Process Document for Joint Verification of the Sorbisense Ground Water Sampler

and

Sorbisense GSW40 Passive Sampler Joint Verification Protocol for Volatile Organic Compounds in Groundwater

and

Sorbisense GWS40 Passive Sampler Joint Test Plan for Volatile Organic Compounds in Groundwater

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FOREWARD

The U.S. Environmental Protection Agency (EPA), Advanced Monitoring Systems (AMS) Center of the Environmental Technology Verification Program (ETV) collaborated with the NOWATECH DHI Water Monitoring Center (DHI WMC), a pilot ETV program in the European Union, to conduct an international joint verification test of a passive ground water sampler.

This document, which is actually a compilation of three separate documents, was used by NOWATECH and the AMS Center to jointly verify the performance of this technology. It is composed of a process document, a verification protocol, and a test plan for joint verification. Combined, these three documents satisfy the requirements of an EPA ETV approved verification test/QA plan and ensure that the requirements of both programs are met.

The verification protocol and test plan were developed by NOWATECH with input from Battelle, EPA, and other stakeholders. Together these documents satisfy NOWATECH's programmatic requirements. The process document was developed by the AMS Center with input from NOWATECH and the EPA. It was developed as a supplement to the NOWATECH documents, to ensure that all of EPA ETV programmatic requirements are met. All three documents were reviewed and approved via the ETV process prior to the start of testing.

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EXECUTIVE SUMMARY

The Nordic Water Technology Verification Center's (NOWATECH) DHI Water Monitoring Center (DHI WMC), a pilot Environmental Technology Verification Program (ETV) in the European Union, and the United States Environmental Protection Agency ETV Program's (EPA ETV) Advanced Monitoring Systems (AMS) Center are jointly verifying the Sorbisense (vendor) GWS40 passive ground water sampler.

Under this joint effort, NOWATECH was responsible for developing the verification protocol, preparing the test plan including quality assurance (test/QA), testing, and generating the verification report/verification statement in accordance with their requirements. The AMS Center provided technical and quality assurance oversight throughout the NOWATECH process to ensure EPA ETV requirements were also met, and facilitated reviews and audits by QA personnel, EPA Program management, and stakeholders of the verification protocol, test plan, testing, and verification report. The AMS center also developed a process document to supplement the protocol and test plan developed by NOWATECH and ensure that all of EPA ETV programmatic requirements are met. The process document, combined with the protocol and test plan, satisfy the requirements of an EPA approved verification test/QA plan. This document is a compilation of these three documents.

The purpose of this verification is to evaluate a passive ground water sampling technology, which is capable of detecting 10 contaminants: mono-, di-, tri- and – tetrachloroethenes, chloroethene, benzene, toluene, ethylbenzene, xylenes (BTEX), and methyl-*tert*-butylether (MTBE). Passive sampling is based upon distribution of solutes between the sampled medium, and a collecting medium. Flow of solute from one medium to the other continues until equilibrium is established in the system. The amount of solute in the sampling medium is then determined analytically and can be used to calculate the concentration in the sampled medium. This result will then be compared to results from a standard analytical method for the contaminants of interest, providing information on the precision, accuracy, and range of the technology being verified. Other verification parameters will include determination of the limit of detection, and the robustness of the monitoring technology.







Environmental Technology Verification Program Advanced Monitoring Systems Center

Process Document for US EPA ETV AMS CENTER and NOWATECH DHI WMC Joint Verification of the Sorbisense Ground Water Sampler



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1 INTRODUCTION

Nordic Water Technology Verification Center's (NOWATECH) DHI Water Monitoring Center (DHI WMC), a pilot Environmental Technology Verification (ETV) program in the European Union, and the United States Environmental Protection Agency ETV (US EPA ETV) program's Advanced Monitoring Systems (AMS) Center will jointly verify the Sorbisense (Vendor) passive ground water sampler named the GWS40. A NOWATECH DHI WMC-US EPA ETV AMS Center joint verification has not yet been performed so the need exists to establish a process to ensure that the requirements of both programs will be met for the joint verification to be successful.

The purpose of both ETV programs is to provide objective and quality-assured performance data on environmental technologies, so that users, developers, regulators, and consultants can make informed decisions about purchasing and applying these technologies. Figure 1 describes the organizational relationships for this joint verification. NOWATECH has DHI as their NOWATECH Water Monitoring Center (DHI WMC). Battelle manages the ETV Advanced Monitoring Systems (AMS) Center under a cooperative agreement with the US EPA. NOWATECH will perform the verification protocol preparation, the test plan preparation, including quality assurance (QA), testing, and verification report/verification statement preparation in accordance with the NOWATECH requirements. To ensure US EPA ETV program acceptance of the verification, the AMS Center will provide technical and quality assurance oversight throughout the NOWATECH process to confirm that each step meets the US EPA ETV program requirements. The AMS Center will facilitate the necessary reviews and audits by US EPA program management, stakeholders, and QA personnel of the verification protocol, the test plan, the testing activities, and the verification report. This will provide the necessary oversight for the US ETV program to ensure a quality process of evaluating, data collection, and reporting. Because DHI is performing the technical work and preparing the drafts of the documents, it is assumed that DHI will follow the necessary process to ensure a NOWATECH verification from their actions. Therefore, this document is focused on what Battelle and DHI must do to ensure that the verification fulfills the requirements of the US EPA ETV program. The efforts of DHI in testing and verification are described in a verification protocol and a test plan (which are the equivalent of an ETV AMS Center Test/QA plan) prepared in compliance with the NOWATECH Center Quality Manual Template¹. The roles of each participant shown in Figure 1 are described in the following

section. The names of the key personnel and their roles during this verification are presented in Appendix A.



Figure 1. Organization Chart for the Joint Verification Test of Sorbisense GWS40 Passive Ground Water Sampler

1.1 Sorbisense Involvement

Sorbisense, the technology vendor, has entered into agreements with both Battelle and DHI for this joint ETV verification. The vendor will provide the following support during joint verification of the vendor's technology:

- A person from the vendor's organization to be Battelle's and DHI's point of contact and to lead vendor's participation in joint verification of the vendor's technology;
- Review and comment on the joint verification testing documents, including Verification Protocol, Test Plan, Verification Report, Verification Statement, and other documents pertaining to joint verification of the vendor's technology as requested by Battelle and/or DHI;
- Permission to post/cite information about the vendor's technology, including the Verification Protocol, Test Plan, Verification Report, and Joint Verification Statement, on the US ETV website (http://www.epa.gov/etv/) and in other program publications;
- Equipment/materials for testing, appropriate training in its operation, and onsite support on an as needed basis;
- At no cost to DHI or Battelle or US EPA, the vendor's technology and associated equipment/materials for testing, appropriate training in its operation, and on-site support on an as needed basis; and
- Written descriptions, diagrams, and/or photographs of the vendor's technology, as input for the Verification Protocol, Test Plan and Verification Report.

1.2 DHI Involvement

During the verification of vendor's technology, DHI will:

- Provide a person from DHI's organization to be Battelle's and the vendor's point of contact and to lead DHI's participation in verification of the vendor's technology;
- Prepare and revise the Verification Protocol, Test Plan, Verification Report, Verification Statement, and other documents pertaining to the verification of

the vendor's technology and allow Battelle, US EPA, and the Expert Group the opportunity to review and comment on these documents;

- Assemble a team of qualified technical staff to conduct the verification test in accordance with the Verification Protocol, Test Plan, and this document;
- Ensure that all quality procedures specified in the Verification Protocol, Test Plan, NOWATECH Center Quality Manual Template¹, and this document are followed;
- Conduct a (virtual) joint kick-off meeting with Battelle prior to test initiation;
- Provide a summary of the QA activities performed by the DHI WMC Internal Auditor during the verification testing; and
- Provide permission to post/cite information about DHI's involvement in the joint verification, including the Joint Verification Protocol, Test Plan, Verification Report, and Verification Statement, on the US ETV website on the US ETV website and in other program publications.

1.3 Battelle Involvement

During the ETV verification of the vendor's technology, Battelle will:

- Provide a person from Battelle's organization to be DHI's and the vendor's point of contact;
- Prepare a procedural document outlining the process of the vendor's technology verification for acceptance by the US EPA (this document);
- Provide input, review, and comment on the Verification Protocol, Test Plan, Verification Report, Verification Statement, and other documents pertaining to verification of the vendor's technology;
- Conduct a (virtual) joint kick-off meeting with DHI (using checklist in Appendix B) prior to test initiation;
- Strive to obtain US EPA approval for the final Verification Protocol, Test Plan, Verification Report, including a Verification Statement; and
- Comply with all quality procedures and program requirements specified in the Test/QA Plan, Quality Management Plan for the ETV AMS Center (ETV AMS Center QMP)², and in the U.S "Environmental Technology Verification Program Quality Management Plan" (US ETV QMP)³, as follows:

- Prepare and get US EPA ETV approval of an audit checklist and provide the checklist to DHI prior to the audit;
- Conduct a technical systems audit once during the verification test;
- Audit at least 10% of the verification data;
- Prepare and distribute an assessment report for each audit;
- o Verify implementation of any necessary corrective action; and
- Provide a summary of the quality assurance/quality control (QA/QC) activities and results for the verification reports.

1.4 NOWATECH Involvement

NOWATECH responsibilities are based on the requirements stated in the NOWATECH Center Quality Manual Template.¹

1.5 US EPA Involvement

A complete list of US EPA's responsibilities in the AMS Center are based on the requirements stated in the AMS Center ETV QMP.² The US EPA will provide technical and quality oversight of all ETV AMS Center activities to ensure compliance with the US ETV program requirements.

1.6 Stakeholder Committee and Expert Group Involvement

The AMS Center's Water Stakeholder Committee is made up of buyers and users of such technologies. This committee assists in prioritizing the types of technologies to be verified and in specific cases, provides testing support. It also has representatives that assist in review of the Test/QA plans, Verification Reports, and Verification Statements. The AMS Center Water Stakeholder Committee provided concurrence for the Center to proceed with testing in this area. The stakeholders have been kept apprised of progress throughout the planning process for this test and have provided input during progress meetings on the test design.

In addition, the US ETV AMS Center obtains the peer review of two external peer reviewers and one EPA peer reviewer who are not directly involved with the verification test. The NOWATECH ETV program uses an Expert Group to perform the external peer review of the documents and give input on the verification. For this test, the Expert Group is made up of three individuals to fulfill the requirements of both programs. These individuals are named with their affiliations in the Verification $Protocol^6$ and Test $Plan^7$.

2 QUALITY SYSTEMS

The Battelle and DHI quality systems to be implemented for this joint verification will conform with the specifications listed in:

 ANSI/ASQ E4-2004, "Specifications and Guidelines for Quality Systems for Environmental Data Collection and Environmental Technology Programs"⁴ or the comparable International Standards Organization (ISO) 9001⁵.

Per the US EPA ETV QMP^{3,} verification organization quality systems, such as DHI's quality systems, are to be reviewed and approved by verification organization management, the AMS Center Manager, and the AMS Center Quality Manager. Since not all of the NOWATECH/DHI quality documents have been finalized, this process document will serve to define the specific quality activities that will be performed by Battelle and DHI for this joint verification.

3 VERIFICATION PLANNING

In performing the verification test, DHI and Battelle will follow the technical and QA procedures specified in the NOWATECH Verification Protocol⁶, NOWATECH Test Plan⁷, and this process document. Because DHI is preparing the Verification Protocol, Test Plan, conducting the testing activities, and preparing the Verification Report and Verification Statement, the procedure and expectations of the US EPA ETV program need to be clarified in a document that explains the process and requirements (this document).

3.1 Planning the Test Design

Initially, the verification test design process produced a Verification Protocol⁶ and Test Plan⁷ based upon the NOWATECH and US EPA ETV processes. These two documents together represent the equivalent of a US ETV AMS Center Test/QA plan.

The protocol includes an Application and Performance Parameter Definition Document (Appendix 3 in the Verification Protocol⁶) that developed relevant parameters and ranges hereof for verification considering the vendor stated performance, government standards, and other technologies and methods in the market. It also evaluated existing data that has been collected to decide whether it could be used as part of the verification or whether it could be used as the vendor stated claims to help decide on relevant performance parameters to test during the verification. The Application and Performance Parameter Definition Appendix was not jointly produced and is a specific process within the NOWATECH program; therefore, it was not reviewed by the US EPA ETV program. In designing this verification test, DHI staff used consensus-accepted test design and a previously peer reviewed US EPA ETV Test/QA Plan⁸. The design also takes into account constraints of time, scheduling, and resources. All relevant activities pertaining to environmental data operations have been identified, as well as performance specifications and the appropriate controls. Finally, a process document (this document) was produced by Battelle to address the process and differences between the programs to ensure a successful joint verification. Collectively, these three documents (the NOWATECH Verification Protocol⁶, NOWATECH Test Plan⁷, and Process Document for the US EPA ETV AMS Center and NOWATECH DHI WMC Joint Verification of the Sorbisense Ground Water Sampler) are referred to as the "testing documents".

The US EPA ETV process utilized its Water Stakeholder Committee to guide the test design process. It provided concurrence for the Center to proceed with testing in this area. The stakeholders have been kept apprise of progress throughout the planning process for this test and have provided input during progress meetings on the test design. The committee also identified potential peer reviewers to perform a formal technical review of the testing documents. The NOWATECH ETV program uses an Expert Group to perform the external peer review of the documents and give input on the verification. For this test, the Expert Group is made up of individuals to fulfill the requirements of both programs. It includes three individuals that have extensive experience in the field of ground water monitoring, one EPA reviewer and two non-EPA reviewers. These reviewers have no direct involvement in the verification test beyond providing their reviews. The comments from the reviews performed by the Expert Group of the testing documents and the Verification Report and Verification Statement will be reconciled by DHI. The review process will utilize the Review Report Form produced by the NOWATECH and is included in Appendix C.

4 VERIFICATION TEST IMPLEMENTATION

This technology performance verification will be implemented according to the Verification Protocol⁶ and the Test Plan⁷ (including technical procedural documents) prepared during planning. Generation of verification test data will not be initiated until the approved Verification Protocol and Test Plan are in place. Any data generated before the required documents are approved will have to be repeated. In performing the verification test, DHI will perform an internal audit of the data collection and handling that follows the technical and QA procedures specified in these documents, as well as, the NOWATECH Center Quality Manual Template¹. The Battelle AMS Center will perform a technical systems audit (TSA) to be sure that these requirements are being met.

A virtual joint kick-off meeting will be held prior to the start of the verification test to review procedures for the test with all verification testing staff. The joint kick-off meeting checklist is provided in Appendix B.

Test personnel will have access to the approved testing documents, approved changes to testing documents, and all referenced documents. When a prescribed sequence for the work is defined in the testing documents, work performed shall follow that sequence. Changes to that sequence need to be documented by either amendment (planned changes) or deviation (unplanned changes). All verification test activities will be documented. Suitable documents are bound notebooks (e.g. laboratory record books, or LRBs), field and laboratory data sheets, spreadsheets, computer records, and output from instruments (both electronic and hardcopy). All documentation is implemented as described in the testing documents. All implementation activities are traceable to the testing documents and to the test personnel. The responsibilities of specific test personnel listed in these testing documents that leave the project before it is completed will be reassigned.

When work cannot be implemented according to the approved testing documents, DHI shall be responsible for providing a written amendment or a deviation report for the test records. Amendments are produced for changes that are made to the testing document before

the proposed change will be made. Amendments must be approved by the DHI WMC Verification Responsible, the DHI WMC Internal Auditor, Battelle AMS Center Manager, and the Battelle AMS Center Quality Manager. Following approval, the amendment will be distributed to all internal personnel holding a copy of the testing documents. A deviation report is produced for any changes to the testing document that occurred during the test. Deviation reports must be retained in the verification test records and summarized in the Verification Report. Frequent deviations from established procedures should result in a retrospective review of the written document and possible revision. Amendments and deviations will include all the information displayed on the forms shown in Appendix D.

All persons responsible for performing verification testing and Sorbisense will receive copies of the final versions of the Verification Protocol⁶ and the Test Plan⁷ and associated documentation provided by DHI. Current versions of the Verification Protocol⁶ and the Test Plan⁷ and any applicable methods and SOPs are required to be physically in place at the technology verification testing sites. Battelle oversight and inspection of the verification test will be provided by the Battelle AMS Center Quality Manager and will be over the course of one week. An audit checklist will be prepared and approved by the EPA AMS Center Project Officer and EPA AMS Center Quality Manager. The audit checklist will be provided to DHI prior to the audit. The audit will begin with an "In Briefing" conducted by the Battelle AMS Center Quality Manager to specify and clarify the necessary points of the audit. Testing during laboratory, standpipe, and field activities will be observed along with viewing the external laboratory performing the reference analyses. To verify full implementation of the testing documents, the inspection will include the testing process and any documentation associated with the process, such as sample chain of custody transfers, instrument maintenance and calibration, sample preparation and analysis, and data records. At the conclusion of the audit there will be an "Exit Briefing" held to discuss the findings and corrective actions necessary. The Battelle AMS Center Quality Manager will also provide a written report, verify the completion of any corrective actions needed, and retain a copy of the report with permanent Battelle AMS Center Quality Manager records. The report will be commented on by DHI WMC and comments addressed before it is distributed. The Assessment Reporting Form is presented in Appendix E. The EPA AMS Center Project Officer will be included in the routing of the inspection results and a written copy provided to both the EPA AMS Center Project Officer and EPA AMS Center Quality Manager.

5 ASSESSMENT AND RESPONSE

Assessments will be planned, scheduled, conducted, and reported in order to measure the efficacy of the Battelle and DHI WMC quality procedures and verification execution. The testing will be audited internally by the WMC Internal Auditor in accordance with the Verification Protocol⁶ and Test Plan⁷. The WMC Document Reviewer and Internal Auditor equate to the Battelle AMS Center Quality Manager. The WMC Document reviewer will perform the technical review of the Test Plan and Verification Report documents. The WMC Internal Auditor will perform an audit based upon identified critical points. The procedure includes two main steps:

- Check that the protocol/plan is prepared and followed in accordance with the DHI QMS and the WMC QM (horizontal audit)
- Check of verification/test parameters and data at the identified critical points, ie a vertical audit in lab, office and/or field.

Data from the testing will be controlled by the Verification Responsible and the Test Responsible when received. Data integrity will be controlled by the Test Responsible (transfer of raw data to spreadsheets) and Verification Responsible (calculations as part of evaluations) as spot checks (5% of the data).

Monitoring of the work process to be conducted by the Battelle AMS Center Quality Manager will be done to:

- Ensure satisfactory performance based on requirements,
- Ensure required actions (as specified in implementation documents) are performed so that routine measurements meet specifications,
- Ensure preventive maintenance is performed and documented as specified in facility and study records,
- Ensure calibrations are performed as planned and prescribed,
- Ensure corrective actions are implemented and documented as planned in response to items of nonconformance.

Assessment types, responsibility, and schedule for this joint verification will be as shown in Table 1.0, and are defined as follows:

Quality Systems Audit (QSA), an on-site review of the implementation of the WMC quality procedures. This review is used to verify the existence of, and

evaluate the adequacy of, the internal quality system. This review will be done in conjunction with the Technical Systems Audit.

Technical Systems Audit (TSA), a qualitative on-site evaluation of sampling and/or measurement systems associated with a particular verification test. The objective of the TSA is to assess and document the acceptability of all facilities, maintenance, calibration procedures, reporting requirements, sampling, and analytical activities, and quality control procedures in the test. Conformance with the testing documents and associated methods and/or Standard Operating Procedures is the basis for this assessment. The Battelle AMS Center Quality Manager will prepare and use an audit checklist that is approved by the EPA AMS Center Quality Manager. The checklist will be available to DHI before the audit takes place. This review will be done in conjunction with the QSA.

Performance Evaluation Audits (PE), a quantitative evaluation of the measurement systems used. The type and frequency of performance evaluation self-audits are specified in the Test plan for the joint verification test. The value or composition of reference materials must be certified or verified prior to use, and the certification or verification must be adequately documented. The Battelle AMS Center Quality Manager will review results of PE audits during the TSA; however, it is most preferable for the PE results to be shared with Battelle as soon as they are available, so that any issues can be resolved.

Audits of Data Quality, an examination of the verification data after they have been collected and verified by project personnel. The Battelle AMS Center Quality Manager will audit at least 10% of all verification data, including equations and calculations.

Assessment Tool	Assessors	Responders	Subject of Assessment	Minimum Frequency	Reason for Assessment	Report Reviewed by
Quality Systems Audit	Battelle AMS Center Quality Manager	DHI	NOWATECH Quality Manual Template	once	assess quality management practices of verification collaborators	EPA directors of quality assurance EPA AMS Center Project Officer Battelle AMS Center Manager and Test Coordinator NOWATECH WMC Verification Responsible
Technical Systems Audits	Battelle AMS Center Quality Manager and DHI Internal Auditor	DHI	Verification Protocol, Test Plan, and Process Document	once	assess technical quality of verification tests	EPA AMS Center Project Officer EPA AMS Center Quality Manager Battelle AMS Center Manager and Test Coordinator NOWATECH WMC Verification Responsible
Performance Evaluation Audits	Battelle AMS Center Quality Manager and DHI Internal Auditor	DHI	Verification Protocol, Test Plan, and Process Document	once	assess measurements performance	EPA AMS Center Project Officer EPA AMS Center Quality Manager Battelle AMS Center Manager and Test Coordinator NOWATECH WMC Verification Responsible
Audits of Data Quality	Battelle AMS Center Quality Manager and DHI Internal Auditor	DHI	raw data and summary data	At least 10% of the verification data	assess data calculations and reporting	EPA AMS Center Project Officer EPA AMS Center Quality Manager Battelle AMS Center Manager and Test Coordinator NOWATECH WMC Verification Responsible

Table 1.0 Assessments for the Joint Verification

5.1 Assessment Reports

Each assessment must be fully documented. The Battelle AMS Center Quality Manager and the DHI WMC Verification Responsible will archive all assessment reports generated for this verification test.

Each assessment must be responded to by the appropriate level of management. The Battelle quality assessment reports shall require a written response by the person performing the inspected activity, and acknowledgment of the assessment by the Battelle AMS Center Test Coordinator. The Assessment Reporting Form is provided in Appendix E. An assessment report will also be prepared by the WMC Internal Auditor and provided to the Battelle AMS Center test coordinator for archive.

Corrective action must be documented and approved on the original assessment report, with detailed narrative in response to the assessor's finding. Initials and date are required for each corrective action response. Acknowledgment of the response will be provided by the Battelle Test Coordinator.

Implementation of corrective actions must be verified by the Battelle AMS Center Quality Manager or the DHI Internal Auditor to ensure that corrective actions are adequate and have been completed. This will be done in real-time if corrective actions can be immediately performed and signed off on the assessment report. Alternatively, should the corrective action require additional approvals not immediately available on-site, the DHI Internal Auditor may need to repeat the inspection, as the designee of the Battelle AMS Center Quality Manager, in order to corroborate the implementation and effectiveness of the corrective action.

5.2 Stop Work

Assessor responsibility and authority to stop work during a verification test for quality considerations is delegated to DHI and Battelle. DHI must ensure compliance with all applicable Danish federal, state, and local safety policies during the performance of verification testing.

Should it be determined during an assessment that test objectives of acceptable quality cannot be achieved during performance of verification testing, the Battelle AMS Center is responsible for immediately notifying the DHI WMC Verification Responsible of the need to consider a stop work order. The DHI WMC Verification Responsible will then direct the staff accordingly. The EPA AMS Center Quality Manager will notify the EPA AMS Center Project Officer if work of inadequate quality is discovered.

Documentation is required of any stop work order and the corrective action implemented and shall be maintained as part of the Battelle quality records, with a copy provided to the EPA AMS Center Project Officer and EPA AMS Center Quality Manager.

5.3 Response

Responses to TSA adverse findings should be addressed within 10 working days after the TSA report is completed. However, it is expected that findings that have a direct impact on the conduct of a verification test will be corrected immediately following notification of the finding.

Responses to each adverse finding will be documented in the assessment report. Ideally, assessment reports will provide space after each adverse finding for a response to be recorded. The response will indicate the corrective action taken or planned to address the adverse finding. The response should be signed and dated by the staff responsible for implementing the corrective action.

Any corrective action that cannot be immediately implemented will be verified following completion by the Battelle AMS Center Quality Manager or designee. Once all corrective action associated with an assessment report has been taken, the Battelle AMS Center Quality Manager or designee will initial the corrective action in the assessment report thus documenting verification of the corrective action. Any impact that an adverse finding had on the quality of verification test data should be addressed in the verification report.

The TSA report, with responses to adverse findings recorded within, will be sent to EPA within 10 working days after the Battelle AMS Center Quality Manager has verified all corrective actions.

6 DOCUMENTATION AND REPORTING

6.1 Responsibilities for these activities concerning documentation and reporting are summarized in Table 2.0 and are detailed below.

6.1.1 Preparation

Individual case requirements and this document shall guide document

and record content and/or format. Guidance for content and/or format are derived by the EPA ETV and NOWATECH directives and the following documents:

- ANSI/ASQ E4-2004⁴.
- ETV AMS Center QMP².
- US EPA document "EPA QA/R-2, *EPA Requirements for Quality Management Plans*, March 2001.
- NOWATECH Center Quality Manual Template¹.

6.1.2 Review/Approval.

Record review/approval for joint verification testing documents shall be performed by qualified technical and/or management personnel as described in Table 2.0. The individual reviewer shall have access to all needed references.

-	Prenaration/			
Record Type	Updating	Review	Approval	Finals Distributed to:
Verification Protocol and Test Plan (including SOPs, amendments and deviations)	DHI	Battelle AMS Center Manager Battelle AMS Center Quality Manager EPA AMS Center Quality Manager EPA AMS Center Project Officer NOWATECH WMC Verification Responsible DHI Document Reviewer Stakeholders/Expert Group Vendor	EPA AMS Center Project Officer EPA AMS Center Quality Manager NOWATECH WMC Verification Responsible	Testing Staff Vendor EPA AMS Center Project Officer EPA AMS Center Quality Manager NOWATECH WMC Verification Responsible
Raw data	DHI	WMC Internal Auditor	N/A	EPA can request copies
Verification Report	DHI	Battelle AMS Center Manager Battelle AMS Center Quality Manager EPA AMS Center Quality Manager NOWATECH WMC Verification Responsible Vendor Stakeholders/Expert Group	EPA AMS Center Project Officer EPA AMS Center Quality Manager NOWATECH WMC Verification Responsible	ETV Program Director EPA AMS Center Project Officer ETV Webmaster Vendor NOWATECH WMC Verification Responsible
ETV Verification Statement	DHI	Battelle AMS Center Manager Battelle AMS Center Quality Manager EPA AMS Center Project Officer EPA AMS Center Quality Manager NOWATECH WMC Verification Responsible WMC Document Reviewer Vendor ETV Program Director Stakeholders/Expert Group	EPA Laboratory Director Battelle Management EPA AMS Center Project Officer EPA AMS Center Quality Manager DHI Director RDI and Quality Management NOWATECH Steering Committee Head	ETV Program Director EPA AMS Center Project Officer Battelle AMS Center Manager NOWATECH WMC Verification Responsible ETV Webmaster Vendor
Audit Reports	DHI Internal Auditor	DHI Test Responsible NOWATECH WMC Verification Responsible	N/A	NOWATECH WMC Verification Responsible Battelle AMS Center Quality Manager Battelle AMS Center Manager Battelle Verification Test Coordinator EPA AMS Center Project Officer EPA AMS Center Quality Manager
Audit Reports	Battelle AMS Center Quality Manager	Battelle AMS Center Manager Battelle Verification Test Coordinator NOWATECH WMC Verification Responsible WMC Test Responsible	N/A	EPA AMS Center Project Officer EPA AMS Center Quality Manager Battelle AMS Center Manager Battelle Verification Test Coordinator NOWATECH WMC Verification Responsible

Table 2.0 Document and Reporting Responsibilities for the Joint Verification*

*See Appendix A for the roles and names of the individuals filling these roles.

A6.2 Reporting

The end result of the joint verification process will be a Verification Report and Verification Statement for the Sorbisense GWS40. The review and approval procedures for the verification report and statement for US EPA ETV program purposes are given in Table 2.0. The Verification Report will be peer-reviewed by external reviewers in the Expert Group and the Verification Statement will be signed by an EPA laboratory director, Battelle management, the DHI Director of Research and Quality Management and the NOWATECH Steering Committee Head. Appendix F presents a preliminary template for a Verification Statement. This document will be expanded and organized to meet US EPA ETV and NOWATECH program requirements.

All logos will appear on the Verification Statement. These will include: US ETV, Battelle, NOWATECH, DHI, and US EPA logos. All logos except the US EPA logo will appear on the cover page of all other joint testing documents (Test Plan, Verification Protocol, Verification Report). All of these testing documents will be made publicly available on the US EPA ETV Web site (www.epa.gov/etv) regardless of the technology's performance.

The vendor will comply with both the NOWATECH and US EPA ETV policies on referencing the verification documents of their technology.

7 **REFERENCES**

- 1. NOWATECH. ETV Test Center and Test Organization: Center Quality Manual Template. December 2008.
- 2. Battelle. Quality Management Plan for the ETV Advanced Monitoring Systems Center. Version 7.0. 11-17-2008.
- 3. United States Environmental Protection Agency. Environmental Technology Verification Program Quality Management Plan. Version 3.0. January 2008.
- 4. American Society for Quality. ANSI/ASQ E4-2004. Quality systems for environmental data and technology programs Requirements with guidance for use. 1-4-2004.
- 5. International Standardization Organization. ISO 9001. Quality management systems Requirements. 11-15-2008.
- 6. DHI. Sorbisense GWS40 Passive Sampler, Joint Verification Protocol. January 2009.
- 7. DHI. Sorbisense GWS40 Passive Sampler, Joint Test Plan. January 2009.
- 8. Sandia National Laboratories. Groundwater Sampling Technologies Verification Test Plan. Environmental Technology Verification Program. Version 2.0. July 1999.

APPENDIX A

ROLES OF KEY PERSONNEL

NOWATECH Role:

NOWATECH person who signs the Verification Statement: NOWATECH Steering Committee Head – Christian Grøn

DHI Water Monitoring Center (DHI WMC) Roles:

DHI person who signs the Verification Statement: Director of Research and Quality – Jørn Rasmussen WMC Verification Responsible: Christian Grøn WMC Test Responsible: Gerald Heinicke WMC Document Reviewer: Anders Lynggaard Jensen WMC Internal Auditor: Louise Schlütter WMC Verification and Test staff: several – see protocol and plan

US EPA ETV Roles:

EPA person who signs the Verification Statement: National Risk Management Research Laboratory (NRMRL) Director – Sally Gutierrez EPA AMS Center Project Officer: John McKernan EPA AMS Center Quality Manager: Lauren Drees, EPA NRMRL Director for Quality Assurance

Battelle Advanced Monitoring Systems (AMS) Center Roles:

Battelle person who signs the Verification Statement: Chemical, Environmental and Materials Operations Manager - Lisa McCauley AMS Center Manager: Amy Dindal AMS Center Quality Manager: Zach Willenberg AMS Center Test Coordinator: Anne Gregg

Parallel Roles between the NOWATECH and US EPA ETV programs:

Verification Responsible = Center Manager Test Responsible = Center Test Coordinator Internal Auditor + Document Reviewer = Center Quality Manager

APPENDIX B KICK OFF MEETING CHECKLIST

ETV JOINT VERIFICATION TEST KICK-OFF MEETING

PURPOSE

To prepare verification testing staff for the NOWATECH and US EPA ETV AMS Center joint verification test and review critical logistical, technical, and administrative aspects of the test. The kick-off meeting will be scheduled prior to the start of testing. It should be near the start of the test but allow time for the test coordinator to address any lingering issues.

FORM

The kick off meeting will be virtual, i.e. based upon phone and WebEx sharing of documents.

STAFF TO ATTEND

- Verification test coordinator/responsible (DHI and Battelle)
- ETV program manager (Battelle)
- QA manager (Battelle)
- US EPA ETV program staff and NOWATECH (invited but optional)

<u>All</u> testing staff involved in <u>all</u> phases of test will subsequently have a kick-off meeting onsite with the DHI WMC verification Responsible. The external laboratory is informed through requisitions of analyses only.

PROJECT MANAGEMENT

- Review roles/responsibilities of all staff attending meeting
- Stakeholders, EPA/ETV program manager, and EPA/QA staff pre-notified of testing schedule and start date?
- Review test schedule
- Formal distribution of final, signed hard-copy Test/QA plan made to all staff involved.
- Documentation of all pertinent forms.
 - Peer review forms on Protocol document and Test plan. Must include one EPA reviewer/two non-EPA peer reviewers.
 - Final Test/QA plan approved by vendor.
 - o Vendor-Collaborator agreement signed and stored in project files?
 - Documentation that the vendor is satisfied that the staff operating the technology are proficient in its use.

QUALITY ASSURANCE

- Copies of all standard methods cited or included in the Test plan available to testing staff and in laboratory where test will be performed?
- EPA QA staff pre-notified of test start date?
- Remind testing staff to sign and date everything.
- If samples are to be transported between labs, field sites, and DHI bring chain-ofcustody form to meeting, review how to complete, and where to obtain form.
- Review deviation/amendment procedures at meeting what to do in the middle of a test if testing document cannot be followed who to notify/what forms to file.
- Review testing document at meeting identify key testing procedures and critical steps to ensure no ambiguity or questions.
- Are or will there be copies of the certificates of analysis in the verification test records?
- When will PE audit be performed? Who will perform? Has materials/equipment been purchased or obtained for the PE audit? What are QC limits? What to do if QC limits are not met? Who to contact?
- Will regular communication between DHI and Battelle be maintained? If so, how? Daily/weekly email updates?

TECHNICAL

- Emphasize to testing staff to document anything and everything that is observed about the technologies, particularly if there are unusual sample results (e.g., sample color).
- Are provisions made to handle daily preparation of solutions/standards, if necessary?
- Take digital photos of all test activities.

DATA/REPORTING

- Review data recording forms or sheets at meeting or discuss how/where will data be recorded for each testing activity
- How are data going to be converted electronically? Are data saved in technology undergoing verification and then exported to Excel? Or will data be recorded manually by the operators? If so, how will transcription errors be avoided?
- Data review who will be doing two week review for each data set collected? If Battelle staff not on-site, how will data be transmitted to Battelle?
- Distribute and review report schedule. Reporting should begin at the same time as testing.

APPENDIX C

REVIEW REPORT FORM

Review report		
Document title:	Document date:	November 2008
Reviewer name:	Review date:	
Name:		
Organization:		
Address:		
Telephone:		
E-mail		

Review results							
Rate items	Satisfactory	Unsatisfactory	Overall recommendation				
Contents							
Scope			Acceptable as is				
Organization			Minor revisions				
Data quality			Major revisions				
Method validity			Not acceptable				
Conclusions							
Other (specify)			Reason				

Revision details					
Торіс	Report chapter, section, page	Revision required	Reason	Revision action(to be filled in by document owner during revision after review)	

Add additional rows, if pertinent.

APPENDIX D

JOINT VERIFICATION TESTING DOCUMENT AMENDMENT AND DEVIATIONS FORMS

AMENDMENT

TESTING DOCUMENT TITLE AND DATE:

AMENDMENT NUMBER: _____

EFFECTIVE DATE: _____

PART TO BE CHANGED/REVISED:

CHANGE/REVISION:

REASON FOR CHANGE:

ORIGINATED BY:

Battelle AMS Center Test Coordinator or DHI WMC Test Responsible

DATE

APPROVED BY:

DHI WMC Internal Auditor

DATE

DHI WMC Verification Responsible

DATE

Battelle AMS Center Quality Manager

Battelle AMS Center Manager

DATE

DATE

Required Distribution with documentation - All individuals/organizations listed on distribution for the applicable Test/QA Plan, including but not limited to:

US ETV AMS Center and NOWATECH DHI WMC Joint Verification Process Document Page 30 of 35 Date: 2/10/2009

Battelle AMS Center Manager NOWATECH WMC Verification Responsible Testing Staff Battelle Quality Manager WMC Internal Auditor Subcontractors (if any) EPA/ETV AMS Center Project Officer EPA/ETV AMS Center Quality Manager Vendor
DEVIATION REPORT

TESTING DOCUMENT TITLE AND DATE:

DEVIATION NUMBER: _____

DATE OF DEVIATION: _____

DESCRIPTION OF DEVIATION:

CAUSE OF DEVIATION:

IMPACT OF DEVIATION ON THE TEST:

CORRECTIVE ACTION:

ORIGINATED BY:

Battelle AMS Center Test Coordinator or DHI WMC Test Responsible

DATE

ACKNOWLEDGED BY:

DHI WMC Quality Manager

Battelle AMS Center Quality Manager

DATE

DATE

Required Distribution with documentation - All individuals/organizations listed below: Battelle AMS Center Manager NOWATECH WMC Verification Responsible Battelle AMS Center Quality Manager WMC Internal Auditor

APPENDIX E

ASSESSMENT REPORTING FORM

Quality Assurance Routing Sheet

Verification Test:

Audit Type:

Test Coordinator:

Vendor:

Auditor:

Date:

Test Coordinator, please complete the attached form indicating CORRECTIVE ACTION TAKEN (IF NEEDED), sign and date this Routing Sheet in the space provided beside your name, and return the entire set when completed to the Battelle AMS Center Quality Manager no later than ______.

Route To	Signature	Date
WMC Test Responsible		
AMS Center Test Coordinator		
Approval		
Battelle AMS Center Manager		
Battelle AMS Center Quality Manager		
NOWATECH WMC Verification Responsible		

Audit Comment Sheet

Instructions: The Battelle AMS Center Quality Manager will fill out the first column for the audit indicated above. The Verification Test Coordinator (or assigned responder) will respond to the comments and initial and date the response in column three. The Battelle AMS Center Quality Manager will verify and document that the response/corrective action has been completed by initialing and dating the final column.

QA Comment	Testing Coordinator	Responder	QA Initials/
	Response/Corrective Actions	Initials/ Date	Date

APPENDIX F

EXAMPLE JOINT VERIFICATION STATEMENT

THE ENVIRONMENTAL TECHNOLOGY VERIFICATION PROGRAM

This is a preliminary template of a Verification Statement that may be expanded and organized to meet NOWATECH and US EPA ETV program requirements. NOWATECH and DHI logos will be added.





Battelle The Business of Innovation

TECHNOLOGY TYPE:

APPLICATION:

TECHNOLOGY NAME:

COMPANY:

ADDRESS:

PHONE: FAX:

WEB SITE: E-MAIL:

ETV Joint Verification Statement

- Description of EV and the organizations involved in this joint verification.
- Name technology category and technology (product) that was jointly verified.

VERIFICATION TEST DESCRIPTION

- Describe the verification test- when, how
- Describe the performance parameters
- Describe the QA performed

TECHNOLOGY DESCRIPTION

• Describe the technology (product)

VERIFICATION RESULTS

• Summary of results by performance parameters

Signature blocks for:

NOWATECH Steering Committee Head - Christian Grøn

DHI Director of Research and Quality – Jørn Rasmussen

Battelle Chemical, Environmental and Materials Operations Manager - Lisa McCauley

US EPA National Risk Management Research Office of Research and Development – Sally Gutierrez

NOTICE: ETV verifications are based on an evaluation of technology performance under specific, predetermined criteria and the appropriate quality assurance procedures. EPA and Battelle make no expressed or implied warranties as to the performance of the technology and do not certify that a technology will always operate as verified. The end user is solely responsible for complying with any and all applicable federal, state, and local requirements. Mention of commercial product names does not imply endorsement.









Sorbisense GSW40 Passive Sampler

Joint verification protocol

Volatile organic compounds in groundwater



Version approved

Sorbisense GSW40 Passive Sampler

Joint verification protocol

Agern Allé 5 DK-2970 Hørsholm Denmark

Tel: +45 4516 9200 Fax: +45 4516 9292 dhi@dhigroup.com www.dhigroup.com

Vendor	Vendors representative
Sorbisense ApS	Hubert de Jonge

Project	t	Project N	0		
	Nordic Water Technology Verification Centers	8014	44		
Author	s Christian Grøn	Date	, 2009		
Christian Cign		Approved	lby		
	Joint verification protocol	CHG	ALJ	ALJ	
Revision	Description	Ву	Checked	Approved	Date
Key wo	^{ords} Environmental technology verification, passive sampler, groundwater	Classifica	tion en ernal eprietary		

Distribution		No of copies
Sorbisense DHI UBA-A Battelle US EPA	HdJ CHG, GHE, MTA DM AMG, ZJW LD	File distribution only

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2 INTRODUCTION

Environmental technology verification (ETV) is an independent (third party) assessment of the performance of a technology or a product for a specified application, under defined conditions and quality assurance.

This verification is a joint verification with the US EPA ETV scheme and the Advanced Monitoring Systems Centre, Battelle, see the verification protocol /1/ for details on organization and implications. The compliance of the test with both scheme's requirements is ensured through a process document /2/.

2.1 Name of product

The product is the Sorbisense GWS40 passive sampling system (106-012-11) with samplers (cartridges) for analysis of volatile organic compounds (VOCs) (no. 043-0091-12, 043-0101-12, 043-0102-12). The analysis of the samplers is performed by AlControl under ISO 17025 accreditation. The passive samplers and the subsequent analysis of the cartridges constitute the product.

2.2 Name and contact of vendor

Sorbisense A/S, Niels Pedersens Allé 2, DK-8830 Tjele, Denmark, phone +45 8999 2505, +45 8999 2599.

Contact: Hubert de Jonge, e-mail <u>hubert@sorbisense.com</u>.

The laboratory responsible for the analysis of samples (subcontractor to the vendor) is: ALcontrol Laboratories, Steenhouwerstraat 15, 3194 AG Hoogvliet, Netherlands,

Contact: Jaap Willem Hutter, e-mail <u>j.hutter@alcontrol.nl</u>

2.3 Name of center/verification responsible

NOWATECH Water Monitoring ETV Center, DHI, Agern Allé 5, DK-2970 Hørsholm, Denmark.

Verification responsible: Christian Grøn, e-mail <u>chg@dhigroup.com</u>, phone +45 95 16 95 70, mobile +45 29 65 34 47.

US EPA Advanced Monitoring System Center, Battelle Memorial Institute, 505 King Avenue, Columbus, Ohio 43201-2693, US.

Verification responsible: Anne M. Gregg (AMG), e-mail gregga@battelle.org, phone +1 614-424-7419

2.4 Verification Test Organization

The verification will be conducted as a joint verification between the Nordic Water Technology Verification Centers (NOWATECH ETV) and the U.S.

Environmental Technology verification (US ETV) Program. The verification is planned and conducted to satisfy the requirements of the ETV scheme currently being established by the European Union (EU ETV) and the US ETV program. Verification and tests will be performed by DHI as NOWATECH Water Monitoring Center (DHI WMC) under contract with Nordic Innovation Centre, Nordic Council of Ministers. Battelle will be participating as the manager of the ETV Advanced Monitoring Systems (AMS) Center through a cooperative agreement with the U.S. Environmental Protection Agency (EPA).

The day to day operations of the verification and tests will be coordinated and supervised by DHI personnel, with the participation of the vendor, Sorbisense. The testing will be conducted in the DHI laboratories, Hørsholm, Denmark and in the field in the Copenhagen area, Denmark. DHI will operate the samplers during the verification. Sorbisense will provide the sampling systems, the samplers and the analysis of samplers for the test. Furthermore, Sorbisense will provide user manuals and operation instructions, and will participate in development of protocol and plans with DHI. Battelle will ensure that the verification and tests is planned and conducted to satisfy the requirements of the US ETV program, including obtaining input and concurrence from its stakeholder group, as described in the process document /2/. Battelle will also participate in the development of the plan document for the verification and tests. EPA will participate in quality assurance of the verification and tests.

An expert group is established to provide independent expert review of the planning, conducting and reporting of the verification and tests.

The organization chart in Figure 1 identifies the relationships of the organization associated with this verification and tests.



Figure 1 Organization of the verification and tests

2.5 Expert group

The expert group assigned to this verification and responsible for review of the verification plan and report documents includes:

Dietmar Müller dietmar.mueller@umweltbundesamt.at, (DM), e-mail Contaminated Sites, Umweltbundesamt, Spittelauer Lände 5, 1090 Wien, Austria, phone +43-(0)1-313 04/5913

Mike Sherrier (MS), e-mail michael.p.sherrier@usa.dupont.com, DuPont, Barley Mill Plaza, Bldg 19-1132, 4417 Lancaster Pike, Wilmington, DE 19805, US, phone +1 302-892-1168

Cynthia Paul (CP), e-mail paul.cindy@epa.gov, U.S. Environmental Protection Agency, 919 Kerr Research Drive, P.O. Box 1198. Ada, OK 74820, US, phone: +1 580-436-8556.

2.6 Verification process

Verification and tests will be conducted in two separate steps, as required by the EU ETV. The steps in the verification are shown in Figure 2.



Figure 2 Verification steps

References for the verification process are the Quality Management Plan for the Battelle AMS /3/ and the Quality Manual for the ETV operations at DHI following the NOWATECH Quality Manual Template /4/.

A joint US EPA ETV and NOWATECH ETV verification statement will be issued after completion of the verification. Ensuring the compliance of the verification with the US ETV requirements is done following a process document developed by Battelle AMS.

This verification protocol, the test plan and the process document shall be seen as one consolidated verification description.

3 DESCRIPTION OF THE TECHNOLOGY

The technology product to be verified is applying the technology of passive sampling.

Passive sampling is based upon distribution of solutes between the sampled medium, *e.g.* a water body, and a collecting medium, the sampler or sampling medium. Flow of solute from one medium to the other continues until equilibrium is established in the system, or until the sampling session is terminated by the user. The amount of solute in the sampling medium is then determined analytically and can be used to calculate the concentration in the sampled medium. With exposure until equilibrium, the sampled medium concentration can be calculated based on the solute distribution between the two media involved as obtained by *e.g.* experimental calibration of the device. With exposure until the sampling session is terminated by the user (before achieving equilibrium), the time-weighted average solute concentration in the sampled medium can be determined from the exposure time and the sampling rate for the solute in question.

A wide range of products are available for passive sampling (equilibrium based and rate controlled) of solutes (inorganic and organic) from waters.

4 DESCRIPTION OF THE PRODUCT

The Sorbisense passive sampler combines the principle of passive sampling with a patented tracer based calculation of the amount of water that the sampler has been exposed to. The sampler consists of a polypropylene cartridge containing, see Figure 3:

- A sorbent that absorbs solutes from water passing the sampler.
- Tracer salt that dissolves proportionally with the volume of water passing the cartridge.
- Filters between sorbent and tracer salt compartments.

Figure 3 Principle of the Sorbisense sampler



When the sampling period is over, the Sorbisense sampler is sent to a laboratory for extraction and analyses whereupon time-weighted average solute concentration is reported.

For analysis, the cartridge is cut and the sorbent taken for batch extraction with acetone followed by quantification of sorbed compounds by headspace GC-

MS. The tracer salt (calcium citrate) is taken for extraction with 0.2 M HCl and quantification of extracted calcium with ICP.

The sampled water volume is calculated from:

$$V = \frac{M_{start, tracersalt} - M_{lab, tracersalt}}{K}$$

The solute water concentration is calculated from:

$$C = M_{solute} / V = \frac{M_{solute} * K}{M_{start, tracersalt} - M_{start, tracersalt}}$$

V= water volume in L; $M_{start, tracersalt}$ = weighed amount of salt in production as mg Ca; $M_{lab, tracersalt}$ = extracted amount of salt in laboratory as mg Ca; C = VOC concentration in ug/L; M_{solute} = mass of VOC detected in ug; K = solubility of the salt with the standard calibration value as 184 mg Ca/L.

The product to be verified here is the Sorbisense GWS40 sampling system intended for sampling of shallow groundwater and equipped with samplers for volatile organic compounds.

Figure 4 Mounting of the GWS40 sampling system



The GWS40 is mounted with air hose, safety string and Sorbisense samplers (can be mounted in top and bottom of the GWS40) and is subsequently lowered to the desired measuring depth, see Figure 4. The water pressure will push water through the sampler slowly filling the GWS40. The air hose enables the air inside the GWS40 to escape to the atmosphere. When the measuring period is over, the samplers are removed and sent to the laboratory for analysis.

5 APPLICATION AND PERFORMANCE PARAMETER DEFINITIONS

The application is defined as detailed in the application definition appendix, Appendix 3, in terms of matrix/matrices for use, targets of monitoring and effects.

The passive sampler is supplied by the vendor as combined sampling and analysis, and the verification shall accordingly see these two steps as one.

5.1 Matrix/matrices

The matrix of the application is groundwater and the field of application is investigations of (potentially) contaminated groundwater (groundwater investigations).

5.2 Target(s)

The targets of the product are volatile organic contaminants (VOC), here mono-, di-, tri- and –tetrachloroethenes, benzene, toluene, ethylbenzene and xylenes (BTEX) and methyl-*tert*-butylether (MTBE), see Table 1.

Target compounds	
Chloroethene	Benzene
1,1-Dichloroethene	Toluene
1,2-Dichloroethenes	Ethylbenzene
Trichloroethene	Xylenes
Tetrachloroethene	MTBE

Table 1 Targets of the Sorbisense GSW40 VOC sampler

5.3 Effects

The effects for the application are set in terms of limit of detection (LoD), precision, trueness, range of application and robustness.

5.4 Performance parameters for verification

The ranges of performance relevant for the application, as derived in Appendix 3, are presented in Table 2. These ranges are used for planning the verification and testing only. For Sorbisense VOC sampling, concentrations above 2,000 μ g/L are not likely to be measurable (vendor information) and are not included in the verification. The calculation of the performance parameters explaining their principle is given in Table 5.

Compound	Limit of	Precision	Trueness	Range of	Robustness
	detection			application	
	µg/L	%	%	µg/L	%
Chloroethene	0.02-0.05	<25	75-125	LoD-1*10 ⁶	100±15
1,1-Dichloroethene	0.1-1	<25	75-125	LoD-1*10 ⁶	100±25
1,2-Dichloroethenes	0.1-1	<25	75-125	LoD-1*10 ⁶	100±25
Trichloroethene	0.1-1	<25	75-125	LoD-1*10 ⁶	100±25
Tetrachloroethene	0.1-1	<25	75-125	LoD-0.1*10 ⁶	100±25
Benzene	0.1-1	<25	75-125	LoD-1*10 ⁶	100±25
Toluene	0.5-5	<25	75-125	LoD-0.1*10 ⁶	100±25
Ethylbenzene	0.5-5	<25	75-125	LoD-0.1*10 ⁶	100±25
Xylenes	0.5-5	<25	75-125	LoD-0.1*10 ⁶	100±25
MTBE	0.2-2	<25	75-125	LoD-1*10 ⁶	100±25

Table 2 Ranges of performance parameters relevant for groundwater investigations

Limit of detection shall be evaluated from the standard deviation of replicate measurements at less than 5 times the detection limit evaluated and will reflect a less than 5% risk of false blanks.

Precision shall be evaluated under repeatability and reproducibility conditions. Repeatability is obtained as the standard deviation of measurements done with the same measurement procedure, same operators, same measuring system, same operating conditions and same location, and replicate measurements on the same or similar objects over a short period of time. Reproducibility is obtained as the standard deviation of measurements that includes different locations, operators, measuring systems, and replicate measurements on the same or similar objects. In laboratory terminology, repeatability is the within series precision and the reproducibility the between series precision.

Trueness is the correspondence between (mean) concentrations found in measurements and corresponding true concentrations.

In addition to conventional trueness, the trueness of time-weighted averages obtained with the sampler shall be verified.

The range of application is the range from the LoD to the highest concentration with linear response.

The parameters of robustness to be verified are sampling depth, sampling time, sampling concentration and groundwater ionic strength. Robustness is basically the trueness as found for different values of the robustness parameters.

The version of the product to be verified is designed for sampling shallow aquifers, i.e. with sampling depths from 0.5 to 5 m below groundwater table (mbgw). The pressure on samplers will vary with depth to the sampling positions, and pressure variations in the range of 1.05-1.5 atmosphere shall accordingly be verified.

Sampling time variations from 3 to 9 days shall be verified covering the different sampling times recommended by the vendor, as the exposure time may impact the performance.

In investigations of contaminated groundwater, both uncontaminated and strongly contaminated groundwater will be included. The concentrations verified shall therefore reflect the range from uncontaminated groundwater to highly contaminated groundwater, with at the least 3 concentrations distributed over a relevant range.

In order to reflect the varying ionic strength of groundwaters, groundwater ionic strengths within the range 10-100 mS/m shall be verified, corresponding to the 5-95 percentile of Danish groundwaters /5/.

Information on the analytical performance for the sampler analysis will be obtained from the responsible laboratory for comparison.

Impact of other factors such as groundwater flow, well construction or presence of other contaminants than the targets can not be ruled out and should be considered in planning the tests for the verification.

5.5 Additional parameters

Besides the performance parameters to be obtained by testing, compilation of parameters describing users manual, product costs and occupational health & safety issues of the product are required as part of the verification.

6 EXISTING DATA

A test of Sorbisense samplers, similar but earlier product version, for volatile organic contaminants in groundwater wells has been conducted by the laboratory used by the vendor for sampler analysis.

6.1 Summary of existing data

The summarized data as provided by the manufacturer is presented in Figure 5.

The test was set up with polyvinylchloride (PVC) pipes simulating groundwater wells (standpipes), filled with spiked water and equipped with Sorbisense samplers inserted directly into the water using a pipe adaptor ("pipe"), Sorbisense samplers mounted in GWS samplers ("GWS") and water samples taken directly from the pipe ("water samples").

Test parameter	Sorbisense	Water Samples
VOC Spike level:	0, 6, 36, 120, 3000 ug/L	
(average of results)		
VOC measuring range:	GWS: 0 - 1980 ug/L	0 - 2160 ug/L
	Pipe: 0 – 1860 ug/L	
VOC recovery:	GWS: 85 % of spiked level	75% of spiked level
	_	
(average of results)	Pipe: 94% of spiked level	
VOC detection limit:	0.2 ug / V	0.2 ug/L
	(V = volume sampled)	4
VOC concentration	GWS: 13,7% of mean	30,5% of mean
precision:	Pipe: 8,9% of mean	
	Calculated from 44 duplicates	Calculated from 44
	each	trinicates

Figure 5 Summarized data on sampler test for selected VOC as provided by the manufacturer



6.2 Quality of existing data

It is not stated whether the testing and analysis were done under the laboratory's ISO 17025 accreditation /6/, the test laboratory can not be considered independent, and the documentation made available for the verification is not sufficient to allow for an assessment of the data quality.

6.3 Accepted existing data

It was decided that the existing data shall not be used as part of the verification due to the data quality issues, see Section 6.2. The data will be used as an indication of the performance range to be expected during planning.

7 TEST PLAN REQUIREMENTS

Based upon the application and performance parameter identification, Section 0, the requirements for test design have been set, see below. The detailed test

plan is prepared separately based upon the specification of test requirements presented below.

7.1 Test design

The outline of the required tests is shown in Table 3. The principle behind the design is that three test scales are used: laboratory tests, standpipe tests and field tests. Each scale is further described below and provides information on specified performance parameters, with the smallest scale chosen for each parameter in order to maintain simplicity and controlled conditions in the test.

Laboratory	Stand pipe	Field
Limit of detection: best possible	Limit of detection: realistic	None
Precision and trueness: best possible	Precision (repeatability), true- ness and range of application	Precision (reproducibility)
Robustness, sampling time and groundwater ionic strength	Robustness, sampling depth	General robustness
Trueness of time-weighted average concentration	None	None

Table 3 Test design scales and associated performance parameters

As an example of the application of the scale principle, consider the test for evaluation of trueness and robustness. Trueness as best possible estimate is evaluated from direct application at the laboratory scale (chloroethene only). Trueness as realistic estimate is evaluated from the stand pipe scale simulating a groundwater well (all but chloroethene), and the variation in trueness between groundwater wells (robustness) is evaluated at the field scale. Combining the scales thus provides the best possible estimates of real conditions performance.

The <u>laboratory tests</u> shall apply direct application of standard solution to the samplers (best possible) or exposure of samplers to spiked water from a sample dispenser (robustness and trueness). The laboratory tests provides information on the response of the samplers to carefully controlled parameters and best possible information on the performance of the samplers with chloroethene, a compound that can not be included in standpipe tests due to practical and health and safety considerations.

The <u>standpipe test</u> is intended to simulate ground water movement through a well established in the laboratory and to enable full control of solute concentrations. The standpipe test provides more realistic information on the performance of the samplers, while minimizing the variability of the test system as compared to field systems.

The <u>field tests</u> shall provide information on the robustness of the sampling system under the real conditions of groundwater investigations. In planning the field tests, varying aquifer and well conditions should be aimed at in order to allow for consideration of any impact of factors such as groundwater flow, well

construction or presence of other contaminants than the targets, as well as the impact of combined variation of robustness parameters.

7.2 Reference analysis

Reference analysis must be done under ISO 17025 accreditation /6/ using a GC-MS-SIM P&T method (EPA 624.2 or equivalent /7/) and must be documented to satisfy the analytical requirements set for groundwater investigations in Denmark, see Table 4 and the application and performance parameter definitions, Appendix 3.

Compound	Limit of detection	Precision	Trueness	Range of application
	µg/L	%	%	µg/L
All	0.03	5	90-110	0.03-2000

Table 4 Required analytical quality for reference analysis

7.3 Data management

Data storage, transfer and control must be done in accordance with the requirements of ISO 9001 /8/ enabling full control and retrieval of documents and records. The filing and archiving requirements of the DHI Quality Manual must be followed (10 years archiving).

7.4 Quality assurance

The quality assurance of the tests must include control of the reference system, control of the test system and control of the data quality and integrity.

The test plan and the test report will be subject to review by the expert group as part of the review of this verification protocol and the verification report, see Figure 2.

As this verification is a joint verification with the US EPA ETV, auditing from Battelle AMS Center is to be included in the test quality assurance.

7.5 Test report

The test report must follow the principles of template of the DHI NOWATECH verification center quality manual template /4/ with data and records from the tests presented. For this joint verification, the principles (contents) of the US ETV format must be complied with as well.

8 EVALUATION

The evaluation includes calculation of the performance parameters, see Section 5.4 for definition, evaluation of the data quality based upon the test quality assurance, see Section 7.4 for requirements, and compilation of the additional parameters as specified in Section 5.5.

8.1 Calculation of performance parameters

Calculations are done according to generally accepted statistical principles such as those described in /9/ and as described in Table 5, referring also to the test design shown in Table 3.

Parameter	Calculation	Explanations
Limit of	$LoD = 2 \times t_{acc}(f) \times S_{cc}$	toss (7) is the Student's t factor for
detection, LoD	200 - 2 11 10 1980 9 11 24	f = n - 1 degrees of freedom n
		being the number of
		measurements.
		\mathfrak{P}_r is the standard deviation of the
		measurements under repeatability
		conditions
Precision	$D_i = x_{i,\text{max}} - x_{i,\text{min}} $	D _i is the range at level i
(repeatability or		x_{imin} and x_{imax} are the lowest and
reproducibility),	$\overline{x}_{i} = \frac{\sum x_{i}}{\sum x_{i}}$	highest measurements at level i
standard	n n	
deviation RSD	$d = D_i$	<i>a</i> is the mean relative range for
	$u_i - \frac{\overline{x_i}}{\overline{x_i}}$	
	$\overline{d} = \frac{\Sigma d_i}{\Sigma d_i}$	
	$m_{\overline{1}}$ + 100	
	$RSD = \frac{d * 100}{\%}\%$	
	1.693	
Trueness, T	$\sum x_i$	\overline{x}_i is the mean of Sorbisense
	$x_i = \frac{m}{n}$	measurements at level i, x _i
	$-\sum_{i=1}^{n} y_i$	y_i is the mean of reference
	$y_i = \frac{m}{n}$	measurements at level i, y _i
	$T_t = \frac{\overline{X_t}}{X_t} \times 10096$	Ti is the trueness at level i
	$r_t = \frac{1}{y_t}$	y is the true value of the analyte
	$T_t = \frac{\overline{x_t}}{m} \times 100\%$	levels
	Y T	
	$T = \frac{d T_1}{m}$	
Range of	Visual identification of linear range,	
application	linear regression of results within linear	
	range to yield slope, intercept and	
Dobuotoooo	coefficient of regression (r).	
Robustness	each specific parameter studied.	
Robustness,	$\sum c_i \times t_i$	y_T is the true, mean concentration
concentration	$\gamma_T = \frac{1}{\Sigma t_t}$	over the exposure period
integration		c _i and t _i are the concentrations and
		exposure times for each
Toot of mean	I≣_dix Ø	concentrations steps
against true	$\frac{ \mu - c \times \sqrt{n}}{ \lambda } > t_{0.97*}(f)$	two sided test at 05% confidence
value	gr - weeking a	level n is number of
		measurements and c is the true
		concentration.

Table 5 Calculations used for the test results

Parameter	Calculation	Explanations
Test of mean against mean value	$\frac{\overline{x_t} - \overline{y_t}}{s_t} > t_{0.978}(v)$ $s_d = \sqrt{\frac{n+m}{n \times m} \times \frac{\Sigma(x-\overline{x})^2 + \Sigma(y-\overline{y})^2}{n+m-2}}$	The test requires that the variances are equal, v is $n+m-2$, and s_d is the standard deviation of the difference between the two means.

For field measurements, the reference measurements will be used as the true values.

For laboratory and standpipe measurements, concentrations obtained from preparation of the matrices are checked against the reference measurements (reference samples). If preparation based concentrations match the reference measurements, mean trueness within 100% \pm 2 x RSD (relative standard deviation), these are used as true values for the test measurements. If not, the reference measurements are used as the true values.

Calculations will be performed in Excel 2007 set up for the purpose with the equations required.

8.2 Evaluation of test data quality

The information of the test report on the reference system, the test system and data quality and integrity control will be evaluated against the requirements set in this protocol and the objectives set in the test plan.

The spreadsheet used for the calculations will subject to control on a sample basis (spot validation).

The external audit reports prepared by Battelle AMS Center, see Section 7.4, will be evaluated and major findings compiled and reported.

8.3 Compilation of additional parameters

8.3.1 User manual

The verification criterion for the users manual is that it describes the use of the samplers adequately and understandable for the typical sampler and sampling planner. This criterion is evaluated through evaluation of a number of specific points of importance, see Table 6 for the parameters to include.

A description is complete, if all essential steps are described, if they are illustrated with a figure or a photo, where relevant, and if the descriptions are understandable without reference to other guidance.

Parameter	Complete	Summary	No	Not relevant
	description	description	description	
Product				
Principle of operation				
Intended use				
Performance expected				
Limitations				
Preparations				
Unpacking				
Transport				
Assembly				
Installation				
Function test				
Operation				
Steps of operation				
Points of caution				
Accessories				
Maintenance				
Trouble shooting				
Safety				
Chemicals				
Power				

Table 6 Criteria for user manual evaluation

8.3.2 Product costs

The capital investment costs and the operation and maintenance cost will be itemized based upon a determined design basis /10/, see Table 7 for the items that will be included.

Table 7 List of capital cost items and operation and maintenance cost items per product unit (sample)

Item type	Item	Number	None
Capital			
Site preparation			
Buildings and land			
Equipment			
Utility connections			
Installation			
Start up/training			
Permits			
Operation and maintenance			
Materials, including chemicals			
Utilities, including water and energy			
Labor			
Waste management			
Permit compliance			

The design basis will be described and the cost items relevant for the Sorbisense sampler listed. Note that the actual costs for each item is not compiled and reported.

8.3.3 Occupational health and environment

The risks for occupational health and safety and for the environment associated with the use of the product will be compiled. The compilation will list chemicals used during product operation and classified as toxic, T, or very toxic, Tx, for human health and/or very environmentally hazardous (N) according to /11/. The information will be given as amount used per product unit (sample), see Table 8 for format.

Table 8 Compilation of classified chemicals used during product operation

Compound	CAS number	Classification	Amount used per product unit

Additional risks from installing, operating and maintaining the product will be evaluated, compiled and reported, if relevant. In particular, risks for human health associated with power supply and danger of infections will be considered.

9 VERIFICATION SCHEDULE

The verification is planned for 2008-9. The overall schedule is given in Table 9.

Task	Timing
Application definition document	May 2008
Verification protocol with test plan	November 2008 to January 2009
Test	January to March 2009
Test reporting	March 2009
Verification	March 2009
Verification report	March 2009
Report document review	April 2009
Verification statement	April 2009

Table 9 Verification schedule

10 QUALITY ASSURANCE

The quality assurance of the verification is described in Table 10 and Figure 2, and the quality assurance of the tests in the test plan but summarized here, as well as in the process document /2/.

Table	10 QA	plan fo	or the	verification
rubic	10 001	piuni		venneauon

	D	HI	Battelle AMS Center	US EPA ETV	Expert Group
Initials	ALJ	LSC	ZW	LD, JMK, EH	CP, DM, MS
Tasks					
Plan document with verification protocol and test plan	Review	-	-	Review	Review
Test system	-	Audit	Audit	-	-
Report document with test report and verification report	Review	-	-	Review	Review

Internal review of plan and report documents is done by chief engineer Anders Lynggaard Jensen (ALJ), and test system audit (see test plan) is done following the GLP audit procedure by a trained auditor: head of laboratory products Louise Schlüter (LSC).

The Battelle quality manager, Zachary Willenberg (ZW), will perform a technical systems audit (TSA) during this verification and test.

EPA QA staff, Lauren Drees (LD), John McKernan (JMK) and Evelyn Hartzell (EH) will do review of the plan and report documents.

The expert group will do review of the plan and report documents.

Reviews will be done using the NOWATECH review report template.

APPENDIX 1

Terms and definitions used in the verification protocol

The abbreviations and definitions used in the verification protocol and the test plan are summarized below.

Where discrepancies exist between NOWATECH and US EPA ETV terminology, definitions from both schemes are given.

Word	NOWATECH	US ETV
ADQ	Audit of data quality: An examination	
	of a set of data after is has been	
	collected and 100% verified by	
	project personnel, consisting of	
	tracing at least 10% of the test data	
	from original recording through	
	transferring, calculating, summarizing	
	and reporting.	
AMS Center	Advanced Monitoring Systems	
	Center at Battelle	
Analysis	Analysis of Sorbisense samplers at	
	the vendor identified laboratory	
Analytical	Independent analytical laboratory	
laboratory	used to analyze reference samples	
Application	The use of a product specified with	
	respect to matrix, target, effect and	
	limitations	
A-UBA	Umweltbundesamt Austria	
BTEX	Benzene, toluene, ethylbenzene and	
	xylenes	
CEN	European Committee for	
	Standardization	
CWA	CEN Workshop agreement	
DHI WMC	(ETV) Water Monitoring Center at	
	DHI	
Direct	A test design where a standard	
application	solution is applied directly to the	
D 00	Sorbisense sampiers	
DUC	Dissolved organic carbon	
Drinking water	Control of drinking water quality	
control	against drinking water maximum	
	Concentrations.	
DS Effort	The way the target is affected in this	
Ellect	verification the way the target	
	compounds are measured	
EN	European standard	
FTV	Environmental technology verification	EPA program that develops generic
	(FTV) is an independent (third party)	verification protocols and verifies the
	assessment of the performance of a	performance of innovative
	technology or a product for a	environmental technologies that have
	specified application, under defined	the potential to improve protection of
	conditions and adequate quality	human health and the environment
	assurance.	
EU	European Union	
Evaluation	Evaluation of test data for a	An examination of the efficiency of a

Word	NOWATECH	US ETV
	technology product for performance	technology
	and data quality	
Experts	Independent persons qualified on a	Peer reviewers appointed for a
	technology in verification or on	verification
	verification as a process	
GC	Gas chromatography	
Groundwater	Investigation of groundwater	
investigation	contamination with measurements	
	controlled against groundwater	
	maximum concentrations.	
Groundwater	Baseline monitoring of groundwater	
monitoring	quality.	
GWS	Groundwater sampler	
ISO	International Standardization	
	Organization	
Laboratory	Test device designed for controlled	
sample	exposure of Sorbisense samplers to	
dispenser	test solutions.	
Limit of	Calculated from the standard	
detection	deviation of replicate measurements	
LoD	at less than 5 times the detection	
	limit evaluated. Corresponding to	
	less than 5% risk of false blanks.	
Limit of	Calculated from the detection limit,	
quantification	typically 3 times the LoD, the	
LoQ	concentration, where the blank	
	variation impacts the precision 20%.	
Matrix	The type of material that the product	
	is intended for	
mbgv	m below groundwater table	
mbs	m below surface	
Method	Generic document that provides	
	rules, guidelines or characteristics for	
	tests or analysis	
MS	Mass spectrometry	
MTBE	Methyl- <i>tert</i> -butylether	
NOWATECH	Nordic Water Technology Verification	
EIV	Centers	
P&T	Purge and trap	
PE	Performance evaluation: A	
	quantitative evaluation of a	
	measurement system, usually	
	involving the measurement or	
	analysis of a reference material of	
Derferences	known value or composition	
Performance	The effects foreseen by the vendor	
ciaim	on the target (s) in the matrix of	
Derferences	Intended use	
Performance	Parameters that can be documented	
parameters	quantitatively in tests and that	
	provide the relevant information on	
	the performance of an environmental	
Drasisian	The standard deviation white the	
Precision	replicate manufacturements have	
	replicate measurements, nere	
	measured under repeatability or	<u> </u>

Word	NOWATECH	US ETV
	reproducibility conditions.	
(Environmen-	Ready to market or prototype stage	(Environmental) technology
tal) product	product, process, system or service	
	based upon an environmental	
	technology	
PVC	Polyvinylchloride	
QA	Quality assurance	
Range of	The range from the LoD to the	
application	highest concentration with linear	
	response,	
Reference	Analysis by a specified reference	
analyses	method in an accredited (ISO 17025)	
	laboratory.	
Reference	Samples taken for and analyzed by a	
samples	specified reference method in an	
	accredited (ISO 17025) laboratory.	
Repeatability	The precision obtained under	
	repeatability conditions, that is with	
	the same measurement procedure,	
	same operators, same measuring	
	and same location, and replicate	
	measurements on the same	
	or similar objects over a short period	
	of time	
Reproducibility	The precision obtained under	
	reproducibility conditions, that is with	
	measurements that includes different	
	locations, operators, measuring	
	systems, and replicate	
	measurements on the same or	
	similar objects	
Robustness	% variation in measurements	
	resulting from defined changes in	
DOD	matrix properties.	
RSD	Relative standard deviation in %.	
Sampler	Sorbisense sorbent cartridge	
Samples	Samples taken with and analyzed	
Correction of	The execution reconnection	
Sampling	The sampling reservoir and venting	
system	Sorbisonso complete	
SIM	Selected ion monitoring	
SM	Standard Methods for the	
SIM	Examination of Water and	
	Wastewater latest edition	
Stakeholder		Buyers and users of technology
Olakonolaci		technology developers/vendors the
		consulting engineers the finance and
		export communities. aovernment
		permitters, regulators, first
		responders, emergency response.
		disaster planners, public interest
		groups, and other groups interested
		in the performance of innovative
		environmental technologies.
Standard	Generic document established by	

Word	NOWATECH	US ETV
	consensus and approved by a	
	recognized standardization body that	
	provides rules, guidelines or	
Standning	Characteristics for tests or analysis	
Standpipe	groundwater well	
Target	The property that is affected by the product, in this verification the target compounds measured.	
(Environmenta	The practical application of	An all-inclusive term used to describe
I) technology	knowledge in the environmental area	pollution control devices and systems, waste treatment processes and storage facilities, and site remediation technologies and their components that may be utilized to remove pollutants or contaminants from, or to prevent them from entering, the environment.
Test/testing	Determination of the performance of a product by parameters defined for the application	
Trueness	The % recovery of true value obtained either from knowledge on the preparation of test solutions or from measurements with reference methods.	
TSA	Technical system audit	
US EPA	United States Environmental Protection Agency	
Vendor	The party delivering the product or service to the customer	The technology developer, owner, or licensee seeking verification
Verification	Evaluation of product performance parameters for a specified application under defined conditions and adequate quality assurance	Establishing or proving the truth of the performance of a technology under specific, predetermined criteria, test plans and adequate data QA procedures
VOC	Volatile organic compounds, here the compounds listed as target compounds/analytical parameters	
VOX	Volatile halogenated organic compounds, here the halogenated compounds listed as target compounds/analytical parameters	
WS	VVORKSNOD (UNDER CEN)	

APPENDIX 2

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APPENDIX 3

Application and performance parameter definitions

This appendix defines the application and the relevant performance parameters application as input for verification and test of an environmental technology following the NOWATECH ETV method.

1 Applications

The intended application of the product for verification is defined in terms of the matrix, the targets and the effects of the product.

The Sorbisense GWS40 passive sampling system with samplers (cartridges) and analysis of the samplers is provided by the vendor as one product, and the verification shall accordingly see these two investigation steps as one.

1.1 Matrix/matrices

The matrix of the application is groundwater and the field of application is investigations on (potentially) contaminated groundwater (groundwater investigations). In groundwater investigations, the groundwater composition generally varies considerably, and the pressure on samplers will vary with depth to the sampling positions. The varying ionic strength, contaminant concentration and water pressure may impact the performance and this impact shall be evaluated as part of the verification.

1.2 Target(s)

The targets of the application are volatile organic contaminants, here mono-, di-, tri- and –tetrachloroethenes, BTEX and MTBE. Investigations of contaminated groundwater generally include both uncontaminated and strongly contaminated groundwater. The concentrations verified shall accordingly reflect the range from uncontaminated groundwater to highly contaminated groundwater. With the claimed application at sampling depths from 0.5 mbs to 5 mbs (m below surface), pressure variation in the range 1-1.5 atmosphere shall be verified. Furthermore, with the claimed application, groundwater ionic strengths within the range 10-100 mS/m shall be verified, corresponding to the 5-95 percentile of Danish groundwaters /5/.

1.3 Effects

The effects for the application are generally reported in terms of limit of detection (LoD), precision, trueness, range of application and robustness. The effects claimed by the vendor are given in Appendix table 1 for all target compounds.

The robustness is the change in trueness within the range of application for defined variations in water pressure, contaminant concentration, groundwater ionic strength and sampling time.

Compound	Limit of detection	Precision	Trueness	Range of application	Robustness
	µg/L	%	%	μg/L	%
Chloroethene	0.5	<20	>80	LoD-2000	100±30
1,1-Dichloroethene	0.5	<20	>80	LoD-2000	100±30
1,2-Dichloroethenes	0.5	<20	>80	LoD-2000	100±30
Trichloroethene	0.5	<20	>80	LoD-2000	100±30
Tetrachloroethene	0.5	<20	>80	LoD-2000	100±30
Benzene	0.5	<20	>80	LoD-2000	100±30
Toluene	0.5	<20	>80	LoD-2000	100±30
Ethylbenzene	0.5	<20	>80	LoD-2000	100±30
Xylenes	0.5	<20	>80	LoD-2000	100±30
MTBE	1	<20	>80	LoD-2000	100±30

Appendix table 1 Vendor claim of performance, general terms

1.4 Exclusions

Passive sampling at waste disposal sites is excluded from the defined application and is thus not covered by the verification, as the conditions with respect to ionic strength and DOC are outside the ranges covered by the verification conditions. Groundwater baseline monitoring and drinking water control are excluded as well, as the passive sampler will not satisfy the detection limit requirements for this purpose, see Chapter 0.

2 General performance Requirements

No formal performance requirements for the application have been identified in the European Union or the US.

The conventional performance parameters of analytical and monitoring methods and equipment are limit of detection (LoD), precision (repeatability and reproducibility), trueness, specificity, linearity and matrix sensitivity. The uncertainty of measurements may be used to summarize the performance. Parameters may be added to characterize e.g. on-line or on-site monitoring instruments. The listed parameters cover the requirements set or implemented in international standards and by testing and verification operators /12-16/.

2.1 **Regulatory requirements**

The general requirement for analytical quality in water monitoring in Europe will be established with the adoption of the Commission Directive on technical specifications for chemical analysis and monitoring of water status /17/ requiring not more than 25% relative standard deviation at the level of the relevant water quality standards. The limit of quantification, LoQ, must be at or below 30% of the relevant water quality standard (WQS), corresponding to a limit of detection at or below 10% of the WQS. The LoQ is as defined in ISO 6107-2: 2006 /18/. The Groundwater Directive /19/ only sets an absolute requirement for monitoring of tri- and tetrachloroethene during groundwater
monitoring without stating the water quality standard and the quality requirement.

The European Directive on drinking water /20/ defines performance requirements for methods used for control of drinking water quality for the VOCs benzene, tri- and tetrachloroethene, among others. These values cover the chemical analysis only, and quality requirements for drinking water control would mostly be seen as stricter than for groundwater investigations. The drinking water based performance requirements for analysis only should therefore be seen as strict compared to groundwater monitoring including also sampling, see Appendix table 2.

Compound	Limit of detection	Precision	Trueness	Range of application	Robustness
	µg/L	%	%	µg/L	%
Trichloroethene	1	25	75-125	-1	-
Tetrachloroethene	1	25	75-125	-	-
Benzene	0.25	25	75-125	-	-

Appendix table 2 Regulatory requirements from the European drinking water directive

The Monitoring Certification Scheme of the British Environment Agency does not provide performance standards for groundwater or drinking water monitoring /12/.

Danish statute on quality requirements for environmental control /21/ specifies the requirements for control and monitoring of mono-, di-, tri- and – tetrachloroethenes and benzene in groundwater as shown in Appendix table 3. The detection limits stated are not justified by the maximum concentrations for groundwater, except for for chloroethene, see Section 2.2.

Again, it should be noted that the requirements cover analysis only and must thus be seen as stricter than required for methods including sampling.

Compound	Limit of detection	Precision	Trueness	Range of application	Robustness
	µg/L	%	%	µg/L	%
Chloroethene	0.03	5	100±10 ²	-	-
1,1-Dichloroethene	0.03	5	100±10	-	-
1,2-Dichloroethenes	0.03	5	100±10	-	-
Trichloroethene	0.03	5	100±10	-	-
Tetrachloroethene	0.03	5	100±10	-	-
Benzene	0.03	5	100±10	-	-

Appendix table 3 Regulatory requirements for groundwater monitoring and control from the Danish analytical quality requirement statute

2.2 Application based requirements

The application of the samplers in groundwater investigations further defines performance requirements in terms of the contaminant concentrations

¹ -: no requirement

² Assuming a 5% relative standard deviation

monitored and controlled during investigations in general. The lower limit of concentrations to be monitored will in most cases be defined by the groundwater maximum concentrations (and as a lower limit the drinking water maximum concentrations) for the compounds in question, see Appendix table 4.

Compound	Groundwater		Drinking water	
	Denmark	EU	US	WHO
	µg/L	µg/L	μg/L	µg/L
Chloroethene	0.2	0.5	2	0.3
1,1-Dichloroethene	1	-	7	30
1,2-Dichloroethenes	1	-	70-100	50
Trichloroethene	1	10	5	70
Tetrachloroethene	1	10	5	40
Benzene	1	1	5	10
Toluene	5	-	1000	700
Ethylbenzene	-	-	100	300
Xylenes	5	-	10*10 ³	500
MTBE	2-5	-	20-40	-

Appendix table 4 Summary of groundwater and drinking water maximum concentrations, as summarized in /22/ and /23/

A general requirement for the limit of detection of 1/10 of the maximum concentration is applied widely, and the derived limits of detection are compiled in Appendix table 5. Required detection limits for both drinking water and groundwater control are in the same ranges in Austria.

For the Danish groundwater monitoring program (GRUMO), requirements for detection limits are as given in Appendix table 5 /24/. It should be noted, that the detection limits required here for groundwater monitoring do not comply with those required in Danish statute on quality requirements for environmental control /21/ covering also monitoring of the compounds in groundwater as shown in Appendix table 5.

Compound	Groundwater maximum concentration based	Drinking water maximum concentration based			Groundwate r monitoring based
	Denmark	EU	US	WHO	Denmark
	µg/L	µg/∟	µg/L	µg/L	µg/∟
Chloroethene	0.02	0.05	0.2	0.03	0.05
1,1-Dichloroethene	0.1	-	0.7	3	-
1,2-Dichloroethenes	0.1	-	7	5	-
Trichloroethene	0.1	1	0.5	7	0.02
Tetrachloroethene	0.1	1	0.5	4	0.02
Benzene	0.1	0.1	0.5	1	0.04
Toluene	0.5	-	100	70	0.04
Ethylbenzene	-	-	10	30	-
Xylenes	0.5	-	1000	50	0.02
MTBE	0.2	-	2	-	-

Appendix table 5 Summary of detection limit requirements derived from the groundwater and drinking water maximum concentrations and for the Danish groundwater monitoring programme, 2003

Application based requirements for trueness and precision have generally not been stated to the same degree as for the limits of detection, mainly because regulatory compliance rules in most cases do not consider the uncertainty of control results.

No requirements for range of application and robustness have been identified. In practical performance of site investigations, the dissolved concentrations range from below detection limit to the limit of solubility. The upper limit of concentrations to be monitored will thus in most cases be defined by the solubilities of the target compounds are summarized in Appendix table 6.

Appendix table 6 Summary of target compound solubilities

Compound	Water solubility
	μg/L
Chloroethene	2.8*10 ⁶
1,1-Dichloroethene	3.3*10 ⁶
1,2-Dichloroethenes	3.5-6.3*10 ⁶
Trichloroethene	1.4*10 ⁶
Tetrachloroethene	0.24*10 ⁶
Benzene	1.8*10 ⁶
Toluene	0.55*10 ⁶
Ethylbenzene	0.17*10 ⁶
Xylenes	0.16-0.20*10 ⁶
MTBE	1.8*10 ⁶

3 State of the art performance

Whereas a broad range of studies on the performance of analytical methods and sampling methods for VOC in groundwater have been published, independent and comparative studies of passive samplers used for VOC monitoring in groundwater are scarce. Examples of reported performances (sampling and analysis) are compiled in Appendix table 7.

Sampler	Limit of detection	Precision	Trueness	Range of applicatio	Robustne ss	Reference
	µg/L	%	%	µg/L	%	
GORE-	-	14-21	-	5-2000	-	/25/
SORBER						
USGS PDB	-	0.9-4.3	86-118	2-500	-	/26/
Dialysis	0.1-5	17	100%	0.2-25*10 ³	-	/27/
membrane						
sampler						
USGS PDB		21			-	

Appendix table 7 Summary of state of the art performance for passive samplers

Reported performance (sampling and analysis) as obtained with reference sampling is given in Appendix table 8.

³ Verified range of application, practical range may differ

Sampler	Limit of detection	Precision	Trueness	Range of applicatio	Robustne ss	Reference
				n		
	µg/L	%	%	µg/L	%	
Grab sampling	-	12%	-	5-2000	-	/25/
Grab sampling	-	1.1-9.8	-	2-500	-	/26/
Low purge pump sampling	-	15	-	0.2-25*10 ³	-	/27/

Appendix table 8 Summary of state of the art performance for reference samplers

The precision results obtained with the passive samplers do not greatly differ from the precision values obtained with reference sampling methods. As the precision data obtained with the reference methods will generally be accepted for groundwater monitoring and control, the precision data obtained with the passive samplers should also be considered acceptable.

4 **Performance parameter definitions**

The statement of regulatory and application based requirements in terms of the analytical quality rather than the combined quality of analysis and sampling, as relevant for passive samplers, makes the identification of relevant criteria difficult for passive samplers.

Only a limited number of studies on the contributions of sampling and analysis, respectively, to the limit of detection, precision and trueness of groundwater monitoring and control have been published. Therefore, the regulatory and application based requirements needs identified for analytical performance can not be directly translated into the combined sampling and analysis performance requirements relevant for passive samplers.

The discrepancies between requirements based upon different approaches when comparing Appendix table 2, Appendix table 3 and Appendix table 5, further hampers the identification of relevant criteria.

Therefore, relevant performance parameters for the application are set in Appendix table 9 based upon regulatory and application based requirements and state of the art performance.

In order to address the general definition of performance parameters in terms of analytical quality only, information on this using the sampler should be obtained from the responsible laboratory for comparison, if possible.

In addition to the straight forward performance parameters of limit of detection, precision, trueness and range of application, the robustness shall be

tested for the critical parameters identified here: variations in water pressure, contaminant concentration, groundwater ionic strength and sampling time.

Compound	Limit of	Precision	Trueness	Range of	Robustness
	uelection	0/	0/	application	0/
	µg/L	%	%	µg/L	%
Chloroethene	0.02-0.05	<25	75-125	LoD-1*10 ⁶	85-115
1,1-Dichloroethene	0.1-1	<25	75-125	LoD-1*10 ⁶	100±25
1,2-Dichloroethenes	0.1-1	<25	75-125	LoD-1*10 ⁶	100±25
Trichloroethene	0.1-1	<25	75-125	LoD-1*10 ⁶	100±25
Tetrachloroethene	0.1-1	<25	75-125	LoD-0.1*10 ⁶	100±25
Benzene	0.1-1	<25	75-125	LoD-1*10 ⁶	100±25
Toluene	0.5-5	<25	75-125	LoD-0.1*10 ⁶	100±25
Ethylbenzene	0.5-5	<25	75-125	LoD-0.1*10 ⁶	100±25
Xylenes	0.5-5	<25	75-125	LoD-0.1*10 ⁶	100±25
MTBE	0.2-2	<25	75-125	LoD-1*10 ⁶	100±25

Appendix table 9 Relevant ranges of performance parameters for groundwater investigations









Sorbisense GWS40 Passive Sampler

Joint test plan

Volatile organic compounds in groundwater



Version approved

Sorbisense GWS40 Passive Sampler

Joint test plan

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	Gerald Heinicke Mette Tjener Andersson		2009		
			by		
	Joint test plan	GHE	CHG	CHG	
Revision	Description	Ву	Checked	Approved	Date
Key wo	ords	Classifica	tion		
	Environmental technology verification, passive	🖾 Open			
	sampler, groundwater	🔲 Internal			
		Prc	prietary		

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2 INTRODUCTION

This joint test plan is the implementation of a test design developed for verification of the performance of an environmental technology following the NOWATECH ETV method.

The verification is a joint verification with the US EPA ETV scheme and the Advanced Monitoring Systems Centre, Battelle, see the verification protocol /1/ for details on organization and implications. The compliance of the test with both scheme's requirements is ensured through a process document /2/.

2.1 Verification protocol reference

This test plan is prepared in response to the test design established in the Sorbisense GWS40 Passive Sampler, verification protocol, Volatile organic compounds in groundwater, Version final draft, December 2008 /1/.

2.2 Name and contact of vendor

Sorbisense A/S, Niels Pedersens Allé 2, DK-8830 Tjele, Denmark, phone +45 8999 2505, +45 8999 2599.

Contact: Hubert de Jonge, e-mail hubert@sorbisense.com.

The laboratory responsible for the analysis of samples (subcontractor to the vendor) is: ALcontrol Laboratories, Steenhouwerstraat 15, 3194 AG Hoogvliet, Netherlands,

Contact: Jaap Willem Hutter, e-mail j.hutter@alcontrol.nl

2.3 Name of center/test responsible

NOWATECH Water Monitoring ETV Center , DHI, Agern Allé 5, DK-2970 Hørsholm, Denmark.

Test responsible: Gerald Heinicke, e-mail ghe@dhigroup.com, phone +45 95 16 92 68, mobile +45 29 91 07 15.

US EPA Advanced Monitoring System Center, Battelle Memorial Institute, 505 King Avenue, Columbus, Ohio 43201-2693, US.

Test responsible: Anne M. Gregg (AMG), e-mail gregga@battelle.org, phone +1 614-424-7419

2.4 Expert group

The expert group assigned to this test and responsible for review of test plan and test report includes:

Dietmar Müller (DM), e-mail <u>dietmar.mueller@umweltbundesamt.at</u>, Contaminated Sites, Umweltbundesamt, Spittelauer Lände 5, 1090 Wien, Austria, phone +43-(0)1-313 04/5913

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Cynthia Paul (CP), e-mail <u>paul.cindy@epa.gov</u>, U.S. Environmental Protection Agency, 919 Kerr Research Drive, P.O. Box 1198. Ada, OK 74820, US, phone: +1 580-436-8556.

3 TEST DESIGN

The test design outlined in the test protocol is summarized in Table 1. The term "samples" is used for samples taken with the Sorbisense sampler, whereas the term "reference samples" is used for water samples taken for reference analysis. Acronyms are explained in Appendix 1.

If nothing else is stated below, the standard conditions for the stand pipe include mid range ionic strength (30-70 mS/m conductivity), a sampling period of 6 days and a sampling depth of 0.5 m (0.05 atm overpressure).

In Table 1, labels are given for each experiment and for experiments with different levels, a new label is given for each level.

Table 1 Test design

Performance parameters	Laboratory ¹	Stand pipe ²	Field
Limit of detection (LoD)	Direct application to samplers of VOX standard dilution in 7 replicates in the range LoD-5 x LoD. Triplicate analysis of VOX standard dilu- tion. Exp. H	 7 replicate samples in the range LoD-5 x LoD, spiked conc. 7 reference samples distributed over the sampling period. Exp. J 	n.a. ³
Precision (repeatability and reproducibility) Range of application Trueness Robustness, general	Direct application in 7 replicates to samplers of VOX standard at 10% of range. Exp. L	Triplicate samples and three ref- erence samples, the later distrib- uted over the sampling period, each at 5 spiked VOC conc. 10, 25, 50, 75, 100 % of range Exp. N, P, R, T, V	Single samples and reference samples at three (Sorbisense) or four (reference) times from a total of 5 wells at 1-3 sites, inherent concentrations. Exp. AA, AB, AC, AD, AE
Robustness, specific Reference for the robust- ness test levels	Triplicate samples at 1 spiked 50% range VOC concentration ⁴ , 1 mid range ionic strength (35 mS/m) and 1 mid range sampling time (6 days) from the sample dispenser. Three reference samples distributed over the sampling period. Exp. BA	Precision test above.	n.a.
Sampling depth	n.a.	Triplicate samples at 1 spiked VOC mid range concentration, 0.5 atm. overpressure. Three reference samples distrib- uted over the sampling period. Exp. CA	n.a.
Ionic strength	Triplicate samples at 1 spiked mid range VOC concentration, 2 ionic strengths (10 and 100 mS/m), (6 days),	n.a.	n.a.

¹ Direct application is done with chloroethene and the other chlorinated compounds, but without the BTEX and MTBE in the standard, other experiments are done with the full VOC set without chloroethene ² Standpipe experiments are done with the full VOC set without chloroethene ³ n.a.: not applicable ⁴ from pure chemicals, without chloroethene

Performance parameters	Laboratory ¹	Stand pipe ²	Field
	from the sample dispenser. Three reference samples distributed over the sampling period for each ionic strength. Exp. DA, EA		
Sampling time	Triplicate samples at 1 spiked VOC mid range concentration, mid range ionic strength and 2 sampling times (3 and 9 days), from the sample dispenser. Three reference samples distributed over the sampling period for each sam- pling time. Exp. FA, GA	n.a.	n.a.
Concentration integration	Triplicate samples at a step VOC con- centration, 3 concentrations (20, 50 and 80 % of range), each at 1/3 of 6 days sampling period, from the sample dis- penser. Three reference samples distributed over the sampling period. Exp. HA	n.a.	n.a.

3.1 Test sites

Both the laboratory tests and the standpipe tests will be conducted in the DHI laboratory buildings, Hørsholm, Denmark.

The field tests will be carried out on contaminated groundwater sites in the Copenhagen area.

3.1.1 Types

The test sites are summarized in Table 2.

Scale	Address/site	Site de- tails	VOC profile for test
Laboratory	DHI premises	None	All target compounds
Standpipe	DHI premises	None	All target compounds
	Søborg hovedgade, Søborg	AFV 6	All target compounds
	Søborg hovedgade, Søborg	AFV 4	All target compounds, low conc.
Field	Søborg hovedgade, Søborg	B109	Chlorinated solvents
Field	Farum Bytorv, Farum	AFV1	BTEX + MTBE, high concentrations
	Røde Vejrmølle, Roskildevej, Al-	K4	BTEX + MTBE, intermediary concen-
	bertslund		trations

Table 2 Summary of test sites

Please, note that the field test sites are preliminary and may change after site data compilation and inspections. Changes will be documented by an amendment to this document.

3.1.2 Addresses

See Table 2.

3.1.3 Descriptions

See Table 2.

3.2 Tests

The test program has been prepared to provide the information and to apply the approaches presented for analytical quality control for water analysis (ISO 13530) /3/ and for performance test of on-line sensors/analysing equipment (ISO 15839) /4/. The field tests have been prepared to comply with the test requirements in the Cost Agreement (pre-standard) on verification of monitoring technologies for groundwater site characterization (CEN/WS 32:2008) /5/.

The test design, as described in Table 1, includes three test scales: laboratory, stand pipe and field.

For chloroethene (vinyl chloride), the performance is only tested in a simplified laboratory design (direct application, best possible LoD, repeatability precision and trueness) and in the field (worst realistic reproducibility precision and robustness) due to difficulties preparing, obtaining and handling chloroethene solutions.

3.2.1 Test methods

No standard methods exist for testing of passive samplers for groundwater monitoring. The test methods have accordingly been prepared for the purpose (see Appendix 3), with reference to the Ground Water Sampling Technologies Verification Test Plan prepared for the US EPA ETV program /6/. Preparation of test solutions, reagents and chemicals are described in Appendix 3 as well. It should be noted that the methods in Appendix 3 are described at the detailed level of a work instruction for direct implementation in the laboratory.

For standpipe tests, groundwater, see Appendix 3.7, was used for preparing test solutions, and for laboratory tests, (clean) water, also see Appendix 3.7, was used. All dilutions were prepared in water.

For the laboratory and standpipe tests, custom-made stainless steel test devices have been being prepared, see below.

For <u>direct application laboratory tests</u>, a standard solution with chlorinated compounds only is applied to the samplers directly with a syringe, followed by equilibration and flushing with water using the sample dispenser, see Appendix 3.1 for method description (no illustration).

For the <u>laboratory tests</u>, a sample dispenser device, Figure 1, is designed as a closed system that enables direct exposure of samplers to test solutions with known and stable VOC concentrations by conveying the test solution from a closed container by gravity, see Appendix 3.2. The container is equipped with spiking port, sampling port and magnetic stirrer to maintain homogeneous conditions in the sample container.





The <u>standpipe test</u> device, Figure 2, is designed with a closed container filled with test solution where the sampling system with samplers can be suspended from the top, see Appendix 3.3. Air from the sample reservoir is vented through an air hose. The container is equipped with sampling ports and mixing is ensured through continuous pumping from top to bottom.

For both the sample dispenser and the standpipe, the air entering the container to replace dispensed liquid is saturated with VOCs at the same concentration as in the container, by using an air wash bottle.

Figure 2 Standpipe



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The <u>field sampling</u>, Figure 3, is done by suspending the sampling systems with samplers in the screened intervals in depth with the pump of established wells with con-

tinuous pumping (monitoring wells or pump-and-treat wells), see Appendix 3.4. Reference samples are taken from the pumped streams (pump depth sampling strategy).

Figure 3 Field sampling



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In cases, where the pump is deeper than the maximum sampling depth or the installations do not allow for positioning the sampler in pump depth, a position above is chosen (above pump sampling strategy). The sampler position is always within the screened interval and within the same aquifer unit as the pump. In such cases, a low volume reference sampling pump is positioned at the same depth as the sampler intake.

In all cases, a low purge sampling strategy is followed, allowing only for flushing of sampling equipment.

3.2.2 Test staff

The test staff is test responsible Ph.D. Gerald Heinicke (GHE), field responsible Mette T. Andersson (MTA) and test technician Susanne Klem (SEK).

3.2.3 Test schedule

The test schedule is given in Table 3, see Table 1 for identification of experiment labels.

Task	Week number 2008						W	eek no.	2009
	47	48	49	50	51	52	1	2	3
Test plan	x	х	х	х	х	х	х		
Pre-testing dispenser	x		х						
Test using dispenser									BA
Direct application	H, L								
Set up standpipe				Х	х				
Test using standpipe									J
Test field									
Test report draft									
Test report QA									
Test report									

Table 3 Test schedule

Task	Week number 2009									
	4	5	6	7	8	9	10	11	12	13
Test plan										
Pre-testing dispenser										
Test using dispenser	DA	EA	FA,	GA	HA					
Direct application										
Set up standpipe										
Test using standpipe	N	Р	R	Т	V	CA				
Test field			AA, A	B, AC, A	D, AE					
Test report draft						х				
Test report QA							Х	Х	х	
Test report										х

3.2.4 Test equipment

The test equipment includes (working procedures):

Laboratory sample dispenser (Appendix 3.2)

Stand pipe (Appendix 3.3)

Field sampling (Appendix 3.4)

The laboratory and stand pipe equipment has been designed and produced for the purpose. Equipment test procedures are described in Appendix 3.5.

General laboratory equipment procedures including cleaning and calibration are those described and ISO 17025 accredited /7/ for the DHI laboratories under the laboratory services manual of the DHI Quality Management System /8/.

3.2.5 Type and number of samples

The types and number of samples are summarized in Table 4.

	Laboratory	Stand pipe	Field
Limit of detection	7 analyses	7 samples	
	3 reference analyses	7 reference samples	
		3 reference analysis	
Precision	7 analyses	15 samples	15 samples
Range of application		15 reference samples	20 reference samples
Trueness		5 reference analysis	
Robustness, general			
Robustness, specific			
Reference for the ro-	3 samples		
bustness test levels	3 reference samples		
	1 reference analysis		
Sampling depth		3 samples	
		3 reference samples	
		1 reference analysis	
Ionic strength	6 samples		
	6 reference samples		
	2 reference analysis		
Sampling time	6 samples		
	6 reference samples		
	2 reference analysis		
Concentration inte-	3 samples		
gration	3 reference samples		
	3 reference analysis		
Samples per test	32 samples	25 samples	15 samples
scale	29 reference sam-	34 reference sam-	20 reference samples
	ples/analyses	ples/analyses	
Samples totally ⁵		72 samples	
	107	reference samples/analy	yses

Table 4 Summary of type and number of samples

The term analysis covers analysis of Sorbisense samplers that have been exposed to standard solutions by direct application. The term "samples" is used for samples taken with the Sorbisense sampler and analyzed accordingly.

The term reference analyses covers analysis of standards and standard dilutions after a reference method in an accredited (ISO 17025) laboratory. The term reference samples covers sampling of water and analysis after a reference method in an accredited (ISO 17025) laboratory.

The analytical program shown above includes 26 reference analyses of standard and stock solutions, as well as of dilutions. If the stability of the solutions and the analytical precision proves satisfactory in the initial part of the program, the number of replicate reference analysis may be reduced.

The water and the groundwater used in laboratory tests will be controlled for blanks, and the groundwater further characterized for general groundwater parameters, see Appendices 2 and 3.7.

⁵ Includes also pretesting samples and analyses not in the above rows

In addition to the number of test samples, samples controlling the test systems will be required as described in Appendix 3.5. The total number of samples for this purpose is 18 reference samples. The samples will include test system blank samples.

3.2.6 Operation conditions

The operation conditions applied during the verification of the product are:

- Sampling temperature: ambient 5-25°C
- Sampling depth: 0.5-5 m below the water surface
- Sample volume: up to 600 mL
- Sampling period: up to 9 days
- Sampling replicates: one sampler per sampling event

3.2.7 Operation measurements

During operation, the following operation conditions are recorded, as relevant, see Appendix 5 for data recording and reporting forms:

- Sampling temperature
- Depth of sample intake
- Sample volume
- Sampling period

3.2.8 Product maintenance

Samplers are kept in sealed bags as delivered from the vendor at ambient temperature until used. Opened bags with unused samplers are resealed until used.

No further maintenance is prescribed for the equipment.

3.2.9 Health, safety and wastes

The use of the product does not imply special health, safety and waste issues.

Laboratory work during testing will be done according to the DHI Safety Rules that are compliant with the extensive Danish rules for safe occupational health and the European regulations of work with chemicals. Work with VOC spiked solutions will be done using nitrile rubber gloves.

Field work will be done according to the DHI rules for safe field work included in the DHI Safety Rules.

Chemicals and test solutions are discarded according to Danish regulations for chemical waste by collection and destruction, *in casu* by collection in drums followed by controlled destruction.

4 REFERENCE ANALYSIS

The reference analysis applies to an aliquot of the test solutions that will be submitted to an analytical laboratory for analysis. These samples will verify the actual concentrations of the test solutions and the results will be compared to the results of the product in this verification.

4.1 Analytical laboratory

Reference analyses are done by Eurofins Danmark A/S, Smedeskovvej 38, DK-8464 Galten, Denmark.

Contact Rita Splidt Pedersen, Eurofins Miljø A/S, +45 70 22 42 66.

4.2 Analytical parameters

The analytical parameters and the target VOC are given in Table 5.

Table 5 Analytical parameters

Analytical parameters	
Chloroethene	Benzene
1,1-Dichloroethene	Toluene
1,2-Dichloroethenes	Ethylbenzene
Trichloroethene	Xylenes
Tetrachloroethene	MTBE

4.3 Analytical methods

The analysis are done using purge and trap gas chromatography with mass spectrometry detection in the selected ion monitoring mode (P&T GC-MS-SIM) according to the packages given in Table 6.

Analytical	Parameter	Limit of	Uncertainty
package		detection	
		µg/l	%
	Trichlorethene	0,02	7,5
	Tetrachlorethene	0,02	9,2
DR124 chlorinated solvents	1,1-Dichlorethene	0,02	8,5
and degradation projects	trans-1,2-dichlorethene	0,02	8,2
	cis-1,2-dichlorethene	0,02	14
	Chloroethene	0,02	7,7
	Benzene	0,02	7,4
	Toluene	0,02	8,9
DR102 BTEX	Ethylbenzene	0,02	9,4
	o-Xylene	0,02	7,4
	m+p-Xylene	0,02	7,3
23145 MTBE	MTBE	0,1	7,0

Table 6 Analytical packages, parameters and performance expectations from the contracted laboratory

The analytical method is based upon EPA Method 624 /9/ and ISO 15680 /10/ (see Appendix 2 for details).

4.4 Analytical performance requirements

The analytical performance requirements are given in Table 7.

It should be noted that the uncertainties stated by the laboratory, Table 6, includes both the random error under reproducibility conditions (requirements set here for the precision under repeatability conditions) and the systematic errors (requirements set here for the trueness).

For MTBE, concern may be raised whether the laboratory will be able to satisfy the required limit of detection.

Compound	Limit of	Precision	Trueness	Range of
	detection			application
	µg/L	%	%	µg/L
Chloroethene	0.03	5	90-110	0.03-2000
1,1-Dichloroethene	0.03	5	90-110	0.03-2000
1,2-Dichloroethenes	0.03	5	90-110	0.03-2000
Trichloroethene	0.03	5	90-110	0.03-2000
Tetrachloroethene	0.03	5	90-110	0.03-2000
Benzene	0.03	5	90-110	0.03-2000
Toluene	0.03	5	90-110	0.03-2000
Ethylbenzene	0.03	5	90-110	0.03-2000
Xylenes	0.03	5	90-110	0.03-2000
MTBE	0.03	5	90-110	0.03-2000

Table 7 Required analytical performances

4.5 Preservation and storage of reference samples

All water samples for VOC reference analysis are taken in 3 x 40 mL autosampler vials with Teflon lined screw caps as delivered from the laboratory contracted for reference sample analysis. The samples are not preserved but stored cold $(1-5^{\circ}C)$ and dark until delivered to the laboratory within a maximum of 3 days.

5 DATA MANAGEMENT

In general, the data filing and archiving procedures of the DHI Quality Management System will be followed.

5.1 Data storage, transfer and control

The data to be compiled and stored are summarized in Table 8.

Analytical raw data will be filed and archived according to the specifications of the laboratories quality management systems under their ISO 17025 accreditation, Eurofins for reference analysis and AlControl for sample analysis (Sorbisense sampler analysis).

Table 8 Data compilation and storage summary

Data type	Data media	Data recorder	Data re- cording tim- ing	Data storage
Test plan and report	Protected PDF files	Test responsi- ble, DHI	When ap- proved	Files and ar- chives at DHI
Test details in laboratory and field	Log book and pre-prepared forms	Technician, DHI	During collec- tion	Files and ar- chives at DHI
Calculations	Excel files	Test responsi- ble, DHI	During calcula- tions	Files and ar- chives DHI
Analytical re- ports	Paper	Test responsi- ble, DHI	When received	Files and ar- chives DHI

Forms for data recording are given in Appendix 5.

6 QUALITY ASSURANCE

The tests are performed under the quality management system of DHI which is ISO 9001 compliant /11/, but not certified. The DHI laboratories have ISO 17025 accreditations /7/ and OECD GLP approvals /12/ for a range of tests and ISO 17025 for sampling of drinking water. As part of the ISO 17025 and GLP inspections, the procedures for general laboratory processes, quality assurance and documentation/archiving are assessed.

6.1 Test plan review

The test plan will be subject to internal review by the verification responsible from DHI WMC Verifications: senior chemist Christian Grøn. Additionally, the test plan will be subject to review by the Battelle Advanced Monitoring Center quality manager (Zachary Willenberg, respectively), as well as by the US EPA ETV AMS project officer, quality manager and ETV coordinator (John McKerna, Lauren Drees and Evelyn Hazell, respectively).

External review of the test plan will be done by the expert group assigned to this verification.

6.2 *Performance control – reference analysis*

General chemistry, reference sample analyses and reference analyses are done under the ISO 17025 accreditation of Eurofins.

The performance of Eurofins for the reference analysis will be evaluated (performance evaluation audit) from laboratory quality control data for the relevant period (precision under repeatability conditions, trueness). Data for the analytical quality control of the laboratory will include duplicate control samples at two concentrations (acceptance within 100% \pm 10%) in each series and at the least one blank sample per 5 samples. The data from participation in a proficiency test arranged by Analytical Products Inc Sep-

tember 2008 will be evaluated for the demonstrated precision and traceability for the compounds in question for relevant matrices.

The detection limits and risks of false positives of the laboratory are controlled by submitting blank samples and low concentration samples as described in the test program.

The precision and trueness of the laboratory is further evaluated by analysis of stock, standard and spike solutions used for the test (26 reference analyses). The reference analysis includes analysis of a standard with analytical certificate and of solutions prepared from pure chemicals at DHI.

The analytical reference performance control is summarized in Table 9, with reference to Appendix 3.6 and 3.7 for information on water, standard solution (purchased standard with analytical certificate) and VOC solutions (prepared by DHI from pure chemicals).

Control type	Limit of detection	Precision	Trueness
VOX standard solution	-	-	Х
VOC solutions	-	Х	Х
Water	Х	-	-
Laboratory quality control	-	Х	Х
Proficiency test	-	Х	Х

Table 9 Summary of analytical reference performance control

6.3 Test system control

The laboratory test design includes test solutions of known concentrations, traceable back to added chemicals of p.a. quality or standards with analytical certificate, see Appendix 3.7 for specifications of purity etc.

The known concentrations will be used to pre-test the test design, see Appendix 3.5.

The water used for preparation of solutions will be controlled for contents of the target VOC as blanks by reference analysis.

The stability of the test concentrations will be controlled continuously during the tests by taking and analyzing reference samples distributed over the sampling periods, considering the "true concentrations" based upon added amounts and the reference analyses.

The control of the field test system is done using analysis of reference samples and field blank samples.

The analytical reference performance control is summarized in Table 10. Laboratory blanks/spiked samples are water, in some cases with ionic strength controlled by add-ing KCl, and the standpipe blanks/stand pipe samples are groundwater (matrix).

Table 10 Summary of test system control

Information/control type	Laboratory	Standpipe	Field
System contamination/blank	v	v	
sample reference analysis	~	^	-
System contamination/field			
blank sample reference	-	-	Х
analysis			
System trueness/spiked	V	v	
sample reference analysis	~	^	-
System variability/spiked	~	v	
sample reference analysis	~	^	-
System trueness/natural			v
sample reference analysis	-	-	^
System variability/natural			v
sample reference analysis	-	-	^

6.4 Data integrity check procedures

All transfer of data from printed media to digital form and between digital media are checked by spot check of not less than 5% of the data (test or field responsible). If errors are found in a spot check, all data from the transfer are checked.

6.5 Test system audits

Internal audit from DHI following the GLP audit procedure by a trained auditor is done, see the verification protocol for details.

The Battelle quality manager, Zachary Willenberg, will perform a technical systems audit (TSA) at least once during this verification and test. The purpose of this audit is to ensure that the verification test is being performed in accordance with the AMS quality management plan /13/, this test plan, published reference methods and any methods used in the tests. In the TSA, the Battelle quality manager, or designee, may review the reference methods used, and compare actual test procedures to those specified or referenced in this plan. In the TSA, the Battelle quality manager will observe testing in progress, observe the reference method sample preparation and analysis (when available), inspect documentation, and review technology-specific record books. He will also check standard certifications and may confer with other Battelle staff. A TSA report will be prepared, including a statement of findings and the actions taken to address any adverse findings. The AMS quality manager and the NOWATECH WMC verification responsible will receive a copy of Battelle's TSA report. The TSA findings will be communicated to technical staff at the time of the audit and documented in a TSA report.

The Battelle Quality Manager will perform an audit of data quality (ADQ). This will be a review of data acquisition and handling procedures and an audit of at least 10% of the data acquired in the test and verification. The Battelle Quality Manager will trace the data from initial acquisition, through reduction and statistical comparisons, to final reporting. All calculations performed on the data undergoing the audit will be checked.

6.6 Test report review

The test report will be subject to internal review by the verification responsible from DHI WMC Verifications: senior chemist Christian Grøn.

External review of the test report will be done by the expert group as part of the review of the verification report, that will include the full test report as an appendix.

7 TEST REPORT

The test report will follow the template of the DHI NOWATECH verification center quality manual /14/ and will be included as an appendix in the verification report. The test report will contain the test plan, except for this Chapter 7 on test report format, with the data and records from the tests to be inserted as new Chapter 7. For this joint verification, the principles (contents) of the US ETV format will be complied with as well.

7.1 Test site report

The test site report will include: well design drawing, well and pump data, operation data as outlined in Section 3.2.7 (forms in Appendix 5).

7.2 Test data report

The test data will include all data recorded during the test and the data reported by the analytical laboratories, see Appendix 5 for data forms.

7.3 Amendment report

The report section on deviations will compile all changes of this test plan occurring before testing with justification of deviations and evaluation of any consequences for the test data quality.

7.4 Deviations report

The report section on deviations will compile all deviations from this test plan occurring during testing with justification of deviations and evaluation of any consequences for the test data quality.

APPENDIX 1

Terms and definitions used in the test plan

The abbreviations and definitions used in the verification protocol and the test plan are summarized below.

Where discrepancies exist between NOWATECH and US EPA ETV terminology, definitions from both schemes are given.

Word	NOWATECH	US ETV
ADQ	Audit of data quality: An examination	
	of a set of data after is has been col-	
	lected and 100% verified by project	
	personnel, consisting of tracing at	
	least 10% of the test data from origi-	
	nal recording through transferring,	
	calculating, summarizing and report-	
	ing.	
AMS Center	Advanced Monitoring Systems Cen- ter at Battelle	
Analysis	Analysis of Sorbisense samplers at	
	the vendor identified laboratory	
Analytical	Independent analytical laboratory	
laboratory	used to analyze reference samples	
Application	The use of a product specified with	
	respect to matrix, target, effect and	
	limitations	
A-UBA	Umweltbundesamt Austria	
BTEX	Benzene, toluene, ethylbenzene and	
051	xylenes	
CEN	European Committee for Standardi-	
0)4/4		
	CEN Workshop agreement	
	DHI	
Direct applica-	A test design where a standard solu-	
tion	tion is applied directly to the Sorbi-	
	sense samplers	
DOC	Dissolved organic carbon	
Drinking water	Control of drinking water quality	
control	against drinking water maximum	
	concentrations.	
DS	Danish Standard	
Effect	The way the target is affected, in this	
	verification the way the target com-	
	pounds are measured	
EN	European standard	
ETV	Environmental technology verification	EPA program that develops generic
	(ETV) is an independent (third party)	verification protocols and verifies the
	assessment of the performance of a	performance of innovative environ-
	fied application under defined condi-	nemal lechnologies that have the
	tions and adoquate quality assur	buman health and the environment
	ance	
FU	European Union	
Evaluation	Evaluation of test data for a technol-	An examination of the efficiency of a
	ogy product for performance and	technology
	data quality	

ExpertsIndependent persons qualified on a technology in verification or on verifi- cation as a processPeer reviewers appointed for a verifi- cationGCGas chromatographyGroundwaterInvestigation of groundwater con- tamination with measurements con- trolled against groundwater maxi- mum concentrations.InvestigationGroundwaterBaseline monitoring of groundwater quality.Groundwater samplerInternational Standardization Organi- zationLaboratoryTest device designed for controlled exposure of Sorbisense samplers to penserLimit of detec-Limit of detec-Calculated from the standard devia-	Word	NOWATECH	US ETV
technology in verification or on verification as a process cation GC Gas chromatography Groundwater Investigation of groundwater contamination with measurements controlled against groundwater maximum concentrations. Groundwater Baseline monitoring of groundwater maximum concentrations. GWS Groundwater sampler ISO International Standardization Organization Laboratory Test device designed for controlled exposure of Sorbisense samplers to penser Limit of detec- Calculated from the standard devia-	Experts	Independent persons gualified on a	Peer reviewers appointed for a verifi-
GC Gas chromatography Groundwater Investigation of groundwater con- investigation tamination with measurements con- trolled against groundwater maxi- mum concentrations. Groundwater Baseline monitoring of groundwater monitoring quality. GWS Groundwater sampler ISO International Standardization Organization Laboratory Test device designed for controlled sample dis- exposure of Sorbisense samplers to penser test solutions.		technology in verification or on verifi-	cation
GC Gas chromatography Groundwater Investigation of groundwater con- tamination with measurements con- trolled against groundwater maxi- mum concentrations. Groundwater Baseline monitoring of groundwater quality. GWS Groundwater sampler ISO International Standardization Organi- zation Laboratory Test device designed for controlled exposure of Sorbisense samplers to penser Limit of detec- Calculated from the standard devia-		cation as a process	
Groundwater investigation Investigation of groundwater con- tamination with measurements con- trolled against groundwater maxi- mum concentrations. Groundwater monitoring Baseline monitoring of groundwater quality. GWS Groundwater sampler ISO International Standardization Organi- zation Laboratory Test device designed for controlled exposure of Sorbisense samplers to penser Limit of detec- Calculated from the standard devia-	GC	Gas chromatography	
investigation tamination with measurements controlled against groundwater maximum concentrations. Groundwater Baseline monitoring of groundwater maximum concentrations. GWS Groundwater sampler ISO International Standardization Organization Laboratory Test device designed for controlled sample dispenser test solutions. Limit of detec- Limit of detec- Calculated from the standard devia-	Groundwater	Investigation of groundwater con-	
International and the standard devia- trolled against groundwater maximum concentrations. Groundwater monitoring of groundwater monitoring quality. GWS Groundwater sampler ISO International Standardization Organization Laboratory Test device designed for controlled sample dis- exposure of Sorbisense samplers to penser Limit of detec- Calculated from the standard devia-	investigation	tamination with measurements con-	
International against groundwater mum concentrations. Groundwater monitoring of groundwater monitoring quality. GWS Groundwater sampler ISO International Standardization Organization Laboratory Test device designed for controlled sample dis- exposure of Sorbisense samplers to penser Limit of detec- Calculated from the standard devia-	invooliguton	trolled against groundwater maxi-	
Groundwater Baseline monitoring of groundwater monitoring quality. GWS Groundwater sampler ISO International Standardization Organization zation zation Laboratory Test device designed for controlled sample dis- exposure of Sorbisense samplers to penser Limit of detec- Calculated from the standard devia-		mum concentrations	
monitoring quality. GWS Groundwater sampler ISO International Standardization Organization Laboratory Test device designed for controlled sample dis- penser test solutions. test solutions.	Groundwater	Baseline monitoring of groundwater	
GWS Groundwater sampler ISO International Standardization Organi- zation Laboratory Test device designed for controlled sample dis- penser test solutions. Limit of detec- Calculated from the standard devia-	monitoring	quality	
ISO International Standardization Organization Laboratory Test device designed for controlled sample dis- exposure of Sorbisense samplers to penser test solutions. Limit of detec- Calculated from the standard devia-	GWS	Groundwater sampler	
Laboratory Test device designed for controlled sample dis- exposure of Sorbisense samplers to penser test solutions.	190	International Standardization Organi-	
Laboratory Test device designed for controlled sample dis- exposure of Sorbisense samplers to penser test solutions. Limit of detec- Calculated from the standard devia-	100	zation	
sample dis- penser exposure of Sorbisense samplers to test solutions. Limit of detec- Calculated from the standard devia-	Laboratory	Test device designed for controlled	
penser test solutions.	sample dis-	exposure of Sorbisense samplers to	
Limit of detec-	penser	test solutions.	
	Limit of detec-	Calculated from the standard devia-	
tion tion of replicate measurements at	tion	tion of replicate measurements at	
LoD less than 5 times the detection limit	LoD	less than 5 times the detection limit	
evaluated. Corresponding to less		evaluated. Corresponding to less	
than 5% risk of false blanks.		than 5% risk of false blanks.	
Limit of guanti- Calculated from the detection limit,	Limit of quanti-	Calculated from the detection limit,	
fication typically 3 times the LoD, the concen-	fication	typically 3 times the LoD, the concen-	
LoQ tration, where the blank variation im-	LoQ	tration, where the blank variation im-	
pacts the precision 20%.		pacts the precision 20%.	
Matrix The type of material that the product	Matrix	The type of material that the product	
is intended for		is intended for	
mbgy m below groundwater table	mbay	m below groundwater table	
mbs m below surface	mbs	m below surface	
Method Generic document that provides	Method	Generic document that provides	
rules, quidelines or characteristics for	mounou	rules quidelines or characteristics for	
tests or analysis		tests or analysis	
MS Mass spectrometry	MS	Mass spectrometry	
MTBE Methyl-tert-butylether	MTRE	Methyl- <i>tert</i> -butylether	
NOWATECH Nordic Water Technology Verification		Nordic Water Technology Verification	
ETV/	FTV	Centers	
P&T Durge and tran	D&T	Purge and tran	
DE Derformance evaluation: A quantita		Performance evaluation: A quantita	
tive evaluation of a measurement	r L	tive evaluation of a measurement	
system usually involving the meas-		system usually involving the meas-	
urement or analysis of a reference		urement or analysis of a reference	
material of known value or composi-		material of known value or composi-	
tion		tion	
Performance The effects foreseen by the vendor	Performance	The effects foreseen by the vendor	
claim on the target (s) in the matrix of in-	claim	on the target (s) in the matrix of in-	
tended use	olainn	tended use	
Performance Parameters that can be documented	Performance	Parameters that can be documented	
parameters quantitatively in tests and that pro-	narameters	quantitatively in tests and that pro-	
vide the relevant information on the	parameters	vide the relevant information on the	
performance of an environmental		performance of an environmental	
technology product		technology product	
Precision The standard deviation obtained from	Precision	The standard deviation obtained from	
renlicate measurements here meas-		renlicate measurements here meas	
ured under repeatability or repro-		ured under reneatability or repro-	
ducibility conditions		ducibility conditions	
(Environmen- Ready to market or prototype stage (Environmental) technology	(Environmen-	Ready to market or prototype stage	(Environmental) technology

Word	NOWATECH	US ETV
tal) product	product, process, system or service	
	based upon an environmental tech-	
	nology	
PVC	Polyvinylchloride	
QA	Quality assurance	
Range of ap-	The range from the LoD to the high-	
plication	est concentration with linear re-	
	sponse,	
Reference	Analysis by a specified reference	
analyses	method in an accredited (ISO 17025)	
	laboratory.	
Reference	Samples taken for and analyzed by a	
samples	specified reference method in an ac-	
	credited (ISO 17025) laboratory.	
Repeatability	The precision obtained under repeat-	
	ability conditions, that is with the	
	same measurement procedure, same	
	operators, same measuring	
	system, same operating conditions	
	and same location, and replicate	
	or similar objects over a short period	
	of time	
Reproducibility	The precision obtained under repro-	
	ducibility conditions, that is with	
	measurements that includes different	
	locations, operators, measuring sys-	
	tems, and replicate measurements	
Poblictnoss	% variation in measurements result	
Robustiless	// variation in measurements result-	
	nroperties	
RSD	Relative standard deviation in %	
Sampler	Sorbisense sorbent cartridge	
Samples	Samples taken with and analyzed	
Campics	after the Sorbisense method	
Sampling sys-	The sampling reservoir and venting	
tem	system used to operate the Sorbi-	
	sense samplers	
SIM	Selected ion monitoring	
SM	Standard Methods for the Examina-	
-	tion of Water and Wastewater, latest	
	edition	
Stakeholder		Buyers and users of technology,
		technology developers/vendors, the
		consulting engineers, the finance and
		export communities, government
		permitters, regulators, first respond-
		ers, emergency response, disaster
		planners, public interest groups, and
		other groups interested in the per-
		formance of innovative environmental
		technologies.
Standard	Generic document established by	
	consensus and approved by a rec-	
	ognized standardization body that	
	provides rules, guidelines or charac-	

Word	NOWATECH	US ETV
	teristics for tests or analysis	
Standpipe	Test device designed to simulate a groundwater well	
Target	The property that is affected by the product, in this verification the target compounds measured.	
(Environ- mental) tech- nology	The practical application of knowl- edge in the environmental area	An all-inclusive term used to describe pollution control devices and sys- tems, waste treatment processes and storage facilities, and site remedia- tion technologies and their compo- nents that may be utilized to remove pollutants or contaminants from, or to prevent them from entering, the envi- ronment.
Test/testing	Determination of the performance of a product by parameters defined for the application	
Trueness	The % recovery of true value ob- tained either from knowledge on the preparation of test solutions or from measurements with reference meth- ods.	
TSA	Technical system audit	
US EPA	United States Environmental Protec- tion Agency	
Vendor	The party delivering the product or service to the customer	The technology developer, owner, or licensee seeking verification
Verification	Evaluation of product performance parameters for a specified application under defined conditions and ade- quate quality assurance	Establishing or proving the truth of the performance of a technology un- der specific, predetermined criteria, test plans and adequate data QA procedures
VOC	Volatile organic compounds, here the compounds listed as target compounds/analytical parameters	
VOX	Volatile halogenated organic com- pounds, here the halogenated com- pounds listed as target com- pounds/analytical parameters	
WS	Workshop (under CEN)	

APPENDIX 2

Reference methods and references

1 Reference analysis, VOC

Water samples are taken as 40 mL samples in autosampler vials filled completely from the bottom and allow to overflow.

A precise volume of subsample is transferred from the sampler vial to the airsparger via a sample loop and using helium as the pressure gas. The subsample is purged with helium and the purged compounds trapped on a VOCARB 3000 adsorbent, followed by thermal desorption at 240°C and transfer of desorbed compounds to the gas chromatograph (GC). GC separation is followed by selected ion monitoring and quantification against external standard.

Selectivity is ensured by applying a maximum limit of 20% deviation of mass ratios for the selected masses from reference run.

The equipment used is Tekmar Aquatek 70/Velocity XPT and Agilent 6890 GC/5973 or 5975 MS

Standard method references are EPA Method 624.2 /9/ and ISO 15680 /10/.

2 General chemistry

Groundwater from wells in the field test will be characterized for general chemistry parameters using the below given methods. Analysis for pH and conductivity is done on-line in the field.

Parameter	Method	Parameter	Method
рН	DS 287	DOC	EN 1484
Conductivity	DS 288	Iron	SM3500C
Nitrate	EN 10304	Ammonium	DS 224
Fluoride	EN 10304	Sodium	SM3500C
Chloride	EN 10304	Potassium	SM3500C
Bicarbonate	DS 256	Calcium	SM3500C
Sulphate	EN 10304	Magnesium	SM3500C

General chemistry data for groundwater for the laboratory tests, see Appendix 3.7, will be obtained from the water work delivering the water.

2 References

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APPENDIX 3

In-house test methods
The in-house test methods are the detailed specifications (work instructions) of the tests to be performed including specific information on the practical work planned, Appendices 3.1 to 3.4.

The pre-testing is described in Appendix 3.5 and the check of solutions used in Appendix 3.6.

Reagents are described in Appendix 3.7 and apparatus in Appendix 3.8.

The volumes of solutions used for different experiments are summarized in Appendix 3.9.

The storage and shipping of samples is described in Appendix 3.10.

Direct application of halogenated hydrocarbons standard to samplers.

For personal safety and to avoid contamination, wear nitrile gloves for all handling of equipment.

- a) Place 7 samplers in the fume cupboard in a vial stand.
- b) Add the spike (50 μ L of 24.7 μ g/mL VOX standard dilution for 5xLoD, 50 μ L standard solution for 10 % of range) directly into the adsorber resin of the sampler using a 50 μ L gas tight syringe.
- c) Place each sampler into a separate 100 mL glass bottle with PTFE-lined screw cap.
- d) Place 3-4 of the 100 mL bottles into a 2 L wide-neck glass bottle with Teflon-lined screw cap.
- e) Let the samplers equilibrate at 4°C for 24 hours at least.
- f) Remove the samplers from the glass bottles.
- g) Connect 3 of the samplers to the sample dispenser using new 1/16" capillaries.
- h) Convey 300 ml (collect in 500 mL graduated cylinders) of water through each sampler during ca. 4 hours. This is done in batches of 2-3 samplers, before the sample dispenser has been used with VOCs, but after the blank test of the sample dispenser.
- i) Prepare the samplers for shipping.

Laboratory sample dispenser.

For personal safety and to avoid contamination, wear nitrile gloves for all handling of equipment.

1 **Preparations**

- a) Start with the clean, empty dispenser, lid attached, air exchange pipe detached from the wash bottle. Make sure the stir bar in the dispenser is positioned in the middle of the dispenser bottom and rotating when stirrer is switched on.
- b) Connect the sampler capillaries to the 1/16 inch fittings. Place the ends of the capillaries above the lid of the lab dispenser to avoid leakage.
- c) Add 35 mL potassium chloride stock solution to the dispenser through the spiking port, using a 100 mL syringe with Luer lock. For the tests with different ionic strength add 10 mL for 10 mS/m or 100 mL for 100 mS/m). Rinse with 100 mL of water
- d) Fill the dispenser with water (see separate instruction below)
- e) Add the VOC spike to the dispenser (see separate instruction below).
- f) Fill the wash bottle with water up to the 5.5 L-mark, using the PTFE tubing.
- g) Add the adequate spike directly to the wash bottle (under the water surface) and close the wash bottle.
- h) Connect the exchange pipe and tighten the fittings.
- i) Start the magnetic stirrers in the wash bottle.
- j) Take down the end of the capillaries from the dispenser. Let at least 1 mL go to drain.
- k) Make sure that 30 minutes have passed since the magnetic stirrers have been started; then, connect a sampler to each capillary.
- l) Start the magnetic valve timer.
- m) Note the time and possible deviations

2 Filling the dispenser with water

- a) Attach one end of the PTFE tube to the vertical nozzle of the sampling port. Attach the other end to the water tap (MilliQ).
- b) Open the two-way valve in the lid of the dispenser.
- c) Open the sampling port valve. Open the water tap to fill the dispenser until water overflows from the open two-way valve.
- d) Close the water tap. Close the spiking port valve.
- e) Remove the PTFE tube and close the open ends with tinfoil.
- f) Drain 200 mL of water from the dispenser through the sampling port. Measure conductivity and temperature in the drained water.
- g) Continue with step 1e

3 Addition of spike to the lab dispenser

- a) Start the dispenser's magnetic stirrer
- b) Open a stock solution vial with appropriate volume.
- c) Fill the appropriate amount into the suitable gastight syringe with Luer lock, with needle attached.

- d) Remove the needle from the syringe and connect the syringe to the dispenser's spiking port.
- e) Open the spiking port valve and add the contents to dispenser.
- f) Close the spiking port valve and remove the syringe.
- g) Attach the needle and fill syringe with methanol. Detach the needle and add also the methanol to the dispenser.
- h) Close the spiking valve, and remove the syringe.
- i) Fill a 100 mL syringe with Luer lock with water. Add the water to the dispenser. Close the spiking port valve and remove the syringe.
- j) Close the two-way valve in the lid of the dispenser.
- k) Continue with step 1f

4 During exposure

- a) Control the amount of liquid that has passed through each sampler after ¹/₂ the sampling time and full sampling time by collecting in pre-weighed 1000 mL bottles.
- b) Take water phase sample from the dispenser after 2 hours, ¹/₂ the sampling time and full sampling time, following the *sampling instruction*.

5 Reference sampling instructions

- a) Wipe the vertical nozzle of the sample tap with acetone-soaked paper tissue. Rinse the nozzle with water from a bottle, dry with paper tissue.
- b) Set the magnetic valves to open.
- c) Open the sample tap and drain 25 mL to waste.
- d) Place the nozzle in a 40 mL P&T vial, open the sample valve by ca. 45 degrees and fill the vial slowly from below. Let the sample overflow for at least 3 seconds. Close the sample tap, and close the vial.
- e) Repeat c) for the 2 following P&T vials.
- f) Start the magnetic valve timer.
- g) Repeat a) to clean the nozzle after sampling.
- h) Store cold 1-5°C and dark for no more than 3 days and transfer to laboratory.

5 End of exposure

- a) Stop both magnetic stirrers.
- b) Open the two-way valve in the lid of the dispenser.
- c) Drain the dispenser through the sample tap, into containers for disposal.
- d) Remove the samplers and send them for analysis.
- e) Detach the air exchange pipe from the wash bottle⁶ and tilt the container towards the sample tap to empty completely.
- f) Detach the magnetic valve from the other side of the wash bottle.
- g) Empty the wash bottle. Attach the air exchange pipe lightly until next use.

⁶ Whenever detaching the connections of the wash bottle, detach the nut on the steel side of the fitting, not on the glass side.

Standpipe

1 Preparations

Wear nitrile gloves for all handling of equipment.

- a) Mount the needed number of samplers with samplers into the empty standpipe. Tighten with the provided strings
- b) Connect the air exchange pipes to the lid.
- c) Close the standpipe.
- d) Open the two-way valve in the lid of the standpipe.
- e) Fill the standpipe from the bottom with ground water, using PTFE tubing, until water overflows from the open two-way valve.
- f) Stop the water flow, close the sampling port valve and remove the PTFE tubing.
- g) Drain 200 mL from the standpipe. Measure conductivity and temperature in the drained water.
- h) Start the circulation pump.
- i) Add the spike to the standpipe (*see separate instruction*).
- j) Fill the wash bottle with water up to the 5.5 L-mark, using the PTFE tubing.
- k) Add an adequate spike directly to the wash bottle (under the water surface) and close the wash bottle.
- 1) Connect the air exchange pipe.
- m) Start the magnetic stirrer in the wash bottle. Start the magnetic valve timer.
- n) Note the time and possible deviations

2 Addition of spike to the standpipe

- a) Make sure the magnetic valves are closed.
- b) Open a stock solution vial with appropriate volume.
- c) Fill the appropriate amount into the suitable gastight syringe with Luer lock, with needle attached.
- d) Remove the needle from the syringe and connect the syringe to the standpipe's spiking port.
- e) Open the spiking port valve and add the contents to standpipe.
- f) Close the spiking port valve and remove the syringe.
- g) Fill a 100 mL syringe with Luer lock with water. Add the water to the dispenser. Close the spiking port valve and remove the syringe.
- h) Close the two-way valve in the lid of the standpipe.
- i) Continue with step 1h

3 During the exposure

Take water phase sample from the dispenser after 2 hours, ½ the sampling time and full sampling time, following the *sampling instruction*.

4 **Reference sampling instructions**

- a) Wipe the vertical nozzle of the sample tap with acetone-soaked paper tissue. Rinse the nozzle with water from a bottle, dry with paper tissue.
- b) Set the magnetic valves to open.
- c) Open the sample tap and drain 25 mL to waste.
- d) Place the nozzle in a 40 mL P&T vial, open the sample valve by ca. 45 degrees and fill the vial slowly from below. Let the sample overflows for at least 3 seconds. Close the sample tap, and close the vial.
- e) Repeat c) for the 2 following P&T vials.
- f) Start the magnetic valve timer.
- g) Repeat a) to clean the nozzle after sampling.
- h) Store cold 1-5°C and dark for no more than 3 days and transfer to laboratory.

4 End of exposure

- a) Stop the magnetic stirrer in the air wash bottle.
- b) Stop the recirculation pump.
- c) Open the two-way valve in the lid of the standpipe.
- d) Remove the air exchange pipe.
- e) Drain the standpipe into containers for disposal, using the sample tap.
- f) Open the standpipe and take up the sampler
- g) Remove the samplers and send them for analysis
- h) Measure the sampled water volume by collecting in 1000 mL pre-weighed glass bottles and weighing
- i) Tilt the container towards the sample tap to empty completely.
- j) Empty the wash bottle. Attach the air exchange pipe lightly.

Field sampling

1 Data compilation

First planning step includes data compilation in order to allow for the detailed planning:

Full Address	Well iden- tification	Well regis- tration number	Contact, name, phone, e-mail
Søborg hovedgade 23, Søborg	AFV 6	201.5751	Region Hovedstaden, Jens Lerche Mortensen, +45 48 20 53 33, jens.lerche.mortensen@ regionh.dk
Søborg hovedgade 17-19, Søborg	AFV 4	201.5749	Region Hovedstaden, Jens Lerche Mortensen, +45 48 20 53 33, jens.lerche.mortensen@ regionh.dk
Søborg hovedgade 189, Søborg	B109	201.5743	Region Hovedstaden, Jens Lerche Mortensen, +45 48 20 53 33, jens.lerche.mortensen@ regionh.dk
Farum Bytorv 36, Farum	AFV1	193.2283	Jord•Miljø, Charlotte Juhl Søegaard, +45 35 82 04 02, cjs@jordmil.dk
Roskildevej 2-4, Al- bertslund	К4	200.5185	Jord•Miljø, Charlotte Juhl Søegaard, +45 35 82 04 02, cjs@jordmil.dk

Well iden- tification	Filter (mbs) ⁷	Diameter (mm)	Groundwa- ter table (mbs)	Geology
AFV 6	5-13.3	125	7.7	Sand
AFV 4	9.5-14.5	160	11.5	Sand
B109	13.5-46.5	165	22	Sand
AFV1	8-15	160	7.3	Sand
K4	7.5-9.5	160	5.3	Limestone

Well iden-	Pump	Yield	Pumping pattern	Access for
tification	(mbs)	(m ³ /hour)		samplers
AFV 6	13	0.2	Start at water level < 12 mbs, stop at 13 mbs	Should be possible to place next to pump

⁷ mbs: meter below surface

Well iden-	Pump	Yield	Pumping	Access for
tification	(mbs)	(m ³ /hour)	pattern	samplers
AFV 4	14	1.5-2.5	Start at water level < 8.5 mbs, stop at 9.5 mbs	Should be possible to place next to pump
B109	37	2-2.5	Continuously, stop for cleaning 15 min/day	Good access
AFV1	12	1.5-2	Continuously	Should be possible to place next to pump
K4	8.5	2.5	3 min pumping, 5 min stop, etc.	Should be possible to place next to pump

2 Reference sampling strategy

The second planning step is to select the reference sampling strategy (pump depth or above pump) and to select the sampling $(0.5-5 \text{ mbgvt}^8)$ and reference sampling positions. The pump depth sampling strategy applies sampling of the pumped stream. In cases where the pump is positioned deeper than the maximum sampler depth, a position above the pump is chosen, the above pump strategy.

Well iden- tification	Sampler position (mbs)	Sampler posi- tion to in well groundwater table distance (m)	Reference sampling position (mbs)	Sampling strategy
AFV 6	13	1-2	13	Pump depth
AFV 4	14	3.5-4.5	14	Pump depth
B109	27	4	27	Above pump
AFV1	12	1	12	Pump depth
K4	8.5	0.5-3.5	8.5	Pump depth

3 Sampling

The sampling is done as follows:

- a) Mount a sampling system with a sampler in the well at the selected depth
- b) Secure the position with the provided string
- c) Leave the sampler in position for 6 days
- d) Take up the sampler
- e) Remove the sampler and send it for analysis
- f) Repeat twice

⁸ mbgv: m below groundwater table

The reference sampling is done as follows, for the pump depth strategy:

- a) Mount sampling tube upon pump exit sampling tap
- b) Flush tap and tube 5-10 minutes with lowest flow filling the tube
- c) Fill sample containers from the bottom and allow to overflow 2-3 times
- d) Store and preserve samples as prescribed and send to the laboratory

The reference sampling is done as follows, for the above pump strategy:

- a) Ensure that well pump is running at routine yield
- b) Lower the pump into the well to the selected depth
- c) Purge with the lowest flow filling the tube for 5-10 minutes, max 5% of stationary pump yield
- d) Ensure stability (within 10%) of indicator parameters (pH, dissolved oxygen, conductivity)
- e) Fill sample containers from the bottom and allow to overflow 2-3 times ensuring no head space in the containers
- e) Store and preserve samples as prescribed and send to the laboratory

Reference sampling is done before, between and after each sampling, totally 4 times.

Field blanks are prepared during the first reference sampling at each site. Water is transferred to sample bottles on site and the samples at stored, transported and analyzed as reference samples.

Sampling is done using a Grundfos MP1 pump equipped with 10 mm Teflon tubes, leaving the tubes in each well. Indicator parameters (pH, dissolved oxygen, conductivity, temperature) are measured in a flow through on-line cell.

Pre-testing

Laboratory sample dispenser

Objective	Test design	Samples
Blank test	Dispenser filled with water.	Triplicate reference sam- ples 1) of water, 2) from dispenser directly 30 min after addition, and 3) from
		dispenser after 6 days.
VOC stability in dispenser	Dispenser filled with water, spiked to 5xLoD.	Triplicate samples 1) of 0.1 g/L VOC solution, 2) from dispenser 30 minutes after addition, and 3) from dis- penser after 6 days.

Preparation of solutions for reference analysis

1 **10 g/L VOC stock solution**

Check of concentrations is done initially and each time a subsample is taken out for use.

Initial testing is done by filling a 1,5 mL capped vial with stock solution at -20 °C, using a low-flow pipette. Close vial. Produce triplicate vials in this way. Place each of the 1 mL capped vials in a larger capped vial. Store cold 1-5°C and dark for no more than 3 days and transfer to laboratory with information of concentration range.

Check during use: after using part the stock solution in the vial for spiking, fill one 1,5 mL capped vials with the solution using a gas-tight syringe. Close the vial and place it in a larger capped vial. Store cold 1-5°C and dark for no more than 3 days and transfer to laboratory with information on concentration range. Produce 2 more vials and keep at -20 °C for possible future reference

2 **0.1 g/L VOC solution**

Check during use is done after using part the solution in the vial for spiking by transferring 1 mL to a cap vial using a low-flow pipette. Close vial, shake well. Produce triplicate vials in this way. Close the vials and place each in a larger capped vial. Store cold 1-5°C and dark for no more than 3 days and transfer to laboratory with information of concentration range.

3 VOX Standard dilution

Check during use is done after using part the stock solution in the 6 mL vial with VOX standard dilution for spiking. Fill three 1.5 mL capped vials with the solution using a gas-tight syringe. Close the vials and place them in a P&T vial. Store cold 1-5°C and dark for no more than 3 days and transfer to laboratory with information of concentration range

Reagents

1 Water

Laboratory grade water from Millipore system with electrical conductivity below 10 M Ω /cm. Dissolved organic carbon below 0.1 mg/L and target VOC below the limit of detection 0.02 μ g/L is expected and verified in first blank test series.

2 Groundwater

Potable water as obtained from the supply network at DHI. Groundwater characteristics are as follows:

Parameter	Value	Parameter	Value
рН	7.6	DOC	1.5 mg C/L
Conductivity	71 mS/m	Iron	0.02 mg/L
Nitrate	2.4 mg/L	Ammonium	0.058 mg/L
Fluoride	0.62 mg/L	Sodium	30 mg/L
Chloride	44 mg/L	Potassium	3.8 mg/L
Bicarbonate	340 mg/L	Calcium	85 mg/L
Sulphate	21 mg/L	Magnesium	22 mg/L

Target VOC is below the limit of detection 0.02 μ g/L.

3 VOX standard

Standard solution "QTM Volatile Halocarbons Mix" produced by Supelco, purchased from Sigma-Aldrich, product number 48001, lot number LB59726, with analytical certificate, MFG date July 2008, nominal concentrations as follows (among other VOCs):

Compound	Nominal concentration
	μg/L
Chloroethene	1998
1,1-Dichloroethene	2000
trans-1,2-Dichloroethene	2000
cis-1,2-Dichloroethene	1951
Trichloroethene	2000
Tetrachloroethene	2000

4 Chemicals

Compound	Pro- ducer	Quality	Purity	Batch
1,1-Dichloroethene	Supelco	Analytical standard	99.9%	LB56468
trans-1,2- Dichloroethene	Supelco	Analytical standard	99.9%	LB57511

Compound	Pro- ducer	Quality	Purity	Batch
cis- 1,2- Dichloroethene	Fluka	Analytical standard	99.7%	7333X
Trichloroethene	Fluka	Puriss. p.a.	>99.5%	1368013
Tetrachloroethene	Supelco	Analytical standard	99.9%	LB56979
Benzene	Fluka	Puriss. p.a.	99.9%	1369911
Toluene	Fluka	Puriss. p.a.	>99.7%	1392028
Ethylbenzene	Fluka	Puriss. p.a.	>99.0%	1388758
m-Xylene	Fluka	Puriss. p.a.	>99.0%	1399073
o-Xylene	Fluka	Puriss. p.a.	>99.0%	1406896
MTBE	Fluka	Puriss. p.a.	>99.5%	1399802
Methanol	Fluka	Puriss. p.a. For trace analysis of chlorinated hydrocar- bons	≥99.8%	1379978
Potassium chloride	Fluka	Puriss. p.a.	>99.5%	80150

5 10 g/L VOC stock solution

Prepare a 10 g/L solution of each target VOC as follows:

- a) Fill a 250 mL volumetric flask (with glass stopper) with methanol, refrigerate to -20°C and mark the level.
- b) Keep 210 mL methanol in the volumetric flask, place on ice in fume cupboard.
- c) Add the volume indicated below of each chemical using low flow pipettes, starting with the highest boiling compound and keeping the pipette below the methanol surface while emptying. Close the flask after each addition.
- d) Refrigerate flask to -20°C
- e) Fill the volumetric flask to the new mark with methanol refrigerated to -20°C.
- f) Shake by hand until no phase difference is visible.
- g) Distribute the stock solution into 1.5, 3.5 and 10 ml capped vials using a low flow pipette. Check cap tightness (tight when cannot be twisted), wrap with aluminum foil and place in freezer, -20°C.

Compound	µL pipetted	Density	µg pipetted	Concentration g/L
		g/L		
Chloroethene	-	-	-	-
1,1-Dichloroethene	2 000	1.218	2 436 000	9.74
trans-1,2-Dichloroethene	2 000	1.2565	2 513 000	10.05
cis-1,2-Dichloroethene	1 500	1.2837	1 925 550	7.70
Trichloroethene	1 750	1.4642	2 562 350	10.25
Tetrachloroethene	1 500	1.6227	2 434 050	9.74
Benzene	2 750	0.87865	2 416 288	9.67
Toluene	2 750	0.8669	2 383 975	9.54
Ethylbenzene	2 750	0.867	2 384 250	9.54
o-Xylene	2 750	0.8802	2 420 550	9.68
m-Xylene	3 000	0.8642	2 592 600	10.37

Compound	µL pipetted	Density g/L	µg pipetted	Concentration g/L
MTBE	3 250	0.74	2 405 000	9.62

6 0.1 g/L VOC solution

Prepare a 0.1 g/L solution from the 10g/L solution as follows, directly before use.

- a) Open a 1.5 mL vial of 10 g/L stock solution
- b) Fill a 10 mL capped vial with 10 mL of methanol using a low flow pipette at 20°C±2°C
- c) Add 100 μ L of 10 g/L stock solution using a gas tight syringe with cemented needle, keep needle under methanol surface while emptying.
- d) Close the vial, check for tightness.
- e) Shake by hand.
- f) Use after letting the solution equilibrate at room temperature for 1 hour.

7 24.7 μg/L VOX standard dilution for direct application

Prepare a 24.7 µg/mL stock solution of halogenated hydrocarbons as follows:

- a) Add 6 mL of methanol to a 6 mL cap vial using a low flow pipette.
- b) Transfer 75 μ l of the 2000 μ g/mL volatile hydrocarbon standard to the cap vial using a 50 μ L gas tight syringe, keeping the needle below the methanol surface while emptying.
- c) Close the vial, check for tightness.
- d) Shake by hand.
- e) Use after letting the solution equilibrate at room temperature for 1 hour.

8 205 g/L KCl stock solution

Prepare a 205 g/l stock solution of potassium chloride:

- a) Place a 1000 mL volumetric flask with 200 mL of water
- b) Weigh the flask with water
- c) Add portions of KCl dried at 105°C overnight and shake well
- d) When all 205 g of KCl is dissolved, allow the solution to equilibrate to room temperature.
- e) Fill the flask to the mark.
- f) Transfer to a 1000 mL glass bottle and close with a blue cap screw lid.

Apparatus

1 Glass syringes and adapters

- a) 1 gas tight glass syringe with stainless steel Luer-lock, 2.5 mL
- b) 1 gas tight glass syringe with stainless steel Luer-lock, 5 mL
- c) 1 gas tight glass syringe with stainless steel Luer-lock, 10 mL
- d) 1 stainless steel Luer/Luer adapter with valve, each for lab dispenser and standpipe.
- e) 6 needles with Luer adapter, 22 ga (0.394 mm ID)
- f) 1 gas tight syringes with fixed needle, $25 \,\mu L$
- g) 2 gas tight syringes with fixed needle, $50 \,\mu L$
- h) 1 gas tight syringe with fixed needle, $100 \ \mu L$
- i) 1 gas tight syringe with fixed needle, $500 \ \mu L$
- j) 1 gas tight syringe with fixed needle, $1000 \ \mu L$
- k) Glass syringe with Luer lock, 100 mL

2 Glassware

- a) Volumetric flasks 250 mL and 1000 mL with glass stopper
- b) Low flow glass pipettes at 250, 500, 7500, 1000, 1250, 1500, 2000 and 2500 μL
- c) 3 stir bars, glass coated
- d) Capped vials for 1.5, 3.5, and 10 mL, caps with PTFE seals.
- e) 25 mL Erlenmeyer flask with mark
- f) 200 mL Erlenmeyer flask with mark
- g) 1000 mL bottle with blue screw cap
- h) 1000 mL bottles with red screw cap
- i) 500 mL graduated cylinders

3 Miscellaneous

- a) Micropipettes with tips at 100 μ L, 1 mL and 5 mL
- b) Dedicated, water flushed PTFE tubes, 8x6 mm diameter
- c) Nitrile gloves

3 Field

- a) Grundfos MP1 pump
- b) Transformer box
- c) 220 V generator
- d) Dedicated, water flushed 10 mm PTFE tubes
- e) On-line flow through devices (WTW) for monitoring of indicator parameters (pH, dissolved oxygen, conductivity)

Spike volumes and solutions

Experiment	Lab dispenser or	Wash bottle	Solution
-	standpipe (mL)	(mL)	
Н	0.050		VOX standard dilution
L	0.050		VOX standard
BA	4.00	0.55	10 g/L VOC stock solution
DA	4.00	0.55	10 g/L VOC stock solution
EA	4.00	0.55	10 g/L VOC stock solution
FA	4.00	0.55	10 g/L VOC stock solution
GA	4.00	0.55	10 g/L VOC stock solution
HA	1.50	0.2	10 g/L VOC stock solution
HA	4.00	0.55	10 g/L VOC stock solution
HA	6.00	0.85	10 g/L VOC stock solution
J	2.50	0.125	0.1 g/L VOC solution
Ν	2.00	0.10	10 g/L VOC stock solution
Р	5.00	0.25	10 g/L VOC stock solution
R	10.00	0.50	10 g/L VOC stock solution
Т	15.00	0.80	10 g/L VOC stock solution
V	20.00	1.00	10 g/L VOC stock solution
CA	10.00	0.50	10 g/L VOC stock solution

Managing, storing and shipping of samples/samplers

1 Managing samples

- a) Prior to each individual test, the sampling responsible (test technician or field responsible) labels the correct type and number of sample vials, according to information in the test plan and the data forms. For cap vials, only the labels are prepared, to avoid contamination.
- b) The test responsible checks the array of labeled sample bottles and labels against the test plan and the data forms.
- c) After sampling, the sampling responsible takes a photo of the sample vials and sends the photo to the test responsible immediately.
- d) The sampling responsible stores the sample vials.
- e) The test responsible prepares a requisition for analysis, and sends it to the sampling responsible.
- f) The sampling responsible ships the samples, and making sure that they are sent within the maximum stated storage time.
- g) The sampling responsible informs the test responsible immediately when the samples have been sent.
- h) The sampling responsible keeps a copy of the requisition with a note of the date of shipping.

2 Sample storing, reference samples

Water samples are taken in 40 mL P&T vials. Samples are stored cold 1-5°C and dark for no more than 3 days prior to transfer to the laboratory.

3 Sample storing, samplers

Samplers are equipped with protective caps in both ends, placed in transportation tubes and stored cold 1-5°C and dark for no more than 7 days prior to transfer to the laboratory.

4 Sample shipping, reference samples

Water samples (P&T vials) are sent in cooling boxes with cooling elements. Cap vials with stock solution or stock dilution are placed individually into a P&T vial and may be sent in a non-isolated package, with one cooling element.

5 Sample shipping, samplers

Samplers are packed in the transport tubes in a cardboard box at ambient temperature.

APPENDIX 4

In-house analytical methods

None

APPENDIX 5

Data reporting forms

Compound		Concentration mg/L				
Date	Dec.1, 2008 A1	Not taken A2	Not taken A3	Jan. 8, 2009 A4	Jan. 8, 2009 A5	Jan. 8, 2009 A6
1,1-Dichloroethene						
trans-1,2-Dichloroethene						
cis-1,2-Dichloroethene						
Trichloroethene						
Tetrachloroethene						
Benzene						
Toluene						
Ethylbenzene						
o-Xylene						
m+p-Xylene						
MTBE						

A Check of 10 g/L VOC stock solution, reference analyses, unopened vial

aA Check of 10 g/L VOC stock solution, reference analyses, used in test BA. Spare vials are not sent for analysis, but kept in the freezer.

Compound	Concentration g/L				
Date	Usage Date aA1	Spare Vial aA2	Spare Vial aA3		
1,1-Dichloroethene					
trans-1,2-Dichloroethene					
cis-1,2-Dichloroethene					
Trichloroethene					
Tetrachloroethene					
Benzene					
Toluene					
Ethylbenzene					
o-Xylene					
m+p-Xylene					
MTBE					

Compound	Concentration g/L			
Date	Usage Date bA1	Spare Vial bA2	Spare Vial bA3	
1,1-Dichloroethene				
trans-1,2-Dichloroethene				
cis-1,2-Dichloroethene				
Trichloroethene				
Tetrachloroethene				
Benzene				
Toluene				
Ethylbenzene				
o-Xylene				
m+p-Xylene				
MTBE				

bA Check of 10 g/L VOC stock solution, reference analyses, used in test DA. Spare vials are not sent for analysis, but kept in the freezer.

Compound	Concentration g/L			
Date	Usage Date cA1	Spare Vial cA2	Spare Vial cA3	
1,1-Dichloroethene				
trans-1,2-Dichloroethene				
cis-1,2-Dichloroethene				
Trichloroethene				
Tetrachloroethene				
Benzene				
Toluene				
Ethylbenzene				
o-Xylene				
m+p-Xylene				
MTBE				

cA Check of 10 g/L VOC stock solution, reference analyses, used in test EA. Spare vials are not sent for analysis, but kept in the freezer.

dA Check of 10 g/L VOC stock solution, reference analyses, used in test FA. Spare vials are not sent for analysis, but kept in the freezer.

Compound	Concentration g/L			
Date	Usage Date dA1	Spare Vial dA2	Spare Vial dA3	
1,1-Dichloroethene				
trans-1,2-Dichloroethene				
cis-1,2-Dichloroethene				
Trichloroethene				
Tetrachloroethene				
Benzene				
Toluene				
Ethylbenzene				
o-Xylene				
m+p-Xylene				
MTBE				

Compound	Concentration g/L				
Date	Usage Date eA1	Spare Vial eA2	Spare Vial eA3		
1,1-Dichloroethene					
trans-1,2-Dichloroethene					
cis-1,2-Dichloroethene					
Trichloroethene					
Tetrachloroethene					
Benzene					
Toluene					
Ethylbenzene					
o-Xylene					
m+p-Xylene					
MTBE					

eA Check of 10 g/L VOC stock solution, reference analyses, used in test GA. Spare vials are not sent for analysis, but kept in the freezer.

fA Check of 10 g/L VOC stock solution, reference analyses, used in test HA for 20 % of range. Spare vials are not sent for analysis, but kept in the freezer.

Compound	Concentration g/L			
Date	Usage Date fA1	Spare Vial fA2	Spare Vial fA3	
1,1-Dichloroethene				
trans-1,2-Dichloroethene				
cis-1,2-Dichloroethene				
Trichloroethene				
Tetrachloroethene				
Benzene				
Toluene				
Ethylbenzene				
o-Xylene				
m+p-Xylene				
МТВЕ				

Compound		Concer	ntration g/L	
Date	Usage Date gA1	Spare Vial gA2	Spare Vial gA3	
1,1-Dichloroethene				
trans-1,2-Dichloroethene				
cis-1,2-Dichloroethene				
Trichloroethene				
Tetrachloroethene				
Benzene				
Toluene				
Ethylbenzene				
o-Xylene				
m+p-Xylene				
MTBE				

gA Check of 10 g/L VOC stock solution, reference analyses, used in test HA for 50 % of range. Spare vials are not sent for analysis, but kept in the freezer.

hA Check of 10 g/L VOC stock solution, reference analyses, used in test HA for 80 % of range. Spare vials are not sent for analysis, but kept in the freezer.

Compound	Concentration g/L			
Date	Usage Date hA1	Spare Vial hA2	Spare Vial hA3	
1,1-Dichloroethene				
trans-1,2-Dichloroethene				
cis-1,2-Dichloroethene				
Trichloroethene				
Tetrachloroethene				
Benzene				
Toluene				
Ethylbenzene				
o-Xylene				
m+p-Xylene				
MTBE				

Compound	Concentration g/L				
Date	Usage Date iA1	Spare Vial iA2	Spare Vial iA3		
1,1-Dichloroethene					
trans-1,2-Dichloroethene					
cis-1,2-Dichloroethene					
Trichloroethene					
Tetrachloroethene					
Benzene					
Toluene					
Ethylbenzene					
o-Xylene					
m+p-Xylene					
MTBE					

iA Check of 10 g/L VOC stock solution, reference analyses, used in test N. Spare vials are not sent for analysis, but kept in the freezer.

jA Check of 10 g/L VOC stock solution, reference analyses, used in test P. Spare vials are not sent for analysis, but kept in the freezer.

Compound	Concentration g/L			
Date	Usage Date jA1	Spare Vial jA2	Spare Vial jA3	
1,1-Dichloroethene				
trans-1,2-Dichloroethene				
cis-1,2-Dichloroethene				
Trichloroethene				
Tetrachloroethene				
Benzene				
Toluene				
Ethylbenzene				
o-Xylene				
m+p-Xylene				
MTBE				

Compound	Concentration g/L			
Date	Usage Date kA1	Spare Vial kA2	Spare Vial kA3	
1,1-Dichloroethene				
trans-1,2-Dichloroethene				
cis-1,2-Dichloroethene				
Trichloroethene				
Tetrachloroethene				
Benzene				
Toluene				
Ethylbenzene				
o-Xylene				
m+p-Xylene				
MTBE				

kA Check of 10 g/L VOC stock solution, reference analyses, used in test R. Spare vials are not sent for analysis, but kept in the freezer.

IA Check of 10 g/L VOC stock solution, reference analyses, used in test T. Spare vials are not sent for analysis, but kept in the freezer.

Compound	Concentration g/L			
Date	Usage Date IA1	Spare Vial IA2	Spare Vial IA3	
1,1-Dichloroethene				
trans-1,2-Dichloroethene				
cis-1,2-Dichloroethene				
Trichloroethene				
Tetrachloroethene				
Benzene				
Toluene				
Ethylbenzene				
o-Xylene				
m+p-Xylene				
MTBE				

Compound	Concentration g/L			
Date	Usage Date mA1	Spare Vial mA2	Spare Vial mA3	
1,1-Dichloroethene				
trans-1,2-Dichloroethene				
cis-1,2-Dichloroethene				
Trichloroethene				
Tetrachloroethene				
Benzene				
Toluene				
Ethylbenzene				
o-Xylene				
m+p-Xylene				
MTBE				

mA Check of 10 g/L VOC stock solution, reference analyses, used in test V. Spare vials are not sent for analysis, but kept in the freezer.

nA Check of 10 g/L VOC stock solution, reference analyses, used in test CA. Spare vials are not sent for analysis, but kept in the freezer.

Compound		Concentration g/L			
Date	Usage Date nA1	Spare Vial nA2	Spare Vial nA3		
1,1-Dichloroethene					
trans-1,2-Dichloroethene					
cis-1,2-Dichloroethene					
Trichloroethene					
Tetrachloroethene					
Benzene					
Toluene					
Ethylbenzene					
o-Xylene					
m+p-Xylene					
MTBE					

Compound		Concentration g/L			
Date	Preparation Date B1	Preparation Date B2	Preparation Date B3		
1,1-Dichloroethene					
trans-1,2-Dichloroethene					
cis-1,2-Dichloroethene					
Trichloroethene					
Tetrachloroethene					
Benzene					
Toluene					
Ethylbenzene					
o-Xylene					
m+p-Xylene					
MTBE					

B Check of 0.1 g/L VOC solution, reference analyses, used in lab dispenser pre-testing

aB Check of 0.1 g/L VOC solution, reference analyses, used in test J

Compound		Concentration mg/L			
	Preparation	Preparation	Preparation		
Date	Date	Date	Date		
	aB1	aB2	aB3		
1,1-Dichloroethene					
trans-1,2-Dichloroethene					
cis-1,2-Dichloroethene					
Trichloroethene					
Tetrachloroethene					
Benzene					
Toluene					
Ethylbenzene					
o-Xylene					
m+p-Xylene					
MTBE					

C Check of VOX standard dilution, reference analyses

Compound	Concentration mg/L			
Date	Preparation Date C1	Preparation Date C2	Preparation Date C3	
Chloroethene				
1,1-Dichloroethene				
trans-1,2-Dichloroethene				
cis-1,2-Dichloroethene				
Trichloroethene				
Tetrachloroethene				

aD Check of MiliQ water from tap

Compound		Concen	tration µg/L	
Date	Sample 1 aD1	Sample 2 aD2	Sample 3 aD3	
Chloroethene				
1,1-Dichloroethene				
trans-1,2-Dichloroethene				
cis-1,2-Dichloroethene				
Trichloroethene				
Tetrachloroethene				
Benzene				
Toluene				
Ethylbenzene				
o-Xylene				
m+p-Xylene				
МТВЕ				

Compound		Concen	tration µg/L	
Date	Sample 1 bD1	Sample 2 bD2	Sample 3 bD3	
Chloroethene				
1,1-Dichloroethene				
trans-1,2-Dichloroethene				
cis-1,2-Dichloroethene				
Trichloroethene				
Tetrachloroethene				
Benzene				
Toluene				
Ethylbenzene				
o-Xylene				
m+p-Xylene				
MTBE				

bD Check of water from lab dispenser sample tap after 30 minutes.

D Check of water from lab dispenser sample tap after 6 days

Compound		Concen	tration µg/L	
Date	Sample 1 D1	Sample 2 D2	Sample 3 D3	
Chloroethene				
1,1-Dichloroethene				
trans-1,2-Dichloroethene				
cis-1,2-Dichloroethene				
Trichloroethene				
Tetrachloroethene				
Benzene				
Toluene				
Ethylbenzene				
o-Xylene				
m+p-Xylene				
МТВЕ				

Compound		Concen	tration µg/L	
Date:	Sample 1 cD1	Sample 2 cD2	Sample 3 cD3	
Chloroethene				
1,1-Dichloroethene				
trans-1,2-Dichloroethene				
cis-1,2-Dichloroethene				
Trichloroethene				
Tetrachloroethene				
Benzene				
Toluene				
Ethylbenzene				
o-Xylene				
m+p-Xylene				
MTBE				

cD Check of ground water (blank)

E VOC stability check of sample dispenser, reference samples after 30 minutes

Compound	Concentration µg/L			
Date	Sample 1 E1	Sample 2 E2	Sample 3 E3	
1,1-Dichloroethene				
trans-1,2-Dichloroethene				
cis-1,2-Dichloroethene				
Trichloroethene				
Tetrachloroethene				
Benzene				
Toluene				
Ethylbenzene				
o-Xylene				
m+p-Xylene				
MTBE				

Compound	Concentration µg/L						
Date	Sample 1 aE1	Sample 2 aE2	Sample 3 aE3				
1,1-Dichloroethene							
trans-1,2-Dichloroethene							
cis-1,2-Dichloroethene							
Trichloroethene							
Tetrachloroethene							
Benzene							
Toluene							
Ethylbenzene							
o-Xylene							
m+p-Xylene							
MTBE							

aE VOC stability check of sample dispenser, reference samples after 6 day

F and G are omitted, covered by experiment J.

	Η	LoD	direct	ap	olication	, samp	les
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Compound	Mass on sampler μg						
Date of spiking	ID	ID	ID	ID	ID	ID	ID
I emperature Chloroethene	H1	H2	H3	H4	H5	H6	H7
1,1-Dichloroethene							
1,2-Dichloroethenes							
Trichloroethene							
Tetrachloroethene							
Date of rinsing							
mL passed (measured)							
mL passed (by tracer salt)							

I is omitted, covered by experiment C.

J LoD standpipe, samples

Compound	Concentration µg/L					Dates	Temperatures		
	ID	ID	ID	ID	ID	ID	ID		
	J1	J2	J3	J4	J5	J6	J7		
1,1-Dichloroethene								Set up	Set up
trans-1,2-Dichloroethene									
cis-1,2-Dichloroethene								Sampled	Sampled
Trichloroethene									
Tetrachloroethene								Pressure head	External
Benzene								at sep-up	pressure
Toluene								cm	bar
Ethylbenzene								-	-
o-Xylene								-	-
m+p-Xylene								-	-
MTBE								-	-
mL sampled (measured)								-	-
mL sampled (by tracer salt)								-	-

K LoD standpipe, reference samples

Compound	Concentration µg/L							
	Sample 1 Date	Sample 2 Date	Sample 3 Date	Sample 4 Date	Sample 5 Date	Sample 6 Date	Sample 7 Date	
	K1	K2	K3	K4	K5	K6	K7	
1,1-Dichloroethene								
trans-1,2-Dichloroethene								
cis-1,2-Dichloroethene								
Trichloroethene								
Tetrachloroethene								
Benzene								
Toluene								
Ethylbenzene								
o-Xylene								
m+p-Xylene								
MTBE								
L Precision direct apr	olication.	samples						
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Compound	Mass on sampler μg						
Date of spiking	ID	ID	ID	ID	ID	ID	ID
Temperature	L1	L2	L3	L4	L5	L6	L7
Chloroethene							
1,1-Dichloroethene							
1,2-Dichloroethenes							
Trichloroethene							
Tetrachloroethene							
Date of rinsing							
mL passed (measured)							
mL passed (by tracer salt)							

M omitted, dilution volume will be insufficient

N Precision standpipe 10%, sam	ples
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Compound	Concentration µg/L			Dates	Temperatures
	ID N1	ID N2	ID N3		
1,1-Dichloroethene				Set up	Set up
trans-1,2-Dichloroethene					
cis-1,2-Dichloroethene				Sampled	Sampled
Trichloroethene					
Tetrachloroethene				Pressure head	External
Benzene				at sep-up	pressure
Toluene				cm	bar
Ethylbenzene				-	-
o-Xylene				-	-
m+p-Xylene				-	-
MTBE				-	-
mL sampled (measured)				-	-
mL sampled (by tracer salt)				-	-

O Precision standpipe 10%, reference samples

Compound	Concentration µg/L						
	Sample 1	Sample 2	Sample 3				
	Date O1	Date O2	Date O3				
1,1-Dichloroethene							
trans-1,2-Dichloroethene							
cis-1,2-Dichloroethene							
Trichloroethene							
Tetrachloroethene							
Benzene							
Toluene							
Ethylbenzene							
o-Xylene							
m+p-Xylene							

Compound	Concentration µg/L	
MTBE		

P Precision standpipe 25%, samples

Compound	Concentration µg/L		Dates	Temperatures	
	ID P1	ID P2	ID P3		
1,1-Dichloroethene				Set up	Set up
trans-1,2-Dichloroethene					
cis-1,2-Dichloroethene				Sampled	Sampled
Trichloroethene					
Tetrachloroethene				Pressure head	External
Benzene				at sep-up	pressure
Toluene				cm	bar
Ethylbenzene				-	-
o-Xylene				-	-
m+p-Xylene				-	-
MTBE				-	-
mL sampled (measured)				-	-
mL sampled (by tracer salt)				-	-

Q Precision standpipe 25%, reference samples

Compound	Concentration µg/L					
	Sample 1	Sample 2	Sample 3			
	Date	Date	Date			
	Q1	Q2	Q3			
1,1-Dichloroethene						
trans-1,2-Dichloroethene						
cis-1,2-Dichloroethene						
Trichloroethene						
Tetrachloroethene						
Benzene						
Toluene						
Ethylbenzene						

Compound	Concentration µg/L			
o-Xylene				
m+p-Xylene				
MTBE				

R Precision standpipe 50%, samples

Compound		Concentration µg/L		Dates	Temperatures
	ID R1	ID R2	ID R3		
1,1-Dichloroethene				Set up	Set up
trans-1,2-Dichloroethene					
cis-1,2-Dichloroethene				Sampled	Sampled
Trichloroethene					
Tetrachloroethene				Pressure head	External
Benzene				at sep-up	pressure
Toluene				cm	bar
Ethylbenzene				-	-
o-Xylene				-	-
m+p-Xylene				-	-
MTBE				-	-
mL sampled (measured)				-	-
mL sampled (by tracer salt)				-	-

S Precision standpipe 50%, reference samples

Compound	Concentration µg/L				
	Sample 1	Sample 2	Sample 3		
	S1	S2	S3		
1,1-Dichloroethene					
trans-1,2-Dichloroethene					
cis-1,2-Dichloroethene					
Trichloroethene					
Tetrachloroethene					
Benzene					

Compound	Concentration µg/L				
Toluene					
Ethylbenzene					
o-Xylene					
m+p-Xylene					
MTBE					

T Precision standpipe 75%, samples

ompound		Concentrat	Dates	Tem-		
						pera-
						tures
	ID	ID	ID			
	T1	T2		T3		
1,1-Dichloroethene					Set up	Set up
trans-1,2-Dichloroethene						
cis-1,2-Dichloroethene					Sampled	Sam-
						pled
Trichloroethene						
Tetrachloroethene					Pressure	Exter-
					head	nal
Benzene					at sep-up	pres-
						sure
Toluene					cm	bar
Ethylbenzene					-	-
o-Xylene					-	-
m+p-Xylene					-	-
MTBE					-	-
mL sampled (measured)					-	-
mL sampled (by tracer salt)					-	-

Compound	Concentration µg/L						
	Sample 1	Sample 2	Sample 3				
	Date	Date	Date				
	U1	U2	U3				
1,1-Dichloroethene							
trans-1,2-Dichloroethene							
cis-1,2-Dichloroethene							
Trichloroethene							
Tetrachloroethene							
Benzene							
Toluene							
Ethylbenzene							
o-Xylene							
m+p-Xylene							
МТВЕ							

U Precision standpipe 75%, reference samples

V Precision standpipe 100%, samples

Compound	Concentration µg/L		Dates	Temperatures	
	ID	ID	ID		
	V1	V2	V3		
1,1-Dichloroethene				Set up	Set up
trans-1,2-Dichloroethene					
cis-1,2-Dichloroethene				Sampled	Sampled
Trichloroethene					
Tetrachloroethene				Pressure head	External
Benzene				at sep-up	pressure
Toluene				cm	bar
Ethylbenzene				-	-
o-Xylene				-	-
m+p-Xylene				-	-
MTBE				-	-
mL sampled (measured)				-	-
mL sampled (by tracer salt)				-	-

Compound	Concentration µg/L						
	Sample 1	Sample 2	Sample 3				
	Date	Date	Date				
	X1	X2	X3				
1,1-Dichloroethene							
trans-1,2-Dichloroethene							
cis-1,2-Dichloroethene							
Trichloroethene							
Tetrachloroethene							
Benzene							
Toluene							
Ethylbenzene							
o-Xylene							
m+p-Xylene							
MTBE							

X Precision standpipe 100%, reference samples

Z omitted, redundant initially.

Well number		C	Concentration µg	/L				
Samples		ID	ID	ID				
Compound	Start	AA1	AA2	AA3				
Chloroethene								
1,1-Dichloroethene								
trans-1,2-Dichloroethene								
cis-1,2-Dichloroethene								
Trichloroethene								
Tetrachloroethene]			
Benzene								
Toluene]			
Ethylbenzene]			
o-Xylene]			
m+p-Xylene]			
MTBE								
L sampled (by tracer salt)								
Date set up						AA Genera	al chemistry	
Date sampled					Parameter	Value	Parameter	Value
Temperature, °C					pН		DOC	mg C/L
Depth water table set up, mbs					Conductivity	mS/m	Iron	mg/L
Depth water table sampling, mbs					Nitrate	mg/L	Ammonium	mg/L
Depth top sampler, mbs					Fluoride	mg/L	Sodium	mg/L
Depth bottom well, mbs					Chloride	mg/L	Potassium	mg/L
Pump yield m ³ /L					Bicarbonate	mg/L	Calcium	mg/L
					Sulphate	mg/L	Magnesium	mg/L

AA Precision field, samples, and groundwater chemistry

AA Precision field, refe	rence samples
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Well number	Concentration µg/L						
	Sample 1 Date	Sample 2 Date	Sample 3 Date	Sample 4 Date			
Compound	AA4	AA5	AA6	AA7			
Chloroethene							
1,1-Dichloroethene							
trans-1,2-Dichloroethene							
cis-1,2-Dichloroethene							
Trichloroethene							
Tetrachloroethene							
Benzene							
Toluene							
Ethylbenzene							
o-Xylene							
m+p-Xylene							
MTBE							

Well number			Concentration µ	g/L				
Samples		ID	ID	ID				
Compound	Start	AB1	AB2	AB3				
Chloroethene								
1,1-Dichloroethene								
trans-1,2-Dichloroethene								
cis-1,2-Dichloroethene								
Trichloroethene								
Tetrachloroethene								
Benzene								
Toluene								
Ethylbenzene								
o-Xylene								
m+p-Xylene								
MTBE								
L sampled (by tracer salt)								
Date set up						AB Genera	al chemistry	
Date sampled					Parameter	Value	Parameter	Value
Temperature, °C					рН		DOC	mg C/L
Depth water table set up, mbs					Conductivity	mS/m	Iron	mg/L
Depth water table sampling, mbs					Nitrate	mg/L	Ammonium	mg/L
Depth top sampler, mbs					Fluoride	mg/L	Sodium	mg/L
Depth bottom well, mbs					Chloride	mg/L	Potassium	mg/L
Pump yield m ³ /L					Bicarbonate	mg/L	Calcium	mg/L
					Sulphate	mg/L	Magnesium	mg/L

AB Precision field, samples, and groundwater chemistry

AB Precision field. ref	erence samples
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Well number	Concentration µg/L						
	Sample 1 Date	Sample 2 Date	Sample 3 Date	Sample 4 Date			
Compound	AB4	AB5	AB6	AB7			
Chloroethene							
1,1-Dichloroethene							
trans-1,2-Dichloroethene							
cis-1,2-Dichloroethene							
Trichloroethene							
Tetrachloroethene							
Benzene							
Toluene							
Ethylbenzene							
o-Xylene							
m+p-Xylene							
MTBE							

Well number			Concentration µ	g/L				
Samples	0 1 1	ID	ID	ID				
Compound	Start	AC1	AC2	AC3	4			
Chloroethene			_		_			
1,1-Dichloroethene								
trans-1,2-Dichloroethene								
cis-1,2-Dichloroethene								
Trichloroethene								
Tetrachloroethene								
Benzene								
Toluene								
Ethylbenzene								
o-Xylene								
m+p-Xylene								
MTBE								
L sampled (by tracer salt)								
Date set up						AC Generation	al chemistry	
Date sampled					Parameter	Value	Parameter	Value
Temperature, °C					рН		DOC	mg C/L
Depth water table set up, mbs					Conductivity	mS/m	Iron	mg/L
Depth water table sampling, mbs					Nitrate	mg/L	Ammonium	mg/L
Depth top sampler, mbs					Fluoride	mg/L	Sodium	mg/L
Depth bottom well, mbs					Chloride	mg/L	Potassium	mg/L
Pump yield m ³ /L					Bicarbonate	mg/L	Calcium	mg/L
					Sulphate	mg/L	Magnesium	mg/L

AC Precision field, samples, and groundwater chemistry

AC FIELISION HEIL, TEIETEILLE Samples	AC	Precision	field,	reference	samples
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Well number	Concentration µg/L						
	Sample 1 Date	Sample 2 Date	Sample 3 Date	Sample 4 Date			
Compound	AC4	AC5	AC6	AC7			
Chloroethene							
1,1-Dichloroethene							
trans-1,2-Dichloroethene							
cis-1,2-Dichloroethene							
Trichloroethene							
Tetrachloroethene							
Benzene							
Toluene							
Ethylbenzene							
o-Xylene							
m+p-Xylene							
MTBE							

Well number			Concentration µ	ıg/L				
Samples		ID	ID	ID				
Compound	Start	AD1	AD2	AD3				
Chloroethene								
1,1-Dichloroethene								
trans-1,2-Dichloroethene								
cis-1,2-Dichloroethene								
Trichloroethene								
Tetrachloroethene								
Benzene								
Toluene								
Ethylbenzene								
o-Xylene								
m+p-Xylene								
MTBE								
L sampled (by tracer salt)								
Date set up						AD Genera	al chemistry	
Date sampled					Parameter	Value	Parameter	Value
Temperature, °C					pН		DOC	mg C/L
Depth water table set up, mbs					Conductivity	mS/m	Iron	mg/L
Depth water table sampling, mbs					Nitrate	mg/L	Ammonium	mg/L
Depth top sampler, mbs					Fluoride	mg/L	Sodium	mg/L
Depth bottom well, mbs					Chloride	mg/L	Potassium	mg/L
Pump yield m ³ /L					Bicarbonate	mg/L	Calcium	mg/L
					Sulphate	mg/L	Magnesium	mg/L

AD Precision field, samples, and groundwater chemistry

AD Precision field, reference samples	•
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Well number	Concentration µg/L							
	Sample 1 Date	Sample 2 Date	Sample 3 Date	Sample 4 Date				
Compound	AD4	AD5	AD6	AD7				
Chloroethene								
1,1-Dichloroethene								
trans-1,2-Dichloroethene								
cis-1,2-Dichloroethene								
Trichloroethene								
Tetrachloroethene								
Benzene								
Toluene								
Ethylbenzene								
o-Xylene								
m+p-Xylene								
MTBE								

Well number			Concentration µo	g/L				
Samples		ID	ID	ID				
Compound	Start	AE1	AE2	AE3				
Chloroethene								
1,1-Dichloroethene								
trans-1,2-Dichloroethene								
cis-1,2-Dichloroethene								
Trichloroethene								
Tetrachloroethene								
Benzene								
Toluene								
Ethylbenzene								
o-Xylene								
m+p-Xylene								
MTBE]			
L sampled (by tracer salt)								
Date set up						AE Genera	al chemistry	
Date sampled					Parameter	Value	Parameter	Value
Temperature, °C					pН		DOC	mg C/L
Depth water table set up, mbs					Conductivity	mS/m	Iron	mg/L
Depth water table sampling, mbs					Nitrate	mg/L	Ammonium	mg/L
Depth top sampler, mbs					Fluoride	mg/L	Sodium	mg/L
Depth bottom well, mbs					Chloride	mg/L	Potassium	mg/L
Pump yield m ³ /L					Bicarbonate	mg/L	Calcium	mg/L
					Sulphate	mg/L	Magnesium	mg/L

AE Precision field, samples, and groundwater chemistry

AE Precision field, reference	samples
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Well number	Concentration µg/L						
	Sample 1 Date	Sample 2 Date	Sample 3 Date	Sample 4 Date			
Compound	AE4	AE5	AE6	AE7			
Chloroethene							
1,1-Dichloroethene							
trans-1,2-Dichloroethene							
cis-1,2-Dichloroethene							
Trichloroethene							
Tetrachloroethene							
Benzene							
Toluene							
Ethylbenzene							
o-Xylene							
m+p-Xylene							
MTBE							

Compound		Concentration µg/L			Temperatures
	ID	ID	ID		
	BA1	BA2	BA3		
1,1-Dichloroethene				Set up	Set up
trans-1,2-Dichloroethene					
cis-1,2-Dichloroethene				Sampled	Sampled
Trichloroethene					
Tetrachloroethene				Pressure head	External
Benzene				at sep-up	pressure
Toluene				cm	bar
Ethylbenzene				-	-
o-Xylene				-	-
m+p-Xylene				-	-
MTBE				-	-
mL sampled (measured)				-	-
mL sampled (by tracer salt)				-	-

BA Reference robustness lab dispenser, samples

BB Reference robustness lab dispenser, reference samples

Compound	Concentration µg/L						
	Sample 1	Sample 2	Sample 3				
	Date	Date	Date				
	BB1	BB2	BB3				
1,1-Dichloroethene							
trans-1,2-Dichloroethene							
cis-1,2-Dichloroethene							
Trichloroethene							
Tetrachloroethene							
Benzene							
Toluene							
Ethylbenzene							
o-Xylene							
m+p-Xylene							
MTBE							

CA Sampling depth robustness standpipe, samples	
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Compound	Concentration µg/L			Dates	Temperatures
	ID ID ID				
	CA1	CA2	CA3		
1,1-Dichloroethene				Set up	Set up
trans-1,2-Dichloroethene					
cis-1,2-Dichloroethene				Sampled	Sampled
Trichloroethene					
Tetrachloroethene				Pressure head	External
Benzene				at sep-up	pressure
Toluene				cm	bar
Ethylbenzene				-	-
o-Xylene				-	-
m+p-Xylene				-	-
MTBE				-	-
mL sampled (measured)				-	-
mL sampled (by tracer salt)				-	-

CB Sampling depth robustness standpipe, reference samples

Compound	Concentration µg/L				
	Sample 1	Sample 2	Sample 3		
	Date	Date	Date		
	CB1	CB2	CB3		
1,1-Dichloroethene					
trans-1,2-Dichloroethene					
cis-1,2-Dichloroethene					
Trichloroethene					
Tetrachloroethene					
Benzene					
Toluene					
Ethylbenzene					
o-Xylene					
m+p-Xylene					
MTBE					

DA lonic strength robustness lab dispenser.	10 mS/cm, samples	
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Compound	C	Concentration µg/L		Dates	Temperatures
	ID	ID	ID		
	DA1	DA2	DA3		
1,1-Dichloroethene				Set up	Set up
trans-1,2-Dichloroethene					
cis-1,2-Dichloroethene				Sampled	Sampled
Trichloroethene					
Tetrachloroethene				Pressure head	External
Benzene				at sep-up	pressure
Toluene				cm	bar
Ethylbenzene				-	-
o-Xylene				-	-
m+p-Xylene				-	-
MTBE				-	-
mL sampled (measured)				-	-
mL sampled (by tracer salt)				-	-

DB lonic strength robustness lab dispenser, 10 mS/cm, reference samples

Compound	Concentration µg/L					
	Sample 1	Sample 2	Sample 3			
	Date	Date	Date			
	DB1	DB2	DB3			
1,1-Dichloroethene						
trans-1,2-Dichloroethene						
cis-1,2-Dichloroethene						
Trichloroethene						
Tetrachloroethene						
Benzene						
Toluene						
Ethylbenzene						
o-Xylene						
m+p-Xylene						
MTBE						

EA lonic strength robustness lab dispenser, 100 mS/cm, samples

Compound	C	Concentration µg/L			Temperatures
	ID	ID	ID		
	EA1	EA2	EA3		
1,1-Dichloroethene				Set up	Set up
trans-1,2-Dichloroethene					
cis-1,2-Dichloroethene				Sampled	Sampled
Trichloroethene					
Tetrachloroethene				Pressure head	External
Benzene				at sep-up	pressure
Toluene				cm	bar
Ethylbenzene				-	-
o-Xylene				-	-
m+p-Xylene				-	-
MTBE				-	-
mL sampled (measured)				-	-
mL sampled (by tracer salt)				-	-

EB Ionic strength robustness lab dispenser, 100 mS/cm, reference samples

Compound	Concentration µg/L					
	Sample 1	Sample 2	Sample 3			
	Date	Date	Date			
	EB1	EB2	EB3			
1,1-Dichloroethene						
trans-1,2-Dichloroethene						
cis-1,2-Dichloroethene						
Trichloroethene						
Tetrachloroethene						
Benzene						
Toluene						
Ethylbenzene						
o-Xylene						
m+p-Xylene						
MTBE						

FA Sampling time robustness	lab dispenser.	3 davs, samples
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Compound		Concentration µg/L		Dates	Temperatures
	ID	ID	ID		
	FA1	FA2	FA3		
1,1-Dichloroethene				Set up	Set up
trans-1,2-Dichloroethene					
cis-1,2-Dichloroethene				Sampled	Sampled
Trichloroethene					
Tetrachloroethene				Pressure head	External
Benzene				at sep-up	pressure
Toluene				cm	bar
Ethylbenzene				-	-
o-Xylene				-	-
m+p-Xylene				-	-
MTBE				-	-
mL sampled (measured)				-	-
mL sampled (by tracer salt)				-	-

FB Ionic strength robustness lab dispenser, 3 days, reference samples

Compound	Concentration µg/L					
	Sample 1	Sample 2	Sample 3			
	Date	Date	Date			
	FB1	FB2	FB3			
1,1-Dichloroethene						
trans-1,2-Dichloroethene						
cis-1,2-Dichloroethene						
Trichloroethene						
Tetrachloroethene						
Benzene						
Toluene						
Ethylbenzene						
o-Xylene						
m+p-Xylene						
MTBE						

GA Sampling time robustness	lab dispenser.	9 davs.	samples
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Compound	Concentration µg/L			Dates	Temperatures
	ID	ID	ID		
	GA1	GA2	GA3		
1,1-Dichloroethene				Set up	Set up
trans-1,2-Dichloroethene					
cis-1,2-Dichloroethene				Sampled	Sampled
Trichloroethene					
Tetrachloroethene				Pressure head	External
Benzene				at sep-up	pressure
Toluene				cm	bar
Ethylbenzene				-	-
o-Xylene				-	-
m+p-Xylene				-	-
MTBE				-	-
mL sampled (measured)				-	-
mL sampled (by tracer salt)				-	-

GB lonic strength robustness lab dispenser, 9 days, reference samples

Compound	Concentration µg/L				
	Sample 1	Sample 2	Sample 3		
	Date	Date	Date		
	GB1	GB2	GB3		
1,1-Dichloroethene					
trans-1,2-Dichloroethene					
cis-1,2-Dichloroethene					
Trichloroethene					
Tetrachloroethene					
Benzene					
Toluene					
Ethylbenzene					
o-Xylene					
m+p-Xylene					
MTBE					

$1 17 \times X X Y H (X Y H (X H (Y H H H (X Y H (Y H ($	HA	Concentration	integration	robustness.	lab dispenser	samples
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Compound		Concentration	Date & time	Temperatures		
	ID					
	HA1	HA2	HA3			
1,1-Dichloroethene				Start 20%	Set up	
trans-1,2-Dichloroethene						
cis-1,2-Dichloroethene				End 20%	Sampled	
Trichloroethene						
Tetrachloroethene				Start 50%	Pressure head	
Benzene					at sep-up	
Toluene				End 50%	cm	
Ethylbenzene					-	
o-Xylene				Start 80%	-	
m+p-Xylene					-	
MTBE				End 80%	-	
mL sampled (measured)					-	
mL sampled (by tracer salt)				-	-	

HB Concentration integration robustness, lab dispenser, reference samples

Compound	Concentration µg/L							
	Sample 1	Sample 2	Sample 3					
	Date	Date	Date					
	HB1	HB2	HB3					
1,1-Dichloroethene								
trans-1,2-Dichloroethene								
cis-1,2-Dichloroethene								
Trichloroethene								
Tetrachloroethene								
Benzene								
Toluene								
Ethylbenzene								
o-Xylene								
m+p-Xylene								
MTBE								

APPENDIX 6

Data management

In general, the data filing and archiving procedures of the DHI Quality Management System will be followed.

All data recording and reporting is done in English, communication with Danish external and internal can be in Danish.

Data storage, transfer and control

The data to be compiled and stored are summarized in Table 8.

Analytical raw data will be filed and archived according to the specifications of the laboratories quality management systems under their ISO 17025 accreditation and are thus not the concern of DHI staff.

Data type	Data media	Data recorder	Data re- cording tim- ing	Data storage
Test plan and report	Protected PDF files	Test responsi- ble, DHI	When ap- proved	Files and ar- chives at DHI
Test details in laboratory and field	Log book and pre-prepared forms	Technician, DHI	During collec- tion	Files and ar- chives at DHI
Calculations	Excel files	Test responsi- ble, DHI	During calcula- tions	Files and ar- chives DHI
Analytical re- ports	Paper	Test responsi- ble, DHI	When received	Files and ar- chives DHI

Table 11 Data compilation and storage summary

Implementation

All <u>e-mail communication</u> is filed in the Outlook Exchange folders, see below structure.

The DHI person receiving an e-mail (to field, not cc field) will file the e-mail. The DHI person sending an e-mail will use the "send and file" option and thereby ensure prompt filing of all e-mails sent. There is generally no need to widespread cc when sending e-mails, unless specific action or communication is required.

All <u>paper communication</u> is immediately filed in the binder established by GHE and available in his office. The title page of the binder will resemble the folder structure at dkstor, see below.

All <u>recordings</u> during testing in the laboratory or in the field are done in water proof writing in hardback log-books with all pages numbered page/total page number. The log books are filed with the staff member using them until the testing is completed, then with GHE and available at his office.

All <u>data</u> needed for the tests are recorded in the data sheets available from Appendix 5 of the Test Plan. The format can be Word tables, Excel worksheets or paper sheets as decided by GHE as test responsible. The outline and format are mandatory and can only be deviated from by recording a deviation with justification, see the Test Plan.

All <u>calculations</u> are done using Excel spreadsheets with names identifying the contents and with headings and notes explaining the calculations.

All <u>electronic files</u> are stored at dkstor in the folder structure shown below. File names are constructed to identify the contents. Subfolders can be established as found convenient, while again constructing folder names that identify the contents. When working away from network connection (offline), copies of files can be used on own PC, but the server version is updated and the offline version deleted immediately after returning to network connection.





APPENDIX 7

Deviations and amendments

Deviation reports

The test plan version approved must be followed. If (or rather when) deviations are needed during testing, the deviations are noted and justified in the format:

Deviation number	Experiment label Test Plan Table 1	Test method step Test Plan Appendix 3	Deviation	Cause	Impact as- sessment	Corrective action, if any	Date	Signature test or field responsible	Date	Signature verification responsible	Date	Signature Battelle AMS QM

The verification protocol version approved must be followed. If deviations are needed during testing, the deviations are noted and justified in the format:

Deviation number	Verification protocol Chapter	Deviation	Cause	Impact as- sessment	Corrective action, if any	Date	Signature verification responsible	Date	Signature internal audi- tor	Date	Signature Battelle AMS QM

Deviation reports are continuously filed in and filed in the appropriate folder at dkstor, see Appendix 6.

Amendment reports

All changes in the protocol and test plan done in advance of verification and testing must be done by the document owner (protocol CHG, plan GHE) and approved by the verification responsible and the internal auditor. Amendments shall be made available for all involved.

The amendments will mostly have the form of a revised section or chapter of the protocol or plan, with the below given front page.

Deviation reports are continuously filed in and filed in the appropriate folder at dkstor, see Appendix 6.

AMENDMENT

TESTING DOCUMENT TITLE AND DATE:

AMENDMENT NUMBER:

DATE OF REVISED PART: ____

PART TO BE CHANGED/REVISED:

CHANGE/REVISION:

Reference to revised part

REASON FOR CHANGE:

ORIGINATED BY:

DHI WMC Verification or Test Responsible

DATE

APPROVED BY:

DHI WMC Internal Auditor

DHI WMC Verification Responsible

DATE

DATE

Battelle AMS Center Quality Manager

Battelle AMS Center Manager

DATE

DATE
