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Bis[4-(4-aminophenylsulfanyl)phenyl]ketone

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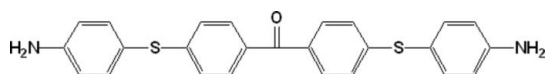
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Key indicators: single-crystal X-ray study; $T = 292$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.050; wR factor = 0.146; data-to-parameter ratio = 13.7.

The molecule of the title compound, $\text{C}_{25}\text{H}_{20}\text{N}_2\text{O}_2\text{S}_2$, has imposed twofold rotation symmetry. The dihedral angle formed by the two crystallographically independent phenyl rings is $79.23(7)^\circ$. In the crystal packing, the molecules are linked by intermolecular $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds, forming chains running parallel to $[102]$.

Related literature

For the properties and applications of the title compound and related derivatives, see: Wang *et al.* (2006a,b); Jiang *et al.* (2006); Aritomi & Terauchi (1985); Aritomi & Fujiwara (1986). For the synthesis of the title compound, see: Yang *et al.* (2007); Chen *et al.* (2009).



Experimental

Crystal data

 $\text{C}_{25}\text{H}_{20}\text{N}_2\text{O}_2\text{S}_2$ $M_r = 428.55$ Monoclinic, $C2/c$ $a = 18.945(3)$ Å $b = 6.025(2)$ Å $c = 20.793(5)$ Å $\beta = 110.64(4)^\circ$ $V = 2221.1(11)$ Å³ $Z = 4$ Mo $K\alpha$ radiation $\mu = 0.26$ mm⁻¹ $T = 292$ K $0.52 \times 0.48 \times 0.42$ mm

Data collection

Enraf–Nonius CAD-4

diffractometer

Absorption correction: spherical

(WinGX; Farrugia, 1999)

 $T_{\min} = 0.877$, $T_{\max} = 0.899$

2261 measured reflections

1990 independent reflections

1441 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.010$

3 standard reflections

every 150 reflections

intensity decay: 2.4%

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.050$ $wR(F^2) = 0.146$ $S = 1.05$

1990 reflections

145 parameters

H atoms treated by a mixture of independent and constrained refinement

 $\Delta\rho_{\text{max}} = 0.22$ e Å⁻³ $\Delta\rho_{\text{min}} = -0.30$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N1}-\text{HN2}\cdots\text{O1}^i$	0.77 (3)	2.52 (3)	3.231 (4)	154 (3)

Symmetry code: (i) $-x + \frac{3}{2}, -y + \frac{1}{2}, -z + 1$.

Data collection: *DIFRAC* (Gabe *et al.*, 1993); cell refinement: *DIFRAC*; data reduction: *NRCVAX* (Gabe *et al.*, 1989); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: RZ2311).

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supplementary materials

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Bis[4-(4-aminophenylsulfanyl)phenyl] ketone

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Comment

The title compound is a major active photo-initiator used in coatings, optics and microelectronic materials (Wang *et al.*, 2006a,b; Jiang *et al.*, 2006) and can be used as monomer in the synthesis of high performance polyamide. Moreover, as photo initiator it has showed superior to the natural compound 4,4'-difluorobenzophenone (Wang *et al.*, 2006a). Besides their properties as photo-initiators, some derivatives of the title compound have also been reported to possess good thermo-stability and chemical resistance (Aritomi & Terauchi, 1985; Aritomi & Fujiwara, 1986). The synthetic procedure of the title compound have been reported elsewhere (Yang *et al.*, 2007; Chen *et al.*, 2009).

The molecule of the title compound (Fig. 1) has crystallographically imposed twofold rotation symmetry. In the asymmetric unit, the phenyl rings form a dihedral angle of 79.23 (7)°. The C2–C1–C2ⁱ–C7ⁱ torsion angle is 29.49 (15)° (symmetry code: $i = 1-x, y, 1/2-z$). In the crystal packing, intermolecular N—H···O hydrogen bonding interactions (Table 1) link the molecules into chains running parallel to the [102] direction.

Experimental

A mixture of 4,4'-difluorobenzophenone (21.8 g, 0.1 mol), 4-aminothiophenol (25 g, 0.2 mol), K₂CO₃ (14.0 g, 0.101 mol) and dimethyl acetamide (120 ml) were charged into a three-necked round-bottomed flask fitted with a mechanical stirrer, a thermometer and a nitrogen inlet. The mixture was stirred vigorously at 120°C for 3 h, then the mixture was heated to 166°C and kept for 5 h under nitrogen atmosphere. After the reactor was cooled to room temperature, the reaction solution was poured into water. The resulting solid was filtered, washed with hot water and methanol, dried and recrystallized from a mixture of dimethyl formamide and water (3:1 v/v) to get a yellow powder. Light yellow crystals suitable for X-ray analysis were obtained by slow evaporation of a formamide/water (3:1 v/v) solution at 60°C.

Refinement

The H atoms bound to the N atom were found in a difference Fourier map and refined freely. All other H atoms were positioned geometrically and refined using a riding model, with C—H = 0.93 Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

Figures

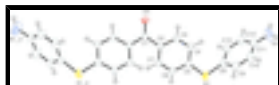


Fig. 1. The molecular structure of the title compound. Unlabelled atoms are related to the labelled atoms by the symmetry operator (1-x, y, 1/2-z).

Bis[4-(4-aminophenylsulfanyl)phenyl] ketone

Crystal data

$C_{25}H_{20}N_2OS_2$	$F_{000} = 896$
$M_r = 428.55$	$D_x = 1.282 \text{ Mg m}^{-3}$
Monoclinic, $C2/c$	Mo $K\alpha$ radiation
Hall symbol: $-C 2yc$	$\lambda = 0.71073 \text{ \AA}$
$a = 18.945 (3) \text{ \AA}$	Cell parameters from 31 reflections
$b = 6.025 (2) \text{ \AA}$	$\theta = 4.3\text{--}9.4^\circ$
$c = 20.793 (5) \text{ \AA}$	$\mu = 0.26 \text{ mm}^{-1}$
$\beta = 110.64 (4)^\circ$	$T = 292 \text{ K}$
$V = 2221.1 (11) \text{ \AA}^3$	Block, yellow
$Z = 4$	$0.52 \times 0.48 \times 0.42 \text{ mm}$

Data collection

Enraf–Nonius CAD-4 diffractometer	$R_{\text{int}} = 0.010$
Radiation source: fine-focus sealed tube	$\theta_{\text{max}} = 25.5^\circ$
Monochromator: graphite	$\theta_{\text{min}} = 2.1^\circ$
$T = 292 \text{ K}$	$h = -22 \rightarrow 21$
ω – 2θ scans	$k = 0 \rightarrow 7$
Absorption correction: for a sphere (WinGX; Farrugia, 1999)	$l = -18 \rightarrow 24$
$T_{\text{min}} = 0.877$, $T_{\text{max}} = 0.899$	3 standard reflections
2261 measured reflections	every 150 reflections
1990 independent reflections	intensity decay: 2.4%
1441 reflections with $I > 2\sigma(I)$	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: mixed
$R[F^2 > 2\sigma(F^2)] = 0.050$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.146$	$w = 1/[\sigma^2(F_o^2) + (0.0979P)^2 + 0.2492P]$
$S = 1.05$	where $P = (F_o^2 + 2F_c^2)/3$
1990 reflections	$(\Delta/\sigma)_{\text{max}} = 0.001$
145 parameters	$\Delta\rho_{\text{max}} = 0.22 \text{ e \AA}^{-3}$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{\text{min}} = -0.30 \text{ e \AA}^{-3}$
	Extinction correction: none

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

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 $F^2 > 2\sigma(F^2)$ 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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
S1	0.59611 (4)	0.88028 (12)	0.52196 (3)	0.0591 (3)
O1	0.5000	0.1894 (4)	0.2500	0.0638 (7)
N1	0.8241 (2)	0.3345 (7)	0.73370 (17)	0.0954 (11)
HN1	0.811 (2)	0.224 (7)	0.7515 (19)	0.096 (13)*
HN2	0.8651 (18)	0.371 (5)	0.7407 (16)	0.071 (11)*
C1	0.5000	0.3937 (5)	0.2500	0.0397 (7)
C2	0.52388 (11)	0.5146 (3)	0.31641 (10)	0.0364 (5)
C3	0.57458 (12)	0.4134 (4)	0.37505 (11)	0.0427 (5)
H3	0.5933	0.2731	0.3714	0.051*
C4	0.59739 (12)	0.5168 (4)	0.43805 (11)	0.0437 (5)
H4	0.6312	0.4466	0.4764	0.052*
C5	0.56977 (12)	0.7271 (4)	0.44448 (11)	0.0409 (5)
C6	0.51783 (12)	0.8281 (4)	0.38664 (10)	0.0396 (5)
H6	0.4979	0.9662	0.3907	0.048*
C7	0.49596 (11)	0.7241 (3)	0.32367 (10)	0.0371 (5)
H7	0.4621	0.7944	0.2854	0.045*
C8	0.66367 (12)	0.7082 (4)	0.58244 (11)	0.0484 (6)
C9	0.73981 (14)	0.7565 (5)	0.60170 (13)	0.0606 (7)
H9	0.7555	0.8750	0.5814	0.073*
C10	0.79254 (14)	0.6301 (5)	0.65085 (14)	0.0650 (8)
H10	0.8435	0.6644	0.6631	0.078*
C11	0.77125 (14)	0.4541 (5)	0.68221 (12)	0.0580 (7)
C12	0.69484 (15)	0.4026 (5)	0.66200 (13)	0.0634 (7)
H12	0.6794	0.2819	0.6816	0.076*
C13	0.64197 (13)	0.5290 (5)	0.61320 (12)	0.0567 (7)
H13	0.5911	0.4940	0.6007	0.068*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0615 (5)	0.0678 (5)	0.0395 (4)	0.0091 (3)	0.0073 (3)	-0.0110 (3)
O1	0.0882 (19)	0.0344 (13)	0.0499 (14)	0.000	0.0009 (13)	0.000

supplementary materials

N1	0.0612 (19)	0.134 (3)	0.084 (2)	0.0128 (19)	0.0163 (16)	0.042 (2)
C1	0.0395 (16)	0.0318 (16)	0.0397 (16)	0.000	0.0038 (13)	0.000
C2	0.0376 (11)	0.0363 (11)	0.0336 (10)	-0.0016 (9)	0.0103 (8)	0.0045 (8)
C3	0.0456 (12)	0.0357 (12)	0.0413 (12)	0.0045 (9)	0.0084 (10)	0.0046 (9)
C4	0.0408 (12)	0.0475 (13)	0.0364 (11)	0.0030 (10)	0.0058 (9)	0.0049 (10)
C5	0.0375 (11)	0.0475 (13)	0.0379 (11)	-0.0045 (10)	0.0137 (9)	-0.0013 (9)
C6	0.0395 (11)	0.0413 (12)	0.0401 (11)	0.0021 (9)	0.0166 (10)	0.0012 (9)
C7	0.0350 (10)	0.0383 (11)	0.0366 (11)	0.0017 (9)	0.0109 (9)	0.0039 (9)
C8	0.0412 (13)	0.0668 (16)	0.0348 (11)	-0.0065 (11)	0.0102 (10)	-0.0071 (11)
C9	0.0500 (14)	0.0760 (18)	0.0540 (15)	-0.0172 (13)	0.0164 (12)	0.0067 (13)
C10	0.0364 (13)	0.092 (2)	0.0602 (16)	-0.0089 (13)	0.0092 (12)	0.0043 (15)
C11	0.0501 (15)	0.0776 (17)	0.0433 (13)	0.0015 (13)	0.0126 (11)	0.0023 (12)
C12	0.0585 (16)	0.079 (2)	0.0537 (15)	-0.0130 (14)	0.0206 (13)	0.0093 (13)
C13	0.0380 (12)	0.0843 (19)	0.0454 (13)	-0.0144 (12)	0.0115 (10)	-0.0014 (13)

Geometric parameters (\AA , $^\circ$)

S1—C5	1.769 (2)	C5—C6	1.397 (3)
S1—C8	1.777 (3)	C6—C7	1.377 (3)
O1—C1	1.231 (3)	C6—H6	0.9300
N1—C11	1.383 (4)	C7—H7	0.9300
N1—HN1	0.84 (4)	C8—C9	1.385 (3)
N1—HN2	0.77 (3)	C8—C13	1.389 (4)
C1—C2	1.484 (2)	C9—C10	1.380 (4)
C1—C2 ⁱ	1.484 (2)	C9—H9	0.9300
C2—C7	1.397 (3)	C10—C11	1.377 (4)
C2—C3	1.398 (3)	C10—H10	0.9300
C3—C4	1.375 (3)	C11—C12	1.393 (4)
C3—H3	0.9300	C12—C13	1.377 (4)
C4—C5	1.395 (3)	C12—H12	0.9300
C4—H4	0.9300	C13—H13	0.9300
C5—S1—C8	103.99 (12)	C6—C7—C2	121.02 (19)
C11—N1—HN1	121 (2)	C6—C7—H7	119.5
C11—N1—HN2	114 (2)	C2—C7—H7	119.5
HN1—N1—HN2	125 (3)	C9—C8—C13	118.5 (2)
O1—C1—C2	119.39 (12)	C9—C8—S1	119.9 (2)
O1—C1—C2 ⁱ	119.39 (12)	C13—C8—S1	121.52 (17)
C2—C1—C2 ⁱ	121.2 (2)	C10—C9—C8	120.4 (2)
C7—C2—C3	118.00 (18)	C10—C9—H9	119.8
C7—C2—C1	122.75 (18)	C8—C9—H9	119.8
C3—C2—C1	119.21 (19)	C11—C10—C9	121.3 (2)
C4—C3—C2	121.4 (2)	C11—C10—H10	119.4
C4—C3—H3	119.3	C9—C10—H10	119.4
C2—C3—H3	119.3	C10—C11—N1	120.9 (3)
C3—C4—C5	120.0 (2)	C10—C11—C12	118.4 (2)
C3—C4—H4	120.0	N1—C11—C12	120.7 (3)
C5—C4—H4	120.0	C13—C12—C11	120.5 (3)
C4—C5—C6	119.15 (19)	C13—C12—H12	119.7

C4—C5—S1	124.48 (16)	C11—C12—H12	119.7
C6—C5—S1	116.37 (17)	C12—C13—C8	120.8 (2)
C7—C6—C5	120.3 (2)	C12—C13—H13	119.6
C7—C6—H6	119.8	C8—C13—H13	119.6
C5—C6—H6	119.8		
O1—C1—C2—C7	-150.51 (15)	C3—C2—C7—C6	0.2 (3)
C2 ⁱ —C1—C2—C7	29.49 (15)	C1—C2—C7—C6	178.04 (18)
O1—C1—C2—C3	27.3 (2)	C5—S1—C8—C9	102.6 (2)
C2 ⁱ —C1—C2—C3	-152.7 (2)	C5—S1—C8—C13	-79.8 (2)
C7—C2—C3—C4	-0.8 (3)	C13—C8—C9—C10	-0.6 (4)
C1—C2—C3—C4	-178.80 (18)	S1—C8—C9—C10	177.1 (2)
C2—C3—C4—C5	0.1 (3)	C8—C9—C10—C11	-0.3 (4)
C3—C4—C5—C6	1.3 (3)	C9—C10—C11—N1	-177.0 (3)
C3—C4—C5—S1	-178.56 (17)	C9—C10—C11—C12	1.5 (4)
C8—S1—C5—C4	1.7 (2)	C10—C11—C12—C13	-1.8 (4)
C8—S1—C5—C6	-178.18 (16)	N1—C11—C12—C13	176.7 (3)
C4—C5—C6—C7	-2.0 (3)	C11—C12—C13—C8	0.9 (4)
S1—C5—C6—C7	177.89 (16)	C9—C8—C13—C12	0.3 (4)
C5—C6—C7—C2	1.3 (3)	S1—C8—C13—C12	-177.4 (2)

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

Hydrogen-bond geometry ($\text{\AA}, ^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1—HN2 ⁱⁱ —O1 ⁱⁱ	0.77 (3)	2.52 (3)	3.231 (4)	154 (3)

Symmetry codes: (ii) $-x+3/2, -y+1/2, -z+1$.

Fig. 1

