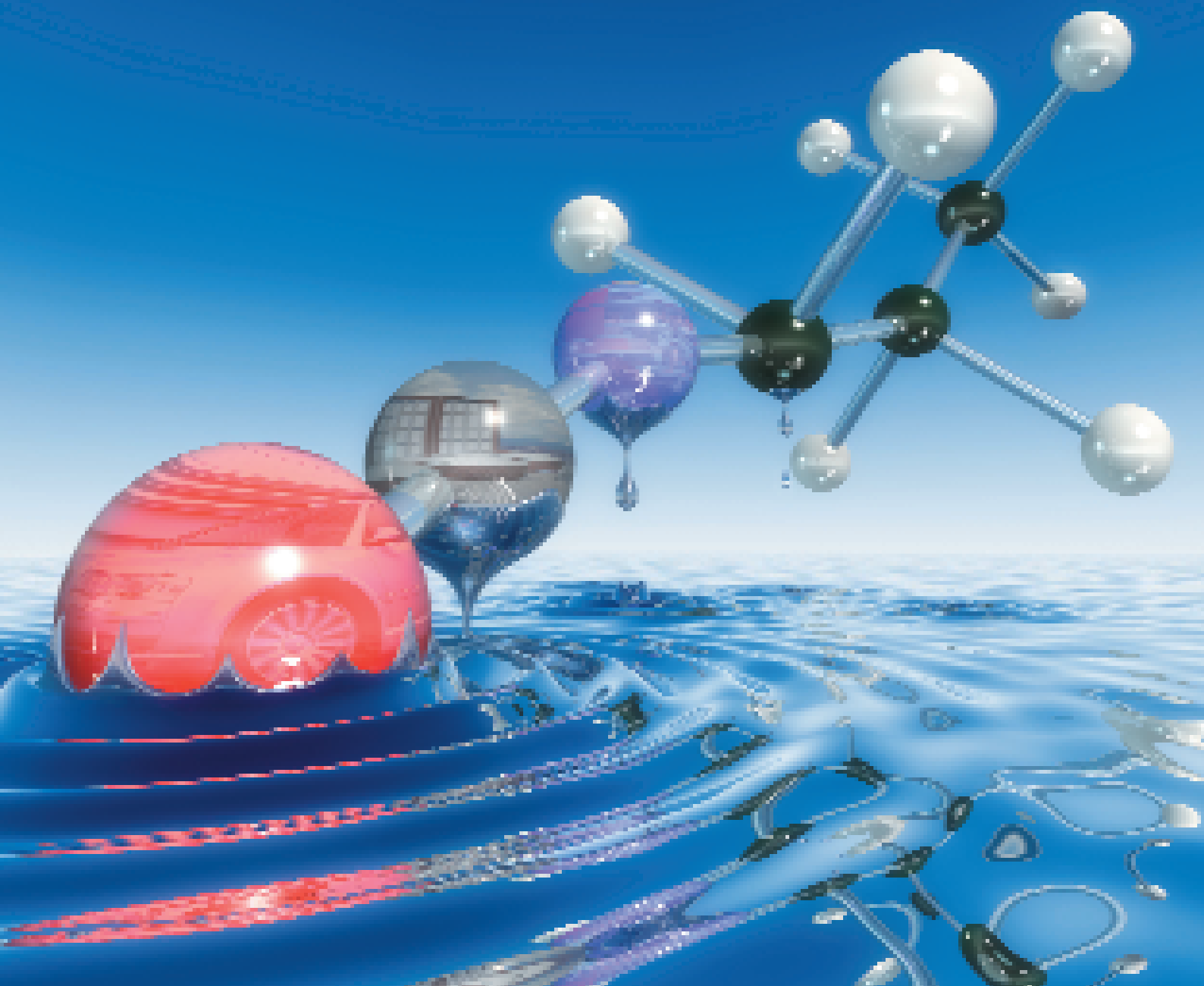
Baxenden
Chemicals Limited

Speciality Chemicals Division

Blocked Isocyanates



The Chemistry of Blocked Isocyanates

Introduction

The isocyanate groups associated with urethane crosslinkers are highly reactive towards many other compounds. This reactivity can lead to stability problems in two pack (2k) coatings and prepolymers, due to pot life limitations and the ability of the NCO groups to react with atmospheric moisture. Blocking technology was developed to overcome these limitations and to give storage stable one-pack systems (1k) which can be thermally or chemically re-activated.

Baxenden's Trixene blocked isocyanates are designed to be compatible with hydroxyl functional polyesters, polyethers and acrylics, as well as epoxy, phenolic and amine functional resins.

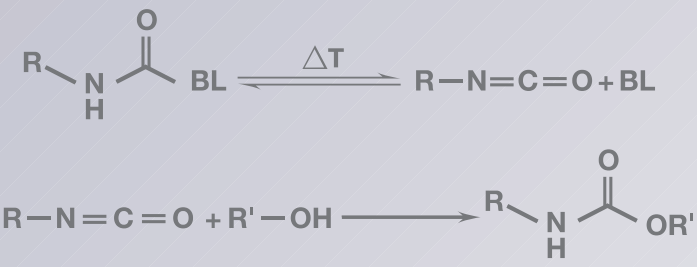
The level of blocked isocyanate required in a given formulation is calculated from the apparent equivalent weight.

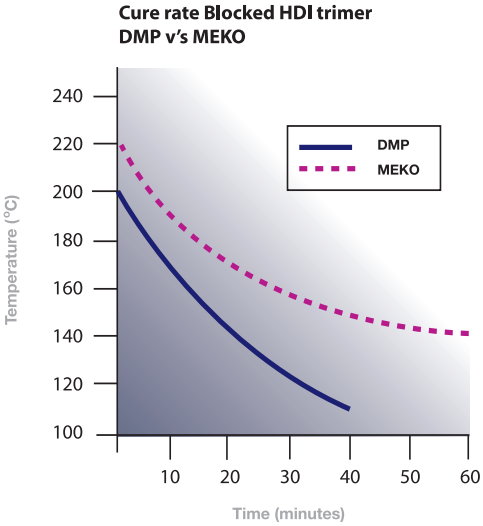
The mechanism for the reaction between blocked isocyanates and hydroxyl functional species is given below.

Mechanism

1. The first step is the thermal liberation of the isocyanate group (R-NCO).
2. The second step is the reaction of the generated isocyanate with the available hydroxyl groups.
3. The blocking agent is either liberated as a VOC or, as in the case of the pyrazole systems, remains mainly in the cured coating.

Blocked Isocyanate Mechanism



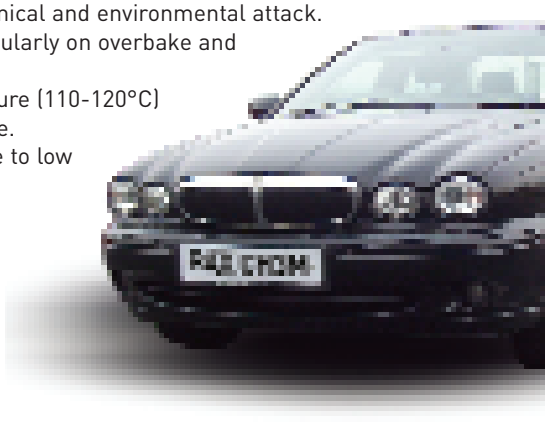


DMP Blocked Isocyanates

In the drive to lower the unblocking temperature of conventional blocked isocyanates, Baxenden Chemicals developed the use of 3,5-Dimethylpyrazole (DMP).

DMP blocked isocyanates have several advantages relative to other established blocking technologies.

- Improved resistance to chemical and environmental attack.
- Better colour stability particularly on overbake and UV exposure.
- Lower unblocking temperature (110-120°C) and increased cure response.
- Reduced VOC emissions due to low volatility of blocking agent.



Speciality Blocked Isocyanates

Baxenden's Speciality Chemicals Division is at the forefront of blocked isocyanate technology. The Trixene range of products is designed for high performance coatings in a wide range of application areas.

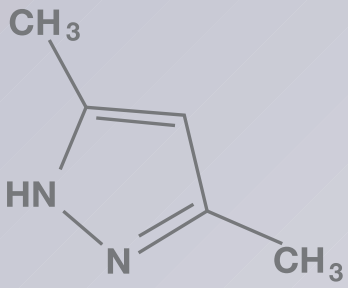
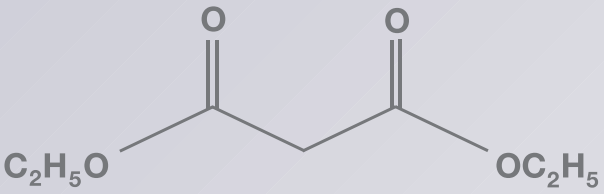
The reduced curing temperature of the Baxenden products also creates opportunities to heat cure coatings on temperature sensitive substrates such as plastics.

Trixene Grade No.	Iso Type	Blocking Agent	Viscosity @ 25°C (mPa.s)	Eq. Wt. (as supplied)	Solids (%)	Solvent	Application
Aromatic Blocked Isocyanate							
BI 7641	TDI Prepolymer	DMP	6,250	744	60	PMA / Xylene	Automotive, coil, leather
BI 7642	TDI Prepolymer	MEKO	25,000	737	60	PMA / Xylene	Coil, automotive, primers/base
Water Based Blocked Isocyanate							
BI 7986	HDI trimer	DMP	150	846	40	Water/NMP	Automotive and coil coating
BI 7987	HDI trimer	DMP	200	933	40	Water/DPGME	Automotive and coil coating
Aliphatic Blocked Isocyanate							
BI 7950	IPDI	DMP	1,200	567	65	PM	Automotive and coil coating
BI 7951	IPDI trimer	DMP	3,500	539	65	C_9 Aromatic/BA	Automotive and coil coating
BI 7960	HDI biuret	DMP	1,100	410	70	PM	Coil and electrodeposition
BI 7961	HDI biuret	DMP	2,250	410	70	C_9 Aromatic	Coil and electrostatic
BI 7963	HDI biuret	DEM	4,500	477	70	PM	Low temp curing or higher reactivity
BI 7981	HDI trimer	ε-CAP	450	476	65	PM Acetate	Coil coating
BI 7982	HDI trimer	DMP	600	410	70	PM	Automotive and coil coating
BI 7984	HDI trimer	MEKO	3,000	373	75	C_9 Aromatic	Automotive and coil coating
Hybrid Aliphatic Blocked Isocyanate							
BI 7990	IPDI trimer	DMP/DEM	5,000	538	65	PM/ PM Acetate	Automotive and coil coatings
BI 7991	HDI biuret	DMP/DEM	1,000	456	70	PM/ PM Acetate	Automotive and coil coatings
BI 7992	HDI trimer	DMP/DEM	1,500	456	70	PM/ PM Acetate	Automotive and coil coatings

Key features of the Trixene BI product range include:

- A range of flexibility, functionality and branching.
- A range of blocking agents to meet unblocking temperature and cure speed requirements.
- Compatibility and stability in the presence of hydroxyl functional resins.
- Monomeric isocyanate emissions well below safety limits during curing process.
- Exceptional colour, colour retention and pigment tolerance.
- No significant VOC increases compared with conventional 2k coatings.
- No significant overbake yellowing when using DMP to block aliphatic systems.
- Solids contents suitable for medium to high solids coatings.
- Water dispersible and emulsified systems for low VOC coatings.

Baxenden's Blocking Technology

3,5-Dimethylpyrazole	Diethyl malonate
	

Baxenden Chemicals pioneered and patented the use of 3,5-Dimethylpyrazole as a blocking agent. Continuous refinement of the in-house manufacturing process yields a very pure and consistent blocking agent for use in high performance coatings.

This gives lower unblocking temperatures than other conventional blocking technologies (e.g. MEKO), whilst offering excellent colour stability.

Recent developments have centred around the use of hybrid blocked isocyanates combining the attributes of both 3,5-dimethylpyrazole (DMP) with diethyl malonate (DEM). These systems give a combined polymer network resulting from two differing reaction mechanisms.

DEM blocked isocyanates have a tendency to crystallise on standing. The process of hybridisation reduces this and also improves the compatibility with OH functional resins.

Blocking Agents

Baxenden Chemicals uses a wide range of different blocking agents in its systems. The choice of blocking agent is governed by several different factors.

1 The choice of co-reactant, i.e. hydroxyl or amine functional.

2 The time and temperature of curing.

3 Method of application.

4 End use application, e.g. primer or top coat.

The table below gives the range of blocking agents available along with their unblocking temperatures when used with aliphatic isocyanates.

Blocking Agent	Unblocking Range (°C)	Melting Point (°C)	Boiling Point (°C)
Diethyl Malonate (DEM)	100 - 120	-50	199
3,5-Dimethylpyrazole (DMP)	110 - 120	106	218
Methylethylketoxime (MEKO)	140 - 160	-30	152
Caprolactam (Ɛ-CAP)	160 - 180	72	138

Blocked Isocyanate Prepolymers

Baxenden Chemicals produces a range of blocked aromatic prepolymers for use as epoxy flexibilisers. These systems are designed for use with ambient temperature processed, amine cured, two pack epoxy systems.

Replacing some of the epoxy resin in the formulation with these products gives increased elongation, impact resistance and flexibility without compromising the chemical resistance of the epoxy.

Trixene Grade No.	Iso Type	Polymer Type	Viscosity @ 25 °C (mPa.s)	Isocyanate Eq. Wt.
BI 7770	TDI	Branched	68,000	1860
BI 7771	TDI	Branched	80,000	1750
BI 7772	TDI	Linear	35,000	2100
BI 7774	TDI	Branched	40,000	1945
BI 7779*	TDI	Branched	30,000	2170

*contains 10% dioctyl adipate

Baxenden Chemicals' policy of customer service means we will undertake product design to suit individual customer requirements. Many of the products listed can be provided in different solvent combinations to accommodate different application techniques and stoving temperatures.

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