2 STUDY OBJECTIVE

To validate the Syngenta residue analytical method Draft GRM030.02A: 'NOA449280: Residue Method for the Determination of NOA449280 and Metabolite SYN503780 in soil', in 2 different characterised soil types.

3 MATERIALS / TEST SYSTEM

3.1 Specimen Receipt

Specimen Identifier CSR	Specimen Type	Origin
3544-002	Standard soil 2.3	LUFA-Speyer
3544-003	Standard soil 6S	LUFA-Speyer

3.2 Specimen Preparation

No specimen preparation was necessary for the soils. Both specimens were stored frozen and will be disposed of on completion of the analysis.

3.3 Reference / Test Items

Identity:	**NOA449280**
Reference:	AMS 1144/1
Purity:	99.9%
Expiry date:	31 October 2007
Storage:	Ambient

Identity:	**SYN503780**
Reference:	KI 6386/18
Purity:	100%
Expiry date:	31 May 2008
Storage:	Refrigerator

Full details of these materials are included in the raw data package for the study along with all analytical and fortification standards prepared from the primary reference items. The reference items will be retained until expiry and then disposed of.

4 METHOD

4.1 Fortification of Specimens

Control specimens were fortified as detailed below:

Matrix	Reference Item	Untreated Replicates	Replicates at Fortification Level (LOQ)	Replicates at Fortification Level (10 x LOQ)
Soil	NOA449280 SYN503780	2	5 at 0.001 mg/kg	5 at 0.01 mg/kg

4.2 Methods of Analysis

The specimens were extracted and analysed according to the Syngenta residue analytical method Draft GRM030.02A: 'NOA449280: Residue Method for the Determination of NOA449280 and Metabolite SYN503780 in soil'.

Representative samples of soil (10 g) were extracted by shaking and centrifuging in 0.05 M ammonium hydroxide solution (20 mL). This extraction was repeated with 0.05M ammonium hydroxide solution:acetone 50:50 v/v (20 mL), followed by a similar extraction procedure in acetone with all supernatants being combined. Aliquots were filtered and diluted with water; final sample concentration was 0.04 g/mL. Final determination was by high performance liquid chromatography with triple quadrupole mass spectrometric detection (LC-MS/MS).

Characterisation of the soil specimens for sand content, silt content, clay content and organic matter was carried out by NRM Ltd. Soil samples were air dried at ≤30 ºC, then rolled or sieved to pass a 2 mm screen. Sub-samples were then ground to pass a 0.5 mm screen.

The specimens were analysed for sand fraction, silt fraction, clay fraction and textural classification according to NRM SOP JAS-096 "Determination of Particle Size Distribution in Soil – Pipette Method – Three Fractions". The specimens were analysed for organic matter according to NRM SOP JAS-093 "Determination of Organic Matter in Soil by the Walkley Black Wet Oxidation Method".

Characterisation of the soil specimens for pH was carried out by CEMAS in accordance with SOP CEM-3089 "Determination of Soil pH".

The NRM characterisation data is stored in Study Number CEMS-3544.

Stability of the final extracts in vials was assessed for 1 soil matrix (Standard Soil 2.3) fortified at LOQ, after storage in a refrigerator for a period of 10 days. Re-analysis was performed against a freshly prepared calibration standard using the primary transition only.