

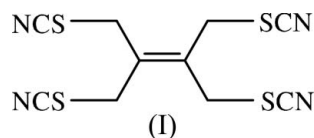
Ethene-1,1,2,2-tetrayltetramethylene
tetrathiocyanateReşat Ustabaş,^a Kemal Sancak,^b
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Key indicators

Single-crystal X-ray study
 $T = 293\text{ K}$
Mean $\sigma(\text{C}-\text{C}) = 0.003\text{ \AA}$
 R factor = 0.042
 wR factor = 0.117
Data-to-parameter ratio = 19.0For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.The title compound, $\text{C}_{10}\text{H}_8\text{N}_4\text{S}_4$, displays an approximate symmetry that closely fits the C_{2h} point group, but it crystallizes in a general position without crystallographic symmetry. The crystal structure is stabilized by two intermolecular $\text{C}-\text{H}\cdots\text{N}$ close contacts.

Comment

Many aromatic or aliphatic nitriles are close relatives of iminoesters, thioamides, carboxamidines and hydrazones. These compounds have been used as starting materials for triazole, thiazole, thiadiazole and imidazole compounds, which are important components of antimicrobial, antiviral, anticancer and antifungal agents (İkizler & Sancak, 1995, 1998; Demirbas *et al.*, 2002, 2004; Shukurov *et al.*, 1994; Klingele & Brooker, 2004). Some thiadiazole compounds with unsaturated double bonds that were synthesized from nitriles have been shown to have high anticancer activity (Chou *et al.*, 2003). Nibentan, a member of a new class of antiarrhythmic drug, was obtained from phenyl acetonitrile derivatives (Nadejda *et al.*, 2000). Moreover, phthalonitriles have been used as key reagents in the syntheses of phthalocyanines (Jin *et al.*, 1994), which are useful in the production of dyes, optical limiters and liquid crystals. Phthalocyanines are also used as catalysts for the oxidation of sulfur compounds in the petroleum industry, and as photoconductors in xerographic processes (Brewis *et al.*, 2003; Dandliker *et al.*, 1995).The molecular structure of the title compound, (I), is shown in Fig. 1. The molecule has an approximate centre of symmetry at the middle of the $\text{C}5-\text{C}6$ bond, and also approximates the other symmetry elements (*viz.* a twofold axis perpendicular to a mirror plane) that constitute the C_{2h} point group. However, since it crystallizes in a general position, the packing environment does not fit this point group, and there are some significant deviations. For example, the four torsion angles $\text{C}11-\text{S}1-\text{C}1-\text{C}5$, $\text{C}21-\text{S}2-\text{C}2-\text{C}6$, $\text{C}31-\text{S}3-\text{C}3-\text{C}5$ and $\text{C}41-\text{S}4-\text{C}4-\text{C}6$ would all be equal in magnitude if C_{2h} were the symmetry group, but instead they have the values $72.5(2)$, $-58.9(2)$, $-39.4(2)$ and $48.6(2)^\circ$, respectively. The $\text{C}11\equiv\text{N}11$ and $\text{C}41\equiv\text{N}41$ distances are $1.139(3)$ and $1.142(3)\text{ \AA}$, respectively, and thus show good agreement with corresponding distances reported previously (Çoruh *et al.*, 2003). The $\text{S}1-\text{C}11$ bond length of $1.684(3)\text{ \AA}$ lies within theReceived 26 April 2005
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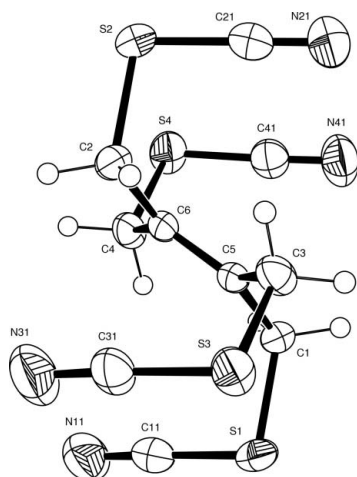


Figure 1
A view of the title compound, with the atom-numbering scheme and 50% probability displacement ellipsoids.

range previously reported for organic thiocyanates [from 1.680 (3) Å (Maartmann-Moe *et al.*, 1984) to 1.693 (2) Å (Belaj *et al.*, 1988)]. The S2–C21–N21 angle is linear at 179.7 (3)°.

The molecules of (I) are linked by C–H···N hydrogen bonds (Table 1) which are roughly parallel to the *a* axis of the unit cell (Fig. 2).

Experimental

A solution of 1,4-dibromo-2,3-bis(bromomethyl)but-2-ene (3.99 g, 0.01 mol) in absolute ethanol (200 ml) with powdered potassium thiocyanate (3.87 g, 0.04 mol) was allowed to warm to ambient temperature and stirred for 8 h. After cooling, the reaction mixture was filtered, the filtrate was removed under reduced pressure and the residue was dried over CaCl₂. The solid residue was crystallized from acetone–petroleum ether (1:1) to give (I) (yield 1.022 g, 87.17%; m.p. 429–430 K. Spectroscopic analysis: IR (KBr, ν , cm⁻¹): CH 2960, C≡N 2147; C=C 1598; CH₂ (def.) 1451, 1426; ¹H NMR (DMSO-*d*₆, δ , p.p.m.): 3.44 (s, 8H, 4CH₂); ¹³C NMR (DMSO-*d*₆, δ , p.p.m.): 20.18 (4S-CH₂), 113.08 (4S-C≡N), 126.02 (C=C).

Crystal data

C ₁₀ H ₈ N ₄ S ₄	$D_x = 1.544 \text{ Mg m}^{-3}$
$M_r = 312.44$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 7754 reflections
$a = 11.5149 (14) \text{ \AA}$	$\theta = 1.9\text{--}28.0^\circ$
$b = 8.6821 (10) \text{ \AA}$	$\mu = 0.69 \text{ mm}^{-1}$
$c = 14.1549 (17) \text{ \AA}$	$T = 293 (2) \text{ K}$
$\beta = 108.200 (2)^\circ$	Prism, yellow
$V = 1344.3 (3) \text{ \AA}^3$	$0.30 \times 0.27 \times 0.26 \text{ mm}$
$Z = 4$	

Data collection

Bruker SMART CCD area-detector diffractometer	3100 independent reflections
φ and ω scans	2390 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (DENZO-SMN; Otwinowski & Minor, 1997)	$R_{\text{int}} = 0.017$
$T_{\text{min}} = 0.809$, $T_{\text{max}} = 0.835$	$\theta_{\text{max}} = 28.0^\circ$
7754 measured reflections	$h = -14 \rightarrow 13$
	$k = -5 \rightarrow 11$
	$l = -18 \rightarrow 18$

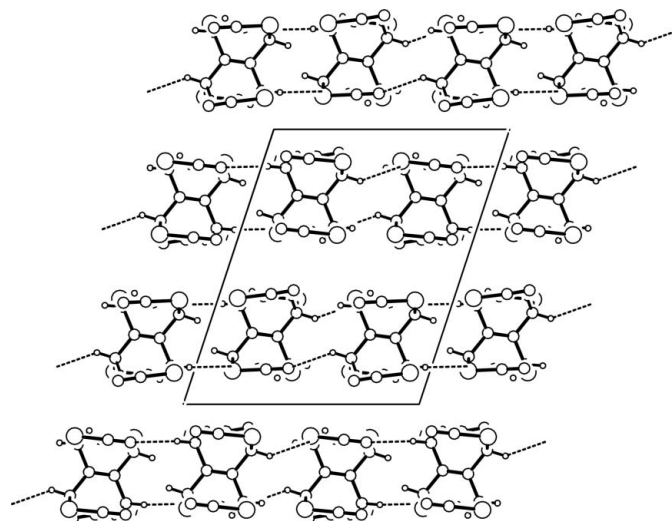


Figure 2
Part of the crystal structure of (I), viewed down the *b* axis. The dashed lines indicate C–H···N hydrogen bonds linking the molecules in the horizontal direction, which is the direction of the *a* axis of the unit cell.

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0477P)^2 + 0.9296P]$
$R[F^2 > 2\sigma(F^2)] = 0.042$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.117$	$(\Delta/\sigma)_{\text{max}} = 0.001$
$S = 1.08$	$\Delta\rho_{\text{max}} = 0.35 \text{ e \AA}^{-3}$
3100 reflections	$\Delta\rho_{\text{min}} = -0.30 \text{ e \AA}^{-3}$
163 parameters	
H-atom parameters constrained	

Table 1
Hydrogen-bond geometry (Å, °).

$D\text{--}H\cdots A$	$D\text{--}H$	$H\cdots A$	$D\cdots A$	$D\text{--}H\cdots A$
C1–H1A···N21 ⁱ	0.97	2.56	3.251 (4)	129
C4–H4A···N31 ⁱⁱ	0.97	2.61	3.257 (4)	125

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

All H atoms were fixed. C–H distances are in the range 0.92 (2)–1.04 (3) Å and $U_{\text{iso}}(\text{H})$ values are in the range 0.44 (5)–0.88 (10) Å².

Data collection: SMART (Bruker, 1997); cell refinement: SAINT (Bruker, 1997); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997a); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997a); molecular graphics: SHELXTL (Sheldrick, 1997b); software used to prepare material for publication: SHELXTL.

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