Crystal structure of N,N-ditosylbenzo-1,5-diazocane-2,6-dione, C_{28}H_{22}N_{2}O_{6}S_{2}

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Abstract

C_{28}H_{22}N_{2}O_{6}S_{2}, monoclinic, C12/c1 (no. 15), a = 22.591(1) Å, b = 13.3972(8) Å, c = 19.874(1) Å, \( \beta = 94.018(1)^\circ \), \( V = 6000.3 \text{ Å}^3 \), Z = 8, \( R_F = 0.068 \), \( wR^2 = 0.178 \), \( T = 298 \text{ K} \).

Source of material

N,N-ditosylbenzo-1,5-diazocane-2,6-dione was prepared by reacting of p-toluenesulfonylchloride (0.01 mol) and a sodium carbonate (0.03 mol) solution (40 mL) with 2-aminobenzoic acid (0.01 mol). The pH of the mixture was adjusted to 3 after reacting 20 hours at room temperature, and then a white solid of the title compound was obtained after filtration and column chromatography separation. The purified product was dissolved in 95 % ethanol and single crystals were separated after 8 days.

Discussion

The sulfonamide family includes a broad-spectrum of synthetic bacteriostatic antibiotics used against most Gram-positive and many Gram-negative microorganisms and protozoa [1]. These compounds are commonly used in human and veterinary medicine for therapeutic and prophylactic purposes to fight common bacterial diseases [2]. Furthermore, in animal husbandry these substances are commonly used for the prevention and treatment of dairy cattle for several infectious diseases, prophylactic, or as feed additives to promote growth in farm animals [3,4].

In the crystal structure of the title compound, no significant differences appear in the bond distances and bond angles comparing to the similar compounds [5]. The C7—N2 and C8—N1 bond distance [1.388(9) Å and 1.38(1) Å] are shorter than C—N single bond of 1.443(4) Å [6]. The C2—C7 and C8—C9 bond lengths [1.48(1) Å and 1.50(1) Å] are both shorter than a typical C—C bond length [1.530(3) Å], but longer than a typical C=C double bond [1.331(2) Å] [7]. The C7—O2 and C8—O1 bond lengths [1.207(9) Å and 1.205(9) Å] are longer than that of standard C=O double bond length 1.170 Å [6]. The partial double bond character of the structure is presumed as a result of the electron delocalization. The para-substituted phenyl ring shows approximately the expected C_{2v} symmetry so that for the two matching endocyclic bond angles this holds true even at the 2o level. Therefore, it may be concluded that the angles at C16 (116.9(8)°), C23 (118.3(8)°) and C19 (121.5(8)°), C26 (120.8(7)°) are significantly diminished and enlarged, respectively. According to the conclusions of Domenicano et al. [8], it follows that the S atom introduces a small electron-withdrawing effect, whereas the methyl substituent introduces electron-releasing properties. The seven atoms of C1 to C7 and N1 are coplanar (r.m.s. deviation = 0.0241(3) Å, plane \( \pi \)). Another seven atoms of C8 to C14 and N2 are coplanar with the r.m.s. deviation of 0.0210(3) Å (plane \( p_2 \)). The sulphur atom S1 and the phenyl group C15 to C21 are coplanar (r.m.s. deviation = 0.0317(6) Å, plane \( p_3 \)). The other sulphur atom S2 and the phenyl group C22 to C28 are coplanar with the r.m.s. deviation of 0.0580(8) Å (plane \( p_4 \)). The dihedral angles formed by the plane p1 and p2, plane p3 and p4 are 76.5(2)° and 64.07°, respectively.

Table 1. Data collection and handling.

| Crystal: | colorless block, size 0.12 × 0.14 × 0.17 mm |
| Wave length: | Mo Ka radiation (0.71073 Å) |
| \( \lambda_{\text{max}} \) | 2.18 cm\(^{-1} \) |
| Diffractometer, scan mode: | Bruker APEX CCD, \( \phi/\omega \) |
| \( N(hkl)^{\text{measured}}, N(hkl)^{\text{unique}} \): | 21334, 5296 |
| Criterion for \( I_{\text{obs}} \): | \( I_{\text{obs}} > 2 \sigma(I_{\text{obs}}) \), 4870 |
| Programs: | SHELXS-97 [9], SHELXL-97 [10], SHELXTL [11], ORTEP-II [12] |