after 10 minutes. It was kept at 90-95°C for 5 hr and then given several hot water washes. No unreacted hydrolyzed Aroclor was found on extraction with caustic. The organic material solidifies on standing and could be ground to a powder (124 g) possessing an mp. of 112°C (not sharp. Conversion was 92.9%.

Anal. Calcd. MW 547.4: C, 35.90; H, 1.83; Cl, 51.6; OH, 6.22
Found: C, 36.92; H, 2.71; Cl, 50.19; 50.83; OH, 6.64, 6.73.

The OH content of starting material was determined 2 years after its preparation when the modified OH method was available.

Hydrolyzed Aroclor 5460 - Expt. 12 (328995-6) The same general procedure as in the previous run was used. Hydrolyzed Aroclor 5460 (121.2 g of mixed di- and tri-hydroxy cpd, 328966-7) was dissolved in NaOH (0.75 mole 2.5 N) and the ethylene chlorohydrin (1 mole 80.4 g) was added at once at 85°C. After 20 more minutes the pH of the mixture turned to 8. It was heated for a total of 3 hr at 85-95°C, and then washed until free of ionic Cl, to give 132 g of a light powder. Conversion was 92.5% calculated as a mixture of di- and tri-ether.

Anal. Calcd. tri-ether: C, 44.95; H, 3.04; Cl, 37.0; OH, 7.98
Cl, 43.6; OH, 5.52
Found: C, 44.21; H, 2.61; Cl, 42.38, 42.44; OH, 5.45, 5.73

Reaction between ethyl chloroacetate and hydrolyzed Aroclors

Hydrolyzed Aroclor 1271 - Expt. 13 (334605-8) Caustic (0.8 mole 32 g) was dissolved in 900 ml of absolute ethanol and hydrolyzed Aroclor 1271 (0.4 mole, 184.9 g, 7.60% OH) was added. Redistilled ethyl chloroacetate (0.88 mole, 108 g) was added at once at 70°C and the system was refluxed for 14 hr; salt precipitation started after 0.5 hr and total salt formed was 98.2 g(0.84 mole). The ethanol was distilled off at reduced pressure. The semi-solid residue is very soluble in the low normal and branched alcohols and dioxane. No precipitation occurred from ethanol solution kept at -40°C over night. Precipitation using solvents such as pentane, ether, water failed to yield a crystalline precipitate; only oils were formed. The crude ester was dissolved in 800 ml of ethanol and refluxed 10 hr with NaOH (0.9 mole, 9 N). The ethanol-water mixture (750 ml) was distilled off, the residue diluted with 1000 ml of water, and refluxed. The water soluble material was filtered off and the filtrate was acidified to yield 162 g of acid.

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