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**Die ersten N,N-disulfonylierten Thioharnstoffe: Synthese von (RSO<sub>2</sub>)<sub>2</sub>N–C(S)–NMe<sub>2</sub> (R = Me, 4-Me–C<sub>6</sub>H<sub>4</sub>) und Kristallstruktur der Ditosylverbindung**

Polysulfonylamines, CXLVII [1].

The First N,N-Disulfonylated Thioureas: Synthesis of (RSO<sub>2</sub>)<sub>2</sub>N–C(S)–NMe<sub>2</sub> (R = Me, 4-Me–C<sub>6</sub>H<sub>4</sub>) and Crystal Structure of the Ditosyl Compound

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Z. Naturforsch. **56b**, 1188–1195 (2001); eingegangen am 4. September 2001

Sulfonylthioureas, Thioamide Single Bond, Weak Hydrogen Bonding

The moisture-sensitive title compounds were obtained in good yields by treating the corresponding silver salts AgN(SO<sub>2</sub>R)<sub>2</sub> with the thiocarbamoyl chloride Me<sub>2</sub>NC(S)Cl in acetonitrile. In the NMR solution spectra of the novel thioureas, as recorded at room temperature for <sup>13</sup>C and up to 120 °C for <sup>1</sup>H, the Me<sub>2</sub>N group invariably gives rise to two distinct resonances, reflecting unusually high barriers to rotation about the C(S)–NMe<sub>2</sub> bond. The crystal structure of the ditosyl compound (triclinic, space group *P* $\bar{1}$ , X-ray diffraction at –130 °C) contains two independent molecules **A** and **B**, in which the bond lengths and angles are nearly identical, whereas the conformations exhibit pronounced discrepancies. The amide N and thiocarbonyl C atoms have trigonal-planar environments, but the S–N–S planes are strikingly rotated into approximately perpendicular orientations relative to the planar C<sub>2</sub>N–C(S)–N moieties. Other remarkable features of the molecular structures are the exceedingly long C(S)–NS<sub>2</sub> bonds [mean: 145.4(3) pm] and the concomitantly short C–S and C(S)–NC<sub>2</sub> bonds [mean values: 164.8(2), 132.6(3) pm]. The packing is governed by a three-dimensional system of weak hydrogen bonds and may be viewed as a self-clathrate, in which (**B**)<sub>2</sub> dimers constructed from C–H⋯S=C interactions are inserted as guest species into parallel tunnels between (**A**)<sub>∞</sub> tapes based upon short C–H⋯O=S contacts.