

ANCAMINE® 2422 Curing Agent

DESCRIPTION

Ancamine 2422 curing agent is a highfunctionality aliphatic amine designed for use in two-package epoxy formulations. It is especially suitable for use with multifunctional epoxy novolac resins. Ancamine 2422 curing agent yields formulations with excellent chemical resistance. It also imparts good working time when compared with alternative high-functionality curing agents. Ancamine 2422 systems are suitable for cure at room temperature or elevated temperatures. These properties make Ancamine 2422 curing agent ideal for formulating chemically-resistant coatings, linings and mortars. It can be also be used as a high functionality accelerator.

ADVANTAGES

- Excellent chemical resistance
- Good pot life
- Suitable for ambient cure or post cure in formulated systems

APPLICATIONS

- Chemically resistant coatings
- Tank linings
- Secondary containment linings
- Mortars
- High functionality accelerator

SHELF LIFE

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

STORAGE AND HANDLING

Refer to the Safety Data Sheet for Ancamine 2422 curing agent.

TABLE 1: TYPICAL PROPERTIES

Appearance	Yellow Liquid
Color¹ (Gardner)	3
Viscosity² @ 77°F (25°C) (cP)	2,000
Amine Value³ (mg KOH/g)	665
Specific Gravity⁴ @ 77°F (25°C)	1.12
Flash Point⁵ (closed cup) (°F)	>212
Equivalent Wt/{H}	49
Recommended Use Level (phr, EEW=190)	

TABLE 2: TYPICAL HANDLING PROPERTIES*

See attached information (Table 1 and Table 2)

TABLE 3: TYPICAL PERFORMANCE

See attached information

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Footnotes:

¹ ASTM D1544-80

² ASTM D445-83, Brookfield, RVTD, Spindle 4

³ Perchloric Acid Titration

⁴ ASTM D1475-85

⁵ Seta Flash Closed Cup

SUPPLEMENTARY DATA

FORMULATING WITH ANCAMINE 2422 CURING AGENT FOR CHEMICAL RESISTANCE

Epoxy novolacs are known for their high chemical resistance relative to other polymer types. However, some in-service performance demands exceed what even the best standard epoxy formulations can deliver. For example, very aggressive solvents, such as methylene chloride, swell rapidly and fracture the polymer network, while high concentrations of strong inorganic acids, such as nitric acid, attack and destroy the protective barrier chemically. Epoxy novolacs can be made more resistant to these aggressive reagents by increasing the crosslink sites per unit volume in the polymer network (crosslink density) or, more simply put, by increasing the functionality of the formulation on both the resin and curing agent sides.

The resins used in this approach are typically epoxy novolacs with average functionality (reactive sites per molecule) of 2.2f to 3.6f. Higher functionality is available with solid epoxy novolac resins, but these resins are difficult to formulate for high-solids, ambient-cured systems.

When used with these resins, conventional curing agents with high functionality give formulations with very short working time, excessive exotherms and very low conversion or through cure at gel. Ancamine 2422 curing agent is a high-functionality (~ 6f) curing agent designed to overcome these drawbacks. When mixed with epoxy novolac resin, it offers good working time and good conversion at room temperature. In addition, the structure of this amine contributes significantly to the formulation's chemical resistance. For ambient temperature cure with epoxy novolac resins, Ancamine 2422 formulations must contain a plasticizer to get adequate through-cure.

SELECTION AND USE OF MULTIFUNCTIONAL RESIN

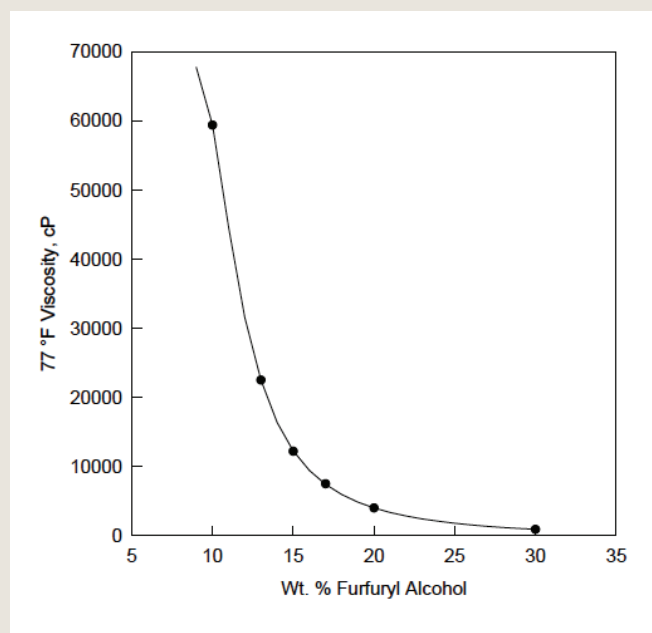
While epoxy novolacs offer the multifunctionality needed for high chemical resistance, their use is limited by their very high viscosity. The 3.6f epoxy novolac is a semisolid at room temperature with a melt viscosity above 20,000 cP at 125°F (52°C). Ancamine 2422 curing agent's low viscosity aids in lowering system viscosity, but a diluent/plasticizer may be required. Diluents lower the viscosity of these resins effectively but should be chosen carefully to avoid reducing crosslink density and subsequent chemical resistance. For this reason, monofunctional reactive diluents should be avoided and multifunctional reactive diluents with aliphatic backbones should be used sparingly.

Nonreactive diluents in multifunctional formulations are effective as both viscosity reducers and plasticizers, promoting

greater through-cure for room temperature conversion. They do not contribute to crosslinking, however, so their volume in the polymer network should be minimized. Too high a level of diluent will ultimately degrade the chemical resistance of the system.

Figure 1 shows the viscosity reduction curve for a 3.6f epoxy novolac resin blended with furfuryl alcohol as the diluent. At 15 weight percent diluent, the resin becomes manageable with a viscosity of 12,500 cP. Combinations of 3.6f and 2.2f resins will give even lower viscosities, but diluent will still be required for through-cure at room temperature.

FIGURE 1: VISCOSITY REDUCTION OF 3.6F EPOXY NOVOLAC WITH FURFURYL ALCOHOL



The low viscosity of Ancamine 2422 curing agent and the use of diluent with the epoxy novolac resin eliminates the need for additional diluent/plasticizer.

COMBINING ANCAMINE 2422 CURING AGENT WITH MULTIFUNCTIONAL RESINS

Two formulations (shown in Table 4) were prepared using Ancamine 2422 curing agent with epoxy novolac resins. Furfuryl alcohol was chosen as the diluent and mixed with the resins prior to adding Ancamine 2422 curing agent.

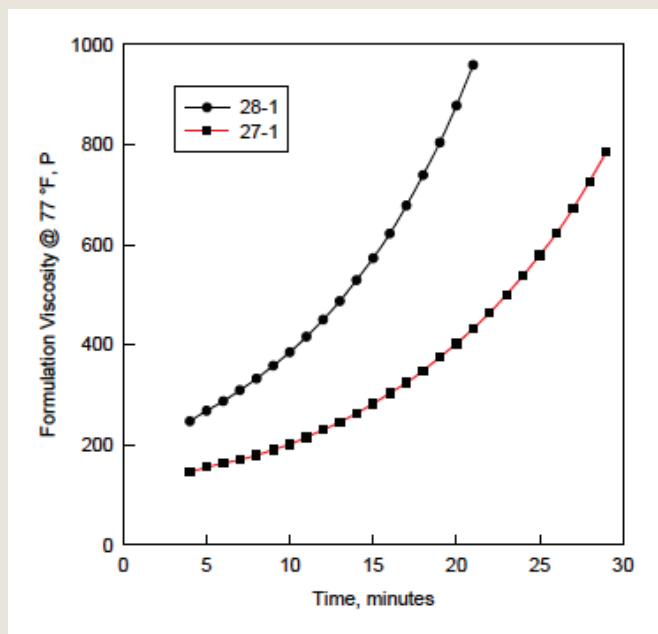
Reactivity was measured by gel time, thin film set time and viscosity increase to determine working time. Table 4 shows the high reactivity of these formulations, with room temperature gel times of 15 to 16 minutes. The results of viscosity build over time in Figure 2 show reasonable working times considering the high functionality of these formulations.

TABLE 4: ANCAMINE 2422 AMBIENT REACTIVITY AND CURE WITH MULTIFUNCTIONAL RESINS

FORMULATION	27-1	28-1
RESIN SIDE (Parts by Weight)		
Epoxy Novolac, 3.6f*	51.0	85.0
Epoxy Novolac, 2.2f**	34.0	-
Furfuryl alcohol	15.0	15.0
HARDENER SIDE (Parts by Weight)		
Ancamine 2422	23.4	23.3
Initial Reactivity		
Gel Time, min, 150g mass	16.1	15.2
Peak Exotherm Temp, °F	399	405
Thin Film Set Time, h		
@ 77 °F (25°C)	1.3	1.0
@ 40 °F (4°C)	6.0	5.3
DSC Reactivity, Initial		
Peak Exotherm @ °F	208	207
Δ H, J/g	401	415
7 Day Cure @ 77°F (25°C)		
Residual Exotherm, J/g	37	74
Tg, °F	131	133

* 3.6f resin: Dow Chemical, D.E.N.® 438 or Shell EPON HPT® Resin 155
 **2.2f to 2.5f resin: Dow Chemical, D.E.N. 431 or Shell EPON® Resin 160

FIGURE 2: VISCOSITY VS. TIME FOR ANCAMINE 2422/EPOXY NOVOLAC FORMULATIONS



Note: Formulation viscosity versus time was monitored for the two formulations using a Rheometrics mechanical spectrometer with parallel plate geometry.

Estimates of the extent of cure can be determined by using a differential scanning calorimeter (DSC). A small residual exotherm is seen for both formulations in Table 4, indicating that, although well-cured at room temperature, some additional crosslinking can take place.

POST CURE: Mild post-cures at slightly elevated temperatures were examined to determine the effect on crosslinking. Both formulations were allowed to gel at room temperature for three days followed by a two-hour post-cure at either 135 °F (57°C) or 250°F (121°C). Results of the analysis are given in Table 5. Analysis of the formulations exposed to the 135°F post-cure show lower residual exotherms and higher Tgs than formulations cured at ambient temperature; both indications of a more tightly crosslinked polymer network. Analysis of the formulations exposed to the 250°F post-cure show a similar result with no residual exotherm and even higher crosslink density. Optimal diluent level will be a balance between handling capabilities and chemical resistance requirements in the finished formulation.

TABLE 5: ANCAMINE 2422 POST CURE WITH MULTIFUNCTIONAL RESINS

FORMULATION	27-1	28-1
DSC Reactivity, Initial		
Peak Exotherm @ °F	208	207
Δ H, J/g	401	415
Post Cure, 2 hr @ 135°F (57°C)		
Residual Exotherm, J/g	33	46
Tg, °F	156	160
Post Cure, 2 hr @ 250°F (121°C)		
Residual Exotherm, J/g	0	0
Tg, °F	207	228

CHEMICAL RESISTANCE: Chemical resistance tests following ASTM D 543 were performed on 1/8-inch castings using each of the three cure schedules:

- 1) 7 days at 77°F (25°C),
- 2) 3 days at 77°F (25°C) plus 2 hours at 135°F (57°C), and
- 3) 3 days at 77°F (25°C) plus 2 hours at 250°F (121°C).

1" X 3" coupons from these castings were immersed in four very aggressive reagents: methylene chloride, 30% nitric acid, 10% acetic acid and 10% phenol. Weight gain was monitored over time to determine chemical resistance (see Table 6). Figures 3 through 6 show the impact of the cure schedule on chemical resistance for the two most aggressive reagents, methylene chloride and nitric acid.

TABLE 6: ANCAMINE 2422 CHEMICAL RESISTANCE WITH MULTIFUNCTIONAL RESINS % WEIGHT GAIN

Formulation	27-1			28-1		
	7-day @ 77°F (25°C)	Gel + 2 hr @ 135°F (57°C)	Gel + 2 hr @ 250°F (121°C)	7-day @ 77°F (25°C)	Gel + 2 hr @ 135°F (57°C)	G Gel + 2 hr @ 250°F (121°C)
Chemical Resistance						
Methylene Chloride						
1 Day	16.2	6.97	0.95	14.31	6.38	0.97
3 Day	D*	15.17	6.91	31.87	15.68	3.38
7 Day	D	33.41	14.45	31.64	30.31	7.01
14 Day	D	30.30	25.50	31.98	29.81	12.16
21 Day	D	29.57	28.41	29.30	28.06	15.74
28 Day	D	28.97	27.57	26.57	26.01	19.78
10% Acetic Acid						
1 Day	0.89	0.32	0.22	1.14	0.42	0.18
3 Day	1.67	0.67	0.43	2.00	0.83	0.36
7 Day	2.33	1.02	0.65	2.91	1.34	0.60
14 Day	3.31	1.61	1.03	3.84	1.87	0.88
21 Day	3.88	1.99	1.29	4.72	2.33	1.11
28 Day	4.40	2.36	1.53	4.93	2.83	1.35
30% Nitric Acid						
1 Day	0.49	0.46	0.26	0.45	0.45	0.33
3 Day	0.77	0.69	0.65	0.71	0.70	0.61
7 Day	1.17	1.02	0.95	1.12	1.14	0.96
14 Day	1.76	1.48	1.36	1.64	1.55	1.34
21 Day	2.28	1.91	1.73	2.22	1.90	1.63
28 Day	2.75	2.31	2.10	2.40	2.45	2.11
10% Phenol						
1 Day	0.58	0.25	0.23	0.50	0.30	0.16
3 Day	1.08	0.50	0.38	0.87	0.58	0.31
7 Day	1.76	0.82	0.63	1.41	1.01	0.53
14 Day	2.62	1.32	0.96	2.14	1.59	0.82
21 Day	3.29	1.70	1.22	2.88	2.23	1.14
28 Day	4.01	2.14	1.50	3.16	2.48	1.24

* D indicates sample destroyed by the test.

Figure 3 shows methylene chloride resistance for formulation 27-1. After 3 days of exposure, the room temperature cured coupons are destroyed. The 135°F (57°C) post-cured coupons show evidence of cracking at 7 days, but remain intact for the 28-day test. The 250°F (121°C) post-cured coupons show no evidence of cracking for the duration of the test, even though there is significant weight gain.

FIGURE 3: METHYLENE CHLORIDE RESISTANCE OF ANCAMINE 2422/EPOXY NOVOLAC FORMULATION 27-1

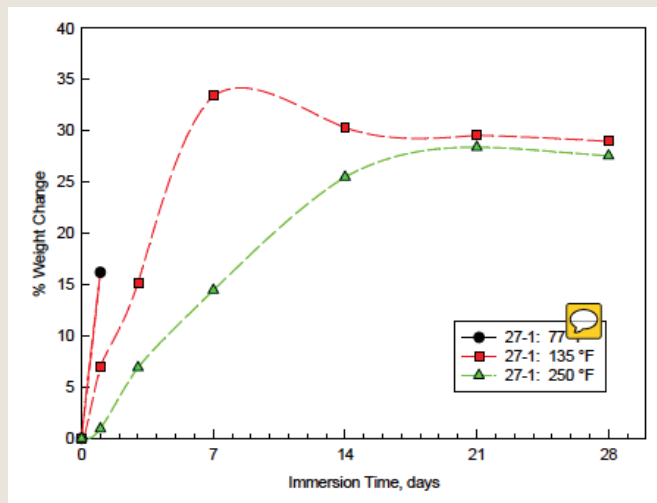
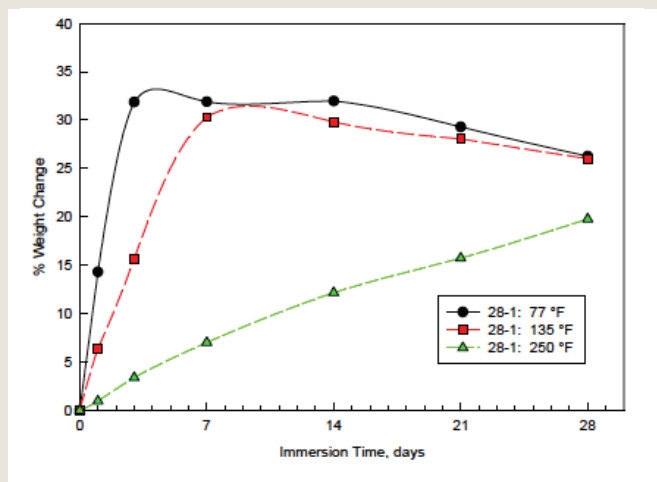


Figure 4 shows methylene chloride resistance for the more tightly crosslinked formulation, 28-1. The room temperature cured coupons show only a very slight fracture at the end of the 28-day test. The 135°F (57°C) post-cured coupons and the 250°F (121°C) coupons show no evidence of cracking and remain intact for the entire test time. This formulation shows excellent resistance to methylene chloride and exceptional resilience during the solvent swell.

FIGURE 4: METHYLENE CHLORIDE RESISTANCE OF ANCAMINE 2422/EPOXY NOVOLAC FORMULATION 28-1



Figures 5 and 6 show 30% nitric acid resistance for formulations 27-1 and 28-1 respectively. Post-cures enhance the resistance to attack within each formulation, but the tighter network of formulation 28-1 shows only a slight improvement over formulation 27-1. Both formulations show outstanding resistance to this high concentration of nitric acid. Similar results are seen with exposure to 10% acetic acid and 10% phenol.

FIGURE 5: 30% NITRIC ACID RESISTANCE OF ANCAMINE 2422/EPOXY NOVOLAC FORMULATION 27-1

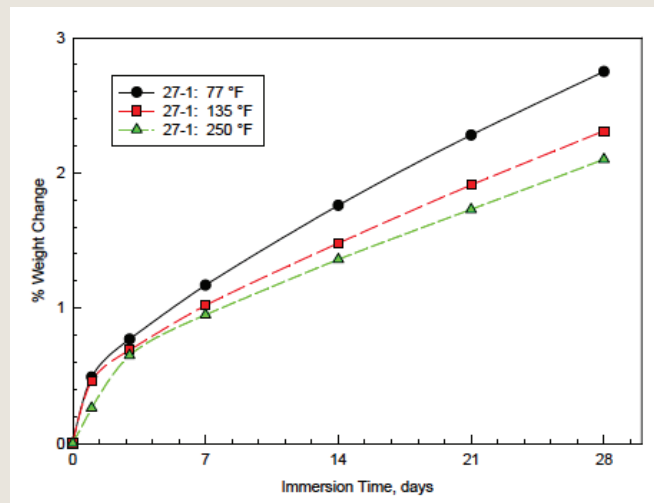


FIGURE 6: 30% NITRIC ACID RESISTANCE OF ANCAMINE 2422/EPOXY NOVOLAC FORMULATION 28-1

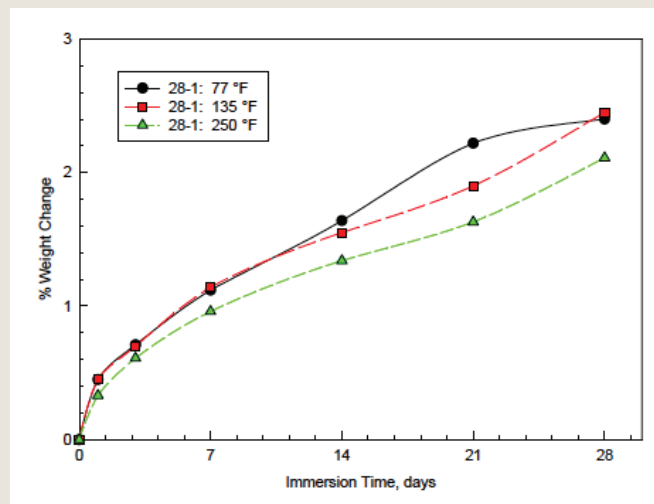


Table 7 shows additional chemical resistance for formulation 28-1 cured under ambient conditions. The results further highlight the outstanding resistance offered to a wide range of aggressive reagents.

TABLE 7: ANCAMINE 2422 CHEMICAL RESISTANCE WITH MULTIFUNCTIONAL RESINS % WEIGHT GAIN

Formulation	28-1		
Cure	7-day @77 °F (25°C)	Cure	7-day @77 °F (25°C)
Chemical Resistance			
Glacial Acetic Acid		98% Sulfuric Acid	
1 Day	2.39	1 Day	0.23
3 Day	4.08	3 Day	0.47
7 Day	6.40	7 Day	0.77
14 Day	9.59	14 Day	1.17
21 Day	11.96	21 Day	1.39
28 Day	13.36	28 Day	1.57
Methanol		10% Lactic Acid	
1 Day	1.90	1 Day	0.59
3 Day	3.14	3 Day	1.11
7 Day	4.74	7 Day	1.83
14 Day	6.78	14 Day	2.63
21 Day	8.15	21 Day	3.18
28 Day	8.02	28 Day	3.51
Toluene		Butyl Cellosolve	
1 Day	0.05	1 Day	-0.08
3 Day	0.10	3 Day	-0.03
7 Day	0.16	7 Day	-0.10
14 Day	0.26	14 Day	-0.12
21 Day	0.37	21 Day	-0.13
28 Day	0.46	28 Day	-0.10
MEK			
1 Day	-0.03		
3 Day	0.04		
7 Day	0.28		
14 Day	0.80		
21 Day	1.29		
28 Day	1.65		

Epoxy Curing Agents and Modifiers

ANCAMINE® 2422 Curing Agent

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