Diffusion Rates of Methyl Chloroform Through Some Insulation Materials

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July 28, 1965

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ABSTRACT

Diffusion rates of methyl chloroform through insulation and decorative materials cemented to aluminum surfaces with Bondmaster No. 596 neoprene contact adhesive have been studied; in addition, the permeability coefficients for Ensolite and surface-covered corkboard to the above-mentioned chlorinated hydrocarbon were measured (182.6×10^{-9} and 6.75×10^{-9} (lb/hr)(in.)/(in.^{2})(psig), respectively). Rate-of-diffusion measurements indicate that methyl chloroform is trapped behind surface-covered corkboard for a longer time (> 6 months) and emanates from the corkboard surface at a slower rate ($\sim 2.5 \times 10^{-6}$ lb/day-ft^2) than for any other insulation material studied. One-inch Ensolite retains methyl chloroform longer than corkboard whose surface has not been covered or 1/2-in. Ensolite. Of the materials studied, the one with the greatest diffusion rate of methyl chloroform was uncovered corkboard. Methyl chloroform did not diffuse through the decorative laminate materials.

On the basis of the data presented, a change of the corkboard cementing procedures in submarines has been recommended. Corkboard should be installed before being coated, and 1/2-in. Ensolite should replace the 1-in. material where possible.

PROBLEM STATUS

This is a final report on this phase of the problem; work on other phases of the problem is continuing.

AUTHORIZATION

NRL Problem C07-02
Project RR 001-01-43-4804

Manuscript submitted May 21, 1965.

DIFFUSION RATES OF METHYL CHLOROFORM THROUGH SOME INSULATION MATERIALS

INTRODUCTION

With the advent of nuclear power for submarine use, the limiting factor in extended submergence has been the submarine atmosphere. The U.S. Naval Research Laboratory has contributed to the long-term livability of the submarine atmosphere through development and evaluation of oxygen-generation and CO_2-removal systems and continuing studies of atmospheric contaminants (1-5).

The presence of some halogenated compounds in the atmosphere has been noted (6) in addition to the long-recognized hydrocarbon contaminants from fuels, paints, cleaning solvents, etc., and various Freons from leaking refrigeration systems. Some corrosion in the submarine has been attributed to hydrochloric acid formed on passage of air containing methyl chloroform through the H_2/CO catalytic burner. Methyl chloroform (CH_3CCl_3) has been detected in the atmosphere and in the main carbon filter beds of several FBM submarines that have had corrosion problems; moreover, vinylidene chloride ($CH_2{=}CCl_2$), a decomposition product of methyl chloroform, has also been detected in these ships (6). The evidence appears to be rather conclusive that methyl chloroform is the major source of the observed corrosion, though some contribution is to be expected from monoethanolamine (MEA) from the CO_2 scrubber and halogen acids from burner-catalyzed decomposition of Freons.

Recent studies have shown that large quantities of methyl chloroform are used during submarine construction as a major component in the solvent system of the neoprene cement used to adhere insulating and decorative materials to extensive surface areas within the submarine (6). While no methyl chloroform is carried aboard as part of the ship's stores, it may be reintroduced at intervals during maintenance periods between cruises.

The general problem seems to concern the quantity of solvent that is trapped behind the insulation or decorative panels and the rate at which it is released to the ship's atmosphere. This report covers the results of a study of the rate of diffusion of methyl chloroform through some of the materials which are attached within the submarines by neoprene contact cements.

EXPERIMENTAL METHOD

Background

The rate of transfer of one material through another is commonly expressed by a permeability coefficient which is obtained by measuring the mass of material transported per unit area in a given time interval under steady-state conditions through a fixed thickness of material. This coefficient Q is defined as

$$Q = \frac{\frac{dn}{dt} h}{A \ \Delta P}$$

where for this report, dn/dt is given in pounds per hour, the thickness h is in inches, A is in square inches, and ΔP is expressed as pounds per square inch.

The study was carried out in two parts:

a. The permeability coefficient Q was determined for the various materials, using as the driving force the vapor pressure of methyl chloroform at room temperature (2.14 lb/in.^{2} or 0.145 atm at 75 ± 2° F (7)), and

b. The rate of mass transfer under non-steady-state conditions was determined as a function of time after cementing the various materials to an aluminum plate; here the driving force was the vapor pressure of methyl chloroform at the cement surface.

A mass spectrometer was used to measure the extent of diffusion of the methyl chloroform; the technique was a modification of that described in a previous report (8). In both parts of this study the sample was placed under the collection cell, and the diffusing vapors were allowed to accumulate for 15 min. After an aliquot of these gases was introduced into the mass spectrometer and analyzed, the remaining vapors in the cell were removed with a stream of air. This procedure was repeated at regular intervals until a steady state of mass transfer was attained or the chlorinated solvent of the neoprene cement could not be detected passing through the sample.

Materials

The materials investigated were Ensolite (U.S. Rubber Co.), corkboard (Armstrong Cork Co.), and high-pressure Formica laminate (Formica Corp.). Ensolite, a unicellular plastic foam made of polyvinyl chloride, is supplied in production sheets either 1 in. or 1/2 in. thick. Three samples of each thickness, 12 in. square, each cut from the center of randomly selected production sheets, were obtained. The appearance of the 1-in. samples differed from the 1/2-in. samples in that an unidentified skin, typical of molded foam material, was on one side of the 1-in. samples. Both sides of the 1/2-in. and the reverse side of the 1-in. samples had a split-pore appearance.

Corkboard, a very porous material fabricated from granular cork and an appropriate binder, is usually covered with a smoothing cement (Navy Formula 62-GM8010-227-1698, Mil-C-15202) before installation in submarines. Samples of corkboard 1 in. thick and a can of smoothing cement were obtained for this study, and the diffusion characteristics of both the surface-covered and uncovered corkboard were investigated.

Diffusion properties of two types of decorative laminates were also studied. Both were 1/16 in. thick; however, one type had an aluminum foil between the laminate layers while the second did not.

Technical grade methyl chloroform used for the permeability studies was purified by gas chromatography. No impurities were found in a subsequent mass spectrometric analysis of the collected fraction.

Bondmaster cement No. 596 (Pittsburgh Plate Glass Co.) was used to adhere the materials to aluminum plates. Mass spectrometric analysis of the vapors emanating from the surface of a cement film indicated a mixture of methyl chloroform (80%) and toluene (20%). A more detailed analysis of the solvent system of this adhesive has been reported by Saunders (9).

In these experiments none of the materials were covered with decorative, pigmented paints which would further retard the diffusion rates. It would not have been possible to determine the extent of interference with the diffusion mechanism caused by these paints because of the wide variety of paints used in submarine construction.

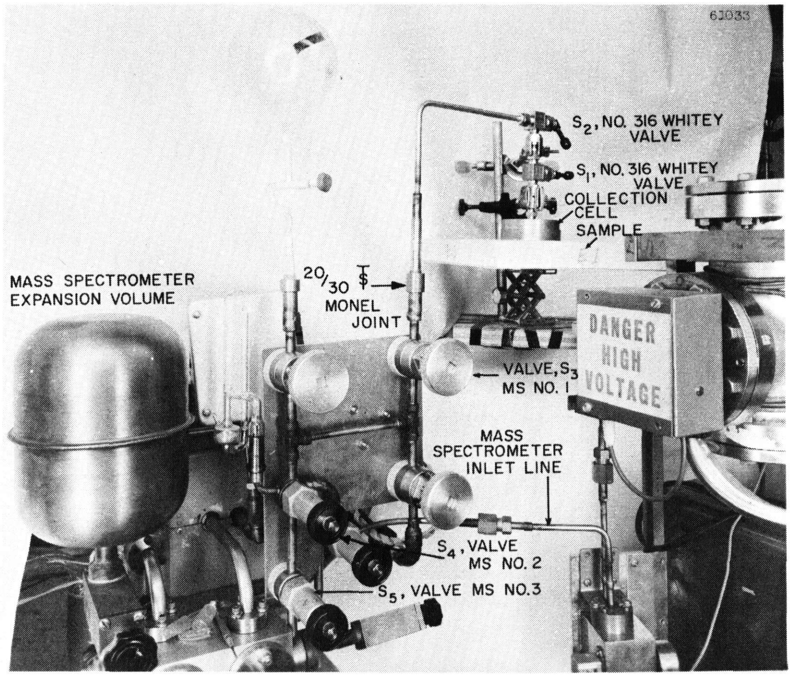

Fig. 1 - Collection cell and mass spectrometer inlet

Procedure and Apparatus

Each sample was placed under the collection cell, and the methyl chloroform diffusing through the samples into the cell was collected for 15 min. The collection cell, shown in Fig. 1, was a brass cup with a 40-cm^3 volume and an exposed area of 31.7 cm^2 (0.034 ft^2). One side of the cup was connected to the inlet system of the mass spectrometer through two No. 316 Whitey valves and a standard taper 12/30 Monel joint, while the opposite side of the cup was open. A sample was held against the open side of the cup with a "lab jack" during these experiments. The wall of the cup was perforated with sixteen 1/4-in. holes which could be sealed with a brass cover band. Raising the band exposed the holes so that gas accumulated in the cell could be removed with a jet of air after each mass spectrometric analysis.

From the ion current I_c observed at the mass-to-charge ratio $m/e = 97$ with a Bendix Time-of-Flight mass spectrometer described in detail elsewhere (8), the pressure of methyl chloroform accumulated in the collection cell was determined. The pressure was obtained by dividing I_c by the sensitivity S of the mass spectrometer for methyl chloroform. The sensitivity was determined by the following technique. Immediately after analysis of the collected gases, the mass spectrometer was evacuated and a known pressure P_μ of methyl chloroform introduced into the instrument. Again, the ion current at 97 was recorded and the value of this measurement divided by the known pressure was S, that is $S = I_c/P_\mu$.

Since the total pressure in the collection cell was 1 atm, the pressure had to be reduced before the collected vapors were introduced into the mass spectrometer. The pressure was reduced by expanding and trapping aliquots of the collected gases between the valves in the cell and inlet system of the mass spectrometer. To calculate the actual mass of methyl chloroform transferred through the sample, it was necessary to develop a relationship between the amount introduced into the spectrometer and that collected in the cell. Figure 2 aids in the derivation of this relationship.

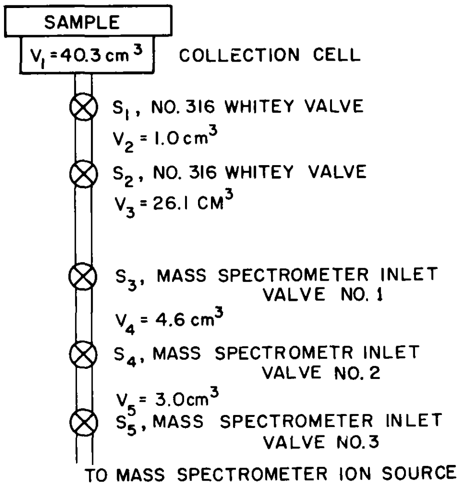

Fig. 2 - Expansion system

A sample was placed under the collection cell having volume V_1. During the collection time a pressure P_1 of methyl chloroform accumulated in this volume. The number of moles collected was

$$n_1 = \frac{P_1 V_1}{RT}$$

where R is the gas constant and T the temperature of the collection cell. Valve S_1 was opened, allowing the gases to expand into the evacuated volume V_2. Closing S_1 trapped n_2 moles in V_2, where

$$n_2 = n_1\left(\frac{V_2}{V_1+V_2}\right).$$

Next, valves S_2 and S_3 were opened, allowing the gas to expand into the evacuated volume V_3 plus V_4. Valve S_3 was closed, trapping n_3 moles of gas in V_4. The number of moles thus trapped is given by

$$n_3 = n_2\left(\frac{V_4}{V_2+V_3+V_4}\right) = n_1\left(\frac{V_2}{V_1+V_2}\right)\left(\frac{V_4}{V_2+V_3+V_4}\right)$$

where n_3 is the number of moles of gas introduced into the mass spectrometer. Thus the ratio n_1/n_3 may be evaluated by experimentally determining the volumes V_1, V_2, V_3 and V_4. This ratio relates the amount of gas collected in the cell to that introduced into the mass spectrometer; the measured ratio was 285 for this study.

After the accumulated gases had been analyzed, the band on the cell was raised, exposing the perforations, and air was passed through the cell to remove the remaining methyl chloroform. This collection and expansion procedure was repeated at 2-hour intervals until a steady state of mass transfer was attained (Background, part a) or until no methyl chloroform was detected passing through the sample (Background, part b). The only difference between the experimental procedures of part a and part b was the way in which the samples were exposed to the source of methyl chloroform.

In part a of this investigation the samples were sealed on aluminum cups containing methyl chloroform, and a separate cup was used for each sample. A steady state of mass transfer was assumed to exist when three or more consecutive collections of methyl chloroform contained the same amount of the chlorinated hydrocarbon.

In part b, where the samples were cemented to aluminum plates with Bondmaster cement No. 596, the source of methyl chloroform was constantly being depleted; thus a steady state was never attained. Furthermore, since ΔP, the pressure drop across the sample from the cement film, was not known, the permeability coefficients could not be calculated. Instead, the rate of mass transfer, dn/dt as a function of time was determined.

RESULTS AND DISCUSSION

Permeability coefficients have been determined for the diffusion of methyl chloroform through Ensolite and surface-covered corkboard at room temperature (75 ± 2°F) (Table 1). From the data shown in Table 1 it is seen that the coefficients for Ensolite are approximately 30 times the values for the corkboard. From this it can be predicted that trapped methyl chloroform would diffuse at a lower rate, but for a longer time through surface-covered corkboard than through Ensolite. Methyl chloroform did not diffuse through the decorative laminates in detectable amounts in any of these experiments.

Table 1
Permeability of Ensolite and Treated Corkboard to Methyl Chloroform Vapor*

Material	Sample No.	Sample Thickness (in.)	No. of Measurements	Permeability Coefficient Q $10^{-9}\frac{(lb/hr)(in.)}{(in.^2)(psig)}$
Ensolite	E-0	1	5	174.0 ± 3.2 †
	E-1	1	5	206.7 ± 12.5
	E-2	1	5	194.2 ± 12.5
	E-3	1	5	215.6 ± 2.8
	E-4	0.5	7	135.2 ± 8.9
	E-5	0.5	7	139.3 ± 6.8
	E-6	0.5	7	145.3 ± 5.2
	E-1 (top)‡	0.5	3	199.8 ± 8.9
	E-1 (bottom)‡	0.5	3	190.5 ± 13.3
	E-2 (top)‡	0.5	3	220.0 ± 3.6
	E-2 (bottom)‡	0.5	3	188.1 ± 2.8
				Avg. 182.6
Corkboard § (surface-treated with smoothing cement)	C-1a	1	4	7.7 ± 0.4
	C-1b	1	4	6.9 ± 0.4
	C-2a	1	4	5.6 ± 0.5
	C-2b	1	4	6.4 ± 0.4
	C-3a	1	4	6.7 ± 0.5
	C-3b	1	4	7.2 ± 0.4
				Avg. 6.75

*Pressure gradient was vapor pressure of methyl chloroform at 75 ± 2°F (2.14 lb/in.^{2}).
†Uncertainties are the standard deviation of the mean.
‡Samples E-1 and E-2 were sliced to give two 0.5-in. sheets. Top sheet retained original surface skin.
§Duplicate samples were taken from 3 production sheets.

A comparison of Q for the 1-in. and 1/2-in. Ensolite would seem to indicate that this constant is dependent upon thickness. However, as mentioned previously, there is a physical difference observable between these materials. A portion of the Ensolite used for E-1 and E-2 was split horizontally to give two 1/2-in. specimens from each sheet. Samples designated E-1 top and E-2 top retained the original molded skin while both surfaces of E-1 bottom and E-2 bottom had a split-pore appearance. Data in Table 1 show that the

Table 2
Rate of Diffusion of Methyl Chloroform from Ensolite and Corkboard Cemented to Aluminum Surface with Bondmaster No. 596 Neoprene Cement

Sample	Sample Thickness (in.)	Elapsed Time* (hr)	Diffusion Rate (10^{-4} lb/day per ft^2)
Ensolite, Sample E-1	1	2	0.81
		4	0.83
		5	0.67
		26	0.46
		30	0.62
		173	0.65
		2880 (120 days)	0.002
		4320 (180 days)	not detectable
Ensolite, Sample E-5	0.5	2	10.3
		4	8.2
		5	7.2
		26	0.6
		30	0.5
		173	not detectable
Corkboard, surface-treated Sample C-1a	1	2	0.030
		4	0.028
		5	0.035
		26	0.033
		30	0.030
		173	0.029
		2880	0.026
		4320	0.023
Corkboard, surface untreated	1	0.5	2.28
		1.0	1.80
		2.4	1.49
		3.7	1.26
		4.5	0.91
		23.6	0.12
		27.6	0.13
		96.0	0.13
Bondmaster No. 596 cement film	-	0.167	5.19
		0.7	1.32
		2.0	0.61
		4.5	0.31
		5.6	0.27
		23.6	0.05

*Cement-coated surfaces were brought in contact after 10-min drying period.

surface skin effect is small compared with sheet-to-sheet variation and the permeability is independent of thickness, since Q for the split samples agrees reasonably well with the Q found for the parent material. However, with the supplied 1/2-in. samples Q varies from the 1-in. materials by a factor of almost two, which is greater than the experimental error.

Data in Table 2 show the diffusion rate of methyl chloroform from Ensolite (both thicknesses) and corkboard (surface-covered and uncovered) cemented to an aluminum

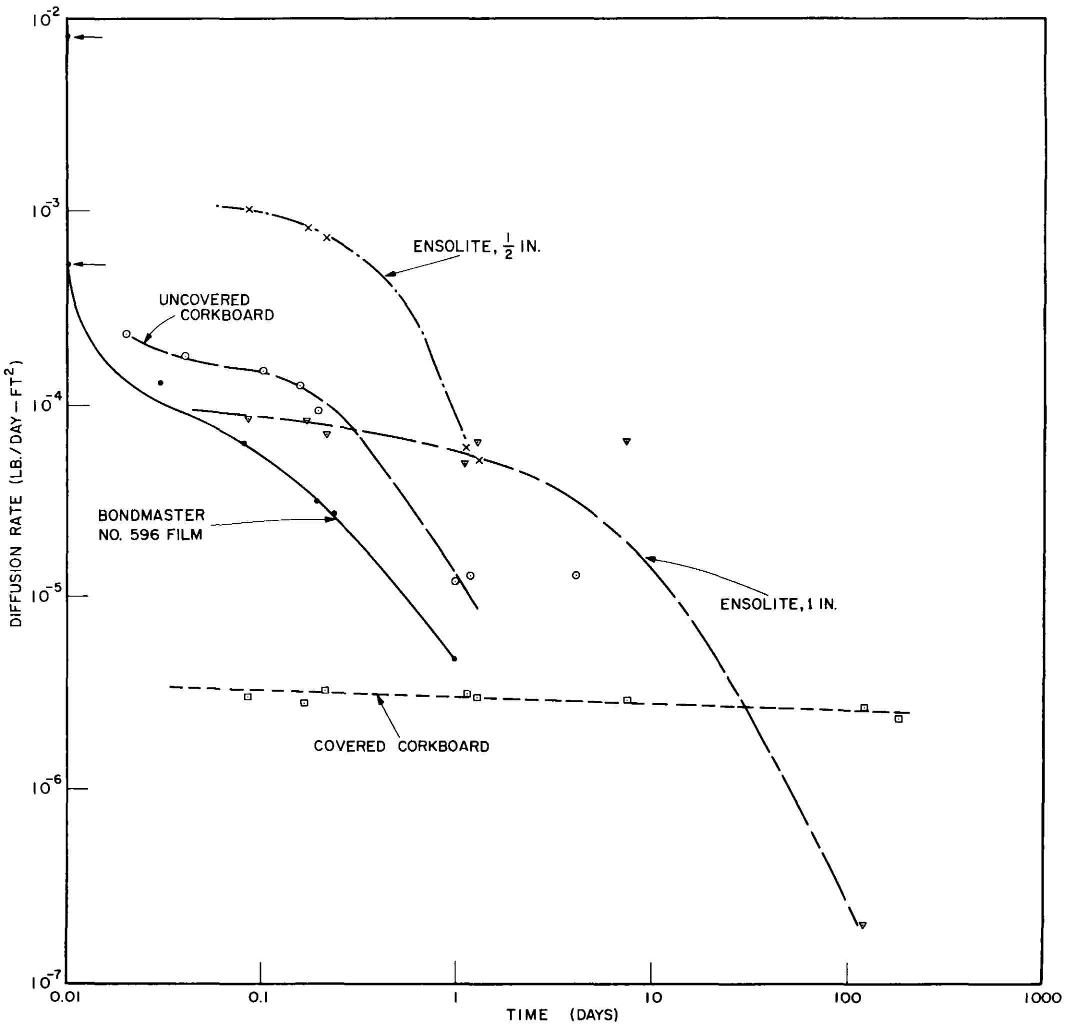

Fig. 3 - Rate of diffusion of methyl chloroform from several materials cemented with Bondmaster No. 596 adhesive (minimum detectable amount was 5.0×10^{-9} lb/day-ft²)

surface with Bondmaster No. 596 neoprene adhesive. Also shown in Table 2 is the diffusion rate of methyl chloroform from an uncovered film of the Bondmaster cement. The results are shown graphically in Fig. 3.

With the exception of the surface-covered corkboard, all of the curves delineated in Fig. 3 have similar shapes. The uncovered Bondmaster cement film initially has a very high diffusion rate of methyl chloroform which drops rapidly to a plateau before descending below the minimum detectable limit of the method (5.0×10^{-9} lb/day-ft²). Corkboard whose surface had not been covered with smoothing cement had a diffusion rate curve similar to that of the cement film. However, the initial peak value was not observed, the plateau had a smaller slope, and the time required to reach the detectability limit was longer. Half-inch Ensolite had a curve whose shape resembled the previously discussed curves, except the chlorinated hydrocarbon was detected diffusing from the material's surface for a longer time; and at the plateau values, the rate of methyl chloroform diffusion was greater than that found for any other material.

Plateau diffusion rates of 1-in. Ensolite were lower in value but longer in time duration than for the 1/2-in. Ensolite. Methyl chloroform diffused from the 1-in. material at a relatively constant rate for approximately 10 days before a noticeable descent in the curve was observed. One hundred twenty days (4 months) after the Ensolite had been cemented to an aluminum surface, detectable quantities of methyl chloroform were still emanating from the surface of this material.

Most unique of all the diffusion rate curves was the surface-covered corkboard, which had a very low rate of passage initially and remained constant for 180 days (6 months). No appreciable decrease in the amount of evolved methyl chloroform was detected during this time. This implies that the methyl chloroform trapped behind this material will diffuse into the atmosphere at a slow but steady rate ($\sim 2.5 \times 10^{-6}$ lb/day-ft^2) for a very long period of time. Although it can be anticipated that this diffusion rate curve will eventually decrease below the detectable limit, the length of time required cannot be predicted from the data.

If the area of insulation material used in submarines is assumed to be 60,000 ft^2, the amount of methyl chloroform diffusing through the surface-covered corkboard can be estimated as 0.1 lb/day, agreeing with the amount found by shipboard measurements (10). It should be noted that in the shipboard measurements the methyl chloroform was found to be diffusing from Ensolite and not from corkboard. Presumably, the shipboard measurements were made at a time before the diffusion rate curve of Ensolite fell below that of the surface-covered corkboard and the rate of diffusion through this latter material was below the detectability limit of the equipment used.

While the Bondmaster No. 596 cement contains methyl chloroform and toluene as solvents in an approximately 3:2 ratio (9), the ratio of vapor pressure of these solvents above a drying cement film is about 4:1. The mixture diffusing through surface-covered corkboard during the first week, however, contained only 10% toluene; after 10 days the toluene could no longer be detected. Therefore, it is concluded that toluene is preferentially retained in the cement film and the bulk of the corkboard and will continue to emanate from this source at a slow rate for a long period of time.

SUMMARY AND RECOMMENDATIONS

The diffusion rates of methyl chloroform through insulation and decorative materials cemented to aluminum surfaces with Bondmaster No. 596 adhesive have been investigated by a mass spectrometric technique. The measurements have shown that methyl chloroform trapped behind surface-covered corkboard diffuses into the atmosphere at a slow rate ($\sim 2.5 \times 10^{-6}$ lb/day-ft^2) for an extended length of time ($>$ 6 months). One-inch Ensolite retains methyl chloroform longer than 1/2-in. Ensolite or corkboard whose surface has not been covered with smoothing cement. Methyl chloroform passes through uncovered corkboard faster than through any other of the materials studied. No chlorinated hydrocarbon could be detected diffusing through the decorative laminates.

If the use of adhesives which have chlorinated solvents must continue, the following changes are recommended:

1. The corkboard should be installed in submarines prior to being covered with smoothing cement. Two weeks after installation, the vast majority of the trapped solvent will have diffused from behind the corkboard and thus can be removed from the ship before it is ready for sea trials.

2. Half-inch Ensolite should replace the 1-in. material where possible since the methyl chloroform trapped behind this material will be removed in a shorter period of time.

ACKNOWLEDGMENT

The authors wish to thank Mr. R.O. Bullock of the Bureau of Ships for obtaining the materials used in this study.

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DOCUMENT CONTROL DATA - R&D

(Security classification of title, body of abstract and indexing annotation must be entered when the overall report is classified)

1. ORIGINATING ACTIVITY *(Corporate author)* U.S. Naval Research Laboratory Washington, D.C. 20390	2a. REPORT SECURITY CLASSIFICATION Unclassified 2b. GROUP

3. REPORT TITLE
DIFFUSION RATES OF METHYL CHLOROFORM THROUGH SOME INSULATION MATERIALS

4. DESCRIPTIVE NOTES *(Type of report and inclusive dates)*
A final report on one phase of the problem

5. AUTHOR(S) *(Last name, first name, initial)*
Saalfeld, F.E., and McDowell, M.V.

6. REPORT DATE July 28, 1965	7a. TOTAL NO. OF PAGES 14	7b. NO. OF REFS 10

8a. CONTRACT OR GRANT NO. NRL Problem C07-02 b. PROJECT NO. RR 001-01-43-4304 c. d.	9a. ORIGINATOR'S REPORT NUMBER(S) NRL Report 6302 9b. OTHER REPORT NO(S) *(Any other numbers that may be assigned this report)*

10. AVAILABILITY/LIMITATION NOTICES
Unlimited Availability
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11. SUPPLEMENTARY NOTES	12. SPONSORING MILITARY ACTIVITY Dept. of the Navy (Office of Naval Research)

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14. KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
Submarine habitability Methyl chloroform Permeability coefficient Diffusion rate Insulation materials Adhesive cement Mass spectrometer						

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