

X-Ray Diffraction Powder Data of Some Normal Alkyl Dithiol Esters of Sebacic Acid

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► X-ray diffraction powder data were obtained for *n*-alkyl dithiol esters of sebacic acid. All the individual compounds can be easily distinguished and identified by the diffraction data. The esters crystallize in tilted monomolecular layers. Long spacings increase regularly with increase in hydrocarbon

chain length, forming two series, one for odd- and one for even-membered series of the alkyl ester groups.

MANY of the dithiol esters of long-chain fatty acids are solid crystalline materials at ordinary temperatures, suitable for characterization by

x-ray diffraction. In this paper the x-ray diffraction powder patterns of methyl, *n*-amyl, *n*-heptyl, *n*-octyl, *n*-nonyl, *n*-decyl, *n*-undecyl, and *n*-dodecyl dithiol sebacic esters are reported.

EXPERIMENTAL

All the dithiol compounds used were

bered series, which is characteristic of long-chain compounds (2). The average increase for each additional carbon atom is approximately 1.12 A. for the dithiol esters of sebacic acid. This is less than the accepted value of 1.27 A. for the projected carbon-to-carbon distance. It appears that the dithiol esters of sebacic acid crystallize in tilted monomolecular layers, as do the diethyl esters of dicarboxylic acids, containing an even number of carbon atoms in the chain (1), and *n*-aliphatic thiol derivatives of monocarboxylic acids (5). The values of the long spacings reported (1) for the diethyl esters of dicarboxylic acids, containing an even number of carbon atoms in the acid chain, are approximately 0.2 A. larger than those of the corresponding dithiol esters given in Table II. This indicates that in both series the compounds crystallize with approximately the same angle of tilt. The dithiol esters containing an even number of carbon atoms in the thiol-ester groups have long spacings which are slightly greater than those containing an odd number of carbon atoms in the thiol ester group. This is the same as was reported for thiol esters of monocarboxylic acids (5). In general the reverse is true for long-chain aliphatic compounds containing odd and even numbers of carbon atoms in the aliphatic chain (2).

SUMMARY

X-ray diffraction powder data were obtained for 9 *n*-alkyl dithiol esters of sebacic acid. All individual compounds can be readily identified and distinguished by the x-ray diffraction data.

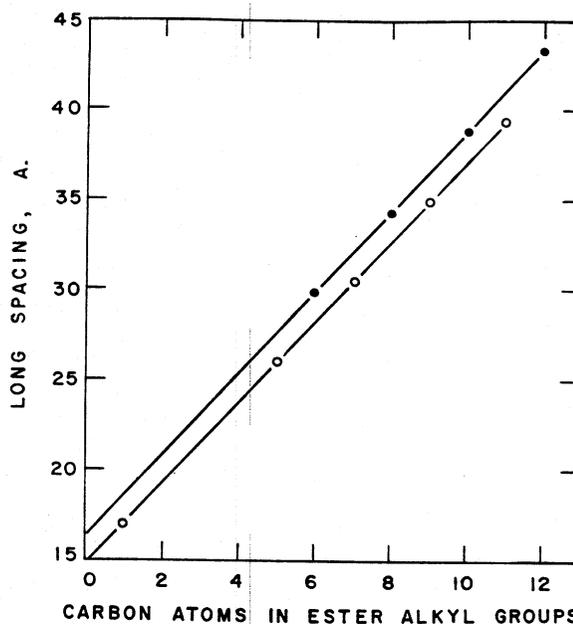


Figure 1. Long spacings of dithiol esters of sebacic acid

- Even number of carbon atoms in each alkyl group in ester
- Odd number of carbon atoms in each alkyl group in ester

The long spacings increase regularly with increasing ester-chain length forming an odd and an even series. The *n*-dialkyl dithiol sebacates crystallize in monomolecular tilted layers.

LITERATURE CITED

- (1) Francis, F., Collins, F. J. E., Piper, S. H., *Proc. Roy. Soc. (London)* **A158**, 691 (1937).
- (2) Malkin, T., *Nature* **127**, 126 (1931).
- (3) Sasin, R., Weiss, G. S., Wilfond, A. E., Sasin, G. S., *J. Org. Chem.* **21**, 1304 (1956).

- (4) Urquhart, G. G., Gates, J. W., Jr., Connor, R., "Organic Syntheses," Vol. 21, pp. 36-8, New York, Wiley, 1941.
- (5) Witnauer, L. P., Lutz, D. A., Sasin, G. S., Sasin, R., *J. Am. Oil Chemists' Soc.* **34**, 71 (1957).

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