

**VULCANIZATION OF RUBBER WITH HIGH-INTENSITY
GAMMA RADIATION**

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FOREWORD

This report was prepared by the Organic Materials Branch under Project No. 7340, "Rubber, Plastic and Composite Materials", Task No. 73405, "Compounding of Elastomers", formerly RDO No. 617-12, "Compounding of Elastomers", and was initiated under the direction of the Materials Laboratory, Directorate of Research, Wright Air Development Center, with Mr. Wallace W. Jackson and Mr. Denver Hale acting as project engineers. Included among others who cooperated in this study was Mr. Donald Alvord of the Phillips Petroleum Company, Idaho Falls, Idaho.

This report covers work performed from May 1954 to March 1955.

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ABSTRACT

Techniques of vulcanizing both natural and synthetic rubber polymers, using high intensity gamma radiation are described.

This process utilizes the gamma radiation from Cobalt 60 or spent uranium reactor fuel elements and is accomplished without heat or chemical vulcanizing agents. Reinforcing agents, anti-oxidants and other additives are still required however, to obtain an optimum balance of physical properties.

After compounding the elastomers in the conventional manner and subjecting them to various dosages of gamma radiation, the resultant physical properties were determined.

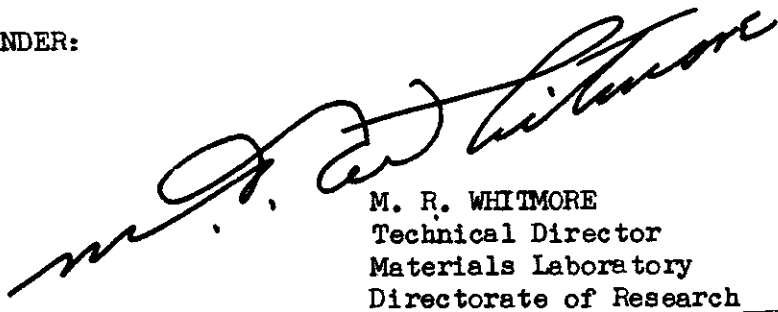
Evaluation of this technique shows considerable promise in developing elastomers for specialized uses, such as producing high temperature oil resistant rubber compounds and for improving the compression set of specialized rubber compounds.

This study further emphasizes the practical usefulness of atomic energy in providing the rubber technologist a new experimental variable or tool for determining fundamental properties of elastomeric compounds.

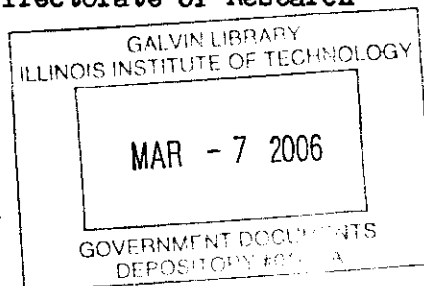
PUBLICATION REVIEW

This report has been reviewed and is approved.

FOR THE COMMANDER:



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SECTION I

DISCUSSION

In spite of recent large scale investigations of new rubber polymers for many highly specialized Air Force applications, generally satisfactory elastomeric compounds have not yet been produced. These applications for the most part require rubber end items resistant to fuel, oil, synthetic base hydraulic fluids or lubricants at extremely high or low temperatures. It has therefore, become necessary to increase the effort for new and improved compounding procedures and vulcanizing techniques for the existing polymers so that more satisfactory performance can be obtained from them.

Vulcanization of rubber was first discovered by Charles Goodyear in 1839. Since that time many refinements of this technique including the discovery and production of a wide variety of synthetic rubber polymers have followed. At the present time, the formulating and processing of natural and synthetic rubber constitute a highly developed art encompassing the use of numerous special compounding materials.

In accordance with most widely accepted theories, vulcanization is primarily a cross-linking process. This cross-linking is stated to occur at points in the rubber molecule which are reactive to such known vulcanizing agents and accelerators as sulfur, sulfur derivatives, metallic oxides, amines and the like under the reaction conditions involving heat, time, amount and type of agent.

The fact that some materials can be vulcanized more readily under a given set of conditions than others is explained in terms of differences in the nature and structure of the polymeric molecule. Natural rubber, for example contains unsaturation to an extent such that vulcanization by means of sulfur can be varied over a wide range. In the making of soft natural rubber goods the amount of sulfur used during vulcanization rarely exceeds 4%. Additional amounts up to as high as a maximum of 47% of its weight may be employed wherein complete saturation of the double bonds occurs and hard rubber or ebonite is formed.

At the present time relatively few of the reaction mechanisms which are involved in the vulcanization of rubber are precisely understood. It is well known, however, that most variations from a given procedure markedly influence the final product.

It has been found, through this study, that uncured vulcanizable elastomers may be cross-linked in the absence of chemical vulcanizing agents and without the use of high curing temperatures. The products obtained closely resemble the chemical vulcanizates in many respects and offer considerable promise in developing elastomers for specialized uses. By subjecting the otherwise compounded base polymer to the action of a high energy gamma radiation source not only can the elastomer be cross

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linked or vulcanized, but the extent of change produced can be controlled directly by adjustment of the radiation dosage.

This cross-linking effect has been noted by previous observers using other sources of irradiation such as X-ray, particle accelerators, etc. Davidson and Geib noted a slight curing action of pile irradiation on natural rubber. Charlesby through pile irradiation studies on certain elastomers has shown that the degree of cross linking is directly proportional to radiation dose.

Gamma radiation is particularly desirable in this study for four reasons:

- (1) High energy gamma rays have great penetrating power and, hence the elastomers are vulcanized uniformly.
- (2) The utilization of Cobalt 60 with proper shielding greatly reduces radiation hazards.
- (3) No residual radioactivity is imparted to the treated elastomer.
- (4) After initial installation of the source together with the proper shielding, maintenance problems are limited as compared to those encountered with X-ray equipment or particle accelerators.

Although no effort is made in this study to determine the specific physico-chemical changes for each polymer, a review of the most recent concepts of the effects of atomic radiations on high polymers aids considerably in a more fundamental understanding of this process.

These effects as described by Sun usually lead to one or more of the following:

- (1) Breaking of chemical bonds with free radical formation.
- (2) Dissociation or degradation of the molecule.
- (3) Re-arrangement of the molecule.

Consequently chemical reactions are initiated. These include gas liberation, double bond formation and elimination, degradation, polymerization, cross-linking, vitrification, hydrogenation and others.

Initial studies included an evaluation of this gamma ray vulcanization technique with respect to the ability to vulcanize a wide range of rubber polymers. These polymers contained conventional amounts of filler to enhance their most useful properties. A comparison of heat aging characteristics of some compounds was made with those obtainable from similar compounds using conventional chemical vulcanization procedures.

Using four representative polymers compounded with varying amounts and types of filler stress-strain changes and changes in other physical properties were noted over varying dosages of gamma radiation. The polymers included natural, GRS, Neoprene WRT and Hycar 1001 (buna N). The fillers used in 20, 40 and 60 parts by weight were Philblack A, MT Thermax, hard processing channel black, Silene EF and Kalvan. Gamma radiation dosages were 10^7 , 3×10^7 and 4×10^7 Roentgens .

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Volume swell and studies on plasticizer extractability were performed on gamma ray vulcanized buna N compounds (Paracril 18) varying in types and amounts of plasticizers. These included dioctyl sebacate, TP90-B, tributoxyethyl phosphate and butyl oleate.

Compression set studies on Hycar 1001 compounds containing 0, 20 and 60 parts of Philblack A were also made using gamma dosages of 10^7 Roentgens.

A discussion of these results is found in the summary. Compounding formulas and test data are shown in the tables.

SECTION II

IRRADIATION TECHNIQUE

Two sources of high energy gamma radiation employed in this study were Cobalt 60 and spent uranium fuel elements. Cobalt 60 is particularly desirable on the basis of its current availability and the simplicity of shielding which greatly reduces health hazards involved.

Portions of this work were conducted at the Materials Test Reactor at the National Reactor Testing Station. When spent uranium fuel elements are removed from the reactor they are usually stored in a deep water canal and allowed to "cool". These elements may be arranged to give a uniform gamma ray field above or inside the submerged fuel element assembly. The gamma fluxes from this source were determined calorimetrically the average dose rate being 3×10^6 Roentgens per hour. The Cobalt 60 source used in this study is located at the Materials Laboratory, Wright Air Development Center. The present dose rate is approximately 5.2×10^5 Roentgens per hour.

The radiation exposures of the elastomers have been expressed in calculated Roentgen values. The Roentgen unit is defined as essentially equivalent to the absorption of 83.8 ergs of energy from gamma radiation in one gram of dry air. For the same radiation field, the amount of energy absorbed varies with the material irradiated, for example in the case of hydrocarbons, the absorption value in a radiation field of one Roentgen is 93 instead of 83.8 ergs.

The dosages to which the elastomers were subjected were regulated by controlling the exposure time and thus were computed by multiplying the gamma flux in Roentgens per hour by the exposure time in hours.

Although temperatures above and below room temperature may be employed, the use of room temperature or thereabouts is preferred in view of the simplicity of operation. Temperatures were maintained at 30°C to

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eliminate fluctuations and variations that could possibly be attributed to differences in temperature.

A sectional view of a conventional Cobalt 60 "pig" is shown in Fig. 1. A chain hoist is used for charging and removing the 1.69 inch diameter aluminum canister in which the aluminum molds are placed for irradiation. Fig. 2 shows the aluminum specimen molds for the elastomeric materials to be irradiated in the source. The overall dimensions of the molds are 4 x 1 x 5/16 inches.

SECTION III

COMPOUNDING AND FABRICATION TECHNIQUE

Using a standard 6 inch rubber mill, 100 parts of each polymer were pre-masticated to uniform breakdown. The fillers and/or plasticizers were slowly added and the mixture cut several times to insure homogeneity and sheeted off the mill at 0.050 ± 0.005 inches. The elastomer was then placed in the aluminum mold as shown in Fig. 2. Compression was maintained by Allen screws. The molds were then placed in the aluminum canister and loaded in the Cobalt 60 source as shown in Fig. 1.

The following general formulas were used for all stocks:

<u>I</u>		<u>II</u>	
Polymer	100	Polymer	100
Filler	0-60	Filler	0-60
		Plasticizer	16, 26

The amounts used were based on a total of 100 parts by weight of polymer.

SECTION IV

TESTING PROCEDURES

Physical Properties:

Using standard test procedures as outlined in ASTM or Federal Specification ZZ-R-601a tensile strength, elongation, modulus, hardness, compression set and volume swell in 70/30 isooctane-toluene mixture were determined.

Plasticizer Extractability:

Tests for plasticizer extractability were conducted by determining the percent weight loss after immersion in 70/30 isooctane-toluene mixture for 96 hours at room temperature and dryout for 48 hours in air at 158°F.

Calculations for percent weight loss are as follows:

$$\frac{W_1 - W_2}{W_1} \times 100 = \% \text{ Weight Loss}$$

W_1 = initial weight

W_2 = weight after immersion and dry out.

SUMMARY

Although the elastomers from this process appear to be modified in a manner closely related to the chemical vulcanizates in many respects, tests indicate that the modifications in most cases provide elastomers having improved aging characteristics.

Attempts to initiate vulcanization in polyisobutylene elastomers proved unsuccessful since polymers of this type readily undergo depolymerization at relatively low dosages of gamma radiation.

Many experimental polymers which offer considerable resistance to vulcanization by chemical means may be readily vulcanized by this process. A specific example of a polymer of this type is seen in Table II, wherein the response of N5400 elastomer (experimental acrylate developed by Monsanto Chemical Company) to irradiation is shown to be effective after relatively low dosage levels despite the fact that the particular polymer is greatly resistant to chemical vulcanization. A chemical vulcanizate was prepared after considerable difficulty and is shown for purpose of comparison with the irradiated product.

Comparative dry heat aging data at 350°F between the two types of vulcanizates indicate a complete loss of rubber-like properties (embrittlement) of the chemically vulcanized N5400 elastomer in 8 hours while the gamma ray vulcanized product attained a similar condition in 36 hours. Oil aging properties at 350°F in synthetic base diester oil also showed a definite superiority of the irradiated product. The chemical vulcanizate exhibited a complete loss of rubber-like properties in 24 hours. The physical properties of the irradiated product after 72 hours oil aging are shown in Table II.

Table III shows a buna N (Hycar 1001) compound irradiated to a dosage of 10^7 Roentgens. Comparative dry heat aging data with a chemically vulcanized compound indicated a complete loss of rubber-like properties in both compounds after air aging 24 hours at 350°F. The degree of brittleness was substantially less in the case of the irradiated vulcanizate, however, on the basis of a 180° bend test.

Figures 3 thru 6 graphically represent the effect of gamma radiation dosages on the tensile strength and elongation percent for four representative elastomers consisting of natural, buna N (Hycar 1001), Neoprene WRT and GRS polymers. Each composition consists of 100 parts of the base polymer and 45 parts by weight of Philblack A. The response to specific dosages is seen to be not the same for the elastomers, supporting previous observations that polymers of differing chain lengths or those containing the benzene ring in their structure, etc. possess varying degrees of resistance to the effect of high energy radiation. The changes in tensile strength and elongation noted in these illustrations after increased gamma ray dosages closely follow those encountered using conventional chemical vulcanization procedures.

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It has been found that dosages ranging from 10^7 and up to about 8×10^7 Roentgens may be employed for some elastomers. An effective dosage is within the range of 2×10^7 and 6×10^7 Roentgens. Optimum results for materials such as natural rubber and several of the synthetic elastomers are indicated at dosage levels of approximately 3 to 5×10^7 Roentgens.

As may be seen in Tables IV thru XIII different types and amounts of fillers also influence the rate and extent of change in physical properties of natural, neoprene, GRS and buna N rubber at various dosage levels.

Analysis of the stress-strain data noted in Tables IV thru IX indicates certain changes which, in some respects follow those encountered in conventional vulcanization procedures. The modulus at 150 percent of all the elastomers increases with increased concentration of filler and as the dosage within the range of 10^7 to 4×10^7 Roentgens is increased. Few exceptions are noted in some compounds showing a slight decrease in modulus at 4×10^7 Roentgens. In comparing the reinforcing properties of hard processing channel black with those of Philblack A generally higher tensile elongation and modulus properties are noted in most compounds using Philblack A. Further examination of the tensile - elongation properties, shown in Tables X thru XIII of these four polymers indicate that as the concentration of filler is increased the tensile properties are increased. Certain irregularities are noted wherein the elongation also increases with increasing amounts of filler. Decreases in elongation are noted in a number of compounds as the gamma radiation dosage is increased within the range of 10^7 and 3×10^7 Roentgens.

Other unique physical and functional properties of elastomers prepared by this process are noted in the compression set data shown in Table XIV. Buna N elastomers containing 0, 20 and 60 parts of Philblack A were irradiated to a dosage of 10^7 Roentgens and the compression set results compared with similar chemically vulcanized buna N elastomers. The compression set after 70 hours at 250°F is seen to be considerably less in the case of the irradiated compounds.

Studies on limiting plasticizer extractability by fuel from a buna N compound containing 10 and 15 percent of various types of plasticizers are shown in Table XV. Limitations in plasticizer extraction by 70/30 isooctane toluene are observed, however, only after considerably high dosages of gamma radiation which to date have resulted in an unfavorable balance of physical properties. Table XVI shows a decrease in volume swell of similar compounds irradiated at dosages of 4×10^7 and 6×10^7 Roentgens. Comparative results of similar chemically vulcanized compounds are also shown in this table.

The results of this study, while not conclusive, indicate areas for further investigation. While commercial feasibility of this process has not been studied yet, its usefulness in providing to the rubber and plastics industry a unique tool for obtaining fundamental information should be emphasized.

BIBLIOGRAPHY

1. Davidson, W. L. and Geib, I. G. - Effects of File Bombardment on Uncured Elastomers, J. Appl. Phys., 19, p427, (May 1948)
2. Charlesby, A. - Crosslinking of Rubber by File Radiation, Atomics 5, p12, (January 1954)
3. Sun, K. H. - Effects of Atomic Radiation on High Polymers, Modern Plastics, (September 1954)

TABLE I

Compounding Data and Physical Properties of Representative Elastomers
Containing Conventional Amounts and Types of Filler and Irradiated as Indicated

POLYMER	Parts	SRF	Philblack A	HAF Black	Dosage	Tensile psi	Elong %	Hardness
Natural	100	45	-	-	4 x 10 ⁷ r	2600	320	40
Neoprene WRT	100	45	-	-	1 x 10 ⁷ r	2650	360	66
Hycar 1001	100	-	45	-	1 x 10 ⁷ r	2640	200	75
GRS	100	-	60	-	1 x 10 ⁷ r	880	500	56
FBA ¹	100	-	-	35	1 x 10 ⁷ r	730	300	66
EA/AN/VTS ²	100	-	-	35	1 x 10 ⁷ r	1730	220	71
Kel F (Elastomer)	100	-	-	35	1 x 10 ⁷ r	1530	470	68
N5400 ³	100	-	-	35	1 x 10 ⁷ r	1330	170	60
Thiokol ST	100	45	-	-	1 x 10 ⁷ r	330	280	51
Silastic 250	100	-	-	-	1 x 10 ⁷ r	860	130	70

- 1 - Poly 1,1, dihydroperfluorobutyl acrylate
- 2 - Terpolymer of ethyl acrylate/acrylonitrile and vinyltriethoxy silane developed by Bjorksten Research Laboratories.
- 3 - Experimental acrylate developed by Monsanto Chemical Company.

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TABLE II

Comparative Heat Aging Properties of Gamma Ray
Vulcanized and Chemically Vulcanized N5400 Elastomers

<u>Gamma Ray Vulcanized</u>		<u>Chemically Vulcanized</u>	
N5400	100 parts	N5400	100 parts
HAF	35 parts	Stearic Acid	3 parts
Gamma ray dosage	$1 \times 10^7 r$	Magnesium Oxide	2 parts
		Ethyl Zimate	0.75 parts
		Cure 20 min. at 400°F	

Original Properties

Tensile	1330 psi	1140 psi
Elongation	170%	200%
Shore A Hardness	60	60

Dry Air Aging at 350°F

embrittlement after 36 hours

embrittlement after 8 hours

Properties after aging 72 hours in MIL-L-7808 engine oil at 350°F

Tensile	575 psi	
Elongation	60%	embrittlement
Shore A Hardness	85	

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TABLE III

Comparative Heat Aging Properties of Gamma Ray
Vulcanized and Chemically Vulcanized Hycar 1001 Elastomers

<u>Gamma Ray Vulcanized</u>		<u>Chemically Vulcanized</u>	
Hycar	100 parts	Hycar 1001	100 parts
Philblack A	35 parts	Zinc Oxide	5 parts
Gamma Ray dosage	1 x 10 ⁷ r	Stearic Acid	1.5 parts
		Sulfur	1.5 parts
		Altax	1.5 parts
		Philblack A	35 parts
		Cure 20' at 310°F	

Original Properties

Tensile	2640 psi	2900 psi
Elongation	200%	350%
Shore A Hardness	75	84

Properties after aging 24 hours in MIL-L-7808 Engine Oil at 350°F

Both samples brittle, however, degree of brittleness substantially less in the irradiated vulcanizate on the basis of 180 degree flat bend test.

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TABLE IV

Stress-Strain Data for 4 Polymers Using No Filler

Polymer	150% Modulus (psi) after 3 Radiation Doses		
	$1 \times 10^7 r$	$3 \times 10^7 r$	$4 \times 10^7 r$
Natural	54	108	120
GRS	107	142	149
Neoprene WRT	147	195	216
Hycar 1001	185	284	393

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TABLE V

Stress-Strain Data for 4 Polymers
Using Various Concentrations of Philblack A

Polymer	Parts Filler/100 of Polymer	<u>150% Modulus psi after 3 Radiation Doses</u>		
		<u>1 x 10⁷r</u>	<u>3 x 10⁷r</u>	<u>4 x 10⁷r</u>
Natural	20	178	153	184
	40	269	272	334
	60	500	538	383
GRS	20	106	261	285
	40	158	457	724
	60	209	715	909
Neoprene WRT	20	257	498	553
	40	665	1260	1970
	60	1182	2009	-
Hycar 1001	20	290	612	1020
	40	582	1610	2020
	60	1065	1620	-

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TABLE VI

Stress-Strain Data for 4 Polymers
Using Various Concentrations of MT Thermax

Polymer	Parts Filler/100 of Polymer	<u>150% Modulus (psi) after 3 Radiation Doses</u>		
		<u>1 x 10⁷r</u>	<u>3 x 10⁷r</u>	<u>4 x 10⁷r</u>
Natural	20	65	102	110
	40	62	135	119
	60	81	211	172
GRS	20	78	118	108
	40	82	129	192
	60	93	137	233
Neoprene WRT	20	190	253	329
	40	141	335	511
	60	232	435	802
Hycar 1001	20	254	406	478
	40	278	636	723
	60	401	959	-

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TABLE VII

Stress-Strain Data for 4 Polymers
Using Various Concentrations of HPC (Channel Black)

Polymer	Parts Filler/100 of Polymer	<u>150% Modulus (psi after 3 Radiation Doses)</u>		
		<u>1 x 10⁷r</u>	<u>3 x 10⁷r</u>	<u>4 x 10⁷r</u>
Natural	20	58	128	103
	40	198	273	307
	60	241	304	407
GRS	20	98	146	186
	40	150	382	293
	60	358	602	585
Neoprene WRT	20	252	456	1069
	40	662	890	-
	60	1070	-	-
Hycar 1001	20	367	665	635
	40	565	1320	1420
	60	725	2145	1960

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TABLE VIII

Stress-Strain Data for 4 Polymers
Using Various Concentrations of Silene EF

Polymer	Parts Filler/100 of Polymer	<u>150% Modulus (psi) after 3 Radiation Doses</u>		
		<u>1 x 10⁷r</u>	<u>3 x 10⁷r</u>	<u>4 x 10⁷r</u>
Natural	20	97	163	192
	40	91	164	209
	60	119	230	353
GRS	20	98	170	159
	40	148	297	268
	60	249	549	459
Neoprene WRT	20	260	-	-
	40	524	-	-
	60	852	-	-
Hycar 1001	20	254	539	492
	40	457	725	787
	60	742	1255	1090

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TABLE IX

Stress-Strain Data for 4 Polymers
Using Various Concentrations of Kalvan

Polymer	Parts Filler/100 of Polymer	<u>150% Modulus (psi) after 3 Radiation Doses</u>		
		<u>1 x 10⁷r</u>	<u>3 x 10⁷r</u>	<u>4 x 10⁷r</u>
Natural	20	83	112	141
	40	99	133	279
	60	122	146	404
GRS	20	88	164	139
	40	100	202	183
	60	108	185	278
Neoprene WRT	20	175	-	-
	40	176	-	-
	60	337	-	-
Hycar 1001	20	233	372	600
	40	311	532	-
	60	433	799	-

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TABLE X

Tensile-Elongation Properties of Natural Rubber
Using Various Types and Amounts of Filler

Filler	Parts Filler/100 of Polymer	Tensile (psi), Elongation (%) after 3 Radiation Doses					
		1 x 10 ⁷ r		3 x 10 ⁷ r		4 x 10 ⁷ r	
		T	E	T	E	T	E
Philblack A	20	256	200	630	390	307	390
	40	500	230	1845	400	1809	310
	60	702	310	2030	330	1690	290
MT Thermax	20	167	670	492	505	502	500
	40	217	620	750	475	1150	535
	60	410	545	1630	495	1995	545
HPC (Channel black)	20	139	445	612	280	738	430
	40	389	255	750	275	1050	235
	60	893	400	1630	210	1947	330
Silene EF	20	239	475	722	445	1042	191
	40	503	510	947	410	1517	209
	60	835	485	1465	420	1725	354
Kalvan	20	137	465	300	310	287	440
	40	249	490	780	450	725	405
	60	542	555	670	370	1607	420

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TABLE XI

Tensile-Elongation Properties of GRS Rubber
Using Various Types and Amounts of Filler

Filler	Parts Filler/100 of Polymer	Tensile (psi), Elongation (%) after 3 Radiation Doses					
		1 x 10 ⁷ r		3 x 10 ⁷ r		4 x 10 ⁷ r	
		T	E	T	E	T	E
Philblack A	20	393	1035	1047	410	935	320
	40	385	750	1055	340	1495	235
	60	343	580	1355	240	1360	190
MT Thermax	20	375	1345	650	1060	143	170
	40	298	1310	239	405	562	545
	60	334	1340	749	1080	730	585
HPC (Channel Black)	20	480	1340	1000	725	812	395
	40	1035	855	2395	620	1430	470
	60	952	585	1730	365	1600	335
Silene EF	20	415	1160	592	635	471	465
	40	665	1040	1130	590	1240	770
	60	920	826	1330	440	1532	660
Kalvan	20	253	1195	340	495	357	680
	40	373	1270	406	485	386	620
	60	246	1370	710	990	903	635

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TABLE XII

Tensile-Elongation Properties of Neoprene WRT Rubber
Using Various Types and Amounts of Filler

Filler	Parts Filler/100 of Polymer	Tensile (psi), Elongation (%) after 3 Radiation Doses					
		1 x 10 ⁷ r		3 x 10 ⁷ r		4 x 10 ⁷ r	
		T	E	T	E	T	E
Philblack A	20	2255	620	1595	305	1485	240
	40	2590	367	2300	210	1965	155
	60	2355	245	2160	155	2055	100
MT Thermax	20	1620	800	1320	640	440	195
	40	1580	890	1215	415	1000	235
	60	1880	950	1485	590	1222	210
HFC (Channel black)	20	1700	490	1150	275	1079	160
	40	2115	340	1345	175	1165	100
	60	2010	225	1570	100	1405	70
Silene EF	20	1045	655	489	100	334	70
	40	1090	390	685	80	550	50
	60	1150	285	855	60	570	20
Kalvan	20	492	525	310	130	279	60
	40	508	400	288	90	234	40
	60	1090	730	555	80	398	40

TABLE XIII

Tensile-Elongation Properties of Hycar 1001 Rubber
Using Various Types and Amounts of Filler

Filler	Parts Filler/100 of Polymer	Tensile (psi), Elongation (%) after 3 Radiation Doses					
		1 x 10 ⁷ r		3 x 10 ⁷ r		4 x 10 ⁷ r	
		T	E	T	E	T	E
Philblack A	20	1535	510	1250	200	1160	175
	40	2650	425	2680	210	2430	180
	60	2770	335	2430	210	2620	130
MT Thermax	20	870	460	562	190	470	155
	40	1185	525	1040	180	1060	190
	60	1575	625	1130	180	820	100
HFC (Channel black)	20	1430	640	1130	205	1850	255
	40	2025	630	2340	180	2380	205
	60	2715	545	3100	190	2930	195
Silene EF	20	1400	635	1145	270	1290	315
	40	1780	560	1610	300	1435	265
	60	2485	595	2220	305	2320	295
Kalvan	20	1125	620	1135	405	880	200
	40	1110	475	867	225	570	130
	60	2390	570	1212	250	1080	100

TABLE XIV

Compression Set Data for Gamma Ray Vulcanized
and Chemically Vulcanized Hycar 1001

Gamma Ray Vulcanized

Compound No.	<u>1</u>	<u>2</u>	<u>3</u>
Hycar 1001	100	100	100
Philblack A	0	20	60
Gamma ray dosage $1 \times 10^7 r$			
Compression Set (%) after 70 hours at 250°F	20.2	55.5	53

Chemically Vulcanized

Compound No.	<u>4</u>	<u>5</u>	<u>6</u>
Hycar 1001	100	100	100
Stearic Acid	1.5	1.5	1.5
Zinc Oxide	5	5	5
Sulfur	1.5	1.5	1.5
Altax	1.5	1.5	1.5
Philblack A	0	20	60
Cure 35 min. at 310°F			
Compression Set (%) after 70 hours at 250°F	64.5	80	77.5

TABLE XV

Plasticizer Extractability Studies on Gamma Vulcanized Paracril 18 Polymer Containing Four Plasticizers

Compound No.	1	2	3	4	5	6	7	8
Paracril 18	100	100	100	100	100	100	100	100
TP90B	16	26	-	-	-	-	-	-
Tributoxyethyl phosphate	-	-	16	26	-	-	-	-
Butyl oleate	-	-	-	-	16	26	-	-
Di-2 ethylhexyl sebacate	-	-	-	-	-	-	16	26
Philblack A	45	45	45	45	45	45	45	45
Percent weight loss after aging 96 hours in 70/30 isooctane/toluene at room temperature then dry-out 24 hours at 158°F after								
Gamma dose 4 x 10 ⁷ r	7.34	16.5	8.12	16.5	8.1	16	7.7	17.8
Gamma dose 6 x 10 ⁷ r	3.65	-	6.47	-	4.46	-	4.5	-

Contrails

TABLE XVI

Comparative Volume Swell Data, on Chemically Vulcanized
and Gamma Ray Vulcanized Paracril 18 Compounds

Chemically Vulcanized

Paracril 18	100	100	100	100
Zinc oxide	5	5	5	5
Stearic Acid	1.5	1.5	1.5	1.5
Sulfur	1.5	1.5	1.5	1.5
Altax	1.5	1.5	1.5	1.5
Philblack A	45	45	45	45
TP90B	17.5	-	-	-
Tributoxyethyl phosphate	-	17.5	-	-
Butyl oleate	-	-	17.5	-
Di-2 ethylhexyl sebacate	-	-	-	17.5

Cure 20 minutes at 310°F

Volume Swell (%) after aging 70 hours at room temperature in
70/30 isooctane/toluene

	46	42.1	43	45
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Gamma Vulcanized

Paracril 18	100	100	100	100
Philblack A	45	45	45	45
TP90B	16	-	-	-
Tributoxyethyl phosphate	-	16	-	-
Butyl oleate	-	-	16	-
Di-2 ethylhexyl sebacate	-	-	-	16

Volume Swell (%) after aging 70 hours at room temperature in 70/30
isooctane/toluene after

Gamma dose	4 x 10 ⁷ r	37.2	38.5	38.4	43
Gamma dose	6 x 10 ⁷ r	35.2	28	35.1	35.6

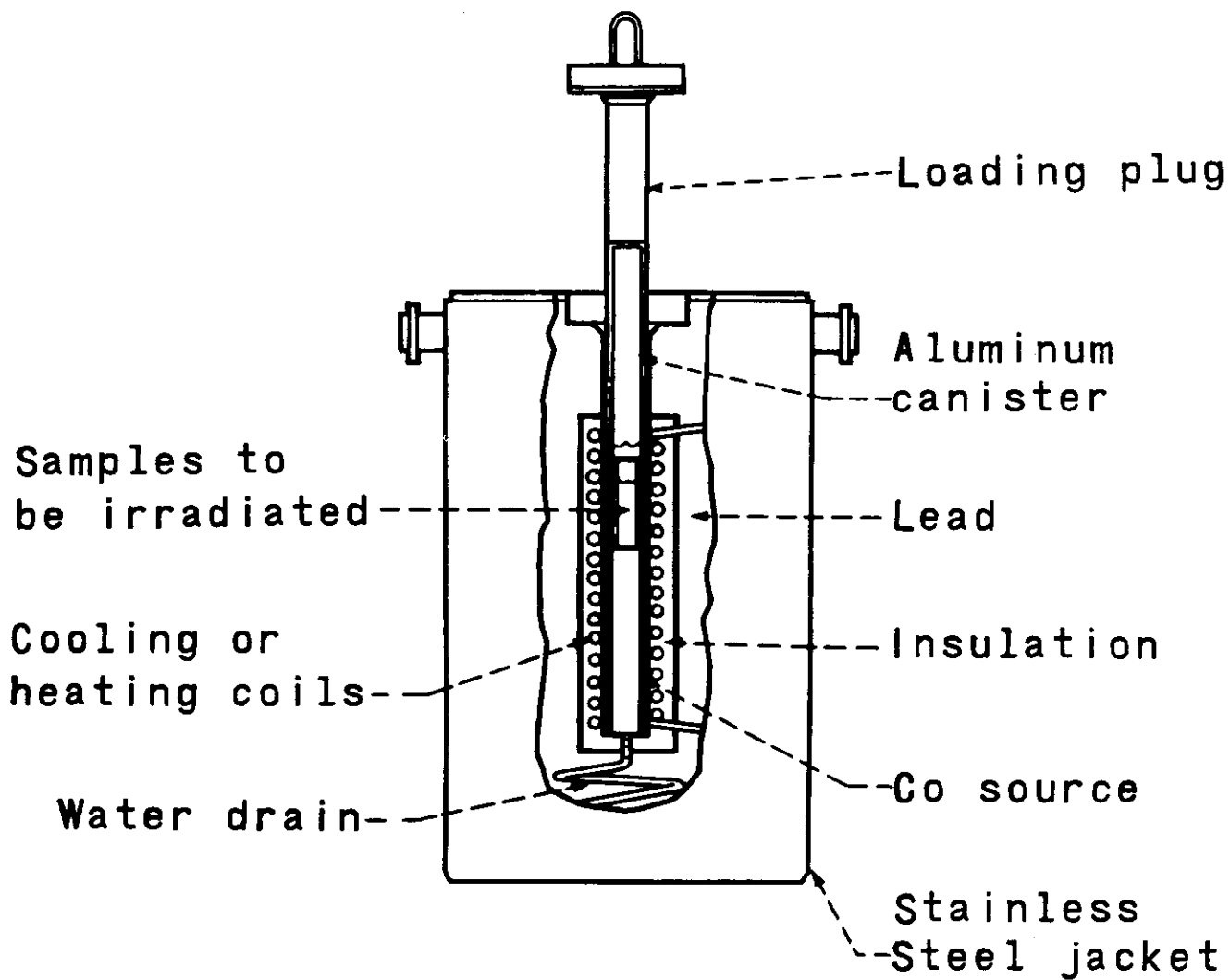


FIG.1. SECTIONAL VIEW OF A
COBALT-60 "PIG"

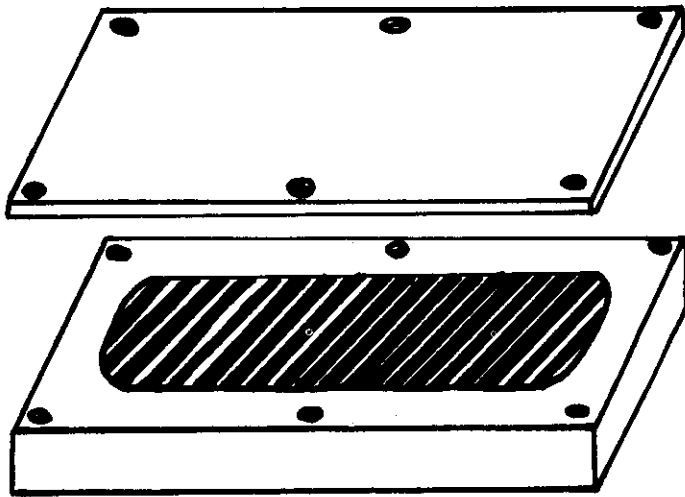
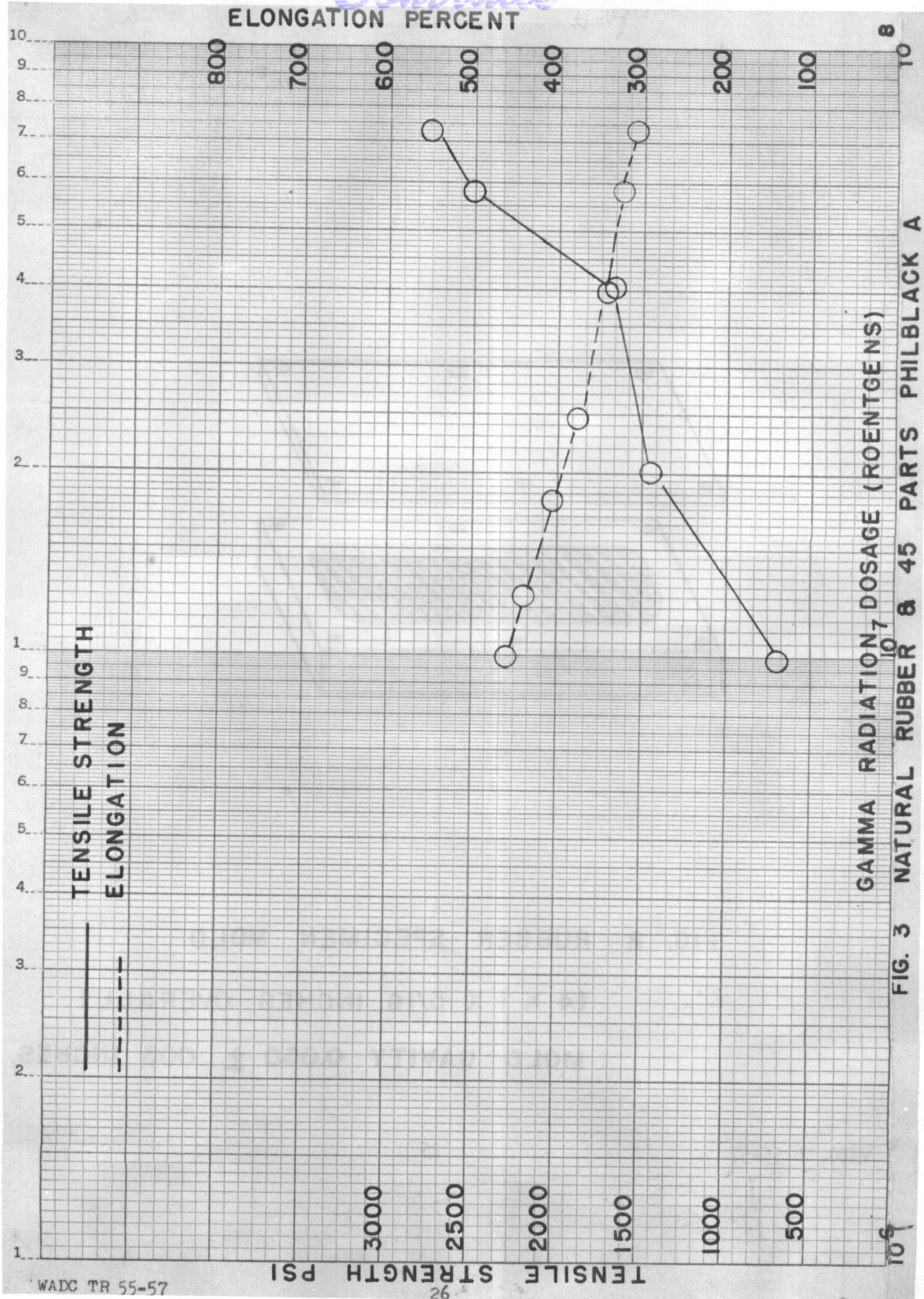


FIG. 2 RUBBER SPECIMEN MOLD
(4 X 1 X 5/16 INCHES OVERALL)
MOLD CAVITY 0.050 ± .005 INCHES.

Contrails



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Contrails

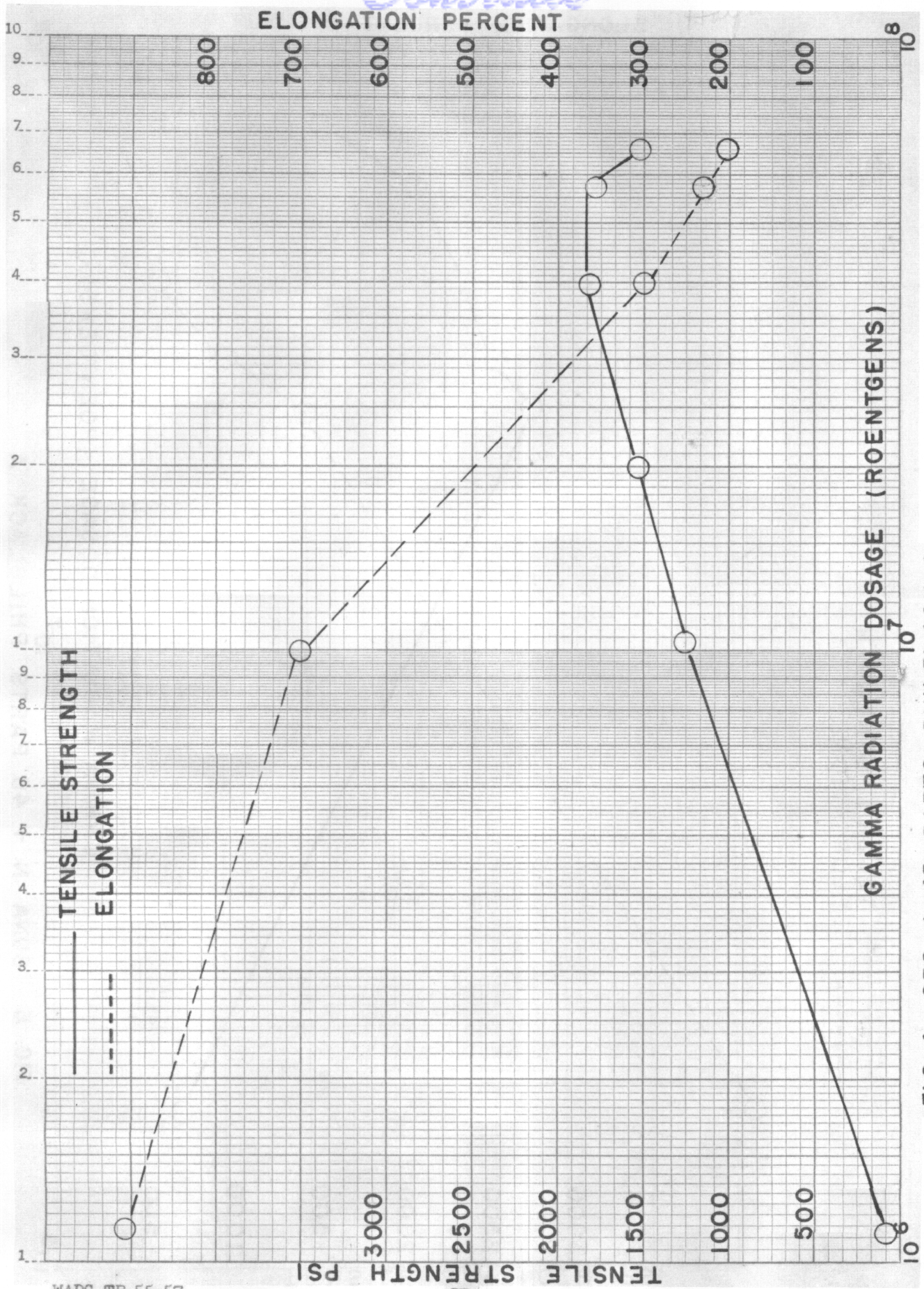
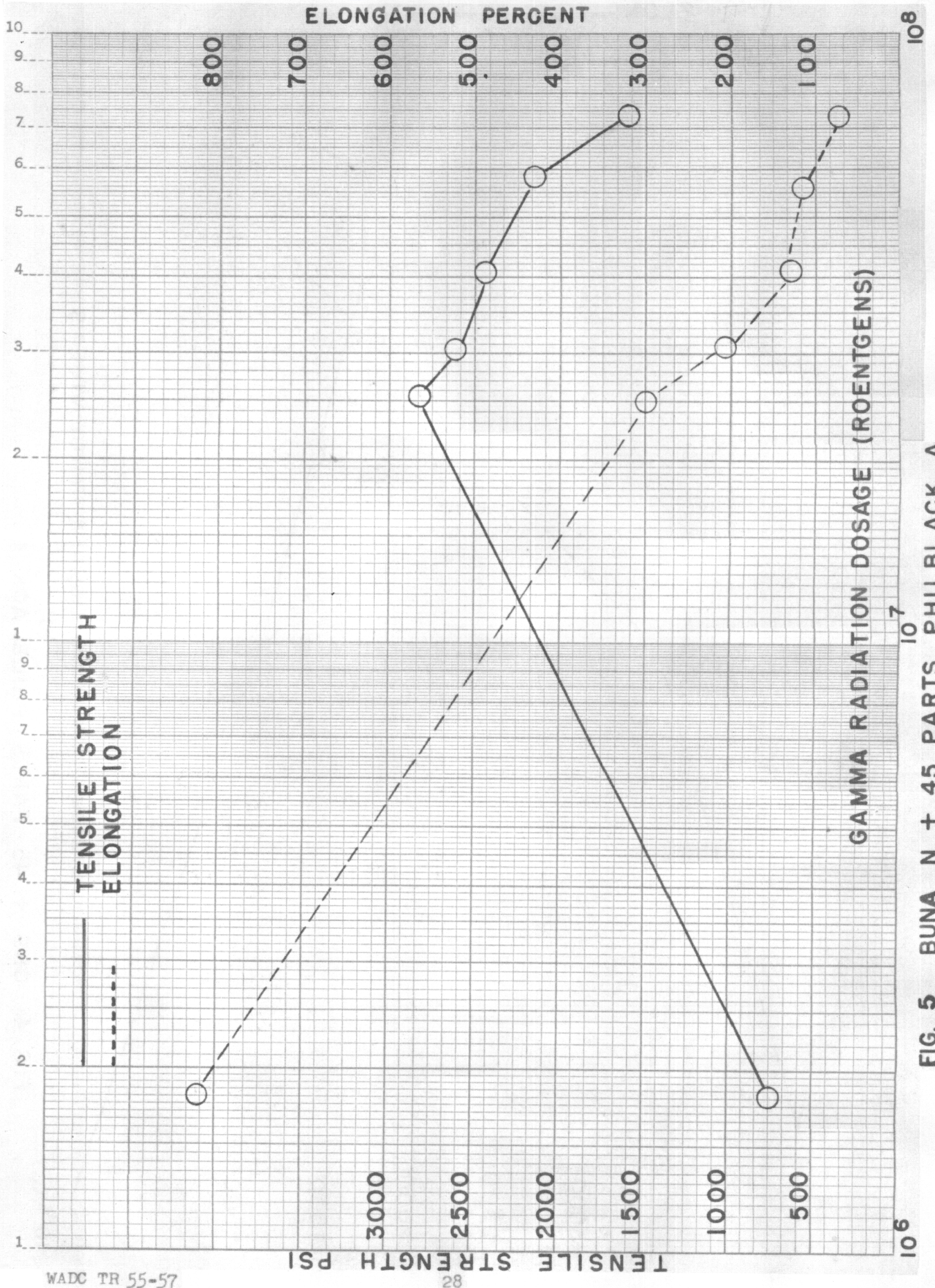


FIG. 4 GRS + 45 PARTS PHILBLACK A



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FIG. 5 BUNA N + 45 PARTS PHILBLACK A

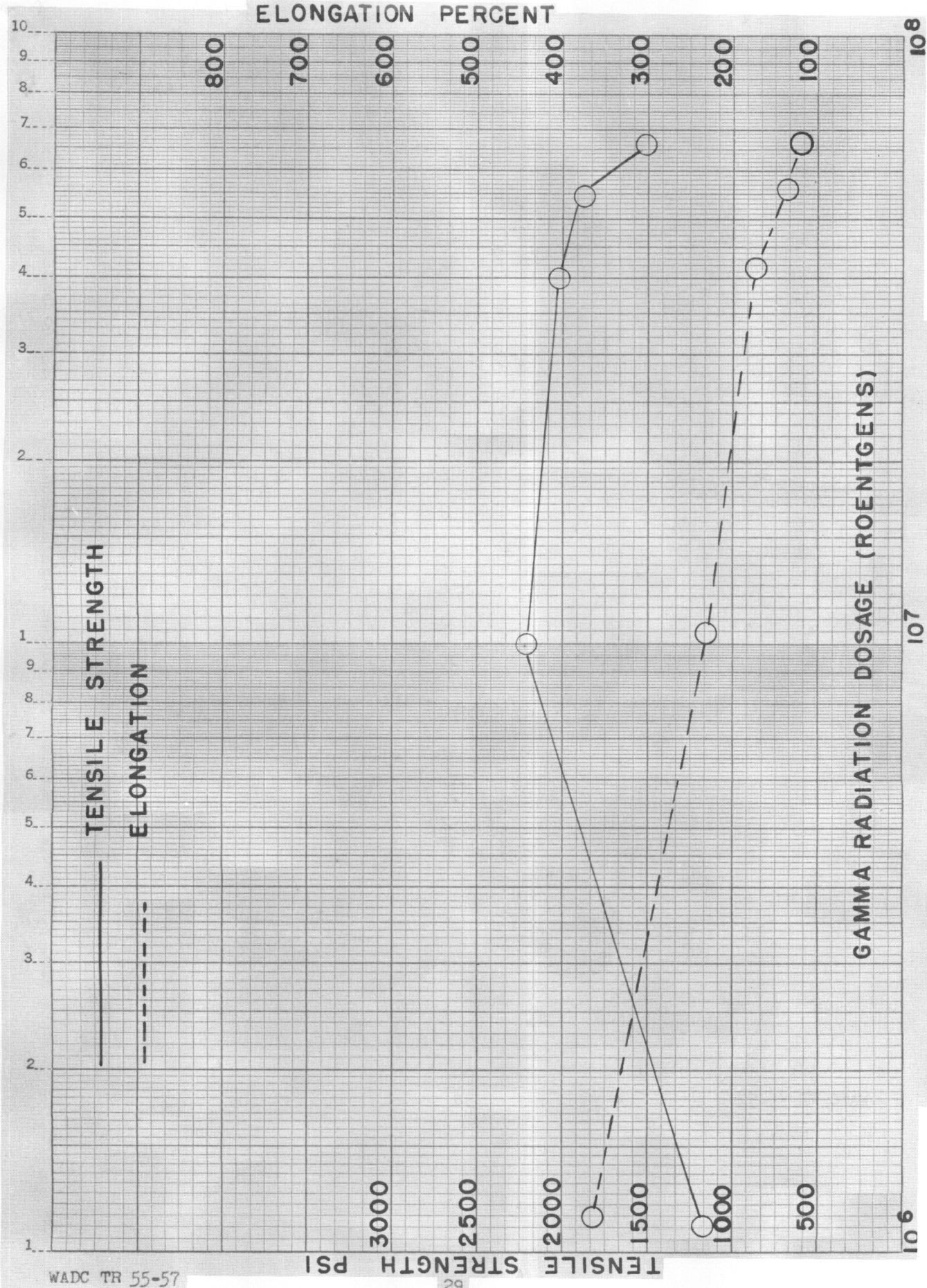


FIG. 6 NEOPRENE WRT + 45 PARTS PHILBLACK A

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FIG. 7. SHOWING TECHNIQUE FOR LOADING RUBBER SAMPLES IN COBALT 60 SOURCE

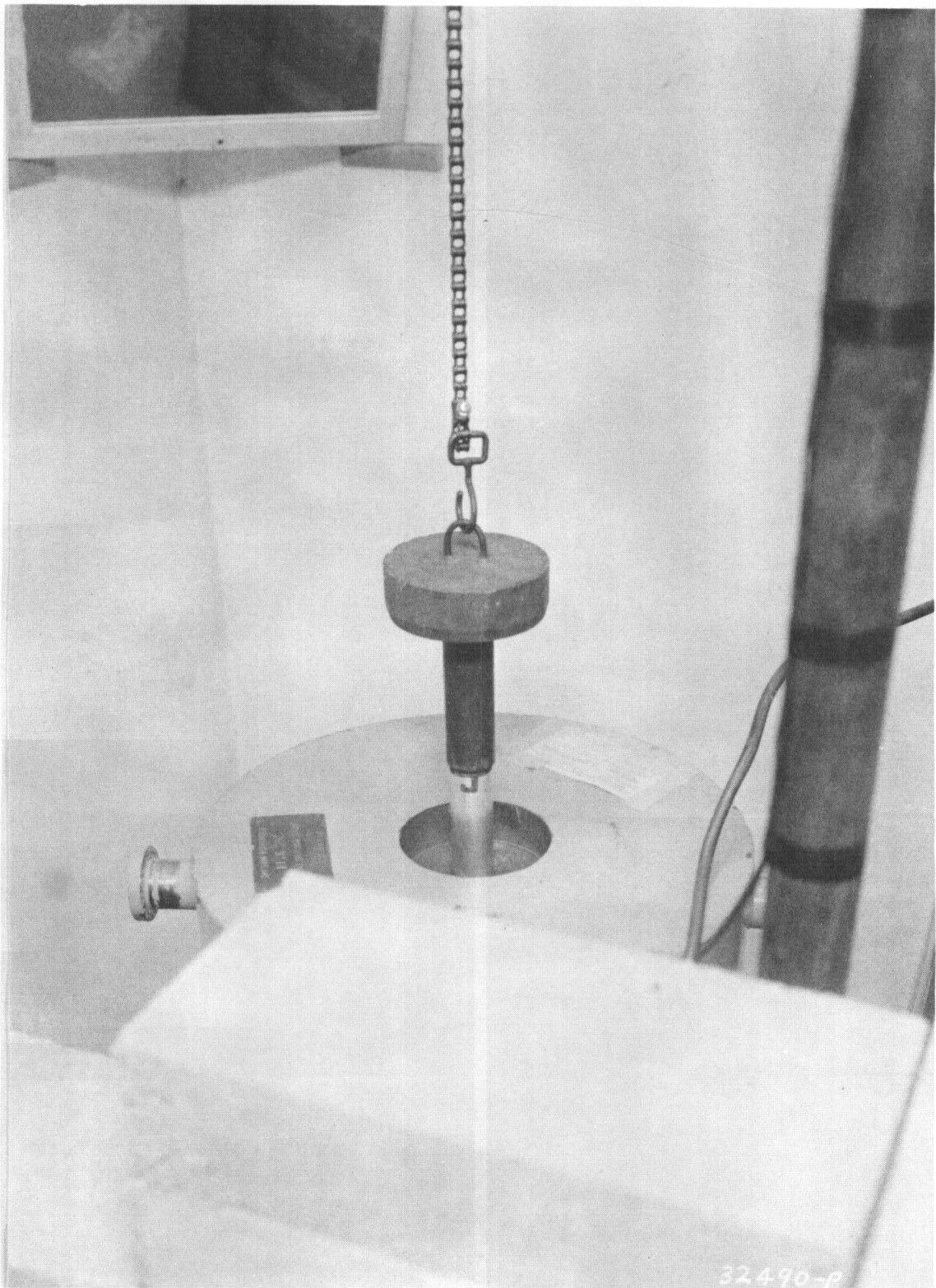


FIG. 8. CLOSE UP VIEW OF COBALT 60 SOURCE SHOWING PLUG WITH CANISTER IN SLIGHTLY ELEVATED POSITION

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