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4-(4-Bromophenyl)-2-methyl-2,6-diphenyl-2*H*-thiopyran

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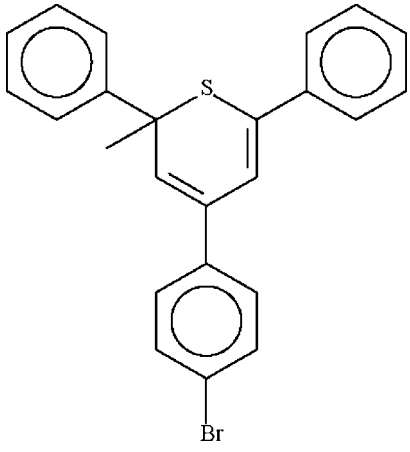
Key indicators: single-crystal X-ray study; T = 115 K; mean σ(C–C) = 0.012 Å; R factor = 0.075; wR factor = 0.190; data-to-parameter ratio = 18.7.

The six-membered thiopyran ring in the title compound, $C_{24}H_{19}BrS$, adopts an approximate envelope conformation, with the S atom displaced by 0.26 (1) Å and the 2-methylene C atom by −0.54 (1) Å from the plane of the other four sp^2-hydridized C atoms. The methyl substituent on the methylene carbon lies in a pseudo-axial position with the phenyl ring in a pseudo-equatorial position.

Related literature

For the background to 4-alkyl-2,4,6-triaryl-4*H*-thiopyrans, see: Rahmani *et al.* (2009). For the general synthesis from a Grignard reaction, see: Suld & Price (1962).



Experimental

Crystal data

$C_{24}H_{19}BrS$
M_r = 419.36
Orthorhombic, $Pca2_1$
a = 23.3348 (8) Å
b = 5.9991 (2) Å
c = 13.6866 (5) Å
V = 1916.0 (1) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 2.26 mm^{-1}
T = 115 K
0.40 × 0.15 × 0.05 mm

Data collection

Bruker SMART APEX diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.465, T_{max} = 0.895
16215 measured reflections
4407 independent reflections
3017 reflections with $I > 2\sigma(I)$
R_{int} = 0.093

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.075
$wR(F^2)$ = 0.190
S = 1.09
4407 reflections
236 parameters
145 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 1.53 e Å^{-3}
$\Delta\rho_{min}$ = −0.91 e Å^{-3}
Absolute structure: Flack (1983), 2108 Friedel pairs
Flack parameter: 0.01 (2)

Data collection: *APEX2* (Bruker, 2008); cell refinement: *SAINT* (Bruker, 2008); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2009).

We thank the Iranian Research Organization for Science and Technology and the University of Malaya for supporting this study.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SJ2581).

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supporting information

Acta Cryst. (2009). E**65**, o606 [doi:10.1107/S1600536809005959]

4-(4-Bromophenyl)-2-methyl-2,6-diphenyl-2*H*-thiopyran

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S1. Experimental

The compound was obtained as the rearranged product from the reaction of methyl magnesium bromide and 4-(4-bromophenyl)-2,6-diphenyl thiopyrylium perchlorate in dry ether under an argon atmosphere according to a reported method (Suld & Price, 1962). The product was isolated by TLC on neutral alumina (petroleum ether 40–60 °C) and purified by recrystalization from ethanol.

S2. Refinement

Carbon-bound H-atoms were placed in calculated positions (C–H 0.95 to 0.98 Å) and were included in the refinement in the riding model approximation, with *U*(H) set to 1.2 to 1.5*U*(C).

The final difference Fourier map had a large peak/deep hole in the vicinity of the bromine.

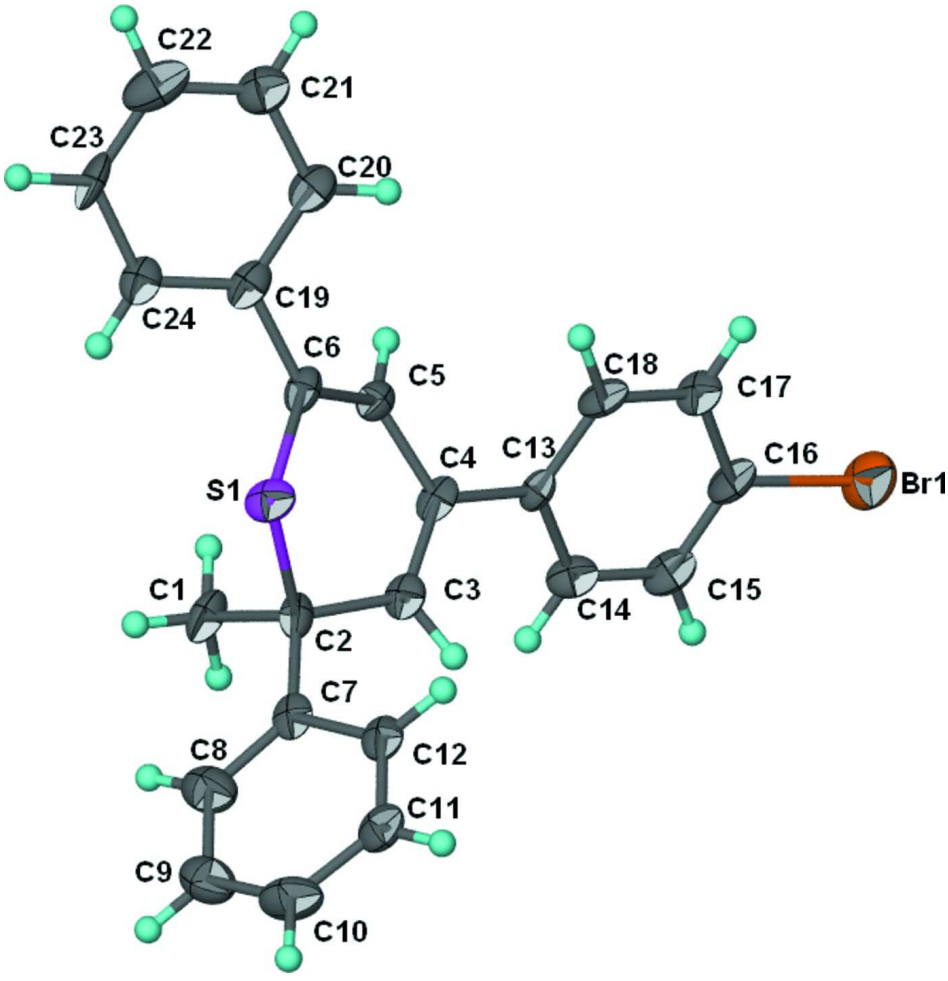

Figure 1

Thermal ellisoid plot (Barbour, 2001) of $C_{24}H_{19}BrS$; probability levels are set at 70% and H-atoms are drawn as spheres of arbitrary radius.

4-(4-Bromophenyl)-2-methyl-2,6-diphenyl-2*H*-thiopyran

Crystal data

$C_{24}H_{19}BrS$
$M_r = 419.36$
Orthorhombic, $Pca2_1$
Hall symbol: P 2c -2ac
$a = 23.3348\ (8)$ Å
$b = 5.9991\ (2)$ Å
$c = 13.6866\ (5)$ Å
$V = 1916.0\ (1)$ Å^3
$Z = 4$
$F(000) = 856$
$D_x = 1.454$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2506 reflections
$\theta = 2.3–22.9°$
$\mu = 2.26$ mm^{-1}
$T = 115$ K
Prism, pale yellow
0.40 × 0.15 × 0.05 mm

Data collection

Bruker SMART APEX diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.465$, $T_{max} = 0.895$

16215 measured reflections
4407 independent reflections
3017 reflections with $I > 2\sigma(I)$
$R_{int} = 0.093$
$\theta_{max} = 27.5°$, $\theta_{min} = 1.8°$
$h = -30 \rightarrow 29$
$k = -7 \rightarrow 7$
$l = -17 \rightarrow 17$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.075$
$wR(F^2) = 0.190$
$S = 1.09$
4407 reflections
236 parameters
145 restraints
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
$w = 1/[\sigma^2(F_o^2) + (0.0672P)^2 + 10.0175P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 1.53$ e Å^{-3}
$\Delta\rho_{min} = -0.91$ e Å^{-3}
Absolute structure: Flack (1983), 2108 Friedel pairs
Absolute structure parameter: 0.01 (2)

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Br1	0.55183 (4)	0.84726 (15)	0.50001 (7)	0.0381 (3)
S1	0.76405 (9)	−0.1684 (4)	0.84041 (14)	0.0245 (4)
C1	0.7867 (4)	−0.2735 (13)	0.6484 (6)	0.0260 (18)
H1A	0.8005	−0.2273	0.5838	0.039*
H1B	0.7469	−0.3246	0.6431	0.039*
H1C	0.8107	−0.3952	0.6730	0.039*
C2	0.7898 (3)	−0.0738 (13)	0.7196 (5)	0.0198 (16)
C3	0.7503 (3)	0.1075 (12)	0.6851 (5)	0.0206 (17)
H3	0.7669	0.2372	0.6571	0.025*
C4	0.6924 (3)	0.0971 (13)	0.6915 (5)	0.0191 (16)
C5	0.6643 (3)	−0.0773 (13)	0.7470 (5)	0.0183 (16)
H5	0.6252	−0.1083	0.7332	0.022*
C6	0.6907 (4)	−0.1988 (12)	0.8174 (5)	0.0204 (17)
C7	0.8509 (3)	0.0024 (14)	0.7371 (5)	0.0202 (17)
C8	0.8986 (4)	−0.1115 (16)	0.7095 (6)	0.032 (2)
H8	0.8937	−0.2494	0.6764	0.039*
C9	0.9534 (4)	−0.0388 (16)	0.7267 (6)	0.033 (2)
H9	0.9852	−0.1234	0.7039	0.040*
C10	0.9623 (4)	0.1547 (18)	0.7762 (7)	0.034 (2)
H10	1.0000	0.2016	0.7922	0.041*
C11	0.9148 (4)	0.2837 (15)	0.8034 (6)	0.0263 (19)
H11	0.9206	0.4238	0.8342	0.032*
C12	0.8604 (4)	0.2107 (13)	0.7863 (5)	0.0225 (17)
H12	0.8287	0.2979	0.8069	0.027*
C13	0.6567 (3)	0.2668 (12)	0.6448 (5)	0.0168 (15)
C14	0.6740 (4)	0.3678 (14)	0.5562 (5)	0.0236 (17)
H14	0.7081	0.3187	0.5253	0.028*
C15	0.6422 (3)	0.5366 (13)	0.5137 (6)	0.0274 (18)

H15	0.6552	0.6058	0.4553	0.033*
C16	0.5923 (4)	0.6028 (14)	0.5560 (6)	0.0276 (19)
C17	0.5729 (4)	0.5021 (14)	0.6414 (5)	0.0223 (17)
H17	0.5376	0.5474	0.6698	0.027*
C18	0.6048 (4)	0.3370 (14)	0.6844 (6)	0.0230 (17)
H18	0.5912	0.2691	0.7427	0.028*
C19	0.6600 (4)	−0.3593 (13)	0.8826 (5)	0.0222 (17)
C20	0.6058 (4)	−0.2976 (14)	0.9198 (6)	0.0258 (19)
H20	0.5896	−0.1564	0.9046	0.031*
C21	0.5766 (4)	−0.4471 (14)	0.9787 (5)	0.0284 (19)
H21	0.5393	−0.4106	1.0017	0.034*
C22	0.6007 (4)	−0.6463 (15)	1.0043 (8)	0.0363 (19)
H22	0.5808	−0.7436	1.0473	0.044*
C23	0.6542 (4)	−0.7082 (13)	0.9679 (6)	0.0259 (19)
H23	0.6702	−0.8489	0.9843	0.031*
C24	0.6841 (4)	−0.5623 (13)	0.9073 (5)	0.0214 (17)
H24	0.7209	−0.6021	0.8831	0.026*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0382 (5)	0.0361 (4)	0.0401 (5)	0.0017 (4)	−0.0071 (5)	0.0148 (5)
S1	0.0254 (10)	0.0295 (11)	0.0186 (9)	−0.0016 (9)	−0.0016 (8)	0.0090 (9)
C1	0.041 (5)	0.014 (4)	0.023 (4)	−0.005 (3)	−0.002 (4)	0.000 (3)
C2	0.029 (4)	0.015 (3)	0.016 (3)	0.004 (3)	0.001 (3)	0.001 (3)
C3	0.027 (4)	0.021 (4)	0.014 (3)	0.001 (3)	−0.003 (3)	0.005 (3)
C4	0.031 (4)	0.018 (4)	0.008 (3)	−0.002 (3)	−0.003 (3)	0.006 (3)
C5	0.023 (4)	0.016 (4)	0.016 (3)	−0.002 (3)	0.001 (3)	0.000 (3)
C6	0.031 (4)	0.015 (4)	0.015 (3)	0.000 (3)	−0.001 (3)	0.002 (3)
C7	0.031 (4)	0.020 (4)	0.010 (3)	0.004 (3)	0.000 (3)	0.007 (3)
C8	0.028 (4)	0.041 (5)	0.028 (4)	0.004 (4)	0.003 (3)	0.010 (4)
C9	0.028 (4)	0.042 (5)	0.028 (4)	0.005 (4)	0.005 (4)	0.005 (4)
C10	0.020 (4)	0.047 (5)	0.036 (4)	−0.007 (4)	−0.004 (3)	0.010 (4)
C11	0.032 (5)	0.023 (4)	0.024 (4)	−0.001 (4)	−0.002 (4)	0.010 (3)
C12	0.033 (4)	0.020 (4)	0.015 (3)	0.002 (3)	0.005 (3)	0.004 (3)
C13	0.022 (4)	0.014 (3)	0.015 (3)	−0.002 (3)	−0.004 (3)	0.004 (3)
C14	0.025 (4)	0.030 (4)	0.016 (3)	−0.004 (3)	−0.002 (3)	0.004 (3)
C15	0.036 (4)	0.029 (4)	0.017 (4)	−0.006 (3)	−0.001 (3)	0.010 (3)
C16	0.035 (5)	0.024 (4)	0.024 (4)	−0.004 (3)	0.002 (4)	0.012 (3)
C17	0.029 (4)	0.025 (4)	0.013 (3)	0.001 (3)	−0.001 (3)	0.007 (3)
C18	0.025 (4)	0.026 (4)	0.017 (3)	−0.007 (3)	−0.001 (3)	0.010 (3)
C19	0.032 (4)	0.019 (4)	0.016 (3)	−0.004 (3)	−0.004 (3)	0.002 (3)
C20	0.032 (4)	0.025 (4)	0.020 (3)	−0.002 (3)	−0.001 (3)	0.007 (3)
C21	0.032 (4)	0.032 (4)	0.022 (4)	−0.002 (3)	−0.001 (3)	0.006 (3)
C22	0.043 (4)	0.041 (4)	0.025 (3)	−0.018 (4)	−0.006 (5)	0.009 (4)
C23	0.046 (5)	0.010 (4)	0.022 (4)	−0.003 (3)	−0.006 (3)	0.006 (3)
C24	0.031 (4)	0.020 (4)	0.013 (3)	0.000 (3)	−0.002 (3)	0.002 (3)

Geometric parameters (Å, º)

Br1—C16	1.905 (8)	C11—C12	1.363 (12)
S1—C6	1.750 (8)	C11—H11	0.9500
S1—C2	1.849 (8)	C12—H12	0.9500
C1—C2	1.547 (11)	C13—C18	1.391 (11)
C1—H1A	0.9800	C13—C14	1.414 (10)
C1—H1B	0.9800	C14—C15	1.383 (11)
C1—H1C	0.9800	C14—H14	0.9500
C2—C3	1.501 (10)	C15—C16	1.360 (12)
C2—C7	1.516 (11)	C15—H15	0.9500
C3—C4	1.356 (11)	C16—C17	1.392 (11)
C3—H3	0.9500	C17—C18	1.373 (11)
C4—C5	1.448 (10)	C17—H17	0.9500
C4—C13	1.463 (10)	C18—H18	0.9500
C5—C6	1.357 (11)	C19—C24	1.383 (11)
C5—H5	0.9500	C19—C20	1.414 (12)
C6—C19	1.495 (11)	C20—C21	1.385 (11)
C7—C8	1.361 (12)	C20—H20	0.9500
C7—C12	1.436 (11)	C21—C22	1.366 (13)
C8—C9	1.372 (12)	C21—H21	0.9500
C8—H8	0.9500	C22—C23	1.395 (13)
C9—C10	1.360 (14)	C22—H22	0.9500
C9—H9	0.9500	C23—C24	1.393 (11)
C10—C11	1.402 (13)	C23—H23	0.9500
C10—H10	0.9500	C24—H24	0.9500
C6—S1—C2	100.9 (4)	C11—C12—C7	120.3 (8)
C2—C1—H1A	109.5	C11—C12—H12	119.8
C2—C1—H1B	109.5	C7—C12—H12	119.8
H1A—C1—H1B	109.5	C18—C13—C14	116.9 (7)
C2—C1—H1C	109.5	C18—C13—C4	122.4 (6)
H1A—C1—H1C	109.5	C14—C13—C4	120.7 (7)
H1B—C1—H1C	109.5	C15—C14—C13	121.5 (8)
C3—C2—C7	114.1 (7)	C15—C14—H14	119.3
C3—C2—C1	109.5 (6)	C13—C14—H14	119.3
C7—C2—C1	112.2 (7)	C16—C15—C14	119.5 (7)
C3—C2—S1	107.7 (5)	C16—C15—H15	120.2
C7—C2—S1	104.9 (5)	C14—C15—H15	120.2
C1—C2—S1	108.1 (5)	C15—C16—C17	120.7 (8)
C4—C3—C2	123.9 (7)	C15—C16—Br1	118.6 (6)
C4—C3—H3	118.0	C17—C16—Br1	120.7 (7)
C2—C3—H3	118.0	C18—C17—C16	119.8 (8)
C3—C4—C5	121.2 (7)	C18—C17—H17	120.1
C3—C4—C13	120.5 (7)	C16—C17—H17	120.1
C5—C4—C13	118.3 (7)	C17—C18—C13	121.6 (7)
C6—C5—C4	123.7 (7)	C17—C18—H18	119.2
C6—C5—H5	118.1	C13—C18—H18	119.2

C4—C5—H5	118.1	C24—C19—C20	120.4 (7)
C5—C6—C19	123.6 (8)	C24—C19—C6	121.2 (8)
C5—C6—S1	121.0 (6)	C20—C19—C6	118.4 (7)
C19—C6—S1	115.3 (6)	C21—C20—C19	118.7 (8)
C8—C7—C12	116.1 (8)	C21—C20—H20	120.6
C8—C7—C2	125.1 (8)	C19—C20—H20	120.6
C12—C7—C2	118.8 (7)	C22—C21—C20	120.9 (8)
C7—C8—C9	123.8 (9)	C22—C21—H21	119.6
C7—C8—H8	118.1	C20—C21—H21	119.6
C9—C8—H8	118.1	C21—C22—C23	120.6 (9)
C10—C9—C8	119.9 (9)	C21—C22—H22	119.7
C10—C9—H9	120.1	C23—C22—H22	119.7
C8—C9—H9	120.1	C24—C23—C22	119.6 (8)
C9—C10—C11	118.9 (8)	C24—C23—H23	120.2
C9—C10—H10	120.5	C22—C23—H23	120.2
C11—C10—H10	120.5	C19—C24—C23	119.7 (8)
C12—C11—C10	120.8 (9)	C19—C24—H24	120.2
C12—C11—H11	119.6	C23—C24—H24	120.2
C10—C11—H11	119.6		

C6—S1—C2—C3	46.9 (6)	C2—C7—C12—C11	179.8 (7)
C6—S1—C2—C7	168.8 (5)	C3—C4—C13—C18	−146.5 (8)
C6—S1—C2—C1	−71.3 (6)	C5—C4—C13—C18	31.7 (11)
C7—C2—C3—C4	−160.3 (7)	C3—C4—C13—C14	33.4 (11)
C1—C2—C3—C4	73.0 (9)	C5—C4—C13—C14	−148.5 (7)
S1—C2—C3—C4	−44.3 (9)	C18—C13—C14—C15	3.2 (12)
C2—C3—C4—C5	10.0 (11)	C4—C13—C14—C15	−176.6 (7)
C2—C3—C4—C13	−171.9 (7)	C13—C14—C15—C16	−2.1 (13)
C3—C4—C5—C6	19.9 (12)	C14—C15—C16—C17	−0.2 (13)
C13—C4—C5—C6	−158.2 (7)	C14—C15—C16—Br1	176.4 (6)
C4—C5—C6—C19	172.4 (7)	C15—C16—C17—C18	1.2 (13)
C4—C5—C6—S1	−5.3 (11)	Br1—C16—C17—C18	−175.3 (7)
C2—S1—C6—C5	−26.8 (7)	C16—C17—C18—C13	0.0 (13)
C2—S1—C6—C19	155.3 (6)	C14—C13—C18—C17	−2.2 (12)
C3—C2—C7—C8	−136.8 (8)	C4—C13—C18—C17	177.7 (8)
C1—C2—C7—C8	−11.5 (11)	C5—C6—C19—C24	139.8 (8)
S1—C2—C7—C8	105.6 (8)	S1—C6—C19—C24	−42.3 (9)
C3—C2—C7—C12	43.0 (9)	C5—C6—C19—C20	−40.9 (11)
C1—C2—C7—C12	168.3 (6)	S1—C6—C19—C20	136.9 (7)
S1—C2—C7—C12	−74.6 (7)	C24—C19—C20—C21	−1.9 (12)
C12—C7—C8—C9	0.4 (12)	C6—C19—C20—C21	178.8 (7)
C2—C7—C8—C9	−179.8 (8)	C19—C20—C21—C22	2.9 (13)
C7—C8—C9—C10	1.9 (13)	C20—C21—C22—C23	−3.0 (14)
C8—C9—C10—C11	−4.1 (13)	C21—C22—C23—C24	2.0 (13)
C9—C10—C11—C12	4.2 (13)	C20—C19—C24—C23	1.0 (11)
C10—C11—C12—C7	−1.9 (12)	C6—C19—C24—C23	−179.7 (7)
C8—C7—C12—C11	−0.4 (11)	C22—C23—C24—C19	−1.0 (12)