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2-[2,6-Bis(4-methoxyphenyl)tetrahydrothiopyran-4-ylidene]malononitrile

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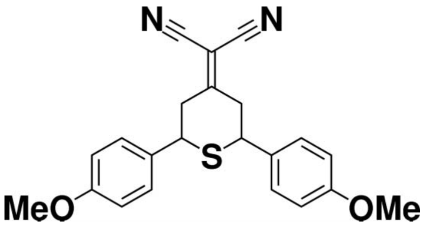
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.008 Å; R factor = 0.055; wR factor = 0.193; data-to-parameter ratio = 8.7.

In the title molecule, $C_{22}H_{20}N_2O_2S$, the tetrahydrothiopyran ring adopts a chair conformation. The dicyanomethylene group and the 4-methoxyphenyl groups have equatorial orientations. A crystallographic mirror plane bisects the molecule, passing through the S and opposite C atoms of the central ring.

Related literature

For related literature, see: Haller & Ludtke (1976); Manimekalai & Anusuya (2005).



Experimental

Crystal data

$C_{22}H_{20}N_2O_2S$
M_r = 376.47
Orthorhombic, $Pnma$
a = 8.4906 (3) Å
b = 24.6848 (7) Å
c = 9.4361 (4) Å
V = 1977.70 (12) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.18 mm^{-1}
T = 298 (2) K
0.15 × 0.11 × 0.11 mm

Data collection

Bruker APEXII diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2004)
T_{min} = 0.748, T_{max} = 1.000 (expected range = 0.733–0.980)
6801 measured reflections
1105 independent reflections
909 reflections with $I > 2\sigma(I)$
R_{int} = 0.024
θ_{max} = 21.0°

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.055
$wR(F^2)$ = 0.193
S = 1.18
1105 reflections
127 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.35 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}

Data collection: *APEX2* (Bruker, 2004); cell refinement: *APEX2*; data reduction: *SAINT-NT* (Bruker, 2004); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *PLATON* (Spek, 2003).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CF2145).

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supplementary materials

Acta Cryst. (2007). E**63**, o4149 [**doi:10.1107/S1600536807046296**]

2-[2,6-Bis(4-methoxyphenyl)tetrahydrothiopyran-4-ylidene]malononitrile

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Comment

In the title molecule, 4-dicyanomethylene-r2,c6-bis(*p*-methoxyphenyl)tetrahydrothiopyran, $C_{22}H_{20}N_2O_2S$, the tetrahydrothiopyran ring adopts a chair conformation. The dicyanomethylene group in the 4-position and the *p*-methoxyphenyl groups at positions 2 and 6 have equatorial orientations. A crystallographic mirror plane bisects the molecule, passing through the S and opposite C atoms of the central ring (Fig. 1). No classical hydrogen bonds are found in the crystal structure.

Experimental

The title compound was prepared from *cis*-2,6-bis(*p*-methoxyphenyl) tetrahydrothiopyran-4-one by adopting a general procedure described in the literature (Haller & Ludtke, 1976) and it was characterized using NMR techniques (Manimekalai & Anusuya, 2005).

Refinement

The H atoms were positioned geometrically and allowed to ride on their parent atoms with C—H = 0.93–0.98 Å and U_{iso} = 1.2–1.5U_{eq}(C).

Figures

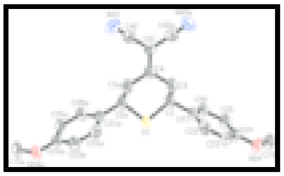


Fig. 1. The molecular structure with the atom numbering scheme. Displacement ellipsoids for non-H atoms are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radius. [Symmetry code: (*a*) *x*, 1/2 − *y*, *z*.]

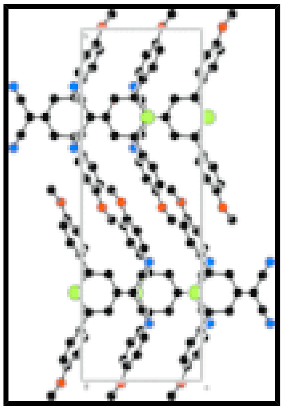


Fig. 2. The molecular packing of the title molecule, viewed down the *c* axis.

2-[2,6-Bis(4-methoxyphenyl)tetrahydrothiopyran-4-ylidene]malononitrile

Crystal data

$C_{22}H_{20}N_2O_2S$

$M_r = 376.47$

Orthorhombic, *Pnma*

Hall symbol: -P 2ac 2n

$a = 8.4906$ (3) Å

$b = 24.6848$ (7) Å

$c = 9.4361$ (4) Å

$V = 1977.70$ (12) Å^3

$Z = 4$

$F_{000} = 792$

$D_x = 1.264$ Mg m^{-3}

Melting point: 485(1) K

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å

Cell parameters from 2118 reflections

$\theta = 1.7$–21.0º

$\mu = 0.18$ mm^{-1}

$T = 298$ (2) K

Block, pale-yellow

0.15 × 0.11 × 0.11 mm

Data collection

Bruker APEXII
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 293(2)$ K

φ and ω scans

Absorption correction: multi-scan
(SADABS; Bruker, 2004)

$T_{\min} = 0.748$, $T_{\max} = 1.000$

6801 measured reflections

1105 independent reflections

909 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.024$

$\theta_{\max} = 21.0$º

$\theta_{\min} = 1.7$º

$h = -8 \rightarrow 7$

$k = -22 \rightarrow 24$

$l = -9 \rightarrow 8$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.055$

$wR(F^2) = 0.193$

$S = 1.18$

1105 reflections

127 parameters

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0939P)^2 + 1.7792P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max} = 0.35$ e Å^{-3}

$\Delta\rho_{\min} = -0.25$ e Å^{-3}

Extinction correction: none

Special details

Geometry. Bond distances, angles *etc*. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit S are based on F^2, conventional *R*-factors *R* are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on F, and R– factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.4518 (2)	0.25000	0.6263 (2)	0.0760 (7)
O24	0.3295 (5)	−0.00687 (13)	0.7469 (4)	0.1023 (16)
N43	1.0637 (7)	0.3378 (3)	0.3432 (5)	0.123 (3)
C2	0.5576 (7)	0.19394 (17)	0.5493 (7)	0.099 (3)
C3	0.7265 (7)	0.19799 (18)	0.5578 (8)	0.106 (3)
C4	0.7984 (9)	0.25000	0.5038 (9)	0.088 (3)
C15	0.2481 (8)	−0.0430 (2)	0.6552 (7)	0.113 (3)
C21	0.4916 (6)	0.14095 (18)	0.6034 (6)	0.0740 (19)
C22	0.5190 (6)	0.12309 (19)	0.7398 (7)	0.087 (2)
C23	0.4615 (6)	0.0735 (2)	0.7861 (6)	0.086 (2)
C24	0.3781 (6)	0.04156 (17)	0.6932 (6)	0.0753 (19)
C25	0.3502 (6)	0.05867 (19)	0.5597 (6)	0.085 (2)
C26	0.4067 (6)	0.10849 (18)	0.5145 (6)	0.085 (2)
C41	0.9279 (9)	0.25000	0.4247 (8)	0.078 (3)
C42	1.0032 (7)	0.2993 (3)	0.3802 (5)	0.090 (2)
H2	0.53261	0.19483	0.44792	0.1190*
H3A	0.75715	0.19345	0.65612	0.1276*
H3B	0.77170	0.16810	0.50488	0.1276*
H15A	0.22139	−0.07538	0.70603	0.1695*
H15B	0.15368	−0.02594	0.62143	0.1695*
H15C	0.31438	−0.05200	0.57620	0.1695*
H22	0.57693	0.14461	0.80170	0.1050*
H23	0.47919	0.06205	0.87858	0.1027*
H25	0.29289	0.03699	0.49771	0.1015*
H26	0.38648	0.11995	0.42242	0.1022*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0838 (13)	0.0425 (11)	0.1016 (14)	0.0000	0.0084 (10)	0.0000
O24	0.116 (3)	0.056 (2)	0.135 (3)	−0.014 (2)	0.011 (2)	0.017 (2)
N43	0.140 (5)	0.115 (4)	0.114 (4)	−0.035 (4)	0.016 (3)	0.017 (3)
C2	0.110 (5)	0.042 (3)	0.146 (5)	0.004 (3)	0.021 (4)	−0.003 (3)

C3	0.097 (4)	0.050 (3)	0.172 (6)	0.005 (3)	0.033 (4)	0.001 (3)
C4	0.090 (5)	0.053 (4)	0.121 (6)	0.0000	0.013 (5)	0.0000
C15	0.123 (5)	0.055 (3)	0.161 (6)	−0.022 (3)	0.020 (4)	−0.009 (3)
C21	0.088 (3)	0.042 (3)	0.092 (4)	0.007 (2)	0.017 (3)	0.004 (3)
C22	0.096 (4)	0.060 (3)	0.106 (4)	−0.005 (3)	−0.008 (3)	−0.013 (3)
C23	0.099 (4)	0.065 (3)	0.093 (4)	−0.002 (3)	−0.010 (3)	0.012 (3)
C24	0.081 (3)	0.040 (3)	0.105 (4)	0.005 (2)	0.012 (3)	0.011 (3)
C25	0.092 (4)	0.057 (3)	0.106 (4)	−0.012 (3)	0.004 (3)	−0.005 (3)
C26	0.096 (4)	0.059 (3)	0.101 (4)	−0.003 (3)	0.013 (3)	0.004 (3)
C41	0.089 (5)	0.069 (5)	0.076 (4)	0.0000	0.006 (4)	0.0000
C42	0.103 (4)	0.087 (4)	0.079 (3)	−0.007 (4)	0.014 (3)	0.005 (3)

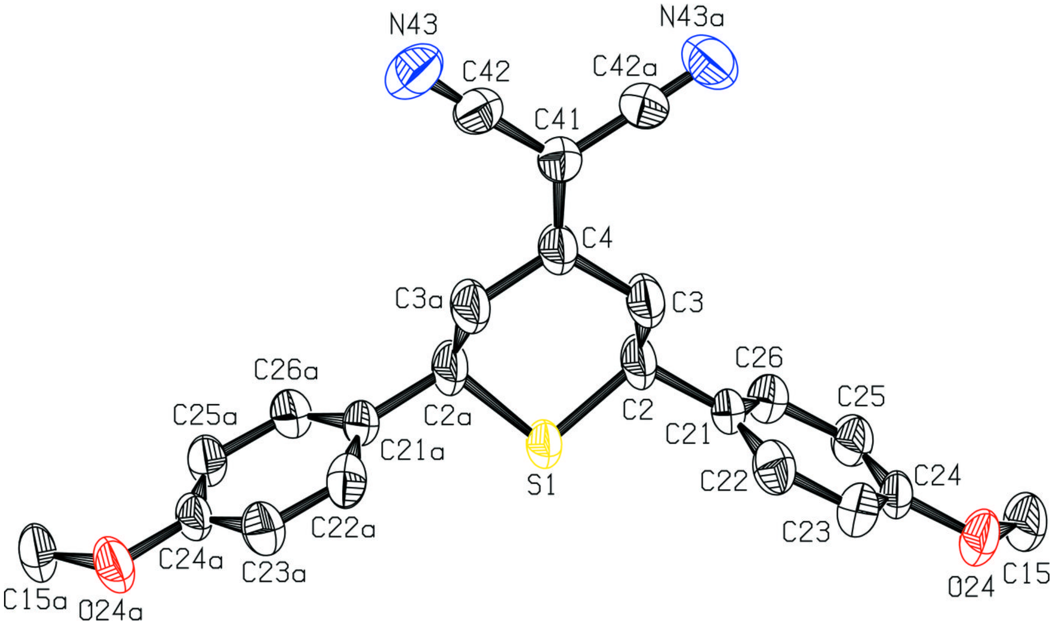
Geometric parameters (Å, °)

S1—C2	1.803 (5)	C25—C26	1.387 (7)
S1—C2^{i}	1.803 (5)	C41—C42	1.437 (8)
O24—C15	1.422 (7)	C41—C42^{i}	1.437 (8)
O24—C24	1.362 (6)	C2—H2	0.980
N43—C42	1.135 (10)	C3—H3A	0.970
C2—C3	1.440 (8)	C3—H3B	0.970
C2—C21	1.512 (7)	C15—H15A	0.960
C3—C4	1.510 (7)	C15—H15B	0.960
C4—C41	1.329 (11)	C15—H15C	0.960
C21—C22	1.380 (8)	C22—H22	0.930
C21—C26	1.366 (7)	C23—H23	0.930
C22—C23	1.388 (7)	C25—H25	0.930
C23—C24	1.375 (7)	C26—H26	0.930
C24—C25	1.350 (8)		
C2—S1—C2^{i}	100.3 (3)	S1—C2—H2	106.0
C15—O24—C24	118.1 (4)	C3—C2—H2	106.0
S1—C2—C3	114.9 (4)	C21—C2—H2	106.0
S1—C2—C21	110.1 (4)	C2—C3—H3A	108.0
C3—C2—C21	114.2 (4)	C2—C3—H3B	108.0
C2—C3—C4	116.3 (5)	C4—C3—H3A	108.0
C3—C4—C41	121.6 (3)	C4—C3—H3B	108.0
C3—C4—C3^{i}	116.5 (6)	H3A—C3—H3B	107.0
C3^{i}—C4—C41	121.6 (3)	O24—C15—H15A	109.0
C2—C21—C22	121.9 (5)	O24—C15—H15B	109.0
C2—C21—C26	119.7 (5)	O24—C15—H15C	109.0
C22—C21—C26	118.3 (4)	H15A—C15—H15B	110.0
C21—C22—C23	121.1 (5)	H15A—C15—H15C	110.0
C22—C23—C24	119.1 (5)	H15B—C15—H15C	109.0
O24—C24—C23	115.0 (5)	C21—C22—H22	119.0
O24—C24—C25	124.7 (5)	C23—C22—H22	119.0
C23—C24—C25	120.4 (4)	C22—C23—H23	120.0
C24—C25—C26	120.3 (5)	C24—C23—H23	120.0
C21—C26—C25	120.9 (5)	C24—C25—H25	120.0
C4—C41—C42	122.2 (4)	C26—C25—H25	120.0

C4—C41—C42$^{\mathrm{i}}$	122.2 (4)	C21—C26—H26	120.0
C42—C41—C42$^{\mathrm{i}}$	115.7 (6)	C25—C26—H26	120.0
N43—C42—C41	178.9 (6)		
C2$^{\mathrm{i}}$—S1—C2—C3	47.6 (5)	C3—C4—C41—C42	−175.9 (6)
C2$^{\mathrm{i}}$—S1—C2—C21	178.2 (4)	C3—C4—C41—C42$^{\mathrm{i}}$	3.0 (12)
C15—O24—C24—C23	−177.5 (5)	C2—C21—C26—C25	177.4 (5)
C15—O24—C24—C25	2.2 (8)	C22—C21—C26—C25	−0.4 (8)
S1—C2—C21—C22	−73.4 (6)	C2—C21—C22—C23	−178.0 (5)
S1—C2—C3—C4	−51.9 (8)	C26—C21—C22—C23	−0.2 (8)
C21—C2—C3—C4	179.5 (6)	C21—C22—C23—C24	1.0 (8)
C3—C2—C21—C26	−120.1 (6)	C22—C23—C24—C25	−1.2 (8)
C3—C2—C21—C22	57.6 (7)	C22—C23—C24—O24	178.5 (5)
S1—C2—C21—C26	108.9 (5)	O24—C24—C25—C26	−179.1 (5)
C2—C3—C4—C3$^{\mathrm{i}}$	49.4 (9)	C23—C24—C25—C26	0.6 (8)
C2—C3—C4—C41	−137.3 (8)	C24—C25—C26—C21	0.2 (8)
C3$^{\mathrm{i}}$—C4—C41—C42	−3.0 (12)		

Symmetry codes: (i) x, $-y+1/2$, z.

Fig. 1


N43
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C26
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S1
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C24
C22a
C23
C24a
C23a
O24
C15
C15a
O24a

Fig. 2