

# SUMMARY COMMENTS AND STATUS OF HORIZONTAL DS 16 NUTRIENTS

## RECOMMENDATIONS FROM THE STEERING COMMITTEE

The Steering Committee was satisfied with the work done on nutrients and suggested **proceeding to Phase II**.

A long discussion followed the question by the Work Package Leader, Mrs Kirsten Andersen, whether a method for soluble phosphorus (and nutrients) was needed (not in the contract specifications). In the end, it was agreed that for policy purposes it was indeed useful to have a reliable method for measuring this important parameter. This latter issue was linked with leaching. It was agreed to ask the team to **address this aspect by March 2004** in a revised version of the desk study.

## SUMMARY OF COMMENTS

### HORIZONTAL DESK STUDY 16. DETERMINATION OF TOTAL PHOSPHORUS, TOTAL NITROGEN AND NITROGEN FRACTIONS

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#### **Summary of the comments and the responses**

Working with the desk study 16 the national working groups and mirror committees were asked for information about standards in their countries. The only information was that the CAT-method is applied in the Nordic countries. This situation made the working out of the desk study difficult.

Task of the desk study 16 was to describe and assess the existing international standards or draft standards. It should show if the existing standards could be developed to Horizontal standards. The assessment of the existing standards could mainly be done referring to the analytical point of view. Standards of total N, (Kjeldahl, Dumas), total P and N-fractions should be proposed.

Here the comments to the desk study are summarised according to the mail from the 23.10.2003.

Kjeldahl-N with and without nitrate and nitrite: It is agreed to have a Kjeldahl standard for all materials. One comment refuses the standard including nitrate and nitrite. The homogeneity, the amount of test sample and the volume of Kjeldahl tubes are points of

discussion in the comments. Two draft standards are formulated and have to be discussed in the CEN TC's.

Total N (Dumas): The comments agree with this standard. A problem may be the sample pretreatment, especially the drying of wet samples with low dry matter content without losses of N and the homogeneity of the test samples. A draft standard is formulated and has to be discussed in the CEN TC's.

Total Phosphorus: It is agreed to use the aqua regia extract for the extraction of total phosphorus. Some comments asked for the Nordic method using nitric acid as the extractant. One other comment says, they would agree with the aqua regia extract if microwave heating is allowed. This is discussed in chapter 3.3.3. The available data for this test allow this modification of the aqua regia extract for phosphorus. Some comments propose to integrate the determination of total phosphorus into the desk study 18, I agree with this proposal. The aqua regia extraction is not only to determine P but also to determine a lot of other elements. If this is followed, the microwave digestion is not validated for some elements, especially in comparison with the extraction in a reflux system (see appendix EN 13656 and EN 13657), the validation data are not satisfactory.

There is a comment on the measuring method ICP-OES. It is not agreed that this method is the reference method.

I can't accept, that this wide spread and multielement technique should not be the reference method. No one is prevented from doing the molybdenum blue method, he has to prove the comparability of results with the ICP-OES method. A draft standard is formulated and has to be discussed in the CEN TC's.

N-Fractions: The extraction of materials with 2 mol/l of KCl is accepted except the TC 223. They demand water and the CAT-solution as extraction solution. These two methods allow the interpretation of results for the purpose of fertilisation, they are not used for waste, sludge and soil to determine the N-fractions, especially for fertilisation. To have a standard for all materials and an information of the „total content“ of N-fractions, is the draft standard formulated to use 2 mol/l KCl as extractant. This is a standard to cover all materials. To use water or CAT in the fields of soil improvers, growing media and biowastes, the scope of this standards has to describe the limitation and the description of the working area has to be limited to the special purpose. There is a different comment saying that for soil improvers the KCl-method is agreed, for growing media it is rejected. A draft standard is formulated and has to be discussed in the CEN TC's.

There is a demand to have standards to differentiate phosphorus (P-fractions). This is discussed in chapters 1.2.4, 3.1.6, 3.4 and 4.3. One comment agrees with the proposal to have an intensive discussion of experts on the topic of P-fractions before formulating a draft standard. There are a lot of methods to extract soils in behalf of fertilisation. These methods do not seem to be suitable for other materials and other fields of investigation. To have a method without satisfactory validation data is not the aim of Horizontal.

The main work to do in the future to make the draft standards to Horizontal standards is the work of validation as described in chapters 1.3, 3.3 and 4 in detail. The results of this validation work will define the working range, the detection limit and the precision (repeatability, reproducibility and accuracy). The implementation of the interlaboratory tests should be planned carefully.

The throughput depends on the laboratory staff, the analytical instruments and equipment. All proposed methods allow a high throughput and they can work automatically in steps or in whole (N-Dumas, N-Kjeldahl, ICP-OES, flow injection analysis).

The evaluation of the whole project „Horizontal“ and especially about the desk study 16 is very different. The predominant opinion is to continue the work of the project and that of the desk study 16.