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VACUUM COMPATIBILITY OF ENGINEERING MATERIALS (LIQUIDS AND SEMI-SOLIDS)

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VACUUM COMPATIBILITY OF ENGINEERING MATERIALS

(LIQUIDS AND SEMI-SOLIDS)

By J. G. Austin and J. B. Gayle

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Huntsville, Alabama

ABSTRACT

The rates of evaporation of 20 liquid and semi-solid materials in a vacuum environment and the effects of temperature on the rates of evaporation of 11 of the 20 materials were determined experimentally. Test conditions were from room temperature to 154°C at pressures of 10^{-5} torr or less. The results are presented in 20 figures and 1 table.

Author

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RESEARCH AND DEVELOPMENT OPERATIONS
PROPULSION AND VEHICLE ENGINEERING LABORATORY

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SUMMARY

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The rates of evaporation of 20 liquid and semi-solid materials in a vacuum environment were determined by both continuous and before-and-after weighings. The effect of temperature on rate of evaporation was determined for 11 of the 20 materials. Tests were made at one to three temperatures, ranging from room temperature to 154 C. The weight loss varied from 0.6 milligram (twice the limit of detection) to 100 percent. Testing times ranged from 1 to 500 hours. Comparisons of the evaporation rates of the various liquids and semi-solids should be attempted cautiously.

Author

INTRODUCTION

Vacuum compatibility is a major factor influencing the selection of materials for space applications. Many materials lose weight when exposed to reduced pressure. Both the extent of this loss and any corresponding changes in material properties depend on a number of factors, including the temperature and radiation levels to which the material is subjected and the chemical, physical, and mechanical characteristics of the material. For this reason, specifications for acceptance or rejection of materials for space applications should include the environmental conditions associated with the intended application.

Weight loss is an ambiguous criterion for the selection of materials for space environments. For example, a grease may evaporate to a considerable extent but continue to function satisfactorily. However, the vaporized portion may condense on cooler adjacent surfaces and, thereby, cause malfunction of the system, as has occurred in at least one known incident with electronic circuitry. It should be noted that materials which are susceptible to oxidation may perform more satisfactorily in vacuum than in air at normal atmospheric pressure. In any event, knowledge obtained by visual examination of the samples before and after testing can be used for preliminary screening, thus reducing the number of materials which must be subjected to altitude simulation tests in hardware configurations.

A program for testing the compatibility of materials in vacuum was initiated in the Materials Division in 1959. The equipment used for this investigation, the test procedures, and the results obtained for a number of materials have been discussed in previous reports (Ref. 1, 2, 3). This report presents additional results obtained for a variety of liquid and semi-solid materials.

EXPERIMENTAL PROCEDURE

Three vacuum systems, designated 4A, 4B, and 4D, were used. All systems used four-inch diffusion pumps and differed primarily in the degree of automatic operation and type of balance. Both weighing and pumping operations used in the 4A system were manual. The 4D system (FIG 1) had an automatic balance (FIG 2) and manually operated pumping equipment. Both weighing and pumping operations used in the 4B system were automatic. The systems were equipped with ceramic ovens (FIG 3) for elevated temperature experimentation.

All weight loss determinations were made with the samples contained in Knudsen cells (FIG 4 and 5). Each sample was weighed into the lower section of the cell; then, the top section, which contained a 0.3302-centimeter diameter orifice, was secured in place by a rolled and crimped mechanical seal of the mated flanges. The initial sample weight was determined by difference.

Two types of balances were used for each run: an ordinary analytical balance for before-and-after weighing and a Cahn magnetic electrobalance (FIG 2) for weighing in vacuum. In the 4B and 4D systems, the automatic balances provided a continuous recording of the weight; however, in the 4A system, the balance was operated manually. The zero settings and calibrations of the balances were checked between runs. By placing thermocouples at various locations in the bell jar, it was determined that heating was confined largely to the ovens; consequently, thermal effects on the balances were small.

After a sample was placed in the vacuum chamber, a pressure of less than 10^{-4} torr was achieved in approximately one hour; then, when applicable, the specimen was heated to the selected test temperature in 15-30 minutes. In all cases, the ultimate pressure was less than 10^{-5} torr. Temperatures were governed by maximum anticipated service temperatures, by the approximate upper temperature limit (for use in air) specified by the manufacturer, or by arbitrary selection.

In most instances, runs were terminated when the sample attained constant weight or exhibited less than 0.1 milligram weight change in any eight-hour period. After the specimens cooled to room temperature, they were removed from vacuum and weighed on the analytical balance.

This procedure has been described in somewhat greater detail in a previous report (Ref. 1). Experience has indicated that variations due to weighing errors and other causes normally do not exceed 0.3 milligram. The corresponding percentage errors based on sample weights used in this investigation ranged from 0.9 to 0.01. Therefore, changes less than this value were not considered significant.

DISCUSSION

Since the demarcation between liquid and semi-solid materials was arbitrary, no attempt has been made to classify the results for the various materials according to the physical state of the samples.

Tests were made on 20 liquid or semi-solid materials which were obtained from commercial manufacturers. Samples were contained within Knudsen cells with 0.3302-centimeter diameter orifices. Sample weights varied from 50 to 450 milligrams with most samples grouped in the 90 to 100-milligram range. Tests were made at one to three temperatures, ranging from room temperature to 154°C. Generally, most samples were tested at room temperature and 100°C. The results are summarized in Table I and are presented graphically in FIG 6 through 25. It should be noted that with the wide range of initial sample weights the percent weight remaining was plotted versus the weight adjusted time to relegate the results for each sample to an initial weight basis of 100 milligrams. For the range of sample weights used in this study, errors introduced by this treatment are considered to be negligible. However, a theoretical analysis of Knudsen cell errors is in progress and should indicate the limitations on the use of this procedure to extrapolate sample results for applications involving large quantities of materials.

The effect of temperature on the rate and extent of weight loss for each material is shown in FIG 6 through 25. These relationships are distinctly nonlinear; therefore, they are indicative of the complex nature of the processes responsible for the weight loss.

The materials tested ranged from pure liquids to fiber filled grease; therefore, because the results are dependent not only on the principal component but also for any filler components, general conclusions regarding the evaporation of liquids and semi-solids should be made with caution.

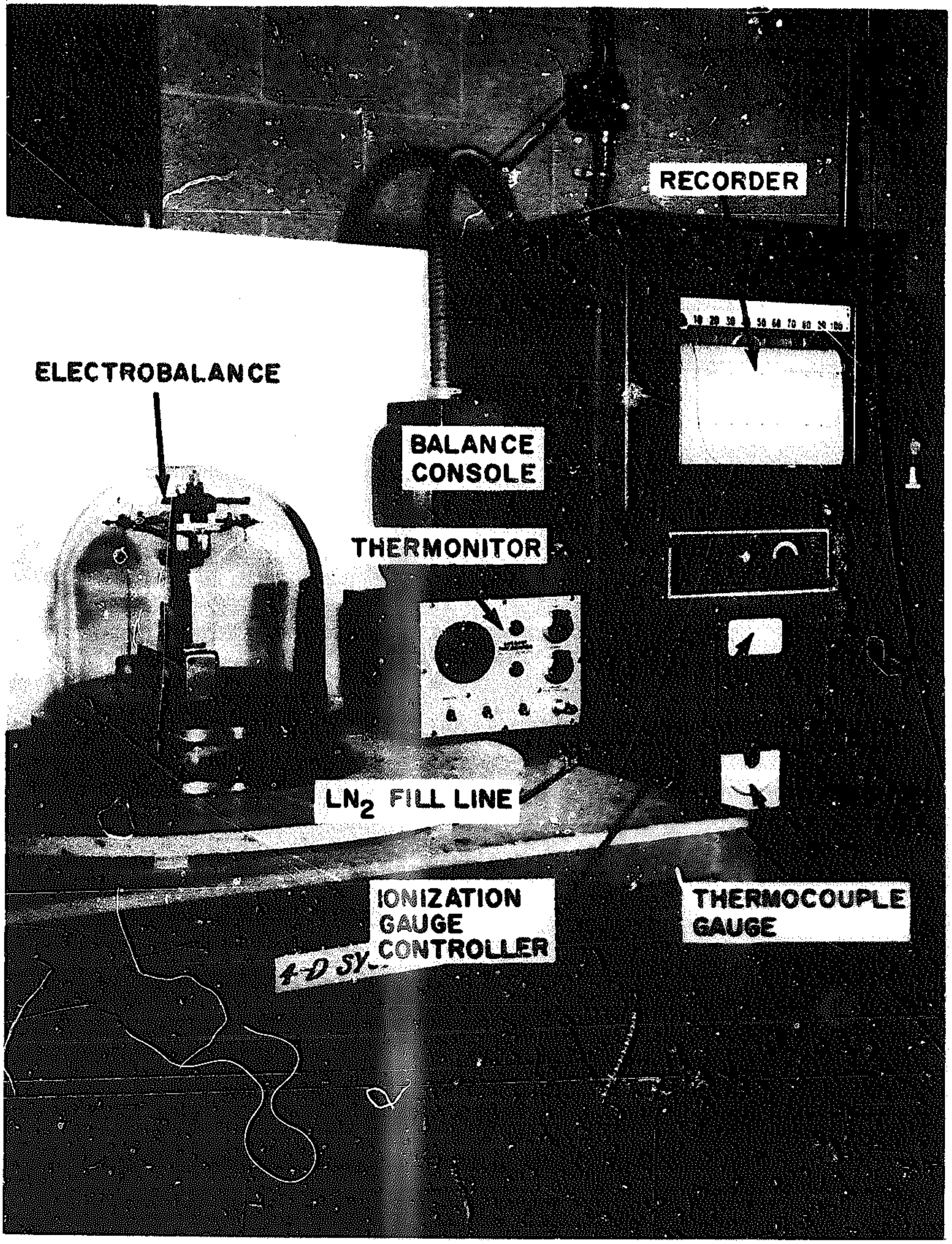

FIGURE 1. 4D VACUUM SYSTEM

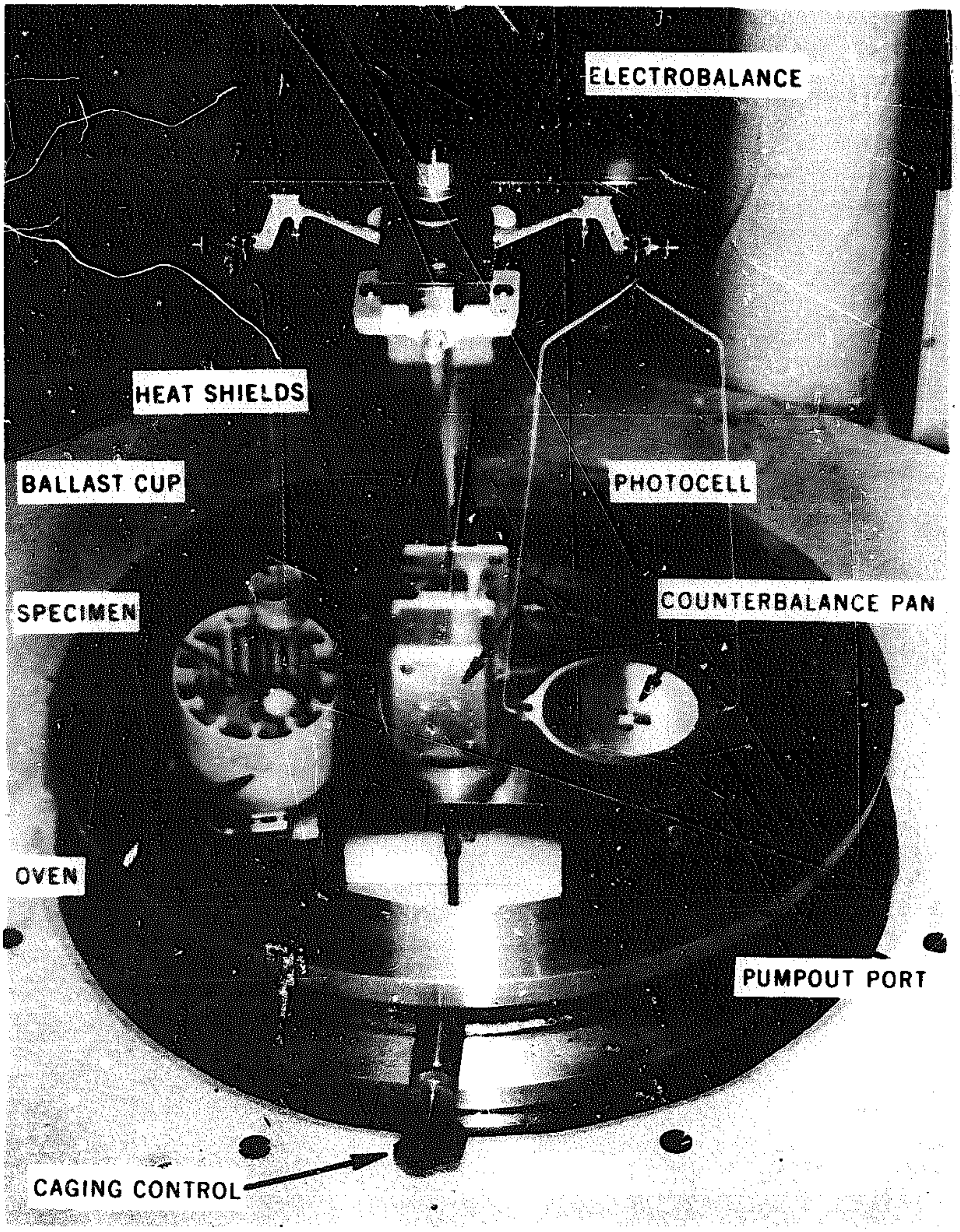

FIGURE 2. CONTINUOUS RECORDING AUTOMATIC ELECTROBALANCE

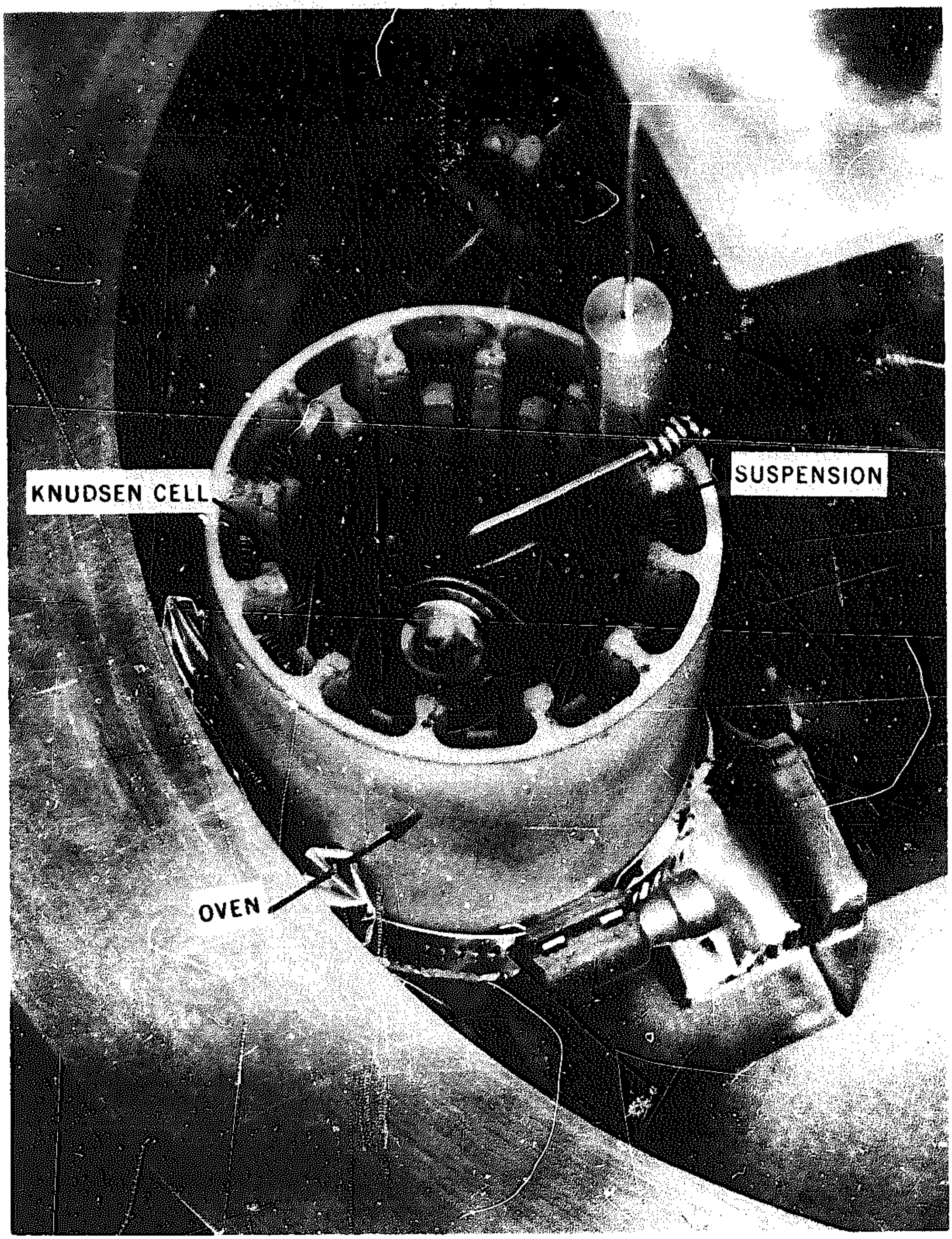

FIGURE 3. CERAMIC OVEN FOR HEATING TEST SPECIMEN IN VACUUM

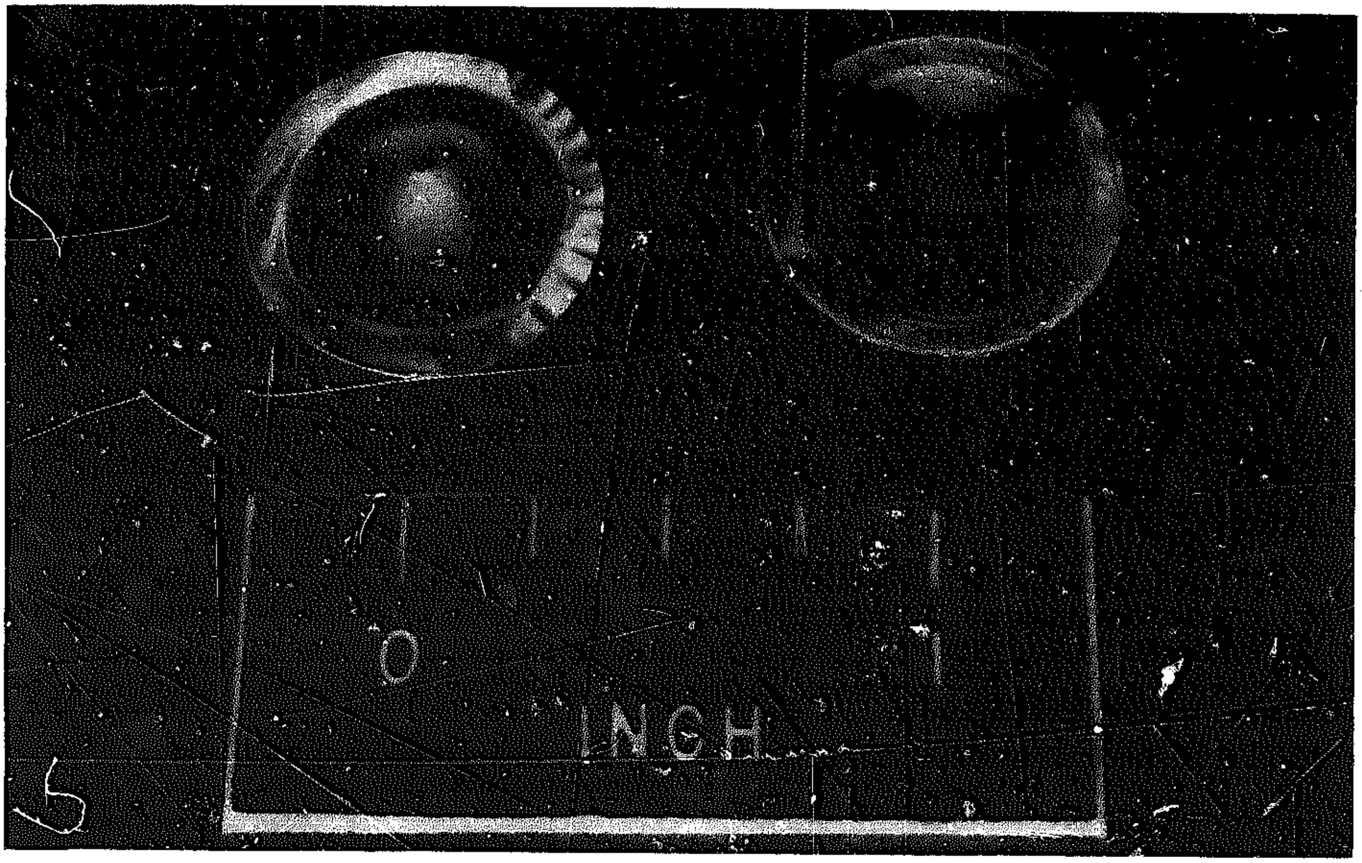

FIGURE 4. KNUDSEN CELL - TOP AND BOTTOM SECTIONS

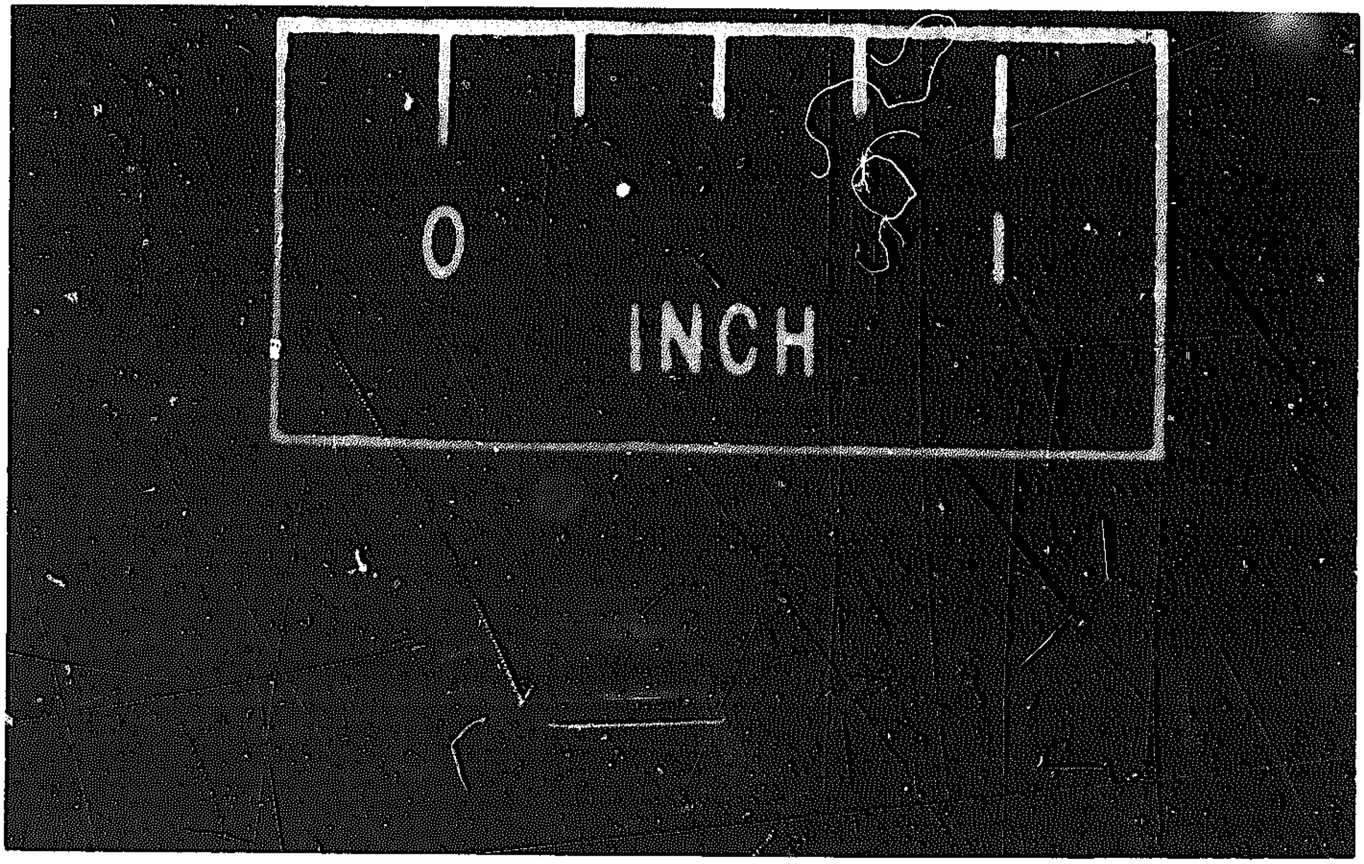

FIGURE 5. ASSEMBLED

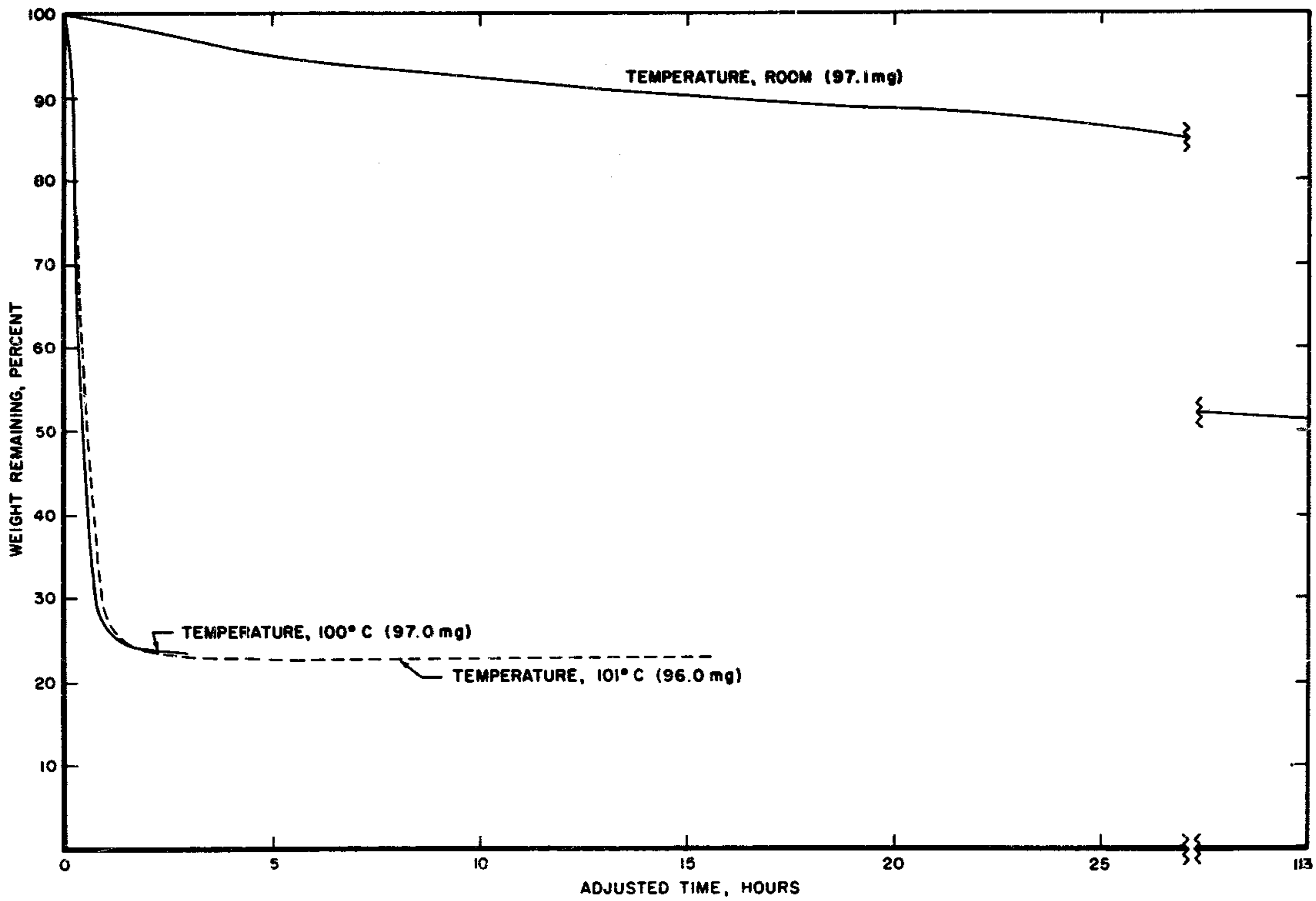

FIGURE 6. TIME-WEIGHT HISTORY FOR ACHESON EC-1730 DURING EXPOSURE TO VACUUM

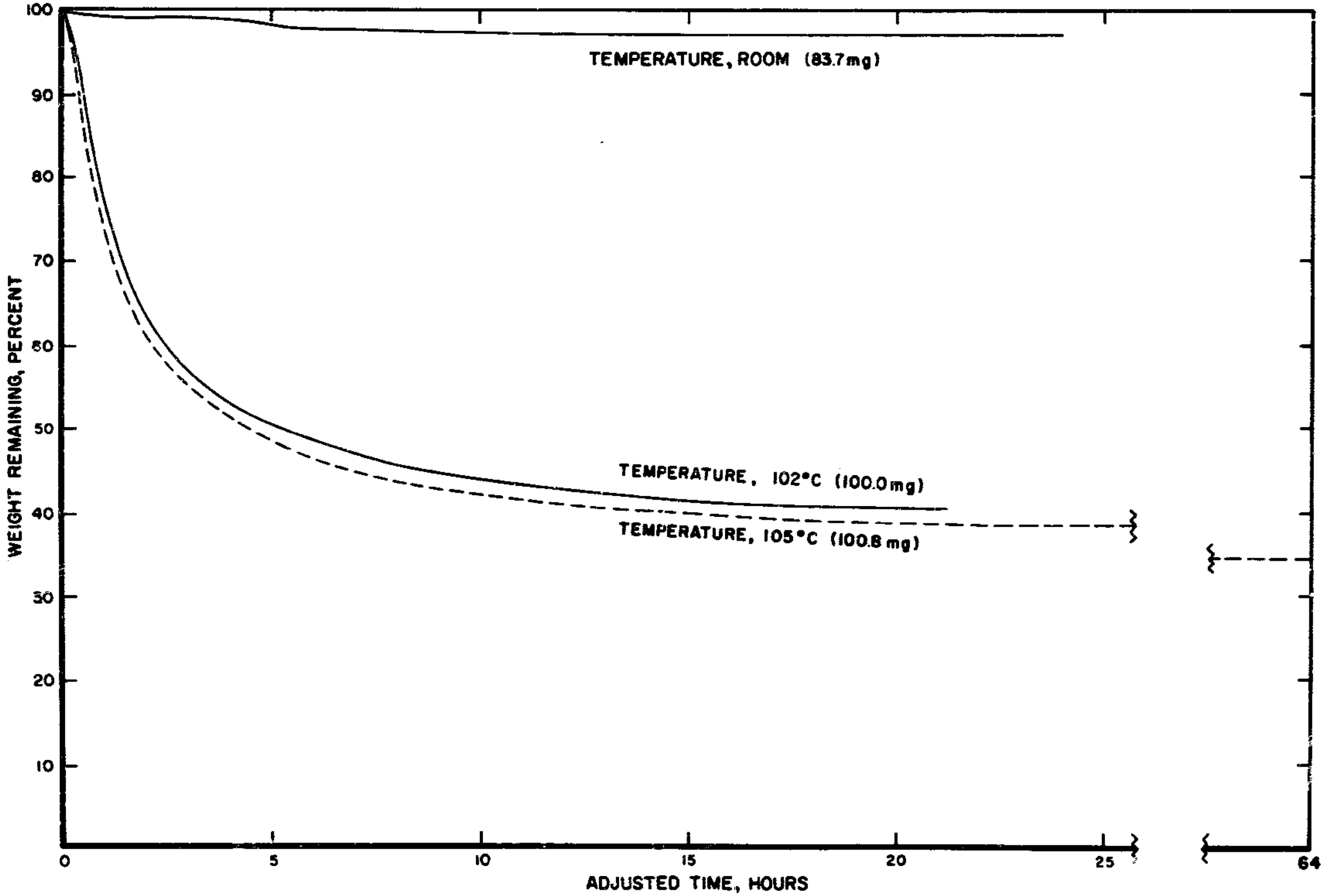

FIGURE 7. TIME-WEIGHT HISTORY FOR ANDOK C DURING EXPOSURE TO VACUUM

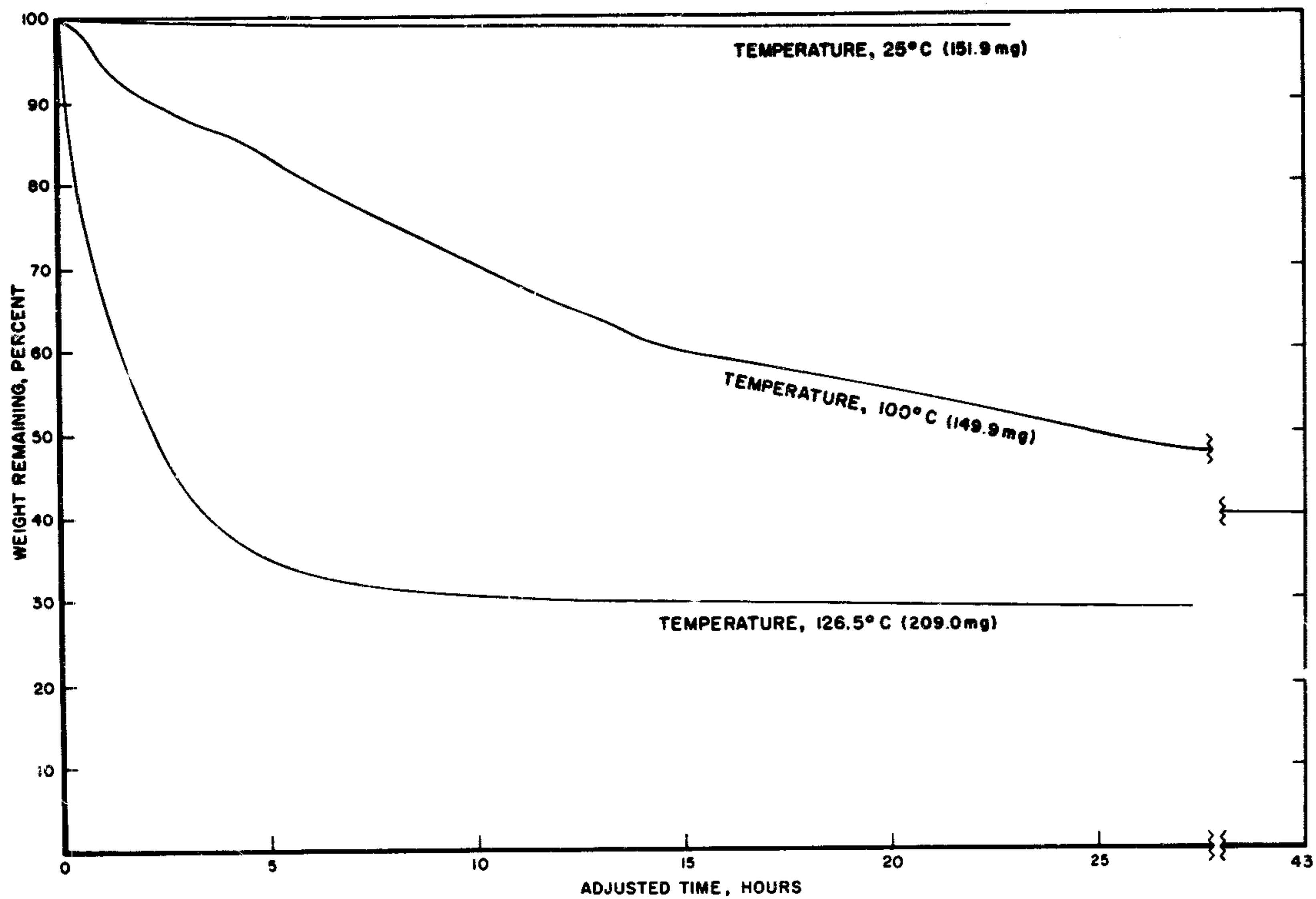

FIGURE 8. TIME-WEIGHT HISTORY FOR CALIFORNIA RESEARCH CORPORATION GREASE NO. 159 DURING EXPOSURE TO VACUUM

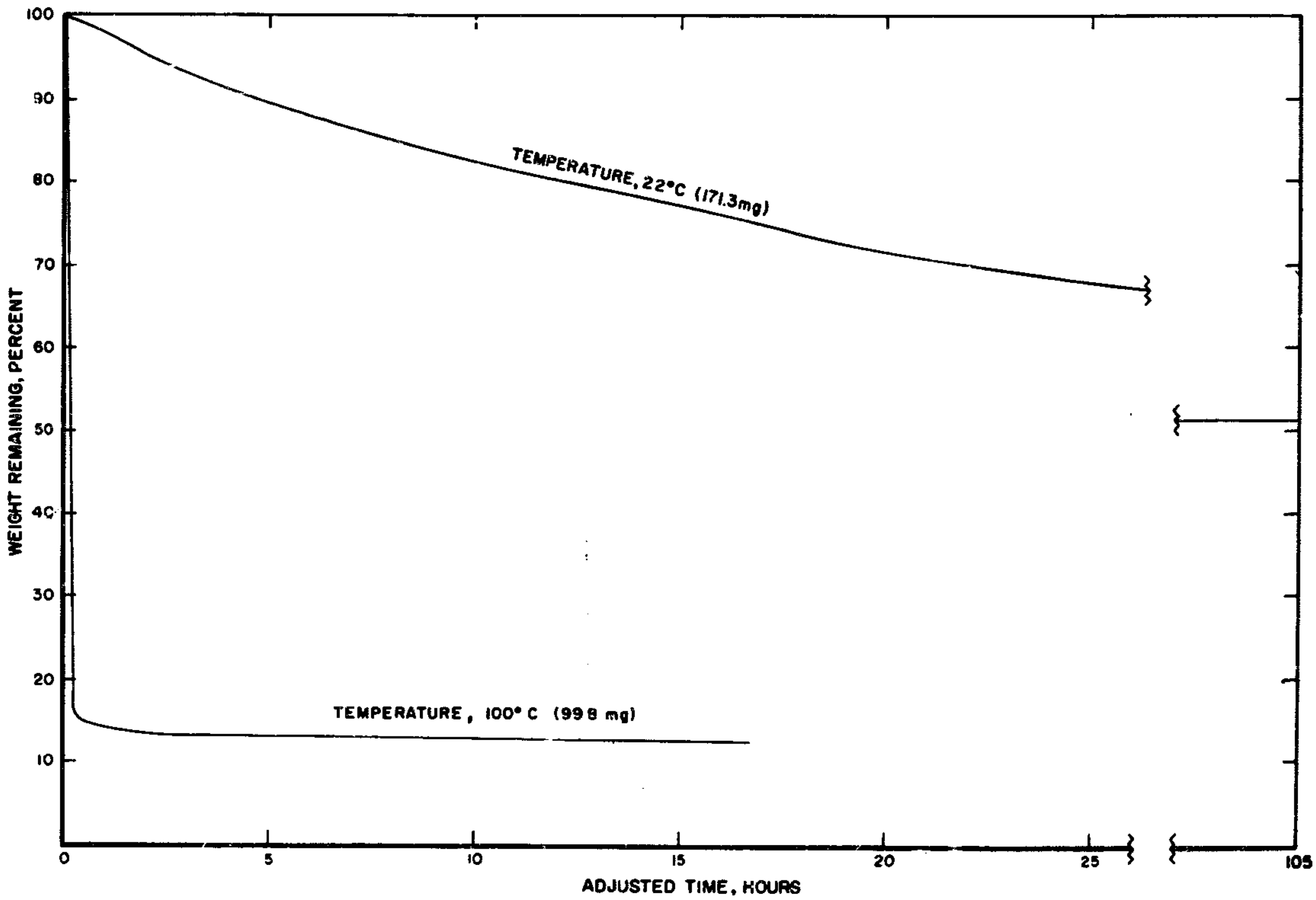

FIGURE 9. TIME-WEIGHT HISTORY FOR CALIFORNIA RESEARCH CORPORATION GREASE NO. 4669-18-1 DURING EXPOSURE TO VACUUM

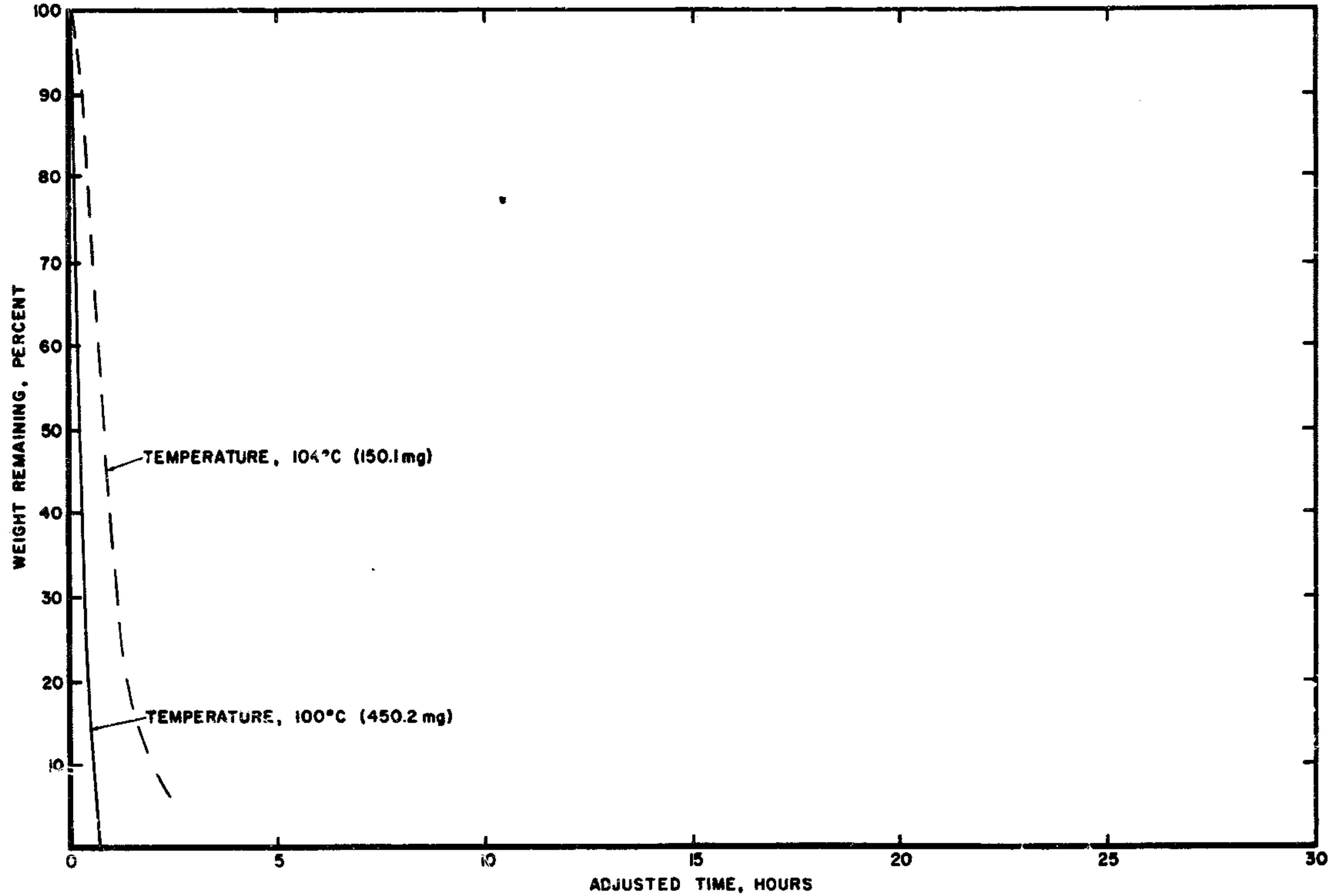

FIGURE 10. TIME-WEIGHT HISTORY FOR DOW-CORNING 704 DURING EXPOSURE TO VACUUM

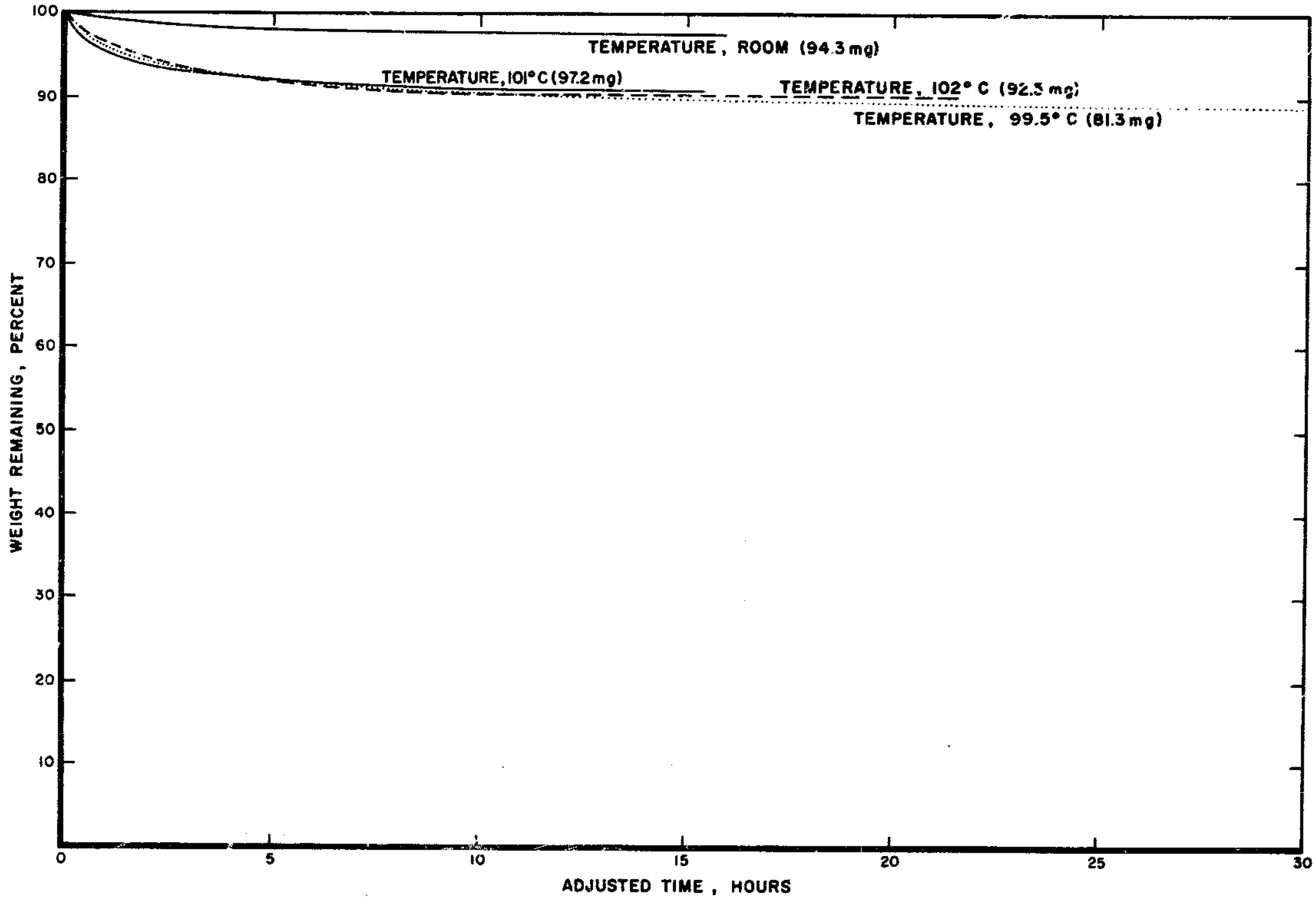

FIGURE 11. TIME-WEIGHT HISTORY FOR DOW-CORNING QC-2-0026 DURING EXPOSURE TO VACUUM

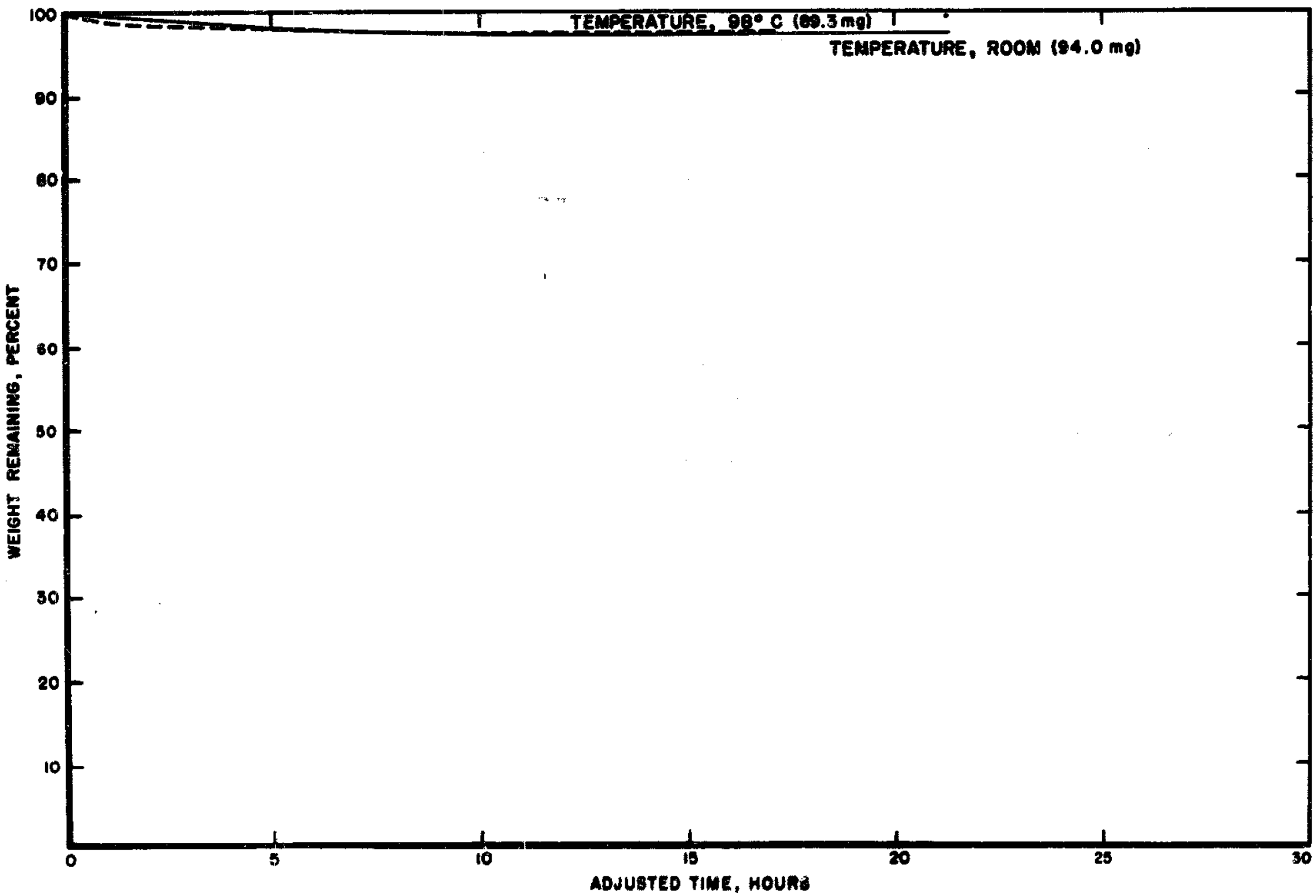

FIGURE 12. TIME-WEIGHT HISTORY FOR DOW-CORNING QC-2-0093 DURING EXPOSURE TO VACUUM

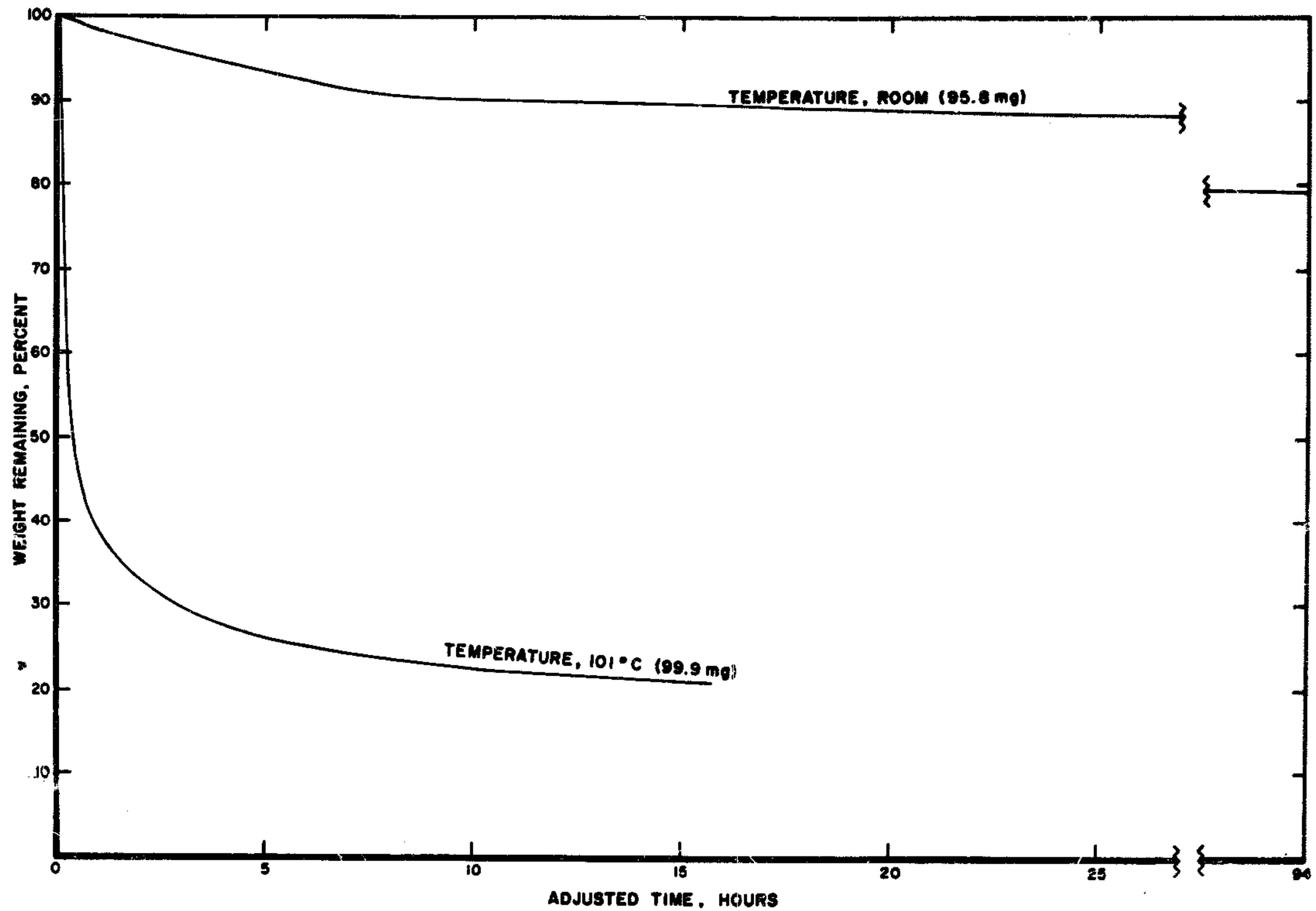

FIGURE 13. TIME-WEIGHT HISTORY FOR FLUOROLUBE LC-160 DURING EXPOSURE TO VACUUM

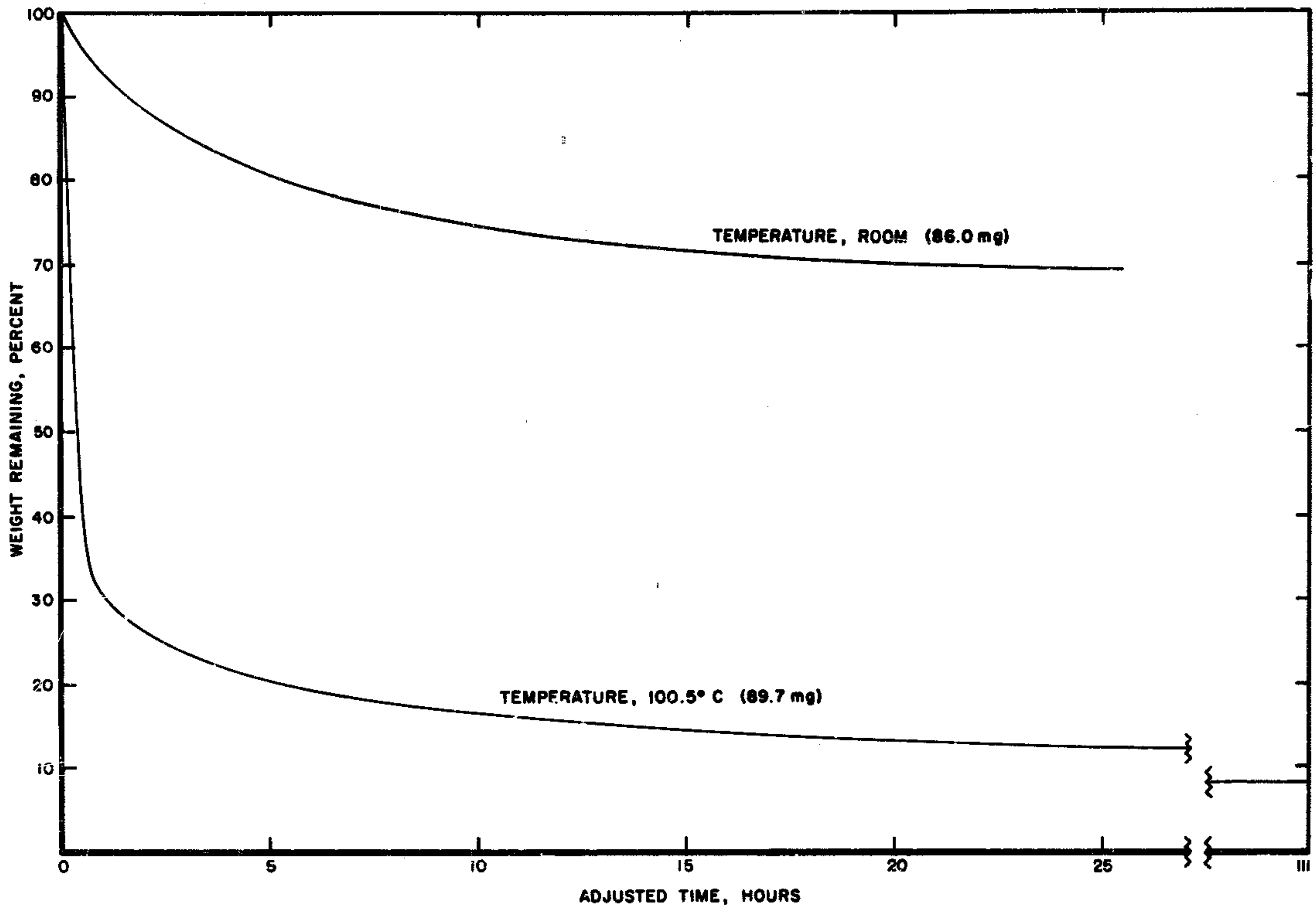

FIGURE 14. TIME-WEIGHT HISTORY FOR FLUOROLUBE T-45 DURING EXPOSURE TO VACUUM

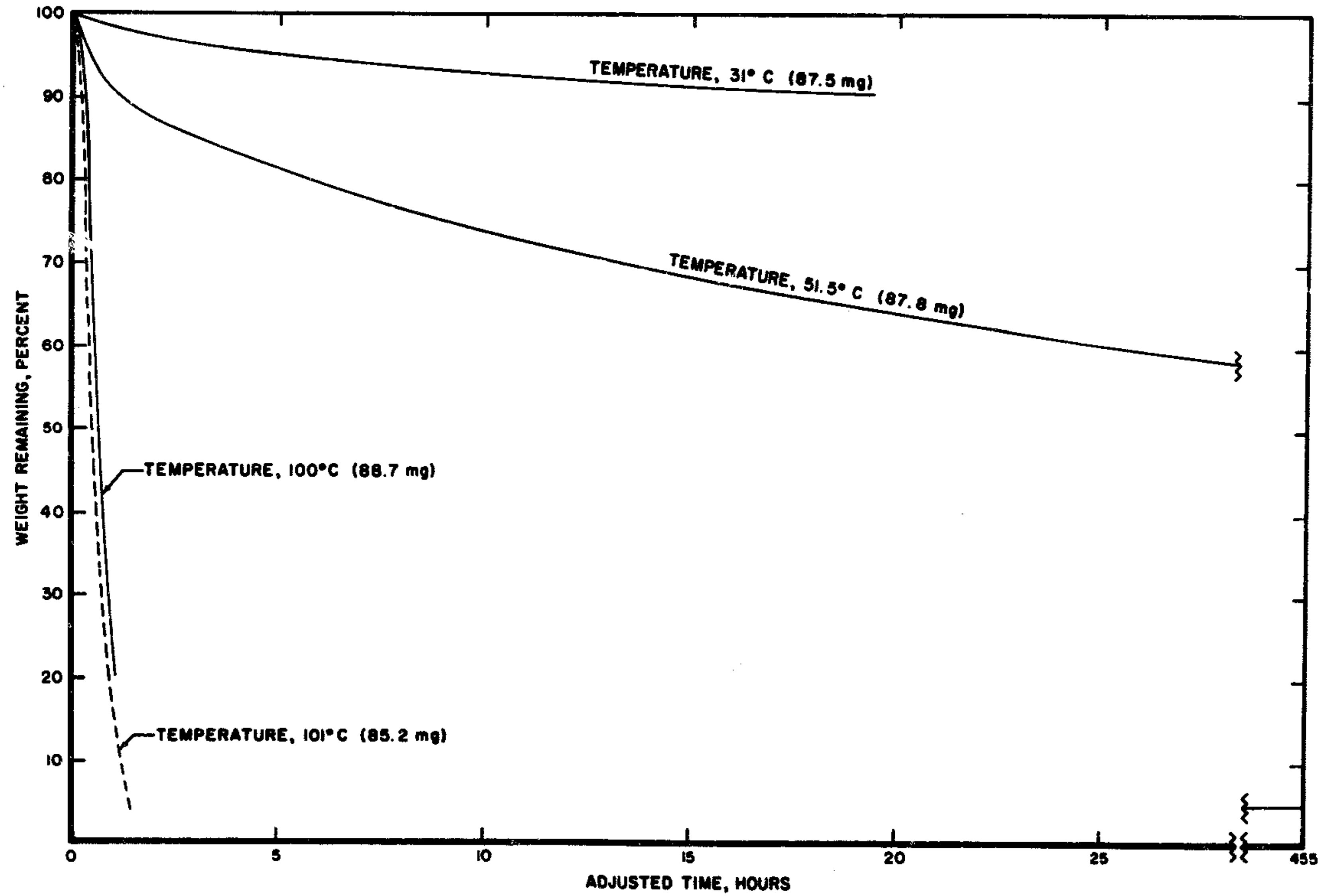

FIGURE 15. TIME-WEIGHT HISTORY FOR FLUOROLUBE T-80 DURING EXPOSURE TO VACUUM

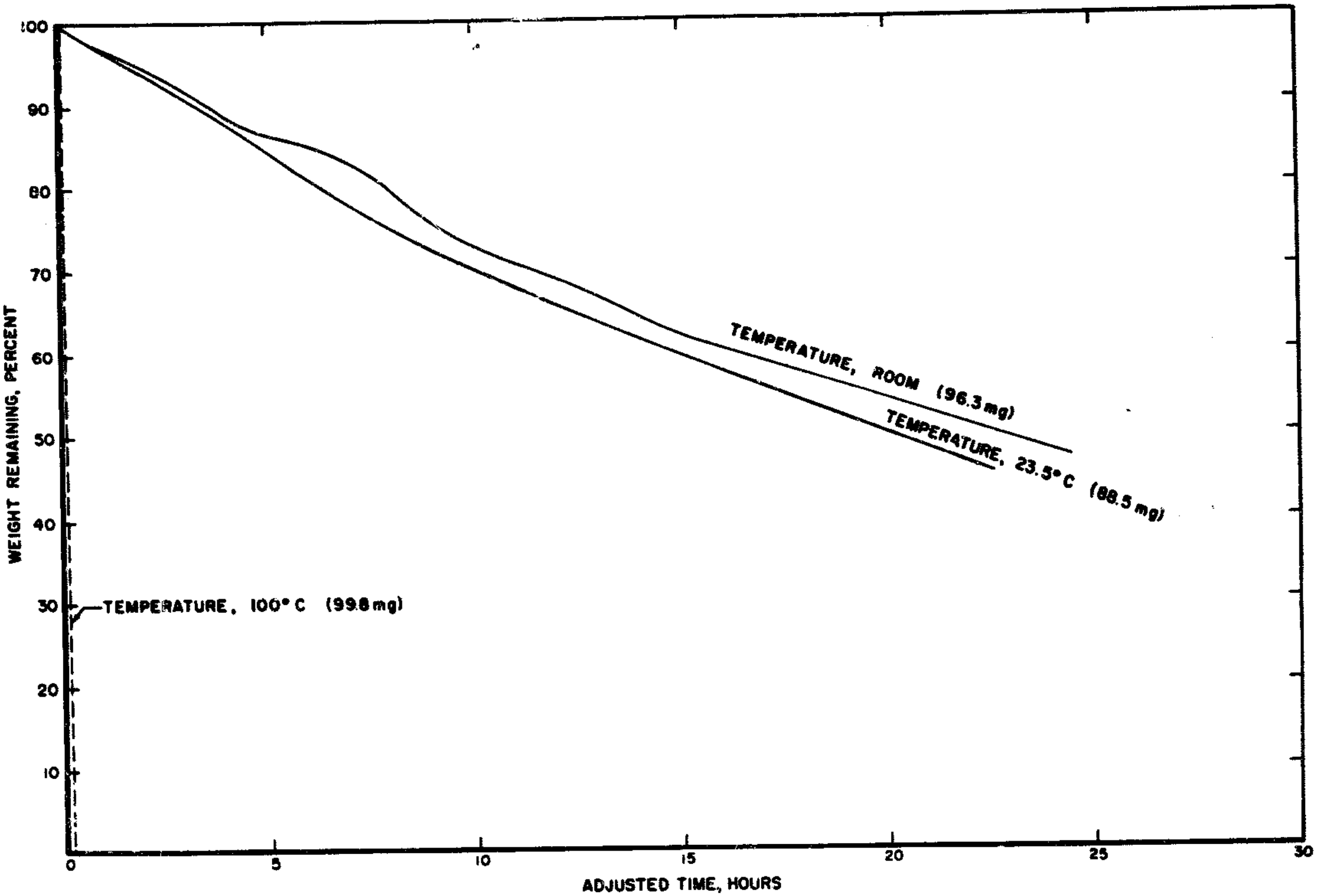

FIGURE 16. TIME-WEIGHT HISTORY FOR GLYCERINE DURING EXPOSURE TO VACUUM

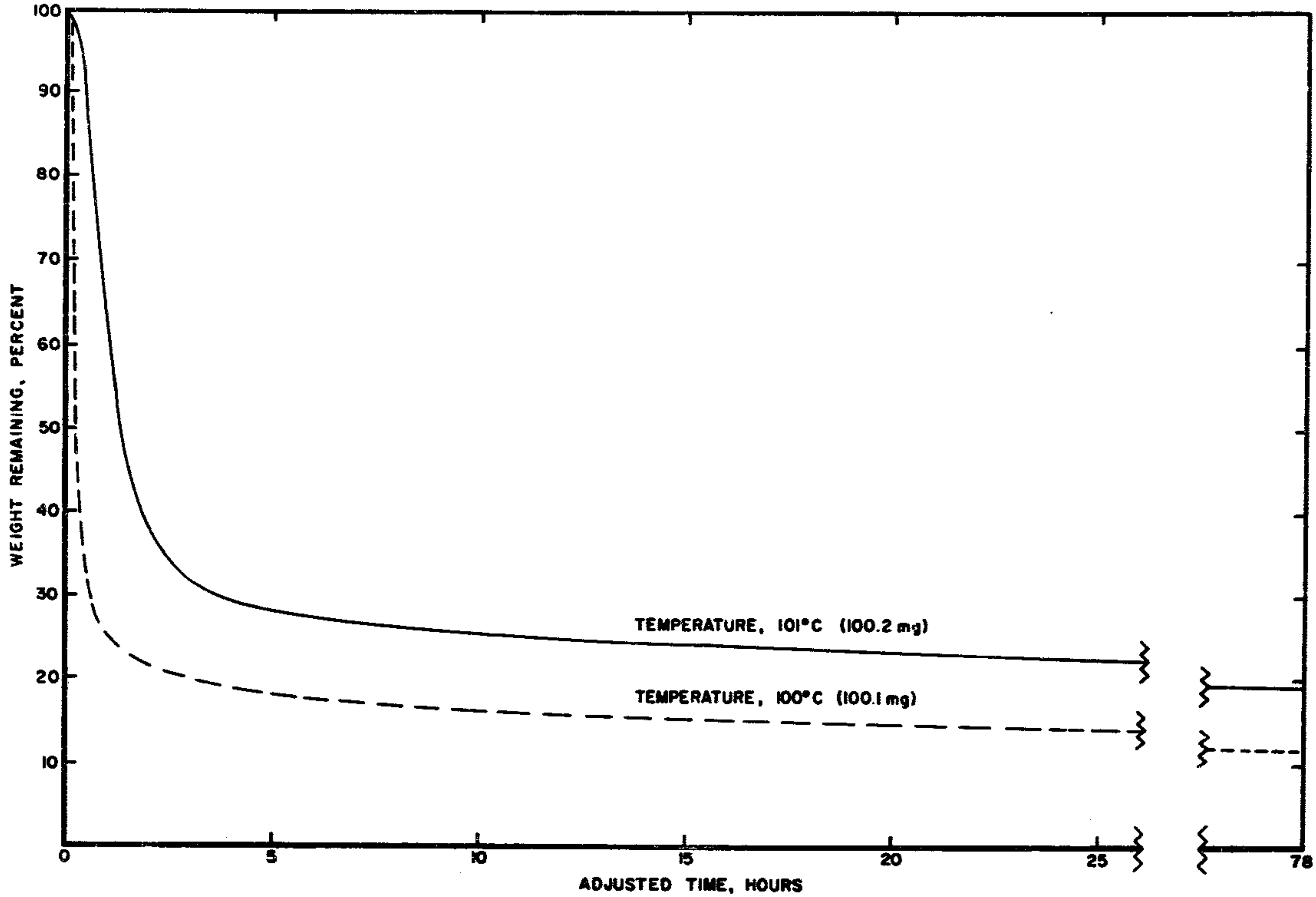

FIGURE 17. TIME-WEIGHT HISTORY FOR MIL-G-3278A DURING EXPOSURE TO VACUUM

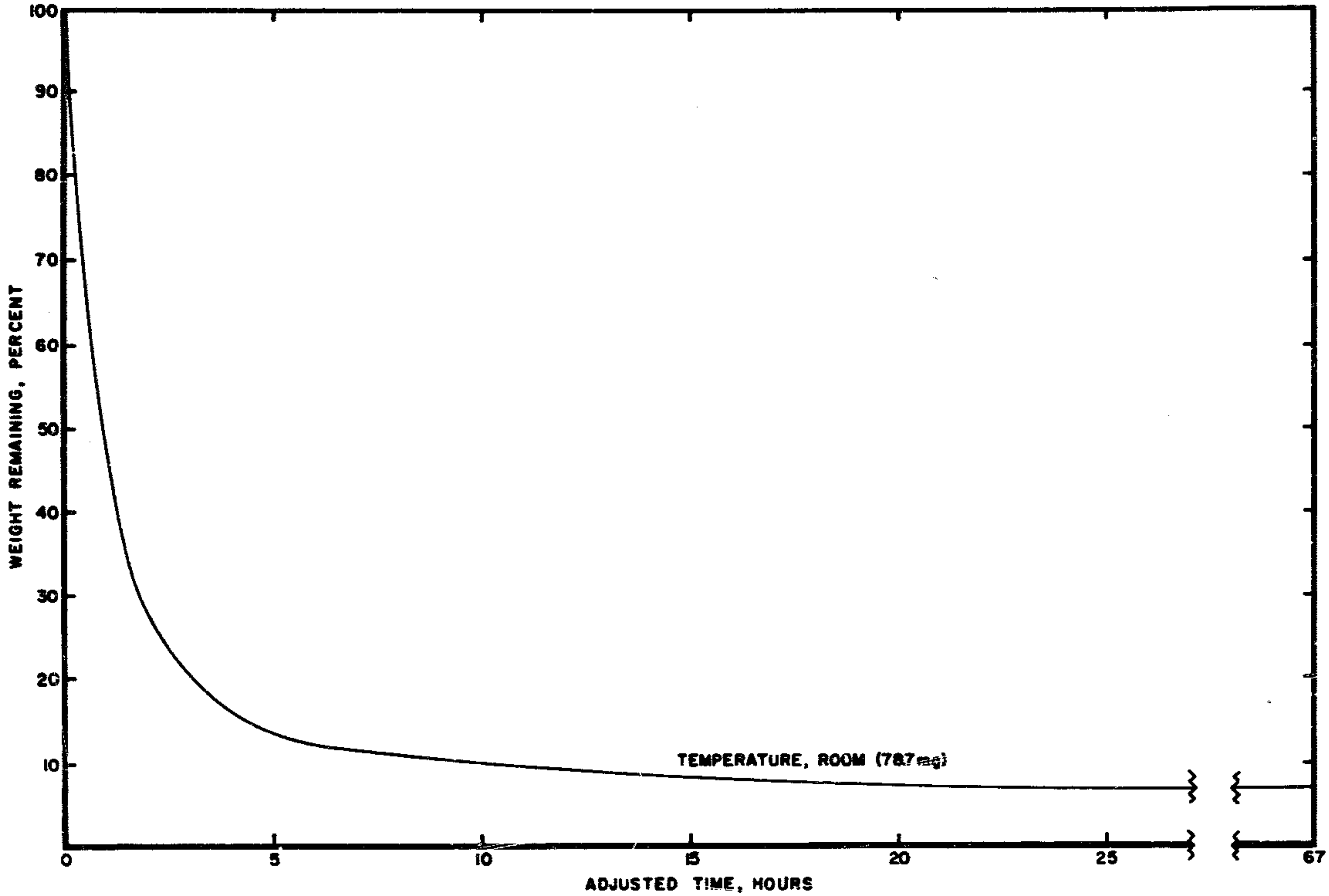

FIGURE 18. TIME-WEIGHT HISTORY FOR MIL-H-5606 DURING EXPOSURE TO VACUUM

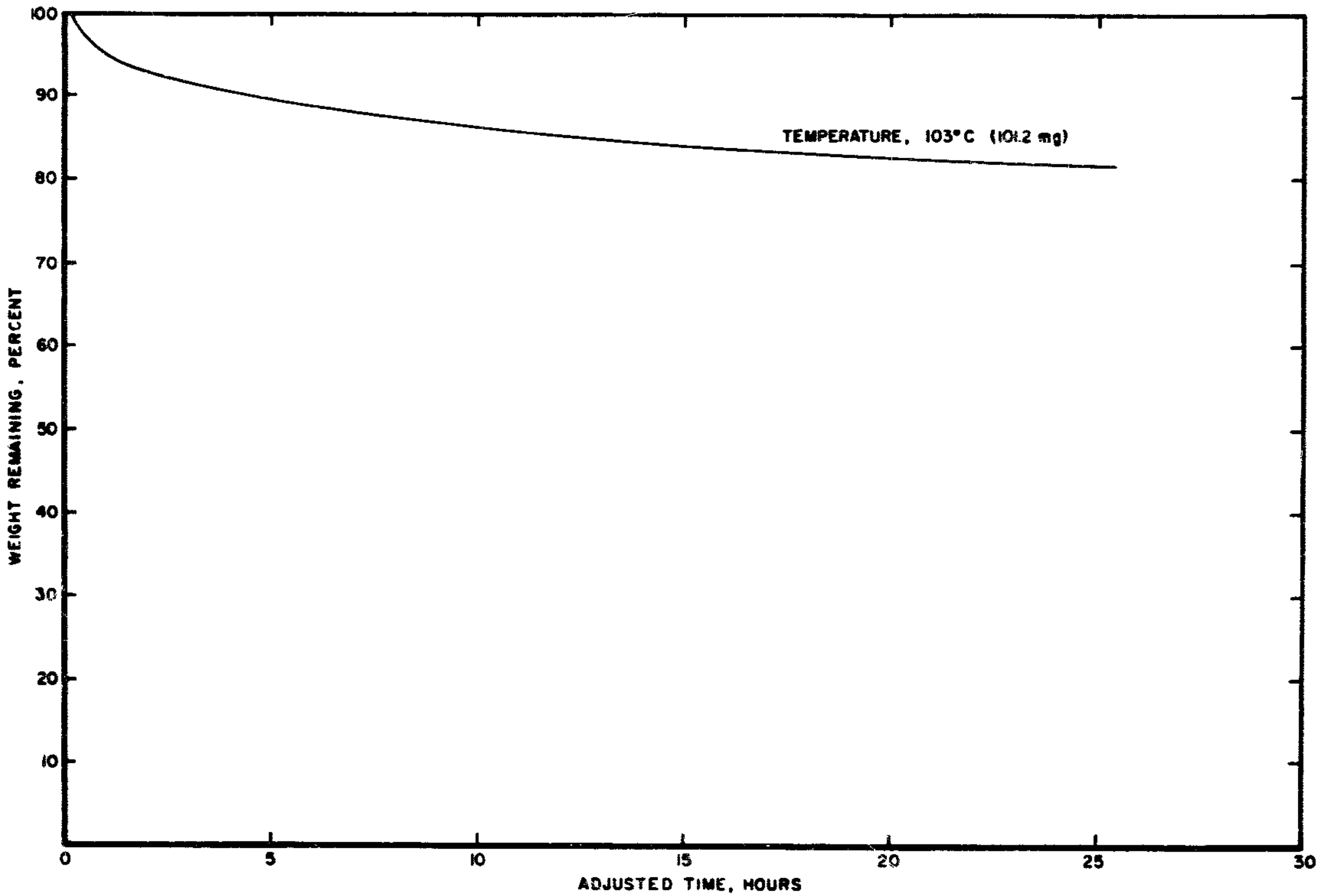

FIGURE 19. TIME-WEIGHT HISTORY FOR MIL-L-6032, 9150-257-5360 DURING EXPOSURE TO VACUUM

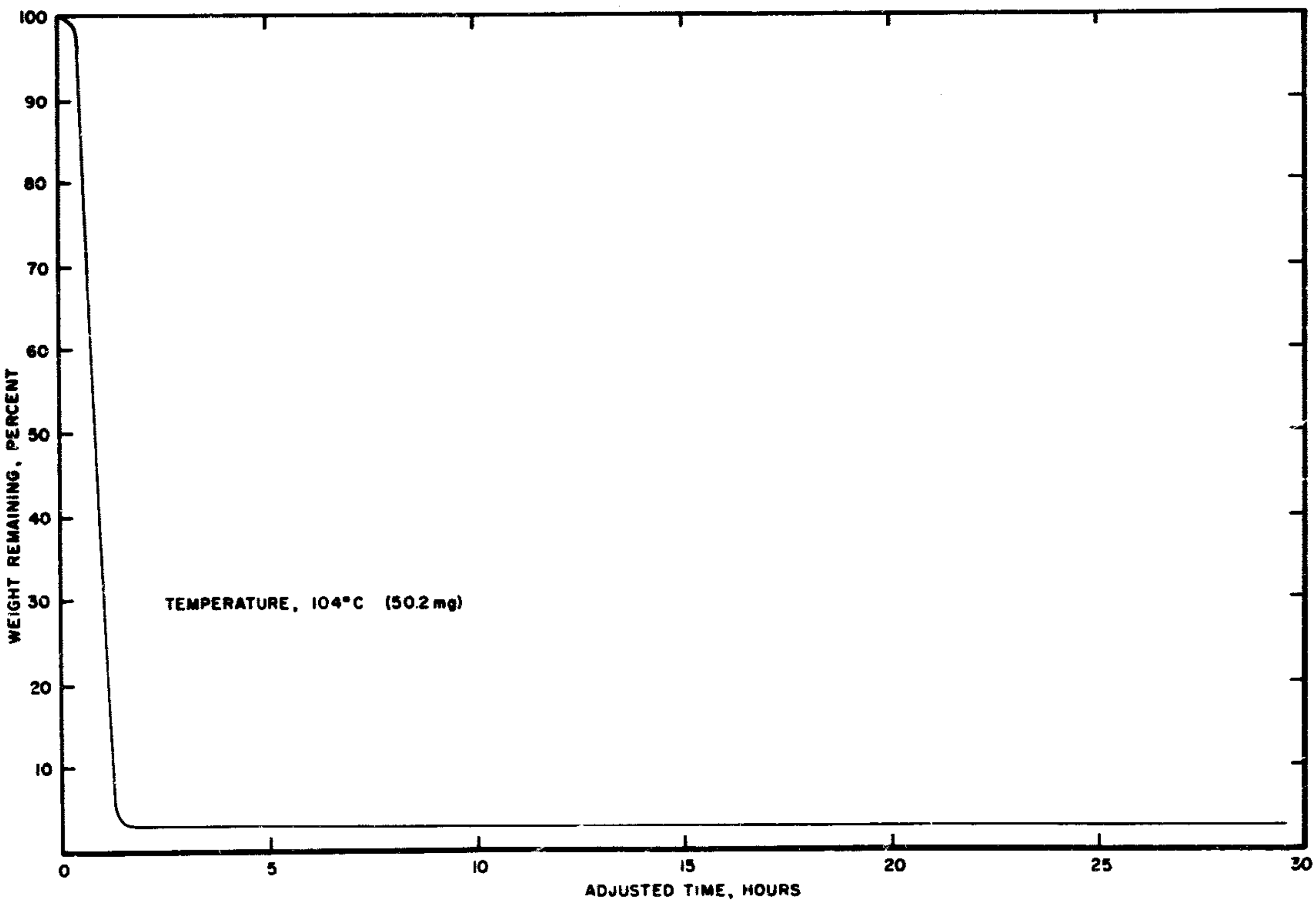

FIGURE 20. TIME-WEIGHT HISTORY FOR OCTOIL DURING EXPOSURE TO VACUUM

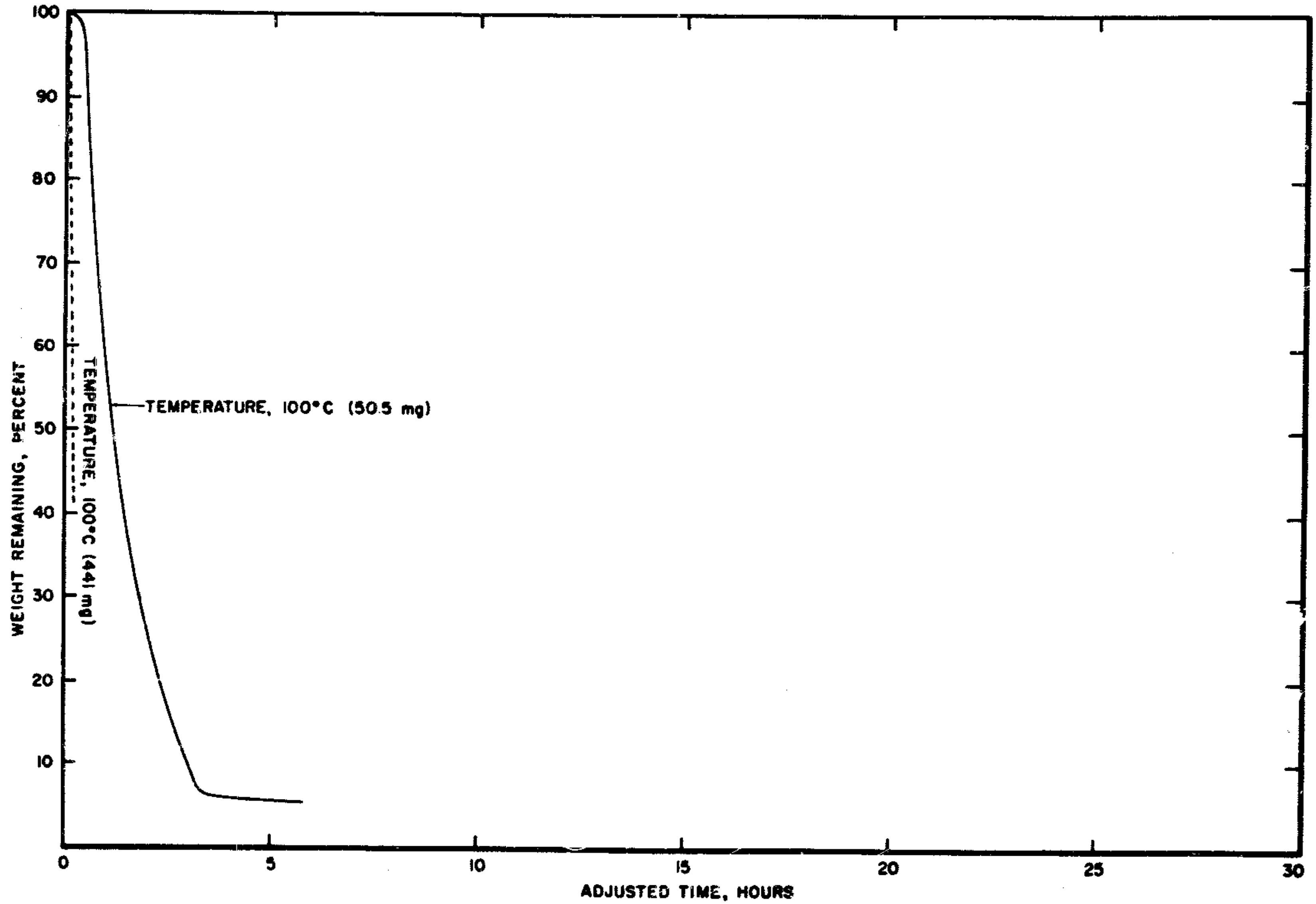

FIGURE 21. TIME-WEIGHT HISTORY FOR OCTOIL-S DURING EXPOSURE TO VACUUM

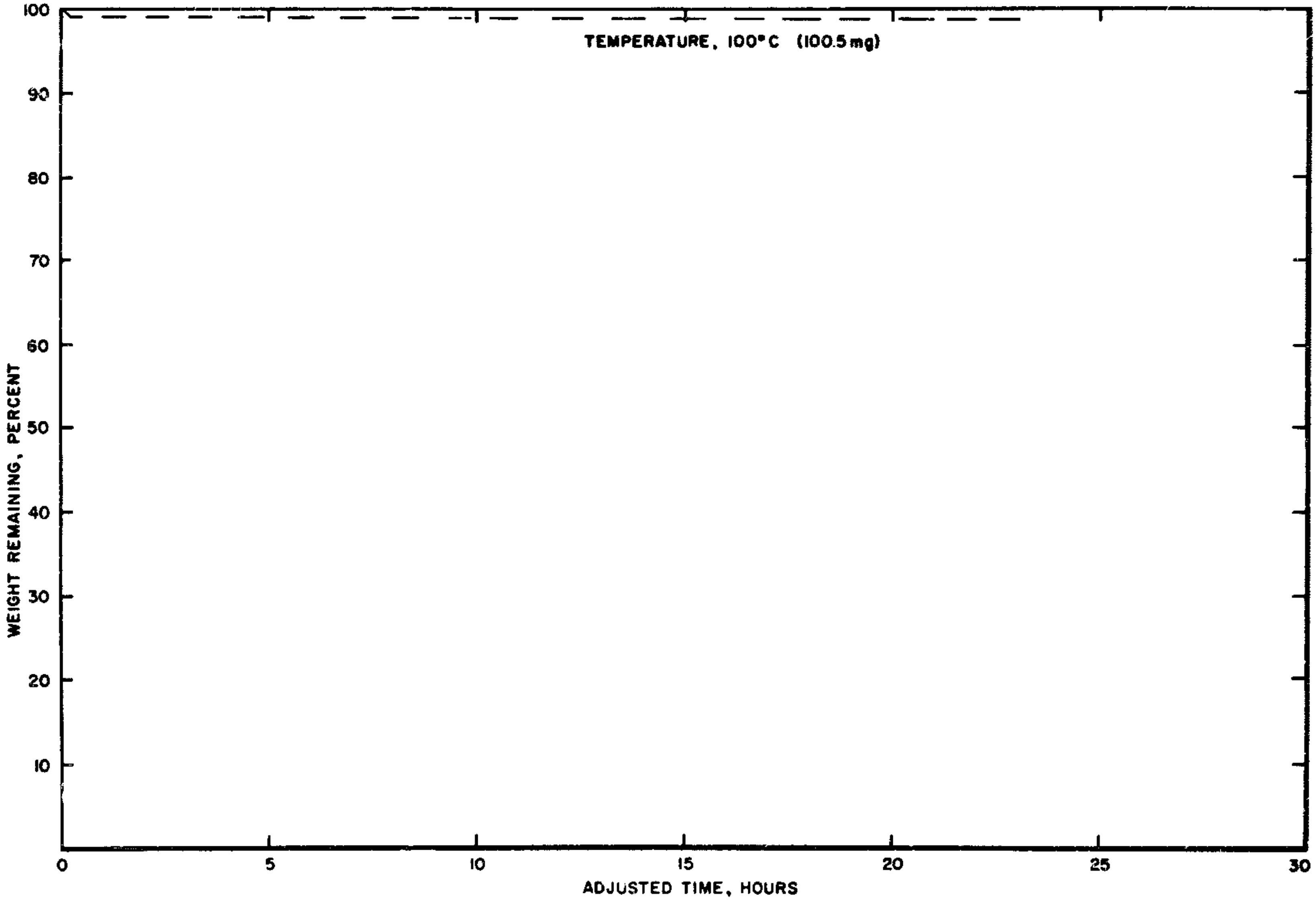

FIGURE 22. TIME-WEIGHT HISTORY FOR RO.KWELL P-4, #950 DURING EXPOSURE TO VACUUM

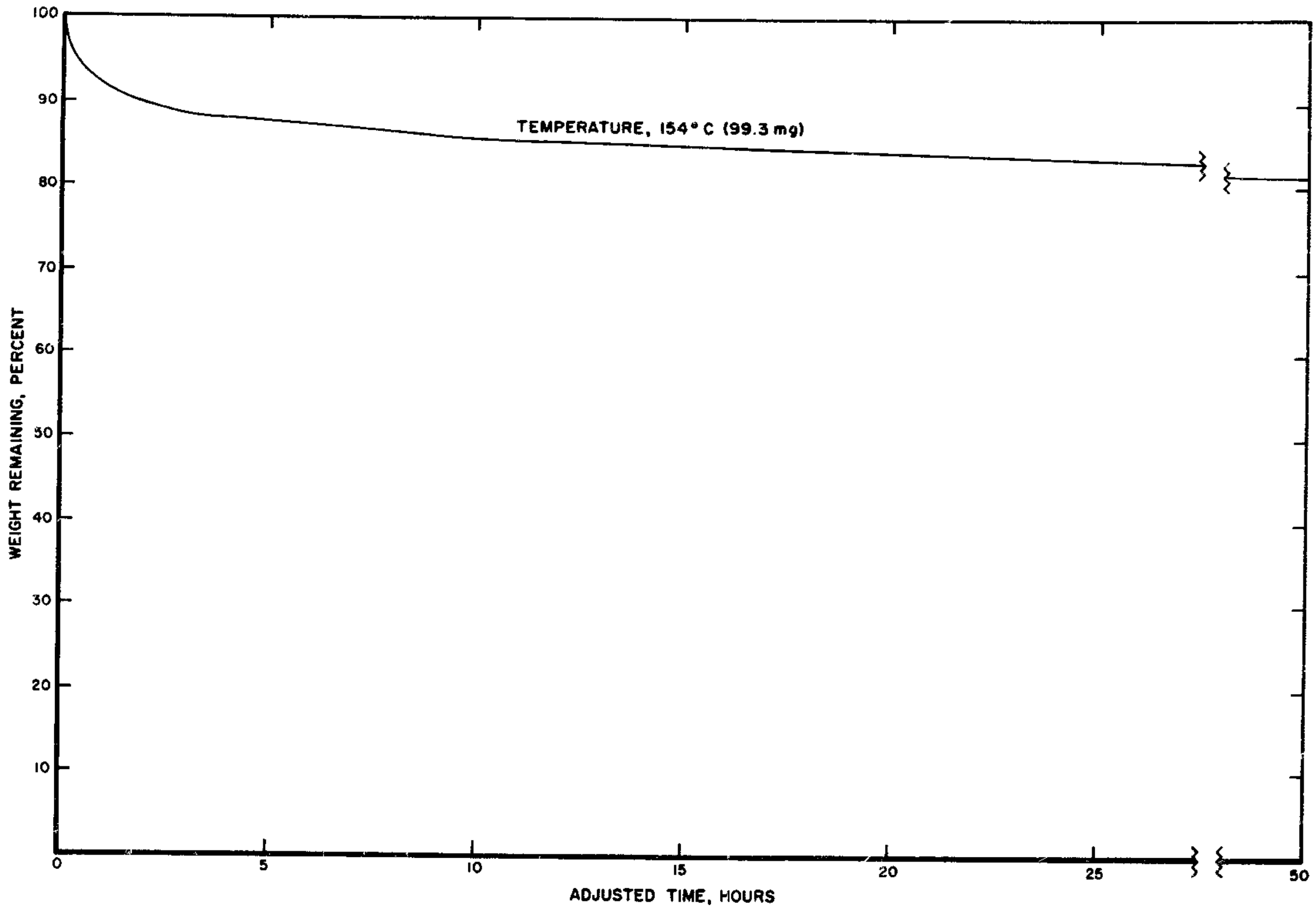

FIGURE 23. TIME-WEIGHT HISTORY FOR SHELL ETR-H DURING EXPOSURE TO VACUUM

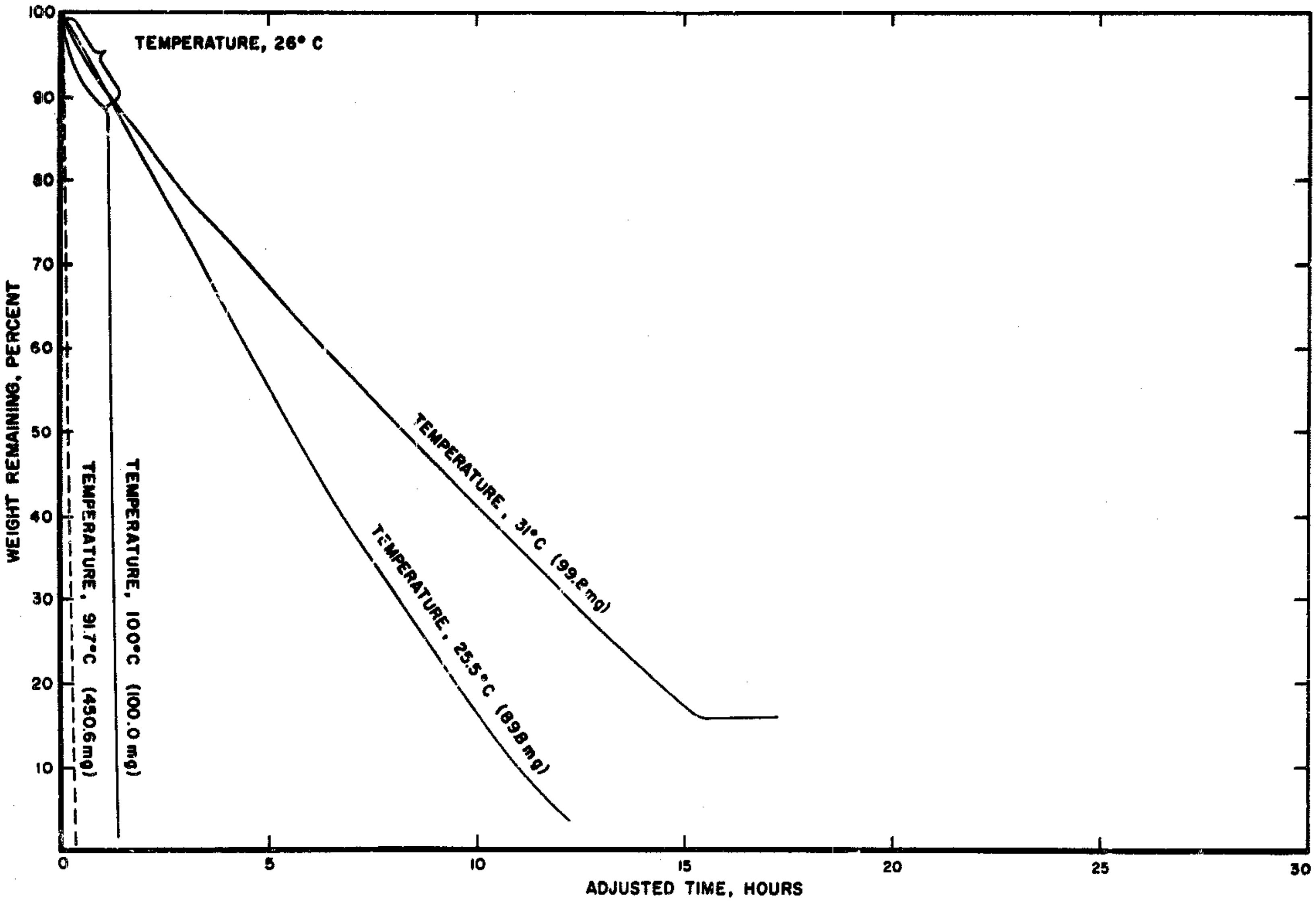

FIGURE 24. TIME-WEIGHT HISTORY FOR TRIETHYLENE GLYCOL DURING EXPOSURE TO VACUUM

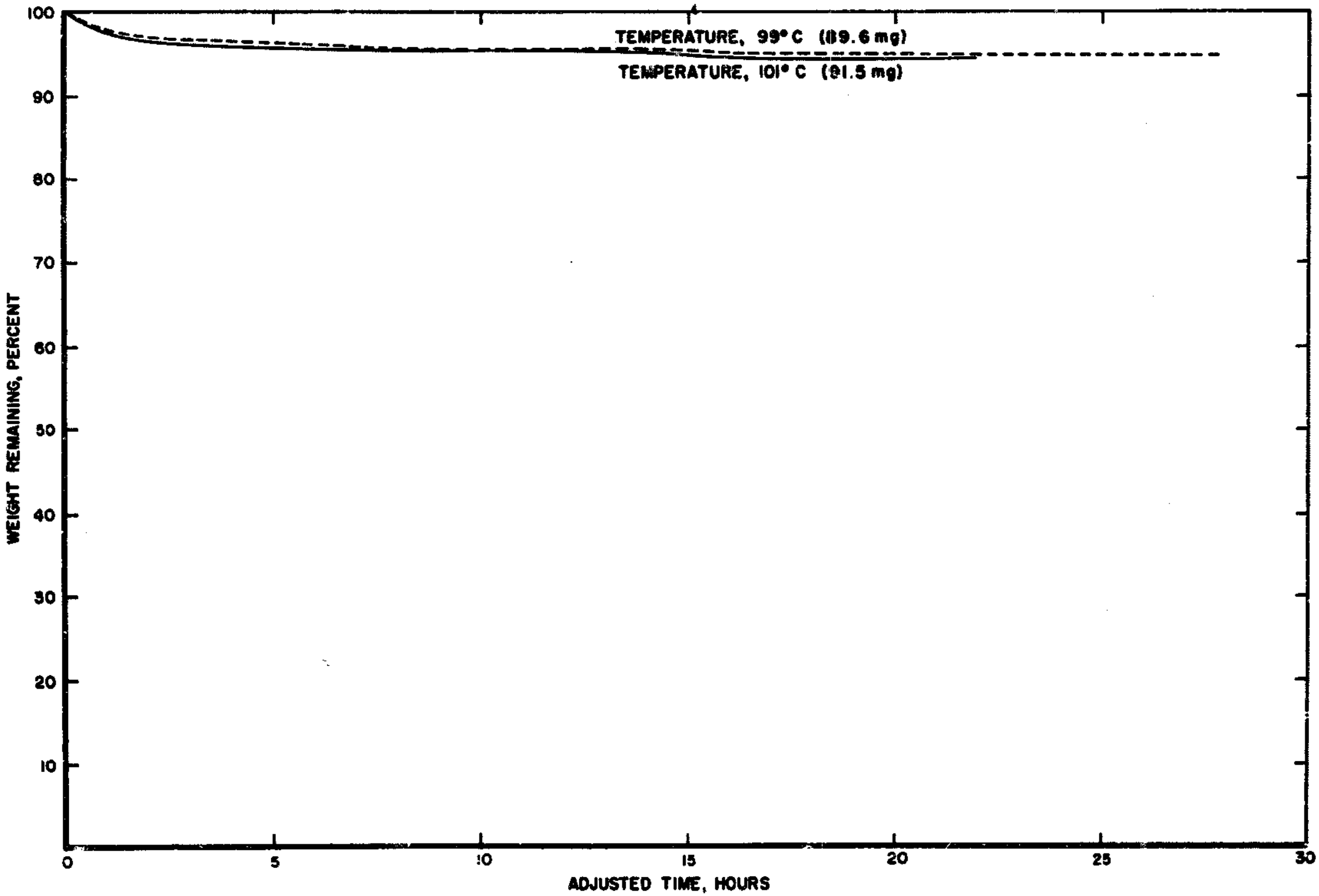

FIGURE 25. TIME-WEIGHT HISTORY FOR UCON LUBE 50-HB-280-X DURING EXPOSURE TO VACUUM

TABLE I

LIQUIDS AND SEMI-SOLIDS VACUUM WEIGHT LOSS

(Sheet 1 of 2)

Material	Temperature, °C Programmed	Temperature, °C Actual	Initial Weight, mg	Weight Loss Total, mg	Weight Loss Total, %	Vacuum System	Figure Number	Date	Run Designation	Run Duration, Hours
Acheson EC 1730	R.T.	R.T.	97.1	49.0	50.5	4B	6	7-13-61	761-1	131
Acheson Colloids	100	100	97.0	74.0	76.4	4B	6	7-20-61	761-3	1.66
	100	101	96.0	74.0	77.1	4B	6	7-19-61	761-2	2.33
Andok C	R.T.	R.T.	83.7	2.7	3.1	4B	7	6-7-61	661-1	9
Humble Oil Refining Company	100	102	100.0	61.6	61.6	4A	7	8-22-63	863-26	21
	100	105	100.8	69.9	69.1	4A	7	8-23-63	863-28	64
California Research Corporation	R.T.	25	151.9	2.0	1.3	4B	8	1-15-62	162-2	25
Grease #159	100	100	149.9	90.0	60.0	4B	8	1-15-62	162-3	65
	125	126.5	209.0	149.4	71.5	4D	8	1-19-62	162-4	57
California Research Corporation	R.T.	22	171.3	88.6	51.6	4B	9	1-4-62	162-1	288
Grease #4669-18-1 (Experimental)	100	100	99.8	84.4	84.6	4B	9	8-23-63	863-29	16
	100	110	99.7	84.8		4B	*	8-22-63	863-27	7.7
Dow Corning 704 Fluid	100	100	450.2	449.3	99.6	4B	10	8-19-63	863-20	2.65
Dow Corning Corporation	100	104	150.1	150.1	100.0	4A	10	8-20-63	863-22	>2
Dow Corning QC-2-0026	R.T.	R.T.	94.3	2.3	2.4	4B	11	7-24-61	761-6	10
Dow Corning Corporation	100	99.5	81.3	8.7	10.7	4B	11	9-14-61	961-1	20
	100	101	97.2	8.7	8.9	4B	11	7-20-61	761-4	10
	100	102	92.5	9.0	9.7	4B	11	7-21-61	761-5	15
Dow Corning QC-2-0093	R.T.	R.T.	94.0	2.5	2.7	4B	12	5-29-61	561-1	7.5
Dow Corning Corporation	100	98	89.3	2.3	2.6	4B	12	7-25-61	761-7	7.5
	100	99	91.8	2.8	3.1	4B	*	7-28-61	761-8	24
Electrolube #2 (Source Unknown)	R.T.	R.T.	86.0	0.6	0.7	4B	*	3-12-62	362-2	24
Fluorolube LC 160	R.T.	R.T.	95.8	20.3	21.2	4B	13	4-6-61	461-2	94
Hooker Chemical Corporation	R.T.	R.T.	91.5	20.0	21.8	4B	*	3-31-61	361-4	94
	100	101	99.9	80.3	80.4	4A	13	8-26-63	863-30	16.5
Fluorolube T45	R.T.	R.T.	86.0	20.0	23.2	4B	14	3-23-61	361-3	20
Hooker Chemical Corporation	100	100.5	89.7	82.2	91.6	4B	14	8-3-61	861-4	100
Fluorolube T80	R.T.	31	87.5	8.5	9.7	4B	15	8-2-61	861-2	17
Hooker Chemical Corporation	50	51.5	87.8	86.2	98.2	4B	15	8-3-61	861-5	500
	100	100	88.7	88.7	100.0	4B	15	8-1-61	861-1	2
	100	100	451.3	451.3	100.0	4B	*	8-20-63	863-23	1.1
	100	100	50.8	50.3	99.1	4A	*	8-21-63	863-24	14.7
	100	100	150.6	150.6	100.0	4B	*	8-21-63	863-25	0.25
	100	101	85.2	85.2	100.0	4B	15	8-2-61	861-3	2

* Not plotted due to its similarity to other curves.

TABLE I

LIQUIDS AND SEMI-SOLIDS VACUUM WEIGHT LOSS
(Sheet 2 of 2)

Material	Temperature, °C		Initial Weight, mg	Weight Loss		Vacuum System	Figure Number	Date	Run Designation	Run Duration, Hours
	Programmed	Actual		Total, mg	Total, %					
Glycerine (Source Unknown)	R.T.	R.T.	96.3	50.0	51.9	4B	16	4-4-61	461-1	23
	R.T.	R.T.	97.4	58.0	59.6	4B	*	4-5-61	461-3	23
	R.T.	23.5	88.5	48.5	54.8	4B	16	3-22-61	361-2	20
	100	100	99.8	99.8	100.0	4B	16	8-26-63	863-31	0.2
MIL-G-3278A	100	100	100.1	85.7	85.6	4B	17	8-30-63	863-38	42
Socony Mobil Oil Company, Inc.	100	101	100.2	83.9	83.6	4A	17	8-30-63	863-37	88
MIL-H-5606 (Source Unknown)	R.T.	R.T.	78.7	70.3	89.4	4B	18	3-9-62	362-1	21
MIL-L-6032, 9150-257-5360 The Parker Appliance Company	100	103	101.2	20.0	19.8	4A	19	8-27-63	863-32	24.8
Octoil Consolidated Vacuum Corporation	100	104	50.2	50.2	100.0	4A	20	8-19-63	863-21	0.8
Octoil S	100	100	50.5	50.5	100.0	4A	21	8-16-63	863-17	2.7
Consolidated Vacuum Corporation	100	100	441.0	441.0	100.0	4B	21	8-16-63	863-18	3 (approx)
Rockwell P-4 #950 Rockwell Manufacturing Company	100	100	100.5	1.0	1.0	4B	22	8-27-63	863-33	0.1 (approx)
Shell ETR-H Shell Oil Company	150	154	99.3	18.0	18.1	4B	23	8-13-62	862-1	50
Triethylene Glycol	R.T.	25.5	89.8	89.8	100.0	4B	24	3-21-61	361-1	12
(Source Unknown)	R.T.	31.0	99.8	84.0	84.2	4A	24	8-28-63	863-34	16.2
	100	91.7	450.6	450.6	100.0	4B	24	8-30-63	863-36	0.1
	100	100	100.0	100.0	100.0	4A	24	8-30-63	863-35	1.1
Ucon Lube 50-HB-280-X	100	99	89.6	4.5	5.0	4B	25	8-9-61	861-7	14
Union Carbide Chemicals Company	100	101	91.5	5.0	5.5	4B	25	8-8-61	861-6	14
Versilube F-50 General Electric Company	50	55	93.5	0.0	0.0	4B	*	3-28-61	361-5	12

* Not plotted due to its similarity to other curves.

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February 5, 1965 APPROVAL NASA TM X-53207

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By

J. G. Austin and J. B. Gayle

The information in this report has been reviewed for security classification. Review of any information concerning Department of Defense or Atomic Energy Commission programs has been made by the MSFC Security Classification Officer. This report, in its entirety, has been determined to be unclassified.

This document has also been reviewed and approved for technical accuracy.

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