DISCUSSION

At temperatures below 40° C., SPS typically crystallizes from hexane in fine-grained β crystals. On standing in solvent these β crystals (accelerated by the presence of finely divided succinic acid) slowly transform to spherulitic β' crystals. This β' form, slowly obtained from solvent, is the highest melting and apparently only stable crystalline form of SPS.

It is now clear that the six triglycerides (optical isomerism disregarded) obtainable from palmitic and stearic acids all show α, β', and β forms and apparently no other in the range 0° C. to the completely melting point. In Table IV an effort is made to codify the crystallization behavior of the six different triglycerides in their different forms. Considerable individuality is evident despite the inevitable great similarity. One feature brought out in the table is the high nucleation tendency of β at low temperatures ($<$ 40° C.). This correlates with the observation that nonpolar solvents (the more powerful solvents for triglycerides) favor β crystallization because crystallization from more powerful solvents tends to occur at lower temperatures.

It is tempting to try to account for the highly individual features of polymorphism exhibited by the C_{16}–C_{18} triglycerides, but there is no reasonable hope of doing so before there is a detailed knowledge of crystal structure for at least one β and one β' form.

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Phenol Esters of 3,4,5-Triiodobenzoic Acid

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The 3,4,5-triiodobenzoic esters of fifty phenols have been prepared by reacting the various phenols with 3,4,5-triiodobenzoyl chloride.

WE HAVE PREVIOUSLY reported the use of 3,4,5-triiodobenzoyl chloride as a reagent for the preparation of derivatives of cellosolves (*2*), carbitols (*2*), alcohols (*3*) and mercaptans (*4*). This simple method has now been extended to the preparation of derivatives of a number of phenols. These compounds have proven useful for the characterization of this class of compounds.

EXPERIMENTAL

The phenols were used as obtained from Eastman Kodak with the exception of *p*-chlorophenol which was purified by distillation. The acid chloride was prepared by the method of Klemme and Hunter (*1*) except that cyclohexane was used for crystallization.

Table I. Properties of Phenols

Phenol Used	M.P. ° C.	Yield %	Formula	Iodine % Calcd.	Iodine % Found
2-Aceto-1-naphthol[a]	237.2–238.0[d]	57	$C_{19}H_{11}O_3I_3$	57.0	57.14
Acetyl-*p*-methylaminophenol	240.2–241.6	32	$C_{16}H_{12}O_3NI_3$	58.85	58.59
p-tert. Amylphenol	170.8–172.0	36	$C_{18}H_{17}O_2I_3$	58.93	58.94
4-*tert.* Butyl-2-chlorophenol	190–190.8	69	$C_{17}H_{14}O_2ClI_3$	57.14	57.05
p-tert. Butylphenol	179–179.8	58	$C_{17}H_5O_2I_3$	60.24	60.24
Catechol	242.6–243.4	12	$C_{26}H_8O_4I_6$	70.92	70.83
Carvacrol	121–122[c]	68	$C_{17}H_{15}O_2I_3$	60.24	60.44
2-Chloro-5-hydroxytoluene	154.2–154.8	57	$C_{14}H_8O_2ClI_3$	60.98	60.53
o-Chlorophenol	162.6–163.8	72	$C_{13}H_6O_2ClI_3$	62.39	62.61
m-Chlorophenol	175–176	63	$C_{13}H_6O_2ClI_3$	62.39	62.26
p-Chlorophenol	146.4–147.3	55	$C_{13}H_6O_2ClI_3$	62.39	62.23
p-Chlorothymol	142.2–143	31	$C_{17}H_{14}O_2ClI_3$	57.14	57.22
m-Cresol	179–180	40	$C_{14}H_9O_2I_3$	64.54	64.47
p-Cresol	124–125	54	$C_{14}H_9O_2I_3$	64.54	64.16
2,4-Dichloro-1-naphthol[a]	235.5–237	20	$C_{17}H_7O_2Cl_2I_3$	54.81	54.76
2,6-Dichloro-4-nitrophenol	259.4–260.6	33	$C_{13}H_4O_4NCl_2I_3$	55.19	54.93
o-Hydroxydiphenyl	168.4–169.4	60	$C_{19}H_{11}O_2I_3$	58.39	58.02
p-Hydroxydiphenyl	195–195.6	84	$C_{19}H_{11}O_2I_3$	58.39	58.32
p-Hydroxypropiophenone[a]	195–195.4	49	$C_{16}H_{11}O_3I_3$	60.27	60.40
o-Iodophenol	179.2–180.4[c]	34	$C_{13}H_6O_2I_4$	72.32	72.32
2-Methoxy-4-methylphenol	149.9–151.3[c]	57	$C_{15}H_{11}O_3I_3$	61.40	61.26
β-Naphthol	225–226[g]	22	$C_{17}H_9O_2I_3$	60.82	60.65
o-Nitrophenol	153.5–154.5	31	$C_{13}H_6O_4NI_3$	61.34	61.24
m-Nitrophenol	180–181	33	$C_{13}H_6O_4NI_3$	61.34	61.16
p-Nitrophenol	199–200	63	$C_{13}H_6O_4NI_3$	61.34	61.02
Phenol	180.8–182	46	$C_{13}H_7O_2I_3$	66.11	66.57
Pyrogallol 1,3-dimethyl ether	185.5–187[f]	32	$C_{15}H_{10}O_3I_3$	59.87	60.15
Resorcinol monoethyl ether	145.2–146	45	$C_{15}H_{11}O_3I_3$	61.41	61.49
Resorcinol monomethyl ether	162.6–163.6	36	$C_{14}H_9O_3I_3$	62.84	62.59
Thymol	163.8–165	49	$C_{17}H_{15}O_2I_3$	60.24	60.70
p(1,1,3,3-Tetramethylbutyl)phenol	149–150.2	60	$C_{21}H_{23}O_2I_3$	55.33	55.36
Toluhydroquinone	238.2–240[g]	21	$C_{21}H_{10}O_4I_6$	70.00	69.87
2,4,5-Trichlorophenol	203.6–204.4	39	$C_{13}H_4O_2Cl_3I_3$	56.05	55.85
2,4-Dichlorophenol	189.8–190.6	36	$C_{13}H_5O_2Cl_2I_3$	59.05	59.00
2,4-Dihydroxybenzaldehyde[a]	250.5–252.2	20[b]	$C_{21}H_8O_5I_6$	69.10	68.73
2,2′-Dihydroxybinaphthyl-1,1′	270.2–270.8[e]	33	$C_{34}H_{16}O_4I_6$	60.92	60.91
3,5-Dinitro-*o*-cresol[a]	269.2–271	40	$C_{14}H_7O_6N_2I_3$	56.00	55.80
2,4-Dinitrophenol	206.0–207.2	35	$C_{13}H_5O_6N_2I_3$	57.18	57.09
m-Ethylphenol	131.6–132.6[c]	22	$C_{15}H_{11}O_2I_3$	63.04	63.17
Eugenol	170.5–171	33	$C_{17}H_{13}O_3I_3$	58.94	59.08
Guiacol	154.6–155.4	73	$C_{14}H_9O_3I_3$	62.83	62.62
Hydroquinone monobenzyl ether[a]	164.2–165	80	$C_{20}H_{13}O_3I_3$	55.85	55.69
Hydroquinone monoethyl ether	150.8–151.4	57	$C_{15}H_{11}O_3I_3$	61.41	61.49
Hydroquinone monomethyl ether	154.4–154.8	55	$C_{14}H_9O_3I_3$	62.84	62.59
p-Hydroxybenzaldehyde[a]	164.8–166[c]	54	$C_{14}H_7O_3I_3$	63.04	62.86
2-Hydroxy-5-chlorobenzaldehyde[a]	195.8–196.8[b]	44	$C_{14}H_6O_3ClI_3$	59.58	59.29
2-Hydroxy-1,4-dimethylbenzene	163.4–164.2	41	$C_{15}H_{11}O_2I_3$	63.05	63.16
4-Hydroxy-1,2-dimethylbenzene	147–148	25	$C_{15}H_{11}O_2I_3$	63.05	62.95
4-Hydroxy-1,3-dimethylbenzene	133–133.8	69	$C_{15}H_{11}O_2I_3$	63.05	63.42
5-Hydroxy-1,3-dimethylbenzene	182.4–183.2	81	$C_{15}H_{11}O_2I_3$	63.05	63.41

[a] Pyridine used. Solvents [b] 60% Dioxane and 40% water. [c] Ethanol. [d] Nitroethane.
[e] 50% Nitrobenzene and 50% nitroethane. [f] 1-Propanol. [g] Toluene.

The method described in (3) was used except as noted in Table I, when 2ml. of pyridine was added to the reaction mixture. Unless otherwise indicaged in Table I, 1-butanol was used as the solvent.

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