C2—O3—C3	60.7 (2)	C8—C9—C4	120.5 (3)
C10—O5—P1	123.3 (2)	O2—C12—N1	127.9 (2)
C11—O6—P1	121.1 (2)	O2—C12—O1	123.0 (2)
C12—N1—C13	125.4 (2)	N1—C12—O1	109.1 (2)
O1—C1—C2	109.4 (2)	C14—C13—C18	119.6 (3)
O1—C1—P1	108.0 (2)	C14—C13—N1	117.2 (2)
C2—C1—P1	110.9 (2)	C18—C13—N1	123.2 (2)
O3—C2—C3	60.5 (2)	C13—C14—C15	120.2 (3)
O3—C2—C1	115.4 (2)	C16—C15—C14	120.5 (3)
C3—C2—C1	121.1 (2)	C17—C16—C15	118.9 (3)
O3—C3—C2	58.80 (15)	C16—C17—C18	121.5 (3)
O3—C3—C4	118.1 (2)	C17—C18—C13	119.3 (3)
C2—C3—C4	124.1 (2)		

Intensity decay was monitored by recollecting the first 50 frames at the end of data collection (8.5 h). A total of 1325 frames (15 s frame^{-1}, 0.3° scan width) was collected. The structure was solved by direct methods and refined successfully in the monoclinic space group $P2_1/c$. Full-matrix least-squares refinement was carried out by minimizing $w(F_o^2 - F_c^2)^2$. The non-H atoms were refined anisotropically, whereas H atoms connected to C1, C2, C3 and N1 were refined isotropically to convergence. The rest of the H atoms were refined using an appropriate riding model. Very fine needles of 0.15 mm in the largest dimension were obtained initially from the diethyl ether solution and data collection was attempted on these crystals. To our surprise, the structure could be solved and partially refined using the CCD system. We are convinced that with the use of sensitive detectors such as the CCD area-detector system, single-crystal X-ray diffraction can truly be used as an analytical tool. Some pertinent data for the small crystal are presented here: $a = 11.9099\,(14)$, $b = 9.4878\,(11)$, $c = 17.946\,(2)$ Å, $\beta = 108.660\,(3)°$, $V = 1921.2\,(4)$ Å^3; 6367 reflections collected, 1793 independent reflections, $R1 = 0.103$, $wR2 = 0.260$, crystal dimensions = 0.15 × <0.03 × <0.03 mm, data collection time 21 h (45 s frame^{-1}).

Data collection: *SMART* (Siemens, 1995). Cell refinement: *SAINT* (Siemens, 1995). Data reduction: *SAINT*. Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *SHELXTL-Plus* (Sheldrick, 1995). Software used to prepare material for publication: *SHELXTL-Plus*.

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The Khellin Quinone

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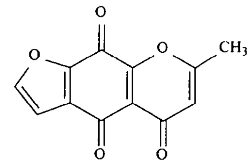
Abstract

The title compound, 2-dehydro-2-methyl-5,8-dioxo-5,8-dihydrofuro[3,2-*g*]chromen-4-one, $C_{12}H_6O_5$, derives from the khellin molecule (7-methyl-4,9-dimethoxy-5*H*-furo[3,2-*g*][1]benzopyran-5-one). The molecular skeleton is nearly planar as in all furobenzopyranones. The intermolecular interactions are strengthened by C—H···O bonds.

Comment

The title compound, (I), has been studied in order to elucidate the transformation of khellin to khellinquinone.



(I)

The molecular skeleton is nearly planar (Fig. 1) as in all furobenzopyranones (El-Sayed, Ammon & Abd El-Rahman, 1988) and the crystal structure is made up of layers. The maximum deviation from the least-squares plane through atoms C1–C12, O1, O3 and O5 is 0.108 (2) Å for C9. Similar to other furobenzopyranones, we have found the O2···O4 distance of 2.808 (2) Å is close to the usual O···O contact distance of 2.8 Å (El-Sayed, Ammon & Abd El-Rahman, 1988). The crystal cohesion is strengthened by non linear C—H···O hydrogen-bonding interactions (Table 2).

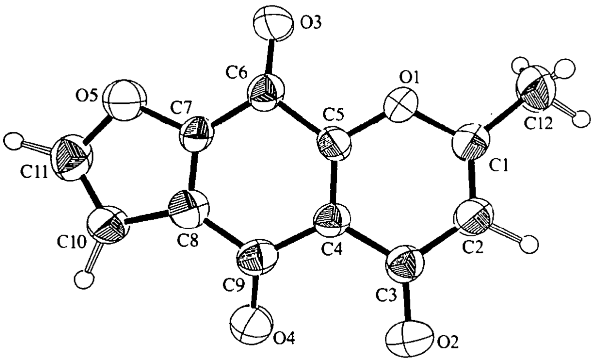


Fig. 1. *ORTEP*II (Johnson, 1971) view of the molecule with the atomic numbering. Displacement ellipsoids are drawn at the 50% probability level. H atoms are shown as small spheres of arbitrary radii.

Experimental

The title compound was prepared by the method of Sreerama Murti & Seshadri (1949). When khellin was treated with nitric acid, the khellin-quinone separated as an orange solid which was filtered and washed free of acid and crystallized from alcohol. Crystals were grown as described previously (Sreerama Murti & Seshadri, 1949; Hila, 1983).

Crystal data

$C_{12}H_6O_5$
$M_r = 230.17$
Monoclinic
$P2_1/c$
$a = 7.552\,(3)$ Å
$b = 8.1720\,(10)$ Å
$c = 15.624\,(3)$ Å
$\beta = 99.29\,(2)°$
$V = 951.6\,(4)$ Å^3
$Z = 4$
$D_x = 1.607$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 25 reflections
$\theta = 13$–$15°$
$\mu = 0.128$ mm^{-1}
$T = 293\,(2)$ K
Parallelepiped
0.30 × 0.08 × 0.05 mm
Orange

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
2154 measured reflections
2076 independent reflections
1353 reflections with $I > 4\sigma(I)$

$R_{\rm int} = 0.010$
$\theta_{\rm max} = 26.97°$
$h = -9 \rightarrow 9$
$k = -10 \rightarrow 0$
$l = 0 \rightarrow 19$
1 standard reflection frequency: 120 min intensity decay: 0.4%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.046$
$wR(F^2) = 0.121$
$S = 1.093$
2076 reflections
179 parameters
All H atoms refined
$w = 1/[\sigma^2(F_o^2) + (0.051P)^2 + 0.2658P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\rm max} = -0.001$
$\Delta\rho_{\rm max} = 0.207$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.174$ e Å^{-3}
Extinction correction: *SHELXL*93
Extinction coefficient: 0.0041 (13)
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

C1—O1	1.380 (2)	C6—O3	1.209 (2)
C1—C12	1.475 (3)	C7—O5	1.364 (2)
C3—O2	1.227 (2)	C9—O4	1.208 (2)
C4—C9	1.493 (3)	C11—O5	1.370 (3)
C5—O1	1.340 (2)		
C2—C1—C12	127.4 (2)	O3—C6—C5	121.9 (2)
O1—C1—C12	111.3 (2)	O4—C9—C8	121.0 (2)
O2—C3—C2	122.8 (2)	O4—C9—C4	123.4 (2)
O2—C3—C4	123.4 (2)	C5—O1—C1	118.9 (2)
O3—C6—C7	126.8 (2)	C7—O5—C11	105.3 (2)

Table 2. *Hydrogen-bonding geometry (Å, °)*

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C2—H2···O2$^{\rm i}$	0.97 (2)	2.50 (2)	3.462 (3)	173 (3)
C11—H11···O3$^{\rm ii}$	0.95 (2)	2.47 (2)	3.389 (3)	161 (3)
C12—H121···O4$^{\rm iii}$	0.85 (3)	2.51 (3)	3.232 (3)	143 (3)
C11—H11···O5$^{\rm ii}$	0.95 (2)	2.65 (3)	3.275 (3)	123 (3)
C12—H122···O1$^{\rm iv}$	1.01 (5)	2.67 (5)	3.644 (4)	162 (3)

Symmetry codes: (i) $2-x, 1-y, -z$; (ii) $-x, \frac{1}{2}+y, \frac{1}{2}-z$; (iii) $x, y-1, z$; (iv) $1-x, -y, -z$.

Data collection: *CAD*-4 *EXPRESS* (Enraf–Nonius, 1994). Cell refinement: *CAD*-4 *EXPRESS*. Data reduction: *MolEN* (Fair, 1990). Program(s) used to solve structure: *MULTAN*80 (Main *et al.*, 1980). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ORTEP*II (Johnson, 1971). Software used to prepare material for publication: *SHELXL*93.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: AB1487). Services for accessing these data are described at the back of the journal.

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