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Department of Food, Nutrition, and Veterinary Public Health
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ITALY**

**Monograph prepared in the context of the inclusion of the following
active substance in Annex I of the Council Directive 91/414/EEC**

Bensulfuron Methyl

Volume 3

Annex B

**Summary, Scientific
Evaluation and Assessments**

May 2006

ANNEX B

Active substance: Bensulfuron Methyl

B.1 Identity

WARNING: This document forms part of an EC evaluation data package and should not be read in isolation. Registration must not be granted on the basis of this document.

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Identity

B.1. Identity

B.1.1 Identity of the active substance (Annex IIA 1 and 3)

B.1.1.1 Applicant (name, address, etc.)

Applicant:

[illegible]

B.1.1.2 **Manufacturer (name, address, including location of plant)**

[illegible]

B.1.1.3 Common name proposed or ISO-accepted, and synonyms

Bensulfuron methyl (BSI and ISO approved)

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B.1.1.4 Chemical name (IUPAC nomenclature)

IUPAC:

METHYL α -(4,6-DIMETHOXYPYRIMIDIN-2-YLCARBAMOYLSULFAMOYL)-O-TOLUATE

CAS:

METHYL 2-[[[(4,6-DIMETHOXY-2- pyrimidinyl)amino]carbonyl]amino]sulfonyl]methyl]benzoate

B.1.1.5 Manufacturers development code number

DPX-F5384

F5384

B.1.1.6 CAS and EEC numbers (if available)

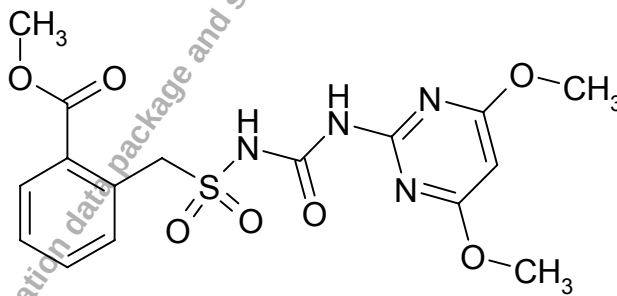
CAS Registry No.: 83055-99-6

EEC No.: 401-340-6

EINECS, ELINCS: See EEC number above

B.1.1.7 Empirical and structural formula, molecular massa. Empirical Formula: $C_{16}H_{18}N_4O_7S$

b. Structural Formula:

Figure B.1 1: Structural formula of bensulfuron methyl

c. Molecular Mass: 410.4 g/mol

B.1.1.8 Method of manufacture (synthesis pathway) of the active substance

Details of the method of manufacture of the active substance are given in Annex C. The applicant points out that information pertaining to the process description is highly confidential and requests it to be treated as such according to Article 14 of Council Directive 91/414/EEC.

B.1.1.9 Specification of purity of the active substance in g/kg or g/l as appropriate

Production scale: Minimum purity of bensulfuron methyl is 975 g/kg. Information is provided in Annex C.

Identity

There are neither additives nor isomers in bensulfuron methyl technical. A list of impurities with their maximum values is provided in Annex C.

Analytical profiles of commercial production batches together with those for the samples used in the main toxicological studies can be found in Annex C.

Postemergence, selective herbicide acting primarily through foliar uptake with little soil activity.

B.1.2.1 Applicant (name, address, etc.)

[illegible]

[REDACTED]

Identity

100

It has been found that the following factors are important in determining the rate of change in the population:

GAO 11-111

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Detailed information on the composition of the preparation is provided in Annex C which contains industrial and commercial data for which confidentiality is requested in accordance with Article 14 of Directive 91/414/EEC.

B.1.2.5 Physical state and nature of the preparation

Water Dispersible Granule (WG).

B.1.2.6 Use category (herbicide, insecticide, etc.)

Selective herbicide.

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B.1.3 Reference relied on

Annex No., EU Data Requirement No., Reference No.	Author(s)	Year	Title Source Company Report No. GLP or GEP Status (where relevant) Published or not	EU Data Protection Claimed (Y/N)	Owner
IIA, 1.8./01 IIA, 1.9./01 IIA, 1.10./01	Lan, J.X.	2002	Technical grade bensulfuron methyl analysis and certification of product ingredients - EU submission DuPont Stine-Haskell Research Center DuPont-8296 GLP: No Published: No	Y	DuPont
IIA, 1.9./02 IIA, 1.10./02 IIA, 1.11./01	Tseng, P. K.	1995	Technical grade bensulfuron methyl analysis and certification of product ingredients DuPont Experimental Station AMR 3139-94 GLP: Yes Published: No	Y	DuPont
IIA, 1.9./03 IIA, 1.10./03 IIA, 1.11./02	Morgan, E.A.	2002	Bensulfuron methyl (DPX-F5384): analysis and certification of product ingredients for technical grade material produced at the manati and Shanghai manufacturing facilities DuPont Stine-Haskell Research Center and Chambers Works DuPont-7095 GLP: Yes Published: No	Y	DuPont

ANNEX B

Active substance: Bensulfuron Methyl

B.2 Physical and chemical properties

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B.2 Physical and chemical properties

B.2.1 Physical and chemical properties of the active substance (Annex IIA 2)

Test or Study & Annex point	Guideline and method	Test material purity and specification	Findings	Comments	GLP	Reference
B.2.1.1 Melting point and boiling point						
<i>B.2.1.1.1 Melting, freezing or solidification point</i>	EEC A.1 OECD 102 EPA 830.7200 1. Method: Apparatus with photocell detection 2. Method: Differential Scanning Calorimetry (DSC)	1. DPX-F5384-328 98.3% purity 2. DPX-F5384-328 98.3% purity	1. The melting point range of bensulfuron methyl was determined to be $179.4 \pm 0.15^{\circ}\text{C}$ (452.6 K) using the Mettler FP900 Thermosystem, with the determination based on the Thiele Tube technique. 2. DSC was used to determine the thermal stability. In three DSC trials, a clearly defined endotherm indicates melting at 190°C .	Measurements were made in two different laboratories in U.S. and Ireland, using photocell detection and DSC respectively (both with estimated accuracy of ± 0.5). Compared to the photocell detection, DSC determination gave a melting point 10°C higher. The applicant claims the results from the test using the Mettler FP900 (179.4°C) as definitive. The 10°C difference is explained possibly to be due to different atmospheric pressures.	Y	Huntley, 2000
<i>B.2.1.1.2 Boiling point</i>	-	NA	Not applicable; the test material is a solid which decomposes after melting.	Accepted	NA	NA
<i>B.2.1.1.3 Decomposition or sublimation temperature</i>	EEC A.1. Method: Differential Scanning Calorimetry	DPX-F5384-328 98.3% purity	Bensulfuron methyl decomposes after melting at approximately 245°C (518 K).	Result accepted	Y	Huntley, 2000

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Test or Study & Annex point	Guideline and method	Test material purity and specification	Findings	Comments	GLP	Reference
B.2.1.2 Relative density						
<i>B.2.1.2.1 Relative density of purified active substance</i>	EEC A.3. OECD 109 EPA 830.7300 Method: Pressure cell gas pycnometer	DPX-F5384-328 98.3% purity	The relative density of bensulfuron methyl was measured in triplicate with an AccuPyc 1330 gas pycnometer and determined to be 1.4862 ± 0.0002 g/cm ³ (1486 kg/m ³), at an average recorded temperature of $19.7 \pm 0.2^\circ\text{C}$. The instrument determines the volume of a known mass of test substance in air, by measuring the pressure change, given by a 0-30 psi pressure transducer in the calibrated volume of a pressure chamber cell.	Acceptable study	Y	Huntley, Edgar, 2000

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Test or Study & Annex point	Guideline and method	Test material purity and specification	Findings	Comments	GLP	Reference
B.2.1.3 Vapour pressure and volatility						
<i>B.2.1.3.1 Vapour pressure of purified active substance</i>	EPA 63-9 Method: Effusion method – “Knudsen method” (extrapolating from high temperatures)	DPX-F5384-83 99.3% purity	The vapor pressure of bensulfuron methyl at 25°C was determined to be 2.1×10^{-14} torr (2.8×10^{-12} Pa). Loss of the compound from a Knudsen cell was measured gravimetrically at five temperatures from 160.8 to 198°C. The value of the vapor pressure at 25°C was obtained by extrapolation of the high temperature data, applying the following experimental equation: $\ln(vp) = -20.983 \times 1000/T + 38.906$ where T is in Kelvin. Conclusion: Volatilisation is not expected to contribute to a significant extent to the dissipation of bensulfuron methyl in the environment.	Acceptable study	N	Schmuckler, 2001
<i>B.2.1.3.2 Henry's law constant at 25 °C</i>	Method: Calculation	NA	6×10^{-10} Pa x m ³ x mol ⁻¹ at pH 5 2×10^{-11} Pa x m ³ x mol ⁻¹ at pH 7 4×10^{-13} Pa x m ³ x mol ⁻¹ at pH 9	Accepted	NA	Schmuckler, 2000

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Test or Study & Annex point	Guideline and method	Test material purity and specification	Findings	Comments	GLP	Reference
B.2.1.4 Appearance (physical state, colour and odour)						
<i>B.2.1.4.1 Physical state, colour</i>	Visual assessment ASTM Method D1535-89	DPX-F5384-328 98.3% purity	Bensulfuron methyl is a white solid (at 22°C). The colour of bensulfuron methyl was compared against the Munsell Color System against both white and black backgrounds. The color is described in terms of value, hue and chroma: bensulfuron methyl was determined to have a value of 9, a hue of 7.5Y and a chroma of 1 at 20°C.	Accepted	Y	Huntley, Edgar, 2000
<i>B.2.1.4.2 Odour</i>	Organoleptic Testing	DPX-F5384-328 98.3% purity	Bensulfuron methyl was characterised as odourless at 23°C.	Accepted	Y	Huntley, Edgar, 2000

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Test or Study & Annex point	Guideline and method	Test material purity and specification	Findings	Comments	GLP	Reference
B.2.1.5 Spectra (UV/Vis, IR, NMR, MS), molecular extinction at relevant wavelengths						
<i>B.2.1.5.1 Spectra, active substance</i>						
UV/Visible spectrum:	OECD 101 EPA 830.7050	DPX-F5384-328 98.3% purity	The UV/Visible spectra at 25°C of methanolic buffer solutions (KH₂PO₄/HCl) at pH 1.91, 7.02, and 12.15, were measured with a Diode Array, UV/visible spectrophotometer. Absorbance maxima of bensulfuron methyl are: pH 1.91: 200nm (logϵ = 4.72) and 236nm (logϵ = 4.13) pH 7.02: 200nm (logϵ = 4.76) and 239nm (logϵ = 4.42) pH 12.15: 210nm (logϵ = 4.47) and 240nm (logϵ = 4.44). At 290 nm the molar extinction coefficient (ϵ) in neutral methanolic buffer (pH 7.06) was $\epsilon = 784 \text{ L} \times \text{mol}^{-1} \times \text{cm}^{-1}$ (see Errore. Il risultato non è valido per una tabella.). No absorbance maxima beyond 290 nm were observed at any pH for aqueous solutions of bensulfuron methyl, at concentrations of 11.88 µg/mL in cell path lengths of 10mm and 100mm (see Figure B.2.2).	Bensulfuron methyl shows little or no absorption in the region above 290 nm. Acceptable study	Y	Jeffery, 2000

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Test or Study & Annex point	Guideline and method	Test material purity and specification	Findings	Comments	GLP	Reference
Infrared Spectrum:	No guidelines are specified	DPX-F5384-237 99.3% purity	The IR spectrum for bensulfuron methyl showed key bands consistent with the structure. The IR spectrum was obtained using a BioRad FT-IR spectrometer, FTS 60 and a KBr pellet. The spectrum was obtained by adding 64 scans at 4 cm ⁻¹ resolution, using a TGS detector (Figure B.2 3). Absorption bands and assignments (in parentheses) are given in Table B.2 1 below.	Accepted	N	Schmuckler, 2000
NMR:	No guidelines are specified	DPX-F5384-237 99.3% purity	The ¹ H NMR spectrum (deuterated DMSO) and the proton chemical shift assignments are shown in Figure B.2 4 and Table B.2 2, respectively. The ¹⁴ C NMR spectrum (deuterated acetonitrile) and chemical shifts are shown in Figure B.2 5 and Table B.2 3, respectively. The spectra obtained are consistent with the structure of bensulfuron methyl.	Accepted	N	Schmuckler, 2000

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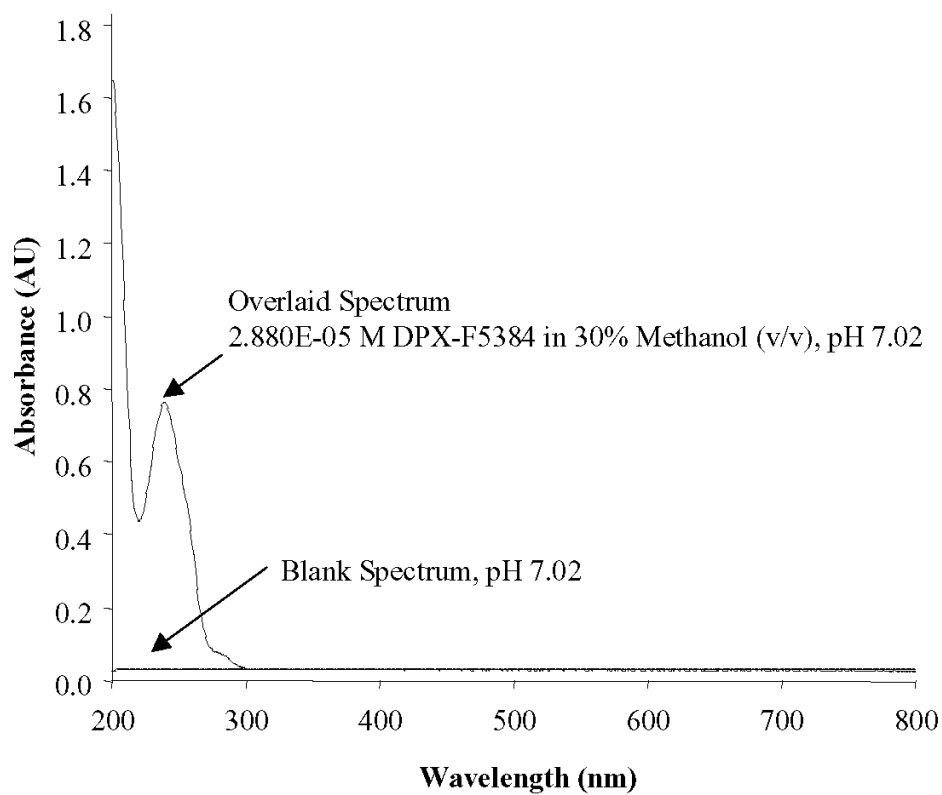
Chemical properties

Test or Study & Annex point	Guideline and method	Test material purity and specification	Findings	Comments	GLP	Reference
Mass Spectrum:	No guidelines are specified	DPX-F5384-237 98.3% purity	The mass spectrum of bensulfuron methyl was obtained by Desorption Chemical Ionisation (DCI) with CH ₄ as a reagent gas (probe) and shows the [M + H] ⁺ ion at m/z = 411, consistent with bensulfuron methyl, as shown in Figure B.2.6	Accepted	N	Schmuckler, 2000
B.2.1.5.2 Spectra, impurities			None of the impurities present in the active substance as manufactured is considered of toxicological or environmental significance.	The toxicological and environmental relevance of impurity and metabolite IN-J290 (pyrimidine amine) is under discussion.		

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Figure B.2 1: UV/Visible spectrum of bensulfuron methyl in neutral solution with 10 mm cell



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Figure B.2 2: UV/Visible spectrum of bensulfuron methyl in aqueous/methanol solution using a 100 mm flow cell

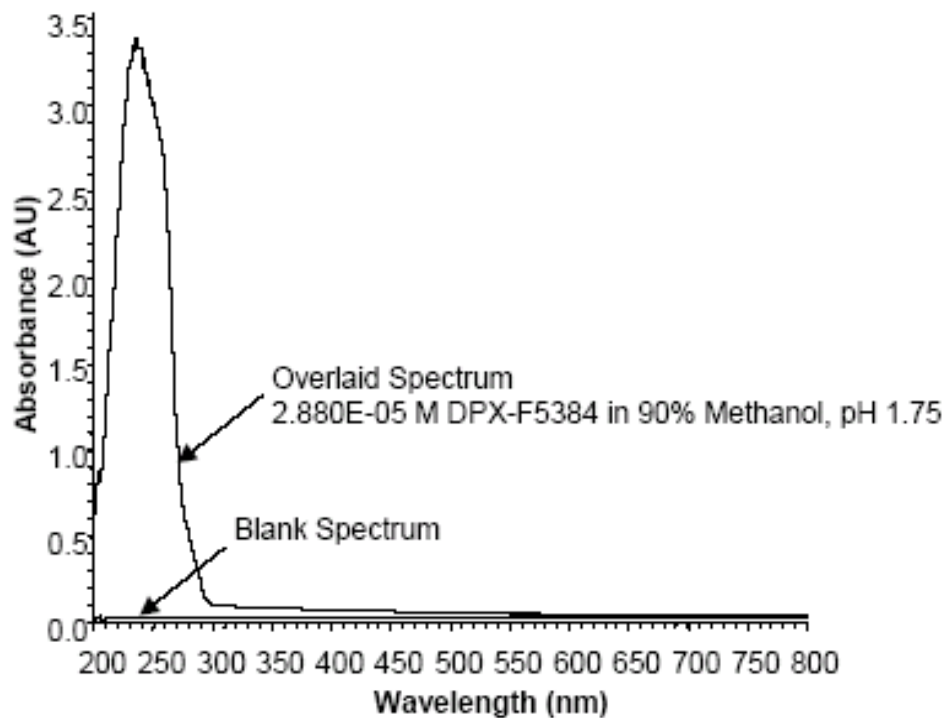
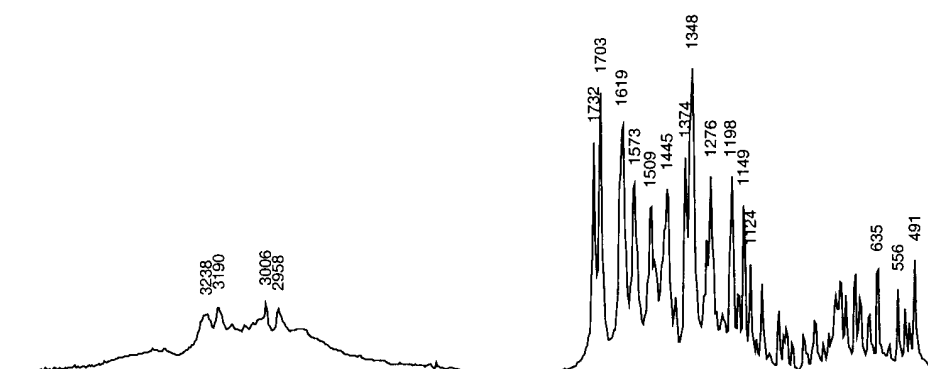


Figure B.2 3: IR Spectrum of bensulfuron methyl



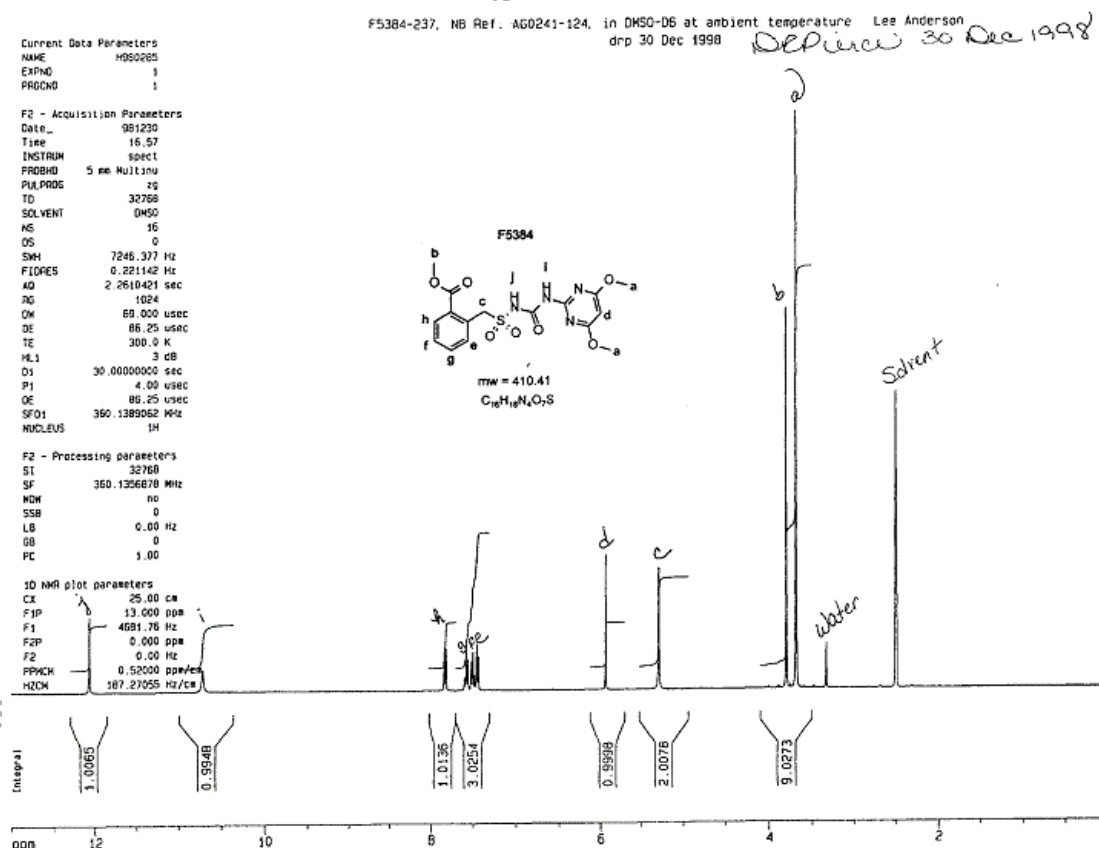
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Table B.2 1: Infrared Absorption Band Assignments for Bensulfuron Methyl

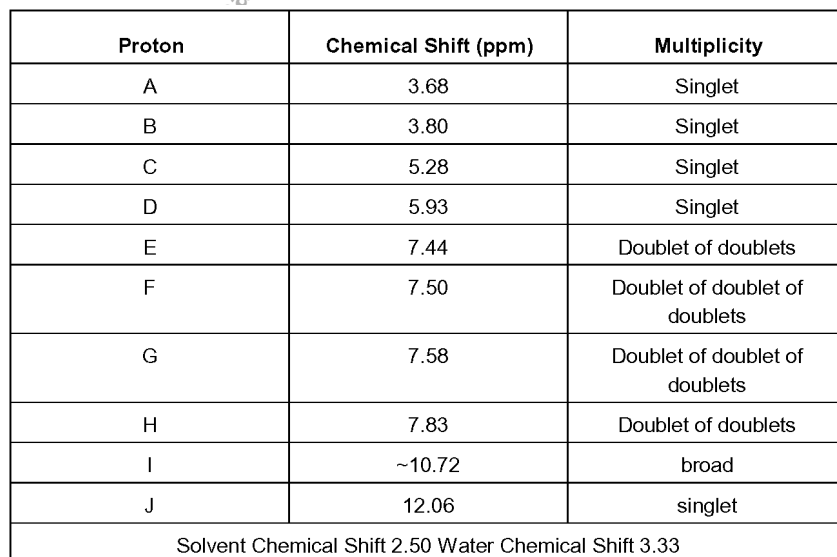
Functional Group	Bond	Range	Intensity	Mode	Absorption Bands in Spectrum
urea	C=O	1705-1635	strong	stretch	1703 cm ⁻¹
	N-C-N	1490-1465	strong	asymmetric stretch	1445 cm ⁻¹
	CO-NH	~1600-1515	medium	deformation (amide II)	1573 cm ⁻¹
	N-H	3300-3200	medium	stretch	3236+3188 cm ⁻¹
SO ₂	SO ₂	1335-1325	very strong	asymmetric stretch	1348 cm ⁻¹
	SO ₂	1160-1150	strong	symmetric stretch	1198 cm ⁻¹
	SO ₂	810-545	medium	SO ₂ scissoring	835 + 556 cm ⁻¹
ester	C=O	1740-1705	strong	stretch	1732 cm ⁻¹
	C-O-C	1310-1250	strong	asymmetric stretch	1276 cm ⁻¹
	C-O-C	1150-1100	strong	symmetric stretch	1149 cm ⁻¹
aromatic structures	ring	1620-1585	medium	ring deformation	1619 cm ⁻¹
	ring	1510-1470	medium	ring deformation	1509 cm ⁻¹

Figure B.2 4: Nuclear Magnetic Resonance diagram of bensulfuron methyl



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Table B.2 2: Chemical shifts of protons in the NMR spectrum of bensulfuron methyl



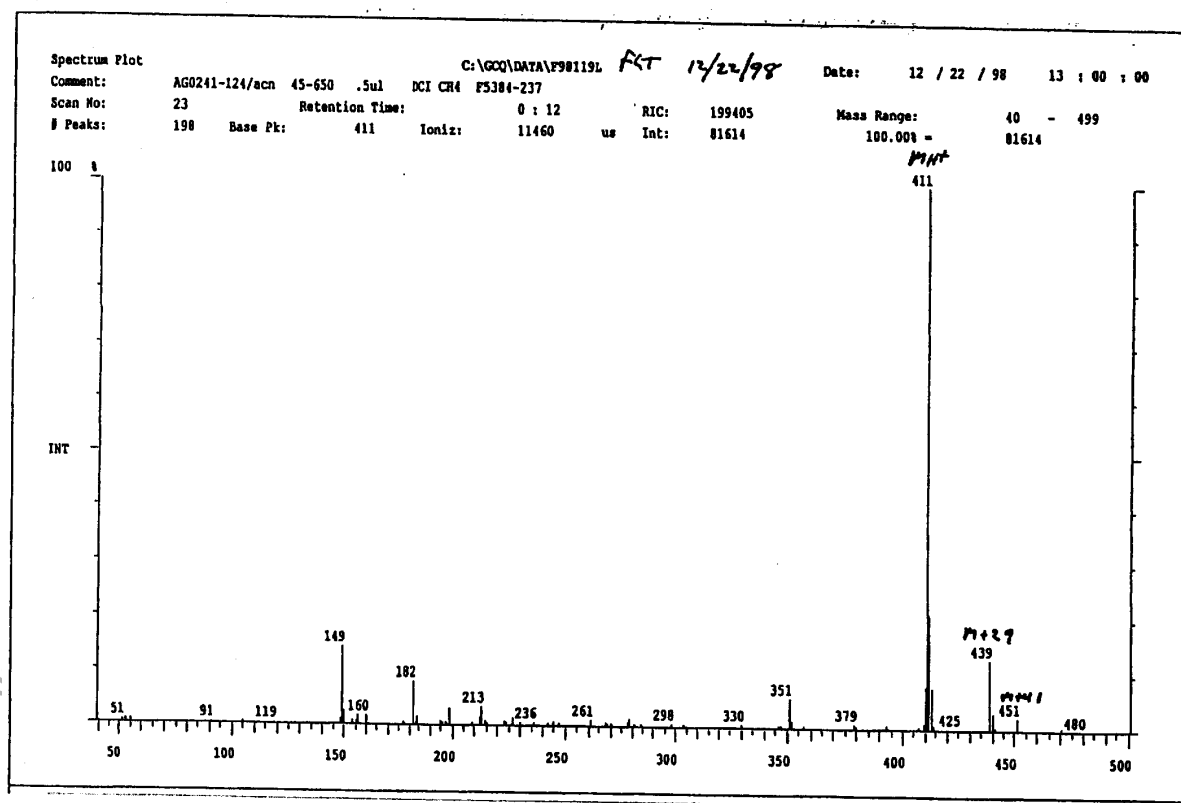
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Table B.2 3: Chemical shifts in the carbon NMR spectrum of bensulfuron methyl

Carbon	Chemical Shift (ppm)
1	52.71
2	168.17
3	129.75
4	129.88
5	132.85
6	131.45
7	134.01
8	132.39
9	56.14
10	156.60
11	150.24
12	172.27
13	84.62
14	55.03
Solvent Chemical Shift	118.00, 1.034

Figure B.2 6: Mass spectrum diagram of bensulfuron methyl



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Table B.2 4: Summary of water solubility of bensulfuron methyl at different pH values

Test pH (Temp.)	Solubility (mg/L)	
	Mean	Range
Un-buffered Distilled (20°C)	6.65	6.09 – 6.96
4.99 (25°C)	2.1	1.99 - 2.22
7.06 (25°C)	67	61.45 - 70.69
9.00 (25°C)	3100	2902 – 3341

Table B.2 5: Solubilities of bensulfuron methyl in various organic solvents at 20°C

Solvent	Solubility (g/L)
	mean
Acetone	5.10
Acetonitrile	3.75
Dichloromethane	18.4
Dimethylformamide	70.8
Ethyl Acetate	1.75
n-Heptane	3.62×10^{-4}
Methanol	0.765
1-Octanol	7.47×10^{-2}
o-Xylene	0.229

Table B.2 6: Summary of log octanol:water partition coefficients of bensulfuron methyl at 25°C and different pH values

pH	Log ₁₀ K _{ow} (S.D.)
5.0	2.18
7.0	0.79
9.0	- 0.99

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B.2.1.6 Solubility in water and effect of pH	1. EPA 63-8 Method: Flask method/HPLC	1. DPX-F5384-83 99.5% purity	1. The solubilities of bensulfuron methyl at 25°C were determined in aqueous buffers at pH 4.99, 7.06 and 9.00 and resulted in: pH 5: 2.1 mg/L, sampling over 25h, pH 7: 67 mg/L, sampling over 30h, pH 9: 3100 mg/L, sampling over 8d	1. Hydrolysis of bensulfuron methyl is supposed not to affect study results, with hydrolytic half-lives of 6 days at pH 4 and of >200 days at pH 7 and 9. pH was measured only once in freshly prepared stock solutions, rather than on each sample as required by EEC A6. Anyhow, the buffer concentration (0.1 M) was selected to prevent any change in pH as a result of dissolution of bensulfuron methyl. Acceptable study	1. Y	1. Moore, 1994
	2. EEC A.6. OECD 105 EPA 830.7840 Method: Flask method/HPLC	2. DPX-F5384-328, 99.3% purity Preparation of LC standards: DPX-F5384-237, 99.3% purity	2. The solubility of bensulfuron methyl in un-buffered distilled water was determined to be 6.65 mg/l at 20°C. Water solubility data are resumed in Table B.2 4.	2. No pH was determined of a.s. in un-buffered distilled water. Anyhow, the pH of the test a.s. in un-buffered distilled water would be close to its pKa value of 5.2. Acceptable study Concluding comment: Bensulfuron methyl is a weak acid (acidic hydrogen on the nitrogen adjacent to the sulfonyl group) with a pKa of 5.2. In aqueous media it exists as an equilibrium mixture of the neutral undissociated form and the negatively charged dissociated form: $R_1-SO_2-NH-CO-NH-R_2 \leftrightarrow [R_1-SO_2-N-CO-NH-R_2]^- + H^+$	2. Y	2. Jeffery, 2000

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				As a consequence pH encountered in the environment (e.g. rice paddies) could affect the mobility of the a.s..		
B.2.1.7 Solubility in organic solvents	Method: Flask method/HPLC	DPX-F5384-328 98.3% purity	Replicate saturated solutions of bensulfuron methyl in nine organic solvents were equilibrated at 20°C. Aliquots were taken at three or more consecutive sampling times and were analyzed by high performance liquid chromatography using UV absorption detection at 254 nm. Test results are given in Table B.2 5.	Acceptable study	Y	Jeffery, 2000
B.2.1.8 Partition coefficient n-octanol/water	EPA 63-11 Method: modified 'shake flask' method/HPLC	DPX-F5384-83 99.5% purity	The study determined the n-octanol/water partition coefficient (K _{ow}) of bensulfuron methyl at two concentrations, respectively 0.02 mg/mL and 0.2 mg/mL, and three pHs: pH 5, pH 7, and pH 9. Results are given in Table B.2 6 and indicated that at any of the test pH, 5, 7 or 9, the K _{ow} is not dependent on the concentration of bensulfuron methyl in either phase of the solvent system. Conclusion: The bioaccumulation potential of bensulfuron methyl in living tissues or other organic matter is moderate to low.	Results at pH 5 (pK _a =5.2) are supposed to be affected by deviations from the Nernst partition law, the extent of ionization being $\alpha = 0.4$. On the other, hand a lower experimental pH would not be significant for the environment. Since these data should be relevant for assessing the potential for bioconcentration and uptake under typical environmental conditions, the study is considered acceptable. Acceptable study	Y	Barefoot, 1989

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Test or Study & Annex point	Guideline and method	Test material purity and specification	Findings	Comments	GLP	Reference
B.2.1.8.1 <i>Partition coefficient n-octanol/water of impurities</i>	Calculation	NA	1. A Log Octanol-Water Partition Coefficient Program (KOWWIN) was used to calculate the log K_{ow} of impurity IN-J290 (pyrimidine amine) given as log K_{ow} = 0.95, 2. The calculated log K_{ow} for impurity IN-N5279 (sulphonamide) is 0.47.	These calculation results are considered indicative and presumably highly dependent on pH.	NA	1. Schmuckler, 2001a 2. Schmuckler, 2001b

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B.2.1.9 Stability in water, hydrolysis rate, photochemical degradation, quantum yield and identity of breakdown product(s), dissociation constant including effect of pH (4 to 9)

Two radiolabeled forms of bensulfuron methyl were used to evaluate the fate of the active substance in the environment. Detailed summaries of the hydrolysis and aqueous photolysis studies are presented in Bensulfuron methyl EU Dossier, Annex IIA, Document M-II, Section 5, DuPont-6185 EU, Points 7.2.1.1 and 7.2.1.2. Below are short summaries of the results obtained in these two studies (points 2.9.1 and 2.9.2).

WARNING: This document forms part of an EC evaluation data package and should not be read in isolation. Registration must not be based on the basis of this document.

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B.2.1.9.1 <i>Hydrolysis rate</i>	OECD 111 EPA 161-1	[Pyrimidinyl-2- ¹⁴ C] bensulfuron methyl, 53.1 µCi/mg, radiochemical purity ≥ 95% [Phenyl(U)- ¹⁴ C] bensulfuron methyl, 57.5 µCi/mg; radiochemical purity ≥ 95%	Buffer solutions prepared at pH 4, 7 and 9 were treated with bensulfuron methyl 14C-labeled in the pyrimidinyl ring or in the phenyl ring (ca. 1 µg/mL) and were incubated for 30 days at 25 ± 1 °C in the dark. The target test substance concentration for this study was approximately 1.0 µg/mL. At selected intervals, samples were analyzed directly by HPLC with radiochemical flow detection (RAD) to determine the distribution of radioactivity. Hydrolysis half-lives of bensulfuron methyl at 25°C were 6 days at pH 4 and 141 days at pH 9. Bensulfuron methyl was essentially stable at pH 7. Pyrimidine amine (IN-J290) and sulphonamide (IN-N5297) were the major hydrolysis products at pH 4 and pH 7. These two degradates appeared to be stable to hydrolysis at pH 4. The major hydrolysis product at pH 9 was the deesterified bensulfuron (IN-R9419). Bensulfuron slowly hydrolyzes to pyrimidine amine (IN-0290) and to homo-saccharin (IN-B6895) in the pH 9 buffer solution. Aqueous abiotic hydrolysis would	Main hydrolysis pathway under acidic and neutral conditions is the cleavage of the sulfonyl-urea bridge and under basic conditions de-esterification followed by cleavage. Acceptable study	Y	Hatzenbeler, 2002

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			be expected to contribute to the degradation of bensulfuron methyl significantly at pH 4, moderately at pH 9 and only slightly at pH 7 under conditions associated with typical use and/or exposure.			
B.2.1.9.2 Photochemical degradation	1. EPA 162-2	1. [Phenyl(U)- ¹⁴ C] bensulfuron methyl, 29.8 µCi/mg, Radiochemical purity 96.9% [Pyrimidine-4- ¹⁴ C] bensulfuron methyl, 16.6 µCi/mg, Radiochemical purity 99.1%	1. Photolysis of bensulfuron methyl was dependent on pH with half-lives ranging from 13 days at pH 5 to greater than 200 days at pH 7 and pH 9 at 25°C. However, when the observed degradation rate is corrected for the hydrolysis component, the photolysis component was insignificant (half-lives greater than 400 days) and independent of pH in the range 5 – 9. This observation is consistent with UV absorption data of bensulfuron methyl which showed little or no absorption above 290 nm; little or no direct photolysis of bensulfuron methyl is expected. IN-N5297 was found to be the major hydrolysis product at pH 5.	Apparent differences in degradation rates are observed in these studies, considered not significant due to the variability in results obtained and the consequent errors in estimation of degradation half-lives. Both studies are considered acceptable. Two further metabolites were determined at significant levels in Lentz (2003): IN-T5831 (pyrimidinyl-urea) and IN-YY142 (phenylglyoxylic acid).	1. N	1. Horne, 1987
	2. JMAFF, Japanese guideline 12 Noshan No.8147 Method: photodegradation in	2. [Phenyl(U)- ¹⁴ C] bensulfuron methyl, 57.5 µCi/mg, Radiochemical purity >95% [Pyrimidine-4- ¹⁴ C]	2. The photolytic stability of bensulfuron methyl was studied in two natural waters and sterile pH 7 aqueous buffer solutions. The test systems were continuously irradiated for 15 days using a xenon	Concluding comment: Degradation of bensulfuron methyl in sterile aqueous buffer solution exposed to natural sunlight is similar to the hydrolytic degradation rate. Indirect rather than direct photolysis may contribute significantly to the elimination of bensulfuron methyl from the environment.	2. Y	2. Lentz, 2003

[illegible][illegible]

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Test or Study & Annex point	Guideline and method	Test material purity and specification	Findings	Comments	GLP	Reference
	natural water by simulated sunlight	bensulfuron methyl, 53.1 µCi/mg, Radiochemical purity >95%	are simulated sunlight source (intensity equal to midday summer sunlight at a latitude of 52°N; equivalent to 28 days of sunlight) at 25°C under sterile conditions. The photolysis half life of bensulfuron methyl in Cavrini Farm and Ten-Mile Creek natural waters were 10 and 89 days as compared to 29 days in sterile pH 7 buffer. The major degradation products (>10% AR) identified were IN-T5831 (pyrimidinyl-urea) and 1N-YY142 (phenylglyoxylic acid).			
B.2.1.9.3 <i>Quantum yield</i>	1. FAO revised guidelines on Environmental Criteria for the Registration of Pesticides 2. JMAFF, Japanese guideline 12 Noshan No.8147	1. Calculation 2. Chemical actinometry, simulated sunlight	1. The quantum yield for bensulfuron methyl was calculated, using data from Horne (1987) obtained at pH 9 sterile buffer, to be 4×10^{-6} (based on 40N summer sunlight). 2. The quantum yield of bensulfuron methyl in sterile pH 7 buffer was calculated using chemical actinometry to be 3×10^{-5} .	Because direct photolysis plays an insignificant role in the degradation of bensulfuron methyl, it is difficult to accurately calculate a quantum efficiency. Both values found in Li (2000) and Lentz (2003) are very low, due to insignificant absorbance of the compound at wavelengths >290 nm. Since the value of 3×10^{-5} was obtained from an experiment that assumed sunlight intensities rather than from a calculation, the applicant suggests the experimental value as the appropriate value for meeting data requirements. Applicants' justification accepted	NA 2. Y	1. Li, 2000 2. Lentz, 2003

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Test or Study & Annex point	Guideline and method	Test material purity and specification	Findings	Comments	GLP	Reference
B.2.1.9.4 <i>Dissociation constant</i>	OECD 112 Method: spectrophotometric	DPX-F5384- 99.0 % purity Lot no: JUN00MA219	The dissociation constant was determined to be 5.2 at 20°C using the spectrometric intensity method.	Acceptable study	Y	Nobuhiro Koike, 2001
B.2.1.10 Stability in air, photochemical degradation, identity of breakdown products(s)	Method: Structure activity relationship methods by R. Atkinson	Calculation	The overall OH rate constant is $210.163 \times 10^{-12} \text{ cm}^3 \text{ molecule}^{-1} \text{ sec}^{-1}$. The estimated half-life for reaction with average daily air concentrations of hydroxyl radicals (12 hour daylight; $1.5 \times 10^6 \text{ OH radicals per cm}^3$) is 0.611 hours.	Accepted	NA	Schmuckler, 2001
B.2.1.11 Flammability including auto-flammability						
B.2.1.11.1 <i>Flammability</i>	EEC A.10.	DPX-F5384-333 98.66% purity	Results were negative. bensulfuron methyl did not support combustion in an initial screening test. No propagation of the product train occurred within the specified time and therefore no further testing was required.	Accepted	Y	Gravell, 2001
B.2.1.11.2 <i>Auto-flammability</i>	EEC A.16	DPX-F5384-333 98.66% purity	Results were negative. There was no exothermic indication up to 400°C. The temperature of bensulfuron methyl reached 400°C, the upper limit of the test, in a 20-mm cubic container.	Accepted	Y	Gravell, 2001

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Test or Study & Annex point	Guideline and method	Test material purity and specification	Findings	Comments	GLP	Reference
B.2.1.12 Flash point	EEC A.9.	NA	Not applicable as the active substance is not a liquid and does not have a melting point below 40°C.	Accepted	NA	NA
B.2.1.13 Explosive properties	EEC A.14. Methods: - thermal sensitivity - mechanical sensitivity with respect to shock - mechanical sensitivity with respect to friction	DPX-F5384-333 98.66% purity	Thermal sensitivity: no explosions were observed for bensulfuron methyl using a long steel tube closed and secured for either 6 or 2 mm orifice plates. Mechanical sensitivity with respect to shock was negative; no positive results were obtained for bensulfuron methyl in 21 drop impact tests conducted at 48 Joules (3.5 kg at 1.4 m) Mechanical sensitivity with respect to friction was negative; no positive results were observed for bensulfuron methyl in 6 trials conducted with a force of 360 N.	Accepted	Y	Gravell, 2001
B.2.1.14 Surface tension	EEC A.5 Method: Ring tensiometer method	DPX-F5384-258 97.9% purity	The surface tension of bensulfuron methyl was determined to be 74.9 ± 0.1 dynes/cm (74.9×10^{-3} N/m) at an average temperature of $20.3 \pm 0.1^\circ\text{C}$, using a calibrated tensiometer.	Accepted	Y	Huntley, 2000

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Test or Study & Annex point	Guideline and method	Test material purity and specification	Findings	Comments	GLP	Reference
B.2.1.15 Oxidising properties	Method: Structural argument	NA	An examination of the chemical structure of bensulfuron methyl establishes that the active substance is incapable of reacting exothermically with a combustible material. All the bensulfuron methyl atoms are in highly reduced oxidation states. The sulfonylurea nitrogens and the pyrimidine nitrogens are in the –3 oxidation state. The oxygen atoms are in the –2 oxidation state and likewise have no oxidation potential	Accepted	NA	Moore, L.A. 2004

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B.2.1.16 Overview of the physical and chemical properties of the active substance

Pure bensulfuron methyl is an odourless, white solid with a melting point of 179.4°C and a density of 1.486 g/cm³. Bensulfuron methyl is a weak acid with a pKa of 5.2, existing in the neutral form at pH values below the pKa and in the anionic form (acidic hydrogen on the nitrogen adjacent to the sulfonyl group) at pH levels above the pKa. As a consequence, solubility in water ranges from 2 mg/L at pH 5 to 3100 mg/L at pH 9. At 25°C bensulfuron-methyl hydrolyses rapidly at pH 4 ($t_{1/2}$ = 6d) but is essentially stable at pH 7 ($t_{1/2}$ = >200d) and pH 9 ($t_{1/2}$ = 141d). The proposed hydrolytic degradation pathway is shown in Figure ... together with the major metabolites formed. Under acidic and neutral conditions cleavage of the sulfonylurea linkage is the primary degradation pathway producing the pyrimidine-amine (IN-J290) and the sulfonamide (IN-N5297) metabolites. Under basic conditions, de-esterification produces the acid bensulfuron (IN-R9419). Bensulfuron methyl is not a surface active material.

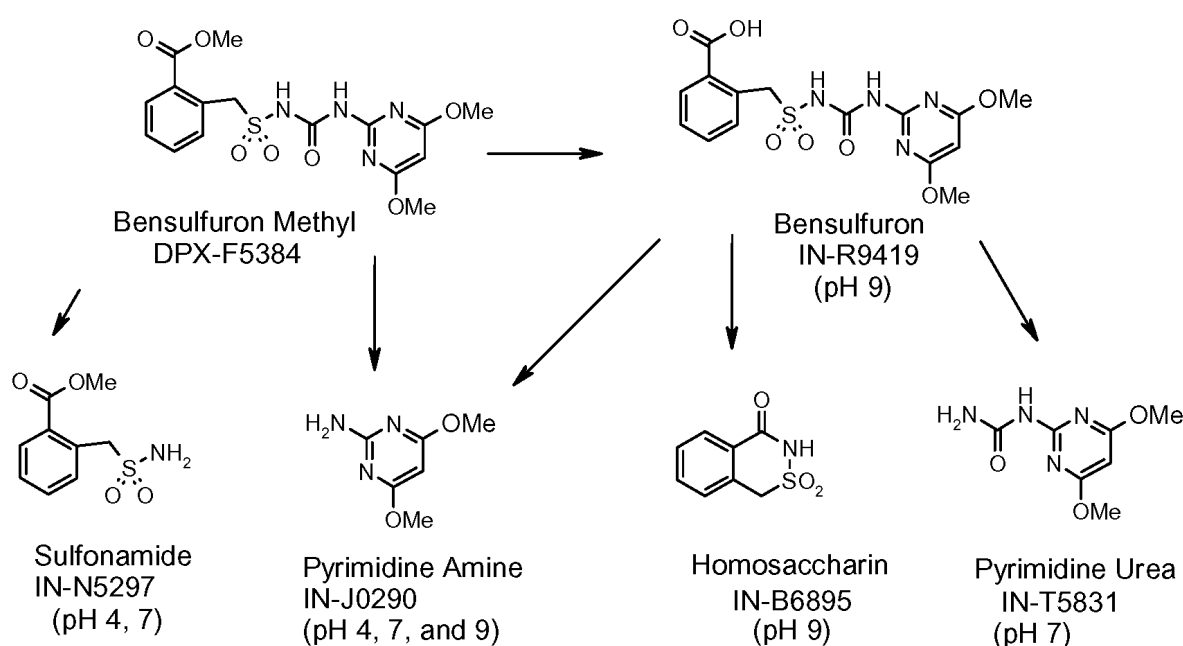
Indirect rather than direct photolysis (low absorption at $\lambda > 290$ nm) may contribute significantly to the degradation of bensulfuron methyl in natural water under sunlight. The octanol:water partition coefficient of bensulfuron methyl and its major metabolites indicate a low potential for bio-accumulation.

Volatilisation does not contribute to the dissipation of bensulfuron methyl in the environment as shown by the low vapour pressure of 2.8×10^{-12} Pa (25°C) and Henry's Law Constant $<10^{-10}$ Pa $\times$ m³ $\times$ mol⁻¹. The half-life of bensulfuron methyl in air for reaction with hydroxyl radicals is approximately 0.611 hours.

Bensulfuron methyl is soluble in dimethylformamide (70.8 g/L) and dichloromethane (18.4 g/L) and less soluble in n-heptane (3.62×10^{-4} g/L) and 1-octanol (7.47×10^{-2} g/L).

The technical material is not classified for explosive, oxidising or flammable properties.

Figure 2. 1: Proposed degradation pathway of Bensulfuron methyl in aqueous buffers at pH 4, pH 7 and pH 9



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B.2.2 Physical, chemical and technical properties of the plant protection products (Annex IIIA 2)

All physico-chemical tests were conducted in the Research Laboratory at Newark, Delaware and the Explosive Properties Flammability and Auto-flammability tests were conducted at Chambers Works Site in Deepwater, NJ. All tests were carried out with a test substance identified as DPX-F5384-332 containing 60.1% of the active ingredient Bensulfuron Methyl (DPX-F5384).

Where appropriate, tests were conducted in compliance with the principles of Good Laboratory Practice (GLP).

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Test or Study & Annex point	Guideline and method	Test material purity and specification	Findings	Comments	GLP	Reference
B.2.2.1 Colour and physical state	Visual assessment	DPX-F5384-332 60WG	The test material is a solid, tan in color, sold as a Water Dispersible Granule.	Accepted	Y	Bloemer, 2000
B.2.2.2 Odour	Olfactory assessment	DPX-F5384-332 60WG	The test material has a mild lignin odour.	Accepted	Y	Bloemer, 2000
B.2.2.3 Explosivity and oxidizing properties						
<i>B.2.2.3.1 Explosive properties</i>	EEC A 14 Methods: - thermal sensitivity - mechanical sensitivity with respect to shock - mechanical sensitivity with respect to friction	DPX-F5384-332 60WG	Thermal sensitivity – the test material was subject to conditions of intense heat and confinement. No explosions were observed. Mechanical Sensitivity of the test material with respect to shock was negative; no positive results were obtained in 21 drop impact tests conducted at 48 Joules. Mechanical Sensitivity of the test material with respect to friction was also negative; no positive results were observed in 6 trials conducted with a force of 360 N.	Accepted	Y	Gravell, 2001
<i>B.2.2.3.2 Oxidising properties</i>	EEC A 17 Method: burning rate tests	DPX-F5384-332 60WG	The test substance self extinguished at the 70/30 ratio but propagated flame at the 80/20 and 90/10 ratio of cellulose/test substance. However the burning rate at these ratios were slower than that of the test standard. The test substance is not an oxidizer.	Accepted	Y	Gravell, 2001
B.2.2.4 Flash point and other indications of flammability or spontaneous ignition						
<i>B.2.2.4.1 Flash point of liquids (containing flammable solvents)</i>	EEC A 9	NA	Not applicable to solid preparations.	Accepted	NA	
<i>B.2.2.4.2 Flammability of</i>	EEC A 10	DPX-F5384-332	Results were negative. The test material did not	Accepted	Y	Gravell, 2001

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Test or Study & Annex point	Guideline and method	Test material purity and specification	Findings	Comments	GLP	Reference
<i>solid preparations</i>		60WG	support combustion in an initial screening test. No propagation of combustion of the product train occurred within the specified time and, therefore, no further testing was required.			
B.2.2.4.3 <i>Auto-flammability of preparations</i>	EEC A 16	DPX-F5384-332 60WG	A sample of the test material was placed in a 20 mm cubic container constructed of stainless steel mesh and heated in an oven capable of reaching a temperature of 400±2°C. The oven temperature was incrementally raised to 400°C while the internal temperature of the sample was monitored. The temperature of the oven, recorded at the time the test sample reaches 400°C, is called the self-ignition temperature. The test substance reached a temperature of 400°C at a corresponding oven temperature of 400°C.	Accepted	Y	Gravell, 2001

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Test or Study & Annex point	Guideline and method	Test material purity and specification	Findings	Comments	GLP	Reference
B.2.2.5 Acidity/alkalinity and if necessary, pH value						
<i>B.2.2.5.1 Acidity /Alkalinity and pH value</i>	CIPAC MT 31	NA	Not applicable as pH of a 1% dilution with de-ionised water is not lower than 4 nor greater than 10.	Accepted	N/A	
<i>B.2.2.5.2 pH of an aqueous dilution</i>	CIPAC MT 75	DPX-F5384-332 60WG	The pH of the test material in a 1% aqueous dilution with de-ionised water is 7.0.	Accepted	Y	Bloemer, 2000
B.2.2.6 Viscosity and surface tension						
<i>B.2.2.6.1 Kinematic Viscosity (ULV preparations)</i>	OECD 114	NA	Viscosity not required as this preparation was not designed for Ultra Low Volume (ULV) use.	Accepted	NA	
<i>B.2.2.6.2 Viscosity</i>	Rotary Viscometer	NA	Not applicable to solid preparations	Accepted	NA	
<i>B.2.2.6.3 Surface tension</i>	EEC A 5	NA	Surface tension measurements are not applicable to solid preparations.	Accepted	NA	
B.2.2.7 Relative density and bulk density						
<i>B.2.2.7.1 Relative density</i>	EEC A 3		Relative density not required for solid preparations.	Accepted		
<i>B.2.2.7.2 Bulk density</i>	CIPAC MT 169	DPX-F5384-332 60WG	80.0 grams of a representative sample of the test material were placed into a 250 mL glass measuring cylinder. It was raised and allowed to fall vertically a distance of 2.5 cm onto a rubber pad. This procedure was repeated 50 times. The bulk density was calculated as 0.79 g/mL (loose) and 0.85 g/mL (tapped).	Accepted		

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Test or Study & Annex point	Guideline and method	Test material purity and specification	Findings	Comments	GLP	Reference
B.2.2.8 Storage stability and shelf life: Effects of light, temperature and humidity on technical characteristics of the plant protection product.						
<i>B.2.2.8.1 Storage Stability after 14 days at 54°C</i>	CIPAC MT46 CIPAC MT 75 pH MT 182 Wet Sieve MT 168 Suspensibility MT 171 Dust MT 47.2 Persistent Foaming MT 174 Dispersibility	DPX-F5384-332 60WG	Test material was stored at a temperature of 54°C for a period of 2 weeks. Results are reported with the initial measurement in brackets: Assay: 59.19% (60.1%; -1.5%) pH: 7.1 (7.0) Wet Sieve: 0.2% (0.3%) on 75µm sieve Suspensibility: 67.3% (68.5%) (analytical) Dust: 1.3 mg ; 0.004% (0.8 mg; 0.0027%) Persistent foam: 0 mL @ 1 min. (idem) Dispersibility: 92% (93%)	Accepted	Y	Bloemer, 2000
<i>B.2.2.8.2 Stability after storage for other periods and at other temperatures</i>	CIPAC MT46.3	NA	Not applicable as Bensulfuron methyl 60WG was stored at a temperature of 54°C for a period of two weeks (See above)	Accepted	NA	
<i>B.2.2.8.3 Minimum content after heat stability testing</i>	DuPont Analytical Method No. KE459.220.01.ES	DPX-F5384-332 60WG	Assay (as made) 60.1% Bensulfuron methyl Assay (after accelerated storage) 59.19% Bensulfuron methyl	Accepted	Y	Bloemer, 2000
<i>B.2.2.8.4 Low temperature stability (for liquid preparations)</i>	CIPAC MT 39	NA	Low temperature stability is not applicable to solid preparations.	Accepted	NA	

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Test or Study & Annex point	Guideline and method	Test material purity and specification	Findings	Comments	GLP	Reference
B.2.2.9 Storage stability and shelf life: Effects of light, temperature and humidity on technical characteristics of the plant protection product						
<i>B.2.2.9.1 Shelf life of the preparation at ambient temperature</i>	GIFAP Monograph No 17 MT 75 MT 168 MT 167 MT 53.3.1 MT 47.2 MT 174 MT 171 MT 178 MT 170	DPX-F5384-332 60WG	The shelf-life stability of the test substance was determined in accordance with the guidelines provided in GIFAP Technical Monograph No. 17. Assay: 59.96% Bensulfuron methyl Appearance/Packaging: No change in product appearance. No evidence of interaction with container. No indication of leakage as the heat seal remained completely intact. pH: 6.1 Suspensibility: 72% (Chemical analysis) Wet Sieve: 0.11% on 75µm sieve Wettability: 1 Second Persistent foam: 0mL @1 minute Dispersibility: 94% Dustiness: 0.6mg (0.002%) Attrition Resistance: 99.6% Dry sieve: >90% retained on 250 µm sieve <10% retained on 1000 µm sieve.	No description of the test in Bloemer 2000; Bloemer 2002 not found. Study has not been submitted	N	Bloemer, 2002

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Test or Study & Annex point	Guideline and method	Test material purity and specification	Findings	Comments	GLP	Reference
B.2.2.10 Technical characteristics of the plant protection product						
<i>B.2.2.10.1 Wettability</i>	CIPAC MT 53.3.1	DPX-F5384-332 60WG	A sample of the test material was dropped from a position level with the rim of a 250-mL beaker into the beaker, containing 100-mL of CIPAC Standard Water "D". The wetting time was recorded as 1 second.	Accepted	Y	Bloemer, 2000
<i>B.2.2.10.2 Persistent foaming</i>	CIPAC MT 47	DPX-F5384-332 60WG	A sample of the test material was added to a 250 mL graduated cylinder filled with ~180 mL of CIPAC Standard Water "D". Additional water was added to bring the volume to within 9 cm of the ground glass joint. The cylinder was stoppered and inverted 180 degrees by hand a total of 30 times in a period of 1 minute. After standing, undisturbed for 1 minute, no foam could be seen in the cylinder.	Accepted	Y	Bloemer, 2000
B.2.2.11 Suspensibility and suspension stability						
<i>B.2.2.11.1 Suspensibility</i>	CIPAC MT 168 (WG)	DPX-F5384-332 60WG	A 0.31 gram sample of the test material was added to CIPAC Standard Water "D" and made to a volume of 250 mL and agitated as directed in the CIPAC Method. The mixture was allowed to sit undisturbed in a 30°C water bath for 30 minutes. The top 225 mL of the suspension was removed by suction and the remaining liquid was taken to an analytical laboratory where suspensibility was determined by chemical analysis. 68.5% of the test material remained in suspension at the conclusion of the test.	Accepted	Y	Bloemer, 2000

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B.2.2.11.2 <i>Suspension stability (dispersibility - WG's)</i>	CIPAC MT 174	DPX-F5384-332 60WG	A 9.0 gram sample of the test material was placed in 900 mL of CIPAC Standard Water "D", maintained at a temperature of 20 +/- 1 degree Celsius. The mixture was agitated for one minute and then allowed to sit undisturbed for one minute. Suction was used to remove the top 810 mL. The remaining 90 mL of the suspension was dried to a constant weight and the dispersibility was assessed gravimetrically. At the conclusion of the test, it was determined that 93% of the test substance was dispersed.	Accepted	Y	Bloemer, 2000

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Test or Study & Annex point	Guideline and method	Test material purity and specification	Findings	Comments	GLP	Reference
B.2.2.11.3 <i>Suspension stability (spontaneity of dispersion - SC's)</i>	CIPAC MT160 or MT 174 as appropriate.		This test is not applicable to Water Dispersible Granules.	Directive 1991L0414 requires: The spontaneity of dispersion of water dispersible products (e.g. suspension concentrates and water dispersible granules) must be determined and reported according to CIPAC Methods MT 160 or MT 174 as appropriate. Justification not accepted	NA	
B.2.2.11.4 <i>Dilution stability</i>	CIPAC MT 41	NA	Only applicable to Water Soluble Products.	Accepted	NA	
B.2.2.11.5 <i>Dry sieve test (dustable powders only) and wet sieve test</i>	CIPAC MT 167	DPX-F5384-332 60WG	The dry sieve test required in this section is only applicable to dustable powders. Wet sieve test: A 10 gram sample of the test material was placed in a beaker and 100 mL of water added. The mixture was allowed to stand, was stirred, and was transferred to a 75 µm sieve as directed by the method. The sieve was washed, and then the residue transferred to a dish and dried. The residue collected was 0.3%.	Accepted	Y	Bloemer, 2000

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Test or Study & Annex point	Guideline and method	Test material purity and specification	Findings	Comments	GLP	Reference
B.2.2.12 Particle size distribution (dustable and wettable powders, granules), content of dust/fines (granules), attrition and friability (granules)						
<i>B.2.2.12.1 Size distribution of particles (in the case of powders) or nominal size range of granules</i>	CIPAC MT 170 (WG)	DPX-F5384-332 60WG	A 85 gram sample of the test material was introduced onto the top sieve of the stack placed upon the receiver pan. The sieves were placed in a Pascal Engineering Sieve Shaker and tapped for a period of 5 minutes as directed by CIPAC Method MT170. - The smallest sieve, upon which $\geq 90\%$ of the material was retained, was 250 microns (60 mesh). The largest sieve, upon which $\leq 10\%$ of the material was retained, was 1000 microns (18 mesh).	Accepted	Y	Bloemer, 2000
<i>B.2.2.12.2 Dust content of granular preparations (particle size of dust, if relevant)</i>	CIPAC MT 171	DPX-F5384-332 60WG	After the airflow was adjusted to a rate of 15 L per minute, a 30 gram sample of the test material was introduced into the gravimetric dust testing apparatus. After a period of 60 seconds the airflow was stopped and the amount of airborne dust collected on a filter was measured. The weight of the dust collected was 0.8 mg (0.003%).	Accepted	Y	Bloemer, 2000
<i>B.2.2.12.3 Friability and attrition resistance of granules</i>	CIPAC MT178	DPX-F5384-332 60WG	A representative sample of the test material was pre-sieved as directed by the method. Fifty grams of this material was placed into a glass jar along with an equal weight of glass beads. The jar was rotated at a speed of 72 rpm for a period of 60 minutes. The test substance was separated from the beads, placed on a 125 μm sieve and agitated for 3 minutes. The material remaining on the sieve was weighed and the attrition resistance calculated as 99.8%.	Accepted	NA	Bloemer, 2000

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Test or Study & Annex point	Guideline and method	Test material purity and specification	Findings	Comments	GLP	Reference
B.2.2.13 Emulsifiability, re-emulsifiability, emulsion stability						
<i>B.2.2.13.1 Emulsifiability, emulsion stability and re-emulsifiability of preparations which form emulsions</i>	CIPAC MT 36.1	NA	Not applicable to water dispersible granules	Accepted	NA	
<i>B.2.2.13.2 Stability of dilute emulsions and of preparations which are emulsions</i>	CIPAC MT 20 (EO,EW)	NA	Not applicable to solids or suspension concentrates.	Accepted	NA	
B.2.2.14 Flowability, pourability (rinsability) and dustability						
<i>B.2.2.14.1 Flowability of granular preparations</i>	CIPAC MT 172	DPX-F5384-332 60WG	A 50 gram sample of the test material was placed in a cylinder, 4.75 cm in diameter. A stamp, exerting a pressure of 25 g/cm ² , was placed on the surface of the test material. The cylinder was then placed in a glass bottle, sealed, and placed in an oven set at a temperature of 54°C for a period of two weeks. After completion of the oven storage, the contents of the cylinder were allowed to cool prior to being transferred to a sieve with 5-mm apertures. The sieve was then lifted to a height of 1 cm and dropped, as specified by the method, onto a rubber pad. 100% of the test substance passed through the sieve spontaneously.	Accepted	Y	Bloemer, 2000
<i>B.2.2.14.2 Pourability of suspensions</i>	CIPAC MT 148	NA	This test is not applicable to suspensions.	Accepted	NA	
<i>B.2.2.14.3 Dustability of dustable powders</i>	CIPAC MT 34	NA	This test is only applicable to dustable powders.	Accepted	NA	

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Chemical properties

Test or Study & Annex point	Guideline and method	Test material purity and specification	Findings	Comments	GLP	Reference
B.2.2.15 Physical and chemical compatibility with other products including plant protection products with which its use is to be authorized.						
<i>B.2.2.15.1 Physical Compatibility of tank mixes</i>	In-house		No tank mix recommendations are included in the label. However, Bensulfuron methyl 60WG was tested with various potential tank mix partners in two or three way combinations. Methods and results are presented in biological dossiers submitted at country level: No physical compatibility problems were observed.	The mentioned dossiers should be submitted.		
<i>B.2.2.15.2 Chemical compatibility of tank mixes</i>			As described in Biological Dossier submitted at country level compatibility with regard to efficacy on weeds and selectivity on rice crop was demonstrated during field trials conducted with following active substances: MCPA, Propanil and Quinclorac.	The mentioned dossiers should be submitted.		
B.2.2.16 Adherence and distribution to seeds			Only applicable to preparations for seed treatment.	Accepted		

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Physical and chemical properties

B.2.2.17 Summary and evaluation of data presented under points 2.2.1 to 2.2.16

Safety: Bensulfuron methyl 60WG is non-flammable, non-explosive and not an oxidiser. The preparation was tested for dustiness in accordance with CIPAC Method MT 171 and found to be “nearly dust free.”

Performance: The active ingredient content of Bensulfuron methyl 60WG, as manufactured was 60.1%. The physical and chemical properties of the preparation were measured using CIPAC and EEC Methods. All physical properties and chemical stability specifications, as defined by “The Manual on the Development and Use of FAO Specifications for Plant Protection Products” were met both prior to and after completion of accelerated storage. The preparation was stored at a temperature of 54°C for a period of 2 weeks.

This plant protection product can be conveniently measured using a calibrated volumetric measuring guide and easily dispersed in water. It can be sprayed through conventional application equipment without any screen or nozzle pluggage. Equipment cleanout is easily accomplished using standard wash out procedures.

Environmental Impact: The plant protection product is a nearly dust free, dry flowable granule that can easily be recovered (scooped up using a shovel) if spilled.

Missing data:

1. Shelf life of the preparation at ambient temperature: Study should be submitted
2. Suspension stability (spontaneity of dispersion - SC's) CIPAC MT160 or MT 174 as appropriate.
Applicants' statement: This test is not applicable to Water Dispersible Granules. Directive 1991L0414 requires: The spontaneity of dispersion of water dispersible products (e.g. suspension concentrates and water dispersible granules) must be determined and reported according to CIPAC Methods MT 160 or MT 174 as appropriate. Justification not accepted
3. Physical Compatibility of tank mixes: Study should be submitted
4. Chemical compatibility of tank mixes: Study should be submitted

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Physical and chemical properties

B.2.3 Reference relied on

Annex No., EU Data Requirement No., Reference No.	Author(s)	Year	Title Source Company Report No. GLP or GEP Status (where relevant) Published or not	EU Data Protection Claimed (Y/N)	Owner
Annex II Data and Information					
IIA, 2.1.1./01 IIA, 2.1.3./01	Huntley, K.	2000	Determination of melting point/melting range and thermal stability for bensulfuron methyl (DPX-F5384) ABC Laboratories Europe Ltd, ABC Laboratories, Inc. (Missouri) DuPont-3574 GLP, unpublished	Y	DuPont
IIA, 2.2./01	Huntley, K., Edgar, L.	2000	Determination of the density of bensulfuron methyl (DPX-F5384) ABC Laboratories, Inc. (Missouri) DuPont-4138 Revision No. 1 GLP, unpublished	Y	DuPont
IIA, 2.3.1./01	Schmuckler, M.E.	2001	Vapor pressure of bensulfuron methyl DuPont Stine-Haskell Research Center AMR 2284-92 Revision No. 1 GLP not applicable, unpublished	Y	DuPont
IIA, 2.3.2./01	Schmuckler, M.E.	2000	Henry's law constant for bensulfuron methyl DuPont Stine-Haskell Research Center DuPont-4663 GLP not applicable, unpublished	Y	DuPont
IIA, 2.4.1./01 IIA, 2.4.2./01	Huntley, K., Edgar, L.	2000	Determination of color, physical state, and odor for bensulfuron methyl (DPX-F5384) ABC Laboratories, Inc. (Missouri) DuPont-3578 GLP, unpublished	Y	DuPont
IIA, 2.5.1./01	Jeffery, D.J.	2000	UV/Visible absorption of bensulfuron methyl DuPont Experimental Station DuPont-1495 GLP, unpublished	Y	DuPont
IIA, 2.5.1./02	Schmuckler, M.E.	2000	¹ H nuclear magnetic resonance (NMR), mass spectrum (MS), infrared (IR) and ¹³ Carbon NMR spectra bensulfuron methyl DuPont Experimental Station DuPont-3534 GLP, unpublished	Y	DuPont
IIA, 2.6./01 IIA, 2.7./01	Jeffery, D.J.	2000	Solubility of bensulfuron methyl in water and organic solvents DuPont Experimental Station DuPont-1496 GLP, unpublished	Y	DuPont
IIA, 2.6./02	Moore, L. A.	1994	Solubility of bensulfuron methyl (DPX-F5384) in pH 5, 7, and 9 aqueous buffers DuPont Experimental Station AMR 2862-93 GLP, unpublished	Y	DuPont

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Physical and chemical properties

Annex No., EU Data Requirement No., Reference No.	Author(s)	Year	Title Source Company Report No. GLP or GEP Status (where relevant) Published or not	EU Data Protection Claimed (Y/N)	Owner
IIA, 2.8./01	Barefoot, A.C.	1989	N-Octanol/Water partition coefficient determination of bensulfuron methyl at pH 5, pH 7, and pH 9 DuPont Experimental Station AMR 1439-89 GLP, unpublished	Y	DuPont
IIA, 2.8./02	Schmuckler, M.E.	2001a	The calculated octanol water partition coefficient (K_{ow}) of IN-J290 DuPont Stine-Haskell Research Center DuPont-4624 GLP not applicable, unpublished	Y	DuPont
IIA, 2.8./03	Schmuckler, M.E.	2001b	The calculated octanol water partition coefficient (K_{ow}) of bensulfuron methyl metabolite sulfonamide (IN-N5297) DuPont Stine-Haskell Research Center DuPont-4625 GLP not applicable, unpublished	Y	DuPont
IIA, 2.9.1./01	Friedman, P.L.	Not specified	Hydrolysis of ^{14}C -DPX-F5384 DuPont Experimental Station AMR 143-83 Not specified GLP, unpublished	N	DuPont
IIA, 2.9.1./02	Hatzenbeler, C.J.	2002	Bensulfuron methyl: Hydrolysis in buffer solutions of pH 4, 7 and 9 Ricerca, LLC DuPont-4499 GLP, unpublished	Y	DuPont
IIA, 2.9.2./01	Horne, P.	1987	Photodegradation of [phenyl(U)- ^{14}C]DPX-F5384 and [pyrimidine-2- ^{14}C] DPX-F5384 in water (conducted in sunlight) DuPont Experimental Station AMR 899-87 Not GLP, unpublished	N	DuPont
IIA, 2.9.2./02	Lentz, N.R.	2003	Photodegradation of [^{14}C] bensulfuron methyl in natural water by simulated sunlight Ricerca, LLC DuPont-8697 GLP, unpublished	Y	DuPont
IIA, 2.9.2./03	Van-Nguyen A.	2003	Photodegradation of bensulfuron methyl in a simulated paddy system DuPont Stine-Haskell Research Center DuPont-11110 GLP, unpublished	Y	DuPont
IIA, 2.9.3./01	Li, Y.	2000	Quantum yield of bensulfuron methyl DuPont Stine-Haskell Research Center DuPont-3739 Not applicable GLP, unpublished	Y	DuPont
IIA, 2.9.4./01	Nobuhiro, K., Mitsubishi, K.K.	2001	Measurement of dissociation constant of bensulfuron methyl Mitsubishi K.K. Safety Science Laboratory D000158 GLP, unpublished	Y	DuPont

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Physical and chemical properties

Annex No., EU Data Requirement No., Reference No.	Author(s)	Year	Title Source Company Report No. GLP or GEP Status (where relevant) Published or not	EU Data Protection Claimed (Y/N)	Owner
IIA, 2.10./01	Schmuckler, M.E.	2000	Photochemical oxidative degradation of bensulfuron methyl DuPont Stine-Haskell Research Center DuPont-4662 Not applicable GLP, unpublished	Y	DuPont
IIA, 2.11.1./01 IIA, 2.11.2./01 IIA, 2.13./01	Gravell, R.L.	2001	Explosive properties, flammability of solids and autoflammability of bensulfuron methyl DuPont Stine-Haskell Research Center DuPont-4477 Revision No. 1 GLP, unpublished	Y	DuPont
Annex III Data and Information					
IIA, 2.14./01	Huntley, K., Sarff, P.	2000	Determination of the surface tension of bensulfuron methyl (DPX-F5384) ABC Laboratories, Inc. (Missouri) DuPont-3811 GLP, unpublished	Y	DuPont
IIA, 2.15./01	Moore, L.A.	2004	Bensulfuron methyl (DPX-F5384): oxidizing properties DuPont-15128 Not applicable GLP, unpublished	Y	DuPont
IIIA, 2.0./01	Anon	1987	Physical and chemical characteristics of Du Pont Londax® 60DF herbicide DuPont Experimental Station PC-5384-1-87 GLP: No Published: No	N	DuPont
IIIA, 2.0./02 IIIA, 2.1./01	Bloemer, D.S.	2000	Physical, chemical and technical properties of bensulfuron methyl 60% water-dispersible granular herbicide formulation DuPont Stine-Haskell Research Center DuPont-4366 GLP, unpublished	Y	DuPont
IIIA, 2.2.1./01 IIIA, 2.2.2./01 IIIA, 2.3./01	Gravell, R.L.	2001	Explosive and oxidizing properties, flammability of solids and relative self-ignition of bensulfuron methyl 60% water-dispersible granular herbicide formulation DuPont Stine-Haskell Research Center and DuPont Explosion Hazards Lab DuPont-4031 GLP, unpublished	Y	DuPont

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Physical and chemical properties

Annex No., EU Data Requirement No., Reference No.	Author(s)	Year	Title Source Company Report No. GLP or GEP Status (where relevant) Published or not	EU Data Protection Claimed (Y/N)	Owner
IIIA, 2.4.2./01 IIIA, 2.6.2./01 IIIA, 2.7.1./01 IIIA, 2.7.3./01 IIIA, 2.8.1./01 IIIA, 2.8.2./01 IIIA, 2.8.3./01 IIIA, 2.8.5./01 IIIA, 2.8.6.1./01 IIIA, 2.8.6.2./01 IIIA, 2.8.6.3./01 IIIA, 2.8.8.1./01	Bloemer, D.S.	2000	Physical, chemical and technical properties of bensulfuron methyl 60% water-dispersible granular herbicide formulation DuPont Stine-Haskell Research Center DuPont-4366 GLP, unpublished:	Y	DuPont
IIIA, 4.2./01	Cahill, W. R.	2002	Bensulfuron methyl 60% water-dispersible granular herbicide formulation: laboratory cleanout DuPont Stine-Haskell Research Center DuPont-10466 Not GLP, unpublished	Y	DuPont

ANNEX B

Active substance: Bensulfuron Methyl

B.3 Data on application and further information

WARNING: This document forms part of an EC evaluation data package and should not be read in isolation. Registration must not be granted on the basis of this document.

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Data on application and further information

B.3 Data on application and further information

B.3.1 Data on application relevant to the active substance (Annex IIA 3.1 to IIA 3.6)

B.3.1.1 Function (Annex IIA 3.1)

Postemergence, selective herbicide acting primarily through foliar uptake with little soil activity.

B.3.1.2 Effects on harmful organisms (Annex IIA 3.2)

B.3.1.2.1 Nature of the effects on harmful organisms

Bensulfuron methyl affects sensitive weeds through inhibition of the enzyme acetolactate synthase (ALS). Inhibition of ALS leads to the cessation of cell division and subsequent growth processes in plants. Symptoms of chlorosis appear in affected weeds in a matter of days with necrosis and subsequent death occurring within 10 to 25 days under optimal conditions for herbicidal activity.

B.3.1.2.2 Translocation in plants

When applied into paddy water, bensulfuron methyl rapidly disperses and is subsequently absorbed by the plants. Bensulfuron methyl can be absorbed through both foliage/shoots and roots with the former being most dominant. Sulfonylureas have a systemic phloem translocation in plants. When sulfonylurea molecules reach the acid cell wall environment either *via* foliar or root uptake, some of the molecules are in the neutral, highly permeable form, and are able to cross the phloem tube/companion cell plasma membrane and enter the phloem. Upon entering the alkaline phloem environment, the molecule dissociates and becomes "trapped" in its anionic form. Once "trapped" the sulfonylurea will move systemically by "mass flow" action with sucrose and other phloem solutes.

B.3.1.3 Field of use envisaged (Annex IIA 3.3)

Bensulfuron methyl is to be used in agriculture under field conditions only.

B.3.1.4 Harmful organisms controlled and crops or products protected or treated (Annex IIA 3.4)

B.3.1.4.1 Intended crop uses

Bensulfuron methyl is intended for control of broadleaved weeds (*Dicotyledonous*), aquatic broadleaved weeds (*Monocotyledonous* not *Graminae*) and *Cyperaceae* in rice.

B.3.1.4.2 Harmful organisms controlled

Bensulfuron methyl, when used according to label recommendations, provides selective control or suppression of key annual weeds in rice crop, including *Alisma plantago aquatica*, *Alisma lanceolata*, *Ammania coccinea*, *Bergia* spp., *Bolboschenus (Scirpus) maritimus*, *Schoenoplectus (Scirpus) mucronatus*, *Scirpus supinus*, *Butomus umbellatus*., *Cyperus* spp., *Lindernia* spp., *Heteranthera reniformis*, *Heteranthera rotundifolia*, *Sparganium erectum*, and *Typha* spp.

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Data on application and further information

B.3.1.4.3 Effects achieved

Sensitive plant species – Within 1-2 hours after the application of bensulfuron methyl, there is a cessation of shoot and root cell division and general plant growth. Secondary plant responses appear a few days after treatment. These visual symptoms include; chlorosis, necrosis, leaf malformation, terminal bud death, enhanced anthocyanin formation and vein discolouration. Plant death generally occurs within 10 to 25 days after the appearance of visual symptoms. During this period they do not take up any water or nutrients from the soil, thus removing the competitive effect from the rice crop.

B.3.1.5 Mode of action (Annex IIA 3.5)

B.3.1.5.1 Biochemical and physiological mechanism(s) and biochemical pathway(s) involved

Biochemical mechanism and pathway

The mode of action of sulfonylurea herbicides is inhibition of the plant enzyme acetolactate synthase (ALS), which is needed to make the essential branched-chain amino acids isoleucine, leucine and valine. The biochemical target site of sulfonylurea herbicides was first identified in the bacterium *Salmonella typhimurium*, where inhibition of bacterial growth by sulfonylureas was fully reversed by supplementing the medium with branched-chain amino acids. Early studies with micro organisms were extended to plants where a strong correlation was shown between herbicidal activity and ALS inhibition using various enzyme assays. Subsequent genetic studies showed that ALS is the only plant enzyme directly inhibited by sulfonylureas and that sulfonylureas do not directly provoke adverse effect on other plant biochemical processes, even at exaggerated concentrations.

Physiological mechanism

Bensulfuron methyl is a member of the sulfonylurea class of herbicides, which are effective inhibitors of plant root and shoot growth.

Bensulfuron methyl affects sensitive weeds through the inhibition of the enzyme acetolactate synthase (ALS). This enzyme is needed to make the essential amino acids isoleucine, leucine, and valine. Inhibition of ALS leads to rapid cessation of growth within 1-2 hours after treatment by arresting cell division in the G1 and/or G2 phases of interphase.

Sulfonylureas inhibit ALS isolated from both target weeds and crop plants. In every case, as been shown for all commercial sulfonylureas, crop selectivity is based on rapid metabolic inactivation of the herbicide by the tolerant plant. Rice crops are able to rapidly metabolise bensulfuron methyl to an inactive form of the molecule that does not inhibit the ALS enzyme whereas weed plants are killed because of their inability to rapidly convert the herbicide to an inactive form. According to all studies conducted to investigate the mode of action and selective action of bensulfuron methyl between rice and weeds, the metabolism of bensulfuron methyl by rice leaves was very rapid compared to that of sensitive weeds; in fact just after 4 hours from the treatment, rice is able to metabolize 50% of the applied active substance to one major metabolite which still contains both phenyl and pyrimidine rings, but without any specific physiological activity, while at the same time after the application 100% of unabridged bensulfuron methyl remains in weeds.

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Data on application and further information

Visual symptoms such as chlorosis, necrosis, leaf malformation and discolouration appear a few days after treatment. Sensitive plants can take 10 to 25 days to appear dead. During this time they do not take up any water or nutrients from the soil, thus removing the competitive effect from the crop.

B.3.1.5.2 Metabolism to the active substance

All data indicate that bensulfuron methyl is the active substance and that neither metabolism nor degradation processes are required for herbicidal activity.

B.3.1.5.3 Process for formation of the active substance

Bensulfuron methyl is the active substance.

B.3.1.6 Information on the occurrence or possible occurrence of the development of resistance, and appropriate management strategies (Annex IIA 3.6)

Resistance to crop protection chemicals is a common biological phenomenon that occurs in insects, fungi and weeds. It usually becomes evident after the repeated use of a particular pesticide selects for the naturally occurring resistant biotypes allowing them to multiply over several seasons until they become an obvious problem.

When herbicides with the same mode of action are used repeatedly over several years, naturally occurring resistant weed biotypes may survive a correctly applied herbicide treatment, propagate and become dominant in that field.

Resistance is the naturally occurring inheritable ability of some weed biotypes within a population to survive an herbicide treatment that should, under normal conditions of use, effectively control that weed population. Development of resistance in a weed population can be avoided or delayed by reducing the selection pressure that a particular herbicide produces on a weed population.

There are two precursors for the evolution of a trait like herbicide resistance in plant populations

- The occurrence of heritable variation for the trait
- Natural selection.

As genetic variation is naturally occurring, the rate of resistance development will be determined by the mode of inheritance of resistance trait(s), together with the intensity of selection pressure. The development of resistance under repeated application of a single herbicide (or mode of action) may be considered as an example of recurrent selection. This results in a progressive and sometimes rapid development of resistance of some species of a weed population exposed to the herbicide.

Three factors contribute to increase the selection pressure imposed by the herbicide:

- The efficacy of the herbicide. Highly effective herbicides control almost all the susceptible biotypes. The initial low level of resistant biotypes goes unnoticed because the overall level of weed control is so high. Resistant biotypes are given a competitive advantage and will continue to multiply until they dominate the population if susceptible biotypes are controlled.
- The frequency of use. The risk of developing a herbicide resistant weed population is higher where the same herbicides are used repeatedly on the same crop, the same fields and the same weeds over a long period of time.
- The duration of effect. The use of herbicides with a long residual soil activity accelerates the selection process that favours resistant biotypes. As susceptible biotypes are controlled over a long period of time, the soil reserve of susceptible seed becomes depleted and replaced by seed from resistant biotypes.

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The selection intensity in response to herbicide application is a measure of the relative efficacy of the product against the target weed species and/or the relative reduction in the seed production by the survivors. This is proportional to the herbicide dose. Selection in response to duration is a measure of the period of time over which phytotoxicity is imposed by a herbicide. Both intensity and duration interact to give a seasonal variation in the selection pressure imposed on weed species according to their phenology and growth.

Herbicide resistance to sulfonylureas

Bensulfuron methyl is a member of the sulfonylurea class of herbicides. The sulfonylureas are highly active herbicides that have been in commercial use since 1982 with the introduction of chlorsulfuron based products. The mode of action of the sulfonylureas as well as the imidazolinone and sulfonanilide herbicides is the inhibition of the enzyme acetolactate synthase (ALS). This enzyme is the first enzyme common to the biosynthesis of branched-chain amino acids like leucine, valine and isoleucine (Brown and Cotterman, 1994). The inhibition of the ALS enzyme results in a rapid cessation of growth and eventual plant death.

Resistance to weeds arising from treatment with sulfonylurea herbicides was first detected in the US in 1987 (Primiani et al., 1990), five years after the first widespread commercial use of chlorsulfuron. Since that discovery, resistance to ALS inhibitors (sulfonylureas, imidazolinones, triazolpyrimidine, etc.) has been documented in more than 60 weeds species (70% dicotyledon and 30% monocotyledon) in a total of 15 countries worldwide. ALS inhibitors-resistant weeds have appeared in cereals, corn, soybeans, rice, highway right-of-way, and forestry. In these situations the use of long residual ALS inhibitors and/or frequent application (more than one per season) and extensive use of unique mode of action herbicide has contributed to the development of resistance.

ALS target site resistance in Europe had been confirmed in 3 species in cereals (*Stellaria media*, *Papaver rhoeas* and *Chrysanthemum segetum*) and 3 species in rice (*Alisma plantago-aquatica*, *Cyperus difformis*, and *Schoenoplectus (Scirpus) mucronatus*). The first case was identified in 1991 where *Stellaria media* developed resistance to chlorsulfuron in Denmark (Saari et al., in Powles and Holtum, 1994). Since then resistance to *Stellaria media* has also been confirmed from fields in Ireland and Sweden, resistance to *Chrysanthemum* has been confirmed in fields from Ireland and Sweden, and *Papaver* resistance has been confirmed from fields in Spain, Italy and Greece. Resistance of rice weeds has been confirmed from fields in Portugal, Spain, Italy and Greece.

Mechanisms of resistance to sulfonylureas

Target site resistance

This is the basis for the vast majority of the weed biotypes with ALS inhibitor resistance (Saari et al., in Powles and Holtum, 1994). A mutation in the gene encoding ALS renders the weeds less sensitive to sulfonylureas. The ability for other ALS inhibitor herbicides to bind at this site and hence their activity is also affected.

Enhancement of Metabolism

This mechanism is based on the selection of plants with an increase in their ability to metabolise the herbicide, in this case the sulfonylureas are metabolised to non phytotoxic compounds rapidly enough to prevent lethal herbicides levels. No cases of resistance due to enhancement of metabolism have been reported in the scientific literature on dicotyledon weeds.

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Other concepts on herbicide weed resistance

Cross Resistance

Cross-resistance occurs in those biotypes that are resistant to one or more herbicides due to only one mechanism. Those resistant biotypes withstand herbicides from different chemical classes due to a mechanism (target site or enhancement of metabolism). For example, *Stellaria media* resistant to chlorsulfuron due to a mutation in the ALS enzyme will also be resistant to the rest of the ALS inhibitor herbicides, or in other words *Stellaria media* will be resistant to chlorsulfuron and cross resistant to all other ALS inhibitor herbicides that bind to the ALS enzyme at the mutated site.

However, ALS resistant biotypes are controlled, similarly to susceptible biotypes, by herbicides with a different mode of action to ALS inhibitors (Kudsk *et al.*, 1995).

Multiple Resistance

Multiple resistance will occur in those biotypes that are resistant to one or more herbicides due to several resistance mechanisms (target site and enhancement of metabolism). Weeds showing multiple resistance have not been reported in Europe up to now.

Risk of development of resistance in rice weeds and proposed strategies for its control

The reported cases of sulfonylurea resistant weeds described above show that the unrestricted use of sulfonylurea products has the risk to develop resistance. Development of resistance in rice weeds is constantly monitored in Europe and mainly in Italy, where a specific Research Group formed by Biologists and Agronomists is working on it from year 1996 (GIRE, “Gruppo Italiano Resistenza Erbicidi”, c/o University of Padova, Padova, Italy).

Table B.3. 1 below reports current updated status in European Countries; it is very important to underline that no new resistant weeds appeared in rice crop cultivated in Europe from year 2000 due to the implementation of correct strategies to prevent, avoid or delay resistance to an herbicide.

Table B.3. 1: Rice weeds resistant to herbicides in european countries

First year of evidence	Country	Weed species	Herbicides mode of action	Active substances
1986	Greece	<i>Echinochloa crus-galli</i>	Ureas and Amides	Propanil
1994	Italy	<i>Alisma plantago-aquatica</i>	ALS inhibitors	Bensulfuron methyl Cinosulfuron
1994	Italy	<i>Bolboschoenus (Scirpus) mucronatus</i>	ALS inhibitors	Bensulfuron methyl Cinosulfuron (Azimsulfuron and Etoxysulfuron in 1999)
1998	Italy	<i>Alisma plantago-aquatica</i> <i>Cyperus difformis</i> <i>Bolboschoenus (Scirpus) mucronatus</i>	ALS inhibitors	Metosulam
1999	Italy	<i>Cyperus difformis</i>	ALS inhibitors	Azimsulfuron Bensulfuron methyl Cinosulfuron
2000	Italy	<i>Echinochloa crus-galli</i>	Ureas and Amides	Propanil
1995	Portugal	<i>Alisma plantago-aquatica</i>	ALS inhibitors	Bensulfuron methyl
2000	Spain	<i>Alisma plantago-aquatica</i>	ALS inhibitors	Bensulfuron methyl
2000	Spain	<i>Cyperus difformis</i>	ALS inhibitors	Bensulfuron methyl

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Rapporteur Member State	Month and year	Active Substance (Name)
Italy	May 2006	Bensulfuron Methyl

Data on application and further information

In addition to specific measures recommended to prevent and/or delay resistance, and according to current knowledge, there are some aspects of bensulfuron methyl profile that can reduce the potential risk of further resistance developing. Most important aspects are the short residual soil activity of bensulfuron methyl and its half-life under field conditions that ranges from 4 to 39 days in rice paddy soil and from 1 to 19 days in rice paddy water. Timing of degradation in rice paddy water is also confirmed by specific water sediment study where the half-life found for bensulfuron methyl in water ranges from 16 to 21 days.

There are many agricultural practices that could help to reduce the selection pressure imposed by the herbicide. These include: crop rotation, rotation of herbicides with a different mode of action, use of mixtures or sequential treatments, use of short residual herbicides and limiting the number of treatments per crop with the same mode of action for control of the same weed. These practices should be designed into an integrated weed control programme that incorporates cultural practices along with chemical methods, e.g., timely use of ploughing/cultivation, use of weed free seed and competitive crop stands and judicious use of non-selective herbicides. Any incidences of poor control brought to DuPont's attention will be monitored. Furthermore it is proposed to include a label statement regarding resistance.

Proposed label text

When herbicides with the same mode of action are used repeatedly over several years in the same field, selection of resistant biotypes can take place. These can propagate and may become dominating. A weed species is considered resistant to an herbicide if it survives a correctly applied treatment at the recommended dose. Development of resistance in a weed species can be avoided or delayed by alternating (or tank-mixing) with suitable products having a different mode of action. In case of demonstrated weed resistance do not apply in pre-sowing of the rice crop.

B.3.2 Data on application relevant to the plant protection product (Annex IIIA 3)

B.3.2.1 Field of use envisaged (Annex IIIA 3.1)

Bensulfuron methyl 60WG is to be used in agricultural situations and under field conditions only. Bensulfuron methyl 60WG is a selective herbicide which controls broadleaved weeds and *Cyperaceae* in rice crop.

B.3.2.2 Effects on harmful organisms (Annex IIIA 3.2)

Bensulfuron methyl 60WG contains bensulfuron methyl, a herbicide from the sulfonylurea class. These herbicides are effective inhibitors of plant root and shoot growth.

Bensulfuron methyl affects sensitive weeds through inhibition of the enzyme acetolactate synthase (ALS). This enzyme is needed to make the essential amino acids isoleucine, leucine and valine. Inhibition of ALS leads to the cessation of cell division and subsequent growth processes in plants. Visual symptoms such as chlorosis, discolouration and leaf malformation appear in affected weeds few days after the herbicide application; necrosis and subsequent death occurring within 10 to 25 days. During this time sensitive weeds do not take up any water or nutrients from the soil, thus removing the competitive effect from the crop.

Rapporteur Member State	Month and year	Active Substance (Name)
Italy	May 2006	Bensulfuron Methyl

Data on application and further information

B.3.2.3 Details of intended use (Annex IIIA 3.3)

Bensulfuron methyl 60WG is intended to be used against broadleaved weeds and *Cyperaceae* in rice crop.

Bensulfuron methyl 60WG can be applied in flooded rice (with 5-10 centimetres of water) or in dry land rice (saturated with 0-1 centimetres of water). In flooded rice the application can occur in pre-sowing of the crop (from 2 to 5 days before sowing) or in post-emergence between 20 to 45 days after sowing (depending on locale this may be between BBCH 12 two leaves and BBCH 29 maximum tillers). In dry land rice the application can only occur in post-emergence between 20 to 50 days after sowing (again depending on locale this may be between BBCH 12 two leaves and BBCH 29 maximum tillers). In any case, according to Good Agricultural Practice, only 1 application per year is requested.

Bensulfuron methyl 60WG, when used according to label recommendations, provides selective control or suppression of key annual weeds in rice crop, including *Alisma plantago aquatica*, *Alisma lanceolata*, *Ammania coccinea*, *Bergia spp*, *Bolboschenus (Scirpus) maritimus*, *Schoenoplectus (Scirpus) mucronatus*, *Scirpus supinus*, *Butomus umbellatus*., *Cyperus spp.*, *Lindernia spp.*, *Heteranthera reniformis*, *Heteranthera rotundifolia*, *Sparganium erectum*, *Typha spp*.

Within the above recommended growth stage of the crop, application should be targeted at the growth stage of weed species to be controlled. Best control is achieved when weeds are treated at an early growth stage. Application timing for weeds is generally from the two leaf stage to maximum tillers, depending on the locale practice and the sensitivity of the weeds to bensulfuron methyl.

B.3.2.4 Application rate (Annex IIIA 3.4)

The maximum recommended dose rate is shown in Table B.3. 2.

Table B.3. 2: Maximum recommended dose rates of Bensulfuron methyl 60WG

Crop	Rate of plant protection product	Rate of active substance	Maximum number of applications per year
Rice	Bensulfuron methyl 60WG	Bensulfuron methyl	1
	100 g/ha	60 g/ha	

B.3.2.5 Concentration of active substance in material used (e.g., in the diluted spray, baits or treated seed) (Annex IIIA 3.5)

Bensulfuron methyl 60WG is typically applied using spray volumes between 200 and 400 L/ha. The suggested application rate of 60 grams bensulfuron methyl per hectare results in an active substance concentrations in the spray tank between 0.3 g/L (as maximum) and 0.15 g/L (as minimum).

B.3.2.6 Method of application(Annex IIIA 3.6)

Bensulfuron methyl 60WG is diluted in water and applied using tractor-mounted hydraulic field sprayers with ground-directed booms.

Rapporteur Member State	Month and year	Active Substance (Name)
Italy	May 2006	Bensulfuron Methyl

Data on application and further information

B.3.2.7 Number and timing of applications(Annex IIIA 3.7)

Bensulfuron methyl 60WG is applied once per year at the recommended application rate of 100 g/ha. Bensulfuron methyl 60WG can be applied in flooded rice (with 5-10 centimetres of water) or in dry land rice (saturated with 0-1 centimetres of water). In flooded rice the application can occur in pre-sowing of the crop (from 2 to 5 days before sowing) or in postemergence between 20 to 45 days after sowing (depending on locale this may be between BBCH 12 two leaves and BBCH 29 maximum tillers). In dry land rice the application can only occur in postemergence between 20 to 50 days after sowing (again depending on locale this may be between BBCH 12 two leaves and BBCH 29 maximum tillers). In any case, according to Good Agricultural Practice (**Errore. L'origine riferimento non è stata trovata.**), only 1 application per year is requested.

B.3.2.8 Necessary waiting periods or other precautions to avoid phytotoxic effects on succeeding crops(Annex IIIA 3.8)

Usually rice is not a rotational crop and if for any reason there will be rotation, according to agronomic science, only few crops like corn or soybean will be sowed. In that case Bensulfuron methyl 60WG applied in preceding rice according to the proposed GAP did not show phytotoxicity to succeeding crops; this is because under field conditions bensulfuron methyl degrades rapidly in rice paddy soil so there will not be significant amount of the active substance available for uptake by succeeding crops. Hence, under normal field conditions, the use of Bensulfuron methyl 60WG does not represent a hazard to rotational crops and does not justify to put on the label a specific waiting period before planting succeeding crops. Nevertheless, as general recommendation, in the event of crop failure for any reason, sow only rice crop in the same growing season.

B.3.2.9 Proposed instructions for use (Annex IIIA 3.9)

A European label proposal is included in Bensulfuron methyl EU Dossier, Document C, DuPont-6162 EU.

B.3.3 Further information on the active substance (Annex IIA 3.7 to IIA 3.9)

B.3.3.1 Recommended methods and precautions concerning handling, storage, transport or fire (Annex IIA 3.7)

Handling and storage: Provide appropriate exhaust ventilation at places where dust is formed. Use only in area provided with appropriate exhaust ventilation. Keep away from heat and sources of ignition. Avoid dust generation in confined areas. Use personal protective clothing when handling the products. Keep tightly closed in a dry and cool place. Keep tightly closed in original packaging. Store in a Keep out of reach of children.

Rapporteur Member State	Month and year	Active Substance (Name)
Italy	May 2006	Bensulfuron Methyl

Data on application and further information

Transport: ADR/RID: 3077 Environmentally hazardous substance, solid, n.o.s. (contains bensulfuron methyl) 9, 9(c) ADR.

IMO: Not regulated. Possible optional classification as per IMDG Code “Special Provision 909”, described as Environmentally hazardous substance, solid, n.o.s. (contains bensulfuron methyl) 9, UN 3077, PG III.

ICAO: Not regulated. Possible optional classification as per IATA “Special Provision A97” Environmentally hazardous substance, solid, n.o.s. (contains bensulfuron methyl) 9, UN 3077, PG III

Fire

Extinguishing media: Water spray, foam, carbon dioxide (CO₂). Extinguishing media that must not be used for safety reasons: High volume water jet (contamination risk).

On small fires if area is heavily exposed to fire and if conditions permit, let fire burn itself out since water may increase the area contaminated.

Special protective equipment for firefighters: In the event of fire, wear self-contained breathing apparatus.

Combustion gases: In the event of fire, the formation of carbon oxides and nitrogen oxides must be anticipated. No halogens.

B.3.3.2 Procedures for destruction or decontamination (Annex IIA 3.8)

B.3.3.2.1 Controlled incineration

A specific study on the thermal decomposition has not been carried out. Current practice used for experimental quantities of bensulfuron methyl is to incinerate at a temperature greater than 900°C with a residence time of 2-4 seconds in the chamber. Oxygen supply should be adjusted to generate <100 ppm carbon monoxide in the stack. Consideration of content of halogens is not relevant.

B.3.3.2.2 Others

Package product wastes: Waste should be disposed of in accordance with local and national regulations and laws. Packaging should be incinerated at a suitable, licensed plant.

B.3.3.3 Emergency measures in the case of an accident (Annex IIA 3.9)

Fire-fighting water must be contained and decontaminated in a suitable sewage plant or incinerated.

In the event of a spill, do not sweep the powder. Clean up by scoop or vacuum. Do not flush to a drain to avoid contamination of water. Cleanup of a spill on the ground should include removal of the upper layer of soil for incineration. Decontaminate equipment by rinsing with a 0.5% by volume solution of chlorine bleach (5% sodium hypochlorite).

Rapporteur Member State	Month and year	Active Substance (Name)
Italy	May 2006	Bensulfuron Methyl

Data on application and further information

B.3.4 Further information on the plant protection product (Annex IIIA 4)

B.3.4.1 Packaging (type, material, size, etc.), compatibility of the preparation with proposed packaging materials (Annex IIIA 4.1)

B.3.4.1.1 Description of the packaging

Bensulfuron methyl 60WG will be packed in 200 mL, 500 mL and 1000 mL High Density PolyEthylene (HDPE) jars, containing 100 g, 200 g and 500 g product, respectively. A description of the packaging and of the corresponding type of cap is given below:

HDPE JAR

100 g pack size:

The jar is set in an inner box (cardboard box). Ten inner boxes are grouped into an outer box.

Material : HDPE

Type of construction : Jar

Shape/size: Approximate diameter/height: 60 mm/100 mm

Capacity: 210 mL overflow

Type of closure: Screw cap with seal

Size of opening: 40 mm minimum

Accessories: One measuring device for each jar

200 g pack size:

The jar is set in an inner box (cardboard box). Ten inner boxes are grouped into an outer box.

Material : HDPE

Type of construction : Jar

Shape/size: Approximate diameter/height: 75 mm/1136 mm

Capacity: 500 mL overflow

Type of closure: Screw cap with seal

Size of opening: 40 mm minimum

Accessories: One measuring device for each jar

500 g pack size:

Ten jars are set into an outer corrugated box.

Material: HDPE

Type of construction : Bottle

Shape/size: Approximate diameter/ height: 100 mm / 175 mm

Capacity: 1100 mL overflow

Type of closure: Screw cap with seal

Size of opening: 40 mm minimum

Accessories: One measuring device for each jar

Rapporteur Member State	Month and year	Active Substance (Name)
Italy	May 2006	Bensulfuron Methyl

Data on application and further information

B.3.4.1.2 Suitability of the packaging

The packaging complies with ADR regulations having been tested using the ADR test methods appropriate to the pack type and material and classification of the contents and an appropriate UN certificate issued.

B.3.4.1.3 Resistance of the packaging

The packaging is a standard DuPont packaging, which has been used for similar formulations of other compounds such as sulfonylureas for nearly 20 years. This long period of use has proved its suitability and resistance, which has been confirmed by additional compatibility and permeability tests.

B.3.4.2 Procedures for cleaning application equipment (Annex IIIA 4.2)

Given the high unit activity of bensulfuron methyl, special care should be applied to cleaning of equipment according to the following procedure:

- Immediately after spraying, drain tank completely. Any contamination on the outside of the spraying equipment should be removed by washing with clean water.
- Rinse inside of tank with clean water and flush through boom and hoses using at least one-tenth of the spray tank volume. Drain tank completely.
- Half fill tank with clean water and add 1/3 litre household ammonia (contains 9.5% ammonia) or 0.5 litre All Clear Extra® for each 100 litres of tank volume. Agitate and then flush the boom and hoses with the cleaning solution. Top up with water making sure the tank is completely full and allow to stand for 15 minutes with agitation. Again flush the boom and hoses and drain tank completely.
- Nozzles and filters should be removed and cleaned separately with ammonia solution, All Clear Extra® at the same concentration as used for the sprayer.
- Rinse the tank with clean water and flush through the boom and hoses using at least one tenth of the spray tank volume. Drain tank completely.
- For disposal of washings, follow specific country guidelines. Do not spray onto sensitive crop or land intended for cropping with sensitive crop.

Bensulfuron methyl 60WG is non-corrosive to equipment, non-flammable and non-volatile.

Jar Test

The Tank Clean-out Procedure was evaluated for effectiveness, using the DuPont Jar Test Protocol.

300 mL of CIPAC 'D' water is placed into a 600-mL beaker and stirred. To this, 0.26 g of Bensulfuron methyl 60WG is added (corresponds to a concentrated spray solution of 85 g/ha bensulfuron methyl 60WG at a spray volume of 100 L/ha). After 2 minutes of stirring, the indicated amount of tank mix partner is added. After 2 more minutes of stirring, 100-mL aliquots are poured into three 4-oz polyethylene bottles, which are capped and allowed to stand at room temperature overnight.

Each polyethylene bottle was subjected to a standard clean-out:

1. The bottle is inverted twice and the liquid is discarded.
2. 10 mL tap water are added, the bottle is inverted twice, and the rinsate is discarded.
3. 100 mL tap water are added, ½ mL of All Clear Extra® is also added. The bottle is inverted twice and allowed to sit for 15 minutes. The bottle is then inverted two more times and the liquid is discarded.

Rapporteur Member State	Month and year	Active Substance (Name)
Italy	May 2006	Bensulfuron Methyl

Data on application and further information

4. Step 2 is repeated.
5. 5 mL acetonitrile and 15 mL water are added to extract any residual herbicide and the bottle is shaken well. The solution is filtered through PTFE 0.45 µm filters to remove solid before analysis.
6. The acetonitrile/water solution is chemically analyzed for tribenuron methyl.

Conclusion:

Bensulfuron methyl can be effectively cleaned out from all tank mix concentrations tested, using an All Clear Extra® clean-out procedure.

Reference: Cahill, W.R. (2002), Bensulfuron methyl 60% Water-Dispersible Granular Herbicide Formulation: Laboratory Cleanout DuPont-10466.

B.3.4.3 Pre-harvest intervals, re-entry and withholding periods, other use restrictions (Annex IIIA 4.3)

B.3.4.3.1 Pre-harvest intervals, re-entry intervals or withholding periods to minimise residues in crops, plants, plant products, treated areas or spaces

Table B.3. 3: Proposed pre-harvest intervals for Bensulfuron methyl 60WG

Crop	Proposed pre-harvest intervals
Rice	Do not apply within 60 days of harvest

Pre harvest interval of 60 days essentially becomes, from agronomic practice; in fact the latest stage to apply Bensulfuron methyl 60WG is BBCH 29 (maximum tillers) corresponding around 60 days before harvest. Residue levels decline to limit of quantification well in advance of the maturity of the rice grains.

Re-entry period for livestock in grazing areas: At latest application stage (maximum tillers) of Bensulfuron methyl 60WG rice grains are not there or can only be present in some plants. In any case very small rice grains are not intended for cattle grazing neither shortly after treatment nor later and exposure of livestock through this way is highly unlikely. For these reasons the establishment of a withholding period to protect livestock is not necessary.

Re-entry period for humans to crops, buildings or spaces treated: Rice crop typically do not require manual operations; agricultural practices performed between treatment and harvest (if any) are mechanical only. In addition Bensulfuron methyl 60WG has a low acute toxicity to mammals (oral or dermal route) and it is neither a primary irritant (eye or skin) nor a skin sensitiser. No claim of discomfort or injury was received from field investigators during all the residues and efficacy trials done for this product. Besides, exposure modeling of field workers entering treated areas showed adequate margins of safety (see Bensulfuron methyl EU Dossier, Annex IIIA, Document M-III, Section 3, DuPont-6195 EU). Therefore the probability for workers to be exposed to the product after treatment of Bensulfuron methyl 60WG to rice paddy is very low and does not justify the establishment of a re-entry period. However, common recommendation valid for all plant protection products is that no access should be given before spray solution has dried on the plants.

Withholding period for animal feedingstuffs: Not applicable since rice grains are highly unlikely to be used for feeding purposes before harvest.

Waiting period between application and handling treated products: Not applicable since rice grains are not collected/handled before harvest. Mature grains do not contain residues. Therefore, no additional waiting period between application and handling treated rice is required.

Rapporteur Member State	Month and year	Active Substance (Name)
Italy	May 2006	Bensulfuron Methyl

Data on application and further information

Waiting period between application and sowing or planting of succeeding crops: Usually rice is not a rotational crop and if for any reason there will be rotation, according to agronomic science, only few crops like corn or soybean will be sowed. In that case Bensulfuron methyl 60WG applied in preceding rice according to the proposed GAP did not show phytotoxicity to succeeding crops; this is because under field conditions bensulfuron methyl degrades rapidly in rice paddy soil so there will not be significant amount of the active substance available for uptake by succeeding crops. Hence, under normal field conditions, the use of Bensulfuron methyl 60WG does not represent a hazard to rotational crops and does not justify to put on the label a specific waiting period before planting succeeding crops. Nevertheless, as general recommendation, in the event of crop failure for any reason, sow only rice crop in the same growing season.

B.3.4.3.2 *Information on any specific agricultural, plant health or environmental conditions under which the preparation may or may not be used*

None of the test results obtained or observations made so far was such that restrictions should be imposed.

B.3.4.4 Statement of the risks arising and the recommended methods, precautions and procedures to minimise those risks relating to handling, storage, transport or fire (Annex IIIA 4.4)

Handling and storage:

Handling: Technical measures/precautions: Provide appropriate exhaust ventilation at places where dust is formed.

Safe handling advice: Use personal protective equipment. Keep away from heat and sources of ignition.

Storage: Technical measures/storage conditions: Keep containers tightly closed in a dry, cool and well-ventilated place. Keep at temperatures below 50°C. Store in a place accessible by authorised persons only.

Incompatible products: Humid air and water.

Packaging material: No materials to be specially mentioned.

Further information: Keep out of reach of children.

Transport information:

ADR/RID: 3077 Environmentally hazardous substance, solid, n.o.s. (contains bensulfuron methyl) 9, 9(c) ADR.

IMO: Not regulated. Possible optional classification as per IMDG Code "Special Provision 909", described as Environmentally hazardous substance, solid, n.o.s. (contains bensulfuron methyl) 9, UN 3077, PG III.

ICAO: Not regulated. Possible optional classification as per IATA "Special Provision A97" Environmentally hazardous substance, solid, n.o.s. (contains bensulfuron methyl) 9, UN 3077, PG III.

Protective clothing and equipment proposed:

Respiratory protection: Respirator with P1 filter.

Eye protection: safety glasses or face-shield.

Hand protection: Protective gloves.

Skin and body protection: Protective suit.

Hygiene measures: Wash hands before breaks and immediately after handling the product. Keep away from food, drink and animal feeding stuff. When using, do not eat, drink or smoke. Avoid contact with skin.

For environmental protection remove and wash all contaminated protective equipment or clothing before re-use.

Dispose of rinse water according with local and national regulations.

Rapporteur Member State	Month and year	Active Substance (Name)
Italy	May 2006	Bensulfuron Methyl

Data on application and further information

Fire-fighting measures:

Suitable extinguishing media: Water spray, foam, carbon dioxide (CO₂).

Extinguishing media which must not be used for safety reasons: High volume water jet (contamination risk).

Special protective equipment for firefighters: In the event of fire, wear self-contained breathing apparatus.

Specific methods (on small fires): If area is heavily exposed to fire and if conditions permit, let fire burn itself out since water may increase the area contaminated. Cool containers / tanks with spray water.

Information on combustion products likely to be generated in the event of a fire: In the event of fire, the formation of carbon oxides and nitrogen oxides must be anticipated. No halogens.

Procedures to minimise the generation of waste: Only purchase and store quantities of product required in the short term. Do not open larger containers than is necessary for immediate use. Do not prepare spray mixture volumes greater than is necessary for immediate use.

Information on combustion products likely to be generated in the event of a fire: In the event of fire, the formation of carbon oxides and nitrogen oxides must be anticipated. No halogens.

Procedures to minimise the generation of waste: Only purchase and store quantities of product required in the short term. Do not open larger containers than is necessary for immediate use. Do not prepare spray mixture volumes greater than is necessary for immediate use.

B.3.4.5 Procedures to be followed in the event of an accident during transport, storage or use
(Annex IIA 4.5)

General procedure: Methods for cleaning up: Clean up promptly by scoop or vacuum. Use approved industrial vacuum cleaner for removal. If spill area is on ground near valuable plants or trees, remove 5 cm of top soil after initial clean-up. Place removed material into suitable container for disposal at an approved site.

Environmental precautions: Do not flush into surface water or sanitary sewer system.

Protection of emergency workers and bystanders: Use personal protective equipment as proposed.

First aid measures:

General information: Remove victims from the danger zone. Remove contaminated clothing immediately. There is no specific antidote, treat symptomatically.

Inhalation: Move to fresh air. Administer oxygen or artificial respiration if needed. Consult a physician after significant exposure.

Skin contact: Wash off immediately with soap and plenty of water removing all contaminated clothes and shoes. If skin irritation persists, call a physician.

Eye contact: Rinse immediately with plenty of water, also under the eyelids for at least 15 minutes. If eye irritation persists see a specialist.

Ingestion: Drink 1 or 2 glasses of water. Do not induce vomiting without medical advice. Never give anything by mouth to an unconscious person. If you feel unwell, seek medical advice (show the label where possible.)

WARNING: This document forms part of an Evaluation data package and should not be read in isolation. Registration must not be gained on the basis of this document.

Rapporteur Member State	Month and year	Active Substance (Name)
Italy	May 2006	Bensulfuron Methyl

Data on application and further information

B.3.4.6 Procedures for destruction or decontamination of the plant protection product and its packaging (Annex IIIA 4.6)

B.3.4.6.1 Neutralisation procedures for use in the event of accidental spillage

Neutralisation by chemical reaction does not apply to Bensulfuron methyl 60WG.

B.3.4.6.2 Pyrolytic behaviour of the active substance under controlled conditions at 800 °C and the content of polyhalogenated dibenzo-p-dioxins in the products of pyrolysis

A specific study on the thermal decomposition has not been carried out. Current practice used for experimental quantities of bensulfuron methyl is to incinerate at a temperature greater than 900°C with a residence time of 2-4 seconds in the chamber. Oxygen supply should be adjusted to generate <100 ppm carbon monoxide in the stack. Consideration of content of halogens is not relevant.

Detailed instructions for safe disposal of the plant protection product and its packaging: Package product wastes: Triple rinse containers and place rinsate into the spray tank. Close and label the waste receptacles. Dispose of them at a suitable waste incineration plant holding a permit delivered by the competent authorities. Do not contaminate water. Disposal should be carried out in accordance with the official local regulations. Where large quantities are concerned, consult the supplier.

B.3.4.6.3 Methods other than controlled incineration for disposal of the plant protection product, contaminated packaging and contaminated materials

No other methods are currently available.

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Rapporteur Member State	Month and year	Active Substance (Name)
Italy	May 2006	Bensulfuron Methyl

Data on application and further information

B.3.5 Reference relied on

Annex No., EU Data Requirement No., Reference No.	Author(s)	Year	Title Source Company Report No. GLP or GEP Status (where relevant) Published or not	EU Data Protection Claimed (Y/N)	Owner
IIIA, 4.2./01	Cahill, W. R.	2002	Bensulfuron methyl 60% water-dispersible granular herbicide formulation: laboratory cleanout DuPont Stine-Haskell Research Center DuPont-10466 GLP: No Published: No	Y	DuPont

ANNEX B

Active substance: Bensulfuron Methyl

B.4 Proposals for classification and labelling

WARNING: This document forms part of an EC evaluation data package and should not be read in isolation. Registration must not be granted on the basis of this document.

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WARNING: This document forms part of an EC evaluation data package and should not be read in isolation. Registration must not be granted on the basis of this document.

Rapporteur Member State	Month and year	Active Substance (Name)
Italy	May 2006	Bensulfuron Methyl

Proposals for classification and labelling

B.4 Proposals for classification and labelling

B.4.1 Proposals for the classification and labelling of the active substance (Annex IIA 10)

B.4.1.1 Proposals including justification for the proposals for the classification and labelling of the active substance according to Council Directive 67/548/EEC

Bensulfuron methyl is currently included in Annex I of Directive 67/548/EEC (16th adaptation to technical progress).

The corresponding classification/labelling is as follows:

Symbols :

Xi (Irritant) – N (Dangerous for the environment)

Risk phrases :

R43 (May cause sensitisation by skin contact)

R51/53 (Toxic to aquatic organisms, may cause long-term adverse effects in the aquatic environment)

Safety phrases:

S2 (Keep out of the reach of children)

S24 (Avoid contact with skin)

S37 (Wear suitable gloves)

S61 (Avoid release to the environment. Refer to special instructions/Safety data sheets)

In the light of available and current testing information a different classification and labeling of bensulfuron methyl active substance is proposed according to the provisions of Directive 67/548/EEC (Table B.4 1 and Table B.4 2 below):

Table B.4 1: Proposed classification of bensulfuron methyl according to Directive 67/548/EEC

Hazard	N	Dangerous for the environment
Risk Phrases	R50/53	Very toxic to aquatic organisms, may cause long-term adverse effects in the aquatic environment
Risk Phrases	R63	Possible risk of harm to the unborn child

Table B.4 2: Proposed labelling of bensulfuron methyl according to Directive 67/548/EEC

Hazard	N	Dangerous for the environment
Risk Phrases	R50/53	Very toxic to aquatic organisms, may cause long-term adverse effects in the aquatic environment
Safety Phrases	S35	This material and container must be disposed of in a safe way.
	S57	Use appropriate containment to avoid environmental contamination
Risk Phrases	R63	Possible risk of harm to the unborn child

Justifications for the proposal:

Mammal toxicity

Bensulfuron methyl, according to Directive 67/548/EEC and its 16th adaptation to technical progress, is classified as skin sensitiser, since when evaluated for dermal sensitisation potential by Closed-patch repeated insult

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Italy	May 2006	Bensulfuron Methyl

Proposals for classification and labelling

(Maximization method) produced an insignificant weak hypersensitivity response in Guinea pigs. However all subsequent testing of bensulfuron methyl showed that the technical ingredient and formulations were negative for dermal sensitisation using Buehler and Magnusson-Kligman Maximisation testing methodologies. In addition, no incidence of skin sensitisation has been noted in the large population of bensulfuron methyl users, who have been in regular contact with bensulfuron methyl for several years. RMS agrees with the notifier about the proposal that bensulfuron methyl should not be classified under R43 (skin sensitiser).

Bensulfuron methyl is of low acute toxicity by the oral, dermal and inhalation routes of exposure with oral LD₅₀ greater than 5000 mg/kg, dermal LD₅₀ greater than 2000 mg/kg and an inhalation LC₅₀ greater than 5 mg/L. According to the provisions of Directive 67/548/EEC, classification for acute oral, dermal, and inhalation toxicity should not apply.

In the developmental toxicity study in rats, the NOAEL for maternal effects was 2000 mg/kg/day based on the absence of maternal toxicity at 2000 mg/kg bw/day. The foetal NOAEL was 500 mg/kg/day, based on the adverse effects on pup weight, and the presence of variations in pups at 2000 mg/kg/day.

In light of these findings the RMS proposes that bensulfuron methyl should be classified under R 63 (possible risk of harm to the unborn child)

Environmental toxicity

Environmental classification of bensulfuron methyl is based on the acute aquatic toxicity to algae (72-hour EC₅₀ of *Selenastrum capricornutum* is 0.020 mg/L) and to aquatic weeds (14-day EC₅₀ on *Lemna gibba* is 0.0008 mg/L). Bensulfuron methyl is not considered readily biodegradable under OECD protocol 301.

Physical and chemical hazards

Bensulfuron methyl should not be classified for physical and chemical hazards, according to Directive 67/548/EEC and according to available information.

B.4.2 Proposals for the classification and labelling of preparations (Annex IIIA 12.3 and 12.4)

Bensulfuron methyl 60WG is classified as follows according to Directive 1999/45/EC:

Symbol: N

Hazard: Dangerous for the environment

This classification is based on the following properties of the plant protection product:

R50: Acute aquatic toxicity to algae, 72-hour E_rC₅₀ on *Selenastrum capricornutum* = 0.020 mg/L and acute aquatic toxicity to aquatic weeds, 14-day E_rC₅₀ on *Lemna gibba* = 0.0008 mg/L

R53: Contains 60% w/w of bensulfuron methyl assigned R53. Bensulfuron methyl being not readily biodegradable. See Table B.4.3: and Table B.4.4: .

B.4.2.1 Proposals for risk and safety phrases

The proposed classification and labelling for Bensulfuron methyl 60WG according to the provisions of Directive 1999/45/EC are shown risk and safety phrases are given in Table B.4.3 and Table B.4.4 below:

Rapporteur Member State	Month and year	Active Substance (Name)
Italy	May 2006	Bensulfuron Methyl

Proposals for classification and labelling

Table B.4 3: Proposed classification for Bensulfuron methyl 60WG according to Directive 1999/45/EC

Component	Abbr.	Full text	Justification
Symbol of danger	N	Dangerous for the environment	Related to R50
Risk phrases	R50/53	Very toxic to aquatic organisms, may cause long-term adverse effects in the aquatic environment.	R50: 72-h E_rC_{50} on <i>Selenastrum capricornutum</i> = 0.020 mg/L and 14-day E_rC_{50} on <i>Lemna gibba</i> = 0.0008 mg/L R53: product contains 60% w/w of Bensulfuron methyl, active substance with assigned R53.
Risk phrases	R63	Possible risk of harm to the unborn child	Developmental toxicity study in rats: NOAEL maternal > 2000 mg/kg/day NOAEL foetal=500mg/kg/day

Table B.4 4: Proposed labeling for Bensulfuron methyl 60WG according to Directive 1999/45/EC

Component	Abbr.	Full text	Justification
Symbols	N	Dangerous for the environment	See classification
Risk phrases	R50/53	Very toxic to aquatic organisms, may cause long-term adverse effects in the aquatic environment.	See classification
Risk phrases	R63	Possible risk of harm to the unborn child	See classification
Safety phrases	S35	This material and its container must be disposed in a safe way	Appropriate for substances and preparations assigned the symbol N and not likely to be used by the general public
	S57	Use appropriate containment to avoid environmental contamination.	
Hazardous components		Contains bensulfuron methyl: May produce an allergic reaction	According to the provisions of Directive 1999/45/EC (article 10) label disclosure is required since bensulfuron methyl content is 60% (600 g/kg)

Note: Bensulfuron methyl 60WG is restricted to agricultural uses. Therefore the product is not destined to be sold or to be used by the general public. The strict application of labelling criteria according to Annex VI of Directive 67/548/EEC (which Directive 1999/45/EC refers to) does not require more safety phrases than those mentioned above.

ANNEX B

Active substance: Bensulfuron Methyl

B.5 Methods of analysis

WARNING: This document forms part of an EC evaluation data package and should not be read in isolation. Registration must not be granted on the basis of this document.

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Rapporteur Member State	Month and year	Active Substance (Name)
Italy	May 2006	Bensulfuron methyl

Methods of analysis

B.5.1 Analytical methods for the analysis of the preparation (Annex IIA 4.1; Annex IIIA 5.1)

B.5.1.1 Methods for the analysis of the active substance as manufactured (Annex IIA 4.1.1)

- Bensulfuron methyl (DPX-F5384): Analysis and certification of product ingredients for technical grade material produced at the Manati and Shanghai manufacturing facilities. Morgan E.A., 2002.*

Cross reference IIA, 4.1.1 This cross reference number denotes whole page(s) deleted from the Non-Confidential section of this report and included in the Confidential Attachment in Volume 4 Annex C of this monograph.

Guidelines:

CIPAC/4181/m

GLP:

Yes

Principle of the method:

“Londax[®] Herbicide, technical and formulations, determination of bensulfuron methyl (DPX-F5384), reversed-phase liquid chromatography (RPLC) method”

The method for assay of bensulfuron methyl as manufactured or Bensulfuron methyl 60WG (a 60% water-dispersible granule formulation) involves dissolution by ultrasonication of the preparation in acetonitrile. Samples are filtered immediately upon preparation before analysis. Analysis is done by reversed-phase liquid chromatography, with quantitation by ultraviolet absorbance at 236 nm. The column used is a Zorbax[®] SB-C8 column, and the mobile phase is an isocratic mixture of acetonitrile and water adjusted to pH 2.7. The weight percent of bensulfuron methyl in each sample is determined by comparing peak area of bensulfuron methyl with a calibration curve generated from the analysis of standard solutions. Weight percent bensulfuron methyl is reported.

Findings:

Information reported below concern the method for the determination of pure active substance in bensulfuron methyl as manufactured (and as formulated, as appropriate).

Specificity: The method was evaluated for interferences from expected manufacturing impurities and formulation inerts. None of the known impurities expected to be present in technical grade bensulfuron methyl co-elute with bensulfuron methyl. None of the *formulation* ingredients in Bensulfuron methyl 60WG are detected or co-elute with bensulfuron methyl. Since the EU requires that any interference present does not contribute more than $\pm 3\%$ to the total quantity determined, this method satisfies the EU criteria for specificity.

Linearity: The linearity of the method proposed for determination of the pure active substance as manufactured, or in the preparation, was demonstrated. The correlation coefficient for three different concentrations of bensulfuron methyl standards over the range of 60 to 130% of the assay level is 0.9999, and the slope is greater than zero.

Accuracy: The accuracy of this method for the analysis of bensulfuron methyl as manufactured samples was evaluated by analysing standard material as surrogate samples in duplicate at two levels that bracket the analyte target concentration for the analysis of real samples. The average percent recovery obtained was 100.0% with a standard deviation of 0.2%. The accuracy of this method for the analysis of formulated samples (Bensulfuron methyl

Rapporteur Member State	Month and year	Active Substance (Name)
Italy	May 2006	Bensulfuron methyl

Methods of analysis

60WG) was evaluated by fortifying the corresponding mixture of formulation inert ingredients with analytical standard to levels above and below the nominal concentration (60% bensulfuron methyl), as recommended in the guidelines. The average percent recovery obtained was 99.7% with a relative standard deviation of 0.65%. The EU has established that the mean recovery of a formulation method must lie within 98% and 102% if the percent active is above 10%. Therefore, the accuracy of this method is adequate.

Repeatability: Repeatability testing of the assay method was determined by calculating the standard deviation of the average percent bensulfuron methyl obtained from the analysis by one analyst of replicate test portions of the same sample of bensulfuron methyl as manufactured and Bensulfuron methyl 60WG. The relative standard deviation was 0.25% for the bensulfuron methyl as manufactured and 0.2% for Bensulfuron methyl 60WG. The maximum allowable relative standard deviation calculated from the modified Horwitz equation was 1.3% for technical material and 1.4% for Bensulfuron methyl 60WG. Therefore, this method fulfils the EU repeatability criteria.

There were no outliers during this testing.

Conclusion:

The method described in Morgan E.A., 2002 (Report: Du Pont-7095) is considered acceptable for the quantitation of the active substance as manufactured.

B.5.1.2 Analytical methods for the determination of isomers, impurities and additives in the active substance as manufactured (Annex IIA 4.1.2)

Analytical methods for the determination of quantitatively significant impurities (>1 g/kg) in bensulfuron methyl as manufactured are included in the batch analytical report (E. A. Morgan, 2002) provided in Bensulfuron methyl EU Dossier, Document J Part 1, Cross reference IIA, 4.1.2. As their disclosure would also disclose the impurity profile of the technical product, the notifier requests them to be treated as confidential information according to article 14 of Directive 91/414/EEC.

A summarised description of the procedure is provided under confidential information in Volume 4 Annex C of this monograph.

The notifier affirms that no impurities should be considered of toxicological or environmental relevance in bensulfuron methyl as manufactured, nor isomers or additives that would justify the submission and disclosure of enforcement methods.

Conclusion:

The methods described in Morgan E.A., 2002 (Report: Du Pont-7095; Confidential Attachment) are considered acceptable for the quantitation of impurities (process residual solvents, water, and inorganic ions) in the active substance as manufactured.

Rapporteur Member State	Month and year	Active Substance (Name)
Italy	May 2006	Bensulfuron methyl

Methods of analysis

B.5.1.3 Method for the determination of pure active substance in the formulation (IIIA 5.1.1)

2. *Method for the determination of the active substance in the formulation (Method for the determination of pure active substance in the active substance as manufactured (Annex IIA 4.1) Bensulfuron methyl (DPX-F5384): Analysis and certification of product ingredients for technical grade material produced at the Manati And Shanghai manufacturing facilities. Morgan E.A., 2002.*

Cross reference IIIA, 5.1.1

Guidelines:

CIPAC/4181/m

GLP:

Yes

Principle of the method:

see method nr. 1 Morgan E.A., 2002 (Report: Du Pont-7095)

The inert ingredients included in bensulfuron methyl 60WG are not expected to interfere with the extraction and analysis of bensulfuron methyl residues in any matrix. Therefore, no residue analytical methods have been developed specifically for bensulfuron methyl 60WG.

Conclusion:

The method described in Morgan E.A., 2002 (Report: Du Pont-7095) is considered acceptable for the quantitation of the pure active substance in the formulation

B.5.1.4 Analytical method for the determination of isomers, impurities, additives and formulants in plant protection products (Annex IIIA 5.1.1)

Analytical methods for the determination of quantitatively significant impurities (>1 g/kg) in the formulation are included in the batch analytical report (E. A. Morgan, 2002) provided in Bensulfuron methyl EU Dossier, Document J Part 1, Cross reference IIA, 5.1.2. As their disclosure would also disclose the impurity profile of the technical product, the notifier requests them to be treated as confidential information according to article 14 of Directive 91/414/EEC.

The inert ingredients included in bensulfuron methyl 60WG are not expected to interfere with the extraction and analysis of bensulfuron methyl residues in any matrix. Therefore, no residue analytical methods have been developed specifically for bensulfuron methyl 60WG.

Conclusion:

The method described in Morgan E.A., 2002 (Report: Du Pont-7095; Confidential Attachment) is considered acceptable for the quantitation of impurities (process residual solvents, water, and inorganic ions) in the formulation

Rapporteur Member State	Month and year	Active Substance (Name)
Italy	May 2006	Bensulfuron methyl

Methods of analysis

B.5.2 Analytical methods for the determination of residues (Annex IIA 4.2; Annex IIIA 5.2)

B.5.2.1 Analytical methods for the determination of residues in and/or on plants and plant products, (Annex IIA 4.2.1)

- Analytical enforcement method for the determination of bensulfuron methyl in rice (Grain and Straw) using column-switching liquid chromatography with ultraviolet detection. Amoo J.S., Jones W., 2001 (Du Pont-4510).*

Cross reference IIA, 4.2.1./01

Guidelines:

European Commission SANCO/825/00 rev.6; Directive 91/414/EEC

Deviations: Not applicable

GLP:

Not applicable

Principle of the method:

Residues of bensulfuron methyl in rice grain and rice straw are extracted from the plant tissue by homogenisation in an aqueous 50 mM ammonium carbonate solution. The extracts are purified and concentrated using disposable Envi™-Carb solid phase extraction columns. Analysis of rice grain and straw sample extracts is performed by reversed-phase HPLC utilising column switching with UV detection, where a fraction taken from the first column (phenyl) is automatically switched to a second column (C8) to improve the separation from matrix. Confirmation analysis is by electrospray LC/MS analysis of samples prepared using the same extraction and cleanup procedure. This method was designed for both a Maximum Residue Limit (MRL) enforcement method and for residue data collection. The data reported in Du Pont residue field studies AMR 3056-94 and Du Pont-9208 were determined using this method procedure.

Findings:

Specificity: The limit of quantification of the method proposed for monitoring bensulfuron methyl residues is 0.010 mg/kg for rice grain and 0.050 mg/kg rice straw. Analysis of control samples resulted in no apparent residues of bensulfuron methyl detected, and any peak response in the region of the bensulfuron methyl elution always corresponded to less than 20% of the limit of determination. Note: The Du Pont sulfonylurea rice herbicides metsulfuron methyl and azimsulfuron were tested and shown to be chromatographically resolved from bensulfuron methyl.

Recovery: The fortification data reported in the method proposed for monitoring bensulfuron methyl residues in rice grain and rice straw are summarised in Table B.5. 1. Fortification data obtained during method validation and concurrent fresh fortification analysis in residue field studies (Du Pont-4139, Du Pont-9208) produced similar results. The average recovery specified in the decision-making criteria is 70 to 110%, with a standard deviation of $\leq 20\%$ at each fortification level. Therefore, the recovery of this method is adequate for the purposes of residue data collection and enforcement of MRLs.

Rapporteur Member State	Month and year	Active Substance (Name)
Italy	May 2006	Bensulfuron methyl

Methods of analysis

Extraction efficiency: Extraction efficiency of this method was not demonstrated, due to the fact that no residues of bensulfuron methyl have been detected in treated rice grain or straw samples generated in plant metabolism studies. Therefore, samples containing weathered residues of bensulfuron methyl were not available.

Linearity: Calibration curves for external standards across a range of 0.0080 to 0.075 µg/mL were linear with correlation coefficients (R^2) greater than 0.99. This range of standard concentrations corresponds to 0.5 (rice straw) or 0.64 (rice grain) to 12 times the LOQ concentration in final extracts. Intercepts obtained by linear regression correspond to levels less than 20% of the LOQ.

Repeatability: Repeatability of the method is addressed by the data in Table B.5. 1. These recovery data were acquired at 2 laboratories, by 3 analysts, over 2 years. Relative standard deviations of less than 20% were obtained for fortifications made at the quantification limit for each matrix, as well as at higher levels. Therefore, the repeatability of this method is adequate for the purposes of residue data collection and enforcement of MRLs.

Reproducibility: The method of Amoo and Jones (2001) was validated by Powley (2003) for rice grain and Weber (2003) for rice grain and straw. During these independent laboratory validation exercises, the instrumental analysis was modified for LC/MS and LC/MS/MS, respectively. The application of mass spectrometric analysis to the original method added robustness by increasing selectivity, sensitivity, and concurrent confirmation for bensulfuron methyl. The recovery data obtained are summarised in Table B.5. 1. The successful independent laboratory validation demonstrates the reproducibility of this method.

Limit of Quantification: The limit of quantification of the method is 0.010 mg/kg for rice grain and 0.050 mg/kg rice straw. Since no quantifiable residues have been observed in mature grain, the proposed MRL is 0.010 mg/kg for rice grain. Rice straw is not a feed or food item within the ERL and a MRL for this commodity will not be proposed. According to guidelines, the MRL may be set at the limit of determination of the enforcement method. Therefore, the sensitivity of this method is adequate to enforce the proposed MRL.

Confirmatory Method: Confirmation of results obtained by this column switching HPLC-UV method is performed by LC/MS analysis of samples prepared using the same extraction and cleanup procedures. Chromatographic separation is on a single reversed-phase column (C8) and a single quadrupole mass spectrometer equipped with an electrospray interface operating in the positive ionisation mode is used for selected ion monitoring (SIM) of the protonated molecular ion response for bensulfuron methyl. The recovery data obtained using the confirmatory procedure is summarised in Table B.5. 1. The application of LC/MS/MS for quantitative analysis by Weber (2003) provides an additional alternative for confirmatory analysis.

Conclusion:

The method described in Amoo J.S., Jones W., 2001 (Du Pont-4510) is considered acceptable. However, it should be emphasized that column switching is here required to overcome the poor selectivity of the detection system, i.e. UV absorption at 245 nm. In order to implement column switching technique a well equipped analytical laboratory is required as well as particularly skilled technical personal. On the other hand, renouncing on this technique would require the use of highly selective detection (LC/MS/MS coupling still not everywhere considered as commonly available equipment).

Rapporteur Member State	Month and year	Active Substance (Name)
Italy	May 2006	Bensulfuron methyl

Methods of analysis

4. *Independent Laboratory Validation of Du Pont-4510, "Analytical enforcement method for the determination of bensulfuron methyl in rice (grain and straw) using column-switching liquid chromatography with ultraviolet detection" on rice grain. Powley C.R., 2003 (Du Pont-5592).*

Cross reference IIA, 4.2.1./04

Guidelines:

OPPTS 860.1340, SANCO/825/00 rev. 6 (June 20, 2000)

Deviations: None

GLP:

Yes

Principle of the method:

Du Pont-5592 is an independent laboratory validation (ILV) for the crop enforcement method Du Pont-4510. The method passed the ILV. The procedure for extraction and analyte purification was followed as written, except that quantification of final extracts was by LC/MS using conditions similar to those specified for the confirmatory analysis in Du Pont-4510. The ILV data are shown in Table B.5. 1.

Findings:

Samples were extracted and purified as specified in Du Pont-4510, Section 4.2.10, up to and including Step 7. After that, samples were reconstituted by adding 1 mL of acetonitrile, vortex mixing, then ultrasonicated for 5 min in a beaker of ice water. Then 9 mL of 0.01 M acetic acid was added to each vial, and the samples were re-vortex mixed and sonicated for another 5 min in a beaker of ice water. Finally, they were vortex mixed a third time. The samples were then filtered into an autosampler vial for HPLC/MS analysis. Reason for change: The independent laboratory decided to perform the analysis part of the procedure by HPLC/MS instead of column-switching HPLC/UV. Therefore, the reconstitution procedure specified in Du Pont-4510 for the LC/MS confirmatory procedure was followed.

The analysis was done using LC/MS conditions similar to those specified for the confirmatory analysis in Du Pont-4510. Reason: The LC/UV analysis specified in Du Pont-4510 requires a 500-µL injection volume. The independent laboratory can only make injections up to 250 µL. Preliminary method evaluation indicated that sufficient sensitivity would not be obtained with reduced injection volumes. Therefore, we chose to use the more sensitive LC/MS approach, with reduced injection volumes. Also, it was decided to use LC/MS instead of column-switching LC/UV because few enforcement labs use column-switching HPLC. LC/MS and LC/MS/MS are now the preferred analytical methods for sulfonylurea herbicides.

Conclusion:

The method described in Powley C.R., 2003 (Du Pont-5592). is considered acceptable for the purpose of validation. However, it could be disputed on whether one quadrupole with single ion monitoring of the molecular peak offers enough selectivity due to extent of matrix constituents eluting (no column switching !).

Rapporteur Member State	Month and year	Active Substance (Name)
Italy	May 2006	Bensulfuron methyl

Methods of analysis

5. *Magnitude of residues of bensulfuron methyl in rice (cereals) following application of bensulfuron methyl (DPX-F5384) 60WG formulation in wetland rice fields - southern Europe, season 2002. Hornshuth, M.J., 2003 (Du Pont-9208).*

Cross reference IIA, 4.2.1./03

Guidelines:

Not specified

GLP:

Yes

Principle of the method:

Du Pont-9208 is a Magnitude of Residue study that contains an independent laboratory validation for the crop enforcement method Du Pont-4510.

Test item code number: F5384-332 (water dispersible granule (WG), containing 600g bensulfuron methyl per kg formulated product). Reference analytical standard code number: F5384-332 (purity: 97.9%)

The procedure for extraction and analyte purification was followed as written, except that quantification of final extracts was by LC/MS/MS using similar conditions to those specified for the confirmatory analysis in Du Pont-4510. The total ion current (TIC) from 2 individual ion transitions are used for the quantitative LC/MS/MS analysis. The recovery data from the validation and fortifications made concurrently with field sample analyses are shown in Table B.5. 1.

Findings:

Rice grain and rice straw were treated with Bensulfuron methyl 60WG herbicide in accordance with GAP (a single application at 60 g as/ha).

- No residues above the defined LOD (0.003 mg/kg) of bensulfuron methyl were found at crop maturity (67- to 80-day PHI/DALA) in rice grain specimens taken from the four southern European field locations in this study.
- No residues above the defined LOD (0.02 mg/kg) of bensulfuron methyl were found at crop maturity (67- to 76-day PHI/DALA) in rice straw specimens taken from the two Italian field locations in this study. For the two Spanish field locations, residues of bensulfuron methyl at crop maturity (78- to 80-day PHI/DALA) bensulfuron methyl residues were detected at less than the LOQ (<0.05 [0.04] mg/kg) in one treated straw specimen and <LOD (<0.02 mg/kg) for the second specimen taken from Test Site No. 4. Bensulfuron methyl residues in rice straw specimens taken from Trial 3 were 0.10/0.10 mg/kg, respectively.
- Recovery and control specimens analyzed concurrently with treated specimens indicated that the method generated acceptable data.
- Test specimens in this study were stored frozen at $-20 \pm 5^{\circ}\text{C}$ from sampling to extraction for no longer than 4 months for bensulfuron methyl analysis. Results from a separate freezer storage stability study (Du Pont-4139) indicate that bensulfuron methyl is stable in rice grain and rice for up to 15 months when stored frozen at $-20 \pm 5^{\circ}\text{C}$.

Rapporteur Member State	Month and year	Active Substance (Name)
Italy	May 2006	Bensulfuron methyl

Methods of analysis

Conclusion:

The method described in Hornshuh, M.J., 2003 (Du Pont-9208) is considered acceptable for the investigation of residues in rice and straw. However, the LC/MS/MS coupling is still not everywhere considered as commonly available equipment.

6. *Analytical method for the determination of bensulfuron methyl residues in rice grain and Rice straw by HPLC using a column and eluent switching system. Zhou M., 1993 Du Pont AMR 2722-93)*

Cross reference IIA, 4.2.1./09

Guidelines:

USEPA 171-4(D), 171-4(C)

Deviations: Not applicable

GLP:

No

Principle of the method:

Reference Standard Material Code: F5384-83 (Purity: 99.5%)

Residues of bensulfuron methyl in rice grain and rice straw were determined by column- and eluent-switching HPLC with UV detection. Bensulfuron methyl is extracted from the plant tissue by homogenisation in methylene chloride. The extracts are purified and concentrated using disposable silica solid-phase extraction cartridges. Analysis of rice grain and straw matrices is performed by reversed-phase HPLC utilising eluent- and column-switching with UV detection, where a fraction taken from the first column (phenyl) is automatically switched to a second column (C8) to improve the separation from matrix. This method was used to generate freezer storage stability recovery data for bensulfuron methyl in rice straw reported in Du Pont residue field study AMR 3056-94.

Findings:

Specificity: The limit of quantification of the method proposed for monitoring bensulfuron methyl residues is 0.010 mg/kg for rice grain and 0.050 mg/kg for rice straw. Analysis of control samples resulted in no detectable apparent residues of bensulfuron methyl, the response in the area of the bensulfuron methyl peak always corresponded to less than 20% of the limit of determination. Therefore, the specificity of this method was sufficient to provide quality data for the application of storage stability sample analysis.

Recovery: The fortification data reported in the method proposed for monitoring bensulfuron methyl residues in rice grain and rice straw are summarised in Table B.5. 1. Fortification data obtained from fresh and freezer aged rice straw fortification samples produced similar results (AMR 3056-94). The average recovery specified in the decision-making criteria is 70 to 110%, with a standard deviation of $\leq 20\%$. Therefore, the recovery of this method is adequate for the purposes of residue data collection.

Extraction efficiency: Extraction efficiency of this method was not demonstrated, due to the fact that no residues of bensulfuron methyl have been detected in treated wheat samples generated in plant metabolism studies. Therefore, samples containing weathered residues of bensulfuron methyl were not available.

Rapporteur Member State	Month and year	Active Substance (Name)
Italy	May 2006	Bensulfuron methyl

Methods of analysis

Linearity: Calibration curves for external standards across a range of 0.040 to 0.625 µg/mL were linear with correlation coefficients (R^2) greater than 0.99. Intercepts obtained by linear regression correspond to levels less than 20% of the LOQ.

Repeatability: Repeatability of the method is addressed by the data in Table B.5. 1. These recovery data were acquired at 1 laboratory over 2 years. Relative standard deviations of less than 20% were obtained for fortifications made at the quantification limit for each matrix, as well as at higher levels. Therefore, the repeatability of this method is adequate for the purposes of residue data collection.

Reproducibility: The method was applied to the analysis of bensulfuron methyl fortified samples in freezer storage stability study (AMR 3056-94) for rice straw. The recovery data from fresh and aged fortified samples are summarised in Table B.5. 1. Recovery results that are consistent with the original method validation demonstrate the reproducibility of this method.

Limit of Quantification: The limit of quantification of the method is 0.010 mg/kg for rice grain and 0.050 mg/kg rice straw.

Confirmatory Method: The confirmatory method for rice grain and straw is contained in the current enforcement method, Du Pont-4510.

Conclusion:

The method described in Du Pont AMR 2722-93 (Zhou M., 1993) is the former version of the one in Du Pont-4510 (Amoo J.S., Jones W., 2001), proposed as the analytical enforcement method for the determination of bensulfuron methyl in rice (Grain and Straw) using column-switching liquid chromatography with ultraviolet detection.

The method is considered acceptable with regard to the results produced. Homogenisation in aqueous 50 mM ammonium carbonate solution as described in Du Pont-4510 should nowadays be preferred in front of methylene chloride extraction.

7. *Determination of rice herbicide candidate DPX-F5384 in rice grain Slates, R. V., 1983b (Du Pont AMR-132-83).*

Cross reference IIA, 4.2.1./05

Guidelines:

Not specified

Deviations: Not applicable

GLP:

No

Principle of the method:

DPX-F5384 is isolated from polished or unpolished rice grain by methylene chloride extraction. The compound is separated from major interfering components by acetonitrile-hexane partitioning and by collection of the DPX-F5384 on a disposable C18 Bond ElutTM column from aqueous solution. After elution of the sample residue from the Bond ElutTM column by acetonitrile, the residue for polished rice samples is transferred to mobile phase, and DPX-F5384 is determined by high performance liquid chromatography HPLC.

Rapporteur Member State	Month and year	Active Substance (Name)
Italy	May 2006	Bensulfuron methyl

Methods of analysis

Additional cleanup is required for unpolished rice samples. The residue eluted from the Bond ElutTM column is redissolved in water at pH 10 and is washed with toluene. After acidification of the aqueous phase to pH 4, DPX-F5384 is extracted into toluene, transferred to mobile phase, and is determined by HPLC.

The detection limit for DPX-F5384 in rice grain is 0.02 ppm. Average recovery efficiency for rice control samples fortified with DPX-F5384 at 0.02 to 0.08 ppm is 96%.

The residue data reported in Du Pont studies BE-A-11-92-09-BG, AMR 4162-96, and Du Pont-8797 were determined using this method procedure.

Findings:

Specificity: The limit of quantification of the method for monitoring bensulfuron methyl residues is 0.020 mg/kg for rice grain. Analysis of control samples resulted in detection of minor apparent residues of bensulfuron methyl that corresponded to less than 20% of the limit of determination. It can therefore be concluded that few, if any, apparent residues or false positive values would arise.

Recovery: The fortification data reported in the method proposed for monitoring bensulfuron methyl residues in rice grain are summarised in Table B.5. 1. Fortification data obtained during method validation and concurrent analysis with residue field samples produced similar results (BE-A-11-92-09-BG, AMR 4162-96). The average recovery specified in the decision-making criteria is 70 to 110%, with a standard deviation of $\leq 20\%$. The recovery of this method is adequate for the purposes of residue data collection.

Extraction efficiency: Extraction efficiency of this method was not demonstrated, due to the fact that no residues of bensulfuron methyl have been detected in treated wheat samples generated in plant metabolism studies. Therefore, samples containing weathered residues of bensulfuron methyl were not available.

Linearity: Calibration curves for external standards across a range of 0.020 to 0.25 $\mu\text{g/mL}$ were linear with correlation coefficients (R^2) greater than 0.99. Intercepts obtained by linear regression correspond to levels less than 20% of the LOQ.

Repeatability: Repeatability of the method is addressed by the data in Table B.5. 1. These recovery data were acquired at 2 laboratories over span of 15 years. Relative standard deviations of less than 20% were obtained for fortifications made at the quantification limit for each matrix, as well as at higher levels. Therefore, the repeatability of this method is adequate for the purposes of residue data collection.

Reproducibility: The method of Slates (2001) was independently validated by Cicotti (1990) for rice grain. The recovery data obtained are summarised in Table B.5. 1. The successful independent laboratory validation demonstrates the reproducibility of this method.

Limit of Quantification: The limit of quantification of the method is 0.020 mg/kg for rice grain. The sensitivity of this method is adequate for residue data collection.

Conclusion:

The method described in Du Pont AMR-132-83 (Slates, R. V., 1983b) is the former version of the method described in Du Pont AMR 2722-93 (Zhou M., 1993). Poor separation of the test substance from the matrix is attained during cleaning steps, resulting in poor chromatographic resolution of the DPX-F5384 peak. Validation would never be attained under nowadays conditions due to insufficient specificity and the method would not be considered 'fit for purpose'. Anyhow, analytical results obtained at that time should be considered sufficiently reliable.

Rapporteur Member State	Month and year	Active Substance (Name)
Italy	May 2006	Bensulfuron methyl

Methods of analysis

8. *Determination of rice herbicide candidate DPX-F5384 in rice grain. Slates R. V., 1986 (AMR-132-83 Amendment 1).*

Cross reference IIA; 4.2.1./06

Guidelines:

Not specified

Deviations: Not applicable

GLP:

No

Principle of the method:

Same principle as the method described in Du Pont AMR-132-83 (Slates, R. V., 1983b).

Conclusion:

see conclusion in point 7: Du Pont AMR-132-83 (Slates, R. V., 1983b).

9. *Determination of rice herbicide candidate DPX-F5384 in rice grain. Slates R.V., 1988 (AMR-132-83 Addendum).*

Cross reference IIA, 4.2.1./07

Findings:

Study contains attachment to validation data; in particular, chromatograms in support of Residue Method AMR-132-83 for registration purposes.

Comment: The chromatograms confirm the poor separation of the test substance from matrix constituents, resulting in poor chromatographic resolution of the DPX-F5384 peak (poor specificity; peak tailing).

10. *Determination of residues of DPX-F5384 in rice grain, straw, water and paddy soil. Validation of the method Cicotti M., 1990 (Study No. BE-A-11-89-21-BG).*

Cross reference IIA, 4.2.1./02

Guidelines:

Not specified

Deviations: Not applicable

GLP:

Yes

Principle of the method:

This study was carried out according to the following residues analytical methods:

For rice grain: Analytical Method for Determination of the Rice Herbicide Candidate DPX-F5384 in Rice Grain elaborated by R.V. Slates, Du Pont Study No. AMR-132-83, Amendment 1, May 31, 1988. For the rice grain no

Rapporteur Member State	Month and year	Active Substance (Name)
Italy	May 2006	Bensulfuron methyl

Methods of analysis

major method modification were made. The extraction and determination was carried out according to the above mentioned methodology. As sample, commercial polished rice was used.

For rice straw: Analytical Method for Determination of the Rice Herbicide Candidate DPX-F5384 in Rice Straw elaborated by R.V. Slates, Du Pont Study No. AMR-163-83, Amendment 1, May 31, 1988. The method was used without any modifications. Italian rice straw from control field was used for the validation.

For water: Determination of Rice Herbicide Candidate DPX-F5384 in Natural Water elaborated by R.V. Slates, Document AMR-232-84. The extraction procedure was used without any modifications. Additional C18 Bond Elute® cartridges cleanup procedure was used, as described in the soil procedure (see below), in order to eliminate some interferences.

Findings:

BE-A-11-89-21-BG is an independent laboratory validation (ILV) of multiple methods, including AMR 132-83 for the analysis of bensulfuron methyl in rice grain. This method passed the ILV. The procedure for extraction and analyte purification was followed as written. The ILV data are shown in Table B.5. 1.

Although being a validation of methods, the only aspects envisaged are limited to recovery and detection limit

Conclusion:

The methods used in Study No. BE-A-11-89-21-BG (Cicotti M., 1990), are those described in Du Pont Study No. AMR-132-83, Amendment 1, Study No. AMR-163-83, Amendment 1 and Document AMR-232-84 and are all characterized by poor separation of the test substance DPX-F5384. Validation would never be attained under nowadays conditions due to insufficient specificity and the method would not be considered 'fit for purpose'.

Anyhow, analytical results obtained at that time should be considered sufficiently reliable.

11. *Determination of residues of DPX-F5384 in rice grain, straw, water and paddy soil. Zenide D., 2002.*

Cross reference IIA, 4.2.1./08

This cross reference contains an amendment to Study No. BE-A-11-89-21-BG (Cicotti M., 1990), requesting the change of the study director from Mihai Cicotti to Dinu Zenide and the transfer of original final report from Battelle archive to E.I. du Pont de Nemours - Crop Protection.

WARNING: This document forms part of an EC evaluation data package and should not be read in isolation. Registration must not be granted on the basis of this document.

Rapporteur Member State	Month and year	Active Substance (Name)
Italy	May 2006	Bensulfuron methyl

Methods of analysis

Table B.5. 1: Validation data for analytical methods for the determination of bensulfuron methyl in food of plant or animal origin

Reference (analyte)	Matrix	Fortification level (mg/kg)	Recovery mean	rate [%] range	RSD [%]	n
Du Pont-4510: Amoo and Jones, 2001	Rice Grain	0.010 ^a	88	84-93	4	5
		0.10	91	87-96	4	5
	Rice Straw	0.050 ^a	102	100-106	3	5
		0.50	94	90-96	3	5
	Rice Grain ^b	0.010 ^a	99	96-102	na	3
		0.10	78	76-79	na	2
	Rice Straw ^b	0.050 ^a	107	104-110	3	3
		0.50	89	88-90	na	2
Du Pont-5592: Powley, 2003	Rice Grain	0.010 ^a	81	68-95	11	5
		0.10	80	69-91	12	5
Du Pont-9208: Weber and Hornshuh, 2003	Rice Grain ^c	0.010 ^a	104	91-113	8	7
		0.10	98	94-102	4	4
	Rice Straw ^c	0.050	105	98-110	4	7
		0.50	94	84-104	11	4
AMR 2722-93: Zhou, 1993	Rice Grain	0.010 ^a	98	95-100	2	4
		0.050	88	83-95	7	4
		0.25	101	98-106	3	4
	Rice Straw	0.050 ^a	106	100-112	5	4
		0.50	95	93-97	2	4
		1.0	100	92-111	8	4
	Rice Straw ^d	0.20	85	73-105	11	14
AMR 132-83 Rev. No. 1: Slates, 1988	Rice Grain	0.020 ^a	96	Not Available	8	16
		0.040	(Overall)		(Overall)	
		0.080				
	Rice Grain ^e	0.020 ^a	103	na	na	na
BE-A-11-89-21-BG: Cicotti, 1990	Rice Grain	0.020 ^a	73	69-78	7	3
		0.10	86	78-93	9	3
		0.020 ^a	79	73-86	8	3
		0.050	78	70-86	na	2
		0.10	85	84-86	na	2
	Rice Grain	0.20	88	87-88	na	2
		Overall	82	70-88	8	9

^a Limit of quantification, defined by a signal-to-noise ratio of approximately 10 and the lowest validated fortification level

^b Confirmatory analyses.

^c Summary of validation and concurrent fresh fortification recoveries (Table B.5. 2 and Table B.5. 3).

^d Summary of fresh and aged fortification recoveries from Du Pont Report AMR 3056-94.

^e Summary of concurrent fresh fortification recovery from Du Pont Report BE-A-11-92-09-BG.

^f Summary of concurrent fresh fortification recoveries from Du Pont Report AMR 4162-96.

na Not applicable

B.5.2.2 Analytical methods for the determination of residues in foodstuffs of plant and animal origin, feedingstuffs

A method for the determination of bensulfuron methyl in foodstuffs of animal origin is not submitted. Due to the absence of residues in feedingstuffs, residues cannot occur in products of animal origin. Therefore, MRL's do not need to be set for these animal products, and enforcement methods are not necessary.

Animal matrices

This data requirement is not relevant for bensulfuron methyl in foodstuffs of animal origin. The EU Guidance Document on Residue Analytical Methods (SANCO/825/00 rev.6, 20/06/00) requires methods for foodstuffs of animal origin to be developed and submitted for active substance and relevant metabolites unless it can be justified

Rapporteur Member State	Month and year	Active Substance (Name)
Italy	May 2006	Bensulfuron methyl

Methods of analysis

that ruminant and poultry metabolism studies would not be required, and that an MRL will not be established. When bensulfuron methyl is used according to the recommended GAP, residues do not occur in animal feed items at levels >0.1 mg/kg. Therefore, no animal metabolism studies would be required, and an MRL for foodstuffs of animal origin is not necessary. Furthermore, no enforcement methods, independent laboratory validations or confirmatory methods are submitted for bensulfuron methyl in foodstuffs of animal origin.

B.5.3 Analytical methods (residue) soil, water, air (Annex IIA 4.2.2 to IIA 4.2.4; Annex IIIA 5.2)

B.5.3.1 Residues in soil

12. *Analytical methods for the determination of 13 Du Pont sulfonylurea herbicides in soil using LC/MS/MS. Hill S.J., Stry J.J., 2001 (DuPont-5082, Revision No. 1).*

Cross reference IIA, 4.2.2./01

Guidelines:

OPPTS 850.7100, SANCO/825/00 Rev.6 (June 20, 2000)

Deviations: Not applicable

GLP:

Yes

Principle of the method:

This is a multianalyte method for the analysis of bensulfuron methyl and 12 other sulfonylurea herbicides from soil using a 0.1 M ammonium carbonate/methanol (9/1, v/v) solution and a wrist action shaker. The sample extracts are purified and concentrated on an OasisTM HLB solid-phase extraction cartridge (SPE) with multiple washes followed by analyte elution using acetonitrile and ethyl acetate. The extracts are evaporated to approximately 1 mL, diluted with 2 mL of water, and evaporated again until only the water layer is remaining (less than 2 mL). The water layer is acidified and partitioned three times with ethyl acetate. The ethyl acetate layers are collected and evaporated to dryness. The extracts are reconstituted in methanol and diluted with an aqueous buffered solution. The purified sample extracts are analysed by reversed-phase liquid chromatography and were detected by tandem mass spectrometric (MS/MS) analysis.

Findings:

Specificity: The limit of quantification of the method proposed for monitoring bensulfuron methyl residues in soil is 0.050 µg/kg. Analysis of control samples of six different soil types, including a soil with high organic matter, resulted in no detectable, apparent residues of bensulfuron methyl. Due to the selective nature of the detection of this method (two individual ion transitions monitored), interference peaks were not observed at the retention time of the analytes. As a result of the selective detection used, interference testing is not necessary for this method.

Recovery: The fortification data reported in the method proposed for monitoring bensulfuron methyl residues in soil is summarised in Table B.5. 2 below. The average recoveries and relative standard deviation are well within the criteria specified in the EU Guidance Document on Residue Analytical Methods (SANCO/825/00 Rev.6, June 20, 2000).

Rapporteur Member State	Month and year	Active Substance (Name)
Italy	May 2006	Bensulfuron methyl

Methods of analysis

Extraction efficiency: The extraction solvent used was selected after reviewing previous SU herbicide soil methods and extraction efficiency studies. Ammonium carbonate/methanol was selected because it is a rigorous extraction solvent for soil. Based on previous extraction efficiency studies, the extraction used here is equivalent to previous SU herbicide methods.

Linearity: Calibration curves for external standards across a range of 0.20 to 5.0 ng/mL were linear with correlation coefficients (R^2) greater than 0.99. This range of standard concentrations corresponds to 0.5 to 12.5 times the LOQ concentration in final extracts. Intercepts obtained by linear regression correspond to levels less than 20% of the LOQ. The response of the MS/MS detector was linear over the range of standards analyzed, as evidenced by relative standard deviation of the response factors consistently being less than 20%.

Repeatability: Repeatability of the method is addressed by the data in Table B.5. 2. The same analyst obtained these recovery data from 6 different soils in 2 separate sets per soil type, each acquired on different day. Relative standard deviations of less than 20% were obtained for fortifications made at the quantification limit for each matrix, as well as at higher levels. Therefore, the repeatability of this method is adequate for the purposes of residue data collection.

Limit of Quantification: The limit of quantification of the method for soil is 0.05 µg/kg (ppb). The lowest level of biological toxicity to the most sensitive non-target plant species is above this level. Therefore, this method is sufficiently sensitive for regulatory purposes.

Confirmatory Method: No confirmatory method is specified because the detection technique used in this method is LC/MS/MS, which, according to the EU Guidance Document on Residue Analytical Methods (SANCO/825/00 Rev.6, June 20, 2000), is sufficiently specific so that confirmatory methods are not required.

Conclusion:

The method described in Hill S.J., Stry J.J., 2001 (DuPont-5082, Revision No. 1) is considered acceptable for the determination of bensulfuron methyl in soil. However, the LC/MS/MS coupling is still not everywhere considered as commonly available equipment.

13. *Method validation for the determination of metsulfuron methyl and bensulfuron methyl residues in soil and water. Zenide D., Leka A., 1997 (Study No. A-II-95-27).*

Cross reference IIA, 4.2.2./02

Guidelines:

Not applicable

Deviations: Not applicable

GLP:

Yes

Principle of the method:

This is a multianalyte method for the analysis of bensulfuron methyl and 1 other sulfonylurea herbicide. Bensulfuron methyl is extracted from soil using an aqueous 100 mM ammonium carbonate solution and a wrist action shaker. The residues are partitioned from aqueous extracts into toluene under acidic conditions and back into water under basic conditions. The sample extracts are further purified by solid-phase extractions using C₁₈ and silica cartridges.

Rapporteur Member State	Month and year	Active Substance (Name)
Italy	May 2006	Bensulfuron methyl

Methods of analysis

Residues of bensulfuron methyl in soil extracts were determined by normal-phase HPLC with photoconductivity detection. The residue data reported in Du Pont study AMR 4162-96 were determined using this method procedure.

Findings:

Specificity: The limit of quantification of the method proposed for monitoring bensulfuron methyl residues in soil is 1.0 µg/kg. This method was applied only to soil taken from northern Italy for method validation and field sample analysis in Du Pont study AMR 4162-96.

Recovery: The fortification data reported in the method proposed for monitoring bensulfuron methyl residues in soil is summarised in Table B.5. 2 below. The average recoveries and relative standard deviation are within the criteria specified in the EU Guidance Document on Residue Analytical Methods (SANCO/825/00 Rev.6, June 20, 2000).

Extraction efficiency: This method procedure was evaluated in Du Pont method AMR 2480-92 by aging 5 sulfonylurea herbicides in high clay soil (worse case) for a minimum of 12 hours prior to extraction. Acceptable recoveries (70-110%) reported from this experiment, and similar hydrophilic and octanol-water partitioning properties for all sulfonylurea herbicides, support the satisfactory extraction efficiency of this method for bensulfuron methyl in soil.

Linearity: Calibration curves for external standards across a range of 0.020 to 0.25 µg/mL were linear with correlation coefficients (R^2) greater than 0.99. This range of standard concentrations corresponds to 1 to 10 times the LOQ concentration in final extracts. Intercepts obtained by linear regression correspond to levels less than 20% of the LOQ.

Repeatability: Repeatability of the method is addressed by the data in Table B.5. 2. The recovery data are from the original method validation and concurrent fortification samples in Du Pont AMR 4162-96 field study. Relative standard deviations of less than 20% were obtained for fortifications made at the quantification limit, as well as at higher levels. Therefore, the repeatability of this method is adequate for the purposes of residue data collection.

Limit of Quantification: The limit of quantification of the method for soil is 1.0 µg/kg (ppb). The lowest level of biological toxicity to the most sensitive non-target plant species is above this level. Therefore, this method is sufficiently sensitive for regulatory purposes.

Confirmatory Method: The method Du Pont 5082 Revision No. 1 provides method confirmation.

Table B.5. 2: Validation data for analytical methods for the determination of bensulfuron methyl in soil

Reference (analyte)	Matrix	Fortification level (µg/kg)	Recovery mean	rate [%] range	RSD [%]	n
Du Pont-5082 Revision No. 1: Hill and Stry, 2001	Soils (6)	0.050 ^a	91	70-107	11	42
		0.50	90	66-110	13	38
A-11-95-27: Zenide and Leke, 1997	Soil	1.0 ^a	83	70-100	18	3
		5.0	83	73-93	na	2
		10.0	80	74-88	9	3
	Soils ^b (3 sites)	5.0	82	61-102	12	29
		10.0	84	55-104	11	30

^a Limit of quantification, defined by a signal-to-noise ratio of approximately 10 and the lowest validated fortification level

^b Concurrent sample fortification data from Du Pont Report AMR 4162-96.

Rapporteur Member State	Month and year	Active Substance (Name)
Italy	May 2006	Bensulfuron methyl

Methods of analysis

Conclusion:

The method described in Zenide D., Leka A., 1997 (Study No. A-II-95-27) for the determination of the a.s. in soil is characterized by insufficient separation of the test substance DPX-F5384 from matrix constituents is attained during cleaning steps, resulting in poor chromatographic resolution of the DPX-F5384 peak. Validation would never be attained under nowadays conditions due to insufficient specificity and the method would not be considered 'fit for purpose'.

Anyhow, analytical results obtained at that time should be considered sufficiently reliable.

B.5.3.2 Residues in water (include drinking water, ground water and surface water)

14. *Analytical method for the determination and confirmation of 13 Du Pont sulfonylurea herbicides in water using LC/MS/MS. Devine T.J., Jin L., 2004 (DuPont-5491, Revision No. 1).*

Cross reference IIA, 4.2.3./01

Guidelines:

SANCO/825/00 Rev.6, June 20, 2000; OPPTS 850.7100

Deviations: Not applicable

GLP:

No

Principle of the method:

This a multianalyte method for the analysis of bensulfuron methyl and 12 other sulfonylurea herbicides from water. In this method, residues of bensulfuron methyl are extracted from water (tap, well, pond, river, and ocean), purified, and concentrated by solid-phase extraction using an ENV cartridge. The final eluate was evaporated to dryness and reconstituted in 5 mM ammonium acetate buffer. Bensulfuron methyl was separated from co-extractants by reversed-phase HPLC and were detected by tandem mass spectrometry (MS/MS). This method is intended to be used for enforcement purposes.

Findings:

Specificity: The limit of determination of the method proposed for monitoring bensulfuron methyl residues in water is 0.05 µg/L. Analysis of control samples of 5 different water sources, including pond water with a high algae content, resulted in no detectable apparent residues of bensulfuron methyl. Due to the selective nature of the LC/MS/MS analysis of this method (two individual ion transitions monitored) and absence interference peaks at the retention time of bensulfuron methyl, interference testing is not necessary for this method.

Recovery: The fortification data reported in the method proposed for monitoring chlorsulfuron residues in water is summarised in Table B.5. 3. The average recoveries and relative standard deviation are well within the criteria specified in the EU Guidance Document on Residue Analytical Methods (SANCO/825/00 Rev.6, June 20, 20002).

Linearity: Calibration curves for external standards across a range of 0.50 to 10.0 ng/mL were linear with correlation coefficients (R^2) greater than 0.99. This range of standard concentrations corresponds to approximately 0.5 to 12.5 times the LOQ concentration in final extracts. Intercepts obtained by linear regression correspond to levels less than 20% of the LOQ.

Rapporteur Member State	Month and year	Active Substance (Name)
Italy	May 2006	Bensulfuron methyl

Methods of analysis

Repeatability: Repeatability of the method is addressed by the data in Table B.5. 3. The same analyst obtained these recovery data from 5 different water sources in 2 separate sets per water source, each acquired on different day. Relative standard deviations of less than 20% were obtained for fortifications made at the quantification limit for each matrix, as well as at higher levels. Therefore, the repeatability of this method is adequate for the purposes of detecting bensulfuron methyl residues in drinking and surface waters of agricultural relevance.

Limit of Quantification: The limit of quantification of the method for water is 0.050 µg/L. The EU guidelines specify that the drinking water limit of quantification must be equal or lower than 0.1 µg/L (EU drinking water limit). Therefore, the sensitivity of this method is sufficient to meet the guidelines as stated by the EU Guidance Document on Residue Analytical Methods (SANCO/825/00 Rev.6, June 20, 2000).

Confirmatory Method: No confirmatory method is specified because the detection technique used in this method is LC/MS/MS, which, according to the EU Guidance Document on Residue Analytical Methods (SANCO/825/00 Rev.6, June 20, 2000), is sufficiently specific so that confirmatory methods are not required.

Conclusion:

The method described in Devine T.J., Jin L., 2004 (DuPont-5491, Revision No. 1) is considered acceptable for the determination of bensulfuron methyl in water. However, the LC/MS/MS coupling is still not everywhere considered as commonly available equipment.

15. *Method validation for the determination of bensulfuron methyl and bensulfuron methyl residues in soil and water. Zenide D., Leka A., 1997.*

Cross reference IIA, 4.2.3./02

Guidelines:

Not applicable

Deviations: Not applicable

GLP:

Yes

Principle of the study:

This is a multianalyte method for the analysis of bensulfuron methyl and 1 other sulfonylurea herbicide. Water samples are acidified to pH 3-3.5 with dilute phosphoric acid and the bensulfuron methyl residue is partitioned into toluene. The residue is back extracted from the toluene phase into aqueous 1 mM ammonium hydroxide. The aqueous fraction pH is adjusted to 3-3.5 with dilute phosphoric acid and the extracts are purified by SPE using C18 and silica cartridges. Bensulfuron methyl residue in water extract was determined by normal-phase HPLC with photoconductivity detection. The residue data reported in Du Pont study AMR 4162-96 were determined using this method procedure.

Findings:

Specificity: The limit of quantification of the method proposed for monitoring bensulfuron methyl residues in water is 0.10 µg/L. This method was applied only to paddy water taken from northern Italy for method validation and field sample analysis in Du Pont study AMR 4162-96.

Rapporteur Member State	Month and year	Active Substance (Name)
Italy	May 2006	Bensulfuron methyl

Methods of analysis

Recovery: The fortification data reported in the method proposed for monitoring bensulfuron methyl residues in soil is summarised in Table B.5. 3 below. The average recoveries and relative standard deviation are within the criteria specified in the EU Guidance Document on Residue Analytical Methods (SANCO/825/00 Rev.6, June 20, 2000).

Linearity: Calibration curves for external standards across a range of 0.020 to 0.25 µg/mL were linear with correlation coefficients (R^2) greater than 0.99. This range of standard concentrations corresponds to 1 to 10 times the LOQ concentration in final extracts. Intercepts obtained by linear regression correspond to levels less than 20% of the LOQ.

Repeatability: Repeatability of the method is addressed by the data in Table B.5. 3. The recovery data are from the original method validation and concurrent fortification samples in Du Pont AMR 4162-96 field study. Relative standard deviations of $\leq 20\%$ were obtained for fortifications made at the quantification limit, as well as at higher levels. Therefore, the repeatability of this method would seem to be adequate for the purposes of detecting bensulfuron methyl residues in water.

Limit of Quantification: The limit of quantification of the method for water is 0.10 µg/L (ppb). The lowest level of biological toxicity to the most sensitive non-target plant species is above this level. Therefore, this method is sufficiently sensitive for regulatory purposes.

Confirmatory Method: The method Du Pont-5491 Revision No. 4 provides method confirmation.

Table B.5. 3: Validation data for analytical methods for the determination of bensulfuron methyl in water

Reference (analyte)	Matrix	Fortification level (mg/kg)	Recovery mean	rate [%] range	RSD [%]	n
Du Pont-5491 Revision No. 1 Devine and Jin, 2004 (bensulfuron methyl)	Tap Water	0.050 ^a	101	85-120	14	5
		0.50	101	88-119	15	5
	Well water	0.05 ^a	94	83-100	8	5
		0.5	94	91-98	3	5
	Pond water	0.05 ^a	98	89-107	8	5
		0.5	101	97-106	3	5
	River water	0.05 ^a	105	98-115	6	5
		0.5	100	94-104	4	5
	Ocean Water	0.05 ^a	99	93-107	5	5
		0.5	99	93-105	5	5
A-11-95-27: Zenide and Leke, 1997	Paddy Water	0.10	75	75, 75	na	2
		0.50	96	74-108	20	3
		1.0	102	95-111	8	3
	Paddy Water ^b (3 sites)	0.5	109	89-138	11	12
		5.0	95	80-130	14	12

^a Limit of quantification, defined by a signal-to-noise ratio of approximately 10 and the lowest validated fortification level

^b Concurrent sample fortification data from Du Pont Report AMR 4162-96.

na not applicable

Conclusion:

The method described in Zenide D., Leka A., 1997 (Study No. A-11-95-27) for the determination of the a.s. in water is characterized by poor chromatographic resolution of the DPX-F5384 peak. Validation would never be attained under nowadays conditions due to insufficient specificity and the method would not be considered 'fit for purpose'.

Anyhow, analytical results obtained at that time should be considered sufficiently reliable.

Rapporteur Member State	Month and year	Active Substance (Name)
Italy	May 2006	Bensulfuron methyl

Methods of analysis

B.5.3.3 Residues in air

16. *Development and validation of analytical methods for the determination of seven sulfonylurea herbicides in air (amended). Bacher R., 2001 (DuPont-4560).*

Cross reference IIA, 4.2.4./01

Guidelines:

Directive 91/414/EEC; SANCO/825/00 Rev. 6 June 20, 2000

Deviations: Not applicable

GLP:

Yes

The study is

Principle of the method:

A multianalyte LC/MS/MS method was developed to monitor residues of bensulfuron methyl and 6 other sulfonylurea herbicides in air. In this method, residues of bensulfuron methyl were extracted from air with XAD-2 porous polymer filters. Bensulfuron residues were extracted from the filters with acetonitrile. After rotary evaporation, the extracts were analysed by LC/MS/MS using electrospray ionisation in the positive ion mode on an ion trap mass spectrometer.

Findings:

Specificity: The limit of quantification of this method used to determine bensulfuron methyl residues trapped on filters from air is 0.3 µg/m³ based on a 0.5 m³ air volume sample. Analysis of control samples showed no detectable bensulfuron methyl residue. Due to the selective nature of the LC/MS/MS analysis of this method (three individual ion transitions monitored) and absence interference peaks at the retention time of bensulfuron methyl; interference testing is not necessary for this method.

Recovery: The bensulfuron methyl fortification data for air obtained during validation of the method is summarised in Table B.5. 4 below. The average recovery of bensulfuron from the XAD-2 filters (97%) is within the acceptance criteria of 70-110% for both ambient and warm, humid air, and confirms this method is adequate for the purpose of determining bensulfuron residues trapped on XAD-2 filters from air.

Linearity: Calibration curves for external standards across a range of 0.015 to 3.0 ng/mL were linear with correlation coefficients (R²) greater than 0.99. This range of standard concentrations corresponds to 0.1 to 20 times the LOQ concentration in final extracts. Intercepts obtained by linear regression correspond to levels less than 20% of the LOQ.

Repeatability: Repeatability of this method was demonstrated by the standard deviation of the recovery values given in Table B.5. 4. The maximum relative standard deviation of 8% for recovery data is within the guideline of ≤20%, for both ambient and warm, humid air. This method is adequate for determining bensulfuron residues trapped on XAD-2 filters from air.

Limit of Quantification: The limit of quantification for bensulfuron methyl trapped on XAD-2 filters from air is 3.0 µg/m³ based on a 0.5 m³ air volume sample. The limit of quantification is well below the estimated acute inhalation

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no-observable-adverse effect level concentration of 7.5 µg/m³ in the rat. This method is capable of determining bensulfuron methyl well below toxicologically significant levels in air.

Confirmatory Method: No confirmatory method is specified because the detection technique used in this method is LC/MS/MS, which, according to the EU Guidance Document on Residue Analytical Methods (SANCO/825/00 Rev.6, June 20, 2000), is sufficiently specific so that confirmatory methods are not required.

Table B.5. 4: Validation data for analytical methods for the determination of bensulfuron methyl in air

Reference (analyte)	Matrix	Fortification level (µg/m ³)	Recovery mean	rate [%] range	RSD [%]	n
Du Pont-4560	Ambient air	3.0 ^a	97	87-104	6	5
Amended Report		30	89	85-97	5	5
Bacher, 2001	Warm,	3.0 ^a	90	81-100	8	5
(bensulfuron methyl)	humid air	30	108	105-111	2	5

^a Limit of quantification, defined by a signal-to-noise ratio of approximately 10 and the lowest validated fortification level

Conclusion:

The method described in Bacher R., 2001 (DuPont-4560) is considered acceptable for the determination of bensulfuron methyl in air. However, the LC/MS/MS coupling is still not everywhere considered as commonly available equipment.

B.5.4 Analytical methods (residue) for body fluids and tissues (Annex IIA 4.2.5; Annex IIIA 5.2)

B.5.4.1 Residues in body fluids and tissues (Annex IIA 4.2.5)

This data requirement is not relevant for bensulfuron methyl. Bensulfuron methyl is not classified as highly toxic or toxic to humans and would not bioaccumulate in human tissues in case of accidental contact or ingestion. Therefore, methods for the determination of bensulfuron methyl in body fluids and tissues would be of no practical use.

B.5.5 Summary and evaluation of studies on analytical methods

All recently developed and validated analytical methods are considered adequate for the determination of bensulfuron methyl (DPX-F5384) in the technical substance, plant protection product, rice grain and rice straw, soil, water and air. Given the revised definition of residue in soil (AE F082671/parent, IN-J0290, IN-N5297, IN-D1R84, IN-R9419), in surface water (AE F082671/parent, IN-J0290, IN-N5297, IN-R9419) and in drinking/groundwater (AE F082671/parent, IN-N5297, IN-T5831, IN-DAT97) a request will follow for the submission of suitable analytical methods for the determination of these metabolites. Adequate methods exist for the quantitation of impurities (process residual solvents, water, and inorganic ions) in the active substance as manufactured. The inert ingredients contained in the plant protection product (bensulfuron methyl 60WG) are not expected to interfere with the extraction and analysis of bensulfuron methyl residues in any matrix. Therefore, no residue methods have been developed specifically for the plant protection product.

Methods for the determination of bensulfuron methyl in foodstuffs of animal origin were not submitted due to the absence of residues in feedingstuffs. Also, no method for the determination of bensulfuron methyl or metabolites in body fluids and tissues were submitted since no of them has been classified as highly toxic or toxic to humans and is not expected to accumulate in human tissues in case of accidental contact or ingestion.

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Analytical methods considered suitable for use by regulatory agencies for monitoring, confirmatory and enforcement purposes are resumed in Table B.5. 5.

Table B.5. 5: Proposed analytical methods for monitoring bensulfuron methyl residues

Matrix	Reference and report	Separation / Quantitation	Limit of determination	Comments
Technical material	Du Pont-7095 Morgan E.A., 2002	LC/UV GC/FID	Active substance and impurities >1g/kg	Method validated for quantitative analysis by LC/UV at 236nm (a.s.) and 230nm (impurities) GC/FID for the determination of solvents (Confidential Attachment)
Plant Protection Product	Du Pont-7095 Morgan E.A., 2002	LC/UV	Active substance	No specific method developed since inert ingredients contained in p.p.p. not expected to interfere with extraction and analysis of the a.s..
Rice Grain Rice Straw	Du Pont-4510: Amoo and Jones, 2001	LC/UV LC/MS LC/MS/MS	0.010 mg/kg 0.050 mg/kg	Method validated for quantitative analysis by LC/UV with column switching, LC/MS, or LC/MS/MS
Animal Products				No method required since residues are not expected in food from animal origin
Soil	Du Pont-5082 Rev. No. 1: Hill and Stry, 2001	LC/MS/MS	0.050 µg/kg	Multiple sulfonylurea herbicides analysis method.
Water	Du Pont-5491 Revision No. 1: Devine and Jin, 2004	LC/MS/MS	0.050 µg/L	Multiple sulfonylurea herbicides analysis method.
Air	Du Pont-4560 Amended Report: Bacher, 2001	LC/MS/MS	0.30 µg/m ³	Multiple sulfonylurea herbicides analysis method.
Body fluids and tissues				No method required (low acute toxicity of a.s. and metabolites, accumulation in tissues not expected)

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B.5.6 References relied on

Annex No., EU Data Requirement No., Reference No.	Author(s)	Year	Title Source Company Report No. GLP or GEP Status (where relevant) Published or not	EU Data Protection Claimed (Y/N)	Owner
IIA, 4.1.1./01	Morgan, E.A.	2002	Bensulfuron methyl (DPX-F5384): Analysis and certification of product ingredients for technical grade material Produced at the Manati and Shanghai manufacturing facilities. Du Pont Stine-Haskell Research Center and Chambers Works Du Pont-7095 (Non confidential) GLP, unpublished	Y	Du Pont
IIA, 4.1.2./01	Morgan, E.A.	2002	Bensulfuron methyl (DPX-F5384): Analysis and certification of product ingredients for technical grade material produced at the Manati and Shanghai manufacturing facilities Du Pont Stine-Haskell Research Center and Chambers Works Du Pont-7095, (Confidential) GLP, unpublished	Y	Du Pont
IIA, 4.2.1./01	Amoo, J.S., Jones, W.	2001	Analytical enforcement method for the determination of bensulfuron methyl in rice (grain and straw) using column- switching liquid chromatography with ultraviolet detection Du Pont Stine-Haskell Research Center Du Pont-4510 Not applicable GLP, unpublished	Y	Du Pont
IIA, 4.2.1./02	Cicotti, M	1990	Determination of residues of DPX-F5384 in rice grain, straw, water and paddy soil Battelle Europe - Geneva Research Centres BE-A-11-89-21-BG GLP, unpublished	Y	Du Pont
IIA, 4.2.1./03	Hornshuh, M.J.	2003	Magnitude of residues of bensulfuron methyl in rice (cereals) following application of bensulfuron methyl (DPX- F5384) 60WG formulation in wetland rice fields - Southern Europe, Season 2002 Du Pont Experimental Station Du Pont-9208 GLP, unpublished	Y	Du Pont
IIA, 4.2.1./04	Powley, C.R.	2003	Independent laboratory validation of Du Pont-4510, "Analytical enforcement method for the determination of bensulfuron methyl in rice (grain and straw) using column-switching liquid chromatography with ultraviolet detection" on rice grain Du Pont Stine-Haskell Research Center Du Pont-5592 GLP, unpublished	Y	Du Pont

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Annex No., EU Data Requirement No., Reference No.	Author(s)	Year	Title Source Company Report No. GLP or GEP Status (where relevant) Published or not	EU Data Protection Claimed (Y/N)	Owner
IIA, 4.2.1./05	Slates, R. V.	1983b	Determination of rice herbicide candidate DPX-F5384 in rice grain Du Pont Experimental Station AMR 132-83, Revision No. 1 Not GLP, unpublished	N	Du Pont
IIA, 4.2.1./06	Slates, R. V.	1988	Determination of rice herbicide candidate DPX-F5384 in rice grain Du Pont Experimental Station AMR 132-83, Amendment No. 1 Not GLP, unpublished	N	Du Pont
IIA, 4.2.1./07	Slates, R. V.	1986	Determination of rice herbicide candidate DPX-F5384 in rice grain Du Pont Experimental Station AMR 132-83, Addendum No. 1 Not GLP, unpublished	N	Du Pont
IIA, 4.2.1./08	Zenide, D.	2002	Determination of residues of DPX-F5384 in rice grain, straw, water and paddy soil Battelle Europe - Geneva Research Centres BE-A-11-89-21-BG Amendment No. 1 GLP, unpublished	Y	Du Pont
IIA, 4.2.1./09	Zhou, M.	1993	Analytical method for the determination of bensulfuron methyl residues in rice grain and rice straw by HPLC using a column and eluent switching system Du Pont Experimental Station AMR 2722-93 Not applicable GLP, unpublished	Y	Du Pont
IIA, 4.2.2./01	Hill, S.J., Stry, J.J.	2001	Analytical methods for the determination of 13 Du Pont sulfonylurea herbicides in soil using LC/MS/MS. Du Pont Stine-Haskell Research Center Du Pont-5082, Revision No. 1 GLP, unpublished	Y	Du Pont
IIA, 4.2.2./02	Zenide, D., Leka, A.	1997	Method Validation for the determination of metsulfuron methyl and bensulfuron methyl residues in soil and water Battelle Europe - Geneva Research Centres A 11-95-27 GLP, unpublished	Y	Du Pont
IIA, 4.2.3./01	Devine, T.J., Jin, L.	2004	Analytical method for the determination and confirmation of 13 Du Pont sulfonylurea herbicides in water using LC/MS/MS Du Pont Stine-Haskell Research Center Du Pont-5491, Revision No. 1 Not GLP, unpublished	Y	Du Pont

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IIA, 4.2.3./02	Zenide, D., Leka, A.	1997	Method validation for the determination of metsulfuron methyl and bensulfuron methyl residues in soil and water Battelle Europe - Geneva Research Centres A 11-95-27 GLP, unpublished	Y	Du Pont
IIA, 4.2.4./01	Bacher, R.	2001	Development and validation of analytical methods for the determination of seven sulfonylurea herbicides in air (Amended) PTRL Europe Du Pont-4560 GLP, unpublished	Y	Du Pont
IIA, 5.1.1./01	Morgan, E.A.	2002	Bensulfuron methyl (DPX-F5384): Analysis and certification of product ingredients for technical grade material Produced at the Manati and Shanghai manufacturing facilities. Du Pont Stine-Haskell Research Center and Chambers Works Du Pont-7095 (Non-Confidential) GLP, unpublished	N	Du Pont