Epoxy Curing Agents and Modifiers

# 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

## 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.

### TABLE 1: TYPICAL PROPERTIES

| | |
|---|---|
| **Appearance** | White, Micronized Powder |
| **Amine Value (Mg Koh/G)** | 210-250 |
| **Particle Size (Micron)** | 90% < 10 |
| **Recommended Use Level (Phr, Eew=190)** | |
| **As A Dicy Cure Accelerator** | 2-8 |
| **As A Sole Curing Agent** | 20 |

### TABLE 2: TYPICAL FORMULATION* (COMPOSITION BY WEIGHT)

| Formulation | I | II |
|---|---|---|
| Liquid Epoxy Resin[1] | 100 | 100 |
| Amicure® CG-1200[2] | 6.0 | 0 |
| Ancamine 2441[3] | 5.0 | 20.0 |
| Fumed Silica[4] | 1.0 | 1.0 |

### TABLE 3: TYPICAL PERFORMANCE*

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

* Ancamine 2441 curing agent formulated with standard Bisphenol-A based (DGEBA, EEW=190) epoxy resin.
(1) Standard liquid epoxy resin with an epoxide equivalent weight of approximately 190.
(2) Dicyandiamide—Product of Evonik
(3) Modified Amine—Product of Evonik
(4) Fumed silica—Product of Cabot Corporation

## 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 (Tg) 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 AND RETENTION:** 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 |
|---|---|---|---|---|
| Liquid Epoxy Resin[1] | 100 | 100 | 100 | 100 |
| Amicure CG-1200[2] | 6 | 6 | — | — |
| Ancamine 2014AS[3] | 5 | — | 28 | — |
| Ancamine 2441[4] | — | 5 | — | 20 |
| Cab-O-Sil TS-720[5] | 1 | 1 | 1 | 1 |
| Onset Temperature (°F) | 271 | 255 | 228 | 210 |
| Peak Temperature (°F) | 302 | 300 | 264 | 241 |
| Heat of Reaction (J/g) | 385 | 430 | 212 | 230 |
| Tg (°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.
(2) Dicyandiamide—Product of Evonik
(3) (4) Modified amines—Products of Evonik
(5) Fumed silica—Product of Cabot Corporation

**FIGURE 1: SHEAR STRENGTH AT 77°F**

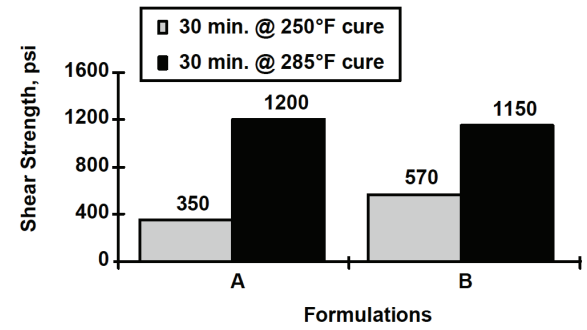
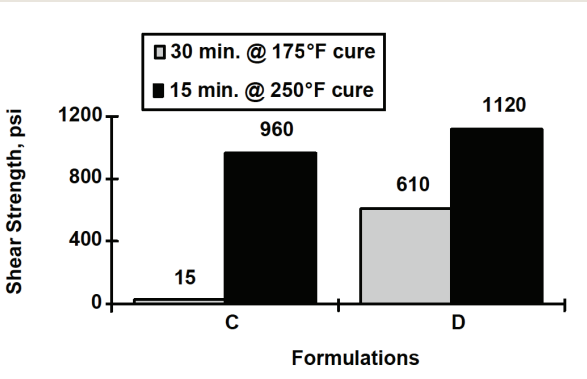



**FIGURE 2: SHEAR STRENGTH AT 77°F**



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 labscale induction heat unit at different cure schedules.

**FIGURE 3: SHEAR STRENGTH* AT 300°F**

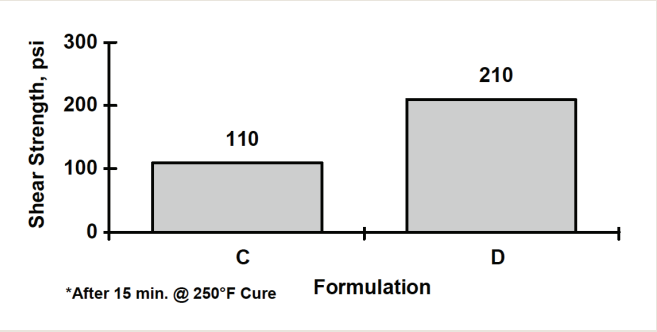



**FIGURE 4: FORMULATIONS SHELF STABILITY % VISCOSITY INCREASE AT 110°F AFTER 4 WEEKS**

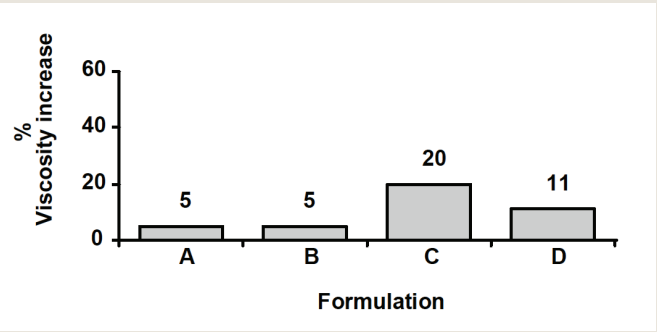



**TABLE 5: COMPARATIVE FORMULATIONS* COMPOSITION ( % BY WEIGHT )**

| Formulation Ingredient | E | F |
|---|---|---|
| Liquid epoxy resin[1] | 87.5 | 87.5 |
| Kelpoxy G292-100[2] | 25.0 | 25.0 |
| Amicure CG-1200[3] | 7.0 | 7.0 |
| Ancamine 2014AS[4] | 10.0 | — |
| Ancamine 2337S[5] | 12.5 | 12.5 |
| Ancamine 2441[6] | — | 10.0 |
| Aluminum 101[7] | 40.0 | 40.0 |
| Cab-O-Sil TS-720[8] | 2.0 | 2.0 |

* Formulations were for comparative purposes only. For maximum performance, optimize components and use additives.
(1) Standard liquid epoxy resin with an epoxide equivalent weight of approximately 190.
(2) Hycar 1300X13—Epoxy adduct from Reichhold Chemicals, Inc.
(3) Dicyandiamide—Product of Evonik
(4) (5) (6) Modified amines—Products of Evonik
(7) Filler—Product of Alcan Toyo of America.
(8) Fumed silica—Product of Cabot Corporation

**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**

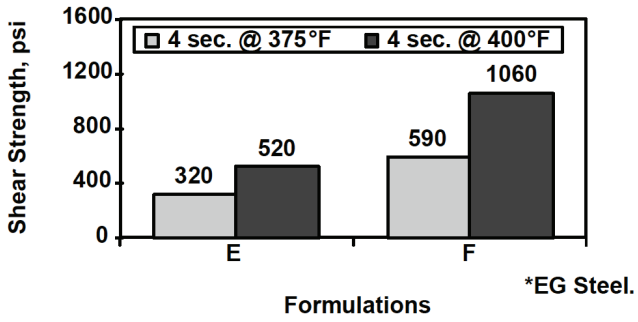



**FIGURE 6: GREEN STRENGTH* AT 77°F WITHIN 24 HOURS**

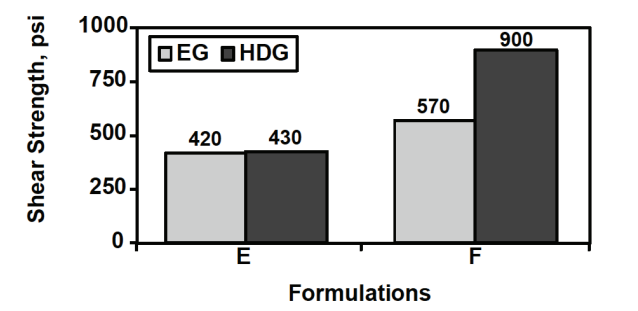



**FIGURE 7: SHEAR STRENGTH AT 300 °F**

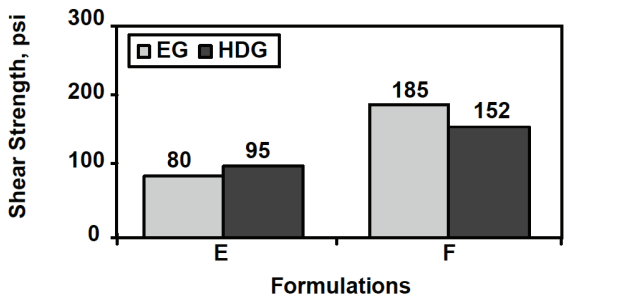


Table 6 contains shear strength and shear strength retention data for induction heat-cured bonded lap joints under different conditions after post cure. Formulation F, containing Ancamine 2441 curing agent, provides higher shear strength and shear strength retention at 300°F, as compared with the control. 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, Tg 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 2441-based formulation C offers improved reactivity, higher Tg, 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 Tg, 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**

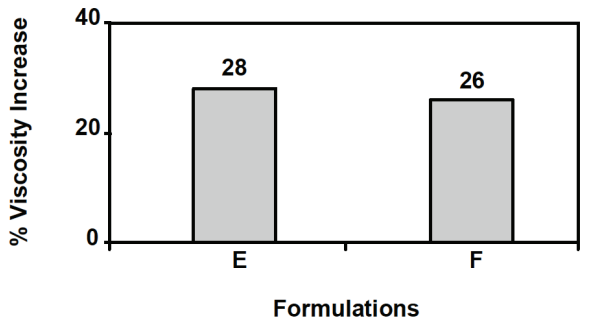



**TABLE 7: COMPARISON OF ANCAMINE 2441 WITH COMPETITIVE CURING AGENTS**

| Formulation | G | H | I | J | K |
|---|---|---|---|---|---|
| Liquid Epoxy Resin | 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 |
|---|---|---|---|---|---|---|
| Liquid Epoxy Resin[1] | 100 | 100 | 100 | 100 | 100 | 100 |
| Amicure CG-1200[2] | 7 | 7 | 7 | 7 | 7 | 7 |
| Ancamine 2441[3] | 2 | 3 | — | — | — | — |
| Amicure UR[4] | — | — | 2 | 3 | — | — |
| Diuron[5] | — | — | — | — | 2 | 3 |
| Cab-O-Sil TS-720[6] | 2 | 2 | 2 | 2 | 2 | 2 |
| 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
(1) Standard liquid epoxy resin with an epoxide equivalent weight of approximately 190
(2) Dicyandiamide—Product of Evonik
(3) Modified amine—Product of Evonik
(4) Substituted urea—Product of Evonik
(5) 3-(3,4-dichlorophenyl)-1,1-dimethylurea.
(6) Fumed silica—Product of Cabot Corporation

### 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

# ANCAMINE® 2441 Curing Agent

### 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

### TABLE 15: POWDER FORMULATIONS

| Ingredient (% by Weight) | Y | Z |
|---|---|---|
| DER 663µ | 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 |

**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.

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