

ChemBrief



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The Effect of Epoxy Hardeners and Accelerators in a Two Part Epoxy Primer Containing Garamite® 1958 Additive

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Introduction: The use of a tertiary amine accelerator, Ancamine™* K54, in epoxy systems containing Garamite® 1958 additive has been reported to cause a loss of sag resistance when allowed to sit for 30 minutes before measurement. Thus we have evaluated various epoxy hardeners and accelerators in a two part, high solids, high build epoxy primer to better understand which epoxy chemistries are beneficial with the use of Garamite® 1958. It is believed that accelerators with high amine values, such as Ancamine K54, Ancamine 1638, and Ancamine 1768, can exchange with the quaternary amine surface modifiers on Garamite®, causing the loss in sag resistance; these should therefore be avoided. See Table III for results. The combination of Ancamine 2432 with Ancamine 1767 (both modified aliphatic amines) gave the highest level of sag resistance, 20 – 25 mils, in this study.

Experimental: The high solids, high build primer formula used for this work is given in Table I.

Table I: High Solids High Build Formula

A Side	grams
Epon 828	100.0
Ti O2	19.1
Sparmite	40.5
G-1958	3.7
Xylene	42.0
1-butanol	11.5
Total wt.	216.8
Solvent wt.	53.5
% Solids	75.3%

Procedure: Add Garamite® to resin and mix on dispermat at 5000 rpm for 5 minutes. Add TiO2 and mix at 5000 rpm for 2 minutes. Add sparmite and mix at 5000 rpm for 2 minutes. Add xylene and butanol and mix at 2000 rpm for 2 minutes; cool to 25°C. Mix A side and B side (hardener) at proper ratio (see Table II) and run sag.

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Allow the two part mix to sit for 30 minutes then measure sag resistance again. Repeat procedure but add 8% accelerator to the hardener; a description of the hardeners and accelerators is given in the Appendix.

* Ancamine is a registered trademark of Air Products and Chemicals, Inc.

Table II: A:B Ratios

Hardener	phr	A:B Ratio	grams
Ancamine 2280	58	3.7:1	18.5:5
Ancamine 2368	52	4.2:1	21.0:5
Ancamine 2432	46	4.7:1	23.5:5
Ancamine 350A	55	3.9:1	19.5:5
Ancamine 1767	100	2.2:1	22.0:10

Results: Each hardener was evaluated alone and with all accelerators. A repeat sample was also run for reproducibility. Sag resistance was measured at initial and after 30 minutes. This data is listed in Table III.

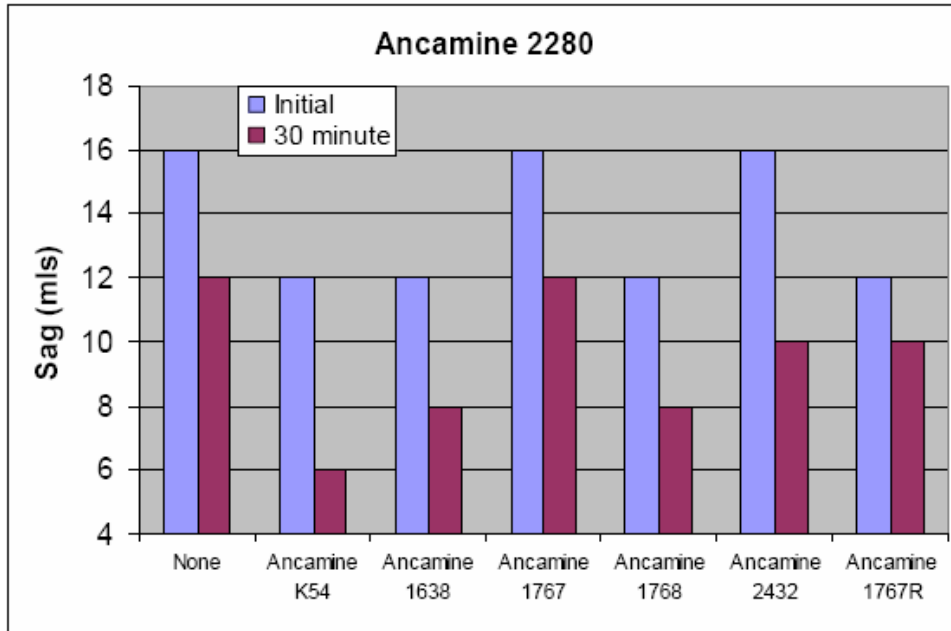
Table III: Initial and 30 Minute Sag Results

Hardener	Accelerator	Sag (mils)	
		Initial	30 minute
Ancamine 2280	None	16	12
	Ancamine K54	12	6
	Ancamine 1638	12	8
	Ancamine 1767	16	12
	Ancamine 1768	12	8
	Ancamine 2432	16	10
	Ancamine 1767 Repeat	12	10
	None	8	6
Ancamine 2368	None	8	6
	Ancamine K54	6	6
	Ancamine 1638	6	6
	Ancamine 1767	6	6
	Ancamine 1768	6	6
	Ancamine 2432	8	6
	Ancamine 2432 Repeat	6	6
	None	25	18
Ancamine 2432	None	25	18
	Ancamine K54	20	10
	Ancamine 1638	16	14
	Ancamine 1767	25	20
	Ancamine 1768	16	12
	Ancamine 2432	N/A	N/A
	Ancamine 1767 Repeat	25	22
	None	14	12
Ancamide 350A	None	14	12
	Ancamine K54	12	10
	Ancamine 1638	12	10
	Ancamine 1767	12	12
	Ancamine 1768	12	12
	Ancamine 2432	12	12
	Ancamine 1767 Repeat	12	12
	None	10	Set up
Ancamine 1767	None	10	Set up
	Ancamine K54	8	Set up
	Ancamine 1638	8	Set up
	Ancamine 1767	N/A	N/A
	Ancamine 1768	8	Set up
	Ancamine 2432	8	Set up
	None	8	Set up



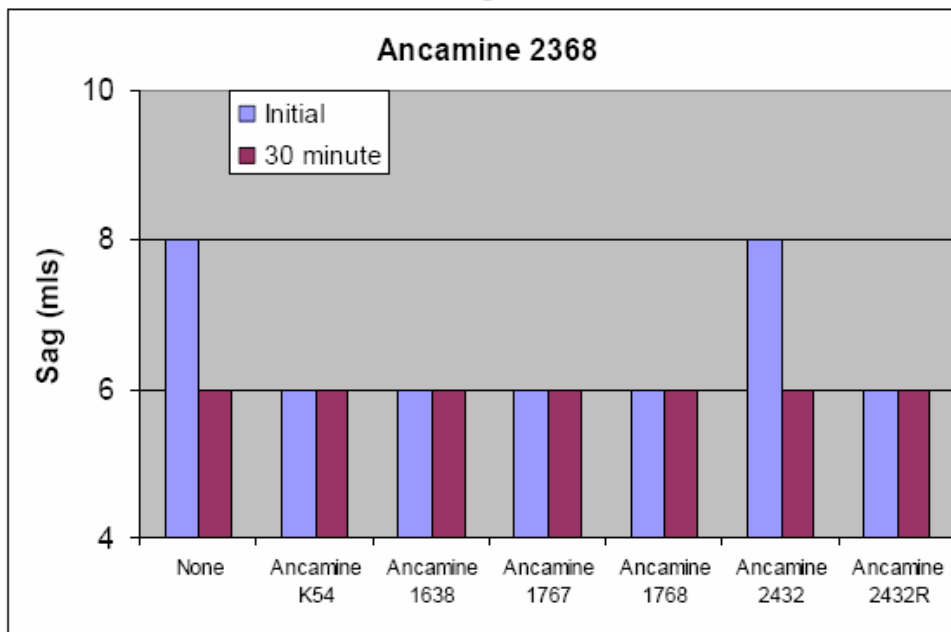
The samples prepared with Ancamine 1767 set up after 30 minutes, therefore a 30 minute sag measurement could not be obtained. The data listed in Table III is illustrated in the following graphs.

Graph 1



Graph 1 indicates that when using Ancamine 2280, a modified cycloaliphatic amine, with a viscosity of 450 cps and an amine value of 250 mg KOH/g, Ancamine 1767 and Ancamine 2432 accelerators give the best sag resistance after 30 minutes.

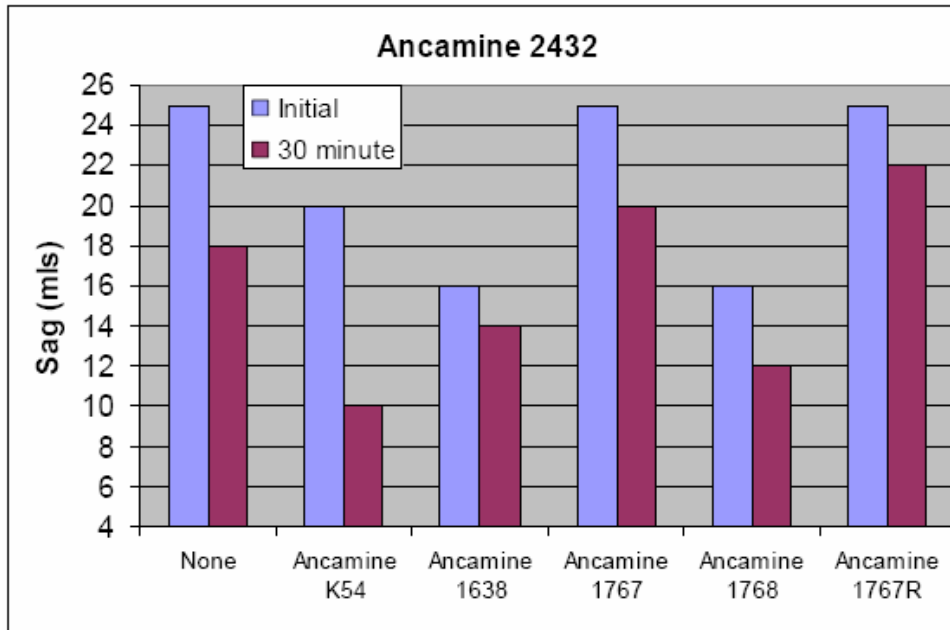
Graph 2



Graph 2 indicates that Ancamine 2368, a modified cycloaliphatic amine with a viscosity of 200 cps and an amine value of 325 mg KOH/g, is a poor choice for an accelerator with Garamite® and Epoxy systems for sag resistance.

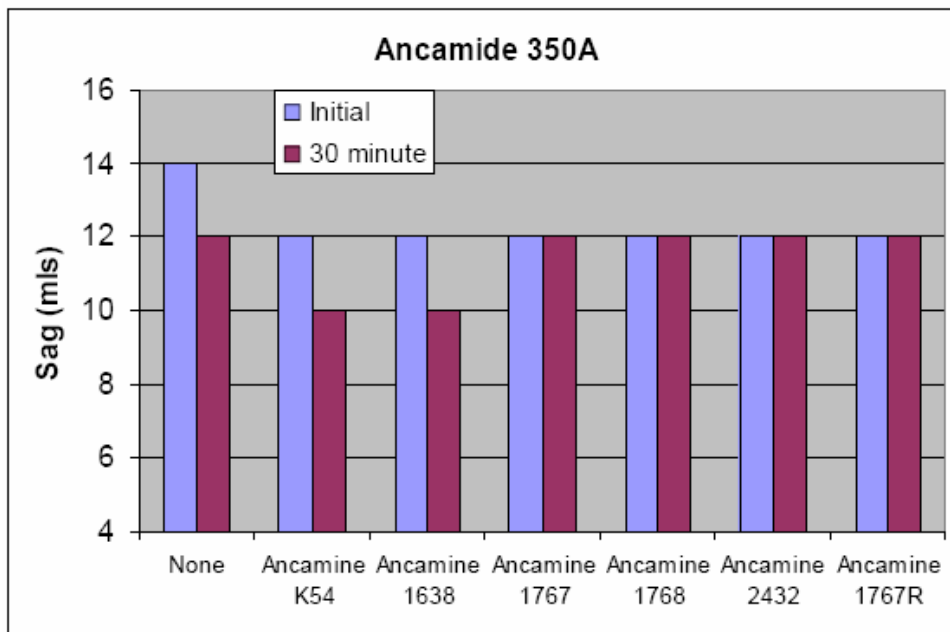


Graph 3



Graph 3 shows that when using Ancamine 2432, a modified aliphatic amine with a viscosity of 300 cps and an amine value of 368 mg KOH/g, excellent sag performance can be achieved. The accelerator that gives the best sag resistance after 30 minutes is Ancamine 1767.

Graph 4



Graph 4 illustrates that when using Ancamide 350A, an imidazoline polyamide with a viscosity of 11000 cps and an amine value of 380 mg KOH/g, the accelerators do not have a significant effect on sag resistance after 30 minutes and very consistent sag performance is achieved.



Conclusions: Out of the five hardeners tested, Ancamine 2280, a modified cycloaliphatic amine, and Ancamine 2432, a modified aliphatic amine, showed the most variance in sag resistance when the accelerators were added. The accelerators that showed the least loss in sag resistance with these two hardeners were Ancamine 1767 and Ancamine 2432, which are the accelerators with the lowest amine values. It seems that accelerators with high amine values may exchange with the quaternary amine surface modifiers on the Garamite®, causing the loss in sag performance. Thus accelerators with high amine values should be avoided in Epoxy systems thixed with Garamite® Mixed Mineral Thixotropes.

Very consistent sag performance was achieved with the Ancamide 350A hardened system; it has a very long gel time, however, so applicability may be limited. The initial samples prepared with the Ancamine 1767 hardened system showed no difference in sag resistance; however all of these samples had set up after 30 minutes so a measurement could not be taken for this series. Sag resistances in the Ancamine 2368 hardened systems were very low. Finally when high sag resistance is required, the system of Ancamine 2432 with Ancamine 1767 should be suggested.

Appendix:

Table IV: Description of Hardeners and Accelerators

<u>Hardener</u>	<u>Type</u>	<u>phr</u>	<u>Viscosity</u> (cps@77°F)	<u>AmineValue</u> (Mg KOH/g)	<u>Gel Time</u> (min@77°F)
Ancamine 2280	Modified cycloaliphatic amine	58	450	250	50
Ancamine 2368	Modified cycloaliphatic amine	52	200	325	32
Ancamine 2432	Modified aliphatic amine	46	300	368	27
Ancamide 350A	Imidazoline polyamide	55	11000	380	200
Ancamine 1767	Modified aliphatic amine	100	6000	310	7
Accelerator					
Ancamine K54	Tertiary amine	1-15	200	630	45
Ancamine 1638	Modified aliphatic amine	15	100	1070	15
Ancamine 1767	Modified aliphatic amine	100	6000	310	7
Ancamine 1768	Modified aliphatic amine	50	220	630	10
Ancamine 2432	Modified aliphatic amine	46	300	368	27

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