Crystal structure of dinaphth[2,1-*e*:1′,2′-*e*]thiepin-3-(5*H*)-thione, $(C_{10}H_6)CS_2(CH_2)(C_{10}H_6)$

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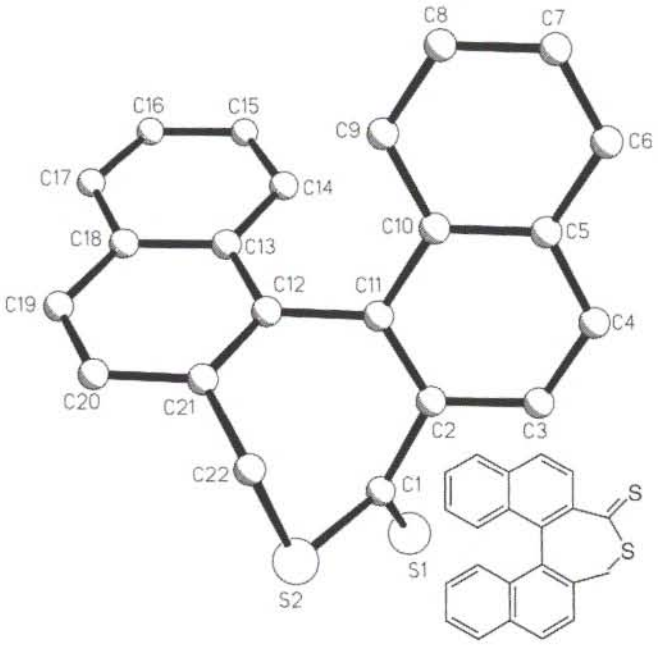

Abstract

$C_{22}H_{14}S_2$, monoclinic, $P12_1/n1$ (No. 14), a = 12.331(1) Å, b = 8.746(1) Å, c = 15.991(1) Å, β = 104.30(1)°, V = 1671.2 Å^3, Z = 4, $R_{gt}(F)$ = 0.050, $wR(F)$ = 0.049, T = 293 K.

Source of material

The racemic title compound was obtained as a two-fold sulfurated by-product during the treatment of the dioxo analog [1] with Lawesson's reagent in toluene (reflux, 5 d) in 9 % yield.

Table 1. Data collection and handling.

Crystal:	orange red plate, size 0.65 × 0.55 × 0.15 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	3.20 cm^{-1}
Diffractometer, scan mode:	Bruker AXS P4, ω
$2\theta_{max}$.	55°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	4281, 3842
Criterion for F_{obs}, $N(hkl)_{gt}$:	$F_{obs} > 3\ \sigma(F_{obs})$, 2953
$N(param)_{refined}$:	217
Program:	SHELXTL-plus [2]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(3)	4*e*	0.8880(2)	−0.2048(3)	1.0437(1)	0.08
H(4)	4*e*	0.8125(2)	−0.4358(3)	1.0685(2)	0.08
H(6)	4*e*	0.6716(3)	−0.6427(3)	1.0219(2)	0.08
H(7)	4*e*	0.5105(3)	−0.7298(3)	0.9279(2)	0.08
H(8)	4*e*	0.4141(2)	−0.5768(3)	0.8129(2)	0.08
H(9)	4*e*	0.4791(2)	−0.3340(3)	0.7926(2)	0.08
H(14)	4*e*	0.4822(2)	−0.1119(3)	0.9254(2)	0.08
H(15)	4*e*	0.3088(2)	−0.0029(3)	0.9207(2)	0.08
H(16)	4*e*	0.2184(2)	0.1486(4)	0.8031(2)	0.08
H(17)	4*e*	0.2998(2)	0.1926(3)	0.6910(2)	0.08
H(19)	4*e*	0.4571(2)	0.1488(3)	0.6204(2)	0.08
H(20)	4*e*	0.6317(2)	0.0487(3)	0.6245(2)	0.08
H(22A)	4*e*	0.7833(2)	−0.2134(3)	0.7707(2)	0.08
H(22B)	4*e*	0.7888(2)	−0.1063(3)	0.6932(2)	0.08

Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
S(1)	4*e*	0.86882(7)	0.1213(1)	0.99463(6)	0.0637(5)	0.0763(6)	0.0818(5)	−0.0255(4)	0.0225(4)	−0.0380(4)
S(2)	4*e*	0.86454(5)	0.01895(8)	0.82291(4)	0.0436(3)	0.0541(4)	0.0555(4)	−0.0127(3)	0.0146(3)	0.0034(3)
C(1)	4*e*	0.8254(2)	−0.0050(3)	0.9187(2)	0.030(1)	0.047(1)	0.049(1)	0.000(1)	0.007(1)	−0.005(1)
C(2)	4*e*	0.7664(2)	−0.1475(3)	0.9334(1)	0.037(1)	0.039(1)	0.036(1)	0.002(1)	0.0108(9)	−0.003(1)
C(3)	4*e*	0.8201(2)	−0.2399(3)	1.0051(1)	0.041(1)	0.059(2)	0.038(1)	0.009(1)	0.006(1)	−0.001(1)
C(4)	4*e*	0.7757(2)	−0.3751(3)	1.0197(2)	0.058(2)	0.058(2)	0.038(1)	0.017(1)	0.012(1)	0.013(1)
C(5)	4*e*	0.6760(2)	−0.4304(3)	0.9650(1)	0.053(1)	0.041(1)	0.041(1)	0.010(1)	0.021(1)	0.007(1)
C(6)	4*e*	0.6332(3)	−0.5784(3)	0.9752(2)	0.079(2)	0.040(1)	0.060(2)	0.013(1)	0.034(2)	0.017(1)

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Table 3. Continued.

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(7)	4*e*	0.5383(3)	–0.6298(3)	0.9196(2)	0.082(2)	0.035(1)	0.083(2)	–0.007(1)	0.043(2)	0.004(1)
C(8)	4*e*	0.4810(2)	–0.5387(3)	0.8516(2)	0.060(2)	0.039(1)	0.068(2)	–0.010(1)	0.028(1)	–0.006(1)
C(9)	4*e*	0.5193(2)	–0.3968(3)	0.8393(2)	0.045(1)	0.037(1)	0.048(1)	–0.002(1)	0.018(1)	–0.002(1)
C(10)	4*e*	0.6190(2)	–0.3378(2)	0.8951(1)	0.042(1)	0.032(1)	0.037(1)	0.0033(9)	0.018(1)	0.0016(9)
C(11)	4*e*	0.6649(2)	–0.1927(2)	0.8806(1)	0.035(1)	0.033(1)	0.031(1)	0.0021(9)	0.0110(9)	0.0002(9)
C(12)	4*e*	0.6044(2)	–0.0935(2)	0.8079(1)	0.033(1)	0.028(1)	0.036(1)	–0.0046(9)	0.0058(9)	–0.0017(9)
C(13)	4*e*	0.4979(2)	–0.0296(2)	0.8081(1)	0.035(1)	0.029(1)	0.045(1)	–0.0033(9)	0.003(1)	–0.0057(9)
C(14)	4*e*	0.4452(2)	–0.0515(3)	0.8766(2)	0.040(1)	0.046(1)	0.058(1)	–0.001(1)	0.012(1)	–0.009(1)
C(15)	4*e*	0.3440(2)	0.0133(3)	0.8742(2)	0.045(1)	0.064(2)	0.083(2)	–0.000(1)	0.020(1)	–0.023(2)
C(16)	4*e*	0.2900(2)	0.1043(4)	0.8044(2)	0.039(1)	0.080(2)	0.099(2)	0.014(2)	–0.000(2)	–0.035(2)
C(17)	4*e*	0.3375(2)	0.1292(3)	0.7382(2)	0.051(2)	0.053(2)	0.077(2)	0.014(1)	–0.018(2)	–0.013(2)
C(18)	4*e*	0.4434(2)	0.0640(3)	0.7372(2)	0.044(1)	0.034(1)	0.057(2)	0.000(1)	–0.009(1)	–0.005(1)
C(19)	4*e*	0.4954(2)	0.0892(3)	0.6692(2)	0.062(2)	0.045(1)	0.049(1)	–0.006(1)	–0.013(1)	0.014(1)
C(20)	4*e*	0.5974(2)	0.0286(3)	0.6712(2)	0.060(2)	0.050(2)	0.041(1)	–0.016(1)	0.004(1)	0.009(1)
C(21)	4*e*	0.6544(2)	–0.0616(3)	0.7418(1)	0.044(1)	0.034(1)	0.036(1)	–0.006(1)	0.008(1)	0.0013(9)
C(22)	4*e*	0.7727(2)	–0.1107(3)	0.7489(2)	0.050(1)	0.047(1)	0.041(1)	–0.006(1)	0.019(1)	0.002(1)

References

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