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Preliminary Communication

Crystal and Molecular Structure of (S)-α-(*p*-Chlorobenzenesulphonamido)-β-Propiothiolactone[a)]

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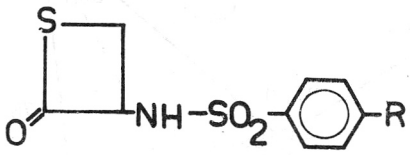
The different α-substituted-β-propiothiolactones were synthesized by Fleš *et al.*[1-3].



R = OCH_3, CH_3, H, Cl, Br, NO_2

These substances give by polymerization the optically active polythiolesters. The polymers may be of chemical and biological interest because they represent a new class of nonamide-bonded polycysteins. The proposed mechanism of polymerization[4] suggests the opening of the β-propiothiolactone ring by S_2—C_8 bond rupture. It has been found that the rate of polymerization of substrates with different substituents on the benzene ring depends on the nature of the *p*-substituents. Since these substituents are distant from the reaction centers we thought that the investigation of the crystal structure might be of some help in the elucidation of the observed dependence.

(S)-α-(*p*-chlorobenzenesulphonamido)-β-propiothiolactone is orthorhombic. The space group is $P2_12_12_1$ (from systematic absences); the unit cell dimensions are $a = 9.34$, $b = 9.95$, $c = 12.26$ Å and $Z = 4$; $D_m = 1.61$, $D_c = 1.62$ g cm^{-3}; $\mu = 63.2$ cm^{-1} (Cu Kα).

Three-dimensional intensity data were collected photometrically from integrated equi-inclination Weissenberg photographs taken around [010]. The intensities of 789 independent reflexions were measured and corrected for absorption and Lorentz polarization factors as well as for spot shape.

The structure was solved by the heavy-atom method. Considering the isotropic thermal parameters, the full-matrix least-squares refinement was carried out up to R = 13.7%. The anisotropic thermal parameters for chlorine and sulphur atoms were then introduced in the refinement as well as the anomalous scattering corrections for chlorine, sulphur and oxygen atoms[5]. The final residual error index is $R = 10.3\%$.

[a)] This work was reported at the Italo-Yugoslav Crystallographic Meeting, Trieste, June 11—14, 1973.

The values for the interatomic distances and angles in this stage of the refinement are in accordance with those reported in the literature for similar compounds.

The most interesting part of this structure is certainly the β-propiothiolactone ring (Fig. 1.) for the first time determined by X-ray investigation. The ring is puckered and the dihedral angle between the plane through the atoms C_7, C_8, S_2 and the plane through C_7, C_9, S_2 is about 13°.

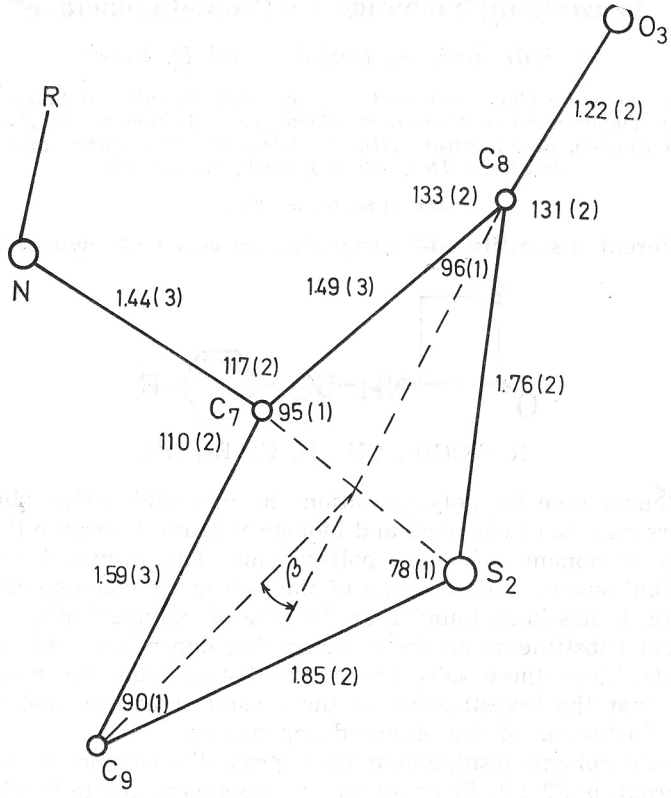


Fig. 1. Bond distances (Å) and angles (°) in β-propiothiolactone ring [R = SO_2 — (p — C_6H_4Cl)].

The intermolecular distance N—H.....O=C of 2.83 Å indicates the existence of a hydrogen bond along the b axis.

Further refinement is in progress.

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IZVOD

Kristalna i molekularna struktura (S)-α-(*p*-klorobenzensulfonamido)-β-propiotiolaktona

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Određeni su kristalografski podaci za (S)-α-(*p*-klorobenzensulfonamido)-β-propiotiolakton: $a = 9{,}34$, $b = 9{,}95$ i $c = 12{,}26$ Å. Spoju pripada prostorna grupa $P2_12_12_1$, $Z = 4$. Mjerena i računana gustoća iznose: $D_m = 1{,}61$ odnosno $D_c = 1{,}62$ g cm^{-3}. Snimanjem kristala brušenog u kuglicu ($\mu r = 1{,}24$) oko osi *b* dobiveni su podaci za rješavanje strukture, koja je određena metodom teškog atoma iz 789 mjerenih intenziteta. Utočnjavanje je provedeno metodom najmanjih kvadrata i vrijednost *R*-faktora uz anizotropne temperaturne faktore za atome S_1, S_2 i Cl iznosi 10,3%.

Dihedralni kut u β-propiotiolaktonskom prstenu je 13°, a duljina intermolekularne vodikove veze NH---O=C iznosi 2,83 Å.

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