

Capa[®] 2201A IN MDI ELASTOMERS

Capa[®] 2201A is a linear polyester diol, derived from Caprolactone and is terminated with primary hydroxyl groups.

This diol has been specifically developed as a raw material for the production of storage stable prepolymer systems based on MDI.

Typical Properties

Physical Form	:	white, waxy solid
Melting Range	:	40° - 50°C
Hydroxyl Value	:	56 mg KOH/g
Acid Value	:	<0.1 mg KOH/g
Water Content	:	<0.01%
Density at 60°C	:	1.06 g/ml
Viscosity at 60°C	:	about 500 cps.
Storage Stability	:	6 months

The reactivity characteristics of Capa[®] 2201A are similar to those of Capa[®] 2201 and it can therefore be used to replace this grade in virtually all applications.

The use Capa[®] 2201A offers the following advantages:

1. Prepolymers are storage stable at room temperature (20° - 25°C) for at least six months.
2. Prepolymers containing 50% - 60% w/w MDI are liquid at 20°C and remain so if stored at 20° - 25°C. At lower levels of MDI the melting point of the system steadily increases.
3. Prepolymers can be stored at 75° - 80°C for 10-14 days without significant change in viscosity, moulding characteristics and rubber properties.

4. Polyurethanes made from MDI and butane diol are almost colourless and yellow only slightly with time in the presence of light.

Polyurethanes based on Capa[®] 2201A show a significant improvement in this respect over products based on Capa[®] 2201 and other polyesters.

5. Thermoplastic polyurethanes based on Capa[®] 2201A have very low odour levels and thus, are suitable for extrusion grade thermoplastic elastomers used in demanding applications such as air hoses.

Prepolymer Preparation

The required quantity of molten Capa[®] 2201A is charged to the reactor, which should be fitted with a stirrer, heating and cooling facilities and a means of applying vacuum.

The Capa[®] 2201A is heated to 95°C and the required amount of flake MDI at 20° - 25°C is added and the mixture stirred, (alternatively, the Capa[®] 2201A may be heated at 80°C and molten MDI at 45° - 50°C is added, again with stirring).

Initially, the temperature will drop and then rise after about one minute. The exotherm should be controlled to a maximum of 80°C, by heating or cooling for one hour. During the last half hour of reaction, vacuum is applied to ensure the prepolymer is degassed.

Urethane Preparation

The required amount of degassed prepolymer, at 80°C is weighed into a suitable mixing vessel. To this is added the correct amount of chain extender, at 20° - 25°C.

The mixture is stirred in such a way that inclusion of air bubbles is avoided, for at least one minute or until the extender has reacted or dissolved in the prepolymer.

The resultant liquid urethane is poured into a heated mould at 110°C and cured at 110°C for at least 20 minutes.

After demould, the resultant elastomer should be cured for a further 24 hours at 110°C and subsequently post cured at room temperature for at least 14 days.

With some formulations, improvements in mixing can be obtained by:

1. Increasing the mixing temperature of the prepolymer with chain extender at 95 °C.
2. Adding the chain extender at 40° - 50 °C, instead of 20° - 25 °C.

Soft Urethanes

With soft urethanes, such as those with hardness from 50-70 Shore A, it may be necessary to use catalysts to obtain rapid demould.

Cobalt octoate and Tri-iso propanolamine, or mixtures of these are recommended.

Suggested levels are 0.003 parts cobalt octoate and 0.1 parts Tri-iso propanolamine per 100 parts of rubber. They should be dissolved in the chain extender prior to mixing with the prepolymer.

For elastomers with hardnesses in this range, quasi prepolymers containing 50-55% w/w MDI should be used, to avoid problems of mixing due to the high viscosity of the true prepolymer. They should then be used with the remaining mixture of chain extender and diol.

1. Cast Urethane Formulations

NCO/OH Index 1.07

Hardness Shore A	Chain Extender Type	% MDI	% Capa [®] 2201A	% Chain Extender	Parts MDI	Parts Capa [®] 2201A	Parts Chain Extender
95	1	33	58.54	8.46	56.37	100	14.45
90	1	30	62.74	7.26	47.81	100	11.57
85	1	28	65.52	6.48	42.73	100	9.89
80	1	26	68.32	5.68	38.05	100	8.31
70	1	20	76.73	3.27	26.06	100	4.26
60	2	20	77.26	2.74	25.89	100	3.55
50	3	20	77.44	2.56	25.83	100	3.31

Chain Extenders are:

Type 1 : 1,4 Butane Diol

Type 2 : 60/40 Butane Diol/Ethane diol,
Average Molecular Weight 76.28

Type 3 : 40/60 Butane Diol/Ethane diol,
Average molecular weight 70.88

2. Prepolymer Storage Stability

These results were obtained with total prepolymer formulated for 90 Shore A, using MDI.

a) Prepolymer Storage at 80 °C

Prepolymer Storage Time	Capa [®] 2201		Capa [®] 2201A	
	Pot Life minutes	Demould minutes	Pot Life minutes	Demould minutes
1 hour	9	27	7	21
2 hours	10	30	7	20
3 hours	8	37	7	20
24 hours	8	60*	7	19
14 hours	-	-	7	18

* A crumbly rubber was obtained.

b) Room Temperature Storage of Capa[®] 2201A Prepolymer

	Pot Life minutes	Demould minutes
Initial result	9	25
After 6 months at 20° - 25 °C	8	30

3. Comparison of Elastomer Properties based on Capa[®] 2201 and Capa[®] 2201A with MDI

PROPERTY	Capa [®] 2201	Capa [®] 2201A
	90	90
100% Modulus, kg/cm ²	78	91
300% Modulus, kg/cm ²	196	159
Tensile Strength, kg/cm ²	621	618
% Elongation	390	440
Tear Strength, kg/cm ²	92.3	100
Resilience at 21 °C	58%	59%
at 70 °C	73%	76%
Compression Set	18%	23%
Hydrolysis Resistance at 100 °C days		
T _{1/2}	4.75	6.5
T100	6.25	8.0

4. Hydrolysis Resistance

The hydrolysis resistance of polyurethane elastomers based on MDI and various grades of polycaprolactone was determined with the samples immersed in flowing demineralised water, in the presence of air.

Capa [®] Grade	T _{1/2} /100 °C Days	T100/100 °C Days	T _{1/2} 80 °C Days	T100 80 °C Days
2201A	7-10	11-14	40-46	45-53
2201	5-6½	6½-8	20-26	28-31
2200A	6-7	9½-11	30-49	39-54
2200	3½-5	6-7	10-17	17-23

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