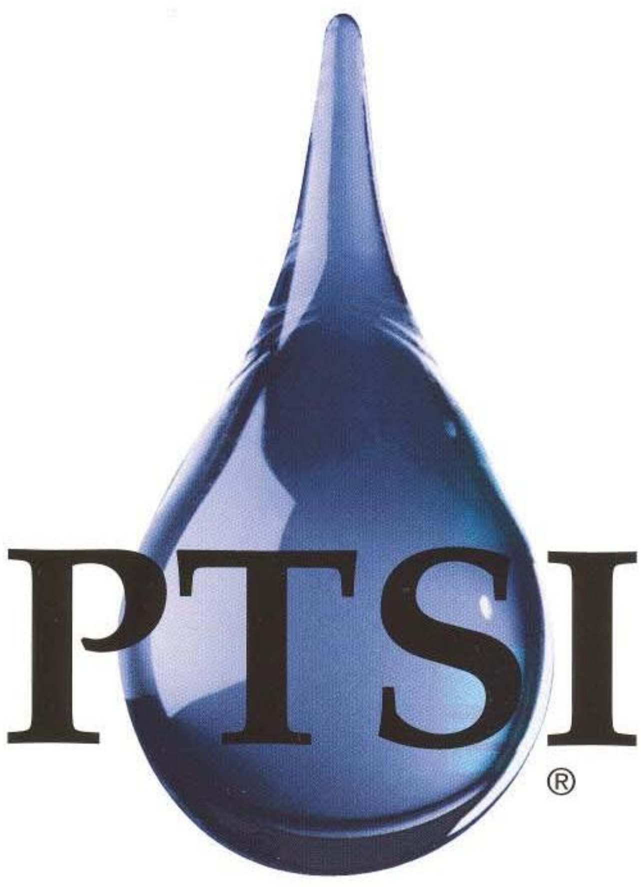
VanDeMark

Phosgene Chemistry – Custom Chemicals

Urethane Applications

for

p-Toluenesulfonyl Isocyanate



Why PTSI?

PTSI (p-toluenesulfonyl isocyanate) is a low viscosity, reactive additive useful as a water scavenger in the formulation of specialty urethane products, including sealants, adhesives, and coatings.

The reaction of PTSI with water generates carbon dioxide and the corresponding toluenesulfonamide which is generally inert towards further reaction with alkyl and aryl isocyanates. The sulfonamide is usually soluble in common coating solvents and presents no significant toxicity hazards.

PTSI provides the formulator of specialty urethane products with an expedient and efficient alternative to physical methods of dehydration in common use. PTSI is recommended further for the storage stabilization of purified diisocyanates against deterioration and discoloration.

Use PTSI for:

- Moisture-Curing Prepolymers
- Catalyzed Prepolymers
- Prepolymer and Polyol Systems
- Urethane Alkyd Production
- Urethane Lacquer Production

Improved Performance

Water is introduced during the formulation process in the form of dissolved water in solvents, adsorbed water in fillers and pigments, and ambient moisture. Subsequent reaction with alkyl and aryl isocyanates leads to the formation of by-products which can adversely affect product quality by the formation of bubbles and urea linkages. These problems are aggravated by the low equivalent weight of water and the irreversible nature of its reactions with isocyanates. The use of PTSI in the formulation process prevents most of these moisture-related problems.

Which Urethanes?

Both one and two-part systems can be formulated with PTSI as a scavenger for water introduced with solvents, pigments, and fillers. PTSI functions also as a packaging stabilizer for one-component systems and the urethane portion of two-component systems.

Moisture-Curing Prepolymers

PTSI is used effectively in moisture-curing systems to remove moisture in both the formulation and packaging steps. This bulletin includes some typical formulations for moisture-curing sealants, adhesives, and coatings.

Catalyzed Prepolymers

The utility of PTSI in catalyzed prepolymer systems depends specifically on the type of catalyst employed. Certain amine catalysts can promote an additional reaction between toluenesulfonamide and the isocyanate prepolymer, thereby reducing the NCO/OH ratio and resulting in an incomplete cure.

In systems where catalyst activity and curing are not affected by toluenesulfonamide, PTSI may be used to dehydrate solvents and solvent/filler/pigment mixtures prior to addition of the polyol and polyisocyanate components. Residual PTSI will react rapidly with the polyol portion.

Prepolymer and Polyol

PTSI is added to solvents and solvent/pigment/filler mixtures prior to treatment with the polyol and polyisocyanate components. As in the case of catalyzed prepolymer systems, excessive residual PTSI can adversely affect the NCO/OH ratio.

Other Urethanes

Urethane Alkyds

PTSI is used in the dehydration of solvents and solvent/pigment mixtures prior to their incorporation with either the isocyanate or hydroxyl portions of the alkyd formulation. As discussed above, excess PTSI will react with polyols and careful attention should be given to the quantity of PTSI necessary for dehydration.

Urethane Lacquers

PTSI may be used to ensure anhydrous conditions during the formulation of the urethane resins used in such systems.

Using PTSI

Solvents, fillers, pigments, and bituminous tars are all dehydrated readily by PTSI. Best results are obtained when the moisture content can be determined accurately.

A. Solvents

Solvents used in the formulation of one and two-component systems are dehydrated rapidly by treating with PTSI. The quantity of PTSI will vary with the water content of the solvent; in general, 1-1.5% PTSI is sufficient to dehydrate solvents with up to 0.1% water. Additional PTSI must be added for water in excess of 0.1%. Following the addition of PTSI, the solvent is ready for use within several hours.

TABLE I lists many of the solvents used in the formulation of sealants, adhesives, and coatings with recommended PTSI charges for varying water levels. Most of the common solvents used in the formulation of specialty urethane products are compatible with PTSI.

Aromatics, including toluene and xylene, can be routinely employed as co-solvents. Sulfoxides and formamides are reactive and should not be mixed with formulations to which PTSI will be added.

PTSI cannot be used, to dehydrate polyols or hydroxylic solvents. Urethane catalysts, solvents, and additives containing amine groups will react immediately with PTSI and should not be added to mixtures containing excess PTSI unless demonstrated to retain their activity on mixing.

TABLE I

Recommended PTSI Charges for Solvent Dehydration

(Pounds of PTSI/100 gallons of solvent containing 0.01% water.)

Acetone	0.72
Methyl Ethyl Ketone	0.73
Methyl Isobutyl Ketone	0.73
Methyl Isoamyl Ketone	0.75
Amyl Acetate	0.80
Butyl Acetate	0.80
Ethyl Acetate	0.82
2-Ethylhexyl Acetate	0.80
Isobutyl Acetate	0.79
Isobutyl Isobutyrate	0.78
Isopropyl Acetate	0.80
Propyl Acetate	0.81
2-Methoxyethyl Acetate	0.92
2-Ethoxyethyl Acetate	0.89
2-Butoxyethyl Acetate	0.86
Diethylene Glycol Acetate Ethyl Ether	0.92
Diethylene Glycol Acetate Butyl Ether	0.89
Dichloromethane	1.25
Ethylene Dichloride	1.15
1,1,1,-Trichloroethane	1.20
Trichloroethylene	1.33

Using PTSI

B. Pigments/Fillers

Pigments and fillers are suspended in the solvent and treated with PTSI. Processing periods of up to 24 hours are recommended to insure complete reaction. Following the water reaction period, the mixture can be treated with the polyol and isocyanate components.

PTSI requirements will vary markedly with the total water content. Experience suggests that a PTSI charge equivalent to 4% of the total formulation weight will be sufficient for the dehydration of pigment/solvent suspensions prior to addition of the polyol and polyisocyanate components. Moisture in most pigments and fillers is determined by ASTM Method D-280 although certain pigments are analyzed by other methods.

This bulletin includes some pigmented and filled polyurethane-based formulations and are representative of the types of systems in which PTSI is used.

C. Bituminous Tars/Resins

Bituminous tars and resins commonly used in the formulation of urethane-based sealants are dehydrated by treatment with PTSI. The tar is conveniently mixed with solvents, pigments, and fillers and treated with PTSI. Following a suitable reaction period, the mixture can be milled and formulated with the remaining components.

As with suspensions of pigments and fillers, PTSI levels will vary markedly, depending on the water present. Moisture in bituminous tars and resins is determined by ASTM Method D-95.

The reaction of PTSI with water in tar solutions can be accompanied by moderate to severe foaming. Caution should be exercised during the addition of PTSI to such systems.

p-Toluenesulfonamide

The reaction of PTSI with water yields p-toluenesulfonamide [88-19-7] and carbon dioxide. In general, the sulfonamide is soluble in all common coating solvents and remains evenly distributed throughout the finished coating. Table II contains solubility data for the sulfonamide in a number of solvents

p-Toluenesulfonamide is reported in the EPA TSCA Inventory 1980. It is not hazardous by definitions in 29CFR section 1501.2. The toxic effects of p-toluenesulfonamide are reported in the Registry of Toxic Effects of Chemical Substances (RTECS) 1980 as XT5075000. Disposal of p-toluenesulfonamide should be in accordance with local, state, and federal regulations.

TABLE II

Solubility of p-toluenesulfonamide in Common Urethane Solvents

SOLVENT	SOLUBILITY, GRAMS/LITER @ 25°C
Methyl Ethyl Ketone	280
Methyl Isobutyl Ketone	80
Ethyl Acetate	105
Ethylhexyl Acetate	170
Methylene Chloride	21
Xylene	15

Typical Formulations

Moisture-Cure Urethane Zinc Primer

Raw Materials	Weight Pounds	Volume Gallons	Weight Solids	Volume Solids	Supplier
1. Novacite L-207A	114.3	5.2	114.3	5.2	Malvern
2. Zinc dust #64	1370.1	23.5	1370.1	23.5	ZCA
3. Vertal 5	114.3	4.9	114.3	4.9	Vermont Talc
4. Acronal 700L (10% in A-100)	2.2	0.3	0.1	0.01	BASF
5. MPA-60X	5.6	0.8	1.4	0.2	Rheox
6. DC-11 (1% in A-100)	4.1	0.6	0.04	0.01	Dow Corning
7. Ircogel 905	9.9	1.1	5.9	0.6	Lubrizol
8. A-100/PMA1:1	265.6	34.6	0.0	0.0	
9. p-Toluenesulfonyl Isocyanate	61.2	7.3	61.2	7.4	VanDeMark
10. Desmodur E-type resin	204.0	21.7	204.0	21.7	Bayer
Total	**2151.3**	**100.0**	**1871.34**	**63.52**	

Formulation Characteristics

Weight Solids %	87.0
Volume Solids %	63.4
Weight per gallon, lbs.	21.5
Recommended film thickness, mils	3-5
Minimum time to recoat, 77°F, 50% RH	4 hours
PVC, %	53.6
P/B Ratio	6.0
VOC, lbs./gal	2.8
Viscosity @ 77°F Kreb Units	73 ± 2

Manufacturing Procedure

1. Grind ingredients 1, 3, 4, 5 and 7 in 75 pounds of #8 to a 5 Hegman. A temperature of 110°F-130°F is necessary to activate the MPA-60X.
2. Stir in the Zinc Dust #64 and 80 lbs. of #8, followed by #6 in the balance of #8.
3. Stir in the PTSI slowly to avoid excessive foaming.
4. Let the PTSI digest for at least 1 hour, but no more than 8 hours, then stir in the Desmodur E resin.

Typical Formulations

MIOX Coating

Raw Materials	Weight Pounds	Volume Gallons	Weight Solids	Volume Solids	Supplier
1. Barytes #1	173.5	4.8	173.5	4.8	Harcros
2. Vertal 5	165.1	7.1	165.1	7.1	Vermont Talc
3. Aluminum 6-334	67.1	5.3	44.3	1.8	Reynolds
4. MIOX-AS	271.4	7.3	271.4	7.2	
5. Acronal 700L (10% in A-100)	2.8	0.4	0.1	0.02	BASF
6. MPA-60X	7.9	1.1	2.0	0.3	Rheox
7. DC-11 (1% in A-100)	11.1	1.5	0.1	0.02	Dow Corning
8. Ircogel 905	8.5	0.9	5.1	0.5	Lubrizol
9. Aromatic 100	234.1	32.2	0.0	0.0	
10. p-Toluenesulfonyl Isocyanate	83.2	10.0	83.2	9.9	VanDeMark
11. Desmodur E-type resin	276.7	29.4	276.7	29.4	Bayer
Total	**1301.4**	**100.0**	**1021.5**	**61.04**	

Formulation Characteristics

Weight Solids %	78.5
Volume Solids %	61.1
Weight per gallon, lbs.	13.0
Recommended film thickness, mils	3-5
Minimum time to recoat, 77°F, 50% RH	4 hours
PVC, %	34.7
P/B Ratio	1.8
VOC, lbs./gal	2.8
Viscosity @ 77°F Kreb Units	63 ± 2

Manufacturing Procedure

1. Grind ingredients 1, 2, 5, 6 and 8 in 110 pounds of #9 to a 5 Hegman. A temperature of 110°F-130°F is necessary to activate the MPA-60X.
2. Stir in the MIOX, Aluminum, DC-11 and balance of #9.
3. Stir in the PTSI slowly to avoid excessive foaming.
4. Let the PTSI digest for at least 1 hour, but no more than 8 hours, then stir in the Desmodur E resin.

Typical Formulations

Moisture-Cure Urethane Coal Tar Formulation

Raw Materials	Weight Pounds	Volume Gallons	Weight Solids	Volume Solids	Supplier
1. Desmodur E-type resin	278.4	31.25	169.8	15.58	Bayer
2. KC-261, Coal Tar	199.7	22.09	199.7	2.09	Koppers
3. p-Toluenesulfonyl Isocyanate	28.5	2.66	28.5	2.66	VanDeMark
4. Bentone 34 Gel (see below)	135.9	17.72	17.0	1.77	NL Industries
5. Cyprufil 325	270.7	11.62	270.7	11.62	Cyprus
6. Xylene	93.7	12.93	0.0	0.00	
7. DC-11 (1% in A-100)	3.5	0.49	0.04	0.00	Dow Corning
8. Additive OF	9.3	1.24	9.3	1.24	Bayer
Total	**1019.7**	**100.0**	**695.0**	**54.96**	

Formulation Characteristics

Weight Solids %	68.2
Volume Solids %	55.0
VOC, lbs./gal	3.2
Weight per gallon, lbs.	10.2
Viscosity @ 77°F Kreb Units	70

Manufacturing Procedure

1. Make Bentone 34 Gel by mixing on Cowles Dissolver 10% by weight of Bentone with 85 parts of xylene and 5 parts of Anti Terra U. Stir until uniform gel is formed.
2. Mix ingredients 1, 2, 3, 4, and 5 in 53.7 pounds of xylene on a Cowles Dissolver until a 6+ Hegman is reached.
3. Turn off the Dissolver and cover the batch with the DC-11, Additive OF and the balance of the xylene.
4. Allow batch to set overnight with last addition covering the top. The next morning, stir in liquid layer and then package in clean dry containers.
5. Note: Additive OF and the PTSI will react with each other if added simultaneously.

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