SENTEX SCENTOGRAPH GAS CHROMATOGRAPH FIELD USE

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1.0 SCOPE AND APPLICATION

This standard operating procedure (SOP) deals primarily with the assessment of gaseous matrix samples. The Sentex Scentograph Gas Chromatograph (GC) can work in two detector configurations: the Electron Capture Detector (ECD) or the Argon Ionization Detector (AID). When the ECD is employed, volatile chlorinated compounds will be analyzed, as the ECD is very sensitive to electrophilic compounds such as chlorinated organics. The AID is a more universal detector, responding to most compounds with ionization potentials at or below 11.7eV. As such, it will respond to most aromatic compounds and many chlorinated compounds of environmental interest.

At present, only vapor phase samples (i.e., soil gas samples, Tedlar gas sampling bags, and ambient air samples) are being analyzed through the activation of the instrument's internal sampling pump. The Sentex GC unit does have a syringe injection port, but this is not being employed for any ERT applications at present. An optional purge and trap unit is available to determine purgeable organics in soil or water matrices. However, this SOP does not cover that capability.

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

The initial step in Sentex Scentograph Gas Chromatograph sampling is to boot up the Toshiba T 1100 computer. The computer runs the data acquisition program and stores all parameters and data. Begin by inserting the program "A" and data "B" disks into the appropriate upper and lower disk drives of the computer. Next, turn on the Sentex GC, and following the menu prompts, enter the GC parameters into the computer. Calibration analysis is then performed and stored. Calibration standards can be run either in the field, or in the laboratory prior to field sampling. In the latter case, a field calibration run must still be conducted to ensure that the lab calibrations are valid. A Tedlar bag containing a standards mixture can be attached to the Sentex GC upper inlet port at this point, or the internal calibration gas cylinder can be used.

Once calibration analysis is validated and stored, a Tedlar bag containing an unknown sample is attached to the Sentex GCs lower sampling inlet port, and the bag valve is opened. By selecting Function #3, a manual analysis can be run.

Once the sampling pump stops, the sample bag's valve is closed, and the entire bag is removed from the inlet port and stored for future laboratory analysis.

While this procedure is standard for all Sentex GC sampling, actual operating conditions (i.e., detector used, column packing material, oven temperature) will vary as required by the sample matrix encountered, and by the physical and chemical nature of the samples analyzed. Appendix A lists the operational parameters menus for entering and storing different GC conditions in the Sentex. New operating parameters are determined as new target compounds are selected for analysis.

3.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

The vast majority of Sentex applications are for soil gas analysis. These soil gas samples are collected and stored in dark plastic bags and placed in coolers to protect the bags from any damage that may occur in the field or in transit.

4.0 INTERFERENCES AND POTENTIAL PROBLEMS

Since the Sentex units employ gas chromatography, target compounds are identified by retention time indices (RTI). If the RTI of the sample peak(s) match the RTI of the standard peak(s), they are assumed to be identical. If any non-target compounds has the same RTI, it can be misidentified as a target compound. This problem occurs more frequently with the AID, since it will respond to any compound at or below 11.7eV. Often soil gas samples will have very high (ppm) levels of C_1 to C_6 hydrocarbons as well as low (ppb) levels of target compounds. The AID will respond to these hydrocarbons, whose signal often can "swamp" or obscure the signal of the lower level target compounds. In this case, it is better to use the ECD. If this is not possible, a different GC column (one able to separate the target compounds from the interference of the sample matrix) must be utilized. Moisture within the Tedlar bags will yield interference peaks that can obscure the resolution of the target compounds. It has not been determined whether it is the moisture itself, or contaminants in the moisture, that yield these contamination peaks, but the effect is seen on both the AID and the ECD. Typically, when water is found in the Tedlar bags the results of that bag's analysis is considered questionable. Typical ambient air relative humidity seems to have no appreciable effect on the signal response.

5.0 EQUIPMENT/APPARATUS

A Sentex model Scentograph GC can be configured for AID or ECD. Interfaced with the unit is a Toshiba model T 1100 lap top computer which runs the data acquisition program and stores all parameters and data. The Sentex unit has an internal battery pack which is charged from a Power Sonic Corp. model PSC-12400, (115 VAC to 12 VDC) charger. Attached to the T 1100 is a Hewlett-Packard model 2225P Think Jet printer. This produces hard copies of chromatograms and peak data information. No other equipment is required to operate the Sentex Scentograph unit.

6.0 REAGENTS

The Sentex AID requires ultra high purity (99.99% or above) Argon as carrier gas. The ECD can use either ultra high purity Argon or Helium (these can be ordered from Scott Gas, Matheson or any other reliable vendor). Gas standards are purchased as certified mixtures from Scott Gas or Matheson, at fairly high concentrations (i.e., 1-50 ppm). These concentrations are for subsequent dilution to various concentrations that enable construction of a standard calibration curve. If the internal calibration cylinder is to be used a low level standard, 0.5-1.0 ppm-v should be used.

If liquid phase reagents are required to make vapor phase standards, they must also be of the highest purity, such as Aldrich Gold Label or Supelco Environmental standards kits. If air is to be used for sample/standards dilutions, it must also be ultra high purity gas.

7.0 PROCEDURES

All operational parameters are entered via the T 1100 computer. This is accessed from the operations Menu, Function #1, which appears once the Sentex unit is turned on (Appendix A). Once the parameters are entered, a calibration run, Function #4, must be run. This is not a true calibration, since the Sentex calculates a pseudo-concentration against only one concentration. The calibration function is used only when operating parameters under Function #1 are changed. In all other cases, it is ignored. The run is typically aborted after the first "junk" or noise peak is identified in the calibration run. Occasionally, a Tedlar bag or the internal calibration gas cylinder can be sampled in the calibration mode but this would be for peak identification only and semi-quantification purposes, since it is only a single point calibration.

7.1 Calibration Procedures

The generation of calibration standard to be run in the field can be performed either in the field or in the laboratory prior to entering the field. If the latter is done, field standards must still be run to ensure calibration runs stored on the data disks are valid and close to standards run in the lab. Dilutions are typically made from the certified gas standards cylinders using Hamilton 500, 1000, and 1500cc model "Super syringes" and Tedlar sampling bags.

Simple volumetric dilutions are made and the set of standards are analyzed as if they were typical samples.

At least three concentrations of each standard must be run. It is preferable that more standards be run to establish the minimum ranges for the linear response of the Sentex detectors for each individual target compound. In the laboratory, a Multi-Channel Mass Flow Control can be used to meter selected flow rates from two to four separate compressed gas cylinders. A continuous flow of selected concentration of mixtures can be established to either fill Tedlar bags for analysis or to create a flow-through-cell from which the Sentex GC can sample. This has been used extensively to establish minimum and maximum detection levels in an efficient and timely manner.

The Sentex GC software does perform a single point calibration on up to 16 compounds contained in the internal calibration cylinder. This is adequate for most field screening needs. A linear calibration curve will be developed on a daily basis while analyzing in the field. Consequently, three concentrations of each standard, per target compound will be analyzed by the Sentex GC as if they were samples, and their GC response will be used to construct a linear regression curve.

7.2 Analytical Procedures

The following are stepwise procedures for analyzing vapor phase samples via the Scentograph Sentex GC:

1. Insert Disks "A" and "B" into upper and lower T 1100 disk drives, respectively, and turn on Sentex GC.
2. Follow menu prompts and input GC parameters as per Appendix A (Scentograph Operating Parameter Menu).
3. Select Function #4 to run and store calibration analysis. A Tedlar bag of a standards mixture may be attached to lower inlet.
4. Attach Tedlar bag with unknown sample to the lower sample inlet port and slide the bag valve down to open.
5. Select Function #3 to run a manual analysis, at prompt enter sample name, press "ENTER" a second time to inject.
6. Immediately after sampling pump stops, pull bag valve out to close, remove bag from inlet port.
7. Sample and calibration analysis can be aborted by holding down the reset key on the GC panel until "RETURN" prompt appears.
8. Any changes in operating parameters entered in Function #1 must be followed by a calibration run prior to an analysis run.

8.0 CALCULATIONS

A calibration curve of at least three concentrations must be constructed for each target compound. A straight line equation in the form of $y = (m)(x) + b$; (where: x = concentration, y = area counts, m = slope and b = the intercept) is fit to the standards raw data. The (y), or the unknown concentration for the sample, is determined from the above straight line equation. Non-linear data is indicative of erroneous detector response, alternatively sample concentration can be calculated as below:

$$\textit{Sample Conc.} = \frac{(\underline{\text{standard conc.}}) \times (\underline{\text{sample area}})}{(\text{standard area})}$$

The Sentex does perform a one point calibration for compounds present in the internal calibration cylinder that were entered in the library. If the samples and library standard are in the linear range, this one point calibration is considered valid for field screening purposes.

9.0 QUALITY ASSURANCE/ QUALITY CONTROL

The following QA/QC protocols are applicable:

1. A complete calibration curve must be run daily.
2. Replicates of a gas standard, in the mid-range of the calibration curve and preferably close to sample results, should be run every 10 samples to ensure detector response is constant.
3. Two or three replicates for each sample and

standard should be run. In terms of area count and retention time values, these replicate responses should be within 10-20% of each other.

4. Matrix spikes, or spiking samples with known levels of standards, are not typically required as the same Tedlar bag may be analyzed by other field instrumentation (i.e., Photovac, organic vapor analyzer [OVA], etc.) and/or collected onto traps for GC/MS confirmation. If Tedlar bags are used to prepare standards, the time of preparation should be noted.

5. During sample analysis, one of the standards should be periodically re-analyzed to ascertain if any sample loss occurs in the bag over time since many compounds will degrade or permeate Tedlar sample bags.

6. A performance evaluation sample (PE) is typically sent along with the sampling efforts to determine if any loss or contamination occurs from transit or handling during sampling.

7. A trip blank, consisting of a Tedlar bag filled with zero air, is also sent along and is analyzed at the end of the sampling run to determine if any contamination of the Tedlar bags occurred during transit.

10.0 DATA VALIDATION

As mentioned previously, peak identification is by Retention Time Index (RTI). Sample spikes, using known levels of target compounds, can be prepared to identify the absence/presence of target compounds in the samples if peaks are eluting close to the target compounds. Typically, only the RTI is needed to identify the peaks of interest. Quantification is determined from the linear calibration curve, and solving for concentration (y) from the straight line equation. The coefficient of variation on the straight line equation should have an R squared, (R^2), of 0.95 or better. The confirmation of the identity of any particular target compound must be done by other analytical methods, typically GC/MS. Standards must be run along with the samples, and should bracket the levels found in the field samples.

Alternatively, a statistical approach to data validation can be sought. Once the linear range is established an appropriate standard, either a low or midrange concentration, will be analyzed 10 or more times throughout the day. The standard deviation of the mean [F(n-1)] for the response of the standard selected is determined. The statistical method detection limit (MDL) will be three times the standard deviation (3F). The method quantitation limit (MQL) will then be 10 times the standard deviation (10F). Results below the MDL will be considered nondetects (ND). Results above the MDL but below the MQL will be considered detected but below the quantitation limit and will be ascribed a "J" value. This "J" value will flag the data to let the user know the results are questionable. Results above the MQL are considered statistically reliable data.

11.0 HEALTH AND SAFETY

When working with potentially hazardous materials follow U.S. EPA, OSHA and corporate health and safety guidelines. More specifically, analysis should be performed in a well ventilated room. When liquid reagents are used to prepare standards etc., disposable protective gloves should be worn, and work should be performed under a vented hood.

12.0 REFERENCES

This section is not applicable to this SOP.

APPENDIX A

Scentograph Main Menus

Operation Menu

Scentograph **	Operating Parameters	1
Scentograph **	Automatic Operation	2
Scentograph **	Manual Operation	3
Scentograph **	Calibration Analysis	4
Scentograph **	Operation Summary	5
Scentograph **	Utility Menu	6
	Enter Function	?

APPENDIX A (con't)

Example of Typical Scentograph Operating Parameter Menu

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** Scentograph                            Operating Parameters

1  - Calibration Sample Name         (enter up to 8 letters)             ... REAC
2  - Sample Time                         (1-300 sec)                     ... 15
3  - Delay Time                         (0.1-4.0 sec)                    ... 1.0
4  - Desorption Time                    (0.1-4.0 sec)                    ... 4.0
5  - Inhibit Time                       (10-999 sec)                     ... 50
6  - Oven Temperature                    (30-140ºC)                      ... 50
7  - Chart Duration                  (1,3,5,10,15,20,30 min)             ... 30
8  - Analysis per Calibration              (1-99)                        ... 99
9  - Auto Analysis Duration              (0-120 min)                     ... Manual
10 - Backflush Option                   (0=off, 1=on)                    ... BCKFL OFF
11 - Detector                        (1-AID, 2-ECD, 3-TCD, F-PID)        ... A.I.D.
12 - Column                          (enter up to 8 letters)             ... 6'10%CP5
13 - Column Pressure                       (5-40)                        ... 20
14 - # of Calibration Peaks                (1-16)                        ... 1
15 - Peak Number 1
          Substance Name              (enter up to 8 letters)            ... U1/AID
          Concentration Range            (PPM=0, PPB=1)                  ... PPM
          Calibration Conc             (99.9 ppm, 9999 ppb)              ... 1.00
          Peak alarm values          (0-99.99 ppm, 9999 ppb)             ... 99
16 - Upload Scentograph parameters

Enter field to be UPDATED?
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