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MEASUREMENT AND CONTROL OF MOISTURE IN MOLDED EXPANDABLE POLYSTYRENE

PETER J. MIHALIK

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DOVER, NEW JERSEY

MEASUREMENT AND CONTROL OF MOISTURE IN MOLDED EXPANDABLE POLYSTYRENE

by

Peter J. Mihalik

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Feltman Research Laboratories
Picatinny Arsenal
Dover, N. J.

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OBJECT

The purposes of this investigation were (a) to develop suitable test methods for measuring the moisture content of expandable polystyrene parts, (b) to determine maximum allowable moisture content for procurement purposes, and (c) to develop sampling plans to provide satisfactory levels of inspection for expandable polystyrene.

SUMMARY

Two methods, solvent extraction and Karl Fischer, were investigated as means of measuring the moisture content of molded expandable polystyrene for use in Army Ordnance ammunition packaging. Both methods were satisfactory and gave results which were in agreement. The maximum allowable moisture content for molded expandable polystyrene was established. This moisture content is recommended for adoption in packing drawings and purchase specifications. A small sample inspection plan from MIL-STD-105B is also recommended for adoption.

CONCLUSIONS

The solvent extraction and Karl Fischer methods are satisfactory for determining the moisture content of expandable polystyrene. Of the two methods, the Karl Fischer method is superior because it employs an electrometric titration which allows a higher degree of accuracy and can be utilized for all sample sizes. A significant conclusion of the investigation was that the loss in weight of molded expandable polystyrene at elevated temperatures is due to volatiles other than moisture. Of the samples tested, no significant changes in moisture content were observed within the material during drying at 160° F.

Liquid water on the surfaces of expandable polystyrene in contact with metal parts is expected to corrode these parts. Drying of the foam at elevated temperatures will control this surface water but will not reduce moisture levels within the material itself. Since values for moisture taken after such drying did not exceed 0.3%, this figure can be recommended as a maximum allowable moisture content in packing drawings and purchase specifications.

Humid atmospheres do not substantially affect the moisture content of molded expandable polystyrene. No significant moisture distribution patterns were observed within the material. It is suggested that a small sample inspection plan taken from MIL-STD-105B be adopted for quality control.

RECOMMENDATIONS

It is recommended that the Karl Fischer method rather than the solvent extraction method be adopted as a standard for measuring moisture content of molded expandable polystyrene. It is also recommended that 0.3% be established as a maximum allowable moisture content in future procurements. The following specific steps should be taken:

(1) Amend appropriate packing drawings and purchase specifications.

(2) Adopt a small sample inspection plan from MIL-STD-105B for quality control, and

(3) Keep liquid water away from the parts prior to use.

INTRODUCTION

1. Ordnance field reports indicated that molded expandable polystyrene supports in contact with certain metal parts contribute to the corrosion of these parts.

2. Since most molded expandable polystyrenes for Ordnance packaging are prepared commercially by the application of steam directly into holes of a mold cavity and are subsequently cooled with water, it was believed that residual water on the surface contributed significantly to the corrosion problem. In evidence of this condition, a number of expandable polystyrene containers for the M525 PD fuze, as received for development testing at Picatinny Arsenal, were found to possess a significant amount of surface water. Apparently, it was retained on the surface of the parts as a result of cooling and/or condensation of steam during manufacture. Usually, such surface water exists as a thin film and is extremely difficult to detect visually.

3. Because of the importance of the corrosion problem in Ordnance packaging, a program was initiated to develop suitable test methods for measuring moisture content, to determine maximum allowable moisture content of expandable polystyrene parts during procurement, and to develop sampling plans to provide satisfactory levels of inspection. Examination of an existing procurement specification (MIL-P-19644) showed that no requirement exists for controlling moisture content of molded expandable polystyrene parts during procurement for Ordnance packaging.

RESULTS

4. Figures 1 and 2 show a number of weight loss vs time curves for the drying at 160° F of molded expandable polystyrene samples that had been room temperature conditioned for 4 months (Fig 1) and 2 years (Fig 2). Solvent extraction tests performed on these materials after their drying periods and also on control samples revealed moisture levels of 0.3% maximum. Karl Fischer tests for moisture were also conducted on control samples and on samples that had previously been dried at 160° F for extended periods (Fig 3). Samples were room temperature aged for one year. Figure 4 indicates the effect of exposure in a humid atmosphere (160° F, 60% RH) for an extended period.

5. Molded samples immersed for 16 hours under a 4-inch head of water absorbed exhibited a moisture content of 23%. Karl Fischer tests conducted on molded samples of expandable polystyrene disclosed no significant moisture distribution patterns.

DISCUSSION

Solvent Extraction Method

6. One of the objectives of this study was to develop a testing technique(s) for measurement of moisture content of molded expandable polystyrene. Two methods, both of which involve destructive sampling, were examined. In the first method, solvent extraction (Moisture, Carbon Tetrachloride Distillation Method, MIL-STD-286, Method 102.1), a sample was dissolved in carbon tetrachloride and heated. The residual water and/or moisture in the sample was evaporated, condensed, and measured.

7. Solvent extraction tests were performed on a number of expandable polystyrene containers (for the M525 PD fuze) which had been room temperature aged for 4 months. This test and all subsequent tests were conducted on natural grades of material qualified under MIL-P-19644. Moisture contents were determined on samples previously dried at 160° F for 4 and 90 hours. Values were 0.19% and 0.14%, respectively. The same test, performed on a control sample with dry surfaces, indicated a moisture content of 0.26% (Fig 1). Two-year room-conditioned samples which were subsequently dried for 95 and 120 hours showed moisture contents of 0.29% and 0.19%, respectively (Fig 2).

8. Examination of results revealed no significant change in moisture content of surface-dry samples during drying at 160° F. Since the moisture content remained substantially constant, the loss in weight of the samples was attributed to loss of volatiles rather than loss of moisture. This fact was corroborated when the molded material was examined by infrared spectroscopy for the presence of organic volatiles. At 165° F, a small amount of hydrocarbon was detected. Apparently, small traces of volatile hydrocarbon remained after expansion. A reported analysis of the material in its pre-expanded state discloses a volatile content of 6.5% - 8.0%.

9. The main advantages of this method are a fairly high degree of accuracy and simplicity. A limitation of this method is that sample sizes outside the 50-100 gram range cannot be used.

Karl Fischer Method

10. The Karl Fischer method (Water by Electrometric Titration with Karl Fischer Reagent, Fed Test Method Std 791, Method 3253) involved dissolving an 8-10 gram sample in 60% carbon tetrachloride/40% methanol solution and titrating with standard Karl Fischer reagent.

11. A number of expandable polystyrene supports for the M517 VT fuze which were room temperature aged for one year were subjected to a titration test using Karl Fischer reagent. Results of tests on a group of surface-dry samples revealed moisture contents of 0.12% - 0.20%. The same test, repeated on samples which were dried at 160° F for 90 hours, showed moisture contents of 0.13% - 0.17%.

12. The results obtained with the Karl Fischer method agree with those obtained by the solvent extraction method. No significant changes in moisture content were observed after drying at 160° F and the loss in weight was therefore attributed to volatiles. Since the values did not exceed 0.3% for surface-dry material, this figure can be recommended as a maximum allowable moisture content in future purchase specifications. To ensure compliance, it is recommended that the supplier be held responsible for quality control. Drying at elevated temperatures can be expected to eliminate residual surface water retained from the cooling operation, but care must be exercised during molding to prevent the amount of moisture within the material from exceeding the maximum figure. To maintain these moisture levels, it is required that the Ordnance Corps keep liquid water away from the material prior to use.

Effect of Humid Environments

13. Figure 4 shows the effect of a humid environment (160° F, 60% RH) on an M517 VT fuze support. Losses in weight were observed after 48 hours. Karl Fischer titration tests conducted after the 48-hour exposure period indicated a moisture content of 0.09%. Other samples subjected to 86° F and 90% RH showed no increase in weight after 120 hours exposure. These results revealed that humid atmospheres do not substantially affect the material.

Water Absorption

14. Solvent extraction tests were performed on 2-year-aged, surface-dry samples of molded expandable polystyrene that had been immersed under a 4-inch head of water for 16 hours. A moisture content of 23% was exhibited. This value represents the residual moisture present in the samples prior to immersion (less than 0.3%) and the water retained from water conditioning. Subsequent drying at 160°F for an extended period evaporated the water retained from immersion but did not eliminate the residual moisture present prior to the conditioning period.

Moisture Distribution

15. A number of expandable polystyrene supports for the M21 mine (Ordnance Dwg F8830861) were subjected to the Karl Fischer test to determine whether significant distribution patterns existed. Samples taken directly from the mold were sectioned and tested. Values ranged from 0.08% – 0.12%. The test, repeated on samples cured for 48 hours, showed values of 0.11% – 0.20%. No significant moisture distribution patterns were observed for any group; that is, moisture content at the center of the support was equivalent to that near the surface.

Sampling

16. Since some parts are purchased in large quantity, sampling plans which would provide adequate levels of inspection were investigated. When this testing method is used, the samples are destroyed. Typical plans which can be readily adopted are listed in MIL-STD-105B. Table III-B outlines several suitable plans for small sample inspection. It is suggested that inspection level L2 be used.

EXPERIMENTAL PROCEDURE

17. The solvent extractions were conducted in accordance with the procedure in MIL-STD-286, Method 102.1 (Moisture, Carbon Tetrachloride Distillation Method). Covers of molded expandable polystyrene containers which weighed approximately 78 grams each were dissolved in carbon tetrachloride.

18. The Karl Fischer tests were conducted in accordance with the procedure outlined in Federal Test Method Standard No. 791, Method 3253 (Water by Electrometric Titration with Karl Fischer Reagent). Molded M517 VT supports of expandable polystyrene which weighed approximately 7 grams each were dissolved in a 60/40 carbon tetrachloride/methanol solution.

19. For the water absorption test, molded samples of M525 containers were immersed for 16 hours under a 4-inch head of water.

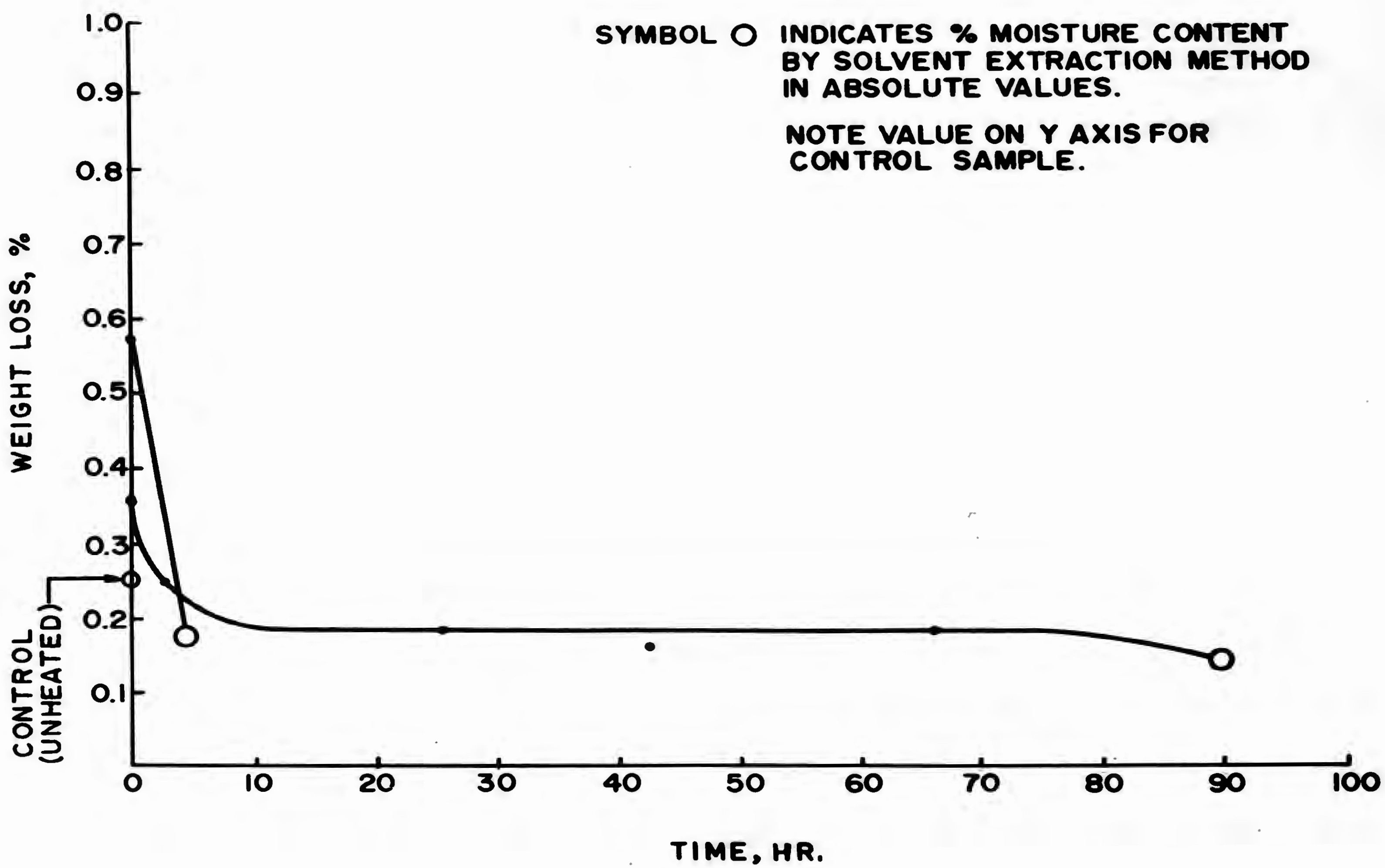

Figure 1. Effect of Elevated Temperature on Moisture Content as Determined by Solvent Extraction Method (Four month Aged Samples)

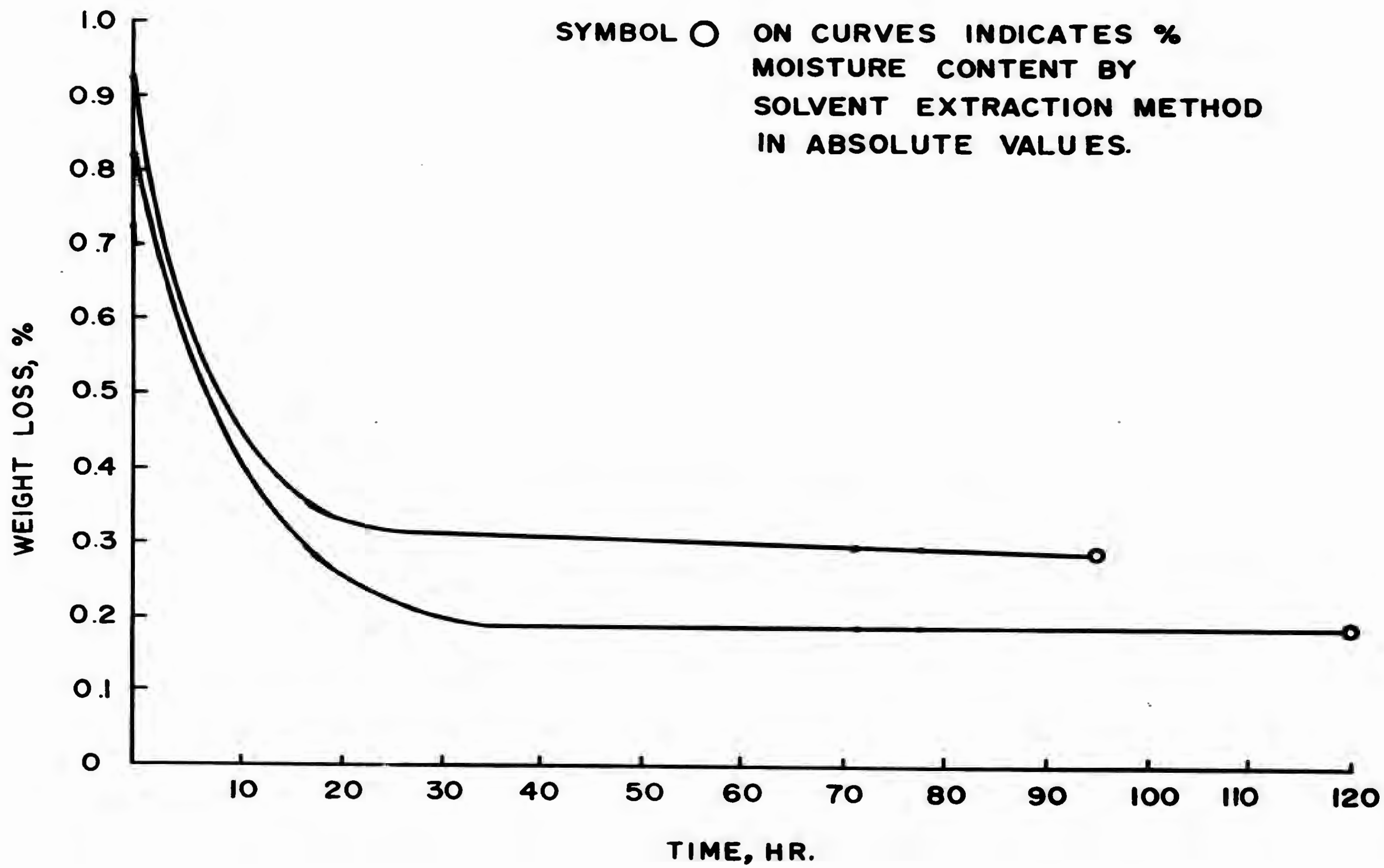

Figure 2. Effect of Elevated Temperature on Moisture Content as Determined by Solvent Extraction Method (Two Year Aged Samples)

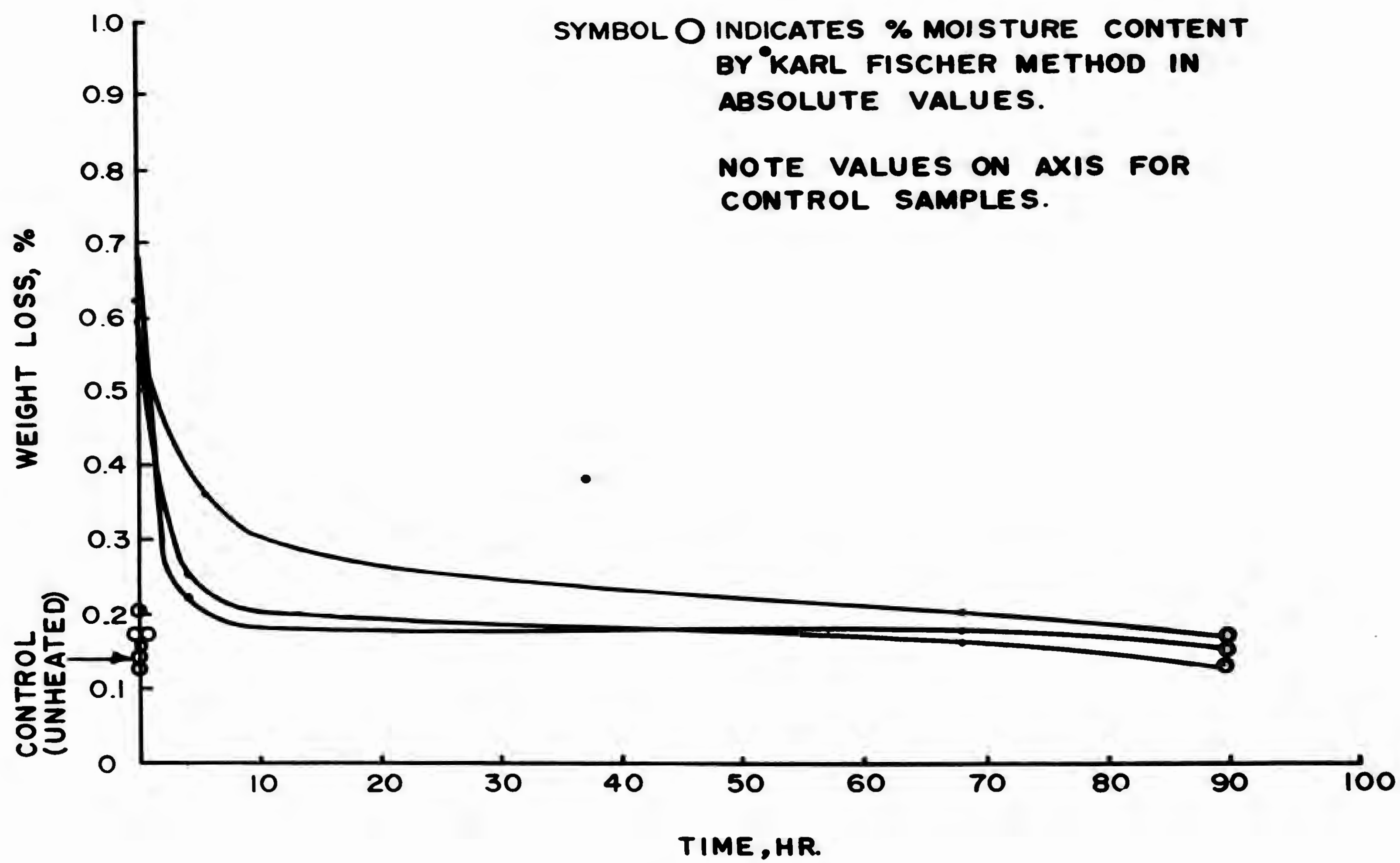

Figure 3. Effect of Elevated Temperature on Moisture Content as Determined by Karl Fischer Method

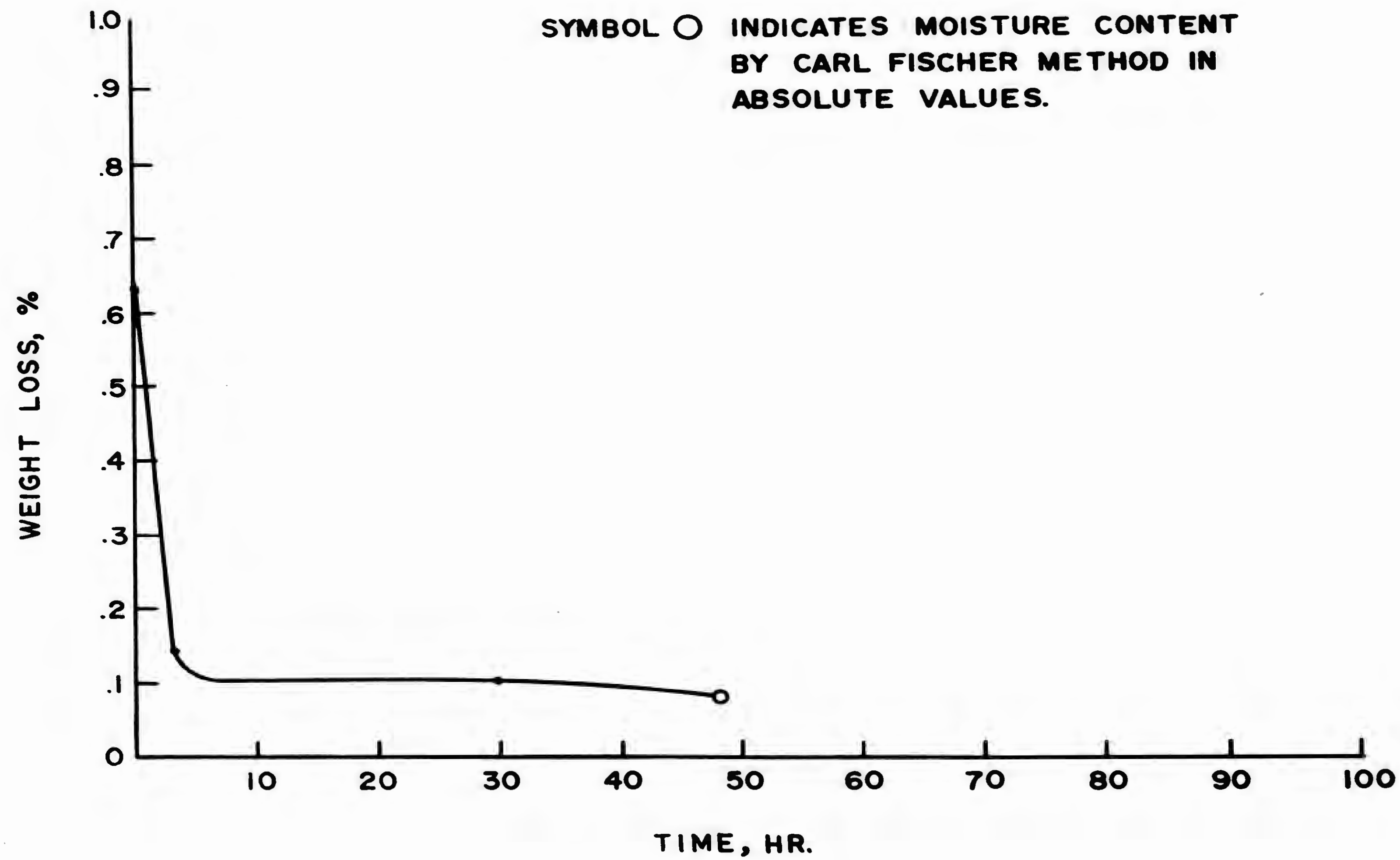

Figure 4. Effect of Humid Atmosphere (160°F, 60% RH) on Moisture Content

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I. Mihalik, Peter J.
II. OMS Code 4110.16.2136.12

UNITERMS

Measurement
Control
Moisture
Molded
Expansion
Polystyrene

AD________________Accession No.________
Feltman Research Laboratories
Picatinny Arsenal, Dover, N. J.
MEASUREMENT AND CONTROL OF MOISTURE IN MOLDED EXPANDABLE POLYSTYRENE
Peter J. Mihalik

Technical Report 3033, September 1962, 21 pp, figures. DA Proj none, OMS Code 4110.16.2136.12. Unclassified Report

Two methods, solvent extraction and Karl Fischer, were investigated as means of measuring the moisture content of molded expandable polystyrene for use in Army Ordnance ammunition packaging. Both methods were satisfactory and gave results which were in agreement. The maximum allowable moisture content is

(over)

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