Honeywell

Developing the Manufacturing Process for Hylene MP Curing Agent

Federal Manufacturing & Technologies

E.A. Eastwood

KCP-613-8526

November 2008

Final Report

Prepared under prime contract DE-ACO4-01AL66850 for the
United States Department of Energy

DISCLAIMER

A prime contractor with the United States Department of Energy under Contract Number DE-AC04-O1AL66850

Honeywell Federal Manufacturing & Technologies
P.O. Box 419159
Kansas City, Missouri, 64141-6159

Honeywell

KCP-613-8526
Distribution Category UC-42

Developing the Manufacturing Process for Hylene MP Curing Agent

E.A. Eastwood
November 2008

Final Report
D.E. Bowen, Project Leader

Project Team:
Eric Eastwood
David Irwin

Contents

Illustrations

Tables

Abstract

This report details efforts to scale-up and re-establish the manufacturing process for the curing agent known as Hylene MP. First, small scale reactions were completed with varying conditions to determine key drivers for yielding high quality product. Once the optimum conditions were determined on the small scale, the scaled-up process conditions were determined. New equipment was incorporated into the manufacturing process to create a closed production system and improve chemical exposure controls and improve worker safety. A safe, efficient manufacturing process was developed to manufacture high quality Hylene MP in large quantities.

Summary

This study is part of a project to re-establish the manufacturing process for diphenol-4,4'-methylenebis (phenylcarbamate), which is known as Hylene MP. Hylene MP is used in filled elastomer parts to cure ethylene/vinyl acetate/vinyl alcohol terpolymer (VCE). Upon heating, VCE hydroxy groups on the vinyl alcohol monomers undergo a urethane type reaction with the Hylene MP to form a crosslinked network. First small scale reactions were completed in 2L reactors with varying conditions to determine key drivers for yielding high quality product. The temperature used to melt/dissolve methylenebis (4-phenyl-isocyanatate) (MDI) in toluene was determined to be an important factor in yielding conforming Hylene MP. Heating MDI for 60 minutes at 150°F resulted in MDI dimerization and a failed TGA result. Thus, the temperature used to melt/dissolve MDI in toluene was lowered to 110°F which eliminated the negative TGA result. The addition rate of the MDI/toluene to the reaction was determined to be another important factor influencing the Hylene MP purity. Faster addition rates and the resulting faster temperature increase yielded Hylene MP product with low purity. Addition rates that gave temperature increases lower than 1.6°F/min were successful at producing high purity material. Another factor that was determined to influence material purity was incorporation of a re-slurry step. After the Hylene MP is formed and filtered, the product is returned to the reactor and re-slurried with toluene to remove any unreacted MDI, unreacted phenol, or TMBDA catalyst. This step was shown to improve purities and thermal stability. Once the optimum conditions were determined on the small scale, production processes to manufacture Hylene MP on larger scales were developed. New equipment was incorporated into the manufacturing process to create a closed production system and improve chemical exposure controls and improve worker safety. Using a 50 gallon reactor, Nutsche Filter Dryer, 5 gallon APC tank, and a Comil 197 Lab Model Grinder (to grind/mill and package), a safe, efficient manufacturing process was developed to manufacture over 70 pounds of high quality Hylene MP by two full scale batches.

Discussion

Scope and Purpose

This report details efforts to scale-up and re-establish the manufacturing process for Hylene MP curing agent. Small scale reactions (260 gram batches in 2L reaction vessels) were completed with varying conditions to determine key drivers for yielding high quality product. Once the optimum conditions were determined on the small scale, the scaled-up process conditions using a 50 gallon reactor, Nutsche Filter Dryer, and 5 gallon APC tank were developed. A Comil 197 Lab Model Grinder, purchased from Quadro, was used to grind and package Hylene MP powder, which significantly reduced the amount of dust generated and processing time.

Activity

Background

Hylene MP is a white powder originally sold by DuPont. It is a bis phenol adduct of methylene bis (4-phenyl-isocyanatate) with the chemical name diphenol-4,4'-methylenebis (phenylcarbamate). The chemical structure for Hylene MP is shown in Figure 1.

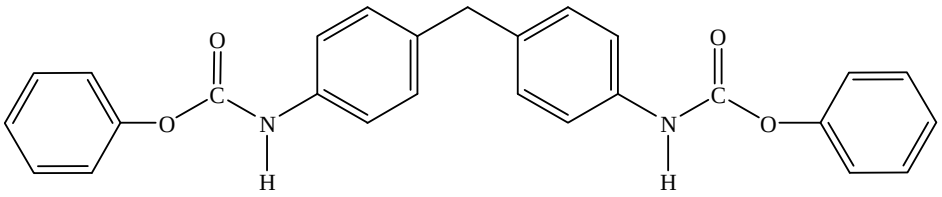

Figure 1. Hylene MP, diphenol-4,4'-methylenebis (phenylcarbamate).

Diphenol-4,4'-methylenebis (phenylcarbamate) is a blocked diisocyanate which is prepared by reacting phenol with methylenebis (4-phenyl-isocyanatate) (MDI), in the presence of an amine catalyst as shown in Figure 2. The catalyst used in this reaction is N, N, N', N'-tetramethyl-1,3 butanediamine (TMBDA) and the reaction is completed in toluene. Toluene dissolves all of the reactants and catalyst, however, the final product is insoluble and will fall out of solution once formed. Hylene MP is used as the curing agent for the ethylene/vinyl acetate/vinyl alcohol terpolymer known as VCE. Upon heating, VCE hydroxy groups on the vinyl alcohol monomers undergo a urethane type reaction with the Hylene MP to form a crosslinked network. Previous reports have detailed the synthesis and curing of VCE terpolymer with Hylene MP.[2]

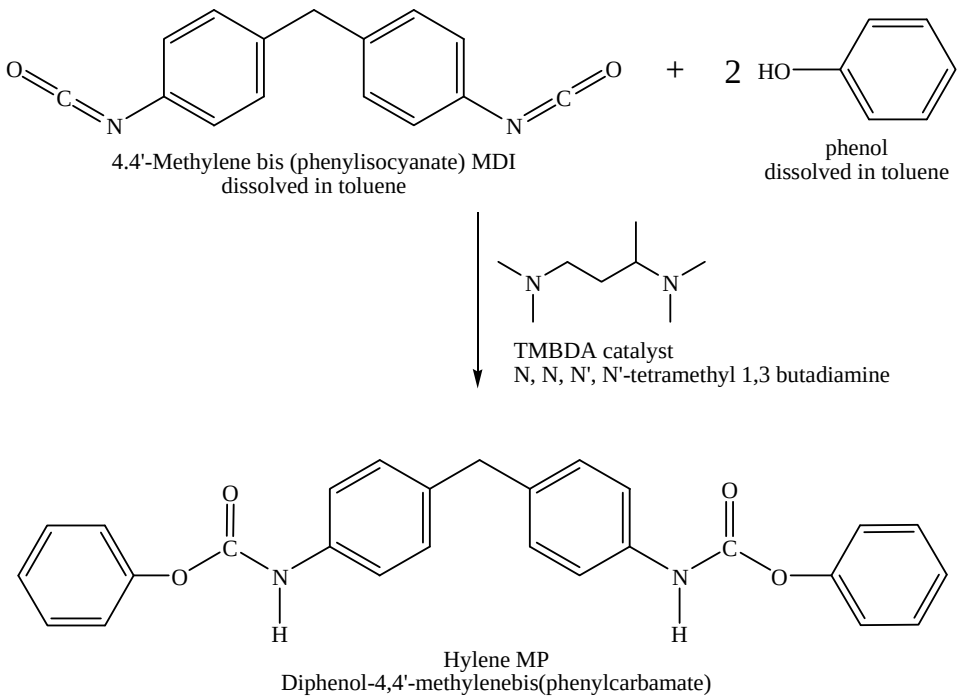

Figure 2. Formation of diphenol-4,4'-methylenebis (phenylcarbamate).

Initial Characterization

Characterization data for different lots of Hylene MP were reported in previous reports.[3,4] The current report was used to generate the testing requirements for quality verification of new lots of Hylene MP manufactured under the seven-digit material specification 4162014. Gel permeation chromatography (GPC) was used to determine purity; differential scanning calorimeter (DSC), thermogravimetric analysis (TGA), and thermogravimetric analysis coupled with mass spectrometry (TGA/MS) were used to determine thermal properties; and nuclear magnetic resonance (NMR) was used to verify structure, estimate purity, and determine impurities for multiple lots of Hylene MP. The requirements for material specification 4162014 were designed to exclude Hylene MP with behavior similar to Lot 040421. This lot has the lowest purity, lowest melting point, and gave a much different thermal decomposition profile than the rest of the lots.

Manufacturing Process Development

Reaction #1 – 2L Reaction, Heated MDI

The first reactions were completed on a small scale in 2L glass reaction vessels with a target theoretical yield of 260 grams. Report BDX-613-1796, "Preparation and Properties of Blocked Diisocyanates", was used as guide for experimental conditions for the laboratory scale reactions.[1] The actual material ratios and amounts used were based on process specification P1270085 and shown in Table 1.

Table 1. Ratios and weights used in 2L Hylene MP batches.

Material	Material Specification #	Parts By Weight	Weight (grams)
MDI	4612129	100	140.5
Toluene for MDI	4612021	43.5	61.1
Phenol	4619061	85	119.5
Toluenefor Phenol	4612021	850	1195
TMBDA	4604247	2	2.8

The 2 liter glass reactor was charged with 1195 grams of toluene and 2.8 grams of TMBDA. 140.5 grams of phenol was melted at 150°F and mixed in with the toluene and TMBDA solution. At 150°F, it took approximately 60 minutes for the phenol to completely melt. 140.5 grams of MDI and 61.1 grams of toluene were mixed together and placed in an oven at 150°F to speed up dissolution. The MDI/toluene solution was heated for 60 minutes until the MDI was completely homogeneous and then transferred to a one liter addition flask. While vigorously stirring the phenol/TMBDA/toluene mixture, MDI/toluene was slowly dripped into the 2L reaction vessel. The MDI/toluene solution volume and temperature was monitored throughout the addition and is shown in Figure 3. It took 25 minutes to add the volume of MDI/toluene solution (~200 mL) to the phenol/TMBDA/toluene mixture, which was an average rate of addition of 8mL/min. The temperature increased 48.5°F to a maximum temperature of 119.2°F which occurred at the 30 minute mark. This corresponded to an average temperature increase of 1.6°F/min.

Once all of the MDI solution was added, the mixture was allowed to stir for an additional 60 minutes to insure complete reaction. The product slurry was removed from the 2L reaction vessel and filtered with a Büchner filter funnel on filter paper. The product was rinsed several times with toluene while on the filter paper. The filtered product was then returned back into the 2L reaction vessel with an additional 1200 grams of toluene. The product was allowed to stir for another hour. The purpose of this step was to remove any remaining unreacted MDI, unreacted phenol, or TMBDA. The re-slurried product was again filtered with a Büchner filter funnel and rinsed several times with toluene while on the filter paper. The wet Hylene MP product was then dried under vacuum at 165°F until dry. The material from Reaction #1 was given lot number

070518 and tested for quality and conformance to 4162014 material specification. The data is compiled in Table 2 with non-conforming data highlighted in red. As can be seen in Table 2, the

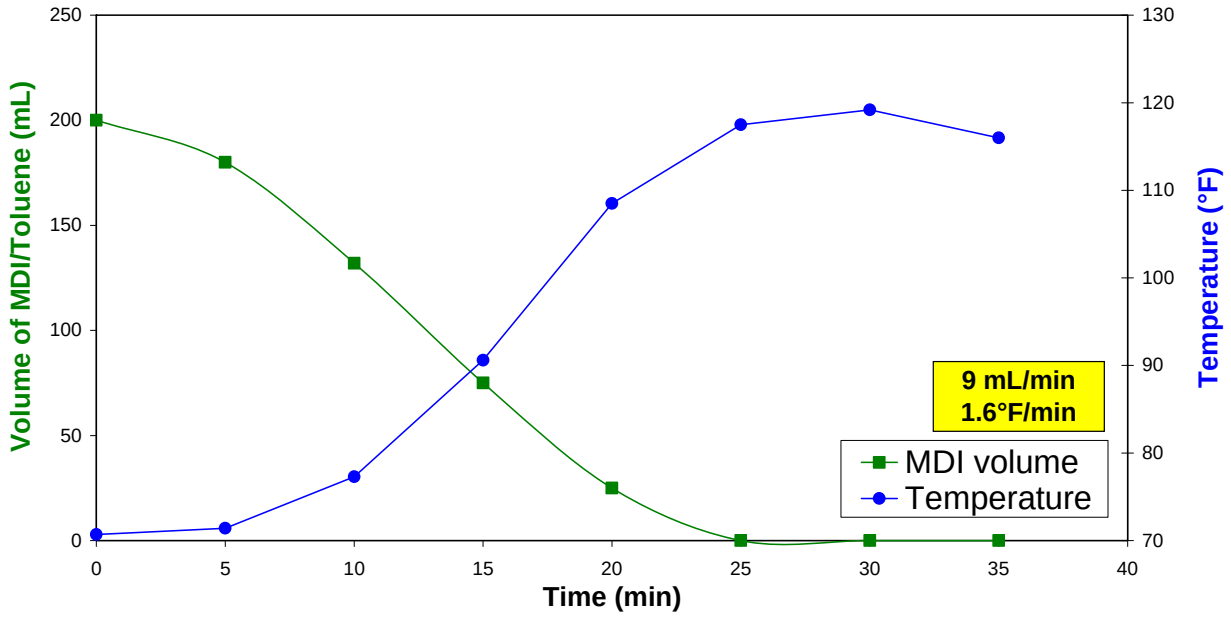

Figure 3. Reaction #1 MDI/toluene volume and temperature data.

purity by GPC, melting point by DSC, and weight percent at 160°C conform to the established requirements; however, the weight percent at 275°C for Lot 070518 was too high (with a value of 51.9% vs. the 25% maximum allowed). Figure 4 shows the TGA data of Lot 070518 along with some of the previous lots analyzed and reported in KCP-613-8192 report.

The requirements of material specification 4162014 were chosen to reject material with data similar to Lot 040421 (red line). Lot 070518 performs very well up to 250°F, however above that temperature, it performs like Lot 040421 resulting in a non-conforming weight % of 51.9% at 275°C. MDI is very reactive and normally stored at sub-ambient conditions to prevent the dimerization reaction from occurring. The bottle of MDI used in this reaction was used previously and thus warmed up to room temperature and opened. One proposed source of the failed TGA requirement was contaminated or degraded MDI by this dimerization process. A new bottle of MDI was sent along with a sample from the first bottle used to the analytical lab for GC/MS analysis to determine purity. The GC/MS data given in Table 3 shows very little difference between the new, unopened bottle of MDI and the older bottle used in the reaction indicating that this was not the source of failure.

Table 2. Characterization data for Lot 070518.

Lot	Purity by GPC	Melting Point (°C) by DSC	Wt% at 160°C by TGA	Wt% at 275°C by TGA
Requirements	**95% minimum**	**190 °C minimum**	**97% minimum**	**25% maximum**
070518	95.6%	194.6 °C	99.96%	51.9%

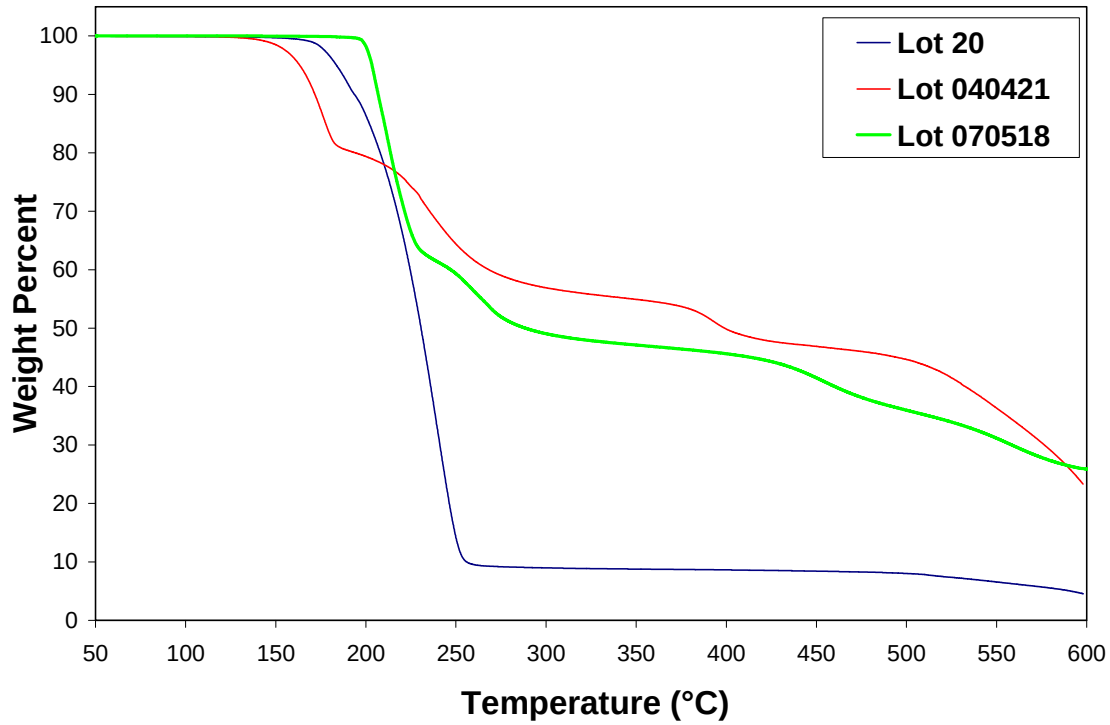

Figure 4. TGA decomposition traces of various Hylene MP lots; Reaction #1- Lot 070518, conforming material – Lot 20, and non-conforming material – Lot 040421.

Table 3. MDI purity by GC/MS.

	MDI	**Dimer**
New Bottle	98.7%	1.3%
Old Bottle	98.6%	1.4%

Another experiment was completed on the MDI to determine how heating the material during dissolution at 150°F effects the purity and dimer content. Two samples of MDI (from the new bottle) were heated at 150°F for 30 minutes and 60 minutes; and then analyzed by GC/MS. This experiment recreated possible conditions the MDI was exposed to during Reaction #1. The GC/MS purities of the heated MDI samples are shown in Table 4. Heating MDI at 150°C for 30 minutes did not cause much change in purity; however, heating for a total of 60 minutes causes a significant reduction in purity and a large increase in the dimer content. To solve this problem, the time MDI is heated at 150°C could be limited or temperatures could be lowered. Since the MDI may not be completely dissolved if only heated for 30 minutes, the temperature will be lowered to 110°F for future batches. It's not clear why the purity of the final Hylene MP product is still 95.6% even though the MDI purity was likely lower due to the dimerization reaction occurring during heating at 150°F. Nonetheless, it appears the TGA requirement failure is due to the dimerization of the MDI during heating at 150°F and subsequent reactions used a much lower temperature of 110°F.

Table 4. MDI purity upon heating for 0, 30, and 60 minutes.

	MDI	Dimer	Other
Heated at 150°C for 0 min	98.7%	1.3%	NA
Heated at 150°C for 30 min	98.5%	1.3%	0.2%
Heated at 150°C for 60 min	87.2%	11.6%	1.2%

Reaction #2 – 2L Reaction, Faster Addition

The amounts and reaction conditions were the same as Reaction #1 except for the following changes. First, the MDI/toluene was placed in an oven at 110°F for dissolution instead of the 150°F temperature (due to the MDI dimerization just described). The MDI/toluene was added to the phenol/TMBDA/toluene mixture at a much faster rate. The 200 mL of MDI/toluene was added in 10 minutes to give an average addition rate of 20 mL/min. The rates of MDI addition for Reaction #2 and Reaction #1 are given in Figure 5. Because of the faster addition, the temperature increased at a faster rate with a value of 2.8°F/min (vs. 1.6°F/min), but the maximum temperature was only 112°F (vs. 119.2°F for Lot 07018). The temperature of Reactions #1 and #2 can be found in Figure 6. To determine if the re-slurry step was necessary, a sample was pulled after the first filtration and toluene rinses (prior to the re-slurry step) and vacuum dried and tested. The remaining material was processed as before. The analytical data for both samples from Lot 070606 are found in Table 5.

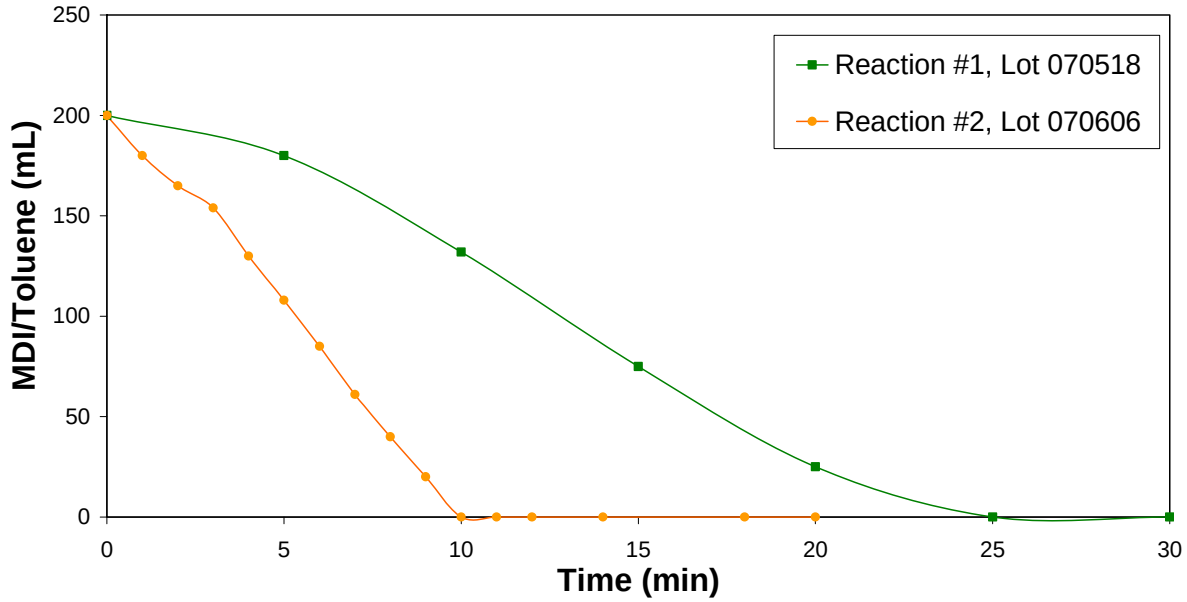

Figure 5. MDI/toluene volume data for Reactions #1 and 2.

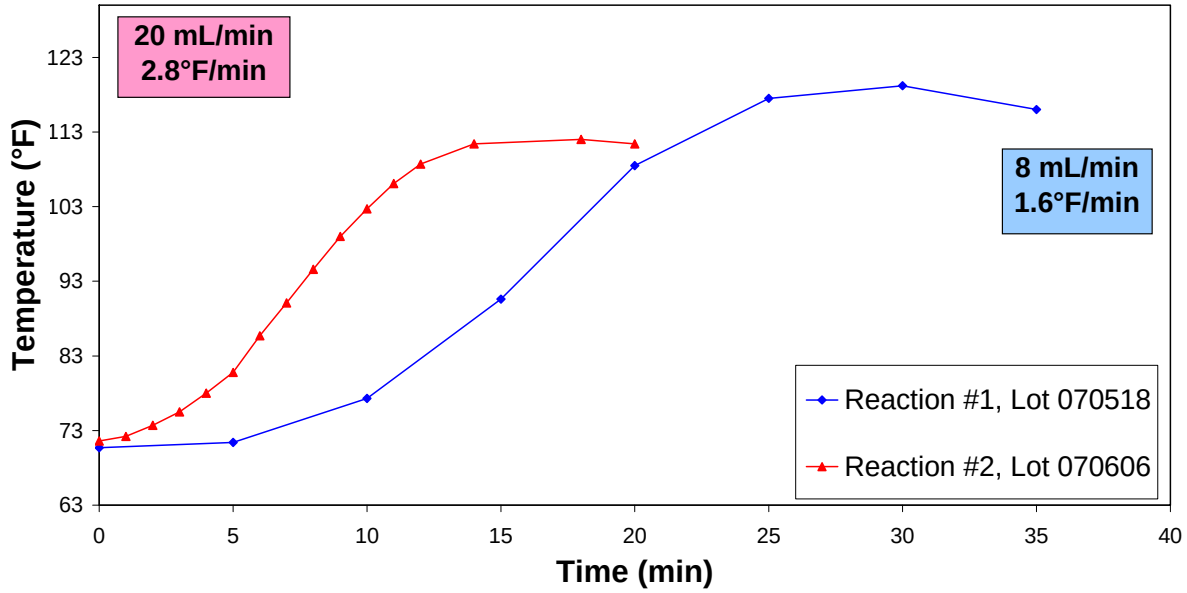

Figure 6. Temperature increase during Reactions #1 and 2.

Table 5. Characterization data for Lot 070606 (with and without re-slurry step).

Lot	Purity by GPC	Melting Point (°C) by DSC	Wt% at 160°C by TGA	Wt% at 275°C by TGA
Requirements	**95% minimum**	**190 °C minimum**	**97% minimum**	**25% maximum**
070606 without re-slurry	86.8%	194.2%	99.99%	7.6%
070606	92.7%	194.8%	99.97%	5.5%

Lot 070606 Hylene MP material conformed to all of the thermal property requirements (DSC and TGA requirements). Thus, it appears that by heating MDI/toluene to only 110°F rather than 150°F the failing TGA result observed in Reaction #1 was avoided. Unfortunately, the purity for Lot 070606, as determined by GPC, did not meet the 95% minimum requirement. Un-slurried material has a purity of 86.8% and a purity of 92.7% is found for re-slurried material. The lower purity could possibly be traced back to the faster addition of the MDI/toluene solution to the reactor. The re-slurry step effects both the purity and the TGA data. Both samples of Lot 070606 conform to the TGA decomposition limits, but the TGA data shown in Figure 7 indicates a more thermally stable material is obtained by completing the re-slurry step. Therefore, the re-slurry step should be completed during the production of Hylene MP.

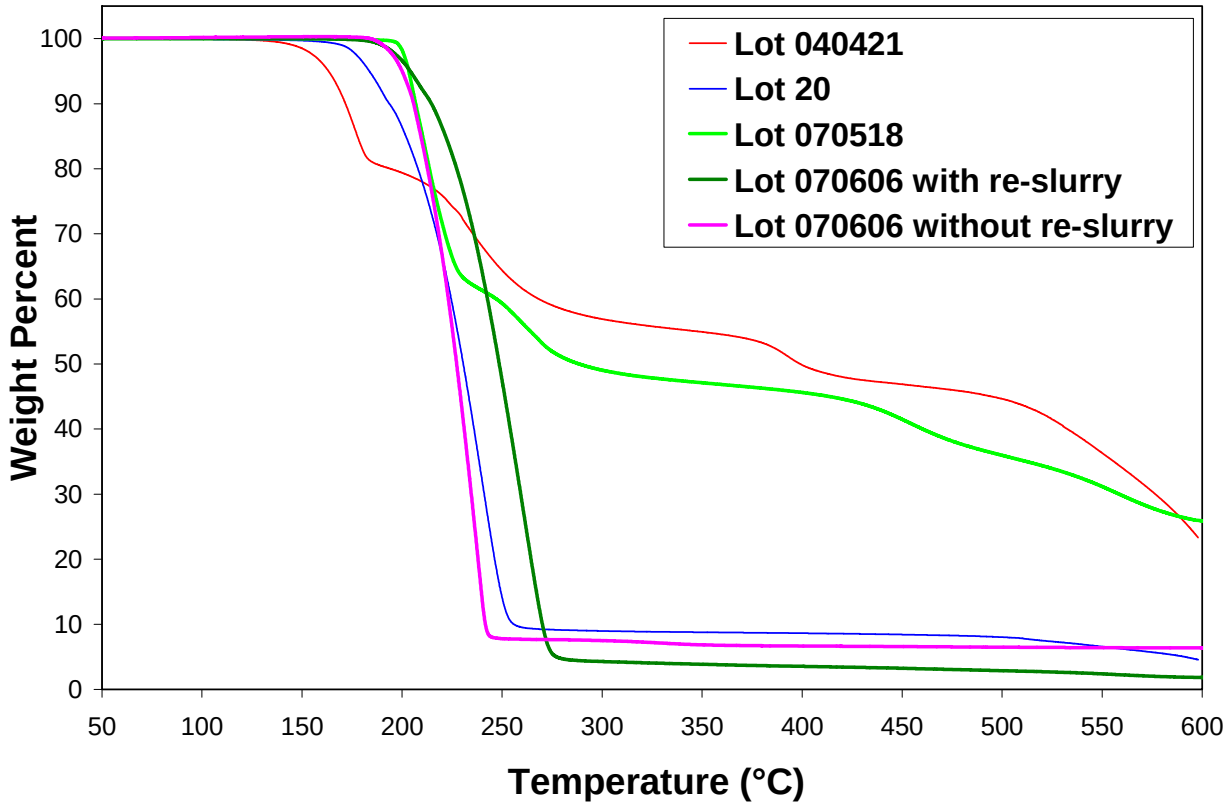

Figure 7. TGA traces of various conforming and non-conforming Hylene MP lots.

Reaction #3 – 2L Reaction, Optimum Conditions

For the third 2L reaction, the lessons learned from Reactions #1 and #2 were incorporated to Lot 070812. The amounts and reaction conditions were the same as Reaction #1 and #2 with the following specific conditions. The MDI/toluene solution was placed in an oven at 110°F for dissolution. The MDI/toluene was added to the phenol/TMBDA/toluene mixture at a rate used in Reaction #1, 8 mL/min. The target time to add the ~200 mL volume was 25-30 minutes. The re-slurry step was completed to further purify the material. All other processing conditions were completed as was done for Reactions #1 and #2. Table 6 provides the analytical data for Lot 070812 and indicates that the Hylene MP material conforms to all of the requirements. The optimum conditions used in Reaction #3 resulted in high quality product that completely conforms to the material specification requirements.

Table 6. Characterization data for Lot 070812.

Lot	Purity by GPC	Melting Point (°C) by DSC	Wt% at 160°C by TGA	Wt% at 275°C by TGA
Requirements	**95% minimum**	**190 °C minimum**	**97% minimum**	**25% maximum**
070812	96.4%	197.8°C	99.96%	5.7%

Reactions #4 and 5 – Full Scale Batches

The optimum conditions used in the Lot 070812 2L reaction were incorporated and used to manufacture two scaled-up lots in a 50-gallon reactor. This chemical process involves some very hazardous chemicals and the original production design did not have adequate controls for chemical exposure. The production process was redesigned to limit worker exposure to these chemicals and incorporate a closed system by using new equipment shown in Figures 8 and 9, the Nutsche Filter Dryer and the 5-gallon APC tank, respectively. The 50-gallon reactor was charged with approximately 200 pounds of toluene. For Lot 071203, six jars of phenol were weighed and placed in an oven at 150°F until melted. Each jar of phenol contains approximately 1.5 kg. The melted phenol was poured into the reactor and empty bottles were weighed again to determine the exact weight of phenol added into the reactor. Then the exact amounts of the other reactants were calculated from this value. The theoretical yield for this batch was determined to be 43.2 pounds. The TMBDA was weighed out and added to the phenol/toluene solution. The correct amounts of MDI and toluene were weighed out and charged into the 5 gallon APC tank. The 5-gallon APC tank was placed in an oven and heated to 110°F for 60 minutes to dissolve and melt the MDI. The rate of addition of the MDI/toluene solution via the pressurized APC tank is difficult to control, and from the 2L reaction experiments it is known that if the addition is too fast the purity of the resulting material will be effected. Since the reactor temperature has shown to correlate to the addition rate, and is much easier to monitor, the reactor temperature was tracked. There was 4 gallons (15 liters) of MDI/toluene added to the reactor in 30 minutes which give a rate of addition of approximately 0.5 L/min or 0.13 gal/min. The temperature data for Reactions #1, 2, and 4 are shown in Figure 10. The starting temperature of 64°F was slightly lower than previous 2L reactions. This lower temperature of the reactor was due to the fact that the toluene drums were left outside overnight and were much cooler than toluene used previously for 2L reactions. The temperature only increased 42°F to a maximum of 106°F with an average rate of 1.1 F/min. These values are lower than what was observed for any of the other 2L reactions completed.

Once all the MDI/toluene was added to the reactor, the mixture was allowed to stir for an additional 60 minutes to insure complete reaction. The product slurry was transferred to the Nutsche Filter Dryer for filtering and washing. The Nutsche Filter Dryer was equipped with a 40 micron filter media. The filter media used was made from 316 stainless steel with a 50 x 250 mesh size (0.0049” x 0.0045” wire diameter) with plain dutch weave to give the 40 micron rating. The Hylene MP was filtered in the Nutsche Filter Dryer until most of the liquid was removed. Approximately 14 gallons (100 pounds) of toluene was charged into the 50 gallon reactor and then transferred to the Nutsche Filter Dryer to wash the product and clean out any remaining material in the lines and on the reactor walls to the Nutsche Filter Dryer. The product was then filtered again until most of the liquid was removed. With the discharge valve on the Nutsche Filter Dryer closed, an additional 28 gallons (200 pounds) of toluene was charged into the Nutsche Filter Dryer (again through the 50-gallon reactor). To re-slurry, the agitator was turned on and the product was allowed to stir for an additional hour in the Nutsche Filter Dryer. Again, the product was filtered until most of the liquid was removed. 14 gallons (100 pounds) of toluene was sprayed onto the product through the Nutsche Filter Dryer spray balls. After this final rinse, the product was filtered a final time until the majority of the toluene was removed from the solid product. While still in the Nutsche Filter Dryer, the Hylene MP product was dried

under a vacuum at 165°F for approximately 36 hours for Lot 071203 and at 180 °F for 36 hours for Lot 071217. A total of 36.5 pounds of Hylene MP was recovered (84.4% yield) for Lot 071203 The same reaction conditions were used for Lot 071217 except 7 bottles of phenol were used which resulted in a slightly higher theoretical yield of 50.2 pounds of Hylene MP. A total of 41.4 pounds (82.5% yield) of Hylene MP was recovered for Lot 071217.

Figure 8. Nutsche Filter Dryer from Pfaudler

Figure 9. 5 gallon APC tank

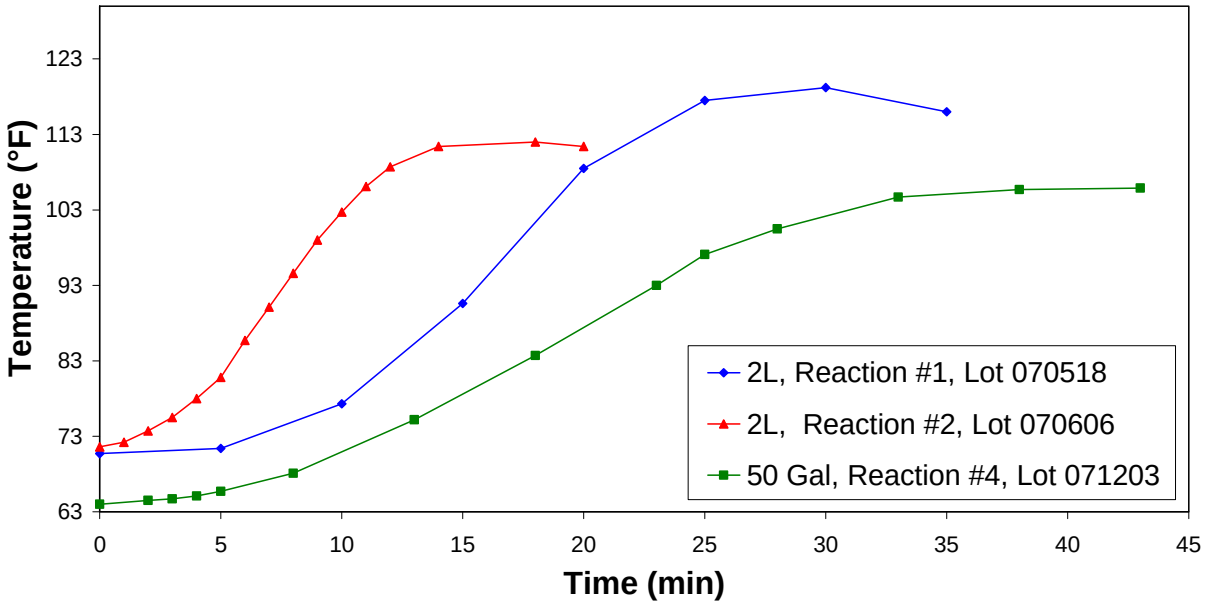

Figure 10. Temperature data for the full scale and 2L batches.

Characterization Data

The characterization data for all lots of Hylene MP analyzed, including scaled up Lots of 071203 and 071217, are compiled in Table 7 (with non-conforming values highlighted in red). The full scale production lots have some of the highest purities, highest nadir melt points, and most stable TGA decomposition traces of Hylene MP materials analyzed. Figure 11 is a bar chart plot of all the nadir melt points for all Hylene MP lots manufactured. Lots 071203 and 071217 have very high melt points compared to the rest of the lots produced. These two lots also have high purities as determined by GPC. The GPC purity data for all lots are shown in bar chart form in Figure 12. Figure 13 shows the TGA decomposition traces for Hylene MP lots manufactured and again the material manufactured using the scaled-up process is very thermally stable and meets all of the previously defined requirements.

Table 7. Characterization data for all Hylene MP lots manufactured.

Lot	Purity by GPC	Melting Point (°C) by DSC	Wt% at 160°C by TGA	Wt% at 275°C by TGA
040421	87.4%	186.6 °C	94.10%	59.0%
060124-1	95.9%	195.1 °C	99.80%	9.2%
060124-2	96.1%	192.8 °C	99.60%	9.3%
050823	96.1%	193.4 °C	99.40%	10.5%
20	96.2%	192.2 °C	99.30%	9.2%
17	98.6%	199.8 °C	99.60%	9.1%
070518	95.6%	194.6 °C	99.96%	51.9%
070606 without re-slurry	86.8%	194.2%	99.99%	7.6%
070606	92.7%	194.8%	99.97%	5.5%
070812	96.4%	197.8°C	99.96%	5.7%
071203	97.2%	200.1°C	99.86%	4.9%
071217	97.2%	198.8°C	99.88%	5.9%
Requirements	**95% minimum**	**190 °C minimum**	**97% minimum**	**25% maximum**

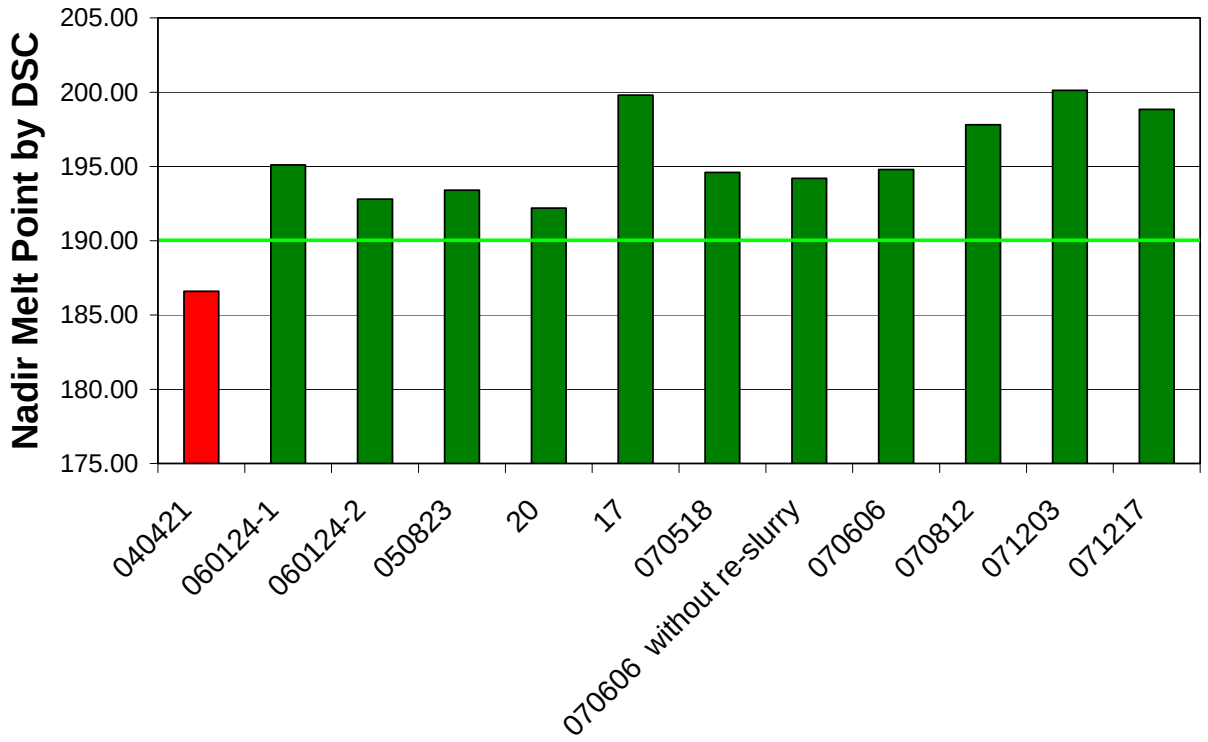

Figure 11. Nadir melt point by DSC for all Hylene MP lots manufactured.

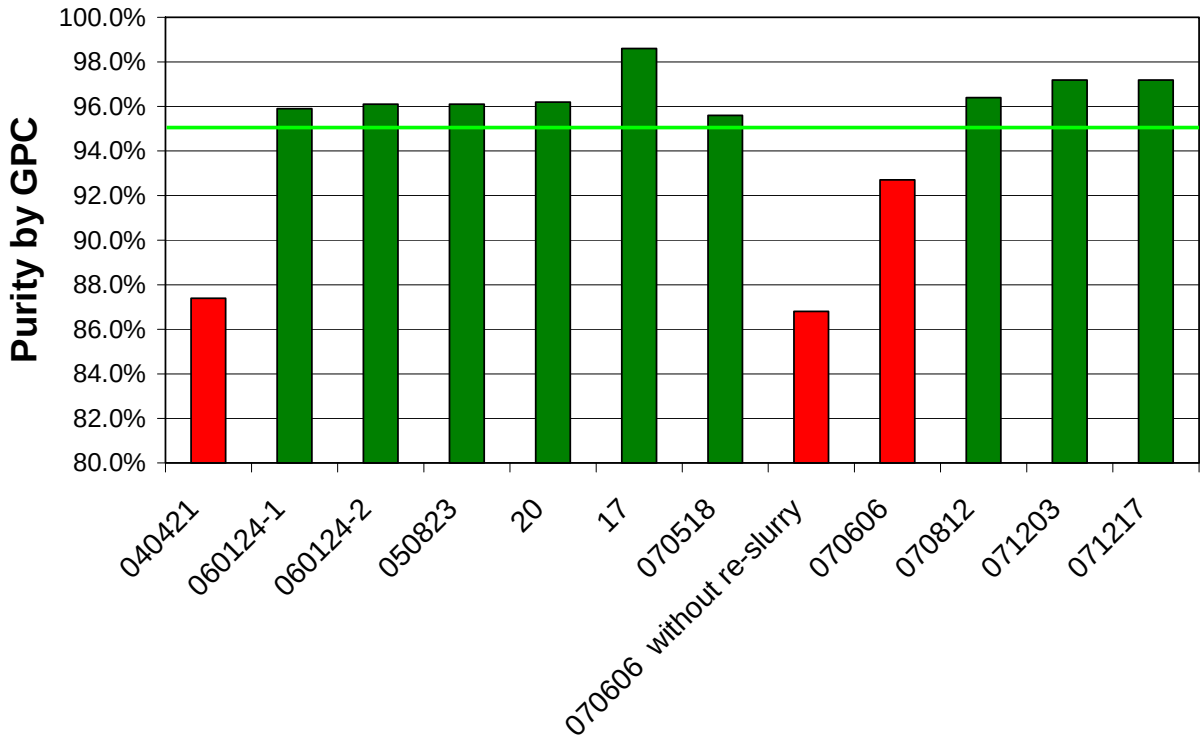

Figure 12. Purity of all Hylene MP lots manufactured as determined by GPC.

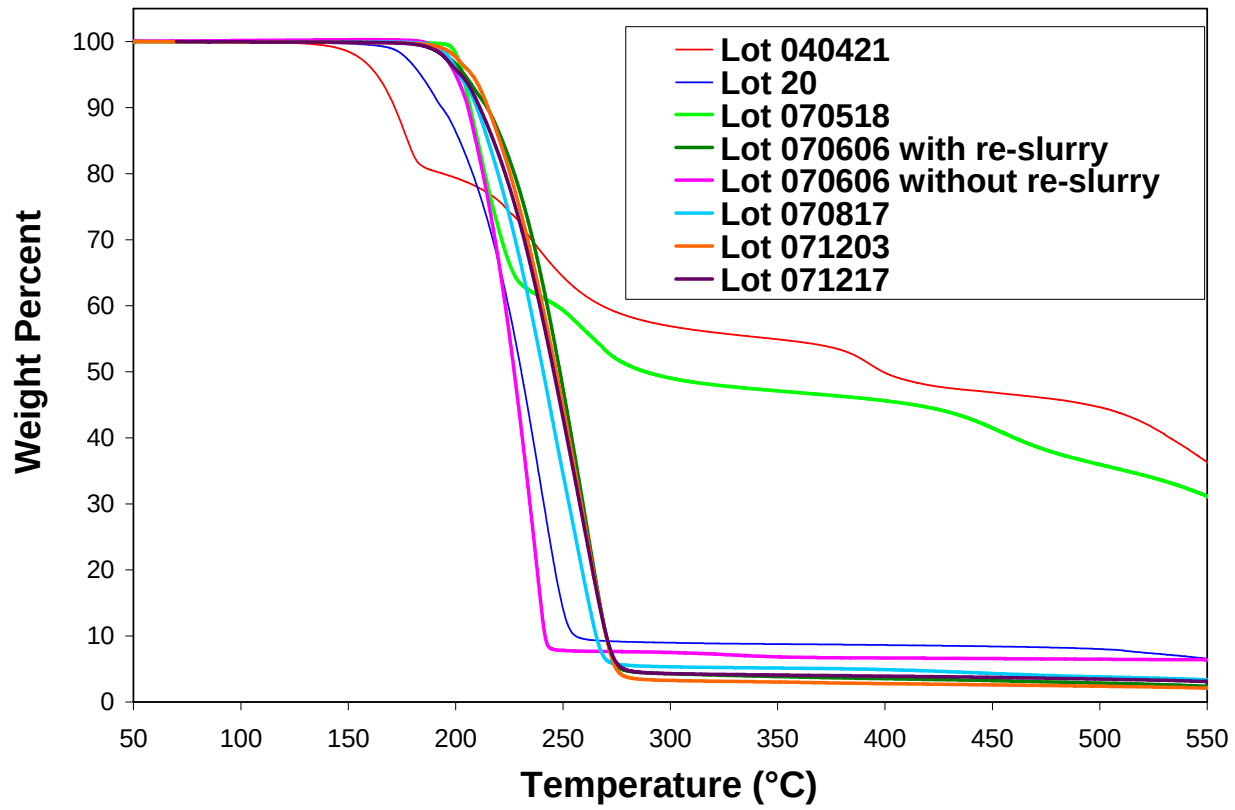

Figure 13. TGA decomposition traces for all Hylene MP lots manufactured.

Final Grinding and Packaging

Once the material was dry, it was discharged from the Nutsche Filter Dryer and stored in a fiber drum in polyethylene bags. A Comil 197 Lab Model Grinder was purchased from Quadro to grind/mill the Hylene MP and package in pint, amber glass jars. The unit purchased came with a Quick Release - “swing away” screen retaining shroud, 6"(152 mm) diameter with custom threaded connection. The custom thread connection fit a 53mm-400 thread, which is 53 mm diameter opening with a 400 GPI thread and allows the packaging jars to be screwed directly into the grinder. This feature significantly reduced the amount of dust generated and time required to package the Hylene MP. The Comil 197 Lab Model was equipped with a round hole (125R) screen (0.125” diameter holes) and standard 1607 square bar Impeller. Non contact parts were made of 304 stainless steel and the contact parts were 316L or 316 stainless steel. An image of the Comil 197 Lab Model Grinder purchased can be seen in Figure 16. This image also shows the amber glass jars screwed into the discharge and a small pile of white Hylene MP powder after grinding. The Comil 197 Lab Model Grinder was run at speed of 2600 rpm to process Lots 071203 and 071217 into a total of 134 pint jars. Lot 071203 was much easier to grind and the resulting powder was much less dense than Lot 071217. This result was likely due to the higher drying temperature used for Lot 071217 (180°F vs. 165°F). It was also observed that the product cake discharged from the Nutsche Filter Dryer for Lot 071217 was a much denser and harder product than Lot 071203 material. The reactor, Nutsche Filter Dryer, process lines, Comil 197 Lab Model Grinder, and other equipment were cleaned up with tetrahydrofuran.

Figure 14. Comil 197 Lab Model Grinder purchased from Quadro.

Melting or Dissociation

A study was completed to determine which peaks observed in the DSC curves are crystalline melt points or due to the thermal dissociation of the phenol (unblocking temperature). A heat/cool/heat DSC method was used to analyze Lot 071217 to answer this question. The sample was heated to 200°C, then cooled to -25°C, followed by a second re-heating cycle up to 250°C. If the first melt peak observed is due to the thermal dissociation of the phenol (unblocking temperature), then the material will have free MDI and phenol thermal behavior in the subsequent cooling and re-heating cycles. MDI has a melt point of 19-21°C and a boiling point of 106-107°C. Phenol has a melt point of 40-43°C and a boiling point of 180°C. Figure 14 shows the first heating cycle and the nadir melt point of 193.7°C. Figure 15 shows the subsequent cooling cycle and the re-heating cycle thermal data. The cooling cycle shows a large exothermic peak at 159°C indicative of a crystallization peak. The second heating cycle looks very similar to the first heating cycle with a nadir melt peak at 192.9°C. The cooling and second heating cycles do not show any thermal behavior indicative of free MDI or phenol. Therefore, it is concluded that the melt peak observed in the 190 to 200°C region is due to the melting of crystalline Hylene MP and not the thermal dissociation of the phenol. At temperatures greater than 200°C there is a second peak (or more commonly observed scattered peaks) which is likely due to the dissociation of phenol. Moreover, in the absence of any alcohol groups to react with, upon heating Hylene MP will first melt and then, at an higher temperature, unblock and "kick off" phenol.

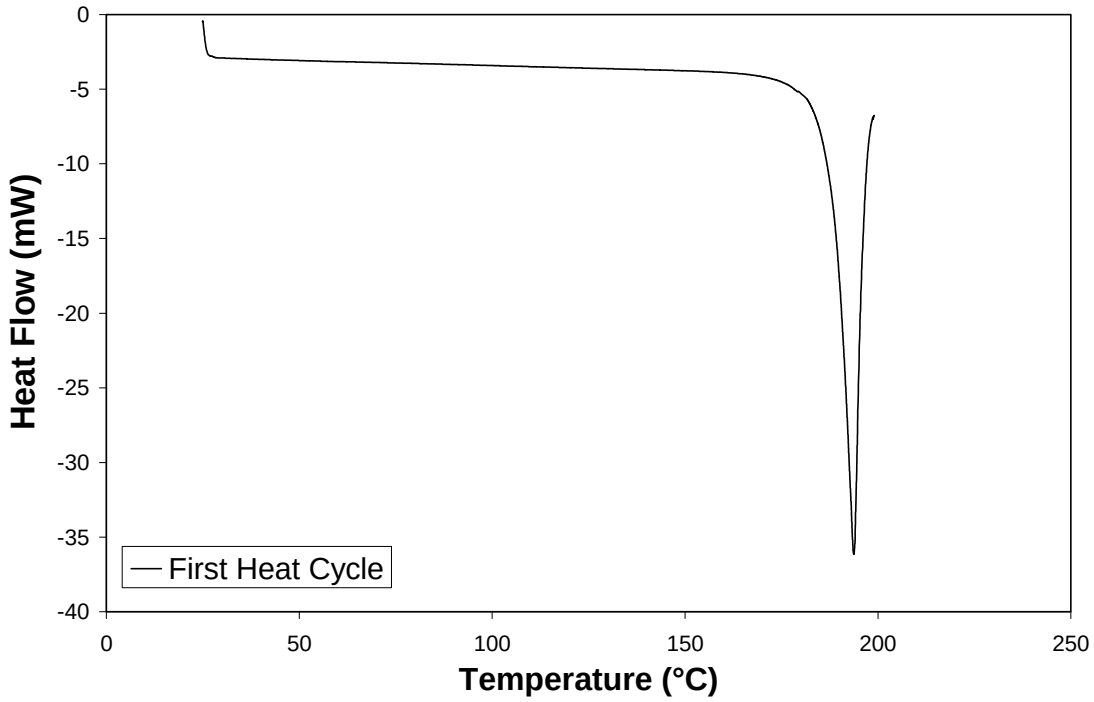

Figure 15. First DSC heat cycle of Lot 071217.

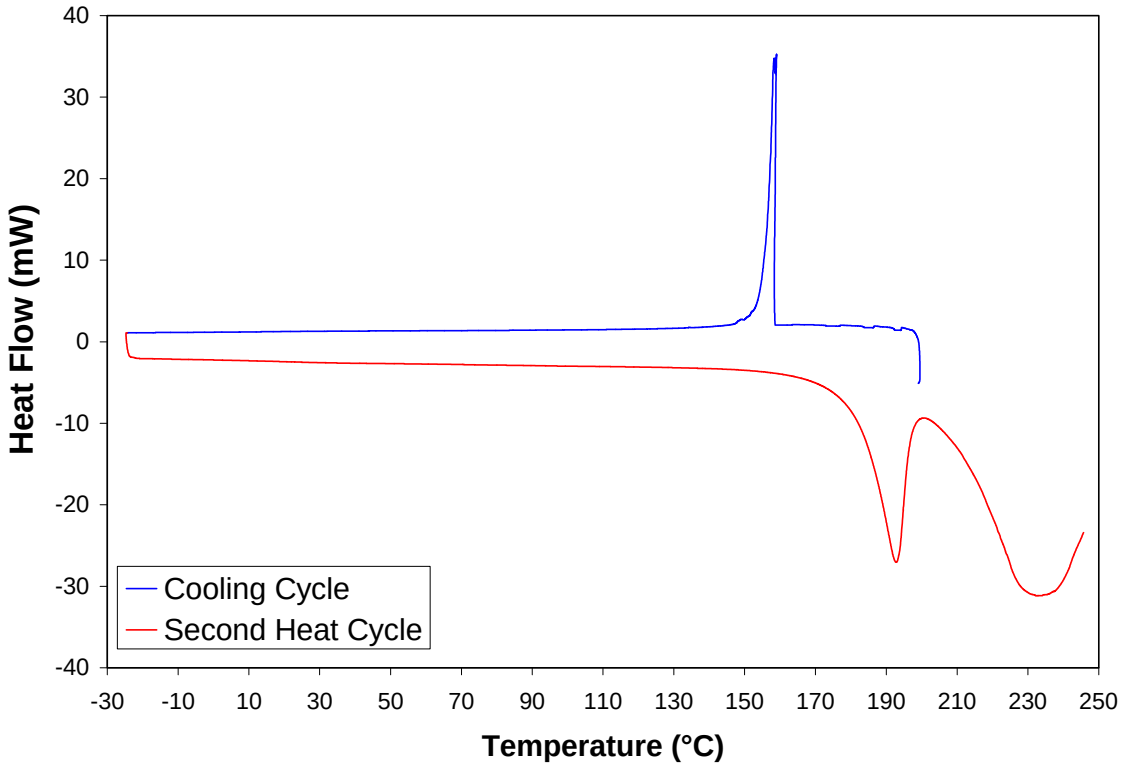

Figure 16. Cooling cycle and second heat cycle of Lot 071217 determined by DSC.

Accomplishments

The optimum reaction conditions were determined first on the small scale (260 grams batches in 2L reactor) and then on the larger batches (35-50 pound batches using 50 gallon reactor). In addition to the larger reactor used, the scaled up batches used the Nutsche Filter and a 5 gallon APC tank to incorporate a closed system to improve chemical exposure controls and worker safety. To further improve worker safety, a Comil 197 Lab Model Grinder was purchased and used to grind and package the Hylene MP powder. The Comil 197 Lab Model Grinder significantly reduced the amount of dust generated and processing time. Therefore, a safe, efficient manufacturing process was developed to manufacture high quality Hylene MP in large quantities.

References

1. B. G. Parker, *Preparation and Properties of Blocked Diisocyanates (Final Report).* UNCLASSIFIED. Federal Manufacturing & Technologies: BDX-613-1796, April 1977

2. E. A. Eastwood, *VCE Replacement Development: Curing Studies.* UNCLASSIFIED. Federal Manufacturing & Technologies. KCP-613-8161, July 2006.

3. Labouriau, A., Densmore, C., Meadows, K., Cordova, B., and Lewis, R., *Characterization of VCE and Starting Materials: Part 1*. LA-UR-06-5294. Los Alamos National Laboratory.

4. E. A. Eastwood, C. Densmore, *Characterization of Hylene MP: Curing Agent for Filled Elastomers*. UNCLASSIFIED. Federal Manufacturing & Technologies. KCP-613-8192.