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Certified Reference Materials for the determination of mineral oil hydrocarbons in water, soil and waste

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The international research project HYCREF, funded by the European Commission in the 5th Framework programme, aimed to develop methods to prepare homogenous and stable water-, soil and waste reference materials contaminated with mineral oil hydrocarbons and to test certify the mineral oil content by gas chromatographic methods.

As mineral oil products are important sources for environmental contaminations a high need exists for certified reference materials for their determination using the new gas chromatographic methods (soil: ISO/FDIS 16703, waste: ENpr 14039, water: ISO 9377-2).

The experimental conditions and results for preparation and characterisation of a total of nine reference materials (3 water-, 3 soil- and 3 waste materials) are described and discussed. Target values for the reference materials were defined at the beginning of the project in order to have clear quality criteria, which could be compared with the achieved results at the end of the project. These target specifications were related to the maximum uncertainty from test certification exercises (< 5 % for soil/waste and < 10 % for water), the maximum inhomogeneity between bottles (< 3 %) and minimum requirements for stability (> 5 years for soil/waste and > 2 years for water). The feasibility studies showed that solid materials (soil, waste) could be prepared sufficiently homogenous and stable. The test certified values of the 6 solid materials comprise a wide range of mineral oil content from about 200-9000 mg/kg with expanded uncertainties between 5.7-13.1 % using a coverage factor k ($k = 2$). The development of new water reference materials –the so-called “spiking pills” for an offshore- and a land-based discharge water represents one of the most innovative aspects of the project. The spiking pill technology facilitates the application and storage and improves the material stability compared with aqueous materials.

Additional to the preparation and test certification of the reference materials investigations on the analytical method for the determination of mineral oil hydrocarbons were performed. The results obtained in relation to the optimisation of analytical method (extraction procedure, sample pretreatment, clean-up and measurement) were provided to the respective working group of ISO/TC 190, ISO/TC 147 and CEN/TC 292 and were incorporated into the ongoing standardisation procedures.

The new version of ISO/FDIS 16703 (July 2004) includes the improvements based on HYCREF results, for example the increase of the solvent/sample ratio, the removal of acetone from the organic extracts and the use of column technique instead of batch technique for clean-up.