Extraction of Soil for the Determination of Pesticides

SpeedExtractor E-916, Syncore® Analyst
Extraction of Soil using SpeedExtractor E-916 for the Determination of Pesticides

This application note describes a fast and reliable way to extract organochlorine pesticides (OCP) from soil with the SpeedExtractor E-916. After reducing the volume with the Syncore® Analyst the extracts were cleaned by using Florisil. The cleaned extracts were concentrated again and quantified by GC-ECD.

1. Introduction

A pesticide is any substance or mixture of substances intended for, preventing, destroying, repelling, or mitigating any pest. There are three main types of pesticides: organochlorine, organophosphate and carbamates. OCP can cause severe health problems, are persistent and tend to bio-accumulate. Therefore the use of some OCP is banned by the Stockholm convention.

2. Experimental

Instrumentation: SpeedExtractor E-916 with 20 mL cells, Syncore Analyst, Thermo Trace GC Ultra

Samples: CRM 847-050 and CRM 804-050 by R.T. Corporation.

Depending on the expected values of the OCP in the samples 1 to 5 g of sample were weighed-in and mixed with sand. The mixture was transferred to the cell and two surrogates were added prior to the extraction. The cells were extracted using the parameters in Table 1. 150 mL Syncore® vessels with appendix were used as collection vials.
After the extraction, 1 mL of Internal Standard (IS) was added to the extracts. After concentration on Syncore® Analyst a clean-up with Florisil was performed. The volume of the cleaned solution was reduced again and quantification of 16 different pesticides was performed by GC-ECD.

A fourfold extraction of the samples was done. Two blanks were extracted in parallel on two positions not used for samples. In addition post-extractions of the samples were to show the performance of the method.

Table 1: Method SpeedExtractor E-916

Temperature	100°C
Pressure	100 bar
Solvent	Cyclohexane 50%, Ethyl acetate 50%
Cells	20 mL
Vials	150 mL Syncore with 1 mL appendix
Cycles	3
Heat-up	1 min
Hold	10 min
Discharge	2 min
Flush with solvent	3 min
Flush with gas	2 min

3. Results

The measurements should be within the prediction interval (P.I.) at least 19 out of 20 times. Out of the 22 quantified values only one value was above the P.I. All pesticide values in the blanks were below the limit of quantification.

Figure 1 and 2 show the results of a few selected pesticides. Error bars of CRM represent the prediction intervall; error bars of SpeedExtractor represent the rsd (n = 4). For detailed results see Application Note 067/2011

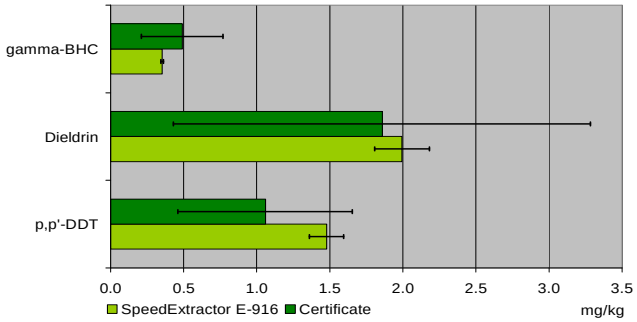

Figure 1: Results for CRM 804-050

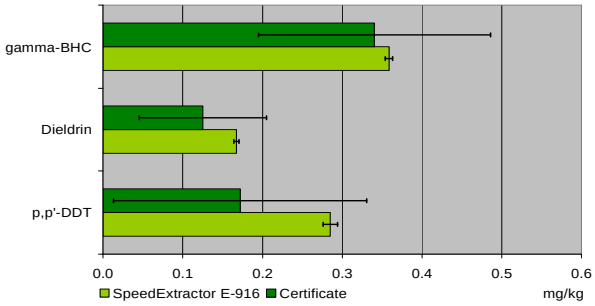

Figure 2: Results for CRM 847-050

4. Conclusion

The method presented in this application note demonstrates a fast and reliable way to extract pesticides from soil using the SpeedExtractor E-916.

5. Ackowledgement

We sincerely thank Bachema AG, Schlieren, Switzerland for the analytical work.

6. References

For more details see Application Note 067/2011