CVC Specialty Chemicals, Inc. | Technical Service Report | TSR #031030

Date: October 30, 2003

Subject: Compare reactivity, viscosity stability, T_g, and other characteristics, of BC-120 against BF_3-Amine Complexes

Background: Information was lacking on the comparative properties of Omicure BC-120 vs. the more widely used BF_3-Amine Complexes. BCl_3 and BF_3 are in a class of curing agents known as Lewis Acids. Both are gaseous under standard temperature and pressure conditions. Complexes are made to provide liquid or solid forms under conditions of normal use. When heated, boron tri-halide is liberated to catalyze the cure of the epoxy resin. The use of different amines will provide complexes that liberate boron trihalide at different temperatures. This study was undertaken to compare the characteristic properties of BC-120 against several different BF_3 Complexes.

US Patent # 3,395,121, titled "Curing Epoxy Resins with Boron Trichloride-Tertiary Amine Complexes" compares use of BF_3 complexes vs. BCl_3 complexes. This patent indicates BCl_3 complexes show much better hydrolytic stability, longer pot life at elevated temperature, shorter cure times, and much less brittle character in cured compositions. In addition, it was noted that both materials liberate a certain amount of halide on decomposition and cure. One disadvantage of BF_3 types is a propensity to etch glass fillers, fibers, or reinforcement in epoxy formulations. This can obviously cause problems in some applications such as prepregs, filiment windings and other applications that may contain glass fiber or utilize glass matting. Since BCl_3 complexes do not contain fluorine, they do not exhibit this disadvantage.

Experimental: Selected BF_3-Amine complex samples were obtained. These were BF_3-MEA from Ato-Tech, Anchor 1040 (BF_3 complexed with benzyl amine and isopropyl amine) from Air Products, Anchor 1115 (BF_3 complexed with isopropyl amine adduct) from Air Products, and Anchor 1170 (BF_3 complexed with chlorobenzyl amine) also from Air Products. Tests were run on each of the BF_3 complexes to determine optimal concentration within their recommended range by T_g. Optimal concentration of BC-120 was already known by previous work to be 8 phr. The recommended range, optimal weight ratio in DGEBA, and resulting T_g is shown in Table 1.

Tests were run using optimal catalyst concentration to measure time to 90% full cure at varying temperatures. These results were obtained from cure curves run on CVC's Perkin Elmer DSC 7, using the kinetics program included with the DSC 7 (See Graph 1). Result for Anchor 1170 is not included since this material is designed for cure at much lower temperatures.

Viscosity stability at 130°C was run by measuring the initial as-made Brookfield viscosity at 25°C of samples made with each curing agent, and samples aged in a 130°F water bath after intervals of 24, 72, 168, and 216 hours (See Graph 2). Result for Anchor 1170 is not included since this material is more reactive and is designed for cure at much lower temperatures. As such it has very limited latency compared with the other products shown.

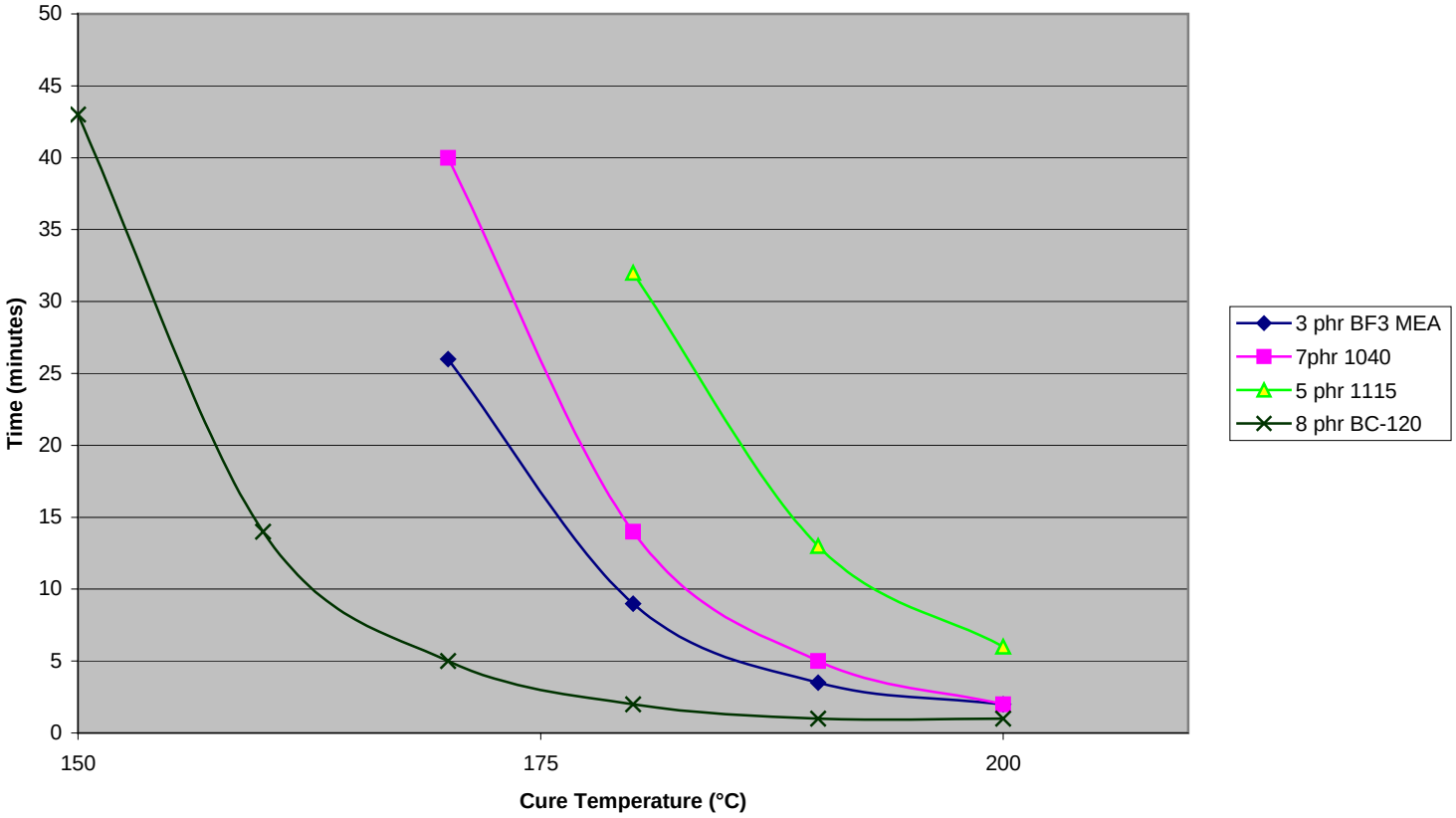
Discussion of Results Results in Table 1 indicate that BC-120 has an optimal T_g within the range of the BF_3 complexes tested. Results shown in Graph 1 indicate that BC-120 shows much shorter time to 90% full cure at any given temperature over the range 150 to 200°C. Results shown in Graph 2 indicate that BC-120 has much longer latency at 130°F than any of the BF_3 complexes tested.

Conclusions: The data generated in this series of experiments indicates several advantages to the use of BC-120 over a variety of different BF_3--Amine Complexes. These include longer latency, better reactivity, and similar thermal performance. Also indicated in the patent literature is improved hydrolytic stability and a decrease in the brittleness of cured resin compositions.

Table 1 – Optimal Concentration of Curing Agent in DGEBA (EEW = 185 – 190)

Curing Agent	Recommended Range (phr)	Optimal Concentration (phr)	T_g (°C)
BC-120	**1 – 9**	**8**	**140**
BF_3-MEA	**1 – 5**	**3**	**154**
Anchor 1040	**7 – 12**	**7**	**129**
Anchor 1115	**5 – 10**	**5**	**140**
Anchor 1170	**5 - 10**	**5**	**147**

Graph 1 --BF3 v. BC-120 Time to 90% Cure



Graph 2 - Viscosity Stability of BF3 v. BC-120 @ 130°F