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**THE MASSACHUSETTS  
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**SUPERCRITICAL FLUID EXTRACTION  
CLEANER APPLICATION**

**TEXAS INSTRUMENTS INCORPORATED**

Technical Report No. 21

1994

# **Supercritical Fluid Extraction (SFE) Cleaner Application**

## **Texas Instruments Incorporated**

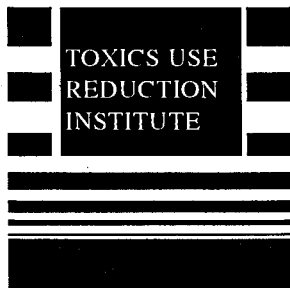
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### **The Toxics Use Reduction Institute Matching Grants Program**

December 1994

The Toxics Use Reduction Institute  
University of Massachusetts Lowell



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## **Toxics Use Reduction Institute Matching Grants Program**

The Institute annually provides direct funding to Massachusetts industries on a matching basis for toxics use reduction (TUR) feasibility and technology studies. The matching Grants Program was initiated in FY 93 to facilitate the development and use of innovative techniques that reduce the use of toxic chemicals or the generation of toxic byproducts in Massachusetts businesses. Grants are awarded on a competitive basis for companies to conduct TUR studies at their facilities. Recipients prepare project reports which assist in transferring toxics use reduction technologies and methods to other companies.

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# Supercritical Fluid Extraction (SFE) Cleaner Application

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## I. INTRODUCTION

Metal cleaning in an Industrial setting is typically an environmentally hazardous activity. Until recently, vapor degreasers utilizing halogenated solvents were prevalent through industry. Halogenated solvents have been relatively cheap, extraordinarily versatile and waste disposal costs have been perceived as insignificant. Today, it is readily apparent that Industry's reliance upon halogenated solvents as metal cleaners have resulted in a myriad of environmental, health and safety concerns, resulting in regulations that has resulted in increased costs for companies who continue using these toxic materials. Consequently, a great deal of effort has been devoted to developing alternative cleaning technologies.

Texas Instruments Incorporated, Materials & Controls Group (TI), with headquarters in Attleboro, MA, has been actively pursuing alternatives to halogenated solvent vapor degreasing since 1984. Aqueous, semi-aqueous, and "no-clean" technologies have been implemented to reduce solvent usage by more than 60%. However, several manufacturing production units have been unable to find a technically feasible and cost-effective alternative to vapor degreasing. In 1990, TI qualified the use of supercritical carbon dioxide as a solvent capable of cleaning mineral oil residue from temperature sensitive metal parts. The conceptual design of the cleaner equipment included a large cleaning chamber capable of meeting TI's cleaning requirements for an entire shift. The high capital cost of such a system prevented TI from employing supercritical fluid extraction technology in its routine cleaning operations. In 1992, TI engaged with CF TECHnologies (CF TECH) to evaluate the application of a smaller scale SFE technology to clean organic residues from metal parts assembled into motor protectors.

This paper documents the efforts of Texas Instruments Incorporated, Materials & Controls Group (TI) to qualify a Supercritical Fluid Extraction (SFE) Cleaner. The paper is divided into five sections:

- The SFE Cleaning Process Described
- SFE Technical Feasibility Evaluation
- Proposed SFE Cleaner Design and Cost
- Financial Assessment
- Conclusions

This project was undertaken as part of a Toxics Use Reduction Institute (TURI) Matching Grant Program for 1993.

## II. The SFE Cleaning Process Described

### A. The SFE Cleaning Process

Supercritical fluids today are being used in a variety of applications as diverse as the decaffeination of coffee and the treatment of hazardous wastes. For the last several years, metal-finishing and electronics industries have been looking at SFE technology as a promising substitute for halogenated solvent usage in manufacturing operations. Environmental concerns related to the use of halogenated solvents and new regulations have motivated companies to find alternative manufacturing techniques that do not rely on these substances. SFE has received a great deal of scrutiny due to the environmental acceptability of the most common SFE solvent, carbon dioxide (CO<sub>2</sub>).

Supercritical fluids have been used as solvents for extracting organic chemicals from mixtures in applications such as pharmaceuticals, petrochemicals, explosives, foods (decaffeinating coffee and tea), and polymers. Also, the technology has been successfully employed in applications such as recrystallization, deposition, impregnation, and

surface modification. The use of supercritical fluid extraction to "extract" organic contamination from metal, ceramic or composite parts are a natural outgrowth of these previous applications. (1)

Precision surface cleaning with critical fluids uses the powerful solvent properties of gases compressed to their critical point. Each substance has a unique critical point, defined by the temperature and pressure at which the liquid and vapor phases become identical. Critical fluids have a useful combination of liquid-like density and solvency, and gas-like viscosity and diffusivity.

Supercritical fluids are effective cleaning agents because of their ability to rapidly penetrate substrates and small interstitial spaces. After dissolving any contaminants, the critical fluid is easily and completely removed because it lacks surface tension.

This cleaning process involves the removal of contaminant, principally organic materials, from the surface, holes, pores and crevices of complex parts, assemblies, instruments and electronic hardware. Organic materials that are removable include solvent and flux residues, lubricants, oils and coatings. Particulate removal is possible and enhanced when the critical fluid solvent is used in conjunction with physical means such as agitation or ultrasound.

The critical fluid of choice for surface cleaning applications is most often carbon dioxide (CO<sub>2</sub>), either pure or in combination with a small amount of cosolvent. Solvent properties can be adjusted by small changes in temperature and pressure, allowing CO<sub>2</sub> to dissolve a range of organic compounds. The advantages of CO<sub>2</sub> include a near ambient critical temperature of 31 C and a moderate critical pressure of 1100 psi. See figure 1. CO<sub>2</sub> is also non-toxic, non-hazardous and non-flammable. It has no ozone-depleting potential and is easily recycled.

### B. Cleaning System Operation

Typically, critical fluid cleaning operations are a batch process. The system consists of two primary pressure vessels, a cleaning chamber and a separator. Other major equipment includes high pressure pump(s), pressure regulator(s), pressure reduction valve(s), and interconnect piping.

Systems are either manually controlled or completely automated, requiring no operator attention during the cleaning cycle. In the latter case a set of pre-programmed operating parameters, optimized for the materials being cleaned, controls the process.

The process schematic in Figure 2 illustrates the typical cleaning operation and several optional features. Materials to be cleaned are loaded into the cleaning chamber using an appropriate container or fixture. The chamber is then sealed using a closure mechanism designed for the operators and working environment.

The cleaning process begins by pumping liquid CO<sub>2</sub> from a storage vessel into the cleaning chamber and pressurizing the chamber to operating conditions. At this point the cleaning chamber is isolated from the rest of the of the system and the materials remain immersed in supercritical CO<sub>2</sub>. The cleaning process then proceeds by using any one or a combination of the following options:

- immersion only for a specific length of time
- agitation of the cleaning chamber contents
- recirculation of the supercritical CO<sub>2</sub>
- displacement of the CO<sub>2</sub> in the cleaning chamber with additional volumes of fresh CO<sub>2</sub>

At the end of the cleaning cycle, additional fresh CO<sub>2</sub> is pumped into the cleaning chamber, displacing the contaminated CO<sub>2</sub> solvent into the separator. As the CO<sub>2</sub> exists the cleaning chamber, it passes through a pressure

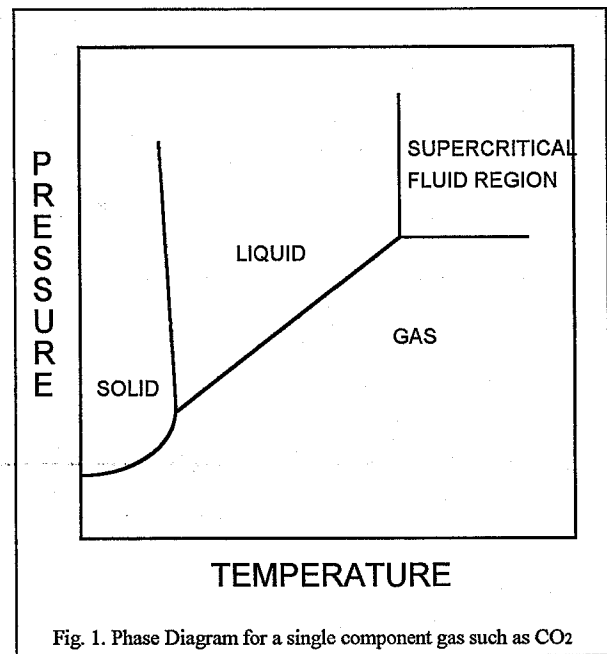


Fig. 1. Phase Diagram for a single component gas such as CO<sub>2</sub>

reduction valve and vaporizes in the separator. Non-volatile contaminants collect in the bottom of the separator and CO<sub>2</sub> vapor exits from the top. Depending upon the scale of the operation, recovery and reuse of the CO<sub>2</sub> may be economically justified.

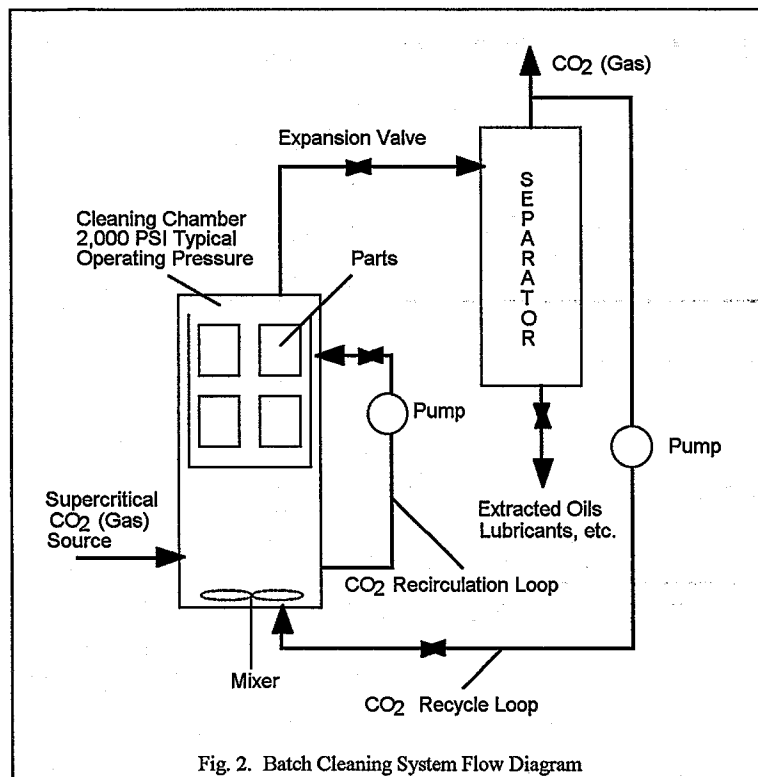


Fig. 2. Batch Cleaning System Flow Diagram

### C. SFE Cleaning System Applicability

Like any cleaning technology, SFE works better on certain classes of soils. SFE has been employed to successfully remove oils such as hydrocarbons, esters, silicones, perfluoropolyethers, halocarbon substituted triazines, etc. They are commonly used in industrial lubricants and are the types of soils removed by vapor degreasing and aqueous cleaning. Inorganic materials, such as metal salts, oxides, dust and lint are not normally removed by SFE. The study described in the Section III evaluates the applicability of SFE as a method of removing a mineral oil and hydrocarbon-based stamping lubricant from product manufacturing in TI's Disc Manufacturing production unit.

### III. SFE EVALUATION PROGRAM RESULTS

#### A. Production Unit 001, Disc

#### Manufacturing

The Disc Manufacturing production unit, designated Production Unit 001 on TI's Toxics Use Reduction Form R, manufactures monometal and bimetal discs (i.e., round 'diaphragms') that change curvature in response to pressure, temperature or current. These discs are the working mechanism of a wide range of precision control switches manufactured by TI in Production Unit 036, Precision Control Devices. The disc forming process is proprietary and the precise nature of the operations is maintained as "TI Strictly Private." The discs are soiled twice during the production process: once during the stamping of the disc from strips of thermostat material (from Production Unit 005, Thermostat Material Strip) and during the calibration operation. Currently, a trichloroethylene vapor degreaser, as described in Table I, is used to remove these soils.

Table 1: Disc Department Vapor Degreaser

Description	Trichloroethylene/Byproduct Generation (lbs)	BRI/Base-Year to Planning Year
Baron-Blakeslee, Model Number	Base Year (1990)	63,191
THLLV, Cross-rod Vapor Degreaser	Planning Year (1993)	39
	Planning Year (1998)	40

#### B. Test Program

The tests were performed in a one liter vessel outfitted with a basket that could be set to rotate at various speeds. The CO<sub>2</sub> was recirculated once the vessel was charged to supercritical conditions (condition 3). Three experiments were conducted as follows:

Table 2: Experiment Conditions

Experiment	Supercritical State	Rotation
I	180oF, 2,000 PSI	Static (no rotation)
II	180oF, 2,000 PSI	High to Moderate rotation
III	180oF, 2,000 PSI, High purity CO2/Filtered	Low rotation

Organic residue evaluation consisted of a surface carbon level analysis performed by TI's Technical Surface Laboratory. Years of experience with the vapor degreasing of bimetal discs had allowed TI to establish an acceptable level of organic residue. Surface carbon level test results are in micrograms. To compare the test to other surface carbon level analyses, a factor is developed relating the surface area of one tested population with another.

### C. Test Program Results

TI provided sample parts to CF TECH to clean in their laboratory. Tests were conducted at the CF TECH facility in Hyde Park, MA and were transported to TI for analyses in TI's Technical Services Laboratory. The following table summarizes the results of the testing program.

Table 3: Surface Carbon Levels (micrograms)

Test No.	Experiment I	Experiment II	Experiment III
1	23.1	19.7	12.6
2	22.8	23.7	17.7
3	33.2	18.2	13.8
4	33.7	31.9	13.1
5	54.7	22.0	13.0
<b>Sigma</b>	12.9	5.3	2.1
<b>Mean</b>	33.5	23.1	14.0

Testing significance was analyzed using an "F" test (degrees of freedom = 4, critical value = 16.0, confidence = 99%) that shows a significant difference exists between experiments 3 and 1. Although these two experiments were testing two variables, it is likely that the use of high purity carbon dioxide and Millipore filtering contributed to the variance. There was little significant difference between experiments 1 and 2 or between experiments 2 and 3, although a testing population of 50 pieces may not have been sufficient to test for the effects of agitation on cleaning.

### D: Comparisons with Appropriate Cleaning Technologies

Overall, the test results demonstrated that SFE Cleaning was comparable with other cleaning technologies. The following table compares the SFE Cleaning experiment runs with similar tests performed using other cleaning technologies such as Conventional Vapor Degreasers (New -- 1990 unit, Old -- Modified 1970 unit) and Aqueous Washing.

Table 4: Comparisons with Appropriate Cleaning Technologies: Surface Carbon Levels (micrograms)

	Old Vapor Degreaser	New Vapor Degreaser	Aqueous Washing	Exp. I	SFE Exp. II	Exp. III
<b>Average SCL</b>	24.0*	10.0*	150.0*	33.5	23.1	14.0

\* Cleaning Study, February 21, 1989, Moveable Contact/Disc of 5HM Motor Protector extrapolated to match existing testing.

Although further testing will be necessary to fully qualify SFE Cleaning, it was determined that the technical feasibility study had sufficiently demonstrated the applicability of the technology and the following conceptual piece of cleaning equipment was designed.

*E: SFE Cleaning System Design*

CF TECH has been investigating the use of SFE cleaning as a method of cleaning precision parts, defluxing circuit boards, and removing conformal coatings. In addition to the project to determine the applicability of SFE to clean hi-reliability thermostats, CF TECH has been working with TI to produce an economically feasible SFE cleaning system for the large-scale degreasing of bimetal discs. From the work in the hi-reliability thermostats area, CF TECH had indicated that an SFE cleaning system with multiple vessels attached to a central charging system would result in a system that was more likely to both meet throughput requirements and be financially justified. Figure 3 provides a schematic drawing of this multi-vessel SFE Cleaning System.

Disc Manufacturing production unit throughput requirements are described in the following table:

Table 5: Disc Manufacturing Production Unit Production Data

Lots/Day*	Average Lot Weight (lbs)	Maximum Lot Weight (lbs)	Lots/Hr
300	6	15	19

\* 16 hours per day, 6 days per week for a total of 4,992 hours per year of operation.

A single vessel SFE Cleaning System can be optimized to clean three maximum weight lots every 30 minutes. Consequently, a three vessel system is capable of cleaning 18 maximum weight lots every hour. Since the average weight of a lot is less than 15 lbs, a three vessel SFE Cleaning System is adequate to meet the anticipated 19 lots per hour from the Disc Manufacturing operation with sufficient capacity in reserve for potential future growth.

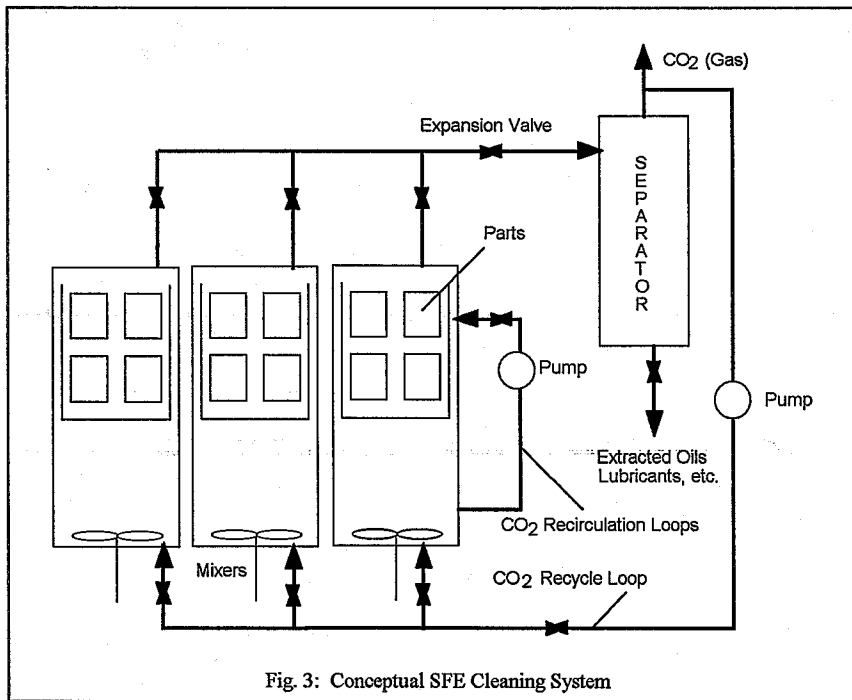


Fig. 3: Conceptual SFE Cleaning System

While there is significant uncertainty regarding the cost of the cleaning system described above, the current level of information provides sufficient information to produce a rough estimate for use in the financial assessment. TI approached CF TECH, a competitor of CF TECH and a pressure vessel manufacturer to provide cost estimate details. In addition to the cost of constructing the cleaning equipment, costs related to material handling, engineering, and installation was included. It was estimated that the capital expenditure for the SFE Cleaning System would total \$350,000.00.

IV FINANCIAL ASSESSMENT

The SFE Cleaner Project was determined to be a Capital Expenditure. TI has an established program under which project managers request funding. TI's Capital Budgeting process consists of:

- Generating the Idea/need by the Project Manager
- Performing the Financial Assessment
- Obtaining Management Approval
- Executing the Project
- Closing the Project



In order for the Capital Budgeting process to result in consistent decisions, Financial Assessments must consider all cash flows throughout the project's lifetime, the time value of money, account for the cost of obtaining capital, and factor risk. The financial assessment consisted of the following six steps:

- Gathering Relevant Cost Information Concerning the Existing Production Unit
- Estimating Relevant Cost Information for the Proposed Project
- Listing Differential Costs Between Existing and Proposed
- Generating the Cash Flow
- Evaluating Intangible Factors
- Interpreting the Results

The first two steps were done in detail and have not been provided as part of this paper. TI applies a cost accounting system that attempts to comprehend total costs and allocate those costs within the operating area that causes them to be generated. Costs that are often considered "overhead" are allocated back to the cost center either directly or via an allocation formula based upon an activity measurement. Although this accounting methodology falls short of activity based costing, it provides sufficiently detailed information to assess a project such as this. The financial assessment is performed by the Project Manager of the manufacturing area directly responsible for generating the concept and savings/expenses are directly accrued by that manufacturing area. Consequently, incentives to comprehend full environmental costs and the savings to be gained through pollution prevention exist within those Tiers responsible for developing pollution prevention options.

To perform the financial assessment, the originator of the Cash Flow must make a number of assumptions. First, operating costs (utilities, labor, etc. per unit of throughput) and some indirect costs (employee training, insurance, repair & maintenance, etc.) were assumed to be equivalent between existing and proposed Production Units. In addition, liability costs caused by potential penalties, fines, personal injury, corrective actions related to the existing and proposed Production Units were not assessed as part of this study.

Tables 6 and 7 describe the differential savings and expenses associated with implementing the SFE Cleaning System. The Annual Savings (Trichloroethylene Purchase & Disposal, Regulatory Fees, and Miscellaneous Costs) are compiled in the SAVINGS line of the SFE Cleaner Cash Flow. The Clean Air Act/MACT for Degreasing Cost is located in the OTHER SAVINGS line. Under Title III of the Clean Air Act Amendments of 1990, the U.S. EPA will be establishing emission standards that reflect the Maximum Achievable Control Technology (MACT) for halogenated solvent degreasing. The cost described in this line reflects an estimate for upgrading the existing equipment to meet the emission standard. The TI Facilities Organization has provided a quotation for wreck-out and disposal of the existing degreasing equipment. Since the Disc Manufacturing Production Unit will be shut-down to accommodate installation, the START-UP EXPENSES includes the cost for lost production. The CAPITAL EXPENDITURE has been split into two payments, 30% of the \$350K upon placement of the Purchase Order with the remainder upon delivery of the equipment. It is estimated that the design-and-build schedule will result in delivery of the SFE Cleaner within 40 weeks of placing the order.

Table 6: Description of Savings

Saving (\$K)	Description
19	Annual Purchase of Trichloroethylene (Note: Increase at 5%/yr.)
4	Annual Disposal of Trichloroethylene/oil wastes (Note: Increase at 8%/yr.)
1	Annual Toxics Use Reduction Fee/Program Administration (Note: Flat each year)
2	Annual Clean Air Act Fee/Program Administration (Note: Flat each year)
2	Annual Miscellaneous Environmental, Safety & Health Cost (Note: Increase at 5%/yr.)
100	Clean Air Act/MACT for Degreasing Cost (Note: One time charge in 1998)

Table 7: Description of Expenses

Expenses (\$K)	Description
-20	Start-up Expense/Wreck-out and Disposal of Existing Degreaser (Note: One time charge in 1996)
-115	First Year Capital Expenditure (30% of \$350K Capital Costs upon placing order)
-235	Second Year Capital Expenditure (Remainder of \$350K Capital Costs upon delivery)

To assess the financial impact of a pollution prevention project such as the proposed SFE Cleaner, listing the costs and savings is not sufficient. The assessment needs to include, not only the current costs, but future conditions. The capital budgeting process utilized by industry to justify investment decisions incorporates the impact of the capital project to the firm's cash flow over time. As indicated under Description in Table 6, the savings change over time as factors change the value of the savings. The estimation of costs and savings over a ten year horizon is complicated and uncertain. Without explicit data to base future costs and savings, TI extrapolates these values based upon the trends of the past 5 years.

The SFE Cleaner is maintained as a 5-year asset in accordance with TI accounting procedures. The depreciation is applied to the Cash Flow as a 150% Double Declining Balance. The depreciation is comprehended on the DEPRECIATION line of the Cash Flow.

The SFE Cleaner Cash Flow has been attached. The economic lifetime of the equipment for this example is 10 years. TI usually evaluates equipment over 5 years even though the expected physical lifetime of the equipment may be longer. The CORP EXPENSE line adjusts the Cash Flow to account for the percentage of profit that goes to maintaining the corporation. The AFTER-TAX SAVINGS line reflects the expected rate at which TI is taxed both at a federal and state level. The Discount Factor selected for this example is 11.5. The corporation sets this value based upon the cost to TI of obtaining capital.

When making pollution prevention decisions, long-term financial indications such as Net Present Value (NPV) and Internal Rate of Return (IRR) are calculated. The Profitability Index (PI) is calculated to allow easy comparison to other projects. The more commonly used Payback Years is also included. Table 8 provides a summary of these results from the SFE Cleaner Cash Flow.

Table 8: Financial Indicators

Financial Indicator	Value from SFE Cleaner Cash Flow (2/1/95)
Net Present Value (NPV)	-94
Internal Rate of Return (IRR)	1.4%
Profitability Index (PI)	-0.27
Payback Years	10

The Net Present Value (NPV) represents the difference between the present value of the cash inflows and the initial investment amount. The NPV provides an objective, undistorted view of the future returns of the project. In general, the rule is that the NPV must be greater than 0 for the project to be accepted. The SFE Cleaner Cash Flow NPV of -94,000 dollars is sufficient to reject the project.

The Internal Rate of Return (IRR) calculates the rate of return that equates the present value of the cash flows with the amount of the investment. The SFE Cleaner IRR of 1.4% is less than TI's desired rate of return or "hurdle rate" and is sufficient to reject the project.

Included is the Profitability Index (PI), an index that can be used to compare the SFE Cleaner proposal with other potential pollution prevention projects. A PI of -0.27 does not stack up well against other options that are being assessed within the Motor Controls Disc Manufacturing production unit.

The Payback Years is an undiscounted (i.e., it does not account for the time value of money) value that describes the number of years the initial investment will be tied up in the project. As such, it is only a quick appraisal of the merits of the project. The 10 year payback is longer than TI usually accepts and may be longer than the economic lifetime of the asset. The conclusion of the quantitative portion of the financial assessment is that the SFE Cleaner project does not display a positive effect on TI's profitability.

Since the above quantitative financial assessment does not include the risks and/or intangible factors, non-monetary concerns need to be addressed. The following intangibles were factored into the assessment:

- The liability associated with the continued use of trichloroethylene in the existing production unit is considered high. In 1984, TI management established as an objective the phase-out of the use of

trichloroethylene; consequently, they favor projects that result in the elimination of trichloroethylene. IMPACT -- High \$ Costs.

- SFE Cleaning of metal parts has not been fully demonstrated. Although this study included a preliminary technical feasibility evaluation, the technology has not undergone the level of evaluation necessary to be qualified as a substitute. The cost of conducting the qualification tests is likely to be moderately costly. IMPACT -- Moderate \$ Costs.
- Highly pressurized equipment is not a "familiar" technology. Unlike vacuum equipment, which has many years of proven use within the metal finishing industry, there is not equivalent experience within TI in the use of the highly pressurized equipment. Employees expressed concern that the maintenance, operation, etc. of the cleaning vessels of the SFE Cleaner may present a problem. IMPACT -- Low \$ Costs.
- In addition to the SFE Cleaner, TI is evaluating other pollution prevention options including "eliminating the need to clean." Inherently, this strategy promises a larger payoff for TI; consequently, more resources are being dedicated to this effort. IMPACT -- High \$ Savings.

In summary, the intangible factors continue to justify TI's efforts to phase out the use of trichloroethylene, but not through input substitution utilizing current SFE cleaner technology.

## V CONCLUSIONS

The purpose of this study was to determine if an SFE cleaning system was a viable substitute to the trichloroethylene vapor degreaser in the Disc manufacturing area, Production Unit 001. Results of the technical feasibility study demonstrated that the technology was workable, but was insufficient to approve the cleaner as a qualified substitute that would be acceptable to TI's customer base. A positive result on a customer approved qualification investigation is necessary before an SFE cleaning system could be approved to replace the trichloroethylene degreaser in the Motor Controls Disc Mfg. area. The study did provide enough information to design a conceptual SFE cleaner. Using this design, a Capital Expenditure request, including a financial assessment, was developed. The measures of profitability (NPV, IRR and Payback) all lead to a determination that SFE cleaning is not, at present, an economically justified technology within the Disc manufacturing area. The conclusion of the paper is that SFE Cleaning technology is an elegant cleaning method with many environmental benefits, but one that is not presently cost effective. The findings of this project have resulted in TI no longer pursuing SFE Cleaning as a Toxics Use Reduction option.

## ACKNOWLEDGMENTS

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**SFE Cleaner Cash Flow**

Factors	Rest of 1995	1996	1997	1998	1999	2000	2001	2002	2003	2004	2005	TOTAL
Capital Expend	115	235	0	0	0	0	0	0	0	0	0	350
Savings	0	28	29	31	32	34	36	37	39	41	44	352
Other Savings	0	0	0	100	0	0	0	0	0	0	0	100
Depreciation	0	0	-105	-74	-57	-57	-57	0	0	0	0	-350
Start-up Costs	0	-20	0	0	0	0	0	0	0	0	0	-20
\$ Savings	0	8	-76	57	-25	-23	-21	37	39	41	44	-18
Corp. Expense	0	0	0	10	0	0	0	9	10	10	11	51
Total Savings	0	8	-76	47	-25	-23	-21	28	30	31	33	-69
After-Tax Savings	0	5	-48	30	-16	-15	-14	18	19	20	21	20
+ Depreciation	0	0	105	74	57	57	57	0	0	0	0	350
Cash Flow	-115	-230	57	104	41	42	43	18	19	20	21	20
Discount Factor	0.944	0.842	0.751	0.67	0.597	0.533	0.475	0.424	0.378	0.337	0.318	
Present Value	-109	-194	43	69	25	23	21	8	7	7	7	-94
Cumm. Cash Flow	-115	-345	-288	-185	-143	-101	-58	-40	-21	-1	20	20

Net Present Value (NPV)	-94
Internal Rate of Return (IRR)	1.4%
Profitable Index (PI)	-0.27
Payback Years	10