state steric destabilizing effect over both the *tert*-butyl (**6a**) and *tert*-heptyl (**6c**) azoalkanes.

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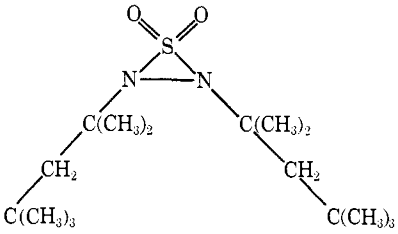
Crystal and Molecular Structure of Bis(1,1,3,3-tetramethylbutyl)thiadiaziridine 1,1-Dioxide

Sir:

We wish to report the first detailed structural information on a three-membered ring composed entirely of heteroatoms. Previously, oxadiaziridines[1,2] represented the only ring system made up solely of heteroatoms which had been isolated and characterized. A crystalline sample of a second and more stable three-membered ring system has now been prepared[3] and the structure of a substituted thiadiaziridine dioxide example (**1**) of this system is reported herein.



1

The title compound crystallizes in the orthorhombic space group $Pca2_1$, with cell dimensions: $a = 14.811 \pm 0.001$, $b = 7.951 \pm 0.001$, and $c = 17.059 \pm 0.002$ Å. A complete set of three dimensional X-ray data was taken on a G.E. XRD-490 automated diffractometer system using Cu Kα radiation and stationary crystal–stationary counter techniques out to a 2θ limit of 120° ($d = 0.89$ Å). A total of 1012 reflections (58% of the 1754 measured) were considered observed based on our statistical criteria. The data were corrected for $\alpha_1 - \alpha_2$ splitting and absorption, followed by the usual $1/LP$ corrections.

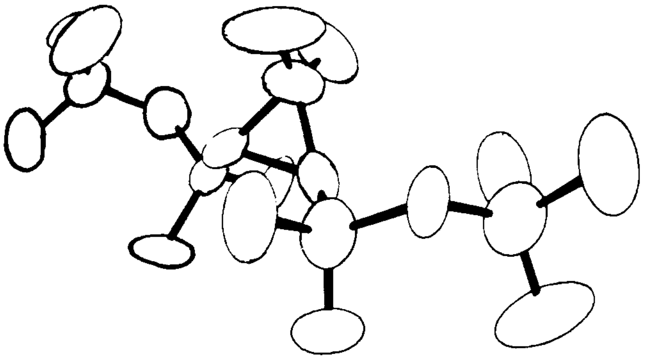


Figure 1. Projective view of bis(1,1,3,3-tetramethylbutyl)thiadiaziridine 1,1-dioxide.

The S–S vectors were identified on a Patterson map and used to phase the subsequent Fourier map (at a value of $R = 0.52$). Two additional peaks were found and assumed to be the oxygen atoms; the process was repeated ($R = 0.43$), and from that point on alternating least-squares and Fourier techniques resulted in all nonhydrogen atoms being identified ($R = 0.117$ for the final isotropic refinement). Conversion to anisotropic temperature factors followed by further least-squares refinements lowered the value of the reliability index to $R = 0.097$. The results, at this stage, are reported herein and are shown in Figure 1. Further work in locating the 34 hydrogen atoms and the final refinement of the structure are complicated by the relatively large thermal motion already evident in the nonhydrogens and the anticipated rotation of the methyl groups.

The trans configuration of the compound is unambiguously determined and substantiates the previous assumption based on chemical and nmr results. The large thermal motion of the atoms evident thus far obscures any trends in the bond distances of the two octyl side chains. The S–N average bond length of 1.62 Å is shorter than a value of 1.67 Å previously determined[4] for an analogous bond in a noncyclic system. The N–N bond (1.67 Å) is significantly longer than any analogous distance (for example, the 1.48-Å distance for the C–N bond in aziridine itself[5]) and is, in fact, bracketed between the values of 1.64 and 1.71 Å found for the admittedly lengthened[6,7] N–N bond in N_2O_4. Such lengthening may well be the only way that the NSN angle of 62° can be maintained at a value near 60°. For example, if one were to assume a "normal" N–N distance even as long as 1.48 Å and still maintain the present S–N distances, then the resultant NSN angle would be lowered to 52°. However, as a consequence of the ring maintaining this angle at the sulfur, the N–N bond is appreciably weakened. This conclusion suggests that the removal of the magnetic nonequivalence of the two methyl groups closest to the ring nitrogens in the nmr[3] may proceed through a bond breaking, inversion, and bond re-formation mechanism.

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Conformation of Olefin–Iron Carbonyl Complexes from Long-Range Proton Magnetic Resonance Couplings

Sir:

Efforts to define the nature of diene–iron carbonyl complexes based upon pmr data have brought forth