

## Environmental Chemistry Method for SYN545192 and SYN546206 (Solatenol Degradates) in Soil (Method GRM042.05A)

**Reports:** *ECM:* Braid, S.; Wiesner, F.; Breyer, N. (2012) SYN545192 - Analytical Method GRM042.05A for the Determination of SYN545192 and its Metabolite SYN546206 in Soil. Project Number: T001407/08/OCR, GRM042/05A. Unpublished study prepared by Jealott's Hill Res. Station EPA MRID No 48604560.

Wiesner, F.; Breyer, N. (2012) SYN545192 - Validation of an Analytical Method for the Determination of Residues of SYN545192 and its Metabolite SYN546206 in Soil: Method Validation. Project Number: TK0105197/OCR, S11/03924. Unpublished study prepared by Eurofins Analytick GmbH, EPA MRID No 48604558.

*ILV:* None

**Document No.:** MRIDs 48604560 & 48604558

**Guideline:** USEPA 850.6100  
PMRA 8.2.2.1

**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 (p. 3).

**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. 48604560 and 48604558). Overall mean recoveries of SYN546246 were less than 90 percent in all cases from clay loam soil and silt loam soils. However, overall mean recoveries were always above 87 percent. Overall mean recoveries of CSCD465008 and CSAA798670 from loamy sand soil was less than 90 percent in all cases, but always greater than 77 percent in the initial validation of the method.

**PC Code:** 122305

**Reviewer:** Gabe Rothman  
Environmental Scientist

**Signature:**



**Date:** August 29, 2013

## **Executive Summary**

10 g sub samples of soil are extracted by reflux in 80/20 v/v acetonitrile/ultra-pure water. After cooling to room temperature, extracts are decanted and centrifuged. Aliquots of the soil extracts are diluted with 50/50 v/v acetonitrile/ultra-pure water for direct injection analysis.

Alternatively, aliquots may be diluted with ultra-pure water and taken through a solid phase extraction procedure. Final determination is by high performance liquid chromatography with triple quadrupole mass spectrometric detection (LC-MS/MS).

The limit of quantification of the method is 0.001 mg/kg (0.001 ppm, 1 ppb).

## I. Recovery Findings

### Initial Validation of Method (Tables 2 – 5 from Study Report, MRID No. 48604560)

**Table 2 :** SYN545192 Recovery Data.  
Primary Transition  $m/z = 396 \rightarrow 368$

Soil Type	Fortification Level (mg/kg)	Recovery (%)**	n	Mean (%)	RSD (%)	Range (%)
Silt Loam	0.001*	88, 106, 95, 89, 92	5	94	7.7	88-106
	0.01	114, 112, 104, 109, 62#	4	110	4.0	104-114
		Overall	9	101	10	88-114
Clay Loam	0.001*	90, 88, 92, 104, 103	5	95	7.9	88-104
	0.01	72, 88, 74, 66, 84	5	77	12	66-88
		Overall	10	86	15	66-104

\*Limit of quantification, defined by the lowest validated fortification level

\*\*Residues in control samples and reagent blanks were less than 30% of the LOQ.

# outlier according to Grubbs test, not taken into account for statistical calculation

**Table 3 :** SYN545192 Recovery Data.  
Confirmatory Transition  $m/z = 396 \rightarrow 91$

Soil Type	Fortification Level (mg/kg)	Recovery (%)**	n	Mean (%)	RSD (%)	Range (%)
Silt Loam	0.001*	98, 108, 91, 116, 119	5	106	11	91-119
	0.01	118, 118, 108, 116, 64#	4	115	4.1	108-118
		Overall	9	110	9.0	91-119
Clay Loam	0.001*	118, 81, 96, 106, 88	5	98	15	81-118
	0.01	71, 94, 76, 67, 81	5	78	14	67-94
		Overall	10	88	18	67-118

\*Limit of quantification, defined by the lowest validated fortification level

\*\*Residues in control samples and reagent blanks were less than 30% of the LOQ

# outlier according to Grubbs test, not taken into account for statistical calculation

**Table 4 :** SYN546206 Recovery Data.  
Primary Transition  $m/z = 382 \rightarrow 342$

Soil Type	Fortification Level (mg/kg)	Recovery (%)**	n	Mean (%)	RSD (%)	Range (%)
Silt Loam	0.001*	80, 83, 79, 82, 85	5	82	2.9	79-85
	0.01	87, 89, 84, 85, 48#	4	86	2.6	84-89
		Overall	9	84	3.8	79-89
Clay Loam	0.001*	81, 67, 89, 84, 84	5	81	10	67-89
	0.01	73, 77, 69, 73, 77	5	74	4.5	69-77
		Overall	10	77	9.2	67-89

\*Limit of quantification, defined by the lowest validated fortification level

\*\*Residues in control samples and reagent blanks were less than 30% of the LOQ.

# outlier according to Grubbs test, not taken into account for statistical calculation

**Table 5 :** SYN546206 Recovery Data.  
Confirmatory Transition  $m/z = 382 \rightarrow 362$

Soil Type	Fortification Level (mg/kg)	Recovery (%)**	n	Mean (%)	RSD (%)	Range (%)
Silt Loam	0.001*	86, 86, 74, 78, 84	5	82	6.6	74-86
	0.01	86, 90, 82, 85, 49#	4	86	3.9	82-90
		Overall	9	83	5.8	74-90
Clay Loam	0.001*	81, 72, 89, 87, 84	5	83	8.1	72-89
	0.01	72, 79, 71, 74, 78	5	75	4.8	71-79
		Overall	10	79	8.3	71-89

\*Limit of quantification, defined by the lowest validated fortification level

\*\*Residues in control samples and reagent blanks were less than 30% of the LOQ

# outlier according to Grubbs test, not taken into account for statistical calculation

## **II. Method Characteristics**

### **Extractability**

The stability of the sample extracts of SYN545192 and its metabolite SYN546206 fortified at the LOQ level was checked after a storage period of at least seven days in a refrigerator at 3-8 °C against freshly prepared calibration standards.

### **Specificity**

LC-MS/MS as a detection technique with two transitions is considered to be highly specific and therefore according to the guidance (see guidance section of this summary) further confirmation is not required. No significant interferences arising from the soil matrices, the lab ware, reagents or solvents tested have been observed at the retention time of interest for any of the analytes.

### **Linearity**

For accurate quantification of residue concentrations, analyses should be carried out within the linear range of the detector. For multi-point calibration, detector range and linearity will be demonstrated within each sample set.

The linearity of the LC-MS/MS detector response for SYN545192 and SYN546206 was tested in the range from 1.5 pg to 250 pg injected on column (equivalent to 0.03 ng/mL to 5.0 ng/mL standards when using a 50 µL injection volume) and was found to be linear. If a residue beyond the tested concentration range is expected, dilute the sample appropriately to bring it within the tested linear range prior to quantification.

### **Accuracy and Precision**

The overall mean recoveries in the stored fortified samples were within the acceptable range of 70-120% with a RSD of  $\leq 20\%$  for both soil types.

### **Limit of Quantification**

The limit of quantification of the method is 0.001 mg/kg (0.001 ppm, 1 ppb).

### **Matrix Effect**

Significant matrix effects (signal enhancement) were found for both soil types. For the validation, matrix-matched standards were therefore used for quantification.

### **Final Extract Stability**

Final soil extracts in 50/50 v/v acetonitrile/ultra-pure water retained in vials and stored at a temperature of approximately 4°C were suitable for SYN545192 and SYN546206 residue analysis, for storage periods of up to 28 days.

### **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. 48604560 and 48604558).
2. Overall mean recoveries of SYN545192 were less than 90 percent in all cases from clay loam soil and silt loam soils. However, overall mean recoveries were always above 87 percent.
3. Overall mean recoveries of SYN546246 from loamy sand soil was less than 90 percent in all cases, but always greater than 77 percent in the initial validation of the method.

### **IV. References**

1. Braid, S (2012). SYN545192 - Analytical Method GRM042.05A for the Determination of SYN545192 and its Metabolite SYN546206 in Soil. Syngenta Ltd., Jealott's Hill International Research Centre, Bracknell, UK. GRM042.05A.