| Test Material: | Sodium Bentazon | | | | | |
|-----------------------------|--|----------------------|-----------------|--|--|--|
| MRID: | 49693503 | | | | | |
| Title: | Validation of Analytical Method L0141/01 for the LC-MS/MS Determination of BAS 351 H (Bentazon) in Surface Water and Groundwater (including Amendment No. 1 and Amendment No. 2) | | | | | |
| MRID: | 49693504 | | | | | |
| Title: | Independent Laboratory Validation of Analytical Method Number L0141/01: LC-MS/MS determination of BAS 351 H (Bentazon) in surface water and groundwater | | | | | |
| EPA PC Code: | 103901 | | | | | |
| OCSPP Guideline: | 850.6100 | | | | | |
| For CDM Smith | | | | | | |
| Primary Reviewer: Lisa Muto | | Signature: | Jara Muto | | | |
| | | Date: 11/4/15 | 5 | | | |
| Secondary Reviewer: | Kathleen Ferguson | Signature: | Kathleen Jeymon | | | |
| | | Date: 11/4/15 | 5 | | | |
| | | | | | | |

QC/QA Manager: Joan Gaidos

Signature: Jour St. Date: 11/4/15

Analytical method for bentazon (BAS 351 H) in water

| Reports: | ECM: EPA MRID No. Validation of Analytica Determination of BAS (including Amendment 334546. BASF Registr sponsored and submitte Limburgerhof, Limbur Amendment 1, 6 pages July 19, 2009; Amendr Amendment No. 2 com ILV: EPA MRID No. 4 Validation of Analytica determination of BAS BASF Study No.: 4330 2015/7001577. Report Missouri, sponsored an Triangle Park, North C Final original report iss completion date, March 16, 2015; amendment (| 49693503. Penning, H., M. Obermann. 2015. Method L0141/01 for the LC-MS/MS 851 H (Bentazon) in Surface Water and Groundwater No. 1 and Amendment No. 2). BASF Study ID No.: tion Document No.: 2015/7001892. Report prepared: d by BASF SE, BASF Agricultural Center erhof, Germany; 63 pages (Original report, 49 pages: Amendment 2, 8 ages). Final original report issued ent No. 1 completion date, February 20, 2014; pletion date, July 09, 2015. 9693504. Grant, J. 2015. Independent Laboratory I Method Number L0141/01: LC-MS/MS 51 H (Bentazon) in surface water and groundwater. 95. BASF Registration Document No.: orepared by ABC Laboratories, Inc., Columbia, a submitted by BASF Crop Protection, Research rolina; 150 pages total (including 60a, 61a and 62a). ued November 16, 2012; amendment (first) 5, 2014; amendment (second) completion date, June hird) completion date, July 16, 2015. |
|--------------------------------|--|---|
| Document No.: | MRIDs 49693503 (EC | 1) & 49693504 (ILV) |
| Guideline: | 850.6100 | |
| Statements: Classification: | ECM: The study was c GLP, and with USEPA standards (p. 3 of MRI Confidentiality, GLP, C statements were provid were also included (pp. ILV: The study was co MRID 49693504). Sign Assurance and Certific 5). A signatures page for included (p. 6). This analytical method | onducted in accordance with OECD and German GLP which corresponds with those other GLP 0 49693503). Signed and dated No Data puality Assurance and Certification of Authenticity ed (pp. 2-5). Signatures pages for the Amendments 55, 63). Iducted in accordance with USEPA GLP (p. 3 of ed and dated No Data Confidentiality, GLP, Quality tion of Authenticity statements were provided (pp. 2- r the Amended Final Report (July 16, 2015) was also is classified as ACCEPTABLE . The determinations |
| Ciassification: | of the LOQ and LOD v ILV linearity coefficien H. The number of samp in groundwater. The so | ere not based on scientifically acceptable procedures ts were <0.995 for all calibration curves of BAS 351 les was insufficient for fortifications of BAS 351 H irce of the ILV surface water was not reported. |
| PC Code: | 103901 | |
| Reviewer: | He Zhong, Ph.D. Biologist | Signature: Date: 2-1-2016 |

<u>Referenced page numbers for ECM MRID 49693503 refer to those listed in the bottom righthand corner of the document. Referenced page numbers for ECM MRID 49693504 refer to those listed in the upper right-hand corner of the document.</u>

Executive Summary

This analytical method, BASF Analytical Method L0141/01, is designed for the quantitative determination of bentazon (BAS 351 H) in water at the LOQ of 0.03 μ g/kg (0.03 ppb) using HPLC/MS/MS. The LOQ is much less than the lowest toxicological level of concern (900 ppb) in water. The method was validated by the ECM using surface water (pond) and groundwater (tap water sourced by four wells). The method was validated by the ILV with the first trial using surface water (source not specified) with minor modifications. ILV calibration data was insufficient to support the method. The number of samples was insufficient for fortifications of BAS 351 H in groundwater at the LOQ (n = 4) due to an experimental error with one sample.

| A malasta (a) | maluta(a) MRID | | | | | | | Limit of Quantitation (LOQ) |
|----------------------------|--------------------------------------|---|----------------------|----------------------|--|---------------------|------------|-----------------------------------|
| by Pesticide | Environmental Chemistry Method | Independent Laboratory Validation | EPA Review Matrix | | Method Date (dd/mm/yyyy) | Registrant | Analysis | |
| Bentazon (BAS 351 H) | 49693503 | 49693504 | | Water ^{1,2} | 14/07/2009 (Original) 20/02/2014 (Amendment 1) 09/07/2015 (Amendment 2) | BASF Corporation | HPLC/MS/MS | 0.03 μg/kg (0.03 ppb) |

Table 1. Analytical Method Summary

1 In the ECM, water matrices were fully characterized (p. 10; Appendices 5.1-5.2, pp. 21-27 of MRID 49693503). The groundwater was the tap water of the test facility which was sourced by four wells: Well Böhl (pH 7.81, total organic carbon 1.3 mg/L, oxygen content 9.2 mg/L); Well Schifferstadt (pH 7.7, total organic carbon 0.9 mg/L, oxygen content 9.1 mg/L); Well Mutterstadt (pH 7.72, total organic carbon 1.3 mg/L, oxygen content 1 mg/L); Well Mutterstadt (pH 7.72, total organic carbon 1.4 mg/L). The surface water was taken from Kelmetschweiher, a small pond in the wood between Schifferstadt and Iffelheim (pH 8.7, total organic carbon 14.0 mg/L, dissolved oxygen 9.5 mg/L).

2 In the ILV, surface water (PA Water; pH 8.6, total dissolved oxygen 6.8 mg/L) was used (source not reported; p. 12; Appendix 5, pp. 144-145 of MRID 49693504).

I. Principle of the Method

Unfiltered water samples (10 g) were measured into 50-mL beakers and fortified, as necessary, then acidified to pH 2 with 30 μ L of S2 (6M HCl; Appendix 5.4, pp. 40-42, 46 of MRID 49693503). The acidified water sample was applied to the Bond Elut-ENV SPE column [pre-conditioned with 3 x 3 mL of methanol then S3 (pH 2 water, prepared with 6M HCl and water); flow *ca*. 1 mL/min.]. The beaker was rinsed with 3 mL of S4 (pH 2 water, prepared with conc. HCOOH and water) then applied to the SPE column (flow *ca*. 1 mL/min.). Air dry the column for 5 min. under vacuum (400 mbar) then with a stream of nitrogen at 40°C for 30-45 min. The column was transferred to the Processing Station (VacMaster Sample; Appendix 5.4, pp. 40, 42; 49). The analyte was eluted with 3 x 3 mL of methanol (Appendix 5.4, pp. 40-42, 46). The collected eluates were reduced to dryness in the evaporator (TurboVap LV Evaporator) at 40°C (water bath). The residue was reconstituted with S1 [methanol:water (50:50, v:v); e.g. final volume = 2 mL for LOQ concentrations). An aliquot (50 μ L) of the final extract was measured for HPLC/MS/MS analysis.

Samples were analyzed using an Agilent 1100 LC Binary Pump LC System coupled to an AB Sciex API 3000 Triple Stage Quadrupole Mass Spectrometer with Electrospray (ESI; Appendix 5.4, pp. 43-44 of MRID 49693503). The instrumental conditions consisted of a Betasil C18 column (2.1 x 100 mm, 5-µm; column temperature RT), 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 0.0min. 50:50, 2.5-4.0 min. 35:65, 4.1-6.0 min. 0:100, 6.1-9.0 min. 50:50] and MS/MS detection (MRM) in negative ion mode for BAS 351 H. Two parent-daughter ion transitions (quantitative = Q, confirmatory = C) were monitored: $m/z 239 \rightarrow 132$ (Q) and $m/z 239 \rightarrow 197$ (C) for bentazon (BAS 351 H). Approximate retention time was *ca*. 2.6 min. for bentazon (BAS 351 H). Injection volume was 50 µL.

Amendments to the original report did not include any changes to the extraction or analytical procedure; however, the alternative use of N-evaporation at *ca*. 40°C for heated SPE column dryer was added to the method based on hardware limitation and the successful testing of the ILV (Appendix 5.4, Amendment 2, p. 57 of MRID 49693503).

ILV

In the ILV, the extraction procedure was the same as the ECM, except that N-evaporation at *ca*. 40°C was used instead of heated SPE column dryer due to hardware limitation (pp. 13-15; Appendix 2, p. 89 of MRID 49693504). HPLC/MS/MS analysis was performed using an Agilent 1100 with a HTC PAL LC system coupled to a MDS Sciex API 4000 Mass Spectrometer. The instrumental conditions were the same as those of the ECM, except that the column temperature was not reported and the injection volume was reduced to 5 μ L. The same two parent-daughter ion transitions (quantitative = Q, confirmatory = C) were monitored. Approximate retention time was *ca*. 2.5 min. for bentazon (BAS 351 H).

LOQ/LOD

For bentazon (BAS 351 H), the LOQ and LOD were reported as 0.03 μ g/kg (0.03 ppb) and 0.005 μ g/kg (17% of the LOQ; 1.25 pg), respectively, in both the ECM and ILV (pp. 6, 19-20; Appendix

5.4, p. 47; Appendix 5.4, Amendment 1, p. 52; Appendix 5.4, Amendment 2, p. 57 of MRID 49693503; pp. 7, 15 of MRID 49693504).

II. Recovery Findings

ECM (MRID 49693503): All mean recoveries and relative standard deviations (RSD) were within guidelines (mean 70-120%; RSD \leq 20%) in groundwater and surface water matrices for bentazon (BAS 351 H) at the LOQ (0.03 µg/kg; 0.01 ppb) and 10×LOQ (0.3 µg/kg; 0.3 ppb; pp. 6-7; Tables 1-4, pp. 15-18). Results of the quantitation and confirmation ions were comparable. The number of samples was five for all analyses, except those for BAS 351 H groundwater at the LOQ (n = 4). Originally five samples were prepared for the fortification level, but one sample was spiked twice unintentionally (Tables 1-2, pp. 15-16). The sample data was discarded by the ECM study authors prior to statistical calculations. Calculations allowed for recoveries to be corrected for residues found in the controls; however, none were quantified (pp. 12-13; Tables 1-4, pp. 15-18). The water matrices were fully characterized by Institut für analytische Chemie (Germany) and DAR (Germany; p. 10; Appendices 5.1-5.2, pp. 21-27). The groundwater was the tap water of the test facility which was sourced by four wells: Well Böhl (pH 7.81, total organic carbon 1.3 mg/L, oxygen content 9.2 mg/L); Well Schifferstadt (pH 7.7, total organic carbon 0.9 mg/L, oxygen content 9.1 mg/L); Well Mutterstadt (pH 7.72, total organic carbon 1.3 mg/L, oxygen content 1 mg/L); and Well Waldsee (pH 7.74, total organic carbon 2.2 mg/L, oxygen content 1.4 mg/L). The surface water was taken from Kelmetschweiher, a small pond in the wood between Schifferstadt and Iffelheim (pH 8.7, total organic carbon 14.0 mg/L, dissolved oxygen 9.5 mg/L).

ILV (MRID 49693504): Mean recoveries and RSDs were within guidelines in surface water matrices for bentazon (BAS 351 H) at the LOQ (0.03 μ g/kg; 0.01 ppb) and 10×LOQ (0.3 μ g/kg; 0.3 ppb; Tables 1-2, pp. 17-19). Results of the quantitation and confirmation ions were comparable. Calculations allowed for recoveries to be corrected for residues found in the controls; however, none were quantified (Table 2, pp. 18-19; Figure 2, p. 21). The method was validated with the first trial with minor modifications (p. 12). The water matrix was provided by BASF and well characterized by Agvise Laboratories, Northwood, North Dakota (p. 12; Appendix 5, pp. 144-145). Surface water (PA Water; pH 8.6, total dissolved oxygen 6.8 mg/L) was used (source not reported).

| Analyte | Fortification | Number of | Recovery | Mean | Standard | Relative Standard | |
|-----------------------------|---------------|----------------|-----------------|--------------|----------------------|--------------------------|--|
| | Level (ppb) | Tests | Range (%) | Recovery (%) | Deviation (%) | Deviation (%) | |
| | | | Groundwat | er | | | |
| | | Pı | rimary Quantifi | cation | | | |
| Bentazon | 0.03 (LOQ) | 4 ³ | 89.3-104.0 | 94.0 | 6.8 | 7.3 | |
| (BAS 351 H) | 0.3 | 5 | 80.0-84.0 | 82.1 | 1.6 | 1.9 | |
| | | Cont | firmatory Quan | tification | | | |
| Bentazon | 0.03 (LOQ) | 4 ³ | 90.7-104.7 | 94.8 | 6.6 | 7.0 | |
| (BAS 351 H) | 0.3 | 5 | 81.3-85.3 | 82.8 | 1.5 | 1.8 | |
| | Surface Water | | | | | | |
| Primary Quantification | | | | | | | |
| Bentazon | 0.03 (LOQ) | 5 | 70.7-82.0 | 77.5 | 5.1 | 6.6 | |
| (BAS 351 H) | 0.3 | 5 | 83.3-86.7 | 85.7 | 1.5 | 1.7 | |
| Confirmatory Quantification | | | | | | | |
| Bentazon (BAS 351 H) | 0.03 (LOQ) | 5 | 70.0-80.7 | 76.1 | 5.0 | 6.6 | |
| | 0.3 | 5 | 82.7-87.3 | 85.3 | 1.7 | 2.0 | |

Table 2. Initial Validation Method Recoveries for Bentazon (BAS 351 H) in Two Waters^{1,2}

Data (uncorrected recovery results; Tables 1-4, pp. 15-18) were obtained from Tables 1-4, pp. 15-18 of MRID 49693503.

1 The water matrices were fully characterized by Institut für analytische Chemie (Germany) and DAR (Germany; p. 10; Appendices 5.1-5.2, pp. 21-27). The groundwater was the tap water of the test facility which was sourced by four wells: Well Böhl (pH 7.81, total organic carbon 1.3 mg/L, oxygen content 9.2 mg/L); Well Schifferstadt (pH 7.7, total organic carbon 0.9 mg/L, oxygen content 9.1 mg/L); Well Mutterstadt (pH 7.72, total organic carbon 1.3 mg/L, oxygen content 1 mg/L); and Well Waldsee (pH 7.74, total organic carbon 2.2 mg/L, oxygen content 1.4 mg/L). The surface water was taken from Kelmetschweiher, a small pond in the wood between Schifferstadt and Iffelheim (pH 8.7, total organic carbon 14.0 mg/L, dissolved oxygen 9.5 mg/L).

2 Ion transitions monitored were as follows (quantitative ion and confirmatory ion, respectively): $m/z 239 \rightarrow 132$ and $m/z 239 \rightarrow 197$ for bentazon (BAS 351 H; Appendix 5.4, pp. 43-44).

3 Originally five samples were prepared for the fortification level, but one sample was spiked twice unintentionally (Tables 1-2, pp. 15-16). The sample yielded 218.7% and 218.0% recoveries in the quantification and confirmation ions, respectively. The sample data was discarded by the ECM study authors prior to statistical calculations.

| Analyte | Fortification Level (ppb) | Number of Tests | Recovery Range (%) | Mean Recovery (%) | Standard Deviation (%) | Relative Standard Deviation (%) | | |
|-----------------------------|------------------------------|--------------------|-----------------------|----------------------|---------------------------|------------------------------------|--|--|
| | Surface Water | | | | | | | |
| | Primary Quantification | | | | | | | |
| Bentazon (BAS 351 H) | 0.03 (LOQ) | 5 | 85-102 | 90 | 11.1 | 12.2 | | |
| | 0.3 | 5 | 85-107 | 94 | 12.6 | 13.4 | | |
| Confirmatory Quantification | | | | | | | | |
| Bentazon (BAS 351 H) | 0.03 (LOQ) | 5 | 72-95 | 84 | 10.2 | 12.1 | | |
| | 0.3 | 5 | 70-97 | 88 | 11.1 | 12.6 | | |

Table 3. Independent Validation Method Recoveries for Bentazon (BAS 351 H) in One Water^{1,2}

Data (uncorrected recovery results, Table 2, pp. 18-19) were obtained from Tables 1-2, pp. 17-19 of MRID 49693504.

1 The water matrix was provided by BASF and well-characterized by Agvise Laboratories, Northwood, North Dakota (p. 12; Appendix 5, pp. 144-145). Surface water (PA Water; pH 8.6, total dissolved oxygen 6.8 mg/L) was used (source not reported).

2 Ion transitions monitored were as follows (quantitative ion and confirmatory ion, respectively): $m/z 239 \rightarrow 132$ and $m/z 239 \rightarrow 197$ for bentazon (BAS 351 H; Appendix 2, p. 89).

III. Method Characteristics

For bentazon (BAS 351 H), the LOQ and LOD were reported as 0.03 μ g/kg (0.03 ppb) and 0.005 μ g/kg (17% of the LOQ; 1.25 pg), respectively, in both the ECM and ILV (pp. 6, 19-20; Appendix 5.4, p. 47; Appendix 5.4, Amendment 1, p. 52; Appendix 5.4, Amendment 2, p. 57 of MRID 49693503; pp. 7, 15 of MRID 49693504). In the ECM, the LOQ was defined as the lowest fortification level successfully tested. The ECM study authors also noted that the LOQ covers "any toxicological or ecotoxicological endpoints in the aquatic compartment" (Appendix 5.4, Amendment 2, p. 57 of MRID 49693503). The LOD was defined as the absolute amount of analyte injected into the LC/MS/MS instrument using the lowest standard of the calibration curve (Appendix 5.4, p. 47; Appendix 5.4, Amendment 1, p. 52 of MRID 49693503).

Table 4. Method Characteristics

| | | Bent (BAS | tazon 351 H) | | | |
|---|------------------|--|--|--|--|--|
| Limit of Quantitation (LOQ) | | 0.03 µg/kg (0.03 ppb) | | | | |
| Limit of Detection (LOD) | | 0.005 μg/kg (17% of the LOQ; corresponds to 1.25 pg) | | | | |
| Linearity ECM ¹ (calibration curve r ² | | $ \begin{array}{c} r^2 = 0.9992 \ (Q) \\ r^2 = 0.9994 \ (C) \end{array} $ | | | | |
| and concentration | | (0.025-1.0 ng/mL) | | | | |
| range) | ILV ² | $r^2 = 0.9912 (Q)$ $r^2 = 0.9920 (C)$ | | | | |
| | | (0.025-0.50 ng/mL) | | | | |
| Repeatable | | Groundwater | Surface water | | | |
| | ECM ³ | Yes at LOQ $(n = 4)$. ⁴ Yes at 10×LOQ $(n = 5)$. | Yes at LOQ and $10 \times LOQ$; (n = 5). | | | |
| | ILV ⁵ | Not performed. | Yes at LOQ and $10 \times LOQ$; (n = 5). | | | |
| Reproducible | ECM ILV | - Yes at LOQ and 10×LOQ (surface water). | | | | |
| Specific | ECM | Groundwater | Surface water | | | |
| | | Matrix interferences at analyte retention time were 17% of the LOQ response (=LOD) in chromatograms. | Matrix interferences at analyte retention time were <5% of the LOQ response (<lod) chromatograms.<="" in="" td=""></lod)> | | | |
| | | Chromatograms were provided for each matrix. | | | | |
| | ILV | Matrix interferences at analyte retention times were <10% of the LOQ response (<lod) (surface="" chromatograms="" chromatograms;="" confirmation="" in="" interferences="" no="" observed="" quantitation="" td="" the="" water).<="" were=""></lod)> | | | | |

Data were obtained from pp. 6, 19-20; Tables 1-4, pp. 15-18; Appendix 5.3, pp. 29-33; Appendix 5.4, pp. 44, 47; Appendix 5.4, Amendment 1, p. 52; Appendix 5.4, Amendment 2, p. 57; Appendix 5.6, pp. 58-60 of MRID 49693503; pp. 7, 15; Tables 1-2, pp. 17-19; Figures 3-5, pp. 22-37 of MRID 49693504. Q = quantitation ion; C = confirmation ion. 1 ECM r² values are reviewer-generated from reported r values of 0.9996- 0.9997 for BAS 351 H (ions combined; Appendix 5.3, p. 33 of MRID 49693503; DER Attachment 2).

- 2 ILV r² values are reviewer-generated from reported r values of 0.9956-0.9960 for BAS 351 H (ions combined; Figure 3, pp. 22-23 of MRID 49693504; DER Attachment 2).
- 3 In the ECM, water matrices were fully characterized (p. 10; Appendices 5.1-5.2, pp. 21-27 16 of MRID 49693503). The groundwater was the tap water of the test facility which was sourced by four wells: Well Böhl (pH 7.81, total organic carbon 1.3 mg/L, oxygen content 9.2 mg/L); Well Schifferstadt (pH 7.7, total organic carbon 0.9 mg/L, oxygen content 9.1 mg/L); Well Mutterstadt (pH 7.72, total organic carbon 1.3 mg/L, oxygen content 1 mg/L); and Well Waldsee (pH 7.74, total organic carbon 2.2 mg/L, oxygen content 1.4 mg/L). The surface water was taken from Kelmetschweiher, a small pond in the wood between Schifferstadt and Iffelheim (pH 8.7, total organic carbon 14.0 mg/L, dissolved oxygen 9.5 mg/L).
- 4 Originally five samples were prepared for the fortification level, but one sample was spiked twice unintentionally (Tables 1-2, pp. 15-16 of MRID 49693503). The sample yielded 218.7% and 218.0% recoveries in the quantification and confirmation ions, respectively. The sample data was discarded by the ECM study authors prior to statistical calculations.
- 5 In the ILV, surface water (PA Water; pH 8.6, total dissolved oxygen 6.8 mg/L) was used (source not reported; p. 12; Appendix 5, pp. 144-145 of MRID 49693504).

Linearity is satisfactory when $r^2 \ge 0.995$.

IV. Method Deficiencies and Reviewer's Comments

 The ECM MRID 49693503 was a combination of BASF Reg. Doc# 2009/1076476 (EPA MRID 48970702), 2014/1083366, & 2015/1111335; ECM MRID 49693503 replaced MRID 48970702 (p. 1 of MRID 49693503). The ILV referenced BASF Reg. Doc# 2009/1076476 (EPA MRID 48970702) and BASF Reg. Doc# 2014/1083366, but not the updated MRID 49693503 or BASF Reg. Doc# 2015/1111335 (p. 16 of MRID 49693504).

The changes to the amended final reports of the ILV were listed in Appendix 6 (p. 150) of MRID 49693504). Among the changes, the ILV BASF Reg. Doc# changed from 2014/7000479 (EPA MRID 49338404) to 2015/7001110 to 2015/7001577.

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 and ILV, the LOQ was defined as the lowest fortification level successfully tested (pp. 6, 19-20; Appendix 5.4, p. 47; Appendix 5.4, Amendment 1, p. 52; Appendix 5.4, Amendment 2, p. 57 of MRID 49693503; pp. 7, 15 of MRID 49693504). The LOD was defined as the absolute amount of analyte injected into the LC/MS/MS instrument using the lowest standard of the calibration curve. No calculations or comparison to background levels were provided for the LOQ or LOD.

Detection limits should not be based on the arbitrarily selected lowest concentration in the spiked samples. Additionally, the lowest toxicological level of concern in water was not reported; however, the ECM study authors noted that the LOQ covers "any toxicological or ecotoxicological endpoints in the aquatic compartment" (Appendix 5.4, Amendment 2, p. 57 of MRID 49693503). An LOQ above toxicological level of concern results in an unacceptable method classification.

- 3. In the ILV, the linearity coefficients were <0.995 for both calibration curves of BAS 351 H (Figure 3, pp. 22-23 of MRID 49693504; DER Attachment 2).
- 4. The number of samples was insufficient for fortifications of BAS 351 H in groundwater at the LOQ (n = 4; Tables 1-2, pp. 15-16 of MRID 49693503). Originally five samples were prepared for the fortification level, but one sample was spiked twice unintentionally. The sample yielded 218.7% and 218.0% recoveries in the quantification and confirmation ions, respectively. The sample data was discarded by the ECM study authors prior to statistical calculations. The reviewer did not recalculate the recovery statistics since the results were due to a gross experimental error.
- 5. The source of the ILV surface water was not reported (p. 12; Appendix 5, pp. 144-145 of MRID 49693504).
- 6. In the ECM, representative chromatograms were not provided for all calibration standards (Appendix 5.3, pp. 28-32; Appendix 5.6, pp. 58-60 of MRID 49693503). Only chromatograms of the LOQ and 10×LOQ, control, reagent blank and one calibration standard were provided for each matrix.

- 7. The ILV study report noted that no recommendations or suggestions for the ECM were needed (p. 15 of MRID 49693504).
- 8. In the ILV, the communications between the ILV study author and study sponsor/ECM study authors was provided (pp. 12, 15; Appendix 4, pp. 91-142 of MRID 49693504). No technical guidance was required. Communication regarded "clarifications of protocol and report preparation, minor method modifications, fortification scheme, receipt of test substance and control matrix, and instrument optimization, including after trial initiation communications concerning recovery results with the study monitor" (p. 15).
- 9. In the ILV, it was reported that a set consisting of 13 samples required approximately 14 work hours, plus additional HPLC-MS/MS determination time (p. 13 of MRID 49693504).
- 10. The ILV listed two protocol changes: 1) the substitution of the drying apparatus and the correction of language in the experimental design regarding QCSs (quality control samples); and 2) the LOD was set to 0.005 ppb (17% of the LOQ; pp. 15-16 of MRID 49693504).

V. References

- U.S. Environmental Protection Agency. 2012. 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

Bentazon (BAS 351 H; Reg. No. 51929)

IUPAC Name:3-Isopropyl-1H-2,1,3-benzothiadiazin-4(3H)-one 2,2-dioxide.CAS Name:Not reportedCAS Number:25057-89-0SMILES String:Not found

Ν