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1,3-Bis(2-phenylethyl)benzimidazolium-2-dithiocarboxylate

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The title compound, $C_{24}H_{22}N_2S_2$, crystallizes with two independent molecules in the asymmetric unit; these have essentially the same geometry. However, the dihedral angles between the planes of the two phenyl rings are 30.8 (1) and 21.6 (1)°, respectively, in the two molecules.

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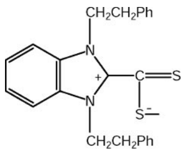
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.035
wR factor = 0.084
Data-to-parameter ratio = 16.7

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Comment

Electron-rich olefins are strongly nucleophlic and highly reactive compounds. They are also effective precursors for the preparations of variety of organic and organometallic compounds. We have used such electron-rich olefins as precursors for the synthesis of carbine–metal complexes (Çetinkaya *et al.*, 1994; Küçükbay *et al.*, 1996), acyloin catalysts (Çetinkaya & Küçükbay, 1995) and benzimidazole derivatives (Küçükbay *et al.*, 1995, 1997, 2001, 2003; Aydın *et al.*, 1998). They have an extensive organic chemistry, and particularly electron-rich olefins containing imidazolidine or benzothiazolidine moieties have long been known. However, there are a limited number of studies on electron-rich olefins containing a benzimidazolidine moiety. Electron-rich olefins will react with carbon disulfide in a molar ratio of 1:2 to yield stable dipoles (Krasuski, 1982). The objectives of the present study were to elucidate the crystal structure of the title compound, (I), and to compare it with those of related benzimidazole derivatives reported previously (İngeç *et al.* 1999; Aydın *et al.*, 1999; Öztürk *et al.*, 2001, 2003; Akkurt *et al.*, 2003).



(I)

The molecular structure of (I), shown in Fig. 1, is composed of a benzimidazole ring with two phenylethyl substituents and a dithiocarboxylate group in the 2-position. There are two crystallographically independent molecules in the asymmetric unit. Selected geometric parameters are given in Table 1. In both molecules, the C—S bonds (C16—S1/C16—S2 and C40—S3/C40—S4] are nearly equal in length. The N1—C15/N2—C15 and N3—C39/N4—C39 bond lengths in the imidazole rings agree with those reported in related structures (Öztürk *et al.*, 2003). The benzimidazole moieties are planar within experimental error. The mean of the N1—C7 and N2—C17 bond distances is 1.4708 (2) Å, in agreement with the values found in a related structure (Akkurt *et al.*, 2003). The dihedral angle between the two phenyl rings (C9–C14 and C19–C24) is 30.8 (1)°, and is 21.6 (1)° for the two phenyl rings (C33–C38

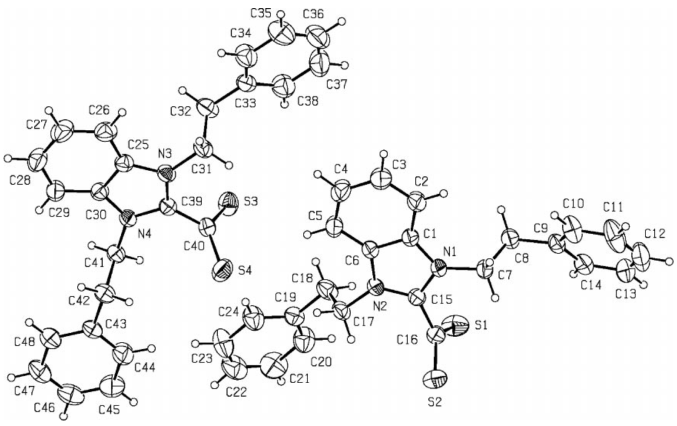


Figure 1
A view of the molecular structure of (I), with the atom-numbering scheme and displacement ellipsoids drawn at the 30% probability level.

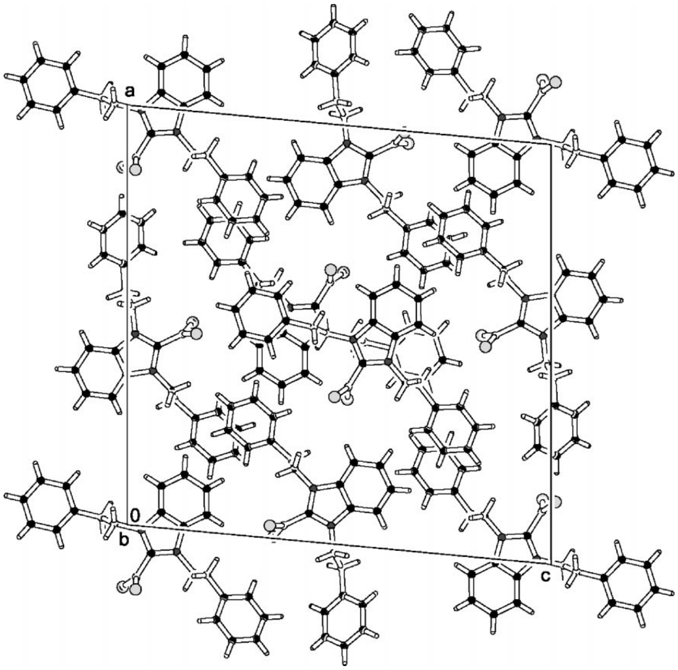


Figure 2
The crystal packing of (I), viewed along the *b* axis.

and C43–C48) in the second independent molecule. In the same sequence for both molecules, the dihedral angles between the fused benzene and imidazole rings are 0.81 (1) and 0.84 (1)°, respectively.

Experimental

All experiments were performed under argon using freshly distilled dry solvents. CS_2 (0.1 ml, 1.65 mmol) was added to a solution of bis[1,3-bis(2-phenylethyl)benzimidazolidine-2-ylidene] (0.5 g 0.77 mmol) in toluene (15 ml). A red precipitate formed instantly. The red compound was washed twice with Et_2O and crystallized from EtOH/Et_2O (yield: 0.45 g, 73%; m.p: 464–465 K). ^{1}H NMR ($CDCl_3$: δ 2.9 (*t*, CH_2CH_2Ph, 4H), 4.2 (*t*, CH_2CH_2Ph, 4H), 6.7–7.2 (*m*, Ar-H, 14H). ^{13}C NMR ($CDCl_3$): δ 35.83, 47.73, 112.70, 126.42, 127.65, 129.29, 129.40, 130.29, 137.29, 152.78, 224.60. Analysis calculated for $C_{24}H_{22}N_2S_2$: C 71.64, H 5.47, N 6.96%; found: C 71.48, H 5.43, N 6.89%.

Crystal data

$C_{24}H_{22}N_2S_2$
$M_r = 402.58$
Monoclinic, $P2_1/n$
$a = 22.1161$ (10) Å
$b = 8.7361$ (5) Å
$c = 22.3793$ (10) Å
$\beta = 95.336$ (4)°
$V = 4305.1$ (4) Å^3
$Z = 8$
$D_x = 1.242$ Mg m$^{-3$}
Mo $K\alpha$ radiation
Cell parameters from 19141 reflections
$\theta = 1.2$–25.3°
$\mu = 0.26$ mm^{-1}
$T = 293$ K
Prism, red
0.40 × 0.29 × 0.19 mm

Data collection

Stoe IPDS-II diffractometer
ω rotation scans
Absorption correction: by integration (*X-RED*32; Stoe & Cie, 2002)
$T_{\min} = 0.920$, $T_{\max} = 0.954$
60783 measured reflections
8470 independent reflections
3928 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.065$
$\theta_{\max} = 26.0°$
$h = -27 \rightarrow 27$
$k = -10 \rightarrow 10$
$l = -27 \rightarrow 27$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.035$
$wR(F^2) = 0.084$
$S = 0.82$
8470 reflections
506 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.04P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.23$ e Å^{-3}
$\Delta\rho_{\min} = -0.22$ e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.00178 (17)

Table 1
Selected geometric parameters (Å, °).

S1—C16	1.6513 (19)	N2—C6	1.396 (2)
S2—C16	1.6645 (19)	N2—C17	1.468 (2)
S3—C40	1.6535 (19)	N3—C31	1.469 (3)
S4—C40	1.6556 (19)	N3—C39	1.343 (2)
N1—C7	1.472 (2)	N3—C25	1.388 (3)
N1—C1	1.392 (2)	N4—C39	1.338 (2)
N1—C15	1.339 (2)	N4—C41	1.467 (2)
N2—C15	1.335 (3)	N4—C30	1.395 (2)
C1—N1—C7	125.69 (16)	N1—C15—C16	124.94 (18)
C1—N1—C15	108.83 (16)	N2—C15—C16	125.84 (16)
C7—N1—C15	125.41 (16)	S2—C16—C15	114.46 (13)
C6—N2—C15	108.66 (15)	S1—C16—S2	130.74 (12)
C6—N2—C17	125.34 (16)	S1—C16—C15	114.80 (13)
C15—N2—C17	126.00 (15)	N2—C17—C18	112.14 (16)
C25—N3—C39	108.82 (16)	N3—C25—C26	131.38 (19)
C31—N3—C39	125.74 (16)	N3—C25—C30	106.87 (16)
C25—N3—C31	125.03 (16)	N4—C30—C25	106.70 (17)
C30—N4—C39	108.72 (14)	N4—C30—C29	131.36 (18)
C39—N4—C41	125.61 (15)	N3—C31—C32	110.31 (16)
C30—N4—C41	125.65 (16)	N3—C39—N4	108.89 (16)
N1—C1—C6	106.59 (15)	N3—C39—C40	125.71 (17)
N1—C1—C2	131.57 (19)	N4—C39—C40	125.40 (16)
N2—C6—C1	106.70 (16)	S3—C40—S4	130.24 (13)
N2—C6—C5	131.52 (18)	S3—C40—C39	114.62 (13)
N1—C7—C8	113.47 (15)	S4—C40—C39	115.13 (13)
N1—C15—N2	109.22 (15)	N4—C41—C42	111.95 (14)

H atoms were placed geometrically and refined with a riding model, with $U_{\rm iso} = 1.2U_{\rm eq}$ of the carrier atom, and C—H = 0.93–0.97 Å.

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-RED*32 (Stoe & Cie, 2002); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*III (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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