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Storable Concentrated Hydrogen Peroxide

J.M. Monger

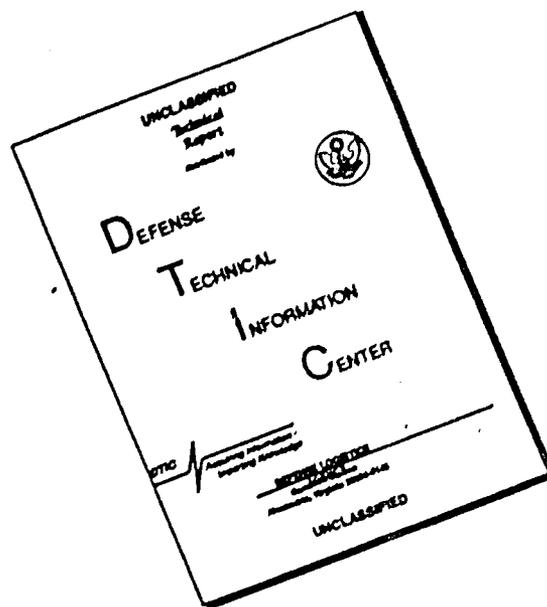
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Quarterly Progress Report No. 3
September, 1966 - November, 1966

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AFRPL-TR-66-353

STORABLE CONCENTRATED HYDROGEN PEROXIDE

Sponsoring Agency:

Air Force Rocket Propulsion Laboratory
Research and Technology Division
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Air Force Systems Command, U.S.A.F

Contract No. AF 04(611)-11416

Quarterly Progress Report No. 3
September-November 1966

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Shell Development Company
A Division of Shell Oil Company
Emeryville, California

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FOREWORD

This report describes research being carried out to improve the storability of concentrated hydrogen peroxide. The work is being conducted for the Air Force under Contract No. AF 04(611)-11416, Project No. 3148, BPSN 623148. The contract project officer is 1/Lt Ralph Fagnoli, USAF, Air Force Rocket Propulsion Laboratory, RPCL, Edwards, California. This Quarterly Report No. 3 describes work carried out by Shell Development Company, Emeryville, California in the period September 1 to November 30, 1966.

Publication of this report does not constitute Air Force approval of the report's findings or conclusions. It is published only for the exchange and stimulation of ideas.

W. H. Ebelke, Colonel, USAF
Chief, Propellant Division

ABSTRACT

Investigation of means for reducing the rate of decomposition of concentrated hydrogen peroxide (HP) by removing the catalytic elements carried in the HP and by reducing the catalytic activity of various materials of construction for use as containers has continued.

A flash distillation unit has been constructed in order to prepare some 5-gal storage samples of HP purified by additional distillation. Samples of HP-98 prepared by distillation at low pressure and temperature will be prepared for storage. Samples of HP-90 will be distilled also and tested by the accelerated test.

An ion exchange bed support unit has been designed and constructed and will be assembled with a feed pump in order to prepare other storage samples of HP-98.

Additional storage samples have been placed in constant temperature baths. Included are two in tin-plated vessels to again test the activity of tin plated from a stannous sulfate-sulfamic acid bath.

CONTENTS

	Page
Abstract.	iii
Introduction.	1
Objectives of the Program	1
Observation of Previously Stored Samples.	2
Purification of HP.	2
Ion Exchange	2
Distillation	6
Studies of Container Surfaces	7
Surface Activities	7
Storage Samples	7
Program	9
Document Control Data - R and D; DD Form 1473 With Abstract and Key Word List	10

LIST OF TABLES

<u>Table</u>		Page
1	Storage Samples at 25°C.	3
2	Decomposition Rates of Ion-Exchange Effluent at 100°C.	5
3	Surface Activities in Stabilized HP-90 at 100°C.	8

LIST OF FIGURES

<u>Figure</u>		Page
1	Decomposition Observed in 5-Gallon Samples at 25°C. . . . follows	3
2	Decomposition Observed in 5-Gallon Samples at 25°C. . . . follows	3
3	Ion Exchange Bed Support. follows	4
4	Hydrogen Peroxide Flash Distillation Apparatus. follows	6
5	Storage Sample Closure. follows	9

STORABLE CONCENTRATED HYDROGEN PEROXIDEIntroduction

The design of systems employing concentrated hydrogen peroxide (HP) requires allowance in one form or another for the decomposition of a small fraction of the peroxide in the system tanks and lines. For systems intended for long term storage under sealed conditions this allowance must be in the form of ullage space and vessel wall strength in order to confine the gas liberated by decomposition of the peroxide. The present investigation has been concerned with means of reducing the amount of decomposition of concentrated HP which would occur under storage conditions as a result of 1) dissolved and suspended contaminants in the liquid phase, 2) catalytic activity of the container surfaces submerged in the HP and 3) catalytic activity of the container surfaces not submerged but in contact with the vapor from the HP. As in the previous reports from these laboratories on this subject the decomposition rates stemming from these sources will be separated whenever possible and will each be treated as first order reactions with respect to HP concentration. The assumption that the reaction is first order rather than a higher or a zero order will cause no significant errors in decomposition calculations as long as minor changes in HP concentration occur. In comparing surface activity measurements made with HP-90 and HP-98 differences may appear if the actual reaction mechanism is other than first order in HP concentration.

In the following data, therefore, the rate constants for the homogeneous liquid phase decomposition will be designated k_1 , that for the heterogeneous reaction on submerged surfaces will be designated k_2 and that for the heterogeneous reaction on non-submerged surfaces will be designated k_3 . In line with previous measurements an Arrhenius type activation energy will be assumed to apply to all three rate constants in extrapolating and comparing rates at various temperatures other than those specified for the particular measurement. In all cases the rates are determined by gas evolution methods instead of chemical analysis since the expected change in composition in most samples is too small for the latter method to give accurate results. The gas evolution method also allows for differential rate determination during the course of observations.

Objectives of the Program

The investigation of methods of purification and stabilization of HP and means of preparing and passivating inactive surfaces for contact with HP will be continued in order that storage of concentrated HP in sealed containers for periods up to 5 years might be feasible.

The scope of the investigation will be:

- 1) to continue laboratory investigation of ion-exchange, distillation and recrystallization techniques for purification of 90% to 98% HP in order to reduce the rate of homogeneous decomposition;
- 2) to continue laboratory investigation of tin plating on aluminum and on steel in order to achieve inactive tin surfaces;

- 3) to investigate additional materials of construction which have attractive physical or chemical properties for tentative use with concentrated HP;
- 4) to continue laboratory investigation of means of passivating surfaces of materials of construction for intended use with concentrated HP;
- 5) to develop ion-exchange equipment for processing quantities of HP;
- 6) to demonstrate with samples of HP of 5 gallon size, stabilized and stored at 25°C, the various purification techniques and surface coatings developed in the laboratory studies;
- 7) to continue the observation of the storage samples already in existence which are demonstrating stabilization formulas in HP-90, and which are stored in pyrex, aluminum and Kel-F lined aluminum containers.

Observation of Previously Stored Samples

Monitoring has continued of the samples in 5-gallon containers which were placed in storage a year and a half or more ago. The results of the samples listed in Table 1 are shown in Figures 1 and 2. Very little change has been observed since the first progress report^{a)} on those samples which were stabilized with stannate at the 34×10^{-6} m/l (4 mg tin/l) level. Those stored in aluminum type 1260 show a gradual increase in rate indicative of a slow contamination by metallic ions probably corroded from the aluminum surface. The sample stabilized with only 1/4 as much stannate (8×10^{-6} m/l) has increased in decomposition rate to 0.009 year^{-1} , slightly below that of the unstabilized samples.

One stabilized sample stored in a Kel-F lined vessel has shown an increase of about 25% during the past quarter, starting from the time of rearrangement of the baths. Since the temperatures of the baths were upset for a short period at that time, rising to 35-40°C, it is possible that the observed rise in rate is due to contamination carried into the sample by condensate in the aluminum gas delivery line.

The three samples stored in aluminum type 1260 without stabilizer have continued to increase in decomposition rate. The decomposition rates are now at about 0.011 to 0.013 year^{-1} , which is nearly 50% greater than their initial measurements.

Purification of HP

Ion Exchange

The effectiveness of acid treated 3-stannic acid solids as ion exchange medium for removing contaminants from concentrated HP has not

a) "Storable Concentrated Hydrogen Peroxide", Quarterly Progress Report No. 1, March-May 1966, AFRPL-TR-66-207, Contract AF 04(611)-11416.

Table 1. STORAGE SAMPLES AT 25°C

Sample No.	HP-90 Source	Container Surface	$\text{Na}_2\text{Sn}(\text{OH})_6$, ^{a)} $10^6 \times \text{m/l}$	Storage, Days	\bar{S}/V , cm^{-1}
1013B	Shell Chemical	Pyrex	35	593	0.197
1001	Shell Chemical	1260 Alum.	0	685	0.198
1005	Shell Chemical	1260 Alum.	0	664	0.203
1007	Shell Chemical	1260 Alum.	0	660	0.204
1006	Shell Chemical	1260 Alum.	8.8	674	0.200
1004	Shell Chemical	1260 Alum.	39	608	0.201
1017	Shell Chemical	1260 Alum.	37 ^{b)}	588	0.205
1018	Shell Chemical ^{d)}	1260 Alum.	36	569	0.206
1002	Electrolytic	1260 Alum.	36	651	0.201
1003	Electrolytic	1260 Alum.	107	651	0.202
1022	Electrolytic ^{d)g)}	1260 Alum.	36	(g)	0.199
1019B	Electrolytic ^{d)g)}	Pyrex ^{e)}	36	(g)	0.220
1012B	Shell Chemical	5052 Alum.	36	594	0.205
1016	Shell Chemical	5052 Alum.	47 ^{c)}	618	0.201
1010	Shell Chemical	Kel-F	36	675	0.212
1011	Shell Chemical	Kel-F	36	675	0.218
1023B	Shell Chemical ^{g)}	Elect. Tin ^{f)}	34	(g)	0.215
1024	Shell Chemical ^{g)}	Elect. Tin ^{f)}	34	(g)	0.204

- a) Nitric acid also added to give direct reading pH ca 0.3.
b) Cyclohexanediamine tetracetic acid added ($3.8 \times 10^{-6} \text{ m/l}$) to protect and aid the stannate.
c) 8-Hydroxyquinoline added ($5.4 \times 10^{-6} \text{ m/l}$) to protect and aid the stannate.
d) Treated with stannic acid ion exchanger.
e) Transferred from 1260 aluminum container after 52 days.
f) Tin-plated over fused tin surface.
g) Samples discontinued (see Quarterly Report AFRPL-TR-66-262).

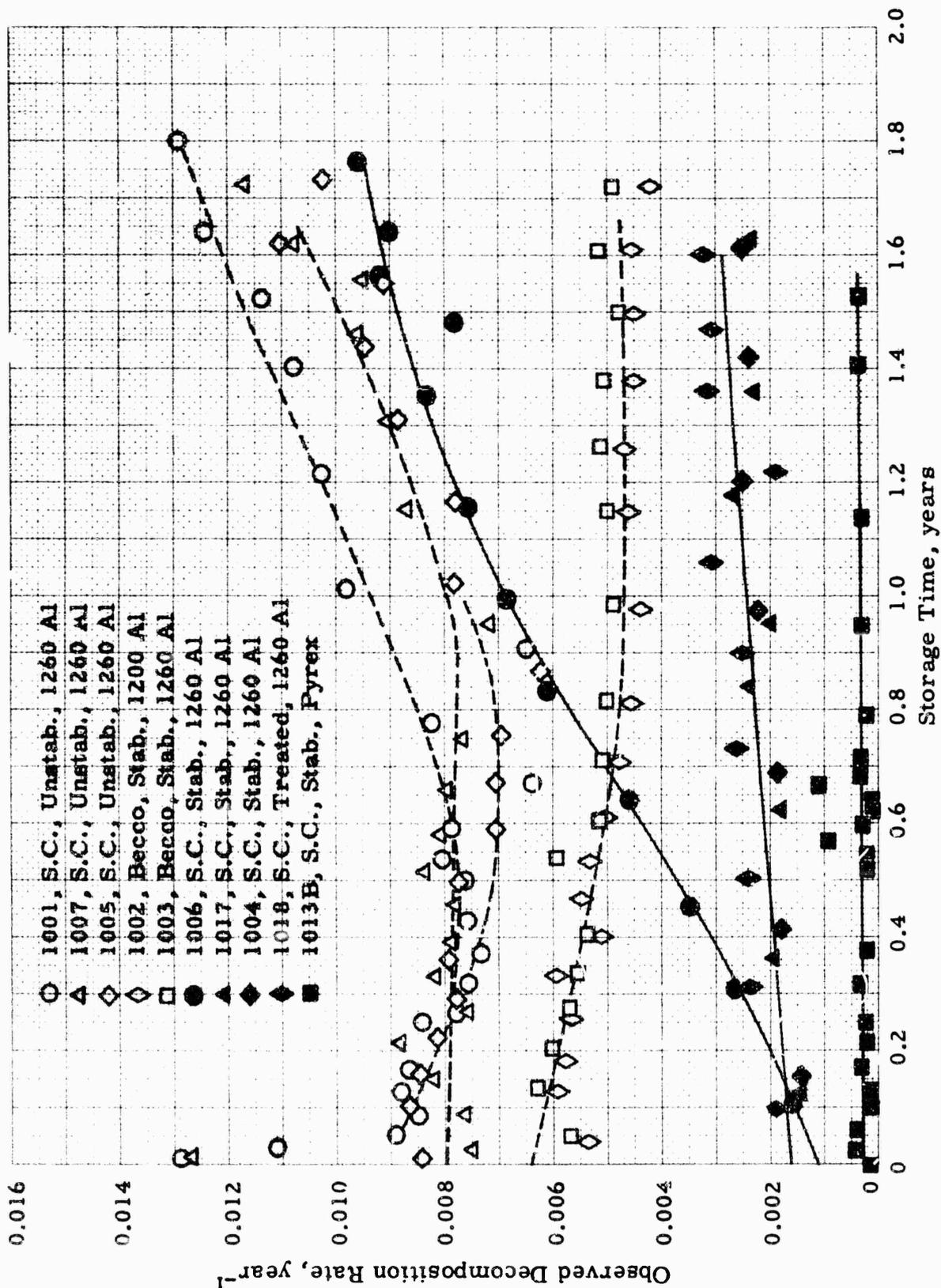


Figure 1. DECOMPOSITION OBSERVED IN 5 GALLON SAMPLES AT 25°C

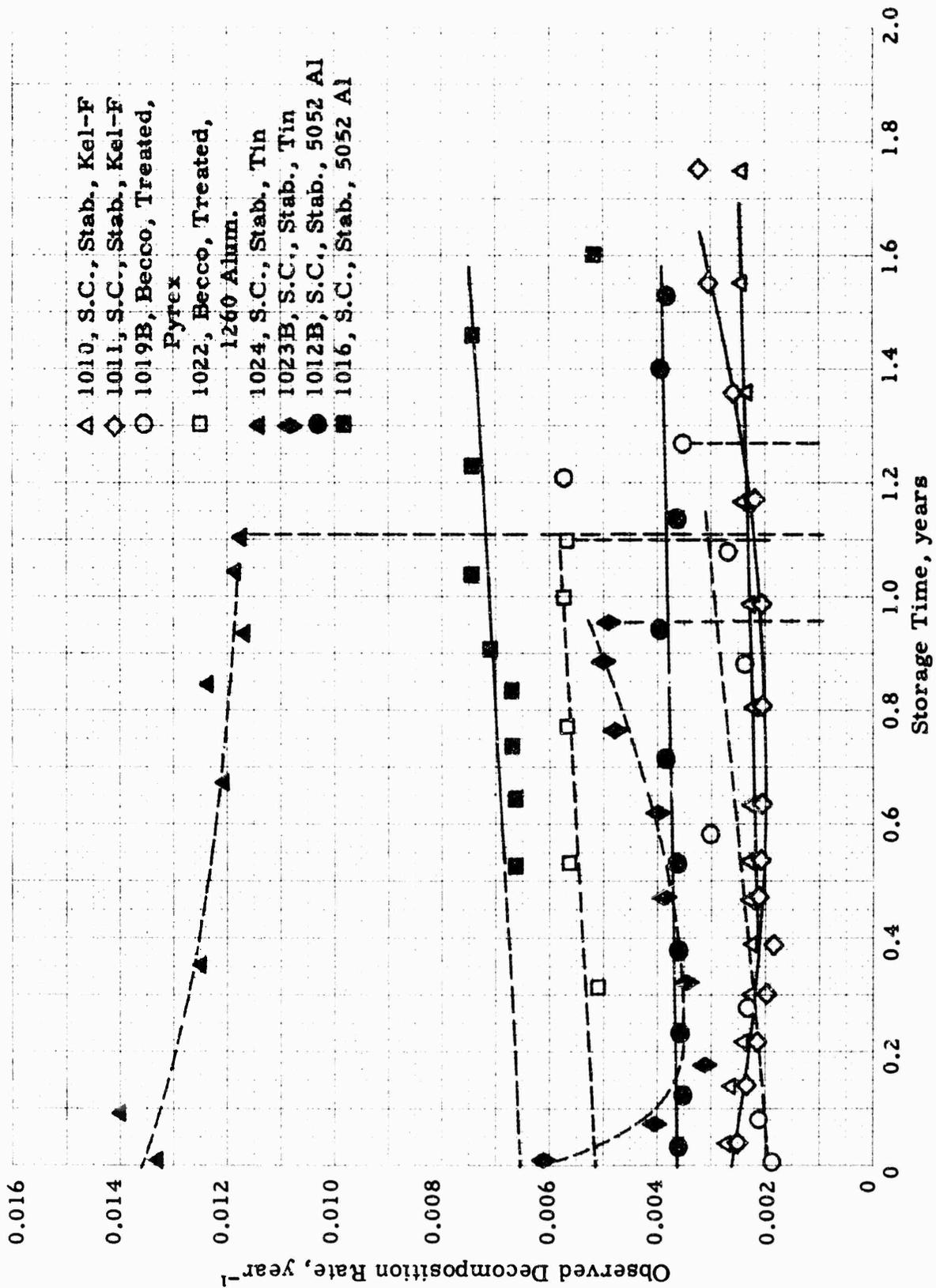


Figure 2. DECOMPOSITION OBSERVED IN 5 GALLON SAMPLES AT 25°C

appeared to be dependent on the flow rates within those limits which have been used in the previous experiments.^{a)} The various bed shapes which have been used also appear to have been of sufficient volume and length to prevent breakthrough of the contaminating ions in the feed HP. It is reasonable, however, that some lower limit exists for contact time in order that these ions may be removed which is a function of the flow rate and the shape, as expressed by the ratio of the length to diameter. As an aid in the design of the larger ion exchange apparatus several experiments were run on short beds of β -stannic acid solids with HP-98 as feedstock. The results are shown in Table 2.

With a ratio of l/d of 0.2 and with flow rates of over 100 LHSV the bed failed to reduce the decomposition rates of the effluent samples below that of the feedstock. With a flow rate of 55 to 80 LHSV the short bed did reduce the decomposition rate of the effluent a significant amount but not to the degree possible with larger beds.

With a ratio of l/d of 0.4 and with flow rates of 15 to 30 LHSV (linear velocities approaching those of the preceding runs in terms of cm/sec) the effluent had a decomposition rate as low as that measured in the pretesting of the sample vessel (approximately 0.0006 day^{-1} at 100°C according to previous measurements).

Since the above bed size is considerably below that recommended for more normal type of ion-exchange systems a compromise size of about 2.5 l/d will be incorporated into the design of the larger apparatus. Provision will also be made for extending the bed to about 7 l/d by using a second chamber intended also as a holder for a dual ion-exchange medium.

Experiments were also run in order to determine the effect of contact of the feed HP with aluminum or steel prior to passage through the ion-exchange bed. As shown in Table 2 the contact with aluminum surface (type 1100) at room temperature prior to passing through the bed did not contaminate the feed HP to the extent that it could not be stabilized by ion-exchange. However, contact with stainless steel surface (type 347) at S/V ratio of about 0.3 cm^{-1} for a period of several hours did appear to cause an increase in the decomposition rate of the effluent. For this reason the design of the larger apparatus avoids all use of metal surface and instead consists of Kel-F and Teflon on all exposed surfaces with the exception of the glass filters.

The ion exchange bed support has been constructed of stainless steel as shown in Figure 3. Kel-F coating is being applied to the inner surfaces and Teflon fittings and gaskets will be used to make the connections to tubing and seal the pyrex filters in place.

A Flexiliner pump, with Teflon block and Kel-F liner will be used to feed the HP to the ion exchanger with Teflon fittings to make connections to the Kel-F tubing. Lubrication of the pump bearings and outside lining

a) "Storable Concentrated Hydrogen Peroxide", Quarterly Progress Report No. 4, February-April 1965, TR-S-13961, Contract DA-04-200-AMC-569(Z).

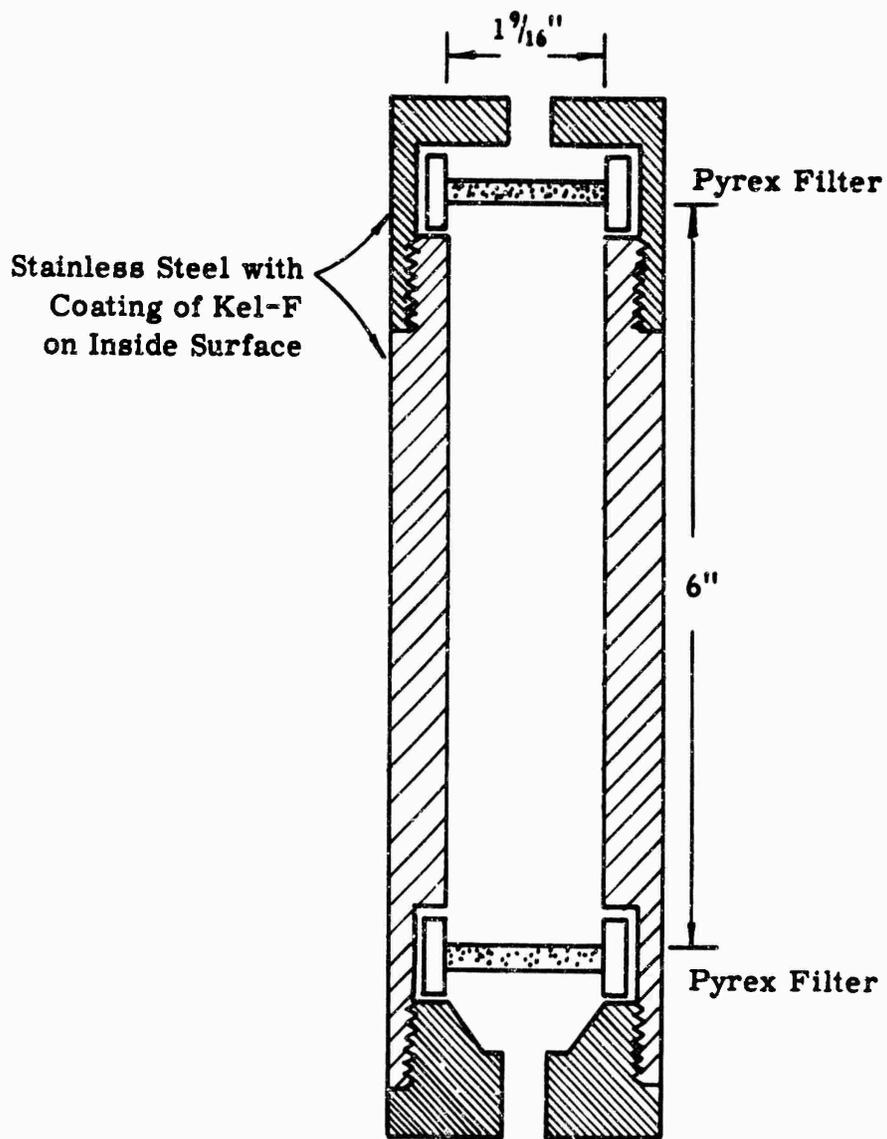


Figure 3. ION EXCHANGE BED SUPPORT

Table 2. DECOMPOSITION RATES OF ION-EXCHANGE EFFLUENT AT 100°C

Ref No. e)	Bed Size		Effluent, Accumulative			Sample No.	pH ^{c)}	Decomposition, d) Day ⁻¹ at 100°C		
	cc	l/d	ml	Bed Vol	LHSV ^{b)}			Pretest	Sample	Stabilized
With β -Stannic Acid Solids, 6										
26	1.4	0.16	200	148	115	199	-3.76	0.0010	0.0076	0.0037
	1.4	0.16	350	260	148	200	-3.75	0.0009	0.0028	0.0023
	1.4	0.16	600	429	79	211	-0.97	0.0015	0.0017	0.0015
	1.4	0.16	700	500	55	212	-0.67	0.0010	0.0019	0.0013
	1.4	0.16	800	571	60	213	-0.54	0.0021	0.0030	0.0022
27	4.2	0.43	300	71	15	217	-3.30	0.0013	0.0012	0.0014
	4.2	0.43	400	95	30	218	-2.50	0.0012	0.0010	0.0013
With β -Stannic Acid Solids, 2; Feed in Contact With Aluminum Type 1100										
24	8.1	6.2	500	61	3.2	201	-1.76	0.0012	0.0011	0.0013
	8.1	6.2	600	74	2.9	202	-1.48	0.0011	0.0013	0.0013
With β -Stannic Acid Solids, 2; Feed in Contact With Stainless Steel Type 347										
24	8.1	6.2	750	93	3.4	214	-1.09	0.0018	0.0020	0.0019
	8.1	6.2	900	111	2.9	216	-1.15	0.0011	0.0017	0.0018

- a) The ratio l/d is the length to average diameter and is approximate.
- b) Liquid hourly space velocity in terms of bed volumes per hour.
- c) Direct reading with glass-Calomel cells. pH of the feed HP-98 is about 0.0; most stable region would be from 0 to about -2.0.
- d) Values are uncorrected for the contribution by the vessel walls.
- e) Becco HP-98 used as feed. Decomposition of untreated feed approximately 0.0025 day⁻¹ at 120°C, corrected for the effect of the vessel walls.

surface will be with fluorolube oil as a precaution against possible leak of HP into these regions and the formation of explosive mixtures.

Distillation

In order to produce larger samples of flash distilled HP of 90-98% strength so that tests of stability may be made without the interference of a relatively high surface/volume ratio of the container a vacuum distillation apparatus has been assembled according to the schematic diagram shown in Figure 4. The apparatus is designed so that the distilled HP comes in contact only with pyrex glass, Kel-F tubing and Teflon fittings in order that the metallic ion contamination may be kept at a minimum. The feed HP is also kept away from contact with steel as only aluminum lines and fittings are used between the feed vessel and the still and on the bottoms take-off lines. A spray disengaging chamber is located above the thermosyphon reboiler in order to prevent carry-over from the feed to the product.

The stillhead is constructed in two parts in order that, with partial condensing in the first, the concentration of the condensate may be kept high with some water and HP carrying through to the second condenser. Although several distillation plates could be used to make this separation with more exactness the more simplified design is being tried first in order to reduce the pressure drop between the reboiler and final condenser. The reduced pressure will allow a greater safety factor against vapor phase HP explosions by allowing operation of the reboiler at a lower temperature. According to measurements previously made in these laboratories^{a)} operation at pressures below 75 torr for HP-90 (50 torr for HP-100) and about 80°C should be possible without danger from spontaneous vapor phase explosions. Explosion of HP vapor at this pressure, if it occurred, would be barely audible and would not result in significant pressure rise.

Besides control of the pressure of operation, other safety factors which have been incorporated into the design are 1) a feed reservoir integral with the reboiler in order to keep the liquid volume relatively high without having large volumes of heated liquid, 2) steam heat in order to keep the absolute skin temperature low, 3) a water purge which can be fed quickly into the reboiler in case vapor burning does occur and 4) bottoms withdrawal to reduce the concentration of impurities which may occur in the reboiler. Temperature measurement and recording at various locations in the apparatus will be made.

Operation of this distillation apparatus will be with water feed followed by HP-90 feed until the apparatus and receivers have been thoroughly cleaned. Tests of the stability of the HP distillate will be used as an indication of the cleanliness of the apparatus.

a) Monger, J. M., Baumgartner, H. J., Hood, G. C., and Sanborn, C. E., Chem. Eng. Data 9, 119 (1964).

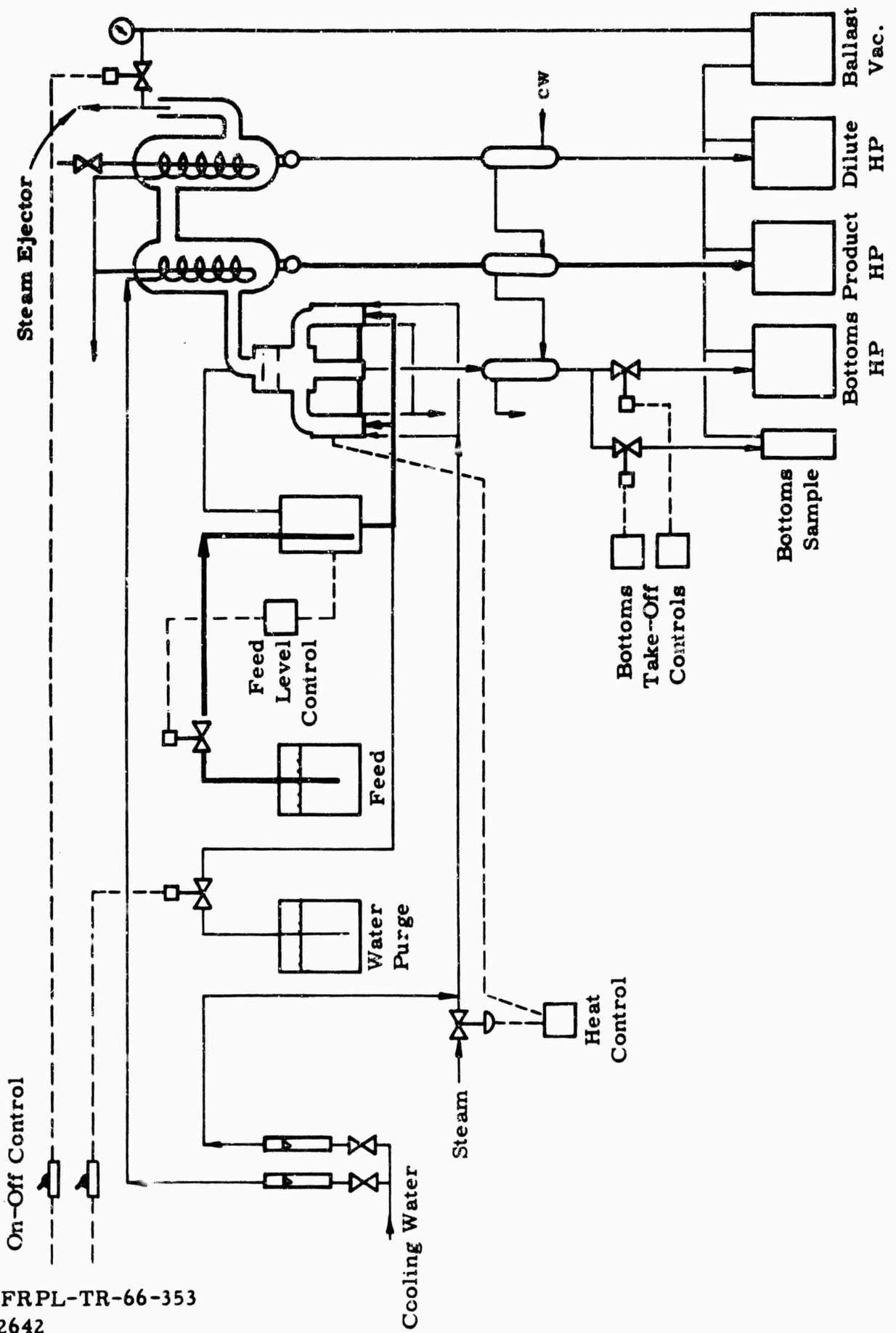


Figure 4. HYDROGEN PEROXIDE FLASH DISTILLATION APPARATUS

Studies of Container Surfaces

Surface Activities

Data reported for the surface activities of aluminum alloys 2014 and 2219 in the previous quarterly report^{a)} were lower than expected because of their copper content. New samples were therefore prepared by machining of specimens available and these samples were tested in HP without previous caustic or acid pretreatment. The results are given in Table 3.

The blank determinations in all tests were made by transfer of the HP to a separate flask since the test specimen formed in integral part of the surface activity test flask. In two sample tests this blank measurement was greater than the combined decomposition just previously made. In these two tests the value shown for the surface activity is less than the value calculated assuming that all of the decomposition occurred on the surface.

The surface activities for both 2014 and 2219 aluminum alloys, after contact with hot HP-90 for 10 to 20 hours, were relatively low considering the copper content of the alloy. However, as shown by the blank determinations, considerable dissolution of the contaminants had occurred such that these alloys would not be recommended for long term storage even after passivation with hot HP. One sample, at 100°C over a 28 hour period, showed an increase in the homogeneous decomposition from about 0.007 day⁻¹ to 0.015 day⁻¹ during this period.

In addition to the aluminum alloys tested, a fluorocarbon type surface coating material made by Diamond Chemical called Dalvor 720 was tested. This sample became very active during the initial warming-up period and was immediately discontinued. Examination showed that bleaching of the surface coating (normally black) had occurred. It is possible that this material had a metallic oxide as pigment which may have been leached into the HP.

No additional surface testing is planned

Storage Samples

The samples proposed for storage test include two separate purposes, 1) testing of the homogeneous stability to demonstrate means of purification of the HP and 2) testing of materials for use as containers or liners to demonstrate practical containers. Samples for demonstrating the homogeneous decomposition will be stored in pyrex carboys in order to take advantage of the low surface activity and the freedom from contamination by the glass.

Two samples have been prepared and installed in the 25°C bath: one sample of Becco HP-98 and one sample of du Pont HP-90. Both were filtered through a porcelain filter (BKH filter candles of 4.4 micron pore diameter cleaned with caustic and nitric acid and passivated with HP-90) in order to reject the aluminum oxide and stannate-metal-oxide precipitate which may have accumulated in the HP in the shipping container. Stannate stabilizer was added to each in the amount equivalent to 4 mg/g as tin plus nitric acid to adjust the pH. The stabilizer solution was prepared by dissolving the sodium

Table 3. SURFACE ACTIVITIES IN STABILIZED HP-90 AT 100°C

Sample No.	HP No. c)	Surface		S/V, cm ⁻¹	Decomposition Rate		
		Type	Preparation a)		Overall, Day ⁻¹	Blank, b)	k ₂ , cm/Day
198	A18-2	2014 Aluminum ^{d)}	1	1.05	0.348	0.012	0.320
198	A18-2	2014 Aluminum ^{e)}	1, 7	1.06	0.018	0.012	0.0057
226	A18-2	2014 Aluminum	None	0.86	1.73	1.62	0.129
226	A18-2	2014 Aluminum	7	0.81	0.024	0.024	<0.03
221	A18-2	2219 Aluminum	1	1.16	0.0258	0.0205	0.0046
221	A18-2	2219 Aluminum	1, 7	1.13	0.0112	0.0046	0.0059
235	A18-2	2219 Aluminum	None	1.14	0.0596	0.0708	<0.05
235	A18-2	2219 Aluminum	7	1.06	0.0199	0.0147	0.0050
254	A18-2	Dalvor 720	None	-	>5 ^{e)}	-	>5 ^{e)}

- a) Surface preparation by solvent degreasing (1) or by passivation with hot HP-90 (7).
- b) The vessel used could not be used for the blank. The HP was transferred to separate test flasks in order to determine the blank.
- c) HP-90 from drum A18 with Na₂Sn(OH)₆ at 34 x 10⁻⁶ m/l and HNO₃ at 69 x 10⁻³ m/l added as stabilizer.
- d) The test samples used were 3-in. diameter discs machined on the surface and fitted to a special test vessel.
- e) The surface became active during the initial warming-up period and was discontinued.

stannate in a small amount of the HP to be stored, partially neutralizing by addition of 1 mole nitric acid per mole of stannate so that stannic oxide polymerization is started, and then after adding this solution to the storage sample an additional mole of nitric acid per mole of stannate was added so that the resultant pH is nearly equivalent to the initial pH of the sample.

Small test samples of the 5-gallon storage samples were obtained in order to determine the stability by the accelerated test at 100°C. In both cases the results of this accelerated test were nearly identical to previous small sample tests made with these two HP stocks with the stated amount of stabilizer added, i.e., 0.0015-0.0018 day⁻¹ for Becco HP-98 and 0.0028-0.0029 day⁻¹ for du Pont HP-90 at 100°C, corrected for the heterogeneous decomposition caused by the sample test vessel.

The carboy samples were sealed by means of Teflon gaskets with Aclar film covering the metal cap as shown in Figure 5. Kel-F tubes were fitted to the cap with Teflon fittings and the assemblies were pressure tested at 6 psi in order to guarantee that no loss of decomposition gas would occur through leakage. The decomposition gas is transferred through Kel-F tubing to collecting burets for measurement.

Other storage samples in tin-plated vessels are in preparation. The tin-lined stainless steel vessels which were used previously for plating experiments were cleaned thoroughly with dilute caustic and 20-35% nitric acid in order to remove the rough treed formation of tin which had been put on one vessel by the previous plating procedure. The vessels were then plated for two hours at 20 amps in a stannous sulfate-sulfamic acid bath such that the cathode current density was about 0.6 amp/square decimeter. The electrolytic-tin anode was cylindrically shaped and of such area that operation was at 0.75 amp/square decimeter. Weight loss from the anode, presumed to have been gained in equal amount on the cathode, was sufficient to provide a tin plate of 0.0045 cm thickness. The plating was fine-grained in appearance and showed no signs of crystalline trees.

These vessels are being filled with stabilized HP-90 and will then be placed in storage. Additional vessels of aluminum will be plated also by the procedure reported in the previous quarterly report^{a)} and then placed in storage.

Other samples of HP-90 and HP-98 are planned for storage in pyrex vessels or Aclar-lined vessels. These will be described in a future report.

Program

The ion-exchange apparatus for preparation of 5-gallon samples of HP will be assembled and put into operation. Quantities of HP-98 and HP-90 will be treated in this apparatus and tested by the accelerated test for stability. When satisfactory operation is achieved several storage samples will be prepared, some in pyrex and some in Aclar-lined vessels.

a) "Storable Concentrated Hydrogen Peroxide", Quarterly Progress Report No. 2, AFRPL-TR-66-262, Contract AF 04(611)-11416.

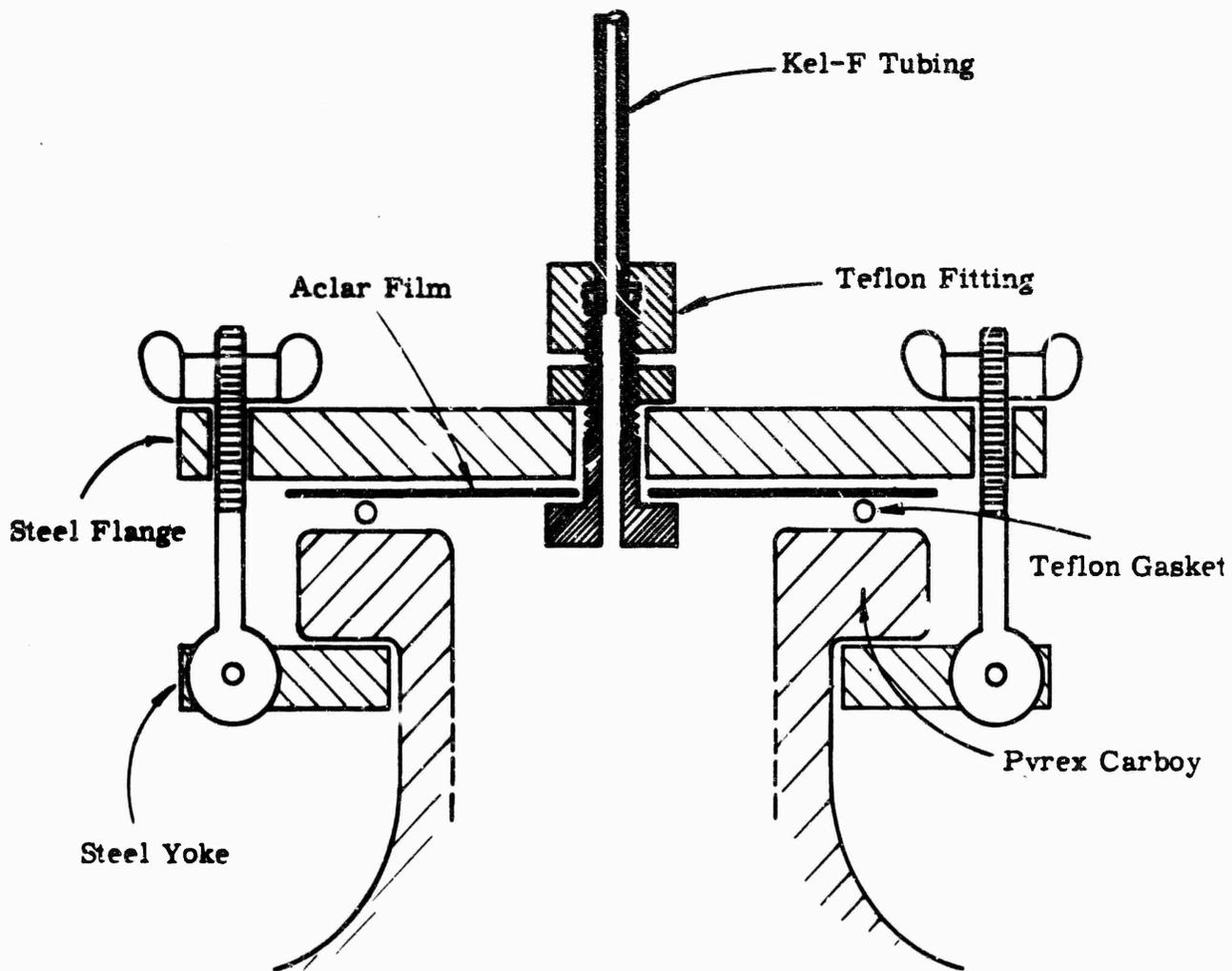


Figure 5. STORAGE SAMPLE CLOSURE

AFRPL-TR-66-353

Distillation of HP-90 and of HP-98 will be started and stability test samples will be run at the accelerated temperature. It is anticipated that considerable flashing of the distillation apparatus will be required before satisfactory stability of the HP distillate is achieved. Larger samples will then be prepared and placed in storage, two in pyrex and one in Aclar-lined vessel.

The vessels for the above storage samples have been prepared and linings of Aclar film will be made for those requiring lining.

Three aluminum vessels will be plated with tin and when of satisfactory appearance will be filled with HP-90 for storage test.

KEY WORDS

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Stabilization
Surface Activity Tests
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Container Linings
Decomposition Rates

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13. ABSTRACT Investigation of means for reducing the rate of decomposition of concentrated hydrogen peroxide (HP) by removing the catalytic elements carried in the HP and by reducing the catalytic activity of various materials of construction for use as containers has continued. A flash distillation unit has been constructed in order to prepare some 5-gal storage samples of HP purified by additional distillation. Samples of HP-98 prepared by distillation at low pressure and temperature will be prepared for storage. Samples of HP-90 will be distilled also and tested by the accelerated test. An ion exchange bed support unit has been designed and constructed and will be assembled with a feed pump in order to prepare other storage samples of HP-98. Additional storage samples have been placed in constant temperature baths. Included are two in tin-plated vessels to again test the activity of tin plated from a stannous sulfate-sulfamic acid bath.		