Crystal structure of 5-ethyl-3-hydroxy-9-*tert*-butylthieno[3,2-*e*]-1,2,4-triazolo[2,3-*c*]pyrimidine, C₁₃H₁₆N₄OS

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Abstract

C₁₃H₁₆N₄OS, monoclinic, *P*12₁/*a*1 (No. 14), *a* = 6.286(4) Å, *b* = 19.268(5) Å, *c* = 11.060(2) Å, β = 91.84(5)°, *V* = 1338.9 Å³, *Z* = 4, *R*_{gt}(*F*) = 0.049, *wR*_{ref}(*F*) = 0.050, *T* = 296 K.

Source of material

A mixture of 100 mmol of ethylcarbазate and 100 mmol of 3-cyano-2-[(ethoxypropylene)amino]-4-*tert*-butylthiophene was heated at refluxing toluene for 6 hours in presence of catalytic amount of acetic acid. Recrystallization from dimethylsulfoxide-ethanol (1:1) afford crystals (mp 551 K) suitable for diffraction study.

Discussion

Studies on the chemistry of 3-cyano-2[(alkoxyalkylidene)amino]-thiophene have shown that these molecules can be attacked by nucleophiles [1,2]. From the X-ray diffraction experiment, the title compound was identified as 5-ethyl-3-hydroxy-9-*tert*-butylthieno[3,2-*e*]-1,2,4-triazolo[2,3-*c*]pyrimidine.

The crystal structure unambiguously shows that the molecular skeleton of heterocyclic rings is nearly planar and the molecules are linked in centrosymmetric pairs by N...H–O and N...H–C hydrogen bonds. Other intramolecular hydrogen bonds C–H...N

participate to the molecular packing cohesion. The mean geometrical characteristics of the thiophene ring are in agreement with the reported values [3].

Table 1. Data collection and handling.

Crystal:	colourless prism, size 0.15 × 0.25 × 0.70 mm
Wavelength:	Ag Kα radiation (0.5608 Å)
μ:	1.31 cm ^{−1}
Diffractometer, scan mode:	CAD4, ω/2θ
2θ _{max} :	49.94°
<i>N</i> (<i>hkl</i>) _{measured} , <i>N</i> (<i>hkl</i>) _{unique} :	5133, 4927
Criterion for <i>I</i> _{obs} , <i>N</i> (<i>hkl</i>) _{gt} :	<i>I</i> _{obs} > 3 σ(<i>I</i> _{obs}), 2132
<i>N</i> (<i>param</i>) _{refined} :	172
Programs:	teXsan [4], SIR92 [5]

Table 2. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso}
H(1)	4e	0.6512	0.0625	0.6959	0.064
H(2)	4e	0.4466	0.1319	0.7259	0.064
H(3)	4e	0.8725	0.1539	0.6293	0.095
H(4)	4e	0.8135	0.1734	0.7750	0.095
H(5)	4e	0.6959	0.2277	0.6625	0.095
H(6)	4e	0.5120	0.1892	0.0321	0.059
H(7)	4e	0.6052	0.1157	0.0957	0.059
H(8)	4e	0.5360	0.1147	−0.0359	0.059
H(9)	4e	0.3687	0.0157	0.1381	0.056
H(10)	4e	0.2493	0.0134	−0.0069	0.056
H(11)	4e	0.1156	0.0188	0.0941	0.056
H(12)	4e	0.1455	0.1175	−0.1021	0.071
H(13)	4e	−0.0461	0.1176	−0.0029	0.071
H(14)	4e	0.1093	0.1865	−0.0271	0.071
H(15)	4e	1.0469	−0.0310	0.3943	0.059
H(16)	4e	−0.0815	0.1996	0.1617	0.052

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Table 3. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	x	y	z	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
S(0)	4e	0.01815(8)	0.19948(3)	0.37551(5)	0.0331(2)	0.0453(3)	0.0625(4)	0.0106(2)	0.0041(2)	-0.0023(3)
O(0)	4e	0.9561(2)	-0.01736(8)	0.3306(1)	0.0396(7)	0.0614(9)	0.0479(8)	0.0224(7)	-0.0041(6)	0.0023(7)
N(1)	4e	0.5905(2)	0.08408(8)	0.4629(1)	0.0293(8)	0.0347(9)	0.0381(9)	0.0018(7)	-0.0023(7)	0.0025(7)
N(2)	4e	0.7726(2)	0.04377(8)	0.4757(1)	0.0291(8)	0.043(1)	0.044(1)	0.0056(7)	-0.0047(7)	0.0042(8)
N(4)	4e	0.6521(2)	0.04512(8)	0.2790(1)	0.0281(7)	0.043(1)	0.0400(9)	0.0055(7)	-0.0029(7)	-0.0010(7)
N(6)	4e	0.3167(3)	0.15198(9)	0.5310(2)	0.0422(9)	0.0411(9)	0.045(1)	0.0027(8)	0.0048(8)	-0.0016(8)
C(3)	4e	0.7981(3)	0.0232(1)	0.3631(2)	0.0276(9)	0.039(1)	0.043(1)	0.0042(8)	-0.0010(8)	0.0030(9)
C(5)	4e	0.2445(3)	0.1533(1)	0.4145(2)	0.0288(9)	0.032(1)	0.049(1)	0.0008(8)	0.0019(8)	-0.0002(9)
C(8)	4e	0.0457(3)	0.1781(1)	0.2261(2)	0.0300(9)	0.043(1)	0.058(1)	0.0040(8)	-0.0032(9)	0.003(1)
C(9)	4e	0.2170(3)	0.1376(1)	0.2027(2)	0.0278(9)	0.036(1)	0.049(1)	-0.0012(8)	-0.0050(8)	0.0007(9)
C(10)	4e	0.3344(3)	0.12220(9)	0.3143(2)	0.0228(8)	0.0308(9)	0.043(1)	-0.0017(7)	-0.0008(7)	-0.0003(8)
C(11)	4e	0.4922(3)	0.1181(1)	0.5550(2)	0.044(1)	0.039(1)	0.039(1)	0.0011(9)	0.0028(8)	0.0025(9)
C(12)	4e	0.5219(3)	0.08368(9)	0.3434(2)	0.0261(8)	0.032(1)	0.038(1)	-0.0024(7)	-0.0014(8)	0.0017(8)
C(13)	4e	0.5896(4)	0.1173(1)	0.6787(2)	0.060(1)	0.062(1)	0.040(1)	0.008(1)	0.000(1)	0.001(1)
C(14)	4e	0.7519(5)	0.1740(2)	0.6948(2)	0.096(2)	0.082(2)	0.066(2)	-0.015(2)	-0.035(2)	0.003(2)
C(15)	4e	0.2688(3)	0.1117(1)	0.0772(2)	0.0300(9)	0.047(1)	0.042(1)	0.0052(8)	-0.0060(8)	0.0012(9)
C(16)	4e	0.4910(3)	0.1363(1)	0.0446(2)	0.044(1)	0.056(1)	0.050(1)	-0.002(1)	0.0033(9)	0.008(1)
C(17)	4e	0.1080(4)	0.1391(1)	-0.0186(2)	0.050(1)	0.077(2)	0.054(1)	0.017(1)	-0.013(1)	0.003(1)
C(18)	4e	0.2579(3)	0.0323(1)	0.0749(2)	0.041(1)	0.052(1)	0.048(1)	-0.004(1)	-0.0067(9)	-0.009(1)

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