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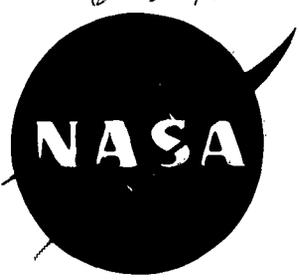
NASA TN D-1284

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TECHNICAL NOTE

D-1284

COMPATIBILITY OF SEVERAL PLASTICS AND ELASTOMERS
WITH SODIUM, POTASSIUM, AND RUBIDIUM

By Louis Rosenblum and Henry Putre

Lewis Research Center
Cleveland, Ohio

62-3-1

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
WASHINGTON
April 1962

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E-1452

SUMMARY

Fourteen representative plastic and elastomer samples were immersed in sodium, potassium, or rubidium for from 3 to 10 days at 250° or 350° F to determine their compatibility with these alkali metals. Changes in weight, hardness, tensile strength, percent elongation, and flexibility were measured, and changes in appearance were noted. A selection was made of materials that were expected to yield minimum contamination to the alkali metals and that showed no appreciable degradation of physical properties under the various conditions of time and temperature.

INTRODUCTION

Experimental investigations involving the use of the alkali liquid metals invariably necessitate the transfer and containment of these metals at temperatures somewhat above their melting points. Therefore, appropriate gasket, pump and valve packing, and valve seat materials are needed that will be compatible with the liquid alkali metals at temperatures of about 250° F.

Previous alkali metal compatibility tests of rubbers and elastomers were made using a sodium-potassium alloy only and were restricted to measurements of weight change and changes in the appearance of the sample (ref. 1). This report discusses an experimental study of the changes in properties of several plastics and elastomers when exposed to sodium at 250° and 350° F and to potassium and rubidium at 250° F. The property changes investigated were those recommended by the ASTM for elastomer immersion tests: weight, hardness, tensile strength, percent elongation, and flexibility (ref. 2).

EXPERIMENTAL DETAILS

Materials

The plastic and elastomer specimens used in this study are listed in table I(a) together with their source of supply and original properties. Since many of the suppliers hold their formulations as proprietary, an incomplete composition list is presented (table I(b)). The materials selected were all commercially available materials. They were chosen to present a comprehensive sampling of the various chemical varieties of polymers that might find use in sealing applications. No attempt was made to select, a priori, a given formulation as to additives, polymer chain length, or curing time. Procurement difficulties with the Kel-F elastomers necessitated a change of supplier and a concomitant change in formulation part way through the test program.

The sodium used in runs 1 and 2 was c.p. Baker and Adams lump sodium that was filtered through a 20-micron stainless-steel filter just before each run. Mine Safety Appliances "High Purity" potassium was used directly from its shipping container for run 3. The potassium from run 3 was filtered through a 20-micron stainless-steel filter for run 4. Rubidium from the American Potash Company was used for run 5.

Test Apparatus

The sodium and potassium compatibility tests, runs 1, 2, 3, and 4, were carried out in an electrically heated stainless-steel test chamber (fig. 1). As a safety precaution, the test chamber was placed in a dry box inerted with argon. On top of the chamber was a filtration unit for filtering the liquid metal when necessary. Two thermocouples for measuring liquid top and bottom temperature, a thermoregulator for temperature control, and openings for vacuum argon lines penetrated the cover of the test chamber. The test specimens were positioned in the specimen holder as shown in figure 2.

In the rubidium test, run 5, a small glass test chamber was used (fig. 3) to minimize the amount of liquid metal needed. The test specimens were held in a slotted stainless steel holder. A thermostated silicone oil bath was used to heat the glass test chamber.

Test Procedure

The test chambers were heated to a temperature just above the melting point of the liquid metal and evacuated to a pressure of 1 micron.

Liquid metal was introduced into the chamber so as to cover the specimens,¹ and the chamber was backfilled with argon to atmospheric pressure. The liquid metal in the test chamber was brought up to test temperature and maintained at this temperature ($\pm 3^{\circ}$ F) for the duration of the run.

After the run the liquid metal was drained from the chamber, and the specimens were washed with ethyl alcohol to remove any liquid metal adhering to their surfaces. This was followed by a water rinse and then air drying.

In runs 1, 2, 3, and 4, Die C specimens (ref. 3) were tested, while in run 5, 3/4-inch-diameter specimens were used.

Properties Tests

Tensile properties (ultimate strength and elongation) were measured in accordance with ASTM specification D 412-51T (ref. 3) using Die C specimens. Instantaneous and 15-second hardness (the latter is a measure of cold flow) were measured with a Shore A Maximum Reading Durometer in accordance with ASTM D 676-55T (ref. 4). An analytical balance was used to measure specimen weight. Flexibility was measured by bending the test specimen back on itself 180° and observing whether or not the specimen cracked.

In each run three specimens of each material were tested. An average value was calculated for each property of interest.

RESULTS AND DISCUSSION

The results for runs 1, 2, 3, 4, and 5 are shown in table II. The percent changes (due to immersion in the liquid metal) in the original properties of weight, instantaneous and 15-second Shore A hardness, tensile strength, and percent elongation are given for runs 1, 2, 3, and 4. For run 5 changes in weight and hardness are given but not changes in tensile strength and percent elongation, because these could not be determined for the small specimens. In addition, in all runs 180° bend test results and remarks concerning the appearance of the specimens after immersion are given. Photographs of the specimens taken before and after the tests are shown for runs 1, 2, 3, and 4 in figures 4, 5, 6, and 7, respectively.

¹In run 4 the specimens were only half covered with the liquid metal.

The final selection of materials most suitable for use with the alkali metals tested was made based on the following criteria:

- (1) Low weight loss of material
- (2) Small losses in tensile strength, hardness, and flexibility
- (3) Absence of powdery decomposition products on the surface of the material

Since minimization of contamination of the alkali metal was of prime concern, the final selection of materials, given by the following table in terms of first and second choice, reflects the fact that the criteria (1) and (3) were weighted more heavily than criteria (2) in the evaluation:

	First	Second
Sodium at 250° F up to 9 days	Kel-F 3700	Buna-N
Sodium at 350° F up to 7 days	Neoprene	Buna-N
Potassium at 250° F up to 3 days	Buna-S	Buna-N
Potassium at 250° F up to 10 days	Buna-S	Buna-N
Rubidium at 245° F up to 3 days	Neoprene	Buna-N

As would be expected, the extent of attack by the alkali metals on the elastomers and plastics increased with time and temperature. The order of severity of attack was found to be potassium > rubidium > sodium. The order of reactivity of the alkali metals is in general cesium > rubidium > potassium > sodium > lithium (ref. 5), which follows as expected from the electronegativity values for the alkali metals. Therefore, the expected order of severity of attack should be rubidium > potassium > sodium. However, reactivity of the alkali metals may also be controlled by steric factors that involve the atomic volume of the reactants (ref. 5). This steric effect produces the reverse order of reactivity of that given by electronegativity effects. Hence, combined electronegativity and steric effects could have lead to the observed results.

It should be noted that most of the materials listed in table I are not pure polymeric compounds but are mixtures of the cross-linked polymer with various additives (fillers, curing agents, plasticizers, etc.) that impart certain desired processing and physical characteristics to the material. The deterioration of a material when exposed to the alkali metal at temperature can be caused by reaction (1) along the polymer chain, (2) at the chain cross linkages, (3) with the additives, or (4) involving any combination of these three.

E-1452

An example of attack along a polymer chain was seen in this investigation in the case of Teflon and Kel-F, where a portion of the polyhalogenated carbon chain was reduced to elemental carbon. This general type of reaction was expected because organic compounds containing chlorine are known to react vigorously with alkali metals to produce carbon as a major reaction product (refs. 6 and 7). It was not expected, however, that Kel-F 3700 and neoprene should show such a slow attack by sodium at 250° F in run 1. Nevertheless, inertness to reaction with the alkali metals should be found for hydrocarbon polymer chains containing no reactive functional groups because, in general, alkali metals do not react with pure hydrocarbons except those possessing conjugated unsaturation (refs. 8 and 9).

Cross-linkage attack was probably present in the case of the sulfur vulcanized polymers such as Buna-N, Buna-S, butyl rubber, and natural rubber. The most likely site of attack in this instance would be the disulphide cross linkage. It is known that the sulfur-sulfur bond of organic disulphides is readily cleaved by sodium and sodium-potassium alloy (ref. 10). Elimination of cross-linkage attack would be effected with cross linkages that are unreactive toward the alkali metals. Carbon-carbon bond cross links formed by beta or gamma irradiation should be of this nature. The Irrathene tested in this program was a radiation-cross-linked polymer. Unfortunately it was procurable only in the form of film, so no hardness or strength measurements were made. However, it showed the smallest weight change of any of the materials tested with sodium at 350° F for 168 hours, and there was no apparent degradation of flexibility.

An example of additive attack may be conjectured in the case of butyl rubber, which contains appreciable amounts of Paraplex plasticizer, an organic ester. Organic esters are known to react with sodium to form acyloins, diketones, and ketoesters (ref. 11). The reaction of the organic ester plasticizer with the alkali metals may have been a major cause in the weight loss and increase in hardness experienced with the butyl rubber specimens. It would be prudent to exclude from elastomers designed for liquid-metal service any additives that are known to react extensively with alkali metals.

SUMMARY OF RESULTS

An evaluation of the compatibility tests conducted with 14 representative elastomers and plastic specimens immersed in sodium, potassium, or rubidium was made based on (1) weight loss of material, (2) powdery deposits formed on the surface of the material, and (3) degradation of material physical properties. Criteria (1) and (2) were given more

weight in this evaluation than (3), since minimizing the contamination of the alkali metal was deemed to be the most important consideration. The materials that appeared most suitable in the light of these criteria are listed in the following table as first and second selections:

	First	Second
Sodium at 250° F up to 9 days	Kel-F 3700	Buna-N
Sodium at 350° F up to 7 days	Neoprene	Buna-N
Potassium at 250° F up to 3 days	Buna-S	Buna-N
Potassium at 250° F up to 10 days	Buna-S	
Rubidium at 245° F up to 3 days	Neoprene	Buna-N

Lewis Research Center
National Aeronautics and Space Administration
Cleveland, Ohio, January 12, 1962

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E-1452

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TABLE I. - PLASTIC AND ELASTOMER SPECIMENS
(a) Source of supply and original properties

Material	Polymer chemical composition	Stock or recipe no.	Source	Original properties ^a			
				Instantaneous Shore A hardness	15 sec Shore A hardness	Tensile strength, psi	Percent elongation
Buna-N	Butadiene-acrylonitrile copolymer	Hycar 1001 recipe II	B. F. Goodrich Chemical Co., Cleveland, Ohio	68	62	3,180	547
Buna-S	Butadiene-styrene copolymer	GR-S-1502 stock no. 444	Buckeye Rubber Co., Cleveland, Ohio	72	67	1,540	390
Butyl rubber	Isobutylene-isoprene copolymer	Hycar 2202 recipe no. 520-44-69-2	B. F. Goodrich Chemical Co., Cleveland, Ohio	67	57	1,100	517
Natural rubber	Polyisoprene	Stock no. 8675	B. F. Goodrich Chemical Co., Cleveland, Ohio	39	39	(b)	(b)
Neoprene	Polychloroisoprene	Stock no. M5565	E. I. Du Pont de Nemours & Co.	74	72	1,610	443
Teflon	Polytetrafluoroethylene	-----	E. I. Du Pont de Nemours & Co. General Electric Co.	(c)	(c)	3,070	290
Silicone rubber	Methyl vinyl siloxane polymer	SE 371	General Electric Co.	74	74	1,010	97
Silicone rubber	Methyl vinyl siloxane polymer	SE 751	General Electric Co.	48	48	390	153
Nylon	Polyhexamethylene adipamide	-----	E. I. Du Pont de Nemours & Co.	(c)	(c)	10,500	90
Kel-F	Chlorotrifluoroethylene-vinylidene fluoride copolymer	5500	Minnesota Mining & Manufacturing Co.	66	64	2,120	400
Kel-F	Chlorotrifluoroethylene-vinylidene fluoride copolymer	3700	Minnesota Mining & Manufacturing Co.	68	66	1,880	300
Kel-F	Chlorotrifluoroethylene-vinylidene fluoride copolymer	3700 VL-1101M4	Vernay Laboratories, Yellow Springs, Ohio	68	66	2,905	515
Irrathene	Irradiated polyethylene	101	General Electric Co. Plastics Manufacturing & Supply Co., Cleveland, Ohio	(d)	(d)	(d)	(d)
Polypropylene	Polypropylene	-----	-----	(c)	(c)	5,110	30

^aAs measured in this investigation.

^bDid not rupture at maximum grip separation.

^cBeyond hardness range of Shore A Durometer.

^dSpecimens available only in form of film.

E-1452

TABLE I. - Concluded. PLASTIC AND ELASTOMER SPECIMENS

(b) Material recipes

Component	Amount of component in recipe, parts			
	Buna-N	Buna-S ^a	Butyl rubber	Natural rubber ^a
Pure gum sheet				✓ <10 parts additives per 100 parts rubber
Hycar 1001	100			
GR-S		✓		
Hycar 2202			100	
Sulfur		✓		
Zinc oxide	5.0	✓	5.0	
Philblack A	40.0	✓	60.0	
EPC Black		✓		
B. I. Whiting		✓		
Silene E. F.			5.0	
Mineral rubber		✓		
Methyl Tuads	3.5		1.5	
Sulfasan R			1.5	
DPG		✓		
Sanicure		✓		
Stearic acid	1.0		3.0	
Paraplex			10.0	
Paraplex G-60			5.0	

^aNumber of parts not available.

TABLE II. - EXPERIMENTAL RESULTS OF IMMERSING SEVERAL PLASTICS AND ELASTOMERS IN SODIUM, POTASSIUM, AND RUBIDIUM

Material	Change in original properties, percent					180° bend flexibility (a)	Change in appearance (a)
	Weight	Instantaneous Shore A hardness	15 sec Shore A hardness (cold flow)	Tensile strength	Percent elongation		
Run 1. Specimens immersed in sodium at 250° F for 219 hours							
Buna-N	-0.70	3	6	-12	-41	Y	M
Buna-S	-0.50	8	11	-57	-69	N	M, br
Butyl rubber	-3.15	11	16	-18	-11	Y	M, G
Natural rubber	-2.88	-13	-13	---	---	Y	D, M, P
Neoprene	-2.19	11	13	-9	-59	Y	G
Teflon	.69	---	---	-19	-18	Y	P, bl
Silicone rubber, 371	-.72	3	3	-70	-67	N	M
Silicone rubber, 751	-2.13	17	17	-65	-65	S	M
Kel-F 5500 Minn. Mining & Mfg. Co.	-.06	-1	-3	---	---	Y	D, P, bl
Kel-F 3700 Minn. Mining & Mfg. Co.	-.22	-1	5	-9	0	Y	D, M, bl
Run 2. Specimens immersed in sodium at 350° F for 168 hours							
Buna-N	-3.13	16	24	-59	-75	Y	M
Buna-S	-.96	18	25	-71	-94	N	M, P, br
Butyl rubber	-6.98	15	25	-12	-39	Y	M, P, br
Natural rubber	-4.98	-10	-10	---	---	Y	D, M
Neoprene	-2.69	11	13	-9	-59	Y	G
Teflon	-38.1	---	---	-73	-95	Y	E, bl
Silicone rubber, 371	-6.99	7	7	-67	-57	N	B, M
Silicone rubber, 751	-7.81	31	31	-58	-66	S	B
Nylon	-2.17	-1	-1	-64	-97	N	D
Irrathene	.81	---	---	---	---	Y	D
Run 3. Specimens immersed in potassium at 250° F for 238 hours							
Buna-N	1.39	21	29	-71	-60	N	M
Buna-S	-2.30	11	16	-22	-37	S	G
Butyl rubber	-5.12	19	12	-21	-53	S	M
Natural rubber	-6.2	33	53	---	---	Y	D
Neoprene	Decomposed completely						
Teflon	Decomposed completely						
Silicone rubber, 371	Extreme attack						
Silicone rubber, 751	Extreme attack						
Nylon	Decomposed completely						
Run 4. Specimens immersed in potassium at 250° F for 70 hours							
Buna-N	b-1.2	12	21	-31	-50	Y	M
Buna-S	b-1.6	6	10	-7	-15	Y	G
Butyl rubber	b-4.5	16	25	-20	-14	Y	G
Natural rubber	b-4.6	28	28	---	---	Y	D
Neoprene	b-3.4	7	10	-28	-47	Y	G
Teflon	b-1.9	---	---	-16	-18	Y	P, bl
Silicone rubber, 371	b-31	5	5	-26	3	Y	M
Silicone rubber, 751	b-33	21	21	-24	-30	Y	B, E
Nylon	b-3.7	---	---	-.6	-22	Y	P, wh
Kel-F 3700 (Vernay)	b-1.8	-1	0	-36	-2	Y	D, P, bl
Polypropylene	b-.7	---	---	-5	7	N	M
Run 5. Specimens immersed in rubidium at 245° F for 68 hours							
Buna-N	-0.1	3	6	---	---	Y	M
Buna-S	-.9	4	1	---	---	Y	G
Butyl rubber	-1.5	7	10	---	---	Y	G
Natural rubber	-2.4	0	0	---	---	Y	D
Neoprene	-.1	0	-1	---	---	Y	G
Kel-F 3700 (Vernay)	-.4	2	0	---	---	Y	P, br

aB bleached surface
 bl black
 br brown
 D darkened surface
 E pitted surface
 G appearance good
 G gummy
 M mottled surface
 N cracks in two or tending 100° or less
 P powdery surface deposits
 S surface cracks or tending 100° or less
 wh white
 Y tensile 100° without cracking.

Percent values for percent weight change multiplied by 100. Values for percent elongation multiplied by 100. Values for percent change in tensile strength multiplied by 100.



C-52936

Figure 1. - Material-compatibility test chamber.



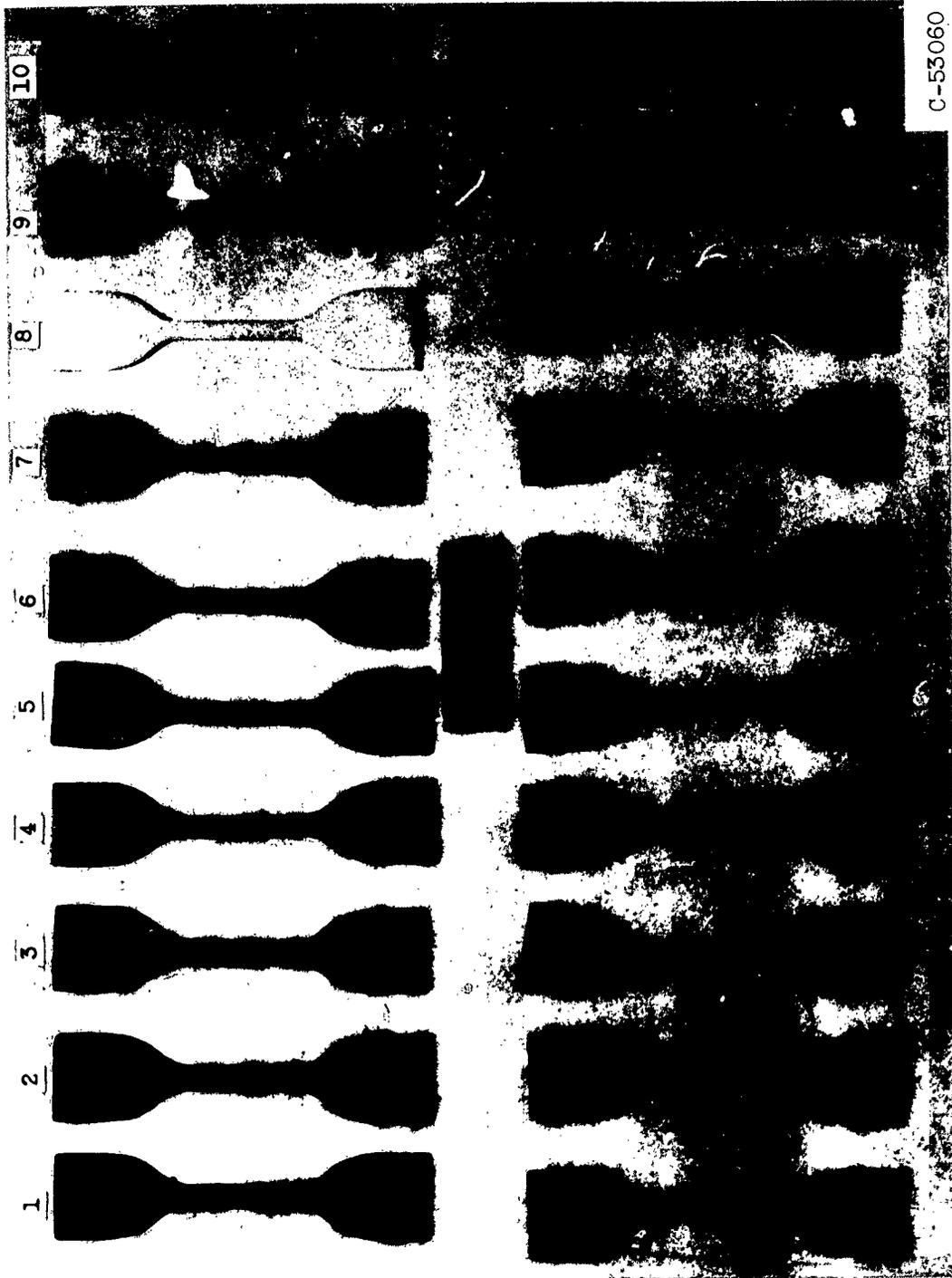
Figure 2. - Specimen holder.

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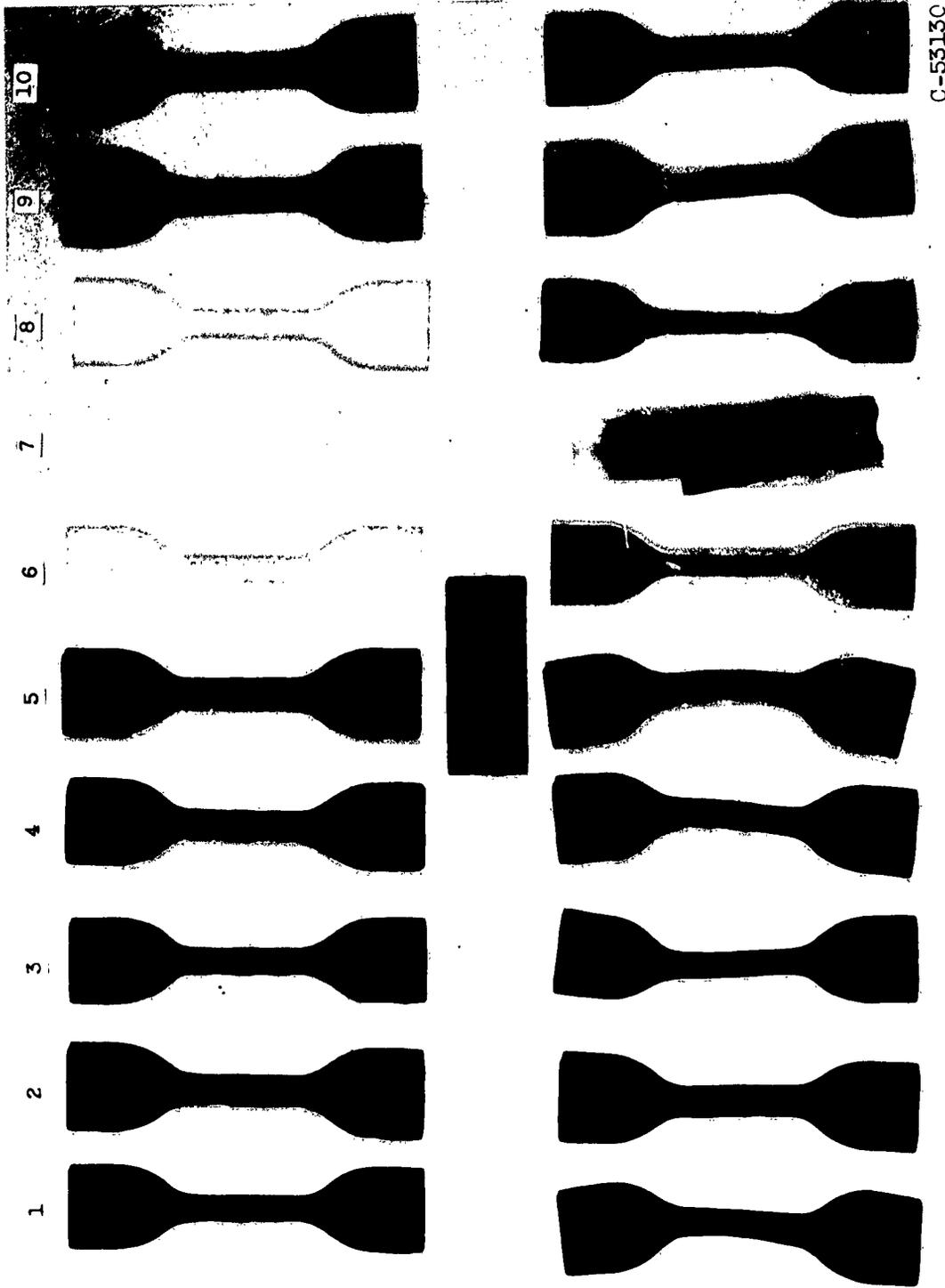
C-56499

Figure 3. - Rubidium-compatibility test chamber.



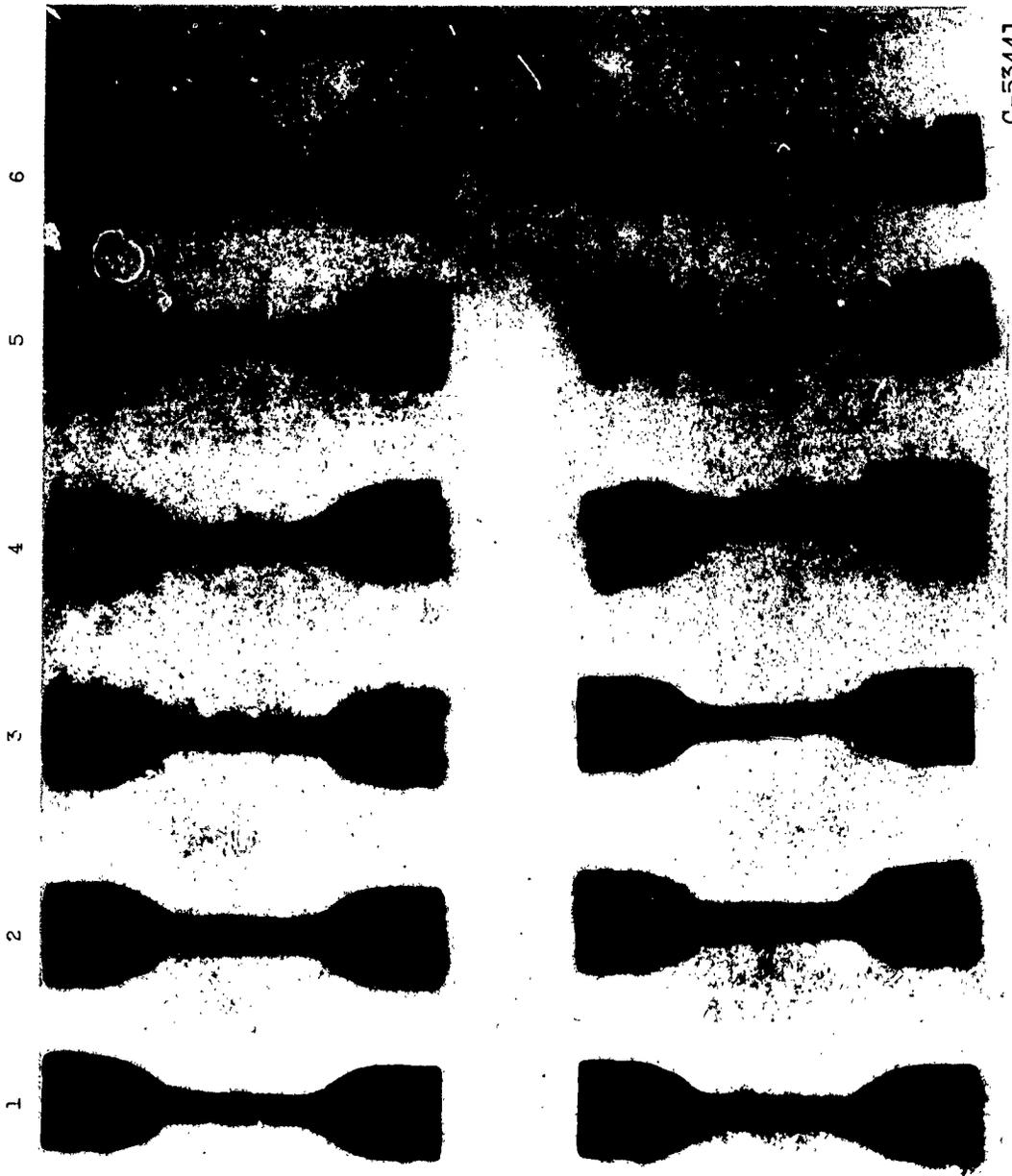
C-53060

Figure 4. - Run 1 specimens before (top row) and after (bottom row) immersion in sodium at 250° F for 219 hours. Specimens: 1, Buna N; 2, Buna S; 3, butyl rubber; 4, Kel-F 5500; 5, Kel-F 3700; 6, natural rubber; 7, neoprene; 8, Teflon; 9, silicone rubber, 371; 10, silicone rubber, 751.



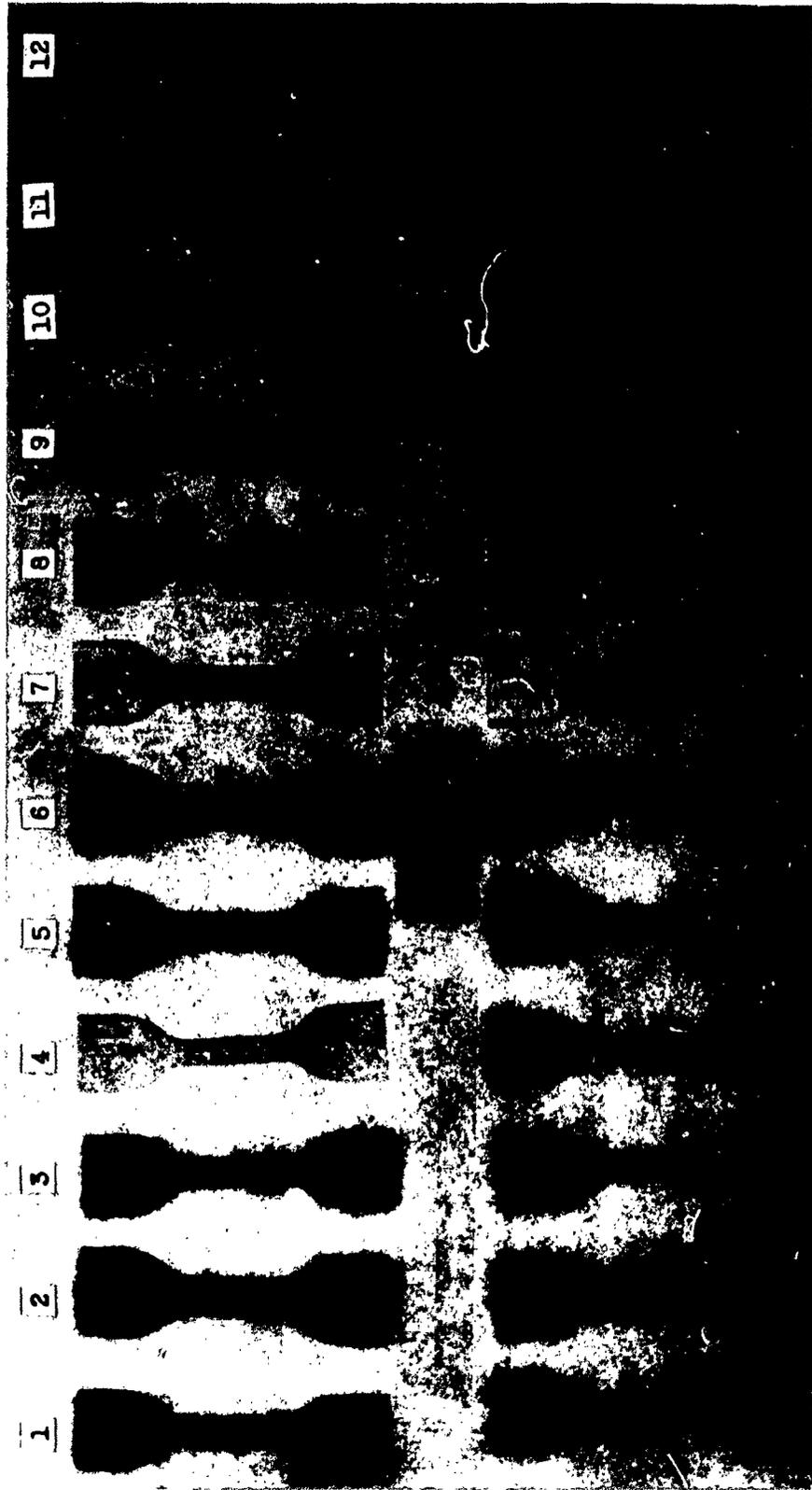
C-53130

Figure 5. - Run 2 specimens before (top row) and after (bottom row) immersion in sodium at 350 F for 169 hours. Specimens: 1, Buna N; 2, Buna S; 3, butyl rubber; 4, natural rubber; 5, neoprene; 6, nylon; 7, irradiated polyethylene; 8, Teflon; 9, silicone rubber, 371; 10, silicone rubber, 751.



C-53441

Figure 6. - Run 3 specimens before (top row) and after (bottom row) immersion in potassium at 250° F for 238 hours. Specimens: 1, Buna N; 2, Buna S; 3, butyl rubber; 4, natural rubber; 5, silicone rubber, 371; 6, silicone rubber, 751.



C-53241

Figure 7. - Put 4 specimens before (top row) and after (bottom row) immersion in potassium at 250 F for 70 hours. Specimens: 1, Buna N; 2, Buna S; 3, butyl rubber; 4, Kel-F 3700; 5, natural rubber; 6, neoprene; 7, nylon; 8, polypropylene; 9, silicone rubber, 317; 10, silicone rubber, 751 (with iron oxide additive); 11, silicone rubber, 751; 12, Teflon.

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