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EVALUATION OF PMB PARTICULATE TESTING

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MATERIALS AND MANUFACTURING DIRECTORATE
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Researchers at the University of Dayton Research Institute (UDRI), acting on behalf of the Air Force Coating Technology Integration Office (CTIO), were asked to investigate several technical issues which had been reported regarding the specification for plastic media for the depainting of aircraft, MIL-P-85891A. The technical issues included the method for specific gravity determination, a reported incompatibility of the reagents called for in testing for heavy and light particulates, and some Type V (acrylic plastic) media meeting the composition requirements but failing to meet the 95 percent methylene chloride solubility requirement.

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Summary

Researchers at the University of Dayton Research Institute (UDRI), acting on behalf of the Air Force Coating Technology Integration Office (CTIO), were asked to investigate several technical issues which had been reported regarding the specification for plastic media for the depainting of aircraft, MIL-P-85891A. The technical issues included the method for specific gravity determination, a reported incompatibility of the reagents called for in testing for heavy and light particulates, and some Type V (acrylic plastic) media meeting the composition requirements but failing to meet the 95 percent methylene chloride solubility requirement.

It is recommended that procedure ASTM D792-66 for testing specific gravity of particulate materials be incorporated in revisions to MIL-P-85891A. Also, revisions of MIL-P-85891A should reference ASTM D788-98 for defining suitable acrylic plastics.

Introduction

The U.S. Air Force purchases plastic media for the depainting of aircraft according to specification MIL-P-85891A. The U.S. Navy, which heretofore has been the responsible agency for this specification has indicated an intent to cease its support. The Air Force has decided to take up support of this specification, update it, requalify vendors, and update the QPL. UDRI was asked to investigate several technical issues which had been reported. These include a reported incompatibility of the reagents called for in testing for heavy and light particulates, and some TYPE V (acrylic plastic) media meeting the composition requirements but failing to pass the 95% solubility requirement. Also, T. O. 1-1-8 calls out a procedure for testing for heavy particulates which differs from that in the MIL-P-85891A specification. Finally, an additional issue which became apparent was that the current MIL-P-85891A specification referenced an ASTM procedure (D792-98) for specific gravity measurement which could not be applied to a particulate material.

Executive Summary

An ASTM D792-66 procedure for testing specific gravity of particulate materials was evaluated and is recommended as the specific gravity procedure to be incorporated into revisions to MIL-P-85891A.

Revisions of MIL-P-85891A should reference ASTM D788-98 to define suitable acrylic plastics. Different grades of Type V media, and of acrylic plastic molding compounds were found to have significantly different methylene chloride solubility. The IR spectra of the materials were all characteristic of acrylic plastics. A decision is necessary as to whether methylene chloride solubility is a requirement which relates to media performance. If solubility in methylene chloride is a continuing requirement, the range of acrylic polymers will be limited to a subset of those currently in use.

The reagents for heavy and lights particulates testing called out in the current version of MIL-P-85891A are incompatible. Of the fluids tested, the recommend replacement is a blend $ZnCl_2$-water/ethanol. One fluid not tested, 3M PF-5060, may be an acceptable alternate and should be evaluated.

The procedure in T.O. 1-1-8 for heavy particulates testing differs sufficiently from that in MIL-P-85891A that it should not be used as the basis for rejection of virgin media without additional validation of the comparability of the two methods. This recommendation makes no statement regarding the method's suitability for in-process control of recycled media.

Results of Laboratory Investigations

I. Specific Gravity Testing Procedures

MIL-P-85891A calls for testing the specific gravity of plastic media according to ASTM D792-98, Test Method for Specific Gravity and Density of Plastics by Displacement. However, the current procedures in this test method are not applicable to particulate materials. The method is limited to specific gravity determination of sheets, plates, rods, etc. A discussion with Ken Clark

at Patuxent River Naval Air Station, who was involved in the initial plastic media development work revealed that the previous version of this procedure, D792-66 (reapproved in 1979) included a procedure for the specific gravity determination of particulate samples using a 25ml pycnometer. A copy of the earlier test method is attached in Appendix A.

ASTM D5965-96 provides methods for determination of the specific gravity of powder coatings. Since plastic blast media and powder coatings are of a similar physical nature, it was thought that this procedure might be applicable. The procedure describes two methods: method A uses a 250ml volumetric flask; method B is an instrumental technique using a gas pycnometer. Gas Pycnometry is also used in other ASTM procedures, including D555-94, Specific Gravity of Soil Solids by Gas Pycnometer, and D6093-97, Percent Volume Nonvolatile Matter in Clear or Pigmented Coatings Using a Helium Gas Pycnometer.

Finally, Mr. Dan Kinsinger of U.S. Technologies, a plastic media supplier, provided a copy their internal specific gravity procedure (Appendix B). He claims that all manufacturers use a similar procedure. This technique is similar to that of D792-66.

A discussion was held with a manufacturer of gas pycnometers, (Quantachrome Corp 1900 Corporate Drive, Boynton Beach FL 33426). The lowest price unit has a cost of approximately $4500. It was decided not to pursue this as a primary technique because of the equipment cost. It may still be considered as a potential "referee" method.

A series of experiments on samples of Type II and Type V media from two vendors, U.S. Technologies and Maxiblast, was run using the procedures of D792-66 and D5965-96. The objective was to determine if there were any significant differences between results of each technique, and whether there were any operational characteristics which favored either technique. The data are shown in **Table 1**.

Table 1. Evaluation of Flask vs. Pycnometer Technique for Specific Gravity Determination.

Evaluation of Flask vs Pycnometer Technique for Specific Gravity Determination				
	UST II	MAX II	UST V	MAX V
	1.492	1.411	1.17	1.177
Pycnometer	1.488	1.446	1.178	1.187
	1.482	1.432	1.173	1.17
mean	1.487	1.430	1.174	1.178
	1.496	1.451	1.181	1.175
Flask	1.435	1.484	1.188	1.193
	1.49	1.46	1.184	1.189
mean	1.474	1.465	1.184	1.186
difference	0.014	-0.035	-0.011	-0.008

A statistical analysis of the type II data showed no significant difference in the mean specific gravity between the two methods. However, the difference between the means of the two methods with the Type V media was statistically significant.

A significant difference in the two techniques is the method of ensuring that entrained air is removed. The pycnometer procedure uses a vacuum dessicator to remove air while no such

procedure is called for in the flask method. While the flask was in the water bath for temperature adjustment it was observed that vibration of the bath led to a release of entrained air. This mechanism appears to be less reliable than the use of the vacuum dessicator. It is recommended that the updated specification for plastic media incorporate the pycnometer method of specific gravity determination as contained in ASTM D792-66. Since this is an obsolete method, the actual procedure should probably be incorporated into the text of the specification.

II. Methylene Chloride Extraction Testing

MIL-P-85891A calls for testing the methylene chloride extractability of all media. For thermoset plastics, this test ensures that the material is properly polymerized. The specification ranges between 1% (Types II, III, IV, and VI) and 5%(Type I) maximum. Type V acrylic media is thermoplastic and the methylene chloride extraction specification is set at 95% minimum. Type VII, a starch acrylic hybrid has an extract specification of 5%min, and 10% max.

The government PJM indicated that vendors report that it is not uncommon that media which conforms to the composition requirements of Type V media will not pass the methylene chloride solubility specification.

This raises the issue as to whether the composition of acceptable raw material for Type V media is adequately defined by the specification.

The required composition of Type V media is defined in Par. 3.2 of the specification, which states "The finished product shall be made from chlorine free cured plastic stock of ...acrylic plastic (for Type V)..." This paragraph further states that the finished product shall contain no inorganic fillers, but may contain anti-static agents.

Paragraph 3.2.3, Sources of Plastic Stock states "The finished product shall be manufactured from selected plastic stock of the exact chemical type required by this specification." This paragraph further states that the stock shall be either virgin or scrap, and "an infrared spectrogram of the finished product shall be essentially identical to those in figures 1 through 7" (Figure 5 is the spectrogram for Type V media).

The degree to which compositions of "acrylic plastic" can vary is documented in ASTM D 788-98, "Standard Classification System for Poly(Methyl Methacrylate) (PMMA) Molding and Extrusion Compounds". There are essentially 4 basic categories of acrylic plastic; unmodified, impact modified, heat resistance modified, and other. Within each of these classes there are further designations determined by the Vicat Softening Temperature and whether the material is UV stabilized, UV transmitting, general purpose, or other. Unmodified materials are defined as "polymerized from 70-100% methacrylate monomer and 0-30% acrylic comonomers.) Impact modified materials contain "50-95% unmodified polymer and 5-50% of impact modifier(s), maintaining the requirement that the overall composition of these resins is polymers made from monomers, at least 70% of which are methyl methacrylate." Heat-resistance modified materials are "polymerized from 70 to 95% methacrylate monomer and 5 to 30% comonomers."

Because a wide range of materials can correctly be identified as "acrylic plastic" the problem appeared to lie in the definition of allowed material.

The extraction procedure in MIL-P-85891A was followed on the two samples of Type V media, one supplied from U.S. Technology, and one supplied by Maxiblast. The results were 58.7% extraction for the U.S. Technology material and 90.6% for the Maxiblast Type V media. The paper used to filter the U.S. Technologies material contained a large quantity of a white brittle material, both inside the paper and outside the paper. In addition, the filtration time was very slow, versus the Maxiblast material which filtered rapidly.

To further test the hypothesis that methylene chloride extractability varied widely between various grades of acrylic plastic, samples of acrylic molding compounds of various grades were obtained from Cyro Industries, a primary manufacturer. The results of the methylene chloride extractability test on these materials are shown in **Table 2**, below.

Table 2. Methylene Chloride Extractability of Cyro Industries Acrylic Molding Compounds.

Methylene Chloride Extractability of Cyro Industries Acrylic Molding Compounds					
Grade	**Acrylite Plus**	**Acrylite Plus**	**Acrylite**	**Acrylite**	**XT Polymer**
Code	ZK-6	ZK-P	H15-002	S10-23	375-301
Type	impact modified	impact modified	unmodified	unmodified	Acrylic based multi-polymer
Filter Rate	slow	slow-mod	Rapid	Rapid	Moderate
Filter Paper Crystals	heavy	moderate	slight	very slight	slight-moderate
Filtrate	hazy	sl haze	clear	clear	sl haze
% Extractability	50.9	74.8	99.7	92.2	45.4

One of the unmodified materials met the extractability criteria in the current specification, and another was very close. The impact modified materials and the acrylic based multipolymer material did not meet the specification. The behavior during testing, and the residue on the filter paper from the latter materials appeared similar to the sample of U.S. Technology Type V media while the unmodified materials acted much like the Maxiblast Type V.

Infrared spectrograms were run on each of the two samples of Type V media and for the Cyro Industries acrylic molding compounds by preparing 1% pellets ground with KBr. The resulting spectra were then compared to the reference spectrum for Type V media in MIL-P-85891A. The spectra are reproduced in Appendix B

The spectra of the two Type V samples are essentially identical. The small differences are attributable to sample preparation differences and differences in concentration. The two Type V samples were qualitatively similar to the reference with the exception that neither had the reference's strong OH stretch absorption peak at 3500 cm^{-1}. The spectrum of the extract from the U.S. Technology Type V media retained on the filter paper was compared to that of the unextracted media. Some qualitative differences were seen, primarily in the falloff of transmittance between 3000 cm^{-1} (C=C-H) and the overtone at 1900 cm^{-1} and the shape of the peaks between 1500 and 1700 cm^{-1} but both materials had the general characteristics of the reference spectrum.

All the Cyro Industries acrylic molding compounds spectra were qualitatively similar to the specification reference for Type V media. As with the U.S. Technology Type V media, the

spectrum of the material retained on the filter paper from the methylene chloride extract test of Acrylite Plus ZK-6 was similar to that of the parent molding compound.

From this investigation, it is apparent that the definition of allowed materials as contained in the current specification, whether by name (acrylic plastic) or qualitative comparison to a reference IR spectrum, is insufficient to ensure that material is selected which meets the methylene chloride extract requirement.

As the specification is now constituted, the requirement is for a base material to be selected which is acrylic plastic, AND which meets the extract specification. This clearly limits the selection of materials suitable for production of type V media. Whether this is an issue depends on the original purpose for the extract test. In the case of thermoset media types, the obvious purpose is to check for polymer crosslinking. In the case of Type V media, the intent appears to have been to exclude impact modified and similar acrylic compounds.

If the intent was to check for occluded materials in the polymer or contamination from other media, there is no basis in the specification for rejection of material based on the nature of small amounts of residue discovered during this test. Contamination appears to be handled adequately through other procedures.

If the requirement was included based on results with initial Type V media samples under the (mistaken) assumption that all acrylic compounds would behave similarly, then there is no technical justification for the solubility requirement for Type V and there is no reason to maintain it as part of the specification.

The testing shows that it is possible to produce Type V media which meets the extract specification by selection of a suitable acrylic compound. Based on the samples of commercial media, the enforcement of this specification may affect the raw material procurement practices of some vendors.

A review of reports generated or discussions with persons involved with the development of Type V media may shed some light on the rationale for the requirement. If there is not a performance basis for the solubility requirement, the specification can be revised. If there is a basis for the requirement, then future qualification, quality control, and first article testing should reject material which does not comply. It is recommended that to maintain future integrity, that the specification reference allowable materials for Type V media based on the use of the classes of acrylic polymers in ASTM D788-98.

III. Heavy and Lights Particulate Testing

MIL-P-85891A calls for the testing of plastic media for heavy and light particulates by using reagents having a heavier or lighter specific gravity than the bulk media. The original specification called for the use of hydrocarbon solvent meeting Federal Specification PD-680 as the light reagent, and trichlorotrifluoroethane (TCTFE, Refrigerant 113) as the heavy reagent. By mixing these two materials in the proper proportions, mixtures having the appropriate specific gravity for each media type could be prepared. Revision A of the specification, dated 1 April 1992, called for reagent specific gravities 0.1 point higher or lower than the specific

gravity of the material to be tested. It was unclear whether the specific gravity reference point was the particular lot under test, or the midpoint, minimum, or maximum of the specific gravity range of the particular media. Amendment 2, dated 26 June 1998 provides clarification of this latter point, requiring the heavy solution to have a specific gravity of 1.40 for media types I, V, and VI and the light solution to have a specific gravity of 0.10 less than the minimum allowed specific gravity of the finished product. The reagent composition was also changed, substituting 3M fluorocarbon FC-40 in place of TCTFE. The requirements are summarized below in **Table 3**.

Table 3. MIL-P-85891A Particulate Testing Requirements.

MIL-P-85891A Particulate Testing Requirements							
Type	**I**	**II**	**III**	**IV**	**V**	**VI**	**VII**
Specific Gravity Specifications							
min	1.15	1.47	1.47	1.47	1.10	1.28	1.38
max	1.25	1.52	1.52	1.52	1.20	1.33	1.43
Particulates Specification, % by Weight, Max.							
heavies	0.02	0.02	0.02	0.02	0.02	0.02	0.02
lights	0.10	1.00	1.00	1.00	0.10	0.10	1.00
Specific Gravity of Test Reagents							
heavies	1.40	1.62	1.62	1.62	1.40	1.40	1.62
lights	1.05	1.37	1.37	1.37	1.00	1.18	1.28

The government PJM reported that vendors, as well as the laboratory providing first article testing, indicated that the reagents specified, (FC-40 and PD-680) were incompatible (immiscible) and could not be used for testing. Vendors also provided recommendations for alternate reagents based on their internal practices. These included:

- 85% w/v KBr in water/ethanol
- ZnCl in water/ethanol
- Perchlorethylene/ethanol

Samples of TCTFE, FC-40, PD-680 and the vendor recommended reagents were obtained, evaluated for compatibility, and where possible, used to conduct heavy and lights particulate testing of plastic media samples.

It was determined that TCTFE and PD-680, the original specification reagents, were compatible in all proportions. It was also confirmed that FC-40 and PD-680 were incompatible in all proportions. As a side experiment, hexane and toluene were tried unsuccessfully as third components to see if they could effect compatability. Also, an alternate fluorocarbon, 3M FC-5311, was tried. It too was incompatible with PD-680. Mixtures with toluene and methylene chloride were also incompatible with FC-5311. Limited compatibility was seen with FC-5311 and Hexane (1/10) but the density range was too limited to be of use. The detailed data is shown below in **Table 4**.

Table 4. Fluorocarbon Reagent Compatibility Data.

Fluorocarbon Reagent Compatibility Data				
		R1/R2 volume ratio		
R1	R2	90/10	50/50	10/90
TCTFE	PD-680	compatible	compatible	compatible
FC-40	PD-680	incompatible	incompatible	incompatible
FC-5311	PD-680	incompatible	incompatible	incompatible
	Three Way Mixtures With Toluene			
	FC-5311	PD-680	Toluene	
	50	50	10	incompatible
	FC-40	PD-680	Toluene	
	50	50	10	incompatible
	Hexane Compatibility			
			Volume of each layer, ml	
	ml Hexane	ml FC-5311	upper	lower
	10	10	10	10
	10	5	3	10
	10	2.5	0.5	10
	10	1	compatible	

Other reagents examined for specific gravity (**Table 5**) included KI-Water/ethanol, perchlorethylene/ethanol (reagents used by U.S. Technologies), and $ZnCl_2$/water/ethanol.

All of these reagents achieved the heavy specific gravity required (1.62). However, the KI-Water/ethanol combination could not be used for the lower specific gravity (1.0) reagent because of limited solubility of KI-water in ethanol. Theoretically is should be possible to use water for the low specific gravity reagent. However, water's surface tension is too high to effectively wet the media. A possibility which was not evaluated is the use of a small amount of a surface active agent with water.

Table 5. Other Regents Examined for Specific Gravity.

KI/water	Ethanol	Sp. Gr.	Perchlorethylene	Ethanol	Sp. Gr.	ZnCl/water	Ethanol	Sp. Gr.
85g/100ml						85g/100ml		
100	0	1.602	100	0	1.6115	100	0	1.612
25	75	ppt.*	25	75	0.998	60	40	1.297
						25	75	1.020
* a precipitate formed which would not dissolve.								

Table 6 shows the results of heavy and lights particulates testing with various reagents. The testing procedure generally followed that of MIL-P-85891. In the case of the KI and $ZnCl_2$ reagents, the procedure was modified to include a deionized water rinse of the 200 mesh screen prior to drying. This was to eliminate the potential for retained salts affecting the method.

Table 6. Results of Heavy and Light Particulates Testing with Various Regents.

Heavies Particulate Testing				
Reagents	**Ratio**	**Reagent Sp. Gr.**	**Media**	**% Heavies**
TCTFE/PD-680	80/20	1.403	UST Type V	0.042
perc/ethanol	62/38	1.299	UST Type V	0.023
perc/ethanol	75/25	1.408	UST Type V	0.010
ZnCl-Water/Ethanol	75/25	1.431	UST Type V	0.019
KI-Water/Ethanol	75/25	1.403	UST Type V	0.015
perc/ethanol	75/25	1.408	Maxiblast Type V	0.033
Lights Particulate Testing				
Reagents	**Ratio**	**Reagent Sp. Gr.**	**Media**	**% Lights**
TCTFE/PD-680	72/28	1.338	UST Type II	0.018
perc/ethanol	70/30	1.368	UST Type II	0.098
ZnCl-Water/Ethanol	70/30	1.345	UST Type II	0.196
TCTFE/PD-680	25/75	0.977	UST Type V	0.019
perc/ethanol	25/75	1.006	UST Type V	0.011
ZnCl-Water/Ethanol	25/75	1.017	UST Type V	0.019
perc/ethanol	25/75	1.006	Maxiblast Type V	0.010

During the heavies particulate testing of Type V media using perchlorethylene/ethanol at a 75/25 ratio, a sticky residue with a polymeric appearance was found in the separatory funnel. Methylene chloride was required to effect its removal. It was hypothesized that the media might be slightly soluble in the test reagent. The filtrate was collected and weighed into a tared 100ml beaker, and evaporated 4 hr @ 60 deg. C, followed by 1 hr @ 105 deg. C. The beaker was reweighed and showed a weight gain of 1.167grams. Based on an initial sample weight of 100 grams charged into the separatory funnel for testing, this represents over a 1% solubility of the material in the test reagent. This results raises questions as to the suitability of this reagent combination as for particulate testing of Type V media. It would seem that a more inert reagent should be used. By process of elimination, this leaves the $ZnCl_2$-Water reagent as the preferred choice. This material is compatible with ethanol in the proportions necessary and is inexpensive and readily available. In addition to its expense, KI's limited solubility with a light solution limits its utility.

IV. T.O. 1-1-8 Procedure Evaluation

T.O. 1-1-8, Par. 2-16 contains process instructions for "Finish System Removal by Plastic Media Blasting (PMB) Method." Paragraph d of this section provides a procedure for testing the heavies contamination level of plastic media. Part 2 provides sampling instructions for a) used media and b) new media, implying that the procedure should be used for incoming quality assurance testing of new material as well as process control for used and recycled media.

The proposed use of the procedure in T.O. 1-1-8 as a quality assurance test for incoming new media is a cause for concern. The specification is so stringent, .02%, that variances due to the different procedures could result in controversies between vendors using the specification procedure, and a receiving location following the procedure in T. O. 1-1-8.

Although the procedure in T.O.1-1-8 generally follows the procedure in MIL-P85891, There are several exceptions, as follows:

a) The reagent called for is a mixture by volume of 95 % 3M Fluorocarbon PF-5060, 5% hexane. The specified sp. gr. of the mixture is between 1.60 and 1.66, as measured with a hydrometer (temperature unspecified), and the same mixture is used for all media types.
b) The T.O. procedure calls for adding approx. 300-350 ml of media to the 500 ml separatory funnel whereas MIL-P-85891A calls for a sample size of 100g, which is approximately 150 ml of Type V media, and less for other types.
c) Multiple extractions are called for by the T.O. 1-1-8 procedure, repeating the process as many times as necessary until no heavy particles are separated.
d) The media and heavy particles are filtered through a filter paper rather than a 200 mesh screen. The filter paper is dried to constant weight, and the residue scraped off with a metal spatula (not a brush) onto a tared watch glass, and weighed. The procedure states "Fine particles or dust may have impregnated the filter paper. This residue is not a major concern and may be disregarded because fine particles (less than 80 mesh, US Standard Sieve) are not damaging to aircraft materials or structure."

To test the potential for disputes, a heavy particulates analysis was conducted using two samples of UST type V media and the original reagent (TCTFE/PD-680). One test was run through a filter paper, the other through a 200-mesh screen as specified in MIL-P-85891A. The screened sample had a residue of .042%, while the sample put through the filter paper had a residue of 0.117%. The filtrate through the screen was cloudy, while the filtrate through the filter paper was crystal clear. In both cases, a significant portion of the residue collected was a white powder, similar to that seen during the methylene chloride extraction tests of the same media. At least for Type V media, this indicates the potential for disputes arising from the use of procedures. These differences may also depend on the polymer type and source, bringing up again the need to resolve this issue. Failure of virgin media to meet the heavy particulates specification when tested by the method in T.O. 1-1-8 is not a valid basis for rejection.

The T.O. 1-1-8 procedure may be appropriate for process control and evaluation of recycled media for reuse. The stated heavy particulate requirements for recycled media are the same as for virgin media. Although this does not seem practical, the determination of the appropriate process control limits for heavy particulates was not within the scope of the project and was not investigated.

V. Conclusions and Recommendations

Based on the investigations conducted, the following recommendations are made for incorporation into a revised MIL-P-85891 specification:

a) The revised specification should adopt the procedure contained in ASTM D792-66 for specific gravity of particulates. The text of this procedure should be incorporated into the specification.

b) There is a significant difference in methylene chloride solubility of different grades of acrylic plastics. Methylene chloride solubility appears to be very useful in the selection of

unmodified acrylic polymers. If modified polymers are acceptable, the specification should be rewritten to remove the solubility requirement. A possible requirement for procurement is to require the vendor to identify the polymer type by reference to ASTM D788-98.

c) The reagents called for heavy and lights particulate testing in the current specification are incompatible. A $ZnCl_2$-water/ethanol blend appears to be the most preferable reagent of those tested. KI-water/ethanol was incompatible at the lower specific gravity. Type V media was partially soluble in the Perchlorethylene/ethanol blend. Although no experiments were conducted using 3M PF-5060 (the reagent called for in T.O. 1-1-8). this material should be considered as a candidate reagent. Its specific gravity of 1.68 is in the right range, and according to 3M literature, it is miscible in all proportions with hexane. Although the high volatility of hexane may not make it suitable as the light particulates solvent, the PF-5060 may be compatible with the similar but less volatile PD-680.

d) The procedure in T.O. 1-1-8 for testing of heavy particulates is sufficiently different from that in MIL-P-85891A that it should not be used as the basis for acceptance or rejection of virgin material.

Appendix A
ADTM D792-66

Designation: D 792 – 66 (Reapproved 1979)[ε1]

An American National Standard

Standard Test Methods for SPECIFIC GRAVITY AND DENSITY OF PLASTICS BY DISPLACEMENT[1]

This standard is issued under the fixed designation D 792; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

These test methods have been approved for use by agencies of the Department of Defense to replace method 5011 and 5012 of Federal Test Method Standard 406 and 14011 and 14021 of FTMS 601, and for listing in the DoD Index of Specifications and Standards.

[ε1] NOTE—Section 2 was added editorially and subsequent sections renumbered in August 1985.

1. Scope

1.1 These test methods cover the determination of the specific gravity and density of solid plastics by displacement of liquid and determination of the change in weight.

1.2 These test methods differ in applicability as follows:

1.2.1 *Methods A-1, 2, 3*—For plastics in forms such as sheets, rods, tubes, or molded items. Nondestructive testing of large items is covered. These methods also may be used to characterize a molding or casting compound by testing a specimen that has been prepared under controlled conditions.

1.2.2 *Method B*—For plastics in forms such as molding powder, flake, or pellets.

NOTE 1—Alternatively, Test Method D 1505, may be applied to many such forms, as well as to films and sheeting.

1.3 The values stated in SI units are to be regarded as the standard.

2. Applicable Documents

2.1 *ASTM Standards:*

D 618 Method for Conditioning of Plastics and Electrical Insulating Materials[2]

D 891 Test Methods for Specific Gravity, Apparent, of Liquid Industrial Chemicals[3]

D 1505 Test Method for Density of Plastics by the Density-Gradient Technique[2]

D 1622 Test Method for Apparent Density of Rigid Cellular Plastics[4]

D 1898 Practice for Sampling of Plastics[4]

E 1 Specifications for ASTM Thermometers[5]

E 12 Definitions of Terms Relating to Density and Specific Gravity of Solids, Liquids, and Gases[6]

3. Definitions

3.1 *specific gravity*—the ratio of the weight in air of a unit volume of the impermeable portion of the material at 23°C (73.4°F) to the weight in air of equal density of an equal volume of gas-free distilled water at the same temperature. The form of expression shall be:

Specific gravity 23/23°C (or sp gr 23/23°C)

NOTE 2—These definitions are essentially equivalent to the definitions of apparent specific gravity and apparent density in Definitions E 12, because the small percentage difference introduced by not correcting for the buoyancy of air is insignificant for most purposes.

3.2 *density*—the weight in air in grams per cubic centimeter of impermeable portion of the material at 23°C. The form of expression shall be:

D^{23}, g/cm^3 (Notes 2, 3, 4)

[1] These test methods are under the jurisdiction of ASTM Committee D-20 on Plastics and are the direct responsibility of Subcommittee D20.70 on Analytical Methods (Section D20.70.01).

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[2] *Annual Book of ASTM Standards*, Vol 08.01.

[3] *Annual Book of ASTM Standards*, Vol 15.05.

[4] *Annual Book of ASTM Standards*, Vol 08.02.

[5] *Annual Book of ASTM Standards*, Vols 05.03 and 14.01.

[6] *Annual Book of ASTM Standards*, Vols 04.02 and 15.05.

NOTE 3—Densities in other units may be obtained, when desired, by multiplying by appropriate factors.

NOTE 4—Specific gravity 23/23°C can be converted to density 23°C, g/cm³, by use of the following equation:

$$D^{23°C},\ g/cm^3 = \text{sp gr } 23/23°C \times 0.9975$$

4. Significance and Use

4.1 The specific gravity or density of a solid is a property that can be measured conveniently to identify a material, to follow physical changes in a sample, to indicate degree of uniformity among different sampling units or specimens, or to indicate the average density of a large item.

4.2 Changes in density of a single specimen may be due to changes in crystallinity, loss of plasticizer, absorption of solvent, or to other causes. Portions of a sample may differ in density because of difference in crystallinity, thermal history, porosity, and composition (types or proportions of resin, plasticizer, pigment, or filler).

NOTE 5—Reference is made to Test Method D 1622.

4.3 Density is useful for calculating strength-weight and cost-weight ratios.

5. Sampling

5.1 The sampling units used for the determination of specific gravity shall be representative of the quantity of product for which the data are required, in accordance with Practice D 1898. Particular attention shall be given to the following:

5.1.1 If it is known or suspected that the sample consists of two or more layers or sections having different specific gravities, either complete finished parts or complete cross sections of the parts or shapes shall be used as the specimens, or separate specimens shall be taken and tested from each layer.

5.1.2 In Method B, if it is known or suspected that fine and coarse particles may have different specific gravities, either the specimens shall contain the same proportions of each size as present in the bulk of the sample, or the sample shall be sieved into several size groups, each size group shall be tested separately, and the weighed average shall be calculated and reported.

6. Conditioning

6.1 *Conditioning*—Condition the test specimens at 23 ± 2°C (73.4 ± 3.6°F) and 50 ± 5 % relative humidity for not less than 40 h prior to test in accordance with Procedure A of Method D618, for those tests where conditioning is required. In cases of disagreement, the tolerances shall be 1°C (1.8°F) and ±2 % relative humidity.

6.2 *Test Conditions*—Conduct tests in the Standard Laboratory Atmosphere of 23 ± 2°C (73.4 ± 3.6°F) and 50 ± 5 % relative humidity, unless otherwise specified in the test methods or in this specification. In cases of disagreement, the tolerances shall be 1°C (1.8°F) and ±2 % relative humidity.

NOTE 6—Certain ASTM specifications for plastic materials that achieve moisture equilibrium very slowly call for the testing of such materials in the "bone dry" condition. Since molding powders or flake are rarely used in the Standard Laboratory Atmosphere of Methods D618, the specific gravity or density of such materials may be determined under any condition considered desirable: as received, conditioned under standard conditioning procedures, or dry, etc., provided that the condition is stated. If conditioning includes immersion in a liquid, the time and temperature shall be stated.

METHOD A-1 FOR TESTING SOLID PLASTICS IN WATER (SPECIMENS 1 TO 50 g)

7. Scope

7.1 This method involves weighing a one-piece specimen of 1 to 50 g in water, using a sinker with plastics that are lighter than water. This method is suitable for plastics that are wet by, but otherwise not affected by water.

8. Apparatus

8.1 *Analytical Balance*—A balance with a precision within 0.1 mg, accuracy within 0.05 % relative (that is, 0.05 % of the weight of the specimen in air), and equipped with a stationary support for the immersion vessel above the balance pan ("pan straddle").

NOTE 7—Assurance that the balance meets the performance requirements should be provided by frequent checks on adjustments of zero point and sensitivity and by periodic calibration for absolute accuracy, using standard weights.

8.2 *Wire*—A corrosion-resistant wire for suspending the specimen.

8.3 *Sinker*—A sinker for use with specimens of plastics that have specific gravities less than 1.000. The sinker shall: (*1*) be corrosion-resistant; (*2*) have a specific gravity of not less than 7.0; (*3*) have smooth surfaces and a regular shape; and (*4*) be slightly heavier than necessary to sink the specimen. The sinker should have an opening to facilitate attachment to the specimen and wire.

8.4 *Immersion Vessel*—A beaker or other

wide-mouthed vessel for holding the water and immersed specimen.

8.5 *Thermometer*—A thermometer with an accuracy of ±1°C (±2°F) is required if the test is not performed in the Standard Laboratory Atmosphere of Methods D618, Conditioning Plastics and Electrical Insulating Materials for Testing[4] (compare 15.4).

9. Materials

9.1 *Water*—The water shall be substantially air-free, distilled, or demineralized water.

NOTE 8—Water may be rendered substantially air-free by boiling and cooling or by shaking under vacuum in a heavy-walled vacuum flask. (**Caution**—Use gloves and shielding.) If the water does not wet the specimen, a few drops of a wetting agent shall be added. If this solution does not wet the specimen, Method A-2 shall be used.

10. Test Specimens

10.1 The test specimen shall be a single piece of the material under test of any size and shape that can conveniently be prepared and tested, provided that its volume shall be not less than 1 cm^3 (0.06 $in.^3$), and its surface and edges shall be made smooth. The thickness of the specimen should be at least 1 mm (0.04 in.) for each 1 g of weight. A specimen weighing 1 to 5 g usually will be found convenient, but specimens up to approximately 50 g may be used (Note 9). Care should be taken in cutting specimens to avoid changes in density resulting from compressive stresses or frictional heating.

NOTE 9—Specifications for certain plastics require a particular method of specimen preparation and should be consulted if applicable.

10.2 The specimen shall be free from oil, grease, and other foreign matter.

11. Procedure

11.1 Weigh the specimen in air to the nearest 0.1 mg or 0.05 % relative, whichever is greater.

11.2 Attach to the balance a piece of fine wire sufficiently long to reach from the hook above the pan to the support for the immersion vessel. Attach the specimen to the wire such that it is suspended about 2.5 cm (1 in.) above the vessel support.

NOTE 10—The specimen may be weighed in air after hanging from the wire. In this case, record the weight of the specimen, a = (weight of specimen + wire, in air) − (weight of wire in air).

11.3 Mount the immersion vessel on the support, and completely immerse the suspended specimen (and sinkers, if used) in water (9.1) at a temperature of 23 ± 2°C. The vessel must not touch wire or specimen. Remove any bubbles adhering to the specimen, wire, or sinker, paying particular attention to holes in the specimen and sinker. Usually these bubbles can be removed by rubbing them with another wire. If the bubbles cannot be removed by this method or if bubbles are continuously formed (as from dissolved gases), the use of vacuum is recommended (Note 12). Weigh the suspended specimen to the required precision (11.1) (Note 11). Record this weight as b (the weight of the specimen, sinker, if used, and the partially immersed wire in liquid). Unless otherwise specified, weigh rapidly in order to minimize absorption of water by the specimen.

NOTE 11—It may be necessary to change the sensitivity adjustment of the balance to overcome the damping effect of the immersed specimen.

NOTE 12—Some specimens may contain absorbed or dissolved gases, or irregularities which tend to trap air bubbles; any of these may affect the density values obtained. In such cases, the immersed specimen may be subjected to vacuum in a separate vessel until evolution of bubbles has substantially ceased before weighing (see Method B). It must also be demonstrated that the use of this technique leads to results of the required degree of precision.

11.4 Weigh the wire (and sinker, if used) in water with immersion to the same depth as used in the previous step (Notes 13 and 14). Record this height as w (weight of the wire in liquid).

NOTE 13—It is convenient to mark the level of immersion by means of a shallow notch filed in the wire. The finer the wire, the greater the tolerance which may be permitted in adjusting the level of immersion between weighings. With wire Awg No. 36 or finer, disregard its degrees of immersion and, if no sinker is used, use the weight of the wire in air as w.

NOTE 14—If the wire is left attached to the balance arm during a series of determinations, the weight a may be determined either with the aid of a tare on the other arm of the balance or as in Note 12. In such cases, care must be taken that the change of weight of the wire (for example, from visible water) between readings does not exceed the desired precision.

11.5 Repeat the procedure for the required number of specimens.

12. Calculations

12.1 Calculate the specific gravity of the plastic as follows:

$$\text{Sp gr } 23/23°C = a/(a + w - b)$$

where:

a = apparent weight of specimen, without wire or sinker, in air,

b = apparent weight of specimen (and of sinker, if used) completely immersed and of the wire partially immersed in liquid, and

w = apparent weight of totally immersed sinker (if used) and of partially immersed wire.

12.2 Calculate the density of the plastic as follows:

$$D^{23}, \text{g/cm}^3 = \text{sp gr } 23/23°C \times 0.9975$$

13. Report

13.1 The report shall include the following:

13.1.1 Complete identification of the material or product tested, including method of specimen preparation and conditioning.

13.1.2 Average specific gravity for all specimens from a sampling unit, reported as sp gr 23/23°C = _____, or average density reported as D^{23} = _____ g/cm³.

13.1.3 A measure of the degree of variation of specific gravity or density within the sampling unit such as the standard deviation and number of determinations on a homogeneous material or the averages plus these measures of dispersion on different layers or areas of a nonhomogeneous product.

13.1.4 Any evidence of porosity of the material or specimen.

13.1.5 The method of test (Method A-1 of Methods D 792), and

13.1.6 Date of test.

METHOD A-2 FOR TESTING SOLID PLASTICS IN LIQUIDS OTHER THAN WATER (SPECIMENS 1 TO 50 g)

14. Scope

14.1 Method A-2 uses a liquid other than water for testing one-piece specimens, 1 to 50 g, of plastics that are affected by water or which are lighter than water.

15. Apparatus

15.1 The apparatus shall include the balance, wire, and immersion vessel of Section 8, and optionally, the following:

15.2 *Pycnometer with Thermometer*—A 25-mL specific gravity bottle with thermometer, or

15.3 *Pycnometer*—A pycnometer of the Weld type, preferably with a capacity of about 25 mL, and an external cap over the stopper.

15.4 *Thermometer*—A thermometer having not fewer than four divisions per °C or two divisions per °F over a temperature range of not less than 5°C or 10°F above and below the standard temperature, and having an ice point for calibration. A thermometer short enough to be handled inside the balance case will be found convenient. ASTM Thermometer 23C (see Specification E 1) and Anschutz-type thermometers have been found satisfactory for this purpose.

15.5 *Constant-Temperature Bath*—An appropriate constant-temperature bath adjusted to maintain a temperature of 23 ± 0.1°C.

16. Materials

16.1 *Immersion Liquid*—The liquid used shall not dissolve, swell, or otherwise affect the specimen, but should wet it and should have a specific gravity less than that of the specimen. In addition, the immersion liquid should be nonhygroscopic, have a low vapor pressure, a low viscosity, and a high flash point, and should leave little or no waxy or tarry residue on evaporation. A narrow cut distilled from kerosine meets these requirements for many plastics. The specific gravity 23/23°C of the immersion liquid shall be determined shortly before and after each use in this method to a precision of at least 0.1 % relative, unless it has been established experimentally in the particular application that a lesser frequency of determination can be used to assure the desired precision.

NOTE 15—For the determination of the specific gravity of the liquid, the use of a standard plummet of known volume (Note 16) or of Method A, C, or D of Test Methods D 891, using the modifications required to give specific gravity 23/23°C instead of specific gravity 60/60°F, is recommended. One suggested procedure is the following:

If a constant-temperature water bath is not available, determine the weight of the clean, dry pycnometer with thermometer to the nearest 0.1 mg on an analytical balance. Fill the pycnometer with water (9.1) cooler than 23°C. Insert the thermometer-stopper, causing excess water to be expelled through the side arm. Permit the filled bottle to warm in air until the thermometer reads 23.0°C. Remove the drop of water at the tip of the side arm with a bit of filter paper, taking care not to draw any liquid from within the capillary, place the cap over the side arm, wipe the outside carefully, and weigh the filled bottle again to the nearest 0.2 mg. Empty the pycnometer, dry, and fill and weigh with the other liquid in the same manner as was done with the water. Calculate the specific gravity 23/23°C of the liquid, d, as follows:

$$d = (b - c)/(w - c)$$

where:

c = apparent weight of empty pycnometer,
w = apparent weight of pycnometer filled with water at 23.0°C, and
b = apparent weight of pycnometer filled with liquid at 23.0°C.

If a constant-temperature water bath is available, a pycnometer without a thermometer may be used (compare 30.2).

NOTE 16—One standard object which has been found satisfactory for this purpose is the Reimann Thermometer Plummet. These are normally supplied calibrated for measurements at temperatures other than 23/23°C, so that recalibration is necessary for the purposes of these methods. Calibration at intervals of 1 week is recommended.

17. Test Specimens

17.1 See Section 10.

18. Procedure

18.1 The procedure shall be similar to Section 11, except for the choice of immersion liquid, and the temperature during the immersed weighing (11.3) shall be 23 ± 0.5°C (73.4 ± 1.0°F).

19. Calculations

19.1 The calculations shall be similar to Section 12, except that d, the specific gravity 23/23°C of the liquid, shall be placed in the numerator:

$$\text{Sp gr } 23/23°C = (a \times d)/(a + w - b)$$

20. Report

20.1 See Section 13.

METHOD A-3 FOR TESTING SOLID PLASTICS (SPECIMENS > 50 g)

21. Scope

21.1 This method is suitable for determining the specific gravity or density of items heavier than 50 g by immersion of the entire item, that is, nondestructively. Any appropriate liquid may be used.

22. Apparatus

22.1 *Balance*—A balance large enough to accommodate the desired specimen conveniently and to achieve a precision and accuracy within 0.05 % relative (that is, of the weight of the specimen in air).

NOTE 17—A chainomatic balance has been found to meet these requirements. Alternatively, a torsion balance will meet these requirements and can be used for immersed weighings if modified as follows: (*1*) a hole is drilled in the base directly under the left hand pan; (*2*) the balance is mounted on a shelf, having a hole aligned with the hole in the base. This shelf is placed over working area so that the specimen can be attached to a wire suspended from the pan. A shield may be necessary to minimize the effects of air currents upon the weighings.

22.2 The apparatus shall also include the wire (8.2), sinker, if used (8.3), immersion vessel of appropriate size (8.4), thermometer (8.5 or 15.4), constant-temperature bath, if used, (15.5), and pycnometer, if used, (15.2).

23. Materials

23.1 See 9.1 or 16.1.

24. Test Specimens

24.1 The test specimen shall be a single item or piece of the material under test, of any shape that can conveniently be prepared and tested. The weight of the specimen in air should preferably be upwards of 50 g, to the capacity limit of the balance, but in any event shall be large enough so that the precision requirements (22.1) can easily be met.

25. Procedure

25.1 The procedure shall be similar to Section 11 or 18, depending upon the choice of immersion liquid. No support for the immersion vessel is necessary if a torsion balance is used and is mounted completely above the immersion vessel.

26. Calculation and Report

26.1 See Section 12 or 19, and Section 13.

METHOD B FOR TESTING MOLDING POWDERS, PELLETS, AND FLAKE

27. Scope

27.1 This method is suitable for determining the specific gravity or density of essentially nonporous plastic powders large enough to be retained on a 20-mesh (850-μm) sieve, flake, pellets, drillings, or small pieces cut from larger shapes (Note 18). The immersed specimen is weighed in a pycnometer. The immersion liquid must have a specific gravity less than that of the specimen.

NOTE 18—Immersion of powders finer than 20 mesh lay lead to incomplete wetting because of static charges and surface tension effects.

28. Apparatus

28.1 The apparatus shall include the balance, pycnometer, thermometer, and constant-temperature bath of Sections 8 and 15, and the following:

28.2 *Vacuum Pump*—A water aspirator or vacuum pump capable of producing a vacuum high enough to cause the liquid to boil at 23°C or to produce a vacuum of 3 mm Hg or better. The pump shall be provided with a trap, for preventing backflow of oil or water, and a stopcock on the inlet side.

28.3 *Vacuum Desiccator*—A vacuum desiccator or bell jar with plate constructed to withstand a vacuum of 1 atm and connections to the source of vaccum.

NOTE 19—Before a new desiccator is used for the first time, it should be wrapped in a towel, placed in a heavy shield, and tested under the vacuum to be used. The operator should be adequately protected by the proper safety devices, such as goggles. The same precautions should be observed under operating conditions.

29. Materials

29.1 *Water* (9.1) or other immersion liquid (16.1).

30. Procedure

30.1 Weigh the clean, dry pycnometer to the nearest 0.1 mg. Record this weight in grams as *e* (weight of empty pycnometer).

30.2 Fill the pycnometer with the liquid to be used at a temperature of approximately 23°C. Place the filled pycnometer without cap in the water bath and allow to remain until it has attained temperature equilibrium with the bath. If the liquid level in the capillary in the stopper has dropped, bring it level with the top of the stopper by adding more liquid. Wipe the outside of the pycnometer dry, cap, and weigh to the nearest 0.2 mg. Record this weight as *b* (weight of pycnometer filled with immersion liquid).

30.3 Clean and dry the pycnometer. Add 1 to 5 g of the material and weigh to the nearest 0.1 mg. Subtract *e* from this weight and record the difference (the weight of the specimen) as *a*.

30.4 Place sufficient liquid in the pycnometer to cover the specimen. Place the pycnometer without stopper in the vacuum desiccator and apply vacuum gradually until all the air has been removed from between the particles of the specimen. Take care not to permit any particles to be splashed out of the pycnometer or onto the ground portion of the neck, where they might prevent the stopper from seating properly.

30.5 Add additional liquid to fill the pycnometer. Insert the stopper, transfer to the bath, adjust to temperature, adjust the final volume, wipe, cap, and weigh. Record this weight as *m* (weight of pycnometer containing the specimen and filled with liquid).

NOTE 20—Since the precision of this method depends on the measurement of a small difference between two large weights, all weighings must be performed with unusual care. The temperature during the determinations of *b* and *m* must be exact in order that the volume of liquid displaced by the specimen can be accurately obtained. Other potential sources of error are retention of air by the specimen and absorption of the liquid.[7]

31. Calculations

31.1 Calculate the specific gravity 23/23°C of the specimen as follows (Note 21):

$$\text{Sp gr } 23/23°C = (a \times d)/(b + a - m)$$

NOTE 21—The weight of the liquid having a volume equal to that of the specimen is $(b + a - m)$ and the volume of this weight of liquid is $(b + a - m)/d$.

31.2 Calculate the density of the plastic as in 12.2.

32. Report

32.1 See Section 13.

33. Precision

33.1 In these methods, the following criterion should be used for judging the acceptability of results at a 95 % confidence level:

33.2 *Repeatability*—Two results (each the average of duplicate determinations) obtained by the same operator should be considered suspect if they differ by more than 0.1 %, relative.

[7] Determination of the specific gravity of powders is discussed in detail, together with methods of obtaining high precision in such measurements, in Hillebrand, W. F., and Lundell, G. E. F., *Applied Inorganic Analysis*, John Wiley & Sons, Inc., New York, NY, 1929, pp. 661–669.

Appendix B

Type V and Acrylic Plastic Spectra

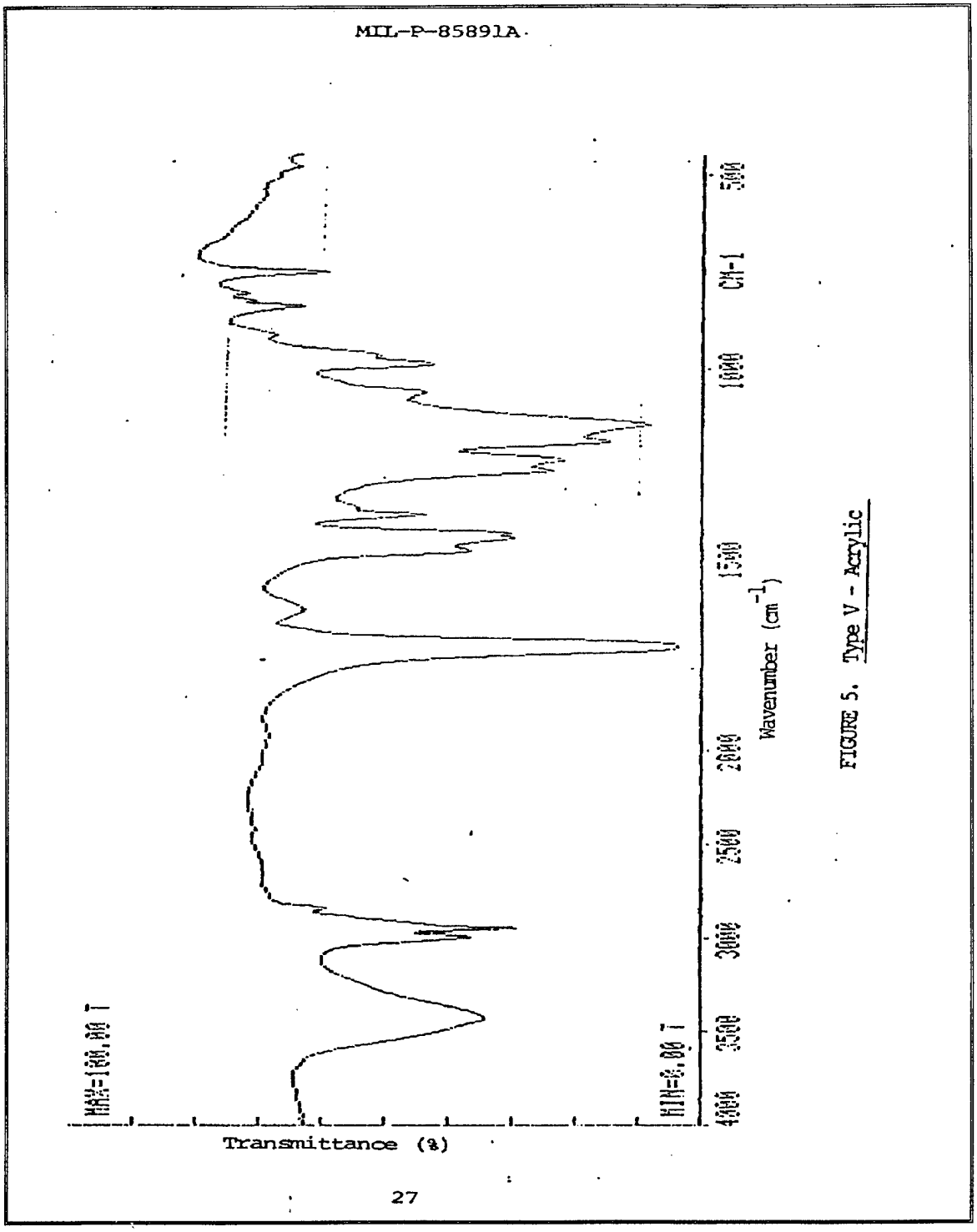
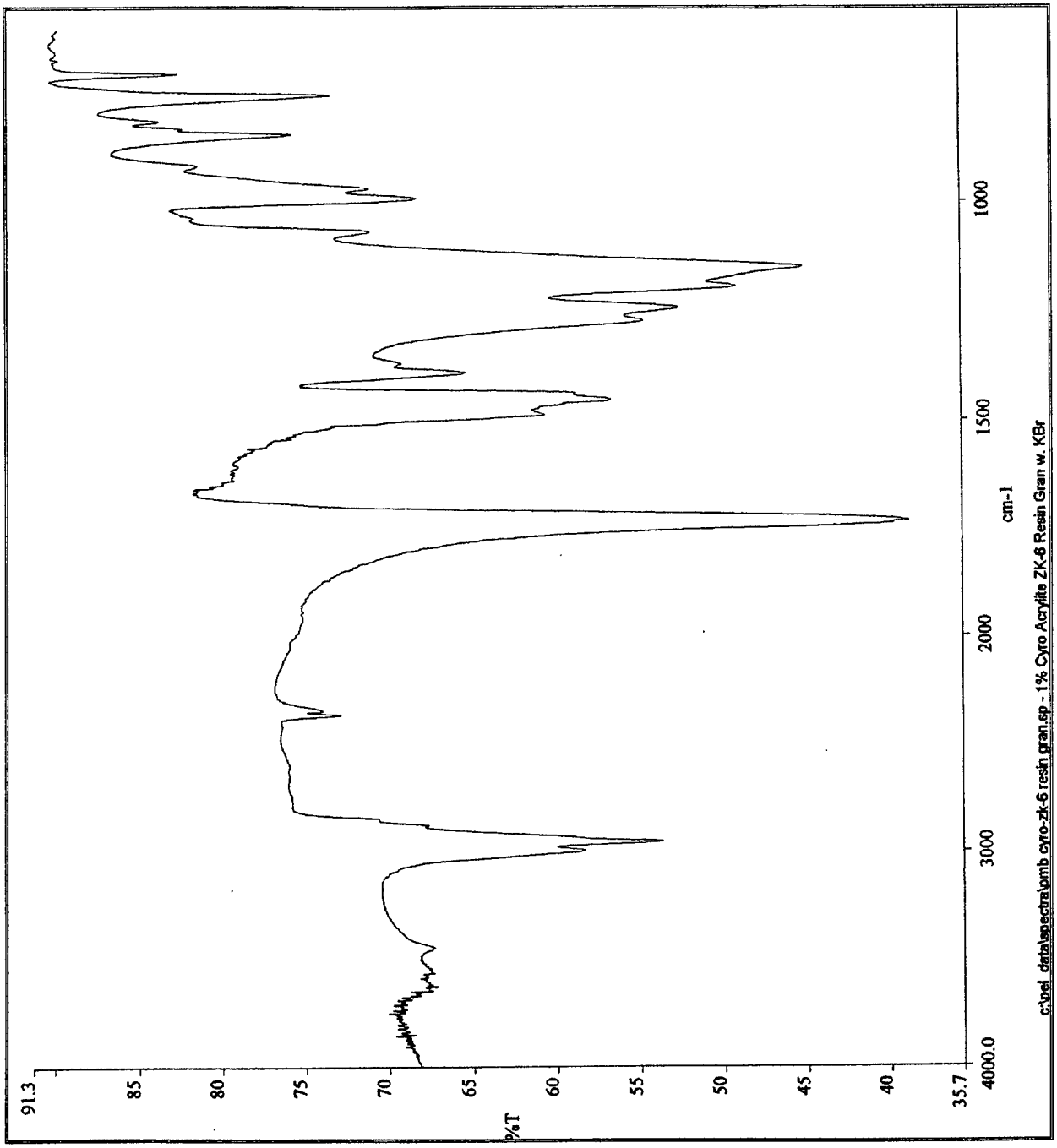
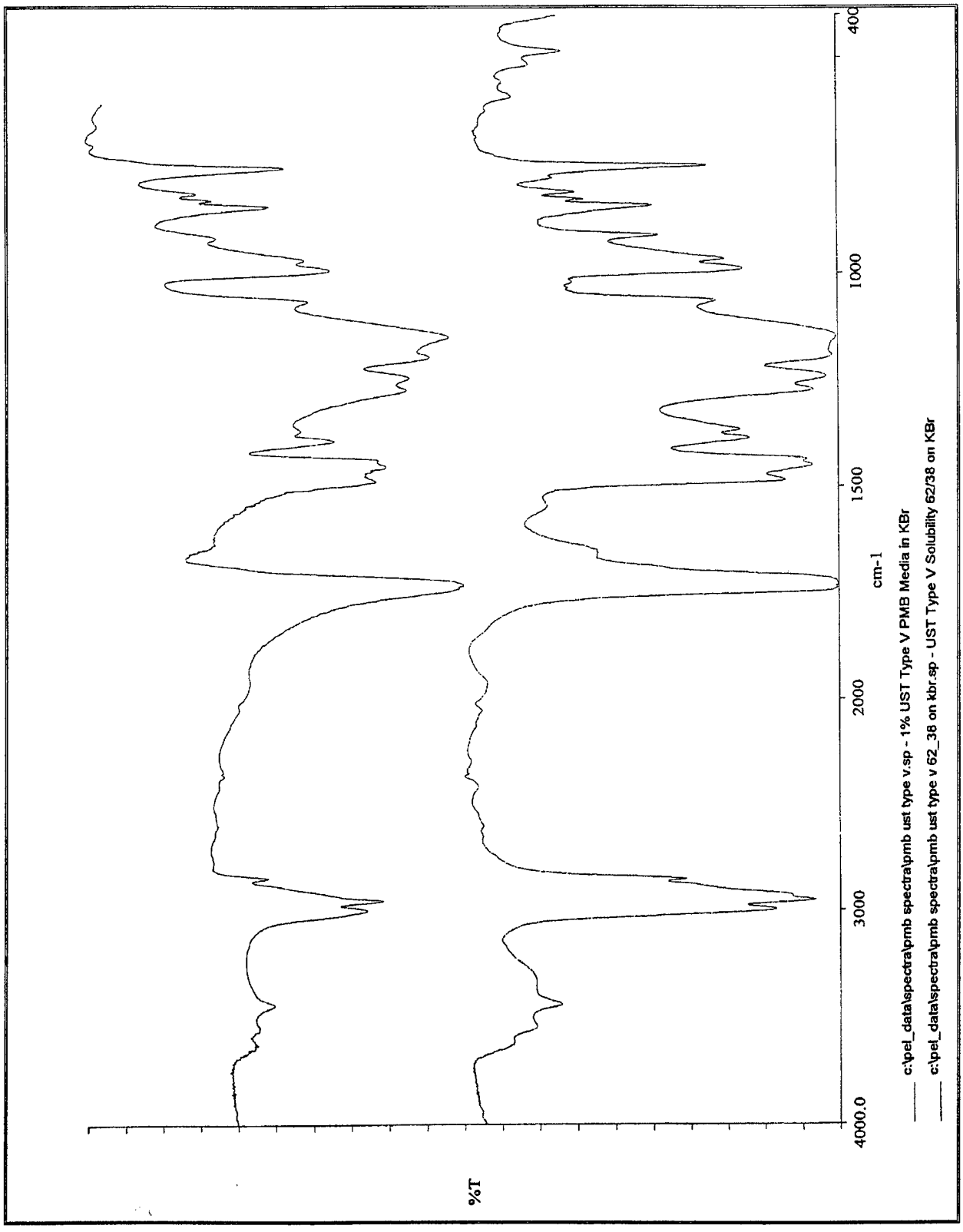


FIGURE 5. Type V - Acrylic

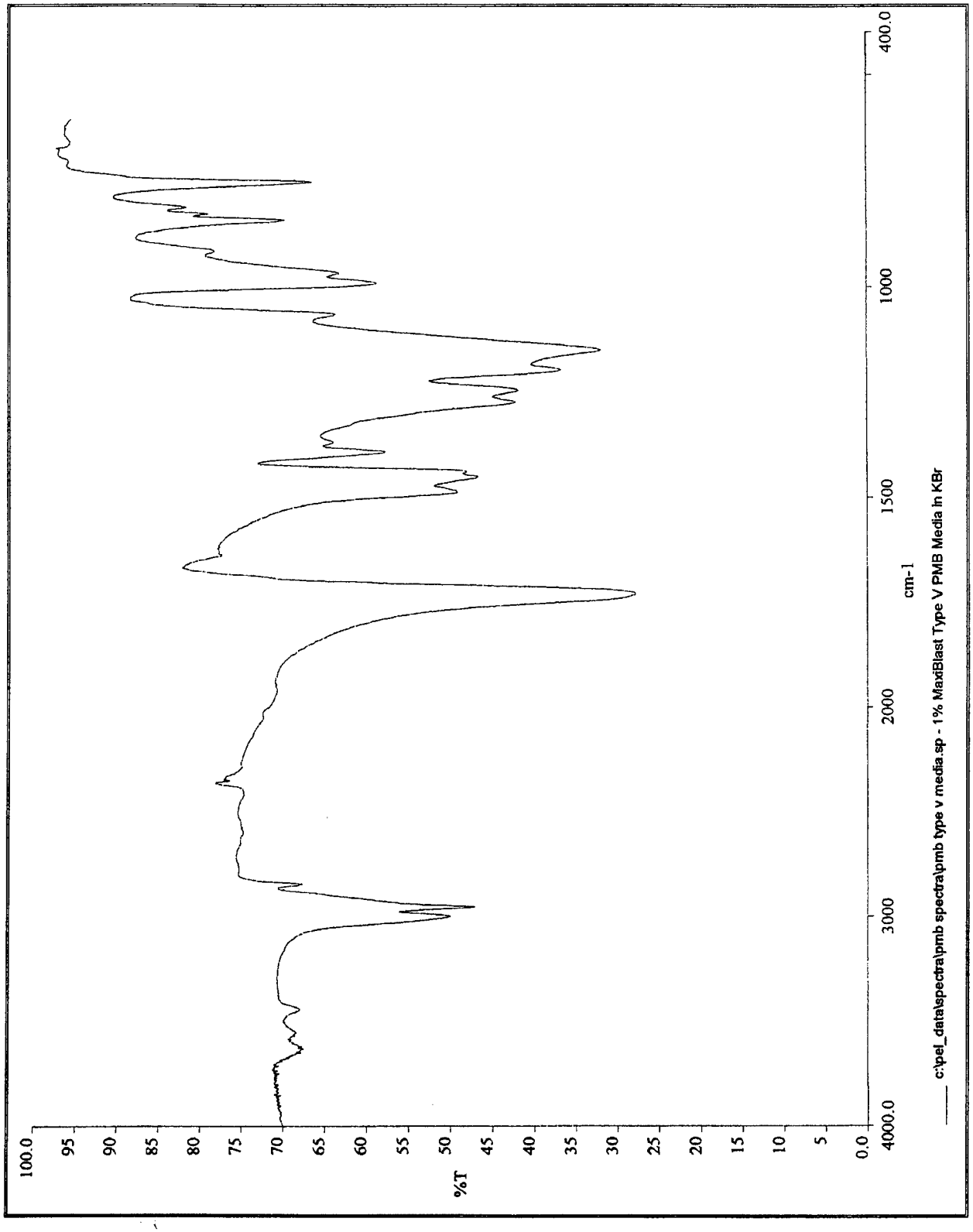
Cyro ZK-6 Acrylic Plastic



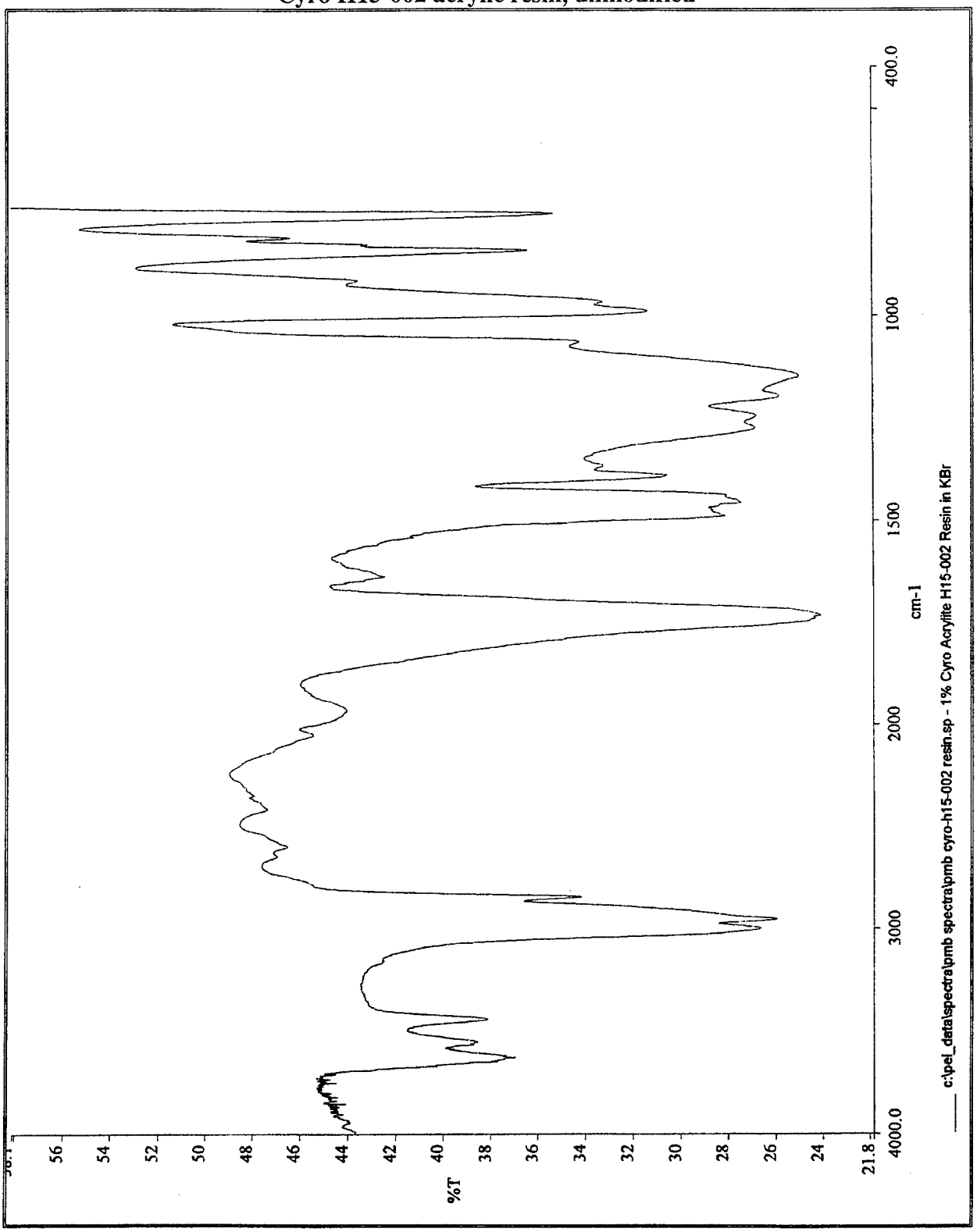
U.S. Technologies Type V Media and Methylene Chloride Extract Retained on Filter Paper



Maxiblast Type V Media



Cyro H15-002 acrylic resin, unmodified



Acrylite ZK-6 Methylene Chloride Extract