

# SUMMARY COMMENTS AND STATUS OF HORIZONTAL DS 18. DIGESTION

## RECOMMENDATIONS FROM THE STEERING COMMITTEE

The Steering Committee **agreed to proceed to Phase II.**

In order to avoid confusion with a digestion method based on HF (prohibited in certain Member States for health and safety reasons, but needed for specific research needs) or other true total method, it was agreed to specify that *aqua regia* or nitric acid digestion might not necessarily release elements completely from the solid matrices.

## SUMMARY OF COMMENTS

### HORIZONTAL DESK STUDY 18. DESK STUDY ON DIGESTION METHODS

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#### **Choice of digestion methods**

The desk study concerns an evaluation of potential for horizontal standardisation for the digestion of samples prior to the determination of trace elements covering sludge, soil, treated biowaste and neighboring fields.

The detailed comparison of standards for the digestion prior to the determination of trace elements demonstrates the possibility of preparing horizontal standards in this field, however investigations has to be initiated prior to the establishment.

From an environmental impact evaluation perspective the composition derived from either "partial" or "total" digestion is hardly relevant, as the fraction which even under extreme environmental condition will be brought into solution (and made available for plants etc.) is several orders of magnitude lower. Thus for control purposes or for monitoring or evaluation of developments in element concentration, the use of "partial" methods are considered acceptable. However, it is of vital importance that the methods provide comparable results with a sufficiently high reproducibility for the elements and matter in question.

In many European countries, digestion methods used for solid environmental samples such as waste, sludge and soil are based on the use of aqua regia in accordance with the relevant European and International standards for the different areas. However, in some European countries, e.g. the Nordic countries, the digestion methods are based on the use of nitric acid. The reason for this latter choice being the smaller interference effects from using nitric acids on some of the subsequent analytical procedures, e.g. ICP-MS, which is

nowadays in some countries and may be in future one of the predominant subsequent equipment to be used for the determination step.

There is no evidence that the two digestion principles give significantly different results. It is recommended that the comparability of the digestion methods be investigated in a coming step of the project.

### **Aqua regia digestion methods**

One major difference in the existing standards on aqua regia is identified. The ISO standard on aqua regia digestion of soil and the EN standard on digestion of soil improvers and growing media include only digestion by the use of hot-plate heating, while the EN standards on digestion of waste and sludge also include methods for microwave heating.

To take into account the developments that have taken place in many European laboratories, the development of a harmonised horizontal standard that includes both hot-plate heating and microwave oven heating (open and closed heating) is proposed. Data exist for waste and sludge samples that suggest that comparable data can be obtained, but for other matrices more investigations are needed.

A draft horizontal standard has been described. Sediments, soil, sludge and waste are proposed to be included in the final scope of the horizontal standard.

### **Nitric acid digestion method**

Autoclave methods exist in the Nordic countries covering sludge and sediment (and water) and the same standards are generally prescribed for soil and biowaste. An equivalent method exists in Sweden covering soil samples. Microwave-assisted heating of nitric acid digests is validated as a US EPA method.

A draft horizontal standard covering nitric acid digestion in an autoclave equivalent to the existing autoclave heating method has been described. It is proposed that sediments, soil, sludge and waste be included in the final scope of the horizontal standard.

It is suggested that investigations on the comparability of autoclave heating and microwave-assisted heating be carried out for consideration of a possible inclusion of microwave heating in the final standard. Inclusion of the conditions given in the US EPA methods is suggested. One advantage to be mentioned is the high throughput enabled by the high temperature and short heating period.

### **Sample pretreatment**

Comparability of data is of high importance. An investigation of the optimum particle size of a test sample is needed for soil, sediment and biowaste for both digestion methods. Directions on sample pretreatment are not included in the proposed standards. It is proposed to develop a horizontal standard on pre-treatment.