



STANDARD OPERATING PROCEDURES

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DETERMINATION OF NITROGLYCERIN IN SOIL GAS BY SCAN X JR.

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SUPERCEDES: SOP #1715; Revision 0; 01/30/90; U.S. EPA Contract EP-W-09-031.



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1.0 SCOPE AND APPLICATION

This procedure was developed to analyze for nitroglycerine in soil gases. A level of action was selected at 1 ppm (part per million) as determined by headspace analysis of a commercially obtained 1 ppm nitroglycerine in soil standard. The actual vapor content in the headspace above the soil standard will be considerably less due to its low vapor pressure. The Scan X Jr. can see nitroglycerine in vapors at the parts per billion (ppb) range, depending upon the sampling time used and background interferences.

These are standard (i.e., typically applicable) operating procedures which may be varied or changed as required, dependent on site conditions, equipment limitations or limitations imposed by the procedure or other procedure limitations. In all instances, the ultimate procedures employed should be documented and associated with the final report.

Mention of trade names or commercial products does not constitute U.S. EPA endorsement or recommendation for use.

2.0 METHOD SUMMARY

Holes were prepared as per the soil gas sampling methodology defined in Environmental Response Team/Scientific, Engineering, Response and Analytical Services (ERT/SERAS) ERT/SERAS SOP# 2042, Soil Gas Sampling. These holes were evacuated via Gilian pump for 7-8 seconds to displace any void volume in the sampling lines. The sampling probe of the Scan X Jr. was inserted directly onto the soil gas sample line and the probe sampled for exactly 10 seconds. Any observable response was noted from an alarm, the LED readout on the Scan X Jr. unit, and a volt meter connected to the recorder output of the Scan X Jr. unit. Periodically, the headspace of a 1-ppm or 10-ppm soil standard vial would be analyzed to insure the instrument was responding per specifications.

3.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

Since the soil gas analysis is performed as a real-time in-situ measurement, no preservation or storage is required. Reference soil standards containing known levels of nitroglycerine should be kept in closed vials and protected from direct sunlight. Standards should be refrigerated when not in use.

4.0 INTERFERENCES AND POTENTIAL PROBLEMS

It has not yet been determined if any environmental or weather conditions adversely effect the performance of the Scan X Jr. However, since the unit employs an Electron Capture Detector (ECD), it is possible that other electrophilic compounds (i.e., halogenated compounds), may respond to the ECD. Since the unit operates at very high temperatures (>200°C), most ECD responding compounds typically present at hazardous waste sites will elute before the peak of the target compound of interest. As of yet, however, this has not yet been tested in the laboratory.

5.0 EQUIPMENT

The Scan X Jr. is a portable gas chromatograph equipped with an ECD and is manufactured by Sentex Sensing Technology of Ridgefield, N.J. The unit is self contained and can run in a completely portable mode using its internal battery pack and helium carrier gas cylinder. During field runs, an additional battery pack was attached to the unit to supply power for the high temperature requirement needed for nitroglycerine analysis. This added power pack is also supplied by Sentex. When the instrument was not field mobilized, it was charged via 12 VDC/115 VAC charger,



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Model PSC-12, 4000, manufactured by Power Sonic Corp., Redwood City, CA. An optional 12 VDC charger which operates from a car cigarette lighter is available from Sentex.

To facilitate determination of the quantitative responses to different levels of nitroglycerine, a Micronta Model 22-188, millimeter, available from Radio Shack, was attached to the recorder output of the Scan X Jr. This was taped to the outside of the unit and was continually observed to record detector performance.

6.0 REAGENTS

The Scan X Jr. requires replacement of the carrier gas, ultra-high purity helium (99.999%), when the internal tank pressure drops below 200 psi. The helium is available from either Scott Specialty Gases or Matheson Gas. The Scan X Jr. is checked against soil standards of nitroglycerin obtained from Divex Corp., Columbia, South Carolina.

7.0 PROCEDURES

7.1 Calibration

1. Follow instructions in the Operator's Manual for instrument start-up. This phase may require 20 minutes or longer.
2. Calibrate the instrument utilizing pre-prepared standards at predetermined sampling exposure intervals.
3. Follow general tuning procedures as outlined in the Operator's Manual.

7.2 Operation

1. Soil gas probe holes are created as per ERT/SERAS SOP# 2042, Soil Gas Sampling, using slam bars and 3/8" diameter stainless steel lines.
2. A small piece of Teflon tubing is placed on top of the stainless steel gas line and connected to a Gilian personal sampling pump.
3. The Gilian pump is activated and the hole is purged for 7-8 seconds at 3000 cc/min.
4. The Gilian pump is removed, and the Scan X Jr.'s probe is immediately inserted into the Teflon fitting on top of the stainless steel line. The probe's sampling pump is then started and allowed to run for exactly ten seconds.
5. Afterwards, the volt meter is observed to determine if any peaks are present and if so, at what intensity (as determined by signal voltage output). In addition, the Scan X Jr.'s audible alarm and LED readout are also noted to aid in identifying and quantitating any target compound (i.e. nitroglycerine) peak that may be present.
6. After approximately one minute, the probe will light the green "ready" indicator and the next sampling station is ready for analysis.

8.0 CALCULATIONS

At present, only three nitroglycerine soil standards are available: 1, 10, and 100 ppm. The signal voltage responses on the volt meter are recorded for all three standards at sampling times of two, five, and ten seconds. The soil gas sampling stations are sampled at two, five, and ten seconds and voltage responses are recorded and compared to those of the three standards. As such, only a quantitative range can be ascribed to soil as samples and compared to the sample calibration procedures above.



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The Scan X Jr. can give only relative quantitative results based on the comparison of samples to the response found for the sampled at selected sampling periods. Therefore, only a greater than/less than concentration level is reported.

9.0 QUALITY ASSURANCE/QUALITY CONTROL

The analysis of nitroglycerine in soil gas by the Scan X Jr. portable GC is performed in-situ. Each soil gas station is analyzed only once as sampling typically voids all the organics from the hole. Sampling spikes and PE samples are not possible with this real-time in-situ sampling procedure. Periodically during the field analysis, the headspace from one of the standard vials is run on the Scan X Jr. to ensure that the unit is operating and responding within the calibration responses. If the unit is not responding correctly, the detector can be adjusted to yield voltage levels similar to levels obtained on previous standard calibrations run during the same sampling time periods.

10.0 DATA VALIDATION

The Scan X Jr., like any other GC, bases its compound identification on retention time indices at specific operating parameters. Positive identification via the Scan X Jr. must be confirmed by other analytical methods, such as GC/MS, to be absolutely sure that nitroglycerin is present. This must be done on soil samples taken within the same proximity as the soil gas samples. This in a sense would be mixing sample matrices, with the Scan X Jr. analyzing vapor phase samples and other methodologies analyzing solid phase soil samples. It would be possible to take Tenax tubes or Tedlar bags of soil gas for analysis by other methods. These would have to be from adjacent soil borings since the Scan X Jr. sampling pump will exhaust the organic vapors from the soil borings. This could result in erroneous results as none of the soil gas borings would be identical, despite close proximity.

A negative response from the Scan X Jr. provides a reliable indication that no Nitroglycerin is present; however, positive response yields only relative quantitative results, requiring independent confirmation for absolute identification.

11.0 HEALTH AND SAFETY

Nitroglycerine is toxic and explosive at high levels. Contact to skin and inhalation of vapors should be avoided. Nitroglycerin is light sensitive and should therefore be protected from direct sunlight. The levels of nitroglycerin used in the soil standards is far below any toxic and explosive levels; however, caution is advised when handling.

When handling hazardous materials, follow U.S. EPA, OSHA, and corporate health and safety practices.