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FREON 113 AS A TEST MATERIAL FOR CHEMICAL CARTRIDGE RESPIRATORS



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by

C. F. Swab, Jr.,¹ and B. I. Ferber²

ABSTRACT

A relatively innocuous halocarbon was investigated as a substitute for the highly toxic carbon tetrachloride in the approval testing of chemical cartridges. The vapor effluent of 1,1,2-trichloro-1,2,2-trifluoroethane from chemical cartridges is continuously monitored by a nondispersive infrared analyzer. Breakthrough times for the cartridges, using the halocarbon, can be equated to those of carbon tetrachloride, if multiplied by the factor, 1.49.

INTRODUCTION

Chemical cartridge respirators, for protection against organic vapors, are certified under the requirements of Bureau of Mines Approval Schedule 23B³ using carbon tetrachloride (CCl₄) as the test medium. Carbon tetrachloride's threshold limit value (TLV⁴) of 10 ppm, coupled with its odor threshold of 50 ppm, makes its use hazardous. Several halocarbons (halogenated hydrocarbons), which are relatively innocuous compounds with physical properties similar to those of CCl₄ (table 1), are investigated as substitutes. Three halocarbons (Freons⁵ 11, 21, and 113) are used in this investigation. A syringe pump is used to generate the test vapor in comparison with the displacer apparatus presently used in approval testing. Approved cartridges are used in all tests. This is the only work known to have been done on the substitution of new materials for those presently employed for chemical cartridge respirator tests.

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³Bureau of Mines. Respiratory Protective Apparatus; Tests for Permissibility; Fees. Schedule 23B. Aug. 8, 1959, 8 pp.; Federal Register, v. 24, No. 159, Aug. 14, 1959, pp. 6619-6624.

⁴Threshold Limit Values of Airborne Contaminants Adopted by ACGIH for 1969.

⁵Reference to specific brands, manufacturers' names, and trade names is made for identification only and does not imply endorsement by the Bureau of Mines.

TABLE 1. - Physical properties of CCl_4 and halocarbons used in tests

Property	CCl_4	Freon 113 ¹	Freon 11 ²	Freon 21 ³
Molecular weight..g/mole.	153.83	187.39	137.37	102.92
Density.....g/ml.	1.584 ²⁵	1.5635 ²⁵	1.476 ²⁵	1.421 ⁰
Boiling point				
° C at 760 mm Hg.	76.8	47.6	24.1	8.9
TLV ⁴	10	1,000	1,000	1,000

¹1,1,2-trichloro-1,2,2-trifluoroethane.²Fluorotrichloromethane.³Dichloromonofluoromethane.⁴Threshold Limit Values of Airborne Contaminants Adopted by ACGIH for 1969.

EXPERIMENTAL WORK

Materials and Equipment

The displacement-type apparatus (fig. 1), presently employed with CCl_4 , is also used for halocarbons. Low-pressure air (approximately 7 psig), at 50-percent relative humidity and either 32 or 64 liters per minute (lpm) flow, passes through the vaporizer picking up the halocarbon vapor. The latter is generated by lowering the displacer rod into the halocarbon reservoir with a synchronous motor to give a known constant flow of liquid halocarbon into the vaporizer. The air and vapor are mixed and then passed through the test stand and into the hood exhaust. A nondispersive infrared analyzer (M-S-A LIRA[®]) monitors the test effluent. Our modification uses a syringe pump instead of the displacer (fig. 1) to supply the required volume of liquid needed for

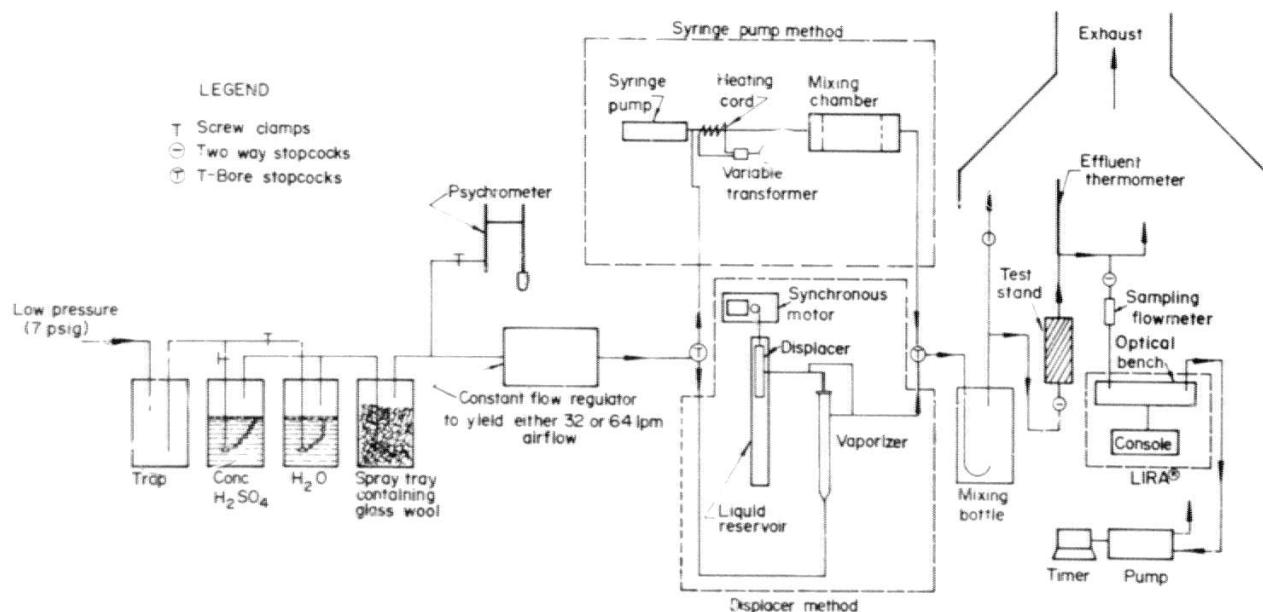


FIGURE 1. - Apparatus for Testing Chemical Cartridges.

vaporization. The syringe needle pierces a rubber septum and allows the liquid emanating from it to mix with the airstream. A heating cord wrapped around the liquid inlet aids vaporization. The mixing chamber and bottle assure homogeneity. One make of chemical cartridges is used throughout the tests to reduce variations in results. Freons 11, 21, and 113 are the hydrocarbons used.

Freon Screening Tests

Three Freons were investigated to determine the best one for testing cartridge service life. Freon 113 was tried because it is closest to CCl_4 in molecular weight and density (table 1). The LIRA® was calibrated and a curve of meter readings versus concentration in parts per million (ppm) was obtained. Cartridges were tested, under each set of conditions shown in table 2, using the displacer apparatus.

TABLE 2. - Conditions under which chemical cartridges were tested against CCl_4

CCl_4 concentration, volume-percent	Airflow, 1pm	CCl_4 life requirements, minutes
0.1	64	50
.1	32	100

Freon 21, a gas at room temperature (table 1), was tried. Two flowmeters were calibrated to provide the proper gas flows for the different vapor concentrations required. The LIRA® was calibrated and curves plotted.

Freon 11, with a boiling point of 24.1° C, vaporizes with slight warming. Breakthrough tests were tried to obtain life data.

Cartridge Tests

All subsequent cartridge tests were performed using Freon 113 (since Freon 11 and Freon 21 were eluted too rapidly). CCl_4 was used for comparison. Tests were performed with both displacer and syringe methods using air at 50-percent relative humidity.⁶ The syringe method was tried both at room temperature and with additional heat at the liquid inlet.

RESULTS AND CONCLUSIONS

Screening Tests

Eleven tests were made using approved cartridges at each condition in table 2. Test lives were compared with data for CCl_4 tests that had been recorded in past years. This may not be a rigorous comparison because of the different grades of charcoal used in chemical cartridges over the period of time involved. The cartridge test lives against Freon 113 were 30 to 38 percent lower than the test lives against CCl_4 (table 3).

⁶Lower humidities were tried, but did not produce any diverse results.

TABLE 3. - Comparison of lives (minutes), chemical cartridges, displacer method, 0.1 percent concentration, CCl_4 and Freon 113

64 lpm airflow		32 lpm airflow	
CCl_4	Freon 113	CCl_4	Freon 113
52.9	39.0	134.2	85.1
53.3	43.4	136.0	89.6
54.8	40.4	131.0	88.6
55.5	36.7	-	87.2
56.0	38.7	-	90.1
61.3	41.9	-	80.1
64.6	40.9	-	77.4
60.0	41.5	-	72.1
58.0	39.4	-	86.2
-	37.6	-	77.9
-	40.6	-	85.8
¹ 57.4±3.93	¹ ² 40.0±1.96	¹ 133.7±2.53	¹ ³ 83.7±5.87

¹Average and standard deviation.

²Decrease in life 17.4 minutes (30.3 percent). Average Freon 113/ CCl_4 ratio 0.70 ± 0.05 .

³Decrease in life 50.0 minutes (37.4 percent). Average Freon 113/ CCl_4 ratio 0.65 ± 0.02 .

Three cartridge tests were made at the high flow rate using Freon 21 (table 4). Test lives were approximately one-fifth of the CCl_4 life requirement of Schedule 23B (table 4). Results at low flow confirmed this observation.

TABLE 4. - Comparison of average cartridge test lives (minutes), Freons 11, 21, and 113; CCl_4 and Schedule 23B, 0.1 percent concentration

	64 lpm airflow	32 lpm airflow
Freon 11.....	21.8	-
Freon 21.....	8.8	21.8
Freon 113.....	40.0	83.5
CCl_4	57.4	133.7
Schedule 23B.....	50.0	100.0

Two cartridge tests at the high flow rate were made using Freon 11. Test lives were approximately 40 percent of the CCl_4 life requirement.

As a result of the screening tests, it was determined that Freon 113 could be a suitable substitute for CCl_4 . The other Freons tested have too low a breakthrough time. The lower service life produced by Freon 113 as compared to that of CCl_4 does not preclude its use as a test material. Test time can be reduced and required changes can be made in the approval schedules.

Cartridge Tests

These tests compare cartridge service lives against CCl_4 and Freon 113. The cartridges were tested under the following conditions:

1. Syringe method, liquid inlet at room temperature.
2. Syringe method, inlet heated to prevent condensation.
3. Displacer method.

The syringe pump, at room temperature, produced essentially the same service lives as the displacer (tables 3 and 5). The heated inlet on the syringe pump decreased service life (table 5). This may be due to the more complete vaporization of the test materials or to the effect of heated mixtures on cartridge performance. Tests with the displacer (table 5) confirmed those made in the screening tests (table 3).

TABLE 5. - Comparison of average lives (minutes), CCl_4 and Freon 113,
0.1 percent concentration at 64 lpm airflow and
50 percent relative humidity

Method	Life		Freon 113/ CCl_4	
	Minutes	Standard deviation	Ratio	Standard deviation
Room temperature syringe method.....	-	-	0.66	± 0.05
CCl_4	54.7	± 1.32	-	-
Freon 113.....	36.3	± 8.94	-	-
Heated inlet syringe method ¹	-	-	.73	$\pm .05$
CCl_4	44.9	± 1.12	-	-
Freon 113.....	32.9	± 1.71	-	-
Displacement method.....	-	-	.62	$\pm .05$
CCl_4	60.5	± 3.86	-	-
Freon 113.....	37.3	± 8.03	-	-

¹Liquid inlet heated to 70 percent of boiling point.

As the result of these experiments it was concluded that Freon 113 can be substituted for CCl_4 in testing chemical cartridge respirators for approval if an appropriate factor is applied. Data (summarized from tables 3 and 5) show this factor to be 0.67:

$$\begin{array}{r}
 0.70 \\
 .65 \\
 .66 \\
 .73 \\
 .62 \\
 \hline
 \text{Average} = 0.67 \\
 \text{Std. dev.} = \pm 0.04
 \end{array}$$

The service lives for cartridges tested with Freon 113 must be divided by a factor of 0.67 (or multiplied by 1.49) to give results comparable to those obtained with CCl_4 .