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Crystal structure of 3-(2-methylbenzyl)thiazolidin-2-one, C₁₁H₁₃ONS

<https://doi.org/10.1515/ncrs-2019-0922>

Received December 19, 2019; accepted February 10, 2020; available online March 25, 2020

Abstract

C₁₁H₁₃ONS, monoclinic, *P*₂₁/*c* (no. 14), *a* = 8.0795(6) Å, *b* = 7.2294(5) Å, *c* = 17.5898(14) Å, β = 98.354(8)°, *V* = 1016.52(13) Å³, *Z* = 4, *R*_{gt}(*F*) = 0.0332, *wR*_{ref}(*F*₂) = 0.0825, *T* = 120.00(10) K.

CCDC no.: 1912357

The molecular structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Source of material

KO^tBu (1 mmol, 112 mg), I₂ (1 mmol, 254 mg), 2-(methylthio)-4,5-dihydrothiazole (0.5 mmol), 2-methylbenzyl bromide

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Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.16 × 0.14 × 0.12 mm
Wavelength:	Mo Kα radiation (0.71073 Å)
μ:	0.28 mm ⁻¹
Diffractometer, scan mode:	SuperNova, ω
θ _{max} , completeness:	25.0°, >99%
<i>N</i> (<i>hkl</i>) _{measured} , <i>N</i> (<i>hkl</i>) _{unique} , <i>R</i> _{int} :	6153, 1789, 0.028
Criterion for <i>I</i> _{obs} , <i>N</i> (<i>hkl</i>) _{gt} :	<i>I</i> _{obs} > 2 σ(<i>I</i> _{obs}), 1590
<i>N</i> (<i>param</i>) _{refined} :	128
Programs:	Bruker [1], SHELX [2], Olex2 [3]

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
C1	0.7100(2)	−0.0415(2)	0.30904(10)	0.0220(4)
C2	0.5656(2)	−0.1431(2)	0.28410(10)	0.0279(4)
H2	0.565878	−0.227039	0.244064	0.034*
C3	0.4219(2)	−0.1219(2)	0.31747(11)	0.0296(4)
H3	0.326884	−0.190752	0.299690	0.036*
C4	0.4197(2)	0.0013(2)	0.37710(10)	0.0253(4)
H4	0.323765	0.015410	0.400062	0.030*
C5	0.5621(2)	0.1043(2)	0.40255(10)	0.0220(4)
H5	0.560620	0.187474	0.442795	0.026*
C6	0.7072(2)	0.0857(2)	0.36904(10)	0.0194(4)
C7	0.8635(2)	0.1971(2)	0.39843(10)	0.0224(4)
H7A	0.943639	0.116698	0.428703	0.027*
H7B	0.913687	0.240417	0.354847	0.027*
C8	0.7434(2)	0.5160(2)	0.40816(10)	0.0223(4)
H8A	0.633339	0.480579	0.382710	0.027*
H8B	0.806007	0.567238	0.370055	0.027*
C9	0.7279(2)	0.6586(2)	0.47061(10)	0.0237(4)
H9A	0.622207	0.723931	0.459761	0.028*
H9B	0.818128	0.748026	0.473574	0.028*
C10	0.83208(19)	0.3356(2)	0.52149(10)	0.0206(4)
C11	0.8663(2)	−0.0710(3)	0.27289(11)	0.0297(4)
H11A	0.893804	0.041113	0.248266	0.045*
H11B	0.847516	−0.168287	0.235497	0.045*
H11C	0.957094	−0.104536	0.311924	0.045*
N1	0.83044(16)	0.35536(18)	0.44510(8)	0.0192(3)
O1	0.88735(14)	0.20338(16)	0.56024(7)	0.0275(3)
S1	0.73907(5)	0.53136(6)	0.55996(2)	0.02501(16)

(1.0 mmol) and DMC (2 mL) were introduced in a tube, equipped with magnetic stirring bar and was stirred at 80 °C. After 16 h, the conversion of the reaction was analyzed by

gas chromatography. The solvent was then evaporated under vacuum and the desired product was purified using a silica gel chromatography column and a mixture of petrol ether/ethyl acetate (4:1, v/v) as eluent. This procedure produces 3-(2-methylbenzyl)thiazolidin-2-one as a colorless solid in 86% yield (89 mg).

Experimental details

Hydrogen atoms were placed in their geometrically idealized positions and constrained to ride on their parent atoms.

Comment

Thiazolidinone derivatives are important moieties in functional materials and nature products [4, 5], such as latrun-culin obtained from the sponge *Cacospongia mycofijiensis* [6]. The convenient synthesis of thiazolidinone derivatives has attracted much attention of synthetic chemists. Recently, the synthesis of thiazolidinone derivatives have been developed by ruthenium catalyzed *O*- to *S*-alkyl migration of *N*-alkyl oxazolidine-2-thiones via Barton-McCombie pathway [7]. However, the crystal structure of 3-(2-methylbenzyl)thiazolidin-2-one has not been reported until now. Herein, we report an easy preparation for the compound 3-(2-methylbenzyl) thiazolidin-2-one, and the crystal structure of the title compound is first described. The single crystal structure verifies that all bond lengths are in normal ranges [8]. The two ring moieties are not coplanar to each other (see the figure).

Acknowledgements: We thank the Support of the National Natural Science Foundation of China (No: 21702148),

the Foundation of Department of Education of Guangdong Province (No: 2018KTSCX230, 2017KZDXM085, 2018KZDXM070 and 2018KQNCX272).

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