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**Environmental Chemistry Method for Solatenol in Water (Method GRM042.01A)**

**Reports:** *ECM:* Braid S and Lin K (2011). SYN545192 – Residue Method for the Determination of SYN545192 in Water. Syngenta Ltd., Jealott's Hill International Research Centre, Bracknell, United Kingdom. Issued March 2011. Unpublished Method No. GRM042.01A (Syngenta Regulatory Document No. SYN545192\_10094), EPA MRID No 48604414.

Validation of SYN545192 – Residue Method for the Determination of SYN545192 in Water. Syngenta Crop Protection, Inc., Greensboro, NC, USA. Study Date: July 2010. Unpublished Report No. TK0002507 (Syngenta Regulatory Document No. SYN545192\_50014), EPA MRID No 48604415.

*ILV:* None

**Document No.:** MRIDs 48604414 & 48604415

**Guideline:** USEPA 850.6100  
PMRA 8.2.2.3

**Statements:** The study was conducted in accordance with USEPA FIFRA Good Laboratory Practice (GLP) Standards, 40 CFR Part 160 (pp. 3, 16). Signed and dated Data Confidentiality, GLP, and Quality Assurance statements were provided (pp. 2-4).

**Classification:** This analytical method is classified as **Acceptable**. However, there has been no independent laboratory validation submitted accompanying the method. There are two reports provided for the method which appear to convey identical information (MRID Nos. 48604414 and 48604415). Overall mean recoveries of SYN545192 from municipal drinking water was less than 90 percent in all cases, but always greater than 80.6 percent in the initial validation of the method.

**PC Code:** 122305

**Reviewer:** Gabe Rothman  
Environmental Scientist

**Signature:** 

**Date:** August 29, 2013

**Executive Summary**

Environmental water samples are concentrated using solid phase extraction (SPE) on a proprietary polymeric sorbent. After elution with acetonitrile, samples are diluted with ultra-pure water and analysed by high performance liquid chromatography with triple quadrupole mass spectrometric detection (LC-MS/MS). The limit of quantification of the method is 0.05 µg/L. Interferences arising from matrices tested (representative water types shown in Table IIA 4.5.01) have not been observed.

The validated limit of quantification is 0.001 mg/kg.

**I. Recovery Findings*****Initial Validation of Method***

**Table 1. Recoveries of SYN545192 from water (primary transition  $m/z = 396/368$ ) in initial validation.**

| Matrix         | Fortification level (µg/L) | Recovery (%)                 | Number of analyses (n) | Mean recovery (%) | RSD (%) | Recovery range (%) |
|----------------|----------------------------|------------------------------|------------------------|-------------------|---------|--------------------|
| Drinking water | 0.05                       | 84.2, 82.9, 97.3, 86.1, 83.2 | 5                      | 86.7              | 6.96    | 82.9-97.3          |
|                | 0.5                        | 82.6, 78.4, 80.0, 76.2, 85.1 | 5                      | 80.5              | 4.34    | 76.2-85.1          |
|                | Overall                    |                              | 10                     | 83.6              | 6.82    | 76.2-97.3          |
| Surface water  | 0.05                       | 86.7, 103, 97.3, 95.7, 98.2  | 5                      | 96.2              | 6.19    | 86.7-103           |
|                | 0.5                        | 99.8, 95.4, 94.4, 90.2, 98.9 | 5                      | 95.7              | 4.01    | 90.2-99.8          |
|                | Overall                    |                              | 10                     | 96.0              | 4.93    | 86.7-103           |
| Ground water   | 0.05                       | 104, 103, 108, 95.4, 109     | 5                      | 104               | 5.18    | 95.4-109           |
|                | 0.5                        | 97.6, 96.3, 96.6, 95.0, 99.8 | 5                      | 97.1              | 1.85    | 95.0-97.6          |
|                | Overall                    |                              | 10                     | 100               | 5.19    | 95.0-109           |

**Table 2: Recoveries of SYN545192 from water (confirmatory transition  $m/z=396/91$ ) in initial validation.**

| Matrix         | Fortification level ( $\mu\text{g/L}$ ) | Recovery (%)                 | Number of analyses (n) | Mean recovery (%) | RSD (%) | Recovery range (%) |
|----------------|---|------------------------------|------------------------|-------------------|---------|--------------------|
| Drinking water | 0.05                                    | 86.4, 85.4, 83.8, 73.9, 79.0 | 5                      | 81.7              | 6.37    | 73.9-86.4          |
|                | 0.5                                     | 80.3, 79.4, 75.2, 81.0, 81.9 | 5                      | 79.6              | 3.27    | 75.2-81.9          |
|                | Overall                                 |                              | 10                     | 80.6              | 5.01    | 73.9-86.4          |
| Surface water  | 0.05                                    | 95.0, 112, 96.6, 91.8, 102   | 5                      | 99.5              | 7.95    | 91.8-112           |
|                | 0.5                                     | 99.8, 93.8, 89.9, 92.8, 97.6 | 5                      | 94.8              | 4.15    | 89.9-99.8          |
|                | Overall                                 |                              | 10                     | 97.1              | 6.58    | 89.9-112           |
| Ground water   | 0.05                                    | 93.4, 87.7, 104, 109, 106    | 5                      | 100               | 9.05    | 87.7-109           |
|                | 0.5                                     | 95.0, 95.7, 94.1, 97.6, 98.9 | 5                      | 96.3              | 2.03    | 94.1-98.9          |
|                | Overall                                 |                              | 10                     | 98.1              | 6.61    | 87.7-109           |

## II. Method Characteristics

### Specificity

LC-MS/MS is a highly specific detection technique and therefore a further confirmatory technique is not required. The method includes a primary and a confirmatory MS/MS transition, both of which have been validated for representative water samples.

### Linearity

The response of the LC-MS/MS detector was shown to be linear ( $r > 0.99$ ) for both primary and confirmatory transitions for SYN545192 over a concentration range of 1.25 to 50 pg injected on column (equivalent to 0.05 pg/ $\mu\text{L}$  – 2 pg/ $\mu\text{L}$  standards using a 25  $\mu\text{L}$  injection) for water.

### Accuracy

The overall SYN545192 mean recovery values for the 396 $\rightarrow$ 368  $m/z$  transition were 100% for ground water, 96.0% for surface water and 83.6% for municipal tap water. The overall SYN545192 mean recovery values for the 396  $\rightarrow$  91  $m/z$  transition were 98.1% for ground water, 97.1% for surface water and 79.6% of municipal tap water demonstrating acceptable accuracy of the method for both transitions.

### Precision

The overall SYN545192 relative standard deviations for the 396 $\rightarrow$ 368  $m/z$  transition were 5.22%, 4.73% and 5.71% for ground water, surface water and municipal tap water respectively. For the 396  $\rightarrow$  91  $m/z$  transition they were 6.61%, 6.58% and 5.01% for ground water, surface water and municipal tap water respectively. The precision of the method is therefore acceptable for both transitions.

### Limit of Quantification

The validated limit of quantification of the method for ground water, surface water and municipal tap water is 0.05 µg/L quantified on both the  $m/z$  396→368 transition and the  $m/z$  396→91 transition.

### Matrix effects

Matrix effects were assessed for each matrix type and are shown in Table 5. They are considered to be insignificant (< 20% enhancement or suppression). Solvent standards were therefore used for quantification.

**Table 5. LC-MS/MS Matrix Effects**

| Analyte/Transition        | Matrix Effect    |                 |                 |
|---------------------------|------------------|-----------------|-----------------|
|                           | Drinking water   | Surface Water   | Groundwater     |
| SYN545192 $m/z$ = 396→368 | -16% suppression | -4% suppression | 1% enhancement  |
| SYN545192 $m/z$ = 396→91  | -19% suppression | -3% suppression | -2% suppression |

### Extract stability

The stability of SYN545192 in the final sample of the extract of a control drinking water sample fortified with SYN545192 at the LOQ (0.05 µg/L) in acetonitrile/ultra-pure water 50/50 v/v prior to and following storage at 4°C for a period of 7 days is shown in Table 6 (primary transition  $m/z$  396→368, non-matrix standard).

**Table 6. SYN545192 Recovery before and after 7 days storage of extract at 4°C**

| Matrix       | Fortification (µg/L) | Storage intervals (days) | Recovery %                   | n | Mean (%) | RSD (%) | Range (%) |
|--------------|----------------------|--------------------------|------------------------------|---|----------|---------|-----------|
| Ground water | 0.05                 | 0                        | 104, 103, 108, 95.4, 109     | 5 | 104      | 5.31    | 95.4-109  |
| Ground water | 0.05                 | 7                        | 119, 110, 94.4, 93.1, 111    | 5 | 105      | 10.7    | 93.1-119  |
| Ground water | 0.5                  | 0                        | 97.6, 96.3, 96.6, 95.0, 99.8 | 5 | 97.1     | 1.84    | 95.0-99.8 |
| Ground water | 0.5                  | 7                        | 104, 104, 113, 101, 107      | 5 | 106      | 4.13    | 101-113   |

No significant degradation of SYN545192 was observed in the extract under these storage conditions. Stability in other water types is assumed to be similar.

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

1. There has been no independent laboratory validation submitted accompanying the method. There are two reports provided for the method which appear to convey identical information (MRID Nos. 48604414 and 48604415).
2. Overall mean recoveries of SYN545192 from municipal drinking water was less than 90 percent in all cases, but always greater than 80.6 percent in the initial validation of the method.

**IV. References**

1. Luxon S G (1992): Hazards in the Chemical Laboratory 5th Edition. The Royal Society of Chemistry. Thomas Graham House, The Science Park, Cambridge CB4 4WF, UK. ISBN 0-85186-229-2.
2. Cardone M J, Palermo P J and Sybrand L B: Potential error in single point ratio calculations based on linear calibration curves with a significant intercept. Anal Chem., 52 pp 1187-1191, 1980.
3. Mayer L (2010): Validation of SYN545192 - Residue Method GRM042.01A for the Determination of SYN545192 in Water. Syngenta report number TK0002507.