TECHNICAL BULLETIN

Polyol HA-0135

Polyol HA-0135 Solvent-Free PUD Process For Soft-Feel Coatings

Polyol HA-0135 is a low molecular weight linear polyester polyol that contains primary hydroxyl and tertiary carboxyl groups in the terminal positions.

The fact that Dimethylol propionic acid is built into a polymeric polyol eliminates the crystalline properties associated with pure ***DMPA®;*** as a result, formulating low VOC PUDs with a solvent-free process is much easier.

At 25°C, ***Polyol HA-0135*** is a viscous liquid; however, when mild heat is applied, the viscosity of the resin decreases rapidly. ***Polyol HA-0135*** has a unique structure that provides excellent hydrolytic stability in PUD formulations.

To illustrate the benefits and properties of ***Polyol HA-0135*** in PUD formulations, a low VOC PUD formulation utilizing a solvent-free process is provided here.

*Typical Physical Properties **

Property	*Value*
Appearance	***Clear / lightly hazy viscous liquid***
Acid Value, mg KOH / g (as supplied)	***100 - 115***
Hydroxyl Number, mg KOH / g (as supplied)	***100 - 115***
Non-volatile, %	***98 min***
Brookfield Viscosity @ 100% Non-volatile	***@ 25°C 4000 - 8000 P @ 55°C 150 - 350 P @ 75°C 60 - 80 P***
Density @ 25°C, grams / litre	***1200 - 1235***
Colour, APHA	***450 max***
Moisture, %	***0.5 max***

* The typical values presented here are believed to be accurate; they should not, however be considered to constitute a specification.

EUROPE

Charleston Road, Hardley,
Hythe, Southampton, Hampshire
SO45 3ZG UK
Call +44 2380 245 437

NORTH AMERICA

300 Brookside Avenue,
Building #23, Suite 100 Ambler,
PA 19002 USA

Call +1 215 773 9280 Toll

Free 888 519 3883

Email: PaintsandCoatings@geosc.com

www.geosc.com

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Table 1 - Pre-polymer Formulation

Materials	EXP 1025-54	EXP 1025-48
Terathane™ 650 [1]	168	174.2
Polyol HA-0135	84.6	87.1
TMXDI® [2]	159.4	0
Desmodur® I [3]	0	150.7
Total	***412***	***412***

1. DuPont
2. Cytec Industries
3. Bayer MaterialScience LLC

Synthesis of the NCO terminated pre-polymer

1. Charge Terathane™ 650 and Polyol HA-0135 to a reactor equipped with agitator, inert gas sparging tube, temperature controller and water-cooled condenser.
2. Heat the contents to 80°C and hold until the solution is homogeneous. Add isocyanate (Desmodur® I or TMXDI®), then reheat the batch to 80°C and hold for 1 hour. After one hour, increase the temperature to 90ºC and hold for the % NCO end point. ***Note:*** % NCO is determined by the di-n-butylamine titration method.
3. When the anticipated % NCO is obtained, prepare for "Neutralization", “Dispersion” and “Chain Extension".
4. Determine and record the final properties of the pre-polymer solution, such as % non-volatile, viscosity, and % NCO.

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Pre-Polymer Properties	EXP 1025-54	EXP 1025-48
Non-Volatile, %	100	100
Viscosity @ 75°C, Brookfield #5 spindle @ 20 rpm, cP	7500	10000
NCO : Hydroxyl	1.9	1.9
Free NCO, %	6.30	6.60
Acid Value, mgKOH / g	23	24

Part II - Neutralization, Dispersion & Chain Extension

Dispersion Formulation	EXP 1025-54	EXP 1025-48
Pre-Polymer	412	412
Triethylamine (TEA)	15.4	15.5
De-ionized Water *	417.5	417.5
Ethylene Diamine	18	18
De-ionized Water	137.1	137
Total parts	1000	1000

* Note: For lab reactions, the pre-polymer was charged to the dispersing vessel and neutralized before adding the first charge of water. The water was heated to 35-40°C prior to charging. For production, TEA and water are first charged to the dispersing vessel ,followed by the pre-polymer.

Dispersion Procedure

Neutralization & dispersing pre-polymer:

1. At 75°C, begin transferring pre-polymer solution to dispersing vessel equipped with high-speed dispersing agitator. Begin agitation on low to medium speed and charge triethylamine (TEA). Allow time for TEA to mix in, typically 3-5 minutes for a 1000g lab batch.
2. Increase agitator speed to 800-1000 rpm and begin charging de-ionized water at a rate sufficient to maintain a vortex. Once water is in and the resin is dispersed, reduce agitator speed and rapidly cool to 25 -30°C.

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Chain Extension:

1. While dispersion is cooling, pre-mix ethylene diamine (EDA) and second charge of water.
2. Once the dispersion is below 30°C, begin addition of EDA / water mixture. Note: it is best to start addition slowly, then gradually increase rate if viscosity is stable. If viscosity increases, stop addition until viscosity decreases. If viscosity does not decrease after 1-2 minutes, add more de-ionised water in small increments until it does. Repeat process if viscosity continues to increase during chain extension.
3. Once all the EDA / water mixture is in, reduce agitator speed to 300-400 rpm and continue mixing for approx. 20 minutes.
4. After 20 minutes, stop agitation and transfer dispersion to a new container and hold for one day before adjusting pH and characterizing dispersion. Note: the one day hold is to allow for entrained air to escape.

Dispersion Properties	EXP 1025-54	EXP 1025-48
Non-Volatile, %	43	43
Viscosity, Brookfield , cP	350	280
KU-2 Viscometer		
Kreb units	64.2	63
cP	387	367
pH	7.5	8.13
Density, lbs / gallon	8.67	8.67
grams /litre	1040	1040
VOC (as supplied), g / l	36	36

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Film Properties EXP 1025-48 * % CoatOSil 2287 Silane** on TW	0.00%	0.60%	0.80%	1.00 %
Chemical Resistance				
Sunscreen	0	2	4	4
DEET, insect repellent	0	2	3	4
Isopropyl alcohol	0	4	5	5
10% Ammonium hydroxide	3	5	5	5
10% Hydrochloric acid	5	5	5	5
10% Sulfuric acid	5	5	5	5
10% Acetic acid	2	4	5	5

* 3 mils / 70 µm wet film baked 5 minutes @ 90°C, 30 minute exposure @ 25° C.

** Momentive Performance Materials

Handling & Storage: Polyol HA-0135 is very hygroscopic. Container should be sealed at all times unless discharging. Due to the reactive nature of the material, containers should be tightly sealed and stored at 0-30°C. If preheating is required for discharging, ***Polyol HA-0135*** should be heated to a maximum temperature of 70°C for no longer than 48 hours.

If the material is exposed to >70°C for extended periods of time, undesirable side reactions may occur that could cause variations in the properties of the prepared formulations.

The shelf life of the material is minimum 1 year if stored in original sealed container at 0-30°C.

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