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TRIFLUOROETHANOL

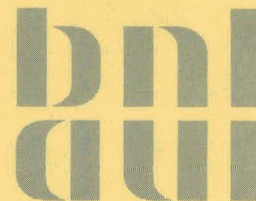
SAMUEL SILBERSTEIN

October 1978

BIOMEDICAL AND ENVIRONMENTAL ASSESSMENT DIVISION
NATIONAL CENTER FOR ANALYSIS OF ENERGY SYSTEMS
BROOKHAVEN NATIONAL LABORATORY
ASSOCIATED UNIVERSITIES, INC.
UPTON, NEW YORK 11973

UNDER CONTRACT NO. EY-76-C-02-0016 WITH THE

DIVISION OF TECHNOLOGY OVERVIEW/ENVIRONMENT
UNITED STATES DEPARTMENT OF ENERGY



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BNL 50964
UC-41
(Health & Safety - TID-4500)

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Printed in the United States of America
Available from
National Technical Information Service
U.S. Department of Commerce
5285 Port Royal Road
Springfield, VA 22161
Price: Printed Copy \$4.00; Microfiche \$3.00

ABSTRACT

Fluorinol-50 and -85 (containing 50 and 85 mole % trifluoroethanol (TFE), respectively) are candidate working fluids for truck and power plant Diesel bottoming cycles, respectively. TFE is self-extinguishing and chemically stable at its proposed operating temperature. Toxic effects were not seen in production workers at Halocarbon Products Corp., the manufacturer, but are apparent in animal studies.

The most obvious effects upon acute or chronic inhalation exposure are intoxication, narcosis, and death, but there is wide disagreement on concentrations producing nervous system effects. Ethanol is an antidote. Sterility and impaired spermatogenesis were first noted at 50 ppm in male rats. These changes may be reversible. Exposure of female rats to 10 ppm produced enlarged ovaries. Functional tests of reproductive ability were not done, but they should be, since the above concentrations are not much higher than the recommended threshold limit value, 2.5 ppm.

Skin contact with TFE can cause nervous system effects and even death; eye contact produces corneal opacity. Because of TFE's ability to penetrate intact skin, the Toxic Materials Advisory Committee of the U.S. Department of Energy strongly recommended that a less toxic working fluid be sought. We fully concur.

It is unknown whether TFE will add to the danger of truck accidents, but leakage of the small amount of TFE used probably will not contaminate drinking water.

ACKNOWLEDGMENT

This work was supported by Contract No. EY-76-C-02-0016 with OTI/Division of Technology Assessments/ASEV, U.S. Department of Energy. I am grateful to S. Kramer for invaluable materials not readily available elsewhere and to him as well as P. Ajmeri, S.C. Morris, and D.A. Blake for useful information and discussions.

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1. Schematic of the Thermo Electron Corporation Organic Rankine Bottoming Cycle 1

TRIFLUOROETHANOL

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TRIFLUOROETHANOL

Bottoming cycles are designed to conserve energy by utilizing usually discarded exhaust heat from generators or engines. For example, Thermo Electron Corporation manufactures Rankine bottoming cycle^{1,2} that use Diesel engine waste heat to heat and pressurize working fluid (Fluorinol-85 for power plants and Fluorinol-50 for trucks), which is then permitted to expand and cool through a turbine, doing useful work (Fig. 1). The cycle is repeated after further cooling. Fluorinol-85 and -50 are the trade names for 85 and 50 mole % trifluoroethanol (TFE, $\text{CF}_3\text{CH}_2\text{OH}$) manufactured by Halocarbon Products Corporation.³ It is estimated that Rankine bottoming cycles can raise the efficiency of Colt-Pielstick PC-2 12-cylinder Diesel electricity generators by 24%⁴ and improve truck Diesel engine efficiency by 15%.^{1,5} Although a Rankine cycle is a sealed system, leaks and accidents can occur, exposing employees and the general public to working fluid and to decomposition and combustion products such as carbonyl fluoride (COF_2). It has previously been found that organic working fluids can be quite hazardous.^{5,6} The present report shows that Fluorinols are no exception. First the toxicity of TFE is evaluated, and then special hazards arising from its use in trucks are examined.

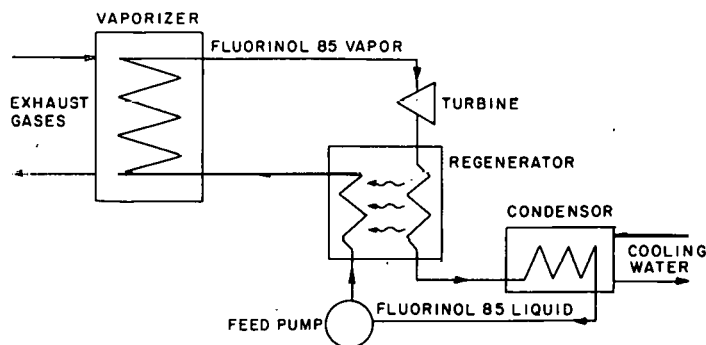


Figure 1. Schematic of the thermo electron corporation organic rankine bottoming cycle (500 kW_e) (from ref. 1)

TRIFLUOROETHANOL TOXICITY

TFE is a volatile, colorless liquid of density 1.382 kg/L at room temperature, miscible with water and alcohols. Its acidity is comparable with that of phenol and higher than that of ethanol. It can burn only when supported by an external fuel source and is self-extinguishing.⁴ It is chemically stable: various Fluorinol-water mixtures were heated to 316°C for 1700 to 2350 hr in the presence of carbon steel, cast iron, or steel A. Decomposition was < 1% although the metals turned black and the liquid sometimes became yellow,⁸ possibly because of TFE's acidity. Under normal operating conditions, working fluid is heated to 290°C by Diesel exhaust whose temperature is 360°C. Whether extensive decomposition occurs above 316°C is unknown, but Halocarbon Products Corp. intends to investigate this.⁹ Maximum recommended operating temperature is 329°C for both Fluorinol-85 and Fluorinol-50.³

TFE has been manufactured by Halocarbon Products Corp. since 1960.⁷ They claim to have seen no toxic effects in production workers, who also produce fluorocarbon anesthetics. Therefore, the U.S. Department of Energy Toxic Materials Advisory Committee¹⁰ used only toxic effects on animals to determine its recommended threshold limit value: a time-weighted airborne concentration average of 2.5 ppm. The toxic effects include intoxication, respiratory irritation, and death,^{11,12} and the airborne levels producing them are controversial as shown below but are well above 2.5 ppm. Chronic exposure to 50 ppm can produce sterility.¹³ TFE can be absorbed through intact skin,^{12,14} and 1 ml/kg entering the body this way can cause death.¹⁴ Ethanol can be used as an antidote. Practically all ingested TFE is excreted in urine as glucuronide conjugate and $\text{CF}_3\text{CO}_2\text{H}$ (trifluoroacetic acid), in mice, dogs,^{15,16} and humans.¹⁷

The material examined below comprises mainly results of inhalation and skin and eye contact experiments conducted by Hazleton Laboratories America,

Inc., 11,13,14,18, and by Nikitenko and by Nikitenko and Tolgskaya¹² in Russia.
Table 1 summarizes animal studies on TFE.

Table 1
Summary of Toxicological Studies on Trifluorethanol²⁰
(Prepared by David A. Blake, Ph.D., 9/12/76)

Type	Duration	Level	(Sex)	Observations	Ref.
Chronic inhal.	4 mo. ? hr/day	30 ppm	Rats, Guinea pigs (?)	Retarded wt. gain (2 mo) ↑N-M irritability (4 mo) ↓RBC, Hbg, benzoate Cl Above reversing 1 mo post Pos. histopath.-URT, liver(?) ↑Body wt. gain	12
Subacute inhal.	6 hr/day 5 days/wk 4 wk	15 ppm (2.5-25)	Rats(M,F)	No deaths, no effect on wt. gain	13
		0,10,50, & 150 ppm		Neg. histopath. (26 tissues) Testicular depress. at 50 ppm (reversible) and 150 ppm (persistent, 5 wk)	18
	4 hr/day 4 wk	0,86 ppm	Rats (?), Guinea pigs (?)	Pronounced intoxication Wt. loss Deaths (4/10 rats, 2/6 GP) ↓RBC, Hbg, benzoate Cl Pos. histopath. lungs, liver, kidney, brain	12
Acute inhal.	6 hr	350-2000 ppm	Rats(M,F)	LC ₅₀ = 550 ppm Narcosis Latency to death (1 day)	11
	2 hr	?	Mice (?)	LC ₅₀ = 710 ppm Narcosis Pos. histopath.-brain	12
	4 hr	24-80 ppm	Rats (?)	Pos. for N-M irritability, O ₂ req., resp. rate, organ wt. coeff.	
Acute oral	1 dose		Rats (M)	LD ₅₀ = 240 mg/kg	11
			Mice (M)	LD ₅₀ = 365 mg/kg Latency to death (5+ hr)	16
Acute I.P.	1 dose		Mice (M)	LD ₅₀ = 350 mg/kg Latency to death (7+ hr)	15
			Mice (M)	LD ₅₀ = 195 mg/kg	21
Acute I.V.	1 dose	400 mg/kg	Dogs (F)	Lethal, 20-24 hr Hypervent., hypertherm. Neg. histopath.	15
				Norm. citrate, lactate	
Acute I.G.	1 dose		Rats	LD ₅₀ = 590 mg/kg	12
Acute Dermal	2 hr	tail(2/3) dip	Mice	Lethal to 80% Local inflamm., necrosis	12
	24 hr	0.1-3.2 ml/kg Abdominal +/- abrasion occluded	Rabbits (M,F)	LD ₅₀ = 1.2 ml/kg = 1.7 cm/kg Non-irritant	14
Acute Eye	1 dose Irrigated & non- irrigated	0.1 ml	Rabbits (M,F)	Severe irritation Corneal opacity Iritis, conjunctivitis Slightly reduced by irrigation	19

Animal Studies on TFE

Hazleton Labs. tested TFE manufactured by Halocarbon Products Corp. and give details of experimental conditions such as doses, animal strains, airborne concentrations, and feeding methods. Nikitenko and Tolgskaya often omit vital information such as the source and purity of the TFE used.

Inhalation Exposure

In the Hazleton Labs. acute inhalation study,¹¹ rats were subjected to various airborne concentrations of TFE for 6 hr, observed for 14 days, and sacrificed. The LC_{50} (concentration lethal to half the animals) was in the range 470 to 640 ppm. Nikitenko and Tolgskaya obtained LC_{50} in the range 560 to 830 ppm for a 2-hr exposure of mice (strain and observation time after exposure unspecified). Although conditions and animals differ, these two values of LC_{50} are close.

Hazleton Labs. found the main physiological effects to be piloerection, humped position, bloody nasal discharge, ataxia, depression, decreased activity, apparent eye irritation, and slight tremors. These effects were noted at the lowest dose tested (350 ppm) and increased in severity with concentration. Nikitenko and Tolgskaya also found symptoms characteristic of narcosis, with death, when it occurred, caused by hyperemia, hemorrhages, and edema in the brain. They found the threshold dose for neuromuscular irritation, oxygen requirement, respiration rate, and internal organ weight coefficient changes for rats (strain unspecified) in a 4-hr exposure to be 24 to 81 ppm.

Both Hazleton Labs.¹³ and the Russians performed similar chronic inhalation studies with rats exposed to various concentrations of TFE 5 days/wk for 4 wk; the daily exposure was to 0, 10, 50 and 150 ppm for 6 hr in the former study and to 0 and 86 ppm for 4-hr in the latter (which also included guinea pigs).

Hazleton Labs. was interested mainly in effects on reproductive function. They observed no deaths during or after exposure and no differences in appearance or behavior among any of the four groups of animals.

After 2 and 4 weeks some of the males were sacrificed. Those exposed to 150 ppm of TFE for either period had lowered mean testis weights and testis wt./body wt. ratios; after 4 weeks rats exposed to 50 ppm also had decreased testis weights. Although there was no statistically significant decrease in body weight for any group of males after 4 weeks, subgroups exposed to 50 and 150 ppm of TFE were significantly lighter than a control subgroup, which indicated that weight loss for the highly exposed groups was of borderline significance.

All females were sacrificed after 4 weeks. Non-control females had elevated ovary weights relative to controls, but the difference was significant only for the group exposed to 10 ppm of TFE. The ovary wt./body wt. ratio increased by similar amounts for all non-control groups, but the increase for rats exposed to 150 ppm was not significant although it was nearly as high as for the other two non-control groups. Thus the gain in ovary weight due to TFE appears to be of borderline statistical significance. It occurs rather sharply between 0 and 10 ppm and levels off for higher concentrations. Histology revealed no differences in ovary appearance attributable to TFE.

Among males sacrificed after both 2 and 4 weeks, those exposed to 50 ppm showed impairment of spermatogenesis and those exposed to 150 ppm showed hypospermatogenesis. Other tissues (26 types in all) from both males and females did not appear different from those of controls.¹⁸

In functional tests, the remaining males¹³ were allowed to recover from TFE exposure for about 10 days, and then each was supplied with a different

unexposed female each week for 5 wk, and conception rates were measured. For the group exposed to 10 ppm, the rate was the same as for controls; for the group exposed to 50 ppm, it was 72% that of controls; and for the group exposed to 150 ppm, no conception occurred. For the group exposed to 50 ppm, conception rates were lower than for controls during the first 3 wk, reaching a minimum during the second week, and preimplantation losses followed an analogous pattern. Animals were sacrificed 2 wk later (for a total recovery period of 57 days) and their testes examined. Males exposed to 50 ppm showed normal spermatogenesis. Those exposed to 150 ppm also showed some recovery and some normal spermatogenesis.

Nikitenko and Tolgskaya did not measure reproductive function. The effects they noted at 86 ppm were markedly different from those found by Hazleton Labs. for any concentration: pronounced intoxication, death of 4 of 10 rats and 2 of 6 guinea pigs after acute weight loss, decrease in hemoglobin and erythrocytes and in urine hippuric acid, and pathological tissue changes.

Contact Exposure

Hazleton Labs.¹⁴ applied 100, 316, 1000, and 3160 μ l/kg of TFE to the abdomens of rabbits, covered the abdomens with gauze for 24 hr, observed the rabbits for 14 days, and sacrificed the survivors for gross examination. No controls were used. Two of 4 rabbits exposed to 1000 μ l/kg and 3 of 4 exposed to 3160 μ l/kg died. Toxic effects and necropsy findings attributable to TFE were observed only for these two groups. Depression was noted in highly exposed animals. Two survivors of high exposures still had slight tremors at the end of observation but the third survivor recovered completely. Animals that died showed congestion and hemorrhage in the lungs. The LD₅₀ (dose lethal to half

the animals) was estimated to be in the range 390 to 3800 $\mu\text{l/kg}$. No dermal effects were seen except for slight erythema at low dose levels.

Nikitenko and Tolgskaya applied TFE to tails of white mice, causing a mortality rate of 80% (total number of animals unspecified).

Hazleton Labs.¹⁹ applied 0.1 ml of TFE into the conjunctival sac of one eye of each of several rabbits. Some of these eyes were irrigated with water 2 or 4 sec later. Animals were observed for 14 days. There was severe eye irritation and corneal opacity that persisted in all the nonirrigated eyes and in 4 of the 6 irrigated ones. Nikitenko and Tolgskaya obtained permanent corneal opacity in a similar experiment.

Discussion and Suggestions for Further Research

Nikitenko and Tolgskaya noted narcosis, death, and tissue changes resulting from chronic exposure to TFE vapor. These were all absent in a similar study performed by Hazleton Labs. It is difficult to explain these results since the Russians do not provide much experimental detail. One possibility is that in their study the concentration underwent large excursions above the nominal 86 ppm. In another study they exposed animals to a nominal concentration of 15 ppm of TFE but the range was 2.4 to 22 ppm, a 50% excursion above the nominal level. The standard deviations from nominal concentrations in the Hazleton Labs. study were about 10%. (However, even a 50% excursion above 86 ppm would still be only 128 ppm.) Other important factors might be presence or absence of food during exposure, different strains of rat, temperature and humidity conditions, and-probably the most important-purity of the TFE used. Nikitenko and Tolgskaya do not supply any of this information. They should be contacted and asked to provide experimental details so that an explanation can be found for

the radical difference between their chronic inhalation results and those of Hazleton Labs.

Since bottoming cycle operation can be expected to create at least small amounts of impurities in TFE,⁸ it is important to determine whether these impurities increase the toxicity of TFE. Halocarbon Products Corp. did not characterize decomposition products resulting from heating various Fluorinols in their study; this should be done. Toxicological studies on slightly impure TFE may also be needed.

In the Hazleton Labs. study, male rats produced fewer sperm and offspring starting at an exposure of 50 ppm. Recovery occurred after exposure to this concentration, and tissue examination suggested that some recovery occurred after exposure to 150 ppm also. However, two weeks before this apparent recovery, rats were unable to sire offspring. Therefore it is possible that nonfunctional although normal-appearing sperm are produced, or alternatively that TFE-produced sterility is reversible. It is not clear why Hazleton Labs. waited two weeks after functional tests to examine testes. As far as setting a standard is concerned, further research is not needed, since even temporary sterility is unacceptable. However, it would be useful to know whether sterility is permanent or not.

In females, ovary wt./body wt. ratios increased as much with exposure to 10 ppm of TFE as with higher doses. No tissue changes were seen, but this does not guarantee that there were no toxic effects, in view of the presence of normal-appearing sperm in male rats who had been sterile 2 wk previously.

One cannot test whether humans react the same way as rats. Effects on humans could be more or less severe, show a different dose response, or vary differently between males and females. Therefore, standards should incorporate

a safety margin in case the response threshold is lower for humans than for rats. The suggested value of 2.5 ppm may be too high since there may be an effect on ovaries at that level; there may be no safety margin at all. The threshold for ovary enlargement should be determined and functional tests on females performed.

Nikitenko and Tolgskaya tested lower doses for acute exposure than Hazleton Labs. They claim that the threshold for intoxication and nervous system effects is about 29 ppm (4-hr exposure; rats). Death first occurred around 480 ppm (2-hr exposure; mice) and LC_{50} was about 710 ppm. Hazleton Labs. obtained LC_{50} of about 550 ppm (6-hr exposure; rats) with death first occurring between 350 and 500 ppm. They found severe nervous system effects at 350 ppm, but did no acute tests with lower doses. Since they observed no nervous system effects at 150 ppm in a chronic inhalation study, their threshold is between 150 and 350 ppm. Nikitenko and Tolgskaya and Hazleton Labs. thus agree closely on concentrations causing death, but Hazleton Labs. found an acute intoxication threshold 10 times as great as that found by the Russians. The intoxication threshold therefore needs to be studied; reproducible conditions should be used to test concentrations below 350 ppm, and Nikitenko and Tolgskaya should be asked to provide more details of their study.

Two workers who had TFE splashed into their eyes²⁰ and washed them immediately showed no ill effects. However, rabbit experiments suggest that in some cases irrigation, although helpful, does not prevent corneal opacity. Therefore, it might be advisable for workers to be provided with eye protection.

Because TFE can be absorbed through intact skin, workers should have access to emergency showers and to the readily available antidote, ethanol.

Since these safety measures are impractical for truck drivers, steps must be taken to ensure that their potential exposure to TFE is minute.

The DOE Toxic Materials Advisory Committee strongly advises that a less toxic fluid be sought since the ability of TFE to be absorbed through intact skin makes it very hazardous. We fully agree.

TRIFLUOROETHANOL USE IN TRUCK-BOTTOMING CYCLES

A proposed use for the Thermo Electron Corporation organic Rankine bottoming cycle is in long-haul heavy-duty trucks, where it would convert Diesel exhaust heat to useful energy by means of a turbine.^{1,2} A standard Mack truck Diesel engine (ENDT676) is currently being tested on a dynamometer by Thermo Electron, and a prototype truck should presently be tested by Mack Truck.² A 100-vehicle demonstration is planned for 1983, when Mack will decide whether to incorporate the bottoming cycle into its fleet as an option.^{1,5} Mack Truck's share of the 150,000 class 8 (\geq 1.5 metric tons) truck market is about 40,000 trucks.⁵

Each truck bottoming cycle holds about 4 liters of working fluid--Fluorinol-50.⁵ The packaging of the bottoming cycle has recently been redesigned.² The power conversion module (turbine, gearbox, feedpump, condenser, and regenerator) is now located behind the Diesel engine. On the engine tested by Thermo Electron, the condenser is air-cooled and located in front of the radiator. The new version uses water cooling, permitting the condenser to be smaller and to be located next to the turbine in a single heat-exchanger unit with the regenerator behind the engine. The water used to cool the condenser is segregated from the water used to cool the Diesel; there are two radiators. The

new design eliminates any tubing carrying organic fluid from the front of the engine. The vapor generator replaces the muffler in the compound engine.

As in power plant bottoming cycles, overheating, leaks, and explosions are conceivable hazards. In addition, trucks can be involved in traffic accidents. Trucks using bottoming cycles will weigh more than 13 metric tons.⁵ Since collision usually affects only the immediate site of impact on a truck of this size,⁵ only a direct hit at the engine might be expected to cause release of working fluid. Jack-knife accidents might also damage the bottoming cycle unit and release fluid. The effects of such release on the driver or others are unknown.

Would 4 liters of Fluorinol-50 affect drinking water if it should enter groundwater or a reservoir? Hazleton Labs.¹¹ reports that LD_{50} for TFE is in the range 126 to 233 $\mu\text{l/kg}$ for ingestion by rats; Nikitenko and Tolgskaya obtained $LD_{50} = 430 \mu\text{l/kg}$ by injecting TFE into the stomach. Hazleton Labs. observed no effects when 46.4 $\mu\text{l/kg}$ were ingested and slight depression without death at 100 $\mu\text{l/kg}$.

Let us assume that a person drinks 1 liter of contaminated water and that this is a newborn infant weighting 4 kg so that a maximal amount of TFE per body weight is ingested (neglecting any differential effects of TFE on different age groups). Then, if the TFE concentration is $< 200 \mu\text{l/liter}$ (0.02% by volume), the ingested amount will be $< 50 \mu\text{l/kg}$.

To dilute 4 liters of Fluorinol-50 (80% vol TFE = 3.2 liters) to 200 μl TFE per liter requires 16,000 liters of water, which has a volume of 16 m^3 , i.e., a cube 2.5 m long on each side (about the size of a very small room). Any stream, rainfall, or reservoir contains thousands (perhaps millions) of times this quantity of water. Thus, even if a person (even an infant) were to consume more than a liter of the contaminated water, even if lower doses affect humans

than rats, even if effects occur at 50 μ l/liter that have not been measured, and even if all 4 liters of Fluorinol-50 were washed into drinking water and did not decompose, still the leakage from a single truck bottoming cycle probably would not present a hazard in drinking water. If microorganisms convert TFE into trifluoroacetic acid, there is probably still no hazard since $LD_{50} = 200$ mg/kg for the metabolite in rats, which is comparable with LD_{50} for TFE, although it is true that compounds with similar LD_{50} may have different effects thresholds. Similar arguments can be used to estimate hazards of leakage of Fluorinol-85 from power plants.

Since bottoming cycles are expected to increase Diesel efficiency by 15%, they should reduce emissions at any speed. However, cooling exhaust gases might condense hydrocarbons, which might increase soot and ultimately affect heat exchanger performance. These problems are currently being studied by Thermo Electron.²

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