

ANCAMINE® 2422 Curing Agent

DESCRIPTION

Ancamine 2422 curing agent is a high functionality 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.

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

SHELF LIFE

At least 12 months from the date of manufacture in the original sealed container at ambient temperature.

PACKAGING AND HANDLING

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

TYPICAL PROPERTIES

Appearance	Yellow liquid
Colour¹ (Gardner)	≤5
Viscosity² @ 25°C, [mPa.s]	1,500-2,500
Amine Value³ (mg KOH/g)	660-675
Specific Gravity @ 21°C	1.12
Equivalent Wt/{H}	49
Recommended use Level⁴ [PHR]	26

TYPICAL CURE SCHEDULE

7 Days at Room Temperature of heat cure

Footnotes:

- (1) ASTM D 1544-80
- (2) Brookfield RVTD, Spindle 4
- (3) Perchloric Acid Titration
- (4) With Bisphenol A diglycidyl ether (EEW=190)

SUPPLEMENTARY DATA

FORMULATING WITH ANCAMINE 2422 CURING AGENT FOR CHEMICAL RESISTANCE

Epoxy resins 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, rapidly swell and fracture the polymer network, while high concentrations of strong inorganic acids, such as nitric acid, chemically attack and destroy the protective barrier. Epoxy resins 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 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 plasticiser to get adequate through-cure.

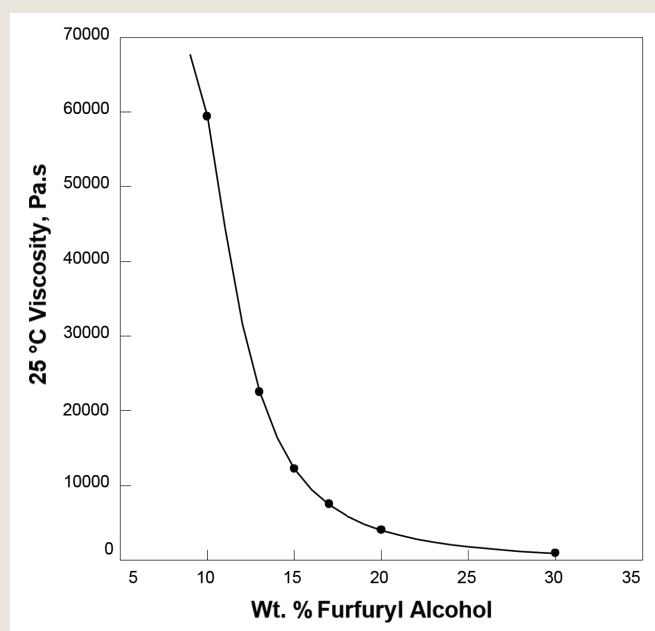
SELECTION AND USE OF MULTIFUNCTIONAL RESIN

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 semi-solid at room temperature with a melt viscosity above 20,000 mPa.s at 57°C. Ancamine 2422's low viscosity aids in lowering system viscosity, but a diluent/plasticizer may be required. Diluents effectively lower the viscosity of these resins 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.

Non-reactive 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 on page 3 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 mPa.s. 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 and the use of diluent with the epoxy novolac resin eliminates the need for additional diluent/plasticizer.

COMBINING ANCAMINE 2422 WITH MULTIFUNCTIONAL RESINS

Two formulations (shown in Table 1) 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.

Reactivity was measured by gel time, thin film set time and viscosity increase to determine working time. Table I 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.

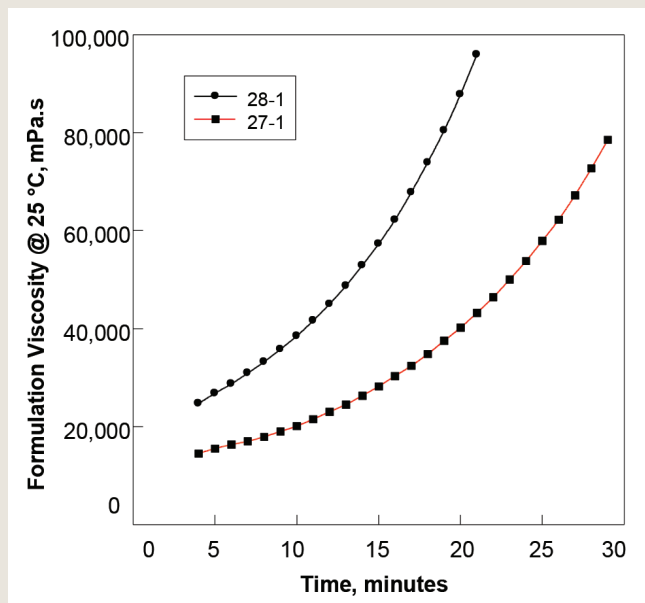
TABLE 1: ANCAMINE 2422 AMBIENT REACTIVITY AND CURE WITH MULTIFUNCTIONAL RESINS

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
Handling Properties Initial Reactivity		
Gel Time, min, 150g mass	16.1	15.2
Peak Exotherm Temp, °C	204	207
Thin Film Set Time, h		
@ 25°C	1.3	1.0
@ 5°C	6.0	5.3
DCS Reactivity, Initial		
Peak Exotherm @ °C	98	97
Δ H, J/g	401	415
7 Day Cure @ 25°C		
Residual Exotherm, J/g	37	74
Tg, °C	55	56

* 3.6f resin: Dow Chemical, D.E.N.® 438

**2.2f resin: Dow Chemical, D.E.N. 431

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



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 1 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 57°C or 121°C. Results of the analysis are given in Table 2. Analysis of the formulations exposed to the 57°C post cure show lower residual exotherms and higher Tg's than formulations cured at ambient temperature, both indicating a more tightly crosslinked polymer network. Analysis of the formulations exposed to the 121°C 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 2: ANCAMINE 2422 POST CURE WITH MULTIFUNCTIONAL RESINS

Formulation	27-1	28-1
DCS Reactivity, Initial		
Peak Exotherm @ °C	98	97
Δ H, J/g	401	415
Post Cure, 2 h @ 57°C		
Residual Exotherm, J/g	33	46
Tg, °C	69	71
Post Cure, 2 h @ 121°C		
Residual Exotherm, J/g	0	0
Tg, °C	97	109

CHEMICAL RESISTANCE

Chemical resistance tests following ASTM D 543 were performed on 3mm castings using each of the three cure schedules:

- 1) 7 days at 25°C
- 2) 3 days at 25°C plus 2 hours at 57°C
- 3) 3 days at 25°C plus 2 hours at 121°C

25mm X 75mm 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 (Table 3).

TABLE 3: ANCAMINE 2422 CHEMICAL RESISTANCE WITH MULTIFUNCTIONAL RESINS % WEIGHT GAIN

Formulation	27-1			28-1		
	Cure	7 day @ 25°C	Gel+2h @ 57°C	Gel+2h @ 121°C	7 day @ 25°C	Gel+2h @ 57°C
Chemical Resistance						
Methylene Chloride						
1 Day	16.2	6.97	0.95	14.31	6.38	0.97
3 Days	D*	15.17	6.91	31.87	15.68	3.38
7 Days	D	33.41	14.45	31.64	30.31	7.01
14 Days	D	30.30	25.50	31.98	29.81	12.16
21 Days	D	29.57	28.41	29.30	28.06	15.74
28 Days	D	28.97	27.57	26.27	26.01	19.78
10% Acetic Acid						
1 Day	0.89	0.32	0.22	1.14	0.42	0.18
3 Days	1.67	0.67	0.43	2.00	0.83	0.36
7 Days	2.33	1.02	0.65	2.91	1.34	0.60
14 Days	3.31	1.61	1.03	3.84	1.87	0.88
21 Days	3.88	1.99	1.29	4.72	2.33	1.11
28 Days	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 Days	0.77	0.69	0.65	0.71	0.70	0.61
7 Days	1.17	1.02	0.95	1.12	1.14	0.96
14 Days	1.76	1.48	1.36	1.64	1.55	1.34
21 Days	2.28	1.91	1.73	2.22	1.90	1.63
28 Days	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 Days	1.08	0.50	0.38	0.87	0.58	0.31
7 Days	1.76	0.82	0.63	1.41	1.01	0.53
14 Days	2.62	1.32	0.96	2.14	1.59	0.82
21 Days	3.29	1.70	1.22	2.88	2.23	1.14
28 Days	4.01	2.14	1.50	3.16	2.48	1.24

* D indicates sample destroyed by the test.

Figures 3 - 6 show the impact of cure schedule on chemical resistance for the two most aggressive reagents, methylene chloride and nitric acid. Figure 3 shows methylene chloride resistance for formulation 27-1. At 3 days exposure the room temperature cured coupons are destroyed. The 57°C post cured coupons show evidence of cracking at 7 days but remain intact for the 28 day test. The 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

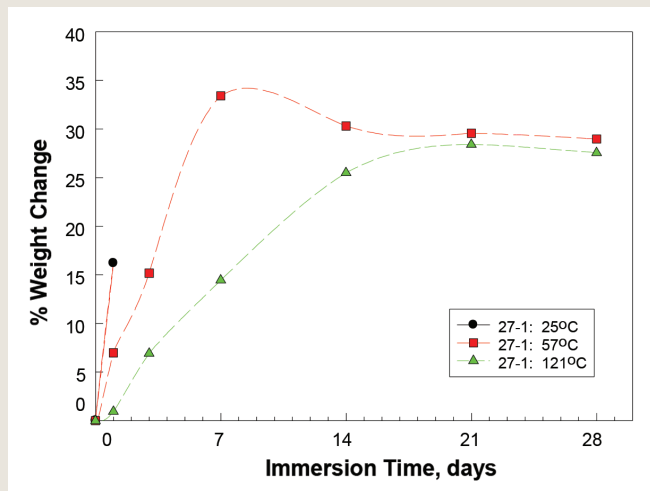


FIGURE 4: METHYLENE CHLORIDE RESISTANCE OF ANCAMINE 2422/EPOXY NOVOLAC FORMULATION 28-1

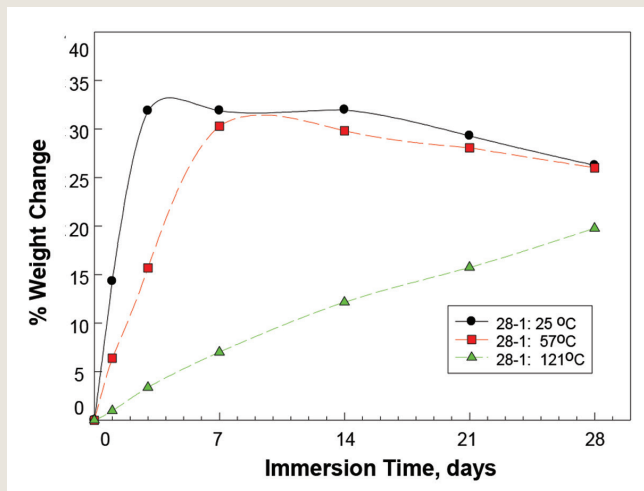


Figure 4 shows methylene chloride resistance for the more tightly crosslinked formulation, 28-1. The room temperature cured coupons show only a very slight fracture at the end of the 28 day test. The 57 °C post cured coupons and the 121°C coupons show no evidence of cracking and remain intact for the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

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: NITRIC ACID OF ANCAMINE 2422/EPOXY NOVOLAC FORMULATION 27-1

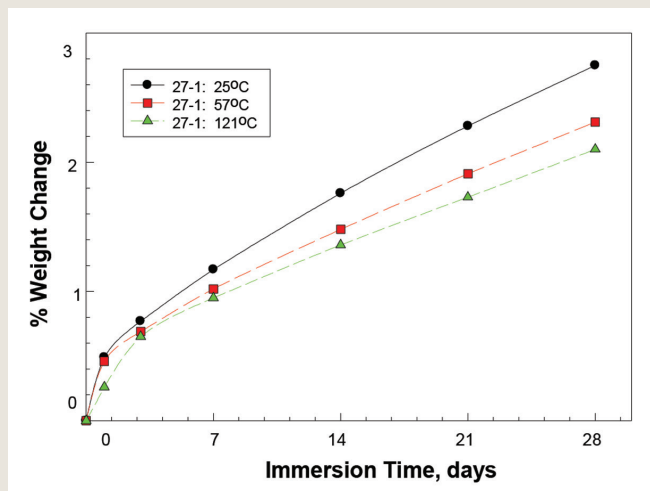


FIGURE 6: NITRIC ACID OF ANCAMINE 2422/EPOXY NOVOLAC FORMULATION 28-1

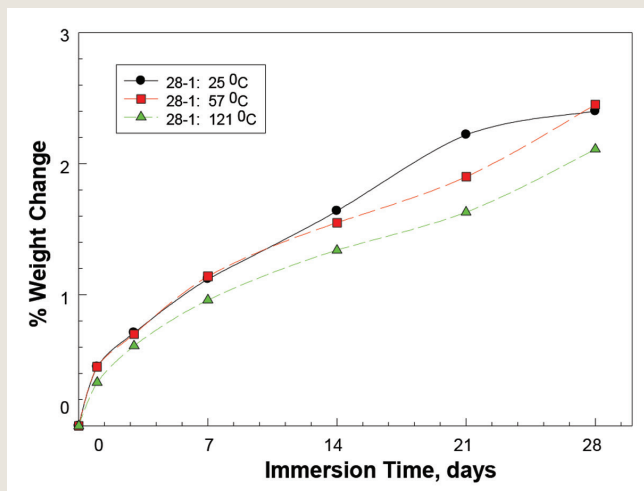


Table 4 shows additional chemical resistance for formulation 28-1 cured under ambient conditions. The results further highlight the outstanding resistance offered to a wide range of aggressive reagents.

TABLE 4: ANCAMINE 2422 CHEMICAL RESISTANCE WITH MULTIFUNCTIONAL RESINS % WEIGHT GAIN

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

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