

Independent Laboratory Validation (ILV) of an Environmental Chemistry Method for Detection of Bensulide in Water

Reports: ILV: Independent Laboratory Validation (ILV) of an Environmental Chemistry Method for Detection of Bensulide in Water

Document No.: [MRIDs 49162801]

Guideline: 850.6100 [U.S.], 8.2.2.3 [Water];

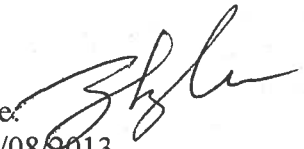
Statements: The study reported herein, "Independent Laboratory Validation (ILV) of an Environmental Chemistry Method for Detection of Bensulide in Water " was conducted and reported in compliance with the following the Good Laboratory Practice (GLP) Regulations set forth in Title 40, part 160 of the code of Federal regulations of the United States of America.

Classification: The ILV is classified as **Supplemental** for monitoring parent bensulide in water.

Deficiencies: 1). Limit of Detection (LOD) is not reported/confirmed. 2). The primary and secondary ions (m/z) cannot be viewed/confirmed from mass spectrum image in the report.

PC Code: 009801

Reviewer: He Zhong, Ph.D.
Biologist

Signature: 
Date: 11/08/2013

Executive Summary

This ILV method, MRID 49162801, has confirmed the determination of parent bensulide in water using LC-MS/MS by the environmental chemistry method (ECM, MRID 48764001) (Table 1). The limit of quantification (LOQ) for bensulide residue in river water is 1.0 µg/L and below the level of concern (LOC) for freshwater invertebrate life cycle (NOAEC = 4.2 ppb, MRID 45063401). However, the ILV did not confirm the limit of detection (LOD) at 0.24 µg/L level as reported by the ECM. No ECM study deficiency was identified by this ILV method.

Table 1. Bensulide Analytical Method Summary

Analyte(s) by Pesticide	MRID	Matrix	Method Date (m/d/y)	Registrant	Instrument	Limit of Detection (µg/L)	Limit of Quantitation (µg/L)
Independent Laboratory Validation	49162801	Water	6/30/2013	Gowan	LC-MS/MS	N/A	1.0
Environmental Chemistry Method	48764001	Water	1/25/2012	Gowan	LC-MS/MS	0.24	1.0

I. Principle of the Method

Water samples fortified with known amount of bensulide (1.0 and 10.0 µg/L) were extracted and cleaned up using solid phase extraction (SPE) cartridges. Quantitation was performed using a Shimadzu LC-MS/MS. The ion transition monitored were m/z 398.03>158.1, primary quantification, m/z 398.03>218, confirmatory quantification, and m/z 398.03>141.1, second confirmatory quantification, for bensulide. The analyte was identified by the similar retention time with that of the calibration standards, and quantified by integration of the peak area relative to the calibration curves.

II. Recovery Findings

The mean recoveries of bensulide and relative standard deviations (RSD) were within guideline requirements (mean 70-120%; RSD ≤20%) for ILV method (**Table 2**).

Table 2. Independent Laboratory Validation (ILV) Method Recoveries for Bensulide in River Water (n=5)

Analyte	Quantitation Ion	Fortification Level (µg/L)	Number of Tests	Recovery Range (%)	Mean Recovery (%)	CV (%)
Bensulide	m/z = 158.1	1	5	91.0-104.0	97.5	5.5
		10	5	80.9-99.7	92.3	7.5
	m/z=218	1	5	95.6-102.0	96.6	4.2
		10	5	81.0-97.7	91.4	6.8
	m/z=141.1	1	5	93.9-106.0	98.5	4.7
		10	5	81.6-104.0	92.8	9.0

III. Method Characteristics

The ILV method confirmation is listed in Tables 4.

Table 4. Bensulide ILV Method Characteristics Confirmation

	m/z = 158.1 Primary Ion	m/z=218 Confirmation	m/z=141.1 Confirmation
Limit of Quantitation (LOQ)	1.0 µg/L	1.0 µg/L	1.0 µg/L
Limit of Detection (LOD)	N/A	N/A	N/A
Linearity (¹ calibration curve r ² and concentration range)	r ² = 0.999 4.85 – 106 µg/L	r ² = 0.999 4.85 – 106 µg/L	r ² = 0.999 4.85 – 106 µg/L
Repeatable	Yes	Yes	Yes
Reproducible	Yes	Yes	Yes
Specific	Yes	Yes	Yes

¹calibration curve is based on linear regression ($y=a+bx$) and r-values are reported in ILV method and r²-values are calculated based on the r-values.

Linearity is established in the calibration ($y=a+bx$) using external standards. The correlation coefficient of the calibration curves was above 0.999. The **limit of quantification** (LOQ) is 1.0 µg/L. The method in general satisfies the **repeatability** criteria with mean recoveries are in the range of 70-120% and RSDs are ≤20%. **Reproducibility** is satisfactory with the independent validation confirmed the LOQ(s) established by the initial validation. This method using LCMS/MS demonstrated excellent **specificity** by selecting the following daughter and parent ions (Table 5). However, Mass Spectrum Graph in ILV method is poor selected to determine those ions visually.

Table 5. Method Specificity—LC-MS/MS Parent and Daughter ions

Analyte	Primary ion	Confirmation ion	Confirmation ion
Bensulide	158.1	218	141.1

IV. Method Deficiencies and Reviewer's Comments

- 1). ILV method fails to report or confirm the limit of detection (LOD) for the ECM.
- 2). The primary and confirmation ions cannot be identified from the mass spectrum graph in the report.

V. References

Ferguson, Ling-Jen 2013. Independent Laboratory Validation (ILV) of an Environmental Chemistry Method for Detection of Bensulide in Water MRID 49162801

Cremin, Peadar, 2012. Independent Laboratory Validation (ILV) of an Analytical Method for the Determination of Bensulide in Water. MRID 48764001