Crystal and molecular structure of Compound 1

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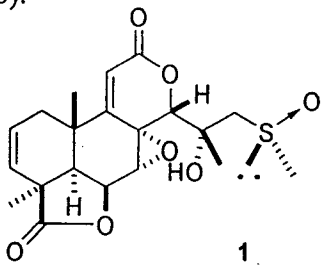
Abstract

We present the crystal and molecular structure of compound **1** (A2224-6).

Comment

The study of the titled structure was undertaken to establish its three dimensional structure. Geometries are tabulated below. All diagrams and calculations were performed using maXus (Bruker Nonius, Delft & MacScience, Japan).



1

Experimental

Crystal data

$C_{20}H_{24}O_7S$
$C_{20}H_{24}O_7S$
$M_r = 408.469$
triclinic
P1
a = 6.2480 (8)Å
b = 7.131 (2)Å
c = 11.521 (2)Å
α = 85.285 (10)°
β = 76.957 (11)°
γ = 73.272 (12)°
V = 478.8 (2)Å^3
Z = 1

$D_x = 1.417$ Mg m^{-3}
Density measured by: not measured
fine-focus sealed tube
Mo *K*α radiation
λ = 0.71073
Cell parameters from 1862
θ = 2.98—27.21 °
μ = 0.210 mm^{-1}
T = 296 K
Prism
Colorless
Crystal source:Local laboratory

Data collection

DIP Image plate
IP
Absorption correction: none
1862 measured reflections
1862 independent reflections
1761 observed reflections

Criterion: >2sigma(I)
$R_{int} = 0.028$
$\theta_{max} = 27.21$ °
h = 0 →7
k = -8 →9
l = -14 →14

Refinement

Refinement on F^2
fullmatrix least squares refinement
R(all) = 0.0367
R(gt) = 0.0349
wR(ref) = 0.0934
wR(gt)= 0.0920
S(ref) = 1.076
1862 reflections
259 parameters
3 restraints
H-atom parameters not refined
Calculated weights calc

$\Delta/\sigma_{max} = 0.000$
$\Delta\rho_{max} = 0.214 e Å^3$
$\Delta\rho_{min} = -0.245 e Å^3$
Extinction correction: *SHELXL*
$Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient = 0.200 (18)
Atomic scattering factors from International Tables Vol C Tables 4.2.6.8 and 6.1.1.4
Flack parameter = 0.20 (8)
Flack H D (1983), *Acta Cryst.* A39, 876-881

Data collection: DIP Image plate
Cell refinement: Scalepack(HKL)
Data reduction: maXus (Mackay et al., 1999)
Program(s) used to refine structure: *SHELXL*-97 (Sheldrick, 1997)

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters ($Å^2$)*
$U_{eq} = 1/3\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	U_{eq}	Occ
C1	0.2094 (5)	0.6431 (4)	0.9302 (3)	0.0388 (6)	1
C2	0.2337 (6)	0.7025 (5)	1.0483 (3)	0.0474 (7)	1
C3	0.3428 (5)	0.5783 (5)	1.1224 (3)	0.0442 (7)	1
C4	0.4396 (5)	0.3638 (4)	1.1004 (2)	0.0373 (6)	1
C5	0.3934 (4)	0.3006 (4)	0.9864 (2)	0.0314 (5)	1
C6	0.5847 (5)	0.1118 (4)	0.9573 (2)	0.0362 (6)	1
C7	0.6547 (5)	0.0551 (4)	0.8279 (2)	0.0349 (6)	1
C8	0.5444 (4)	0.1788 (3)	0.7377 (2)	0.0289 (5)	1
C9	0.3780 (4)	0.3739 (4)	0.7747 (2)	0.0282 (5)	1
C10	0.4015 (4)	0.4588 (4)	0.8857 (2)	0.0298 (5)	1
C11	0.2386 (5)	0.4607 (4)	0.7013 (2)	0.0337 (5)	1
C12	0.2494 (5)	0.3644 (4)	0.5910 (2)	0.0334 (5)	1
C14	0.6360 (4)	0.1801 (4)	0.6051 (2)	0.0295 (5)	1
C15	0.8048 (4)	-0.0052 (4)	0.5421 (2)	0.0324 (5)	1
C16	0.6905 (5)	-0.1703 (4)	0.5447 (3)	0.0377 (6)	1
C17	0.9086 (5)	0.0550 (5)	0.4154 (2)	0.0418 (7)	1
C18	0.3451 (7)	0.2498 (6)	1.2115 (3)	0.0527 (8)	1
C19	0.6997 (5)	0.2873 (5)	1.0792 (3)	0.0444 (8)	1
C20	0.6306 (5)	0.5126 (4)	0.8504 (2)	0.0373 (6)	1
C22	0.7779 (7)	-0.3566 (7)	0.3337 (4)	0.0622 (10)	1

O13	0.4434 (3)	0.2246 (3)	0.54607 (17)	0.0358 (4)	1
O23	0.3750 (4)	-0.2850 (4)	0.4743 (3)	0.0555 (6)	1
O24	0.8292 (4)	0.3357 (5)	1.1250 (2)	0.0670 (8)	1
O25	0.7772 (4)	0.1411 (3)	0.99867 (18)	0.0445 (5)	1
O26	0.4696 (4)	0.0102 (3)	0.79081 (17)	0.0375 (5)	1
O27	0.9781 (4)	-0.0673 (3)	0.60998 (19)	0.0433 (5)	1
O28	0.0934 (4)	0.4001 (3)	0.5394 (2)	0.0476 (5)	1
S21	0.55226 (10)	-0.17811 (8)	0.42317 (6)	0.0385 (2)	1
H1A	0.2162	0.7490	0.8722	0.047	1
H1B	0.0622	0.6174	0.9392	0.047	1
H2	0.1692	0.8326	1.0703	0.057	1
H3	0.3595	0.6274	1.1911	0.053	1
H5	0.2446	0.2727	1.0035	0.038	1
H6	0.5364	0.0047	1.0046	0.043	1
H7	0.8090	-0.0311	0.8006	0.042	1
H11	0.1344	0.5823	0.7201	0.040	1
H14	0.7083	0.2871	0.5860	0.035	1
H16A	0.5771	-0.1612	0.6185	0.045	1
H16B	0.8061	-0.2942	0.5472	0.045	1
H17A	0.9827	0.1541	0.4197	0.050	1
H17B	0.7894	0.1055	0.3718	0.050	1
H17C	1.0183	-0.0571	0.3757	0.050	1
H18A	0.3875	0.2843	1.2802	0.063	1
H18B	0.4080	0.1116	1.1988	0.063	1
H18C	0.1815	0.2829	1.2241	0.063	1
H20A	0.7526	0.3974	0.8245	0.045	1
H20B	0.6582	0.5652	0.9180	0.045	1
H20C	0.6235	0.6086	0.7868	0.045	1
H22A	0.8201	-0.4740	0.3803	0.075	1
H22B	0.9078	-0.3061	0.3065	0.075	1
H22C	0.7269	-0.3855	0.2664	0.075	1
H27	1.0993	-0.1240	0.5666	0.052	1

Table 2. *Anisotropic displacement parameters ($Å^2$)*

	U_{11}	U_{12}	U_{13}	U_{22}	U_{23}	U_{33}
C1	0.0370 (15)	-0.0016 (10)	-0.0075 (11)	0.0350 (14)	-0.0090 (11)	0.0403 (14)
C2	0.0490	-0.0045	-0.0056	0.0426	-0.0167	0.0459

	(19)	(13)	(13)	(16)	(13)	(17)
C3	0.0378	-0.0074	-0.0048	0.0557	-0.0178	0.0370
	(17)	(13)	(11)	(18)	(13)	(14)
C4	0.0297	-0.0081	-0.0045	0.0504	-0.0056	0.0298
	(14)	(11)	(10)	(16)	(11)	(13)
C5	0.0291	-0.0094	-0.0048	0.0361	-0.0005	0.0285
	(13)	(10)	(9)	(13)	(9)	(12)
C6	0.0409	-0.0059	-0.0102	0.0349	0.0023	0.0306
	(16)	(10)	(10)	(13)	(10)	(13)
C7	0.0399	-0.0005	-0.0104	0.0278	-0.0028	0.0331
	(15)	(10)	(10)	(13)	(10)	(13)
C8	0.0307	-0.0065	-0.0081	0.0256	-0.0014	0.0304
	(13)	(9)	(10)	(11)	(9)	(12)
C9	0.0250	-0.0063	-0.0049	0.0275	-0.0014	0.0311
	(13)	(9)	(9)	(12)	(9)	(12)
C10	0.0281	-0.0048	-0.0038	0.0281	-0.0040	0.0306
	(13)	(9)	(9)	(12)	(9)	(12)
C11	0.0325	-0.0007	-0.0092	0.0277	-0.0039	0.0374
	(14)	(9)	(10)	(12)	(10)	(13)
C12	0.0322	-0.0040	-0.0156	0.0284	-0.0017	0.0409
	(14)	(9)	(11)	(12)	(10)	(14)
C14	0.0318	-0.0049	-0.0103	0.0271	-0.0023	0.0297
	(13)	(9)	(9)	(11)	(9)	(12)
C15	0.0308	-0.0037	-0.0079	0.0337	-0.0031	0.0304
	(14)	(10)	(9)	(13)	(9)	(12)
C16	0.0462	-0.0037	-0.0084	0.0285	-0.0045	0.0348
	(16)	(11)	(11)	(12)	(10)	(14)
C17	0.0389	-0.0112	-0.0005	0.0503	-0.0044	0.0326
	(17)	(12)	(11)	(17)	(11)	(14)
C18	0.0476	-0.0095	-0.0050	0.073 (2)	0.0051	0.0304
	(18)	(15)	(12)		(14)	(14)
C19	0.0310	-0.0064	-0.0069	0.067 (2)	-0.0082	0.0320
	(17)	(14)	(11)		(13)	(14)
C20	0.0364	-0.0143	-0.0024	0.0402	-0.0039	0.0355
	(16)	(11)	(11)	(15)	(11)	(14)
C22	0.052 (2)	-0.0179	0.0041	0.081 (3)	-0.0351	0.0522
		(18)	(15)		(19)	(19)
O13	0.0370	0.0001 (7)	-0.0165	0.0331	-0.0064	0.0364
	(11)		(8)	(10)	(7)	(10)
O23	0.0367	-0.0159	0.0019	0.0572	-0.0151	0.0704
	(13)	(10)	(10)	(14)	(11)	(16)
O24	0.0345	-0.0140	-0.0133	0.109 (2)	-0.0333	0.0605
	(15)	(13)	(11)		(15)	(15)
O25	0.0340	0.0025 (9)	-0.0122	0.0562	-0.0080	0.0372

	(12)		(8)	(14)	(9)	(11)
O26	0.0493	-0.0134	-0.0087	0.0289 (9)	0.0003 (7)	0.0356
	(13)	(8)	(9)			(10)
O27	0.0299	0.0043 (9)	-0.0093	0.0536	-0.0090	0.0385
	(11)		(8)	(12)	(8)	(10)
O28	0.0446	0.0001 (9)	-0.0261	0.0414	-0.0099	0.0580
	(13)		(10)	(11)	(9)	(13)
S21	0.0362 (4)	-0.0065	-0.0097	0.0369 (3)	-0.0046	0.0415 (4)
		(2)	(2)		(3)	

Crystal and molecular structure for Compound 2

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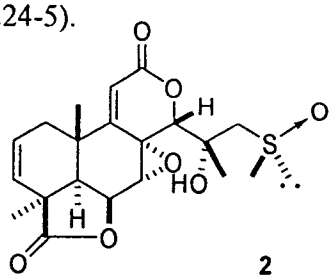
Abstract
We present the crystal and molecular structure of compound **2** (A2224-5).

Comment
The study of the titled structure was undertaken to establish its three dimensional structure. Geometries are tabulated below. All diagrams and calculations were performed using maXus (Bruker Nonius, Delft & MacScience, Japan).



2
(podolactone D)

Experimental
Crystal data

$C_{20}H_{24}O_7S$
$C_{20}H_{24}O_7S$
$M_r = 408.469$
Orthorhombic
$P2_12_12_1$
a = 7.6030 (5)Å
b = 10.470 (2)Å
c = 24.785 (3)Å
α = 90.00°
β = 90.00°
γ = 90.00°
V = 1973.0 (4)Å^3
Z = 4

$D_x = 1.375$ Mg m^{-3}
Density measured by: not measured
fine-focus sealed tube
Mo *K*α radiation
λ = 0.71073
Cell parameters from 3850
θ = 0—27.17 °
μ = 0.204 mm^{-1}
T = 296 K
Prism
Colorless
Crystal source:Local laboratory

Data collection

DIP Image plate
IP
Absorption correction: multi-scan
$T_{min} = 0.890$, $T_{max} = 0.964$
3850 measured reflections

3849 independent reflections
2919 observed reflections
Criterion: >2sigma(I)
$R_{int} = 0.036$
$\theta_{max} = 27.17$ °
h = -9 →0

k = -13 →0

l = -31 →31

Refinement

Refinement on F^2
fullmatrix least squares refinement
R(all) = 0.0664
R(gt) = 0.0494
wR(ref) = 0.1338
wR(gt)= 0.1250
S(ref) = 0.969
3849 reflections
259 parameters
0 restraints
H-atom parameters not refined
Calculated weights calc

Δ/σ_{max} = 0.001
$\Delta\rho_{max}$ = 0.454eÅ^3
$\Delta\rho_{min}$ = -0.264eÅ^3
Extinction correction: *SHELXL*
$Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient = 0.031 (3)
Atomic scattering factors from International Tables Vol C Tables 4.2.6.8 and 6.1.1.4
Flack parameter = 0.06 (11)
Flack H D (1983), *Acta Cryst.* A39, 876-881

Data collection: DIP Image plate
Cell refinement: Scalepack(HKL)
Data reduction: maXus (Mackay et al., 1999)
Program(s) used to refine structure: *SHELXL*-97 (Sheldrick, 1997)

Table 3. *Fractional atomic coordinates and equivalent isotropic thermal parameters (Å^2)*
$U_{eq} = 1/3\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* a_i.a_j$.

	x	y	z	U_{eq}	Occ
C1	0.6426 (5)	0.1330 (4)	0.26983 (11)	0.0517 (8)	1
C2	0.5928 (5)	0.2003 (4)	0.21840 (13)	0.0574 (9)	1
C3	0.4290 (5)	0.2198 (4)	0.20334 (12)	0.0534 (9)	1
C4	0.2729 (5)	0.1658 (3)	0.23252 (11)	0.0458 (7)	1
C5	0.3243 (4)	0.0868 (3)	0.28195 (11)	0.0397 (7)	1
C6	0.1607 (4)	0.0972 (3)	0.31694 (11)	0.0422 (7)	1
C7	0.1870 (4)	0.0775 (3)	0.37665 (11)	0.0424 (7)	1
C8	0.3651 (4)	0.0512 (3)	0.39749 (10)	0.0372 (6)	1
C9	0.5203 (4)	0.0589 (3)	0.36037 (10)	0.0368 (6)	1

C10	0.4921 (4)	0.1401 (3)	0.31021 (11)	0.0395 (6)	1
C11	0.6640 (4)	-0.0014 (3)	0.37634 (11)	0.0446 (7)	1
C12	0.6590 (4)	-0.0820 (3)	0.42547 (12)	0.0456 (7)	1
C14	0.4291 (4)	0.0592 (3)	0.45500 (10)	0.0393 (6)	1
C15	0.2999 (4)	0.0742 (3)	0.50213 (11)	0.0398 (7)	1
C16	0.1622 (4)	-0.0328 (3)	0.50588 (11)	0.0427 (7)	1
C17	0.4012 (5)	0.0860 (4)	0.55501 (11)	0.0512 (8)	1
C18	0.1612 (6)	0.0874 (4)	0.19204 (14)	0.0671 (11)	1
C19	0.1504 (5)	0.2635 (3)	0.25664 (12)	0.0491 (8)	1
C20	0.4755 (5)	0.2787 (3)	0.33064 (12)	0.0484 (8)	1
C22	-0.1193 (5)	-0.1300 (4)	0.55772 (14)	0.0603 (9)	1
O13	0.5355 (3)	-0.0559 (2)	0.46311 (7)	0.0439 (5)	1
O23	-0.1471 (4)	0.0816 (3)	0.49996 (14)	0.0792 (9)	1
O24	0.1027 (4)	0.3645 (3)	0.23753 (10)	0.0661 (7)	1
O25	0.0883 (3)	0.2237 (2)	0.30528 (8)	0.0489 (5)	1
O26	0.2434 (3)	-0.0533 (2)	0.38569 (8)	0.0442 (5)	1
O27	0.2051 (3)	0.1892 (2)	0.49165 (9)	0.0461 (5)	1
O28	0.7553 (4)	-0.1735 (3)	0.43187 (9)	0.0598 (7)	1
S21	-0.02728 (12)	0.02239 (9)	0.54238 (4)	0.0561 (3)	1
H1A	0.7467	0.1726	0.2851	0.062	1
H1B	0.6699	0.0444	0.2622	0.062	1
H2	0.6825	0.2296	0.1961	0.069	1
H3	0.4089	0.2699	0.1730	0.064	1
H5	0.3433	-0.0024	0.2714	0.048	1
H6	0.0759	0.0333	0.3043	0.051	1
H7	0.1002	0.1142	0.4014	0.051	1
H11	0.7679	0.0068	0.3568	0.054	1
H14	0.5100	0.1320	0.4568	0.047	1
H16A	0.2126	-0.1061	0.5241	0.051	1
H16B	0.1275	-0.0591	0.4699	0.051	1

H17A	0.4856	0.1539	0.5521	0.061	1
H17B	0.4611	0.0072	0.5624	0.061	1
H17C	0.3206	0.1044	0.5838	0.061	1
H18A	0.0583	0.0552	0.2099	0.080	1
H18B	0.2296	0.0173	0.1786	0.080	1
H18C	0.1264	0.1413	0.1625	0.080	1
H20A	0.3839	0.2835	0.3572	0.058	1
H20B	0.4474	0.3340	0.3010	0.058	1
H20C	0.5849	0.3050	0.3465	0.058	1
H22A	-0.2341	-0.1189	0.5731	0.072	1
H22B	-0.0449	-0.1735	0.5830	0.072	1
H22C	-0.1284	-0.1796	0.5253	0.072	1
H27	0.2701	0.2508	0.4962	0.055	1

Table 4. *Anisotropic displacement parameters ($Å^2$)*

	U_{11}	U_{12}	U_{13}	U_{22}	U_{23}	U_{33}
C1	0.051 (2)	0.0037 (16)	0.0117 (13)	0.061 (2)	0.0055 (15)	0.0431 (17)
C2	0.063 (2)	0.0042 (19)	0.0141 (16)	0.065 (2)	0.0110 (16)	0.0435 (17)
C3	0.071 (2)	0.0066 (17)	0.0057 (14)	0.051 (2)	0.0087 (14)	0.0377 (16)
C4	0.057 (2)	0.0027 (15)	-0.0035 (12)	0.0454 (19)	0.0001 (13)	0.0347 (15)
C5	0.0506 (18)	-0.0007 (13)	-0.0027 (11)	0.0337 (15)	-0.0004 (12)	0.0348 (14)
C6	0.0489 (19)	-0.0042 (13)	-0.0047 (12)	0.0390 (17)	0.0048 (12)	0.0387 (15)
C7	0.0409 (17)	0.0005 (13)	-0.0022 (11)	0.0451 (18)	0.0023 (13)	0.0412 (16)
C8	0.0410 (17)	-0.0051 (12)	-0.0022 (11)	0.0366 (16)	0.0011 (11)	0.0339 (13)
C9	0.0421 (16)	-0.0006 (12)	0.0007 (11)	0.0340 (15)	-0.0011 (11)	0.0343 (13)
C10	0.0449 (18)	0.0006 (12)	0.0010 (11)	0.0371 (15)	0.0006 (12)	0.0365 (14)
C11	0.0420 (17)	0.0011 (13)	0.0007 (11)	0.052 (2)	0.0007 (13)	0.0399 (15)
C12	0.0464 (19)	0.0061 (15)	-0.0071 (12)	0.0471 (19)	-0.0023 (14)	0.0433 (16)
C14	0.0413 (16)	0.0018 (12)	0.0001 (11)	0.0416 (16)	0.0019 (12)	0.0350 (13)
C15	0.0426	0.0017	0.0020	0.0397	0.0013	0.0371

	(17)	(12)	(11)	(17)	(12)	(14)
C16	0.0530	0.0007	0.0058	0.0378	0.0016	0.0372
	(18)	(14)	(12)	(16)	(12)	(14)
C17	0.057 (2)	-0.0071	0.0026	0.061 (2)	-0.0010	0.0357
		(17)	(13)		(14)	(15)
C18	0.092 (3)	-0.005 (2)	-0.0162	0.064 (2)	-0.0016	0.0450
			(18)		(18)	(18)
C19	0.058 (2)	0.0030	-0.0072	0.047 (2)	0.0056	0.0426
		(15)	(14)		(14)	(16)
C20	0.056 (2)	-0.0073	-0.0005	0.0447	-0.0032	0.0449
		(16)	(14)	(19)	(13)	(16)
C22	0.058 (2)	-0.0028	0.0054	0.063 (2)	0.0110	0.060 (2)
		(18)	(16)		(17)	
O13	0.0498	0.0055	-0.0007	0.0462	0.0039 (9)	0.0358
	(12)	(10)	(8)	(12)		(10)
O23	0.0544	-0.0009	-0.0220	0.0458	0.0107	0.137 (2)
	(17)	(12)	(15)	(15)	(16)	
O24	0.0736	0.0158	-0.0031	0.0566	0.0144	0.0681
	(19)	(13)	(13)	(17)	(13)	(15)
O25	0.0459	0.0084	-0.0018	0.0529	0.0059	0.0478
	(13)	(10)	(9)	(14)	(10)	(12)
O26	0.0535	-0.0091	-0.0026	0.0386	0.0009 (9)	0.0406
	(13)	(10)	(9)	(13)		(11)
O27	0.0488	-0.0021	0.0037	0.0334	-0.0006	0.0560
	(13)	(9)	(10)	(12)	(10)	(13)
O28	0.0682	0.0199	-0.0040	0.0593	0.0031	0.0519
	(17)	(14)	(11)	(16)	(11)	(13)
S21	0.0496 (5)	-0.0029	0.0128 (4)	0.0511 (5)	-0.0168	0.0676 (5)
		(4)			(4)	