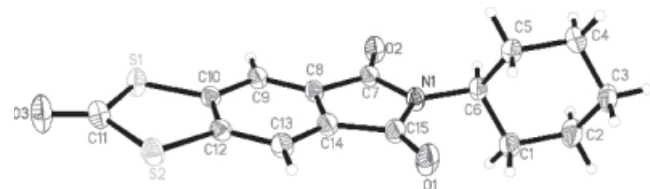


Crystal structure of 6-cyclohexyl-5*H*-[1,3]dithiolo[4,5-*f*]isoindole-2,5,7-(6*H*)-trione, C₁₅H₁₃NO₃S₂

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Abstract

C₁₅H₁₃NO₃S₂, triclinic, *P* $\bar{1}$ (no. 2), *a* = 5.5064(6) Å, *b* = 10.075(1) Å, *c* = 13.844(1) Å, α = 104.474(9)°, β = 92.046(8)°, γ = 101.598(8)°, *V* = 725.5 Å³, *Z* = 2, *R*_{gt}(*F*) = 0.0419, *wR*_{ref}(*F*²) = 0.0987, *T* = 296 K.

Table 1. Data collection and handling.

Crystal:	colourless blocks, size 0.20×0.28×0.40 mm
Wavelength:	Mo <i>K</i> _α radiation (0.71073 Å)
μ :	3.75 cm ⁻¹
Diffractometer, scan mode:	Bruker APEXII CCD, φ and ω
2 θ _{max} :	50.02°
<i>N</i> (<i>hkl</i>) _{measured} , <i>N</i> (<i>hkl</i>) _{unique} :	7498, 2560
Criterion for <i>I</i> _{obs} , <i>N</i> (<i>hkl</i>) _{gt} :	<i>I</i> _{obs} > 2 σ (<i>I</i> _{obs}), 2015
<i>N</i> (<i>param</i>) _{refined} :	190
Programs:	SHELX [6]

Source of material

A solution of 5,6-bis(benzylthio)-2-cyclohexylisoindoline-1,3-dione (0.8 g, 1.7 mmol) and AlCl₃ (1.8 g, 13.5 mmol) in dry toluene (40 mL) was stirred under nitrogen at room temperature for 2 h, then, 1,1-carbonyldiimidazole (0.54 g, 3.4 mmol) was added to the mixture and it was stirred at 373 K for an additional 6 h. The solvent was removed under reduced pressure. Chromatography of the residue on a silica gel column with ethyl acetate / petroleum ether (1:6, v:v) as eluent afforded 0.25 g (46.3%) of the title compound as a white solid [1]. Quality crystals were obtained from an ethyl acetate/petroleum ether mixture (1:3, v:v) by slow evaporation at room temperature.

Discussion

The advances in the electronic performance of organic devices in the past few years have attracted a great interest not only from the academic world but also from large international companies [2,3]. Tetrathiafulvalenes (TTF) derivatives have gone beyond the field of organic conductors and superconductors, and have been used as building blocks for molecular machines, supramolecular assemblies, electrochemical sensors, OFETs, and solar cells [4,5]. The title compound was an important intermediate in the preparation of tetrathiafulvalenes derivatives. The title compound, the title crystal structure is only built up by the C₁₅H₁₃NO₃S₂ molecules, in which all bond lengths are in normal ranges. The distances *d*(O3–C11) = 1.201(3) Å, *d*(O1–C15) = 1.205(3) Å and *d*(O2–C7) = 1.211(3) Å are in the range of a typi-

cal double bond (1.19–1.23 Å). The lengths of N1–C6, N1–C7 and N1–C15 are 1.486(3) Å, 1.392(3) Å, 1.398(3) Å, respectively, with the C7–N1–C15 angle of 111.16(19)°. The phenyl ring and five-ring group planes (S1–C11–S2–C12–C10, C7–N1–C15–C14–C8) are almost planar, with maximum dihedral angles 1.8°(3) and 2.7°(3), respectively. The torsion angles of C7–N1–C6–C1, N1–C7–C8–C14, C11–S1–C10–C9 and C10–S1–C11–O3 are -121.6(3)°, 0.5(3)°, -175.7(2)° and 176.6(3)°, respectively. The distances from S (S1, S2) to C (C10, C11, C12) are in the range of 1.74 Å to 1.78 Å, with the angle of C10–S1–C11 = 96.27(13)°, C12–S2–C11 = 96.25(12)°, S2–C11–S1 = 113.93(15)°, respectively.

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(1A)	2i	0.8371	0.5751	0.7512	0.065
H(1B)	2i	0.5551	0.5425	0.7710	0.065
H(2A)	2i	0.7889	0.5195	0.9071	0.075
H(2B)	2i	0.9551	0.6704	0.9216	0.075
H(3A)	2i	0.4469	0.6132	0.9565	0.067
H(3B)	2i	0.6678	0.6815	1.0404	0.067
H(4A)	2i	0.7557	0.8818	0.9828	0.076
H(4B)	2i	0.4755	0.8538	1.0056	0.076
H(5A)	2i	0.3478	0.7573	0.8356	0.066
H(5B)	2i	0.5150	0.9079	0.8496	0.066
H(6A)	2i	0.8570	0.8133	0.8023	0.050
H(9A)	2i	0.8084	0.9257	0.4479	0.046
H(13A)	2i	0.0495	0.6163	0.4545	0.054

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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(1)	2i	0.5079(1)	0.89182(8)	0.26423(5)	0.0431(4)	0.0665(5)	0.0406(4)	0.0044(3)	0.0015(3)	0.0252(3)
S(2)	2i	0.0226(1)	0.70323(7)	0.27097(5)	0.0405(4)	0.0579(4)	0.0411(4)	0.0032(3)	−0.0053(3)	0.0121(3)
O(1)	2i	0.2326(4)	0.5937(2)	0.6524(1)	0.059(1)	0.075(1)	0.053(1)	−0.027(1)	−0.004(1)	0.033(1)
O(2)	2i	0.9631(3)	0.9044(2)	0.6525(1)	0.039(1)	0.065(1)	0.043(1)	−0.0098(9)	−0.0015(8)	0.0204(9)
O(3)	2i	0.1269(4)	0.8318(2)	0.1294(2)	0.068(1)	0.108(2)	0.050(1)	0.004(1)	−0.012(1)	0.038(1)
N(1)	2i	0.6173(4)	0.7484(2)	0.6786(1)	0.037(1)	0.046(1)	0.031(1)	−0.0050(9)	0.0001(9)	0.0135(9)
C(1)	2i	0.7134(6)	0.6083(3)	0.7935(2)	0.068(2)	0.057(2)	0.045(2)	0.023(1)	0.014(1)	0.017(1)
C(2)	2i	0.7879(6)	0.6137(3)	0.9018(2)	0.072(2)	0.075(2)	0.053(2)	0.027(2)	0.009(2)	0.031(2)
C(3)	2i	0.6108(6)	0.6748(3)	0.9718(2)	0.068(2)	0.064(2)	0.040(2)	0.009(2)	0.008(1)	0.023(1)
C(4)	2i	0.5947(6)	0.8181(3)	0.9619(2)	0.091(2)	0.061(2)	0.036(2)	0.014(2)	0.014(2)	0.012(1)
C(5)	2i	0.5155(6)	0.8135(3)	0.8545(2)	0.079(2)	0.051(2)	0.044(2)	0.021(2)	0.014(1)	0.019(1)
C(6)	2i	0.6917(5)	0.7512(3)	0.7835(2)	0.040(1)	0.050(2)	0.032(1)	−0.001(1)	0.001(1)	0.015(1)
C(7)	2i	0.7586(4)	0.8297(2)	0.6242(2)	0.036(1)	0.040(1)	0.034(1)	−0.000(1)	0.005(1)	0.010(1)
C(8)	2i	0.6094(4)	0.8071(2)	0.5280(2)	0.034(1)	0.041(1)	0.031(1)	0.003(1)	0.004(1)	0.010(1)
C(9)	2i	0.6580(4)	0.8645(2)	0.4487(2)	0.033(1)	0.043(1)	0.035(1)	0.000(1)	0.005(1)	0.011(1)
C(10)	2i	0.4722(4)	0.8272(2)	0.3695(2)	0.038(1)	0.039(1)	0.033(1)	0.007(1)	0.006(1)	0.010(1)
C(11)	2i	0.2031(5)	0.8133(3)	0.2062(2)	0.049(2)	0.062(2)	0.039(2)	0.012(1)	0.000(1)	0.013(1)
C(12)	2i	0.2441(4)	0.7357(2)	0.3717(2)	0.036(1)	0.039(1)	0.035(1)	0.004(1)	0.001(1)	0.006(1)
C(13)	2i	0.1990(5)	0.6775(3)	0.4525(2)	0.039(1)	0.048(2)	0.041(1)	−0.007(1)	0.001(1)	0.013(1)
C(14)	2i	0.3859(4)	0.7147(2)	0.5290(2)	0.040(1)	0.042(1)	0.035(1)	−0.003(1)	0.003(1)	0.011(1)
C(15)	2i	0.3902(5)	0.6744(3)	0.6249(2)	0.046(2)	0.046(2)	0.039(1)	−0.007(1)	0.003(1)	0.014(1)

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