NATIONAL BUREAU OF STANDARDS REPORT

10 029

COMPATABILITY OF PROTECTIVE COATINGS, MATERIALS, AND LIQUID PROPELLANTS

QUARTERLY PROGRESS REPORT NO. 13
January 1 to March 31, 1969

For
Picatinny Arsenal Project
Order No. A1-8-RF034-01-D1-GG

To

Commanding Officer
Picatinny Arsenal, Dover, N. J. 07801
Attn: Code SMUPA DL-2
Liquid Rocket Propulsion Laboratory

U.S. DEPARTMENT OF COMMERCE
NATIONAL BUREAU OF STANDARDS

NATIONAL BUREAU OF STANDARDS

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[1] Headquarters and Laboratories at Gaithersburg, Maryland, unless otherwise noted; mailing address Washington, D. C. 20234.
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COMPATABILITY OF PROTECTIVE COATINGS, MATERIALS, AND LIQUID PROPELLANTS

By
J. P. Young and G. I. Reid

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U.S. DEPARTMENT OF COMMERCE
NATIONAL BUREAU OF STANDARDS

COMPATIBILITY OF PROTECTIVE COATINGS, MATERIALS, AND LIQUID PROPELLANTS

A. Objectives of Project

1. To investigate the effect of various coatings and materials for their ability to withstand the corrosive effects of hydrazine fuels and oxidizers, and for freedom from catalytic effect on the decomposition of fuels and oxidizers.

2. To develop procedures for applying coatings, disclosed as satisfactory under part (1), to the interior surfaces of rocket and missile fuel tanks of complex shape.

B. Summary of Progress in Preceding Quarter

1. Decomposition of MHF-3 fuel in contact with various materials

There was little change in the activity of metals in contact with MHF-3 at 170°F listed in Table 1. Several stainless steels were giving considerably different rates for comparable units and a variation in the oxide coating on otherwise similar specimens was suggested as the cause. To check this idea, several stainless specimens and nickel were oxidized in air before exposure to fuel. Initial indications were that AM355 stainless and nickel were considerably less reactive in the oxidized state with MHF-3.

2. Experiments on kinetics of fuel decomposition

The rate of fuel decomposition decreased further for the Maraging steel specimens partially coated with cadmium indicating continued protection of the exposed MS by the cadmium. A specimen with one-half of the MS surface coated with electroless nickel gave a higher rate than MS alone.

A specimen of large area plated with electroless nickel gave the same rate coefficient as those having one-tenth the area/volume-of-fuel ratio.

3. Decomposition of Aerozine-50 fuel in contact with various materials at 160°F

The background rate for this fuel continued to decline and the high fuel decomposition rate for Teflon coated aluminum and Maraging steel were confirmed.

4. Exposure of various materials to oxidizers at 160°F

Sudden rapid increases in the decomposition rate of IRFNA was considered to result from gradual consumption of the HF inhibitor at 160°F. A change in the system for evaluating IRFNA was considered necessary. There was no significant change in units containing NTO.

C. Summary of Progress During Current Period

1. The rates of fuel decomposition of specimens exposed to MHF-3 at 160°F did not change significantly. Several units which had a high rate, including some stainless steels, were discontinued. Sixteen new units were assembled and testing started. These new units included specimens of varying thicknesses of cadmium and electroless nickel, solid specimens of cadmium and electroless nickel and specimens of thermally oxidized Maraging steel and electroless nickel. Data on these units containing MHF-3 are given in Table 1 and Figure 1.

2. Experiments on kinetics of fuel decomposition

Another unit was added to test the effect of porosity in coatings. It contained a Maraging steel specimen with end caps of cadmium in mechanical contact with the steel (Figure 3). The fuel decomposition

rate is lower than that for Maraging steel alone and is decreasing. Further testing to determine the effect of ratio of metal area to fuel volume has been stopped. The results indicate that the rate coefficient is independent of the A/V ratio, at least for electrolytic nickel and electroless nickel, the two materials tested.

3. Decomposition of Aerozine-50 fuel

The units containing Maraging steel and Teflon coated aluminum have been discontinued because of excessive rate of fuel decomposition. The other units containing 301 stainless steel, titanium and Teflon did not change in rate appreciably (Table 4). A new unit is being set up containing MP35N high alloy steel.

4. Exposure of materials to UDMH fuel

Two units containing uncoated Maraging steel specimens show a low rate of fuel decomposition at 160°F.

5. Exposure of materials to oxidizers

The rate coefficient of gas evolution for specimens in NTO decreased slightly this period. The rate also decreased slightly for specimens in IRFNA but the data was clouded by the uncertainty of the concentration of the HF inhibitor. Modification of the test units to lessen this difficulty is planned. Meanwhile the test temperature has been reduced from 160°F to 90°F. At the lower temperature no reaction is evident.

D. Details of Progress During the Current Report Period

1. Decomposition of MHF-3 fuel in contact with various materials at 160°F

a. "Background" rate

The average rate of evolution of gas in blank (no specimen) mercury manometer test units Hg-2 and Hg-15 has decreased slightly from 0.0039 cm^3/day at the end of the preceding quarterly period to 0.0037 cm^3/day at the end of the present period. Since units having a larger bulb and manometer are used for specimens of larger surface area or those expected to have a high fuel decomposition rate, a third "background" unit of the larger size was set up. So far it shows no increase in the background rate over the standard size units but was not included in determination of the average value because of its relatively short test period.

b. Effects of various metals

Table 1 and Figure 1 contain the cumulative results to the end of March 1969. The relative reactivity of the various specimens is as follows:

Low activity: silver, cadmium, 50/50 lead-tin solder, electroless nickel (standard acid bath), zinc, tin-nickel alloy, tin, 347 stainless steel, tungsten, aluminum, lead, titanium alloys 6Al-4V and 3 Al-11Cr-13V, Teflon coated Maraging steel, oxidized AM355 stainless steel, oxidized nickel and 301 stainless steel.

Moderate activity: gold, nickel, stainless Maraging steel and chromium.

High activity: cobalt, 18% Maraging steel, molybdenum, iron, AM 355 stainless steel, Inco-718.

(1) Behavior of recent units

The performance of the oxidized stainless steel, AM355, in unit Hg-74 lends credence to the theory that nonuniform oxide coatings cause wide variation in the performance of otherwise similar stainless steel specimens. In this case, a thermally oxidized specimen has a rate, thus far, lower by a factor of twenty than the best untreated specimen. The reduced rate of nickel after oxidizing is down in the range of that for electroless nickel. Combined with its good ductility, this low rate gives nickel possibilities as a collapsable liner material. The oxidized stainless Maraging steel showed no improvement in rate. Oxidized Maraging steel also showed no improvement in fuel decomposition rate.

(2) Deposit thickness tests on Maraging steel

Deposits of cadmium and electroless nickel of from 0.5 to 2 mils in thickness have given low rates. The initial tests of deposits of these metals, 0.1 mil thick, give rates in the low activity range (Hg-81, Hg-82).

(3) Other new tests

A Maraging steel specimen with zinc phosphate coating gave no initial improvement over untreated MS. However, the 301 stainless steel specimen (Hg-89) gave a low rate. A Maraging steel specimen dip-coated with 50-50 lead-tin alloy (Hg-91) gave a very low rate. A specimen plated with electroless nickel in the citrate type bath (Hg-78) gave a higher initial rate than expected and probably should be confirmed by a check run. A unit containing oxidized electroless nickel (Hg-90) showed no improvement over a similar untreated coating.

2. Results of experiments on the kinetics of fuel decomposition

a. The rate of fuel decomposition of the cadmium plated Maraging steel specimen with one-half of the steel surface exposed (Hg-51) and the cadmium plated Maraging steel specimen with pores drilled to expose the steel (Hg-63) continued to decrease (Table 2). A third Maraging steel specimen (Figure 3) with the entire surface exposed except where caps of cadmium were mechanically clamped (Hg-84) gave a rate about one-half the average for uncoated Maraging steel alone. These tests show the considerable cathodic protection afforded Maraging steel by cadmium resulting in reduced decomposition of MHF-3 at 160°C.

The specimen consisting of electroless nickel on Maraging steel with one-half of the steel surface exposed (Hg-71) continued to give a rate greater than that for either Maraging steel or electroless nickel alone although its rate did decrease from the previous quarter.

b. Effect of ratio of metal area to fuel volume

The rate of fuel decomposition resulting from the exposure of a large area of electroless nickel to fuel (Unit Hg-70, Table 3) continues to parallel the rate for much smaller specimens for the second quarter. This further confirms the independence between the rate coefficient of fuel decomposition and the specimen-area fuel-volume ratio for electroless nickel. This test series is being discontinued.

3. Fuel tanks plated for Picatinny Arsenal

Two one-half scale fuel tanks were plated, one with 0.88 mil average thickness of electroless nickel and one with 1.2 mils average thickness of cadmium and given to Mr. Ng to be tested at Picatinny Arsenal.

4. Decomposition of Aerozine-50 fuel in contact with various materials at 160°F

Results of the exposure tests are given in Table 4. The background rate for this fuel in a test unit without a specimen has changed slightly from a previous value of 0.0063 to 0.0074 cm^3/day. The units containing Maraging steel and Teflon coated aluminum have been discontinued because of excessive rate. At the request of Mr. Ng of Picatinny Arsenal a new unit is being set up with MP35N high alloy steel as the specimen. There was little change in the rates for 301 stainless steel, titanium and Teflon (TFE).

5. Decomposition of UDMH in contact with specimens at 160°F

Two units containing UDMH (unsymmetrical dimethyl hydrazine) fuel were set up during the period of this report at the request of Mr. Ng of Picatinny Arsenal. The specimen in each is 18% Maraging steel. So far there has been very little reaction with the fuel. The results to date are shown in Table 5.

6. Exposure of various materials to oxidizers at 160°F

Results of the nine bomb-type test units are given in Table 6 and Figure 2. The two oxidizers used are NTO (nitrogen tetroxide inhibited with 0.6±0.2% of nitric oxide) and IRFNA (red fuming nitric acid inhibited with 0.7±0.1% hydrogen fluoride).

Because of the suspected gradual loss of the inhibitor in the IRFNA units and subsequent rapid increase in pressure, only the data obtained during the period after refilling the units and the start of a sudden pressure was used. At the suggestion of Mr. Ng, the temperature of the IRFNA units was reduced from 160°F to 90°F. At this lower

temperature no reaction has been observed. It has been learned that the 3-M Company markets an easily applied Teflon coating. It is planned to coat a couple of the stainless steel units to try to reduce consumption of the IRFNA inhibitor and to obtain longer uninterrupted tests of specimens at 160°F. According to information from our mechanical design group, bombs made wholly from Teflon would not be suitable for our application because of the problem of cold creep of the Teflon subjected to the pressures necessary to insure leak-proof sealing of the bombs. In the meantime the runs will be continued at 90°F.

The units containing NTO are relatively unchanged from the previous period.

D. New Work

1. Tests of special materials

Materials in test units now being set up at the request of Mr. Ng include: MP35N high alloy steel from Latrobe Steel Co.; vapor deposited aluminum coating on Maraging steel from Commonwealth Scientific Corp.; 2021 aluminum from Alcoa; and 301 stainless steel (untreated). Other new units will contain 50-50 cadmium-tin alloy dip coated, a duplicate 50-50 lead-tin alloy dip coated, and a blank (no specimen) "background" unit containing UDMH.

In addition to the above, and also at the suggestion of Mr. Ng, two companies have been contacted in regard to coating specimens with aluminum by their processes. Dow Chemical Company has agreed to coat two specimens with their solvated aluminum hydride electroless aluminum process in return for a description of the test procedure and results.

Samples of Maraging steel have already been sent to Continental Oil's Organometallic Division to be aluminum coated in their aluminum diethyl hydride bath.

An inquiry to the Astro-Power Laboratory of McDonnell Douglas Astronautics Company revealed that their Astro-coat-T fluocarbon polymer coating process has not been tested on the type of material of interest to us (Maraging steel) and will require further investigation on their part before coated samples can be furnished.

2. Examination of terminated units

Fuel and metals in units for which there is no need for further testing will be examined as necessary.

3. Coating fuel tanks

Two more one-half scale fuel tanks are on hand and will be modified and plated as required.

TABLE 1

Summary of Test Data for Materials

Exposed to MHF-3 at 160°F

Test unit number	Specimen	Area of specimen cm^2	Time under test days	Gas* evolved cm^3	Rate* coefficient $cm^3/day/cm^2$ previous	Rate* coefficient $cm^3/day/cm^2$ present	Calculated** tank pressure after 1 year psig
Hg-2	Background	--)					
		)av.	818	3.04	0.0039	0.0037	--
Hg-15	Background	--)					
Hg-48	Aluminum	12.5	475	1.1	0.00015	0.00019	2
Hg-31	Cadmium	14.7	614	1.1	0.00008	0.00012	1
Hg-16	Cadmium (thin)	9.4	770	11	0.0012	0.0015	16
Hg-27	Cadmium (thin)	10.6	629	6.1	0.0009	0.0009	10
Hg-81	Cadmium (0.1 mil)	14.4	27	2.3	--	0.0059	62
Hg-80	Cadmium[a]	13.4	64	5.1	--	0.0060	63
Hg-50	Chromium[x]	17.0	482[xx]	113	0.0199	0.0201	213
Hg-12	Electroless nickel	22.2	788	17	0.00098	0.00097	10
Hg-49	Electroless nickel (Alk)[x]	12.6	461[xx]	169	0.228	0.2198	2319
Hg-52	Electroless nickel (Alk)[x]	13.0	320[xx]	42.5	0.0187	0.0125	132
Hg-58	Electroless nickel (BH_4)[x]	14.2	390	113	0.0196	0.0203	214
Hg-61	Electroless nickel (800°C)	12.2	305	4.1	0.0011	0.0011	12
Hg-77	Electroless nickel[a]	15.0	57	4	--	0.0047	49
Hg-78	Electroless nickel (citrate bath)	9.8	59	9.7	--	0.0169	178
Hg-82	Electroless nickel (0.1 mil)	15.3	63	7.5	--	0.0078	82
Hg-90	Electroless nickel (oxidized)	14.4	40	3.5	--	0.0061	64
Hg-39	Inco (718)[a,x]	12.0	482	207	0.1120	0.1142	1205
Hg-87	Maraging steel (oxidized)	15.0	34	42.6	--	0.0835	882

TABLE 1 (cont.)

Summary of Test Data for Materials

Exposed to MHF-3 at 160°F

Test unit number	Specimen	Area of specimen cm^2	Time under test days	Gas* evolved cm^3	Rate* coefficient $cm^3/day/cm^2$ previous	present	Calculated** tank pressure after 1 year psig
Hg-57	Nickel	14.0	405	24	0.0050	0.0042	45
Hg-75	Nickel (oxidized)[a]	18.5	93	7	--	0.0041	43
Hg-29	Silver	14.5	626	2.3	0.0002	0.00025	3
Hg-22	Silver (thin)	16.0	709	128	0.0104	0.0112	119
Hg-33	Solder (Pb/Sn, 50-50)[a]	15.3	614	Zero	Zero	Zero	Zero
Hg-91	Solder (Pb/Sn, 50-50)(Dip coated)	14.3	19	0.4	--	0.0015	16
Hg-14	Stainless steel (347)[a]	13.2	797	Zero	Zero	Zero	Zero
Hg-28	Stainless steel (347)[a]	6.5	625	7.7	0.0020	0.0019	20
Hg-43	Stainless Maraging steel[a]	15.0	525	43.5	0.0058	0.0058	61
Hg-55	Stainless Maraging steel[a,x]	16.6	398	77.2	0.0176	0.0171	180
Hg-44	Stainless steel (AM355)[a,x]	11.4	398	148	0.0883	0.0920	972
Hg-54	Stainless steel (AM355)[a,x]	11.5	398	75.6	0.0335	0.0391	413
Hg-74	Stainless steel (AM355)[a] (oxidized in air at 500°F)	18.5	105	4	--	0.0021	22
Hg-89	Stainless (301, aged)[a]	14.7	40	2.5	--	0.0043	45
Hg-68	Teflon coated MS	14.2	290	13.5	0.0041	0.0033	35
Hg-32	Tin	13.0	616	26.3	0.0035	0.0033	35
HG-30	Titanium[a,b]	15.0	456	Zero	Zero	Zero	Zero
Hg-45	Titanium (6Al,4V)[a]	13.3	478	Zero	Zero	Zero	Zero

*, **Cumulative total, corrected for background rate and to 1 atm. pressure.

**Based on a tank in the form of a cube, 1 cu. ft. volume, 10% ullage.

[x]Discontinued.

[xx]Total days under test are not equal to number of days during which gas was collected, due to interrupted collection.

[a]These specimens are solid metal. All others are coatings on 18% Marging steel.

[b]Titanium alloy, 13% V, 11% Cr, 3% Al. Similar to alloy B120VCA.

[c]Vapor-deposited foil from Commonwealth Scientific Corp.

TABLE 2

Data on Decomposition of MHF-3 Fuel at 160°F
in Special Tests

Effect of Porosity in Coatings of Cadmium on Maraging Steel

Test unit number	Description	Time under test days	Gas evolved cm^3	Rate coefficient $cm^3/day/cm^2$ * previous	present
Hg-51	Cadmium on Maraging steel with 1/2 of MS surface exposed - 8 cm^2	423	33.8	0.013	0.0100
Hg-63	Cadmium on Maraging steel with 200 0.026" diam. pores. MS area 1.04 cm^2	310	8	0.027**	0.0248
Hg-71	Electroless nickel on Maraging steel with 1/2 of MS surface exposed - 14 cm^2	161xx	113	0.355	0.202x
Hg-84	Maraging steel in electrical contact with Cd end caps.	28	9.9	--	0.0410

*Rate coefficient is calculated on basis of exposed Maraging steel only.

**Rate coefficient based on pore area: 1.04 cm^2.

xHighest rate for Maraging steel alone was 0.098 and for standard electroless nickel 0.0047.

xxTotal days under test are not equal to days during which gas was collected.

TABLE 3

Effect of Variation of Ratio of Metal Area to Fuel Volume at 160°F

Test unit number	Description				$\frac{A}{V}$	Test under test	Gas evolved	Rate coefficient
	Coating or metal	Volume (V) Liquid cm^3	Volume (V) Vapor cm^3	Metal Area (A) cm^2	cm^{-1}	days	cm^3	$cm^3/day/cm^2$
Hg-70	El nickel on Ni wire coil[x]	18	6	108	6.0	150	18.1	0.0011
Hg-4	El nickel on MS[x]	37	4.7	19.6	0.5	458	14.5	0.0016
Hg-12	El nickel on MS	29	5.9	22.2	0.8	788	17	0.0010

[x]Discontinued.

TABLE 4

Test Data for Materials Exposed to Aerozine-50

at 160°F

Test unit number	Coating or metal	Area of specimen cm^2	Time under test days	Gas* evolved cm^3	Rate* coefficient $cm^3/day/cm^2$ previous	present	Calculated** tank pressure after 1 year psig
Hg-53A	301 Stainless steel[a] (cryogenic form)	14.7	218	83.8	0.0290	0.0262	276
Hg-56A	None (Background)		368	2.71	0.0063	0.0074	--
Hg-59A	Maraging steel steel[a,x]	15.2	0.8	41.2	--	3.39	35,760
Hg-60A	Titanium[a]	14.5	370	0.3	Zero	0.00006	1
Hg-62A	Teflon (TFE)[a]	17.5	370	0.7	0.00002	0.00011	1
Hg-69A	Teflon coated 7075-T6 Al[x]	14.2	0.33	9.4	--	2.01	21,170

*Cumulative total, corrected for background rate and to 1 atm. pressure.

**Based on a tank in the form of a cube, 1 cu. ft. volume, 10% ullage.

[a]These specimens are uncoated, solid material.

[x]Discontinued.

TABLE 5

Test Data for Materials Exposed to UDMH

at 160°F

Test unit number	Coating or metal	Area of specimen cm^2	Time under test days	Gas* evolved cm^3	Rate* coefficient $cm^3 day/cm^2$ previous	present	Calculated** tank pressure after 1 year psig
Hg-85U	Maraging steel[a]	14.0	26	0.7	--	0.0019	20
Hg-92U	Maraging steel[a]	14.0	20	0.3	--	0.0011	11

*Cumulative total corrected to 1 atm. pressure, no background.

**Based on a tank in the form of a cube, 1 cu. ft. volume, 10% ullage.

[a]These specimens are uncoated, solid material.

TABLE 6

Results of Tests of Materials Exposed to Oxydizers

at 160°F in

Stainless Steel Bomb-Type Test Units

Test unit number	Specimen	Area of specimen cm^2	Time under test days	Gas* evolved cm^3	Rate* coefficient $cm^3/day/cm^2$ previous	present	Calculated** tank pressure after 1 year psig
	Inhibited red fuming nitric acid						
RFN-1	Teflon (TFE)	14.5	202	14.4	0.0074	0.0049	52
RFN-2	Stainless steel (301 Cryogenic form aged)	14.7	210	13.4	0.0042	0.0043	46
RFN-3	Maraging steel	13.6	193	Zero	Zero	Zero	Zero
RFN-4	Teflon on 6061-T6 Al	15.1	183	9.5	0.0026	0.0034	36
RFN-5	None (Background)	--	32	1.27	--	0.038	--
	Nitrogen tetroxide						
301-NTO***	Stainless steel (301 Cryogenic form un-aged)	338	302	Zero	Zero	Zero	Zero
NTO-1	None (Background)	--	115	5.8	0.0417	--	--
NTO-2	Teflon (TFE)	19.9	246	23	0.0066	0.0047	50
NTO-3	Stainless steel (301 Cryogenic form aged)	14.7	265	2.8	0.0013	0.0007	8
NTO-4	Maraging steel	13.6	238	2.4	0.00099	0.00090	9
NTO-5	Titanium (6Al, 4V)	13.4	120	0.8	0.00097	0.00050	5

*Cumulative total, corrected for background rate and to 1 atm. pressure.

**Based on a tank in the form of a cube, 1 cu. ft. volume, 10% ullage.

***Tested at 90°F.

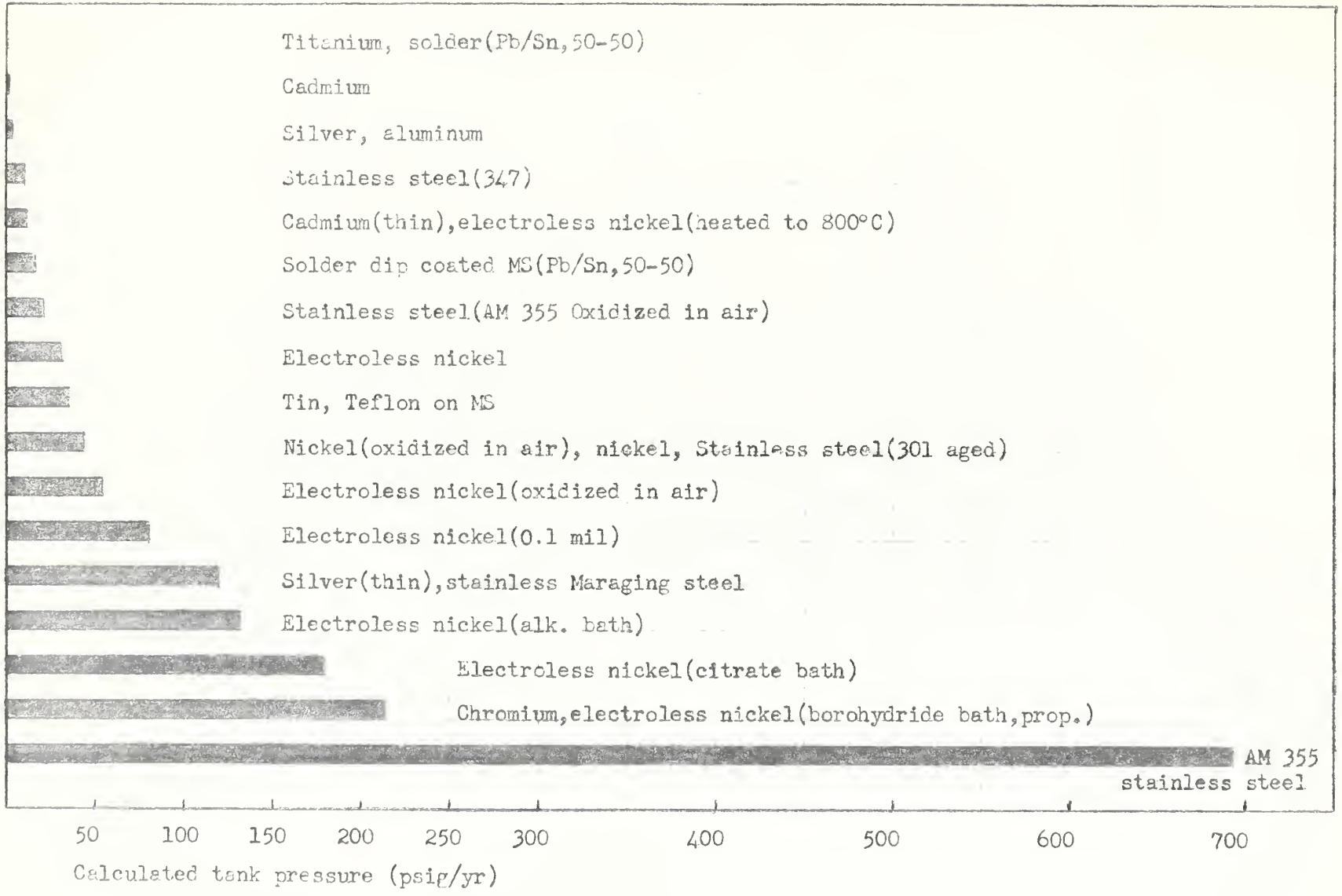


Figure 1

Rate of gas evolution from MHF-3 in contact with indicated metals at 160°F. (Some short term units not in average)

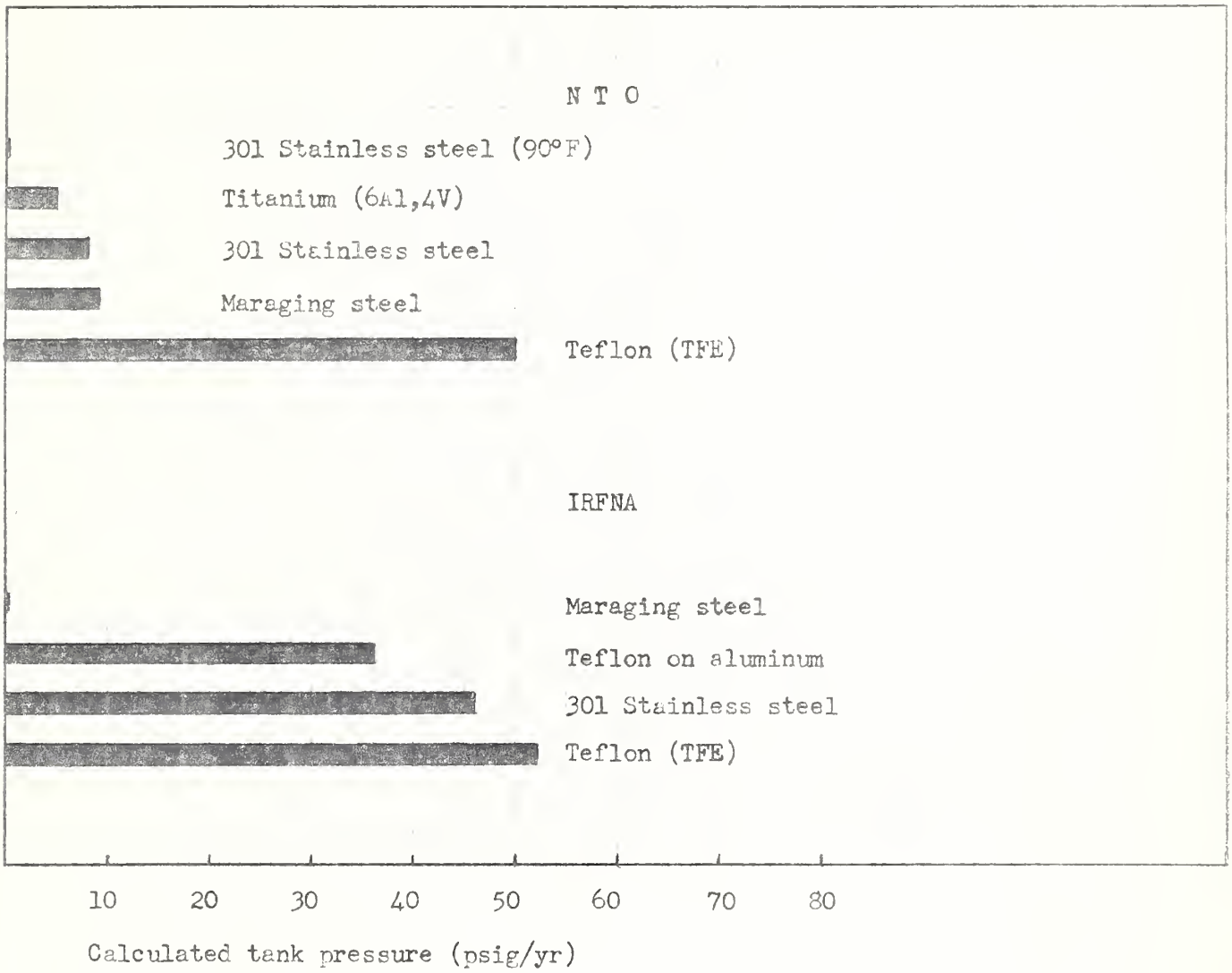
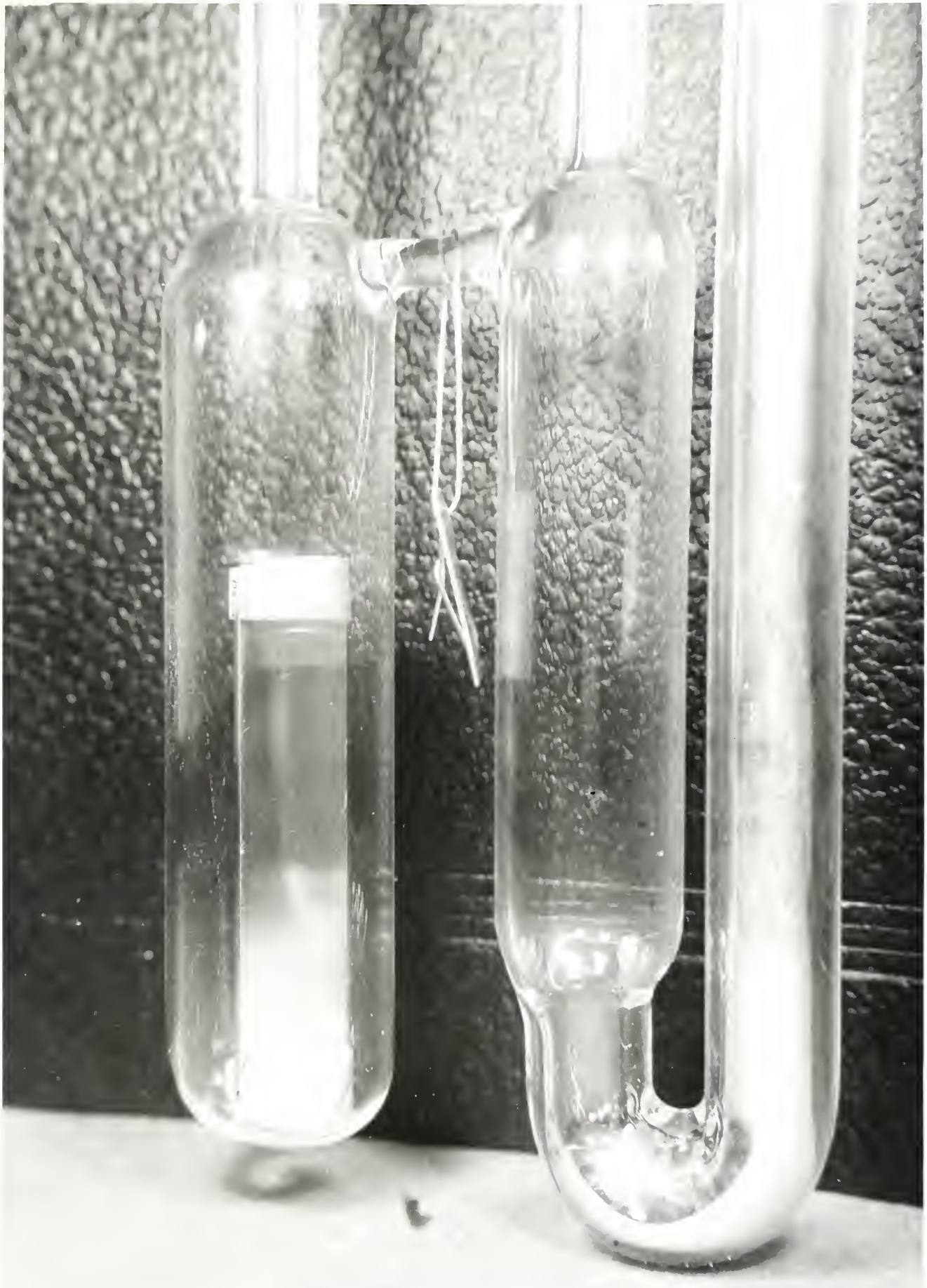


Figure 2

Cumulative gas evolution as indicated by pressure increase resulting from contact of above materials with NTO and IRFNA at 160°F.

Figure 3. At the left of center is the specimen chamber of a test unit containing a Maraging steel specimen protected by caps of cadmium clamped on each end. At the right is the mercury reservoir and the manometer column. There is no fuel in the specimen chamber and the mercury level is below the bottom of the reservoir because the unit is being leak tested under full manometer pressure. Magnification - 2X.

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