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GAMMA IRRADIATION TESTING OF MONTAN WAX BARRIER MATERIALS FOR IN-SITU WASTE CONTAINMENT

Peter Soo and John Heiser

February 1996

Environmental and Waste Technology Center
Department of Advanced Technology
Brookhaven National Laboratory
Upton, NY 11973-5000

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ABSTRACT

A scoping study was carried out to quantify the potential use of montan wax as a barrier material for subsurface use. If it possesses resistance to chemical and structural change, it could be used in a barrier to minimize the migration of contaminants from their storage or disposal locations. Properties that were evaluated included hardness, melting point, molecular weight, and biodegradation as a function of gamma radiation dose. Based on the data obtained to date the wax is extremely resistant to radiation-induced change. Coupled with low permeability, the material shows promise as a barrier material.

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EXECUTIVE SUMMARY

Some preliminary work has been carried out to characterize montan wax for potential use as a major waste barrier constituent for subsurface use. The main emphasis was to quantify the wax's long-term ability to withstand radiation-induced mechanical, chemical, and microbial degradation. The study included the measurement of mechanical property changes (specifically hardness), chemical changes (molecular weight) and microbial attack as a function of gamma-irradiation doses in air. The following conclusions have been made based on results obtained to date:

a) Irradiation up to doses of 10^8 rad did not cause any consistent change in the hardness of pre-melted montan wax as measured using a standard needle penetrometer test.

b) Irradiation gives an increase in the weight of montan wax. It is probably caused by irradiation-induced oxidation. The increases are very small; about 0.07 % after an irradiation dose of 10^8 rad. The weight increase is proportional to the dose.

c) Differential scanning calorimetry was conducted to determine if irradiation causes any changes in structure that could be detected through changes in melting point. Irradiation did cause small changes in the distribution of the melting points of constituents in the wax but the overall main melting point remained at about 88^0C regardless of the amount of irradiation. In a few cases, specimens showed a higher-melting-point constituent was present. There was no correlation with radiation dose. It is speculated that the constituent is associated with local differences in the amount of crystallinity in the samples that was caused by small differences in the wax melting procedure during sample preparation.

d) Gamma radiation does not cause any consistent changes in the molecular weight of the wax. It appears to have high resistance to radiation-induced structural change.

e) Using the standard ASTM G22 test there was no detectable bacterial degradation on unirradiated or irradiated wax. A small amount of fungal growth was observed using the ASTM G21 test. It is quite common to observe such fungal attack in polymeric materials since the test is, apparently, a severe scoping test. Under actual soil service conditions fungal attack of montan wax by indigenous fungi may not occur. Prototypic tests are recommended to confirm this. Based on the resistance to molecular weight decreases caused by irradiation, the wax should not suffer increased susceptibility to biodegradation as a result of long-term radiation during service.

In summary, the limited tests on montan wax show that there are no major degradation modes that would disqualify its use as a component in a subsurface waste barrier. Its low cost and resistance to mechanical, chemical, and microbial deterioration warrant an expanded evaluation of its potential applications.

ACKNOWLEDGMENTS

The authors gratefully acknowledge Mr. Martin Clark of Strohmeyer and Arpe, Inc., for supplying the montan wax that was evaluated in this study. Dr. Janardan Upadhyaya of Petrofin Corporation performed the needle penetrometer tests. Mr. Jay Adams was of great help in demonstrating the use of the differential scanning calorimeter and the data analysis. Finally, the authors express their gratitude to Ms. Grace Webster for preparing the report for publication.

1. INTRODUCTION

The U.S. Department of Energy is sponsoring the development of low-permeability barriers that can be emplaced around waste storage and disposal facilities. One promising technology involves the use of a wax extracted from lignite coal (montan wax) that can be injected as an emulsion into soil surrounding these facilities. Upon solidification, a high-integrity, low-permeability barrier will be formed. The wax is inexpensive (about 75 cents/lb) and does not require a high-temperature process for emplacement.

In order for such a technology to be successful, the barrier material must be cost effective, be capable of penetrating soil to form a continuous barrier, and maintain its properties (especially its low permeability) in the service environment. Montan wax/bentonite grouts were first evaluated in Germany. To date, three field-scale pilot studies have been completed. Recently, laboratory tests on montan wax/sodium silicate grouts have also been completed by Golder Associates.[1]

As polymeric materials, waxes are susceptible to damage from radiation. Gamma ionizing radiation is likely to be the more important type of radiation because of its presence in many radioactive waste streams and its ability to penetrate large thicknesses of matter. Therefore, if wax-based materials are to be qualified as barriers for wastes containing radioactive constituents, a knowledge of the effects of radiation on the durability and mechanical properties of montan wax over extended periods of time needs to be obtained.

Among the important gamma-induced damage mechanisms that could affect barrier life include:

a) crosslinking of the polymer chains that would increase the molecular weight, increase strength, and reduce plasticity,

b) chain scission that would decrease the molecular weight and strength, and

c) gas generation (mainly H_2, CH_4, CO, CO_2), that could conceivably cause grout swelling and cracking.

Polymer chain scission is one of the more important damage mechanisms since lowering of the molecular weight of the wax could make it susceptible to biodegradation from microbes in the soil. It is generally accepted that the higher the molecular weight of a polymer, the less likely is microbial degradation.

2. EXPERIMENTAL

The present study is centered on four separate areas which are outlined below.

2.1. Gamma Irradiation Effects on Mechanical Properties

This Task evaluated irradiation-induced changes in hardness of the wax. Since many DOE sites contain radioactive constituents, gamma irradiation may cause degradation of the organic wax component. As a conservative estimate it will be assumed that a barrier will be subjected to a constant gamma field of 500 rad/h. Over an estimated barrier lifetime of 25 y an integrated dose of about 10^8 rad will be reached. Individual montan wax samples were prepared and irradiated in the BNL cobalt-60 Gamma Irradiation Facility. The dose rate was 1.45×10^6 rad/h and the nominal irradiation temperature 20°C. Target doses of 10^7, 5×10^7, and 10^8 rad were used in order to bracket the possible dose levels. Irradiation times were 6.9 h, 34.5 h, and 69 h, respectively.

After irradiation, the samples underwent the following tests:

a) they were checked for weight changes,

b) they were examined to determine if any surface changes had occurred (e.g. had they become sticky, changed color, or developed a different odor?),

c) they were tested for hardness using a needle penetrometer to determine if the mechanical properties had been altered, and

d) selected samples were chilled to liquid nitrogen temperature and fractured by an impact load. The fractured surfaces were examined under a high-powered microscope for the presence of gas bubbles generated by radiolysis.

All of the above observations for the irradiated samples were correlated with those taken from unirradiated control samples maintained at the irradiation temperature.

2.2. Differential Scanning Calorimetry

This technique was used to detect any significant chemical and structural changes in the montan wax caused by the irradiation. These include phase changes and changes in molecular weight. If such changes occur during irradiation, then it should be indicated by the appearance of a spectrum of melting points in the wax. The calorimeter is an instrument that provides the ability to quantify these changes after gamma irradiation. It measures the heat inputs to an irradiated specimen and an unirradiated control while their temperatures are increased at an identical predetermined rate. A large increase in the heat input rate to the irradiated specimen

compared to the control shows that some new component is melting or vaporizing.

2.3 Molecular Weight Determinations

A rule-of-thumb for polymeric materials is that biodegradation is possible if the molecular weight is less than approximately 500. If irradiation-induced chain scission in the wax is capable of reducing the molecular weight into this range, then the long-term stability of these barrier could be compromised. Note that the measured molecular weight would be an average but, if it has been substantially reduced from the value for unirradiated material, there is a potential for biodegradation to occur. This would be a warning that long-term losses in barrier integrity from this mechanism are possible. Molecular weight measurements were made to determine if there were any radiation-induced changes.

2.4 Biodegradation Evaluation

Task 4 is important to determine whether biodegradation may occur in irradiated wax-based grouts. Since irradiation may reduce the molecular weight of polymeric materials, biodegradation may be initiated. (It is generally accepted that biodegradation is more likely in low molecular weight polymers). Material irradiated to 5×10^7 and 10^8 rad was used to determine if biodegradation would occur under specific protocols.

The NRC-recommended biodegradation tests were used (ASTM G21 and G22). The first test is for degradation caused by fungi, whereas the second is concerned with bacterial-induced attack. These tests are commonly used to determine the susceptibility of low-level waste forms to microbial degradation.

3. RESULTS

3.1 Sample Preparation and Irradiation

The montan wax starting material was donated by Strohmeyer and Arpe Company, Inc., NJ, who are the US distributors for this German material. The sample received is designated Romonta Type R montan wax. It was in the form of almost black granules that were from 0.8 to 1.0 mm in diameter. The standard test samples that were used in the current study measured 47 mm in diameter by about 10 mm in thickness. They were prepared by placing petri dishes filled with the wax granules onto a hotplate where they became molten after about eight minutes. The wax was allowed to remain at the meting point, about 87^{0}C, for another minute to allow the small numbers of minute air bubbles to be released at the surface. The petri dishes were removed from the hotplate and placed on the bench top to allow the wax to re-solidify. This took approximately 15 to 20 minutes. Except for the surface layer, which became light brown after solidification, the wax discs were almost black in color. The brown surface layer appears to consist of less-dense wax constituents that separate out during melting. The petri dishes were covered with standard glass lids to minimize contamination.

Irradiation was carried out using the BNL Gamma Irradiation Facility (GIF). It consists of a number of four-inch diameter stainless steel irradiation tubes that are placed vertically in a deep pool of water. The tubes are closed at the bottom ends so that the tubes are filled with air on the inside and in contact with 10^{0}C water on the outside. Cobalt-60 irradiation sources are placed around the base of the tubes to provide gamma radiation to samples that are lowered into the tubes from above. Figure 1 shows a view of four irradiation tubes surrounded by the Co-60 sources.

As mentioned above, the wax samples were irradiated to three different dose levels. For each dose, nine wax discs contained in covered petri dishes were stacked on top of each other and place inside a stainless steel "bucket" which was lowered down the irradiation tube using a metal chain. Care was taken to ensure that they resided in the location of constant gamma flux. In an initial trial test, a sample containing an embedded thermocouple was examined for increases in temperature as a result of gamma heating. It was found that the normal irradiation temperature had only increased from 10 to 20^{0}C.

After irradiation, the wax discs were examined for superficial changes. Figure 2 shows that radiation causes the top surfaces to change in color from brown to brown-black. The most highly irradiated samples seemed to have a slightly stronger smell, but the samples lost virtually all odor after a few weeks.

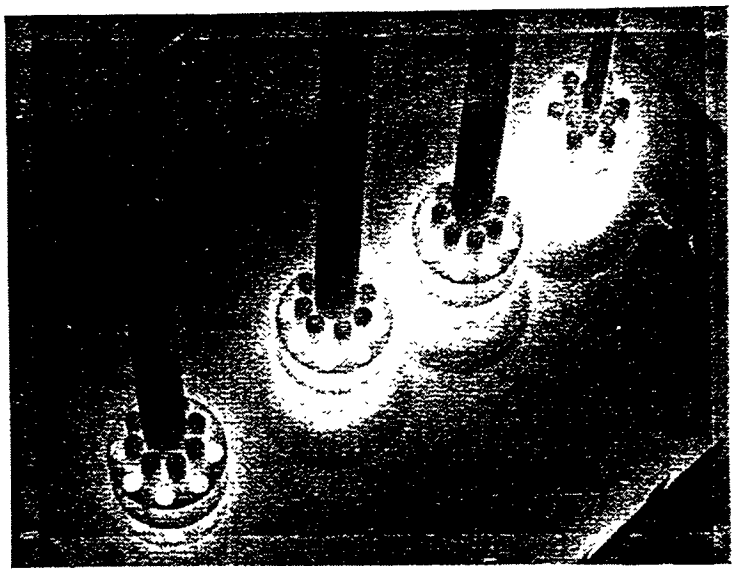

Figure 1. View of Four Cobalt-60 Source Arrays Surrounding Specimen-Irradiation Tubes.

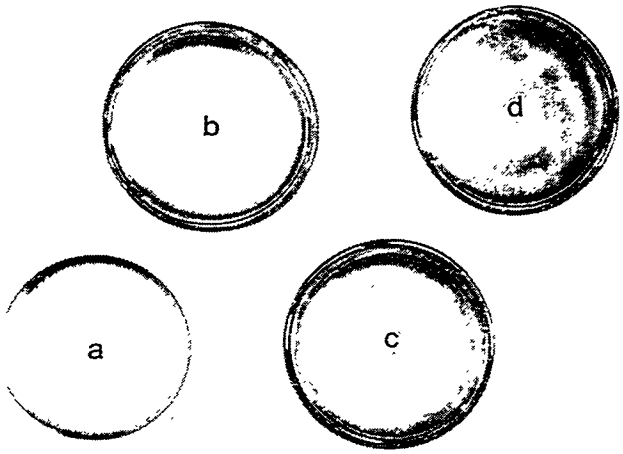

Figure 2. Montan Wax Discs Irradiated to Different Gamma Doses; (a) 0 rad, (b) 10^7 rad, (c) $5x10^7$ rad, and (d) 10^8 rad.

3.2 Effect of Irradiation on Weight Change

Gamma irradiation may be expected to cause a weight change in a polymer as a result of chemical changes. For example, it is known that polyethylene will suffer from irradiation-induced oxidation[2]. The resultant absorption of oxygen from the surrounding air should cause an increase in weight. On the other hand, the generation of radiolytic gases such as hydrogen. methane, and carbon oxides would cause a weight loss. Table 1 gives the results of weight change measurements taken before and after irradiation. The individual weights include those of the wax samples and the petri dishes and covers.

Table 1. Weight increases for montan wax during gamma irradiation

Dose (rad)	Spec. #	Weight (g)		Increase (%)
		Initial	Final	
1.0×10^7	1	75.29279	75.29856	0.0077
	2	71.27171	71.27743	0.0080
	3	74.55838	74.56246	0.0055
	4	71.75123	71.75738	0.0086
				Ave. 0.0075
5.0×10^7	1	73.64054	73.64701	0.0088
	2	72.30672	72.34486	0.0527
	3	76.25410	76.29153	0.0491
	4	73.17751	73.21198	0.0471
	5	72.03828	72.06936	0.0431
	6	74.75781	74.78863	0.0412
	7	69.96099	69.98227	0.0304
	8	69.85092	69.87892	0.0401
	9	76.68692	76.70691	0.0260
				Ave. 0.0420
1.0×10^8	7	75.15920	75.18624	0.0360
	10	46.40338	46.45789	0.1170
	11	50.10208	50.13338	0.0625
	12	52.33697	52.37452	0.0717
				Ave. 0.0718

For samples irradiated to 10^7 rad there is a very small weight increase of 0.0075 %. At the 5.0×10^7 and 10^8 rad dose levels the weights increase by 0.042 % and 0.072 %, respectively. Based on these data the weight increase is proportional to the dose.

A limited amount of microscopical examination was performed on selected wax samples to determine if gas generation and bubble formation could be present as a result of irradiation. In order to increase the possibility of observing gas bubbles the irradiated samples were chilled to liquid nitrogen temperature (77^0K) and struck with a hammer. The intent was to try to produce a flat cleaved fracture surface that would be relatively featureless. However, the samples did not cleave well and the ability to observe minute features was restricted. Examination to magnifications up to about 200X did not reveal any evidence of bubble generation. Gas bubbles could conceivably form but, if present, they would be extremely small.

3.3 Effect of Irradiation on Mechanical Properties

The wax samples were tested for hardness after irradiation by Petrofin Corp., NY. A needle penetrometer was used according to the ASTM D-1321 methodology. The instrument was a Lab Line Instrument Penetrometer (Cat. # 4100T). The test involves measuring the depth of penetration of a needle into the wax surface under a load of 100 g. Prior to and during test the samples were immersed in a water bath at 25^0C (77^0F), the test temperature. Hardness measurements were taken on two duplicate samples. The top surfaces of the samples as well as on the bottom surfaces that were in contact with the petri dishes. Three separate indentations were made on each surface to check for reproducibility.

Table 2 shows the results obtained. It is noticed that the top surfaces of the wax discs are relatively soft with a needle penetration depth of about 0.3 mm. One of the samples that was irradiated to 5×10^7 rad had a harder surface with a penetration of only 0.1 mm. The reason for this anomalously low value is not known. Based on these data there is no significant affect of gamma irradiation on the hardness of the top surfaces of the wax samples.

The results also show that the hardness of the bottom surfaces of the samples are also not significantly influenced by radiation to 10^8 rad. However, the depth of penetration is much less than that observed for the top surfaces showing that the material in this location is much harder. It is probable that the softer top surface of the samples is caused by a less dense wax component that floated to the top of the samples as they were prepared during melting in the petri dishes.

Table 2. Effect of irradiation on the hardness of montan wax.

Irradiation Dose (rad)	Penetration (mm x 0.1)	
	Top Surface	Bottom Surface
0	3, 3, 3	1, 1, 1
0	3, 3, 3	1, 1, 1
10^7	3, 3, 3	0.5, 0.5, 0.5
10^7	3, 2.5, 3.5	3, 2, 2
5.0×10^7	3, 2.5, 3	1, 1, 1
5.0×10^7	1, 1, 1	1, 1, 1
10^8	3, 3, 3.5	1, 1, 1
10^8	3, 3, 2	1, 1, 1

3.4 Differential Scanning Calorimetry Evaluations

Figures 3 through 7 show the differential scanning calorimetry data obtained for as-received montan wax granules in the unirradiated state as well as results for melted wax samples that received various gamma doses. Triplicate specimens were tested for each condition. The specimens were all heated individually at a rate of 5^0C/min. Inflections in the curves indicate that the heat being supplied to the specimens increases at a faster rate showing that some component in the wax is beginning to melt.

For the as-receive unirradiated granules there is an initial steep change in the milliwatts of heat supplied to each milligram of sample (mW/mg) indicating that some wax component is being melted or vaporized. At about 45^0C another inflection is seen. The very large dip in the curves at about 88^0C is the main melting point for the wax. For the granules (Fig. 3) there is excellent reproducibility for the three specimens. However, for unirradiated wax samples that had been prepared by melting in the petri dishes (Fig. 4) there is more scatter in the data. These differences in the rate of heat input for supposedly similar samples suggest that subtle changes were induced in the wax samples as they were prepared by melting. The melting points, however, were still at about 88^0C.

Two of the samples displayed a higher-temperature melting point component at about 106^0C. At this time it is conjectured that this may be a result of crystallinity differences that were caused when the samples were prepared by melting.

For the three sets of irradiated samples, there is no major observable effect of irradiation (see Figs. 5 through 7). The melting points remain at about 88^0C although the melting point ranges (width of the V-shaped sections of the curves) are narrower than for the unirradiated wax granules. In fact, the V-shaped sections for all melted wax samples, regardless of irradiation dose, have developed secondary inflections. This shows that after melting the wax in the petri dishes is became converted into two main components that have discrete but very similar melting points.

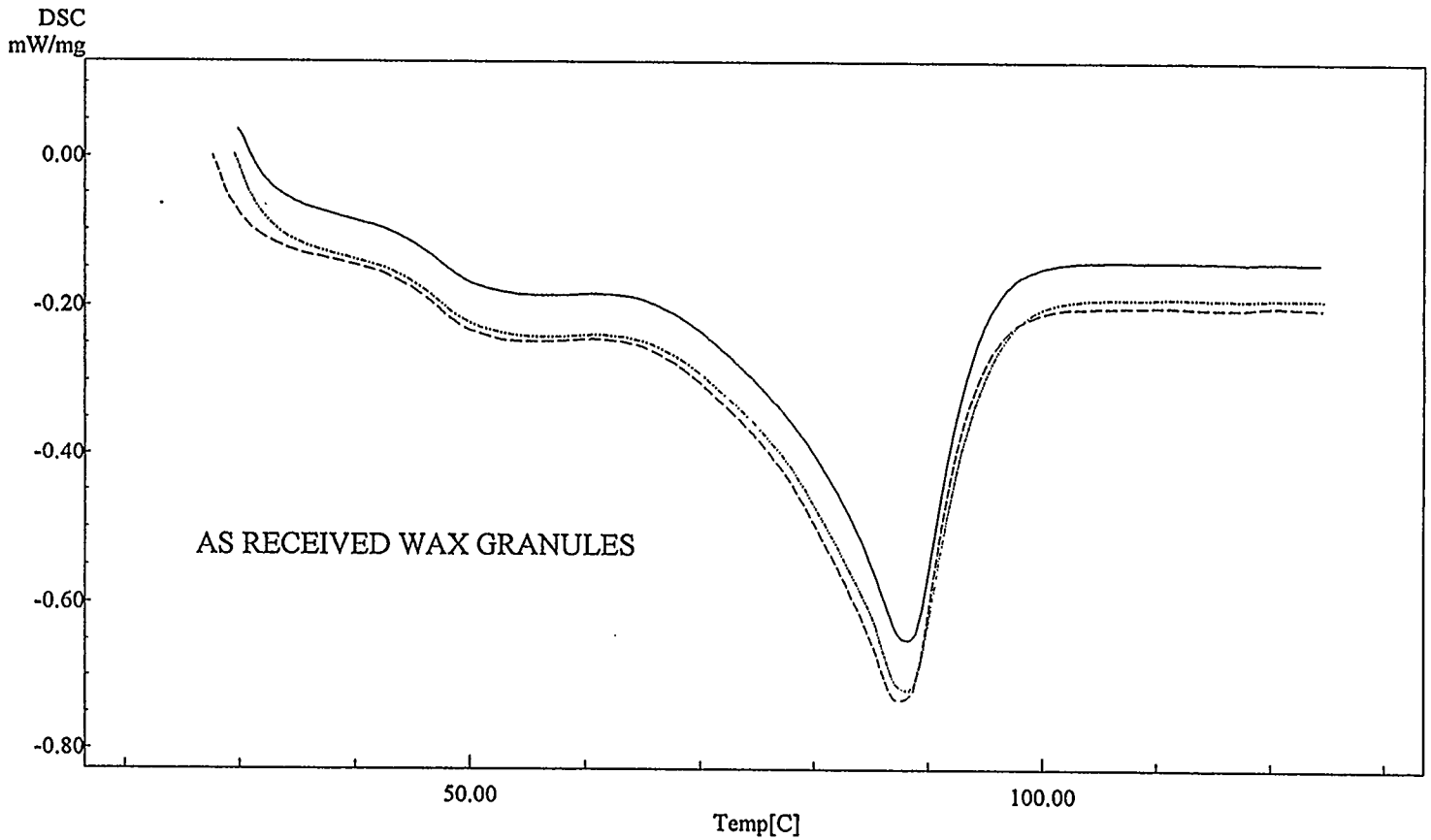

Figure 3. Differential Scanning Calorimetry Results from Unirradiated Montan Wax Granules.

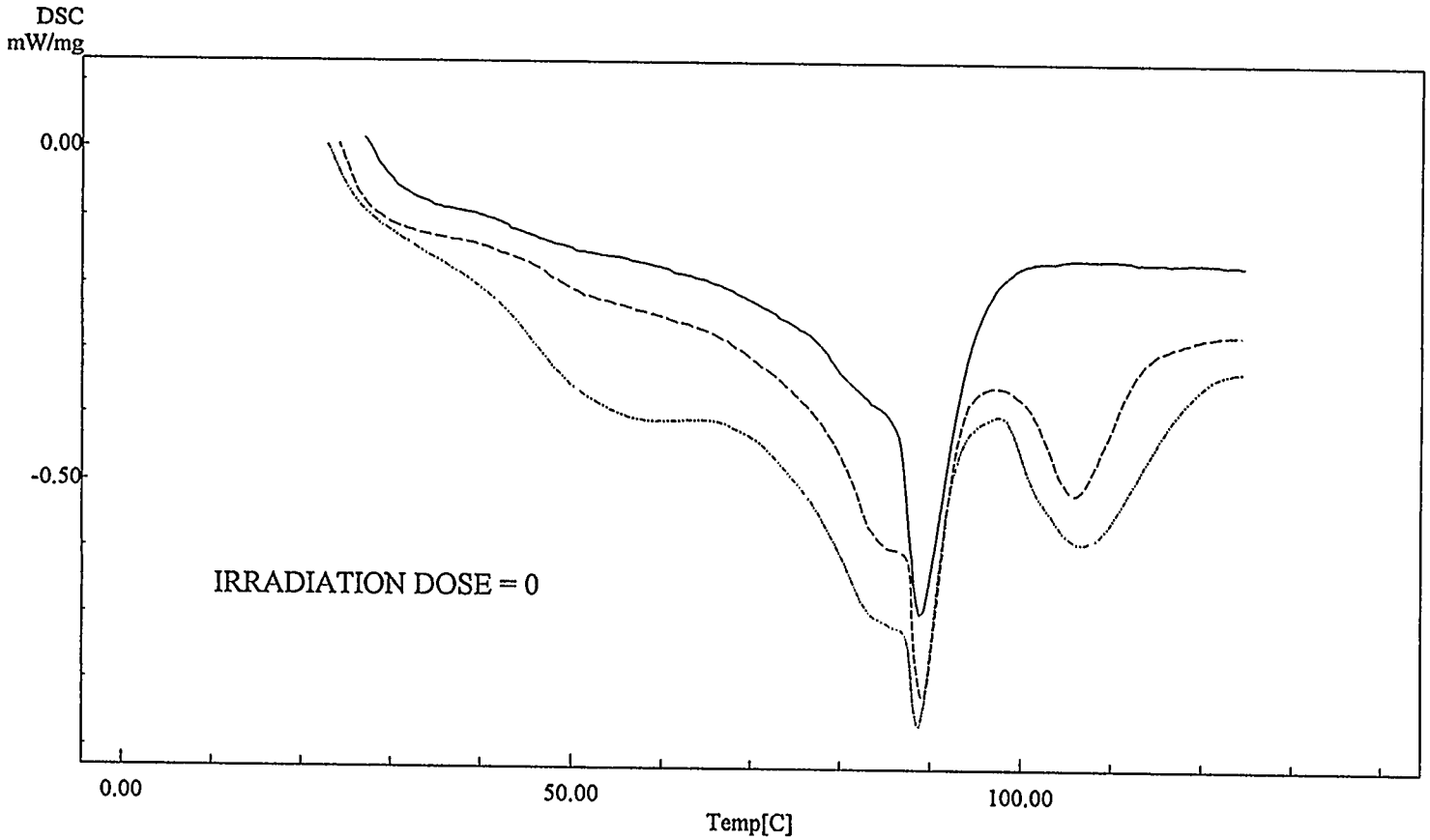

Figure 4. Differential Scanning Calorimetry Results for Unirradiated Pre-Melted Montan Wax.

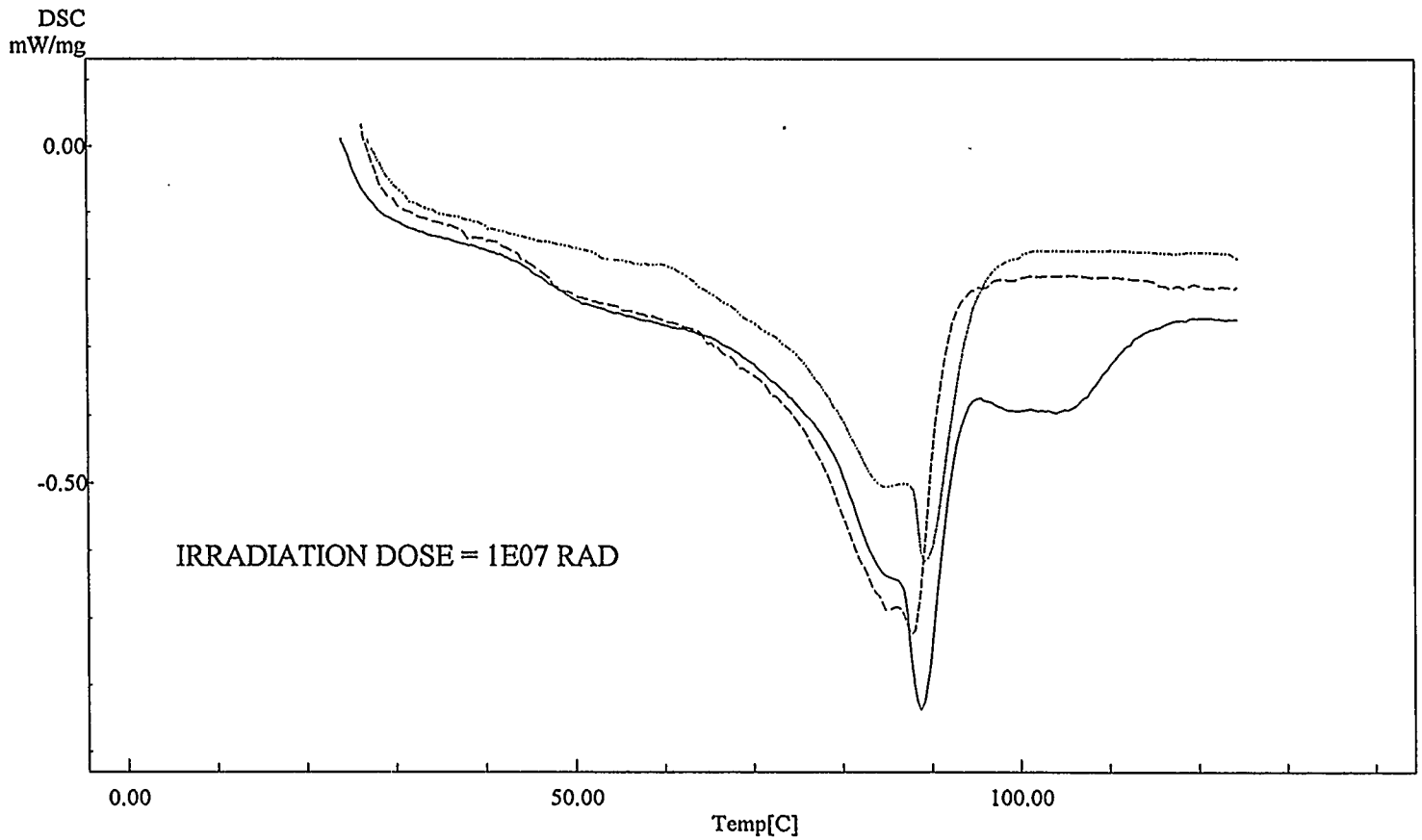

Figure 5. Differential Scanning Calorimetry Results for Pre-Melted Montan Wax Irradiated to 10^7 Rad.

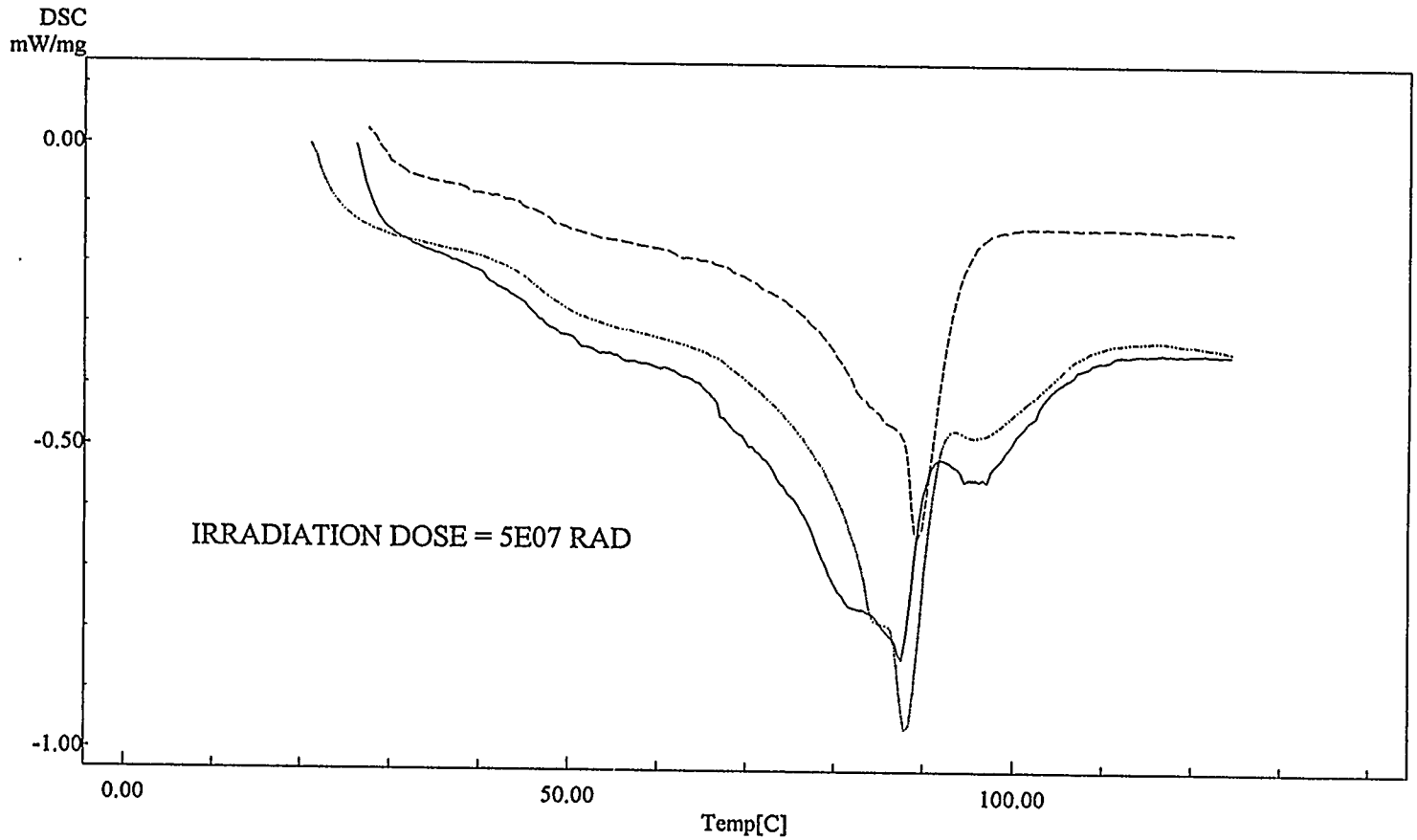

Figure 6. Differential Scanning Calorimetry Results for Pre-Melted Montan Wax Irradiated to 5 x 10^7 Rad.

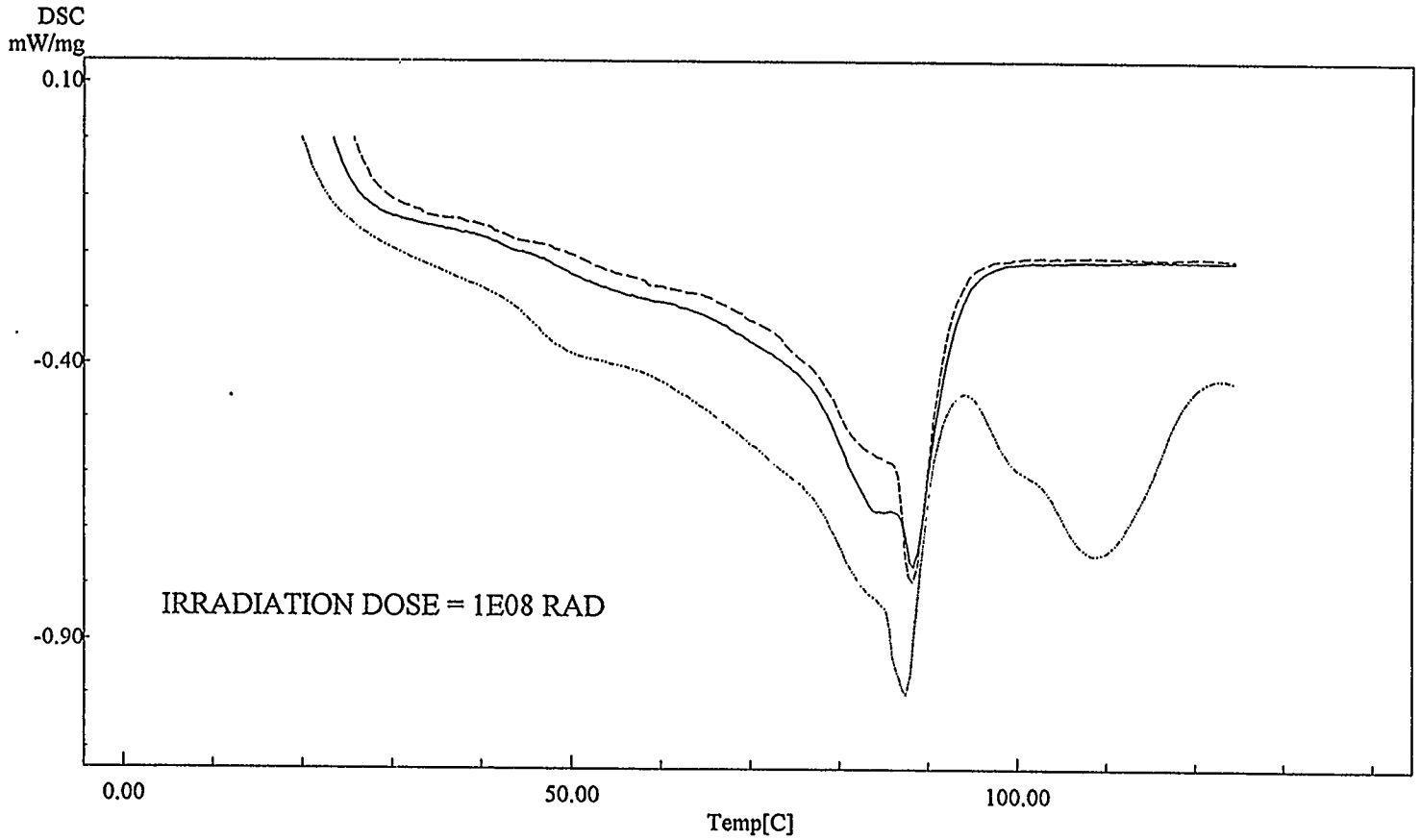

Figure 7. Differential Scanning Calorimetry Results for Pre-Melted Montan Wax Irradiated to 10^8 Rad.

It may be seen that one of the samples irradiated to 10^8 rad has a higher melting point component similar to those seen in the unirradiated material (compare Figures 4 and 7). Again this is best ascribed to crystallinity differences in the wax granules, rather than to irradiation-induced change. Another point to note is that there could be a minor effect of irradiation on the maximum heat input to the specimens during melting. Close examination of the data in Figures 3 through 7 indicates that as the dose increases, the maximum heat input increases. The most highly-irradiated samples (Fig. 7) have a maximum heat input rate approaching 0.9 mW/mg of wax. This could be a result of irradiation-induced structural changes that make the wax more resistant to melting even though the melting points do not change significantly with the irradiation dose.

3.5 Molecular Weight Determinations

Samples that had been irradiated over a wide range of doses were sent for molecular weight determination. The results allow a more direct assessment of the effects of irradiation on the wax structure. Table 3 summarizes the molecular weight determinations carried out by the American Polymer Standards Corporation in Mentor, Ohio. The analytical technique used was gel permeation chromatography. Samples were usually in the form of as-melted discs, but molecular weights were also obtained for as-received granules. Three different molecular weights were measured which are defined by the relationships given below:

$$M_n = \frac{\sum N_i M_i}{\sum N_i} \tag{1}$$

$$M_w = \frac{\sum N_i M_i^2}{\sum N_i^2} \tag{2}$$

$$M_z = \frac{\sum N_i M_i^3}{\sum N_i^3} \tag{3}$$

In these equations N_i is the number of molecules of species i with a molar mass of M_i.

Table 3. Effect of gamma radiation on the molecular weight of montan wax

Sample #	Irradiation Dose (rad)	Molecular Weight		
		M_n	M_w	M_z
1 (as rec. granules)	0	590	980	1400
4	0	440	920	1400
2	1×10^7	590	1000	1500
3	5×10^7	600	1000	1500
5	1×10^8	460	970	1500

The number averaged molecular weight (M_n) is the one most commonly cited. Irradiations up to 10^8 rad do not alter the molecular weight in a consistent manner. This result reinforces the conclusion reached above that irradiation to the doses studied here does not cause marked changes in the wax structure. This wax shows great resistance to gamma radiation. Since the molecular weight is in the 500 range it could be susceptible to biodegradation. This is discussed below in Section 3.6.

The molecular weight distribution results shown in Appendix A appear to be correlatable with the differential scanning calorimetric data in Figures 3 through 7. Specifically, the molecular weight distribution of the wax is dominated by a double peak centered at 1000. This correlates with the observed double melting point observation centered at 88^{0}C in the calorimetric data. The wax component with the smaller molecular weight peak at about 250 (log M_w=2.4) also correlates with the 50^{0}C melting point inflection that can be detected in Figures 3 through 7.

3.6 Biodegradation Testing

Biodegradation tests using the ASTM G-21 and G-22 methodologies were carried out by NAmSA of Kennesaw, GA. The G-21 test is designed to determine a polymer's resistance to a mixture of five different fungi. Test specimens in the form of the standard 47 mm discs were placed on top of a layer of nutrient-salts agar. The test surfaces were those that were in contact with the bases of petri dishes during solidification. These were facing upward during test, and were not in direct contact with the agar. A suspension of inoculant containing the mixture of fungi was then sprayed onto the agar and test sample surfaces and they were maintained over a period of 21 days at a temperature in the range 28 to 30^{0}C, at a relative humidity of 85 %. Triplicate specimens were tested for irradiation levels of 4.7×10^7 and 10^8 rad, as well as for non-irradiated material. Samples were periodically examined visually to check for the presence

of fungal growth.

Appendix B shows the results obtained from NAmSA. The NAmSA report shows that traces of fungal growth were present on the samples, i.e. less than 10 % of the test surface was affected. This represents a rating of "1" on a scale of 1 to 4, of which a rating of "4" specifies 60 % to complete coverage of the surface.

In the G-22 bacterial tests the bacterium used is Pseudomonas aeruginosa. Wax samples, in triplicate for a given irradiation condition, were placed on nutrient-salts agar that was inoculated with the bacteria. The test surfaces were the bottom surfaces of the samples that contacted the bases of the petri dishes during melting and solidification. The specimens were maintained for 21 days at 35 to 37^{0}C at a relative humidity of 85 %. The bottoms of the wax sample discs were again examined for bacterial growth. Appendix B shows the results of the NAmSA tests. No bacterial growth was observed for any irradiation level. Also, uninoculated agar on a glass plate did not show any bacterial attack. This control test was carried out to confirm that stray bacteria in the system did not give rise to a false positive result in the main tests.

It should be noted that the tests carried out are only standard scoping tests. They do not necessarily represent the possible action of microbes that exist in soils at a site where a barrier is to be emplaced. If, therefore, a barrier is to be qualified for an actual site it would be desirable to obtain representative soil samples to obtain microbial cultures that can be used for prototypic microbial degradation studies. At this time, based on the observed minor fungal attack using the ASTM G21 test, it appears that montan wax itself is relatively resistant to fungal attack. The ASTM G22 test indicates that the wax is immune to attack by bacteria.

4. CONCLUSIONS

Some preliminary work has been carried out to characterize montan wax for potential use as a major waste barrier constituent for subsurface use. The main emphasis was to quantify the wax's long-term ability to withstand radiation-induced mechanical, chemical, and microbial degradation. The study included the measurement of mechanical property changes (specifically hardness), chemical changes (molecular weight) and microbial attack as a function of gamma-irradiation doses in air. The following conclusions have been made based on results obtained to date:

a) Irradiation up to doses of 10^8 rad did not cause any consistent change in the hardness of pre-melted montan wax as measured using a standard needle penetrometer test.

b) Irradiation causes an increase in the weight of montan wax. It is probably caused by irradiation-induced oxidation. The increases are very small; about 0.07 % after an irradiation dose of 10^8 rad. The weight increase is proportional to the dose.

c) Differential scanning calorimetry was conducted to determine if irradiation caused any changes in structure that could be detected through changes in melting point. Irradiation did cause small changes in the distribution of the melting points of constituents in the wax but the overall main melting point remained at about 88°C regardless of the amount of irradiation. In a few cases, specimens showed a higher-melting-point constituent was present. There was no correlation with radiation dose. It is speculated that the constituent is associated with local differences in the amount of crystallinity in the samples that was caused by small differences in the wax melting procedure during sample preparation.

d) Gamma radiation does not cause any consistent changes in the molecular weight of the wax. It appears to have high resistance to radiation-induced structural change.

e) Using the standard ASTM G22 test there was no detectable bacterial degradation on unirradiated or irradiated wax. A small amount of fungal growth was observed using the ASTM G21 test. It is quite common to observe such fungal attack in polymeric materials since the test is, apparently, a severe scoping test. Under actual soil service conditions fungal attack of montan wax by indigenous fungi may not occur. Prototypic tests are recommended to confirm this. Based on the resistance to molecular weight decreases caused by irradiation, the wax should not suffer increased susceptibility to biodegradation as a result of long-term radiation during service.

In summary, the limited tests on montan wax show that there are no major degradation modes that would disqualify its use as a component in a subsurface waste barrier. Its low cost and resistance to mechanical, chemical, and microbial deterioration warrant an expanded evaluation of its potential applications.

5. REFERENCES

1. Golder Associates, "Laboratory Test Results for Evaluating the Performance of Montan Wax and Sodium Silicate Barrier Materials in Unconsolidated Soils," Report No. 913-1248-600a, 1993.

2. P.Soo, et al., "A Study of the Use of Cross-linked, High-density Polyethylene for Low-Level Radioactive Waste Containers," Report No. BNL-52429, Brookhaven National Laboratory, 1989.

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BROOKHAVEN NATIONAL LABORATORY
ASSOCIATED UNIVERSITIES, INC.
P. O. BOX 5000
UPTON, NY 11973 5000
ATTN: PETER SOO

ASTM G-21-90

Test Article: Montan Wax

Test Organism Mixture: *Aspergillus niger* ATCC 9642
Penicillium pinophilum ATCC 11797
Chaetomium globosum ATCC 6205
Gliocladium virens ATCC 9645
Aureobasidium pullulans ATCC 15233
Inoculum Level: 8.0×10^5 - 1.2×10^6 CFU/ml
Sample Size: 2 inch disk
Incubation Parameters: 21 days at 28°C - 30°C and 85% RH
Date Received: 9-5-95

	Results [a]		
Sample Identification	Replicate 1	Replicate 2	Replicate 3
As solidified	1	1	1
5×10^7 rad	1	1	1
10^8 rad	1	1	1

[a] Observed growth on specimens: 0 = none, 1 = traces (<10%), 2 = light (10-30%), 3 = medium (30-60%), 4 = heavy (60% - complete coverage)

ism BSP · Completed 9-28-95 Approved James R. Kautz, B.S. James R. Kautz
MG107-000, MG107-001

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BROOKHAVEN NATIONAL LABORATORY
ASSOCIATED UNIVERSITIES, INC.
P. O. BOX 5000
UPTON, NY 11973 5000
ATTN: PETER SOO

ASTM G-22 BACTERIAL

Test Article: Montan Wax

Test Organism Mixture: *Pseudomonas aeruginosa* ATCC 13388
Inoculum Level: 2.2×10^5 CFU/mL
Sample Size: 2 inch disk
Incubation Parameters: 21 days at 35-37°C and 85% Relative Humidity
Date Received: 9-5-95

	Results		
Sample Identification	Replicate 1	Replicate 2	Replicate 3
As solidified	No growth	No growth	No growth
5×10^7 rad	No growth	No growth	No growth
10^8 rad	No growth	No growth	No growth
Uninoculated control	No growth	No growth	No growth

Comment: Bacterial attack is not apparent in the form of visible growth on the specimen surface.
NG = No Growth

ism
BRP

Completed 9-29-95

Approved James R. Kautz, B.S. James R. Kautz
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PHONE (216) 255-2211 • FAX (216) 255-8397

November 21, 1995 1

Peter Soo
BrookHaven National Laboratory
Building 830
Upton, NY 11973

Dear Mr. Soo:

Enclosed are the gel permeation chromatographic results in Chloroform on your polymer samples.

Results: In Polyethylene MW Numbers

Sample	Mn	Mw	Mz	Mw/Mn
Montan Wax #1	590	980	1,400	1.66
Montan Wax #2	590	1,000	1,500	1.69
Montan Wax #3	600	1,000	1,500	1.67
Montan Wax #4	440	920	1,400	2.09
Montan Wax #5	460	970	1,500	2.11

Experimental

Columns:	AM Gel (10^3Å + 500Å + 100Å)
Solvent:	Chloroform
Temperature:	30°C
Flow Rate:	1.0 ml/min
Injection Volume:	100 μl
Detector:	M-150-C (-64/25)
Data Module:	GPC PRO 3.13 IBM AT

Comments

All samples and standards were dissolved and filtered in mobile phase using 0.45 micron filters.

Enclosed are the original GPC tracings for your inspection.

A copy of the column calibration used in assigning your sample molecular weights is enclosed along with a copy of the integration parameters.

Thank you for allowing us to be of service. Please feel free to contact me if you have any questions concerning these results or if we can be of assistance to you in the future.

Sincerely,

Ronald E Alleman

Ronald E. Alleman
Technical Department

1-3

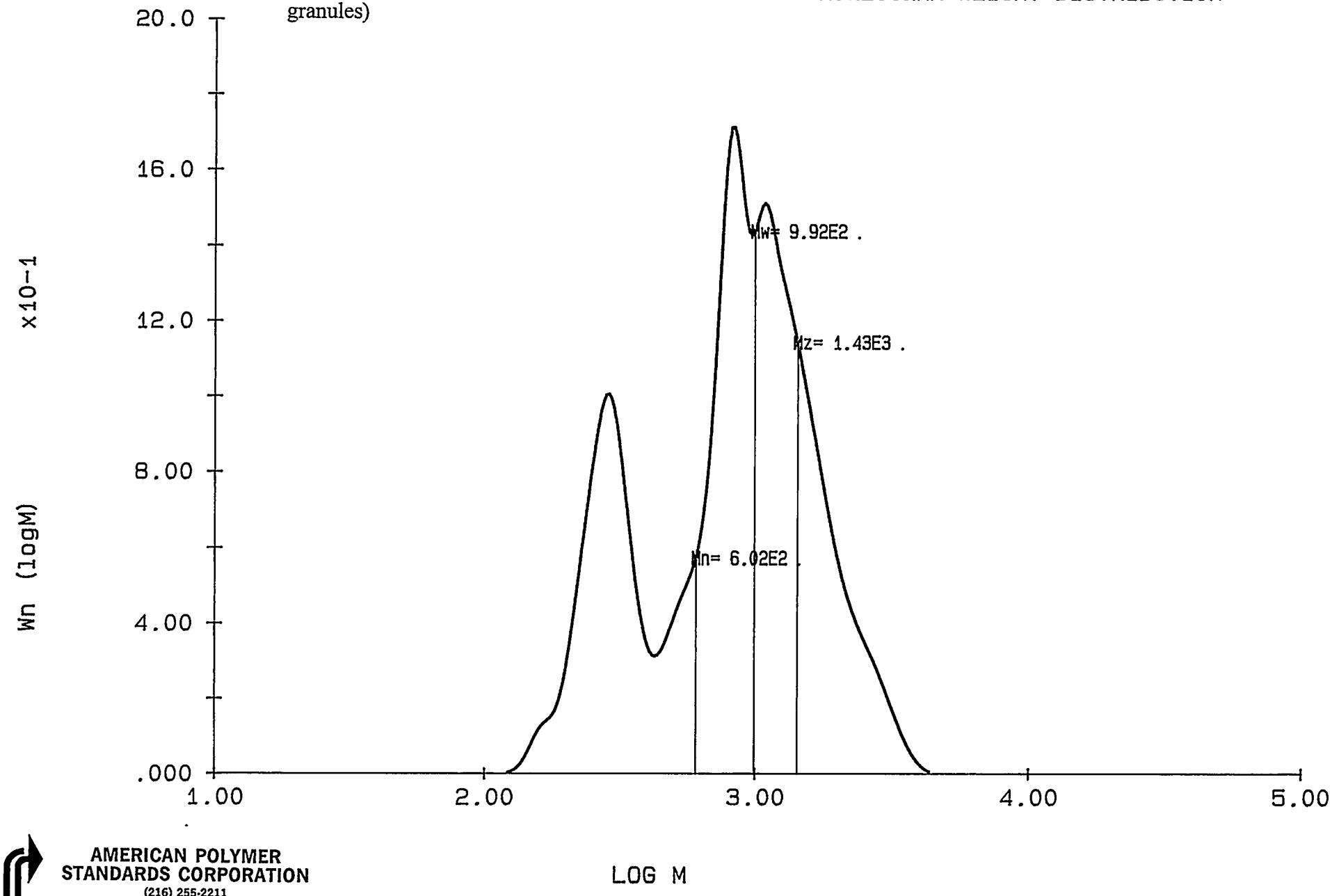
GPC-PRO 3.13

Sample: Montan Wax #1

(unirradiated as-received granules)

ENDED: 11/14/95 03:

MOLECULAR WEIGHT DISTRIBUTION



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2-1

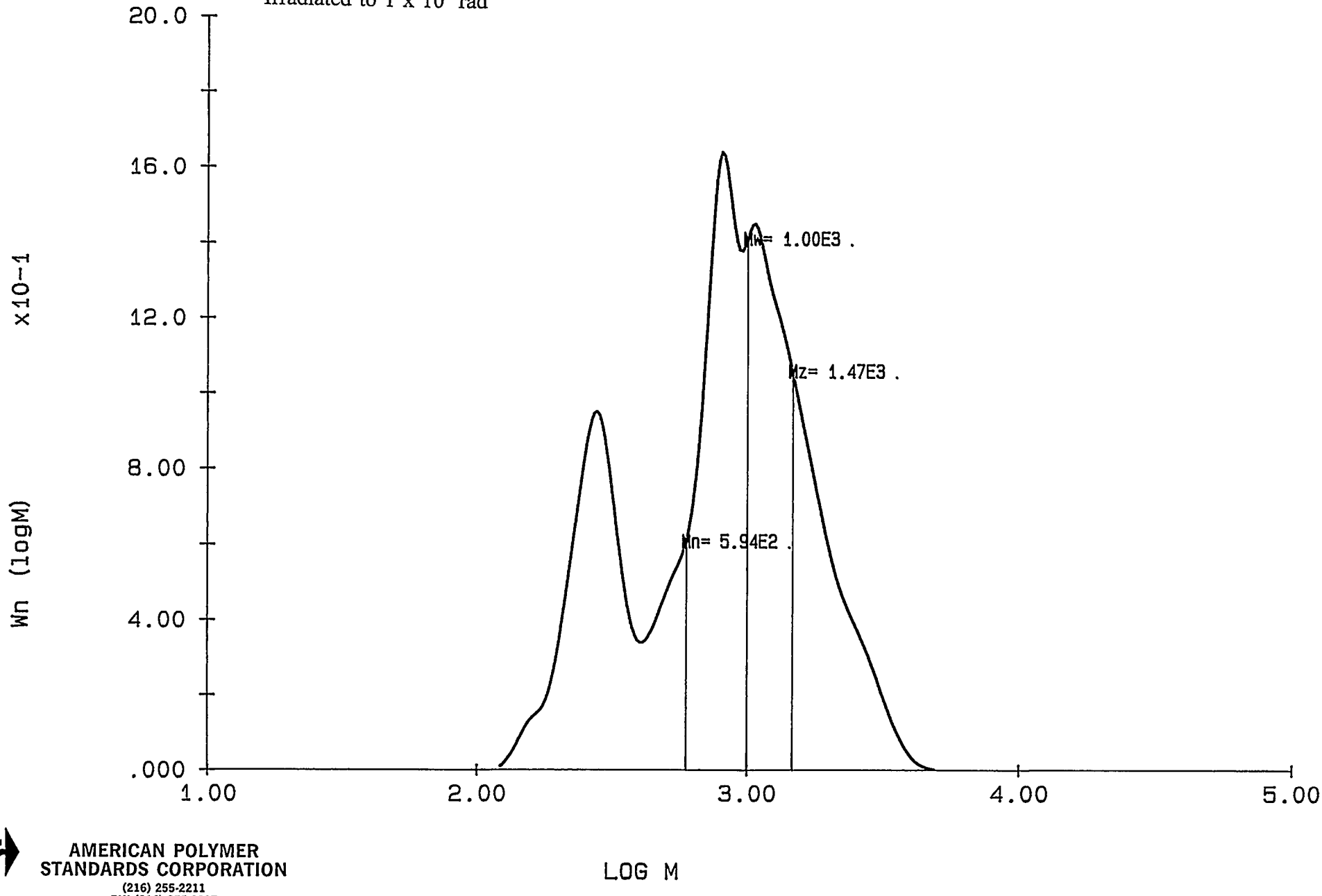
GPC-PRO 3.13

Sample: Montan Wax #2

Irradiated to 1 x 10^7 rad

ENDED: 11/14/95 13:.

MOLECULAR WEIGHT DISTRIBUTION



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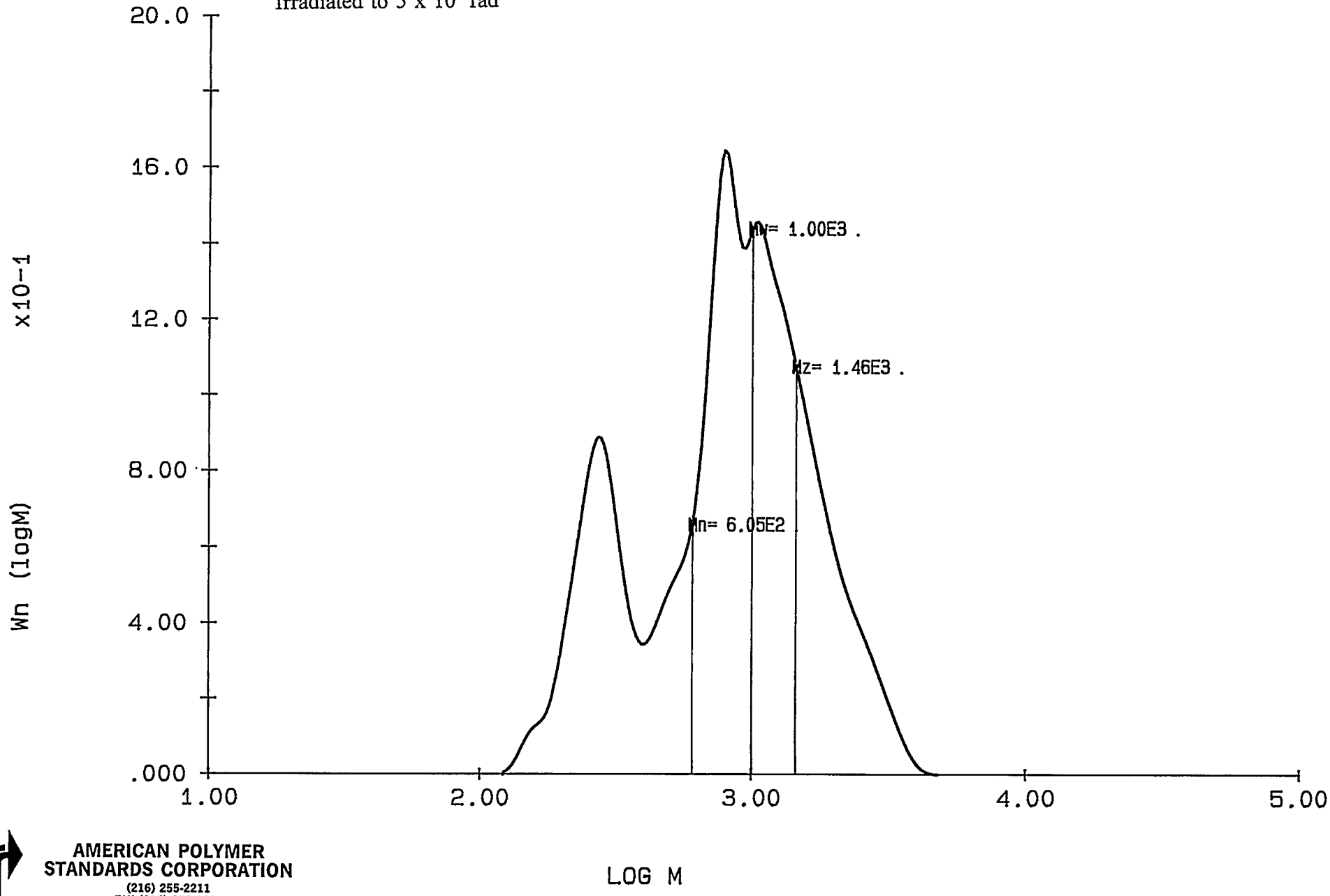
3-1 Sample: Montan Wax #3 ENDED: 11/14/95 16:.

GPC-PRO 3.13

Irradiated to 5 x 10^7 rad

MOLECULAR WEIGHT DISTRIBUTION



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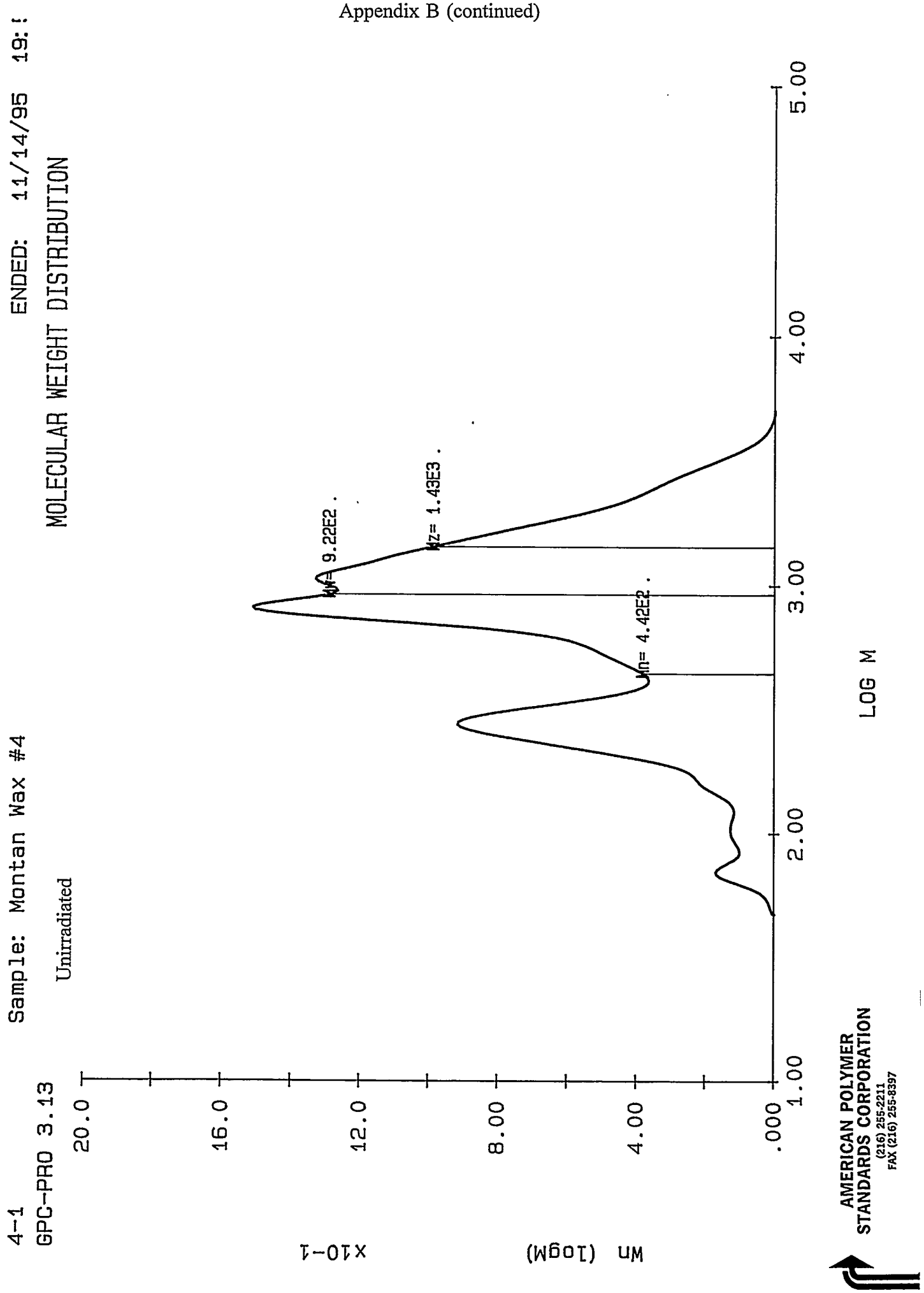
Appendix B (continued)


4-1
GPC-PRO 3.13
Sample: Montan Wax #4
Unirradiated
MOLECULAR WEIGHT DISTRIBUTION
ENDED: 11/14/95 19:
Mz= 1.43E3
Mw= 9.22E2
Mn= 4.42E2
20.0
16.0
12.0
8.00
4.00
.000
x10-1
Wn (logM)
1.00
2.00
3.00
4.00
5.00
LOG M
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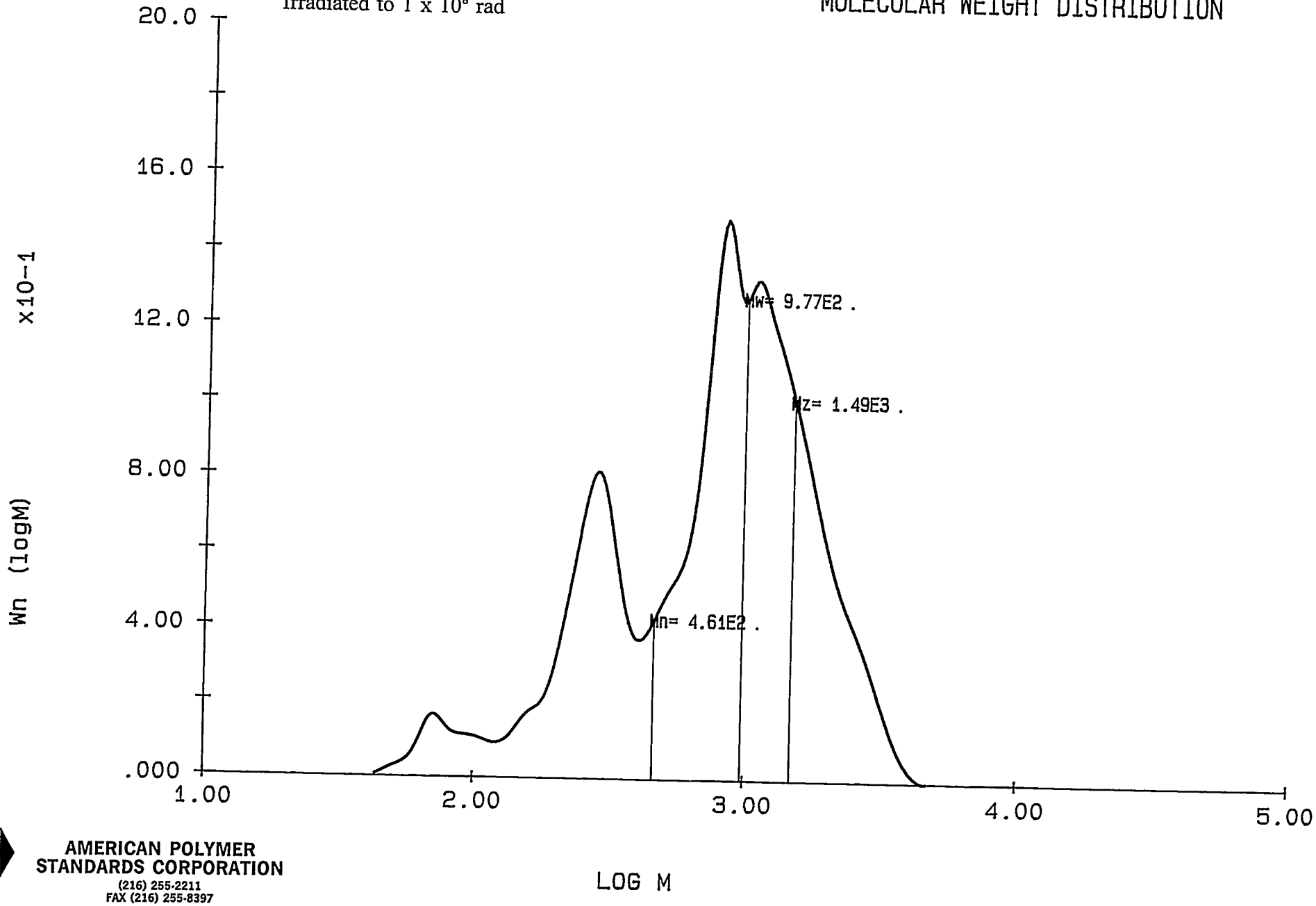
5-1
GPC-PRO 3.13

Sample: Montan Wax #5

Irradiated to 1 x 10^8 rad

ENDED: 11/14/95 21:

MOLECULAR WEIGHT DISTRIBUTION



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