



# **DESCRIPTION**

Incozol EH is a urethane bis-oxazolidine latent hardener for use in 1-K polyurethane coatings, adhesives, sealants and elastomers.

Used in conjunction with a low NCO polyurethane prepolymer, this hardener accelerates the cure of the prepolymer through a moisture triggered mechanism to afford crosslinking benefits to the 1-K urethane. In addition, it enhances properties such as through cure development and mechanical strength through the elimination of CO<sub>2</sub> gassing in high build, high solids PU systems.

Incozol EH confers tolerance to the repeated opening of containers and can be used as a versatile curing agent with 2 or 4 functional mix ratios. It can also be handled and stored at temperatures as low as -15°C without risk of crystallisation.

# **PROPERTIES**

Functionality	4
Equivalent Weight	150
Viscosity @ 20ºC (mPa.s)	ca 7000
Density (g/cm³)	1.033
Colour (APHA)	<400
Purity (%)	>98%

# **FEATURES & BENEFITS**

- Can be stored at sub-zero temperatures (down to -15°C) without signs of crystallisation, making handling and storage easier and more convenient than other types of bis-oxazolidines.
- Excellent in-can stability for 1-K aliphatic and aromatic PU systems.
- Increased through-cure and tensile profile of formulated PU system leading to tougher, more durable product.
- Reduced odour the reduced volatility of the aldehyde leaving group (2-ethyl hexanal) in this product results in less pungent odour than bis-oxazolidines based on isobutyraldehyde. Useful for low odour 1-K PU formulations.

# **TYPICAL APPLICATIONS**

- To improve the aesthetics and performance of PU systems, by eliminating CO<sub>2</sub> gas formation.
- To increase cure speed of a 1-K PU systems where the addition of metal based catalysts are restricted or unwanted.
- Incozol EH is particularly suited for use in aromatic 1-K PU systems where in-can stability may be an issue.

# **TEST DATA**

## Non-crystallising behaviour of Incozol EH.

As can be seen from the table below, **Incozol EH** shows excellent storage stability, with no signs of crystallisation even when seeded with a potential nucleation site (to mimic dirt contamination). This is in contrast to isobutyraldehyde based oxazolidines (e.g. Incozol 4) that are currently sold by Incorez, which provide limited storage stability at these temperatures.

Product	Seeded (Y/N)	Storage Temp. (ºC)	1 week	2 weeks	1 month	2 months	4 months	6 months
	N		CRY	CRY	CRY	CRY	CRY	CRY
Incozol 4	Y	-15	CRY	CRY	CRY	CRY	CRY	CRY
11002014	N		PC	PC	CRY	CRY	CRY	CRY
	Y	20	PC	PC	CRY	CRY	CRY	CRY
	N		NC	NC	NC	NC	NC	NC
Incozol EH	Y	-15	NC	NC	NC	NC	NC	NC
	N		NC	NC	NC	NC	NC	NC
	Y	20	NC	NC	NC	NC	NC	NC
	N		NC	NC	NC	NC	NC	NC
Competitor	Y	-15	NC	NC	NC	NC	NC	NC
material	N		NC	NC	NC	NC	NC	NC
	Y	20	NC	NC	NC	NC	NC	NC

KEY: NC - Not crystallised, PC - Partly crystallised, CRY - Crystallised (solid)

# Enhancing the performance of aromatic PU sealant prepolymers using oxazolidine latent hardeners

# **Study Results**

All test data was acquired using the prepolymer with oxazolidine only; no fillers or additives were included in the mixes.

# **Dealing with moisture ingress**

It is vitally important when handling PU prepolymer mixes with oxazolidine latent hardeners that moisture ingress is minimised. Good practice recommends nitrogen purging of all mixing reactors and minimum exposure to atmospheric moisture during handling.

# **Oxazolidines eliminate gassing**

Traditionally, PU sealants are cured by the reaction of terminal isocyanate groups with moisture. A side product of this reaction is the generation of carbon dioxide which remains trapped in the cured sealant system. The pictures below illustrate cured PU prepolymer (MDI: Desmoseal M280 & TDI: Desmodur E15) used in sealants mixed with oxazolidine latent hardener (typically up to 2% w/w) in comparison to the same prepolymer cured solely with moisture.



MDI prepolymer Desmoseal M280 cured without oxazolidine (left), cured with 1% Incozol EH (centre) and cured with 2% Incozol EH (right).



TDI prepolymer Desmodur E15 cured without oxazolidine (left) cured with 1% Incozol EH (centre) and cured with 2% Incozol EH (right).

#### Impact on storage stability

The change in viscosity was measured for unopened containers of PU prepolymer (MDI & TDI) mixes with different oxazolidine latent hardeners. Containers were sealed and stored at ambient temperature (23°C) for 56 days.





As expected, TDI based mixes are more stable than MDI systems due to the increased reactivity of MDI prepolymers. For TDI systems, mixing with a latent hardener shows an initial viscosity rise where moisture ingress from the mixing/handling is consumed. Excellent stability follows with little viscosity rise whilst the containers are sealed from the atmosphere.

With MDI mixes, the stability is reduced compared to TDI. The data reflects 1% oxazolidine in Desmoseal M280 prepolymer mixes; dilution of the binder and curing agent in fully formulated systems with dry, inert fillers affords improved stability.

# **Through Cure/Hardness Development**

The addition of oxazolidine latent hardener not only eliminates sealant defects created by gassing, but also promotes faster through cure than by moisture alone. This reflects the ability of oxazolidine latent hardeners to hydrolyse quickly and crosslink the PU prepolymer in preference to the water and isocyanate reaction.



Hardness development of oxazolidine/Desmoseal M280 systems





Above figures of hardness development indicate faster cure when oxazolidines are blended with less reactive TDI PU prepolymer as opposed to MDI PU prepolymers. This is due to the competitive nature of the MDI-water reaction over hydrolysis of the oxazolidine.





As can be seen from the graphs above, no appreciable change in ultimate shore A hardness was observed for all oxazolidine crosslinked systems over the prepolymer control – only the cure speed has been increased (for the TDI system).

## Impact on tensile strength

In addition to shore hardness, the use of latent hardeners does not significantly alter the tensile profiles of the cured PU system at the low levels required to eliminate CO<sub>2</sub> gassing.

In general, Incozol EH slightly increases the hardness and tensile strength of the PU prepolymer while Incozol LV slightly reduces the hardness and tensile strength due to the nature of the oxazolidine bridging group.





# Enhancing the performance of 1K PU flooring & balcony systems using oxazolidine latent hardeners

# Study 1 – Use of an oxazolidine latent hardener in a floor coating system

This study investigated the impact on the cure and film properties of blending oxazolidine latent hardeners, Incozol 4 and Incozol EH, with an aliphatic polycarbonate prepolymer, Incorez 705. Both latent hardeners were combined with the control prepolymer (Incorez 705) at 1:1 stoichiometry based on % NCO of the prepolymer (approximately 4% w/w).

# **Dealing with moisture ingress**

It is vitally important when handling PU prepolymer mixes with oxazolidine latent hardeners that moisture ingress is minimised. Good practice recommends nitrogen purging of all mixing reactors and minimum exposure to atmospheric moisture during handling.

# **Oxazolidines eliminate gassing**

The addition of the oxazolidine latent hardener to the aliphatic PU prepolymer, not only eliminates coating defects created by gassing, but also promotes faster through cure than by moisture alone. This reflects the ability of the oxazolidine latent hardener to hydrolyse quickly and crosslink the PU prepolymer in preference to the water and isocyanate reaction.

# Faster through cure = Early walk on time

One of the key benefits of using an oxazolidine latent hardener is the ability to accelerate the through cure of the 1K PU floor coating without the need for additional metal catalyst. The latent hardener is able, through hydrolysis, to speed up the crosslinking of the PU floor paint to reduce touch-dry and walk-on times.



The use of an oxazolidine more than halves the touch dry and walk on times compared to the moisture triggered control system. A similar increase in cure speed for the control prepolymer can only be achieved using high levels of metal catalyst loading (>0.5% w/w), which is undesirable for in-can stability.

# Improved durability through crosslinking

Formulating with a latent hardener, such as Incozol 4 or Incozol EH, significantly boosts the ultimate hardness and tensile strength of the system.





The increase in hardness and tensile strength over the control prepolymer is due to the crosslinking nature of the bis-oxazolidine.

# **Chemical and stain resistance**

Formulating Incozol 4 or Incozol EH latent hardener into the flooring system improves the resistance to many chemicals including dilute acids, alkalis and hydrocarbon based reagents.

The spider diagrams above clear shows the benefits that oxazolidine crosslinking brings to a simple flooring system.



In addition to chemical resistance, crosslinking of the PU coating also significantly improves stain resistance properties and subsequently long term aesthetics of the flooring system.



# Storage stability



The change in viscosity was measured for unopened containers of PU prepolymer (Incorez 705) with the addition of Incozol 4 and Incozol EH at a 1:1 stoichiometry based on the total % NCO of the flooring system.

As can be seen from the stability graph, both oxazolidines show very good in-can stability even after storage at 40°C for 56 days.

# **Guide Formulations**

Materials	Parts by weight (g)	Supplier
Incorez 705	380.00	Incorez
Incozol EH	54.29	Incorez
Bannersolve C573	45.84	Samuel Banner
BAS 125	68.60	Samuel Banner
DBTDL	0.75	Akcros
Tinuvin 292	2.50	Ciba
Tinuvin 1130	2.50	Ciba
Total	554.48	

#### Durable clear topcoat based on Incorez 705 prepolymer and Incozol EH latent hardener

#### **Properties**

% Solids	65
Viscosity @ 20ºC (cps)	418
Specific Gravity	0.998

#### **Recommended Application Details**

Application	Brush/Roller/Float
Walk-on time (hours)	13
Surface-dry time (hours)	4

#### **Manufacturing Instructions**

1. Materials are moisture sensitive so all preparations must be performed under an inert atmosphere.

2. Add Bannersolve C573 and BAS 125 to the Incorez 705 and stir the mixture for 10 minutes.

3. If necessary, the water content can be checked at this point and corrected using Incozol 2.

4. DBTDL is then added and stirred for 1 hour at 80°C.

5. The mixture is then cooled to 40°C and the additives Tinuvin 292 and Tinuvin 1130 added and stirred for a further 10 minutes.

6. Finally, Incozol EH is added and mixed until homogenous and packaged under an inert atmosphere.

## Durable floor paint based on Incorez 705 prepolymer and Incozol EH latent hardener

Materials	Parts by weight (g)	Supplier
Incorez 705	161.20	Incorez
Incozol EH	23.00	Incorez
Filler TiO2	62.49	Tioxide
Barytes B10	223.44	Deutsche Baryt - Industrie
DBTDL	0.59	Akcros
BAS 125	40.65	Samuel Banner
Tinuvin 292	0.81	Ciba
Tinuvin 1130	0.81	Ciba
Black colour paste	10.02	Clariant
Total	523.01	

#### **Properties**

% Solids	65
Viscosity @ 20ºC (cps)	4400
Specific Gravity	1.1

#### **Recommended Application Details**

Application	Brush/Roller/Float
Walk-on time (hours)	14
Surface-dry time (hours)	9

#### Typical Properties for Incorez 705 prepolymer

NCO Content (%)	4.0
Viscosity @ 20ºC (cps)	6,000
Density (g/cm <sup>3</sup> )	1.05
Colour (Gardner)	1
% Solids	80

#### **Manufacturing Instructions**

1. Materials are moisture sensitive so all preparations must be performed under an inert atmosphere.

2. A premix of BAS 125, Tinuvin 292, Tinuvin 1130 and DBTDL is mixed until homogenous.

3. To Incorez 705, half of the Barytes B10 is added slowly under high shear ensuring good dispersion. Once homogenous half of the premix is then added.

4. The remaining Barytes B10 is added followed by the remaining half of the premix.

5. The black colour paste is added and allowed to stir for 1 hour before the addition of Incozol EH.

6. The final paint formulation is mixed until homogeneous and then packaged under an inert atmosphere.

## Study 2 - Use of an oxazolidine latent hardener in a balcony coating system

This study investigated the impact on the cure and film properties of blending oxazolidine latent hardeners, Incozol 4 and Incozol EH, with an aliphatic polyether prepolymer, Incorez 718. Both latent hardeners were combined with the control prepolymer (Incorez 718) at 1:1 stoichiometry based on % NCO of the prepolymer (approximately 8.5% w/w).

## **Dealing with moisture ingress**

It is vitally important when handling PU prepolymer mixes with oxazolidine latent hardeners that moisture ingress is minimised. Good practice recommends nitrogen purging of all mixing reactors and minimum exposure to atmospheric moisture during handling.

## **Oxazolidines eliminate gassing**

The addition of the oxazolidine latent hardener to the aliphatic PU prepolymer, not only eliminates coating defects created by gassing, but also promotes faster through cure than by moisture alone. This reflects the ability of the oxazolidine latent hardener to hydrolyse quickly and crosslink the PU prepolymer in preference to the water and isocyanate reaction.

### Faster through cure = Early walk on time

One of the key benefits of using an oxazolidine latent hardener is the ability to accelerate the through cure of the 1K PU floor coating without the need for additional metal catalyst. The latent hardener is able, through hydrolysis, to speed up the crosslinking of the PU floor paint to provide a reduced touch-dry and walk-on time.



The use of bis-oxazolidines significantly reduces the walk on time of the balcony system and can therefore lead to increased productivity and turnaround.

## Improved durability through crosslinking

The use of a latent hardener brings the benefit of crosslinking without the need to reformulate the system. Through the choice of oxazolidine, it is possible to modify the hardness of a coating. This allows the performance of the coating to be easily tailored to meet different application requirements.



The aliphatic prepolymer, Incorez 718 (control), was cured by moisture alone and with the addition of an oxazolidine latent hardener. Addition of the oxazolidine into the system significantly increased the hardness through chemical crosslinking.

# Storage stability

The change in viscosity was measured for unopened containers of PU prepolymer (Incorez 718) with the addition of Incozol 4 and Incozol EH at a 1:1 stoichiometry based on the total % NCO of the prepolymer.



As can be seen from the stability graph, both oxazolidines show very good in-can stability even after storage at 40°C for 56 days.

## **Guide Formulation**

#### Clear balcony coating based on Incorez 718 prepolymer and Incozol EH latent hardener

Materials	Parts by Weight (g)	Supplier
Incorez 718	379.86	Incorez
Incozol EH	115.31	Incorez
Bannersolve C573	45.82	Samuel Banner
BAS 125	68.57	Samuel Banner
DBTDL	0.75	Akcros
Tinuvin 292	2.50	Ciba
Tinuvin 1130	2.50	Ciba
Total	615.31	

Note: The amount of Incozol EH has been calculated at a 1:1 stoichiometry to maximise crosslink density.

#### **Properties**

% Solids	63
Viscosity @ 20ºC (cps)	240
Specific Gravity	0.997

#### **Recommended Application Details**

Application	Brush/Roller/Float
Walk-on time (hours)	11
Surface-dry time (hours)	4

#### Typical Properties for Incorez 718 prepolymer

NCO Content (%)	8.5
Viscosity @ 20ºC (cps)	4500
Density (g/cm <sup>3</sup> )	1.04
Colour (Gardner)	1
% Solids	70

#### **Manufacturing Instructions**

1. Materials are moisture sensitive so all preparations must be performed under an inert atmosphere.

2. Add Bannersolve C573 and BAS 125 to the Incorez 718 and stir the mixture for 10 minutes.

3. If necessary, the water content can be checked at this point and corrected using Incozol 2.

4. DBTDL is then added and stirred for 1 hour at 80°C.

5. The mixture is then cooled to 40°C and the additives Tinuvin 292 and Tinuvin 1130 added and stirred for a further 10 minutes.

6. Finally, Incozol EH is added and mixed until homogenous and packaged under an inert atmosphere.

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