Data Evaluation Report on the Aquatic Field Dissipation of Orthosulfamuron

PMRA Submission Number {.....} EPA MRID Number 46578985

Data Requirement: PMRA Data Code:
EPA DP Barcode: D320283
OECD Data Point:
EPA Guideline: 164-2

Test material: IR5878 50WG

End Use Product name: IR5878 50WG **Concentration of a.i.:** 50%
Formulation type: Water dispersible granule

Active ingredient

Common name: Orthosulfamuron

Chemical name:

IUPAC name: 1-(4,6-Dimethoxypyrimidin-2-yl)-3-[2-(dimethylcarbamoyl)phenyl-sulfamoyl]urea.

CAS name: 2-[[[[[(4,6-Dimethoxy-2-pyrimidinyl)amino]carbonyl]amino]sulfonyl]-amino]-N,N-dimethylbenzamide.

CAS No.: 213464-77-8.

Synonyms IR5878.

Smiles string: CN(C(=O)c1ccccc1NS(=O)(=O)NC(=O)Nc1nc(cc(n1)OC)OC)C (ISIS v2.3/Universal SMILES).
No EPI Suite, v3.12 SMILES String found as of 11/21/05.

Primary Reviewer: Dan Hunt
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Company Code
Active Code
Use Site Category
EPA PC Code: 108209

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Data Evaluation Report on the field dissipation/aquatic field dissipation of orthosulfamuron

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EPA Guideline: 164-1 and 164-2

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CITATION: Cooley, T.A. 2005. IR5878 Field Dissipation Study in Bare Ground Rice Soil. Unpublished study performed by Excel Research Services, Inc., Fresno, CA and PTRL West, Inc., Hercules, CA; and sponsored and submitted by ISAGRO S.p.A., Milano, Italy. Study No.: ERS23044. PTRL West Project No.: 1204W. Experiment initiation July 15, 2003 and completion February 14, 2005. Final report issued May 19, 2005. 1013 pp.

EXECUTIVE SUMMARY

Orthosulfamuron (1-(4,6-dimethoxypyrimidin-2-yl)-3-[2-(dimethylcarbamoyl)phenyl-sulfamoyl]urea; IR5878 50WG, 50% a.i.) was applied once at a target application rate of 375 g a.i./ha (0.335 lb a.i./A; which is 500% of the normal use rate) onto a bareground plot of silt loam soil in Arkansas and a bareground plot of silty clay loam soil in California. The plots were flooded with water 1-2 days following the test application to approximate the timing of herbicide application to rice. Soil (0-60 cm depth), water, and sediment (0-5 cm depth) samples were collected for analysis of orthosulfamuron and three transformation products: IR7825 (4,6-dimethoxy-2-pyrimidinyl urea), IR8181 (1-[2(dimethylcarbamoyl)phenyl-sulfamoyl]-3-(4-hydroxy-6-methoxypyrimidin-2-yl)urea), and IR7863 (sodium (2-dimethylcarbamoylphenyl)-sulfamate). Soil samples were collected through 550-553 days posttreatment and water and sediment samples were collected from 3 days to 2 months posttreatment, at which time the water was drained from the plots.

At the Arkansas test site, orthosulfamuron dissipated in the soil, sediment, and water with In-linear half-lives of 4.1 days (r^2 = 0.9119), 6.2 days (r^2 = 0.8864), and 6.4 days (r^2 = 0.7323), respectively.

Orthosulfamuron was detected in the 0- to 15-cm soil depth at a mean concentration of 0.069 mg/kg at day 0, which is 33.7% of the theoretical amount (calculated based on a theoretical value of 0.205 mg/kg), compared to a recovery of 91.3% from the application rate monitoring devices. Orthosulfamuron decreased to 0.046 mg/kg by 1 day and 0.015 mg/kg by 7 days, and was last detected at 0.006 mg/kg (single replicate) at 15 days posttreatment. Orthosulfamuron was only detected once in the 15-30 cm soil depth, at 0.006 mg/kg (single replicate) at 1 day, and was not detected below that depth.

The major transformation product **IR7825** was initially detected in the 0-15 cm soil at 0.007 mg/kg (single replicate) at day 0, ranged from a mean of 0.023-0.026 mg/kg from 7-60 days, was a maximum of 0.049 mg/kg at 186 days, and decreased to 0.017 mg/kg by 550 days, the last sampling interval. The half-life of IR7825 in soil was 330 days (r^2 = 0.2476; based on 186-550 day data).

The transformation product **IR7863** was detected in the soil above the LOQ at 7 days (0.041-0.052 mg/kg; 2 of 3 replicates) and 22 days (0.025 mg/kg; 2 of 3 replicates).. IR7825 and IR7863 were not detected below the 0-15 cm soil depth. Mean concentrations of orthosulfamuron residues in soil are presented in Table 1 below.

Table 1. Mean concentration of orthosulfamuron residues in Arkansas soil (0-15 cm), expressed as mg/kg soil.

Days posttreatment	Average residue in the 0-15 cm soil depth (mg/kg)			
	Orthosulfamuron	IR7825	IR8181	IR7863
0	**0.069**	**0.007**[a]	ND	ND
1	**0.046**	**0.006**[a]	ND	ND
7	**0.015**	**0.026**	ND	**0.047**[b]
15	**0.006**[a]	**0.026**	ND	ND
22	<0.005	**0.025**	ND	**0.025**[b]
31	<0.005	**0.023**	ND	ND
60	<0.005	**0.026**	ND	ND
123	<0.005	**0.023**[a]	ND	--
186	--	**0.049**	--	--
273	--	**0.033**	--	--
364	--	**0.018**	--	--
462	--	**0.037**	--	--
550	--	**0.017**	--	--

Orthosulfamuron, IR7825 and IR8181: LOD = 0.0006 mg/kg and LOQ = 0.005 mg/kg. IR7863: LOD = 0.008 mg/kg and LOQ = 0.02 mg/kg.
a = single replicate value (other replicates <LOQ).
b = mean of duplicate detects (with the third replicate <LOQ).

Orthosulfamuron was detected in the sediment at a mean concentration of 0.182 mg/kg at 3 days, decreased to 0.055 mg/kg by 7 days and 0.019 mg/kg by 15 days, and was last detected at 0.008 mg/kg at 1 month posttreatment.

The major transformation product **IR7825** was detected in the sediment at 0.042 mg/kg at 3 days, increased to a maximum of 0.141 mg/kg by 15 days, and then decreased to 0.049 mg/kg by 2 months, the last sampling interval. The half-life of IR7825 in sediment was 37.1 days (r^2 = 0.4408; based on 15 day to 2 month data).

The major transformation product **IR7863** was detected in the sediment at 0.061-0.065 mg/kg from 3-7 days, was not detected at 15 days, was 0.053 mg/kg at 22 days, and was not detected at 2 months posttreatment.

Orthosulfamuron was detected in the water at a mean concentration of 0.024 mg/L at 3 days, decreased to 0.012 mg/L by 7 days, and was last detected at 0.006 mg/L at 15 days posttreatment.

The transformation product **IR7863** was detected in the water at 0.006 mg/L at 3 days, increased to a maximum of 0.011 mg/L by 15 days, and was not detected above the LOQ at 2 months posttreatment. Mean concentrations of orthosulfamuron residues in sediment and water are presented in Tables 2 and 3 below.

Table 2. Mean concentration of orthosulfamuron residues in Arkansas sediment (mg/kg soil).

Sampling interval	Concentration in sediment (mg/kg)			
	Orthosulfamuron	**IR7825**	**IR8181**	**IR7863**
3 days	**0.182**	**0.042**	ND	**0.061**
7 days	**0.055**	**0.126**	<0.005	**0.065**
15 days	**0.019**	**0.141**	<0.005	ND
22 days	**0.011**	**0.085**	<0.005	**0.053**
1 month	**0.008**	**0.062**	<0.005	**0.031**[a]
2 months	<0.005	**0.049**	ND	ND

Orthosulfamuron, IR7825 and IR8181: LOD = 0.0006 mg/kg and LOQ = 0.005 mg/kg. IR7863: LOD = 0.008 mg/kg and LOQ = 0.02 mg/kg.
a = single replicate value (other replicates <LOQ).

Table 3. Mean concentration of orthosulfamuron residues in Arkansas water (mg/L).

Sampling interval	Concentration in water (mg/L)			
	Orthosulfamuron	**IR7825**	**IR8181**	**IR7863**
3 days	**0.024**	<0.005	ND	**0.006**
7 days	**0.012**	<0.005	ND	**0.009**[a]
15 days	**0.006**[a]	<0.005	ND	**0.011**[a]
22 days	<0.005	<0.005	ND	**0.009**
1 month	<0.005	<0.005	<0.005	**0.006**[a]
2 months	<0.005	<0.005	ND	<0.005

Orthosulfamuron, IR7825 and IR8181: LOQ = 0.005 mg/L. IR7863: LOQ = 0.02 mg/L.
a = mean from replicates with a detection.

At the California test site, orthosulfamuron dissipated in the soil and sediment with ln-linear half-lives of 2.3 days (r^2 = 0.8678) and 4.3 days (r^2 = 0.438), respectively. A half-life for orthosulfamuron in water could not be calculated because the chemical was not detected in the water.

Orthosulfamuron was detected in the 0- to 15-cm soil depth at a mean concentration of 0.094 mg/kg at day 0, which is 45.9% of the theoretical amount (based on a theoretical value of 0.205

mg/kg), compared to a recovery of 86.6% from the application rate monitoring devices. Orthosulfamuron decreased to 0.031 mg/kg (single replicate) by 3 days and was last detected at 0.013 mg/kg at 7 days posttreatment.

The major transformation product **IR7825** was initially detected in the 0-15 cm depth soil at 0.008 mg/kg at 1 day, increased to a maximum of 0.040 mg/kg by 194 days, and decreased to 0.021-0.036 mg/kg by 284-372 days, and was 0.024 mg/kg at 553 days, the last sampling interval. The half-life of IR7825 in soil was 178 days (r^2 = 0.0039; based on 194-553 day data). Orthosulfamuron and IR7825 were not detected in soil below the 0-15 cm depth. Mean concentrations of orthosulfamuron residues in soil are presented in Table 4 below.

Table 4. Mean concentration of orthosulfamuron residues in California soil (0-15 cm), expressed as mg/kg soil.

Days posttreatment	Average residue in the 0-15 cm soil depth (mg/kg)			
	Orthosulfamuron	IR7825	IR8181	IR7863
0	**0.094**	<0.005	ND	<0.02
1	**0.095**	**0.008**	ND	<0.02
3	**0.031**[a]	**0.012**[a]	ND	<0.02
7	**0.013**	**0.022**	ND	<0.02
15	<0.005	**0.019**	ND	ND
31	<0.005	**0.017**[b]	ND	ND
62	ND	**0.010**	ND	ND
122	ND	**0.031**	ND	ND
194	<0.005	**0.040**	ND	ND
284	--	**0.021**	--	--
372	--	**0.036**	--	--
464	--	**0.015**[a]	--	--
553	--	**0.024**	--	--

Orthosulfamuron, IR7825, and IR8181: LOD = 0.0006 mg/kg and LOQ = 0.005 mg/kg. IR7863: LOD = 0.008 mg/kg and LOQ = 0.02 mg/kg.
a = single replicate value (other replicates <LOQ).
b = mean of duplicate detects (with the third replicate <LOQ).

Orthosulfamuron was detected in the sediment at a mean concentration of 0.062 mg/kg at 3 days, decreased to 0.050 mg/kg by 7 days, and was last detected at 0.007 mg/kg (single replicate) at 15 days posttreatment.

The major transformation product **IR7825** was detected in the sediment at 0.028 mg/kg at 3 days, increased to a maximum of 0.100 mg/kg by 7 days, and then decreased to 0.033 mg/kg by

15 days and 0.017 mg/kg by 2 months, the last sampling interval. The half-life of IR7825 in sediment was 29.6 days (r^2 = 0.3627; based on 7 day to 2 month data).

Orthosulfamuron, IR7825, IR8181, and IR7863 were not detected in the water above the LOQ at any sampling intervals. Mean concentrations of orthosulfamuron residues in sediment and water are presented in Tables 5 and 6 below.

Table 5. Mean concentration of orthosulfamuron residues in California sediment (mg/kg soil).

Sampling interval	Concentration in sediment (mg/kg)			
	Orthosulfamuron	IR7825	IR8181	IR7863
3 days	**0.062**	**0.028**	ND	<0.02
7 days	**0.050**	**0.100**	ND	<0.02
15 days	**0.007**[a]	**0.033**	ND	ND
1 month	<0.005	**0.023**[b]	ND	ND
2 months	ND	**0.017**	ND	ND

Orthosulfamuron, IR7825 and IR8181: LOD = 0.0006 mg/kg and LOQ = 0.005 mg/kg. IR7863: LOD = 0.008 mg/kg and LOQ = 0.02 mg/kg.
a = single replicate value (other replicates <LOQ).
b = mean of duplicate detects (with the third replicate <LOQ).

Table 6. Mean concentration of orthosulfamuron residues in California water (mg/L).

Sampling interval	Concentration in water (mg/L)			
	Orthosulfamuron	IR7825	IR8181	IR7863
3 days	<0.005	<0.005	ND	ND
7 days	<0.005	<0.005	<0.005	ND
15 days	<0.005	<0.005	ND	<0.005
1 month	<0.005	<0.005	<0.005	ND
2 months	ND	ND	ND	ND

Orthosulfamuron, IR7825 and IR8181: LOQ = 0.005 mg/L. IR7863: LOQ = 0.02 mg/L.

Study Acceptability: This study is classified as **supplemental**. The storage stability data (from field spikes) could not demonstrate stability of the parent and transformation products for the maximum storage interval of test samples (with the exception of parent and IR7825 from the Arkansas water samples). Recoveries for IR7863 from Arkansas soil and sediment and for orthosulfamuron, IR7825, and IR7863 from California soil and sediment were low, indicating that the compounds were not stable for the storage interval.

MATERIALS AND METHODS

The aquatic field dissipation of orthosulfamuron (1-(4,6-dimethoxypyrimidin-2-yl)-3-[2-(dimethylcarbamoyl)phenyl sulfamoyl]urea; IR5878 50WG, 50% a.i.; Appendix 3, p. 186), was conducted on bareground plots in Jackson County, Arkansas and Glenn County, California (pp. 22-23; Appendix B, Figure 1, p. 201). The sites selected were representative of climatic conditions in major rice-growing regions of Arkansas and California (Appendix 3, p. 171).

GUIDELINE FOLLOWED: This study was conducted in accordance with USEPA Subdivision N guideline §164-2 (p. 1). One significant deviation from the objectives of Subdivision N guidelines was noted:

> The stability of the parent and transformation products was not demonstrated for the maximum storage interval of test samples (with the exception of parent and IR7825 from the Arkansas water samples). Subdivision N guidelines specify that the stability of samples under typical storage conditions be determined.

COMPLIANCE: The field phase of this study was conducted in compliance with USEPA Good Laboratory Practices with seven exceptions; the analytical phase of this study was conducted in compliance with USEPA Good Laboratory Practices with no exceptions (p. 3). Signed and dated Good Laboratory Practice, Quality Assurance, Certification of Authenticity and Data Confidentiality statements were provided with the study (pp. 2-6).

Arkansas test site. The soil at the test site was an Amagon and Forestdale silt loam (0-6 inch depth: 32% sand, 56% silt, 12% clay, pH 6.7, 0.8% organic matter, CEC 8.8 meq/100 g, bulk density 1.22 g/cm^3; p. 23; Table 2, p. 57). The site has a plow layer at approximately 15 cm and a hardpan layer at approximately 61 cm (p. 23). The depth to the water table was estimated to be 5.5 m. The treated plot (slope 0.2%) was divided into triplicate 20.3 m x 6.1 m plots laid out end to end; each of the three replicate plots was further divided into 75 subplots for sampling (p. 24; Appendix B, Figures 3-4, pp. 203-204). A control plot (20.3 m x 6.1 m) was separated from the treated plots by a buffer of 36.5 m. The plot was planted to soybeans in the three years prior to study initiation (p. 23). A three-year plot history indicated that the pesticides Dual Magnum 7.62EC (metolachlor), Reflex 2LC (fomesafen), and Basagran 4SL (bentazon) were applied to the test plot (Appendix 3, Table 2, p. 187). The test area was cultivated three times in May and June 2003 and rototilled (p. 24). Glyphosate was used during the study period to control weeds. The plots were flooded approximately 3 weeks prior to the test application to prepare the plots for the study; the water was drained at the time of application. Meteorological measurements were made approximately 2 miles west of the test site (p. 25). Total precipitation during the study period was 68.12 inches, compared to a historical average of 70.70 inches; an additional 39-48 inches of flood/irrigation were applied following application through the last sampling

date (p. 41). No extreme or abnormal environmental conditions were observed during the conduct of the field phase.

California test site. The soil at the test site was a Zamora silty clay loam (0-6 inch depth: 18% sand, 49% silt, 33% clay, pH 7.7, 2.2% organic matter, CEC 22.5 meq/100 g, bulk density 1.22 g/cm^3; p. 23; Table 2, p. 57). The site has a plow layer at approximately 20 cm and does not have a discernable hardpan layer (p. 23). The depth to the water table was estimated to be 7.6 m. The treated plot (slope <1.0%) was divided into triplicate 11.4 m x 7.6 m plots laid out side by side; each of the three replicate plots was further divided into 75 subplots for sampling (p. 24; Appendix B, Figures 6-7, pp. 206-207). A control plot (dimensions not reported) was separated from the treated plots by a buffer of 33.4 m. The plot was fallow in 2000 and 2001 and planted with tomatoes in 2002 (p. 23). A three-year plot history indicated that the pesticides Treflan (trifluralin), Sencor (metribuzin), and Shadeout (rimsulfuron; a sulfonylurea) were applied to the test plot (Appendix B, Table 2, p. 187). The test area was prepared by discing and landplaning (p. 24). Glyphosate was used during the study period to control weeds. The plots were flooded approximately 2 weeks prior to the test application to prepare the plots for study; the water was drained at the time of application. Meteorological measurements were made approximately 13 miles northeast or 17 miles northwest of the test site (p. 25). Total precipitation during the study period was 29.86 inches, compared to a historical average of 35.20 inches; an additional 31 inches of flood/irrigation were applied following application through January 18, 2005 (p. 41). No extreme or abnormal environmental conditions were observed during the conduct of the field phase.

Both test sites: Orthosulfamuron was broadcast sprayed on August 11, 2003 (Arkansas site) and July 15, 2003 (California site), at a target application rate of 375 g a.i./ha (750 g product/ha) onto the test plots (pp. 25-26; Table 1, p. 56). The target application rate was 500% of the normal use rate (p. 17). The application was made at both sites using a CO_2 powered boom sprayer equipped with 12-15 TeeJet flat fan nozzles spaced 20 inches apart and 15-18 inches above the target. A commercial adjuvant was added to the spray mixture at 0.2% v/v for the application at each test site. The concentration of orthosulfamuron in the spray tank was verified by taking four 25-mL spray solution samples from the spray tank (two before the application and two following the application; p. 27). Samples were diluted with acetonitrile followed by further dilution with acetonitrile:aqueous buffer prior to HPLC/UV analysis at 254 nm (pp. 34-35). The mean recovery of orthosulfamuron from the spray tank samples was 88.8 ± 3.9% for the Arkansas site and 90.5 ± 1.7% for the California site (p. 41; Table 3, p. 58). Meteorological conditions during application at the Arkansas site were: wind speed 0-1 mph (N), air temperature 21.1EC, relative humidity 90% (Appendix 3, Table 6, p. 193). Meteorological conditions during application at the California site were: wind speed 0 mph, air temperature 26.7EC, relative humidity 35%.

The plots were flooded 1 day (Arkansas site) or 2 days (California site) following the test application to maintain a flood of approximately 3-4 inches (pp. 24-25). For the Arkansas plots, the levees were cut in October 2003 to allow for drainage; the plots were again flooded on June 10, 2004 to maintain a water level of 3 inches throughout the growing season. The California

plots were drained on September 20, 2003, and were periodically flushed with irrigation water through the remainder of the study to simulate commercial practices.

The application rate was verified at both test sites using filter paper squares (20 cm x 20 cm) backed by aluminum foil (p. 27). Five paper squares were placed in each of the three replicate plots prior to the test application and were collected immediately following the application by folding into quarters, wrapping in clean aluminum foil, and placing in a freezer bag and into the freezer. Filter paper target pads were stored frozen for 43-45 days (Arkansas samples) or 9 days (California samples) prior to analysis (Appendix D, Table 1, p. 269). The application monitoring pads were exhaustively extracted with methanol:$NaHCO_3$ (7:3, v:v), and the combined extract was diluted and analyzed by HPLC/UV at 254 nm (p. 35).

Soil samples were collected from the Arkansas test plot at –14, 0, 1, 7, 15, 22, 31, 60, 123, 186, 273, 364, 462, and 550 days posttreatment (Appendix 3, Table 9, p. 196). Soil samples were collected from the California test plot at –4, 0, 1, 3, 7, 15, 31, 62, 122, 194, 284, 372, 464, and 553 days posttreatment. For non-flooded sampling intervals, five soil cores were collected from the control plot and each of the three replicate plots at each test site. Soil cores were collected in two segments, 0-15 cm and approximately 15-60 cm, using a Concord tractor-mounted hydraulic probe (Arkansas site) or a manual system (California site; pp. 27-28). The core diameter was not specified for either test site. The actual depth reached was dictated by the underlying hardpan layer and varied from sample to sample. Following flooding, a 1-m length probe lined with an acetate liner was used to collect the soil samples (probe diameter not specified). Prior to freezing or after freezing, the soil cores were sectioned into 15-cm increments and the five samples from each replicate plot were composited to form a single sample. Arkansas soil samples were stored frozen for up to 155 days prior to analysis for orthosulfamuron, IR7825, and IR8181 and for up to 309 days prior to analysis for IR7863 (Appendix 4, Table 1, pp. 270-271). California soil samples were stored frozen for up to 258 days prior to analysis for orthosulfamuron, IR7825, and IR8181, and for up to 351 days prior to analysis for IR7863 (Appendix 4, Table 1, pp. 274-275).

Sediment and water samples were scheduled for collection at 3, 7, and 15 days and 1 and 2 months posttreatment (pp. 28-29). Sediment samples were collected to a depth of 5 cm using an acetate liner that was inserted into the water and sediment/soil by hand (Appendix A, p. 82). Samples were collected by sealing the top of the tube to provide suction. Following collection, the water was drained or poured off and the sediment samples were frozen. At each sampling interval, two water samples were collected from the control plot and from each of the three replicate plots. Samples were collected in 500-mL glass bottles and stored frozen. Arkansas sediment samples were stored frozen for up to 202 days prior to analysis for orthosulfamuron, IR7825, and IR8181, and for up to 162 days prior to analysis for IR7863 (Appendix 4, Table 1, pp. 272-273). Arkansas water samples were stored frozen for up to 70 days prior to analysis for orthosulfamuron, IR7825, and IR8181, and for up to 98 days prior to analysis for IR7863. California sediment samples were stored frozen for up to 204 days prior to analysis for orthosulfamuron, IR7825, and IR8181, and for up to 331 days prior to analysis for IR7863 (Appendix 4, Table 1, pp. 276-277). California water samples were stored frozen for up to 125

days prior to analysis for orthosulfamuron, IR7825, and IR8181, and for up to 145 days prior to analysis for IR7863.

Water, soil and sediment samples were analyzed for orthosulfamuron and the transformation products IR7825 (4,6-dimethoxy-2-pyrimidinyl urea), IR8181 (1-[2(dimethylcarbamoyl)-phenylsulfamoyl]-3-(4-hydroxy-6-methoxypyrimidin-2-yl)urea) and IR7863 (sodium (2-dimethylcarbamoylphenyl)sulfamate; pp. 33-34; Appendix D, pp. 245-247).

Table 7. CAS chemical names and numbers for orthosulfamuron and its degradates.

Applicant's Code Name	CAS Chemical Name	CAS No.	Molecular Weight (g/mol)
IR5878 (Orthosulfamuron)	2-[[[[[(4,6-Dimethoxy-2-pyrimidinyl)amino]carbonyl]-amino]-sulfonyl]amino]-N,N-dimethylbenzamide	213464-77-8	424.4
IR7825	4,6-Dimethoxy-2-pyrimidinyl urea	151331-81-6	198
IR8181	1-[2(Dimethylcarbamoyl)phenylsulfamoyl]-3-(4-hydroxy-6-methoxypyrimidin-2-yl)urea	N/A	410.4
IR7863	Sodium (2-dimethylcarbamoylphenyl)sulfamate	N/A	266

Table data were obtained from pp. 33-34.

Analytical method for orthosulfamuron, IR7825, and IR8181 in soil/sediment. Soil/sediment samples were extracted using an accelerated solvent extractor with a solvent mixture of acetonitrile and 0.02M triethylamine, and the extract was partitioned with hexane in the presence of large quantities of sodium chloride (12-15 g; p. 35). The acetonitrile layer was concentrated, and the residue was reconstituted in acetonitrile:aqueous sodium carbonate and analyzed by LC/MS/MS. HPLC parameters were as follows (Appendix 4, p. 399):

Column	Phenomenex SPHEREX 5, 15 cm x 3.2 mm X 5µ
Flow rate	0.6 mL/minute
Injection volume	5 or 10 µL
Solvent system	Solvent A = water (0.05% formic acid) Solvent B = acetonitrile (0.05% formic acid)
Solvent program	A:B, 80:20 to 40:60 to 0:100 to 80:20
Retention times	IR8181: 3 minutes for m/z242 + m/z268 IR7825: 3 minutes for m/z156 + m/z182 Orthosulfamuron: 6 minutes for m/z199 + m/z227

The LOQ was 0.005 mg/kg for each analyte in soil and the LOD was 0.0006 mg/kg (p. 42).

Analytical method for IR7863 in soil/sediment. Soil/sediment samples were extracted with a pH 8 borate buffer using an accelerated solvent extractor. The resultant borate buffer extract was treated with acid under reflux in the presence of sodium thiosulfate to yield 2-amino-N,N-

dimethylbenzamide (DBAmine; p. 35). The hydrolysate was basified and DBAmine was extracted into dichloromethane. An aliquot of trifluoroacetic acid was added to protonate DBAmine. The extract was then concentrated by roto-evaporation and the residue was reconstituted in toluene, partitioned with aqueous sodium hydroxide, and analyzed for DBAmine by GC with nitrogen/phosphorous detection (Appendix 4, p. 410). A separate method was developed for the California soil samples, and was also used for the 1- and 2-month Arkansas soil samples due to the clay-like nature of the soil, which rendered the samples difficult for the accelerated solvent extractor to handle. As a result, these soils were dried, ground, and mixed with sand and Hydromatrix prior to ASE extraction, and the number of ASE extraction cycles was increased. The LOQ was 0.02 mg/kg for IR7863 in soil and the LOD was 0.008 mg/kg (p. 43).

Analytical method for orthosulfamuron, IR7825 and IR8181 in water. Water samples were thawed and filtered through a Whatman No. 4 filter prior to analysis (p. 34). Samples were then analyzed by direct injection LC/MS/MS relative to external mixed standards (p. 36). HPLC parameters were reported in Appendix 4 of the study report (pp. 422-424). The LOQ was 0.005 mg/L for each analyte in water (p. 50).

Analytical method for IR7863 in water. As with the soil samples, the water samples were hydrolyzed at reflux in the presence of acid (p. 36). The hydrolysate was then basified and DBAmine was extracted into dichloromethane. An aliquot of trifluoroacetic acid was added and the extract was then concentrated. The residue was reconstituted in toluene, partitioned with aqueous sodium hydroxide, and analyzed for DBAmine by GC with nitrogen/phosphorous detection (Appendix 4, p. 433). The LOQ was 0.02 mg/L for IR7863 in water (p. 50).

Method validation studies were conducted for the analysis of orthosulfamuron, IR7825, IR8181, and IR7863 in soil and water from Arkansas and California (pp. 35-36). Method validations were conducted on soil samples at 0.005 and 0.05 mg/kg for orthosulfamuron, IR 7825, and IR8181 (plus 0.50 mg/kg for IR7825 in Arkansas soil), and at 0.02 and 0.05 mg/kg for IR7863 (p. 35). Method validations were conducted on water samples at 0.005 and 0.05 mg/L for orthosulfamuron, IR7825, IR8181, and IR7863. Method validation for orthosulfamuron and IR7825 was determined in a different manner than used for the field samples. Samples were repeatedly extracted with dichloromethane and the combined dichloromethane extract was concentrated, reconstituted in acetonitrile:aqueous $NaHCO_3$, and analyzed by LC/MS/MS (compared to direct analysis by LC/MS/MS for the field samples).

Field spikes were prepared for orthosulfamuron, IR7825, and IR7863 using untreated soil, sediment, and water samples at the 2-month posttreatment sampling interval (p. 37; Appendix 3, p. 182). Soil and sediment samples (5-10 g) were fortified with orthosulfamuron at 0.05 mg/kg, with IR7825 at 0.5 mg/kg, and with IR7863 at 1.0 mg/kg. Water samples (100 mL) were individually spiked with 0.05 mg/L orthosulfamuron, IR7825, and IR7863. The field spikes were kept under the same conditions as the field cores until analysis. Field spikes were analyzed following 7 to 114 days of storage (Tables 8-10, pp. 64-66). Field spikes were not prepared for IR8181 in soil, sediment or water.

RESULTS AND DISCUSSION

Orthosulfamuron (1-(4,6-dimethoxypyrimidin-2-yl)-3-[2-(dimethylcarbamoyl)phenyl-sulfamoyl]urea; IR5878 50WG, 50% a.i.) was applied once at a target application rate of 375 g a.i./ha onto a bareground plot of silt loam soil in Arkansas and a bareground plot of silty clay loam soil in California. The plots were flooded with water 1-2 days posttreatment for 2 months.

Arkansas test site. Orthosulfamuron dissipated in the soil, sediment, and water with calculated half-life values of 4.1 days (r^2 = 0.9119), 6.2 days (r^2 = 0.8864), and 6.4 days (r^2 = 0.7323), respectively. Half-lives were calculated using linear regression analysis performed on a plot of ln-transformed orthosulfamuron concentrations vs. time.

Orthosulfamuron was detected in the 0- to 15-cm soil depth at a mean concentration of 0.069 mg/kg at day 0, which is 33.7% of the theoretical amount (calculated based on a theoretical value of 0.205 mg/kg determined for the 0-15 cm soil depth based on the application rate at 0.375 kg a.i./ha and the site specific bulk density of 1.22 g/cm^3). Orthosulfamuron decreased to 0.046 mg/kg by 1 day and 0.015 mg/kg by 7 days, and was last detected at 0.006 mg/kg (single replicate) at 15 days posttreatment (Table 5, p. 61; Appendix 4, Table IX, pp. 288-289). Orthosulfamuron was only detected once in the 15- to 30-cm soil depth, at 0.007 mg/kg (single replicate) at day 0, and was not detected below that depth (Appendix 4, Table IX, pp. 290-293).

The major transformation product **IR7825** was initially detected in the 0-15 cm soil at 0.006 mg/kg (single replicate) at 1 day, ranged from a mean of 0.023-0.026 mg/kg from 7-60 days, was a maximum of 0.049 mg/kg at 186 days, and decreased to 0.017 mg/kg by 550 days, the last sampling interval (Appendix 4, Table X, pp. 300-302). The half-life of IR7825 in soil was 330 days (r^2 = 0.2476; based on 186-550 day data).

The transformation product **IR7863** was only detected in the soil above the LOQ at 7 days (0.041-0.052 mg/kg; 2 of 3 replicates) and 22 days (0.025 mg/kg; 2 of 3 replicates; Appendix 4, Table XII, pp. 330-331). IR7825 and IR7863 were not detected below the 0-15 cm soil depth (Appendix 4, Tables X and XII, pp. 303-308 and 332-335, respectively). The transformation product **IR8181** was not detected in soil above the LOD (Appendix 4, Table XI, pp. 318-323). Mean concentrations of orthosulfamuron residues in soil are presented in Table 8.

Table 8. Mean concentration of orthosulfamuron residues in Arkansas soil (mg/kg soil).

Compound	Soil depth (cm)	Sampling times (days posttreatment)												
		0	1	7	15	22	31	60	123	186	273	364	462	550
Orthosulfamuron	0-15	**0.069**	**0.046**	**0.015**	**0.006a**	<0.005	<0.005	<0.005	<0.005	--	--	--	--	--
	15-30	**0.006**[a]	<0.005	ND	ND	ND	ND	ND	ND	--	--	--	--	--
	30-45	<0.005	ND	ND	ND	ND	ND	ND	ND	--	--	--	--	--
IR7825	0-15	**0.007**[a]	**0.006**[a]	**0.026**	**0.026**	**0.025**	**0.023**	**0.026**	**0.023**[a]	**0.049**	**0.033**	**0.018**	**0.037**	**0.017**
	15-30	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
	30-45	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
IR8181	0-15	ND	ND	ND	ND	ND	ND	ND	ND	--	--	--	--	--
	15-30	ND	ND	ND	ND	ND	ND	ND	ND	--	--	--	--	--
	30-45	ND	ND	ND	ND	ND	ND	ND	ND	--	--	--	--	--
IR7863	0-15	ND	ND	**0.047**[b]	ND	**0.025**[b]	ND	ND	--	--	--	--	--	--
	15-30	ND	ND	ND	ND	<0.02	ND	ND	--	--	--	--	--	--
	30-45	ND	ND	ND	ND	<0.02	ND	ND	--	--	--	--	--	--

Residue data obtained from Table 5, p. 61 and Appendix 4, Tables IX, X, XI and XII, pp. 288-293, 300-308, 318-323 and 330-335, respectively.
Orthosulfamuron, IR7825, and IR8181: LOD = 0.0006 mg/kg and LOQ = 0.005 mg/kg. IR7863: LOD = 0.008 mg/kg and LOQ = 0.02 mg/kg.
a = single replicate value (other replicates <LOQ).
b = mean of duplicate detects (with the third replicate <LOQ).

Orthosulfamuron was detected in the sediment (0-5 cm depth) at a mean concentration of 0.182 mg/kg at 3 days, decreased to 0.055 mg/kg by 7 days and 0.019 mg/kg by 15 days, and was last detected at 0.008 mg/kg at 1 month posttreatment (Table 6, p. 62; Appendix 4, Table XIV, p. 344).

The major transformation product **IR7825** was detected in the sediment at 0.042 mg/kg at 3 days, increased to a maximum of 0.141 mg/kg by 15 days, and then decreased to 0.049 mg/kg by 2 months, the last sampling interval (Appendix 4, Table XV, p. 346). The half-life of IR7825 in sediment was 37.1 days (r^2 = 0.4408; based on 15-day to 2-month data).

The major transformation product **IR7863** was detected in the sediment at 0.061-0.065 mg/kg from 3-7 days, was not detected at 15 days, was 0.053 mg/kg at 22 days, and was not detected at 2 months posttreatment (Appendix 4, Table XVII, p. 350). The transformation product **IR8181** was not detected in sediment above the LOQ (Appendix 4, Table XVI, p. 348). Mean concentrations of orthosulfamuron residues in sediment are presented in Table 9.

Table 9. Mean concentration of orthosulfamuron residues in Arkansas sediment (mg/kg soil).

Sampling interval	Concentration in sediment (mg/kg)			
	Orthosulfamuron	IR7825	IR8181	IR7863
3 days	**0.182**	**0.042**	ND	**0.061**
7 days	**0.055**	**0.126**	<0.005	**0.065**
15 days	**0.019**	**0.141**	<0.005	ND
22 days	**0.011**	**0.085**	<0.005	**0.053**
1 month	**0.008**	**0.062**	<0.005	**0.031**[a]
2 months	<0.005	**0.049**	ND	ND

Residue data obtained from Table 6, p. 62 and Appendix 4, Tables XIV, XV, XVI and XVII, pp. 344, 346, 348 and 350, respectively. Orthosulfamuron, IR7825 and IR8181: LOD = 0.0006 mg/kg and LOQ = 0.005 mg/kg. IR7863: LOD = 0.008 mg/kg and LOQ = 0.02 mg/kg.
a = single replicate value (other replicates <LOQ).

Orthosulfamuron was detected in the water at a mean concentration of 0.024 mg/L at 3 days, decreased to 0.012 mg/L by 7 days, and was last detected at 0.006 mg/L (mean from replicates with a detection) at 15 days posttreatment (Table 7, p. 63; Appendix 4, Table XIX, pp. 354-355).

The transformation product **IR7863** was detected in the water at 0.006 mg/L at 3 days, increased to a maximum of 0.011 mg/L (mean of the 5 detects) by 15 days, and was not detected above the LOQ following 1 month posttreatment. The transformation products **IR7825** and **IR8181** were not detected in the water above the LOQ. Mean concentrations of orthosulfamuron residues in water are presented in Table 10.

Table 10. Mean concentration of orthosulfamuron residues in Arkansas water (mg/L).

Sampling interval	Concentration in water (mg/L)			
	Orthosulfamuron	IR7825	IR8181	IR7863
3 days	**0.024**	<0.005	ND	**0.006**
7 days	**0.012**	<0.005	ND	**0.009**[a]
15 days	**0.006**[a]	<0.005	ND	**0.011**[a]
22 days	<0.005	<0.005	ND	**0.009**
1 month	<0.005	<0.005	<0.005	**0.006**[a]
2 months	<0.005	<0.005	ND	<0.005

Residue data obtained from Table 7, p. 63 and Appendix 4, Table XIX, pp. 354-355. Orthosulfamuron, IR7825, and IR8181: LOQ = 0.005 mg/L. IR7863: LOQ = 0.02 mg/L.
a = mean from replicates with a detection.

California test site. Orthosulfamuron dissipated in the soil and sediment with half-life values of 2.3 days (r^2 = 0.8678) and 4.3 days (r^2 = 0.438), respectively. Half-lives were calculated using linear regression analysis performed on a plot of ln-transformed orthosulfamuron concentrations vs. time. A half-life for orthosulfamuron in water could not be calculated because the chemical was not detected in the water.

Orthosulfamuron was detected in the 0- to 15-cm soil depth at a mean concentration of 0.094 mg/kg at day 0, which is 45.9% of the theoretical amount (calculated based on a theoretical value of 0.205 mg/kg determined for the 0-15 cm soil depth based on the application rate at 0.375 kg a.i./ha and the site specific bulk density of 1.22 g/cm^3). Orthosulfamuron decreased to 0.031 mg/kg (single replicate) by 3 days and was last detected at 0.013 mg/kg at 7 days posttreatment (Table 5, p. 61; Appendix 4, Table IX, pp. 294-295).

The major transformation product **IR7825** was initially detected in the 0-15 cm soil at 0.008 mg/kg at 1 day, increased to a maximum of 0.040 mg/kg by 194 days, then decreased to 0.021-0.036 mg/kg by 284-372 days and was 0.024 mg/kg at 553 days, the last sampling interval (Appendix 4, Table X, pp. 309-311). The half-life of IR7825 in soil was 177.7 days (r^2 = 0.0039; based on 194-553 day data). Orthosulfamuron and IR7825 were not detected below the 0-15 cm soil depth (Appendix 4, Tables IX and X, pp. 296-299 and 312-317, respectively).

The transformation product **IR7863** was not detected in the soil above the LOQ at any depth (0.02 mg/kg; Appendix 4, Table XII, pp. 336-341). The transformation product **IR8181** was not detected in soil above the LOD (Appendix 4, Table XI, pp. 324-329). Mean concentrations of orthosulfamuron residues in soil are presented in Table 11.

Table 11. Mean concentration of orthosulfamuron residues in California soil (mg/kg soil).

Compound	Soil depth (cm)	Sampling times (days posttreatment)												
		0	1	3	7	15	31	62	122	194	284	372	464	553
Orthosulfamuron	0-15	**0.094**	**0.095**	**0.031**[a]	**0.013**	<0.005	<0.005	ND	ND	<0.005	--	--	--	--
	15-30	<0.005	<0.005	<0.005	ND	ND	ND	ND	ND	ND	--	--	--	--
	30-45	<0.005	ND	<0.005	<0.005	<0.005	ND	ND	ND	ND	--	--	--	--
IR7825	0-15	<0.005	**0.008**	**0.012**[a]	**0.022**	**0.019**	**0.017**[b]	**0.010**	**0.031**	**0.040**	**0.021**	**0.036**	**0.015**[a]	**0.024**
	15-30	ND	ND	ND	ND	<0.005	ND	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	ND
	30-45	ND	ND	ND	<0.005	ND	<0.005	<0.005	<0.005	<0.005	<0.005	ND	ND	ND
IR8181	0-15	ND	ND	ND	ND	ND	ND	ND	ND	ND	--	--	--	--
	15-30	ND	ND	ND	ND	ND	ND	ND	ND	ND	--	--	--	--
	30-45	ND	ND	ND	ND	ND	ND	ND	ND	ND	--	--	--	--
IR7863	0-15	<0.02	<0.02	<0.02	<0.02	ND	ND	ND	ND	ND	--	--	--	--
	15-30	ND	ND	ND	ND	ND	ND	ND	ND	ND	--	--	--	--
	30-45	ND	ND	ND	ND	ND	ND	ND	ND	ND	--	--	--	--

Residue data obtained from Table 5, p. 61 and Appendix 4, Table IX, X, XI and XII, pp. 294-299, 309-317, 324-329 and 336-341, respectively.
Orthosulfamuron, IR7825, and IR8181: LOD = 0.0006 mg/kg and LOQ = 0.005 mg/kg. IR7863: LOD = 0.008 mg/kg and LOQ = 0.02 mg/kg.
a = single replicate value (other replicates <LOQ).
b = mean of duplicate detects (with the third replicate <LOQ).

Orthosulfamuron was detected in the sediment (0-5 cm depth) at a mean concentration of 0.062 mg/kg at 3 days, decreased to 0.050 mg/kg by 7 days and was last detected at 0.007 mg/kg (single replicate) at 15 days posttreatment (Table 6, p. 62; Appendix 4, Table XIV, p. 345).

The major transformation product **IR7825** was detected in the sediment at 0.028 mg/kg at 3 days, increased to a maximum of 0.100 mg/kg by 7 days, and then decreased to 0.033 mg/kg by 15 days and 0.017 mg/kg by 2 months, the last sampling interval (Appendix 4, Table XV, p. 347). The half-life of IR7825 in sediment was 29.6 days (r^2 = 0.3627; based on 7 day to 2 month data).

The transformation product **IR7863** was not detected in the sediment above the LOQ, and the transformation product **IR8181** was not detected in the sediment above the LOD (Appendix 4, Tables XVI and XVII, pp. 349 and 351, respectively). Mean concentrations of orthosulfamuron residues in sediment are presented in Table 12.

Table 12. Mean concentration of orthosulfamuron residues in California sediment (mg/kg soil).

Sampling interval	Concentration in sediment (mg/kg)			
	Orthosulfamuron	IR7825	IR8181	IR7863
3 days	**0.062**	**0.028**	ND	<0.02
7 days	**0.050**	**0.100**	ND	<0.02
15 days	**0.007**[a]	**0.033**	ND	ND
1 month	<0.005	**0.023**[b]	ND	ND
2 months	ND	**0.017**	ND	ND

Residue data obtained from Table 6, p. 62 and Appendix 4, Tables XIV, XV, XVI and XVII, pp. 345, 347, 349 and 351, respectively. Orthosulfamuron, IR7825, and IR8181: LOD = 0.0006 mg/kg and LOQ = 0.005 mg/kg. IR7863: LOD = 0.008 mg/kg and LOQ = 0.02 mg/kg.
a = single replicate value (other replicates <LOQ).
b = mean of duplicate detects (with the third replicate <LOQ).

Orthosulfamuron, IR7825, IR8181, and **IR7863** were not detected in the water above the LOQ at any sampling intervals (Table 7, p. 63; Appendix 4, Table XIX, pp. 356-357).

Table 13. Mean concentration of orthosulfamuron residues in California water (mg/L).

Sampling interval	Concentration in water (mg/L)			
	Orthosulfamuron	IR7825	IR8181	IR7863
3 days	<0.005	<0.005	ND	ND
7 days	<0.005	<0.005	<0.005	ND
15 days	<0.005	<0.005	ND	<0.005
1 month	<0.005	<0.005	<0.005	ND
2 months	ND	ND	ND	ND

Residue data obtained from Table 7, p. 63 and Appendix 4, Table XIX, pp. 356-357. Orthosulfamuron, IR7825, and IR8181: LOQ = 0.005 mg/L. IR7863: LOQ = 0.02 mg/L.

The mean recovery of orthosulfamuron from the application monitoring pads was 91.3 ± 19.8% for the Arkansas site and 86.6 ± 26.9% for the California site (pp. 41-42; Table 4, pp. 59-60). The mean recovery of orthosulfamuron from filter paper target pads fortified in triplicate at 1.0 and 1.5 mg/pad was 101 ± 8%.

Method validation recoveries of orthosulfamuron and its transformation products in soil and water were generally acceptable with the exception of IR8181 in soil, as reported in Table 14.

Table 14. Method validation recoveries (mean ± SD) of orthosulfamuron and its transformation products from soil and water.

Analyte	Fortification level – Soil			Fortification level - Water	
Orthosulfamuron	0.005 mg/kg	0.05 mg/kg	0.5 mg/kg	0.005 mg/L	0.05 mg/L
Arkansas	78%	76%	NA	118.7%	94.4%
California	74%	77%	NA		
Average	**76 ± 3%**			**106.5 ± 16.3%**	
IR7825	0.005 mg/kg	0.05 mg/kg	0.5 mg/kg	0.005 mg/L	0.05 mg/L
Arkansas	NA	92%	92%	101.0%	95.2%
California	89%	91%	NA		
Average	**91 ± 2.8%**			**98.1 ± 4.5%**	
IR8181	0.005 mg/kg	0.05 mg/kg	0.5 mg/kg	0.005 mg/L	0.05 mg/L
Arkansas	64%	62%	NA	92.0%	99.3%
California	63%	49%	NA		
Average	**59 ± 11%**			**95.7 ± 5.6%**	
IR7863	0.02 mg/mg	0.05 mg/kg		0.005 mg/L	0.05 mg/L
Arkansas	86%	59%	NA	70.0%	77.6%
California	99%	81%	NA		
Average	**81 ± 25%**			**73.8 ± 7.7%**	

Means were obtained from Appendix 4, Tables IV-V, pp. 280-281, p. 223. Recoveries reported for Arkansas and California are mean values across multiple replicates. Mean values for the recoveries from water for each fortification level were calculated from the reported replicate values; recoveries were not reported separately for the Arkansas and California samples.

Mean recoveries from soil and sediment field spikes were low (<70%) for IR7863 in Arkansas soil and sediment, and for orthosulfamuron, IR7825, and IR7863 in California soil and sediment, following correction for concurrent recoveries (Tables 8-9, pp. 64-65). Mean recoveries of all three analytes from Arkansas and California water samples were ≥69% (Table 10, p. 66). Field spikes were not prepared for IR8181 in soil, sediment or water. Mean recoveries from soil, sediment, and water fortification samples and total storage time are reported in Table 13.

Table 13. Total storage time and mean fortification recoveries of orthosulfamuron, IR7825, and IR7863 from soil, sediment, and water.

Analyte	Total storage time (days)	Percent recovery (%)		
		Lab fortification	Field fortification	Corrected field fortification
Arkansas soil				
Orthosulfamuron	89	82.2	67.3	**81.8**
IR7825	89	78.8	73.7	**93.5**
IR7863	74	69	23	**33**
California soil				
Orthosulfamuron	7	83	51	**61**
IR7825	12	100	69	**69**
IR7863	97	69	16	**23**
Arkansas sediment				
Orthosulfamuron	90	73	82	**112**
IR7825	80	90	79	**88**
IR7863	73	65	31	**47**
California sediment				
Orthosulfamuron	114	109	67	**67**
IR7825	104	90	48	**54**
IR7863	97	65	31	**48**
Arkansas water				
Orthosulfamuron	73	89	82	**92**
IR7825	73	107	100	**100**
IR7863	67	89	94	**106**
California water				
Orthosulfamuron	19	109	69	**69**
IR7825	21	101	92	**92**
IR7863	62	72	68	**95**

Data obtained from Tables 8-10, pp. 64-66.

Mean recoveries from **soil** samples that were fortified with orthosulfamuron, IR7825 or IR8181 at approximately 0.01-0.02 mg/kg and analyzed concurrently with the test samples were 84 ± 12%, 88 ± 6%, and 82 ± 11%, respectively, for Arkansas soil and 86 ± 13%, 88 ± 10%, and 94 ± 15%, respectively, for California soil (Appendix 4, Summary Table C, pp. 235-236). Mean recoveries of IR7863 from soil samples that were fortified at 0.02-0.05 mg/kg (AK) or 0.05 mg/kg (CA) were 80 ± 23% for the Arkansas samples and 65 ± 18% for the California samples (Appendix 4, Summary Table C, p. 237).

Mean recoveries from **sediment** samples that were fortified with orthosulfamuron, IR7825, and IR8181 at 0.02 mg/kg and analyzed concurrently with the test samples were 96 ± 6%, 87 ± 5%, and 96 ± 13%, respectively, for Arkansas sediment and 94 ± 8%, 88 ± 5%, and 80 ± 15%, respectively, for California sediment (Appendix 4, Summary Table D, p. 238). Mean recoveries of IR7863 from sediment samples that were fortified at 0.04 mg/kg (AK) or 0.05 mg/kg (CA) were 72 ± 16% for the Arkansas samples and 36 ± 10% for the California samples (Appendix 4, Summary Table D, p. 239).

Mean recoveries from **water** samples that were fortified with orthosulfamuron, IR7825, and IR8181 at 0.005-0.01 mg/L and analyzed concurrently with the test samples were 87 ± 18%, 93 ± 18%, and 103 ± 35%, respectively, for Arkansas water and 73 ± 13%, 101 ± 14%, and 86 ± 14%, respectively, for California water (Appendix 4, Summary Table E, p. 240). Mean recoveries of IR7863 from water samples that were fortified at 0.01 mg/L (AK) or 0.01-0.02 mg/L (CA) were 84 ± 12% for the Arkansas samples and 88 ± 7% for the California samples (Appendix 4, Summary Table E, p. 241).

STUDY DEFICIENCIES

1. Storage stability data (from field spikes) could not demonstrate stability of the parent and transformation products for the maximum storage interval of the test samples (with the exception of parent and IR7825 from the Arkansas water samples) due to the shorter storage intervals employed for the field spikes compared to the test samples prior to analysis. The field spikes were stored 7-114 days prior to analysis, whereas soil/sediment/water samples were stored for up to 351 days prior to analysis (Tables 8-10, pp. 64-66); Appendix 4, Table 1, pp. 270-277). Field spikes were not prepared for IR8181 in soil, sediment or water. In addition, recoveries of IR7863 from the Arkansas soil and sediment and recoveries of orthosulfamuron, IR7825, and IR7863 from the California soil and sediment were low, indicating that the compounds were not stable for the storage interval.

   Laboratory freezer storage stability studies conducted in separate experiments outside the scope of this study showed that orthosulfamuron, IR7825, and IR7863 were stable for 1 year in soil, sediment, and surface water samples stored below –20°C (MRID 46578977, 46578978, 46578979, 46578980, 46578981). A laboratory freezer storage stability study of IR8181 in soil, sediment, and water was ongoing (p. 37).

REVIEWER'S COMMENTS

1. The recovery of orthosulfamuron from the day-0 soil cores was low, accounting for only 33.7% (0.069 mg/kg; Arkansas site) and 45.9% (0.094 mg/kg; California site) of the theoretical amount (0.205 mg/kg), and was not in agreement with the recovery of parent from the application monitoring devices (91.3% and 86.6%, respectively, for the

Arkansas and California sites). The theoretical day-0 value was calculated for the 0-15 cm soil depth based on the application rate at 0.375 kg a.i./ha and the site-specific bulk density of 1.22 g/cm^3.

The low recovery from day-0 soil cores may be the result of degradation of orthosulfamuron during storage. This possibility is supported by the low mean recovery of orthosulfamuron from field spiked soil samples (81.8% for Arkansas soil following 89 days of storage and 61% for California soil following only 7 days of storage; Table 8, p. 64).

2. Application at the maximum label rate is generally required for kinetic studies because dissipation rates can vary unpredictably at different application rates. The test substance in this study was applied at an exaggerated target application rate of 375 g a.i./ha (750 g product/A) or 500% of the normal use rate in order to provide levels of the metabolites IR7863 and IR7825 greater than the LOQ, and therefore, allow establishment of their rates of decline (Appendix 2, p. 112), which is an acceptable modification in this case.

3. Rimsulfuron, a sulfonylurea, had been applied to the California test site within the past three years (Appendix B, Table 2, p. 187). Orthosulfamuron and rimsulfuron may share DOP urea (IR7863) as a common transformation product. However, DOP urea was not found above the level of detection in soil at the California test site prior to treatment and was never found above the level of quantitation in soil, sediment, or water throughout the study. Therefore, the possible presence of residues of rimsulfuron and its degradates onsite is not expected to have affected the outcome of the study.

LITERATURE CITED

MRID 46578977. Zini, G. and T. Crisippi. 2004. Storage Stability of IR7863 in Soil and Sediment Stored in the Dark Below -20°C. Unpublished study performed by ISAGRO RICERCA S.r.l., Novara, Italy; sponsored and submitted by ISAGRO S.p.A., Milano, Italy. Study No.: 2405. October 22, 2004. 138 pp.

MRID 46578978. Zini, G. and T. Crisippi. 2004. Storage Stability of IR7863 in Surface Water Stored in the Dark Below -20°C. Unpublished study performed by ISAGRO RICERCA S.r.l., Novara, Italy; sponsored and submitted by ISAGRO S.p.A., Milano, Italy. Study No.: 2403. October 12, 2004. 94 pp.

MRID 46578979. Zini, G. and T. Crisippi. 2004. Storage Stability of IR7825 in Surface Water Stored in the Dark Below -20°C. Unpublished study performed by ISAGRO RICERCA S.r.l., Novara, Italy; sponsored and submitted by ISAGRO S.p.A., Milano, Italy. Study No.: 2402. October 12, 2004. 105 pp.

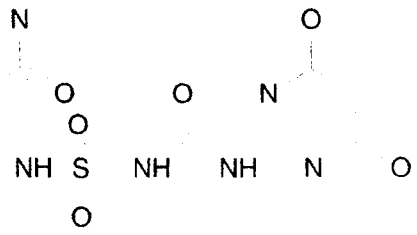
MRID 46578980. Zini, G. and T. Crisippi. 2004. Storage Stability of IR7825 in Soil and Sediment Stored in the Dark Below -20°C. Unpublished study performed by ISAGRO RICERCA S.r.l., Novara, Italy; sponsored and submitted by ISAGRO S.p.A., Milano, Italy. Study No.: 2404. October 8, 2004. 131 pp.

MRID 46578981. Zini, G. and T. Crisippi. 2004. Storage Stability of IR5878 in Soil, Sediment and Surface Water Stored in the Dark Below -20°C. Unpublished study performed by ISAGRO RICERCA S.r.l., Novara, Italy; sponsored and submitted by ISAGRO S.p.A., Milano, Italy. Study No.: 2377. May 24, 2004. 164 pp.

Attachment 1: Structures of Parent Compound and Transformation Products

Orthosulfamuron [IR5878; S3]

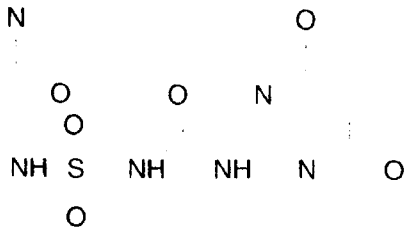
IUPAC Name:	1-(4,6-Dimethoxypyrimidin-2-yl)-3-[2-(dimethylcarbamoyl)phenylsulfamoyl]urea.
CAS Name:	2-[[[[[(4,6-Dimethoxy-2-pyrimidinyl)amino]carbonyl]amino]sulfonyl]amino]-N,N-dimethylbenzamide.
CAS Number:	213464-77-8.
SMILES String:	CN(C(=O)c1ccccc1NS(=O)(=O)NC(=O)Nc1nc(cc(n1)OC)OC)C (ISIS v2.3/Universal SMILES). No EPI Suite, v3.12 SMILES String found as of 11/21/05.



Identified Compounds

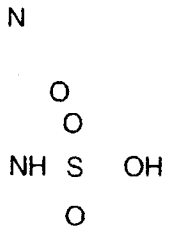
Orthosulfamuron [IR5878; S3]

IUPAC Name:	1-(4,6-Dimethoxypyrimidin-2-yl)-3-[2-(dimethylcarbamoyl)phenylsulfamoyl]urea.
CAS Name:	2-[[[[[(4,6-Dimethoxy-2-pyrimidinyl)amino]carbonyl]amino]sulfonyl]amino]-N,N-dimethylbenzamide.
CAS Number:	213464-77-8.
SMILES String:	CN(C(=O)c1ccccc1NS(=O)(=O)NC(=O)Nc1nc(cc(n1)OC)OC)C (ISIS v2.3/Universal SMILES). No EPI Suite, v3.12 SMILES String found as of 11/21/05.



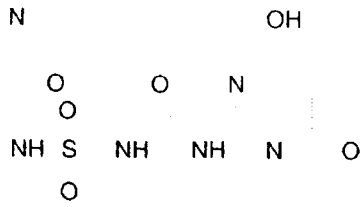
S1 [IR7863; DBS acid]

IUPAC Name:	(2-Dimethylcarbamoylphenyl)sulfamic acid.
CAS Name:	Sodium (2-dimethylcarbamoylphenyl)sulfamate.
CAS Number:	Not reported.



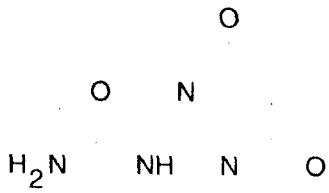
S9 [IR8181; O-desmethyl IR5878]

IUPAC Name: 1-(4-Hydroxy-6-methoxypyrimidin-2-yl)-3-[2-(2-dimethylcarbamoyl)phenylsulfamoyl]urea.
CAS Name: 1-[2(Dimethylcarbamoyl)phenylsulfamyl]-3-(4-hydroxy-6-methoxypyrimidin-2-yl)urea.
CAS Number: Not reported.



S12 [IR7825; DOP urea]

IUPAC Name: N-(4,6-Dimethoxypyrimidin-2-yl)-urea.
CAS Name: 4,6-Dimethoxy-2-pyrimidinyl urea.
CAS Number: Not reported.



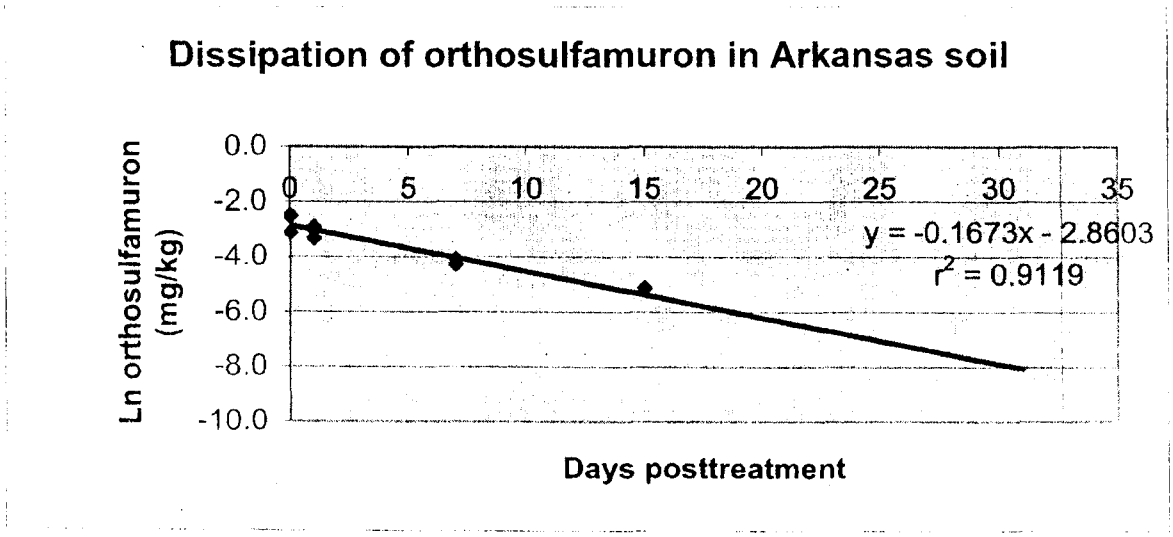
Attachment 2: Excel and SigmaPlot Spreadsheets

Chemical name	Orthosulfamuron	**Arkansas soil**
PC code	108209	
MRID	46578985	
Guideline No.	164-1 and 164-2	

Half-life (days) = 4.1 ***Calculated using all available data**

Days posttreatment	Orthosulfamuron (mg/kg)	Ln (orthosulfamuron)
0	0.083	-2.489
0	0.044	-3.124
0	0.080	-2.526
1	0.048	-3.037
1	0.036	-3.324
1	0.055	-2.900
7	0.014	-4.269
7	0.014	-4.269
7	0.017	-4.075
15	<0.005	
15	0.006	-5.116
15	<0.005	
22	<0.005	
22	<0.005	
22	<0.005	
31	<0.005	
31	ND	
31	<0.005	
60	<0.005	
60	ND	
60	<0.005	
123	<0.005	
123	ND	
123	ND	

* Data obtained from Appendix 4, Table IX, pp. 288-289 in the study report.

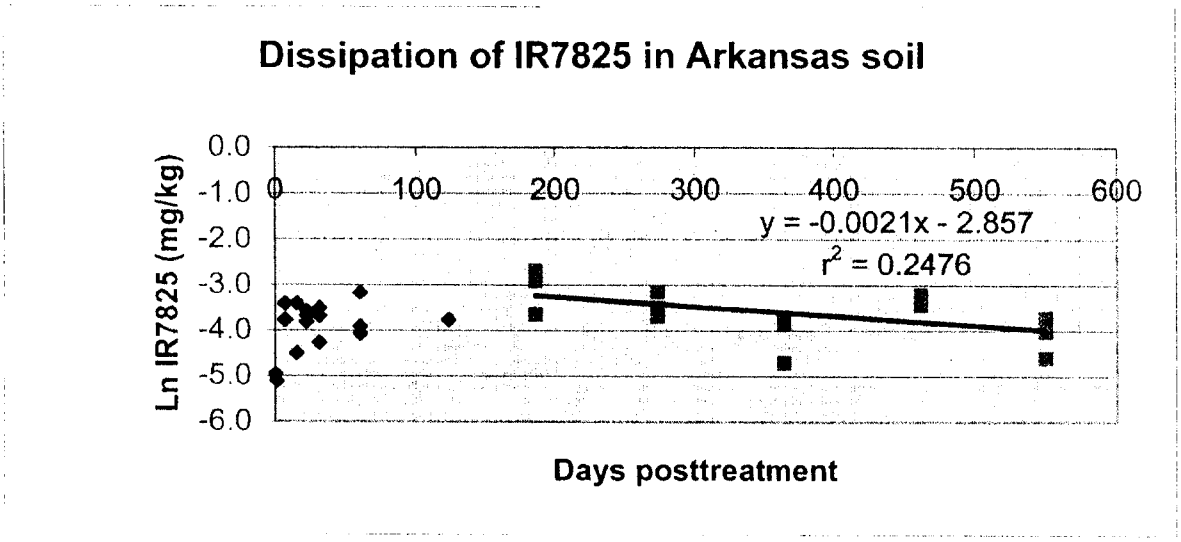


IR7825

Half-life (days) = 330.1 ***Calculated using 186-550 day data**

Days posttreatment	IR7825 (mg/kg)	Ln (IR7825)
0	0.007	-4.962
0	<0.005	
0	<0.005	
1	0.006	-5.116
1	<0.005	
1	<0.005	
7	0.023	-3.772
7	0.023	-3.772
7	0.033	-3.411
15	0.033	-3.411
15	0.033	-3.411
15	0.011	-4.510
22	0.022	-3.817
22	0.025	-3.689
22	0.028	-3.576
31	0.030	-3.507
31	0.014	-4.269
31	0.025	-3.689
60	0.020	-3.912
60	0.017	-4.075
60	0.042	-3.170
123	0.023	-3.772
123	<0.005	
123	<0.005	
186	0.054	-2.919
186	0.068	-2.688
186	0.026	-3.650
273	0.025	-3.689
273	0.030	-3.507
273	0.043	-3.147
364	0.024	-3.730
364	0.009	-4.711
364	0.021	-3.863
462	0.040	-3.219
462	0.038	-3.270
462	0.032	-3.442
550	0.010	-4.605
550	0.018	-4.017
550	0.024	-3.730

* Data obtained from Appendix 4, Table X, pp. 300-302 in the study report.

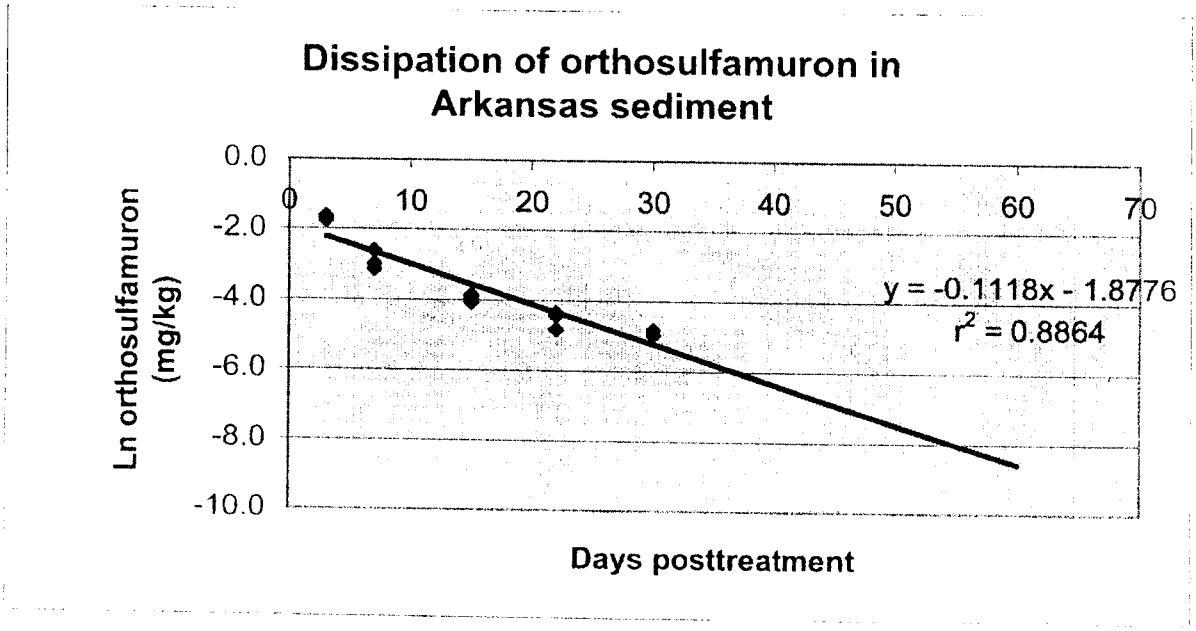

Dissipation of IR7825 in Arkansas soil
Ln IR7825 (mg/kg)
0.0
-1.0
-2.0
-3.0
-4.0
-5.0
-6.0
0
100
200
300
400
500
600
y = -0.0021x - 2.857
r2 = 0.2476
Days posttreatment

Chemical name	Orthosulfamuron	**Arkansas sediment**
PC code	108209	
MRID	46578985	
Guideline No.	164-1 and 164-2	

Half-life (days) = 6.2 ***Calculated using all available data**

Days posttreatment	Orthosulfamuron (mg/kg)	Ln (orthosulfamuron)
3	0.171	-1.766
3	0.182	-1.704
3	0.193	-1.645
7	0.050	-2.996
7	0.043	-3.147
7	0.073	-2.617
15	0.021	-3.863
15	0.019	-3.963
15	0.017	-4.075
22	0.013	-4.343
22	0.008	-4.828
22	0.012	-4.423
30	0.008	-4.828
30	0.007	-4.962
30	0.008	-4.828
60	<0.005	
60	<0.005	
60	<0.005	

* Data obtained from Appendix 4, Table XIV, p. 344 in the study report.

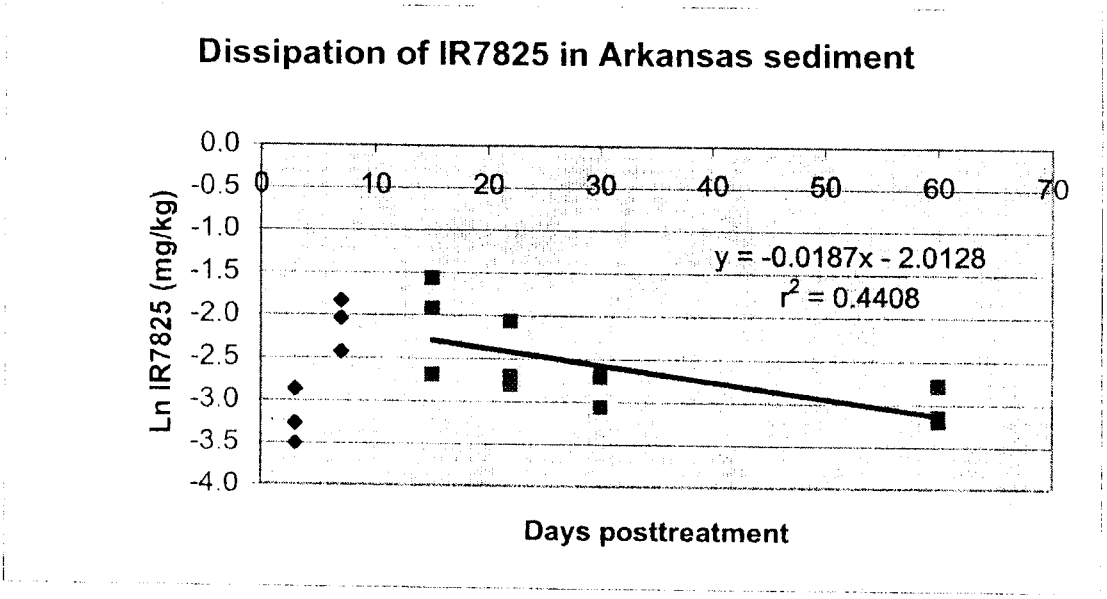


IR7825

Half-life (days) = 37.1 ***Calculated using 15-60 day data**

Days posttreatment	IR7825 (mg/kg)	Ln (IR7825)
3	0.038	-3.270
3	0.057	-2.865
3	0.030	-3.507
7	0.160	-1.833
7	0.130	-2.040
7	0.088	-2.430
15	0.209	-1.565
15	0.147	-1.917
15	0.068	-2.688
22	0.127	-2.064
22	0.061	-2.797
22	0.067	-2.703
30	0.067	-2.703
30	0.047	-3.058
30	0.071	-2.645
60	0.043	-3.147
60	0.062	-2.781
60	0.041	-3.194

* Data obtained from Appendix 4, Table XV, p. 346 in the study report.

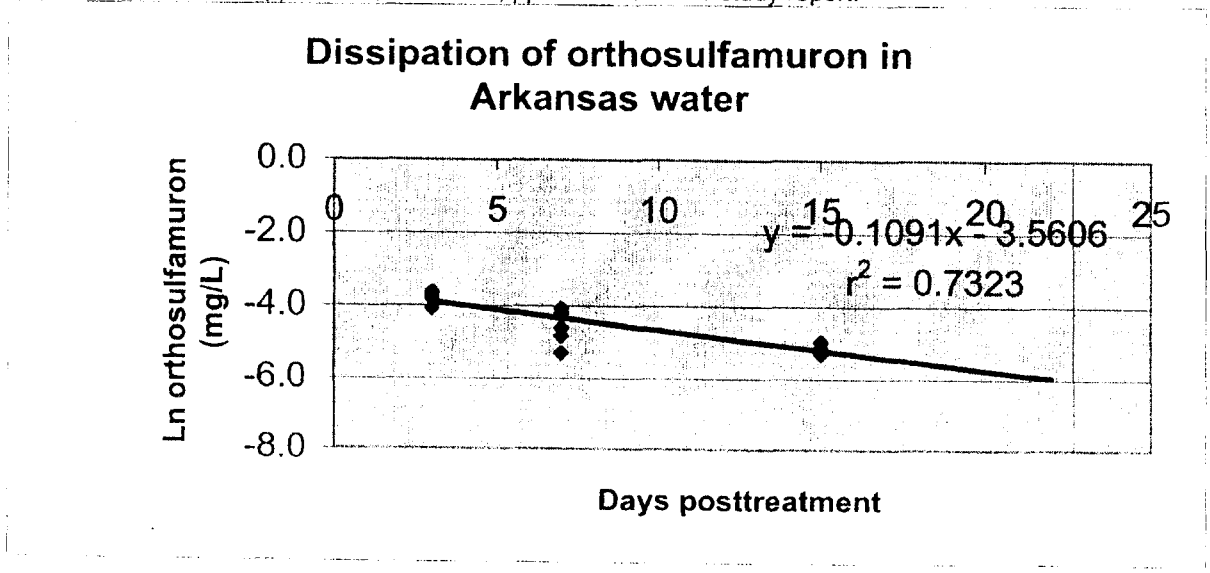


Chemical name	Orthosulfamuron	**Arkansas water**
PC code	108209	
MRID	46578985	
Guideline No.	164-1 and 164-2	

Half-life (days) = 6.4 ***Calculated using all available data**

Days posttreatment	Orthosulfamuron (mg/L)	Ln (orthosulfamuron)
3	0.017	-4.075
3	0.022	-3.817
3	0.027	-3.612
3	0.026	-3.650
3	0.024	-3.730
3	0.025	-3.689
7	0.005	-5.298
7	0.008	-4.828
7	0.015	-4.200
7	0.010	-4.605
7	0.017	-4.075
7	0.017	-4.075
15	0.007	-4.962
15	0.006	-5.116
15	0.006	-5.116
15	0.005	-5.298
15	<0.005	
15	0.006	-5.116
22	<0.005	
22	<0.005	
22	<0.005	
22	<0.005	
22	<0.005	
22	<0.005	

* Data obtained from Appendix 4, Table XIX, pp. 354-355 in the study report.

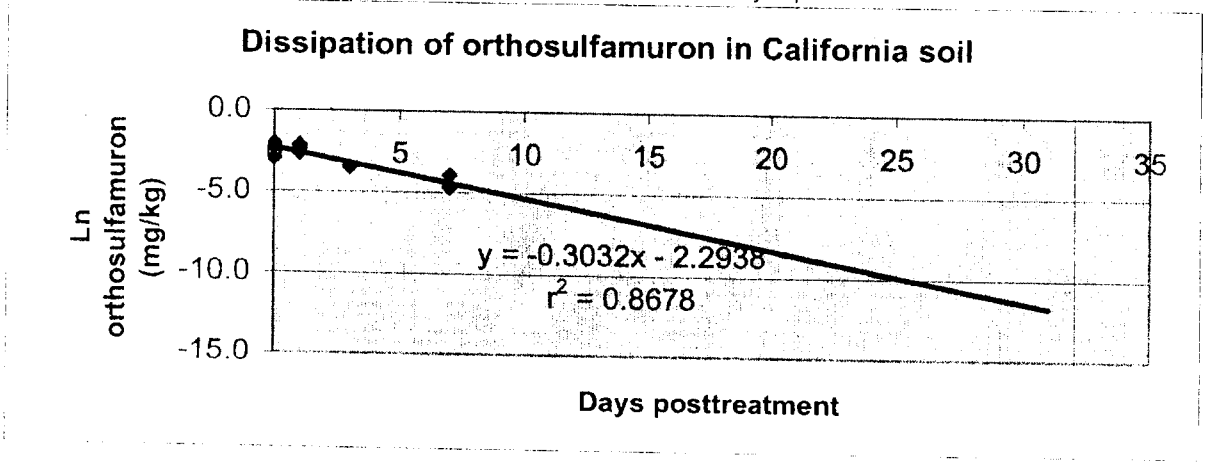


Chemical name	Orthosulfamuron	**California soil**
PC code	108209	
MRID	46578985	
Guideline No.	164-1 and 164-2	

Half-life (days) = 2.3 ***Calculated using all available data**

Days posttreatment	Orthosulfamuron (mg/kg)	Ln (orthosulfamuron)
0	0.103	-2.273
0	0.052	-2.957
0	0.127	-2.064
1	0.072	-2.631
1	0.095	-2.354
1	0.117	-2.146
3	0.031	-3.474
3	<0.005	
3	<0.005	
7	0.009	-4.711
7	0.010	-4.605
7	0.020	-3.912
15	<0.005	
15	<0.005	
15	<0.005	
31	ND	
31	ND	
31	<0.005	
62	ND	
62	ND	
62	ND	
122	ND	
122	ND	
122	ND	
194	ND	
194	ND	
194	<0.005	

* Data obtained from Appendix 4, Table IX, pp. 294-295 in the study report.

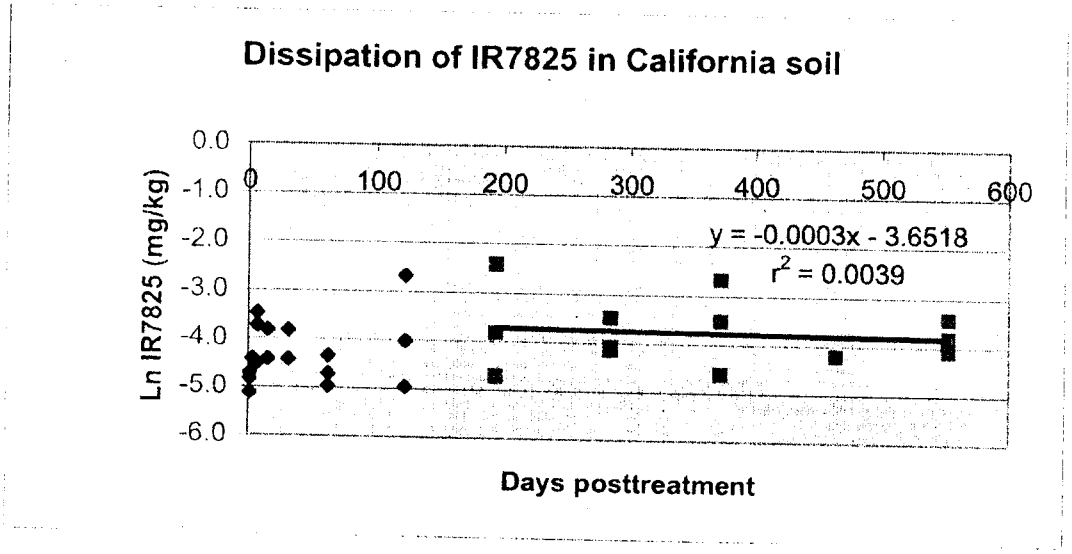


IR7825

Half-life (days) = 177.7 ***Calculated using 194-553 day data**

Days posttreatment	IR7825 (mg/kg)	Ln (IR7825)
0	<0.005	
0	<0.005	
0	<0.005	
1	0.006	-5.116
1	0.008	-4.828
1	0.009	-4.711
3	0.012	-4.423
3	<0.005	
3	<0.005	
7	0.011	-4.510
7	0.024	-3.730
7	0.031	-3.474
15	0.022	-3.817
15	0.022	-3.817
15	0.012	-4.423
31	<0.005	
31	0.012	-4.423
31	0.022	-3.817
62	0.007	-4.962
62	0.009	-4.711
62	0.013	-4.343
122	0.007	-4.962
122	0.018	-4.017
122	0.069	-2.674
194	0.022	-3.817
194	0.009	-4.711
194	0.089	-2.419
284	0.017	-4.075
284	0.016	-4.135
284	0.031	-3.474
372	0.069	-2.674
372	0.010	-4.605
372	0.030	-3.507
464	0.015	-4.200
464	<0.005	
464	<0.005	
553	0.022	-3.817
553	0.017	-4.075
553	0.033	-3.411

* Data obtained from Appendix 4, Table X, pp. 309-311 in the study report.

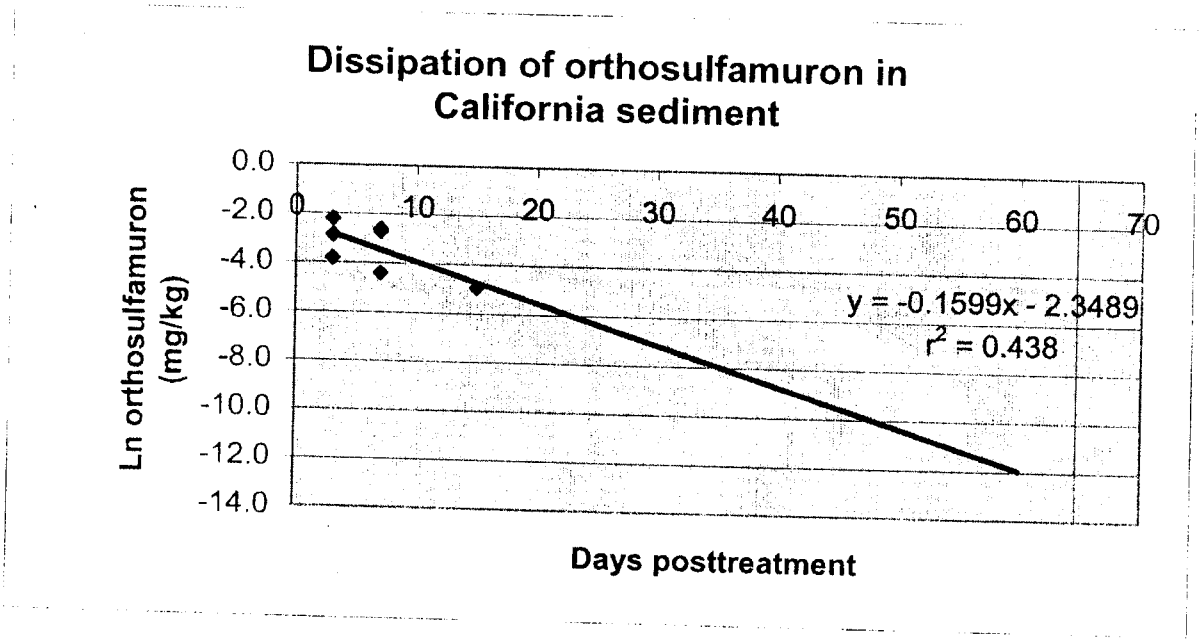

Dissipation of IR7825 in California soil
0.0
-1.0
-2.0
-3.0
-4.0
-5.0
-6.0
Ln IR7825 (mg/kg)
0
100
200
300
400
500
600
y = -0.0003x - 3.6518
r2 = 0.0039
Days posttreatment

Chemical name	Orthosulfamuron	**California sediment**
PC code	108209	
MRID	46578985	
Guideline No.	164-1 and 164-2	

Half-life (days) = **4.3** ***Calculated using all available data**

Days posttreatment	Orthosulfamuron (mg/kg)	Ln (orthosulfamuron)
3	0.056	-2.882
3	0.022	-3.817
3	0.109	-2.216
7	0.073	-2.617
7	0.012	-4.423
7	0.066	-2.718
15	ND	
15	0.007	-4.962
15	<0.005	
30	ND	
30	ND	
30	<0.005	
60	ND	
60	ND	
60	ND	

* Data obtained from Appendix 4, Table XIV, p. 345 in the study report.



IR7825

Half-life (days) = 29.6 ***Calculated using 7-60 day data**

Days posttreatment	IR7825 (mg/kg)	Ln (IR7825)
3	0.014	-4.269
3	0.013	-4.343
3	0.056	-2.882
7	0.162	-1.820
7	0.027	-3.612
7	0.110	-2.207
15	0.012	-4.423
15	0.047	-3.058
15	0.040	-3.219
30	<0.005	
30	0.014	-4.269
30	0.032	-3.442
60	0.016	-4.135
60	0.013	-4.343
60	0.023	-3.772

* Data obtained from Appendix 4, Table XV, p. 347 in the study report.