

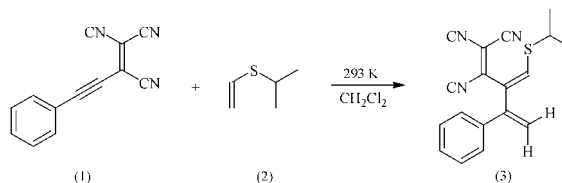
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Key indicators

Single-crystal X-ray study
 $T = 178$ K
Mean $\sigma(\text{C}-\text{C}) = 0.005$ Å
 R factor = 0.055
 wR factor = 0.161
Data-to-parameter ratio = 14.4For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.**(Z)-3-[(1-Methylethyl)thiomethylene]-4-phenyl-1,4-pentadiene-1,1,2-tricarbonitrile**In the title compound, $\text{C}_{18}\text{H}_{15}\text{N}_3\text{S}$, the limited extent of delocalization between the double-bond systems is shown by the mutual rotation of substituents about the formal single bonds $\text{NC}-\text{C}(\text{=C}-\text{S})$ and $(\text{S}-\text{C}=\text{C})-\text{C}(\text{=CH}_2)$ by 26 and 49°, respectively. Molecules are linked by four $\text{C}-\text{H}\cdots\text{N}$ and one $\text{S}\cdots\text{N}$ contact.

Comment

In our studies concerning the activating influence of the 1,1,2-tricyanovinyl group on double and triple bonds (Hopf *et al.*, 1991), we were interested in the cycloaddition of the electron-rich thioether (2) to 4-phenyl-1-buten-3-yne-1,1,2-tricarbonitrile, (1). When these two components were reacted at room temperature in dichloromethane, (Z)-3-[(1-methylethyl)thiomethylene]-4-phenyl-1,4-pentadiene-1,1,2-tricarbonitrile, (3), was produced in 68% yield (Kreutzer, 1993). Clearly [2 + 2]-cycloaddition takes place under these mild conditions, followed by ring-opening of the intermediate cyclobutene adduct.The molecule of (3) is shown in Fig. 1. Molecular dimensions (Table 1) may be considered normal. The atom sequence $\text{C4}-\text{C3}-\text{C9}-\text{S}-\text{C10}-\text{C11}$ displays an extended conformation. The extent of delocalization between the double-bond systems must be limited, as shown by the mutual rotation of substituents at $\text{C2}-\text{C3}$ and $\text{C3}-\text{C4}$ (the formal single bonds) by *ca* 26 and 49°, respectively.The molecules are connected by four $\text{C}-\text{H}\cdots\text{N}$ contacts that could be regarded as weak hydrogen bonds (Table 2); three of these involve atom N2 as acceptor. There is also a contact $\text{S}\cdots\text{N1}(1-x, -y, 1-z)$ of 3.367 (3) Å.

Experimental

The title compound was synthesized as described by Kreutzer (1993) and recrystallized by diffusion of pentane into a chloroform solution.

Crystal data

 $\text{C}_{18}\text{H}_{15}\text{N}_3\text{S}$
 $M_r = 305.39$
Monoclinic, $P2_1/c$
 $a = 10.766$ (3) Å
 $b = 8.243$ (2) Å
 $c = 18.470$ (5) Å
 $\beta = 93.37$ (2)°
 $V = 1636.3$ (7) Å³
 $Z = 4$ $D_x = 1.240$ Mg m⁻³
Mo $K\alpha$ radiation
Cell parameters from 50
reflections
 $\theta = 10-12^\circ$
 $\mu = 0.20$ mm⁻¹
 $T = 178$ (2) K
Prism, red
 $0.6 \times 0.3 \times 0.3$ mm

Data collection

Nicolet R3 diffractometer
 ω scans
 Absorption correction: none
 2987 measured reflections
 2886 independent reflections
 1898 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.015$

$\theta_{\text{max}} = 25.1^\circ$
 $h = -12 \rightarrow 12$
 $k = -9 \rightarrow 1$
 $l = -22 \rightarrow 0$
 3 standard reflections
 every 147 reflections
 intensity decay: 4%

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.055$
 $wR(F^2) = 0.161$
 $S = 1.02$
 2886 reflections
 201 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0653P)^2 + 2.0333P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.002$
 $\Delta\rho_{\text{max}} = 0.27 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.34 \text{ e } \text{\AA}^{-3}$

Table 1

Selected geometric parameters (\AA , $^\circ$).

S—C9	1.704 (3)	C2—C3	1.445 (4)
S—C10	1.827 (3)	C3—C9	1.363 (4)
C1—C2	1.372 (4)		
C9—S—C10	100.29 (16)		
C1—C2—C3—C4	−26.0 (5)	C10—S—C9—C3	163.1 (3)
C9—C3—C4—C5	−49.1 (5)	C9—S—C10—C12	−70.5 (3)
C9—C3—C4—C13	125.8 (3)	C9—S—C10—C11	167.0 (2)
C4—C3—C9—S	170.3 (2)		

Table 2

Hydrogen-bonding geometry (\AA , $^\circ$).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
C9—H9 \cdots N2 ⁱ	0.95	2.71	3.626 (5)	163
C10—H10 \cdots N2 ⁱ	1.00	2.64	3.484 (5)	142
C17—H17 \cdots N2 ⁱⁱ	0.95	2.69	3.564 (5)	154
C14—H14 \cdots N3 ⁱⁱⁱ	0.95	2.63	3.392 (5)	137

Symmetry codes: (i) $1 - x, \frac{1}{2} + y, \frac{1}{2} - z$; (ii) $2 - x, \frac{1}{2} + y, \frac{1}{2} - z$; (iii) $1 - x, 1 - y, 1 - z$.

Methyl H atoms were identified in difference syntheses, placed in idealized positions and then refined using rigid methyl groups ($C-H = 0.98 \text{ \AA}$ and $H-C-H = 109.5^\circ$) allowed to rotate but not tip.

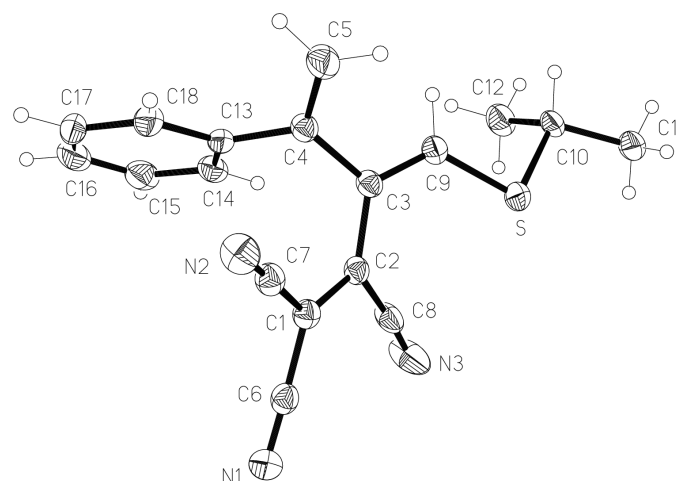


Figure 1

The molecule of compound (3) in the crystal. Ellipsoids are shown at the 30% probability level. H-atom radii are arbitrary.

Other H atoms were included using a riding model, with $C-H = 0.95$ (sp^2) or 1.00 \AA (methine).

Data collection: *P3* (Nicolet, 1987); cell refinement: *P3*; data reduction: *XDISK* (Nicolet, 1987); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* (Siemens, 1994); software used to prepare material for publication: *SHELXL97*.

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