Crystal structure of (*Z*)-2-amino-4-(2-amino-2-(4-bromophenylthio)-1-cyanovinyl)-6-bromo-4*H*-chromene-3-carbonitrile, $C_{19}H_{12}Br_2N_4OS$

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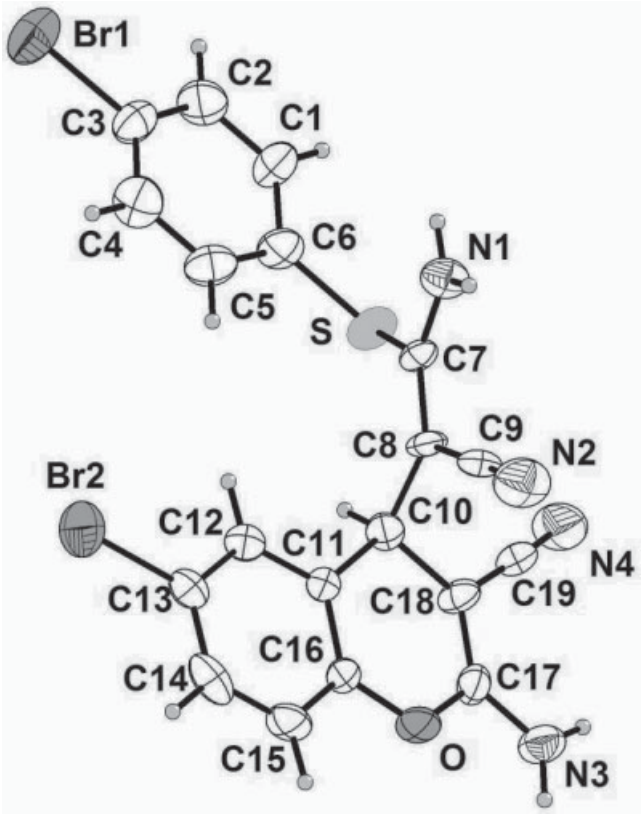

Abstract

$C_{19}H_{12}Br_2N_4OS$, triclinic, $P\overline{1}$ (no. 2), a = 9.536(2) Å, b = 9.977(2) Å, c = 10.160(2) Å, α = 84.16(3)°, β = 81.34(3)°, γ = 86.25(3)°, V = 949.4 Å^3, Z = 2, $R_{gt}(F)$ = 0.0680, $wR_{ref}(F^2)$ = 0.1198, T = 293 K.

Table 1. Data collection and handling.

Crystal:	colourless blocks, size 0.10×0.10×0.20 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	43.96 cm^{-1}
Diffractometer, scan mode:	Nonius CAD4, $\omega/2\theta$
$2\theta_{max}$:	50.74°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	3708, 3483
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1726
$N(param)_{refined}$:	244
Programs:	SHELX [2]

Source of material

5-bromo-2-hydroxybenzaldehyde (5.0 mmol, 2.02 g), malononitrile (10.1 mmol, 0.67 g) and 4-bromobenzenethiol (5.0 mmol, 0.97 g) were dissolved in ethanol (20 mL). Piperidine (0.5 mmol, 0.05 mL) was added to the solution and the reaction mixture stired at room temperature for 1.5h. The solution was filtered, and a white solid was obtained (2.16 g, 85.7% yield). Recrystallization from a tetrahydrofuran solution at room temperature yielded crystals suitable for single-crystal X-ray diffraction.

Experimental details

H atoms were positioned geometrically with C–H = 0.93, 0.98 and N–H = 0.86 Å for aromatic, methyl, and amino groups, respectively, and riding on their parent atoms, with U_{iso}(H) = 1.2 (or 1.5 for methyl groups) times U_{eq}(C).

Discussion

(*Z*)-2-amino-4-(2-amino-2-(4-bromophenylthio)-1-cyanovinyl)-6-bromo-4*H*-chromene-3-carbonitrile is an important derivative of Chromene [1]. In the ctystal structure, intermolecular N–H···N hydrogen bonds link the molecules into chains, in which may be effective in the stabilization of the structure.

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(1A)	2*i*	0.7213	0.4059	–0.0713	0.052
H(1B)	2*i*	0.8311	0.3241	–0.0041	0.052
H(1C)	2*i*	1.0502	0.2158	0.2455	0.060
H(2A)	2*i*	1.0005	0.0083	0.3558	0.067
H(3A)	2*i*	0.7837	1.0887	0.0358	0.056
H(3B)	2*i*	0.6357	1.1466	0.0662	0.056
H(4A)	2*i*	0.5900	0.1178	0.4191	0.066
H(5A)	2*i*	0.6398	0.3300	0.3159	0.062
H(10A)	2*i*	0.7196	0.6696	0.2555	0.040
H(12A)	2*i*	0.4875	0.5344	0.3091	0.048
H(14A)	2*i*	0.1306	0.7655	0.3552	0.065
H(15A)	2*i*	0.2508	0.9449	0.2457	0.050

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Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
S	2*i*	0.8979(2)	0.4586(2)	0.1939(2)	0.041(1)	0.038(1)	0.063(1)	0.0023(9)	–0.020(1)	0.005(1)
O	2*i*	0.5080(5)	0.9618(4)	0.1595(5)	0.027(3)	0.036(3)	0.065(4)	0.010(2)	0.005(2)	0.001(2)
Br(1)	2*i*	0.7471(1)	–0.12684(9)	0.48594(9)	0.0903(8)	0.0545(6)	0.0667(7)	–0.0120(5)	–0.0243(5)	0.0138(4)
N(1)	2*i*	0.7761(5)	0.3955(5)	–0.0107(5)	0.035(4)	0.046(4)	0.050(4)	0.010(3)	–0.014(3)	–0.007(3)
C(1)	2*i*	0.9571(8)	0.1979(7)	0.2820(7)	0.055(5)	0.038(5)	0.056(5)	–0.006(4)	–0.009(4)	0.007(4)
Br(2)	2*i*	0.19446(9)	0.48168(9)	0.4242(1)	0.0556(6)	0.0674(6)	0.0765(7)	–0.0157(5)	0.0174(5)	0.0039(5)
C(2)	2*i*	0.9275(8)	0.0736(8)	0.3467(8)	0.044(5)	0.058(6)	0.061(6)	0.017(4)	–0.009(4)	0.000(4)
N(2)	2*i*	0.5575(7)	0.6746(7)	–0.1214(7)	0.070(5)	0.066(5)	0.061(5)	0.016(4)	–0.026(4)	–0.007(4)
C(3)	2*i*	0.7910(9)	0.0457(7)	0.3977(7)	0.055(5)	0.037(5)	0.045(5)	–0.001(4)	–0.015(4)	0.003(4)
N(3)	2*i*	0.6959(6)	1.0785(5)	0.0677(6)	0.041(4)	0.035(4)	0.062(4)	0.005(3)	–0.005(3)	0.000(3)
C(4)	2*i*	0.6831(8)	0.1385(8)	0.3857(8)	0.039(5)	0.064(6)	0.063(6)	–0.003(4)	–0.011(4)	–0.002(5)
N(4)	2*i*	1.0038(7)	0.8392(6)	0.0645(7)	0.036(4)	0.057(5)	0.098(6)	0.003(4)	0.009(4)	–0.004(4)
C(5)	2*i*	0.7135(8)	0.2651(8)	0.3228(8)	0.049(5)	0.043(5)	0.063(6)	0.010(4)	–0.017(4)	–0.004(4)
C(6)	2*i*	0.8502(8)	0.2977(7)	0.2701(7)	0.042(5)	0.043(5)	0.048(5)	0.013(4)	–0.016(4)	0.002(4)
C(7)	2*i*	0.7756(6)	0.4928(6)	0.0756(6)	0.026(4)	0.023(3)	0.036(4)	0.005(3)	–0.010(3)	0.003(3)
C(8)	2*i*	0.6965(6)	0.6060(6)	0.0755(7)	0.035(4)	0.016(3)	0.044(4)	0.005(3)	–0.005(3)	–0.003(3)
C(9)	2*i*	0.6187(8)	0.6413(7)	–0.0355(8)	0.041(5)	0.027(4)	0.048(5)	0.014(3)	0.001(4)	–0.009(4)
C(10)	2*i*	0.6716(6)	0.7069(6)	0.1801(6)	0.028(4)	0.037(4)	0.034(4)	0.005(3)	–0.007(3)	0.000(3)
C(11)	2*i*	0.5151(7)	0.7248(6)	0.2318(6)	0.037(4)	0.029(4)	0.034(4)	0.003(3)	0.004(3)	0.000(3)
C(12)	2*i*	0.4398(7)	0.6177(7)	0.2950(7)	0.039(5)	0.036(4)	0.042(5)	0.006(3)	–0.001(4)	–0.003(3)
C(13)	2*i*	0.2938(8)	0.6304(7)	0.3388(7)	0.038(5)	0.045(5)	0.047(5)	0.005(4)	0.012(4)	–0.005(4)
C(14)	2*i*	0.2268(8)	0.7542(9)	0.3230(7)	0.034(5)	0.079(7)	0.045(5)	0.002(5)	0.009(4)	–0.017(4)
C(15)	2*i*	0.2991(7)	0.8620(7)	0.2606(7)	0.027(4)	0.043(5)	0.053(5)	0.010(3)	–0.002(4)	–0.007(4)
C(16)	2*i*	0.4459(7)	0.8482(6)	0.2188(6)	0.038(4)	0.026(4)	0.032(4)	–0.005(3)	0.001(3)	–0.003(3)
C(17)	2*i*	0.6528(7)	0.9567(7)	0.1198(6)	0.045(5)	0.032(4)	0.031(4)	–0.010(4)	–0.001(3)	0.001(3)
C(18)	2*i*	0.7312(7)	0.8413(6)	0.1302(7)	0.029(4)	0.022(4)	0.052(5)	–0.005(3)	–0.007(3)	–0.001(3)
C(19)	2*i*	0.8825(9)	0.8427(6)	0.0918(7)	0.061(6)	0.021(4)	0.048(5)	–0.001(4)	0.001(4)	0.000(3)

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