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RESEARCH MEMORANDUM

TRANSPORTATION OF LIQUID FLUORINE

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NATIONAL ADVISORY COMMITTEE
FOR AERONAUTICS

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SUMMARY

An investigation was conducted to determine whether a liquid-fluorine storage tank, designed and constructed under Air Force Contract AF 33(616)-2229, would be satisfactory for the transportation of liquid fluorine within the Lewis laboratory property. In these studies liquid fluorine was safely transported and stored. The investigation included 82 hours of transportation over both smooth and rough roadways in a total storage time of 2570 hours. Recommendations for improving the transportable tank are given.

INTRODUCTION

Liquid fluorine as a rocket oxidant has considerable promise of meeting the high performance requirements for long-range rocket missiles. At the present time, fluorine is supplied as a gas in cylinders containing approximately 6 pounds each at a pressure of 400 pounds per square inch gage. If, for example, fluorine in these containers was used for engines of 100,000-pounds thrust, about 33 cylinders would be required for 1-second operation. The shipping of small quantities of fluorine in separate cylinders under extremely high pressures (thus the handling of large numbers of cylinders) calls for considerable effort and results in a high cost of operation. This method is inadequate to meet the demands of medium- and large-scale experimental studies.

To study alternate methods of supplying fluorine, the U. S. Air Force contracted for a preliminary survey and design of a storage system for liquid fluorine. The specifications called for a system to serve as either a fixed or transportable tank of from 500- to 1000-pounds capacity. The completed portable storage tank (Air Force Contract AF 33(616)-2229) was used for a series of liquid-fluorine loading and storage tests at the construction site. At the suggestion of Headquarters, Wright Air Development Center and the NACA, the storage tank and equipment were loaned to the NACA Lewis laboratory to investigate the use of the tank under mobile conditions when loaded with liquid fluorine.

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APPARATUS AND PROCEDURE

Description of Apparatus

A complete description of the fluorine storage unit is given in reference 1 and a schematic diagram is presented in figure 1. The vessel consists of an inner tank for liquid fluorine (500-lb capacity) surrounded by a liquid-nitrogen bath. The liquid-nitrogen jacket can hold up to 100 gallons. A vacuum jacket, filled with granular powder insulation, between the liquid-nitrogen container and outer jacket ensures a low liquid-nitrogen loss rate. Two such containers with associated vacuum pump, weighing system, and electrical accessories were mounted on a flat four-wheel trailer.

The method of transferring both liquid nitrogen and liquid fluorine from the tank is by gas pressure. For the transfer of liquid nitrogen, gaseous nitrogen pressure is built up over the liquid by vaporization. The liquid fluorine may be transferred by building up the fluorine pressure by passing warm nitrogen gas through a warm-up coil located in the liquid-fluorine tank.

The nitrogen tanks are protected by a safety relief valve and a burst diaphragm. In addition, each fluorine tank is equipped with a pressure switch set to operate a buzzer alarm. The nitrogen level is monitored by a liquid-level gage, equipped with a switch that operates the same buzzer alarm when the liquid level falls below a set value.

The complete tank assembly is attached to a weighing system, which consists of two strain-gage load cells, cables, and indicating instrument. One side of the tank is supported by the two strain-gage load cells and the other side by a channel attached to the trailer bed. The channel serves as a flexible support.

Loading Fluorine in Tank

The procedure followed in filling the tank with fluorine was to manifold a number of fluorine cylinders (400 lb/sq in. gage, 6 lb net each) and empty these cylinders into the tank by opening one cylinder at a time. The fluorine feed rate to the tank was controlled by an air-operated control valve. During the exploratory feeding of the first two cylinders, each was opened to full 400 pounds per square inch on the manifold with the control valve about one-eighth open. Subsequent cylinders were emptied individually by opening them to about 150 to 200 pounds per square inch manifold pressure and using the control valve about one-fourth open for actual throttling. As the cylinder emptied, the manifold pressure was kept in this range as long as possible; then the cylinder valve was opened wide and the system allowed to discharge to the tank to 10 pounds per square inch gage.

This procedure was used to feed 100 cylinders to the tank over a 3-day period. The actual weight of material transferred was calculated as 586 pounds, because of the residual pressure of 10 pounds per square inch gage remaining in the cylinder. The amount of liquid nitrogen used to condense the initial 293 pounds of fluorine was 66 gallons or 1.51 pounds of nitrogen per pound of fluorine. The theoretical quantity of liquid nitrogen required to condense 1 pound of fluorine from 298° K is 1.72 pounds. This value is based on the heat of vaporization of nitrogen. If nitrogen gas (vaporized by the condensation of fluorine) is heated to 298° K by means of a heat exchanger, the theoretical amount of liquid nitrogen required for the condensation of 1 pound of fluorine is 0.76 pound. The flow rate of fluorine into the tank during the loading varied from 111 to 200 pounds per hour. Detailed data of the fluorine loading procedure is presented in the appendix.

RESULTS AND DISCUSSION

Liquid-Oxygen Storage

The storage tank was loaded with liquid oxygen at Baton Rouge, Louisiana, and driven to the Lewis laboratory, Cleveland, Ohio. Measurements of the liquid-nitrogen level, pressure in the product tank, and vacuum in the insulating jacket were taken during the trip and at the Lewis laboratory. Data taken during the trip are presented in table I. The total time of the trip was about 100 hours with 38 hours of actual driving time. No nitrogen was added during the trip. The nitrogen loss in percentage per day and the heat loss in Btu per hour during the trip are shown by figure 2(a). The loss rate of nitrogen per day based on a full nitrogen jacket varied from an initial rate of about 5.5 percent at the beginning to 4.25 percent after 80 hours. The nitrogen loss rates correspond to a heat leak of from about 131 to 103 Btu per hour.

Measurements to determine the liquid-nitrogen loss rate with liquid oxygen in the product tank were continued at the Lewis laboratory. The tank was kept stationary without the addition of further liquid nitrogen to the jacket. Figure 2(b) indicates values of about 3.6 percent nitrogen loss (heat leak of about 85 Btu/hr) after 250 hours of stationary tests.

The heat leak during transit with liquid oxygen in the tank was approximately 17 percent greater than under stationary conditions. This increase was probably due to greater heat rejection, caused by the sloshing of the liquid during transit and the weather conditions. Temperature during the trip varied from 39° to 62° F. The total time of liquid-oxygen storage, including the trip, was 434 hours (18 days), at which time, the liquid-nitrogen level gage indicated 35 gallons and a product pressure of -20 inches of mercury. The tests with liquid oxygen were considered satisfactory, and the liquid oxygen was transferred. Oxygen pressure for transfer was built up by passing warm nitrogen gas through the warm-up coil. The time required to build up sufficient pressure was extremely long.

Liquid-Fluorine Storage

After the tank was loaded with liquid fluorine, the entire unit was inspected and found to be unaffected by the fluorine. For the mobile tests the trailer upon which the storage tank was supported was attached to laboratory trucks. The driving was done in two laboratory areas, the main area, which permitted relatively smooth driving up to speeds of 30 miles per hour, and an adjoining area with extremely rough terrain, which allowed maximum speeds of 10 miles per hour. The data taken during the mobile test with liquid fluorine in the tank are presented in table II. 3896

The total time of storage as of August 17, 1955, was about 2570 hours (107 days) with 82 hours under transit conditions. No difficulty was encountered during the entire program, indicating that the storage tank is satisfactory for the transportation of liquid fluorine. The pressure in the fluorine tank varied from only -17 to -19 inches of mercury during the entire program. The loss rate of the liquid nitrogen and the heat leak during this test are given by figure 2(c). The values varied from an initial value of about 4.5 to 3.75 percent per day after 150 hours, corresponding to a heat leak of from 109 to 90 Btu per hour. The heat leak during the stationary storage of liquid fluorine obtained from reference 1 indicates values of about 109 Btu per hour.

Recommendations for Improvements

The following items would be considered for the improvement of the liquid-fluorine portable vessel:

(1) Pressure gages designed to withstand the vibrations encountered during transit should be used on the fluorine system. In addition, it might be advantageous to separate the gage from the product tank by means of a packless valve. The pressure gages and related equipment should be introduced to the vapor side of the product tank rather than the liquid side.

(2) A more suitable load-cell weighing system should be designed for the tank. It would be highly desirable to be able to check the loss of liquid nitrogen and to be able to transfer the required amounts of liquid fluorine when necessary. The frame of the present design was

apparently twisted during transit; furthermore, the design is not easily adaptable to the leveling necessary for reading the load cell. A comparison of the liquid-nitrogen loss obtained from the level indicator and from the load cell is shown by figure 3.

(3) The storage tank should be mounted on an approved highway trailer and should be positioned to permit access to the related equipment.

3896 (4) The warning system should be modified to permit a separate warning for each storage tank, and some means should be incorporated to distinguish between warnings in the liquid-fluorine tank and in the liquid-nitrogen cooling system.

(5) The method presently used to pressurize the liquid-fluorine tank requires the removal of most of the liquid nitrogen and, at best, takes several hours. To permit the loading of rocket propellant tanks with liquid fluorine in a short time, a more efficient heat-exchange system should be included on the nitrogen cycle.

SUMMARY OF RESULTS

Liquid fluorine, was safely transported and stored at the Lewis laboratory in a storage tank designed and constructed under Air Force Contract AF 33(616)-2229. Tests included 82 hours of transportation in a total 2570 hours of storage.

Lewis Flight Propulsion Laboratory
National Advisory Committee for Aeronautics
Cleveland, Ohio, September 22, 1955

REFERENCE

1. Siegmund, J. M.: Research on the Production and Handling of Fluorine. Prog. Rep. No. 7, General Chem. Div., Allied Chem. & Dye Corp., Feb. 1, 1955. (Contract AF 33(616)-2229.)

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APPENDIX - FLUORINE FEED TO STORAGE TANK

The following data describe the transfer of gaseous fluorine to the portable storage tank. This history covers the filling of tank number 1 with liquid fluorine at the Lewis laboratory:

April 26, 1955[a]

Time	Liquid-fluorine tank pressure, in. Hg	Nitrogen level, gal	Fluorine condensed, lb		Remarks
			Cylinders	lb	
Initial	-28.5	82	--	--	
1435	Started fluorine feed	--	--	--	
1440	-17.5	--	1	6	
1443-45	-17	--	2	12	
1445	-15	--	4	24	
1457	-13	--	5	30	
1502	-11	--	7	42	
1510	-10	--	10	60	
1511[b]					
1600	-18	70	10	60	Equilibrium conditions

[a]Daily feed, 60 (405/415) = 59 lb gaseous fluorine; flow rate for cylinders 3 to 10, 111 lb/hr; nitrogen consumption, 12 gal or approximately 1.4 lb nitrogen per lb gaseous fluorine.

[b]Stopped feeding and liquid fluorine tank pressure dropped to -16 in. Hg.

April 27, 1955[a]

Time	Liquid-fluorine tank pressure, in. Hg	Nitrogen level, gal	Fluorine condensed, lb		Remarks
			Cylinders	lb	
1000	-18	87	--	--	Initial
1045	Started feeds	--	--	--	
1050	-15	--	3	18	
1055	-13	--	5	30	
1105	-12.5	--	9	54	
1115	-10	--	12	72	
1127	-13	--	17	102	
1135	-11.5	--	19	114	
1143	-11	57	23	138	Stopped feeds
1303	-18	57	23	138	Resumed feeds
1320	-6	--	31	186	
1328	-3.5	--	34	204	
1330	-2	--	35	210	Slowed down feeds; low N_2 level
1333	-4	--	36	216	
1338	0	--	38	228	
1345	0	--	40	240	Stopped
1500	-17.5	33	40	240	Static equilibrium with only 33 gal N_2

[a]Daily feed, 240 (405/415) = 234 lb gaseous fluorine; flow rate for cylinders 1 to 34, 144 lb/hr; flow rate for cylinders 35 to 40, 127 lb/hr; nitrogen consumption, 54 gal or approximately 1.56 lb nitrogen per lb gaseous fluorine.

April 28, 1955[a]

Time	Liquid-fluorine tank pressure, in. Hg	Nitrogen level, gal	Fluorine condensed, lb		Remarks
			Cylinders	lb	
1415	-18.5	98	--	--	Start
1425	-15	--	4	24	
1435	-13.5	--	9	54	
1443	-13	--	13	78	
1452	-13	--	17	102	
1501	-13	--	21	126	Increase feeds
1512	-12.5	--	26	156	
1519	-12	--	30	180	
1525	-12	--	34	204	(b)(c)
1531	-11	--	37	222	
1544	-9	--	43	258	
1552	-7	--	47	282	
1602	2[d]	--	50	300	Stopped
1640	-16	47	50	300	Not yet at equilibrium

[a]Daily feed, 300 (405/415) = 293 lb gaseous fluorine; flow rate for cylinders 1 to 21 164 lb/hr; flow rate for cylinders 22 to 34, 200 lb/hr; nitrogen consumption indeterminate because of concurrent liquid nitrogen addition. Total gaseous fluorine fed to tank number 1, 586 lb.
[b]Added more nitrogen; level too low for good transfer.
[c]After 1525 time, some interruptions in feed were caused by truck traffic through area.
[d]lb/sq in. gage

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TABLE I. - DATA OBTAINED DURING TRIP FROM BATON ROUGE, LOUISIANA, TO CLEVELAND, OHIO, WITH LIQUID OXYGEN IN TANK

Data	Time	Liquid-nitrogen level, gal	Liquid-oxygen tank pressure, in. Hg	Vacuum in jacket, micron
3/17/55	0830	$96\frac{1}{2}$	-23	36
3/17/55	1830	93	-23	--
3/18/55	1230	90	-23	36
3/18/55	1900	90	-23	--
3/19/55	0530	89	-23	36
3/19/55	1600	86	-23	--
3/20/55	1700	81	-23	31
3/21/55	0700	80	-22	--
3/21/55	1000	Arrived at Lewis laboratory		

TABLE II. - MOBILE TESTS WITH LIQUID FLUORINE

Date	Start of run, time	End of run, time	Liquid-nitrogen level, gal	Cumulative driving time	
				hr	min
5/2/55	1103	1143	85		40
	1245	1325	83.5		
	1335	1400	--		
	1415	1430	--		
	1450	1520	83	2	30
	1800	1900	84	3	30
	1905	2005	84	4	50
5/3/55	1040	1110	81	5	0
	1120	1140	81	5	20
	1300	1330	81	5	50
	1335	1400	81	6	15
	1415	1445	80	6	45
	1515	1600	79	7	30
	1610	1640	79	8	0
	1915	2015	80	9	0
	2045	2105	80	9	20
5/4/55	0905	0935	78	9	50
	0945	1025	78	10	30
	1115	1130	78	10	45
	1230	1320	78	11	35
	1330	1420	78	12	25
	1430	1515	78	13	10
	1525	1635	78	14	20
	2040	2125	75	15	0
5/5/55	0035	0645	75	21	15
	0900	0945	73	22	0
	0955	1050	72	22	55
	1100	1145	72	23	40
	1230	1330	72	24	40
	1335	1410	72	25	15
	1415	1500	72	26	0
	1505	1610	72	27	0
	1615	1645	72	27	35
	1655	1810	72	28	50
	1810	2350	72	34	30

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TABLE II. - Concluded. MOBILE TESTS WITH LIQUID FLUORINE

Date	Start of run, time	End of run, time	Liquid-nitrogen level, gal	Cumulative driving time, hr	Cumulative driving time, min
5/6/55	0900	0935	71	35	0[a]
	1030	1200	69	36	35
	1245	1630	68	40	20
	1735	2335	66	46	20
5/9/55	1005	1045	59	47	0[b]
	1050	1155	59	48	5
	1230	1630	57	52	5
	1815	2340	55	57	30
5/10/55	0045	0345	55	60	30
	0415	0730	53	63	45
	0830	0915	54	64	30
	0920	1040	54	65	50
	1045	1145	54	66	50
	1230	1915	53	73	35
5/11/55	0845	1630	50	81	15
5/12/55[c,d]					
5/14/55[c]			42		
5/15/55[c]			38		
5/17/55[c]			31		

[a]Check weight on load cell, 319 lb; vacuum in jacket, 50 micron.
[b]Check weight on load cell, 263 lb; vacuum in jacket, 50 micron.
[c]No transportation
[d]Check weight on load cell, 170 lb; vacuum in jacket, 50 micron.

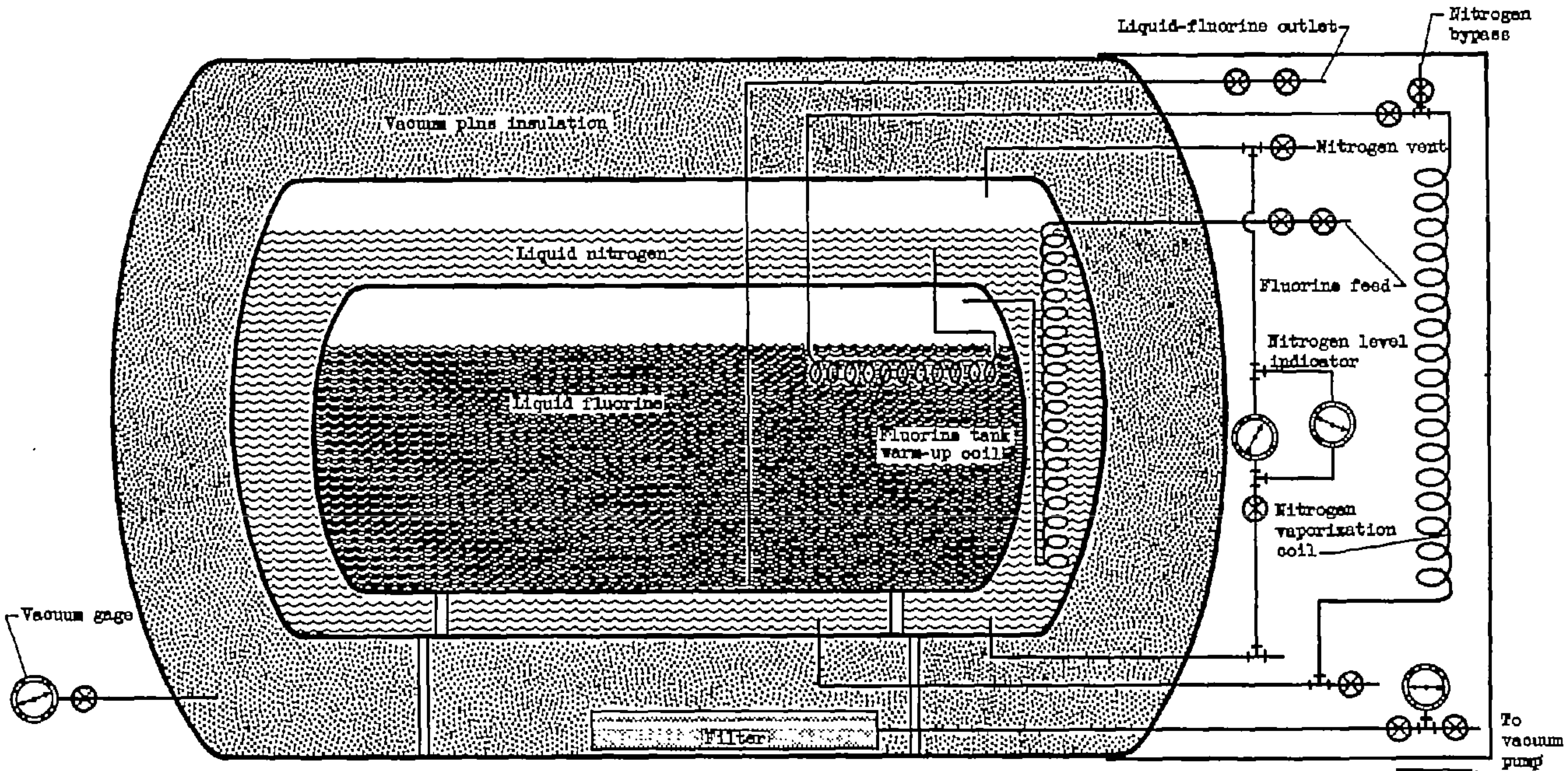
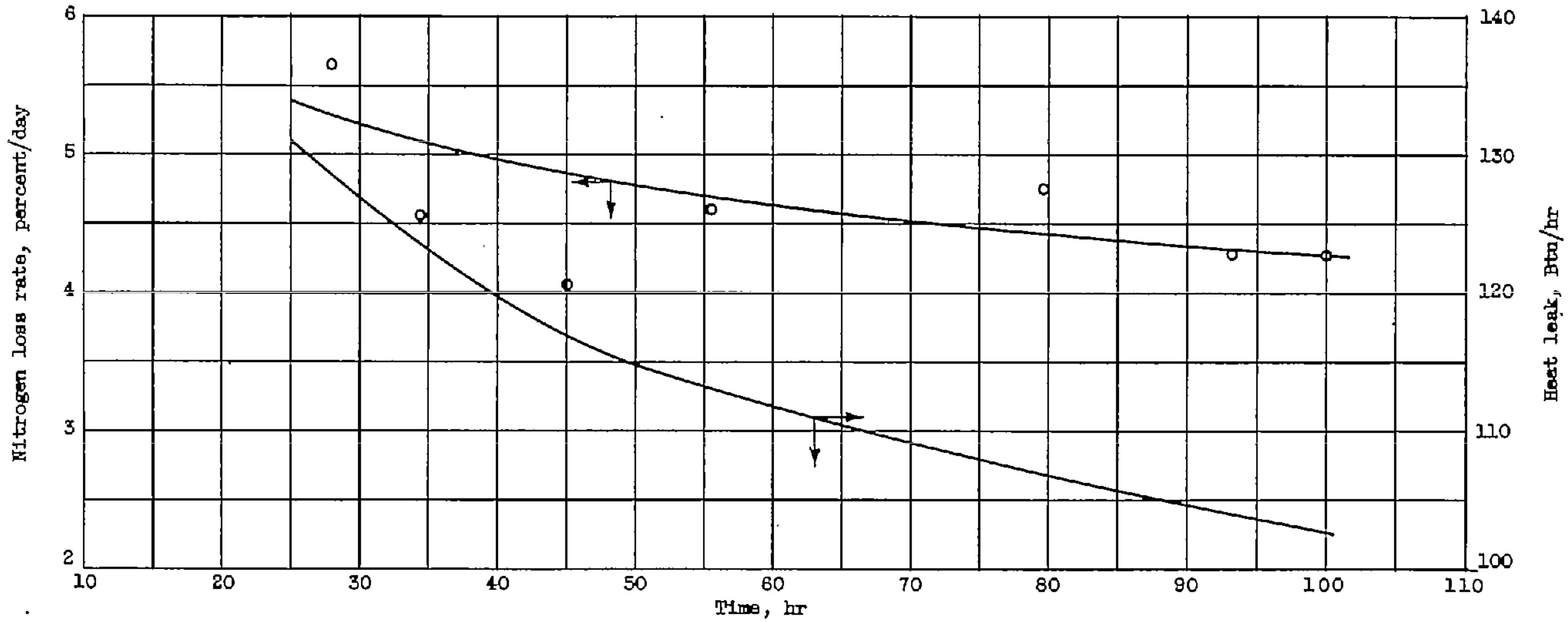
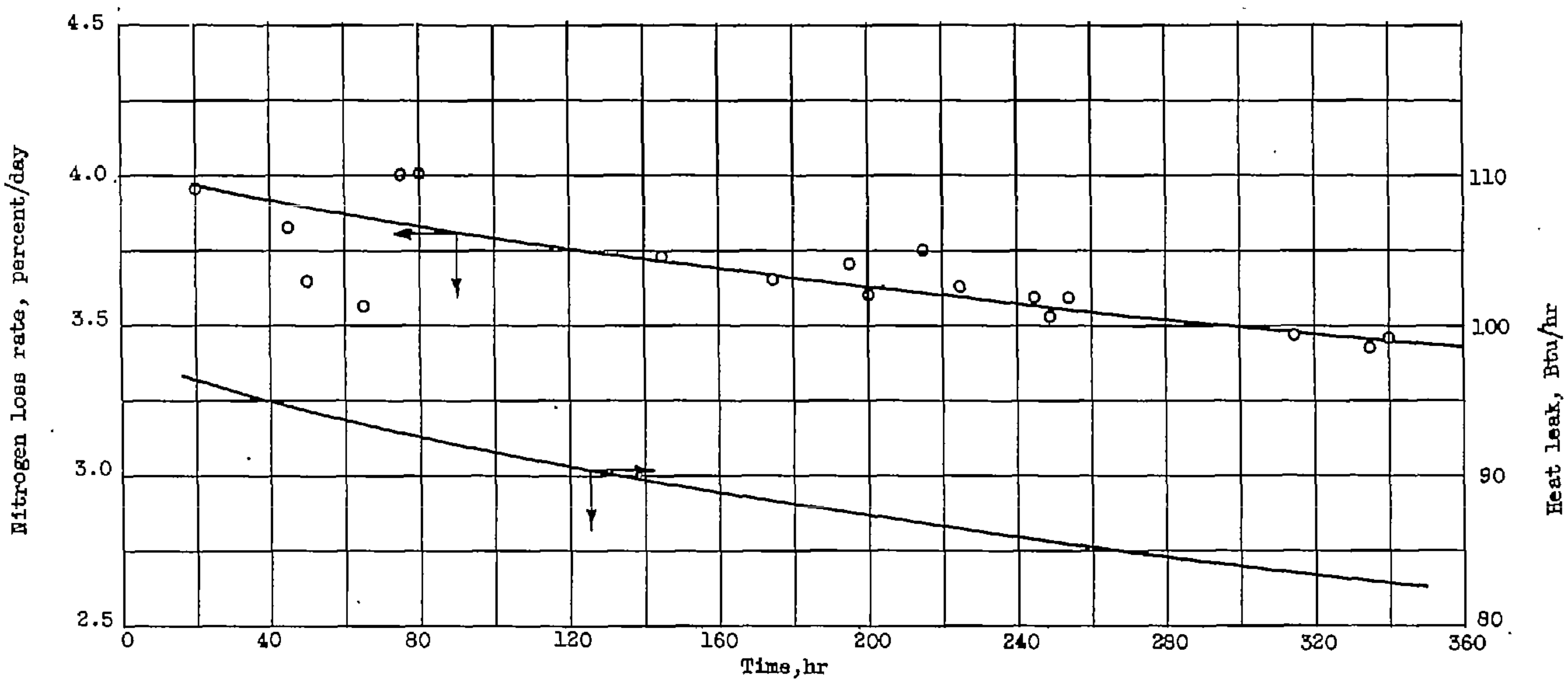


Figure 1. - Liquid-fluorine storage vessel.



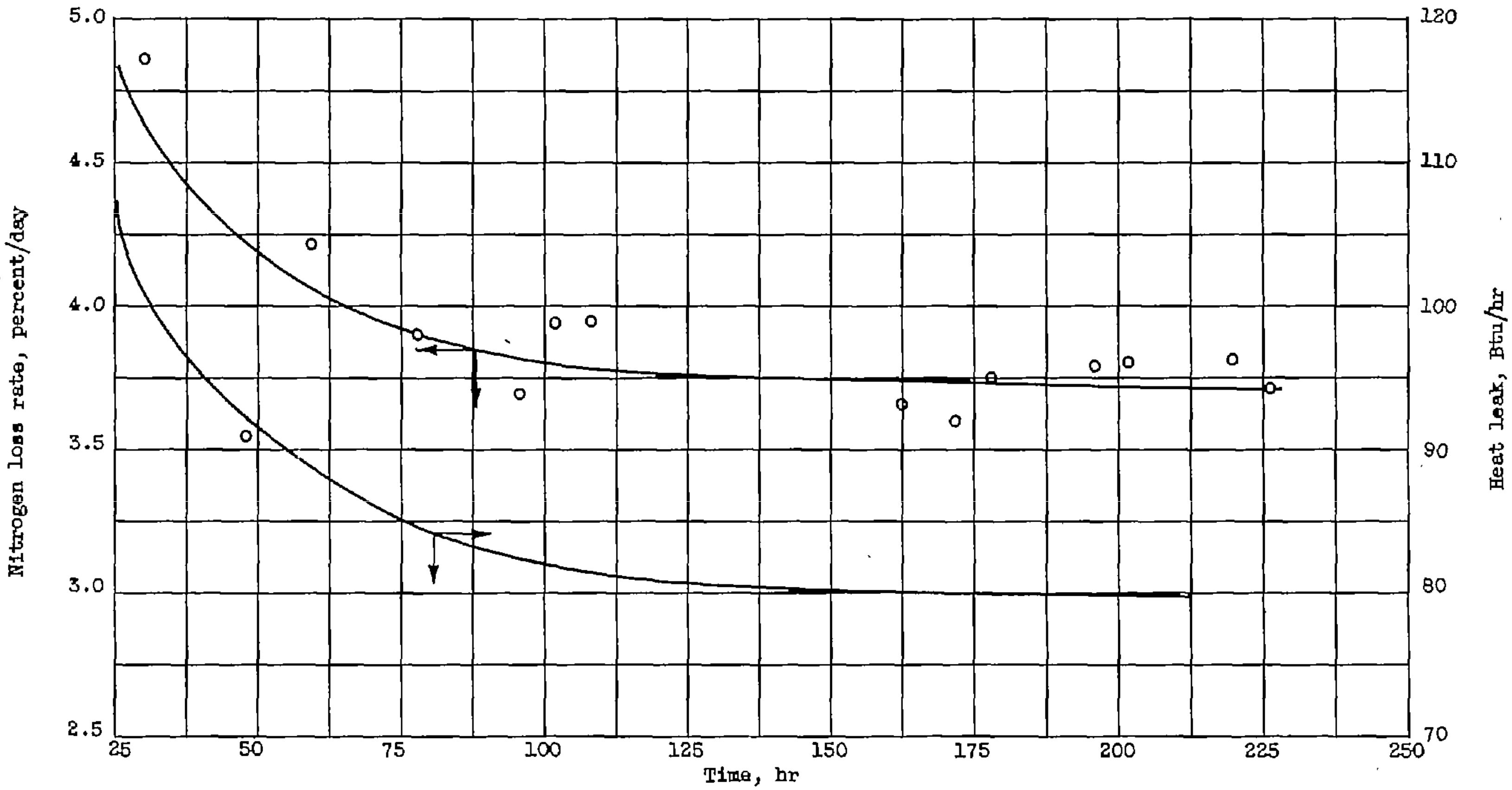
(a) Transportation from Baton Rouge, Louisiana, to Lewis laboratory; oxygen in tank.

Figure 2. - Liquid-nitrogen loss rate based on full jacket under various conditions.



(b) Stationary at Lewis laboratory; oxygen in tank.

Figure 2. - Continued. Liquid-nitrogen loss based on full jacket under various conditions.



(c) Transportation at Lewis laboratory; fluorine in tank.

Figure 2. - Concluded. Liquid-nitrogen loss rate based on full jacket under various conditions.

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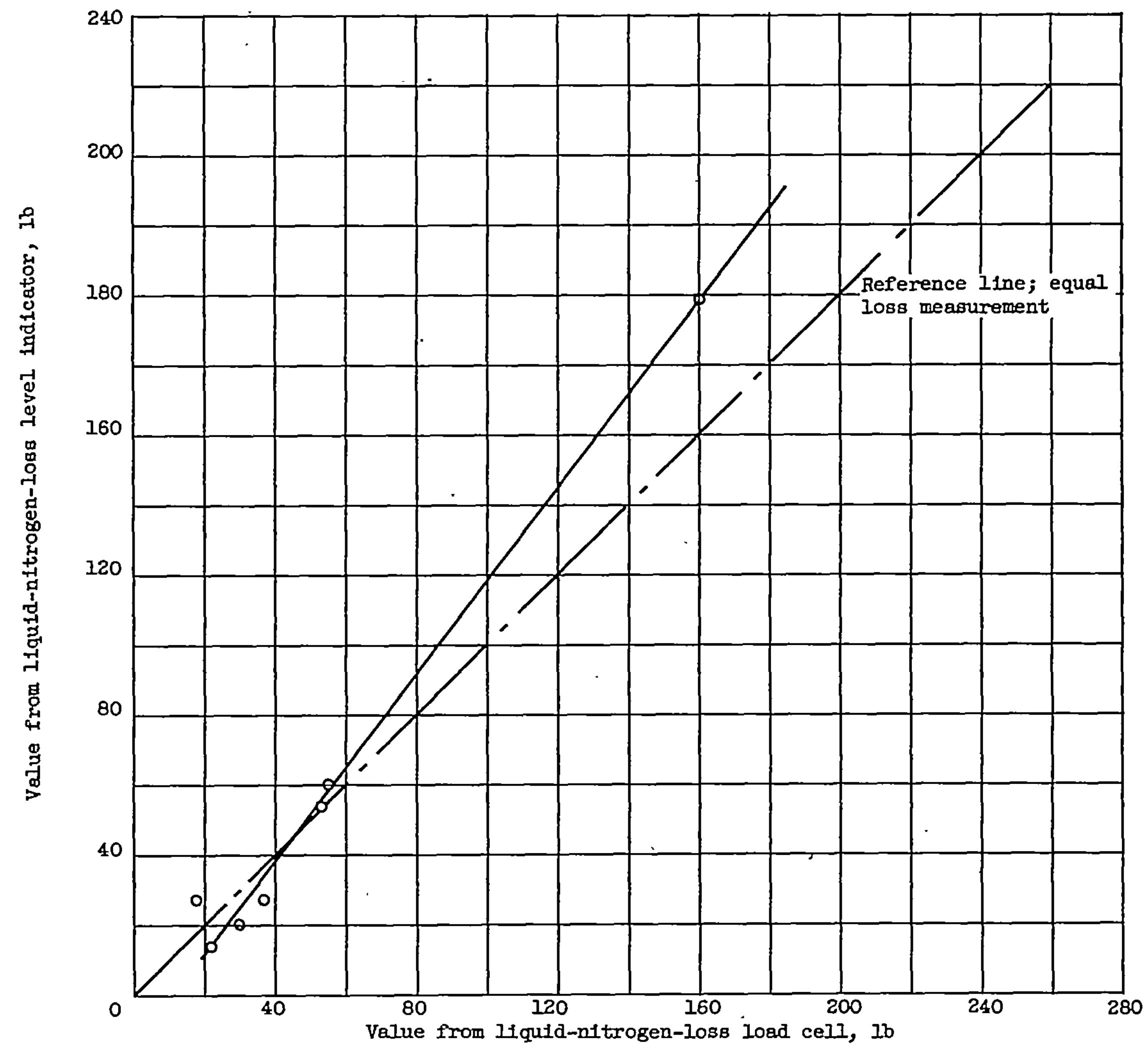

Figure 3. - Comparison of liquid nitrogen loss determined by level indicator and load cell.

NACA - Langley Field, Va.