

**Test Material:** Mepiquat Chloride

**MRID:** 49586601

**Title:** Validation of Analytical Method No. 477/0: Determination of BAS 083 W (Mepiquat-chloride) in Water

**MRID:** 49569101

**Title:** Independent Laboratory Validation of Analytical Method Number 477/0: Determination of BAS 083 W (Mepiquat-chloride) in Water

**EPA PC Code:** 109101

**OCSPP Guideline:** 850.6100

### Analytical method for Mepiquat Cl in water

**Reports:** ECM: EPA MRID No. 49586601. Grote, C. 2003. Validation of Analytical Method No. 477/0: Determination of BAS 083 W (Mepiquat-chloride) in Water. BASF Study Code: 58417. BASF Registration Document No.: 2001/1014999. Report prepared and sponsored by BASF Aktiengesellschaft, Limburgerhof, Germany; and submitted by BASF Corporation, Research Triangle Park, North Carolina; 50 pages. Final report issued March 26, 2003. ILV: EPA MRID No. 49569101. Vincent, T.P. 2015. Independent Laboratory Validation of Analytical Method Number 477/0: Determination of BAS 083 W (Mepiquat-chloride) in Water. ABC Study No.: 80506. BASF Document ID: 2015/1036739. Report prepared by ABC Laboratories, Inc., Columbia, Missouri; sponsored by BASF SE, Ludwigshafwen, Germany; and submitted by BASF Crop Protection, Research Triangle Park, North Carolina; 80 pages. Original final report issued February 9, 2015. Amended final report issued February 11, 2015.

**Statements:** ECM: The study was conducted in accordance with OECD and German Good Laboratory Practice (GLP) standards (pp. 3-5 of MRID 49586601). Signed and dated No Data Confidentiality, GLP, Quality Assurance and Certification of Authenticity statements were provided (pp. 2-7).

ILV: The study was conducted in accordance with the USEPA FIFRA GLP standards (40 CFR Part 160), which are compatible with the OECD Principles of GLP (p. 3 of MRID 49569101). Signed and dated No Data Confidentiality, GLP, Quality Assurance and Certification of Authenticity statements were provided (pp. 2-5).

**Classification:** This analytical method is classified as **Supplemental**. The ILV did not validate the ion chromatography (IC) portion of BASF Analytical Method No. 477/0. The ILV did not validate the method using the most difficult

matrix available. The determinations of the LOQ and LOD were not based on scientifically acceptable procedures. The LOD for the HPLC/MS/MS analysis was not reported in the ECM. An updated ECM was not provided incorporating the significant ILV modification of the method. Insufficient recovery data was provided by the ECM to validate the LC/MS/MS portion of BASF Analytical Method No. 477/0. Recoveries were corrected for residues found in the controls in both the ECM and ILV. A reagent blank was not included in the ECM

**PC Code:** 109101

**Reviewer:** Faruque Khan, Senior Fate Scientist

**Signature:**

**Date:** 09-30-15

## Executive Summary

This analytical method, BASF Analytical Method No. 477/0, is designed for the quantitative determination of mepiquat chloride (mepiquat Cl; BAS 083 W) in water at the LOQ of 0.05 µg/kg (0.05 ppb) using ion chromatography (IC) for primary analysis and LC/MS/MS for confirmatory analysis. The LOQ is less than the lowest toxicological level (6.3 (12.6\*0.5) mg/L; USEPA, 2012a, D393458) of concern in water. The ECM laboratory used surface (lake) water and tap (sourced from three wells) water, while the ILV laboratory used only drinking (tap) water. The ILV validated the method using LC/MS/MS analysis in the second trial after one modification of the extraction procedure, but the most difficult matrix available was not used. An updated ECM was not provided incorporating the significant ILV modification of the method. The ECM provided only partial data for validating the LC/MS/MS analysis. The ILV could not validate the IC portion of BASF Analytical Method No. 477/0.

**Table 1. Analytical Method Summary**

Analyte(s) by Pesticide	MRID		EPA Review	Matrix	Method Date (dd/mm/yyyy)	Registrant	Analysis	Limit of Quantitation (LOQ)
	Environmental Chemistry Method	Independent Laboratory Validation						
Mepiquat Cl (BAS 083 W)	49586601	49569101		Water <sup>1</sup>	26/03/2003	BASF Corporation	Ion Chromatography & LC/MS/MS <sup>2</sup>	0.05 µg/kg

<sup>1</sup> Water matrices of ECM were tap and surface water (Appendices 6.1-6.2, p. 21). Tap water was taken from the water pipe of the Agricultural Center Limburgerhof. The tap water was supplied by three different wells: Well Mutterstadt, Well Schifferstadt and Well Waldsee. The surface water was taken from a lake in the palatinate forest in Kelmetschweiher. The water matrix of the ILV was uncharacterized tap water.

<sup>2</sup> Only the LC/MS/MS validated in the ILV.

## I. Principle of the Method

Water samples (1000 g) were filtered (paper filter) then fortified with 1 mL of the appropriate fortification solution in a 1 L separatory funnel (p. 14; Appendix 6.4, pp. 38, 40-41, 44 of MRID 49586601). The solution was extracted once with methylene chloride (1 x 100 mL). The organic layer was discarded. The aqueous layer was mixed with 1 mL of the Na-tetraphenylborate solution (2 g/100 mL total volume water) then extracted twice with methylene chloride (2 x 100 mL) by

shaking for *ca.* 1 minute. The organic layer was transferred to a 500 mL round bottom flask containing 100 mL of 2M HCl. The mixture was shaken for 1 minute then transferred to a separatory funnel. The organic layer was discarded; the aqueous layer was extracted with 50 mL of methylene chloride. The aqueous layer was isolated and reduced to dryness (60°C starting with 150 mbar and reducing pressure further to full vacuum). 25 mL of water was added to the dry residue, and the solution was reduced to dryness again. The method noted that HCl should be removed completely. The dry residue was dissolved in 10 mL of acetonitrile:methanol (90:10, v:v) and applied to an Al<sub>2</sub>O<sub>3</sub> column [(12 mm diameter glass column with 10 g of Al<sub>2</sub>O<sub>3</sub>:water (97:3, v:v)]. The flask was rinsed with 5 mL of acetonitrile:methanol (90:10, v:v), and the rinsate was applied to the column. The analyte was eluted from the column with 85 mL of acetonitrile:methanol (90:10, v:v). The eluate was reduced to dryness using a rotary evaporator (60°C, 150 mbar). The residue was transferred into either a 25 mL tapered flask using 3 x 5 mL methanol or a 10 mL tapered flask using 3 x 2.5 mL methanol (used for concentrations at the LOQ). After reducing to dryness (evaporation technique not described), the dry residue was dissolved in ultra pure water [0.5 mL for LOQ level; 10 mL for 5.0 ppb (100×LOQ level)], using sonication as necessary. The method noted to use transfer pipettes for methanol solvent transfers and Pasteur-pipets for solvent transfers into vials.

Samples were analyzed for mepiquat chloride using a Dionex Ion Chromatograph with suppressed conductivity detection (p. 14; Appendix 6.4, pp. 38, 41-42 of MRID 49586601). A Dionex DX-500 IC instrument was equipped with a stainless steel, PRP-1 (4.1 x 150 mm, 10- $\mu$ m) pre-column and a stainless steel, PRP-1 (4.1 x 250 mm, 10- $\mu$ m) column. The mobile phase was hexanesulfonic acid (*ca.* 2 mM in water; prepared using a cation exchange column); the regenerant was 32 g tetrabutylammonium hydroxide in 2 L total volume water. A Dionex ED 50 detector was used. The injection volume was 200  $\mu$ L. In the original technical procedure, the instrumentation and instrumental methods were the same, except that a Dionex DX-300 IC instrument and Dionex ED 40 detector were used and the injection volume was 100  $\mu$ L. The retention time was *ca.* 16-17 min. for mepiquat Cl (15.57-17.27 min. in tap water and 15.65-16.09 min. in surface water; Appendix 6.3, pp. 22-28).

The confirmation method was LC/MS/MS (Appendix 6.4, pp. 38, 45-46 of MRID 49586601). The sample processing was the similar to that described above, except that it was adapted for a reduced sample weigh-in. Fortified, filtered water samples (200 g) were extracted once with methylene chloride (1 x 50 mL). The organic layer was discarded. The aqueous layer was mixed with 1 mL of the Na-tetraphenylborate solution (2 g/100 mL total volume water) then extracted twice with methylene chloride (2 x 50 mL) by shaking for *ca.* 1 minute. The organic layer was transferred to a round bottom flask containing 50 mL of 2M HCl. The mixture was shaken for 1 minute then transferred to a separatory funnel. The organic layer was discarded; the aqueous layer was extracted with 25 mL of methylene chloride. The aqueous layer was isolated and reduced to dryness (60°C starting with 150 mbar and reducing pressure further to full vacuum). 25 mL of water was added to the dry residue, and the solution was reduced to dryness again. The method noted that HCl should be removed completely. The dry residue was dissolved in 10 mL of acetonitrile:methanol (90:10, v:v) and applied to an Al<sub>2</sub>O<sub>3</sub> column [(12 mm diameter glass column with 10 g of Al<sub>2</sub>O<sub>3</sub>:water (97:3, v:v)]. The flask was rinsed with 5 mL of acetonitrile:methanol (90:10, v:v), and the rinsate was applied to the column. The analyte was eluted from the column with 85 mL of acetonitrile:methanol (90:10, v:v). The eluate was reduced to dryness using a rotary evaporator (60°C, 150 mbar). The residue was transferred into either a 25 mL or 10 mL tapered flask small amounts of methanol. After reducing to dryness (evaporation technique not described), the dry

residue was dissolved in water:formic acid (1000:1, v:v; 2.0 mL for LOQ level; higher volumes for higher fortifications).

Samples were analyzed for mepiquat chloride using an Agilent 1100 LC binary pump HPLC system with a PE Sciex API 3000 triple stage quadrupole mass spectrometer (Appendix 6.4, p. 46 of MRID 49586601). The HPLC/MS conditions consisted of an Altima C18 column (4.6 x 100 mm, 3- $\mu$ m; column temperature room temperature), a mobile phase gradient of (A) water:formic acid (1000:1, v:v) and (B) methanol:formic acid (1000:1, v:v) [percent A:B (v:v) at 0.0-0.1 min. 100:0, 4.6-9.0 min. 10:90, 9.1-12.1 min. 100:0] and MS/MS detection in positive ESI mode with MRM. Two parent-daughter ion transitions (quantitative = Q, confirmatory = C) were monitored:  $m/z$  114.1  $\rightarrow$  98.1 (Q) and  $m/z$  114.1  $\rightarrow$  58.0 (C) for mepiquat Cl. Injection volume was 25  $\mu$ L. The retention time was *ca.* 3.6 min. for mepiquat Cl (Appendix 6.4, pp. 48-50).

In the ILV, only the confirmation method was performed for validation using the reduced sample weigh-in processing and LC/MS/MS (pp. 14-15, 19-20 of MRID 49569101). Mepiquat Cl was extracted in the same manner as the ECM, except that the organic layer was partitioned twice, instead of once, with 50 mL of 2M HCl. The extracts were analyzed for mepiquat Cl using a MDS Sciex API 5000 UPLC coupled to AB Sciex 5000 mass spectrometer. The UPLC column was an Alltima HP C18 column (4.6 x 100 mm, 3- $\mu$ m; column temperature 40°C). The mobile phase and gradient were the same as the ECM. The injection volume was 2-10  $\mu$ L. MS/MS detection used positive turbo spray ionization. The ion transitions were the same; retention time was *ca.* 2.8 min. for mepiquat Cl (Figures 10.1.15-10.1.18, pp. 37-40).

In both the ECM and ILV, the LOQ was reported as 0.05  $\mu$ g/kg for mepiquat Cl for the IC and LC/MS/MS analyses (pp. 8, 19; Appendix 6.4, pp. 42, 45 of MRID 49586601; p. 19 of MRID 49569101). In the ECM, the LOD was reported as 5 ng for the IC analysis; no LOD was reported for the HPLC/MS/MS analysis. In the ILV, the LOD was reported as 0.005  $\mu$ g/kg for UPLC/MS/MS analysis.

## II. Recovery Findings

ECM (MRID 49586601): Mean recoveries and relative standard deviations (RSD) were within guidelines (mean 70-120%; RSD  $\leq$ 20%) for primary ion chromatography analysis of mepiquat Cl in surface (lake) and tap (sourced by wells) water at the LOQ, 10 $\times$ LOQ and 100 $\times$ LOQ (Tables 1-2, pp. 17-18). Mean recoveries and relative standard deviations (RSD) were within guidelines (mean 70-120%; RSD  $\leq$ 20%) for confirmatory LC/MS/MS analysis of mepiquat Cl in surface (lake) and tap (sourced by wells) water at the LOQ (n = 2-3; no results were reported for 10 $\times$ LOQ; Appendix 6.4, Table 1, p. 47). For LC/MS/MS analysis, two parent-daughter ion transitions were reportedly monitored, but only results and spectra from the primary ion transition were reported. Quantitative and confirmatory analysis results were fairly comparable. The water matrices were characterized (Appendices 6.1-6.2, p. 21). Tap water was taken from the water pipe of the Agricultural Center Limburgerhof. The tap water was supplied by three different wells: Well Mutterstadt, Well Schifferstadt and Well Waldsee. The surface water was taken from a lake in the palatinate forest in Kelmetschweiher.

ILV (MRID 49569101): Mean recoveries and RSDs were within guidelines for LC/MS/MS analysis of mepiquat Cl in tap water at the LOQ and 10 $\times$ LOQ (pp. 7, 18-20). Ion chromatography analysis

was not validated. The LC/MS/MS portion of the method was validated with the second trial (p. 20). Two parent-daughter ion transitions were monitored. Quantitative ion and confirmatory ion results were comparable. The drinking (tap) water was not characterized (p. 10).

**Table 2. Initial Validation Method Recoveries for Mepiquat Cl in Water<sup>1,2</sup>**

Analyte	Fortification Level (µg/kg)	Number of Tests	Recovery Range (%)	Mean Recovery (%)	Standard Deviation (%)	Relative Standard Deviation (%)
<b>IC analysis</b>						
<b>Tap Water (sourced from three different wells)</b>						
Mepiquat Cl (BAS 083 W)	0.05 (LOQ)	5	67.85-79.43	72.5	4.8	6.6
	0.5	5	85.25-87.63	86.8	1.0	1.2
	5.0	5	89.77-99.37	94.7	3.5	3.7
<b>Surface Water (Lake)</b>						
Mepiquat Cl (BAS 083 W)	0.05 (LOQ)	5	77.31-86.75	81.8	4.1	5.0
	0.5	5	76.34-93.25	85.0	6.9	8.2
	5.0	5	84.09-99.05	92.2	6.6	7.2
<b>HPLC/MS/MS analysis<sup>3</sup></b>						
<b>Tap Water (sourced from three different wells)</b>						
Quantitative ion						
Mepiquat Cl (BAS 083 W)	0.05 (LOQ)	3	77.7-83.3	81.2	3.1	3.8
<b>Surface Water (Lake)</b>						
Quantitative ion						
Mepiquat Cl (BAS 083 W)	0.05 (LOQ)	2	70.7, 71.1	--	--	--

Data (corrected recovery results, pp. 14-15) were obtained from Tables 1-2, pp. 17-18 (IC results) and Appendix 6.4, Table 1, p. 47 (LC/MS/MS results) of MRID 49586601.

- The water matrices were characterized (Appendices 6.1-6.2, p. 21). Tap water was taken from the water pipe of the Agricultural Center Limburgerhof. The tap water was supplied by three different wells: Well Mutterstadt, Well Schifferstadt and Well Waldsee. The surface water was taken from a lake in the palatinate forest in Kelmetschweiher.
- Ion transitions monitored were as follows (quantitative ion and confirmatory ion, respectively):  $m/z$  114.1 → 98.1 (Q) and  $m/z$  114.1 → 58.0 (C; Appendix 6.4, p. 46).
- Statistics were not reported by the study author. Mean, s.d. and RSD values were reviewer-calculated based on recovery results reported in the study report. Statistical significant results could not be calculated for  $n = 2$ .

**Table 3. Independent Validation Method Recoveries for Mepiquat Cl in Water<sup>1,2</sup>**

Analyte	Fortification Level (µg/kg)	Number of Tests	Recovery Range (%)	Mean Recovery (%)	Standard Deviation (%)	Relative Standard Deviation (%)
<b>Drinking (tap) water</b>						
Quantitative ion						
Mepiquat Cl (BAS 083 W)	0.05 (LOQ)	5	66-79	74	5.0	6.7
	0.5	5	93-94	88	5.5	6.3
Confirmatory ion						
Mepiquat Cl (BAS 083 W)	0.05 (LOQ)	5	68-84	75	7.1	9.5
	0.5	5	75-86	82	4.2	5.1

Data (corrected recovery results, pp. 15-16) were obtained from Table 1, p. 18 of MRID 49569101.

- The drinking water was not characterized (p. 10).
- Ion transitions monitored were as follows (quantitative ion and confirmatory ion, respectively):  $m/z$  114.1 → 98.1 (Q) and  $m/z$  114.1 → 58.0 (C; Table 1, p. 18).

### III. Method Characteristics

In both the ECM and ILV, the LOQ was reported as 0.05 µg/kg for mepiquat Cl for the IC and LC/MS/MS analyses (pp. 8, 19; Appendix 6.4, pp. 42, 45 of MRID 49586601; p. 19 of MRID 49569101). In the ECM, the LOQ was defined as the lowest fortification level which obtained acceptable results. In the ECM, the LOD was reported as 5 ng for the IC analysis; no LOD was reported for the HPLC/MS/MS analysis. The LOD was defined in the ECM as the absolute amount of analyte injected into the ion chromatography instrument using the lowest standard of calibration curve (0.05 µg/mL). In the ILV, the LOD was reported as 0.005 µg/kg for UPLC/MS/MS analysis. In the ILV, the LOQ and LOD were reported from the ECM, and no justification was provided. No calculations or comparison to background levels was provided for the LOQ or LOD.

**Table 4. Method Characteristics**

		Mepiquat Cl	
		Ion Chromatography	LC/MS/MS
Limit of Quantitation (LOQ)		0.05 µg/kg	
Limit of Detection (LOD)		5 ng (0.05 µg/mL)	0.005 µg/kg
Linearity (calibration curve $r^2$ and concentration range)	ECM	$r^2 = 0.9998$ (0.05-1.0 µg/mL)	Not reported <sup>1</sup>
	ILV	Not performed	$r^2 = 0.99552028$ (Q) $r^2 = 0.99688820$ (C) (0.5-1.25 ng/mL)
Repeatable	ECM	Yes at the LOQ, 10×LOQ and 100×LOQ for surface (lake) and tap (sourced from wells) water.	Yes at the LOQ for surface (lake) water and tap (sourced from wells) water, but <b>n = 2-3</b> .
	ILV <sup>2</sup>	Not validated	Yes at the LOQ and 10×LOQ for drinking (tap) water.
Reproducible		<b>No</b>	Yes for drinking (tap) water only.
Specific	ECM	Matrix interferences were <i>ca.</i> 65% of the LOQ near the retention time of the analyte in surface water. The retention time of the analyte was fairly variable in tap water.	Matrix interferences were <i>ca.</i> 15-20% of the LOQ at the retention time of the analyte.
	ILV <sup>2</sup>	Not applicable	Matrix interferences were <i>ca.</i> 20% of the LOQ at the retention time of the analyte. Matrix effects: slight suppression (<20%).

Data were obtained from pp. 8, 17-19; Appendix 6.3, pp. 25-29; Appendix 6.4, pp. 42, 45, 47; Appendix 6.4, Figures 2-6, pp. 48-50 of MRID 49586601; pp. 18-20; Appendix 10.1, Figures 10.1.2-10.1.3, pp. 24-25; Figures 10.1.13-10.1.40, pp. 35-40 of MRID 49569101.

<sup>1</sup> Appendix 6.4, Figure 2, p. 48 of MRID 49586601 (quantitative ion transition only). No data was provided.

<sup>2</sup> The ILV was unable to successfully validate the IC analysis portion of the ECM, and the LC/MS/MS portion of the ECM using surface water (based on communications between ILV and study sponsor; Appendix 10.3, pp. 42-80 of MRID 49569101).

#### IV. Method Deficiencies and Reviewer's Comments

1. The ILV could not validate the ion chromatography (IC) portion of BASF Analytical Method No. 477/0 and did not validate the LC/MS/MS portion of the method using the most difficult matrix available. The ILV validated the method using uncharacterized tap water. Based on communications between ILV and study sponsor (Appendix 10.3, pp. 42-80 of MRID 49569101), the ILV attempted to validate the ion chromatography analysis, but was unable to validate it. The sponsor directed the ILV to terminate attempts to validate the IC analysis based on the method being out-of-date and "highly dependent on the instrumentation" (Appendix 10.3, pp. 63, 69). Also, the ILV was provided with surface water for the validation, but was unable to produce satisfactory results with this matrix, even with a second DCM/HCl extraction and "numerous tactics to attempt to get the method to work" (Appendix 10.3, pp. 69, 73, 75). The sponsor directed the ILV to provide a draft report which only included "work relating to the drinking water validation" (Appendix 10.3, p. 80).

2. The estimations of the LOQ and LOD in the ECM were not based on scientifically acceptable procedures as defined in 40 CFR Part 136. In the ECM, the LOQ was defined as the lowest fortification level which obtained acceptable results. The LOD was defined in the ECM as the absolute amount of analyte injected into the ion chromatography instrument using the lowest standard of calibration curve; no LOD was reported for the HPLC/MS/MS analysis. In the ILV, the LOD was reported for UPLC/MS/MS analysis. In the ILV, the LOQ and LOD were reported from the ECM, and no justification was provided. No calculations or comparison to background levels was provided for the LOQ or LOD.

Detection limits should not be based on the arbitrarily selected lowest concentration in the spiked samples.

3. **The only ILV modification of the extraction procedure was that the organic layer was partitioned twice, instead of once, with 50 mL of 2M HCl (pp. 14-15, 19-20; Appendix 10.3, p. 69 of MRID 49569101). This modification was considered significant since it was necessary for the successful validation of the method. An updated ECM should have been provided incorporating this modification.**

4. In the ECM, the LC/MS/MS data was insufficient for validation of that portion of the method (Appendix 6.4, Table 1, p. 47 of MRID 49586601). An insufficient number of samples were prepared ( $n = 2-3$ ). No samples were prepared for  $10\times$ LOQ. OCSPP guidelines recommend that a minimum of five spiked replicates were analyzed at each concentration (*i.e.*, minimally, the LOQ and  $10\times$  LOQ) for each analyte.

Also, two parent-daughter ion transitions were reportedly monitored (primary and confirmatory), but only results and spectra from the primary ion transition were reported (Appendix 6.4, Table 1, p. 47; Appendix 6.4, pp. 48-50 of MRID 49586601).

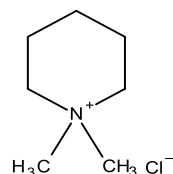
5. Recoveries were corrected for residues found in the controls in both the ECM and ILV (pp. 14-15 of MRID 49586601; pp. 15-16 of MRID 49569101). In the ECM and ILV, matrix interferences were *ca.* 15-20% of the LOQ at the retention time of the analyte for the LC/MS/MS analysis (Figures 2-6, pp. 48-50 of MRID 49586601; Figures 10.1.13-10.1.40,



- pp. 35-40 of MRID 49569101). Analyte residues were not observed in the IC control chromatograms of the ECM (Appendix 6.3, pp. 25-29 of MRID 49586601).
6. A reagent blank was not included in the ECM. LC/MS/MS chromatograms for the 10×LOQ fortification were not included, as well, since this fortification was not prepared.
  7. In the IC control chromatograms of the ECM, matrix interferences were *ca.* 65% of the LOQ near the retention time of the analyte in surface water (Appendix 6.3, pp. 25-29 of MRID 49586601). This contamination was observed to interfere with the integration of the 5 µg/kg fortification peak; it could not be determined if the contamination interfered with other fortification peaks.
  8. Matrix effects were reported in the ILV as “slight suppression (<20%)”; however, data for the quantification of the matrix effects was not provided (p. 20 of MRID 49569101).
  9. It was reported for the ILV that a set consisting of 7 samples required approximately 8-10 hours of work, including calculation of the results (LC/MS/MS analysis; p. 19 of MRID 49569101). In the ECM, the time requirement for one series of samples (7 unknown, 2 fortified and 1 blank) required two working days (15 hours) per laboratory assistant, including calculation and equipment GLP preparation (p. 19 of MRID 49586601).
  10. A significant typographical error was noted in the Discussion of the ECM, where the tested fortification levels were reported as “0.05, 0.5 and 5.0 mg/kg” instead of “0.05, 0.5 and 5.0 µg/kg”
  11. Communications between the ILV study authors and study sponsor representative were well documented in the ILV (Appendix 10.3, pp. 42-80 of MRID 49569101). Communications regarded the failed validation trials and sponsor-directed change of protocol for ILV validation.

## V. References

- U.S. Environmental Protection Agency. 2012b. Registration Review: Preliminary Problem Formulation for Environmental Fate, Ecological Risk, Endangered Species, and Drinking Water Exposure Assessments for Mepiquat Chloride and Mepiquat Pentaborate. Environmental Fate and Effects Division, Office of Pesticide Programs. Washington, DC.
- U.S. Environmental Protection Agency. 2012b. Ecological Effects Test Guidelines, OCSPP 850.6100, Environmental Chemistry Methods and Associated Independent Laboratory Validation. Office of Chemical Safety and Pollution Prevention, Washington, DC. EPA 712-C-001.
- 40 CFR Part 136. Appendix B. Definition and Procedure for the Determination of the Method Detection Limit-Revision 1.11, pp. 317-319.

**Attachment 1: Chemical Names and Structures****Mepiquat Chloride (Mepiquat Cl; BAS 083 W)****IUPAC Name:** 1,1-Dimethylpiperidinium chloride**CAS Name:** 1,1-Dimethylpiperidinium chloride**CAS Number:** 24307-26-4**SMILES String:** C1CCCCN1(Cl)(C)C

**Information related to Primary Review**

**For CDM Smith**

**Primary Reviewer:** Lisa Muto

**Signature:** 

**Date:** 8/25/15

**Secondary Reviewer:** Kathleen Ferguson

**Signature:**

**Date:** 8/25/15

**QC/QA Manager:** Joan Gaidos

**Signature:** 

**Date:** 8/25/15