

Report	IIA 4.4/04 (OECD) / CA 4.1.2/08 (EU)	
Authors (year):	Robaugh, D. (2013)	
Title:	Independent laboratory validation of ISK method H-855 for the determination of SL-573 and MT-2153 in soil	
PMRA No.	2522684	
MRID	49580118	PC: 573101
Laboratory report no. and date:	2460, 8 April 2013	
Owner:	Ishihara Sangyo Kaisha, Ltd., Japan	
Testing facility and address:	Pyxant Labs Inc, 4720 Forge Road, Suite 108, Colorado Springs, CO 80907, USA	
Dates of experimental work:	October 2012	
Guideline(s) followed:	US EPA OPPTS 850.7100 SANCO/825/00 rev. 8.1 (2010)	
Deviations from guidelines:	None	
GLP	Yes	US EPA

Study Classification:

EPA: Acceptable

Summary written by: Katherine Keppel-Jones, PMRA, on November 25, 2015

Peer reviewed by: Kim Davis, PMRA, on January 26, 2016

Secondary review by: Marianne Mannix, EPA
Rochelle Bohaty EPA

Marianne A. Mannix
Rochelle Bohaty

Digitally signed by MARIANNE MANNIX
Date: 2017.10.04 17:09:29 -04'00'

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Executive summary

The analytical method H-855 for the determination of SL-573 and MT-2153 in soil (Furuo, R., 2012, "Validation of the residue analytical method for the determination of SL-573 and its metabolite MT-2153 in soil") was successfully validated, in an independent laboratory, for the determination of SL-573 and MT-2153 in untreated soil from North Dakota, USA. Several minor modifications were made to the original method, none of which were considered critical to the success of the method.

The results demonstrated similar accuracy and precision to the original methodology and confirmed the applicability of the method for the determination of SL-573 and MT-2153 in soil.

Analyte / reference substance 1

ISO common name: Tolpyralate
Code no.: SL-573
CAS no.: 1101132-67-5
Lot/batch no.: 20110128
Purity: 99.81%

Analyte / reference substance 2

Code no.: MT-2153
CAS no.: Not available
Lot/batch no.: 20120125
Purity: 99.8%

Test matrix

Untreated soil from North Dakota, USA

Principle of the method

Soil samples were extracted using methanol/water (80/20, v/v) with ammonium formate (0.1M), citric acid (0.05M) and hydrochloric acid (0.5%, v/v) and a reciprocal shaker. Extracts were cleaned by solid phase extraction and diluted with acetonitrile/water (50/50, v/v). Analysis was by LC-MS/MS.

The trial comprised one batch of samples consisting of solvent blank, reagent blank, two unfortified controls, five control samples fortified at the LOQ (0.001 µg/g) and five control samples fortified at 100 × LOQ (i.e. 0.10 µg/g).

Specificity

Interferences at the retention times of SL-573 and MT-2153 were not observed.

Linearity

Correlation coefficients (r) for each analyte were ≥ 0.995 .

Accuracy

Mean recoveries for both analytes at each fortification level were within the range 70 - 110%. Individual recovery values for each fortified sample were within the range 70 - 120%, with one exception (one MT-2153 sample fortified at the LOQ: 137%).

Table 5. ILV validation data for tolpyralate (SL-573) and MT-2153 in soil

Analyte	Fortified at LOQ (0.001 µg/g)		Fortified at 100 × LOQ (0.10 µg/g)	
	Mean ± SD (RSD)	Range (n = 5)	Mean ± SD (RSD)	Range (n = 5)
SL-573	79% ± 4.0% (5%)	72 - 83	81% ± 3.0% (4%)	77 - 85
MT-2153	107% ± 18% (17%)	93 - 137	85% ± 1.3% (2%)	84 - 87

SD Standard deviation

RSD Relative standard deviation

Precision (repeatability)

Results obtained were within the guideline requirements (relative standard deviation $\leq 20\%$).

Limit of quantification (LOQ)

The limit of quantification (LOQ) was 0.001 µg/g for both SL-573 and MT-2153.

Conclusion

The analytical method H-855 for the determination of SL-573 and MT-2153 in soil (Furuo, R., 2012, "Validation of the residue analytical method for the determination of SL-573 and its metabolite MT-2153 in soil") was successfully validated, in an independent laboratory, for the determination of SL-573 and MT-2153 in untreated soil from North Dakota, USA. Several minor modifications were made to the original method, none of which were considered critical to the success of the method; these were primarily amendments to the solvents used in the preparation of the standards.

The results demonstrated similar accuracy and precision to the original methodology and confirmed the applicability of the method for the determination of SL-573 and MT-2153 in soil.

Study Classification:

EPA: Acceptable

Summary written by: Katherine Keppel-Jones, PMRA, on November 25, 2015

Peer reviewed by: Kim Davis, PMRA, on January 26, 2016

Secondary review by: Marianne Mannix, Rochelle Bohaty EPA, on May 20, 2016