XCPM80

Experimental. A prismatic crystal (0.1x0.1x0.2 mm) was selected and mounted on a MAR345 diffractometer with an image plate detector. Unit-cell parameters were determined from automatic 185 reflections ($3 < \theta < 31°$) and refined by least-squares method. Intensities were collected with graphite monochromatized Mo Kα radiation. 16387 reflections were measured in the range $1.58 \leq \theta \leq 32.35$. 5891 of which were non-equivalent by symmetry (Rint(on I) = 0.076). 5116 reflections were assumed as observed applying the condition $I > 2\sigma(I)$. Lorentz-polarization but no absorption corrections were made.

The structure was solved by Direct methods, using SHELXS computer program (Sheldrick, G.M., (1997), A computer program for automatic solution of crystal structure. UNIVER goettingen, Germany) and refined by full-matrix least-squares method with SHELX97 computer program (Sheldrick, G.M., (1997), A program for crystal structure refinement. Univer Goettinhen, Germany), using 16387 reflections, (very negative intensities were not assumed). The function minimized was $\Sigma\ w\ ||Fo|^2 - |Fc|^2|^2$, where $w = [\sigma^2(I) + (0.0785P)^2 + 5.2849P]^{-1}$, and $P = (|Fo|^2 + 2\ |Fc|^2)/3$, f, f' and f" were taken from International Tables of X-Ray Crystallography (International Tables of X-Ray Crystallography, (1974), Ed. Kynoch press, Vol. IV, pp 99-100 and 149). 15H atoms were located from a difference synthesis and refined with an overall isotropic temperature factor and 7H atoms were computed and refined, using a riding model, with an isotropic temperature factor equal to 1.2 time the equivalent temperature factor of the atom which are linked. The final R(on F) factor was 0.077, wR(on $|F|^2$) = 0.181 and goodness of fit = 1.09 for all observed reflections. Number of refined parameters was 351. Max. shift/esd = 0.00, Mean shift/esd = 0.00. Max. and min. peaks in final difference synthesis was 0.562 and -0.767 e.$Å^{-3}$, respectively.

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Table 1. Crystal data and structure refinement for xcpm80.

Identification code	xcpm80
Empirical formula	C_{24} H_{22} O_6 S_2
Formula weight	470.54
Temperature	105(2) K
Wavelength	0.71073 Å
Crystal system, space group	Orthorhombic, $P2_12_12_1$
Unit cell dimensions	a = 5.919(3) Å α= 90 °. b = 14.531(5) Å β= 90 °. c = 25.735(11) Å γ= 90 °.
Volume	2213.4(17) $Å^3$
Z, Calculated density	4, 1.412 Mg/m^3
Absorption coefficient	0.280 mm^{-1}
F(000)	984
Crystal size	0.1 x 0.09 x 0.08 mm
Theta range for data collection	1.58 to 32.35 °.
Limiting indices	-8<=h<=7, -19<=k<=20, -38<=l<=38
Reflections collected / unique	16387 / 5891 [R(int) = 0.0768]
Completeness to theta = 25.00	94.0 %
Absorption correction	None
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	5891 / 0 / 351
Goodness-of-fit on F^2	1.098
Final R indices [I>2σ(I)]	R1 = 0.0779, wR2 = 0.1815
R indices (all data)	R1 = 0.0965, wR2 = 0.2035
Absolute structure parameter	-0.09(13)
Extinction coefficient	0.016(2)
Largest diff. peak and hole	0.562 and -0.767 $e.Å^{-3}$

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$ x 10^3) for xcpm80.
U(eq) is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U(eq)
S(1)	8655(2)	4630(1)	8015(1)	28(1)
S(2)	10291(2)	4130(1)	9067(1)	29(1)
O(1)	6249(7)	8109(2)	8349(1)	38(1)
O(2)	9733(7)	7753(2)	8630(1)	39(1)
O(3)	11039(5)	4712(2)	7917(1)	31(1)
O(4)	7140(6)	5216(2)	7731(1)	33(1)
O(5)	12410(5)	4614(2)	9104(1)	31(1)
O(6)	10277(6)	3241(2)	8822(1)	33(1)
C(1)	10478(8)	6472(3)	9675(2)	30(1)
C(2)	10411(9)	6668(3)	10208(2)	33(1)
C(3)	8517(9)	6458(3)	10499(2)	32(1)
C(4)	6640(8)	6054(3)	10259(2)	32(1)
C(5)	6672(7)	5883(3)	9723(2)	29(1)
C(6)	8617(7)	6075(3)	9429(2)	26(1)
C(7)	8628(8)	5896(3)	8840(2)	27(1)
C(8)	6908(8)	6561(3)	8600(2)	28(1)
C(9)	7802(8)	7516(3)	8531(2)	31(1)
C(10)	6910(12)	9067(4)	8303(2)	47(1)
C(11)	4767(8)	6365(4)	8491(2)	33(1)
C(12)	8249(8)	4870(3)	8716(2)	28(1)
C(13)	7802(7)	3476(3)	7920(2)	29(1)
C(14)	5607(8)	3200(3)	8053(2)	34(1)
C(15)	5043(9)	2276(4)	7984(2)	38(1)
C(16)	6591(9)	1645(4)	7780(2)	38(1)
C(17)	8711(10)	1941(3)	7642(2)	35(1)
C(18)	9345(8)	2856(3)	7709(2)	31(1)
C(19)	9159(8)	3977(3)	9699(2)	30(1)
C(20)	10492(8)	4205(3)	10123(2)	32(1)
C(21)	9708(10)	3983(4)	10620(2)	40(1)
C(22)	7618(10)	3567(4)	10682(2)	43(1)
C(23)	6317(10)	3344(4)	10250(2)	39(1)
C(24)	7081(8)	3540(4)	9753(2)	34(1)

Table 3. Bond lengths [Å] and angles [°] for xcpm80.

S(1)-O(4)	1.436(3)
S(1)-O(3)	1.439(3)
S(1)-C(13)	1.768(5)
S(1)-C(12)	1.853(4)
S(2)-O(6)	1.437(3)
S(2)-O(5)	1.442(3)
S(2)-C(19)	1.773(5)
S(2)-C(12)	1.853(4)
O(1)-C(9)	1.344(6)
O(1)-C(10)	1.450(7)
O(2)-C(9)	1.221(6)
C(1)-C(6)	1.395(6)
C(1)-C(2)	1.401(6)
C(1)-H(1)	1.07(5)
C(2)-C(3)	1.382(7)
C(2)-H(2)	0.95(7)
C(3)-C(4)	1.400(7)
C(3)-H(3)	1.06(5)
C(4)-C(5)	1.401(6)
C(4)-H(4)	1.06(5)
C(5)-C(6)	1.406(6)
C(5)-H(5)	0.9300
C(6)-C(7)	1.537(6)
C(7)-C(8)	1.534(6)
C(7)-C(12)	1.542(6)
C(7)-H(7)	1.04(5)
C(8)-C(11)	1.328(7)
C(8)-C(9)	1.496(7)
C(10)-H(10)	0.9600
C(10)-H(10A)	0.9600
C(10)-H(10B)	0.9600
C(11)-H(11)	0.94(7)
C(11)-H(11A)	1.01(6)
C(12)-H(12)	0.99(5)
C(13)-C(18)	1.393(7)
C(13)-C(14)	1.402(7)
C(14)-C(15)	1.394(7)
C(14)-H(14)	1.03(5)
C(15)-C(16)	1.399(8)
C(15)-H(15)	1.02(9)
C(16)-C(17)	1.373(8)
C(16)-H(16)	0.9300
C(17)-C(18)	1.392(7)
C(17)-H(17)	1.04(6)
C(18)-H(18)	1.05(7)
C(19)-C(20)	1.387(6)
C(19)-C(24)	1.391(7)
C(20)-C(21)	1.397(7)
C(20)-H(20)	0.9300
C(21)-C(22)	1.387(8)
C(21)-H(21)	1.02(6)
C(22)-C(23)	1.392(8)
C(22)-H(22)	1.06(6)
C(23)-C(24)	1.384(7)
C(23)-H(23)	0.9300
C(24)-H(24)	1.05(6)

O(4)-S(1)-O(3)	118.3(2)
O(4)-S(1)-C(13)	108.3(2)
O(3)-S(1)-C(13)	109.6(2)
O(4)-S(1)-C(12)	107.5(2)
O(3)-S(1)-C(12)	106.3(2)
C(13)-S(1)-C(12)	106.0(2)
O(6)-S(2)-O(5)	118.2(2)
O(6)-S(2)-C(19)	106.7(2)
O(5)-S(2)-C(19)	109.3(2)
O(6)-S(2)-C(12)	107.7(2)
O(5)-S(2)-C(12)	108.4(2)
C(19)-S(2)-C(12)	105.9(2)
C(9)-O(1)-C(10)	117.3(5)
C(6)-C(1)-C(2)	120.3(4)
C(6)-C(1)-H(1)	119(3)
C(2)-C(1)-H(1)	120(3)
C(3)-C(2)-C(1)	120.6(4)
C(3)-C(2)-H(2)	123(4)
C(1)-C(2)-H(2)	116(4)
C(2)-C(3)-C(4)	119.9(4)
C(2)-C(3)-H(3)	121(3)
C(4)-C(3)-H(3)	119(3)
C(3)-C(4)-C(5)	119.8(4)
C(3)-C(4)-H(4)	119(3)
C(5)-C(4)-H(4)	121(3)
C(4)-C(5)-C(6)	120.4(4)
C(4)-C(5)-H(5)	119.8
C(6)-C(5)-H(5)	119.8
C(1)-C(6)-C(5)	119.0(4)
C(1)-C(6)-C(7)	120.9(4)
C(5)-C(6)-C(7)	120.0(4)
C(8)-C(7)-C(6)	106.8(3)
C(8)-C(7)-C(12)	115.3(4)
C(6)-C(7)-C(12)	111.6(4)
C(8)-C(7)-H(7)	105(3)
C(6)-C(7)-H(7)	108(3)
C(12)-C(7)-H(7)	110(3)
C(11)-C(8)-C(9)	120.8(5)
C(11)-C(8)-C(7)	125.7(5)
C(9)-C(8)-C(7)	113.4(4)
O(2)-C(9)-O(1)	122.2(5)
O(2)-C(9)-C(8)	124.6(4)
O(1)-C(9)-C(8)	113.2(4)
O(1)-C(10)-H(10)	109.5
O(1)-C(10)-H(10A)	109.5
H(10)-C(10)-H(10A)	109.5
O(1)-C(10)-H(10B)	109.5
H(10)-C(10)-H(10B)	109.5
H(10A)-C(10)-H(10B)	109.5
C(8)-C(11)-H(11)	115(4)
C(8)-C(11)-H(11A)	122(3)
H(11)-C(11)-H(11A)	122(5)
C(7)-C(12)-S(2)	111.4(3)
C(7)-C(12)-S(1)	111.5(3)
S(2)-C(12)-S(1)	106.3(2)
C(7)-C(12)-H(12)	115(3)
S(2)-C(12)-H(12)	106(3)
S(1)-C(12)-H(12)	106(3)
C(18)-C(13)-C(14)	121.2(4)
C(18)-C(13)-S(1)	118.7(4)
C(14)-C(13)-S(1)	120.1(4)

C(15)-C(14)-C(13)	117.8(5)
C(15)-C(14)-H(14)	124(3)
C(13)-C(14)-H(14)	118(3)
C(14)-C(15)-C(16)	121.5(5)
C(14)-C(15)-H(15)	120(4)
C(16)-C(15)-H(15)	119(4)
C(17)-C(16)-C(15)	119.3(5)
C(17)-C(16)-H(16)	120.3
C(15)-C(16)-H(16)	120.3
C(16)-C(17)-C(18)	120.9(5)
C(16)-C(17)-H(17)	121(3)
C(18)-C(17)-H(17)	118(3)
C(17)-C(18)-C(13)	119.3(5)
C(17)-C(18)-H(18)	122(4)
C(13)-C(18)-H(18)	119(4)
C(20)-C(19)-C(24)	122.2(4)
C(20)-C(19)-S(2)	118.5(4)
C(24)-C(19)-S(2)	118.9(4)
C(19)-C(20)-C(21)	118.4(5)
C(19)-C(20)-H(20)	120.8
C(21)-C(20)-H(20)	120.8
C(22)-C(21)-C(20)	120.2(5)
C(22)-C(21)-H(21)	118(4)
C(20)-C(21)-H(21)	121(4)
C(21)-C(22)-C(23)	120.1(5)
C(21)-C(22)-H(22)	120(3)
C(23)-C(22)-H(22)	120(3)
C(24)-C(23)-C(22)	120.6(5)
C(24)-C(23)-H(23)	119.7
C(22)-C(23)-H(23)	119.7
C(23)-C(24)-C(19)	118.4(5)
C(23)-C(24)-H(24)	123(3)
C(19)-C(24)-H(24)	119(3)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2$ x 10^3) for xcpm80. The anisotropic displacement factor exponent takes the form: $-2\pi^2 [h^2 a^{*2} U_{11} + ... + 2 h k a^* b^* U_{12}]$

	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
S(1)	28(1)	25(1)	30(1)	0(1)	1(1)	0(1)
S(2)	28(1)	27(1)	31(1)	0(1)	-2(1)	2(1)
O(1)	45(2)	26(2)	43(2)	4(1)	1(2)	7(2)
O(2)	39(2)	31(2)	47(2)	0(1)	-2(2)	-7(2)
O(3)	28(2)	27(2)	37(2)	-5(1)	6(1)	-3(1)
O(4)	37(2)	30(2)	32(2)	2(1)	0(1)	3(1)
O(5)	28(2)	32(2)	35(2)	4(1)	0(1)	-1(1)
O(6)	40(2)	26(2)	33(2)	-2(1)	-6(1)	5(1)
C(1)	26(2)	23(2)	40(2)	2(2)	-1(2)	1(2)
C(2)	35(2)	26(3)	37(2)	-2(2)	-8(2)	-2(2)
C(3)	38(2)	28(2)	31(2)	-5(2)	-5(2)	6(2)
C(4)	32(2)	29(3)	36(2)	-1(2)	2(2)	8(2)
C(5)	27(2)	28(2)	31(2)	-1(2)	0(1)	2(2)
C(6)	22(2)	24(2)	33(2)	3(1)	-1(1)	4(2)
C(7)	27(2)	25(2)	30(2)	0(2)	2(1)	-1(2)
C(8)	32(2)	25(2)	27(2)	-2(2)	2(1)	1(2)
C(9)	36(2)	25(3)	32(2)	0(2)	2(2)	4(2)
C(10)	71(4)	25(3)	44(3)	7(2)	9(2)	9(3)
C(11)	30(2)	37(3)	33(2)	1(2)	3(2)	6(2)
C(12)	28(2)	25(2)	31(2)	2(2)	-1(1)	5(2)
C(13)	29(2)	27(2)	31(2)	2(2)	-4(2)	-1(2)
C(14)	31(2)	27(3)	44(2)	-2(2)	-3(2)	-1(2)
C(15)	35(3)	34(3)	46(3)	2(2)	-5(2)	-6(2)
C(16)	49(3)	25(3)	39(2)	-1(2)	-8(2)	-5(2)
C(17)	46(3)	24(2)	36(2)	-5(2)	-4(2)	1(2)
C(18)	33(2)	29(3)	32(2)	-2(2)	-2(2)	2(2)
C(19)	31(2)	24(2)	35(2)	-1(2)	1(2)	2(2)
C(20)	33(2)	23(2)	39(2)	1(2)	-4(2)	1(2)
C(21)	51(3)	37(3)	33(2)	3(2)	-5(2)	-2(2)
C(22)	54(3)	39(3)	35(2)	5(2)	4(2)	-5(3)
C(23)	41(3)	28(3)	47(3)	5(2)	3(2)	-3(2)
C(24)	34(2)	27(3)	41(2)	2(2)	0(2)	1(2)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å^2 x 10^3) for xcpm80.

	x	y	z	U(eq)
H(5)	5400	5640	9561	34
H(10)	5588	9441	8257	56
H(10A)	7892	9140	8009	56
H(10B)	7688	9253	8613	56
H(16)	6188	1030	7738	45
H(20)	11875	4498	10078	38
H(23)	4921	3061	10294	80(20)
H(1)	11970(90)	6610(40)	9453(19)	28(13)
H(2)	11720(110)	6950(40)	10350(20)	48(17)
H(3)	8490(90)	6550(40)	10906(19)	30(13)
H(4)	5210(90)	5880(30)	10487(18)	25(12)
H(7)	10190(90)	6110(30)	8696(17)	21(11)
H(11)	3890(120)	6870(50)	8380(20)	52(19)
H(11A)	4100(100)	5740(40)	8550(20)	40(16)
H(12)	6730(90)	4630(40)	8807(18)	23(12)
H(14)	4480(90)	3690(40)	8170(19)	28(13)
H(15)	3480(140)	2040(50)	8090(30)	80(20)
H(17)	9910(110)	1490(40)	7500(20)	39(15)
H(18)	10990(120)	3090(50)	7620(30)	55(19)
H(21)	10640(120)	4130(50)	10940(20)	55(19)
H(22)	7020(100)	3400(40)	11060(20)	36(15)
H(24)	6190(100)	3350(40)	9420(20)	41(15)

C(7) - C(6) C(8) C(12) H(7) sp3 R