

REQUIREMENTS ON THE HORIZONTAL STANDARDISATION OF THE PRETREATMENT OF DIFFERENT SOLID SAMPLES CONCERNING THE PHTHALATES

Sampling

The use of glass containers is indispensable, the samples should be protected from contamination by the caps by aluminium foil. The cleaned containers should be heated to 250 °C (or 400 °C if possible, as recommended for analytical glassware) and rinsed with solvent (also in order to deactivate the surface). Cleaned metal containers are also appropriate for phthalates though not for all organic analytes because of possible catalytic effects (see desk study of M. Gfrerer, 4.1).

Drying

According to CEN/TC 308/WG 1/TG 4 N0058 the samples should contain at least 85 % dry matter for the drying with sodium sulphate. Sodium sulphate is appropriate for the phthalates if it is pre-treated in the usual manner.

Samples with low water content may be air-dried, but contamination is to be avoided. Drying in an oven at 105 °C is not advisable because DBP will be lost partly, the drying at 35 °C was not tested.

In the case of the phthalates small amounts of water do not disturb the analysis. Comparative extractions of sewage sludge, that had been stored at ambient conditions in the laboratory, with and without the addition of sodium sulphate gave no significantly differing results. Samples with less than 85 % dry matter should be freeze dried. At the end of the drying process care should be taken that the sample is cooled as long as it is under vacuum, in order to avoid losses of more volatile phthalates.

For samples that contain free water it is recommended to decant it, if losses of phthalates with a relatively high water solubility occur has not been tested.

Homogenisation

If the samples are only slightly humid and finely crumbling, they may be directly sieved to the required size and homogenised.

In the case of sewage sludge and other liquid samples the homogenisation should be done preferably before the drying process by intense shaking. Lyophilised samples have to be ground in a mill or mortar. Some times this may be difficult when the freeze dried sewage sludge is forming a kind of fibre mat.

Sieving: see desk study M. Gfrerer, 4.6

Sub-sampling: see desk study M. Gfrerer, 4.7

Storage

It is generally accepted that phthalates are stable in dry samples at 4 °C for longer times.

Dry samples are best stored in glass vessels whose (preferably PTFE-lined) caps are separated from the sample by aluminium foil, or in metal containers which are cleaned in an appropriate manner.

Due to the ubiquitous occurrence of phthalates all containers have to be protected rather against contamination than against loss of analyte.

Aqueous samples or samples with a high water content are stored frozen in glass containers. After thawing these samples the previous structure may be lost, e.g. sewage sludge may flocculate.

Conclusion

To our opinion the pre-treatment scheme in the desk study of M. Gfrerer is applicable for phthalates and the mentioned matrices as long as the samples come into contact only with glass, PTFE, or metal. Special measures against evaporation losses of phthalates are not necessary if extreme conditions like higher temperatures or low pressure at ambient temperature are avoided.