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IAEA-TECDOC-1783

Supporting Sampling and Sample Preparation Tools for Isotope and Nuclear Analysis

Joint FAO/IAEA Programme
Nuclear Techniques in Food and Agriculture

IAEA
International Atomic Energy Agency

SUPPORTING SAMPLING AND SAMPLE PREPARATION TOOLS FOR ISOTOPE AND NUCLEAR ANALYSIS

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SUPPORTING SAMPLING AND SAMPLE PREPARATION TOOLS FOR ISOTOPE AND NUCLEAR ANALYSIS

PREPARED BY THE
JOINT FAO/IAEA DIVISION OF NUCLEAR TECHNIQUES IN FOOD AND AGRICULTURE

INTERNATIONAL ATOMIC ENERGY AGENCY
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FOREWORD

The IAEA and the Food and Agriculture Organization of the United Nations (FAO), through the Joint FAO/IAEA Division of Nuclear Techniques in Food and Agriculture, help scientists and farmers worldwide to ensure food security and to promote sustainable agricultural development. The Joint Division's programme and activities are problem oriented and demand driven, and focus on developing and transferring technologies in response to real and practical needs. This programme assists Member States in the implementation of appropriate nuclear and related techniques where they have a competitive advantage to enhance, improve or increase agricultural production and to protect natural resources.

Sustainable soil, water and nutrient management is fundamental to ensuring food security. The Soil and Water Management and Crop Nutrition (SWMCN) Subprogramme of the Joint Division focuses on the development of improved soil, water and crop management technologies and practices for sustainable agricultural intensification through the use of nuclear and related techniques.

Nuclear and related techniques can help to develop climate smart agriculture practices by optimizing water and nutrient use efficiency, promoting soil organic carbon sequestration and assisting in the evaluation of soil erosion control measures. The knowledge of the behaviour of radioactive materials in soil, water and foodstuffs is also essential in enhancing nuclear emergency preparedness and response in food and agriculture.

Appropriate sampling and sample preparation are the first steps to ensure the quality and good use of the measurements. It is often difficult to find the procedures described in a comprehensive manner, including illustrations of each step. This publication aims to bridge this gap for scientists, technicians and students. It presents a selection of five standard operating procedures (SOPs), providing information on the application of sampling and sample preparation tools that are mandatory when conducting reliable isotope and nuclear analysis of soil, water and plant materials. Of the five SOPs presented, two were developed by the SWMCN Laboratory, and the other three focus on the clarification and illustration of existing procedures.

The IAEA wishes to thank all the contributors involved in the preparation of this publication. The IAEA officers responsible for this publication were G. Dercon, L. Mabit, A. Wahbi and L. Heng of the Joint FAO/IAEA Division of Nuclear Techniques in Food and Agriculture.

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CONTENTS

SUMMARY

This publication on Standard Operating Procedures (SOP) was developed to provide illustrated, step by step, comprehensive guidance for sampling and processing of soil, water and plant materials. It assists scientists, technicians and students in implementing procedures and tools to take and prepare samples for isotope and nuclear analysis. Member States require this step by step guidance to improve soil, water and nutrient management practices for making agriculture resilient to impacts of climate change (climate-smart agriculture), and to prepare and respond to nuclear emergency in food and agriculture.

Five examples of SOPs on (1) fractionating soil organic matter into different pools, (2) purifying inorganic phosphate in soil and sediment, (3) rapid extraction of water from soil and plant, (4) precise soil and sediment sampling, and (5) soil bulk density, are presented in this publication.

The first SOP covers fractionating of soil organic matter into different pools, particularly the separation of the particulate organic matter. This fractionation procedure is essential in conventional soil organic carbon dynamics studies and modelling, but is also important to assess soil organic carbon stability using nitrogen-15 stable isotope techniques.

The second SOP provides step by step information to purify inorganic phosphate in soil and sediment samples prior to oxygen-18 isotopic abundance analysis in phosphate. The oxygen-18 isotope composition of phosphate has been used in the study of biological cycling of phosphorus in seawater and marine sediments. This approach is now being applied as well to understand phosphate cycling in agricultural soils.

The SOP for extracting water from soil and plant focused on rapid and cost effective approach for oxygen-18/oxygen-16 and Deuterium/Hydrogen ratio measurements in soil and plant water. This technique is used to quantify soil evaporation and plant transpiration in agro-ecological systems and hydrological investigation.

Procedures for precise soil and sediment sampling are provided for a new sampling device. Such precise sampling can be used for assessing soil erosion through radionuclide techniques or providing information on the amount of the soil to be treated or removed in case of contamination linked to nuclear power plant accidental releases or industrial pollution.

The last SOP shows detailed guidelines for measuring soil bulk density. Many guidelines on bulk density are available in literature, but the lack of illustrated step by step guidelines warranted this SOP in this publication. Soil bulk density measurements are used in a wide range of soil assessments such as soil structure (compaction), soil water and soil organic carbon dynamics.

1. PARTICULATE ORGANIC MATTER (POM) SEPARATION

M. HEILING, G. DERCON

1.1. SCOPE AND FIELD OF APPLICATION

Information on soil organic matter (SOM) pools is of vital importance for studying the impact of soil management and environmental factors on soil organic carbon, an important part of the global carbon cycle. Several conceptual SOM pools with different turnover rates are available to feed models or to study carbon cycles. The fractionation scheme of Zimmermann (Fig. 1.1) allows isolating the labile particulate organic matter (POM) pool. Besides its use in conventional soil organic carbon dynamics studies and modelling, this pool can be determining as well in the evaluation of soil organic carbon stability based on the use of stable ^{15}N and ^{13}C isotopes [1.1, 1.2].

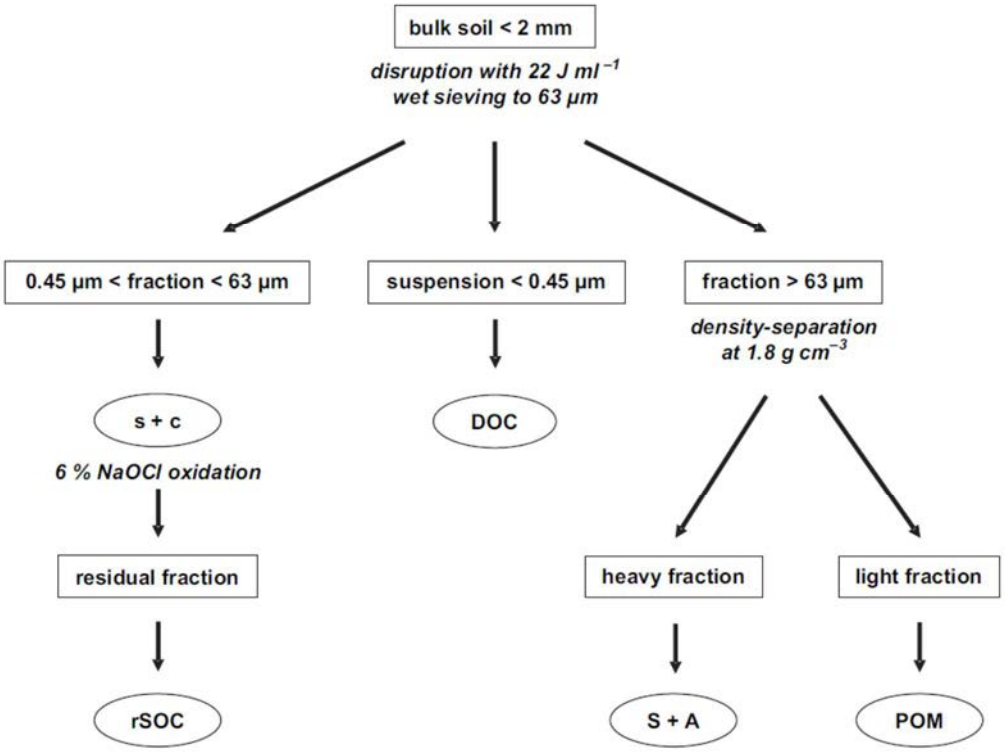


FIG 1.1. Diagram of the fractionation procedure: s + c = silt and clay, rSOC = resistant soil organic carbon, DOC = dissolved organic carbon, S + A = sand and stable aggregates, and POM = particulate organic matter; Reproduced courtesy of Zimmermann, M., [1.3].

1.2. PRINCIPLE

The presented method for the isolation of POM is a physical method, based on particle size and density separation after disrupting the soil aggregates with soft energy (22 J ml^{-1}). POM is defined as the light fraction (> 1.8 g cm^{-3}) with a particle size > 63 µm.

1.3. TYPICAL SAMPLE

Air-dry soil, sieved to < 2 mm

1.4. REAGENTS

sodium polytungstate (SPT) solution at a density of 1.8 g cm^{-3}

- 243 g $3Na_2WO_4.9WO_3.H_2O$ + 200g H_2O give a density of 1.8 g cm^{-3}
- check the density of the solution by weighing out

Caution: The waste has to be collected and treated as hazardous to environment material.

1.5. EQUIPMENT

The following materials are needed:

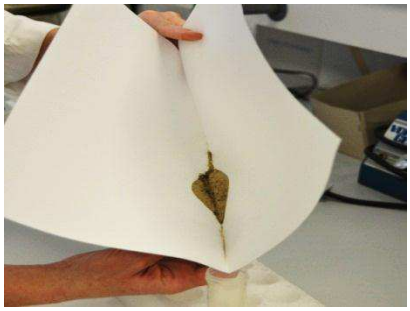
- drying oven (set at 40°C);
- stainless steel sieves, 63 µm;
- aluminium dishes or similar for drying;
- dispenser, set to 150 ml;
- beakers;
- balance, ±0.0001 g;
- balance, ±0.001 g;
- ultrasonic probe with an output-energy of 22 J ml^{-1};
- centrifuge vials, 50 ml;
- deep freezer, (approx. –18°C);
- nylon filter with a mesh size smaller than 60 µm;
- funnels;
- small vials for drying and storing samples.

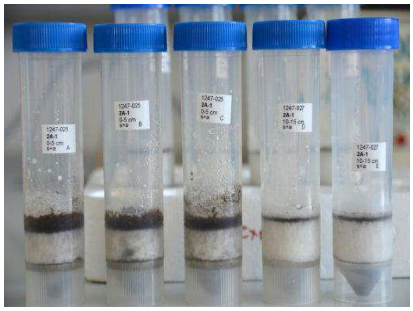
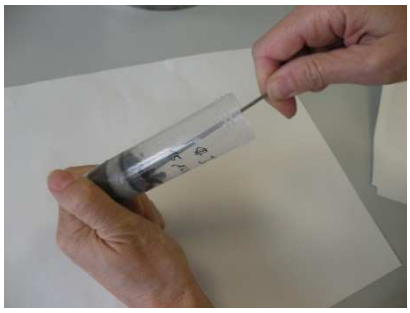
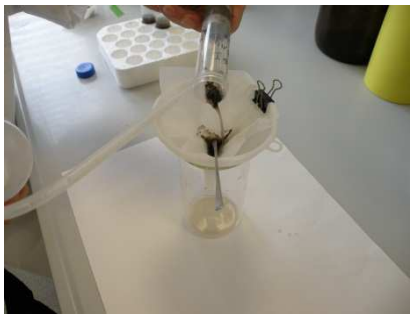
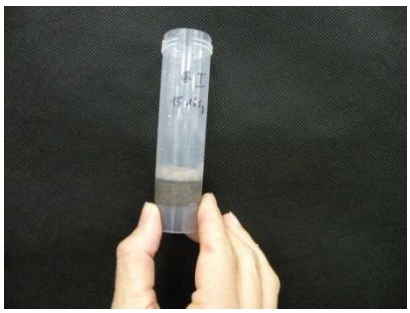
1.6. PROCEDURE

Step	Procedure	
Step 1 **Dry Sieving**	Sieve air-dry soil to < 2mm, mix well to obtain one homogeneous sample and transfer it to a clean aluminium dish.	

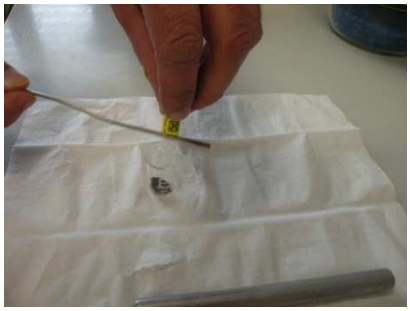
	Dry the soil at 40°C overnight.	
Step 2 **Weighing**	Weigh out 30g ± 1g of dry soil (W1) into a 250 ml glass beaker and note the soil weight with a precision of ±0.001g.	
Step 3	Add 150 ml of demineralized water using a dispenser.	
Step 4 **Ultrasonic dispersion**	Disperse the sample using a calibrated ultrasonic probe with an output-energy of 22 J ml^{-1}	
Step 5 **Washing procedure**	Transfer the dispersed suspension into a sieve with a mesh size of 63 µm.	

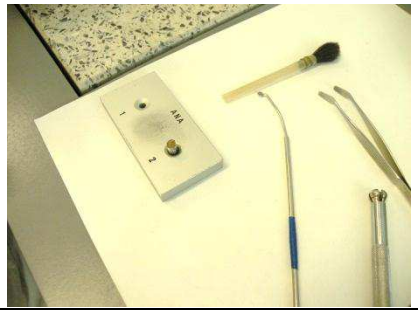
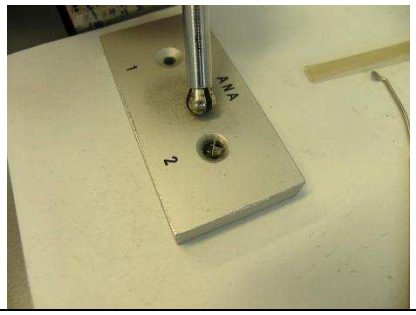
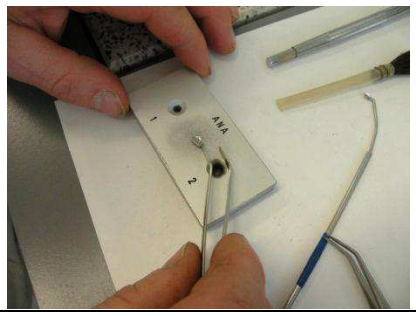
	Wash the soil in the sieve with demineralized water until the rinsing water is clear.	
	Check by carefully touching the bottom site of the sieve with a clean paper towel.	
Step 6	Dry the sieved sample, consisting of **sand, stable aggregates and POM**, on the sieve at 40°C in a non-ventilated oven. **Remark:** It is also possible to wash all particles directly into an aluminium dish and dry the sample in the dish.	
Step 7 **Transfer of sample**	Transfer sample using a brush. Take care not to lose any brush hair.	
	Possibility 1: Quantitatively transfer the dry sample to a 50 ml centrifuge vial, use a glass funnel.	

	Possibility 2: Transfer all sample onto a smooth sheet of paper and fold it in the middle for direct transfer into the centrifuge vial	
	Use a brush to clean the sheet of paper completely and transfer all particles quantitatively!	
Step 8 **Density Gradient**	Add 10 ml sodium polytungstate (SPT) solution at a density of 1.8g cm^{-3}	
	Stir the suspension gently with a small spatula; avoid splashing onto the centrifuge vial walls	

	Wait until heavy sediments (sand and stable aggregates) settle and until light fraction (POM) floats on the surface (usually 2–4 hours). Then place in deep freezer (approx. –18°C) overnight.	
Step 9 **Isolation and cleaning of POM fraction**	Scrap off the top layer of the frozen sample, which represents the **light fraction (POM).**	
	Transfer the POM fraction and let it melt on a nylon filter with a mesh size smaller than 63 µm, placed in a funnel	
	Transfer all POM with demineralized water using a wash bottle.	
	Check: the black layer has to be completely removed.	

	Wash the light fraction with demineralized water	
	Collect the dilute polytungstate solution for safe disposal.	
	Transfer the POM as quantitatively as possible from the nylon filter into a **pre-weighed** glass or poly-ethylene centrifuge vial (2 ml).	
Step 10 **Drying of POM**	Dry the POM sample at 40°C until weight constancy.	
Step 11 **Calculation of POM fraction**	Take the weight of the POM fraction (W2) with a precision of ±0.1 mg and calculate percentage POM of total soil sample. W2= POM plus vial weight – vial weight % POM = W2 *100/ W1	

Step 12 **Preparation for isotope analysis**	Homogenization of sample: Crush the sample as described or use a sample disrupter if available	
	Transfer the dry POM quantitatively into a small glass beaker with flat bottom.	
	Crush and homogenize the POM with a metal or glass rod.	
	Put the powdered POM back into the sample vial.	
	Brush all traces of powder back into the sample vial. Clean the used equipment for homogenization and sample transfer with pressurized air to avoid cross contamination.	

	Weigh about 7 mg of powdered POM into a tin capsule for Isotope Ratio Mass Spectrometry (IRMS) analysis.	
	Place the tin capsule in a special sample holder.	
	Seal the tin capsule carefully with a special tool.	
	Form a small ball using forceps (do not touch the tin capsule with your fingers).	
	Place sample in a labelled micro-plate rack and record sample position. Close the micro-plate properly (wrap with tape or para-film) and store in a dry place for transport. The sample is now ready for measurement of stable nitrogen and carbon isotopes (^{15}N, ^{13}C) and percentage total N and C by IRMS.	

1.7. CALCULATION

$$\% \text{ POM} = W2 \times 100 / W1 \qquad (1.1)$$

Where: % POM is the percentage of particulate organic matter
W1 is the initial weight of dry soil sample [g]
W2 is the weight of POM fraction after samples processing [g]

REFERENCES TO SECTION 1

[1.1] CONEN, F., ZIMMERMANN, M., LEIFELD J., SETH B., ALEWELL C., Relative stability of soil carbon revealed by shifts in $\delta^{15}N$ and C:N ratio. Biogeosciences, **5** (2008) 123–128.

[1.2] DE CLERCQ T., HEILING M., DERCON G., RESCH C., AIGNER M., MAYER L., MAO Y., ELSEN A., STEIER P., LEIFELD J., MERCKX R. Predicting soil organic matter stability in agricultural fields through carbon and nitrogen stable isotopes. Soil Biology & Biochemistry **88** (2015) 29-38

[1.3] ZIMMERMANN, M., LEIFELD, J., SCHMIDT, M.W., SMITH, P., FUHRER J., Measured soil organic matter fractions can be related to pools in the RothC model. European Journal of Soil Science **58** (2007) 658–667.

2. METHOD FOR THE PURIFICATION OF INORGANIC PHOSPHATE IN SOIL- AND SEDIMENT SAMPLES PRIOR TO ANALYSIS OF THE $\delta^{18}O$ ISOTOPIC ABUNDANCE IN PHOSPHATE

M. AIGNER, F. TAMBURINI, M. HEILING, G. DERCON

2.1. SCOPE AND FIELD OF APPLICATION

The ratio of stable oxygen isotopes in phosphate has been used successfully to study the biological cycling of phosphorus in seawater and marine sediments, and now this approach is being applied to study phosphorus cycling in agricultural soils [2.1, 2.2]. As an important major element, phosphorus can limit agricultural production and on the other hand excess of phosphorus can lead to water pollution. A better understanding of phosphorus cycling is essential to improve agricultural and environmental management.

2.2. PRINCIPLE

This SOP provides step by step guidance on how to extract and purify inorganic P (Pi) from organic matter in soils and other organic rich materials and precipitate it as a stable, non-hygroscopic pure chemical compound of low solubility, needed for the analysis of stable isotope ^{18}O in phosphate by Thermal Conversion Elemental Analyser – Isotope Ratio Mass Spectrometer (TC/EA-IRMS).

The method includes following steps:

1. HCl-extraction
2. Dissolved organic matter removal
3. Ammonium phospho-molybdate (APM) precipitation and dissolution
4. Magnesium ammonium phosphate (MAP, struvite) precipitation and dissolution
5. Cation removal
6. Silver phosphate precipitation
7. Analysis by Thermal Conversion Elemental Analyser – Isotope Ratio Mass Spectrometer (TC/EA-IRMS)

2.3. TYPICAL SAMPLE

- Soil samples, sediments
- Rock phosphates, manure samples, inorganic fertilizers
- All samples should be cooled to reduce microbial activities dried as soon as possible.

2.4. REAGENTS

Ammonium nitrate solution, 35 % (w/v):
dissolve 538.5 g NH_4NO_3 in 1000 ml of DDW

Ammonium heptamolybdate solution:
dissolve 10 g of $(NH_4)6Mo_7O_{24}\cdot 4H_2O$ in 90 ml of DDW (i.e. 66 g / 600 ml)

NH_4NO_3 solution, 5 % (w/v):
dissolve 52.7 g NH_4NO_3 in DDW and make to 1L

Citric acid-NH_4OH solution:
dissolve 10 g citric acid and 140 ml conc. NH_4OH in 300 ml DDW

Magnesia solution:
dissolve 50g $MgCl_2 \cdot 6H_2O$ and 100g of NH_4Cl in 500 ml DDW, acidify to pH 1 with 12M HCl, adjust volume to 1L

Ammonia solution 1:1 (v/v):
add 50 ml NH_4OH conc. to 50 ml DDW and mix

Ammonia solution 1:20 (v/v):
add 100 ml NH_4OH conc. to 1900 ml DDW and mix

1M HCl:
dissolve 82.7 ml of HCl conc. in 900 ml DDW and make to 1L

0.5M HNO_3:
add 33 ml of HNO_3 conc. to 967 ml of DDW

1M HNO_3:
pour 66 ml of HNO_3 conc.(69%) to 800 ml of DDW, mix and make to 1L

7M HNO_3:
pour 454 ml of HNO_3 conc. (69%) to 400 ml of DDW, mix and make to 1L

Ag-ammine solution:
10.2 g $AgNO_3$, 9.6 g of NH_4NO_3, 18.5ml of NH_4OH conc. and 81.5 ml of DDW

Resins:
- Supelite™, DAX-8 for organic matter removal, Supelco Analytical, 21567-U
- AG® 50W-X8 Resin for cation removal, Bio-Rad, Cat. No. 142-1441

2.5. EQUIPMENT AND MATERIALS

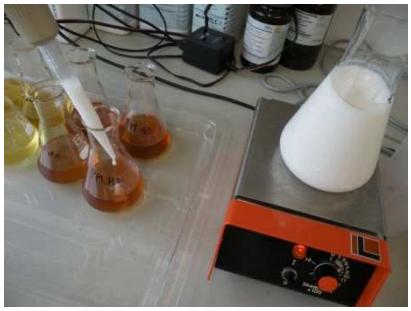
Detailed equipment and materials information can be found on:
Website: www-naweb.iaea.org/nafa/about-nafa/biotechnology-lab.html,
Email: Official.mail@iaea.org

- Standard lab glass ware and equipment;
- Disposable 50 ml-polypropylene-centrifuge-tubes, skirted design (freestanding);
- GF/F filters (Ø 47 mm);
- Cellulose Acetate Membrane filters, 0.2 µm, Ø 47 mm;
- Cellulose Nitrate Membrane filters, 0.2 µm, Ø 47 mm;
- Polycarbonate filters, 0.2 µm, Ø 47 mm;
- Silver capsules, pressed, 4 × 3.2 mm, Elemental Microanalysis;
- Lamp black (Gasruß), conditioned, 10 ml, Elementar, Germany, Art. No. 23.00-0097;
- Fume hood;
- Fridge (max. +8°C);
- Drying oven (no ventilation);
- Water bath shaker set at 50°C;

- Multi-position magnetic stirrer, 15 positions;
- Reference Material IAEA-601 Benzoic acid standard for ^{18}O-PO_4-analysis;
- Reference Material IAEA-602 Benzoic acid standard for ^{18}O-PO_4-analysis.

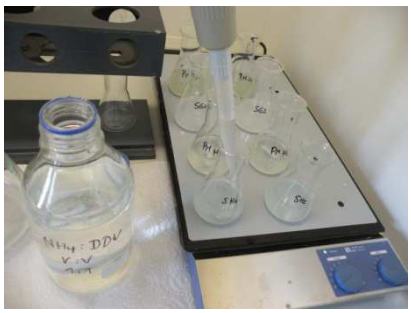
2.6. PROCEDURE

Step	Procedure	
Step 1: **HCl-extraction**	**Preparation day 1:** ▪ dry the samples at 40°C ▪ sieve the samples to < 2 mm ▪ weigh 20–25 g soil sample into a 250 ml Polypropylen (PP) bottle (fertilizer samples: 2 g, manure samples: 1 g)	
	▪ add 100 ml of 1M HCl using a dispenser	
	▪ extract by shaking 16 hours (overnight) at 180 rpm	
	Preparation day 2: centrifuge for 15 min at 1300 g (e.g. 2500 rpm with rotor radius 19.2 cm)	

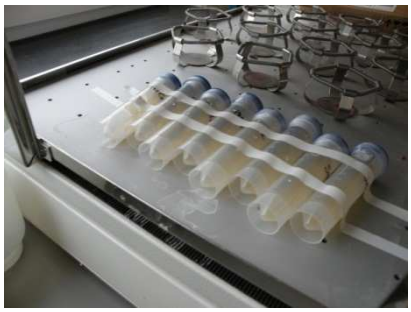
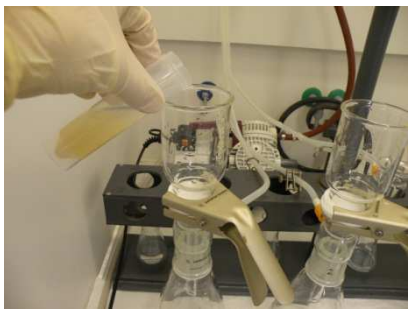
	filter supernatant solution through GF/F filter (diameter 47/50 mm)	
	use a vacuum filtration apparatus connected to a vacuum pump collect filtrate in a dry 200 ml EM-flask => possibility of storage	
Step 2: **Dissolved Organic Matter (DOM) removal**	**Cleaning of resin:** Consider 20 ml of DAX-8 Amberlite resin (about 10-15 g) per sample. ▪ In a bottle, add 3 g of HPLC-grade methanol for each 20 ml of resin. ▪ Shake for 15 min ▪ rinse resin with DDW for 10–15 min **Note:** DOM removal only necessary for soils with high organic content	
	DOM-removal from sample: ▪ prepare a resin – DDW slurry (1:1 v/v) ▪ load 20 ml of the resin slurry onto the filtered sample solution in a 200 ml Erlenmeyer flask	

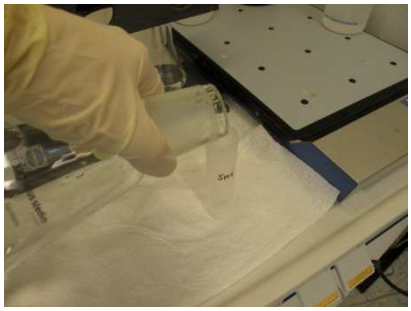
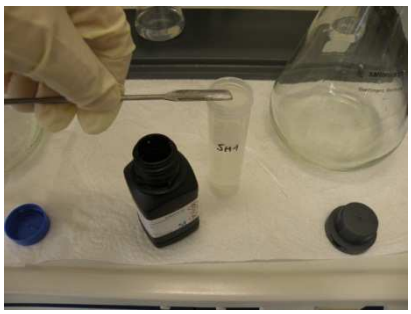
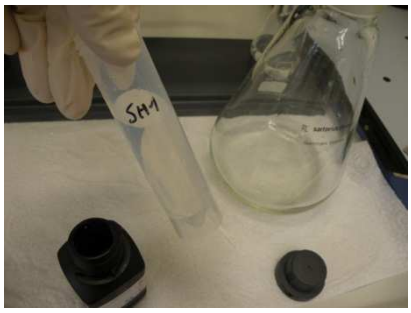
	▪ cover EM-flasks with parafilm ▪ shake for 3 hours at 130 rpm	
	▪ filter solution through a 0.2 µm polycarbonate filter into a new 200 ml Erlenmeyer (EM) flask (attention: do not stir before filtration, better decant and pipette solution into the filter apparatus – filtration takes long time!!) ▪ rinse resin with 10 ml DDW and add wash-solution to the sample solution	
	Storage of the resin ▪ recover resin and recondition with wash-solution: mixture of 1M HCl and methanol: mix resin with wash-solution for some time ▪ store resin in methanol in the fridge	
Step 3: **APM-precipitation and dissolution**	▪ add 25 ml of Ammonia nitrate solution, 35% (w/v) to the filtrates ▪ place flasks in a shaking waterbath at 50°C ▪ slowly add 40 ml of Ammonium heptamolybdate – solution, 10% (w/v)	

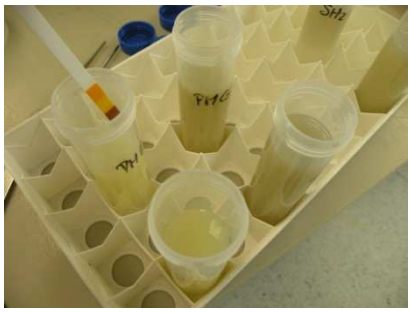
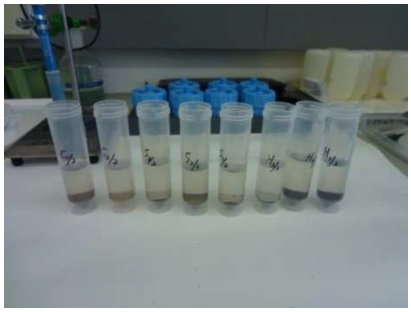
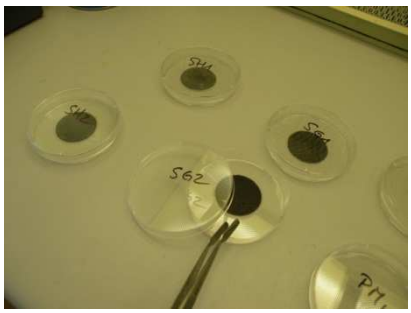
	▪ shake in the waterbath overnight => APM precipitates!!	
	Note: if colour of the solution is green (e.g. calcareous soils, or if extraction is done with H_2O, resin, etc.) => add some conc. H_2SO_4, (max. 1 ml) and if still no precipitate is formed, add optionally some ammoniumheptamolybdate salt crystals to start precipitation.	
	Preparation day 3: ▪ recover APM precipitate by filtering through a 0.2 µm cellulose acetate filter ▪ rinse filters 2–3 times (about 200 ml in total) of a 5% NH_4NO_3 solution ▪ collect the NH_4NO_3 – wash-solution in separate waste container for safe disposal	
	▪ place filter with APM in a 100 ml Erlenmeyer → possibility of storage	

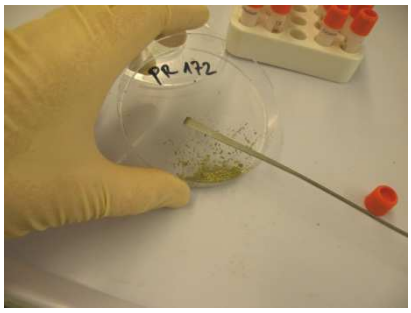
	Store APM crystals in fridge covered with parafilm	
Step 4: **Magnesium ammonium phosphate (MAP, struvite) precipitation and dissolution**	**Preparation day 4:** ▪ place flask on a magnetic stirrer ▪ dissolve the APM crystals in 30–50 ml of citric acid-NH_4OH solution ▪ remove the filter	
	▪ while stirring add 25 ml of magnesia solution ▪ slowly add 7–15 ml of NH_4OH/DDW solution until the pH rises to 8–9 => MAP precipitation **Note:** slow addition of the ammonia solution, pH should remain stable around 8–9!!! ▪ cover with flasks with parafilm, make a small hole and leave solution on the magnetic stirrer overnight	
	Preparation day 5: ▪ filter the white MAP-precipitate through a 0.2 µm cellulose nitrate filter ▪ rinse thoroughly 3 times with 100 ml of 1:20 v:v NH_4OH/DDW solution	

	Note: to avoid co-precipitation of AgCl, check with some $AgNO_3$-crystals – should not be white at dissolution: Discard the first NH_4OH/DDW wash and rinse again with another 20 ml, check this solution for AgCl. ▪ transfer the filter with the MAP-precipitate into a 50 ml PP-tube	
	▪ dissolve the MAP-crystals in a minimal quantity (approx. 20 ml) of 0.5M HNO_3 ▪ remove the filter => possibility of storage	
	▪ condition **New** BioRad AG50 X8 cation resin (H+ form, 100–200 mesh) with 7M HNO_3 overnight (approx. 6 ml resin/sample)	
Step 5: **Cation Removal**	**Preparation day 6:** ▪ thoroughly wash the BioRad AG50 X8 cation resin with 2 × 1L of DDW on the magnetic stirrer, decant washing water, check if pH is neutral (or the pH of the used DDW is reached).	

	▪ decant most excess water of the centrifuged resin-slurry ▪ leave the slurry on the magnetic stirrer and use a 10 ml pipette with a cut tip	
	▪ pipette about 6 ml of resin-slurry (stirred on magnetic stirrer while pipetting) to the sample solution	
	▪ close the tubes tightly with parafilm around the screw caps	
	▪ fix the tubes by taping them horizontally on the shaker and shake slowly overnight (70 rpm)	
	Preparation day 7: ▪ filter the solution through a 0.2 um polycarbonate filter into the clean glass bottle ▪ wash the resin twice with max. 2 × 5 ml (!!) DDW and add the washing water to the sample solution	

	▪ transfer filtrate into a new 50 ml PP-tube	
	▪ collect, recondition and store the cation resin in 1M HNO_3 (66 ml conc. HNO_3 + 934 ml of DDW) in the fridge	
	▪ check filtrate for Chloride: add some $AgNO_3$-crystals to the filtrate	
	▪ if AgCl (white clouds) precipitates => wait 5–10 min and filter again through a new polycarbonate filter	
Step 6: **Silver Phosphate Precipitation**	**Preparation day 6, continued:** ▪ add about 5 ml of Ag-ammine solution	

	▪ place tube in an oven at 50°C for 1-2 days and repeatedly add DDW to keep the volume constant	
	Preparation day 8–9: ▪ if no Ag_3PO_4 crystals are formed after 1–2 days, check the pH and readjust to 7.0 by adding conc. HNO_3 or NH_4OH	
	▪ filter the precipitated Ag_3PO_4-crystals through 0.2 um polycarbonate filter	
	▪ wash thoroughly (3–4 times) with about 50 ml DDW to wash off any remaining NO_3-traces which would increase the oxygen-yield	
	▪ place each filter with Ag_3PO_4 precipitate in a separate, labelled petridish and close with lid	

	▪ dry the crystals in the oven at 50°C overnight **Note:** residual organic matter can be eliminated by treating the Ag_3PO_4 crystals with 15% H_2O_2 at RT for 4–8 days), extensive washing with DDW and drying.	
Step 7: **Silver phosphate sample preparation for TC/EA IRMS:**	▪ scratch off and transfer the Ag_3PO_4 crystals to a 2 ml (cryogenic) vial with screw cap	
	▪ weigh 200–400 µg of the crystals into silver capsules (3.5 × 4 mm) in triplicates ▪ add glassy carbon powder (Elementar Analysen Systeme GmbH, Germany) to the sample	
	Note: use benzoic acid (IAEA-601 and IAEA-602) as standard. There is no need to add glassy carbon. ▪ weigh 300 µg of IAEA-601 respectively 602 standard into silver capsules (3.5 × 4 mm)	

	▪ close silver capsules tightly using a special tool	
	▪ close capsules tightly by forming little balls using tweezers and gloves ▪ transfer closed capsules to coded racks	
	▪ analyse ^{18}O in Ag_3PO_4 by TC/EA IRMS (High Temperature Conversion Elemental Analyser Isotope Ratio Mass Spectrometry)	
Sample sequence	5 blanks, 5 empty spaces for Ag_3PO_4 stds, 2 IAEA-601, 2 IAEA-602 (Benzoic acid), 12 samples, 1 empty place for Ag_3PO_4, 1 IAEA-601, 12 samples, 1 empty space, 1IAEA-602, 12 samples, etc. 4 empty spaces for Ag_3PO_4 stds, 2 IAEA-601 and 2 IAEA 602 closing the sequence.	

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3. EXTRACTION OF WATER FROM SOIL AND PLANT SAMPLES FOR $^{18}O/^{16}O$ AND D/H ISOTOPE RATIO MEASUREMENTS

L. MAYR, M. AIGNER, L. HENG

3.1. BACKGROUND INFORMATION

The need for a rapid, inexpensive technique for routine extraction of water from plant and soil samples for Oxygen-18/Oxygen-16 ($^{18}O/^{16}O$) and Hydrogen-2/Hydrogen-1 (D/H) isotope ratio measurements is increasing due to the greater demand for isotopic data in agro-ecological, soil water, evaporation and transpiration partitioning and hydrological studies [3.1-3.4]. The common sample extraction technique is vacuum distillation [3.5-3.7]. Most of the current techniques are laborious, time consuming and involve complicated setups with specially-made glass apparatus. In addition, liquid nitrogen or dry ice is needed to freeze and trap water vapour evaporated during extraction. Both of these cooling agents can be difficult to acquire in many developing countries. With water isotope analyses becoming cheaper, easier and faster (e.g. through the development of modern laser isotope analysers), the bottleneck in sample throughput is often the water extraction time from the soil and plant samples instead of the isotopic analysis of water.

3.2. PRINCIPLE

A simple, fast and accurate vacuum distillation method using a commercial immersion cooler and a Dewar container filled with 2-propanol at -50°C, in place of the liquid nitrogen or dry ice for freezing water vapour is described here. The method can be easily adopted at a relatively low cost and allows large number of soil and plant samples to be extracted quickly for subsequent $^{18}O/^{16}O$ isotopic analyses.

3.3. EQUIPMENT

TABLE 3.1. LIST OF ITEMS FOR ASSEMBLING THE EXTRACTION SETUP FOR 12 SAMPLES AND APPROXIMATE PRICES (AS OF 2013)

	Quantity	Item	Estimated Cost (Euro)	Euro total
Assembly:	12	Valve cock, glass 10mm, PTFE spindle, 90°, 0-4mm	35	420
	25	Screw joint GL18 for tubing 10mm, PTFE, Bola D590-18	13	325
	12	Glassblower work including 10mm glass tube	30	360
	1 pack	Culture tubes, thread GL18, glass, 16x160mm, 100 pieces	46	46
	1 pack	Culture tubes, thread GL18, glass, 16x100mm, 100 pieces	38	38
Evacuation:	1	Rotary vane pump, two stage, NW10 flange (e.g. Edwards E2M0.7)	1500	1500
	1	Diaphragm valve (e.g., Edwards Speedivalve SP10K, NW10)	90	90
	1	Adapter, NW10 to Pipe 1/4" female	18	18
	1	T-piece, Aluminium, NW10	22	22
	1	Elbow 90°, Aluminium, NW10	18	18
	5	Clamping ring, NW10/16	5	25
	5	Polymer centering ring N, NW10	3	15
	1	Digital vacuum gauge, 1080 to 1 mbar (e.g. Vaccubrand DVR 2)	540	540
	1	Screw-in tube fitting, GL18 to NPT 1/4" male (e.g. Bola D516-32)	15	15
Distillation:	1	Immersion cooler, -50°C (e.g. Peter Huber,TC50)	3000	3000
	2	Block heater, analog, 2 blocks, up to 150°C	561	1122
	4	Aluminium block, 12 holes for 16mm tubes	95	380
	1	Dewar vessel, stainless steel (e.g. AirLiquide Agil 6)	693	693
	2	Laboratory jacks, stainless steel, 200x200mm	164	328
	1	2-Propanol, technical, 10L	75	75
			TOTAL	**9030**

3.4. REAGENTS

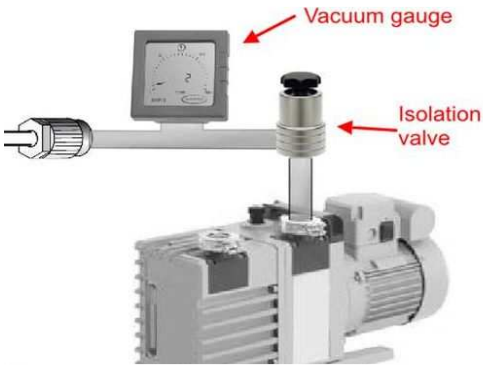
- 2-propanol, technical grade, 10 L

3.5. TYPICAL SAMPLE

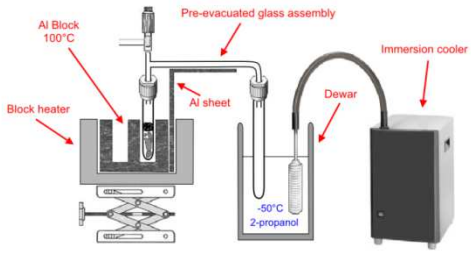
- soil samples, sediments, plant material

3.6. PREPARATIONS PRIOR TO EXTRACTION

3.6.1. Vacuum pump setup

Assemble the evacuation setup. See items under 'evacuation' in Table 3.1 above.	
It consists of a rotary vacuum pump, an isolation valve, a digital vacuum gauge, and a connector to attach the sample assembly.	

3.6.2. Extraction device

Prepare the extraction setup. See items under 'distillation' in Table 3.1 above.	

It consists of either one or two heating blocks, a six-liter stainless steel Dewar cold bath, submerged within is an immersion cooler and an electronic thermometer to monitor temperature of the cold bath.	
Fill Dewar container with four litres of technical grade 2-propanol. Submerge the cooler coil and switch on the immersion cooler.	
After about 2-3 hours, a temperature of -50°C should be reached. Leave the immersion cooler switched on during the whole extraction/distillation procedure.	

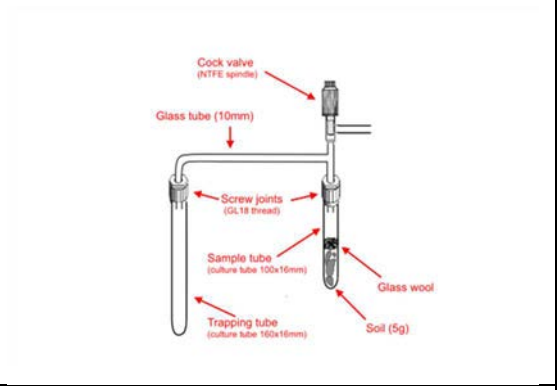
3.7. PROCEDURE

Step 1: Sample treatment applied to both plant and soil samples

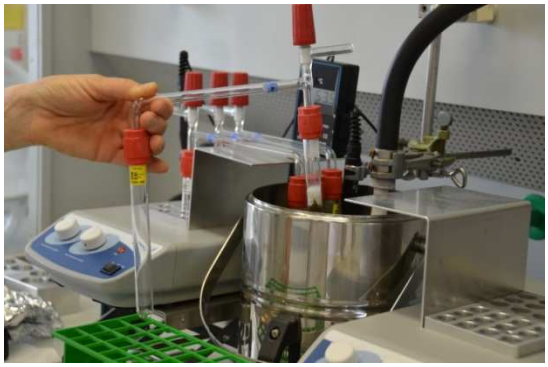
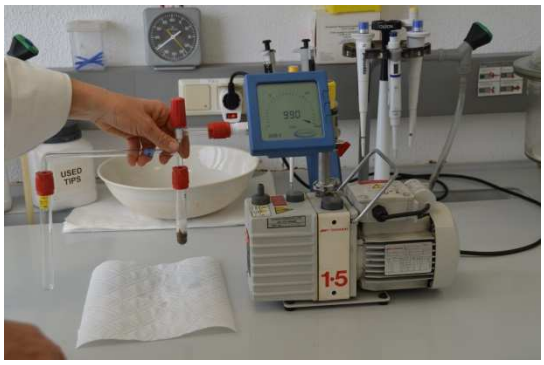
Weigh sample into glass tubes (culture tube with glass thread GL18).	

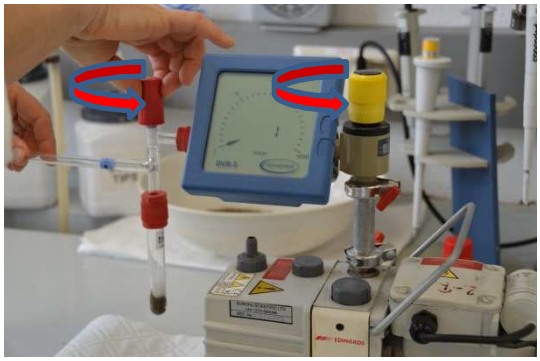
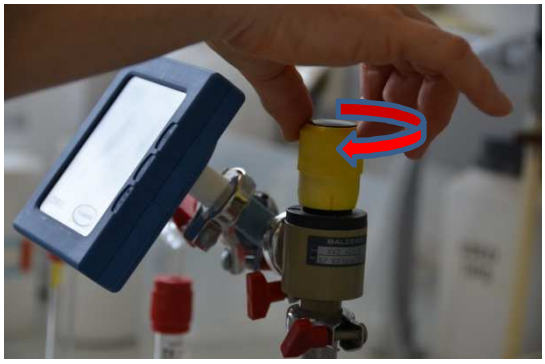
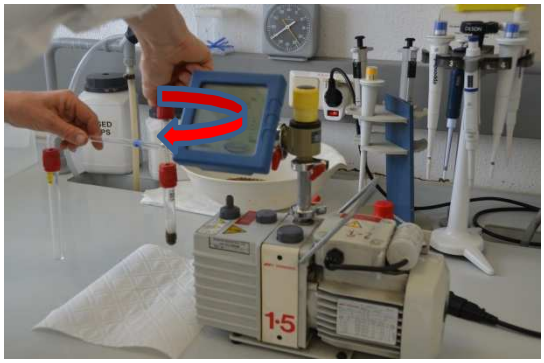
Sample size: approximately 3-5 grams of fresh soil or plant samples, depending on the moisture content for a maximum of 1-2 ml water to be collected.	
Cover sample with a small amount of glass wool	
Label and close the tubes with screw caps. Keep them away from direct sunlight.	
Freeze samples if they are not extracted immediately.	

Step 2: Assembly of tube components

Assemble the following components (sample tube and empty trapping tube) together. See items under 'assembly' in Table 3.1 above.	
Both tubes must be tightened with their respective screw connectors. Close the cock valve. Note: trapping tube is longer than sample tube.	

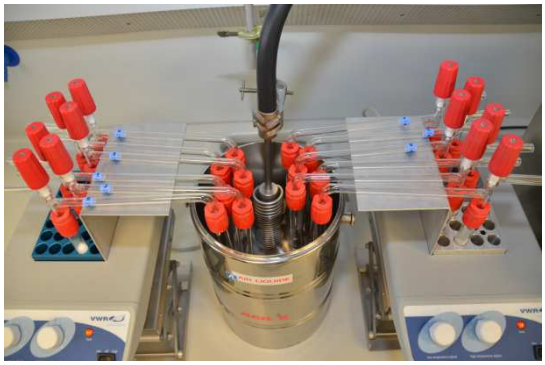
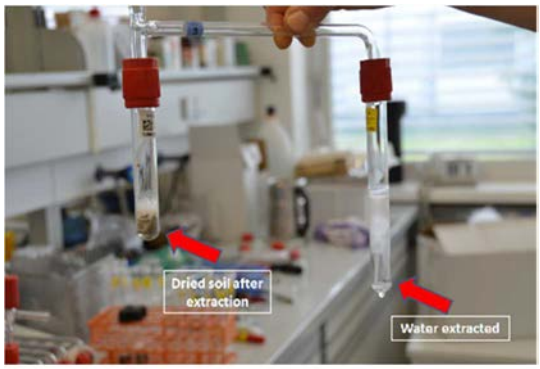
Step 3: Evacuation of sample tube and leak test

Prior to extraction, sample tubes (if not taken out of the freezer) need to be chilled in the Dewar for about 3 min to minimize water vapour in the gas phase and to avoid losses of water during evacuation.	
Take the glass tubing assembly out of the cooling trap and connect it to the vacuum pump system. Tighten the connecting joint. Switch on the pump.	

Open isolation valve and cock valve. The digital vacuum gauge should read 1 mbar within 5 seconds. If the desired pressure can't be reached retighten the joints or freeze the sample tube again.	
Close the isolation valve. The created vacuum should stay for at least 15 seconds. If the pressure rises to more than 2 mbar within this time period, retighten the tubes and redo the evacuation procedure.	
Close the cock valve to maintain the vacuum in the sample and disconnect the glass assembly from the pumping system.	

Step 4: Extraction / Distillation

Put the sample side of the unit on the heating block set to 100°C, and insert the other side with the water collection tube to about one third of the total length into the cooling bath at -50°C.	

Depending on the size of the heating block and the Dewar container for the immersion cooler, at least eight units can be distilled at one time. With two heating blocks, a maximum of 15 samples can be processed at a time.	
Turn on the heating block and distil for 2 hours at 100°C. Note: aluminium foil can be wrapped around the horizontal arm of the glass tubing to minimize water condensation in the transfer tube.	
Once completed, take the glass assembly off the distillation device and ventilate the glass assembly (open and close cock valve) and let the ice melt.	
Remove the water collection tube from the glass assembly. If not pipetted immediately, close the tube tightly until sampling.	

Pipet the extracted soil or plant water into a 2 ml sample vial. In case the sample amount is little, use sample vials with a 0.3 ml insert.	
Extracted soil/plant water ready for stable isotope analysis.	

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4. HOW TO PERFORM PRECISE SOIL AND SEDIMENT SAMPLING? ONE SOLUTION: THE FINE INCREMENT SOIL COLLECTOR (FISC)

L. MABIT, A. TOLOZA, K. MEUSBURGER, A.R. IURIAN, P.N. OWENS, C. ALEWELL, A. NIRSCHL

4.1. BACKGROUND

Soil as well as sediment analysis for agri-environmental research may require the ability to take precise depth increments. Existing equipment (e.g. soil augers and corers, classical bulk density cylinders, scraper plate systems) does not allow collecting millimeter fine increments of soil and there are no standardized existing guidelines for conducting such precise vertical sampling.

4.2. SCOPE AND FIELD OF APPLICATION

The Fine Increment Soil Collector (FISC), developed at the Soil and Water Management & Crop Nutrition Laboratory with the collaboration of the Technical Support Team from the Office of Safeguards Analytical Services, allows precise millimetre incremental sampling. It assists in the easy recovery of the soil/sediment by using a simple and robust extraction mechanism (see the following SOP).

The FISC has been designed specifically to support scientific investigation of shallow soil/sediment samples. Initially, the FISC has been developed to determine accurate soil profile content in Fallout Radionuclides [FRNs] (e.g. ^{7}Be, ^{137}Cs, $^{210}Pb_{ex}$, $^{239+240}Pu$), which are widely used for soil/sediment redistribution investigation [4.1-4.5].

This innovative item also offers significant potential advantage to study the depth distributions of contaminants, which can be linked to nuclear power plant accidental releases or industrial pollution. Prior to any remediation actions, this device hence offers the possibility to determine with precision the amount of soil to be removed.

Accurate incremental sampling through the FISC represents a potential valuable asset for various scientific disciples. For example, soil scientists and agronomists could obtain more precise soil physical and chemical characterization. For instance, soil profile bulk density can be measured at mm resolution.

4.3. ADVANTAGES AND POTENTIAL LIMITATIONS

The design of the FISC is freely available for the public, and can be found at the end of this section.

The FISC, which can be easily operated by only one person in the field or at the laboratory, permits also to adapt the amount of sample to be collected according to the analytical needs. The FISC is suitable for investigating most soil types. However, the material to be collected should be cohesive with reduced coarse soil particles content. Consequently, it will not be appropriate to investigate dry sandy soil and should not be recommended for soil with a gravel and/or stone content above 20%.

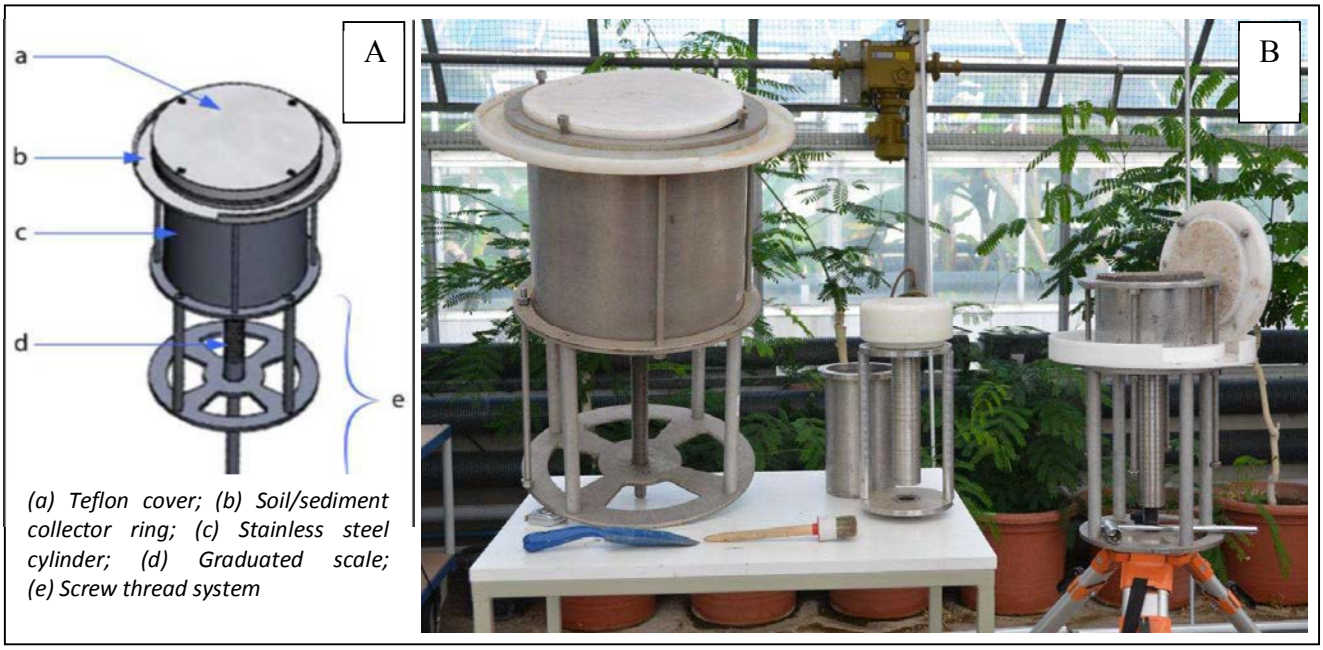
4.4. PRINCIPLE

Step 1: Hammering the FISC cylinder into the ground
Step 2: Inserting and levelling the FISC cylinder with soil surface
Step 3: Extracting the FISC cylinder from the ground
Step 4: Mounting the FISC cylinder on the tripod
Step 5: Checking the scale
Step 6: Determining the preferred depth of the soil collection
Step 7: Gathering the soil from the desired depth and putting the material collected into paper sacks

4.5. TYPICAL SAMPLE

Soils and sediments

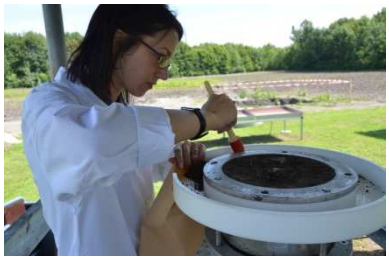
4.6. EQUIPMENT

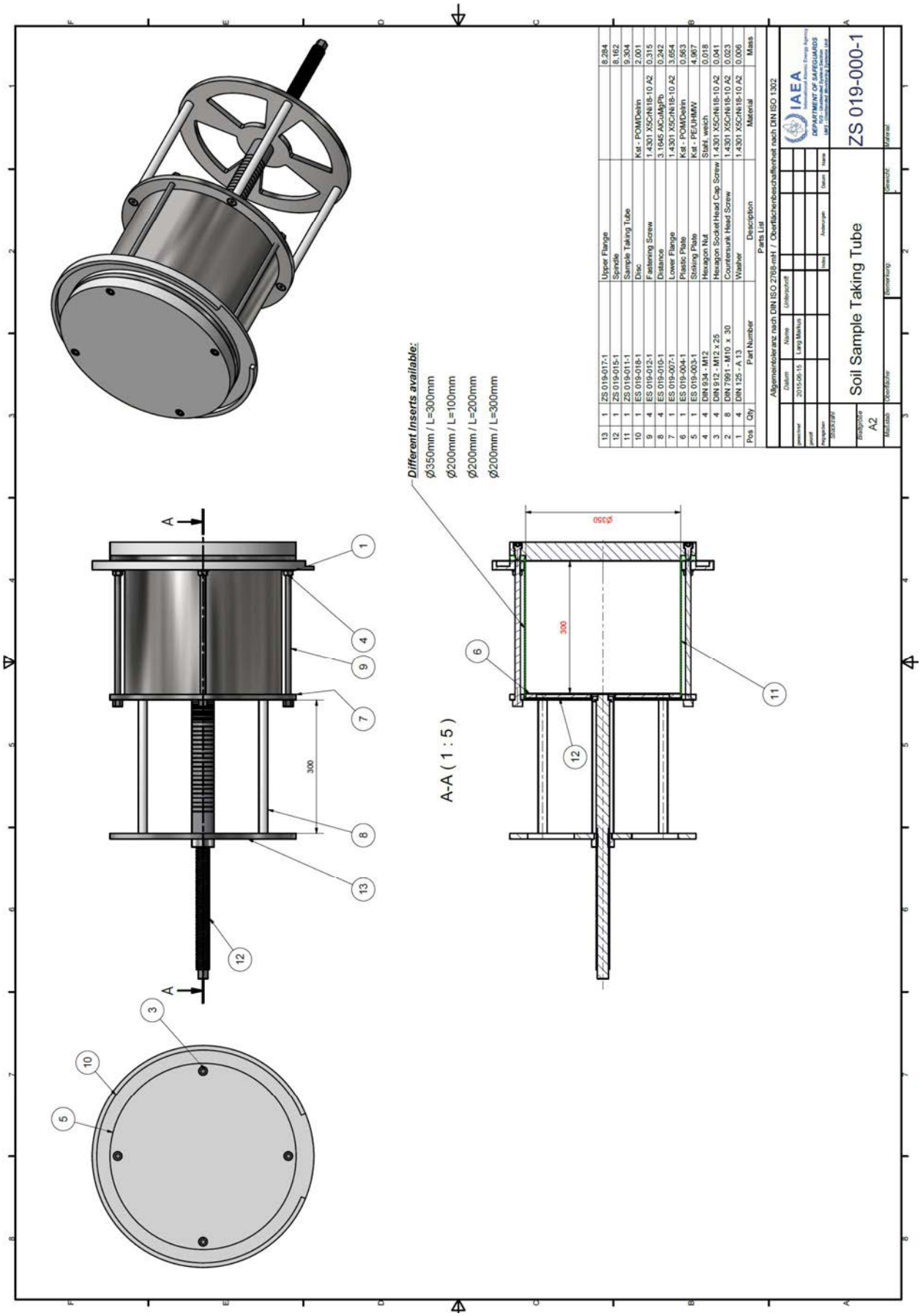


(a) Teflon cover; (b) Soil/sediment collector ring; (c) Stainless steel cylinder; (d) Graduated scale; (e) Screw thread system

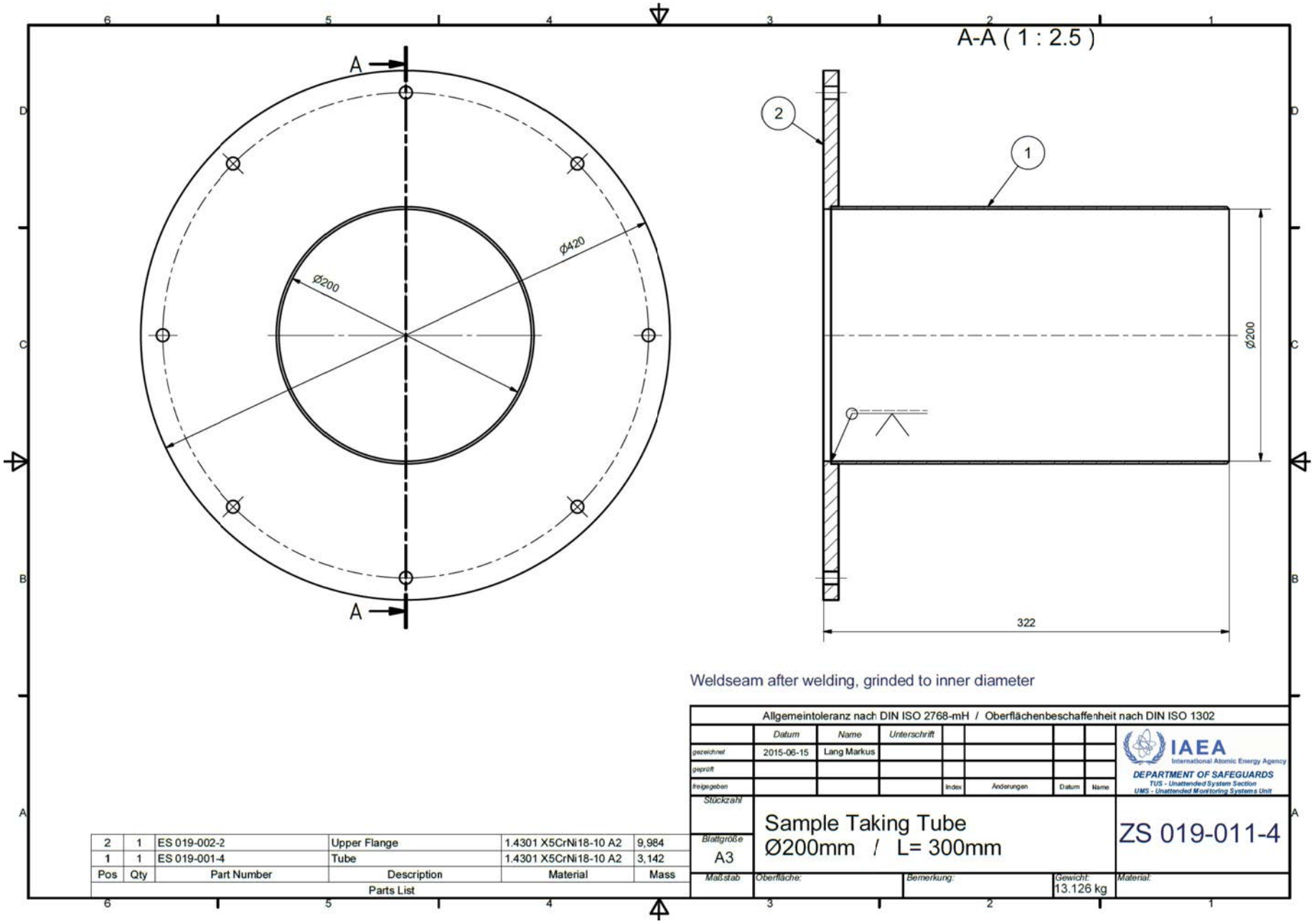
4.7. PROCEDURE

Step	Procedure	
Step 1 Hammering	The FISC is hammered into the ground using a plastic hammer and teflon covers for protecting the cylinder. 	
Step 2 Inserting and levelling	The FISC cylinder is inserted until it is completely into the soil and levelled with the surface.	
Step 3 Extracting	To extract the FISC cylinder, a shovel is used for digging. Then the FISC cylinder is removed slowly to avoid soil falling out.	
Step 4 Mounting	The FISC cylinder is mounted on the tripod making sure all screws are tightened.	

Step	Procedure	
Step 5 Checking scale	Check carefully the graduated scale, which will be the reference throughout the sampling of the soil increments.	
Step 6 Determining sampling depth increments	By rotating the screw thread system clockwise, the soil will emerge from the FISC cylinder at selected soil increment depths.	
Step 7 Collecting, packaging and labelling	To gather the soil sample, the emerged soil is slowly scraped of the cylinder using a field spatula. The soil is gathered with a brush in the collector ring.	
	The collected soil samples from the collector ring are placed in a paper sack or plastic bag and labelled correctly (indicating sample code and sampling depth).	



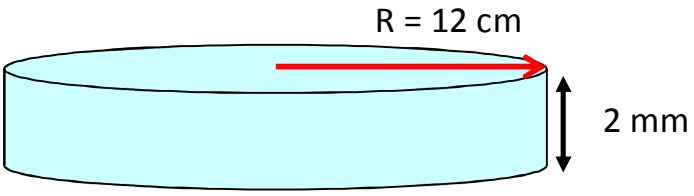
Pos	Qty	Part Number	Description	Material	Mass
		Parts List			
13	1	ZS 019-017-1	Upper Flange		8.284
12	1	ZS 019-015-1	Spindle		8.162
11	1	ZS 019-011-1	Sample Taking Tube		9.304
10	1	ES 019-018-1	Disc	Kst - POM/Delrin	2.001
9	4	ES 019-012-1	Fastening Screw	1.4301 X5CrNi18-10 A2	0.315
8	4	ES 019-010-1	Distance	3.1645 AlCuMgPb	0.242
7	1	ES 019-007-1	Lower Flange	1.4301 X5CrNi18-10 A2	3.654
6	1	ES 019-004-1	Plastic Piste	Kst - POM/Delrin	0.563
5	1	ES 019-003-1	Striking Plate	Kst - PEUHMW	4.967
4	4	DIN 934 - M12	Hexagon Nut	Stahl, weich	0.018
3	4	DIN 912 - M12 x 25	Hexagon Socket Head Cap Screw	1.4301 X5CrNi18-10 A2	0.041
2	8	DIN 7991 - M10 x 30	Countersunk Head Screw	1.4301 X5CrNi18-10 A2	0.023
1	4	DIN 125 - A 13	Washer	1.4301 X5CrNi18-10 A2	0.006



Pos	Qty	Part Number	Description	Material	Mass
2	1	ES 019-002-2	Upper Flange	1.4301 X5CrNi18-10 A2	9,984
1	1	ES 019-001-4	Tube	1.4301 X5CrNi18-10 A2	3,142

Parts List

4.9. CALCULATION



The volume (V) and dry weight (W) of material collected with a FISC having a radius (R) of 12 cm and an increment of 2 mm can be calculated as follows:

$$V = \pi \times R^2 \, h = 3.14 \times 144 \text{ cm}^2 \times 0.2 \text{ cm} = 90 \text{ cm}^3$$

$$W = V \times d = 108\text{g}$$

With density (d) of 1.2 g cm^3

REFERENCES TO SECTION 4

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5. GUIDELINES FOR MEASURING BULK DENSITY OF SOIL

C. RESCH, A. WAHBI

5.1. SCOPE AND FIELD OF APPLICATION

Bulk density is defined as the dry weight of soil per unit volume of undisturbed soil.

- Bulk density can be used to give an indication of the porosity and structure of the soil influencing O_2 and H_2O movement in the soil.
- Soils with a bulk density higher than 1.6 g/cm^3 may restrict root development.
- Bulk density is also a measurement of the degree of compaction of the soil.
- Bulk density increases with compaction and tends to increase with soil depth.
- Sandy soils tend to have higher bulk density (1.4–1.5 g/cm^3) than clay soils (1.2–1.3g/cm^3).

5.2. PRINCIPLE

The measurement of soil bulk density is carried out by collecting undisturbed soil samples through inserting metal rings (with a known volume) into the soil, and determining the weight of the collected soil after drying.

5.3. TYPICAL SAMPLE

Soil bulk density determination is most effective in moist soils without gravel. If the soil is very dry, it is recommended to increase first the moisture content of the soil. This can be done best by putting a bottomless drum filled with water on the soil surface for 24 hours.

5.4. REAGENTS

No reagents needed.

5.5. EQUIPMENT

Following equipment is needed (Fig. 5.1):

- Drying oven (set at 105°C);
- Balance, ±0.01g;
- Set of undisturbed soil sampling ring kits;
- Closed ring holder, bottom part for rings;
- Plastic mallet;
- Chisel for scraping off and clearing the soil sample;
- Shovel, spade, trowel;
- Lath;
- Folding yardstick or measuring tape.

FIG. 5.1. Equipment needed for the soil bulk density measurements.

For advice on equipment and estimated costs, please contact the Soil and Water Management & Crop Nutrition Laboratory, indicating the following reference "BULK DENSITY SWMCNL".

Website: www-naweb.iaea.org/nafa/about-nafa/biotechnology-lab.html
Email: Official.mail@iaea.org

5.6. PROCEDURE

a. Collecting soil cores at the soil surface

Step	Procedure
Select an undisturbed site and prepare a horizontal surface with a spade, trowel or chisel at the required sampling depth. Do not compact the soil!	

Place the soil sampling ring in the ring holder.	
A good sampling ring should have bevelled edges to minimize soil compaction during hammering. Plastic caps are ideal for sample retention.	
Use a folding yardstick or a measuring tape and a lath to check the depth.	
Push or hammer gently the sampling ring into the soil.	
Do not push the sampling ring in too far to avoid soil compaction.	

Remove the soil around the sampling ring slowly to ensure that the soil in the ring remains undisturbed or will not be loosened. Carefully remove the ring with the undisturbed soil.	
Cut off carefully any excess soil from outside the ring so that you end up with an even and flat surface on both sides.	 
Put the soil into a plastic bag, seal the bag, clearly identify the sample with a unique ID, and mark the sampling date, lower depth and location where the sample was taken. In case you have sufficient sampling rings, just put the ring with soil into a plastic bag, seal the bag, and mark the required information as indicated above.	
Dry the soil core at 105°C for 24–48 h or until the oven-dry weight is constant.	

b. Collecting soil cores from the deeper layers

Select an undisturbed site and prepare a horizontal surface with a spade, trowel or chisel at the required sampling depth. Do <u>not</u> compact the soil!	
Prepare a sampling ring.	
Place the soil sampling ring in the ring holder.	
Use a folding yardstick or a measuring tape and a lath to check the depth.	

Push or gently hammer the steel ring into the soil.	
Carefully pull out the ring holder.	
It is also possible to take a sample from the side of the pit.	
Push or hammer gently the sampling ring into the soil.	

Remove the soil around the sampling ring slowly to ensure that the soil in the ring remains undisturbed or will not be loosened. Carefully remove the ring with the undisturbed soil.	
Carefully remove any exceeding soil from outside the ring so that you end up with an even and flat surface on both sides.	
Close the sampling ring with the plastic lids.	
Put the soil into a plastic bag, seal the bag, clearly identify the sample with a unique ID, and mark the sampling date, lower depth and location where the sample was taken. In case you have sufficient sampling rings, just put the ring with soil into a plastic bag, seal the bag, and mark the required information as indicated above.	
Dry the soil core at 105 °C for 24–48 h or until the oven-dry weight is constant.	

5.7. CALCULATION

a. *Soil volume*

The undisturbed soil volume is the volume of the sampling ring.

To calculate the volume of the soil sampling ring, following measurements are needed:

- Measure the height (H) of the ring with a glide gauge in cm to 0.1 mm precision
- Measure the inner diameter (D) of the ring with a glide gauge in cm to 0.1 mm precision
- Ring volume (cm^3) = $(D/2)^2 \times \pi \times H$ (where π is 3.1416)

Example:

If the diameter of the ring is 7 cm and the height is 5 cm, then the ring volume is calculated as follows: $(3.5)^2 \times \pi \times 5 = 192.42$ cm^3

b. *Dry soil weight and volumetric water content calculation*

To calculate the dry weight of the soil and, if required, the volumetric water content, following steps should be followed:

- Weigh an oven-proof container in grams (W1)
- Carefully remove all soil from the bag into the container and weigh them (W2). Dry the soil in an oven at 105ºC for 24–48 h or until the oven-dry weight is constant.
- When the soil is dry, weigh the sample (W3)
- Dry soil weight (g) = W3 – W1
- Volumetric water content (g water/ g dry soil) = (W2–W3) / (W3–W1)

5.8. BULK DENSITY

The final calculation of the bulk density (g/cm^3) is as follows:

Bulk density = Dry soil weight (g) / Undisturbed Soil volume (cm^3) (5.1)

5.9. NOTE

It is recommended to avoid taking samples when the soil is very dry (near wilting point) or very wet (saturation), as the samples could be compacted.

FURTHER READING

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6. ADDITIONAL INFORMATION ON METHODOLOGY

6.1. SECTION 3: EXTRACTION OF WATER FROM SOIL AND PLANT SAMPLES FOR $^{18}O/^{16}O$ AND D/H ISOTOPE RATIO MEASUREMENTS

Below is the outline of the study carried out for obtaining and validating the above SOP results. Two different soil types: Ebendorf silty clay loam and Reisenberg sandy loam were used. They were adjusted to two different soil moistures: close to field capacity (FC) and near permanent wilting point (PWP). This is equivalent to 0.1 bar for Ebendorf soil and 0.3 bar for Reisenberg soil for the FC study; and equivalent to 12.5 bar for the Ebendorf soil and 1 bar for the Reisenberg soil, see Table 6.1 below for their physical properties.

TABLE 6.1. CHARACTERISTICS AND MOISTURE LEVEL OF TEST SOILS

Soil type	test	% sand	% silt	% clay	Applied pressure	% moisture
Ebendorf silty clay loam	wet	16	57	27	0.1 bar	33.3
	dry	16	57	27	12.5 bar	19.8
Reisenberg sandy loam	wet	65	28	7	0.3 bar	18.7
	dry	65	28	7	1.0 bar	12.6

In order to avoid fractionation of delta oxygen-18 ($\delta^{18}O$) and delta hydrogen-2 ($\delta^{2}H$) during the moisture adjustment, the soil moisture at both levels was adjusted using a ceramic pressure plate extractor (Soil Moisture Equipment Corp., Santa Barbara, California, USA).

The two bulk soils were initially air dried and sieved to 2 mm. About 500 g of each soil type was equilibrated with one liter of water of known $\delta^{18}O$ and $\delta^{2}H$ content ($\delta^{18}O$ = –9.28 ‰; $\delta^{2}H$ = –67.76 ‰). The slurry, made by stirring the soil-water suspension thoroughly, was left standing covered and light-protected for two days at room temperature (25°C). After the equilibration time the excess water was decanted and the wet soil poured into three metal rings (55 mm diameter, 40 mm height) for each soil type (three replicates) and placed on a ceramic pressure plate. Water was removed from the soil by applying the appropriate pressure until no more water was found flowing out of the pressure chamber (Fig. 6.1).

After moisture adjustment, five subsamples of 3–5 g per soil type and per moisture level were taken from each of the metal rings using an auger (10 mm diameter) and weighed into tared 100 × 16 mm glass culture tubes, and the extraction procedure in the SOP was used to extract water from the soil samples

FIG. 6.1. Pressure plate was used to adjust the soil moistures for the two soils.

Three replicate samples per moisture level were prepared for water extraction at five different extraction times (15, 30, 60, 120 and 180 min). Fifteen samples of each soil were put on the block heater and after the corresponding extraction time three replicates of each extraction time were removed, vented to air by opening and closing the stopcock valve, and left standing in a rack until the ice in the water trap had melted. Then the sample and water tubes were disconnected from the glass assembly and closed immediately with screw caps. All tubes were weighed after having reached room temperature (25°C).

Water was transferred from the trapping tubes into 2-ml auto-sampler glass vials (32 × 12 mm) using disposable pipettes. For water volumes of less than 0.5 ml, vials with a 0.3 ml insert were used. The samples were analysed for $\delta^{18}O$ and $\delta^{2}H$ using a cavity ring down laser spectrometer (CRDS Picarro Isotopic Water Analyser L2130-i), as described earlier.

After extraction the soil dry weight was recorded and the tubes containing soil were placed on a separate block heater at 110°C and closed with silicone stoppers holding two syringe needles. One was connected to an aquarium pump blowing dry air onto the soil using a molecular sieve filter. The other needle was for release of the moist air. With this special drying procedure all remaining moisture could be removed overnight. After cooling to room temperature the soil samples were again weighed and the difference between soil weight after extraction and soil weight at "complete dryness" was determined.

Measurements and calculations:

$$\% \text{ water recovery} = (S_{wet}-S_{dis})/(S_{wet}-S_{dry}) \times 100\% \qquad (6.1)$$

Where: S_{wet} is the weight of wet soil
S_{dis} is the weight of soil after distillation
S_{dry} is the weight of soil after oven drying (48h at 110°C)

For stable isotope analyses a water recovery of at least 98% is required [6.1].

Main results of validation: (See [6.2] for more detailed information)

A simple, fast, affordable and portable vacuum distillation method for extraction soil samples for isotopic analysis was developed. The extraction time (with 98% water recovery) was 30 minutes for sandy soil and 120 minutes for clay soils (Table 6.2 and Fig. 6.2).

The method does not require the use of liquid nitrogen or dry-ice hence can be adapted easily for developing countries. The storage vials can be connected directly to minimize the transfer of sample and hence fractionation of the isotopic signature of the water.

Other advantages of this system are the portability, the relatively low price (9000 Euro for a setup of 12 samples) and the high throughput (12 to 15 samples at a time).

Precision (Standard Deviation of measurements) is:

- Sandy soil: ±0.09‰ $\delta^{18}O$ and 0.43 ‰ $\delta^{2}H$
- Clay soil: ±0.13‰ $\delta^{18}O$ and 0.83 ‰ $\delta^{2}H$

TABLE 6.2. MINIMUM DISTILLATION TIME REQUIRED TO ACHIEVE A WATER RECOVERY OF > 98 PERCENT AND THEIR CORRESPONDING $\delta^{18}O$ AND $\delta^{2}H$ PRECISIONS AT ONE STANDARD DEVIATION

Soil type		**Time (min)**	**Precision (1σ SD)**	
			$\delta^{18}O$ (‰)	**$\delta^{2}H$ (‰)**
Clay soil	Wet (near FC)	≥ 30	± 0.09	± 0.58
	Dry (near PWP)	≥ 120	± 0.13	± 0.83
Sandy soil	Wet (near FC)	≥ 15	± 0.11	± 0.51
	Dry (near PWP)	≥ 60	± 0.08	± 0.43

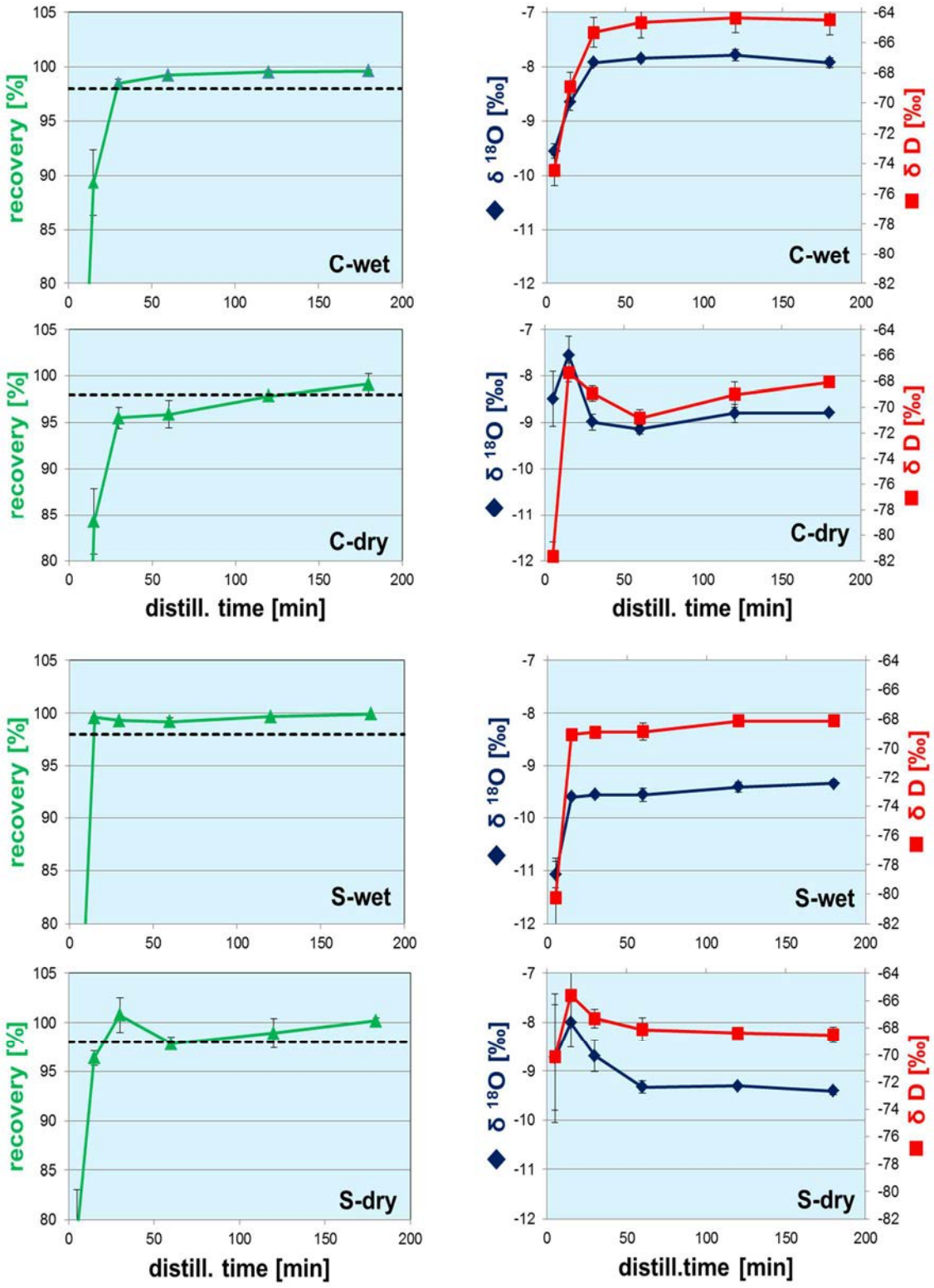

FIG. 6.2. Extraction time for 98 percent water recovery of clay soil (C) and sandy soil (S) under wet and dry conditions (left); corresponding $\delta^{18}O$ and $\delta^{2}H$ values under various extraction times are also shown (right).

6.2. SECTION 5: GUIDELINES FOR MEASURING BULK DENSITY OF SOIL

Common sources of error when collecting soil cores for bulk density are:

- Disrupting the soil while sampling;
- Inaccurate trimming;
- Inaccurate measuring of the volume of the ring;
- Gravel can make trimming the core difficult and give inaccurate values, so it is best to take more samples to decrease error in this way;
- The below ring samplers are not recommended as shown below (Fig. 6.3).

FIG. 6.3. Ring samplers and its parts.

The ring sampler illustrated above has the advantage of obtaining two soil cores. However, the large cross-sectional area of the beveled cutting edge causes soil compression ahead of the sampler in many soils. The resulting cores may appear undisturbed even though the bulk density has been increased by compression. It is difficult to remove the screw-on sampler head from the cylindrical body in order to check for compression before removing the sampler from the soil. Samplers of this type are therefore not recommended.

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