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3-(4-Cyanophenyl)-2,2-dimethyl-1-phenylpropane-1-thione†

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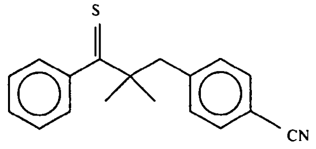
Abstract

The title molecule, $C_{18}H_{17}NS$, contains an all-*trans* arrangement of the central aliphatic system and exhibits an intramolecular S···βH contact of 2.71 Å, which is favourable for hydrogen abstraction in a photochemical reaction. The angular parameters (describing the orientation of the H atom relative to the thiocarbonyl plane) are close to those expected for reaction *via* the (*n*,π*) excited state.

Comment

Photolysis of thioketones proceeds *via* the (π,π*) excited state, with hydrogen abstraction preferably from the δ position, but with γ or ε abstraction in some derivatives (Couture *et al.*, 1981). For thioketones which have only βH atoms available, reaction appears to occur by two separate pathways, following (π,π*) or (*n*,π*) excitations, to give cyclopropylthiols as photoproducts. In our study of the molecular parameters involved in hydrogen abstraction in the photochemical reactions of thioketones (Fu *et al.*, 1997), we have determined the crystal structure of one of the compounds described by Couture *et al.* (1981), namely, 3-(4-cyanophenyl)-2,2-dimethyl-1-phenylpropane-1-thione, (1) [3-(4-cyanophenyl)thiopivalophenone, compound (7*b*), in Couture *et al.* (1981)]. This compound reacts in solution *via* βH-atom abstraction, to yield a mixture of *cis* and *trans* cyclopropylthiols.



(I)

The title molecule (Fig. 1) contains an all-*trans* arrangement of the central aliphatic chain, with both aromatic rings roughly normal to the plane of the chain [torsion angles: C2—C1—C6—C7 −102.4 (3), C2—C1—C6—C11 79.4 (4), C2—C3—C12—C13 −95.6 (4) and C2—C3—C12—C17 88.1 (4)°]. There are intramolecular S···βH (on C3) contacts of 2.71 (H atom on the right in Fig. 1) and 2.93 Å (H atom on the left). The relevant parameters in the hydrogen abstractions are *d*, ω, Δ and θ (S···βH, angular displacement of βH from the thiocarbonyl plane, C=S···H and C—H···S), with ideal values: for (π,π*), <3.0 Å, 90, <90, 180°, and

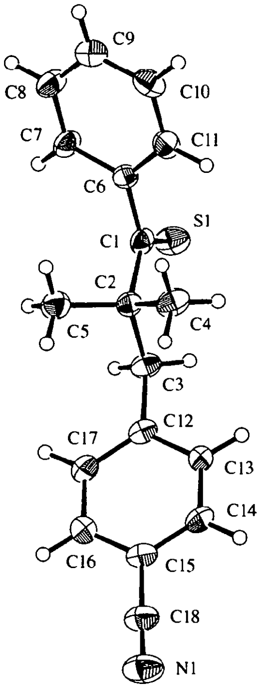


Fig. 1. View of the title molecule (33% probability displacement ellipsoids).

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† Alternative name: 4-(2-methyl-2-thiobenzoylpropyl)benzonitrile.

for (n,π^*), <3.0 Å, 0, 90, 180°. The measured values for the closest S···βH contact in compound (1) are: 2.71 Å, 13, 70, 97°. The S···H contact is shorter than the sum of van der Waals radii (3.00 Å), very favourable for hydrogen abstraction, with the ω angular parameter more favourable for abstraction *via* (n,π^*). Compounds studied previously which undergo photochemical reaction *via* (π,π^*) have average γH-abstraction parameters of about 3.04 Å, 51, 53, 122° (Fu *et al.*, 1997).

Experimental

The title compound was synthesized according to the procedure of Couture *et al.* (1981).

Crystal data

$C_{18}H_{17}NS$
$M_r = 279.40$
Orthorhombic
Pbca
$a = 23.106\ (13)$ Å
$b = 16.445\ (12)$ Å
$c = 7.985\ (7)$ Å
$V = 3034\ (7)$ Å^3
$Z = 8$
$D_x = 1.223$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.7107$ Å
Cell parameters from 24 reflections
$\theta = 5.8–13.0°$
$\mu = 0.193$ mm^{-1}
$T = 294$ K
Prism
0.50 × 0.25 × 0.20 mm
Purple

Data collection

Rigaku AFC-6*S* diffractometer
ω–2θ scans
Absorption correction: ψ scans (North *et al.*, 1968)
$T_{\min} = 0.787$, $T_{\max} = 0.962$
4429 measured reflections
4429 independent reflections

1485 reflections with $I > 3\sigma(I)$
$\theta_{\max} = 30.05°$
$h = 0 \rightarrow 32$
$k = 0 \rightarrow 23$
$l = -11 \rightarrow 0$
3 standard reflections every 200 reflections intensity decay: 3.1%

Refinement

Refinement on F^2
$R(F) = 0.063$
$wR(F^2) = 0.206$
$S = 1.225$
4429 reflections
181 parameters
H atoms not refined
$w = 1/[\sigma^2(F_o^2) + 0.00123(F_o^2)^2]$

$(\Delta/\sigma)_{\max} = 0.0002$
$\Delta\rho_{\max} = 0.38$ e Å^{-3}
$\Delta\rho_{\min} = -0.37$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

S1—C1	1.619 (3)	C15—C18	1.437 (5)
N1—C18	1.140 (4)		
S1—C1—C2	124.4 (2)	C2—C1—C6	118.1 (3)
S1—C1—C6	117.5 (2)	N1—C18—C15	178.3 (4)

H atoms were placed in calculated sites, with C—H = 0.95 Å and U(H) = 1.2U(bonded C).

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1989). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1995). Program(s) used to solve structure: *SIR*92 (Altomare *et al.*, 1993). Program(s) used to refine structure: *TEXSAN*. Software used to prepare material for publication: *TEXSAN*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: FG1388). Services for accessing these data are described at the back of the journal.

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Mephentermine Hemisulfate Monohydrate: an Adrenergic Agent

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Abstract

The title molecule, a hydrated hemisulfate salt of ethyl(2-methyl-1-phenyl-2-propyl)ammonium, $C_{11}H_{18}N^+.0.5SO_4^{2-}.H_2O$, consists of a phenethylamine skeleton in which the N atom is protonated. There are two molecules in the asymmetric unit, with the S atom of the SO_4^{2-} ion lying on a pseudo-twofold axis. The ethylamine side chain is in an extended conformation in both the symmetry-independent molecules. The distance of the N atom from the centre of the benzene ring is 5.1 Å for molecule 1 and 5.3 Å for molecule 2. The packing is stabilized by N—H···O and O—H···O hydrogen bonds.