

Ensuring Data Integrity

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NEWMOA Vapor Intrusion Seminar

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- Measurement of VOCs & non-VOCs
 - Soil Gas, Indoor and Ambient Air
 - Collection and analytical techniques
 - TO-15
 - SVOC methods
 - Mobile Laboratories (8260B)
 - Passive Technology

- Data Quality Objectives
 - Reporting limits
 - Method QA/QC
 - Ensuring legally defensible data

OSWER's Draft Guidance Analyte List

- 114 compounds in Table 2 of OSWER Guidance
 - VOCs, SVOCs, PCBs, Metals
- TO-15 is only applicable for VOCs
- Sorbent collection/analysis methods necessary for all non-VOCs
 - Sorbent types (PUF/XAD)
 - Sampling rates/volumes
 - Breakthrough

Questions that need to be Addressed

1. Target Analyte Lists
 - ➔ Determined by your contaminants of concern

2. Detection Limits
 - ➔ Determined by RBCs from state agency

3. Methodology
 - ➔ Determined by answers to 1 & 2

4. Canister Certification (TO-15)
 - ➔ Batch
 - ➔ Individual

5. Laboratory Certification
 - ➔ Required or available

Data Quality Objectives

- Analyte list:
 - May vary at every site;
 - Discuss your target list with the lab
- LOWER RLs DO NOT EQUAL BETTER QUALITY!
 - RLs need to meet your site's screening criteria and do not have to be the lowest RLs possible;
 - E.g. Using TO-15 SIM for soil gas samples
- RLs (Reporting Limits): Lowest standard of the instrument's initial calibration- also referred to as PQLs or DLs
 - MDLs (Method Detection Limits): Statistical number that is derived from 7 replicate analyses of a standard; concentration analyzed is targeted to be ~5 times that of the suspected MDL

Data Quality Objectives

continued

- RLS continued:
 - May not always be able to achieve method reporting limits
 - Instrument calibration ranges:
 - E.g. 0.2 ppbv to 40 ppbv
 - If you have an analyte concentration above the calibrated range, a sample dilution needs to be performed
 - The sample dilution will bring the higher analyte's concentration into the calibrated range, but other analytes may end up being ND above the RLS
- Methodology:
 - Dependent upon your analyte list and RLS

Data Quality Objectives

continued

- QA/QC requirements differ between the methodologies that are available
- Need to consider the intended use of the methods as written before making decisions as to if the QA/QC requirements are stringent enough
 - 8260B & TO-15 were written for different matrices
 - Calibration acceptance & recoveries of internal standards are built around these matrices
- Sampling Media Certification
- Laboratory Certification

Tracer Testing



- Add 10% to 50% helium to shroud
- Fill Tedlar bag via Lung Box and screen for helium reading
- Quantitative proof of sample integrity

Source: T. McAlary,
GeoSyntec

Unfortunately, we all cannot
have a TAGA bus...



Trace Atmospheric Gas Analyzer Mobile Laboratory

Source: D. Mickunas,
EPA

Field Screening



PID or FID



O₂/CO₂/CH₄/H₂S



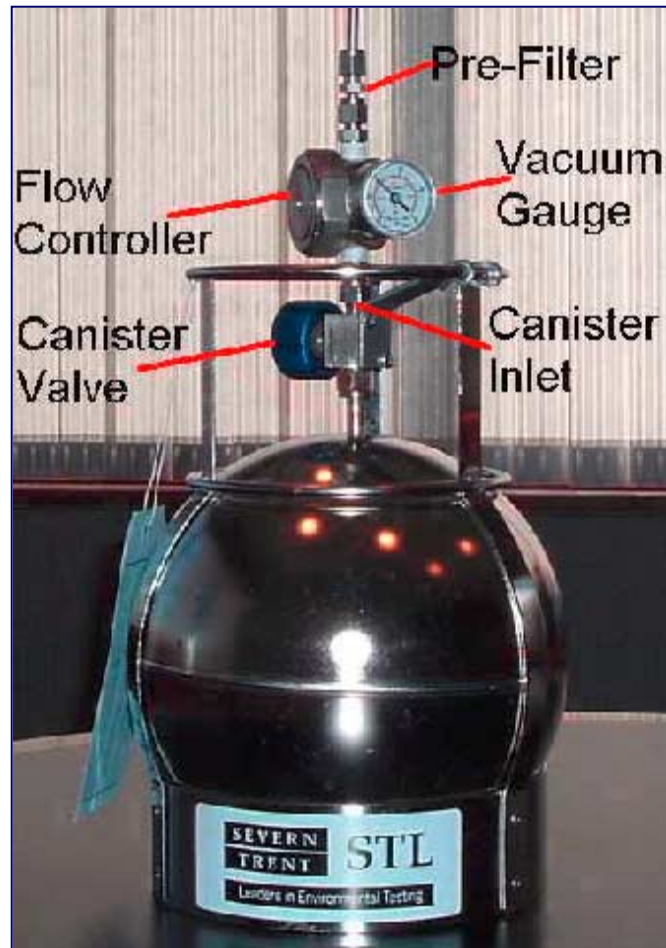
Helium detector



Optional compound specific analysis (Draeger chips)

Source: T. McAlary,
GeoSyntec

VOC Sample Collection



Indoor Air Sampling

- 1) Pre-sample walk-through & building survey,
- 2) Remember to collect an Ambient Air sample,
- 3) Sample at appropriate human risk exposure locations- breathing zone, good circulation, high use areas,
- 4) Select a certified & experienced laboratory.

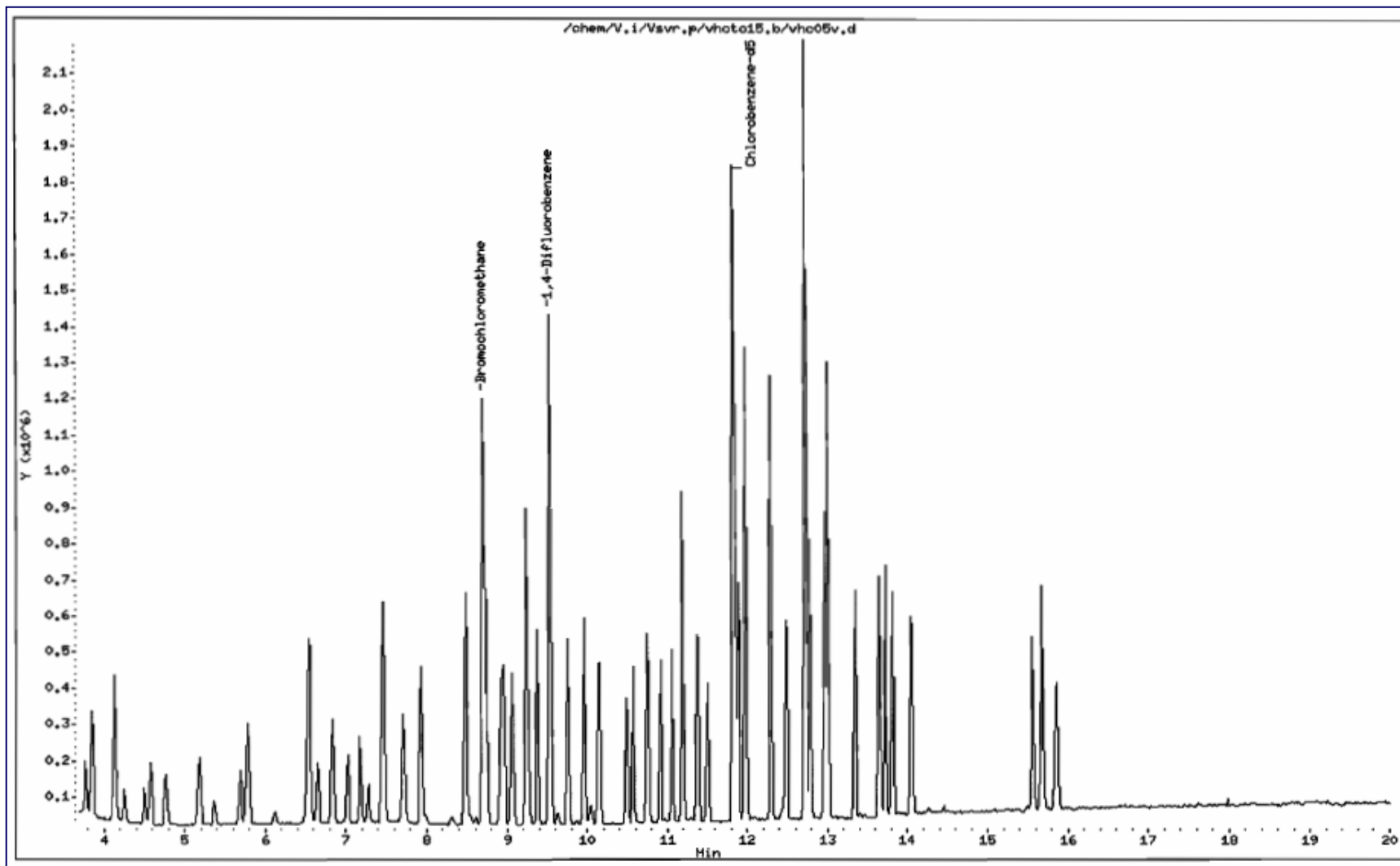
- Sampling & Analysis for subsets of the 97 hazardous VOCs listed in Title III of the 1990 Clean Air Act.
- Reporting limits :
 - Robust method if followed properly
 - Full scan: 0.2 to 0.5 ppbv ($\sim 1 \text{ ug/m}^3$)
 - SIM: 0.002 to 0.010 ppbv (0.011 to 0.02 ug/m^3)
- Samples collected in canisters
 - Holding times: VOCs stable for up to 30 days
 - Shipping procedures
- Field QA-
 - Trip or Field blanks; what do they really give you?
- Lab QA:
 - Calibration standards:
 - NIST traceable gaseous standards
 - Second Source standards

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 - Holding times: VOCs stable for up to 30 days
 - Shipping procedures
- Time integrated vs. grab samples

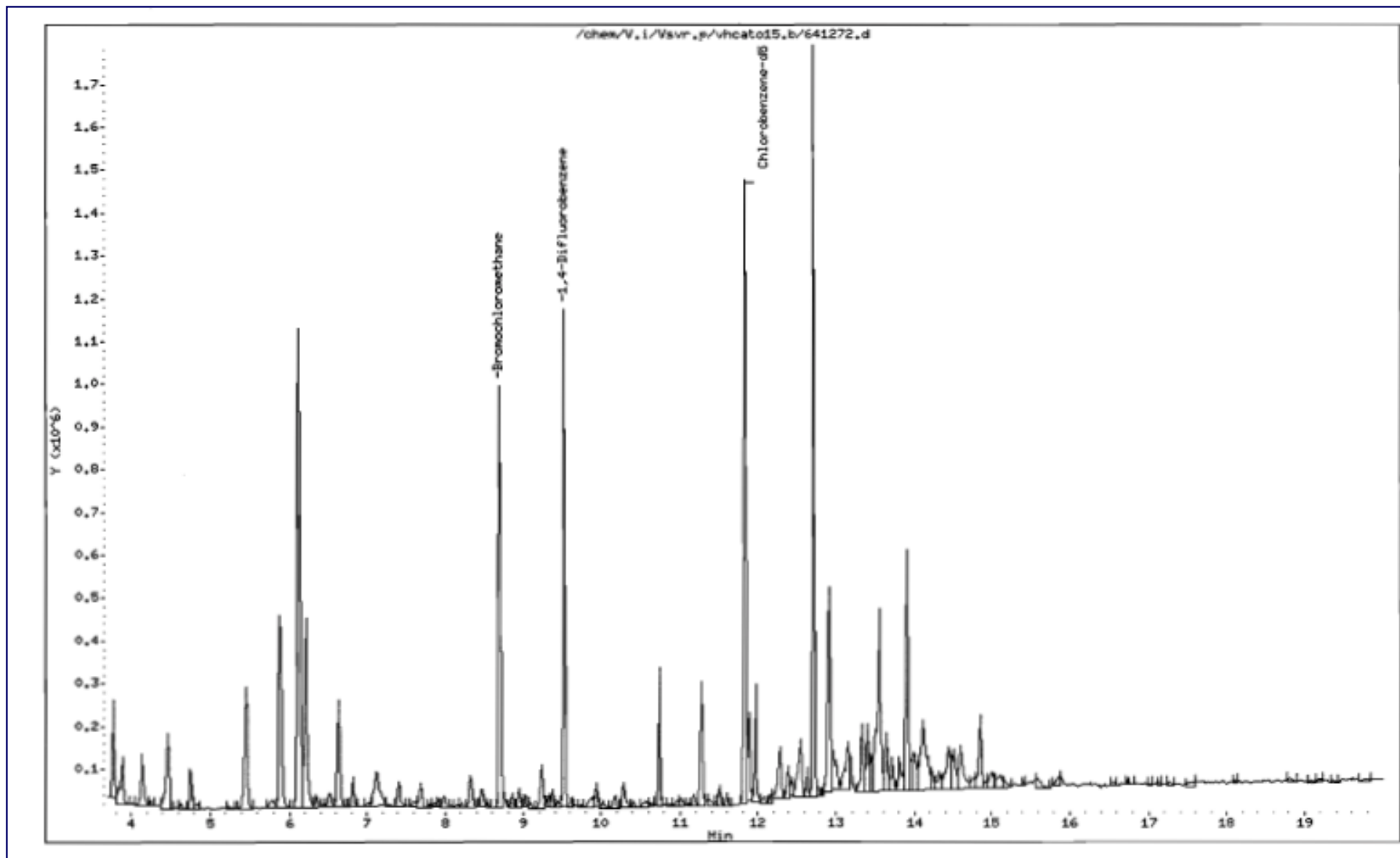
TO-15 Analytical System



TO-15 Chromatogram (5 ppbv Standard)



Sample from Basement



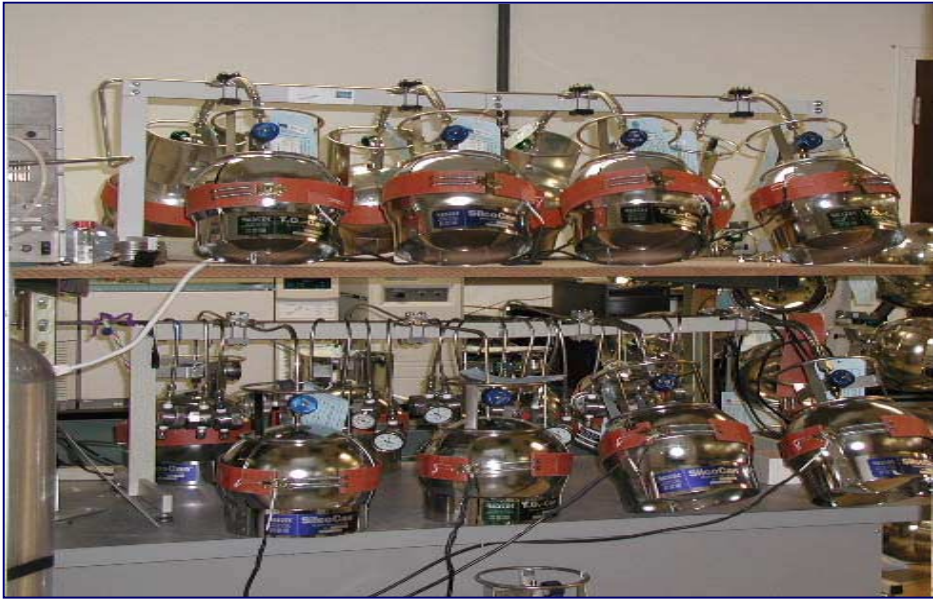
Interpreting Air Data

- Remember:
1 ppb TCE does not = 1 ppbv TCE
1 ppb (ug/L) = 180 ppbv
- Formulas are chemical specific:
$$\text{ppbv} = (\text{ug/m}^3 \times 24.45) / \text{MW}$$

Canister Certification & Management

- All canisters should have a unique ID and tracked from day one
- Canisters should be segregated
 - Low level (ambient & indoor)
 - Source level (soil gas)
- Batch certification
 - How canisters are cleaned
 - Evacuated, heated, pressurized w/humidified zero air
 - Certification requirements
 - Canister that had sample w/highest levels of VOCs analyzed
 - No detections above RLs
 - Leak free
- Individual certification
 - Each canister is analyzed after being cleaned

Canister Cleaning Systems



TO-15 In Review

Advantages

- Sensitivity allows for RLs down to the 10^{-6} risk range.
- Robust enough to handle high ppmv level samples.
- Media is certified clean before use.

Disadvantages

- May be difficult to collect soil gas samples from tight soils.
- May be difficult to transport canisters around site for large sampling event.

TO-17 Analysis of VOCs in Ambient Air

- ◆ Samples collected on a sorbent tube with use of pump
- ◆ Collect duplicate samples per sampling point
- ◆ Analyzed by GC/MS
- ◆ Holding time is 14 days from collection
- ◆ Must be chilled <4 C

SVOCs Sample Collection & Analysis

- TO-13A:

- PAHs and many other 8270 compounds;

- TO-4A:

- Pesticides & PCBs (including congeners);

- PUF/XAD media for sample collection

- Sample volumes needed to meet screening criteria may not be practical for many screening levels

- Modified versions: low flow pump

SVOC Sample Collection



High volume sampler



PUF/XAD
"sandwich"

Soil gas
sampling



- Can be a useful, powerful tool;
- 8260B modified, commonly used;
- Analytical instrumentation is consistent to TO-15: GC/MS;
- 8260B calibration, standards and sample introduction differ significantly vs. TO-15
 - Liquid phase standards
 - Sample concentration not as effective with removing moisture
- Syringes, Tedlar bags and glass vials commonly used for modified 8260B are not certified clean to RLs;
- Vapor and Air analysis by modified 8260B may lead to bias low results.



Mobile Laboratories In Review

Advantages

- Near-real time data
- Ability to adjust sampling plan as data becomes available
- Can identify problems with sample integrity and correct in the field

Disadvantages

- May not be able to achieve the low detection limits necessary
- Data often are not acceptable for human health risk assessment or for litigation support
- Sample collection media is not certified clean

Passive Sampling

- Published literature, methods for passive collection of indoor air
 - Including calculating concentration
- Current and Future work
 - EPA Detroit Exposure & Aerosol Research Study (DEARS)
 - Perkin Elmer diffusion tubes
 - 25 VOCs: ambient and indoor air samples
 - Extension of TO-17
 - Research being conducted by technology vendors for calculating soil gas concentration
- Examples:
 - GORE™ Module
 - EMFLUX®
 - SKC Ultra I and Ultra II Badges
 - Perkin Elmer Passive Diffusion Tubes

Legally defensible data

Data Validation is a critical step:



- ✓ DQOs met?
- ✓ Sample media certified appropriately?
- ✓ Technically sound, proven analytical method used?
- ✓ Laboratory certified by NELAC? and state (if applicable)?
- ✓ Analytical QA/QC met?
 - ✓ Calibration, 2nd Source Standards, Method Blanks, etc.
 - ✓ Samples analyzed within holding time?
 - ✓ If no, was data flagged appropriately?

- ✓ Multiple tools for assessing VI pathway
- ✓ Each technique offers different advantages and disadvantages
- ✓ Analyte Lists & Reporting Limits vary
- ✓ Air sampling media needs special preparation
- ✓ Developing & meeting DQOs is critical
- ✓ Communication is key

Questions or Comments?

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