Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

1,3-Bis[4-(4-pyridyl)pyrimidin-2-ylsulfanyl]propane

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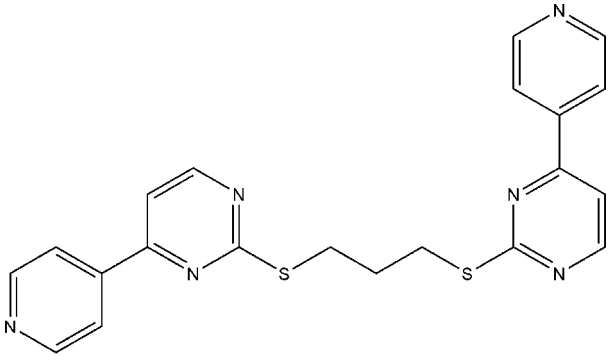
Received 8 April 2008; accepted 18 April 2008

Key indicators: single-crystal X-ray study; T = 291 K; mean σ(C–C) = 0.004 Å; R factor = 0.059; wR factor = 0.157; data-to-parameter ratio = 14.2.

In the title compound, $C_{21}H_{18}N_6S_2$, the dihedral angles between the aromatic rings in the two 4-(4-pyridyl)pyrimidine residues are 23.45 (13) and 2.67 (14)°. Whereas one of the C—S—C—C torsion angles corresponds to a staggered conformation, the other is gauche.

Related literature

For related structures, see: Awaleh *et al.* (2005); Xie *et al.* (2005).



Experimental

Crystal data

$C_{21}H_{18}N_6S_2$
M_r = 418.55
Triclinic, $P\overline{1}$
a = 9.986 (3) Å
b = 10.057 (3) Å
c = 10.645 (3) Å
α = 98.972 (5)°
β = 90.688 (5)°
γ = 112.632 (5)°
V = 971.6 (5) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.29 mm^{-1}
T = 291 (2) K
0.30 × 0.20 × 0.20 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
T_{min} = 0.915, T_{max} = 0.943
5163 measured reflections
3720 independent reflections
2986 reflections with $I > 2\sigma(I)$
R_{int} = 0.028

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.059
$wR(F^2)$ = 0.156
S = 1.02
3720 reflections
262 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.52 e Å^{-3}
$\Delta\rho_{min}$ = −0.46 e Å^{-3}

Data collection: *SMART* (Bruker, 2000); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 2000); program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

The authors thank the Program for Excellent Talents Introduction of Southeast University for financial support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT2695).

References

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supporting information

Acta Cryst. (2008). E**64**, o901 [doi:10.1107/S1600536808010854]

1,3-Bis[4-(4-pyridyl)pyrimidin-2-ylsulfanyl]propane

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S1. Comment

In the title compound, the dihedral angles between the aromatic rings in the two(4-pyridyl)-pyrimidine residues are 23.45 (13)° and 2.67 (14)°. Whereas one of the C-S-C-C torsion angles adopts a staggered conformation [-176.74 (17)°], the other one is gauche [87.6 (2)°].

S2. Experimental

NaOH (0.80 g, 20 mmol) in ethanol (10 mL) was added with stirring to a solution of 4-(pyridin-4-yl)pyrimidine-2-thiol (3.78 g, 20 mmol) in acetone (30 mL) at ambient temperature for 20 min. A solution of 1,3-dibromopropane (2.00 g, 10.0 mmol) was added slowly over 1 h, and the resulting mixture was further refluxed for 6 h. The solids isolated from the reaction mixture were washed with water and acetone. The yellow products were obtained by vacuum dryness in 91% yield (3.80 g). The filtrate was allowed to evaporate slowly at room temperature. After several days, yellow block shaped crystals suitable for X-ray diffraction analyses were obtained.

S3. Refinement

H atoms were positioned geometrically and refined using a riding model, with C—H = 0.93 and 0.97 Å for $C_{aromatic}$ and $C_{methylene}$, respectively, and with $U_{iso}(H) = 1.2U_{eq}(C)$.

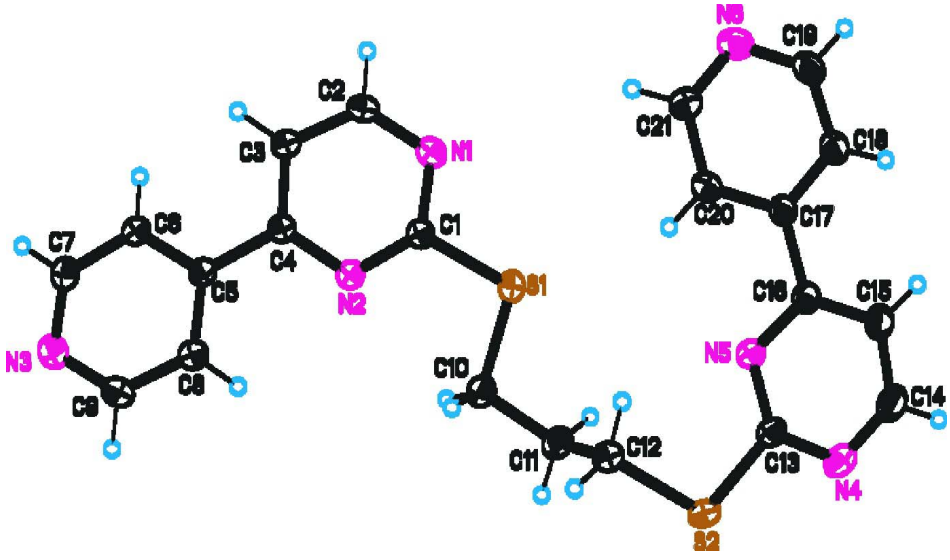

Figure 1

The molecular structure of the title compound.

1,3-Bis[4-(4-pyridyl)pyrimidin-2-ylsulfanyl]propane

Crystal data

$C_{21}H_{18}N_6S_2$
$M_r = 418.55$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 9.986$ (3) Å
$b = 10.057$ (3) Å
$c = 10.645$ (3) Å
$\alpha = 98.972$ (5)°
$\beta = 90.688$ (5)°
$\gamma = 112.632$ (5)°
$V = 971.6$ (5) Å^3
$Z = 2$
$F(000) = 436.0$
$D_x = 1.431$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 765 reflections
$\theta = 2.5$–$28.0°$
$\mu = 0.30$ mm^{-1}
$T = 291$ K
Block, yellow
0.30 × 0.20 × 0.20 mm

Data collection

Bruker CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
$T_{min} = 0.915$, $T_{max} = 0.943$
5163 measured reflections
3720 independent reflections
2986 reflections with $I > 2\sigma(I)$
$R_{int} = 0.028$
$\theta_{max} = 26.0°$, $\theta_{min} = 1.9°$
$h = -12 \rightarrow 11$
$k = -12 \rightarrow 10$
$l = -12 \rightarrow 13$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.059$
$wR(F^2) = 0.156$
$S = 1.02$
3720 reflections
262 parameters
0 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
$w = 1/[\sigma^2(F_o^2) + (0.1031P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.52$ e Å^{-3}
$\Delta\rho_{min} = -0.46$ e Å^{-3}

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 $F^2 > \sigma(F^2)$ 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	0.66814 (7)	0.18543 (8)	0.02844 (6)	0.0501 (2)
S2	0.31122 (8)	0.33035 (8)	0.24761 (7)	0.0551 (2)
N1	0.9037 (2)	0.1412 (2)	−0.0039 (2)	0.0484 (5)

N2	0.88209 (19)	0.3272 (2)	−0.10815 (17)	0.0362 (4)
N3	1.1505 (2)	0.6455 (2)	−0.4191 (2)	0.0517 (6)
N4	0.1388 (2)	0.1026 (2)	0.3337 (2)	0.0516 (5)
N5	0.3748 (2)	0.1065 (2)	0.29548 (17)	0.0394 (5)
N6	0.6716 (3)	−0.1928 (3)	0.3320 (2)	0.0558 (6)
C1	0.8365 (2)	0.2254 (3)	−0.0352 (2)	0.0374 (5)
C2	1.0326 (3)	0.1682 (3)	−0.0486 (2)	0.0488 (6)
H2	1.0859	0.1158	−0.0270	0.059*
C3	1.0916 (3)	0.2709 (3)	−0.1260 (2)	0.0449 (6)
H3	1.1826	0.2879	−0.1565	0.054*
C4	1.0107 (2)	0.3474 (2)	−0.1565 (2)	0.0346 (5)
C5	1.0584 (2)	0.4510 (2)	−0.2471 (2)	0.0355 (5)
C6	1.1556 (3)	0.4399 (3)	−0.3360 (2)	0.0438 (6)
H6	1.1925	0.3676	−0.3397	0.053*
C7	1.1961 (3)	0.5387 (3)	−0.4185 (3)	0.0520 (7)
H7	1.2605	0.5294	−0.4784	0.062*
C8	1.0070 (3)	0.5595 (3)	−0.2489 (2)	0.0423 (6)
H8	0.9392	0.5687	−0.1928	0.051*
C9	1.0560 (3)	0.6538 (3)	−0.3333 (3)	0.0504 (6)
H9	1.0216	0.7279	−0.3311	0.061*
C10	0.6054 (3)	0.3160 (3)	−0.0212 (2)	0.0454 (6)
H10A	0.6765	0.4146	0.0080	0.055*
H10B	0.5920	0.2998	−0.1135	0.055*
C11	0.4614 (3)	0.2964 (3)	0.0364 (2)	0.0475 (6)
H11A	0.4109	0.3412	−0.0101	0.057*
H11B	0.4011	0.1928	0.0263	0.057*
C12	0.4802 (3)	0.3627 (3)	0.1758 (2)	0.0446 (6)
H12A	0.5352	0.4674	0.1852	0.054*
H12B	0.5367	0.3228	0.2213	0.054*
C13	0.2733 (3)	0.1608 (3)	0.2964 (2)	0.0416 (5)
C14	0.1092 (3)	−0.0215 (3)	0.3770 (2)	0.0519 (7)
H14	0.0168	−0.0673	0.4037	0.062*
C15	0.2062 (3)	−0.0862 (3)	0.3847 (2)	0.0464 (6)
H15	0.1824	−0.1720	0.4179	0.056*
C16	0.3416 (3)	−0.0181 (2)	0.3409 (2)	0.0373 (5)
C17	0.4557 (3)	−0.0781 (2)	0.3402 (2)	0.0373 (5)
C18	0.4364 (3)	−0.2049 (3)	0.3858 (2)	0.0491 (6)
H18	0.3500	−0.2550	0.4204	0.059*
C19	0.5447 (3)	−0.2568 (3)	0.3800 (2)	0.0546 (7)
H19	0.5286	−0.3423	0.4117	0.066*
C20	0.5885 (3)	−0.0082 (3)	0.2938 (2)	0.0443 (6)
H20	0.6092	0.0798	0.2650	0.053*
C21	0.6903 (3)	−0.0697 (3)	0.2905 (3)	0.0536 (7)
H21	0.7781	−0.0217	0.2569	0.064*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0461 (4)	0.0571 (4)	0.0600 (4)	0.0260 (3)	0.0245 (3)	0.0303 (3)
S2	0.0617 (5)	0.0605 (4)	0.0689 (5)	0.0449 (4)	0.0295 (4)	0.0283 (4)
N1	0.0516 (13)	0.0521 (12)	0.0558 (13)	0.0287 (10)	0.0163 (10)	0.0272 (10)
N2	0.0346 (10)	0.0392 (10)	0.0392 (10)	0.0173 (8)	0.0077 (8)	0.0118 (8)
N3	0.0481 (13)	0.0537 (13)	0.0615 (14)	0.0215 (10)	0.0138 (10)	0.0283 (11)
N4	0.0447 (12)	0.0597 (14)	0.0598 (13)	0.0287 (11)	0.0183 (10)	0.0142 (11)
N5	0.0438 (11)	0.0451 (11)	0.0400 (10)	0.0264 (9)	0.0124 (8)	0.0134 (8)
N6	0.0701 (16)	0.0584 (13)	0.0590 (14)	0.0434 (12)	0.0147 (12)	0.0192 (11)
C1	0.0389 (12)	0.0410 (12)	0.0381 (12)	0.0193 (10)	0.0083 (10)	0.0132 (10)
C2	0.0470 (14)	0.0536 (15)	0.0620 (16)	0.0306 (12)	0.0109 (12)	0.0270 (12)
C3	0.0391 (13)	0.0505 (14)	0.0575 (15)	0.0257 (11)	0.0139 (11)	0.0226 (12)
C4	0.0326 (11)	0.0355 (11)	0.0385 (12)	0.0148 (9)	0.0052 (9)	0.0103 (9)
C5	0.0296 (11)	0.0382 (12)	0.0414 (12)	0.0145 (9)	0.0036 (9)	0.0113 (9)
C6	0.0381 (13)	0.0482 (14)	0.0553 (14)	0.0232 (11)	0.0144 (11)	0.0206 (11)
C7	0.0458 (15)	0.0595 (16)	0.0604 (16)	0.0242 (13)	0.0210 (12)	0.0277 (13)
C8	0.0409 (13)	0.0445 (13)	0.0485 (13)	0.0219 (11)	0.0099 (10)	0.0139 (11)
C9	0.0516 (15)	0.0439 (14)	0.0654 (16)	0.0244 (12)	0.0104 (13)	0.0218 (12)
C10	0.0450 (14)	0.0548 (15)	0.0446 (13)	0.0251 (12)	0.0125 (11)	0.0164 (11)
C11	0.0392 (13)	0.0628 (16)	0.0494 (14)	0.0265 (12)	0.0092 (11)	0.0172 (12)
C12	0.0447 (14)	0.0477 (13)	0.0508 (14)	0.0251 (11)	0.0122 (11)	0.0160 (11)
C13	0.0477 (14)	0.0485 (13)	0.0392 (12)	0.0294 (11)	0.0123 (10)	0.0097 (10)
C14	0.0455 (15)	0.0552 (15)	0.0556 (15)	0.0197 (12)	0.0189 (12)	0.0098 (12)
C15	0.0501 (15)	0.0438 (13)	0.0455 (14)	0.0172 (12)	0.0153 (11)	0.0109 (11)
C16	0.0441 (13)	0.0399 (12)	0.0309 (11)	0.0200 (10)	0.0073 (9)	0.0057 (9)
C17	0.0488 (14)	0.0389 (12)	0.0299 (11)	0.0226 (10)	0.0091 (9)	0.0077 (9)
C18	0.0627 (16)	0.0483 (14)	0.0488 (14)	0.0303 (13)	0.0199 (12)	0.0201 (11)
C19	0.080 (2)	0.0489 (15)	0.0537 (15)	0.0400 (14)	0.0211 (14)	0.0233 (12)
C20	0.0506 (15)	0.0425 (13)	0.0508 (14)	0.0256 (11)	0.0134 (11)	0.0193 (11)
C21	0.0560 (16)	0.0590 (16)	0.0627 (16)	0.0353 (14)	0.0207 (13)	0.0242 (13)

Geometric parameters (Å, º)

S1—C1	1.743 (2)	C7—H7	0.9300
S1—C10	1.799 (3)	C8—C9	1.365 (3)
S2—C13	1.762 (3)	C8—H8	0.9300
S2—C12	1.796 (2)	C9—H9	0.9300
N1—C2	1.320 (3)	C10—C11	1.525 (3)
N1—C1	1.342 (3)	C10—H10A	0.9700
N2—C1	1.325 (3)	C10—H10B	0.9700
N2—C4	1.343 (3)	C11—C12	1.507 (3)
N3—C7	1.320 (3)	C11—H11A	0.9700
N3—C9	1.338 (3)	C11—H11B	0.9700
N4—C14	1.325 (3)	C12—H12A	0.9700
N4—C13	1.336 (3)	C12—H12B	0.9700
N5—C13	1.323 (3)	C14—C15	1.367 (4)

N5—C16	1.337 (3)	C14—H14	0.9300
N6—C21	1.326 (3)	C15—C16	1.385 (3)
N6—C19	1.330 (4)	C15—H15	0.9300
C2—C3	1.379 (3)	C16—C17	1.480 (3)
C2—H2	0.9300	C17—C18	1.381 (3)
C3—C4	1.379 (3)	C17—C20	1.380 (3)
C3—H3	0.9300	C18—C19	1.368 (4)
C4—C5	1.478 (3)	C18—H18	0.9300
C5—C8	1.376 (3)	C19—H19	0.9300
C5—C6	1.386 (3)	C20—C21	1.377 (4)
C6—C7	1.376 (3)	C20—H20	0.9300
C6—H6	0.9300	C21—H21	0.9300

C1—S1—C10	103.68 (11)	H10A—C10—H10B	108.4
C13—S2—C12	102.41 (11)	C12—C11—C10	113.0 (2)
C2—N1—C1	114.9 (2)	C12—C11—H11A	109.0
C1—N2—C4	115.73 (19)	C10—C11—H11A	109.0
C7—N3—C9	115.9 (2)	C12—C11—H11B	109.0
C14—N4—C13	114.3 (2)	C10—C11—H11B	109.0
C13—N5—C16	116.7 (2)	H11A—C11—H11B	107.8
C21—N6—C19	115.5 (2)	C11—C12—S2	113.52 (17)
N2—C1—N1	127.7 (2)	C11—C12—H12A	108.9
N2—C1—S1	119.77 (17)	S2—C12—H12A	108.9
N1—C1—S1	112.50 (17)	C11—C12—H12B	108.9
N1—C2—C3	122.8 (2)	S2—C12—H12B	108.9
N1—C2—H2	118.6	H12A—C12—H12B	107.7
C3—C2—H2	118.6	N5—C13—N4	127.3 (2)
C4—C3—C2	117.5 (2)	N5—C13—S2	119.94 (18)
C4—C3—H3	121.2	N4—C13—S2	112.72 (18)
C2—C3—H3	121.2	N4—C14—C15	124.0 (2)
N2—C4—C3	121.2 (2)	N4—C14—H14	118.0
N2—C4—C5	116.95 (19)	C15—C14—H14	118.0
C3—C4—C5	121.8 (2)	C14—C15—C16	116.9 (2)
C8—C5—C6	117.4 (2)	C14—C15—H15	121.6
C8—C5—C4	122.0 (2)	C16—C15—H15	121.6
C6—C5—C4	120.6 (2)	N5—C16—C15	120.7 (2)
C7—C6—C5	118.4 (2)	N5—C16—C17	116.6 (2)
C7—C6—H6	120.8	C15—C16—C17	122.7 (2)
C5—C6—H6	120.8	C18—C17—C20	116.4 (2)
N3—C7—C6	124.9 (2)	C18—C17—C16	122.6 (2)
N3—C7—H7	117.6	C20—C17—C16	121.0 (2)
C6—C7—H7	117.6	C19—C18—C17	119.9 (2)
C9—C8—C5	119.8 (2)	C19—C18—H18	120.1
C9—C8—H8	120.1	C17—C18—H18	120.1
C5—C8—H8	120.1	N6—C19—C18	124.3 (2)
N3—C9—C8	123.7 (2)	N6—C19—H19	117.8
N3—C9—H9	118.1	C18—C19—H19	117.8
C8—C9—H9	118.1	C21—C20—C17	119.5 (2)

C11—C10—S1	108.09 (17)	C21—C20—H20	120.3
C11—C10—H10A	110.1	C17—C20—H20	120.3
S1—C10—H10A	110.1	N6—C21—C20	124.4 (2)
C11—C10—H10B	110.1	N6—C21—H21	117.8
S1—C10—H10B	110.1	C20—C21—H21	117.8