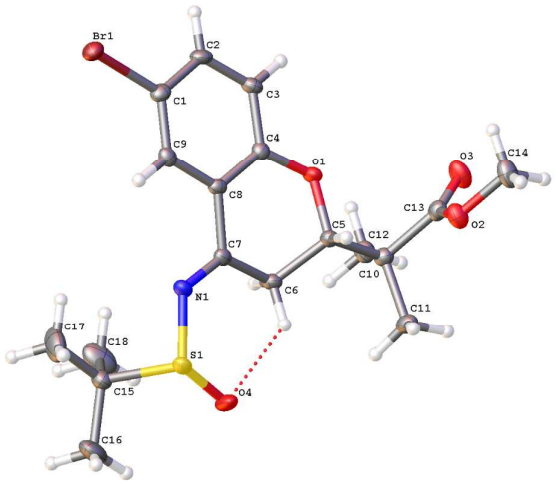
Submitted by: **Anita Mattson**
Ohio State
Solved by: **Charlotte L. Stern**
Sample ID: **cx0744**

Crystal Submitted on**: 2016-04-18**

Data Collected on: **2016-04-25**

Crystal Data and Experimental



Compound	**cx0744**
Formula	$C_{18}H_{24}BrNO_4S$
$D_{calc.}$/ g cm^{-3}	1.435
μ/mm^{-1}	3.974
Formula Weight	430.35
Colour	colourless
Shape	plate
Size/mm^3	0.36×0.23×0.11
T/K	100(2)
Crystal System	orthorhombic
Flack Parameter	-0.009(13)
Hooft Parameter	0.007(5)
Space Group	$P2_12_12_1$
a/Å	9.8294(8)
b/Å	10.8457(9)
c/Å	18.6856(15)
α/°	90
β/°	90
γ/°	90
V/Å^3	1992.0(3)
Z	4
Z'	1
Wavelength/Å	1.541838
Radiation type	CuK_α
Θ_{min}/°	4.714
Θ_{max}/°	67.881
Measured Refl.	19717
Independent Refl.	3557
Reflections Used	3541
R_{int}	0.0365
Parameters	232
Restraints	0
Largest Peak	0.207
Deepest Hole	-0.305
GooF	1.039
wR_2 (all data)	0.0515
wR_2	0.0515
R_1 (all data)	0.0213
R_1	0.0212

Experimental. Single colourless plate-shaped crystals of (**cx0744**) were recrystallised from a mixture of hexane and DCM by slow evaporation. A suitable crystal (0.36×0.23×0.11) mm^3 was selected and mounted on a glass fibre in Paratone oil on a Bruker Kappa APEX CCD area detector diffractometer. The crystal was kept at T = 100(2) K during data collection. Using **Olex2** (Dolomanov et al., 2009), the structure was solved with the **ShelXT** (Sheldrick, 2015) structure solution program, using the Direct Methods solution method. The model was refined with version of **ShelXL** (Sheldrick, 2015) using Least Squares minimisation.

Crystal Data. $C_{18}H_{24}BrNO_4S$, M_r = 430.35, orthorhombic, $P2_12_12_1$ (No. 19), a = 9.8294(8) Å, b = 10.8457(9) Å, c = 18.6856(15) Å, $\alpha = \beta = \gamma = 90°$, V = 1992.0(3) Å^3, T = 100(2) K, Z = 4, Z' = 1, $\mu(CuK_\alpha)$ = 3.974, 19717 reflections measured, 3557 unique (R_{int} = 0.0365) which were used in all calculations. The final wR_2 was 0.0515 (all data) and R_1 was 0.0212 (I > 2(I)).

Structure Quality Indicators

Reflections:	d min	0.83	I/σ	38.6	Rint	3.65%	complete	98%	
Refinement:	Shift	0.002	Max Peak	0.2	Min Peak	-0.3	GooF	1.039	-0.009(13)

A colourless plate-shaped crystal with dimensions 0.36×0.23×0.11 mm^3 was mounted on a glass fibre in Paratone oil. X-ray diffraction data were collected using a Bruker Kappa APEX CCD area detector diffractometer equipped with an Oxford Cryosystems low-temperature device, operating at T = 100(2) K.

Data were measured using ω and ϕ scans scans of 0.50 ° per frame for 5.00 s using CuK_α radiation (microsource, 45 kV, 1 mA). The total number of runs and images was based on the strategy calculation from the program **APEX2** (Bruker).The maximum resolution achieved was Θ = 67.881°

Cell parameters were retrieved using the **SAINT** (Bruker, V8.34A, 2013) software and refined using **SAINT** (Bruker) on 9113 reflections, 46 % of the observed reflections. Data reduction was performed using the **SAINT** (Bruker) software which corrects for Lorentz polarisation. The final completeness is 99.10 out to 67.881 in Θ. The absorption coefficient μ of this material is 3.974 at this wavelength (λ = 1.54178) and the minimum and maximum transmissions are 0.6020 and 0.7530.

The structure was solved in the space group $P2_12_12_1$ (# 19) by Direct Methods using the **ShelXT** (Sheldrick, 2015) structure solution program and refined by Least Squares using version of **ShelXL** (Sheldrick, 2015). All non-hydrogen atoms were refined anisotropically. Hydrogen atom positions were calculated geometrically and refined using the riding model.

_refine_special_details: No Special refinement necessary.

There is a single molecule in the asymmetric unit, which is represented by the reported sum formula. In other words: Z is 4 and Z' is 1.

The Flack parameter was refined to -0.009(13). Determination of absolute structure using Bayesian statistics on Bijvoet differences using the Olex2 results in 0.007(5). Note: The Flack parameter is used to determine chirality of the crystal studied, the value should be near 0, a value of 1 means that the stereochemistry is wrong and the model should be inverted. A value of 0.5 means that the crystal consists of a racemic mixture of the two enantiomers.

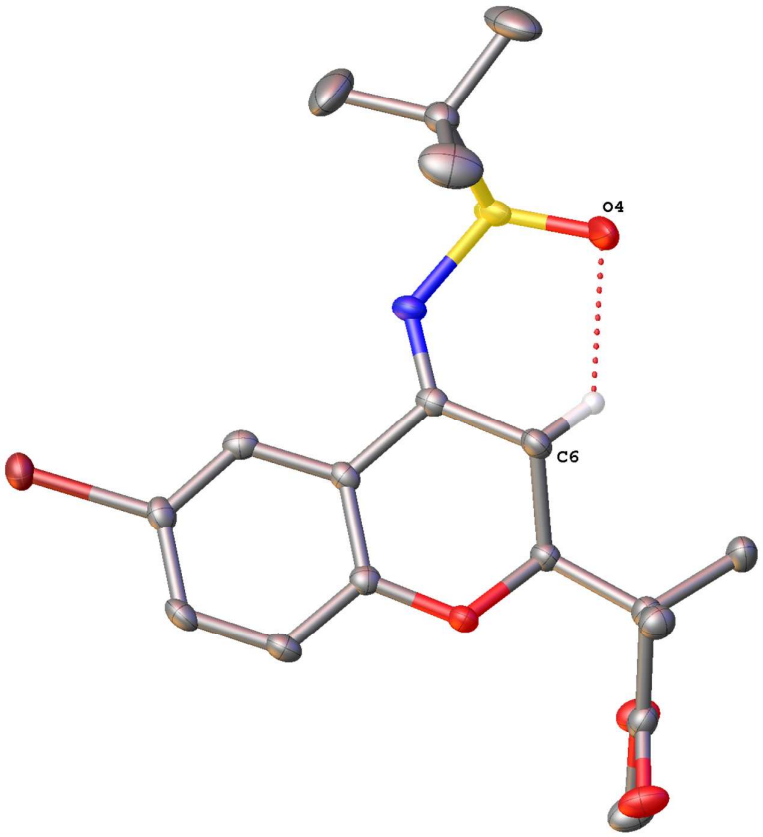


Figure 1: The following hydrogen bonding interactions with a maximum D-D distance of 3.1 Å and a minimum angle of 120 ° are present in **cx0744**: C6-O4 =2.952 Å.

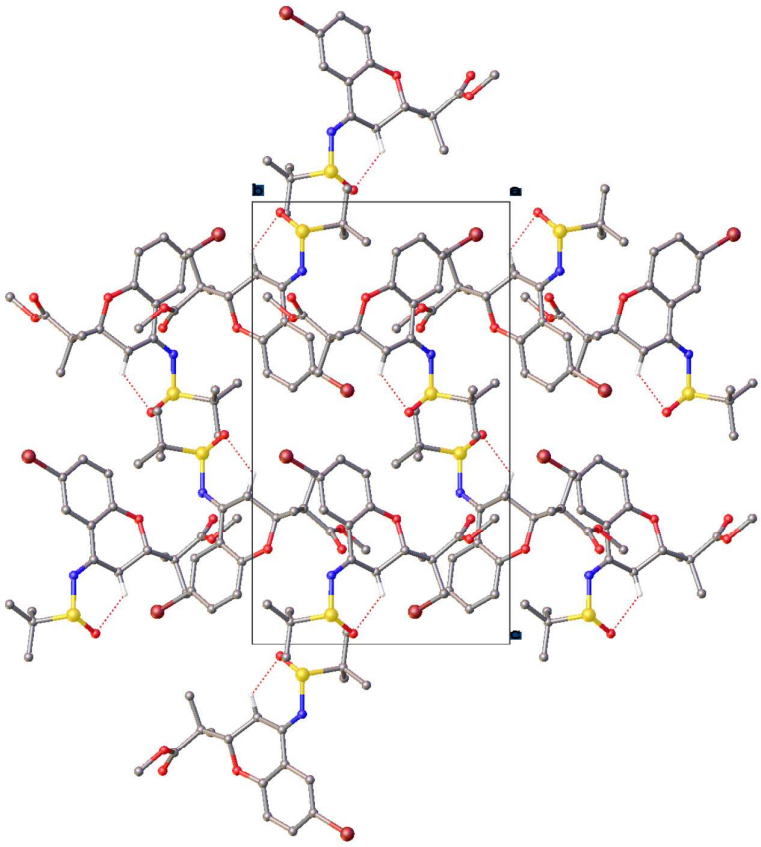


Figure 2: Packing diagram of cx0744.

Table 1: Fractional Atomic Coordinates (×10⁴) and Equivalent Isotropic Displacement Parameters (Å²×10³) for **cx0744**. U_{eq} is defined as 1/3 of the trace of the orthogonalised U_{ij}.

Atom	x	y	z	U_{eq}
Br1	4631.8(3)	8684.3(3)	5750.6(2)	22.85(9)
S1	4094.2(8)	6945.2(6)	9320.4(4)	24.05(16)
O1	1788(2)	4404.2(17)	7154.7(10)	19.1(4)
O2	2801(2)	1573.6(18)	7619.0(12)	25.0(5)
O3	706(2)	1521.4(19)	7149.6(12)	30.3(5)
O4	3238(3)	6085(2)	9737.6(11)	42.5(7)
N1	3828(3)	6932(2)	8437.4(12)	18.8(5)
C1	3691(3)	7369(3)	6218.1(15)	19.2(6)
C2	2897(3)	6573(3)	5813.0(15)	21.1(6)
C3	2267(3)	5588(3)	6138.5(15)	20.7(6)
C4	2405(3)	5412(2)	6874.6(15)	16.5(5)
C5	2175(3)	4074(2)	7875.4(14)	17.7(6)
C6	2138(3)	5193(2)	8362.1(14)	18.3(6)
C7	3108(3)	6157(2)	8082.5(14)	16.5(5)
C8	3139(3)	6258(3)	7290.6(14)	15.4(5)
C9	3816(3)	7232(3)	6947.1(15)	17.8(5)
C10	1214(3)	3013(3)	8082.1(15)	19.1(6)
C11	1522(3)	2561(3)	8843.1(16)	24.8(6)
C12	-271(3)	3386(3)	8022.8(16)	22.7(6)
C13	1504(3)	1964(3)	7559.5(15)	20.1(6)
C14	3194(3)	595(3)	7138.2(19)	30.0(7)
C15	3402(3)	8517(3)	9479.3(15)	24.3(6)
C16	3504(5)	8685(4)	10287.6(18)	49(1)
C17	4284(5)	9421(3)	9084(2)	52.5(11)
C18	1933(4)	8560(4)	9242(2)	46.5(9)

Table 2: Anisotropic Displacement Parameters (×10^4) **cx0744**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2} \times U_{11} + ... + 2hka^* \times b^* \times U_{12}]$

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
Br1	27.62(15)	22.64(14)	18.29(14)	3.84(11)	2.80(12)	-1.46(12)
S1	37.7(4)	19.4(3)	15.1(3)	-2.5(3)	-8.7(3)	0.8(3)
O1	26.1(10)	18.8(9)	12.5(9)	-1.0(8)	-2.7(8)	-3.6(8)
O2	21.5(10)	22.7(11)	30.9(11)	-6.5(8)	-2.0(9)	1.7(8)
O3	27.2(11)	26.0(11)	37.8(12)	-10.7(9)	-9.8(9)	-0.9(9)
O4	88(2)	25.8(12)	13.7(10)	3.9(9)	-6.4(11)	-15.8(13)
N1	23.8(13)	18.1(12)	14.5(11)	-2.6(9)	-3.9(10)	0.4(10)
C1	19.8(14)	21.5(14)	16.5(13)	1.9(11)	4.5(11)	2.3(11)
C2	26.3(14)	26.4(14)	10.7(12)	-0.9(11)	0.7(12)	3.9(11)
C3	24.1(15)	24.3(14)	13.7(13)	-4.2(11)	-1.2(11)	0.2(12)
C4	16.7(13)	16.4(13)	16.5(13)	-1.4(10)	2.0(11)	1.8(11)
C5	21.3(14)	17.7(13)	14.1(13)	-0.2(10)	-1.6(11)	-1.0(11)
C6	21.6(15)	20.8(14)	12.7(13)	-0.2(11)	-1.2(11)	0.3(11)
C7	17.8(13)	16.2(13)	15.4(12)	-0.9(11)	-2.2(10)	4.1(11)
C8	15.2(12)	18.0(12)	13.2(12)	-0.4(11)	-0.6(9)	3.3(11)
C9	16.3(13)	17.1(13)	20.1(14)	-3.3(11)	-2.0(11)	2.5(10)
C10	20.7(15)	19.6(14)	17.1(13)	0.8(11)	-0.7(11)	-1.7(12)
C11	31.1(16)	23.6(15)	19.6(14)	3.5(12)	-2.3(13)	-6.0(12)
C12	20.8(14)	22.3(14)	25.1(14)	-1.7(11)	0.7(12)	-1.8(12)
C13	21.2(14)	17.5(13)	21.5(14)	2.1(11)	-1.4(12)	-2.8(11)
C14	24.6(16)	23.0(15)	42(2)	-8.5(14)	1.5(14)	2.3(13)
C15	33.2(16)	20.9(14)	18.7(14)	-2.8(11)	1.4(11)	-0.1(12)
C16	82(3)	39(2)	26.1(18)	-15.0(17)	-7.9(18)	13(2)
C17	74(3)	23.7(17)	60(3)	-6.1(16)	23(2)	-15.0(18)
C18	34.7(18)	57(2)	48(2)	-20(2)	0.2(17)	14.5(17)

Table 3: Bond Lengths in Å for **cx0744**.

Atom	Atom	Length/Å
Br1	C1	1.911(3)
S1	O4	1.478(3)
S1	N1	1.671(2)
S1	C15	1.860(3)
O1	C4	1.355(3)
O1	C5	1.444(3)
O2	C13	1.348(4)
O2	C14	1.443(4)
O3	C13	1.197(4)
N1	C7	1.284(4)
C1	C2	1.388(4)
C1	C9	1.376(4)
C2	C3	1.376(4)

Atom	Atom	Length/Å
C3	C4	1.395(4)
C4	C8	1.402(4)
C5	C6	1.516(4)
C5	C10	1.539(4)
C6	C7	1.509(4)
C7	C8	1.484(3)
C8	C9	1.404(4)
C10	C11	1.534(4)
C10	C12	1.519(4)
C10	C13	1.526(4)
C15	C16	1.525(4)
C15	C17	1.502(5)
C15	C18	1.510(5)

Table 4: Bond Angles in ° for **cx0744**.

Atom	Atom	Atom	Angle/°
O4	S1	N1	115.24(13)
O4	S1	C15	106.62(15)
N1	S1	C15	96.19(13)
C4	O1	C5	116.2(2)
C13	O2	C14	115.7(2)
C7	N1	S1	127.0(2)
C2	C1	Br1	119.1(2)
C9	C1	Br1	119.4(2)
C9	C1	C2	121.5(3)
C3	C2	C1	119.7(3)
C2	C3	C4	119.9(3)
O1	C4	C3	116.6(2)

Atom	Atom	Atom	Angle/°
O1	C4	C8	122.9(2)
C3	C4	C8	120.5(3)
O1	C5	C6	110.8(2)
O1	C5	C10	104.9(2)
C6	C5	C10	115.7(2)
C7	C6	C5	109.4(2)
N1	C7	C6	128.6(2)
N1	C7	C8	117.1(3)
C8	C7	C6	114.1(2)
C4	C8	C7	119.6(3)
C4	C8	C9	118.9(2)
C9	C8	C7	121.4(2)

Atom	Atom	Atom	Angle/°
C1	C9	C8	119.5(3)
C11	C10	C5	110.5(2)
C12	C10	C5	111.9(2)
C12	C10	C11	110.0(3)
C12	C10	C13	109.4(2)
C13	C10	C5	106.4(2)
C13	C10	C11	108.6(2)
O2	C13	C10	111.0(2)
O3	C13	O2	123.1(3)
O3	C13	C10	125.9(3)
C16	C15	S1	104.1(2)
C17	C15	S1	107.9(2)
C17	C15	C16	111.8(3)
C17	C15	C18	112.8(3)
C18	C15	S1	109.3(2)
C18	C15	C16	110.5(3)

Table 5: Torsion Angles in ° for **cx0744**.

Atom	Atom	Atom	Atom	Angle/°
Br1	C1	C2	C3	176.9(2)
Br1	C1	C9	C8	-179.22(19)
S1	N1	C7	C6	10.9(4)
S1	N1	C7	C8	-173.8(2)
O1	C4	C8	C7	-8.2(4)
O1	C4	C8	C9	176.0(2)
O1	C5	C6	C7	-59.7(3)
O1	C5	C10	C11	179.6(2)
O1	C5	C10	C12	-57.4(3)
O1	C5	C10	C13	62.0(3)
O4	S1	N1	C7	-9.9(3)
O4	S1	C15	C16	59.0(3)
O4	S1	C15	C17	177.9(3)
O4	S1	C15	C18	-59.1(3)
N1	S1	C15	C16	177.7(2)
N1	S1	C15	C17	-63.4(3)
N1	S1	C15	C18	59.6(3)
N1	C7	C8	C4	178.1(2)
N1	C7	C8	C9	-6.2(4)
C1	C2	C3	C4	1.4(4)
C2	C1	C9	C8	0.7(4)
C2	C3	C4	O1	-178.2(2)
C2	C3	C4	C8	2.5(4)
C3	C4	C8	C7	171.2(3)
C3	C4	C8	C9	-4.7(4)
C4	O1	C5	C6	48.2(3)
C4	O1	C5	C10	173.6(2)
C4	C8	C9	C1	3.1(4)
C5	O1	C4	C3	166.7(2)
C5	O1	C4	C8	-13.9(4)
C5	C6	C7	N1	-146.3(3)
C5	C6	C7	C8	38.3(3)
C5	C10	C13	O2	61.6(3)
C5	C10	C13	O3	-119.0(3)
C6	C5	C10	C11	-58.0(3)
C6	C5	C10	C12	65.0(3)
C6	C5	C10	C13	-175.7(2)
C6	C7	C8	C4	-6.0(4)
C6	C7	C8	C9	169.8(2)
C7	C8	C9	C1	-172.7(3)
C9	C1	C2	C3	-3.0(4)
C10	C5	C6	C7	-178.8(2)
C11	C10	C13	O2	-57.3(3)
C11	C10	C13	O3	122.1(3)
C12	C10	C13	O2	-177.4(2)
C12	C10	C13	O3	2.0(4)
C14	O2	C13	O3	1.7(4)
C14	O2	C13	C10	-178.9(2)
C15	S1	N1	C7	-121.6(3)

Table 6: Hydrogen Fractional Atomic Coordinates (×10^4) and Equivalent Isotropic Displacement Parameters (Å^2×10^3) for **cx0744**. U_{eq} is defined as 1/3 of the trace of the orthogonalised U_{ij}.

Atom	x	y	z	U_{eq}
H2	2790	6706	5314	25
H3	1739	5030	5863	25
H5	3127	3750	7865	21
H6A	1203	5532	8378	22
H6B	2402	4951	8854	22
H9	4355	7792	7216	21
H11A	2505	2440	8897	37
H11B	1050	1779	8928	37
H11C	1208	3177	9190	37
H12A	-455	4069	8352	34
H12B	-850	2682	8146	34
H12C	-465	3649	7531	34
H14A	3127	885	6643	45
H14B	2588	-113	7206	45
H14C	4133	348	7239	45
H16A	2979	8038	10528	74
H16B	3138	9495	10420	74
H16C	4459	8632	10434	74
H17A	5240	9270	9204	79
H17B	4036	10262	9223	79
H17C	4151	9318	8568	79
H18A	1886	8458	8721	70
H18B	1535	9355	9375	70
H18C	1426	7893	9474	70

Table 7: Hydrogen Bond information for **cx0744**.

D	H	A	d(D-H)/Å	d(H-A)/Å	d(D-A)/Å	D-H-A/deg
C6	H6B	O4	0.99	2.22	2.952(4)	129.9

Citations

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Sheldrick, G.M., Crystal structure refinement with ShelXL, *Acta Cryst.*, (2015), **C27**, 3-8.

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