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SUBJECTING SIMULATED LUNAR ROCK SAMPLES TO VACUUM AND THERMAL ENVIRONMENT

FINAL REPORT

September 1968

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FOREWORD

This document is prepared in accordance with Contract NAS9-8375. This report describes and documents, in narrative form, the task of subjecting simulated lunar rock samples to a vacuum and thermal environment for the purpose of removing entrapped gases.

CONTENTS

OBJECTIVE

The objective of this program was to expose approximately 25 kilograms of simulated lunar rock samples, supplied by the government, to a vacuum and thermal environment for thirty (30) days. The purpose of this environmental exposure to the samples was to remove entrapped gases. The required vacuum level was that which was achieved using an ion pumped system capable of attaining 1×10^{-9} torr under a gas load of 60,000 liters per second. The temperature was to be raised and maintained at 300°C + 25°C.

Upon completion of the thirty-day exposure the lunar rock samples were to be transferred to sample containers which would be placed in an oven and connected to the vacuum system. The containers would then be subjected to the full pumping capacity of the vacuum system for eight hours. During this period, the oven temperature would be increased and maintained at 300°C + 25°C. At the completion of the final bakeout, the sample containers, containing the treated lunar rock samples, would be hermetically sealed and shipped to the Manned Spacecraft Center.

SUMMARY

The thirty (30) day exposure of the simulated lunar rock samples, extended to thirty-one (31) days and was accomplished from 5 August 1968 through 4 September 1968. The extension was provided to recover a sixteen (16) hour period during which only the temperature environment was interrupted. The vacuum environment was not interrupted. The vacuum and thermal environment during this exposure is shown on Figures 1 and 2 respectively.

These data show that the pressure inside the vacuum chamber reached the 10^{-6} torr range during the period; the temperature reached and was maintained at 305°C. Upon completion of the thirty (30) day period the temperature environment was decreased to 149°C while continuing to pump the vacuum chamber with the ion pumps. The chamber pressure at this time reached 10^{-7} torr.

The vacuum chamber was then returned to atmospheric pressure while the temperature was maintained slighly above room temperature. Dry nitrogen gas was used to repressurize the chamber to minimize the admission of moisture to the lunar rock samples. The temperature of the lunar rock samples was kept above room temperature throughout the transfer from the sample trays to the sample containers, again to minimize the absorption of moisture into the samples from the room environment.

The lunar rock sample transfer from the trays into the containers was accomplished as rapidly as possible. The lunar rock samples were extremely dry, emitting dust as they were transferred into the final bakeout containers. The samples, however, were very difficult to transfer. The intended pouring method from trays to containers was not possible without the assistance of "spooning" of the lunar rock samples and tapping of the trays. The simulated lunar rock samples acted as "damp, sticky soil" not sliding from the sample trays to the containers without physical assistance. It was also noted that the dust, emitted during transfer of the smaller diameter samples, was irritating to the eyes and breathing passages.

The sample containers, after filling, were immediately connected to the vacuum manifold inside the oven. The final bakeout was then performed for a nine (9) hour period. The pressure attained, utilizing the full pumping capacity of the ion-pumped vacuum chamber was 10^{-6} torr with an oven temperature of 315°C.

At the completion of this final bakeout period the sample containers were hermetically sealed, utilizing a pinch-off tool. As each container was sealed off the temperature and pressure were recorded and are tabulated in Table 1.

DESCRIPTION AND EVENTS

This program was conducted using a nominal 4x8-foot ion-titanium pumped vacuum chamber which has a pumping speed in excess of 60,000 liters per second when operating at maximum capacity. Prior to placing the simulated lunar rock samples into the vacuum chamber for the thirty (30) day exposure to vacuum and thermal environment, the chamber was thoroughly cleaned. The temperature shroud was then installed and a pre-test bakeout at 315°C at a pressure of 3×10^{-4} torr was accomplished. This bakeout served to clean and dry the vacuum chamber and shroud and to verify the operation of the heated shroud and temperature control equipment. The temperature was then allowed to return to ambient and the chamber pressure was decreased into the 10^{-6} torr range.

A residual gas analysis spectrum was then taken to assure that the test chamber was free of hydrocarbons. The results of this gas analysis verified the chamber cleanliness and showed only trace quantities of water, methane, nitrogen, argon and hydrogen.

The simulated lunar rock samples were then placed in the sample trays (Figures 3, 4, 5, 6 and 7 show this installation). The distribution of the rock samples is presented in Table 2. The thirty (30) day vacuum and thermal exposure was initiated on 5 August 1968. The chamber pressure is presented in Figure 1 and the temperature environment is presented in Figure 2. The titanium sublimator filaments were expended during the first five (5) days of operation in an attempt to reduce and maintain the system pressure in the ion pump range (10^{-5} torr) as rapidly as possible. After reaching 10^{-5} torr and while operating with only the ion pumps the shroud temperature was increased at a rate which did not allow the gas load, resulting from the rock samples, to exceed the pumping speed of the ion pumps (900 liters/second). The allowable rate of temperature rise after ten (10) days of operation was not sufficient; therefore, the cold trapped roughing system was used to assist the ion pumps while the temperature was increased to 350°C. After three (3) days of chamber evacuation, using both the cold trapped roughing system and the ion pumps, the temperature was reduced to 300°C while operating with only the ion pumps. The thirty (30) day exposure was continued in this mode through completion. A sixteen-hour interruption occurred during the 23 days. The temperature environment was lost due to a facility electrical power momentary failure. This brief failure, a result of an electrical storm, caused the heater power supply breakers to open. The chamber pressure was not interrupted. One extra day was added to the required thirty-day exposure to recover this interrupted period. The exposure period was, therefore, completed on 4 September 1968. The chamber pressure during the exposure reached 5×10^{-6} torr while the temperature environment was stabilized at 305°C.

Upon completion of the required thirty (30) day exposure, the temperature was reduced to 149°C. System pumping was continued during this period using the ion pumps. System pressure reached 1.7×10^{-7} torr at the reduced temperature.

The system was then repressurized using dry nitrogen to minimize the admission of moisture to the rock samples. The temperature was reduced and maintained at approximately 32°C throughout the sample transfer operation to again minimize the absorption of moisture from the room environment into the rock samples. Figure 8 shows the samples inside the vacuum chamber immediately after the thirty (30) day exposure. Figures 9 and 10 show the transfer from the trays to the sample containers. The transfer of the rock samples into the containers required a six (6) hour period. The samples were kept inside the vacuum chamber with the door closed and at a temperature above room temperature during this period, except when an individual tray was transferred.

The transfer operation was difficult to accomplish using the planned pouring method. The samples would not slide from the sample trays and "spooning" of the samples and tapping of the trays were necessary to move the samples. The lunar rock samples were very dry, emitting dust as they were transferred; however, they acted "sticky" when pouring. The rock samples did not adhere to themselves or to the sample trays. The location of rock samples in the sample containers is presented in Table 3.

As the sample containers were filled they were immediately connected to the vacuum manifold for the final bakeout. After all containers were connected to the vacuum manifold, the oven was placed around the manifold assembly. The final bakeout was then performed utilizing the full pumping capacity of the ion-titanium sublimation pumping system while increasing the oven temperature. The system pressure and oven temperature are shown in Table 1.

The containers, placed in a 315°C oven, reached a maximum temperature of 280°C during the bakeout. This temperature, as recorded by a thermocouple attached to sample container No. 1, was the maximum attainable due to the large mass of the containers which were directly connected to the ambient vacuum chamber.

The sample containers were then hermetically sealed from the vacuum system. This seal was made with a pinch-off assembly at the inlet to each container. The containers were sealed in the following sequence and the pressure and temperature recorded just prior to sealing.

Container Number S/N	System Pressure Torr	Temperature °C
003	5.5×10^{-6}	170
010	5.0×10^{-6}	160
005	4.0×10^{-6}	140
002	2.8×10^{-6}	124
009	2.4×10^{-6}	105
008	1.5×10^{-6}	93
004	1.0×10^{-6}	93
006	8.5×10^{-7}	87
007	4.5×10^{-7}	87
001	2.5×10^{-7}	85

It should be noted that sample container No. 1 contains three samples of different types. These samples are separated inside the container with shim stock. In the bottom of this container is the Basalt 415 chips, the center sample is Andesite chips and the top sample is Dunite chips.

RECOMMENDATIONS

After encountering the difficulties of transferring the treated simulated lunar rock samples, an improved technique would be desirable to prevent extended exposure to the atmosphere and excessive handling of these samples. Future programs involving transfer of the treated samples should include improved techniques and equipment to accomplish the transfer.

TABLE 1

FINAL BAKEOUT

SYSTEM PRESSURE & TEMPERATURE

TIME		SYSTEM PRESSURE TORR	CONTAINER PRESSURE °C	REMARKS
2130	9/6/68	3×10^{-3}	41	Start of 8-hour bakeout
2210		2.2×10^{-3}	57	
2240		1.8×10^{-5}	127	
2310		2.5×10^{-5}	160	
2340		3.6×10^{-5}	167	
0010	9/7/68	4.5×10^{-5}	201	
0035		4.4×10^{-5}	243	
0120		4.4×10^{-5}	255	
0155		3.6×10^{-5}	265	
0220		3.1×10^{-5}	280	
0335		1.6×10^{-5}	280	
0410		1.3×10^{-5}	280	
0440		1.2×10^{-5}	280	
0510		1.0×10^{-5}	280	
0640		8.8×10^{-6}	280	Bakeout complete
0655		6.3×10^{-6}	181	*

* Upon completion of the eight (8) hour final bakeout the temperature was reduced to permit handling of the sample containers during the pinch-off operation.

TABLE 2

SIMULATED LUNAR ROCK SAMPLE DISTRIBUTION IN SAMPLE TRAYS

TRAY NO.		TRAY NO.	
101	Diorite 472 < 40	124	Knippa Basalt < 40 Mesh
102	Diorite 472 < 40	125	Diorite 472 > 40
104	Diorite 472 < 40	126	Diorite 472 > 40
105	Diorite 472 < 40	127	Diorite 472 > 40
106	Andesite < 40	128	Diorite 472 > 40
107	Andesite < 40	129	Diorite 472 < 40
108	Basalt 415 < 40	130	Diorite 472 < 40
109	Basalt 415 < 40	131	Knippa Basalt < 40 Mesh
110	Basalt 415 < 40	132	Knippa Basalt Chips
111	Andesite < 40	133	Knippa Basalt Chips
112	Dunite Chips	134	Knippa Basalt Chips
113	Dunite < 40	135	Knippa Basalt Chips
114	Dunite < 40	136	Dunite < 40
115	Basalt 415 > 40	137	Dunite < 40
116	Knippa Basalt > 40	138	Dunite < 40
117	Knippa Basalt Chips	139	Dunite < 40
118	Oceanic Basalt (Gast) > 40	140	Dunite < 40
119	Oceanic Basalt (Gast) > 40	141	Andesite > 40
120	Oceanic Basalt (Gast) < 40	142	Andesite Chips
121	Knippa Basalt < 40 Mesh	143	Andesite > 40
122	Knippa Basalt < 40 Mesh	144	Dunite > 40
123	Knippa Basalt < 40 Mesh	145	Basalt 415 Chips
		146	Dunite > 40

NOTE: Sample tray number 103 was not used.

TABLE 3

DISTRIBUTION OF SIMULATED LUNAR ROCK SAMPLE IN SAMPLE CONTAINERS

SAMPLE CONTAINER S/N	TRANSFERRED FROM TRAY NO.	SAMPLE DESCRIPTION
001	145	Basalt 415 Chips
001	142	Andesite Chips
001	112	Dunite Chips
002	115	Basalt 415 > 40 Mesh
002	108	Basalt 415 < 40 Mesh
002	109	Basalt 415 < 40 Mesh
002	110	Basalt 415 < 40 Mesh
003	106	Andesite < 40 Mesh
003	107	Andesite < 40 Mesh
003	111	Andesite < 40 Mesh
003	141	Andesite > 40 Mesh
003	143	Andesite > 40 Mesh
004	130	Diorite 472 < 40 Mesh
004	125	Diorite 472 > 40 Mesh
004	127	Diorite 472 > 40 Mesh
004	128	Diorite 472 > 40 Mesh
005	113	Dunite < 40 Mesh
005	114	Dunite < 40 Mesh
005	136	Dunite < 40 Mesh
005	137	Dunite < 40 Mesh
005	138	Dunite < 40 Mesh
005	139	Dunite < 40 Mesh
005	140	Dunite < 40 Mesh
006	144	Dunite > 40 Mesh
006	146	Dunite > 40 Mesh
007	116	Knippa Basalt > 40 Mesh
007	121	Knippa Basalt < 40 Mesh
007	122	Knippa Basalt < 40 Mesh
007	123	Knippa Basalt < 40 Mesh
007	124	Knippa Basalt < 40 Mesh
007	131	Knippa Basalt < 40 Mesh
008	117	Knippa Basalt Chips
008	132	Knippa Basalt Chips
008	133	Knippa Basalt Chips
008	134	Knippa Basalt Chips
008	135	Knippa Basalt Chips
009	101	Diorite 472 < 40 Mesh
009	104	Diorite 472 < 40 Mesh
009	105	Diorite 472 < 40 Mesh
009	126	Diorite 472 > 40 Mesh
010	118	Oceanic Basalt
010	119	Oceanic Basalt
010	120	Oceanic Basalt

Note: Portions of samples from trays number 102, 117, 124, 129 and 140 were placed in clean glass bottles and returned with the sample containers.

10 A

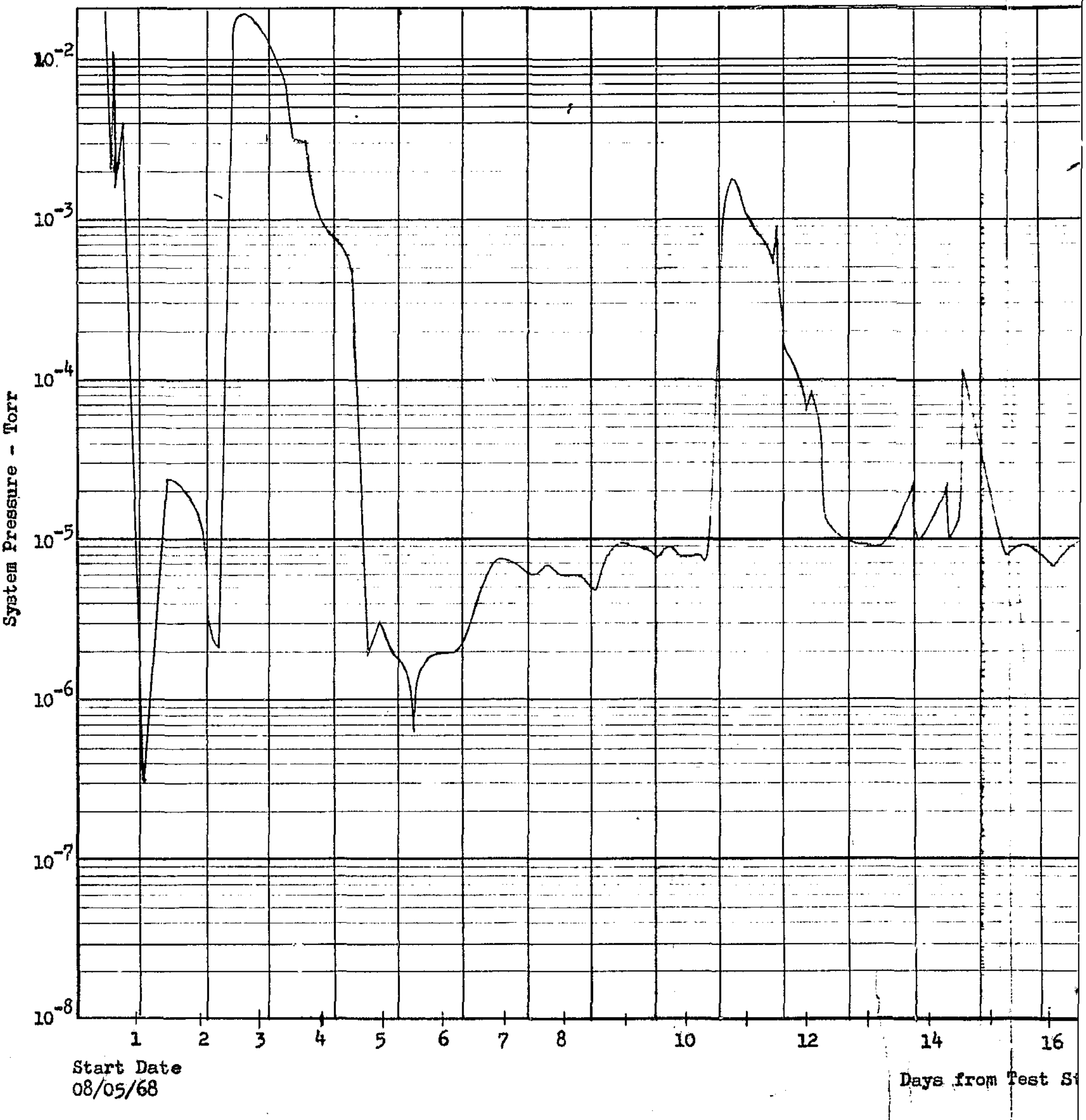
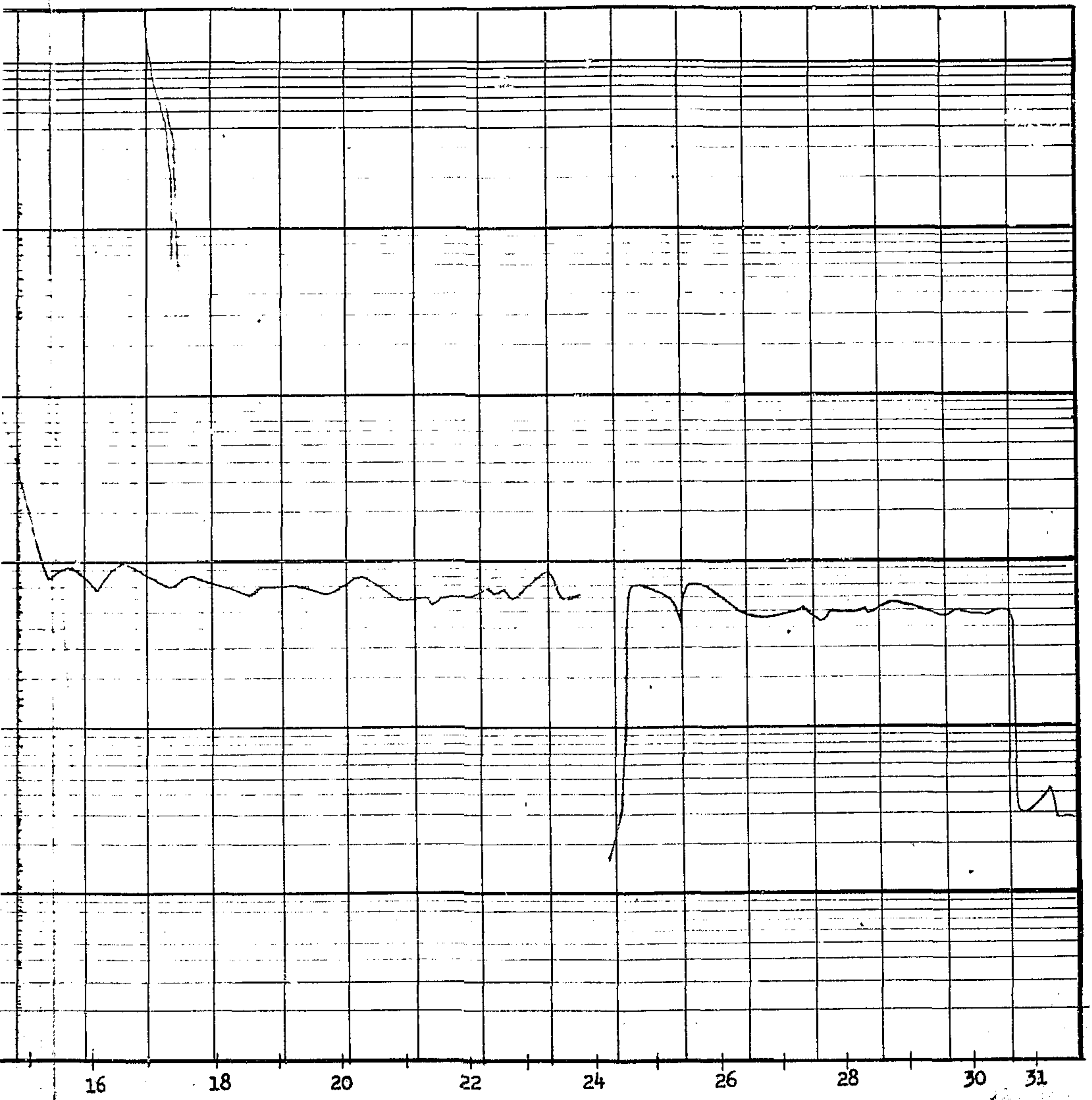
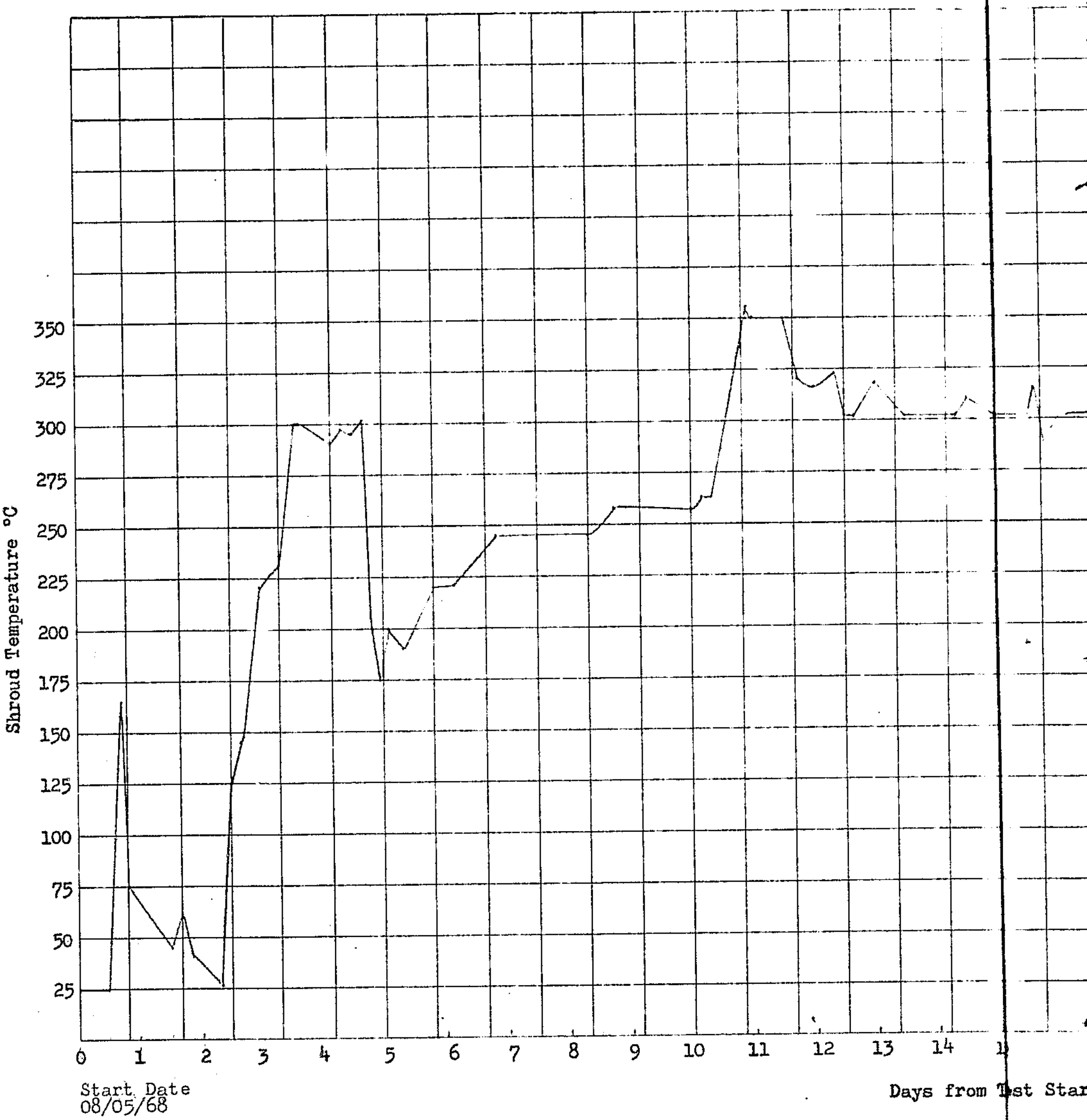

System Pressure - Torr
10-2
10-3
10-4
10-5
10-6
10-7
10-8
1
2
3
4
5
6
7
8
10
12
14
16
Start Date
08/05/68
Days from Test St

FIGURE 1: SIMULATED LUNAR ROCK BAKEOUT 10
SYSTEM PRESSURE VS TIME

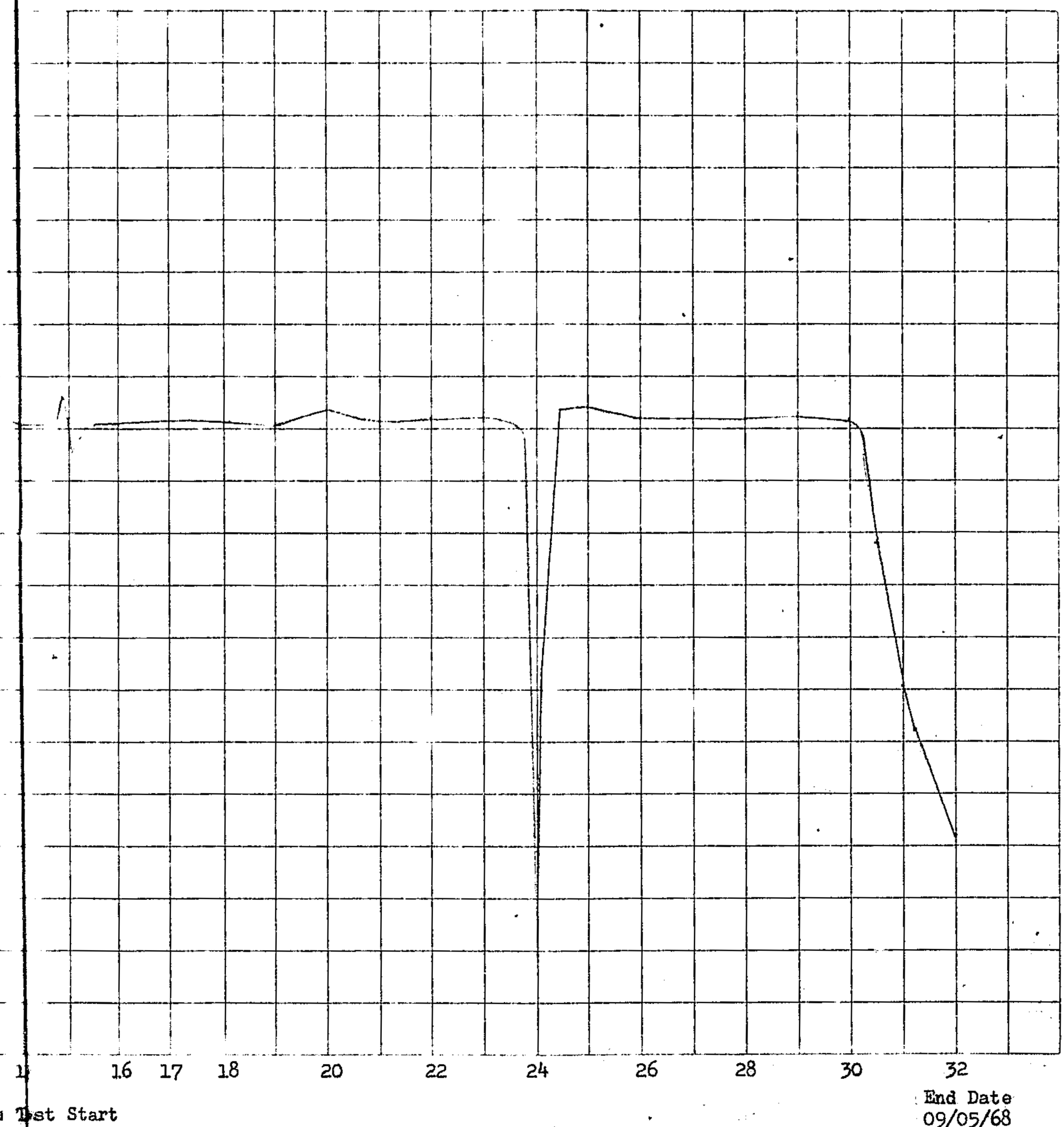


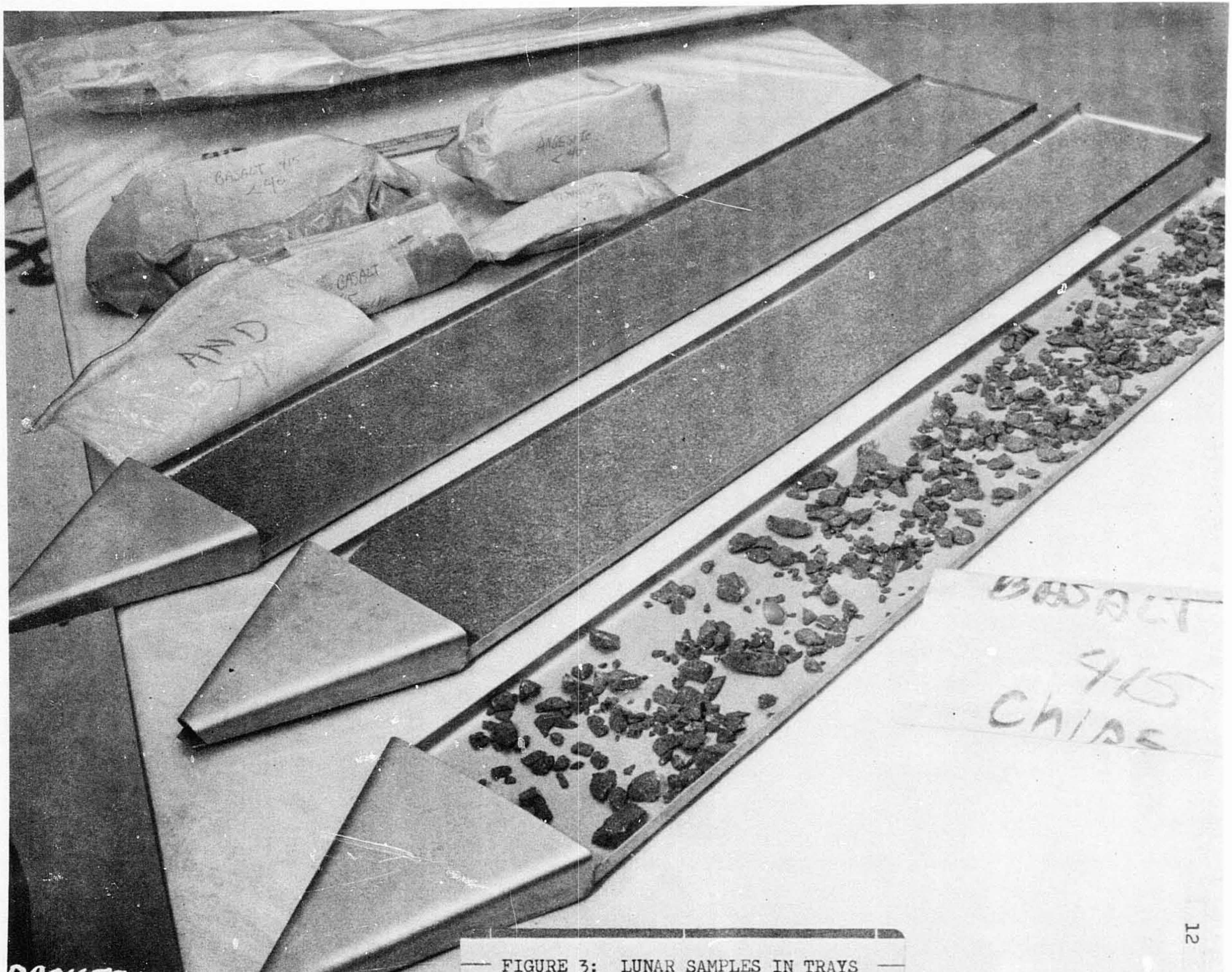
11A



Shroud Temperature °C
350
325
300
275
250
225
200
175
150
125
100
75
50
25
0
1
2
3
4
5
6
7
8
9
10
11
12
13
14
Start Date
08/05/68
Days from Test Star

FIGURE 2: SIMULATED LUNAR ROCK BAKEOUT
SHROUD TEMPERATURE VS TIME





— FIGURE 3: LUNAR SAMPLES IN TRAYS —

FIGURE 4: LUNAR SAMPLES IN TRAYS

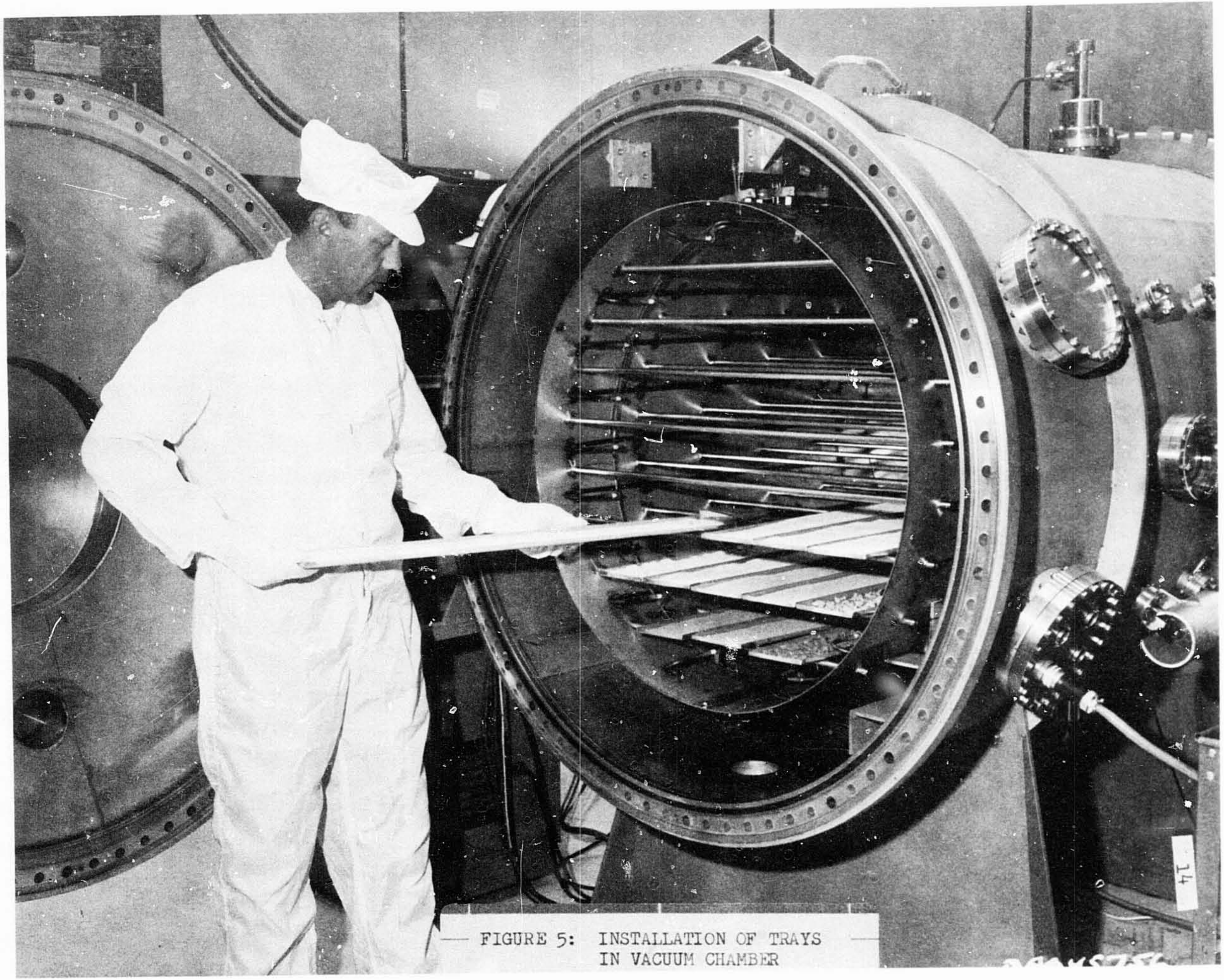


FIGURE 5: INSTALLATION OF TRAYS IN VACUUM CHAMBER

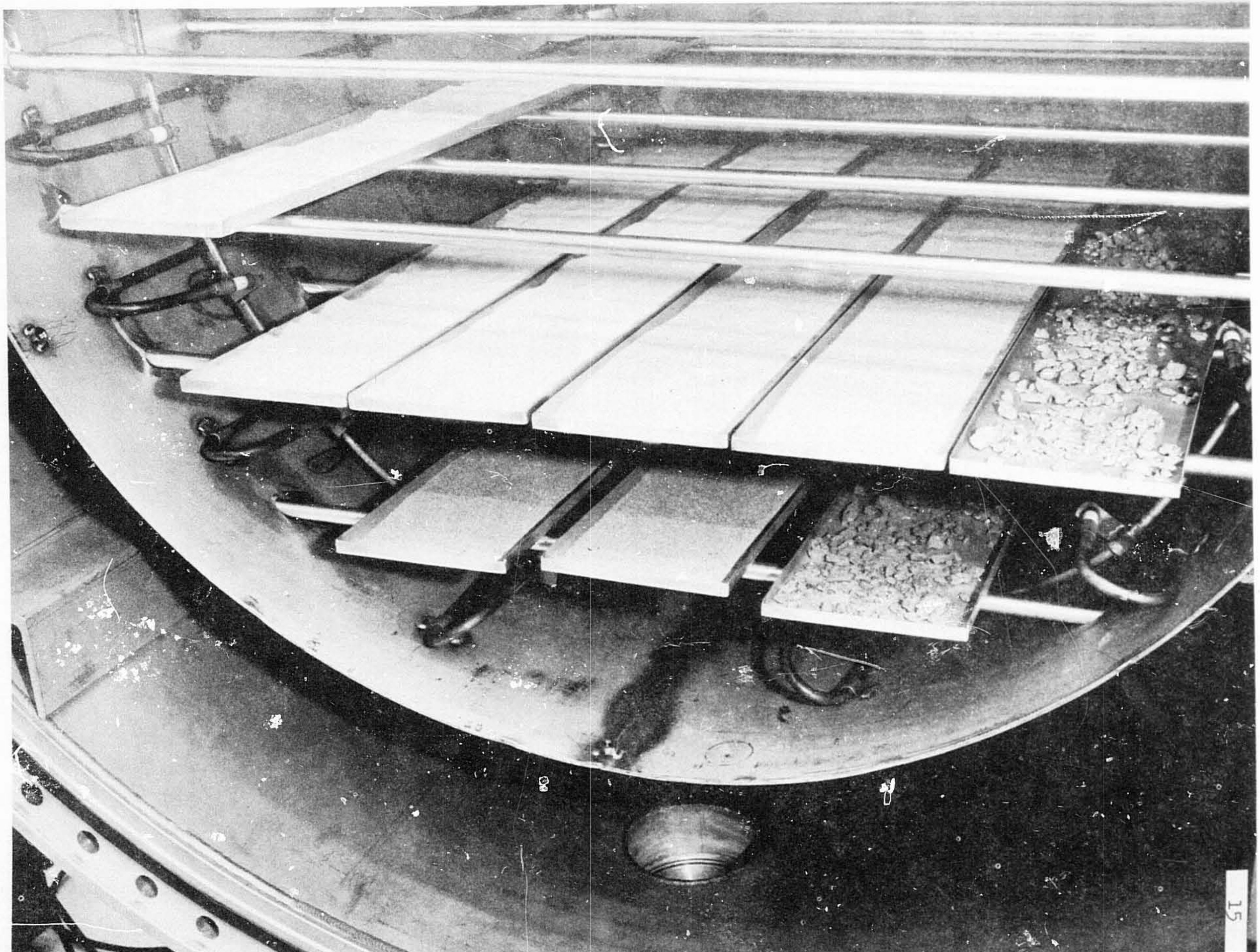


FIGURE 6: TRAYS ON OVEN SHELVES
IN VACUUM CHAMBER

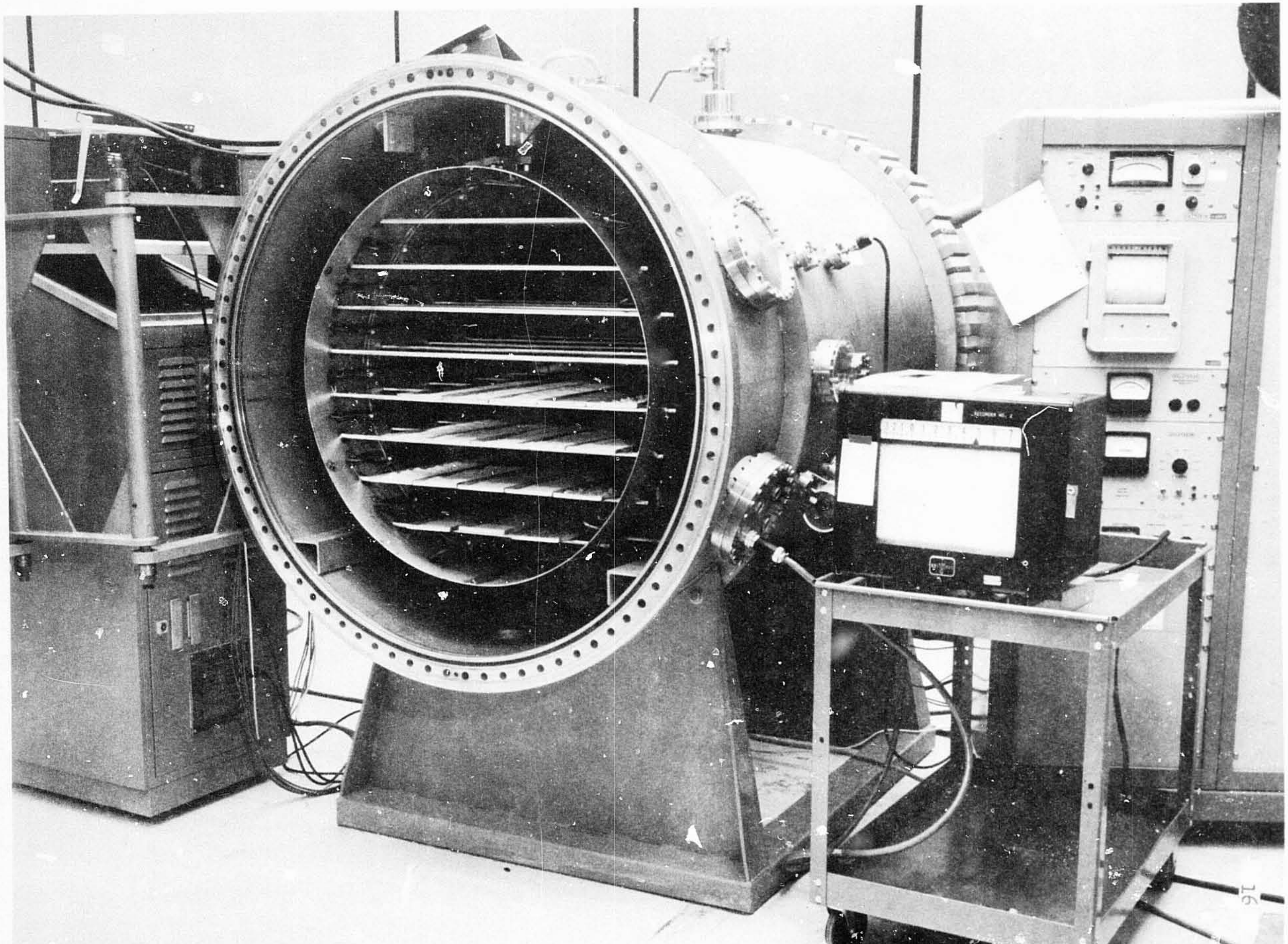


FIGURE 7: TEST CHAMBER WITH SAMPLE TRAYS INSTALLED

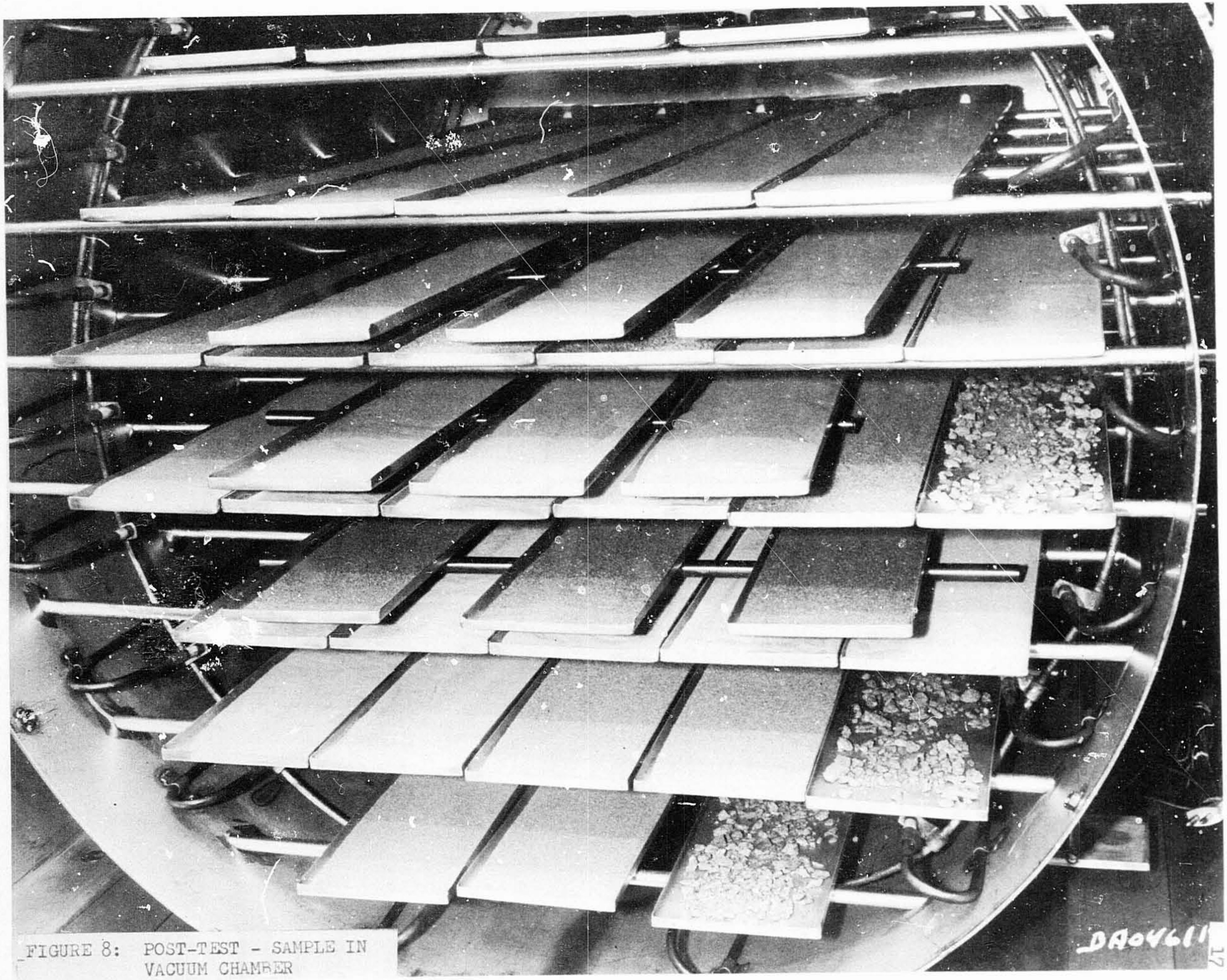


FIGURE 8: POST-TEST - SAMPLE IN VACUUM CHAMBER

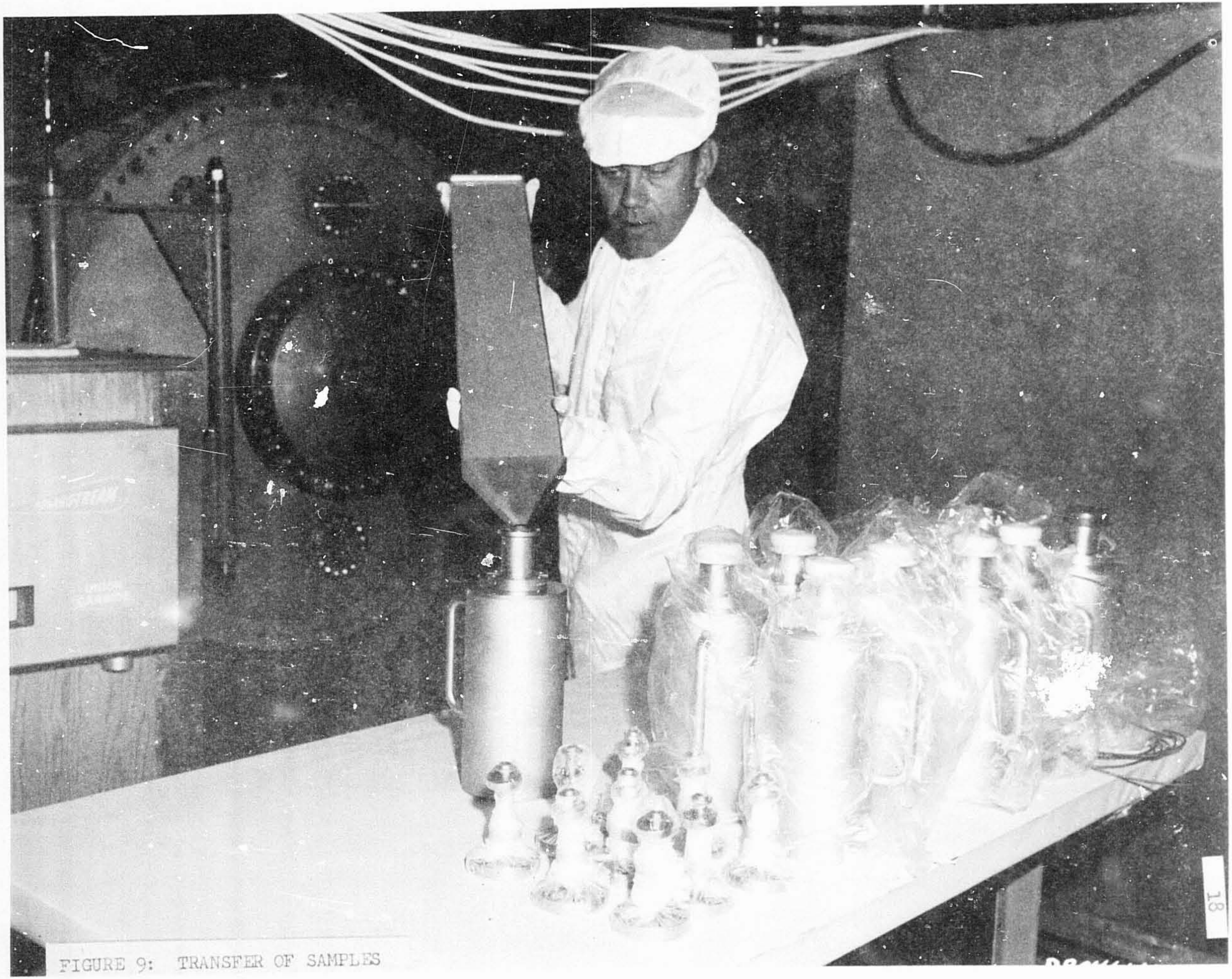


FIGURE 9: TRANSFER OF SAMPLES

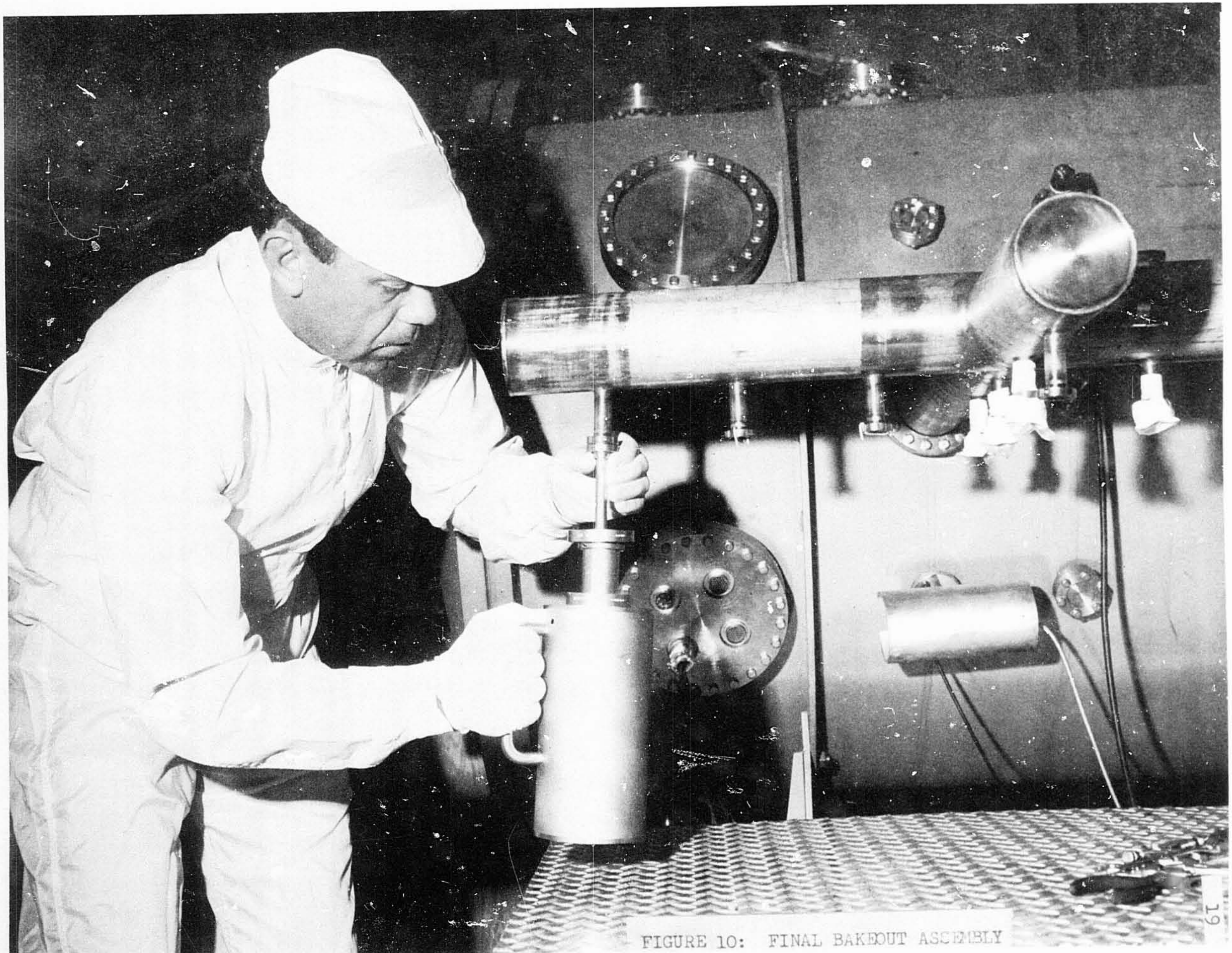


FIGURE 10: FINAL BAKEOUT ASSEMBLY