Asulam sodium

Annex B (Volume 3)

B.7 Residues

Version history

When	What
2016-04-18	Initial DAR

B.7 Residues data

B.7.1 Metabolism, distribution and expression of residues in plants (IIA 6.1, IIIA 8.1)

These studies have been evaluated at EU level and the conclusions presented are consistent with those in the EFSA report, with the exception of the new spinach metabolism studies (Roohi, 2008 and Piskorski, 2012)

B.7.1.1.1 Spinach (2003 study – Post emergence treatment PHI = 14 days)

A study was carried out in Germany in 2003. Phenyl ring labelled [^{14}C] asulam (radiochemical purity >99%) was applied (formulated as a soluble concentrate) to spinach grown in a greenhouse, as a foliar application, at a rate of 2.4 kg as/ha (N), at GS 14 (four true leaves – which is in-line with the proposed latest time of application of before GS 15). Samples were taken at maturity, 14 days after application. All the samples were analysed by LSC following combustion. The samples were also extracted with acetonitrile/water and acetonitrile. The resulting extracts were analysed by LSC, HPLC-UV, HPLC-MS, HPLC-MS/MS and FT/MS. The remaining unextractable material was combusted and analysed by LSC.

The total [^{14}C] residue at harvest in the spinach was (expressed as parent equivalent) 184 mg/kg.

The percentage recoveries of radiolabelled material are summarised in Table B.7.1. On characterisation of the extractable radioactivity (see Table B.7.2) one major component was identified in the spinach at harvest as parent asulam, which accounted for 77% (93% free and conjugated) of the total radioactivity in the spinach. Several other metabolites were identified plus several unknowns, which individually did not represent more than 5% (unknowns less than 1%) of the total radioactivity in the spinach at harvest for the foliar treatments. The remaining unextractable radioactivity accounted for less than 1% of the total radioactivity in the spinach.

(Sur 2004)

Table B.7.1 Partitioning of extractable radioactivity from spinach (in % of total radioactivity)

Plant part	Number of days after the first application	Label	Total residue (mg/kg parent equivalent)	Parent residues (mg/kg)	Extractable radioactivity (%)	Non-extractable radioactivity (%)
Spinach	14	Phenyl	184	143	99.5	0.5

Table B.7.2 Distribution of asulam and its metabolites in spinach in % of the total radioactivity (parent equivalent in mg/kg)

	Day 14
Asulam	77 (143)
Asulam-malonyl	9.0 (17)
Asulam-glucocside	6.4 (12)

Asulam-dimer1*	4.2 (7.7)
Asulam-dimer2*	1.6 (2.9)

*proposed by applicant as possible structure

In addition, two further studies were submitted on leafy crops, applying phenyl ring [^{14}C] asulam to spinach and alfalfa. In the case of spinach, on reading the summary, the results of the study were not considered to be valid, as the samples had been stored frozen for 6 months and a freezer stability study had indicated that residues of parent asulam had fallen by 50% over this period, therefore the above study was commissioned. However, the study did further clarify the metabolism pathway, with acetyl-asulam and acetyl-sulfanilamide being identified in the study at low levels (<0.1 mg/kg) and malonyl-asulam at high levels (50-72%). In the case of alfalfa the study was carried out in 1974, the summary indicated that a number of metabolites were isolated, but only parent could be reliably identified.

(Sur 2003a, and Maycey 1975a)

B.7.1.1.2 Spinach (2008 study – Pre-emergence Treatment)

Study was carried out in the UK in 2007. Phenyl ring labelled [^{14}C] asulam (radiochemical purity >99%) was applied (formulated as a soluble concentrate) pre-emergence to field grown spinach, at a rate of 2.4 kg as/ha (N). Samples were taken 20 (GS 45 – 50% maturity) and 41 (GS49 – Maturity) days after the application. All samples were analysed by LSC following combustion. The samples were also extracted sequentially with acetonitrile/water; hot acetonitrile/water, dilute HCl and dilute KOH. The resulting extracts were analysed by LSC, HPLC/UV and LC/MS/MS. The resulting unextractable material was further extracted by acid hydrolysis, followed by combustion and analysed by LSC.

The total [^{14}C] residues at harvest in the spinach were (expressed as parent equivalent) 0.062 mg/kg.

The percentage recoveries of radiolabelled material are summarised in Table B.7.2.1. On characterisation of the extractable radioactivity (see Table B.7.2.2) one major component was identified in the spinach at harvest as parent asulam (plus its conjugate malonyl-asulam), which accounted for 45% of the total radioactivity in the spinach at harvest. One other component was identified as desamino asulam, which was present at a level of less than 14% (<0.01 mg/kg) of the total radioactivity in the spinach. One unknown was also present representing 7.3% (<0.01 mg/kg) of the total radioactivity in the spinach. The remaining unextractable radioactivity accounted for less than 28% (0.02 mg/kg) of the total radioactivity in the spinach. On further extraction of the unextractable material, using acid hydrolysis, a further 6.8% (0.004 mg/kg) was extracted.

(Roohi, 2008)

Table B.7.2.1 Partitioning of extractable radioactivity from various plant parts (in % of total radioactivity)

Plant part	Number of days after last application	Label	Total residue (mg/kg parent equivalent)	Parent residues** (mg/kg)	Extractable radioactivity (%)	Extractable Radioactivity after acid hydrolysis (%)	Non-extractable radioactivity (%)
Spinach*	20 41	Phenyl	0.57 0.062	0.34 0.028	75 79	4.5 6.8	25 21

*Whole plant cut and samples at just above soil surface
**Parent plus its conjugate malonyl asulam

Table B.7.2.2 Distribution of asulam and its metabolites in spinach in % of the total radioactivity (parent equivalent in mg/kg)

	Sampled 20 days after the application	Sampled 41 days after the last application
	Spinach (Immature)	Spinach (Mature)
Asulam plus its conjugate malonyl asulam*	61 (0.34)	45 (0.028)
Asulam glucoside	0.2 (0.001)	-
Deasamino asulam	1.7 (0.01)	14 (0.008)

*HPLC system employed could not sufficiently separate the two components, however there presence was confirmed by LC/MS/MS

B.7.1.1.3 Spinach (2012 study – Post emergence – Longer PHI of up to 28 days in line with GAP)

Study was carried out in the Switzerland in 2011. Phenyl ring labelled asulam (radiochemical purity >99%) was applied (formulated as a soluble concentrate) to field grown spinach, as a foliar application at before GS 14 (4th true leaf unfolded), at a rate of 2.4 kg as/ha (N). Samples were taken 0, 7 (GS41/42 – 10/20% maturity), 14 (GS41 - 50% maturity), 21 (GS49 – maturity) and 28 (proposed PHI; GS53 30% stem elongation) days after application. All samples were analysed by LSC following combustion. The samples were also extracted sequentially with water; acetonitrile/water and acetonitrile. The resulting extracts were analysed by LSC, TLC; HPLC/UV and LC/MS/MS. The resulting unextractable material was further extracted by acid/base and enzyme (cellulose) hydrolysis, followed by combustion and analysed by LSC.

The total [^{14}C] residues at harvest (day 21) in the spinach were (expressed as parent equivalent) 1.4 mg/kg (by day 28 the crop had reached GS53 at which point the crop was no longer viable as a harvestable commodity).

The percentage recoveries of radiolabelled material are summarised in Table B.7.2.3. On characterisation of the extractable radioactivity (see Table B.7.2.4) one major component was identified in the spinach at harvest as parent asulam (plus its conjugate malonyl-asulam), which accounted for 77% of the total radioactivity in the spinach at harvest. One other component was identified as asulam glucoside, which was present at a level of less than 1.7% (0.02 mg/kg) of the total radioactivity in the spinach. Several unknown were also present representing individually representing up to 6.8% (<0.1 mg/kg) of the total radioactivity in the spinach. The remaining unextractable

radioactivity accounted for less than 8.4% (0.12 mg/kg) of the total radioactivity in the spinach. On further extraction of the unextractable material, using acid/base hydrolysis, a further 5.3% (0.08 mg/kg) was extracted , the remaining unextractable radioactivity was probably associated with fragmentation of the compound and the natural incorporation of these fragments into the plant tissue.

(Piskorski, 2012)

Table B.7.2.3 Partitioning of extractable radioactivity from various plant parts (in % of total radioactivity)

Plant part	Number of days after last application	Label	Total residue (mg/kg parent equivalent)	Parent residues (mg/kg)	Extractable radioactivity (%)	Extractable Radioactivity after acid/ base/enzyme hydrolysis (%)	Non-extractable radioactivity (%)
Leaves	0	Phenyl	180	172	99.8	-	0.2
	7		19	4	92	5.3	5.6
	14		5.4	1.5	94.3	3.4	3.9
	21		1.4	0.31	85.3	5.3	3.1
	28		0.22	-	79	6.9	4.9

Table B.7.2.4 Distribution of asulam and its metabolites in spinach in % of the total radioactivity (parent equivalent in mg/kg)

	Sampled 0 days after the application	Sampled 7 days after the last application	Sampled 14 days after the application	Sampled 21 days after the last application
	Spinach (Immature)	Spinach (Immature)	Spinach (Immature)	Spinach (Mature)
Asulam	96 (172)	21 (4)	27 (1.5)	22 (0.31)
Malonyl asulam	0.8 (1.5)	36 (6.7)	51 (2.8)	55 (0.78)
Asulam glucoside	0.6 (1.1)	26 (5)	12 (0.64)	1.7 (0.02)

*Day 28 sample could not be successfully chromatographed - crop had reached GS53 at which point the crop was no longer viable as a harvestable commodity

B.7.1.2 Grass (Rye)

A study was carried out in UK in 1974. Phenyl ring labelled [^{14}C] asulam was applied to rye grass grown in a greenhouse, as a foliar application, at a rate of 1.12 kg as/ha. Samples were taken 0, 2, 7, 14 and 28 days after application. All the samples were analysed by LSC following combustion. The samples were also extracted with acetone and water. The resulting extracts were analysed by LSC and TLC. The remaining unextractable material was further extracted by acid hydrolysis followed by chlorite digestion (day 7 only), before being combusted and analysed by LSC.

The total [^{14}C] residues at harvest in the rye grass (expressed as parent equivalent) declined from 63 mg/kg on day 0 to 10 mg/kg on day 28.

The percentage recoveries of radiolabelled material are summarised in Table B.7.3. On characterisation (7 day sample only) of the extractable radioactivity (see Table B.7.4) one major component was identified in the grass at harvest as parent asulam, which accounted for 11% of the total radioactivity in the grass. One other metabolite

was identified (acetyl-asulam - 6.3%) plus numerous tentatively identified/unknown metabolites, which individually did not represent more than 3% (unknowns less than 1%) of the total radioactivity in the grass at harvest. The remaining unextractable radioactivity accounted for 6% (chlorate digestion released 20%) of the total radioactivity in the grass and was probably associated with the fragmentation of the compound and the natural incorporation of these fragments into the plant tissue (lignin), which is supported by the tentative identification of natural plant products (salicylic acid, quinic acid, etc) containing ^{14}C.

(Maycey 1975b)

Table B.7.3 Partitioning of extractable radioactivity from grass (in % of total radioactivity)

Plant part	Number of days after the first application	Label	Total residue (mg/kg parent equivalent)	Parent residues (mg/kg)	Extractable radioactivity (%)	Acid extractable	Non-extractable radioactivity (%)
Grass	0	Phenyl	63	-	91	3	6
	2		37	-	75	7	18
	7		32	3.4	66	7	27
	14		22	-	61	11	27
	28		10	-	51	9	41

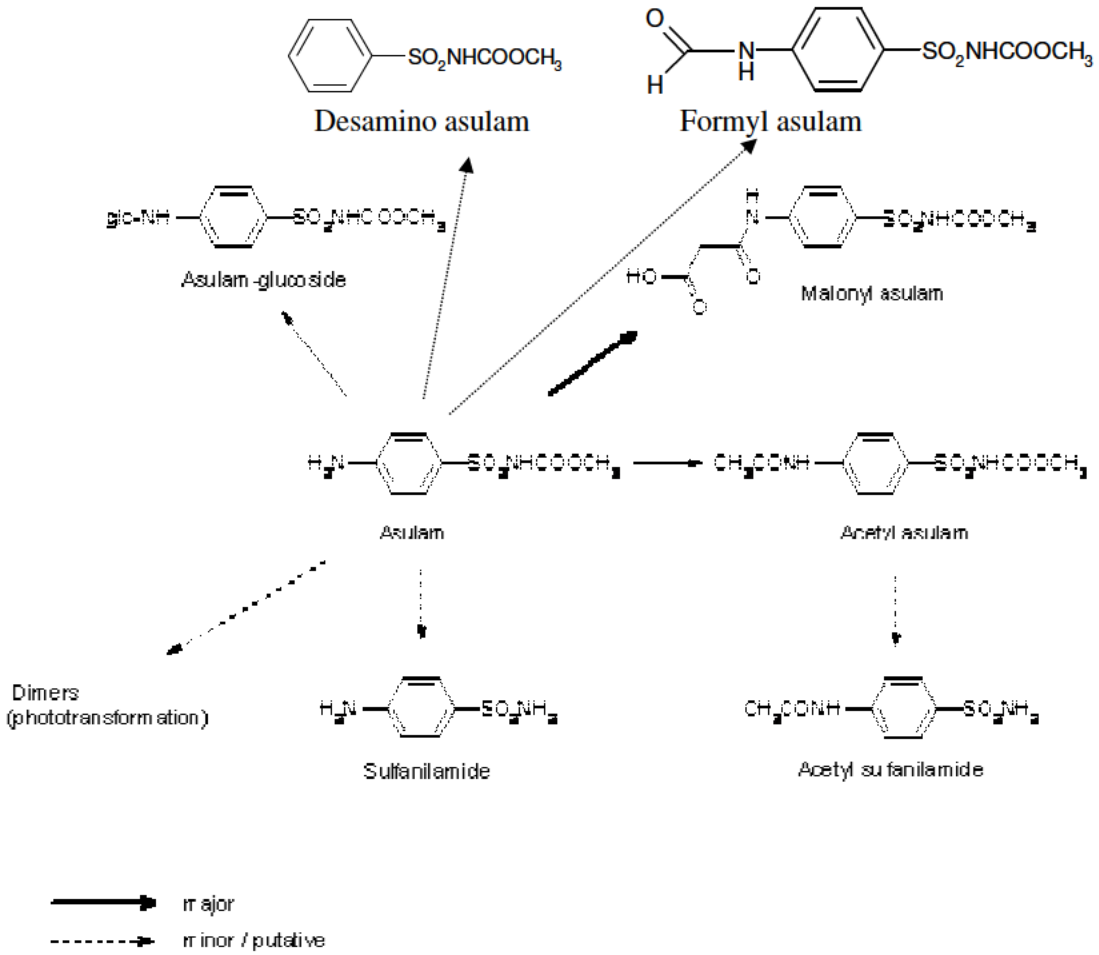
Table B.7.4 Distribution of asulam and its metabolites in grass in % of the total radioactivity (parent equivalent in mg/kg)

	Day 7
Asulam	11 (3.4)
Acetyl-asulam	6.5 (2.1)

In addition, one further study was submitted on sugar cane (1971), however on reading the summary it contained no additional data to that presented in the above report.

(Mulcock, 1971a)

Figure B.7.1 Proposed Metabolic Pathway of Asulam in Plants



B.7.1.3 Metabolism, distribution and expression of the residue in rotational crops

Field trials were conducted (under a polyethylene canopy) in 1990 in the USA, growing wheat, spinach and radish in soil treated (bare ground application) with phenyl ring labelled [^{14}C] asulam (formulated as a soluble concentrate; radiochemical purity >96%), at a rate of 3.74 kg as/ha (1.6N). The crops were planted 28, 118 (soil from the 29 day study was used for radish and spinach) and 365 (soil from the 29 day study was used for wheat and from the 118 day study for radish and spinach) days after application. All samples were analysed by LSC, following combustion. Samples were also sequentially extracted with acidified water, acetone/water, ethyl acetate/methanol. The resulting extracts were analysed by LSC, TLC and HPLC-UV. The remaining unextractable material was further extracted by acid and base hydrolysis, before being combusted and analysed by LSC.

The total [^{14}C] residues in the mature crops at harvest (expressed as parent equivalent) were less than 0.07 mg/kg, with the exception of wheat straw which gave residues of

0.47 mg/kg in the 28 day study falling to 0.39 mg/kg in the 365 day studies and radish root 0.18 mg/kg in the 28 day study falling to 0.03 mg/kg in the 365 day study.

The percentage recoveries of radiolabelled material are summarised in Table B.7.5. On characterisation of the extractable radioactivity (see Tables B.7.6) several components were identified in the crops at harvest in the 29 day study as parent asulam and its metabolites acetyl asulam, sulphanilamide, 4-acetyl benzene sulphonamide and Sulfanilic acid. Individually the components did not account for more than 10% (0.04 mg/kg) of the radioactivity in the crops at harvest, with the majority of the extractable radioactivity remaining uncharacterised. It was surmised that the remaining uncharacterised radioactivity was associated with the fragmentation (soil and plant) of the compound and the natural incorporation of these fragments into the plant tissue. In addition, the total [^{14}C] residues in the crops at harvest was low (less than 0.2 mg/kg), with the exception of wheat straw (0.6 mg/kg) which is not directly consumed by humans. The remaining unextractable radioactivity accounted for less than 12% (0.02 mg/kg) of the total radioactivity in the crops.

(Jesudason, 1996)

Table B.7.5 Partitioning of extractable radioactivity from various crops (in % of total radioactivity)

Crop and days after treatment	Number of days after application	Label	Total residue (mg/kg parent equivalent)*	Parent residues (mg/kg)	Extractable radioactivity (%)	Released by acid hydrolysis (%)	Released by base hydrolysis (%)	Non-extractable radioactivity (%)
Spinach		Phenyl						
28 days			0.06	-	57	22	11	<1
118 days*			0.05	-	67	58	30	<1
365 days			0.03	-	65	24	<1	<1
Wheat		Phenyl						
28 days								
Forage*			0.07	<0.01	87	28	9	<1
Grain			0.04	-	55	47	<1	<1
Straw			0.47	<0.01	87	14	4	4
128 days								
Forage*			0.05	-	119	30	16	<1
Grain			0.04	-	36	60	<1	<1
Straw*			0.59	-	18	3	1	7
365 days								
Forage			0.09	-	63	22	13	<1
Grain			0.02	-	22	46	<1	<1
Straw*			0.39	<0.01	46	13	4	3
Radish		Phenyl						
28 days								
Tops			0.12	<0.01	66	31	14	8
Roots			0.18	<0.01	39	32	16	11
118 days								
Tops*			0.05	-	117	44	20	<1
Roots			0.19	-	-	-	-	-
365 days								
Tops			0.03	-	77	20	<1	<1
Roots			0.03	-	49	19	<1	<1

*The extraction efficiencies were significantly greater or less than 100%, the applicant stated that this was possibly due to the homgeniety of the sample

Table B.7.6 Distribution of asulam and its metabolites in rotational crops in % of the total radioactivity (parent equivalent in mg/kg)

	Wheat forage	Wheat straw	Radish tops	Radish roots
Asulam				
28 day study	-	-	-	3 (<0.01)
365 day study	8 (<0.01)	-	-	-
Acetyl asulam				
28 day study	-	-	-	3 (<0.01)
365 day study	-	-	-	-
Sulfanilamide				
28 day study	-	-	2 (<0.01)	5 (<0.01)
365 day study	5 (<0.01)	-	-	-
4-acetyl benzene sulphonamide				
28 day study	24 (0.02)	-	10 (0.01)	1 (<0.01)
365 day study	6 (<0.01)	-	-	-
Sulfanilic acid				
28 day study	-	8 (0.04)	5 (<0.01)	1 (<0.01)
365 day study	3 (<0.01)	-	-	-

B.7.1.4 Summary/assessment

The metabolism of asulam was investigated in spinach and rye grass, by applying phenyl ring labelled [^{14}C] asulam as a foliar application, at rate of 2.4 kg as/ha (N) to spinach (both pre and post emergence) and 2 x 1.12 kg as/ha rye grass. At harvest the total [^{14}C] residues (expressed as parent equivalent) in spinach were 184 mg/kg and in rye grass 32 mg/kg (day 7), in the glasshouse studies and in the field spinach studies 0.062 mg/kg (day 41) in the pre-emergence study and 1.4 mg/kg (day 21) in the post emergence study.

On characterisation of the extractable radioactivity one major component was identified in the spinach and rye at harvest as parent asulam (plus its conjugate malonyl asulam).
For the glasshouse studies, parent asulam (plus its conjugate malonyl asulam) accounted for 86% of the total radioactivity in the spinach and 11% of the total radioactivity in the rye grass. Several other metabolites were identified (tentatively in the case of rye grass) plus several unknowns, which individually did not represent more than 5% (unknowns less than 1%) of the total radioactivity in the spinach at harvest for the foliar treatments. The remaining unextractable radioactivity accounted for less than 7% of the total radioactivity in the crops.
For the pre-emergence field study, parent asulam (plus its conjugate malonyl-asulam) accounted for 45% of the total radioactivity in the spinach at harvest. One other

component was identified as desamino asulam, which was present at a level of less than 14% (<0.01 mg/kg) of the total radioactivity in the spinach. One unknown was also present representing 7.3% (<0.01 mg/kg) of the total radioactivity in the spinach. The remaining unextractable radioactivity accounted for less than 28% (0.02 mg/kg) of the total radioactivity in the spinach. On further extraction of the unextractable material, using acid hydrolysis, a further 6.8% (0.004 mg/kg) was extracted.
For the post emergence field study, parent asulam (plus its conjugate malonyl-asulam) accounted for 77% of the total radioactivity in the spinach at harvest. One other component was identified as asulam glucoside, which was present at a level of less than 1.7% (0.02 mg/kg) of the total radioactivity in the spinach. Several unknown were also present representing individually representing up to 6.8% (<0.1 mg/kg) of the total radioactivity in the spinach. The remaining unextractable radioactivity accounted for less than 8.4% (0.12 mg/kg) of the total radioactivity in the spinach. On further extraction of the unextractable material, using acid/base hydrolysis, a further 5.3% (0.08 mg/kg) was extracted , the remaining unextractable radioactivity was probably associated with fragmentation of the compound and the natural incorporation of these fragments into the plant tissue.

For the proposed lead use on spinach, appropriate plant metabolism data are available to support this use and potential uses on other leafy crops, however the main uses of asulam are on grassland and fruit crops. In the case of grassland, a plant metabolism study on rye grass is available, however the study was carried out in 1974, the characterisation work is tentative at best and there may be an issue with the stability of asulam during freezer storage of the samples (unclear how long the samples were stored frozen – issue with spinach see Section B.7.7). In the case of fruit crops (e.g. strawberries) no plant metabolism data were available. Therefore, it is recommended that if approval is to be sought on grassland and fruit crops, at least two further metabolism studies are required on grass and a fruit crop (e.g. strawberries), applying phenyl ring labelled [^{14}C] asulam at the required GAP, storing the samples for the minimum period possible.

Based on the plant metabolism data submitted for spinach, residues in leafy crops should be defined as the sum of parent asulam plus its metabolite asulam-malonyl expressed as asulam.

The metabolism and distribution in rotational crops was investigated in wheat, spinach and radish. The crops were grown in soil treated (bare ground application) with phenyl and ring labelled [^{14}C] asulam at a rate of 3.74 kg as/ha (1.6N). At harvest total [^{14}C] residues (expressed as parent equivalent) were less than 0.07 mg/kg, with the exception of wheat straw which gave residues of 0.47 mg/kg in the 28 day study falling to 0.39 mg/kg in the 365 day studies and radish root 0.18 mg/kg in the 28 day study falling to 0.03 mg/kg in the 365 day study.

On characterisation of the extractable radioactivity several components were identified in the crops at harvest in the 29 day study as parent asulam and its metabolites acetyl asulam, sulphanilamide, 4-acetyl benzene sulphonamide and sulfanilic acid. Individually the components did not account for more than 10% (0.04 mg/kg) of the radioactivity in the crops at harvest, with the majority of the extractable radioactivity remaining uncharacterised. It was surmised that the remaining uncharacterised

radioactivity was associated with the fragmentation (soil and plant) of the compound and the natural incorporation of these fragments into the plant tissue. In addition, the total [^{14}C] residues in the crops at harvest was low (less than 0.2 mg/kg), with the exception of wheat straw (0.6 mg/kg) which is not directly consumed by humans. The remaining unextractable radioactivity accounted for less than 12% (0.02 mg/kg) of the total radioactivity in the crops.

For the proposed lead use on spinach, the rotational crop metabolism data is sufficient to cover the use on spinach, however the main use of asulam is on grassland, at an increased rate of 4.4 kg as/ha. Therefore, consideration should be given to the generation of a new rotation crop metabolism study, applying phenyl ring labelled [^{14}C] asulam at the required GAP, storing the samples for the minimum period possible and the characterisation of a greater amount of the extractable radioactivity.

The majority of plant metabolites identified in the plant and rotational crop metabolism studies were also identified in the rat metabolism studies, however the glucoside conjugate (malonyl included in the residues definition) of asulam and the asulam dimers were not, but were not considered to be of toxicological concern (see Section B.6.1.4). In the case of desamino asulam, 4-acetyl benzene sulphonamide and sulfanilic acid, residue levels in crops consumed by humans were less than 0.02 mg/kg (less than 0.05 mg/kg in fodder crops).

B.7.2 Metabolism, distribution and expression of the residues in livestock (AII 6.2, IIIA 8.1)

These studies have been evaluated at EU level and the conclusions presented are consistent with those in the EFSA report

B.7.2.1 Goats

The studies were carried out in 1989. Two lactating goats each received seven daily doses of phenyl ring labelled [^{14}C] asulam (radiochemical purity >96%) dosed at rates of 20 mg/kg in feed. The goats were sacrificed 23 hours after the last dose. All samples were analysed by LSC following combustion. The samples were extracted with acidified water (milk), or acidified acetone (liver and kidney) and analysed by LSC, TLC and HPLC-UV. The unextractable material was further extracted by enzyme (glucanase, protease and cellyase) hydrolysis, before being combusted and analysed by LSC.

Overall recovery of radioactivity was 89-102%, the bulk of the radioactivity was excreted (89-102%), with less than 0.1% in the milk and less than 0.1% in the tissues.

Total [^{14}C] residues in the milk (expressed as parent equivalent) reach a plateau after 2 days of 0.01-0.02 mg/kg.

The percentage recoveries of radiolabelled material are summarised in Table B.7.7. On characterisation of the extractable radioactivity (see Table B.7.8) one major component was identified in the milk as parent asulam which accounted for 85% of the

total radioactivity in the milk. The remaining unextractable radioactivity, accounted for 15% (<0.01 mg/kg) of the total radioactivity in the milk.

Total [^{14}C] residues in the tissues (expressed as parent equivalent) were less than 0.01 mg/kg for muscle and fat (samples were not characterised due to the low levels of radioactivity in the samples), 0.17-0.69 mg/kg for kidney (the 0.69 mg/kg level was thought to be due to residual urine in the kidney at sacrifice - urine was the main route of elimination of radiocarbon and the goat in question consistently produced low amounts of urine compared to the other goats in the study) and 0.06-0.09 mg/kg for liver.

The percentage recoveries of radiolabelled material are summarised in Table B.7.7. On characterisation of the extractable radioactivity (see Table B.7.8) one major component was identified in the kidney as parent asulam, which accounted for 95% of the total radioactivity in the kidney. The remaining unextractable radioactivity, accounted for 5% (<0.01 mg/kg) of the total radioactivity in the kidney. For liver, one major component was identified in the liver as acetyl sulphanilamide, which accounted for 64% of the total radioactivity in the liver. One unknown was isolated, which accounted for 26% (0.02 mg/kg) of the total radioactivity in the liver. The remaining unextractable radioactivity accounted for less than 10% (<0.01 mg/kg) of the total radioactivity in the liver.

([redacted], 1990b)

Table B.7.7 Partitioning of extractable radioactivity in milk and tissues (in % of total radioactivity)

Animal Product and number of days after treatment	Number of doses	Label	Total residue (mg/kg parent equivalent)	Parent residues (mg/kg)	Extractable Radioactivity (%)	Non-extractable radioactivity (%)
Milk		Phenyl				
Day 1	1		0.01	-	-	-
Day 2	2		0.02	-	-	-
Day 3	3		0.02	-	-	-
Day 4	4		0.02	-	-	-
Day 5	5		0.02	-	-	-
Day 6	6		0.02	0.02	81	14
Day 7	7		0.02	-	-	-
Liver	7	Phenyl	0.08	-	81	9
Muscle	7	Phenyl	<0.01	-	-	-
Fat	7	Phenyl	<0.01	-	-	-
Kidney	7	Phenyl	0.16	0.18	109	6

Table B.7.8 Distribution of asulam and its metabolites in animal products in % of the total radioactivity (parent equivalent in mg/kg)

	Milk (day 6)	Kidney	Liver
Asulam	85 (0.02)	95 (0.18)	-
Acetyl sulphanilamide	-	-	64 (0.05)

B.7.2.2 Poultry

The studies were carried out in 1999. Twenty laying hens each received seven daily doses of phenyl ring labelled [^{14}C] asulam (radiochemical purity >96%) dosed at a rate of 23 mg/kg in feed. The hens were sacrificed 9 hours after the last dose. All samples were analysed by LSC following combustion. The samples were extracted with acidified acetone (liver, kidney and muscle) and ethyl acetate/alkaline ethyl acetate (egg white and yolk) and methanol/acetonitrile (egg) and analysed by LSC, TLC and HPLC-UV. The unextractable material was combusted and analysed by LSC.

Overall recovery of radioactivity was 86%, the bulk of the radioactivity was excreted (85-89%), with less than 0.1% in the eggs and less than 0.1% in the tissues.

Total [^{14}C] residues in the egg yolk and white (expressed as parent equivalent) appeared to have reached a plateau after 7 days of 0.03 and 0.05 mg/kg respectively.

The percentage recoveries of radiolabelled material are summarised in Table B.7.9. On characterisation of the extractable radioactivity (see Table B.7.10) two major components were identified in the eggs as parent asulam and its metabolite acetyl sulfanilamide, which accounted for more than 90% of the total radioactivity in the egg yolk and white. The remaining unextractable radioactivity, accounted for less than 0.01 mg/kg in the egg yolk and white.

Total [^{14}C] residues in the tissues (expressed as parent equivalent) were 0.05-0.07 mg/kg for muscle; <0.01-0.01 mg/kg for fat (sample was not characterised due to the low levels of radioactivity in the sample), 0.28-0.44 mg/kg for kidney and 0.08-0.14 mg/kg for liver.

The percentage recoveries of radiolabelled material are summarised in Table B.7.9. On characterisation of the extractable radioactivity (see Table B.7.10) two major components were identified in the tissues as parent asulam and its metabolite acetyl sulphanilamide, which accounted for more than 80% of the radioactivity in the tissues. One unknown was isolated, which did not exceed 0.04 mg/kg in the tissues. The remaining unextractable radioactivity accounted for less than 0.04 mg/kg in the tissues.

([redacted], 1990a)

Table B.7.9 Partitioning of extractable radioactivity in eggs and tissues (in % of total radioactivity)

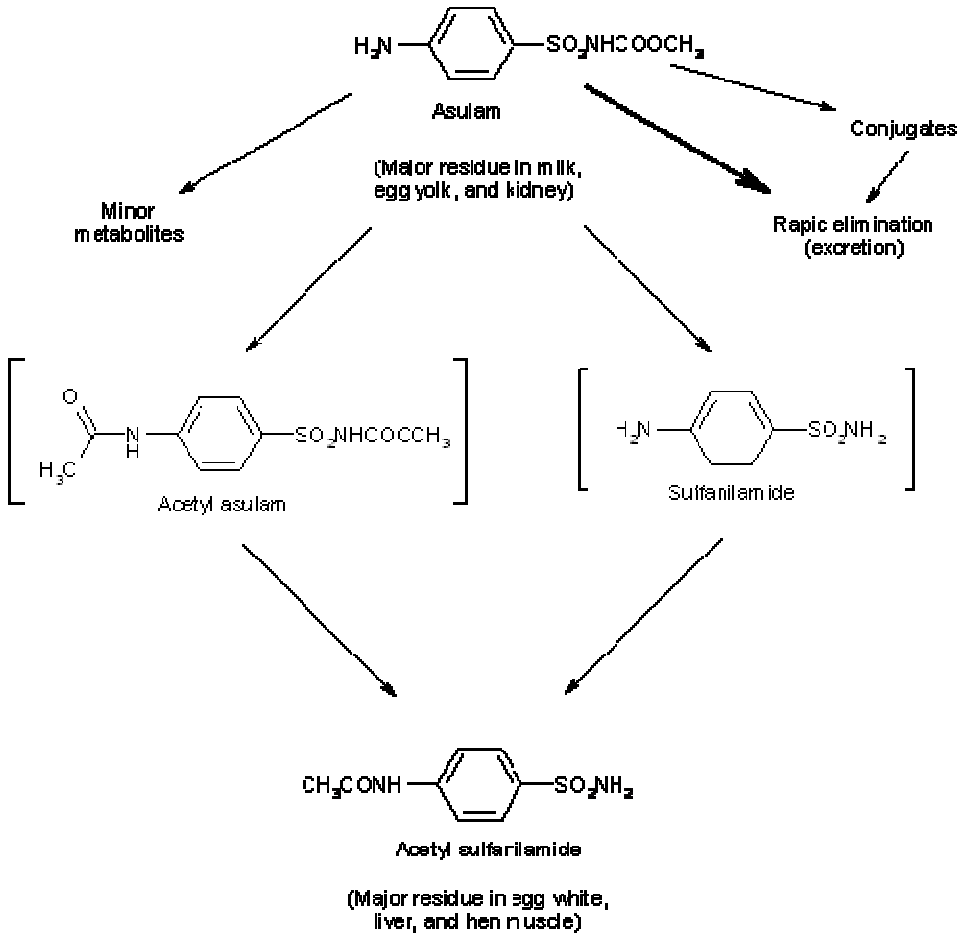
Animal Product and number of days after treatment	Number of doses	Label	Total residue (mg/kg parent equivalent)	Parent residues (mg/kg)	Extractable Radioactivity (%)	Non-extractable radioactivity (%)
Egg yolk		Phenyl				
Day 1	1		<0.01	-	-	-
Day 2	2		<0.01	-	-	-
Day 3	3		<0.01	-	-	-
Day 4	4		0.01	-	-	-
Day 5	5		0.01	-	-	-
Day 6	6		0.02	0.02	113*	29*
Day 7	7		0.03	-	-	-
Egg White						
Day 1	1		<0.01	-	-	-
Day 2	2		0.03	-	-	-
Day 3	3		0.04	-	-	-
Day 4	4		0.03	-	-	-
Day 5	5		0.04	-	-	-
Day 6	6		0.05	0.01	98	7
Day 7	7		0.05	-	-	-
Liver	14	Phenyl	0.09	-	104	16
Muscle	14	Phenyl	0.07	0.03	91	4
Fat	14	Phenyl	<0.01	-	-	-
Kidney	14	Phenyl	0.44	0.37	98	7

*high recovery was possibly due to the residue levels in the sample being low (<0.03 mg/kg)

Table B.7.10 Distribution of asulam and its metabolites in animal products in % of the total radioactivity (parent equivalent in mg/kg)

	Egg yolk (Day 6)	Egg white (Day 6)	Liver	Kidney	Muscle
Asulam	63 (0.02)	26 (0.01)	-	82 (0.37)	35 (0.03)
Acetyl sulfanilamide	44 (0.01)	66 (0.03)	81 (0.07)	14 (0.06)	51 (0.04)

Figure B.7.2: Proposed metabolic pathway of asulam in domestic animals



[ ] Not isolated

B.7.2.3 Summary/assessment

The metabolism and distribution in animals was investigated in lactating goats and chickens, using phenyl ring labelled [^{14}C] asulam. For lactating goats dosed at rates of 20 mg/kg in feed, overall recovery of radioactivity was 89-102%, the bulk of the radioactivity was excreted (89-102%), with less than 0.1% in the milk and less than 0.1% in the tissues.

On characterisation of the extractable radioactivity one major component was identified in the milk as parent asulam which accounted for 85% of the total radioactivity in the milk. The remaining unextractable radioactivity, accounted for 15% (<0.01 mg/kg) of the total radioactivity in the milk.

On characterisation of the extractable radioactivity one major component was identified in the kidney as parent asulam, which accounted for 95% of the total radioactivity in the kidney. The remaining unextractable radioactivity, accounted for 5% (<0.01 mg/kg) of the total radioactivity in the kidney. For liver, one major component was identified in the liver as acetyl sulphanilamide, which accounted for

64% of the total radioactivity in the liver. One unknown was isolated, which accounted for 26% (0.02 mg/kg) of the total radioactivity in the liver. The remaining unextractable radioactivity accounted for less than 10% (<0.01 mg/kg) of the total radioactivity in the liver.

For chickens dosed at a rate of 23 mg/kg in feed overall recovery of radioactivity was 86%, the bulk of which was excreted (86%), with less than 0.1% in the eggs and less than 0.1% in the tissues.
On characterisation of the extractable radioactivity two major components were identified in the eggs as parent asulam and its metabolite acetyl sulfanilamide, which accounted for more than 90% of the total radioactivity in the egg yolk and white. The remaining unextractable radioactivity, accounted for less than 0.01 mg/kg in the egg yolk and white.
On characterisation of the extractable radioactivity two major components were identified in the tissues as parent asulam and its metabolite acetyl sulphanilamide, which accounted for more than 80% of the radioactivity in the tissues. One unknown was isolated, which did not exceed 0.04 mg/kg in the tissues. The remaining unextractable radioactivity accounted for less than 0.04 mg/kg in the tissues.

A proposed metabolic pathway was submitted for asulam in domestic animals (see Figure B.7.2).

The animal metabolite (edible animal products) identified in the animal metabolism study was also identified in the rat metabolism study.

Based on the metabolism data submitted for domestic animals, residues in products of ruminant origin should be defined as the sum of parent asulam plus its metabolite acetyl sulphanilamide expressed as asulam.

B.7.3 Definition of the residue (IIA 6.7, IIIA 8.6)

Based on the metabolism data submitted for spinach, residues should be defined as follows:

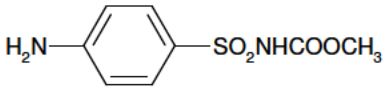
Leafy crops (MRL and monitoring):	Asulam plus its metabolite asulam-malonyl expressed as asulam.
Leafy crops (Risk Assessment):	Asulam plus its metabolite asulam-malonyl expressed as asulam.

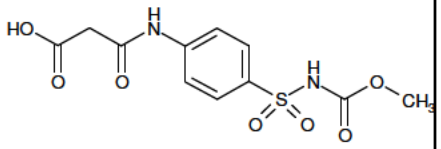
Based on the metabolism data submitted for goats and hens, residues should be defined as follows:

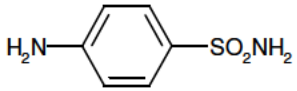
Animal products (MRL and monitoring):	Asulam plus its metabolite acetyl sulphanilamide expressed as asulam.
Animal products (Risk Assessment):	Asulam plus its metabolite acetyl sulphanilamide expressed as asulam.

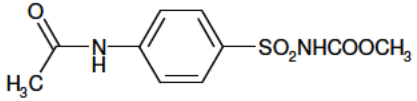
See Table B.7.10.1 for structures of plant and animal metabolites.

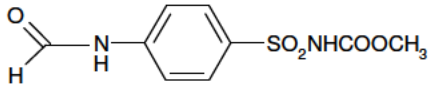
Table B.7.10.1 The occurrence of asulam and metabolites in crops at harvest and products of animal origin

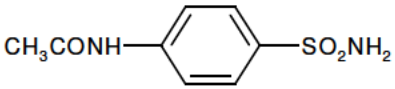
Component	Structure	Occurrence (max % TRR)	Level (mg/kg)	PHI	Comment
Asulam		**Spinach field studies (N)**			Proposed for inclusion in the residue definition for monitoring and risk assessment for plants and products of animal origin *Asulam plus its conjugate malonyl asulam
		Pre-emergence (45%)*	0.03*	41	
		Post emergence (22%)	0.31	21	
		Rye Grass (1.12 kg as/ha)			
		Grass (11%)	3.4	7	
		Rotational crops (1.6N)			
		28 day			
		Wheat grain (-)			
		Wheat straw (-)			
		Spinach (-)			
		Radish root (3%)	<0.01		
		Radish tops (-)			
		Goat (20 mg/kg in feed)			
		Goat kidney (95%)	0.18		
		Goat liver (-)			
		Goat milk (85%)	0.02		
		Hen (23 mg/kg in feed)			
		Poultry muscle (35%)	0.03		
		Poultry kidney (82%)	0.37		
		Poultry liver (-)			
		Egg yolk (63%)	0.02		
		Egg white (26%)	0.01		

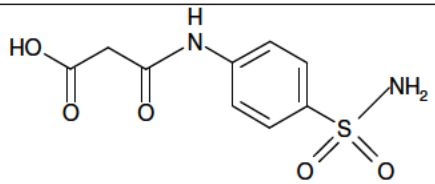
Component	Structure	Occurrence (max % TRR)	Level (mg/kg)	PHI	Comment
Malonyl-asulam		**Spinach field studies (N)** Pre-emergence (45%)* Post emergence (55%) **Rye Grass (1.12 kg as/ha)** Grass (-) **Rotational crops (1.6N)** <u>28 day</u> Wheat grain (-) Wheat straw (-) Spinach (-) Radish root (-) Radish tops (-) **Goat** Goat kidney (-) Goat liver (-) Goat milk (-) **Hen** Poultry muscle (-) Poultry kidney (-) Poultry liver (-) Egg yolk (-) Egg white (-)	0.03* 0.78	41 21	Proposed for inclusion in the residue definition for monitoring and risk assessment for plants *Asulam plus its conjugate malonyl asulam

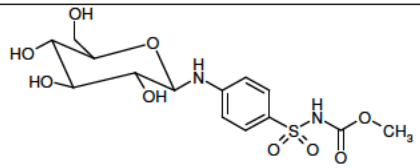
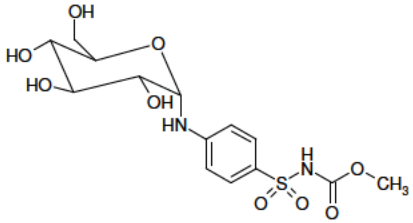
Component	Structure	Occurrence (max % TRR)	Level (mg/kg)	PHI	Comment
Sulphanilamide		**Spinach field studies (N)** Pre-emergence (-) Post emergence (-) **Rye Grass (1.12 kg as/ha)** Grass (-) **Rotational crops (1.6N)** 28 day Wheat grain (-) Wheat straw (-) Spinach (-) Radish root (5%) Radish tops (2%) **Goat** Goat kidney (-) Goat liver (-) Goat milk (-) **Hen** Poultry muscle (-) Poultry kidney (-) Poultry liver (-) Egg yolk (-) Egg white (-)	

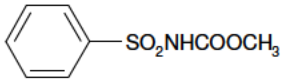
Component	Structure	Occurrence (max % TRR)	Level (mg/kg)	PHI	Comment
Acetyl asulam		**Spinach field studies (N)** Pre-emergence (-) Post emergence (-) **Rye Grass (1.12 kg as/ha)** Grass (6.5%) **Rotational crops (1.6N)** <u>28 day</u> Wheat grain (-) Wheat straw (-) Spinach (-) Radish root (3%) Radish tops (-) **Goat** Goat kidney (-) Goat liver (-) Goat milk (-) **Hen** Poultry muscle (-) Poultry kidney (-) Poultry liver (-) Egg yolk (-) Egg white (-)	 2.1

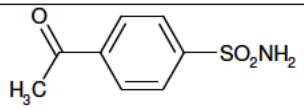
Component	Structure	Occurrence (max % TRR)	Level (mg/kg)	PHI	Comment
Formyl asulam		**Spinach field studies (N)** Pre-emergence (-) Post emergence (-) **Rye Grass (1.12 kg as/ha)** Grass (-) **Rotational crops (1.6N)** <u>28 day</u> Wheat grain (-) Wheat straw (-) Spinach (-) Radish root (-) Radish tops (-) **Goat** Goat kidney (-) Goat liver (-) Goat milk (-) **Hen** Poultry muscle (-) Poultry kidney (-) Poultry liver (-) Egg yolk (-) Egg white (-)			

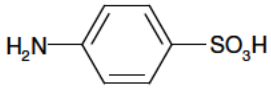
Component	Structure	Occurrence (max % TRR)	Level (mg/kg)	PHI	Comment
Acetyl sulphanilamide		**Spinach field studies (N)** Pre-emergence (-) Post emergence (-) **Rye Grass (1.12 kg as/ha)** Grass (-) **Rotational crops (1.6N)** 28 day Wheat grain (-) Wheat straw (-) Spinach (-) Radish root (-) Radish tops (-) **Goat** Goat kidney (-) Goat liver (64%) Goat milk (-) **Hen** Poultry muscle (51%) Poultry kidney (14%) Poultry liver (81%) Egg yolk (44%) Egg white (66%)	 0.05 0.04 0.06 0.07 0.01 0.03		

Component	Structure	Occurrence (max % TRR)	Level (mg/kg)	PHI	Comment
Malonyl-sulphanilamide		**Spinach field studies (N)** Pre-emergence (-) Post emergence (-) **Rye Grass (1.12 kg as/ha)** Grass (-) **Rotational crops (1.6N)** <u>28 day</u> Wheat grain (-) Wheat straw (-) Spinach (-) Radish root (-) Radish tops (-) **Goat** Goat kidney (-) Goat liver (-) Goat milk (-) **Hen** Poultry muscle (-) Poultry kidney (-) Poultry liver (-) Egg yolk (-) Egg white (-)			

Component	Structure	Occurrence (max % TRR)	Level (mg/kg)	PHI	Comment
Asulam glucoside	 	**Spinach field studies (N)** Pre-emergence (-) Post emergence (1.7%) **Rye Grass (1.12 kg as/ha)** Grass (-) **Rotational crops (1.6N)** <u>28 day</u> Wheat grain (-) Wheat straw (-) Spinach (-) Radish root (-) Radish tops (-) **Goat** Goat kidney (-) Goat liver (-) Goat milk (-) **Hen** Poultry muscle (-) Poultry kidney (-) Poultry liver (-) Egg yolk (-) Egg white (-)	0.02	21	

Component	Structure	Occurrence (max % TRR)	Level (mg/kg)	PHI	Comment
Desamino asulam		**Spinach field studies (N)** Pre-emergence (14%) Post emergence (-) **Rye Grass (1.12 kg as/ha)** Grass (-) **Rotational crops (1.6N)** 28 day Wheat grain (-) Wheat straw (-) Spinach (-) Radish root (-) Radish tops (-) **Goat** Goat kidney (-) Goat liver (-) Goat milk (-) **Hen** Poultry muscle (-) Poultry kidney (-) Poultry liver (-) Egg yolk (-) Egg white (-)	0.008	41	

Component	Structure	Occurrence (max % TRR)	Level (mg/kg)	PHI	Comment
4-Acetylbenzene-sulphonamide		**Spinach field studies (N)** Pre-emergence (-) Post emergence (-) **Rye Grass (1.12 kg as/ha)** Grass (-) **Rotational crops (1.6N)** 28 day Wheat grain (%) Wheat straw (%) Spinach (%) Radish root (1%) Radish tops (10%) **Goat** Goat kidney (-) Goat liver (-) Goat milk (-) **Hen** Poultry muscle (-) Poultry kidney (-) Poultry liver (-) Egg yolk (-) Egg white (-)	

Component	Structure	Occurrence (max % TRR)	Level (mg/kg)	PHI	Comment
Sulfanilic acid		**Spinach field studies (N)** Pre-emergence (-) Post emergence (-) **Rye Grass (1.12 kg as/ha)** Grass (-) **Rotational crops (1.6N)** 28 day Wheat grain (-) Wheat straw (8%) Spinach (-) Radish root (1%) Radish tops (5%) **Goat** Goat kidney (-) Goat liver (-) Goat milk (-) **Hen** Poultry muscle (-) Poultry kidney (-) Poultry liver (-) Egg yolk (-) Egg white (-)	 0.04 <0.01 <0.01		

B.7.4 Use pattern

Full details of registered Good Agricultural Practice are given in Volume 1 Table 1.5.1.

B.7.5 Identification of critical GAPs

Table B.7.23 Summary of critical GAP

Crop	Rate of application (kg as/ha)	Number of treatments	Last time of application	PHI (days)
Spinach-outdoor (NMS)	2.4	1	GS14 (4th true leaf unfolded)	28
			OR	
			Pre-emergence	-

B.7.6 Residues arising from supervised trials (IIA 6.3; IIIA 8.2)

These study have been evaluated at EU level and the conclusions presented are consistent with those in the EFSA report, with the exception of the new method of analysis (Chambers, 2007) and new residue trials data (Boileau, 2013 and Sandt, 2008/9/13)

Unless specified, the trials were reported in sufficient detail. The basic criteria for acceptability are listed below:

Trials details
Crop/variety
Location/position/year
Formulation type
Application/dilution rate
Maximum number of treatments
Growth stage of crop at treatment
PHI
Residue level (control treated)
Geo-climatic information

Analytical aspects
Method specified and submitted (see Section B.5.2 and [a] below and summary Table B.7.16.0)
Residues in control plots were below the limit of determination (0.1 mg/kg 2003/4 trials and 0.02 mg/kg 2008/9/13 trials – asulam plus its metabolite malonyl asulam [and all the other determined metabolites])
Storage of samples prior to analysis (samples were stored frozen for up to 4 months {only one sample stored for longer than 3 months – Spanish sample])
Limit of determination at acceptable level (0.1 mg/kg 2003/4 trials and 0.02 mg/kg 2008/9/13 trials – asulam plus its metabolite malonyl asulam)
Acceptable recovery (usually 70 - 110%), with the exception of -

two recoveries of 64 and 67% at a fortification level of 0.01 mg/kg malonyl asulam (Sandt, 2008/2013), however mean recovery was 71% at 0.01 mg/kg.
one recovery of 69% at a fortification level of 0.05 mg/kg asulam (Boileau, 2013a), however overall mean recovery was 98%.
two recoveries of 64 and 69% at a fortification level of 0.01 and 0.1 mg/kg acetyl asulam (Boileau, 2013a), however mean recovery was 96% at 0.01 mg/kg.
one recovery of 64% at a fortification level of 0.1 mg/kg acetyl sulfanilamide (Boileau, 2013a), however overall mean recovery was 91%.
one recovery of 68% at a fortification level of 0.05 mg/kg acetyl sulfanilamide (Boileau, 2013a), however overall mean recovery was 99%.
two recoveries of 62 and 66% at a fortification level of 0.05 and 0.1 mg/kg formyl asulam (Boileau, 2013a), however overall mean recovery was 94%.
three recoveries of 62, 63 and 66% at a fortification level of 0.05, 0.01 and 0.1 mg/kg malonyl asulam (Boileau, 2013a), however mean recovery was 88% at 0.01 mg/kg.
one recovery of 66% at a fortification level of 0.01 mg/kg asulam (Boileau, 2013b), however mean recovery was 98% at 0.01 mg/kg.
one recovery of 68% at a fortification level of 2 mg/kg asulam glucosides (Boileau, 2013b), however overall mean recovery was 98%.
two recoveries of 65 and 68% at a fortification level of 0.01 and 2 mg/kg acetyl malonyl asulam (Boileau, 2013b), however mean recovery was 88% at 0.01 mg/kg.
one recovery of 69% at a fortification level of 0.01 mg/kg malonyl sulfanilamide (Boileau, 2013b), however mean recovery was 98% at 0.01 mg/kg.
two recoveries of 65 and 69% at a fortification level of 0.01 and 2 mg/kg acetyl sulfanilamide (Boileau, 2013b), however mean recovery was 86% at 0.01 mg/kg.

A full list of studies available is given in Annex A. Those evaluated are listed in Section B.7.18.

a) <u>2008/9 residue trial samples</u>

Samples were extracted three times with acetonitrile/water and the resulting extracts evaporated to an aqueous remainder (dilute formic acid/methanol added) and analysed by LC/MS/MS (monitoring for in the case of parent asulam the precursor ion m/z 231 and the product ion m/z 156 [and 92 for confirmation] and for malonyl asulam the precursor ion m/z 317 and the product ion m/z 242 [and precursor ion m/z 315 and product ion m/z 229 for confirmation], using a C_{18} column. The supporting method validation data are shown in table B.7.16.0. Representative chromatograms were submitted and were acceptable.

(Chambers, 2007)

Table B.7.16.0 Summary of method description and validation (treated plants)

Substrate	Analyte	Dissolution/ extraction	Partition, clean-up	Quantification	Limit of quantif-ication (mg/kg)	Recovery fortifica-tion level (mg/kg)	Recoveries % range (mean)	Repeatabil-ity RSD (%) (n)	Linearity demon-strated (mg/l)	Ref.
Plant										
Spinach	Asulam	Acetonitrile/ water	Evaporate to aqueous remainder	LC/MS/MS m/z 156	0.01	0.01 0.1	72-105 (94) 73-105 (89)	15 (6) 16 (5)	0.0008-0.02	Chambers, 2007
	Malonyl asulam			m/z 242	0.01	0.01 0.1	77-104 (92) 76-112 (96)	14 (6) 15 (5)	0.0008-0.02	

Table B.7.16.1 Summary of residues of asulam in spinach (outdoor foliar) – Northern Europe (trials which represent critical GAP are underlined)

Crop/ Variety	Country/ year	Application rate per treatment			No. of treat-ments	Growth stage at last treatment	Portion analysed	PHI (days)	Residues (asulam plus asulam-malonyl) (mg/kg)	Comments	Reference
		kg as/ha seed	Water (l/ha)	kg as/hl							
Spinach		**2.4**			**1**	**GS14**					
Spinach Triptiek	UK 2003	2.4	200	1.2	1	GS12-14	Leaves	0	80	Asulam = 70 mg/kg Malonyl asulam = 9.4 mg/kg	Bardel, 2004
								28	9.7	Asulam = 0.12 mg/kg Malonyl asulam = 12 mg/kg	
Spinach Cobra	Belgium 2003	2.4	200	1.2	1	GS12-13	Leaves	0	170	Asulam = 170 mg/kg Malonyl asulam = 3.1 mg/kg	Bardel, 2004
								28	1.1	Asulam = <0.05 mg/kg Malonyl asulam = 1.3 mg/kg	
Spinach MonetF1	Germany 2003	2.4	200	1.2	1	GS13	Leaves	0	160	Asulam = 160 mg/kg Malonyl asulam = 0.82 mg/kg	Bardel, 2004
								28	23	Asulam = 3.9 mg/kg Malonyl asulam = 22 mg/kg	
Spinach Matador	Germany 2003	2.4	200	1.2	1	GS13	Leaves	0	140	Asulam = 140 mg/kg Malonyl asulam = 0.5 mg/kg	Bardel, 2004
								28	0.19	Asulam = <0.05 mg/kg Malonyl asulam = 0.24 mg/kg	
Spinach Matador	Germany 2004	2.4	300	0.8	1	GS12-13	Leaves	0	140	Asulam = 140 mg/kg Malonyl asulam = 0.29 mg/kg	Bardel, 2004a
								1	77	Asulam = 71 mg/kg Malonyl asulam = 7.1 mg/kg	
								4	5.8	Asulam = 2 mg/kg Malonyl asulam = 4.7 mg/kg	
								5	5.4	Asulam = 1.4 mg/kg Malonyl asulam = 5 mg/kg	
								7	3.6	Asulam = 0.28 mg/kg Malonyl asulam = 4.1 mg/kg	
								14	1.9	Asulam = 0.1 mg/kg Malonyl asulam = 2.3 mg/kg	
								26	0.54	Asulam = <0.05 mg/kg Malonyl asulam = 0.61 mg/kg	

Crop/ Variety	Country/ year	Application rate per treatment			No. of treat-ments	Growth stage at last treatment	Portion analysed	PHI (days)	Residues (asulam plus asulam-malonyl) (mg/kg)	Comments	Reference
		kg as/ha seed	Water (l/ha)	kg as/hl							
Spinach		**2.4**			**1**	**GS14**					
Spinach Lazio	Netherlands 2004	2.4	300	0.8	1	GS13	Leaves	0	250	Asulam = 250 mg/kg Malonyl asulam = mg/kg	Bardel, 2004a
								1	200	Asulam = 190 mg/kg Malonyl asulam = mg/kg	
								3	91	Asulam = 76 mg/kg Malonyl asulam = mg/kg	
								5	24	Asulam = 8.3 mg/kg Malonyl asulam = mg/kg	
								7	14	Asulam = 3.4 mg/kg Malonyl asulam = mg/kg	
								14	16	Asulam = 1.9 mg/kg Malonyl asulam = mg/kg	
								28	1.6	Asulam = <0.05 mg/kg Malonyl asulam = mg/kg	
Spinach Czesanne	Germany 2004	2.4	300	0.8	1	GS12-13	Leaves	0	120	Asulam = 120 mg/kg Malonyl asulam = 0.29 mg/kg	Bardel, 2004a
								1	84	Asulam = 77 mg/kg Malonyl asulam = 8.6 mg/kg	
								3	8	Asulam = 2.5 mg/kg Malonyl asulam = 6.9 mg/kg	
								5	7	Asulam = 1.9 mg/kg Malonyl asulam =6.4 mg/kg	
								7	5	Asulam = 0.3 mg/kg Malonyl asulam = 5.9 mg/kg	
								14	2.4	Asulam = 0.22 mg/kg Malonyl asulam = 2.7 mg/kg	
								28	0.29	Asulam = <0.05 mg/kg Malonyl asulam = 0.36 mg/kg	

Crop/ Variety	Country/ year	Application rate per treatment			No. of treat-ments	Growth stage at last treatment	Portion analysed	PHI (days)	Residues (asulam plus asulam-malonyl) (mg/kg)	Comments	Reference
		kg as/ha seed	Water (l/ha)	kg as/hl							
Spinach		**2.4**			**1**	**GS14**					
Spinach Amazon	UK 2013	2.4	256	0.94	1	GS14	Leaves	0	250	Asulam = 250 mg/kg Malonyl asulam = 0.25 mg/kg Acetyl asulam = <0.01mg/kg Asulam glucosides = 3.3 mg/kg Acetyl sulphanilamide = <0.01mg/kg Desamino asulam = 0.03 mg/kg Formyl asulam = 0.07mg/kg Malonyl sulphanilamide=<0.05mg/kg Suphanilamide = 8.1 mg/kg	Boileau, 2013b
							Leaves GS41-45 (10-50% leaf mass – immature spinach)	15	2.6	Asulam = 0.07 mg/kg Malonyl asulam = 3.1 mg/kg Acetyl asulam = 0.02mg/kg Asulam glucosides = 3.1 mg/kg Acetyl sulphanilamide = <0.01mg/kg Desamino asulam = 0.03mg/kg Formyl asulam = 0.07mg/kg Malonyl sulphanilamide=<0.05mg/kg Suphanilamide = <0.01 mg/kg	
							Leaves GS47-49 (70% leaf mass – commercial harvest)	23	0.14	Asulam = <0.01 mg/kg Malonyl asulam = 0.17 mg/kg Acetyl asulam = <0.01mg/kg Asulam glucosides = 0.68 mg/kg Acetyl sulphanilamide = <0.01mg/kg Desamino asulam = <0.01mg/kg Formyl asulam = <0.01mg/kg Malonyl sulphanilamide=<0.05mg/kg Suphanilamide = <0.01 mg/kg	
Spinach Junius	N.France 2013	2.39	204	1.2	1	GS14	Leaves	0	180	Asulam = 180 mg/kg Malonyl asulam = 2.7 mg/kg Acetyl asulam = 0.02mg/kg Asulam glucosides = 49 mg/kg Acetyl sulphanilamide = <0.01mg/kg Desamino asulam = 0.02 mg/kg Formyl asulam = 0.24mg/kg Malonyl sulphanilamide=<0.05mg/kg Suphanilamide = 5.4 mg/kg	Boileau, 2013b

Crop/ Variety	Country/ year	Application rate per treatment			No. of treat- ments	Growth stage at last treatment	Portion analysed	PHI (days)	Residues(asulam plus asulam-malonyl)(mg/kg)	Comments	Reference
		kg as/ha seed	Water (l/ha)	kg as/hl							
Spinach		**2.4**			**1**	**GS14**					
							Leaves	3	23	Asulam = 3.5 mg/kg Malonyl asulam = 24 mg/kg Acetyl asulam = 0.05mg/kg Asulam glucosides = 104 mg/kg Acetyl sulphanilamide = 0.01mg/kg Desamino asulam = <0.01 mg/kg Formyl asulam = 0.24mg/kg Malonyl sulphanilamide=<0.05mg/kg Suphanilamide = 0.1 mg/kg	
							Leaves	7	20	Asulam = 1.2 mg/kg Malonyl asulam = 24 mg/kg Acetyl asulam = 0.07mg/kg Asulam glucosides = 50 mg/kg Acetyl sulphanilamide = 0.02mg/kg Desamino asulam = <0.01mg/kg Formyl asulam = 0.17mg/kg Malonyl sulphanilamide=<0.05mg/kg Suphanilamide = 0.03 mg/kg	
							Leaves GS41-45 (10-50% leaf mass – immature spinach)	21	7.3	Asulam = 0.06 mg/kg Malonyl asulam = 9 mg/kg Acetyl asulam = 0.05 mg/kg Asulam glucosides = 3.4 mg/kg Acetyl sulphanilamide = <0.01 mg/kg Desamino asulam = <0.01 mg/kg Formyl asulam = 0.03 mg/kg Malonyl sulphanilamide=<0.05mg/kg Suphanilamide = <0.01 mg/kg	
							Leaves GS47-49 (70% leaf mass – commercial harvest)	35	0.43	Asulam = <0.01 mg/kg Malonyl asulam = 0.54 mg/kg Acetyl asulam = <0.01mg/kg Asulam glucosides = 0.06 mg/kg Acetyl sulphanilamide = <0.01mg/kg Desamino asulam = <0.01mg/kg Formyl asulam = <0.01mg/kg Malonyl sulphanilamide=<0.05mg/kg Suphanilamide = <0.01 mg/kg	

Crop/ Variety	Country/ year	Application rate per treatment			No. of treat- ments	Growth stage at last treatment	Portion analysed	PHI (days)	Residues(asulam plus asulam-malonyl)(mg/kg)	Comments	Reference
		kg as/ha seed	Water (l/ha)	kg as/hl							
Spinach		**2.4**			**1**	**GS14**					

Spinach Beaver	N.France 2013	2.25	288	0.78	1	GS14	Leaves	0	140	Asulam = 140 mg/kg Malonyl asulam = 1.3 mg/kg Acetyl asulam = 0.01mg/kg Asulam glucosides = 8.5 mg/kg Acetyl sulphanilamide = <0.01mg/kg Desamino asulam = 0.15 mg/kg Formyl asulam = 0.06mg/kg Malonyl sulphanilamide=<0.05mg/kg Suphanilamide = 3.9 mg/kg	Boileau, 2013b
							Leaves	3	8.4	Asulam = 0.9 mg/kg Malonyl asulam = 8.4 mg/kg Acetyl asulam = <0.01mg/kg Asulam glucosides = 49 mg/kg Acetyl sulphanilamide = <0.01mg/kg Desamino asulam = <0.01 mg/kg Formyl asulam = 0.08mg/kg Malonyl sulphanilamide=<0.05mg/kg Suphanilamide = 0.03 mg/kg	
							Leaves	7	5.4	Asulam = 0.12 mg/kg Malonyl asulam = 6.7 mg/kg Acetyl asulam = <0.01mg/kg Asulam glucosides = 18 mg/kg Acetyl sulphanilamide = <0.01mg/kg Desamino asulam = <0.01mg/kg Formyl asulam = 0.03 mg/kg Malonyl sulphanilamide=<0.05mg/kg Suphanilamide = <0.01 mg/kg	
							Leaves GS41-45 (10-50% leaf mass – immature spinach)	21	2.6	Asulam = 0.08 mg/kg Malonyl asulam = 3.2 mg/kg Acetyl asulam = <0.01 mg/kg Asulam glucosides = 5.4 mg/kg Acetyl sulphanilamide = <0.01 mg/kg Desamino asulam = <0.01 mg/kg Formyl asulam = <0.01 mg/kg Malonyl sulphanilamide=<0.05mg/kg Suphanilamide = <0.01 mg/kg	
							Leaves GS47-49 (70% leaf mass – commercial harvest)	35	0.24	Asulam = <0.01 mg/kg Malonyl asulam = 0.3 mg/kg Acetyl asulam = <0.01mg/kg Asulam glucosides = 0.2 mg/kg Acetyl sulphanilamide = <0.01mg/kg Desamino asulam = <0.01mg/kg Formyl asulam = <0.01mg/kg Malonyl sulphanilamide=<0.05mg/kg Suphanilamide = <0.01 mg/kg	

Crop/ Variety	Country/ year	Application rate per treatment			No. of treat-ments	Growth stage at last treatment	Portion analysed	PHI (days)	Residues (asulam plus asulam-malonyl) (mg/kg)	Comments	Reference
		kg as/ha seed	Water (l/ha)	kg as/hl							
Spinach		**2.4**			**1**	**GS14**					
Spinach Symphonie/ F1 hybrid	N.France 2013	2.41	205	1.2	1	GS14	Leaves GS41-45 (10-50% leaf mass – immature spinach) Leaves GS47-49 (70% leaf mass – commercial harvest)	20 27	1.3 <u><0.02</u>	Asulam = 0.01 mg/kg Malonyl asulam = 1.6 mg/kg Acetyl asulam = <0.01mg/kg Asulam glucosides = 0.93 mg/kg Acetyl sulphanilamide = <0.01mg/kg Desamino asulam = <0.01 mg/kg Formyl asulam = 0.01mg/kg Malonyl sulphanilamide=<0.05mg/kg Suphanilamide = <0.01 mg/kg Asulam = <0.01 mg/kg Malonyl asulam = <0.01 mg/kg Acetyl asulam = <0.01 mg/kg Asulam glucosides = <0.01 mg/kg Acetyl sulphanilamide = <0.01mg/kg Desamino asulam = <0.01 mg/kg Formyl asulam = <0.01 mg/kg Malonyl sulphanilamide=<0.05mg/kg Suphanilamide = <0.01 mg/kg	Boileau, 2013b
Spinach Junius	N.France 2013	2.32	197	1.2	1	GS14	Leaves GS47-49 (70% leaf mass – commercial harvest)	27	<u>0.22</u>	Asulam = <0.01 mg/kg Malonyl asulam = 0.28 mg/kg Acetyl asulam = <0.01mg/kg Asulam glucosides = 0.06 mg/kg Acetyl sulphanilamide = <0.01mg/kg Desamino asulam = <0.01 mg/kg Formyl asulam = <0.01 mg/kg Malonyl sulphanilamide=<0.05mg/kg Suphanilamide = <0.01 mg/kg	Boileau, 2013b

Table B.7.16.1 Summary of residues of asulam in spinach (outdoor pre-emergence) – Northern Europe (trials which represent critical GAP are underlined)

Crop/ Variety	Country/ year	Application rate per treatment			No. of treat-ments	Growth stage at last treatment	Portion analysed	PHI (days)	Residues (asulam plus asulam-malonyl) (mg/kg)	Comments	Reference
		kg as/ha seed	Water (l/ha)	kg as/hl							
Spinach		**2.4**			**1**	**GS00**					
Spinach Bali	Netherlands 2008	2.42	707	0.34	1	GS00	Leaves	21 28 35 42 44	0.19 0.02 <0.02 <0.02 <u><0.02</u>	Asulam = 0.09 mg/kg Malonyl asulam = 0.12 mg/kg Asulam = <0.01 mg/kg Malonyl asulam = 0.02 mg/kg Asulam = <0.01 mg/kg Malonyl asulam = 0.01 mg/kg Asulam = <0.01 mg/kg Malonyl asulam = <0.01 mg/kg Asulam = <0.01 mg/kg Malonyl asulam = 0.01 mg/kg	Sandt, 2008/2013
Spinach Bali	Netherlands 2008	2.39	699	0.34	1	GS00	Leaves	28 48	0.63 <u>0.06</u>	Asulam = 0.03 mg/kg Malonyl asulam = 0.078 mg/kg Asulam = <0.01 mg/kg Malonyl asulam = 0.07 mg/kg	Sandt, 2008/2013
Spinach Yabi	Netherlands 2008	2.36	689	0.34	1	GS00	Leaves	21 28 35 42 44	0.02 <0.02 <0.02 <0.02 <u><0.02</u>	Asulam = <0.01 mg/kg Malonyl asulam = 0.03 mg/kg Asulam = <0.01 mg/kg Malonyl asulam =<0.01 mg/kg Asulam = <0.01 mg/kg Malonyl asulam = <0.01 mg/kg Asulam = <0.01 mg/kg Malonyl asulam = <0.01 mg/kg Asulam = <0.01 mg/kg Malonyl asulam = <0.01 mg/kg	Sandt, 2009
Spinach Toucan	Netherlands 2008	2.39	699	0.34	1	GS00	Leaves	28 40	0.02 <u><0.02</u>	Asulam = <0.01 mg/kg Malonyl asulam = 0.02 mg/kg Asulam = <0.01 mg/kg Malonyl asulam = <0.01 mg/kg	Sandt, 2009

Crop/ Variety	Country/ year	Application rate per treatment			No. of treat-ments	Growth stage at last treatment	Portion analysed	PHI (days)	Residues (asulam plus asulam-malonyl) (mg/kg)	Comments	Reference
		kg as/ha seed	Water (l/ha)	kg as/hl							
Spinach		**2.4**			**1**	**GS00**					
Spinach Amazon	UK 2013	2.4	256	0.94	1	GS00	Leaves GS41-45 (10-50% leaf mass – immature spinach)	42	<0.02	Asulam = <0.01 mg/kg Malonyl asulam =<0.01 mg/kg Acetyl asulam = <0.01mg/kg Asulam glucosides = <0.01mg/kg Acetyl sulphanilamide = <0.01mg/kg Desamino asulam = <0.01mg/kg Formyl asulam = <0.01mg/kg Malonyl sulphanilamide=<0.05mg/kg Suphanilamide = <0.01 mg/kg	Boileau, 2013a
							Leaves GS47-49 (70% leaf mass – commercial harvest)	50	<0.02	Asulam = <0.01 mg/kg Malonyl asulam = <0.01 mg/kg Acetyl asulam = <0.01mg/kg Asulam glucosides = <0.01mg/kg Acetyl sulphanilamide = <0.01mg/kg Desamino asulam = <0.01mg/kg Formyl asulam = <0.01mg/kg Malonyl sulphanilamide=<0.05mg/kg Suphanilamide = <0.01 mg/kg Rainfall = 12mm 12 hours after application	
Spinach Junius	N.France 2013	2.44	208	1.2	1	GS00	Leaves GS41-45 (10-50% leaf mass – immature spinach)	44	0.09	Asulam = <0.01 mg/kg Malonyl asulam = mg/kg Acetyl asulam = <0.01mg/kg Asulam glucosides = <0.01mg/kg Acetyl sulphanilamide = <0.01mg/kg Desamino asulam = <0.01mg/kg Formyl asulam = <0.01mg/kg Malonyl sulphanilamide=<0.05mg/kg Suphanilamide = <0.01 mg/kg	Boileau, 2013a
							Leaves GS47-49 (70% leaf mass – commercial harvest)	58	0.07	Asulam = <0.01 mg/kg Malonyl asulam = mg/kg Acetyl asulam = <0.01mg/kg Asulam glucosides = 0.13mg/kg Acetyl sulphanilamide = <0.01mg/kg Desamino asulam = <0.01mg/kg Formyl asulam = <0.01mg/kg Malonyl sulphanilamide=<0.05mg/kg Suphanilamide = <0.01 mg/kg	

Crop/ Variety	Country/ year	Application rate per treatment			No. of treat-ments	Growth stage at last treatment	Portion analysed	PHI (days)	Residues (asulam plus asulam-malonyl) (mg/kg)	Comments	Reference
		kg as/ha seed	Water (l/ha)	kg as/hl							
Spinach		**2.4**			**1**	**GS00**					
Spinach Symphonie Hybride F1	N.France 2013	2.28	388	0.59	1	GS00	Leaves GS41-45 (10-50% leaf mass – immature spinach)	47	0.03	Asulam = <0.01 mg/kg Malonyl asulam = mg/kg Acetyl asulam = <0.01mg/kg Asulam glucosides = <0.01mg/kg Acetyl sulphanilamide = <0.01mg/kg Desamino asulam = <0.01mg/kg Formyl asulam = <0.01 Malonyl sulphanilamide=<0.05mg/kg Suphanilamide = <0.01 mg/kg	Boileau, 2013a
							Leaves GS47-49 (70% leaf mass – commercial harvest)	54	0.02	Asulam = <0.01 mg/kg Malonyl asulam = mg/kg Acetyl asulam = <0.01mg/kg Asulam glucosides = <0.01 mg/kg Acetyl sulphanilamide = <0.01mg/kg Desamino asulam = <0.01mg/kg Formyl asulam = <0.01mg/kg Malonyl sulphanilamide=<0.05mg/kg Suphanilamide = <0.01 mg/kg	

Spinach Beaver	N.France 2013	2.36	302	0.78	1	GS00	Leaves GS41-45 (10-50% leaf mass – immature spinach) Leaves GS47-49 (70% leaf mass – commercial harvest)	33 47	<0.02 <0.02	Asulam = <0.01 mg/kg Malonyl asulam = <0.01 mg/kg Acetyl asulam = <0.01mg/kg Asulam glucosides = <0.01mg/kg Acetyl sulphanilamide = <0.01mg/kg Desamino asulam = <0.01mg/kg Formyl asulam = <0.01mg/kg Malonyl sulphanilamide=<0.05mg/kg Suphanilamide = <0.01 mg/kg Asulam = <0.01 mg/kg Malonyl asulam = <0.01 mg/kg Acetyl asulam = <0.01mg/kg Asulam glucosides = <0.01 mg/kg Acetyl sulphanilamide = <0.01mg/kg Desamino asulam = <0.01mg/kg Formyl asulam = <0.01mg/kg Malonyl sulphanilamide=<0.05mg/kg Suphanilamide = <0.01 mg/kg	Boileau, 2013a

Table B.7.17 Summary of residues of asulam in spinach (outdoor foliar) – Southern Europe (trials which represent critical GAP are underlined)

Crop/ Variety	Country/ year	Application rate per treatment			No. of treat-ments	Growth stage at last treatment	Portion analysed	PHI (days)	Residues (asulam plus asulam-malonyl) (mg/kg)	Comments	Reference
		kg as/ha seed	Water (l/ha)	kg as/hl							
Spinach		**2.4**			**1**	**GS14**					
Spinach Goya	Spain 2003	2.4	200	1.2	1	GS12-13	Leaves	0 28	230 0.7	Asulam = 230 mg/kg Malonyl asulam = 4.5 mg/kg Asulam = <0.05 mg/kg Malonyl asulam = 0.88 mg/kg	Bardel, 2004d
Spinach Monstrueux De Viroflay	S.France 2004	2.4	300	0.8	1	GS14	Leaves	0 28	180 0.47	Asulam = 180 mg/kg Malonyl asulam = 3.9 mg/kg Asulam = <0.05 mg/kg Malonyl asulam = 0.59 mg/kg	Bardel, 2004f

Note: Malonyl asulam residue is multiplied by 0.8 to convert it to parent asulam

B.7.6.1 Summary of residues resulting from supervised trials

B.7.6.1.1 Spinach

Northern Europe (Post emergence use)
Residues trials data were available for spinach grown in N.France, Germany, UK, Belgium and Netherlands. Twelve trials support the proposed critical GAP with residues in Spinach up to 23 mg/kg (appropriate EUMRL = 30 mg/kg; STMR = 0.36 mg/kg).

Northern Europe (Pre-emergence use)
Residues trials data were available for spinach grown in UK, N.France and Netherlands. Eight trials support the proposed critical GAP with residues in Spinach up to 0.07 mg/kg (appropriate EUMRL = 30 mg/kg; STMR = <0.02 mg/kg).

Southern Europe
Residues trials data were available for spinach grown in Spain and S.France (treated post emergence). Only two trail are available giving residues in Spinach up to 0.88 mg/kg, however no approval is being sort in SMS.

Residues of metabolites in trial samples
Residue trial samples from 2013 were also analysed for the following metabolites, as outlined in the EFSA conclusion.

Acetyl asulam
Residues in all the 2013 trials were below the limit of determination (0.01 mg/kg) at harvest (in immature crops were up to 0.07 mg/kg).

Asulam glucosides
Residues in the 2013 post emergence trials were up to 0.68 mg/kg (residues of asulam and malonyl asulam in the sample were 0.17 mg/kg) at harvest, with 4 out of the 5 trials containing residues above 0.01 mg/kg (1 out of the 3 pre-emergence 2013 trials gave a residues of 0.13 mg/kg at harvest). Immature crops gave residues of up to 104 mg/kg on day 3, decreasing to up to 5.4 mg/kg by day 21.
Consideration should be given to the inclusion of asulam glucosides into the residues definition (monitoring method has been validated for this metabolite). However, the levels seen at harvest were well below the maximum level of asulam and its metabolite malonyl asulam seen at harvest (12 mg/kg) and would only increase the STMR slightly to 0.61 mg/kg. Only 5 of the 12 residue trials were analysed for asulam glucoside, however possibly in the 2003/4 trials where high levels of malonyl asulam were detected at harvest, the method employed was not capable of separating malonyl asulam from asulam glucosides and both give similar mass fragments of 242 (malonyl asulam) and 239 (asulam glucosides).

Acetyl sulphanilamide
Residues in all the 2013 trials were below the limit of determination (0.01 mg/kg) at harvest (in immature crops were up to 0.02 mg/kg).

Desamino asulam
Residues in all the 2013 trials were below the limit of determination (0.01 mg/kg) at harvest (in immature crops were up to 0.15 mg/kg).

Formyl asulam
Residues in all the 2013 trials were below the limit of determination (0.01 mg/kg) at harvest (in immature crops were up to 0.24 mg/kg).

Malonyl sulphanilamide
Residues in all the 2013 trials were below the limit of determination (0.05 mg/kg) at harvest.

Sulphanailamide
Residues in all the 2013 trials were below the limit of determination (0.01 mg/kg) at harvest (in immature crops were up to 8.1 mg/kg on day 0 declining quickly to up to 0.1 mg/kg by day 3, 0.03 mg/kg on day 7 and less than 0.01 mg/kg by day 21).

Asulam Dimers (Applicants case for non-submission of residues trials data)
A spinach study not conducted to the GAP suggested that low-level unidentified components of the residue could be asulam 'dimers' of some sort or another. EFSA concluded that in crops grown according to GAP these 'dimers' might reach significant levels and so should be taken into account in residue trials. A new study conducted outdoors in support of the post-emergence spinach GAP showed conclusively that such compounds did not reach significant levels in the residue and that there was no evidence for their existence. Therefore there was no justification for any additional metabolites to be included in the target analytes. The list of analytes for the new residue studies conducted in 2013 [see section CA 6.3] was very comprehensive including parent, asulam glucosides and malonyl asulam, as seen to be significant in the pre- and post-emergence spinach studies but also desamino asulam (seen at low levels in the pre-emergence application study), acetyl asulam and formyl asulam as well as acetyl sulfanilamide, malonyl sulfanilamide (which could theoretically be formed from acetyl asulam and malonyl asulam) and sulfanilamide (to demonstrate that it was not a significant plant metabolite). All credible potential residue components were covered by the validated analytical method. It can be concluded that there is no justification to also analyse for asulam related dimer compounds.

(Lowden and Peatman 2014)

B.7.7 Stability of residues prior to analysis

This study has been evaluated at EU level and the conclusions presented are consistent with those in the EFSA report

B.7.7.1 Crops

Samples of spinach were fortified at 0.5 mg/kg with asulam and its metabolite asulam-malonyl and were stored in a freezer (at -18°C) for up to 11 months. Samples were taken from storage at intervals and analysed by the method described in Section B.5.2. Recoveries are detailed in Table B.7.18 and show that residues of asulam are stable for up to 3 months in spinach and its metabolite asulam-malonyl for at least 11 months.

(Bardel 2005 and Bardel 2004b)

Table B.7.18 Stability of residues in spinach following storage at -18°C

Freezer storage period (months)	Recoveries (%) Spinach
0	
Asulam	93
Asulam-malonyl	99
0.5	
Asulam	92
Asulam-malonyl	-
1	
Asulam	84
Asulam-malonyl	-
3	
Asulam	67
Asulam-malonyl	96
6	
Asulam	53
Asulam-malonyl	112
9	
Asulam	37
Asulam-malonyl	-
11	-
Asulam	105
Asulam-malonyl	

B.7.7.2 Summary

Residues of asulam are stable for up to 3 months in spinach and its metabolite asulam-malonyl for at least 11 months, when stored at -18°C.

B.7.8 Effects of industrial processing and/or household processing (IIA 6.5, IIIA 8.4)

These study have been evaluated at EU level and the conclusions presented are consistent with those in the EFSA report, with the exception of the new cold processing study (Boileau, 2013)

B.7.8.1 Effects on the nature of the residue

Studies of the stability of aqueous solutions of asulam and its metabolite were conducted in 2003. Stocks of [^{14}C] phenyl labelled asulam (radiochemical purity >99%) and [^{14}C] phenyl labelled asulam-malonyl (radiochemical purity >98%) in deionised water was used to spike ammonium acetate/acetic acid buffer, under three test conditions: pH 4 at 90°C, pH 5 at 100°C and pH 6 at 120°C. Buffers were prepared using drinking water. The solutions contained 1 mg/l asulam and 4 mg/l asulam-malonyl. The tests were of duration 20, 60 and 20 minutes respectively, and concentrations of substance at the start and end of the exposure periods were measured. The total amount of [^{14}C] phenyl labelled asulam and [^{14}C] phenyl labelled asulam-malonyl were determined by LSC, and recovery values were obtained by HPLC-UV and LC/MS. For the solutions pre-treatment, recoveries were greater than 99%, however after treatment recoveries of asulam were 39-75% and for asulam-malonyl were 70-95%. Therefore, it can be concluded that asulam and its metabolite asulam-malonyl degrade abiotically under conditions representative of industrial or domestic food processing, most notable at pH 5 and 6 (see table B.7.19), forming the metabolite sulphanilamide [23-62%] (or the malonyl derivative [7-27%]), which was also identified in the rat metabolism study.

(Fitzmaurice 2003a and b)

Table B.7.19 Levels of asulam and its metabolite after industrial or domestic food processing

	Hydrolysis conditions		
	pH 4, 90ºC, 20mins pasteurisation	pH 5, 100ºC, 60mins baking, brewing and boiling	pH 6, 120ºC, 20mins Sterilisation
Asulam Sulfanilamide	75% 23%	39% 62%	48% 49%
Asulam-malonyl Sulfanilamide-malonyl	95% 7%	75% 26%	70% 27%

B.7.8.2 Effects on residue levels

B.7.8.2.1 Spinach

Five spinach processing studies were carried out in Germany in 2003/4. The spinach samples were obtained from residue trials carried out in Germany. On analysis of the spinach by the method in Section B.5.2 (in the case of sulphanilamide-malonyl by a virtually identical LC/MS/MS method monitoring for the precursor ion m/z 259 and the product ion 162), residues of asulam, asulam-malonyl and sulphanilamide-malonyl in the spinach were 4 (only one of the five samples contained positive residues), 0.24-22 and less than 0.05 mg/kg respectively. On cooking the spinach samples and analysing the processed samples by the method in Section B.5.2 (samples were stored frozen for up to 2 months), residues of asulam and its metabolite asulam-malonyl had decreased by a factor of 0.9 and 0.8 respectively (residues of sulphanilamide-malonyl remained below the limit of determination [0.05 mg/kg]).

(Hoffmann, 2004a and b)

Four spinach processing studies were carried out in 2013, in France, [see Table B.7.20 and Table B.7.21]. The spinach samples were obtained from residue trials carried out in N.France. On analysis of the spinach by method B.5.2, residues of asulam plus its metabolite malonyl asulam (residues definition) in the samples were 3.2 (immature spinach), 0.34 (mature spinach); 13 (mature spinach treated late at GS 43 instead of GS 14 to increase residue in samples at harvest) and 0.28 (mature spinach) mg/kg; total asulam in the samples were 5.7 (immature spinach), 0.34 (mature spinach); 13 (mature spinach treated late at GS 43 instead of GS 14 to increase residue in samples at harvest) and 0.28 (mature spinach) mg/kg . On processing the spinach samples and analysing the processed samples by method B.5.2 (Spinach and processed samples were stored frozen for only 2 days; Brine water analysed by direct injection into LC/MS/MS – See table B.7.22 for additional validation data;), residues of asulam/malonyl asulam [and total asulam] in the processed samples had decreased (canned spinach by a factor of 0.5 [0.4]; cooked spinach by a factor of 0.5 [0.4] and frozen spinach by a factor of 0.7 [0.7]). Residues of Sulfanilamide and Malonyl sulphanilamide in mature spinach and processed spinach were below the limit of determination, the only exception being spinach treated at a later growth stage. Desamino asulam and Asulam glucosides showed significant residues in processed products, with levels of desamino asulam increasing, however in both cases significant residues were only seen in either immature spinach or spinach treated at a later growth stage.

(Boileau, 2013b)

Table B.7.20 Residues of asulam and its metabolites in spinach and processed products (mg/kg)

	Study 1 (Immature spinach) (mg/kg)	Study 2 (Mature spinach) (mg/kg)	Study 3 (Mature spinach treated at GS43)** (mg/kg)	Study 4 (Mature spinach) (mg/kg)
Asulam				
Fresh spinach	0.026	<0.01	0.8	<0.01
Canned spinach	0.013	<0.01	0.41	<0.01
Brine water*	0.087	0.008	1.2	0.006
Cooked	0.044	<0.01	0.67	<0.01
Frozen	0.014	<0.01	0.78	<0.01
Acetyl asulam				
Fresh spinach	<0.01	<0.01	0.16	<0.01
Canned spinach	<0.01	<0.01	0.1	<0.01
Brine water*	<0.002	<0.002	0.038	<0.002
Cooked	<0.01	<0.01	0.12	<0.01
Frozen	<0.01	<0.01	0.14	<0.01
Asulam glucoside				
Fresh spinach	4.8	0.092	21	0.064
Canned spinach	0.84	0.029	5.2	0.014
Brine water*	1.2	0.026	5.8	0.011
Cooked	1.1	0.032	9.6	0.015
Frozen	2.2	0.071	13	0.054
Acetyl sulfanilamide				
Fresh spinach	<0.01	<0.01	<0.01	<0.01
Canned spinach	<0.01	<0.01	<0.01	<0.01
Brine water*	0.021	<0.01	0.028	<0.01
Cooked	<0.01	<0.01	<0.01	<0.01
Frozen	<0.01	<0.01	<0.01	<0.01
Desamino asulam				
Fresh spinach	<0.01	<0.01	0.016	<0.01
Canned spinach	0.014	<0.01	0.38	<0.01
Brine water*	<0.002	<0.002	0.004	<0.002
Cooked	<0.01	<0.01	0.12	<0.01
Frozen	0.012	<0.01	0.049	<0.01
Formyl asulam				
Fresh spinach	0.016	<0.01	0.11	<0.01
Canned spinach	<0.01	<0.01	0.082	<0.01
Brine water*	0.005	<0.002	0.064	0.003
Cooked	<0.01	<0.01	0.11	<0.01
Frozen	<0.01	<0.01	0.11	<0.01
Malonyl asulam				
Fresh spinach	3.2	0.34	12	0.28
Canned spinach	0.81	0.17	7.7	0.15
Brine water*	1	0.24	14	0.14
Cooked	0.87	0.21	7.6	0.095
Frozen	1.4	0.21	9.8	0.23
Malonyl sulfanilamide				
Fresh spinach	<0.05	<0.05	0.031	<0.05
Canned spinach	<0.05	<0.05	0.083	<0.05
Brine water*	<0.01	<0.01	0.084	<0.01
Cooked	<0.05	<0.05	0.077	<0.05

Frozen	<0.05	<0.05	0.052	<0.05
Sulfanilamide				
Fresh spinach	<0.01	<0.01	<0.01	<0.01
Canned spinach	<0.01	<0.01	<0.01	<0.01
Brine water*	0.019	<0.01	0.23	<0.01
Cooked	<0.01	<0.01	0.021	<0.01
Frozen	<0.01	<0.01	0.014	<0.01

*From canned spinach in mg/l
**Spinach was treated at GS43 (30% leaf mass) instead of the required GS14 in order to increase the residue in the spinach at harvest and more give a more accurate processing factor

Table B.7.21 Transfer factors

	Study 1 (Immature spinach) (mg/kg)	Study 2 (Mature spinach) (mg/kg)	Study 3 (Mature spinach treated at GS43)** (mg/kg)	Study 4 (Mature spinach) (mg/kg)	Transfer factor
Asulam/Malonyl asulam					
Fresh spinach	2.6	0.27	10	0.22	-
Canned spinach	0.66	0.14	6.6	0.12	0.5
Brine water*	0.89	0.2	12	0.12	0.7
Cooked	0.74	0.17	6.8	0.08	0.5
Frozen	1.1	0.17	8.6	0.18	0.7
Total asulam***					
Fresh spinach	5.7	0.33	24	0.26	-
Canned spinach	1.2	0.16	11	0.13	0.4
Brine water*	1.7	0.22	17	0.13	0.5
Cooked	1.5	0.19	13	0.085	0.4
Frozen	2.6	0.22	17	0.22	0.7

*From canned spinach in mg/l
**Spinach was treated at GS43 (30% leaf mass) instead of the required GS14 in order to increase the residue in the spinach at harvest and more give a more accurate processing factor
***Total asulam = parent plus all metabolites

Table B.7.22 Additional validation data for processing studies on Brine water

Substrate	Analyte	Fortification level (mg/1)	Recovery % range (mean)	%RSD (n)	Limit of determination (mg/l)
Brine water	Asulam	0.002-0.14	61-88 (71)*	13(6)	0.002
	Acetyl asulam	0.002-5	75-97 (84)	12 (5)	0.002
	Asulam glucosides	0.01-5	100-107(103)	3(4)	0.01
	Acetyl sulphanilamide	0.01-0.25	73-81(77)	5.6(3)	0.01
	Desamino asulam	0.002-5	65-101(83)**	14(6)	0.002
	Formyl asulam	0.002-5	73-88(82)	7.9(6)	0.002
	Malonyl asulam	0.002-10	70-113(99)	4.7(6)	0.002
	Malonyl sulphanilamide	0.01-0.25	103-109(108)	4.7(3)	0.01
	Sulphanilamide	0.01-0.26	69-73(71)***	-(2)	0.01

*Recoveries at 0.002 mg/l were 61 and 69%
**Recovery at 0.004 mg/l was 65%
***Recovery at 0.26 mg/l was 69%

B.7.8.3 Summary/assessment

Processing studies were carried out on spinach and showed that residues of asulam plus its metabolite asulam-malonyl in the canned/cooked/frozen samples had decreased (by a factor of 0.5, 0.6 and 0.7 respectively). With regards to Sulfanilamide and Malonyl sulphanilamide, residues in mature spinach and processed spinach were below the limit of determination. Studies on the nature of the residue indicate that asulam and its metabolite asulam-malonyl degrade abiotically under conditions representative of industrial or domestic food processing, most notable at pH 5 and 6 forming the metabolite sulphanilamide [23-62%] (or the malonyl derivative [7-27%]).

B.7.9 Livestock feeding studies (IIA 6.4, IIIA 8.3)

B.7.9.1 Cows

No data were submitted or required, due to approval only being required on spinach which is not normally fed to cattle.

B.7.9.2 Chickens

No data were submitted or required, due to approval only being required on spinach, which is not normally fed to chickens.

B.7.10 Residues in succeeding or rotational crops (IIA 6.6, IIIA 8.5)

No data (US study was submitted but was deemed not to be relevant) were submitted or required, due to total [^{14}C] residues in rotational crops at harvest being less than 0.2 mg/kg in the rotational crop metabolism study, with the exception of wheat straw which gave residues of up to 0.6 mg/kg. However, it is unlikely that residues of asulam in the soil would contribute significantly to the residue in following cereal crops from the application of asulam to spinach for the following reasons:

a) The study being carried out at 1.6N
b) radiolabelled asulam being applied to bare soil instead of a crop.
c) the low levels of parent asulam and its metabolites found in rotational crops at harvest (<0.05 mg/kg)
d) the DT50 and DT90 for asulam being 3.2 and 11 days respectively and for sulphanilamide 28 and 94 days respectively.
e) total [^{14}C] residues at harvest in crops consumed by humans being less than 0.2 mg/kg.
f) the low probability of crop failure

B.7.11 Proposed pre-harvest intervals for envisaged uses or witholding periods, in the case of post harvest uses (Annex IIA 6.8, Annex IIIA 8.7)

Current Good Agricultural Practice is summarised in Volume 1 Table 1.1. No additional information has been submitted.

B.7.12 Community MRL's and MRL's in EU Member States (IIIA 12.2)

Code number	Groups and examples of individual products to which the MRLs apply [a]	asulam
100000	1. FRUIT FRESH OR FROZEN; NUTS	0,5
110000	(i) Citrus fruit	0,5
110010	Grapefruit (Shaddocks, pomelos, sweeties, tangelo, ugli and other hybrids)	0,5
110020	Oranges (Bergamot, bitter orange, chinotto and other hybrids)	0,5
110030	Lemons (Citron, lemon)	0,5
110040	Limes	0,5
110050	Mandarins (Clementine, tangerine and other hybrids)	0,5
110990	Others	0,5
120000	(ii) Tree nuts (shelled or unshelled)	0,5
120010	Almonds	0,5
120020	Brazil nuts	0,5
120030	Cashew nuts	0,5
120040	Chestnuts	0,5
120050	Coconuts	0,5
120060	Hazelnuts (Filbert)	0,5
120070	Macadamia	0,5
120080	Pecans	0,5
120090	Pine nuts	0,5
120100	Pistachios	0,5
120110	Walnuts	0,5
120990	Others	0,5
130000	(iii) Pome fruit	0,5
130010	Apples (Crab apple)	0,5
130020	Pears (Oriental pear)	0,5
130030	Quinces	0,5
130040	Medlar	0,5
130050	Loquat	0,5
130990	Others	0,5
140000	(iv) Stone fruit	0,5
140010	Apricots	0,5
140020	Cherries (sweet cherries, sour cherries)	0,5
140030	Peaches (Nectarines and similar hybrids)	0,5
140040	Plums (Damson, greengage, mirabelle)	0,5

Code number	Groups and examples of individual products to which the MRLs apply [a]	asulam
140990	Others	0,5
150000	(v) Berries & small fruit	0,5
151000	(a) Table and wine grapes	0,5
151010	Table grapes	0,5
151020	Wine grapes	0,5
152000	(b) Strawberries	0,5
153000	(c) Cane fruit	0,5
153010	Blackberries	0,5
153020	Dewberries (Loganberries, Boysenberries, and cloudberries)	0,5
153030	Raspberries (Wineberries)	0,5
153990	Others	0,5
154000	(d) Other small fruit & berries	0,5
154010	Blueberries (Bilberries cowberries (red bilberries))	0,5
154020	Cranberries	0,5
154030	Currants (red, black and white)	0,5
154040	Gooseberries (Including hybrids with other ribes species)	0,5
154050	Rose hips	0,5
154060	Mulberries (arbutus berry)	0,5
154070	Azarole (mediteranean medlar)	0,5
154080	Elderberries (Black chokeberry (appleberry), mountain ash, azarole, buckthorn (sea sallowthorn), hawthorn, service berries, and other treeberries)	0,5
154990	Others	0,5
160000	(vi) Miscellaneous fruit	0,5
161000	(a) Edible peel	0,5
161010	Dates	0,5
161020	Figs	0,5
161030	Table olives	0,5
161040	Kumquats (Marumi kumquats, nagami kumquats)	0,5
161050	Carambola (Bilimbi)	0,5

Code number	Groups and examples of individual products to which the MRLs apply [a]	asulam
161060	Persimmon	0,5
161070	Jambolan (java plum) (Java apple (water apple), pomerac, rose apple, Brazilean cherry (grumichama), Surinam cherry)	0,5
161990	Others	0,5
162000	(b) Inedible peel, small	0,5
162010	Kiwi	0,5
162020	Lychee (Litchi) (Pulasan, rambutan (hairy litchi))	0,5
162030	Passion fruit	0,5
162040	Prickly pear (cactus fruit)	0,5
162050	Star apple	0,5
162060	American persimmon (Virginia kaki) (Black sapote, white sapote, green sapote, canistel (yellow sapote), and mammey sapote)	0,5
162990	Others	0,5
163000	(c) Inedible peel, large	0,5
163010	Avocados	0,5
163020	Bananas (Dwarf banana, plantain, apple banana)	0,5
163030	Mangoes	0,5
163040	Papaya	0,5
163050	Pomegranate	0,5
163060	Cherimoya (Custard apple, sugar apple (sweetsop) , llama and other medium sized Annonaceae)	0,5
163070	Guava	0,5
163080	Pineapples	0,5
163090	Bread fruit (Jackfruit)	0,5
163100	Durian	0,5
163110	Soursop (guanabana)	0,5
163990	Others	0,5
200000	2. VEGETABLES FRESH OR FROZEN	
210000	(i) Root and tuber vegetables	
211000	(a) Potatoes	0,05*
212000	(b) Tropical root and tuber	0,5

Code number	Groups and examples of individual products to which the MRLs apply [a]	asulam
	vegetables	
212010	Cassava (Dasheen, eddoe (Japanese taro), tannia)	0,5
212020	Sweet potatoes	0,5
212030	Yams (Potato bean (yam bean), Mexican yam bean)	0,5
212040	Arrowroot	0,5
212990	Others	0,5
213000	(c) Other root and tuber vegetables except sugar beet	0,5
213010	Beetroot	0,5
213020	Carrots	0,5
213030	Celeriac	0,5
213040	Horseradish	0,5
213050	Jerusalem artichokes	0,5
213060	Parsnips	0,5
213070	Parsley root	0,5
213080	Radishes (Black radish, Japanese radish, small radish and similar varieties)	0,5
213090	Salsify (Scorzonera, Spanish salsify (Spanish oysterplant))	0,5
213100	Swedes	0,5
213110	Turnips	0,5
213990	Others	0,5
220000	(ii) Bulb vegetables	0,5
220010	Garlic	0,5
220020	Onions (Silverskin onions)	0,5
220030	Shallots	0,5
220040	Spring onions (Welsh onion and similar varieties)	0,5
220990	Others	0,5
230000	(iii) Fruiting vegetables	0,5
231000	(a) Solanacea	0,5
231010	Tomatoes (Cherry tomatoes,)	0,5
231020	Peppers (Chilli peppers)	0,5
231030	Aubergines (egg plants) (Pepino)	0,5
231040	Okra, lady's fingers	0,5
231990	Others	0,5

Asulam sodium - Volume 3, Annex B.7 : Residues

Code number	Groups and examples of individual products to which the MRLs apply [a]	asulam
232000	(b) Cucurbits - edible peel	0,5
232010	Cucumbers	0,5
232020	Gherkins	0,5
232030	Courgettes (Summer squash, marrow (patisson))	0,5
232990	Others	0,5
233000	(c) Cucurbits-inedible peel	0,5
233010	Melons (Kiwano)	0,5
233020	Pumpkins (Winter squash)	0,5
233030	Watermelons	0,5
233990	Others	0,5
234000	(d) Sweet corn	0,5
239000	(e) Other fruiting vegetables	0,5
240000	(iv) Brassica vegetables	0,5
241000	(a) Flowering brassica	0,5
241010	Broccoli (Calabrese, Chinese broccoli, Broccoli raab)	0,5
241020	Cauliflower	0,5
241990	Others	0,5
242000	(b) Head brassica	0,5
242010	Brussels sprouts	0,5
242020	Head cabbage (Pointed head cabbage, red cabbage, savoy cabbage, white cabbage)	0,5
242990	Others	0,5
243000	(c) Leafy brassica	0,5
243010	Chinese cabbage (Indian (Chinese) mustard, pak choi, Chinese flat cabbage (tai goo choi), peking cabbage (pe-tsai), cow cabbage)	0,5
243020	Kale (Borecole (curly kale), collards)	0,5
243990	Others	0,5
244000	(d) Kohlrabi	0,5
250000	(v) Leaf vegetables & fresh herbs	0,5
251000	(a) Lettuce and other salad plants including Brassicacea	0,5
251010	Lamb´s lettuce (Italian cornsalad)	0,5
251020	Lettuce (Head lettuce, lollo rosso (cutting lettuce), iceberg lettuce, romaine (cos) lettuce)	0,5
251030	Scarole (broad-leaf endive) (Wild chicory, red-leaved chicory, radicchio, curld leave endive, sugar loaf)	0,5
251040	Cress	0,5
251050	Land cress	0,5
251060	Rocket, Rucola (Wild rocket)	0,5
251070	Red mustard	0,5
251080	Leaves and sprouts of Brassica spp (Mizuna)	0,5
251990	Others	0,5
252000	(b) Spinach & similar (leaves)	0,5
252010	Spinach (New Zealand spinach, turnip greens (turnip tops))	0,5
252020	Purslane (Winter purslane (miner's lettuce), garden purslane, common purslane, sorrel, glassworth)	0,5
252030	Beet leaves (chard) (Leaves of beetroot)	0,5
252990	Others	0,5
253000	(c) Vine leaves (grape leaves)	0,5
254000	(d) Water cress	0,5
255000	(e) Witloof	0,5
256000	(f) Herbs	0,5
256010	Chervil	0,5
256020	Chives	0,5
256030	Celery leaves (fennel leaves , Coriander leaves, dill leaves, Caraway leaves, lovage, angelica, sweet cisely and other Apiacea)	0,5
256040	Parsley	0,5
256050	Sage (Winter savory, summer savory,)	0,5
256060	Rosemary	0,5
256070	Thyme (marjoram, oregano)	0,5
256080	Basil (Balm leaves, mint, peppermint)	0,5
256090	Bay leaves (laurel)	0,5
256100	Tarragon (Hyssop)	0,5
256990	Others	0,5
260000	(vi) Legume vegetables (fresh)	0,5
260010	Beans (with pods) (Green bean (french beans, snap beans), scarlet runner bean, slicing bean, yardlong beans)	0,5
260020	Beans (without pods) (Broad beans, Flageolets, jack bean, lima bean, cowpea)	0,5
260030	Peas (with pods) (Mangetout (sugar peas))	0,5
260040	Peas (without pods) (Garden pea, green pea, chickpea)	0,5
260050	Lentils	0,5
260990	Others	0,5
270000	(vii) Stem vegetables (fresh)	0,5
270010	Asparagus	0,5
270020	Cardoons	0,5
270030	Celery	0,5
270040	Fennel	0,5
270050	Globe artichokes	0,5
270060	Leek	0,5
270070	Rhubarb	0,5
270080	Bamboo shoots	0,5
270090	Palm hearts	0,5
270990	Others	0,5
280000	(viii) Fungi	0,5
280010	Cultivated (Common mushroom, Oyster mushroom, Shi-take)	0,5
280020	Wild (Chanterelle, Truffle, Morel ,)	0,5
280990	Others	0,5
290000	(ix) Sea weeds	0,05*
300000	3. PULSES, DRY	0,05*
300010	Beans (Broad beans, navy beans, flageolets, jack beans, lima beans, field beans, cowpeas)	0,05*
300020	Lentils	0,05*
300030	Peas (Chickpeas, field peas, chickling vetch)	0,05*
300040	Lupins	0,05*
300990	Others	0,05*
400000	4. OILSEEDS AND OILFRUITS	
401000	(i) Oilseeds	0,05*
401010	Linseed	0,05*
401020	Peanuts	0,05*
401030	Poppy seed	0,05*
401040	Sesame seed	0,05*
401050	Sunflower seed	0,05*
401060	Rape seed (Bird rapeseed, turnip rape)	0,05*
401070	Soya bean	0,05*
401080	Mustard seed	0,05*
401090	Cotton seed	0,05*
401100	Pumpkin seeds	0,05*
401110	Safflower	0,05*
401120	Borage	0,05*
401130	Gold of pleasure	0,05*
401140	Hempseed	0,05*
401150	Castor bean	0,05*
401990	Others	0,05*
402000	(ii) Oilfruits	
402010	Olives for oil production	0,5
402020	Palm nuts (palmoil kernels)	0,05*
402030	Palmfruit	0,05*
402040	Kapok	0,05*
402990	Others	0,05*
500000	5. CEREALS	0,05*
500010	Barley	0,05*
500020	Buckwheat	0,05*
500030	Maize	0,05*
500040	Millet (Foxtail millet, teff)	0,05*
500050	Oats	0,05*
500060	Rice	0,05*
500070	Rye	0,05*
500080	Sorghum	0,05*
500090	Wheat (Spelt Triticale)	0,05*
500990	Others	0,05*
600000	6. TEA, COFFEE, HERBAL INFUSIONS AND COCOA	0,05*
610000	(i) Tea (dried leaves and stalks, fermented or otherwise of Camellia sinensis)	0,05*
620000	(ii) Coffee beans	0,05*
630000	(iii) Herbal infusions (dried)	0,05*
631000	(a) Flowers	0,05*
631010	Camomille flowers	0,05*
631020	Hybiscus flowers	0,05*
631030	Rose petals	0,05*
631040	Jasmine flowers	0,05*
631050	Lime (linden)	0,05*
631990	Others	0,05*
632000	(b) Leaves	0,05*
632010	Strawberry leaves	0,05*
632020	Rooibos leaves	0,05*
632030	Maté	0,05*
632990	Others	0,05*
633000	(c) Roots	0,05*
633010	Valerian root	0,05*
633020	Ginseng root	0,05*
633990	Others	0,05*
639000	(d) Other herbal infusions	0,05*
640000	(iv) Cocoa (fermented beans)	0,05*
650000	(v) Carob (st johns bread)	0,05*
700000	7. HOPS (dried) , including hop pellets and uncoacentrated powder	0,05*
800000	8. SPICES	0,05*
810000	(i) Seeds	0,05*
810010	Anise	0,05*
810020	Black caraway	0,05*
810030	Celery seed (Lovage seed)	0,05*
810040	Coriander seed	0,05*
810050	Cumin seed	0,05*
810060	Dill seed	0,05*
810070	Fennel seed	0,05*
810080	Fenugreek	0,05*
810090	Nutmeg	0,05*
810990	Others	0,05*
820000	(ii) Fruits and berries	0,05*
820010	Allspice	0,05*
820020	Anise pepper (Japan pepper)	0,05*
820030	Caraway	0,05*
820040	Cardamom	0,05*
820050	Juniper berries	0,05*
820060	Pepper, black and white (Long pepper, pink pepper)	0,05*
820070	Vanilla pods	0,05*
820080	Tamarind	0,05*
820990	Others	0,05*
830000	(iii) Bark	0,05*
830010	Cinnamon (Cassia)	0,05*
830990	Others	0,05*
840000	(iv) Roots or rhizome	0,05*
840010	Liquorice	0,05*
840020	Ginger	0,05*
840030	Turmeric (Curcuma)	0,05*
840040	Horseradish	0,05*
840990	Others	0,05*
850000	(v) Buds	0,05*
850010	Cloves	0,05*
850020	Capers	0,05*
850990	Others	0,05*
860000	(vi) Flower stigma	0,05*
860010	Saffron	0,05*
860990	Others	0,05*
870000	(vii) Aril	0,05*
870010	Mace	0,05*
870990	Others	0,05*
900000	9. SUGAR PLANTS	
900010	Sugar beet (root)	0,05*
900020	Sugar cane	0,5
900030	Chicory roots	0,05*
900990	Others	0,05*
1000000	10. PRODUCTS OF ANIMAL ORIGIN-TERRESTRIAL ANIMALS	
1010000	(i) Meat, preparations of meat, offals, blood, animal fats fresh chilled or frozen, salted, in brine, dried or smoked or processed as flours or meals other processed products such as sausages and food preparations based on these	0,1
1011000	(a) Swine	0,1
1011010	Meat	0,1
1011020	Fat free of lean meat	0,1
1011030	Liver	0,1
1011040	Kidney	0,1
1011050	Edible offal	0,1
1011990	Others	0,1
1012000	(b) Bovine	0,1
1012010	Meat	0,1
1012020	Fat	0,1
1012030	Liver	0,1
1012040	Kidney	0,1
1012050	Edible offal	0,1
1012990	Others	0,1
1013000	(c) Sheep	0,1
1013010	Meat	0,1
1013020	Fat	0,1
1013030	Liver	0,1
1013040	Kidney	0,1
1013050	Edible offal	0,1
1013990	Others	0,1
1014000	(d) Goat	0,1
1014010	Meat	0,1
1014020	Fat	0,1
1014030	Liver	0,1
1014040	Kidney	0,1
1014050	Edible offal	0,1
1014990	Others	0,1
1015000	(e) Horses, asses, mules or hinnies	0,1
1015010	Meat	0,1
1015020	Fat	0,1
1015030	Liver	0,1
1015040	Kidney	0,1
1015050	Edible offal	0,1
1015990	Others	0,1
1016000	(f) Poultry -chicken, geese, duck, turkey and Guinea fowl-, ostrich, pigeon	0,1
1016010	Meat	0,1
1016020	Fat	0,1
1016030	Liver	0,1
1016040	Kidney	0,1
1016050	Edible offal	0,1
1016990	Others	0,1
1017000	(g) Other farm animals (Rabbit, Kangaroo)	0,1
1017010	Meat	0,1
1017020	Fat	0,1
1017030	Liver	0,1
1017040	Kidney	0,1
1017050	Edible offal	0,1
1017990	Others	0,1
1020000	(ii) Milk and cream, not concentrated, nor containing added sugar or sweetening matter, butter and other fats derived from milk, cheese and curd	0,1
1020010	Cattle	0,1
1020020	Sheep	0,1
1020030	Goat	0,1
1020040	Horse	0,1
1020990	Others	0,1
1030000	(iii) Birds' eggs, fresh preserved or cooked	0,05*
	Shelled eggs and egg yolks fresh, dried, cooked by steaming or boiling in water, moulded, frozen or otherwise preserved whether or not containing added sugar or sweetening matter	
1030010	Chicken	0,05*
1030020	Duck	0,05*
1030030	Goose	0,05*
1030040	Quail	0,05*
1030990	Others	0,05*
1040000	(iv) Honey (Royal jelly, pollen)	0,05*
1050000	(v) Amphibians and reptiles (Frog legs, crocodiles)	0,05*
1060000	(vi) Snails	0,05*
1070000	(vii) Other terrestrial animal products	0,05*

(*) Indicates lower limit of analytical determination

B.7.13 Proposed EU MRLs and justification for the acceptability of those MRL's (IIA 6.7, IIIA 8.6)

Sufficient data have been submitted to propose an MRL for spinach:

Commodity	Existing EC MRL (mg/kg)	Proposed EC MRL (mg/kg)	Justification for the proposal
Spinach	0.5	30	HR (asulam plus malonyl asulam) = 23 mg/kg Rber = 3 mg/kg Rmax = 22 mg/kg OECD = 30 mg/kg Codex = - mg/kg

Based on the acute intakes, the proposed EU MRL for spinach would not lead to an exceedence of the ARfD.

B.7.14 Proposed EU Import tolerances and justification for the acceptability of those residues

No requests have been made for EU import tolerances for asulam.

B.7.15 Basis for differences, if any, in conclusions reached having regard to established or proposed CAC MRL's

No CAC MRLs are available for asulam.

B.7.16 Estimates of potential and actual dietary exposure through diet and other means (IIA 6.9, IIIA 8.8)

B.7.16.1 Intakes by domestic animals.

Possible intake via consumption of animal products is deemed very unlikely, as only on very rare occasions will animals consume spinach.

B.7.16.2 Intakes by humans

B.7.16.2.1 Theoretical Daily Intakes

Table B.7.36 NEDIs of asulam

Commodity	Residue (mg/kg)	NEDI for Adult (mg/kg bw/day)	NEDI for Infant (mg/kg bw/day)	NEDI for Toddlers (mg/kg bw/day)	NEDI for Children Years 4-6 (mg/kg bw/day)	NEDI for Children Years 7-10 (mg/kg bw/day)	NEDI for Children Years 11-14 (mg/kg bw/day)	NEDI for Children Years 15-18 (mg/kg bw/day)	NEDI for Vegeta-rian (mg/kg bw/day)	NEDI for Elderly (Own home) (mg/kg bw/day)	NEDI for Elderly (Residential) (mg/kg bw/day)
Spinach	0.36	0.00015	0.00036	0.00056	0.00031	0.00031	0.00023	0.00013	0.00025	0.0002	0.00013

The NEDIs for spinach are all below (less than 0.2%) the ADI of 0.36 mg/kg bw/day.

Table B.7.37 TMDIs of asulam – EFSA Model (highest intake group for children and adults)

Crop	MRL (mg/kg)	TMDI children (% of ADI)	TMDI adults (% of ADI)
Spinach	30	5.9 (FR Toddler)	1.2 (NL)

The TMDIs for adults and children are all well below (less than 10%) the ADI of 0.36 mg/kg bw/day.

B.7.16.2.2 Acute exposure

Table B.7.38 NESTIs of asulam

Commodity	Residue (mg/kg)	NESTI for Adult (mg/kg bw/day)	NESTI for Infant (mg/kg bw/day)	NESTI for Toddlers (mg/kg bw/day)	NESTI for Children Years 4-6 (mg/kg bw/day)	NESTI for Children Years 7-10 (mg/kg bw/day)	NESTI for Children Years 11-14 (mg/kg bw/day)	NESTI for Children Years 15-18 (mg/kg bw/day)	NESTI for Vegeta-rian (mg/kg bw/day)	NESTI for Elderly (Own home) (mg/kg bw/day)	NESTI for Elderly (Residential) (mg/kg bw/day)
Spinach	23	0.25	0.6	0.46	0.65	0.37	0.35	0.18	0.41	0.25	0.16

The NESTIs for spinach are all below (less than 70%) the ARfD of 1 mg/kg bw/day.

Table B.7.38a IESTIs of asulam – EFSA Model

Crop	MRL (mg/kg)	IESTI children (% of ARfD)	IESTI adults (% of ARfD)
Spinach	30	68 (BE)	27 (NL)

The IESTIs for spinach are all below (less than 70%) the ARfD of 1 mg/kg bw/day.

B.7.16.2.3 Summary of estimates of potential and actual dietary exposure through diet and other means

Individual TMDIs and IESTIs, NEDIs and NESTIs from the consumption of spinach for adults, infants, toddlers, children, vegetarians and the elderly are all below the ADI and ARfD of 0.36 and 1 mg/kg bw/day respectively. With regards to sulphanilamide (and malonyl sulphanilamide), residues in processed spinach were below the limit of determination (0.01 and 0.05 mg/kg respectively), with the exception of spinach treated at a later grow stage (GS 43 – proposed treatment GS14), which gave residues of up to 0.021 and 0.083 mg/kg respectively, even at these levels the resulting individual TMDIs and IESTIs, NEDIs and NESTIs would be well below (less than 1%) the ADI and ARfD of sulphanilamide of 0.05 and 0.3 mg/kg bw/day respectively.

B.7.17 Summary

The data submitted are sufficient to establish the nature and magnitude of residues in crops and in foodstuffs of animal origin that are likely to result from the proposed use of asulam on spinach. Estimates of animal and human dietary intakes of residues indicate that the risk to consumers is well within acceptable limits.

B.7.18 Literature search

It is considered that the literature searches are acceptable in terms of databases searched and the search criteria applied. On checking the references list, agree with the applicant assessment that the search did not reveal any references of relevance to this section. In addition, the applicant submitted new studies to cover the areas of concern raised in the EFSA conclusion.

B.7.19 References relied on

Active substance

*details to be added during Peer Review

Data point	Author(s)	Year	Title Company name, Report No. Source (where different from company) GLP or GEP status Published or not	Vertebrate Study Y/N	Data protection Y/N	Justification if data protection is claimed	Owner	*Previous evaluation
CA 6.1.1/01	Bardel P.	2005	Storage Stability of asulam in/on Spinach (Leaves) during Freezer Storage for 6 Months at 5.0 mg/kg fortification level Generated by: Bayer CropScience AG, DEU; Bayer CropScience AG, DEU; Document No: **C042938** GLP / GEP Yes unpublished	N	Y	Data protection is claimed in accordance with Article 59 of Regulation (EC) No 1107/2009	UPL	
CA 6.1.1/04	Bardel P.	2004b	Storage Stability of Malonyl-asulam in/on Spinach (Leaves) during Freezer Storage for 11 Months at 0.5 mg/kg Fortification Level Code: AE 1388213 Generated by: Bayer CropScience AG, DEU; Bayer CropScience AG, DEU; Document No: **C042942** GLP / GEP Yes unpublished	N	Y	Data protection is claimed in accordance with Article 59 of Regulation (EC) No 1107/2009	UPL	
CA 6.1/04	McEwen A., Sur R., Miebach D.	2003a	(14C)-Asulam: Metabolism in spinach Generated by: BioDynamics Research Ltd., Northamptonshire, GBR; Bayer CropScience S.A., Lyon, FRA; Document No: **C032013** GLP / GEP Yes unpublished	N	Y	Data protection is claimed in accordance with Article 59 of Regulation (EC) No 1107/2009	UPL	

Data point	Author(s)	Year	Title Company name, Report No. Source (where different from company) GLP or GEP status Published or not	Vertebrate Study Y/N	Data protection Y/N	Justification if data protection is claimed	Owner	*Previous evaluation
CA 6.1.1/03	Sur R.	2004	Metabolism of (phenyl-UL-14C) asulam in spinach after post-emergence application Generated by: Bayer CropScience AG, DEU; Development-Metabolism & Environmental Fate Document No: **C043598** GLP / GEP Yes unpublished	N	Y	Data protection is claimed in accordance with Article 59 of Regulation (EC) No 1107/2009	UPL	
CA 6.2.1/01	Roohi, A.	2008	[14C]-Asulam: Metabolism, Distribution and Expression of Residue in Spinach Following Pre-emergence Application United Phosphorus Limited Report No.: OU/07/013 Battelle UK Limited, United Kingdom GLP, Unpublished	N	Y	Data protection is claimed in accordance with Article 59 of Regulation (EC) No 1107/2009	UPL	
CA, 6.1.2/01	Piskorski, R.	2012	[14C]-Asulam: Metabolism in Spinach United Phosphorus Limited Report No.: 20110093 Innovative Environmental Services Ltd, Switzerland GLP, Unpublished	N	Y	Data protection is claimed in accordance with Article 59 of Regulation (EC) No 1107/2009	UPL	
CA 6.2.1/03	Lowden, P. and Peatman, M.	2014	The Non-relevance of 'Asulam Dimers' in Asulam Spinach Metabolism Studies Conducted to GAP United Phosphorus Limited Report No. NZ/13/017A Battelle UK Ltd., United Kingdom and JSC International Limited, United Kingdom Not GLP, Unpublished	N	Y	Data protection is claimed in accordance with Article 59 of Regulation (EC) No 1107/2009	UPL	

Data point	Author(s)	Year	Title Company name, Report No. Source (where different from company) GLP or GEP status Published or not	Vertebrate Study Y/N	Data protection Y/N	Justification if data protection is claimed	Owner	*Previous evaluation
CA 6.2.3/01	[redacted]	1990b	A metabolism study with 14C-asulam in dairy goats (Capra hircus) [redacted] GLP / GEP Yes unpublished	N	Y	Data protection is claimed in accordance with Article 59 of Regulation (EC) No 1107/2009	UPL	
CA 6.2.2/01	[redacted]	1990a	A metabolism study with 14C-asulam in laying hens (Gallus gallus) [redacted] GLP / GEP Yes unpublished	N	Y	Data protection is claimed in accordance with Article 59 of Regulation (EC) No 1107/2009	UPL	
CA 6.3.2/02	Bardel P.	2004a	Determination of the residues of asulam in/on spinach after spraying of AE F074383 00 SL33 A1 (400 SL) in Spain Generated by: Bayer CropScience AG, DEU; Bayer CropScience AG, DEU; Document No: **C042944** GLP / GEP Yes unpublished	N	Y	Data protection is claimed in accordance with Article 59 of Regulation (EC) No 1107/2009	UPL	
CA, 6.3.2/03	Bardel P.	2004d	Determination of the residues of Asulam in/on spinach after spraying of AE F074383 00 SL33 A1 (400 SL) in Great Britain, Belgium and Germany Generated by: Bayer CropScience AG, DEU; Document No: **C042946** GLP / GEP Yes unpublished	N	Y	Data protection is claimed in accordance with Article 59 of Regulation (EC) No 1107/2009	UPL	

Data point	Author(s)	Year	Title Company name, Report No. Source (where different from company) GLP or GEP status Published or not	Vertebrate Study Y/N	Data protection Y/N	Justification if data protection is claimed	Owner	*Previous evaluation
CA 6.3.2/01	Bardel P., Oel D.	2004a	Determination of the residues of asulam in/on spinach after spraying of AE F074383 00 SL33 A1 (400 SL) in the field in Germany and the Netherlands Generated by: Bayer CropScience AG, DEU; Bayer CropScience AG, DEU; Document No: **C032852** GLP / GEP Yes unpublished	N	Y	Data protection is claimed in accordance with Article 59 of Regulation (EC) No 1107/2009	UPL	
CA 6.3.2/04	Bardel P.	2004f	Determination of the residues of asulam in / on spinach after spraying of AE F074383 00 SL33 A1 (400 SL) in France Generated by: Bayer CropScience AG, DEU; Bayer CropScience AG, DEU; Document No: **C043215** GLP / GEP Yes unpublished	N	Yes	Data protection is claimed in accordance with Article 59 of Regulation (EC) No 1107/2009	UPL	
CA 6.3.1/01	van de Sandt, H.J.	2008	Determination of the decline and magnitude of the residues of asulam and malonyl asulam in/on spinach after one soil application with Asulam 400 g/l SL to spinach in the Netherlands, 2008 + Amendment No. 1 United Phosphorus Limited Report No.: R08-16-NL-09 Research Company for Plant Protection De Bredelaar BV, The Netherlands GLP, Unpublished	N	Y Y	Data protection is claimed in accordance with Article 59 of Regulation (EC) No 1107/2009	UPL	

Data point	Author(s)	Year	Title Company name, Report No. Source (where different from company) GLP or GEP status Published or not	Vertebrate Study Y/N	Data protection Y/N	Justification if data protection is claimed	Owner	*Previous evaluation
CA 6.3.1/02	van de Sandt, H.J.	2009	Determination of the decline and magnitude of the residues of asulam and malonyl asulam in/on spinach after one soil application with Asulam 400 g/l SL to spinach in the Netherlands, 2008 United Phosphorus Limited Report No.: R08-16-NL-08 Research Company for Plant Protection De Bredelaar BV, The Netherlands GLP, Unpublished	N	Y	Data protection is claimed in accordance with Article 59 of Regulation (EC) No 1107/2009	UPL	
CA, 6.3.1/03	Boileau, G.	2013a	Magnitude of the residues of asulam and its metabolites in spinach (RAC leaves), following one pre-emergence application of Asulox, in 4 harvest trials, northern Europe (northern France and the United Kingdom) – 2013 United Phosphorus Limited Report No.: GBU-13-16062 Staphyt, France GLP, Unpublished	N	Y	Data protection is claimed in accordance with Article 59 of Regulation (EC) No 1107/2009	UPL	
CA 6.3.2/05	Boileau, G.	2013b	Magnitude of the residues of asulam and its metabolites in spinach (RAC leaves and processed fractions), following one post-emergence application of Asulox, in 5 trials (2 harvest and 3 decline), northern Europe (northern France and the United Kingdom) – 2013 United Phosphorus Limited Report No.: GBU-13-16064 Staphyt, France GLP, Unpublished	N	Y	Data protection is claimed in accordance with Article 59 of Regulation (EC) No 1107/2009	UPL	

Data point	Author(s)	Year	Title Company name, Report No. Source (where different from company) GLP or GEP status Published or not	Vertebrate Study Y/N	Data protection Y/N	Justification if data protection is claimed	Owner	*Previous evaluation
CA, 6.5.1/01	Fitzmaurice M.J.	2003a	[14C]-Asulam: Nature of the Potential Residue in the Products of Industrial Processing or Household Preparation Generated by: Battelle AgriFood Ltd, UK; Fyfield Business and Research Park Bayer CropScience AG, DEU; Development Document No: **C033605** GLP / GEP Yes unpublished	N	Y	Data protection is claimed in accordance with Article 59 of Regulation (EC) No 1107/2009	UPL	
CA 6.5.1/02	Fitzmaurice M.J.	2003b	[14C]-AE 1388213: Nature of the Potential Residue in the Products of Industrial Processing or Household Preparation. Generated by: Battelle AgriFood Ltd, UK; Fyfield Business and Research Park Bayer CropScience AG, DEU; Development Document No: **C034761** GLP / GEP Yes unpublished	N	Y	Data protection is claimed in accordance with Article 59 of Regulation (EC) No 1107/2009	UPL	
CA 6.5.3/01	Bardel P., Hoffmann M.	2004a	Determination of the residues of Asulam in/on spinach in processing products from spinach (leaves, cooking leaves, cooking water, washing water) after spraying of AE F074383 00 SL33 A1 (400 SL) in Germany Generated by: Bayer CropScience AG, DEU; Bayer CropScience AG, DEU; Document No: **C042948** GLP / GEP Yes unpublished	N	Y	Data protection is claimed in accordance with Article 59 of Regulation (EC) No 1107/2009	UPL	

Data point	Author(s)	Year	Title Company name, Report No. Source (where different from company) GLP or GEP status Published or not	Vertebrate Study Y/N	Data protection Y/N	Justification if data protection is claimed	Owner	*Previous evaluation
CA 6.5.3/02	Bardel P., Hoffmann M.	2004b	Determination of the residues of asulam in/on spinach in processing products from spinach (leaves, cooking leaves, cooking water, washing water) after spraying of AE F074383 00 SL33 A1 (400 SL) in the field in Germany Generated by: Bayer CropScience AG, DEU; Bayer CropScience AG, DEU; Document No: **C043216** GLP / GEP Yes unpublished	N	Y	Data protection is claimed in accordance with Article 59 of Regulation (EC) No 1107/2009	UPL	
CA 6.5.3/03	Boileau, G.	2013b	Magnitude of the residues of asulam and its metabolites in spinach (RAC leaves and processed fractions), following one post-emergence application of Asulox, in 5 trials (2 harvest and 3 decline), northern Europe (northern France and the United Kingdom) – 2013 United Phosphorus Limited Report No.: GBU-13-16064 Staphyt, France GLP, Unpublished ⇒ CA 6.3.2/05	N	Y	Data protection is claimed in accordance with Article 59 of Regulation (EC) No 1107/2009	UPL	
CA 6.6.2/01	Jesudason, P.A.	1996	A Confiined Rotational Crop Study with 14C-Asulam Using Radishes (Raphanus sativus), Spinach (Spinacia oleracea), and Wheat (Triticum aestivum) Generated by: Rhone-Poulenc; Rhone-Poulenc Ag Company, USA; Document No: **R003651** GLP / GEP Yes unpublished	N	Y	Data protection is claimed in accordance with Article 59 of Regulation (EC) No 1107/2009	UPL	

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None

B.7.20 References used in assessment of data

1. Gregory, J., Foster, K., Tyler, H. and Wiseman, M. (1990). The Dietary and Nutritional Survey of British Adults. HMSO.

2. Report on Health and Social Subjects, No. 36 : 'The Diets of British Schoolchildren'. HMSO.

3 Mills, A and Tyler, H. (1992). Food and Nutrient Intakes of British Infants Aged 6-12 months. HMSO.

4. Rees, N and Harris, C. (1995). UK Methods for the Estimation of Dietary Intakes of Pesticide Residues (unpublished)