Melting Point and Index of Refraction Data for *n*-Alkyl Bromides, Thiols, and Disulfides

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Melting point and index of refraction data are reported for a number of *n*-alkyl bromides, thiols, and symmetrical disulfides ranging in carbon number from 12 to 56.

RECENT electron-spin resonance studies on irradiated *n*-alkyl disulfides and thiols necessitated the preparation of highly pure samples and the intermediate bromides. The bromides were obtained from commercial sources or by brominating purified alcohols and were purified by repeated vacuum distillation in a platinum spinning-band column. The thiols were prepared by the method of Urquhart, Gates, It is possible that small quantities of isomers were present, but their effect on refractive index is small.

Melting points were determined with short range thermometers calibrated against National Bureau of Standards references using a Thomas-Hoover capillary tube apparatus. and Connor (*2*) and purified by vacuum distillation. The disulfides were prepared by the method of Hesse and Truby (*1*). All compounds are estimated to be at least 99.95% pure on the basis of temperature programmed gas chromatography and neutron activation analysis. Flame ionization and thermal conductivity detectors were used on the gas chromatograph. Each material was dissolved in benzene and run in duplicate on the gas chromatograph. One sample was large enough to give a full scale deflection, while the other was approximately 100 times larger. Secondary impurity peaks were generally not observed, even with the larger samples. The neutron activation analysis showed an oxygen content of 0.03 ± 0.005% (*1*) and negligible amounts of bromine and sodium. Carbon, hydrogen, sulfur, and molecular weight determinations were not definitive. Refractive indices were determined with a Bausch & Lomb Precision Abbe Refractometer modified for high temperature operation and controlled to ±0.005° C. Sample temperature was read with a platinum resistance thermometer inserted in the heat transfer medium in the exit prism of the refractometer. The indices of refraction were all determined at 85° C. to obtain a comparison between the higher and lower members of the series. Selected literature values are included in Table I.

Table I. Melting Point and Index of Refraction Data for Various *n*-Alkyl Compounds

No. of C Atoms	M.P., ° C.	n_D^{85}
	Bromides	
12	-5.5 (Lit.)	1.43595
14	5.67 (Lit.)	1.43830
16	15	1.43973
18	27.4 (Lit.)	1.44135
20	36.2-37.0	1.44261
24	49.0-50.0	1.44434
26	54.5-55.5	1.44520
28	59.0-59.5	1.44600
	Thiols	
20	37.5-38.5	
22	42.5-43.0	
24	51.0-52.0	
26	56.5-57.5	
28	61.0-62.0	
	Disulfides	
16	30-31 (Lit.)	1.45985
24	55-55.5 (Lit.)	1.45820
32	62.5 (Lit).	1.45741
36	67.2-67.8	1.45713
40	71.0-72.0	1.45683
44	75.0-76.0	1.45655
48	75.0-76.0	
52	78.5-79.5	
56	80.5-81.5	

LITERATURE CITED

(1) Hesse, J.E., Truby, F.K., *Chem. & Ind. (London)* **1965**, p. 680.
(2) Urquhart, G.G., Gates, J.W., Jr., Connor, R., "Organic Syntheses," Collective Vol. **3**, p. 363, Wiley, New York, 1955.

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Compound Formation in the Methylene Chloride–Triethylamine System

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A RECENT investigation (*1*) showed the existence of a 1 to 1 binary compound in the chloroform-triethylamine system. The authors presumed that the compound was formed by a hydrogen bond between the nitrogen of the triethylamine and the electron deficient hydrogen on the chloroform. This report describes a similar study involving methylene chloride and triethylamine. The hydrogens of methylene chloride presumably are less electron deficient than that of chloroform. Exploratory phase diagram studies of these and related systems are being undertaken to choose