OSHA COMPLIANCE

IN THE

ACADEMIC ORGANIC CHEMISTRY LABORATORY

by

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A dissertation presented to the Graduate Faculty of Middle Tennessee State University in partial fulfillment of the requirements for the degree, Doctor-of-Arts

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APPROVED:

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ABSTRACT

OSHA COMPLIANCE IN THE ACADEMIC ORGANIC CHEMISTRY LABORATORY

by Marlyn Cupell Newhouse

The Federal Regulation, 29 CFR 1910.1450, "The Occupational Exposures to Hazardous Chemicals in Laboratories", applies to school science laboratories where chemicals are found. This may be a new finding to those who thought Occupational Safety and Health Act (OSHA) regulations only apply to industries and manufacturing.

This study documents several steps taken to bring a university chemistry laboratory into compliance with 29 CFR 1910.1450: (1) the process of changing an academic organic chemistry instructional program to microscale, (2) the incorporation of a Chemical Hygiene Plan (CHP), (3) the determination of hood velocities, and (4) using instrumental analysis to monitor chemically the tasks a student worker would do. Overhead transparency masters are included in the appendices for use in preparing training presentations.

The measurement of the air quality and hood velocities was accomplished using standardized procedures outlined in 29 CFR 1910.1450. Air sample collection was accomplished using personal air pump with charcoal filter tube attached

Marlyn Cupell Newhouse to the worker's collar. While grab sampling with evacuated canisters is usually conducted in EPA air quality studies, the air pump procedure coincides with OSHA studies. Gas chromatography was used to analyze the air samples. The target chemicals were benzene and toluene. The data collected was compared to the American Conference of Governmental and Industrial Hygienists (ACGIH) threshold limit values (TLVs), the Occupational Safety and Health Act permissible exposure limits (PEL), and the National Institute for Occupational Safety and Health (NIOSH) recommended exposure limits (RELs). Air Sample #1 was the only sample to have a contaminant, benzene, near the legal limits.

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CHAPTER 1

HAZARDS AND LAWS

Introduction

Recent federal legislation has included schools under the jurisdiction of the Occupational Safety and Health Administration (OSHA). Prior to this time, academia was not concerned with OSHA laws because, for the most part, these laws pertained to industry and manufacturing--not the "business" of education (1,2).

It has always been an intention of Congress for school laboratories eventually to be included under OSHA laws because of the hazards that exist in them (3). These hazards were first graphically reported in the early 1970's. Articles described accidents and cases where chemicals (picric acid and ether, especially) had been stored so long in high school and college laboratories that it was a danger even to touch them (4,5)!

At first glance, colleges might not seem to be big producers of hazardous waste, at least in comparison to manufacturing and industrial companies. And they're not, in terms of volume. But their laboratories, grounds departments, and art studios all generate hundreds of tons of waste a year all of which must be handled and disposed of in a safe manner.

Common wastes produced by colleges include solvents, which are used in research; pesticides, which are used by grounds departments and in agricultural programs; and metals in glazes used by art departments. All of those substances are considered hazardous and are regulated under federal law (6).

Since the mid-1970's, a string of safety and health and environmental legislation

regulating the use and disposal of hazardous materials has been enacted by all levels of government. Until recently, ... federal, state, and local government agencies have pretty much ignored the use of these materials by colleges and universities ... the amount of hazardous materials used by America's institutions of higher learning represented only a fraction of the volume used by American industry. The responsible enforcement agencies adopted a "Bigger Fish to Fry" philosophy, and many institutions ... could choose to ignore or only partially address the compliance requirements of these laws without fear of penalty. This is no longer true.

Due to the fact that relatively small amounts of hazardous materials have caused significant damage to both human life and the environment, existing laws have been modified incorporate smaller to volume users. Reporting requirements for these laws have also become more stringent and interrelated. For example, one EPA-enforced federal standard requires hazardous material users to provide their local governments with an inventory of chemicals located at their facilities. This means an inspector from your local fire department can find your campus in violation of EPA regulations (7).

Importance of the Study

Most of the recent literature is so technical with regard to air quality or so broad in scope that it is not specific enough to apply to academic laboratories. The purpose of this work was: (a) to acquaint school officials and science teachers with the particulars of the law that affect academia directly, (b) to offer a suggested process or procedure to achieve compliance with as little disruption to the normal school routines as possible, (c) to report the specific data and results of this study of air sampling and hood ventilation in a typical organic chemistry laboratory, and (d) to provide materials that can be used to train

instructors, graduate teaching assistants, student workers, and staff in chemical safety on the job.

Limitations

The law, 29 CFR 1910.1450, is an OSHA regulation "to protect the employee from health hazards associated with hazardous chemicals in the laboratory" (8). The Laboratory Standard and its Chemical Hygiene Plan requirement is a major topic of this study. The setting under consideration is an academic organic chemistry laboratory at the university level. The chemicals focused upon are certain volatile organics identified in "Subpart Z of 29 CFR 1910.1000- Air Contaminants". Inorganic chemicals, concentrated acids and cryogenic gases are not included. Clinical and industrial laboratories have their own specific problems which are not addressed in this study. Pathological and radiological hazards are regulated by other, more specific laws. Certain hazardous chemicals, such as formaldehyde, are also regulated in other laws. Definitions of Terms

In the realm of safety literature, a number of terms are commonly referred to by acronyms. Following are some of the most important acronyms and related terms (9).

<u>ACGIH</u> - American Conference of Governmental Industrial Hygienists publishes TLVs for chemical and physical agents. The Conference is not an official Government Agency. Membership is limited to professional personnel in government agencies or educational institutions engaged in occupational safety and health programs.

ACS - American Chemical Society

<u>ANSI</u> - The American National Standards Institute publishes consensus standards of acceptable concentrations for chemical and physical agents.

<u>Ceiling</u> - A ceiling limit is given to a substance that is relatively fast acting. It is measured by one or more brief samples, usually 15 minutes in duration. This limit should not be exceeded.

CFR - Code of Federal Regulations

<u>CHP</u> - Chemical Hygiene Plan

DOT - Department of Transportation

<u>EPA</u> - Environmental Protection Act (or Agency)

<u>Excursions</u> - Short exposures above a time-weighted average level. These must be compensated by levels below the TWA.

FID - Flame Ionization Detector

GC - Gas Chromatogram or Chromatography

<u>MSDS</u> - Material Safety Data Sheets - an information sheet provided by the chemical manufacturer.

<u>NFPA</u> - National Fire Prevention Association

<u>NIOSH</u> - National Institute for Occupational Safety and Health.

OSHA - Occupational Safety and Health Act(or Administration)

<u>PEL</u> - Permissible Exposure Limits are legally allowed concentrations in the workplace.

<u>REL</u> - Recommended Exposure Limit

SOP's - Standard Operating Procedures

<u>TSD</u> - Treatment, Storage or Disposal facility

<u>TLV</u> - Threshold Limit Values- Refers to airborne concentrations of substances and represents conditions under which it is believed that nearly all workers may be repeatedly exposed, day after day, without adverse effect.

<u>TOSHA</u> - Tennessee Occupational Safety and Health Administration.

<u>TWA</u> - Time-weighted averages represent airborne concentrations averaged with regard to their duration.

Organization of the Study

Chapter 1 is an introduction to the hazards encountered in a chemistry laboratory and the laws that govern those hazards. Chapter 2 is a review of related literature and studies that others have done. Chapter 3 is a brief summary of the laboratory standard, 29 CFR 1910.1450. Chapter 4 is a report of what was done to bring an academic laboratory into compliance. Chapter 5 describes the methodology for the air quality study. Chapter 6 shows the data and describes the results. Chapter 7 recommends how academia can monitor its own activities and states the conclusion of the study.

CHAPTER 2

REVIEW OF RELATED LITERATURE

In any Occupational Safety and Health Administration (OSHA) training seminar, hazards are categorized as "Health Hazards" or "Physical Hazards". For the purpose of training school employees, whether they be from the janitorial staff or student workers, these same categories should be used (3).

CHEMICAL HAZARDS

HEALTH HAZARDS (Illness)

6 Classes:

Corrosive Irritant Sensitizer Carcinogen Acute Toxicity Organ Damage PHYSICAL HAZARDS (Injury) 9 Classes: Combustible Liquid Compressed Gas Explosive Flammable Organic Peroxides Oxidizer Pyrophroic Unstable (Reactive) Water Reactive

The simple language and limited number of definitions will enhance the retention of learning. The presentation of the hazards could be made as short or as long as needed depending on whether the presentation is to be used as introductory material or as a complete training session on hazards specific to the workplace.

The evolution of the Laboratory Standard (29 CFR

1910.1450) is more readily seen in its proper perspective with the history of the other laws and in the relationship of OSHA laws with Department of Transportation (DOT) and Environmental Protection Agency (EPA) laws. The same hazardous chemical has different safety laws applied to it depending on the situation. OSHA laws apply to the chemical in the workplace. DOT laws apply to the chemical in transportation, packaging, and placarding. EPA laws apply to the chemical in the environment or community. They are especially applicable in the case of a chemical spill. Overhead transparency masters follow the discussion of the laws (10). They are repeated in Appendix C.

Five federal regulations form the core of the standard of conduct by which all hazardous materials users are measured.

(a) HAZARD COMMUNICATION STANDARD: An OSHA rule which requires employers to inform their employees of the dangers of hazardous materials found in the workplace. This standard also requires employers to provide the training and information employees need to protect themselves from these materials.

OCCUPATIONAL (b) EXPOSURE TO HAZARDOUS MATERIALS IN THE LABORATORY STANDARD: Also known as the OSHA Lab Standard, this mandate requires that employers protect laboratory staff from over-exposure to hazardous materials. Compliance with this standard therequires adoption of an integrated Chemical Hygiene Plan.

(c) SUPERFUND AMENDMENTS AND REAUTHORIZATION ACT (SARA): Enforced by EPA, Title III of this law is extremely important to academic institutions. This part of SARA is known as the Emergency Planning and Community Right-to-

Know Act. It grants any member of the community, in which your school is located, access to information on chemicals present on your campus. SARA also requires that your institution provide EPA, your state government, and local community officials with an inventory of hazardous materials located on your campus.

(d) RESOURCE CONSERVATION AND RECOVERY ACT (RCRA): Provides for the safe treatment and disposal of hazardous waste. Violations of this EPA-enforced law have cost colleges and universities around the country millions of dollars in fines and associated compliance costs.

(e) COMPREHENSIVE ENVIRONMENTAL RESPONSE, COMPENSATION, AND LIABILITY ACT (CERCLA): Authorizes EPA to finance the clean-up of abandoned hazardous waste dump sites and provides the federal agency with the legal means to recover clean-up costs. Colleges and universities can be named as co-violators in CERCLA-related cases when hazardous waste traced to their institution was discovered at these illegal sites (7). Figure 1. Related Laws

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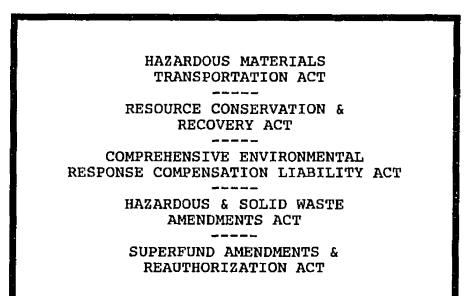


Figure 2. Relationship of Laws

OSHA	DOT	ЕРА
EMPLOYER EMPLOYEE HAZARDOUS CHEMICALS HAZARDOUS MATERIALS WORKER RIGHT-TO-KNOW	SHIPPERS CARRIERS DRIVERS HAZARDOUS MATERIALS HAZARDOUS SUBSTANCES "RQ"IN ONE CONTAINER	GENERATORS TRANSPORTERS TREATMENT STORAGE & DISPOSAL FACILITY UNCONTROLLED HAZARDOUS WASTE SITE COMMUNITY RIGHT-TO-KNOW HAZARDOUS WASTE HAZARDOUS SUBSTANCES

.

The disposal of hazardous chemical waste is costly. Brown University listed "rapidly increasing costs for disposing of hazardous wastes" as one of the reasons for raising their tuition 7.5%. They budgeted \$21,000 in 1986-87 for handling wastes on campus and \$120,000 in 1988-89. The University of Vermont paid less than \$20,000 in 1984 and \$160,000 in 1988 (6).

When the subject of wastes comes up, a whole new vocabulary needs to be incorporated. The portion of the law that applies here is Section 261.5 of CFR 40. These regulations are part of the EPA laws. Most of the universities and colleges could qualify to be "small quantity generators" of hazardous wastes if they produce no more than 100 kilograms of hazardous waste in one calendar month. A total of 1000 kilograms is the limit that can be stored on-site. Some wastes are classified as "acute hazardous wastes" in Sections 261.31, 261.32, 261.33 of this same chapter. An accumulation of a total of one kilogram is the limit on these.

In determining the quantity of hazardous waste generated, a generator need not include: (1) Hazardous waste when it is removed from the on-site storage; or (2) Hazardous waste produced by on-site treatment (including reclamation) of his hazardous waste, so long as the hazardous waste that is treated was counted once; or (3) Spent materials that are generated, reclaimed, and subsequently reused on-site, so long as such spent materials have been counted once (11).

The problem is finding a contractor who is willing to handle such a diverse waste stream (12).

Another cost of hazardous waste disposal could be a fine if it is not disposed of properly or if documentation is not followed through correctly. Stanford University opened an \$8-million facility to store and dispose of hazardous and biological wastes but an operations manager of the university's environmental health and safety department was made to resign after charging the university of mismanaging chemical wastes. After the state issued 21 citations for violations and the university paid a \$200,000 lawsuit to the employee for wrongful termination, the department has been reorganized and chemical wastes are receiving more attention (6). Iowa State University has a special facility for handling hazardous wastes but it received \$37,000 in fines from EPA for violations of federal hazardous waste regulations (13).

The difference between a hazardous chemical and a hazardous waste is the point in time at which it is called, "WASTE". Until the chemical has reached its disposal point, it could be called "used" or "to be recycled". Many solvents can be redistilled and used again.

The minimization of waste can be accomplished by simply using smaller amounts in the beginning. Microscale techniques in instruction can be profitable not only in the cost of disposal, but also in reducing the cost of purchasing the reagents. Using the product of one experiment to be a reagent in another was a common occurrence in organic instructional laboratories (14,15). Rendering a hazardous product innocuous is another way of reducing the disposal problem to a controllable size (16).

An intended major attraction of this study is the Laboratory Standard. "This final standard published today shall become effective on May 1, 1990. ... Employers shall have completed an appropriate Chemical Hygiene Plan and commenced carrying out its provisions by January 31, 1991." So states the Federal Register, Vol. 55, No. 21 published January 31, 1990. Why then was it such a surprise to academia to find that this ruling and regulation on Occupational Exposures to Hazardous Chemicals in Laboratories applies to academic laboratories?

A copy of the Federal Register, January 31, 1990, should be given to every science teacher who uses or has chemicals in their work station and to every school official who has supervision over science teachers. The entire pamphlet is necessary. The law itself is short and written in "legalese". For a true understanding of the depth and breadth - the "spirit of the law" - the preliminary section should be read in its entirety. The preamble contains a record of the testimonies and comments of controversies on the various parts of the law.

The regulation, 29 CFR 1910.1450, does not specify

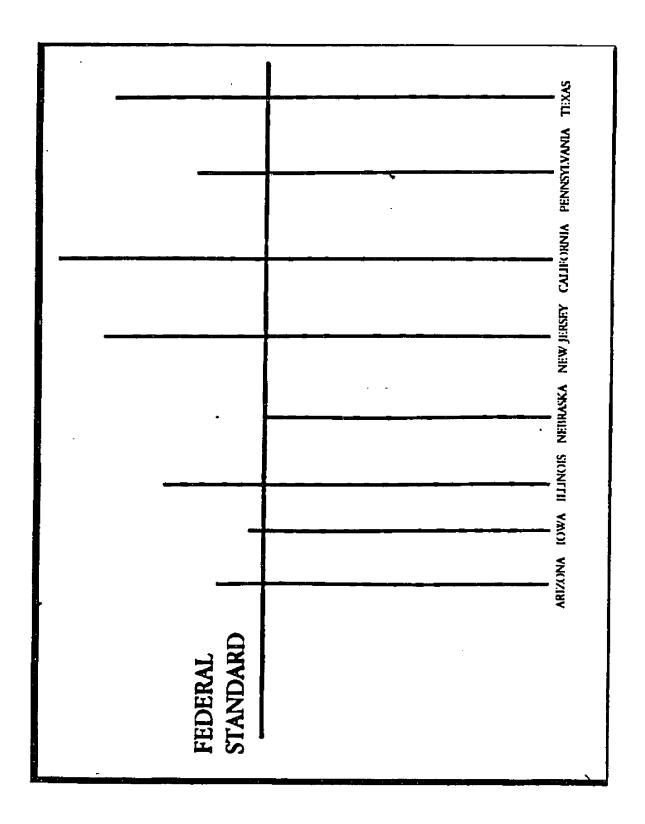
"school laboratories". The spirit of the law in its preamble includes the estimated cost of compliance for industrial, clinical, and academic laboratories. The definitions of "laboratory" and "laboratory scale" in the law itself carry the weight of the where and to whom this law applies.

The law applies to academic laboratories. It only remains now for the schools to fit their current standard operating procedures and rules of safety to the prescribed format. Teachers are cautious and caring, but do sometimes become lax and overlook some things. Students are not employees, but they do have a right to know where and what Material Safety Data Sheets are (17). Students are not employees, but instructors, secretaries, stockroom personnel, graduate teaching assistants, student workers, custodial staff, and research assistants who receive a paycheck are employees.

It has always been an issue to have safe laboratories in schools. Now has come the time for this issue to be made more consistent in its adherence to standardized practices of safety. <u>Prudent Practices for Handling Hazardous</u> <u>Chemicals in Laboratories</u> by the National Research Council is cited repeatedly as a suggested reference for laboratories (18a,19). Other suggested references are given on the last page of the Standard. Most of these are the "classics" of safety literature and can be found in almost

any college library: Sax's <u>Dangerous Properties of</u> <u>Industrial Materials</u>, Patty's <u>Industrial Hygiene and</u> <u>Toxicology</u>, Sittig's <u>Handbook of Toxic and Hazardous</u> <u>Chemicals and Carcinogens</u> (20,21,22). Bretherick's <u>Handbook</u> <u>of Reactive Chemical Hazards</u> is a nice, "handy" handbook that gives incompatibilities in a convincing manner (23). Proctor and Hughes' book, <u>Chemical Hazards of the Workplace</u> is good for how to recognize hazards and has some helpful surveillance charts (24).

Tennessee is one of the "state plan states". This means their Occupational Safety and Health laws must be the same or more stringent than the federal laws. Other "state plan states" include: Arizona, Iowa, Illinois, Nebraska, New Jersey, California, Pennsylvania, and Texas (10). Figure 3. Diagram of State Plan States



Occasionally there is a paragraph inserted into a law that makes an individual state's enforcement a little different, but Tennessee has adopted the Laboratory Standard almost verbatim.

The process by which Tennessee became a "state plan state" was somewhat lengthy. In 1975, there were three OSHA inspectors whose job it was to cover the whole state of Tennessee. They just could not do it all. In the period from 1976 to 1983, the Tennessee Occupational Safety and Health Administration (TOSHA) was formed and was run in a probationary fashion. The three areas of criteria were the lab facility, industrial hygienists, and accreditation. There are now three sections of TOSHA: Consultation, for free services; Public Sector, for governmental agencies and parks; and Compliance, for enforcement, complaints and routine inspections. The TOSHA lab is technically under the Compliance section, although, they do some work for the Consultation and Public sections. Each section is funded a little differently with federal funds and state funds coming from various departments of the respective governments (25).

TOSHA has prepared a handout to try to simplify compliance to the Laboratory Standard. Appendix A of their handout is a copy of the Laboratory Standard minus the preamble. Topics covered by the TOSHA handout include: Personnel Responsible for Program Implementation, Hazardous Chemical List, Employee Knowledge and Implementation,

Chemical Hygiene Plan (CHP), and Recordkeeping. In addition, there are numerous appendices. The title page has a note, "What TOSHA inspectors will look for when they evaluate facilities for compliance with the <u>Occupational</u> <u>Exposure to Hazardous Chemicals in Laboratories, 29 CFR</u> <u>1910.1450</u>" (8b).

The people responsible for seeing that a Chemical Hygiene Plan is implemented are: the Chief Executive Officer (a college president or a school board president or a superintendent of schools), a Chemical Hygiene Officer ("an employee designated by the employer and who is qualified by training or experience, to provide technical guidance" ...(18b)), and a Chemical Hygiene Committee (if appropriate). A hazardous chemical list is to be compiled. The hazardous chemicals list should be available at all times at each workplace and cover only those chemicals at that particular place. A master list should be kept by the chemical hygiene officer.

Employees need to know the chemicals and the hazards they are working with. This becomes a little cumbersome in an academic setting where each work session entails the use of a different chemical. If a routine (standard operating procedure) is developed so the worker knows to look at the Material Safety Data Sheets (MSDS) and labels to the chemicals they will be working with, they should be able to answer the following questions any time (and when an

inspector shows up).

- 1. What is the objective of the <u>Occupational Exposure</u> to <u>Hazardous Chemicals in Laboratories</u>, 29 CFR <u>1910.1450</u>?
- 2. What hazardous chemicals do you work with, and where?
- 3. What long and short term effects can they have on your body?
- 4. How can you detect presence, concentration level and/or release of the hazardous chemicals you work with?
- 5. What measures are or can be taken to protect you against 1) overexposure during normal use, and 2) overexposure in the event of an emergency?
- 6. What are the medical provisions of this standard?
- 7. Where is the information, such as MSDS and the Chemical Hygiene Plan, located, and have they been explained to you?*
- * Such information shall be provided at the time of an employee's initial assignment to a work area where hazardous chemicals are present and prior to assignments involving new exposure situations. The frequency of refresher information and training shall be determined by the employer (8b).

CHAPTER 3

ANNOTATED 29 CFR 1910.1450 SPECIFICS

"Laboratory" means a facility where the "laboratory use of hazardous chemicals" occurs. It is a workplace where relatively small quantities of hazardous chemicals are used on a non-production basis.

"Laboratory scale" means work with substances in which the containers used for reactions, transfers, and other handling of substances are designed to be easily and safely manipulated by one person. "Laboratory scale" excludes those workplaces whose function is to produce commercial quantities of materials ...

"Laboratory use of hazardous chemicals" means handling or use of such chemicals in which all of the following conditions are met:

(i) Chemical manipulations are carried out on a"laboratory scale";

(ii) Multiple chemical procedures of chemicals are used;

(iii) The procedures involved are not part of a production process, nor in any way simulate a production process; and

(iv) "Protective laboratory practices and equipment" are available and in common use to minimize the potential for employee exposure to hazardous chemicals (18a).

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The Chemical Hygiene Plan (CHP) has eleven parts.

1. Basic Rules and Procedures.

These are the "standard operating procedures" (SOP's). Every laboratory class has them. They can be the second sheet in the lab manual or the middle paragraphs on the course syllabus. In a college setting, one set of rules can cover the whole department or each class can have their own. Whichever is decided, the hygiene officer needs a copy.

2. Chemical Procurement, Distribution, and Storage.

These are written already in the faculty handbook. The student workers may have written or verbal instructions on how the chemicals are obtained for their work. The storage is what gets prickly. The following information is taken from the appendix of the Lab Standard (18c) with the numbers in parentheses being page numbers from <u>Prudent Practices</u>.... (Since this information is in the appendix, it is suggested, not mandated.)

(b) Stockrooms/storerooms. Toxic substances should be segregated in a wellidentified area with local exhaust ventilation (221). Chemicals which are highly toxic (227) or other chemicals whose containers have been opened should be in unbreakable secondary containers (219). Stored chemicals should be examined periodically (at least annually) for replacement, deterioration, and container integrity (218-19).

Stockrooms/storerooms should not be used as preparation or repackaging areas, should be open during normal working hours, and should be controlled by one person (219). (c) Distribution. When chemicals are hand carried, the container should be placed in an outside container or bucket. Freightonly elevators should be used if possible (223).

(d) Laboratory storage. Amounts permitted should be as small as practical. Storage on bench tops and in hoods is inadvisable. Exposure to heat or direct sunlight should be avoided. Periodic inventories should be conducted, with unneeded items being discarded or returned to the storeroom/stockroom (225-6, 229).

3. Environmental Monitoring.

Any air quality testing and hood air flow measurements need to be documented. The law is not too specific on when these tests need to be done but at least every 3 months for hood inspections. A good routine time for schools is once or twice each semester, depending on how long summer and winter sessions are. Air flow must be 80 to 100 linear feet per minute (18c). The hood sash may be lowered to achieve that measurement, in which case, a piece of tape must mark the spot and a note signifying the measure (26). A hot wire anemometer is nice for determining air flow but there are several other less expensive instruments on the market (27,28a). A good way to keep track of the inspections is to adhere a sticker on the side of the hood with lines for the dates of the inspections and the initials of the inspectors (29).

Air quality testing is not mandated unless there is reason to believe the concentration of a contaminant is above its legal limits. Spot testing with a hand pump and analyzer tubes is convenient to determine if further testing and quantifying is necessary. The analyzer tubes are specific for designated chemicals (28b).

4. Housekeeping, Maintenance, and Inspections.

The following information is again taken from the appendix of the Lab Standard (8c) with the numbers in parentheses representing page numbers from <u>Prudent</u> <u>Practices...</u>

(b) Inspections. Formal housekeeping and chemical hygiene inspections should be held at least quarterly (6, 21) for units which have frequent personnel changes and semiannually for others; informal inspections should be continual (21).

(c) Maintenance. Eye wash fountains should be inspected at intervals of not less than 3 months (6). Respirators for routine use should be inspected periodically by the laboratory supervisor (169). Safety showers should be tested routinely (169). Other safety equipment should be inspected regularly (e.g., every 3-6 months)....

One instructor made these safety inspections part of every semester's check-out procedure. He would write a clean-up task on a 3x5 card. At the beginning of lab on check-out day each pair of students received a task card which was to be completed before he would sign their checkout sheets (14). Safety showers may or may not have a drain. There is a two-fold purpose for not having a drain. One is to contain any spills or washes of hazardous chemicals from entering the community water system. The other is to discourage anyone from deliberately releasing water in horseplay (30). To inspect the shower, one need only to borrow the janitor's big plastic waste barrel on wheels. Put the barrel under the shower and pull the ring.

5. Medical Program.

Every school or department has already an accident report form and a procedure to follow in the case of an emergency. The hygiene officer just needs a copy to put in the CHP along with the name and telephone number of the physician. Medical monitoring may be necessary for some employees, like, professors or graduate students who receive a paycheck and are doing research with toxic chemicals. These "Exposure and Medical" records must essentially be kept for 30 years (8c).

6. Personal Protective Apparel and Equipment.

Again, from the appendix of the Laboratory Standard:

These should include for each laboratory: (a) Protective apparel compatible with the required degree of protection for substances being handled (158-161); (b) An easily accessible drench-type shower (162-169); (c) An eye wash fountain (162); (d) A fire extinguisher (162-164); (e) Respiratory protection (164-9), fire alarm and telephone for emergency use (162) should be available nearby; and (f) Other items designated by the laboratory supervisor (156, 160).

The term "easily accessible" is pertinent. Many safety

showers have been unused so long that things become stacked under them; a desk, fire blanket, ice containers, <u>etc</u>. A person would literally have to clean out from under it to use it. If a situation is severe enough to use the shower, time is essential and the things in the way would be a hazard to the person fumbling or groping for the shower! 7. Records.

Some of the kinds of information that need to be kept have already been mentioned: accident records, CHP records which document that the facilities and procedures were "compatible with current knowledge and regulations" (18c), inventory and use records for high risk substances, and medical records. Other records to be discussed include: training and waste disposal documentation.

8. Signs and Labels.

Some signs, like the National Fire Prevention Association (NFPA) colored decals, are regulated in other laws. Especially useful in chemistry laboratories are signs for emergency posting of telephone numbers of emergency personnel and location signs for safety equipment. Identity labels and warning signs at areas where special or unusual hazards exist are critical. Unfortunately, many working with hazardous chemicals or situations become so used to the hazards, they are not cognizant the hazard exists and object to posting signs so frequently.

9. Spills and Accidents.

"All accidents or near accidents should be carefully analyzed with the results distributed to all who might benefit" (18d). The person to whom the report is made is not specified. Each school, department or organization has its own accident policy in which these details are decided. Some chemical spills are regulated by other laws, for example, formaldehyde. Special spill kits are available commercially. A wonderfully inexpensive way to handle normal spills is to use "kitty litter" (30).

10. Information and Training Program.

The aim of this part of the CHP is to assure that all employees are adequately informed of the hazards they may encounter. Material Safety Data Sheets (MSDS) are to be kept in an accessible, centrally located place for all chemicals purchased. Additional emergency and first aid training for designated personnel may be in order. The other employees need to know who these designated people The key is to communicate (and document)! "The are. training ... should be a regular, continuing activity - not simply an annual presentation (15).... Literature and consulting advice concerning chemical hygiene should be readily available to laboratory personnel who should be encouraged to use these information resources (14)"(18d). "Readily available" is a prickly phrase. It is good to have the MSDS in a centrally located place, but if that place is

locked up when the student worker needs access to it after 5

pm, it is not "readily available".

(3) Information. Employees shall be informed of:

(i) The contents of this standard and its appendices which shall be made available to employees;

(ii) The location and availability of the employer's Chemical Hygiene Plan;

(iii) The permissible exposure limits for OSHA regulated substances or recommended exposure limits for other hazardous chemicals where there is no applicable OSHA standard;

(iv) Signs and symptoms associated with exposures to hazardous chemicals used in the laboratory; and

(v) The location and availability of known reference material on the hazards, safe handling, storage and disposal of hazardous chemicals found in the laboratory including, but not limited to, Material Safety Data Sheets received from the chemical supplier.
(4) Training. (i) Employee training shall include:

(A) Methods and observations that may be used to detect the presence or release of a hazardous chemical (such as monitoring conducted by the employer, continuous monitoring devices, visual appearance or odor of hazardous chemicals when being released, etc.);

(B) The physical and health hazards of chemicals in the work area; and

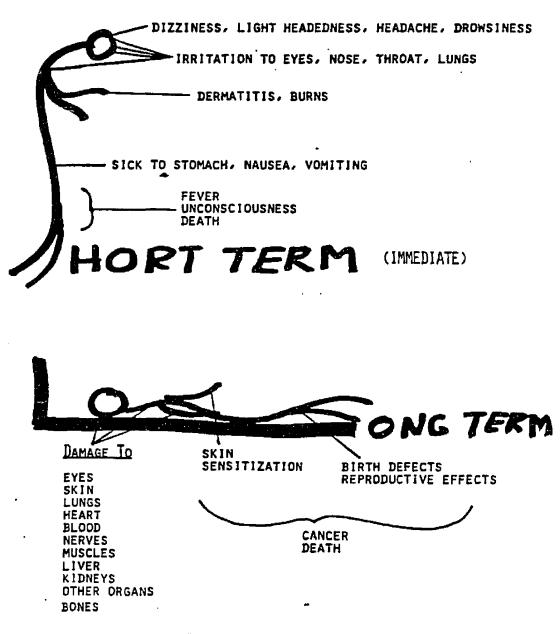
(C) The measures employees can take to protect themselves from these hazards, including specific procedures the employer has implemented to protect employees from exposure to hazardous chemicals, such as appropriate work practices, emergency procedures, and personal protective equipment to be used.

(ii) The employee shall be trained on the applicable details of the employer's written Chemical Hygiene Plan (18f).

The employer's CHP is not to be simply an exercise in

writing documents. It is intended to be used in the workplace and its use must be verified. In the questions a Tennessee Occupational Safety and Health Administration (TOSHA) inspector might ask, as previously discussed, an outline of the information a worker may need to know is again apparent. "Any method that produces verbal recall of the information required ... will be adequate. Person to person checking of this information is a most effective way to comply with the requirements" (8d). Appendix F of the TOSHA Booklet has an excellent presentation of the short term (immediate) and long term bodily effects of chemicals. It is reproduced here and with the overhead masters. Figure 4. TOSHA's Appendix F

BODILY EFFECTS OF CHEMICALS



HAVE A HEALTHY RESPECT FOR CHEMICALS

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11. <u>Waste Disposal</u>.

The plan for each laboratory operation (SOP) should include procedures and training for waste disposal. Chemical wastes should be put in appropriately labeled receptacles. Concentrated acids or bases, highly toxic materials, or any substances which might interfere with the biological activity of waste water treatment plants, create fire or explosion hazards,... or obstruct flow should not be discharged into the sewer (18g). EPA and DOT regulations need to be considered. For many schools the easiest answer is to hire a contractor. The waste disposal plan should specify how waste is to be collected, segregated, stored, and transported.

CHAPTER 4

WHAT WAS DONE TO COMPLY

Listing of chemicals and tasks

An inventory of all the chemicals in the organic lab area was made. This was easily accomplished by a student worker. Each day of work, she would take a section of chemicals (Solids, Liquids, Solutions, Dilute acids, Flammables cupboard, or Preparation room shelves) and list the chemicals. This was not difficult because the chemicals were in alphabetical order in their respective groups. After compiling these lists into a master list, chemicals were identified as to whether they were classified as hazardous by comparing the master list to 29 CFR 1910, Subpart Z.

Numerous chemicals were no longer being used in the organic lab area after the conversion to microscale instruction and because lab texts had been changed. These chemicals were returned to the main stockroom area. Some chemicals were being stored in gallon and liter bottles, which were larger quantities than needed. These, also, were returned. This action cut down on the inventory considerably, making it easier to identify specifically the hazardous chemicals most likely to be encountered by a student worker in laboratory preparation.

The various tasks a student worker would be called upon to do were identified. Preparing septum reagent bottles and

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distilling reagents seemed to be two of the tasks that would involve the most exposure of a chemical. Since the change to microscale, large distillations are no longer as common as they once were. To duplicate the "before" type conditions, a large scale distillation was done to see what kinds of values could be related to PEL's, and TLV's.

Hood sinks are notorious for leaking. For that reason, often the distillation would be done at a student bench work area which has easy access to sink and water for cooling the condenser. In the microscale technique, water cooling is not necessary. The relative size and length of the condenser provides sufficient cooling by air. Other tasks which may expose a worker to chemicals are the washing and rinsing of glassware. The chemicals in the containers being washed and the type of rinse (for example, acetone) might pose a hazard, especially if done in an area without hood ventilation.

Waste: Reduction, Disposal, and Documentation

(1) <u>Waste Reduction</u>

Hazardous chemicals and hazardous chemical waste disposal are the stuff nightmares are made of for department chairpersons and maintenance managers. The disposal of hazardous chemical waste is costly. The cost goes up dramatically if there are bottles or jars of chemicals to be disposed of that have no labels. These "unknowns" are handled with extreme care.

The American Chemical Society provides a booklet entitled, Less Is Better, Laboratory Chemical Management For Waste Reduction (31). The topics discussed are: The Impact of Purchasing Strategy, Purchasing-Disposal Economics, Inventory Control, Recycling, and Waste Reduction in the Laboratory. Some of the techniques recommended for waste reduction include replacing glassware cleaning solutions (like chromic acid solution) with less hazardous proprietary laboratory detergents. Experiments that call for benzene or carbon tetrachloride as reagents or solvents can be modified to use the less hazardous toluene and cylcohexane. This was done in the modification of the organic lab experiments. Cyclohexane is equally efficient for extracting halogens as carbon tetrachloride in the standard qualitative test for halides. In some organic syntheses, the hypochlorite ion can replace the chromate ion as an oxidizer, thereby avoiding the hassle of disposing of the chromium (VI) ion. The section further discusses more techniques that could be used in other courses.

(2) <u>Waste Gathering</u>

In this situation, the waste bottles are set up in one hood. The bottles are labelled according to their functional groups (hydrocarbons, aromatics, halogenated hydrocarbons, oxygen containing, nitrogen containing). The labels are needed to identify the containers when they are put in a lab-pack and are used to determine the type of

disposal procedure that will ultimately be used.

Most colleges hire hazardous waste contractors.

The wastes come in thousands of bottles and containers of old chemicals - used and unused- ranging in size from tiny 10-gram bottles to 5-gallon jugs, or larger. These bottles are packed into 55-gallon drums and transported to privately operated disposal sites, such as incinerators that are authorized to burn hazardous materials (6).

Lab-packs are usually prepared by the contractor for the hazardous waste disposal. The are numerous publications and articles on the subject (32,31b). "The contractor, who must have the necessary EPA permits and authorizations, performs some or all of the following services: packs the wastes properly, labels the containers properly, prepares the necessary forms including a manifest, distributes the forms to the proper destinations, transports the wastes to the disposal site for disposal, certifies that the wastes have been properly disposed of." (33) The confusion is further multiplied by the need to adhere to not only EPA standards but also the rules for a particular treatment, storage or disposal (TSD) facility and DOT standards.

(3) <u>Waste Recycling</u>

In another situation, an acetone rinse station is set up in another hood area. The used acetone is collected and redistilled to be used over again for the same purpose. In classes where microtechniques are not incorporated yet, there are often solvents used for extractions and recrystallizations that are in sufficient quantities to be

reclaimed.

Training and Documentation

In reference to TOSHA inspector questions mentioned before, these things are discussed as a matter of course in academia. If we are fortunate enough to have student workers, they are told and cautioned about each task. At the beginning of their employment, they are escorted around, pointing out the hazards and locations of safety equipment. The only additional thing that may or may not be accomplished is to have that training <u>documented</u>: The student workers and instructors who attend the training sessions should sign a roll. One university gave their three-hour training session during a week-day evening and provided the evening meal (34):

CHAPTER 5

METHODS AND MATERIALS FOR AIR QUALITY STUDY

Hood Velocities

The measuring of hood velocities was accomplished using a Data Metrics, model 800-VTP, air velocity meter borrowed from the Industrial Studies Department (35). Later, a local, private industrial hygiene concern was found willing to loan equipment and instruments - for a small rental fee. Drawings that showed the hoods' locations, model numbers, capacities, and maintenance records were available from the Campus Planning Department.

The 29 CFR 1910 does not dictate a specific face velocity, but only that the CHP will "contain measures to assure the proper functioning of fume hoods and other protective equipment" (18e). Even though not mandated, the average of the air flow measurements is to be between 80 and 100 linear feet per minute as suggested in the appendix of the standard (18c). The hood face area is divided into an imaginary 12-line grid. The intersections of the four vertical lines and three horizontal lines are where the measurements are taken. This takes a bit of doing, because if a person or object is in front of the hood, eddies in the air current interfere with the measurement. The method used was to sit on the floor on a wheeled pallet and hold the long, thin arm of the instrument up to the imaginary intersections. The average face velocity was determined. If

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the hood's face velocity does not meet the criteria, simply lower the window until it does. A piece of tape or a nonerasable mark is to be put on the window sash. The window should be kept at that level or lower when the hood is being operated.

The student work bench areas are supplied with surface vents. There are no specific criteria mentioned for these. The air velocities were measured in the center of the opening for the smaller vents and an average of four measurements were taken for the larger vents. Chronic problems occur with individual student vents: paper, pens, and even small glassware is drawn into the air flow, clogging it. Traditionally, these mishaps are overlooked and maintenance orders are either not written or not given priority until remodeling. Keeping records can help identify clog locations.

Air Sample Collection

The measurement of the air quality was accomplished using standardized procedures outlined in 29 CFR 1910.1450. Air sample collection was accomplished using a personal air pump with charcoal filter tube attached to the worker's collar (36). While grab sampling with evacuated canisters is usually conducted in EPA air quality studies, the air pump procedure coincides with OSHA studies. Gas chromatography was used to analyze the air samples. The data collected was compared to the American Conference of

Governmental and Industrial Hygienists (ACGIH) threshold limit values (TLVs), the Occupational Safety and Health Act permissible exposure limits (PEL), and the National Institute for Occupational Safety and Health (NIOSH) recommended exposure limits (RELs) (37,38,39).

Air sampling methods and evaluative methods used in a study depend on ∞ whom the resulting information is to be reported. Since this is an OSHA law and because the air a <u>worker was to breathe</u> was being tested, a personal air sampling pump with a small charcoal collection tube was used instead of an evacuated canister (40). The evacuated canister method is used for EPA type studies. The procedures followed were OSHA and NIOSH methods as published in their respective manuals (41,42a).

The pump used was the Bendix Super Sampler BDX 44 Pump (43). OSHA laws and methods use the concentrating effects of the pump attached to the worker's collar to collect the air being breathed. The calibration of the personal air sampling pump was accomplished by the use of an inverted buret, soapy water to make bubbles, and a stop watch (41).

In the gas chromatography analyses of the air samples, again, OSHA methods were adhered. Some EPA procedures use thermal desorption or purge and trap attachments to the GC instrumentation. While these are very attractive and a lot less smelly, the OSHA method stipulates desorption with carbon disulfide in a number of the procedures used in this

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study.

Sample preparation and storing can be tricky. It is best to store the collected charcoal tubes with their plastic end covers in a refrigerator until they can be desorbed. It even works out to put the charcoal in a sample vial with a tightly sealed screw cap as long as the desorbent is NOT added. It is not recommended to try to store samples in the desorbent (25). The very best choice is to do the collection and running of the samples all in the same day but that is often impractical.

Next, the samples were desorbed by adding the carbon disulfide, placing the sample vials on a mechanical shaker and shaking for 15-30 min prior to analysis (42a). The amount of time required seems to vary depending on the chemical in the sample: acetone, 15 min; toluene and benzene, 30 min. (42b). More time will not affect the results (25). This had to be done just prior to the injection of the sample into the GC. It would be a mistake to shake all the samples and then have them sitting around three hours until it is their turn to be injected.

Gas Chromatographic Parameters

The Varian Series 3700 Gas Chromatograph was used with the Aerograph Flame Ionization Detector (FID). The column was 6-foot, packed with 0.1% Carbowax 1500 with Supelcoport SP-2100. Hewlett Packard Chemstation program was the computing integrator system used. Samples were run at 140

degrees C. Most of the samples injected were 0.5 uL. Some difficulties were encountered when the FID developed a ceramic-type coating due to the use of the carbon disulfide.

The air samples collected from the worker's collar were the main concern. Several ancillary studies needed to be done:

- Retention times of specific members of certain classes of organic compounds (alcohols, alkanes, aromatics, amines, and alkyl halides but NOT acids nor phenyl hydrazine)
- Calibration curves for selected common solvents (benzene and toluene)

Standards Preparation

An internal standard was necessary because the process by which carbon disulfide is produced always leaves a little benzene in the reagent. The internal standard chosen was chlorobenzene. Its retention time is much later than any of the other chemicals that were expected to be encountered in this study. External standards were still necessary because the air samples collected needed to be compared to known quantities of the expected components.

Each of the expected chemical components have differing desorption efficiencies. For example, benzene has a desorption efficiency of 98%, which means for this procedure, 98% of the benzene absorbed on the charcoal will be desorbed. For toluene, the desorption efficiency is 95%.

Benzene and toluene are so well documented the literature values for them were used (25).

CHAPTER 6

RESULTS AND DISCUSSION

The data tabulated included the hood velocities, the surface desk vent velocities, and a summary of the air quality samples. The hood velocities were measured in standard feet per minute (fpm). The air quality samples were gathered by identifying several tasks a student worker would normally be expected to do, wearing a personal air sampling pump with charcoal filter while doing those tasks, desorbing the charcoal filter in carbon disulfide, and injecting the samples into a gas chromatograph. The gas chromatograms were then analyzed by comparing with standard curves and by calculation using the internal standard.

Calibration curves for benzene and toluene were prepared. The graph for benzene does not go through the origin because of the small quantity of benzene present in the solvent.

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TABLE 1. Hood Velocities, fpm.

With Bottles	1	2	3	4	5	9	7	8	6	10	11	12	average
Hood 1	140	120	130	100	150	110	110	105	120	06	60	01	601
Hood 2	90	100	80	70	85	85	90	80	70	85	80	75	83
Hood 3	70	70	80	90	80	85	90	80	80	90	96	80	82
Hood 4	Hood	was	empty										
Without													
Hood 1	150	140	110	110	140	135	130	105	130	130	120	120	127
Hood 2	90	90	90	80	100	100	100	80	100	100	100	06	69
Hood 3	70	70	80	70	70	90	90	80	80	100	011	90	83
Hood 4	100	100	100	100	110	120	120	120	110	06	120	125	110

(in linear feet per minute)

Hood Velocities

TABLE 2. Desk Vent Velocities, fpm.

	top	bottom	average
1	210	130	170
2	110,110	110,110	110
3	130	170	150
4	150	160	155
5	120,120	90,90	105
6	210	220	215
7	210	190	200
8	90,70	85,50	74
9	80	100	90
10	100	100	100
11	50,60	50,50	53
12	40	110	75
13	210	190	200
	120,70	130,80	100
14	120	140	130
15	120	120	120
16	_50,50	40,70	53
17	10	120	65
18	150	150	150
19	100,50	80,50	70
20	80	100	90
21	70	90	80
22	80,100	80,100	90
23	150	170	160

Desk Vent Velocities

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(in linear feet per minute)

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FIGURE 5. Gas Chromatogram of Carbon Disulfide.

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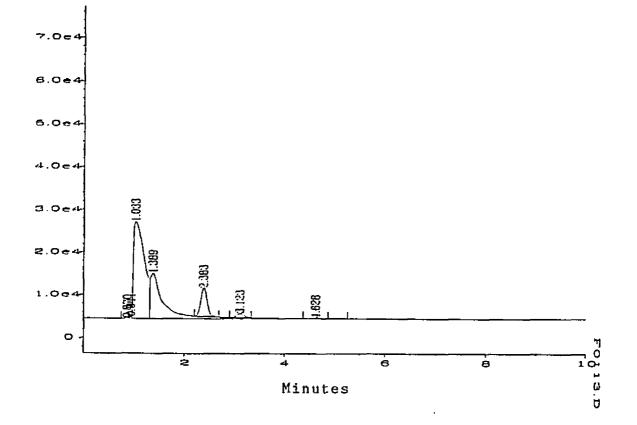


FIGURE 6. Gas Chromatogram of Carbon Disulfide with Chlorobenzene as Internal Standard.

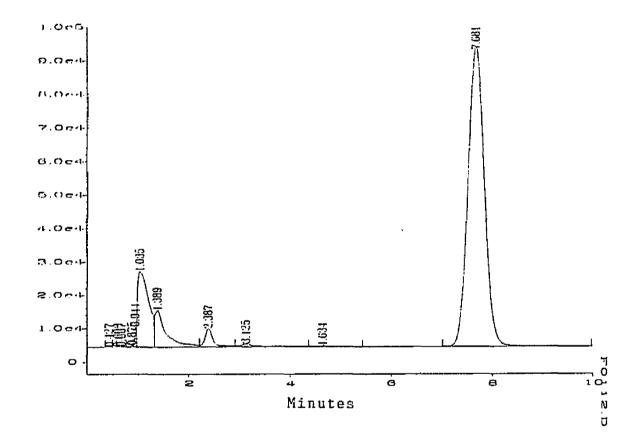


TABLE 3. Tabulated Retention Times and Peak Areas of Carbon Disulfide With Chlorobenzene as Internal Standard

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	0.964 1.235				
Start Time	0.964	1.235	2.061	6.602 6.602	7.812
Area	22211	5986	3540	177343	20
Width 0 041	0.159	0.087	0.111.0	0.307	0.039
Туре РV	ΡV	22	24 24	ΡV	Δ
Ret Time 0.920	1.040	1.267	162.2	7.196	7.835
Peak# 1	6	ר יז ד	. 0	9	٢.

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FIGURE 7. Gas Chromatogram of Air Sample # 1

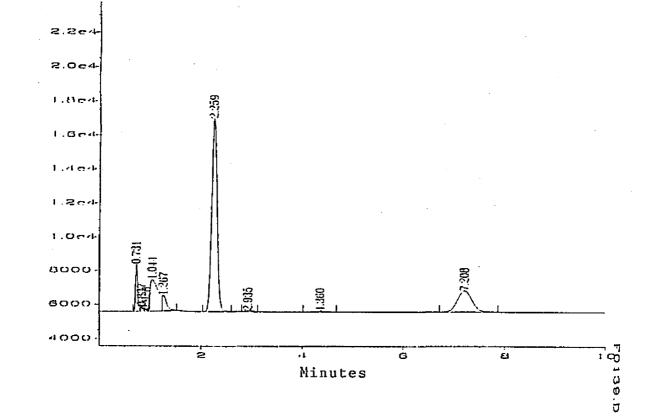


TABLE 4. Tabulated Results of All Air Samples

Tabulated Results

3	Tasks	Benzene	Toluene	Chlorobenzene	
	dismantled distillation	3991	434	25819	area count 1st
30 min	apparatus, washed and	2490	0	22801	area count 1st
	rinsed		1		area count and
	filled Toluene bottles		1		
	and Cyclohexane bottles				
	(on bench top)				
		0	0.0928		parts per million
			L		(first half only)
4		L	L		
	washed beakers and bottles	4168	7581	23442	area count 1st
40 min	in prep room (no hood)	2469	0	23426	area count 2nd
	MEK, Toluene, Cyclohexane				
	cleaned hood, acetone				
	+				
		0	1.336		parts per million
					(first half only)

Tabulated Results

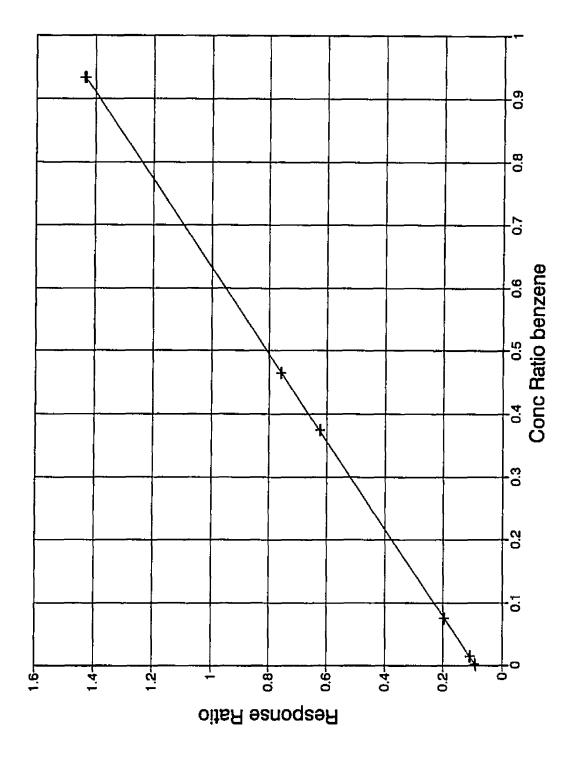
Sample	Tasks	Benzene	Toluene	Chlorobenzene	
<u> </u>	Benzene distillation	84348	534	24990	area_count_lst
	set up on bench top	2499	0	24087	area count 2nd
55 min	distilled benzene,				
	washed bottles				
		8.18	0.0642		parts per million
			ļ		(first half only)
2	cont. distillation of Benzene	5455	232	26007	area count 1st
	rinsed bottles, acetone	2424	0	23243	area count 2nd
45 min	(in hood)				
	changed collecting vessel				
	filled Benzene bottles				
	(in hood)				
		0.0783	0.0327		parts per million
					(first half only)

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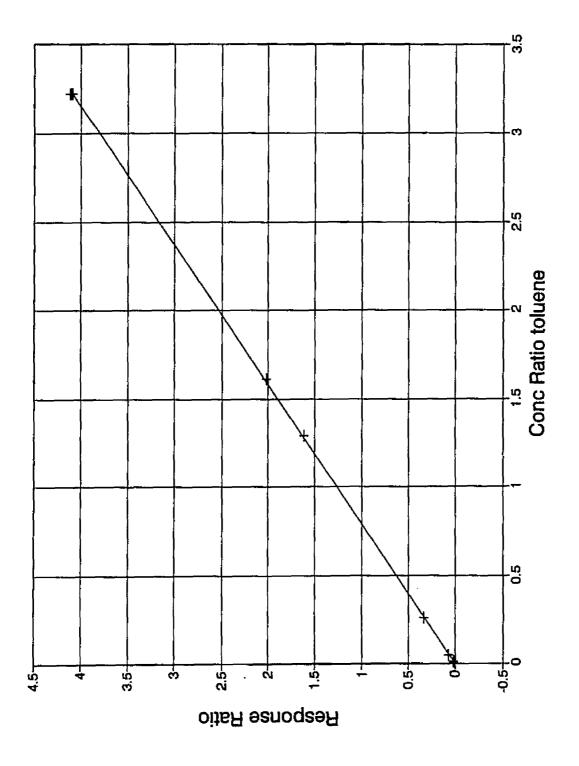
FIGURE 8. Calibration Curve for Benzene

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FIGURE 9. Calibration Curve for Toluene



From the gas chromatogram of the solvent, carbon disulfide, in Figure 5, the small peak at about 2.3 minutes represents the benzene. This same amount will be in all the air samples and standard curve runs. In order to quantify the amount of benzene in each run, especially since it is one of the air contaminates, chlorobenzene was selected as the internal standard, Figure 6. Chlorobenzene's retention time is much later, about 7.2 minutes.

Four air samples were collected. The chromatogram for Sample # 1 is shown here. The chromatograms of the remaining samples are in Appendix B. In each air sample, the second half of the charcoal filter was processed separately. The quantity of contaminate was compared to the amount present in the first half to see that no "break through" had occurred. A "break through" is indicated if the amount of contaminate in the second half is 0.25 or more times the amount in the first half and is due to overloading the capacity of the charcoal filter. Table 4 shows that no "break through" had occurred in any of the air samples.

Benzene is asterisked in Table 4 to indicate an example of the calculations involved. The area under the peak will be adjusted to compensate for the amount present in the solvent. Sample calculations for benzene are shown here, while all others are in Appendix A.

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Calculations Worksheet

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I. Air Quantity

Time (min) x Pump Calibration (L/min) = L
L x 1.179 g/L (as per ideal gas equation) = grams $_{air}$
$_{air}$ grams $_{air} \times 10^3 = mg_{air}$
II. Contaminant Quantity
<u>area_of_contaminant</u> concentration = [<u>area_of_chlorobenzeneb</u>] (0.2214 mg/mL)
of contaminant m
area of contaminant benzene adjustment =
area of chlorobenzene
b = m =
concentration of contaminant (mg/mL) calculated from graph equation
mg/mL from graph equation x volume mL = mg_{contaminant}
mg _{contaminant} / desorbtion efficiency = final mg _{contaminant}
III. Parts per million
final mg contaminant \times 106 / mg air = ppm

Calculations Worksheet for Benzene, Sample 1.

I. Air Quantity

Time <u>55</u> (min) x Pump Calibration <u>2088</u> (L/min) = <u>11.484</u> L 11.484 L x 1.179 g/L (as per ideal gas equation) = $\frac{13.5398}{2000}$ grams air 13. 5396 grams air x 10³ = <u>mg air</u>

II. Contaminant Quantity

area of contaminantconcentration=[area of chlorobenzene - b]of contaminantm

area of contaminant $\underline{34348}$ - benzene adjustment $\underline{3522} = \underline{81826}$ area of chlorobenzene 34990

b = 0.08783 m = (.500)

concentration of contaminant (mg/mL) 0.1095 calculated from graph equation

0.1015 mg/mL from graph equation x volume 1.00 mL = 0.1015 mg_{contaminant} 0.1025 mg_{contaminant} / desorbtion efficiency 0.98 = 0.1108 final mg_{contaminant}

III. Parts per million

0.1109 final mg contaminant x 106 //3 539.6 mg air = 9.180 ppm

Discussion

From Table 4 several observations can be made. The area counts of the chlorobenzene internal standard are all around 24227. This is an indication that the sample sizes injected into the GC were fairly constant.

The second area counts for toluene were less than 0.25 of the first area counts. Indeed, most of the second area counts were zero. As discussed before, the second half of the sample needed to have less than 0.25 of the first half of the sample to insure that "break through" had not occurred.

The second area counts for benzene were averaged to be 2470. This reassures both sample size consistency and that "break through" had not occurred. Air Sample # 1 had a much higher area count for benzene in the first half of the sample than the other three air samples.

The area counts for the chlorobenzene were also constant in the preparation of the calibration curves, but they were different than the area counts in the air samples. This introduces an uncertainty in the calculations. It is believed the difference in the area counts occurred due to the FID problem. The detector's response became much less sensitive and all the peaks were diminished in size. The area ratios of the chlorobenzene in the air samples compared to the benzene and to the solvent, carbon disulfide, were consistent with the area ratios in the calibration runs. For this reason, the graphs, Figures 8 and 9, were prepared usually the ratios of the areas of the contaminants vs the area of chlorobenzene for the y-axis and the ratios of the concentrations of the contaminants vs the concentration of chlorobenzene for the x-axis.

Calculations

The calculations for the concentrations in parts per million for each contaminant were as follows. The formula, y = mx + b, was utilized, where y equaled the ratio of the areas and x equaled the ratios of the concentration of the contaminant (the unknown) to the concentration of the chlorobenzene which was 0.2214 mg/mL in all runs.

Rearranging the formula to solve for the unknown concentration of the contaminant yielded:

	<u>area_of_contaminant</u>	
concentration =	[area_of_chlorobenzene - b](0.2214	mg/mL)
of contaminant	m	

In the calculation for benzene an adjustment was needed to correct for the amount of benzene already in the solvent. The average area of 2522 obtained from blank air samples was subtracted from each air sample's area count for benzene.

For the contaminant, benzene, b equaled the y intercept which was 0.08783. The slope of the line, m, was 6.500. For the contaminant, toluene, b was essentially zero. The slope of the line, m, was 5.730. These values came from the least squares regression of the data and the respective graphs.

Each of the air samples were desorbed into 1.00 mL of carbon disulfide. The desorbtion efficiency of benzene was the accepted 98%. The volume of air collected for Air Sample # 1 was:

55 min x 0.223 L/min = 12.265 L where 0.223 L/min was the calibrated air pump volume/time. Since 1 m³ = 10³ L then volume₁ = 1.23 x 10⁻² m³. Sample conversions were shown in the Calculations Worksheet for Benzene, Sample 1, to convert to parts per million (ppm).

ACGIH has TLVs, OSHA uses PELs, NIOSH has RELs and then there are TWAs. The way the number is to be reported depends upon who the report goes to, what kind of time limits are controlling the exposure, and the nature of the chemical. Benzene has its own standard, 29 CFR 1910.1028. It is a suspected carcinogen. The mandated levels are much lower than most other volatile chemicals. Table 4 lists the mandated concentrations in the most common method for the chemicals listed. Table 5 attempts to show the relationships of these various methods of reporting. Table 6 is a summary table.

TABLE 5. Exposure Limits

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Exposure Limits

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Chemical	OSHA's PEL	TWA	OSHA Celling	NIOSH'S REL TWA	NiOSH Ceiling	ACGIH's TLV
Acetone	1000 ppm (2400 mg/m3)			250 ppm (590 mg/m3) 10 hr		750 ppm
Benzens	10 ppm		50 ppm, 10 min	0.1 ppm, 8 hr,	1 ppm, 15 min	10 ррш
yclohexane	300 ppm (1050 mg/m3)					
Tokiene	200 ppm	300 ppm ceil	300 ppm ceil 500 ppm. 10 min peak	100 ppm. 10 hr	200 port. 10 min	100 oom

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TABLE 6. Summary Table

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J	um	m	a	гy

Material/Task	Measured	PEL
# in () = Air Sample #	Concentration	ppm
	in Air Sample	
Benzene		
set up distillation (1)	8.180	10
change collection (2)	0.0783	10
dismantle (3)	0	10
Toluene		
fill bottles (3)	0.0928	200
clean up, wash (4)	1.336	200

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CHAPTER 7

CONCLUSIONS

One of the unspoken contentions of this study was that academia could easily monitor its own activities. The key word here is "easily". The procedures call for one versed in OSHA Protocol and familiar with GC on a daily basis. The actual work involved covers several job descriptions: industrial hygienist, laboratory director, GC operator, laboratory manager and safety officer. While the university professor often wears several hats, the work here is too much for anyone to keep up and still carry a full teaching load. The person(s) assigned would need to have a goodly block of released time designated for this task. The normal university safety officer could not do this monitoring without hiring an outside concern. Cost comparisons may be in order here.

Further study in this area would include the actual monitoring of student workers on a regular basis. The activities in this study were, for the most part, somewhat contrived, under the more strenuous conditions of summer school. Continued study during a regular school semester would be more "real".

The benzene distillation was an "eye opener". Air Sample #1 was the only sample to have a contaminant, benzene, near legal limits. Even though this procedure is

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no longer being used, it is really scary to know that once the student workers were asked to do a task that exposed them to a chemical in concentrations so close to the now accepted limits of safety. The study reinforces the commitment to adhere to standard safety practices of insisting the distillations of volatile organics are carried out in a hood, to monitor the hoods working conditions and to monitor the student workers' operating procedures.

Academia should have the same sentiments as expressed by Ken Spaulding, UAW Safety and Security Advisor for Saturn Corporation, "We are not concerned about OSHA compliance (because) we concentrate on doing what is right" (44).

APPENDICES

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- A. Sample Calculations
- B. Spectra and Data
- C. Overhead Transparency Masters

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APPENDIX A: Sample Calculations

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Calculations Worksheet

I. Air Quantity

Time <u>45</u> (min) x Pump Calibration 0.2088 (L/min) = <u>9.376</u> L 9.396 L x 1.179 g/L (as per ideal gas equation) = <u>11.0779</u> grams _{air} 11.0779 grams _{air} x 10³ = <u>11.0779</u> mg _{air}

II. Contaminant Quantity

concentration = [area of contaminant of contaminant m

area of contaminant 5455 - benzene adjustment 2522 = 2933area of chlorobenzene 26007

b = 0.02783 m = 4.500 -4

concentration of contaminant (mg/mL) $\frac{8.411}{8.411}$ calculated from graph equation

 g_{491}^{H0} mg/mL from graph equation x volume $\frac{1.00}{1.00}$ mL = $\frac{8497}{M}$ mg_{contaminant} g_{491}^{H0} mg_{contaminant} / desorbtion efficiency $\underline{0.98} = g_{10}^{H0}$ final mg_{contaminant}

III. Parts per million 4 **S.L.71×10** final mg contaminant × 106 / <u>(10779</u> mg air = <u>0.0783</u> ppm

81

Calculations Worksheet

I. Air Quantity

Time <u>30</u> (min) x Pump Calibration 0.2011 (L/min) = <u>6.264</u> L

4.164 L x 1.179 g/L (as per ideal gas equation) = 2.3853 grams _{air}

7, 3153 grams $_{air} \times 10^3 = 7375.3 \text{ mg}_{air}$

II. Contaminant Quantity

 $\begin{array}{rcl} & \underline{area \ of \ contaminant}}\\ concentration & = & [\underline{area \ of \ chlorobenzene \ - \ b}] (0.2214 \ mg/mL)\\ of \ contaminant & m \end{array}$

area of contaminant <u>3991</u> - benzene adjustment <u>2522</u> = <u>1469</u> area of chlorobenzene <u>258</u>19

b = 0.08783 m = 0.500-.001054 concentration of contaminant (mg/mL) _____ calculated from graph equation

 $_$ mg/mL from graph equation x volume $_$ mL = $_$ mg_{contaminant}

_____ mg_{contaminant} / desorbtion efficiency _____ = _____ final mg_{contaminant}

III. Parts per million

_____ final mg contaminant x 106 / 73853 mg air = _____ ppm

Benjene, Sample #4

82

Calculations Worksheet

I. Air Quantity

Time <u>40</u> (min) x Pump Calibration <u>0.2088</u> (L/min) = <u>8.352</u> L 8.352 L x 1.179 g/L (as per ideal gas equation) = <u>9.9470</u> grams _{air} 9.4470 grams _{air} x 10³ = <u>9847.0</u> mg _{air}

II. Contaminant Quantity

 $\frac{\operatorname{area of contaminant}}{\operatorname{of contaminant}} = \underbrace{\operatorname{area of chlorobenzene} - b}_{\mathrm{m}} (0.2214 \text{ mg/mL})$ $\operatorname{area of contaminant} = \underbrace{\operatorname{m}}_{\mathrm{m}}$ $\operatorname{area of contaminant} \underbrace{4169}_{\mathrm{m}} - \operatorname{benzene adjustment} \underbrace{2522}_{2.522} = \underbrace{1646}_{4.560}$ $\operatorname{area of chlorobenzene} \underbrace{23442}_{-.01761}$ $\operatorname{concentration of contaminant} (\operatorname{mg/mL}) \underbrace{-.01761}_{-.01761}$ $\operatorname{calculated from graph equation x volume} \underbrace{\mathrm{mL}}_{-...} = \underbrace{\mathrm{mg_{contaminant}}}_{\mathrm{final mg_{contaminant}}}$ III. Parts per million

_____ final mg contaminant x 10⁶ /9847.0 mg air = _____ ppm

Calculations Worksheet

I. Air Quantity

Time 55 (min) x Pump Calibration 0.2089 (L/min) = 1.484 L 13.5396 13.5396 13.5396 13.5396 13.5396 13.5396 13.5396 13.5396 13.5396 13.5396

II. Contaminant Quantity

area_of contaminantconcentration=[area of chlorobenzene - b]of contaminantm

area of contaminant <u>534</u> - benzene adjustment ____ = ____ area of chlorobenzene <u>24.99</u>0

 $b = \Phi_{\underline{n}}$ m = 5.730concentration of contaminant (mg/mL) $\underbrace{\$.256}^{\times/0}$ calculated from graph equation

8. 256 mg/mL from graph equation x volume $\frac{0.00}{0.95}$ mL = 8.256 mg_{contaminant} 4. $\frac{4}{3.25610}$ mg_{contaminant} / desorbtion efficiency 0.95 = 8.6911 final mg_{contaminant}

III. Parts per million -4 $\textbf{g.bgM} = 10^{-4}$ final mg contaminant × 10⁶ / **13532** / mg air = 0.0642 ppm

Toluener Sample #2 Calculations Worksheet

I. Air Quantity

Time <u>45</u> (min) x Pump Calibration <u>0.2088</u> (L/min) = <u>9,396</u> L 9<u>,396</u> L x 1.179 g/L (as per ideal gas equation) = $l_{1,0}$ grams _{air} 11.0<u>179</u> grams _{air} x 103 = l_{10} mg _{air}

II. Contaminant Quantity

area of contaminantconcentration=[area of chlorobenzene - b]of contaminantm

area of contaminant <u>232</u> - benzene adjustment _____ = ____ area of chlorobenzene <u>2600</u>7

 $b = -\frac{1}{2}$ m = 5.730

concentration of contaminant (mg/mL) 3.447 calculated from graph equation

.4 3.447.0mg/mL from graph equation x volume 1.00 mL = 3.447.0mg .4 3.447.0mg_{contaminant} / desorbtion efficiency 0.95 = 3.428.20final mg_{contaminant}

III. Parts per million -4 3.<u>629 w</u>bfinal mg contaminant × 10⁶ /16.17.5 mg air = 0.0527 ppm

Tolune, Sample #3 **Calculations Worksheet**

I. Air Quantity

Time <u>30</u> (min) x Pump Calibration 0.203% (L/min) = <u>6.264</u> L

 $\frac{1}{2}$ L x 1.179 g/L (as per ideal gas equation) = 7. 503 grams air

7, 3853 grams air x 103 = 7385.3 mg air

II. Contaminant Quantity

area of contaminantconcentration=[area of chlorobenzene - b]of contaminantm

area of contaminant <u>434</u> - benzene adjustment ____ = ____ area of chlorobenzene <u>2581</u>

 $b = -\frac{1}{2}$ m = 5.730concentration of contaminant (mg/mL) $\frac{6.475}{6.475}$ calculated from graph equation

 $\frac{.4}{6.495 \times 10} \text{ mL from graph equation x volume } \frac{1.00}{1.00} \text{ mL } = \frac{.495 \times 100}{400} \text{ mg}_{\text{contaminant}}$

III. Parts per million -4 ($. < \frac{3}{2} \times 10^{6}$ final mg contaminant × 10⁶ / -1 365.3 mg air = 0.0928 ppm

I. Air Quantity

Time <u>40</u> (min) x Pump Calibration <u>0,2088</u> (L/min) = <u>8,357</u> L **8.352** L x 1.179 g/L (as per ideal gas equation) = <u>9,8470</u> grams _{air} **9,6470** grams _{air} x 10³ = <u>9847.0</u> mg _{air}

II. Contaminant Quantity

concentration = <u>[area of chlorobenzene - b]</u> (0.2214 mg/mL) of contaminant m

area of contaminant <u>7581</u> - benzene adjustment _____ = ____ area of chlorobenzene <u>2344</u>2

b = 0 m = <u>5.730</u>

concentration of contaminant (mg/mL)0.01250 calculated from graph equation

0.01250 mg/mL from graph equation x volume <u>1.00</u> mL = 0.01250 mg_{contaminant} 0.01250 mg_{contaminant} / desorbtion efficiency 0.95 = 0.01315 final mg_{contaminant}

III. Parts per million

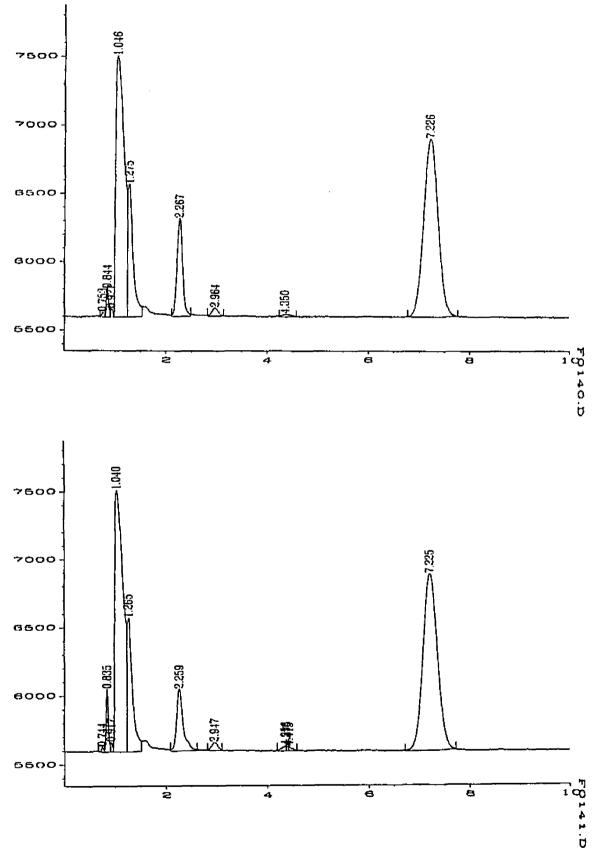
0.01315 final mg contaminant x 106 /9947.0 mg air = 1.334 ppm

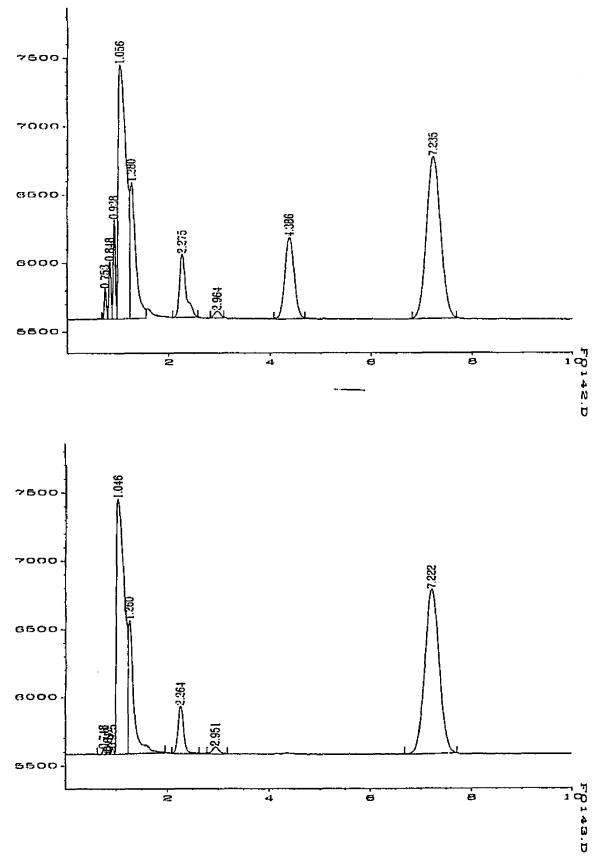
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APPENDIX B: Spectra and Data

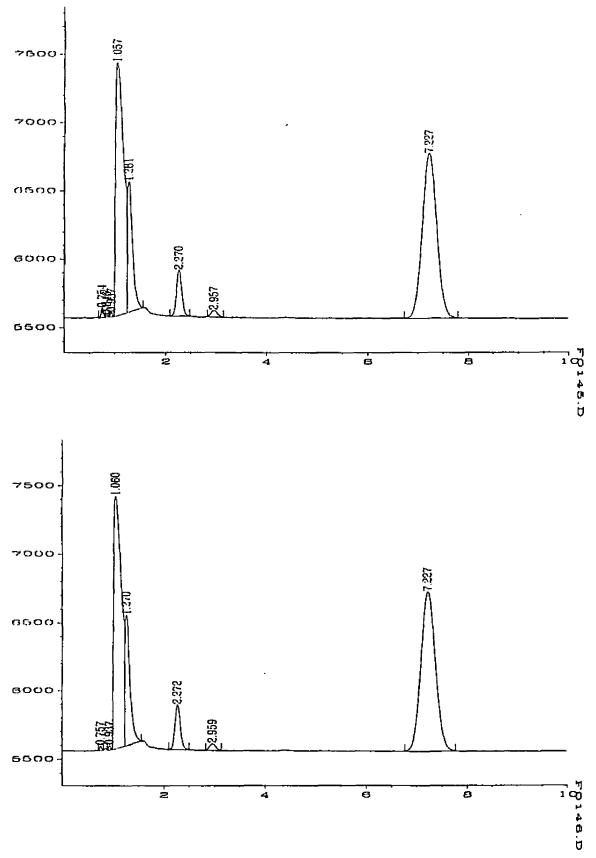
Run #	Description
F0139.D	first half of Sample # 1
F0140.D	<pre>first half of Sample # 2</pre>
F0141.D	first half of Sample # 3
F0142.D	first half of Sample # 4
F0145.D	second half of Sample # 1
F0146.D	<pre>second half of Sample # 2</pre>
F0147.D	<pre>second half of Sample # 3</pre>
F0148.D	<pre>second half of Sample # 4</pre>

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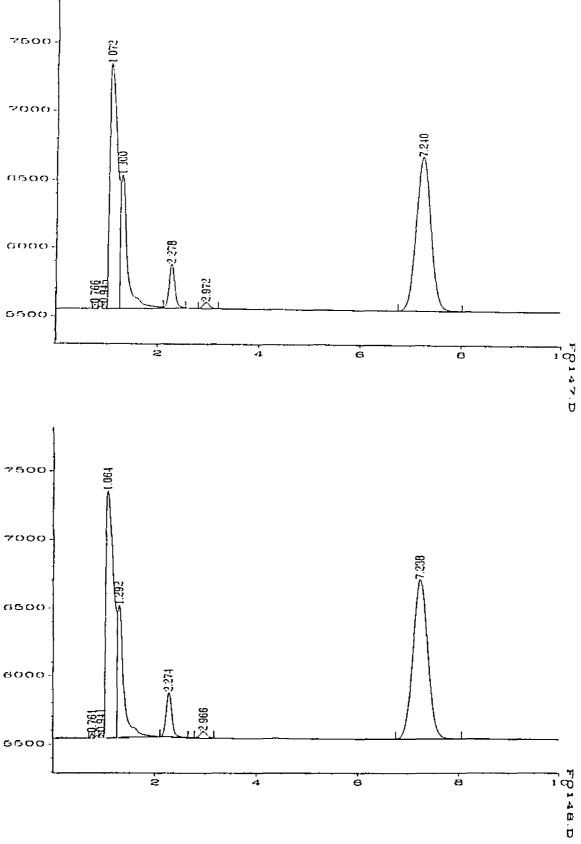


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End Time 0.806 0.975 1.523 3.122 7.862 7.865		- 6 - 6 - 6 - 6 - 6 - 6 - 6 - 6 - 6 - 6	1.240 2.479 2.479 4.559 7.762
Start Time 0.669 0.896 0.892 0.975 1.238 1.238 2.034 2.034 6.719 6.719		rt . 49 . 49	0.838 0.972 2.109 2.806 4.230 6.775
Area 7818 852 852 20716 5511 84348 509 24990	F0140.D	a o n o	21753 21753 5455 512 232 232
Width 0.043 0.050 0.164 0.116 0.116 0.116 0.283 0.283		idt. 04	0.157 0.157 0.099 0.114 0.114 0.290
Type PV VV VV VV VV VV VV VV		Туре РV VV	
Ret Time 0.731 0.837 1.044 1.267 2.259 2.935 7.208 7.208		Tim .75 .84	0.927 1.046 2.267 4.350 7.226
₽ ● 3 ● 3 2 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5		peak# 1 2	ოფიადი ფი ოფიადი იი ოფიადი იი ოფიადი იი იი იი იი იი იი იი იი იი იი იი იი იი

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End Time 0.780 0.898 0.957 1.232 2.609 4.410 7.725 7.725	End Time 0.802 0.988 0.988 1.242 1.555 1.555 3.086 4.686 7.691
Start Time 0.665 0.780 0.957 0.957 1.232 2.809 4.188 4.410 6.719 6.719	Start Time 0.689 0.802 0.988 1.242 2.085 2.829 4.072 6.809
Area 104 1375 1758 6795 3991 203 203 203 203 203 25819	·F0142.D Area 646 1233 2072 20327 7903 412 412 7581 23442
Width 0.041 0.046 0.157 0.157 0.127 0.038 0.038 0.038 0.239	Width 0.045 0.046 0.152 0.152 0.114 0.129 0.245 0.245
$\mathbf{F}_{\mathbf{F}}^{T}$	Type VV VV VV VV VV VV VV VV VV VV VV VV VV
Ret Time 0.744 0.835 1.040 1.265 4.353 4.375 7.225 7.225	Ret Time 0.753 0.938 1.056 1.280 2.275 2.275 7.235 7.235
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End Time 0.803 0.873 0.975 1.229	1.955 2.625 3.179 7.719	טיי דירי ניי	00		10 0	3.139
Start Time 0.622 0.803 0.873 0.975	. 22	Start Time	0.68 0.80	0.888 0.981	1.242 2.086	2.825 6.727
Area 124 132 132 20427	7375 2696 480 23985	·F0145.D Area	215	94 20250	6675 2499	410 24087
Width 0.049 0.036 0.156	282.	Wiđth	0.038	<u><u></u></u>	<u>н</u> н	
Type PV VV VV	BV VV VV	Туре	PV VV	VV Vq	V V V	BV PV
Ret Time 0.748 0.842 0.925 1.046	522	Ret Time	0.7	0.937 1.057	.28	.22
Ре а 4 у у 2 н 4 4 у 2 н 4	ら り り の	Peak#	10	ጣላ፣	ഗഗ	78

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Ret Time 0.766 0.945 1.072 1.300 2.278 2.972 7.240		7.227	2,959	1.270	1.060	0.937	0.757	Ret Time
Type VV BV BB		Υ.	PV PV	N N	ΡV	V	BV	Туре
Width 0.049 0.159 0.159 0.130 0.120 0.121 0.310		0.283	0.106	0.096	0.157	0.043	0.043	Width
Area 113 19422 9187 2490 420 22801	F0147.D	23243	4 4 4	6441	5	06	56	Area
Start Time 0.709 0.901 0.996 1.254 2.112 2.802 6.752		6.761	2.822	1.236	0.984	0.889	0,709	Start Time
End Time 0.824 0.996 1.254 2.112 2.551 3.205 8.019		. 76	2.499 3.129	ათ ით	.23	8	.81	End Time

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End Time	0.812	0.988	1.249	2.099	2.655	3.169	8.062
	0.692						
Area	101	114	19780	8614	2469	429	23426
Width	0.047	0.047	0.161	0.124	0.117	0.124	0.316
Type	ΒV	22	Ŵ	VB	BB	BB	BB
Ret Time	0.761	0.941	1.064	1.292	2.274	2.966	7.238
Peak#	r-4	ы	m	দ	ŋ	9	7

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Air Samples Calculation Information

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Chlorobenzene Concentration	6 LCC 0	10 0 0115 ELS	5177.0 V 7172	81777.0 V 7777	4 C 4 C	•	1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1	0.2214	.221 .221 .221
Benzene Area	84348	54	σ	4168	2499	2424 2424	7400	2469	2696 2506 2569
Chlorobenzene Area	24990	26007	25819	23442	24087	324	28	342	23985 23520 24639
Run #	139	140	141	142	145	146	147	148	bl 143 bl 136 bl 149

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APPENDIX C: Overhead Transparency Masters

Health Hazards

(Illness)

6 Classes

Corrosive Irritant Sensitizer Carcinogen Acute Toxicity Organ Damage

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Physical Hazards

(Injury)

9 Classes

Combustible Liquid Compressed Gas Explosive Flammable Organic Peroxides Oxidizer Pyrophroic Unstable (Reactive) Water Reactive



CODE OF FEDERAL REGULATIONS TITLE 29 OCCUPATIONAL SAFETY & HEALTH ADMINISTRATION

CODE OF FEDERAL REGULATIONS TITLE 40 ENVIRONMENTAL PROTECTION AGENCY

CODE OF FEDERAL REGULATIONS TITLE 49 DEPARTMENT OF TRANSPORTATION

OSHA	DOT	EPA
EMPLOYER EMPLOYEE HAZARDOUS CHEMICALS HAZARDOUS MATERIALS WORKER RIGHT-TO-KNOW	SHIPPERS CARRIERS DRIVERS HAZARDOUS MATERIALS HAZARDOUS SUBSTANCES "RQ" IN ONE CONTAINER	GENERATORS TRANSPORTERS TREATMENT, STORAGE & DISPOSAL FACILITY UNCONTROLLED HAZARDOUS WASTE SITE COMMUNITY RIGHT-TO-KNOW HAZARDOUS WASTE HAZARDOUS SUBSTANCES "RQ" DISCHARGES

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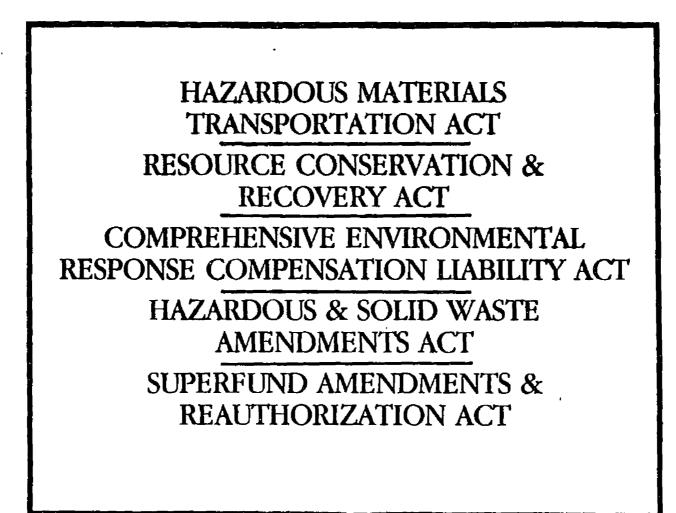
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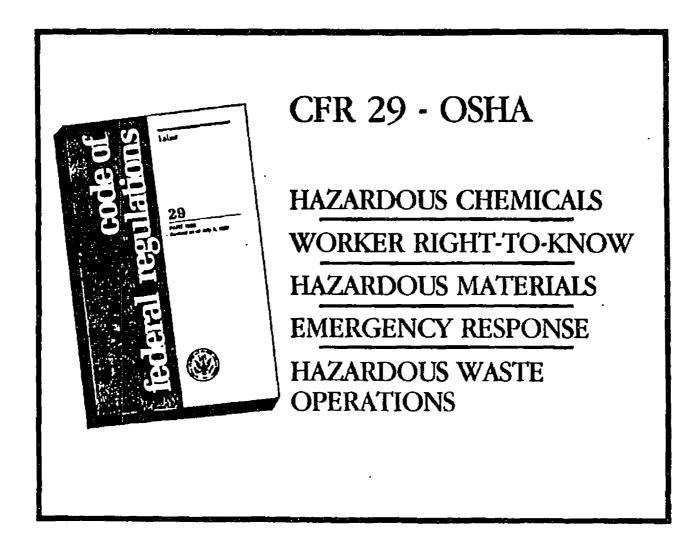
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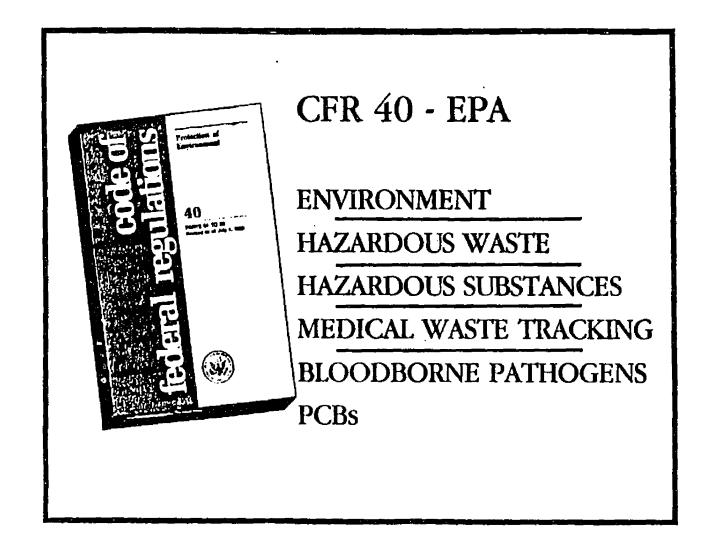
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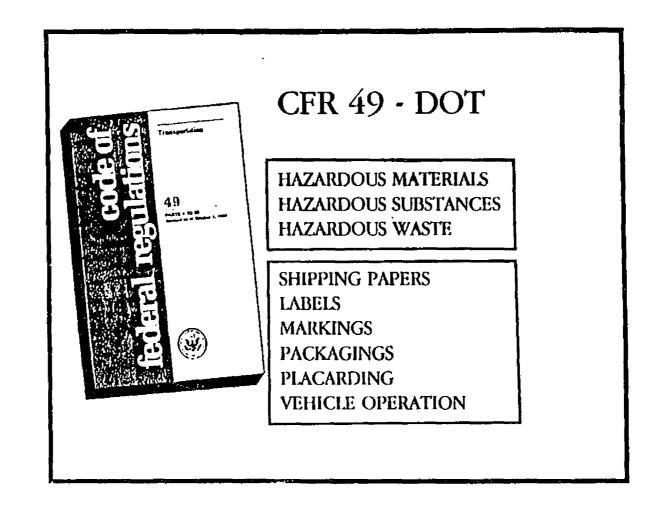
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MANDATORY REGULATORY TRAINING					
DOT	OSHA	EPA			
SHIPPERS/GENERATORS 173.1 CARRIERS/TRANSPORTERS 177.800 DRIVERS 177.800 390/399 ANY PERSON IN PROXIMITY TO HAZARDOUS MATERIALS & WASTE 172.700 - 1910.120	EMPLOYEES/EMPLOYERS WORKER RIGHT-TO-KNOW 1910.1200 HAZARDOUS WASTE OPERATIONS EMERGENCY RESPONSE 1910-120 1926 1910.1200	GENERATORS TREATMENT, STORAGE & DISPOSAL FACILITY 265.16 COMMUNITY RIGHT-TO-KNOW 355 BLOODBORNE PATHOGENS			

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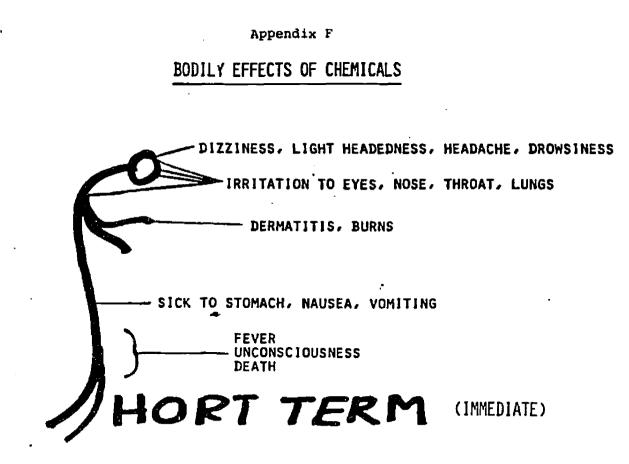
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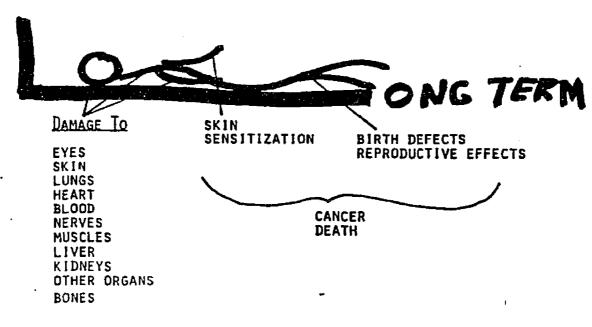
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HAVE A HEALTHY RESPECT FOR CHEMICALS

Flinn Scientific, Inc. MATERIAL SAFETY DATA SHEET

CHEMICAL NAME & SYNONYMS			FLINN CATALOG NUMBER		
MALONIC ACID (PROPANEDIOIC ACID)		M0091	M0091		
FORMULA CH2(CO2H)2	FORMULA OR ATOMIC WEIGHT CAS NU 104,07 141-8				
Physical OATA (DENSITY, SOLUBILITY, ETC.) Specific Gravity: 1.63 Melting Point: 132-134 C Soluble: water, alcohol, ether.					
APPEARANCE AND ODOR While crystal. Acetic acid odor.	<u> </u>				
COMPATIBLE CHEMICAL FAMILY	DOT CLASS	REACTIVITY	REACTIVITY		
Organic #1	Not Regulated		Stable		
See Finn Chemical Catalog/Relevance Manual CONDITIONS TO AVOID (IF ANY):					
Avoid contact with strongly alkaline subs HEALTH HAZAROS (IF ANY): Strong irritant; moderately toxic. This sub some states. orl-rat LDSO: 1310 mg/kg. Not all health aspects of this substance hav HRE HAZAROS (IF ANY): Non combustible.	stance is regulated (as a drug in	uermediate) in	THRESHOLD LIMIT VALUE (TLV) IF ESTABLISHED None Established		
SPILLS AND LEAKS: Gather up material in a pile (do not sweep). disposal method listed on the right.	Place in a suitable container. U	ise the	DISPOSAL METHOD 24a See Finn Chemical Catalog Reference Manual		
SPECIAL PRECAUTIONS (IF ANY): Wear chemical gloves and goggles. Prudent laboratory practices should be observed. Material should be stored in a cool, dark place. Check your local regulations since, in some states, malonic acid is a controlled substance.					
FIRST AID (IF SUBSTANCE DANGEROUS); External: Wash affected parts with copion Internal: Wash mouth; see a physician.		for 15 minutes.	See a physician.		
Consult your copy of the Flinn Chemical Catalog/Reference Manual					
	for even more information about laboratory chemicals				

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