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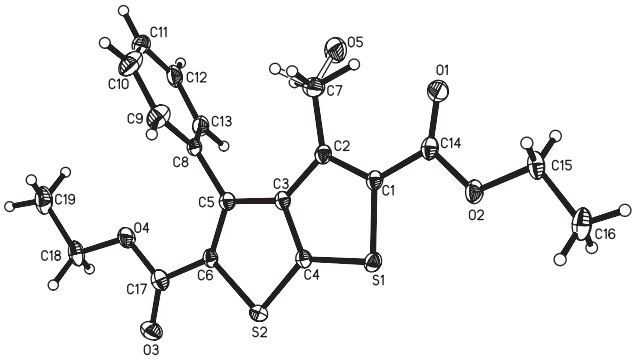
Crystal structure of diethyl-3-methyl-4-phenyl-thieno[2,3-*b*]thiophene-2,5-dicarboxylate, $C_{19}H_{18}O_4S_2$



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Abstract

$C_{19}H_{17.75}O_{4.13}S_2$, monoclinic, $P2_1/n$ (no. 14), $a = 7.1921(6)$ Å, $b = 18.6304(14)$ Å, $c = 13.3739(11)$ Å, $\beta = 92.681(4)°$, $V = 1790.0(2)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0478$, $wR_{ref}(F^2) = 0.0913$, $T = 100$ K.

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Table 1: Data collection and handling.

Crystal:	Colourless, prism, size 0.11×0.15×0.49 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	3.19 cm^{-1}
Diffractometer, scan mode:	Bruker Kappa APEXII Duo, ω + φ Scans
$2\theta_{max}$:	56.66°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	16128, 4455
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2809
$N(param)_{refined}$:	311
Programs:	SAINT program [14], SHELX [15]

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

Atom	Site	*x*	*y*	*z*	U_{iso}
H(7A)	4*e*	0.058(4)	0.823(2)	1.032(2)	0.038(8)
H(7B)	4*e*	0.275(4)	0.818(2)	1.093(2)	0.040(1)
H(7C)	4*e*	0.112(4)	0.842(2)	1.154(2)	0.037(9)
H(7D)	4*e*	0.230(3)	0.804(8)	1.060(2)	0.0370
H(9)	4*e*	0.435(3)	0.805(1)	0.864(2)	0.023(6)
H(10)	4*e*	0.377(4)	0.681(1)	0.848(2)	0.050(8)
H(11)	4*e*	0.076(3)	0.637(1)	0.815(2)	0.026(6)
H(12)	4*e*	−0.173(4)	0.717(1)	0.793(2)	0.041(8)
H(13)	4*e*	−0.123(3)	0.842(1)	0.806(2)	0.027(6)
H(15A)	4*e*	0.175(3)	1.025(1)	1.406(2)	0.028(7)
H(15B)	4*e*	0.397(3)	1.016(1)	1.401(1)	0.010(5)
H(16A)	4*e*	0.222(4)	1.151(1)	1.381(2)	0.042(8)
H(16B)	4*e*	0.336(4)	1.127(1)	1.473(2)	0.046(8)
H(16C)	4*e*	0.440(3)	1.140(1)	1.370(2)	0.037(7)
H(18A)	4*e*	0.060(3)	0.899(1)	0.501(2)	0.016(6)
H(18B)	4*e*	0.292(3)	0.899(1)	0.505(2)	0.024(6)
H(19A)	4*e*	0.065(4)	0.774(1)	0.554(2)	0.036(7)
H(19B)	4*e*	0.175(3)	0.780(1)	0.455(2)	0.047(8)
H(19C)	4*e*	0.282(4)	0.771(2)	0.562(2)	0.059(9)

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 3: Fractional atomic coordinate and displacement parameters (Å^2).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
S(1)	4*e*	0.28487(7)	1.05569(3)	1.06346(4)	0.0166(3)	0.0158(3)	0.0176(3)	−0.0015(2)	0.0007(2)	−0.0039(2)
O(1)	4*e*	0.2209(2)	0.91872(8)	1.2820(1)	0.037(1)	0.0214(8)	0.0175(9)	0.0065(7)	0.0000(7)	0.0011(7)
C(1)	4*e*	0.2400(3)	0.9737(1)	1.1213(2)	0.013(1)	0.016(1)	0.015(1)	0.0039(9)	0.0005(9)	−0.0011(9)
S(2)	4*e*	0.26054(8)	1.04872(3)	0.82988(4)	0.0195(3)	0.0155(3)	0.0174(3)	−0.0016(2)	0.0042(2)	0.0013(2)
O(2)	4*e*	0.2822(2)	1.03710(7)	1.2694(1)	0.0250(9)	0.0226(9)	0.0139(9)	0.0018(7)	−0.0011(7)	−0.0045(7)
C(2)	4*e*	0.2049(3)	0.9184(1)	1.0546(2)	0.017(1)	0.017(1)	0.015(1)	0.0052(9)	0.0004(9)	−0.0006(9)
O(3)	4*e*	0.2296(2)	1.00775(8)	0.6177(1)	0.035(1)	0.0253(9)	0.019(1)	−0.0029(8)	0.0039(7)	0.0050(7)
C(3)	4*e*	0.2152(3)	0.9432(1)	0.9536(2)	0.010(1)	0.016(1)	0.014(1)	0.0028(9)	0.0005(8)	−0.0003(9)
O(4)	4*e*	0.1773(2)	0.88993(8)	0.6422(1)	0.032(1)	0.0241(8)	0.0122(9)	−0.0044(7)	0.0027(7)	−0.0044(7)
C(4)	4*e*	0.2538(3)	1.0160(1)	0.9487(2)	0.011(1)	0.016(1)	0.013(1)	0.0002(8)	0.0007(8)	−0.0009(9)
C(5)	4*e*	0.1946(3)	0.9120(1)	0.8559(2)	0.013(1)	0.016(1)	0.015(1)	0.0016(9)	0.0003(9)	−0.0004(9)
C(6)	4*e*	0.2144(3)	0.9630(1)	0.7827(2)	0.014(1)	0.018(1)	0.012(1)	−0.0014(9)	0.0009(8)	−0.0033(9)
C(7)	4*e*	0.1545(4)	0.8428(1)	1.0831(2)	0.040(2)	0.020(1)	0.020(2)	−0.002(1)	0.001(1)	0.001(1)
O(5)	4*e*	0.029(2)	0.8132(7)	1.119(1)	0.026(6)	0.031(6)	0.036(7)	0.001(6)	0.011(6)	−0.003(6)
C(8)	4*e*	0.1615(3)	0.8337(1)	0.8387(2)	0.027(1)	0.015(1)	0.009(1)	0.001(1)	0.0020(9)	−0.0020(9)
C(9)	4*e*	0.3074(4)	0.7856(1)	0.8502(2)	0.028(2)	0.023(1)	0.026(2)	0.003(1)	−0.007(1)	−0.004(1)
C(10)	4*e*	0.2754(4)	0.7119(1)	0.8404(2)	0.043(2)	0.023(1)	0.028(2)	0.012(1)	−0.009(1)	−0.005(1)
C(11)	4*e*	0.0967(4)	0.6871(1)	0.8207(2)	0.054(2)	0.016(1)	0.018(1)	−0.002(1)	0.008(1)	−0.005(1)
C(12)	4*e*	−0.0480(4)	0.7348(1)	0.8067(2)	0.035(2)	0.026(1)	0.020(1)	−0.008(1)	0.012(1)	−0.009(1)
C(13)	4*e*	−0.0163(3)	0.8081(1)	0.8155(2)	0.024(1)	0.020(1)	0.021(1)	−0.002(1)	0.006(1)	−0.006(1)
C(14)	4*e*	0.2456(3)	0.9713(1)	1.2307(2)	0.014(1)	0.021(1)	0.016(1)	0.0049(9)	−0.0009(9)	−0.004(1)
C(15)	4*e*	0.2938(4)	1.0422(1)	1.3786(2)	0.022(1)	0.032(1)	0.013(1)	0.004(1)	−0.001(1)	−0.006(1)
C(16)	4*e*	0.3236(4)	1.1201(1)	1.4034(2)	0.028(2)	0.036(2)	0.023(2)	0.000(1)	−0.002(1)	−0.014(1)
C(17)	4*e*	0.2085(3)	0.9572(1)	0.6730(2)	0.012(1)	0.025(1)	0.020(1)	0.001(1)	0.0010(9)	−0.000(1)
C(18)	4*e*	0.1747(4)	0.8761(1)	0.5345(2)	0.027(2)	0.036(1)	0.012(1)	0.001(1)	0.002(1)	−0.006(1)
C(19)	4*e*	0.1728(4)	0.7959(2)	0.5246(2)	0.032(2)	0.040(2)	0.026(2)	−0.005(1)	0.008(1)	−0.016(1)

Source of material

The chemical reagents and solvents used in this study are commercially available and were used as purchased. The synthesis of diethyl 3-methyl-4-phenylthieno[2,3-b]thiophene-2,5-dicarboxylate was carried out according to procedure reported by Comel & Kirsch [1]. This target compound was obtained as colorless crystals (ethanol), Yield: 86%; m.p. 130 °C; ^{1}H-NMR (400 MHz, $CDCl_3$): $\delta = 1.17$ (t, 3H, CH3, J = 7.1 Hz), 1.39 (t, 3H, CH_3, J = 7.1 Hz), 2.22 (s, 3H, CH_3), 4.07 (q, 2H, CH_2, J = 7.1 Hz), 4.26 (q, 2H, CH2, J = 7.1 Hz), 7.30 (m, 2H, Ph), 7.43 ppm (m, 3H, Ph); ^{13}C-NMR (100 MHz, $CDCl_3$): $\delta = 14.4$ (CH_3), 15.9 (CH_3), 16.5 (CH_3), 63.6 ($2CH_2$), 125.2 (2 CAr), 127.1 (CAr), 128.5 (2 CAr), 131.8(CAr), 133.2 (CAr), 135.2 (CAr), 139.8 (CAr), 142.9 (CAr), 145.7 (CAr),149.0 (CAr), 161.8 (CO_2), 162.8 ppm (CO_2).

Experimental details

Data were collected on a Bruker APEX-II CCD area diffractometer. Cell refinement and data reduction were carried out by Bruker SAINT [14]. There is a disorder situation of the moiety attached on C2 (see figure). The main part of the disorder is formed by the methyl group and the minor part represents an aldehyde function. The ratio of the population factors methyl/aldehyde is 7:1.

Discussion

Thieno[2,3-*b*]thiophene scaffold represents the key pharmacophore of several pharmaceutically agents. Thus, several thieno [2,3-*b*] thiophene were reported to exhibit marked chemotherapeutic activities such as antibacterial, antiviral, antitumor, anti-glaucoma drugs, β-glucuronidase, α-glucosidase inhibition, and anti-oxidant potential [2–13]. In continues of our research program we report her crystal structure of title compound elucidated by X-ray diffraction analysis.

In the title compound, $C_{19}H_{18}O_4S_2$, the thieno [2,3-*b*] thiophene nucleus is tetra-substituted at C1, C2, C5 and C6. All bond lengths and angles are in the expected ranges.

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