Introduction to IH Analytical Chemistry

Sampling and Analysis

AIHA®
Protecting Worker Health

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(Apr 2013)

Foreword

Thank you for purchasing the Introduction to IH Analytical Chemistry self-study workbook.

Course Format

The workbook is divided into 9 chapters, each with review questions. Read through each of these chapters and take the time to answer the review questions at the end. You can type your answers in the document. Answers to the review questions are included at the back of the workbook so you can check your work.

Final Exam & Evaluation

Please submit for credit when you have reviewed the materials and feel ready to complete the final exam. A brief evaluation is included as part of the credit submission process. Once you have successfully passed the final exam and submitted for credit, your AIHA transcript will be updated.

Scoring

Final Exam scoring is as follows:

- 90-100% Excellent
- 80-89% Good
- 70-79% Fair
- <70% Exam must be retaken

Retaking the Final

If you fail to score 70% on the second test, you will not be awarded credit.

Course Credit

The course will award 6.0 contact hours of credit on the AIHA Transcript.

Completion Time

You have 3 months from the date of purchase to complete the course and exam. If more time is needed please contact the AIHA eLearning Department Staff at dlassistant@aiha.org.

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1. DEFINITIONS

Air Sample — The contents of a sampler which has been used in an air sampling event

American Conference of Governmental Industrial Hygienists (ACGIH®) — A private organization that has established guidelines and Occupational Exposure Limits to promote chemical safety.

Analyte or Contaminant — The chemical substance that is being sampled and analyzed.

Certified Industrial Hygienist (CIH) — An IH practitioner certified by the American Board of Industrial Hygiene (ABIH) based on meeting work experience requirements, and passing a comprehensive examination.

IH Practitioner — An industrial (occupational) hygienist who identifies, measures, and controls exposures in a workplace, and conducts exposure assessments, including conducting air sampling events.

IH — Industrial hygiene or occupational hygiene, a profession that identifies, measures, and controls exposures in a workplace.

IH Lab — An AIHA®-accredited Industrial Hygiene Laboratory (AIHA Registry Programs LLC) that analyzes Samples

National Institute for Occupational Safety and Health (NIOSH) — An agency of the US government (in the Department of Health and Human Services) charged with doing research and making recommendations in support of the Occupational Safety and Health Act of 1970. NIOSH publishes the NMAM (NIOSH Manual of Analytical Methods) which describes non-binding OELs and sampling methods used by NIOSH in doing their research studies in occupational exposure.

Occupational Exposure Limit (OEL) — A generic name for any exposure limit established by any organization to control TWA exposures to workers.

Occupational Safety and Health Administration (OSHA) — An agency of the U.S. government charged with enforcing the Occupational Safety and Health Act of 1970. OSHA establishes PELs (below) that are legally binding regulations under the OSH Act. OSHA also publishes OSHA Sampling and Analytical Methods (OSAM) that it uses for sampling events in enforcement inspections. OSAM are recommended, but are not legally binding (as the agency desires to support the development of new and improved methods that may be more suitable for the private sector.)

5. CHARACTERISTICS of PERSONAL SAMPLERS

Sampling Tubes

Used for sampling gases and vapors, sampling tubes are glass ampoules usually containing two sections of loosely packed sorbent particles separated by an inert spacer. Sorbents for different sampling tubes are selected based on chemical affinity for analytes to be sampled. To begin sampling, each tube is broken at both ends, then attached via tubing to a sampling pump which draws air though the tube at a metered sampling rate (ca. 100 mL/min).

The open end of the sampling tube is attached near the worker's lapel, while a sampling pump is attached at the worker's belt. Air passes through the larger (front) sorbent section first, and, then, subsequently, through the smaller (back-up) section of the tube. The two sorbent sections are analyzed separately by the lab. The finding of a significant amount of analyte in the back section of sorbent indicates "breakthrough" of analyte through the sorbent and suggests an invalid sample. Different organizations may have different guidelines as to how much back-section analyte (as % of total sample) is grounds for rejecting a sample. (As a rule of thumb, 10% of analyte in the back-section suggests at least 1% has been lost from the back section, while 20% in the back-section suggests at least 4% has been lost.)

Sampling Cassettes

Sampling cassettes are used in sampling of aerosols (solid or liquid particles) as they retain analytes by mechanical entrapment rather than by chemical affinity. Like sampling tubes, cassettes are used in conjunction with a 1-lb sampling pump that draws air through tubing connected to the cassette (typical sampling rates 1–2 L/min). Each cassette contains a porous filter disc that is matched to the size of the particles to be collected. For analysis, the cassette is disassembled and the filter disc removed for examination and extraction. In some cases, the entire cassette is extracted to remove particles adhered to the cassette body. 2 piece cassettes are used for closed face sampling methods. 3 piece cassettes are used when "open face" sampling is required.

Some so-called "treated" sampling cassettes contain filters treated with chemical reagents which form a stable derivative when exposed to a reactive vapor or aerosol. It is important to recognize that "treated cassettes" are able to sample for vapors or aerosols that are reactive with the treating reagent, while "un-treated cassettes" are unable to retain gases or vapors at all.

6. PERSONAL SAMPLING – PARAMETERS and CALCULATIONS

Calculating Time-Weighted Average (TWA) Concentrations

In normal IH practice, air samples are collected in the field and the sampler is shipped to a remote IH lab that analyzes the sampler. In so doing, the IH lab extracts and measures the quantity (mass) of analyte found in the sampler.

The average air concentration of chemical present in the worker's breathing zone can be determined as the amount of analyte found in the sampler divided by the volume of air sampled, as follows.

TWA Conc'n = [**Amount Found** (*µg*)] / [**Sample Volume** (*L*)]

Sample Volume

The effective volume of air sampled during an air sampling event is equal to the sampling time (typ., *minutes*) multiplied by the sampling rate (typ., *mL/min*). Assuming typical units are employed the calculation of sample volume is as follows.

[**Sampling Time** (min)] X [**Sampling Rate** (*mL/min*)] = **Sample Volume** (*mL*)
[**Sample Volume** (*mL*)] X [*L/1000 mL*] = **Sample Volume** (*L*)

Different time units (*seconds, minutes, hours*), Rate (*mL/min, L/hr,* etc.), and volume (*mL, L,* M^3) may also be used, as long as the units of time and rate are properly matched for the calculation. When sampling with badges it is also important for the IH to record the temperature and uncorrected barometric pressure or elevation of the worksite. *It is important to note that the sample volume has as much impact on the calculated result as the mass of analyte found by the lab. Thus, any error in measuring the sample volume is as costly as an error in lab analysis.*

Sampling Rate and Sample Volume

Sampling devices used by the IH in the field are characterized by their sampling rate (*mL/min*). Some sampling devices (sampling pumps) can be adjusted in the field to a desired sampling rate, while others (personal monitoring badges) have a fixed sampling rate for each chemical sampled. The sampling rate for a particular analyte on a particular badge is obtained from its manufacturer.

8. LAB ANALYSIS – TECHNIQUES

Some of the fundamentals of the analytical methods most commonly employed in industrial hygiene analysis are included in the following section.

Gas Chromatography (GC)

The most popular single technique in industrial hygiene analysis, gas chromatography is specified for the analysis of more than 150 analytes in OSHA methods, the NIOSH Manual, and in the standard test methods of many accredited industrial hygiene labs. In gas chromatography, a sample (usually a solvent containing analytes) is injected into a flowing gas stream (helium or hydrogen) which immediately passes through a heated column containing a polymer material (or sorbent). During chromatography, each analyte tends to travel through the column at a slightly different rate (based on its affinity for the column material) emerging from the column at a different time (the retention time). In gas chromatography, the column is often programmed to increase in temperature at a pre-determined rate, taking advantage of the fact that each analyte's affinity for the column is altered dramatically as its boiling point in approached. In so-called temperature programmed GC, analytes travel very slowly down the column at temperatures below their boiling point, and begin to travel very rapidly when their boiling point is approached. Scanning the column temperature in this way provides a method for analyzing a large number of analytes in a small amount of time.

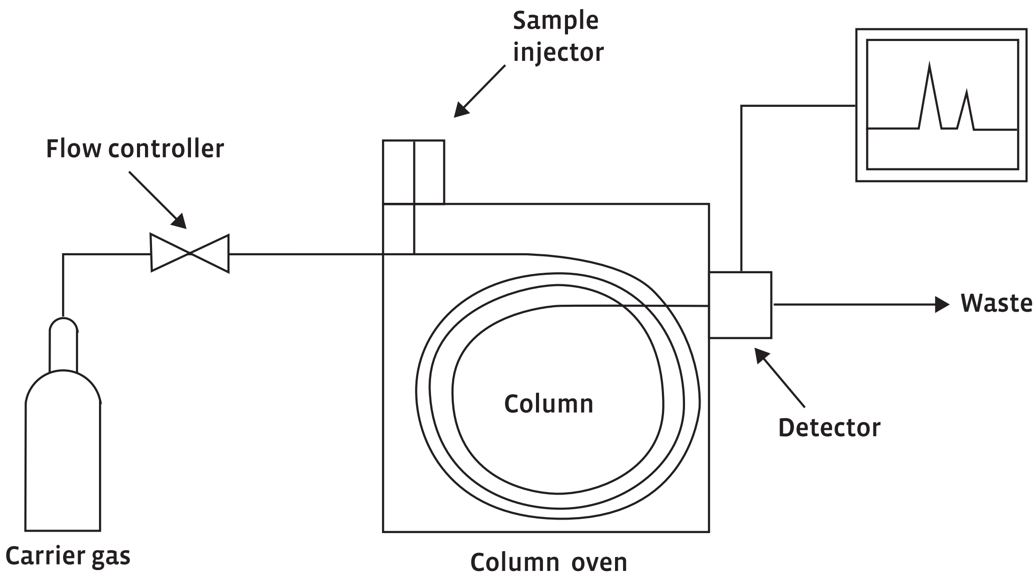

FIGURE 1.
A schematic of a gas chromatograph

REVIEW QUESTIONS

Section 8. Lab Analysis – Techniques

1. What is the most commonly used Lab analysis method for organic vapors? Why?

2. Analytes are separated in gas chromatography based on which of the following physical properties?

- (a) Affinity for the GC column stationary phase
- (b) Number of carbon atoms
- (c) Heat of vaporization
- (d) Volatility (vapor pressure & boiling point)
- (e) Specific gravity

3. What is the most commonly used lab analysis method for metal dust? Why?

4. What class of compounds is best suited for HPLC?

5. The detector commonly used for gas chromatographic analysis of carbon containing analytes is:

- (a) Electron capture detector
- (b) Flame ionization detector
- (c) Electron capture detector
- (d) Flame photometric detector

Answer Key for Questions

Section 1. Definitions:

1. **What is an IH practitioner? What is a CIH?**

   ANSWER:
   An IH is anyone who practices industrial hygiene, regardless of credentials. A CIH is a "Certified Industrial Hygienist," i.e., one whose professional standing has been recognized by the American Board of Industrial Hygiene (ABIH), based on a review of qualifications, passing of a comprehensive exam, and meeting maintenance requirements.

2. **What is the difference between a STEL and a Full-Shift PEL?**

   ANSWER:
   A STEL is a PEL for short-term exposure, a 15-min TWA. A full-shift PEL is a PEL for the duration of the work shift, usually a 8-hr TWA.

3. **Which of the following Occupational Exposure Limits are legally enforceable?**
   (a) OSHA PEL (b) ACGIH® TLV® (c) NIOSH REL

   ANSWER:
   (a) OSHA PEL

4. **Which of the following performs accreditation of industrial hygiene laboratories?**
   (a) NIOSH (b) OSHA (c) AIHA® (d) EPA

   ANSWER:
   (c) AIHA®

5. **What organization enforces the Occupational Safety and Health Act of 1970?**

   ANSWER:
   OSHA

6. **Which of the following organizations performs certification of industrial hygienists?**
   (a) NIOSH (b) OSHA (c) ABIH (d) ACGIH®

   ANSWER:
   The American Board of Industrial Hygiene (ABIH).