

SUMMARY COMMENTS AND STATUS OF HORIZONTAL DS 20. AAS

RECOMMENDATIONS FROM THE STEERING COMMITTEE

The Steering Committee suggested discussing the interference problems of ICP with certain types of soils rich, for instance, in tin or iron. It was agreed to add flame AAS and the determination of mercury by atomic fluorescence to this Work Package, if necessary by increasing the budget of €4,000 to cover the extra costs. **The revised desk study with these integrations should be submitted by March 2004.**

Concerning Phase II, it was decided to merge experimental works on digestion and measurement with ICP and AAS as both aspects are linked. A detailed programme on what will be performed for these investigations should be previously sent to the Steering Committee.

SUMMARY OF COMMENTS

HORIZONTAL DESK STUDY 20.TRACE ELEMENTS DETERMINATION - AAS

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General

Under the clause "Deliverables" in the contract for the desk study it is indicated that the desk study should include

- determination of trace elements --- by AAS graphite furnace.
- determination of mercury --- by AAS cold vapour technique.
- determination of arsenic --- by AAS hydride technique.

As the contract underlined that these methods should be included in the study, I decided to keep strictly to these ones only, in spite of the fact that I know that there are many other methods being fully applicable for the determinations of these elements. Because of the general trend in the comments received, this was obviously a wrong decision, and other, related methods have to be included in the draft horizontal standards.

Trace metals

Several comments on why the flame AAS is not included in the draft were received, because some of the elements of interest will be present in rather high concentrations in several samples (e.g. zinc, iron, manganese, aluminium, etc). In these cases the graphite furnace method is far too sensitive, and the samples have to be diluted extensively. For

such samples it is preferable to use a less sensitive method, and the flame AAS is a good alternative. It is therefore suggested that the draft horizontal standard is extended, and is describing the use of flame AAS and graphite furnace AAS in two separate parts of the standard.

The draft standard may also include more elements than in the first proposal, and the working ranges and detection limits should be more clearly adapted to the soil and waste extracts.

Mercury

For this element some comments have been given on why the atomic fluorescence spectrometric method is not included in the draft. This technique is used by several laboratories, the method is standardized and the performance of the method is thus well documented. It is therefore suggested that the AFS technique is included in the draft standard as a separate part of the draft.

The traditional reduction of mercury compounds to elemental mercury is done by addition of stannous chloride solution to the sample. However, sodium borohydride is used by several laboratories, and the method itself is also described in a standard. Because some comments express a clear wish to include this reduction agent in the method, I suggest that it is included in the draft standard.

Arsenic

In some comments there is pointed out that there is already existing a draft standard for the determination of arsenic in digests (e.g. ISO DIS 20280). This method may be used as a basis for a draft horizontal standard, or even used as it is. Performance data for both the soil and waste extracts must be included.

Recommendations

- 1 The draft horizontal standard for the determination of trace metals is extended to include the flame AAS as well as the graphite furnace AAS, the two methods described in separate sections of the draft.
- 2 The draft horizontal standard for the determination of mercury is extended to include the atomic fluorescence technique, the two methods being described in separate parts of the draft. In both cases the use of sodium borohydride as reduction agent is included in addition to the stannous chloride.
- 3 The draft international standard ISO DIS 20280 is used as a basis for the preparation of a draft horizontal standard for the determination of arsenic by the hydride technique.