Evidence for Nonplanarity in an Oxalamide*

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Mizushima[1] concluded that steric repulsion is the most important force determining the stable configuration of rotational isomers. We wish to report an interesting demonstration of the correctness of this conclusion.

Tetraisopropyl oxalamide (I) was obtained by the reaction of diisopropyl amine with oxalyl chloride, followed by repeated crystallization from a variety of solvents. Proton magnetic resonance (PMR) spectra were obtained with a Varian A-60 spectrometer equipped with a variable temperature insert.

The spectra of I in CD_3COCD_3 show two sets of methyl proton signals at 10°C and higher temperatures as would be expected from analogy with simple amides.[2] At -15°C these signals broaden and finally emerge as four sets at -40°C. The two isopropyl radicals on a nitrogen atom are nonequivalent as also are the two methyl groups within a radical. There can be nonequivalent methyl groups only if there is no molecular symmetry plane between the groups.[3] Since there must be such a plane if the whole molecule is planar, it must be that the halves of the molecule are not coplanar and, further, that rotation around the rotation axis $(-\overset{O}{\overset{\|}{C}}\overset{\downarrow}{—}\overset{O}{\overset{\|}{C}}-)$ is slow on the PMR time scale below about -15°C. Double bond character[1] in the rotation axis favors coplanarity. In spite of this, steric factors, that arise from the bulky amide groups, stabilize the nonplanar structure.

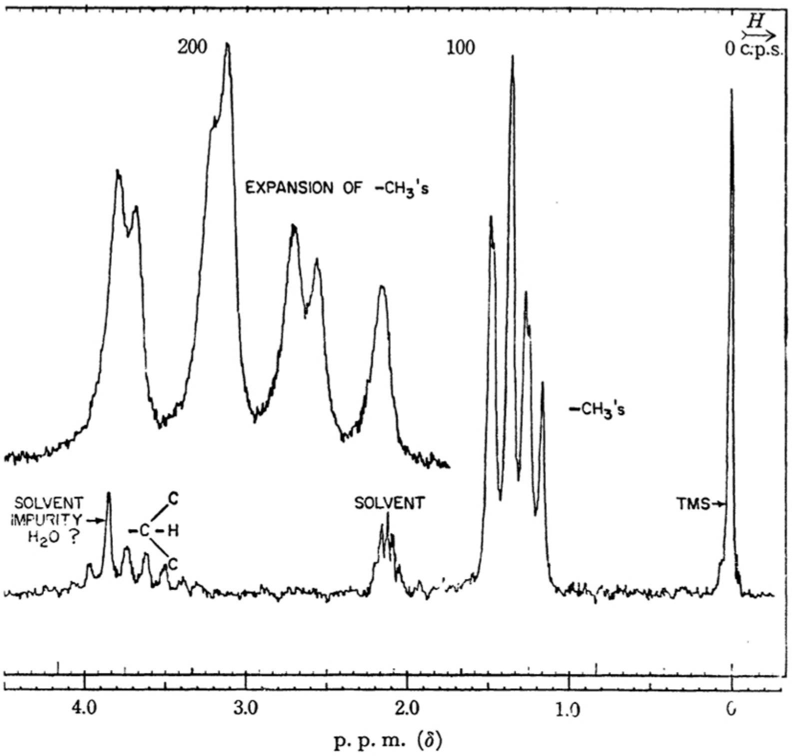

Fig. 1. PMR spectrum of I at -40°C 50 mg./ml.

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[1] S. Mizushima, "Structure of Molecules and Internal Rotation," Academic Press, New York (1954).

[2] H. S. Gutowsky and C. S. Holm, *J. Chem. Phys.*, **25**, 1228 (1956).

[3] T. H. Siddall, accepted for publication in *J. Phys. Chem.*