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Synthesis of MDIPA, an Aromatic Diamine Curative for Polyurethane Adhesives

By F. N. Larsen

Published September 1980

Final Report

Prepared for the United States Department of Energy
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Bendix Kansas City Division

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SYNTHESIS OF MDIPA, AN AROMATIC DIAMINE CURATIVE FOR POLYURETHANE ADHESIVES

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Final Report
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The compound 4,4'-methylene bis-(2,6-diisopropylaniline), or MDIPA, has shown good thermal stability, high butt tensile strength, and excellent compatibility with salt when used as a curative for LLL's Halthane 87 prepolymer. Laboratory-scale synthesis of MDIPA by condensation of commercially-available 2,6-diisopropylaniline and formaldehyde has been accomplished, using the amine sulfate as an intermediate. Polymer Pilot Plant scale-up has been achieved; kilogram quantities at acceptable purity levels (98+ percent) have been produced.

WPC-TR4/k

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A prime contractor with the United States Department of Energy under Contract Number DE-AC04-76-DP00613

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SUMMARY

The chemical compound 4,4'-methylene bis-(2,6-diisopropylaniline), or MDIPA, when used as a cure agent with isocyanate-terminated prepolymers, has shown excellent adhesive strength and compatibility properties in certain applications. During development and evaluation of polyurethane adhesive systems at LLL and UCC/Y-12, the sole supplier of MDIPA ceased production. Because Bendix Kansas City was manufacturing the prepolymer, Halthane 87 (an LLL-developed polyurethane prepolymer), for use at other DOE agencies, Bendix was suggested as an alternate supplier of the MDIPA compound.

After existing technology to synthesize MDIPA was transferred to Bendix from UCC/Y-12, laboratory experiments were performed to optimize synthetic parameters, resulting in successful production of gram quantities of MDIPA. Test methods were developed to analyze the starting amine (2,6-diisopropylamine) and the reaction product (MDIPA). The primary method used was gel permeation chromatography; gas chromatography, infrared spectrophotometry, liquid chromatography, and mass spectrometry also were used.

Scale-up procedures were prepared and several kilogram batches were produced in the Polymer Pilot Plant with purity levels essentially identical to the previously available product.

DISCUSSION

SCOPE AND PURPOSE

Restrictions imposed by the Occupational Safety and Health Administration (OSHA) on the use of methylene bis (2-chloroaniline), MOCA, has prompted the development of alternates to replace a widely used polyurethane adhesive system. One such system is a Lawrence Livermore National Laboratory-developed prepolymer, Halthane 87, cured with aromatic diamines.[1] The 87 prepolymer is a methylene bis(4-cyclohexyl isocyanate), Hylene W, terminated polytetramethylene ether glycol, MW=2000. This prepolymer is manufactured by the Bendix Polymer Pilot Plant.

One of the aromatic diamine cure agents for Halthane 87 showing excellent properties and good salt compatibility has been investigated recently. This material, 4,4'-methylene bis(2,6-diisopropylaniline) is commonly referred to as MDIPA. As a result of the early evaluation and test data, materials and engineering personnel at Los Alamos National Scientific Laboratory and LLNL expressed an interest in this cure agent. Unfortunately, the supplier had only very limited quantities available and indicated they would not supply additional material in the future.

This project was initiated to investigate the synthesis and scale-up capabilities and to provide limited quantities of MDIPA to the DOE agencies.

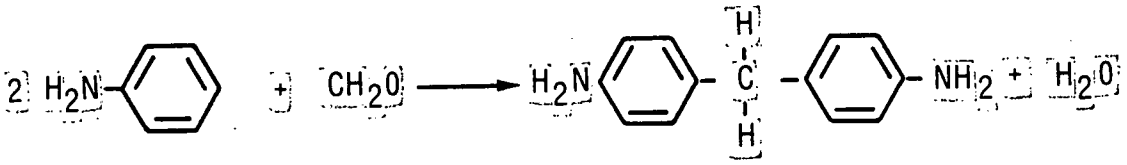
PRIOR WORK

No previous efforts to synthesize MDIPA have been made at Bendix. However, Dr. G. F. Dorsey at Union Carbide Company, Y-12 Plant, Oak Ridge, Tennessee, had developed general synthetic procedures for methylene bridged aromatic diamines and provided a specific procedure for the MDIPA through the amine hydrochloride intermediate. Dr. Dorsey also provided small quantities of his synthetic product for properties and purity comparison.

ACTIVITY

General Synthesis Scheme

The synthesis of MDIPA follows the general reaction of aniline-formaldehyde condensations; the active para position hydrogens of the aromatic ring react with formaldehyde, splitting off water and joining the phenyl groups with a methylene bridge. Unsubstituted aniline reacts with formaldehyde at the ortho and para positions, resulting in cross-linked thermosetting resins. Two advantages accrue from using ortho substituted anilines. One



(1)

is the blocking for reactions at the ortho site, eliminating the crosslinking actions. The other advantage is the reduction of the amine reactivity by steric and electronic effects of the side groups; this slows the reaction with isocyanates, giving longer pot life and handling times to the adhesive systems. Diisopropylaniline (DIPA) is one such disubstituted aniline and is the starting amine for MDIPA synthesis. The condensation with formaldehyde must be preceded by the formation of the amine salt using either hydrochloric or sulfuric acid. This step is required to prevent unwanted formylation at the amine group.

After the methylene coupling of the DIPA, the diamine salt must be neutralized and the free amine recovered and purified by recrystallization.

Raw Materials

The 2,6-diisopropylaniline (95 percent) was purified by fractional distillation under vacuum. Freshly distilled material was used in the MDIPA reactions as soon as possible, or converted to the hydrochloride or sulfate for storage stability. The properties of this compound are listed in the Appendix. The commercially available product is usually dark brown in color and normally contains 95 percent 2,6-diisopropylaniline, 4 percent 2-n-propyl-6-isopropylaniline and 1 percent other amine impurities. This product can be redistilled under vacuum to a pale yellow clear liquid.

Formaldehyde, 37 percent aqueous solution, was assayed using the sodium sulfite procedure and found to be 37.55 percent formaldehyde.

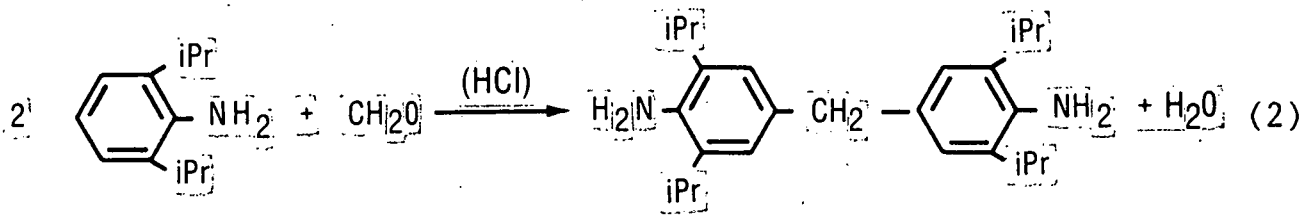
Absolute ethanol, isopropanol, hexane, petroleum ether and diethyl ether of reagent grade quality were used as received.

The sulfuric and hydrochloric acids (concentrated) used were reagent grade quality.

Concentrated ammonium hydroxide of reagent grade also was used in the synthesis.

Synthesis Via Amine Chloride Salt

The overall reaction using the amine hydrochloride is as follows:



(2)

where iPr = isopropyl.

The three separate reaction steps, together with the stoichiometry and yield, are as follows.

100	20.6	121	(Amount, g)	
DIPA + HCl		DIPA·HCl	(Reaction)	(3)
177.3	36.5	213.8	(Molecular Weight, g/mol)	

121	8.5	124		
2 DIPA·HCl +	CH_2O	MDIPA·2HCl +	H_2O	(4)
427.6	30	439.6		

124	22.6	103.4		
MDIPA·2HCl +	2NaOH	MDIPA +	2NaCl + H_2O	(5)
439.6	80.0	366.6		

At 100 percent yield, 100 parts by weight (pbw) of DIPA will yield 103.4 pbw of MDIPA.

Reaction 3, the preparation of the amine hydrochloride, was carried out by adding by drops, with constant stirring, 100 g of as received (95 percent) DIPA into a 2-L beaker containing a solution of 60 mL concentrate HCl in 1000 mL distilled water. The solution will solidify, and additional 10 percent HCl is added until a slurry is obtained. The slurry is chilled to lower the solubility of DIPA·HCl and then filtered, washed with diethyl ether, and finally vacuum dried. The product is a very light, white powder.

The coupling reaction (4) is carried out in a 4-necked reaction flask equipped with stirrer, nitrogen inlet, thermometer, and addition funnel. Twenty grams of the DIPA·HCl were added to a 500 mL flask containing 150 mL H_2O and 7 mL concentrated HCl. The solution was heated to 100°C and 3.75 mL (1.405 g) of CH_2O were added fairly rapidly (2 to 3 minutes) through the addition funnel. After about 10 minutes reaction time, the solid MDIPA-2 HCl product forms. The slurry is held at 100°C for 30 minutes, then allowed to cool to 50°C. The slurry is then filtered, washed with 25 percent HCl and finally with diethyl ether.

The neutralization step (5) is carried out by transferring the MDIPA·2 HCl from the previous procedure into a 600 mL beaker. Then 100 mL of hexane, 100 mL H_2O and 15 mL of a 6.25N NaOH solution is added and followed by vigorous stirring. After extraction of the free MDIPA into the hexane layer, separation is effected using a separating funnel. The MDIPA/hexane layer is dried over K_2CO_3 and the hexane removed by vacuum distillation. The oily MDIPA product then is allowed to crystallize by cooling or seeding with MDIPA crystals.

Originally, a procedure was suggested which called for performing all the reaction in one reaction flask set-up with no separation of the hydrochloride salt. The major disadvantage in that procedure is that the starting DIPA must be very pure because all impurities are carried through the various reactions.

The discoloring impurities can be separated by filtering and washing the DIPA·HCl and the MDIPA·2HCl.

Early in the project it was learned that the reaction involving strong HCl solutions and formaldehyde could possibly form dichlorodimethyl ether which is toxic. This compound has been restricted by OSHA to a few parts per billion human exposure. Therefore, all Bendix syntheses use sulfuric acid.

Synthesis Via Amine Sulfate Salt

The procedure for synthesis of MDIPA using sulfuric acid is essentially identical to that for the hydrochloride salt intermediate. Several batches using the sulfate intermediate were attempted where a number of variables were examined to optimize the synthetic procedure.

Several conclusions were drawn from these series of experiments and are listed below.

> It is better to form the amine salt (DIPA · sulfate) as a first step procedure. The salt is a stable white, free flowing powder that can be easily stored and used as needed; it is not as susceptable to oxidation as the free amine.

The amine sulfate can be prepared using the as-received (95 percent purity) DIPA because the dark impurities can be removed during filtration and subsequent washing. This method eliminates the difficult vacuum distillation of the DIPA.

When using the amine salt for the condensation with formaldehyde, it is not necessary to exclude air from the environment. Thus, the reaction can all be carried out in open beakers or flasks. There was no noticable change in color or reaction rates when the entire synthesis was carried out in open beakers rather than a closed, nitrogen-flushed reaction vessel. This expedites handling and reduces equipment needs, particularly for scale-up.

Isopropanol (IPA) can be substituted for ethanol in the DIPA·sulfate preparation and the subsequent condensation procedure. IPA is more readily available and obviates the presence of denaturants in the ethanol.

The MDIPA·sulfate can be easily isolated as a white, free flowing powder. Preparation and isolation before forming the free amine by neutralization has the advantage of removing additional impurities, particularly any unreacted DIPA by filtration and washing.

Ammonium hydroxide is used rather than sodium or potassium hydroxide in the neutralization step primarily for convenience in handling.

Petroleum ether is the solvent of choice for the extraction of MDIPA. It is low boiling (for easy removal) does not form perioxides, and has high solubility for MDIPA and very low solubility for any unreacted amine sulfates.

The generalized procedure then for the laboratory-scale experiments is preparation of DIPA·sulfate, preparation of MDIPA·sulfate, and conversion of MDIPA·sulfate to a free amine.

Preparation of DIPA·Sulfate

With a high rate of stirring, add 100 g of the as-received DIPA to a 2-L beaker containing 35 mL of concentrated sulfuric acid and 300 mL distilled water. As the slurry thickens, add 200 mL water and 25 mL isopropanol (IPA). Continue stirring and heat to 70 to 80°C, adding enough additional IPA to effect solution. Cool to ambient, chill overnight, and then filter. Wash with petroleum ether and dry.

Preparation of MDIPA·Sulfate

Add 125 g of DIPA·sulfate to a 2-L beaker containing 650 mL water, 150 mL IPA and 15 mL concentrated sulfuric acid. Stir to a slurry and heat to 70 to 80°C. Add sufficient IPA to solubilize the DIPA·sulfate (~70 mL). Add slowly 18.5 mL of 37.5 percent aqueous formaldehyde. Continue heating at 70°C for 1 hour. Cool and chill to lower solubility of the MDIPA·sulfate. Filter product on a Buchner funnel and wash successively with 50 mL of 50 percent IPA/water, 200 mL IPA, 150 mL of 50 percent IPA/Petroleum ether and 150 mL Petroleum ether. Air dry the MDIPA·sulfate and then vacuum dry to remove the residual solvents.

Conversion of MDIPA·Sulfate to Free Amine

Place 100 g of MDIPA·sulfate in a 2-L beaker. Add 1.0 to 1.2 L of distilled water and stir to form a slurry. Add 80 mL of concentrated ammonium hydroxide. Coagulation of the free diamine will occur. Add 300 mL of petroleum ether and stir or agitate to extract the free amine into the organic layer. Separate the petroleum ether layer using a separatory funnel. Re-extract the aqueous layer with 100 mL petroleum ether and combine extracts. Remove the petroleum ether by ambient evaporation using a drafted hood. A viscous liquid will remain which will crystallize on standing. This is the MDIPA product.

Purification

The overall yield of the MDIPA synthesis will vary from 25 to 80 percent; the wide range is caused by the sensitive solubility of the amine salts and the complete solubility of the MDIPA in all solvents. The presence of the two ortho alkyl groups in the molecular structure of MDIPA imparts very high oil solubility (nonpolar solvents) and the primary amine moeity contributes polar solvent solubility. This high solubility of the MDIPA makes purification by crystallization or solvent fractionation very difficult. However, a recrystallization procedure using methanol as a solvent and water as a non-solvent was devised. This procedure for the recrystallization of MDIPA is as follows.

Dissolve 100 g of crude MDIPA in 500 mL of methanol. Transfer to a 2-L beaker.

Add cold distilled water slowly while rapidly stirring the solution. An amount of water equal to 25 percent of the volume of methanol (~125 mL) should be sufficient to cause the MDIPA to separate as a solid phase. Continue stirring for 15 minutes.

Chill overnight in a cold box (0°C) and then filter solids on a Buchner funnel. Wash the filter cake quickly with a well chilled 5:1 methanol/water solution.

Vacuum dry the white to light tan solid MDIPA product for several hours. Use no heat, as the MDIPA will melt at approximately 60°C.

This recrystallization step will remove most of the free DIPA and oliomer impurities. However, there will be residual water and methanol in the product. The removal of water and methanol is accomplished by vacuum drying the MDIPA at 80°C and 5 to 10 mm Hg, where the MDIPA is liquid and the volatile solvents can be easily removed by heat and vacuum. One hour is usually sufficient. The dried, melted MDIPA is placed in aluminum foil or polyethylene lined trays and allowed to crystallize. The solid product is broken up and packaged for storage.

Pilot Plant Scale-Up

The development of a laboratory procedure using essentially open beaker reactions and handling made the transfer and scale-up to the Polymer Pilot Plant relatively easy. Starting with 1 kg of DIPA, three separate Pilot Plant batches were produced. The yields varied somewhat, but a total of about 1.5 kg of MDIPA was made.

No particular problems were encountered. However, precautions must be taken to ensure that no metal vessels or stirring blades are used where the acid solutions are required, and careful handling of the petroleum ether is necessary because of its low flash point and high volatility. Some minor adjustments in the processing equipment and temperature control can probably lead to better yeilds in future production.

Product Analysis and Properties

Typical properties for 2,6-diisopropylaniline (DIPA) are given in the Appendix. It is of interest here to note the purity analysis for DIPA as determined by gas chromatography. The original lot of material analyzed at 93.7 percent 2,6-diisopropylaniline; 4.2 percent 2-n-propyl-6-isopropylaniline; 1 percent N-diethylphenylenediamine; and traces of 2,4-dimethylaniline, aniline, and toluene. The lot of DIPA which had been freshly prepared showed 93.1 percent DIPA; the balance of impurities was identical. A sample of redistilled DIPA showed an increase in the total dipropylaniline content from 98 to 99 percent. The impurity peaks were identified by mass spectrometry. An infrared spectrum of the 2,6-diisopropylaniline is shown in Figure 1. The only impurities of significance in the MDIPA product are unreacted diisopropylaniline and a 3 phenyl ring oligomer. The presence of free or unreacted DIPA in the MDIPA would have the

effect of polymer chain growth stopping as a curative because of its monofunctionality. Oligomers of 3, 4, or more phenyl rings would have the effect of crosslinking because of a functionality greater than 2. Therefore, the analysis for these components in the MDIPA product is required. Infrared spectrophotometry, gel permeation chromatography, and melting point were used as methods to evaluate the purity of the MDIPA.

Infrared (IR) spectra of the various MDIPA samples are shown in Figures 2 through 6. These spectra are identical except for Batch 3 (Figure 2) which is used to show that IR can be used to detect significant amounts of impurities. The free DIPA is detected as an absorption peak at 746 cm^{-1} and can be used to monitor free DIPA down to 1 or 2 percent concentration in the MDIPA.

Figure 3 is the spectrum of the original material. Figure 4 is the sample of material from UCC/Y-12 using the hydrochloride procedure and Figures 5 and 6 are spectra of the Bendix Pilot Plant Batches PP-1 and PP-2, respectively.

Gel permeation chromatography (GPC) utilizing low porosity Styragel columns was the most powerful tool for analysis of the MDIPA products. Figures 7 through 11 show the distribution and quantitation of components in the products. Figure 7 is the GPC of an early laboratory synthesis (Batch 3) containing significant amounts of free DIPA and oligomers. The original and GFD-15-62 UCC/Y-12 materials are shown in Figures 8 and 9. The first sample is obviously quite free of impurities. The GPCs of the Bendix Pilot Plant Batches PP-1 and PP-2 are shown in Figures 10 and 11, respectively.

The melting point of MDIPA is extremely sensitive to the presence of impurities. The MDIPA has a tendency to supercool and remain a viscous liquid even in the pure state. The presence of a few percent of unreacted DIPA almost obviates crystallization of the product. The melting point and analytical values for free DIPA and oligomers are given in Table 1 for selected MDIPA samples.

It should be noted that separation and identification of the 3 ring oligomer shows a structure that is not trifunctional with respect to the amine. The structure shown here indicates that N-alkylation has occurred in the synthesis. This implies migration or rearrangement of one ortho isopropyl group, as no mono-isopropylaniline has been detected in the starting DIPA. The structure shown is still only difunctional and the presence of

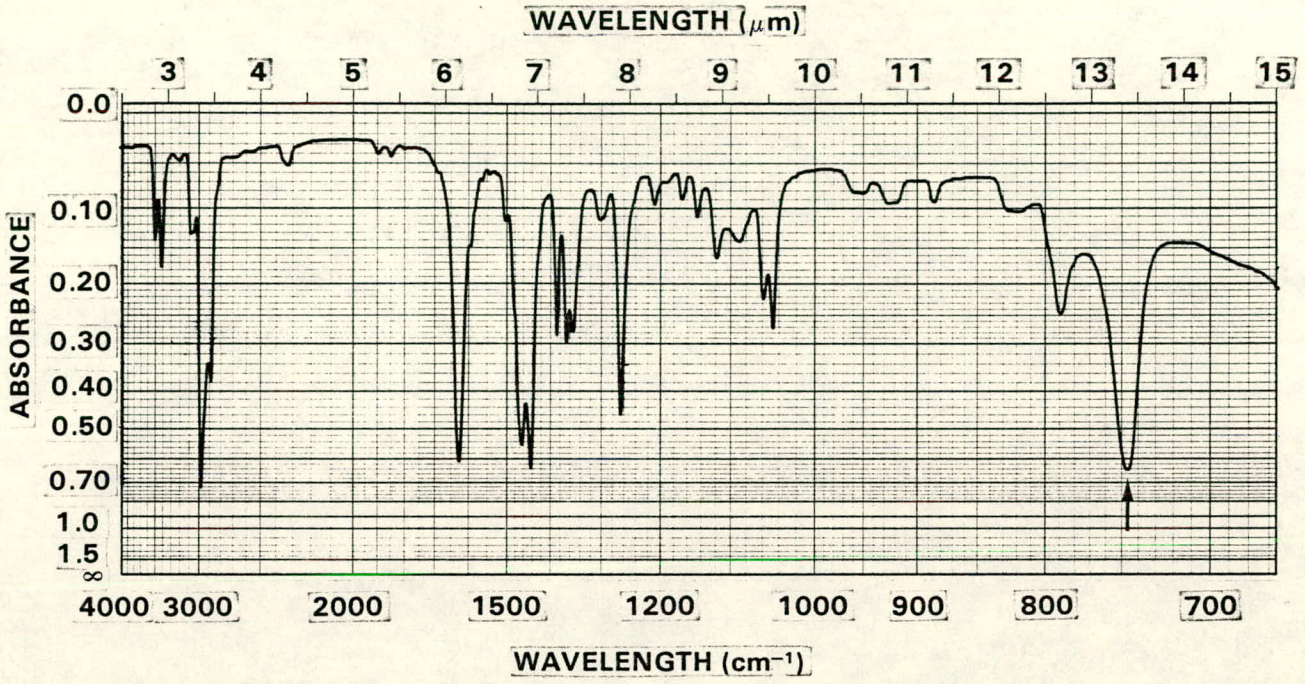


Figure 1. Infrared Spectrum of 2,6-Diisopropylaniline

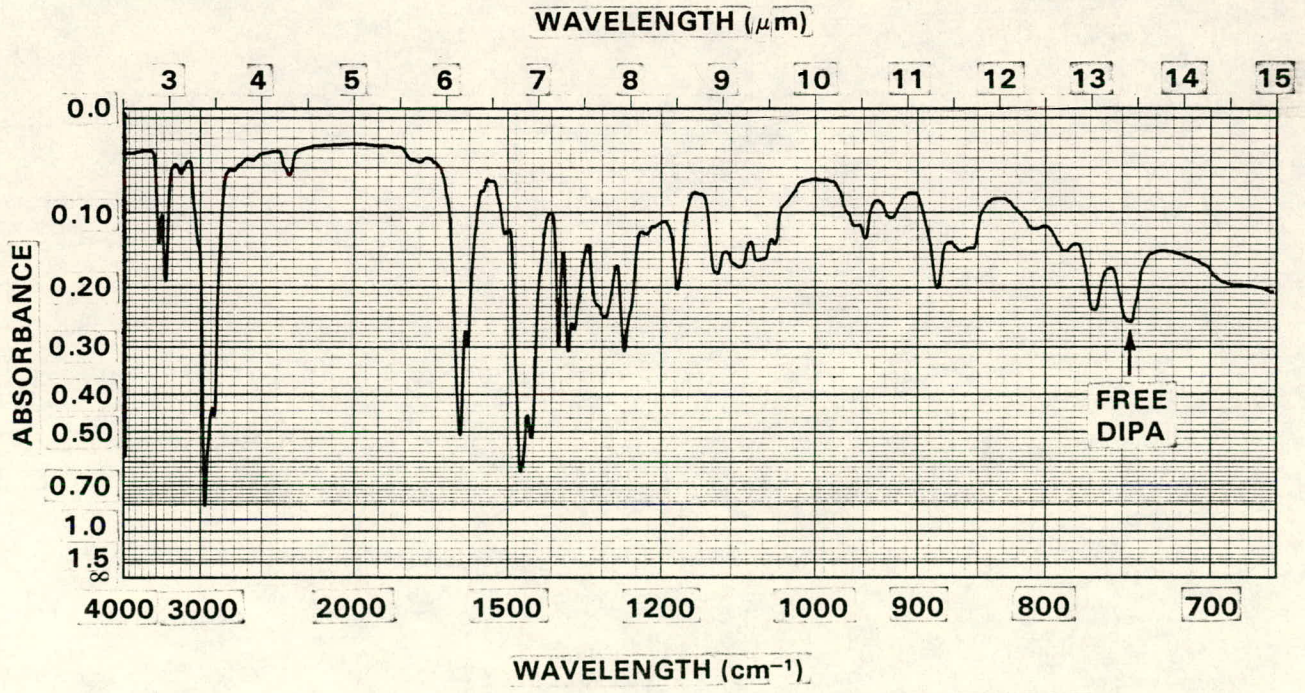


Figure 2. Infrared Spectrum of MDIPA, Batch 3

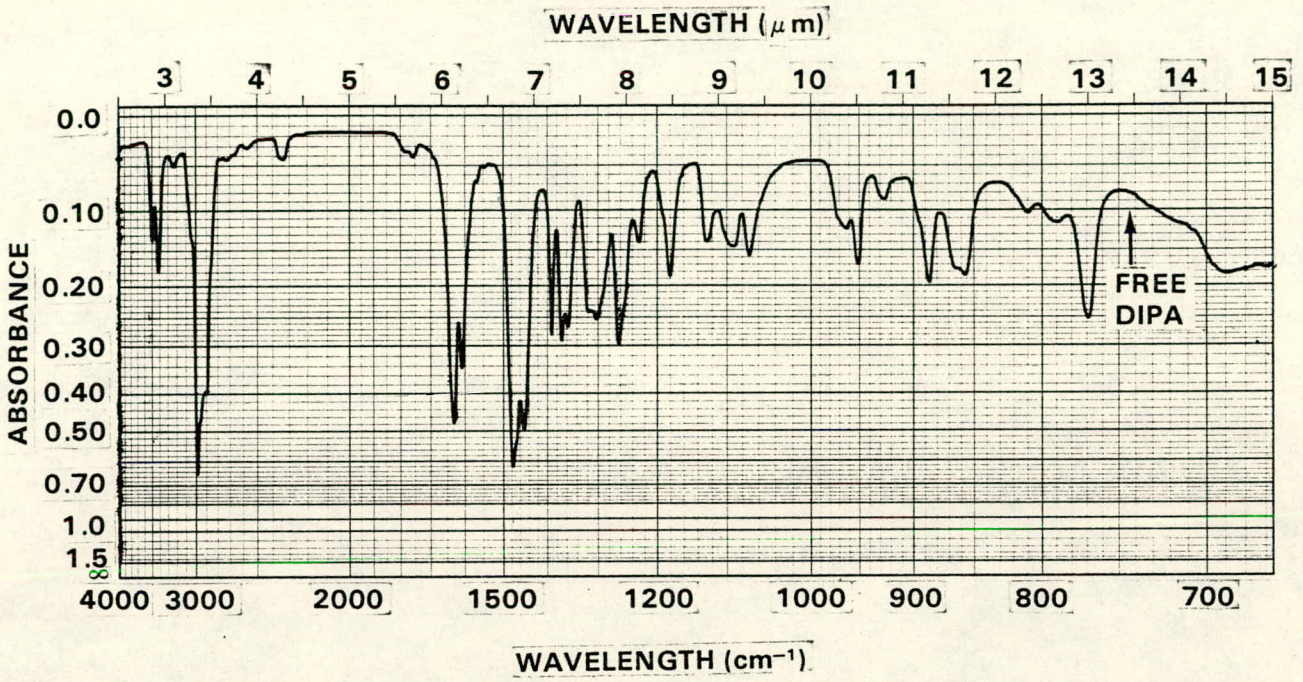


Figure 3. Infrared Spectrum of MDIPA

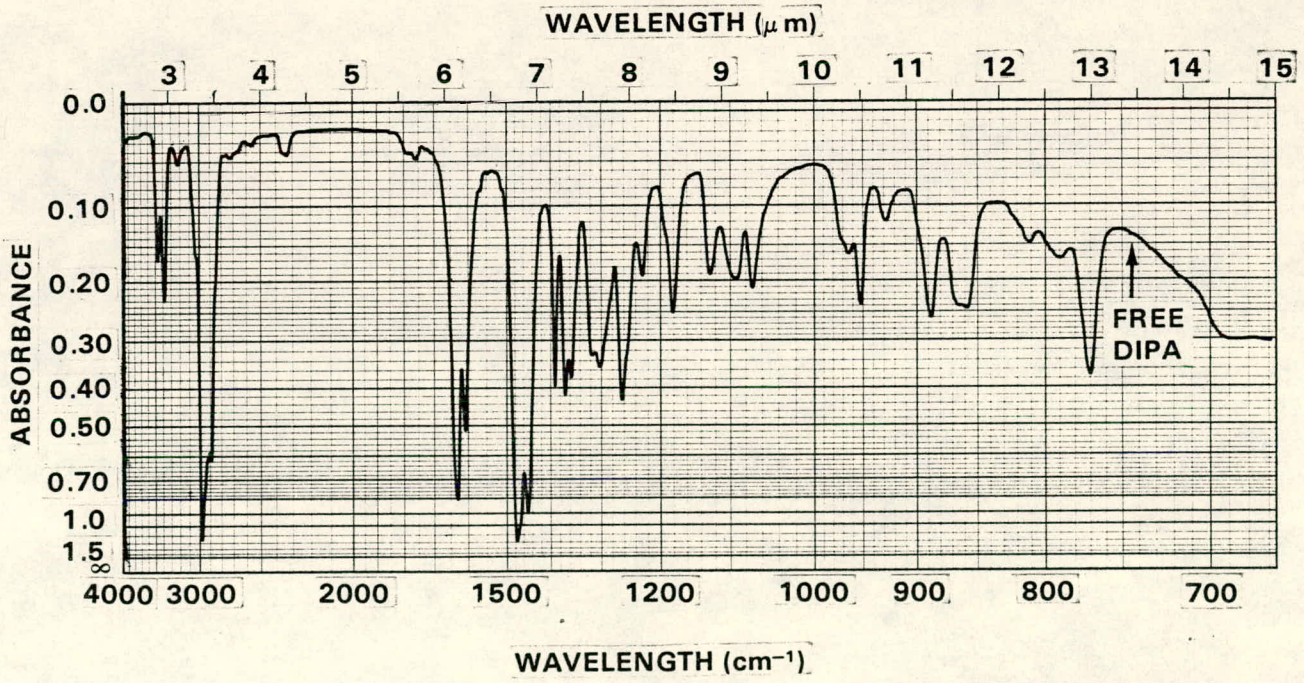


Figure 4. Infrared Spectrum of MDIPA, UCC/Y-12 GFD 15-62

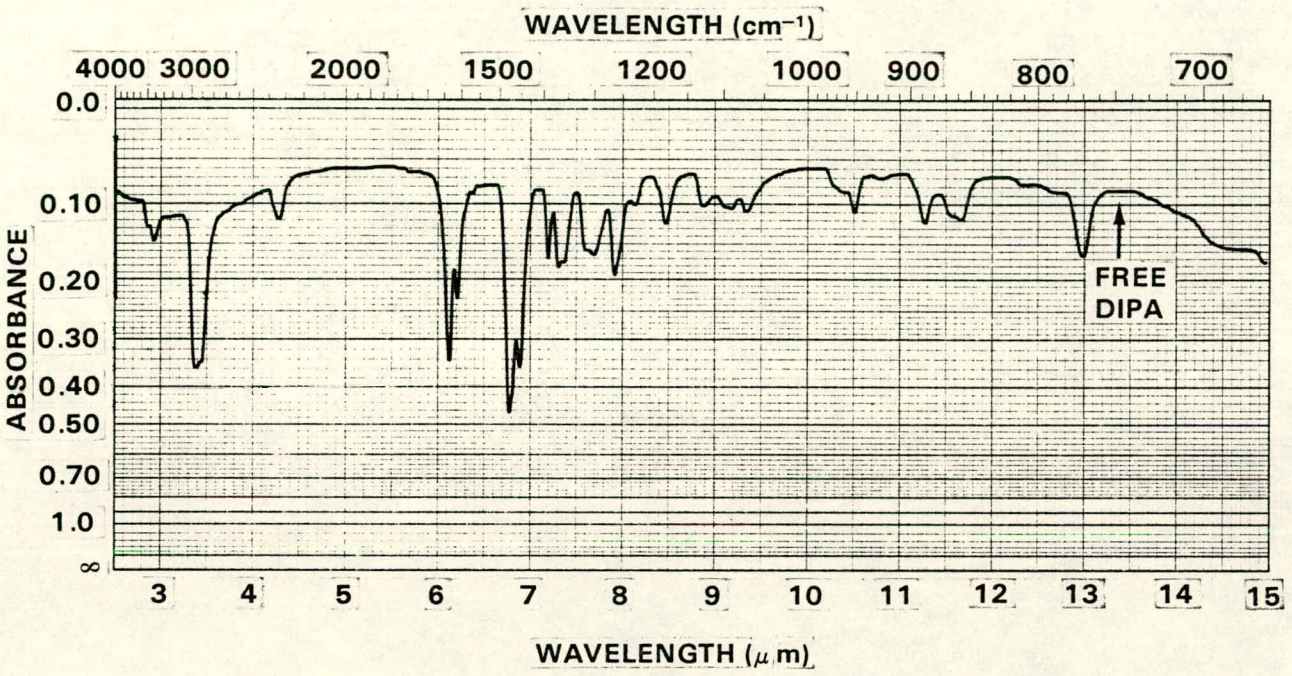


Figure 5. Infrared Spectrum of MDIPA, PP-1

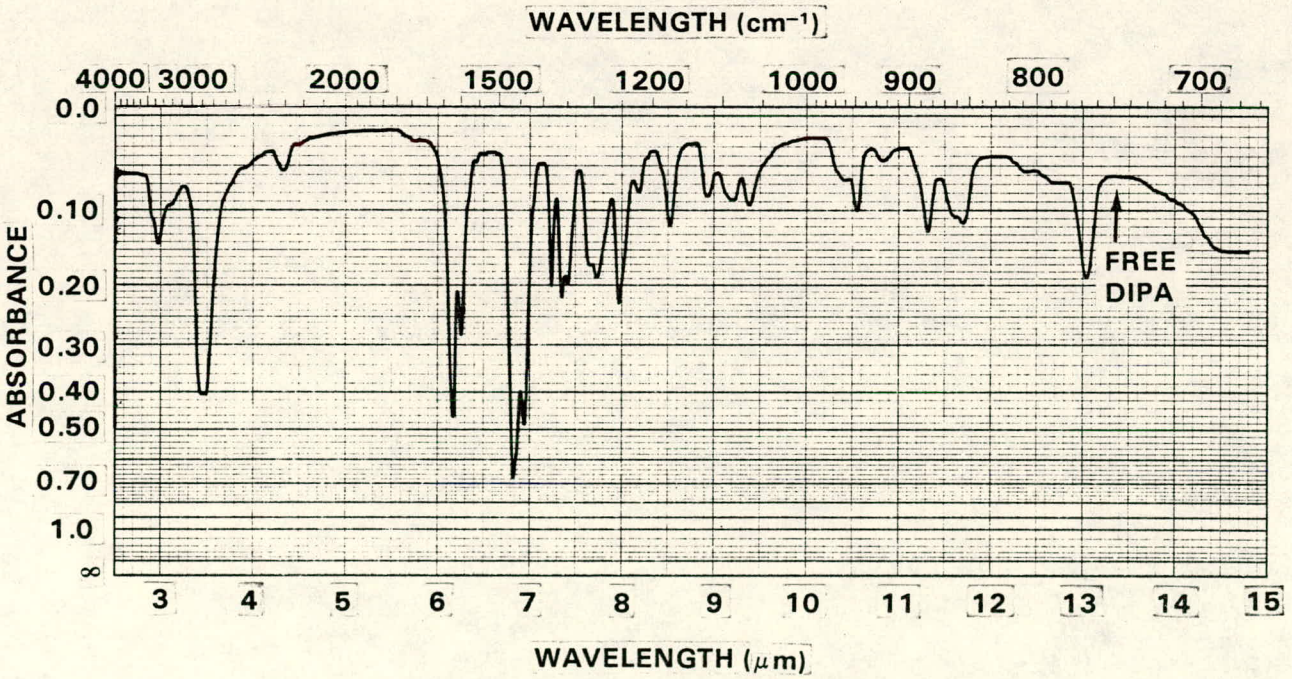


Figure 6. Infrared Spectrum of MDIPA, PP-2

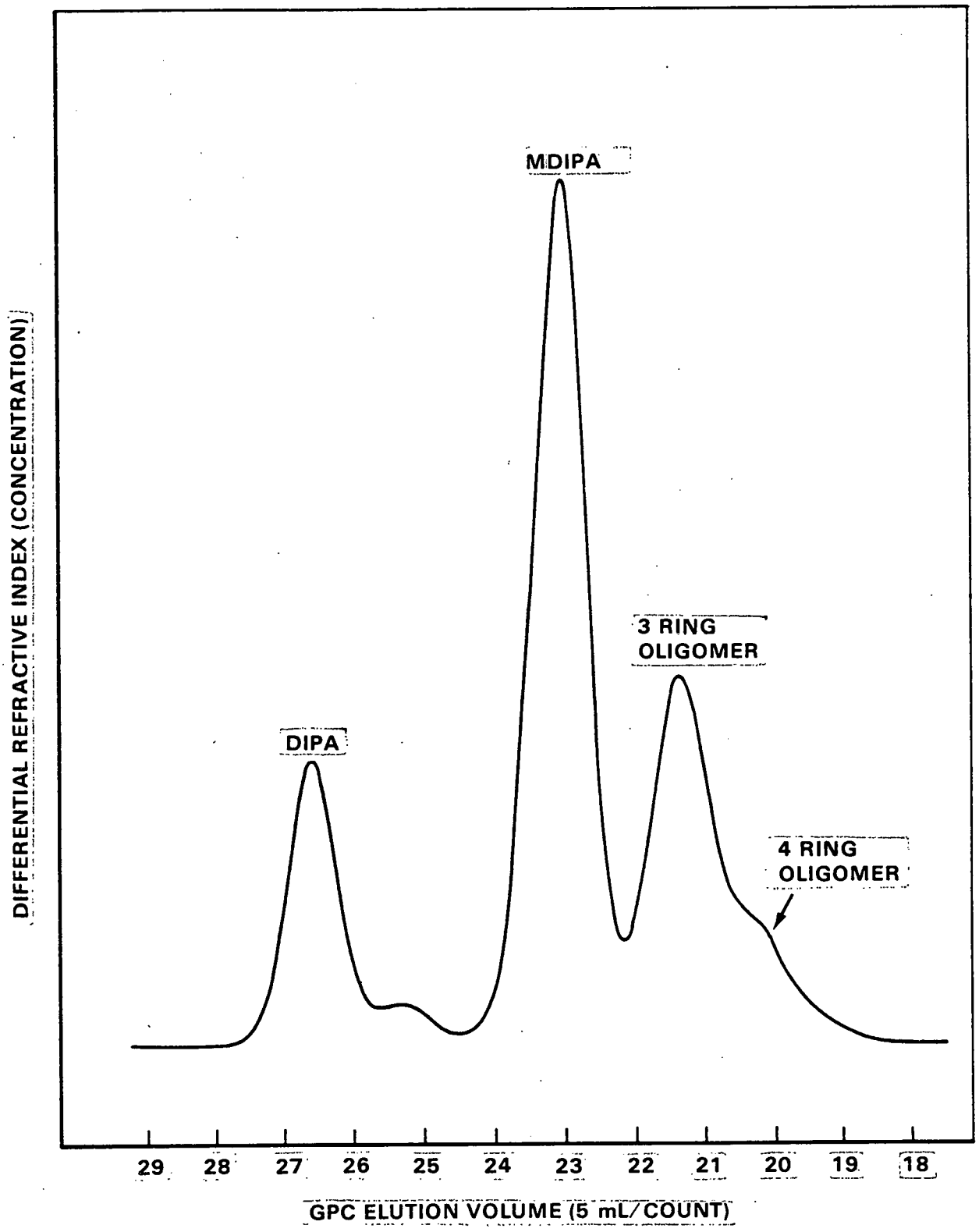


Figure 7. Gel Permeation Chromatogram of MDIPA, Batch 3

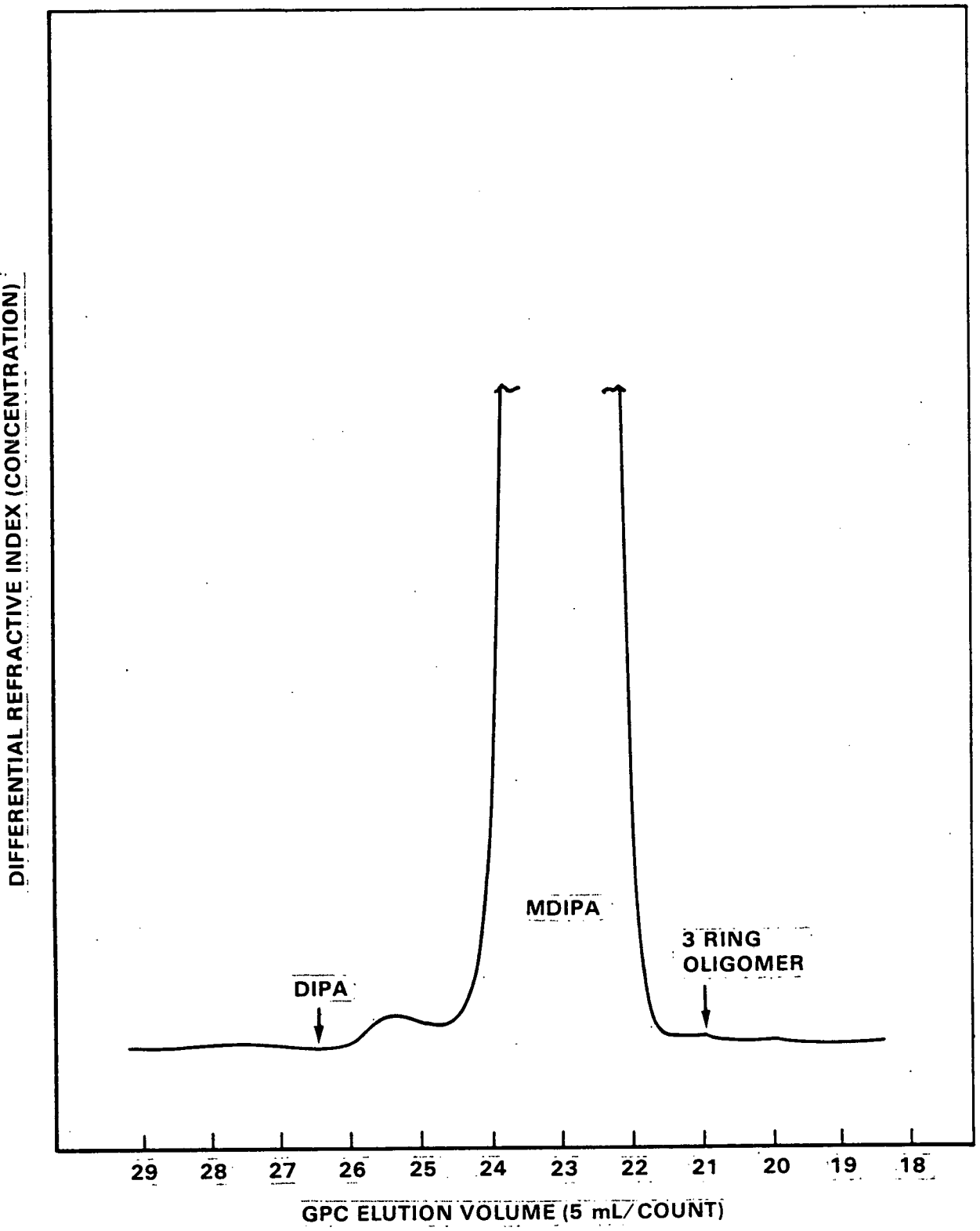


Figure 8. Gel Permeation Chromatogram of MDIPA

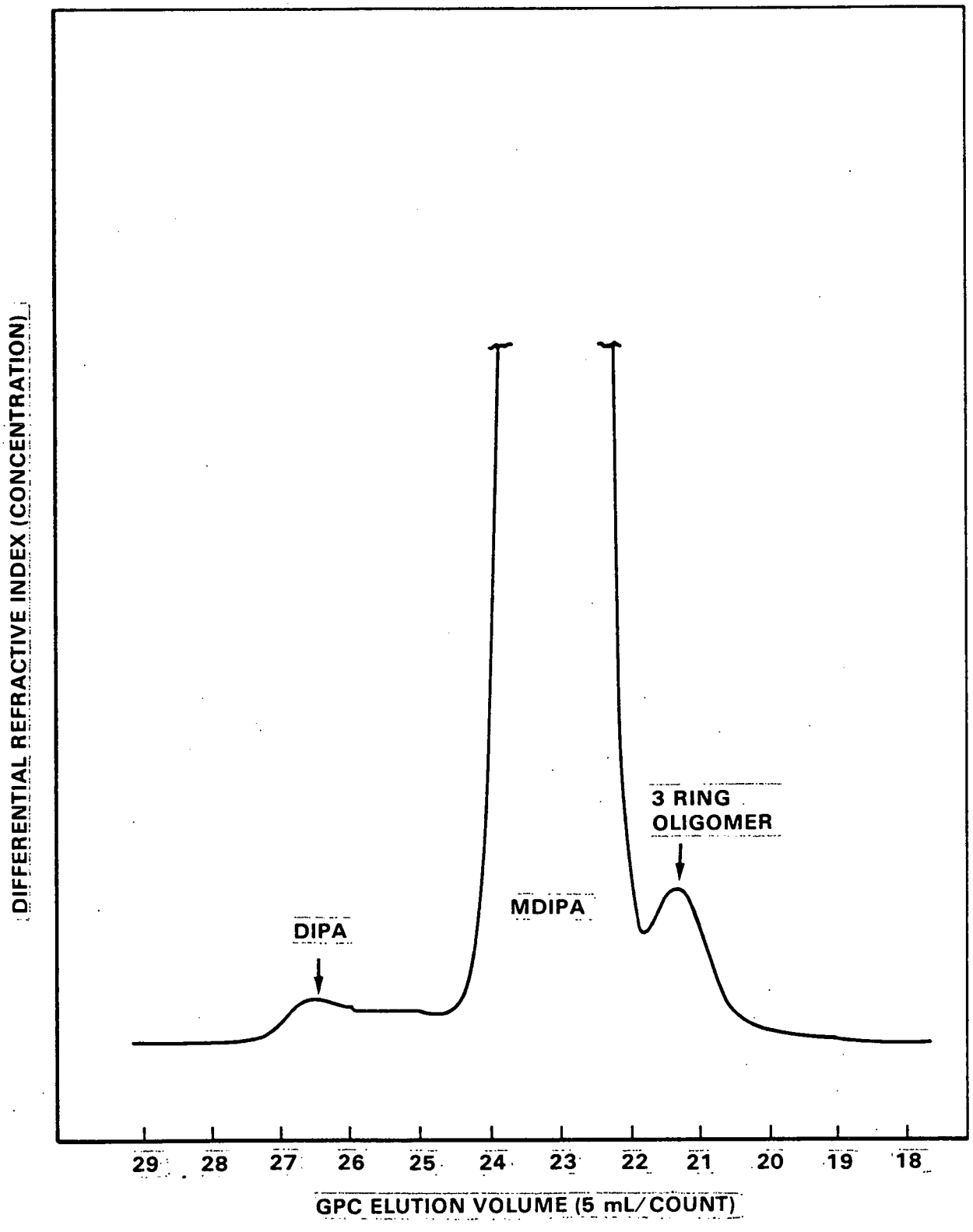


Figure 9. Gel Permeation Chromatogram of MDIPA, UCC/Y-12 GFD 15-62

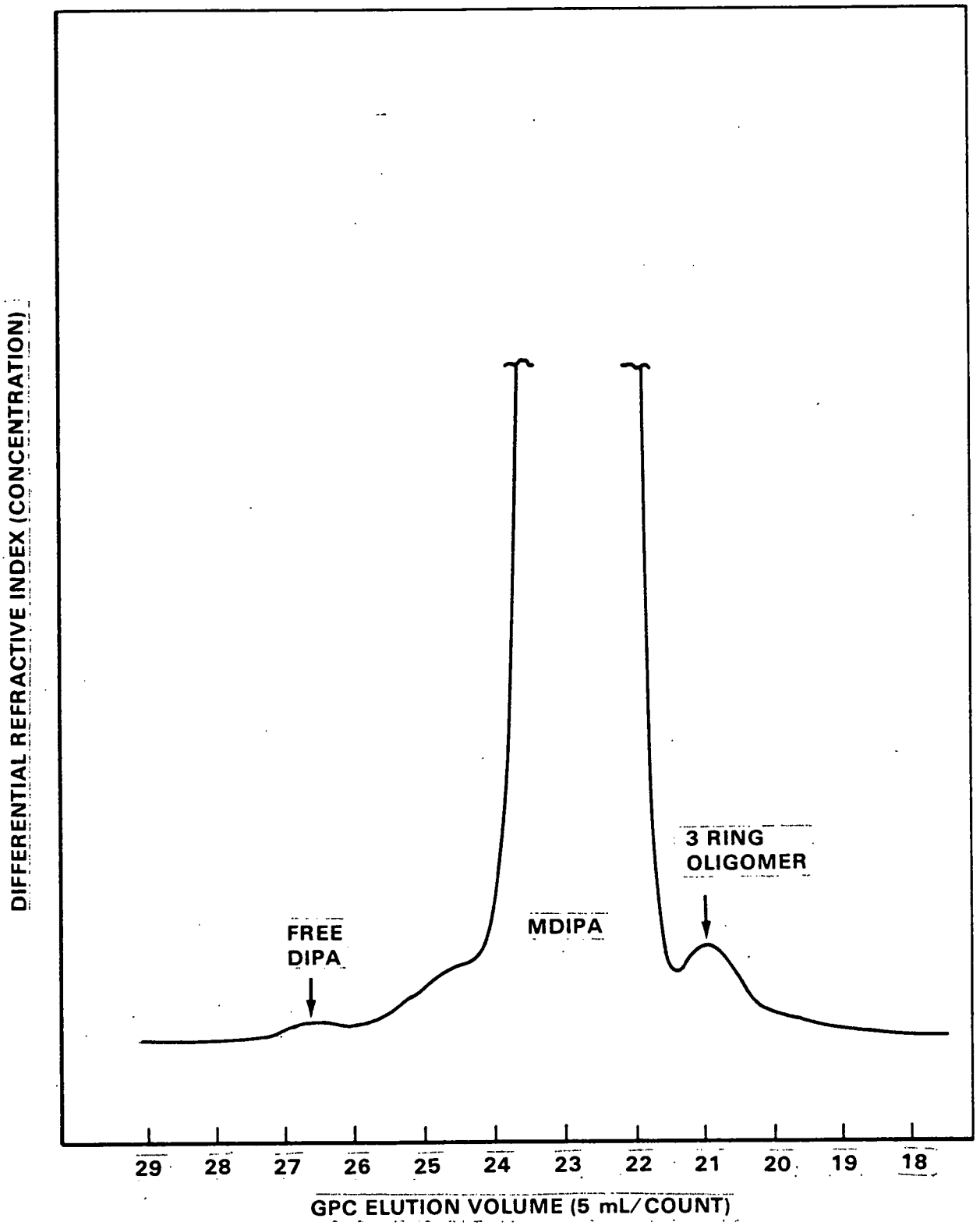


Figure 10. Gel Permeation Chromatogram of MDIPA, PP-1

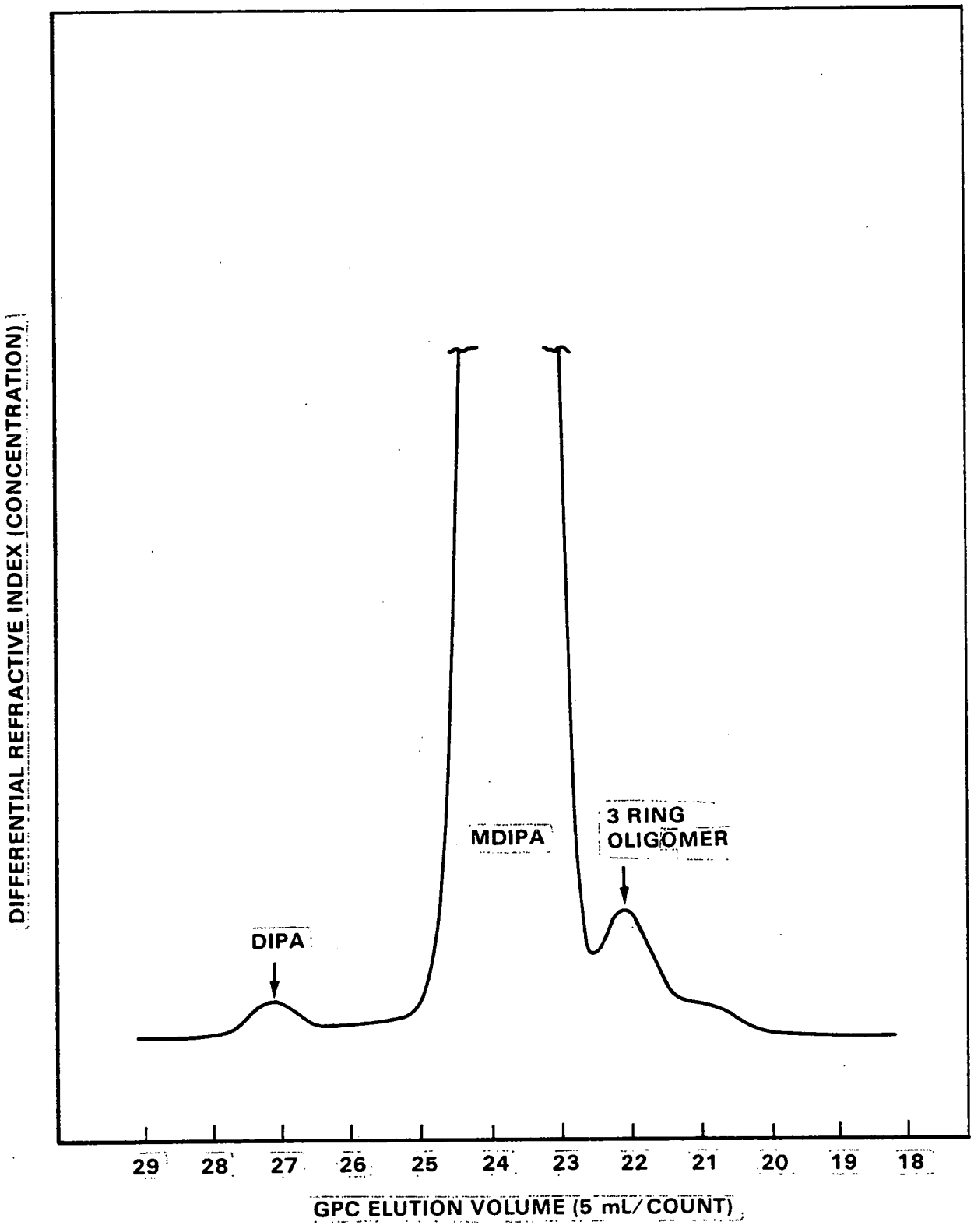


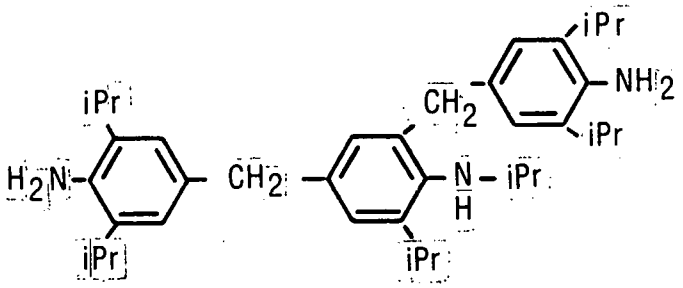
Figure 11. Gel Permeation Chromatogram of MDIPA, PP-2

Table 1. Properties and Analysis of MDIPA

	Melt Point (°C)	GPC Analysis, Percent		
		DIPA	MDIPA	3-ring
Original	66 to 68	0.11	99.8	0.1
GFD/Y-12, 15-62	52 to 55	0.67	97.0	2.3
Batch 3*	Liquid	17.3	53.3	22.4
Batch 12	52 to 58	2.74	96.0	1.3
Batch 12A**	63 to 65	0.47	98.4	1.1
Batch 13	59 to 61	1.36	97.6	1.0
Batch 14	52 to 56	2.61	95.9	1.5
Batch PP-1	66	0.24	98.6	1.2
Batch PP-2	53 to 56	1.08	95.4	3.5

*Contained ~7 percent 4 phenyl ring oligomer
**Recrystallized Batch 12

small amounts in the MDIPA product should not be deliterious in urethane curing reactions.



ACCOMPLISHMENTS

Laboratory experimentation has provided the required parameters for successful synthesis of the MDIPA diamine cure agent. The synthetic method, stoichiometry, reaction times and temperatures, product recovery, and purification procedures have been established and successfully transferred to the Polymer Pilot Plant for scale-up to provide kilogram quantites of MDIPA.

Test procedures, primarily gel permeation chromatography, have been developed to monitor product quality. Upon request, small quantities of MDIPA product have been distributed to materials and engineering personnel at LANSL, LLNL, UCC/Y-12, and GEND for further evaluation as a curative in polyurethane adhesives.

FUTURE WORK

To satisfy LLNL requirements that the MDIPA product synthesized by Bendix is equivalent to the previously available product it will be necessary to demonstrate equivalency in properties for Halthane 87 cured with both MDIPA materials. Another project is underway to characterize the Halthane 87/MDIPA system using viscosity versus time, butt tensile strength, hardness, shear modulus, solvent swell, and residual functional group analysis.

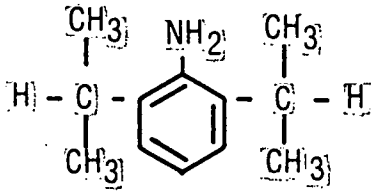
As a spin-off, there will most likely be future interest in other aromatic diamines as polyurethane curatives, particularly as more and more specific aromatic diamines fall under OSHA restrictions. The technology to synthesize and the capability to produce this type of fine organic compound is now available and can be easily applied to other diamines.

REFERENCE

[1]B. G. Parker, Preparation of Urethane Adhesives (Final Report). Bendix Kansas City: BDX-613-1549 (Rev.), September, 1976 (Available from NTIS).

APPENDIX

PROPERTIES OF 2,6-DIISOPROPYLANILINE



Form	Clear Liquid
Molecular Weight	177.3
Freezing Point	-45°C
Boiling Point	257°C
Flash Point (COC)	124°C
Density (20°C)	0.940 g/cm^3
Refractive Index, n_D^{20}	1.5332
Vapor Pressure, 180°C	100 mm Hg
Vapor Pressure, 120°C	10 mm Hg
Vapor Pressure, 80°C	1 mm Hg
Elemental Formula	$C_{12}H_{19}N$
Carbon	81.3 percent
Hydrogen	10.8 percent
Nitrogen	7.9 percent
Water Solubility, 20°C	0.02 percent

Completely miscible in ethanol, iso-octane, and toluene. Freshly distilled product is almost colorless, but usually is pale yellow.

BDX-613-2509

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PLASTICS: Cure Agents

SYNTHESIS OF MDIPA, AN ARMOATIC DIAMINE CURATIVE FOR POLYURETHANE ADHESIVES, F. N. Larsen, Final, BDX-613-2509, September 1980

The compound 4,4'-methylene bis-(2,6-dissopropylaniline), or MDIPA, has shown good thermal stability, high butt tensile strength, and excellent compatibility with salt when used as a curative for LLL's Halthane 87 prepolymer. Laboratory-scale synthesis of MDIPA by condensation of commercially-available 2,6-diisopropylaniline and formaldehyde has been accomplished, using the amine sulfate as an intermediate. Polymer Pilot Plant scale-up has been achieved; kilogram quantities at acceptable purity levels (98+ percent) have been produced.

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