Title: Non-thermal Plasma for Point-of-Use Abatement of VOC emissions:
Final Report ESHC003(b)

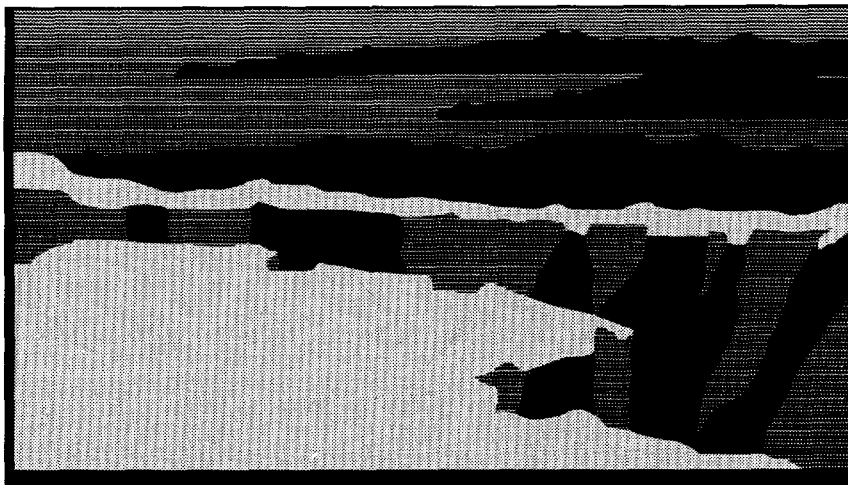**Author(s):** John J. Coogan
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ESHC003(b)
Silent Discharge Plasma for Point-of-Use Abatement of VOC Emissions
Final Report

Abstract:

Los Alamos and SEMATECH have evaluated a silent discharge plasma (SDP) device for point-of-use (POU) control of specific semiconductor VOC emissions at the source. Destruction efficiencies were initially determined at the bench scale using controlled gas mixtures and system performance was measured for simulated emissions containing a variety of volatile organic compounds (including HMDS) and PFCs. Based on this work, a field-pilot unit was designed and tested at a SEMATECH member site using two slip-streams : 1) PGMEA and HMDS gas mixture from lithography tools and the 2) acetone, PCE and methanol from a wet bench cleaning tool. Based on the pilot test data , CoO estimates for the SDP technology show annual operating expenses (including amortized capital and installation costs, maintenance, and utilities) are $8.3K for a single 250 scfm lithotrack tool. End-of-pipe (EOP) system costs are $33.3K per 1000 scfm as compared to about $22K per 1000 scfm for a typical EOP concentrator/thermal abatement system. LANL does not recommend replacing existing EOP systems with SDP. However SDP could be easily installed in "niche" circumstances for POU control of VOCs from lithotrack tools.

Keywords:

Nonthermal plasma, silent discharge plasma, volatile organic compounds, emissions control equipment, gas cleaning, beta-testing, point-of-use

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Nonthermal Plasma for Point-of-Use Abatement of VOC Emissions
Final Report

Contents:

1.0 Background

Los Alamos and SEMATECH have collaborated to demonstrate a novel, potentially cost effective, air emissions control technology, applicable to point-of-use (POU) destruction of volatile organic compounds (VOCs). The process is based on the silent discharge plasma, or SDP [1,2,3], and is focused on the efficient treatment of process emissions containing VOCs, specific to the semiconductor manufacturing industry. SDP treatment is an advanced oxidation process which relies on free radical chemistry [4] at ambient temperatures and pressures to destroy air pollutants. The financial support of this work is cost-shared (50/50) between the Department of Energy (DOE), through Los Alamos National Laboratory (LANL), and SEMATECH.

SEMATECH and LANL specifically evaluated SDP for point-of-use treatment as an alternative to thermal treatment, catalytic, and carbon adsorption technologies. Our primary goal was to acquire the data required for an evaluation of this developing technology. The drivers for the project arise from: (1) Semiconductor Industry Association (SIA) ESH roadmap calls for emissions and energy reduction (2) the lower projected process costs relative to other POU technologies, and (3) the need for alternatives to existing combustion devices, especially for off-gases containing HMDS.

Three potential applications of SDP equipment were explored: 1) removal of vapors from a solvent clean operation - focusing on methanol, isopropanol (IPA) and acetone, 2) treatment of photoresist (lithography) streams containing mixtures of hexamethyldisilazane (HMDS), ethyl 3-ethoxy propionate (EEP), and propylene glycol monomethyl ether acetate (PGMEA) and 3) destruction of perfluorinated compounds (PFCs), focusing on the refractory compounds CF_4 and C_2F_6.

The primary objective of this work was to determine the reliability and CoO of the SDP technology for potential POU treatment of volatile organic emissions. This Final Report documents the results from laboratory and pilot tests and provides a cost of ownership estimate.

1.1 Why Point-of-Use?

The current approach to air emissions control combines all VOC exhausts from process tools, cleaning benches, and VOC fume hoods into a single large stream duct and treated by an "end of pipe" (EOP) abatement system. To increase fab flexibility, reduce operating expenses, and provide a higher level of emissions control, the industry is evaluating POU air emissions control

technologies. In POU control, semiconductor processes are individually controlled, replacing the single large control system now used. This approach will, if suitable technologies can be identified, increase manufacturing flexibility and treat VOCs at the source while they are at higher concentrations. The ability of SDP to go from essentially zero power to full power in a fraction of a second conserves utilities and allows this system to be used on transient off-gas streams. Additionally, the system maintains an economy of scale at the lower (10-500 scfm) flows intrinsic to a POU system. Key to the success of the SDP, or any other POU technology, is the identification of an appropriate niche VOC stream.

2.0 Plasma Sources:

What is a "nonthermal" discharge? By nonthermal we mean that the various species in the plasma are at very different temperatures. Nonthermal is used to differentiate these sources from "thermal" plasmas where all particles, electrons, ions and non-charged species are at the same temperature. In SDP, and all the other nonthermal plasmas, the lightest particles, the electrons are heated to very high temperatures (in air discharges typical electron temperatures are 3 eV and are approximately equal to 30,000°K) where the bulk gas remains at ambient temperatures. This means that a "nonthermal" or "cold" plasma is able to access very energetic chemistry without heating the bulk of the gas. For example, the plasma used to etch wafers is nonthermal, and uses the chemical energy of atomic fluorine to selectively remove silicon.

SDP treatment is an advanced oxidation process which relies on free radical chemistry at ambient temperatures and pressures to oxidize gas phase contaminants. Within the SDP myriad micro-arcs, similar to tiny lightning bolts and each lasting only a few nanoseconds, produce energetic electrons. While the bulk of the gas remains at ambient temperatures, the high temperature electrons efficiently dissociate oxygen and water to produce large concentrations of free radicals. These radicals then attack and oxidize the organic species in a process similar to thermal oxidation. The desired chemistry is very similar to thermal oxidation, except that the radicals are produced by electronic dissociation of water and oxygen, not by thermal dissociation. Usually this nonthermal regime is accessed at low pressures where collisions are less likely and electrons have a much longer mean free path. As pressure increases, so do collision rates and the transfer of energy from one species to the other. Atmospheric cold plasmas must interrupt the collisions before equilibrium is reached.

The other atmospheric pressure plasma source, corona, applies a short pulse that allows electronic excitation in the high field region of the reactor near the center wire. The high voltage pulse

applied to the center wire must be short enough to eliminate the electric field before the arc can thermalize - that is before enough collisions can take place to transfer energy from the very light and very quick electrons to the bulk of the gas. SDP exploits the interaction of the discharge with the dielectric surface to produce a homogeneous distribution of self-terminating pulses (microdischarges) even when the high voltage is applied slowly (typically 1-5 kHz). Other plasma sources are summarized in TABLE 1 [5].

TABLE 1 : Summary of plasma sources.

Plasma Source	Advantages	Disadvantages
Radio frequency	Simple power supply Already used in semiconductor industry	Low pressure Low throughput
Corona	Atmospheric pressure	More complicated power supply Low power density Large footprint
Microwave	Simple power supply	Reduced efficiency at high pressures
Electron beams	Potentially more chemically efficient	More complicated system Significant R&D required
SDP	Atmospheric pressure Small footprint Simple power supplies	Limited commercial use at high flow rates

What makes the SDP different from other plasma sources? SDP is the simplest and provides the highest power density (i.e. most compact) atmospheric cold-plasma source [5]. Power supplies and reactors are inexpensive, and silent discharges in pure oxygen and air have been studied extensively for ozone generation applications since the 1850's - by 1920, the city of Nice, France was using a SDP based system to treat their water supply. SDP is such a mature, reliable, and economical process that SDP driven ozonation is the method of choice for sanitary water treatment in Europe and a growing number of cities within the United States. Compounds destroyed by SDP methods include: oxides of sulfur and nitrogen (SO_x and NO_x), chlorinated hydrocarbons (trichloroethylene, perchloroethylene, trichloroethane, and carbon tetrachloride), aromatic hydrocarbons (benzene, xylene, toluene, p-cumene) and surrogates for polychlorinated biphenyls [6].

The key parameter that governs the degree of reduction of a given gas species in the SDP unit is the plasma energy per unit volume (or deposited electrical power divided by gas flow rate - P/Q). In many cases [7], the removal of a contaminant X can be approximated by an exponential decay

$$[X] = [X]_0 \exp(-\alpha/\beta) \qquad (1)$$

where $[X]$ is the resulting concentration, $[X]_0$ is the initial concentration, α is the applied specific energy or P/Q, and β is the e-fold energy density. Supplying one β of energy density to the reactor reduces the concentration by $1/e$, two β's reduces it by $1/e^2$, and so on. β-values are given in base e units. It is more convenient to express β in base ten units because then it represents the energy density required to reduce the contaminant concentration by a factor of ten. For base ten units the base-ten exponential-folding factor, or "nine-factor", is equal to $-\beta*\ln(0.1)$ or $2.3*\beta$. The lower the "nine-factor" the more efficient the process. The removal of VOCs is often expressed in terms of a destruction efficiency (DE) of so many "nines"; e.g., three "nines" removal equals 99.9% DE. β varies strongly from compound to compound and is related to the chemical kinetic rates of the compound with O and OH.

To increase the removal fraction $[X]/[X]_0$ for a given gas mixture within the SDP, α must be increased. Because $\alpha= P/Q$, α can be increased by either increasing system power, P, or decreasing the flow rate, Q, for a given cell volume. Alternatively, by dividing a given gas flow into several parallel cells, the overall energy density for the total flow can be effectively increased. We prefer the second approach of modularization, whereby a cell of desirable mechanical properties is replicated many times. Such scaling of silent discharge cells has been previously demonstrated for the industrial-scale synthesis of ozone, where municipal water treatment plants frequently require the on-site generation of thousands of kilograms per day. The nine-factor depends on the target compound, and the gas mixture.

The required power density is one of two variables that dominate the cost of ownership calculations (the other one is the cost of the power supply). If the "nine-factor" can be reduced by 50%, the cost of ownership is also reduced by close to 50%. For example, if a compound requires 200 joules/liter for 90% destruction, a flow of 210 scfm (6,000 standard liters per minute or 100 liters per second) would require 20 kW of power. If the nine-factor can be reduced to 100 joules/liter the power required drops to 10 kW. SDP hardware (power supplies and related equipment) cost about $0.50 to $2.00 per watt, depending on system scale.

3.0 Experimental Plan

A detailed experimental plan was jointly produced by LANL and SEMATECH and presented to the PTAB for review. An abridged version follows.

TABLE 2: Summary of workplan.

TASK 1(6/15/95 to 8/31/95) :	Identify compounds of interest and generate a detailed work plan. (After the presentation of this work plan to the PTAB on September 28, 1995 several changes were made in the experimental plan to better address industry needs. The schedule and deliverables of Tasks 2 and 3 have been adjusted to reflect these modifications.)
TASK 2 (8/15/95 to 1/15/96) :	Lab testing of simulated gas mixtures. Tests will determine destruction efficiency and byproducts from the SDP treatment of the following gas mixtures: room temperature tests with isopropanol, methanol, and acetone , additional testing of isopropanol, methanol, and acetone at higher temperatures, tests with perfluorinated compounds (CF_4/C_2F_6) in nitrogen/oxygen mixtures, and simulated lithography streams containing mixtures of hexamethyldisilazane (HMDS), ethyl 3-ethoxy propionate (EEP), and propylene glycol monomethyl ether acetate (PGMEA). Additional tests quantified byproduct formation. This data is summarized in Section 4 of this report.
TASK 3 (10/1/95 to 3/31/96) :	Design, fabricate and test a pilot SDP unit suitable for on-site testing. System details and SEMI-S2 review findings are summarized in Section 5. The computer control system was based on the LABVIEW software package and proprietary control codes written at LANL.
TASK 4 (3/31/96 to 11/8/96) :	Field testing at a member site. Two tests were completed - lithotrack and cleaning hood.
TASK 5 (11/8/96 to 1/24/97)	Generate a detailed data summary and cost of ownership estimate. These results are given in Sections 5 and 6.

4.0 Laboratory Testing

A wide range of electrical drivers have been developed for driving the SDP.   In the work
described in this report, an ELGAR SL-3000 provides a 0-300 V sine wave to the primary of a
high voltage transformer. The voltage applied to the cell can be adjusted from 0 to 30 kV and from
50 to 5000 Hz. Because of the transient nature of the current and the relatively low applied
frequency, the usual method of deriving electrical power, power equals voltage times current , is
difficult to implement. In all our work, power is measured using well established methods develop
by Manley [8] and later extended by Kuchler and Pietsch for ozonizer research.

4.1 Bench Tests with VOCs from Solvent Cleaning Operations

The destruction efficiency of IPA / methanol / acetone at 133 ppmv (combined mixture 400 ppmv)
in "dry" and "humid air" (approximately 45% relative humidity at room temperature) were
measured using gas chromatography. Compound specific results are shown in Figure 1. For the
three-compound mix, the measured nine-factors are 200 J/liter for IPA, 800 J/liter for methanol,
and 1100 J/liter for acetone. That is to say, 90% removal of IPA requires 200 J/liter, 90% removal
of methanol requires 800 J/liter and 90% removal of acetone requires 1100 J/liter. The same
removal efficiencies were measured with single compound mixtures. Figure 2 shows that similar
to photocatalytic systems, small amounts of acetone are generated during the destruction of IPA.
The destruction of total hydrocarbons, measured using a flame ionization detector, is dominated by
the power required for the acetone. Figure 3 shows a comparison of total hydrocarbons, measured
with a flame ionization detector, and the sum of all hydrocarbons measured with gas
chromatography/mass spectrometry (GC/MS). Because acetone is produced during the oxidation
of IPA, 1250 J/liter is required to destroy 90% of the hydrocarbons from this mix of VOCs, so a
500 scfm air stream would require approximately 300 kW of electrical power - a very large system.
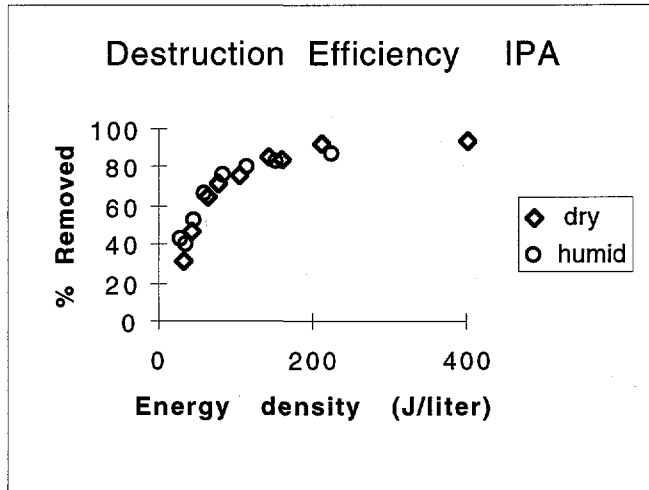A system removing only IPA (a nine factor of 200 joules/liter) would require only 50 kW.

Figure 1: Plot of the destruction efficiency of 133 ppmv of IPA from dry and humid air streams: percent removed versus energy density.
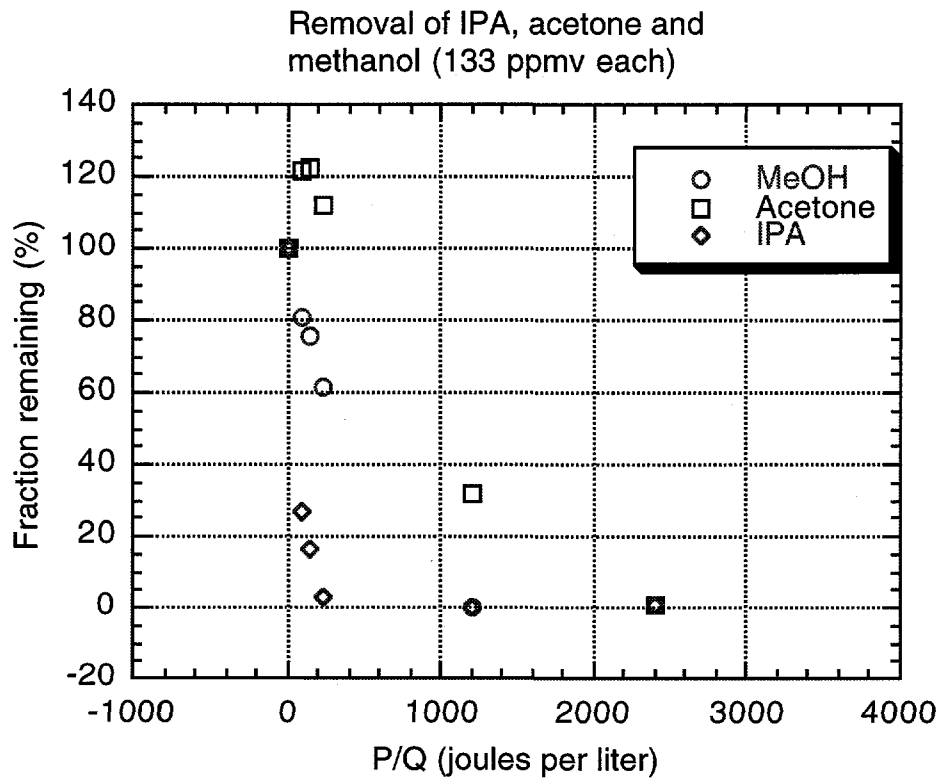

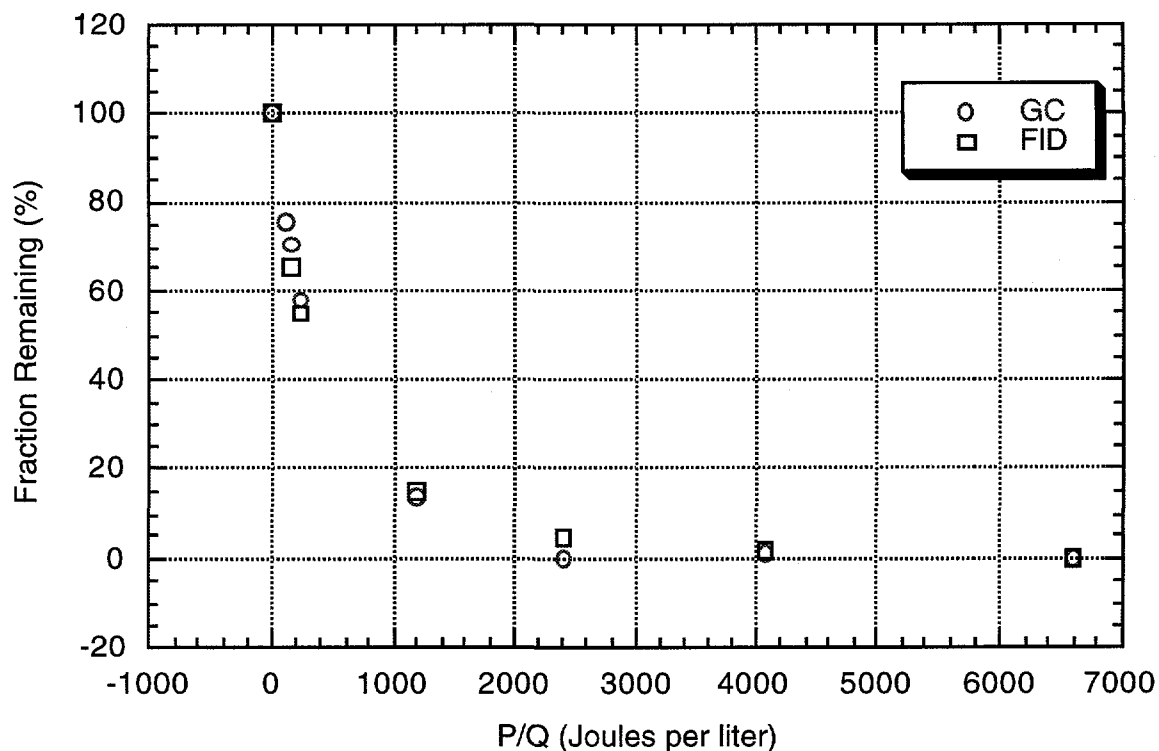Figure 2: Destruction of methanol, IPA, and acetone mix (133 ppmv each) from humid air streams.

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Figure 3: The destruction of methanol, IPA and acetone mix (133 ppmv each) from humid air streams. Total hydrocarbons are plotted as a "methane equivalent". Independent measurements using a flame ionization detector (FID) and GC/MS are both plotted.

Corona plasma reactors have demonstrated [9] a "nine" factor of 770 J/liter for the removal of acetone at a temperature of 120 °C and have shown that raising the temperature of the gas from 120 to 300 °C reduces the required energy consumption by approximately four times. We confirmed this result for the SDP in a follow-up experiment on IPA/methanol/acetone removal at elevated temperatures. Figure 4 shows that at a temperature of 300 °C, the acetone nine factor is reduced to 250 J/liter reducing the required power for a 500 scfm system to from 300 to 65 kW. Figures 5 and 6 quantify the production of other compounds. Ozone production is eliminated at temperatures above 150 °C, and NO_x is generated at only a few ppmv.
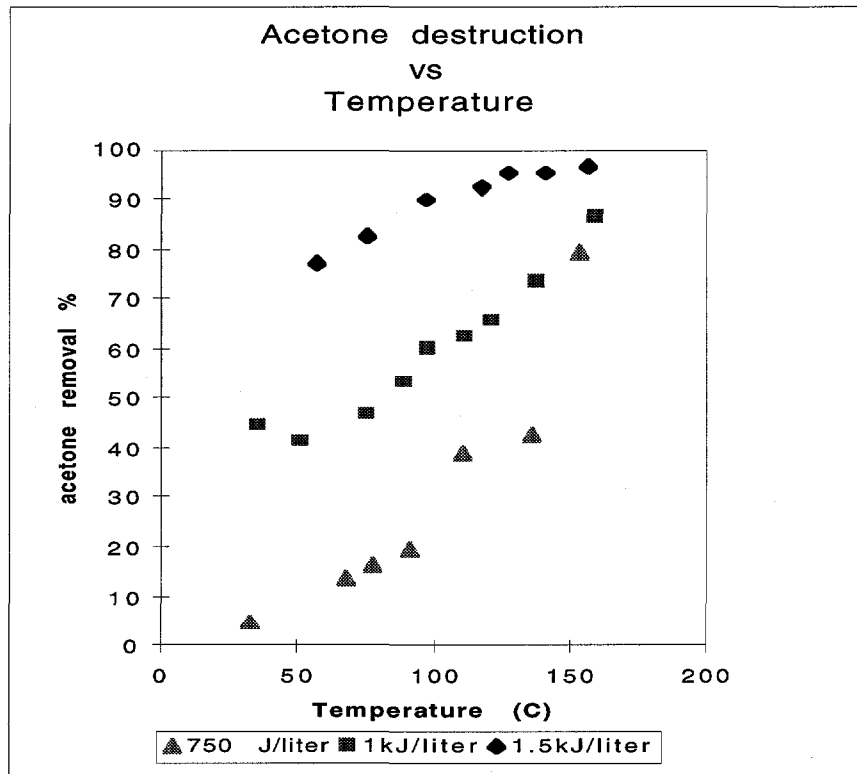
Figure 4: Effect of temperature on the removal of 133 ppmv of acetone from air.
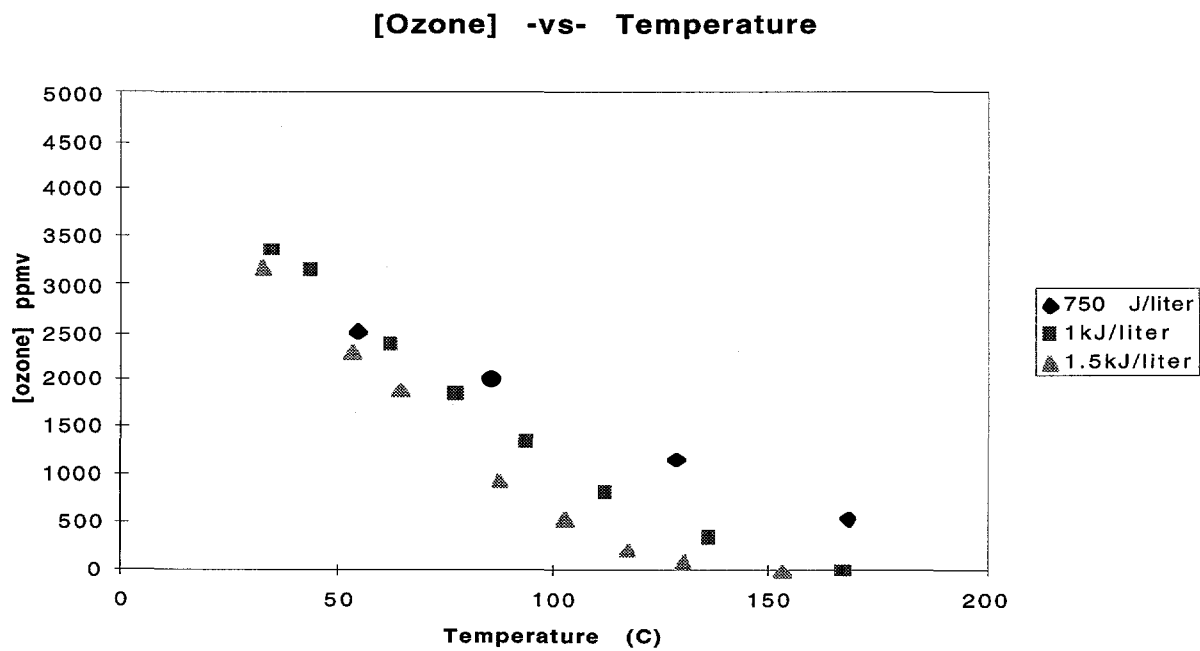

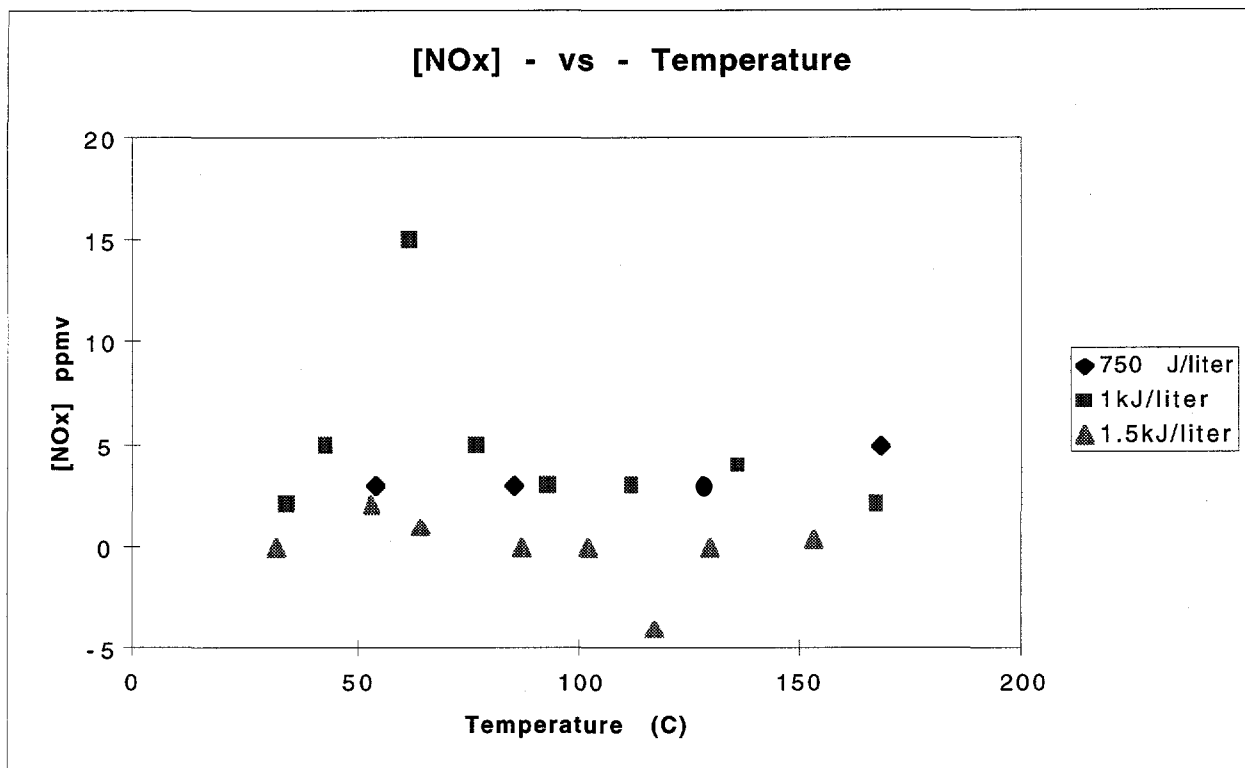Figure 5: Effect of temperature on the formation of ozone.

[NOx] - vs - Temperature

Legend:
- ◆ 750 J/liter
- ■ 1kJ/liter
- ▲ 1.5kJ/liter

Y-axis: [NOx] ppmv
X-axis: Temperature (C)

Figure 6: Effect of temperature on NOx formation. This measurement does not include the production of N_2O.

4.2 Bench Tests with Lithotrack VOCs

Simulated air streams containing HMDS were combined with EEP and PGMEA at 10-20 ppmv in humid (~45% relative humidity) air and treated in the lab-scale SDP reactor. Tests of EEP in both dry and humid air showed better than 90% destruction at 40 joules/liter (see Figure 7). A nine-factor of 40 J/liter means that 90% removal of a 15,000 slpm (500 scfm) air stream would require approximately 12 kW of electrical power, less than 30 times the cost of a similar flow system removing room temperature acetone. Tests were then continued to evaluate the effect of HMDS and its principle byproduct, silicon dioxide (SiO_2), on SDP cell performance. The laboratory system treated 3,000 standard liters of air contaminated with 50 ppmv of HMDS. A flow of 10 slpm of the air/HMDS mixture combined with a 2 slpm flow of air saturated with water at 35 °C (the resulting 12 slpm stream contained 42 ppmv of HMDS at 20 °C and 40% relative humidity) was fed through an SDP cell for 4.2 hours. SiO_2 was produced with no reduction in SDP efficiency. The hexamethyldisilazane was destroyed with a 95% destruction efficiency measured at 1.4 kJ/liter. This yields a nine-factor close to 1 kJ/liter. Very few other byproducts were seen.

Tentatively identified compounds include trimethylsilanol, pentamethyldisiloxane, and diethoxydimethylsilane (combined, the concentrations of these compounds is less than 0.5 ppmv). A second test with a more complex air/water/PGMEA and HMDS mixture produced similar results to the individual tests - removal was consistently unaffected by the presence of HMDS. Figure 8 shows the expected scaling of the SDP system for higher flows.
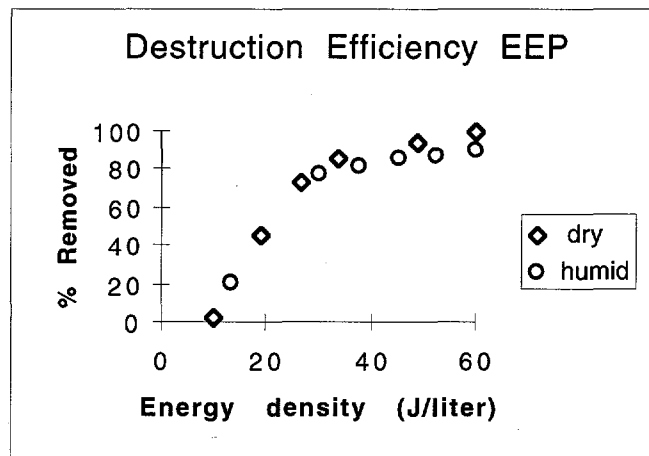

Figure 7: Destruction efficiency of 20 ppmv of EEP from dry and humid air streams. Removal rates for PGMEA are almost identical.

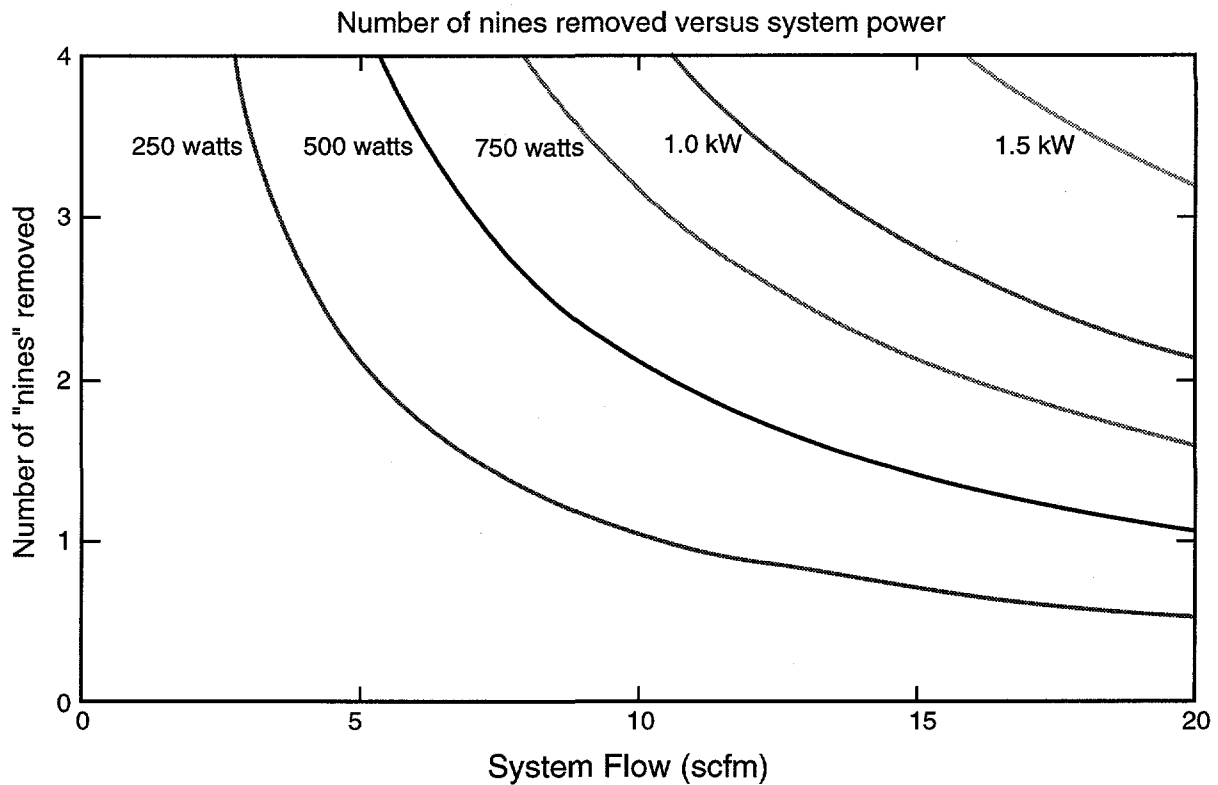**Number of nines removed versus system power**

Figure 8 : Predicted scaling for PGMEA removal at higher flows (this scaling has been confirmed at concentrations from 15 to 1000 ppmv) The plot predicts that 99% removal from a flow of 15 scfm will require 750 watts.

4.3 Bench Tests with PFCs

Other plasma-based systems have been used to treat perfluorinated compounds (PFCs) with mixed success. NF_3 can be destroyed with reasonable efficiency, but significantly more power is required to destroy CF_4 and C_2F_6. Tests with pulsed corona reactors [10] measured only 4% removal of CF_4 at the same reactor power that destroyed 84% of NF_3. Assuming a first order removal rate, this means that CF_4 requires 40-50 time more energy than NF_3. For this evaluation we have focused exclusively on the more refractory compounds. A standard SDP unit was used to treat a 1000 ppmv of CF_4 and C_2F_6 with 1000 ppmv of O_2 in dry nitrogen. Removal rates of >95% for C_2F_6 and > 45% for CF_4 were demonstrated and are shown in Figure 9. Still, as shown in Figure 10, the 95% removal of C_2F_6 resulted in the production of 400 ppmv of CF_4. This

suggests that while 5% of the fluoride ion remained in C_2F_6, at most only 68 % of the fluoride ion was converted to HF. The energy required for this removal was substantial, approaching 120 kJ/liter. Optimization of gas mix (oxygen, argon, and hydrogen dilution) showed modest increases, with CF_4 removal increased to only 75-80% at 100 kJ/liter. This data suggests that simple, homogeneous (gas phase) reactions are not effective at destroying CF_4 and that heterogeneous (gas-wall) interactions may be the key to effective removal of CF_4.

Follow on work designed and tested a heterogeneous reactor concept by partially filling a SDP cell with a sacrificial aluminum electrode. Surfaces can remove atomic fluorine by enabling the production of F_2 (recombination) or by forming non-volatile fluorides (reaction). The use of this modified reactor and the addition of a small quantity of H_2 resulted in a 80% destruction efficiency of CF_4 at 80 kJ/liter. Figure 11 summarizes tests with the combination reactor using different materials to "fill" the SDP gap. A more complete summary of our work with PFCs can be found in reference 11.
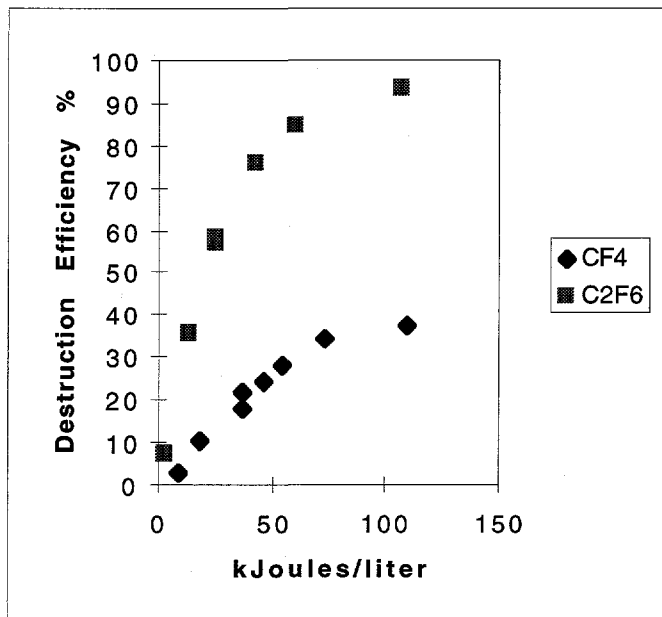

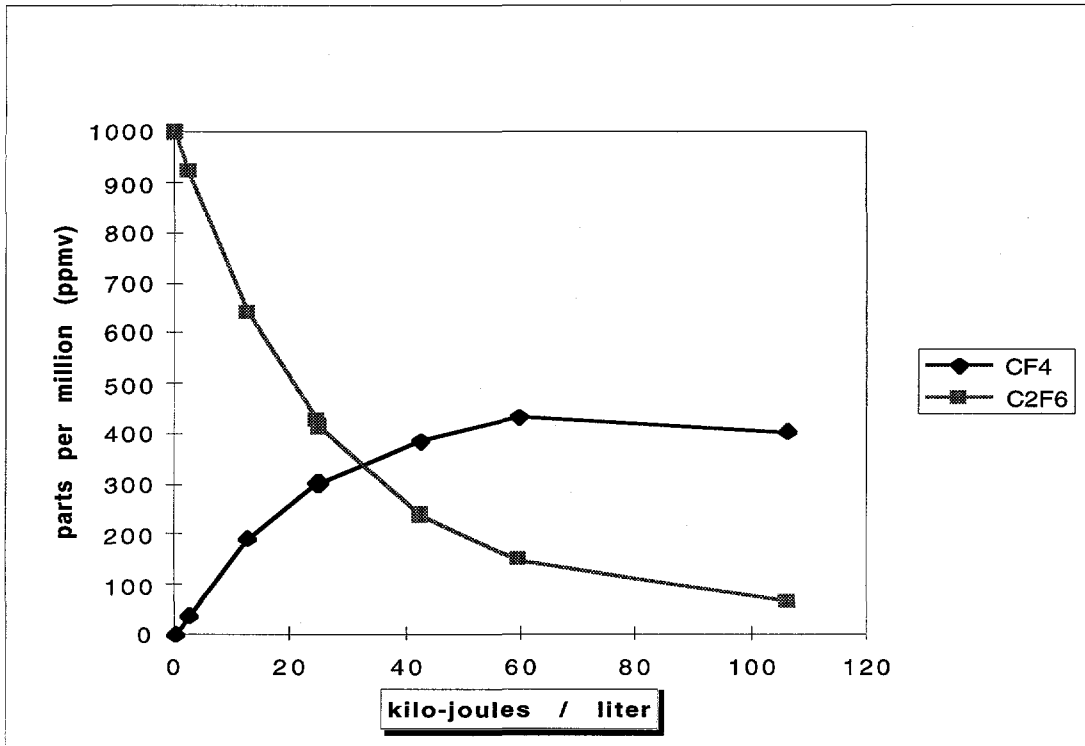Figure 9: DE versus energy density for 1000 ppmv of PFCs, with 1000 ppmv of O_2, in nitrogen.

Figure 10: Destruction of 1000 ppmv of C_2F_6, with 1000 ppmv of O_2 in nitrogen. The destruction of C_2F_6 and the formation of its byproduct CF_4 are both plotted
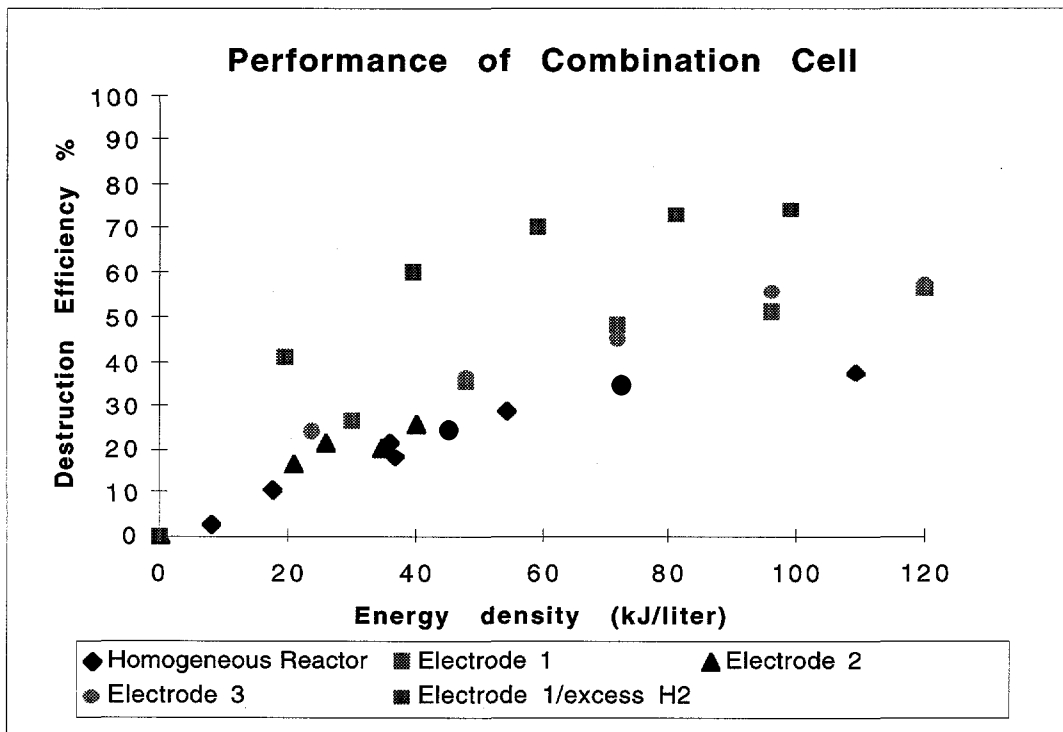

Figure 11: Removal of CF_4 using both homogeneous and heterogeneous reactions.

5.0 Field Testing

Based on this series of lab tests our focus on the POU narrowed to organic solvent vapors produced from cleaning hoods and organic solvent/HMDS streams produced from lithography tools.

5.1 Preparation for Field tests

Before any equipment was shipped to the member company for tests, a system review using the SEMI S2-93 guidelines was performed by a third party, Safeflow Systems Inc. Copies of the summary report prepared by Safeflow are available [12]. The field unit is made of the several subassemblies described below.

Electrical: System power is provided by a commercial power supply made by ELGAR (SW5250). This unit is rated at an output of 5250 watts into a matched load and can provide output wave forms from 50 to 5000 Hz. A matching inductor is placed across the output of the supply and in parallel with the HV transformer that takes the high-current (up to 50 amps peak) low voltage (up to 255 volts peak) and transforms it to the high-voltage (15-18 kV) low current (<1 amp) signal that drives the SDP cells. The inductors are cooled by fans, and the transformer by a small heat exchanger. Target discharge powers are 1000-4500 watts. A summary of the electrical utilities required by the pilot unit is shown in Table 3.

TABLE 3 : Electrical specification of the SDP pilot unit.

Electrical Requirements	Voltage	Maximum Current (amps)
Main power Supply	208 three phase	40-50
Oil Pump	115/230	5.4/2.7
Heat exchanger	115	1.1
Oil Heater	115	15
Computer	115	4.1
SCXI control	115	4.1
Oscilloscope	115	4.1
Air blower	115	15
Totals	208 three phase	40-50
	115/120	45

Gas flow and control: Gas flow can be measured using either analog or digital flow meters. Target flows vary from 2 to 20 scfm. A blower is available to boost existing pressures and flows. The secondary containment tank is rated at 5 psi. A pressure drop of about 5 inches of water is required for an SDP flow of 10 scfm. Pressure relief valves insure that the ΔP between the interior of the SDP cells and the tank pressure do not exceed 0.33 psi. The relief valves should prevent any damage to the SDP cells. However, to reduce the risk of cells cracking the system should not be connected to any vacuum systems. Figure 12 shows the system pressure versus flow for both lab and field tests. The system was designed for a maximum pressure of 0.33 psi (~10 inches of water), which limited total flow to ~ 15 scfm.
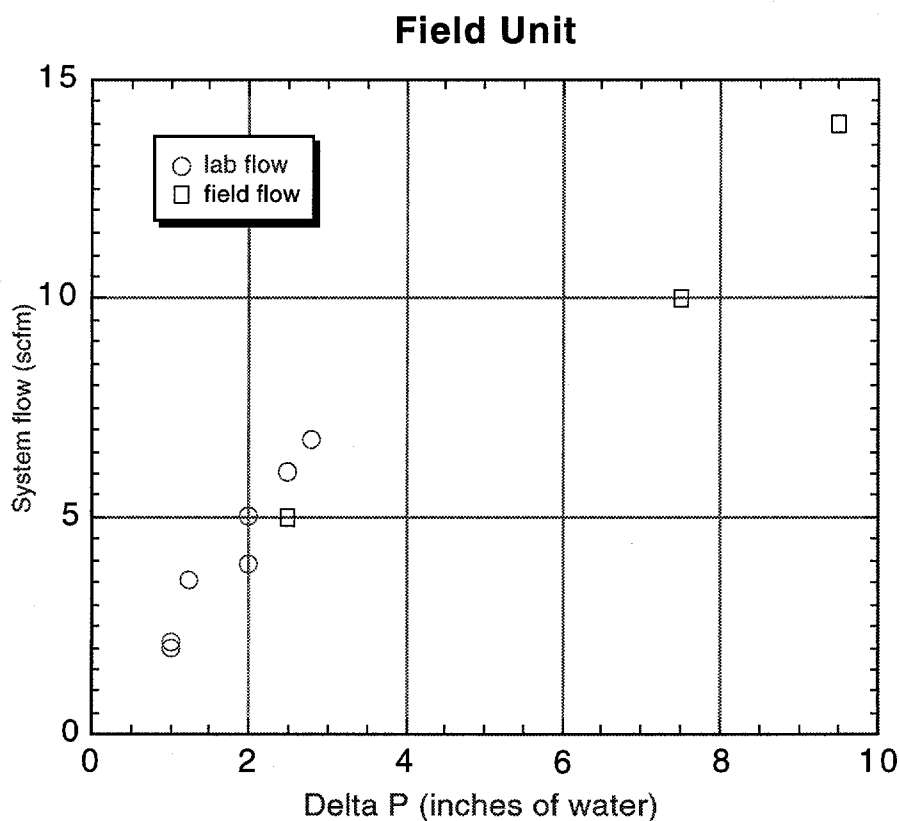
Field Unit

Figure 12: Measured pressure drop across SDP unit as a function of gas flow rate.

System temperature control: The system temperature is controlled by an oil flow system. The oil both preheats the cells, preventing any water condensation, and cools the cell during operation. The fan on the heat exchanger can be on, off, and or thermostatic.

Control system: The computer system controls the SDP electrical power, gas flows and oil temperatures. The computer system (SCXI) also logs operating data (temperatures, pressures, powers, etc.).

Effluent monitoring: System performance is monitored by Fourier transform infra-red (FTIR) spectrometers provided by a third party (Radian International). Twin FTIRs continuously sampled and analyzed both the input and output of the SDP system. The data is accurate to ±20-30%, and typical detection limits are 1 ppmv or less for all compounds.

Process and Instrumentation diagrams: Figure 13 shows the mechanical layout of the system. The figure shows the locations of thermocouples (TC), flow meters (FM), and pressure transducers (P). To minimize disruption of fab operations, a slipstream of process exhaust is passed through the SDP system and then returned to the main exhaust system as shown in Figure 14.
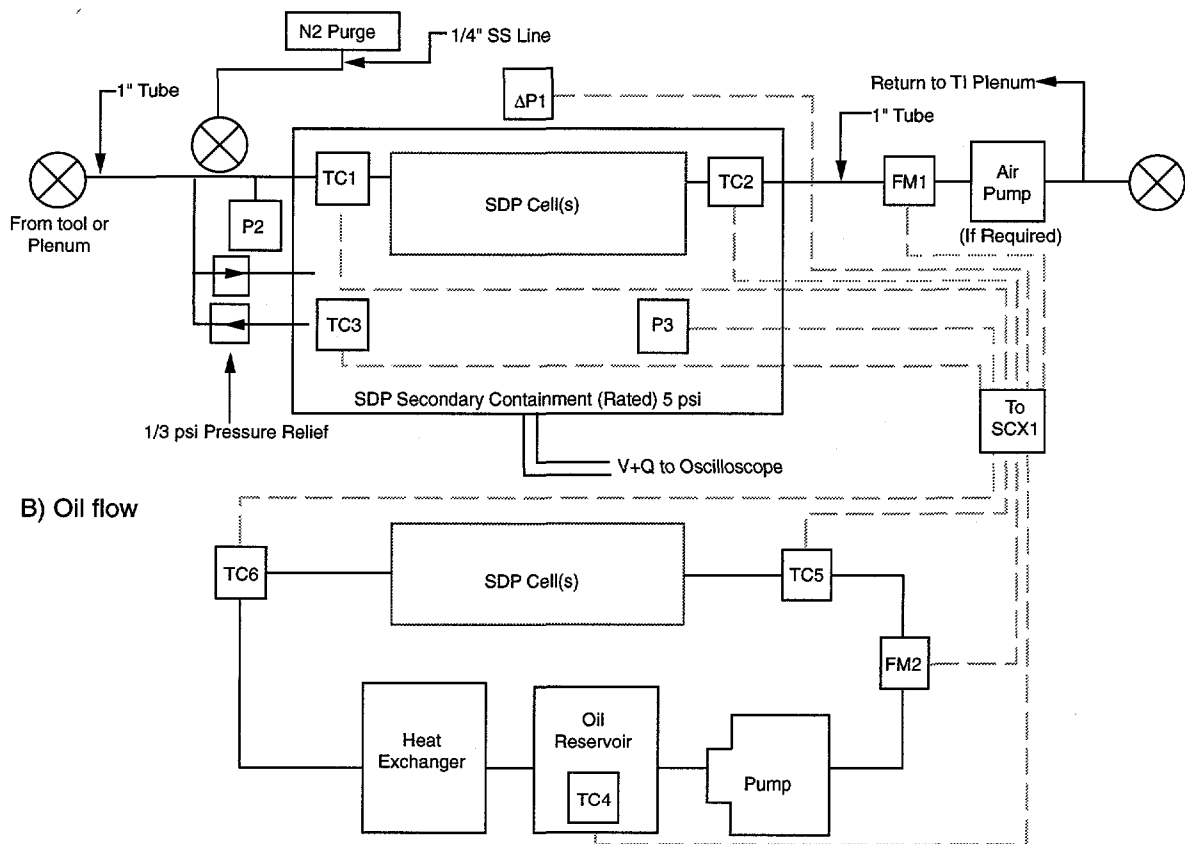

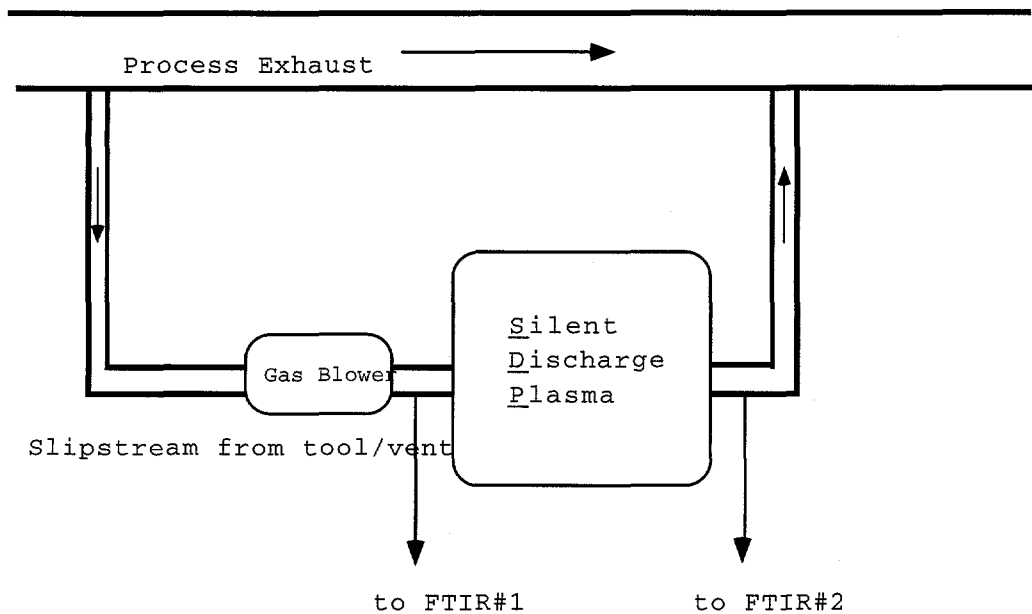Figure 13: Mechanical layout: System flows, temperatures, and pressures

Figure 14: Schematic of set-up used with the SDP pilot unit.

5.2 Description of Field Tests

The equipment was transferred to the test facility and installed by LANL personnel. Figures 15a and 15b show the interior of the SDP system. The 6 individual SDP cells can be clearly seen. Figure 16 shows a mechanical drawing of the equipment. The gas flow volume between the two glass plates (hatched) is shown in red, and the electrodes in gray. One of two FTIRs is shown in Figure 17. The SDP power supply and computer control are shown in Figure 18, the tank containing the cells can be seen to the left of the supply. Figure 19 shows a better view of the external SDP tank.
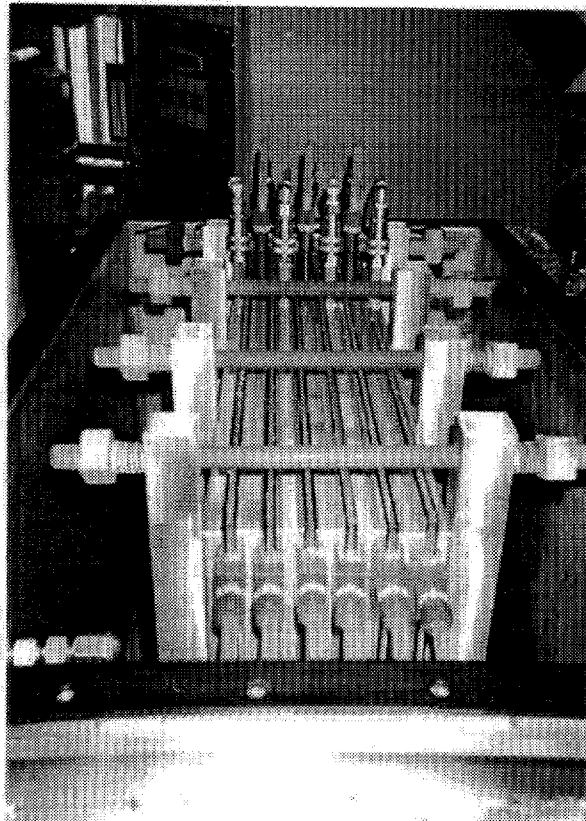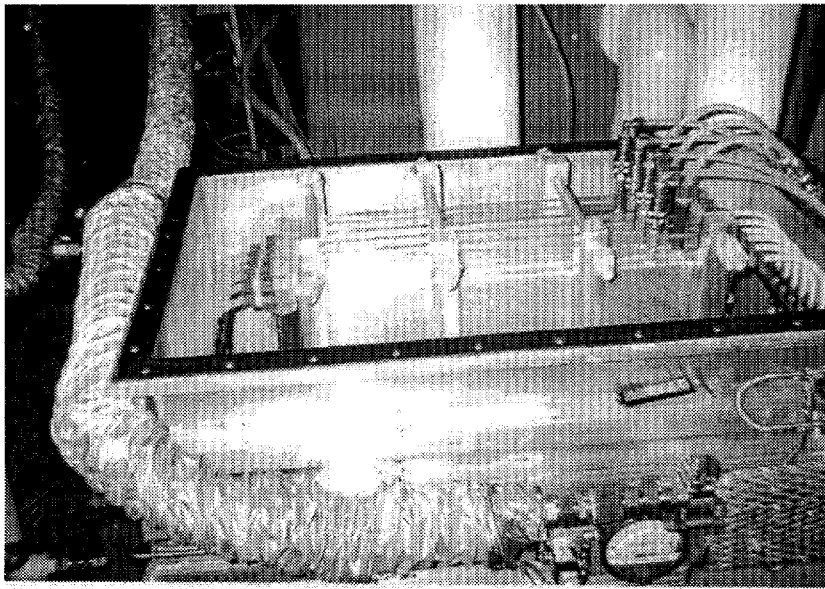
Figure 15: Photographs of the inside of the field unit. The top photo, Figure 15a shows a side view of the system with the lid removed. Figure 15b, the bottom photo shows an end-on view of the six individual SDP cells used.
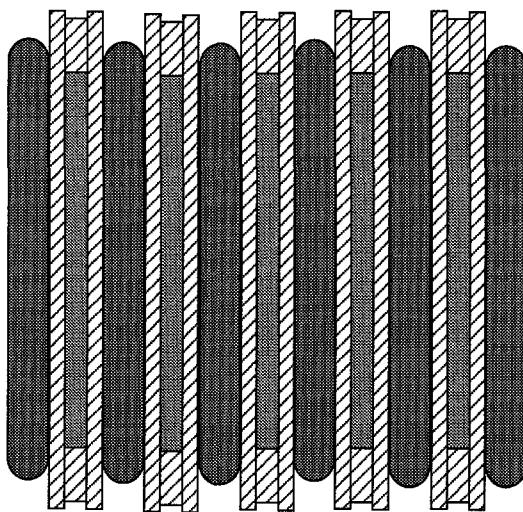
Figure 16 : Mechanical drawing showing an end-on cross section of the field unit. Power is applied to the gray electrodes. Gas flows between the glass plates (hatched). The discharge zone is shown in red.
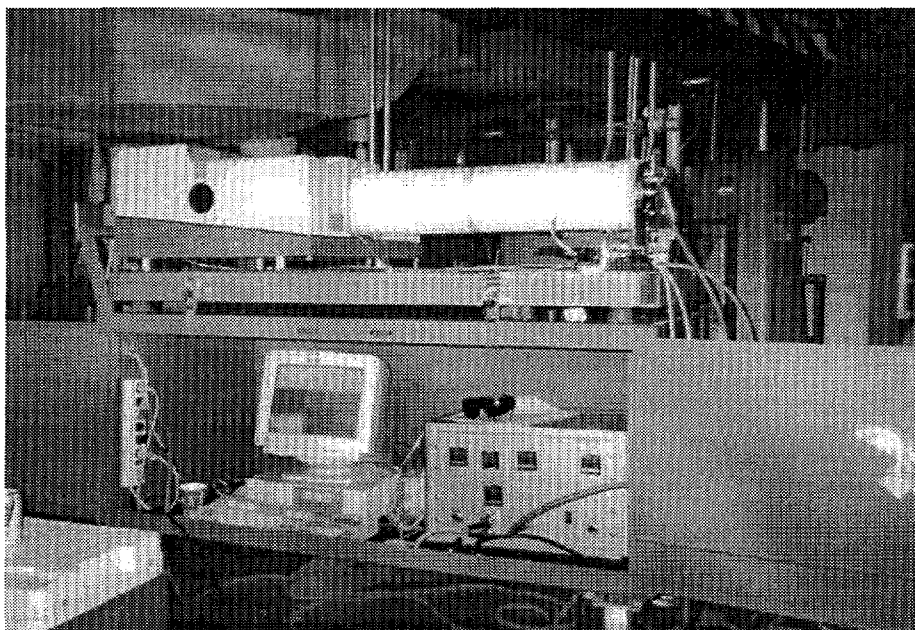
Figure 17: Photograph of one of the two FTIRs from Radian. Both input and output of the SDP system were monitored continuously.
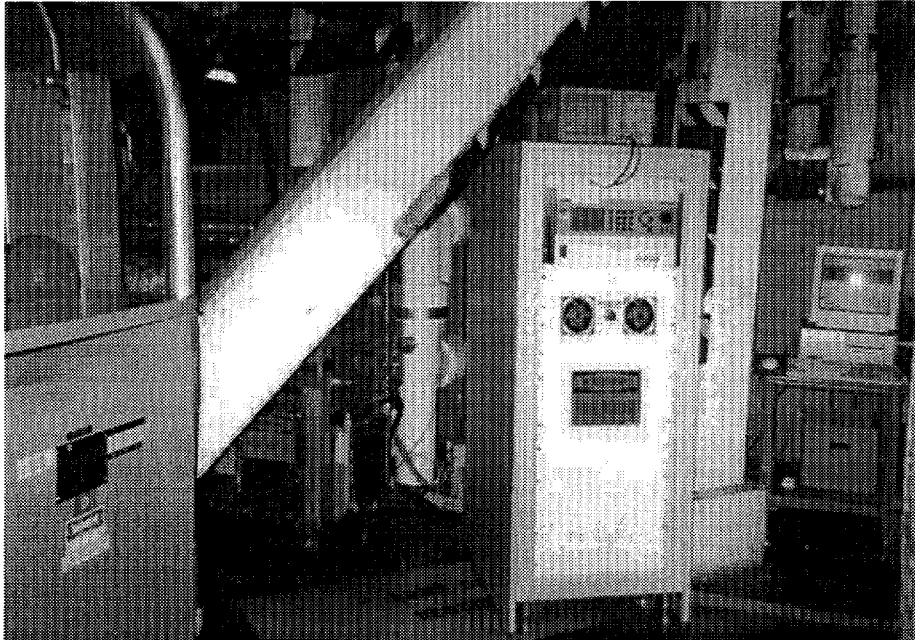
Figure 18: Photograph showing power supply and computer control system.
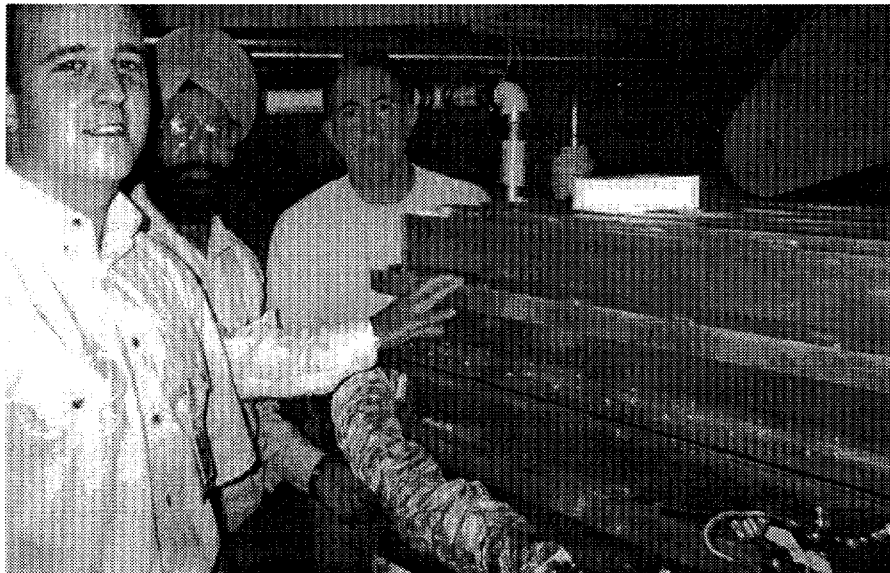
Figure 19: Photograph showing exterior of SDP secondary containment tank.

5.2.1 Results from Lithotrack VOC Emissions

A 14-20 scfm slip stream of the exhaust from an operating lithography tool containing both PGMEA and HMDS, was treated for 30 continuous hours using the SDP equipment. SDP electrical powers ranged from 700 to 3,000 watts. Typical energy densities were 100 to 150

J/liter. As predicted by earlier lab tests, destruction efficiencies for PGMEA consistently exceeded the target 95%, and, depending on the power delivered, often exceeded 99% and/or the detection limits (0.3 ppmv) of the analytical system. The final six hours of operation provided an accelerated test of the long-term effects of HMDS. A bubbler system was set up and provided a steady HMDS source at 450 +/- 100 ppmv for six hours. From a mass balance perspective, this is approximately equal to the amount of HMDS expected during 60-90 operating days. Destruction efficiencies of both the HMDS and PGMEA were continuously measured (Figure 20), and within experimental error PGMEA removal was unaffected by the presence of several hundred ppmv of HMDS. Ozone, carbon and nitrogen containing by-products are shown in Figures 21 and 22, respectively. As in many other room temperature oxidation processes, CO formation is significant. Ozone production can be controlled by allowing the system temperature to increase above 100 °C. Figure 23 shows that 10's of ppmv of formaldehyde, formic acid, and acetic acid were also detected. Inspection of the SDP plumbing after operation found approximately 1-2 grams of what was thought to be SiO_2. To confirm the make up of this material the sample was analyzed using x-ray diffraction. The results show that a fraction of this powder is not SiO_2 but NH_4NO_3, or ammonium nitrate. This compound is manufactured in large quantities as a fertilizer and is routinely produced during electron beam scrubbing of NOx and SOx from power plant flue gases. In those systems [13], urea or ammonia is injected into the off-gas to remove nitric acid (HNO_3), and it is known that HMDS water mixtures produce ammonia. Figure 22 shows that in our equipment HNO_3 formation is eliminated in the presence of HMDS. The small quantities of ammonium nitrate present no operating concerns for the SDP, and standard particulate removal (filters, etc.) methods can still be used.

Although the FTIR spectra is very complex, especially during the HMDS "spike", these results show that the energy densities required for PGMEA removal during this field test are essentially the same as those measured in the lab. Operational data showing PGMEA destruction and byproduct formation is summarized in Figures 20-23. A photo showing the appearance of the SDP cells before and after operation is shown in Figure 24. The passivation of the Pyrex can be seen very clearly. This discoloration did not reduce the gas gap, reduce system performance, or effect the electrical stability of the SDP system.
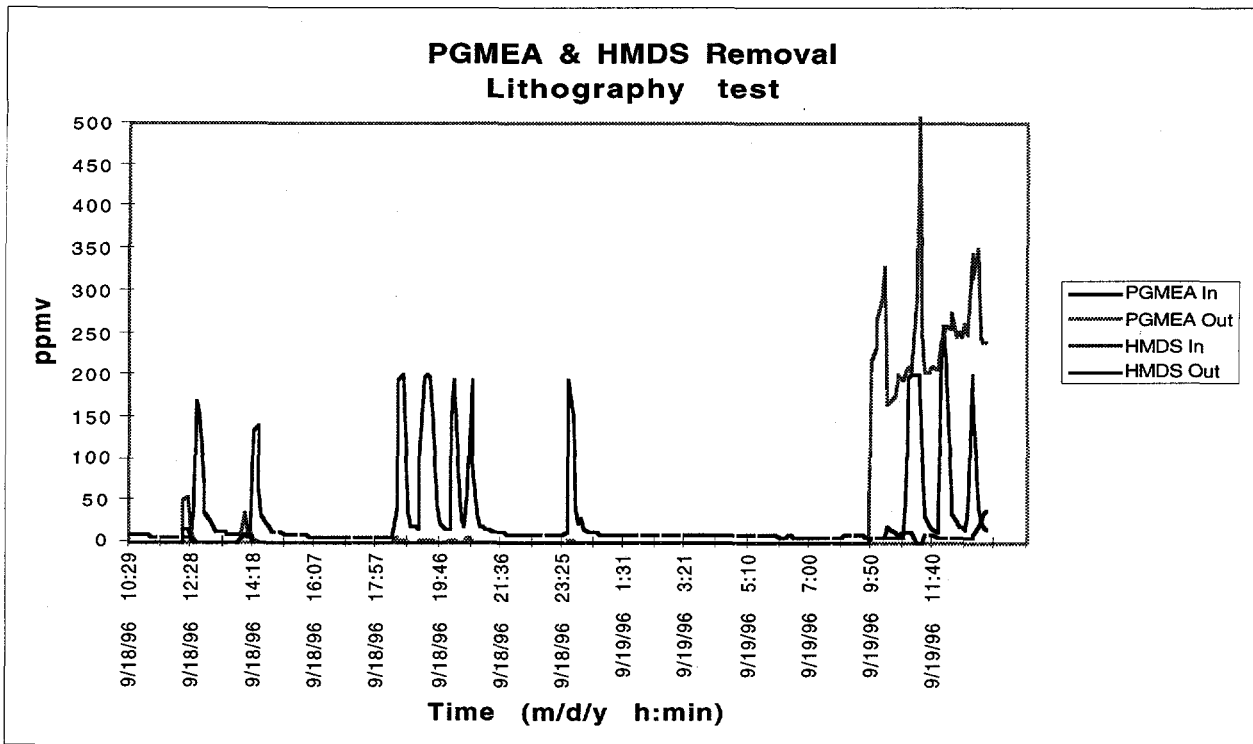
Figure 20: FTIR data summary from tests on Lithotrack exhaust - PGMEA in/out and HMDS in/out.

Figure 21: FTIR data summary from tests on Lithotrack exhaust- CO, CO_2 and ozone generated in SDP cells.
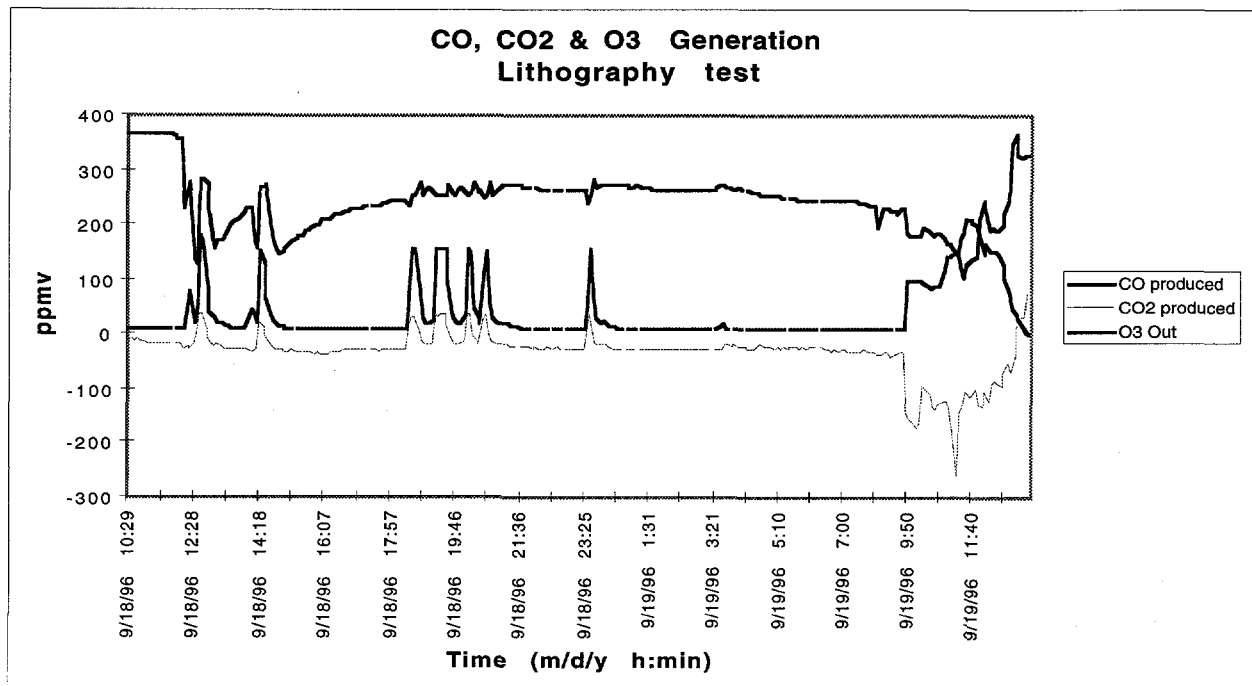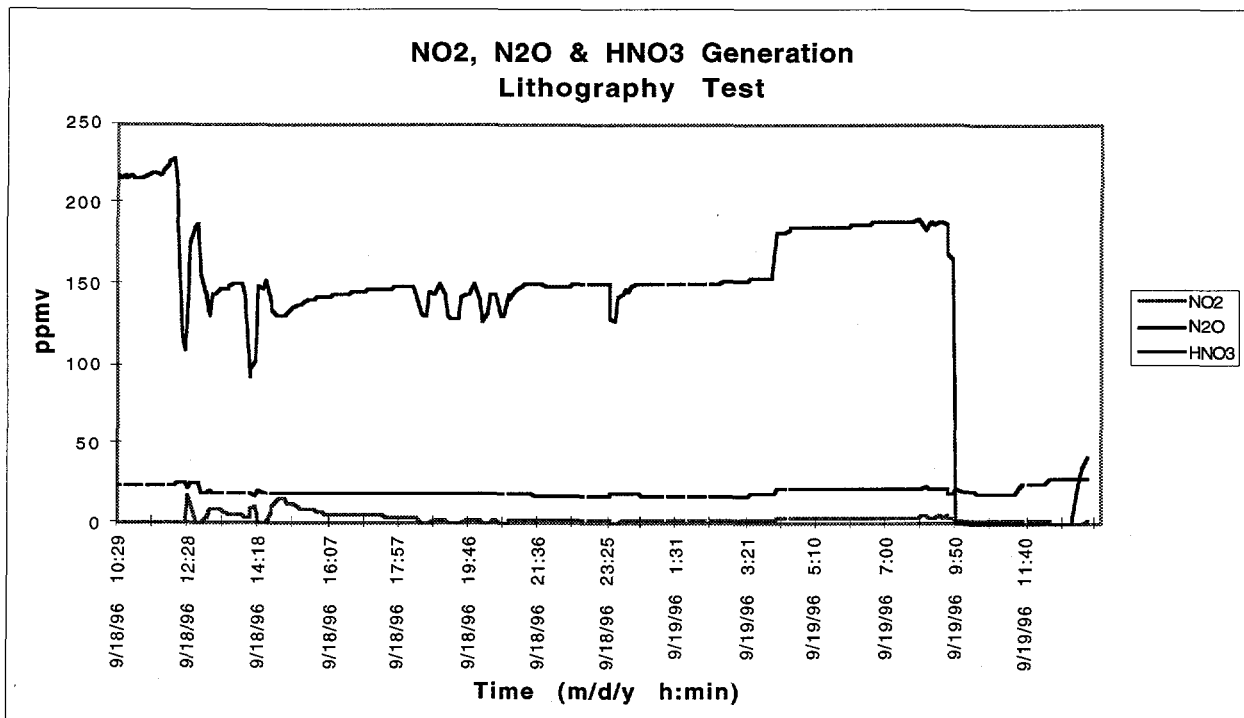
Figure 22: FTIR data summary from tests on Lithotrack exhaust - NO_2, N_2O and HNO_3 generated in SDP cells.
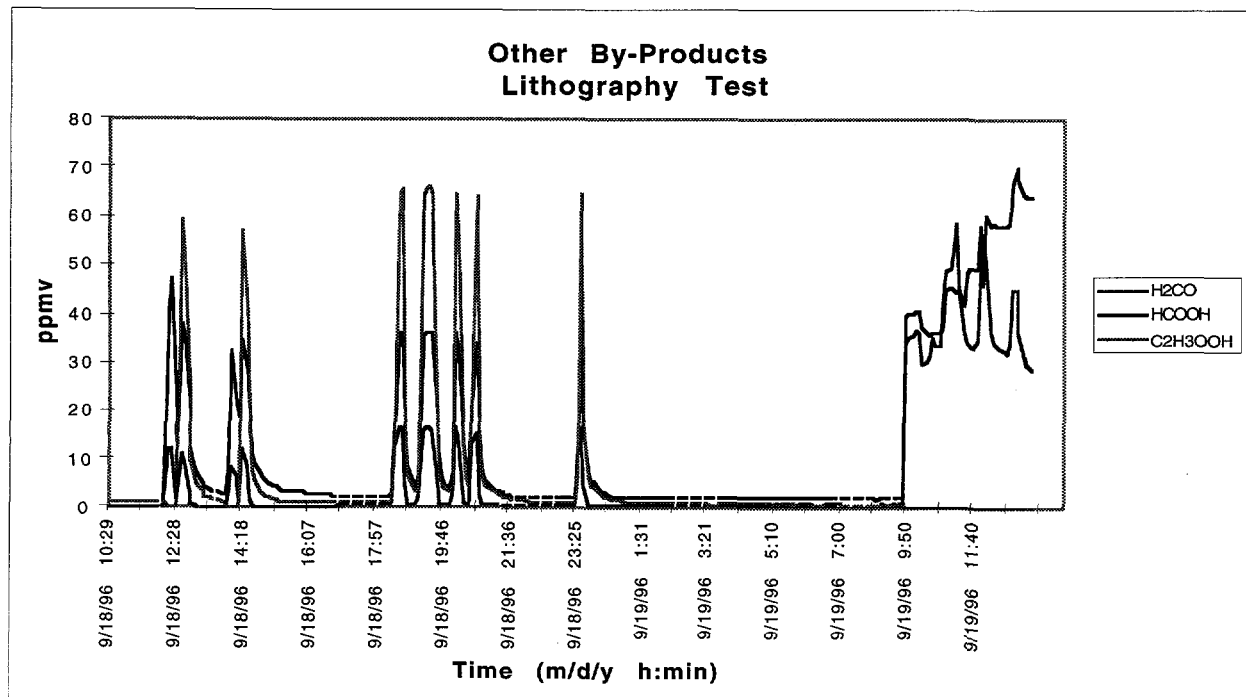

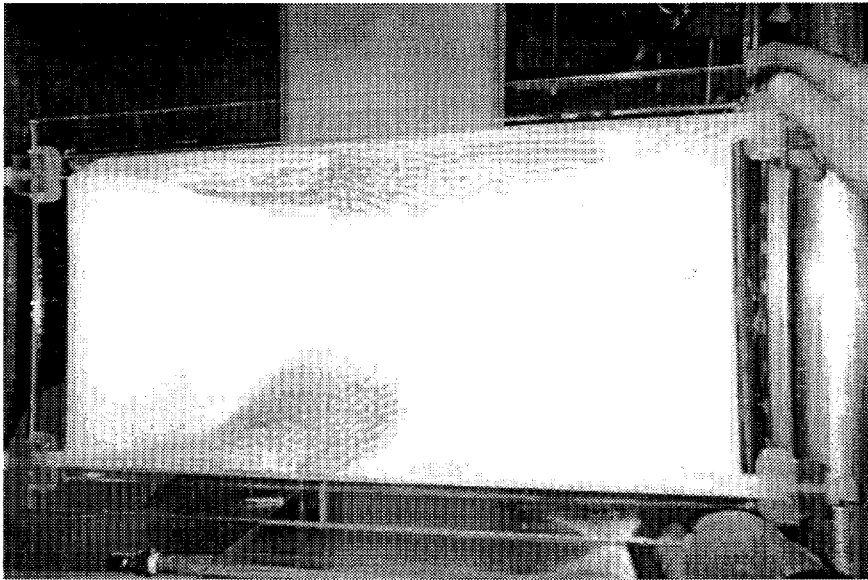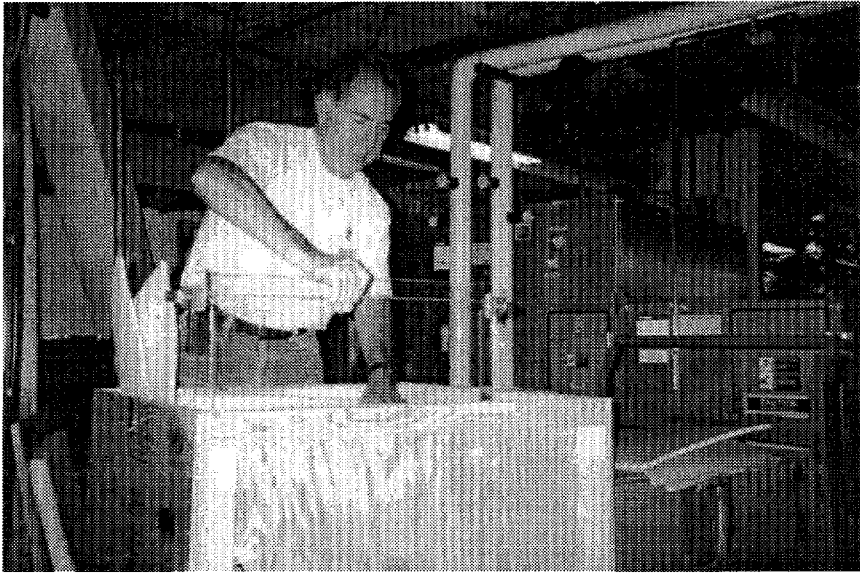Figure 23: FTIR data summary from tests on Lithotrack exhaust - Other tentatively identified compounds.

Figure 24: Photographs showing before and after run condition of SDP cells. A "zero-thickness' passivation layer built up on the SDP glass.

5.2.2 Results from Solvent Cleaning VOC Emissions

The same equipment used for the lithotrack test was then used to treat the off-gases from a cleaning hood. The SDP system was operated and the performance measured for 30 continuous hours. Because the target compounds in this experiment required a higher energy density, flows were reduced to 3-10 scfm. The SDP treated vent hood exhaust, containing acetone (C_3H_6O), perchloroethylene or PCE (C_2Cl_4), and methanol (CH_3OH). SDP electrical power ranged from 1,500 to 3,500 watts. Typical energy densities were 200 to 2,000 J/liter. destruction efficiencies for methanol consistently exceeded 99% and, depending on the power delivered, usually exceeded the detection limits of the analytical system. Destruction efficiencies for acetone showed the expected dependence on power and flow, and scaled consistent with earlier laboratory testing. Because PCE reaction rates with O and OH are comparatively slow, PCE destruction requires higher energy densities. Because of the low inlet concentrations PCE output levels were reduced to below the detection limit of the FTIR (0.12 ppmv) for all but the lowest power densities. Close inspection of the equipment after the test showed no discoloration of the SDP cells. Figures 25 and 26 show the destruction of acetone and methanol during the test. Figures 27 and 28 show the major byproducts produced. HNO_3 production is increased over the previous tests since the applied energy density was increased from 700 - 1,000 J/liter to 1,000 - 2,000 J/liter. The removal of PCE, and production of the desired byproduct HCl is shown in Figures 29 and 30. It is unclear whether the missing chlorine is due to surface losses within the SDP's stainless steel plumbing, or measurement error. Other byproducts identified in the SDP exhaust are shown in Figure 31. The data in Figure 32, showing acetone removal levels as a function of time and SDP power, is similar to Figure 25. However Figure 32 replots acetone removal by the number of nine's removed (1 nine is 90% destroyed, 2 nines is 99% destroyed) showing that the number of nine's removed scales roughly as the applied energy density. During the peak energy density of 2 kJ/liter (from 5 am to 9 am on 11/6/96) acetone was reduced to below the FTIR detection limit of 0.1 ppmv.

Figure 25: FTIR data summary from tests on cleaning hood - acetone removal.
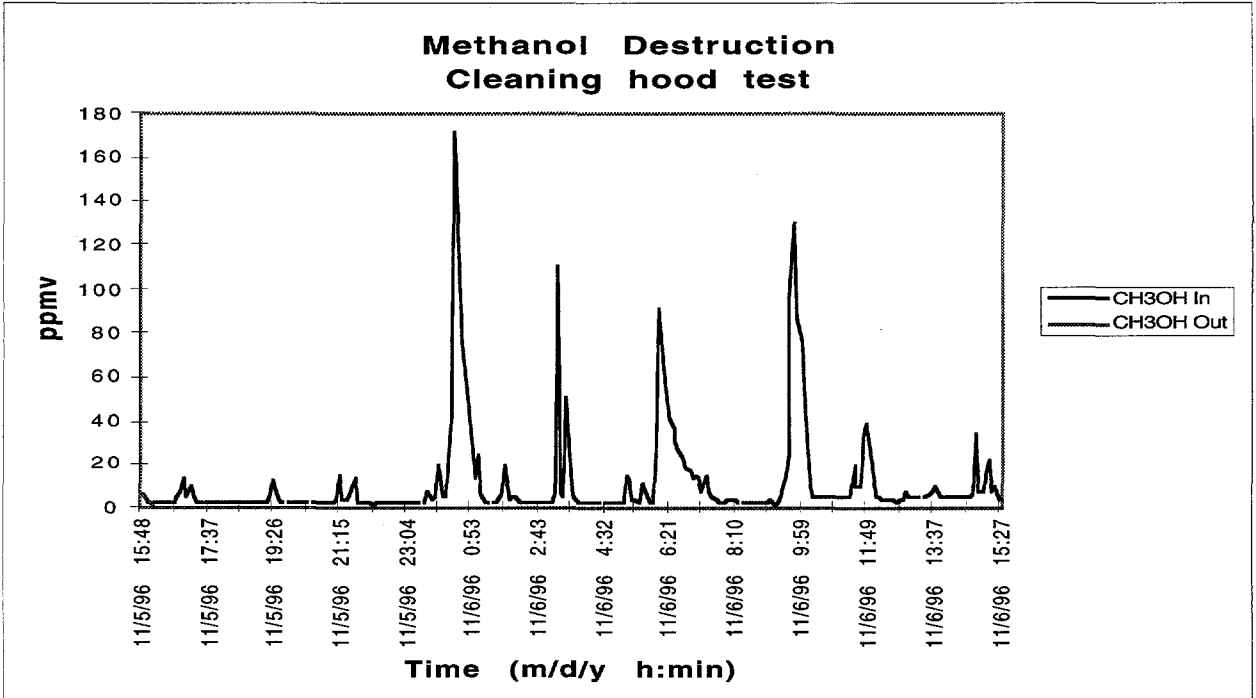
Figure 26: FTIR data summary from tests on cleaning hood - methanol destruction.

Figure 27: FTIR data summary from tests on cleaning hood- CO, CO_2 and ozone generated in SDP cells

Figure 28: FTIR data summary from tests on cleaning hood - NO_2, N_2O and HNO_3 generated in SDP cells
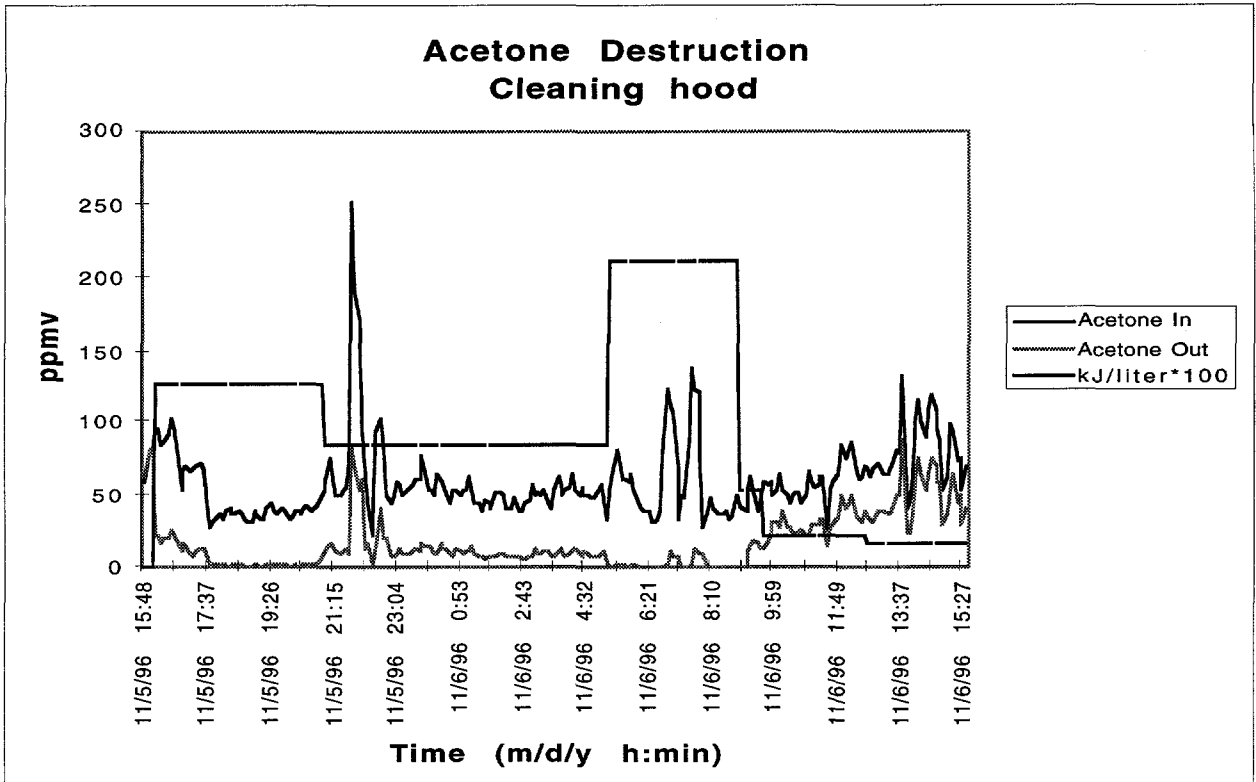
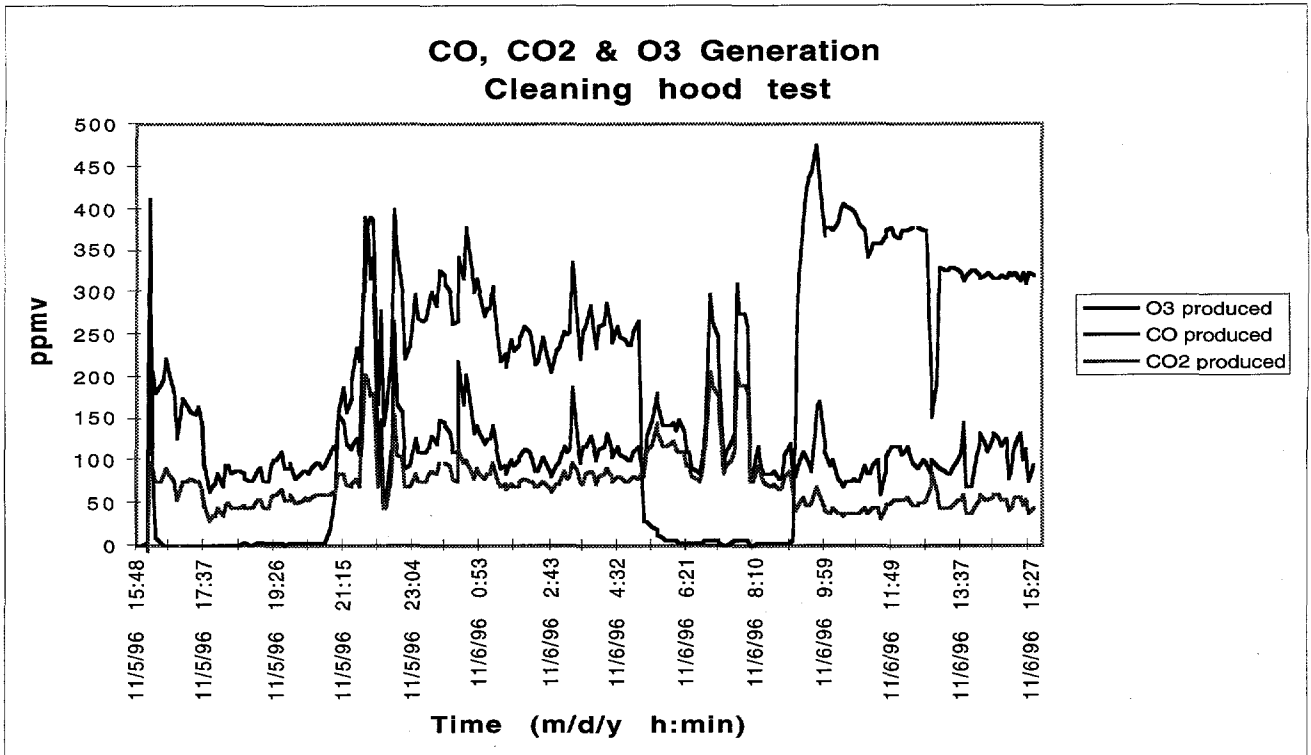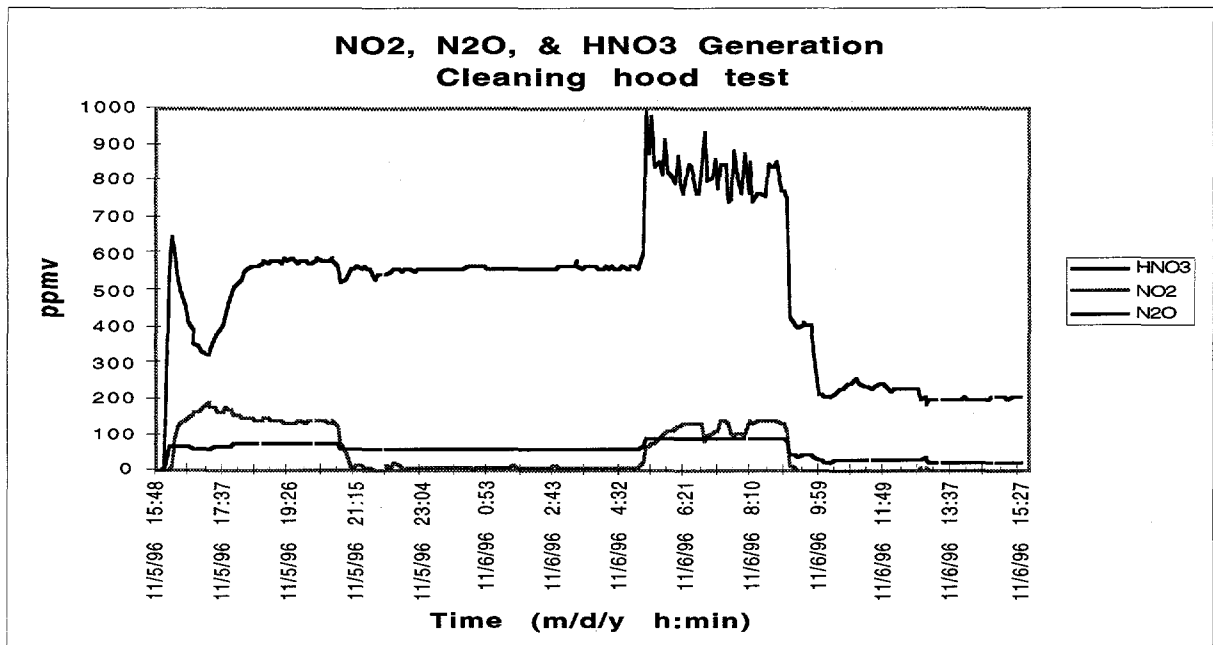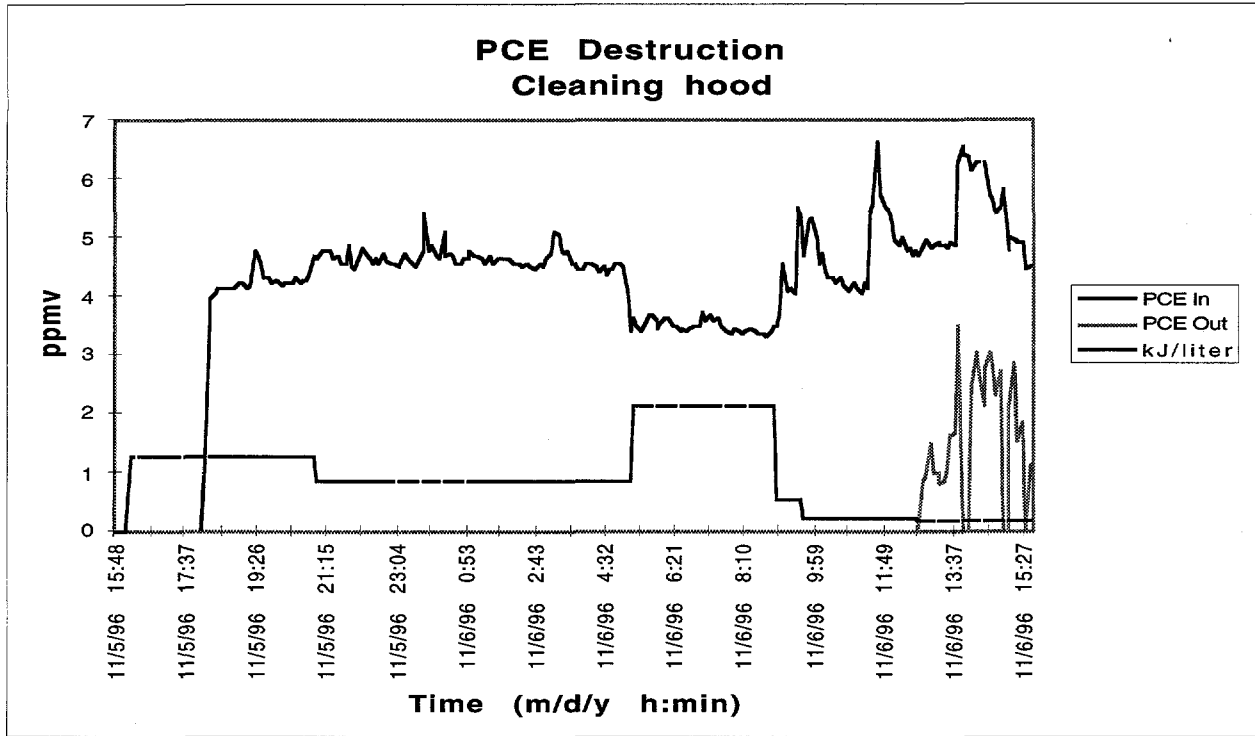Figure 29: FTIR data summary from tests on cleaning hood - perchloroethylene removal.
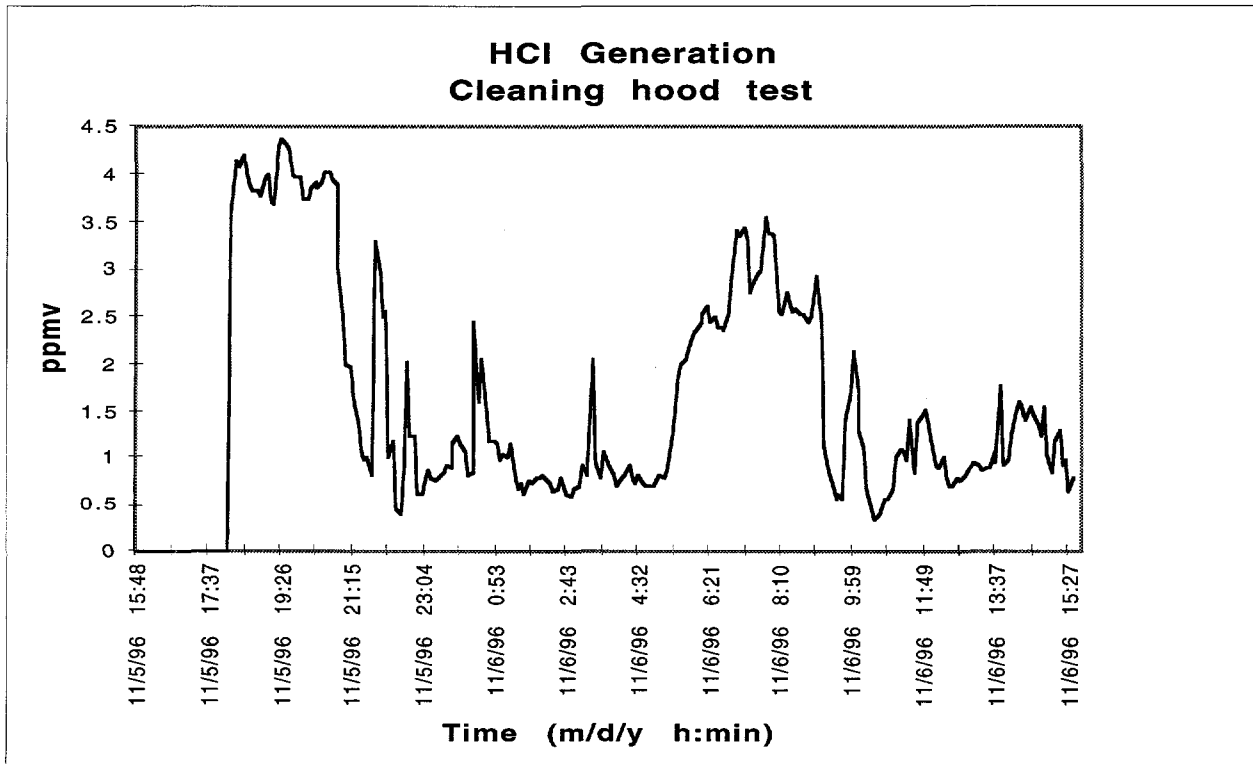

Figure 30: FTIR data summary from tests on cleaning hood - HCl generated in SDP cells
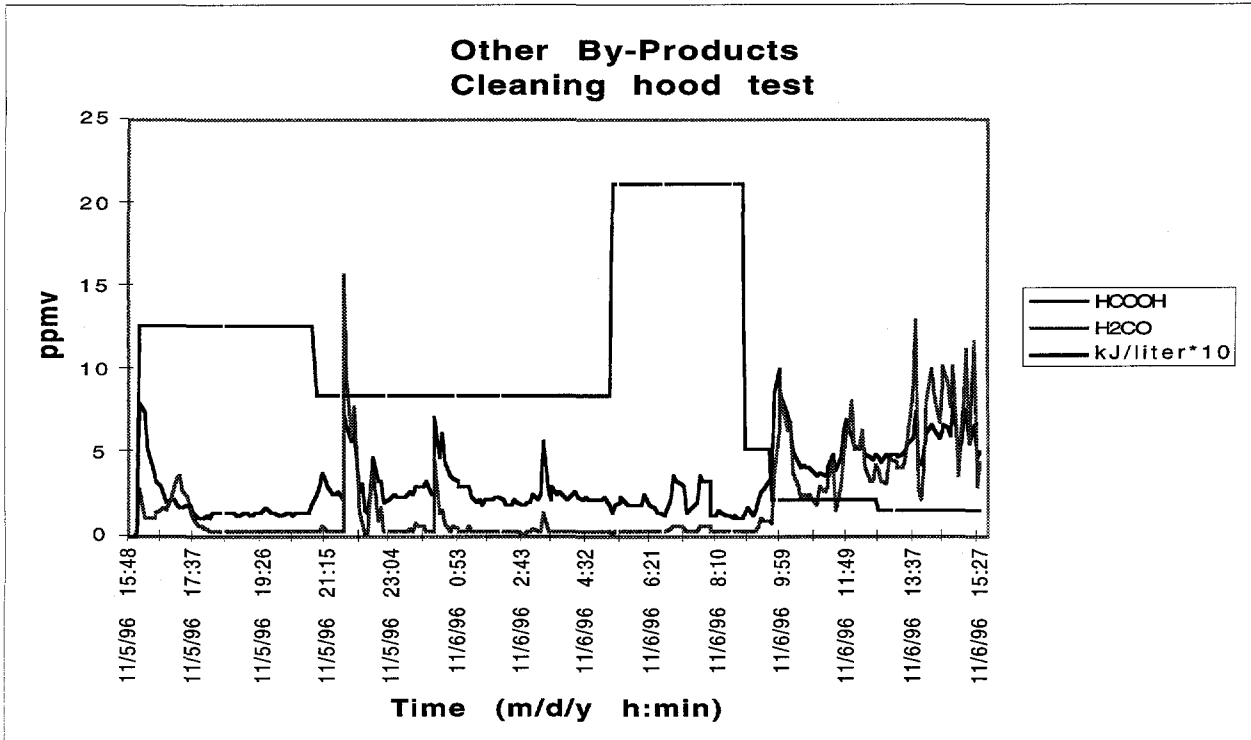
Figure 31: FTIR data summary from tests on cleaning hood - other tentatively identified compounds.
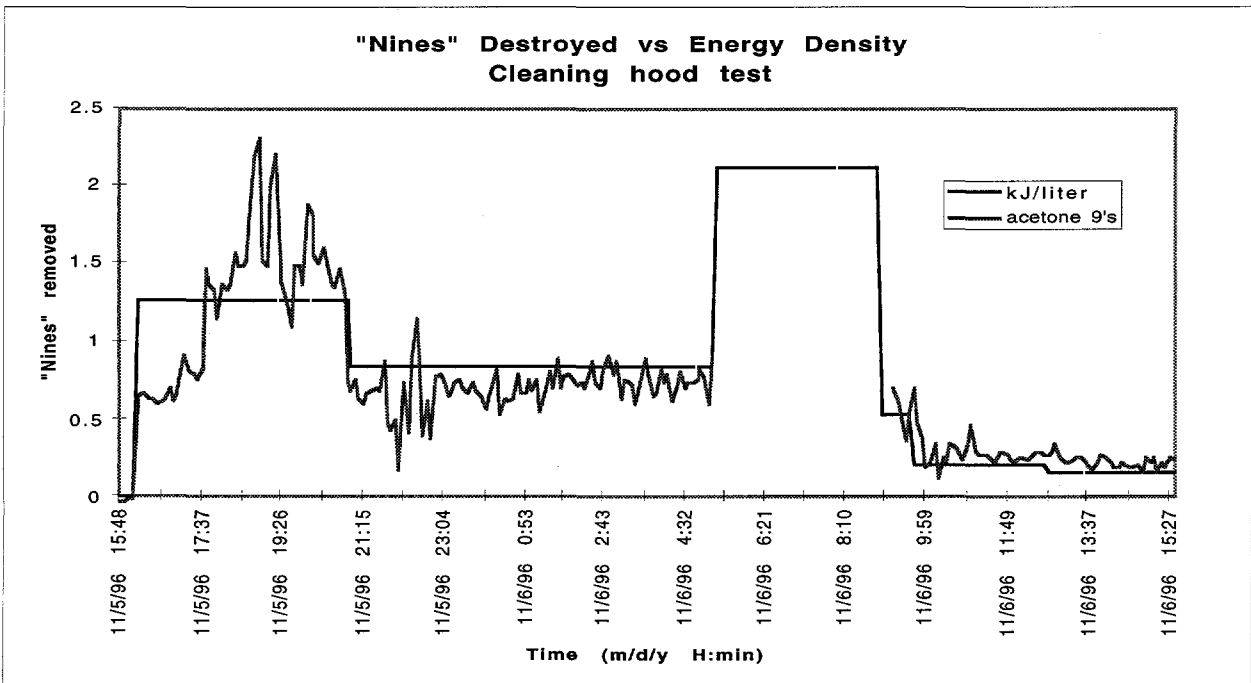
Figure 32: FTIR data summary from tests on cleaning hood - comparison of acetone removal and power density.

5.2.3 Summary Field Tests : Byproducts and DE

The principle decomposition products are CO, CO_2, and water. At low powers and temperatures ozone concentrations can exceed 200 ppmv. At higher powers HNO_3 is produced which could corrode ductwork. Other byproducts of interest are the oxides of nitrogen, which at reasonable energy densities is generated at levels typically less than 10-20 ppmv. The higher power densities used to destroy acetone can produce more NOx, but it is unlikely that the economics of those systems would allow their implementation. During the input spikes of methanol and PGMEA, formaldehyde, formic acid and acetic acid have all been identified in the SDP exhaust. Hydrogen chloride is produced when chlorinated compounds are present.

Within experimental error, the measured DE of the target compounds during pilot scale testing are all the same as those predicted by the laboratory tests. With adequate SDP powers, input levels of 0 to 200 ppmv of PGMEA, 5 to 500 ppmv of HMDS, 50 to 250 ppmv of acetone, 0 to 170 ppmv of methanol, and 0 to 6 ppmv of PCE were all destroyed to the target 95% or to below the detection limit of the FTIR. PGMEA was consistently reduced by >99%, HMDS by >90%, acetone by >90% and methanol by >99%. PCE removal is more difficult to quantify due to the low levels in the input, but for all but the lowest power levels output concentrations were below the detection limit (1 ppmv) of the FTIR.

6.0 Cost of Ownership

The SDP technology has not yet been demonstrated at full-scale over a long-term period. For this reason, there is necessarily some uncertainty associated with operation and maintenance costs. At the present stage of maturity costs shown can be expected to fall in the +30%/-15% range. Cost projections are based upon lab and field tests and vendor recommendations. Values provided for the SDP power supply and related equipment are based upon retail prices and do not take into consideration discounts that may apply for quantity. Finally, it is commonplace for thermal technologies (i.e., RTOs) to present unit costs in $/1000 scfm because it is economically favorable to do so for systems that treat small concentrations of contaminants in large volumes of air. However, as POU emissions control takes the opposite approach, unit costs in $/lb. VOC destroyed may be a more appropriate way to judge SDP cost of ownership. We present both metrics.

6.1   Cost Estimates

Table 4 presents the cost of ownership for SDP in terms of unit costs in both $/lb. of VOC destroyed and $/1000 scfm treated. Total annual costs and unit costs are dependent upon values given for the important parameters of flow rate, energy density requirement for a particular compound, desired DE, and exhaust stream duty cycle. Table 4 presents a summary of costs for lithotrack emissions control. A flow rate of 250 scfm and an energy density requirement of 50 J/liter per nine destroyed are assumed. A duty cycle of 25% is assumed. Table 4 does not include cost projections for treatment of acetone/methanol due to the high energy density requirements (>1000 J/liter) and excessive air flow rates (>3000 scfm) associated with solvent bench exhausts. Table 4 is a static depiction of the cost of ownership, a dynamic sensitivity analysis is presented in Table 5 showing the relationship of the major parameters to cost.

Table 4. Cost of Ownership for point-of-use SDP treatment of Lithotrack emissions.

	Litho Tool
Target Compound	**PGMEA**
Molecular Weight	132.16
Influent Concentration (ppmv)	50
Allowable Effluent Conc. (ppmv)	0.5
Target DE	99%
Nine-factor (based on Target DE)	2
Peak Flow Rate (scfm)	**250**
Energy Density Required (J/liter)	**50**
Power Requirement (kW)	12
VOC Loading (lb./hr)	0.28
Equipment Cost	
Power Supply Size Factor ($/W)	$1.50
Power Supply	$17,700
Cells and Support Equipment	$1,200
Sales tax (3%)	$567
Freight (5%)	$945
Total Equipment Cost (TEC)	**$20,400**
Indirect Costs (0.25 x TEC)	$5,100
(incl. piping, elec., installation)	
Total Capital Cost (TCC)	**$25,500**
Operating & Maintenance Costs	
Operating Labor (0.02 x TCC)	$520
Maintenance Labor	$1,900
Maintenance Materials (0.04 x TCC)	$1,000
Footprint (total equip sq. ft.)	12
Floor Space Unit Cost ($/sq.ft.)	$75
Floor Space Annual Cost	$900
Total O&M	$4,300
Utilities	
Electricity Unit Cost ($/kWhr)	$0.05
Duty Cycle	25%
Electricity Usage (kWhr/yr.)	25,835
Annual Electricity Cost ($/yr.)	$1,290
Annual Capital	
(@ 7.0%, 10 yr. term)	$3,630
Annual Operating Cost	$520
Annual Maintenance	$2,900
Annual Utilities	$1,290
Total Annual Cost	**$8,300**
PV 10 yr. O&M Cost (at 2.8%)	$40,600
Total PV Cost (TCC + 10 yr. O&M)	$66,100
Annual VOCs Destroyed (lb./yr)	2,420
CoO ($/lb. VOC)	**$3**
CoO ($/1,000 scfm)	**$33,200**

As summarized in Table 5, the four major parameters that influence SDP cost of ownership are flow rate, energy density (i.e., power) requirement, target effluent concentration or destruction efficiency, and duty cycle (the percentage of time that the exhaust output is actually being emitted). The total annual cost rises with the increase in flow rate; however, the unit cost stays relatively constant due to the linear dependence of cost on flow rate.

The unit cost of the power supply, the major capital investment for an SDP system, decreases as the size of the power supply increases. This is significant because it results in some economy of scale for the SDP systems. Referring to Table 4, for PGMEA destruction at 99% the unit cost is $33,300 in $/1,000 scfm; however if we want to treat the combined flow from 10 lithography tools using a larger SDP system, a 2500 scfm flow rate would require 118 kW of power instead of only 12 kW for the 250 scfm flow rate. The power supply unit cost decreases by half at this level of electrical power (> 100 kW ==> $0.75/W; < 15 kW ==> $1.50/W), so the total unit cost for the larger SDP system would decrease to approximately $23,700. In addition, the annual cost for a system designed to treat 2500 scfm is less than the cost of 10 SDP units designed to treat 250 scfm -- $59,000 as opposed to 10x the annual cost ($8,400) of the smaller systems = $84,000.

The energy density requirement is empirically derived from the specific compound(s) of interest in the exhaust stream. For example, the energy density required to destroy isopropanol (200 J/liter) is four times greater than that required for PGMEA (only 50 J/liter). The energy density is a function of power, so an increase in the energy requirement means a necessary increase in the power supply at a fixed flow rate, and hence, capital cost and utilities. From Table 5 it is evident that unit cost is especially dependent on the compound of interest. Similarly, each "nine" increases the power required to maintain the energy density for greater percentage destruction of a given compound. Inlet concentration does not effect the cost of ownership. There is little cost increase with an increase in duty cycle. This is because duty cycle does not affect the design specifications for the power supply, so capital cost is independent of the duty cycle. The change in cost as duty cycle increases reflects an increase in electrical utility cost requirements. Details on the specific inputs used in Table 4 and 5 are shown in Appendix 1.

Table 5. Summary of sensitivity data.

Flow Rate				DE		
Flow rate	Annual cost	Unit cost		Target DE	Annual cost	Unit cost
(scfm)		($/1,000 scfm)		(%)		($/1,000 scfm)
85	$2,800	$33,300		90	$4,200	$16,700
100	$3,500	$33,300		99	$8,300	$33,300
145	$4,800	$33,300		99.9	$12,500	$50,000
191	$6,400	$33,300		99.99	$16,700	$66,700
200	$6,700	$33,300				
265	$8,800	$33,300				
500	$16,700	$33,300				
636	$21,200	$33,300				
706	$23,500	$33,300				
911	$30,400	$33,300				
1000	$33,300	$33,300				
assume energy density req. = 50J/l				assume flow rate = 250 scfm		
assume DE = 99%				assume energy density = 50 J/l		
assume duty cycle = 25%				assume duty cycle = 25%		

Energy Density				Duty Cycle		
Energy req.,	Annual cost	Unit cost		Duty cycle	Annual cost	Unit cost
(J/l)		($/1,000 scfm)		(%)		($/1,000 scfm)
40	$6,700	$26,000		5	$7,300	$29,200
50	$8,300	$33,600		10	$7,600	$30,200
75	$12,500	$50,400		25	$8,300	$33,300
100	$16,700	$67,200		50	$9,600	$38,500
200	$33,300	$133,200				
400	$66,700	$266,000				
800	$133,000	$534,000				
assume flow rate = 250 scfm				assume flow rate = 250 scfm		
assume DE = 99%				assume energy density = 50 J/l		
assume duty cycle = 25%				assume DE = 99%		

6.2 Summary CoO

SDP shows optimal performance under a high concentration, low-flow regime. It is not particularly sensitive to influent exhaust stream concentration, but is very sensitive to flow rate, specific exhaust compound(s), and the target DE. Cost of ownership calculations depend upon these latter three parameters. This results in both advantages and disadvantages to the system. If the target compounds are favorable to destruction via SDP, such as those emitted by lithography tool exhaust (i.e., PGMEA, EEP), and if flow rates are relatively low (i.e. less than 500-1000 scfm), then the cost of ownership is competitive. If, on the other hand, target compounds are

unfavorable to SDP treatment, such as those emitted by organic solvent benches (i.e., acetone), then cost of ownership becomes prohibitive, particularly at the high flow rates (3000-5000 scfm) associated with solvent bench exhaust output. As we have seen, cost is also dependent on the specific target destruction efficiency, or nine-factor. Increasing the required destruction efficiency to 99.9% or greater is technically achievable, but operating costs increase accordingly.

This cost of ownership calculation was performed by LANL's Environmental Technology Cost Savings Analysis Project (ETCAP) [14]. Costs scale with total flow, DE, and target compound. Total capital costs (including installation) for a POU lithotrack tool, treating 250 scfm to > 99%, are ~$26,000. Annual operating expenses (maintenance, utilities and depreciation of capital expenses) of this device are approximately $8,300. Annual expenses for this system are $33,300 per 1000 scfm.

7.0 Commercialization

In 1992 LANL and the Electric Power Research Institute (EPRI) entered into a cooperative research and development agreement, or CRADA, to transfer the SDP to industry. Through a competitive call for proposals High Mesa Technologies (HMT) of Santa Fe, NM was selected as the third partner to commercialize and manufacture SDP equipment. HMT has subsequently acquired the licensing rights from LANL for the SDP for off-gas control. Based on the results of the tests described in this report HMT plans to design and build a prototype SDP system optimized for lithotrack off-gases (high flow/low power with particulate collection). This POU tool will have a flow capacity of 100 to 500 scfm, will be built using low flow impedance cells to avoid pressure build up, and will consume 10-15 kW of power.

As described in the previous section, a critical component of this technology is the power supply. The costs of the supply dominate all the fixed expenses within the cost of ownership calculations. LANL and HMT have recently started a $100,000 effort (supported by LANL and the DOE) to optimize new high power electronic circuits developed at LANL to drive SDP cells. If successful, this effort could reduce the cost of power supplies, and therefore reduce the cost of ownership of the technology, by as much as 50% for all applications.

HMT is currently seeking opportunities to demonstrate their tool, and anticipates commercial units will be available in 12-18 months. Any member companies interested in a demonstration can contact Peter Chase of HMT at jkjn38a@prodigy.com or 505-988-8979 or 505-988-4440.

8.0 Summary / Conclusions

SDP may be a cost effective treatment system for specific POU applications. While the SDP has achieved the target destruction or higher for the VOCs tested, the capital costs of the power supply limit its field of use. At this point, the cost of ownership associated with treating high flow (>1,000 scfm) streams of acetone/methanol are prohibitive because of the costs of the power supply. The most significant finding of this work is the ability of SDP to operate with streams containing HMDS. The low energy densities (power/flow) required for destruction of lithotrack organic vapors suggest that, compared to other emerging technologies, the SDP is a cost effective POU tool. Field testing of a prototype unit at a member company confirmed these laboratory findings. Analysis of the field test data has identified byproduct formation as a potential issue for implementation and additional work is required to respond to that concern. Future SDP systems can control these byproducts by either adjusting SDP power (e.g., more power will reduce formaldehyde emissions), or adding a post-SDP treatment (e.g., an acid scrubber). For specific VOC streams, cost-of-ownership calculations demonstrate that the SDP system can compete with conventional and regenerative thermal oxidation for low flow streams containing HMDS, PGMEA, EEP, and IPA. Commercialization efforts are advancing and POU systems optimized for lithotrack tools are anticipated within 18 months.

9.0 Acknowledgments

10.0 References

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Appendix 1:   Basis of Cost Estimate

A spread sheet was developed using the SEMATECH cost of ownership guidelines. The input to that spread sheet is summarized in the following section.

Row 1   **Title** and **date** of latest revision

Row 2   Applicable **Operational Component** within semiconductor fab

Row 3   The **Target Compound** dictates the SDP energy density requirement. Some compounds require much greater input power to destroy than others. For solvent benches we assume a mix of IPA, methanol, and acetone. Acetone is the designated target constituent because it requires the highest energy density for destruction. For dryers, IPA is the target. For litho tools, PGMEA is the target compound, but HMDS is also readily destroyed at an equivalent energy density.

Row 4   The **Molecular Weight** of the target compound is presented for calculating the VOC loading and subsequent unit cost in $/lb. VOC destroyed.

Row 5   **Influent Concentration** to the SDP treatment system in ppmv, averaged over time. The exhaust emission spikes have been measured at approximately 200 ppmv. Assuming actual output occurs 25% of the time, we give a an average value of 50 ppmv.

Row 6   The **Allowable Effluent Concentration** as governed by state and federal air emissions regulations. For purposes of cost calculation we assume 90% or 99% destruction will be required.

Row 7   **Target DE** = 1 – (B6/B5) This result gives the required percent destruction which is used in calculating the power requirement.

Row 8   The **Nine-factor** is the number of orders of magnitude of desired destruction of the target compound; thus 99% DE is equivalent to a required nine-factor of 2.

Row 9   **Peak Flow Rate** is the maximum output air flow rate assumed to apply to the given operational component.

Row 10   **Energy Density Required** is the empirically derived amount of pulsed power necessary to destroy one nine-factor (i.e., 90%) of the target compound.

Row 11   **Power Requirement** = (B10*B9*B8*3.785*7.48)/60/1000 This result yields the necessary size of the power supply that drives the SDP system. It is dependent upon the energy density required, flow rate, and desired DE.

Row 12   **VOC loading** = (((B9/0.1336)*3.785*60*(B5/1000000))/22.4)*B4/454. The loading is a function of influent concentration, flow rate, and the molecular weight of the target compound.

Rows 15–19   **Equipment Cost**
                Power Supply Size Factor is a function of the power requirement. Costs for power supplies under 5kW are assumed to be $2/W; from 5kW up to 15kW costs

are \$1.50/W; from 15kW to 100kW, \$1/W, and >100kW systems are costed at \$0.75/W (Elgar Power, 1996).
Power Supply = ROUND(B11,)*1000*B15 This is the single largest cost associated with SDP and dependent upon the power requirement and power supply size factor.
Cells and Support Equipment =ROUND(B11,)*100 include primarily Pyrex and aluminum to construct the SDP cells and holding tank; this cost is based on \$0.10/W, a percentage of the power requirement.
Sales Tax is assumed to be 3% of power supply and support equipment.
Freight is assume to apply to 5% of power supply and support equipment.

Row 20   **Total Equipment Cost (TEC)** is the sum of the power supply, cells and support equipment, sales tax, and freight.

Row 21   **Indirect Costs** are 25% of TEC and include the piping, electrical, labor, and G&A costs incurred to install the system. Installation requires 2 man-days once piping and electrical hookups are completed.

Row 23   **Total Capital Cost** is the sum of TEC and indirect costs.

Rows 26–31 **O&M Costs**
    Operating Labor is considered to be approximately 2% of TCC, as the SDP equipment is self-sustaining and only requires periodic monitoring.
    Maintenance Labor = B11*0.1*32*50
Based upon 10% of power requirement. For a 10kW system, it is assumed that SDP cells will need cleaning twice annually, requiring 2 employees 2 days for disassembly, assembly, and cleaning. Larger systems have a greater number of cells; hence the dependence on power requirement.
    Maintenance Materials are conservatively costed at 4% of TCC.
    Footprint takes into consideration both power supply and the SDP cell holding tank dimensions. Approximate equipment footprints are as follows:

10kW:	2' x 4' tank	$12 ft^2$
	2' x 2' power supply	
20kW:	3' x 4' tank	$16 ft^2$
	2' x 2' power supply	
50kW:	6' x 4' tank	$30 ft^2$
	3' x 2' power supply	
100kW:	6' x 8' tank	$56 ft^2$
	4' x 2' power supply	

    Floor Space Unit Cost is based on \$75/sq. ft. (Jassal, 1996).
    Floor Space Annual Cost is the footprint x floor space unit cost.

Row 32   **Total O&M** =SUM(B26+B27+B28+B31) This is the sum of O&M labor, maintenance materials, and floor space.

Rows 35–38 **Utilities**

Electricity Unit Cost is the price per kWhr for electricity and is assumed
     to be $0.05

Duty Cycle is the percentage of time that the output exhaust from a particular tool is actually being emitted. 25% is considered a conservative estimate.

Electricity Usage = 8760*B11*B36 This value is dependent upon power requirement and duty cycle, and assumes continuous operation.

Annual Electricity Cost = electricity unit cost x usage.

Row 41   **Annual Capital** = 0.1423*B23 The multiplier is based on amortization of TCC at 7% over a 10-year term.

Row 42   **Annual Operating Cost** is the same as operating labor (Row 26).

Row 43   **Annual Maintenance** is the sum of maintenance labor and materials.

Row 44   **Annual Utilities** is the same as annual electricity cost (Row 38).

Row 45   **Total Annual Cost** is the sum of amortized capital, operating, maintenance, and utilities.

Row 46   **Present Value 10-year O&M Cost** = 8.618*(B42+B43+B44)
This value is the total cost of O&M plus utilities over 10 years. The multiplier is based on the current discount rate of 2.8%.

Row 47   **Total Present Value Cost** is the sum of the present value 10-year O&M cost plus the TCC.

Row 48   **Annual VOCs Destroyed** are the pounds per year of target exhaust compounds treated, assuming continuous operation (VOC loading x 8760 hours).

Row 49   **Cost of Ownership** = B45/48 This value is the unit cost in $/lb. VOCs destroyed, and is derived by dividing the total annual cost by the annual VOCs destroyed.

Row 50   **Cost of Ownership** = ROUND((B45/B9)*1000,-2) This is COO in terms of $/1,000 scfm and is calculated by dividing total annual cost by the peak flow rate (Row 9).