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N-[4-(3-methyl-3-mesityl-cyclobutyl)-thiazol-2-yl]-succinamic acid: X-ray structure, spectroscopic characterization and quantum chemical computational studies

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HIGHLIGHTS

- *N*-[4-(3-methyl-3-mesityl-cyclobutyl)-thiazol-2-yl]-succinamic acid (CBTSA).
- Spectroscopic and structural characterization of CBTSA.
- HF and DFT electronic structure investigation of CBTSA.
- The conformational analysis of CBTSA.

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ABSTRACT

The aim of this study is to present results of a detailed investigation of the title compound, *N*-[4-(3-methyl-3-mesityl-cyclobutyl)-thiazol-2-yl]-succinamicacid ($C_{21}H_{26}O_3N_2S$). The compound was prepared in the laboratory and crystallized in the monoclinic space group $P\bar{3}$ with $a = b = 22.4066$ (5) Å, $c = 8.0744$ (2) Å, $\gamma = 120$, and $Z = 6$. The molecule characterized by experimental methods such as ^{1}H NMR, ^{13}C NMR, IR and single-crystal X-ray diffraction. The molecular geometry, vibrational frequencies, gauge including atomic orbital (GIAO) ^{1}H and ^{13}C NMR chemical shift values of the title compound in the ground state was optimized quantum chemistry methods(Hartree–Fock (HF) and density functional method (DFT) (B3LYP) with 6-31G(d,p) basis set). In order to identify low energy conformation, molecular energy profile of the title molecule was obtained by semi-empirical quantum chemistry method (AM1) calculations with respect to a selected degrees of torsional freedom, which were varied from −180° to +180° in steps 10°. In addition to the molecular electrostatic potential (MEP), frontier molecular orbital (FMO) and Mulliken population analysis of the title compound were investigated by theoretical calculation results.

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1. Introduction

The mesitylene or 1,3,5-trimethylbenzene is commonly used as a solvent in the laboratory and has been used as a developer for photopatternable silicones due to its solvent properties in the electronics industry. Also, it plays a significant role in aerosol and tropospheric ozone formation as well as other reactions in atmospheric chemistry [1]. Cyclobutane itself is of no commercial or biological significance, but more complex derivatives are important in biology and biotechnology [2]. It is well known that 3-substituted cyclobutane carboxylic acid derivatives exhibit anti-inflammatory and anti-depressant activity [3], and also liquid crystal properties [4]. Heterocyclic compounds containing nitrogen and sulfur are used for medical purposes for the treatment of different kinds of fungal and bacterial infection along with treatment [5–7]. Thiazole derivatives exhibit different pharmaceutical properties, among them are: anticancer [8,9], anticonvulsant [10], antipsychotic-like [11], antibacterial, antifungal [12,13], antitubercular [14], antimicrobial [15], analgesic and anti-inflammatory [16] activities. Also, the thiazole ring system could be found in natural compounds like thiamine (vitamin B1) [17], bistratamide H, archazolid A and B, siomycin A, didmolamide A, scleritodermin A, etc. [18]. The title compound is a novel compound firstly synthesized in our laboratories by us.

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Investigations into the structural stability of these compounds using both experimental techniques and theoretical methods have been of interest for many years. With recent advances in computer hardware and software, it is possible to correctly describe the physico-chemical properties of molecules from first principles using various computational techniques [19]. Hartree–Fock (HF) and density functional theory (DFT) methods have been very popular for calculations in theoretical modeling. HF and DFT methods calculated a great variety of molecular properties: molecular structures, vibrational frequencies, chemical shifts, non-linear optical effects, conformational analysis, molecular electrostatic potential, frontier molecular orbitals and Mulliken atomic charges, etc.

In this present study, the title compound cyclobutane derivative, *N*-[4-(3-methyl-3-mesityl-cyclobutyl)-thiazol-2-yl]-succinamic acid, CBTSA, has been investigated both experimentally and theoretically. In experimental study, the molecule was prepared and characterized by ^{1}H NMR, ^{13}C NMR, IR and single-crystal X-ray diffraction methods. In theoretical study, the molecular geometry, vibrational frequencies, ^{1}H and ^{13}C NMR chemical shift values of the title compound in the ground state have been calculated using the Hartree–Fock (HF) and density functional method (DFT) (B3LYP) with 6-31G(d,p) basis set. The calculated geometric parameters, theoretical scaled vibrational frequencies and chemical shifts values compared with their experimental data.

2. Experimental and theoretical methods

2.1. Synthesis of the title compound

A mixture of 10 mmoL of 4-(3-methyl-3-mesityl-cyclobutyl)-thiazol-2-ylamine and 10 mmoL succinic acid anhydride in 30 mL of acetonitrile was refluxed by monitoring the reaction course with IR technique. After completion of the reaction, and cooling to room temperature, the mixture was poured into stirred water. Thus formed solid substance was separated by suction, washed with copious water and recrystallized from ethanol. White crystals. Yield: 86%. M.p.: 449 K (EtOH) (see Scheme 1).

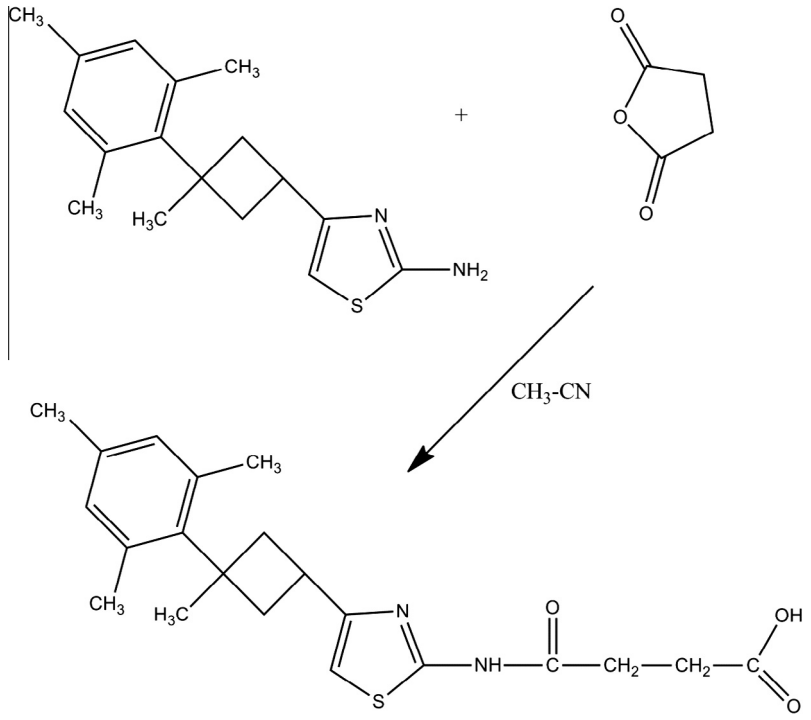
2.2. General remarks

All chemicals were of reagent grade and used as commercially purchased without further purification. Melting point was determined by Gallenkamp melting point apparatus. The IR spectrum of the title compound was recorded in the range 4000–400 cm^{-1} using a Mattson 1000 FT-IR spectrometer with KBr pellets. The ^{1}H and ^{13}C nuclear magnetic resonance (NMR) spectra were recorded on a Varian-Mercury-Plus 400 MHz spectrometer using TMS as internal standard and $CDCl_3$ (chloroform) as solvent.

2.3. X-ray diffraction data

The single-crystal X-ray data was collected on a STOE diffractometer with an IPDS(II) image plate detector. All diffraction measurements were performed at room temperature (296 K) using graphite monochromated Mo Kα radiation ($\lambda = 0.71073$ Å). Intensity data were collected in the θ range 1.8–26.2°. Reflection data was recorded in the rotation mode using the scan technique by using X-AREA software [20]. The structure was solved by direct methods using SHELXS-97 [21] implemented in the WinGX [22] program suite. The refinement was carried out by full-matrix least-squares method on the positional and anisotropic temperature parameters of the non-hydrogen atoms, or equivalently corresponding to 278 crystallographic parameters, using SHELXL-97 [23]. All H atoms were positioned geometrically and treated using a riding model, fixing the bond lengths at 0.86, 0.93, 0.97 and 0.96 Å for NH, CH, CH_2 and CH_3 atoms, respectively. The general-purpose crystallographic tool PLATON [24] was used for the structure analysis and presentation of the results. Details of the data collection conditions and the parameters of the refinement process are given in Table 1.

Crystallographic data for the structure reported in this paper have been deposited with the Cambridge Crystallographic Data



Scheme 1. Synthesis scheme of title compound.