Environmental Control in Semiconductor Manufacturing

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Semiconductor manufacturing is among the world's most important and technologically advanced industries. Although it once enjoyed a reputation as a "clean" industry, in recent years that reputation has been tarnished by allegations of environmental, worker health, and human reproductive problems stemming from semiconductor manufacturing operations. It is well-documented[1-3] that making semiconductor devices requires hazardous chemicals that, if not properly used or controlled, could result in serious problems to the facility, its work force, or the community where it exists. This paper first discusses early attempts at semiconductor manufacturing, then tracks technology, early environmental controls, growth in regulatory requirements, and programs implemented to insure regulatory compliance in all areas of environmental concern.

Introduction

AT&T Microelectronics' Allentown Works, opened in 1947, is AT&T's oldest existing manufacturing facility. Despite its age, it has long enjoyed the role of leading-edge manufacturing facility, first for electron tubes and switches, then for transistors and thin film devices, and currently for sophisticated silicon integrated circuits. From Allentown's earliest days as a Western Electric production site, both proper handling of hazardous materials and treating the process wastes they generated were high priorities. Though the original treatment processes might seem archaic by current standards, at the time they represented the best in environmental engineering. As long ago as the late 1940s, Allentown operated a wastewater treatment facility that could neutralize acid wastes and oxidize cyanide wastes from electroplating operations.

Allentown was the first AT&T manufacturing location to see the need to consolidate existing environmental health and safety functions, and then restructure them in a single organization with overall responsibility for these issues. This reorganization proved timely in two respects.

- It helped Allentown bring environmental, health, and safety issues into focus at a time when there was not much regulatory pressure.
- When Federal legislation was enacted (The Clean Air Act Amendments of 1970, the Occupational Safety and Health Act of 1970, and the Clean Water Act of 1972), and regulations were promulgated, Allentown was in a particularly strong position to comply.

Allentown was years ahead of the semiconductor industry in implementing environmental, health, and safety programs and controls that anticipated future requirements. Several examples of operations and facilities in Allentown during the late 1960s and early 1970s included:

- Segregation and on-site treatment of fluoride wastes
- Segregation of solvent wastes
- Reclamation and beneficial use of solvent wastes
- Breakpoint chlorination of ammonia wastes
- Introduction of polymer coagulants to reduce sludge volume produced during wastewater treatment
- Process stack sampling
- Installation of wet scrubbers on process exhausts
- Internal review of new processes and new chemical use, including factors such as suitability, compatibility, allowable

Panel 1. Terms and Acronyms in This Paper

AA	atomic absorption spectroscopy
CFC	chlorofluorocarbon
GC	gas chromatography
GC-IR	gas chromatography-infrared spectroscopy
GC-MS	gas chromatography-mass spectrometry
IC	ion chromatography
ICP	inductively coupled plasma
IR	infrared spectroscopy
mg	milligram
L	liter
MS	mass spectrometry
NPDES	national pollutant discharge elimination system
EPA	Environmental Protection Agency (United States government)

quantities, toxicity, flammability, treatment, and disposal
- Establishing administrative controls for hydride use, such as maximum allowable quantities, outside supply, and solid sources as a replacement for gases
- Design, development and production of a standard gas panel by Allentown engineers to provide safe distribution of hazardous gases.

Much has changed since the early days of semiconductor manufacturing, especially in the regulatory environment. In this regard, the proliferation of environmental regulations has far outpaced the development of new environmental control technologies. With increased new regulations, the role, responsibility, and perceived importance of environmental health and safety organization also has greatly expanded. Also, manufacturing technology that depended on wet chemistry—especially for cleaning and etching—now uses sophisticated processes such as low pressure chemical vapor deposition, ion implantation, and plasma etching. These processes—most notably plasma etching—have greatly reduced the quantities of waste generated while simultaneously creating new problems associated with handling toxic, pyrophoric, and corrosive by-products. Finally, pollution prevention is replacing end-of-pipe treatment as the primary means of environmental control. The emphasis is now on reducing hazardous material usage, substituting chemicals, and recovering waste products for later use.

This paper describes the programs an advanced producer of semiconductor devices requires to keep pace with the current manufacturing and regulatory requirements. Specifically, it focuses on four program areas:
- Wastewater treatment
- Hazardous waste management
- Air pollution control
- The environmental engineering laboratory.

Wastewater Treatment

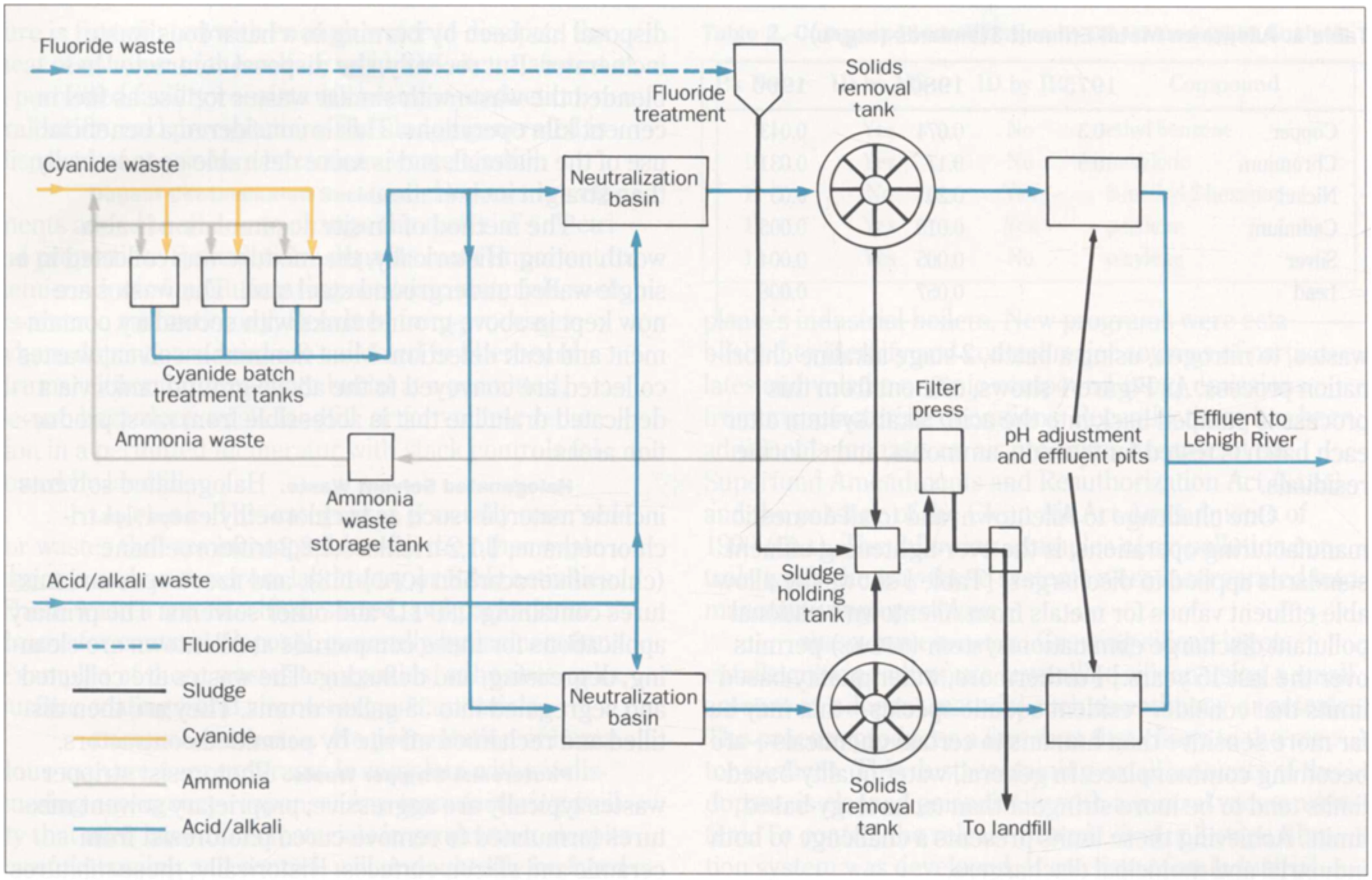
The Allentown Works wastewater treatment plant treats inorganic wastes generated from manufacturing operations that include:
- Lapping, etching, and polishing of silicon wafers
- Wet etching of silicon oxides, and cleaning during wafer fabrication
- Dry (plasma) etching of surface layers during wafer fabrication
- Inorganic acid stripping of photoresists before metallization, during wafer fabrication.

The treatment process consists of this series of interconnected unit operations:
- Neutralization
- Batch fluoride treatment
- Cyanide/ammonium oxidation
- Metals precipitation
- Solids removal
- Sludge drying.

Figure 1 presents a flow diagram of the treatment system. Note that each principal influent stream is conveyed to the treatment plant via a separate pipe to a distinct process. After the appropriate treatments, the waste streams are mingled for additional treatment before discharge into the Lehigh River. A singular feature of the plant is its ability to treat concentrated fluoride wastes (hydrofluoric acid and ammonium fluoride-hydrofluoric acid) and ammonium wastes.[4]

Acid and alkali wastes are processed through a continuous flow-through system with two identical interconnected flow trains that can each handle the entire waste flow hydraulically. The goal is to provide neutralization, metals precipitation, and solids removal even if one train is lost. Hence, three unit operations—neutralization, clarification, and final pH adjustment—are provided.

The chemicals used include lime, sodium hydroxide, sulfuric acid, polymeric coagulants, and polymeric flocculants (long chain polymers developed to



help increase the size of particles formed in coagulation). The polymers help agglomerate charged colloidal precipitates by surface charge-reduction and bridging mechanisms. (*Bridging* is a process by which colloidal particles are attracted to the surface of the polymer.) Most flocculation takes place in the rapid mix tank at the center of each clarifier unit. The clarifiers are circular, upflow units designed to maintain and use a fluidized bed of sludge called a "sludge blanket." Periodically, sludge generated during waste treatment is removed from each clarifier and is transferred to a holding tank to balance the amount generated during treatment. The last treatment step for the acid and alkali waste stream is final pH adjustment to assure that the effluent does not exceed a pH of 8.0.

Fluoride wastes are batch-treated in one of two available 1,000 gallon plastic lined tanks, using a high-lime precipitation process. Like the acid/alkali treatment, polymers aid the coagulation and flocculation processes;

Figure 1. A flow diagram for the treatment system. Each principal influent stream is conveyed to the treatment plant via a separate pipe to a distinct process. After the appropriate treatments, the waste streams are mingled for additional treatment before discharge into the Lehigh River. The plant is able to treat concentrated fluoride wastes and ammonium wastes.

sulfuric acid is used to adjust the pH level. The sludge generated—primarily calcium fluoride—is transferred to a holding tank where it is commingled with sludge from the acid and alkali clarifiers. This sludge is then pumped to the plate and frame filter press for dewatering. The partially dewatered sludge—about 50 percent solids—is hauled to a permitted landfill for disposal. The dewatering process produces a filtrate that is rich in ammonia from ammonium fluoride. It is piped to another holding tank, and is pumped to the cyanide treatment system where it is oxidized along with small amounts of cyanide

Table 1. Allentown Metal Effluent Standards (mg/L)

	1975	1980	1990
Copper	0.3	0.074	0.043
Chromium	0.5	0.17	0.031
Nickel	-	0.20	0.05
Cadmium	-	0.010	0.005
Silver	-	0.005	0.004
Lead	-	0.057	0.008

wastes, to nitrogen, using a batch, 2-stage alkaline chlorination process. As Figure 1 shows, effluent from this process is pumped back into the acid/alkali system after each batch is tested for cyanide, ammonia, and chlorine residuals.

One challenge to Allentown, and to all domestic manufacturing operations, is the ever-tightening effluent standards applied to discharges. [Table I shows the allowable effluent values for metals from Allentown's national pollutant discharge elimination system (NPDES) permits over the last 15 years.] Furthermore, water quality-based limits that consider resident aquatic species—that may be far more sensitive than humans to certain chemicals—are becoming commonplace. In general, water quality-based limits tend to be more stringent than technology-based limits. Achieving these limits presents a challenge to both industrial and municipal dischargers.

Hazardous Waste Management

Although a hazardous waste management program has existed at Allentown since 1971, the enactment of the Resource Conservation Recovery and Act (RCRA) has led to more formalized control of these wastes. The Pennsylvania Department of Environmental Resources (PaDER) and the United States Environmental Protection Agency (EPA) carefully scrutinize how these wastes are managed, how they are classified, where they are accumulated before disposal, how they are shipped, where they are sent for disposal, and how they are accounted for during the process. What follows describes the categories of wastes at Allentown, their principal sources, and the means to handle and dispose of them.

Flammable Solvent Waste. This waste is a heterogeneous mixture of aliphatic, aromatic, and chlorinated solvents, along with oils and other combustible liquids. The principal operations that produce these wastes are photolithography and surface cleaning. Since 1971, disposal has been by burning in a hazardous waste incinerator. But recently, the disposal contractor has blended the waste with similar wastes for use as fuel in cement kiln operations. This is considered a beneficial use of the material, and is more desirable economically than straight incineration.

The method of on-site accumulation is also worth noting. Historically, the mixture was collected in a single-walled underground steel tank. The wastes are now kept in above-ground tanks with secondary containment and leak detection. Most flammable solvent wastes collected are conveyed to the above-ground tanks via a dedicated drainline that is accessible from most production areas.

Halogenated Solvent Waste. Halogenated solvents include materials such as trichloroethylene, 1,1,1 trichloroethane, 1,1,2-trichloro,1,2,2-trifluoroethane (chlorofluorocarbon [CFC]-113), and azeotropes and mixtures containing CFC-113 and other solvents. The primary applications for these compounds at Allentown are cleaning, degreasing, and defluxing. The wastes are collected and segregated into 55-gallon drums. They are then distilled and reclaimed off-site by permitted contractors.

Photoresist Stripper Waste. Photoresist stripper wastes typically are aggressive, proprietary solvent mixtures formulated to remove cured photoresist from ceramic and silicon surfaces. Historically, these mixtures contained aromatic and chlorinated solvents as well as detergent wetting agents. These materials were generally acidic and corroded steel; this meant they could not be drained into the flammable solvent waste tank. Thus, the disposal procedure had been to collect these materials in plastic-lined drums for burning at a permitted facility. In recent years, new photoresist strippers have been introduced that do not have the same properties as the original strippers. Several of these new formulations are non-corrosive and are compatible with the waste flammable solvents. At least one material used is recycled in concentrated form by the supplier.

Chromic Acid Waste. Chromic acid waste is derived from two production operations. In one, a mixture of chromic acid and hydrofluoric acid is used to find defects in the crystalline structure of newly grown silicon ingots. In the other, a mixture of chromic acid and sulfuric acid is used to clean chrome on glass masks. Both wastes are conveyed via a common drainline and are commingled in a common tank. The chrome/sulfuric mixture is first diluted with enough water to dissipate the heat of solution of the sulfuric acid. Off-site treatment at a permitted facility consists of chromium reduction, neutralization, and precipitation. The sludge generated is disposed of at a secure hazardous waste landfill.

Dopant Contaminated Residues. Dopants are elements added to silicon to change the material's electrical properties. Typically, the dopants most important to semiconductor manufacturing are toxic materials such as arsenic and antimony. Residues from processes where dopants are used are collected in fiberboard drums, either for immediate burial at a permitted, secure hazardous waste landfill, or for volume reduction in a permitted incinerator with stack controls followed by landfill.

Lab Packs. This category is generally reserved for wastes that are incompatible with the other waste streams and wastes from laboratory or R&D activities. These wastes are classified, sorted, and packed into drums for contract disposal, generally by incineration. Examples of these wastes are epoxies, adhesives, inks, surface coatings, and viscous organic materials.

Emergency Response. No presentation of hazardous management programs is complete without discussing emergency response. Any manufacturing facility that uses hazardous materials in production, despite careful planning and control, will undoubtedly be faced with the task of dealing with spills or leaks. Securing areas where these have occurred, containment, implementing proper cleanup procedures, and residue disposal, becomes the charge of trained volunteers on the emergency response, or hazardous materials (HAZMAT), team. The team receives hazardous material cleanup training, and appropriate personal protective equipment including respirators, boots, and chemical-resistant coveralls. The Allentown HAZMAT team—on call 24 hours a day, every day of the year—is vital to insure that spills and leaks are controlled quickly. Its actions keep employees from being unnecessarily exposed to toxic materials or hazardous conditions, and minimize potential property damage.

Air Pollution Control

Allentown's air pollution control program was established in the late 1960s to meet Pennsylvania's requirements. Initial efforts were directed at controlling emissions of sulfur oxides and particulates from the plants's industrial boilers. New programs were established to identify and control major sources of particulates and volatile organic compound (VOC) emissions from manufacturing operations. Recently, there has been additional emphasis on air programs because of the Superfund Amendments and Reauthorization Act (SARA), and the passage of the Clean Air Act Amendments of 1990 (CAA). The following examples of air pollution controls typify the way these systems were incorporated into manufacturing operations.

Table 2. Compound Identification by GC-MS and GC-IR Analysis

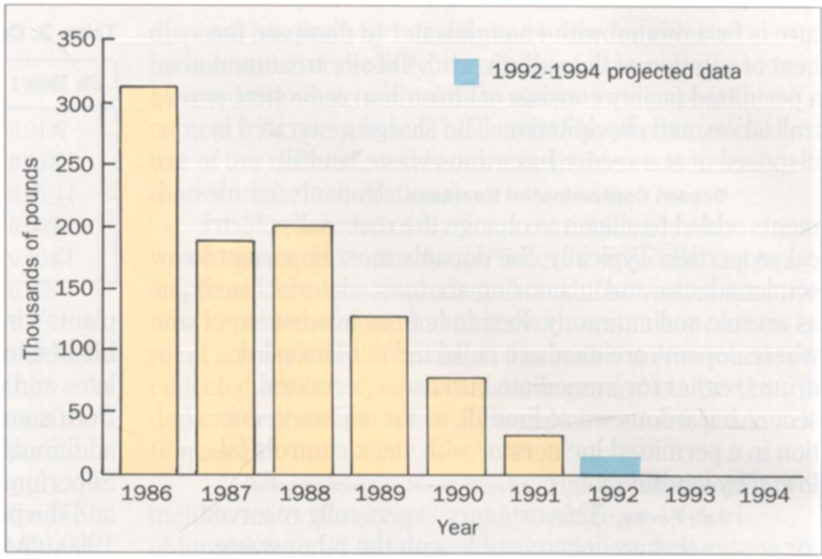
Pk No.	ID by MS	ID by IR	Compound
9	Yes	No	ethyl benzene
10	Yes	No	m-xylene
11	No	Yes	5-methyl-2-hexanone
12	Yes	Yes	p-xylene
13	Yes	No	o-xylene

Silicon Ingot Growing. Growing silicon ingots entails growing ultrapure crystalline silicon using a small amount of a dopant material such as antimony or arsenic. The process produces a fine dust that clings to the reactor surfaces. This dust, containing small amounts of the dopant, is cleaned periodically with a central vacuum system. To control the release of toxic dust, a 3-stage filtration system was developed. It consists of an industrial vacuum/bag housing, a particle filtration system, and a high efficiency particulate air (HEPA) filter to remove submicrometer particles.

Epitaxial Deposition. Epitaxial deposition involves growing thin layers of monocrystalline silicon on the surface of silicon wafers. At first, silicon tetrachloride was the source of silicon. This was switched to dichlorosilane in the 1970s and to trichlorosilane in the 1980s. While the source material has varied since then, the byproducts have remained hydrogen chloride and a polymeric form of silicon believed to be a variety of polysiloxanes. Since the 1960s, these materials have been scrubbed with water. At first, horizontal, packed, cross-flow units, designed to handle multiple reactors, were used. When the silicon materials production areas were moved to new quarters in the early 1980s, baffle-type wet scrubbers were installed, one per reactor.

Plasma Etching. In many respects, plasma etching represented a major improvement over the wet chemistry it replaced. Fewer reactants were needed in the

Figure 2. Graph showing reduction in CFC emissions at the Allentown Works, beginning in 1986. Projections of future reductions are shown in the unfilled areas. It is expected that all CFCs will be eliminated by the end of 1992.



plasma process than in a process using wet etching with acids. This minimized handling and disposal problems by trading substantial quantities of bottled or drummed chemicals for small cylinders of plasma source gases. If there was a negative environmental aspect to plasma etch, it was that the plasma by-products, especially those from the early source materials, were noxious, corrosive, and toxic. To protect the vacuum pumps and pump oils from attack, liquid nitrogen freeze-out traps were incorporated. However, cleaning these traps required an exhausted work station and extreme caution to ensure workers would not be exposed to the byproducts.

The solution to this problem came in several steps in the late 1970s. First, a fluorinated corrosion-resistant vacuum pump oil became available. New, inorganic source gas recipes with more predictable by-products for etching were developed in AT&T Bell Laboratories. Finally, individual wet scrubbers were brought into the plasma process to remove corrosive by-products from the exhaust stream.

Current Air Issues

By the late 1980s, the weight of scientific evidence pointed to CFCs as a major contributor to stratospheric ozone depletion. Clearly, air pollution—particularly toxic emissions into the atmosphere—was high on the Congressional agenda, and this belief was confirmed with the passage of the 1990 Clean Air Act Amendments. Provisions to phase out CFCs and reduce the emission of toxics and volatile organic compounds were of special importance to manufacturing facilities such as Allentown.

Because of AT&T's environmental policy and its local implementation, Allentown is advanced in eliminating CFC usage and reducing solvent emissions. The key initial element is preparing a comprehensive chemical use and emission inventory that will help identify major users, focus attention on key operations, and help rank the programs.

The Allentown CFC strategy has two phases:
- Improved housekeeping and conservation
- Developing an alternative materials program.

Improved housekeeping includes enhanced maintenance, leak detection, checking exhaust ventilation, keeping covers on chemical baths during idle periods, and turning off sump heaters in degreaser units during idle periods. In the second phase, alternative materials and processes have been researched, developed, and incorporated into Allentown's business plan for future funding. Allentown's emission reduction program has three phases:
- Maximize processes to reduce emissions
- Develop alternative materials programs
- Use exhaust abatement techniques.

For the last phase, Allentown is evaluating thermal oxidizing systems to reduce emissions from solvent operations. Other processes being considered include adsorption, dry scrubbing, and chemisorption.

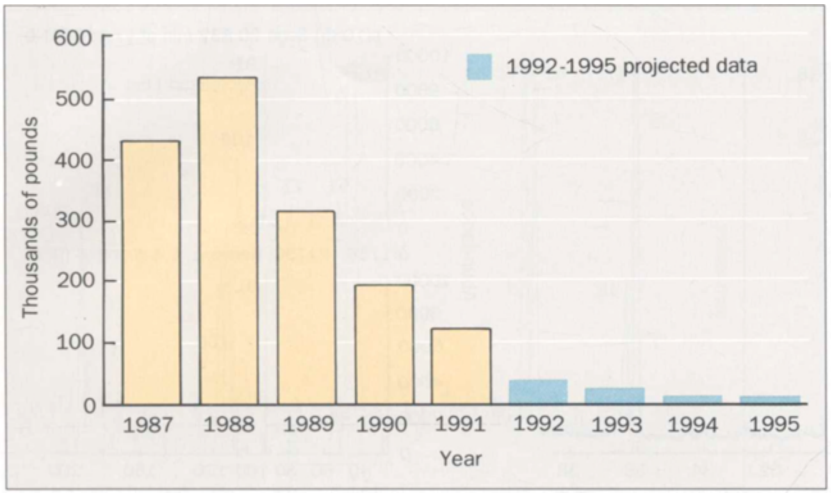


Figure 3. Graph showing reduction in toxic air emissions at the Allentown Works, beginning in 1987. Projections of future reductions are shown in the unfilled areas. It is expected that the poundage volume of toxics emitted to the air will be significantly reduced by 1995.

Significant progress has been achieved to reduce both CFC and toxic air emissions from Allentown processes. These efforts are summarized in Figures 2 and 3, showing the reductions achieved to date.

Environmental Engineering Laboratory

Allentown has an Environmental Engineering Laboratory that is central to the Environmental Health and Safety Organization. The laboratory, staffed with two environmental chemists, is certified by the American Industrial Hygiene Association (AIHA) to do solvent vapor and metals analyses. The laboratory's primary function is support of environmental health and safety programs at Allentown. However, because it is the only AT&T facility with AIHA certification, Allentown also performs analyses, particularly of industrial hygiene, for other AT&T facilities. The laboratory serves two principal functions.

- It enables ongoing, in-house identification and characterization of wastes and other collected samples.
- It can analyze samples *while* an incident is occurring.

Analytical Instrumentation. The Allentown Environmental Engineering Laboratory is an advanced facility for analyzing both large volumes of similar samples and specialized samples requiring individual attention. A variety of analytical instrumentation is used by the laboratory staff to aid in sample analysis. Of particular note are the following:

- Fourier transform infrared spectroscopy to fingerprint materials and identify functional chemical groups
- Gas chromatography (GC) to separate mixtures of volatile organic compounds and identify their components by retention time. Detection is achieved through flame ionization and electron capture; and the signal response is quantified by electronic integration.
- Gas chromatography-mass spectrometry (GC-MS) to separate volatile organic compounds and identify components by mass spectrometry, using a 60,000 compound, on-line library of mass spectra.
- Gas chromatography-infrared (GC-IR) to separate volatile organic compounds and identify components by infrared spectroscopy, using an EPA, on-line 3,000 compound library.
- A purge and trap concentrator to strip and concentrate trace amounts of volatile organic compounds from aqueous samples, sludges, or solids for subsequent gas chromatographic analysis.
- Atomic absorption spectrometry (AA) to identify and quantify specific metals in solutions or dissolved solids.
- Inductively coupled plasma (ICP) to identify total metal content of a material in a single spectral distribution.
- Ion chromatography (IC) to identify and quantify particular organic or inorganic ions.

Waste Treatment Plant. The laboratory provides analytical support for the waste treatment plant. One example is the laboratory's job of verifying the status of a particular treatment process by identifying reactants or by-products. Another is administering quality control programs for treatment plant operators to assure proficiency in analyzing for process residuals. The laboratory has been an important contributor to Allentown's effort to classify low levels of metals in water supply and effluent lines.

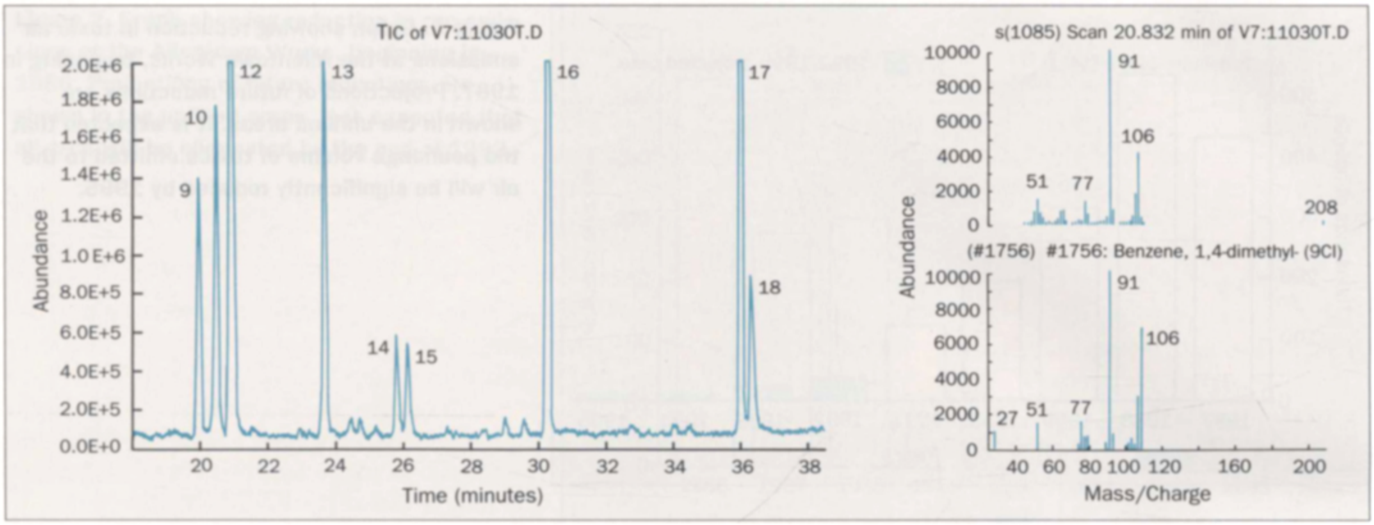


Figure 4. A partial gas chromatogram of a waste flammable solvent analyzed under the same gas chromatographic conditions by GC-MS and GC-IR, showing the MS generated, total ion chromatogram and the mass spectum for the identified peak number 12 with the library match for xylene.

Hazardous Waste Disposal. The laboratory routinely analyzes all classified waste streams to insure that the material conforms to the description provided to the disposal contractor. Also, materials of unknown origin or composition are given to the laboratory for analysis using the equipment identified above. Analysis of materials suspected of containing polychlorinated biphenyls (PCB) falls into this category.

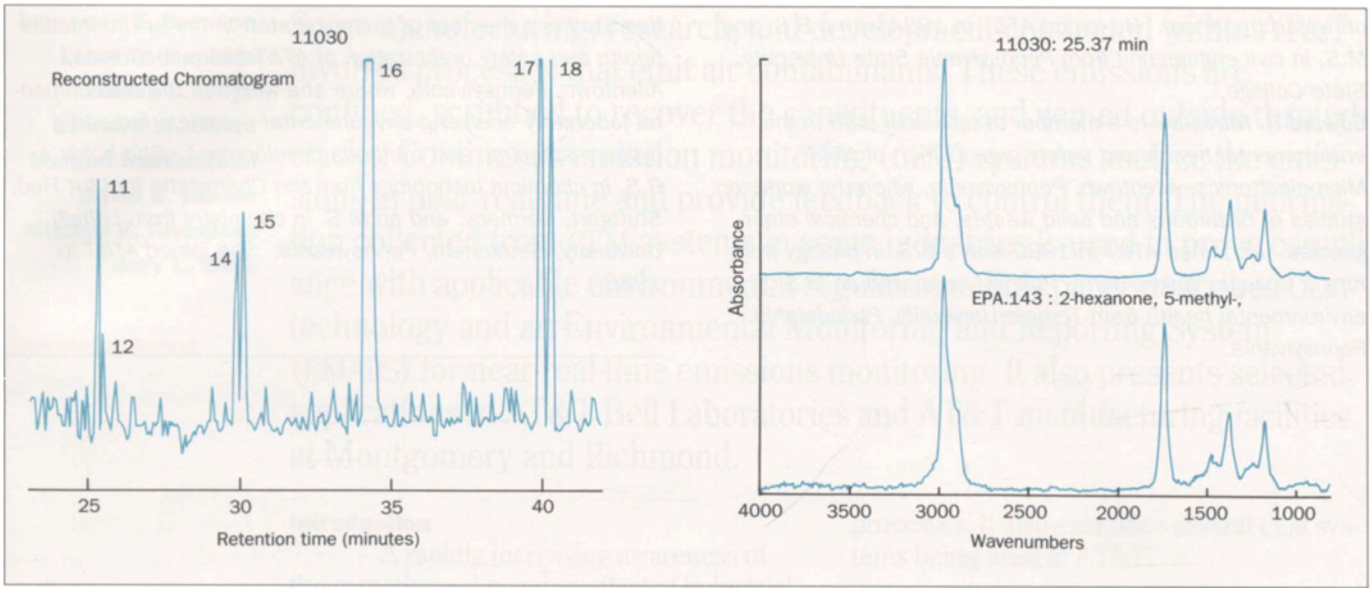
To illustrate the application of this instrumentation, Figures 4 and 5 show a partial chromatogram of a waste flammable solvent. Figure 4 shows the sample analyzed by GC-MS, and Figure 5 shows it analyzed by GC-IR under the same gas chromatographic conditions. Similarities are shown between peaks 14-18 on the two chromatograms. Peaks 9, 10, 12, and 13—identified by the MS library as ethylbenzene, and the three xylene isomers—showed strongly in the MS analysis (Figure 4), whereas only peak 12 was detected by the IR analysis. However, peak 11 was only detectable by IR (Figure 5) and did not show in the MS analysis. The IR library identified this peak as 5-methyl-2-hexanone (Methylisoamylketone). The combined compound identification is shown in Table 2. This agrees with the common findings that aromatic compounds, such as xylenes, show strong response by MS and weak response by IR; whereas ketones show strong response by IR and weak response by MS. This illustrates the use of a combination of GC detectors to obtain the information desired.[5]

Air Pollution Control. The laboratory identifies components of process stacks and quantifies the emissions. Furthermore, the laboratory has helped evaluate the removal efficiencies of air scrubbers.

Industrial Hygiene. The laboratory supports the industrial hygiene program at Allentown and other AT&T locations by providing timely, thorough analyses of work area samples collected by industrial hygienists throughout AT&T on adsorbents such as activated carbon, filters, silica gel, and polymer beads. These samples are used to evaluate workplaces for potential exposures to toxic materials, and to assure compliance with governmental regulations. The laboratory develops new analytical methods where a method does not currently exist.

Conclusion

It is an understatement to say the importance and complexity of environmental engineering programs at manufacturing facilities have grown over the last two decades. Today's environmental engineers must not only be aware of the latest technologies, but must also understand complex and often contradictory local, state, and federal regulations. They must understand manufacturing technologies and be able to communicate with both process engineers and the research community. Today's environmental control program must be flexible and



responsive to major, often rapid change both in the manufacturing processes as well as the rules that dictate how manufacturing will be conducted. Such a program must be well-directed, well-staffed, well-focused, goal-oriented, and supported by local management.

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Figure 5 also shows the partial gas chromatogram of a waste flammable solvent analyzed under the same gas chromatographic conditions by GC-MS and GC-IR. It shows the IR reconstructed chromatogram and the IR spectrum for the identified peak number 11 with the library match for 5-methyl-2-hexanone (Methylisoamylketone).

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