Test Material:	Flazasulfuron
MRID:	49360101
Title:	Validation of Method GPL-MTH-082: Analytical Method for the Determination of SL-160 and its Metabolites DTPU, DTPP, and TPSA in Water by LC-MS/MS
MRID:	49360102
Title:	Independent Laboratory Validation of Ishihara Sangyo Kaisha (ISK) Analytical Method for the Determination of SL-160 and Its Metabolites DTPU, DTPP, and TPSA in Surface and Drinking Water by LC-MS/MS (Document Number: GPL-MTH-082)
EPA PC Code:	119011
OCSPP Guideline:	850.6100

For CDM Smith

Primary Reviewer: Lynne Binari **Signature:** 

Date: 4/2/15

Secondary Reviewer: Lisa Muto **Signature:** 

Date: 4/2/15

QC/QA Manager: Joan Gaidos **Signature:** 

Date: 4/2/15

Analytical method for flazasulfuron (SL-160) and its transformation products DTPU, DTPP, and TPSA in water

Reports: ECM: EPA MRID No.: 49360101. Schoenau, E. 2014. Validation of Method GPL-MTH-082: Analytical Method for the Determination of SL-160 and its Metabolites DTPU, DTPP, and TPSA in Water by LC-MS/MS. GPL Study No.: 140546 and Report No.: IB-2014-JLW-009-01-01. Report prepared by Golden Pacific Laboratories, LLC (GPL), Fresno, California, sponsored by Ishihara Sangyo Kaisha, Ltd., Osaka, Japan, and submitted by ISK Biosciences Corporation, Concord, Ohio; 172 pages. Final report issued April 4, 2014.
ILV: EPA MRID No.: 49360102. Testman, R. 2014. Independent Laboratory Validation of Ishihara Sangyo Kaisha (ISK) Analytical Method for the Determination of SL-160 and Its Metabolites DTPU, DTPP, and TPSA in Surface and Drinking Water by LC-MS/MS (Document Number: GPL-MTH-082). GPL Study No.: 140547 and Report No.: IB-2014-JLW-010-00-01. Report prepared by Golden Pacific Laboratories, LLC (GPL), Fresno, California, sponsored by Ishihara Sangyo Kaisha, Ltd., Osaka, Japan, and submitted by ISK Biosciences Corporation, Concord, Ohio; 189 pages. Final report issued April 10, 2014.

Document No.: MRIDs 49360101 & 49360102

Guideline: 850.6100

Statements: ECM: The study was conducted in accordance with USEPA Good Laboratory Practice (GLP) standards, with the exception of the surface water characterization (p. 3 of MRID 49360101). Signed and dated Data Confidentiality, GLP, and Quality Assurance Statements were provided (pp. 2-4). The certification of the authenticity of the study report is included in the Quality Assurance Statement (p. 4).
ILV: The study was conducted in accordance with USEPA GLP standards, with the exception of the surface water characterization (p. 3 of MRID 49360102). Signed and dated Data Confidentiality, GLP, and Quality Assurance Statements were provided (pp. 2-4). The certification of the authenticity of the study report is included in the Quality Assurance Statement (p. 4).

Classification: This analytical method is classified as unacceptable. An updated ECM implementing an ILV modification to the method was not provided. The determinations of the LOQ and LOD were not based on scientifically acceptable procedures. The LOQs are three orders of magnitude greater than the lowest toxicological level of concern in water.

PC Code: 119011

Reviewer:

Andrew Shelby
Physical Scientist

Signature:
Date: 5/22/2015

Executive Summary

This analytical method, GPL-MTH-082, is designed for the quantitative determination of flazasulfuron and its transformation products DTPU, DTPP, and TPSA in water using LC/MS/MS. The method is quantitative for flazasulfuron, DTPU, and DTPP at the stated LOQ of 0.05 µg/L and for TPSA at the stated LOQ of 0.20 µg/L. The LOQs are three orders of magnitude greater than the lowest toxicological level of concern in water. The independent laboratory validated the method for analysis of flazasulfuron, DTPU, DTPP, and TPSA at the LOQ and 10x LOQ in surface water after one trial and in drinking water after two trials. The independent laboratory found that water containing chlorine needs to be neutralized with sodium bisulfite prior to fortification and analysis to yield accurate recoveries of flazasulfuron and DTPP. An updated ECM implementing this ILV modification was not provided.

Table 1. Analytical Method Summary

Analyte(s) by Pesticide	MRID		EPA Review	Matrix	Method Date (dd/mm/yyyy)	Registrant	Analysis	Limit of Quantitation (LOQ)
	Environmental Chemistry Method	Independent Laboratory Validation						
Flazasulfuron (SL-160)	49360101	49360102		Surface and drinking water	Original method: 26/03/2014 ECM validation: 04/04/2014	ISK Biosciences Corporation	LC/MS/MS	0.05 µg/L
DTPU								
DTPP								
TPSA								0.2 µg/L

I. Principle of the Method

Water (10 mL) was fortified with a mixed standard solution of flazasulfuron (SL-160), DTPU, DTPP, and TPSA in acetonitrile for procedural recoveries (pp. 16-19; Appendix B, pp. 63-71; Appendix C, p. 77 of MRID 49360101). Reference substances were supplied by Midwest Research Institute, Kansas City, Missouri. Surface water was obtained from Fresno Irrigation District "Herndon Canal No. 39" near Gates Avenue Bridge. An aliquot of water (volume variable) is combined with acetonitrile (ACN) at a 9:1 ratio (***e.g.*** 4.5 mL sample:0.5 mL ACN). The sample is then manually mixed (*ca*. 5 seconds), filtered (PTFE 0.45-µm), and analyzed by LC/MS/MS.

Samples are analyzed using an AB Sciex API 4000 LC/MS/MS with TurboIonSource and electrospray ionization (ESI) interface (pp. 19-21; Appendix B, pp. 71-72 of MRID 49360101). The following LC conditions were used: Phenomenex Luna C18(2) column (3.00 mm x 50 mm, 3 µm, 100 Å, column temperature ambient) preceded by a SecurityGuard™ C18 cartridge (2.00 x 4 mm), using a mobile phase of (A) 0.2% formic acid in acetonitrile and (B) 0.2% formic acid in water [percent A:B (v:v) at 0.0 min. 30:70, 5.0-6.0 min. 90:10, 6.1-8.0 min 30:70]. Injection volume was 10 µL. The following MS/MS conditions were used: ESI in positive ion mode detection and multiple reaction monitoring (MRM). Analytes are identified using two ion transitions; one for quantitation (Q, "primary") and one for confirmation (C). Ion transitions monitored were as follows: *m/z* 407.9→181.8 (Q) and *m/z* 407.9→139.1 (C) for flazasulfuron (SL-160), *m/z* 343.9→300.9 (Q) and *m/z* 343.9→281.1 (C) for DTPU, *m/z* 300.9→281.1 (Q) and *m/z* 300.9→238.1 (C) for DTPP, and *m/z* 227.0→145.8 (Q) and *m/z* 227.0→126.0 (C) for TPSA. Expected retention times were *ca*. 3.2, 2.4, 2.7, and 1.5 minutes for flazasulfuron (SL-160), DTPU, DTPP, and TPSA, respectively.

ILV: Reference substances were supplied by Midwest Research Institute (p. 15 of MRID 49360102). Drinking (tap) water was obtained at the independent laboratory (Fresno municipal supply, p. 18). Surface water was obtained from the San Joaquin River in Fresno, California, near Gravel Haut Road. The independent laboratory performed the method as written, with the following exception: for Trial 2/drinking water the matrix was treated with 1 mL of 5 mg/mL sodium bisulfite solution per liter of drinking water to neutralize the chlorine (pp. 17-21; Appendix B, p. 60).

LOQ and LOD: In the ECM and ILV, the LOQ for flazasulfuron (SL-160), DTPU, and DTPP was 0.05 µg/L and the LOQ for TPSA was 0.2 µg/L (pp. 17-18, 26; Appendix B, p. 70 of MRID 49360101; pp. 17, 26-27 of MRID 49360102). LODs in the ECM and ILV were 0.01 µg/L for flazasulfuron, DTPU, and DTPP, and 0.088 µg/L for TPSA.

II. Recovery Findings

ECM (MRID 49360101): Mean recoveries and relative standard deviations (RSDs) were within guidelines (mean 70-120%; RSD ≤20%) for analysis of flazasulfuron (SL-160) and its transformation products DTPU and DTPP in surface (irrigation canal) water at fortification levels of 0.05 µg/L (LOQ), 0.50 µg/L (10x LOQ), and 5.0 µg/L (100x LOQ; Tables I-VI, pp. 30-40). Mean recoveries and RSDs were within guidelines for analysis of flazasulfuron transformation product TPSA in surface water at fortification levels of 0.20 µg/L (LOQ), 2.0 µg/L (10x LOQ), and 20.0 µg/L (100x LOQ), with the following exception: confirmation ion at 0.20 µg/L (mean recovery 134%; Tables VII-VIII, pp. 41-44; DER Attachment 2). The method defines that the TPSA confirmation ion pair (*m/z* 227.0→126.0) can only be used for peak identity confirmation below 10x LOQ (2.0 µg/L) due to limited sensitivity (p. 26; Appendix B, p. 65). Analytes were identified and quantified using two ion transitions; quantitation ion and confirmation ion recovery results were comparable, except for TPSA at the LOQ. The water matrix was characterized (non-GLP) by BSK Laboratories, Fresno, California (p. 18).

ILV (MRID 49360102): Mean recoveries and relative standard deviations (RSDs) were within guidelines (mean 70-120%; RSD ≤20%) for analysis of flazasulfuron and its products DTPU and DTPP in surface (river) water and drinking (tap) water at fortification levels of 0.05 µg/L (LOQ) and 0.50 µg/L (10x LOQ), with the following exception: confirmation ion for 0.50 µg/L flazasulfuron (RSD 21.3%) in surface water (Tables I-VI, pp. 30-35; Tables IX-XIV, pp. 38-43). Mean recoveries and RSDs were within guidelines for analysis of TPSA in surface and drinking water at fortification levels of 0.20 µg/L (LOQ) and 2.0 µg/L (10x LOQ), with the following exceptions: confirmation ion for 2.0 µg/L TPSA in surface water (RSD 28.0%) and confirmation ion for 0.20 µg/L TPSA (mean recovery 125%, RSD 61.4%) and 2.0 µg/L TPSA (RSD 80.2%) in drinking water (Tables VII-VIII, pp. 36-37; Tables XV-XVI, pp. 44-45; DER Attachment 2). The study author reported that the TPSA confirmation ion pair "may not meet all validation requirements for quantitation" (pp. 13, 25-26). The method was validated for all four analytes at both fortification levels in surface water after one trial and in drinking water after two trials (pp. 12, 18, 22, 27). The first drinking water trial was unsuccessful due to degradation of flazasulfuron and DTPP from residual chlorine; therefore, the drinking water was treated with sodium bisulfite to neutralize the chlorine for the second trial. A City of Fresno Department of Public Utilities Water Quality Report was provided for the drinking water matrix, while the surface water was characterized (non-GLP) by BSK Laboratories, Fresno, California (p. 18; Appendix C, pp. 61-68).

Table 2. Initial Validation Method Recoveries for Flazasulfuron (SL-160) and Its Transformation Products DTPU, DTPP, and TPSA in Surface (Irrigation Canal) Water

Analyte	Fortification Level (µg/L)	Number of Tests	Recovery Range (%)	Mean Recovery (%)	Standard Deviation (%)	Relative Standard Deviation (%)[1]
	Quantitation Ion					
Flazasulfuron *m/z* 407.9→181.8	0.05 (LOQ)	7	84.6-110	100	9.67	9.67
	0.50	7	103-106	104	1.25	1.20
	5.00	7	103-110	106	2.82	2.66
DTPU *m/z* 343.9→300.9	0.05 (LOQ)	7	97.0-103	99.9	2.28	2.28
	0.50	7	100-106	104	2.37	2.28
	5.00	7	100-104	102	1.57	1.54
DTPP *m/z* 300.9→281.1	0.05 (LOQ)	7	97.6-108	103	3.37	3.27
	0.50	7	100-105	103	1.77	1.72
	5.00	7	101-106	104	1.80	1.73
TPSA *m/z* 227.0→145.8	0.20 (LOQ)	7	97.0-115	104	6.47	6.22
	2.00	7	93.0-103	98.6	3.14	3.18
	20.0	7	94.5-105	101	3.99	3.95
	Confirmation Ion					
Flazasulfuron *m/z* 407.9→139.1	0.05 (LOQ)	7	93.0-121	108	9.59	8.88
	0.50	7	97.4-109	104	4.21	4.05
	5.00	7	92.4-109	103	5.61	5.45
DTPU *m/z* 343.9→281.1	0.05 (LOQ)	7	90.4-105	96.6	4.74	4.91
	0.50	7	101-106	104	1.81	1.74
	5.00	7	95.4-105	100	2.97	2.97
DTPP *m/z* 300.9→238.1	0.05 (LOQ)	7	90.6-105	99.1	5.75	5.80
	0.50	7	97.0-105	100	2.83	2.83
	5.00	7	97.2-106	102	2.69	2.64
TPSA *m/z* 227.0→126.0	0.20 (LOQ)	7	NA[2] [94.5-168]	NA [134]	NA [24.0]	NA [17.9]
	2.00	7	94.5-110	105	5.82	5.54
	20.0	7	97.5-112	106	6.18	5.83

Data (uncorrected recovery results, p. 23) were obtained from Tables I-VIII, pp. 29-44 of MRID 49360101.

1 Coefficient of Variance in study tables (Tables I-VIII, pp. 29-44).

2 Study author did not present tabular results for TPSA at the LOQ stating that the confirmatory ion pair cannot be used to quantitate below 10xLOQ due to limited sensitivity, and the confirmatory ion pair can only be used for peak identity confirmation under 10x LOQ (pp. 12, 25-26; Table VIII, p. 43; Appendix B, p. 65). Bracketed results were determined by reviewer using data obtained from Raw Data Summary Spreadsheets (Appendix E, p. 120; DER Attachment 2).

Table 3. Independent Validation Method Recoveries for Flazasulfuron (SL-160) and Its Transformation Products DTPU, DTPP, and TPSA in Surface and Drinking Water

Analyte	Fortification Level (µg/L)	Number of Tests	Recovery Range (%)	Mean Recovery (%)	Standard Deviation (%)	Relative Standard Deviation (%)[1]
	Surface (River) Water					
	Quantitation Ion					
Flazasulfuron *m/z* 407.9→181.8	0.05 (LOQ)	5	94.4-109	102	5.63	5.52
	0.50	5	92.8-105	99.8	5.43	5.44
DTPU *m/z* 343.9→300.9	0.05 (LOQ)	5	93.2-100	96.4	3.33	3.45
	0.50	5	85.6-97.6	89.6	4.70	5.25
DTPP *m/z* 300.9→281.1	0.05 (LOQ)	5	102-110	105	3.46	3.30
	0.50	5	92.6-103	98.9	4.34	4.39
TPSA *m/z* 227.0→145.8	0.20 (LOQ)	5	98.0-118	104	8.26	7.94
	2.00	5	91.0-113	103	7.95	7.72
	Confirmation Ion					
Flazasulfuron *m/z* 407.9→139.1	0.05 (LOQ)	5	97.2-121	105	10.1	9.62
	0.50	5	81.6-135	104	22.2	**21.3**
DTPU *m/z* 343.9→281.1	0.05 (LOQ)	5	80.4-101	91.2	8.00	8.77
	0.50	5	64.8-94.0	78.1	13.2	16.9
DTPP *m/z* 300.9→238.1	0.05 (LOQ)	5	101-113	107	4.95	4.63
	0.50	5	95.4-109	102	6.40	6.27
TPSA *m/z* 227.0→126.0	0.20 (LOQ)	5	91.0-151	117	23.0	19.7
	2.00	5	61.0-144	116	32.5	**28.0**
	Drinking (Tap) Water					
	Quantitation Ion					
Flazasulfuron *m/z* 407.9→181.8	0.05 (LOQ)	5	97.2-113	105	5.87	5.59
	0.50	5	92.2-102	99.8	4.29	4.30
DTPU *m/z* 343.9→300.9	0.05 (LOQ)	5	79.2-83.8	80.8	1.76	2.18
	0.50	5	74.2-76.8	75.4	1.07	1.42
DTPP *m/z* 300.9→281.1	0.05 (LOQ)	5	111-118	115	3.11	2.70
	0.50	5	110-118	113	3.78	3.35
TPSA *m/z* 227.0→145.8	0.20 (LOQ)	5	91.5-102	95.2	4.42	4.64
	2.00	5	73.0-110	90.6	13.8	15.2
	Confirmation Ion					
Flazasulfuron *m/z* 407.9→139.1	0.05 (LOQ)	5	82.0-114	104	13.2	12.7
	0.50	5	82.8-114	103	12.7	12.3
DTPU *m/z* 343.9→281.1	0.05 (LOQ)	5	60.6-89.2	79.7	11.0	13.8
	0.50	5	65.2-79.8	72.7	5.85	8.05
DTPP *m/z* 300.9→238.1	0.05 (LOQ)	5	108-121	116	5.17	4.46
	0.50	5	111-116	114	1.87	1.64
TPSA *m/z* 227.0→126.0	0.20 (LOQ)	5	NA[2] [ND-203]	NA [**125**]	NA [76.6]	NA [**61.4**]
	2.00	5	NA [ND-217]	NA [99.4]	NA [79.7]	NA [**80.2**]

Data (uncorrected recovery results, p. 23) were obtained from Tables I-XVI, pp. 30-45 of MRID 49360102.

1 Coefficient of Variance in study tables (Tables I-XVI, pp. 30-45).

2 Study author did not present tabular results for TPSA stating that the confirmatory ion pair "may not meet all validation requirements for quantitation", and the confirmatory ion pair was used for verification of peak identity (Table XVII, p. 45). Confirmation ion for TPSA was not quantitated due to low signal/noise at LOQ (pp. 13, 26). Bracketed results were determined by reviewer using data obtained from Raw Data Summary Spreadsheets (Appendix E, p. 121; DER Attachment 2). ND = not detected and for statistics ND = LOD = 0.088 µg/L.

III. Method Characteristics

In the ECM and ILV, the LOQ and LOD for flazasulfuron (SL-160), DTPU, and DTPP in water were 0.05 µg/L and 0.01 µg/L, respectively, and for TPSA were 0.2 µg/L and 0.088 µg/L, respectively (pp. 17-18, 26 of MRID 49360101; pp. 17, 26-27 of MRID 49360102). The ECM defined the LOQs as the lowest fortification levels at which acceptable recovery data were obtained. The ECM set the LODs as the lowest calibration standards used in the calibration curves, or 0.02 ng/mL (equivalent to 0.01 µg/L in sample matrix) for flazasulfuron, DTPU, and DTPP, and 0.08 ng/mL (equivalent to 0.088 µg/L in sample matrix) for TPSA.

Table 4. Method Characteristics for Flazasulfuron (SL-160) and Its Transformation Products DTPU, DTPP, and TPSA in Surface and Drinking Water

			Flazasulfuron	**DTPU**	**DTPP**	**TPSA**
Limit of Quantitation (LOQ)			0.05 µg/L			0.2 µg/L
Limit of Detection (LOD)			0.01 µg/L			0.088 µg/L
Linearity (calibration curve r^2 and concentration range)[1]	ECM:	Q ion:	$r^2 = 0.9992$	$r^2 = 0.9992$	$r^2 = 0.9998$	$r^2 = 0.9986$
		C ion:	$r^2 = \mathbf{0.9900}$	$r^2 = 0.9988$	$r^2 = 0.9994$	$r^2 = 0.9998$
	ILV:	Q ion:	$r^2 = 0.9992$-0.9994	$r^2 = 0.9996$-0.9998	$r^2 = 0.9996$-0.9998	$r^2 = 0.9994$-0.9998
		C ion:	$r^2 = 0.9990$-0.9994	$r^2 = 0.9994$-0.9996	$r^2 = 0.9984$-0.9996	$r^2 = 0.9954$-0.9982
	Range:		0.02-1.00 ng/mL			0.08-4.00 ng/mL
Repeatable	ECM:	Surface water:	Yes at LOQ, 10x LOQ, and 100x LOQ, except for confirmation ion at LOQ for TPSA.			
	ILV:	Surface water:	Yes at LOQ and 10x LOQ, except for confirmation ion at 10x LOQ for flazasulfuron and TPSA.			
		Drinking water:	Yes at LOQ and 10x LOQ, except for confirmation ion at LOQ and 10x LOQ for TPSA.			
Reproducible (LOQ)	ILV:	Surface water:	Yes for quantitation and confirmation ions.			
		Drinking water:	Yes for quantitation and confirmation ions.			Yes for quantitation ion only.[2]
Specific	ECM:		Yes; interferences at the analyte retention times were ≤50% (based on peak height) of the LOD (lowest calibration standard), except for the TPSA confirmation ion analyses which were <LOD.			
	ILV:					

Data were obtained from pp. 17-18, 26; Appendix B, p. 65; Appendix E, pp. 113-120; Appendix F, pp. 125, 132-133, 137, 144-145, 149, 156-157, 161, 168-169 of MRID 49360101; pp. 17, 26-27; Appendix E, pp. 106-121; Appendix F, pp. 134, 141-142, 145, 148, 155-156, 159, 162, 169-170, 173, 176, 183-184, 187 of MRID 49360102; DER Attachment 2.

Linearity is satisfactory when $r^2 \geq 0.995$.

1 ECM and ILV coefficient of determination (r^2) values from 1/x weighted linear regression (Appendix E, pp. 113-120 of MRID 49360101; Appendix E, pp. 106-121 of MRID 49360102). Q = quantitation (primary) ion, C = confirmation ion.

2 The method defines that the TPSA confirmation ion pair (*m/z* 227.0→126.0) can only be used for peak identity confirmation below 10x LOQ (2.0 µg/L) due to limited sensitivity (p. 26; Appendix B, p. 65 of MRID 49360101).

IV. Method Deficiencies and Reviewer's Comments

1. Due to low recoveries of flazasulfuron and DTPP in Trial 1 using drinking water, the matrix was treated with 1 mL of 5 mg/mL sodium bisulfite solution per liter of drinking water to neutralize the chlorine for Trial 2 (pp. 17-21; Appendix B, p. 60 of MRID 49360102). This modification was approved by the Study Sponsor (p. 22). The independent laboratory noted that a limitation to the ECM is that "Water containing chlorine needs to be treated with sodium bisulfite at the time of collection if accurate SL-160 [flazasulfuron] and DTPP data are desired." (p. 27). An updated ECM implementing the ILV modification was not provided.

2. The determination of the LOQ and LOD were not based on scientifically acceptable procedures as defined in 40 CFR Part 136, Appendix B. The ECM defined the LOQs as the lowest fortification levels at which acceptable recovery data were obtained and set the LODs as the lowest calibration standards used in the calibration curves (p. 26 of MRID 49360101). Detection limits should not be based on the arbitrarily selected lowest concentration in the spiked samples. Additionally, the lowest toxicological level of concern in water was not reported. An LOQ above toxicological levels of concern results in an unacceptable method classification.

3. Method recoveries met OCSPP Guideline 850.6100 criteria for precision and accuracy (mean recoveries for replicates at each spiking level between 70% and 120% and relative standard deviations (RSD) ≤20%) at the stated LOQ and at higher concentrations with the following exceptions:

   For the ECM, confirmation ion for 0.20 µg/L (LOQ) TPSA (mean recovery 134%; Table I-VIII, pp. 29-44 of MRID 49360101; DER Attachment 2).

   For the ILV, confirmation ion for 0.50 µg/L (10x LOQ) flazasulfuron (RSD 21.3%) and 2.00 µg/L (10x LOQ) TPSA (RSD 28.0%) in surface water and 0.20 µg/L (LOQ, mean recovery 125%, RSD 61.4%) and 2.00 µg/L (RSD 80.2%) TPSA in drinking water (Tables I-XVI, pp. 30-45 of MRID 49360102; DER Attachment 2).

   The method specifies that the TPSA confirmation ion pair (*m/z* 227.0→126.0) can only be used for peak identity confirmation below 10x LOQ (2.0 µg/L) due to limited sensitivity (pp. 12, 26; Appendix B, p. 65 of MRID 49360101). The ILV did not quantitate the TPSA confirmation ion at the LOQ or 10x LOQ in drinking water due to low signal/noise (p. 13; Table XVI, p. 45; Appendix F, pp. 188-189 of MRID 49360102).

4. Although the ILV was conducted at the same testing facility that developed and validated the method, the ILV was conducted using an independent Study Director and Chemist, separate equipment and instruments, and different lots of reagents. Care was taken to ensure the independence of the ILV staff from those that conducted the original validation. (pp. 6, 12 of MRID 49360102; p. 6 of MRID 49360101).

5. It was reported for the ILV that 1 person-hour is required to prepare a sample set, with overnight LC/MS/MS analysis, followed by 0.5 hour of data calculation and tabulation (p.

22 of MRID 49360102). Therefore, at a maximum, two calendar days are required for sample preparation, analysis, and data calculation/tabulation.

V. References

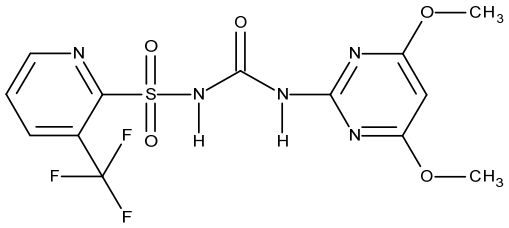
U.S. Environmental Protection Agency. 2012. Ecological Effects Test Guidelines, OCSPP 850.6100, Environmental Chemistry Methods and Associated Independent Laboratory Validation. Office of Chemical Safety and Pollution Prevention, Washington, DC. EPA 712-C-001.

40 CFR Part 136. Appendix B. Definition and Procedure for the Determination of the Method Detection Limit-Revision 1.11, pp. 317-319.

Attachment 1: Chemical Names and Structures

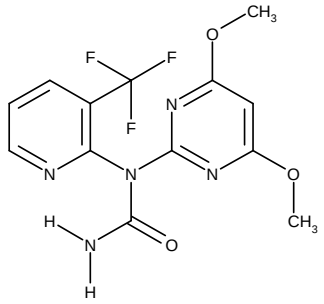
Flazasulfuron (SL-160)

IUPAC Name: 1-(4,6-Dimethoxypyrimidin-2-yl)-3-(3-trifluoromethyl-2-pyridylsulphonyl)urea
CAS Name: N-[[(4,6-dimethoxy-2-pyrimidinyl)amino]carbonyl]-3-(trifluoromethyl)-2-pyridinesulfonamide
CAS Number: 104040-78-0
SMILES String: n1cccc(C(F)(F)F)c1S(=O)(=O)NC(=O)Nc2nc(OC)cc(OC)n2



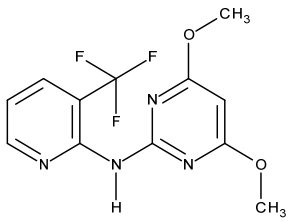
DTPU

IUPAC Name: 1-(4,6-Dimethoxypyrimidin-2-yl)-1-[3-(trifluoromethyl)-2-pyridyl]urea
CAS Name: Not reported.
CAS Number: Not reported.
SMILES String: COc1cc(nc(n1)N(c2c(cccn2)C(F)(F)F)C(=O)N)OC



DTPP

IUPAC Name: 4,6-Dimethoxy-2-(3-trifluoromethyl-2-pyridylamino)pyrimidine
CAS Name: Not reported.
CAS Number: Not reported.
SMILES String: COc1cc(nc(n1)Nc2c(cccn2)C(F)(F)F)OC



TPSA

IUPAC Name: 3-Trifluoromethyl-2-pyridinesulphonamide

CAS Name: Not reported.

CAS Number: Not reported.

SMILES String: c1cc(c(nc1)S(=O)(=O)N)C(F)(F)F