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Radioactive Oil Decontamination Development – An Overview

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Abstract

Insulating, hydraulic and vacuum pump oils are used extensively in CANDU plants. It is inevitable that at some point in the equipment life cycle that these oils will become waste products and their disposal needs to be properly managed. The presence of radioactivity and conventional contamination in the oils (mixed waste) pose special challenges to the waste manager.

This paper provides an overview of waste oil streams that have been problematic in the CANDU nuclear industry and the decontamination processes that were developed for each to effectively remove a variety of radioactive species including tritium as well as conventional hazardous materials such as PCB, lead and cadmium.

1.0 Introduction

It is inevitable that oils used in nuclear power production become contaminated with process fluids and piping system corrosion products that may also be radioactive.

Currently there are about 2800 drums equivalent of waste oil stored in Low Level Storage building #5.

Insulating oils used in transformers in some of the older nuclear plants also contain PCB contamination. Good waste segregation practices are essential to keep such hazardous contaminants from mixing with radioactive waste oils. However in spite efforts to segregate problem waste streams, small volumes of such wastes have been generated.

The chemical and radiochemical characteristics of these waste oils make them difficult to dispose of and over the years the processing options for such wastes have been influenced greatly by the political climate. For example the thermal energy content of lubricating oils is sufficiently high to make incineration an attractive option for effective destruction of the waste oil and currently this is OPG’s plan when the new incinerator at the Western Waste Facility is fully operational. However a decade ago this was not the case and a significant amount of research effort was expended in looking at low temperature techniques for making the oils suitable for recycling, reuse or conventional disposal.

It is instructive to review the results of this work because the political climate may once again shift or the economics of recycling may make a more favourable business case in the near future.

2.0 Lubricating Oil

The primary source of this oil is from the Pickering Fueling machines which are hydraulically operated. The acceptance criteria for the storage of the oil at the Western Waste Management Facility (WWMF) are given in Table 1.

Table 1

PCB	<50ppm
Total Chlorine	<20000ppm
Lead	<5ppm
Cadmium	<0.5ppm
Flash Point	>61°C
Beta-gamma Activity	$<3.14\text{x}10^{-4}$Ci/L
Tritium	$<9.59\text{x}10^{-2}$Ci/L

The objective of the research work was to develop decontamination techniques to allow free release of the oil as non active ie:

Tritium concentration <2 microcuries/kg (74 Bq/g)
Beta gamma activity $<3.14 \times 10^{-4}$ microcuries/kg (1Bq/g)
Lead <5mg/kg
Cadmium<0.5mg/kg

Loop tests[1-6] involving low temperature filtration, centrifuging, acid washing, vacuum degassing and the use of sold absorbents were all tried with disappointing results:

- This work established conclusively that while bulk of the tritiated water in the oil was associated with dissolved and suspended water in the oil, a significant amount of the tritium is associated with oil oxidation products which become tritium labeled.
- It also confirmed earlier studies that the beta gamma radionuclides were not in particulate form in the oil but were associated with the polar additives in the oil.
- The soluble radionuclides were not able to be reduced to 3.7×10^{-7}microCuries/g, the limit at which the oil could be free released for conventional disposal.
- Lead and cadmium species present in the oil could be oxidized by acid washing and were found in the particulate sludge.

We also concluded that if the gamma activity was not associated with very fine particulate, low temperature techniques were unlikely to achieve a decontamination factor of >100 in a practical time frame since the absorption kinetics that these processes rely on are slow. To achieve a 100 fold improvement, more aggressive measures are required.

In the ASTM D 2440 accelerated aging test, oil is aged at high temperature under flowing oxygen in the presence of a solid copper catalyst, and the production of sludge and acid products is monitored periodically by a gravimetric method and titration respectively. In this test, unstable or poor quality oils will show evidence of significant oxidative degradation in as little as 24 hours. Given that the radioactivity (except tritium) was associated with the oil additives and oxidation products this test formed the basis for converting the contamination into a form that could be easily separated from the oil.

A peroxide based initiator, and a copper catalyst was used. The degree of sludging at ~200°C in the presence of oxygen was used as a measure of successful oxidation, with a target minimum being 0.25 % on a weight/weight basis. The results of the three active runs are summarized in Table 2 in terms of the remaining activity in the oil portion. Table 3 shows the corresponding quantities of the radionuclides filtered out with the filterable sludge. The activity distribution of the major radionuclides in the adherent sludge component, corrected for residual losses to the flasks, is shown in Table 4. It is noted that the process effectively reduced the radionuclide levels in all samples.

TABLE 2
CONCENTRATION OF RADIONUCLIDES IN DECONTAMINATED OIL

		RUN 1	RUN 2	RUN 3
	Flow Rate Temperature Duration Wt. of oil	1L/h O_2 185-200°C 3h 732g	1L/h air 185°C 6h 780g	1.6L/h O_2 200°C 3h 886g
NUCLIDES	BEFORE (μCi/g)	AFTER (μCi/g)	AFTER (μCi/g)	AFTER (μCi/g)
H-3	0.027	0.004	0.003	0.003
Co-60	(1.3±0.1)E-5	(1.8±0.7)E-8	(1.2±0.3)E-7	(3.0±1)E-8
Ru-106	Not detected (n.d)	n.d	n.d	n.d
Sb-124	n.d	n.d	<6.9E-8	<6.8E-8
Sb-125	(3.5±1.0)E-7	n.d	<8.2E-8	<6.7E-8
Cs-134	(2.1±0.1)E-7	(3.1±0.7)E-8	<3.1E-8	<1.5E-8
Cs-137	(3.4±0.3)E-6	(4.9±0.4)E-7	<3.2E-8	<1.5E-8
Ce-144	(5.0±2.1)E-7	n.d	n.d	n.d
Eu-152	(2.8±1.0)E-7	n.d	n.d	n.d
Eu-154	n.d	n.d	n.d	n.d
Eu-155	n.d	n.d	n.d	n.d
Am-241	n.d	n.d	n.d	n.d
TOTAL(γ)	1.77E-5	5.4E-7	<3.3E-7	<1.9E-7

TABLE 3

QUANTITY AND COMPOSITION OF ACTIVITY (uCi) IN FILTERED, LOOSE SLUDGE

	RUN 1	RUN 2	RUN 3
WT. OF SLUDGE (g)	4.6	1.5	6.1
SLUDGE AS A PERCENT OF OIL TREATED	0.63	0.19	0.69
Co-60	(7.7±0.1)E-3	(9.2±0.1)E-3	(8.5±0.1)E-3
Ru-106	(8.6±5.0)E-5	n.d	(1.6±0.7)E-4
Sb-124	<1.6E-5	(1.0±0.1)E-5	<1.4E-5
Sb-125	(1.7±0.1)E-4	(1.6±0.1)E-4	(2.0±0.2)E-4
Cs-134	(1.0±0.1)E-4	(1.1±0.1)E-4	(1.2±0.1)E-4
Cs-137	(1.3±0.02)E-3	(1.4±0.02)E-3	(1.6±0.02)E-3
Ce-144	(4.0±0.2)E-4	(4.6±0.1)E-4	(4.5±0.2)E-4
Eu-152	(1.6±0.2)E-4	(1.8±0.1)E-4	(1.8±0.2)E-4
Eu-154	(8.9±1.4)E-5	(2.0±0.1E-4	(9.7±2.0)E-5
Eu-155	n.d	(5.8±0.6)E-5	n.d
Am-241	(3.4±1.0)E-5	(4.5±1.0)E-5	n.d
TOTAL	1.0E-3	1.2E-3	1.1E-03

The majority of the Co-60 radioactivity is found in the filtered sludge. It should be noted that the activity data for the catalyst and reaction vessel is approximate because there is no standard geometry for these items for an accurate calibration to be carried out.

TABLE 4

DISTRIBUTION OF Co-60 (μCi) REMOVED FROM OIL

	RUN 1	RUN 2	RUN 3
AMOUNT REMOVED FROM OIL	9.5E-3	1.0E-2	1.1E-2
AMOUNT REMOVED FROM LOOSE SLUDGE	7.7E-3 (81%)	9.2E-3 (92%)	8.5E-3 (77%)
AMOUNT REMOVED FROM ADHERED SLUDGE ON CATALYST	1.6E-5 (0.2%)	3.5E-5 (0.3%)	7.5E-5 (0.7%)
AMOUNT REMOVED FROM ADHERED SLUDGE ON REACTION VESSEL	2.6E-3 (27%)	8.5E-4 (8%)	not measured

The lead concentration in the oil was reduced from 6.7mg/kg to the detection limit (<0.12mg/kg). The cadmium concentration in the untreated oil was already below the detection level (0.2mg/kg).

Pilot scale testing with two 100L batches of oil confirmed that the chemical oxidation process can effectively remove both radioactive contaminants as well as lead and cadmium.

Some of the contaminants appear to be removed more slowly than others suggesting that a real time sampling and analysis method would be required to determine when the oil can be considered to be clean. These pilot runs indicate that a minimum processing time of 12 hours per batch will most likely be required.

The pilot plant design needs to consider the water content (suspended and dissolved) in the oil to ensure effective tritium removal.

The end product of the oxidation is a very dark oil with high acidity (1.6 mg KOH/g oil). Treating the oil with activated alumina to remove the organic acids (to <0.05mg KOH/g) indicated that it is possible to reduce the acid number of the decontaminated oil but this requires a significant cost in activated alumina or similar absorbent. The economics of using the decontaminated oil as feedstock for standard, off-site re-refining may be more favourable.

2.1 SUMMARY

(1) Controlled oxidation in the presence of copper catalyst at 200°C can reduce the gamma activity in waste oil by 2 orders of magnitude to the detection limit ($<2\times10^{-7}\mu$Ci/g). The tritium concentration was reduced in this work to 3μCi/kg but this level can be further reduced by pre-drying the oil.

(2) All of the gamma activity is retained in the filtered sludge. Water is a byproduct of the oxidation and when condensed will contain the bulk of the tritium contamination from the oil as well as some of the volatile light ends. This represents a very small volume of secondary waste that can easily be managed. It is estimated that the volume reduction factor achieved by this process is of the order of 100.

(3) Pure oxygen is more effective than air at a 1L/h flow rate under the test conditions and results in a reduced processing time.

(4) Lead contamination is very effectively removed in this process.

3.0 PCB and Radioactive Contaminated Lubricating Oil

The patented[7] waste oil decontamination process described in the previous section removes heavy metals (Pb/Cd) from oil as well as beta/gamma radioactive contamination but is ineffective in destroying PCBs. Small quantities of this type of oil were also identified in storage at one of the generating stations.

Following the decontamination process described in the previous section the oil can be classified as conventional chemical waste, thereby allowing the oil to be either incinerated as waste derived fuel or recycled through a refining process.

TABLE 5
Chemical Characteristics of Oil Following Decontamination

CONTAMINANT	Before	After	Decon Factor
Total Chlorine (mg/kg)	3480	58	60
Lead (mg/kg)	21	<0.12	168
Cadmium (mg/kg)	0.12	<0.12	>1
PCB (mg/kg)	8	7	1

The criterion for waste derived fuel however requires that the PCB contamination in the oil be less than 2ppm, which could not be achieved with this process. PCBs are very resistant to low temperature oxidation hence their former use as insulating fluids in transformers. To destroy them thermally requires very high temperatures in excess of 1200°C.

A PCB destruction process based on elemental sodium reaction with PCB in insulating oil has been extensively used[8] on a commercial scale by Ontario Power Generation to decontaminate transformer oils. Sodium metal is a very powerful reducing agent and is known to be an effective dechlorination agent. The sodium in turn forms sodium chloride, which can be mechanically separated from the oil. The reaction can be represented by equation 1.

$$2RCl + 2Na \longrightarrow R\text{-}R + 2NaCl \qquad \text{(Equation 1)}$$

It was assumed that in order to produce waste derived fuel from radioactive contaminated waste oils containing PCB contamination would require a two step process whereby radioactivity and heavy metals would be removed in the first step and PCB would be destroyed in the second step. A major disadvantage of this scheme is that a significant degree of oil oxidation takes place in the first step and this leads to a much greater amount of sodium required to destroy the PCBs in the second step.

A literature review of the mechanism of the two processes revealed that it is very likely that both the catalytic destruction of the antioxidants in lubricating oils and the dechlorination of hydrocarbons both proceed via radical intermediates. An obvious question was whether the sodium dechlorination reaction would also destroy the antioxidant in lubricating oil, which is associated with the radioactive and heavy metal contaminant. This would lead to a one step process for both radioactive and heavy metal decontamination as well as PCB destruction. Furthermore sodium consumption would be less than in a two step process.

Several laboratory tests were conducted and the results summarized in Table 6. As noted the second exposure (required due to insufficient sodium after the first exposure) resulted in a further reduction of the gamma emitting radionuclides by a factor of 10 and reduced the PCB content to below the 2 mg/kg detection limit. It is interesting to note that the PCB concentration was reduced by a factor of only 2 after the first exposure showing that the dechlorination reaction is slower than the reaction of sodium with the polar additives in the oil which carry the radioactivity and the heavy metals.

As expected there was essentially negligible removal of tritium in the oil. Past experience indicates that tritium does not become incorporated into the insoluble species formed in these reactions.

TABLE 6
Chemical/Radiochemical Characteristics of Oil
Following Reaction with Sodium.

CONTAMINANT	Before (uCi/g)	After (uCi/g) Exposure 1	After (uCi/g) Exposure 2	Decon. Factor
Co-60 ($t_{1/2}$ 5.3y)	2.97E-05	7.28E-06	6.95E-07	42
Sb-125 ($t_{1/2}$ 2.7y)	3.6E-06	7.00E-07	not detectable	
Cs-137 ($t_{1/2}$ 30y)	3.7E-06	2.70E-07	6.76E-08	55
Ce-144 ($t_{1/2}$ 285d)	1.20E-06	3.90E-07	not detectable	
Eu-152 ($t_{1/2}$ 13y)	1.50E-06	8.00E-07	not detectable	
Eu-154 ($t_{1/2}$ 16y)	9.00E-07	4.0E-07	1.99E-07	4.5
TOTAL Beta/Gamma	4.09E-05	1.03E-05	0.97E-06	42
Tritium (uCi/kg)	37	27	24	1.5
Lead (mg/kg)	12.9	1.32	0.4	32
Cadmium (mg/kg)	<0.2	<0.125	not measured	
PCB (mg/kg)	12	6	<2	

3.1 SUMMARY

(1) The sodium treatment method is effective for both PCB and radionuclide removal from active waste oils. Sodium dispersion (40% w/w) consumption for effective treatment is estimated at between 1 and 2% by weight of waste oil, for oils containing oxidized oil components in the typical 0.5% by weight concentration range.

(2) Based on the test results and previous experience with PCB destruction in multiple and single reactions, a reaction temperature in the range 130-150°C and a single reaction period of up to 5 hours appears to be sufficient for both PCB dechlorination and activity reduction. This assumes that a 2% sodium charge is used.

(3) As expected, tritium is not affected significantly by sodium treatment and would have to be removed from the oil matrix by other methods such as vacuum degassing at slightly elevated temperature.

4.0 PCB and Radioactive Contaminated Lubricating Oil Heavily Emulsified with Tritiated Heavy Water

A third class of waste oil whose disposal has been problematic is similar to the previous type. The added complication was that the oil was heavily emulsified with tritiated heavy water to a degree that made heavy water recovery desirable. In fact application of the previous technique would have necessitated the application of a water removal step prior to PCB destruction and radioactivity decontamination by virtue of the amount of water in the oil. Fortunately there was only 600 liters of this type of oil.

Chemical and radiochemical characteristics of the drummed liquid are shown in Table 7.

Table 7
Characteristics of Radioactive/PCB Contaminated Liquid Waste

DRUM CONTENTS	VOLUME (L)	PCB CONC. (ppm)	Co-60 (nCi/kg)	Cs-137 (nCi/kg)	Cs-134 (nCi/kg)	Sb-125 (nCi/kg)	TRITIUM (µCi/kg)
Water Phase	100	1.66	93	271	6.6	7.7	180,000
Oil Phase	100	130	12.4	6.1	0.56	-	175
Oil Phase	40	260	0.05	0.05	-		34
Oil Phase	60	94	12.5	9.6	0.32	0.97	171
Water Phase	90	42 ppb	120.5	451	10.5	9.2	170,000
Water – 90%	180	650,000	n.d	1.9	n.d	n.d	14,700
Low level oil – 10 %	20	51	0.06	0.54	n.d	n.d	1,330

The preferred disposal option for this small volume low activity waste is to send it to a conventional PCB destruction facility in Canada. Implementation of this option however required that the generator complete a pathways analysis to show that the most critical individual handling the waste is not exposed to a risk in excess of the CNSC R104 limits.

Derived clearance levels are levels at which the risk based R104 limits are adhered to. For the proposed hazardous waste treatment facility, the pathways analysis showed that the derived clearance levels for all of the radionuclides associated with the oil phase were above the measured radionuclide concentrations in the oil. However, transportation regulations are very much more stringent and under these regulations the tritium levels in the oil are close to 100 times higher than acceptable (2μC/kg).

Since tritium is the only radionuclide above the transport regulation levels, the path forward for the disposal of this waste stream was to process the oil to only reduce its tritium concentration to <2μCi/kg and transfer to Swan Hills for destruction.

4.1 FORMS OF TRITIUM IN OIL

Hydraulic and lubricating oils consist of a mixture of saturated and unsaturated hydrocarbons derived from paraffinic distillate and additives. The purpose of the additives is to increase the oxidation resistance of the oil and provide corrosion inhibition to the components being lubricated. These additives are proprietary mixtures of phenolic hydrocarbons and can be described simplistically as having a molecular formula of $[Ar\text{-}CH_2]\text{-}OH$. Over a period of time the additives will degrade and become ineffective. The base oil then oxidizes to form alcohols, aldehydes and acidic species.

When these degradation reactions take place in the presence of tritiated water, complex mixtures of tritiated hydrocarbons can result. The tritium atom can be bonded to an oxygen atom on the alcohol, aldehyde or acid functional group and be easily exchanged with hydrogen in water. The tritium can also form strong carbon tritium bonds by incorporation through a series of radical intermediates.

Four general types of tritiated species can therefore exist in oil exposed to tritiated water.

(1) Tritiated water (HTO) dissolved or suspended as an emulsion,
(2) Tritiated polar hydrocarbons with tritium on exchangeable sites e.g. $[R\text{-}CH_2]_n\text{-}OT$, $[R\text{-}CH_2]_n\text{-}COOT$,
(3) Tritiated polar hydrocarbon species with tritium on non-exchangeable sites e.g. $[R\text{-}CHT]_n\text{-}OH$, $[R\text{-}CHT]_n\text{-}COOH$,
(4) Tritiated non-polar hydrocarbon species with tritium on non-exchangeable sites e.g. $[R\text{-}CH_T]_n\text{-}H$.

An effective tritium decontamination process must address the removal of all four of the species. In previous work we have shown that unless the tritium is in the elemental form the concentration of tritiated species in category (4) is negligible.

A laboratory scale treatability study confirmed that:

(a) the predominant tritiated species in oil was tritiated water removable by vacuum degassing,
(b) 10% of the total tritium (12μCi/kg) was not volatile,
(c) a polar hydrocarbon absorbent such as Florisil or activated alumina can remove this residual tritium
(d) less than 0.1% of the total tritium is associated with non polar hydrocarbons that will not be removable by a combination of degassing and polar hydrocarbon absorption.

4.2 OIL PROCESSING

4.2.1 Vacuum Degassing

A small (200L/h) mini dehydrator was procured for this job and the four drums of PCB contaminated radioactive oil were transported to Kinectrics' laboratory for processing. Florisil was replaced by activated alumina because it was more readily available and much less expensive even after the reduced capacity of activated alumina is taken into account. The mass of alumina used in the absorption cartridge was approximately 4.5kg sufficient to process 30kg of oil.

The equipment is shown in Figure 1. It consists of a polyethylene processing tank of 132L capacity, the degasser in the right foreground and the housing which contains the activated alumina cartridge.

From the relative composition of each phase it was determined that the total volume of oil requiring processing in the four waste drums was 330L. A drum pump with its inlet positioned such that only the oil phase would be transferred to the processing tank was used. Approximately 100L of oil was transferred over and the system placed in the recirculation mode at 50°C. A higher temperature would make the degassing more efficient due to the higher vapour pressure of water but it was found that the condensate entrained more oil at 80°C which required additional separation. Tritium in the oil was determined periodically.

The profile for the reduction of tritium in the oil for two distinct batches is shown in Figure 2. The first batch of oil was tan coloured highly turbid and contained a large quantity of both suspended and emulsified water because it was taken from the oil water interface of one of the drums to ensure all of the oil was removed. The presence of the suspended tritiated water carried over explains why the tritium concentration was initially so high. In fact, as the figure also shows, the tritium concentration increased further as more suspended water was emulsified in the system as a result of recirculating through the degasser. As long as there was free water held up in the system the tritium concentration tended to fluctuate but there was a decreasing trend over about 150 hours and the tritium levelled off at 13.5μCi/kg when further vacuuming was not beneficial. In the second batch of oil processed, the tritium concentration levelled off at 9.4μCi/kg. This confirmed the conclusions of the treatability study that between 9-12μCi/kg could be attributed to tritium residing on non-volatile, non-exchangeable sites since the addition of water and its removal by degassing did not further reduce the tritium concentration in the oil.

Figure 1
Oil Degassing Unit and Activated Alumina Cartridge

Figure 2
Effect of Vacuum Degassing on the Tritium Concentration in Oil

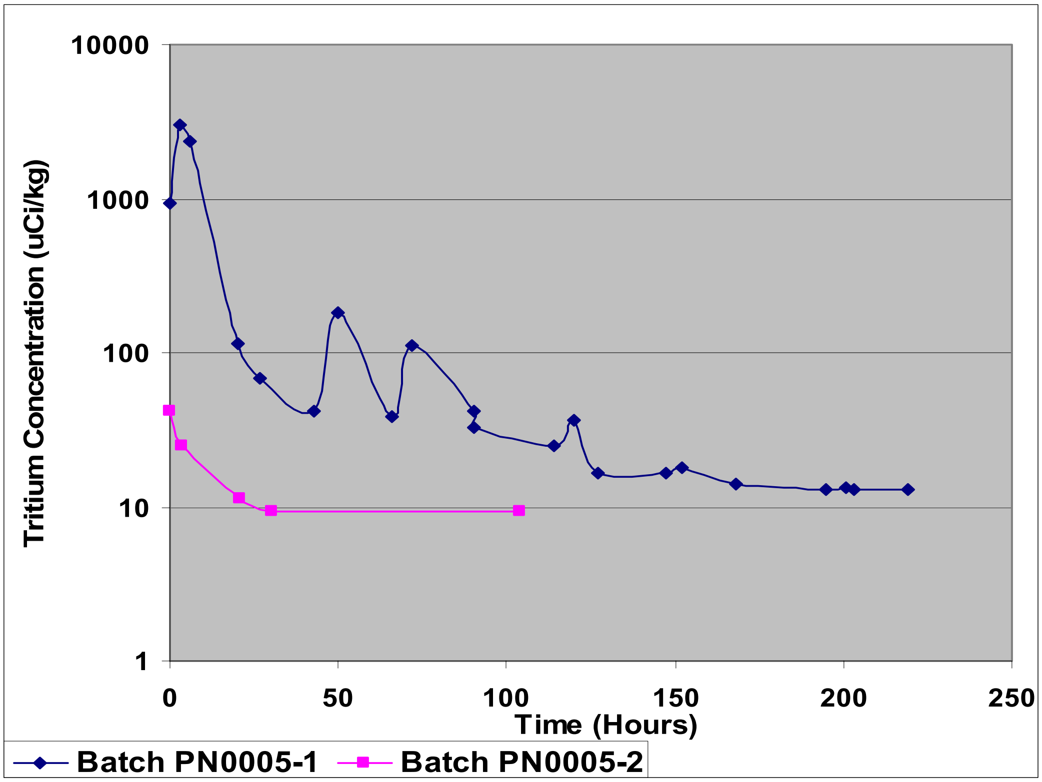
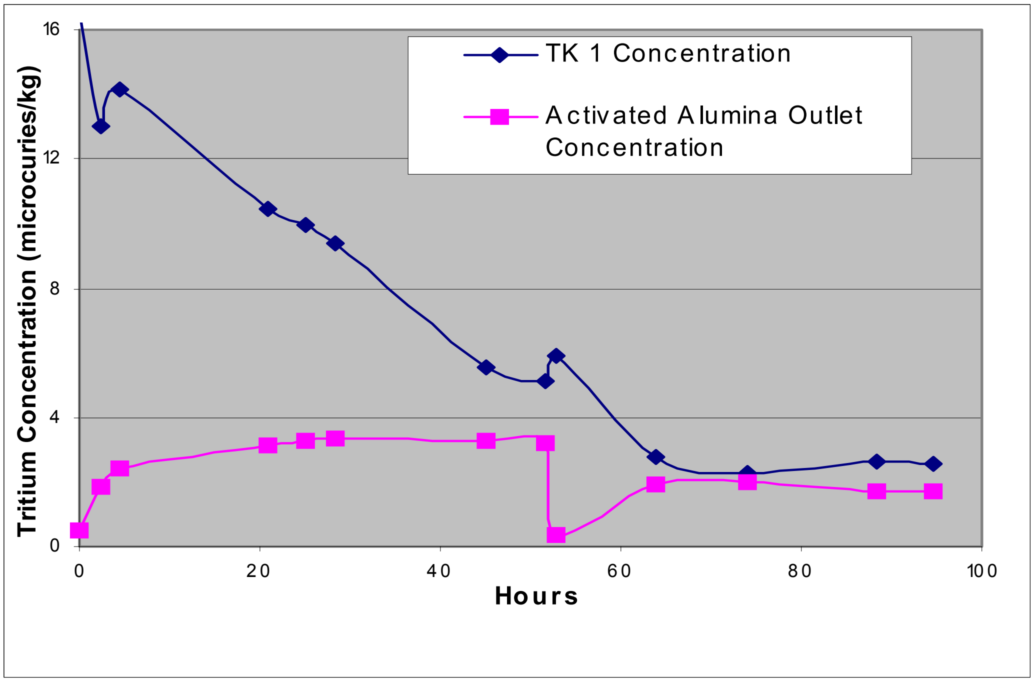


Figure 3
Polar Hydrocarbon Absorption on Activated Alumina



4.2.2 Polar Hydrocarbon Absorption

The tritiated oil at 50°C containing between 9.4-13.5μCi/kg was then diverted through the activated alumina bed at a flow rate of 30-50mL/min. This was a factor of 100 higher than in the treatability study. The bulk of the flow (3L/min) was recirculated back to the process tank.

The tritium profile (Figure 3) shows that at this temperature and flow rate, there are tritiated hydrocarbon species that pass through the activated alumina. The first fraction of effluent was very light brown in colour and progressively darkened as more of the oil breakdown products passed through the alumina. However, as the Figure shows, the column continues to pull tritium even though the tritium concentration in the effluent is >2μCi/kg. The first 10L collected from the activated alumina bed effluent had a tritium content of approximately 1.5μCi/kg. After the 10L of oil was collected the effluent from the absorption cartridge was routed back to the process tank and left in this mode until the effluent and the tank tritium concentration were equal indicating exhaustion of the absorption column.

4.2.3 Oil Processing Summary

(1) Vacuum Degassing was effective in reducing the tritium in the oil to 9-14μCi/kg. Further degassing was unable to reduce the tritium concentration. A tritium decontamination factor of 250 was obtained across the degasser.

(2) A further tritium decontamination factor of 8 was obtained with activated alumina absorption.

(3) Activated Alumina absorption was effective but the kinetics was slow at ambient temperature.

(4) Decontamination was selective for tritium.

(5) Processing is slow but equipment is highly reliable and can be left unattended.

(6) Tritiated heavy water was returned for upgrading and the oil containing PCB contamination was sent to Swan Hills for destruction.

CONCLUSIONS

The behaviour of radioactive contaminants in waste oils has been elucidated in the work presented in this paper. This has resulted in the development of a number of low temperature unit operations allowing custom decontamination solutions for a variety of waste oils.

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