

Product information

ANCAMINE[®] 2441

Curing Agent

DESCRIPTION

Ancamine 2441 curing agent is a modified polyamine which was designed for use as a latent curing agent for one-component adhesive formulations. The product can also be used as an accelerator for dicyandiamide (dicy). Its combination of low temperature activation, good shelf life and high glass transition temperature permits use in many applications requiring latency and high performance

TYPICAL PROPERTIES

Property	Value	Unit
Appearance	White, Micronized Powder	
Amine Value	210-250	mg KOH/g
Particle Size	90% < 10	micron
Recommended Use Level (EEW=190)		phr
As A Dicy Cure Accelerator	2-8	
As A Sole Curing Agent	20	

ADVANTAGES

- Excellent reactivity
- Improved green strength development
- Good formulation shelf stability
- High glass transition temperature

APPLICATIONS

- One-component convection or induction heat-cured adhesives
- Powder coatings
- Potting compounds
- Coatings
- Hot-melt prepregs
- Anhydride accelerator

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 Ancamine 2441 curing agent.

TYPICAL CURE SCHEDULE

30 minutes at 250°F.

TYPICAL FORMULATION* (COMPOSITION BY WEIGHT)

Property	I	II	Method
Liquid Epoxy Resin	100	100	Standard liquid epoxy resin with an epoxide equivalent weight of approximately 190.
Amicure® CG-1200	6.0	0	Dicyandiamide—Product of Evonik
Ancamine 2441	5.0	20.0	Modified Amine—Product of Evonik
Fumed Silica	1.0	1.0	Fumed silica—Product of Cabot Corporation

TYPICAL PERFORMANCE*

Property	I	II	Unit
DSC Activation Temperature	255	211	°F
Pot Life (150g mix @ 110°F)	>3	>3	month
Glass Transition Temperature (DSC)	276	238	°F
Lap Shear Strength (CRS)	1,200	1,600	psi

* Ancamine 2441 curing agent formulated with standard Bisphenol-A based (DGEBA, EEW=190) epoxy resin.

SUPPLEMENTARY INFORMATION

REACTIVITY:

To evaluate the reactivity of Ancamine 2441 curing agent, an initial screening was conducted that compared basic formulations containing standard liquid epoxy resin, Ancamine 2441 with and without dicy, and as a control, Ancamine 2014AS with and without dicy.

As shown in Table 4, the formulations containing Ancamine 2441 curing agent exhibit lower onset temperatures than the control as a dicy cure accelerator or as a sole curing agent, indicating improved reactivity. In addition, when used as a sole curative, Ancamine 2441 shows much improved low-temperature reactivity versus when it is used as a dicy accelerator

GLASS TRANSITION TEMPERATURE:

Samples of cured formulations were analyzed for glass transition temperature (T_g) using DSC. The scan rate was 18°F/min starting at 68°F. Whether used as a dicy cure accelerator or as a sole curative, Ancamine 2441 curing agent imparts significantly higher glass transition temperatures than the control.

SHEAR STRENGTH DEVELOPMENT:

Shear strength development was measured using cold rolled steel coupons that were bonded together with a ½ inch overlap and a 20 mil bondline thickness. The samples were cured in an air circulated oven for 30 minutes at 248°F and 284°F. The purpose of this evaluation was to demonstrate adhesive utility prior to post-baking. Due to the improved low-temperature reactivity, Ancamine 2441 curing agent, both with and without dicy, demonstrates improved shear strength development as compared with the control (see Figures 1 and 2).

TABLE 4: COMPARATIVE FORMULATIONS* AND REACTIVITY** COMPOSITION (% BY WT)

INGREDIENT	A	B	C	D	Method
Liquid Epoxy Resin	100	100	100	100	Standard liquid epoxy resin with an epoxide equivalent weight of approximately 190.
Amicure CG-1200	6	6	-	-	Dicyandiamide—Product of Evonik
Ancamine 2014AS	5	-	28	-	Modified Amine—Product of Evonik
Ancamine 2441	-	5	-	20	Fumed silica—Product of Cabot Corporation
Cab-O-Sil TS-720	1	1	1	1	Fumed silica—Product of Cabot Corporation
Onset Temperature (°F)	271	255	228	210	
Peak Temperature (°F)	302	300	264	241	
Heat of Reaction (J/g)	385	430	212	230	
T _g (°F)	239	277	131	237	

* Formulations were for comparative purposes only. For maximum performance, optimize components and use additives.

** By DSC at a 18°F/min scan rate 1 Standard liquid epoxy resin with an epoxide equivalent weight of approximately 190.



FIGURE 1: SHEAR STRENGTH AT 77°F

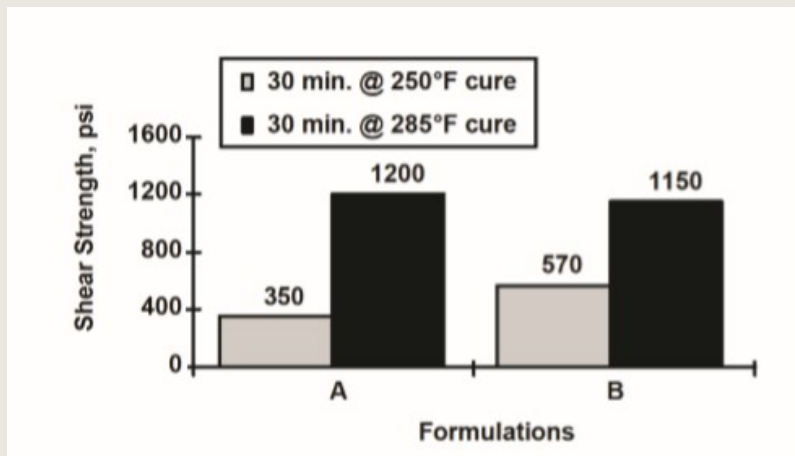
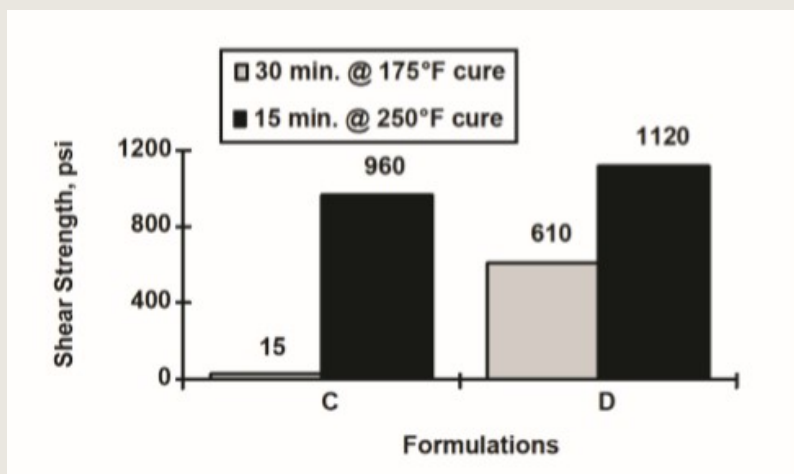


FIGURE 2: SHEAR STRENGTH AT 77°F



Partially cured samples (15 minutes @ 250°F) were tested at 300°F, a higher temperature than cure temperature, to evaluate the shear strength retention at higher temperatures. As Figure 3 indicates, the formulation containing Ancamine 2441 curing agent (D) also exhibit improved strength retention as compared with the control.

FORMULATION SHELF STABILITY:

To evaluate the shelf stability of the model formulations, an accelerated aging study was conducted for four weeks at 110°F. Twenty-four hours after mixing and degassing, the viscosity of the formulations was measured at room temperature. The formulations were stored at 110°F, and their viscosities were measured (after equilibrating to room temperature) every week.

As Figure 4 indicates, the formulations containing Ancamine 2441 curing agent (B and D) exhibit a small increase in viscosity after four weeks at 110°F. The improved reactivity, as demonstrated in previous figures, did not have an adverse affect on shelf stability.

EVALUATION IN INDUCTION HEAT-CURED ADHESIVE FORMULATIONS:

Two formulations, E and F (Table 5), were prepared to characterize Ancamine 2441 as a co-curing agent for induction heat-cured adhesive applications. Formulation E contains Ancamine 2014AS curing agent as the control. The formulations were characterized for green strength/shear strength development, shear strength retention, shear strength after post-bake, shear strength after environmental exposure and formulation shelf stability.

Formulations E and F were tested for shear strength under different conditions using EG and HDG coupons that were coated with Ferrocote 61-MAL-HCL-1 oil. The coupons were bonded together with a ½ inch overlap and a 10 mil bondline thickness and were cured using a lab scale induction heat unit at different cure schedules.

FIGURE 3: SHEAR STRENGTH* AT 300°F

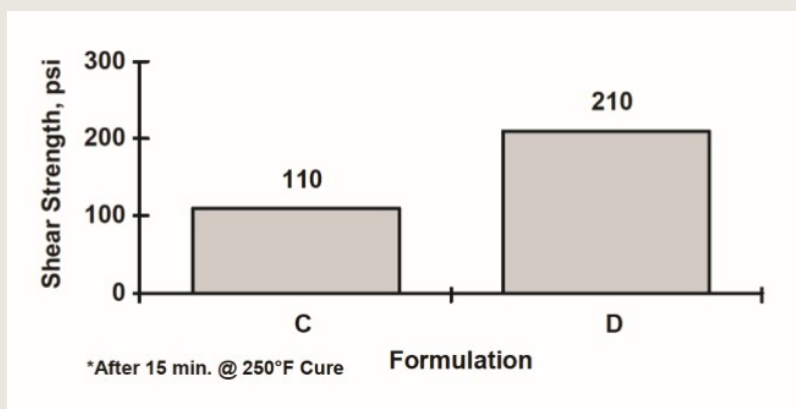


FIGURE 4: FORMULATIONS SHELF STABILITY % VISCOSITY INCREASE AT 110°F AFTER 4 WEEKS

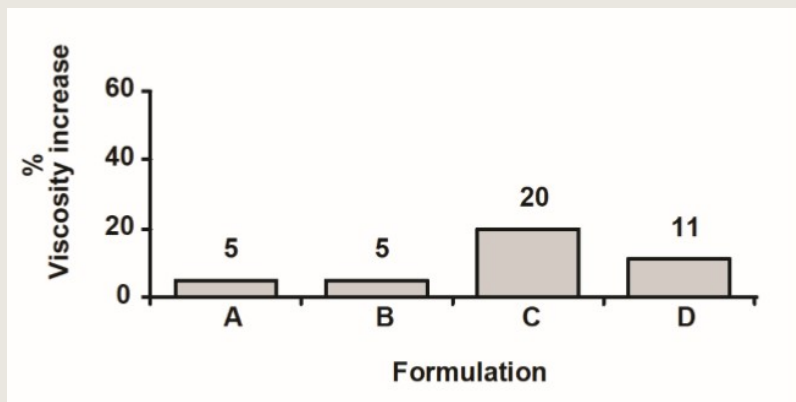


TABLE 5: COMPARATIVE FORMULATIONS* COMPOSITION (% BY WEIGHT)

Formulation Ingredient	E	F	Method
Liquid epoxy resin	87.5	87.5	Standard liquid epoxy resin with an epoxide equivalent weight of approximately 190.
Kelpoxy G292-100	25.0	25.0	Hycar 1300X13—Epoxy adduct from Reichhold Chemicals, Inc.
Amicure CG-1200	7.0	7.0	Dicyandiamide—Product of Evonik,
Ancamine 2014AS	10.0	-	Modified amines—Products of Evonik
Ancamine 2337S	12.5	12.5	Modified amines—Products of Evonik
Ancamine 2441	-	10.0	Modified amines—Products of Evonik
Aluminum 101	40.0	40.0	Filler—Product of Alcan Toyo of America.
Cab-O-Sil TS-720	2.0	2.0	Fumed silica—Product of Cabot Corporation

* Formulations were for comparative purposes only. For maximum performance, optimize components and use additives.

GREEN STRENGTH DEVELOPMENT AND STRENGTH RETENTION:

The purpose of this evaluation was to demonstrate the adhesive utility prior to post-baking. Each formulation was induction-cured for four seconds at 375°F and 400°F (one-second ramp and three-second hold) and evaluated under three different conditions:

- 1) Strength within 10 minutes of induction heat cure
- 2) Strength at room temperature after 24 hours
- 3) Strength at 245°F and 300°F after 24 hours

Green strength was measured using electro-galvanized steel coupons that were bonded together with a ½ inch overlap and a 10 ml bondline thickness. Figures 5 and 6 show green strength development for the two formulations within 10 minutes and 24 hours at room temperature after specific induction cure cycles. As shown in Figure 5, the system containing Ancamine 2441 curing agent (F), due to its improved reactivity, imparts twice as much green strength as the control. Results in Figure 7 indicate that the Ancamine 2441-based formulation (F) offers significantly higher strength retention on HDG and EG substrates than the control.

SHEAR STRENGTH AND SHEAR STRENGTH RETENTION AFTER POST-CURE:

The ultimate lap shear strength capabilities of the model formulations were determined following a full cure achieved with a combination of induction and convection heating. Samples were induction-heated and post-cured for 30 minutes at 300°F, prior to testing at room temperature. Samples were also tested at 300°F for strength retention after the post-cure. In addition, the formulations were evaluated for shear strength retention after seven days of soaking in water at 130°F.



FIGURE 5: GREEN STRENGTH* AT 77°F WITHIN 10 MINUTES

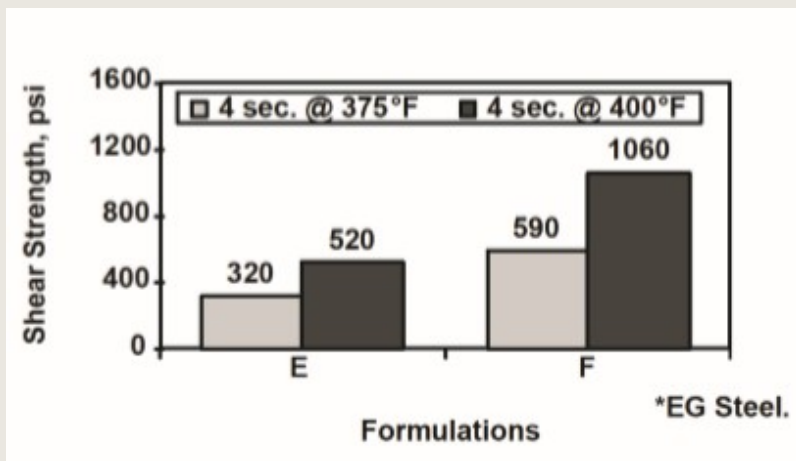


FIGURE 6: GREEN STRENGTH* AT 77°F WITHIN 24 HOURS

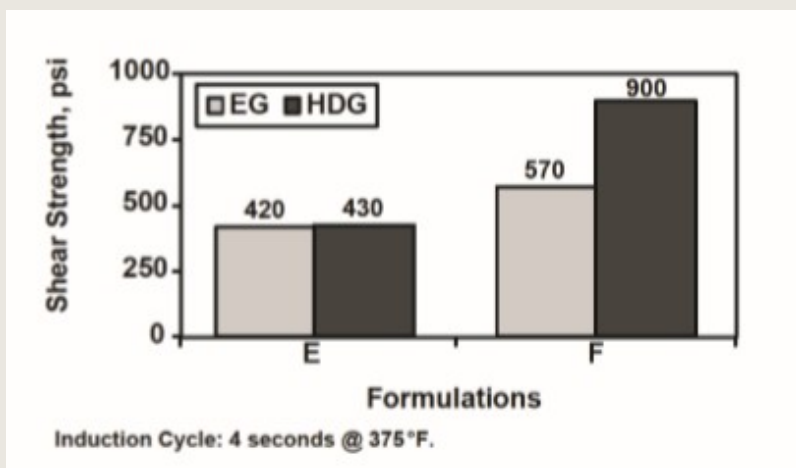


FIGURE 7: SHEAR STRENGTH AT 300 °F

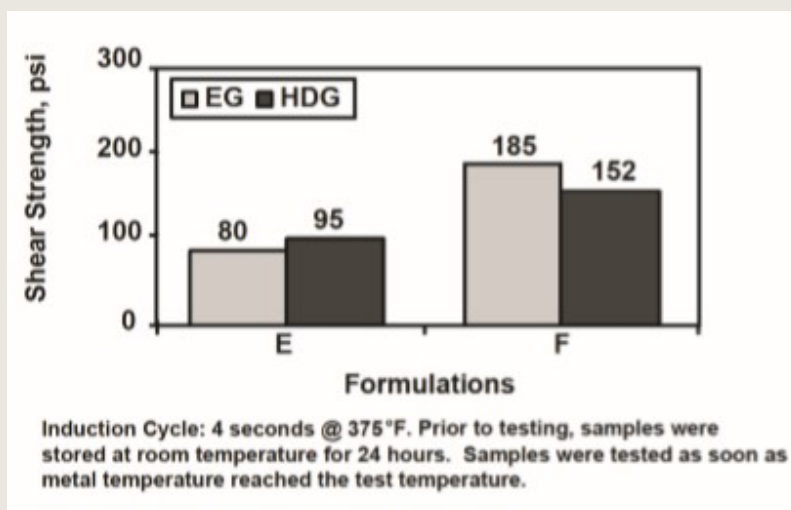


Table 6 contains shear strength and shear strength retention data for induction heat-cured bonded lap joints under different conditions after post cure. Formulation F, containing Ancamine 2441 curing agent, provides higher shear strength and shear strength retention at 300°F, as compared with the control. Also, shear strength was not affected adversely by the exposure to hot water.

SHELF STABILITY: As shown in Figure 8, the formulations containing Ancamine 2441 curing agent exhibit a small and acceptable viscosity increase after four weeks at 110°F, demonstrating that the improved reactivity of Ancamine 2441 does not have an adverse affect on the shelf stability of the formulation. Additional studies indicate that alkyl glycidyl ethers, such as Epodil® 748 (mono-functional) and Epodil 757 (di-functional) diluents, are the most effective reactive diluents for formulations containing Ancamine 2441 curing agent and do not adversely affect the shelf life of the formulation.

Comparison with competitive curing agents Ancamine 2441 curing agent has been evaluated in comparison with two other competitive curing agents, in one case as a sole curative and dicy accelerator, and in the other as a sole curative only (the competitive product is not a dicy accelerator). Table 7 contains the test formulations. As a sole curative, Ancamine 2441 curing agent has the lowest loading. Table 8 reviews the reactivity of the products tested by DSC, and Table 9 compares the shelf stability of the formulations upon storage for four weeks at 110°F.

As a dicy cure accelerator, Ancamine 2441 curing agent based formulation A offers reactivity, T_g and shelf life comparable to that of Competitive Product 1, a more expensive curing agent. Therefore, Ancamine 2441-based formulations are more cost effective without compromising performance. As a sole curative versus Competitive Product 1, the Ancamine 2441based formulation C offers improved reactivity, higher T_g, better shelf stability and lower cost. Compared with Competitive Product 2 as a sole curative (Competitive Product 2 cannot accelerate dicy), the Ancamine 2441 formulation offers significantly higher T_g, a greater degree of cure and improved reactivity, but it has a somewhat lower shelf stability. Overall formulation cost is also favorable.

In summary, Ancamine 2441 curing agent offers greater formulating versatility than the competitive products, while also offering excellent performance and handling benefits.

TABLE 6: SHEAR STRENGTH AND SHEAR STRENGTH RETENTION AT 302°F AFTER POST CURE*

Formulation	E	F
Shear Strength @ 77°F, psi	1,120	1,300
Shear Strength @ 300°F, psi	340	660
% Strength Retention	30	50
Shear Strength after 7 days Immersion in 130°F water	1,000	1,280

* 4 seconds @ 375°F induction cure followed by 30 minutes at 350°F cure.

FIGURE 8: FORMULATIONS' SHELF STABILITY % VISCOSITY INCREASE AFTER 4 WEEKS AT 110°F

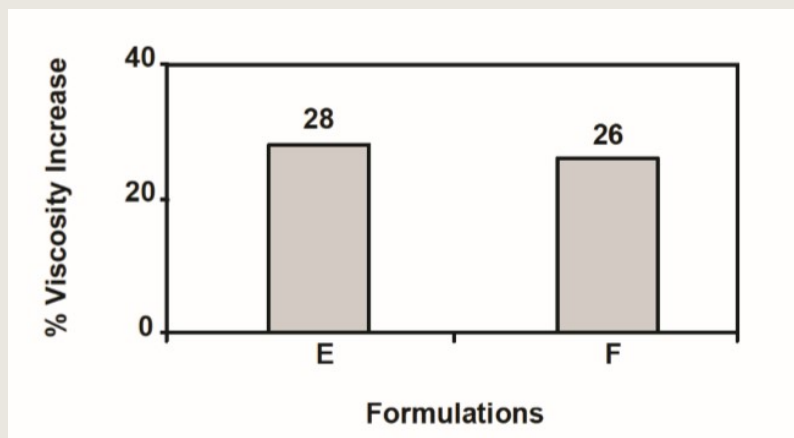


TABLE 7: COMPARISON OF ANCAMINE 2441 WITH COMPETITIVE CURING AGENTS

INGREDIENT	G	H	I	J	K
Liquid DGEBA (EEW=190)	100	100	100	100	100
Amicure CG-1200	6	6	-	-	-
Ancamine 2441	5	-	20	-	-
Competitive Product 1	-	5	-	25	-
Competitive Product 2	-	-	-	-	35
Cab-O-Sil TS-720	2	2	2	2	2

TABLE 8: FORMULATION REACTIVITY BY DSC

Formulation	G	H	I	J	K
Onset Temperature (°F)	248	246	201	212	216
Peak Temperature (°F)	293	286	275	266	241
Heat of Reaction (J/g)	385	400	360	345	295
Tg (°F)	271	279	239	228	203

ANCAMINE 2441 CURING AGENT AS A REPLACEMENT FOR SUBSTITUTED UREAS

REACTIVITY: To evaluate and compare the reactivity of Ancamine 2441 curing agent, model formulations containing two levels of Amicure UR and diuron as dicy cure accelerators were tested by DSC (Table 10). All formulations show nearly equivalent onset temperature and peak temperature; however, formulations containing Ancamine 2441 curing agent show higher heat of reaction than the formulations containing the substituted urea's, indicating a higher degree of cure. Also, the formulations containing Ancamine 2441 exhibit significantly higher glass transition temperature than the substituted urea-based formulations.

FORMULATION SHELF STABILITY: Twenty-four hours after mixing and degassing, the viscosity of the formulations was measured at room temperature. The formulations were stored at 110°F, and their viscosities were measured (after equilibrating to room temperature) every week. As Table 11 indicates, in the formulations containing Ancamine 2441 curing agent (L and M), elevated storage temperature had no affect on viscosity, suggesting that Ancamine 2441 offers excellent latency. Formulations containing Amicure UR and diuron, on the other hand, gelled after 2 and 3 weeks, respectively

TABLE 9: FORMULATION SHELF STABILITY*

Formulation	G	H	I	J	K
Initial Viscosity, cPs	90,000	90,000	126,000	135,000	170,000
After 4 Weeks	73,000	83,000	160,000	195,000	130,000
% Increase	-	-	27%	44%	-

*Storage at 108°F

TABLE 10: COMPARATIVE FORMULATIONS* AND REACTIVITY** COMPOSITION (% BY WEIGHT)

Formulation	L	M	N	O	P	Q	Methods
Liquid Epoxy Resin	100	100	100	100	100	100	Standard liquid epoxy resin with an epoxide equivalent weight of approximately 190
Amicure CG-1200	7	7	7	7	7	7	Dicyandiamide—Product of Evonik
Ancamine 2441	2	3	-	-	-	-	Modified amine—Product of Evonik
Amicure UR	-	-	2	3	-	-	Substituted urea—Product of Evonik
Diuron	-	-	-	-	2	3	3-(3,4-dichlorophenyl) 1,1-dimethylurea.
Cab-O-Sil TS-720	2	2	2	2	2	2	Fumed silica—Product of Cabot Corporation
Onset Temperature(°F)	295	282	293	288	295	291	
Peak Temperature(°F)	342	324	320	313	325	320	
Heat of Reaction (J/g)	290	310	220	240	180	210	
Tg (°F)	273	270	243	237	244	241	-

* Formulations were for comparative purposes only. For maximum performance, optimize components and use additives.

** By DSC at 50°F/min scan rate

TABLE 11: FORMULATION SHELF STABILITY VISCOSITY (PS) AT 108°F

Formulation	L	M	N	O	P	Q
Initial Viscosity	88.4	87.6	83.0	85.1	85.8	90.9
After 2 weeks	71.5	65.9	gelled	gelled	1,123	58.460
After 3 weeks	61.5	58.0	-	-	gelled	gelled
After 10 weeks	90.0	89.0	-	-	-	-



ANCAMINE 2441 CURING AGENT IN EPOXY- ANHYDRIDE SYSTEMS

Ancamine 2441 curing agent has had preliminary evaluation for use as an anhydride cure accelerator. Table 12 details the model formulations used for this comparison, all with methyl tetrahydrophthalic anhydride (MTHPA) and liquid epoxy resin (EEW=190). Alternative accelerators evaluated as comparisons were 2-methyl imidazole (2 MI), benzyldimethylamine (BDMA) and a competitive product used as an accelerator in epoxyanhydride systems. Formulation P is the recommended use level from the competitive product's manufacturer.

Table 13 summarizes the results on reactivity by DSC and glass transition temperature, and Table 14 shows the viscosity change upon storage at 77°F in a 150 gram mass. Compared with the 2MI formulation (R) and the BDMA formulation (S), the Ancamine 2441 formulation (V) has better shelf stability, it reacts more quickly, but with a higher activation temperature, and it has a lower Tg. Achieving a more comparable Tg and reactivity would require a higher loading, and the good shelf stability would be maintained. For example, Formulation X, containing 4 phr of Ancamine 2441 curing agent, has comparable reactivity, a higher Tg than Formulation S but comparable to that of Formulation R, and a significantly increased shelf stability.

In the comparison with the competitive product, evaluations were done at two different loading levels (2 phr and 4 phr). At the lower loading, the activation temperature of the competitive product formulation is lower than for the Ancamine 2441 formulation, while the glass transition temperature is also lower. At the higher loading, the competitive product formulation's onset temperature is still lower, but not by as much as in the lower loading case, and the Tg of the Ancamine 2441 formulation is significantly higher. At both loadings, the higher peak temperature of the Ancamine 2441 formulations indicates that these systems complete the cure reaction faster than the competitive systems. Both systems have good pot life, which are superior to those of either the 2MI or the BDMA formulations.

TABLE 12: COMPARISON OF ANCAMINE 2441 WITH COMPETITIVE CURING AGENTS

Formulation	R	S	T	U	V	W	X
Liquid Epoxy Resin	100	100	100	100	100	100	100
MTHPA	80	80	80	80	80	80	80
2MI	1	-	-	-	-	-	-
BDMA	-	1	-	-	-	-	-
Competitive Product	-	-	2	4	-	-	-
Ancamine 2441	-	-	-	-	1	2	4

TABLE 13: FORMULATION REACTIVITY AND GLASS TRANSITION TEMPERATURE*

Formulation	R	S	T	U	V	W	X
Onset Temp °F	255	257	277	264	318	302	282
Peak Temp °F	345	316	329	316	367	352	333
Heat of Reaction, J/g	255	220	166	217	133	215	240
Tg after 2 hr @ 302°F	275	194	194	212	167	203	266

*Reactivity by DSC

TABLE 14: FORMULATION SHELF STABILITY*

Formulation	R	S	U	X
Initial Viscosity,cPs	3,900	5,700	2,200	2,400
After 4 days	6,300	29,000	2,400	2,400
After 8 days	20,000	gel	2,400	2,400
After 60 days	gel	-	5,500	5,600

*Storage at 77°F

USE IN POWDER COATINGS: Table 15 outlines two basic powder formulations which have been evaluated. Both have been successfully cured using several cure schedules ranging from 5 to 10 minutes at 401°F to 30 minutes at 250°F. Low temperature cure capabilities have been demonstrated. Film appearances varied from smooth to grainy, and further optimization work is underway.

TABLE 15: POWDER FORMULATIONS

Ingredient (% by Weight)	Y	Z
DER 663#	100	100
Resiflow P-67	1.2	1.2
TiO ₂ R960	50	50
Amicure® CG-1200	-	5.0
Ancamine 2337S	-	-
Ancamine 2441	6.0	0.4



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