TECHNICAL BULLETIN

Polyol HA-0135

Polyol HA-0135 in Low VOC PUDs for Heat Activated Laminated Adhesives

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 Dimethylol propionic acid***;*** 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.

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Table 1 - Pre-polymer Formulation

Materials	EXP 1025-35	EXP 1025-42	EXP 1025-2
Diexter-G 6600-37 [1]	256.4	273.4	269.2
Polyol HA-0135	51.3	54.7	64.7
Desmodur® I [2]	68.4	0	58.8
TMXDI® [3]	0	79.2	0
Acclaim® Polyol N2220 [4]	51.3	54.7	0
Dipropyleneglycol DME [5]	22.4	0	69.3
Total	***449.8***	***462***	***462***

1 hydroxyl terminated saturated linear polyester which is a poly(hexamethylene adipate/isophthalate) glycol, product of Coim USA, Inc.

2 Desmodur® I is a product of Bayer

3 Cytec Industries

4 Bayer MaterialScience LLC

5 Clariant Corporation

Synthesis of the NCO terminated pre-polymer

1. Charge Diexter-G 6600-37 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, charge N2220 and solvent (DPG -DME) if needed, heat to 90°C and hold for the % NCO end point. Note: % NCO is determined by the di-n-butylamine titration method.
3. When the anticipated percent 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 1025-35	EXP 1025-42	EXP 1025-2
Non-Volatile, %	95	100	85
Viscosity @ 75°C, Brookfield #5 spindle @ 20 rpm, cP	10000	13500	7500
NCO:OH	1.93	1.90	1.77
Free NCO, %	2.90	2.80	1.90
Acid Value on solids, mgKOH / g	13.5	13.3	18.5

Part II - Neutralization, Dispersion & Chain Extension

Dispersion Formulation	EXP 1025-35	EXP 1025-42	EXP 1025-2
Pre-Polymer	449.8	462	462
Triethylamine (TEA)	9.3	9.2	12
Di-ionized Water *	394.8	378.6	384
Ethylene Diamine	7.7	8	5.7
De-ionized Water	138.4	142.2	136.3
Total parts	1000	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 agitation of EDA and water. Note: it is best to start addition slowly then gradually increase rate if viscosity is stable. If viscosity increases, stop addition until viscosity reduces. If viscosity does not reduce 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 pre-mix is in, reduce agitator speed to 300-400 rpm and continue mixing for approx. 20 minutes.

Dispersion Properties	*EXP 1025-35*	*EXP 1025-42*	*EXP 1025-2*
Non-volatile, %	44	47	41.1
Viscosity @ 25°C, Brookfield #2 spindle @ 20 rpm, cP	620	2880	2200
KU-2 Viscometer			
Kreb units	57	83.8	67
cP	292	917	2260
pH	7.5	7.5	7.54
Density lbs/gallon, grams/litre	8.76 / 1050	8.78 / 1050	8.75 / 1050
VOC (as supplied), g / l	77	21	156
Minimum activation temperature, °C	45 - 55	45 - 55	45 - 55

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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