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ELASTOMERS IN UNDERWATER APPLICATIONS, (U)
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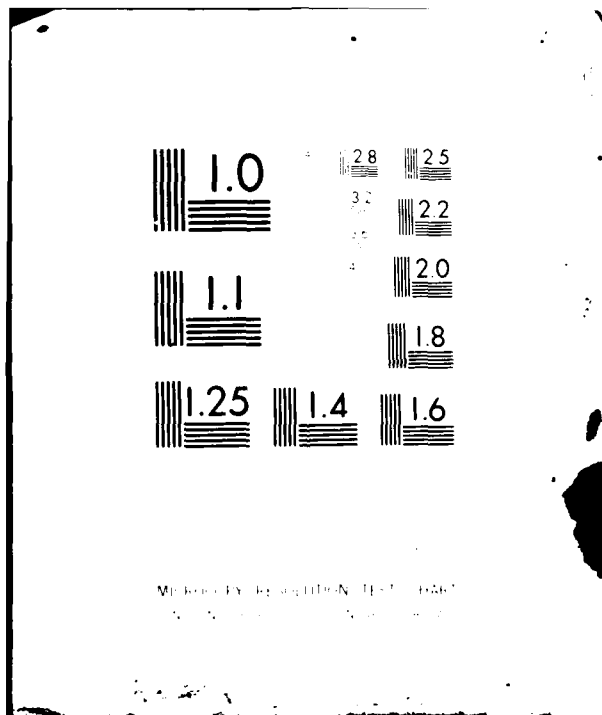
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**DEPARTMENT OF DEFENCE
DEFENCE SCIENCE AND TECHNOLOGY ORGANISATION
MATERIALS RESEARCH LABORATORIES
MELBOURNE, VICTORIA**

REPORT

MRL-R-824

ELASTOMERS IN UNDERWATER APPLICATIONS

David Oldfield and Ted Symes

Approved for Public Release



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MATERIALS RESEARCH LABORATORIES

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David Oldfield and Ted Symes

ABSTRACT

The successful longterm operation of the Australian designed Mulloka sonar system was found to be dependent on the correct choice of material for an elastomeric membrane intended to prevent moisture ingress into the underwater transducer array. The amount of moisture removed after a period in service was in good agreement with the amount predicted from laboratory measurements. A study of the influence of compounding ingredients on the water vapour permeability of bromobutyl rubber vulcanizates was carried out in order to produce low permeability elastomeric membranes for sonar applications. The factors affecting the water vapour permeabilities of nitrile rubber vulcanizates are reported here for the first time.

*Presented at the Fifth Australasian Rubber Technology Convention organised by the PRI, Australasian Section, Canberra, October, 1980.

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16. ABSTRACT (if this is security classified, the announcement of this report will be similarly classified):

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C O N T E N T S

	<u>Page No</u>
1. INTRODUCTION	1
2. EXPERIMENTAL	2
2.1 Materials	2
2.2 Vulcanizate Preparation	2
2.3 Permeability Measurements	2
3. RESULTS AND DISCUSSION	2
3.1 Water Vapour Permeability	2
3.2 Butyl/polyurethane Permeabilities	3
3.3 Bromobutyl Vulcanizates	4
3.4 Nitrile Rubber Vulcanizates	4
3.5 Blends	5
3.6 Carbon Black Permeability	5
3.7 Temperature Dependence	5
4. CONCLUSIONS	6
5. ACKNOWLEDGEMENTS	6
6. REFERENCES	6
TABLE 1 MATERIALS	7
TABLE 2 PERMEABILITIES OF CAST POLYURETHANE MEMBRANES	8
TABLE 3 BROMOBUTYL RUBBER COATING	9
TABLE 4 BROMOBUTYL RUBBER FORMULATIONS	10
TABLE 5 NITRILE RUBBER FORMULATIONS	11
TABLE 6 BROMOBUTYL/NATURAL RUBBER BLEND FORMULATIONS	12
TABLE 7 BROMOBUTYL/NITRILE RUBBER BLEND FORMULATIONS	13



ELASTOMERS IN UNDERWATER APPLICATIONS

1. INTRODUCTION

Elastomeric components incorporated into modern defence equipment often have to function under adverse conditions for long periods of time. The underwater transducer array for the Mulloka sonar system [1], designed and developed within the Defence Science and Technology Organisation at Defence Research Centre, Salisbury, South Australia and the Royal Australian Navy Research Laboratory, Sydney, provided an example of the crucial role that elastomers can play in the successful operation of complex equipment. The cylindrical array comprised a large number of individual sonar staves which house the transducers and was mounted inside a free-flooding glass-reinforced polyester resin dome attached to the keel of HMAS Yarra. An elastomeric membrane was used to prevent water ingress into the sonar staves while maintaining acoustic isolation between the transducer heads and their housing. The interior of the sonar staves had to remain dry to prevent electrical and corrosion problems which would have made the whole sonar system inoperable. The design of the staves was such that an elastomeric membrane was required which had a much lower permeability than was previously specified for sonar equipment. The importance of the permeability of the membrane was not fully recognised at the initial design stage, and this eventually led to the transducer array being withdrawn from service due to severe internal corrosion.

A complete refurbishment of the transducer array was carried out by the US contractor, Honeywell Defense Electronics Division, during which two elastomeric membranes consisting of a butyl coating over cast polyurethane were assessed for suitability. Subsequently it was discovered that the method of application of the protective butyl rubber coating produced significant differences in properties between test specimens and the coating on the actual sonar staves. The amount of water which permeated into the array during operational service with the Royal Australian Navy on HMAS Yarra was found to be in good agreement with the amount predicted from laboratory measurements.

In order to improve the reliability of the transducer array it was desirable to develop low permeability membranes for use in future production of sonar arrays. A limited amount of published data [2,3] was available on the water vapour permeability of elastomers but it was difficult to establish the specific formulations used in every case. Diffusion in elastomers has been reviewed [4] and the effects of fillers on water absorption have been reported [5] but there appeared to be little work on the influence of

compounding variables on water vapour permeability. It has been reported [2] that the physical state of the water in contact with the membrane, either vapour or liquid, can affect the permeability values. Most of the literature data were determined with water vapour on the 'wet' side of the membranes whereas liquid water is more relevant to the sonar application. The present study set out to investigate some of the effects of different compounding ingredients on the permeabilities of bromobutyl vulcanizates in contact with liquid water.

2. EXPERIMENTAL

2.1 Materials

The materials used are shown in Table 1. Other compounding ingredients comparable in chemical composition and activity should be equally satisfactory. The formulations of the vulcanizates are shown in Tables 3 to 7.

2.2 Vulcanizate Preparation

Compounding was performed on a laboratory two-roll mill (200 mm x 75 mm) and the curing characteristics of each compound were determined on a Monsanto model LSD Rheometer. Test sheets were cured in a two cavity mould (225 mm x 125 mm x 1.9 mm) in an electrically heated press at 160°C for the time taken to reach 90 percent of the maximum torque in the Rheometer.

2.3 Permeability Measurements

The method used was based on ASTM practice [6] with an improved method of securing the membrane. The specimen holder consisted of a cylindrical anodised aluminium cup (60 mm internal diam. x 25 mm deep) as shown in Figure 1. Distilled water (10 ml) was poured into the cup, a membrane disc (56 mm in diameter) was placed on the inner ledge, a suitable washer was placed on top of the membrane and secured by a threaded retaining ring. The thickness of the membrane was measured in at least 5 places before insertion in the test cup. The cup assembly was inverted so that the inner face of the membrane was covered with water and then placed in a desiccator containing activated silica gel at $21^{\circ} \pm 2^{\circ}\text{C}$ or $40^{\circ}\text{C} \pm 1^{\circ}\text{C}$. The cup was removed and weighed at intervals of not less than two days. The loss in weight was plotted against elapsed time and measurements were continued until a straight line could be drawn through four consecutive points.

3. RESULTS AND DISCUSSION

3.1 Water Vapour Permeability

The water vapour permeability of a membrane may be defined as the mass of water vapour which passes in unit time through unit area and unit thickness

of the membrane when there is unit water vapour pressure difference across the membrane. In this report permeability is expressed as

$$\frac{\text{g cm}}{\text{cm}^2 \text{ h torr}}$$

The amount of water vapour passing through a given area and thickness of the membrane in a specified time may be readily calculated from the permeability. In practical applications it is important to know the water vapour transmission rate for the particular membrane in use.

3.2 Butyl/polyurethane Permeabilities

During the refurbishment of the Mulloka sonar array by the US contractor in 1977 it was proposed to use a polyurethane membrane with an outer butyl rubber coating applied from a solvent solution. Two candidate butyl coatings were submitted to MRL for evaluation. The test specimens were in the form of brush applied coatings on a polyurethane substrate and the results, shown in Table 2 indicated that butyl A was the preferred material.

The refurbishment program had to meet a very tight schedule to allow the sonar array to be replaced on HMAS Yarra during the routine dry docking period. In order to achieve this the butyl coating solution was sprayed on to the sonar array. Specimens of the sprayed coating were not made available for permeability testing until after the array had been refitted to the ship. Measurements of the permeability of the sprayed coating indicated that it was at least ten times more permeable than anticipated from the previous results (Table 2). Examination of the sprayed coating under a scanning electron microscope (SEM) revealed microporosity. This porosity was later found to be even more obvious in samples of sprayed butyl coating removed from the actual sonar stave surfaces.

During the refurbishment a quantity of desiccant, molecular sieve 4A, had been placed inside each stave and a limited number of staves were also fitted with internal relative humidity sensors. These sensors indicated that the relative humidity inside the staves had risen to 20 percent after 90 days immersion and to 75 percent after 200 days. The amounts of water adsorbed by the desiccant at these relative humidities were calculated from a calibration curve obtained by relating the adsorbed water content to equilibrium relative humidity over the desiccant. The amount of water adsorbed within each stave for an indicated relative humidity of 75 per cent was estimated as 13 g. An unexpected corrosion problem later necessitated the removal and repair of the array and the opportunity was taken to completely dry the staves. The amount of water removed from each stave under high vacuum varied from 12 g to 14 g, which agreed well with the predicted value. The average water vapour transmission rate calculated from the amount of water removed was $7.3 \text{ g/m}^2\text{d}$ compared to the value of $6.6 \text{ g/m}^2\text{d}$ calculated from laboratory measurements.

Before the sonar array was returned to service an approximately 0.2 mm thick coating of a bromobutyl rubber compound, formulated at MRL, was applied by brush to each stave and cured for 24 h at 100°C. The formulation, together with the permeability measured on a cured film, is shown in Table 3. The

superior resistance to water vapour permeation of this material was demonstrated by the fact that the relative humidity inside the array after return to service had risen to only 23 percent after 220 days and to 40 percent after 440 days. The improved performance enabled sea trials of the Mulloka sonar system to be successfully completed which in turn led to the placing of an order for the production of further sonar sets worth \$9 million to Australian industry.

3.3 Bromobutyl Vulcanizates

The formulations and permeability results of a series of bromobutyl vulcanizates are presented in Table 4. The permeabilities of filled systems did not appear to be very sensitive to changes in compounding ingredients. Permeabilities between 18×10^{-10} and 40×10^{-10} were obtained for vulcanizates with several different formulations. For an unfilled vulcanizate, the permeability was found to be 8×10^{-10} whilst those for compounds containing 30 phr to 70 phr of N550 or N219 carbon black ranged between 18×10^{-10} and 24×10^{-10} . Substitution of part of the carbon black by an equal volume of a clay filler produced little change in the permeability of the vulcanizate. The addition of 10 phr of litharge to a formulation containing 70 phr of N550 carbon black reduced the permeability of the resulting vulcanizate to 6×10^{-10} but similar additions in the presence of clay fillers had little effect. Bromobutyl rubber vulcanizates with useful mechanical properties would seem from these results to display water vapour permeabilities of about 20×10^{-10} in the units previously stated.

3.4 Nitrile Rubber Vulcanizates

The literature [1,2] did not appear to contain any water vapour permeability data for nitrile rubbers. It was expected that increasing the polarity of the polymer by increasing the bound acrylonitrile content would affect the water vapour permeability. A series of vulcanizates was prepared containing 70 phr of N550 carbon black in nitrile rubbers containing 18, 27, 34, 38 and 50 percent bound acrylonitrile in the polymer. The formulations and the permeability results are shown in Table 5. The permeabilities, with the exception of the one from the polymer containing 38 percent acrylonitrile, fell on a smooth curve when plotted against the bound acrylonitrile content of the polymer as shown in Figure 2.

The permeabilities of a series of vulcanizates consisting of different amounts of N550 carbon black in a nitrile rubber containing 34 percent bound acrylonitrile are shown in Figure 3. Substitution of 40 parts of the carbon black by equivalent volumes of the lamellar fillers mica or talc reduced the permeability by about 35 percent compared to a compound containing 70 phr of N550 carbon black.

Thus it can be seen that the water vapour permeability of nitrile rubber vulcanizates was reduced either by increasing bound acrylonitrile content of the polymer or by increasing the loading of carbon black or by partial substitution of carbon black by lamellar fillers.

3.5 Blends

Blends of bromobutyl rubber with natural rubber or nitrile rubber were prepared to the formulations listed, together with the permeabilities of the vulcanizates, in Tables 6 and 7. These permeabilities, when plotted against the percentage by weight of bromobutyl rubber in the blended polymers, produced a smooth curve as shown in Figures 4 and 5. In Figure 6 the logarithm of the permeability of the blended vulcanizates was plotted against the volume percentage of bromobutyl rubber in the total vulcanizate. The permeability results for the previous vulcanizates in which bromobutyl rubber was the only elastomer were treated in the same way and recorded on the same graph. A linear regression analysis of the plotted values was found to give a correlation coefficient of 0.97 which confirmed the graphical indication that a straight line could be drawn through the points. Thus the logarithm of the permeability of the vulcanizates was inversely proportional to the volume percentage of bromobutyl rubber in the vulcanizates.

3.6 Carbon Black Permeability

The addition of increasing amounts of carbon black increased the water vapour permeabilities of bromobutyl rubber vulcanizates but decreased the permeabilities of nitrile and natural rubber vulcanizates. This suggested that the carbon black was behaving as if it had an apparent permeability value which was higher than that of a bromobutyl gum stock but lower than that of nitrile and natural rubber gum stocks. A plot of the logarithm of the permeability against the volume percentage of carbon black in the vulcanizates gave a straight line for the nitrile rubber system as shown in Figure 7. Extrapolation to 100 percent carbon black gave an apparent permeability of 140×10^{-10} for the carbon black. This should be regarded as an estimate only and additional work could be done to pursue this concept further.

3.7 Temperature Dependence

Previous authors [7] noted that the permeability of several materials, including polystyrene, poly (vinyl chloride) and rubber hydrochloride was very temperature dependent. It was stated that the magnitude of this temperature dependence varies so much from one material to another that the performance cannot be predicted with certainty at one temperature on the basis of measurements at another temperature. The permeability results obtained in the present study at 22°C and 40°C for bromobutyl vulcanizates are in agreement with this conclusion. The low values of the bromobutyl permeabilities made experimental errors more significant than in more permeable materials. However the permeabilities of the bromobutyl vulcanizates did not appear to be influenced by temperature in any manner which was consistent from one vulcanizate to another.

4. CONCLUSIONS

4.1 Laboratory water vapour permeability measurements were confirmed as being useful for predicting the actual amount of water which would permeate into sonar equipment in service. Good agreement was found between the actual amount of water present inside the equipment and that predicted from laboratory measurements.

4.2 Addition of increasing amounts of carbon black to bromobutyl rubber compounds increased their water vapour permeability but similar increases of carbon black in the case of nitrile rubber and natural rubber decreased the permeabilities. An inherent permeability could be derived for carbon black which was intermediate between the permeabilities of bromobutyl and nitrile rubber gum stocks.

4.3 The water vapour permeability of nitrile rubber vulcanizates decreased with increasing bound acrylonitrile content in the polymer.

4.4 The logarithm of the permeability of bromobutyl/natural rubber or bromobutyl/nitrile rubber blends was inversely proportional to the volume fraction of bromobutyl rubber in the total compound.

5. ACKNOWLEDGEMENTS

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T A B L E 1

MATERIALS

<u>MATERIALS</u>	<u>TRADE NAME</u>	<u>SUPPLIER</u>
Antioxidant A	Permanax B	Vulnax International Ltd.
Antioxidant B	Flectol H	Monsanto Aust. Ltd.
Bromobutyl rubber	Bromobutyl X2	Polysar Ltd.
Butyl A coating	Elastron 860 X	United Coatings, Spokane, Wa. USA
Butyl B coating	Vapolon	Hempels Industrial coatings San Francisco, Cal. USA
Carbon black	Acarb	Australian Carbon Black Pty. Ltd.
Clay	Kaolin 37	ACI Minerals Pty. Ltd.
Silane treated clay	Nucap 200	A. Victor Leggo & Co. Pty. Ltd.
Coumarone resin	Polymer 300	Union Carbide Aust. Ltd.
Calcined clay	Glomax LL	A. Victor Leggo & Co. Pty. Ltd.
N-cyclohexyl-2- benzothiazyl sulphenamide	Vulcafor CBS	Vulnax International Ltd.
Dibenzthiazyl disulphide	Vulcafor MBTS	Vulnax Internation Ltd.
Litharge	Canary Litharge	Commonwealth Litharge & Red Lead Pty. Ltd.
Mercaptobenzthiazole	Vulcafor MBT	Vulnax International Ltd.
Mica	Mica D	Steeley Industries Ltd.
Nitrile rubbers	Krynac	Polysar Ltd.
Nitrile rubber (18% ACN only)	Hycar 1094-80	B.F. Goodrich Chemicals Pty. Ltd.
Polyurethane	PR-1538	Products Research and Chemical Corp., Burbank, Cal.
Talc	Talc TX	Steeley Industries Ltd.
Tetramethyl thiuram disulphide	Vulcafor TMTD	Vulnax Internat. Ltd.
Zinc oxide	White Seal Zinc Oxide	Lysacht Durham Chemical Co. Pty. Ltd.

T A B L E 2

PERMEABILITIES OF CAST POLYURETHANE MEMBRANES

Material	Thickness mm	Permeability ^{a)}	Temperature °C
Polyurethane	1.57	4490	20
Polyurethane/butyl A ^{b)}	1.57/0.10 ^{c)}	560	20
Polyurethane/butyl B ^{b)}	1.57/0.10 ^{c)}	970	20
Polyurethane	3.12	4750	20
Polyurethane	3.12	6340	40
Polyurethane/butyl A ^{d)}	3.12/0.52 ^{c)}	5720	20
Polyurethane/butyl A ^{d)}	3.12/0.52 ^{c)}	6580	40

(a) Permeability in $\frac{\text{g cm}}{\text{cm}^2 \text{ h torr}} \times 10^{10}$

(b) Coating applied by brush, water against coating during measurements.

(c) Thickness polyurethane/thickness butyl coating.

(d) Coatings applied by spray, water against coating during measurements.

T A B L E 3

BROMOBUTYL RUBBER COATING

Ingredients	Parts by weight
Bromobutyl rubber	100.00
Stearic acid	1.00
Zinc oxide	5.00
N219 carbon black	5.00
Dibenzthiazyl disulphide	1.00
Tetramethyl thiuram disulphide	1.00
Sulphur	2.00
Antioxidant A	1.00

Compound mill mixed, diced and dissolved in toluene at a concentration of 150 g/l.

Permeability of a film cured for 24 h at 100°C was

$$25 \times 10^{-10} \frac{\text{g cm}}{\text{cm}^2 \text{ h torr}} \quad \text{at } 20^\circ\text{C}$$

BROMOBUTYL RUBBER FORMULATIONS

FORMULATION NO	1	2	3	4	5	6	7	8	9	10	11	12
<u>Ingredients^{a)}</u>												
Carbon Black N550	70.00	30.00			50.00	50.00	50.00	70.00				
Carbon Black N774				50.00								
Clay				70.00								
Silane treated clay					70.00			101.00	101.00		101.00	
Calcined clay						70.00	70.00			101.00		101.00
Litharge							10.00	10.00			10.00	10.00
TOTAL	178.80	138.80	108.80	228.80	228.80	228.80	238.80	188.80	209.80	209.80	219.80	219.80
Permeability at 22°C ^{b)}	18	24	8	53	23	24	36	6	38	22	32	22
Permeability at 40°C ^{b)}	26	22	29	-	36	21	31	31				

a) All formulations contained the following ingredients (parts by weight):

Bromobutyl rubber 100.00, Stearic acid 1.00, Zinc oxide 3.00, TMTD* 0.50, CBS* 3.00, MC Sulphur 0.30, Antioxidant A 1.00.

* TMTD = Tetramethyl thiuram disulphide.

* CBS = N-cyclohexyl-2-benzothiazyl sulphenamide.

b) Units of permeability, $\frac{\text{g cm}}{\text{cm}^2 \text{ h torr}}$ x 10⁻¹⁰

T A B L E 5

NITRILE RUBBER FORMULATIONS

FORMULATION NO	13	14	15	16	17	18	19	20	21
<u>Ingredients a)</u>									
	<u>parts by weight</u>								
Nitrile Rubber (18% ACN)	100.00								
Nitrile Rubber (27% ACN)		100.00							
Nitrile Rubber (34% ACN)			100.00	100.00	100.00	100.00	100.00		
Nitrile Rubber (38% ACN)								100.00	
Nitrile Rubber (50% ACN)									100.00
Carbon black N550	70.00	70.00		30.00	70.00	30.00	30.00	70.00	70.00
Talc						62.00			
Mica							62.00		
TOTAL	191.25	191.25	121.25	151.25	191.25	213.25	213.25	191.25	191.25
Permeability at 21°C ^{b)}	1020	950	1520	1090	860	525	580	970	495

a) All formulations also contained the following, expressed as parts per hundred parts of rubber:

Stearic acid 1.00, Zinc oxide 3.00, Antioxidant B 1.00,
Coumarone resin 10.00, Sulphur 0.25, CBS* 3.00, TMTD* 3.00.

*CBS = N-cyclohexyl-2-benzothiazyl sulphenamide.

*TMTD = Tetramethyl thiuram disulphide.

b) Units of permeability, $\frac{\text{g cm}}{\text{cm}^2 \text{ h torr}} \times 10^{-10}$

T A B L E 6

BROMOBUTYL/NATURAL RUBBER BLEND FORMULATIONS

FORMULATION NO	22	23	24	25	26	27	28	29	30	31
<u>Ingredients,</u>										
<u>parts by weight</u>										
Natural rubber		25.00	50.00	75.00	25.00	50.00	75.00		100.00	100.00
Bromobutyl X2	100.00	75.00	50.00	25.00	75.00	50.00	25.00	100.00		
Stearic acid	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00
Zinc oxide	3.00	3.00	3.00	3.00	3.00	3.00	3.00	3.00	3.00	3.00
Carbon black N219	50.00	50.00	50.00	50.00	70.00	70.00	70.00	70.00		
Carbon black N550								50.00	50.00	
TMTD ^{a)}	0.50	0.50	0.50	0.50	0.50	0.50	0.50	0.50	0.50	
CBS ^{b)}	1.25	2.00	3.00	3.00	3.00	3.00	3.00	3.00	3.00	
MC Sulphur			0.20	0.30	0.30	0.30	0.30	0.30	0.30	0.30
Antioxidant A			1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00
TOTAL	155.75	157.70	158.80	178.80	178.80	178.80	178.80	158.80	178.80	178.80
Permeability at 22°C ^{c)}	15	30	140	240	41	116	164	21	412	354

a) TMTD = Tetramethyl thiuram disulphide.

b) CBS = N-cyclohexyl-2-benzothiazyl sulphenamide.

c) Units of permeability, $\frac{\text{g cm}}{\text{cm}^2 \text{ h torr}} \times 10^{-10}$

T A B L E 7

BROMOBUTYL/NITRILE RUBBER BLEND FORMULATIONS

FORMULATION NO	32	33	34
<u>Ingredients,</u>			
<u>parts by weight</u>			
Bromobutyl rubber	67.00	33.00	
Nitrile rubber (34% ACN)	33.00	67.00	100.00
Stearic acid	1.00	1.00	1.00
Zinc oxide	3.00	3.00	3.00
Antioxidant A	1.00	1.00	1.00
Carbon black N550	70.00	70.00	70.00
Coumarone resin	3.30	6.70	10.00
MC Sulphur	0.30	0.30	0.30
CBS ^{a)}	2.70	2.30	2.00
TMTD ^{b)}	0.67	0.84	1.00
TOTAL	181.97	185.14	188.30
Permeability at 22°C ^{c)}	82	350	840

a) CBS = N-cyclohexyl-2-benzothiazyl sulphenamide

b) TMTD = Tetramethyl thiuram disulphide

c) Units of permeability, $\frac{\text{g cm}}{\text{cm}^2 \text{ h torr}} \times 10^{-10}$

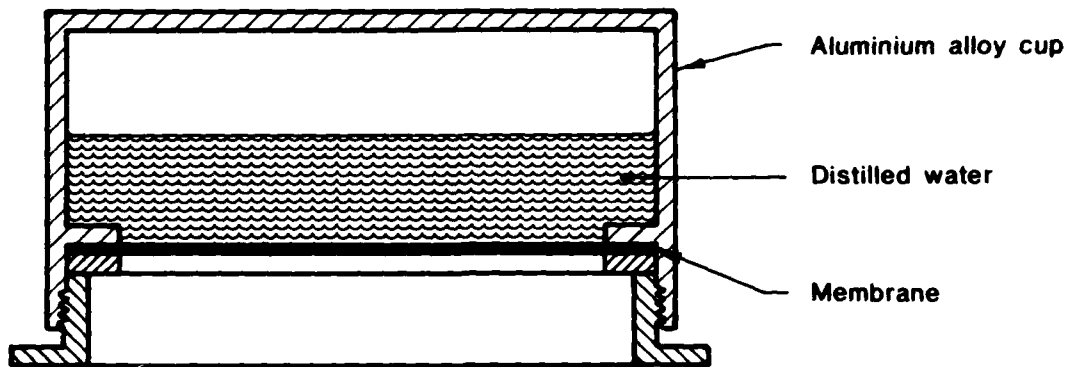


FIG. 1 - Permeability measuring cup.

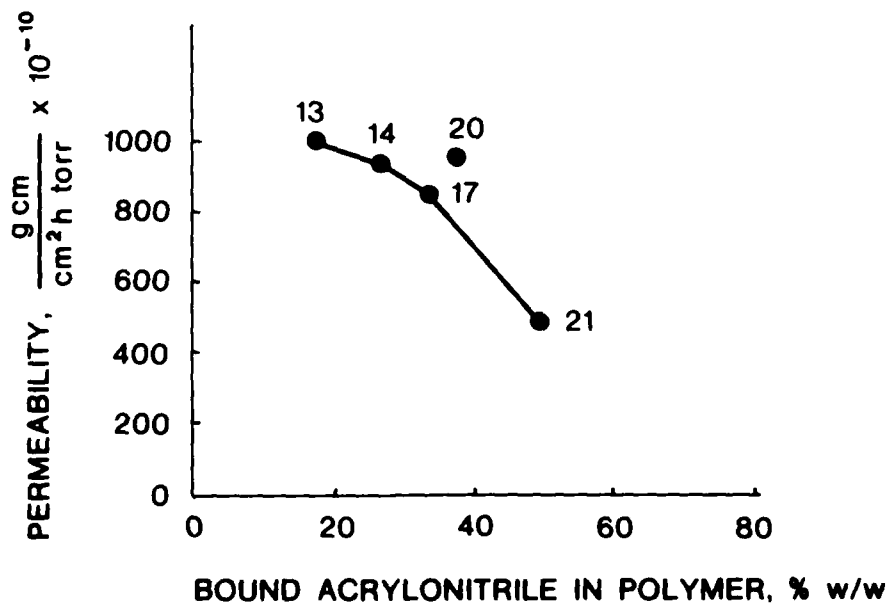


FIG. 2 - Permeability of nitrile vulcanizates with different amounts of bound acrylonitrile in polymer. Formulation numbers as shown.

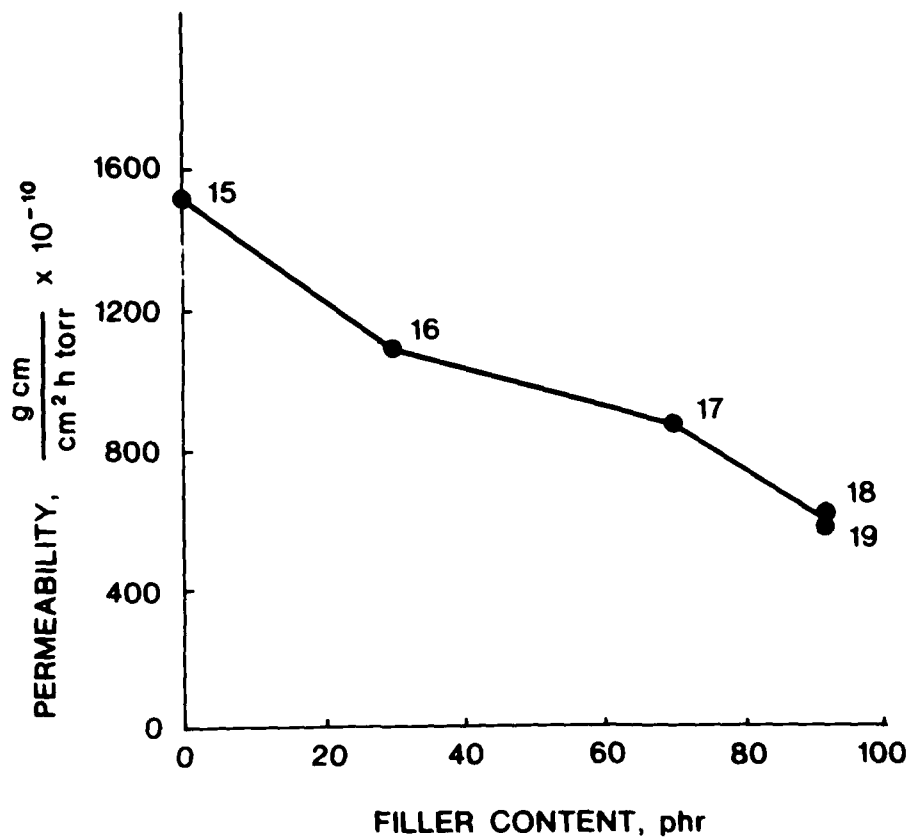


FIG. 3 - Permeability of nitrile vulcanizates (34% ACN) with various amounts of fillers, formulation numbers as shown.

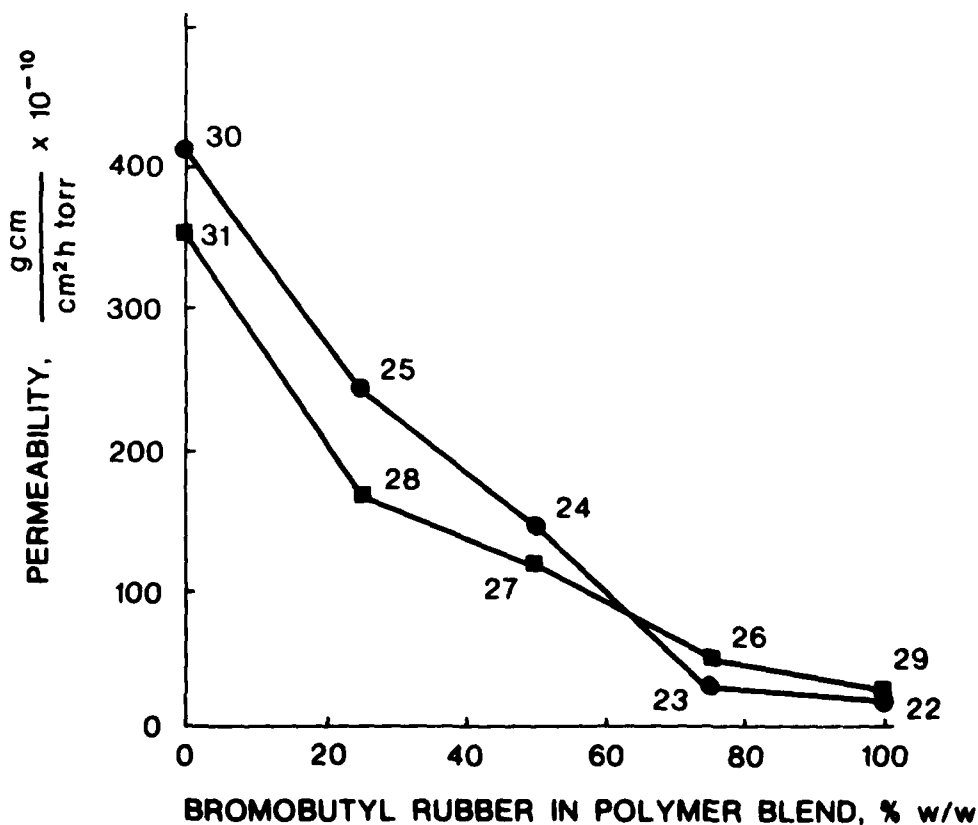


FIG. 4 - Permeabilities of bromobutyl/natural rubber blends, formulation numbers as shown.

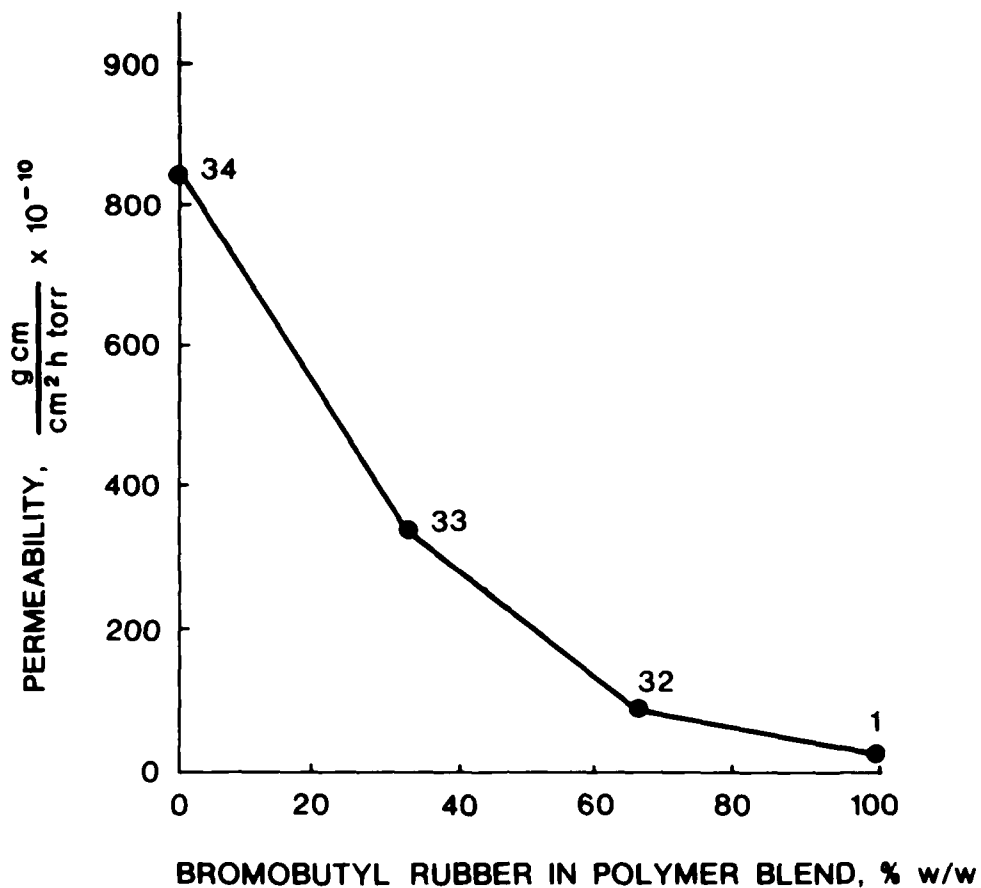


FIG. 5 - Permeabilities of bromobutyl/nitrile rubber blends, formulation numbers as shown.

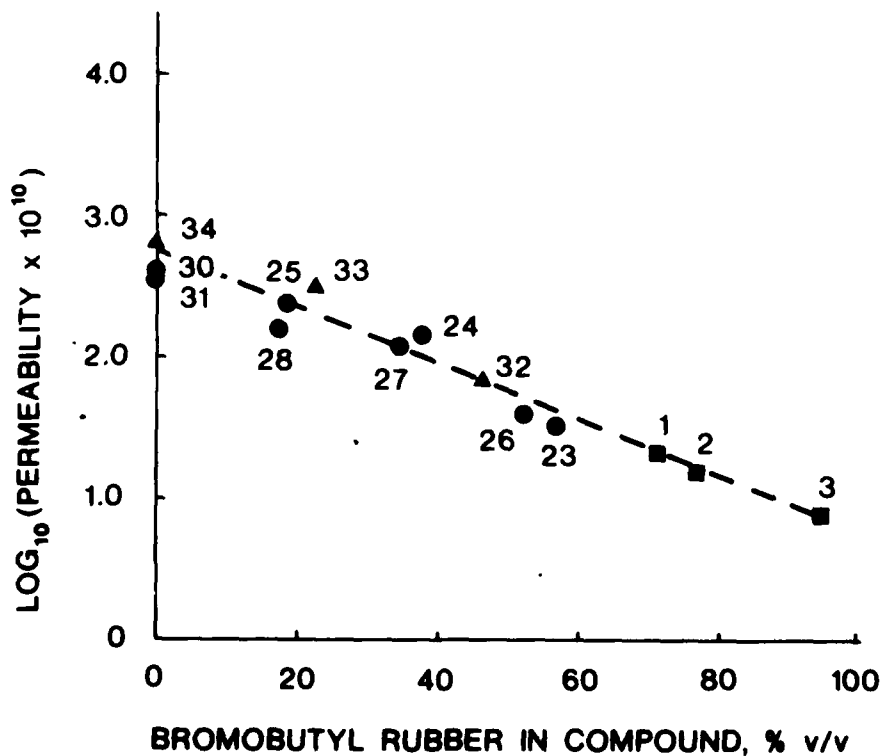


FIG. 6 - Logarithms of permeabilities of vulcanizates containing bromobutyl rubber, formulation numbers as shown.

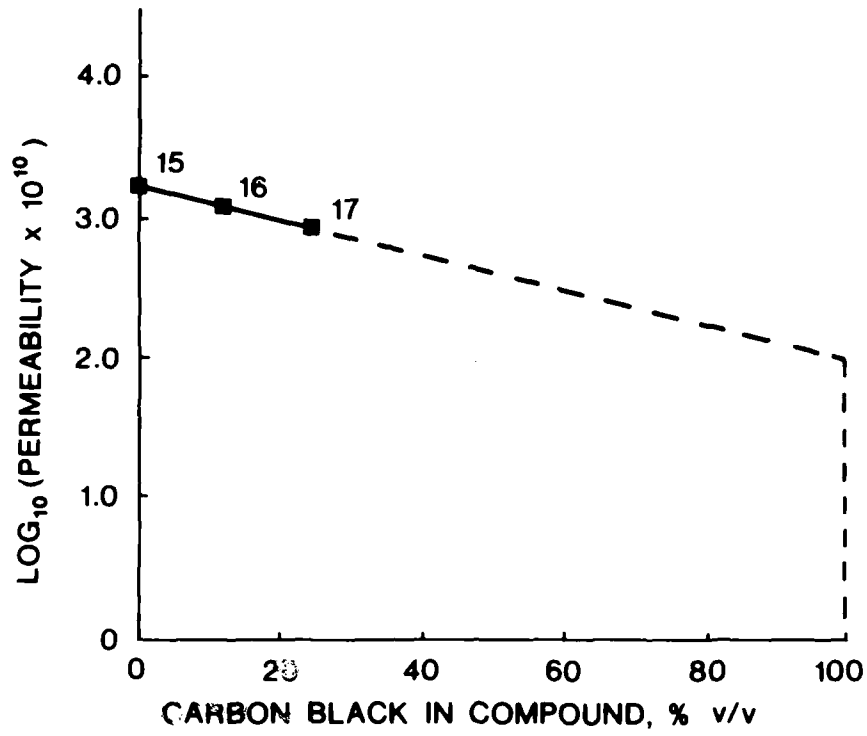


FIG. 7 - Logarithms of permeability of nitrile rubber vulcanizates, formulation numbers as shown.

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