

Report		
Authors (year):	Schoenau, E. (2015)	
Title:	Independent Laboratory Validation of Ishihara Sangyo Kaisha (ISK) Residue Analytical Method for the Determination of MMTA in Soil	
PMRA No.	2581982	
MRID	49796201	PC: 573101
Laboratory report no. and date:	JSM0757, 22 June 2015	
Owner:	Ishihara Sangyo Kaisha, Ltd., Japan	
Testing facility and address:	Golden Pacific Laboratories, LLC (GPL) 4720 W. Jennifer Avenue, Suite 105 Fresno, California 93722	
Dates of experimental work:	15 January 2015 to 16 January 2015	
Guideline(s) followed:	US EPA Ecological Effects Test Guidelines, OCSP 850.6100	
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*

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2018.03.19  
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### Executive Summary

The method for the Residue Analytical Method for the Determination of MMTA in Soil, Miller, C. (2014) "MMTA: Validation of methodology for the determination of residues in two soil types" was successfully validated, in an independent laboratory for the determination of MMTA in a soil from North Rose, NY. No significant deviations from the protocol were made.

The results demonstrated similar accuracy and precision to the original methodology and confirmed the applicability of the method for the determination of MMTA in soil.

### Analyte / reference

Chemical name: 3-(2-ethoxyethoxy)-2 methyl-4-(methylsulfonyl) benzoic acid (MMTA)

Code no.:

CAS #: NA

Lot/batch no.: 20140226

Purity: 100%

### Test Matrix

The test matrix was a soil sample obtained from a previous non-GLP ISK Biosciences study conducted at GPL. The soil was obtained from North Rose, NY.

### Principle of the method

Soil samples were extracted twice Soil samples were extracted twice with methanol/water (80:20, v/v) containing 0.1 M ammonium formate, 0.05 M citric acid, and 0.5% v/v hydrochloric acid. The combined extract was taken through an Oasis HLB solid-phase extraction (SPE) clean-up. Analysis was by LC-MS/MS.

The study design consisted of one reagent blank sample, two control samples, five LOQ laboratory fortification samples (1 ppb) and five 10x LOQ laboratory fortification samples (10 ppb).

**Specificity**

No interferences were observed for MMTA with respect to the matrix, reagent/solvent, or labware interfaces.

**Linearity**

Correlation coefficients (r) for MMTA were  $\geq 0.995$ .

**Accuracy**

Mean and individual recovery values for MMTA at each fortification level were within the range of 70 – 110%.

**ILV Validation Data for MMTA in Soil**

Matrix	Analyte	Fortification Level (ppb)	Recovery Range (%)	Coefficient of Variation (%)	Mean Recovery (%)
Soil	MMTA	1	91.4 – 97.2	2.51	93.2
		10	90.3 – 94.3	1.91	92.0
		1	84.5 – 102	8.94	93.4
		10	88.1 – 93.3	2.26	91.7

**Precision (repeatability)**

Results obtained were within the guideline requirements ( $CV \leq 20\%$ ).

**Limit of Quantitation (LOQ)**

The limit of quantitation (LOQ) for MMTA was 1 ppb (ng/g). The limit of detection (LOD) was 0.25 ppb.

**Conclusion:**

The method for the Residue Analytical Method for the Determination of MMTA in Soil, Miller, C. (2014) “MMTA: Validation of methodology for the determination of residues in two soil types” was successfully validated, in an independent laboratory for the determination of MMTA in a soil from North Rose, NY. No significant deviations from the protocol were made.

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**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**