

**Safer Alternatives for the Chemical, Pharmaceutical and Biotechnology Industries: Screening Tests of Alternatives**

**Prepared by:**

Katy Wolf

Institute for Research and Technical Assistance

**Prepared For:**

Cal/EPA's Department of Toxic Substances Control and  
U.S. Environmental Protection Agency Region IX

September 2011

## **DISCLAIMER**

This report was prepared as a result of work sponsored and paid for by the California Environmental Protection Agency's (Cal/EPA's) Department of Toxic Substances Control (DTSC) and the United States Environmental Protection Agency (U.S. EPA). The opinions, findings, conclusions and recommendations are those of the author and do not necessarily represent the views of the sponsors. Mention of trade names, products or services does not convey and should not be interpreted as conveying Cal/EPA, DTSC or U.S. EPA approval, endorsement or recommendation. DTSC, U.S. EPA, their officers, employees, contractors and subcontractors make no warranty, expressed or implied, and assume no legal liability for the information in this report. The sponsors have not approved or disapproved this report nor have the sponsors passed upon the accuracy or adequacy of the information contained herein.

## **ACKNOWLEDGMENTS**

The analysis in this report benefited considerably from the efforts of many persons within and outside the Institute for Research and Technical Assistance (IRTA). I would particularly like to acknowledge the valuable contributions made by Diana Phelps from DTSC and John Katz from U.S. EPA. I am especially grateful to Martha Murray, Jill Dapremont, Chris Holmes and Renato Murrer from AMPAC Fine Chemicals for all of their work on helping to structure the project and testing the alternatives. Finally, I would like to give special thanks to Holly Brown for her assistance in preparing the document.

## EXECUTIVE SUMMARY

It is estimated that there are more than 1,200 chemical manufacturing facilities in California. Various types of solvents are used extensively to clean reactor tanks and associated equipment employed by pharmaceutical and chemical manufacturers. Companies with batch and campaign operations particularly rely on solvent cleaning operations when they are changing products that require different input chemicals. Many of the solvents that are used for cleaning are classified as Volatile Organic Compounds (VOCs) which contribute to photochemical smog. Some of the solvents may pose toxicity problems. Use of the solvents can result in air emissions and the generation of large amounts of hazardous waste.

This project focused on finding and evaluating safer alternatives for cleaning reactor tanks and other related equipment. The project was sponsored by U.S. EPA Region IX and Cal/EPA's Department of Toxic Substances Control (DTSC). The project was conducted by the Institute for Research and Technical Assistance (IRTA), a small nonprofit technical environmental organization with specific expertise in solvent alternatives. IRTA partnered with a pharmaceutical company in northern California, AMPAC Fine Chemicals, to perform screening tests of a range of different alternatives including water-based cleaners, low-VOC, low toxicity solvents and a few other cleaning agents. The results of the screening tests can be used by chemical, pharmaceutical and biotechnology companies for guidance on which alternatives may be promising candidates for scaled-up testing.

For the screening tests, IRTA and AMPAC selected low-VOC, low toxicity alternatives for testing, developed a testing protocol and conducted the tests over a two month period. The protocol involved testing six alternatives at various concentrations at both room and elevated temperatures. Beakers were used for the testing and the cleaners were agitated with stir bars to mimic the agitated immersion cleaning method employed by AMPAC. The cleaning capability of the alternatives was tested on coupons contaminated with three soils including one of AMPAC's pharmaceutical intermediates and oil and grease and carbon encountered in maintenance activities.

The results of the screening tests indicated that two of the water-based cleaners, called CIP 100/ProKlenz and Metalnox M6321, performed well. Both are alkaline cleaners. Other alternatives that performed well for some of the soil types included a soy based cleaner called Soy Gold 2500, propylene carbonate and another neutral water-based cleaner called Metalnox M6441. Table E-1 summarizes the qualitative results of the testing.

<b>Table E-1</b>		
<b>Best Performing Alternatives in Screening Tests</b>		
	<b>Best Performers</b>	<b>Next Best Performers</b>
AMPAC Intermediate	CIP 100/ProKlenz	Metalnox M6321
	Soy Gold 2500	Metalnox M6441
		Propylene Carbonate
Oil and Grease	Metalnox M6321— 10%	Soy Gold 2500
Carbon Soil	CIP 100/ProKlenz	Metalnox M6321

IRTA and AMPAC had originally planned to conduct scaled-up testing of the alternatives that performed well in the screening tests. Because of resource constraints, however, AMPAC was not able to devote the staff time that would have been required to this effort. Based on the results of the testing however, AMPAC did adopt one of the screening test candidates, CIP 100/ProKlenz, in some of their cleaning operations. This indicates that at least one of the alternatives has promise for being used by other companies in operations where they are currently using solvents for cleaning.

## TABLE OF CONTENTS

Disclaimer.....	i
Acknowledgements.....	ii
Executive Summary.....	iii
Table of Contents.....	v
List of Tables.....	vi
List of Figures.....	vii
I. Introduction and Background.....	1
II. Preliminary Screening Tests for Reactor Tank Cleaning Alternatives.....	3
Analyzing/Selecting Alternative Cleaning Agents.....	3
Water-Based Cleaning Alternatives.....	3
Solvent Cleaning Alternatives.....	4
Other Cleaning Alternatives.....	5
Screening Test Protocol.....	5
Results of Screening Tests.....	7
AMPAC Intermediate .....	7
Oil and Grease.....	8
Carbon.....	10
Summary of Overall Results.....	10
Scaled-Up Testing.....	10
III. Results and Conclusions.....	12

## LIST OF TABLES

Table E-1. Best Performing Alternatives in Screening Tests.....	iii
Table 2-1. Alternative Cleaning Agents Included in Screening Tests.....	5
Table 2-2. Results of Intermediate Cleaning Tests at 70 Degrees C (158 Degrees F).....	9
Table 2-3. Results of Oil and Grease Cleaning Tests at 70 Degrees C (158 Degrees F).....	9
Table 2-4. Results of Carbon Cleaning Tests at 70 Degrees C (158 Degrees F).....	10
Table 3-1. Results of Screening Tests.....	12

## LIST OF FIGURES

Figure 2.1. Test Coupons with Three Soils.....	6
Figure 2-2. Beaker Setup For Coupon Tests.....	7
Figure 2-3. Soil Delaminating from Coupon.....	7
Figure 2-4. Soil Floating to Top of Beaker.....	8



## I. INTRODUCTION AND BACKGROUND

Californians are strongly concerned about the quality of their environment and are vitally interested in minimizing the releases and generation of toxic and other hazardous materials. In response to continuing concerns about pollution, in 1998, the Legislature augmented the State's Pollution Prevention (P2) Program at California's Environmental Protection Agency Department of Toxic Substances Control (DTSC) through legislation called Senate Bill 1916. The program involves selecting certain industries every few years for detailed focus to address P2 priorities and promote implementation of source reduction measures. For the fiscal year 07/08 cycle of SB 1916, DTSC selected the Chemical Industry. The Chemical Industry project is a voluntary program that addresses an industry primarily composed of large businesses but many members of the industry are also small and medium sized businesses. DTSC has established a partnership with the Chemical Industry Council of California (CICC) as part of the project which is designed to reduce hazardous waste generation and other multimedia releases.

According to the American Chemistry Council, the California chemical industry produces about \$27 billion worth of goods and contributes nearly \$17 billion to the gross state product. The most recent data on the industry are from 2004 which was a peak manufacturing year. In that year, there were 1,206 chemical manufacturing facilities in the State with 90,970 employees. This industry includes a wide variety of manufacturing operations used to produce inorganic chemicals, organic chemicals, plastics and synthetic resins, drugs, soap, detergent and cleaning preparations, paints, varnishes, lacquers, enamels and agricultural chemicals. The chemical industry ranks first in hazardous waste generation and fifth in total hazardous releases in the federal Toxics Release Inventory (TRI) among all California industries. The highest concentrations of TRI releases are in southern California, especially in Los Angeles, Orange and Riverside counties, and around the Bay Area and the northern Central Valley. The industry's air emissions account for 95 percent of the industry's total on-site disposal and other releases and 75 percent of the industry's total on- and off-site releases.

According to DTSC manifest data, the chemical industry generated about 70,000 tons of hazardous wastes in 2005, the most recent year data for this industry are available. Major waste generating processes in the chemical industry include cleaning activities like washing out reactor vessels and other production equipment, bottles, glassware, containers and tanks and flushing lines, valves and hoses. Waste streams classified as oxygenated, hydrocarbon and unspecified solvents account for 17 percent of major waste stream generation and inorganic aqueous solutions also account for 17 percent. The spent solvents and aqueous streams include materials used in cleaning activities. The California Air Resources Board (CARB) reports that the chemical industry was responsible for 4.6 million pounds of toxic air emissions in 2002. Many of the chemicals that comprise these toxic air emissions are solvents of various types and it is likely they are used for cleaning activities. Most of the solvents used for cleaning are classified as Volatile Organic Compounds (VOCs) which contribute to photochemical smog and many are considered toxic as well.

The focus of this project was to identify, test, develop and evaluate alternative safer methods of cleaning reactor tanks and related equipment for the chemical, pharmaceutical and biotechnology industries. The aim of the project was to reduce or eliminate the use of toxic and VOC cleaning materials and minimize the generation of hazardous waste and air emissions. Use of safer cleaning alternatives protects workers and community members in California. The Institute for Research and

Technical Assistance (IRTA) is a nonprofit organization established in 1989. IRTA's mission is to identify, develop, test and demonstrate safer alternatives in a variety of applications. A significant focus of IRTA's work has been on solvent alternatives. IRTA partnered with DTSC's Pollution Prevention group to conduct the project which was sponsored under EPA's Pollution Prevention Grants Program.

In the original project formulation, IRTA and DTSC wanted to recruit three companies in the chemical industry to work on the project to test and evaluate alternatives. The best candidates for the project are companies that have batch operations. Because they are producing or blending many different products in shorter runs, their cleaning needs between products are greater and they use a number of solvents and high volumes of solvents for cleaning. At the outset, IRTA recruited one company, AMPAC Fine Chemicals, a pharmaceutical company with many batch operations, to work on the project. IRTA also worked with the Chemical Industry Council of California (CICC) to identify and recruit additional companies. Several companies were interested but could not commit staff to work on the project because of the poor economy at the time. As a consequence, the project was scaled down and AMPAC was the single project participant.

AMPAC is an active pharmaceutical ingredient and fine chemical manufacturer subject to strict oversight by the U.S. Food and Drug Administration (FDA) and other international food and drug regulatory agencies. The manufacture of products within the pharmaceutical industry is controlled by the customer and regulatory agencies and is driven by the quality of the final product. When alternatives are considered, companies must ensure that there are no detrimental impacts to product quality and verify for the customers and the FDA that product quality is not affected.

When the project was initiated, IRTA and AMPAC scoped out a program designed to test alternatives for reactor tank cleaning and cleaning of related equipment like valves, pumps and hoses. The program involved developing a protocol and conducting detailed preliminary screening tests of a variety of alternatives. Section II of the report summarizes the screening tests in more detail. Due to resource constraints, AMPAC could not continue the scaled-up tests that were planned for the best performing alternatives in the screening tests. The company did, however, adopt one of the best performing alternatives in some operations. This suggests that other companies might find this alternative and perhaps some of the other alternatives that performed well in the screening tests appropriate for substitution for solvents in their cleaning operations. Finally, Section III of the document summarizes the results of the testing.

## II. PRELIMINARY SCREENING TESTS FOR REACTOR TANK CLEANING ALTERNATIVES

This section presents the approach to and results for the screening tests that were conducted to find suitable alternatives to the solvents currently used for reactor tank and related equipment cleaning. The first task involved analyzing several different potential alternative cleaning agents and determining which ones should undergo the screening tests. The second task involved developing a protocol for the screening tests. The third task was to conduct the screening tests and to analyze and compare the results to identify the best candidates for scaled-up testing. Each of these tasks is discussed in more detail in this section below.

### ANALYZING/SELECTING ALTERNATIVE CLEANING AGENTS

The approach to selecting alternative cleaning agents that would undergo the screening tests involved identifying alternatives that had low or no Volatile Organic Compound (VOC) content, were low in toxicity and had a reasonable chance of performing well in terms of cleaning capability.

The use of VOCs in California is carefully controlled. The California Air Resources Board (CARB) is responsible for regulating the VOC content of materials used in consumer products and mobile sources. Local air districts in California are responsible for regulating the VOC content of materials used by stationary sources like chemical companies. Because the project team wanted the results to be transferrable to companies throughout California, the selected cleaners that were tested had to have low or no VOC content so they could be used anywhere, taking into account the most stringent of the VOC regulations in the state and anticipating possible future regulations. Accordingly, the criterion for selection of alternative cleaning agents was that they have no more than 25 grams per liter VOC content in the “as applied” formulation.

Another major criterion for selection of alternative test candidates was that they be low in toxicity. Virtually all solvents contribute to central nervous system toxicity at very high concentrations. Many solvents have been shown to cause chronic toxicity problems. Only alternatives with a relatively low toxicity profile were selected for testing.

The third criterion for selection of alternatives was that the alternative should have a good chance of performing well in cleaning a variety of soils that might be encountered during reactor tank and associated equipment cleaning. AMPAC has experience in evaluating alternative cleaning agents for their operations and IRTA has extensive experience in testing alternative cleaning agents for use in a variety of applications over a 30 year period. Together IRTA and AMPAC selected potentially promising candidates.

### Water-Based Cleaning Alternatives

The team’s preference for test candidates was water-based cleaners rather than solvents. AMPAC already used a particular water-based cleaner in other gross cleaning operations. The company wanted to test this cleaner, a combination of a water-based cleaning agent called CIP 100 and a booster called ProKlenz, in the screening tests as well. This cleaner is referred to as CIP 100/ProKlenz. IRTA had worked with a company that formulated water-based cleaners for a variety of precision and metal cleaning applications and wanted to conduct tests with this company, Kyzen, to identify a few different additional water-based cleaners that could also be tested.

AMPAC contacted the supplier of the CIP 100/ProKlenz cleaner and arranged for the company to conduct some tests on the ability of the cleaner to clean a few of their products. The results indicated that the cleaner could clean the products at elevated temperature but only within a fairly long timeframe. AMPAC decided to test the cleaner further in the structured comparative tests that would be conducted for all of the selected cleaning agents. This cleaner is classified as an alkaline cleaner with a very high pH at full concentration. Material Safety Data Sheets (MSDSs) for the CIP 100 and the ProKlenz booster are provided in the Appendix. It's worth noting that one of the ingredients in the ProKlenz booster, according to the MSDS, is hydrogen peroxide at a 3 to 7% concentration. As discussed below, hydrogen peroxide was one of the additional materials included in the cleaning tests.

IRTA worked with Kyzen to structure some cleaning tests on a few of AMPAC's products so the best performing water-based cleaners could be selected for the comparative screening tests. AMPAC provided four of their products which might be soils on the reactors and associated components that would ultimately require cleaning. Kyzen soiled small 1018 steel matte finish panels with the four cleaning agents according to instructions for the soil mix, baking time and temperature provided in instructions from AMPAC. The company conducted cleaning tests of the soiled panels with four water-based cleaners. The cleaners were tested in a 10 percent concentration in water at a temperature of 60 degrees C (140 degrees F) in a beaker with a spin bar for gentle agitation. The panels were partially submerged and evaluated after one, two, five and 15 minutes. The two water-based cleaners that performed best in Kyzen's tests were Metalnox M6441, a fairly neutral cleaner with a pH at full concentration between 7.8 and 9.8 and Metalnox M6321, a more alkaline cleaner with a pH of more than 13 at full concentration. MSDSs for these two cleaners are provided in the Appendix.

#### Solvent Cleaning Alternatives

IRTA has conducted testing over the last several years on a vegetable based cleaner called Soy Gold 2500. The South Coast Air Quality Management District (SCAQMD) has tested the VOC content of the cleaner; it was found to be less than 25 grams per liter. The cleaner is based on soy and it also contains a high level of surfactants which make it water rinseable. IRTA tested the cleaner extensively for cleaning ink formulations for the lithographic and screen printing industries and it was very effective. IRTA has also tested it for oil and grease removal and it is effective in some of these applications as well. Although it has a very low vapor pressure and does not evaporate from surfaces after cleaning, it can be removed by rinsing the surface with water. IRTA wanted to include this cleaner in the screening tests. An MSDS for the cleaner is provided in the Appendix.

IRTA had recently begun testing another solvent, propylene carbonate. This solvent was deemed exempt from VOC regulations by EPA based on its low reactivity in the lower atmosphere. In California, chemicals must also be deemed exempt from VOC regulations by local air districts before it can be considered a non-VOC for use by sources. Local air districts generally follow the lead of EPA in exempting chemicals except in cases where toxicity or other factors like global warming and ozone depletion potential might be high. The chemical does not contribute to global warming or ozone depletion. According to a toxicologist consulted by IRTA, the chemical is likely to be low in toxicity. IRTA conducted limited testing of propylene carbonate and found it to be a very aggressive solvent capable of removing oils and paint. The team decided to test the solvent in the screening tests. An MSDS for the solvent is shown in the Appendix.

## Other Cleaning Alternatives

IRTA also wanted to include hydrogen peroxide at fairly low concentration in the screening tests. IRTA had tested the chemical, primarily for soil removal in textile cleaning operations and it performed fairly well. Because the chemical is inorganic, it is not classified as a VOC. It is also fairly low in toxicity and is sold, at low concentration, in drugstores for consumer use. The team decided to include the chemical in the screening tests. An MSDS for the chemical is shown in the Appendix.

The team also decided to include plain deionized water in the screening tests for reference.

Table 2-1 summarizes the cleaning agent, the test concentrations and the pH of the test concentrations that were tested in the screening tests. Soy Gold 2500, propylene carbonate and deionized water were all tested at full concentration. The CIP 100/ProKlenz was tested at a 2% concentration. The other two water-based cleaners, Metalnox M6441 the neutral cleaner and Metalnox M6321 an alkaline cleaner, were tested at three concentrations, 3%, 5% and 10%. The lower the concentration that is effective for the water-based cleaners, the less costly it would be to use it. Hydrogen peroxide was tested at three concentrations, 1%, 3% and 5%.

<b>Table 2-1 Alternative Cleaning Agents Included in Screening Tests</b>		
<b>Cleaning Agent</b>	<b>Test Concentration</b>	<b>pH</b>
CIP 100/ProKlenz	2%	12.54
Metalnox M6441	3%	9.19
	5%	9.1
	10%	8.94
Metalnox M6321	3%	12.86
	5%	12.98
	10%	13.22
Soy Gold 2500	100%	5
Propylene Carbonate	100%	11.16
Hydrogen Peroxide	1%	5.17
	3%	5.12
	5%	7.01
Deionized Water	100%	

## SCREENING TEST PROTOCOL

AMPAC and IRTA developed a protocol for the comparative screening tests. In general terms, the purpose of the protocol was to describe the comparative testing procedure that would be used for the cleaning agents listed in Table 2-1 in the screening tests. These tests would involve soiling coupons with three types of soil likely to be encountered at AMPAC during reactor or associated equipment cleaning. The soils were one of AMPAC's intermediates and oil and grease and carbon which are encountered in maintenance activities. The aim of the tests was to identify which cleaning agents are best for cleaning each type of soil and to find the lowest concentration level that is effective for the cleaning agents not used at full strength.

The coupons were fabricated from 316 stainless steel one-sixteenth inch thick 2B finish. These were cut into 3 inch by 6 inch coupons with a half inch hole approximately one-half inch from the top and centered across the width of the coupon. They were each stamped with an ID number for tracking. The coupons were cleaned in a glassware washer with a powdered residue free water-based cleaner, dried at 70 degrees C (158 degrees F) and placed in an oven for 45 to 60 minutes at a temperature of 100 degrees C (212 degrees F). They were then placed in a dessicator for further moisture control.

The coupons were weighed on an analytic balance before they were soiled. They were coated with the soil and then baked for seven days, placed in a dessicator and weighed again. The difference between the weight before soiling and after soiling is the weight of the soil. Examples of coupons with the AMPAC intermediate, oil and grease and carbon from left to right are shown in Figure 2-1.



**Figure 2-1. Test Coupons With Three Soils**

For the cleaning tests three coupons, one with each of the three soils, were placed in three two liter beakers containing 1.5 liters of the appropriate cleaning solution on a hot plate. The three soils were cleaned simultaneously at three cleaning stations at one of two temperatures. The two different cleaning temperatures that were tested are 25 degrees C (77 degrees F) to represent room temperature and 70 degrees C (158 degrees F) to represent a common elevated cleaning temperature. The coupons were agitated with stir bars during cleaning and the process was continued until the coupons were visually clean based on observations every 30 minutes up to two hours and, in some cases, 150 minutes. Figure 2-2 shows the beaker setup for the cleaning tests. One additional test, with CIP 100/ProKlenz with no agitation was conducted; AMPAC was particularly interested in the results of this cleaner since they were already using it for other purposes.

When they were judged to be visually clean, the coupons were removed from the beakers and inspected, run under tap water for 10 seconds, rinsed with deionized water, placed in an oven at 90 degrees C (194 degrees F) for 60 minutes and then put into a dessicator. When the coupons were removed from the dessicator, they were weighed. The standard used to determine if the coupons were clean is that they were visually clean and that the clean coupon weight is not heavier than 5 ppm (a 100 gram coupon is not heavier than 100.5 grams in final weight).



**Figure 2-2. Beaker Setup For Coupon Tests**

### RESULTS OF THE SCREENING TESTS

The tests were conducted over the period between January 26, 2010 and March 15, 2010. Tests at the higher concentrations for some of the cleaning agents were conducted first. If the soils were not removed at the higher concentration, then the tests at lower concentration were not conducted. A total of 71 cleaning tests were performed. The results of the tests for each of the soils are discussed below.

#### AMPAC Intermediate

In general terms, the intermediate soil was the easiest of the three soils to remove. In all cases where the soil was removed, it delaminated from the surface of the coupon in a sheet, most often within five minutes and floated to the top of the solution where it dissolved. Figure 2-3 shows an example of the soil delaminating from the coupon and Figure 2-4 shows it floating to the top of the beaker.



**Figure 2-3. Soil Delaminating From Coupon**



**Figure 2-4. Soil Floating To Top Of Beaker**

At the lower temperature, only propylene carbonate and Soy Gold 2500 removed the soil and they accomplished this within 30 minutes. All other cleaning agents failed to remove the soil in a period of 150 minutes.

Table 2-2 summarizes the results of the cleaning tests at the higher temperature, 70 degrees C (158 degrees F), for all of the cleaning agents. At the higher temperature, all but three of the cleaning agents removed the soil within 30 minutes. The Metalnox M6441 at 3% concentration required 90 minutes to remove the soil. Hydrogen peroxide at 1% concentration and deionized water did not completely remove the soil within 150 minutes. The CIP 100/ProKlenz and Soy Gold 2500 were observed to remove the soils within about one minute. The Metalnox M6321 and Metalnox M 6441 at 10% concentration and propylene carbonate removed the soil within two minutes.

#### Oil and Grease

This soil was more difficult to remove than the intermediate soil. At the lower temperature, none of the cleaning agents removed the soil within 150 minutes. Table 2-3 summarizes the results for the tests conducted at the higher temperature. In this case, only two cleaning agents, Metalnox M6321 at 10 percent concentration and Soy Gold 2500, effectively removed the soil and they both required 120 minutes to accomplish the task.



<b>Table 2-2</b>			
<b>Results of AMPAC Intermediate Cleaning Tests at 70 Degrees C (158 Degrees F)</b>			
<b>Cleaning Agent</b>	<b>Concentration (%)</b>	<b>Soil Removed (%)</b>	<b>Cleaning Time (Minutes)</b>
CIP 100/ ProKlenz	2	100	30
CIP 100/ProKlenz no agitation	2	100	30
Metalnox M6441	10	100	30
	5	100	30
	3	100	90
Metalnox M6321	10	100	30
	5	100	30
	3	100	30
Soy Gold 2500	100	100	30
Propylene Carbonate	100	100	30
Hydrogen Peroxide	5	100	30
	3	100	30
	1	97.7	150
Deionized Water	100	83.7	150

<b>Table 2-3</b>			
<b>Results of Oil and Grease Cleaning Tests at 70 Degrees C (158 Degrees F)</b>			
<b>Cleaning Agent</b>	<b>Concentration (%)</b>	<b>Soil Removed (%)</b>	<b>Cleaning Time (Minutes)</b>
CIP 100/ ProKlenz	2	21.2	Not Clean
CIP 100/ProKlenz No agitation	2	3.9	Not Clean
Metalnox M6441	10	1.0	Not Clean
	5	0.0	Not Clean
	3	0.0	Not Clean
Metalnox M6321	10	100	120
	5	33.6	Not Clean
	3	4.5	Not Clean
Soy Gold 2500	100	99.97	120
Propylene Carbonate	100	18.7	Not Clean
Hydrogen Peroxide	5	0.6	Not Clean
	3	1.8	Not Clean
	1	0.5	Not Clean
Deionized Water	100	1.0	Not Clean

## Carbon

Like the oil and grease, this soil was more difficult to remove than the intermediate soil. Table 2-4 shows the results at the higher temperature for the carbon cleaning tests. The values show that the CIP 100/ProKlenz with agitation cleaned the coupon in 45 minutes. This formulation with no agitation and the Soy Gold 2500 did remove the soil in 120 minutes but only after the rinse. The 10% concentration Metalnox cleaners were able to remove the soil and the Metalnox M6321 did remove the carbon at all three concentrations.

<b>Table 2-4 Results of Carbon Cleaning Tests at 70 Degrees C (158 Degrees F)</b>			
<b>Cleaning Agent</b>	<b>Concentration (%)</b>	<b>Soil Removed (%)</b>	<b>Cleaning Time (Minutes)</b>
CIP 100/ ProKlenz	2	100	45
CIP 100/ProKlenz No agitation	2	100	120, rinse
Metalnox M6441	10	100	120
	5	87.5	Not Clean
	3	100	150
Metalnox M6321	10	100	90
	5	100	90
	3	100	150
Soy Gold 2500	100	100	120, rinse
Propylene Carbonate	100	74.2	Not Clean
Hydrogen Peroxide	5	31.3	Not Clean
	3	11.1	Not Clean
	1	14.3	Not Clean
Deionized Water	100	0.0	Not Clean

### Summary of Overall Results

The cleaners that performed best on the AMPAC intermediate soil were the CIP 100/ProKlenz and Soy Gold 2500 and they removed the soil in one minute. The next best cleaners were the two 10% Metalnox formulations and propylene carbonate; these cleaners removed the soil in two minutes. For the oil and grease soil, the 10% Metalnox M6321 cleaner performed best and the Soy Gold 2500 was the next best cleaner. For the carbon soil, the best cleaner was the CIP 100/ProKlenz formulation; the next best cleaner was the 10% and 5% Metalnox M6321 cleaner.

### SCALED-UP TESTING

The project team met in February 2011 to scope out a scaled-up testing program. The plan was to focus on valve and hose cleaning and the cleaning agents that would be tested further were selected based on the desire to move to water-based products and the results of the screening tests. The program would involve testing the CIP 100/ProKlenz, Metalnox M6321 and Soy Gold 2500 in these cleaning operations. At this stage, because of resource constraints, AMPAC was not able to continue the scaled-up testing

phase because it would have required considerable resources. The company did adopt CIP 100/ProKlenz in some of their operations as a replacement for solvents.

### III. RESULTS AND CONCLUSIONS

This project focused on finding safer alternatives to the solvents used today for cleaning reactor tanks and associated equipment like valves, hoses and driers. The results should be useful as a starting point for more extensive individual testing for companies like pharmaceutical, biotechnology and chemical manufacturers. The project involved conducting comprehensive screening tests of potential alternative cleaning agents with one company that manufactures active pharmaceutical ingredients, AMPAC Fine Chemicals.

IRTA and AMPAC developed a protocol for conducting screening tests of alternatives to solvents used in cleaning operations throughout chemical, pharmaceutical and biotechnology manufacturing plants. A variety of candidate alternatives were selected for testing based on a desire to use water-based cleaners and the stringent regulations on VOCs and toxics in California.

The tests were conducted for six potential alternatives and deionized water. A range of concentrations for some of the potential alternatives were included in the testing as appropriate. The tests were conducted at two different temperatures to assess the cleaners' capability of removing three types of contaminants, one of AMPAC's intermediates, oil and grease and a carbon soil. Over a two month period, 71 total tests were performed.

The results of the testing indicated that two of the water-based cleaners performed well. One of these cleaners, called CIP 100/ProKlenz, was already used by AMPAC for maintenance operations in the plant. The other water-based cleaner, called Metalnox M6321, also performed well on some of the soils. Both water-based cleaners are alkaline cleaners. Other alternatives that performed well for some of the soil types included Soy Gold 2500, propylene carbonate and another water-based cleaner called Metalnox M6441. Table 3-1 summarizes the qualitative results of the testing.

<b>Table 3-1 Results of Screening Tests</b>		
	<b>Best Performers</b>	<b>Next Best Performers</b>
AMPAC Intermediate	CIP 100/ProKlenz	Metalnox M6321— 10%
	Soy Gold 2500	Metalnox M6441— 10%
		Propylene Carbonate
Oil and Grease	Metalnox M6321— 10%	Soy Gold 2500
Carbon Soil	CIP 100/ProKlenz	Metalnox M6321— 10%
		Metalnox M6321— 5%

The original intention was to select the best alternatives from the screening tests and to conduct scaled-up testing on valves and hoses. Because of resource constraints, however, AMPAC was not able to devote the staff time to this extensive testing program. AMPAC did adopt one of the candidate alternatives, CIP 100/ProKlenz, in certain cleaning operations in place of solvents. This demonstrates the feasibility of converting to an alternative water-based cleaner. The results of the screening tests should be useful to other companies interested in moving away from high VOC and toxic solvents to safer alternatives for their cleaning needs.