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(5 12-04)

Data Evaluation Report on the leaching of penoxsulam (XR-638) in unaged soil columns

PMRA Submission Number {.....} EPA MRID Number 45834802

Data Requirement: PMRA Data Code:
EPA DP Barcode: D288160
OECD Data Point:
EPA Guideline: 163-1

Test material:
Chemical names:

IUPAC: 6-(2,2-Difluoroethoxy)-N-(5,8-dimethoxy-s-triazolo[1,5-c]pyrimidin-2-yl)-α,α,α-trifluoro-o-toluenesulfonamide.
3-(2,2-Difluoroethoxy)-N-(5,8-dimethoxy[1,2,4]triazolo[1,5-c]pyrimidin-2-yl)-α,α,α-trifluorotoluene-2-sulfonamide.

CAS : 2-(2,2-Difluoroethoxy)-N-(5,8-dimethoxy[1,2,4]triazolo[1,5-c]pyrimidin-2-yl)-6-(trifluoromethyl)benzenesulfonamide.

CAS No: 219714-96-2.

Synonyms: XDE-638.

SMILES string: n1c(nc2n1c(ncc2OC)OC)NS(=O)(=O)c3c(cccc3C(F)(F)F)OCC(F)F.

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(1)

Data Evaluation Report on the leaching of penoxsulam (XR-638) in unaged soil columns

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Study Acceptability: This study is classified supplemental. Both portions of this study, conducted using [triazolopyrimidine-2-^{14}C]penoxsulam in a free-draining and saturated leaching conditions, are scientifically valid, but do not satisfy Subdivision N Guideline §163-1 data requirements for a mobility study using aged soil because: (i) only one ring in penoxsulam was radiolabeled, (ii) only two test soils were studied in one portion of the primary study, rather than the required minimum of four test soils, (iii) three test soils were foreign in origin and not completely characterized according to the USDA Soil Textural Classification System or compared to U.S. soils, (iv) no test soil contained an organic matter content less than 1%, and (v) the test soils were leached with 20 cm of water, rather than the required 50.8 cm.

ABSTRACT

Leaching - Unaged Column

The column leaching of [triazolopyrimidine-2-^{14}C] 3-(2,2-difluoroethoxy)-*N*-(5,8-dimethoxy[1,2,4]triazolo[1,5-c]pyrimidin-2-yl)-α,α,α,-trifluorotoluene-2-sulfonamide (penoxsulam; XR-638; radiochemical purity 99%; specific activity 949.1 Mbq/mmol, 25.26 mCi/mmol, 117900 dpm/μg; Letcombe Ref. No.: INV 98/05; Inventory No.: INV1352) was studied in the dark for 48 hours in four unaged soils: sandy silt loam (Ogori, Japan; pH 6.5, organic carbon 1.8%); clay loam (Nagaoka, Japan; pH 5.8, organic carbon 2.1%); silt loam (Arkansas, USA; pH 5.7, organic carbon 1.2%); and sandy loam (Ottobiano, Italy; pH 6.4, organic carbon 1.0%). This experiment was conducted in accordance with the Procedures for Assessing the Environmental Fate and Ecotoxicity of Pesticides Part 1, Section 5, SETAC-Europe guidelines (1995), and in compliance with OECD Principles of Good Laboratory Practice. Prior to study initiation, the four test soils were air-dried, sieved (2 mm), and stored in the dark at approximately 4°C. Two types of unaged soil column leaching experiments were performed in the study: a free-draining experiment and a saturated (flooded) experiment. All four soils were used in the free-draining experiment. Only the Nagaoka and Ottobiano soils were used in the saturated experiment. In the free-draining experiment, the flow of aqueous solution was controlled at the top of the column and uncontrolled at the bottom. In the saturated experiment, the flow of aqueous solution was restricted at the bottom so as to maintain a 5 cm layer of the solvent on the top of the column.

Prior to treatment, seven cylindrical glass columns (5 cm height x 5 cm i.d.) with round glass joints were connected together and evenly packed with air-dried soil to a height of 30 cm. The four soil columns of the free-draining experiment were saturated with the 0.01M $CaCl_2$ solution, drained, and maintained in a free-draining condition before application of test compound. The two soil columns of the saturated experiment were saturated with the 0.01M $CaCl_2$ solution, drained, and re-saturated so that there was 5 cm of solution in the top of the soil columns before application of test compound. The amounts of aqueous solution required to saturated all soil columns were recorded (recorded as weight; assumed density of 1 g/mL; p.11; Table 3, p.24).

The [^{14}C]penoxsulam application solution was prepared by diluting a 178-µL aliquot of a [^{14}C]penoxsulam stock solution to 5 mL with distilled water. Aliquots (962 µL) of the [^{14}C]penoxsulam application solution (14.54 µg/mL final concentration; 3.6% acetone by volume) were applied dropwise to the soil columns of the free-draining (on the soil surface) and saturated (on the aqueous solution surface) experiments. The application rate was 14.0 µg of [^{14}C]penoxsulam (0.74 µCi), which was equivalent to 71 g/ha based on the 19.2-cm^2 surface area of the soil column (approximately twice the maximum field application rate of 35 g/ha).

The 0.01M $CaCl_2$ aqueous solution was applied to the four soil columns of the free-draining experiment (20.0-21.5 cm depth) and to the two soil columns of the saturated experiment (depth not recorded) at a constant flow rate over 48 hours (flow maintained by a peristaltic pump). During leaching of the columns, leachates were collected in four (0-5 cm, 5-10 cm, 10-15 cm, and 15-20 cm) and two (0-10 cm and 10-20 cm) approximately equal-volume fractions for the free-draining soil experiment and saturated experiment, respectively.

Following leaching, all of the soil columns were disassembled. The weights of the soil columns post-leaching were determined for each entire column and the six individual sections (0-5 cm, 5-10 cm, 10-15 cm, 15-20 cm, 20-25 cm, and 25-30 cm). The soil in each section was removed, combusted, and analyzed for total radioactivity using LSC. The glass column segments were washed with acetone:water (1:1, v:v) which was then measured for radioactivity via LSC analysis. No further analysis of the saturated experiment soil columns was performed.

The soil sections of the free-draining experiment were extracted with acetonitrile:water (9:1, v:v; best solvent for extraction as determined by a preliminary study), purified with a pre-conditioned Isolute C18 solid phase extraction (SPE) column, and further analyzed by LSC and reverse-phase HPLC. The extracted soils were air-dried, finely ground via mortar and pestle, combusted, and analyzed for total radioactivity using LSC. Leachates were analyzed for total radioactivity using LSC, and only one leachate fraction (15-20 cm fraction of the Ottobiano soil column in the free-draining experiment) contained >2% of the applied radioactivity. The aqueous leachate (*ca.* 100 mL) was passed through the SPE column, the SPE column was eluted with acetonitrile, and the leachate was analyzed by LSC and reverse-phase HPLC.

Free-draining experiment: In unaged Ogori sandy silt loam soil treated with [triazolopyrimidine-2-^{14}C]penoxsulam, total residues were 100.2% of the applied radioactivity (100.2% from the soil columns, 0% from the leachate). The distribution of total radioactivity was 40.2%, 25.5%, 20.0%, 13.0%, 1.5%, and not detected (the 0-5 cm, 5-10 cm, 10-15 cm, 15-20 cm, 20-25 cm, and 25-30 cm soil column depths, respectively. Total extractable [^{14}C]residues were 39.8% of the applied for the 0-5 cm soil column depth, and were 25.3%, 19.8%, 12.8%, 1.4%, and not detected for the 5-10 cm, 10-15 cm, 15-20 cm, 20-25 cm, and 25-30 cm soil column depths, respectively. [Triazolopyrimidine-2-^{14}C]penoxsulam accounted for 36.1% of the applied in the 0-5 cm soil column depth, and were 23.1%, 18.2%, 12.4%, 1.3%, and not detected in the 5-10 cm, 10-15 cm, 15-20 cm, 20-25 cm, and 25-30 cm soil column depths, respectively. The minor transformation product (15-minute retention time) was only measured in the 5-10 cm

and 10-15 cm soil column depths (0.5% and 0.6% of the applied respectively). Nonextractable [^{14}C]residues were a maximum of 3.7% of the applied in the 0-5 cm soil column depth, and were ≤1.7% in all other soil layers.

In unaged Nagaoka clay loam soil treated with [triazolopyrimidine-2-^{14}C]penoxsulam, total residues were 102.9% of the applied radioactivity (102.9% from the soil columns, 0% from the leachate). The distribution of total radioactivity was 102.9% for the 0-5 cm and below detection in all other soil column depths. Total extractable [^{14}C]residues were 101.9% of the applied for the 0-5 cm soil column depth, and were not detected in all other soil column depths (reviewer-calculated). [Triazolopyrimidine-2-^{14}C]penoxsulam accounted for 95.8% of the applied in the 0-5 cm soil column depth, and was not detected in all other soil column depths. The minor transformation product (15-minute retention time) was not detected in any soil column depths. Nonextractable [^{14}C]residues were a maximum of 6.1% of the applied in the 0-5 cm soil column depth, and were not detected in all other soil column depths.

In unaged Arkansas silt loam soil treated with [triazolopyrimidine-2-^{14}C]penoxsulam, total residues were 98.2% of the applied radioactivity (98.2% from the soil columns, 0% from the leachate). The distribution of total radioactivity was 86.1% for the 0-5 cm soil layer, 12.1% for the 5-10 cm soil layer, and below detection in all other soil layers. Total extractable [^{14}C]residues were 85.6% of the applied for the 0-5 cm soil layer, 12% for the 5-10 cm soil layer, and were not detected for all other soil column depths. [Triazolopyrimidine-2-^{14}C]penoxsulam accounted for 80.6% of the applied in the 0-5 cm soil layer, 10.7% in the 5-10 cm soil layer, and was not detected in all other soil column layers. The minor transformation product (15-minute retention time) was a maximum of 1.9% of the applied in the 0-5 cm soil column depth, accounted for 1.0% in the 5-10 cm, and was not detected in any other soil column depths. Nonextractable [^{14}C]residues were a maximum of 3.1% of the applied in the 0-5 cm soil layer, accounted for 0.3% in the 5-10 cm layer, and were not detected in all other soil layers.

In unaged Ottobiano sandy loam soil treated with [triazolopyrimidine-2-^{14}C]penoxsulam, total residues were 98.9% of the applied radioactivity (95.8% from the soil columns, 3.1% from the leachate). The distribution of total radioactivity was 9.7%, 19.9%, 22.6%, 18.6%, 12.3%, and 12.7% for the 0-5 cm, 5-10 cm, 10-15 cm, 15-20 cm, 20-25 cm, and 25-30 cm soil column depths, respectively. Total extractable [^{14}C]residues were 9.6% of the applied for the 0-5 cm soil column depth, and 19.7%, 22.5%, 18.5%, 12.2%, and 12.6% for the 5-10 cm, 10-15 cm, 15-20 cm, 20-25 cm, and 25-30 cm soil column depths, respectively. [Triazolopyrimidine-2-^{14}C]penoxsulam accounted for 8.5% of the applied in the 0-5 cm soil column depth, and 18.9%, 20.9%, 16.8%, 11.4%, and 11.7% in the 5-10 cm, 10-15 cm, 15-20 cm, 20-25 cm, and 25-30 cm soil column depths, respectively. The minor transformation product (15-minute retention time) was a maximum of 0.9% of the applied in the 10-15 cm soil layer. Nonextractable [^{14}C]residues were a maximum of 0.9% of the applied in the 0-5 cm soil column depth, and <0.8% in all other soil layers.

Volatile [^{14}C]organic compounds, $^{14}CO_2$, and bound residues were not measured individually in the free-draining experiment.

Saturated experiment: In unaged Nagaoka clay loam soil treated with [triazolopyrimidine-2-^{14}C]penoxsulam, total residues were 98.1% of the applied radioactivity (97.1% from the soil columns, 1.0% from the leachate). The distribution of total radioactivity was 35.7%, 15.8%, 15.1%, 9.9%, 14.7%, and 5.9% for the 0-5 cm, 5-10 cm, 10-15 cm, 15-20 cm, 20-25 cm, and 25-30 cm soil column depths, respectively.

In unaged Ottobiano sandy loam soil treated with [triazolopyrimidine-2-^{14}C]penoxsulam, total residues were 97.7% of the applied radioactivity (97.5% from the soil columns, 0.2% from the leachate). The distribution of total radioactivity was 20.9%, 28.3%, 24.5%, 15.4%, 6.9%, and 1.5% for the 0-5 cm, 5-10 cm, 10-15 cm, 15-20 cm, 20-25 cm, and 25-30 cm soil column depths, respectively.

Total extractable [^{14}C]residues, [triazolopyrimidine-2-^{14}C]penoxsulam, transformation products, nonextractable [^{14}C]residues, volatile [^{14}C]organic compounds, $^{14}CO_2$, and bound residues were not measured individually in either soil column of the saturated soil leaching experiment.

PRELIMINARY STUDY

The most efficient solvent for extracting [^{14}C]penoxsulam from the definitive study soils was determined in a preliminary study (p.10). [^{14}C]Penoxsulam (*ca* 0.2 µg) was applied to 2 g of three of the four definitive study soils (Ogori sandy silt loam, Nagaoka clay loam, and Arkansas silt loam; Table 1, pp.22). Five samples of each soil were prepared, one per extraction solvent (p.10). The five extraction solvents were aqueous 0.01M $CaCl_2$, methanol, acetone:ethyl acetate: 1 M HCl (85:10:5, v:v:v), acetonitrile:water (9:1, v:v), and acetonitrile:water (9:1, v:v) with 2% acetic acid (p.10). After two days at room temperature, the [^{14}C]penoxsulam-treated soils were extracted with 5 mL of each of the extraction solvents. The extracts were analyzed by LSC for radioactivity (p.10). The results of the LSC analysis showed that acetonitrile:water (9:1, v:v) extracted the most radioactivity from the soil samples (p.10; Table 2a, p.23). As a further investigation, two 100- Nagaoka soil samples were treated with a larger amount of [^{14}C]penoxsulam (*ca.* 4.9 µg; p.10). One soil sample was extracted (3 x 100 mL) with acetonitrile:water (9:1, v:v), and the other was extracted (3 x 100 mL) with acetone:water (9:1, v:v; p.10). The LSC analysis of the combined extracts from both soils showed that acetonitrile:water (9:1, v:v) and acetone:water (9:1, v:v) were equivalent in extraction efficiency (p.10; Table 2b, p.23). The acetonitrile:water (9:1, v:v) was chosen as the extraction solvent for the definitive study (p.13).

MATERIALS AND METHODS (DEFINITIVE STUDY)

The mobility of [^{14}C]penoxsulam in unaged soils was investigated using [triazolopyrimidine-2-^{14}C] 3-(2,2-difluoroethoxy)-*N*-(5,8-dimethoxy[1,2,4]triazolo[1,5-c]pyrimidin-2-yl)-α,α,α-trifluorotoluene-2-sulfonamide (penoxsulam; XR-638; radiochemical purity 99%; specific activity 949.1 Mbq/mmol, 25.26 mCi/mmol, 117900 dpm/µg; Letcombe Ref. No.: INV 98/05; Inventory No.: INV1352; p.9).

Prior to study initiation, sandy silt loam, clay loam, silt loam, and sandy loam soils were air-dried, sieved (2 mm), and stored in the dark at approximately 4°C (p.10). The four test soils were collected from rice-growing areas around the world (p.10). Characteristics of the test soils were as follows:

Property	Ogori	Nagaoka	Arkansas	Ottobiano
Source	Japan	Japan	USA	Italy
Taxonomic class	Sandy silt loam	Clay loam	Silt loam	Sandy loam
Soil texture [1]				
% sand	33.3	24.7	4.7	67.2
% silt	50.4	51.7	78.2	25.0
% clay	16.3	23.6	17.2	7.8
pH (water)	6.5	5.8	5.7	6.4
pH (0.01M $CaCl_2$)	5.7	4.9	4.8	5.5
Organic carbon (%)	1.8	2.1	1.2	1.0
Organic matter (%)[2]	3.10	3.61	2.06	1.72
CEC (meq/100 g soil)	18.5	24.0	12.5	7.4
Microbial biomass (mg C/100 g soil)	Not reported.			
Bulk density (g/mL)	Not reported.			
Percent water holding capacity at 1/3 bar (g/100 g soil dry wt)	Not reported.			
Maximum water capacity (g water/100 g soil)	Not reported.			
Field capacity (g water/100 g soil)	Not reported.			

Data were obtained from Table 1, p.22 of the study report. Soil characterization was performed by the Soil Survey and Land Research Centre, Silsoe, UK (p.10).

[1] Soil textural classes could not be confirmed because the particle size distribution used in soil characterization was not according to the USDA Soil Textural Classification System.

[2] Calculated; organic matter = organic carbon x 1.72.

Two types of unaged soil leaching experiments were performed in the study, a free-draining experiment and a saturated (flooded) experiment (p.8). All four soils were used in the free-draining experiment. Only the Nagaoka and Ottobiano soils were used in the saturated experiment (p.11). In the free-draining experiment, the flow of aqueous solution was controlled at the top of the column and uncontrolled at the bottom (p.8). In the saturated experiment, the flow of aqueous solution was restricted at the bottom so as to maintain a 5-cm layer of the solvent on the top of the column (pp.8, 11).

Prior to treatment, seven cylindrical glass columns (5 cm height x 5 cm i.d.) with round glass joints were connected together (35-cm total height; p.11). The joints were water-sealed with silicon grease (p.11). The bottom section contained a glass fibre filter paper (to contain fine

particles) on top of a perforated ceramic plate (to allow flow of the solvent; p.11). The aqueous solution (0.01M $CaCl_2$) flow was controlled by a funnel at the bottom of the column (below the ceramic plate; p.11). An illustration of the assembled soil column is provided in Figure 1 of the study report (p.27). After the assembled columns were evenly packed with air-dried soil to a height of 30 cm, the weights of the filled columns were recorded (p.11). The four soil columns of the free-draining experiment were saturated with the 0.01M $CaCl_2$ solution, drained, and maintained in a free-draining condition before application of test compound (p.11). The two soil columns of the saturated experiment were saturated with the 0.01M $CaCl_2$ solution, drained, and re-saturated so that there was 5 cm of solution in the top of the soil columns before application of test compound (p.11). The amounts of aqueous solution required to saturated all soil columns were recorded (recorded as weight; assumed density of 1 g/mL; p.11; Table 3, p.24).

The [^{14}C]penoxsulam application solution was prepared by diluting a 178-µL aliquot of a [^{14}C]penoxsulam stock solution to 5 mL with distilled water (14.54 µg/mL final volume; 3.6% acetone by volume; p.11). Then, 962-µL aliquots of the [^{14}C]penoxsulam application solution were applied dropwise to the soil columns of the free-draining (on the soil surface) and saturated experiments (on the aqueous solvent surface; p.11). The application rate was 14.0 µg of [^{14}C]penoxsulam (0.74 µCi), which was equivalent to 71 g/ha based on the 19.2-cm^2 surface area of the soil column (approximately twice the maximum field application rate of 35 g/ha; pp.11-12).

After application of the [^{14}C]penoxsulam test solution, all of the soil columns were wrapped with aluminum foil to exclude light. However, only the soil columns of the free-draining experiment were protected by placing a piece of filter paper on top of the soil (a 5-cm layer of aqueous solution was on top the soil of the saturated experiment; p.12). A total of 393-422 mL of the 0.01M $CaCl_2$ aqueous solvent (20.0-21.5 cm depth) was applied to the four soil columns of the free-draining experiment at a constant flow rate over 48 hours (flow maintained by a peristaltic pump; p.12; Table 3, p.24). The 0.01M $CaCl_2$ aqueous solution was also applied to the two soil columns of the saturated experiment at a constant flow rate over 48 hours, but no volume/weight of the solvent was recorded (p.12; Table 3, p.24). An illustration of the assembled soil column with the peristaltic pump is provided in Figure 1 of the study report (p.27). During leaching of the columns, leachates were collected in four (0-5 cm, 5-10 cm, 10-15 cm, and 15-20 cm) and two (0-10 cm and 10-20 cm) approximately equal-volume fractions for the free-draining soil experiment and saturated experiment, respectively (p.12). After the 48-hour leaching period, the remaining 0.01M $CaCl_2$ aqueous solution was drained from the column and added to the final fraction (p.12). The volumes of 0.01M $CaCl_2$ aqueous solution were measured for the entire column and the individual fractions (p.12; Table 3, p.24).

Following leaching, all of the soil columns were disassembled. The weights of the soil columns post-leaching were determined for each entire column and the six individual sections (0-5 cm, 5-10 cm, 10-15 cm, 15-20 cm, 20-25 cm, and 25-30 cm; Table 3, p.24). The soil in each section was removed, combusted, and analyzed for total radioactivity using LSC (a preliminary analysis; p.13). The glass column segments were washed with acetone:water (1:1, v:v), which was then

measured for radioactivity using LSC (p.13). No further analysis of the saturated experiment soil columns was performed (p.13).

The soil sections of the free-draining experiment which contained >2% of the applied radioactivity in the preliminary radioactivity analysis were extracted and further analyzed by HPLC (p.13). To isolate and quantify [^{14}C]residues, each soil section of the four soil columns was extracted three times by shaking (method not specified) with 100 mL of acetonitrile:water (9:1) for one hour (approximately 125-166 g of soil per section; p.13; Table 3, p.24). After centrifugation, the supernatants were combined, analyzed for total radioactivity using LSC, and evaporated to a 50 mL aqueous solution (p.13). The aqueous concentrate was passed through a pre-conditioned Isolute C18 solid phase extraction (SPE) column. The SPE column was eluted with 2 x 4 mL of acetonitrile (p.13). The acetonitrile solution was evaporated to dryness, re-dissolved in 1.0 mL of acetonitrile:water (1:1, v:v), and aliquots were analyzed using LSC and reverse-phase HPLC (p.13). The operating conditions for HPLC analyses were as follows (pp.14-15):

Column	Prodigy ODS-3 (260 x 4.6 mm; 5 µm particle size; Phenomenex, Macclesfield, Cheshire, UK).
Detectors	Varian 9050 UV detector (260 nm). Packard Radiomatic 515TR radioactivity detector fitted with a SolarScint™ solid phase flow cell (200 µL).
Mobile phase	(A) Water with 2% acetic acid; (B) Acetonitrile with 2% acetic acid.
Mobile phase gradient	75:25 to 0:100 (v:v; A:B).
Flow rate	1 mL/minute.
Sample aliquots	Soil extracts- 28-200 µL. Leachate extracts- 250 µL.
Column recovery	96.7% mean (range = 92.4-99.4%).

Although not stated in the study report, [^{14}C]penoxsulam in the soil and leachate of the soil columns was apparently identified by comparison to the retention time of [^{14}C]penoxsulam test substance (Figures 3-6, pp.29-32). No transformation products were reported at >4% of the applied radioactivity (Table 5, p.26). The retention times of the [^{14}C]compounds were as follows (Table 5, p.26):

Compound Code- Number	Retention Time (min)
[^{14}C]XR-638	17
[^{14}C]minor degradation product	15

Limits of detection and quantification for HPLC analyses of penoxsulam and its transformation products were not reported (pp.15-16). The extracted soils were air-dried, finely ground via mortar and pestle, combusted, and analyzed for total radioactivity using LSC (p.13). Leachates were analyzed for total radioactivity using LSC, and only one leachate fraction (15-20 cm fraction of the Ottobiano soil column in the free-draining experiment) contained >2% of the applied radioactivity (p.12; Table 3, p.24). The aqueous leachate (*ca.* 100 mL) was passed through a pre-conditioned Isolute C18 solid phase extraction (SPE) column; the SPE column was eluted with 2 x 4 mL of acetonitrile (p.13). The acetonitrile solution was evaporated to dryness, re-dissolved in 1.0 mL of acetonitrile:water (1:1, v:v), and aliquots were analyzed by LSC and reverse-phase HPLC (p.13).

Radioactivity analyses on all liquid samples in the study were performed by mixing triplicate samples with UltimaGold™ XR scintillation cocktail and counting for five minutes on a Tri-Carb 2700TR liquid scintillation analyzer (Canberra Packard, Pangbourne, UK; p.14). Radioactivity analyses of soil samples (*ca.* 0.3-0.6 g) were performed by absorbing the $^{14}CO_2$ combustion product into a mixture of Carbo-Sorb™ carbon dioxide absorbing solution and Permafluor™ scintillation cocktails and counting for five minutes on the liquid scintillation analyzer (p.14). The background values for the analysis of the liquid samples and soil samples were 23 and 41 dpm, respectively (p.15). All combustion of soil samples was performed with oxygen in a Harvey OX500 Biological Sample Oxidizer (Laboratory Impex Ltd., Hampton, Middlesex, UK; p.14).

RESULTS/DISCUSSION:

Free-draining experiment: In unaged Ogori sandy silt loam soil treated with [triazolopyrimidine-2-^{14}C]penoxsulam, total residues were 100.2% of the applied radioactivity (100.2% from the soil columns, 0% from the leachate; Table 4, p.25). The distribution of total radioactivity was 40.2%, 25.5%, 20.0%, 13.0%, 1.5%, and below detection in the 0-5 cm, 5-10 cm, 10-15 cm, 15-20 cm, 20-25 cm, and 25-30 cm soil column depths, respectively (Table 4, p.25). Total extractable [^{14}C]residues were 39.8% of the applied in the 0-5 cm soil column depth, and were 25.3%, 19.8%, 12.8%, 1.4%, and not detected in the 5-10 cm, 10-15 cm, 15-20 cm, 20-25 cm, and 25-30 cm soil column depths, respectively (Table 5, p.26). [Triazolopyrimidine-2-^{14}C]penoxsulam accounted for 36.1% of the applied in the 0-5 cm soil column depth, and were 23.1%, 18.2%, 12.4%, 1.3%, and not detected in the 5-10 cm, 10-15 cm, 15-20 cm, 20-25 cm, and 25-30 cm soil column depths, respectively (Table 5, p.26). A minor unidentified transformation product (15-minute retention time) was not detected in the 0-5 cm, 15-20 cm, 20-25 cm, and 25-30 cm soil column depths, but accounted for 0.5% and 0.6% of the applied in the 5-10 cm and 10-15 cm soil column depths, respectively (Table 5, p.26). Nonextractable [^{14}C]residues accounted for 3.7% of the applied in the 0-5 cm soil column depth, and were 1.7%, 1.0%, 0.4%, 0.1%, and not detected in the 5-10 cm, 10-15 cm, 15-20 cm, 20-25 cm, and 25-30 cm soil column depths, respectively (Table 5, p.26). Volatile [^{14}C]organic compounds, $^{14}CO_2$, and bound residues were not measured individually.

In unaged Nagaoka clay loam soil treated with [triazolopyrimidine-2-^{14}C]penoxsulam, total residues were 102.9% of the applied radioactivity (102.9% from the soil columns, 0% from the leachate; Table 4, p.25). The distribution of total radioactivity was 102.9% for the 0-5 cm and not detected in all other soil column depths (Table 4, p.25). Total extractable [^{14}C]residues were 101.9% of the applied in the 0-5 cm soil column depth, and were not detected in all other soil column depths (Table 5, p.26). [Triazolopyrimidine-2-^{14}C]penoxsulam accounted for 95.8% of the applied in the 0-5 cm soil column depth, and was not detected in all other soil column depths (Table 5, p.26). The minor transformation product (15-minute retention time) was not detected in any soil column depths (Table 5, p.26). Nonextractable [^{14}C]residues accounted for 6.1% of the applied in the 0-5 cm soil column depth, and were not detected in all other soil column depths (Table 5, p.26). Volatile [^{14}C]organic compounds, $^{14}CO_2$, and bound residues were not measured individually.

In unaged Arkansas silt loam soil treated with [triazolopyrimidine-2-^{14}C]penoxsulam, total residues were 98.2% of the applied radioactivity (98.2% from the soil columns, 0% from the leachate; Table 4, p.25). The distribution of total radioactivity was 86.1% in the 0-5 cm soil column depth, 12.1% in the 5-10 cm soil column depth, and not detected in any other soil column depths (Table 4, p.25). Total extractable [^{14}C]residues were 85.6% of the applied in the 0-5 cm soil column depth, 12% in the 5-10 cm soil column depth, and were not detected in any other soil column depth (Table 5, p.26). [Triazolopyrimidine-2-^{14}C]penoxsulam accounted for 80.6% of the applied in the 0-5 cm soil column depth, 10.7% in the 5-10 cm soil column depth, and was not detected in any other soil column depth (Table 5, p.26). The minor transformation product (15-minute retention time) accounted for 1.9% of the applied in the 0-5 cm soil column depth, 1.0% in the 5-10 cm, and was not detected in any other soil column depth (Table 5, p.26). Nonextractable [^{14}C]residues accounted for 3.1% of the applied in the 0-5 cm soil column depth, 0.3% in the 5-10 cm soil column depth, and were not detected in any other soil column depth (Table 5, p.26). Volatile [^{14}C]organic compounds, $^{14}CO_2$, and bound residues were not measured individually.

In unaged Ottobiano sandy loam soil treated with [triazolopyrimidine-2-^{14}C]penoxsulam, total residues were 98.9% of the applied radioactivity (95.8% from the soil columns, 3.1% from the leachate; Table 4, p.25). The distribution of total radioactivity was 9.7%, 19.9%, 22.6%, 18.6%, 12.3%, and 12.7% in the 0-5 cm, 5-10 cm, 10-15 cm, 15-20 cm, 20-25 cm, and 25-30 cm soil column depths, respectively (Table 4, p.25). Total extractable [^{14}C]residues were 9.6% of the applied for the 0-5 cm soil column depth, and 19.7%, 22.5%, 18.5%, 12.2%, and 12.6% for the 5-10 cm, 10-15 cm, 15-20 cm, 20-25 cm, and 25-30 cm soil column depths, respectively (Table 5, p.26). [Triazolopyrimidine-2-^{14}C]penoxsulam accounted for 8.5% of the applied in the 0-5 cm soil column depth, and 18.9%, 20.9%, 16.8%, 11.4%, and 11.7% in the 5-10 cm, 10-15 cm, 15-20 cm, 20-25 cm, and 25-30 cm soil column depths, respectively (Table 5, p.26). The minor transformation product (15-minute retention time) accounted for 0.2% of the applied in the 0-5 cm soil column depth, and 0.3%, 0.9%, 0.9%, 0.6%, and 0.5% in the 5-10 cm, 10-15 cm, 15-20 cm, 20-25 cm, and 25-30 cm soil column depths, respectively (Table 5, p.26). Nonextractable [^{14}C]residues accounted for 0.9% of the applied in the 0-5 cm soil column depth, and 0.5%,

0.7%, 0.8%, 0.2%, and 0.4% in the 5-10 cm, 10-15 cm, 15-20 cm, 20-25 cm, and 25-30 cm soil column depths, respectively (Table 5, p.26). Volatile [^{14}C]organic compounds, $^{14}CO_2$, and bound residues were not measured individually.

Saturated experiment: In unaged Nagaoka clay loam soil treated with [triazolopyrimidine-2-^{14}C]penoxsulam, total residues were 98.1% of the applied radioactivity (97.1% from the soil columns, 1.0% from the leachate; Table 4, p.25). The distribution of total radioactivity was 35.7%, 15.8%, 15.1%, 9.9%, 14.7%, and 5.9% in the 0-5 cm, 5-10 cm, 10-15 cm, 15-20 cm, 20-25 cm, and 25-30 cm soil column depths, respectively (Table 4, p.25).

In unaged Ottobiano sandy loam soil treated with [triazolopyrimidine-2-^{14}C]penoxsulam, total residues were 97.7% of the applied radioactivity (97.5% from the soil columns, and 0.2% from the leachate; Table 4, p.25). The distribution of total radioactivity was 20.9%, 28.3%, 24.5%, 15.4%, 6.9%, and 1.5% for the 0-5 cm, 5-10 cm, 10-15 cm, 15-20 cm, 20-25 cm, and 25-30 cm soil column depths, respectively (Table 4, p.25).

Total extractable [^{14}C]residues, [triazolopyrimidine-2-^{14}C]penoxsulam, transformation products, nonextractable [^{14}C]residues, volatile [^{14}C]organic compounds, $^{14}CO_2$, and bound residues were not measured individually in either soil column of the saturated soil leaching experiment.

DEFICIENCIES/DEVIATIONS

1. The soil columns were leached with approximately 20 cm of 0.01M $CaCl_2$ aqueous solvent, rather than 50.8 cm, as required by Subdivision N guidelines.

2. Three of the four test soils were foreign in origin (Table 1, p.22). The three foreign soils were not completely characterized according to the USDA Soil Textural Classification System. The reviewer notes that the textural classes reported in the study were based on the UK Soil Textural Classification System. Soil particle size distributions were as follows: sand, 2 mm - 63 µm; silt, 63 µm - 2 µm; and clay, <2 µm (Table 1, p.22).

3. No soil in the study contained an organic matter content less than 1%, as required by Subdivision N guidelines.

4. The chemical structure of penoxsulam has two rings, a phenyl ring and a pyrimidinyl ring. The test substance was only radiolabeled on the pyrimidinyl ring. Subdivision N guidelines specify that experiments should be done with each respective ring. The reviewer notes that additional experiments with [^{14}C-phenyl]penoxsulam are unnecessary, due to the experimental stability of penoxsulam in the unaged soil column and the suitable mass balance in this study.

5. The application rate of [^{14}C]penoxsulam in this study was approximately twice the maximum field application rate of 35 g/ha (pp.11-12). Subdivision N guidelines specify that the application rate of the test substance is equivalent to the highest recommended single application rate.

6. It was not stated in the study if one of the test soils was used in the aerobic metabolism study, as required by Subdivision N guidelines.

7. Volatiles were not collected during the leaching portion of the study. During combustion of the wet soils and extracted soils, the study authors reported a $^{14}CO_2$ trapping scintillation mixture (Carbo-Sorb™ carbon dioxide absorbing solution and Permafluor™ scintillation cocktails; p.14). The study authors did not report or determine the percentage/amount of $^{14}CO_2$ present in the nonextractable residues.

8. Microbial viability of the test soil was not determined.

9. For each soil type and label, only one soil column was prepared for each soil type/test substance treatment combination. Subdivision N guidelines specify that at least two soil columns be prepared for each soil type/test substance treatment combination.

10. The study authors reported that the test soils were typical rice-growing soils (high clay content; 16-24%). However, the Ottobiano sandy loam soil may represent a "worse-case" for mobility of penoxsulam based on its low clay content (7.8% clay content; p.17). Additionally, the study authors noted that the low pH values of the test soils is expected to retard leaching since penoxsulam was acidic in nature (p.17).

11. The limits of detection and quantification used for LSC and HPLC analyses were not reported.

12. Representative HPLC chromatograms of soil (from different soil layers) and leachate from the four test soils treated with radiolabeled test substance showed good separation of peaks (Figures 4-6, pp.30-32).

13. 3-(2,2-Difluoroethoxy)-*N*-(5,8-dimethoxy[1,2,4]triazolo[1,5-c]pyrimidin-2-yl)-α,α,α-trifluorotoluene-2-sulfonamide was identified as the IUPAC name of penoxsulam by the Compendium of Pesticide Common Names (http://www.hclrss.demon.co.uk/penoxsulam.html). 3-(2,2-difluoroethoxy)-*N*-(5,8-dimethoxy[1,2,4]triazolo[1,5-c]pyrimidin-2-yl)-6-(trifluoromethyl)benzenesulfonamide was identified as the CAS name of penoxsulam by the Compendium of Pesticide Common Names and the USEPA/OPP Chemical Database (http://www.cdpr.ca.gov/cgi-bin/epa/chemidetriris.pl?pccode=119031). CAS Reg. No. 219714-96-2 for penoxsulam was obtained from the USEPA/OPP Chemical Database.

Attachment 1

Excel Spreadsheets

Chemical Name: Penoxsulam
PC Code: 119031
MRID: 45834802
Guideline No.: 163-1
Data obtained from Table 5, p. 26 of the study report.

Ogori

Compound	Total	0-5cm	5-10cm	10-15cm	15-20cm	20-25cm	25-30cm	Leachate
XR-638	91.1	36.1	23.1	18.2	12.4	1.3		
Deg. Prod	1.1		0.5	0.6				
NER	6.9	3.7	1.7	1	0.4	0.1		
Total	99.1	39.8	25.3	19.8	12.8	1.4		

Nagaoka

Compound	Total	0-5cm	5-10cm	10-15cm	15-20cm	20-25cm	25-30cm	Leachate
XR-638	95.8	95.8						
Deg. Prod	0							
NER	6.1	6.1						
Total	101.9	101.9						

Arkansas

Compound	Total	0-5cm	5-10cm	10-15cm	15-20cm	20-25cm	25-30cm	Leachate
XR-638	91.3	80.6	10.7					
Deg. Prod	2.9	1.9	1					
NER	3.4	3.1	0.3					
Total	97.6	85.6	12					

Ottobiano

Compound	Total	0-5cm	5-10cm	10-15cm	15-20cm	20-25cm	25-30cm	Leachate
XR-638	90.9	8.5	18.9	20.9	16.8	11.4	11.7	3
Deg. Prod	3.4	0.2	0.3	0.9	0.9	0.6	0.5	0.1
NER	3.5	0.9	0.5	0.7	0.8	0.2	0.4	
Total	97.8	9.6	19.7	22.5	18.5	12.2	12.6	3.1

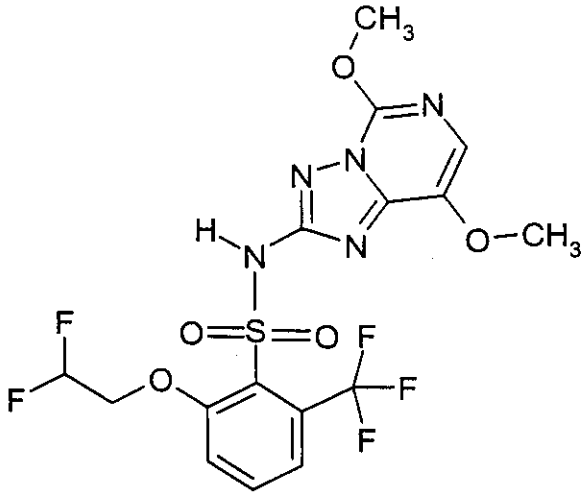
Attachment 2

Structures of Parent and Transformation Products

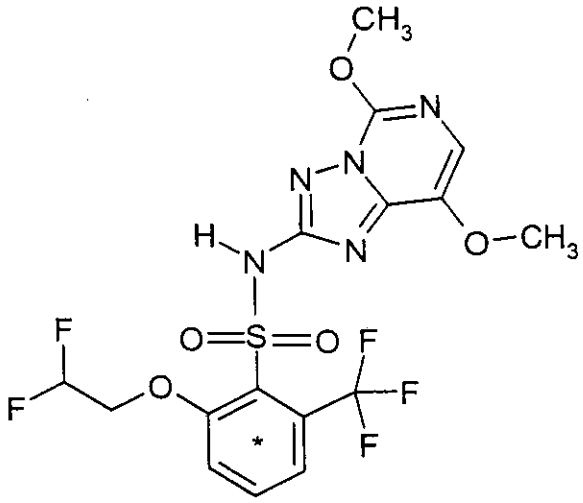
Penoxsulam

IUPAC name:	3-(2,2-Difluoroethoxy)-N-(5,8-dimethoxy[1,2,4]triazolo[1,5-c]pyrimidin-2-yl)-□,□,□-trifluorotoluene-2-sulfonamide
CAS name:	2-(2,2-Difluoroethoxy)-N-(5,8-dimethoxy[1,2,4]triazolo[1,5-c]pyrimidin-2-yl)-6-(trifluoromethyl)benzenesulfonamide
CAS No:	219714-96-2

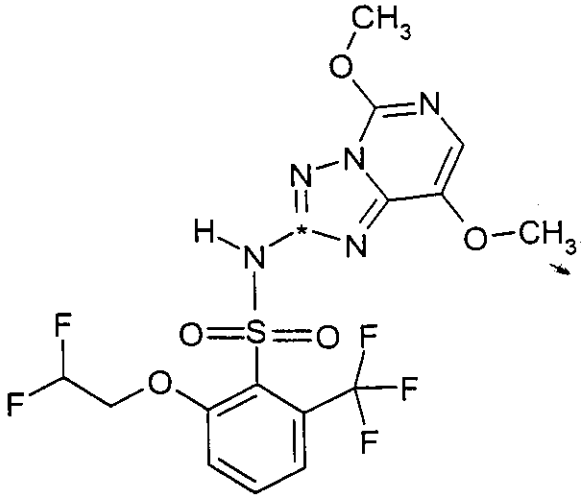
Unlabeled



[Phenyl-U-^{14}C] label



[Triazolopyrimidine-2-^{14}C] label



* Position of the radiolabel.

5-OH-XDE-638

IUPAC name: 6-(2,2-Difluoroethoxy)-N-(5,6-dihydro-8-methoxy-5-oxo-s-triazolo[1,5-c]pyrimidin-2-yl)-□,□,□-trifluoro-o-toluenesulfonamide
CAS name: (2,2-Difluoroethoxy)-N-(5,6-dihydro-8-methoxy-5-oxo[1,2,4]triazolo[1,5-c]pyrimidin-2-yl)-6-(trifluoromethyl)benzenesulfonamide
CAS No: NA

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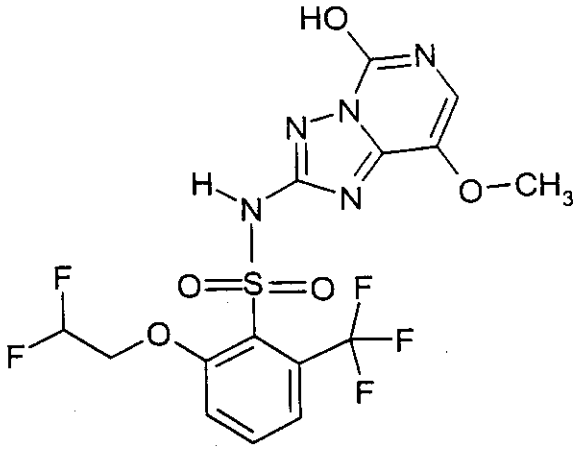
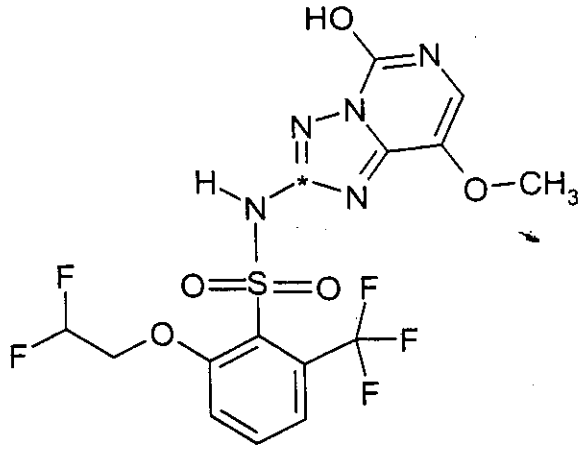


[Triazolopyrimidine-2-^{14}C] label



* Position of the radiolabel.

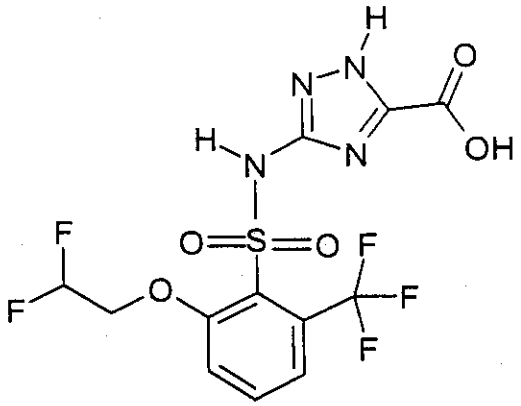
BSTCA

IUPAC name: 3-[6-(2,2-Difluoroethoxy)-□,□,□-(trifluoro-o-toluenesulfonamido]-s-triazole-5-carboxylic acid

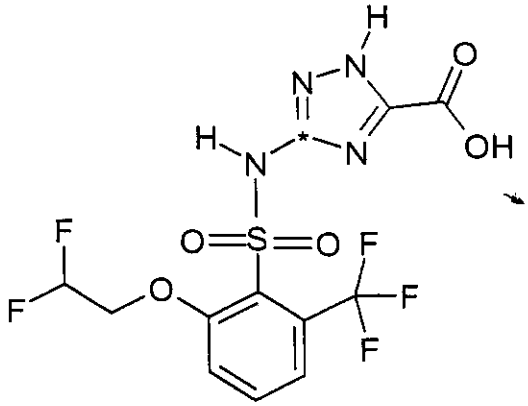
CAS name: 3-[[[2-(2,2-Difluoroethoxy)-6-(trifluoromethyl)phenyl]-sulfonyl]amino]-1H-1,2,4-triazole-5-carboxylic acid

CAS No: NA

Unlabeled



[Triazolopyrimidine-2-^{14}C] label

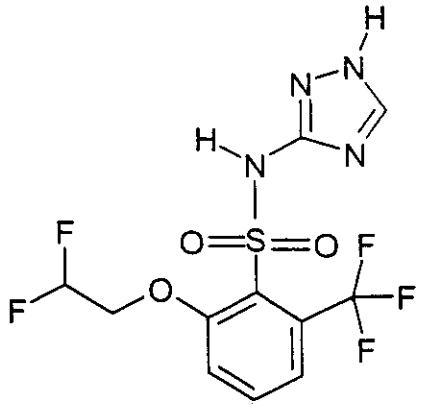


* Position of the radiolabel.

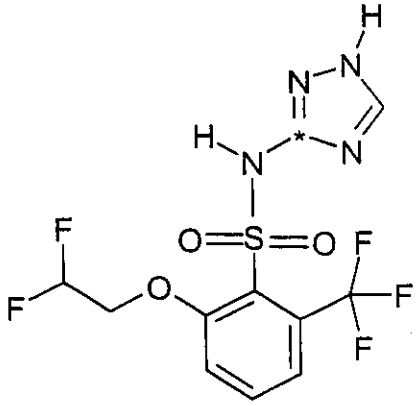
BST

IUPAC name: 6-(2,2-Difluoroethoxy)-□,□,□-trifluoro-N-s-triazol-3-yl-o-toluenesulfonamide
CAS name: 2-(2,2-Difluoroethoxy)-N-1H-1,2,4-triazole-3-yl-6-(trifluoromethyl)benzenesulfonamide
CAS No: NA

Unlabeled



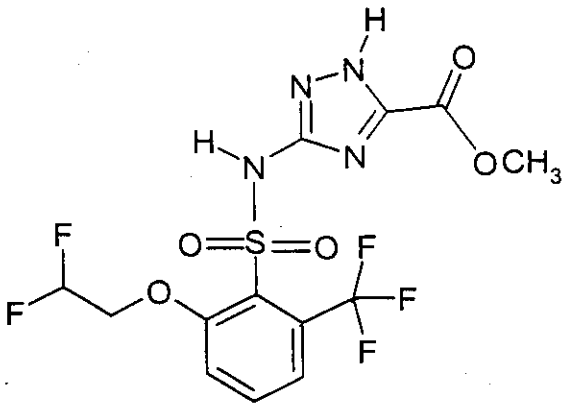
[Triazolopyrimidine-2-^{14}C] label



* Position of the radiolabel.

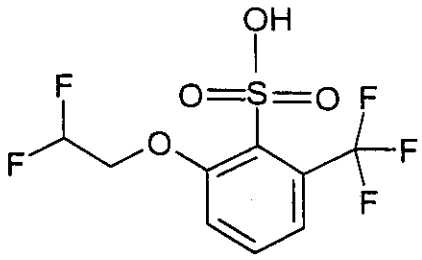
BSTCA-methyl

IUPAC name: Methyl 3-[6-(2,2-difluoroethoxy)-□,□,□-trifluoro-o-toluenesulfonamido]-s-triazole-5-carboxylate
CAS name: Methyl 3-[[[2-(2,2-difluoroethoxy)-6-(trifluoromethyl)phenyl]sulfonyl]amino]-1H-1,2,4-triazole-5-carboxylate
CAS No: NA



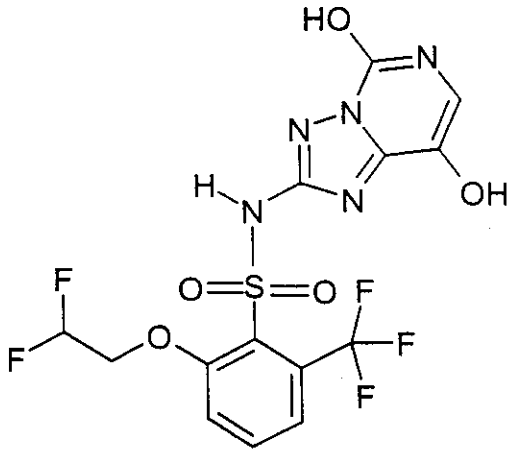
BSA

IUPAC name: 6-(2,2-Difluoroethoxy)-□,□,□-trifluoro-o-toluenesulfonic acid
CAS name: 2-(2,2-Difluoroethoxy)-6-(trifluoromethyl)benzenesulfonic acid
CAS No: NA



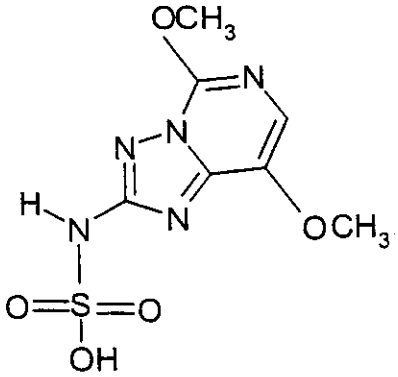
5,8-diOH

IUPAC name: NA
CAS name: 2-(2,2-Difluoroethoxy)-6-trifluoromethyl-N-(5,8-dihydroxy-[1,2,4]triazolo[1,5-c]pyrimidin-2-yl)benzenesulfonamide
CAS No: NA



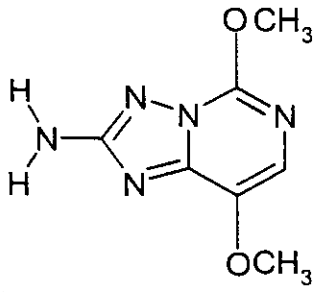
TPSA

IUPAC name: NA
CAS name: 5,8-Dimethoxy[1,2,4]triazolo-[1,5-c]pyrimidin-2-yl-sulfamic acid
CAS No: NA



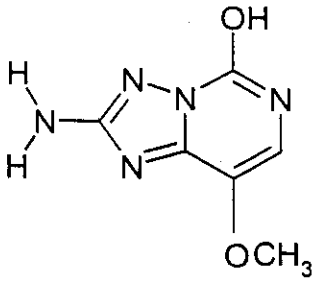
2-Amino TP

IUPAC name: 2-Amino-5,8-dimethoxy-s-triazolo[1,5-c]pyrimidine
CAS name: 5,8-Dimethoxy[1,2,4]triazolo[1,5-c]pyrimidin-2-amine
CAS No: NA



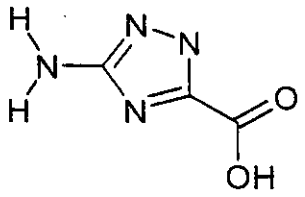
5-OH, 2-Amino TP

IUPAC name: NA
CAS name: 8-Methoxy[1,2,4]triazolo-[1,5-c]pyrimidin-5-ol-2-amine
CAS No: NA



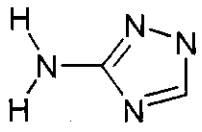
2-Amino TCA

IUPAC name: NA
CAS name: 2-Amino-1,3,4-triazole-5-carboxylic acid
CAS No: NA



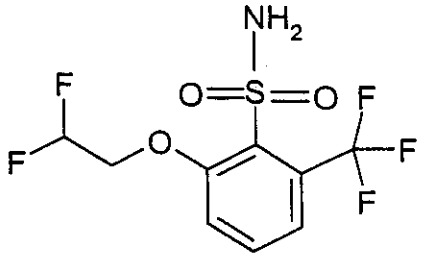
2-Amino-1,3,4-triazole

IUPAC name: NA
CAS name: 2-Amino-1,3,4-triazole
CAS No: NA



Sulfonamide

IUPAC name: 2-(2,2-Difluoroethoxy)-6-(trifluoromethyl)-benzenesulfonamide
CAS name: 2-(2,2-Difluoroethoxy)-6-(trifluoromethyl)-benzenesulfonamide
CAS No: NA



Sulfonylformamidine

IUPAC name: 2-(2,2-Difluoroethoxy)-N-[(E)iminomethyl-6-(trifluoromethyl)benzenesulfonamide
CAS name: 2-(2,2-Difluoroethoxy)-N-(iminomethyl-6-(trifluoromethyl)-benzenesulfonamide
CAS No: NA