



Project Summary

Onsite Solvent Recovery

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This study evaluated the product quality, waste reduction/pollution prevention, and economic aspects of three technologies for onsite solvent recovery: atmospheric batch distillation, vacuum heat-pump distillation, and low-emission vapor degreasing. The atmospheric and vacuum distillation units were tested on spent methyl ethyl ketone and spent methylene chloride, respectively. Samples of spent, recycled, and virgin solvents at two industrial sites underwent physical and chemical tests to determine solvent quality. The quality of the recycled solvent was found to be acceptable for use in the specific applications. Significant waste reduction was achieved by reducing the volume of spent solvent to a few gallons of distillation residue needing disposal.

The low-emission vapor degreaser is a fully enclosed alternative to conventional, open-top vapor degreasing. It was found to reduce air emissions by more than 99%, compared to a conventional vapor degreaser of the same production capacity.

Compared to disposal, the atmospheric and vacuum distillation units reduced operating costs significantly. The estimated payback period for these units was found to be less than 2 yr. The low-emission vapor degreaser reduced operating costs by reducing solvent losses and labor costs. The estimated payback for this unit was approximately 10 yr. The cost estimates were based on a full range of considerations including equipment, engineer-

ing, installation, operation, maintenance, and energy use. The estimates did not, however, include potential changes in liabilities or impacts due to regulations planned or in the process of being implemented.

This Project Summary was developed by EPA's Risk Reduction Engineering Laboratory, Cincinnati, OH, to announce key findings of the research project that is fully documented in a separate report of the same title (see Project Report ordering information at back).

Introduction

This study, performed under the U.S. Environmental Protection Agency's (EPA's) Waste Reduction and Innovative Technology Evaluation (WRITE) Program, was a cooperative effort between EPA's Risk Reduction Engineering Laboratory (RREL) and the Washington Department of Ecology. The objective of the WRITE Program is to evaluate, in a typical workplace environment, examples of prototype or innovative commercial technologies that have potential for source reduction or recycling. The study evaluated three technologies for recovering and reusing waste solvent on site: atmospheric batch distillation, vacuum heat-pump distillation, and low-emission vapor degreasing. Comparing the three units was not an objective of this study. Rather, the suitability of each technology to its respective application was examined. In each technology category, a specific unit offered by a specific manufacturer was tested. Other variations of these units (with varying capabilities) may be available from several vendors.



The two liquid-distillation units were tested at industrial sites that have purchased and are using the units. The atmospheric unit was tested on spent methyl ethyl ketone (MEK) at a site where MEK is used to clean the spray painting lines between colors. The recycled solvent was reused for the same purpose, with the residue shipped off as hazardous waste. The vacuum unit was tested on spent methylene chloride (MC) at a site that manufactures wires and cables. The MC is used for cold (immersion) cleaning of wires and cables to remove markings (ink).

Atmospheric Batch Distillation and Vacuum Heat-Pump Distillation

Atmospheric distillation is the simplest technology available to recover liquid spent solvents. Units that can distill as little as 5 gal or as much as 55 gal/batch are available. Some units can be modified to operate under vacuum for higher-boiling solvents (>135°C). Contaminant components with lower boiling points than the solvent or that form an azeotrope with the solvent cannot be separated (without fractionation) and may end up in the distillate. The unit used in this study (Figure 1) was Model LS-55D,* manufactured by Finish Thompson, Inc. The distillation residue,

* Mention of trade names or commercial products does not constitute endorsement or recommendation for use.

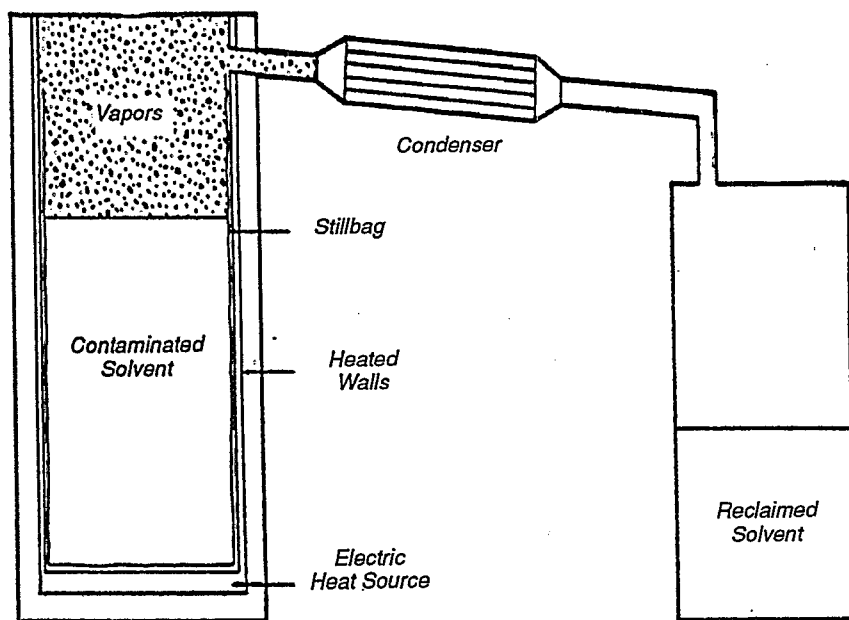


Figure 1. Atmospheric distillation unit. (Source: Finish Thompson, Inc.)

often a relatively small fraction of the spent solvent, is disposed of as hazardous waste.

The vacuum unit tested, Model 040 is manufactured by Mentec AG in Switzerland and supplied in the United States by Vaco-Solv Chicago, Inc. It is configured similar to a conventional vacuum distillation system except that the pump, in addition to drawing a vacuum, functions as a heat pump (Figure 2). No external heating or cooling is applied. The heat pump generates a vacuum for distillation and compresses vapors for condensation. Model 040 is suitable for solvents with boiling points up to 80°C. Spent solvent is continuously sucked into the evaporator by a valve. The vacuum drawn generates vapors, which are sucked into the heat pump, compressed, and sent to the condenser. The temperature stabilizes automatically according to the specific solvent and the ambient air. The condenser surrounds the evaporator to allow heat exchange between the cool spent solvent and the warm condensing vapors.

The product quality objective for the two liquid-distillation units was to show that the recycled solvent was of sufficient quality for reuse. One 55-gal drum of spent solvent was processed each day through the batch and continuous units. For each unit, one drum of spent solvent was pro-

cessed in ~12 hr. The atmospheric unit left 16 gal of residue and the vacuum unit left 3 gal. The amount of residue left behind is a function of the application and not the distillation units. Samples of the spent and recycled solvents were analyzed by standard ASTM methods to determine the improvement in quality. Virgin solvent samples also were collected at each site and subjected to the same tests for comparison.

During the vacuum unit test, the "virgin" sample was found to be a sample of MC obtained by the site from a solvent recycling company. The "virgin" solvent specifications meet the requirements for the company's application, and it has been used satisfactorily at the site in the past. The vacuum unit was being operated at a faster rate than recommended by the manufacturer. Because the unit's built-in condenser-evaporator heat exchange was not sufficient for this rate, site personnel had attached an air-cooled condenser at the outlet to restrict vapor loss to ~4 gal/55 gal of spent solvent. To prevent the release of this vapor into the work area, the vapor was led through a pipe to the roof of the facility and discharged per state regulations.

Table 1 shows the characterization results for samples from the atmospheric and vacuum units. In appearance and color, the spent samples varied vastly from the clear recycled and virgin samples. All the measured parameters showed a significant improvement from spent to recycled samples but were not quite up to virgin grade. The water content increase in the recycled samples from the atmospheric unit was traced to a slight leakage from the water-cooled condenser that was worn out due to long use. Repairing the leak after the testing, site personnel reported that the problem had been corrected.

MEK purity of the recycled sample from the atmospheric unit substantially increased from 78% to ~85%. The large decrease in nonvolatile matter during recycling accounts for most of this increase. Of the 15% impurity in the recycled sample, 5% is water as discussed above. The remaining 10% impurity probably is due to the codistilling out of paint thinner solvents (proprietary blends) present in the spent solvent. MC purity of the recycled solvent from the vacuum unit was 86%, comparing favorably with the "virgin" sample purity of 90%.

Some performance characteristics of MC (a halogenated solvent) also were evaluated. The pH of the water extract of the recycled solvent was fairly close to the "virgin" value of 7. The spent sample pH

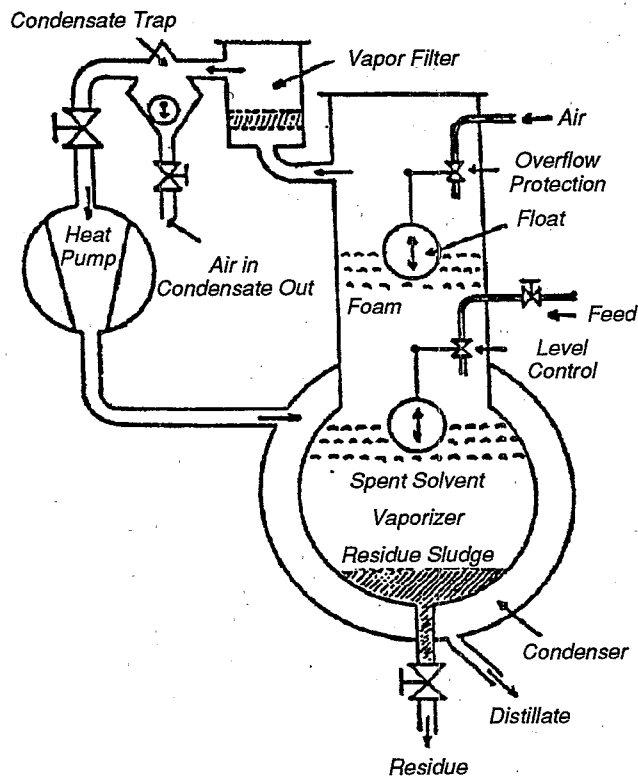


Figure 2. Vacuum heat-pump distillation unit. (Source: Vaco-Solv Chicago, Inc.)

of 5 indicates the presence of potentially corrosive components. The corrosion test on steel and aluminum (ASTM D2251) yielded noticeable corrosion only in the case of the steel strip placed in the spent solvent sample. No such corrosion was

evident due to the recycled solvent, indicating that recycling improved the quality.

Table 2 shows the waste reduction achieved by the two distillation technologies at the respective sites. Through recycling, large volumes of spent solvent waste were reduced to small volumes of distilla-

tion residue, which is disposed of as RCRA hazardous waste. Both MEK and MC are hazardous chemicals listed on the Toxic Releases Inventory (TRI). These solvents also are on EPA's list of 17 chemicals targeted for 33% reduction by 1992 and 50% reduction by 1995.

The economic evaluation compares the costs of each new technology to conventional practice. Table 3 shows the major operating costs associated with disposal and the atmospheric batch unit. For the unit, recycling saved ~\$10,000/yr. The purchase price of the atmospheric batch unit is \$12,995. A detailed calculation based on worksheets provided in the *Facility Pollution Prevention Guide* (EPA, 1992) indicated a payback period of less than 2 yr.

For the vacuum unit (Table 4), savings from recycling are ~\$18,300/yr. An explosion-proof vacuum unit costs \$23,500. The payback period for this unit also was less than 2 yr.

Low-Emission Vapor Degreasing (LEVD)

LEVD currently is used in Europe, where vapor degreasers are regulated as a point source. Previous studies (Battelle, 1992) on conventional open-top vapor degreasers have shown that a large part of the solvent (more than 90% in some cases) is lost through air emissions, which are considerable even though vapor degreasers are required to have primary cooling coils (tapwater cooled) and a certain freeboard height. Air emissions are mainly workload-related, caused either by dragout of solvent on the workload itself (and subsequent vaporization) or by disturbance in the air-vapor interface during entry and

Table 1. Characterization of Solvent Samples

Sample	Appearance	Color ^a	Specific Gravity	Nonvolatile Matter mg/100 mL	Conductivity μ hos/cm	Water Content % by wt	Acid Acceptance ^b	Purity % ^c
Atmospheric Unit (MEK)								
Spent	Dark grey w/sediment	— ^a	0.845	6,951	7.05	1.89	NA ^f	78.41
Recycled	Clear	5	0.827	2.6	3.30	5.42	NA	85.02
Recycled Dup ^d	Clear	5	0.821	2.0	3.40	5.56	NA	85.54
Virgin	Clear	5	0.800	2.2	1.15	0.09	NA	99.09
Vacuum Unit (MC)								
Spent	Dirty grey-brown	— ^a	1.220	34,101	1,063	0.27	0.032	NA
Recycled	Clear	5	1.286	20.37	137	0.25	0.004	86.4
Recycled Dup ^d	Clear	5	1.288	17.88	136	0.24	0.005	NA
Virgin	Clear, tinge of yellow	10	1.298	57.16	36	0.14	0.003	90.1

^a On a scale of 5 to 500, with 500 being the darkest color. ASTM D1209 and D2108.

^b Measured as equivalent NaOH wt%. ASTM D2942.

^c Gas chromatography analysis based on ASTM D2804.

^d Duplicate analysis of the same sample.

^e Not comparable with standards. Sample was too dirty.

^f NA = not analyzed.

Table 2. Waste Reduction by Atmospheric and Vacuum Units

Wastestream	-Disposal Option -		- Recycling Option -	
	Annual Volume		Wastestream	Annual Volume
Atmospheric Unit Test Site:				
Spent MEK	880 gal		Distillation residue	262 gal
Drums	17 drums		Still bags	17 bags
			Cooling water	18,360 gal
			Drums	5 drums
Vacuum Unit Test Site:				
Spent MC	3,000 gal		Distillation residue	136 gal
Drums	55 drums		Air emission	218 gal
			Drums	3 drums
			Used oil	1 gal

Table 3. Major Operating Costs for Atmospheric Unit

Item	Annual Usage	Unit Cost (\$)	Annual Cost (\$)
Disposal Option			
Virgin solvent	880 gal	10.50/gal	9,240
Disposal			
- labor	8 hr	8/hr	64
- drums	17	40/drum	680
- disposal fee	900 gal	400/55 gal	6,545
		Total	16,529
Atmospheric Unit			
Virgin solvent	245 gal	10.50/gal	2,573
Operating labor	17 hr	8/hr	136
Routine maintenance			
- spare parts	1	86/each	86
- labor	12 hr	8/hr	96
Energy	1,265 kWh	0.04/kWh	51
Cooling water	18,360 gal	1/1000 gal	18
Disposal			
- labor	3	8/hr	24
- drums	5	40/drum	200
- residue disposal	262 gal	675/55 gal	3,215
- still bags	17	84/12 bags	119
		Total	6,518

exit of the workload. Other sources are convection and diffusion during startup, operation, idling, shutdown, and, to a small extent, equipment leaks. Air emissions are a concern for metal finishers because many solvents used in vapor degreasing have been targeted by EPA in the 33/50 Program. Environmental and Occupational Safety and Health Administration (OSHA) regulations have become more stringent.

Pollution control devices available for conventional vapor degreasers include increased freeboard height, refrigerated coils, and covers to eliminate drafts and reduce diffusion. In contrast, LEVDs are completely enclosed, airtight units. This evaluation used Model 83S (Size 1), manufactured in the United States by Durr Au-

tomation, Inc. Figure 3 shows its operation. About 1 hr before the shift begins, a timer switches on the heat to the sump. When the solvent in the sump reaches vapor temperature, the vapor is still confined to the enclosed jacket around the working chamber. The parts to be cleaned (workload) are placed in a galvanized basket and lowered into the working chamber. Loads can range from 330 to 110 lb (of steel parts) in this model. When the lid is shut and the unit is switched on, compressed air hermetically seals the lid shut for the duration of the cycle.

Table 5 shows typical cleaning cycle stages. During "vapor fill," solvent vapors enter the chamber from the outer jacket, and degreasing begins. During "conden-

sation," solvent vapors are condensed out by a refrigerated cooling coil at the bottom of the chamber. During "air recirculation," the air-solvent mixture is recirculated through a chiller to condense out more solvent. During "carbon heatup," solvent adsorbed in the previous cycle is released (desorbed) to the circulating air and condenses out in the chiller. During "adsorption," the chamber air is recirculated in the reverse direction—first through the chiller and then through the carbon. Most residual solvent vapor in the cold air is adsorbed on the carbon. A photoionization detector (PID) probe verifies that the chamber air has less than 1 g/m³ of solvent and signals the air compressor to release the seal on the lid to end the cycle. If the chamber air has more than 1 g/m³ of solvent, the cycle loops back to the desorption stage. The entire cycle is programmed and requires no operator attention except to load and unload the workload. Only a very small amount of solvent exhausts at the end when the lid is opened. The LEVD also works as a distillation unit to clean the liquid solvent in the sump. During distillation, the unit is switched on without any workload in the chamber.

Testing was conducted on the LEVD using perchloroethylene (PCE) solvent. Test runs were conducted on machined steel parts with and without cutting oil on the parts. Total cycle times were recorded for all completed runs. Because the same batch of parts was used for each run, parts were either cold (ambient) or hot depending on the cooling time between runs. Adding oil to the parts did not greatly affect the total cycle time, but the workload mass did. In all the runs starting with parts dipped in cutting oil, the cleaned parts were visually examined for traces of oil or dirt contamination. No contamination was noticed on the parts from any of these runs.

The pollution prevention aspect of the LEVD was the main focus of this technology. The completely enclosed design of the working chamber allows the potential for air emissions only when the cleaning cycle is complete and the lid is opened. Any solvent vapor not evacuated from the chamber during condensation or adsorption releases to the atmosphere.

Table 6 shows the total cycle times and emissions recorded from the LEVD by a flame ionization detector (FID) probe inserted (for this test) into the working chamber below the designated vapor level. FID measurements began during the adsorption stage and continued until after the lid was opened. A second FID probe (ambi-

Table 4. Major Operating Costs for Vacuum Unit

Item	Annual Usage	Unit Cost (\$)	Annual Cost (\$)
Disposal Option			
Virgin solvent	3000 gal	3.57/gal	10,710
Disposal			
- labor	16 hr	8/hr	128
- drums	55	40	2,200
- disposal fee	3000 gal	2.50/gal	7,500
		Total	20,538
Recycling			
Virgin solvent	246 gal	3.57/gal	878
Operating labor	55 hr	8/hr	220
Energy	985 kWh	0.04/kWh	39
Disposal			
- still bottoms	136 gal	2.50	340
- used oil	4 quarts	3.00/quart	12
- labor	3 hr	8/hr	24
- drums	3	40/drum	120
Routine Maintenance			
- oil	4 quarts	3.50/quart	14
- spare parts	— ^a	480 (max) ^a	480
- labor	16 hr	8/hr	128
		Total	2,255

^a The \$480 cost for spare parts is a maximum, which assumes that the manufacturer's recommendations are exactly followed and that the maximum number of parts will be replaced during each overhaul. Actual maintenance costs could be lower.

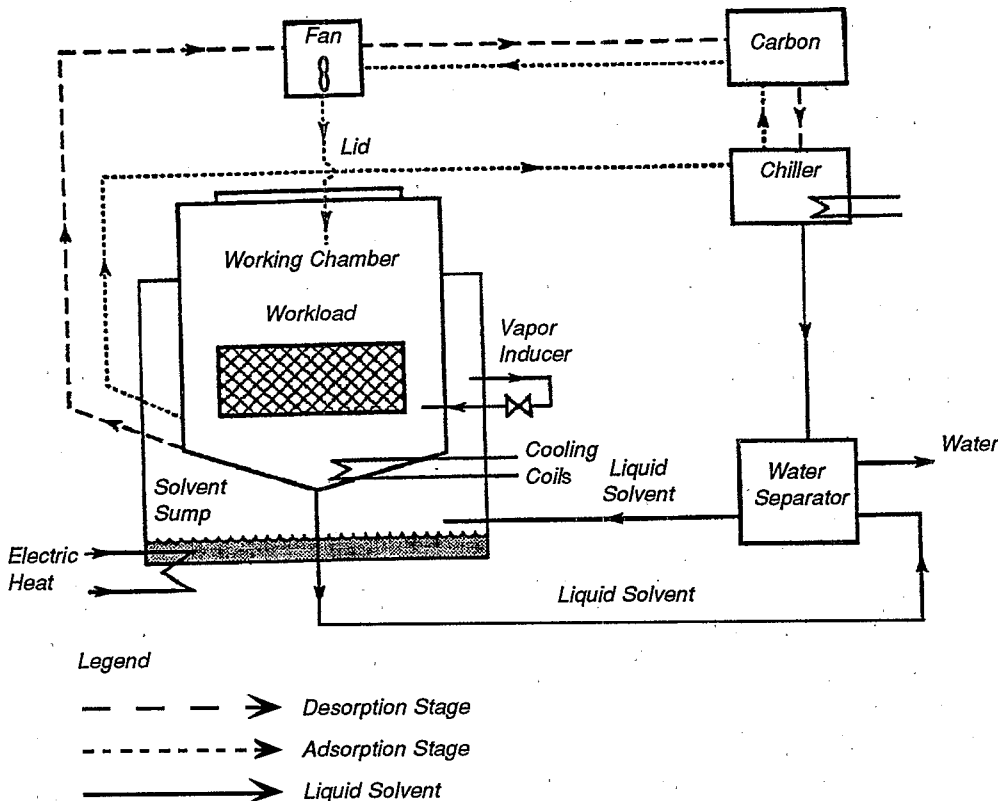


Figure 3. Low-emission vapor degreaser.
(Source: Durr Automation, Inc.)

ent), positioned outside the unit near the lid seal, took continuous measurements all around the unit during operation, with special emphasis around the lid to ensure leak-proof design. Ambient levels (3 to 4 ppm) in the indoor facility on the test days were consistent.

Figure 4 shows how a typical LEVD cleaning cycle ends. The same pattern was evident in the other runs. Time zero corresponds to the start of measurements when the FID probe in the working chamber was activated.

Just before the adsorption cycle ended, the chamber FID read 52 ppm, well below the targeted 1 g/m³ (150 ppm of PCE). When the lid was retracted, the chamber air had full access to the ambient. At this point, the chamber concentration dropped sharply as the residual solvent vapor in the chamber dispersed. The ambient FID probe showed a corresponding increase (to 6 ppm). Both FID readings soon stabilized to facility ambient levels (3 to 4 ppm).

Later, as the basket of cleaned parts was raised out of the chamber, the second FID probe was thrust into the basket near the parts. No elevated readings above ambient were sensed, indicating that the parts were free of solvent. Thus, there is a very small air emission from the LEVD when the lid is opened. In all the test runs, the solvent concentration was well below the targeted 1 g/m³ (150 ppm PCE), so 1 g/m³ is an achievable concentration. The volume of the working chamber is 0.6 m³. Assuming that all the residual solvent vapor (1 g/m³ maximum) in the chamber is discharged to the ambient area, the typical air emission through the opened top is 0.6 g (0.00132 lb)/cycle or less. It takes 1 hr to clean 560 lb of oiled steel parts. Therefore, the air emission from this LEVD mode is 0.00132 lb of solvent/hr.

A typical conventional open-top vapor degreaser cleaning at a similar rate (~ 560 lb of steel parts/hr) typically would emit 0.147 lb of solvent/ft²/hr (EPA, 1989), or 0.662 lb of solvent/hr from its 4.5-ft² opening during continuous operation. Therefore, the LEVD reduces air emissions by more than 99% compared to air emissions from the typical conventional open-top vapor degreaser (i.e., with a 0.75 freeboard ratio, primary cooling coil, electric hoist, and no lip exhausts) used in this calculation.

The OSHA exposure limit for PCE is 25 ppm for an 8-hr time-weighted average (TWA). Personnel air sampling (in accordance with OSHA guidelines) was not conducted during this evaluation, but PCE levels measured with the ambient FID at all times during operation (3 to 4 ppm)

Table 5. LEVD Cleaning Cycle

Stage	Vendor-Recommended Time Settings (sec)	Times Set for This Testing (sec)
Solvent heatup (once a day)	Variable ^a	Variable ^a
Solvent spray (optional)	10-180	not used
Vapor fill	Variable ^b	Variable ^b
Degreasing	20-180	60
Condensation	120	120
Air recirculation	120	120
Carbon heatup	Variable ^c	Variable ^c
Desorption	60	60
Adsorption	60-240 ^d	240

- ^a Requires ~1 hr on days following overnight shutdown when sump solvent temperature drops to 70°C. After weekend shutdowns, when sump solvent temperature drops to 20°C, it may take 1.5 hr for solvent to reach vapor temperature. Timer on unit allows automatic heatup.
- ^b Depending on the workload mass and type of metal. Varied from 8.5 min for 165 lb to 36.5 min for 915 lb of steel parts.
- ^c Carbon heatup took approximately 22.5 min during testing.
- ^d At 60 sec, if monitor shows that chamber concentration is above 1 g/m³, then the adsorption stage proceeds to the full 240-sec stage. This sequence repeats if necessary.

Table 6. Emissions from LEVD

Run No. ^a	Mass of Steel Parts (lb)	Final Chamber Concentration ^b (ppm)	Total PCE Emission ^c (lb/cycle)	Total Cycle Time (min)	Emission Rate (lb/hr)
1	165	52	0.0005	—	—
2	165	75	0.0007	39 ^f	0.0011
3	900	92	0.0008	67.5	0.0007
5	165 ^e	43	0.0004	50.5	0.0005
6	165 ^e	47	0.0004	40 ^f	0.0006
8	915 ^e	78	0.0007	69	0.0006
Target ^d	560	150	0.0013	60 ^g	0.0013

- ^a Runs 4, 7, and 9 were interrupted to allow other measurements.
- ^b At the moment when the seal on the lid is released.
- ^c Based on 150 ppm = 1 g/m³ of PCE and a chamber volume of 0.6 m³.
- ^d Normally the machine is programmed to release the lid when solvent concentration in the chamber falls below 1 g/m³ (150 ppm of PCE). This target was easily met in all the test runs.
- ^e Workload parts were dipped in cutting oil before the run.
- ^f Workload parts were already hot from being used in previous runs when inserted into working chamber. Hence, total cycle times for these runs are lower than normally expected.
- ^g Expected cycle time for 560 lb of steel parts (workload).

and at the edge of the chamber opening for about 2.5 min when the lid is retracted completely (<6 ppm) (Figure 4) are well under the OSHA exposure limit. The pollution prevention potential of this unit is further enhanced by its ability to perform as a liquid solvent distillation system for cleaning the sump solvent; this capability was not a part of this evaluation. When pollution prevention is an objective, the LEVD also affords greater production flexibility because it has none of the idling losses between loads or downtime losses during shutdown of the conventional degreaser.

Table 7 lists the LEVD's major operating costs and the operating costs for a conventional open-top vapor degreaser with similar production capacity. With a vendor-quoted purchase price for the LEVD of \$210,000, the unit results in savings in annual total operating costs of ~\$25,000 mainly from reduced labor costs (due to larger batch size) and lower solvent requirement (due to solvent recovery). The LEVD pays for itself in ~10 yr. The above is a straightforward cost comparison between the LEVD and a conventional vapor degreaser of similar production capacity. Other cost-benefit factors must be taken into account when making eco-

nomic decisions. The LEVD does not require capital and operating expenditures for auxiliary equipment that may be required for a standard conventional vapor degreaser (increased freeboard ratio, refrigerated coils, lip exhausts, room ventilation) in order to meet or anticipate increasingly stringent environmental and worker safety regulations. The LEVD is a self-contained unit that requires no additional facility modifications to achieve significant emission reductions.

Another consideration is the LEVD's production rate. The above calculation used a production rate of 560 lb/hr of steel parts (workload) because most vendors of conventional degreasers quote capacities based on steel parts. However, production capacity per machine can vary depending on the metal processed. Based on the thermal diffusivity of various metals, total cycle times versus production rates are plotted in Figure 5. Brass and copper can be processed faster than steel with the LEVD, and aluminum can be processed faster up to a point determined, for a certain shape of parts, by the maximum mass of aluminum parts that fit into the basket.

The shape of the parts also may affect cycle time. Parts with recesses that can trap solvent should be arranged in the basket so that the solvent liquid drains out. Other features offered by the vendor (oscillating or rotating baskets) should be used. Otherwise, either the air recirculation stage time must be increased, or the unit will loop into several adsorption cycles until the chamber concentration falls below 1 g/m³.

Conclusions and Discussion

All three technologies evaluated in this study demonstrated good potential for pollution prevention/waste reduction. The two onsite solvent distillation technologies reduced large volumes of hazardous solvent to a few gallons of distillation residue and produced a reusable recycled product. The total U.S. solvent demand is approximately 160 billion gal/yr. Therefore, there is considerable potential for recycling and reusing spent solvent. Between onsite and offsite recovery, onsite recovery is preferable because of the reduced transportation hazard.

The largest single use for solvents in the United States is for vapor degreasing. The LEVD reduced air emissions significantly compared to emissions from a conventional vapor degreaser.

Payback periods for both distillation technologies are less than 2 yr. The LEVD is a slightly higher capital investment (with

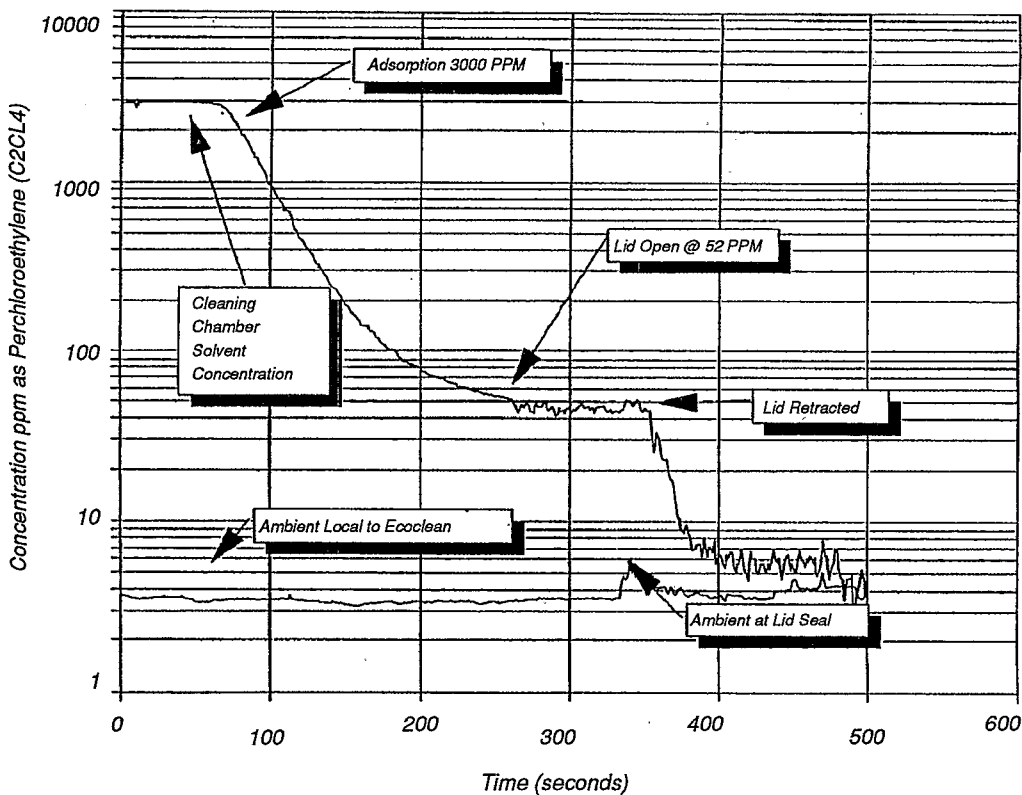


Figure 4. Concentrations at the end of the cleaning cycle for Run 1.

Table 7. Operating Costs for Low-Emission Vapor Degreasing-

Item	Annual Volume	Unit Cost (\$)	Total Cost (\$)
Conventional Degreaser			
Operating labor	4,000 hr	8/hr	32,000
Electricity	25,500 kWh	0.04/kWh	1,020
Cooling water	480,000 gal	1/1000 gal	480
Maintenance			
- labor	22 hr	8/hr	176
- materials			\$ 88
Net solvent loss	2,642 lb	0.71/lb	1,876
		Total	35,640
LEVD			
Operating labor	333 hr	8/hr	2,664
Electricity	93,725 kWh	0.04/kWh	3,749
Maintenance			
- labor	262.5 hr	8/hr	2,100
- materials			2,100
		Total	10,613

a payback period of approximately 10 yr), but it eliminates the need for other potentially expensive auxiliary equipment that conventional vapor degreasers would require to meet comparable pollution prevention objectives.

The full report was submitted in partial fulfillment of Contract Number 68-CO-0003, Work Assignment 2-36, by Battelle under the sponsorship of the U.S. Environmental Protection Agency.

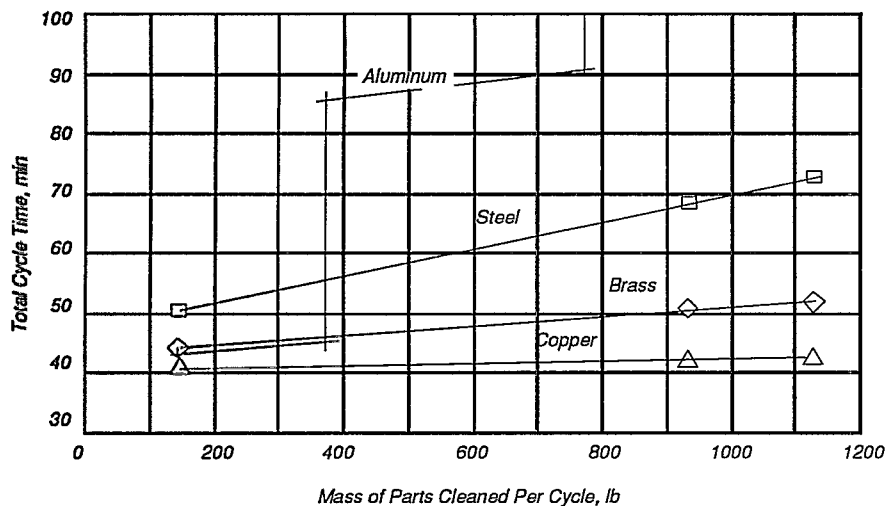


Figure 5. Variation of LEVD cycle time for various metals.

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The complete report, entitled "Onsite Solvent Recovery," (Order No. PB94-144508; Cost: \$19.50, subject to change) will be available only from

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The EPA Project Officer can be contacted at

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