STUDY TITLE

Independent Laboratory Validation of XDE-659 and Metabolite in Water

DATA REQUIREMENTS

SANCO/825/00 Rev. 8.1 (2010); U.S. EPA Guidance OPPTS 860.1000, Background, OCSPP 850.6100, Independent Laboratory Validation

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STUDY COMPLETED ON

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PERFORMING LABORATORY

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STUDY SPONSOR

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INTRODUCTION

The purpose of this study was to demonstrate that "Method Validation of XDE-659 and its Metabolite in Water," AU-2019-11 (DAS ID 180503), could be performed successfully at an outside facility with no prior experience with the method (Reference 1).

Principle of the method. Residues of XDE-659 and X12485649 are determined by brief shaking and direct injection via HPLC-MS/MS.

Test conditions. For validation, the analytical set consisted of one reagent blank, two matrix controls, one control fortified at LOD (limit of detection), five replicates fortified at LOQ (limit of quantitation), and five replicates fortified at 10X LOQ. The mass transitions used for analysis are listed below.

	Quantitation (*m/z*)
XDE-659	*m/z* 513.1 → 231.1
XDE-659[1]	*m/z* 513.1 → 109.0
X12485649	*m/z* 471.1 → 231.1
X12485649[1]	*m/z* 471.1 → 109.0

[1]Denotes confirmatory ion.

Limit of Quantification (LOQ) and Limit of Detection (LOD). During the independent laboratory validation of the method, the limit of quantitation (LOQ) of XDE-659 and X12485649 was confirmed to be 0.1 µg/L (ppb) for surface, tap, and well water. The LOD for XDE-659 and X12485649 was set at 30% of the defined LOQ (0.03 ng/mL).

EXPERIMENTAL

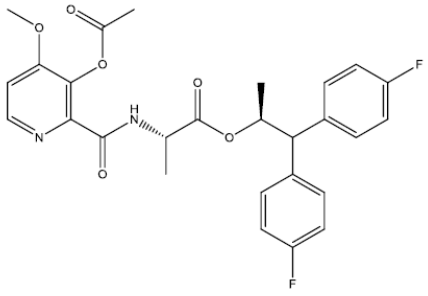
Test Systems

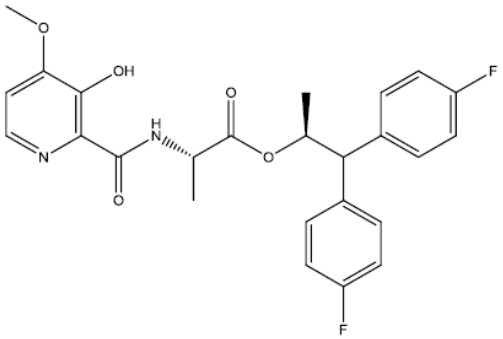
The test system considered in this study was surface (pond/lake), tap (drinking), and well (ground) water. The control samples were collected by SGS. The tap water came from the SGS GLP laboratory (44.3220743, -96.7554363) the well water from SGS Brookings field site (44.304195, -96.667977), and the surface water from a lake in Brookings, SD (44.266182, -96.761461). The test system was stored at ambient temperature at all times. All three water matrices were characterized under GLPs by Agvise Laboratories (Northwood, ND). These characterizations can be found in Appendix E.

Test and Reference Substances

The XDE-659 and X12485649 standards were stored at room temperature. The Sponsor has retained a reserve sample of these chemicals, and has documentation specifying the location of the synthesis and characterization information available at Dow AgroSciences in Indianapolis, Indiana.

The XDE-659 and X12485649 reference substances were provided by the sponsor and received on October 24, 2019. Upon receipt, the standard substances were stored in the ambient lockbox with the temperature ranging from 19 to 21 °C. All standards were stored under locked conditions. The certificates of analysis are presented in Appendix A. A detailed summary of the reference substances is presented below.

Common Name	XDE-659
Chemical Formula	$C_{27}H_{26}F_2N_2O_6$
Test Substance Structure	
CAS Number	1961312-55-9
Supplier	Dow AgroSciences
Lot / Batch #	XZ7-169400-18A
Purity	99%
Expiration	May 17, 2020

Common Name	X12485649
Chemical Formula	$C_{25}H_{24}F_2N_2O_5$
Test Substance Structure	
CAS Number	N/A
Supplier	Dow AgroSciences
Lot / Batch #	YN4-148985-060
Purity	95.0%
Expiration	May 25, 2023

Analytical Method

"Method Validation of XDE-659 and its Metabolite in Water," AU-2019-11 (DAS ID 180503) was used for the analysis of the samples.

Residues of XDE-659 and X12485649 are determined by brief shaking and direct injection via HPLC-MS/MS. The primary (quantitative) and secondary (confirmatory) transition ions monitored are presented below:

Analyte	Transition (*m/z*)		Ionization Mode	Retention Time (min)
	Primary	Secondary		
XDE-659	513.1 → 231.1	513.1 → 109.0	Positive	~4.0
X12485649	471.1 → 231.1	471.1 → 109.0		~3.6