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Textiles — Determination of stable nitrogen isotope ratio in cotton fibres

Textiles — Détermination du rapport isotopique stable de l'azote dans les fibres de coton

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Foreword

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This document was prepared by Technical Committee ISO/TC 38, *Textiles*.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

Nitrogen (N) is the most essential element for cotton fibre production, since cotton crops require large amounts of N to sustain their growth and productivity. For this purpose, considerable amounts of nitrogen are applied to the soil as chemical fertilizers (e.g. urea) or organic fertilizers (e.g. composted manure).

The nitrogen applied in the form of a chemical fertilizer or a composted manure is absorbed from the soil to cotton crops through their root systems during cultivation, leaving a fertilizer-specific nitrogen isotopic signature in cotton tissues (particularly in cotton fibres). The inerasable nitrogen isotopic fingerprint engraved in cotton fibres remains unchanged during manufacturing of yarns, textiles and fabrics.

This document employs the principle of nitrogen isotope discrimination that fractionates against ^{15}N (heavier nitrogen isotope), resulting in ^{15}N-enriched reactants and ^{15}N-depleted products. In general, composted manure has a much higher $\delta^{15}N$ (natural ^{15}N abundance, defined hereinafter) than chemical fertilizer due to faster NH_3 (ammonia) volatilization of the lighter nitrogen isotope (^{14}N) than ^{15}N during the composting process. In contrast, chemical fertilizers (e.g. urea) are produced from the atmospheric N_2 via the Haber-Bosch process, resulting in low $\delta^{15}N$ values close to atmospheric N_2, which means that similar isotopic nitrogen compositions of chemical fertilizers to the atmospheric N_2.

Organic farming strictly bans the use of genetically modified crops (GMO), synthetic pesticides, and chemical fertilizers. Therefore, the use of chemical fertilizer during organic cotton production can be detected by determining $\delta^{15}N$ of cotton fibres, since the difference in $\delta^{15}N$ values between the two isotopically different nitrogen inputs (chemical vs. organic fertilizers) leaves a fertilizer-specific isotopic fingerprint in cotton fibres that can thereafter provide a forensic evidence for organic cotton fibre production.

Textiles — Determination of stable nitrogen isotope ratio in cotton fibres

1 Scope

This document specifies the determination of the ratio of nitrogen isotopes in cotton fibres that are used for textile production. It applies not only to cotton textiles but also to raw cotton taken from cotton fields.

2 Normative references

There are no normative references in this document.

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

3.1
stable isotope
atom whose nucleus contains the same number of protons but a different number of neutrons

Note 1 to entry: Stable isotopes do not decay into other elements, while radioactive isotopes are unstable and will decay into other elements.

3.2
isotope-ratio mass spectrometry
IRSM
specialization of mass spectrometry, in which mass spectrometric methods are used to measure the relative abundance of isotopes in given sample

Note 1 to entry: The analysis of isotopes is normally related to measuring isotopic variations caused by mass-dependent isotopic fractionation in natural systems.

3.3
isotopic fractionation
isotopic discrimination
process that affect the relative abundance of isotopes

Note 1 to entry: Normally, the focus is on *stable isotopes* (3.1) of the same element. Isotopic fractionation in the natural environment can be measured by isotope analysis, using isotope-ratio mass spectrometry, to separate different element isotopes on the basis of their mass-to-charge ratios.

Note 2 to entry: Both heavy and light stable isotopes participate freely in biochemical reactions and in geochemical processes, but the rate at which heavy and light stable isotopes react differs. As a result, the lighter isotopes react faster than the heavier isotopes leading to isotopic fractionation between reactant and product in the reactions.

3.4
natural ^{15}N abundance
relative amount of the isotopes of nitrogen, as it occurs in nature

Note 1 to entry: Natural ^{15}N abundance is expressed in parts per thousand (‰, per mil) deviations from the atmospheric N_2 (a standard).

4 Principle

Isotope-ratio mass spectrometry (IRMS) measures the relative abundance of stable isotopes (e.g. ^{14}N and ^{15}N for nitrogen) in a cotton fibre sample. The abundance of stable isotopes varies according to physical, physiological and biochemical isotopic fractionation processes. Consequently, variations in the isotopic ratio of nitrogen (i.e. $^{15}N/^{14}N$) can be used both as fingerprints of the nitrogen source and to track the fate and transformation of nitrogen-containing compounds.

Nitrogen (N) is the most essential element that can increase the production of cotton fibres that are used to manufacture cotton yarns, textiles and fabrics. To ensure cotton fibre production, sufficient amount of nitrogen should be supplied as fertilizers (chemical or organic fertilizers) to the soil to meet the nitrogen demand for the growth and productivity of cotton crops.

Since most of fertilizer-nitrogen released into the soil is absorbed through root systems, translocated, and stored in the cotton fibres during cultivation, the isotopic nitrogen composition of the cotton fibre, expressed as $\delta^{15}N$, reflects the type of fertilizer from which it originated. In general, $\delta^{15}N$ of chemical fertilizers is close to 0 ‰, ranging from −3,9 ‰ to +0,5 ‰, while that of composted manure is much higher, ranging between +15,4 ‰ and +19,4 ‰. Cotton fibres harvested are processed to produce cotton yarns, textiles, and fabrics. Therefore, it is believed that $\delta^{15}N$ of a cotton fibre that contains information about the type of nitrogen fertilizers can serve as an isotopic fingerprint to identify authenticity and trace origin of cotton products, such as yarns, textiles and fabrics.

An accurate measurement of $\delta^{15}N$ of an aliquot of the dried cotton fibres can be obtained by using a continuous-flow type isotope ratio mass spectrometer linked to an elemental analyser (EA-IRMS). An aliquot of the test sample is washed, dried, ground to very fine powders using a ball-mill, weighed in a tin capsule on a microbalance, and then analysed for cotton-$\delta^{15}N$.

In particular, the use of chemical fertilizers can be attested by simultaneous measurements of stable nitrogen isotope ratio ($\delta^{15}N$) of cotton fibre and soil samples at harvest by using the determination process specified herein, and the determination procedure is specified in Annex A. Annex B shows some main outcomes of feasibility testing performed on cotton fabrics from organic certified textiles (see Table B.1) and raw cotton fibres obtained from several countries (see Table B.2).

5 Reagent

5.1 Reference material, **nitrogen isotopes in ammonium sulfate (IAEA-N2)** provided by the International Atomic Energy Agency (IAEA), necessary to check the accuracy and precision of $\delta^{15}N$ analysis, and available as NIST RM8548.

The $\delta^{15}N$ of NIST RM8548 is +20,3 ‰, and the accuracy and reproducibility of $\delta^{15}N$ analysis checked periodically with this standard should be lower than 0,4 ‰ and 0,2 ‰, respectively.

6 Apparatus

6.1 Drying oven, capable of heating up to around 80 °C.

6.2 Ball-mill, capable of grinding to final size smaller than 5 µm, and vibrational frequency ranging 3 Hz to 30 Hz (180 ~1 800) r/min are at least required.

6.3 Microbalance, with readability of 1 µg.

6.4 Continuous-flow elemental analyser-isotope ratio mass spectrometer (EA-IRMS).

7 Preparation

7.1 Sampling of test specimen

Cotton fibre samples are taken from fabricated textiles and prepared as test specimens.

7.2 Pre-treatment of test specimen

Cotton fibre samples are immersed with 100 ml distilled waters for 3 min and oven-dried at 80 °C for 24 h. Dried samples are chopped, homogeneously ground into a very fine powder using a ball-mill and used in triplicate for ^{15}N analysis.

8 Test procedure

8.1 Weighing of aliquot

8.1.1 Determination of weighing amount needed

Each finely ground test specimen is weighed with a tin capsule (or can) using a microbalance (readability: at least 1 µg).

Provide between (100 ~1 200) µg of nitrogen for each aliquot of the test specimen to meet EA-IRMS detection limits.

However, the minimum amount needed for accurate and precise analysis of $\delta^{15}N$ is typically about 100 µg of nitrogen.

A simple formula to determine the amount of an aliquot to weigh for the EA-IRMS measurements is given as Formula (1):

$$m_a = \frac{m_N}{1\,000} \times \frac{100}{w_p} \quad (1)$$

where

m_a is the dry mass of an aliquot needed for EA-IRMS measurements (mg);

m_N is the dry mass of nitrogen needed (µg);

w_p is the mass fraction percentage of nitrogen in the test specimen (%).

For example, if the test specimen has 1 % nitrogen and 100 µg of nitrogen for the IRMS measurements is provided, then an aliquot that weighs 10 mg shall be prepared. For samples with unknown % nitrogen, it is recommended to determine the % nitrogen of the test specimen before making isotope analysis.

In general, the optimal mass of an aliquot for the IRMS measurements is 33 mg to 56 mg for cotton products (cotton fibres in yarns or textiles), since cotton products typically contain between 0,18 and 0,30) % nitrogen.

8.1.2 General weighing procedure

a) Clean the plate and tools using lint-free papers or equivalents moistened with alcohol or acetone.