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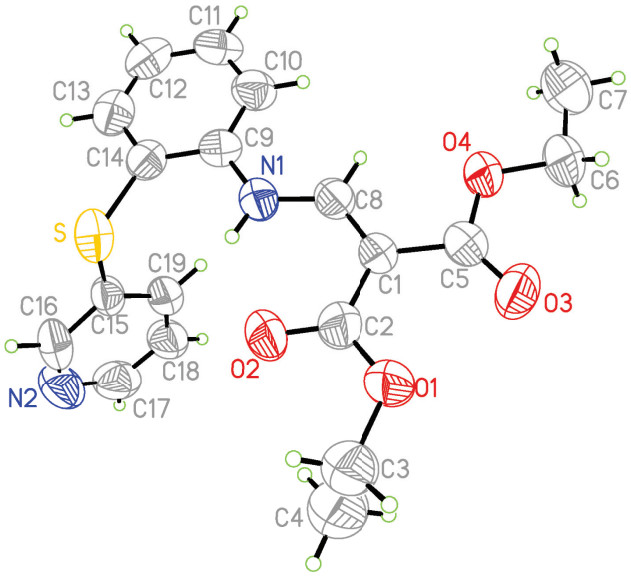
Crystal structure of diethyl 2-(((2-(pyridin-3-ylthio)phenyl)amino)methylene)malonate, $C_{19}H_{20}N_2O_4S$



DOI 10.1515/ncrs-2015-0108
Received May 28, 2015; accepted December 17, 2015; available online January 13, 2016

Abstract

$C_{19}H_{20}N_2O_4S$, monoclinic, $P2_1/c$, $a = 18.983(4)$ Å, $b = 5.057(1)$ Å, $c = 20.304(4)$ Å, $\beta = 106.10(3)°$, $V = 1872.7(7)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0680$, $wR_{ref}(F^2) = 0.1423$, $T = 293(2)$ K.

CCDC no.: 1442997

The crystal structure is shown in the figure, Tables 1–3 contain details of the measurement method and a list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Colorless, block, size 0.10×0.10×0.30 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	1.99 cm^{-1}
Diffractometer, scan mode:	Nonius CAD4, $\omega/2\theta$ scan
$2\theta_{max}$:	50.76°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	3539, 3429
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1574
$N(param)_{refined}$:	235
Programs:	SHELX [3]

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

Atom	Site	x	y	z	U_{iso}
H(1A)	4e	0.7072	0.4919	0.9161	0.071
H(3A)	4e	0.9363	1.2059	1.0024	0.108
H(3B)	4e	0.8551	1.1526	1.0047	0.108
H(4A)	4e	0.9363	1.0289	1.1061	0.174
H(4B)	4e	0.9739	0.8337	1.0663	0.174
H(4C)	4e	0.8932	0.7821	1.0687	0.174
H(6A)	4e	0.9574	0.1789	0.7881	0.105
H(6B)	4e	0.9312	0.4117	0.7356	0.105
H(7A)	4e	0.9163	0.0111	0.6779	0.148
H(7B)	4e	0.8396	0.1430	0.6695	0.148
H(7C)	4e	0.8655	−0.0889	0.7218	0.148
H(8A)	4e	0.7611	0.2674	0.8210	0.067
H(10A)	4e	0.6714	0.0193	0.7888	0.082
H(11A)	4e	0.5724	−0.2616	0.7606	0.088
H(12A)	4e	0.4840	−0.2353	0.8199	0.089
H(13A)	4e	0.4982	0.0657	0.9074	0.082
H(16A)	4e	0.5971	0.4693	1.1030	0.096
H(17A)	4e	0.7249	−0.0806	1.2015	0.111
H(18A)	4e	0.7547	−0.2075	1.1049	0.094
H(19A)	4e	0.7008	0.0042	1.0029	0.079

Source of material

The title compound was obtained from the reaction of 2-(pyridin-3-ylthio)aniline and diethyl 2-(ethoxymethylene)malonate as described in literature [1] and recrystallized from ethanol to give the desired crystals suitable for single-crystal X-ray diffraction.

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Table 3: Atomic displacement parameters (Å^2).

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
S	4*e*	0.60035(6)	0.4549(2)	0.96832(6)	0.0719(8)	0.0650(7)	0.0863(8)	0.0128(7)	0.0386(7)	0.0027(7)
N(1)	4*e*	0.7064(2)	0.3793(6)	0.8842(2)	0.057(2)	0.068(2)	0.057(2)	−0.003(2)	0.023(2)	−0.006(2)
O(1)	4*e*	0.8918(2)	0.8714(7)	0.9481(2)	0.083(2)	0.112(3)	0.084(2)	−0.036(2)	0.037(2)	−0.046(2)
C(1)	4*e*	0.8238(2)	0.5425(8)	0.8764(2)	0.051(2)	0.057(3)	0.049(2)	0.002(2)	0.016(2)	−0.007(2)
O(2)	4*e*	0.7843(2)	0.7492(6)	0.9629(2)	0.074(2)	0.116(3)	0.100(2)	−0.018(2)	0.046(2)	−0.054(2)
C(2)	4*e*	0.8311(2)	0.7276(9)	0.9318(2)	0.059(3)	0.073(3)	0.074(3)	−0.005(3)	0.023(3)	−0.016(3)
O(3)	4*e*	0.9411(2)	0.6047(7)	0.8568(2)	0.070(2)	0.143(3)	0.132(3)	−0.042(2)	0.052(2)	−0.069(3)
C(3)	4*e*	0.9014(3)	1.070(1)	1.0056(2)	0.091(4)	0.107(4)	0.076(3)	−0.020(3)	0.027(3)	−0.011(3)
O(4)	4*e*	0.8614(1)	0.3312(6)	0.7900(1)	0.060(2)	0.103(2)	0.068(2)	−0.007(2)	0.030(2)	−0.027(2)
C(4)	4*e*	0.9284(3)	0.916(1)	1.0667(2)	0.107(4)	0.151(6)	0.089(4)	−0.013(4)	0.026(3)	−0.004(4)
C(5)	4*e*	0.8824(2)	0.5048(9)	0.8421(2)	0.064(3)	0.071(3)	0.060(3)	0.000(3)	0.021(2)	−0.011(2)
C(6)	4*e*	0.9151(2)	0.257(1)	0.7557(2)	0.077(3)	0.116(4)	0.082(3)	0.005(3)	0.042(3)	−0.027(3)
C(7)	4*e*	0.8811(3)	0.064(1)	0.7015(2)	0.104(4)	0.107(4)	0.093(4)	0.010(3)	0.041(3)	−0.035(3)
C(8)	4*e*	0.7632(2)	0.3840(8)	0.8569(2)	0.057(3)	0.061(3)	0.049(2)	0.006(2)	0.016(2)	−0.001(2)
C(9)	4*e*	0.6458(2)	0.2076(8)	0.8656(2)	0.058(3)	0.055(3)	0.051(2)	−0.001(2)	0.008(2)	0.001(2)
C(10)	4*e*	0.6366(2)	0.0280(9)	0.8131(2)	0.072(3)	0.076(3)	0.058(3)	−0.016(3)	0.019(2)	−0.001(3)
C(11)	4*e*	0.5772(3)	−0.1394(9)	0.7958(2)	0.083(3)	0.081(3)	0.049(3)	−0.013(3)	0.006(2)	−0.001(2)
C(12)	4*e*	0.5247(2)	−0.1246(9)	0.8313(2)	0.058(3)	0.075(3)	0.079(3)	−0.007(3)	0.004(3)	0.014(3)
C(13)	4*e*	0.5335(2)	0.0560(9)	0.8836(2)	0.064(3)	0.069(3)	0.073(3)	0.007(3)	0.022(2)	0.020(3)
C(14)	4*e*	0.5935(2)	0.2243(8)	0.9018(2)	0.052(3)	0.061(3)	0.055(2)	−0.001(2)	0.014(2)	0.009(2)
C(15)	4*e*	0.6428(2)	0.2639(8)	1.0418(2)	0.053(3)	0.057(3)	0.062(3)	−0.005(2)	0.028(2)	−0.008(2)
C(16)	4*e*	0.6294(2)	0.3309(9)	1.1031(3)	0.079(3)	0.085(4)	0.090(4)	0.010(3)	0.050(3)	−0.008(3)
N(2)	4*e*	0.6599(3)	0.210(1)	1.1627(2)	0.111(4)	0.120(4)	0.070(3)	−0.008(3)	0.042(3)	−0.015(3)
C(17)	4*e*	0.7043(3)	0.011(1)	1.1610(2)	0.091(4)	0.117(5)	0.065(3)	−0.021(4)	0.017(3)	0.001(3)
C(18)	4*e*	0.7219(2)	−0.0694(9)	1.1032(2)	0.080(3)	0.090(4)	0.060(3)	0.018(3)	0.012(3)	0.000(3)
C(19)	4*e*	0.6903(2)	0.0572(9)	1.0430(2)	0.062(3)	0.076(3)	0.061(3)	0.009(3)	0.019(2)	−0.008(3)

A mixture of 2-(pyridin-3-ylthio)aniline (1 g, 4.95 mmol) and diethyl 2-(ethoxymethylene)malonate (1.1 g, 5.10 mmol) was heated at 130 °C for eight minutes and then allowed to cool to 100 °C. The resulting oil was dissolved in 10 mL of 2-propanol and cyclohexane . This solution was treated with charcoal, filtered and cooled to yield 1.66 g (90%) of diethyl 2-(((2-(pyridin-3-ylthio)phenyl)amino)methylene)malonate. The product can be further purified by recrystallization from ethanol.

Discussion

Diethyl 2-(((2-(pyridin-3-ylthio)phenyl)amino)methylene)malonate is a new derivative of quinolone antibacterial agents, which show broad-spectrum antibacterial activity and are widely used to treat patients with infections [2]. We report herein the synthesis and crystal structure of the title compound. In the crystal structure, intermolecular C—H$\cdots$O hydrogen bonds link the molecules into chains, in which may be effective in the stabilization of the structure.

Experimental details

In the refinement H atoms were positioned geometrically with C—H = 0.93, 0.97 and 0.98 Å for aromatic, and methyl, respectively, and constrained to ride on their parent atoms, with $U_{iso}(H) = 1.2$ (or 1.5 for methyl groups) times $U_{eq}(C)$.

Acknowledgements: We thank the National key Basic Research Program of China (973 Program) 2011CB710803 and 2012CB721104. We also thank the Program for Changjiang Scholars and Innovative Research Team in University (Grant NO. IRT1066).

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