Crystal Structure and Molecular Mechanics Modeling of 2-(4-Amino-3-benzyl-2-thioxo-2,3-dihydrothiazol-5-yl)benzoxazole

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Abstract

Introduction

Experimental

Synthesis and crystallization

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1. Changes in illuminated volume were kept to a minimum, and were taken into account (Görbitz, 1999) by the multi-scan inter-frame scaling (DENZO/SCALEPACK, Otwinowski & Minor, 1997).

Görbitz, C. H. (1999). Acta Cryst. B55, 1090-1098.

The H atoms were all located in a difference map, but those attached to carbon atoms were repositioned geometrically. The H atoms were initially refined with soft restraints on the bond lengths and angles to regularise their geometry (C—H in the range 0.93–0.98, N—H in the range 0.86–0.89 N—H to 0.86 O—H = 0.82 Å) and U_{iso}(H) (in the range 1.2-1.5 times U_{eq} of the parent atom), after which the positions were refined with riding constraints (Cooper *et al.*, 2010).

Cooper, R. I., Thompson, A. L. & Watkin, D. J. (2010). J. Appl. Cryst. 43, 1100-1107.

Results and discussion

Computing details

Data collection: *COLLECT* (Nonius, 2001).; cell refinement: *DENZO*/*SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO*/*SCALEPACK* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *CRYSTALS* (Betteridge *et al.*, 2003); molecular graphics: *CAMERON* (Watkin *et al.*, 1996); software used to prepare material for publication: *CRYSTALS* (Betteridge *et al.*, 2003).

References

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Betteridge, P. W., Carruthers, J. R., Cooper, R. I., Prout, K. & Watkin, D. J. (2003). *J. Appl. Cryst.* **36**, 1487.

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(1)

Crystal data

$C_{17}H_{13}N_3OS_2$
M_r = 339.44
Triclinic, *P*1
Hall symbol: P 1
a = 5.1738 (3) Å
b = 6.4111 (6) Å
c = 12.3694 (10) Å
α = 86.021 (4)°
β = 84.384 (5)°
γ = 77.191 (5)°
V = 397.68 (5) Å^3
Z = 1
F(000) = 176
D_x = 1.417 Mg m^{-3}
Mo *K*α radiation, λ = 0.71073 Å
Cell parameters from 3630 reflections
θ = 3–27°
μ = 0.34 mm^{-1}
T = 298 K
Cube, colourless
× × mm

Data collection

Nonius KappaCCD diffractometer
Graphite monochromator
φ & ω scans
Absorption correction: multi-scan *DENZO*/*SCALEPACK* (Otwinowski & Minor, 1997)
T_{min} = 1.00, T_{max} = 1.00
2530 measured reflections
1761 independent reflections
1644 reflections with $I > 2.0\sigma(I)$
R_{int} = 0.025
θ_{max} = 27.4°, θ_{min} = 3.3°
h = −5→6
k = −7→8
l = −14→16

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.030
$wR(F^2)$ = 0.073
S = 1.00
1644 reflections
209 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Hydrogen site location: difference Fourier map
H-atom parameters constrained
Method, part 1, Chebychev polynomial, (Watkin, 1994, *P*rince, 1982) [*w*eight] = 1.0/[A_0*T_0(x) + A_1*T_1(x) ⋯ + A_{n-1}]*T_{n-1}(x)] where A_i are the Chebychev coefficients listed below and x = *F*/*F*max Method = Robust Weighting (*P*rince, 1982) W = [*w*eight] * [1-(delta*F*/6*sigma*F*)^2]^2 A_i are: 40.9 61.0 42.5 16.6 7.38
$(\Delta/\sigma)_{max}$ = 0.0001
$\Delta\rho_{max}$ = 0.15 e Å^{-3}
$\Delta\rho_{min}$ = −0.14 e Å^{-3}
Absolute structure: Flack (1983), 0 Friedel-pairs
Absolute structure parameter: 0.53 (17)

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

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.48152 (19)	0.65929 (16)	0.99348 (14)	0.0416
S2	0.1501 (2)	0.65613 (17)	0.80895 (14)	0.0495
N1	0.8611 (6)	0.0685 (4)	0.9690 (3)	0.0542
N2	1.0837 (6)	0.2530 (5)	1.1338 (2)	0.0526

N3	0.5214 (5)	0.3265 (3)	0.8880 (2)	0.0382
O1	0.8927 (5)	0.6032 (4)	1.15943 (19)	0.0496
C1	1.0941 (6)	0.5417 (6)	1.2296 (3)	0.0531
C2	1.1719 (9)	0.6668 (9)	1.3000 (3)	0.0735
C3	1.3812 (9)	0.5647 (10)	1.3596 (3)	0.0853
C4	1.5020 (9)	0.3539 (10)	1.3469 (3)	0.0843
C5	1.4219 (8)	0.2272 (8)	1.2731 (4)	0.0743
C6	1.2103 (7)	0.3280 (6)	1.2132 (3)	0.0545
C7	0.9041 (6)	0.4200 (5)	1.1062 (2)	0.0433
C8	0.7178 (6)	0.4324 (5)	1.0270 (2)	0.0395
C9	0.7105 (6)	0.2686 (4)	0.9621 (2)	0.0391
C10	0.3805 (5)	0.5355 (4)	0.8914 (2)	0.0374
C11	0.4649 (6)	0.1784 (5)	0.8126 (3)	0.0449
C12	0.6625 (5)	0.1424 (4)	0.7137 (2)	0.0380
C13	0.6838 (7)	−0.0397 (5)	0.6573 (3)	0.0507
C14	0.8638 (8)	−0.0806 (6)	0.5660 (3)	0.0624
C15	1.0229 (8)	0.0602 (7)	0.5314 (3)	0.0619
C16	0.9986 (8)	0.2443 (7)	0.5867 (3)	0.0614
C17	0.8192 (6)	0.2853 (5)	0.6771 (3)	0.0500
H111	0.4673	0.0442	0.8507	0.0568*
H112	0.2929	0.2345	0.7888	0.0568*
H171	0.8007	0.4134	0.7141	0.0593*
H161	1.1083	0.3423	0.5629	0.0745*
H151	1.1464	0.0313	0.4694	0.0757*
H141	0.8774	−0.2065	0.5277	0.0751*
H131	0.5758	−0.1384	0.6820	0.0594*
H51	1.5082	0.0816	1.2642	0.0925*
H41	1.6438	0.2902	1.3900	0.0966*
H31	1.4444	0.6407	1.4110	0.1064*
H21	1.0895	0.8137	1.3068	0.0884*
H12	0.9863	0.0318	1.0222	0.0656*
H11	0.8431	−0.0356	0.9208	0.0656*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0466 (4)	0.0338 (3)	0.0439 (4)	−0.0052 (3)	−0.0041 (3)	−0.0085 (3)
S2	0.0508 (5)	0.0410 (4)	0.0569 (5)	−0.0051 (3)	−0.0146 (4)	−0.0051 (3)
N1	0.0583 (16)	0.0371 (12)	0.0635 (17)	0.0019 (11)	−0.0116 (13)	−0.0071 (11)
N2	0.0478 (15)	0.0513 (15)	0.0551 (16)	−0.0043 (12)	−0.0064 (12)	0.0034 (13)
N3	0.0390 (12)	0.0317 (11)	0.0426 (12)	−0.0064 (9)	0.0024 (9)	−0.0057 (9)
O1	0.0491 (12)	0.0563 (13)	0.0436 (11)	−0.0089 (10)	−0.0060 (9)	−0.0087 (10)
C1	0.0477 (17)	0.077 (2)	0.0361 (15)	−0.0165 (16)	−0.0043 (13)	−0.0010 (14)
C2	0.073 (2)	0.103 (3)	0.051 (2)	−0.030 (2)	−0.0078 (18)	−0.014 (2)
C3	0.070 (3)	0.140 (5)	0.054 (2)	−0.035 (3)	−0.015 (2)	−0.009 (3)
C4	0.057 (2)	0.147 (5)	0.055 (2)	−0.037 (3)	−0.0201 (19)	0.022 (3)
C5	0.052 (2)	0.099 (3)	0.068 (2)	−0.016 (2)	−0.0104 (18)	0.028 (2)
C6	0.0437 (16)	0.074 (2)	0.0445 (16)	−0.0142 (15)	−0.0023 (13)	0.0105 (15)
C7	0.0439 (16)	0.0483 (16)	0.0374 (15)	−0.0119 (13)	0.0016 (12)	−0.0004 (12)
C8	0.0392 (14)	0.0379 (14)	0.0395 (14)	−0.0053 (11)	−0.0014 (11)	−0.0012 (11)
C9	0.0401 (14)	0.0309 (13)	0.0447 (15)	−0.0064 (11)	0.0013 (12)	−0.0010 (11)

C10	0.0372 (14)	0.0323 (13)	0.0425 (14)	−0.0084 (11)	0.0008 (11)	−0.0029 (11)
C11	0.0481 (15)	0.0367 (13)	0.0529 (17)	−0.0145 (12)	0.0003 (13)	−0.0123 (12)
C12	0.0381 (13)	0.0350 (13)	0.0411 (14)	−0.0049 (10)	−0.0104 (11)	−0.0036 (11)
C13	0.064 (2)	0.0411 (16)	0.0492 (18)	−0.0143 (15)	−0.0060 (15)	−0.0091 (13)
C14	0.077 (2)	0.059 (2)	0.0503 (18)	−0.0077 (18)	−0.0028 (17)	−0.0195 (15)
C15	0.0586 (19)	0.077 (2)	0.0465 (18)	−0.0073 (18)	0.0001 (15)	−0.0104 (16)
C16	0.059 (2)	0.076 (2)	0.0531 (19)	−0.0268 (18)	0.0004 (16)	−0.0012 (17)
C17	0.0534 (17)	0.0472 (16)	0.0536 (17)	−0.0185 (13)	−0.0023 (14)	−0.0103 (13)

Geometric parameters (Å, º) for (1)

S1—C8	1.735 (3)	C4—H41	0.950
S1—C10	1.717 (3)	C5—C6	1.394 (5)
S2—C10	1.667 (3)	C5—H51	0.950
N1—C9	1.347 (4)	C7—C8	1.427 (4)
N1—H12	0.950	C8—C9	1.375 (4)
N1—H11	0.950	C11—C12	1.511 (4)
N2—C6	1.399 (5)	C11—H111	0.950
N2—C7	1.302 (4)	C11—H112	0.950
N3—C9	1.380 (4)	C12—C13	1.380 (4)
N3—C10	1.377 (3)	C12—C17	1.379 (4)
N3—C11	1.468 (4)	C13—C14	1.392 (5)
O1—C1	1.393 (4)	C13—H131	0.950
O1—C7	1.373 (4)	C14—C15	1.372 (6)
C1—C2	1.368 (6)	C14—H141	0.950
C1—C6	1.389 (5)	C15—C16	1.380 (6)
C2—C3	1.386 (7)	C15—H151	0.950
C2—H21	0.950	C16—C17	1.382 (5)
C3—C4	1.370 (8)	C16—H161	0.950
C3—H31	0.950	C17—H171	0.950
C4—C5	1.411 (7)		
C8—S1—C10	92.35 (14)	S1—C8—C9	110.3 (2)
C9—N1—H12	119.7	N3—C9—C8	112.8 (2)
C9—N1—H11	120.3	N3—C9—N1	121.4 (3)
H12—N1—H11	120.0	C8—C9—N1	125.8 (3)
C6—N2—C7	103.4 (3)	N3—C10—S1	109.7 (2)
C9—N3—C10	114.8 (2)	N3—C10—S2	126.2 (2)
C9—N3—C11	123.6 (2)	S1—C10—S2	124.09 (16)
C10—N3—C11	121.5 (3)	N3—C11—C12	113.9 (2)
C1—O1—C7	103.2 (3)	N3—C11—H111	108.3
O1—C1—C2	127.3 (4)	C12—C11—H111	108.2
O1—C1—C6	107.3 (3)	N3—C11—H112	108.5
C2—C1—C6	125.3 (4)	C12—C11—H112	108.4
C1—C2—C3	115.3 (5)	H111—C11—H112	109.5
C1—C2—H21	122.3	C11—C12—C13	118.5 (3)
C3—C2—H21	122.4	C11—C12—C17	122.7 (2)
C2—C3—C4	121.7 (4)	C13—C12—C17	118.9 (3)
C2—C3—H31	120.1	C12—C13—C14	120.6 (3)
C4—C3—H31	118.2	C12—C13—H131	119.2
C3—C4—C5	122.5 (4)	C14—C13—H131	120.2

C3—C4—H41	118.7	C13—C14—C15	120.2 (3)
C5—C4—H41	118.8	C13—C14—H141	120.0
C4—C5—C6	116.3 (5)	C15—C14—H141	119.8
C4—C5—H51	121.9	C14—C15—C16	119.3 (3)
C6—C5—H51	121.8	C14—C15—H151	119.9
N2—C6—C5	131.5 (4)	C16—C15—H151	120.8
N2—C6—C1	109.7 (3)	C15—C16—C17	120.5 (3)
C5—C6—C1	118.9 (4)	C15—C16—H161	119.6
O1—C7—N2	116.4 (3)	C17—C16—H161	119.9
O1—C7—C8	116.7 (3)	C16—C17—C12	120.5 (3)
N2—C7—C8	126.9 (3)	C16—C17—H171	120.2
C7—C8—S1	125.1 (2)	C12—C17—H171	119.2
C7—C8—C9	124.5 (3)		

Hydrogen-bond geometry (Å, º) for (1)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H12···N2	0.95	2.21	2.877 (5)	126 (1)