

November 2010

Environmental Technology Verification Report

GAS IMAGING TECHNOLOGY, LLC
SHERLOCK[®] VOC

Prepared by
Battelle

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November 2010

Environmental Technology Verification Report

ETV Advanced Monitoring Systems Center

GAS IMAGING TECHNOLOGY, LLC
SHERLOCK[®] VOC CAMERA

by

Brian Boczek and Amy Dindal, Battelle
John McKernan, U.S. EPA

Notice

The U.S. Environmental Protection Agency, through its Office of Research and Development, funded and managed, or partially funded and collaborated in, the research described herein. It has been subjected to the Agency's peer and administrative review process. Any opinions expressed in this report are those of the author(s) and do not necessarily reflect the views of the Agency, therefore, no official endorsement should be inferred. Any mention of trade names or commercial products does not constitute endorsement or recommendation for use.

Foreword

The EPA is charged by Congress with protecting the nation's air, water, and land resources. Under a mandate of national environmental laws, the Agency strives to formulate and implement actions leading to a compatible balance between human activities and the ability of natural systems to support and nurture life. To meet this mandate, the EPA's Office of Research and Development provides data and science support that can be used to solve environmental problems and to build the scientific knowledge base needed to manage our ecological resources wisely, to understand how pollutants affect our health, and to prevent or reduce environmental risks.

The Environmental Technology Verification (ETV) Program has been established by the EPA to verify the performance characteristics of innovative environmental technology across all media and to report this objective information to permittees, buyers, and users of the technology, thus substantially accelerating the entrance of new environmental technologies into the marketplace. Verification organizations oversee and report verification activities based on testing and quality assurance protocols developed with input from major stakeholders and customer groups associated with the technology area. ETV consists of six environmental technology centers. Information about each of these centers can be found on the Internet at <http://www.epa.gov/etv/>.

Effective verifications of monitoring technologies are needed to assess environmental quality and to supply cost and performance data to select the most appropriate technology for that assessment. Under a cooperative agreement, Battelle has received EPA funding to plan, coordinate, and conduct such verification tests for "Advanced Monitoring Systems for Air, Water, and Soil" and report the results to the community at large. Information concerning this specific environmental technology area can be found on the Internet at <http://www.epa.gov/etv/centers/center1.html>.

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List of Abbreviations

ACC	American Chemistry Council
AMS	Advanced Monitoring Systems
CH ₄	Methane
DQI	Data Quality Indicator
EPA	Environmental Protection Agency
ETV	Environmental Technology Verification
ft	Feet, foot
f/	Focal ratio, F-number
GC	Gas Chromatography
GIT	Gas Imaging Technology, LLC.
g/hr	Grams per hour
IR	Infrared
kg/hr	Kilogram per hour
LCD	Liquid crystal display
LOD	Limit of Detection
Mph	Miles per hour
NRMRL	National Risk Management Research Laboratory
PID	photoionization
ppmv	Parts per million by volume
QA	Quality assurance
QC	Quality control
QMP	Quality Management Plan
Sherlock [®] VOC	Sherlock [®] VOC imaging spectrometer
TCC	Texas Chemical Council
TQAP	Test Quality Assurance Plan
TVA	Toxic Vapor Analyzer
U.S.	United States
VOC	Volatile organic compounds
°F	Degrees Fahrenheit

Chapter 1 Background

The U.S. Environmental Protection Agency (EPA) supports the Environmental Technology Verification (ETV) Program to facilitate the deployment of innovative environmental technologies through performance verification and dissemination of information. The goal of the ETV Program is to further environmental protection by accelerating the acceptance and use of improved and cost-effective technologies. ETV seeks to achieve this goal by providing high-quality, peer-reviewed data on technology performance to those involved in the design, distribution, financing, permitting, purchase, and use of environmental technologies.

ETV works in partnership with recognized testing organizations; with stakeholder groups consisting of buyers, vendor organizations, and permittees; and with the full participation of individual technology developers. The program evaluates the performance of innovative technologies by developing test plans that are responsive to the needs of stakeholders, conducting field or laboratory tests (as appropriate), collecting and analyzing data, and preparing peer-reviewed reports. All evaluations are conducted in accordance with rigorous quality assurance (QA) protocols to ensure that data of known and adequate quality are generated and that the results are defensible. The definition of ETV verification is to establish the performance of a technology under specific, pre-determined criteria or protocols and a strong quality management system. High quality data are assured through the implementation of the ETV Quality Management Plan. ETV does not endorse, certify, or approve technologies.

The EPA's National Risk Management Research Laboratory (NRMRL) and its verification organization partner, Battelle, operate the Advanced Monitoring Systems (AMS) Center under ETV. The AMS Center recently evaluated the performance of the Sherlock[®] VOC imaging spectrometer by Gas Imaging Technology, LLC. (GIT), a portable, passive infrared (IR) imaging spectrometer operating in the spectral range of 3 to 5 micrometers

Chapter 2 Technology Description

This verification report provides results for the verification testing of GIT's Sherlock[®] VOC imaging spectrometer. Following is a description of the GIT Sherlock[®] VOC imaging spectrometer technology (hereafter referred to as the Sherlock[®] VOC), based on information provided by the vendor. The information provided below was not verified in this test. Figure 1 shows the Sherlock[®] VOC imaging spectrometer.

The Sherlock[®] VOC is an infrared optical imaging instrument for video imaging of gas leaks. The Sherlock[®] VOC is man portable and battery operated. The Sherlock[®] VOC is based on patented Image multi spectral Sensor imaging technology.

The Sherlock[®] VOC can be used for IR imaging purposes in many types of industries: oil, gas, chemical, power generation, mining, pulp and paper, to name a few.

The Sherlock[®] VOC has a single focal ratio (F-number or $f/$) of 2.5 in a 75-millimeter (mm) focal length lens that is embedded in the body of the instrument. The horizontal field of view is approximately seven degrees. The Sherlock[®] VOC can be carried by an operator using an optional EasyRig harness which enables pointing and scanning while looking at the liquid crystal display (LCD). This design leaves the operator with both eyes free to watch for safety hazards in the environment of a processing plant.

In addition to displaying the infrared image to the operator on a hooded LCD display the Sherlock[®] VOC can store both 14-bit digital video clips on an embedded frame grabber or to an external 8-bit digital video recorder.



**Figure 1. Sherlock[®] VOC
Camera**

Chapter 3

Test Design and Procedures

3.1 Test Overview

This verification test was conducted according to procedures specified in the *Test/QA Plan for Verification of Leak Detection and Repair Technologies*⁽¹⁾(TQAP) and adhered to the quality system defined in the ETV AMS Center Quality Management Plan (QMP).⁽²⁾ Battelle conducted this verification test with support from British Petroleum (BP), Innovative Environmental Solutions, Inc., The Dow Chemical Company, Sage Environmental Consulting, and Enthalpy Analytical, Inc.

This verification test simulated gas leaks of various chemicals in a controlled laboratory environment. The ability of the Sherlock[®] VOC imaging spectrometer to qualitatively detect gas leaks of select chemicals species by visual images under controlled environmental conditions – including varied stand-off distances, wind speeds, and background materials – was verified and the method detection limits under each test condition were determined. This passive IR camera has not been evaluated under the ETV Program for other compounds or species other than those tested under this verification test. The potential exists for the identification of other species that have an IR absorbance feature(s) in this spectral range under ideal test conditions.

Additionally during laboratory testing, the ability of the Sherlock[®] VOC imaging spectrometer to qualitatively detect the gas leak by visual images relative to a quantitative concentration measurement made by a portable monitoring device acceptable under U.S. EPA Method 21 – *Determination of Volatile Organic Compound (VOC) Leaks*⁽³⁾ for the determination of VOC leaks from process equipment was verified for each chemical at each test condition during laboratory testing. During laboratory testing, “acceptable under U.S. EPA Method 21” meant that the portable monitoring device used met all of the performance requirements of Section 6 in U.S. EPA Method 21 with the exception of those requirements related to a specific leak definition concentration specified in any applicable regulation. A specific leak definition concentration was not used to qualify leaks during laboratory testing in a regulatory sense.

This verification test also verified the ability of the Sherlock[®] VOC imaging spectrometer to detect gas leaks of various chemicals relative to a portable monitoring device acceptable under U.S. EPA Method 21 under “real world” conditions at a chemical plant in Freeport, TX. During field testing, “acceptable under U.S. EPA Method 21” meant that the portable monitoring device used met all of the performance requirements of Section 6 in U.S. EPA Method 21; a specific leak definition concentration of 500 parts per million by volume (ppmv) was utilized. Reference sampling was conducted to determine the mass rate of specific chemical species emitted from each leaking component observed with the Sherlock[®] VOC imaging spectrometer and with the portable monitoring device acceptable under U.S. EPA Method 21.

This verification test of the Sherlock[®] VOC imaging spectrometer was conducted October 20 through October 24, 2008 at the BP research complex in Naperville, Illinois (laboratory testing) and December 1 through December 5, 2008 at the Dow Chemical Company plants (field testing) in Freeport, TX in compliance with the data quality requirements in the AMS Center Quality Management Plan (QMP). The TQAP for this verification test indicated that field testing would be conducted at two field sites. Due to production scheduling issues, a second field site could not be obtained in a timely manner and this verification test was completed using only one field test location. Confirmation from a second field site was obtained during the writing of these reports and field testing occurred outside of this verification test in March 2010. The reader is encouraged to contact either Gas Imaging Technology or the Texas Chemical Council (TCC) to obtain the results of testing completed at the second field site. As indicated in the test/QA plan, the testing conducted satisfied EPA QA Category III requirements. The test/QA plan, the verification statement, and this verification report were reviewed by the following experts.

- Dave Fashimpaur, BP
- Julie Woodward, Dow Chemical
- Jim Griffin, American Chemistry Council
- Christina Wisdom, Texas Chemical Council
- Eben Thoma, U.S. EPA.

One technical expert came to the laboratory testing, and one technical expert came to the field site to observe testing. Verification testing was conducted by appropriately trained personnel following the safety and health guidelines for BP and Dow's facilities.

The Sherlock[®] VOC imaging spectrometer was verified by evaluating the following parameters:

- Method detection limit – The minimum mass leak rate that three separate individuals can observe using the imaging spectrometer under controlled laboratory conditions. This parameter was not evaluated during the field testing phase.
- Detection of chemical gas species relative to a portable monitoring device– The ability of the imaging spectrometer to qualitatively detect a gas leak by visual images relative to a quantitative concentration measurement made by a portable monitoring device acceptable under U.S. EPA Method 21. This parameter was evaluated in both the laboratory and field testing phases.
- Confounding factors effect – Background materials, wind speed, and stand-off distance were carefully controlled during laboratory testing to observe their effects on the method detection limit. During field testing, these variables as well as meteorological conditions were recorded.
- Operational factors – Factors such as ease of use, technology cost, user-friendliness of vendor software, and troubleshooting/downtime were evaluated.

Due to unavailability of a second Sherlock[®] VOC imaging spectrometer during the laboratory and field testing portions of this verification test, inter-unit comparability could not be completed during laboratory and field testing.

Prior to the start of the verification test, personnel from Industrial Scientific Corporation (a vendor licensed to distribute the Sherlock[®] VOC imaging spectrometer) setup the imaging spectrometer according to their recommended configuration for optimal performance.

3.2 Experimental Design

3.2.1 Detection of a Chemical Gas Leak Using the Sherlock[®] VOC Imaging Spectrometer

During both the laboratory testing and field testing, the Sherlock[®] VOC imaging spectrometer was operated by a representative of Industrial Scientific Corporation. This verification test used two additional confirming individuals beyond the camera operator to confirm the observation of a leak in an effort to eliminate potential operator bias. The two additional confirming individuals were the Battelle verification test coordinator and an additional verification test team member. The use of three individuals to observe a chemical leak with the imaging spectrometer is not standard practice when using the imaging spectrometer; typical operation relies on a single camera operator to observe the presence of a chemical gas leak.

The detection of a chemical gas leak in either the laboratory or field setting was determined by the camera operator, as well as, two confirming individuals who reported the results qualitatively as either “detect” or “non-detect” observation. All three individuals must have agreed on the results for the observation to be considered a “detect.” When all three individuals did not agree on a detection, the observation was reported as a “non-detect.” A non-detect was also recorded if the camera operator did not observe a detection (i.e., no confirmation of a non-detect was performed). Each observation was conducted using the viewing screen of the imaging spectrometer.

The TQAP for this verification test required that camera observers have five seconds to identify the origin of the leak or be able to track the plume back to the leaking component when observing chemical gas leaks (i.e., identify the source of the leak). However, during laboratory and field testing the observers were allowed two minutes. This change was made during laboratory testing to account for system hysteresis and upon discovering that several liquid compounds at very low flow rates did not generate a continuous plume. Rather, the leaks were observable as intermittent “puffs” of chemicals emanating from the valve at a frequency on the order of 10 seconds to two minutes. This lag resulted at lower syringe pump feed rate settings and the reduced hot nitrogen carrier gas volume flow rates.

3.2.2 Method Detection Limit

Method detection limits were determined only in the laboratory portion of this verification test. To determine the method detection limit, a known mass leak rate from the packing of a 1-inch valve attached to certified gas cylinders and calibrated flow meters was set at a nominally detectable level either specified by the vendor’s limit of detection (LOD) for a particular test condition, or based on previous literature by Panek et al.⁽⁴⁾ When all three observers identified the leak, the leak rate was reduced by the testing staff using calibrated flow meters. Once a leak rate that was not identifiable by all three people was reached, the mass emission rate was again increased using the calibrated flow meters to the level where all three could again identify the leak using the passive infrared imager. This rate was the method detection limit for the Sherlock[®] VOC imaging spectrometer under the tested conditions. This process was completed for every testing trial identified in Section 3.2.3. Table 1 identifies the type of chemical leaks evaluated with the Sherlock[®] VOC imaging spectrometer during laboratory testing.

The TQAP for this verification test stated that 1,2-dichloropropane (i.e., propylene dichloride) and hydrochloric acid also would be used during laboratory testing. The stock solution of propylene dichloride was suspected by laboratory personnel of having been cross-contaminated by a different chemical compound. A second stock solution of propylene dichloride could not be obtained from a chemical vendor before the conclusion of laboratory testing. Thus, propylene dichloride was not used during laboratory testing. The laboratory staff also expressed concerns of causing damage to the delivery syringe in the chemical delivery system with the use of hydrochloric acid. Because hydrochloric acid could not be delivered through the chemical delivery system without causing damage to the system, a known leak rate could not be generated during laboratory analysis, therefore hydrochloric acid was not evaluated.

Table 1. Chemical Leaks Evaluated with the GIT Sherlock® VOC Imaging Spectrometer During Laboratory Testing

Chemical	Chemical Group
1,3-butadiene	Olefin
Acetic acid	Acetate
Acrylic acid	Acid
Benzene	Aromatic
Dichloromethane (methylene chloride)	Chlorinated
Ethylene	Olefin
Methane	Alkane
Methanol	Alcohol
Pentane	Alkane
Propane	Alkane
Styrene	Aromatic

3.2.3 *Confounding Factors*

Because passive IR imagers rely on the physical characteristics of the environment and the molecules being imaged to create an image viewed by the operator (via temperature/emissivity differences between naturally occurring ambient IR radiation and the thermal emission or absorption of the leaking gas), environmental characteristics may confound the measurement. For example, if there is not sufficient thermal emission or absorption by the leaking gas, the passive IR imager may not be able to detect a leak against an ambient thermal background.

During laboratory testing, experimental factors of background materials, wind speed, and stand-off distance were altered for each chemical tested. These experimental factors were chosen, because the performance of passive imagers is dependent on physical characteristics of the leak, atmospheric conditions, and background materials. The change of background material demonstrates the ability of the Sherlock® VOC imaging spectrometer to passively detect the leak with a background scene similar to petrochemical process piping and vessels (curved metal gas cylinders) and with a background that is different than the leaking component and more uniform in nature (cement board – representing control buildings, sidewalks, and other uniform flat background surfaces). The wind speed variations and the stand-off distances inform on the atmospheric and optical pathway effects on the method detection limit, and in turn on real-world limitations. Table 2 presents the specific test conditions evaluated during laboratory testing.

It was originally intended that all test conditions would be completed for all chemicals; however, it was not possible for 1,3-butadiene, acrylic acid, methylene chloride, methane, and styrene for the following reasons.

Previous testing of the Sherlock® VOC imaging spectrometer using methane had been completed by the laboratory facility outside of the verification test. Consequently, methane was used during test equipment setup to confirm that the equipment produced method detection limits for methane that were consistent with those produced during previous testing by the laboratory.

Table 2. Test Conditions Evaluated During Laboratory Testing

Chemical Species	Laboratory Test Conditions											
	10 ft stand-off distance; 0-mph wind speed; cement board background	10 ft stand-off distance; 0-mph wind speed; curved metal gas cylinder background	10 ft stand-off distance; 2.5-mph wind speed; cement board background	10 ft stand-off distance; 2.5-mph wind speed; curved metal gas cylinder background	10 ft stand-off distance; 5-mph wind speed; cement board background	10 ft stand-off distance; 5-mph wind speed; curved metal gas cylinder background	30 ft stand-off distance; 0-mph wind speed; cement board background	30 ft stand-off distance; 0-mph wind speed; curved metal gas cylinder background	30 ft stand-off distance; 2.5-mph wind speed; cement board background	30 ft stand-off distance; 2.5-mph wind speed; curved metal gas cylinder background	30 ft stand-off distance; 5-mph wind speed; cement board background	30 ft stand-off distance; 5-mph wind speed; curved metal gas cylinder background
1,3-butadiene	✓	✓					✓	✓				
Acetic acid	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓
Acrylic acid	✓	✓					✓	✓	✓			
Benzene	✓	✓	✓	✓			✓	✓	✓	✓	✓	✓
Methylene chloride	✓	✓	✓	✓			✓	✓	✓			
Ethylene	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓
Methane	✓	✓	✓	✓				✓	✓			
Methanol	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓
Pentane	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓
Propane	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓
Styrene	✓	✓					✓	✓				

The 2.5 and 5-mph wind speed test conditions were not completed for acrylic acid. After completion of the 0-mph wind speed test condition, laboratory personnel indicated that the acrylic acid was dissolving the rubber plunger gasket in the liquid delivery syringe in the vapor generator system. Laboratory personnel indicated that the rubber seemed to be “dissolving” inside the syringe and the syringe was no longer providing a steady flow of acrylic acid into the chemical delivery system. Additional testing using this compound was abandoned due to safety and chemical handling concerns.

The 2.5 and 5-mph wind speed test conditions were not completed for 1,3-butadiene and styrene due to safety and potential exposure concerns. During laboratory setup the week prior to verification testing, the exhaust of the test apparatus, which feeds into the general laboratory building exhaust, was balanced and smoke tested to ensure that compounds leaking from the system were captured in either the vertical hood canopy mounted over the leaking component or the downwind hood mounted adjacent to the test system. Unbeknownst to laboratory personnel, the building general exhaust system was operating at a lower setting during air balancing and smoke testing due to decreased occupancy in the building. During the week of the test, the general building exhaust was increased due to the presence of the test compounds entering the exhaust system. The change in building exhaust flows caused the capture of the chemical compound by the overhead hood and the hood mounted next to the test system to decrease. Neither the vendor nor laboratory personnel had documentation of respirator fit-testing, so respirators could not be used during testing.

To address this problem, the leaking valve was placed next to the side hood during wind speed testing and testing of those chemical compounds which are liquids at standard conditions commenced in order of increasing boiling point. Upon completion of wind speed testing for acetic acid, the laboratory had a slight odor of acetic acid. This indicated to laboratory personnel that locating the leaking valve next to the side hood during wind speed testing did not adequately capture all of the chemical compounds exhausting from the test system. At this point, wind speed testing of 1,3-butadiene and styrene was abandoned because these compounds have higher chemical toxicity and exposure by the verification test team, vendor, and laboratory staff to these compounds would have occurred during wind speed testing.

During benzene, methylene chloride, and ethylene testing, several of the wind speed tests and background tests were not conducted because the method detection limit for lower wind speed (or background) conditions exceeded the highest reliable flow rate capable of being provided by the chemical leak delivery system at test conditions which were expected to produce a lower method detection limit (refer to Section 6.3 for discussion of the observed influence of confounding factors). For example, a 5-mph wind speed test was not conducted at a 10 ft stand-off distance with a cement board background with benzene because the method detection limit exceeded the highest reliable flow rate of the chemical delivery system for the 10 ft stand-off distance, cement board background, and 2.5-mph wind speed.

3.2.4 Detection of a Chemical Gas Species Relative to a Portable Monitoring Device

The detection of a single chemical gas leak in either the laboratory or field environments was determined by the operator as well as two confirming individuals as previously described in Section 3.2.1 and reported qualitatively as either “detect” or “non-detect.”

During laboratory testing a portable monitoring device acceptable under U.S. EPA Method 21 sampled the leak after the method detection limit was determined for the specified test conditions. During laboratory testing, “acceptable under U.S. EPA Method 21” meant that the portable monitoring device used met all of the performance requirements of Section 6 in U.S. EPA Method 21 with the exception of those requirements related to a specific leak definition concentration specified in any applicable regulation. A specific leak definition concentration was not used to qualify leaks during laboratory testing in a regulatory sense. The portable monitoring device used during laboratory testing was an Industrial Scientific IBRID MX6 with photoionization (PID) sensor and SP6 motorized sampling pump which was supplied calibrated from the instrument supplier; no additional calibrations were performed during laboratory

testing. During field testing a portable monitoring device acceptable under U.S. EPA Method 21 was used to screen each leaking component as part of the bagging reference method used. During field testing, “acceptable under U.S. EPA Method 21” meant that the portable monitoring device used met all of the performance requirements of Section 6 in U.S. EPA Method 21; a specific leak concentration of 500 ppmv was utilized. The portable monitoring device used during field testing was a Thermo-Environmental Toxic Vapor Analyzer (TVA).

3.2.5 Field Testing Procedures

Field testing was conducted to allow for performance evaluation under “real world” conditions. Chemicals that were tested in the laboratory were targeted for evaluation at the field sites. The flow rates of field leaks were quantitatively determined by a reference method called *EPA Protocol for Equipment Leak Emission Estimates*⁽⁵⁾, which is also referred to as the “bagging method.” Specific details and procedures for this reference method are provided in the TQAP for this verification test. This method involves completely enclosing the leak with non-permeable material, collecting the leak with ambient air entering the bag, and performing mass measurement of the bagged leak by an analytical method. Only those leaks above the field test-assigned 500 ppmv leak definition concentration, as measured by the Thermo-Environmental TVA, were observed with the passive infrared imagers and collected as reference samples under this verification test.

The verification test team moved through the plant screening for possible leaking components using a Thermo-Environmental TVA, a portable monitoring device acceptable under U.S. EPA Method 21. Once a leak was detected with the portable monitoring device, leak characteristics and environmental factors (i.e., type of component, background material, data and time, temperature, etc.) were recorded qualitatively. Meteorological data was retrieved from the nearest meteorological data station, the Dow Chemical Company’s on-site weather station. As space permitted, the camera operator took readings at three stand-off distances (10, 30, and greater than 30 ft if possible). Every reading was verified by an additional two confirming individuals and recorded as either “detect” or “non-detect” as specified in Section 3.2.1. Once all cameras had scanned the leak, the bagging team members (Sage Environmental Consulting) commenced collecting duplicate reference samples of the leak into evacuated SUMMA canisters. Reference sampling concluded with a final screening by the portable monitoring device acceptable under U.S. EPA Method 21 to verify that the leak concentration had not changed from the beginning to the end of testing the component. Only those leaks which showed less than a 20% difference between the pre- and post-screening with the portable monitoring device were considered consistent enough to report in the results without a data qualifier. The concentration of the collected reference samples was determined according to the analytical method in U.S. EPA Method 18 – *Measurement of Gaseous Organic Compound Emissions by Gas Chromatography*.⁽⁶⁾ Upon conclusion of the five days of field testing, all reference samples were shipped to Enthalpy Analytical, Inc. for U.S. EPA Method 18 analysis.

3.3 Qualitative Evaluation Parameters

Operational factors such as maintenance needs, ease of use, data output, and software requirements were documented based on observations by Battelle.

Chapter 4

Quality Assurance/Quality Control

QA/quality control (QC) procedures were performed in accordance with the QMP for the AMS Center and the TQAP for this verification test. As noted throughout Chapter 3, there were deviations from the TQAP, but the work was performed as described in the previous sections. None of the deviations from the test/QA plan resulted in any adverse impacts on the quality of the data produced by this verification test. QA/QC procedures and results are described in the following subchapters.

4.1 Reference Method Quality Control

Laboratory testing did not use a specified reference method for determining the leak rate of the test conditions. Rather, certified gas cylinders and laboratory grade liquid compounds were used with calibrated flow meters and a calibrated syringe pump to generate a known leak rate in terms of mass per unit time from the leaking valve. As a laboratory QC measure, laboratory personnel, randomly and without the knowledge of the camera operator or the additional confirming individuals, increased or decreased the mass leak rate to reduce the opportunity to predetermine an outcome. In addition, laboratory blanks (i.e., pure nitrogen gas) and replicate tests were used to reduce uncertainties and verify method detection limits established in prior tests.

The field testing portion of this verification test used accepted methods to generate reference samples. Reference samples were collected using *EPA Protocol for Equipment Leak Emission Estimates* and the concentrations of compounds in the collected reference samples were determined according to the analytical method in U.S. EPA Method 18 *Measurement of Gaseous Organic Compound Emissions by Gas Chromatography*.

The quality of the reference measurements collected during field testing was assured by adherence to the requirements of the data quality indicators (DQIs) and criteria for the reference collection and analytical method critical measurements, including requirements to perform initial calibrations and calibration checks of the portable monitoring device acceptable under U.S. EPA Method 21, confirming the leak rates changed less than 20% before and after bagging, assessing the bias and accuracy of the bagging procedure, and assessing the bias and accuracy of the gas chromatography (GC) laboratory analysis by developing calibration curves traceable to certified gas standards, and performing positive and negative control checks. The following sections present key data quality results from these methods.

4.1.1 Bias and Accuracy of Sample Screening Measurements Using Portable Monitoring Device

A DQI is established in the TQAP for this verification test for the bias and accuracy of sample screening measurements using a portable monitoring device. This DQI is assessed by performing calibrations of the Thermo-Environmental TVA used to screen leaking components during the field portion of the verification test and analyzing calibration check samples. During laboratory testing the portable monitoring device was an Industrial Scientific IBRID MX6 with PID sensor and SP6 motorized sampling pump which was supplied calibrated from the instrument supplier; per the TQAP for this verification test, no additional calibrations were performed during laboratory testing.

Calibration of the TVA was conducted using various levels of certified methane (CH₄)-in-air gas standards. The TQAP for this verification test required the use of five calibration points (an un-spiked gas standard plus four additional concentrations); however, only three additional gas standard concentrations were obtained. Because component leaks were only bagged as reference samples if their concentration was greater than 500 ppmv and because the calibration response of the TVA was evaluated using an un-spiked gas standard (0 ppmv) and three additional concentrations of gas standards (500, 1000, and 9600 ppmv) thereby bounding the 500 ppmv reference sample bagging threshold, there was no effect on data quality.

The calibration response of the TVA was analyzed at the start and end of each verification test day or if the overall TVA sensitivity changed by greater than 10% (based on the calibration check data, which are presented in Table 5). The minimum acceptance criterion for this reference method DQI was that the TVA calibration response must agree within 10% of the concentration of each gas standard. Table 3 presents the results of all TVA calibration responses collected during this verification test. Inspection of the data present in Table 3 shows that all calibration response measurements were confirmed to be within 10% of the calibration gas standard concentration.

The TQAP for this verification test required that a calibration check sample be analyzed using one concentration of the calibration gas standards at a minimum frequency of 5% of all bagged reference samples collected. Sixteen calibration check samples were analyzed with the TVA during the course of field testing and nine duplicate reference samples were collected resulting in a calibration check sample frequency of 178% of all bagged reference samples collected (i.e., 16 calibration check samples completed during the collection of nine duplicate reference samples). These checks were performed more frequently to ensure no drifting of the instrument occurred during downtimes to ensure optimum performance. The minimum acceptance criterion specified in the TQAP for this verification test is that the check standard must be within less than or equal to a 10% change in response from the previous calibration of the TVA. If the calibration check sample showed a change in response greater than 10%, then recalibration of the TVA was performed and any affected reference sample components collected would be rescreened. During this verification test, calibration check samples were performed using a certified 500 ppmv CH₄-in-air gas standard. Table 4 presents the results of all calibration check standards performed during verification testing. Inspection of the data presented in Table 4 indicate that reference samples 08A and 08B should have been rescreened after recalibration of the TVA and, therefore, are considered suspect data and reported with a data qualifier.

Table 3. TVA Calibration Responses

Date [Time]	Calibration Gas Standard Concentration (ppmv CH ₄)			
	0	500	1000	9600
	TVA Output Concentration (ppmv CH ₄) ^(b)	TVA Calibration Response (as % Error) ^(c)		
12/1/2008 [13:33] ^(a)	0.70	0.40	-1.3	-0.80
12/2/2008 [09:01]	0.40	-0.80	-0.10	-0.60
12/2/2008 [14:08]	1.0	1.2	11	2.1
12/2/2008 [16:05]	1.0	5.6	4.2	4.2
12/3/2008 [08:41]	0.80	-1.4	ND	-0.70
12/3/2008 [09:30]	0.70	-0.60	-4.4	-4.9
12/3/2008 [10:12]	0.80	-1.2	-0.60	0.10
12/3/2008 [17:06]	0.60	-7.2	-8.2	-8.0
12/4/2008 [10:04]	0.60	-0.60	-0.30	-1.0
12/4/2008 [13:20]	ND	ND	-0.10	-0.30
12/4/2008 [16:12]	0.60	-0.80	-1.5	-1.0
12/4/2008 [17:23]	0.20	-1.4	-1.7	-1.1
12/5/2008 [08:59]	0.60	ND	-0.70	-0.70
12/5/2008 [11:20]	1.2	4.0	3.0	-8.3
12/5/2008 [14:01]	0.20	3.4	3.3	-3.1

^(a) An end-of-day TVA response was not collected on 12/1/2008. Data for leak location 1 is included but flagged because there are acceptable reference and bagging measurements.

^(b) Concentration data presented for un-spiked gas standard, since % error calculation is not possible. This point is used in calibrating the Thermo-Environmental TVA.

^(c) Percent error is calculated as [(TVA calibration response, ppmv CH₄ – Calibration Gas Standard Concentration, ppmv CH₄)/ Calibration Gas Standard Concentration, ppmv CH₄] x 100%.

ND - Not detected

4.1.2 Confirmation of Detected Leaks

A DQI is established in the TQAP for this verification test for the confirmation of detected leaks. This DQI is assessed by analyzing the concentration of a leaking component before and after bagging the component. These measurements were completed for all leaking components which were bagged and collected as reference samples. The acceptance criterion for this DQI is that the pre and post screening measurements collected with the TVA agree within 20%. Table 5 presents the results of all pre- and post-bagging measurements completed during the collection of reference samples.

4.1.3 Bias and Accuracy of Enclosure Equilibration Gas

A DQI is established in the TQAP for this verification test for bias and accuracy of the enclosure equilibration gas. This DQI requires that if the blow-through bagging procedure is used to collect reference samples, then the equilibration gas in the bag is collected and analyzed for contamination prior to collection of reference samples. During the verification testing, reference samples were collected using the vacuum-method which does not require the use of an equilibration gas; therefore, this DQI was not applicable.

Table 4. TVA Calibration Check Samples

Date [Time]	Calibration Check		Comments
	Response (as % Error) ^(a)		
12/2/2008 [11:17]	0.40		
12/2/2008 [12:15]	-5.2		
12/2/2008 [14:05]	-16		Recalibration only. No rescreening necessary because no reference samples had been collected between this calibration check sample and TVA calibration.
12/2/2008 [14:08]	1.2		
12/2/2008 [15:10]	1.4		
12/2/2008 [15:43]	2.0		
12/3/2008 [9:23]	64		Found leak; recalibrated only. No rescreening necessary because reference samples had yet to be collected this day.
12/3/2008 [10:30]	0.80		
12/3/2008 [11:32]	-0.60		
12/3/2008 [13:57]	0.60		
12/3/2008 [15:45]	0.60		
12/4/2008 [11:43]	1.6		
12/4/2008 [13:23]	-17		Recalibration only. No rescreening necessary because no reference samples had been collected between this calibration check sample and the previous check.
12/4/2008 [15:30]	24		Recalibration only. Reference samples 08A and 08B were inadvertently not rescreened and are therefore considered suspect and results reported with qualifier.
12/4/2008 [17:25]	-1.4		
12/5/2008 [10:38]	-3.0		

(a) Percent (%) error is calculated as [(TVA calibration check response, ppmv CH₄ – Calibration Gas Standard Concentration, 500 ppmv CH₄)/ Calibration Gas Standard Concentration, 500 ppmv CH₄] x 100%.

Table 5. Confirmation of Detected Leaks by TVA

Reference Sample Numbers	Concentration Measured by TVA (ppmv CH ₄)			Relative % Difference ^(b)	Comments
	Pre-bagging	Post-bagging			
01C, 01D	>100,000 ^(a)	>100,000 ^(a)		0%	
02A, 02B	20,500	20,500		0%	
03A, 03B	>100,000 ^(a)	>100,000 ^(a)		0%	
05A, 05B	>100,000 ^(a)	>100,000 ^(a)		0%	
06A, 06B	18,000	23,000		24%	Data is considered suspect and results reported with qualifier.
07A, 07B	18,000	17,000		5.7%	
08A, 08B	8,000	8,000		0%	
09A, 09B	800	870		8.4%	
10A, 10B	>100,000 ^(a)	>100,000 ^(a)		0%	

(a) The concentration of the leak at the component was high enough to cause the TVA to flameout. Concentration estimated as greater than 100,000 ppmv CH₄.

(b) Relative percent (%) difference calculated using the following calculation:

$$\text{Relative \% difference} = \frac{2 \times |\text{Pre - bagging concentration} - \text{Post bagging concentration}|}{\text{Pre - bagging concentration} + \text{Post bagging concentration}} \times 100\%$$

4.1.4 Bias and Accuracy of Bagging Procedure

A DQI is established in the TQAP for this verification test for the bias and accuracy of the bagging procedure. This DQI is assessed by bagging an artificial leak at a known rate in the middle of the analytical calibration curve. The procedure followed is that specified in U.S. EPA *Protocol for Equipment Leak Emission Estimates* using certified CH₄-in-air gas standards and calibrated flow meters. This DQI indicator was assessed at the beginning and end of the week of field sampling. An acceptance criterion of 80 to 120% recovery is required for the bagging equipment to pass the known leak rate test. Table 6 presents the results of the known leak rate test. As shown in Table 6, this DQI was met before and after reference sampling.

Table 6. Known Leak Rate Test Results

Date [Time]	Leak Rate Level	Emission Rate (kilogram per hour [kg/hr] CH ₄)		% Recovery ^(a)
		Theoretical	Measured	
Pre-Test				
11/28/2008 [12:45]	Low	4.31 x 10 ⁻⁴	4.23 x 10 ⁻⁴	98.1%
11/28/2008 [12:20]	High	1.75 x 10 ⁻³	1.60 x 10 ⁻³	91.4%
Post-Test				
12/5/2008 [14:35]	Low	1.25 x 10 ⁻³	1.32 x 10 ⁻³	106%
12/5/2008 [14:43]	High	2.43 x 10 ⁻³	2.50 x 10 ⁻³	103%

(a) Percent Recovery is calculated as (measured emission rate, kg/hr CH₄) / (theoretical emission rate, kg/hr CH₄) x 100%

4.1.5 Bias and Accuracy of Gas Chromatography Analytical Method

A DQI is established in the TQAP for this verification test for the bias and accuracy of the GC analytical method used to quantify the concentration of leaks collected during reference sampling. This DQI was assessed through initial calibration, and by performing positive and negative control samples. These assessments are discussed in the following paragraphs.

Initial Calibration. Initial calibration of the GC was conducted by using various levels of certified calibration gases starting with an un-spiked gas standard and then a minimum of four additional concentrations of gas standards. The TQAP for this verification test required that the initial calibration be performed at the start and end of every analytical sequence or if overall instrument sensitivity changed by greater than 10%. To ensure accuracy of the initial calibration, the instrument must be calibrated using certified gas standards. The minimum acceptance criteria specified for this assessment is that all gas standards must be within 2% of their certified value.

The analytical laboratory that performed the GC analytical method (Enthalpy Analytical, Inc.) purchased gas standards with certification accuracies of ± 2%, as specified by the gas supplier. In addition, the GC analytical laboratory produced diluted gas standards from these purchased standards using a gas dilution system compliant with U.S. EPA Method 205⁽⁷⁾ which specifies gas dilution systems must produce calibration gases whose measured values are within ± 2% of the predicted levels from a certified gas standard.

Positive Control Checks. The TQAP for this verification test required that positive control checks be performed at a minimum frequency of 10% of all samples tested using one concentration of calibration gas standard. The minimum acceptance criteria for positive control checks is that the positive control check response is less than or equal to a 10% change in response from the initial calibration after adjustment of the overall instrument sensitivity. Forty sample measurements were conducted by the GC analytical laboratory using triplicate injections and 19 positive control checks were performed exceeding the minimum frequency of 10% of samples tested. The results of the positive control checks are provided in Table 7. As demonstrated by Table 7, all positive control checks met this acceptance criterion.

Negative Control Checks. The TQAP for this verification test required that negative control checks be performed at a minimum frequency of one out of every 10 samples tested. The minimum acceptance criterion for this assessment is that all negative control responses must remain lower than the lowest calibration standard for the chemical analyzed. Forty sample measurements were conducted by the GC analytical laboratory using triplicate injections and four negative control checks were performed meeting the minimum frequency of one negative control check per 10 samples analyzed. All negative control checks performed were non-detect for the compounds analyzed indicating an analytical result below the method detection limit for the compound. The method detection limit for methane, ethylene, styrene, benzene, 1,3-butadiene, methylene chloride, and propylene dichloride was 1.00 ppmv for each compound.

4.2 Audits

Two types of audits were performed during the verification test, a technical systems audit (TSA) of the verification test procedures, and a data quality audit. Because of the nature of bagging reference method, a performance evaluation audit, as is usually performed to confirm the accuracy of the reference method, was not applicable for this verification test. Audit procedures for the TSA and the data quality audit are described further below.

4.2.1 Technical Systems Audit

The Battelle AMS Center Quality Manager performed a TSA during the both the laboratory and field testing portions of this verification test to ensure that the verification test was performed in accordance with the QMP for the AMS Center and the test/QA plan.

The TSA of the laboratory portion of the verification test was performed on October 22, 2008. During this TSA, the Battelle AMS Center Quality Manager observed the test procedures used to determine method detection limits and the response of the Industrial Scientific IBRID MX6 with PID sensor and SP6 motorized sampling pump at the each method detection limit. These procedures were observed during some of the testing conducted with acrylic acid, benzene, methylene chloride, and styrene.

The TSA of the field testing portion of the verification test was performed on December 3, 2008. During this TSA, the Battelle AMS Center Quality Manager observed the procedures of the bagging reference method, including the confirmation of the detected leaks by means of pre- and post-bagging screening of the leaking component with the Thermo-Environmental TVA, construction of the bagging enclosure, and duplicate reference sample collection, as well as the audited the observations of the leak component with camera. In addition, the Battelle AMS

Center Quality Manager observed both the performance of a calibration drift check and recalibration as well as an end-of-day calibration response check of the Thermo-Environmental TVA.

Table 7. Summary of Positive Control Check Responses

Positive Control Check Sample ID	Compounds Measured by GC Method	Expected Response (Picoampere Second)	Actual Response (Picoampere Second)	Percent Error^(a)
GC100pg167 #2	Benzene	39.8	39.3	-1.1%
GC100pg167 #2	Benzene	39.8	39.0	-1.9%
GC100pfl69F #4	Ethylene	13.7	13.8	+0.39%
	1,3-butadiene	27.3	26.9	-1.6%
GC100pfl69F #4	Ethylene	13.7	13.7	-0.61%
	1,3-butadiene	27.3	26.7	-2.4%
GC100pfl69F #4	Ethylene	13.7	13.5	-1.6%
	1,3-butadiene	27.3	26.3	-3.9%
GC100pfl69F #4	Ethylene	13.7	13.4	-2.4%
	1,3-butadiene	27.3	25.7	-5.8%
GC100pfl69F #4	Ethylene	13.7	13.7	-0.44%
	1,3-butadiene	27.3	26.9	-1.5%
GC100pfl69F #4	Ethylene	13.7	13.8	+0.39%
	1,3-butadiene	27.3	27.2	-0.43%
GC102pg44 #3	Methane	22.4	22.8	+1.6%
GC102pg44 #3	Methane	22.4	22.7	+1.3%
GC100pg169 #2	Methane	7.10	6.95	-2.1%
GC100pg169 #2	Methane	7.10	6.73	-5.3%
GC100pg169 #3	Methane	15.7	15.3	-3.3%
GC100pg169 #4R	Methane	15.9	15.5	-2.5%
GC100pg169 #4R	Methane	15.9	15.8	-0.39%
GC102pg52 #4	Pentane	122	127	+4.2%
	Methylene chloride	17.6	17.7	+0.60%
	Benzene	148	150	+1.1%
	Propylene dichloride	36.1	35.4	-2.1%
	Styrene	31.9	34.0	+6.7%
GC102pg52 #4	Pentane	122	125	+2.7%
	Methylene chloride	17.6	17.3	-1.9%
	Benzene	148	147	-0.75%
	Propylene dichloride	36.1	34.4	-4.6%
	Styrene	31.9	32.7	+2.4%
GC102pg52 #4	Pentane	67.7	67.5	-0.35%
	Methylene chloride	10.2	9.86	-3.4%
	Benzene	82.0	79.5	-3.1%
	Propylene dichloride	21.1	20.5	-2.9%
	Styrene	17.8	18.4	+3.8%
GC102pg52 #4	Pentane	67.7	70.3	+3.7%
	Methylene chloride	10.2	10.2	+0.16%
	Benzene	82.0	82.3	+0.35%
	Propylene dichloride	21.1	21.2	+0.49%
	Styrene	17.8	18.6	+4.5%

(a) Percent error is calculated as [(Actual Peak Response, peak area – Expected Response, peak area)/ Actual Peak Response, peak area] x 100%.

The TSA of both the laboratory and field testing portions resulted in one finding and one observation. The finding identified that only one field test (at a chemical plant) has been

conducted as part of this verification test as opposed to the two field sites (one a chemical plant and the other a petrochemical plant) identified in the TQAP for this verification test. The observation noted documentation errors and improvements to the manner in which data were recorded were discussed on-site with the verification test leader; immediate changes based on the discussed improvements were implemented.

A TSA report was prepared, and a copy was distributed to the EPA AMS Center Quality Manager.

4.2.2 Data Quality Audit

Records generated in the verification test received a one-over-one review before these records were used to calculate, evaluate, or report verification results. Data were reviewed by a Battelle technical staff member involved in the verification test. The person performing the review added his/her initials and the date to a hard copy of the record being reviewed.

100% of the verification test data was reviewed for quality by the Verification Test Coordinator, and at least 10% of the data acquired during the verification test were audited. The data were traced from the initial acquisition, through reduction and statistical analysis, to final reporting to ensure the integrity of the reported results. All calculations performed on the data undergoing the audit were checked.

The data quality audit resulted in four findings (on three separate topics) that were addressed related to the documentation of the number of confirming individuals at the method detection limits in the laboratory phase raw data, exclusion from the verification report of concentration measurements made by the PID sensor for dichloromethane (methylene chloride), methanol, and propane during the laboratory phase of this verification test, and data transcription errors.

A data audit report was prepared, and a copy was distributed to the EPA AMS Center Quality Manager.

Chapter 5 Statistical Methods

The statistical methods used to evaluate the quantitative performance factors listed in Section 3.2 are presented in this chapter. Qualitative observations were also used to evaluate verification test data.

5.1 Method Detection Limit

The method detection limit was assessed using the procedures described in Section 3.2.2 and the TQAP for this verification test. The overall detection limit variation was calculated as the standard deviation of the method detection limits determined under all the conditions tested for each chemical of interest. The equation for standard deviation is as follows:

$$S_x = \left[\frac{1}{n-1} \sum_{k=1}^n (C_k - \bar{C})^2 \right]^{0.5} \quad (1)$$

where S_x is the standard deviation of all method detection limits determined for chemical x , n is the number of replicate samples, C_k is the leak rate measured for the k th sample, and \bar{C} is the average leak rate of the replicate samples. If the sample sizes were small ($n < 10$), standard deviations provide a biased estimate of variability. Therefore the range is provided when there were fewer than 10 samples collected.

5.2 Percent Agreement

Percent agreement was used to assess the agreement between the Sherlock[®] VOC imaging spectrometer and the monitoring device acceptable under U.S. EPA Method 21 in the laboratory for each compound tested. The inverse of the percent agreement is the percentage of the results that the technology would detect a leak when U.S. EPA Method 21 would not. The equation for percent agreement is as follows:

$$\text{Percent Agreement} = \frac{A}{T} \times 100\%$$

where A the number of tests that both units agree and T is the total number of tests. To determine if both the monitoring device acceptable under U.S. EPA Method 21 and the Sherlock[®] VOC imaging spectrometer agreed, the method detection limits at each test condition were first reviewed. If the method detection limit of the Sherlock[®] VOC imaging spectrometer was below

the highest reliable flow rate of the chemical delivery system (reported as \leq), then the Sherlock[®] VOC imaging spectrometer was noted as being able to detect the chemical gas leak under those specified test conditions. Similarly, if the method detection limit of the Sherlock[®] VOC imaging spectrometer was above the highest reliable flow rate of the chemical delivery system (reported as \geq), then the Sherlock[®] VOC imaging spectrometer was noted as not being able to detect the chemical gas leak under those specified test conditions.

Next, the response of the monitoring device acceptable under U.S. EPA Method 21 was reviewed for the same test conditions. If the monitoring device acceptable under U.S. EPA Method 21 produced a response greater than zero, the monitoring device was considered capable of detecting the chemical gas leak. Similarly, if the monitoring device acceptable under U.S. EPA Method 21 produced a response equal to zero, the monitoring device was considered incapable of detecting the chemical gas leak.

The responses of both the Sherlock[®] VOC imaging spectrometer and the monitoring device acceptable under U.S. EPA Method 21 under the same test conditions were compared. If both the Sherlock[®] VOC imaging spectrometer and the monitoring device acceptable under U.S. EPA Method 21 proved capable of detecting the chemical gas leak, then both units were considered to have agreed under the specific test condition. Likewise, if either the Sherlock[®] VOC imaging spectrometer or the monitoring device acceptable under U.S. EPA Method 21 proved incapable of detecting the chemical gas leak under the specified test conditions, then the units were considered to have disagreed. Test conditions, under which a response from the either the Sherlock[®] VOC imaging spectrometer or the monitoring device acceptable under U.S. EPA Method 21 were not obtained, were excluded from the comparison.

Chapter 6

Test Results

As mentioned previously, this verification test included both quantitative and qualitative evaluations. The quantitative evaluation was conducted to assess the method detection limits of the Sherlock[®] VOC imaging spectrometer, the detection of chemical gas species relative to a portable monitoring device acceptable under U.S. EPA Method 21, as well as, by testing the influence of confounding factors. The qualitative evaluation was performed to document the operational aspects of imaging spectrometer when they were used during verification testing. The following sections provide the results of the quantitative and qualitative evaluations.

6.1 Method Detection Limit

The method detection limit of each chemical compound was determined according to the procedures discussed in Section 3.2.2. Table 8 through Table 11 present the method detection limits of each chemical compound determined during laboratory testing. Table 8 through Table 11 identify each test condition evaluated (i.e., stand-off distance, background material, and wind speed), the temperatures of the laboratory and of the chemical leak, the response of the portable monitoring device acceptable under U.S. EPA Method 21, and, the method detection limits for each test condition. Table 12 summarizes the range of method detection limits in units of grams per hour (g/hr) found during laboratory testing as well as presents the overall detection limit variation for each compound. The overall detection limit variation presented in Table 12 was calculated using Equation 1 in Chapter 5.

6.2 Detection Agreement to a Portable Monitoring Device

The detection of a single chemical gas leak in either the laboratory or field environments was determined by the operator as well as two confirming individuals as discussed in Section 3.2.1. The leak rate was known from certified gas cylinders and calibrated flow meters in the laboratory, or was determined through the bagging method during field testing. During both the laboratory and field tests, a portable monitoring device acceptable under U.S. EPA Method 21 was used to sample the leaks. The following section presents results on the ability of the Sherlock[®] VOC imaging spectrometer to detect a chemical gas species relative to a portable monitoring device acceptable under U.S. EPA Method 21.

Table 8. Sherlock® VOC Method Detection Limits at 10 Feet with Cement Board Background

Compound	Wind Speed (mph)	Ambient Temp. (°F)	Leak Temp. (°F)	M21 Device Conc. (ppmv)	Method Detection Limit (g/hr)
1,3-butadiene	0	70.4	71.0	≥ 2,000	8.1
Acetic acid	0	72.2	77.6	25	1.7
	2.5	74.9	84.6	511	9.2
	5	74.9	79.4	675	23
Acrylic acid	0	71.0	95.7	43	7.4
Benzene	0	73.0	80.3	877	3.2
	2.5	72.3	81.5	≥ 2,000	≥ 70 ^(a)
Methylene chloride	0	70.9	74.8	N.A. ^(b)	> 70 ^(a)
	2.5	72.3	78.4	N.A. ^(b)	> 70 ^(a)
Ethylene	0	71.2	71.3	No data ^(c)	3.3
	2.5	71.3	72.2	842	250
	5	71.5	72.2	382	≥ 275 ^(a)
Methanol	0	71.5	76.6	N.A. ^(b)	2.1
	2.5	70.0	91.5	N.A. ^(b)	29
	5	70.0	83.3	N.A. ^(b)	69
Pentane	0	71.9	79.3	0.70	0.83
	2.5	71.3	82.6	190	55
	5	70.9	77.9	164	≥ 55 ^(a)
Propane	0	70.5	70.4	N.A. ^(b)	0.88
	2.5	71.8	71.9	N.A. ^(b)	9.8
	5	71.1	71.6	N.A. ^(b)	13
Styrene	0	71.5	80.5	≥ 2000	15

(a) The leak could not be detected below the highest reliable flow rate supplied by the delivery system.

(b) N.A. – not applicable. The ionization potential of this compound is higher than is capable of detection by the device used. Therefore, any raw data measured with this device is not reported in this table.

(c) No data – the leak concentration was inadvertently not collected by laboratory personnel using the M21 device.

Table 9. Sherlock® VOC Method Detection Limits at 30 Feet with Cement Board Background

Compound	Wind Speed (mph)	Ambient Temp. (°F)	Leak Temp. (°F)	M21 Device Conc. (ppmv)	Method Detection Limit (g/hr)
1,3-butadiene	0	71.5	72.2	≥ 2,000	27
Acetic acid	0	71.7	76.5	44	1.9
	2.5	74.8	87.4	39	32
	5	74.7	81.5	516	81
Acrylic acid	0	71.7	92.0	0.80	0.92
Benzene	0	70.0	77.8	≥ 2,000	14
	2.5	74.8	83.0	≥ 2,000	≥ 70 ^(a)
	5	74.6	79.2	≥ 2,000	≥ 70 ^(a)
Methylene chloride	0	69.5	74.3	N.A. ^(b)	> 70 ^(a)
	0	70.5	70.7	No data ^(c)	17
Ethylene	2.5	71.3	72.2	863	278
	0	71.7	83.3	N.A. ^(b)	3.5
Methanol	2.5	72.2	92.2	N.A. ^(b)	66
	5	70.0	82.1	N.A. ^(b)	≥ 69 ^(a)
	0	72.0	76.6	168	2.5
Pentane	2.5	70.3	85.8	36	39
	5	70.3	80.1	111	≥ 55 ^(a)
	0	70.8	70.7	N.A.	1.3
Propane	2.5	71.8	71.7	N.A.	22
	5	72.3	72.0	N.A.	82
	0	71.0	80.0	≥ 2000	25

(a) The leak could not be detected below the highest reliable flow rate supplied by the delivery system.

(b) N.A. – not applicable. The ionization potential of this compound is higher than is capable of detection by the device used. Therefore, any raw data measured with this device is not reported in this table.

(c) No data – the leak concentration was inadvertently not collected by laboratory personnel using the M21 device.

Table 10. Sherlock® VOC Method Detection Limits at 10 Feet with Gas Cylinder Background

Compound	Wind Speed (mph)	Ambient Temp. (°F)	Leak Temp. (°F)	M21 Device Conc. (ppmv)	Method Detection Limit (g/hr)
1,3-butadiene	0	70.5	71.5	≥ 2,000	13
Acetic acid	0	71.8	80.5	No data ^(a)	1.7
	2.5	74.0	89.4	0.40	9.2
	5	74.7	78.9	377	23
Acrylic acid	0	71.7	97.0	26	2.3
Benzene	0	72.6	92.3	≥ 2,000	7.0
	2.5	74.3	83.7	1022	≥ 70 ^(b)
Methylene chloride	0	70.9	80.9	N.A. ^(c)	> 70 ^(b)
	2.5	74.1	81.3	N.A. ^(c)	> 70 ^(b)
Ethylene	0	71.6	71.6	No data ^(a)	22
	2.5	71.1	72.0	1,790	≥ 209 ^(b)
	5	71.5	71.9	547	≥ 278 ^(b)
Methanol	0	70.9	83.3	N.A. ^(c)	2.1
	2.5	70.0	91.2	N.A. ^(c)	29
	5	70.2	83.4	N.A. ^(c)	69
Pentane	0	70.6	79.5	12	1.9
	2.5	72.4	85.0	185	19
	5	72.0	79.6	190	47
Propane	0	71.2	71.0	N.A. ^(c)	1.1
	2.5	71.8	71.8	N.A. ^(c)	9.8
	5	70.9	71.5	N.A. ^(c)	14
Styrene	0	72.4	85.6	≥ 2000	18

- (a) No data – the leak concentration was inadvertently not collected by laboratory personnel using the M21 device.
- (b) The leak could not be detected below the highest reliable flow rate supplied by the delivery system.
- (c) N.A. – not applicable. The ionization potential of this compound is higher than is capable of detection by the device used. Therefore, any raw data measured with this device is not reported in this table.

Table 11. Sherlock® VOC Method Detection Limits at 30 Feet with Gas Cylinder Background

Compound	Wind Speed (mph)	Ambient Temp. (°F)	Leak Temp. (°F)	M21 Device Conc. (ppmv)	Method Detection Limit (g/hr)
1,3-butadiene	0	71.2	72.0	≥ 2,000	27
Acetic acid	0	71.5	78.4	39	1.9
	2.5	74.6	88.7	777	32
	5	74.4	79.2	560	81
Acrylic acid	0	71.4	88.4	3.7	3.2
Benzene	0	71.8	77.1	≥ 2,000	14
	2.5	75.0	83.8	≥ 2,000	≥ 70 ^(a)
	5	74.6	79.4	≥ 2,000	≥ 70 ^(a)
Methylene chloride	0	69.8	77.2	N.A. ^(b)	> 70 ^(a)
Ethylene	0	71.5	72.0	No data ^(c)	32
	2.5	71.0	72.2	1402	≥ 278 ^(a)
Methanol	0	71.7	80.1	N.A. ^(b)	3.5
	2.5	72.2	93.7	N.A. ^(b)	66
	5	70.1	84.0	N.A. ^(b)	≥ 69 ^(a)
Pentane	0	71.9	76.5	228	2.8
	2.5	75.1	88.8	443	≥ 55 ^(a)
	5	72.0	81.7	113	≥ 55 ^(a)
Propane	0	70.6	70.3	N.A. ^(b)	1.3
	2.5	71.6	71.5	N.A. ^(b)	22
	5	71.0	71.8	N.A. ^(b)	235
Styrene	0	73.0	85.4	≥ 2000	25

(a) The leak could not be detected below the highest reliable flow rate supplied by the delivery system.

(b) N.A. – not applicable. The ionization potential of this compound is higher than is capable of detection by the device used. Therefore, any raw data measured with this device is not reported in this table.

(c) No data – the leak concentration was inadvertently not collected by laboratory personnel using the M21 device

Table 12. Sherlock® VOC Range of Method Detection Limits and Overall Method Detection Limit Variation (g/hr)^(a)

Compound	Minimum	Maximum	Overall Variation ^(b)
1,3-butadiene	8.0	27	
Acetic acid	1.7	81	29
Acrylic acid	0.92	7.4	
Benzene	3.2	≥ 70 ^{(c), (d)}	31
Methylene chloride	≥ 70 ^(c)	≥ 70 ^(c)	
Ethylene	3.3	≥ 278 ^(c)	127
Methanol	2.1	≥ 69 ^(d)	31
Pentane	0.83	≥ 55 ^{(c), (d)}	25
Propane	0.88	235 ^(c)	67
Styrene	15	25	

(a) Minimum and maximum values shown were measured at a 0-mph wind speed unless otherwise noted.

(b) When sample sizes are small ($N < 10$), standard deviations provide a biased estimate of the variability, therefore only the range is provided when there were fewer than 10 method detection limits were determined.

(c) Measured at a 2.5-mph wind speed condition.

(d) Measured at a 5-mph wind condition.

6.2.1 Laboratory Testing

Table 13 presents the percent agreement between the ability of the Sherlock® VOC imaging spectrometer and of a portable monitoring device acceptable under U.S. EPA Method 21 to detect a chemical gas leak under the conditions tested. Percent agreement was calculated according to Equation 2 in Chapter 5. The calculation of percent agreement excludes those laboratory test measurements for which a response was not collected using a portable monitoring device acceptable under U.S. EPA Method 21. In addition, percent agreement was not evaluated for methylene chloride, methane, methanol, and propane because these compounds have an ionization potential greater than that which could be supplied by the Industrial Scientific IBRID MX6 with PID sensor (i.e., the device is incapable of detecting these compounds).

Table 13. Summary of Detection Agreement Between Sherlock® VOC and a Method 21 Portable Monitoring Device

Gas Tested	No. of Tests in which Agreed	Total No. of Tests Completed	Percent Agreement
1,3-butadiene	4	4	100%
Acetic acid	11	11	100%
Acrylic acid	4	4	100%
Benzene	4	10	40%
Ethylene	2	6	33%
Pentane	8	12	75%
Styrene	4	4	100%

6.2.2 Field Testing

During field testing, nine leaking components were viewed using the Sherlock® VOC imaging spectrometer using the procedures described in Section 3.2.1. Table 14 identifies whether each chemical species gas leak was observed by the camera and the concentration of the leak as

determined by a portable monitoring device acceptable under U.S. EPA Method 21. In addition, this table identifies the type of component that was leaking, the average chemical-specific mass leak rate from the component as determined by reference sampling, the distance the leak was observed and the wind speed. Daily meteorological conditions were obtained from the Dow Chemical Company's on-site weather station. Although the wind speed and daily maximum and minimum temperatures were obtained from this meteorological tower, the actual wind speed and ambient and background temperatures at each leak location at the time of observation are unknown. Additional discussions describing each leak location are provided in the following sections.

Table 14. Summary of Field Testing Results Using the Sherlock[®] VOC

Leak Location	Leaking Component Type	Wind Speed (mph)	Stand-off Distance (ft)	M21 Device Screening Conc. (ppmv)	Leak Detected by Camera?	Bagging Results: Average Leak Rate (g/hr)
1	3-inch Plug	8	12	> 100,000	No	8.79 (methane) 4.31 (ethylene)
2	¼-inch Tube	21	10 30	20,500	Yes No	0.951 (ethylene)
3	½-inch Connector	21	10	> 100,000	No	2.32 x 10 ⁻³ (ethylene) 7.78 (methane)
5	6-inch Block Valve	21	10	> 100,000	No	5.24 x 10 ⁻² (ethylene) 8.68 x 10 ⁻³ (styrene) 0.077 (benzene)
6	8-inch Block Valve	21	10	20,500	No	3.44 ^(a) (benzene)
7	Control Valve Flange	18	10	17,500	No	1.95 x 10 ⁻³ (ethylene) 0.282 (benzene)
8	2-inch Block Valve	18	10	8,000 ^(b)	No	1.92 ^(b) (1,3-butadiene)
9	1-inch Valve Plug	18	10	835	No	0.350 (methylene chloride)
10	6-inch Pressure Relief Valve	5	10	> 100,000	No	6.78 (propylene dichloride)

- (a) As reported in Table 5, the pre- and post-bagging leak concentrations, as measured by the TVA, differed by 24.4%. This exceeds the minimum acceptance criterion of 20% for the DQI for the confirmation of detected leaks. Thus, this data is considered suspect and reported with this data qualifier.
- (b) As reported in Table 4, the calibration check response for the TVA, conducted after screening this component, resulted in a 24% difference. This exceeded the minimum acceptance criterion of 10% for the DQI for the bias and accuracy of sample screening measurements using a portable monitoring device. After recalibration of the TVA, the leak concentration from this component was not reconfirmed with the TVA. Thus, this data is considered suspect and reported with this data qualifier.

Leak Location 1. A leak was identified originating from a 3-inch plug in service with a process stream containing ethane, ethylene, methane, and propane. Screening of the component with the TVA caused an over range reading (estimated as > 100,000 ppmv). The leak was viewed but not detected with the Sherlock[®] VOC imaging spectrometer at a stand-off distance of 12 ft with the sun at the observers back. The leak was bagged and a duplicate reference sample was collected into two evacuated SUMMA canisters. The SUMMA canisters were shipped to the off-site GC laboratory and analyzed for ethylene and methane concentrations. Daily weather conditions, as

reported by the on-site weather station, were clear conditions, a daily minimum and maximum temperature of 41 and 61 degrees Fahrenheit (°F), respectively, with wind out of the east at 8 mph.

The average mass leak rate of ethylene measured at this leak location was 4.31 g/hr. This value is lower than the lowest method detection limit measured for this compound during the laboratory phase of this verification test.

Leak Location 2. A leak was identified originating from a ¼-inch tube in service with a process stream containing ethane and ethylene. Screening of the component with the TVA resulted in a concentration reading of 20,500 ppmv. The leak was viewed with the Sherlock® VOC imaging spectrometer at stand-off distances of 10 and 30 ft with the sun to the left of the observer. The leak was detected by the imaging spectrometer at the 10 ft stand-off distance but was not detected at the 30 ft stand-off distance. Wind direction at the location was noted as originating from behind the observer and the site was shaded by piping and other equipment. The leak was bagged and a duplicate reference sample was collected into two evacuated SUMMA canisters. The SUMMA canisters were shipped to the off-site GC laboratory and analyzed for ethylene concentration. Daily weather conditions, as reported by the on-site weather station, were clear conditions, a daily minimum and maximum temperature of 42 and 70 °F with wind out of the south southeast at 21 mph.

The average mass leak rate of ethylene measured at this leak location was 0.951 g/hr. This value is lower than the lowest method detection limit measured for this compound during the laboratory phase of this verification test.

Leak Location 3. A leak was identified originating from a ½-inch connector in service with a process stream containing acetylene, ethane, ethylene, methane, propane, and propylene. Screening of the component with the TVA caused an over range reading (estimated as > 100,000 ppmv). The leak was viewed with the Sherlock® VOC at a stand-off distance of 10 ft with the sun to the right of the observer. The leak was not detected by the imaging spectrometer at this stand-off distance. Wind direction at the location was noted as originating from the right of the observer and the site was shaded by piping and other equipment. The leak was bagged and a duplicate reference sample was collected into two evacuated SUMMA canisters. The SUMMA canisters were shipped to the off-site GC laboratory and analyzed for ethylene and methane concentrations. Daily weather conditions, as reported by, were clear conditions, a daily minimum and maximum temperature of 42 and 70 °F with wind out of the south southeast at 21 mph.

The average mass leak rate of ethylene measured at this leak location was 2.32×10^{-3} g/hr. This value is lower than the lowest method detection limit measured with the Sherlock® VOC for this compound during the laboratory phase of this verification test.

Leak Location 4. Leak location 4 contained a leaking component that was misidentified as being in service with styrene. This sample location was confirmed to be in ethylbenzene service and thus no analytical results are reported for this leak location. The Sherlock® VOC imaging spectrometer was not able to detect this leak.

Leak Location 5. A leak was identified originating from a 6-inch block valve in service with a process stream containing benzene, ethane, ethylene, ethylbenzene, styrene, and toluene. Screening of the component with the TVA caused an over range reading (estimated as

> 100,000 ppmv). The leak was viewed with the Sherlock[®] VOC at stand-off distance of 10 ft; the leak could not be detected at this distance. The site was shaded and the viewing background was concrete. The leak was bagged and a duplicate reference sample was collected into two evacuated SUMMA canisters. The SUMMA canisters were shipped to the off-site GC laboratory and analyzed for benzene, ethylene, and styrene concentrations. Daily weather conditions, as reported by the on-site weather station, were clear conditions, a daily minimum and maximum temperature of 48 and 79 °F with wind out of the north at 21 mph.

The average mass leak rates of ethylene, styrene, and benzene measured at this leak location were 5.24×10^{-2} , 8.68×10^{-3} , and 0.077 g/hr, respectively. These values are all below the lowest method detection limits measured with the Sherlock[®] VOC for these compounds during the laboratory phase of this verification test.

Leak Location 6. A leak was identified originating from an 8-inch block valve in service with a process stream containing benzene, toluene, hexane, and other aromatic hydrocarbons. Screening of the component with the TVA resulted in a concentration reading of 20,500 ppmv. The leak was viewed with the Sherlock[®] VOC at a stand-off distance of 10 ft with the sun to the right of the camera observer; the leak could not be detected at this distance. The site was an exterior location and weather conditions were noted as slightly overcast with moderate wind originating from the right of the observer. The leak was bagged and a duplicate reference sample was collected into two evacuated SUMMA canisters. The SUMMA canisters were shipped to the off-site GC laboratory and analyzed for benzene concentration. Daily weather conditions, as reported by the on-site weather station, were clear conditions, a daily minimum and maximum temperature of 48 and 79 °F with wind out of the north at 21 mph.

Leak Location 7. A leak was identified originating from a control valve flange in service with a process stream containing benzene, butane, butylbenzene, all isomers of diethylbenzene, ethane, ethylbenzene, ethylene, hexane, toluene, and other aromatic hydrocarbons. Screening of the component with the TVA resulted in a concentration reading of 17,500 ppmv. The leak was viewed with the Sherlock[®] VOC at a stand-off distance of 10 ft with the sun behind the camera observer; the leak could not be detected at this distance. The site was located on the second deck of the chemical plant and weather conditions were qualitatively noted as very windy. The viewing background was other plant piping and equipment. The leak was bagged and a duplicate reference sample was collected into two evacuated SUMMA canisters. The SUMMA canisters were shipped to the off-site GC laboratory and analyzed for benzene and ethylene concentrations. Daily weather conditions, as reported by the on-site weather station, were partly cloudy conditions, a daily minimum and maximum temperature of 43 and 65 °F with wind out of the north at 18 mph.

The average mass leak rates of ethylene and benzene measured at this leak location were 1.95×10^{-3} and 0.282 g/hr, respectively. These values are all below the lowest method detection limits measured with the Sherlock[®] VOC for these compounds during the laboratory phase of this verification test.

Leak Location 8. A leak was identified originating from a 2-inch block valve in service with a process stream containing 1,3-butadiene. Screening of the component with the TVA resulted in a concentration reading of 8,000 ppmv. The leak was viewed with the Sherlock[®] VOC at a stand-off distance of 10 ft; the leak could not be detected at this distance. The site was an exterior location on a marine vapor recovery line at a marine vapor recovery system and weather conditions were qualitatively noted to be very windy and overcast. The leak was bagged and a

duplicate reference sample was collected into two evacuated SUMMA canisters. The SUMMA canisters were shipped to the off-site GC laboratory and analyzed for 1,3-butadiene concentration. Daily weather conditions, as reported by the on-site weather station, were partly cloudy conditions, a daily minimum and maximum temperature of 43 and 65 °F with wind out of the north at 18 mph.

The average mass leak rate of 1,3-butadiene measured at this leak location was 1.92 g/hr. This value is lower than the lowest method detection limit measured with the Sherlock[®] VOC for this compound during the laboratory phase of this verification test.

Leak Location 9. A leak was identified originating from a 1-inch valve plug in service with a process stream containing dichloromethane (methylene chloride). Screening of the component with the TVA resulted in a concentration reading of 835 ppmv. The leak was viewed with the Sherlock[®] VOC at a stand-off distance of 10 ft; the leak could not be detected at this distance. The site was an exterior location and weather conditions were qualitatively noted as overcast with calm winds. The viewing background was concrete ground and a few metal pipe supports. The leak was bagged and a duplicate reference sample was collected into two evacuated SUMMA canisters. The SUMMA canisters were shipped to the off-site GC laboratory and analyzed for dichloromethane concentration. Daily weather conditions, as reported by the on-site weather station, were partly cloudy conditions, a daily minimum and maximum temperature of 43 and 65 °F with wind out of the north at 18 mph.

The average mass leak rate of dichloromethane (methylene chloride) measured at this leak location was 0.350 g/hr. This value is lower than the lowest method detection limit measured with the Sherlock[®] VOC for this compound during the laboratory phase of this verification test.

Leak Location 10. A leak was identified originating from a 6-inch pressure relief valve in service with a process stream containing 1,2,3-trichloropropane, 2,3-dichloropropanol, 2-methyl-2-pentenal, 1-chloro-2,3-epoxypropane, and 1,2-dichloropropane (propylene dichloride). Screening of the component with the TVA caused an over range reading (estimated as > 100,000 ppmv). The leak was viewed with the Sherlock[®] VOC at a stand-off distance of 10 ft; the leak could not be detected at this distance. The site was an exterior location (on top of a storage tank platform) and weather conditions were qualitatively noted as overcast, breezy, and cold. The leak was bagged and a duplicate reference sample was collected into two evacuated SUMMA canisters. The SUMMA canisters were shipped to the off-site GC laboratory and analyzed for 1,2-dichloropropane concentration. Daily weather conditions, as reported by the on-site weather station, were partly cloudy conditions, a daily minimum and maximum temperature of 41 and 50 °F with wind out of the north at 5 mph.

6.3 Confounding Factors

The method detection limits generated during laboratory testing presented in Table 8 through Table 11 were inspected to identify general trends that the confounding factors of stand-off distance, wind speed, and background materials impart on the method detection limits for the gaseous chemical species leaks observed using the Sherlock[®] VOC imaging spectrometer. The following general trends were noted when using the imaging spectrometer.

- Stand-off Distance – Method detection limits generally increased as the viewing distance increased. Two exceptions to this general observation were found. The first occurred

when viewing a pentane leak at a 2.5-mph wind speed and a cement board background. The second occurred when viewing an acrylic acid leak with no wind and a cement board background.

- Wind Speed – Method detection limits generally increased with increased wind speed;
- Background Materials – Method detection limits were generally lower when viewed against the cement board background. A single exception to this observation was noted when viewing an acrylic acid leak at a 0-mph wind speed and at a 10 ft viewing distance.

During field testing, confounding factors were recorded either quantitatively or qualitatively and are reported in Table 14. A rigid analysis of the influence of confounding factors was not undertaken using field testing data, however, it is generally noted that because the optical imaging instrument detected only a few of the chemical leaks in the field, the confounding factors of wind speed, stand-off distance, and background materials affected the detection capability of the cameras.

6.4 Operational Factors

The Sherlock[®] VOC imaging spectrometer was found to be easily setup in a small, three ft by three ft area and deployed within approximately 10 minutes for portable gas leak observations. In terms of field portability, the imaging spectrometer was moderate in weight (weighing approximately 19 pounds with battery), easily carried by one person and was provided with a rugged shipping case for transportation.

The Sherlock[®] VOC imaging spectrometer may be powered with either an Anton Bauer Titan Power Supply/Charger, a 70 watt unit that accepts 90 to 265 volt (alternating current) at 50 to 60 Hz, for stationary applications or with an Anton Bauer Compatible Digital Hytron Nickel Metal-Hydride battery for mobile field observations. The battery for each instrument was used and held its charge when performing visual screening of leaking components. The camera observer sees the infrared image through a standard, mounted 3.5-inch on diagonal liquid crystal display viewing screen when using the imaging spectrometer; these images may be recorded to either an internal CompactFlash card (up to 2 gigabytes) or to a Sony DV-1000 Digital Video Recorder system or any other digital recording system.

Ease of use was not investigated with a newly trained operator, as the personnel from Industrial Scientific Corporation operated the Sherlock[®] VOC during both laboratory and field testing. Verification test team members, however, did observe that the instrument was used by the camera operator with relative ease. The Sherlock[®] VOC imaging spectrometer is not intrinsically safe, and cannot be used in explosive atmospheres or environments.

During this verification test, all chemical leaks were required to be observed by the instrument operator and two additional confirming individuals to be considered as “detected” by the optical imaging device. During verification testing, there were instances where either one or two of the three observers were able to observe the chemical leak. This indicates that the ability of the operator using the instrument to positively identify the chemical leak may have an influence on the operation of the system.

The cost of the Sherlock[®] VOC imaging spectrometer is \$89,000 and includes the LCD video display, a Pelican shipping case, a battery and battery charger, personal computer, HYPAT software, and all necessary cables.

Chapter 7

Performance Summary

Method Detection Limits. Method detection limits were determined during the laboratory testing. Table 15 summarizes the minimum and maximum method detection limit obtained during laboratory testing using the Sherlock[®] VOC imaging spectrometer. Specific details, including the test conditions at which these method detection limits were obtained, are provided in Table 8 through Table 11 in Chapter 6. The overall detection limit variation for each chemical obtained using the imaging spectrometer are presented in Table 12 in Chapter 6.

Detection of Chemical Gas Species Relative to a Portable Monitoring Device. The ability of the Sherlock[®] VOC imaging spectrometer to detect a gaseous leak of a chemical relative to a portable monitoring device acceptable under U.S. EPA Method 21 was assessed during both laboratory and field testing. During laboratory testing, after the method detection limit had been reached for a particular chemical under the specified test conditions, the leak was sampled by the portable monitoring device. Table 15 presents the percent agreement between the ability of the Sherlock[®] VOC imaging spectrometer and of a portable monitoring device acceptable under U.S. EPA Method 21 to detect a chemical gas leak under the conditions tested in the laboratory.

During field testing a portable monitoring device acceptable under U.S. EPA Method 21 was used to screen each leaking component as part of the bagging reference method used. Table 16 reports the responses of the portable screening device when screening leaking components, identifies whether the Sherlock[®] VOC imaging spectrometer was able to detect the chemical leak from the leaking component, and reports the chemical-specific mass rate of emissions from the leaking component as obtained through reference sampling.

Table 15. Summary of Sherlock[®] VOC Method Detection Limits^(a) and Percent Agreement with a Method 21 Monitoring Device During Laboratory Testing

Compound	Method Detection Limit (g/hr)		Agreement with Method 21 Monitoring Device	
	Minimum	Maximum	Total No. of Tests Performed	Percent Agreement
1,3-butadiene	8.0	27	4	100%
Acetic acid	1.7	81	11	100%
Acrylic acid	0.92	7.4	4	100%
Benzene	3.2	≥ 70 ^{(b), (c)}	4	40%
Methylene chloride	≥ 70 ^(b)	≥ 70 ^(b)		No data ^(d)
Ethylene	3.3	≥ 278 ^(b)	2	33%
Methanol	2.1	≥ 69 ^(c)		No data ^(d)
Pentane	0.83	≥ 55 ^{(b), (c)}	8	75%
Propane	0.88	235 ^(b)		No data ^(d)
Styrene	15	25	4	100%

(a) Minimum and maximum method detection limits were measured at a 0-mph wind speed unless otherwise noted.

(b) Measured at a 2.5-mph wind speed.

(c) Measured at a 5-mph wind speed.

(d) Percent agreement was not evaluated for methylene chloride, methanol, and propane because these compounds have an ionization potential greater than the energy which could be supplied by the Industrial Scientific IBRID MX6 with PID sensor.

Confounding Factors. Stand-off distance, wind speed, and background materials generally impacted the performance of the Sherlock[®] VOC imaging spectrometer (e.g., increasing the viewing distance from the leak increased the method detection limits). Details of the effects of confounding factors can be found in Section 6.3.

Operational Factors. The Sherlock[®] VOC imaging spectrometer was found to be easily set up and ready to deploy in 10 minutes. The camera is moderate in weight (19 pounds with battery) and operated on batteries when performing visual screening of leaking components. Because the cameras were operated by Industrial Scientific personnel and there were some disagreements on detections with the two confirming individuals, the ability of the operator may influence the operation of the camera. The Sherlock[®] VOC imaging spectrometer is not intrinsically safe, and cannot be used in explosive atmospheres or environments.

The cost of the Sherlock[®] VOC imaging spectrometer is \$89,000 and includes the LCD video display, a Pelican shipping case, a battery and battery charger, personal computer, HYPAT software, and all necessary cables.

Table 16. Summary of Field Testing Results of the Sherlock[®] VOC

Leak Location	Leaking Component Type	Wind Speed (mph)	Stand-off Distance (ft)	M21 Device Screening Conc. (ppmv)	Leak Detected by Camera?	Bagging Results: Average Leak Rate (g/hr)
1	3-inch Plug	8	12	>100,000	No	8.79 (methane) 4.31 (ethylene)
2	¼-inch Tube	21	10 30	20,500	Yes No	0.951 (ethylene)
3	½-inch Connector	21	10	>100,000	No	2.32 x 10 ⁻³ (ethylene) 7.78 (methane)
5	6-inch Block Valve	21	10	>100,000	No	5.24 x 10 ⁻² (ethylene) 8.68 x 10 ⁻³ (styrene) 0.077 (benzene)
6	8-inch Block Valve	21	10	20,500	No	3.44 ^(a) (benzene)
7	Control Valve Flange	18	10	17,500	No	1.95 x 10 ⁻³ (ethylene) 0.282 (benzene)
8	2-inch Block Valve	18	10	8,000 ^(b)	No	1.92 ^(b) (1,3-butadiene)
9	1-inch Valve Plug	18	10	835	No	0.350 (methylene chloride)
10	6-inch Pressure Relief Valve	5	10	>100,000	No	6.78 (propylene dichloride)

- (a) As reported in Table 5, the pre- and post-bagging leak concentrations, as measured by the TVA, differed by 24.4 %. This exceeds the minimum acceptance criterion of 20% for the DQI for the confirmation of detected leaks. Thus, this data is considered suspect and reported with this data qualifier.
- (b) As reported in Table 4, the calibration check response for the TVA, conducted after screening this component, resulted in a 24% difference. This exceeded the minimum acceptance criterion of 10% for the DQI for the bias and accuracy of sample screening measurements using a portable monitoring device. After recalibration of the TVA, the leak concentration from this component was not reconfirmed with the TVA. Thus, this data is considered suspect and reported with this data qualifier.

Chapter 8 References

1. *Test/QA Plan for Verification of Leak Detection and Repair Technologies*, Battelle, Columbus, Ohio, September 18, 2008.
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3. *EPA Method 21- Detection of Volatile Organic Compound Leaks*, EPA-600/2-18-110; U.S. EPA, September 1981.
4. Panek, J., P. Drayton, and D. Fashimpaur. *Controlled Laboratory Sensitivities and Performance Evaluation of Optical Leak Imaging Infrared Cameras for Identifying Alkane, Alkene, and Aromatic Compounds*, Proceedings of the 99th Annual Conference and Exposition of the Air and Waste Management Association, New Orleans, June 20 – 23, 2006, Manuscript number 06-A-159-AWMA, Curran Associates, Inc., Red Hook, New York, March 2007.
5. *EPA Protocol for Equipment Leak Emissions Estimates*, EPA-453/R-95-017; U.S. EPA: Research Triangle Park, NC, November 1995.
6. *EPA Method 18 – Measurement of Gaseous Organic Compound Emissions by Gas Chromatography*, 40 CFR, Part 60, Appendix A; April, 1994.
7. *EPA Method 205 – Verification of Gas Dilution Systems for Field Instrument Calibrations*, 40 CFR, Part 51, Appendix M, September, 1996.