lengths and angles that differ by up to ~0.06 Å [for C(1)—C(2)] and ~4° [for C(1)—C(2)—C(3) and C(2)—C(3)—C(4)] (Table 1). These discrepancies may be explained by the fact that Roychowdhury obtained the reflections photographically and estimated their intensities visually.

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Structure of 1,3,6,8-Tetrahydrodithieno[3,4-*a*:3′,4′-*c*]benzene

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Abstract. 1,3,6,8-Tetrahydrothieno[3,4-*e*][2]benzothiophene, $C_{10}H_{10}S_2$, $M_r = 194.32$, tetragonal, $I4_1/a$, $a = 22.386$ (4), $c = 13.883$ (7) Å, $V = 6957$ (4) Å^3, $Z = 32$, two independent molecules, $D_x = 1.482$ Mg m^{-3}, λ(Mo $K\alpha$) = 0.71069 Å, $\mu = 0.52$ mm^{-1}, $F(000) = 3264$, $T = 120$ K, final $R = 0.042$ for 1567 observed reflections. The central ring of the title compound has an almost unchanged benzene geometry and for the annelated 2,5-dihydrothiophene rings typical values of C—S and C—C single and C═C double bonds were observed. While the carbon skeleton of the molecule is planar, the S atoms show deviations of 0.142 [S(1)], 0.082 [S(4)], 0.072 [S(21)] and 0.289 Å [S(24)] from the mean plane. The crystal structure is stabilized by intermolecular S···H hydrogen bonds with S···H distances of 2.90 ± 0.06 Å (mean value) and C—H···S angles of 146 ± 10° (mean value); S···S distances of 3.64 Å were observed also.

Introduction. In cycloalkene-annelated benzenes, ring contraction may introduce strain into the aromatic ring and this could lead to double-bond fixation. The effect of bond fixation in a benzene ring towards one of the Kekulé structures was first proposed by Mills & Nixon (1930) and is known as the Mills–Nixon effect. Much effort was undertaken to prove the existence or non-existence of this effect (Baldridge & Siegel, 1992; Faust, Glendening, Streitwieser & Vollhardt, 1992; Stanger, 1991). To our knowledge little is known about systematic changes of the benzene geometry caused by the annelation of heterocycloalkenes. Here we report the structure of 1,3,6,8-tetrahydrodithieno[3,4-*a*:3′,4′-*c*]benzene, where two 2,5-dihydrothiophenes are annelated to a benzene moiety. The main aim of this study was to investigate the possibility of a heteroatom Mills–Nixon effect.

Experimental. 1,3,6,8-Tetrahydrodithieno[3,4-*a*:3′,4′-*c*]benzene was synthesized from 1,2,3,4-tetramethylbenzene (prehnitene) as described in the literature (Giovannini & Vuilleumier, 1977). Crystal size: 0.10 × 0.12 × 0.25 mm, Nicolet *R*3*m*/*V* four-circle diffractometer, Wyckoff scan, scan width 0.8°, lattice parameters determined with 50 reflections ($15 \leq 2\theta \leq 25°$), no absorption correction applied, maximum value of $(\sin\theta)/\lambda = 0.54$ Å^{-1}, *hkl* range $-24 \leq h \leq 24$, $-13 \leq k \leq 18$, $-18 \leq l \leq 7$, two standard reflections ($hkl = 224$ and $0\bar{4}0$) with no significant variation during the experiment, 7130 measured intensities, $R_{\rm int} = 0.039$, 2143 unique reflections, 846 unobserved reflections [$F_o \geq 4\sigma(F_o)$], structure solved by direct methods and full-matrix refinement on F with *SHELXTL-Plus* (Sheldrick, 1988). H atoms located from difference Fourier synthesis and refined

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Table 1. *Atomic coordinates* ($\times 10^4$) *and equivalent isotropic thermal parameters* (Å$^2 \times 10^4$) *with e.s.d.'s in parentheses*

U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
S(1)	2429 (1)	4067 (1)	1229 (1)	359 (5)
C(2)	2913 (2)	3421 (2)	1101 (3)	272 (17)
C(3)	3772 (2)	2392 (2)	1588 (3)	316 (18)
S(4)	4218 (1)	1860 (1)	2253 (1)	442 (5)
C(5)	3902 (2)	2035 (2)	3422 (3)	311 (17)
C(6)	3147 (2)	2817 (2)	3991 (4)	270 (17)
C(7)	2769 (2)	3282 (2)	3770 (4)	278 (17)
C(8)	2306 (2)	3967 (2)	2515 (3)	325 (18)
C(9)	3024 (2)	3194 (2)	2098 (3)	244 (16)
C(10)	3408 (2)	2722 (2)	2316 (3)	249 (17)
C(11)	3470 (2)	2539 (2)	3259 (4)	250 (16)
C(12)	2706 (2)	3472 (2)	2822 (3)	244 (16)
S(21)	6117 (1)	7275 (1)	1087 (1)	330 (5)
C(22)	6776 (2)	7749 (2)	1078 (3)	286 (17)
C(23)	7757 (2)	8627 (2)	1703 (3)	280 (16)
S(24)	8131 (1)	9215 (1)	2377 (1)	380 (5)
C(25)	7811 (2)	9007 (2)	3531 (3)	293 (17)
C(26)	6881 (2)	8397 (2)	3961 (3)	262 (16)
C(27)	6442 (2)	8000 (2)	3684 (3)	254 (16)
C(28)	5976 (2)	7355 (2)	2364 (3)	279 (17)
C(29)	6840 (2)	7988 (2)	2072 (3)	227 (16)
C(30)	7287 (2)	8392 (2)	2359 (3)	222 (16)
C(31)	7306 (2)	8588 (2)	3301 (3)	230 (16)
C(32)	6427 (2)	7792 (2)	2737 (3)	231 (16)

as rigid groups, 227 parameters refined, $R = 0.042$ and $wR = 0.037$ with $w^{-1} = [\sigma^2(F_o) + 0.000117F_o^2]$, $S = 1.347$, $(\Delta/\sigma)_{\max} = 0.008$, maximum and minimum electron densities based on final Fourier synthesis, $(\Delta\rho)_{\max} = 0.25$ and $(\Delta\rho)_{\min} = -0.25$ e Å^{-3}, no extinction correction applied, atomic scattering factors as incorporated in *SHELXTL-Plus* (Sheldrick, 1988) and from Cromer & Mann (1968), f',f'' values from Cromer & Liberman (1970).

Discussion. Final atomic coordinates and equivalent isotropic thermal parameters are listed in Table 1.* Selected bond distances and angles are given in Table 2. The numbering scheme and the geometry of the two independent molecules are shown in Fig. 1. In the title compound, two 2,5-dihydrothiophene units are connected *via* a benzene unit. Therefore, the influence of the annelation on these structural subunits can be discussed.

In the crystal the two independent molecules have the same geometry within the standard deviations. The central benzene ring reveals an essentially unchanged benzene geometry (Bacon, Curry & Wilson, 1964). The C(9)—C(10) and C(29)—C(30) bonds are slightly elongated (up to 0.02 Å). This effect is typical for angular annelated polycyclic compounds like phenanthrene (Kay, Okaya & Cox, 1971) or phenanthridine (Brett, Rademacher & Boese, 1993), but it is less pronounced and within 3σ in the title compound. In the annelated 2,5-dihydrothiophene rings the bond lengths are in the normal range of C(sp^3)—S and C(sp^3)—C(sp^2) single and C(sp^2)=C(sp^2) double bonds (Rademacher, 1987), indicating that the annelation has no distinct effect on this part of the molecule. The carbon skeleton of the title compound was found to be planar with a mean deviation from the mean plane of ~ 0.007 Å for molecule (I) and ~ 0.013 Å for molecule (II). The S atoms deviate from these mean planes by 0.142 [S(1)], 0.082 [S(4)], 0.072 [S(21)] and 0.289 Å [S(24)]. The data reveal no indication of a hetero-atom Mills–Nixon effect.

Table 2. *Selected bond lengths* (Å), *bond angles* (°) *and torsion angles* (°) *with e.s.d.'s in parentheses*

Molecule (I)		Molecule (II)	
S(1)—C(2)	1.815 (4)	S(21)—C(22)	1.818 (4)
S(1)—C(8)	1.819 (5)	S(21)—C(28)	1.810 (5)
C(2)—C(9)	1.496 (6)	C(22)—C(29)	1.487 (6)
C(3)—C(10)	1.494 (6)	C(23)—C(30)	1.486 (6)
S(4)—C(3)	1.808 (4)	S(24)—C(23)	1.819 (4)
S(4)—C(5)	1.813 (5)	S(24)—C(25)	1.816 (5)
C(5)—C(11)	1.503 (5)	C(25)—C(31)	1.503 (5)
C(6)—C(7)	1.375 (5)	C(26)—C(27)	1.379 (5)
C(6)—C(11)	1.395 (6)	C(26)—C(31)	1.389 (6)
C(7)—C(12)	1.390 (7)	C(27)—C(32)	1.396 (7)
C(8)—C(12)	1.488 (6)	C(28)—C(32)	1.496 (5)
C(9)—C(10)	1.394 (5)	C(29)—C(30)	1.407 (5)
C(9)—C(12)	1.380 (6)	C(29)—C(32)	1.379 (6)
C(10)—C(11)	1.379 (7)	C(30)—C(31)	1.381 (7)
H(8*B*)···S(1*A*)	2.94	H(23*B*)···S(24*B*)	2.91
H(25*A*)···S(1*B*)	2.94	S(21)···S(21*A*)	3.64
H(26)···S(1*C*)	2.81	S(21)···S(21*B*)	3.64
H(27)···S(4*A*)	2.93	H(7)···S(24*A*)	2.86
S(1)—C(2)—C(9)	106.2 (3)	S(21)—C(22)—C(29)	106.3 (3)
S(1)—C(8)—C(12)	106.3 (3)	S(21)—C(28)—C(32)	106.6 (3)
C(2)—S(1)—C(8)	95.1 (2)	C(22)—S(21)—C(28)	95.2 (2)
C(2)—C(9)—C(12)	115.8 (3)	C(22)—C(29)—C(32)	116.2 (3)
C(3)—S(4)—C(5)	95.7 (2)	C(23)—S(24)—C(25)	95.0 (2)
C(3)—C(10)—C(11)	116.2 (3)	C(23)—C(30)—C(31)	116.5 (3)
S(4)—C(3)—C(10)	106.3 (3)	S(24)—C(23)—C(30)	105.4 (3)
S(4)—C(5)—C(11)	106.2 (3)	S(24)—C(25)—C(31)	105.6 (3)
C(5)—C(11)—C(10)	115.5 (4)	C(25)—C(31)—C(30)	115.0 (4)
C(6)—C(11)—C(10)	120.5 (4)	C(26)—C(31)—C(30)	120.4 (4)
C(7)—C(6)—C(11)	119.6 (4)	C(27)—C(26)—C(31)	120.2 (4)
C(8)—C(12)—C(9)	116.0 (4)	C(28)—C(32)—C(29)	115.4 (4)
C(9)—C(10)—C(11)	119.5 (4)	C(29)—C(30)—C(31)	119.6 (4)
C(8)—H(8*B*)···S(1*A*)	139.5	C(7)—H(7)···S(24*A*)	150.5
C(25)—H(25*A*)···S(1*B*)	134.7	C(23)—H(23*B*)···S(24*B*)	159.5
C(26)—H(26)···S(1*C*)	155.2		
C(27)—H(27)···S(4*A*)	137.3		
S(1)—C(2)—C(9)—C(10)	−175.7	S(21)—C(22)—C(29)—C(30)	−177.8
S(1)—C(8)—C(12)—C(7)	176.1	S(21)—C(28)—C(32)—C(27)	176.0
C(2)—C(9)—C(10)—C(3)	1.8	C(22)—C(29)—C(30)—C(23)	−0.6
C(2)—C(9)—C(12)—C(7)	178.5	C(22)—C(29)—C(32)—C(27)	−178.7
S(4)—C(5)—C(11)—C(6)	−177.9	S(24)—C(25)—C(31)—C(26)	−169.2
S(4)—C(5)—C(10)—C(9)	176.8	S(24)—C(25)—C(30)—C(29)	170.8
C(5)—C(11)—C(6)—C(7)	178.9	C(25)—C(31)—C(26)—C(27)	−178.5
C(5)—C(11)—C(10)—C(9)	−179.0	C(25)—C(31)—C(30)—C(29)	178.5
C(6)—C(7)—C(12)—C(8)	179.6	C(26)—C(27)—C(32)—C(28)	179.1
C(6)—C(7)—C(12)—C(9)	−0.1	C(26)—C(27)—C(32)—C(29)	−1.3
C(7)—C(12)—C(9)—C(10)	0.1	C(27)—C(32)—C(29)—C(30)	1.3
C(11)—C(6)—C(7)—C(12)	0.4	C(31)—C(26)—C(27)—C(32)	0.1

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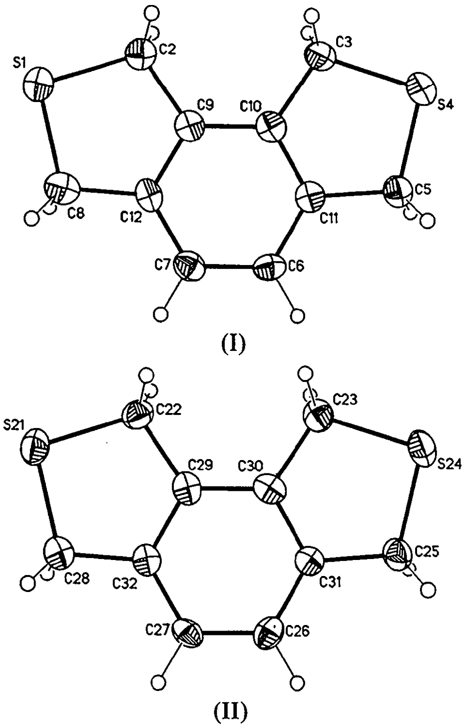
* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and a complete list of bond lengths and angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71148 (15 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: SE1016]



(I)

(II)

Fig. 1. Perspective view of molecules (I) and (II) showing the atomic labelling. Ellipsoids are drawn to include 50% probability for non-H atoms.

The packing is determined by the formation of intermolecular C—H···S hydrogen bonds with H···S distances of 2.90 ± 0.06 Å (mean value) and C—H···S angles of 146 ± 10° (mean value). Furthermore, intermolecular S···S distances of 3.64 Å, shorter than the sum of the van der Waals radii, were observed.

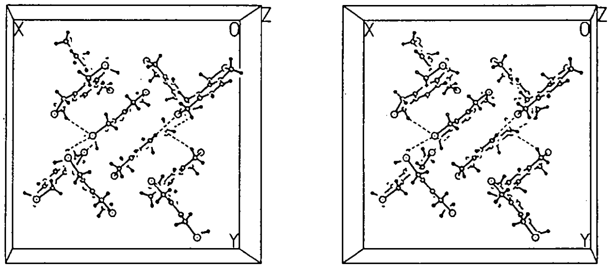


Fig. 2. Stereoview showing the crystal packing along the *z* axis.

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Structure of the Product from a Novel Cyclization Reaction Involving a C(6)-Substituted Uridine Analog

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Abstract. 5,6,7,7a,9,10,14b,14c-Octahydro-4-[2,3-*O*-(1-methylethylidene)-β-D-ribofuranosyl]cyclopenta[4,5]pyrimido[5′,4′:3,4]pyrrolo[2,1-*a*]isoquinoline-1,3(2*H*,4*H*)-dione, $C_{25}H_{31}N_3O_6$, $M_r = 469.54$, tetragonal, $P4_32_12$, $a = 12.577$ (2), $b = 12.577$ (2), $c = 29.893$ (4) Å, $V = 4729$ (1) Å^3, $Z = 8$, $D_x = 1.319$ g cm^{-3}, λ(Cu $K\alpha$) = 1.5418 Å, $\mu = 7.9$ cm^{-1}, $F(000) = 2000$, $T = 298$ K, final $R = 0.038$, $wR =$

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