Monsanto Chemical Company • Organic Chemicals Division
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CHEMICAL SPECIALTIES DATA REPORT 1460

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AROCLOR* FIRE-RETARDING PLASTICIZERS AND MODIFIERS FOR EPOXY RESINS

Because of their outstanding chemical resistance and adhesive characteristics, epoxy resins are widely used for the production of surface coatings, adhesives, laminations and plastic parts.

To impart flame resistance -- with attendant lowering of cost -- Aroclor compounds can be added to the resins in various proportions. The flame retardant resins so produced have many important uses. In laminations and plastic parts, for example, it is often necessary or even mandatory that flame resistance be built into the finished product.

By selecting the proper Aroclor, formulators can meet requirements for fire resistance while, at the same time, adjusting such other important properties as flexibility, hardness and viscosity.

Chemically, the Aroclor compounds are di- and poly-phenyls chlorinated to a definite per-cent-by weight of inert, bound chlorine. Physically, they range in form and appearance from Aroclor 1221, mobile oily liquids, to Aroclor 5460, hard transparent resins. Chemically, the Aroclor compounds are impervious to moisture, acids and alkalis; they are extremely heat stable and will not support combustion. When added to resins, the Aroclor compounds function as low-cost plasticizers imparting fire-retardant properties to the resin formulation as well as other desirable end-product characteristics.

This report covers the application of Aroclor plasticizers in epoxy resin formulations.

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* Aroclor is a trademark of Monsanto Chemical Company for its chlorinated aromatic hydrocarbons and their derivatives, including chlorinated diphenyl. Reg. U. S. Pat. Ofc.

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THE NEED FOR AROCLOR PLASTICIZERS

When incorporated into epoxy resin formulations, Aroclor compounds can be used to impart fire resistance; to increase flexibility; to improve processing; to maintain chemical resistance; to maintain adhesive characteristics and to lower finished product costs. The quality advantage of self-extinguishing epoxy compositions is of great interest in electrical, structural and coatings applications. Aroclor compounds are outstanding for providing fire retardancy since they offer the additional advantages of high chemical resistance (inertness), resistance to water, acid and alkali extraction -- and very low per pound cost. Frequently, the addition of Aroclor results in lower over-all finished product costs by their replacing a percentage of the higher cost ingredients.

SELECTED PROPERTIES OF AROCLOR COMPOUNDS

The Aroclor compounds, from Aroclor 1221 to Aroclor 5460, span a wide range of physical properties. Over the series, the properties vary in a regular gradient; giving a choice for optimum usefulness in any particular application. Adjustment of formulation properties can further be made by "blending" two or more Aroclor compounds to give a mixture for the specific use. The five* (of twelve) Aroclor compounds listed illustrate the variety of properties available.

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* For a complete summary describing the properties of the entire series of twelve Aroclor compounds, request a copy of "The Aroclor Compounds."

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TABLE I
THE PROPERTIES OF AROCLOR COMPOUNDS

Form	Aroclor 1221 Colorless mobile oil	Aroclor 1242 Practically colorless mobile oil	Aroclor 1260 Light yellow soft sticky resin	Aroclor 1268 White opaque resin	Aroclor 5460 Yellow Transparent resin
Acidity - Maximum (Mgm.KOH per Gm.).....	0.015	0.015	0.015	0.05	0.07
Typical Density Specific Gravity 25°/25°C (77°/77°F).....	1.182	1.380	1.620	1.810	1.670
Lbs./Gallon-25°C (77°F)..	9.85	11.50	13.50	15.09	13.91
Distillation Range - ASTM D-20 (Mod.) Corr. °C.....	275°-320°	325°-360°	385°-420°	435°-450°	280°-335° at 5 mm. Hg.
Evaporation Loss - % - ASTM D-6 Mod. 163°C - 5 hrs.....	-	3.0 to 3.6	0.5 to 0.8	0.1 to 0.2	0.03
100°C - 6 hrs.....	1.0 to 1.5	0.0 to 0.4	0.0 to 0.1	0.0 to 0.6	1.5 to 1.7 (at 260°-5 hrs.)
Flash Point - Cleveland Open Cup-°C....	141°-150°	176°-180°	None	None	None
°F....	286°-302°	348°-356°	"	"	"
Fire Point - Cleveland Open Cup - °C.....	176°	None*	None	None	None
°F.....	349°	"	"	"	"
Pour Point-ASTM D-97-°C..	Crystals at 1°C	-19°	31°	--	--
°F..	Crystals at 34°F	2°	88°	--	--
Softening Point -- ASTM E-28 - °C.....	--	--	--	135° to 160° (hold pt.)	100°to 105.5°
°F.....	--	--	--	275° to 320° (hold pt.)	212°to 222°
Refractive Index - D-line - 20°C.....	1.617-1.618	1.627-1.629	1.647-1.649	--	1.660-1.665
Viscosity-Saybolt U. 210°F (98.9°C).....	30-31	34-35	72-78	--	--
130°F (54.4°C).....	35-37	49-56	3200-4500	--	--
100°F (37.8°C).....	40-42	80-93	--	--	--

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* None indicates "No fire point up to boiling temperature."

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PROPERTY IMPROVEMENTS OF EPOXY FORMULATIONS

The Aroclor compounds represent one of the few plasticizer type materials that possess a high degree of compatibility with epoxy resins. The lower chlorinated members, 1221 and 1232, impart a high degree of flexibility to epoxies while the more resinous and solid Aroclor compounds, e.g., 5460, have little flexibilizing effect, in fact, tend to act as reinforcing resins. Aroclor compounds, in general, have negligible influence on the hardness, tensile or compressive yield strength of epoxy resins. In phthalic anhydride cured systems, the ultimate compressive strengtn can even be improved by addition of solid Aroclor compounds to the formulation.

When used in the range of 15 to 20 parts per hundred of resin, all of the Aroclor compounds greatly retard the burning rate of epoxy compositions. A small amount of antimony oxide added to the Aroclor formulations further increases the fire-retardance greatly and the epoxy formulations become non-burning. Aroclor creates self-extinguishing characteristic; the antimony oxide functions to reduce afterglow. In addition, the antimony oxide can replace a portion of any fillers that may be employed.

Aroclor compounds are especially useful whenever finished product cost plus other specific qualities, such as good resistance to chemicals (especially acid and alkali), are important factors. In surface coatings, for example, Aroclor 1254, 1260 and 1262 provide both formulating economy and excellent chemical resistance. However, Aroclor should not be used where maximum organic solvent resistance is desired since it is a true plasticizer and not chemically bound to the resin.

In potting compounds, Aroclor compounds can provide added flexibility to improve the shock resistance of the encapsulated device. In addition, by proper selection of the Aroclor compound, the coating viscosity can be greatly reduced.

Better tackiness can be developed in epoxy adhesives by the "stickiness" property of Aroclor. In epoxy sealants, besides fire resistance, easier workability develops in the formulations.

Wherever fire-resistance, inertness, better flow and improved handling characteristics are needed, Aroclor compounds should be evaluated. Their unique properties and the effect on the epoxy resin as well as their low cost per pound recommend their evaluation for a variety of epoxy resin end uses.

LABORATORY DATA -- AROCLOR COMPOUNDS IN LIQUID EPOXY RESINS

The following data have been assembled as a guide for formulators of epoxy resins. Careful study of these data disclose several generalizations.

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The use of liquid Aroclor in an aliphatic amine-cured system -- 20 parts per 100 parts of resin basis -- results in a self-extinguishing formulation. Replacement of five parts of Aroclor with five parts of antimony oxide produces materials with still better flame retardancy. Flexural strength and compressive yield strength is improved while tensile strength remains unaffected. There is a slight lowering of heat distortion temperature and ultimate compressive strength.

In phthalic anhydride cured systems, solid Aroclor compounds greatly improve ultimate compressive strength, flexural strength and tensile properties. Compressive yield strength is comparable to the unplasticized control while heat distortion temperature is lowered slightly.

The liquid epoxy resins used in the experiments recorded in Table II are representative of commercial types such as Ciba's Araldite 6010, Jones and Dabney's EPI-Rez 510, Shell's Epon 828, Bakelite's ERL 2774 or Dow's DER 332.

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TABLE II

AROCLOR IN LIQUID EPOXY RESIN

Formulation (parts by weight)	Cure: 16 Hr. R.T. + 2 hrs.@ 100°C. Control 1	Modified 2	3	4	5	20 Hrs. @ 150°C. 6	7	8	9	Control 10
Liquid Epoxy Resin	100	100	100	100	100	100	100	100	100	100
Diethylene Triamine	13	13	13	13	13	-	-	-	-	-
Aroclor 1260	-	20	15	-	-	-	-	-	-	-
Aroclor 1242	-	-	-	20	15	-	-	-	-	-
Antimony Oxide	-	-	5	-	5	-	5	-	5	-
Aroclor 1268	-	-	-	-	-	20	15	-	-	-
Aroclor 5460	-	-	-	-	-	-	-	20	15	-
Phthalic Anhydride	-	-	-	-	-	75	75	75	75	75
DMP 30 (Rohm & Haas)	-	-	-	-	-	.1	.1	.1	.1	.1
Flammability ASTM 635-56T	.82 in/ min.	S.E. 1-1/8"	N.B.	S.E. 2-1/8"	N.B.	.38 in/ min.	N.B.	.38 in/ min.	N.B.	.6 in/ min.
Heat Distortion Temp. °C.	117.5	87	92	79.5	81.0	128	134	135.5	135	143
Rockwell "M"	104	103	106	108	107	112	112	113	111	111
Flex Strength PSI	20,000	23,800	22,100	21,800	22,200	25,900	20,000	26,300	19,000	16,900
Tensile PSI	11,400	13,000	9,800	10,700	12,800	13,500	11,100	11,500	10,200	11,000
Compressive Yield Strength PSI	15,700	17,250	16,950	17,900	16,700	22,300	20,500	21,000	21,200	20,900
Compressive Ultimate Strength PSI	43,000	32,900	38,150	32,500	32,400	36,500	36,700	41,700	40,700	23,400

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S.E. = Self extinguishing
N.B. = No burning

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Table III illustrates some other typical physical property modifications imparted by Aroclor 1221 and 1248. In each case, the formulation consisted of 100 parts epoxy resin (as in Table II), 25 parts of Aroclor and 8 parts of tetraethylenepentamine.

TABLE III

Compatibility	Control	Aroclor 1221	Aroclor 1248
Liquid	Compatible	Compatible	Compatible
Room Temp. Cure	Compatible	Compatible	Compatible
30 Min. Cure 100°C.	Compatible	Compatible	Compatible
VISCOSITY*			
Before adding curing agent	12,800	1,650	7,700
After adding curing agent	7,900	1,200	4,300
PHYSICAL PROPERTIES**			
Izod Impact Resistance	0.2	-	0.6
Clarity: % light reflected	3.0	3.0	3.0
Shore D Hardness (10 sec.)	78	8	40
PHYSICAL PROPERTIES AFTER HEAT AGING***			
Shore D Hardness (10 sec.)	86	81	88
% Weight Change	-0.012	-2.118	-0.682

* Brookfield LVF #4 spindle at 60 r.p.m.
** Samples cured 30 min. at 100°C. with 8 p.hr. tetraethylenepentamine
*** Aged 24 hours at 86°C. in activated carbon

EFFECT OF CONCENTRATION OF ANTIMONY OXIDE AND AROCLOR ON FLAMMIBILITY

To determine the effective concentration of Antimony oxide and Aroclor (equal parts by weight), several test resin mixtures were prepared. Liquid epoxy resin (Shell Epikote 834) was mixed with Aroclor and antimony oxide and cured with 8% diethylenetriamine. Specimens were prepared in wax molds 9" x ½" x ¼". The flammibility tests were based upon the British Ministry of Supply Aircraft Specifications DTD 5537 Appendix 10. This procedure consisted of clamping the test specimen in a support with its longitudinal axis horizontal and its transverse axis inclined at 45°. A bunsen burner was applied with the tip of the flame 1/2" to 3/4" just in contact with the end of the specimen. At the end of 30 seconds, the flame was removed and the specimen allowed to burn. If burning did not continue, the flame was replaced for 30 seconds and if the specimen again failed to continue burning, the material was classed as self-extinguishing. These results are listed in Table IV.

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TABLE IV

AROCLOR AND ANTIMONY OXIDE (50-50 Mix)

Weight % in Resin	Aroclor 1242	Aroclor 1260	Aroclor 4465	Aroclor 5460
5	Inflammable	Inflammable	Inflammable	Inflammable
7.5	--	S.I.	S.I.	S.E. (40 sec.)
10	Inflammable	S.E. (30 sec.)	S.E. (20 sec.)	S.E. (20 sec.)
15	S.E. (30 sec.)	S.E. immediate	S.E. immediate	S.E. immediate

S.E. = Self-extinguishing
S.I. = Semi-inflammable

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For samples of the Aroclor products mentioned in this report, contact the nearest branch office (see attached list) or write to:

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