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AMENDMENT 1
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Fertilizers and soil conditioners — Determination of humic and hydrophobic fulvic acids concentrations in fertilizer materials

AMENDMENT 1: DAX-8 resin alternative

Engrais et amendements minéraux basique — Détermination des acides humique et fulvique

AMENDEMENT 1: Alternative à la résine DAX-8

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This document was prepared by Technical Committee ISO/TC 134, *Fertilizers, soil conditioners and beneficial substances*.

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Fertilizers and soil conditioners — Determination of humic and hydrophobic fulvic acids concentrations in fertilizer materials

AMENDMENT 1: DAX-8 resin alternative

4.3

Replace paragraph 4.3 with the following:

The liquid supernatant remaining after the removal of the HA is called the fulvic fraction. The fulvic fraction, which can contain hydrophobic fulvic acids (HFA), is treated to determine the quantity of HFA in the fulvic fraction by selective adsorption onto a macroporous polymeric adsorbent resin. The substances retained by the resin are referred to in the literature as the hydrophobic acid fraction of soluble organic matter[3].

6.7

Replace paragraph 6.7 with the following:

Methacrylic-ester resin, 20–60 mesh, 0,75 ml·g^{-1} to 1,15 ml·g^{-1} pore volume, mean pore size of 225 Å to 600 Å, 140 m^2·g^{-1} to 550 m^2·g^{-1} surface area, for adsorption of materials up to 60 000 MM, e.g. Supelite DAX-8 Resin, or Amberlite XAD-7HP, or any other available resin meeting equivalent properties.

7.18

Replace paragraph 7.18 with the following:

Glass chromatography column, 4 cm × 25 cm for the adsorbent resin.

11.1

Replace the first sentence in 11.1 with the following:

Hydrophobic fulvic acids (HFA) are separated from other acid-soluble substances in the fulvic fraction by selective adsorption to a macroporous adsorbent resin (i.e. DAX-8 or Amberlite XAD-7HP), to which hydrophilic acid-soluble components do not bind and can thus be removed from the solution.

11.1

Add the following sentence at the end of 11.1:

Annex C provides supporting data comparing alternative resins used for HFA separation.

Annex C

Add the following Annex after Annex B, before the Bibliography:

Annex C
(informative)

Resin comparison laboratory study

C.1 General

Currently, the ISO 19822 method for determining humic and hydrophobic fulvic acids concentrations involves separating the hydrophobic fulvic acids (HFA) fraction from the fulvic fraction as per Clause 11. The separation of HFA involves selective adsorption to an acidified hydrophobic resin that does not bind hydrophilic acid-soluble components. The standard resin widely employed across research and industry for HFA separation is the Supelite DAX-8 resin, which has been discontinued by the manufacturer. The purpose of this resin comparison is to identify a suitable alternative, as DAX-8 is no longer commercially available for purchase.

C.2 Properties of the two resins tested

The DAX-8 and XAD-7HP resins possess physical properties suitable for adsorbing hydrophobic fulvic acids. The DAX-8 resin is slightly more polar than the XAD-7HP. The molecular mass capacity differs between the resins, with DAX-8 cited as having a capacity of up to 150 000 and XAD-7HP up to 60 000. DAX-8 has a surface area of 140 $m^2\ g^{-1}$, while XAD-7HP has a larger surface area of 520 $m^2\ g^{-1}$. The particle diameter for DAX-8 is 250 µm to 420 µm and 250 µm to 841 µm for XAD-7HP. The mean pore size for DAX-8 is 225 Å, whereas it is approximately 350 Å for XAD-7HP, and pore volumes are 0,79 ml g^{-1} and 0,50 ml g^{-1}, respectively.

It should be noted that XAD-7 was improved to create XAD-7HP, which has a broader pH application range, making it suitable for this application.

C.3 Multiple laboratory study

The purpose of the laboratory study was to evaluate the comparative performance of XAD-7HP resin as an alternative to DAX-8 resin for separating hydrophobic fulvic acids from the fulvic fraction as per Clause 11. The study design was based on previous experiments performed to discover alternative resins.[9][10] Three laboratories generated three replicated analyses for HFA on three different materials.

C.4 Materials

Materials traceable to their vetted sources, with varying concentrations and chemical compositions of humic substances, were chosen for quantitative analysis using the two different resins. The selected materials were:

— QCM 1.1, a quality control material prepared using best manufacturing practices, which is the same material used as the quality control material in the development of this document;

— PPS, Pahokee Peat soil reference material obtained from the International Humic Substances Society (IHSS) has a higher degree of heterogeneity than QCM 1.1, as it is derived from partially humified peat;

— GL, Gascoyne leonardite reference material, also sourced from IHSS, has a higher degree of heterogeneity than the QCM 1.1.

C.5 Methods

The materials were analysed for hydrophobic fulvic acid (HFA) content using the method described in this document. The precision of the data was evaluated in accordance with ISO 5725-2. Since the data were non-parametric, correlation analysis was conducted using Spearman's rank correlation coefficient (r_s) to compare the data generated by each resin. Raw data and statistical analysis for QCM 1.1 is displayed for DAX-8 (Table C.1), XAD-7HP (Table C.2), and QCM 1.1 resin correlation (Table C.3). Raw data and statistical analysis for material GL is displayed for DAX-8 (Table C.4), XAD-7HP (Table C.5), and GL resin correlation (Table C.6). Raw data and statistical analysis for material PPS is displayed for DAX-8 (Table C.7), XAD-7HP (Table C.8), and PPS resin correlation (Table C.9).

C.6 Results of quantitative analysis

C.6.1 Results for Material QCM 1.1