in 44% yield. It was a crystalline solid melting at 167.5–169° C. (recrystallized from hexane).

Anal. Calcd. for $C_{14}H_6Cl_6O_4$: C, 37.29; H, 1.34; Cl, 47.18. Found: C, 37.40; H, 160; Cl, 47.12.

Octachloronaphthalene. β-Methyl naphthoate (10 grams) was chlorinated in 250 ml. of tetrachloroethylene for 20 hours at 110° C. in the presence of 2.0 grams of ferric chloride and 1.0 gram of iron powder. After steam distillation to remove solvent, a brown solid (20.0 grams) was collected which on recrystallization from chloroform gave yellow needles (13.0 grams) melting at 196–198° C. It was identified as octachloronaphthalene by comparison of its IR spectrum with that reported in the literature (*8*).

Perchloro-1,4-dihydronaphthalene. α-Methyl naphthoate (10 grams) was chlorinated in 200 ml. of 1,1,2,2-tetrachloroethane at 110° C. for 6 hours in the presence of 1.0 gram each of ferric chloride and iron powder. After removing solvent by steam distillation, the solid product (21.0 grams) collected by filtration was dissolved in chloroform and precipitated with methanol to give 5.8 grams of a dark brown solid. After several recrystallizations from tetrachloroethylene a light yellow solid melting at 216–218° C. was obtained. The NMR spectrum of the material showed the absence of hydrogens. The IR spectrum showed carbonyl absorption typical of a quinone [IR in KBr: ν(cm.$^{-1}$)—1818(w), 1678(s), 1588(s), 1505(m), 1353(s), 1279(s), 1217(s), 1127(s), 944(m), 915(s), 895(m), 816(m), 730(s), 600(s)]. The material analyzed for hexachloro-1,4-naphthoquinone whose melting point has been reported as 216–217° C. (*12*).

Anal. Calcd. for $C_{10}Cl_6O_2$: C, 32.92; Cl, 58.31. Found: C, 32.96; Cl, 58.46. Hexachloro-1,4-naphthoquinone was presumably formed from perchloro-1,4-dihydronaphthalene by hydrolysis during the workup.

Decachloroindan. α-Methyl naphthoate (25 grams) was chlorinated in 250 ml. of 1,1,2,2-tetrachloroethane at 140° C. for 22 hours. The crude product was dissolved in chloroform and precipitated with methanol to give 18.6 grams of a solid which on recrystallization from hexane melted at 133–135° C. One reported melting point for decachloroindan is 136–137° C. (*9*). The infrared spectrum of this material was typical of a perhalogenated hydrocarbon without functional groups [IR in KBr: ν(cm.$^{-1}$)—1375(s), 1262(m), 1210(m), 1170(m), 985(m), 868(m), 800(m), 748(s), 637(s), 562(m)]. The elemental analysis agreed with the decachloroindan structure.

Anal. Calcd. for C_9Cl_{10}: C, 23.36; Cl, 76.64. Found: C, 23.43; Cl, 76.58.

Decachloroindan was the only product isolated when 2,6-dicarbomethoxynaphthalene was chlorinated in hexachlorobutadiene-1,3 at 160° C. for 8 hours.

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Halogenated Phthalimidoacetic Acids

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Twelve halogenated phthalimidoacetic acids have been prepared by condensation of glycine with halogenated phthalic acids or anhydrides at 170° to 185° C.

THE PHTHALOYLATION of an amino acid is conveniently achieved by condensation of the amino acid with the phthalic anhydride at 170° to 185° C. This reaction may be accomplished either by fusion of an equimolar mixture of the reactants (*1*) (Method A), or by reaction in a suitable solvent, such as nitrobenzene (*2, 3*) (Method B). The phthalic acid may be used instead of the anhydride (*2, 6*). Twelve halogenated phthalimidoacetic acids, nine of which had not previously been reported, have been prepared from glycine by these methods. The choice of halogenated phthalic acid or anhydride as the starting material was determined by which compound was more readily accessible. The experimental results for the method giving the best yield of each halogenated phthalimidoacetic acid are summarized in Table I.

Comparison of the ultraviolet absorption spectra of these compounds in alcoholic and in aqueous base solutions showed that basic conditions produced the hypsochromic and hypochromic effects characteristic of saponification of the imide ring (*2, 3*). Table II summarizes the spectral data for the bands corresponding to the phthalimidoacetic acid bands at 292.5 nanomoles (log ϵ_{max} 3.38) in 95% ethanol and at 269 nanomoles (log ϵ_{max} 3.04) in dilute sodium hydroxide (*3*).

Table I. Halogenated Phthalimidoacetic Acids

						Analysis[b]							
						Calcd.				Found			
Substituents	Prepd. from	Method	Yield, %	Recryst. from	M.P.,[a] ° C.	C	H	N	Halogen	C	H	N	Halogen
3-Fluoro	Acid	A	94	Water	223–225	53.82	2.71	6.28	...	54.03	3.11	6.44	...
4-Fluoro	Acid	A	57	Water	176–178	53.82	2.71	6.28	...	54.12	3.01	6.42	...
3-Chloro	Acid	A	99	Water	205.5–207	50.13	2.52	5.85	14.80	50.00	2.64	5.88	14.81
4-Chloro	Acid	A	66	Water	206–207[c]	...	...	...	...	...	...	...	...
3,4,5,6-Tetrachloro	Anhydride	B	89	Acetone	307–309[d]	...	...	4.08	41.35	...	...	4.09	41.38
3-Bromo	Acid	A	76	Aq. alcohol	202.5–203	42.28	2.13	4.93	28.13	42.29	2.19	5.08	28.34
4-Bromo	Acid	A	79	Aq. alcohol	203.5–205	42.28	2.13	4.93	28.13	42.33	2.15	4.98	28.25
3,4,5,6-Tetrabromo	Anhydride	B	85	Dioxane	334–336	...	...	2.69	61.38	...	...	2.80	61.16
3-Iodo	Acid	B	63	Water	213.5–214.5	...	...	4.23	38.33	...	...	4.21	38.61
4-Iodo	Acid	A	45	Aq. alcohol	190–192	...	...	4.23	38.33	...	...	4.43	38.52
3,6-Diiodo	Anhydride	B	57	Water	257–258.5	...	...	3.07	55.54	...	...	3.02	55.78
3,4,5,6-Tetraiodo	Anhydride	B	74	Aq. dioxane	329–333[e,f]	...	...	1.98	71.62	...	...	2.19	71.56

[a] Melting points are those of analytical samples, and were determined with an "Electrothermal" apparatus. [b] All microanalyses were done by Midwest Microlab, Inc., Indianapolis, Ind. [c] Reported (*5*) m.p. 205° C. [d] Reported (*5*) m.p. 298° C. (dec.). [e] Decomposition. [f] Reported (*4*) m.p. 327–9° C. (dec.).

Table II. Absorption Spectra of Halogenated Phthalimidoacetic Acids[a]

	In 95% EtOH		In Dilute NaOH[b]	
Substituents	λ_{max}, nm.	Log ϵ_{max}	λ_{max}, nm.	Log ϵ_{max}
3-Fluoro	300	3.56	271	3.29
4-Fluoro	288	3.15	269	3.19
3-Chloro	309	3.47	277	2.95
4-Chloro	296	3.26	284[c]	2.90
3,4,5,6-	332	3.40	294	2.77
Tetrachloro	316	3.51	287	2.77
3-Bromo	310	3.46	276	2.96
4-Bromo	299	3.28	284[c]	2.90
3,4,5,6-	339	3.25	296	2.68
Tetrabromo	330	3.24	287	2.71
3-Iodo	320	3.44	289[c]	2.95
4-Iodo	309	3.47	...	...[d,e]
3,6-Diiodo	340	3.38	...	...[d,f]
3,4,5,6-	355	3.45	...	...[d,g]
Tetraiodo	340[c]	3.47		

[a] Absorption spectra were determined with a Cary Model 15 spectrophotometer, using concentrations of 10^{-5} to 10^{-2} molar. [b] Average pH 12.26. [c] Approximate wave length at shoulder. [d] The wavelength range in which these peaks might be expected showed only end absorption. [e] Shoulder at 249 nm., log ϵ 4.10. [f] Peak at 242 nm., log ϵ_{max} 4.22. [g] Shoulder at 258 nm., log ϵ 4.36; and peak at 238 nm., log ϵ_{max} 4.71.

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