

ANCAMIDE® 351A

Curing Agent

DESCRIPTION

Ancamide 351A curing agent is a member of a new series of reactive polyamides developed for use in the curing of epoxy resins in coatings and adhesives applications. Special features include good colour, colour stability, excellent corrosion resistance and superior adhesion properties. It is freely compatible with a wide range of solvents, including those generally used in epoxy resin based coatings.

TYPICAL PROPERTIES

Property	Value	Unit	Method
Appearance	Clear Amber Liquid		
Colour	8 max	Gardner	ASTM D 1544-80
Viscosity @ 25°C	10,000-20,000	mPa.s	Brookfield RVTD, Spindle 4
Amine Value	370±20	mg KOH/g	Perchloric Acid Titration
Specific Gravity @ 21°C	0.97		
Equivalent	100	Wt/{H}	
Recommended use Level	50-55	PHR	With Bisphenol A diglycidyl ether (EEW=190)
Solids Content	100	%	

ADVANTAGES

- Good corrosion resistance
- Excellent adhesion to a variety of substrates
- Good colour and light stability
- Compatible with wide range of solvents

APPLICATIONS

- Structural adhesives
- Solvent based marine and protective coatings
- Primers, sealers and coatings for concrete

SHELF LIFE

At least 24 months from the date of manufacture in the original sealed container at ambient temperature. Store away from excessive heat and humidity in tightly closed containers.

STORAGE AND HANDLING

Refer to the Safety Data Sheet for Ancamide 351A curing Agent.

TYPICAL HANDLING PROPERTIES [@ 50 PHR]*

Property	Value	Unit	Method
Gel Time (150g mix @ 25°C)	150	mins	Techne GT-3 Gelation Timer
Thin Film Set Time 25°C	10	h	BK Drying Recorder Phase II

* With Bisphenol A diglycidyl ether (EEW=190)

TYPICAL CURE SCHEDULE

2-7 Days at Room Temperature

FILM PROPERTIES AFTER 10 DAYS CURE @ 23°C

Property	Value	Unit	Method
Direct impact	40	cm.kg	
Reverse impact	10	cm.kg	
Flexibility Cylindrical Mandrel	3	mm pass	Erichsen mandrel tester 266

STRUCTURAL ADHESIVES APPLICATIONS

SUPPLEMENTARY INFORMATION

Ancamide 351A curing agent is medium viscosity polyamide, supplied at 100% solids. The product is intended for use in combination with or without other amine curing agent in two part room temperature or heat cured adhesives formulations. When used with a standard DGEBA type liquid epoxy resin (EEW=190) Ancamide 351A curing agent offers excellent handling and physical properties and adhesion to various substrates.

Ancamide 351A curing agent has a non-critical loading and can be used at levels ranging from 40 to 70 phr with the preferred range being 50-60 phr with a standard DGEBA type liquid epoxy resin.

PHYSICAL PROPERTIES

Physical properties of Ancamide 451A curing agent cured standard liquid epoxy resin was compared with Ancamide 350A curing agent cured epoxy after seven days ambient temperature cure and after a 30 minutes cure @ 120°C. The tensile data after seven days ambient cure schedule shows that Ancamide 351A curing agent cured epoxy resin offers greater than 30% higher tensile strength and greater than 80% higher tensile elongation without any adverse effect on tensile modulus. This behavior indicates that Ancamide 351A curing agent provides inherent toughness to the cured epoxy resin.

The tensile data after heat cure schedule shows that Ancamide 351A curing agent demonstrated more than 25% increase in tensile elongation as compared to Ancamide 350A curing agent without any adverse impact on tensile strength. In addition, increased elongation is observed without significant reduction in glass transition temperature.

TABLE 1: PHYSICAL PROPERTIES

	Ancamide 350A	Ancamide 351A
DGEBA liquid epoxy resin	100	100
Ancamide 350A	55	0
Ancamide 351A	0	55
After 7 Days RT Cure		
Tensile Strength, MPa (psi)	44.1 (6400)	59.3 (8600)
Tensile elongation at failure, %	1.8	3.4
Tensile Modulus, GPa (ksi)	2.81 (407)	2.84 (412)
Tg, °C	50	50
After 30 minutes @ 120°C Cure		
Tensile Strength, MPa (psi)	58.6 (8500)	57.2 (8300)
Tensile elongation at failure, %	5.5	7.0
Tensile Modulus, GPa (ksi)	2.51 (364)	2.36 (342)
Tg, °C	100	96

STARTING POINT FORMULATION

A two-part model formulation (Appendix 1) was prepared using Ancamide 351A curing agent as a main curative. Ancamine 2641 curing agent, a modified amine, was used as a co-curing agent along with Ancamide 351A curing agent. Typical fillers and additives were used in the same formulation. A control formulation was prepared using Ancamide 350A curing agent.

Epoxy portion and a curative portion were mixed together based on theoretical EEW of epoxy portion and AHEW of a curative using 1:1 stoichiometric ratio. The mixed adhesive system was used to make single lap shear samples and peel samples for further testing. Substrates used were cold rolled steel (CRS), ABS and polycarbonate. All substrates were used without any surface treatment prior to bonding.

For CRS lap shear samples, 0.5" overlap was used whereas for ABS and polycarbonate substrates 1.0" overlap was used. Only CRS substrate was used to prepare peel samples. In all cases 10 mils bondline thickness was maintained using 10 mils diameter glass beads.

All samples were cured following two cure schedules, (1) seven days ambient temperature cure or (2) 30 minutes at 120°C cure. The samples were tested at ambient temperature following standard test methods.

PERFORMANCE EVALUATION

TABLE 1: PHYSICAL PROPERTIES

	Ancamide 351A based B component	Ancamide 350A based B component Control
7 days @ 25°C cure		
CRS/CRS shear strength, MPa (psi)	12.4 (1800)	11.7 (1700)
ABS/ABS shear strength, MPa (psi)	3.6 (520)	2.5 (360)
Polycarbonate/PolycarbonateShear strength, MPa (psi)	1.6 (230)	1.7 (250)
CRS/CRS peel strength, N/mm (pli)	1.8 (10.5)	1.7 (9.8)
30 minutes @ 120°C cure		
CRS/CRS shear strength, MPa (psi)	13.8 (2000)	14.1 (2050)
CRS/CRS peel strength, N/mm (pli)	2.5 (14.0)	2.4 (13.5)

LAP SHEAR STRENGTH: After room temp cure and after heat cure schedules, formulation based on Ancamide 351A curing agent demonstrated performance very similar or slightly higher than the formulation based on Ancamide 350A curing agent. The mode of failure for CRS was 100% cohesive and adhesive for thermoplastic substrates.

PEEL STRENGTH: After both cure schedules, Ancamide 351A curing agent based formulation exhibited little higher peel strength compared to the Ancamide 350A curing agent based formulation. In both cases, the mode of failure was cohesive.

SOLVENT BASED MARINE AND PROTECTIVE COATINGS APPLICATION

FILM PROPERTIES

In a clear coat formulation Ancamide 351A curing agent also exhibits a good degree of flexibility. As shown in Table 3, the direct impact resistance of Ancamide 351A curing agent is comparable to Ancamide 350A curing agent. Ancamide 351A curing agent formulations can also be accelerated using Ancamine® K54 (phenolic, tertiary amine accelerator). Levels can be varied to achieve the desired dry speed properties, with the preferred level in the 2.5-5.0% range based on Ancamide 351A use level.

TABLE 3: CLEAR COAT PERFORMANCE PROPERTIES

Property	Ancamide 351A(*)	Ancamide 350A(*)
Direct Impact	40	40
Reverse Impact	10	10
Dry Speed BK Phase II [h]	9:30	10:00
Dry Speed (+2.5% K54) [h]	5:00	5:30
Specular Gloss 60°	90	95
Cross Hatch Adhesion (dry)	Gt0	Gt0

(*) Ancamide 351A and Ancamide 350A tested with liquid Bis A diglycidyl ether (EEW 190), Cross hatch adhesion Gt0 = no loss of adhesion.

STARTING POINT FORMULATIONS

Appendix 2 contains an anti-corrosive primer formulation based on Ancamide 351A. As with all solvent based coatings, dry times are influenced by solvent choice, so dry times may vary depending upon the ratio of polar and non-polar solvents incorporated into the coating formulation. Pot lives as measured by the time for the mix to double in viscosity are in the 3-6 hours range, which is typical for such solvent based polyamide systems.

Formulation A351A-1, with Liquid Epoxy Resin, is 64% volume solids. PVC is 35% for both formulations. The primer formulation is compatible with a variety of different let down solvents (including xylene, n-butanol, MIBK, methoxy-propanol, etc) and can be further let down if required for additional spray applications. The coating formulation can be applied with conventional spray equipment or brush applied to the steel substrate.

Formulation A351A-1 was evaluated for corrosion resistance properties using salt spray, Distilled Water Immersion and NaCl 3.5% immersion at room temperature. After 1000 h exposure, coatings exhibit excellent corrosion resistance. No signs of field blisters have been detected using the above tests, with panels exhibiting no scribe creep in the salt spray test. For comparative purposes, Ancamide 350A curing agent was also included in the test study as the “industry” standard reference. In all the corrosion resistance tests carried out, both Ancamide 351A and Ancamide 350A curing agents demonstrated comparable performance.

PERFORMANCE EVALUATION

Formulation A351A-1 was evaluated in 5% salt spray, distilled water immersion at room temperature and NaCl 3.5% immersion at room temperature, following a 10 day ambient cure of applied coatings.

Coatings were applied to grit blasted, hot rolled steel (SA2.5), using conventional spray equipment, in double coats to give coatings with a 75-100 μ dry film thickness (DFT). In salt spray, (ASTM B-117) panels were scribed and evaluated for field blisters using the US Federal Standard Test Method 141a, Method 6461 and the scribe creep was rated in accordance with ASTM D-1654. Similar evaluations were made for panels placed in NaCl 3.5% immersion at room temperature and distilled water immersion. Panels exposed to distilled water immersion were not scribed and coatings were assessed for blistering only. These tests also included evaluations for changes in visual appearance.

CORROSION RESISTANCE

Anti-corrosive primers based on Ancamide 351A and Ancamide 350A curing agents were evaluated for salt spray, distilled water and NaCl 3.5% immersion at room temperature. The results obtained are presented in Table 4, 5 and 6. After 1000hrs salt fog exposure, formulations with Ancamide 351A curing agent or Ancamide 350A curing agent exhibit excellent resistance. Both formulations also demonstrated excellent water and salt water resistance with no signs of field blistering being observed following 1,000 h continuous testing.

TABLE 4: SALT SPRAY RESISTANCE — ANCAMIDE 351A AND ANCAMIDE 350A [1000h]

	Ancamide 351A	Ancamide 350A
Overall rating	9-10	9-10
Scribe creep	0-1mm	0-1mm
X-hatch adhesion after 1,000 h	5B	5B

5% salt spray, cabinet temperature 35°C - ASTM B-117, film thickness 75-100 μ ; Rating: 10 = Best (no blisters), 0 = Worst

TABLE 5: DISTILLED WATER IMMERSION @ RT — ANCAMIDE 351A AND ANCAMIDE 350A [1000h]

	Ancamide 351A		Ancamide 350A	
	Before Immersion	After 1,000 h	Before Immersion	After 1,000 h
X-Hatch Adhesion	5B	5B	5B	5B
Gloss/Appearance	No change	No change	No change	No change

TABLE 6: NaCl 3.5% IMMERSION @ RT — ANCAMIDE 351A AND ANCAMIDE 350A [1000h]

	Ancamide 351A		Ancamide 350A	
	Before Immersion	After 1,000 h	Before Immersion	After 1,000 h
X-Hatch Adhesion	5B	5B	5B	5B
Gloss/Appearance	No change	No change	No change	No change
Scribe creep	0 mm	0 mm	0 mm	0 mm

APPENDIX 1

ANCAMIDE 351A CURING AGENT: TWO-PART ADHESIVE FORMULATION

A-Component (g)

1. Epoxy Resin	DER 331	Olin	60
2. Filler	Microtuff AG 445	Speciality Minerals	38
3. Additive	Cab-O-Sil TS 720	Cabot Industries	2
TOTAL A			100

A-Component Manufacture Procedure:

Charge components 1-3 and stir homogeneous at medium to high shear

B-Component (g) Control

1. Curing agent	Ancamide 350A	Evonik	40
2. Curing agent	Ancamine 2641	Evonik	10
3. Filler	Microtuff AG 445	Speciality Minerals	22
4. Filler	Toyol 101 Aluminium powder	Toyol-America	27
5. Additive	Cab-O-Sil TS 720	Cabot Industries	1
TOTAL B			100

B-Component (g) Ancamide 351A

1. Curing agent	Ancamide 351A	Evonik	40
2. Curing agent	Ancamine 2641	Evonik	10
3. Filler	Microtuff AG 445	Speciality Minerals	22
4. Filler	Toyol 101 Aluminium powder	Toyol-America	27
5. Additive	Cab-O-Sil TS 720	Cabot Industries	1
TOTAL B			100

B-Component Manufacture Procedure:

Charge components 1-5 and stir homogeneous at medium to high shear

TECHNICAL DATA

EEW of A Component	317
AHEW of B Components	201
Mixing ratio (by weight)	100 of A : 63 of B
Solid content wt. (%)	100

APPENDIX 2

FORMULATION A351-1: ANCAMIDE 351A CURING AGENT ANTI-CORROSIVE PRIMER
FORMULATION

Anti-Corrosive Primer			
A-Component (g)			
1. Epoxy Resin	DER 331	Olin	229.40
2. Diluent	Epodil LV5	Evonik	22.90
3. Diluent	Epodil 748	Evonik	22.20
4. Anti-Terra U80	Anti-Terra U-80	Byk Chemie	3.10
5. Additive	Bentone SD-2		11.50
6. Solvent	MIBK		15.30
7. Solvent	Xylene		45.90
8. Solvent	Dowanol PM	Dow	15.30
9. Filler	Barite		152.90
10. Filler	Talc 400 mesh		191.10
11. Filler	Quartz 400 mesh		191.10
12. Pigment	Zinc Phosphate	Heubach	30.60
13. Pigment	Bayferrox 130M	Bayer	68.70
TOTAL A			1000.00
B-Component (g)			
1. Curing Agent	Ancamide 351A	Evonik	118.87
2. Additive	Ancamine K54	Evonik	4.99
3. Solvent	Xylene		120.01
4. Solvent	Butanol		72.03
			315.90
TOTAL A+B			1315.90

After mixing Part A and B, apply a 30 minute induction time prior to application.

TECHNICAL DATA

Mixing Ratio	Volume A:B	3:2
Density (g/ml)	- Mix	1.47
Solid Content (Weight %)	- Mix	79.55
Solid Content (Volume %)	- Mix	64.17
PVC	%	35.19%
Pot-life (h)	h	3.5
Gloss (60°)		17
Hard Dry Thumb twist	h	16.00
Epoxy/Amine		1.08

SALT SPRAY PANELS 1000h EXPOSURE

Ancamide 350A

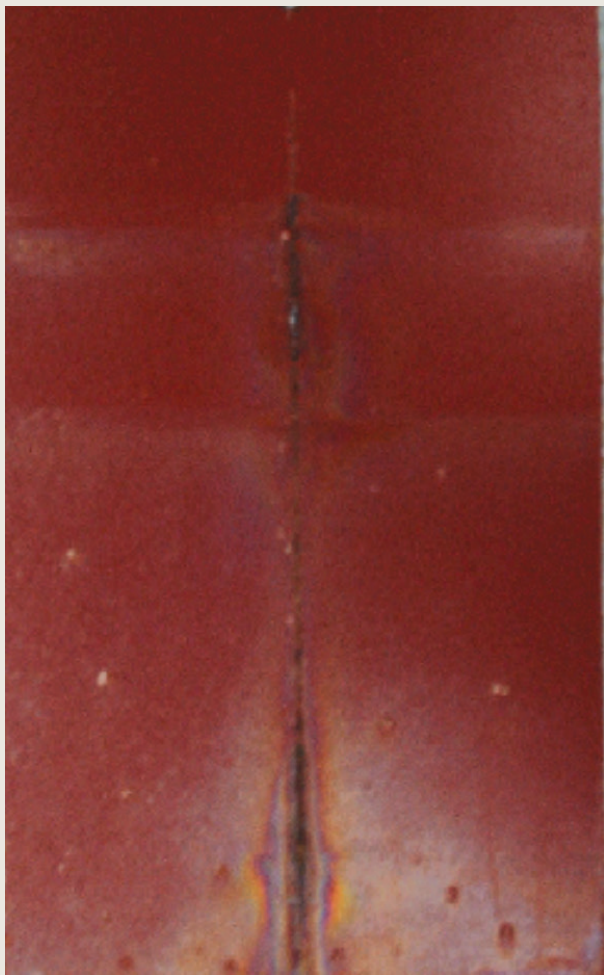


Ancamide 351A



NACL 3.5 IMMERSION @ ROOM TEMPERATURE 1000h EXPOSURE

Ancamide 350A



Ancamide 351A



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