

Renewal Assessment Report

Dimethenamid-P

Volume 3 – B.7 Residue data

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B.7 Residue data

Identity of dimethenamid-P

Dimethenamid-P and dimethenamid are herbicides of the chloroacetamide group. They are identical in chemical structure containing one stereochemical centre in the side chain. Dimethenamid-P is the S-enantiomer of the racemic dimethenamid.

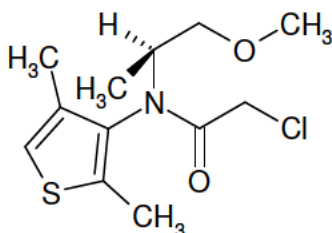


Figure B.7.1-1: Dimethenamid-P

For the active substance a data-package conducted with the racemic mixture was taken into consideration and a bridging concept was applied and accepted for the previous Annex I inclusion of dimethenamid-P. It is therefore concluded that studies performed with dimethenamid can be used to supplement the dimethenamid-P data base for all types of residue studies.

The data already evaluated in the process of Annex I inclusion under Directive 91/414/EEC (DAR, 2000, [ASB2010-10566](#)) and for MRL setting under Reg. (EU) 396/2005 (EFSA 2013, [ASB2013-6081](#)) are summarised and re-evaluated in this assessment report according to current standards and data requirements.

B.7.1 Storage stability of residue

B.7.1.1 Storage stability of residues in plant products

Reference:	KCA 6.1/1
Report	Lehmann A. (2014): Investigation of the storage stability of BAS 656 H (dimethenamid-P) and its metabolites M23, M26, M27 and M30 in plant matrices, BASF Doc ID: 2013/1335905, Study No.: 390391, ASB2014-8339
Guideline(s):	EEC 7032/VI/97 rev. 5, EEC 1607/VI/97 rev. 2 10.06.1999, EEC 91/414, EPA 860.1380, OECD 506
Deviations:	None
GLP:	Yes
Acceptability:	The study is acceptable.

Materials and methods

The study was performed with dimethenamid and its metabolites M23, M26, M27 and M30 in plant matrices. Samples were fortified with the compounds at 0.1 mg/kg each. The fortified samples were stored frozen at about -20 °C in the dark and analysed after 0, 31-52, 103-105, 179-181, 365-369, 544-550 and 745-748 days.

Fortified samples from day 0 and 31-52 were analysed using BASF method No L0179/01. The method

included extraction of dimethenamid-P with methanol, followed by partitioning into dichloromethane and clean-up by SPE. All other samples were analysed using BASF method No L0179/02, which only included extraction with methanol, centrifugation and dilution of the supernatant. Both methods employed LC-MS/MS detection with an LOQ of 0.01 mg/kg. BASF method No L0179/02 is proposed as a surveillance method in Volume 3 CA, B.5 and has been fully validated.

Results and discussions

The results of the storage stability recoveries for the matrices investigated are summarised in Table B.7.1-1. Recoveries are not corrected for concurrent recoveries or for residues in the control sample. However, results at the different sampling time points are compared to the effective fortification level at day zero rather than to the nominal level to compensate for a matrix dependent degradation of the analytes before freezing.

In strawberry dimethenamid-P and the metabolites M23 and M27 were found to be stable over the full 24 month the study. M26 was stable up to 12 month and showed decline to 39 % thereafter, while M30 was stable up to 18 month.

In maize whole plant dimethenamid-P and the metabolites M23, M27 and M30 were found to be stable over the full 24 month of the study. M26 was stable up to 3 month and showed decline to 29 % thereafter. The value at day 42 (63 % recovery) was not considered relevant as the concurrent recoveries for this time point are as well significantly lower compared to the other values for whole wheat plant.

In maize seed dimethenamid-P and the metabolites M23, M26 and M30 were found to be stable over the full 24 month of the study. Recoveries of M27 declined after 45 day to 60 % and remained on this level throughout the study.

In dry beans dimethenamid-P and the metabolites M23, M27, M26 and M30 were found to be stable over the full 24 month of the study.

In oilseed rape neither dimethenamid-P, nor any of the metabolites were found to be stable. Recovery rates after one month ranged between 44-69 %. The applicant argued that problems with the extraction could have been the reason for these low recoveries, despite acceptable concurrent recoveries.

Conclusion

A storage stability study with the commodity categories high acid content (strawberry), high water content (maize whole plant), high starch content (maize seed), high protein content (dry beans) and high oil content (oilseed rape) was performed for dimethenamid-P and its metabolites M23, M26, M27 and M30. For most matrices a storage stability of at least 24 month could be demonstrated. A shorter storage time had to be set for M26 in strawberry and whole maize plant with 12 and 3 month, respectively. M30 was stable in strawberry for 18 month. Oilseed rape showed for all analytes significant degradation already after about one month. Switching the method from L0179/01 to L0179/02 seemed not to be the reason, as recoveries were already low when L0179/01 was still used. The same was found for M27 in maize seed. Therefore an acceptable storage time could not be determined for these analyte crop combinations.

Table B.7.1-1: Results of storage stability experiments with dimethenamid-P and its metabolites M23, M26, M27 and M30 in strawberry, maize whole plant, maize grain, dry bean and oilseed rape and fodder following storage at -20 °C (uncorrected values; assessment based on recoveries standardised to 100 % at day 0)

		0 months (0 d)		1 month (31-51 d)		3 months (103-105 d)		6 months (179-181 d)		12 months (365-369 d)		18 months (544-550 d)		24 months (745-748 d)	
		Stability recoveries	Concurrent recoveries	Stability recoveries	Concurrent recoveries	Stability recoveries	Concurrent recoveries	Stability recoveries	Concurrent recoveries	Stability recoveries	Concurrent recoveries	Stability recoveries	Concurrent recoveries	Stability recoveries	Concurrent recoveries
Dimethenamid-P															
Strawberry (acidic matrix)	Treated (mg/kg)	0.089	0.075	0.086	0.083	0.103	0.100	0.098	0.099	0.092	0.104	0.101	0.097	0.084	0.098
		0.104	0.079	0.092	0.083	0.105	0.098	0.098	0.099	0.101	0.096	0.091	0.097	0.080	0.097
	Mean recovery (mg/kg)	0.096	0.077	0.089	0.083	0.104	0.099	0.098	0.099	0.097	0.100	0.096	0.097	0.082	0.097
	Mean recovery (%) ¹	96	77	89	83	104	99	98	99	97	100	96	97	82	97
	Mean recovery (%)²	100		92		108		102		101		100		85	
Maize whole plant (water matrix)	Treated (mg/kg)	0.088	0.090	0.090	0.098	0.096	0.100	0.091	0.100	0.094	0.106	0.096	0.102	0.090	0.097
		0.089	0.091	0.092	0.097	0.095	0.100	0.092	0.099	0.094	0.106	0.098	0.106	0.091	0.096
	Mean recovery (mg/kg)	0.089	0.090	0.091	0.098	0.095	0.100	0.091	0.100	0.094	0.106	0.097	0.104	0.090	0.097
	Mean recovery (%) ¹	89	90	91	98	95	100	91	100	94	106	97	104	90	97
	Mean recovery (%)²	100		102		107		102		106		109		101	
Maize seed (starch containing matrix)	Treated (mg/kg)	0.098	0.097	0.093	0.111	0.080	0.108	0.086	0.092	0.100	0.110	0.090	0.100	0.076	0.101
		0.090	0.096	0.098	0.111	0.094	0.105	0.091	0.098	0.100	0.113	0.069	0.099	0.079	0.101
	Mean recovery (mg/kg)	0.094	0.097	0.095	0.111	0.087	0.106	0.089	0.095	0.100	0.112	0.080	0.099	0.077	0.101
	Mean recovery (%) ¹	94	97	95	111	87	106	89	95	100	112	80	99	77	101
	Mean recovery (%)²	100		101		92		95		106		85		82	

		0 months (0 d)		1 month (31-51 d)		3 months (103-105 d)		6 months (179-181 d)		12 months (365-369 d)		18 months (544-550 d)		24 months (745-748 d)	
		Stability recoveries	Concurrent recoveries	Stability recoveries	Concurrent recoveries	Stability recoveries	Concurrent recoveries	Stability recoveries	Concurrent recoveries	Stability recoveries	Concurrent recoveries	Stability recoveries	Concurrent recoveries	Stability recoveries	Concurrent recoveries
Dried beans (protein containing matrix)	Treated (mg/kg)	0.089	0.071	0.104	0.103	0.099	0.104	0.091	0.103	0.095	0.110	0.096	0.106	0.086	0.105
		0.084	0.075	0.103	0.104	0.097	0.104	0.093	0.104	0.098	0.108	0.096	0.104	0.086	0.103
	Mean recovery (mg/kg)	0.087	0.073	0.103	0.104	0.098	0.104	0.092	0.103	0.096	0.109	0.096	0.105	0.086	0.104
	Mean recovery (%) ¹	87	73	103	104	98	104	92	103	96	109	96	105	86	104
	Mean recovery (%) ²	100		118		113		106		110		110		99	
Oilseed rape (oil matrix)	Treated (mg/kg)	0.106	0.104	0.072	0.106	0.060	0.100	0.052	0.100	0.066	0.106	0.069	0.103	0.049	0.106
		0.105	0.104	0.074	0.104	0.063	0.100	0.057	0.104	0.066	0.106	0.068	0.102	0.042	0.107
	Mean recovery (mg/kg)	0.106	0.104	0.073	0.105	0.062	0.100	0.054	0.102	0.066	0.106	0.068	0.103	0.045	0.107
	Mean recovery (%) ¹	106	104	73	105	62	100	54	102	66	106	68	103	45	107
	Mean recovery (%) ²	100		69		59		51		62		64		42	
M23															
Strawberry (acidic matrix)	Treated (mg/kg)	0.089	0.086	0.093	0.086	0.093	0.094	0.091	0.101	0.096	0.100	0.089	0.098	0.088	0.095
		0.088	0.092	0.069	0.089	0.096	0.097	0.096	0.099	0.085	0.088	0.092	0.095	0.075	0.098
	Mean recovery (mg/kg)	0.089	0.089	0.081	0.088	0.094	0.096	0.094	0.100	0.091	0.094	0.091	0.096	0.081	0.096
	Mean recovery (%) ¹	89	89	81	88	94	96	94	100	91	94	91	96	81	96
	Mean recovery (%) ²	100		91		106		106		102		102		91	
Maize whole plan (water matrix))	Treated (mg/kg)	0.085	0.091	0.080	0.084	0.090	0.096	0.094	0.103	0.094	0.103	0.098	0.096	0.087	0.096
		0.092	0.089	0.081	0.083	0.091	0.096	0.096	0.104	0.098	0.100	0.096	0.097	0.085	0.097
	Mean recovery (mg/kg)	0.089	0.090	0.081	0.084	0.091	0.096	0.095	0.103	0.096	0.102	0.097	0.096	0.086	0.096

		0 months (0 d)		1 month (31-51 d)		3 months (103-105 d)		6 months (179-181 d)		12 months (365-369 d)		18 months (544-550 d)		24 months (745-748 d)	
		Stability recoveries	Concurrent recoveries	Stability recoveries	Concurrent recoveries	Stability recoveries	Concurrent recoveries	Stability recoveries	Concurrent recoveries	Stability recoveries	Concurrent recoveries	Stability recoveries	Concurrent recoveries	Stability recoveries	Concurrent recoveries
	Mean recovery (%) ¹	89	90	81	84	91	96	95	103	96	102	97	96	86	96
	Mean recovery (%) ²	100		91		102		107		108		109		97	
Maize seed (starch containing matrix)	Treated (mg/kg)	0.085	0.875	0.068	0.094	0.064	0.098	0.066	0.095	0.080	0.105	0.075	0.094	0.066	0.090
		0.084	0.086	0.069	0.095	0.066	0.091	0.067	0.090	0.076	0.103	0.072	0.095	0.062	0.091
	Mean recovery (mg/kg)	0.084	0.480	0.069	0.094	0.065	0.094	0.067	0.093	0.078	0.104	0.073	0.095	0.064	0.091
	Mean recovery (%) ¹	84	480	69	94	65	94	67	93	78	104	73	95	64	91
	Mean recovery (%) ²	100		82		77		80		93		87		76	
Dried beans (protein containing matrix)	Treated (mg/kg)	0.073	0.074	0.084	0.101	0.079	0.099	0.078	0.099	0.087	0.106	0.070	0.102	0.074	0.116
		0.073	0.072	0.084	0.097	0.079	0.096	0.078	0.100	0.083	0.102	0.077	0.101	0.069	0.108
	Mean recovery (mg/kg)	0.073	0.073	0.084	0.099	0.079	0.098	0.078	0.099	0.085	0.104	0.073	0.101	0.072	0.112
	Mean recovery (%) ¹	73	73	84	99	79	98	78	99	85	104	73	101	72	112
	Mean recovery (%) ²	100		115		108		107		116		100		99	
Oilseed rape (oil matrix)	Treated (mg/kg)	0.103	0.108	0.057	0.116	0.045	0.100	0.046	0.100	0.043	0.114	0.044	0.112	0.034	0.109
		0.104	0.104	0.053	0.105	0.043	0.098	0.043	0.101	0.041	0.110	0.047	0.104	0.036	0.108
	Mean recovery (mg/kg)	0.104	0.106	0.055	0.110	0.044	0.099	0.045	0.100	0.042	0.112	0.045	0.108	0.035	0.109
	Mean recovery (%) ¹	104	106	55	110	44	99	45	100	42	112	45	108	35	109
	Mean recovery (%) ²	100		53		42		43		40		43		34	
M26															

		0 months (0 d)		1 month (31-51 d)		3 months (103-105 d)		6 months (179-181 d)		12 months (365-369 d)		18 months (544-550 d)		24 months (745-748 d)	
		Stability recoveries	Concurrent recoveries	Stability recoveries	Concurrent recoveries	Stability recoveries	Concurrent recoveries	Stability recoveries	Concurrent recoveries	Stability recoveries	Concurrent recoveries	Stability recoveries	Concurrent recoveries	Stability recoveries	Concurrent recoveries
Strawberry (acidic matrix)	Treated (mg/kg)	0.086	0.083	0.087	0.073	0.102	0.102	0.072	0.102	0.080	0.099	0.048	0.093	0.036	0.094
		0.091	0.089	0.063	0.078	0.105	0.103	0.082	0.100	0.094	0.092	0.063	0.092	0.031	0.094
	Mean recovery (mg/kg)	0.088	0.086	0.075	0.075	0.104	0.102	0.077	0.101	0.087	0.095	0.056	0.093	0.034	0.094
	Mean recovery (%) ¹	88	86	75	75	104	102	77	101	87	95	56	93	34	94
	Mean recovery (%) ²	100		85		118		88		99		64		39	
Maize whole plant (water matrix)	Treated (mg/kg)	0.086	0.084	0.054	0.073	0.064	0.101	0.048	0.098	0.043	0.101	0.037	0.098	0.027	0.093
		0.088	0.084	0.056	0.072	0.058	0.098	0.048	0.101	0.038	0.100	0.037	0.100	0.025	0.096
	Mean recovery (mg/kg)	0.087	0.084	0.055	0.073	0.061	0.099	0.048	0.099	0.041	0.101	0.037	0.099	0.026	0.095
	Mean recovery (%) ¹	87	84	55	73	61	99	48	99	41	101	37	99	26	95
	Mean recovery (%) ²	100		63		70		55		47		43		29	
Maize seed (starch containing matrix)	Treated (mg/kg)	0.063	0.060	0.069	0.088	0.069	0.102	0.070	0.094	0.078	0.108	0.074	0.099	0.064	0.096
		0.062	0.062	0.072	0.087	0.069	0.100	0.069	0.096	0.074	0.107	0.074	0.097	0.066	0.097
	Mean recovery (mg/kg)	0.062	0.061	0.070	0.087	0.069	0.101	0.069	0.095	0.076	0.107	0.074	0.098	0.065	0.096
	Mean recovery (%) ¹	62	61	70	87	69	101	69	95	76	107	74	98	65	96
	Mean recovery (%) ²	100		113		111		111		123		119		105	
Dried beans (protein containing matrix)	Treated (mg/kg)	0.067	0.064	0.054	0.073	0.078	0.100	0.086	0.096	0.044	0.105	0.089	0.102	0.069	0.106
		0.064	0.064	0.060	0.074	0.080	0.101	0.082	0.097	0.081	0.102	0.082	0.106	0.066	0.094
	Mean recovery (mg/kg)	0.066	0.064	0.057	0.074	0.079	0.100	0.084	0.097	0.062	0.104	0.086	0.104	0.068	0.100
	Mean recovery (%)	66	64	57	74	79	100	84	97	62	104	86	104	68	100

		0 months (0 d)		1 month (31-51 d)		3 months (103-105 d)		6 months (179-181 d)		12 months (365-369 d)		18 months (544-550 d)		24 months (745-748 d)	
		Stability recoveries	Concurrent recoveries	Stability recoveries	Concurrent recoveries	Stability recoveries	Concurrent recoveries	Stability recoveries	Concurrent recoveries	Stability recoveries	Concurrent recoveries	Stability recoveries	Concurrent recoveries	Stability recoveries	Concurrent recoveries
	(%) ¹														
	Mean recovery (%) ²	100		86		120		127		94		130		103	
Oilseed rape (oil matrix)	Treated (mg/kg)	0.117	0.108	0.054	0.106	0.047	0.113	0.044	0.103	0.048	0.105	0.034	0.102	0.036	0.111
		0.118	0.104	0.054	0.098	0.045	0.106	0.048	0.100	0.047	0.105	0.039	0.104	0.035	0.107
	Mean recovery (mg/kg)	0.117	0.106	0.054	0.102	0.046	0.109	0.046	0.101	0.047	0.105	0.037	0.103	0.035	0.109
	Mean recovery (%) ¹	117	106	54	102	46	109	46	101	47	105	37	103	35	109
	Mean recovery (%) ²	100		46		39		39		40		32		30	
M27															
Strawberry (acidic matrix)	Treated (mg/kg)	0.088	0.087	0.086	0.090	0.083	0.096	0.094	0.100	0.092	0.099	0.086	0.096	0.074	0.096
		0.088	0.090	0.074	0.091	0.090	0.095	0.087	0.098	0.079	0.089	0.089	0.095	0.074	0.098
	Mean recovery (mg/kg)	0.088	0.088	0.080	0.090	0.086	0.095	0.091	0.099	0.085	0.094	0.088	0.095	0.074	0.097
	Mean recovery (%) ¹	88	88	80	90	86	95	91	99	85	94	88	95	74	97
	Mean recovery (%) ²	100		91		98		103		97		100		84	
Maize whole plant (water matrix))	Treated (mg/kg)	0.085	0.081	0.066	0.079	0.086	0.098	0.092	0.100	0.086	0.099	0.084	0.097	0.077	0.099
		0.085	0.083	0.072	0.079	0.086	0.098	0.095	0.102	0.088	0.101	0.086	0.099	0.073	0.097
	Mean recovery (mg/kg)	0.085	0.082	0.069	0.079	0.086	0.098	0.093	0.101	0.087	0.100	0.085	0.098	0.075	0.098
	Mean recovery (%) ¹	85	82	69	79	86	98	93	101	87	100	85	98	75	98
	Mean recovery (%) ²	100		81		101		109		102		100		88	
Maize seed	Treated	0.091	0.081	0.052	0.086	0.052	0.088	0.061	0.085	0.065	0.092	0.061	0.092	0.058	0.085

		0 months (0 d)		1 month (31-51 d)		3 months (103-105 d)		6 months (179-181 d)		12 months (365-369 d)		18 months (544-550 d)		24 months (745-748 d)	
		Stability recoveries	Concurrent recoveries	Stability recoveries	Concurrent recoveries	Stability recoveries	Concurrent recoveries	Stability recoveries	Concurrent recoveries	Stability recoveries	Concurrent recoveries	Stability recoveries	Concurrent recoveries	Stability recoveries	Concurrent recoveries
(starch containing matrix)	(mg/kg)	0.090	0.080	0.056	0.085	0.056	0.088	0.062	0.086	0.068	0.092	0.060	0.092	0.059	0.088
	Mean recovery (mg/kg)	0.090	0.081	0.054	0.086	0.054	0.088	0.061	0.086	0.066	0.092	0.061	0.092	0.058	0.087
	Mean recovery (%) ¹	90	81	54	86	54	88	61	86	66	92	61	92	58	87
	Mean recovery (%) ²	100		60		60		68		73		68		64	
Dried beans (protein containing matrix)	Treated (mg/kg)	0.086	0.088	0.084	0.098	0.078	0.098	0.076	0.096	0.078	0.102	0.079	0.102	0.066	0.104
		0.083	0.088	0.082	0.094	0.075	0.097	0.075	0.096	0.083	0.102	0.079	0.104	0.067	0.091
	Mean recovery (mg/kg)	0.084	0.088	0.083	0.096	0.076	0.098	0.075	0.096	0.080	0.102	0.079	0.103	0.067	0.098
	Mean recovery (%) ¹	84	88	83	96	76	98	75	96	80	102	79	103	67	98
	Mean recovery (%) ²	100		99		91		89		95		94		80	
Oilseed rape (oil matrix)	Treated (mg/kg)	0.095	0.104	0.046	0.112	0.042	0.101	0.040	0.097	0.040	0.112	0.045	0.107	0.029	0.106
		0.090	0.102	0.052	0.106	0.040	0.103	0.043	0.102	0.041	0.110	0.039	0.110	0.030	0.110
	Mean recovery (mg/kg)	0.092	0.103	0.049	0.109	0.041	0.102	0.041	0.099	0.040	0.111	0.042	0.108	0.029	0.108
	Mean recovery (%) ¹	92	103	49	109	41	102	41	99	40	111	42	108	29	108
	Mean recovery (%) ²	100		53		45		45		44		46		32	
M30															
Strawberry (acidic matrix)	Treated (mg/kg)	0.089	0.090	0.088	0.087	0.089	0.096	0.089	0.100	0.077	0.094	0.089	0.092	0.062	0.092
		0.092	0.093	0.080	0.090	0.092	0.093	0.081	0.097	0.077	0.091	0.086	0.095	0.058	0.096
	Mean recovery (mg/kg)	0.091	0.091	0.084	0.089	0.090	0.094	0.085	0.098	0.077	0.092	0.088	0.094	0.060	0.094
	Mean recovery (%)	91	91	84	89	90	94	85	98	77	92	88	94	60	94

		0 months (0 d)		1 month (31-51 d)		3 months (103-105 d)		6 months (179-181 d)		12 months (365-369 d)		18 months (544-550 d)		24 months (745-748 d)	
		Stability recoveries	Concurrent recoveries	Stability recoveries	Concurrent recoveries	Stability recoveries	Concurrent recoveries	Stability recoveries	Concurrent recoveries	Stability recoveries	Concurrent recoveries	Stability recoveries	Concurrent recoveries	Stability recoveries	Concurrent recoveries
	(%) ¹														
	Mean recovery (%)²	100		92		99		93		85		97		66	
Maize whole plan (water matrix)	Treated (mg/kg)	0.079	0.084	0.065	0.081	0.083	0.095	0.079	0.100	0.084	0.093	0.084	0.098	0.068	0.091
		0.076	0.084	0.068	0.082	0.085	0.095	0.082	0.103	0.081	0.095	0.088	0.100	0.073	0.089
	Mean recovery (mg/kg)	0.078	0.084	0.067	0.081	0.084	0.095	0.080	0.102	0.083	0.094	0.086	0.099	0.070	0.090
	Mean recovery (%) ¹	78	84	67	81	84	95	80	102	83	94	86	99	70	90
	Mean recovery (%)²	100		86		108		103		106		110		90	
Maize seed (starch containing matrix)	Treated (mg/kg)	0.066	0.074	0.056	0.081	0.057	0.089	0.059	0.092	0.075	0.106	0.065	0.094	0.058	0.093
		0.072	0.076	0.056	0.079	0.056	0.091	0.058	0.095	0.071	0.103	0.066	0.096	0.059	0.096
	Mean recovery (mg/kg)	0.069	0.075	0.056	0.080	0.057	0.090	0.059	0.093	0.073	0.104	0.065	0.095	0.058	0.095
	Mean recovery (%) ¹	69	75	56	80	57	90	59	93	73	104	65	95	58	95
	Mean recovery (%)²	100		81		83		86		106		94		84	
Dried beans (protein containing matrix)	Treated (mg/kg)	0.072	0.085	0.060	0.084	0.070	0.093	0.070	0.093	0.072	0.100	0.077	0.101	0.048	0.104
		0.070	0.094	0.060	0.085	0.078	0.088	0.070	0.089	0.073	0.100	0.078	0.098	0.060	0.093
	Mean recovery (mg/kg)	0.071	0.089	0.060	0.085	0.074	0.091	0.070	0.091	0.073	0.100	0.078	0.100	0.054	0.098
	Mean recovery (%) ¹	71	89	60	85	74	91	70	91	73	100	78	100	54	98
	Mean recovery (%)²	100		85		104		99		103		110		76	
Oilseed rape (oil)	Treated (mg/kg)	0.085	0.104	0.036	0.108	0.041	0.098	0.040	0.098	0.040	0.106	0.032	0.100	0.030	0.105
		0.086	0.101	0.038	0.100	0.040	0.097	0.037	0.095	0.038	0.109	0.036	0.100	0.027	0.105

		0 months (0 d)		1 month (31-51 d)		3 months (103-105 d)		6 months (179-181 d)		12 months (365-369 d)		18 months (544-550 d)		24 months (745-748 d)	
		Stability recoveries	Concurrent recoveries	Stability recoveries	Concurrent recoveries	Stability recoveries	Concurrent recoveries	Stability recoveries	Concurrent recoveries	Stability recoveries	Concurrent recoveries	Stability recoveries	Concurrent recoveries	Stability recoveries	Concurrent recoveries
matrix)	Mean recovery (mg/kg)	0.085	0.103	0.037	0.104	0.040	0.097	0.038	0.097	0.039	0.107	0.034	0.100	0.028	0.105
	Mean recovery (%) ¹	85	103	37	104	40	97	38	97	39	107	34	100	28	105
	Mean recovery (%) ²	100		44		47		45		46		40		33	

¹ Nominal

² Standardised

Reference:	KCA 6.1/2
Report	Oppinger, M. (2014): Investigation of the storage stability of BAS 656 H (dimethenamid-P) and its metabolites M23, M26, M27 and M30 in oily matrices, BASF DocID: 2013/1335906, Study code: 432630, ASB2014-8340
Guideline(s):	EEC 7032/VI/97 rev. 5, EEC 1607/VI/97 rev. 2 10.06.1999, EEC 91/414, OPPTS 860.1380, OECD 506
Deviations:	None
GLP:	Yes
Acceptability:	The study is acceptable.

Materials and methods

The study was performed with dimethenamid and its metabolites M23, M26, M27 and M30 in oilseed rape seed, soya bean seed and sunflower seed. Samples were fortified with the compounds at 0.1 mg/kg each. The fortified samples were stored frozen at about -20 °C in the dark and analysed after 0, 3, 7, 14, 30, 60, 90 and 180 days.

All samples were analysed using BASF method No L0179/02, which included extraction with methanol, centrifugation and dilution of the supernatant. However, a high speed homogeniser was used here, compared to a low speed homogeniser, being used in the study by Lehmann (2014, [ASB2014-8339](#)). The method employed LC-MS/MS detection with an LOQ of 0.01 mg/kg. BASF method No L0179/02 is proposed as a surveillance method in Volume 3 CA, B.5 and has been fully validated.

Results and discussions

The results of the storage stability recoveries for the matrices investigated are summarised in Table B.7.1-2. Recoveries are not corrected for concurrent recoveries or for residues in the control sample. However, results at the different sampling time points are compared to the effective fortification level at day zero rather than to the nominal level to compensate for a matrix dependent degradation of the analytes before freezing.

No degradation was observed during the full study time of 90 days for any of the crop analyte combination tested. For M26 at day 60 nominal recoveries >130 % were detected. However, since the concurrent recoveries at this time point were similar, it was concluded that these excessive recoveries were of analytical origin. Consequently, the time point was disregarded.

Conclusion

Since in the study by Lehmann (2014, [ASB2014-8339](#)) neither dimethenamid-P nor its metabolites were found to be stable in high oil content matrices (oilseed rape), the applicant provided a second storage stability study with commodities of high oil content (oilseed rape, soya bean seed and sunflower seed) using as well method No L0179/02 as in Lehmann (2014, [ASB2014-8339](#)). In this interim report storage stability of at least 3 month (90 days) could be demonstrated for dimethenamid-P and its metabolites M23, M26, M27 and M30. The improved stability recoveries compared to the study by Lehmann (2014, [ASB2014-8339](#)) were explained by the applicant by the use of a high speed homogeniser instead of a low speed homogeniser. Assuming a smaller particle size when using the high speed homogeniser, the RMS considered this as an acceptable explanation, as it is known that extraction efficiency increases with smaller particles size.

Table B.7.1-2: Results of storage stability experiments with dimethenamid-P and metabolites M23, M26, M27 and M30 in strawberry oilseed rape, soya bean seed and sunflower seed following storage at -20 °C (uncorrected values; assessment based on recoveries standardised to 100 % at day 0)

		0 months (0 d)		0.1 months (3 d)		0.25 months (7 d)		0.5 months (14 d)		1 months (29 d)		2 months (59 d)		3 months (94 d)	
		Stability recoveries	Concurrent recoveries	Stability recoveries	Concurrent recoveries	Stability recoveries	Concurrent recoveries	Stability recoveries	Concurrent recoveries	Stability recoveries	Concurrent recoveries	Stability recoveries	Concurrent recoveries	Stability recoveries	Concurrent recoveries
Dimethenamid-P															
Oilseed rape	Treated (mg/kg)	0.101	0.101	0.104	0.108	0.095	0.101	0.099	0.106	0.115	0.104	0.100	0.110	0.100	0.108
		0.102	0.102	0.086	0.107	0.090	0.106	0.098	0.104	0.108	0.113	0.095	0.111	0.094	0.110
	Mean recovery (mg/kg)	0.102	0.102	0.095	0.108	0.093	0.103	0.098	0.105	0.112	0.108	0.098	0.111	0.097	0.109
	Mean recovery (%) ¹	102	102	95	108	93	103	98	105	112	108	98	111	97	109
	Mean recovery (%)²	100		93		91		96		110		96		95	
Soya bean seed	Treated (mg/kg)	0.099	0.092	0.102	0.105	0.095	0.103	0.109	0.110	0.114	0.106	0.108	0.108	0.106	0.114
		0.098	0.093	0.098	0.104	0.100	0.103	0.114	0.112		0.109	0.106	0.108	0.106	0.111
	Mean recovery (mg/kg)	0.098	0.092	0.100	0.105	0.097	0.103	0.111	0.111	0.114	0.107	0.107	0.108	0.106	0.112
	Mean recovery (%) ¹	98	92	100	105	97	103	111	111	114	107	107	108	106	112
	Mean recovery (%)²	100		102		95		113		116		102		108	
Sunflower seed	Treated (mg/kg)	0.101	0.100	0.102	0.104	0.101	0.105	0.112	0.113	0.112	0.109	0.114	0.110	0.109	0.106
		0.100	0.098	0.100	0.104	0.104	0.105	0.112	0.112	0.116	0.107	0.111	0.109	0.111	0.106
	Mean recovery	0.101	0.099	0.101	0.104	0.102	0.105	0.112	0.112	0.114	0.108	0.113	0.109	0.110	0.106

		0 months (0 d)		0.1 months (3 d)		0.25 months (7 d)		0.5 months (14 d)		1 months (29 d)		2 months (59 d)		3 months (94 d)	
		Stability recoveries	Concurrent recoveries	Stability recoveries	Concurrent recoveries	Stability recoveries	Concurrent recoveries	Stability recoveries	Concurrent recoveries	Stability recoveries	Concurrent recoveries	Stability recoveries	Concurrent recoveries	Stability recoveries	Concurrent recoveries
	(mg/kg)														
	Mean recovery (%) ¹	101	99	101	104	102	105	112	112	114	108	113	109	110	106
	Mean recovery (%)²	100		100		101		111		113		112		109	
M23															
Oilseed rape	Treated (mg/kg)	0.103	0.107	0.085	0.114	0.082	0.099	0.089	0.115	0.103	0.104	0.098	0.111	0.098	0.108
		0.103	0.108	0.080	0.112	0.077	0.098	0.095	0.111	0.110	0.109	0.096	0.112	0.099	0.108
	Mean recovery (mg/kg)	0.103	0.107	0.082	0.113	0.080	0.098	0.092	0.113	0.106	0.107	0.097	0.112	0.099	0.108
	Mean recovery (%) ¹	103	107	82	113	80	98	92	113	106	107	97	112	99	108
	Mean recovery (%)²	100		80		78		89		103		94		96	
Soya bean seed	Treated (mg/kg)	0.086	0.082	0.075	0.106	0.071	0.102	0.090	0.105	0.104	0.109	0.094	0.112	0.101	0.104
		0.085	0.084	0.076	0.103	0.074	0.098	0.088	0.108	0.105	0.109	0.096	0.108	0.101	0.107
	Mean recovery (mg/kg)	0.085	0.083	0.075	0.105	0.072	0.100	0.089	0.106	0.105	0.109	0.095	0.110	0.101	0.105
	Mean recovery (%) ¹	85	83	75	105	72	100	89	106	105	109	95	110	101	105
	Mean recovery (%)²	100		88		85		105		124		112		119	
Sunflower seed	Treated (mg/kg)	0.090	0.081	0.081	0.105	0.082	0.098	0.089	0.104	0.108	0.104	0.103	0.108	0.108	0.101
		0.083	0.084	0.080	0.106	0.080	0.105	0.086	0.100	0.106	0.104	0.102	0.106	0.110	0.100

		0 months (0 d)		0.1 months (3 d)		0.25 months (7 d)		0.5 months (14 d)		1 months (29 d)		2 months (59 d)		3 months (94 d)	
		Stability recoveries	Concurrent recoveries	Stability recoveries	Concurrent recoveries	Stability recoveries	Concurrent recoveries	Stability recoveries	Concurrent recoveries	Stability recoveries	Concurrent recoveries	Stability recoveries	Concurrent recoveries	Stability recoveries	Concurrent recoveries
	Mean recovery (mg/kg)	0.087	0.082	0.081	0.105	0.081	0.101	0.088	0.102	0.107	0.104	0.102	0.107	0.109	0.101
	Mean recovery (%) ¹	87	82	81	105	81	101	88	102	107	104	102	107	109	101
	Mean recovery (%)²	100		93		93		101		123		117		125	
M26															
Oilseed rape	Treated (mg/kg)	0.100	0.100	0.080	0.130	0.090	0.117	0.105	0.137	0.094	0.100	0.129	0.165	0.080	0.140
		0.099	0.103	0.072	0.124	0.084	0.121	0.102	0.134	0.074	0.111	0.130	0.167	0.075	0.145
	Mean recovery (mg/kg)	0.100	0.102	0.076	0.127	0.087	0.119	0.104	0.136	0.084	0.106	0.130	0.166	0.077	0.142
	Mean recovery (%) ¹	100	102	76	127	87	119	104	136	84	106	130	166	77	142
	Mean recovery (%)²	100		76		87		104		84		130		77	
Soya bean seed	Treated (mg/kg)	0.081	0.088	0.076	0.131	0.072	0.121	0.093	0.123	0.095	0.105	0.135	0.189	0.082	0.134
		0.078	0.078	0.079	0.131	0.081	0.125	0.100	0.138	0.090	0.104	0.132	0.184	0.087	0.133
	Mean recovery (mg/kg)	0.079	0.083	0.078	0.131	0.077	0.123	0.096	0.130	0.093	0.105	0.133	0.187	0.084	0.133
	Mean recovery (%) ¹	79	83	78	131	77	123	96	130	93	105	133	187	84	133
	Mean recovery (%)²	100		99		97		122		117		168		106	
Sunflower	Treated	0.076	0.083	0.081	0.118	0.092	0.116	0.109	0.126	0.100	0.105	0.156	0.176	0.103	0.133

		0 months (0 d)		0.1 months (3 d)		0.25 months (7 d)		0.5 months (14 d)		1 months (29 d)		2 months (59 d)		3 months (94 d)	
		Stability recoveries	Concurrent recoveries	Stability recoveries	Concurrent recoveries	Stability recoveries	Concurrent recoveries	Stability recoveries	Concurrent recoveries	Stability recoveries	Concurrent recoveries	Stability recoveries	Concurrent recoveries	Stability recoveries	Concurrent recoveries
seed	(mg/kg)	0.077	0.080	0.086	0.116	0.093	0.119	0.106	0.121	0.101	0.111	0.158	0.178	0.103	0.131
	Mean recovery (mg/kg)	0.077	0.082	0.083	0.117	0.093	0.118	0.107	0.123	0.101	0.108	0.157	0.177	0.103	0.132
	Mean recovery (%) ¹	77	82	83	117	93	118	107	123	101	108	157	177	103	132
	Mean recovery (%) ²	100		108		121		139		131		204		134	
M27															
Oilseed rape	Treated (mg/kg)	0.102	0.103	0.076	0.120	0.073	0.099	0.085	0.119	0.106	0.110	0.094	0.108	0.082	0.117
		0.102	0.103	0.070	0.116	0.070	0.094	0.083	0.115	0.105	0.114	0.093	0.108	0.081	0.119
	Mean recovery (mg/kg)	0.102	0.103	0.073	0.118	0.071	0.096	0.084	0.117	0.106	0.112	0.093	0.108	0.082	0.118
	Mean recovery (%) ¹	102	103	73	118	71	96	84	117	106	112	93	108	82	118
	Mean recovery (%) ²	100		71		70		82		104		91		80	
Soya bean seed	Treated (mg/kg)	0.088	0.081	0.077	0.103	0.076	0.105	0.086	0.107	0.114	0.105	0.094	0.108	0.109	0.118
		0.090	0.081	0.078	0.106	0.074	0.103	0.089	0.107	0.115	0.109	0.090	0.108	0.105	0.117
	Mean recovery (mg/kg)	0.089	0.081	0.077	0.105	0.075	0.104	0.087	0.107	0.115	0.107	0.092	0.108	0.107	0.118
	Mean recovery (%) ¹	89	81	77	105	75	104	87	107	115	107	92	108	107	118
	Mean recovery (%) ²	100		87		84		98		129		103		120	

		0 months (0 d)		0.1 months (3 d)		0.25 months (7 d)		0.5 months (14 d)		1 months (29 d)		2 months (59 d)		3 months (94 d)	
		Stability recoveries	Concurrent recoveries	Stability recoveries	Concurrent recoveries	Stability recoveries	Concurrent recoveries	Stability recoveries	Concurrent recoveries	Stability recoveries	Concurrent recoveries	Stability recoveries	Concurrent recoveries	Stability recoveries	Concurrent recoveries
Sunflower seed	Treated (mg/kg)	0.075	0.079	0.076	0.107	0.073	0.098	0.076	0.103	0.104	0.101	0.098	0.103	0.103	0.106
		0.078	0.077	0.077	0.106	0.072	0.105	0.077	0.097	0.105	0.103	0.095	0.099	0.104	0.105
	Mean recovery (mg/kg)	0.077	0.078	0.077	0.107	0.072	0.101	0.077	0.100	0.104	0.102	0.096	0.101	0.104	0.106
	Mean recovery (%) ¹	77	78	77	107	72	101	77	100	104	102	96	101	104	106
	Mean recovery (%) ²	100		100		93		100		135		125		135	
M30															
Oilseed rape	Treated (mg/kg)	0.104	0.099	0.078	0.097	0.067	0.089	0.076	0.097	0.109	0.103	0.061	0.087	0.085	0.136
		0.095	0.098	0.070	0.100	0.057	0.087	0.073	0.098	0.113	0.104	0.051	0.090	0.090	0.141
	Mean recovery (mg/kg)	0.099	0.098	0.074	0.098	0.062	0.088	0.075	0.097	0.111	0.104	0.056	0.089	0.088	0.138
	Mean recovery (%) ¹	99	98	74	98	62	88	75	97	111	104	56	89	88	138
	Mean recovery (%) ²	100		75		63		76		112		57		89	
Soya bean seed	Treated (mg/kg)	0.086	0.076	0.070	0.092	0.066	0.089	0.084	0.102	0.102	0.103	0.068	0.091	0.102	0.130
		0.086	0.080	0.075	0.094	0.063	0.092	0.082	0.094	0.097	0.107	0.067	0.089	0.094	0.127
	Mean recovery (mg/kg)	0.086	0.078	0.073	0.093	0.065	0.090	0.083	0.098	0.100	0.105	0.067	0.090	0.098	0.128
	Mean recovery (%) ¹	86	78	73	93	65	90	83	98	100	105	67	90	98	128
	Mean	100		85		76		97		116		78		114	

		0 months (0 d)		0.1 months (3 d)		0.25 months (7 d)		0.5 months (14 d)		1 months (29 d)		2 months (59 d)		3 months (94 d)	
		Stability recoveries	Concurrent recoveries	Stability recoveries	Concurrent recoveries	Stability recoveries	Concurrent recoveries	Stability recoveries	Concurrent recoveries	Stability recoveries	Concurrent recoveries	Stability recoveries	Concurrent recoveries	Stability recoveries	Concurrent recoveries
	recovery (%) ²														
Sunflower seed	Treated (mg/kg)	0.078	0.077	0.068	0.099	0.063	0.092	0.068	0.092	0.112	0.105	0.080	0.082	0.088	0.105
		0.074	0.075	0.069	0.100	0.063	0.093	0.064	0.084	0.106	0.102	0.079	0.080	0.088	0.113
	Mean recovery (mg/kg)	0.076	0.076	0.068	0.099	0.063	0.092	0.066	0.088	0.109	0.104	0.079	0.081	0.088	0.109
	Mean recovery (%) ¹	76	76	68	99	63	92	66	88	109	104	79	81	88	109
	Mean recovery (%) ²	100		90		83		87		143		104		116	

¹ Nominal

² Standardised

Reference:	KCA 6.1/3
Report	Bade, T. R. (1992): Stability of SAN-582H and its metabolites in stored frozen corn samples, BASF DocID: 92/12400, Project No: 414108, RIP1999-859
Guideline(s):	US EPA Pesticide Assessment Guidelines, Subdivision O, Residue Chemistry, Series 171-4(e)
Deviations:	None
GLP:	Yes
Acceptability:	The study is considered supplementary only.

Materials and methods

The study was performed with dimethenamid and its oxalamide metabolite (M23). Two different approaches were used in this study. In the first approach, samples of maize forage, silage, grain and fodder were fortified with dimethenamid and M23 at 0.5 mg/kg each, stored under deep freeze conditions at -20 °C and analysed after 0, 3, 6, 12 and 21 months. The samples taken at the respective time points were extracted with water/methanol/conc. HCl (19.5 + 80 + 0.5, v/v/v), followed by liquid/liquid partitioning into ethyl ether/dichloromethane (1+1, v/v). After derivatisation the residue were cleaned-up on C18 SPE column.

In the second approach, a re-analysis of radioactive metabolism samples was performed that had been analysed several years prior. Treated samples from the radioactive maize metabolism study were analysed in the winter of 1989-1990 and then re-analysed in October, 1991, after 1.5 to 2 years of frozen storage. Samples were initially extracted with methanol/water (50/50, v/v) followed by sequential extraction with hexane, dichloromethane, methanol and water. Results of the re-analyses were compared to the original analyses.

Fortified samples from 0, 3, 6 and 12 month intervals were analysed using Sandoz method AM-0840-0790-0 (superseded by method #0840-0391-1) using GC-MSD with a validated LOQ of 0.1 mg/kg. The samples from the 21 month interval were analysed using Sandoz method BS-2304 using GC-NPD with a validated LOQ of 0.02 mg/kg.

Results and discussions

The results of the storage stability recoveries for maize forage, silage, grain and fodder are summarised in Table B.7.1-3. Recoveries are not corrected for concurrent recoveries or for residues in the control sample. However, results at the different sampling time points are compared to the effective fortification level at day zero rather than to the nominal level to compensate for a matrix dependent effect on the analytes before freezing.

Concurrent and stability recoveries for dimethenamid and M23 in all matrices showed generally a large degree of analytical variability. For example, concurrent recoveries ranged between 37 and 143 % and were frequently accompanied by similar results for the stability recoveries at the respective time points. Also, as nominal stability recoveries at day 0 often exceed 100 %, standardised recoveries resulted frequently below 70 %. Consequently normalisation to day 0 provides only limited information on the stability.

The re-analysis of radioactive samples from the metabolism study showed a similar variability ranging from 30 % to 205 % of the amounts detected during the original analysis.

Conclusion

In the Study by Bade (1992, [RIP1999-859](#)) storage stability of dimethenamid and its metabolite M23 was investigated in maize forage, silage, grain and fodder when stored at -20 °C. Due to the analytical variability it was not possible to determine storage stabilities based on neither nominal recoveries nor day 0 standardised recoveries. However, if the stability recoveries are adjusted for concurrent recoveries, a storage stability of 21 and 12 month can be set in maize forage, silage, grain and fodder for dimethenamid and M23, respectively. Also, the re-analysis of samples from metabolism studies

using ^{14}C -labelled incurred residues provides only limited information, since it remains unknown whether or not equilibrium for the degradation was reached before the first analysis. In conclusion, the study can be considered as supplementary only.

Table B.7.1-3: Results of storage stability experiments with dimethenamid in maize forage, silage, grain and fodder following storage at -20 °C (uncorrected values; assessment based on recoveries standardised to 100 % at day 0)

Sample		Storage interval (month)									
		0 months (0 d)		3 months (90 d)		6 months (180 d)		12 months (366 d)		21 months (632-657 d)	
		Stability recoveries	Concurrent recoveries	Stability recoveries	Concurrent recoveries	Stability recoveries	Concurrent recoveries	Stability recoveries	Concurrent recoveries	Stability recoveries	Concurrent recoveries
Dimethenamid											
Forage (water matrix)	Control (mg/kg)	0.000		0.000		0.013		0.000		0.009	
	Treated (mg/kg)	0.710	0.664	0.339	0.313	0.506	0.508	0.363	0.431	0.387	0.439
		0.679	0.765	0.376	0.284	0.542	0.551	0.310	0.419	0.414	0.479
		0.617		0.220		0.502		0.383		0.411	
	Mean recovery (mg/kg)	0.669	0.715	0.312	0.299	0.517	0.530	0.352	0.425	0.404	0.459
	Mean recovery (%) ¹	134	143	62	60	103	106	70	85	81	92
	Mean recovery (%)²	100		46		77		52		60	
Silage (water matrix)	Control (mg/kg)	0		0		0.001		0.006		0	
	Treated (mg/kg)	0.743	0.713	0.372	0.184	0.583	0.563	0.337	0.474	0.346	0.453
		0.727		0.389	0.183	0.510	0.507	0.347	0.743	0.448	0.513
		0.688		0.402		0.585		0.404		0.422	
	Mean recovery (mg/kg)	0.719	0.713	0.388	0.184	0.559	0.535	0.363	0.609	0.405	0.483
	Mean recovery (%) ¹	144	143	78	92	112	107	73	122	81	97
	Mean recovery (%)²	100		54		78		51		56	
Grain (starch containing matrix)	Control (mg/kg)	0		0		0.005		0		0	
	Treated (mg/kg)	0.581	0.649	0.341	0.201	0.498	0.508	0.497	0.534	0.354	0.427
		0.579	0.574	0.334	0.165	0.559	0.573	0.462	0.522	0.328	

Sample		Storage interval (month)									
		0 months (0 d)		3 months (90 d)		6 months (180 d)		12 months (366 d)		21 months (632-657 d)	
		Stability recoveries	Concurrent recoveries	Stability recoveries	Concurrent recoveries	Stability recoveries	Concurrent recoveries	Stability recoveries	Concurrent recoveries	Stability recoveries	Concurrent recoveries
		0.274		0.343		0.523		0.491		0.367	
	Mean recovery (mg/kg)	0.478	0.612	0.339	0.183	0.527	0.541	0.483	0.528	0.350	0.427
	Mean recovery (%) ¹	96	122	68	92	105	108	97	106	70	85
	Mean recovery (%)²	100		71		109		101		73	
Fodder (water matrix)	Control (mg/kg)	0		0		0.003		0.006		0.003	
	Treated (mg/kg)	0.529	0.651	0.331	0.181	0.601	0.533	0.483	0.663	0.454	0.507
		0.536		0.371	0.189	0.575	0.513	0.321		0.321	0.470
				0.284		0.464		0.520		0.480	
	Mean recovery (mg/kg)	0.533	0.651	0.329	0.185	0.547	0.523	0.441	0.663	0.418	0.489
	Mean recovery (%) ¹	107	130	66	37	109	105	88	133	84	98
	Mean recovery (%)²	100		62		102		82		79	
M23											
Forage (water matrix)	Control (mg/kg)	0.000		0.000		0.000		0.002			
	Treated (mg/kg)	0.650	0.619	0.314	0.273	0.541	0.547	0.407	0.481		
		0.617	0.672	0.352	0.229	0.527	0.507	0.280	0.469		
		0.486		0.224		0.504		0.396			
	Mean recovery (mg/kg)	0.584	0.646	0.297	0.251	0.524	0.527	0.361	0.475		
	Mean recovery (%) ¹	117	129	59	50	105	105	72	95		
	Mean recovery (%)²	100		50		90		62			

Sample		Storage interval (month)									
		0 months (0 d)		3 months (90 d)		6 months (180 d)		12 months (366 d)		21 months (632-657 d)	
		Stability recoveries	Concurrent recoveries	Stability recoveries	Concurrent recoveries	Stability recoveries	Concurrent recoveries	Stability recoveries	Concurrent recoveries	Stability recoveries	Concurrent recoveries
Silage (water matrix)	Control (mg/kg)	0.003		0.000		0.003		0.005			
	Treated (mg/kg)	0.696	0.685	0.308	0.142	0.517	0.544	0.383	0.538		
		0.671		0.307	0.147	0.481	0.528	0.393	0.934		
		0.672		0.345		0.552		0.490			
	Mean recovery (mg/kg)	0.680	0.685	0.320	0.145	0.517	0.536	0.422	0.736		
	Mean recovery (%) ¹	136	137	64	72	103	107	84	147		
	Mean recovery (%)²	100		47		76		62			
Grain (starch containing matrix)	Control (mg/kg)	0.022		0		0.001		0.002			
	Treated (mg/kg)	0.571	0.648	0.241	0.146	0.478	0.684	0.616	0.682		
		0.563	0.582	0.244	0.104	0.528	0.538	0.699	0.612		
		0.199		0.224		0.534		0.515			
	Mean recovery (mg/kg)	0.444	0.615	0.236	0.125	0.513	0.611	0.610	0.647		
	Mean recovery (%) ¹	89	123	47	63	103	122	122	129		
	Mean recovery (%)²	100		53		116		137			
Fodder (water matrix)	Control (mg/kg)	0.000		0.000		0.003		0.012			
	Treated (mg/kg)	0.517	0.656	0.130	0.168	0.603	0.547	0.541	0.728		
		0.477		0.236	0.171	0.523		0.406			
				0.215		0.508		0.663			
	Mean recovery (mg/kg)	0.497	0.656	0.194	0.170	0.545	0.547	0.537	0.728		
	Mean recovery (%) ¹	99	131	39	34	109	109	107	146		

Sample		Storage interval (month)									
		0 months (0 d)		3 months (90 d)		6 months (180 d)		12 months (366 d)		21 months (632-657 d)	
		Stability recoveries	Concurrent recoveries	Stability recoveries	Concurrent recoveries	Stability recoveries	Concurrent recoveries	Stability recoveries	Concurrent recoveries	Stability recoveries	Concurrent recoveries
	Mean recovery (%) ²	100		39		110		108			

¹ Nominal

² Standardised

³ Samples fortified with 0.2 mg/kg

B.7.1.2 Storage stability of residues in organic solvents

Reference:	KCA 6.1/4
Report	Wisson, M. (1994): Dimethenamid: Stability in various organic solvents, BASF DocID: 94/11821, Project No: 41'205, RIP1999-860
Guideline(s):	No guideline available
Deviations:	Not applicable
GLP:	Yes
Acceptability:	The study is considered supplementary only.

Materials and methods

A study has been conducted to investigate the stability of dimethenamid and dimethyl sulfoxide in hexane, toluene, dichloromethane, ethyl acetate, acetone, methanol, acetonitrile. Solvent samples were spiked with 1 mg/mL dimethenamid and stored at 22 °C for 12 month (except dimethyl sulfoxide at 22 °C for 4 weeks and at 54 °C for 1 week). All samples were analysed by GC-FID, except dimethyl sulfoxide, which was analysed by HPLC-UV.

Results and discussions

Measured residues of dimethenamid in organic solvents stored at 22 °C are presented in Table B.7.1-4. No decline in measured concentrations of dimethenamid was observed over a period of 12 month. Dimethyl sulfoxide was stable at 22 °C for 4 weeks and at 54 °C for 1 week with a mean recovery equal to 97.4 %.

Table B.7.1-4: Results of storage stability experiments with dimethenamid in organic solvents following storage at 22 °C

Matrix	Average of duplicate analyses (% dimethenamid) and storage duration (month)									
	0	1	2	3	4	5	6	8	10	12
Hexane	97.1	98.5	97.3	97.7	94.5	N/A	95.8	95.7	96.7	99.1
Toluene	105.6	106.1	106.4	106.1	105.8	105.3	106.1	100.9	103.9	104.5
Dichloromethane	98.9	96.7	95.5	100.5	96.9	102.3	100.6	94.1	92.9	92.9
Ethyl acetate	99.2	99.0	98.4	97.7	101.3	N/A	100.1	104.9	105.0	101.1
Acetone	99.7	98.7	97.5	98.8	98.6	N/A	97.2	100.6	97.7	99.6
Methanol	95.5	97.1	95.6	95.0	94.4	97.2	96.0	98.2	95.8	93.7
Acetonitrile	95.5	94.9	92.2	89.9	96.8	N/A	96.9	98.6	97.7	99.6

Conclusion

It is concluded that dimethenamid is stable for at least 12 month in hexane, dichloromethane, ethyl acetate, acetone, methanol, acetonitrile when stored at 22°C. Dimethyl sulfoxide is stable at 22 °C for 4 weeks and at 54 °C for 1 week.

B.7.1.3 Overall conclusion on storage stability of dimethenamid-P in samples and extracts

It is concluded that residues of dimethenamid-P and its metabolite M23 are stable under deep frozen conditions (<-20 °C) for at least 24 months in commodities with high acid content (strawberry), high water content (maize whole plant), high starch content (maize seed), high protein content (dry beans). Shorter storage times are determined for M26 in strawberry and whole maize plant with 12 and 3 month, respectively, and for M30 in strawberry with 18 month. Additionally, storage stability could not be demonstrated for M27 in maize seed (recoveries after 45 days at 60 %).

In the study by Lehmann (2014, [ASB2014-8339](#)) neither dimethenamid-P nor its metabolites were found to be stable in high oil content matrices (oilseed rape) with recoveries ranging between 44 - 69 % after 31 days of storage. The applicant argued that problems with the extraction could have been the reason for these low recoveries, despite acceptable concurrent recoveries. Consequently an additional storage stability study using the identical extraction method was provided by Oppinger (2014, [ASB2014-8340](#)) for high oil matrices only (oilseed rape seed, soya bean seed and sunflower seed). In this interim report a storage stability of dimethenamid-P and its metabolites could be proven for at least 3 months.

The storage stability of dimethenamid in various organic solvents could be demonstrated to be at least 12 months.

B.7.2 Metabolism, distribution and expression of residues

B.7.2.1 Plants

During the initial EU review of the active substance dimethenamid-P the metabolism was investigated for foliar application of the racemic mixture on root and tuber vegetables (sugar beet) and for soil application on cereals (maize) (DAR, 2000, [ASB2010-10566](#)). After Annex I inclusion, one additional metabolism studies with soil application of the racemic mixture on soya beans (pulses and oilseeds) was evaluated by France in 2013. For Annex I renewal two new metabolism studies with foliar respective soil application of pure dimethenamid-P on soya beans (pulses and oilseeds) and maize (cereals) were conducted.

B.7.2.1.1 Maize

Reference:	KCA 6.2.1/1
Report:	Bross, M.; Gläßgen, W. E. (2007): Metabolism of ^{14}C -Dimethenamid (^{14}C -BAS 656 H) in corn. BASF DocID: 2006/1024513, Report 132251 ASB2013-9749
Guidelines:	US EPA OPPTS 860.1300, US EPA OPPTS 860.1000, Commission of the European Communities 7028/VI/95 rev .3
Deviations:	None
GLP:	Yes
Acceptability:	The study is acceptable.

Material and methods

Test Material:	Dimethenamid-P, BAS 656 PH; racemic dimethenamid, BAS 656 H
Lot/Batch #:	824-1030 (thienyl-3- ^{14}C): 96.8 % (radiochemical purity) 824-1029 (thienyl-3- ^{14}C): 96.1 % (radiochemical purity)
Purity:	824-1030: 100 % (chemical purity) 824-1029: 99.7 % (chemical purity)
Specific activity:	824-1030: 8.48 MBq/mg 824-1029: 8.27 MBq/mg
CAS#:	163515-14-8 (dimethenamid-P) 87674-68-8 (racemic dimethenamid)
Test Commodity:	Cereals
Crop:	Maize
Variety:	Benicia
Botanical name:	<i>Zea mays</i>
Crop parts(s):	Forage, forage/husks, grain/cobs, straw, husks, cobs and grain

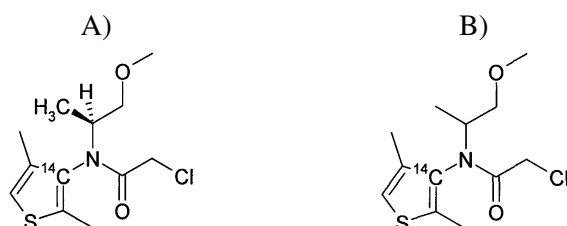


Figure B.7.2-1: Chemical structure and position of radioactive label of A) dimethenamid-P and B) racemic dimethenamid

Experimental conditions:

The study was conducted at the Agricultural Research Center of BASF in Limburgerhof, Germany. The cultivation of the crop took place in plastic containers located in a glass house, climatic chambers (phytotron) and in a glass roofed vegetation hall.

Maize was sown into 10 plastic containers with soil. Four containers were used for dimethenamid-P and six containers were used for racemic dimethenamid. The maintenance of the crop was performed in accordance with normal agricultural practice. In experiment 1, the crop was treated once with racemic ¹⁴C-dimethenamid at a rate of 1300 g as/ha. In experiment 2, the crop was treated once with ¹⁴C-dimethenamid-P at a rate of 720 g as/ha (0.8 N). The application took place at growth stage BBCH 13-15.

For each experiment, a separate application formulation was prepared and applied with an automatic spray track. Unlabelled ¹²C-dimethenamid-P was mixed (1:1, ¹⁴C:¹²C) with the radiolabelled test item ¹⁴C-dimethenamid-P and blank EC formulation BAS 656 08 H. Likewise, unlabelled racemic ¹²C-dimethenamid was mixed with the radiolabelled test item ¹⁴C-dimethenamid (1:3, ¹⁴C:¹²C) and blank EC formulation BAS 656 02 H. The application formulations were analysed by radio-HPLC for identity and purity check.

Sampling and sample storage:

For both experiments, forage samples were taken 30 days after application (30 DAT). Samples of forage/husks and grain/cobs were taken at growth stage BBCH 83-85, 81 DAT for dimethenamid-P/ 80 DAT for racemic dimethenamid). At harvest (BBCH 89, 120 DAT for dimethenamid-P/ 119 DAT for racemic dimethenamid), mature plants were separated into straw, husks, cobs and grain. All samples were stored in a freezer at -18 °C or below. Extracts were stored in a refrigerator or, for longer periods, in a freezer.

In order to demonstrate the storage stability of the residues in plants, the extraction and HPLC investigations were carried out at the beginning of the study. At the end of the metabolism study, a sample of maize forage (dimethenamid-P) was re-extracted. In addition, stored methanol extracts of maize forage (dimethenamid-P) and straw (racemic dimethenamid) were re-analysed by HPLC.

Table B.7.2-1: Design of the plant-uptake part – maize

Position of label	Racemic dimethenamid		Dimethenamid-P	
Intended use rate [g as/ha]	1300		720 (0.8 N)	
Number of applications	1		1	
Interval between applications [days]	-		-	
Sampling material	forage, husks, cobs, grain, straw		forage, husks, cobs, grain, straw	
PHI [days after last application]	forage	30	forage	30
	Forage/husks	81	Forage/husks	80
	Grain/cobs	81	Grain/cobs	80
	Straw	120	Straw	119
	Husks	120	Husks	119
	Cobs	120	Cobs	119
	Grain	120	Grain	119

Extraction of residues and analytical determination:

Samples of the different corn matrices were mixed with liquid nitrogen and homogenised. The total radioactive residues (TRR) were determined by direct combustion analysis of small aliquots of homogenised sample material as well as by summarizing the extractable radioactive residues (ERR) and the residual radioactive residues (RRR) after solvent extraction. For the latter, weighted subsamples of homogenised plant material were extracted three times at ambient temperature with methanol, followed by two extractions with water. Radioactivity in both, the combined methanol extract and aqueous extracted were determined by LSC. The results of the methanol extractions and

the water extraction referred to as ERR. Liquid-liquid partitioning was carried out in order to characterise and purify the methanol extracts. These extracts were partitioned with iso-hexane (3x), dichloromethane (3x) and ethyl acetate (3x). The residue after solvent extraction was dried and subjected to combustion analysis for the determination of the RRR. Moreover, the residue was treated using alkaline and enzymatic hydrolysis, in order to release the residual radioactive residues.

Radioactivity was measured in aliquots using common combustion and LSC techniques. To characterise and purify the metabolites extracted with methanol, liquid/liquid partitioning was carried out and aliquots of the liquid phases were analysed by LSC measurement.

For HPLC analysis, gradient elution on reversed-phase columns was applied. For metabolite profiles and quantitation of metabolites, one HPLC system was used, while further HPLC systems were used for comparison of the metabolite profiles and for confirmation of metabolite identification, for storage stability investigations, for fractionation of some phases obtained after liquid/liquid partition of the methanol extracts of maize forage/husks (racemic dimethenamid), for purification of some collected metabolite fractions, and for analysis of the diluted application solutions.

Identification of the metabolites is mainly based on LC-MS/MS investigations performed with purified fractions from forage (30 DAT) and forage/husks (80 DAT; both racemic dimethenamid). The metabolites in the other samples were assigned by comparison of the retention times with those purified fractions or reference items and of the elution profiles with those of the methanol extracts and the fractions after liquid/liquid partition, from which the fractions had been isolated. If identification was not possible, the peaks were characterised by their retention times. Quantitation is based on HPLC with radio-detection.

Results and discussion

The uptake and the metabolism of dimethenamid (BAS 656 H) in maize was investigated after post-emergence spray application at a rate of 1 x 1300 g as/ha racemic ¹⁴C-dimethenamid, or 1 x 720 g as/ha (0.8 N) ¹⁴C-dimethenamid-P, respectively.

Total radioactive residues (TRRs):

The results for the samples under investigation are summarised in Table B.7.2-2. No major differences were observed when comparing these calculated values with the results of replicate combustion analyses. Minor variations were due to inhomogeneities of the sample material and the limited amounts used for combustion. The TRR values calculated as the sum of ERR and RRR were used as 100 % TRR for all further calculations.

Table B.7.2-2: Total radioactive residues (TRRs) in maize samples following post emergence treatment with ¹⁴C-dimethenamid-P and racemic ¹⁴C-dimethenamid

TRRs in treated maize			
Matrix	DAT*	TRR determined by direct combustion [mg eq/kg]	TRR calculated [mg eq/kg]**
Dimethenamid-P			
Forage	30	0.748	0.718
Forage (re-extraction)	30	0.806	0.692
Forage/husks	81	0.189	0.179
Grain/cobs	81	0.015	0.015
Straw	120	0.538	0.483
Husks	120	0.044	0.042
Cobs	120	0.018	0.018
Grain	120	0.026	0.026
Racemic dimethenamid			
Forage	30	2.125	2.495
Forage/husks	80	0.279	0.278
Grain/cobs	80	0.020	0.019
Straw	119	0.822	0.694
Husks	119	0.075	0.069
Cobs	119	0.029	0.028
Grain	119	0.039	0.039

* DAT = Days after treatment

** TRR was calculated as the sum of ERR and RRR

Extractability:

The extractability of radioactive residues from maize forage, straw and husks after post emergence treatment was high. As given in Table B.7.2-3, 61.5 - 89.2 % of the TRR were extracted for dimethenamid-P and 74.4 - 85.7 % of the TRR for racemic dimethenamid. For cobs and grain a lower extractability was observed ranging from 46.1 - 64.4 % of the TRR for dimethenamid-P and 52.4 - 68.9 % of the TRR for racemic dimethenamid. The major part of the radioactivity was extracted with methanol, releasing five to ten times more radioactivity than the aqueous extraction. The RRR ranged between 10.8 - 53.9 % TRR for dimethenamid-P and 14.3 - 47.6 % TRR for racemic dimethenamid. The subsequent alkaline and enzymatic hydrolysis released an additional 7.1 - 41.1 % TRR for dimethenamid-P and 7.7-38.0 % TRR for dimethenamid.

Table B.7.2-3: Extraction efficiency for residues of ^{14}C -dimethenamid-P and racemic ^{14}C -dimethenamid in maize samples

Matrix	DAA*	TRR calc.**	Distribution of radioactive residues							
			Combined methanol extract		Combined aqueous extract		ERR***		RRR****	
		[mg eq/kg]	[mg eq/kg]	[% TRR]	[mg eq/kg]	[% TRR]	[mg eq/kg]	[% TRR]	[mg eq/kg]	[% TRR]
Dimethenamid-P										
Forage	30	0.718	0.588	81.8	0.053	7.4	0.641	89.2	0.077	10.8
Forage/husks	81	0.179	0.126	70.3	0.011	6.3	0.137	76.6	0.042	23.4
Grain/cobs	81	0.015	0.009	60.5	0.001	4.0	0.010	64.4	0.005	35.6
Straw	120	0.483	0.292	60.4	0.068	14.1	0.360	74.5	0.123	25.5
Husks	120	0.042	0.024	57.0	0.002	4.5	0.026	61.5	0.016	38.5
Cobs	120	0.018	0.010	54.5.	0.001	5.2	0.011	59.6	0.007	40.4
Grain	120	0.026	0.010	36.4	0.003	9.7	0.012	46.1	0.014	53.9
Racemic dimethenamid										
Forage	30	2.495	1.946	78.0	0.192	7.7	2.138	85.7	0.357	14.3
Forage/husks	80	0.278	0.198	71.1	0.018	6.6	0.216	77.6	0.062	22.4
Grain/cobs	80	0.019	0.013	65.0	0.001	3.9	0.013	68.9	0.006	31.1
Straw	119	0.694	0.443	63.9	0.084	12.1	0.527	75.9	0.167	24.1
Husks	119	0.069	0.048	68.9	0.004	5.5	0.052	74.4	0.018	25.6
Cobs	119	0.028	0.016	58.2	0.002	5.8	0.018	63.9	0.010	36.1
Grain	119	0.039	0.017	42.7	0.004	9.7	0.020	52.4	0.019	47.6

* DAA = Days after application

** TRR was calculated as the sum of ERR + RRR

*** ERR = Extractable Radioactive Residue

**** RRR = Residual Radioactive Residue (after solvent extraction)

Partition characteristics:

In order to classify the metabolites into organo-soluble and water-soluble ones and to support metabolite identification and assignment, the combined methanol extracts of forage, forage/husks, grain/cobs, straw, husks, cobs and grain were reduced to the aqueous phase and then partitioned with iso-hexane, dichloromethane and ethyl acetate. The phases obtained were concentrated and examined by means of HPLC and identification of metabolites was achieved by LC-MS/MS analyses of purified fractions from the dichloromethane phase of the methanol extract of maize forage and from three phases obtained after liquid/liquid partition of the methanol extract of forage/husks. Results are presented in Table B.7.2-4.

Table B.7.2-4: Extraction efficiency for residues of ^{14}C -dimethenamid-p and racemic ^{14}C -dimethenamid in maize samples

Matrix	DAA *	Combined methanol extract		Organosoluble						Sum organosoluble		Water soluble		Recovery % *
				Iso-hexane		Dichlormethane		Ethyl acetate						
		mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	
Dimethenamid-P														
Forage	30	0.588	81.8	0.024	3.4	0.125	17.4	0.068	9.5	0.217	30.3	0.394	54.9	104.1
Forage/ husks	81	0.126	70.3	0.004	2.3	0.015	8.2	0.012	6.8	0.031	17.3	0.094	52.8	99.7
Grain/ cobs	81	0.009	60.5	0.001	3.7	0.002	16.2	0.001	6.3	0.004	26.2	0.004	26.7	87.5
Straw	120	0.292	60.4	0.009	1.9	0.051	10.6	0.034	7.0	0.094	19.5	0.207	42.9	103.2
Husks	120	0.024	57.0	0.001	3.2	0.004	9.3	0.003	6.7	0.008	19.2	0.015	36.1	97.0
Cobs	120	0.010	54.5	0.001	3.0	0.001	6.1	0.001	5.7	0.003	14.8	0.007	38.2	97.4
Grain	120	0.010	36.4	0.001	2.7	0.002	7.7	0.001	5.2	0.004	15.6	0.004	15.1	84.5
Racemic dimethenamid														
Forage	30	1.946	78.0	0.016	0.6	0.303	12.1	0.307	12.3	0.626	25.0	1.269	50.9	97.3
Forage/ husks	80	0.198	71.1	0.007	2.5	0.027	9.7	0.022	7.8	0.056	20.0	0.141	50.7	99.5
Grain/ cobs	80	0.013	65.0	0.001	3.9	0.004	20.0	0.001	7.7	0.006	31.6	0.006	30.1	94.8
Straw	119	0.443	63.9	0.015	2.1	0.073	10.5	0.050	7.3	0.138	19.9	0.306	44.2	100.3
Husks	119	0.048	68.9	0.002	3.4	0.008	11.5	0.006	8.1	0.016	23	0.030	43.0	95.7
Cobs	119	0.016	58.2	0.001	3.1	0.002	7.6	0.002	6.2	0.005	16.9	0.011	37.6	93.7
Grain	119	0.017	42.7	0.001	2.3	0.004	9.8	0.002	6.1	0.007	18.2	0.006	16.6	81.5

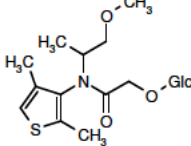
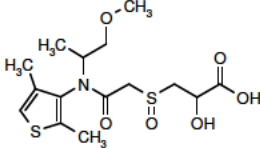
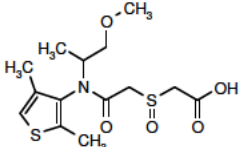
* DAA = Days after application

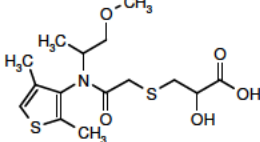
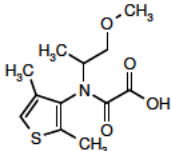
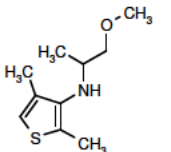
** Recovery calculated as iso-hexane + dichlormethane + ethyl acetate + water soluble [mg/kg] x 100 / combined methanol extract [mg/kg]

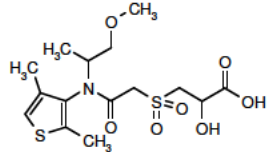
Identification and characterisation of the extractable radioactive residues (ERRs):

The composition of the ERRs in maize samples is specified in Table B.7.2-5.

Table B.7.2-5: Summary of identified components in maize samples after treatment with ¹⁴C-dimethenamid-P (720 g as/ha, 0.8N) and racemic ¹⁴C-dimethenamid (1300 g as/ha)

Metabolite code (Reg. No of reference substance)	Metabolite identity	Matrix	Dimethenmid-P [mg eq/kg] (% TRR) ¹	Racemic Dimethenamid [mg eq/kg] (% TRR) ¹
M40 (one to several isomers)		Forage	0.017 (2.3)	0.098 (3.9%)
		Forage/ husks	0.021 (11.7)	0.028 (10.0)
		Grain/ cobs	<0.0005 (2.7)	<0.0005 (2.3)
		Straw	0.023 (4.7)	0.028 (4.1)
		Husks	0.001 (2.3)	0.003 (4.2)
		Cobs	<0.0005 (1.1)	<0.0005 (1.5)
		Grain	<0.0005 (1.1)	<0.0005 (1.0)
M30		Forage	0.128 (17.9)	0.461 (18.5)
		Forage/ husks	0.008 (4.6)	0.016 (5.6)
		Grain/cobs	<0.0005 (0.6)	<0.0005 (1.1)
		Straw	0.031 (6.4)	0.073 (10.5)
		Husks	0.001 (2.2)	0.001 (1.1)
		Cobs	<0.0005 (0.8)	<0.0005 (0.7)
		Grain	n.d. (n.d.)	<0.0005 (0.2)
M31 (or isomer) (Reg. No.: 360712)		Forage	0.031 (4.4)	0.052 (2.1)
		Forage/ husks	0.005 (2.6)	0.007 (2.6)

Metabolite code (Reg. No of reference substance)	Metabolite identity	Matrix	Dimethenamid-P [mg eq/kg] (% TRR) ¹	Racemic Dimethenamid [mg eq/kg] (% TRR) ¹
		Grain/cobs	<0.0005	<0.0005
			(0.6)	(1.7)
		Straw	0.012	0.018
			(2.5)	(2.7)
		Husks	<0.0005	0.001
			(0.4)	(2.0)
		Cobs	<0.0005	<0.0005
			(0.4)	(1.0)
		Grain	<0.0005	<0.0005
			(0.2)	(0.2)
M26 (Reg. No.: 360716)		Forage	0.077	0.175
			(10.8)	(7.0)
		Forage/ husks	0.002	0.003
			(1.3)	(1.0)
		Grain/cobs	<0.0005	<0.0005
			(0.6)	(1.9)
		Straw	0.006	0.012
			(1.2)	(1.7)
		Husks	n.q.	n.q.
			n.q.	(n.q.)
		Cobs	<0.0005	n.d.
			(0.4)	(n.d.)
		Grain	n.d.	n.d.
			(n.d.)	(n.d.)
M23 (Reg. No.: 360715) and / or M39 (PL1588) and / or C ₁₁ H ₁₃ NO ₃ S (isomer 2, charac- terised) and / or M37 (or isomer)	 M23	Forage	n.d.	One to four of these components were represented by the corresponding HPLC peak and counted as "characterised".
			(n.d.)	
		Forage/ husks	n.d.	0.005
			(n.d.)	(1.9)
	 M39 (PL1588)	Grain/cobs	n.d.	One to four of these components were represented by the corresponding HPLC peak and counted as "characterised"
			(n.d.)	
		Straw	n.d.	n.d.
			(n.d.)	(n.d.)

Metabolite code (Reg. No of reference substance)	Metabolite identity	Matrix	Dimethenamid-P [mg eq/kg] (% TRR) ¹	Racemic Dimethenamid [mg eq/kg] (% TRR) ¹
	 M37 (or isomer)	Husks	n.d.	n.d.
			(n.d.)	(n.d.)
		Cobs	n.d.	n.d.
			(n.d.)	(n.d.)
		Grain	n.d.	n.d.
			(n.d.)	(n.d.)

n.d. Not detected

n.q. Not quantified; the metabolite M26 was detected in the concentrated methanol extract as a shoulder eluting after the peak at approximately 60.225 minutes and no separate quantitation was achieved; The complete peak was therefore counted as "characterised"

¹ Expressed as the sum of the methanol extract and aqueous extract

Proposed metabolic pathway:

Dimethenamid is intensively metabolised in maize: the parent molecule was not detected in extracts (only traces were detectable after liquid/liquid partition), but a large number of different HPLC peaks was observed. The metabolic pathway is comparable for both isomeric test items. The main degradation route proceeds via glutathione conjugation, enzymatic cleavage of the tripeptide and subsequent metabolic reactions on the resulting cysteine conjugate (loss of the amino group of the cysteine moiety and S-oxidation). Various sulfur-containing secondary metabolism products (modified cysteine conjugates) M30, M31 (or isomer), M26 and M37 (or isomer) represented the major part of the extracted radioactive residues. A second metabolic route is hydrolytic/oxidative displacement of the chlorine atom, followed by glycosylation (to form one to several isomers of the metabolite M40) or further transformation (e.g. oxidation, leading to the oxalamide M23, or cleavage of the acetamide to form the amine metabolite M39).

The proposed metabolic pathway of dimethenamid-P in maize is shown in Figure B.7.2-2. The results of this study are in accordance with previous investigations in maize, with metabolism studies in sugar beet and soya bean.

Conclusion

The maize metabolism study was conducted with ¹⁴C-dimethenamid-P and racemic ¹⁴C-dimethenamid after post emergence spray application at a rate of 720 g as/ha (dimethenamid-P) or 1300 g as/ha (racemic dimethenamid), respectively. The application rate was 0.8 N of the rate stated for maize in the list of representative uses. The highest levels of total radioactive residues (TRR) were found in maize forage (sampled 30 days after treatment), straw (harvested 120/119 DAT) and forage/husks (sampled 81/80 DAT). In maize grain/cobs (sampled 81/80 DAT) and in husks, cobs and grain (120/119 DAT), the total radioactive residues were much lower. The extractability of radioactive residues with methanol and water were acceptable with similar results for dimethenamid-P and racemic dimethenamid.

Racemic dimethenamid and dimethenamid-P were both intensively metabolised in maize with no parent detectable in the extracts. However, a large number of different HPLC peaks were observed. The metabolic pathway is sufficiently elucidated and is comparable for both isomeric test items.

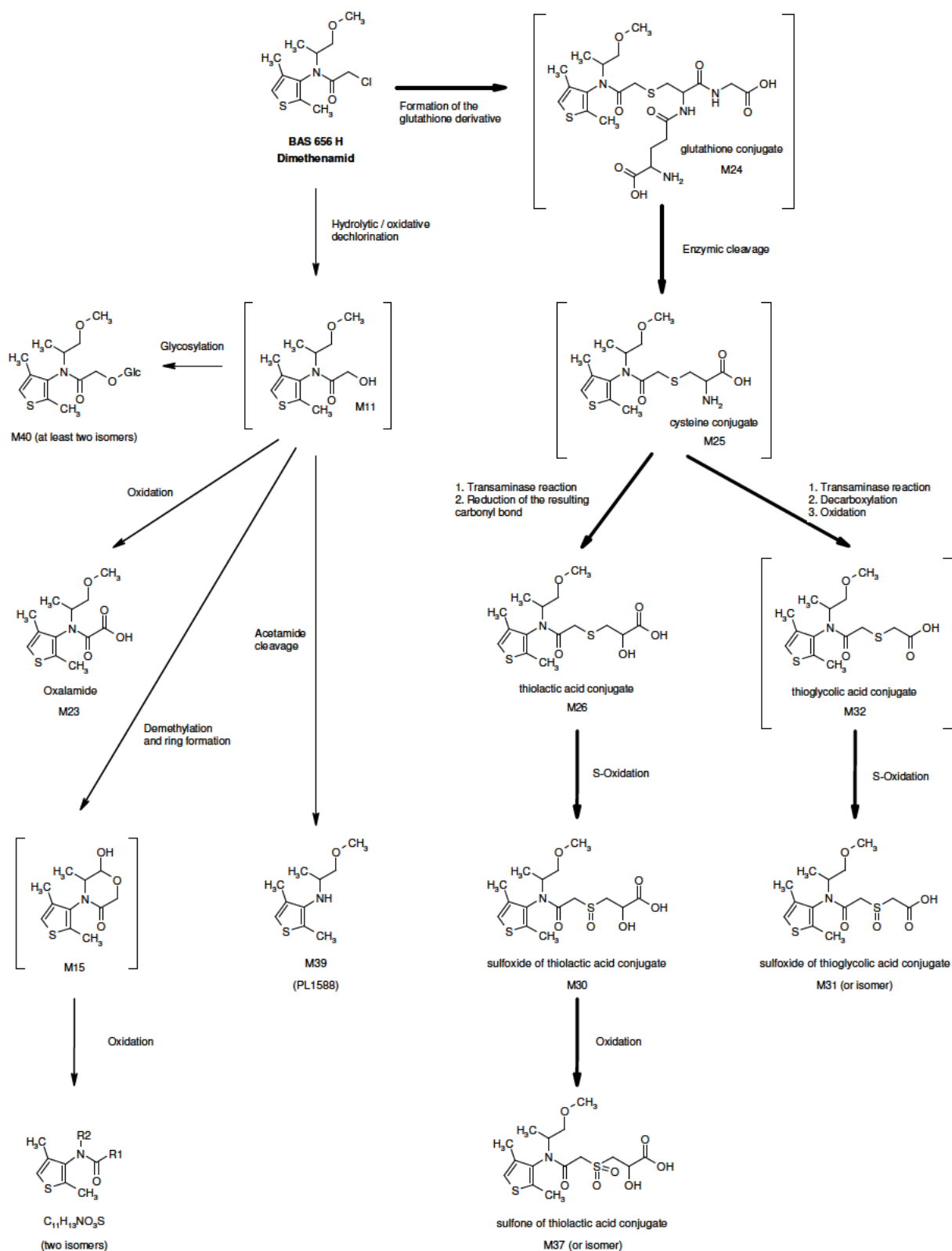


Figure B.7.2-2: Metabolic pathway of ¹⁴C-BAS 656 H in maize after post emergence treatment

Reference:	KCA 6.2.1/2
Report:	Moore, P. A.; Wendt, D. R. (1995): Corn (zea mays) metabolism of ^{14}C -[3-thienyl]-dimethenamid BASF DocID: 95/10129, Project No: 414105, Report No: 31 RIP1999-827
Guidelines:	None stated
Deviations:	N/A
GLP:	Yes
Acceptability:	The study is considered supplementary only.

Material and methods

Test Material:	Dimethenamid, SAN582H
Lot/Batch #:	RS-582-021288 (thienyl-3- ^{14}C): 99 % (radiochemical purity)
Purity:	100 % (chemical purity)
Specific activity:	43.2 mCi/mmol (5.75 MBq/mg)
CAS#:	87674-68-8
Test Commodity:	Cereals
Crop:	Maize
Variety:	Pioneer 3377
Botanical name:	<i>Zea mays</i>
Crop parts(s):	Seedlings, forage, silage, immature grain/cobs, straw, cobs and grain

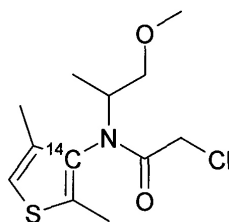


Figure B.7.2-3: Chemical structure and position of radioactive label of racemic dimethenamid

Experimental conditions:

The metabolic fate of dimethenamid in maize plants grown in plant containers outdoors was studied at a field site in Illinois, United States. Radiolabelled active substance was formulated as an emulsifiable concentrate and pipetted uniformly to a loamy soil surface after dilution with distilled water. This application simulated a pre-emergence herbicide use at 1.68 kg R,S-dimethenamid/ha (2 N) and at an exaggerated rate of 4.4 kg R,S-dimethenamid/ha (5.3 N) in containers of 0.25 m². Seeds were planted the day prior to application. Samples were taken at the following intervals after application:

- DAT 19 - field seedlings,
- DAT 50 - forage samples,
- DAT 116 - silage, immature cob and grain samples and
- DAT 130 - straw, mature cob and grain samples.

All samples were stored in a freezer at -18 °C or below.

Samples of maize grown in soil treated at exaggerated rate of 4.4 kg as/ha were only used for analysis in case of very low residues in corresponding samples from the 1.68 kg as/ha treatment. All residue levels given in the text are for the lower treated samples. The residue levels in maize samples after exaggerated soil treatment reveal a linear relationship with the application rate. Also the residue pattern and rates of recovery were shown to be very similar as for those samples from lower use rate.

Extraction of residues and analytical determination:

The samples were extracted with methanol/water, the methanol was evaporated off, and the aqueous fraction was sequentially partitioned with hexane and methylene chloride under neutral and acidic conditions. The remaining aqueous fraction was lyophilised and the resulting solids were dissolved in

methanol and water. The aqueous layer following methanol/water extraction and evaporation of the methanol was also lyophilised directly and the co-extractives removed by counter-current chromatography (single step method). The solids after methanol/water extraction were hydrolysed under acidic and alkaline conditions.

The total radioactivity in all plant parts was determined by combustion and radioassay of $^{14}\text{CO}_2$. The organosoluble extracts from the multiple step procedure were analysed by Thin Layer Chromatography (TLC) and High Pressure Liquid Chromatography (HPLC) systems. The R_f and R_t values were compared to authentic standards. The isolated fractions were further purified and subjected to mass spectrometry (MS) and/or nuclear magnetic resonance (NMR) analysis.

Results and discussion

Total radiocarbon determination after the 1.68 kg as/ha rate treatment yielded residues (as parent equivalent) of 0.3 mg/kg in forage (DAT 50), 0.4 mg/kg in silage (DAT 116) and 0.5 mg/kg fodder (DAT 130). Maize grain contained 0.02 mg/kg equivalent R,S-dimethenamid at DAT 116 and 130. Extractable radiocarbon residues, accompanied by large quantities of co-extractives, were found to be highly polar in nature. Bound radiocarbon in the RACs of the normal dose treatment increased from 12 % (DAT 50 forage) to 51 % (DAT 130 grain). Table B.7.2-6 contains the results on all applied extraction steps:

Table B.7.2-6: Extraction characteristics of dimethenamid in maize plants after soil treatment at 1.68 kg as/ha (2 N) expressed as the % of methanol/water extractable radioactivity and mg/kg dimethenamid equivalents

Crop Part	Forage		Silage		Grain		Fodder		Grain	
DAT	50		116		116		130		130	
TRR [mg eq/kg] ¹	0.307		0.403		0.021		0.504		0.022	
Total recovery [% TRR]	83.9		84.6		78.8		87.5		106.2	
	%	mg/kg	%	mg/kg	%	mg/kg	%	mg/kg	%	mg/kg
MeOH ²	0.21	0.0006	0.10	0.0004	nd ¹⁰		0.47	0.0024	1.11	0.0002
Hexane	2.74	0.0084	0.88	0.0036	nd		1.36	0.0068	4.44	0.0010
CH ₂ Cl ₂ ³	7.82	0.0240	5.16	0.0208	8.19	0.0017	5.99	0.0302	3.72	0.0008
CH ₂ Cl ₂ acid ⁴	16.3	0.0500	10.5	0.0423	5.28	0.0011	7.14	0.0360	5.54	0.0012
Methanol-extr. ⁵	32.2	0.0990	35.5	0.1430	17.6 ⁹	0.0037	10.7	0.0539	22.2 ⁹	0.0049
Water ⁶	9.45	0.0290	3.0	0.0012			13.0	0.0653		
Residual ⁷	2.87	0.0088	4.84	0.0195	1.01	0.0002	5.74	0.0290	17.8	0.0039
Solids ⁸ (unextracted)	12.3	0.0378	24.6	0.0992	46.7	0.0098	43.1	0.2170	51.4	0.0113

¹) Average of four replicates.

²) Radiocarbon in methanol distillate following extraction with methanol/ethanol and evaporation of the methanol.

³) Partitioned in a neutral solution of methylene chloride.

⁴) Partitioned in an acidified solution of methylene chloride.

⁵) Methanol-extractable lyophilisate.

⁶) Water soluble lyophilisate.

⁷) Total radioactivity remaining on walls of Roto-Vap flask and traces of emulsion layers formed during organic / aqueous extraction.

⁸) Values for solids were the sum of the acid hydrolysate values and combustion values of the remaining solid pellet.

⁹) The aqueous fraction was not freeze-dried to determine the methanol soluble radiocarbon because of low radioactivity in these samples (<0.01 mg/kg).

¹⁰) nd = not detected, below the limit of detection of the scintillation counter.

Numerous procedures were employed to maximise radiocarbon extraction, remove co-extractives and identify the specific TLC or HPLC separated bands. When it became apparent that the individual residue species concentrations were so small, large samples (600 g of exaggerated rate) were extracted

to obtain sufficient radiocarbon to work with through the clean-up steps. Successive separations of the various fractions were performed in attempting characterisation by HPLC and/or GC and MS and comparison to known animal and projected synthesised metabolites. The net result of the various procedures was elucidation of more than 30 radiocarbon residues species from the extractable (including hydrolytic) fractions; the largest of which was about 0.03 mg/kg.

Translocation of radiocarbon to grain was minimal. Total radiocarbon in grain was 0.022 mg/kg of which more than 50 % remained unextracted. All extracted fractions in grain were <0.01 mg/kg. Therefore due to this low level in grain, no individual residues could be characterised further. The extracted radiocarbon was distributed between four fractions each containing no more than 0.0049 mg/kg dimethenamid equivalents thus all individual extractable residues were <0.01 mg/kg.

Dimethenamid was rapidly metabolised to several weak acids and other highly polar residues. Consistently in all plant parts there are a large number of individual residue species present in very small amounts. In Table B.7.2-7 the findings are summarised.

Unchanged parent compound was not present in any commodity from the Day 50 forage sample through mature grain, neither at the 1.68 kg as/ha application rate nor at the 4.4 kg as/ha exaggerated rate.

The metabolic profiles in all vegetative raw agricultural commodities (RACs) were qualitatively similar and six metabolites were identified in various extracts, these were: M32, the thioglycolic acid conjugate; M26, the thiolactic acid conjugate; M23, the oxalamide; M31 the sulfoxide of the thioglucolic acid conjugate; M30, the sulfoxide of the thiolactic acid conjugate and M27 the sulfonate. M27 was the most abundant metabolite found in the vegetative RACs. Its highest level was in silage at 116 days at 0.03 mg/kg or 7.38 % of TRR. It was extracted in the methanol soluble freeze-dried aqueous fraction.

In the organic extracts, combined levels of M23, oxalamide and M26, thiolactic acid were highest at the forage stage at 0.011 mg/kg dimethenamid equivalents. In seedlings (not an RAC) the extract in the TLC band(s) with R_f similar to M32 thioglycolic acid was found to contain M11 and/or M9 upon further TLC characterisation. The same band(s) in the fodder extract represented 0.029 mg/kg or 5.6 % total radiocarbon in fodder. The highest level of M30 occurred at the silage stage at 0.012 mg/kg or 2.9 % radiocarbon.

Table B.7.2-7: Characteristics and identification of R/S-dimethenamid residues in maize plants after soil treatment at 1.68 kg as/ha (2 N) (expressed as the % of TRR and mg/kg dimethenamid equivalent)

Crop Part	Forage		Silage		Fodder	
DAT	50		116		130	
TRR [mg eq/kg]	0.307		0.403		0.504	
	%	mg/kg	%	mg/kg	%	mg/kg
Parent	nd ¹²		nd		nd	
Unknown in methylene extract	1.40 ²	0.0043	1.49 ⁵	0.0060	-	
Sulfoxide of thiolactic acid (M31)	1.60	0.0051	3.70	0.0149	2.02	0.0102
Unknown in methylene extract	2.37 ³	0.0073	-		-	
Sulfoxide of thioglycolic acid (M30)	1.66	0.0049	2.90 ⁶	0.0117	0.67	0.0034
Oxalamide (M23)	3.58	0.0110	0.57 ^{7,9} -3.57	0.0023-0.0144	1.43 ¹⁰	0.0072
Thiolactic acid (M26)	2.28	0.0070	1.19 ^{7,9} -4.19	0.0048-0.0169		
TGA (M32 plus ¹)	3.71	0.0114	0.60 ^{7,9} -3.60	0.0024-0.0145	5.62	0.0283
Unknown in methylene extract	6.84 ⁴	0.0210	2.28 ^{7,8,9} -5.28	0.0092-0.0213	-	
Sulphonate (M27 ¹¹)	6.06	0.186	7.38	0.0297	2.5	0.0126

¹) Thioglycolic acid M32 plus M11 plus other unknowns which additional TLC analysis of soil demonstrated this band to be a multi-component as found also in maize seedlings (non-GLP study).

²) TLC* Rf values of 0 to 0.15 and 0 to 0.19 with neutral and acidic methylene chloride, respectively.

³) TLC* Rf values of 0.24 to 0.30 and 0.29 to 0.33 with neutral and acidic methylene chloride, respectively.

⁴) TLC* Rf values of 0.70 to 0.78 and 0.68 to 0.83 with neutral and acidic methylene chloride, respectively.

⁵) TLC* Rf values of 0.0 to 0.18 and 0.0 to 0.15 with neutral and acidic methylene chloride, respectively.

* Solvent System 1 ethyl acetate/toluene/formic acid/water 87:3:5:5.

⁶) A combination of the components of two TLC bands at Rf values of 0.286 to 0.390 and 0.390 to 0.48 with neutral methylene chloride and 0.28 to 0.38 and 0.38 to 0.47 with acidic methylene chloride.

⁷) additional TLC analysis of soil demonstrated the band resulting from neutral methylene chloride extraction to be a multicomponent one as found also in maize seedlings (non-GLP study).

⁸) TLC Rf values of 0.48 to 0.92 and 0.77 to 0.85 with neutral and acidic methylene chloride, respectively.

⁹) The radiocarbon on the TLC band resulting from neutral methylene chloride extraction was very diffuse.

¹⁰) Oxalamide and thiolactic acid.

¹¹) TLC with solvent system 2:1-butanol/glacial acetic acid/water 60:15:25.

¹²) nd = not detected, limit of detection ranged from 0.001 to 0.005 mg/kg.

Attempts with various model metabolites (conjugates) were made to quantify the potential residues of dimethenamid in maize by use of a common moiety method. Among others, the following tests were conducted:

1. Base hydrolysis with 1 N KOH at 90 °C/1 h
2. Acid hydrolysis with 1 N HCl at 90 °C/1 h
3. Acid hydrolysis with 6 N HCl at 85 °C/16 h
4. Desulfuration with Raney Nickel.

None of the methods proved to give a single degradation product with dimethenamid and various conjugates nor did they give satisfactory and consistent recoveries.

Proposed metabolic pathway:

Based on the identified metabolites the proposed metabolic pathway involves conjugation of dimethenamid with glutathion and hydrolysis of the glutathion conjugate to the cysteine conjugate, both are considered transient intermediates undergoing rapid oxidation, deamination and/or decarboxylation forming a high number of products as shown in Figure B.7.2-2.

Conclusion

Dimethenamid applied to soil at 1.68 kg as/ha (2 N) was rapidly metabolised in maize leading to a large number of mostly polar metabolites, the largest of which is less than 0.05 mg/kg and less than 10 % of total radioactive residue. Unextracted radiocarbon increased with time and indicated incorporation of residues into the plant matrix.

Five metabolites of this pathway were identified in extracts of the RACs:

M32 - thioglycolic acid conjugate,

M26 - thiolactic acid conjugate,

M31 - sulfoxide of thioglycolic acid,

M30 - sulfoxide of the thiolactic acid conjugate,

M27 - sulfonate.

In addition, oxalamide (M23) was also present via either uptake from the soil, or through a secondary mechanism involving cytochrome P-450. These metabolites have all been identified in the rat, with many also found to be present in the goat and/or hen, demonstrating that animal metabolism proceeds through similar pathways. No parent dimethenamid was detected in any RACs at any sampling interval.

The study confirms the results of the more recent study by Bross and Gläßgen (2007, [ASB2013-9749](#)). However, due to limitations with regards to metabolite identification, the study can only be considered as supplementary.

B.7.2.1.2 Soya beans

Reference:	KCA 6.2.1/3
Report:	Schweda, Z.; Lutz, T.; Deppermann, N. (2012): Metabolism of ¹⁴ C-BAS 656-P H (¹⁴ C-labelled Dimethenamid-P) in soybean BASF DocID: 2012/1144379, Study code 380191 ASB2014-8341
Guidelines:	US EPA OPPTS 860.1300, US EPA OPPTS 860.1000, Commission of the European Communities 7028/VI/95 rev. 3
Deviations:	None
GLP:	Yes
Acceptability:	The study is acceptable.
Reference:	KCA 6.2.1/4
Report:	Schweda, Z.(2014): Amendment No. 1 to report: Metabolism of ¹⁴ C-BAS 656-P H (¹⁴ C-labelled dimethenamid-P) in soybean BASF DocID: 2014/1036947, Study code 380191 ASB2014-10169
Guidelines:	US EPA OPPTS 860.1300, US EPA OPPTS 860.1000, Commission of the European Communities 7028/VI/95 rev. 3
Deviations:	None
GLP:	Yes
Acceptability:	The study is acceptable.

Material and methods

Test Material:	Dimethenamid-p, BAS 656 PH
Lot/Batch #:	824-5070 (thienyl-2(5)- ¹⁴ C): 98.9 % (radiochemical purity) L74-174 (unlabelled compound)
Purity:	824-5070: 87.4 % (chemical purity) L74-174: 99.4 % (chemical purity)
Specific activity:	8.78 MBq/mg
CAS#:	87674-68-8
Test Commodity:	Oilseed
Crop:	Soya bean
Type	Oilseed
Variety:	Pioneer 9091
Botanical name:	<i>Glycine max.</i> L.
Crop parts:	Leaf, bean, hull and rest of plant

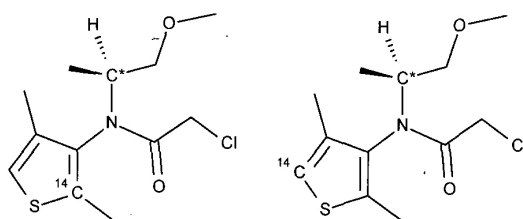


Figure B.7.2-4: Chemical structure and position of radioactive label of dimethenamid-P

Experimental conditions:

The study was carried out at the Agricultural Research Centre of BASF in Limburgerhof, Germany. The plant uptake part of the study was conducted in a phytotron, vegetation hall, or greenhouse.

Soya bean was sowed into eight containers filled with soil. According to the study protocol, a single application was carried out on the soil (directly after sowing) with ¹⁴C-dimethenamid-P at a nominal rate of 1008 g as/ha (1.2 N). The plants were harvested at 119 DAT.

For the preparation of the application formulation, ¹⁴C-dimethenamid-P (dissolved in acetonitrile) and unlabelled dimethenamid-P were mixed to obtain a ratio of approximately 40:60. Thereafter, the test item was dissolved in a mixture of the blank formulation BAS 656 AB H and water. The purity of the application solution was confirmed using HPLC and the isotopic pattern was determined by HPLC-MS analysis.

Sampling and sample storage:

At growth stage BBCH 89, the leaves were picked up from the plants. The beans were picked from the stipes and separated from the hulls. The stipes were cut and minced. The samples were weighed and frozen.

Extraction of residues and analytical determination:

Homogenisation/solvent extraction: All samples (leaf, seed, hull and rest of plant) were homogenised. All samples were extracted three times with methanol and two times with water. After each extraction step solid material was separated from the extract by centrifugation and filtration. The filtered supernatants (methanol extracts and water extracts) were pooled and adjusted to a defined volume. The residue after solvent extraction was dried in a lyophilisation device, homogenised and radio assayed.

Solubilisation of the RRR: The residue after solvent extraction was extracted two times with 1 % ammonia. The extraction procedure was similar to the solvent extraction. The residue after ammonium solubilisation was resuspended in a sodium acetate buffer (pH 5), Macerozyme R-10 and Cellulase Onozuka R-10 were added in a ratio of 1:1 and the mixture was incubated on a shaker at 37 °C for 48 h. After centrifugation, the resulting residue was resuspended in a phosphate buffer (pH 5.95), α-amylase, β-amylase and amyloglucosidase were added and the mixture was incubated on a shaker at 37 °C over the weekend. After centrifugation, the resulting residue was dried in a lyophilisation

device. The dried residue was resuspended in 10 % NaOH and was heated for 3 h under reflux. Thereafter, the sample was filtered and the residue was washed with 10 % NaOH at 80 °C and then with water at 25 °C. The extract and the washing solutions were pooled. The residue after NaOH solubilisation was dried in an oven at 50 °C. The NaOH solubilisate was mixed with concentrated HCl until pH 1 was reached. The mixture was stored overnight in a refrigerator and lignin was separated by centrifugation.

Analytical determination: Homogenised solid plant samples were weighed and combusted by means of an automatic sample oxidiser. The limit of quantitation in mg/kg was calculated from the twofold background radioactivity level (dpm/g matrix) divided by the corresponding specific radioactivity. For the quantitation of radioactivity in liquid samples a liquid scintillation counter was used.

Gradient elution on reversed-phase columns was applied, using different HPLC systems for metabolite profiles, for confirmation of metabolite identification and for fractionation of the methanol extracts. The identification of the metabolites is based on the LC-MS/MS analysis of soya bean leaf methanol extract. The peak assignment for leaf methanol extract was based on co-chromatography experiments with isolated MS fractions. The peak assignment for all other extracts was based on comparison of retention times with those of the leaf methanol extract. In order to identify the components of an early eluting peak group, the polar fraction was isolated from leaf methanol extract. Thereafter, co-chromatography with the reference items glucose, fructose and sucrose were carried out, using two saccharide specific HPLC methods.

Results and discussion

Total Radioactive Residue (TRR):

In the present study, the TRR was calculated by summarising the extractable radioactive residue (ERR) and the residual radioactive residue (RRR) after solvent extraction. The measured TRR of all four matrices showed no major differences to the calculated TRR values.

Table B.7.2-8: Total radioactive residues (TRRs) in soya bean samples following the application of BAS 656 PH

TRRs in treated soya bean			
Matrix	DAT*	TRR determined by direct combustion [mg eq/kg]	TRR calculated [mg eq/kg]**
Leaf	119	2.816	2.595
Seed	119	0.648	0.693
Hull	119	0.719	0.821
Rest of plant	119	0.666	0.629

* DAT = Days after treatment

** TRR was calculated as the sum of ERR and RRR

Extractability:

The extractability of the soya bean matrices with methanol and water is summarised in Table B.7.2-9.

Table B.7.2-9: Extraction efficiency for residues of dimethenamid-P in soya bean samples

Matrix	DAA*	TRR calc.**	Distribution of radioactive residues							
			Combined methanol extract		Combined water extract		ERR***		RRR****	
		[mg eq/kg]	[mg eq/kg]	[% TRR]	[mg eq/kg]	[% TRR]	[mg eq/kg]	[% TRR]	[mg eq/kg]	[% TRR]
Leaf	119	2.595	1.215	46.8	0.607	23.4	1.822	70.2	0.773	29.8
Seed	119	0.693	0.110	15.8	0.216	31.2	0.326	47.1	0.367	52.9
Hull	119	0.821	0.107	13.0	0.103	12.6	0.210	25.6	0.611	74.4
Rest of plant	119	0.629	0.188	29.9	0.051	8.0	0.239	38.0	0.390	62.0

* DAA = Days after application

** TRR was calculated as the sum of ERR + RRR

*** ERR = Extracted radioactive residue

**** RRR = Residual radioactive residue (after solvent extraction)

Identification, characterisation and quantification of radioactive residues in leaf:

In the analysis of the leaf methanol extract with HPLC, metabolite M27 and was the most abundant component (0.276 mg eq/kg or 10.7 % TRR). The polar fraction accounted for 0.051 mg eq/kg or 2.0 % TRR). All other identified components (M81, M50, M40, M14 isomer A/M14 isomer B/M30/others, M31, M23/M51, M26/M11) were present at significantly lower amounts and accounted from 0.7 % TRR (metabolite M50) up to 3.6 % TRR (M81). HPLC analysis of the leaf water extract revealed the polar fraction as the most abundant component (0.066 mg eq/kg or 2.5 % TRR). Other identified components were M14 isomer A/M14 isomer B/M30/others, M27, M23/M51 and M26/M11, which ranged from 0.3 % TRR (M26/M11) up to 2.0 % TRR (M14 isomer A/M14 isomer B/M30/others). In the solvent extracts 0.806 mg/kg or 31.1 % TRR were identified and 1.016 mg/kg or 39.2 % TRR were characterised by HPLC (each peak below or equal to 1.7 % TRR). Taken together a total of 1.822 mg/kg or 70.2 % TRR was identified and characterised in the ERR of soya bean leaf.

In the ammonia solubilisate the polar fraction accounted for 0.057 mg eq/kg and 2.2 % TRR. Other identified metabolites were M14 isomer A/M14 isomer B/M30/others, M23/M51 and M26/M11, which were significantly less abundant and accounted for up to 0.7 % TRR (M14 isomer A/M14 isomer B/M30/others). The polar fraction found during the analysis of the macerozyme and amylase solubilisate with another HPLC method accounted for 0.111 mg/kg or 4.3 % TRR (macerozyme solubilisate) and 0.057 mg/kg or 2.2 % TRR (amylase solubilisate). The residue after amylase solubilisation was further characterised by NaOH solubilisation and lignin precipitation. In the solubilisates 0.254 mg/kg or 9.8 % TRR were identified and 0.149 mg/kg or 5.7 % TRR were characterised by HPLC (each peak below or equal to 0.5 % TRR). Taken together, a total of 0.618 mg/kg or 23.8 % TRR were identified and characterised in the RRR of soya bean leaf.

A summary of identified and characterised residues is compiled in Table B.7.2-10. In soya bean leaf, amounts of 2.440 mg/kg or 94.0 % TRR were identified and characterised.

Identification, characterisation and quantification of radioactive residues in seeds:

In the protease solubilisate of the methanol extract the polar fraction accounted for 0.046 mg eq/kg or 6.7 % TRR. In the analysis of the corresponding supernatant (supernatant of methanol extract) with HPLC each peak was below or equal to 0.4 % TRR. The cyclohexane phase of the methanol extract and the supernatant of the water extract were also analysed with HPLC. Similarly to the methanol extract, analysis of the protease solubilisate of the water extract with HPLC resulted predominantly in a polar fraction (0.154 mg/kg or 22.3 % TRR). A significant part of the radioactive residues in the methanol and water extract was precipitated with acetone and solubilised by protease. Therefore, the corresponding radioactivity, which was mainly assigned as polar fraction, was most probably incorporated into proteins. In the solvent extracts 0.201 mg/kg or 29.0 % TRR were identified and 0.048 mg/kg or 7.0 % TRR were characterised by HPLC (each peak below or equal to 1.2 % TRR). Taken together, a total of 0.293 mg/kg or 42.4 % TRR was identified and characterised in the ERR of soya bean.

HPLC analysis of the solubilisate of the RRR (supernatant of ammonia solubilisate, macerozyme solubilisate and amylase solubilisate) with HPLC resulted predominantly in early eluting peaks, which were assigned as polar fraction. The polar fraction of the supernatant of the ammonia solubilisate accounted for 0.021 mg/kg or 3.0 % TRR, the polar fraction of the macerozyme solubilisate for 0.112 mg/kg or 16.2 % TRR and the polar fraction of the amylase solubilisate for 0.021 mg/kg or 3.1 % TRR. The residue after amylase solubilisation was further characterised by NaOH solubilisation and lignin precipitation. In the solubilisates 0.154 mg/kg or 22.3 % TRR were identified and 0.007 mg/kg or 1.1 % TRR were characterised by HPLC (each peak below or equal to 0.7 % TRR). The residues in the pellet of the ammonia solubilisate (0.109 mg/kg or 15.8 % TRR) were most likely proteins, since they were precipitated by acetone, similar to the methanol and water extract. Taken together, 0.343 mg/kg or 49.5 % TRR were identified and characterised in the RRR of soya bean. A summary of identified and characterised residues is compiled in Table B.7.2-10. In soya bean, amounts of 0.637 mg/kg or 91.9 % TRR were identified and characterised.

Identification, characterisation and quantification of radioactive residues in hull:

In the HPLC analysis of the hull methanol extract, the polar fraction accounted for 0.022 mg/kg or 2.6 % TRR. The remaining identified components (M14 isomer A/M14 isomer B/M30/others, M31, M23/M51 and M27) were less abundant and accounted from 0.7 % TRR (M14 isomer A/M14 isomer B/M30/others) up to 1.1 % TRR (M23/M51 and M27). In the analysis of the water extract with HPLC, the polar fraction represented the main component (0.036 mg eq/kg or 4.4 % TRR). The two other identified peaks (peak group) M14 isomer A/M14 isomer B/M30/others and M23/M51 were significantly less abundant and accounted for 0.5 % TRR and 0.8 % TRR, respectively. In the solvent extracts 0.099 mg eq/kg or 12.0 % TRR were identified and 0.112 mg eq/kg or 13.6 % TRR were characterised by HPLC (each peak below or equal to 0.4 % TRR). Taken together, a total of 0.210 mg eq/kg or 25.6 % TRR was identified and characterised in the ERR of soya bean hull.

The polar fractions of the ammonia solubilisate, the macerozyme solubilisate and the amylase solubilisate accounted for 0.085 mg eq/kg or 10.4 % TRR, 0.230 mg/kg or 28.0 % TRR and 0.051 mg/kg or 6.3 % TRR, respectively. The residue after amylase solubilisation was further characterised by NaOH solubilisation and lignin precipitation. In the solubilisates 0.367 mg/kg or 44.6 % TRR were identified. Taken together, 0.486 mg eq/kg or 59.1 % TRR were identified and characterised in the RRR of soya bean hull.

A summary of identified and characterised residues is compiled in Table B.7.2-10. In soya bean hull, amounts of 0.696 mg/kg or 84.7 % TRR were identified and characterised.

Identification, characterisation and quantification of radioactive residues in rest of plant:

In the HPLC analysis of the rest of plant methanol extract the polar fraction accounted for 0.017 mg eq/kg or 2.7 % TRR. The remaining identified components (M81, M14 isomer A/M14 isomer B/M30/others, M31, M27, M23/M51) were less abundant and accounted from 0.7 % TRR (M23/M51) up to 2.6 % TRR (M27). In the analysis of the water extract with HPLC, the polar fraction represented the main component (0.012 mg eq/kg or 2.0 % TRR). In the solvent extracts 0.078 mg eq/kg or 12.5 % TRR were identified and 0.160 mg eq/kg or 25.5 % TRR were characterised by HPLC (each peak below or equal to 1.2 % TRR). Taken together, a total of 0.239 mg eq/kg or 38.0 % TRR was identified and characterised in the ERR of soya bean rest of plant.

The polar fractions of the ammonia solubilisate, macerozyme solubilisate and amylase solubilisate accounted for 0.046 mg eq/kg or 7.3 % TRR, 0.089 mg eq/kg or 14.2 % TRR and 0.020 mg eq/kg or 3.2 % TRR, respectively. The residue after amylase solubilisation was further characterised by NaOH solubilisation and lignin precipitation. In the solubilisates 0.156 mg/kg or 24.7 % TRR were identified. Taken together, 0.244 mg eq/kg or 38.8 % TRR were identified and characterised in the RRR of soya bean rest of plant.

A summary of identified and characterised residues is compiled in Table B.7.2-10. In soya bean rest of plant, amounts of 0.482 mg eq/kg or 76.7 % TRR were identified and characterised.

Table B.7.2-10: Summary of identified components in soya bean matrices

Metabolite	Leaf		Seed		Hull		Rest of plant	
	[mg eq/kg]	[% TRR]	[mg eq/kg]	[% TRR]	[mg eq/kg]	[% TRR]	[mg eq/kg]	[% TRR]
Polar fraction	0.342	13.2	0.355	51.2	0.425	51.7	0.185	29.4
M81	0.093	3.6	n.r.		n.r.		0.011	1.7
M50	0.019	0.7	n.r.		n.r.		n.r.	
M40	0.034	1.3	n.r.		n.r.		n.r.	
M14 isomer A/ M14 isomer B/ M30/others	0.135	5.2	n.r.		0.010	1.2	0.007	1.2
M31	0.025	1.0	n.r.		0.006	0.8	0.010	1.6
M27	0.321	12.4	n.r.		0.009	1.1	0.016	2.6
M23/M51	0.053	2.1	n.r.		0.015	1.9	0.005	0.7
M26/M11	0.038	1.5	n.r.		n.r.		n.r.	
Total identified	1.060	40.9	0.355	51.2	0.465	56.6	0.234	37.2
Total characterised	1.380	53.2	0.282	40.7	0.231	28.1	0.248	39.5
Total identified and characterised	2.440	94.0	0.637	91.9	0.696	84.7	0.482	76.7
Final residue	0.135	5.2	<0.001	<0.1	0.121	14.7	0.151	24.0
Grand total	2.576	99.3	0.637	91.9	0.816	99.4	0.633	100.8

n.r. Not reported

Metabolic pathway

The proposed metabolic pathway of dimethenamid-P in soya bean is shown in Figure B.7.2-5. A summary of the detected metabolites is given in Table B.7.2-10. The initial step of most metabolites of dimethenamid-P is a presumed substitution of the chlorine atom by glutathione, which leads to the intermediate M24, which was proposed in the metabolic pathway in maize (DocID 2006/1024513). Cleavage of the amino acids glutamic acid and glycine from the glutathione side-chain of M24, followed by deamination and reduction, leads to the 3-mercapto-lactic acid derivative M26. Oxidation of the thioether group of M26 (S-oxidation) yields the sulfoxide M30. Decarboxylation of M30 followed by oxidation leads to metabolite M31. Oxidation of the sulfoxide group of M31 results in the sulfone M51. Further decarboxylation and oxidation (C- and S-oxidation) steps on M51 yield the sulfonic acid derivative M27. Alternatively, after the first decarboxylation step, the ether group is cleaved, which results in the isomers M14 isomer A and B. Glycosylation of M14 isomer A or M14 isomer B yields metabolite M81. Alternatively to the glutathione conjugation and thioether cleavage, hydrolytic dechlorination of dimethenamid-P yields the hydroxylated derivative M11. Oxidation of the hydroxyl group to a carboxyl residue leads to metabolite M23. Cleavage of the ether group of M23 results in metabolite M50. Metabolite M40 is the result of glycosylation of M11. The radioactivity of dimethenamid-P was also partially incorporated into sugar molecules. Since incorporation of dimethenamid-P moieties into sugar molecules requires the transformation of the degradation products into suitable compounds for the biosynthesis of carbohydrates, it is likely that C1 and/or C2 entities of dimethenamid-P enter anabolic biosynthetic pathways. Considerable portions of non-extractable residues were solubilised with macerozyme/cellulose and amylases/amyloglucosidase, and thereafter, identified by HPLC as polar fraction. Release of ¹⁴C-labelled polar components upon treatment of the residues after solvent extraction with polysaccharide-cleaving enzymes most probably reflects an incorporation of radioactivity into polysaccharides. Likewise, detection of polar components after protease treatment of the solvents extracts of bean reflects an incorporation of radioactivity into proteins.

Conclusion

The present study describes the metabolism of dimethenamid-P (BAS 656 PH) in soya beans after soil application of 1008 g as/ha (directly after sowing). The applied rate was 1.2 N of the rate stated for soya beans in the list of representative uses. Soya bean plants were harvested 119 days after treatment/sowing and were separated into leaf, seeds, hull and rest of plant.

The highest total radioactive residue (TRR) was found in soya bean leaf accounting for 2.595 mg eq/kg. TRR in the other matrices (seeds, hull and rest of plant) was approximately 3-4 times lower. For seeds the TRR accounted for 0.693 mg eq/kg, for hull 0.821 mg eq/kg and for rest of plant 0.629 mg eq/kg. The extractability of leaves with methanol and water was high and accounted for 70.2 % of the TRR. The extractability for the remaining matrices was lower and accounted for 47.1 % (seeds), 25.6 % (hull) and 38.0 % (rest of plant) of the TRR. The residue after solvent extraction of all matrices was further solubilised, whereby 23.9 % (leaf) to 59.0 % (hull) of the TRR were additionally released.

HPLC-MS investigations of isolated fractions from the soya bean leaf methanol extract led to the identification of metabolites M11, M40, M23, M50, M26, M30, M31, M51, M14 isomer A, M14 isomer B, M81 and M27. Metabolites M11, M40, M23 and M50 are dechlorinated derivatives of the parent compound. The remaining metabolites result from substitution of the chlorine atom with glutathione and subsequent degradation of the glutathione moiety. The isolated MS fractions were used for co-chromatography experiments for peak assignment. Additionally, sugars were identified by co-chromatography experiments with a polar fraction from soya bean leaf methanol extract, whereby two saccharide-specific HPLC methods were applied.

In all matrices the polar fraction was the most abundant (leaf, hull and rest of plant) and accounted from 13.2 % (leaf) up to 51.7 % (hull) of the TRR. The polar fraction was partially composed of sugars. Generally, the major part of the radioactivity of the polar fraction was incorporated into polysaccharides and/or proteins, because polar components were released upon treatment with polysaccharide cleaving enzymes (RRR of all matrices) and/or protease (ERR of bean). In soya bean leaves metabolite M27, a sulfonic acid derivative of the parent compound, was the second most abundant component and accounted for 12.4 % TRR. Other identified metabolites in soya bean leaf, hull and rest of plant were significantly less abundant and were present at concentrations from 0.7 % TRR (M23/M51 in rest of plant) up to 5.2 % TRR (M14 isomer A/M14 isomer B/M30/others in leaf).

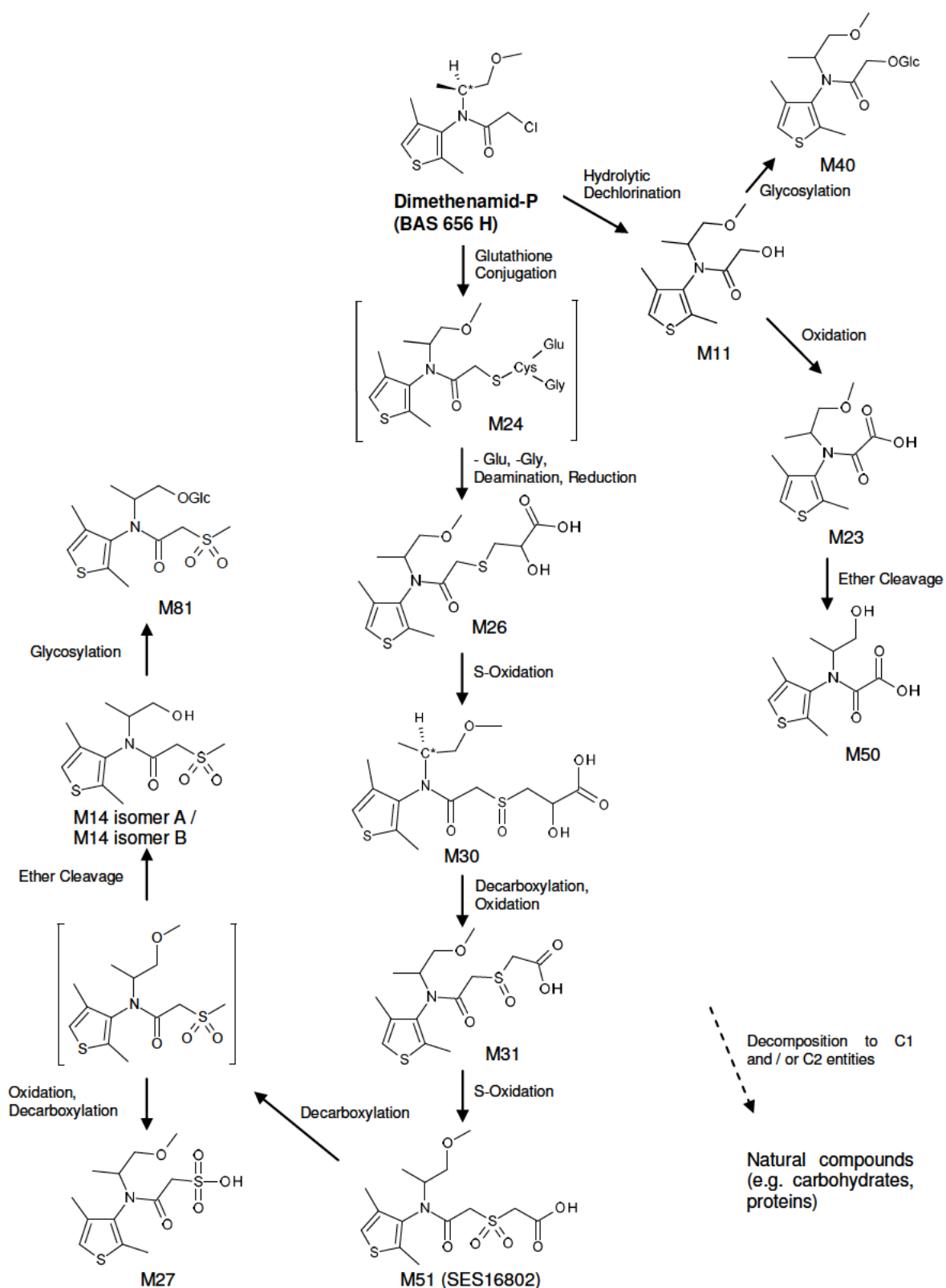


Figure B.7.2-5: Proposed metabolic pathway of dimethenamid-P in soya bean

Reference:	KCA 6.2.1/5
Report:	Atallah, Y. H.; Moore, P. A.; Bade, T. R. (1991): Uptake, translocation and metabolism of the herbicide SAN-582H in soybean BASF DocID: 91/11879, Project No: 414105, Report No: 19 RIP2006-564
Guidelines:	None stated
Deviations:	N/A
GLP:	Yes
Acceptability:	The study is considered supplementary only.

Material and methods

Test Material:	Dimethenamid (SAN582H)
Lot/Batch #:	RS-582-021288 (thienyl-3- ¹⁴ C): 99 % (radiochemical purity)
Purity:	98 % (chemical purity)
Specific activity:	43.2 mCi/mmol (5.75 MBq/mg)
CAS#:	87674-68-8
Test Commodity:	Oilseed
Crop:	Soya bean
Variety:	Pioneer 9271
Botanical name:	<i>Glycine max.</i> L.
Crop parts(s):	Forage, hay, immature bean, mature bean, straw

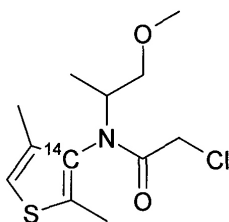


Figure B.7.2-6: Chemical structure and position of radioactive label of racemic dimethenamid

Experimental conditions:

The metabolic fate of dimethenamid in soya beans grown in plant containers was studied. Radiolabelled active substance was formulated as an emulsifiable concentrate and diluted with distilled water before making a pre-emergence application to the soil. Application rates were at 1.68 kg R,S-dimethenamid/ha (2 N) and at an exaggerated rate of 3.3 kg R,S-dimethenamid/ha (4 N) in containers of 0.25 m². Seeds were planted the day prior to application. Samples were taken at the following intervals after application:

- DAT 49 - forage
- DAT 100 - hay and immature bean
- DAT 118 - mature and bean

All samples were stored in a freezer at -20 °C.

Extraction of residues and analytical determination:

The samples were sequentially extracted with hexane (soya beans only), methylene chloride, acetone, methanol, then with water. The aqueous extract was acidified to 1 N HCl, refluxed for 1 h at 90 °C then partitioned with ethyl acetate. Solids, after sequential extraction, were hydrolysed under acidic conditions (1 N HCl, 90 °C, 1 h), followed by alkaline conditions (10 % NaOH, 130 °C, 4 h).

The total radioactivity in all plant parts was determined by combustion and radioassay of ¹⁴CO₂. The organosoluble extracts from the multiple step procedure were analysed by Thin Layer Chromatography (TLC) and High Pressure Liquid Chromatography (HPLC) systems. The R_f and R_t values were compared to authentic standards. The isolated metabolites were further purified and subjected to mass spectrometry (MS) and/or nuclear magnetic resonance (NMR) analysis.

Results and discussion

Extractability:

Dimethenamid is rapidly and extensively metabolised by field grown soya beans to an array of metabolites. Total radiocarbon determination after the 1.68 kg as/ha rate treatment yielded residues (as parent equivalent) of 2.8 mg eq/kg in forage (DAT 49), 2.62 mg eq/kg in hay (DAT 100), 0.123 mg eq/kg in immature bean (DAT 100), 0.412 mg eq/kg in beans (DAT 118) and 1.49 mg eq/kg in straw (DAT 118).

Extractable radiocarbon residues, accompanied by large quantities of co-extractives, were found to be highly polar in nature. Bound radiocarbon in the RACs of the normal dose treatment increased from 5.5 % (DAT 49 forage) to 16 % (DAT 118 straw). Table B.7.2-11 contains the results on all applied extraction steps:

Table B.7.2-11: Extraction characteristics of dimethenamid in soya bean plants after soil treatment at 1.68 kg as/ha (2N) expressed as the % of extractable radioactivity and mg/kg dimethenamid equivalents¹

Crop Part		Forage		Hay		Immature bean		Straw ⁴		Bean	
DAT		49		100		100		118		118	
TRR [mg eq/kg]		2.80		2.62		0.123		1.49		0.412	
Total recovery [% TRR]		87.8		94.4		97.3		99.4		96.5	
		%	mg eq/kg	%	mg eq/kg	%	mg eq/kg	%	mg eq/kg	%	mg eq/kg
Hexane ²		n/a		n/a		1.38	0.002	n/a		5.10	0.021
CH ₂ Cl ₂		7.89	0.221	5.80	0.153	17.5	0.022	5.76	0.086	11.3	0.046
Acetone		44.5	1.250	40.0	1.048	41.5	0.051	25.7	0.383	6.70	0.028
Methanol		18.8	0.526	22.4	0.588	22.9	0.028	29.8	0.443	22.6	0.093
Water (acidified and refluxed) ⁵	Ethyl acetate under acidic conditions	5.32	0.149	9.20	0.240	2.11	0.003	11.1	0.165	26.7	0.110
	Ethyl acetate at pH 7	<0.5	n/a	1.20	0.033	0.65	0.001	2.19	0.033	<0.5	n/a
	Aqueous soluble fraction	5.83	0.164	6.90	0.182	7.40	0.009	8.54	0.127	14.3	0.060
Solids (unextractable) ³		5.46	0.153	8.90	0.234	3.90	0.005	16.3	0.243	9.80	0.040

¹⁾ Values are from one of four replicates, except for 10 day seed and straw in which two replicates were combined due to low total radiocarbon content; n/a = not applicable.

²⁾ Used for seed only to remove lipid content which reduced emulsification for more polar solvent extractions.

³⁾ Prior to acid and base hydrolysis.

⁴⁾ Not a RAC plant part

⁵⁾ Water extract was acidified in 1N HCl prior to refluxing for 1 hr. at 90 °C, which was then followed by ethyl acetate extraction.

Isolation and identification:

The TLC and HPLC isolated radiocarbon from different samples was subjected to extensive clean-up using several systems: TLC, HPLC, ion exchange columns, counter ion chromatography and other separation techniques. The cleaned-up samples were subject to MS and NMR analysis. Because of the polar nature of the metabolites, the cleaned-up samples still contained a relatively large amount of co-extractives. The NMR and MS analysis yielded only limited information. The oxalamide (M23) was confirmed. All other samples failed confirmation by MS, HRMS, NMR and HRNMR despite extraction of large samples and (up to 160 grams) and extensive clean-up. The sample size was not the limiting factor but the presence of the co-extractives and the polar nature of the metabolites were the

limiting factors. To resolve this problem TLC bands corresponding to analytical reference standards metabolites were extracted and subjected to HPLC qualitative and quantitative confirmation. The sulfonate (M27) and sulfoxide (M30) of thiolactic acid metabolites were thus confirmed by HPLC. The sulfoxide of thioglycolic acid (M31) was confirmed by a second TLC system.

Table B.7.2-12: Characteristics and identification of dimethenamid residues in soya bean plants after soil treatment at 1.68 kg as/ha (2 N) (expressed as the % of TRR and mg/kg dimethenamid equivalent)

Crop Part		Forage		Hay		Seed	
DAT		49		100		118	
TRR [mg/kg] ⁵		2.80		2.62		0.41	
		% ^{1,2}	mg/kg	%	mg/kg	%	mg/kg
Parent		ND ⁴		ND		ND	
Oxalamide (M23)		16.76	0.47	5.28	0.14	6.55	0.03
Sulfonate (M27)		7.03	0.20	10.60	0.28	7.54	0.03
Sulfoxides (M30 and M31)		6.02	0.17	7.77	0.20	11.7	0.04
Unidentifiable ³	Methylene chloride	7.00	n/a	4.10	n/a	8.40	n/a
	Acetone	27.1	n/a	25.4	n/a	1.9	n/a
	Methanol	8.0	n/a	16.2	n/a	8.6	n/a
	Ethyl acetate	4.6	n/a	9.3	n/a	22.8	n/a
	Water	5.8	n/a	6.9	n/a	14.3	n/a
Unextractable		5.5	n/a	n/a	n/a	n/a	n/a

¹⁾ % of total radioactive residue. Residue isolated by TLC, qualitative and quantitative confirmation by HPLC with limited NMR and MS confirmation because of the polar nature of the metabolites and co-extractables. Because of the low residue in seeds some values are from TLC analysis only, no qualitative or quantitative confirmation.

²⁾ Summation of metabolites from different extracts. Some values are extrapolated from the 3.3 kg/ha treatment. Reported residue is from the highest replicate in two studies. Residue can be as low as one tenth of reported value.

³⁾ TLC and/or HPLC analysis showed at least 12 peaks in each extract, each peak generally representing <0.02 mg/kg.

⁴⁾ ND = <0.003 mg/kg.

⁵⁾ Replicate with the highest residue (total radiocarbon).

Proposed metabolic pathway:

Metabolism is probably via glutathione and/or cysteine conjugation, hydrolysis of the glutathion conjugate to the cysteine conjugate, deamination of the cysteine conjugate to the thiolactic conjugate followed by oxidation to form the sulfoxide conjugate and β -Lyase cleavage plus oxidation to the sulfonate (Figure B.7.2-8).

Conclusion

Dimethenamid was rapidly metabolised in soya bean leading to a large number of mostly polar metabolites. The maximum concentration of any of the isolated metabolites is not greater than 0.47 mg/kg in any of the RAC's from the maximum label application rate of 1.68 kg as/ha (2 N). There is no evidence of parent compound present. Furthermore, an increasingly large proportion of the radiocarbon in RAC's is converted to conjugated and unextractable forms and the longer the sampling period, the larger the amount of conjugated and unextractable radiocarbon

Three metabolites of this pathway were identified in extracts of the RACs:

M27 - sulfonate,

M31 - sulfoxide of thioglycolic acid,

M30 - sulfoxide of the thiolactic acid conjugate.

In addition, oxalamide (M23) was also present via either uptake from the soil. These metabolites have all been identified in the rat, with many also found to be present in the goat and/or hen, demonstrating

that animal metabolism proceeds through similar pathways.

The study confirms the results of the more recent study by Schweda (2012, [ASB2014-8341](#) and 2014, [ASB2014-10169](#)). However, due to limitations with regards to metabolite identification, the study can only be considered as supplementary.

B.7.2.1.3 Sugar beet

Reference:	KCA 6.2.1/6
Report:	Lam, W. W. (1996): Amended final report: [3- ¹⁴ C-Thienyl]dimethenamid: Metabolism in sugarbeet (<i>beta vulgaris</i> L.) BASF DocID: 98/5173, Protocol No. E95-04 RIP1999-828
Guidelines:	Yes (EPA Pesticide Assessment Guideline, Residue Chemistry Series 171-4[a])
Deviations:	No
GLP:	Yes
Acceptability:	The study is acceptable.

Material and methods

Test Material:	Dimethenamid (SAN582H)
Lot/Batch #:	RA 683-3 (thienyl-3- ¹⁴ C)
Purity:	99.2 % (chemical purity)
Specific activity:	183.6 µCi/mg (6.79 MBq/mg)
CAS#:	87674-68-8
Test Commodity:	Root vegetable
Crop:	Sugar beet
Variety:	GALA
Botanical name:	<i>Beta Vulgaris</i> L.
Crop parts(s) :	Roots and tops

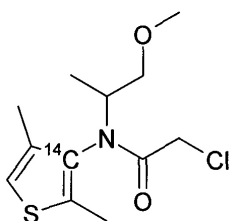


Figure B.7.2-7: Chemical structure and position of radioactive label of racemic dimethenamid

Experimental conditions:

The field portion of this study was conducted in Switzerland. Sugar beet seeds were planted into top soil in containers at a depth of 1.5 cm. The sugar beet variety used in this study was *Beta vulgaris* L. var. GALA, a variety widely used in Europe. The soil used in this study was a sandy loam topsoil. Sugar beets were grown in 7 containers with a surface area of 0.5 m² per container. One container was the control container, three containers were used for the maximum labelled rate and three for the exaggerated rate. The plants were kept in a greenhouse for the first 41 days and then were moved to an outdoor shed until harvest. For the maximum label use rate, dimethenamid was applied three times as early post-emergence and the application rate for each treatment was 450 g/ha. The total application rate was 1.35 kg/ha (1.9 N). The first application was conducted after the cotyledons were completely unfolded (BBCH 10). Subsequent treatments were conducted after 9 and 21 days of the first treatment. For the three exaggerated rate plots, sugar beet plants in each plot were treated with 900, 1800 and 1350 g as/ha, respectively, totalling to 5400 g as/ha per plot. Phytotoxicity occurred after the second

treatment with 1800 g/ha. Therefore, instead of treating the plants with 2700 g/ha, the third treatment was split into two 1350 g/ha applications separated by 8 days. The RACs from the exaggerated rate were not analysed.

Sampling and sample storage:

Samples were taken at the following intervals after application:

DAT 126 - roots & leaves with tops after 3 x 0.45 kg as/ha soil treatment

DAT 105 - roots & leaves with tops after 0.9, 1.8, 1.35, 1.35 kg as/ha soil treatment

All samples were kept in a freezer at approximately -18 °C until analysis took place.

Extraction of residues and analytical determination:

The samples were extracted with a mixture of methanol:water (80:20 v/v), the methanol was evaporated and the aqueous layer was extracted with methylene chloride. The aqueous from the methylene chloride extraction was freeze-dried, redissolved and the radioactivity was determined by LSC. Afterwards, the plant materials were combusted in order to determine their remaining radioactivities. Unextracted residues in roots were subjected to acidic hydrolysis (6 N HCl at 20 °C for 24 h) followed by alkaline hydrolysis (6 N NaOH at 30 °C and 40 °C for each 24 h).

Total radioactive residue (TRR) levels in roots and leaves with tops were determined using both the fresh and freeze-dried samples by means of a sample oxidiser. The radiocarbon content of liquid samples was determined by using a liquid scintillation counter. Identification and characterisation of sample extracts was performed by TLC and HPLC.

Results and discussion

Extractability:

The results from this study showed that the ¹⁴C levels in roots were approximately 3.5 times lower than in leaves with tops. Regardless of the samples matrices, a large amount of the ¹⁴C radioactivities were found in the methanol:water (80:20 v/v) extracts. Analyses of the methylene chloride extracts showed that dimethenamid was rapidly metabolised into numerous polar and low level components in sugar beet. The results are summarised in Table B.7.2-13.

Combustion of the remaining bound residue for roots and tops indicated their ¹⁴C levels to be 0.016 mg/kg (20.54 % TRR) and 0.017 mg/kg (5.99 % TRR), respectively. The bound residues of roots were further hydrolysed with 6 N HCl and 6 N NaOH solutions. The individual acid and base hydrolysates and the plant material were <0.01 mg/kg and they were not further characterised.

Table B.7.2-13: Extraction characteristics of dimethenamid in sugar beet plants treated at 1.35 kg as/ha (1.9 N), expressed as % of methanol/water extractable radioactivity and mg/kg dimethenamid equivalents

Crop part	Roots		Tops	
	%	mg eq/kg	%	mg eq/kg
¹⁴ C TRR	100	0.0783	100	0.284
MeOH:H ₂ O (80:20, v/v)	80.49	0.063	93.63	0.266
Water	3.04	0.0024	1.73	0.0049
ERR ¹	83.54	0.0654	95.35	0.271
RRR ²	20.54	0.0161	5.99	0.017
Recovery	104.07	0.0815	101.35	0.288

¹) ERR = Extracted Radioactive Residues

²) RRR = Remaining Radioactive Residues

Isolation and identification

The organosoluble extracts from the multiple step procedure were analysed by Thin Layer Chromatography (TLC) and High Pressure Liquid Chromatography (HPLC) systems. The R_f and R_t values were compared to authentic standards.

Dimethenamid underwent extensive metabolism once it was sprayed into the field. No parent

compound was detected and none of the metabolites had a level ≥ 0.01 mg/kg and ≥ 10 % TRR. In addition, all the metabolites present were more polar than parent dimethenamid as shown in Table B.7.2-14.

Table B.7.2-14: Characteristics and identification of dimethenamid residue in organic extracts from sugar beet plants treated at 1.35 kg as/ha (1.9 N) (expressed as % of total radioactive residue and mg/kg dimethenamid equivalent)

	Residues in roots		Residues in leaves with tops	
	mg eq/kg	% TRR	mg eq/kg	% TRR
Starting material	0.0783	100	0.2843	100
Extracted material	0.0654	83.54	0.2710	95.35
Parent	ND	ND	ND	ND
M23 Oxalamide	0.0009	1.09	-	-
M27 Sulfonate	0.0047	6.01	0.0185	6.5
M28 Sulfoxide of cysteine conj.	0.0018	2.25	-	-
M29 N-Malonylcysteine conjugate	0.0044	5.66	0.0029	1.01
M30 Sulfoxide of thiolactic acid conj.	-	-	0.0267	9.39
Total identified	0.0118	15.01	0.0481	16.90
Total characterised	0.0477	61.21	0.2134	75.11
Total identified / characterised	0.0596	76.23	0.2615	92.01

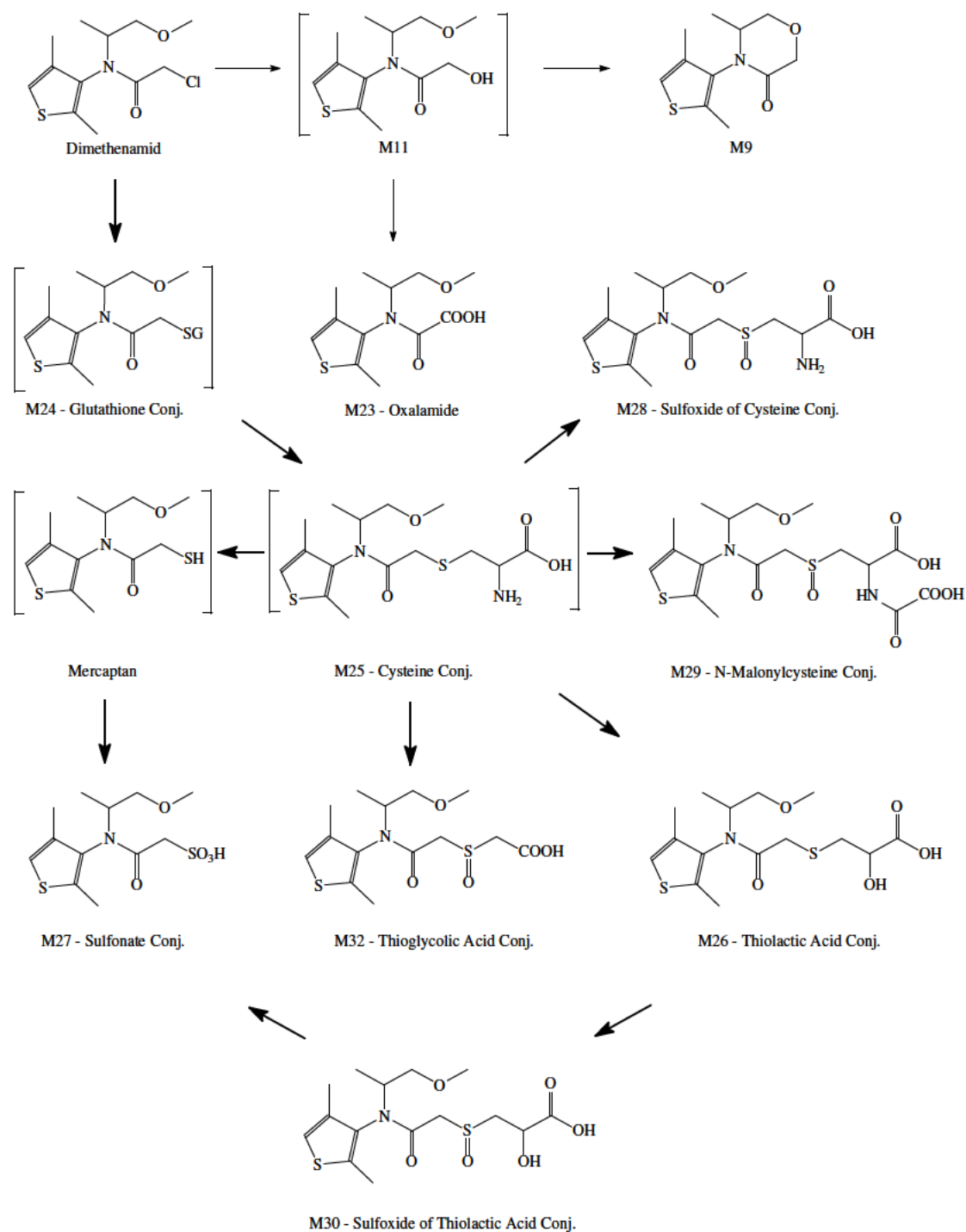
Proposed metabolic pathway:

Based on the identified metabolites the proposed metabolic pathway involves conjugation of dimethenamid with glutathion and hydrolysis of the glutathion conjugate to the cysteine conjugate, both are considered transient intermediates undergoing rapid oxidation, deamination and/or decarboxylation.

Conclusion

Dimethenamid applied on sugar beet at a rate of 1.35 kg as/ha (1.9 N) was rapidly metabolised to a large number of polar metabolites. The major pathway is via glutathione conjugation. Four metabolites of this pathway were identified in extracts of the sugar beet roots and three in sugar beet leaves with roots:

The metabolites that were detected in both roots and tops were sulfonate (M27) and N-malonylcysteine conjugate (M29). Oxalamide (M23) and sulfoxide of cysteine conjugate (M28) were also detected in sugar beet roots. The sulfoxide of thiolactic acid (M30) was identified only in sugar beet leaves. The parent dimethenamid was not detected in sugar beet tops or roots. Figure B.7.2-8 summarises the main degradation steps.



Note: Solid arrows indicate glutathione conjugation pathways.

Figure B.7.2-8: Proposed metabolic pathway of dimethenamid in sugar beet

B.7.2.1.4 Overall conclusion plant metabolism

Plant metabolism studies have been provided for the crop categories cereal (maize), pulses and oilseeds (soya bean) and root crops (sugar beet) covering three different crops groups and all representative uses. New studies were provided for maize and soya beans, confirming the findings of the previous evaluated studies for these crops. Due to analytical limitations with regards to analyte identification, the older studies for maize and soya bean were therefore considered as supplementary only.

Residues in edible parts of the plants were all below 0.01 mg/kg. The only metabolites contributing to more than 10 % TRR were M26 and M30 in maize forage (DAT 30) and M26 in soya bean leaves. No parent was detected all crops investigated.

The main route of metabolism was similar in all crops investigated. It involves rapid glutathione conjugation of dimethenamid-P, enzymatic cleavage of the tripeptide and subsequent metabolic reactions on the resulting cysteine conjugate (loss of the amino group of the cysteine moiety and S-oxidation). Various sulfur-containing secondary metabolism products (modified cysteine conjugates) M30, M31 (or isomer), M26 and M37 (or isomer) represented the major part of the extractable radioactive residues. A second metabolic route is hydrolytic/oxidative displacement of the chlorine atom, followed by glycosylation (to form one to several isomers of the metabolite M40) or further transformation (e.g. oxidation, leading to the oxalamide M23, or cleavage of the acetamide to form the amine metabolite M39).

B.7.2.2 Poultry

During the initial EU Review of the active substance dimethenamid-P the metabolism was investigated in laying hen.

Reference:	KCA 6.2.2/1
Report:	[REDACTED]: Metabolism of SAN 582 H in laying hens BASF DocID: 90/11110, Project No. 414105 RIP1999-834
Guidelines:	EPA Pesticide Assessment Guideline, Residue Chemistry Series 171-4
Deviations:	No
GLP:	Yes
Acceptability:	The study is acceptable.
Reference:	KCA 6.2.2/2
Report:	[REDACTED] SAN 582 H: Addendum to a previous hen metabolism study BASF DocID: 92/12430, Project No. 414105 RIP1999-835
Guidelines:	EPA Pesticide Assessment Guideline, Residue Chemistry Series 171-4
Deviations:	No
GLP:	Yes
Acceptability:	The study is acceptable.

Material and methods

Test Material:	Dimethenamid (SAN582H)
Lot/Batch #:	RA 683-3 (thienyl-3- ¹⁴ C),
Purity:	98 % radiochemical purity
Specific activity:	43.2 mCi/mmol (5.75 MBq/mg)
CAS#:	87674-68-8
Test animal	Laying hens (Rhode Island Red)
Animal species:	<i>Gallus gallus domesticus</i>
Commodities:	Eggs, excreta, tissues

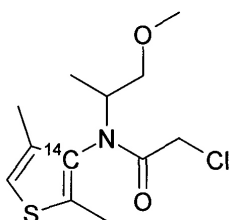


Figure B.7.2-9: Chemical structure and position of radioactive label of racemic dimethenamid

Experimental conditions:

Three laying hens were fed with a gelatine capsule a day for four days. Details of the study are summarised in Table B.7.2-15. The nominal dose 167 mg/kg of feed level was used to simulate an exaggerated dose compared to the expected residue level of 0.01 mg/kg in plant parts as feed items. Thus, it will allow a judgement if residues are likely to occur in poultry under normal agricultural practices.

Table B.7.2-15: Dosing of laying hens with [3-¹⁴C-thienyl] dimethenamid

Number of doses	Nominal dose [mg/kg day]	Actual dose [mg/kg]		Sacrifice time [h]
		feed concentration	per body weight*	
4	10	167	10	7

*) body weight at start of dosing

Sampling and sample storage:

Excreta and eggs were collected daily and the animals killed 7 hours after last dosing. Because the three ¹⁴C dosed hens produced an egg about only every other day, a total of eight eggs only could be collected during the study. The samples of eggs were separated into yolk and egg white for analysis. All samples were kept in a freezer at -20 °C.

Extraction of residues and analytical determination:

Excreta, egg yolk, egg white, liver, muscle and fat were first extracted with organic solvents. Non-extractables were released using acid and base hydrolyses. For egg yolk and egg white, the non-extractables were further released using enzymatic methods.

Metabolites in excreta, liver and fat were identified by co-chromatography with reference standards, MS and/or GC/MS techniques.

Results and discussion

Radioactive residue and extractability:

Following the administration of ¹⁴C-dimethenamid to laying hens, the radioactivity was rapidly excreted (see Table B.7.2-16). Based on the ¹⁴C residue in excreta alone, it accounted for >77 % of the total applied dose at sacrifice.

Table B.7.2-16: Material balance in laying hens

Fraction	% of total dose of 3-thienyl label
Egg yolk	0.02
Egg white	0.02
Liver	0.48
Muscle	0.68
Fat	0.07
Excreta	77.19
Gastro-intestinal tract	*
Cagewash	*
Total	78.46

*) not collected

With the 167 mg/kg dose level, the residue concentrations in egg white were 0.19 (24 h), 0.20 (72 h) and 0.30 mg eq/kg (79 h). The egg yolk residue concentrations at 24, 72 and 79 h were 0.01, 0.24 and 0.62 mg eq/kg, respectively (Table B.7.2-17). Residue concentrations for fat, muscle (breast), muscle (thigh) and liver were 0.29, 0.45, 0.58 and 8.33 mg eq/kg, respectively. The low residue concentration in fat indicated that there was no risk of bioaccumulation. Organosolubles in egg, different tissues and excreta ranged from approximately 1 to 75 % TRR. Additional residues were partitioned into organic solvents after acid and bases hydrolyses, and/or enzymatic studies with the exception of excreta (Table B.7.2-18).

Table B.7.2-17: Total radioactive residue levels in tissues of laying hens

Tissue	TRR [mg eq/kg]*
Egg yolk	0.01, 0.24, 0.62**
Egg white	0.19, 0.2, 0.3**
Muscle - breast	0.45
Muscle - thigh	0.58
Fat	0.29
Liver	8.33

*) average from three hens

**) samples collected at 24, 72 and 79 h

Liver:

21 metabolites were detected by TLC analysis in organic extracts. Besides of the M3 and M8 metabolite, other metabolites each accounting 0.1 to 8 % of liver radioactivity were detected, but none of these could be identified. The aqueous phase was further characterised to consist of several components each of less than 4 % of liver radioactivity.

Muscle:

12 radiocarbon spots were detected, each accounted for less than 10 % of TRR. Due to low radioactivity no identification was possible. The aqueous phase was further characterised to consist of several components each less than 2 to 4 % of liver ¹⁴C.

Fat:

12 radiocarbon spots were detected upon TLC analysis. Besides of parent no other metabolite could be identified; each of these accounted for less than 9 % of TRR in fat. Further characterisation was performed, but no metabolite identification was possible.

Egg white and egg yolk:

The organic extractable radioactivity of egg white was separated into 14 spots by TLC analysis, each

accounted for less than 10 % of TRR in egg white, the concentration were 0.003 to 0.03 mg eq/kg. The extractable radioactivity of egg yolk was separated into 8 spots by TLC analysis. Due to the low concentration (0.01 to 0.04 mg eq/kg each, less than 10 %) no metabolite could be identified. In the water soluble fraction of egg white and yolk were several components characterised but none identified.

Table B.7.2-18: Extraction of radioactivity from eggs, tissues and excreta

	Residue [mg eq/kg] (% TRR)					
	Egg white*	Egg yolk*	Fat	Muscle	Liver	Excreta
Organic solvent extracts	0.100 (33.41)	0.006 (0.99)	0.217 (74.76)	0.257 (44.23)	4.223 (50.68)	(64.33)
Organosoluble post acid and base released (1 N and/or 5 N)	0.125 (41.62)	0.318 (51.18)	0.039 (13.58)	0.231 (39.74)	3.579 (43.29)	n.p.**
Aqueous, including ethyl acetate from partitioning of hydrolysates	0.008 (2.80)	0.024 (3.83)	0.022 (7.68)	0.011 (5.22)	0.23 (2.86)	n.p.
Enzyme released organosoluble	0.059 (19.51)	0.267 (43.03)	n.p.	n.p.	n.p.	n.p.
Unextractable or soluble fat	n.a.**	n.a.	0.003 (0.90)	0.030 (2.64)	0.57 (6.81)	(26.04)
Total	0.292 (97.34)	0.614 (99.03)	0.281 (96.92)	0.529 (91.83)	8.602 (103.65)	(90.36)

*) egg collected at 79 h after dose initiation (7 h after last dose)

**) n.p. = not performed; n.a. = not applicable

Characterisation and identification:

The analysis of fat indicated parent dimethenamid was above 0.05 mg eq/kg. Moreover M3 and M8 were identified in liver samples (Table B.7.2-19).

Table B.7.2-19: Summary of metabolites in eggs, tissues and excreta of laying hens

Metabolite	Excreta	Egg yolk	Egg white	Muscle	Fat	Liver
	[mg eq/kg] (%TRR)	[mg eq/kg] (%TRR)	[mg eq/kg] (%TRR)	[mg eq/kg] (%TRR)	[mg eq/kg] (%TRR)	[mg eq/kg] (%TRR)
Dimethenamid	(2.03)	ND*	ND	ND	0.075 (26.23)	ND
M1	(1.43)	ND	ND	ND	ND	ND
M3	(10.03)	ND	ND	ND	ND	0.429 (5.14)
M7	(2.23)	ND	ND	ND	ND	ND
M8	(0.48)	ND	ND	ND	ND	0.650 (7.80)
M11	(0.79)	ND	ND	ND	ND	ND
M12	(4.64)	ND	ND	ND	ND	ND
M17	(1.52)	ND	ND	ND	ND	ND
M22	(1.00)	ND	ND	ND	ND	ND
M25	(1.10)	ND	ND	ND	ND	ND
M27	(0.51)	ND	ND	ND	ND	ND
M30	(3.52)	ND	ND	ND	ND	ND
M32	(1.05)	ND	ND	ND	ND	ND

*) ND = not detected

Proposed metabolic pathway:

Dimethenamid was rapidly and extensively metabolised and then excreted. The metabolic pathway was via glutathione conjugation, reductive dechlorination followed by the formations of cysteine and mercapturate conjugations, and dimerisation of a mercaptan intermediate as can be seen in excreta. The other pathways included O-demethylation and reductive dechlorination. In liver, metabolites ≥ 0.05 mg/kg were M3 (reductive dechlorination) and M8 (O-demethylation and cyclisation) (Figure B.7.2-13).

Conclusion

This study was conducted using dimethenamid (racemic mixture). Consequently, the metabolism of dimethenamid-P was included since the racemic mixture contained 50 % of the R-enantiomer (dimethenamid-P). Most of the residues were excreted (77 % TRR), while only a minor fraction of the TRR was found in edible tissues and egg. No individual metabolites were detected in edible tissues and egg, with the exception of dimethenamid in fat and M3 and M8 in liver. Metabolite M30, which is major plant metabolite, was detected in excreta equal to 3.5 % TRR. As the study was performed with the parent dimethenamid, which was not detected in any of the plant metabolism studies, it does not reflect the actual exposure scenario if poultry is fed with feed treated with dimethenamid. However, the study can still be considered acceptable since M30 was detected in excreta and is therefore bioavailable in poultry.

B.7.2.3 Lactating ruminants

During the initial EU Review of the active substance dimethenamid-P the metabolism was investigated in lactating ruminant. For the renewal of the approval, an additional goat metabolism study was performed using M30.

Reference:	KCA 6.2.3/1
Report:	XXXXXXXXXX M656PH030 - Metabolism of ^{14}C -M656PH030 in the lactating goat BASF DocID: 2013/7002636, BASF Study ID: 390493, Ricerca Study ID: 030825 ASB2014-8342
Guidelines:	US EPA OPPTS 860.1300, US EPA OPPTS 860.1000, Commission of the European Communities 7028/VI/95 rev .3, OECD Guideline for the testing of chemicals: Metabolims in Livestock Document 503 (8 th January 2007)
Deviations:	No
GLP:	Yes
Acceptability:	The study is acceptable.

Material and methods

Test Material:	M30 (M656PH030)
Lot/Batch #:	1089-1003 (thienyl-3- ^{14}C), 1088-1005 (thienyl-5- ^{13}C), L74-138 (unlabelled)
Purity:	^{14}C : 98 % (radiochemical purity) ^{13}C : 92.6 % (chemical purity)
Specific activity:	5.17 MBq/mg
CAS#:	Not specified
Test animal:	Goat
Species:	<i>Capra hircus</i>
Gender:	Lactating female
Age:	2-4 years

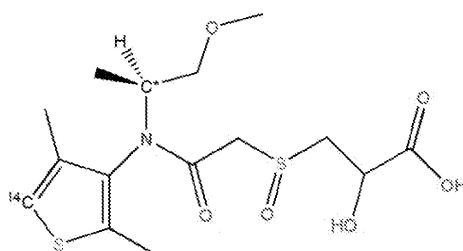


Figure B.7.2-10: Chemical structure and position of radioactive label of metabolite M30

Experimental conditions:

The dosing solution, ^{14}C -M30 was prepared by isotopic dilution with ^{13}C -M30 and ^{12}C -M30 in a 1:1:1 ratio to facilitate metabolite identification by mass spectrometry. The total amount of M30 was 256.1 mg.

An aliquot of the dosing solution containing 23.9 mg of test material corresponding to a nominal dose of 12 mg/kg diet was added to each gelatine capsule half containing cellulose. The solvent was allowed to evaporate overnight and capped with the remaining capsule half. A total of ten dosing capsules were prepared. The capsules were stored under freezer conditions. Two stability capsules were prepared similarly and were used to verify concentration and stability of the test substance in the capsules during the dosing period.

One lactating goat was administered ten consecutive daily oral doses of ^{14}C -M30 (nominal dose of 12 mg/kg feed/day). The average actual dose was 13.79 mg/kg of feed/day and 0.5663 mg/kg body weight/day. The average actual dose was 115 % of the nominal dose.

Sampling and sample storage:

Day 1 blood was collected at 0.5, 1, 2, 3, 4, 6, 8, 10, 12 and 24 h after the first dosing for combustion and LSC counting to determine T_{\max} . T_{\max} was determined to be 2 h. As specified in the protocol, the goat was sacrificed 2 h (T_{\max}) following the last (10th) dose. During the course of the study, urine, faeces and cage wash were collected daily. Milk was collected twice daily. Tissues (liver, kidney, loin muscle, flank muscle, omental fat, renal fat, subcutaneous fat and GI tract and contents), blood, and

bile were collected at termination. Aliquots of all samples were assayed by combustion (except for milk), and LSC for ^{14}C content.

Extraction of residues and analytical determination:

Faeces:

An aliquot of homogenised Day 8 faeces was weighed and extracted three times with methanol and twice with water. Each extract was centrifuged to separate solids, decanted volumes recorded and aliquots of each were analysed by LSC. The methanol extracts were pooled and concentrated to the aqueous phase to give an aqueous concentrate which was analysed by HPLC.

Liver:

An aliquot of homogenised liver tissue was weighed and extracted once with acetonitrile, twice with acetonitrile/water (1:1, v/v) and once with acetonitrile. Each extract was centrifuged and aliquots of each were analysed by LSC. The residual radioactive residues (RRR) were allowed to dry in the hood, and then aliquots were assayed by combustion analysis. The acetonitrile extracts were pooled and concentrated to the aqueous phase to give an aqueous concentrate which was analysed by HPLC. The post-extracted solid (PES) was subjected to protease digestion followed by extraction with acetonitrile. The acetonitrile extract was concentrated and then analysed by HPLC. The PES2 was further subjected to HCl hydrolysis and then extracted with ethyl acetate. The ethyl acetate extract was concentrated and reconstituted in HPLC mobile phase prior to HPLC analysis.

Kidney:

An aliquot of homogenised kidney tissue was weighed and extracted once with acetonitrile and twice with acetonitrile/water (1:1, v/v). Each extract was centrifuged and aliquots of each were analysed by LSC. The ACN extracts were pooled and concentrated to the aqueous phase to give an aqueous concentrate which was analysed by HPLC.

Loin muscle:

An aliquot of homogenised loin muscle tissue was weighed and extracted once with acetonitrile, three times with acetonitrile/water (1:1, v/v) and once with acetone. Each extract was centrifuged and aliquots of each were analysed by LSC. The ACN extracts were pooled and concentrated to the aqueous phase to give an aqueous concentrate which was analysed by HPLC.

Milk:

An aliquot of Day 8 PM and Day 8 milk was weighed and extracted once with acetonitrile and twice with acetonitrile/water (1:1, v/v). Each extract was centrifuged and aliquots of each were analysed by LSC. The extracts were pooled and concentrated to the aqueous phase to give an aqueous concentrate which was analysed by HPLC.

Renal fat:

An aliquot of homogenised renal fat tissue was weighed and extracted once with acetonitrile, once with acetonitrile/water (1:1, v/v), once with acetone and once with methanol. Each extract was centrifuged and aliquots of each were analysed by LSC. The extracts were pooled and concentrated to the aqueous phase to give an aqueous concentrate. The aqueous concentrate was analysed by HPLC with fractions collected every 30 seconds for LSC.

Metabolite patterns and isolation and characterisation of metabolites in urine:

Day 1-9 urine was analysed by HPLC equipped with a liquid cell to determine the metabolite pattern. Day 2 urine and Day 8 urine were injected onto an HPLC equipped with a solid cell to isolate major and minor urinary metabolites for LC-MS identification. Day 1-9 urine samples were further analysed by a second HPLC method to determine the isomeric metabolite pattern. The low residues found in edible tissues precluded metabolite isolation, purification, and characterisation/identification of metabolites from tissues. Since the radio profile in urine was qualitatively similar to the observed radio profile in tissues, identification of metabolites was accomplished from pooled urine sample.

The isolated HPLC fractions were directly analysed by LC-MS without further treatment. All isolated HPLC fractions were first analysed by Q1 scanning in search for the distinct isotope pattern. If a metabolite was observed, MS2 scanning of all three isotopes (^{12}C , ^{13}C and ^{14}C) of the M30 ion to reveal the daughter ions was performed.

Results and discussion

Radioactive residue and extractability:

The overall recovery of radioactivity is provided in Table B.7.2-20. Approximately 100.6 % of the

administered dose was recovered, the majority of which was present in the urine (51.7 %), faeces (36.8 %) and GI tract contents (11.7 %) indicating that urinary and faecal excretion is the major elimination pathway. A small amount of radioactivity was recovered in bile (0.03 %) and cage wash (0.3 %). Radioactivity associated with edible portions (milk and tissues) accounted for 0.2 % of the administered dose.

Table B.7.2-20: Recovery of radioactivity after administration of ^{14}C -M30 to lactating goats and total radioactive residues (TRRs) in milk, excreta and tissues

	^{14}C -M30	
	TRR [mg eq/kg]	% of the dose
Urine	5.525-8.043	51.7
Cage wash	n.r.	0.3
Faeces	4.903-8.013	36.8
Muscle - loin	0.016	n.r.
Muscle - flank	0.017	n.r.
Liver	0.219	0.1
Kidney	0.243	<0.1
Fat - omental	0.011	n.r.
Fat - renal	0.014	n.r.
Fat - subcutaneous	0.017	n.r.
GI tract and intestinal contents	n.r.	11.7
Termination bile	1.221	<0.1
Milk	0.012-0.018	0.1
Total	n.r.	100.6

n.r. Not reported

The concentration of radioactivity in blood was measured at regular intervals throughout the 24 h period following the first dose administration. Results are provided in Table B.7.2-21. The level of radioactivity in the blood reached to a maximum of 0.124 mg eq/kg at 2 h post first dose.

Table B.7.2-21: Residues after administration of ^{14}C -M30 to lactating goats and total radioactive residues (TRRs) in blood from Day 1

Day 1 Blood	^{14}C -M30
	TRR [mg eq/kg]
0.5 h	0.016
1 h	0.046
2 h	0.124
3 h	0.111
4 h	0.094
6 h	0.083
8 h	0.055
10 h	0.047
12 h	0.040
24 h	0.013

TRR data for milk are provided in Table B.7.2-22. Residues in the milk reached to a plateau maximum of 0.018 mg eq/kg within 3 days. Recovery of radioactivity in milk accounted for 0.1 % of the administered dose, indicating that transfer of residues to milk was low. The ratio of radioactive residues in the cream and skimmed milk fractions was determined in a representative 24 h milk sample from the plateau region. The ratio of residues in the cream to skimmed milk was 1.1 : 1, with residues equivalent to 0.009 mg eq/kg and 0.008 mg eq/kg in the cream and skimmed milk, respectively.

Table B.7.2-22: Residues after administration of ^{14}C -M30 to lactating goats and total radioactive residues (TRRs) in milk

Day	^{14}C -M30
	TRR [mg eq/kg]
1	0.012
2	0.016
3	0.018
4	0.015
5	0.016
6	0.015
7	0.016
8	0.018
9	0.017
10	NA

Urine samples were directly measured and Day 8 faeces sample was extracted with methanol and water, resulting in 58.9 % TRR in the extract. The remaining 51.5 % TRR was present in the solid debris which was analysed by combustion analysis. The extracts were combined and concentrated for HPLC analysis (see Table B.7.2-23).

Table B.7.2-23: Residues after administration of ^{14}C -M30 to lactating goats and total radioactive residues (TRRs) in urine, faeces and cage wash

Time [h]	Faeces		Urine	
	TRR [mg eq/kg]	% of dose	TRR [mg eq/kg]	% of dose
24	4.903	2.1	6.565	5.1
48	7.497	3.9	6.692	5.7
72	8.013	4.1	5.797	6.0
96	7.205	4.5	6.236	5.9
120	6.663	4.3	7.096	5.8
144	5.877	4.3	6.350	5.9
168	6.274	4.0	6.377	5.6
192	7.260	4.8	8.043	5.8
216	6.091	4.2	5.525	5.9
218	5.209	0.5	n.r.	n.r.

n.r. Not reported

Faeces, milk, liver, kidney, loin muscle and renal fat were extracted to determine the nature of the radioactive residues. Urine and bile samples were amenable to HPLC analysis without any further purification. The extractability is summarised in Table B.7.2-24.

Table B.7.2-24: Extractability of residues of ^{14}C -M30 in goat matrices

Matrix	TRR		ERR ¹		RRR ²		Recovery ³
	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	
Liver	0.219	100.0	0.096	43.7	0.149	68.0	111.7
Kidney	0.243	100.0	0.240	99.1	0.031	12.9	112.0
Loin muscle	0.016	100.0	0.011	64.6	0.006	35.2	99.7
Renal fat	0.014	100.0	0.008	54.3	0.004	28.8	83.1
Day 8 PM milk	0.027	100.0	0.021	79.5	0.003	11.0	90.5
Day 8 milk	0.018	100.0	0.016	87.6	0.001	5.8	93.4

¹⁾ ERR = Extractable radioactive residue

²⁾ RRR = Residual radioactive residues

The homogenised liver sample was extracted with acetonitrile and acetonitrile/water (1:1, v/v), resulting in extraction of 43.7 % TRR (0.096 mg eq/kg). The solid debris was analysed by combustion analysis and contained 68.0 % TRR (0.149 mg eq/kg). The extracts were combined and concentrated for HPLC analysis. The nature of unextracted radioactivity in the liver debris was investigated by treatment with digestive enzyme (protease) and further extraction of the digested material with acetonitrile. The process resulted in a further 10.6 % TRR (0.023 mg eq/kg) being liberated. This protease hydrolysate was concentrated for HPLC analysis.

The liver debris after initial extraction and protease treatment/extraction was investigated by acid hydrolysis. The process resulted in a further 36.0 % TRR (0.079 mg eq/kg) being liberated.

The kidney sample was extracted with acetonitrile and acetonitrile/water (1:1, v/v), resulting in 99.1 % TRR (0.240 mg eq/kg) in the extract. The remaining 12.9 % TRR was present in the solid debris which was determined by combustion analysis. The extracts were combined and concentrated for HPLC analysis.

The homogenised loin muscle sample was extracted with acetonitrile, acetonitrile/water (1:1, v/v), and acetone, resulting in extraction of 64.6 % TRR (0.011 mg eq/kg). The solid debris was analysed by

combustion analysis and contained 35.2 % TRR (0.006 mg eq/kg). The extracts containing significant radioactivity were combined and concentrated for HPLC analysis.

The homogenised renal fat sample was extracted with acetonitrile, acetonitrile/water (1:1, v/v), and acetone, resulting in extraction of 54.3 % TRR (0.008 mg eq/kg). The solid debris was analysed by combustion analysis and contained 28.8 % TRR (0.004 mg eq/kg). The extracts containing significant radioactivity were combined and concentrated for HPLC analysis.

Characterisation and identification:

Analysis of Day 2 urine using HPLC showed a pattern of 10 peaks, the most predominant component was identified as M26. Three more metabolites were identified as M98, M96 and M30. The relatively low concentration of other minor metabolites precluded further identification and they collectively accounted for 0.693 mg eq/kg (10.4 % TRR). Analysis of the same urine sample using another HPLC method revealed the numbers of isomers (rotamers\stereoisomers) observed for M98 (three peaks in a 1:2:1 ratio, consistent with four isomers) and in M96 (two peaks, consistent with two isomers). Co-chromatography of Day 2 urine and a diluted ¹⁴C-M30 dosing solution further confirmed the identification of M30 in urine (see Table B.7.2-25 for numerical results).

Analysis of Day 8 urine using HPLC showed a pattern of 8 peaks, similar to Day 2 urine, except M96 was no longer detected and two additional metabolites M2 and M14 were also identified. The other minor components were not identified due to relatively low concentration and they collectively accounted for 0.615 mg eq/kg (8.5 % TRR). Analysis of the same urine sample using another HPLC method revealed the numbers of isomers (rotamers\stereoisomers) observed for, in M2 (three peaks in a 1:2:1 ratio, consistent with four isomers) and for M14 (two peaks were observed, consistent with two isomers) (see Table B.7.2-25 for numerical results).

Analysis of bile using HPLC showed a pattern of 3 peaks and analysis of faeces extract using HPLC led to a pattern of 10 peaks. The metabolites observed were similar to those found in urine (see Table B.7.2-25).

Table B.7.2-25: Summary of identified components in various goat excreta

Metabolite	Urine (Day 2)		Urine (Day 8)		Faeces (Day 8)		Bile	
	[mg eq/kg]	[% TRR]	[mg eq/kg]	[% TRR]	[mg eq/kg]	[% TRR]	[mg eq/kg]	[% TRR]
M30	0.410	6.1	0.101	1.3	0.948	13.1	0.484	39.7
M98	1.345	20.1	0.602	7.5	n.r.		0.418	34.2
M2	n.r.		0.770	9.6	0.163	2.2	n.r.	
M96	0.673	10.1	n.r.		n.r.		n.r.	
M14	n.r.		0.683	8.5	0.235	3.2	n.r.	
M26	3.570	53.4	5.205	64.7	2.436	33.5	n.r.	

n.r. Not reported

Analysis of milk extract using HPLC led to a pattern of 5 peaks which were identified with similar metabolites to urine. Analysis of liver extract using HPLC led to a pattern of 11 peaks. Analysis of kidney extract using HPLC led to a pattern of 10 peaks. Four unknown components in kidney accounted for 0.039 mg eq/kg (16.2 % TRR) were detected. Analysis of loin muscle extract using HPLC led to a pattern of 8 peaks. Three unknown components in loin muscle accounting for 0.002 mg eq/kg (14.7 % TRR) were detected. Analysis of renal fat extract using HPLC led to a pattern of 3 peaks. The radioactive residues identified in urine, faeces, milk and edible tissues are summarised in Table B.7.2-26.

Table B.7.2-26: Summary of identified components in various goat tissues and milk

Metabolite	Milk (Day 8)		Liver		Kidney		Loin muscle		Renal fat	
	[mg eq/kg]	[% TRR]	[mg eq/kg]	[% TRR]	[mg eq/kg]	[% TRR]	[mg eq/kg]	[% TRR]	[mg eq/kg]	[% TRR]
M30	0.001	7.6	0.027	12.4	0.048	19.8	0.002	14.1	0.003	23.7
M98	0.002	13.4	0.011	5.3	0.032	13.4	0.002	12.8	0.002	14.1
M2	n.r.		0.010	4.5	0.014	5.9	n.r.		n.r.	
M96	n.r.		0.001	0.6	0.035	14.4	0.001	3.5	n.r.	
M14	0.001	5.8	0.013	6.1	0.011	4.6	0.001	7.3	n.r.	
M26	0.002	13.2	0.011	5.0	0.060	24.8	0.002	12.2	0.002	16.5
Total identified	0.007	40.1	0.074	33.8	0.201	82.9	0.008	49.9	0.008	54.3
Total characterised	0.008	47.5	0.045	20.5	0.039	16.2	0.002	14.7	n.r.	
Total identified and characterised	0.016	87.6	0.119	54.3	0.240	99.1	0.011	64.6	0.008	54.3
PES	0.001	5.8	0.095	43.4	0.031	12.9	0.006	35.2	0.004	28.8
Grand total	0.017	93.4	0.214	97.7	0.272	112.0	0.016	99.7	0.012	83.1

n.r. Not reported

Proposed metabolic pathway:

The proposed metabolic pathway of [thienyl-5-¹⁴C]-M30 in lactating goat is provided in Figure B.7.2-11. [Thienyl-5-¹⁴C]-M30 was extensively metabolised in the lactating goat. The unchanged M30 was found in portions below 24 % TRR in matrices except in bile where M30 comprised 40 % TRR. The main component in urine and faeces was M26, formed by reduction of M30, indicating that urinary and faecal excretion is the major elimination pathway accounting for 89 % of the administered dose. In extracts of milk, liver, kidney, loin muscle, and renal fat the main components were M26 and M30. The metabolite M2 was formed by demethylation of the ether group and substitution of the 2-hydroxypropanoic acid with a methyl group, followed by oxidation of the sulphur group to sulfoxide and subsequent conjugation with glucuronic acid leading to M98. This metabolite was present in significant levels in milk, kidney, loin muscle, and renal fat. Further oxidation of M2 to the corresponding sulfone led to M14 followed by glucuronidation yielding M96. M2, M14 and M96 which were present in some tissue extracts, generally at lower levels.

Thus the following metabolic transformations occurred:

- mainly *via* reduction of the sulfoxide moiety yielding the metabolite M26
- demethylation of the ether group and cleavage of the 2-hydroxypropanoic acid in M26 followed by S-methylation and S-oxidation led to M2 and M14
- glucuronidation of M2 and M14 yielded M98 and M96.

Conclusion

One lactating goat was administered ten consecutive daily oral doses of ¹⁴C-M656PH030 (nominal dose of 12 mg/kg feed/day). The average actual dose was 13.79 mg/kg of feed/day and 0.5663 mg/kg body weight/day. The average actual dose was 115 % of the nominal dose.

The total radioactive residues in milk, muscle and fat were very low and accounted for a maximum of 0.018 mg eq/kg. The residues in the other edible matrices accounted for 0.219 mg eq/kg (liver) and 0.243 mg eq/kg (kidney).

Radioactivity in blood reached a maximum of 0.124 mg eq/kg at 2 h post first dose. Residues in milk had reached steady state within 3 days and the sacrifice time was set as 2 h following the tenth dose.

Approximately 100.6 % of the total dose was recovered, the majority of which was present in the urine (51.7 %), faeces (36.8 %) and GI tract contents (11.7 %). A small amount of radioactivity was recovered

in bile (<0.1 %) and cage wash (0.3 %). Radioactivity associated with edible portions (milk and tissues) accounted for 0.2 % of the administered dose.

Radioactive residues in milk accounted for a plateau concentration of approximately 0.018 mg eq/kg. The ratio of residues associated with skimmed milk and cream was determined in a representative 24 h composite sample from the plateau and found to be 1:1.1.

The identification of metabolites was based on co-chromatography and confirmation by LC-MS/MS of isolated HPLC fractions from pooled urine samples. A summary of the metabolites identified in all faeces, milk and tissue extracts is provided in Table B.7.2-25 and Table B.7.2-26.

The metabolic pathway was sufficiently elucidated.

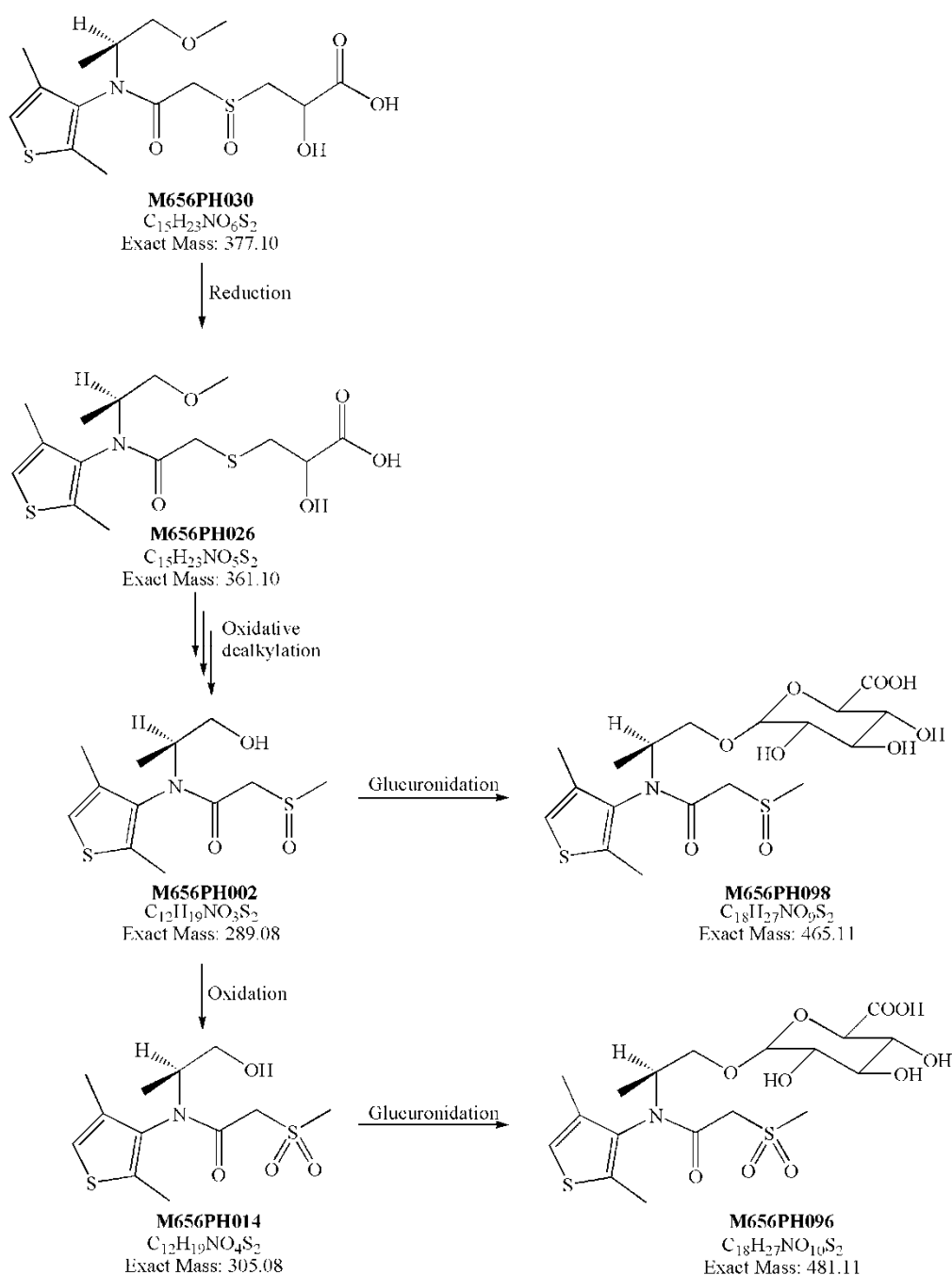


Figure B.7.2-11: Proposed metabolic pathway of ¹⁴C-M30 in lactating goat

Reference:	KCA 6.2.3/2
Report:	<div></div> Metabolism of SAN 582 H in a lactating goat BASF DocID: 90/11112, Project No. 414105 RIP1999-829
Guidelines:	EPA Pesticide Assessment Guideline, Residue Chemistry Series 171-4
Deviations:	No
GLP:	Yes
Acceptability:	The study is considered supplementary only.
Reference:	KCA 6.2.3/3
Report:	<div></div> Material balance investigation in a goat orally administered 14C-SAN 582 H BASF DocID: 90/11113, Project No. 414105 RIP1999-831
Guidelines:	EPA Pesticide Assessment Guideline, Residue Chemistry Series 171-4
Deviations:	No
GLP:	Yes
Acceptability:	The study is considered supplementary and only in combination with the previous study.
Reference:	KCA 6.2.3/4
Report:	<div></div> SAN 582 H: Addendum to a previous goat metabolism study BASF DocID: 92/12431, Project No. 414105 RIP1999-830
Guidelines:	EPA Pesticide Assessment Guideline, Residue Chemistry Series 171-4
Deviations:	No
GLP:	Yes
Acceptability:	The study is considered supplementary and only in combination with the previous study.
Reference:	KCA 6.2.3/5
Report:	<div></div> SAN 582 H: Addendum to previous goat metabolism studies BASF DocID: 92/12432, Project No. 414105 RIP1999-832
Guidelines:	EPA Pesticide Assessment Guideline, Residue Chemistry Series 171-4
Deviations:	No
GLP:	Yes
Acceptability:	The study is considered supplementary and only in combination with the previous study.
Reference:	KCA 6.2.3/6
Report:	<div></div> SAN 582 H: Determination of the presence of sulfonate metabolite in goat excreta BASF DocID: 92/12499, Project No. 414105 RIP1999-833
Guidelines:	EPA Pesticide Assessment Guideline, Residue Chemistry Series 171-4
Deviations:	No
GLP:	Yes
Acceptability:	The study is considered supplementary and only in combination with the previous study.

Material and methods

Test Material:	Dimethenamid (SAN582H)
Lot/Batch #:	RA 683-3 (thienyl-3- ¹⁴ C),
Purity:	98 % radiochemical purity
Specific activity:	43.2 mCi/mmol (5.75 MBq/mg)
CAS#:	87674-68-8
Animal species:	Lactating goat (Nubian)
Commodities:	Milk, excreta, tissues

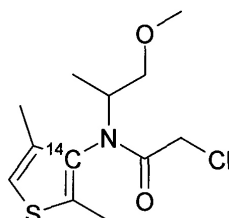


Figure B.7.2-12: Chemical structure and position of radioactive label of racemic dimethenamid

Experimental conditions:

A lactating goat was orally administered the radiolabelled test article contained within a gelatine capsule at a dose of 8.9 mg/kg bw/d for four consecutive days, corresponding to a nominal feed concentration of 223 mg/kg, as detailed in Table B.7.2-27. A second control goat received the vehicle only. The nominal concentration of 223 mg/kg was chosen to simulate an exaggerated dose compared to the expected residue levels (max. 0.01 mg/kg) in plant parts to be used as feed items and thus to allow a judgement if residues are likely to occur in ruminants under normal agricultural practice.

Table B.7.2-27: Dosing of lactating goat with [3-¹⁴C-thienyl] dimethenamid

Number of doses	Nominal dose [mg/kg day]	Actual dose [mg/kg]		Sacrifice time [h]
		feed concentration	per body weight*	
4	10	223	8.9	7

*) body weight at start of dosing

Sampling and sample storage:

Urine and faeces were collected separately 7 and 24 hours after the first dosing and daily thereafter. Milk samples were collected twice daily (in the morning prior to each dosing and in the afternoon, approx. 7 hours after dosing). The animals were sacrificed 7 hours after last dosing. Liver, kidney and samples of thigh muscle and shoulder muscle and omental fat were collected, weighted and frozen. All samples were kept in a freezer at -20 °C.

Extraction of residues and analytical determination:

Faeces, urine, liver, kidney and muscle were extracted using acetone and methanol. In addition, urine was further extracted using ethyl acetate. Non-extractables from faeces, liver, kidney and muscle were released using enzymatic, acidic and/or alkaline hydrolysis. Milk was extracted using methanol. The remaining radioactivity was further released by acidic and alkaline hydrolysis. Fat was extracted with hexane, methanol and chloroform. Non-extractables were released using acidic and alkaline hydrolyses.

Metabolites in urine, faeces, milk, and tissues were identified using GC/MS and/or co-chromatography with reference standards. A separate material balance study (██████, [RIP1999-831](#)) was conducted due to the low recovery of total applied dose.

Results and discussion

Radioactive residue and extractability:

Following the administration of ¹⁴C-dimethenamid to lactating goat, the radioactivity was rapidly excreted (see Table B.7.2-28). Based on the results from the material balance study, more than 59 % and 28 % of administered dose was excreted in the urine and faeces, respectively, at the end of the study.

Table B.7.2-28: Material balance in lactating goat

Fraction	% of total dose of 3-thienyl label	
	main study	separate balance study
Milk	0.022	0.09
Liver	0.75	-
Kidneys	0.08	-
Fat	0.05	-
Muscle	1.36	-
Other tissues	**	-
Cage wash	**	-
Urine*	27.26	59.17
Faeces	8.94	28.08
Total	38.46	87.3

*) On Day 3, the animal urinated immediately after the excreta receiving pan was pulled from the bottom of the metabolism cage to collect faecal sample. The urine sample was lost and not recovered. Therefore, a separate study was conducted for material balance over the period of 5 days. The study indicated a reasonable recovery from urine, faeces, and milk totalling to 87.3 % of the applied dose (second table column).

**) Gastro-intestinal tract contents which can contain considerable residue levels and cage wash were not collected.

At the feed concentration of 223 mg eq/kg, radioactivity was also rapidly excreted into milk: In milk sampled 7 hours and 24 hours after the first treatment, residue levels were approx. 0.51 mg/kg and 0.17 mg/kg, respectively, while the respective levels of 0.9 mg eq/kg and 0.69 mg eq/kg were attained after the second treatment, and 0.98 mg eq/kg and 0.62 mg eq/kg, respectively, on the third day. Thus, the residue level reached a plateau within 3 days.

Residue concentrations in kidney, fat, muscle and liver were 9.92, 0.97, 0.97 and 16.62 mg eq/kg, respectively, (see Table B.7.2-29).

Table B.7.2-29: Total radioactive residue levels in tissues of lactating goat

Tissue	TRR [mg eq/kg]
Fat	0.97
Kidney	9.92
Liver	16.62
Muscle	0.97

Total organosolubles in urine, faeces, milk and tissues ranged from approximately 30 to 82 % TRR. Non-extractable residues were released by acid and base hydrolyses. Additional residues were partitioned into organic solvents, which varied from approximately 1 to 62 % TRR (see Table B.7.2-30).

Table B.7.2-30: Extraction of radioactivity from milk, tissues, and excreta

	Residue [mg eq/kg] (% TRR)						
	Milk	Fat	Muscle	Kidney	Liver	Urine	Faeces
Organic solvent extracts	0.007 (55.94)	0.733 (75.57)	0.773 (79.74)	6.279 (63.30)	5.067 (30.49)	(82.35)	(46.39)
Glucuronidase and sulfatase treatment	n.a.*	n.a.	n.a.	0.105 (1.06)	0.128 (0.77)	n.a.	n.a.
Ethyl acetate extracts from acid and base released (1N and/or 5N)	0.098 (10.57)	0.068 (7.01)	0.011 (1.12)	2.970 (29.94)	10.298 (61.96)	n.a.	(37.43)
Aqueous and/or after ethyl acetate partitioning of hydrolysates	0.052 (5.54)	0.065 (6.66)	0.047 (4.86)	0.346 (3.49)	1.080 (6.50)	6.71	(11.16)
Fat and protein	0.082 (8.69)	n.a.	0.054 (5.62)	n.a.	n.a.	(9.89)	n.a.
Unextractables	0.054 (5.70)	0.040 (4.17)	0.051 (5.23)	0.909 (9.16)	0.515 (3.10)	(0.69)	(9.84)
Total	0.903 (95.99)	0.906 (93.41)	0.937 (96.57)	10.61 (106.96)	16.57 (102.82)	(99.64)	(104.82)

*) n.a. = not applicable

Characterisation and identification:

In liver, the metabolites ≥ 0.05 mg eq/kg were M22 (dimer), M17 (mercapturate conjugate), M24 (glutathione conjugate) and M25 (cysteine conjugate). In kidney, metabolites M7 (O-demethylated parent), M17, M24 and M25 were above 0.05 mg eq/kg (see Table B.7.2-31). The analysis of milk indicated M17, M24 and M25 levels were about 0.05 to 0.1 mg eq/kg. For muscle, the metabolites above 0.05 mg eq/kg were: M17, M24 and M25 (about 0.08 to 0.14 mg eq/kg). Metabolites in fat above 0.05 mg eq/kg were M7 and M17 (about 0.05 and 0.24 mg eq/kg). In urine and faeces, metabolites M3 (reductive dechlorinated), M7, M17, M24, M25, and M31 (sulfoxide of thioglycolic acid) were identified (Table B.7.2-31).

Table B.7.2-31: Summary of metabolites in kidney, liver, and excreta of goat

Metabolite	Kidney		Liver		Urine	Faeces
	[mg eq/kg]	%TRR	[mg eq/kg]	%TRR	[mg eq/kg]	[mg eq/kg]
M3	ND	ND	ND	ND	1.83	4.65
M7	2.388	24.1	ND	ND	1.53	15.48
M17	0.889	9.0	0.445	2.7	3.91	5.01
M22	ND	ND	1.016	6.1	ND	ND
M24	0.516	5.2	0.366	2.2	6.70	2.00
M25	0.122	1.23	1.198	7.2	24.62	4.97
M31	ND	ND	ND	ND	2.44	2.93

*) ND = not detected

Proposed metabolic pathway:

Dimethenamid was rapidly and extensively metabolised in the goat. The major metabolic pathway was through glutathione conjugation, followed by the formations of cysteine, mercapturate, sulfoxide of thioglycolic acid conjugations, and dimerization of a mercaptan intermediate (Figure B.7.2-13). The other pathways included O-demethylation and reductive dechlorination.

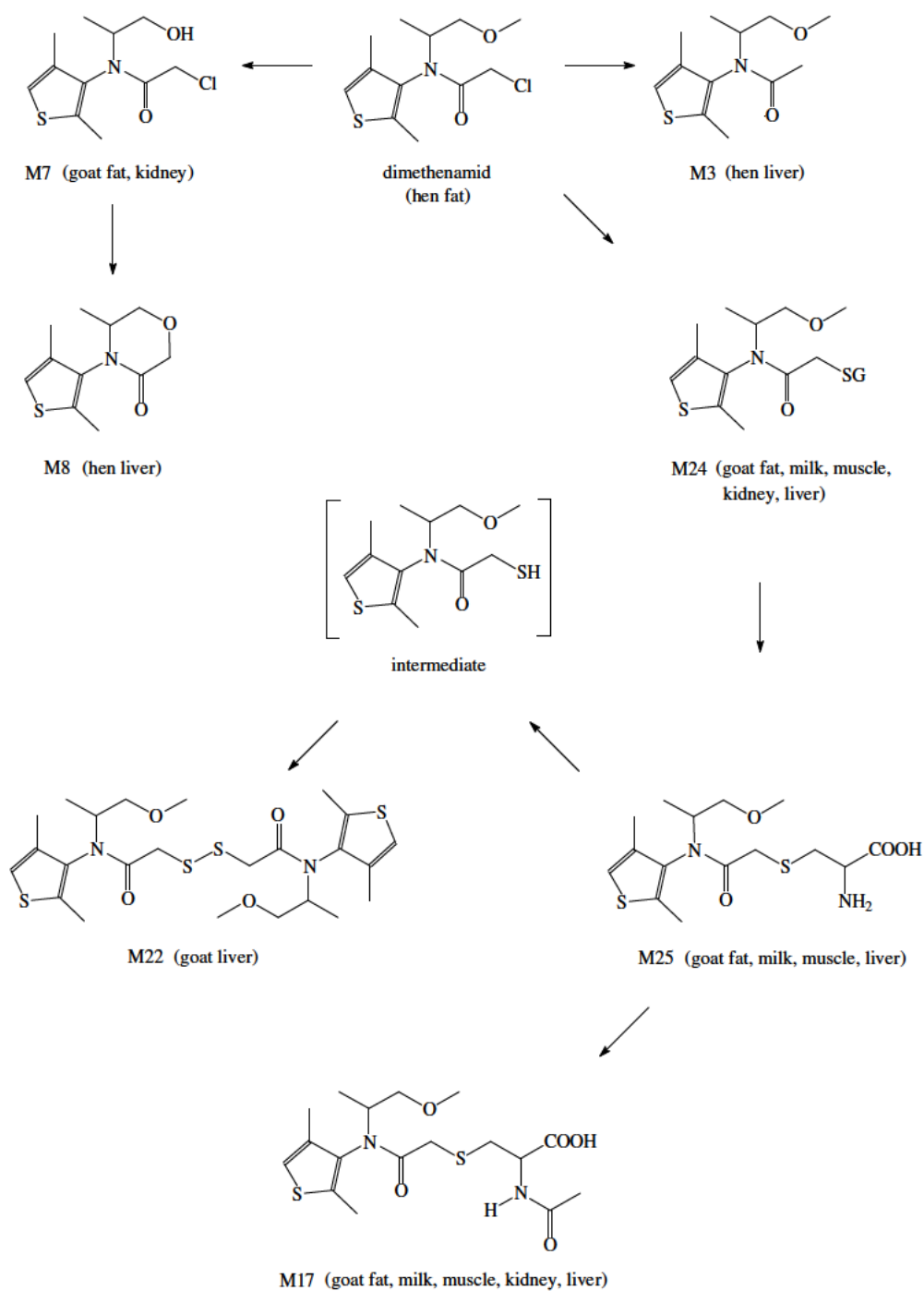
Conclusion

This study was conducted using racemic dimethenamid. Consequently, the metabolism of dimethenamid-P was included since the racemic mixture contained 50 % of the R-enantiomer (dimethenamid-P). Up to 87 % TRR was excreted via faeces and urine. Residues equal to 0.022 % TRR were found in milk, while residues in edible tissues ranged between 0.05 % TRR in fat and 1.36 % TRR in muscle. Identified metabolites in milk, liver, kidney and muscle were M17, M24 and M25. Additionally metabolites M7 and M22 were identified in kidney and liver, respectively.

As the study was performed with the parent dimethenamid, which was not detected in any of the plant metabolism studies, it does not reflect the actual exposure scenario when ruminants are fed with feed treated with dimethenamid. Since with the study by [REDACTED] [ASB2014-8342](#)) an acceptable goat metabolism performed with metabolite M30 was available, this study is only considered as supplementary.

Note:

It is noted by the RMS that after oral dimethenamid treatment, the goat exhibited signs of toxicity (pronounced decrease of water intake, decreased feed intake and body weight). The applicant explained that oral administration of undiluted dimethenamid has been shown to result in temporarily decreased feed intake and body weight loss in other animal studies; therefore the possibility of substance-related toxicity could not be excluded. The applicant pointed out that for reasons of metabolite identification, the administration of a lower dose level was not deemed feasible. The RMS also notes that, compared with the follow-up experiment, a very low overall recovery in urine, faeces and tissues was established in the main study, which could only partly be explained by the loss of a urine sample, since the faecal sample was considerably reduced, too. Moreover, since results from metabolism studies in plant and from field trials showed no residue of the parent dimethenamid-P at all, it can be assumed that animals will not be exposed to parent dimethenamid-P.



*) excreta not included

Figure B.7.2-13: Metabolic pathway of dimethenamid in lactating goat and laying hen*

B.7.2.4 Pigs

No metabolism study in pigs was performed, since the metabolite patterns in rodents (rats) and ruminants (goat) did not differ significantly.

B.7.2.5 Fish

As the indented uses comprises crops that can be considered as feed items for fish (e.g. maize grain, soya bean seed and sunflower seed as well by products such as meals of maize, oilseed rape and soya bean), a metabolism study would be required according to Commission Regulation 283/2013. However, no residues of parent dimethenamid-P or its metabolites were detected in commodities with a potential use as fish feed. Additionally, according to SANCO/11187/2013 rev. 3, a fish metabolism study is required only if the log P_{ow} is ≥ 3 . For dimethenamid-P the log P_{ow} is 1.98 and therefore accumulation can be excluded. Consequently no fish metabolism study is necessary.

B.7.3 Magnitude of residue trials in plants

B.7.3.1 Representative uses

New trials were provided for maize, soya bean, sunflower, sugar beet and oilseed rape for N-EU and S-EU. Trials evaluated for the first approval were not considered in the renewal as those were only performed with racemic dimethenamid. Metabolites were not considered in these trials.

B.7.3.1.1 Maize

The cGAP in N- and S-EU is 1 x 0.864 kg as/ha spray application at early post-emergence stage up to BBCH 16. PHI is covered by the growing period between application and harvest.

Reference:	KCA 6.3.4/2
Report	Perny A. (2013): Study on the residue behaviour of BAS 656 H (dimethenamid-P) after treatment with BAS 656 12 H in corn under field conditions in Northern and Southern Europe, 2011, BASF Doc ID 2012/1209625, Study code: 390501, ASB2014-3704
Guideline(s):	EEC 1607/VI/97 rev. 2 10.06.1999, EEC 91/414 Annex II (Part A Section 6), EEC 91/414 Annex III (Part A Section 8), EEC 7029/VI/95 rev. 5 Appendix B, EEC 7525/VI/95 rev. 7
Deviations:	None
GLP:	Yes
Acceptability:	The study is acceptable.

Reference:	KCA 6.3.4/1
Report	Gabriel E.J., Meyer M., (2013): Study on the residue behaviour of dimethenamid-P (BAS 656 H) in maize after treatment with BAS 656 12 H under field conditions in Germany, Northern France, United Kingdom, the Netherlands, Southern France, Greece, Italy and Spain, 2012, BASF Doc ID 2012/1272621, Study code: 423130, ASB2014-10046

Guideline(s):	EC 1107/2009 of the European Parliament and of the Council of 21 Oct 2009, EEC 79/117, EEC 91/414 Annex II (Part A Section 6), EEC 91/414 Annex III (Part A Section 8), EEC 1607/VI/97 rev. 2 10.06.1999, EEC 7029/VI/95 rev. 5, EEC 7525/VI/95 rev. 9 (March 2011)
Deviations:	None
GLP:	Yes
Acceptability:	The study is acceptable.

Materials and methods

Out of 24 supervised residue trials in maize, 12 trials were conducted in N-EU and 12 trials in S-EU during 2011 and 2012.

Maize plants were treated according to GAP with 1 applications at nominal rates of 0.847-1.058 kg as/ha. Grain and forage were harvested at a PHI of 125-149 and 59-115 days, respectively. The samples were stored deep frozen for max. 11 months.

All samples were analysed for dimethenamid-P and metabolites M23, M26, M27 and M30 according to method BASF L0179/02. For a detailed method description see B.5.2.1.1.

Results and Discussion

Procedural recoveries and precisions were within acceptable limits (Table B.7.3-1), except for the RSD for M27 in whole plant at the 10xLOQ level. Storage of samples with high starch content was covered by acceptable storage stability studies for all analytes, except for metabolite M27. Storage stability of high water content samples was validated for dimethenamid-P, M23 and M30, but not for M26. Residue levels in individual samples are reported in Table B.7.3-2 and Table B.7.3-3. No residues were found in untreated control samples.

Table B.7.3-1: Procedural recoveries for dimethenamid-P in maize

Matrix	Fortification Level [mg/kg]	Summary recoveries							
		Recovery [%]	Mean [%]	RSD [%]	n	Recovery [%]	Mean [%]	RSD [%]	n
		Dimethenamid-p				M23			
Whole plant	0.01	77, 88, 88	84	8	3	78, 108, 101	96	16	3
	0.1	79	79	-	1	74	74	-	1
	10	73, 98, 98	90	16	3	72, 107, 97	92	20	3
Cob with husks	0.01	101, 112, 96, 79	97	14	4	109, 112, 95, 92	102	10	4
	0.1	101, 108, 99, 85	98	10	4	96, 97, 98, 92	96	3	4
Rest of plant	0.01	102, 103, 106, 107, 95, 102, 99, 103	102	4	8	98, 101, 107, 107, 95, 99, 109, 104	103	5	8
	0.1	104, 105, 106, 106, 99, 100, 101, 103	103	3	8	101, 102, 104, 109, 96, 102, 108, 100	103	4	8
	1	103, 106, 107	105	2	3	102, 103, 109	105	4	3
	45	99	99	-	1	93	93	-	1
Grain	0.01	108, 98	103	-	2	100, 100	100	-	2
	0.1	102, 99	101	-	2	94, 95	95	-	2
		M26				M27			
Whole plant	0.01	77, 102, 91	90	14	3	76, 108, 96	93	17	3
	0.1	77	77	-	1	72	72	-	1
	10	72, 101, 99	91	18	3	68, 110, 100	93	24	3
Cob with husks	0.01	101, 112, 90, 86	97	12	4	106, 107, 81, 82	94	15	4
	0.1	100, 99, 96, 82	94	9	4	105, 99, 95, 91	98	6	4
Rest of plant	0.01	100, 99, 98, 99, 94, 92, 99, 95	97	3	8	101, 105, 103, 103, 74, 102, 99, 108	99	11	8
	0.1	101, 103, 107, 107, 90, 97, 105, 94	101	6	8	100, 97, 103, 103, 79, 104, 102, 100	99	8	8
	1	105, 102, 109	105	3	3	101, 101, 104	102	2	3
	45	95	95	-	1	92	92	-	1
Grain	0.01	99, 90	95	-	2	98, 92	95	-	2
	0.1	95, 95	95	-	2	93, 93	93	-	2
		M30							
Whole plant	0.01	67, 90, 98	85	19	3				
	0.1	70	70	-	1				
	10	65, 94, 94	84	20	3				
Cob with husks	0.01	99, 107, 102, 75	96	15	4				
	0.1	98, 101, 93, 82	94	9	4				
Rest of plant	0.01	100, 92, 95, 107, 81, 110, 104, 83	97	11	8				
	0.1	99, 95, 97, 95, 98, 103, 105, 103	99	4	8				
	1	99, 97, 99	98	1	3				
	45	87	87	-	1				
Grain	0.01	100, 98	99	-	2				
	0.1	90, 97	94	-	2				

Conclusion

Of the 24 trials submitted for N-EU and S-EU, 16 were considered acceptable. Eight trials were performed at the same site, but at different plots. On these sites one plot was treated with an application rate according to the cGAP (0.864 kg as/ha) and the other plot using a higher application rate (1.008 kg as/ha). Here, the higher dosed trials were chosen for risk assessment, as they are within ± 25 % of the cGAP and therefore considered more critical. For metabolite M26 the storage stability in high water content commodities could not be validated as maximum storage stability was experimentally determined equal to 3 months (see Table B.7.1-1). Metabolite M27 was found to be instable in high starch commodities, but was not considered in the residue definition anyway.

1	2	3	4			5	6	7	8.1	8.2	8.3	8.4	8.5	9	10
Report-No. Location incl. Postal code and date	Commodity/ Variety	Date of 1) Sowing or planting 2) Flowering 3) Harvest	Application rate per treatment			Dates of treatments or no. of treatments and last date	Growth stage at last treatment or date	Portion analysed	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	PHI (days)	Remarks
	(a)	(b)	kg as/ha	Water L/ha	kg as/hL	(c)		(a)						(d)	(e)
		- 2011-07-22 3) 2011-10-06						rest of plant without root	<0.010 <0.010 <0.010	<0.010 <0.010 <0.010	<0.010 <0.010 <0.010	<0.010 <0.010 <0.010	0.030 0.020 0.020	60 79 88	validated 8.3: rest of plant without root, whole plant 8.4: maize cob with husks
								maize cob with husks	<0.010 <0.010 <0.010	<0.010 <0.010 <0.010	<0.010 <0.010 <0.010	<0.010 <0.010 <0.010	<0.010 <0.010 <0.010	60 79 88	Analytical method: BASF No. L0179/02 (HPLC-MS/MS), LOQ(s): 0.01 mg/kg Max. sample storage time in month(s): 8 (high water content) 6 (high starch content) ASB2014-3704
report 2012/1209625, study 390501 (B1017), trial L110042 (B1017 AN1), plot 2 and 3, decline trial France (FR) 67160 Seebach 2013-02-28	Koherence	1) 2011-04-20 (sowing) 2) 2011-07-07 - 2011-07-18 3) 2011-10-03	0.98	190	0.51	2011-05-23 ⁴⁾	BBCH 16-17	whole plant	34.4	<0.010	0.030	<0.010	<0.010	0	4) spraying
								rest of plant without root	<0.010 <0.010 <0.010	<0.010 <0.010 <0.010	<0.010 <0.010 <0.010	<0.010 <0.010 <0.010	<0.010 <0.010 <0.010	59 93 128	Sample storage time not validated 8.3: rest of plant without root, whole plant 8.4: maize cob with husks, grain
								maize cob with husks	<0.010 <0.010	<0.010 <0.010	<0.010 <0.010	<0.010 <0.010	<0.010 <0.010	59 93	Analytical method: BASF No. L0179/02 (HPLC-MS/MS), LOQ(s): 0.01 mg/kg
								grain	<0.010	<0.010	<0.010	<0.010	<0.010	128	Max. sample storage time in month(s): 9 (high water content) 6 (high starch content) ASB2014-3704
	Koherence	1) 2011-04-20 (sowing) 2) 2011-07-07	0.89	210	0.43	2011-05-23 ⁴⁾	BBCH 16-17	whole plant	15.2	<0.010	0.020	<0.010	<0.010	0	4) spraying Sample storage time not

1	2	3	4			5	6	7	8.1	8.2	8.3	8.4	8.5	9	10
Report-No. Location incl. Postal code and date	Commodity/ Variety	Date of 1) Sowing or planting 2) Flowering 3) Harvest	Application rate per treatment			Dates of treatments or no. of treatments and last date	Growth stage at last treatment or date	Portion analysed	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	PHI (days)	Remarks
			kg as/ha	Water L/ha	kg as/hL										
	(a)	(b)				(c)		(a)						(d)	(e)
		- 2011-07-18 3) 2011-10-03						rest of plant without root	<0.010 <0.010 <0.010	<0.010 <0.010 <0.010	<0.010 <0.010 <0.010	<0.010 <0.010 <0.010	<0.010 <0.010 <0.010	59 93 128	validated 8.3: rest of plant without root, whole plant 8.4: maize cob with husks, grain
								maize cob with husks	<0.010 <0.010	<0.010 <0.010	<0.010 <0.010	<0.010 <0.010	<0.010 <0.010	59 93	Analytical method: BASF No. L0179/02 (HPLC-MS/MS), LOQ(s): 0.01 mg/kg
								grain	<0.010	<0.010	<0.010	<0.010	<0.010	128	Max. sample storage time in month(s): 9 (high water content) 6 (high starch content) ASB2014-3704
report 2012/1209625, study 390501 (B1017), trial L110043 (B1017 UK1), plot 2 and 3, decline trial United Kingdom (UK) OX279AS Stratton Audley 2013-02-28	Podium	1) 2011-04-29 (sowing) 2) 2011-07-28 - 2011-08-11 3) 2011-10-10	1.0	200	0.49	2011-06-06 ⁴⁾	BBCH 16	whole plant	64.4 <0.010	<0.010 <0.010	<0.010 <0.010	<0.010 <0.010	<0.010 <0.010	0 60	4) spraying Sample storage time not validated 8.3: rest of plant without root, whole plant 8.4: grain, maize cob with husks
								rest of plant without root	<0.010 <0.010 <0.010	<0.010 <0.010 <0.010	<0.010 <0.010 <0.010	<0.010 <0.010 <0.010	<0.010 <0.010 <0.010	91 120 142	Analytical method: BASF No. L0179/02 (HPLC-MS/MS), LOQ(s): 0.01 mg/kg
								maize cob with husks	<0.010 <0.010	<0.010 <0.010	<0.010 <0.010	<0.010 <0.010	<0.010 <0.010	91 120	Max. sample storage time in month(s): 8 (high water content) 5 (high starch content) ASB2014-3704
								grain	<0.010	<0.010	<0.010	<0.010	<0.010	142	
	Podium	1) 2011-04-29 (sowing)	0.88	200	0.43	2011-06-06 ⁴⁾	BBCH 16	whole plant	50.0 <0.010	<0.010 <0.010	<0.010 <0.010	<0.010 <0.010	<0.010 <0.010	0 60	4) spraying

1	2	3	4			5	6	7	8.1	8.2	8.3	8.4	8.5	9	10
Report-No. Location incl. Postal code and date	Commodity/ Variety	Date of 1) Sowing or planting 2) Flowering 3) Harvest	Application rate per treatment			Dates of treatments or no. of treatments and last date	Growth stage at last treatment or date	Portion analysed	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	PHI (days)	Remarks
			kg as/ha	Water L/ha	kg as/hL										
	(a)	(b)				(c)		(a)						(d)	(e)
		2) 2011-07-28 - 2011-08-11 3) 2011-10-10						rest of plant without root	<0.010 <0.010 <0.010	<0.010 <0.010 <0.010	<0.010 <0.010 <0.010	<0.010 <0.010 <0.010	<0.010 <0.010 <0.010	91 120 142	Sample storage time not validated 8.3: rest of plant without root, whole plant 8.4: maize cob with husks, grain
								maize cob with husks	<0.010 <0.010	<0.010 <0.010	<0.010 <0.010	<0.010 <0.010	<0.010 <0.010	91 120	Analytical method: BASF No. L0179/02 (HPLC-MS/MS), LOQ(s): 0.01 mg/kg,
								grain	<0.010	<0.010	<0.010	<0.010	<0.010	142	Max. sample storage time in month(s): 8 (high water content) 5 (high starch content) ASB2014-3704
report 2012/1209625, study 390501 (B1017), trial L110044 (B1017 NL1), plot 2 and 3, decline trial Netherlands (NL) 5856 AB Wellerlooi, Gennep 2013-02-28	Fallove	1) 2011-05-13 (sowing) 2) 2011-07-18 - 2011-08-01 3) 2011-10-14	1.0	200	0.49	2011-06-08 ⁴⁾	BBCH 16	whole plant	36.4 <0.010	<0.010 <0.010	0.020 <0.010	<0.010 <0.010	<0.010 <0.010	0 61	4) spraying
								rest of plant without root	<0.010 <0.010 <0.010	<0.010 <0.010 <0.010	<0.010 <0.010 <0.010	<0.010 <0.010 <0.010	<0.010 <0.010 <0.010	92 110 128	Sample storage time not validated 8.3: rest of plant without root, whole plant 8.4: grain, maize cob with husks
								maize cob with husks	<0.010 <0.010	<0.010 <0.010	<0.010 <0.010	<0.010 <0.010	<0.010 <0.010	92 110	Analytical method: BASF No. L0179/02 (HPLC-MS/MS), LOQ(s): 0.01 mg/kg
								grain	<0.010	<0.010	<0.010	<0.010	<0.010	128	Max. sample storage time in month(s): 8 (high water content) 5 (high starch content) ASB2014-3704

1	2	3	4			5	6	7	8.1	8.2	8.3	8.4	8.5	9	10
Report-No. Location incl. Postal code and date	Commodity/ Variety	Date of 1) Sowing or planting 2) Flowering 3) Harvest	Application rate per treatment			Dates of treatments or no. of treatments and last date	Growth stage at last treatment or date	Portion analysed	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	PHI (days)	Remarks
			kg as/ha	Water L/ha	kg as/hL										
	(a)	(b)				(c)		(a)						(d)	(e)
	Fallove	1) 2011-05-13 (sowing) 2) 2011-07-18 - 2011-08-01 3) 2011-10-14	0.87	200	0.43	2011-06-08 ⁴⁾	BBCH 16	whole plant	23.6 <0.010	<0.010 <0.010	0.040 <0.010	<0.010 <0.010	<0.010 <0.010	0 61	4) spraying
								rest of plant without root	<0.010 <0.010 <0.010	<0.010 <0.010 <0.010	<0.010 <0.010 <0.010	<0.010 <0.010 <0.010	<0.010 <0.010 <0.010	92 110 128	Sample storage time not validated 8.3: rest of plant without root, whole plant 8.4: grain, maize cob with husks
								maize cob with husks	<0.010 <0.010	<0.010 <0.010	<0.010 <0.010	<0.010 <0.010	<0.010 <0.010	92 110	Analytical method: BASF No. L0179/02 (HPLC-MS/MS), LOQ(s): 0.01 mg/kg
								grain	<0.010	<0.010	<0.010	<0.010	<0.010	128	Max. sample storage time in month(s): 8 (high water content) 5 (high starch content)
															ASB2014-3704

RESIDUES DATA SUMMARY FROM SUPERVISED TRIALS (SUMMARY)
(Application on agricultural and horticultural crops)

Federal Institute for Risk Assessment, Berlin
Federal Republic of Germany

Content of a.s. (g/kg or g/L) : 720 g/L
Formulation (e.g. WP) : EC (emulsifiable concentrate)
Commercial product (name) : BAS 656 12 H
Applicant : BASF SE

Active substance : Dimethenamid-P
Crop / crop group : Maize
Crop Code : ZEAMX

Submission date : 2014-08-21

Indoors / Outdoors : Outdoors (European North)

Other a.s. in formulation
(content and common name) :

Residues calculated as : 8.1 dimethenamid-P
8.2 M23 oxalamide
8.3 M26 thiolactic acid conjugate
8.4 M27 sulfonate
8.5 M30 sulfoxide of thiolactic acid conjugate

1	2	3	4			5	6	7	8.1	8.2	8.3	8.4	8.5	9	10
Report-No. Location incl. Postal code and date	Commodity/ Variety	Date of 1) Sowing or planting 2) Flowering 3) Harvest	Application rate per treatment			Dates of treatments or no. of treatments and last date	Growth stage at last treatment or date	Portion analysed	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	PHI (days)	Remarks
	(a)	(b)	kg as/ha	Water L/ha	kg as/hL	(c)		(a)						(d)	(e)
BASF DocID 2012/1272621, study 423130 (IF-12/02223803), trial L120284, decline trial Germany (DE) 49685 Garthe 2013-11-19	Aurelia	1) 2012-05-02 (sowing) 2) 2012-07-20 - 2012-08-07 3) 2012-10-23	0.91	210	0.43	2012-06-21 ⁴⁾	BBCH 16	whole plant	46.0	<0.010	0.044	<0.010	<0.010	0	4) spraying
								whole plant, milk stage	<0.010	<0.010	<0.010	<0.010	0.031	60	Sample storage time not validated 8.3: rest of plant, silage stage, whole plant, whole plant, milk stage, rest of plant
								rest of plant	<0.010 <0.010	<0.010 <0.010	<0.010 <0.010	<0.010 <0.010	0.021 <0.010	91 125	8.4: grain, maize cob with husks, silage stage, maize cob with husks
								rest of plant, silage stage	<0.010	<0.010	<0.010	<0.010	<0.010	111	Analytical method: BASF No. L0179/02 (HPLC-MS/MS), LOQ(s): 0.01 mg/kg
								maize cob with husks	<0.010	<0.010	<0.010	<0.010	<0.010	91	Max. sample storage time in month(s): 10 (high water content) 7 (high starch content)
								maize cob with husks, silage stage	<0.010	<0.010	<0.010	<0.010	<0.010	111	
								grain	<0.010	<0.010	<0.010	<0.010	<0.010	125	ASB2014-10046

1	2	3	4			5	6	7	8.1	8.2	8.3	8.4	8.5	9	10
Report-No. Location incl. Postal code and date	Commodity/ Variety	Date of 1) Sowing or planting 2) Flowering 3) Harvest	Application rate per treatment			Dates of treatments or no. of treatments and last date	Growth stage at last treatment or date	Portion analysed	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	PHI (days)	Remarks
			kg as/ha	Water L/ha	kg as/hL										
(a)	(a)	(b)				(c)		(a)						(d)	(e)
BASF DocID 2012/1272621, study 423130 (IF-12/02223803), trial L120285, decline trial France (FR) 37380 Reugny 2013-11-19	DKC 3850	1) 2012-04-01 (sowing) 2) 2012-07-15 - 2012-08-15 3) 2012-11-06 - 2012-11-08	0.91	210	0.43	2012-06-12 ⁴⁾	BBCH 16	whole plant rest of plant rest of plant, silage stage maize cob with husks, milk stage maize cob with husks, silage stage grain	0.77 <0.010 <0.010 <0.010 <0.010 <0.010 <0.010	<0.010 <0.010 <0.010 <0.010 <0.010 <0.010	<0.010 <0.010 <0.010 <0.010 <0.010 <0.010	<0.010 <0.010 <0.010 <0.010 <0.010 <0.010	<0.010 <0.010 <0.010 <0.010 <0.010 <0.010	0 59 149 92 59 92 149	4) spraying Sample storage time not validated 8.3: rest of plant, whole plant, rest of plant, silage stage 8.4: maize cob with husks, milk stage, maize cob with husks, silage stage, grain Analytical method: BASF No. L0179/02 (HPLC-MS/MS), LOQ(s): 0.01 mg/kg Max. sample storage time in month(s): 10 (high water content) 7 (high starch content) ASB2014-10046
BASF DocID 2012/1272621, study 423130 (IF-12/02223803), trial L120286, decline trial United Kingdom (UK) OX27 75L Tusmore, Oxfordshire 2013-11-19	Kougar	1) 2012-04-14 (sowing) 2) 2012-08-10 - 2012-08-24 3) 2012-10-26	0.89	210	0.43	2012-06-10 ⁴⁾	BBCH 16	whole plant rest of plant rest of plant, silage stage maize cob with husks, silage stage	36.0 <0.010 <0.010 <0.010 <0.010	<0.010 <0.010 <0.010 <0.010 <0.010	0.011 <0.010 <0.010 <0.010 <0.010	<0.010 <0.010 <0.010 <0.010 <0.010	<0.010 <0.010 <0.010 <0.010 <0.010	0 61 138 91 91	4) spraying Sample storage time not validated 8.3: whole plant, rest of plant, rest of plant, silage stage 8.4: maize cob with husks, silage stage, grain Analytical method: BASF No. L0179/02 (HPLC-MS/MS), LOQ(s): 0.01 mg/kg Max. sample storage time

1	2	3	4			5	6	7	8.1	8.2	8.3	8.4	8.5	9	10
Report-No. Location incl. Postal code and date	Commodity/ Variety	Date of 1) Sowing or planting 2) Flowering 3) Harvest	Application rate per treatment			Dates of treatments or no. of treatments and last date	Growth stage at last treatment or date	Portion analysed	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	PHI (days)	Remarks
	(a)	(b)	kg as/ha	Water L/ha	kg as/hL	(c)		(a)						(d)	(e)
								grain	<u><0.010</u>	<0.010	<u><0.010</u>	<0.010	<u><0.010</u>	138	in month(s): 10 (high water content) 7 (high starch content) ASB2014-10046
BASF DocID 2012/1272621, study 423130 (IF-12/02223803), trial L120287, decline trial Netherlands (NL) 9541 XH Vlagdwedde 2013-11-19	P 8057 (Pioneer)	1) 2012-05-09 (sowing) 2) 2012-07-30 - 2012-08-17 3) 2012-10-15	0.82	190	0.43	2012-06-19 ⁴⁾	BBCH 16	whole plant	21.0 <0.010	<0.010 <0.010	0.060 <0.010	<0.010 <0.010	0.019 <0.010	0 59	4) spraying Sample storage time not validated 8.3: rest of plant, rest of plant, silage stage, whole plant 8.4: maize cob with husks, silage stage, grain, maize cob with husks, milk stage Analytical method: BASF No. L0179/02 (HPLC-MS/MS), LOQ(s): 0.01 mg/kg Max. sample storage time in month(s): 10 (high water content) 7 (high starch content) ASB2014-10046
								rest of plant	<0.010 <0.010	<0.010 <0.010	<0.010 <0.010	<0.010 <0.010	<0.010 <0.010	91 140	
								rest of plant, silage stage	<u><0.010</u>	<0.010	<u><0.010</u>	<0.010	<u><0.010</u>	115	
								maize cob with husks, milk stage	<0.010	<0.010	<0.010	<0.010	<0.010	91	
								maize cob with husks, silage stage	<u><0.010</u>	<0.010	<u><0.010</u>	<0.010	<u><0.010</u>	115	
								grain	<u><0.010</u>	<0.010	<u><0.010</u>	<0.010	<u><0.010</u>	140	

Remarks:

- (a) According to CODEX Classification / Guide
- (b) Only if relevant
- (c) Year must be indicated
- (d) Days after last application (Label pre-harvest interval, PHI, underline)
- (e) Remarks may include: Climatic conditions; Reference to analytical method and information which metabolites are included

Table B.7.3-3: S-EU residues trials with maize conducted according to GAP

RESIDUES DATA SUMMARY FROM SUPERVISED TRIALS (SUMMARY)
(Application on agricultural and horticultural crops)

Federal Institute for Risk Assessment, Berlin
Federal Republic of Germany

Content of a.s. (g/kg or g/L) : 720 g/L
Formulation (e.g. WP) : EC (emulsifiable concentrate)
Commercial product (name) : BAS 656 12 H
Applicant : BASF SE

Active substance : Dimethenamid-P
Crop / crop group : Maize
Crop Code : ZEAMX

Submission date : 2014-03-19

Indoors / Outdoors : Outdoors (European South)
Other a.s. in formulation :
(content and common name) :
Residues calculated as : 8.1 dimethenamid-P
8.2 M23 oxalamide
8.3 M26 thiolactic acid conjugate
8.4 M27 sulfonate
8.5 M30 sulfoxide of thiolactic acid conjugate

1	2	3	4			5	6	7	8.1	8.2	8.3	8.4	8.5	9	10
Report-No. Location incl. Postal code and date	Commodity/ Variety	Date of 1) Sowing or planting 2) Flowering 3) Harvest	Application rate per treatment			Dates of treatments or no. of treatments and last date	Growth stage at last treatment or date	Portion analysed	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	PHI (days)	Remarks
			kg as/ha	Water L/ha	kg as/hL										
	(a)	(b)				(c)		(a)						(d)	(e)
report 2012/1209625, study 390501 (B1017), trial L110045 (B1017 TL1), plot 2 and 3, decline trial France (FR) 31330 Grenade Sur Garonne 2013-02-28	33Y74	1) 2011-04-25 (sowing) 2) 2011-07-05 - 2011-07-12 3) 2011-10-10 - 2011-10-12	1.1	210	0.53	2011-05-27 ⁴⁾	BBCH 16	whole plant	21.9	<0.010	0.32	<0.010	0.090	0	4) spraying
								rest of plant	<0.010	<0.010	<0.010	<0.010	<0.010	61	Sample storage time not validated
								plant without	<0.010	<0.010	<0.010	<0.010	<0.010	76	8.3: whole plant, rest of
								root	<0.010	<0.010	<0.010	<0.010	<0.010	91	plant without root
								maize cob with	<0.010	<0.010	<0.010	<0.010	<0.010	138	8.4: grain, maize cob with
								husks	<0.010	<0.010	<0.010	<0.010	<0.010	61	husks
								grain	<0.010	<0.010	<0.010	<0.010	<0.010	76	Analytical method: BASF No. L0179/02 (HPLC-MS/MS), LOQ(s): 0.01 mg/kg
	33Y74	1) 2011-04-25 (sowing)	0.94	220	0.43	2011-05-27 ⁴⁾	BBCH 16	controls > LOQ whole plant	0.030					91	Max. sample storage time in month(s): 9 (high water content) 7 (high starch content) ASB2014-3704
								whole plant	19.8	<0.010	0.40	<0.010	0.13	0	4) spraying

1	2	3	4			5	6	7	8.1	8.2	8.3	8.4	8.5	9	10
Report-No. Location incl. Postal code and date	Commodity/ Variety	Date of 1) Sowing or planting 2) Flowering 3) Harvest	Application rate per treatment			Dates of treatments or no. of treatments and last date	Growth stage at last treatment or date	Portion analysed	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	PHI (days)	Remarks
			kg as/ha	Water L/ha	kg as/hL										
(a)	(a)	(b)				(c)		(a)						(d)	(e)
		2) 2011-07-05 - 2011-07-12 3) 2011-10-10 - 2011-10-12						rest of plant without root maize cob with husks grain controls > LOQ whole plant	<0.010 <0.010 <0.010 <0.010 <0.010 <0.010 <0.010 0.030	<0.010 <0.010 <0.010 <0.010 <0.010 <0.010 <0.010 	<0.010 <0.010 <0.010 <0.010 <0.010 <0.010 <0.010 	<0.010 <0.010 <0.010 <0.010 <0.010 <0.010 <0.010 	<0.010 <0.010 <0.010 <0.010 <0.010 <0.010 <0.010 	61 76 91 138 61 76 91 138 0	Sample storage time not validated 8.3: rest of plant without root, whole plant 8.4: grain, maize cob with husks Analytical method: BASF No. L0179/02 (HPLC-MS/MS), LOQ(s): 0.01 mg/kg Max. sample storage time in month(s): 9 (high water content) 7 (high starch content) ASB2014-3704
report 2012/1209625, study 390501 (B1017), trial L110046 (B1017 GR1), plot 2 and 3, decline trial Greece (GR) 59100 Kavasila 2013-02-28	Sansia	1) 2011-05-25 (sowing) 2) 3) 2011-10-12	0.99	200	0.50	2011-07-12 ⁴⁾	BBCH 16	whole plant rest of plant without root maize cob with husks grain	14.4 <u><0.010</u> <0.010 <0.010 <u><0.010</u>	<0.010 <0.010 <0.010 <0.010 <0.010	0.14 <u>0.22</u> 0.010 <0.010 <u><0.010</u>	<0.010 <0.010 <0.010 <0.010 <0.010	0.020 <u>1.1</u> 0.16 <0.010 <u><0.010</u>	0 59 92 59 92	4) spraying Sample storage time not validated 8.3: whole plant, rest of plant without root 8.4: grain, maize cob with husks Analytical method: BASF No. L0179/02 (HPLC-MS/MS), LOQ(s): 0.01 mg/kg Max. sample storage time in month(s): 9 (high water content) 6 (high starch content) ASB2014-3704

1	2	3	4			5	6	7	8.1	8.2	8.3	8.4	8.5	9	10
Report-No. Location incl. Postal code and date	Commodity/ Variety	Date of 1) Sowing or planting 2) Flowering 3) Harvest	Application rate per treatment			Dates of treatments or no. of treatments and last date	Growth stage at last treatment or date	Portion analysed	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	PHI (days)	Remarks
			kg as/ha	Water L/ha	kg as/hL										
	(a)	(b)				(c)		(a)						(d)	(e)
	Sansia	1) 2011-05-25 (sowing) 2) 3) 2011-10-12	0.89	210	0.43	2011-07-12 ⁴⁾	BBCH 16	whole plant	16.7	<0.010	0.19	<0.010	0.020	0	4) spraying
								rest of plant without root	<0.010 <0.010	<0.010 <0.010	0.050 0.010	<0.010 <0.010	0.28 0.060	59 92	Sample storage time not validated 8.3: rest of plant without root, whole plant 8.4: grain, maize cob with husks
								maize cob with husks	<0.010	<0.010	<0.010	<0.010	<0.010	59	Analytical method: BASF No. L0179/02 (HPLC-MS/MS), LOQ(s): 0.01 mg/kg
								grain	<0.010	<0.010	<0.010	<0.010	<0.010	92	Max. sample storage time in month(s): 7 (high water content) 5 (high starch content) ASB2014-3704
report 2012/1209625, study 390501 (B1017), trial L110047 (B1017 IT1), plot 2 and 3, decline trial Italy (IT) 27050 Corana 2013-02-28	PR32F73	1) 2011-04-07 (sowing) 2) 2011-06-15 - 2011-07-03 3) 2011-09-15 - 2011-09-16	1.0	200	0.50	2011-05-17 ⁴⁾	BBCH 16	whole plant	12.5	<0.010	0.080	<0.010	0.020	0	4) spraying
								rest of plant without root	<0.010 <0.010 <0.010 <0.010	<0.010 <0.010 <0.010 <0.010	<0.010 <0.010 0.010 <0.010	<0.010 <0.010 <0.010 <0.010	0.050 0.080 0.13 0.030	59 91 100 121	Sample storage time not validated 8.3: whole plant, rest of plant without root 8.4: maize cob with husks, grain
								maize cob with husks	<0.010 <0.010 <0.010	<0.010 <0.010 <0.010	<0.010 <0.010 <0.010	<0.010 <0.010 <0.010	<0.010 <0.010 <0.010	59 91 100	Analytical method: BASF No. L0179/02 (HPLC-MS/MS), LOQ(s): 0.01 mg/kg
								grain	<0.010	<0.010	<0.010	<0.010	<0.010	121	Max. sample storage time in month(s): 9 (high water content) 7 (high starch content) ASB2014-3704

1	2	3	4			5	6	7	8.1	8.2	8.3	8.4	8.5	9	10
Report-No. Location incl. Postal code and date	Commodity/ Variety	Date of 1) Sowing or planting 2) Flowering 3) Harvest	Application rate per treatment			Dates of treatments or no. of treatments and last date	Growth stage at last treatment or date	Portion analysed	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	PHI (days)	Remarks
			kg as/ha	Water L/ha	kg as/hL										
	(a)	(b)				(c)		(a)						(d)	(e)
	PR32F73	1) 2011-04-07 (sowing) 2) 2011-06-15 - 2011-07-03 3) 2011-09-15 - 2011-09-16	0.88	200	0.43	2011-05-17 ⁴⁾	BBCH 16	whole plant	5.0	<0.010	0.030	<0.010	<0.010	0	4) spraying
								rest of plant	<0.010	<0.010	<0.010	<0.010	0.030	59	Sample storage time not validated
								without root	<0.010	<0.010	<0.010	<0.010	0.030	91	8.3: whole plant, rest of plant without root
									<0.010	<0.010	<0.010	<0.010	0.080	100	8.4: maize cob with husks, grain
									<0.010	<0.010	<0.010	<0.010	<0.010	121	
								maize cob with husks	<0.010	<0.010	<0.010	<0.010	<0.010	59	Analytical method: BASF No. L0179/02 (HPLC-MS/MS), LOQ(s): 0.01 mg/kg

1	2	3	4			5	6	7	8.1	8.2	8.3	8.4	8.5	9	10
Report-No. Location incl. Postal code and date	Commodity/ Variety	Date of 1) Sowing or planting 2) Flowering 3) Harvest	Application rate per treatment			Dates of treatments or no. of treatments and last date	Growth stage at last treatment or date	Portion analysed	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	PHI (days)	Remarks
			kg as/ha	Water L/ha	kg as/hL										
	(a)	(b)				(c)		(a)						(d)	(e)
Montgri 2013-02-28								grain	<0.010	<0.010	<0.010	<0.010	<0.010	134	LOQ(s): 0.01 mg/kg Max. sample storage time in month(s): 9 (high water content) 7 (high starch content) ASB2014-3704
	N28	1) 2011-04-11 (sowing) 2) 2011-06 3) 2011-09-28	0.85	200	0.43	2011-05-17 ⁴⁾	BBCH 16	whole plant rest of plant without root maize cob with husks grain	6.2 <0.010 <0.010 <0.010 <0.010 <0.010 <0.010 <0.010 <0.010	<0.010 <0.010 <0.010 <0.010 <0.010 <0.010 <0.010 <0.010	0.020 <0.010 <0.010 <0.010 <0.010 <0.010 <0.010 <0.010	<0.010 <0.010 <0.010 <0.010 <0.010 <0.010 <0.010 <0.010	<0.010 0.030 <0.010 <0.010 <0.010 <0.010 <0.010 <0.010 <0.010	0 59 87 91 134 59 87 91 134	4) spraying Sample storage time not validated 8.3: rest of plant without root, whole plant 8.4: maize cob with husks, grain Analytical method: BASF No. L0179/02 (HPLC-MS/MS), LOQ(s): 0.01 mg/kg Max. sample storage time in month(s): 9 (high water content) 7 (high starch content) ASB2014-3704

RESIDUES DATA SUMMARY FROM SUPERVISED TRIALS (SUMMARY)
(Application on agricultural and horticultural crops)

Federal Institute for Risk Assessment, Berlin
Federal Republic of Germany

Content of a.s. (g/kg or g/L) : 720 g/L
Formulation (e.g. WP) : EC (emulsifiable concentrate)
Commercial product (name) : BAS 656 12 H
Applicant : BASF SE

Active substance : Dimethenamid-P
Crop / crop group : Maize
Crop Code : ZEAMX

Submission date : 2014-08-21

Indoors / Outdoors : Outdoors (European South)
Other a.s. in formulation :
(content and common name) :
Residues calculated as : 8.1 dimethenamid-P
8.2 M23 oxalamide
8.3 M26 thiolactic acid conjugate
8.4 M27 sulfonate
8.5 M30 sulfoxide of thiolactic acid conjugate

1	2	3	4			5	6	7	8.1	8.2	8.3	8.4	8.5	9	10
Report-No. Location incl. Postal code and date	Commodity/ Variety	Date of 1) Sowing or planting 2) Flowering 3) Harvest	Application rate per treatment			Dates of treatments or no. of treatments and last date	Growth stage at last treatment or date	Portion analysed	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	PHI (days)	Remarks
	(a)	(b)	kg as/ha	Water L/ha	kg as/hL	(c)		(a)						(d)	(e)
BASF DocID 2012/1272621, study 423130 (IF-12/02223803), trial L120288, decline trial France (FR) 26730 Hostun 2013-11-19	DKC 5007	1) 2012-05-04 (sowing) 2) 2012-07-15 - 2012-07-28 3) 2012-10-04	0.89	210	0.43	2012-06-09 ⁴⁾	BBCH 16	whole plant	48.0	<0.010	0.057	<0.010	<0.010	0	4) spraying
								rest of plant	<0.010 <0.010	<0.010 <0.010	<0.010 <0.010	<0.010 <0.010	<0.010 <0.010	60 117	Sample storage time not validated 8.3: rest of plant, rest of plant, silage stage, whole plant
								rest of plant, silage stage	<0.010 <0.010	<0.010 <0.010	<0.010 <0.010	<0.010 <0.010	<0.010 <0.010	86 89	8.4: maize cob with husks, milk stage, maize cob with husks, silage stage, grain
								maize cob with husks, milk stage	<0.010	<0.010	<0.010	<0.010	<0.010	60	Analytical method: BASF No. L0179/02 (HPLC-MS/MS), LOQ(s): 0.01 mg/kg
								maize cob with husks, silage stage	<0.010 <0.010	<0.010 <0.010	<0.010 <0.010	<0.010 <0.010	<0.010 <0.010	86 89	Max. sample storage time in month(s): 10 (high water content) 7 (high starch content)
								grain	<0.010	<0.010	<0.010	<0.010	<0.010	117	ASB2014-10046
BASF DocID 2012/1272621,	A5 72	1) 2012-04-29 (sowing)	0.88	200	0.43	2012-05-22 ⁴⁾	BBCH 16	whole plant	35.0	<0.010	<0.010	<0.010	<0.010	0	4) spraying

1	2	3	4			5	6	7	8.1	8.2	8.3	8.4	8.5	9	10
Report-No. Location incl. Postal code and date	Commodity/ Variety	Date of 1) Sowing or planting 2) Flowering 3) Harvest	Application rate per treatment			Dates of treatments or no. of treatments and last date	Growth stage at last treatment or date	Portion analysed	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	PHI (days)	Remarks
			kg as/ha	Water L/ha	kg as/hL										
	(a)	(b)				(c)		(a)						(d)	(e)
study 423130 (IF-12/02223803), trial L120289, decline trial Greece (GR) 59032 Platanos, Imathia 2013-11-19		2) 2012-06-30 - 2012-07-15 3) 2012-09-01 - 2012-09-20						rest of plant	<0.010 <0.010	<0.010 <0.010	<0.010 <0.010	<0.010 <0.010	<0.010 <0.010	59 99	Sample storage time not validated 8.3: whole plant, rest of plant, rest of plant, silage stage 8.4: maize cob with husks, silage stage, maize cob with husks, milk stage, maize cob with husks, grain Analytical method: BASF No. L0179/02 (HPLC-MS/MS), LOQ(s): 0.01 mg/kg Max. sample storage time in month(s): 11 (high water content) 8 (high starch content) ASB2014-10046
								rest of plant, silage stage	<0.010 <0.010	<0.010 <0.010	<0.010 <0.010	<0.010 <0.010	<0.010 <0.010	86 90	
								maize cob with husks	<0.010	<0.010	<0.010	<0.010	<0.010	90	
								maize cob with husks, milk stage	<0.010 <0.010	<0.010	<0.010 <0.010	<0.010	<0.010 <0.010	59	
								maize cob with husks, silage stage	<0.010	<0.010	<0.010	<0.010	<0.010	86	
								grain	<0.010 <0.010	<0.010	<0.010 <0.010	<0.010	<0.010 <0.010	99	
BASF DocID 2012/1272621, study 423130 (IF-12/02223803), trial L120290, decline trial Italy (IT) 20080 Zibido San Giacomo (MI)	Armonico	1) 2012-04-13 (sowing) 2) 2012-06-28 - 2012-07-04 3) 2012-08-31	0.90	210	0.43	2012-06-01 ⁴⁾	BBCH 16	whole plant	37.0	<0.010	0.053	<0.010	<0.010	0	4) spraying
								rest of plant	<0.010	<0.010	<0.010	<0.010	<0.010	91	Sample storage time not validated 8.3: rest of plant, rest of plant, silage stage, whole plant 8.4: maize cob with husks, silage stage, grain Analytical method:
								rest of plant, silage stage	<0.010 <0.010	<0.010	<0.010 <0.010	<0.010	<0.010 <0.010	60	

1	2	3	4			5	6	7	8.1	8.2	8.3	8.4	8.5	9	10
Report-No. Location incl. Postal code and date	Commodity/ Variety	Date of 1) Sowing or planting 2) Flowering 3) Harvest	Application rate per treatment			Dates of treatments or no. of treatments and last date	Growth stage at last treatment or date	Portion analysed	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	PHI (days)	Remarks
			kg as/ha	Water L/ha	kg as/hL										
	(a)	(b)				(c)		(a)						(d)	(e)
2013-11-19								maize cob with husks, silage stage	<u><0.010</u>	<0.010	<u><0.010</u>	<0.010	<u><0.010</u>	60	BASF No. L0179/02 (HPLC-MS/MS), LOQ(s): 0.01 mg/kg
								grain	<u><0.010</u>	<0.010	<u><0.010</u>	<0.010	<u><0.010</u>	91	Max. sample storage time in month(s): 10 (high water content) 8 (high starch content) ASB2014-10046
BASF DocID 2012/1272621, study 423130 (IF-12/02223803), trial L120291, decline trial Spain (ES) 18128 Zafarraya (Granada) 2013-11-19	Castellano	1) 2012-06-13 (sowing) 2) 2012-08-17 - 2012-08-24 3) 2012-10-15	0.84	200	0.43	2012-07-16 ⁴⁾	BBCH 16	whole plant	8.8	<0.010	0.14	<0.010	0.036	0	4) spraying
								rest of plant	<0.010 <0.010	<0.010 <0.010	0.017 0.037	<0.010 <0.010	0.14 0.28	60 91	Sample storage time not validated 8.3: rest of plant, whole plant, rest of plant, silage stage
								rest of plant, silage stage	<u><0.010</u>	<0.010	<u>0.036</u>	<0.010	<u>0.23</u>	78	8.4: maize cob with husks, milk stage, maize cob with husks, silage stage, grain
								maize cob with husks, milk stage	<0.010	<0.010	<0.010	<0.010	<0.010	60	Analytical method: BASF No. L0179/02 (HPLC-MS/MS), LOQ(s): 0.01 mg/kg
								maize cob with husks, silage stage	<u><0.010</u>	<0.010	<u><0.010</u>	<0.010	<u><0.010</u>	78	Max. sample storage time in month(s): 9 (high water content) 7 (high starch content)
								grain	<u><0.010</u>	<0.010	<u><0.010</u>	<0.010	<u><0.010</u>	91	ASB2014-10046

Remarks:

- (a) According to CODEX Classification / Guide
- (b) Only if relevant
- (c) Year must be indicated
- (d) Days after last application (Label pre-harvest interval, PHI, underline)
- (e) Remarks may include: Climatic conditions; Reference to analytical method and information which metabolites are included

B.7.3.1.2 Soya bean

The cGAP in N-EU is 1 x 0.864 kg as/ha spray application at pre-emergence stage up to BBCH 09. PHI is covered by the growing period between application and harvest.

Reference:	KCA 6.3.3/1
Report	Oxspring S. (2014): Study on the residue behaviour of dimethenamid-P (BAS 656 H) in soybean after treatment with BAS 656 12 H in Northern and Southern Europe during 2013, BASF Doc ID: 2013/1335421, Study code: 697666, ASB2014-8347
Guideline(s):	None stated
Deviations:	None
GLP:	Yes
Acceptability:	The study is acceptable.

Materials and methods

Out of 16 supervised residue trials in soya bean, 8 trials were conducted in N-EU and 8 trials in S-EU in 2013.

Soya bean plants were treated according to the GAP with 1 application at nominal rates of 0.86 - 0.95 kg as/ha. Seeds and forage were harvested at DALA 112-168 and 51-90, respectively. The samples were stored deep frozen for max. 8 months.

All samples were analysed for dimethenamid-P and metabolites M23, M26, M27 and M30 according to method BASF L0179/02. For a detailed method description see B.5.2.1.1.

Results and Discussion

Procedural recoveries and precisions were within acceptable limits (Table B.7.3-4), except for the recovery of M30 in the rest of the plants at the 10 and 1000xLOQ level. Storage of soya bean seed samples was covered by acceptable storage stability studies for all analytes. For high water content samples, storage stability was validated for dimethenamid-P, M23 and M30, but not for M26. Residue levels in individual samples are reported in Table B.7.3-5 and Table B.7.3-6. No residues were found in untreated control samples.

Table B.7.3-4: Procedural recoveries for dimethenamid-P in soya bean

Matrix	Fortification Level [mg/kg]	Summary recoveries							
		Recovery [%]	Mean [%]	RSD [%]	n	Recovery [%]	Mean [%]	RSD [%]	n
		Dimethenamid-P				M23			
Whole plant	0.01	94	94	-	1	100	100	-	1
	0.1	98	98	-	1	96	96	-	1
	1	101	101	-	1	101	101	-	1
	10	92	92	-	1	90	90	-	1
Seed	0.01	98, 99, 104, 125, 91, 95, 105	102	11	7	82, 104, 114, 118, 86, 99, 96	100	13	7
	0.1	97, 98, 103, 108, 91, 97, 104	100	6	7	89, 98, 102, 112, 88, 95, 94	97	9	7
	1	97, 101, 102, 104	101	3	4	92, 108, 103, 99	101	7	4
Rest of plant	0.01	96	96	-	1	99	99	-	1
	0.1	103	103	-	1	93	93	-	1
	1	101	101	-	1	93	93	-	1
	10	98	98	-	1	90	90	-	1
		M26				M27			
Whole plant	0.01	97	97	-	1	100	100	-	1
	0.1	81	81	-	1	101	101	-	1
	1	92	92	-	1	104	104	-	1
	10	77	77	-	1	93	93	-	1
Seed	0.01	73, 104, 111, 105, 97, 82, 84	94	15	7	78, 90, 112, 106, 76, 98, 96	94	14	7
	0.1	85, 103, 106, 115, 107, 80, 79	96	15	7	83, 88, 103, 101, 77, 94, 95	92	10	7
	1	97, 116, 94, 87	99	13	4	92, 96, 102, 95	96	4	4
Rest of plant	0.01	80	80	-	1	96	96	-	1
	0.1	81	81	-	1	91	91	-	1
	1	80	80	-	1	90	90	-	1
	10	71	71	-	1	85	85	-	1
		M30							
Whole plant	0.01	88	88	-	1				
	0.1	90	90	-	1				
	1	88	88	-	1				
	10	84	84	-	1				
Seed	0.01	74, 80, 101, 104, 77, 84, 73	85	15	7				
	0.1	81, 90, 100, 113, 68, 85, 70	87	18	7				
	1	91, 100, 94, 71	89	14	4				
Rest of plant	0.01	82	82	-	1				
	0.1	68	68	-	1				
	1	70	70	-	1				
	10	64	64	-	1				

Conclusion

Of the 16 trials submitted for N-EU and S-EU, all were considered acceptable. For metabolite M26 the storage stability in high water content commodities (rest of the plants and whole plant) could not be validated as the maximum storage stability was determined equal to 3 months only. This is however relevant for the matrix rest of the plants only, since the matrix whole plant was not considered in the assessment.

Table B.7.3-5: N-EU residues trials with soya bean conducted according to GAP

RESIDUES DATA SUMMARY FROM SUPERVISED TRIALS (SUMMARY)
(Application on agricultural and horticultural crops)

Federal Institute for Risk Assessment, Berlin
Federal Republic of Germany

Content of a.s. (g/kg or g/L) : 720 g/L
Formulation (e.g. WP) : EC (emulsifiable concentrate)
Commercial product (name) : BAS 656 12 H
Applicant : BASF SE

Active substance : Dimethenamid-P
Crop / crop group : Soya Bean
Crop Code : GLXMA

Submission date : 2014-08-21

Indoors / Outdoors : Outdoors (European North)
Other a.s. in formulation (content and common name) :
Residues calculated as : 8.1 dimethenamid-P
8.2 M23 oxalamide
8.3 M26 thiolactic acid conjugate
8.4 M27 sulfonate
8.5 M30 sulfoxide of thiolactic acid conjugate

1	2	3	4			5	6	7	8.1	8.2	8.3	8.4	8.5	9	10
Report-No. Location incl. Postal code and date	Commodity/ Variety	Date of 1) Sowing or planting 2) Flowering 3) Harvest	Application rate per treatment			Dates of treatments or no. of treatments and last date	Growth stage at last treatment or date	Portion analysed	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	PHI (days)	Remarks
	(a)	(b)	kg as/ha	Water L/ha	kg as/hL	(c)		(a)						(d)	(e)
BASF DocID 2013/1335421, study 697666 (Eurofins S13- 01079), trial L130372 (S13- 01079-01), plot 2, harvest trial Germany (DE) 71665 Vaihingen/Enz 2014-04-02	Sultana	1) 2013-05-15 (sowing) 2) 2013-06 - 2013-07 3) 2013-10-02	0.94	110	0.86	2013-06-02 ⁴⁾	BBCH 09	whole plant rest of plant seed(s)	<0.010 <0.010 <0.010	0.010 <0.010 <0.010	<0.010 <0.010 <0.010	0.024 <0.010 <0.010	<0.010 <0.010 <0.010	58 122 122	4) spraying Sample storage time not validated 8.3: rest of plant, whole plant Analytical method: BASF No. L0179/02 (HPLC-MS/MS), LOQ(s): 0.01 mg/kg Max. sample storage time in month(s): 8 (rest of plant) 1 (seeds) ASB2014-8347
BASF DocID 2013/1335421, study 697666	Silvia	1) 2013-04-16 (sowing) 2) 2013-07	0.92	110	0.86	2013-05-02 ⁴⁾	BBCH 09	whole plant	<0.010	<0.010	<0.010	0.043	<0.010	70	4) spraying Sample storage time not

1	2	3	4			5	6	7	8.1	8.2	8.3	8.4	8.5	9	10
Report-No. Location incl. Postal code and date	Commodity/ Variety	Date of 1) Sowing or planting 2) Flowering 3) Harvest	Application rate per treatment			Dates of treatments or no. of treatments and last date	Growth stage at last treatment or date	Portion analysed	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	PHI (days)	Remarks
			kg as/ha	Water L/ha	kg as/hL										
	(a)	(b)				(c)		(a)						(d)	(e)
(Eurofins S13-01079), trial L130373 (S13-01079-02), harvest trial Germany (DE) 74349 Ingersheim 2014-04-02		- 2013-08 3) 2013-10-04						rest of plant	<0.010	<0.010	<0.010	<0.010	<0.010	155	validated
								seed(s)	<0.010	<0.010	<0.010	<0.010	<0.010	155	8.3: whole plant, rest of plant Analytical method: BASF No. L0179/02 (HPLC-MS/MS), LOQ(s): 0.01 mg/kg Max. sample storage time in month(s): 8 (rest of plant) 1 (seeds) ASB2014-8347
BASF DocID 2013/1335421, study 697666 (Eurofins S13-01079), trial L130374 (S13-01079-03), harvest trial Hungary (HU) H-8143 Sarszentmihaly, Fejer 2014-04-02	Pannonia Kincse	1) 2013-04-30 (sowing) 2) 2013-07-15 - 2013-07-30 3) 2013	0.94	110	0.87	2013-04-30 ⁴⁾	BBCH 00	whole plant	<0.010	<0.010	<0.010	0.014	<0.010	90	4) spraying
								rest of plant	<0.010	<0.010	<0.010	<0.010	<0.010	168	Sample storage time not validated
								seed(s)	<0.010	<0.010	<0.010	<0.010	<0.010	168	8.3: whole plant, rest of plant Analytical method: BASF No. L0179/02 (HPLC-MS/MS), LOQ(s): 0.01 mg/kg Max. sample storage time in month(s): 8 (rest of plant) 1 (seeds) ASB2014-8347
BASF DocID 2013/1335421, study 697666 (Eurofins S13-01079), trial L130375 (S13-	Isidor	1) 2013-05-10 (sowing) 2) 2013-07-15 - 2013-07-30 3) 2013	0.94	110	0.86	2013-05-10 ⁴⁾	BBCH 00	whole plant	<0.010	<0.010	<0.010	0.061	<0.010	80	4) spraying
								rest of plant	<0.010	<0.010	<0.010	0.016	<0.010	164	Sample storage time not validated 8.3: whole plant, rest of plant

1	2	3	4			5	6	7	8.1	8.2	8.3	8.4	8.5	9	10
Report-No. Location incl. Postal code and date	Commodity/ Variety	Date of 1) Sowing or planting 2) Flowering 3) Harvest	Application rate per treatment			Dates of treatments or no. of treatments and last date	Growth stage at last treatment or date	Portion analysed	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	PHI (days)	Remarks
			kg as/ha	Water L/ha	kg as/hL										
	(a)	(b)				(c)		(a)						(d)	(e)
01079-04), harvest trial Hungary (HU) H-8710 Balatonszentgyprgy, Somogy 2014-04-02								seed(s)	<0.010	<0.010	<0.010	<0.010	<0.010	164	Analytical method: BASF No. L0179/02 (HPLC-MS/MS), LOQ(s): 0.01 mg/kg Max. sample storage time in month(s): 8 (rest of plant) 2(seeds) ASB2014-8347
BASF DocID 2013/1335421, study 697666 (Eurofins S13- 01079), trial L130376 (S13- 01079-05), harvest trial Poland (PL) 64-500 Szamotuly, Wielkopolska 2014-04-02	Augusta	1) 2013-05-13 (sowing) 2) 2013-07-05 - 2013-08-01 3) 2013-09-25	0.88	100	0.86	2013-05-21 ⁴⁾	BBCH 09	whole plant	<0.010	0.014	<0.010	0.15	<0.010	72	4) spraying
								rest of plant	<0.010	<0.010	<0.010	0.033	<0.010	127	Sample storage time not validated 8.3: rest of plant, whole plant
								seed(s)	<0.010	<0.010	<0.010	<0.010	<0.010	127	Analytical method: BASF No. L0179/02 (HPLC-MS/MS), LOQ(s): 0.01 mg/kg Max. sample storage time in month(s): 7 (rest of plant) 2 (seeds) ASB2014-8347
BASF DocID 2013/1335421, study 697666 (Eurofins S13- 01079), trial L130377 (S13-	Augusta	1) 2013-05-15 (sowing) 2) 2013-07-08 - 2013-08-07 3) 2013-09-28	0.86	100	0.86	2013-05-27 ⁴⁾	BBCH 09	whole plant	<0.010	0.012	<0.010	0.045	<0.010	71	4) spraying
								rest of plant	<0.010	<0.010	<0.010	0.016	<0.010	124	Sample storage time not validated 8.3: rest of plant, whole plant

1	2	3	4			5	6	7	8.1	8.2	8.3	8.4	8.5	9	10
Report-No. Location incl. Postal code and date	Commodity/ Variety	Date of 1) Sowing or planting 2) Flowering 3) Harvest	Application rate per treatment			Dates of treatments or no. of treatments and last date	Growth stage at last treatment or date	Portion analysed	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	PHI (days)	Remarks
			kg as/ha	Water L/ha	kg as/hL										
	(a)	(b)				(c)		(a)						(d)	(e)
01079-06), harvest trial Poland (PL) 88-400 Znin, Kujawsko- Pomorskie 2014-04-02								seed(s)	<0.010	<0.010	<0.010	<0.010	<0.010	124	Analytical method: BASF No. L0179/02 (HPLC-MS/MS), LOQ(s): 0.01 mg/kg Max. sample storage time in month(s): 7 (rest of plant) 2 (seeds) ASB2014-8347
BASF DocID 2013/1335421, study 697666 (Eurofins S13- 01079), trial L130378 (S13- 01079-07), harvest trial France (FR) 45130 Baule, Loiret 2014-04-02	Isidor NT	1) 2013-05-10 (sowing) 2) 2013-07-15 - 2013-08-05 3) 2013-11-04	0.94	110	0.87	2013-05-22 ⁴⁾	BBCH 09	whole plant	<0.010	<0.010	<0.010	0.017	<0.010	68	4) spraying
								rest of plant	<0.010	<0.010	<0.010	<0.010	<0.010	166	Sample storage time not validated 8.3: rest of plant, whole plant
								seed(s)	<0.010	<0.010	<0.010	<0.010	<0.010	166	Analytical method: BASF No. L0179/02 (HPLC-MS/MS), LOQ(s): 0.01 mg/kg Max. sample storage time in month(s): 8 (rest of plant) 1 (seeds) ASB2014-8347
BASF DocID 2013/1335421, study 697666 (Eurofins S13- 01079), trial L130379 (S13-	Sultana	1) 2013-06-08 (sowing) 2) 2013-08-01 - 2013-08-20 3) 2013-10-07	0.92	110	0.86	2013-06-12 ⁴⁾	BBCH 09	whole plant	<0.010	0.014	<0.010	0.017	<0.010	65	4) spraying
								rest of plant	<0.010	<0.010	<0.010	<0.010	<0.010	117	Sample storage time not validated 8.3: rest of plant, whole plant

1	2	3	4			5	6	7	8.1	8.2	8.3	8.4	8.5	9	10
Report-No. Location incl. Postal code and date	Commodity/ Variety	Date of 1) Sowing or planting 2) Flowering 3) Harvest	Application rate per treatment			Dates of treatments or no. of treatments and last date	Growth stage at last treatment or date	Portion analysed	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	PHI (days)	Remarks
			kg as/ha	Water L/ha	kg as/hL										
	(a)	(b)				(c)		(a)						(d)	(e)
01079-08), harvest trial France (FR) 91150 Mespuits, Essonne 2014-04-02								seed(s)	<u><0.010</u>	<0.010	<u><0.010</u>	<0.010	<u><0.010</u>	117	Analytical method: BASF No. L0179/02 (HPLC-MS/MS), LOQ(s): 0.01 mg/kg. Max. sample storage time in month(s): 7 (rest of plant) 1 (seeds) ASB2014-8347

Remarks:

- (a) According to CODEX Classification / Guide
- (b) Only if relevant
- (c) Year must be indicated
- (d) Days after last application (Label pre-harvest interval, PHI, underline)
- (e) Remarks may include: Climatic conditions; Reference to analytical method and information which metabolites are included

Table B.7.3-6: S-EU residues trials with soya bean conducted according to GAP

RESIDUES DATA SUMMARY FROM SUPERVISED TRIALS (SUMMARY)
(Application on agricultural and horticultural crops)

Federal Institute for Risk Assessment, Berlin
Federal Republic of Germany

Content of a.s. (g/kg or g/L) : 720 g/L
Formulation (e.g. WP) : EC (emulsifiable concentrate)
Commercial product (name) : BAS 656 12 H
Applicant : BASF SE

Active substance : Dimethenamid-P
Crop / crop group : Soya Bean
Crop Code : GLXMA

Submission date : 2014-08-21

Indoors / Outdoors : Outdoors (European South)
Other a.s. in formulation (content and common name) :
Residues calculated as : 8.1 dimethenamid-P
8.2 M23 oxalamide
8.3 M26 thiolactic acid conjugate
8.4 M27 sulfonate
8.5 M30 sulfoxide of thiolactic acid conjugate

1	2	3	4			5	6	7	8.1	8.2	8.3	8.4	8.5	9	10
Report-No. Location incl. Postal code and date	Commodity/ Variety	Date of 1) Sowing or planting 2) Flowering 3) Harvest	Application rate per treatment			Dates of treatments or no. of treatments and last date	Growth stage at last treatment or date	Portion analysed	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	PHI (days)	Remarks
	(a)	(b)	kg as/ha	Water L/ha	kg as/hL	(c)		(a)						(d)	(e)
BASF DocID 2013/1335421, study 697666 (Eurofins S13- 01079), trial L130380 (S13- 01079-09), harvest trial France (FR) 82400 St. Paul D'Epsis, Tarn-et-Garonne 2014-04-02	Astaffort	1) 2013-06-09 (sowing) 2) 2013-08-07 - 2013-08-21 3) 2013-10-07	0.92	110	0.86	2013-06-17 ⁴⁾	BBCH 10	whole plant rest of plant seed(s)	<0.010 <0.010 <0.010	<0.010 <0.010 <0.010	<0.010 <0.010 <0.010	<0.010 <0.010 <0.010	<0.010 <0.010 <0.010	63 112 112	4) spraying Sample storage time not validated 8.3: whole plant, rest of plant Analytical method: BASF No. L0179/02 (HPLC-MS/MS), LOQ(s): 0.01 mg/kg Max. sample storage time in month(s): 7 (rest of plant) 1 (seeds) ASB2014-8347
BASF DocID 2013/1335421, study 697666	Fukui	1) 2013-05-13 (sowing) 2) 2013-08-10	0.95	110	0.86	2013-06-04 ⁴⁾	BBCH 09	whole plant	<0.010	<0.010	<0.010	0.030	<0.010	76	4) spraying Sample storage time not

1	2	3	4			5	6	7	8.1	8.2	8.3	8.4	8.5	9	10
Report-No. Location incl. Postal code and date	Commodity/ Variety	Date of 1) Sowing or planting 2) Flowering 3) Harvest	Application rate per treatment			Dates of treatments or no. of treatments and last date	Growth stage at last treatment or date	Portion analysed	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	PHI (days)	Remarks
	(a)	(b)	kg as/ha	Water L/ha	kg as/hL	(c)		(a)						(d)	(e)
(Eurofins S13-01079), trial L130381 (S13-01079-10), harvest trial France (FR) 82500 Serignac, Tarn-et-Garonne 2014-04-02		- 2013-08-2 3) 2013-10-07						rest of plant seed(s)	<u><0.010</u> <u><0.010</u>	<0.010 <0.010	<u><0.010</u> <u><0.010</u>	<0.010 <0.010	<u><0.010</u> <u><0.010</u>	125 125	validated 8.3: whole plant, rest of plant Analytical method: BASF No. L0179/02 (HPLC-MS/MS), LOQ(s): 0.01 mg/kg Max. sample storage time in month(s): 7 (rest of plant) 1 (seeds) ASB2014-8347
BASF DocID 2013/1335421, study 697666 (Eurofins S13-01079), trial L130382 (S13-01079-11), harvest trial Greece (GR) 59032 Plantos, Imathia, Central Macedonia 2014-04-02	Blancas	1) 2013-05-02 (sowing) 2) 2013-06-20 - 2013-07-05 3) 2013-09-05	0.86	100	0.86	2013-05-09 ⁴⁾	BBCH 09	whole plant rest of plant seed(s)	<0.010 <u><0.010</u> <u><0.010</u>	<0.010 <0.010 <0.010	<0.010 <u><0.010</u> <u><0.010</u>	<0.010 <0.010 <0.010	<0.010 <u><0.010</u> <u><0.010</u>	53 119 119	4) spraying Sample storage time not validated 8.3: whole plant, rest of plant Analytical method: BASF No. L0179/02 (HPLC-MS/MS), LOQ(s): 0.01 mg/kg Max. sample storage time in month(s): 8 (rest of plant) 1 (seeds) ASB2014-8347
BASF DocID 2013/1335421, study 697666 (Eurofins S13-01079), trial L130383 (S13-	Blancas	1) 2013-05-06 (sowing) 2) 2013-06-25 - 2013-07-05 3) 2013-09-04	0.86	100	0.86	2013-05-13 ⁴⁾	BBCH 09	whole plant rest of plant	<0.010 <u><0.010</u>	<0.010 <0.010	<0.010 <u><0.010</u>	0.012 <0.010	<0.010 <u><0.010</u>	51 114	4) spraying Sample storage time not validated 8.3: whole plant, rest of plant

1	2	3	4			5	6	7	8.1	8.2	8.3	8.4	8.5	9	10
Report-No. Location incl. Postal code and date	Commodity/ Variety	Date of 1) Sowing or planting 2) Flowering 3) Harvest	Application rate per treatment			Dates of treatments or no. of treatments and last date	Growth stage at last treatment or date	Portion analysed	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	PHI (days)	Remarks
			kg as/ha	Water L/ha	kg as/hL										
(a)	(a)	(b)				(c)		(a)						(d)	(e)
01079-12), harvest trial Greece (GR) 57200 Profitis, Central Macedonia 2014-04-02								seed(s)	<0.010	<0.010	<0.010	<0.010	<0.010	114	Analytical method: BASF No. L0179/02 (HPLC-MS/MS), LOQ(s): 0.01 mg/kg Max. sample storage time in month(s): 8 (rest of plant) 1 (seeds) ASB2014-8347
BASF DocID 2013/1335421, study 697666 (Eurofins S13- 01079), trial L130384 (S13- 01079-13), harvest trial Italy (IT) 44012 Finale Emilia, Emilia Romagna 2014-04-02	Deka Big	1) 2013-05-15 (sowing) 2) 2013-07 - 2013-07 3) 2013-09-30	0.89	100	0.87	2013-05-27 ⁴⁾	BBCH 09	whole plant	<0.010	<0.010	<0.010	<0.010	<0.010	52	4) spraying
								rest of plant	<0.010	<0.010	<0.010	<0.010	<0.010	126	Sample storage time not validated 8.3: whole plant, rest of plant
								seed(s)	<0.010	<0.010	<0.010	<0.010	<0.010	126	Analytical method: BASF No. L0179/02 (HPLC-MS/MS), LOQ(s): 0.01 mg/kg Max. sample storage time in month(s): 8 (rest of plant) 1 (seeds) ASB2014-8347
BASF DocID 2013/1335421, study 697666 (Eurofins S13- 01079), trial L130385 (S13-	Demetra	1) 2013-05-29 (sowing) 2) 2013-07 - 2013-07 3) 2013-09-11	0.92	110	0.86	2013-05-08 ⁴⁾	BBCH 09	whole plant	<0.010	<0.010	<0.010	<0.010	<0.010	71	4) spraying
								rest of plant	<0.010	<0.010	<0.010	<0.010	<0.010	126	Sample storage time not validated 8.3: rest of plant, whole plant

1	2	3	4			5	6	7	8.1	8.2	8.3	8.4	8.5	9	10
Report-No. Location incl. Postal code and date	Commodity/ Variety	Date of 1) Sowing or planting 2) Flowering 3) Harvest	Application rate per treatment			Dates of treatments or no. of treatments and last date	Growth stage at last treatment or date	Portion analysed	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	PHI (days)	Remarks
			kg as/ha	Water L/ha	kg as/hL										
(a)	(a)	(b)				(c)		(a)						(d)	(e)
01079-14), harvest trial Italy (IT) 40051 Malalbergo, Emilia Romagna 2014-04-02								seed(s)	<0.010	<0.010	<0.010	<0.010	<0.010	126	Analytical method: BASF No. L0179/02 (HPLC-MS/MS), LOQ(s): 0.01 mg/kg Max. sample storage time in month(s): 8 (rest of plant) 1 (seeds) ASB2014-8347
BASF DocID 2013/1335421, study 697666 (Eurofins S13- 01079), trial L130386 (S13- 01079-15), harvest trial Spain (ES) 06180 Gevora, Badajoz 2014-04-02	PR92B63	1) 2013-05-21 (sowing) 2) 2013-07-26 - 2013-08-12 3) 2013-09-26	0.93	110	0.86	2013-05-30 ^{d)}	BBCH 09	whole plant	<0.010	<0.010	<0.010	<0.010	<0.010	75	4) spraying
								rest of plant	<0.010	<0.010	<0.010	<0.010	<0.010	119	Sample storage time not validated 8.3: rest of plant, whole plant
								seed(s)	<0.010	<0.010	<0.010	<0.010	<0.010	119	Analytical method: BASF No. L0179/02 (HPLC-MS/MS), LOQ(s): 0.01 mg/kg Max. sample storage time in month(s): 7 (rest of plant) 1 (seeds) ASB2014-8347
BASF DocID 2013/1335421, study 697666 (Eurofins S13- 01079), trial L130387 (S13-	PR92B63	1) 2013-05-21 (sowing) 2) 2013-07-26 - 2013-08-12 3) 2013-09-26	0.89	100	0.86	2013-05-30 ^{d)}	BBCH 09	whole plant	<0.010	<0.010	<0.010	0.022	0.012	75	4) spraying
								rest of plant	<0.010	0.023	<0.010	0.037	0.014	119	Sample storage time not validated 8.3: rest of plant, whole plant

1	2	3	4			5	6	7	8.1	8.2	8.3	8.4	8.5	9	10
Report-No. Location incl. Postal code and date	Commodity/ Variety	Date of 1) Sowing or planting 2) Flowering 3) Harvest	Application rate per treatment			Dates of treatments or no. of treatments and last date	Growth stage at last treatment or date	Portion analysed	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	PHI (days)	Remarks
			kg as/ha	Water L/ha	kg as/hL										
	(a)	(b)				(c)		(a)						(d)	(e)
01079-16), harvest trial Spain (ES) 06184 Pueblo Nuevo del Guadiana, Bajadoz 2014-04-02								seed(s)	<u><0.010</u>	<0.010	<u><0.010</u>	<0.010	<u><0.010</u>	119	Analytical method: BASF No. L0179/02 (HPLC-MS/MS), LOQ(s): 0.01 mg/kg Max. sample storage time in month(s): 7 (rest of plant) 2(seeds) ASB2014-8347

Remarks:

- (a) According to CODEX Classification / Guide
- (b) Only if relevant
- (c) Year must be indicated
- (d) Days after last application (Label pre-harvest interval, PHI, underline)
- (e) Remarks may include: Climatic conditions; Reference to analytical method and information which metabolites are included

B.7.3.1.3 Sunflower

The cGAP in N- and S-EU is 1 x 0.864 kg as/ha spray application at pre-emergence stage up to BBCH 09. PHI is covered by the growing period between application and harvest.

Reference:	KCA 6.3.1/1
Report	Meyer M. (2014): Residue behaviour of dimethenamid-P (BAS 656 H) in sunflower after treatment with BAS 656 12 H under field conditions in Germany, Northern France, The Netherlands, United Kingdom, Belgium, Southern France, Italy, Greece and Spain, 2013, BASF Doc ID: 2013/1335422, Study ID: 694884, ASB2014-8343
Guideline(s):	EC 1107/2009 of the European Parliament and of the Council of 21 Oct 2009, EEC 79/117, EEC 91/414, EEC 91/414 Annex II (Part A Section 6), EEC 91/414 Annex III (Part A Section 8), EEC 1607/VI/97 rev. 2 10.06.1999, EEC 7029/VI/95 rev. 5, EEC 7525/VI/95 rev. 9 (March 2011)
Deviations:	None
GLP:	Yes
Acceptability:	The study is acceptable.
Reference:	KCA 6.3.1/2
Report	Ertus C. (2014): Study on the residue behaviour of BAS 656 H (dimethenamid-P) after treatment with BAS 656 12 H in sunflower under field conditions in Northern Europe, 2012, BASF Doc ID: 2013/1335405, Study code: 390502_1, ASB2014-8344
Guideline(s):	EC 1107/2009 of the European Parliament, EEC 7029/VI/95 rev. 5 Appendix B, EEC 7525/VI/95 rev. 9 (March 2011)
Deviations:	None
GLP:	Yes
Acceptability:	The study is acceptable.
Reference:	KCA 6.3.1/3
Report	Gabriel E.J. (2013): Study on the residue behaviour of dimethenamid-P (BAS 656 H) in sunflower after treatment with BAS 656 12 H under field conditions in Germany, the Netherlands, Northern France, Southern France, Greece, Italy and Spain, 2012, BASF Doc ID: 2012/1272620, Study code: 423129, ASB2014-8345
Guideline(s):	EC 1107/2009 of the European Parliament and of the Council of 21 Oct 2009, EEC 91/414 Annex II (Part A Section 6), EEC 91/414 Annex III (Part A Section 8), EEC 1607/VI/97 rev. 2 10.06.1999, EEC 7029/VI/95 rev. 5, EEC 7525/VI/95 rev. 9 (March 2011)
Deviations:	None
GLP:	Yes
Acceptability:	The study is acceptable.

Materials and methods

Out of 25 supervised residue trials in sunflowers, 13 trials were conducted in N-EU and 12 trials in S-EU in 2013.

Sunflower plants were treated according to the GAP with 1 applications at nominal rates of 0.828 - 1.022 kg as/ha. Seeds were harvested at DALA 80-154. The samples were stored deep frozen for max. 7 months.

All samples were analysed for dimethenamid-P and metabolites M23, M26, M27 and M30 according to method BASF L0179/02. For a detailed method description see B.5.2.1.1.

Results and Discussion

Procedural recoveries and precisions were within acceptable limits (Table B.7.3-7). Storage of sunflower seed samples was covered by acceptable storage stability studies for all analytes only partially. Residue levels in individual samples are reported in Table B.7.3-8 and Table B.7.3-9. No residues were found in untreated control samples.

Table B.7.3-7: Procedural recoveries for dimethenamid-P in sunflower

Matrix	Fortification Level [mg/kg]	Summary recoveries														
		Recovery [%]				Mean [%]	RSD [%]	n	Recovery [%]			Mean [%]	RSD [%]	n		
		Dimethenamid-P						M23								
Seeds	0.01	94, 106, 106, 102, 109, 103, 102, 98				103	5	8	95, 107, 106, 108, 104, 103, 106, 100				104	4	8	
	0.1	89, 98, 108, 100, 110, 100, 101, 98				101	6	8	93, 109, 107, 106, 102, 99, 103, 103				103	5	8	
	1	91, 102, 101, 99, 106, 97				99	5	6	93, 108, 106, 102, 104, 103				103	5	6	
		M26						M27								
Seeds	0.01	102, 98, 94, 108, 100, 98, 99, 98				100	4	8	102, 105, 103, 107, 100, 107, 108, 91				103	5	8	
	0.1	96, 106, 109, 104, 114, 103, 97, 95				103	7	8	96, 106, 106, 107, 103, 96, 98, 93				101	5	8	
	1	98, 98, 103, 100, 102, 98				100	2	6	95, 110, 104, 108, 104, 101				104	5	6	
		M30														
Seeds	0.01	103, 100, 91, 94, 113, 104, 116, 93				102	9	8								
	0.1	84, 119, 115, 94, 121, 99, 96, 93				103	13	8								
	1	101, 99, 86, 97, 124, 108				103	12	6								

Conclusion

Two N-EU trials (L120044) were performed at the same site, but at different plots. One plot was treated with an application rate according to the cGAP (0.87 kg as/ha) and the other plot using a higher application rate (1 kg as/ha). Here, the higher dosed trial was chosen for risk assessment, as the trial is within ± 25 % of the cGAP and therefore is considered more critical. Also, two S-EU trials (L130200 and L130201) dosed according to the cGAP and at 1 kg as/ha were performed at the same location in the during vegetation period. Here only the higher dosed trial was considered as the dose was within ± 25 % of the cGAP and therefore is considered more critical. For several trials the sample storage time was not in the validated range, as maximum time determined during storage stability studies was 3 months only (see Table B.7.1.1). Consequently, for sunflower seeds these trials are considered provisionally acceptable only, until storage stability has been proven in the ongoing stability study by Oppinger (2014, [ASB2014-8340](#)).

Table B.7.3-8: N-EU residues trials with sunflower conducted according to GAP

RESIDUES DATA SUMMARY FROM SUPERVISED TRIALS (SUMMARY)
(Application on agricultural and horticultural crops)

Federal Institute for Risk Assessment, Berlin
Federal Republic of Germany

Content of a.s. (g/kg or g/L) : 720 g/L
Formulation (e.g. WP) : EC (emulsifiable concentrate)
Commercial product (name) : BAS 656 12 H
Applicant : BASF SE

Active substance : Dimethenamid-P
Crop / crop group : Sunflower, Common
Crop Code : HELAN

Submission date : 2014-08-21

Indoors / Outdoors : Outdoors (European North)
Other a.s. in formulation :
(content and common name) :
Residues calculated as : 8.1 dimethenamid-P
8.2 M23 oxalamide
8.3 M26 thiolactic acid conjugate
8.4 M27 sulfonate
8.5 M30 sulfoxide of thiolactic acid conjugate

1	2	3	4			5	6	7	8.1	8.2	8.3	8.4	8.5	9	10
Report-No. Location incl. Postal code and date	Commodity/ Variety	Date of 1) Sowing or planting 2) Flowering 3) Harvest	Application rate per treatment			Dates of treatments or no. of treatments and last date	Growth stage at last treatment or date	Portion analysed	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	PHI (days)	Remarks
	(a)	(b)	kg as/ha	Water L/ha	kg as/hL	(c)		(a)						(d)	(e)
BASF DocID 2013/1335422, study 694884 (IF-13/02545612), trial L130186, harvest trial Germany (DE) 16818 Reinsberg OT Braunsberg 2014-02-28	Alisson	1) 2013-04-19 (sowing) 2) 2013-07-08 - 2013-07-23 3) 2013-09-30	0.90	210	0.43	2013-04-23 ⁴⁾	BBCH 09	seed(s)	<u><0.010</u>	<0.010	<u><0.010</u>	<0.010	<u><0.010</u>	154	4) spraying analytical method: BASF No. L0179/02 (HPLC-MS/MS), LOQ(s): 0.01 mg/kg max. sample storage time in month(s): 1 (seeds) ASB2014-8343

1	2	3	4			5	6	7	8.1	8.2	8.3	8.4	8.5	9	10
Report-No. Location incl. Postal code and date	Commodity/ Variety	Date of 1) Sowing or planting 2) Flowering 3) Harvest	Application rate per treatment			Dates of treatments or no. of treatments and last date	Growth stage at last treatment or date	Portion analysed	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	PHI (days)	Remarks
			kg as/ha	Water L/ha	kg as/hL										
	(a)	(b)				(c)		(a)						(d)	(e)
BASF DocID 2013/1335422, study 694884 (IF-13/02545612), trial L130187, harvest trial Germany (DE) 49685 Höltinghausen 2014-02-28	Maestro	1) 2013-05-08 (sowing) 2) 2013-06-26 - 2013-08-10 3) 2013-10-09	0.85	200	0.43	2013-05-17 ⁴⁾	BBCH 09	seed(s)	<u><0.010</u>	<0.010	<u><0.010</u>	<0.010	<u><0.010</u>	145	4) spraying analytical method: BASF No. L0179/02 (HPLC-MS/MS), LOQ(s): 0.01 mg/kg, max. sample storage time in month(s): 1 (seeds) ASB2014-8343
BASF DocID 2013/1335422, study 694884 (IF-13/02545612), trial L130189, harvest trial France (FR) 08190 Vieux les Asfeld 2014-02-28	PE64HE01	1) 2013-04-16 (sowing) 2) 2013-07-10 - 2013-07-20 3) 2013-09-24	0.88	200	0.43	2013-05-06 ⁴⁾	BBCH 09	seed(s)	<u><0.010</u>	<0.010	<u><0.010</u>	<0.010	<u><0.010</u>	141	4) spraying analytical method: BASF No. L0179/02 (HPLC-MS/MS), LOQ(s): 0.01 mg/kg max. sample storage time in month(s): 1 (seeds) ASB2014-8343
BASF DocID 2013/1335422, study 694884 (IF-13/02545612), trial L130190, harvest trial Netherlands (NL) 9541 XH Vlagdwedde 2014-02-28	Maestro	1) 2013-05-16 (sowing) 2) 2013-06-30 - 2013-08-14 3) 2013-10-08	0.86	200	0.43	2013-05-24 ⁴⁾	BBCH 09	seed(s)	<u><0.010</u>	<0.010	<u><0.010</u>	<0.010	<u><0.010</u>	137	4) spraying analytical method: BASF No. L0179/02 (HPLC-MS/MS), LOQ(s): 0.01 mg/kg max. sample storage time in month(s): 1 (seeds) ASB2014-8343

1	2	3	4			5	6	7	8.1	8.2	8.3	8.4	8.5	9	10
Report-No. Location incl. Postal code and date	Commodity/ Variety	Date of 1) Sowing or planting 2) Flowering 3) Harvest	Application rate per treatment			Dates of treatments or no. of treatments and last date	Growth stage at last treatment or date	Portion analysed	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	PHI (days)	Remarks
			kg as/ha	Water L/ha	kg as/hL										
	(a)	(b)				(c)		(a)						(d)	(e)
BASF DocID 2013/1335422, study 694884 (IF-13/02545612), trial L130191, harvest trial Netherlands (NL) Rekken 2014-02-28	Maestro	1) 2013-05-30 (sowing) 2) 2013-07-14 - 2013-08-30 3) 2013-10-16	0.87	200	0.43	2013-06-05 ⁴⁾	BBCH 09	seed(s)	≤0.010	<0.010	≤0.010	<0.010	≤0.010	133	4) spraying analytical method: BASF No. L0179/02 (HPLC-MS/MS) LOQ(s): 0.01 mg/kg, max. sample storage time in month(s): 1 ASB2014-8343
BASF DocID 2013/1335422, study 694884 (IF-13/02545612), trial L130192, harvest trial United Kingdom (UK) OX15 6EP Banbury, Oxfordshire 2014-02-28	ES Paulina	1) 2013-05-04 (sowing) 2) 2013-08-10 - 2013-09-15 3) 2013-10-10 - 2013-10-15	0.86	200	0.43	2013-05-23 ⁴⁾	BBCH 09	seed(s)	≤0.010	<0.010	≤0.010	<0.010	≤0.010	145	4) spraying analytical method: BASF No. L0179/02 (HPLC-MS/MS), LOQ(s): 0.01 mg/kg max. sample storage time in month(s): 1 (seeds) ASB2014-8343
BASF DocID 2013/1335422, study 694884 (IF-13/02545612), trial L130650, harvest trial Belgium (BE) 6221 Saint-Amand 2014-02-28	P64HE01	1) 2013-05-28 (sowing) 2) 3) 2013-10-29	0.88	200	0.43	2013-06-10 ⁴⁾	BBCH 09	seed(s)	≤0.010	<0.010	≤0.010	<0.010	≤0.010	141	4) spraying analytical method: BASF No. L0179/02 (HPLC-MS/MS), LOQ(s): 0.01 mg/kg max. sample storage time in month(s): 1 (seeds) ASB2014-8343

1	2	3	4			5	6	7	8.1	8.2	8.3	8.4	8.5	9	10
Report-No. Location incl. Postal code and date	Commodity/ Variety	Date of 1) Sowing or planting 2) Flowering 3) Harvest	Application rate per treatment			Dates of treatments or no. of treatments and last date	Growth stage at last treatment or date	Portion analysed	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	PHI (days)	Remarks
			kg as/ha	Water L/ha	kg as/hL										
	(a)	(b)				(c)		(a)						(d)	(e)
BASF DocID 2013/1335405, study 390502_1 (ANADIAG B2016), trial L120044 (B2016 DE1), plot 2 and 3, harvest trial Germany (DE) 47574 Goch-Nierswalde 2014-01-17	Metharoc	1) 2012-04-22 (sowing) 2) 2012-06-25 - 2012-07-16 3) 2012-09-21	1.0	200	0.49	2012-04-27 ⁴⁾	BBCH 09	seed(s)	<0.010	<0.010	<0.010	<0.010	<0.010	147	4) spraying sample storage time not validated 8.1: seed(s) 8.2: seed(s) 8.3: seed(s) 8.4: seed(s) 8.5: seed(s) analytical method: BASF No. L0179/02 (HPLC-MS/MS), LOQ(s): 0.01 mg/kg max. sample storage time in month(s): 7 ASB2014-8344
	Metharoc	1) 2012-04-22 (sowing) 2) 2012-06-25 - 2012-07-16 3) 2012-09-21	0.87	200	0.43	2012-04-27 ⁴⁾	BBCH 09	seed(s)	<0.010	<0.010	<0.010	<0.010	<0.010	147	4) spraying sample storage time not validated 8.1: seed(s) 8.2: seed(s) 8.3: seed(s) 8.4: seed(s) 8.5: seed(s) analytical method: BASF No. L0179/02 (HPLC-MS/MS), LOQ(s): 0.01 mg/kg max. sample storage time in month(s): 7 ASB2014-8344

1	2	3	4			5	6	7	8.1	8.2	8.3	8.4	8.5	9	10
Report-No. Location incl. Postal code and date	Commodity/ Variety	Date of 1) Sowing or planting 2) Flowering 3) Harvest	Application rate per treatment			Dates of treatments or no. of treatments and last date	Growth stage at last treatment or date	Portion analysed	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	PHI (days)	Remarks
	(a)	(b)	kg as/ha	Water L/ha	kg as/hL	(c)		(a)						(d)	(e)
BASF DocID 2012/1272620, study 423129 (IF-12/02225097), trial L120276, harvest trial Germany (DE) 49685 Garthe 2013-07-16	Faro	1) 2012-05-18 (sowing) 2) 2012-06-25 - 2012-07-10 3) 2012	0.83	190	0.43	2012-05-25 ⁴⁾	BBCH 09	seed(s)	<u>≤0.010</u>	<0.010	<u>≤0.010</u>	<0.010	<u>≤0.010</u>	138	4) spraying sample storage time not validated 8.1: seed(s) 8.2: seed(s) 8.3: seed(s) 8.4: seed(s) 8.5: seed(s) analytical method: BASF No. L0179/02 (HPLC-MS/MS), LOQ(s): 0.01 mg/kg max. sample storage time in month(s): 6 ASB2014-8345
BASF DocID 2012/1272620, study 423129 (IF-12/02225097), trial L120278, harvest trial France (FR) 37370 Chemillé-sur- Dême 2013-07-16	Durban	1) 2012-05-17 (sowing) 2) 2012-07-15 - 2012-07-30 3) 2012-10-25 - 2012-10-28	0.86	200	0.43	2012-05-25 ⁴⁾	BBCH 08-09	seed(s)	<u>≤0.010</u>	<0.010	<u>≤0.010</u>	<0.010	<u>≤0.010</u>	152	4) spraying sample storage time not validated 8.1: seed(s) 8.2: seed(s) 8.3: seed(s) 8.4: seed(s) 8.5: seed(s) analytical method: BASF No. L0179/02 (HPLC-MS/MS), LOQ(s): 0.01 mg/kg max. sample storage time in month(s): 6 ASB2014-8345

1	2	3	4			5	6	7	8.1	8.2	8.3	8.4	8.5	9	10
Report-No. Location incl. Postal code and date	Commodity/ Variety	Date of 1) Sowing or planting 2) Flowering 3) Harvest	Application rate per treatment			Dates of treatments or no. of treatments and last date	Growth stage at last treatment or date	Portion analysed	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	PHI (days)	Remarks
			kg as/ha	Water L/ha	kg as/hL										
	(a)	(b)				(c)		(a)						(d)	(e)
BASF DocID 2012/1272620, study 423129 (IF-12/02225097), trial L120279, harvest trial France (FR) 02190 Amifontaine 2013-07-16	PE 64HE01	1) 2012-03-26 (sowing) 2) 2012-06-26 - 2012-07-10 3) 2012-09-17	0.89	210	0.43	2012-04-19 ⁴⁾	BBCH 09	seed(s)	<u>≤0.010</u>	<0.010	<u>≤0.010</u>	<0.010	<u>≤0.010</u>	151	4) spraying sample storage time not validated 8.1: seed(s) 8.2: seed(s) 8.3: seed(s) 8.4: seed(s) 8.5: seed(s) analytical method: BASF No. L0179/02 (HPLC-MS/MS), LOQ(s): 0.01 mg/kg max. sample storage time in month(s): 7 ASB2014-8345

1	2	3	4			5	6	7	8.1	8.2	8.3	8.4	8.5	9	10
Report-No. Location incl. Postal code and date	Commodity/ Variety	Date of 1) Sowing or planting 2) Flowering 3) Harvest	Application rate per treatment			Dates of treatments or no. of treatments and last date	Growth stage at last treatment or date	Portion analysed	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	PHI (days)	Remarks
			kg as/ha	Water L/ha	kg as/hL										
	(a)	(b)				(c)		(a)						(d)	(e)
BASF DocID 2012/1272620, study 423129 (IF-12/02225097), trial L120578, harvest trial Netherlands (NL) 9541 XH Vlagdwedde 2013-07-16	Faro	1) 2012-06-12 (sowing) 2) 2012-07-14 - 2012-07-30 3) 2012	0.87	200	0.43	2012-06-19 ⁴⁾	BBCH 09	seed(s)	<u>≤0.010</u>	<0.010	<u>≤0.010</u>	<0.010	<u>≤0.010</u>	115	4) spraying sample storage time not validated 8.1: seed(s) 8.2: seed(s) 8.3: seed(s) 8.4: seed(s) 8.5: seed(s) analytical method: BASF No. L0179/02 (HPLC-MS/MS), LOQ(s): 0.01 mg/kg max. sample storage time in month(s): 6 ASB2014-8345

Remarks:

- (a) According to CODEX Classification / Guide
- (b) Only if relevant
- (c) Year must be indicated
- (d) Days after last application (Label pre-harvest interval, PHI, underline)
- (e) Remarks may include: Climatic conditions; Reference to analytical method and information which metabolites are included

Table B.7.3-9: S-EU residues trials with sunflower conducted according to GAP

RESIDUES DATA SUMMARY FROM SUPERVISED TRIALS (SUMMARY)
(Application on agricultural and horticultural crops)

Federal Institute for Risk Assessment, Berlin
Federal Republic of Germany

Content of a.s. (g/kg or g/L) : 720 g/L
Formulation (e.g. WP) : EC (emulsifiable concentrate)
Commercial product (name) : BAS 656 12 H
Applicant : BASF SE

Active substance : Dimethenamid-P
Crop / crop group : Sunflower, Common
Crop Code : HELAN

Submission date : 2014-08-21

Indoors / Outdoors : Outdoors (European South)
Other a.s. in formulation (content and common name) :
Residues calculated as : 8.1 dimethenamid-P
8.2 M23 oxalamide
8.3 M26 thiolactic acid conjugate
8.4 M27 sulfonate
8.5 M30 sulfoxide of thiolactic acid conjugate

1	2	3	4			5	6	7	8.1	8.2	8.3	8.4	8.5	9	10
Report-No. Location incl. Postal code and date	Commodity/ Variety	Date of 1) Sowing or planting 2) Flowering 3) Harvest	Application rate per treatment			Dates of treatments or no. of treatments and last date	Growth stage at last treatment or date	Portion analysed	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	PHI (days)	Remarks
	(a)	(b)	kg as/ha	Water L/ha	kg as/hL	(c)		(a)						(d)	(e)
BASF DocID 2013/1335422, study 694884 (IF-13/02545612), trial L130194, harvest trial France (FR) 84420 Piolenc 2014-02-28	Olencia	1) 2013-06-06 (sowing) 2) 2013-08-01 - 2013-08-14 3) 2013-10-07	0.85	200	0.43	2013-06-14 ⁴⁾	BBCH 09	seed(s)	<0.010	<0.010	<0.010	<0.010	<0.010	115	4) spraying analytical method: BASF No. L0179/02 (HPLC-MS/MS), LOQ(s): 0.01 mg/kg max. sample storage time in month(s): 1 (seeds) ASB2014-8343

1	2	3	4			5	6	7	8.1	8.2	8.3	8.4	8.5	9	10
Report-No. Location incl. Postal code and date	Commodity/ Variety	Date of 1) Sowing or planting 2) Flowering 3) Harvest	Application rate per treatment			Dates of treatments or no. of treatments and last date	Growth stage at last treatment or date	Portion analysed	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	PHI (days)	Remarks
	(a)	(b)	kg as/ha	Water L/ha	kg as/hL	(c)		(a)						(d)	(e)
BASF DocID 2013/1335422, study 694884 (IF-13/02545612), trial L130195, harvest trial France (FR) 47120 St. Pierre sur Dropt 2014-02-28	ES-ETHIC	1) 2013-06-08 (sowing) 2) 2013-08-19 - 2013-09-03 3) 2013-10-25 - 2013-10-30	0.83	190	0.43	2013-06-19 ⁴⁾	BBCH 09	seed(s)	<u><0.010</u>	<0.010	<u><0.010</u>	<0.010	<u><0.010</u>	128	4) spraying analytical method: BASF No. L0179/02 (HPLC-MS/MS), LOQ(s): 0.01 mg/kg max. sample storage time in month(s): 1 (seeds) ASB2014-8343
BASF DocID 2013/1335422, study 694884 (IF-13/02545612), trial L130196, harvest trial Italy (IT) 25030 Pompiano 2014-02-28	Orasole	1) 2013-05-14 (sowing) 2) 2013-07-16 - 2013-07-25 3) 2013-10-01	0.85	200	0.43	2013-05-22 ⁴⁾	BBCH 09	seed(s)	<u><0.010</u>	<0.010	<u><0.010</u>	<0.010	<u><0.010</u>	132	4) spraying analytical method: BASF No. L0179/02 (HPLC-MS/MS), LOQ(s): 0.01 mg/kg max. sample storage time in month(s): 1 (seeds) ASB2014-8343
BASF DocID 2013/1335422, study 694884 (IF-13/02545612), trial L130197, harvest trial Italy (IT) 20090 Caleppio di Settala 2014-02-28	Orasole	1) 2013-06-15 (sowing) 2) 2013-08-12 - 2013-08-20 3) 2013-10-02	0.85	200	0.43	2013-06-18 ⁴⁾	BBCH 09	seed(s)	<u><0.010</u>	<0.010	<u><0.010</u>	<0.010	<u><0.010</u>	106	4) spraying analytical method: BASF No. L0179/02 (HPLC-MS/MS), LOQ(s): 0.01 mg/kg max. sample storage time in month(s): 1 (seeds) ASB2014-8343

1	2	3	4			5	6	7	8.1	8.2	8.3	8.4	8.5	9	10
Report-No. Location incl. Postal code and date	Commodity/ Variety	Date of 1) Sowing or planting 2) Flowering 3) Harvest	Application rate per treatment			Dates of treatments or no. of treatments and last date	Growth stage at last treatment or date	Portion analysed	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	PHI (days)	Remarks
	(a)	(b)	kg as/ha	Water L/ha	kg as/hL	(c)		(a)						(d)	(e)
BASF DocID 2013/1335422, study 694884 (IF-13/02545612), trial L130198, harvest trial Greece (GR) 59032 Platanos, Central Macedonia 2014-02-28	Sikklos	1) 2013-05-02 (sowing) 2) 2013-06-20 - 2013-06-30 3) 2013-08-15 - 2013-08-30	0.86	200	0.43	2013-05-09 ⁴⁾	BBCH 09	seed(s)	<u><0.010</u>	<0.010	<u><0.010</u>	<0.010	<u><0.010</u>	104	4) spraying analytical method: BASF No. L0179/02 (HPLC-MS/MS), LOQ(s): 0.01 mg/kg max. sample storage time in month(s): 1 (seeds) ASB2014-8343
BASF DocID 2013/1335422, study 694884 (IF-13/02545612), trial L130199, harvest trial Greece (GR) 57200 Profitis, Central Macedonia 2014-02-28	Sikklos	1) 2013-05-06 (sowing) 2) 2013-07-05 - 2013-07-15 3) 2013-08-15 - 2013-08-30	0.86	200	0.43	2013-05-13 ⁴⁾	BBCH 09	seed(s)	<u><0.010</u>	<0.010	<u><0.010</u>	<0.010	<u><0.010</u>	101	4) spraying analytical method: BASF No. L0179/02 (HPLC-MS/MS), LOQ(s): 0.01 mg/kg max. sample storage time in month(s): 1 (seeds) ASB2014-8343
BASF DocID 2013/1335422, study 694884 (IF-13/02545612), trial L130200, harvest trial Spain (ES) 29700 Velez Malaga 2014-02-28	LG5520	1) 2013-04-03 (sowing) 2) 2013-06-16 - 2013-07-05 3) 2013-08-20	0.90	210	0.43	2013-04-12 ⁴⁾	BBCH 09	seed(s)	<u><0.010</u>	<u><0.010</u>	<u><0.010</u>	<0.010	<u><0.010</u>	130	4) spraying analytical method: BASF No. L0179/02 (HPLC-MS/MS), LOQ(s): 0.01 mg/kg max. sample storage time in month(s): 1 (seeds) ASB2014-8343

1	2	3	4			5	6	7	8.1	8.2	8.3	8.4	8.5	9	10
Report-No. Location incl. Postal code and date	Commodity/ Variety	Date of 1) Sowing or planting 2) Flowering 3) Harvest	Application rate per treatment			Dates of treatments or no. of treatments and last date	Growth stage at last treatment or date	Portion analysed	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	PHI (days)	Remarks
	(a)	(b)	kg as/ha	Water L/ha	kg as/hL	(c)		(a)						(d)	(e)
BASF DocID 2013/1335422, study 694884 (IF-13/02545612), trial L130201, harvest trial Spain (ES) 29700 Velez Malaga 2014-02-28	LG5520	1) 2013-06-07 (sowing) 2) 2013-07-22 - 2013-08-02 3) 2013-09-06	0.89	210	0.43	2013-06-18 ⁴⁾	BBCH 09	seed(s)	<0.010	<0.010	<0.010	<0.010	<0.010	80	4) spraying analytical method: BASF No. L0179/02 (HPLC-MS/MS), LOQ(s): 0.01 mg/kg max. sample storage time in month(s): 1 (seeds) ASB2014-8343
BASF DocID 2012/1272620, study 423129 (IF-12/02225097), trial L120280, harvest trial France (FR) 84100 Orange 2013-07-16	Tutti	1) 2012-04-28 (sowing) 2) 2012-07-15 - 2012-07-30 3) 2012-09-22	0.84	200	0.43	2012-05-07 ⁴⁾	BBCH 09	seed(s)	<0.010	<0.010	<0.010	<0.010	<0.010	150	4) spraying sample storage time not validated 8.1: seed(s) 8.2: seed(s) 8.3: seed(s) 8.4: seed(s) 8.5: seed(s) analytical method: BASF No. L0179/02 (HPLC-MS/MS), LOQ(s): 0.01 mg/kg max. sample storage time in month(s): 6 ASB2014-8345

1	2	3	4			5	6	7	8.1	8.2	8.3	8.4	8.5	9	10
Report-No. Location incl. Postal code and date	Commodity/ Variety	Date of 1) Sowing or planting 2) Flowering 3) Harvest	Application rate per treatment			Dates of treatments or no. of treatments and last date	Growth stage at last treatment or date	Portion analysed	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	PHI (days)	Remarks
			kg as/ha	Water L/ha	kg as/hL										
	(a)	(b)				(c)		(a)						(d)	(e)
BASF DocID 2012/1272620, study 423129 (IF-12/02225097), trial L120281, harvest trial Greece (GR) 59032 Platanos, Imathia 2013-07-16	Sikklos	1) 2012-06-20 (sowing) 2) 2012-07-25 - 2012-08-05 3) 2012-09-15 - 2012-09-30	0.89	210	0.43	2012-06-22 ⁴⁾	BBCH 09	seed(s)	<u><0.010</u>	<0.010	<u><0.010</u>	<0.010	<u><0.010</u>	90	4) spraying sample storage time not validated 8.1: seed(s) 8.2: seed(s) 8.3: seed(s) 8.4: seed(s) 8.5: seed(s) analytical method: BASF No. L0179/02 (HPLC-MS/MS), LOQ(s): 0.01 mg/kg max. sample storage time in month(s): 7 ASB2014-8345
BASF DocID 2012/1272620, study 423129 (IF-12/02225097), trial L120282, harvest trial Italy (IT) 29010 Lusurasco di Alseno 2013-07-16	Orasole	1) 2012-04-28 (sowing) 2) 2012-07-16 - 2012-07-25 3) 2012-09-06	0.86	200	0.43	2012-05-11 ⁴⁾	BBCH 09	seed(s)	<u><0.010</u>	<0.010	<u><0.010</u>	<0.010	<u><0.010</u>	118	4) spraying sample storage time not validated 8.1: seed(s) 8.2: seed(s) 8.3: seed(s) 8.4: seed(s) 8.5: seed(s) analytical method: BASF No. L0179/02 (HPLC-MS/MS), LOQ(s): 0.01 mg/kg max. sample storage time in month(s): 7 ASB2014-8345

1	2	3	4			5	6	7	8.1	8.2	8.3	8.4	8.5	9	10
Report-No. Location incl. Postal code and date	Commodity/ Variety	Date of 1) Sowing or planting 2) Flowering 3) Harvest	Application rate per treatment			Dates of treatments or no. of treatments and last date	Growth stage at last treatment or date	Portion analysed	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	PHI (days)	Remarks
			kg as/ha	Water L/ha	kg as/hL										
	(a)	(b)				(c)		(a)						(d)	(e)
BASF DocID 2012/1272620, study 423129 (IF-12/02225097), trial L120283, harvest trial Spain (ES) 18128 Zafarraya 2013-07-16	Bosfora	1) 2012-06-13 (sowing) 2) 2012-08-15 - 2012-08-25 3) 2012-10-15	0.89	210	0.43	2012-07-16 ⁴⁾	BBCH 09	seed(s)	<u><0.010</u>	<0.010	<u><0.010</u>	<0.010	<u><0.010</u>	91	4) spraying sample storage time not validated 8.1: seed(s) 8.2: seed(s) 8.3: seed(s) 8.4: seed(s) 8.5: seed(s) analytical method: BASF No. L0179/02 (HPLC-MS/MS), LOQ(s): 0.01 mg/kg max. sample storage time in month(s): 6 ASB2014-8345

Remarks:

- (a) According to CODEX Classification / Guide
- (b) Only if relevant
- (c) Year must be indicated
- (d) Days after last application (Label pre-harvest interval, PHI, underline)
- (e) Remarks may include: Climatic conditions; Reference to analytical method and information which metabolites are included

B.7.3.1.4 Sugar beet

The cGAP in N- and S-EU is 1 x 0.864 kg as/ha spray application at pre-emergence stage up to BBCH 09. PHI is covered by the growing period between application and harvest.

Reference:	KCA 6.3.5/1
Report	Erdmann H.-P. (2013): Study on the residue behaviour of BAS 656 H (dimethenamid-P) in beets after application of BAS 656 12 H under field condition in United Kingdom, Northern and Southern France, The Netherlands, Italy and Germany, 2012, BASF Doc ID: 2013/1003729, Study code: 423128, ASB2014-3705
Guideline(s):	EC 1107/2009 of the European Parliament and of the Council of 21 Oct 2009, EEC 79/117, EEC 91/414, EEC 7029/VI/95 rev. 5 Appendix B (July 22 1997), EEC 7525/VI/95 rev. 9 (March 2011)
Deviations:	None
GLP:	Yes
Acceptability:	The study is acceptable.

Reference:	KCA 6.3.5/2
Report	Perny A. (2012): Study on the residue behaviour of BAS 656 H (dimethenamid-P) after treatment with BAS 656 12 H in beets (sugar) under field conditions in Northern and Southern Europe, 2011, BASF Doc ID: 2012/1182982, Study code: 390503, ASB2014-3703
Guideline(s):	EEC 1607/VI/97 rev. 2 10.06.1999, EEC 91/414 Annex II (Part A Section 6), EEC 91/414 Annex III (Part A Section 8), EEC 7029/VI/95 rev. 5 Appendix B, EEC 7525/VI/95 rev. 7
Deviations:	None
GLP:	Yes
Acceptability:	The study is acceptable.

Materials and methods

Out of 12 supervised residue trials in sugar beets, 8 trials were conducted in N-EU and 4 trials in S-EU in 2011 and 2012.

Sugar beet plants were treated according to the GAP with 1 application at nominal rates of 0.616-0.704 kg as/ha. Seeds were harvested at DALA 103-142. The samples were stored deep frozen for max. 11 months.

All samples were analysed for dimethenamid-P and metabolites M23, M26, M27 and M30 according to method BASF L0179/02. For a detailed method description see B.5.2.1.1.

Results and Discussion

Procedural recoveries and precisions were within acceptable limits (Table B.7.3-10). However, in the study Perny (2013) no individual recoveries were reported. Storage stability was not validated for metabolite M26 in sugar beet whole plant and leaves and for M27 in root body. Residue levels in individual samples are reported in Table B.7.3-11 and Table B.7.3-12. No residues were found in untreated control samples.

Table B.7.3-10: Procedural recoveries for dimethenamid-P in sugar beet

Matrix	Fortification Level [mg/kg]	Summary recoveries							
		Recovery [%]	Mean [%]	RSD [%]	n	Recovery [%]	Mean [%]	RSD [%]	n
		Dimethenamid-p				M23			
Whole plant	0.01	95,	-	-	1	104	-	-	1
	1	106	-	-	1	108	-	-	1
	2	97	-	-	1	99	-	-	1
	0.01-45	-	98	3.4	4	-	100	6.0	4
Leaves	0.01	101	-	-	1	103	-	-	1
	0.1	99	-	-	1	98	-	-	1
	10	95	-	-	1	94	-	-	1
	0.01-1	-	102	2.3	3	-	100	0.8	3
Roots	0.01	98	-	-	1	101	-	-	1
	0.1	97	-	-	1	98	-	-	1
	0.01-0.1	-	106	-	2	-	106	-	2
		M26				M27			
Whole plant	0.01	93	-	-	1	104	-	-	1
	1	108	-	-	1	110	-	-	1
	2	99	-	-	1	101	-	-	1
	0.01-45	-	101	4.5	4	-	99	2.7	4
Leaves	0.01	96	-	-	1	103	-	-	1
	0.1	92	-	-	1	103	-	-	1
	10	83	-	-	1	93	-	-	1
	0.01-1	-	100	1.8	3	-	103	2.6	3
Roots	0.01	88	-	-	1	98	-	-	1
	0.1	98	-	-	1	97	-	-	1
	0.01-0.1	-	106	-	2	-	107	-	2
		M30							
Whole plant	0.01	94	-	-	1				
	1	105	-	-	1				
	2	98	-	-	1				
	0.01-45	-	96	2.4	4				
Leaves	0.01	91	-	-	1				
	0.1	105	-	-	1				
	10	88	-	-	1				
	0.01-1	-	99	2.7	3				
Roots	0.01	98	-	-	1				
	0.1	94	-	-	1				
	0.01-0.1	-	104	-	2				

Conclusion

All trials submitted were considered acceptable. The application rates of some trials were slightly below 75 % of the cGAP. However, these trials were still considered acceptable since the plants were treated at a later BBCH stadium and all trials were <LOQ. For metabolite M26 the storage stability in high water content commodities (leaf with top, whole plant) was not validated as maximum storage stability was determined equal to 3 months only (see Table B.7.1-1). This was however relevant for the matrix leaf with top only, since the matrix whole plant was not considered in the assessment. Metabolite M27 was found to be instable in high starch commodities, but was not considered in the residue definition anyway.

Table B.7.3-11: N-EU residues trials with sugar beet conducted according to GAP

RESIDUES DATA SUMMARY FROM SUPERVISED TRIALS (SUMMARY)
(Application on agricultural and horticultural crops)

Federal Institute for Risk Assessment, Berlin
Federal Republic of Germany

Content of a.s. (g/kg or g/L) : 720 g/L
Formulation (e.g. WP) : EC (emulsifiable concentrate)
Commercial product (name) : BAS 656 12 H
Applicant : BASF SE

Active substance : Dimethenamid-P
Crop / crop group : Sugar Beet
Crop Code : BEAVA

Submission date : 2014-03-19

Indoors / Outdoors : Outdoors (European North)
Other a.s. in formulation :
(content and common name) :
Residues calculated as : 8.1 dimethenamid-P
8.2 M23 oxalamide
8.3 M26 thiolactic acid conjugate
8.4 M27 sulfonate
8.5 M30 sulfoxide of thiolactic acid conjugate

1	2	3	4			5	6	7	8.1	8.2	8.3	8.4	8.5	9	10
Report-No. Location incl. Postal code and date	Commodity/ Variety	Date of 1) Sowing or planting 2) Flowering 3) Harvest	Application rate per treatment			Dates of treatments or no. of treatments and last date	Growth stage at last treatment or date	Portion analysed	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	PHI (days)	Remarks
	(a)	(b)	kg as/ha	Water L/ha	kg as/hL	(c)		(a)						(d)	(e)
report 2012/1182982, study 390503 (B1019), trial L110035 (B1019 DE1), harvest trial Germany (DE) 47574 Goch-Pfalzdorf 2012-09-28	Naute	1) 2011-03-23 (sowing) 2) 3) 2011-09-20	0.64	200	0.32	2011-05-03 ⁴⁾	BBCH 16	whole plant	29.3	<0.010	<0.010	<0.010	<0.010	0	4) spraying
								leaf with top	<0.010	<0.010	<0.010	<0.010	<0.010	140	ample storage time not validated 8.3: whole plant, leaf with top
								root body	<0.010	<0.010	<0.010	<0.010	<0.010	140	8.4: root body analytical method: BASF No. L0179/02 (LC-MS/MS), LOQ(s): 0.01 mg/kg max. sample storage time in month(s): 10 (whole plant, leaf with top) 6 (root body) ASB2014-3703

1	2	3	4			5	6	7	8.1	8.2	8.3	8.4	8.5	9	10
Report-No. Location incl. Postal code and date	Commodity/ Variety	Date of 1) Sowing or planting 2) Flowering 3) Harvest	Application rate per treatment			Dates of treatments or no. of treatments and last date	Growth stage at last treatment or date	Portion analysed	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	PHI (days)	Remarks
			kg as/ha	Water L/ha	kg as/hL										
	(a)	(b)				(c)		(a)						(d)	(e)
report 2012/1182982, study 390503 (B1019), trial L110036 (B1019 AN1), harvest trial France (FR) 67160 Seebach 2012-09-28	Muraille	1) 2011-04-02 (sowing) 2) 3) 2011-09-30	0.63	190	0.33	2011-05-10 ⁴⁾	BBCH 15-17	whole plant leaf with top root body	0.71 <u><0.010</u> <u><0.010</u>	<0.010 <0.010 <0.010	<0.010 <u><0.010</u> <u><0.010</u>	<0.010 <0.010 <0.010	0.060 <u><0.010</u> <u><0.010</u>	0 136 136	4) spraying sample storage time not validated 8.3: leaf with top, whole plant 8.4: root body analytical method: BASF No. L0179/02 (LC-MS/MS), LOQ(s): 0.01 mg/kg max. sample storage time in month(s): 10 (whole plant, leaf with top) 6 (root body) ASB2014-3703
report 2012/1182982, study 390503 (B1019), trial L110037 (B1019 UK1), harvest trial United Kingdom (UK) PE22 7PQ Boston, Lincolnshire 2012-09-28	Bull Finch	1) 2011-03-25 (sowing) 2) 3) 2011-09-23	0.66	200	0.32	2011-05-10 ⁴⁾	BBCH 16	whole plant leaf with top root body	11.4 <u><0.010</u> <u><0.010</u>	<0.010 <0.010 <0.010	<0.010 <u><0.010</u> <u><0.010</u>	<0.010 <0.010 <0.010	<0.010 <u><0.010</u> <u><0.010</u>	0 136 136	4) spraying sample storage time not validated 8.3: whole plant, leaf with top 8.4: root body analytical method: BASF No. L0179/02 (LC-MS/MS), LOQ(s): 0.01 mg/kg max. sample storage time in month(s): 10 (whole plant, leaf with top) 6 (root body) ASB2014-3703

1	2	3	4			5	6	7	8.1	8.2	8.3	8.4	8.5	9	10
Report-No. Location incl. Postal code and date	Commodity/ Variety	Date of 1) Sowing or planting 2) Flowering 3) Harvest	Application rate per treatment			Dates of treatments or no. of treatments and last date	Growth stage at last treatment or date	Portion analysed	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	PHI (days)	Remarks
			kg as/ha	Water L/ha	kg as/hL										
	(a)	(b)				(c)		(a)						(d)	(e)
report 2012/1182982, study 390503 (B1019), trial L110038 (B1019 NL1), harvest trial Netherlands (NL) 6595 Ottersum 2012-09-28	Pyranja	1) 2011-04-15 (sowing) 2) 3) 2011-09-21	0.65	200	0.33	2011-05-18 ⁴⁾	BBCH 16	whole plant leaf with top root body	24.7 <u><0.010</u> <u><0.010</u>	<0.010 <0.010 <0.010	<0.010 <u><0.010</u> <u><0.010</u>	<0.010 <0.010 <0.010	<0.010 <u><0.010</u> <u><0.010</u>	0 126 126	4) spraying sample storage time not validated 8.3: whole plant, leaf with top 8.4: root body analytical method: BASF No. L0179/02 (LC-MS/MS), LOQ(s): 0.01 mg/kg max. sample storage time in month(s): 10 (whole plant, leaf with top) 6 (root body) ASB2014-3703
report 2013/1003729, study 423128 (AC/BASF/12/07), trial L120262 (AC/12/062),	Klaxon	1) 2012-03-21 (sowing) 2) 3) 2012-09-06	0.62	190	0.33	2012-05-07 ⁴⁾	BBCH 16	whole plant leaf with top	41.0 <u><0.010</u>	<0.010 <0.010	<0.010 <u><0.010</u>	<0.010 <0.010	<0.010 <u><0.010</u>	0 122	4) spraying sample storage time not validated 8.3: whole plant, leaf with top

1	2	3	4			5	6	7	8.1	8.2	8.3	8.4	8.5	9	10
Report-No. Location incl. Postal code and date	Commodity/ Variety	Date of 1) Sowing or planting 2) Flowering 3) Harvest	Application rate per treatment			Dates of treatments or no. of treatments and last date	Growth stage at last treatment or date	Portion analysed	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	PHI (days)	Remarks
			kg as/ha	Water L/ha	kg as/hL										
	(a)	(b)				(c)		(a)						(d)	(e)
harvest trial Germany (DE) 16833 Lentzke 2013-06-12								root body	<u><0.010</u>	<0.010	<u><0.010</u>	<0.010	<u><0.010</u>	122	8.4: root body analytical method: BASF No. L0179/02 (HPLC-MS/MS), LOQ(s): 0.01 mg/kg max. sample storage time in month(s): 10 (whole plant, leaf with top) 7 (root body) ASB2014-3705
report 2013/1003729, study 423128 (AC/BASF/12/07), trial L120263 (AC/12/063), harvest trial France (FR) 51420 Witry les Reims 2013-06-12	Vienna	1) 2012-03-20 (sowing) 2) 3) 2012-10-20	0.70	220	0.32	2012-05-16 ⁴⁾	BBCH 18	whole plant leaf with top root body	22.0 <u><0.010</u> <u><0.010</u>	<0.010 <0.010 <0.010	<0.010 <u><0.010</u> <u><0.010</u>	<0.010 <0.010 <0.010	<0.010 <u><0.010</u> <u><0.010</u>	0 125 125	4) spraying sample storage time not validated 8.3: whole plant, leaf with top 8.4: root body analytical method: BASF No. L0179/02 (HPLC-MS/MS), LOQ(s): 0.01 mg/kg max. sample storage time in month(s): 10 (whole plant, leaf with top) 7 (root body) ASB2014-3705
report 2013/1003729, study 423128 (AC/BASF/12/07), trial L120264	Cayman and Isabella	1) 2012-03-25 (sowing) 2) 3) 2012-09-03	0.68	210	0.32	2012-05-23 ⁴⁾	BBCH 14-16	whole plant leaf with top	29.0 <u><0.010</u>	<0.010 <0.010	<0.010 <u><0.010</u>	<0.010 <0.010	<0.010 <u><0.010</u>	0 103	4) spraying sample storage time not validated 8.3: leaf with top, whole

1	2	3	4			5	6	7	8.1	8.2	8.3	8.4	8.5	9	10
Report-No. Location incl. Postal code and date	Commodity/ Variety	Date of 1) Sowing or planting 2) Flowering 3) Harvest	Application rate per treatment			Dates of treatments or no. of treatments and last date	Growth stage at last treatment or date	Portion analysed	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	PHI (days)	Remarks
			kg as/ha	Water L/ha	kg as/hL										
	(a)	(b)				(c)		(a)						(d)	(e)
(AC/12/064), harvest trial United Kingdom (UK) CO11 2NF Essex 2013-06-12								root body	<u>≤0.010</u>	<0.010	<u>≤0.010</u>	<0.010	<u>≤0.010</u>	103	plant 8.4: root body analytical method: BASF No. L0179/02 (HPLC-MS/MS), LOQ(s): 0.01 mg/kg max. sample storage time in month(s): 10 (whole plant, leaf with top) 7 (root body) ASB2014-3705

1	2	3	4			5	6	7	8.1	8.2	8.3	8.4	8.5	9	10
Report-No. Location incl. Postal code and date	Commodity/ Variety	Date of 1) Sowing or planting 2) Flowering 3) Harvest	Application rate per treatment			Dates of treatments or no. of treatments and last date	Growth stage at last treatment or date	Portion analysed	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	PHI (days)	Remarks
	(a)	(b)				(c)		(a)						(d)	(e)
report 2013/1003729, study 423128 (AC/BASF/12/07), trial L120265 (AC/12/065), harvest trial Netherlands (NL) 6599 CJ Ven- Zelderheide 2013-06-12	Coyote	1) 2012-04-09 (sowing) 2) 3) 2012-09-17 - 2012-09-18	0.65	200	0.33	2012-05-23 ⁴⁾	BBCH 16	whole plant leaf with top root body	29.0 <u><0.010</u> <u><0.010</u>	<0.010 <0.010 <0.010	<0.010 <u><0.010</u> <u><0.010</u>	<0.010 <0.010 <0.010	<0.010 <u><0.010</u> <u><0.010</u>	0 117 117	4) spraying sample storage time not validated 8.3: whole plant, leaf with top 8.4: root body analytical method: BASF No. L0179/02 (HPLC-MS/MS), LOQ(s): 0.01 mg/kg max. sample storage time in month(s): 10 (whole plant, leaf with top) 7 (root body) ASB2014-3705

Remarks:

- (a) According to CODEX Classification / Guide
- (b) Only if relevant
- (c) Year must be indicated
- (d) Days after last application (Label pre-harvest interval, PHI, underline)
- (e) Remarks may include: Climatic conditions; Reference to analytical method and information which metabolites are included

Table B.7.3-12: S-EU residues trials with sugar beet and fodder beet conducted according to GAP

RESIDUES DATA SUMMARY FROM SUPERVISED TRIALS (SUMMARY)
(Application on agricultural and horticultural crops)

Federal Institute for Risk Assessment, Berlin
Federal Republic of Germany

Content of a.s. (g/kg or g/L) : 720 g/L
Formulation (e.g. WP) : EC (emulsifiable concentrate)
Commercial product (name) : BAS 656 12 H
Applicant : BASF SE

Active substance : Dimethenamid-P
Crop / crop group : Sugar Beet
Crop Code : BEAVA

Submission date : 2014-03-19

Indoors / Outdoors : Outdoors (European South)
Other a.s. in formulation (content and common name) :
Residues calculated as : 8.1 dimethenamid-P
8.2 M23 oxalamide
8.3 M26 thiolactic acid conjugate
8.4 M27 sulfonate
8.5 M30 sulfoxide of thiolactic acid conjugate

1	2	3	4			5	6	7	8.1	8.2	8.3	8.4	8.5	9	10
Report-No. Location incl. Postal code and date	Commodity/ Variety	Date of 1) Sowing or planting 2) Flowering 3) Harvest	Application rate per treatment			Dates of treatments or no. of treatments and last date	Growth stage at last treatment or date	Portion analysed	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	PHI (days)	Remarks
	(a)	(b)	kg as/ha	Water L/ha	kg as/hL	(c)		(a)						(d)	(e)
report 2012/1182982, study 390503 (B1019), trial L110039 (B1019 SA1), harvest trial France (FR) 64300 Castétis 2012-09-28	Koala	1) 2011-05-27 (sowing) 2) 3) 2011-10-20	0.68	210	0.32	2011-06-28 ⁴⁾	BBCH 16	whole plant	25.2	<0.010	<0.010	<0.010	<0.010	0	4) spraying
								leaf with top	<0.010	<0.010	<0.010	<0.010	<0.010	114	sample storage time not validated 8.3: whole plant, leaf with top
								root body	<0.010	<0.010	<0.010	<0.010	<0.010	114	8.4: root body analytical method: BASF No. L0179/02 (LC-MS/MS), LOQ(s): 0.01 mg/kg max. sample storage time in month(s): 9 (whole plant, leaf with top) 5 (root body) ASB2014-3703
report 2012/1182982,	Massima	1) 2011-03-07 (sowing)	0.66	200	0.33	2011-04-28 ⁴⁾	BBCH 16	whole plant	30.5	<0.010	<0.010	<0.010	<0.010	0	4) spraying

1	2	3	4			5	6	7	8.1	8.2	8.3	8.4	8.5	9	10
Report-No. Location incl. Postal code and date	Commodity/ Variety	Date of 1) Sowing or planting 2) Flowering 3) Harvest	Application rate per treatment			Dates of treatments or no. of treatments and last date	Growth stage at last treatment or date	Portion analysed	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	PHI (days)	Remarks
			kg as/ha	Water L/ha	kg as/hL										
	(a)	(b)				(c)		(a)						(d)	(e)
study 390503 (B1019), trial L110040 (B1019 IT1), harvest trial Italy (IT) 15055 Bruciata Pontecurone 2012-09-28		2) 3) 2011-08-10						leaf with top root body	<u><0.010</u> <u><0.010</u>	<0.010 <0.010	<u><0.010</u> <u><0.010</u>	<0.010 <0.010	<u><0.010</u> <u><0.010</u>	104 104	sample storage time not validated 8.3: whole plant, leaf with top 8.4: root body analytical method: BASF No. L0179/02 (LC-MS/MS), LOQ(s): 0.01 mg/kg max. sample storage time in month(s): 11 (whole plant, leaf with top) 7 (root body) ASB2014-3703
report 2013/1003729, study 423128 (AC/BASF/12/07), trial L120267 (AC/12/067), harvest trial Italy (IT) 40018 San Pietro in Casale 2013-06-12	Bruna	1) 2012-02-27 (sowing) 2) 3) 2012-08-20	0.69	210	0.33	2012-04-17 ⁴⁾	BBCH 14-16	whole plant leaf with top root body	33.0 <u><0.010</u> <u><0.010</u>	<0.010 <0.010 <0.010	<0.010 <u><0.010</u> <u><0.010</u>	<0.010 <0.010 <0.010	<0.010 <u><0.010</u> <u><0.010</u>	0 125 125	4) spraying sample storage time not validated 8.3: whole plant, leaf with top 8.4: root body analytical method: BASF No. L0179/02 (HPLC-MS/MS), LOQ(s): 0.01 mg/kg max. sample storage time in month(s): 11 (whole plant, leaf with top) 8 (root body) ASB2014-3705

RESIDUES DATA SUMMARY FROM SUPERVISED TRIALS (SUMMARY)
(Application on agricultural and horticultural crops)

Federal Institute for Risk Assessment, Berlin
Federal Republic of Germany

Content of a.s. (g/kg or g/L) : 720 g/L
Formulation (e.g. WP) : EC (emulsifiable concentrate)
Commercial product (name) : BAS 656 12 H
Applicant : BASF SE

Active substance : Dimethenamid-P
Crop / crop group : Fodder Beet
Crop Code : BEAVC

Submission date : 2014-03-19

Indoors / Outdoors : Outdoors (European South)
Other a.s. in formulation :
(content and common name) :
Residues calculated as : 8.1 dimethenamid-P
8.2 M23 oxalamide
8.3 M26 thiolactic acid conjugate
8.4 M27 sulfonate
8.5 M30 sulfoxide of thiolactic acid conjugate

1	2	3	4			5	6	7	8.1	8.2	8.3	8.4	8.5	9	10
Report-No. Location incl. Postal code and date	Commodity/ Variety	Date of 1) Sowing or planting 2) Flowering 3) Harvest	Application rate per treatment			Dates of treatments or no. of treatments and last date	Growth stage at last treatment or date	Portion analysed	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	PHI (days)	Remarks
			kg as/ha	Water L/ha	kg as/hL										
	(a)	(b)				(c)		(a)						(d)	(e)
report 2013/1003729, study 423128 (AC/BASF/12/07), trial L120266 (AC/12/066), harvest trial France (FR) 17330 Bernay Saint- Martin 2013-06-12	Starmon	1) 2012-03-26 (sowing) 2) 3) 2012-11-27	0.64	200	0.32	2012-05-23 ⁴⁾	BBCH 16	whole plant leaf with top root body	22.0 <u><0.010</u> <u><0.010</u>	<0.010 <0.010 <0.010	<0.010 <u><0.010</u> <u><0.010</u>	<0.010 <0.010 <0.010	<0.010 <u><0.010</u> <u><0.010</u>	0 142 142	4) spraying sample storage time not validated 8.3: leaf with top, whole plant 8.4: root body analytical method: BASF No. L0179/02 (HPLC-MS/MS), LOQ(s): 0.01 mg/kg max. sample storage time in month(s): 10 (whole plant, leaf with top) 6 (root body) ASB2014-3705

Remarks:

- (a) According to CODEX Classification / Guide
(b) Only if relevant

- (c) Year must be indicated
- (d) Days after last application (Label pre-harvest interval, PHI, underline)
- (e) Remarks may include: Climatic conditions; Reference to analytical method and information which metabolites are included

B.7.3.1.5 Oilseed rape

The cGAP in N- and S-EU is 1 x 0.500 kg as/ha spray application at post-emergence stage up to BBCH 18. PHI is covered by the growing period between application and harvest.

Reference:	KCA 6.3.2/1
Report	Meyer M. (2014): Residue behaviour of Quinmerac (BAS 518 H) and dimethenamid-P (BAS 656 H) in oilseed rape after treatment with BAS 830 01 H under field conditions in Germany, The Netherlands, United Kingdom, Southern France, Italy and Spain, 2013, BASF Doc ID: 2013/1335420, Study ID: 694886, ASB2014-8346
Guideline(s):	EC 1107/2009 of the European Parliament and of the Council of 21 Oct 2009, EEC 79/117, EEC 91/414, EU Regulation Regulation 544/2011 (10 June 2011) implementing Regulation No 1107/2009, EEC 7029/VI/95 rev. 5 Appendix B (July 22 1997), EEC 7525/VI/95 rev. 9 (March 2011)
Deviations:	None
GLP:	Yes
Acceptability:	The study is acceptable.
Reference:	KCA 6.3.
Report	Erdmann, H.-P. (2013): Study on the residue behaviour of BAS 656 H (dimethenamid-P) and BAS 479 H (Metazachlor) in spring oilseed rape after application of BAS 769 00 H under field condition in United Kingdom, Belgium, The Netherlands and Germany, 2012, BASF Doc ID: 2013/1003730, Study code: 422462, ASB2015-284
Guideline(s):	EC 1107/2009 of the European Parliament and of the Council of 21 Oct 2009, EEC 79/117, EEC 7029/VI/95 rev. 5 Appendix B (July 22 1997), EEC 7525/VI/95 rev. 9 (March 2011)
Deviations:	None
GLP:	Yes
Acceptability:	The study is acceptable.
Reference:	KCA 6.3.
Report	Erdmann, H.-P. (2013): Amendment No. 1 - Study on the residue behaviour of BAS 656 H (dimethenamid-P) and BAS 479 H (Metazachlor) in spring oilseed rape after application of BAS 769 00 H under field condition in United Kingdom, Belgium, The Netherlands and Germany, 2012, BASF Doc ID: 2013/1377012, Study code: 422462, ASB2015-298
Guideline(s):	EC 1107/2009 of the European Parliament and of the Council of 21 Oct 2009, EEC 79/117, EEC 7029/VI/95 rev. 5 Appendix B (July 22 1997), EEC 7525/VI/95 rev. 9 (March 2011)
Deviations:	None
GLP:	Yes
Acceptability:	The study is acceptable.

Materials and methods

Out of 11 supervised residue trials in oilseed rape, 8 trials were conducted in N-EU and 3 trials in S-EU in 2012 and 2013.

Oilseed rape plants were treated according to the GAP with 1 application at nominal rates of 0.487 - 0.605 kg as/ha. Whole plants at forage stage were harvested at 5-41 DALA, while seed and rest of the plants were harvested 69-143 DALA. The samples were stored deep frozen for max. 6 months.

All samples were analysed for dimethenamid-P and the metabolites M23, M26, M27 and M30 according to method BASF L0179/02. For a detailed method description see B.5.2.1.1.

Results and Discussion

With a few exceptions, procedural recoveries and precisions were within acceptable limits (Table B.7.3-13). Storage of oilseed rape seed samples was covered by acceptable storage stability studies for all analytes. For high water content samples, storage stability was validated for dimethenamid-P, M23, M27 and M30, but not for M26. Residue levels in individual samples are reported in Table B.7.3-14 and Table B.7.3-15. No residues were found in untreated control samples.

Table B.7.3-13: Procedural recoveries for dimethenamid-P in oilseed rape

Matrix	Fortification Level [mg/kg]	Summary recoveries							
		Recovery [%]	Mean [%]	RSD [%]	n	Recovery [%]	Mean [%]	RSD [%]	n
		Dimethenamid-p				M23			
Whole plant	0.01	112, 112, 96, 112, 92, 95, 78	100	13	7	96, 108, 107, 113, 98, 105, 84	102	10	7
	0.1	96, 109, 97	101	7	3	108, 113, 101	107	6	3
	1	109, 115	112	-	2	99, 109	104	-	2
	2	96	-	-	1	99	-	-	1
	10	113, 110, 115, 108	112	3	4	99, 100, 110, 106	104	5	4
	20	108, 106	107	-	2	100, 105	103	-	2
	30	116	-	1	1	108	-	-	1
	100	109, 102	106	-	2	-	-	-	-
Seed	0.01	116, 117, 114, 111, 115, 96, 74	106	15	7	105, 105, 106, 111, 115, 102, 84	104	9	7
	0.1	116, 117, 116, 115, 118, 96, 77	108	14	7	104, 115, 106, 106, 118, 100, 80	104	12	7
	1	116, 114	115	-	2	113, 112	113	-	2
Rest of plant	0.01	107, 111, 88, 82	97	15	4	105, 103, 102, 82	98	11	4
	0.1	113, 105, 103, 81	101	14	4	112, 102, 102, 78	99	15	4
	1	111	-	-	1	107	-	-	1
	10	108	-	-	1	106	-	-	1
	20	108	-	-	1	107	-	-	1
		M26				M27			
Whole plant	0.01	100, 105, 94, 112, 77, 85, 72	92	16	7	104, 103, 106, 108, 87, 100, 77	98	12	7
	0.1	99, 120, 91	103	14	3	99, 108, 98	102	5	3
	1	109, 112	111	-	2	110, 110	110	-	2
	2	95	-	-	1	100	-	-	1
	10	95, 111, 113, 118	109	9	4	101, 99, 105, 106	103	3	4
	20	95, 101	98	-	2	106, 104	105	-	2
	30	108	-	-	1	107	-	-	1
Seed	0.01	111, 105, 107, 103, 117, 88, 69	100	16	7	102, 102, 104, 116, 118, 84, 72	100	12	7
	0.1	102, 119, 104, 108, 120, 98, 78	104	14	7	108, 105, 109, 112, 120, 85, 75	102	6	7
	1	121, 122	122	-	2	115, 111	113	-	2
Rest of plant	0.01	97, 81, 76, 68	81	15	4	95, 92, 92, 71	88	13	4
	0.1	109, 76, 82, 78	86	18	4	106, 93, 93, 76	92	13	4
	1	89	-	-	1	97	-	-	1
	10	86	-	-	1	96	-	-	1
	20	97	-	-	1	98	-	-	1

Matrix	Fortification Level [mg/kg]	Summary recoveries									
		Recovery [%]	Mean [%]	RSD [%]	n	Recovery [%]	Mean [%]	RSD [%]	n		
		M30									
Whole plant	0.01	119, 105, 104, 110, 99, 98, 81	102	12	7						
	0.1	105, 113, 100	106	6	3						
	1	135, 95	115	-	2						
	2	99	-	-	1						
	10	97, 84, 113, 115	102	14	4						
	20	94, 97	96	-	2						
	30	109	-	-	1						
Seed	0.01	109, 116, 94, 112, 125, 86, 82	103	16	7						
	0.1	110, 119, 84, 105, 111, 91, 73	99	17	7						
	1	112, 108	110	-	2						
Rest of plant	0.01	108, 75, 77, 87	87	17	4						
	0.1	87, 77, 91, 73	82	10	4						
	1	71	-	-	1						
	10	119	-	-	1						
	20	101	-	-	1						

Conclusion

For S-EU only 3 trials were submitted. This was still considered acceptable as 8 trials were submitted for N-EU (total of 11 trials for both N-EU and S-EU) were submitted and residues in all trials were <LOQ. For metabolite M26 the storage stability in high water content commodities (whole plant) was not validated in some trials as the maximum storage stability was determined equal to 3 months only. This is however not considered relevant since the matrix whole plant was not considered in the assessment.

Table B.7.3-14: N-EU residues trials with oilseed rape conducted according to GAP

RESIDUES DATA SUMMARY FROM SUPERVISED TRIALS (SUMMARY)
(Application on agricultural and horticultural crops)

Federal Institute for Risk Assessment, Berlin
Federal Republic of Germany

Content of a.s. (g/kg or g/L) : 333 g/L
Formulation (e.g. WP) : SE (suspo-emulsion)
Commercial product (name) : BAS 830 01 H
Applicant : BASF SE

Active substance : Dimethenamid-P
Crop / crop group : Spring Rape
Crop Code : BRSNS

Submission date : 2014-08-21

Indoors / Outdoors : Outdoors (European North)
Other a.s. in formulation :
(content and common name) : 167 g/L quinmerac
Residues calculated as : 8.1 dimethenamid-P
8.2 M23 oxalamide
8.3 M26 thiolactic acid conjugate
8.4 M27 sulfonate
8.5 M30 sulfoxide of thiolactic acid conjugate

1	2	3	4			5	6	7	8.1	8.2	8.3	8.4	8.5	9	10
Report-No. Location incl. Postal code and date	Commodity/ Variety	Date of 1) Sowing or planting 2) Flowering 3) Harvest	Application rate per treatment			Dates of treatments or no. of treatments and last date	Growth stage at last treatment or date	Portion analysed	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	PHI (days)	Remarks
	(a)	(b)	kg as/ha	Water L/ha	kg as/hL	(c)		(a)						(d)	(e)
BASF DocID 2013/1335420, study 694886 (IF-13/02536186), trial L130132, decline trial Germany (DE) 49685 Emstek 2014-02-21	Makro	1) 2013-04-15 (sowing) 2) 2013-06-14 - 2013-07-09 3) 2013-08-26	0.50	200	0.25	2013-05- 31 ⁴⁾	BBCH 18	whole plant rest of plant seed(s)	7.1 <0.010 <0.010 <0.010	<0.010 <0.010 <0.010 <0.010	0.10 0.016 <0.010 <0.010	<0.010 <0.010 <0.010 <0.010	0.050 0.34 0.010 <0.010	0 10 87 87	4) spraying analytical method: BASF No. L0179/02 (HPLC-MS/MS), LOQ(s): 0.01 mg/kg max. sample storage time in month(s): 3 (whole plant, rest of plant) 1 (seeds) ASB2014-8346
BASF DocID 2013/1003730, study 422462 (IF- 12/02351240), trial L120272, decline trial Germany (DE)	Ability	1) 2012-03-21 (sowing) 2) 2012-05-25 - 2012-06-11 3) 2012-08-17	0.57	190	0.30	2012-05- 10 ⁴⁾	BBCH 14-51	whole plant rest of plant seed(s)	27.2 <0.010 <0.010 <0.010	<0.010 <0.010 <0.010 <0.010	0.12 0.23 <0.010 <0.010	<0.010 <0.010 <0.010 <0.010	0.044 2.9 0.086 <0.010	0 8 99 99	4) spraying sample storage time not validated 8.3: whole plant analytical method: BASF No. L0179/02

1	2	3	4			5	6	7	8.1	8.2	8.3	8.4	8.5	9	10
Report-No. Location incl. Postal code and date	Commodity/ Variety	Date of 1) Sowing or planting 2) Flowering 3) Harvest	Application rate per treatment			Dates of treatments or no. of treatments and last date	Growth stage at last treatment or date	Portion analysed	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	PHI (days)	Remarks
			kg as/ha	Water L/ha	kg as/hL										
	(a)	(b)				(c)		(a)						(d)	(e)
16833 Lentzke 2013-08-23															(HPLC-MS/MS), LOQ(s): 0.01 mg/kg max. sample storage time in month(s): 5 (whole plant, rest of plant) 1 (seeds) ASB2015-284 ASB2015-298 (amendment)
BASF DocID 2013/1003730, study 422462 (IF- 12/02351240), trial L120273, decline trial United Kingdom (UK) CO11 2NF Lawford, Manningtree, Essex 2013-08-23	Delight	1) 2012-03-28 (sowing) 2) 2012-05-30 - 2012-07-03 3) 2012-09-03	0.57	190	0.30	2012-05- 25 ⁴⁾	BBCH 15-16	whole plant rest of plant seed(s)	23.4 <0.010 <0.010 <0.010	<0.010 <0.010 <0.010 <0.010	0.21 0.13 <0.010 <0.010	<0.010 <0.010 <0.010 <0.010	0.10 2.8 0.016 <0.010	0 5 101 101	4) spraying sample storage time not validated 8.3: whole plant analytical method: BASF No. L0179/02 (HPLC-MS/MS), LOQ(s): 0.01 mg/kg max. sample storage time in month(s): 5 (whole plant, rest of plant) 1 (seeds) ASB2015-284 ASB2015-298 (amendment)
BASF DocID 2013/1003730, study 422462 (IF- 12/02351240), trial L120274, decline trial	Heros	1) 2012-05-15 (sowing) 2) 2012-07-20 - 2012-07-30 3) 2012-09-12	0.61	200	0.30	2012-07- 03 ⁴⁾	BBCH 18	whole plant rest of plant seed(s)	30.2 <0.010 <0.010 <0.010	<0.010 <0.010 <0.010 <0.010	0.080 0.029 <0.010 <0.010	<0.010 <0.010 <0.010 <0.010	0.037 2.6 0.045 <0.010	0 15 71 71	4) spraying analytical method: BASF No. L0179/02 (HPLC-MS/MS), LOQ(s): 0.01 mg/kg

1	2	3	4			5	6	7	8.1	8.2	8.3	8.4	8.5	9	10
Report-No. Location incl. Postal code and date	Commodity/ Variety	Date of 1) Sowing or planting 2) Flowering 3) Harvest	Application rate per treatment			Dates of treatments or no. of treatments and last date	Growth stage at last treatment or date	Portion analysed	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	PHI (days)	Remarks
			kg as/ha	Water L/ha	kg as/hL										
	(a)	(b)				(c)		(a)						(d)	(e)
Belgium (BE) 3534 Halen 2013-08-23															max. sample storage time in month(s): 3 (whole plant, rest of plant) 1 (seeds) ASB2015-284 ASB2015-298 (amendment)
BASF DocID 2013/1003730, study 422462 (IF- 12/02351240), trial L120275, decline trial Netherlands (NL) 6599 CJ Ven- Zelderheide 2013-08-23	Heros	1) 2012-04-24 (sowing) 2) 2012-07-01 - 2012-07-15 3) 2012-09-05	0.61	200	0.30	2012-06- 16 ⁴⁾	BBCH 18	whole plant rest of plant seed(s)	40.8 <0.010 <u><0.010</u> <u><0.010</u>	<0.010 <0.010 <0.010 <0.010	0.24 0.029 <u><0.010</u> <u><0.010</u>	<0.010 <0.010 <0.010 <0.010	0.096 2.9 <u>0.060</u> <u><0.010</u>	3 32 81 81	4) spraying sample storage time not validated 8.3: whole plant analytical method: BASF No. L0179/02 (HPLC-MS/MS), LOQ(s): 0.01 mg/kg max. sample storage time in month(s): 4 (whole plant, rest of plant) 1 (seeds) ASB2015-284 ASB2015-298 (amendment)

RESIDUES DATA SUMMARY FROM SUPERVISED TRIALS (SUMMARY)
(Application on agricultural and horticultural crops)

Federal Institute for Risk Assessment, Berlin
Federal Republic of Germany

Content of a.s. (g/kg or g/L) : 333 g/L
Formulation (e.g. WP) : SE (suspo-emulsion)
Commercial product (name) : BAS 830 01 H
Applicant : BASF SE

Active substance : Dimethenamid-P
Crop / crop group : Winter Rape
Crop Code : BRSNW

Submission date : 2014-08-21

Indoors / Outdoors : Outdoors (European North)
Other a.s. in formulation :
(content and common name) : 167 g/L quinmerac
Residues calculated as : 8.1 dimethenamid-P
8.2 M23 oxalamide
8.3 M26 thiolactic acid conjugate
8.4 M27 sulfonate
8.5 M30 sulfoxide of thiolactic acid conjugate

1	2	3	4			5	6	7	8.1	8.2	8.3	8.4	8.5	9	10
Report-No. Location incl. Postal code and date	Commodity/ Variety	Date of 1) Sowing or planting 2) Flowering 3) Harvest	Application rate per treatment			Dates of treatments or no. of treatments and last date	Growth stage at last treatment or date	Portion analysed	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	PHI (days)	Remarks
	(a)	(b)	kg as/ha	Water L/ha	kg as/hL	(c)		(a)						(d)	(e)
BASF DocID 2013/1335420, study 694886 (IF-13/02536186), trial L130133, decline trial Netherlands (NL) 9695 AK Bellingwohlde 2014-02-21	Expower	1) 2012-09-07 (sowing) 2) 2013-05-15 - 2013-06-04 3) 2013-08-09	0.49	200	0.25	2013-04-18 ⁴⁾	BBCH 18	whole plant rest of plant seed(s)	4.8 <0.010 <0.010 <0.010	<0.010 <0.010 <0.010 <0.010	0.23 0.053 <0.010 <0.010	<0.010 <0.010 <0.010 <0.010	0.11 0.61 <0.010 <0.010	0 11 110 110	4) spraying sample storage time not validated 8.3: whole plant analytical method: BASF No. L0179/02 (HPLC-MS/MS), LOQ(s): 0.01 mg/kg max. sample storage time in month(s): 5 (whole plant, rest of plant) 1 (seeds) ASB2014-8346
BASF DocID 2013/1335420, study 694886 (IF-13/02536186), trial L130134, decline trial	Cabernet	1) 2012-08-13 (sowing) 2) 2013-05-18 - 2013-06-28 3) 2013-08-20	0.51	200	0.25	2013-04-02 ⁴⁾	BBCH 18	whole plant rest of plant	16.0 <0.010 <0.010	<0.010 <0.010 <0.010	0.020 <0.010 <0.010	<0.010 <0.010 <0.010	<0.010 0.098 <0.010	0 28 139	4) spraying sample storage time not validated 8.3: whole plant

1	2	3	4			5	6	7	8.1	8.2	8.3	8.4	8.5	9	10
Report-No. Location incl. Postal code and date	Commodity/ Variety	Date of 1) Sowing or planting 2) Flowering 3) Harvest	Application rate per treatment			Dates of treatments or no. of treatments and last date	Growth stage at last treatment or date	Portion analysed	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	PHI (days)	Remarks
	(a)	(b)	kg as/ha	Water L/ha	kg as/hL	(c)		(a)						(d)	(e)
United Kingdom (UK) OX17 IDX Banbury, Oxfordshire 2014-02-21								seed(s)	<u><0.010</u>	<0.010	<u><0.010</u>	<0.010	<u><0.010</u>	139	analytical method: BASF No. L0179/02 (HPLC-MS/MS), LOQ(s): 0.01 mg/kg max. sample storage time in month(s): 5 (whole plant, rest of plant) 1 (seeds) ASB2014-8346
BASF DocID 2013/1335420, study 694886 (IF-13/02536186), trial L130135, decline trial Germany (DE) 51399 Burscheid 2014-02-21	Visby	1) 2012-09-01 (sowing) 2) 2013-04-30 - 2013-05-14 3) 2013-08-03	0.52	210	0.25	2013-03-08 ⁴⁾	BBCH 18	whole plant rest of plant seed(s)	8.6 <u><0.010</u> <u><0.010</u> <u><0.010</u>	<0.010 <u><0.010</u> <0.010	0.19 <u><0.010</u> <u><0.010</u>	<0.010 <u><0.010</u> <0.010	0.13 0.056 <u>0.015</u> <u><0.010</u>	0 41 143 143	4) spraying sample storage time not validated 8.3: whole plant analytical method: BASF No. L0179/02 (HPLC-MS/MS), LOQ(s): 0.01 mg/kg max. sample storage time in month(s): 6 (whole plant, rest of plant) 1 (seeds) ASB2014-8346

Remarks:

- (a) According to CODEX Classification / Guide
- (b) Only if relevant
- (c) Year must be indicated
- (d) Days after last application (Label pre-harvest interval, PHI, underline)
- (e) Remarks may include: Climatic conditions; Reference to analytical method and information which metabolites are included

Table B.7.3-15: S-EU residues trials with oilseed rape conducted according to GAP

RESIDUES DATA SUMMARY FROM SUPERVISED TRIALS (SUMMARY)
(Application on agricultural and horticultural crops)

Federal Institute for Risk Assessment, Berlin
Federal Republic of Germany

Content of a.s. (g/kg or g/L) : 333 g/L
Formulation (e.g. WP) : SE (suspo-emulsion)
Commercial product (name) : BAS 830 01 H
Applicant : BASF SE

Active substance : Dimethenamid-P
Crop / crop group : Spring Rape
Crop Code : BRSNS

Submission date : 2014-08-21

Indoors / Outdoors : Outdoors (European South)
Other a.s. in formulation :
(content and common name) : 167 g/L quinmerac
Residues calculated as : 8.1 dimethenamid-P
8.2 M23 oxalamide
8.3 M26 thiolactic acid conjugate
8.4 M27 sulfonate
8.5 M30 sulfoxide of thiolactic acid conjugate

1	2	3	4			5	6	7	8.1	8.2	8.3	8.4	8.5	9	10
Report-No. Location incl. Postal code and date	Commodity/ Variety	Date of 1) Sowing or planting 2) Flowering 3) Harvest	Application rate per treatment			Dates of treatments or no. of treatments and last date	Growth stage at last treatment or date	Portion analysed	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	PHI (days)	Remarks
	(a)	(b)	kg as/ha	Water L/ha	kg as/hL	(c)		(a)						(d)	(e)
BASF DocID 2013/1335420, study 694886 (IF- 13/02536186), trial L130138, decline trial Italy (IT) 25030 Pompiano 2014-02-21	Mosaik	1) 2013-04-22 (sowing) 2) 2013-06-29 - 2013-07-04 3) 2013-08-05	0.49	200	0.25	2013-05-28 ⁴⁾	BBCH 18	whole plant rest of plant seed(s)	7.6 <0.010 <0.010 <0.010	<0.010 <0.010 <0.010 <0.010 <0.010	0.33 <0.010 <0.010 <0.010 <0.010	<0.010 <0.010 <0.010 <0.010 <0.010	0.39 0.28 <0.010 <0.010	0 24 69 69	4) spraying sample storage time not validated 8.3: whole plant analytical method: BASF No. L0179/02 (HPLC-MS/MS), LOQ(s): 0.01 mg/kg max. sample storage time in month(s): 4 (whole plant, rest of plant) 1 (seeds) ASB2014-8346
BASF DocID 2013/1335420, study 694886	Jura	1) 2013-03-26 (sowing) 2) 2013-06-05	0.50	200	0.25	2013-04-26 ⁴⁾	BBCH 18	whole plant	37.0 <0.010	<0.010 <0.010	0.32 <0.010	<0.010 <0.010	0.31 0.079	0 26	4) spraying sample storage time not

1	2	3	4			5	6	7	8.1	8.2	8.3	8.4	8.5	9	10
Report-No. Location incl. Postal code and date	Commodity/ Variety	Date of 1) Sowing or planting 2) Flowering 3) Harvest	Application rate per treatment			Dates of treatments or no. of treatments and last date	Growth stage at last treatment or date	Portion analysed	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	PHI (days)	Remarks
			kg as/ha	Water L/ha	kg as/hL										
	(a)	(b)				(c)		(a)						(d)	(e)
(IF- 13/02536186), trial L130139, decline trial Spain (ES) 29700 Velez Malaga 2014-02-21		- 2013-07-06 3) 2013-08-05						rest of plant	<u><0.010</u>	<0.010	<u><0.010</u>	<0.010	<u><0.010</u>	101	validated 8.3: whole plant
								seed(s)	<u><0.010</u>	<0.010	<u><0.010</u>	<0.010	<u><0.010</u>	101	analytical method: BASF No. L0179/02 (HPLC-MS/MS), LOQ(s): 0.01 mg/kg max. sample storage time in month(s): 5 (whole plant, rest of plant) 1 (seeds) ASB2014-8346

RESIDUES DATA SUMMARY FROM SUPERVISED TRIALS (SUMMARY)
(Application on agricultural and horticultural crops)

Federal Institute for Risk Assessment, Berlin
Federal Republic of Germany

Content of a.s. (g/kg or g/L) : 333 g/L
Formulation (e.g. WP) : SE (suspo-emulsion)
Commercial product (name) : BAS 830 01 H
Applicant : BASF SE

Active substance : Dimethenamid-P
Crop / crop group : Winter Rape
Crop Code : BRSNW

Submission date : 2014-08-21

Indoors / Outdoors : Outdoors (European South)
Other a.s. in formulation :
(content and common name) : 167 g/L quinmerac
Residues calculated as : 8.1 dimethenamid-P
8.2 M23 oxalamide
8.3 M26 thiolactic acid conjugate
8.4 M27 sulfonate
8.5 M30 sulfoxide of thiolactic acid conjugate

1	2	3	4			5	6	7	8.1	8.2	8.3	8.4	8.5	9	10
Report-No. Location incl. Postal code and date	Commodity/ Variety	Date of 1) Sowing or planting 2) Flowering 3) Harvest	Application rate per treatment			Dates of treatments or no. of treatments and last date	Growth stage at last treatment or date	Portion analysed	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	Residues (mg/kg)	PHI (days)	Remarks
	(a)	(b)	kg as/ha	Water L/ha	kg as/hL	(c)		(a)						(d)	(e)
BASF DocID 2013/1335420, study 694886 (IF-13/02536186), trial L130136, decline trial France (FR) 47120 Levignac de Guyenne 2014-02-21	Atenzo	1) 2012-09-27 (sowing) 2) 2013-04-15 - 2013-05-10 3) 2013-07-10	0.51	200	0.25	2013-03-29 ⁴⁾	BBCH 18	whole plant	0.36 <0.010	<0.010 <0.010	0.98 <0.010	<0.010 <0.010	0.23 0.18	0 18	4) spraying
								rest of plant	<0.010	<0.010	<0.010	<0.010	<0.010	103	sample storage time not validated 8.3: whole plant
								seed(s)	<0.010	<0.010	<0.010	<0.010	<0.010	103	analytical method: BASF No. L0179/02 (HPLC-MS/MS), LOQ(s): 0.01 mg/kg max. sample storage time in month(s): 5 (whole plant, rest of plant) 2 (seeds) ASB2014-8346

Remarks:

- (a) According to CODEX Classification / Guide
- (b) Only if relevant
- (c) Year must be indicated
- (d) Days after last application (Label pre-harvest interval, PHI, underline)
- (e) Remarks may include: Climatic conditions; Reference to analytical method and information which metabolites are included

B.7.3.1.6 Studies not considered acceptable

The following studies were generally not considered as acceptable as they were either performed with racemic dimethenamid, did not include the metabolites considered in the residue definition(s), or were not conducted according to the GAP.

Table B.7.3-16: List of field trials, which do not fulfil requirements

Raunft, E.; Benz, A.; Mackenroth, C., Study on the residue behaviour of dimethenamid in maize after treatment with BAS 656 02 H and BAS 656 07 H under field conditions in Belgium, Germany and the Netherlands, 1998 99/10005 ! NEU/HR/04/98, MET1999-754
Meumann, H.; Benz, A.; Mackenroth, C., Study on the residue behaviour of dimethenamid in sugar beet after application with BAS 656 02 H and BAS 656 07 H under field conditions in Germany, France and the Netherlands, 1998, 99/10006 ! NEU/HR/05/98, MET1999-755
Schulz, H., Determination of the residues of dimethenamid and dimethenamid-P in grain maize following treatment with BAS 656 02 H and BAS 656 07 H under field conditions in Italy and Southern France, 1998 99/10007 ! IF-98/04958-00, MET1999-756
Hertl, P., Residues of SAN 582 H in corn (grain) following treatment with SAN 582 H 900 EC in France, 1990 (residues at harvest) - Final report [Northern France] 91/11897 ! R 9929 ! BS 2133, RIP1999-836
Hertl, P., Residues of SAN 582 H in corn (grain) following treatment with SAN 582 H 900 EC in France, 1990 (residues at harvest) - Final report [Northern France] 91/11895 ! R 9930 ! BS 2132, RIP1999-837
Hertl, P., Residues of SAN 582 H in corn (grain) following treatment with SAN 582 H 900 EC in France, 1990 (residues at harvest) - Final report [Northern France] 91/11894 ! R 9931 ! BS 2130, RIP1999-838
Hertl, P., Residues of SAN 582 H in corn taken at different time intervals following treatment with SAN 582 H 900 EC in France, 1990 (degradation curve) - Final report [Northern France] 91/11888 ! R 9903 ! BS 2142, RIP1999-839
Hertl, P., Residues of SAN 582 H in corn (grain) taken at different time intervals following treatment with SAN 582 H 900 EC in France, 1990 (degradation curve) - Final report [Northern France] 91/11896 ! R 9905 ! BS 2145, RIP1999-840
Hertl, P., Residues of SAN 582 H in corn (grain) taken at different time intervals following treatment with SAN 582 H 900 EC in France, 1990 (degradation curve) - Final report [Northern France] 91/11889 ! R 9904 ! BS 2143, RIP1999-841
Perny, A., Determination of residue of SAN 582 H at harvest in corn after treatment with SAN 582 H 900 EC in France, 1991 [Southern France] 97/11159 ! RF 108103, RIP1999-842
Perny, A., Determination of residue of SAN 582 H at harvest in corn after treatment with SAN 582 H 900 EC in France, 1991 [Southern France] 97/11160 ! RF 108102, RIP1999-843
Perny, A., Determination of residue of SAN 582 H at harvest in corn after treatment with SAN 582 H 900 EC in France, 1991 [Southern France] 97/11161 ! RF 108101, RIP1999-844
Hertl, P., Residues of SAN 582 H in corn (zea mays) taken at different time intervals following treatment with SAN 582 H 900 EC in Italy, 1990 (degradation curve) - Final report 91/11893 ! R 9973 ! BS 2243, RIP1999-845
Hertl, P., Residues of SAN 582 H in corn (zea mays) taken at different time intervals following treatment with SAN 582 H 900 EC in Italy, 1990 (degradation curve) - Final report 91/11891 ! R 9971 ! BS 2283, RIP1999-846
Hertl, P., Residues of SAN 582 H in corn (zea mays) taken at different time intervals following treatment with SAN 582 H 900 EC in Italy, 1990 (degradation curve) - Final report 91/11892 ! R 9972 ! BS 2284, RIP1999-847
Hertl, P., Residues of SAN 582 H in corn (zea mays) taken at different time intervals following treatment with SAN 582 H 900 EC in Italy, 1990 (degradation curve) - Final report 91/11890 ! R 9970 ! BS 2282, RIP1999-848

Hertl, P.; Fricker, P., Determination of residues of dimethenamid in corn taken at different time intervals after the application with SAN 582 H 900 EC under field conditions in Germany 1991 (degradation curve) 95/11381 ! TDS BS4775, RIP1999-849
Fricker, P.; Hertl, P., Determination of residues of dimethenamid in corn (zea mais) after application with SAN 582 H 900 EC under field conditions in Germany, 1993 (degradation curve) 94/10643 ! R 10279, RIP1999-850
Hertl, P.; Vogler, F., Determination of residues of dimethenamid in corn taken at different time intervals after the application with SAN 582 H 900 EC under field conditions in Switzerland 1991 (degradation curve) 94/10861 ! TDS BS 4753, RIP1999-851
Kaethner, M., Determination of residues of dimethenamid at various timings in maize (zea mays) after application with SAN 582 H 900 EC under field conditions in Spain, 1992 93/11660 ! TDS BS 3935, RIP1999-852
Puy, E.; Wasser, C., Determination of residues of dimethenamid in corn treated with SAN 582 H 900 EC applied under field conditions in Greece, 1992 (residues at harvest) 93/11617 ! RF 3094 02 ! TDS BS 4207, RIP1999-853
Puy, E.; Wasser, C., Determination of residues of dimethenamid in corn treated with SAN 582 H 900 EC applied under field conditions in Greece, 1992 (residues at harvest) 93/11644 ! R GR92 ! TDS BS 4206 ! RF 3094 01, RIP1999-854
Dorn, C.; Fricker, P., Determination of dimethenamid residues in sugarbeets (beta vulgaris) matrices after application of FRONTIER or of a tank-mixture of FRONTIER, Trammat and Betanal, in France, 1995 (DC, RAH) 96/11031 ! R95-002 ! TDS BS 8789, RIP1999-856
Fuchs, A.; Fegert, A.; Mackenroth, C., Determination of dimethenamid residues in sugar beets matrices after application of FRONTIER under field conditions in Germany and Switzerland, 1996 98/11306 ! R96-014, RIP1999-857

B.7.3.2 MRL application

B.7.3.2.1 Welsh/spring onion

The cGAP in N- and S-EU is 1 x 0.864 kg as/ha spray application at post-emergence stage up to BBCH 14. PHI is covered by the growing period between application and harvest.

Reference:	KCA 6.3.
Report	Erdmann, H.P. (2014). Study on the residue behaviour of BAS 656 H (dimethenamid-P) in Welsh/spring onion (EU-N) and leek (EU-S) after application of BAS 656 12 H under field condition in Germany, Belgium, Northern and Southern France United Kingdom, Greece, Italy and Spain, 2013, Study code: 428107, Doc ID 2013/1335411, ASB2015-291
Guideline(s):	EC 1107/2009 of the European Parliament and of the Council of 21 Oct 2009, EEC 79/117, EEC 7029/VI/95 rev. 5 Appendix B (July 22 1997), EEC 7525/VI/95 rev. 9 (March 2011)
Deviations:	None
GLP:	Yes
Acceptability:	The study is acceptable.

Materials and methods

A total of 4 supervised residue trials (all N-EU) conducted in 2013 were provided by the applicant. Spring onion were treated according to the GAP with 1 application at nominal rates of 0.792 - 0.893 kg as/ha. Plants were harvested at 28-43 DALA. The samples were stored deep frozen for max. 8 months.

All samples were analysed for dimethenamid-P and metabolites M23, M26, M27 and M30 according to method BASF L0179/02. For a detailed method description see B.5.2.1.1.

Results and Discussion

Procedural recoveries and precisions were within acceptable limits (Table B.7.3-17). Storage of spring onion samples was validated for dimethenamid-P, M23, M27 and M30, but not for M26. Residue levels in individual samples are reported in Table B.7.3-18. No residues were found in untreated control samples.

Table B.7.3-17: Procedural recoveries for dimethenamid-P and metabolites in Welsh/spring onion

Matrix	Fortification Level [mg/kg]	Summary recoveries							
		Recovery [%]	Mean [%]	RSD [%]	n	Recovery [%]	Mean [%]	RSD [%]	n
		Dimethenamid-P				M23			
Whole plant	0.01	112, 106, 108	109	3	3	105, 81, 102	96	14	3
	1	105, 99	102	-	2	95, 97	96	-	2
	25	94	-	-	1		-	-	1
		M26				M27			
Whole plant	0.01	106 ,98, 101	102	4	3	107, 90, 107	101	10	3
	1	93, 103	98	-	2	93, 97	95	-	2
		M30							
Whole plant	0.01	102, 103, 103	103	1	3				
	1	98, 95	97	7	-				

Conclusion

Residues were > 0.01 mg/kg per analyte in some samples. However, sample storage time for metabolite M26 is not covered by storage stability studies evaluated in Vol. 3, section B.7.1.1 of the RAR. Trials for S-EU were not provided.

Table B.7.3-18: N-EU residues trials with Welsh/spring onion conducted according to GAP

Reference:	Study on the residue behaviour of BAS 656 H (dimethenamid-P) in welsh/spring onion (EU-N) and leek (EU-S) after application of BAS 656 12 H under field condition in Germany, Belgium, Northern and Southern France United Kingdom, Greece, Italy and Spain, 2013 Erdmann, H.P., 2014; Study code: 428107, Doc ID 2013/1335411, ASB2015-291				
GLP:	Yes	Sample storage conditions:	5 - 7 month at -18 °C		
Crop/crop group:	Welsh onion (ALLFI) Spring onion (ALLKU)	Analytical method:	BASF No. L0179/02		
Indoor/Outdoor:	Outdoors (NEU)	Limit of Quantification (mg/kg):	0.01 mg/kg		
Formulation:	EC	Limit of Detection (mg/kg):	-		
Content of active substance (g/kg or g/L):	720 g/L	Residues calculated as:	(1) dimethenamid-P (2) M23 oxalamide (3) M26 thiolactic acid conjugate (4) M27 sulfonate (5) M30 sulfoxide of thiolactic acid conjugate		

Trial No./ Location/ Year	Commodity/ Variety	Date of 1.Sowing or planting 2.Flowering 3. Harvest	Application rate per treatment			Dates of treatment or number and last date	Growth stage at last treatment	Portion analysed	Residues (mg/kg)					PHI (days)	Remarks
			g a.s./ha	Water (L/ha)	g a.s./hL				(1)	(2)	(3)	(4)	(5)		
BASF DocID 2013/1335411, study 428107 (PTRL Europe ID P 3082 G), trial L130450 (AC/13/121), harvest trial Germany (DE) 16833 Lentzke 2014-04-29	Welsh onion/ Damast	1) 2013-04-19 (planting) 2) 3)2013-08-05	0.85	200	0.43	2013-07-01	BBCH 14-15	whole plant	1.5 <u><0.010</u>	<0.010 <0.010	<0.010 <u><0.010</u>	<0.010 <0.010	<0.010 <u>0.016</u>	0 35	spraying sample storage time not validated 3: whole plant analytical method: BASF No. L0179/02 (HPLC- MS/MS), LOQ(s): all 0.01 mg/kg max. sample storage time in month(s): 7 ASB2015-291

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Trial No./ Location/ Year	Commodity/ Variety	Date of 1.Sowing or planting 2.Flowering 3. Harvest	Application rate per treatment			Dates of treatment or number and last date	Growth stage at last treatment	Portion analysed	Residues (mg/kg)					PHI (days)	Remarks
			g a.s./ha	Water (L/ha)	g a.s./hL				(1)	(2)	(3)	(4)	(5)		
BASF DocID 2013/1335411, study 428107 (PTRL Europe ID P 3082 G), trial L130451 (AC/13/122), harvest trial Belgium (BE) 3534 Halen, Limburg 2014-04-29	Spring onion/ Parade	1) 2013-06-01 (sowing) 2) 3) 2013-08-01	0.89	210	0.43	2013-06-26	BBCH 13	whole plant	2.7 <u><0.010</u>	<0.010 <0.010	<0.010 <u><0.010</u>	<0.010 <0.010	<0.010 <u><0.010</u>	0 36	spraying sample storage time not validated 3: whole plant analytical method: BASF No. L0179/02 (HPLC- MS/MS), LOQ(s): all 0.01 mg/kg max. sample storage time in month(s): 8 ASB2015-291
BASF DocID 2013/1335411, study 428107 (PTRL Europe ID P 3082 G), trial L130452 (AC/13/123), harvest trial France (FR) 37340 Ambillou, Centre/Indre et Loire 2014-04-29	Spring onion/ Cristoball	1) 2013-04-08 (sowing) 2) 3) 2013-07-24	0.79	180	0.43	2013-06-26	BBCH 13	whole plant	2.6 <u><0.010</u>	<0.010 <0.010	<0.010 <u><0.010</u>	<0.010 <0.010	<0.010 <u>0.034</u>	0 28	spraying sample storage time not validated 3: whole plant analytical method: BASF No. L0179/02 (HPLC- MS/MS), LOQ(s): all 0.01 mg/kg max. sample storage time in month(s): 8 ASB2015-291

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Trial No./ Location/ Year	Commodity/ Variety	Date of 1.Sowing or planting 2.Flowering 3. Harvest	Application rate per treatment			Dates of treatment or number and last date	Growth stage at last treatment	Portion analysed	Residues (mg/kg)					PHI (days)	Remarks
			g a.s./ha	Water (L/ha)	g a.s./hL				(1)	(2)	(3)	(4)	(5)		
BASF DocID 2013/1335411, study 428107 (PTRL Europe ID P 3082 G), trial L130453 (AC/13/124), harvest trial United Kingdom (UK) CO11 2NF Lawford, Manningtree, Essex 2014-04-29	Spring onion/ White Lisbon	1) 2013-07-26 (sowing) 2) 3) 2013-11-07 - 2013-11-21	0.81	190	0.43	2013-09-25	BBCH 13	whole plant	11.0 <u><0.010</u>	<0.010 <0.010	<0.010 <u><0.010</u>	<0.010 <0.010	<0.010 <u><0.010</u>	0 43	spraying sample storage time not validated 3: whole plant analytical method: BASF No. L0179/02 (HPLC- MS/MS), LOQ(s): all 0.01 mg/kg max. sample storage time in month(s): 5 ASB2015-291

B.7.3.2.2 Melon

The cGAP in N-EU is 1 x 0.864 kg as/ha spray application at post-emergence stage up to BBCH 16. PHI is covered by the growing period between application and harvest.

Reference:	KCA 6.3.
Report	Roche, A. (2014): Study on the residue behaviour of BAS 656 H (dimethenamid-P) after soil treatment with BAS 656 12 H in melon and pumpkin under field conditions in Northern Europe, 2013, Study code: 428112, Doc ID 2013/1335406, ASB2015-286
Guideline(s):	EC 1107/2009 of the European Parliament and of the Council of 21 Oct 2009, EEC 79/117, EEC 7029/VI/95 rev. 5 Appendix B (July 22 1997), EEC 7525/VI/95 rev. 9 (March 2011)
Deviations:	None
GLP:	Yes
Acceptability:	The study is acceptable.

Materials and methods

A total of 4 supervised residue trials (all N-EU) conducted in 2013 were provided by the applicant. Melons were treated according to the GAP with 1 application at nominal rates of 0.878 - 0.986 kg as/ha. Plants were harvested at 67-96 DALA. The samples were stored deep frozen for max. 8 months. All samples were analysed for dimethenamid-P and metabolites M23, M26, M27 and M30 according to method BASF L0179/02. For a detailed method description see B.5.2.1.1.

Results and Discussion

Procedural recoveries were with the exception of M26 at LOQ level within acceptable limits (Table B.7.3-19). Storage of melon samples was validated for dimethenamid-P, M23, M27 and M30, but not for M26. Residue levels in individual samples are reported in Table B.7.3-20. No residues were found in untreated control samples.

Table B.7.3-19: Procedural recoveries for dimethenamid-P and metabolites in melon

Matrix	Fortification Level [mg/kg]	Summary recoveries							
		Recovery [%]	Mean [%]	RSD [%]	n	Recovery [%]	Mean [%]	RSD [%]	n
		Dimethenamid-P				M23			
Melon	0.01	97	-	-	1	100	-	-	1
	0.1	103	-	-	1	98	-	-	1
	5	117	-	-	1	-	-	-	1
		M26				M27			
Melon	0.01	65	-	-	1	109	-	-	1
	1	97	-	-	1	100	-	-	1
		M30							
Melon	0.01	119	-	-	1				
	1	92	-	-	1				

Conclusion

Residues were <0.01 mg/kg per analyte in all samples. However, the sample storage time for metabolite M26 is not covered by storage stability studies evaluated in Vol. 3 CA, section B.7.1.1 of the RAR.

Table B.7.3-20: N-EU residues trials with melon conducted according to GAP

Reference:	Study on the residue behaviour of BAS 656 H (dimethenamid-P) after soil treatment with BAS 656 12 H in melon and pumpkin under field conditions in Northern Europe, 2013, Roche, A., 2014; Study code: 428112, Doc ID 2013/1335406, ASB2015-286				
GLP:	Yes	Sample storage conditions:			4 - 8 month at -18 °C
Crop/crop group:	Melon (CUMME)	Analytical method:			BASF No. L0179/02
Indoor/Outdoor:	Outdoors (NEU)	Limit of Quantification (mg/kg):			0.01 mg/kg
Formulation:	EC	Limit of Detection (mg/kg):			-
Content of active substance (g/kg or g/L):	720 g/L	Residues calculated as:			(1) dimethenamid-P (2) M23 oxalamide (3) M26 thiolactic acid conjugate (4) M27 sulfonate (5) M30 sulfoxide of thiolactic acid conjugate

Trial No./ Location/ Year	Commodity/ Variety	Date of 1.Sowing or planting 2.Flowering 3. Harvest	Application rate per treatment			Dates of treatment or number and last date	Growth stage at last treatment	Portion analysed	Residues (mg/kg)					PHI (days)	Remarks
			g a.s./ha	Water (L/ha)	g a.s./hL				(1)	(2)	(3)	(4)	(5)		
BASF DocID 2013/1335406, study 428112 (B3137), trial L130548, harvest trial Germany (DE) 69124 Heidelberg 2014-08-11	Stellio	1) 2013-07-12 (planting) 2) 2013-08-01 - 2013-09-01 3) 2013-09-20	0.99	230	0.43	2013-07-12	BBCH 13-14	fruit	<0.010	<0.010	<0.010	<0.010	<0.010	70	spraying sample storage time not validated 3: fruit analytical method: BASF No. L0179/02 (HPLC- MS/MS), LOQ(s): all 0.01 mg/kg max. sample storage time in month(s): 8 ASB2015-286

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Trial No./ Location/ Year	Commodity/ Variety	Date of 1.Sowing or planting 2.Flowering 3. Harvest	Application rate per treatment			Dates of treatment or number and last date	Growth stage at last treatment	Portion analysed	Residues (mg/kg)					PHI (days)	Remarks
			g a.s./ha	Water (L/ha)	g a.s./hL				(1)	(2)	(3)	(4)	(5)		
BASF DocID 2013/1335406, study 428112 (B3137), trial L130549, harvest trial Poland (PL) 99-122 Góra Swietej Malgorzaty 2014-08-11	Oliwin	1) 2013-06-14 (planting) 2) 3) 2013-08-16 - 2013-09-03	0.88	200	0.43	2013-06-14	BBCH 10-11	fruit	<0.010	<0.010	<0.010	<0.010	<0.010	67	spraying sample storage time not validated 3: fruit analytical method: BASF No. L0179/02 (HPLC- MS/MS), LOQ(s): all 0.01 mg/kg max. sample storage time in month(s): 6 ASB2015-286
BASF DocID 2013/1335406, study 428112 (B3137), trial L130550, harvest trial France (FR) 49480 St Sylvain d'Anjou 2014-08-11	Galia	1) 2013-06-26 (planting) 2 2013-07-10 - 2013-08-20 3) 2013-09-30	0.88	200	0.43	2013-06-26	BBCH 13	fruit	<0.010	<0.010	<0.010	<0.010	<0.010	96	spraying sample storage time not validated 3: fruit analytical method: BASF No. L0179/02 (HPLC- MS/MS), LOQ(s): all 0.01 mg/kg max. sample storage time in month(s): 4 ASB2015-286

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Trial No./ Location/ Year	Commodity/ Variety	Date of 1.Sowing or planting 2.Flowering 3. Harvest	Application rate per treatment			Dates of treatment or number and last date	Growth stage at last treatment	Portion analysed	Residues (mg/kg)					PHI (days)	Remarks
			g a.s./ha	Water (L/ha)	g a.s./hL				(1)	(2)	(3)	(4)	(5)		
BASF DocID 2013/1335406, study 428112 (B3137), trial L130551, harvest trial Netherlands (NL) 5853 AB Siebengewald 2014-08-11	Charentais	1) 2013-06-21 (planting) 2) 2013-07-19 - 2013-09-07 3) 2013-09-17	0.89	210	0.43	2013-06-24	BBCH 12-13	fruit	<0.010	<0.010	<0.010	<0.010	<0.010	85	spraying sample storage time not validated 3: fruit analytical method: BASF No. L0179/02 (HPLC- MS/MS), LOQ(s): all 0.01 mg/kg max. sample storage time in month(s): 5 ASB2015-286

B.7.3.2.3 Cucumber

The cGAP in N-EU is 1 x 0.864 kg as/ha spray application at post-emergence stage up to BBCH 16. PHI is covered by the growing period between application and harvest.

Reference:	KCA 6.3.
Report	Roche, A. (2014): Study on the residue behaviour of dimethenamid-P after soil treatment with BAS 656 12 H in cucumber and zucchini under field conditions in Northern Europe, 2013, Study code: 428113, Doc ID 2013/1335415, ASB2015-294
Guideline(s):	EC 1107/2009 of the European Parliament and of the Council of 21 Oct 2009, EEC 79/117, EEC 7029/VI/95 rev. 5 Appendix B (July 22 1997), EEC 7525/VI/95 rev. 9 (March 2011)
Deviations:	None
GLP:	Yes
Acceptability:	The study is acceptable.

Materials and methods

A total of 4 supervised residue trials (all NEU) conducted in 2013 were provided by the applicant. Cucumbers were treated according to the GAP with 1 application at nominal rates of 0.792 - 0.864 kg as/ha. Plants were harvested at 47-80 DALA. The samples were stored deep frozen for max. 8 months. All samples were analysed for dimethenamid-P and the metabolites M23, M26, M27 and M30 according to method BASF L0179/02. For a detailed method description see B.5.2.1.1.

Results and Discussion

Procedural recoveries were with the exception of M26 at LOQ level within acceptable limits (Table B.7.3-21). Storage of melon samples was validated for dimethenamid-P, M23, M27 and M30, but not for M26. Residue levels in individual samples are reported in Table B.7.3-22. No residues were found in untreated control samples.

Table B.7.3-21: Procedural recoveries for dimethenamid-P and metabolites in cucumber

Matrix	Fortification Level [mg/kg]	Summary recoveries							
		Recovery [%]	Mean [%]	RSD [%]	n	Recovery [%]	Mean [%]	RSD [%]	n
		Dimethenamid-P				M23			
Cucumber	0.01	108	-	-	1	109	-	-	1
	0.1	99	-	-	1	98	-	-	1
		M26				M27			
Cucumber	0.01	66	-	-	1	110	-	-	1
	1	100	-	-	1	96	-	-	1
		M30							
Cucumber	0.01	119	-	-	1				
	1	94	-	-	1				

Conclusion

Residues were <0.01 mg/kg per analyte in all samples. However, the sample storage time for metabolite M26 is not covered by storage stability studies evaluated in Vol. 3 CA, section B.7.1.1 of the RAR.

Table B.7.3-22: N-EU residues trials with cucumber conducted according to GAP

Reference:	Study on the residue behaviour of dimethenamid-P after soil treatment with BAS 656 12 H in cucumber and zucchini under field conditions in Northern Europe, 2013, Roche, A., 2014; Study code: 428113, Doc ID 2013/1335415, ASB2015-294				
GLP:	Yes	Sample storage conditions:	5 - 8 month at -18 °C		
Crop/crop group:	Cucumber (CUMSA)	Analytical method:	BASF No. L0179/02		
Indoor/Outdoor:	Outdoors (NEU)	Limit of Quantification (mg/kg):	0.01 mg/kg		
Formulation:	EC	Limit of Detection (mg/kg):	-		
Content of active substance (g/kg or g/L):	720 g/L	Residues calculated as:	(1) dimethenamid-P (2) M23 oxalamide (3) M26 thiolactic acid conjugate (4) M27 sulfonate (5) M30 sulfoxide of thiolactic acid conjugate		

Trial No./ Location/ Year	Commodity/ Variety	Date of 1.Sowing or planting 2.Flowering 3. Harvest	Application rate per treatment			Dates of treatment or number and last date	Growth stage at last treatment	Portion analysed	Residues (mg/kg)					PHI (days)	Remarks
			g a.s./ha	Water (L/ha)	g a.s./hL				(1)	(2)	(3)	(4)	(5)		
BASF DocID 2013/1335415, study 428113 (ANADIAG study No. B3065), trial L130427 (B3065 GE1), harvest trial Germany (DE) 69124 Heidelberg 2014-08-12	Marketer	1) 2013-08-06 (planting) 2) 2013-09-15 - 2013-10-15 3) 2013-10-25	0.79	180	0.43	2013-08-06	BBCH 12-13	fruit	<u>≤0.010</u>	<0.010	<u>≤0.010</u>	<0.010	<u>≤0.010</u>	80	spraying sample storage time not validated 3: fruit analytical method: BASF No. L0179/02 (HPLC- MS/MS), LOQ(s): all 0.01 mg/kg max. sample storage time in month(s): 5 ASB2015-294

Trial No./ Location/ Year	Commodity/ Variety	Date of 1.Sowing or planting 2.Flowering 3. Harvest	Application rate per treatment			Dates of treatment or number and last date	Growth stage at last treatment	Portion analysed	Residues (mg/kg)					PHI (days)	Remarks
			g a.s./ha	Water (L/ha)	g a.s./hL				(1)	(2)	(3)	(4)	(5)		
BASF DocID 2013/1335415, study 428113 (ANADIAG study No. B3065), trial L130428 (B3065 BM1), harvest trial France (FR) 72800 Thorée Les Pins, Pays del Loire 2014-08-12	Byblos	1) 2013-05-17 (planting) 2) 2013-06-10 - 2013-07-10 3) 2013-07-20	0.84	190	0.44	2013-05-17	BBCH 12-13	fruit	<u><0.010</u>	<0.010	<u><0.010</u>	<0.010	<u><0.010</u>	64	spraying sample storage time not validated 3: fruit analytical method: BASF No. L0179/02 (HPLC- MS/MS), LOQ(s): all 0.01 mg/kg max. sample storage time in month(s): 8 ASB2015-294
BASF DocID 2013/1335415, study 428113 (ANADIAG study No. B3065), trial L130429 (B3065 BE1), harvest trial Belgium (BE) 3534 Halen, Limburg 2014-08-12	Delikatess	1) 2013-06-26 (planting) 2) 2013-07-01 - 2013-08-12 3) 2013-08-12	0.86	200	0.43	2013-06-26	BBCH 12-13	fruit	<u><0.010</u>	<0.010	<u><0.010</u>	<0.010	<u><0.010</u>	47	spraying sample storage time not validated 3: fruit analytical method: BASF No. L0179/02 (HPLC- MS/MS), LOQ(s): all 0.01 mg/kg max. sample storage time in month(s): 7 ASB2015-294

Trial No./ Location/ Year	Commodity/ Variety	Date of 1.Sowing or planting 2.Flowering 3. Harvest	Application rate per treatment			Dates of treatment or number and last date	Growth stage at last treatment	Portion analysed	Residues (mg/kg)					PHI (days)	Remarks
			g a.s./ha	Water (L/ha)	g a.s./hL				(1)	(2)	(3)	(4)	(5)		
BASF DocID 2013/1335415, study 428113 (ANADIAG study No. B3065), trial L130430 (B3065 NL1), harvest trial Netherlands (NL) 6595 ME Ottersum, Gennep, Limburg 2014-08-12	Delikatess	1) 2013-06-27 (planting) 2) 2013-07-03 - 2013-08-14 3) 2013-08-14	0.86	200	0.43	2013-06-27	BBCH 12-13	fruit	<u><0.010</u>	<0.010	<u><0.010</u>	<0.010	<u><0.010</u>	48	spraying sample storage time not validated 3: fruit analytical method: BASF No. L0179/02 (HPLC- MS/MS), LOQ(s): all 0.01 mg/kg max. sample storage time in month(s): 7 ASB2015-294

B.7.3.2.4 Pumpkin

The cGAP in N-EU is 1 x 0.864 kg as/ha spray application at post-emergence stage up to BBCH 16. PHI is covered by the growing period between application and harvest.

Reference:	KCA 6.3.
Report	Roche, A. (2014): Study on the residue behaviour of BAS 656 H (dimethenamid-P) after soil treatment with BAS 656 12 H in melon and pumpkin under field conditions in Northern Europe, 2013, Study code: 428112, Doc ID 2013/1335406, ASB2015-286
Guideline(s):	EC 1107/2009 of the European Parliament and of the Council of 21 Oct 2009, EEC 79/117, EEC 7029/VI/95 rev. 5 Appendix B (July 22 1997), EEC 7525/VI/95 rev. 9 (March 2011)
Deviations:	None
GLP:	Yes
Acceptability:	The study is acceptable.

Materials and methods

A total of 4 supervised residue trials (all NEU) conducted in 2013 were provided by the applicant. Pumpkins were treated according to the GAP with 1 application at nominal rates of 0.806 - 0.936 kg as/ha. Plants were harvested at 74-85 DALA. The samples were stored deep frozen for max. 5 months. All samples were analysed for dimethenamid-P and the metabolites M23, M26, M27 and M30 according to method BASF L0179/02. For a detailed method description see B.5.2.1.1.

Results and Discussion

Procedural recoveries were within acceptable limits (Table B.7.3-23). Storage of melon samples was validated for dimethenamid-P, M23, M27 and M30, but not for M26. Residue levels in individual samples are reported in Table B.7.3-24. No residues were found in untreated control samples.

Table B.7.3-23: Procedural recoveries for dimethenamid-P and metabolites in pumpkin

Matrix	Fortification Level [mg/kg]	Summary recoveries							
		Recovery [%]	Mean [%]	RSD [%]	n	Recovery [%]	Mean [%]	RSD [%]	n
		Dimethenamid-p				M23			
Pumpkin	0.01	108	-	-	1	111	-	-	1
	0.1	104	-	-	1	98	-	-	1
	5	111	-	-	1	-	-	-	-
		M26				M27			
Pumpkin	0.01	73	-	-	1	105	-	-	1
	1	91	-	-	1	103	-	-	1
		M30							
Pumpkin	0.01	120	-	-	1				
	1	98	-	-	1				

Conclusion

Residues were <0.01 mg/kg per analyte in all samples. However, sample storage time for metabolite M26 is not covered by storage stability studies evaluated in Vol. 3 CA, section B.7.1.1 of the RAR.

Table B.7.3-24: N-EU residues trials with pumpkin conducted according to GAP

Reference: Study on the residue behaviour of BAS 656 H (dimethenamid-P) after soil treatment with BAS 656 12 H in melon and pumpkin under field conditions in Northern Europe, 2013, Roche, A., 2014; Study code: 428112, Doc ID 2013/1335406, [ASB2015-286](#)

GLP: Yes Sample storage conditions: 4 - 5 month at -18 °C

Crop/crop group: Pumpkin (CUUPE) Analytical method: BASF No. L0179/02

Indoor/Outdoor: Outdoors (NEU) Limit of Quantification (mg/kg): 0.01 mg/kg

Formulation: EC Limit of Detection (mg/kg): -

Content of active substance (g/kg or g/L): 720 g/L Residues calculated as: (1) dimethenamid-P
(2) M23 oxalamide
(3) M26 thiolactic acid conjugate
(4) M27 sulfonate
(5) M30 sulfoxide of thiolactic acid conjugate

Trial No./ Location/ Year	Commodity/ Variety	Date of 1.Sowing or planting 2.Flowering 3. Harvest	Application rate per treatment			Dates of treatment or number and last date	Growth stage at last treatment	Portion analysed	Residues (mg/kg)					PHI (days)	Remarks
			g as/ha	Water (L/ha)	g as/hL				(1)	(2)	(3)	(4)	(5)		
BASF DocID 2013/1335406, study 428112 (B3137), trial L130552, harvest trial Germany (DE) 69124 Heidelberg 2014-08-11	Spaghetti squash	1) 2013-08-07 (planting) 2) 2013-09-15 - 2013-10-15 3) 2013-10-25	0.91	210	0.43	2013-08-07	BBCH 12-13	fruit	<0.010	<0.010	<0.010	<0.010	<0.010	79	spraying sample storage time not validated 3: fruit analytical method: BASF No. L0179/02 (HPLC- MS/MS), LOQ(s): all 0.01 mg/kg max. sample storage time in month(s): 4 ASB2015-286

Trial No./ Location/ Year	Commodity/ Variety	Date of 1. Sowing or planting 2. Flowering 3. Harvest	Application rate per treatment			Dates of treatment or number and last date	Growth stage at last treatment	Portion analysed	Residues (mg/kg)					PHI (days)	Remarks
			g as/ha	Water (L/ha)	g as/hL				(1)	(2)	(3)	(4)	(5)		
BASF DocID 2013/1335406, study 428112 (B3137), trial L130553, harvest trial Poland (PL) 95-061 Dmosin 2014-08-11	unknown	1) 2013-06-18 (planting) 2) 3) 2013-09-10	0.94	220	0.43	2013-06-18	BBCH 11-13	fruit	<0.010	<0.010	<0.010	<0.010	<0.010	84	spraying sample storage time not validated 3: fruit analytical method: BASF No. L0179/02 (HPLC- MS/MS), LOQ(s): all 0.01 mg/kg max. sample storage time in month(s): 5 ASB2015-286
BASF DocID 2013/1335406, study 428112 (B3137), trial L130554, harvest trial France (FR) 67203 Ober- schaeffolsheim 2014-08-11	Uchiki Kuri	1) 2013-06-11 (planting) 2) 2013-07-15 - 2013-08-10 3) 2013-09-12	0.81	190	0.43	2013-06-19	BBCH 11-12	fruit	<0.010	<0.010	<0.010	<0.010	<0.010	85	spraying sample storage time not validated 3: fruit analytical method: BASF No. L0179/02 (HPLC- MS/MS), LOQ(s): all 0.01 mg/kg max. sample storage time in month(s): 5 ASB2015-286

Trial No./ Location/ Year	Commodity/ Variety	Date of 1.Sowing or planting 2.Flowering 3. Harvest	Application rate per treatment			Dates of treatment or number and last date	Growth stage at last treatment	Portion analysed	Residues (mg/kg)					PHI (days)	Remarks
			g as/ha	Water (L/ha)	g as/hL				(1)	(2)	(3)	(4)	(5)		
BASF DocID 2013/1335406, study 428112 (B3137), trial L130555, harvest trial Netherlands (NL) 6599 AV Ven- Zelderheide 2014-08-11	Hokaido	1) 2013-06-24 (planting) 2) 2013-07-15 - 2013-08-20 3) 2013-09-06	0.88	200	0.43	2013-06-24	BBCH 13-14	fruit	<u><0.010</u>	<0.010	<u><0.010</u>	<0.010	<u><0.010</u>	74	spraying sample storage time not validated 3: fruit analytical method: BASF No. L0179/02 (HPLC- MS/MS), LOQ(s): all 0.01 mg/kg max. sample storage time in month(s): 5 ASB2015-286

B.7.3.2.5 Zucchini

The cGAP in N-EU is 1 x 0.864 kg as/ha spray application at post-emergence stage up to BBCH 16. PHI is covered by the growing period between application and harvest.

Reference:	KCA 6.3.
Report	Roche, A. (2014): Study on the residue behaviour of dimethenamid-P after soil treatment with BAS 656 12 H in cucumber and zucchini under field conditions in Northern Europe, 2013, Study code: 428113, Doc ID 2013/1335415, ASB2015-294
Guideline(s):	EC 1107/2009 of the European Parliament and of the Council of 21 Oct 2009, EEC 79/117, EEC 7029/VI/95 rev. 5 Appendix B (July 22 1997), EEC 7525/VI/95 rev. 9 (March 2011)
Deviations:	None
GLP:	Yes
Acceptability:	The study is acceptable.

Materials and methods

A total of 4 supervised residue trials (all NEU) conducted in 2013 were provided by the applicant. Zucchini plants were treated according to the GAP with 1 application at nominal rates of 0.878 - 0.936 kg as/ha. Plants were harvested at 36-80 DALA. The samples were stored deep frozen for max. 8 months. All samples were analysed for dimethenamid-P and the metabolites M23, M26, M27 and M30 according to method BASF L0179/02. For a detailed method description see B.5.2.1.1.

Results and Discussion

Procedural recoveries were within acceptable limits Table B.7.3-25. Storage of zucchini samples was validated for dimethenamid-P, M23, M27 and M30, but not for M26. Residue levels in individual samples are reported in Table B.7.3-26. No residues were found in untreated control samples.

Table B.7.3-25: Procedural recoveries for dimethenamid-P and metabolites in zucchini

Matrix	Fortification Level [mg/kg]	Summary recoveries							
		Recovery [%]	Mean [%]	RSD [%]	n	Recovery [%]	Mean [%]	RSD [%]	n
		Dimethenamid-P				M23			
Zucchini	0.01	110	-	-	1	108	-	-	1
	0.1	96	-	-	1	101	-	-	1
		M26				M27			
Zucchini	0.01	74	-	-	1	110	-	-	1
	0.1	99	-	-	1	96	-	-	1
		M30							
Zucchini	0.01	102	-	-	1				
	0.1	94	-	-	1				

Conclusion

Residues were <0.01 mg/kg per analyte in all samples. However, sample storage time for metabolite M26 is not covered by storage stability studies evaluated in Vol. 3 CA, section B.7.1.1 of the RAR.

Table B.7.3-26: N-EU residues trials with zucchini conducted according to GAP

Reference:	Study on the residue behaviour of dimethenamid-P after soil treatment with BAS 656 12 H in cucumber and zucchini under field conditions in Northern Europe, 2013, Roche, A., 2014; Study code: 428113, Doc ID 2013/1335415, ASB2015-294				
GLP:	Yes	Sample storage conditions:	5 - 8 month at -18 °C		
Crop/crop group:	Zucchini (CUUPG)	Analytical method:	BASF No. L0179/02		
Indoor/Outdoor:	Outdoors (NEU)	Limit of Quantification (mg/kg):	0.01 mg/kg		
Formulation:	EC	Limit of Detection (mg/kg):	-		
Content of active substance (g/kg or g/L):	720 g/L	Residues calculated as:	(1) dimethenamid-P (2) M23 oxalamide (3) M26 thiolactic acid conjugate (4) M27 sulfonate (5) M30 sulfoxide of thiolactic acid conjugate		

Trial No./ Location/ Year	Commodity/ Variety	Date of 1.Sowing or planting 2.Flowering 3. Harvest	Application rate per treatment			Dates of treatment or number and last date	Growth stage at last treatment	Portion analysed	Residues (mg/kg)					PHI (days)	Remarks
			g as/ha	Water (L/ha)	g as/hL				(1)	(2)	(3)	(4)	(5)		
BASF DocID 2013/1335415, study 428113 (ANADIAG study No. B3065), trial L130431 (B3065 GE2), harvest trial Germany (DE) 69124 Heidelberg 2014-08-12	unknown	1) 2013-08-06 (planting) 2) 2013-09-15 - 2013-10-15 3) 2013-10-25	0.94	220	0.43	2013-08-06	BBCH 12-13	fruit	<0.010	<0.010	<0.010	<0.010	<0.010	80	spraying sample storage time not validated 3: fruit analytical method: BASF No. L0179/02 (HPLC- MS/MS), LOQ(s): all 0.01 mg/kg max. sample storage time in month(s): 5 ASB2015-294

Trial No./ Location/ Year	Commodity/ Variety	Date of 1. Sowing or planting 2. Flowering 3. Harvest	Application rate per treatment			Dates of treatment or number and last date	Growth stage at last treatment	Portion analysed	Residues (mg/kg)					PHI (days)	Remarks
			g as/ha	Water (L/ha)	g as/hL				(1)	(2)	(3)	(4)	(5)		
BASF DocID 2013/1335415, study 428113 (ANADIAG study No. B3065), trial L130432 (B3065 BE2), harvest trial Belgium (BE) 3534 Halen, Limburg 2014-08-12	Black Beauty	1) 2013-06-26 (planting) 2) 2013-07-01 - 2013-08-01 3) 2013-08-01	0.88	200	0.43	2013-06-26	BBCH 13	fruit	<0.010	<0.010	<0.010	<0.010	<0.010	36	spraying sample storage time not validated 3: fruit analytical method: BASF No. L0179/02 (HPLC- MS/MS), LOQ(s): all 0.01 mg/kg max. sample storage time in month(s): 7 ASB2015-294
BASF DocID 2013/1335415, study 428113 (ANADIAG study No. B3065), trial L130433 (B3065 UK1), harvest trial United Kingdom (UK) OX27 9AS Bicester, Oxfordshire 2014-08-12	Alexander	1) 2013-06-11 (planting) 2) 2013-07-11 - 2013-07-26 3) 2013-07-29	0.91	210	0.43	2013-06-11	BBCH 12	fruit	<0.010	<0.010	<0.010	<0.010	<0.010	48	spraying sample storage time not validated 3: fruit analytical method: BASF No. L0179/02 (HPLC- MS/MS), LOQ(s): all 0.01 mg/kg max. sample storage time in month(s): 8 ASB2015-294

Trial No./ Location/ Year	Commodity/ Variety	Date of 1.Sowing or planting 2.Flowering 3. Harvest	Application rate per treatment			Dates of treatment or number and last date	Growth stage at last treatment	Portion analysed	Residues (mg/kg)					PHI (days)	Remarks
			g as/ha	Water (L/ha)	g as/hL				(1)	(2)	(3)	(4)	(5)		
BASF DocID 2013/1335415, study 428113 (ANADIAG study No. B3065), trial L130434 (B3065 NL2), harvest trial Netherlands (NL) 6595 ME Ottersum, Gennep, Limburg 2014-08-12	Black Beauty	1) 2013-07-08 (planting) 2) 2013-07-15 - 2013-08-14 3) 2013-08-14	0.92	210	0.43	2013-07-08	BBCH 10-11	fruit	<u><0.010</u>	<0.010	<u><0.010</u>	<0.010	<u><0.010</u>	37	spraying sample storage time not validated 3: fruit analytical method: BASF No. L0179/02 (HPLC- MS/MS), LOQ(s): all 0.01 mg/kg max. sample storage time in month(s): 7 ASB2015-294

B.7.3.2.6 Cauliflower

The cGAP in N- and S-EU is 1 x 0.720 kg as/ha spray application at post-emergence stage up to BBCH 16. The proposed PHI is set to 35 days.

Reference:	KCA 6.3.
Report	Martín, T. (2013): Study on the residue behaviour of metazachlor (BAS 479 H) and dimethenamid-P (BAS 656 H) on cabbages (cauliflower and white cabbage) after the application of BAS 769 00 H under field conditions in France (South), Italy and Spain, 2012, Study code: 418108, Doc ID 2013/1182806, ASB2015-285
Guideline(s):	EC 1107/2009 of the European Parliament and of the Council of 21 Oct 2009, EEC 79/117, EEC 7029/VI/95 rev. 5 Appendix B (July 22 1997), EEC 7525/VI/95 rev. 9 (March 2011)
Deviations:	None
GLP:	Yes
Acceptability:	The study is acceptable.
Reference:	KCA 6.3.
Report	Schneider, E. (2014): Study on the Residue Behaviour of Metazachlor (BAS 479 H) and Dimethenamid-P (BAS 656 H) in Cauliflower and Broccoli after Treatment with BAS 769 00 H under field conditions in Northern and Southern Europe, 2013 – 2014, Study code: 428106, Doc ID 2013/1335410, ASB2015-290
Guideline(s):	EC 1107/2009 of the European Parliament and of the Council of 21 Oct 2009, EEC 79/117, EEC 7029/VI/95 rev. 5 Appendix B (July 22 1997), EEC 7525/VI/95 rev. 9 (March 2011)
Deviations:	None
GLP:	Yes
Acceptability:	The study is acceptable only if the fall-back GAP with an application rate of 500 g/ha is used.

Materials and methods

Out of 6 supervised residue trials in cauliflower, 2 trials were conducted in N-EU and 4 trials in S-EU in 2012-2014.

Cauliflower plants were not treated according to the GAP but with 1 application at nominal rates of 0.464 - 0.526 kg as/ha. Plants were harvested at 49-148 DALA. The samples were stored deep frozen for max. 11 months. All samples were analysed for dimethenamid-P and the metabolites M23, M26, M27 and M30 according to method BASF L0179/02. For a detailed method description see B.5.2.1.1.

Results and Discussion

Procedural recoveries were within acceptable limits (Table B.7.3-27). Storage of cauliflower samples was validated for dimethenamid-P, M23, M27 and M30, but not for M26. Residue levels in individual samples are reported in Table B.7.3-28 and Table B.7.3-29. No residues were found in untreated control samples.

Table B.7.3-27: Procedural recoveries for dimethenamid-P and metabolites in cauliflower

Matrix	Fortification Level [mg/kg]	Summary recoveries							
		Recovery [%]	Mean [%]	RSD [%]	n	Recovery [%]	Mean [%]	RSD [%]	n
		Dimethenamid-p				M23			
Whole plant	0.01	105, 106, 102	104	2	3	101, 114, 102, 108	106	6	4
	25	92, 90	91	-	2	82, 79, 86	82	4	3
Cauliflower	0.01	84, 86, 95, 97, 102, 97	94	7	6	92, 84, 100, 87, 92, 84	90	7	6
	0.1	92, 102	100	-	2	77, 90	84	-	2
	1	94, 98, 89	94	5	3	92, 92, 81	88	7	3
	10	109	-	-	1	-	-	-	1
		M26				M27			
Whole plant	0.01	81, 75, 82	79	5	3	99, 88, 92	93	6	3
	25	81, 81, 87	83	4	3	83, 90, 93	89	6	3
Cauliflower	0.01	90, 87, 98, 79, 76, 79	85	10	6	101, 95, 112, 86, 94, 85	96	11	6
	0.1	73, 81	77	-	2	74, 84	79	-	2
	1	83, 94, 72	83	13	3	90, 95, 89	91	4	3
		M30							
Whole plant	0.01	102, 81, 69, 78	83	17	4				
	25	75, 90, 90	85	10	3				
Cauliflower	0.01	105, 104, 114, 96, 99, 88	101	9	6				
	0.1	76, 85	81	-	2				
	1	94, 92, 80	89	9	3				

Conclusion

Residues were <0.01 mg/kg per analyte in all samples. With the application rate according to the cGAP being 0.720 kg/ha, all trials have to be considered underdosed, even if the 25 % rule is applied. However, the data may be used in support of a fall-back GAP with an application rate of 500 g/ha. Sample storage time for metabolite M26 is not covered by storage stability studies evaluated in Vol. 3 CA, section B.7.1.1 of the RAR.

Table B.7.3-28: S-EU residues trials with cauliflower conducted according to GAP

Reference:	Study on the residue behaviour of metazachlor (BAS 479 H) and dimethenamid-P(BAS 656 H) on cabbages (cauliflower and white cabbage) after the application of BAS 769 00 H under field conditions in France (South), Italy and Spain, 2012, Martín, T., 2013; Study code: 418108, Doc ID 2013/1182806, ASB2015-285				
GLP:	Yes	Sample storage conditions:	5 - 11 month at -18 °C		
Crop/crop group:	Cauliflower (BRSOB)	Analytical method:	BASF No. L0179/02		
Indoor/Outdoor:	Outdoors (SEU)	Limit of Quantification (mg/kg):	0.01 mg/kg		
Formulation:	EC	Limit of Detection (mg/kg):	-		
Content of active substance (g/kg or g/L):	200 g/L	Residues calculated as:	(1) dimethenamid-P (2) M23 oxalamide (3) M26 thiolactic acid conjugate (4) M27 sulfonate (5) M30 sulfoxide of thiolactic acid conjugate		

Trial No./ Location/ Year	Commodity/ Variety	Date of 1.Sowing or planting 2.Flowering 3. Harvest	Application rate per treatment			Dates of treatment or number and last date	Growth stage at last treatment	Portion analysed	Residues (mg/kg)					PHI (days)	Remarks
			g as/ha	Water (L/ha)	g as/hL				(1)	(2)	(3)	(4)	(5)		
BASF DocID 2013/1182806, study 418108 (PTRL Europe ID P 2804 G), trial L120382, harvest trial France (FR) 84700 Sorgues, Provence Alpes Côte d' Azur 2013-06-27	Drakkar	1) 2012-08-16 (planting) 2) 3) 2013-02-06	0.53	260	0.20	2012-09-11	BBCH 18	whole plant	5.3	<0.010	0.020	<0.010	0.040	0	spraying
								flower	<0.010	<0.010	<0.010	<0.010	<0.010	148	sample storage time not validated 3: whole plant analytical method: BASF No. L0179/02 (HPLC- MS/MS), LOQ(s): all 0.01 mg/kg max. sample storage time in month(s): 7 ASB2015-285
BASF DocID 2013/1182806, study 418108 (PTRL Europe ID P 2804 G), trial L120383,	Castellum	1) 2012-04-26 (planting) 2) 3) 2012-07-10	0.46	93	0.50	2012-05-22	BBCH 18	whole plant	2.1	<0.010	0.060	<0.010	0.060	0	spraying
								flower	<0.010	<0.010	<0.010	<0.010	<0.010	49	sample storage time not validated 3: all matrices

Trial No./ Location/ Year	Commodity/ Variety	Date of 1.Sowing or planting 2.Flowering 3. Harvest	Application rate per treatment			Dates of treatment or number and last date	Growth stage at last treatment	Portion analysed	Residues (mg/kg)					PHI (days)	Remarks
			g as/ha	Water (L/ha)	g as/hL				(1)	(2)	(3)	(4)	(5)		
harvest trial Italy (IT) 40057 Granarolo dell' Emilia, Emilia Romagna 2013-06-27															analytical method: BASF No. L0179/02 (HPLC- MS/MS), LOQ(s): all 0.01 mg/kg max. sample storage time in month(s): 11 ASB2015-285

Reference:	Study on the Residue Behaviour of Metazachlor (BAS 479 H) and dimethenamid-P (BAS 656 H) in Cauliflower and Broccoli after Treatment with BAS 769 00 H under field conditions in Northern and Southern Europe, 2013 - 2014, Schneider, E., 2014; Study code: 428106, Doc ID 2013/1335410, ASB2015-290				
GLP:	Yes	Sample storage conditions:	5 - 11 month at -18 °C		
Crop/crop group:	Cauliflower (BRSOB)	Analytical method:	BASF No. L0179/02		
Indoor/Outdoor:	Outdoors (SEU)	Limit of Quantification (mg/kg):	0.01 mg/kg		
Formulation:	EC	Limit of Detection (mg/kg):	-		
Content of active substance (g/kg or g/L):	200 g/L	Residues calculated as:	(1) dimethenamid-P (2) M23 oxalamide (3) M26 thiolactic acid conjugate (4) M27 sulfonate (5) M30 sulfoxide of thiolactic acid conjugate		

Trial No./ Location/ Year	Commodity/ Variety	Date of 1.Sowing or planting 2.Flowering 3. Harvest	Application rate per treatment			Dates of treatment or number and last date	Growth stage at last treatment	Portion analysed	Residues (mg/kg)					PHI (days)	Remarks
			g as/ha	Water (L/ha)	g as/hL				(1)	(2)	(3)	(4)	(5)		
BASF DocID 2013/1335410, study 428106 (PTRL Europe ID P 3080 G), trial L130445 (B3066 ES1), harvest trial Spain (ES) 08902 L'Hospitalet de Llobregat; Cataluña 2014-06-17	Skywalker	1) 2013-09-20 (planting) 2) 3) 2014-01-25 - 2014-02-08	0.48	190	0.25	2013-10-08	BBCH 18	whole plant	4.7	<0.010	0.046	<0.010	0.11	0	spraying
								flower	<0.010	<0.010	<0.010	<0.010	<0.010	114	sample storage time not validated 3: whole plant analytical method: BASF No. L0179/02 (HPLC- MS/MS), LOQ(s): all 0.01 mg/kg max. sample storage time in month(s): 5 ASB2015-290
BASF DocID 2013/1335410, study 428106 (PTRL Europe ID P 3080 G), trial L130446 (B3066 GR1), harvest trial Greece (GR) 57011	Concept	1) 2013-09-04 (planting) 2) 3) 2013-12-10 - 2013-12-20	0.50	200	0.25	2013-10-15	BBCH 18	whole plant	10.0	<0.010	0.10	<0.010	0.12	0	spraying
								flower	<0.010	<0.010	<0.010	<0.010	<0.010	58	sample storage time not validated 3: whole plant analytical method: BASF No. L0179/02 (HPLC- MS/MS), LOQ(s): all 0.01

Trial No./ Location/ Year	Commodity/ Variety	Date of 1.Sowing or planting 2.Flowering 3. Harvest	Application rate per treatment			Dates of treatment or number and last date	Growth stage at last treatment	Portion analysed	Residues (mg/kg)					PHI (days)	Remarks
			g as/ha	Water (L/ha)	g as/hL				(1)	(2)	(3)	(4)	(5)		
Kastanas, Thessaloniki 2014-06-17															mg/kg max. sample storage time in month(s): 5 ASB2015-290

Table B.7.3-29: N-EU residues trials with cauliflower conducted according to GAP

Reference: Study on the Residue Behaviour of Metazachlor (BAS 479 H) and dimethenamid-P (BAS 656 H) in Cauliflower and Broccoli after Treatment with BAS 769 00 H under field conditions in Northern and Southern Europe, 2013 - 2014, Schneider, E., 2014; Study code: 428106, Doc ID 2013/1335410, ASB2015-290

GLP: Yes Sample storage conditions: 5 - 11 month at -18 °C

Crop/crop group: Cauliflower (BRSOB) Analytical method: BASF No. L0179/02

Indoor/Outdoor: Outdoors (NEU) Limit of Quantification (mg/kg): 0.01 mg/kg

Formulation: EC Limit of Detection (mg/kg): -

Content of active substance (g/kg or g/L): 200 g/L Residues calculated as:

(1) dimethenamid-P
(2) M23 oxalamide
(3) M26 thiolactic acid conjugate
(4) M27 sulfonate
(5) M30 sulfoxide of thiolactic acid conjugate

Trial No./ Location/ Year	Commodity/ Variety	Date of 1.Sowing or planting 2.Flowering 3. Harvest	Application rate per treatment			Dates of treatment or number and last date	Growth stage at last treatment	Portion analysed	Residues (mg/kg)					PHI (days)	Remarks
			g as/ha	Water (L/ha)	g as/hL				(1)	(2)	(3)	(4)	(5)		
BASF DocID 2013/1335410, study 428106 (PTRL Europe ID P 3080 G), trial L130441 (B3066 GE1), harvest trial Germany (DE) 69124 Heidelberg 2014-06-17	Cornell	1) 2013-07-01 (planting) 2) 3)2013-10-25 - 2013-10-30	0.52	210	0.25	2013-08-02	BBCH 18	whole plant	1.8	<0.010	0.022	<0.010	0.20	0	spraying
								flower	<0.010	<0.010	<0.010	<0.010	<0.010	84	sample storage time not validated 3: all matrices analytical method: BASF No. L0179/02 (HPLC- MS/MS), LOQ(s): all 0.01 mg/kg max. sample storage time in month(s): 6 ASB2015-290
BASF DocID	Gregor	1) 2013-07-02	0.49	190	0.25	2013-07-25	BBCH 18	whole plant	2.7	<0.010	0.022	<0.010	0.092	0	spraying

Trial No./ Location/ Year	Commodity/ Variety	Date of 1.Sowing or planting 2.Flowering 3. Harvest	Application rate per treatment			Dates of treatment or number and last date	Growth stage at last treatment	Portion analysed	Residues (mg/kg)					PHI (days)	Remarks
			g as/ha	Water (L/ha)	g as/hL				(1)	(2)	(3)	(4)	(5)		
2013/1335410, study 428106 (PTRL Europe ID P 3080 G), trial L130442 (B3066 NL1), harvest trial Netherlands (NL) 6599 AV Ven- Zelderheide, Limburg 2014-06-17		(planting) 2) 3)2013-10-01 - 2013-10-04						flower	<u><0.010</u>	<0.010	<u><0.010</u>	<0.010	<u><0.010</u>	68	sample storage time not validated 8.3: all matrices analytical method: BASF No. L0179/02 (HPLC- MS/MS), LOQ(s): all 0.01 mg/kg max. sample storage time in month(s): 7 ASB2015-290

B.7.3.2.7 Broccoli

The cGAP in N- and S-EU is 1 x 0.720 kg as/ha spray application at post-emergence stage up to BBCH 16. The proposed PHI is set to 35 days.

Reference:	KCA 6.3.
Report	Schneider, E. (2014): Study on the Residue Behaviour of Metazachlor (BAS 479 H) and dimethenamid-P (BAS 656 H) in Cauliflower and Broccoli after Treatment with BAS 769 00 H under field conditions in Northern and Southern Europe, 2013 – 2014, Study code: 428106, Doc ID 2013/1335410, ASB2015-290
Guideline(s):	EC 1107/2009 of the European Parliament and of the Council of 21 Oct 2009, EEC 79/117, EEC 7029/VI/95 rev. 5 Appendix B (July 22 1997), EEC 7525/VI/95 rev. 9 (March 2011)
Deviations:	None
GLP:	Yes
Acceptability:	The study is acceptable only if the fall-back GAP with an application rate of 500 g/ha is used.

Materials and methods

A total of 2 supervised residue trials (both N-EU) conducted in 2013-2014 were provided by the applicant.

Broccoli plants were not treated according to the GAP with 1 application at nominal rates of 0.468 - 0.508 kg as/ha. Plants were harvested at 69-74 DALA. The samples were stored deep frozen for max. 8 months. All samples were analysed for dimethenamid-P and the metabolites M23, M26, M27 and M30 according to method BASF L0179/02. For a detailed method description see B.5.2.1.1.

Results and Discussion

Procedural recoveries were with the exception of M30 in whole plant within acceptable limits (Table B.7.3-30). Storage of broccoli samples was validated for dimethenamid-P, M23, M27 and M30, but not for M26. Residue levels in individual samples are reported in Table B.7.3-31. No residues were found in untreated control samples.

Table B.7.3-30: Procedural recoveries for dimethenamid-P and metabolites in broccoli

Matrix	Fortification Level [mg/kg]	Summary recoveries							
		Recovery [%]	Mean [%]	RSD [%]	n	Recovery [%]	Mean [%]	RSD [%]	n
		Dimethenamid-P				M23			
Whole plant	0.01	105, 92, 82	93	10	3	97, 83, 82, 93, 92, 102	92	9	6
	1	-	-	-	-	107, 106	106	-	2
	25	80, 84	82	-	2	77, 75	76	-	2
Broccoli	0.01	96, 102, 93	97	5	3	93, 100, 97	97	4	3
	1	95, 96	96	-	2	90, 94	92	-	2
		M26				M27			
Whole plant	0.01	84, 72, 76, 72	76	7	4	92, 77, 70, 98, 88, 102	88	14	6
	1	110, 106	108	-	2	108, 107	108	-	2
Cauliflower	0.01	80, 96, 90	89	9	3	85, 111, 112	103	15	3
	1	89, 95	92	-	2	92, 95	94	-	2
		M30							
Whole plant	0.01	97, 66, 94, 102, 104	93	17	5				
	1	123, 124	124	-	2				
Cauliflower	0.01	103, 116, 120	113	8	3				
	1	90, 99	95	-	2				

Conclusion

Residues were <0.01 mg/kg per analyte in all samples. With the application rate according to the cGAP being 0.720 kg/ha, all trials have to be considered underdosed, even if the 25 % rule is applied. However, the data may be used in support of a fall-back GAP with an application rate of 500 g/ha. Sample storage time for metabolite M26 is not covered by storage stability studies evaluated in Vol. 3 CA, section B.7.1.1 of the RAR.

Table B.7.3-31: N-EU residues trials with broccoli conducted according to GAP

Reference: Study on the Residue Behaviour of Metazachlor (BAS 479 H) and dimethenamid-P (BAS 656 H) in Cauliflower and Broccoli after Treatment with BAS 769 00 H under field conditions in Northern and Southern Europe, 2013 - 2014, Schneider, E., 2014; Study code: 428106, Doc ID 2013/1335410, ASB2015-290

GLP: Yes Sample storage conditions: 6 month at -18 °C

Crop/crop group: Broccoli (BRSOK) Analytical method: BASF No. L0179/02

Indoor/Outdoor: Outdoors (NEU) Limit of Quantification (mg/kg): 0.01 mg/kg

Formulation: EC Limit of Detection (mg/kg): -

Content of active substance (g/kg or g/L): 200 g/L Residues calculated as:

(1) dimethenamid-P
(2) M23 oxalamide
(3) M26 thiolactic acid conjugate
(4) M27 sulfonate
(5) M30 sulfoxide of thiolactic acid conjugate

Trial No./ Location/ Year	Commodity/ Variety	Date of 1.Sowing or planting 2.Flowering 3. Harvest	Application rate per treatment			Dates of treatment or number and last date	Growth stage at last treatment	Portion analysed	Residues (mg/kg)					PHI (days)	Remarks
			g as/ha	Water (L/ha)	g as/hL				(1)	(2)	(3)	(4)	(5)		
BASF DocID 2013/1335410, study 428106 (PTRL Europe ID P 3080 G), trial L130443 (B3066 BE1), harvest trial Belgium (BE) 3534 Halen, Limburg 2014-06-17	Lord	1) 2013-06-01 (planting) 2) 3) 2013-09-03	0.51	200	0.25	2013-06-26	BBCH 18	whole plant	4.3	<0.010	0.044	<0.010	0.13	0	spraying
								flower	<0.010	<0.010	<0.010	<0.010	<0.010	69	sample storage time not validated 3: all matrices analytical method: BASF No. L0179/02 (HPLC- MS/MS), LOQ(s): all 0.01 mg/kg max. sample storage time in month(s): 8 ASB2015-290

Trial No./ Location/ Year	Commodity/ Variety	Date of 1.Sowing or planting 2.Flowering 3. Harvest	Application rate per treatment			Dates of treatment or number and last date	Growth stage at last treatment	Portion analysed	Residues (mg/kg)					PHI (days)	Remarks
			g as/ha	Water (L/ha)	g as/hL				(1)	(2)	(3)	(4)	(5)		
BASF DocID 2013/1335410, study 428106 (PTRL Europe ID P 3080 G), trial L130444 (B3066 BM1), harvest trial France (FR) 49480 St. Sylvain d'Anjou, Pays de Loire 2014-06-17	Monaco	1) 2013-07-16 (planting) 2) 3)2013-10-08 - 2013-10-21	0.47	190	0.25	2013-08-05	BBCH 18	whole plant	5.0	<0.010	0.039	<0.010	0.18	0	spraying
								flower	<0.010	<0.010	<0.010	<0.010	<0.010	74	sample storage time not validated 3: all matrices analytical method: BASF No. L0179/02 (HPLC- MS/MS), LOQ(s): all 0.01 mg/kg max. sample storage time in month(s): 6 ASB2015-290

B.7.3.2.8 Brussels sprouts

The cGAP in N-EU is 1 x 0.720 kg as/ha spray application at post-emergence stage up to BBCH 16. The proposed PHI is set to 90 days.

Reference:	KCA 6.3.
Report	Aitken, A.; Munro, M. (2014): Study on the residue behaviour of dimethenamid-P (BAS 656 H) in Brussels sprouts following one application of BAS 656 12 H to 4 trials in 2013 - NEU, Study code: 428125, Doc ID 2013/1335418, ASB2015-296
Guideline(s):	EC 1107/2009 of the European Parliament and of the Council of 21 Oct 2009, EEC 79/117, EEC 7029/VI/95 rev. 5 Appendix B (July 22 1997), EEC 7525/VI/95 rev. 9 (March 2011), SANCO/3029/99 rev. 4
Deviations:	None
GLP:	Yes
Acceptability:	The study is acceptable.

Materials and methods

A total of 4 supervised residue trials (all NEU) conducted in 2013 were provided by the applicant. Brussels sprout plants were treated according to the GAP with 1 application at nominal rates of 0.716 - 0.756 kg as/ha. Plants were harvested at 88-143 DALA. The samples were stored deep frozen for max. 12 months. All samples were analysed for dimethenamid-P and the metabolites M23, M26, M27 and M30 according to method BASF L0179/02. For a detailed method description see B.5.2.1.1.

Results and Discussion

Procedural recoveries were within acceptable limits (Table B.7.3-32). Storage of Brussels sprout samples was validated for dimethenamid-P, M23, M27 and M30, but not for M26. Residue levels in individual samples are reported in Table B.7.3-33. No residues were found in untreated control samples.

Table B.7.3-32: Procedural recoveries for dimethenamid-P and metabolites in Brussels sprouts

Matrix	Fortification Level [mg/kg]	Summary recoveries							
		Recovery [%]	Mean [%]	RSD [%]	n	Recovery [%]	Mean [%]	RSD [%]	n
		Dimethenamid-P				M23			
Whole plant	0.01	100, 115	108	-	2	95	-	-	1
	0.1	94, 113	104	-	2	86	-	-	1
	10	74	-	-	1	-	-	-	-
	40	107	-	-	1	-	-	-	-
Sprouts	0.01	78	-	-	1	78	-	-	1
	0.1	97	-	-	1	94	-	-	1
		M26				M27			
Whole plant	0.01	88, 66	77	-	2	87	-	-	1
	0.1	79, 70	75	-	2	82	-	-	1
	1	66	-	-	1	-	-	-	-
Sprouts	0.01	74	-	-	1	75	-	-	1
	0.1	88	-	-	1	86	-	-	1
		M30							
Whole plant	0.01	108, 94	101	-	2				
	0.1	82, 72	77	-	2				
	1	66	-	-	1				
Sprouts	0.01	94	-	-	1				
	0.1	98	-	-	1				

Conclusion

Residues were <0.01 mg/kg per analyte in all samples. However, sample storage time for metabolite M26 is not covered by storage stability studies evaluated in Vol. 3 CA, section B.7.1.1 of the RAR.

Table B.7.3-33: N-EU residues trials with Brussels sprouts conducted according to GAP

Reference: Study on the residue behaviour of dimethenamid-P (BAS 656 H) in brussel sprouts following one application of BAS 656 12 H to 4 trials in 2013 - NEU, Aitken, A.; Munro, M., 2014; Study code: 428125, Doc ID 2013/1335418, ASB2015-296

GLP: Yes Sample storage conditions: 11 - 12 month at -18 °C

Crop/crop group: Brussels sprouts (BR5OF) Analytical method: BASF No. L0179/02

Indoor/Outdoor: Outdoors (NEU) Limit of Quantification (mg/kg): 0.01 mg/kg

Formulation: EC Limit of Detection (mg/kg): -

Content of active substance (g/kg or g/L): 720 g/L Residues calculated as: (1) dimethenamid-P
(2) M23 oxalamide
(3) M26 thiolactic acid conjugate
(4) M27 sulfonate
(5) M30 sulfoxide of thiolactic acid conjugate

Trial No./ Location/ Year	Commodity/ Variety	Date of 1.Sowing or planting 2.Flowering 3. Harvest	Application rate per treatment			Dates of treatment or number and last date	Growth stage at last treatment	Portion analysed	Residues (mg/kg)					PHI (days)	Remarks
			g as/ha	Water (L/ha)	g as/hL				(1)	(2)	(3)	(4)	(5)		
BASF DocID 2013/1335418, study 428125 (Charles River study No.698005), trial L130417, harvest trial United Kingdom (UK) EH49 7LP Linlithgow, West Lothian 2014-10-24	Maximus	1) 2013-05-17 (planting) 2) 3) 2013-10-01	0.72	200	0.36	2013-07-05	BBCH 18-19	whole plant	2.0	<0.010	0.026	0.012	0.31	0	spraying
								brussels sprouts	<0.010	<0.010	<0.010	<0.010	<0.010	88	sample storage time not validated 3: all matrices analytical method: BASF No. L0179/02 (HPLC-MS/MS), LOQ(s): all 0.01 mg/kg max. sample storage time in month(s): 11 ASB2015-296

Trial No./ Location/ Year	Commodity/ Variety	Date of 1.Sowing or planting 2.Flowering 3. Harvest	Application rate per treatment			Dates of treatment or number and last date	Growth stage at last treatment	Portion analysed	Residues (mg/kg)					PHI (days)	Remarks
			g as/ha	Water (L/ha)	g as/hL				(1)	(2)	(3)	(4)	(5)		
BASF DocID 2013/1335418, study 428125 (Charles River study No.698005), trial L130418, harvest trial Netherlands (NL) 6599 AV Ven- Zelderheide, Limburg 2014-10-24	Genius	1) 2013-06-28 (planting) 2) 3) 2013-11-05	0.75	210	0.36	2013-07-25	BBCH 18	whole plant	1.5	<0.010	0.16	<0.010	0.67	0	spraying
								brussels sprouts	<0.010	<0.010	<0.010	<0.010	<0.010	103	sample storage time not validated 3: all matrices analytical method: BASF No. L0179/02 (HPLC- MS/MS), LOQ(s): all 0.01 mg/kg max. sample storage time in month(s): 11 ASB2015-296
BASF DocID 2013/1335418, study 428125 (Charles River study No.698005), trial L130416, harvest trial France (FR) 59237 Parquet, Verlinghem 2014-10-24	Aurelius	1) 2013-04-28 (planting) 2) 3) 2013-11-04	0.76	210	0.36	2013-06-14	BBCH 18-19	whole plant	21.0	<0.010	0.23	<0.010	0.22	0	spraying
								brussels sprouts	<0.010	<0.010	<0.010	<0.010	<0.010	143	sample storage time not validated 3: all matrices analytical method: BASF No. L0179/02 (HPLC- MS/MS), LOQ(s): all 0.01 mg/kg max. sample storage time in month(s): 12 ASB2015-296

Trial No./ Location/ Year	Commodity/ Variety	Date of 1.Sowing or planting 2.Flowering 3. Harvest	Application rate per treatment			Dates of treatment or number and last date	Growth stage at last treatment	Portion analysed	Residues (mg/kg)					PHI (days)	Remarks
			g as/ha	Water (L/ha)	g as/hL				(1)	(2)	(3)	(4)	(5)		
BASF DocID 2013/1335418, study 428125 (Charles River study No.698005), trial L130415, harvest trial Germany (DE) 47574 Goch 2014-10-24	Genius	1) 2013-06-28 (planting) 2) 3) 2013-11-06	0.74	210	0.36	2013-07-25	BBCH 18	whole plant	1.0	<0.010	0.040	<0.010	0.16	0	spraying
								<0.010	<0.010	<0.010	<0.010	<0.010	104	<0.010	sample storage time not validated 3: all matrices analytical method: BASF No. L0179/02 (HPLC- MS/MS), LOQ(s): all 0.01 mg/kg max. sample storage time in month(s): 11 ASB2015-296

B.7.3.2.9 Head cabbage

The cGAP in N- and S-EU is 1 x 0.720 kg as/ha spray application at post-emergence stage up to BBCH 16. PHI is covered by the growing period between application and harvest.

Reference:	KCA 6.3.
Report	Martín, T. (2013): Study on the residue behaviour of metazachlor (BAS 479 H) and dimethenamid-P (BAS 656 H) on cabbages (cauliflower and white cabbage) after the application of BAS 769 00 H under field conditions in France (South), Italy and Spain, 2012, Study code: 418108, Doc ID 2013/1182806, ASB2015-285
Guideline(s):	EC 1107/2009 of the European Parliament and of the Council of 21 Oct 2009, EEC 79/117, EEC 7029/VI/95 rev. 5 Appendix B (July 22 1997), EEC 7525/VI/95 rev. 9 (March 2011)
Deviations:	None
GLP:	Yes
Acceptability:	The study is acceptable.

Reference:	KCA 6.3.
Report	Schneider, E. (2014): Study on the residue behaviour of metazachlor (BAS 479 H) and dimethenamid-P (BAS 656 H) in head cabbage after treatment with BAS 769 00 H under field conditions in Northern and Southern Europe, 2013, Study code: 428108, Doc ID 2013/1335412, ASB2015-292
Guideline(s):	EC 1107/2009 of the European Parliament and of the Council of 21 Oct 2009, EEC 79/117, EEC 7029/VI/95 rev. 5 Appendix B (July 22 1997), EEC 7525/VI/95 rev. 9 (March 2011), SANCO/825/00 rev.8.1, SANCO/3029/99 rev.4
Deviations:	None
GLP:	Yes
Acceptability:	The study is acceptable.

Materials and methods

Out of 7 supervised residue trials in white cabbage, 3 trials were conducted in N-EU and 4 trials in S-EU in 2012-13.

Cabbage plants were treated according to the GAP with 1 application at nominal rates of 0.465 - 0.547 kg as/ha. Plants were harvested at 46-124 DALA. The samples were stored deep frozen for max. 12 months. All samples were analysed for dimethenamid-P and the metabolites M23, M26, M27 and M30 according to method BASF L0179/02. For a detailed method description see B.5.2.1.1.

Results and Discussion

Procedural recoveries were within acceptable limits (Table B.7.3-34). Storage of head cabbage samples was validated for dimethenamid-P, M23, M27 and M30, but not for M26. Residue levels in individual samples are reported in Table B.7.3-35 and Table B.7.3-36. No residues were found in untreated control samples.

Table B.7.3-34: Procedural recoveries for dimethenamid-P and metabolites in head cabbage

Matrix	Fortification Level [mg/kg]	Summary recoveries							
		Recovery [%]	Mean [%]	RSD [%]	n	Recovery [%]	Mean [%]	RSD [%]	n
		Dimethenamid-P				M23			
Whole plant	0.01	108, 98, 105, 99, 92	100	6	5	100, 94, 96, 127, 126	109	15	5
	0.1	103	-	-	1	97	-	-	1
	25	89, 93	91	-	2	82, 85	84	-	2
Cabbage head	0.01	133, 127, 104, 102, 106, 102, 108, 99	110	12	8	93, 96, 94, 108, 101, 100, 91, 94, 95, 88, 100	97	6	11
	0.1	71, 116, 99	95	24	3	83, 84, 102	90	12	3
	1	100, 94	97	-	2	96, 90	93	-	2
	10	97	-	-	1	84	-	-	1
		M26				M27			
Whole plant	0.01	74, 66, 69, 75, 81	73	8	5	100, 100, 98, 89, 89	95	6	5
	0.1	91	-	-	1	97	-	-	1
	25	90, 82	86	-	2	87, 90	89	-	2
Cabbage head	0.01	81, 88, 78, 72, 82, 92, 76, 84, 85	82	8	9	89, 88, 101, 116, 94, 107, 92, 90, 85, 87, 88	97	10	11
	0.1	76, 76, 94	82	13	3	75, 76, 103	85	19	3
	1	98, 85	92	-	2	98, 88	93	-	2
	10	79	-	-	1	81	-	-	1
M30									
Whole plant	0.01	120, 112, 106, 80, 81	100	18	5				
	0.1	94	-	-	1				
	25	82, 85	84	-	2				
Cabbage head	0.01	108, 103, 112, 118, 84, 96, 98, 103, 102	103	10	6				
	0.1	80, 82, 101	88	13	3				
	1	93, 86	90	-	2				
	10	80	-	-					

Conclusion

Residues were <0.01 mg/kg per analyte in all samples. Two trials were not considered acceptable as their application rate was not within the 25 % rule of the cGAP. However, sample storage time for metabolite M26 is not covered by storage stability studies evaluated in Vol. 3, section B.7.1.1 of the RAR.

Table B.7.3-35: S-EU residues trials with head cabbage conducted according to GAP

Reference:	Study on the residue behaviour of Metazachlor (BAS 479 H) and dimethenamid-P(BAS 656 H) on cabbages (cauliflower and white cabbage) after the application of BAS 769 00 H under field conditions in France (South), Italy and Spain, 2012, Martín, T., 2013; Study code: 418108, Doc ID 2013/1182806, ASB2015-285				
	Study on the residue behaviour of Metazachlor (BAS 479 H) and dimethenamid-P (BAS 656 H) in head cabbage after treatment with BAS 769 00 H under field conditions in Northern and Southern Europe, 2013, Schneider, E., 2014; Study code: 428108, Doc ID 2013/1335412, ASB2015-292				
GLP:	Yes	Sample storage conditions:			4 - 10 month at -18 °C
Crop/crop group:	White cabbage (BRSOL)	Analytical method:			BASF No. L0179/02
Indoor/Outdoor:	Outdoors (SEU)	Limit of Quantification (mg/kg):			0.01 mg/kg
Formulation:	EC	Limit of Detection (mg/kg):			-
Content of active substance (g/kg or g/L):	200 g/L	Residues calculated as:			(1) dimethenamid-P (2) M23 oxalamide (3) M26 thiolactic acid conjugate (4) M27 sulfonate (5) M30 sulfoxide of thiolactic acid conjugate

Trial No./ Location/ Year	Commodity/ Variety	Date of 1.Sowing or planting 2.Flowering 3. Harvest	Application rate per treatment			Dates of treatment or number and last date	Growth stage at last treatment	Portion analysed	Residues (mg/kg)					PHI (days)	Remarks
			g as/ha	Water (L/ha)	g as/hL				(1)	(2)	(3)	(4)	(5)		
BASF DocID 2013/1182806, study 418108 (PTRL Europe ID P 2804 G), trial L120384, harvest trial Spain (ES) 41710 Utrera, Sevilla, Andalucia 2013-06-27	Leopard	1) 2012-04-09 (planting) 2) 3) 2012-06-12	0.55	110	0.50	2012-04-26	BBCH 18	whole plant	1.2	<0.010	0.020	<0.010	0.030	0	spraying
								head	<0.010	<0.010	<0.010	<0.010	<0.010	46	sample storage time not validated 3: all matrices analytical method: BASF No. L0179/02 (HPLC- MS/MS), LOQ(s): all 0.01 mg/kg max. sample storage time in month(s): 12 ASB2015-285

Trial No./ Location/ Year	Commodity/ Variety	Date of 1. Sowing or planting 2. Flowering 3. Harvest	Application rate per treatment			Dates of treatment or number and last date	Growth stage at last treatment	Portion analysed	Residues (mg/kg)					PHI (days)	Remarks
			g as/ha	Water (L/ha)	g as/hL				(1)	(2)	(3)	(4)	(5)		
BASF DocID 2013/1182806, study 418108 (PTRL Europe ID P 2804 G), trial L120385, harvest trial France (FR) 84700 Sorgues, Provence Alpes Côte d' Azur 2013-06-27	Caïd	1) 2012-06-06 (planting) 2) 3) 2012-10-25	0.48	240	0.20	2012-06-23	BBCH 18	whole plant	0.34	<0.010	<0.010	<0.010	0.040	0	spraying
								head	<0.010	<0.010	<0.010	<0.010	<0.010	124	sample storage time not validated 3: all matrices analytical method: BASF No. L0179/02 (HPLC- MS/MS), LOQ(s): all 0.01 mg/kg max. sample storage time in month(s): 10 ASB2015-285
BASF DocID 2013/1335412, study 428108 (PTRL Europe ID P 3079 G), trial L130440 (B3067 IT1), harvest trial Italy (IT) 12100 Cuneo, San Pietro del Gallo, Piemonte 2014-06-23	Dama	1) 2013-06-10 (planting) 2) 3) 2013-09-25	0.54	110	0.50	2013-07-17	BBCH 18	whole plant	3.7	<0.010	0.029	<0.010	0.32	0	spraying
								head	<u><0.010</u>	<0.010	<u><0.010</u>	<0.010	<u><0.010</u>	70	sample storage time not validated 3: all matrices analytical method: BASF No. L0179/02 (HPLC- MS/MS), LOQ(s): all 0.01 mg/kg max. sample storage time in month(s): 7 ASB2015-292

Trial No./ Location/ Year	Commodity/ Variety	Date of 1.Sowing or planting 2.Flowering 3. Harvest	Application rate per treatment			Dates of treatment or number and last date	Growth stage at last treatment	Portion analysed	Residues (mg/kg)					PHI (days)	Remarks
			g as/ha	Water (L/ha)	g as/hL				(1)	(2)	(3)	(4)	(5)		
BASF DocID 2013/1335412, study 428108 (PTRL Europe ID P 3079 G), trial L130439 (B3067 ES1), harvest trial Spain (ES) 08902 L'Hospitalet de Llobregat; Cataluña 2014-06-23	Col del pais	1) 2013-09-20 (planting) 2) 3) 2013-11-25 - 2013-12-09	0.54	110	0.50	2013-10-08	BBCH 18	whole plant	6.2	<0.010	0.047	<0.010	0.12	0	spraying
								head	<u><0.010</u>	<0.010	<u><0.010</u>	<0.010	<u><0.010</u>	52	sample storage time not validated 3: whole plant analytical method: BASF No. L0179/02 (HPLC- MS/MS), LOQ(s): all 0.01 mg/kg max. sample storage time in month(s): 4 ASB2015-292

Table B.7.3-36: N-EU residues trials with head cabbage conducted according to GAP

Reference: Study on the residue behaviour of Metazachlor (BAS 479 H) and dimethenamid-P (BAS 656 H) in head cabbage after treatment with BAS 769 00 H under field conditions in Northern and Southern Europe, 2013, Schneider, E., 2014; Study code: 428108, Doc ID 2013/1335412, ASB2015-292

GLP: Yes Sample storage conditions: 7 month at -18 °C

Crop/crop group: White cabbage (BRSOL) Analytical method: BASF No. L0179/02

Indoor/Outdoor: Outdoors (NEU) Limit of Quantification (mg/kg): 0.01 mg/kg

Formulation: EC Limit of Detection (mg/kg): -

Content of active substance (g/kg or g/L): 200 g/L Residues calculated as:

(1) dimethenamid-P
(2) M23 oxalamide
(3) M26 thiolactic acid conjugate
(4) M27 sulfonate
(5) M30 sulfoxide of thiolactic acid conjugate

Trial No./ Location/ Year	Commodity/ Variety	Date of 1.Sowing or planting 2.Flowering 3. Harvest	Application rate per treatment			Dates of treatment or number and last date	Growth stage at last treatment	Portion analysed	Residues (mg/kg)					PHI (days)	Remarks
			g as/ha	Water (L/ha)	g as/hL				(1)	(2)	(3)	(4)	(5)		
BASF DocID 2013/1335412, study 428108 (PTRL Europe ID P 3079 G), trial L130435 (B3067 GE1), harvest trial Germany (DE) 69124 Heidelberg 2014-06-23	Verdeco	1) 2013-06-10 (planting) 2) 3) 2013-09-10 - 2013-09-29	0.47	93	0.51	2013-07-09	BBCH 18-19	whole plant	1.2	<0.010	0.015	<0.010	0.20	0	spraying
								head	<0.010	<0.010	<0.010	<0.010	<0.010	63	sample storage time not validated 3: all matrices analytical method: BASF No. L0179/02 (HPLC- MS/MS), LOQ(s): all 0.01 mg/kg max. sample storage time in month(s): 7 ASB2015-292
BASF DocID 2013/1335412,	Zerlina	1) 2013-05-28 (planting)	0.54	110	0.50	2013-07-01	BBCH 18	whole plant	4.5	<0.010	0.025	<0.010	0.058	0	spraying

Trial No./ Location/ Year	Commodity/ Variety	Date of 1.Sowing or planting 2.Flowering 3. Harvest	Application rate per treatment			Dates of treatment or number and last date	Growth stage at last treatment	Portion analysed	Residues (mg/kg)					PHI (days)	Remarks
			g as/ha	Water (L/ha)	g as/hL				(1)	(2)	(3)	(4)	(5)		
study 428108 (PTRL Europe ID P 3079 G), trial L130436 (B3067 AN1), harvest trial France (FR) 67203 Oberschaeffolsheim, Alsace 2014-06-23		2) 3) 2013-09-13 - 2013-09-30						head	<0.010	<0.010	<0.010	<0.010	<0.010	74	sample storage time not validated 3: all matrices analytical method: BASF No. L0179/02 (HPLC- MS/MS), LOQ(s): all 0.01 mg/kg max. sample storage time in month(s): 7 ASB2015-292
BASF DocID 2013/1335412, study 428108 (PTRL Europe ID P 3079 G), trial L130437 (B3067 NL1), harvest trial Netherlands (NL) 6599 AV Ven- Zelderheide, Limburg 2014-06-23	Tolsma RZ F1	1) 2013-07-02 (planting) 2) 3) 2013-10-01	0.53	110	0.50	2013-07-25	BBCH 18	whole plant	4.5	<0.010	0.029	<0.010	0.23	0	spraying
								head	<0.010	<0.010	<0.010	<0.010	<0.010	68	sample storage time not validated 3: all matrices analytical method: BASF No. L0179/02 (HPLC- MS/MS), LOQ(s): all 0.01 mg/kg max. sample storage time in month(s): 7 ASB2015-292

B.7.3.2.10 Chinese cabbage

The cGAP in N- and S-EU is 1 x 0.720 kg as/ha spray application at post-emergence stage up to BBCH 16. The proposed PHI is set to 60 days.

Reference:	KCA 6.3.
Report	Erdmann, H.-P. (2014): Study on the residue behaviour of BAS 656 H (dimethenamid-P) and BAS 479 H (metazachlor) in leafy cabbages (curly kale EU-N and Chinese cabbage EU-S) after application of BAS 769 00 H under field condition in Germany, Belgium, Northern and Southern France, The Netherlands, Greece, Italy and Spain, 2013, Study code: 428096, Doc ID 2013/1335419, ASB2015-297
Guideline(s):	EC 1107/2009 of the European Parliament and of the Council of 21 Oct 2009, EEC 79/117, EEC 7029/VI/95 rev. 5 Appendix B (July 22 1997), EEC 7525/VI/95 rev. 9 (March 2011)
Deviations:	None
GLP:	Yes
Acceptability:	The study is acceptable only if the fall-back GAP with an application rate of 500 g/ha is used.

Materials and methods

A total of 4 supervised residue trials (all S-EU) conducted in 2013 were provided by the applicant. Chinese cabbage plants were treated according to the GAP with 1 application at nominal rates of 0.489 - 0.525 kg as/ha. Plants were harvested at 26-64 DALA. The samples were stored deep frozen for max. 5 months. All samples were analysed for dimethenamid-P and the metabolites M23, M26, M27 and M30 according to method BASF L0179/02. For a detailed method description see B.5.2.1.1.

Results and Discussion

Procedural recoveries and precisions were within acceptable limits (Table B.7.3-37). Storage of Chinese cabbage samples was validated for dimethenamid-P, M23, M27 and M30, but not for M26. Residue levels in individual samples are reported in Table B.7.3-38. No residues were found in untreated control samples.

Table B.7.3-37: Procedural recoveries for dimethenamid-P and metabolites in Chinese cabbage

Matrix	Fortification Level [mg/kg]	Summary recoveries							
		Recovery [%]	Mean [%]	RSD [%]	n	Recovery [%]	Mean [%]	RSD [%]	n
		Dimethenamid-P				M23			
Whole plant	0.01	103, 105, 108	105	2	3	109, 106, 97	104	6	3
	0.1	100	-	-	1	98	-	-	1
	25	104	-	-	1	101	-	-	1
Cabbage Head	0.01	104, 102, 102	103	1	3	107, 106, 114	109	4	3
	0.1	101	-	-	1	98	-	-	1
	1	99	-	-	1	100	-	-	1
		M26				M27			
Whole plant	0.01	87, 71, 65	74	15	3	104, 103, 83	97	12	3
	0.1	91	-	-	1	102	-	-	1
	25	98	-	-	1	98	-	-	1
Cabbage head	0.01	75, 76, 76	76	1	3	104, 101, 104	103	2	3
	0.1	93	-	-	1	102	-	-	1
	1	98	-	-	1	102	-	-	1
		M30							
Whole plant	0.01	97, 112, 100	103	8	3				
	0.1	102	-	-	1				
	25	98	-	-	1				
Cabbage head	0.01	110, 106, 114	110	4	3				
	0.1	100	-	-	1				
	1	97	-	-	1				

Conclusion

Residues were up to 0.062 mg/kg for metabolite M30. With the application rate according to the cGAP being 0.720 kg/ha, all trials have to be considered underdosed, even if the 25 % rule is applied. However, the data may be used in support of a fall-back GAP with an application rate of 500 g/ha. It should be noted that sample storage time for metabolite M26 is not covered by storage stability studies evaluated in Vol. 3 CA, section B.7.1.1 of the RAR.

Table B.7.3-38: S-EU residues trials with Chinese cabbage conducted according to GAP

Reference:	Study on the residue behaviour of BAS 656 H (dimethenamid-P) and BAS 479 H (Metazachlor) in leafy cabbages (curly kale EU-N and chinese cabbage EU-S) after application of BAS 769 00 H under field condition in Germany, Belgium, Northern and Southern France, The Netherlands, Greece, Italy and Spain, 2013, Erdmann, H.-P., 2014; Study code: 428096, Doc ID 2013/1335419, ASB2015-297				
GLP:	Yes	Sample storage conditions:			5 month at -18 °C
Crop/crop group:	Chinese cabbage (BRSPK)	Analytical method:			BASF No. L0179/02
Indoor/Outdoor:	Outdoors (SEU)	Limit of Quantification (mg/kg):			0.01 mg/kg
Formulation:	EC	Limit of Detection (mg/kg):			-
Content of active substance (g/kg or g/L):	200 g/L	Residues calculated as:			(1) dimethenamid-P (2) M23 oxalamide (3) M26 thiolactic acid conjugate (4) M27 sulfonate (5) M30 sulfoxide of thiolactic acid conjugate

Trial No./ Location/ Year	Commodity/ Variety	Date of 1.Sowing or planting 2.Flowering 3. Harvest	Application rate per treatment			Dates of treatment or number and last date	Growth stage at last treatment	Portion analysed	Residues (mg/kg)					PHI (days)	Remarks
			g as/ha	Water (L/ha)	g as/hL				(1)	(2)	(3)	(4)	(5)		
BASF DocID 2013/1335419, study 428096 (PTRL Europe ID P 3078 G), trial L130423 (AC/13/117), harvest trial France (FR) 84170 Monteux, Vaucluse, Provence Alpes Côtes d' Azur 2014-07-14	Chorus	1) 2013-09-06 (planting) 2) 3) 2013-11-11	0.53	210	0.25	2013-09-23	BBCH 18	whole plant	9.5	<0.010	0.050	<0.010	0.19	0	spraying
								head with wrapper leaves	<0.010	<0.010	<0.010	<0.010	0.031	45	sample storage time not validated 3: whole plant analytical method: BASF No. L0179/02 (HPLC- MS/MS), LOQ(s): all 0.01 mg/kg max. sample storage time in month(s): 5 ASB2015-297

Trial No./ Location/ Year	Commodity/ Variety	Date of 1. Sowing or planting 2. Flowering 3. Harvest	Application rate per treatment			Dates of treatment or number and last date	Growth stage at last treatment	Portion analysed	Residues (mg/kg)					PHI (days)	Remarks
			g as/ha	Water (L/ha)	g as/hL				(1)	(2)	(3)	(4)	(5)		
BASF DocID 2013/1335419, study 428096 (PTRL Europe ID P 3078 G), trial L130424 (AC/13/118), harvest trial Greece (GR) 57007 Chalkidona, Thessaloniki 2014-07-14	Guest star	1) 2013-08-23 (planting) 2) 3) 2013-10-15 - 2013-10-30	0.50	200	0.25	2013-09-19	BBCH 18	whole plant	2.4	<0.010	0.012	<0.010	0.14	0	spraying
								head with wrapper leaves	<u><0.010</u>	<0.010	<u><0.010</u>	<0.010	<u>0.020</u>	26	sample storage time not validated 3: all matrices analytical method: BASF No. L0179/02 (HPLC- MS/MS), LOQ(s): all 0.01 mg/kg max. sample storage time in month(s): 5 ASB2015-297
BASF DocID 2013/1335419, study 428096 (PTRL Europe ID P 3078 G), trial L130425 (AC/13/119), harvest trial Italy (IT) 12100 Cuneo, Piemonte 2014-07-14	Yuki F1	1) 2013-08-01 (planting) 2) 3) 2013-11-05 - 2013-11-10	0.49	200	0.25	2013-09-03	BBCH 18	whole plant	12.0	<0.010	0.44	<0.010	0.20	0	spraying
								head with wrapper leaves	<u><0.010</u>	<0.010	<u><0.010</u>	<0.010	<u><0.010</u>	64	sample storage time not validated 3: whole plant analytical method: BASF No. L0179/02 (HPLC- MS/MS), LOQ(s): all 0.01 mg/kg max. sample storage time in month(s): 5 ASB2015-297

Trial No./ Location/ Year	Commodity/ Variety	Date of 1.Sowing or planting 2.Flowering 3. Harvest	Application rate per treatment			Dates of treatment or number and last date	Growth stage at last treatment	Portion analysed	Residues (mg/kg)					PHI (days)	Remarks
			g as/ha	Water (L/ha)	g as/hL				(1)	(2)	(3)	(4)	(5)		
BASF DocID 2013/1335419, study 428096 (PTRL Europe ID P 3078 G), trial L130426 (AC/13/120), harvest trial Spain (ES) 41710 Utrera (Sevilla), Andalucia 2014-07-14	Kazumy	1) 2013-08-23 (planting) 2) 3) 2013-10-17	0.50	200	0.25	2013-09-20	BBCH 18	whole plant	4.5	<0.010	<0.010	<0.010	0.052	0	spraying
								head with wrapper leaves	<u><0.010</u>	<0.010	<u><0.010</u>	<0.010	<u>0.062</u>	26	sample storage time not validated 3: all matrices analytical method: BASF No. L0179/02 (HPLC- MS/MS), LOQ(s): all 0.01 mg/kg max. sample storage time in month(s): 5 ASB2015-297

B.7.3.2.11 Curly kale

The cGAP in N- and S-EU is 1 x 0.720 kg as/ha spray application at post-emergence stage up to BBCH 16. The proposed PHI is set to 60 days.

Reference:	KCA 6.3.
Report	Erdmann, H.-P. (2014): Study on the residue behaviour of BAS 656 H (dimethenamid-P) and BAS 479 H (metazachlor) in leafy cabbages (curly kale EU-N and Chinese cabbage EU-S) after application of BAS 769 00 H under field condition in Germany, Belgium, Northern and Southern France, The Netherlands, Greece, Italy and Spain, 2013, Study code: 428096, Doc ID 2013/1335419, ASB2015-297
Guideline(s):	EC 1107/2009 of the European Parliament and of the Council of 21 Oct 2009, EEC 79/117, EEC 7029/VI/95 rev. 5 Appendix B (July 22 1997), EEC 7525/VI/95 rev. 9 (March 2011)
Deviations:	None
GLP:	Yes
Acceptability:	The study is acceptable only if the fall-back GAP with an application rate of 500 g/ha is used.

Materials and methods

A total of 4 supervised residue trials (all N-EU) conducted in 2013 were provided by the applicant. Curly kale plants were treated according to the GAP with 1 application at nominal rates of 0.475 - 0.514 kg as/ha. Plants were harvested at 35-87 DALA. The samples were stored deep frozen for max. 8 months. All samples were analysed for dimethenamid-P and the metabolites M23, M26, M27 and M30 according to method BASF L0179/02. For a detailed method description see B.5.2.1.1.

Results and Discussion

Procedural recoveries and precisions were within acceptable limits (Table B.7.3-39). Storage of curly kale samples was validated for dimethenamid-P, M23, M27 and M30, but not for M26. Residue levels in individual samples are reported in Table B.7.3-40. No residues were found in untreated control samples.

Table B.7.3-39: Procedural recoveries for dimethenamid-P and metabolites in curly kale

Matrix	Fortification Level [mg/kg]	Summary recoveries							
		Recovery [%]	Mean [%]	RSD [%]	n	Recovery [%]	Mean [%]	RSD [%]	n
		Dimethenamid-p				M23			
Whole plant	0.01	95, 92, 97	95	3	3	94, 104, 101	100	5	3
	0.1	96	-	-	1	86	-	-	1
	1	95	-	-	1	91	-	-	1
	25	100	-	-	1	-	-	-	-
Leaves	0.01	92, 92, 94	93	1	3	99, 101, 106	102	4	3
	0.1	88	-	-	1	97	-	-	1
	1	91	-	-	1	95	-	-	1
		M26				M27			
Whole plant	0.01	83, 98, 82	88	10	3	96, 109, 101	102	6	3
	0.1	82	-	-	1	89	-	-	1
	1	87	-	-	1	90	-	-	1
Leaves	0.01	91, 86, 95	91	5	3	93, 94, 102	96	5	3
	0.1	96	-	-	1	100	-	-	1
	1	97	-	-	1	98	-	-	1
		M30							
Whole plant	0.01	91, 100, 99	97	5	3				
	0.1	85	-	-	1				
	1	85	-	-	1				
Leaves	0.01	107, 94, 104	102	7	3				
	0.1	107	-	-	1				
	1	101	-	-	1				

Conclusion

Residues were up to 0.013 mg/kg for metabolite M30. With the application rate according to the cGAP being 0.720 kg/ha, all trials have to be considered underdosed, even if the 25 % rule is applied. However, the data may be used in support of a fall-back GAP with an application rate of 500 g/ha. It should be noted that sample storage time for metabolite M26 is not covered by storage stability studies evaluated in Vol. 3 CA, section B.7.1.1 of the RAR.

Table B.7.3-40: N-EU residues trials with curly kale conducted according to GAP

Reference:	Study on the residue behaviour of BAS 656 H (dimethenamid-P) and BAS 479 H (Metazachlor) in leafy cabbages (curly kale EU-N and chinese cabbage EU-S) after application of BAS 769 00 H under field condition in Germany, Belgium, Northern and Southern France, The Netherlands, Greece, Italy and Spain, 2013, Erdmann, H.-P., 2014; Study code: 428096, Doc ID 2013/1335419, ASB2015-297				
GLP:	Yes	Sample storage conditions:			7 - 8 month at -18 °C
Crop/crop group:	Curly kale (BRSOC)	Analytical method:			BASF No. L0179/02
Indoor/Outdoor:	Outdoors (NEU)	Limit of Quantification (mg/kg):			0.01 mg/kg
Formulation:	EC	Limit of Detection (mg/kg):			-
Content of active substance (g/kg or g/L):	200 g/L	Residues calculated as:			(1) dimethenamid-P (2) M23 oxalamide (3) M26 thiolactic acid conjugate (4) M27 sulfonate (5) M30 sulfoxide of thiolactic acid conjugate

Trial No./ Location/ Year	Commodity/ Variety	Date of 1.Sowing or planting 2.Flowering 3. Harvest	Application rate per treatment			Dates of treatment or number and last date	Growth stage at last treatment	Portion analysed	Residues (mg/kg)					PHI (days)	Remarks
			g as/ha	Water (L/ha)	g as/hL				(1)	(2)	(3)	(4)	(5)		
BASF DocID 2013/1335419, study 428096 (PTRL Europe ID P 3078 G), trial L130419 (AC/13/113), harvest trial Germany (DE) 16833 Lentzke 2014-07-14	Winnetou	1) 2013-06-14 (planting) 2) 3) 2013-08-05	0.48	190	0.25	2013-07-01	BBCH 18	whole plant	11.0	<0.010	0.017	<0.010	0.029	0	spraying
								leaf	<0.010	<0.010	<0.010	<0.010	0.013	35	sample storage time not validated 3: all matrices analytical method: BASF No. L0179/02 (HPLC- MS/MS), LOQ(s): all 0.01 mg/kg max. sample storage time in month(s): 7 ASB2015-297

Trial No./ Location/ Year	Commodity/ Variety	Date of 1.Sowing or planting 2.Flowering 3. Harvest	Application rate per treatment			Dates of treatment or number and last date	Growth stage at last treatment	Portion analysed	Residues (mg/kg)					PHI (days)	Remarks
			g as/ha	Water (L/ha)	g as/hL				(1)	(2)	(3)	(4)	(5)		
BASF DocID 2013/1335419, study 428096 (PTRL Europe ID P 3078 G), trial L130420 (AC/13/114), harvest trial France (FR) 37340 Ambillou, Centre/Indre et Loire 2014-07-14	Reflex	1) 2013-05-15 (planting) 2) 3) 2013-08-13	0.51	200	0.25	2013-06-14	BBCH 18	whole plant	20.0	<0.010	<0.010	<0.010	<0.010	0	spraying
								leaf	<u><0.010</u>	<0.010	<u><0.010</u>	<0.010	<u><0.010</u>	61	sample storage time not validated 3: all matrices analytical method: BASF No. L0179/02 (HPLC- MS/MS), LOQ(s): all 0.01 mg/kg max. sample storage time in month(s): 8 ASB2015-297
BASF DocID 2013/1335419, study 428096 (PTRL Europe ID P 3078 G), trial L130421 (AC/13/115), harvest trial Netherlands (NL) 6599 AV Ven- Zelderheide, Limburg 2014-07-14	Reflex	1) 2013-05-30 (planting) 2) 3) 2013-09-20	0.51	210	0.25	2013-06-25	BBCH 18	whole plant	6.4	<0.010	<0.010	<0.010	0.014	0	spraying
								leaf	<u><0.010</u>	<0.010	<u><0.010</u>	<0.010	<u><0.010</u>	87	sample storage time not validated 3: all matrices analytical method: BASF No. L0179/02 (HPLC- MS/MS), LOQ(s): all 0.01 mg/kg max. sample storage time in month(s): 8 ASB2015-297

Trial No./ Location/ Year	Commodity/ Variety	Date of 1.Sowing or planting 2.Flowering 3. Harvest	Application rate per treatment			Dates of treatment or number and last date	Growth stage at last treatment	Portion analysed	Residues (mg/kg)					PHI (days)	Remarks
			g as/ha	Water (L/ha)	g as/hL				(1)	(2)	(3)	(4)	(5)		
BASF DocID 2013/1335419, study 428096 (PTRL Europe ID P 3078 G), trial L130422 (AC/13/116), harvest trial Belgium (BE) 3534 Halen, Limburg 2014-07-14	Reflex	1) 2013-06-01 (planting) 2) 3) 2013-09-20	0.49	200	0.25	2013-06-26	BBCH 18	whole plant	2.4	<0.010	<0.010	<0.010	0.037	0	spraying
								leaf	<u><0.010</u>	<0.010	<u><0.010</u>	<0.010	<u><0.010</u>	86	sample storage time not validated 3: all matrices analytical method: BASF No. L0179/02 (HPLC- MS/MS), LOQ(s): all 0.01 mg/kg max. sample storage time in month(s): 8 ASB2015-297

B.7.3.2.12 Garden bean

The cGAP in N- and S-EU is 1 x 0.850 kg as/ha spray application at post-emergence stage up to BBCH 14. PHI is covered by the growing period between application and harvest.

Reference:	KCA 6.3.
Report	Aitken, A.; Munro, M. (2014): Study on the residue behaviour of dimethenamid-P and pendimethalin (BAS 656 H and BAS 455 H) in green beans following one application of either BAS 656 12 H or BAS 659 03 H to 8 trials in 2013 – NEU, Study code: 428110, Doc ID 2013/1335413, ASB2015-293
Guideline(s):	EC 1107/2009 of the European Parliament and of the Council of 21 Oct 2009, EEC 79/117, EEC 7029/VI/95 rev. 5 Appendix B (July 22 1997), EEC 7525/VI/95 rev. 9 (March 2011)
Deviations:	None
GLP:	Yes
Acceptability:	The study is acceptable.

Materials and methods

A total of 8 supervised residue trials (all N-EU) conducted in 2013 were provided by the applicant. Bean plants were treated according to the GAP with 1 application at nominal rates of 0.885 - 0.998 kg as/ha. Plants were harvested at 42-125 DALA. The samples were stored deep frozen for max. 16 months. All samples were analysed for dimethenamid-P and the metabolites M23, M26, M27 and M30 according to method BASF L0179/02. For a detailed method description see B.5.2.1.1.

Results and Discussion

Procedural recoveries and precisions were within acceptable limits (Table B.7.3-41). Storage of bean samples was validated for dimethenamid-P, M23, M27 and M30, but not for M26 in whole plant. Residue levels in individual samples are reported in Table B.7.3-42. No residues were found in untreated control samples.

Table B.7.3-41: Procedural recoveries for dimethenamid-P and metabolites in beans

Matrix	Fortification Level [mg/kg]	Summary recoveries								
		Recovery [%]	Mean [%]	RSD [%]	n	Recovery [%]	Mean [%]	RSD [%]	n	
		Dimethenamid-P				M23				
Whole plant	0.01	83, 80, 73, 93, 94, 95	86	10	6	86, 96, 97	93	7	3	
	1	89, 84	87	-	2	90, 87	89	-	2	
	100	104, 106	105	-	2	-	-	-	-	
Pods with/without seeds	0.01	92, 99, 98	96	4	3	83, 78, 83	81	4	3	
	1	96, 97	97	-	2	93, 90	92	-	2	
Rest of plant	0.01	98, 100, 106, 94	100	5	4	96, 100, 106, 77, 108	97	13	5	
	1	92, 96	94	-	2	85, 88	87	-	2	
Seeds	0.01	91, 91, 86	89	3	3	81, 80, 75	79	4	3	
	0.1	105, 106	106	-	2	91, 94	93	-	2	
	1	93, 95	94	-	2	86, 88	87	-	2	
		M26				M27				
Whole plant	0.01	74, 83, 64, 85, 90, 91	81	13	6	88, 92, 77, 86, 108, 101	92	12	6	
	1	94, 91	93	-	2	98, 96	97	-	2	
Pods with/without seeds	0.01	94, 111, 108	104	9	3	96, 101, 103	100	4	3	
	1	94, 90	92	-	2	96, 92	94	-	2	
Rest of plant	0.01	100, 91, 104, 78, 107	96	12	5	98, 102, 106, 77, 75	92	16	5	
	1	86, 87	87	-	2	86, 87	87	-	2	
Seeds	0.01	89, 90, 90	90	1	3	83, 93, 89	88	6	3	
	0.1	89, 98	94	-	2	87, 98	93	-	2	
	1	90, 96	93	-	2	91, 93	92	-	2	
		M30								
Whole plant	0.01	100, 100, 87	96	8	3					
	1	77, 81	79	-	2					
Pods with/without seeds	0.01	82, 92, 103	92	11	3					
	1	85, 76	81	-	2					
Rest of plant	0.01	76, 76, 101, 75, 96	85	15	5					
	1	74, 77	76	-	2					
Seeds	0.01	81, 80, 76	79	3	3					
	0.1	88, 87	88	-	2					
	1	81, 82	82	-	2					

Conclusion

Residues were <0.01 mg/kg per analyte in all samples. However, sample storage time for metabolite M26 in high water content matrices is not covered by storage stability studies evaluated in Vol. 3 CA, section B.7.1.1 of the RAR.

Table B.7.3-42: N-EU residues trials with garden bean conducted according to GAP

Reference:	Study on the residue behaviour of dimethenamid-P and Pendimethalin (BAS 656 H and BAS 455 H) in green beans following one application of either BAS 656 12 H or BAS 659 03 H to 8 trials in 2013 – NEU, Aitken, A.; Munro, M., 2014; Study code: 428110, Doc ID 2013/1335413, ASB2015-293				
GLP:	Yes	Sample storage conditions:	7 - 16 month at -18 °C		
Crop/crop group:	Garden bean (PHSVX)	Analytical method:	BASF No. L0179/02		
Indoor/Outdoor:	Outdoors (NEU)	Limit of Quantification (mg/kg):	0.01 mg/kg		
Formulation:	EC	Limit of Detection (mg/kg):	-		
Content of active substance (g/kg or g/L):	212.5 g/L and 720 g/L	Residues calculated as:	(1) dimethenamid-P (2) M23 oxalamide (3) M26 thiolactic acid conjugate (4) M27 sulfonate (5) M30 sulfoxide of thiolactic acid conjugate		

Trial No./ Location/ Year	Commodity/ Variety	Date of 1.Sowing or planting 2.Flowering 3. Harvest	Application rate per treatment			Dates of treatment or number and last date	Growth stage at last treatment	Portion analysed	Residues (mg/kg)					PHI (days)	Remarks
			kg as/ha	Water L/ha	kg as/hL				(1)	(2)	(3)	(4)	(5)		
BASF DocID 2013/1335413, study 428110 (Charles River study No. 697991), trial L130572, harvest trial United Kingdom (UK) 47574 Goch- Nierswalde 2014-11-27	Flaforte	1)2013-07-23 (sowing) 2) 3)2013-11-28	0.92	210	0.43	2013-08-22	BBCH 14	whole plant	44.8	<0.010	<0.010	<0.010	<0.010	0	spraying
								rest of plant	<0.010 <0.010	<0.010 0.019	<0.010 <0.010	<0.010 0.025	<0.010 0.016	69 98	sample storage time not validated 3: whole plant, rest of plant, pod with seed, green, pod w/o seed, fresh
								pod with seed, dry	<0.010	<0.010	<0.010	<0.010	<0.010	98	
								pod with seed, green	<u><0.010</u>	<0.010	<u><0.010</u>	<0.010	<u><0.010</u>	69	analytical method: BASF No. L0179/02 (HPLC- MS/MS), LOQ(s): all 0.01 mg/kg
								pod w/o seed, fresh	<0.010	<0.010	<0.010	<0.010	<0.010	69	max. sample storage time in month(s): 7
								pod w/o seed, dry	<0.010	<0.010	<0.010	<0.010	<0.010	98	
								seed, fresh	<0.010	<0.010	<0.010	<0.010	<0.010	69	
								seed, dry	<u><0.010</u>	<0.010	<u><0.010</u>	<0.010	<u><0.010</u>	98	ASB2015-293

Trial No./ Location/ Year	Commodity/ Variety	Date of 1.Sowing or planting 2.Flowering 3. Harvest	Application rate per treatment			Dates of treatment or number and last date	Growth stage at last treatment	Portion analysed	Residues (mg/kg)					PHI (days)	Remarks
			kg as/ha	Water L/ha	kg as/hL				(1)	(2)	(3)	(4)	(5)		
BASF DocID 2013/1335413, study 428110 (Charles River study No. 697991), trial L130575, harvest trial Belgium (BE) 4347 Fexhe le Haut Clocher, Liege 2014-11-27	Flagrano	1) 2013-06-06 (sowing) 2) 3) 2013-09-11	0.89	210	0.43	2013-07-11	BBCH 13-14	whole plant	56.8	<0.010	0.018	<0.010	<0.010	0	spraying spraying
								rest of plant	<0.010 <0.010	<0.010 <0.010	<0.010 <0.010	<0.010 <0.010	<0.010 <0.010	42 62	sample storage time not validated
								pod with seed, dry	<0.010	<0.010	<0.010	<0.010	<0.010	62	3: whole plant, rest of plant, pod with seed, green, pod w/o seed, fresh
								pod with seed, green	<0.010	<0.010	<0.010	<0.010	<0.010	42	analytical method: BASF No. L0179/02 (HPLC- MS/MS), LOQ(s): all 0.01 mg/kg
								pod w/o seed, fresh	<0.010	<0.010	<0.010	<0.010	<0.010	42	max. sample storage time in month(s): 8
								pod w/o seed, dry	<0.010	<0.010	<0.010	<0.010	<0.010	62	
								seed, fresh	<0.010	<0.010	<0.010	<0.010	<0.010	42	ASB2015-293
								seed, dry	<0.010	<0.010	<0.010	<0.010	<0.010	62	
BASF DocID 2013/1335413, study 428110 (Charles River study No. 697991), trial L130576, harvest trial Germany (DE) 04827 Gerichshain, Leipzig 2014-11-27	Flaforte	1) 2013-06-12 (sowing) 2) 3) 2013-10-25	0.82	200	0.42	2013-07-08	BBCH 14	whole plant	61.6	<0.010	<0.010	<0.010	<0.010	0	spraying
								rest of plant	<0.010 <0.010	<0.010 0.015	<0.010 <0.010	<0.010 0.028	<0.010 <0.010	64 109	sample storage time not validated
								pod with seed, dry	<0.010	<0.010	<0.010	<0.010	<0.010	109	3: whole plant, rest of plant, pod with seed, green, pod w/o seed, fresh
								pod with seed, green	<0.010	<0.010	<0.010	<0.010	<0.010	64	analytical method: BASF No. L0179/02 (HPLC- MS/MS), LOQ(s): all 0.01 mg/kg
								pod w/o seed, fresh	<0.010	<0.010	<0.010	<0.010	<0.010	64	max. sample storage time in month(s): 16
								pod w/o seed, dry	<0.010	<0.010	<0.010	<0.010	<0.010	109	ASB2015-293
								seed, fresh	<0.010	<0.010	<0.010	<0.010	<0.010	64	
								seed, dry	<0.010	<0.010	<0.010	<0.010	<0.010	109	

Trial No./ Location/ Year	Commodity/ Variety	Date of 1.Sowing or planting 2.Flowering 3. Harvest	Application rate per treatment			Dates of treatment or number and last date	Growth stage at last treatment	Portion analysed	Residues (mg/kg)					PHI (days)	Remarks
			kg as/ha	Water L/ha	kg as/hL				(1)	(2)	(3)	(4)	(5)		
BASF DocID 2013/1335413, study 428110 (Charles River study No. 697991), trial L130577, harvest trial Netherlands (NL) 6599 AV Ven- Zelderheide, Limburg 2014-11-27	Flaforte	1) 2013-07-23 (sowing) 2) 3) 2013-11-28	0.82	190	0.42	2013-08-22	BBCH 14	whole plant	60.8	<0.010	<0.010	<0.010	<0.010	0	spraying
								rest of plant	<0.010 <0.010	<0.010 0.013	<0.010 <0.010	<0.010 0.025	<0.010 <0.010	68 98	sample storage time not validated
								pod with seed, dry	<0.010	<0.010	<0.010	<0.010	<0.010	98	3: whole plant, rest of plant, pod with seed, green, pod w/o seed, fresh
								pod with seed, green	<0.010	<0.010	<0.010	<0.010	<0.010	68	analytical method: BASF No. L0179/02 (HPLC- MS/MS), LOQ(s): all 0.01 mg/kg
								pod w/o seed, fresh	<0.010	<0.010	<0.010	<0.010	<0.010	68	max. sample storage time in month(s): 15
								pod w/o seed, dry	<0.010	<0.010	<0.010	<0.010	<0.010	98	ASB2015-293
								seed, fresh	<0.010	<0.010	<0.010	<0.010	<0.010	68	
								seed, dry	<0.010	<0.010	<0.010	<0.010	<0.010	98	
BASF DocID 2013/1335413, study 428110 (Charles River study No. 697991), trial L130578, harvest trial France (FR) 62111 Hebuterne, Nord-Pas de Calais 2014-11-27	Flagrano	1) 2013-07-02 (sowing) 2) 3) 2013-10-15	0.85	200	0.43	2013-08-06	BBCH 14	whole plant	26.0	<0.010	0.036	<0.010	<0.010	0	spraying
								rest of plant	<0.010 <0.010	<0.010 <0.010	<0.010 <0.010	0.014 0.017	<0.010 <0.010	49 70	sample storage time not validated
								pod with seed, dry	<0.010	<0.010	<0.010	<0.010	<0.010	70	3: whole plant, rest of plant, pod with seed, green, pod w/o seed, fresh
								pod with seed, green	<0.010	<0.010	<0.010	<0.010	<0.010	49	analytical method: BASF No. L0179/02 (HPLC- MS/MS), LOQ(s): all 0.01 mg/kg
								pod w/o seed, fresh	<0.010	<0.010	<0.010	<0.010	<0.010	49	max. sample storage time in month(s): 7
								pod w/o seed, dry	<0.010	<0.010	<0.010	<0.010	<0.010	70	ASB2015-293
								seed, fresh	<0.010	<0.010	<0.010	<0.010	<0.010	49	
								seed, dry	<0.010	<0.010	<0.010	<0.010	<0.010	70	

Trial No./ Location/ Year	Commodity/ Variety	Date of 1.Sowing or planting 2.Flowering 3. Harvest	Application rate per treatment			Dates of treatment or number and last date	Growth stage at last treatment	Portion analysed	Residues (mg/kg)					PHI (days)	Remarks
			kg as/ha	Water L/ha	kg as/hL				(1)	(2)	(3)	(4)	(5)		
BASF DocID 2013/1335413, study 428110 (Charles River study No. 697991), trial L130579, harvest trial United Kingdom (UK) EH35 5NG Ormiston, East Lothian 2014-11-27	Flagrano	1) 2013-06-12 (sowing) 2013-06-24 (planting) 2) 3) 2013-10-11	0.83	200	0.42	2013-07-08	BBCH 14	whole plant	19.1	<0.010	<0.010	<0.010	<0.010	0	spraying
								rest of plant	<0.010 <0.010	<0.010 <0.010	<0.010 <0.010	0.010 0.010	<0.010 <0.010	74 125	sample storage time not validated 3: whole plant, rest of plant, pod with seed, green, pod w/o seed, fresh analytical method: BASF No. L0179/02 (HPLC- MS/MS), LOQ(s): all 0.01 mg/kg max. sample storage time in month(s): 8 ASB2015-293
								pod with seed, dry	<0.010	<0.010	<0.010	<0.010	<0.010	125	
								pod with seed, green	<u><0.010</u>	<0.010	<u><0.010</u>	<0.010	<u><0.010</u>	74	
								pod w/o seed, fresh	<0.010	<0.010	<0.010	<0.010	<0.010	74	
								pod w/o seed, dry	<0.010	<0.010	<0.010	<0.010	<0.010	125	
								seed, fresh	<0.010	<0.010	<0.010	<0.010	<0.010	74	
								seed, dry	<u><0.010</u>	<0.010	<u><0.010</u>	<0.010	<u><0.010</u>	125	

B.7.3.2.13 Leek

The cGAP in N- and S-EU is 1 x 0.864 kg as/ha spray application at post-emergence stage up to BBCH 18. PHI is covered by the growing period between application and harvest.

Reference:	KCA 6.3.
Report	Erdmann, H.P. (2014). Study on the residue behaviour of BAS 656 H (dimethenamid-P) in Welsh/spring onion (EU-N) and leek (EU-S) after application of BAS 656 12 H under field condition in Germany, Belgium, Northern and Southern France United Kingdom, Greece, Italy and Spain, 2013, Study code: 428107, Doc ID 2013/1335411, ASB2015-291
Guideline(s):	EC 1107/2009 of the European Parliament and of the Council of 21 Oct 2009, EEC 79/117, EEC 7029/VI/95 rev. 5 Appendix B (July 22 1997), EEC 7525/VI/95 rev. 9 (March 2011)
Deviations:	None
GLP:	Yes
Acceptability:	The study is acceptable.

Materials and methods

A total of 4 supervised residue trials (all S-EU) conducted in 2013 were provided by the applicant. Leek plants were treated according to the GAP with 1 application at nominal rates of 0.828 - 0.883 kg as/ha. Plants were harvested at 79-132 DALA. The samples were stored deep frozen for max. 8 months. All samples were analysed for dimethenamid-P and the metabolites M23, M26, M27 and M30 according to method BASF L0179/02. For a detailed method description see B.5.2.1.1.

Results and Discussion

Procedural recoveries and precisions were within acceptable limits (Table B.7.3-43). Storage of leek samples was validated for dimethenamid-P, M23, M27 and M30, but not for M26. Residue levels in individual samples are reported in Table B.7.3-44. No residues were found in untreated control samples.

Table B.7.3-43: Procedural recoveries for dimethenamid-P and metabolites in leek

Matrix	Fortification Level [mg/kg]	Summary recoveries							
		Recovery [%]	Mean [%]	RSD [%]	n	Recovery [%]	Mean [%]	RSD [%]	n
		Dimethenamid-P				M23			
Whole plant	0.01	101, 108, 107, 102	105	3	4	118, 117, 106, 101	111	8	4
	1	108, 103, 88	100	10	3	99, 104, 102	102	2	3
	25	86	-	-	1	-	-	-	-
		M26				M27			
Whole plant	0.01	109, 99, 105, 97	103	5	4	106, 112, 100, 106	106	5	4
	1	99, 112, 96	102	8	3	98, 104, 104	102	3	3
		M30							
Whole plant	0.01	101, 113, 109, 111	109	5	4				
	1	102, 96, 99	99	3	3				

Conclusion

Residues were <0.01 mg/kg per analyte in all samples. However, sample storage time for metabolite M26 is not covered by storage stability studies evaluated in Vol. 3 CA, section B.7.1.1 of the RAR.

Table B.7.3-44: S-EU residues trials with leek conducted according to GAP

Reference:	Study on the residue behaviour of BAS 656 H (dimethenamid-P) in welsh/spring onion (EU-N) and leek (EU-S) after application of BAS 656 12 H under field condition in Germany, Belgium, Northern and Southern France United Kingdom, Greece, Italy and Spain, 2013 Erdmann, H.P., 2014; Study code: 428107, Doc ID 2013/1335411, ASB2015-291				
GLP:	Yes	Sample storage conditions:	8 month at -18 °C		
Crop/crop group:	Leek (ALLPO)	Analytical method:	BASF No. L0179/02		
Indoor/Outdoor:	Outdoors (SEU)	Limit of Quantification (mg/kg):	0.01 mg/kg		
Formulation:	EC	Limit of Detection (mg/kg):	-		
Content of active substance (g/kg or g/L):	720 g/L	Residues calculated as:	(1) dimethenamid-P (2) M23 oxalamide (3) M26 thiolactic acid conjugate (4) M27 sulfonate (5) M30 sulfoxide of thiolactic acid conjugate		

Trial No./ Location/ Year	Commodity/ Variety	Date of 1.Sowing or planting 2.Flowering 3. Harvest	Application rate per treatment			Dates of treatment or number and last date	Growth stage at last treatment	Portion analysed	Residues (mg/kg)					PHI (days)	Remarks
			g as/ha	Water (L/ha)	g as/hL				(1)	(2)	(3)	(4)	(5)		
BASF DocID 2013/1335411, study 428107 (PTRL Europe ID P 3082 G), trial L130454 (AC/13/125), harvest trial France (FR) 26800 Etoile sur Rhône, Rhône-Alpes/ Drôme 2014-04-29	Kenton	1) 2013-07-05 (planting)	0.88	200	0.43	2013-07-26	BBCH 14	whole plant	4.4	<0.010	0.074	<0.010	0.021	0	spraying
		2)							<0.010	<0.010	<0.010	<0.010	<0.010	81	sample storage time not validated 3: whole plant analytical method: BASF No. L0179/02 (HPLC-MS/MS), LOQ(s): all 0.01 mg/kg max. sample storage time in month(s): 8 ASB2015-291
		3) 2013-12-20							<0.010	<0.010	<0.010	<0.010	<0.010	132	

Trial No./ Location/ Year	Commodity/ Variety	Date of 1.Sowing or planting 2.Flowering 3. Harvest	Application rate per treatment			Dates of treatment or number and last date	Growth stage at last treatment	Portion analysed	Residues (mg/kg)					PHI (days)	Remarks
			g as/ha	Water (L/ha)	g as/hL				(1)	(2)	(3)	(4)	(5)		
BASF DocID 2013/1335411, study 428107 (PTRL Europe ID P 3082 G), trial L130455 (AC/13/126), harvest trial Greece (GR) 57007 Chalkidona, Thessaloniki 2014-04-29	Kalamoprasso	1) 2013-06-26 (planting) 2) 3) 2013-09-25 - 2013-11-10	0.87	200	0.43	2013-07-02	BBCH 13-15	whole plant	3.6	<0.010	0.044	<0.010	0.018	0	spraying
									<0.010	<0.010	<0.010	<0.010	<0.010	79	sample storage time not validated
									<0.010	<0.010	<0.010	<0.010	<0.010	111	3: whole plant analytical method: BASF No. L0179/02 (HPLC-MS/MS), LOQ(s): all 0.01 mg/kg max. sample storage time in month(s): 8 ASB2015-291
BASF DocID 2013/1335411, study 428107 (PTRL Europe ID P 3082 G), trial L130456 (AC/13/127), harvest trial Italy (IT) 12050 Castagnito d' Alba, Piemont/Cueno 2014-04-29	Curling F1	1) 2013-07-18 (planting) 2) 3) 2013-10-31	0.86	200	0.43	2013-07-24	BBCH 13-15	whole plant	2.6	<0.010	0.054	<0.010	<0.010	0	spraying
									<0.010	<0.010	<0.010	<0.010	<0.010	79	sample storage time not validated
									<0.010	<0.010	<0.010	<0.010	<0.010	99	3: whole plant analytical method: BASF No. L0179/02 (HPLC-MS/MS), LOQ(s): all 0.01 mg/kg max. sample storage time in month(s): 8 ASB2015-291

Trial No./ Location/ Year	Commodity/ Variety	Date of 1.Sowing or planting 2.Flowering 3. Harvest	Application rate per treatment			Dates of treatment or number and last date	Growth stage at last treatment	Portion analysed	Residues (mg/kg)					PHI (days)	Remarks
			g as/ha	Water (L/ha)	g as/hL				(1)	(2)	(3)	(4)	(5)		
BASF DocID 2013/1335411, study 428107 (PTRL Europe ID P 3082 G), trial L130457 (AC/13/128), harvest trial Spain (ES) 41710 Utrera (Sevilla), Andalucia 2014-04-29	Monstruoso de plata	1) 2013-09-25 (planting) 2) 3) 2013-12-21	0.83	190	0.43	2013-10-02	BBCH 13	whole plant	6.3 <u><0.010</u>	<0.010 <0.010	<0.010 <u><0.010</u>	<0.010 <0.010	<0.010 <u><0.010</u>	0 79	spraying sample storage time not validated 3: whole plant analytical method: BASF No. L0179/02 (HPLC-MS/MS), LOQ(s): all 0.01 mg/kg max. sample storage time in month(s): 5 ASB2015-291

B.7.4 Feeding studies

B.7.4.1 Poultry

No feeding studies with poultry were provided.

B.7.4.2 Ruminants

No feeding studies with ruminants were provided.

B.7.4.3 Pigs

No studies were provided and also considered not necessary due to the similar metabolism of ruminant and rat.

B.7.4.4 Fish

No studies were provided and are also not considered as necessary (see B.7.2.5).

B.7.5 Effects of processing

According to the data requirements as laid down in Commission regulation (EC) 283/2013, such a study has to be provided when residues in products of plant or animal origin subject to processing may occur at a level of or higher than 0.01 mg/kg (based on the residue definition for risk assessment for the raw commodity). As parent dimethenamid-P was not detected in food or feed from controlled field trials, no processing study is necessary.

On the other hand, metabolite M30 is the predominant residue at relevant harvest intervals in edible commodities and animal feed items. Thus a new hydrolysis study has been provided by the applicant.

B.7.5.1 Nature of the residue

Reference:	KCA 6.5.1
Report:	Habeeb S.B., (2014): Simulated processing practices: Hydrolysis of [14-C]-M656PH030 at pH 4 (90 °C), pH 5 (100 °C) and pH 6 (120 °C), BASF DocID: 2013/7002635, Ricerca Doc No: 030930-1, BASF Study No: 390494, Ricerca Study No: 030930 ASB2014-8366
Guidelines:	Yes (OECD Guideline 507)
Deviations:	No
GLP:	Yes
Acceptability:	The study is acceptable.

Materials and methods

Test Material:

Description:	M30 (C ¹⁴ , C ¹³ and unlabelled)
Lot/Batch #:	1089-1003 (C ¹⁴), 1088-1005 (C ¹³), L74-138 (unlabelled)
Purity:	Radiochemical: 98 % (C ¹⁴)

Spiking level: Chemical: 95.8 % (C¹⁴), 92.6 % (C¹³), 83.7 % (unlabelled)
10 µg/mL

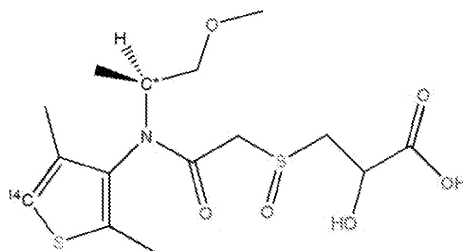


Figure B.7.5-1: Chemical structure and position of radioactive label of metabolite M30

Buffers of pH 4.0, 5.0 and 6.0 were prepared according and tested for accuracy to the protocol. Individual test systems (in triplicate) were prepared in triplicate in amber glass vials containing ¹⁴C-M30 test material in buffered aqueous solutions. Triplicate test vessels were placed in the water bath set at 90±5 °C for the pH 4 buffer for 20 minutes and 100±5 °C for the pH 5 buffer for 60 minutes. Triplicate test vessels were placed in the autoclave set at 120±5 °C for the pH 6 buffer for 20 minutes. Each test system was sterilised prior to the use in this study.

At sampling for each buffer spiked with ¹⁴C-M30, triplicate test samples for pH 4, pH 5 and pH 6 were removed from the constant temperature water bath. Triplicate aliquots were analysed by LSC to determine the radioactivity in the sample. An aliquot of each sample was transferred into an amber sterile HPLC vial and analysed by HPLC with radio-detection for distribution of radioactivity.

Results and discussions

Total ¹⁴C recovery (based on the applied radioactivity) for the pH 4 hydrolysis samples at 90±5 °C ranged from 94.5-109 % TAR, for the pH 5 hydrolysis samples at 100±5 °C ranged from 102-110 % TAR, and for pH 6 hydrolysis samples at 120±5 °C ranged from 103-108 % TAR.

Table B.7.5-1: High temperature hydrolysis of M656PH030

Process represented	Test condition	Recovery of ¹⁴ C-M656PH030 (%)		
		Average	Range	RSD
Pasteurization	pH 4, 90 °C, 0 min	97.8	90.2-105	7.6
	pH 4, 90 °C, 20 min	96.3	90.8-102	5.8
Baking/brewing/boiling	pH 5, 100 °C, 0 min	98.6	96.8-98.3	0.8
	pH 5, 100 °C, 60 min	98.0	95.4-102	3.7
Sterilisation	pH 6, 120 °C, 0 min	100	98-102	2.0
	pH 6, 120 °C, 20 min	95.1	94.4-96.3	1.1

Conclusion

This study demonstrated that M30 does not hydrolyse at pH 4, pH 5 and pH 6 after 20 min, 60 min and 20 min, respectively, of incubation of the samples at 90±5, 100±5, and 120±5 °C.

Only minor hydrolysis products were observed at pH 4, pH 5, and pH 6 and none of the products were greater than 5 % TAR and were not identified. The test compound M30 is hydrolytically stable at the pH and the temperature levels tested in the study. On the basis of these results, M30 is not expected to hydrolyse during the pasteurisation (90 °C), baking/brewing/boiling (100 °C), and sterilisation (120 °C).

B.7.5.2 Distribution of the residue in peel and pulp

Not relevant based on the intended uses.

B.7.5.3 Magnitude of residues in processed commodities

Studies on the magnitude of residues in processed commodities is not necessary since residues in the commodities to be processed were below LOQ (<0.01 mg/kg). Additionally, the contribution of the commodities under consideration to the theoretical maximum daily intake (TMDI) is <10 % of the ADI and the estimated daily intake is <10 % of the ARfD.

B.7.6 Residues in rotational crops

B.7.6.1 Metabolism in rotational crops

During the initial EU review of the active substance, dimethenamid-P residues in rotational crops were investigated using [3-¹⁴C-thienyl]-labelled dimethenamid (DAR, 2000, [ASB2010-10566](#)). After Annex I inclusion, one additional rotational crop study using ¹⁴C-labelled dimethenamid-P was evaluated by France in 2013. Both studies were also reviewed by EFSA under Article 12 (EFSA 2013, [ASB2013-6081](#)).

Reference: KCA 6.6.1/3
Report: Wenzel, N.; Ockert, M.; Possienke, M. (2012): Nachbaustudie mit ¹⁴C-BAS 656 H (14C-markiertes Dimethenamid-P) - Confined rotational crop study with 14C-BAS 656 H (¹⁴C-labelled dimethenamid-P)
BASF DocID: 2012/1162137, Study code: 380192
[ASB2014-8367](#)
Guidelines: US EPA OPPTS 860.1850, US EPA OPPTS 860.1000, Commission of the European Communities 7524/VI/95 rev .2, Appendix C, OECD Guideline for the testing of chemicals No 502: Metabolism in Rotational Crops (January 2007)
Deviations: No
GLP: Yes
Acceptability: The study is acceptable.

Reference: KCA 6.6.1/3
Report: Wenzel, N.(2014): Amendment No. 1 to report: Nachbaustudie mit ¹⁴C-BAS 656 H (¹⁴C-markiertes Dimethenamid-P) - Confined rotational crop study with ¹⁴C-BAS 656 H (¹⁴C-labelled dimethenamid-P)
BASF DocID: 2014/1046558, Study code: 380192
[ASB2014-8368](#)
Guidelines: US EPA OPPTS 860.1850, US EPA OPPTS 860.1000, Commission of the European Communities 7524/VI/95 rev .2, Appendix C, OECD Guideline for the testing of chemicals No 502: Metabolism in Rotational Crops (January 2007)
Deviations: No
GLP: Yes
Acceptability: The study is acceptable.

Material and methods

Test Material: Dimethenamid-p (BAS 656 H)
Lot/Batch #: 824-4024 (thienyl-5-¹⁴C)

L74-174 (unlabelled)
Purity: Radiochemical purity: 99.3 % (thienyl-5-¹⁴C)
Chemical purity a.s.: 99.4 % (unlabelled)
Specific activity: 7.63 MBq/mg
CAS#: 163515-14-8

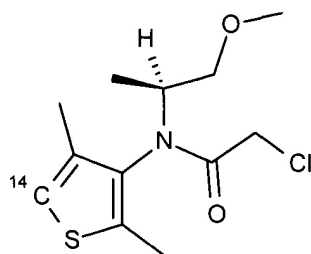


Figure B.7.6-1: Chemical structure and position of radioactive label of dimethenamid-P

Crop:	Spring wheat	white radish	spinach
Type:	Cereals	root & tuber vegetables	leafy vegetables
Variety:	ThassosApril	Cross	Corvette F1
Botanical name:	<i>Triticum aestivum</i>	<i>Raphanus sativus</i>	<i>Lactuca sativa</i>
Crop part/processed			
Commodity:	Wheat forage, wheat hay, wheat straw, wheat grain Radish top, radish root Immature spinach, mature spinach		
Soil:	A sandy loam soil was used. The soil physicochemical properties are described below (see Table B.7.6-1).		

Table B.7.6-1: Soil physicochemical properties

Soil series	Soil type	pH	TOC % ²	Sand %	Silt %	Clay %	Max. water holding capacity	CEC ¹ meq/100 g
Bruch West	Sandy loam*	7.0**	1.36	65.9*	22.1*	11.9*	27.2*	9.6

¹ Cation exchange capacity

² Total organic carbon

* USDA scheme

** CaCl₂

Experimental conditions:

The study was conducted during the period of May 2010 to August 2012 at the BASF Agricultural Center Limburgerhof, in Limburgerhof, Germany. Plastic containers with sandy loam were used for the plant back intervals (PBI) of 30, 120 and 365 days. One single spray application of ¹⁴C-dimethenamid-P was performed on the soil. After 30 days and simulated ploughing, three crops were sowed (spinach, white radish and spring wheat). After harvest of the mature crops and completion of the respective plant back interval, the top layer of the soil was mixed before replanting (individual plant back intervals of 120 days and 365 days). The maintenance of the growing crops was performed using normal agricultural practice. For preparation of the test item ¹⁴C-dimethenamid-P (dissolved in acetonitrile) and unlabelled dimethenamid-P were mixed at a ratio of about 1:1 and dissolved in a mixture of blank formulation of BAS 656 AB H and water. The homogenous emulsion was then applied to the containers at a rate of 1.006 g as/ha (1.2 N) in a spray volume of 214 L/ha. Considering losses during the application process 99.31 % of the application solution was actually applied.

Sampling and sample storage:

Immature and mature spinach leaves were sampled 27-30 and 34-37 days after days after sowing, respectively. Mature white radishes were harvested at 64-71 days after sowing and separated into the root and green parts (tops). Immature green plants of spring wheat (wheat forage) were sampled and partly dried to wheat hay at 43-52 days after sowing. In addition, mature wheat ears and straw were collected at 100-119 days after sowing. The ears were separated into grain and chaff and the chaff was mixed to the straw. Soil samples were taken after the individual plant back intervals and after harvest of the mature crops. All samples were stored in a freezer at -18 °C or below until analysis. The extracts were stored in a refrigerator or, for longer periods, in a freezer.

Extraction of residues and analytical determination:

Homogenisation/TRR determination:

Plant matrices with a sufficient level of radioactivity were extracted with solvents, while for samples with low concentrations of radiolabelled compounds the amount of the total radioactive residues (TRR) was obtained only by combustion analysis. Prior to extraction, the plant and soil sample materials were frozen in liquid nitrogen and chopped/ground to obtain homogenisates. Aliquots of the homogenised samples were dried and combusted using a sample oxidiser.

Extraction/TRR determination:

Aliquots of homogenised plant material were extracted three times with methanol. After each extraction step, the liquid phase was separated from the solid by centrifugation, decanted and filtered, while the remaining plant material was subjected to the next extraction step. The methanol extracts of the three steps were combined, adjusted to volume, and aliquots of the combined extract were measured by LSC. The residue was further extracted in the same way with appropriate volumes of water twice. The water extracts were also combined and aliquots of both extracts were radio-assayed by LSC. The combined results of methanol extractions and water extractions are referred to as extractable radioactive residues (ERR).

The residues after solvent extraction were dried and combusted for the determination of the residual radioactive residue (RRR). The total radioactive residues (TRR calculated) were obtained by calculating the sum of ERR and RRR values and additionally by combustion of sample aliquots (TRR measured). All calculations throughout the present study were based on the TRR calculated.

Partitioning/ERR determination:

Subsamples of methanol extracts of plant matrices after a plant back interval of 30 DAT (with exception of white radish root and spring wheat grain) were evaporated to the water phase and partitioned three times with dichloromethane. Afterwards, the remaining water phases of the respective plant samples were partitioned three times with ethyl acetate. Aliquots of the dichloromethane, ethyl acetate and water phases were analysed by LSC measurement and the ERR was calculated.

Solubilisation/RRR determination:

The residual radioactive residues after extraction with methanol and water (RRR) with a sufficient level of radioactivity were subsequently extracted twice with 1 % ammonia. After ammonia extraction, the residues were solubilised with different carbohydrate and protein cleaving enzymes. For selected wheat samples, additional acidic (boiling for 3 h with 6 M HCl) and alkaline hydrolyses (boiling for 3 h with 6 M NaOH) were performed. The amount of radioactivity in the final residue was determined by combustion.

Radioanalysis:

Aliquots of homogenised solid plant and soil samples were combusted by means of a sample oxidiser. ¹⁴C standards were combusted to determine the recovered radioactivity and the measurements were corrected accordingly (recovery values between 91 % and 97 %). For determination of the background radioactivity, aliquots of untreated wheat straw samples and white radish root samples were combusted under the same conditions.

Identification of components:

The identification of the metabolites was based on yeast fermentation procedures (investigation of the highly polar components observed in the HPLC analyses of white radish root), on co-chromatography experiments with a blend of carbohydrates (fructose, glucose and sucrose), the two metabolites M81 and M51 identified in a dimethenamid-P metabolism study in soya bean and on HPLC retention time comparison. HPLC with LSC detection was used for the analysis of extracts, partition phases, solubilisates and reference items. Identification/structure elucidation and quantification of metabolites

was accomplished by mass spectrometry (ESI-MS/MS).

Results and discussion

Radioactive residue and extractability:

Plant

All control samples were subjected to combustion analysis and no ^{14}C above background was detected in these samples. The treated samples were subjected to combustion analysis and the results are presented in Table B.7.6-2 for plants and in Table B.7.6-3 for soil. The calculated TRR of all extracted rotational crop matrices showed no major differences to the TRR values obtained by combustion.

For the representative leafy crop, spinach, total radioactive residues (TRR calculated) in both immature and mature plants sowed 30 days after soil treatment with ^{14}C -dimethenamid-P were determined. The total residue level in immature spinach accounted for 0.084 mg/kg, while the residues in mature spinach (PBI 30 days) amounted to 0.095 mg/kg. Lower residue levels for both immature and mature spinach were found at a PBI of 120 days (both 0.008 mg/kg, TRR combusted). After a year of soil aging (PBI 365 days, TRR combusted), the residue concentrations in immature and mature spinach had decreased further, down to 0.005 mg/kg and 0.004 mg/kg, respectively.

For the representative root crop, white radish, total radioactive residues in roots and in the top part of the mature crop were determined. The total radioactive residues in the top part of mature crops were 0.089 mg/kg at a PBI of 30 days, decreasing to 0.012 mg/kg and to 0.002 mg/kg (TRR combusted) after a PBI of 120 days and 365 days of soil aging, respectively. The total radioactive residues in roots of white radish decreased from 0.028 mg/kg (PBI 30 days), to 0.009 mg/kg (PBI 120 days, TRR combusted) and finally to 0.001 mg/kg (PBI 365 days, TRR combusted).

For the representative cereal crop, spring wheat, the matrix with the highest residue levels was hay with an initial TRR of 0.818 mg/kg (PBI 30 days), which decreased to 0.121 mg/kg (PBI 120 days), and finally to 0.032 mg/kg (PBI 365 days) followed by straw where the levels decreased as follows: 0.514 mg/kg (PBI 30 days), 0.137 mg/kg (PBI 120 days) and 0.036 mg/kg (PBI 365 days). Residue levels in grain were 0.193 mg/kg (PBI 30 days), 0.083 mg/kg (PBI 120 days) and 0.035 mg/kg (PBI 365 days).

Spring wheat forage was the matrix with the lowest residue levels. Again, the total radioactive residues after the different soil aging intervals decreased from 0.128 mg/kg (PBI 30 days) to 0.019 mg/kg (PBI 120 days) and to 0.008 mg/kg (PBI 365 days, TRR combusted).

The limit of quantitation (LOQ), determined for spring wheat straw and white radish root was found to be approximately 0.0002 mg/kg and below 0.00005 mg/kg, respectively.

Table B.7.6-2: TRR, ERR and RRR in rotational crop samples after treatment with ¹⁴C-dimethenamid-P

Matrix	Days after sowing	TRR ¹	TRR ²	Methanol extract		Water extract		ERR ³		RRR ⁴	
		[mg/kg]	[mg/kg]	[mg/kg]	[% TRR]	[mg/kg]	[% TRR]	[mg/kg]	[% TRR]	[mg/kg]	[% TRR]
PBI: 30 days											
Immature spinach	28	0.090	0.084	0.054	63.8	0.003	3.9	0.057	67.7	0.027	32.3
Mature spinach	37	0.094	0.095	0.061	63.8	0.007	7.7	0.068	71.5	0.027	28.5
White radish top	71	0.106	0.089	0.057	64.3	0.007	8.2	0.065	72.5	0.025	27.5
White radish root	71	0.028	0.028	0.018	63.2	0.001	4.3	0.019	67.5	0.009	32.5
Spring wheat forage	50	0.142	0.128	0.056	44.0	0.004	3.2	0.060	47.2	0.067	52.8
Spring wheat hay	50	0.925	0.818	0.284	34.7	0.084	10.3	0.368	44.9	0.451	55.1
Spring wheat straw	119	0.537	0.514	0.159	31.0	0.074	14.3	0.233	45.4	0.280	54.6
Spring wheat grain	119	0.201	0.193	0.010	5.1	0.013	6.5	0.022	11.5	0.171	88.5
PBI: 120 days											
Immature spinach	30	0.008	-	-	-	-	-	-	-	-	-
Mature spinach	42	0.008	-	-	-	-	-	-	-	-	-
White radish top	65	0.013	0.012	0.007	55.8	0.001	9.3	0.008	65.1	0.004	34.9
White radish root	65	0.009	-	-	-	-	-	-	-	-	-
Spring wheat forage	52	0.023	0.019	0.008	40.8	0.001	4.2	0.009	45.0	0.011	55.0
Spring wheat hay	52	0.137	0.121	0.034	27.9	0.014	11.2	0.047	39.1	0.073	60.9
Spring wheat straw	107	0.156	0.137	0.048	35.5	0.021	15.4	0.070	50.9	0.067	49.1
Spring wheat grain	107	0.076	0.083	0.005	5.6	0.012	13.9	0.016	19.5	0.067	80.5
PBI: 365 days											
Immature spinach	27	0.005	-	-	-	-	-	-	-	-	-
Mature spinach	34	0.004	-	-	-	-	-	-	-	-	-
White radish top	64	0.002	-	-	-	-	-	-	-	-	-
White radish root	64	0.001	-	-	-	-	-	-	-	-	-
Spring wheat forage	43	0.008	-	-	-	-	-	-	-	-	-
Spring wheat hay	43	0.039	0.032	0.007	20.5	0.004	13.0	0.011	33.5	0.021	66.5
Spring wheat straw	100	0.030	0.036	0.010	27.6	0.005	13.2	0.015	40.8	0.021	59.2
Spring wheat grain	100	0.036	0.035	0.002	5.6	0.003	7.4	0.005	13.0	0.031	87.0

1 Total radioactive residue (TRR): was measured after combustion

2 Total radioactive residue (TRR): was calculated as the sum of ERR + RRR

3 Extractable radioactive residue (ERR) calculated as sum of methanol and water extract

4 Residual radioactive residue

Soil

For all soil samples, the residue levels (TRR) were determined by direct combustion analysis of subsamples (see Table B.7.6-3). Measurements were carried out before as well as after soil aging and ploughing. Additionally, soil was sampled after harvest of the individual mature crops for each plant back interval. The residue concentration in the top soil layer after application accounted for 7.589 mg/kg. After aging and ploughing, the residue concentration decreased from 0.152 mg/kg at PBI 30 days to 0.085 mg/kg at PBI 120 days and 0.096 mg/kg at PBI 365 days. After harvest of the mature

crops, the residue levels in soil remained more or less stable for the plant back intervals of 30, 120 and 365 days (except for a slightly higher value for the soil after harvest of spinach at plant back interval 30 days).

Table B.7.6-3: Total radioactive residues in soil after treatment with ^{14}C -dimethenamid-P

Soil samples	Time after sowing	TRR determined by direct combustion
	[days]	[mg/kg]
PBI: 30 days		
<u>After application (petri dishes)</u>		
0 DAT	-	7.589
PBI: 30 days		
<u>After ploughing</u>		
30 DAT	0	0.152
<u>After harvest of mature crops</u>		
Spinach (67 DAT)	37	0.119
White radish (101 DAT)	71	0.063
Spring wheat (149 DAT)	119	0.095
PBI: 120 days		
<u>After ploughing</u>		
120 DAT	0	0.085
<u>After harvest of mature crops</u>		
Spinach (162 DAT)	42	0.057
White radish (185 DAT)	65	0.075
Spring wheat (227 DAT)	107	0.080
PBI: 365 days		
<u>After ploughing</u>		
365 DAT	0	0.096
<u>After harvest of mature crops</u>		
Spinach (399 DAT)	34	0.057
White radish (429 DAT)	64	0.087
Spring wheat (465 DAT)	100	0.077

Characterisation and identification:

Extraction and characterisation of residues in rotational crops

The extractability of the radioactive residues with methanol and water ranged from 33.5 % to 72.5 % TRR for spinach, white radish and spring wheat forage, hay and straw (see Table B.7.6-2). For spring wheat grain, the extractability was relatively low with approximately 12 to 20 %. The major portions of the radioactive residues were generally extracted with methanol, except for spring wheat grain where almost similar portions were extracted with methanol and water.

In order to characterise the methanol extractable radioactive residues as organosoluble or water soluble fractions, liquid/liquid partition of the rotational crop matrices harvested at PBI 30 days (except for white radish root and spring wheat grain) was carried out using dichloromethane and ethyl acetate as organic solvents (data not shown). In most cases, major portions of the radioactive residues extracted with methanol were water soluble, and only lower portions were found in the organic

fractions. In the case of spring wheat forage, comparable portions were found in the organic phases (sum) and in the water phase.

In most of the rotational crop matrices analysed, considerable amounts of the radioactive residues were not extractable with methanol and water. The residual radioactive residues after solvent extraction of particular matrices of the plant back intervals 30, 120 and 365 days were further characterised using an individual combination of sequential solubilisation steps including treatment with ammonia, hydrochloric acid and sodium hydroxide and solubilisation with enzymes. The solubilised residues had possibly been associated with or embedded/incorporated insoluble plant material such as proteins, cell wall polymers and starch. The most effective solubilisation steps were treatments with macerozyme and glucosidase, followed by hydrochloric acid and sodium hydroxide treatment and solubilisation with aqueous ammonia. In the case of wheat grain, incubation with amylases also released substantial amounts of radioactivity.

Identification and quantification of extractable residues in rotational crops

The identification of the metabolites was based on yeast fermentation procedures, on co-chromatography experiments with a blend of carbohydrates (fructose, glucose and sucrose) and the two metabolites M81 and M51 identified in a dimethenamid-P metabolism study in soya bean and on HPLC retention time comparison.

The radioactive residues in the extracts and solubilisates of all rotational crop matrices consisted of a highly polar fraction (6.8 to 81.2 % TRR), which is mainly composed of carbohydrates. In addition, the metabolites M81 (glucose conjugate of a demethoxylated and twofold S-oxidised methanethiol conjugate of the dechlorinated parent compound) and M51 (twofold S-oxidised 2-mercapto-acetic acid conjugate of the dechlorinated parent compound) were identified in individual rotational crop matrices and accounted for a maximum of 11.2 and 4.2 % TRR, respectively. Furthermore, degradation products in minor concentrations were characterised by their chromatographic properties (ranging from <0.1 to 7.6 % TRR, each below 0.023 mg/kg). The parent molecule was neither detected in the extracts nor in the solubilisates.

The residual radioactive residues after solvent extraction with methanol and water (Table B.7.6-2) ranged from a minimum of 27.5 % TRR (white radish top, PBI 30 days) to a maximum of 88.5 % TRR (spring wheat grain, PBI 30 days). The residual radioactive residues were further characterised using an individual combination of sequential solubilisation steps applying ammonia treatment, incubations with macerozyme, glucosidase, amylases, tyrosinase and treatments with hydrochloric acid and sodium hydroxide (data not shown). The most effective solubilisation steps were the treatments with macerozyme, hydrochloric acid and/or sodium hydroxide, glucosidase and solubilisation with aqueous ammonia releasing up to 35.7 % TRR (spring wheat hay, PBI 365 days), up to 26.6 % TRR (spring wheat forage, PBI 120 days), up to 22.3 % TRR (spring wheat grain, PBI 30 days) and up to 10.9 % TRR (spring wheat grain, PBI 30 days), respectively (data not shown). In addition, wheat grain incubation with amylases released up to 18.4 % TRR (spring wheat grain, PBI 30 days). The high solubilisation of residual radioactive residues after enzyme treatments indicates extensive metabolism of dimethenamid-P to C1 and C2 units with subsequent incorporation into natural products.

Table B.7.6-4: Summary of identified components in rotational crop matrices

Crop matrix	Polar fraction		M81		M51	
	[mg/kg]	[% TRR]	[mg/kg]	[% TRR]	[mg/kg]	[% TRR]
PBI: 30 days						
Immature spinach	0.026	31.4	n.d.		n.d.	
Mature spinach	0.022	22.7	n.d.		n.d.	
White radish top	0.016	18.0	0.10	11.2	n.d.	
White radish root	0.021	74.8	n.d.		n.d.	
Spring wheat forage	0.030	23.8	0.006	4.4	0.005	4.2
Spring wheat hay	0.273	33.3	0.026	3.2	0.021	2.6
Spring wheat straw	0.098	19.2	0.014	2.8	0.008	1.5
Spring wheat grain	0.157	81.2	n.d.		n.d.	
PBI: 120 days						
White radish top	0.002	18.0	<0.001	1.7	<0.001	1.3
Spring wheat forage	0.003	13.5	0.001	5.3	0.001	2.8
Spring wheat hay	0.016	13.5	0.004	3.4	0.003	2.9
Spring wheat straw	0.009	6.8	0.005	3.3	0.002	1.5
Spring wheat grain	0.012	13.9	n.d.		n.d.	
PBI: 365 days						
Spring wheat straw	0.004	10.7	n.d.		n.d.	

n.d. Not detected

Proposed metabolic pathway:

The radioactive residues in the extracts and solubilisates of all rotational crop matrices consisted of a highly polar fraction primarily containing carbohydrates (6.8 to 81.2 % TRR). In addition, the metabolites M81 and M51 were identified in individual rotational crop matrices and accounted for a maximum of 11.2 and 4.2 % TRR, respectively. Furthermore, degradation products in minor concentrations were characterised by their chromatographic properties (ranging from <0.1 to 7.6 % TRR, each below 0.023 mg/kg). These components probably were intermediates in the degradation of dimethenamid-P to C1 or C2 units which finally entered anabolic biosynthetic pathways to natural products. The solubilisation and characterisation of considerable parts of these non-extractable residues by enzymatic cleavage of natural macromolecules might indicate a subsequent incorporation into plant polysaccharides. The identified metabolic pathway is shown in Figure B.7.6-2.

Conclusion

Dimethenamid-P was extensively metabolised in rotational crop matrices after application to soil and translocation into the plants. Soil metabolites of dimethenamid-P were taken up and transformed in rotational crops primarily into carbohydrates which were identified in all matrices examined. In addition, the metabolites M81 (glucose conjugate of a demethoxylated and twofold S-oxidised methanethiol conjugate of the dechlorinated parent compound) and M51 (twofold S-oxidised 2-mercapto-acetic acid conjugate of the dechlorinated parent compound) were identified in rotational crop matrices. Unchanged parent compound was neither detected in extracts nor in solubilisates. The solubilisation and characterisation of considerable parts of the non-extractable residues by enzymatic cleavage of natural macromolecules indicates a subsequent incorporation of ¹⁴C labelled C1 or C2 units into plant polysaccharides.

Taking under consideration of the cGAP approximated by the application rate of this study, no residues of parent dimethenamid-P, M26, M27 or M30 have to be expected in rotational crops. In radish tops and wheat feed commodities (forage, hay and straw) residues of M81 were found at levels up to 0.1 mg/kg under the situation of crop failure (PBI 30 d). M81 is the glycosylated metabolite of M14, which was also identified in the rat metabolism. However, M14 did occur in very small amounts only and can therefore not be considered as toxicologically covered by the parent substance. Therefore its toxicological relevance remains unclear. Most of the radioactivity was characterised as polar residues, mainly containing carbohydrates.

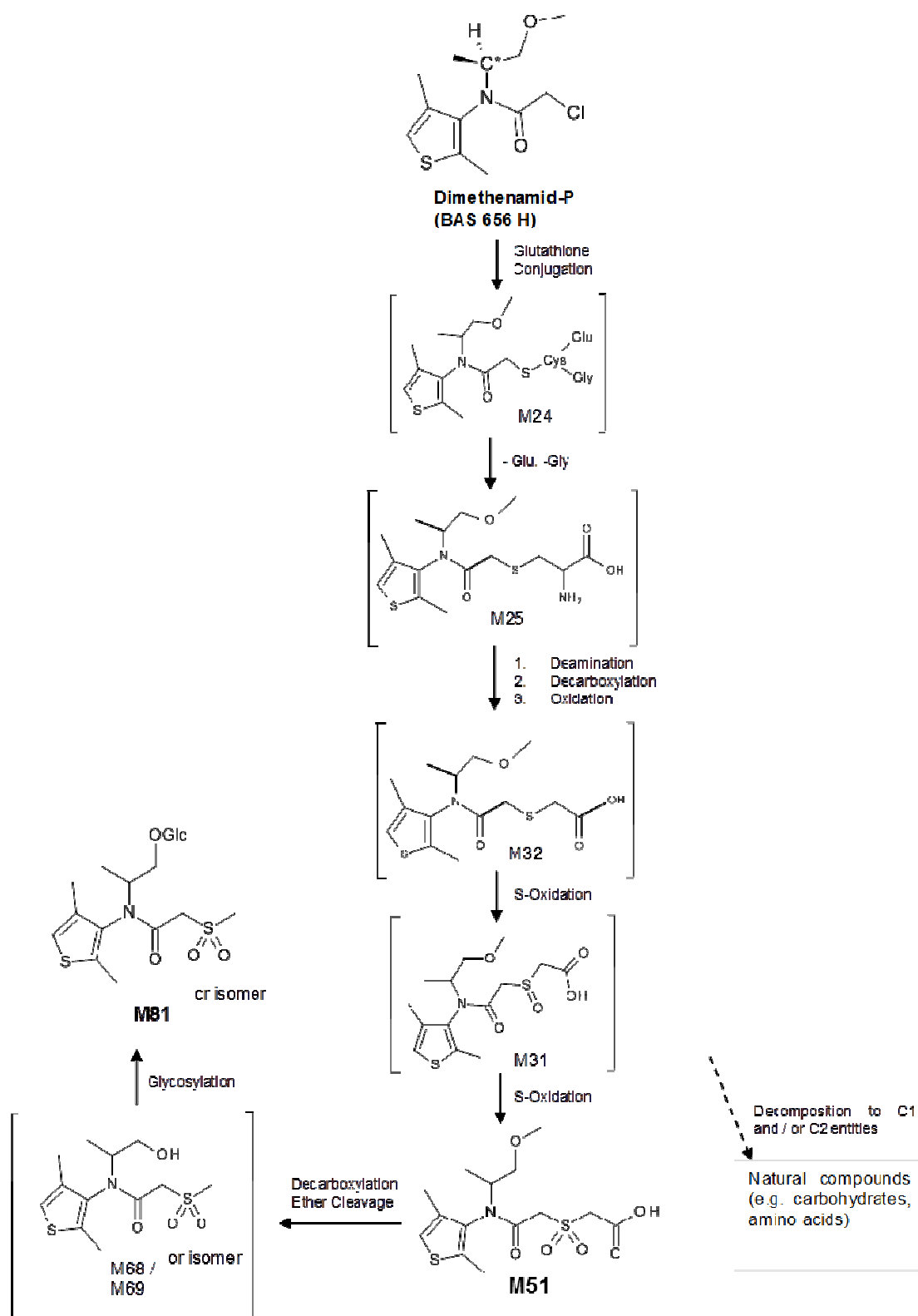


Figure B.7.6-2: Metabolic pathway of dimethenamid-P (BAS 656 PH) in rotational crops

Reference:	KCA 6.6.1/1
Report:	Pierotti, M. V.; Moore P. A. (1992): Confined accumulation study of SAN-582H on rotational crops BASF DocID: 92/12425, Project No. 414105, Report No. 17 RIP1999-861
Guidelines:	EPA Pesticide Assessment Guideline, Environmental Fate, Series 165-1
Deviations:	No
GLP:	Yes
Acceptability:	The study is considered supplementary only.
Reference:	KCA 6.6.1/2
Report:	Moore P. A. (1992): Amendment to final report "Confined accumulation study of SAN-582H on rotational crops" BASF DocID: 92/12426, Project No. 414105, Report No. 17 RIP1999-862
Guidelines:	EPA Pesticide Assessment Guideline, Environmental Fate, Series 165-1
Deviations:	No
GLP:	Yes
Acceptability:	The study is considered supplementary only.

Material and methods

Test Material:	Dimethenamid (SAN582H)
Lot/Batch #:	RA 683-1 (thienyl-3- ¹⁴ C), RS-SAN-110389 (unlabelled)
Purity:	RA 683-1: 99.6 % radiochemical purity RS-SAN-110389: 99.6 % chemical purity
Specific activity:	17196 dpm/μg (0.29 MBq/mg)
CAS#:	87674-68-8

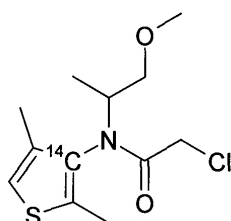


Figure B.7.6-3: Chemical structure and position of radioactive label of racemic dimethenamid

Test commodity			
Type:	Cereals	Leafy vegetable	Root vegetable
Crop:	Wheat	Lettuce	Carrot
Botanical name:	<i>Triticum</i> L.	<i>Lactuca sativa</i>	<i>Daucus carota</i>
Commodity:	Immature wheat, wheat straw, wheat grain		
	Lettuce		
	Carrot tops, root		

Experimental conditions:

A confined rotational crop study was performed on plots in Illinois, USA where maize and soya beans have been grown as primary crops (used for metabolism studies). The residues of dimethenamid in succeeding crops were investigated after soil treatment with racemic ¹⁴C-dimethenamid:

Treatment rates were in maize 1.68 (1.9 N) and 4.4 kg as/ha (5.1 N) and in soya bean 1.68 (1.9 N) and 3.36 kg as/ha (4 N). Maize and soya bean RACs were collected for the metabolism studies were harvested at DAT 130 and DAT 128, respectively. The confined rotation study began with planting of winter wheat at a PBI of 11 days. Additional crops were planted the following spring at a PBI of

192/194 days (spring wheat) and a PBI of 202/204 days (lettuce and carrot). The soil characteristic is summarised in Table B.7.6-5.

Table B.7.6-5: Soil used to investigate radioactive residues in succeeding crops

Soil designation / Origin	Geneseo, Illinois, USA
Soil Type	Silty Clay Loam
% Organic matter	2.5 %
Textural analysis: Sand Silt Clay	19 % 53 % 28 %
Cation exchange capacity [meq/100 g]	15.8
Soil pH	6.3

Sampling and sample storage:

RACs harvested for residue determination were forage (winter wheat: 348 DAT, spring wheat: 364 DAT), seed and straw (winter wheat: 387 DAT, spring wheat: 396 DAT), roots of carrots (408 DAT) and above ground portion of lettuce (364 DAT). All samples were stored at -18 °C to -24 °C.

Extraction of residues and analytical determination:

The TRR of each sample was determined by combustion analysis. Soil and crops were extracted in 1:1 CH₃OH/H₂O. After removal of the methanol, the aqueous fraction was partitioned with hexane and methylene chloride, subsequently. The extracted aqueous fraction was freeze-dried and the resulting residue dissolved in methanol and then deionised water. Bound residue in plant samples was released by acidic and/or alkaline hydrolysis. The soil bound residue was released by alkaline hydrolysis. Extracts yielding residues ≤0.01 mg/kg were analysed by thin layer chromatography.

Results and discussion

Radioactive residue and extractability:

The TRRs for all rotational crop RACs after treatment at a normal use rate (1.68 kg as/ha) were <0.18 mg/kg (maximum in wheat straw). The TRR in edible parts such as wheat grain, carrots, and lettuce were <0.04 mg/kg. The TRR distribution is summarised in Table B.7.6-6:

Table B.7.6-6: TRR in succeeding crops and soil

Sample	Primary crop	Days after Planting	Days after Harvest	Plant back interval (days)	Residues [mg/kg]	
					Maximum rate ¹	Exaggerated rate ²
Soil 0-10 cm 10-14/15 cm	Maize	0	-		1.94 ³ -	3.57 ³ 0.07 ³
Soil 0-10 cm 10-13 cm	Soya bean	0	-		0.85 ³ -	1.52 ³ 0.009
Winter wheat: immature winter wheat grain straw	Maize	141	387	11	0.0208 0.0264 0.1744	0.0662 0.0728 0.3944
Spring wheat: Immature spring wheat grain straw	Maize	322	396	192	0.035 0.02 0.121	0.115 0.052 0.450
Spring wheat: Immature spring wheat grain straw	Soya bean	322	396	194	0.062 0.023 0.142	0.153 0.051 0.484
Lettuce	Maize	332	364	202	0.018	0.073
Lettuce	Soya bean	332	364	204	0.038	0.093
Carrot: top root	Maize	332	408	202	0.059 0.013	0.147 0.038
Carrot: top root	Soya bean	332	408	204	0.073 0.013	0.125 0.026

¹) maximum rate for maize and soya bean was 1.68 kg as/ha

²) exaggerated rate for maize was 4.48 kg as/ha and for soya bean was 3.36 kg as/ha

³) average of four replicate soil samples

The extractable radioactivity (ERR) and non-extractable (RRR) in all rotational crops from the 1.68 kg as/ha treatment are presented in Table B.7.6-7. In all RACs the extractability in organic solvents ranged from 56 - 95 % of total TRR except for wheat grain where the range was 19.9 - 33.7 %. All extractable residues in edible rotational crop RACs were below or slightly above 0.01 mg/kg. The bound residues present in wheat straw were further released by acid and base hydrolysis and additional 0.0074-0.0454 mg/kg (6.1 - 26 % TRR) was released. The extracts of immature spring wheat, spring wheat straw, winter wheat straw, lettuce and carrot tops were the only extracts that were further characterised by TLC.

Table B.7.6-7: Extraction of radioactivity from rotational crops planted after maize and soya bean [mg/kg] (% TRR)

Sample	Maize plots (1.68 kg as/ha)		Soya bean plots (1.68 kg as/ha)	
	ERR	RRR	ERR	RRR
Winter wheat immature	0.0148 (71.2)	0.0096 (46.2)	- ¹	-
Winter wheat grain	0.0089 (33.7)	0.0110 (42.0)	- ¹	-
Winter wheat straw	0.0984 (56.4)	0.0345 (19.8)	- ¹	-
Spring wheat immature	0.0229 (64.9)	0.0089 (25.2)	0.0407 (66.0)	0.0103 (16.7)
Spring wheat grain	0.0049 (25.0)	0.0059 (29.6)	0.0046 (19.9)	0.0072 (31.2)
Spring wheat straw	0.0727 (60.2)	0.0454 (37.6)	0.0801 (56.5)	0.0437 (30.8)
lettuce	0.0111 (60.6)	0.0086 (47.0)	0.0256 (67.0)	0.0126 (33.0)
carrot tops	0.0460 (78.0)	0.0110 (17.0)	0.0610 (77.0)	0.0130 (16.4)
carrot roots	0.0124 (95.4)	0.0047 (36.2)	0.0100(76.9)	0.0030 (23.1)

¹⁾ samples were not collected

Characterisation and identification:

Three metabolites were identified in the rotational crops:

- M23 (Oxalamide),
- M27 (the sulfonate)
- M30 (the sulfoxide of the thiolactic acid conjugate).

All metabolites were present at ≤ 0.01 mg/kg. Unidentified metabolites were also ≤ 0.01 mg/kg. The oxalamide was identified in carrot tops (0.0013 mg/kg, 2.2 % TRR). The sulfoxide of the thiolactic acid conjugate was identified in winter wheat straw (0.01 mg/kg, 5.7 % TRR), lettuce (0.0041 mg/kg, 10.7 % TRR), spring wheat straw (0.0037 mg/kg, 3.1 % TRR) and carrot tops (0.0018 - 0.003 mg/kg, 3.1 - 4.1 % TRR). The sulfonate was identified in immature spring wheat (0.0024 - 0.0077 mg/kg, 6.6-12.5 % TRR). Bound residues were incorporated into the natural carbon pool of the rotational crop as lignin (≤ 0.0247 mg/kg, ≤ 14.2 % TRR) and cellulose (≤ 0.0207 mg/kg, ≤ 11.9 % TRR). Dimethenamid was not detected in any RAC. Unidentified metabolites were also ≤ 0.01 mg/kg.

Proposed metabolic pathway:

Based on the identified metabolites the proposed metabolic pathway involves conjugation of dimethenamid with glutathion and hydrolysis of the glutathion conjugate to the cysteine conjugate, which is the same metabolic pathway identified in the maize, sugar beet, hen, goat and rat metabolism studies.

Conclusion

Regarding the study design, the rotational interval covering the scenario of crop failure (7 - 30 days) is missing. Also, the plant back after 141 days was performed only for wheat, but not for carrots and lettuce. Radioactivity in samples was sufficiently characterised and/or identified, showing no accumulation of dimethenamid or its degradation products in the parts of plants used for human food or animal feed in the case of root vegetable (carrots), green vegetables (lettuce) and cereal grains. Levels of the individual metabolites present were below 0.01 mg/kg. As the study cannot be considered as state-of-the art and is furthermore superseded by the study by Wenzel (2014, [ASB2014-8368](#)) it is only considered as supplementary.

B.7.6.2 Magnitude of residues in rotational crops

No studies provided.

B.7.7 Other studies

No studies provided.

B.7.7.1 Effect on the residue level in pollen and bee products

No studies provided.

B.7.8 References relied on

The applicant has not provided a search of published literature. Therefore, a literature search was performed in order to identify relevant studies in the peer reviewed literature. The search strategy comprised of combining “dimethenamid” with words or word fragments typical in residue studies such as residue*, metabol*, limit*, stabilit*, degrade*, crop*, soil* and water*. These combinations were used with the following literature databases: Pub Med, Scopus, Tox Net, Web of Science. None of the identified studies from these sources was considered relevant with regards to residues as they were either analytical method studies, efficacy studies or environmental studies.

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KCA 6.1	Lehmann A.	2014	Investigation of the storage stability of BAS 656 H (Dimethenamid-P) and its metabolites M23, M26, M27 and M30 in plant matrices 2013/1335905 BASF SE, Limburgerhof, Germany Fed.Rep. GLP, unpublished BVL-2630276, ASB2014-8339	N	Y	New data for AIR3 renewal	BASF	N
KCA 6.1	Oppinger M.	2014	Investigation of the storage stability of BAS 656 H (Dimethenamid-P) and its metabolites M23, M26, M27 and M30 in oily matrices 2013/1335906 BASF SE, Limburgerhof, Germany Fed.Rep. GLP, unpublished BVL-2630277, ASB2014-8340	N	Y	New data for AIR3 renewal	BASF	N
KCA 6.2.1	Atallah, Y.H.; Moore, P.A.; Bade, T.R.	1991	Uptake, translocation and metabolism of the herbicide SAN-582H in soybean 1991/11879 ! DP-300212 ! 414105 GLP, unpublished BVL-1913765, RIP2006-564	N	Y	-	BASF	
KCA 6.2.1	Moore P.A., Wendt, D.R.	1995	Corn metabolism of 14C-[3-thienyl]-dimethenamid SANDOZ Agro, Inc., Des Plaines, Illinois 60018 BASF RegDoc.#95/10129 GLP, unpublished BVL-1913761, RIP1999-827	N	N	Not applicable	BASF	Y Relevant IIA. 6.1.1 [6.1/01]
KCA 6.2.1	Lam W.	1999	[3- ¹⁴ C-thienyl]dimethenamid: metabolism in sugarbeet BASF Corp, ARC, RTP, NC USA BASF RegDoc.#98/5173 GLP, unpublished BVL-1913800, RIP1999-828	N	N	Not applicable	BASF	Y Relevant IIA. 6.1.2 [6.1/02]

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KCA 6.2.1	Bross M., Glaessgen W. E.	2007	Metabolism of 14C-Dimethenamid (14C-BAS 656 H) in corn 2006/1024513 BASF AG Agrarzentrum Limburgerhof, Limburgerhof, Germany Fed.Rep. GLP, unpublished BVL-2630278, ASB2013-9749	N	Y	New data for AIR3 renewal	BASF	N IIA. 6.1
KCA 6.2.1	Schweda Z. et al.	2012	Metabolism of 14C-BAS 656-P H (14C-labeled Dimethenamid-P) in soybean 2012/1144379, 380191, 2014/1036947 BASF SE, Limburgerhof, Germany Fed. Rep. GLP, unpublished BVL-2630280, ASB2014-8341	N	Y	New data for AIR3 renewal	BASF	N IIA. 6.1
KCA 6.2.1	Schweda Z.	2014	Amendment No. 1 - Metabolism of 14C-BAS 656-P H (14C-labeled Dimethenamid-P) in soybean 2014/1036947 BASF SE, Limburgerhof, Germany Fed. Rep. GLP, unpublished BVL-2705592, ASB2014-10169	N	Y	New data for AIR3 renewal	BASF	N IIA. 6.1
KCA 6.2.2	██████████ ██████████ ██████████.	1990	Metabolism of SAN 582 H in laying hens ██ ██ BASF RegDoc.# 90/11110 GLP, unpublished BVL-1913799, RIP1999-834	Y	N	Not applicable	BASF	Y relevant IIA. 6.2.2 [6.2/06]
KCA 6.2.2	██████████ ██████████ ██████████	1992	SAN 582 H: Addendum to a previous hen metabolism study ██ ██ BASF RegDoc.# 92/12430 GLP, unpublished BVL-1913795, RIP1999-835	Y	N	Not applicable	BASF	Y relevant IIA. 6.2.2 [6.2/07]
KCA 6.2.3	██████████ ██████████ ██████████	1990	Metabolism of SAN 582 H in a lactating goat ██ ██ BASF RegDoc.# 90/11112 GLP, unpublished BVL-1913760, RIP1999-829	Y	N	Not applicable	BASF	Y relevant IIA. 6.2.1 [6.2/01]
KCA 6.2.3	██████████ ██████████ ██████████	1992	SAN 582 H: Addendum to a previous goat metabolism study ██ ██ BASF RegDoc.# 92/12431 GLP, unpublished BVL-1913808, RIP1999-830	Y	N	Not applicable	BASF	Y relevant IIA. 6.2.1 [6.2/02]

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KCA 6.2.3	[REDACTED]	1990	Material balance investigation in a goat orally administered 14C-SAN 582 H [REDACTED] [REDACTED] BASF RegDoc.# 90/11113 GLP, unpublished BVL-1913775, RIP1999-831	Y	N	Not applicable	BASF	Y relevant IIA. 6.2.1 [6.2/03]
KCA 6.2.3	[REDACTED]	1992	Addendum to previous goat metabolism studies [REDACTED] BASF RegDoc.# 92/12432 GLP, unpublished BVL-1913783, RIP1999-832	Y	N	Not applicable	BASF	Y relevant IIA. 6.2.1 [6.2/04]
KCA 6.2.3	[REDACTED]	1992	SAN 582 H: Determination of the presence of sulfonate metabolite in goat excreta [REDACTED] BASF RegDoc.# 92/12499 GLP, unpublished BVL-1913794, RIP1999-833	Y	N	Not applicable	BASF	Y relevant IIA. 6.2.1 [6.2/05]
KCA 6.2.3/1	[REDACTED]	2014	M656PH030 ? Metabolism of 14C-M656PH030 in the lactating goat 2013/7002636 [REDACTED] [REDACTED] GLP, unpublished BVL-2360282, ASB2014-8342	Y	Y	New data for AIR3 renewal	BASF	N II A 6.2
KCA 6.3	Aitken, A.; Munro, M.	2014	Study on the residue behaviour of Dimethenamid-P and Pendimethalin (BAS 656 H and BAS 455 H) in green beans following one application of either BAS 656 12 H or BAS 659 03 H to 8 trials in 2013 - NEU 428110 ! 2013/1335413 ! 697991 GLP, unpublished BVL-2746757, ASB2015-293	N	Y	-	BASF	N
KCA 6.3	Aitken, A.; Munro, M.	2014	Study on the residue behaviour of Dimethenamid-P (BAS 656 H) in brussel sprouts following one application of BAS 656 12 H to 4 trials in 2013 - NEU 428125 ! 2013/1335418 ! 698005 GLP, unpublished BVL-2746765, ASB2015-296	N	Y	-	BASF	N

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KCA 6.3	Erdmann, H.P.	2014	Study on the residue behaviour of BAS 656 H (Dimethenamid-P) in welsh/spring onion (EU-N) and leek (EU-S) after application of BAS 656 12 H under field condition in Germany, Belgium, Northern and Southern France United Kingdom, Greece, Italy and Spain, 2013 2013/1335411 ! 428107 ! AC/BASF/13/12 ! P 3082 G GLP, unpublished BVL-2746749, ASB2015-291	N	Y	-	BASF	N
KCA 6.3	Erdmann, H.P.	2014	Study on the residue behaviour of BAS 656 H (Dimethenamid-P) and BAS 479 H (Metazachlor) in leafy cabbages (curly kale EU-N and chinese cabbage EU-S) after application of BAS 769 00 H under field condition in Germany, Belgium, Northern and Southern France, The Netherlands, Greece, Italy and Spain, 2013 428096 ! 2013/1335419 ! AC/BASF/13/11 GLP, unpublished BVL-2746766, ASB2015-297	N	Y	-	BASF	N
KCA 6.3	Erdmann, H.-P.	2013	Study on the residue behaviour of BAS 656 H (Dimethenamid-P) and BAS 479 H (Metazachlor) in spring oilseed rape after application of BAS 769 00 H under field condition in United Kingdom, Belgium, The Netherlands and Germany, 2012 2013/1003730 ! 422462 23.08.2013 GLP, unpublished BVL-2746711, ASB2015-284	N	Y	New data for AIR3 renewal	BASF	-
KCA 6.3	Erdmann, H.-P.	2013	Amendment No. 1 - Study on the residue behaviour of BAS 656 H (Dimethenamid-P) and BAS 479 H (Metazachlor) in spring oilseed rape after application of BAS 769 00 H under field condition in UK, BE, NL and DE, 2012 422462 ! 2013/1377012 21.11.2013 GLP, unpublished BVL-2746771, ASB2015-298	N	Y	New data for AIR3 renewal	BASF	-

Data Point EU as of 2014	Author(s)	Year	Title Company Report No. Source (where different from company) GLP or GEP status Published or not	Vertebrate study Y/N	Data Protection Claimed Y/N	Justification if data protection is claimed	Owner	Previously submitted Y/N If yes, old data point
KCA 6.3	Martín, T.	2013	Study on the residue behavior of Metazachlor (BAS 479 H) and Dimethenamid-P (BAS 656 H) on cabbages (cauliflower and white cabbage) after the application of BAS 769 00 H under field conditions in France (South), Italy and Spain, 2012 2013/1182806 !418108!12/PH/008 ! PTRL Europe ID P 2804 G GLP, unpublished BVL-2746719, ASB2015-285	N	Y	-	BASF	N
KCA 6.3	Meyer M.	2014	Residue behaviour of Dimethenamid-P (BAS 656 H) in sunflower after treatment with BAS 656 12 H under field conditions in Germany, Northern France, The Netherlands, United Kingdom, Belgium, Southern France, Italy, Greece and Spain, 2013 2013/1335422 SGS Institut Fresenius GmbH, Taunusstein, Germany Fed. Rep. GLP, unpublished BVL-2630283, ASB20148343	N	Y	New data for AIR3 renewal	BASF	N II A 6.3
KCA 6.3	Ertus C.	2014	Study on the residue behaviour of BAS 656 H (Dimethenamid-P) after treatment with BAS 656 12 H in sunflower under field conditions in Northern Europe, 2012 2013/1335405 Anadiag SA, Haguenau, France GLP, unpublished BVL-2630285, ASB2014-8344	N	Y	New data for AIR3 renewal	BASF	N II A 6.3
KCA 6.3	Gabriel E.J.	2013	Study on the residue behaviour of Dimethenamid-P (BAS 656 H) in sunflower after treatment with BAS 656 12 H under field conditions in Germany, the Netherlands, Northern France, Southern France, Greece, Italy and Spain, 2012 2012/1272620 SGS Institut Fresenius GmbH, Taunusstein, Germany Fed. Rep. GLP, unpublished BVL-2630287, ASB2014-8345	N	Y	New data for AIR3 renewal	BASF	N II A 6.3

Data Point EU as of 2014	Author(s)	Year	Title Company Report No. Source (where different from company) GLP or GEP status Published or not	Vertebrate study Y/N	Data Protection Claimed Y/N	Justification if data protection is claimed	Owner	Previously submitted Y/N If yes, old data point
KCA 6.3	Meyer M.	2014	Residue behaviour of Quinmerac (BAS 518 H) and Dimethenamid-P (BAS 656 H) in oilseed rape after treatment with BAS 830 01 H under field conditions in Germany, The Netherlands, United Kingdom, Southern France, Italy and Spain, 2013 2013/1335420 SGS Institut Fresenius GmbH, Taunusstein, Germany Fed. Rep. GLP, unpublished BVL-2630289, ASB2014-8346	N	Y	New data for AIR3 renewal	BASF	N II A 6.3
KCA 6.3	Oxspring S.	2014	Study on the residue behaviour of Dimethenamid-P (BAS 656 H) in soybean after treatment with BAS 656 12 H in Northern and Southern Europe during 2013 2013/1335421 Eurofins Agroscience Services, Melbourne Derbyshire DE73 8AG, United Kingdom GLP, unpublished BVL2630291, ASB2014-8347	N	Y	New data for AIR3 renewal	BASF	N II A 6.3
KCA 6.3	Gabriel E.J., Meyer M.	2013	Study on the residue behaviour of Dimethenamid-P (BAS 656 H) in maize after treatment with BAS 656 12 H under field conditions in Germany, Northern France, United Kingdom, the Netherlands, Southern France, Greece, Italy and Spain, 2012 2012/1272621 SGS Institut Fresenius GmbH, Taunusstein, Germany Fed. Rep. GLP, unpublished BVL-12630293, ASB2014-10046	N	Y	New data for AIR3 renewal	BASF	N II A 6.3
KCA 6.3	Perny A.	2013	Study on the residue behaviour of BAS 656 H (Dimethenamid-P) after treatment with BAS 656 12 H in corn under field conditions in Northern and Southern Europe, 2011 2012/1209625 Anadiag SA, Haguenau, France GLP, unpublished BVL-2630295, ASB2014-3704	N	Y	New data for AIR3 renewal	BASF	N II A 6.3

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KCA 6.3	Erdmann H.-P.	2013	Study on the residue behaviour of BAS 656 H (Dimethenamid-P) in beets after application of BAS 656 12 H under field condition in United Kingdom, Northern and Southern France, The Netherlands, Italy and Germany, 2012 2013/1003729 Agro-Check Dr. Teresiak & Erdmann GbR, Lentzke, Germany Fed. Rep. GLP, unpublished BVL-2630297, ASB2014-3705	N	Y	New data for AIR3 renewal	BASF	N II A 6.3
KCA 6.3	Perny A.	2012	Study on the residue behaviour of BAS 656 H (Dimethenamid-P) after treatment with BAS 656 12 H in beets (sugar) under field conditions in Northern and Southern Europe, 2011 2012/1182982 Anadiag SA, Haguenau, France yes Unpublished BVL-2630299 ASB2014-3703	N	Y	New data for AIR3 renewal	BASF	N II A 6.3
KCA 6.3	Roche, A.	2014	Study on the residue behaviour of BAS 656 H (Dimethenamid-P) after soil treatment with BAS 656 12 H in melon and pumpkin under field conditions in Northern Europe, 2013 2013/1335406 ! 428112 ! B3137 GLP, unpublished BVL-2746721, ASB2015-286	N	Y	-	BASF	N
KCA 6.3	Roche, A.	2014	Study on the residue behaviour of Dimethenamid-P after soil treatment with BAS 656 12 H in cucumber and zucchini under field conditions in Northern Europe, 2013 428113 ! 2013/1335415 ! B3065 GLP, unpublished BVL-2746758, ASB2015-294	N	Y	-	BASF	N

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KCA 6.3	Schneider, E.	2014	Study on the Residue Behaviour of Metazachlor (BAS 479 H) and Dimethenamid-P (BAS 656 H) in Cauliflower and Broccoli after Treatment with BAS 769 00 H under field conditions in Northern and Southern Europe, 2013 - 2014 428106 ! 2013/1335410 ! B3066 ! PTRL Europe ID P 3080 G GLP, unpublished BVL-2746745, ASB2015-290	N	Y	-	BASF	N
KCA 6.3	Schneider, E.	2014	Study on the residue behaviour of Metazachlor (BAS 479 H) and Dimethenamid-P (BAS 656 H) in head cabbage after treatment with BAS 769 00 H under field conditions in Northern and Southern Europe, 2013 428108 ! 2013/1335412 ! B3067 GLP, unpublished BVL-2746756, ASB2015-292	N	Y	-	BASF	N
KCA 6.5	Bade T.R.	1992	Stability of SAN-582 H and its metabolites in stored frozen corn samples BASF Corp, ARC, RTP, NC USA BASF RegDoc.# 92/12400 GLP, unpublished BVL-1913772 RIP1999-859	N	N	Not applicable	BASF	Y not relevant IIA. 6.3.2 [6.3/25]
KCA 6.5	Wisson M.	1994	Dimethenamid stability in various solvents SANDOZ Agro, Ltd., Basle, CH BASF RegDoc.# 94/11821 GLP, unpublished BVL-1913768 RIP1999-860	N	N	Not applicable	BASF	Y not relevant IIA. 6.3.2 [6.3/26]
KCA 6.5.1	Habeeb S.B.	2014	Simulated processing practices: Hydrolysis of [14-C]-M656PH030 at pH 4 (90°C), pH 5 (100°C) and pH 6 (120°C) 2013/7002635 Ricerca Biosciences LLC, Concord OH, United States of America GLP, unpublished BVL-2630301, A, OSB2014-8366	N	Y	New data for AIR3 renewal	BASF	N II A 6.5.1
KCA 6.6	Pierotti M.V., Moore P.A.	1992	Confined accumulation study of SAN-582H on rotational crops SANDOZ Agro, Inc., Des Plaines, Illinois 60018 BASF RegDoc.#92/12425 GLP, unpublished BVL-1913786, RIP1999-861	N	N	Not applicable	BASF	Y not relevant IIA. 6.6 [6.6/01]

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KCA 6.6	Moore P.A.	1992	Amendment to final report "confined accumulation study on SAN-582H on rotational crops" project no. 414105 SANDOZ Agro, Inc., Des Plaines, Illinois 60018 BASF RegDoc.#92/12426 GLP, unpublished BVL-1913787, RIP1999-862	N	N	Not applicable	BASF	Y not relevant IIA. 6.6 [6.6/02]
KCA 6.6.1	Wenzel N. et al.	2012	Nachbaustudie mit 14C-BAS 656 H (14C-markiertes Dimethenamid-P) - Confined rotational crop study with 14C-BAS 656 H (14C-labelled Dimethenamid-P) 2012/1162137 BASF SE, Limburgerhof, Germany Fed. Rep. GLP, unpublished BVL-2630303, ASB2014-8367	N	Y	New data for AIR3 renewal	BASF	N IIA. 6.6
KCA 6.6.1	Wenzel N.	2014	Amendment No. 1 - Nachbaustudie mit 14C-BAS 656 H (14C-markiertes Dimethenamid-P) - Confined rotational crop study with 14C-BAS 656 H (14C-labelled Dimethenamid-P) 2014/1046558 BASF SE, Limburgerhof, Germany Fed. Rep. GLP, unpublished BVL-2630304, ASB2014-8368	N	Y	New data for AIR3 renewal	BASF	N IIA. 6.6
	EFSA	2013	Reasoned opinion on the review of the existing maximum residue levels (MRLs) for Dimethenamid-P according to Article 12 of Regulation (EC) No 396/2005 EFSA Journal 2013;11(4):3216 ! EFSA-Q-2008-526 ASB2013-6081	N	N	-	LIT	-
	Germany	2000	Dimethenamid-P (Monograph) Not GLP, published ASB2010-10566	N	N	-	LIT	-

Highlighted in yellow – not part of the dossier