SOLVENT USAGE AND RECYCLING POTENTIAL IN A RESEARCH AND DEVELOPMENT SETTING

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Argonne National Laboratory (ANL) is a multi-program research and development facility owned by the U.S. Department of Energy and operated by the University of Chicago. This organization, with more than 1,100 scientists, utilizes thousands of gallons of chemicals each year.

Laboratory wastes can be broadly characterized as coming from three focus areas: 1) Restoration and decommissioning associated wastes generate larger quantities of waste on a one-time basis. The wastes may be non-hazardous to highly toxic and the quantities are variable. 2) Laboratory operations generate approximately 50% of all waste disposed. Operational waste can be characterized as less hazardous, reasonably consistent in nature, generally in larger quantities. 3) The final waste stream is ongoing research and development waste. This material consists of small quantities of many different materials coming from many different waste streams. This waste stream is at the center of ANL's pollution prevention program. Although the individual generator does not produce a significant amount of waste the aggregate of each individual project contributes to the volume. As an example, methanol is produced by 47 generators ranging in size from over 100 liters to less than one.

The research areas have implemented many pollution prevention techniques. Solvent substitution has been effective in reducing hazardous cleaning wastes, scintillation cocktail wastes, and other chlorinated wastes. Micro chemistry is effective at minimizing certain chemical process wastes, developing new analytical chemistry procedures has reduced and eliminated other waste forms. New instrumentation has provided first level reductions in many waste streams.

Despite these new techniques solvent usage remains the largest research related waste stream. The present solvents are generated from instruments such as electrophoresis and high pressure liquid chromatographs (HPLC), solvent extractions, biological staining, and cleaning practices.

ANL recognizes the significant role recycling this waste stream is in Pollution Prevention Program implementation. ANL initiated a study to quantify solvent usage, characterization of the waste solvent, and match the purity requirements exploring all opportunities to substitute and recycle.

1Argonne National Laboratory Research Aid, University of Kansas.

2Argonne National Laboratory.
solvent, and match the purity requirements exploring all opportunities to substitute and recycle.

The first step in the process was to determine what solvents and which research processes could yield the best results. The study began by generating a list of potential solvents and querying the Waste Management Data base for quantities disposed of and numbers of users. This step allowed ANL to reduce the list of solvents from more than 50 to less than 25. Solvents that had low annual usage or a corresponding large number of generators with small individual usage were eliminated from further study because the likelihood of achieving successful results were not cost effective at this time.

The second phase of the study ranked individual solvents from large users to small users and an arbitrary cutoff of 5 liters per year was established. For the solvents that met the screening criteria detailed characterization data was obtained, (name of generator, generation history, contaminants and concentration). The information was compiled into a questionnaire and for each of the solvents and waste generators in question. Figure 1 depicts solvents and number of users.

The third phase of the project involved discussions with the individual solvent users to verify the data accuracy and collect missing information. The users were also quizzed about potential barriers to recycling opportunities. Could they recycle directly or by filtering or purifying their material? Under what circumstances could they use recycled materials? Could their material be recycled? Could they substitute a different less toxic solvent?

The data validation step was essential because waste requisitions may not fully characterize dilution, particularly with water, or contaminants occurring in the part per million or lower ranges.

The study indicated that an excellent opportunity existed to recycle solvents from "high end users" such as analytical chemistry where purity is a given, to middle users that can use a clean solvent for a process, and finally to a low end user that is more than likely using the material for cleaning.

The study indicated that ethanol containing dirt and oils was the most likely candidate for recycling. The majority of ethanol users could clean the material by filtration and or evaporation and the ethanol could be reused at least once. Other solvents could be recycled with similar results but at greater costs.

The final phase of the project selected solvent from one user, identified a purification technique, and conducted a bench scale test of concept. The stream selected was an ethanol stream from a biological staining process. The stream contained 12% ethanol, silver nitrate, ethyl acetate, and formalin. Removal of the silver was critical to reuse. A 100 mL sample was distilled using
laboratory glassware in a simple distillation process. The recovered product was 95% ethanol as determined by instrumental analysis. A sample of pure solvent was obtained from the user and a simple analytical test conducted (the material was mixed with sodium sulfide). The fresh solvent and the distilled solvent showed no indication of silver sulfide precipitation indicating lack of silver ion. Spent solvent failed this test. The next step was to substitute recovered solvent into the biological staining process. The researcher was actually more pleased with the recovered solvent than they had been with fresh material.

The result of the simple demonstration was outstanding. First we verified spent solvent could be purified to meet research standards, and second the researcher was pleased with the results and wanted more.

The next step in this process will be a larger demonstration using better equipment on a larger number of waste streams. Current thinking will be to use spinning band distillation apparatus in a monitored setting. Various different solvents from several waste streams will be tested.

This project is a first step at identifying pollution prevention opportunities in a research and development setting. The next steps in the program will be to optimize recycling techniques and implement recycling on a small scale in test programs.

![Figure 1. Solvents and Number of Users](Image)

*Figure 1. Solvents and Number of Users (Information from waste requisitions forms of 1994)*