SILQUEST* A-Link* 25 silane
SILQUEST A-Link 35 silane

SILANES - ADHESIVES & SEALANTS ADDITIVES

Silquest A-Link 25 silane and Silquest A-Link 35 silane are unique isocyanate functional silanes. The isocyanate functionality exhibits the typical reactivity of an alkyl isocyanate and will react with most active hydrogen containing compounds. The silane portion of these products can then crosslink with each other or be used to adhere to a variety of substrates.

Key Features and Typical Benefits

Their unique functionality makes them excellent candidates for use as:

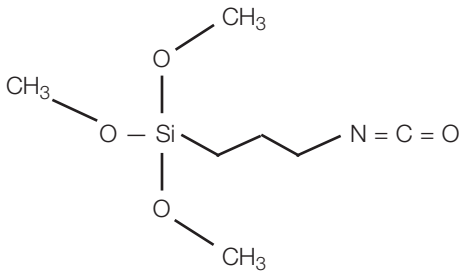
- crosslinkers for one-part moisture curable urethane adhesives, sealants and coatings when used to endcap diol or polyol polymers
- adhesion promoters for one-part moisture curable and two-part reactive urethane systems
- adhesion promoters for silicone sealants or coatings seeking enhanced adhesion to organic substrates on which active hydrogen species are present, such as nylon or other plastics

Typical Physical Properties

	Silquest A-Link 25 silane	Silquest A-Link 35 silane
Molecular Weight	247	205
Physical Form	Transparent Clear Liquid	Transparent Clear Liquid
Active Material	> 95%	> 95%
Specific Gravity (25/25°C)	0.999	1.073
Flashpoint (PMCC ASTM D 93	77 (171)	99 (210)

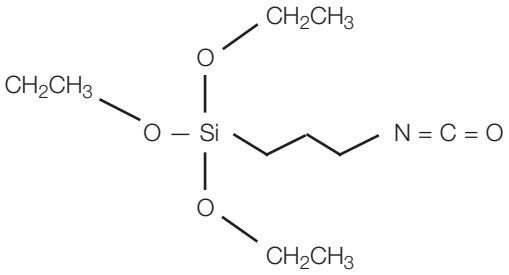
Chemical Structure

Silquest A-Link 35 silane



3-isocyanatopropyltrimethoxysilane

Silquest A-Link 25 silane



3-isocyanatopropyltriethoxysilane

Features and Typical Benefits of Silylated Urethanes: Isocyanate Functionality

Trimethoxysilyl (Silquest A-Link 35 silane)

- reactive with -OH, -NH_2, and -SH functional polyols and polymers, providing a moisture-cure silane crosslink mechanism
- provides good adhesion to difficult substrates when employed as an adhesion promoter
- offers fast hydrolysis in the presence of atmospheric moisture
- provides superior wet adhesion to glass, metal and other inorganic substrates
- resulting bond offers excellent thermal, chemical and UV stability

Triethoxysilyl (Silquest A-Link 25 silane)

- provides slower hydrolysis for applications requiring greater open time or enhanced shelf stability
- provides superior wet adhesion to glass, metal and other inorganic substrates
- resulting bond offers excellent thermal, chemical and UV stable performance

Application: Crosslinker for Polyurethane Polymers

Preparation of Silylated Polyurethane Polymers (SPUR+ Prepolymer):*

Silylated polyurethanes used in the following examples are prepared from a two-step reaction.

Step One: a hydroxyl terminated polyurethane intermediate is prepared by the reaction of a polyol or a blend of polyols with a diisocyanate in the presence of tin catalyst at 65 - 75°C. The reaction is pursued until NCO content reaches zero, by titration. Alternatively this step can be eliminated via the use of a high molecular weight diol.

Step Two: a stoichiometric amount of isocyanatosilane (Silquest A-Link 35 silane) is added to cap the hydroxyl terminated intermediates. The reaction is maintained at 65 - 75°C until NCO content drops to zero again.

Application: Crosslinker for Polyurethane Polymers (continued)

Table 1: The Properties of the SPUR+* (Silylated) Prepolymers A, B and C

Prepolymer Type	A	B	C
Polyols	8,000 mw polypropyleneoxide diol low monol	8,000 mw polypropyleneoxide diol low monol	4,000 mw polypropyleneoxide diol low monol with polyester polyol (70/30)
Diisocyanates	H12MDI	IPDI	MDI
Capped by silane	Silquest A-Link 35 silane	Silquest A-Link 35 silane	Silquest A-Link 35 silane
Viscosity of silylated prepolymer cps @ 25°C	49,300	56,700	44,000
Properties of Cured Pre-polymer (by ASTM D 412, ASTM C 661 and ASTM D 624)[a]			
Tensile Strength at break (psi)	50.2	65.5	93.7
Elongation at break (%)	277	310	100
Young's Modulus (psi)	–	–	290.9
Modulus at 100% extension (psi)	37.4	34.3	–
Tear Strength (lbs/in)	11.8	16	15.4
Shore A Hardness	12	17	30

(a) Those test methods are equivalent to ISO 34, 37 and 868

Application: Crosslinker for Polyurethane Polymers (continued)

Table 2: Formulation and Properties of SPUR+* Prepolymer-based Sealants

Sealant	Low Modulus	Medium Modulus	High Modulus
Sealant Formulation			
Silylated polyurethane (B type – Table 2)	100	100	–
Silylated polyurethane (C type – Table 2)	–	–	100
Plasticizer (DIDP, phr)	80	80	40
$CaCO_3$ Filler	240	240	90
UV stabilizer (phr)	2	2	2
Thixotrope, SiO_2,	5	5	5
Whitener, TiO_2	5	5	5
Desiccant Silquest A-171* silane (phr)	1.5	1.5	1
Adhesion Promoter: Silquest A-1120 silane (phr)	–	2.5	2
Silquest Y-11637 silane (phr)	2.5	–	–
Catalyst Fomrez** SUL 4 tin catalyst (phr)	0.2	0.2	0.15
Sealant Properties			
Viscosity (cps @ 25°C)	128,000	134,400	131,200
Tear strength (lbs/in)	35.4	39.5	42.2
Tensile strength at break (psi)	203.0	169.7	295.9
Elongation at break (%)	741	536	222
Young's Modulus (psi)	–	–	554
Modulus at 100% extension (psi)	50.8	79.8	210.3
Hardness (Shore A)	27	31	44

**Fomrez is a trademark of Chemtura Corporation

Table 3: Adhesion of the Sealants Tested According to ASTM C 794 (equivalent to ISO 8510-2)

Promoter Adhesion	SPUR+ Prepolymer-Based Sealant Using Silquest A-1120 silane	SPUR+ Prepolymer-Based Sealant Using Silquest Y-11637 silane
Aluminum, lbs/in	36 / 100% CF	30 / 85% CF
Glass, lbs/in	24 / 100% CF	33 / 90% CF

CF = Cohesive failure

Process for Making Silane Modified Polymers

Aside from the above-mentioned silylated polyurethanes, silanes are also used widely in the modification of other polymers to improve products' toughness, cohesive adherence to different materials, and heat and/or chemical resistance. Most of the modifications are conducted by grafting vinylsilanes or methacryloxysilanes to polymers via free radical reactions.

Silquest A-Link 25 silane and Silquest A Link 35 silane can make the modification more convenient over a wider range of polymers. For polymers containing functional groups -OH, -SH or $-NH_2$, the grafting (or capping) process can take place in production lines using either batch processing or continuous extrusion.

Two in-line processing examples follow:

1) Batch process:

In this laboratory experiment, Silquest A-Link 35 silane is used to modify 12,000 mw, low monol polypropyleneoxide diol in a Molteni mixer as part of a sealant production. These diols and Silquest A-Link 35 silane are charged to the mixer first. A tin catalyst is added after the temperature of the mixer has reached to 60 - 70°C. The reaction is pursued for one to one and half hours under that temperature until the NCO content reaches zero. Then the remaining ingredients of the formulation are added, in the sequence indicated above. The entire process of reaction and sealant mixing are under nitrogen protection.

In this experiment, the NCO/OH ratio is 1 and the high modulus sealant formulation (Table 2) is used. Properties of the sealant are listed in the table below:

Table 4: Properties of the Sealant Based on Silquest A-Link Silane-Modified Polyols

Tear strength (lbs/in)	20.2
Tensile strength at break (psi)	155
Elongation at break (%)	136
Young's Modulus (psi)	177
Hardness (Shore A)	46
Adhesion, (lbs/in)	
Aluminum	6.8 / 100% CF
Glass	6.5 / 100% CF

Process for Making Silane Modified Polymers (continued)

2) Brabender or extruder process:

In this silane-grafting experiment a terpolymer of ethylene-vinyl-acetate-hydroxyethyl acrylate, is used. Both Silquest A-Link 35 silane and Silquest A-Link 25 silane are grafted to the polymer in a Brabender mixer (Brabender Instruments) at 70 - 90°C for 12 minutes in the presence of tin catalyst. The silane-grafted terpolymers are then pressed into a film for testing. After fully cured, the gel content and high-fail temperature shear of the polymers are measured. Comparisons are shown in the following table:

Table 5: Properties of Silquest A-Link Silane-Grafted EVA Terpolymer

	Control (terpolymer)	Silquest A-Link 35 silane 1%	2%	Silquest A-Link 25 silane 2%	3%
Gel Content (%)	91	96	98	98	95
HFTS (°C)	65	69	71	70	75

HFTS = High-Fail Temperature in Shear tested according to ASTM D 4498.

These silane-modified polymers are used in hot melt adhesives to increase heat-fail temperature and can be used in a variety of construction applications to improve toughness. They also can be used to obtain excellent adhesion in sealants for automotive and insulated glass applications.

Application: Urethane Adhesion Promotion

One-Part Urethane

When incorporated at 0.5 to 1.5% in a one-part moisture curable urethane adhesive, sealant or coating, Silquest A-Link 25 silane and Silquest A-Link 35 silane provide excellent wet adhesion to glass, metal and more difficult plastic substrates such as nylon. The resulting adhesive bond offers excellent thermal, chemical and UV stability.

The silanes will react with isocyanate functional pre-polymers via the silane's isocyanate functionality in the presence of atmospheric or substrate surface moisture.

Two-Part Urethane

In a two-part urethane system, Silquest A-Link 25 silane and Silquest A-Link 35 silane are stable in the isocyanate side of the formulation. Again recommended use levels are 0.5 to 1.5% of the total system.

Application: Silicone Adhesion Promotion

Isocyanatosilanes also can be incorporated into silicone adhesives and sealants. In this case, the silane will bond into the silicone
network via its silane functionality in the presence of atmospheric or formulated moisture. The -NCO group however provides urethane like adhesion. As such, this material can interact with the surface via either its silane or NCO functionality.

Commercial Status

The silanes discussed in this bulletin are available commercially.

Patent Status

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US 5990257

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