



Contract No: 518074

PROMOTE

Efficiency control and performance verification
of improved approaches for soil-groundwater protection and
rehabilitation

Specific Targeted Research Project
Thematic priority: Global Change and Ecosystems

04.16 Soil coring kit, Eijkelkamp- Verification report

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Dissemination Level

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ENVIRONMENTAL TECHNOLOGY VERIFICATION (ETV) I. FACT SHEET

TECHNOLOGY TYPE:	Soil Coring kit for volatiles in soil
APPLICATION:	Soil sampling for determination of the presence of very volatile components with prevention of volatilization and oxidation as much as possible.
TECHNOLOGY NAME:	04.16 Coring tubes for volatiles in soil
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What is ETV

The Environmental Technology Verification (ETV) system currently prepared by the European Commission as part of the European Technologies Action Plan, is designed to support the environmental industry by providing credible and independent verification of technology performance claims. The objective of ETV is to enhance the confidence of investors and potential users in promising innovative eco-technologies. PROMOTE is one of 4 research projects funded under FP 6 which are designed to explore the characteristic features of an ETV on different technology sectors. The overall aim of the PROMOTE project is to set up a complete efficiency control and performance verification system for soil-groundwater protection and rehabilitation technologies. The system is based on a stepwise concept including generic testing at reference site and field site scale.

The given technology fact sheet, as an extended summary of the verification report, has been elaborated within PROMOTE, following the procedures described by PROMOTE and further laid down in the CEN Workshop Agreement CEN/WS 32 *Environmental technology verification – Soil and groundwater site characterization, monitoring and remediation technologies* as a first standardisation document in this field.

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Technology description

04.16 soil coring kit (Soil corer) contains: an Edelman auger to pre-bore or clean the bore hole, in various types of soil, a stainless steel soil coring apparatus with a number of sample tubes, filling blocks and insulation plates. Further bottom caps, a sample extruder and maintenance material. The complete set is packaged in an aluminum transport case. It is possible with this set to take samples to a depth of 5 meters.

The samples are taken with a small coring apparatus fitted with thin-walled stainless steel sample tubes. The sampling tubes preferably are pushed or hammered into the soil.

After taking the sample, the sample tubes can be locked and cooled for transport to the laboratory.

Here sub-samples can be taken from the sample tube, applying a small gauge or apple-corer.

The samples can also be removed using an extruder. After decontamination, the equipment can be used again.

The sample has a volume of 226 ml. The set therefore also is suitable for the determination of volume percentages of humidity in samples from undisturbed soil.



04.16 Soil coring kit for chemical soil research from Eijkelkamp



Sample Tubes with caps and filling blocks



Coring apparatus

Relevance

The application field for the soil corer is for research into the presence of very volatile components, such as benzene, toluene, xylene and chlorinated hydrocarbon. It is also applicable for determining the volume percentage of moist.

With the soil coring kit, the samples are not exposed to air during sampling and transport. Therefore, the volatilization and oxidation of components in the soil samples can be prevented as much as possible. The sample under no condition comes in touch with synthetic material.

Because of the small penetration resistance and the hammer with nylon heads, the soil coring set is also suitable for harder soils. For application in very loose soils a sampler tube with core catcher and liner is available. The auger fitted with valve ensures, through creating a vacuum that the sample remains in the tube during extraction.

With the soil coring kit the transportation of samples in pots is not necessary, thus usage of methanol in the field is not needed. However, it can be combined with the methanol method.

Performance claim

Eijkelkamp defines one claim for the Soil coring kit:

1. The Eijkelkamp sampling protocol is as good as direct field extraction in methanol or water. The losses in VOC for the Eijkelkamp sampling protocol will not be more than 10% in comparison to the standard methods.

Test description

To verify the claim a literature study and tests in the laboratory under controlled conditions have been carried out.

First, a literature study was undertaken, analysing and comparing the different methods used for volatile hydrocarbons. On basis of the results of the literature study, tests were set up for proving the claim. The test was to determine VOC (volatile organic compound) losses upon storage.

Three different methods were tested, with each different storage temperatures and storage times. Metal cylinders were filled with sand, simulating a compacted non-contaminated sand layer. A fixed spiking solution, containing 6 volatile aromatic organic compounds and 2 chlorinated organic components, was added to the sand.

The methods tested were:

1. Eijkelkamp (AP04)
 - a. Immediately after spiking
 - b. After 20hours at 10°C storage
 - c. After 48h at 4°C storage
 - d. After 48h at -20°C storage
 - e. Cryogen grinding method

2. EPA 5035A
 - a. 110ml methanol, 20h at 10°C
 - b. 110ml methanol, 48h at 4°C
 - c. 400ml methanol, 20h at 10°C
 - d. 400ml methanol, 48h at 4°C
 - e. Water, 20h at 10°C
 - f. Water, 48h at 4°C

The tests were prepared at Deltares, Stieltjesweg 2, 2628 CK, Delft and analysed by Alcontrol, Steenhouwerstraat 15, 3194 AG Hoogvliet. Dianne den Hamer and Jaap-Willem Hutter did the implementation of the tests.

Results and verification

For each different test method three samples were spiked and analysed. The methods cryogen grinding and 110ml methanol were not taken into account with the verification, because the VOC loss was too high. The results from the Eijkelkamp method stored at 4°C and -20°C are comparable with the results from EPA 5035A with water and with 400ml methanol. The Eijkelkamp results differ less than 10% with the EPA 5035A 400ml methanol method (which had the best results from the different standard methods).

The results of the test validate the claim. The Eijkelkamp method, where samples were stored at low temperatures, showed comparable or better results with the standardised VOC analyse methods, with a maximum deviation of 10%.

Conclusion of verification: The test results provided by verification in lab tests prove the claim defined for the 04.16 soil coring kit. The VOC loss when the 04.16 soil coring kit and protocol is used is comparable with standard VOC analytical methods.

Date:

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The elaboration of this fact sheet was funded by the 6 Framework Programme of the European Commission within the project PROMOTE.

ENVIRONMENTAL TECHNOLOGY VERIFICATION (ETV)

III. Verification report

ABSTRACT: PROMOTE is one of 4 research projects funded by EC under FP 6 which are designed to explore the characteristic features of an ETV on different technology sectors. The given verification report for 04.16 soil coring kit of Eijkelkamp, Giesbeek, The Netherlands, has been elaborated within PROMOTE, following the procedures described by PROMOTE and further laid down in the CEN Workshop Agreement CEN/WS 32: Environmental technology verification – Soil and groundwater site characterization, monitoring and remediation technologies as a first standardisation document in this field.

Eijkelkamp defined one claim to describe performance of the 04.16 soil coring kit. To verify this claim a literature study and tests in the laboratory have been carried out under controlled conditions. The test in the reference lab was focused on determining the VOC (volatile organic compound) losses during storage, either preceded by extrusion in methanol or water, for a mixture of VOC's over time. The tests consider the requirements on quality assurance of PROMOTE ETV.

Evaluation of test results reveal that the data obtained for the 04.16 soil coring kit prove the claim.

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1. Introduction

The Environmental Technology Verification (ETV) system currently prepared by the EC as part of the European Technologies Action Plan is designed to support environmental industry by providing credible and independent verification of technology performance claims. Convincing the market of the merits of an environmental technology is often very difficult for producers, and especially for small and medium sized enterprises. The objective of ETV is to enhance the confidence of investors and potential buyers in promising innovative eco-technologies, thus facilitating their entrance into the EU and international marketplace.

PROMOTE is one of 4 RTD projects funded under FP 6 which are designed to explore the characteristic features of ETV in different technology sectors. The overall aim of the PROMOTE project is to elaborate a proposal for an environmental technology verification system for soil-groundwater characterisation, monitoring and remediation technologies. The system is based on a stepwise concept including generic testing at reference site and field site scale.

The given verification report has been elaborated within PROMOTE, following the procedures described by PROMOTE and further laid down in the CEN Workshop Agreement CEN/WS 32 *Environmental technology verification – Soil and groundwater site characterization, monitoring and remediation technologies* as a first guidance document in this field. The focus of the related research was to test the workflow of the proposed procedure, including the elaborated protocols and guidelines related to the design, implementation and interpretation of tests, thus testing the methodological concept of verification itself.

In addition, the aim of this verification exercise was to test a specific product under ETV-conditions, thus providing the vendor with an independent proof of the claims made for his product and to publish a best practise example for verifying a specific product according to CEN/WS 32.

The verification has primarily been aimed at testing protocols within budgetary and time-constraints of the PROMOTE-project and subsequently produce a defensible report that also is of use to the vendor of the technology.

The vendor (Eijkelkamp Agrisearch Equipment BV) and members of the verification board have discussed the claim for the 04.16 coil coring kit. Initially the expectation was that the claim could be verified by existing literature data. However, this was shown not to be possible since the literature contained insufficient vendor and trademark/technology type specific data. The literature provided test data for similar sampling techniques that were helpful in developing the necessary test set up. The test design was developed by the vendor, lab experts and members of the verification board based on the literature relating to the same technique of sampling.

To verify the claim given by the vendor for the Soil core tests had been carried out in the laboratory within Deltares and the laboratory of Alcontrol laboratories.

Experts and institutions involved in this verification:

Role in the process	Institution	Address institution	Responsible actor	Date for tests
Verification expert	Deltares , unit Geo Engineering, Department FOW	P.O. Box 177 2600 MH Delft The Netherlands Stieltjesweg 2 2628 CK Delft	-Dr. Derk van Ree, Derk.vanRee@deltares.nl T+31(0)15 2 69 37 04 -Ester Marsman, Ester.marsman@deltares.nl T+31(0)15 2 69 36 25 -Cindy Braat Msc, cindy.braat@delatres.nl T+31(0)15 2 69 37 49	
Lab tests	Deltares , unit Geo Engineering, Department GeoLab	P.O. Box 177 2600 MH Delft The Netherlands Stieltjesweg 2 2628 CK Delft	-Dianne den Hamer Dianne.denhamer@deltares.nl T+31(0)15 2 69 35 72 T+31(0)15 2 69 35 27	August 2008
	Alcontrol Laboratories	P.O. Box 240 3190 AE Hoogvliet The Netherlands Steenhouwerstraat 15 3194 AG Hoogvliet	-Jaap-Willem Hutter, j.hutter@alcontrol.nl T+31(0)10 2 31 47 00 -Marjanne van den Berg	August 2008
Vendor, Stakeholder perspectives	Eijkelkamp Agrisearch BV	P.O. Box 4 6987 ZG Giesbeek The Netherlands Nijverheidsstraat 30 6987 EM Giesbeek	-Gerard van Dijk, G.vanDijk@eijkelkamp.com T+31(0)31 3 88 02 62	

DISCLAIMER

The verification of a technology means always the specific product of the technology provider, not a group or class of technologies.

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2. Product description

2.1 Name of the technology and vendor

Name of the technology: 04.16 Soil coring kit

Vendor's contact information: Eijkelkamp Agrisearch Equipment, Postbox 4, 6987 ZG, Giesbeek, The Netherlands

2.2 Brief description of the technology

Soil sampling for the determination of the presence of very volatile components such as benzene, toluene, xylene and chlorinated hydrocarbons should be executed with samplers that prevent the sample from being exposed to air. Preferably, the sampling must be done without disturbance of the soil aggregates to prevent mixing with air.

These conditions must be maintained during transport to the laboratory.

With the special coring kit (figure 1), volatilization and oxidation of components in soil samples can be prevented as much as possible. The sample under no condition comes in touch with synthetic material. The method meets the NEN 5743 norm (soil or sediment sampling with volatile components).



Figure 1: 04.16 Soil coring kit for chemical soil research from Eijkelkamp



Figure 2: Sample Tubes with caps and filling blocks



Figure 3: Coring apparatus

The sample has a volume of 226 ml (figure 2). The set therefore also is suitable for the determination of volume percentages of humidity in samples from undisturbed soil.

The samples are taken with a small coring apparatus fitted with thin-walled stainless steel sample tubes (figure 3). The sampling tubes preferably are pushed or hammered into the soil (figure 4 and 5).

After taking the sample, the sample tubes can be locked and cooled for transport to the laboratory. Here sub-samples can be taken from the sample tube, applying a small gauge or apple-corer. The samples can also be removed using an extruder. After decontamination, the equipment can be used again.

The set contains among other things: an Edelman auger to pre-bore or clean the bore hole, in various types of soil, a stainless steel soil coring apparatus with a number of sample tubes, filling blocks and insulation plates.

Further bottom caps, a sample extruder and maintenance material. The complete set is packaged in an aluminum transport case. It is possible with this set to take samples to a depth of more than 5 meters.



Figure 4: Surface sampling. Step 1-Push the core apparatus with sample core in the soil and take the sample from the soil. Step 2-Add the filling blocks in the sample tube and cap the sample tube.

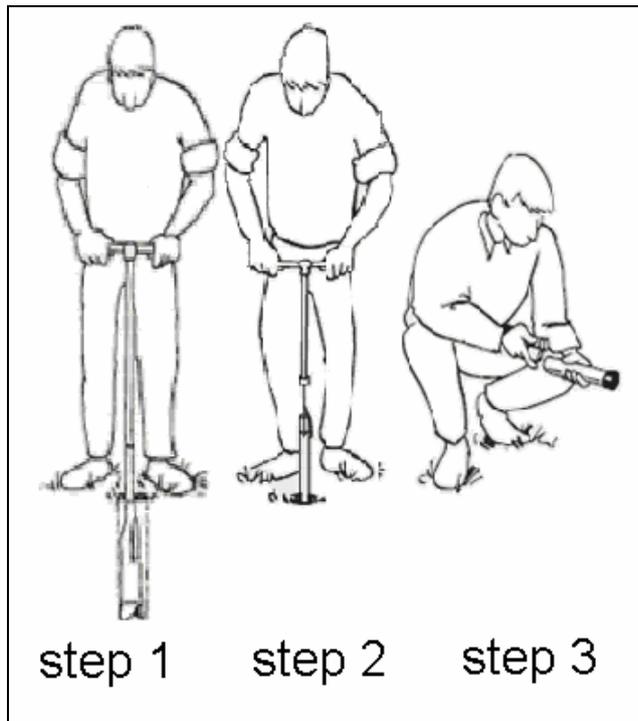


Figure 5: Sampling under surface. Step 1: Pre-bore to the required depth with the edelman auger bore. Next the Riverside auger can be used to make the bottom of the hole flat. Step 2: Push the core apparatus with sample core in the soil and take the sample from the soil. Step 3: Add the filling blocks in the sample tube and cap the sample tube.

Application field:

- Soil sampling for research into the presence of very volatile components such as benzene, toluene, xylene and chlorinated hydrocarbon according to NEN 5743.
- Soil sampling for determining the volume percentage of moist.

Advantages:

- Sampling system that meets the norm for determination of volatile components in soil or sediment.
- Suitable for the determination of the volume percentage of humidity.
- No exposure of the sample to air.
- Transportation of samples in pots is not necessary.
- Because of the small penetration resistance and the hammer with nylon heads, the set is also suitable for harder soils.
- The auger fitted with valve ensures through creating a vacuum, that the sample remains in the tube during extraction.
- For application in very loose soil a sampler tube with core catcher and liner is available.
- Prevents loss of volatiles during sampling
- Prevents loss of volatiles during transport
- No hassle in the field with methanol
- Can be combined with methanol method
- Tubes can be decontaminated over and over

More about soil core method:

For the operation instructions, please refer to:

- Operating Instructions 04.16 soil coring kit for chemical soil research, Eijkelkamp

For the summary of efficiency, please refer to:

- Gerard P. van Dijk, ISO/TC 190/SC2 N 267, Letter for ISO/TC 190-SC2+3, 1 August 2007.

Detailed product information in Annex 1.

3. Claims

3.1 Claim

A claim defines the effects foreseen by the vendor of an environmental technology on the target(s) in the matrix (matrices) of the intended use of a technology (according to CEN Workshop Agreement CEN/WS 32).

The following claim should be proven by literature study and tests of this ETV report:

1. Sampling and field cooling or freezing with subsequent extraction and analysis within 48 hours (before extraction), when cooled, is as good as direct field extraction in methanol or water. (The system of field cooling allows less evaporation of volatiles than field extraction.)
The VOC loss by the soil coring kit does not deviate more than 10% of VOC loss of the reference methods.

3.2 Claim development

Background information

If during a spill organic compound leaks into the soil, it is necessary to know whether the site has to be remediated, how much has to be remediated, which compounds have to be remediated and at which location the organic compound is present.

To investigate this, soil samples have to be taken from the site location and sent to a laboratory for analyses on different types of contaminants. One of the types of contaminants is volatile organic compounds (VOC). From the moment the samples are taken until the samples are analyzed they have to be handled in such a way to prevent VOC disappearing from the sample. When the sample is preserved with a leak to the open air or is exposing to the open air and not treated against evaporation, VOC will quickly disappear from the sample. This evaporation of VOC increases with the exposure time and size of exposure leak.

These losses will lead to lower analytical results of VOC concentrations. In combination with the uncertainties, this will lead to a lower estimation of the VOC concentration at the remediation site potentially underestimating risks and possibly a sub optimal remediation design. This will lead to financial losses for remediation companies.

Data collection chain

The process between the sampling and the VOC concentration results is divided into the following steps:

1. Sample is taken from the soil
 - a. Drilling, Penetration, Tool placement
 - b. Sampling
2. Pre treatment of sample with or without extruding to a vial (with or without extraction with methanol) and conditioning.
3. During storage and transport, conserving, maintaining of temperature. Keeping transport and storage time within limits.
4. Measurement in the laboratory of the VOC concentrations
5. Data processing

Drilling/ penetration/ tool placement	Sampling	Pre-treatment Conditioning	Extraction	Conserving storage and transport	Measurement	Data collection Data evaluation	Translation into information Writing report
Step 1		Step 2		Step 3	Step 4	Step 5	

Figure 6: Data collection chain

Conservation methods and regulations

The current Methods used for sampling, pre-treatment, conservation and transport are described in:

- EPA Method 5035A “closed-system Purge-and-trap and extraction for volatile organics in soil and waste samples”
- NEN 5743 “Soil. Sampling of soil and sediments for the determination of volatile compounds.

The claim of the Quick scan is suggesting two methods for testing the claim:

- EPA Method 5035A “closed-system Purge-and-trap and extraction for volatile organics in soil and waste samples”
- NEN 5743 “Soil, Sampling of soil and sediments for the determination of volatile compounds.

In these methods, more pretreatment and conditioning options are possible for samples where the volatile organic compound are measured. A schematic representation of all options for these methods are given in table 1.

4. Literature Study

Literature review

During literature review, five articles about VOC losses with different sampling (or spiking), preservation, storage and analysing methods were studied. Some parts of the data collection chains are similar to the methods described in NEN 5743 and Method EPA 5035A. The VOC losses at these parts are shown in table 1.

The experiments in the articles can be separated roughly in the following two methods:

1. Method without methanol preservation in the field, without extruding of sample from core to vial in the field
2. Method with methanol preservation, in the field or after 30 min, with extruding of sample from core to vial, in the field or after 30 min.

In the following articles experiments are done using method 1:

1. Siegrist R.L et al., 2006, “Quantifying PCE and TCE in DNAPL Source Zones: Effects of Sampling Methods Used for intact Cores at Varied Contaminant Levels and Media Temperatures”, Ground Water Monitoring & Remediation 26. (*first experiment*)
2. Soilcore Inc., n.d. Analytical report: SoilCore’s New Sampler for VOC discrete Soil Sampling. From: <http://www.soilcore.com/study1.htm> [cited: 2007-08-13]
3. Soilcore Inc., n.d. Analytical report: Study of Soilcore™ Capability to contain BTEX/MTBE for period of 48 hours. Available from: <http://www.soilcore.com/study2.htm>. [cited: 2007-08-13]
4. Soilcore Inc., n.d. Analytical report: Study to document Soilcore™ discrete sampling device as an equivalent and an alternative to En Core™ in EPA method 5035. Available from : <http://www.soilcore.com/study3.htm> [cited: 2007-08-13]

The following articles contain experiments done with method 2:

1. Siegrist R.L et al., 2006, “Quantifying PCE and TCE in DNAPL Source Zones: Effects of Sampling Methods Used for intact Cores at Varied Contaminant Levels and Media Temperatures”, Ground Water Monitoring & Remediation 26. (*second experiment*)
5. Sorini et al., 2002. Evaluation of VOC loss from soil samples: Extrusion into empty VOA vials, refrigerated storage”

Results of the average VOC losses from the test methods in the articles, similar to NEN 5743 and EPA method 5035A, are shown in table 1.

Requirements of literature for verification

Not all literature about the technique/(equivalent) equipment for verification is suitable. The literature is suitable for verification of equipment when for the following requirements are fulfilled:

- The tests performed in the literature have to be performed with the equipment of the vendor. The ETV verification system is equipment and trademark specific.
- The results from reliable experiments in literature have to verify the claim qualitatively and quantitatively.

- The tests in the literature have to be executed by an independent, recognised organisation and not by the producer.
- Results from different literature sources, which have to be compared, have to be performed by the same analytical method and laboratory.

When existing data or literature are used for the ETV verification, the information should be incorporated the generic test design protocol. The test implementation protocol/report can then be filled with the results from the literature study. Thus the verification can be done based on existing literature and test data including conclusions, as long as these data fulfil the requirements of the ETV-system and protocols.

Verification of the Eijkelkamp soil coring kit based on literature

It is not possible to verify the claim based on these data, because the literature is about the technique in general and not specific about the 04.16 soil coring sampler of Eijkelkamp. Because verification is equipment specific, all tests (for the different methods) were done with the 04.16 soil coring kit of Eijkelkamp to verify the claim.

Information from literature used for test design

- Amount of VOC loss depends on the concentrations (article 1). Methanol method (EPA 5035A) is only used for high VOC concentrations, therefore high concentration samples are tested.
- Losses can take place by incomplete extraction of PCE and TCE to the methanol before analyses (article 1).
Because extraction was performed in a certified lab, this would not influence the methanol extraction method.
- Storage time less than 63 hours gives no significant difference in VOC loss (article 2, 3, 4 and 5). Not many different storage times have to be tested for verification.
- Different types of soil samplers show different quantities of VOC loss (article 4). For all the verification tests the same core samples are used.
- Different VOC compounds show different VOC losses (article 1, 4 and 5). Therefore, different VOC compounds were tested for the verification.
- There is a positive relation between increase of VOC losses and time before preserved with methanol (air exposure) (article 1 and 5).
- Increase of temperature, before preservation with methanol, causes an increase of VOC loss (article 1). This indicates that the temperature must not vary much during extrusion and spiking for the ETV verification.

Table 1 shows the different methods which were tested.

Table 1: Overview of methods described in NEN 5743 and in Method 5035A, including additional information obtained from the articles. Grey arrows indicate methods which are performed for the ETV verification.

Method		1 Methanol method with extruding of sample from core to a vial. VOC dilute in water miscible solvents (VOC >200 µg/kg & 0.5 to 200 µg/kg)		2 Method without methanol preservation in the field with soil core without extruding of sample from core to vial. VOC dilute in water miscible solvents (VOC >200 µg/kg)		3 Method without methanol preservation in the field with soil core with extruding of sample in to vial. VOC dilute in water miscible solvents (VOC >200 µg/kg)		4 Method with reagent water with soil core with extruding of sample to vial. Low concentrations (VOC =0.5 to 200 µg/kg)		
Regulation document		EPA Method 5035A		NEN 5743 EPA Method 5035A		EPA Method 5035A		EPA Method 5035A		
Steps in data collection chain:	1. Drilling, Penetration, Tool placement, sampling	Sampling is done by a core sampler								
	2. Pretreatment with or without extraction	<p>Sample in the core is extruded to a vial with methanol and the vial is closed with a screw cap <i>No loss for PCE and TCE* (storage overnight at 20 °C) (article 1)</i></p>	<p>Sample in the core is extruded to a vial <u>without</u> methanol and the vial is closed with a screw cap</p>	<p>Core is capped <i>33% TCE loss* (high conc.) 36% PCE loss* (high conc.) 31% TCE loss* (low conc.) 64% PCE loss* (low conc.) (after 16 to 24 h) (article 1)</i></p> <p><i>3,6 % VOC loss* (after 63 h) (article 3)</i></p> <p><i>10% and 13% VOC loss* (after 63 h) (article 4)</i></p>	<p>Sample in the core is extruded to a vial <u>without</u> methanol and the vial is closed with a screw cap</p>	<p>Sample in the core is extruded to a vial with reagent water and the vial is closed with a screw cap</p>	<p>Sample in the core is extruded to a vial with reagent water and 1 g NaHSO₄ and the vial is closed with a screw cap</p>			
	3. Conditioning during storage and transport Temperature (T) ± 2 °C Maximal accepted holding time (t) (d=day, h=hour)	<p>T: 4 °C t: 14 d <i>11% VOC loss without vinyl chloride. 37% vinyl chloride loss. (after 24 h) (article 5)</i></p>	<p>T: 4 °C t: 48h Preserve sample with methanol upon laboratory receipt T: 4 °C Total t: 14 d</p>	<p>T: 4 °C t: 48 h <i>13% BTEX/MTBE loss* (after 6 h) -12% BTEX/MTBE loss* (after 24 h) 6% BTEX/MTBE loss* (after 48 h) (article 2)</i></p>	<p>T: -7 °C t: 48 h</p>	<p>T: 4 °C t: 48 h Frozen to T=<-7 °C upon laboratory t: 14 d</p>	<p>T: <-7 °C >-20 °C t:14 d</p>	<p>T: 4 °C t: 48 h Frozen to T=<-7 °C upon laboratory t: 14 d</p>	<p>T: <-7 °C >-20 °C t: 14 d</p>	<p>T: 4 °C t: 14 d</p>
	4. Measurement in laboratory	Gas chromatographic procedure								
	5. Data processing	Data collection, evaluation and results writing in a report								

*Remarks *italic text*: the VOC losses depend not only on the method (storage time, preservation), but also on the different VOC compounds that are used during the tests. The losses are average losses. See for precision and VOC compounds the article.

5. Tests

The claim is verified by a lab test. The test and results are described in the following paragraphs.

5.1 Test documentation table

Test 1	
Name/short description of the test	Determination volatile organic compounds (VOC) losses upon storage, either preceded by extrusion in methanol or water, for several VOC in time.
part of claim to be tested	Losses in VOC will be equal or less when applying the Eijkelkamp sampling protocol as when applying direct extrusion in the field, either in methanol or in water.
locality	Deltares: Preparation Samples,
date	Alcontrol: quantification amount of remaining VOC of the different methods Week 36-38
involved parties and persons	Deltares, Dianne den Hamer Eijkelkamp, Gerard v. Dijk Alcontrol, J. Hutter, Marjanne van den Berg

5.2 Test facility

Preparation of the samples and part of the extrusion activities were performed under the fume hood in the SmartSoils laboratory, Deltares Stieltjesweg 2, Delft. For sample storage on the long-term 10 °C, the cooling cell at the mechanical laboratory was used. Filling, compaction and sand properties analysis were also performed in the mechanical laboratory. The samples were analysed by Alcontrol, Steenhouwerstraat 15 in Hoogvliet.

5.3 Tests implementation

The metal cylinders were filled with (silt) sand to simulate a compacted non-contaminated sand layer. The sand characteristics minimal/maximal density and grain size distribution were determined. The pore volumes of all cores should be similar (imitating a homogeneously compacted sand layer), for comparing the different test methods. Therefore, an equal amount of dry sand was weighed. The bottom of the cylinder was closed waterproof. The sand was compacted in degassed demineralised water while the cylinder was vibrating, for compaction. The water excess on top of the sand was removed.

After compaction, the spiking fluid was added to the sand. The spiking fluid consisted out of 6 volatile aromatic organic components (benzene, toluene, ethyl benzene, o-xylene, m-xylene, p-xylene) and two chlorinated organic components (1,2,4-trichloormethane and 1,1,1-trichloormethane). The spike solution had a fixed composition for all the tests. With a syringe, the solution was injected into the sand core. The core was capped and stored at 10 °C for 20 hours.

Because spiking does not lead to direct homogeneously distributed contamination, analysis after 20 hours at 10°C are the zero measurement. After the standard 20 hours storage, the samples were stored at different temperatures, storage time and storage position, for verification of the claim. The different used methods are shown in table 2 and more precisely described in the test design protocol.

For the extrusion of the specimen from the Eijkelkamp cylinder, the official Eijkelkamp pistol is used. After extrusion, the vial is directly closed. Samples are then analysed by Alcontrol. The methods, which were performed with the Eijkelkamp core sampler, are AP04, cryogen grinding and the dry weight test.

The reference method, which was executed, is the water and methanol method (Method EPA 5035 A) to verify the claim stated by Eijkelkamp in the test design. Two different tests were executed with methanol. First, 110ml methanol was added, but the results showed high VOC loss which does not correspond with the predicted results. Therefore, a second methanol method was tested. Hereby, 400ml methanol was added to the jar. With a higher methanol volume more VOC can be dissolved into it, causing less evaporation.

The chloride test was performed to determine the error that is caused by the spiking procedure itself. Chloride is not a volatile compound, meaning that chloride loss is not induced by evaporation but during the spiking step. When VOC loss is measured for the different methods, it can be retrieved what the accuracy is for each method with respect to volatile evaporation.

5.4 Test results

In table 2, the results of the different test methods and reference methods are presented. For each method, three samples were spiked and analysed, and the average values are shown in the table. Appendix 2 shows the results for all analysed samples. For each method, different storage time and storage temperatures have been tested for a proper verification of the claim. All the samples were first stored for 20 hours at 10 °C. Except for “AP04, immediately after spiking” method, here the measurements were done directly after spiking. For this method, twice samples were prepared for analysis, in September and in October. This was done because the variation between the samples (from September) was very high.

The results show that there is a small difference between the measurements “AP04, immediately after spiking” and the “AP04, with 20h at 10°C storage”. This indicates that hardly any loss of VOC occurred during the standard storage step.

A high VOC loss has occurred for all the methods. In literature it was stated that the loss-percentage would be even or lower than 11% for the standard methods. This is the loss after 20 hours storage. Table 2 shows the loss percentages for the different methods compared with the original spiking composition. For all methods, a loss percentage higher than 20% was measured. However, the reference test (chloride spiking test) shows a loss of 40%. Because chloride does not evaporate, the loss of chloride is caused during the spiking step. The spiking step for chloride has been performed the same way as the spiking step for the other methods, indicating that the main loss of VOC's has occurred during spiking and is not caused by evaporation. Table 3 shows the percentage of VOC losses after chloride correction. It can be determined that the loss for the soil corer (Eijkelkamp) and for 400ml methanol is less than 11%. The loss during spiking is probably caused by liquid loss when the cores are capped and by dilution.

Complete loss, especially with the compounds with a vapour pressure higher than 29 mbar, is measured with the cryogen grinding method. This is due to the preparation method of cryogen grinding. Sodium sulphate and talc are added before grinding the samples. These compounds produce heat, causing evaporation of high vapour pressure volatiles.

Table 1: Results: samples with different preparation and analyse methods

Analysed: 2 until 4 Sept 2008	Soil core			Chloride	1,1,1-trichloro-ethane	benzene	toluene	ethyl-benzene	m/p-xylene	o-xylene	1,2,4-trichloro-benzene
	Vapour pressure	dry weight sand [g]	wet weight sand after spiking [g]								
	mbar			486.45	133	110	29	9.3	8.1	7	2
Spiked amount				486.45	15.06	15.06	15.05	15.07	30.09	15.04	15.06
<i>Eijkelkamp</i> ¹ AP04 immediately after spiking	408.05	479.3	85		7.6	7.9	9.0	8.7	24.4	9.6	12.2
<i>Eijkelkamp</i> ¹ AP04 20h diffusion at 10 °C	408.09	481.4	85		6.9	8.3	9.2	9.5	18.1	9.8	12.5
<i>Eijkelkamp</i> ¹ AP04 48h storage at 4 °C	408.06	481.1	85		8.2	9.5	10.4	10.5	19.7	10.5	12.5
<i>Eijkelkamp</i> ¹ AP04 48h storage at -20 °C	408.03	483.5	84		8.6	10.7	11.0	10.9	20.6	10.9	12.2
<i>Eijkelkamp</i> ¹ Cryogen grinding 20h diffusion at 10 °C	408.09	476.7	86		<0.12	<0.12	0.6	3.8	10.2	6.1	16.8
<i>Eijkelkamp</i> ¹ Cryogen grinding 48h storage at 4 °C	408.02	477.2	86		<0.12	<0.05	0.6	3.5	8.9	5.3	14.5
EPA 5035A ³ 110 ml Methanol 20h diffusion at 10 °C	408.08	-			4.7	5.2	4.1	3.7	6.1	3.9	5.7
EPA 5035A 110 ml Methanol 48h storage at 4 °C	408.06	-			4.7	5.5	4.4	4.1	6.7	4.3	6.3
EPA 5035A 400 ml Methanol 20h diffusion at 10 °C	408.04	-			9.6	11.6	11.4	11.2	21.7	11.0	13.0
EPA 5035A 400 ml Methanol 48h storage at 4 °C	408.05	-			8.1	10.5	10.5	10.3	20.0	10.1	11.0
EPA 5035 Water 20h diffusion at 10 °C	408.02	-			6.0	6.3	7.5	7.8	14.3	8.0	10.9
EPA 5035A Water 48h storage at 4 °C	408.05	-			5.6	5.3	6.8	6.8	12.6	6.9	9.1
dry material (spiked with VOCl)	408.10	480.2	85								
chloride spike check (reference)	408.07	475.67	86	290.8							

¹ Samples prepares in Eijkelkamp cylinder from the 04.16 soil coring kit for chemical soil research.

² Weight percentage of the sand after drying for 24 hours at 105 °C.

³ Standard method where samples are prepared in jars filled with methanol or water.

Table 2: VOC loss for the different volatiles in percentages and with standard deviation.

Volatile organic compound	1,1,1-trichloro-ethane		benzene		toluene		ethyl-benzene		m/p-xylene		o-xylene		1,2,4-trichloro-benzene	
	% losses	%std	% losses	%std	% losses	%std	% losses	%std	% losses	%std	% losses	%std	% losses	%std
Reference spike (chloride): 40% loss														
method														
Eijkelkamp AP04 immediately after spiking duplicate	47	10	47	11	38	9	41	6	36	7	33	7	9	13
<i>Eijkelkamp</i> AP04 20h diffusion at 10 °C	54	12	45	15	39	14	37	12	40	10	30	11	17	6
<i>Eijkelkamp</i> AP04 48h storage at 4 °C	44	3	37	4	31	3	31	3	35	2	30	3	17	2
<i>Eijkelkamp</i> AP04 48h storage at -20 °C	43	2	31	3	27	2	28	2	32	2	28	2	19	4
EPA 5035A 110 ml Methanol 20h diffusion at 10 °C	69	2	66	4	73	5	75	5	80	4	74	6	62	10
EPA 5035A 110 ml Methanol 48h storage at 4 °C	64	3	60	3	61	2	62	2	67	1	60	2	43	3
EPA 5035A 400 ml Methanol 20h diffusion at 10 °C	36	4	23	3	24	3	25	4	28	4	27	3	14	7
EPA 5035A 400 ml Methanol 48h storage at 4 °C	46	5	30	6	30	5	31	5	33	5	33	5	26	9
EPA 5035A water 20h diffusion at 10 °C	60	14	58	19	50	23	48	22	52	20	47	23	28	28
EPA 5035A water 48h storage at 4 °C	63	8	65	15	55	17	55	15	58	15	54	16	39	15

Table 3: Relative VOC loss, after correction with chloride spiking test

Volatile organic compound	1,1,1-trichloro-ethane	benzene	toluene	ethyl-benzene	m/p-xylene	o-xylene	1,2,4-trichloro-benzene
	% losses	% losses	% losses	% losses	% losses	% losses	% losses
Reference spike (chloride): 40% loss							
method							
Eijkelkamp AP04 immediately after spiking duplicate	7	7	-2	1	-4	-7	-31
<i>Eijkelkamp</i> AP04 20h diffusion at 10 °C	14	5	-1	-3	0	-10	-23
<i>Eijkelkamp</i> AP04 48h storage at 4 °C	4	-3	-9	-9	-5	-10	-23
<i>Eijkelkamp</i> AP04 48h storage at -20 °C	3	-9	-13	-12	-8	-12	-21
EPA 5035A 110 ml Methanol 20h diffusion at 10 °C	29	26	33	35	40	34	22
EPA 5035A 110 ml Methanol 48h storage at 4 °C	24	20	21	22	27	20	3
EPA 5035A 400 ml Methanol 20h diffusion at 10 °C	-4	-17	-16	-15	-12	-13	-26
EPA 5035A 400 ml Methanol 48h storage at 4 °C	6	-10	-10	-9	-7	-7	-14
EPA 5035A water 20h diffusion at 10 °C	20	18	10	8	12	7	-12
EPA 5035A water 48h storage at 4 °C	23	25	15	15	18	14	-1

Temperature seems to influence the VOC loss for the Eijkelkamp method. VOC loss is higher when the cylinders were stored at higher temperatures (Fig. 3). Only for 1,2,4-trichlorobenzene the storage temperature had not much influence on the loss, which is probably because of its low vapour pressure. For the standard methods, the storage temperature had an opposite effect for the VOC loss; here VOC loss was less at higher storage temperatures (Fig. 4).

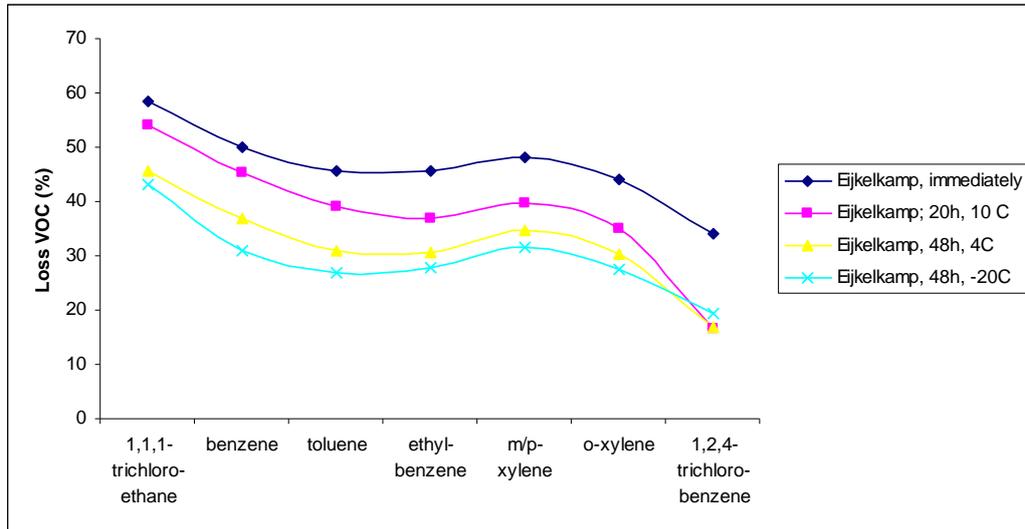


Figure 3. VOC loss for different Eijkelkamp methods.

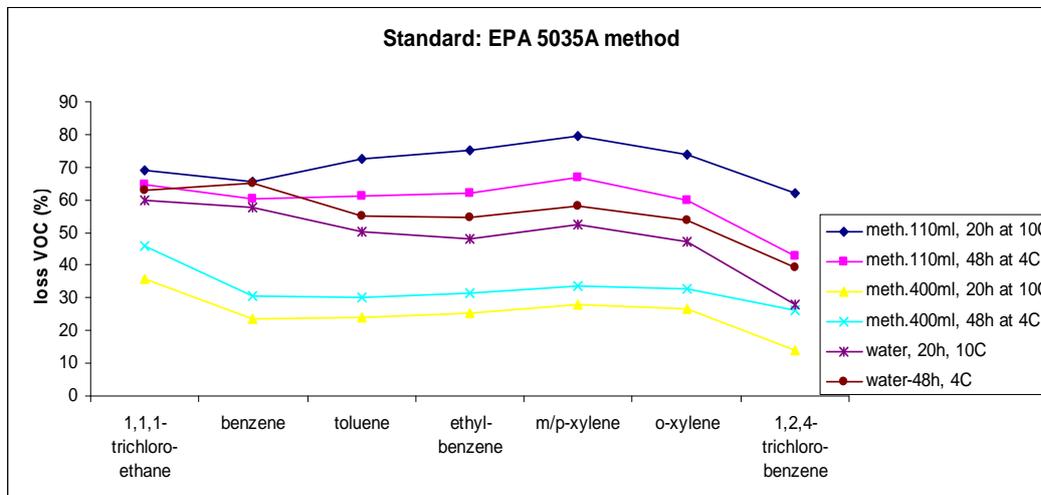


Figure 4. VOC loss for the three standard methods (with water or methanol)

Figure 4 shows that the 110ml methanol method (for both storage times and temperature) shows more VOC losses than the water method and the 400ml methanol method. When the 110ml methanol results are compared with the Eijkelkamp results, it can be concluded that the 110ml methanol method causes the highest VOC loss of all performed methods. This is probably because there is not enough methanol in the cylinder to solve all the compounds, causing a higher compound loss. When 400ml methanol is added to the cylinder the VOC loss decreases significantly.

In general, it can be concluded that the vapour pressure has a slight influence on the amount of loss. Compounds with a high vapour pressure show a higher concentration loss. However, the compounds benzene, toluene, ethylbenzene, m/p-xylene and o-xylene show for most methods the same concentration loss.

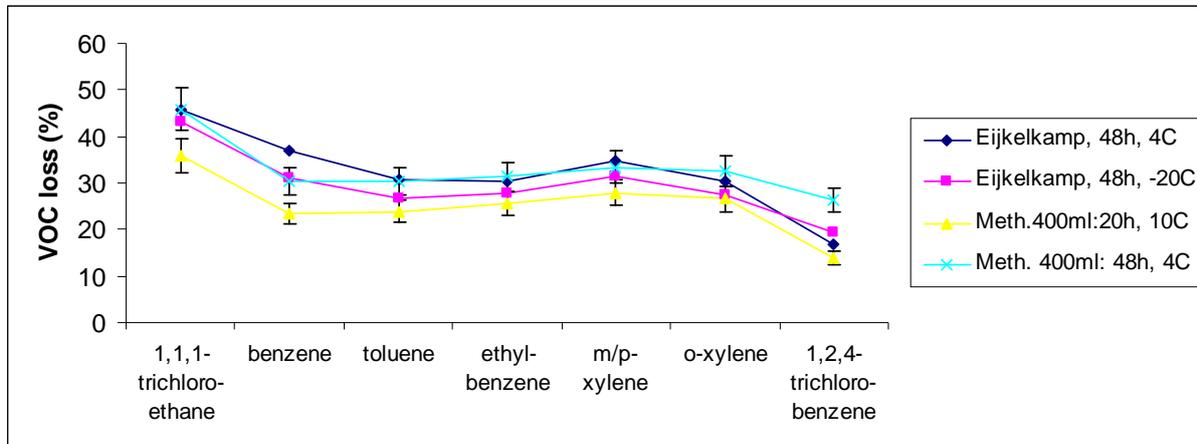


Figure 5. VOC loss (%) for Eijkelkamp method (48h, 4°C; 48h, -20°C) compared with 400ml methanol method (20h, 10°C; 48h, 4°C).

Figure 5 shows that the Eijkelkamp method stored at 4°C and at -20°C is comparable with the reference method 400ml methanol, EPA 5035A. These methods also show the least variation in measurements, indicating that they have a small error margin in their procedure.

The Eijkelkamp methods differ less than 10% in VOC loss, compared with the 400ml methanol methods. VOC losses induced by the soil corer are less or equal compared with the standard methods.

The loss of dry sand when it is taken out of the soil core, is negligible for the Eijkelkamp method, namely 0.86%.

6. Evaluation

The claim was successfully verified.

The losses obtained by the 04.16 soil coring method of Eijkelkamp are comparable to the 400ml methanol (EPA 5035A) standard analyse method. The difference in losses are not more than 10%, meaning that the Eijkelkamp soil corer can be used as a storage and sampling device for samples without much loss of VOC's. The losses induced by the soil coring kit and protocol are less than when water is used as a solvent.

In addition, the margin of error is small for the 04.16 soil coring kit, similar to the 400ml methanol method.

When samples are frozen the least losses in VOC's occur with the Eijkelkamp method.

Technical specification

For the technical components, their functions, setups, material of components, please refer to:

- Operating Instructions 04.16 soil coring kit for chemical soil research, Eijkelkamp, chapter 1 introduction and chapter 2 Technical specifications

Weight of set: 33.5 kg

Energy consumption: manpower

Consumables: detergent and water during cleaning of parts of the soil coring kit for chemical soil research, for soil with volatile components.

Performance of the device

Type of measurements and sampling: This technique is only specified for undisturbed soil sampling with no hassle in the field with methanol. The measurement of concentrations of volatiles takes place in the laboratory is done by different analyses techniques.

Prevention of possible interferences: please refer to:

- Operating Instructions 04.16 soil coring kit for chemical soil research, Eijkelkamp, chapter 3 Safety, chapter 5 use of the equipment, chapter 7 troubleshooting and chapter maintenance

Operation conditions

Type of sample: Soil sample with or without volatiles. The sampling must be done without disturbance of soil aggregates to prevent mixing with air. Sample volume 226 ml. sample preferably is taken at least 1 meter below soil level to prevent soil ventilation by wind, or daily temperature variations. In addition, the strong influence of bacteria in the organic material rich top soil is then eliminated.

Prevention of possible interferences causes of matrix and Sampling and storage: please refer to:

- Gerard P. van Dijk, ISO/TC 190/SC2 N 267, Letter for ISO/TC 190-SC2+3, 1 August 2007.

Preparation for use: please refer to:

- Operating Instructions 04.16 soil coring kit for chemical soil research, Eijkelkamp, chapter 4 preparation for use

Processing and adjustment steps: please refer to:

- Operating Instructions 04.16 soil coring kit for chemical soil research, Eijkelkamp, chapter 5 Use of the equipment.

Conditions equipment: please refer to:

- Operating Instructions 04.16 soil coring kit for chemical soil research, Eijkelkamp, chapter 8 Maintenance

Sampling depth: It is possible to take samples to a depth of 5 meters.

Specific instructions: Please refer to:

- Operating Instructions 04.16 soil coring kit for chemical soil research, Eijkelkamp.

Costs

Purchase costs: € 1566.00

Running costs: cost of working hours of field workers + two plastic PE caps per sample art. Nr 10.01.52 25 pieces €5.10 so €0.41 per sample.

Maintenance costs: Depending on soil type. In stony soil, the coring tubes may become immediately damaged. Otherwise the coring tubes can be used around 50 times.

Additional information

User manual/instruction sheet and operator manual: please refer to:

- Operating Instructions 04.16 soil coring kit for chemical soil research, Eijkelkamp

Maintenance manual: please refer to:

- Operating Instructions 04.16 soil coring kit for chemical soil research, Eijkelkamp, chapter 8 Maintenance

Required personal qualification: moderately trained field workers

Guaranty on the purchased device: 1 year

Network of service: g.vandijk@eijkelkamp.com

Troubleshooting and concept to optimize the implementation of the technology: troubleshooting is described in:

- Operating Instructions 04.16 soil coring kit for chemical soil research, Eijkelkamp, chapter 7 Troubleshooting

Scope, field of application

Contaminants

Ascertainable parameters and mix of contaminants: volatile components are ascertainable in the laboratory from the taken samples with the soil core

Equipment resistance against contaminants: Parts contacting the sample are made from stainless steel 304 type.

Environmental conditions

Water: It is no problem if water is trapped in the sample.

Chemical conditions of water: Every chemical condition of water is possible except in conditions where stainless steel may be attacked on short term (very high or very low pH).

Solids: The equipment can be hammered in hard soils. The very local influence of bacteria and fungi in the organic material rich top soil is excluded by taking the sample below this biologically very active layer. Generally, a depth of 0.5-1 meter will suffice.

Soil air: Soil air (and solids and water) will be trapped in the sample in concentrations that are representative for the soil in-situ. To prevent a strong influence of ambient air ventilating the top soil layer and the layer directly beneath, the sample is taken below these layers. Generally, a depth of 1 meter will suffice to avoid this influence.

Geological and hydro-geological situation: Equipment can be hammered in hard and various soils. Very stony soil layers cannot be sampled with this equipment.

Locality of application

Sample location: in-situ

Level depth: It is possible to take samples to a depth of 5 meters. With a cable attached to a hammering device larger depths are possible

