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Climate change monitoring - Analysis of the oxygen isotopes of soil organic carbon - Mass spectrometry

气候变化监测 土壤有机碳氧同位素分析 质谱法

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Foreword

This document complies with the provisions of GB/T 1.1-2020 "Standardization Work Guidelines Part

1.Structure and Drafting Rules of Standardization Documents". Drafting. This document was proposed by the China Meteorological Administration. This document is under the jurisdiction of the National Technical Committee on Standardization of Climate and Climate Change (SAC/TC540). This document was drafted by: Qinghai Provincial Meteorological Bureau, Nanjing Agricultural University, Institute of Urban Environment, Chinese Academy of Sciences, and Qinghai Provincial Forestry and Grassland Bureau. Bureau, Qinghai University, Chinese Research Academy of Environmental Sciences, Qinghai Provincial Fifth Geological Exploration Institute, Nanjing Deshui Energy Conservation Technology Co., Ltd., Qinghai Yihu Sheng Wu Technology Co., Ltd. The main drafters of this document are. Zhang Zhichun, Pan Genxing, Zhang Han, Li Gang, Zhou Wanfu, Pang Xinwei, Sheng Haiyan, Wang Jing, Xin Yanjun, and Ji Bingyan. Xiang Feng, Lin Chunying, Ou Jianfang, Han Huibang, Wang Yujuan, Kang Xiaoyan, Guo Shiyu, Liu Na, Xin Yuanchun, Wei Lai.

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addition to the patents mentioned above, some content in this document may still involve patents. The issuing organization of this document does not assume responsibility for identifying patents. responsibility. Climate change monitoring Soil organic carbon and oxygen isotope analysis by mass spectrometry

1.Scope This document describes a mass spectrometry method for analyzing the oxygen isotope ratio (18O/16O) in soil organic carbon. This document applies to the use of elemental analysis-stability isotope ratio mass spectrometry (ESRMS) in climate change monitoring to analyze soil organic carbon. Oxygen isotope analysis.

4.Abbreviations The following abbreviations apply to this document. AR. Analytical Reagent EA-IRMS. Elemental Analyzer-Isotope Ratio Mass Spectrometer ter) VSMOW. Vienna Standard Mean Ocean Water

5 Reagents and Materials

5.1 Water used in experiments It should meet the water quality requirements of Grade II or above in GB/T 6682.

5.2 Hydrofluoric acid The purity is no less than AR grade.

5.3 Hydrochloric acid The purity is no less than AR grade.

5.4 Helium Purity greater than or equal to 99.999% (mole fraction).

5.5 Carbon monoxide (reference gas) The purity is greater than or equal to 99.999% (mole fraction), and its oxygen isotope ratio is determined using oxygen isotope standard samples/standard substances.

5.6 Silver Foil Cup We recommend using an inner diameter of 4mm and a height of 5mm.

5.7 Organic carbon and oxygen isotope standard samples/standard substances See Appendix A.

6 Instruments and equipment

6.1 EA-IRMS The basic requirements for operating parameters are given in Appendix B.

6.2 Balance Maximum sample weight is 2g (graduation value 0.001mg) or 100g (graduation value 0.1mg).

6.3 Water Bath Constant Temperature Oscillator The oscillation frequency is 80r/min~100r/min, and the temperature is 20°C~100°C.

6.4 Centrifuge The rotational speed should not be less than 4000 r/min.

6.5 Centrifuge tubes Made of polyethylene, with a volume of not less than 100mL, for use with a centrifuge.

6.6 Oven Volume 400L~500L, temperature 20°C~100°C.

7.1 Sample Preparation

7.1.1 Reagent Preparation The preparation of reagents should meet the following requirements.

a) Dilute hydrochloric acid. 5% hydrochloric acid concentration;

b) Solution I. 4% hydrochloric acid, 8% hydrofluoric acid;

c) Solution II. hydrochloric acid concentration 8%, hydrofluoric acid concentration 16%;

d) Solution III. hydrochloric acid concentration 16%, hydrofluoric acid concentration 24%.

7.1.2 Soil Sample Processing Crush the dried soil sample, remove stones, gravel, plant debris, and roots, then weigh 20g and grind it through a 100-mesh mortar using an agate mortar. Use the sieve for later use.

7.1.3 Sample Preparation The sample preparation steps are as follows:

a) Weigh 1.0g of soil sample as specified in 7.1.2, grind it through a.200-mesh sieve, and transfer the entire sample to a 100mL polyethylene centrifuge tube. In the process, use a dropper to take 1-2 mL of experimental water to moisten the sample, and then add dilute hydrochloric acid solution dropwise until no more bubbles are produced;

b) Add 80 mL of Solution I to the centrifuge tube, shake in a constant temperature water bath at 40°C~45°C for 3 hours, then remove and... Centrifuge at 4000 rpm for 30 min, discard the supernatant and retain the precipitate. Repeat this step 4 times.

c) Add 80 mL of solution II to the centrifuge tube, and repeat the steps in 7.1.3b) four times.

d) Add 80 mL of solution III to the centrifuge tube, shake in a constant temperature water bath at 40°C~45°C for 3 hours, then remove the centrifuge tube and keep it in place. After standing vertically for 12 hours, remove the sample and centrifuge at 4000 r/min for 30 min. Discard the supernatant and retain the precipitate.

e) Add 80 mL of solution III to the centrifuge tube, and repeat the steps in 7.1.3b) four times.

f) Add 25 mL of experimental water to the centrifuge tube, incubate in a constant temperature water bath at 40°C~45°C for 10 min, then remove and centrifuge at 4000 r/min. Centrifuge for 30 minutes, discard the supernatant, and retain the precipitate. Repeat this step three times.

g) Tilt the centrifuge tubes at 40°~50° and place them in an oven at 50°C~60°C for no less than 24 hours, until the precipitate is completely dry;

7.2.1 Instrument Preparation

7.2.1.1 Instrument Parameter Settings The instrument and equipment are in normal working condition. The EA pyrolysis furnace temperature is set to 1450°C, the EA column oven temperature to 80°C, and the helium (carrier) is set to... The gas flow rate is 100 mL/min.

7.2.1.2 Instrument stability check Within the instrument's measurement range, perform 5 to 10 equal volumes of carbon monoxide that meet the requirements of 5.5, until the standard deviation of the delta measurement result is reached. Less than or equal to 0.1 per mille.

7.2.1.3 Instrument linearity check Within the instrument's measurement range, perform 5 to 10 different volumes of carbon monoxide that meet the requirements of 5.5, until the delta measurement results are linear. The deviation is less than or equal to 0.1 per mille.

7.2.2 Measurement Procedure The sample determination steps are as follows:

0.30 mg of the analytical sample prepared according to 7.1.3h), place it in a silver foil cup, and press and wrap it with tweezers to seal it. Small ball;

b) Place the small ball, which is wrapped and sealed in a silver foil cup, into the autosampler to determine the oxygen isotope ratio.

8.1 Expression of Measurement Results

8.2 Conversion of Measurement Results to VSWOM Calculate according to formula (3), and round the result to two decimal places.

9.Quality Control Quality control includes.

a) Instrument blank and silver foil cup blank tests should be performed before each measurement. The mass-to-charge ratio (m/z) of the blank should be 28, and the signal value of the peak should be less than... 100mV (or 100nA);

b) At least 10% of the samples in each batch of analysis shall be measured in parallel;

c) During the analysis of each batch of analytical samples, 10% of the total number of samples should be analyzed using standard samples/standard substances;

d) Calculate the precision according to GB/T 6379.2;

e) The absolute value of the difference between two measurements of the same analytical sample does not exceed the repeatability limit (r) of 0.20 per mille;

f) The absolute difference between two independent determinations of the same analytical sample does not exceed the reproducibility limit (R) of 0.37 per mille.