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Torlon® AI-10 Coatings

Torlon® AI-10 is a soluble polyamide-imide sold in powder form. The polymer is tough, chemically resistant, and thermally stable. It has thermal capabilities similar to many polyimides, at a lower price. Coatings based on Torlon® AI-10 have been shown to be cost effective in electrical/electronic, high-temperature decorative, and corrosion prevention applications.

Magnet wire insulation and protective coatings for printed circuit boards are some of the electrical uses. Industrial applications include primers and decorative topcoats for cookware, appliances, and housewares. Torlon® AI-10 has been combined with fluoropolymers to produce high-performance, low-friction, corrosion-resistant coatings that provide protection to saw blades, gears, carburetor needles, and lawn and garden tools.

Torlon® AI-10 is also used for high-strength, high-temperature adhesives. Excellent bond strengths have been observed with stainless steel, aluminum and titanium alloys, and polyimide films.

This bulletin will briefly discuss amide-imide chemistry and the preparation of Torlon® AI-10 solutions and coatings based on them. In addition, the performance properties of the coatings, including corrosion resistance, will also be presented.

Product Description and Chemistry

Torlon® AI-10 is a reactive polyamide-imide designed to have a relatively low initial molecular weight for easy solubility and application ease. Typical properties of Torlon® AI-10 polymer are shown in Table 1.

Table 1: Typical properties of Torlon® AI-10

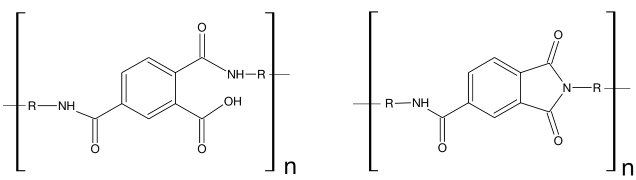
Property	Value[1]	Test Method[2]
Appearance	Yellow powder	
Volatile content	10 %	TTM 6510
Viscosity of solution with 25 % polymer	800 cps	TTM 6520 TTM 6535
Acid number	80 mg KOH/g	TTM 6540

[1] *Properties of individual batches will vary within specification limits.*
[2] *Test methods can be provided as necessary.*

The polymer is composed of trimellitic, aromatic amide, and aromatic imide moieties. As supplied, approximately 50 % of the polymer is in the un-imidized or amic acid form. When heated, the polymer will undergo cyclization to the imide form. Figure 1 shows the generalized structures for both forms.

Most Torlon® AI-10 polymer applications are coatings or films. The powder is dissolved in an appropriate solvent, formulated if desired, applied to a substrate, and then heated to achieve drying and cure.

Figure 1: Structure of Torlon® AI-10



Amic Acid Form   Imide Form

Heat treatment or curing is required to develop the high-performance properties of the polyamide-imide. Three processes occur during curing: removal of the solvent, imidization, and chain extension or molecular weight increase. At 93 °C – 149 °C (200 °F – 300 °F), the imidization reaction occurs through cyclization of the ortho carboxylic acid with the amide to form the five-membered imide ring with the evolution of water. Continued heating at 149 °C – 232 °C (300 °F – 450 °F) will remove most of the solvent with some chain extension occurring. Peak temperatures of 249 °C – 260 °C (480 °F – 500 °F) should be used to remove final traces of solvent and to develop optimum molecular weight and properties. A typical cure schedule for a clear film approximately 0.025 mm (0.001 inch) thick is 60 minutes at 149 °C (300 °F), 15 minutes at 260 °C (500 °F), and 5 minutes at 315 °C (600 °F).

The removal of the evolved water is the factor limiting cure speed. Thin films of uniform thickness can be cured rapidly. In general, if bubbling occurs reduce the rate of temperature increase. Torlon® AI-10 polymer is quite thermally stable, therefore it is quite difficult to "overcure".

Properties of Torlon® AI-10 films

Torlon® AI-10 is stronger and tougher than other thermally stable polymers, such as polyimides and polybenzimidazoles. Films made from Torlon® AI-10 have high flexural modulus and hardness, low coefficient of friction, and good adhesion.

Torlon® AI-10 polymers provide excellent electrical insulating properties at brief excursions to 454 °C (850 °F) without significant damage or severe loss of properties. Magnet wire coated with Torlon® AI-10 is rated for 20,000 hours at 225 °C or 437 °F (Class 220, ASTM D 2307). Electrical properties are retained in high humidity. Some typical properties of a cured Torlon® AI-10 film are shown in Table 2.

Table 2: Typical Torlon® AI-10 film properties

Property	Value
Color	Light amber
Refractive index	1.656
Glass transition temperature	272 °C (522 °F)
Tensile strength	117 MPa (17,000 psi)
Tensile modulus	3,032 MPa (440,000 psi)
Tensile elongation	23 %

When properly cured, Torlon® AI-10 polymers are highly resistant to most solvents and chemicals. Immersion tests have shown that the following chemicals have little or no effect:

- Acetone
- Benzene
- Dilute acids
- Dimethylacetamide
- Ethanol
- Hydraulic fluids
- Jet fuel
- Methylene chloride
- Perchloroethylene
- Refrigerants
- Toluene
- Xylene

Mild caustic solutions used at moderate temperatures, such as detergents, will not damage an Torlon® AI-10 film, but strong oxidizing acids, such as fuming sulfuric acid, or strong caustics will cause degradation.

Preparing AI-10 Solutions

Only strong aprotic materials are true solvents for polyamide-imide. Table 3 lists the usual solvents for Torlon® AI-10 polymer and the viscosity of a 32 weight percent solution. Of the listed solvents, N-methyl pyrrolidone (NMP) is preferred because it has low odor and a relatively low level of toxicity.

Table 3: Solvents for Torlon® AI-10

Solvent	Viscosity at 25 °C, poise
Dimethyl acetamide	13-21
Dimethyl sulfoxide	35-60
Dimethylformamide	Poor storage stability
N-methyl pyrrolidone	45-75
Acetone	Insoluble
Formamide	Insoluble

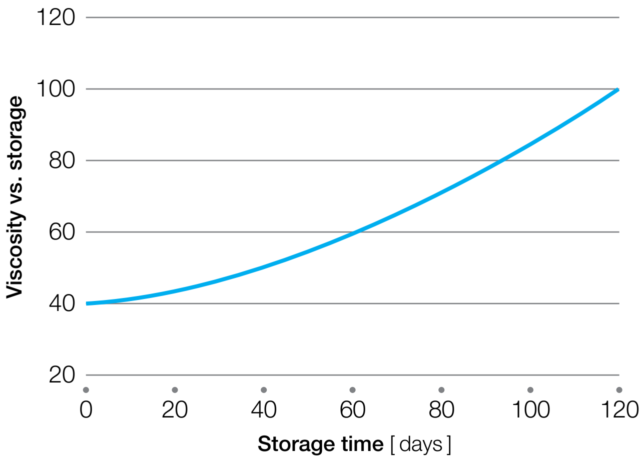
Because the true solvents for Torlon® AI-10 are relatively expensive, lower-cost materials, called diluents, are often used to reduce the viscosity of polyamide-imide solutions. Diluents can only be used within their solubility limits. Table 4 lists typical diluents and the solubility limits.

Table 4: Diluents for Torlon® AI-10 solutions

Diluent	% of Total Solvent (maximum)
Aromatic hydrocarbons	40-50
Ethyl acetate	40-50
Acetone	60
Cycolhexanone	80
Acetanilide	40

To prepare a solution of Torlon® AI-10 in NMP, add the AI-10 powder slowly to the solvent while stirring for 15 to 30 minutes. Continue stirring for 1 to 2 hours and then filter. The solution should be stirred again the day after preparation and again prior to use. Solution viscosity will increase with time; the rate depends upon the solvent used, the polymer concentration, and the storage conditions.

Figure 2: Viscosity vs. storage time*



** AI-10 solution in N-methly pyrrolidone, 35 % polymer*

Figure 2 shows the change in viscosity of a 35 wt. % Torlon® AI-10 solution over a four-month period. The viscosity increase is not detrimental to polymer performance. Usually the solution's viscosity can be adjusted with additional solvent and the solution used without loss of performance.

High-Temperature, Corrosion-Resistant, Decorative Enamels

To demonstrate the exceptional performance of enamels based on Torlon® AI-10 polymer, our laboratory has formulated four enamels, applied them to various substrates, and evaluated their performance.

Prior to preparation of the enamels, a solution of Torlon® AI-10 in NMP at a concentration of 35 wt. % polymer was prepared. See Table 5 below.

Table 5: Properties of the Torlon® AI-10 solution used in enamel preparation

Property	Value
Appearance	Amber liquid
Viscosity (1) at 25 °C (77 °F)	4,520 centipoise
Density	9.42 pounds/gallon
Actual % polymer	35
Measured % polymer (2)	37

(1) ASTM D2196, Brookfield Viscometer, Model HAT, Spindle #2 at 10 rpm

(2) Measured by weighing residue after heating 2 grams for 2 hours at 200 °C (392 °F) in an aluminum weighing dish.

The formulations of the enamels are shown in Table 6 and can be considered "starting points" for the development of commercially acceptable coatings.

The enamels were prepared by adding the silicone resin to the Torlon® AI-10 solution and then dispersing the pigments with a high speed disperser to a Hegman 7 grind. Additional solvent was then added to achieve spraying viscosity. The enamels were then spray applied to untreated cold rolled steel, Bonderite B-37 treated steel, and aluminum test panels. All panels were cured for 15 minutes at 260 °C (500 °F). The white and yellow enamels were air flashed for 15 minutes, and the red and green enamels were oven flashed for 10 minutes at 82 °C (180 °F) before curing.

The physical properties of the enamels on the various substrates are shown in Tables 7 through 9. The coatings show excellent adhesion and hardness with very good gloss and flexibility. Also shown is the effect of overbake, exposing the coating to two and three times the normal cure time. This test gives an indication of thermal stability, as well as processing flexibility. After overbake, the only effects observed were slight yellowing and minor loss of flexibility.

Table 10 lists the results of our evaluation of corrosion resistance by the salt spray technique. The results show excellent corrosion resistance, with very little rust creepage, or adhesion loss.

Table 6: Enamel formulations [parts by weight]

Formulation	White	Yellow	Red	Green
AI-10 polymer solution (Table 5)	538.2	577.0	576.9	576.9
Silicone resin [(1)]	3.4	3.3	3.5	3.5
Titanium dioxide [(2)]	188.3	100.9	100.9	100.9
Red iron oxide [(3)]			30.4	
Nickel titanate yellow [(4)]		30.4		
Chrome oxide green [(5)]				30.4
Grind on Cowles Dissolver, then add:				
N-methyl pyrrolidone	194.0	207.7	207.6	207.6
150 type aromatic solvent [(6)]	76.0	80.8		
Total	1,000.0	1,000.0	1,000.0	1,000.0
Non volatile	38.0	33.7	33.7	33.7
Pigment/binder ratio	1.0	0.65	0.65	0.65

[(1)] SR-112, General Electric, Silicones Division
[(2)] Ti-Pure® R-900, E. I. duPont de Nemours and Co.
[(3)] R-3200, Pfizer Minerals, Pigments and Metals Division
[(4)] #14, Shepard Chemical Company
[(5)] G-6099, Pfizer Minerals, Pigments and Metals Division
[(6)] Hi-Sol®, 15, Ashland Chemicals

Table 7: Physical properties of Torlon® AI-10 enamels applied to aluminum

Property	White		Yellow		Red		Green		ASTM Test Method
Hardness, sward	30		18		22		28		D2134
Hardness, pencil	2H		2H		3H		4H		D3363
Impact, direct [in-lbs]	30		30		40		30		D2794
Impact, reverse [in-lbs]	20		20		40		30		D2794
Crosshatch-adhesion [% pass]	100		100		100		100		D3359B
Conical bend [% pass]	100		100		100		100		D522
Yellowness index	41.4		51.1						E313
Gloss, 20°	39		48		62		76		D523
Gloss, 60°	84		89		98		102		D523
Overbake* [%]	**100**	**200**	**100**	**200**	**100**	**200**	**100**	**200**	
Hardness, sward	22	20	22	26	26	26	24	28	D2134
Hardness, pencil	3H	3H	2H	3H	3H	3H	3H	2H	D3363
Impact, direct [in-lbs]	20	10	20	20	20	30	30	40	D2794
Impact, reverse [in-lbs]	10	10	30	20	30	20	30	30	D2794
Crosshatch-adhesion [% pass]	100	100	100	100	100	100	100	100	D3359B
Conical bend [% pass]	95	95	100	100	100	100	100	100	D522
Yellowness index increase [%]	5	16	5	7					E313
Gloss, 20°; retention [%]	103	95	96	96	97	97	96	97	D523
Gloss, 60°; retention [%]	100	102	101	97	101	100	100	100	D523

** 15 min at 260 °C (500 °F)*

Table 8: Physical properties of Torlon® AI-10 enamels applied to Bonderite 37

Property	White		Yellow		Red		Green		ASTM Test Method
Hardness, sward	24		28		30		34		D2134
Hardness, pencil	> 5H		> 5H		> 5H		> 5H		D3363
Impact, direct [in-lbs]	100		120		100		120		D2794
Impact, reverse [in-lbs]	60		120		80		80		D2794
Crosshatch-adhesion [% pass]	100		100		100		100		D3359B
Conical bend [% pass]	100		100		90		90		D522
Yellowness index	42.4		49.6						E313
Gloss, 20°	41		57		51		46		D523
Gloss, 60°	92		98		97		98		D523
Overbake* [%]	**100**	**200**	**100**	**200**	**100**	**200**	**100**	**200**	
Hardness, sward	30	32	22	26	30	32	22	36	D2134
Hardness, pencil	> 5H	> 5H	> 5H	> 5H	> 5H	> 5H	> 5H	> 5H	D3363
Impact, direct [in-lbs]	70	80	100	120	70	60	80	80	D2794
Impact, reverse [in-lbs]	20	30	80	70	40	40	50	60	D2794
Crosshatch-adhesion [% pass]	100	100	100	100	100	100	100	100	D3359B
Conical bend [% pass]	70	75	90	80	100	100	100	100	D522
Yellowness index increase [%]	9	8	2	9					E313
Gloss, 20°; retention [%]	100	97	94	97	93	96	93	100	D523
Gloss, 60°; retention [%]	99	99	96	99	100	100	101	100	D523

** 15 min at 260 °C (500 °F)*

Table 9: Physical properties of Torlon® AI-10 enamels applied to cold rolled steel

Property	White		Yellow		Red		Green		ASTM Test Method
Hardness, sward	24		24		32		40		D2134
Hardness, pencil	> 5H		> 5H		> 5H		> 5H		D3363
Impact, direct [in-lbs]	100		140		160		160		D2794
Impact, reverse [in-lbs]	60		120		160		160		D2794
Crosshatch-adhesion [% pass]	100		100		100		100		D3359B
Conical bend [% pass]	100		100		100		100		D522
Yellowness index	49.4		54.1						E313
Gloss, 20°	39		72		41		48		D523
Gloss, 60°	94		108		99		102		D523
Overbake* [%]	**100**	**200**	**100**	**200**	**100**	**200**	**100**	**200**	
Hardness, sward	26	24	24	30	28	28	26	24	D2134
Hardness, pencil	> 5H	> 5H	> 5H	> 5H	> 5H	> 5H	> 5H	> 5H	D3363
Impact, direct [in-lbs]	60	60	120	120	100	60	120	120	D2794
Impact, reverse [in-lbs]	20	30	70	50	50	40	80	70	D2794
Crosshatch-adhesion [% pass]	100	100	100	100	100	100	100	100	D3359B
Conical bend [% pass]	90	90	100	100	100	100	100	100	D522
Yellowness index increase [%]	7	5	5	8					E313
Gloss, 20°; retention [%]	98	97	97	95	97	97	96	98	D523
Gloss, 60°; retention [%]	100	99	100	99	100	100	100	100	D523

** 15 min at 260 °C (500 °F)*

Table 10: Salt spray resistance of enamels (ASTM B-177)

Property	White		Yellow		Red		Green	
Substrate	CRS	B37	CRS	B37	CRS	B37	CRS	B37
Test length [hours]	96	240	96	240	96	240	96	24
Rust creepage [32nds inch]	1	2	2	2	2	1 – 2	2	1 – 2
Adhesion loss [32nds inch]	0	1 – 2	4	1 – 2	4	0	4	0

www.solvay.com

SpecialtyPolymers.EMEA@solvay.com | Europe, Middle East and Africa

SpecialtyPolymers.Americas@solvay.com | Americas

SpecialtyPolymers.Asia@solvay.com | Asia Pacific

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