Mostafa M. Ghorab, Mansour S. Alsaid, Hazem A. Ghabbour*, Abdullah A. Al-Mishari and Obaid S. AlRuqi

Crystal structure of 4-((4,4-dimethyl-2, 6-dioxocyclohexylidine)methylamino)-N-(3,4-dimethylisoxazol-5-yl)benzenesulfonamide, C_{20}H_{23}N_{3}O_{5}S

Abstract

C_{20}H_{23}N_{3}O_{5}S, monoclinic, P2_1/c (no. 14), a = 6.7503(2) Å, b = 14.0026(7) Å, c = 21.954(1) Å, β = 96.892(3)°, V = 2060.14(15) Å^3, Z = 4, R_{int}(F) = 0.0545, wR_{ref}(F^2) = 0.1192, T = 100 K.

CCDC no.: 1468116

The asymmetric unit of the title crystal structure is shown in the figure. Tables 1 and 2 contain details of the measurement method and a list of the atoms including atomic coordinates and displacement parameters.

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Table 2 (continued)

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<th>$y$</th>
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Source of material
2-((Dimethylamino)methylene)-5,5-dimethylcyclohexane-1,3-dione (1.95 g, 0.01 mol) and 4-amino-N-(3,4-dimethylisoxazol-5-yl)benzenesulfonamide (2.67 g, 0.01 mol) were added into ethanol (20 mL) containing glacial acetic acid (5 mL). The mixture was heated under reflux for 13 h, and the solid obtained was recrystallized from ethanol to give 4-((4,4-dimethyl-2,6-dioxycyclohexylidene)methylamino)-N-(3,4-dimethylisoxazol-5-yl)benzenesulfonamide. Yield: 89%, m.p. = 245.7 °C, IR (cm⁻¹): 2332, 2185 (NH), 1647, 1650 (C=O), 1612 (CN), 3078 (arom.), 2966, 2839 (aliph.), 1377, 1157 (SO₂).

Discussion
Carbon-bound H atoms were placed in calculated positions (C—H 0.95 Å) and were included in the refinement in the riding model approximation, with $U_{eq}$(H) set to 1.2$U_{eq}$(C). The H atoms of the methyl groups were allowed to rotate with a fixed angle about the C—C bond to best fit the experimental electron density, with $U_{eq}$(H) set to 1.5$U_{eq}$(C).

Experimental details
Attributable to the extensive applicability of benzenesulfonamides, it is sought-after to acquire universal and successful methods for their production. Benzene-sulfonamides were the primarily essentially affianced and increasingly used for demand and chemotherapeutic agents hostile to an assortment of diseases [1]. Greater than thirty drugs having this functionality are in quantifiable utilize, including antiseptic [2, 3], and extra biological activities [4–8]. Particular vital benzenesulfonamide derivatives are used for instance carbonic anhydrase inhibitors of gainful magnitude [9]. A dumpy period ahead, benzenesulfonamides are registered as an anticancer proxies [10]. In persistence of our marvel in the synthesis of talented heterocyclic compounds [11–13], we testimony here a novel 4-((4,4-dimethyl-2,6-dioxycyclohexylidene)methylamino)-N-(3,4-dimethylisoxazol-5-yl)benzenesulfonamide. The ambition of this effort was to prepare of innovative 4-((4,4-dimethyl-2,6-dioxycyclohexylidene)methylamino)-N-(3,4-dimethylisoxazol-5-yl)benzenesulfonamide. Therefore, interaction of 2-((dimethylamino)methylene)-5,5-dimethylcyclohexane-1,3-dione with 4-amino-N-(3,4-dimethylisoxazol-5-yl)benzenesulfonamide furnished 4-((4,4-dimethyl-2,6-dioxycyclohexylidene)methylamino)-N-(3,4-dimethylisoxazol-5-yl)benzene-sulfonamide. The structure of this new compound is supported by microanalysis, IR, 1H-NMR, mass spectral data and X-ray analysis. In the crystal structure of title molecule, the C6–C9 bond length is 1.38(3) Å and this is typical C—C double bond. The structure was bent and the dihedral angle between the central phenyl ring (C10–C15) and the isoxazole ring (N3/C16/C17/C18/O5) is 53.75°. In the crystal structure the molecules are stabilized by two classical and three potential non-classical intermolecular hydrogen bonds, of which O1, O2 and O4 work as hydrogen bond acceptors and N1, C4, C11, C19 and C20 work as hydrogen bond donors. The distance of the interactions between N2–H1N2⋯O2, C4–H4A⋯O4i, C11–H11A⋯O1iii, C19–
H19C—O4v and C20—H20A···O2 are 2.791(3) Å, 3.345(3) Å, 3.330(3) Å, 3.489(3) Å and 3.334(3) Å, respectively. The angles are 174.5(19)°, 156°, 152°, 163° and 141°, respectively.

Symmetry codes: (i) $-x, y+1/2, -z+1/2$; (ii) $-x, y-1/2, -z+1/2$; (iii) $-x+1, -y+1, -z+1$; (iv) $-x+1, y+1/2, -z+1/2$.

Acknowledgements: The authors would like to extend their sincere appreciation to the Deanship of Scientific Research at King Saud University for funding of this research through the Research Group Project no. RGP-302.

References


