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N-Ethyldithiophthalimide, the first structure of a dithiophthalimide derivative

Anton Meden,[a]* Miroslav Huskić[b] and Marjan Bele[b]

[a]Faculty of Chemistry and Chemical Technology, University of Ljubljana, Aškerčeva 5, SI-1000, Ljubljana, Slovenia, and [b]National Institute of Chemistry, Hajdrihova 19, Si-1000, Ljubljana, Slovenia

Correspondence e-mail: tone.meden@uni-lj.si

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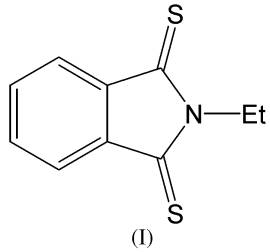
The title compound, $C_{10}H_9NS_2$, represents the first structurally studied derivative of dithiophthalimide. The C=S bond lengths are 1.629 (3) and 1.637 (3) Å; other geometric parameters are very close to the analogous values observed in the phthalimide structures. The least-square planes of the dithiophthalimide parts of the molecules in the crystal are parallel; these parts do not form any short contacts, leaving space for the ethyl groups pointing out of the plane [the 'out-of-plane torsion angles' C—N—C—C being equal to 90.7 (4) and −91.6 (4)°].

Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.045
wR factor = 0.048
Data-to-parameter ratio = 11.8

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



(I)

Experimental

A mixture of 42 mmol of phthalimide and 14 mmol of P_4S_{10} in 50 ml of xylene was heated to reflux. After 20 h, the mixture was cooled to 293 K and excess P_4S_{10} was filtered off. Xylene was removed under reduced pressure. The residue was dissolved in acetone, filtered and dried. The separation of dithiophthalimide from thiophthalimide was made by column chromatography on silica gel in hexane. Crystals of (I) were prepared by recrystallization from hot methanol.

Crystal data

$C_{10}H_9NS_2$
M_r = 207.32
Monoclinic, $P2_1/a$
a = 7.6119 (5) Å
b = 8.8460 (4) Å
c = 15.611 (1) Å
β = 101.321 (5)°
V = 1030.71 (11) Å^3
Z = 4
D_x = 1.336 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 100 reflections
θ = 10.0–16.9°
μ = 0.47 mm^{-1}
T = 293 K
Plate, brown
0.71 × 0.53 × 0.12 mm

Data collection

CAD-4 diffractometer
ω–2θ scans
Absorption correction: analytical (Alcock, 1974)
T_{min} = 0.754, T_{max} = 0.934
9710 measured reflections
2470 independent reflections
1816 reflections with $F > 2.5\sigma(F)$
R_{int} = 0.043
θ_{max} = 27.9°
$h = -10 \rightarrow 10$
$k = -11 \rightarrow 11$
$l = -20 \rightarrow 20$
3 standard reflections
frequency: 333.3 min
intensity decay: 6.3%

Refinement

Refinement on F	All H-atom parameters refined
$R = 0.045$	*REGINA* weighting scheme (Wang & Robertson, 1985), see below
$wR = 0.048$	$(\Delta/\sigma)_{max} = 0.042$
$S = 0.90$	$\Delta\rho_{max} = 0.38$ e Å^{-3}
1810 reflections	$\Delta\rho_{min} = -0.30$ e Å^{-3}
154 parameters	

Table 1
Selected geometric parameters (Å).

S1—C1	1.629 (3)	C3—C4	1.392 (3)
S2—C2	1.637 (3)	C3—C8	1.377 (3)
N—C1	1.395 (3)	C4—C5	1.386 (3)
N—C2	1.374 (4)	C5—C6	1.382 (4)
N—C9	1.471 (4)	C6—C7	1.387 (4)
C1—C4	1.457 (3)	C7—C8	1.389 (4)
C2—C3	1.462 (3)	C9—C10	1.505 (6)

H atoms were found in the difference Fourier map and were refined isotropically with restrained C—H bonds [prescribed value was 1.04 (1) Å] and C—C—H (or N—C—H) angles [prescribed values were 109.5 (5)° for ethyl and 120.0 (5)° for phthalimide H atoms]. A *REGINA* (Wang & Robertson, 1985) weighting scheme using the normal equation of the second order was applied for individual reflections so that $w = A(0,0) + A(1,0)V(F) + A(0,1)V(S) + A(2,0)V(F)^2 + A(0,2)V(S)^2 + A(1,1)V(F)V(S)$, where $V(F) = F_{obs}/F_{obs}(max)$, $F_{obs}(max) = 187.37$ and $V(S) = (\sin\theta/\lambda)/[\sin\theta/\lambda(max)]$, $\sin\theta/\lambda(max) = 0.6582$. The parameters were: $A(0,0) = 2.850259$, $A(1,0) = -0.0428708$, $A(0,1) = -5.150394$, $A(2,0) = 0.0017199$, $A(0,2) = 2.617157$ and $A(1,1) = -0.0495276$. The final geometric parameters are given in Table 1, while the molecular geometry and labeling scheme are presented in Fig. 1.

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989); cell refinement: *Xtal*3.6 *LATCON* (Hall *et al.*, 1999); data reduction: *Xtal*3.6 *DIFDAT SORTRF ADDREF*; program(s) used to solve structure: *SIR*97 (Altomare *et al.*, 1999); program(s) used to refine structure: *Xtal*3.6 *CRYLSQ*; molecular graphics: *Xtal*3.6 *ORTEP*; software used to prepare material for publication: *Xtal*3.6 *BONDLA CIFIO*.

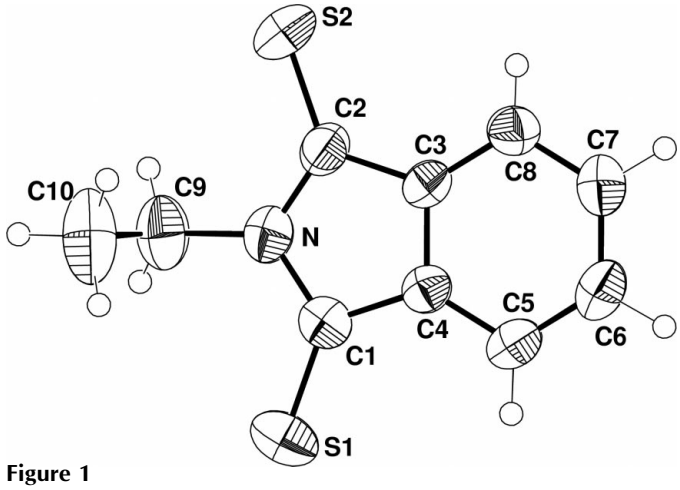


Figure 1
The molecular structure of *N*-ethyldithiophthalimide shown with 50% probability displacement ellipsoids.

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