FINAL REPORT FOR GRANT 85-3529

"SCINTILLATION FLUID RECYCLING PROGRAM"

SUPPORTED BY THE

POLLUTION PREVENTION PAYS CHALLENGE GRANTS PROGRAM

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INTRODUCTION

Low-level radioactive waste is the primary component of the toxic waste stream generated from biomedical research and patient care activities. Historically, this radioactive waste has been shipped to Richland, Washington for landfill disposal. Several years ago, the Nuclear Regulatory Commission "deregulated" liquid scintillation vials containing tritium and carbon 14. More recently, the State of Washington closed their landfill to these deregulated wastes.

These regulatory changes have prompted a critical review of radioactive waste management practices at Duke University Medical Center. We have determined that volume reduction in the form of crushing and compacting could save $200,000 in annual disposal costs. Since there were no appropriate facilities to house these operations, construction of a new Environmental Safety Building was commissioned. The building is scheduled for occupancy by April 1986, and will house specialized facilities for the management of chemical and radioactive wastes.

The original proposed method of volume reduction for scintillation fluid was to crush the vials and collect the fluid. The deregulated liquid waste would then be shipped off-site for incineration as a fuel supplement. The iodinated fluid would be stored on-site for decay and then incinerated off-site as a non-radioactive fuel supplement.

Further benefits would be derived if the scintillation fluid could be recycled within the institution. Redistillation is a possibility since the new facility will be equipped with two efficient spinning-band stills. Recycling could be implemented shortly after occupancy. However, the feasibility of segregating, redistilling, and distributing the scintillation fluid from 137 laboratories had not been investigated.

SCOPE OF THE PROJECT

Duke University Medical Center will undertake a feasibility study of a scintillation fluid recycling program. The program scope will be as follows:

1. Perform an audit of each laboratory which uses scintillation fluid. Identify for each laboratory the chemical composition of the scintillation cocktails used, radioactive isotope(s) employed, monthly volume generated, and ameniability of the investigator to utilize on-site prepared cocktails.

2. Analyze the collected data to identify wastestreams which can be segregated for batch distillation.

3. Review and evaluate distillation equipment suitable for redistillation of scintillation fluid.

4. Develop a working system for the collection of used fluid and the eventual distribution of the redistilled fluid.

5. Perform bench scale studies of the effectiveness of the redistillation process.

6. Evaluate the feasibility of segregating, redistilling and distributing the recovered scintillation fluid.

SUMMARY OF FINDINGS AND RESULTS

1. Laboratory Audits -

The initial phase of the study was auditing all laboratories using scintillation fluid. The audit was directed at identifying the types and quantities of scintillation fluids used, the radioactive isotopes employed, whether the fluid was commercially or laboratory prepared, and the ameniability of the investigator to utilize on-site prepared cocktails, among other variables.

Efforts to develop administrative support and heighten the investigator's understanding of the goals of the project were undertaken before the audit process was begun. Administrative directors received a memorandum that both outlined the project and requested their assistance in gaining the cooperation of their investigators during the audit process (Appendix 1). In addition, an article was published in the facility's weekly research oriented newspaper - the "Intercom". This publicity clearly contributed to the positive response to the audits because most investigators were familiar with our project before we contacted them for an audit appointment.

There were two stages to the audit process. First, all users of radioisotopes were mailed a simple questionnaire to ascertain if scintillation fluid was used in their laboratory (Appendix 2). All non-respondents were subsequently queried by telephone to assure that all laboratories were included in the database.

In the second stage, the laboratories of all positive respondents were visited in a formal audit process. All scintillation fluid users cooperated in the audit process. A total of 137 laboratories were audited during a 3 week period. One hundred and twelve (112) of these laboratories were identified as long-term users of scintillation fluid, and were included as final participants. Within these laboratories, 229 individual wastestreams were identified because more than one isotope was used in many laboratories. A copy of the audit form is presented in Appendix 3.

2. Analysis of the Audit Data -

Computerized data management was utilized for the compilation and analysis of all audit information. A data analysis matrix was developed in a spreadsheet format on Symphony Software (Lotus Corporation). The program was driven on an IBM PC-AT.

The data was analyzed by numerous variables and the significant findings are as follows:

A. Greater than 90% of the investigators expressed interest in participating in an in-house recycling program. All investigators agreed to segregate the scintillation waste based on the isotope it contained. Additionally, many agreed to consider the use of either in-house distilled solvents or prepared cocktail. Use of these products was dependent on quality and supply and several investigators expressed an interest in comparing in-house prepared products with those presently being used.

B. It was estimated that approximately 15,000 liters or 4,000 gallons of scintillation waste are generated annually. The wastestream is characterized by several large volume generators and numerous small volume producers. Because of this distribution, it is possible to collect 95% of all potentially recyclable waste through pick-ups in only 40% of the laboratories that generate waste. This distribution suggests a more simplified scheme for collecting candidate waste than had been anticipated.

C. Commercial cocktails comprise 83% of the waste volume generated or approximately 3300 gallons/year. The primary commercial cocktails include: Aguasol-2, Biofluor, Hydrofluor, Lefko-Fluor, ScintiVerse and Ultrafluor (More specific information in the composition of these cocktails will be presented in Section 5). A graphic representation of the relative volume of the weekly production of each cocktail is presented in Figure A.

The majority of laboratory prepared cocktail (approximately 700 gallons/year) were toluene based. The relative volumes of laboratory prepared cocktail is presented in Figure B. While each of the waste streams had different additives, the distillation efficiency of each waste stream was found to be comparable.

D. The distribution of isotope in the weekly generated wastestream is presented in Figure C. Tritium represents the most commonly used isotope, followed by the short half-lived phosphorous, and 14-carbon labelled materials occurred in approximately 16% of the total waste volume.

The low volume of iodine containing waste was a surprising finding that results from the experimental procedures used for counting this isotope. Most investigators process I125 labelled experimental materials for dry counting of radioactivity. Most of the radioactive waste from this processing is in the form of aqueous wastes that obviously would not be candidates for recycling.

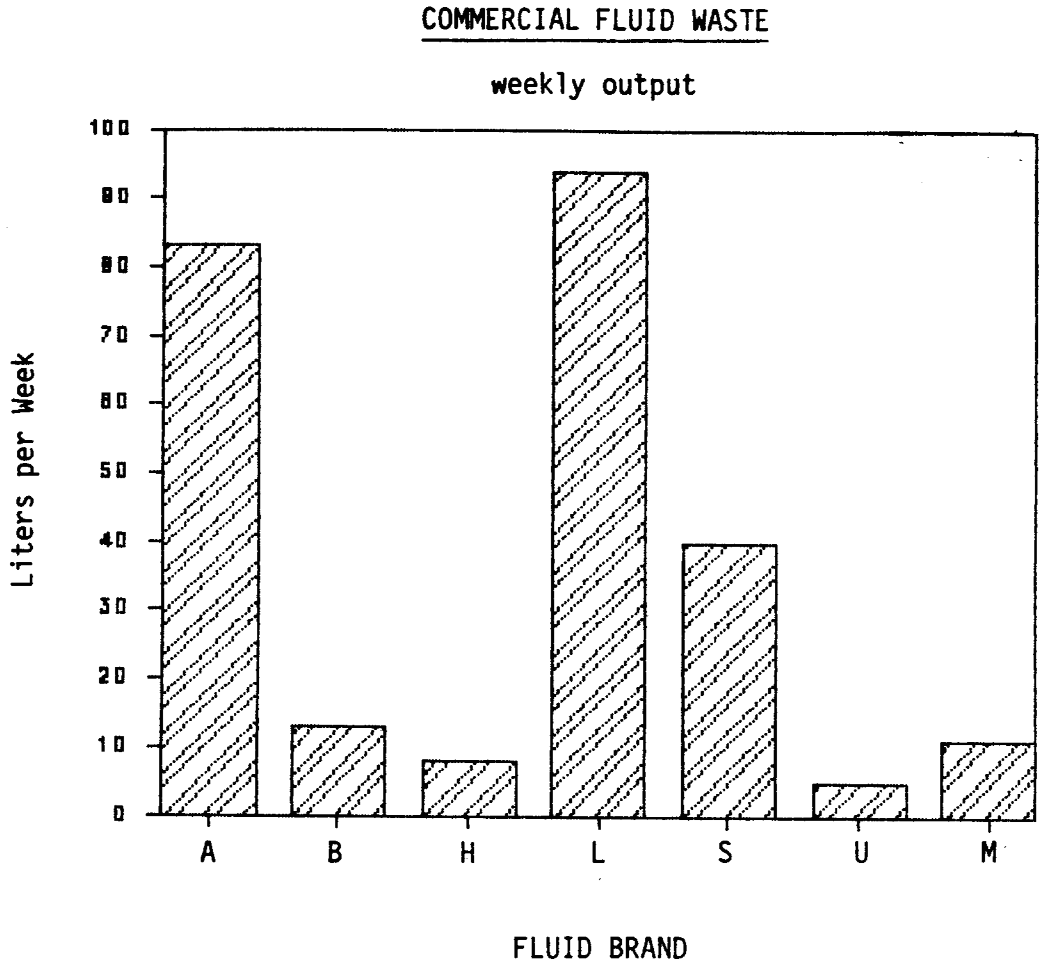
The individual wastestreams that are included in the miscellaneous category are primarily wastes that contain more than one type of isotope which were integrated into the experimental plan. While many of these wastes are recyclable, processing is more complicated and requires the application of two or more management strategies.

E. Potential variations in the wastestream were noted during the audit process. Several investigators noted that they may be changing their isotopes or scintillation cocktails as current experiments ended and new studies were initiated. This observation indicated that frequent monitoring of the generators would be necessary. It also suggests possible opportunities to convert investigators to the use of in-house prepared cocktail - this could most easily be accomplished during the development stage of a new study - thus strengthening the institutional recycling program.

3. Review and Evaluation of Distillation Equipment -

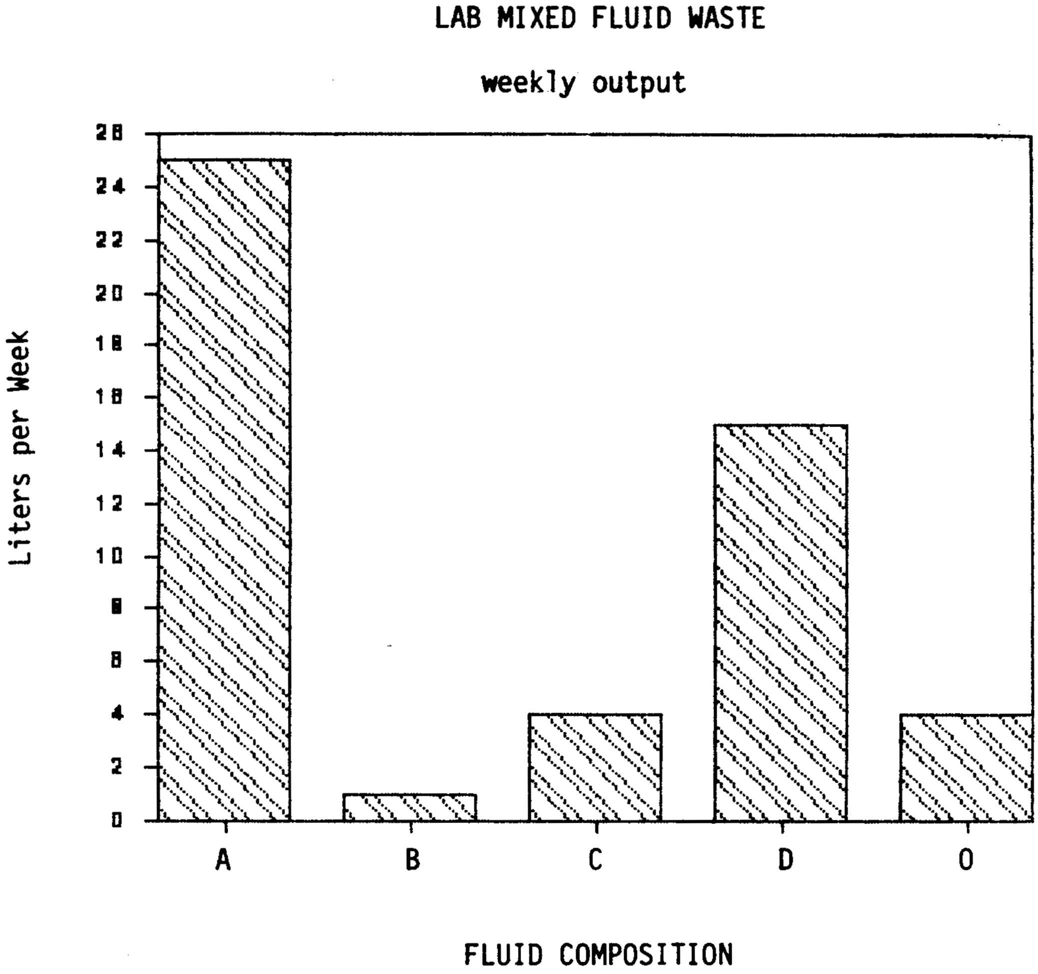
The primary determining factor in the selection of distillation equipment is the required purity of the distillate. There are numerous commercial stills available that will support volume reduction. A relatively simple still can be employed for volume reduction, that is, the separation of volatile from non-volatile components of the waste. However, the purity of the distillate will be too low for re-use in a laboratory setting. These stills do have the advantages of being relatively inexpensive and providing rapid processing of waste.

The production of a high-quality distillate for recycling as a pure reagent requires a more efficient still. Because many of the commercial scintillation fluids are complex mixture of solvents with relatively close boiling point, it was determined that a still with approximately 30 theoretical plates would be



KEY: A - Aquasol-2
B - Biofluor
H - Hydrafluor
L - Lefkofluor
S - ScintiVerse
U - Ultrafluor
M - Miscellaneous

Figure A: Relative weekly output of commercial scintillation fluid waste.



<u>KEY</u>: A - Toluene with moderate concentration PPO/POPOP

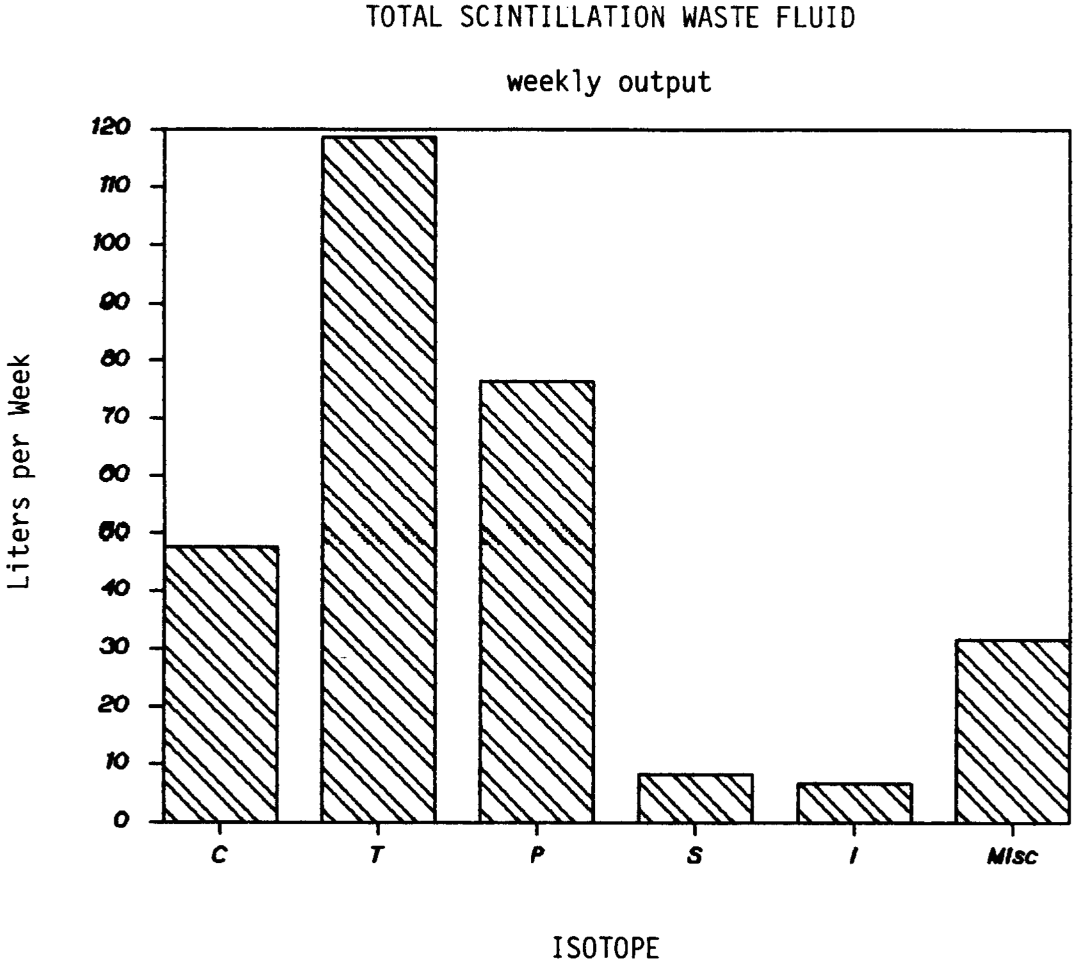
B - Toluene with Triton X-100 and PPO/POPOP

C - Toluene with Protosol and Omnifluor

D - Toluene with low concentration PPO/POPOP

O - Other (predominantly xylene based fluids)

Figure B: Relative weekly output of laboratory-prepared scintillation fluid waste.



KEY:
C - ^{14}C
T - ^{3}H
P - ^{32}P
S - ^{35}S
I - ^{125}I
Misc- Double tagged compounds and miscellaneous isotopes

Figure C: Distribution of radioisotopes in the scintillation fluid wastestream.

necessary to assure separation of pure distillates. A 30 plate still will provide separation of components with a boiling point difference of 6C or greater.

Another consideration in selecting a still is the processing method to be employed - that is, batch processing versus continuous run. Continuous run stills generally operate over a narrow range of parameters and are best suited for large volume constant constituent wastestreams. Since the scintillation fluid wastestream is highly variable, a more flexible batch processing still was selected. A batch still provides the capacity to more widely vary the operating parameters.

The B/R instrument model 8400 spinning-band solvent recycling system satisfies this requirement and was selected for this study. This still is also suitable for the distillation of other chemical waste, which would expand in utility in a research facility.

4. Developing a Working System for the Collection and Eventual Distribution of the Scintillation Fluid -

Collection of scintillation waste was coordinated with the existing radioactive waste management system. Investigator cooperation was excellent. Fortunately, each laboratory used only one type of scintillation fluid and they were supplied with an individual collection barrel for each isotope used. The investigators then notified our office when the container was full. A pick-up of the waste was scheduled within 2 days and a replacement collection container was left in the laboratory.

Distribution of either a redistilled starting solvent or in-house prepared cocktail could be accomplished through the Biochemistry Stockroom which is located in one of the research buildings. Most of the scintillation fluid is presently distributed through this location, therefore, distribution of the new products would simply require a stock substitution.

5. BENCH SCALE STUDIES

Liquid Scintillation Counting

Introduction

Liquid scintillation counting (LSC) was chosen as a method of measuring radioactivity in the distilled samples. LSC was chosen as the detection system because of its capacity to permit counting of multiple isotopes. 14-Carbon and tritium are easily distinguishable using LSC because of their differences in average beta energy, i.e. 14-Carbon : 0.049 meV; tritium : 0.0057 meV. We have utilized two channels in the liquid scintillation counter for the detection of tritium and 14-carbon. Each channel corresponds to a window. Each window corresponds to a specific isotope, i.e. channel 2 - 14-carbon, channel 3 - tritium. Each channel will then count for the specific isotope indicated.

Quenching, which is the inherent interference between the ionizing event and the detector, can affect the values obtained from LSC. Ionizing events can be quenched by impurity, chemical or color interference. External standardization, using a 137-cesium gamma source, was chosen as a method of correcting for the effect of quenching.

Materials

Scintillation counting fluid - Aquasol-2 (New England Nuclear)

Kimble 20 milliliter borosilicate glass scintillation vials

1000 microliter micropipette, disposable tips

10 milliliter disposable glass pipette

Beckman model LS 100C liquid scintillation counter

3H and 14C standards prepared by N. Niehaus,
Radiopharmacist, Duke University Medical Center

Methods

Counting Efficiency Determination

It is necessary to express results of liquid scintillation counting in absolute units. Data from liquid scintillation counting should be expressed in disintegrations per minute (dpm). Dpm's can then be converted to an absolute radioactivity unit, i.e. curie or becquerel (Kobayashi,Y).

The determination of counting efficiency begins with the preparation of known activity standards. The standards are prepared to mimic as closely as possible the experimental samples being counted. These standards were prepared as follows:

Tritium Standard - 10 milliliters Aquasol-2 spiked with 104,900.0 dpm tritium

14-Carbon Standard - 10 milliliters Aquasol-2 spiked with 20,000.0 dpm 14-carbon

The prepared standards were counted on a two minute cycle, and the corresponding counts for each isotope recorded (see Table 1). The counts per minute (CPM) data and known standard activities were then used to convert the CPM's to DPM's (Table 2). Counting efficiency was determined using the following equation:

dpm = (net cpm) / (sample counting efficiency)

where 100% sample counting efficiency = 1.00

Using the above equations and the data in Table 2, the following calculations can be made to determine counting efficiency.

14-Carbon

Average cpm (14-Carbon) = 19931.833
dpm (14-Carbon) = 20,000

sample counting efficiency = (net cpm) / dpm

= 19931.833 / 20,000

= 0.9965917

Tritium

Average cpm (Tritium) = 43713.833
dpm (Tritium) = 104,900

sample counting efficiency = (net cpm) / dpm

= 43713.833 / 104,900

= 0.4167191

Table 1

Actual CPM's Obtained from Standards

ISOTOPE	EXTERNAL STANDARD	CPM/10 ML 14-CARBON CHANNEL 2
14-CARBON	6.95	19920.00
14-CARBON	6.93	19924.00
14-CARBON	6.89	19951.50
AVG.CPM/10ML 14-CARBON		19931.833

ISOTOPE	EXTERNAL STANDARD	CPM/10 ML TRITIUM CHANNEL 3
TRITIUM	6.96	42469.00
TRITIUM	6.96	44257.50
TRITIUM	6.96	44415.00
AVG.CPM/10ML TRITIUM		43713.833

Table 2

Conversion to Counting Efficiency

ISOTOPE	CPM/10 ML 14-CARBON CHANNEL 2	DPM/10 ML 14-CARBON	COUNTING EFFICIENCY
14-CARBON	19920.00	20,000	0.996
14-CARBON	19924.00	20,000	0.9962
14-CARBON	19951.50	20,000	0.997575
AVERAGES 14-CARBON	19931.833	20,000	0.9965917

ISOTOPE	CPM/10 ML TRITIUM CHANNEL 3	DPM/10 ML TRITIUM	COUNTING EFFICIENCY
TRITIUM	42469.00	104,900	0.4048522
TRITIUM	44257.50	104,900	0.4219018
TRITIUM	44415.00	104,900	0.4234032
AVERAGES TRITIUM	43713.833	104,900	0.4167191

Preparation and Counting of Blanks

Blanks were prepared and run through the scintillation counter to determine background counts in the scintillation counting fluid (Aquasol-2). Blanks were prepared with 11 milliliters of Aquasol-2 which corresponds to the total volume for each scintillation vial counted. The cpm's obtained from counting the vials can be found in Table 3. The corresponding dpm conversions can be found in Table 4. Dpm's were obtained using the following equation:

dpm = (net cpm) / (sample counting efficiency)

Table 3

Actual CPM's Obtained for Blanks

EXTERNAL STANDARD	CPM/11 ML 14-CARBON CHANNEL 2	CPM/11 ML TRITIUM CHANNEL 3
6.89	10.50	21.50
6.88	10.50	21.00
6.85	8.50	26.50
6.86	12.00	27.50
AVERAGES		
6.87	10.375	24.125

Table 4

Conversion from CPM's to DPM's for Blanks

CPM/11 ML 14-CARBON CHANNEL 2	DPM/11 ML 14-CARBON CHANNEL 2	CPM/11 ML TRITIUM CHANNEL 3	DPM/11 ML TRITIUM CHANNEL 3
10.50	10.54	21.50	51.59
10.50	10.54	21.00	50.39
8.50	8.53	26.50	63.59
12.00	12.04	27.50	65.99
AVERAGES			
10.375	10.41	24.125	57.89

Sample preparation

Samples were prepared for liquid scintillation counting as follows:

10 milliliters of Aquasol-2 was placed in a 20 milliliter glass scintillation vial.

The material to be sampled was shaken for approximately 5 seconds to assure a homogeneous mixture.

A micropipette was used to draw a one milliliter sample.

The sample was drawn from the sample container approximately 1/4" from the bottom of the sample vial.

This drawn sample was then added to the 10 milliliters of Aquasol-2 in the scintillation vial.

The scintillation vial was then capped.

Sample counting

Samples were counted using a Beckman LS 100C liquid scintillation counter. The samples were run using an external standard, and a two minute per channel counting cycle. Each sample was counted for both tritium and 14-carbon.

Gas Chromatography

Gas chromatography (GC) was used both to identify the components in the stock scintillation fluids and processed distillates, and to quantitate the purity of the distillates. A Perkin Elmer series 800 gas chromatograph with flame ionization detector was used. A solvent flush injection method (using carbon disulfide as the flush) was employed with duplicate testing of each test sample.

Standard curves of signal strength versus concentration were obtained for the following chemicals - hexane, benzene, m-xylene, o-xylene, p-xylene, ethyl benzene, psuedocumene, toluene and mesitylene. The signal strength-volume relationship remained linear throughout the investigated range. In addition, stock scintillation fluids were analyzed by GC and the types and relative concentration of volatile components was determined. Then, samples taken from the distillation runs were analyzed against these standard profiles. Then, calculations were made converting sample results from signal strength to percent component volume.

DISTILLATION RUN S12

Background

The scintillation fluid for run S12 is prepared by the laboratory. It consists mainly of toluene, with Omnifluor (NEN) - a premixed liquid scintillation counting powder containing primary and secondary fluors, and Protosol (NEN) - a tissue and gel solubilizer added to the toluene. The samples being counted in this scintillation fluid are amino acids tagged with tritium. Occasionally the samples will contain digested tissue, which will contain a small amount of water. Also, amino acids will have the tendency to break down, forming water as a breakdown product.

Discussion

Run S12 showed good separation of radioactivity from the distilled solvent. Table 5 indicates the original starting volume of material being distilled as well as distillate collected for each indicated temperature range. The volume remaining in the still bottom is also indicated. There is a loss of 95 milliliters from the start of the run to the end. This can most likely be attributed to evaporation or experimental error. Table 6 indicates the actual cpm's obtained for each 11 milliliter sample counted. All samples for run S12 were counted for tritium and 14-carbon. The results from channel 2, which identifies 14-carbon activity have been omitted, since only background counts were obtained. Only results from channel 3 will be used for this discussion. Table 7 takes the cpm data from Table 6 and converts it from cpm/11 milliliters to dpm/11 milliliters, and eventually to dpm/milliliter.

An overall comparison between cut temperature and average dpm\ml can be seen in Graph 1. The starting material contained an average of 279.3 dpm of tritium per milliliter. The radioactivity in the starting material is not as important as the overall reduction in radioactivity throughout the distillation process, along with the increase in purity of the material as determined by gas chromatography (GC).

The volatile material distilled off in the first cut (<60.108C) consisted of 2.7% hexane and 95.4% toluene. The overall percent volatility of the first cut was 98.1%. Therefore, the hexane and toluene distilled off in this first cut comprised the entire amount of volatile material distilled off. The majority of tritium activity was concentrated in this first cut, which amounted to 4.5% of the original starting material. Cut 2 showed background counts for radioactivity and a purity for toluene of 99.1% as determined by GC. The percent volatility of the sample was also 99.1%, showing that only toluene was coming off at this temperature range. 90 milliliters of toluene were collected in cut 2. Toluene collected during cut 2 would be a candidate for reuse, due to its high purity and background radiation counts. Cut 3 showed a 98.1% volatility for the total sample. Toluene comprised 98.1%, showing the entire cut to be toluene. 2460 milliliters of toluene were collected in cut 3. Combining the amounts of toluene collected from cuts 2 and 3, we show 2550 milliliters of recoverable toluene. This converts to an 85% recovery rate for toluene. 11.8% of the original starting material had to be disposed of as regulated radioactive waste. 3.1% of the original sample volume was lost due to evaporation.

A scintillation cocktail of the type used in run S12 yielded a recycled product both high in recoverability and high in purity. This waste stream would be a good candidate for an in-house recycling program due to its above mentioned qualities.

Table 5

Distillation Information

CUT NUMBER	VOLUME COLLECTED (MILLILITERS)	CUT TEMP. oC
START VOLUME	3000	0.00
S12 - 1	135	60.1
S12 - 2	90	109.1
S12 - 3	2460	112.2
S12 - BOTTOM	220	178.0

GRAPH 1

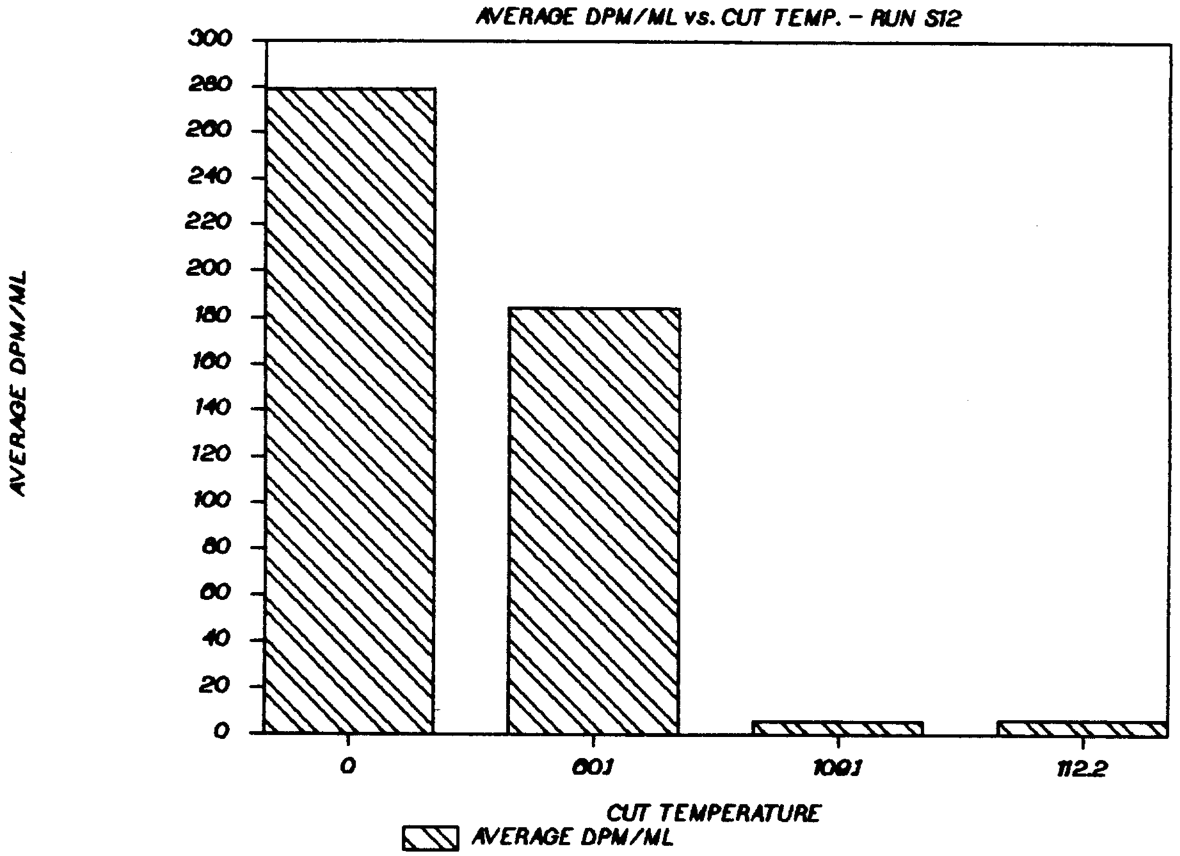

AVERAGE DPM/ML vs. CUT TEMP. - RUN S12
AVERAGE DPM/ML
300
280
260
240
220
200
180
160
140
120
100
80
60
40
20
0
0
601
1001
112.2
CUT TEMPERATURE
AVERAGE DPM/ML

Table 6

S12 - Scintillation Counting Data

SAMPLE ID	EXTERNAL STANDARD	CPM/11 ML TRITIUM CHANNEL 3
UNPROCESSED WASTE	6.36	1325.00
	6.41	1263.50
	6.42	1275.50
	6.34	1256.50
S12 - 1	6.27	1049.50
	6.43	702.00
	6.51	683.00
	6.26	944.00
S12 - 2	6.82	27.50
	6.95	28.50
	6.88	26.50
	6.92	24.00
S12 - 3	6.98	29.50
	6.89	27.00
	6.97	26.00
	6.96	30.00
S12 - BOTTOM	5.36	2190.50
	5.37	2263.50
	5.35	2176.00
	5.35	2147.00
	5.32	2721.00
	5.34	2417.00
	5.42	2542.50
	5.35	2314.00

Table 7

S12 Conversion of CPM's to DPM's

SAMPLE ID	CPM/11 ML TRITIUM CHANNEL 3	DPM/11 ML TRITIUM	DPM/ML TRITIUM
UNPROCESSED WASTE	1325.00	3179.60	289.05
	1263.50	3032.02	275.64
	1275.50	3060.81	278.26
	1256.50	3015.22	274.11
S12 - 1	1049.50	2518.48	228.95
	702.00	1684.59	153.14
	683.00	1638.99	149.00
	944.00	2265.31	205.94
S12 - 2	27.50	65.99	6.00
	28.50	68.39	6.22
	26.50	63.59	5.78
	24.00	57.59	5.24
S12 - 3	29.50	70.79	6.44
	27.00	64.79	5.89
	26.00	62.39	5.67
	30.00	71.99	6.54
S12 - BOTTOM	2190.50	5256.54	477.87
	2263.50	5431.71	493.79
	2176.00	5221.74	474.70
	2147.00	5152.15	468.38
	2721.00	6529.58	593.60
	2417.00	5800.07	527.28
	2542.50	6101.23	554.66
	2314.00	5552.90	504.81

DISTILLATION RUN S13

Background

The scintillation fluid for run S13 is prepared by the laboratory. It is the same cocktail used for run S12 and consists mainly of toluene, with Omnifluor (NEN) - a premixed liquid scintillation counting powder containing primary and secondary fluors, and Protosol (NEN) - a tissue and gel solubilizer added to the toluene. The samples being counted in this scintillation fluid are amino acids tagged with tritium. Occasionally the samples will contain digested tissue, which will contain a small amount of water. Also, amino acids will have the tendency to break down, forming water as a breakdown product.

Discussion

Run S13 showed good separation of radioactivity from the distilled solvent. Table 8 indicates the original starting volume of material being distilled as well as distillate collected for each indicated temperature range. There is a gain of 25 milliliters from the start of the run to the end. This small difference can be attributed to variance in methods of measurement. Table 9 indicates the actual cpm's obtained for each 11 milliliter sample counted. All samples for run S13 were counted for tritium and 14-carbon. The results from channel 2, which identifies 14-carbon activity have been omitted, since only background counts were obtained. Only results from channel 3 will be used for this discussion. Table 10 takes the cpm data from Table 9 and converts it from cpm/11 milliliters to dpm/11 milliliters, and eventually to dpm/milliliter.

An overall comparison between cut temperature and average dpm/ml can be seen in Graph 2. The starting material contained an average of 34.93 dpm of tritium per milliliter. One would expect a higher activity for the original unprocessed waste. The low counts obtained were due to quenching of the sample during counting resulting from impurities which were present in the sample.

Cut 1 contained an average of 864.95 dpm/ml. The high counts obtained for cut 1 would not make it a candidate for recycling. Cut 2, distillate collected between 60.7C and 71.1C, came off the column as a bilayered mixture. The differences in radioactivity can be seen in Graph 2 at the cut temperature of 71.1C. The bar on the left represent the lower aqueous phase, which contained an average dpm/ml of 1130.86. Only background radiation was detected when the upper phase was counted, which is indicated by the small bar adjacent and directly to the right of the large bar. The high activity in the aqueous phase was probably due to tritium exchange. Tritium exchange involves a radiochemical transfer from one molecule to another. In this case, possibly from the labeled amino acids to water molecules. GC analysis revealed that the upper layer was virtually all toluene with a trace of hexane present. Dewatering of the scintillation fluid prior to distillation could have eliminated most of this problem. The amount of water distilled off in each cut can be controlled by the adjustment of the cut temperature ranges. For cut 3, GC analysis showed 100% toluene, and scintillation counting revealed background counts. The majority of the tritium activity was concentrated in the first and second cut. These two cuts comprise only 6.4% of the original starting volume. The material collected in cut 3 would be a candidate for recycling due to the low background radiation combined with high solvent purity. 2310 milliliters were collected in cut 3. This volume amounts to an 84.7% recovery rate for toluene in this run. 15.2% of the original starting material had to be disposed of as regulated radioactive waste.

A scintillation cocktail of the type used in run S13 yielded a product both high in recoverability and high in purity. This waste stream would be a good candidate for an in-house recycling program due to its above mentioned qualities.

Table 8

Distillation Information

CUT NUMBER	VOLUME COLLECTED (MILLILITERS)	CUT TEMP. oC
START VOLUME	2700	0.00
S13 - 1	100	60.7
S13 - 2	75	71.1
S13 - 3	2310	102.1
S13 - BOTTOM	240	109.0+

GRAPH 2

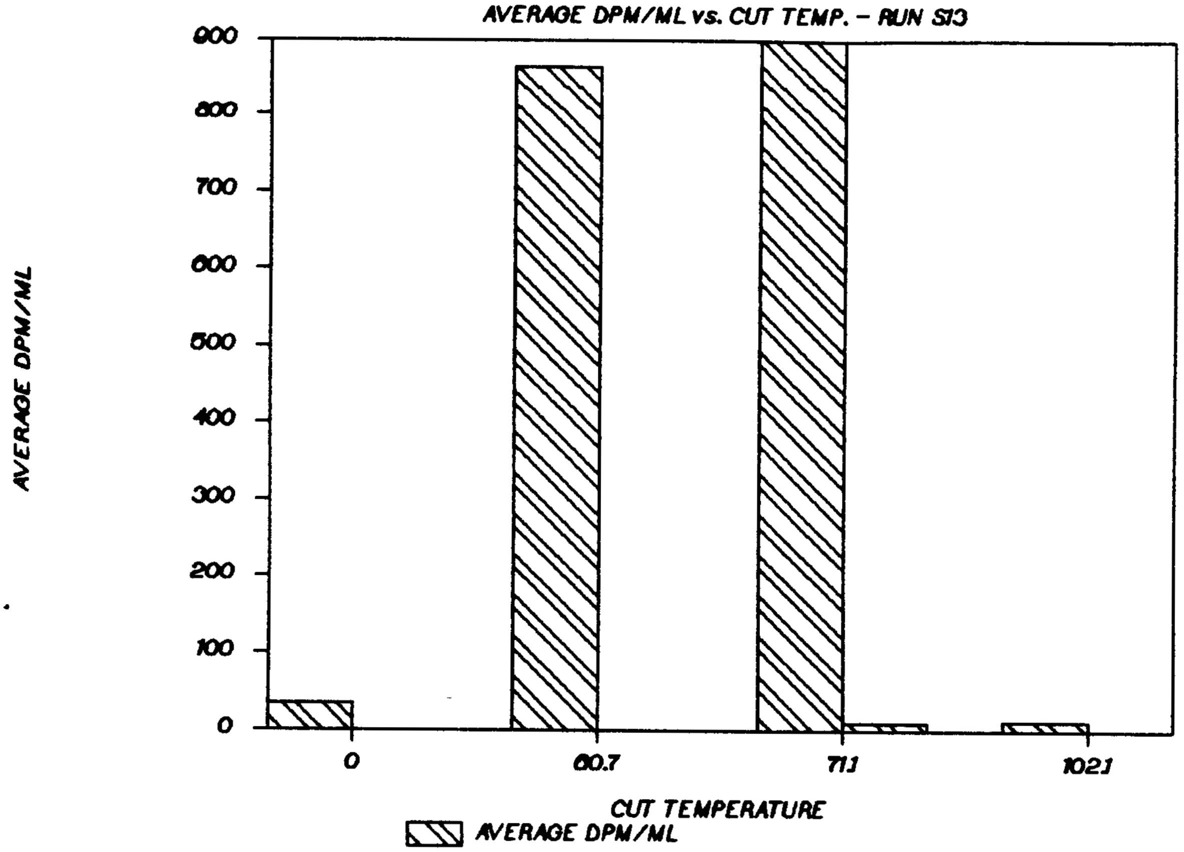

AVERAGE DPM/ML vs. CUT TEMP. - RUN S13
AVERAGE DPM/ML
900
800
700
600
500
400
300
200
100
0
0
60.7
71.1
102.1
CUT TEMPERATURE
▧ AVERAGE DPM/ML

Table 9

S13 - Scintillation Counting Data

SAMPLE ID	EXTERNAL STANDARD	CPM/11 ML TRITIUM CHANNEL 3
UNPROCESSED WASTE	6.95	108.50
	6.90	317.00
	6.90	101.00
	6.82	114.00
S13 - 1	6.21	3222.00
	6.10	4752.50
	6.06	4653.50
	6.22	3231.50
S13 - 2 (see note 1)	6.17	5046.50
	6.07	5321.00
S13 - 2	6.94	45.50
	6.95	44.00
S13 - 3	6.97	48.50
	6.97	65.50
	6.92	59.50
	6.96	55.50
S13 - BOTTOM	5.52	4289.00
	5.54	4582.50
	5.45	4460.00
	5.44	4689.00
	5.56	4522.00
	5.54	4708.00
	5.59	4643.50
	5.44	4631.00

Note 1 - This cut was an aqueous layer, and is explained in the discussion section.

Table 10

S13 - Conversion of CPM's to DPM's

SAMPLE ID	CPM/11 ML TRITIUM CHANNEL 3	DPM/11 ML TRITIUM	DPM/ML TRITIUM
UNPROCESSED WASTE	108.50	260.37	23.67
	317.00	760.70	69.15
	101.00	242.37	22.03
	114.00	273.57	24.87
S13 - 1	3222.00	7731.83	702.89
	4752.50	11404.57	1036.78
	4653.50	11167.00	1015.18
	3231.50	7754.62	704.97
S13 - 2 (see note 2)	5046.50	12110.08	1100.92
	5321.00	12768.79	1160.80
S13 - 2	45.50	109.19	9.93
	44.00	105.59	9.60
S13 - 3	48.50	116.39	10.58
	65.50	157.18	14.29
	59.50	142.78	12.98
	55.50	133.18	12.11
BOTTOM	4289.00	10292.31	935.66
	4582.50	10996.62	999.69
	4460.00	10702.65	972.97
	4689.00	11252.18	1022.93
	4522.00	10851.43	986.50
	4708.00	11297.78	1027.06
	4643.50	11143.00	1013.00
	4631.00	11113.00	1010.27

Note 2 - This cut was an aqueous layer, and is explained in the discussion section.

DISTILLATION RUN S14

Background

The scintillation fluid for run S14 is the commercially prepared fluid - "Lefko-Fluor". This product is marketed by Research Products International Corp.. The company describes this product as an aromatic hydrocarbon mixture with a boiling point range of 156-179.4C, and a flash point of greater then 100F. Lefko-Fluor also contains added fluors and Triton X-100 - a surfactant. The samples being counted in this scintillation fluid are aqueous antibodies and proteins labeled with tritium.

Discussion

Lefko-Fluor posed a problem as far as recycling of this product was concerned. Several factors were involved - 1) this product is comprised of many different components, 2) these components have high boiling points, 3) in order to recycle this scintillation fluid, reduced pressure distillation would have to be employed, and 4) dewatering would have to occur prior to the distillation process. According to current regulations, this waste stream is considered deregulated, and can be disposed of through incineration at several licensed sites. However, the scope of this study was to look at alternatives to existing disposal options.

Volume reduction techniques, rather than recycling would be more appropriately applied in this situation. The volatile fraction of Lefko-Fluor could be distilled off to eliminate the radioactivity and then disposed of as high BTU solvents to be used for recovery of their fuel value. Although the findings from run S14 were not as hoped for, the study did add to our understanding of the distillation potential. Each of the above mentioned points will be discussed, and recommendations for alternatives to recycling suggested.

During this discussion, purity of the various cuts will not be addressed. It would not be feasible to recycle products such as Lefko-Fluor for reuse because of the many components which make up the product, along with the inability to distill most of the product at atmospheric pressure. Instead, the obtainability of a non-radioactive, high BTU solvent after the distillation process will be discussed.

Table 11 indicates the original starting volume of material being distilled as well as distillate collected for each indicated temperature range. There is a loss of 85 milliliters from the start of the run to the end. This can most likely be attributed to evaporation, and experimental error. Table 12 indicates the actual cpm's obtained for each 11 milliliter sample counted. All samples for run S14 were counted for tritium and 14-carbon. The results from channel 2, which identifies 14-carbon activity have been omitted, since only background counts were obtained. Only results from channel 3 will be used for this discussion. Table 13 takes the cpm data from Table 12 and converts it from cpm/11 milliliters to dpm/11 milliliters, and eventually to dpm/milliliter.

An overall comparison between cut temperature and average dpm/ml can be seen in Graph 3. The starting material contained an average of 30.25 dpm of tritium per milliliter. One would expect a higher activity in the original unprocessed waste. The observed low counts can most likely be attributed to quenching during scintillation counting, caused by contaminants in the unprocessed waste. The scintillation counts obtained for cuts 1 and 2 indicate a radioactivity level higher than the starting material. The distillate from these cuts would produce very little quenching during scintillation counting, due to the purity of the material. Therefore, it is expected that the actual radioactivity level in the unprocessed waste is higher the experimental values obtained.

The distillate from cut 1 and cut 2 contained two phases, an aqueous phase and an organic phase. The organic phase from cut 1 had an average dpm/ml of 11.62, while the aqueous phase had an average dpm/ml of 417.58. The high activity in the aqueous phase was probably due to tritium exchange. Tritium exchange involves a radiochemical transfer from one molecule to another. In this case, possibly from the labeled antibodies and proteins to water molecules. The distillate from cut 2 was similar and the organic phase had an average dpm/ml of 9.38, while the aqueous phase had an average dpm/ml of 600.55. Again, tritium exchange could be attributed to the high dpm/ml in the aqueous phase. Cut 1 and cut 2 make up 11.8% of the original starting volume. Although this quantity may seem small, there are alternatives for eliminating the radioactivity from the aqueous phases. This can be done by dewatering the material prior to distillation, using a drying agent. Cut 3 contained only background counts of radiation. This factor would make the distillate a candidate for volume reduction techniques. Cut 3 comprised only 24.9% of the original starting material. After processing cuts 1 and 2 to eliminate any water present, the distillate, amounting to approximately 6% of the original starting volume could be combined with the distillate from cut 3 to yield a 31% recovery rate. The distilled material from these cuts could then be disposed of by incineration or used in fuel recovery. After the distillation process for run S14, 75.1% of the original starting material had to be disposed of as regulated radioactive waste.

The low overall recovery rate can be attributed to the solvent system used in Lefko-Fluor. The majority of its components are high boiling point materials (i.e. methylated benzenes), which could not be distilled with the apparatus used in this study. However, increased recovery of an end product with low radioactivity and a high BTU value would probably result from the utilization of reduced pressure distillation techniques along with dewatering prior to distillation. This expanded end product would then be suitable for disposal through incineration or a fuel recovery program.

Table 11

Distillation Information

CUT NUMBER	VOLUME COLLECTED (MILLILITERS)	CUT TEMP. oC
START VOLUME	2800	0.00
S14 - 1	20	60.9
S14 - 2	300	91.1
S14 - 3	675	104.2
S14 - BOTTOM	1720	163.0+

GRAPH 3

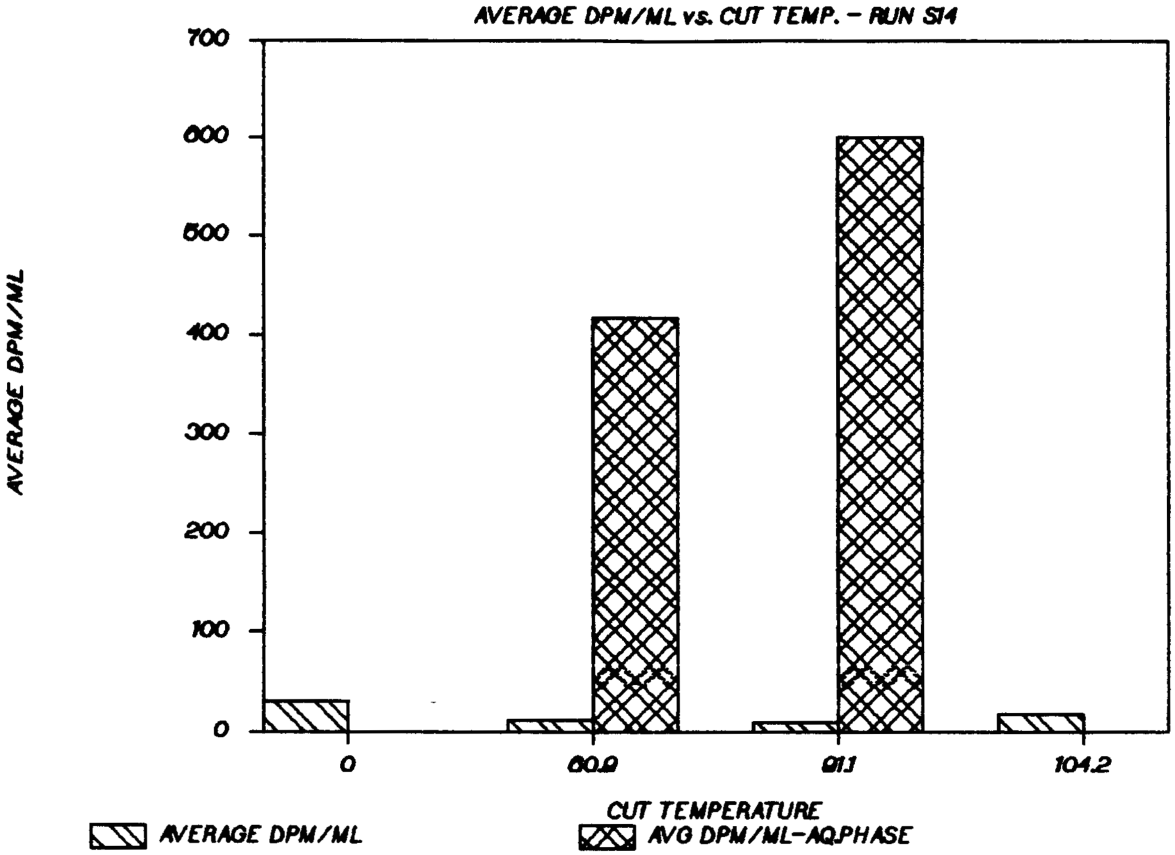

AVERAGE DPM/ML vs. CUT TEMP. - RUN S14
AVERAGE DPM/ML
700
600
500
400
300
200
100
0
0
60.9
91.1
104.2
CUT TEMPERATURE
AVERAGE DPM/ML
AVG DPM/ML-AQ.PHASE

Table 12

S14 - Scintillation Counting Data

SAMPLE ID	EXTERNAL STANDARD	CPM/11 ML TRITIUM CHANNEL 3
UNPROCESSED	6.88	176.50
WASTE	6.85	202.00
	6.85	190.50
	6.90	199.50
	6.83	85.50
	6.91	98.00
	6.87	76.00
	6.90	81.50
S14-1-TOP	6.95	40.50
	6.82	43.50
	6.87	80.50
	6.91	48.50
S14-1-BOTTOM	5.80	1831.00
	6.06	2048.50
	5.95	1975.50
(see note 3)	5.93	1801.50
S14-2-TOP	6.95	45.50
	6.87	40.00
	6.96	43.00
	6.97	43.50
S14-2-BOTTOM	6.00	2986.00
	6.05	2904.50
	6.10	2614.50
(see note 3)	6.14	2506.50
S14 - 3	6.87	77.00
	6.87	77.00
	6.88	89.50
	6.88	73.50
BOTTOM	6.22	1251.50
	6.16	1345.50
	6.18	1389.00
	6.16	1265.00
	6.19	1340.00
	6.09	1269.00
	6.13	1330.50
	6.21	1277.50

Note 3 - This cut contained two phases, and is explained in the discussion of section.

Table 13

S14 - Conversion of CPM's to DPM's

SAMPLE ID	CPM/11 ML TRITIUM CHANNEL 3	DPM/11 ML TRITIUM	DPM/ML TRITIUM
UNPROCESSED	176.50	423.55	38.50
WASTE	202.00	484.74	44.07
	190.50	457.14	41.56
	199.50	478.74	43.52
	85.50	205.17	18.65
	98.00	235.17	21.38
	76.00	182.38	16.58
	81.50	195.58	17.78
S14-1-TOP	40.50	97.19	8.84
	43.50	104.39	9.49
	80.50	193.18	17.56
	48.50	116.39	10.58
S14-1-BOTTOM	1831.00	4393.85	399.44
	2048.50	4915.78	446.89
	1975.50	4740.60	430.96
(see note 4)	1801.50	4323.06	393.01
S14-2-TOP	45.50	109.19	9.93
	40.00	95.99	8.73
	43.00	103.19	9.38
	43.50	104.39	9.49
S14-2-BOTTOM	2986.00	7165.50	651.41
	2904.50	6969.92	633.63
	2614.50	6274.01	570.36
(see note 4)	2506.50	6014.84	546.80
S14 - 3	77.00	184.78	16.80
	77.00	184.78	16.80
	89.50	214.77	19.52
	73.50	176.37	16.03
BOTTOM	1251.50	3003.22	273.02
	1345.50	3228.79	293.53
	1389.00	3333.18	303.02
	1265.00	3035.62	275.97
	1340.00	3215.60	292.33
	1269.00	3045.22	276.84
	1330.50	3192.80	290.25
	1277.50	3065.61	278.69

Note 4 - This cut contained two phases, and is explained in the discussion section.

DISTILLATION RUN S15

Background

The scintillation fluid for run S15 is prepared by the laboratory. It consists mainly of toluene, with Triton X-100 - a surfactant added to the toluene. The samples being counted in this scintillation fluid are aqueous phenolic samples labeled with 14-carbon.

Discussion

Run S15 did not show good separation of radioactivity from the distilled solvent. Table 14 indicates the original starting volume of material being distilled as well as distillate collected for each indicated temperature range. The volume remaining in the still bottom is also indicated. There is a loss of 35 milliliters from the start of the run to the end. This can most likely be attributed to evaporation and experimental error. Table 15 indicates the actual cpm's obtained for each 11 milliliter sample counted. All samples were counted for tritium and 14-carbon. The results from channel 3, which identifies tritium activity have been omitted, since only background counts were obtained. Only results from channel 2 will be used for this discussion. Table 16 takes the cpm data from Table 15 and converts it from cpm/11 milliliters to dpm/11 milliliters, and eventually to dpm/milliliter.

An overall comparison between cut temperature and average dpm/ml can be seen in Graph 4. The starting material contained an average of 11.8 dpm of 14-carbon per milliliter.

An overall decrease in dpm/ml was not seen during the distillation of run S15. However, an increase in product purity was seen throughout the run. The volatile material distilled off in the first cut was comprised of two phases, an organic phase and an aqueous phase. GC analysis revealed that the organic phase was pure toluene, but it still contained radioactivity. GC analysis of cut 2 revealed a distillate of toluene at a purity of 99.25%. Again, the level of radioactivity has not decreased significantly from the level of the starting material.

The purity of the material obtained in run S15 indicates a potential for recycling of 14-carbon labeled scintillation fluid. Pretreatment would be required to eliminate water and 14-carbon activity prior to distillation. A search of the literature revealed an article which discusses the pretreatment of liquid scintillation waste (Sasaki,T). The article outlines experiments to remove radioactivity from liquid scintillation waste using various absorbents - including calcium chloride, calcium hydroxide, aluminum oxide, silicon dioxide, molecular sieves and synthetic zeolite. 14-Carbon labeled methanol and 14-carbon labeled glucose were used along with various scintillating fluids, including toluene, dioxane, toluene-triton and Biofluor. Results of the study showed the greatest success with the removal of radioactivity from 14-carbon labeled methanol using calcium chloride.

14-Carbon labeled scintillation waste of types similar to that used in run S15 would have the potential to be included in a scintillation fluid recycling program if pretreatment were found to be successful in eliminating radioactivity. The purity of the distillate has already been demonstrated. Further studies would be needed to look at the effects of pretreatment of 14-carbon labeled scintillation wastes.

Table 14

Distillation Information

CUT NUMBER	VOLUME COLLECTED (MILLILITERS)	CUT TEMP. OC
START VOLUME	1800	0.0
S15 - 1	590	60.1
S15 - 2	1005	103.1
S15 - BOTTOM	170	111.0+

GRAPH 4

AVERAGE DPM/ML vs. CUT TEMP. - RUN S15

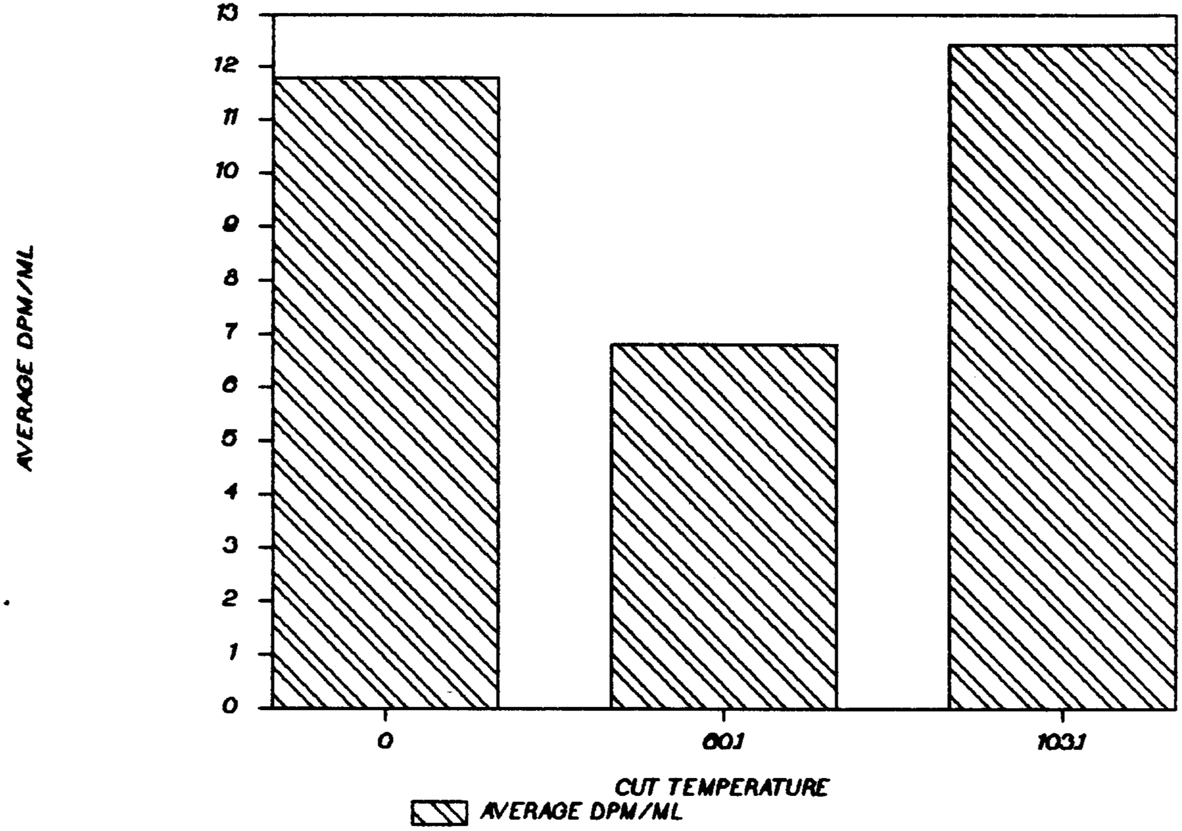


Table 15

S15 - Scintillation Counting Data

SAMPLE ID	EXTERNAL STANDARD	CPM/11 ML 14-CARBON CHANNEL 2
UNPROCESSED WASTE	6.83	138.00
	6.78	136.50
	6.82	136.00
	6.88	124.00
	6.81	114.50
	6.88	115.50
	6.78	118.50
	6.93	154.00
S15 - 1	6.92	98.00
	6.99	90.00
	6.92	55.00
	6.88	53.50
S15-1-TOP	6.98	66.00
	7.03	59.00
	6.94	50.50
	6.98	53.00
S15-1-BOTTOM	6.06	73.50
	6.37	53.50
	6.17	59.50
(see note 5)	6.09	78.50
S15 - 2	6.92	127.50
	6.89	131.00
	6.92	157.00
	6.88	130.00
S15 -BOTTOM	6.38	74.00
	6.27	100.00
	6.31	60.00
	6.28	86.00
	6.48	111.50
	6.43	111.50
	6.43	110.00
	6.39	105.50

Note 5 - This cut contained two phases, and is explained in the discussion section.

Table 16

S15 - Conversion of CPM's to DPM's

SAMPLE ID	CPM/11 ML 14-CARBON CHANNEL 2	DPM/11 ML 14-CARBON	DPM/ML 14-CARBON
UNPROCESSED WASTE	138.00	138.47	12.59
	136.50	136.97	12.45
	136.00	136.47	12.41
	124.00	124.42	11.31
	114.50	114.89	10.44
	115.50	115.90	10.54
	118.50	118.90	10.81
	154.00	154.53	14.05
S15 - 1	98.00	98.34	8.94
	90.00	90.31	8.21
	55.00	55.19	5.02
	53.50	53.68	4.88
S15-1-TOP	66.00	66.23	6.02
	59.00	59.20	5.38
	50.50	50.67	4.61
	53.00	53.18	4.83
S15-1-BOTTOM	73.50	73.75	6.70
	53.50	53.68	4.88
	59.50	59.70	5.43
(see note 6)	78.50	78.77	7.16
S15 - 2	127.50	127.94	11.63
	131.00	131.45	11.95
	157.00	157.54	14.32
	130.00	130.44	11.86
S15 -BOTTOM	74.00	74.25	6.75
	100.00	100.34	9.12
	60.00	60.21	5.47
	86.00	86.29	7.84
	111.50	111.88	10.17
	111.50	111.88	10.17
	110.00	110.38	10.03
	105.50	105.86	9.62

Note 6 - This cut contained two phases, and is explained in the discussion section.

DISTILLATION RUN S16

Background

The scintillation fluid for run S16 is prepared by the laboratory. It consists primarily of toluene, with fluors added as needed. Samples are applied to a filter paper disk and evaporated to dryness prior to their addition to the scintillation fluid. This eliminates the need for added surfactants. The samples being counted in this scintillation fluid are biochemical compounds labeled with tritium.

Discussion

Run S16 showed background radiation counts throughout the entire distillation process. It is important to note that the laboratory does not add their tritiated samples directly to their scintillation fluid. Instead, they apply their samples to filter paper disks. These disks are then placed in the vials with the scintillation fluid, and counted. The majority of radioactivity remains on the disks; therefore allowing the laboratory to reuse their scintillation fluid as long as only background counts are present. Table 17 indicates the original starting volume of material being distilled as well as distillate collected for each indicated temperature range. The volume remaining in the still bottom is also indicated. There is a loss of only 8 milliliters from the start of the run to the end. This can be attributed to evaporation or experimental error. All samples for run S16 were counted for tritium and 14-carbon. The results from channel 2, which identifies 14-carbon activity have been omitted, since only background counts were obtained. Only results for channel 3 will be used for this discussion. Table 19 takes the cpm data from Table 18 and converts it from cpm/11 milliliters to dpm/11 milliliters, and eventually to dpm/milliliter.

An overall comparison between cut temperature and average dpm/ml can be seen in Graph 5. The starting material contained an average of 4.4 dpm of tritium per milliliter. This is considered a background level.

As shown in runs S12 and S13, it is possible to eliminate tritium activity from toluene based scintillation fluid. It has also been shown by GC analysis that a high purity toluene can be obtained from this distillation process. GC analysis of cut 1 confirmed this showing a 97.4% purity for the recovered toluene. This distillate comprised the entire amount of volatile material distilled off. The distillate obtained from cut 1 showed a 97.5% recovery rate for this run, leaving 2.1% of the original starting material to be disposed of as regulated radioactive waste.

GRAPH 5

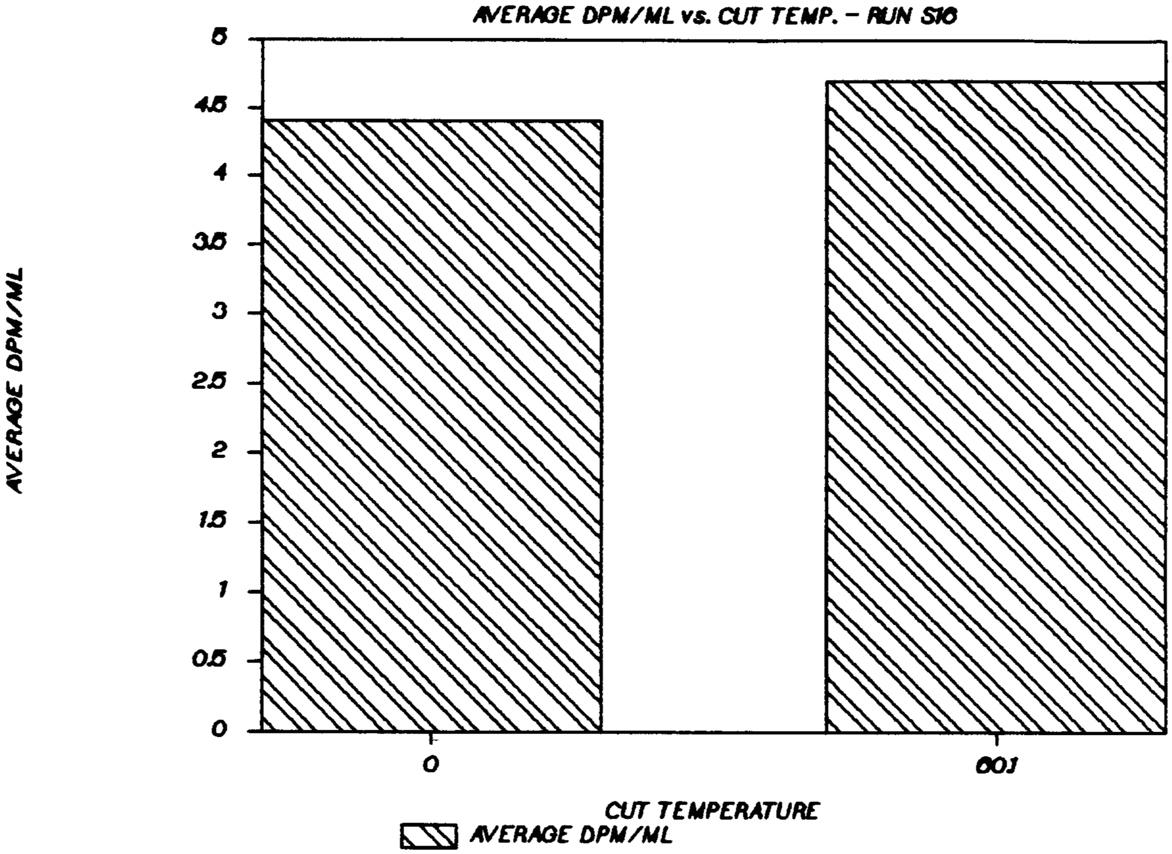

AVERAGE DPM/ML vs. CUT TEMP. - RUN S10
AVERAGE DPM/ML
5
4.5
4
3.5
3
2.5
2
1.5
1
0.5
0
0
001
CUT TEMPERATURE
▨ AVERAGE DPM/ML

Table 17

Distillation Information

CUT NUMBER	VOLUME COLLECTED (MILLILITERS)	CUT TEMP. oC
START VOLUME	2000	0.00
S16 - 1	1950	60.1
S16 - BOTTOM	42	111.0+

Table 18

S16 - Scintillation Counting Data

SAMPLE ID	EXTERNAL STANDARD	CPM/11 ML TRITIUM CHANNEL 3
UNPROCESSED WASTE	6.93	23.50
	6.83	20.00
	6.93	20.00
	6.91	17.00
	6.99	22.00
	6.98	23.00
	6.92	19.00
	6.97	17.50
S16 - 1	6.96	16.00
	6.96	20.50
	6.86	23.50
	6.90	26.50
S16 - BOTTOM	6.45	382.00
	6.53	376.00
	6.46	368.00
	6.34	365.00
	6.45	395.00
	6.44	388.50
	6.45	397.50
	6.58	308.50

Table 19

Conversion of CPM's to DPM's

SAMPLE ID	CPM/11 ML TRITIUM CHANNEL 3	DPM/11 ML TRITIUM	DPM/ML TRITIUM
UNPROCESSED WASTE	23.50	56.39	5.13
	20.00	47.99	4.36
	20.00	47.99	4.36
	17.00	40.79	3.71
	22.00	52.79	4.80
	23.00	55.19	5.02
	19.00	45.59	4.14
	17.50	41.99	3.82
S16 - 1	16.00	38.40	3.49
	20.50	49.19	4.47
	23.50	56.39	5.13
	26.50	63.59	5.78
S16 - BOTTOM	382.00	916.68	83.33
	376.00	902.29	82.03
	368.00	883.09	80.28
	365.00	875.89	79.63
	395.00	947.88	86.17
	388.50	932.28	84.75
	397.50	953.88	86.72
	308.50	740.31	67.30

Distillation of Non-Radioactive Scintillation Fluid

Several commercial scintillation fluids were distilled, and the collected distillate analyzed by GC to determine their feasibility for recycling. Based on the results of the radioactive distillation runs (S12-S16) a correlation could be made as to the practicality of recycling these commercial fluids. The following commercial scintillation fluids were analyzed:

1. OCS
2. Aquasol-2 (NEN)
3. Biofluor (NEN)
4. Ultrafluor (National Diagnostics)
5. Hydrofluor (National Diagnostics)

Each commercial scintillation fluid will be discussed in terms of their components and recycling potential.

OCS

OCS is a xylene based scintillation fluid. This type of fluid is highly desirable for an in-house recycling program due to the easy separability of volatile components. During the distillation of OCS, two cut temperatures were used - 60.1C and 134.1C. Twenty-five milliliters were collected from cut 1. The distillate was 92.4% xylene (ortho, meta and para), 3.3% toluene and 1% hexane as analyzed by GC. During a distillation run using radioactive scintillation fluid of this type, one might expect some radioactivity in the distillate of cut 1. This assumption is based on the results from runs S12-S16. 1800 milliliters of distillate were collected in cut 2. The composition of cut 2 was 94.9% xylene (ortho, meta and para) and 0.45% toluene as determined by GC. Using the volume collected from cut 2, the recovery rate obtained was 90%, based on a starting volume of 2000 milliliters. Several factors would determine the actual recovery rate obtained from distillation of radioactive scintillation fluid - these include whether or not the sample is aqueous and whether or not other contaminants are present in the fluid.

Based on this preliminary data, OCS scintillation fluid would be a good candidate for an in-house scintillation fluid recycling program.

Biofluor

Biofluor is primarily a psuedocumene based scintillation fluid. There is a drawback to distillation of this type of material at atmospheric pressure. Due to the high boiling point of the material (173C), recovery of distillate at atmospheric pressure is low.

Two cut temperatures were used for the distillation of Biofluor - 60.2C and 166.2C. The volatile material collected from cuts 1 and 2 averaged 96.8% psuedocumene as analyzed by GC. This comprised the entire amount of volatile material collected from the two cuts. A 46% recovery rate was obtained from distillation at atmospheric pressure. Because Biofluor is primarily psuedocumene, recycling of this material would be preferred over volume reduction, and ultimate incineration. Further studies would be necessary to look at the applicability of reduced pressure distillation of this material.

Aquasol-2

Aquasol-2 is a scintillation fluid comprised of toluene, m-xylene, o-xylene and mesitylene. Like Biofluor, Aquasol-2 contains a high boiling point solvent - mesitylene. The recovery of all components of the fluid is impossible at atmospheric pressure. We showed a 45% recovery rate. Cut 1, which contained only 5 milliliters, consisted of toluene, m-xylene and o-xylene. Cut 2, which contained 600 milliliters was comprised of toluene, m-xylene, o-xylene and mesitylene. Cut 3, containing 200 milliliters, contained a slight amount of toluene, the remainder m-xylene and mesitylene. 1000 milliliters remained in the still bottom after distillation. It is speculated that the still bottom material contains methylated benzene compounds with boiling points greater than 160C.

The potential exists to recover various fractions from the Aquasol-2 for reuse in other applications. Reduced pressure distillation would increase the recovery rate of the higher boiling point solvents.

Ultrafluor, Hydrofluor

Ultrafluor and Hydrofluor are scintillation fluids comprised of several high boiling point solvents (>140C). These fluids are similar to Lefko-Fluor (refer to Run S14), in that they do not yield a good recovery from distillation at atmospheric pressure. Because of the complex make-up of these solvents, along with their high boiling points, these types of scintillation fluids are candidates for volume reduction, rather then recycling.

Applying reduced pressure distillation techniques, recovery rates for these types of fluids could be increased. Assuming, the distillate would be free of radioactivity, it could then be disposed of through incineration, or through a fuel recovery program.

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6. Feasibility of Segregating, Redistilling and Distributing the Recovered Scintillation Fluid -

The factors that impact on the implementation of an effective volume reduction/recycling program for scintillation fluid include an accurate characterization of the wastestream, the potential to control the types of fluid used on-site, logistics, facility requirements and technical limitations. The summary of the relative impact of each of these factors on the feasibility of implementing a program, as evaluated in the present study, is as follows:

A. Characterization and analysis of the wastestream was accomplished during a six week laboratory audit process. We increased the investigators understanding of the goals of this process through personal communication, seeking administrative support and the publication of an article outlining the study in a facility publication. The willingness of investigators to cooperate in this process has contributed to the accumulation of an accurate profile of the wastestream. The probability of variations in the wastestream was identified. However, these changes should be easily documented because of the open communication that was established during the laboratory audits.

CONCLUSION - The scintillation fluid wastestream can be easily characterized through a comprehensive audit program. Investigator cooperation is critical in all phases of the proposed program and must be developed through education. The audit process is an integral part of this education.

B. The possibility of limiting and controlling the types of scintillation fluids being used on-site was identified during the audit process. First, numerous investigators expressed interest in using a high-quality product that could be supplied at a reduced cost. Many investigators also were interested in supporting an environmentally sound alternative to landfill disposal of the waste they generated. Finally, the fact that investigators could plan to substitute a new scintillation fluid at a start of new studies provides an opportunity to standardize the product being used.

CONCLUSION - It was obvious that several investigators were committed to long-term use of one product and were not capable of switching (although they agreed to support segregation and improved management). However, the opportunity to convert to a more standard and consistent wastestream was found to exist. This possibility would be dependent on the quality and supply of the fluid, as well as effective salesmanship by personnel in the Environmental Safety Division.

C. Logistics concerns are primarily related to the segregation, collection and distribution of the scintillation fluid. All investigators had agreed to support intra-laboratory segregation of their waste during the audit process. Subsequently, a segregation system was established in 12 laboratories during a period when materials were being collected for bench scale distillation studies. The collection and distribution of scintillation fluid would only require minor modifications in existing management mechanisms.

CONCLUSIONS - A scintillation fluid volume reduction/recycling program is logistically possible at our institution.

D. The facility requirements for a volume reduction/recycling program include both building floor space and equipment. Floor space is required for special processing, such as, crushing the scintillation vials for efficient collection of the fluid, on-site storage for decay, pre-processing of the scintillation fluid waste (precipitation out of radioactive components and dehydrating), and distillation. Specialized equipment includes a crusher/compactor, spinning-band stills, mixer or reaction vessels, and localized exhaust and fume hoods.

The new Environmental Safety Building, which is presently under construction, was designed to provide space and specialized ventilation for these processes. In addition, the spinning band stills that are intended to process chemical waste could readily be applied to the distillation of the scintillation fluids.

CONCLUSION - A volume reduction/recycling program is feasible in light of the new building and equipment that will be available at our institution.

E. Technical limitations produced the greatest constraints to the implementation of a volume reduction/recycling program. As was discussed in Section 5., several wastestreams provided technical hurdles that impact on the potential level of volume reduction/recycling that can be achieved. Specific problems included; 1) reduced efficiency of distillation because of contamination with water; 2) carry-over of 14-carbon labelled compounds into the distillate; and 3) complex scintillation fluids composed primarily of high boiling point solvents.

While each of these problems have a significant impact on the efficiency of recycling, potential solutions were identified during the study. First, the use of dewatering agents (calcium chloride, silica gel, molecular sieves, etc.) could reduce the effect of water contamination and may result in as much as a 16% increase in recyclable product.

The Japanese have reported that some 14-carbon labelled compounds can be removed from scintillation fluids by precipitation. If precipitation or other pre-processing could be effectively applied to our wastestream an additional 700 gallons of waste per year would be available for recycling.

Finally, the level of volume reduction, and perhaps the capacity to recycle, complex scintillation cocktails (Lefkofluor, Hydrofluor, and Ultarafluor) would be theoretically greatly enhanced through the application of reduced pressure distillation. Considering that these fluids comprise approximately one-third of the total volume of waste generated, any improvements in their management would have a significant impact in the efficiency of our program.

Unfortunately, It was beyond the scope of the current study to further explore the possible solutions to the identified technical problems. However, the preliminary findings suggest some exciting areas for future research.

DUKE UNIVERSITY MEDICAL CENTER
DUKE UNIVERSITY HOSPITAL

Hospital Epidemiology and
Environmental Safety Office

APPENDIX 1.

MEMORANDUM

TO: Administrative Directors

FROM: Dr. Wayne Thomann, Director Environmental Safety

SUBJECT: Laboratory Audit

DATE: May 21, 1985

Scintillating fluid wastes containing Carbon-14, Tritium, and Iodine-125 recently have been deregulated by the Nuclear Regulatory Commision. These deregulated wastes are candidates for conventional waste stream processing to reduce the volume of waste which must be landfilled. Duke Medical Center has received a grant from the State of North Carolina's Pollution Prevention Pays Program to study the feasibility of recycling scintillating fluids.

In accordance with this study, an audit of all laboratories using scintillating fluids within the University and Medical Center will be conducted. This audit will allow for a quantification of scintillating fluid wastes produced at the facilities. The feasibility study will examine the use of distillation techniques to recycle solvents and to reduce the volume of radioactive waste to be landfilled. Solvent recycling could reduce both the cost of scintillating fluids to the investigator through on-site repurchase and the cost of disposal through volume reduction. The implementation of a volume reduction and recycling program would be a significant step toward achieving an environmentally and fiscally responsible waste management program.

Investigator cooperation is vital to the success of the feasibility study. Your assistance in gaining this cooperation is greatly appreciated. If you have any questions, please contact me at 684-6320.

cc: Mr. Conrad Knight
Mr. Kevin Moore

Box 3914 • Durham, North Carolina 27710 • Telephone (919) 684-5457, 684-2794

DUKE UNIVERSITY MEDICAL CENTER
DUKE UNIVERSITY HOSPITAL

Hospital Epidemiology and
Environmental Safety Office

APPENDIX 2.

MEMORANDUM

TO: Users of Radioisotopes

FROM: Dr. Wayne R. Thomann, Director, Environmental Safety

SUBJECT: Scintillation Fluid Use

DATE: May 18, 1985

The Division of Environmental Safety is studying the use of scintillation fluid within Duke University and Medical Center. We are requesting your cooperation in identifying all scintillation fluid users.

Is scintillation fluid used in your laboratory? ( ) YES ( ) NO

Licensee ______________________________

Telephone Number ______________________

Please return through campus mail to: Box 3914, Environmental Safety

Box 3914 • Durham, North Carolina 27710 • Telephone (919) 684-5457, 684-2794

APPENDIX 3.

Form #__ of __

LABORATORY AUDIT FORM

Scintillation Fluids In Use

Building:________________ Room #:_______________

Principal Investigator:__________________________________

Lab Representative:______________________________________

Department:________________ Phone #:______________

Date of Audit:___________________

Audit performed by:___________________________

Is Scintillation Fluid in use in this lab?____________

What radioactive isotopes are used in this laboratory?

C-14:_____ Tritium:_____ I-125:_____

P-32:_____ S-35:_____ Other:__________________

What chemical compounds are tagged? _______________________

How are samples processed for counting?

____

____

____

____

Are you detecting the parent compound or metabolites?

parent compound:_____ metabolites:_____

List all known metabolites that are detected:

____

____

What other compounds/biological material may be present in your scintillation waste stream associated with your process?

____

____

What type of scintillation vials do you use?

plastic:________ glass:________

What volume of scintillation fluid per vial do you routinely use?

___________:ml

What is the approximate number of waste scintillation vials that you produce?

____

________:per day _________:per week _________:per month

How consistently do you produce that volume?

____

Have you considered using microvials for scintillation counting in the future?

_____Haven't considered it
_____Have considered it but have no future plans for using
_____Will try to implement the use by _________________

Is the Scintillation Fluid in use based on a commercial product? __________

What is the commercial product used?

Bioflour:____ Hydroflour:____

Ultraflour:____ Aquasol-2:____

Other:______________ Supplier:____________________

Are there any modifications made to the commercial fluids? __________

What are the modifications made by your lab to the scintillation fluids?

____

____

____

Is there any other information about the fluid which might prove to be useful?

____

Do you mix your own scintillation fluids?________________

What are the components by weight or volume of that fluid?

Component	Amount

Is this fluid recipe standard or is it modified as needed?

Standard:__________ Modified:__________

How is it modified? ______________________________________

____

How often is it modified? ________________________________

Are you planning on changing scintillating fluid in the future?

no:_____ yes:_____ to:______________________

Are you presently involved in a study in which you cannot change scintillating fluids?

no:_____ yes:_____ study ending date:____________

An in-house scintillating fluid recycling program is being considered for Duke University. This program would reduce the cost of scintillating fluid to the investigator, reduce the cost of disposal to the University, and help implement an environmentally sound waste program. If solvent purity and elimination of radioactivity could be guaranteed, would you be interested in participating?

no:_____ yes:_____ unsure:_____

Additional Notes:______________________________________

CONCLUSIONS

All scintillation fluids, except the complex or high boiling point mixtures, were redistilled to high purity parent solvents. High boiling point mixtures could not be purified for recycling; however, the volatile fractions were separated and could be disposed of as non-radioactive, high BTU fuel replacements for incineration. Reduced pressure distillation would increase the yield of such volume reduction efforts and may ultimately permit recycling of a pure distillate from the mixed cocktails.

When each isotope is evaluated separate from the scintillation fluid constraints, the following management profile can be proposed:

1) Tritiated wastes are the primary component of our wastestream and are immediate candidates for recycling. Radioactivity remains in the aqueous fractions of the waste and the distillate contains only background levels of activity. Dewatering of the tritium waste prior to distillation should result in an increased yield of distillate.

2) Scintillation fluid containing 14-carbon is not a candidate for recycling because radioactivity is carried-over into the distillate. It is possible that pretreatment of this wastestream would remove the radioactivity and make it a viable candidate for recycling.

3) The phosphorous isotope wastes comprise 28% of the processable wastes. Due to the relatively short half-life (14 days), these wastes could best be stored for decay prior to distillation. After storage (through 10 half-lives) the waste would be processed as non-radioactive scintillation fluid.

4) Sulfur containing waste is a small volume stream representing only 2.5% of all processable waste. This waste could be processed similarly to the phosphorous waste.

5) The I125 waste is small volume but can be inexpensively processed to remove the radioactivity. One possible volume reduction strategy would be the fixation of the radioactive iodine by phosphate/thiosulfate precipitation. Since this wastestream is primarily aqueous washes, the filtered water could be discharged into the sanitary sewer and the radioactive slurry could be decayed on-site as a method of volume reduction.

Summary - The results from the feasibility study are very encouraging. Approximately one-half to two-thirds of the scintillation wastes appear to be candidates for immediate recycling. The other segment of the wastestream could also be processed if preprocessing and reduced pressure distillation procedures can be developed. These technologies are clearly exciting prospects for needed future research.

Based on this preliminary study, we have estimated that an aggressive volume reduction/recycling program for scintillation fluid waste could result in an annual savings of $52,000 in disposal and repurchase costs. This estimate must be tested by upgrading the bench scale studies to a production level status, in order to evaluate the cost/benefit (including personnel requirements) for a full scale volume reduction/recycling program.

ACKNOWLEDGEMENTS

We are grateful to the B/R Instrument Corporation, P.O. Box 7, Pasadena, Maryland 21122, (301)647-2894, which generously supplied the 8400 solvent recycling system that was used in this study. A special thanks to Rubin Lovelady with Southeastern Scientific Corporation, (704) 542-3508, for technical support in initiating the distillation studies. Finally, we thank Ms. Emma Duffin for her clerical support.