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Structure of 3-Acetyl-4-hydroxy-6-phenyl-2-pyrone, C₁₃H₁₀O₄

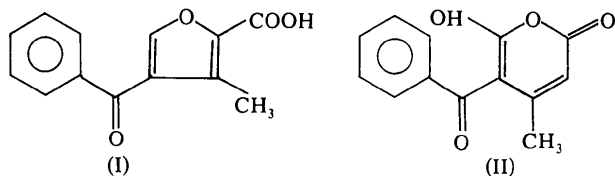
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Abstract. $M_r = 230.21$, m.p. ~ 443 K, monoclinic, $P2_1/c$, $a = 5.558$ (5), $b = 24.301$ (5), $c = 7.982$ (3) Å, $\beta = 92.22$ (5)°, $V = 1077$ (1) Å³, $Z = 4$, D_m (floatation) = 1.423, $D_x = 1.420$ g cm⁻³, Mo $K\alpha$ radiation, $\lambda = 0.7107$ Å, $\mu = 1.36$ cm⁻¹, $F(000) = 480$, $T = 293$ K, $R = 0.051$ for 1008 observed reflections. The O of the acetyl group of the pyrone ring is hydrogen-bonded to the O of the hydroxyl group of the same molecule. This intramolecular hydrogen bond, though very strong [O...O 2.464 (7) Å], is asymmetrical. There are no unusual bond lengths or angles.

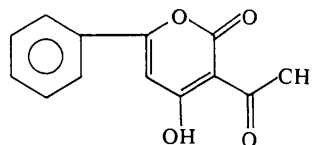
Introduction. C₁₃H₁₀O₄ is a plant product isolated from *Pogostemon heyneanus* Benth-Sym. (Purushothaman, Sarada & Connolly, 1984). The compound was supplied by Dr Purushothaman of Captain Srinivasamurti Research Institute for Ayurveda and Siddha. It has been reported to have 70% regression against methylcholanthrene-induced fibrosarcoma (Purushothaman, Sarada & Connolly, 1984). The present study was undertaken to establish the chemical structure, the following having been suggested:



* Contribution No. 660.

0108-2701/85/050802-03\$01.50

However, the structure arrived at from single-crystal X-ray studies is:



which also agrees with spectroscopic results.

Experimental. Pale-yellow rectangular crystals (from ethyl acetate). Crystal $0.7 \times 0.3 \times 0.3$ mm. Nonius CAD-4 diffractometer, monochromatic Mo $K\alpha$. 1528 independent reflections with $2\theta \leq 48^\circ$; 1008 with $I \geq 3\sigma(I)$. Two standard reflections ($\bar{3}62$ and $\bar{2}8\bar{2}$), maximum variation in intensity 2.6%. 18 reflections with $9 < \theta < 16^\circ$ for lattice-parameter measurement and refinement. Max. $h = 6$, max. $k = 26$, max. $l = 8$, min. $l = -8$. Lp correction, no absorption correction. Structure solution by *MULTAN80* (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980). Anisotropic block-diagonal least-squares refinement on F (Shiono, 1968) for non-hydrogen atoms; H atoms fixed geometrically and checked by ΔF synthesis and isotropic refinement. Final $R = 0.051$, $wR = 0.083$ for 194 parameters, goodness of fit 0.50. Cruickshank's weighting scheme (Cruickshank, Bujosa, Lovell & Truter, 1961). Final ΔF map had no peak > 0.3 e Å⁻³. $(\Delta/\sigma)_{\max} = 0.5$, $(\Delta/\sigma)_{\text{mean}} = 0.2$. No correction for secondary extinction. Scattering factors for non-

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Table 1. Fractional positional parameters ($\times 10^4$, for H $\times 10^3$) and isotropic thermal parameters, with e.s.d.'s in parentheses

For non-H atoms $B_{eq} = \frac{1}{3}(\beta_{11}a^2 + \beta_{22}b^2 + \beta_{33}c^2 + \beta_{13}acc\beta)$;
 $\sigma B_{eq} = \frac{1}{3}[(a^2\sigma\beta_{11})^2 + (b^2\sigma\beta_{22})^2 + (c^2\sigma\beta_{33})^2 + (2acc\sigma\beta_{13})^2]^{1/2}$.

	x	y	z	B_{eq} or $B(\text{\AA}^2)$
O(1)	8596 (7)	2394 (2)	8892 (5)	3.9 (1)
C(2)	8885 (11)	2962 (2)	9058 (8)	3.8 (1)
C(3)	7089 (10)	3302 (2)	8226 (7)	3.6 (1)
C(4)	5176 (11)	3044 (2)	7346 (8)	3.9 (1)
C(5)	4980 (11)	2468 (3)	7268 (8)	4.0 (1)
C(6)	6688 (10)	2156 (3)	8046 (7)	3.5 (1)
C(7)	6798 (11)	1549 (3)	8116 (7)	3.8 (1)
C(8)	4930 (12)	1238 (3)	7389 (9)	4.8 (1)
C(9)	5024 (14)	671 (3)	7495 (11)	5.7 (2)
C(10)	6909 (13)	413 (3)	8318 (9)	5.5 (2)
C(11)	8763 (14)	719 (3)	9027 (10)	5.3 (2)
C(12)	8717 (12)	1288 (2)	8937 (8)	4.4 (1)
O(13)	10611 (7)	3102 (2)	9914 (6)	5.0 (1)
O(14)	3479 (8)	3335 (2)	6546 (6)	5.1 (1)
C(15)	7205 (11)	3899 (3)	8280 (8)	4.3 (1)
C(16)	9261 (13)	4198 (3)	9118 (10)	5.6 (2)
O(17)	5554 (9)	4171 (2)	7583 (7)	5.8 (1)
H(1)	867 (15)	460 (3)	882 (10)	8 (2)
H(2)	938 (14)	413 (4)	1039 (12)	8 (2)
H(3)	1078 (16)	408 (4)	878 (12)	8 (2)
H(C5)	357 (10)	231 (2)	668 (7)	4 (1)
H(C8)	362 (11)	139 (3)	672 (7)	5 (1)
H(C9)	362 (12)	47 (3)	696 (9)	6 (2)
H(C10)	675 (12)	3 (3)	841 (9)	7 (2)
H(C11)	1021 (10)	54 (2)	956 (7)	4 (1)
H(C12)	998 (12)	150 (3)	943 (8)	5 (1)
H(O14)	392 (17)	372 (4)	688 (10)	10 (2)

hydrogen atoms from *International Tables for X-ray Crystallography* (1968) and for H atoms from Stewart, Davidson & Simpson (1965).

Discussion. Final atomic parameters and equivalent isotropic temperature coefficients are listed in Table 1.* The atom numbering is given in Fig. 1. The bond lengths and bond angles are in Table 2. The average e.s.d. in bond lengths is 0.008 Å and that in bond angles is 0.5°. The packing of the molecule, viewed down the *a* axis, is shown in Fig. 1. The distance between H(C5) and H(C8) is 2.24 (9) Å which is less than (but within 2σ of) the minimum contact distance between H...H atoms. The best plane through the molecule makes an angle of 58.4 (1)° with the *bc* plane, and the longest axis of the molecule is almost parallel to the *b* axis. There is an intramolecular hydrogen bond between the O of the acetyl group and the O of the hydroxyl group. This hydrogen bond is strong with an O...O distance of 2.464 (7) Å, but is asymmetrical, as the proton obtained from the difference Fourier map is closer to O(14) than O(17). The torsion angles around the pyrone ring agree well with those observed in

* Lists of structure factors, anisotropic thermal parameters and least-squares-planes' data have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42010 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

similar structures. The pyrone ring is almost planar [$\sum(\Delta/\sigma)^2 = 10.8$] with C(2) deviating -0.013 (5) Å from the least-squares plane. The angle between the phenyl-ring plane and the best plane through the pyrone-ring atoms is 3.5 (2)° and that between the pyrone ring and the acetyl group is 2.6 (3)°.

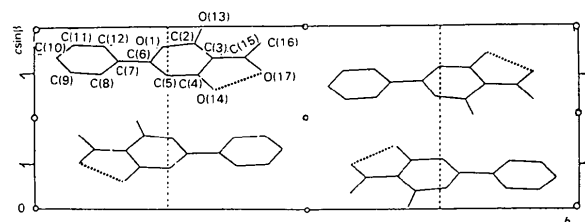


Fig. 1. Packing of the molecule with the atom numbering; projection is down the *a* axis.

Table 2. Bond lengths (Å) and angles (°) with e.s.d.'s in parentheses

O(1)—C(2)	1.394 (7)	C(11)—C(12)	1.383 (9)
O(1)—C(6)	1.364 (7)	C(15)—C(16)	1.491 (10)
C(2)—C(3)	1.438 (8)	C(15)—O(17)	1.245 (8)
C(2)—O(13)	1.205 (7)	C(5)—H(C5)	0.98 (5)
C(3)—C(4)	1.400 (8)	C(8)—H(C8)	0.96 (6)
C(3)—C(15)	1.452 (9)	C(9)—H(C9)	1.00 (7)
C(4)—C(5)	1.406 (8)	C(10)—H(C10)	0.94 (7)
C(4)—O(14)	1.322 (7)	C(11)—H(C11)	1.00 (6)
C(5)—C(6)	1.348 (8)	C(12)—H(C12)	0.94 (7)
C(6)—C(7)	1.476 (9)	O(14)—H(O14)	1.00 (10)
C(7)—C(8)	1.393 (9)	C(16)—H(1)	1.06 (7)
C(7)—C(12)	1.385 (9)	C(16)—H(2)	1.03 (9)
C(8)—C(9)	1.381 (10)	C(16)—H(3)	0.94 (9)
C(9)—C(10)	1.367 (10)	O(17)—H(O14)	1.52 (9)
C(10)—C(11)	1.375 (10)	O(14)...O(17)	2.464 (7)
O(1)—C(2)—C(3)	116.7 (5)	C(4)—C(5)—C(6)	119.2 (5)
O(1)—C(2)—O(13)	114.8 (4)	C(5)—C(4)—O(14)	117.2 (4)
O(1)—C(6)—C(7)	112.0 (4)	C(5)—C(6)—C(7)	127.3 (5)
O(1)—C(6)—C(5)	120.6 (5)	C(6)—C(7)—C(8)	119.8 (5)
C(2)—O(1)—C(6)	123.5 (4)	C(6)—C(7)—C(12)	120.4 (5)
C(2)—C(3)—C(4)	118.4 (5)	C(7)—C(8)—C(9)	119.3 (5)
C(2)—C(3)—C(15)	122.1 (5)	C(7)—C(12)—C(11)	119.7 (5)
C(3)—C(2)—O(13)	128.5 (5)	C(8)—C(7)—C(12)	119.8 (5)
C(3)—C(4)—C(5)	121.6 (5)	C(8)—C(9)—C(10)	120.9 (6)
C(3)—C(4)—O(14)	121.2 (4)	C(9)—C(10)—C(11)	119.9 (6)
C(3)—C(15)—C(16)	122.2 (5)	C(10)—C(11)—C(12)	120.4 (6)
C(3)—C(15)—O(17)	119.1 (5)	C(16)—C(15)—O(17)	118.7 (5)
C(4)—C(3)—C(15)	119.6 (5)		

Table 3. Comparison of the pyrone ring geometry (where the ring is almost planar)

(a) Distances (Å) and angles (°)					
C—O	O—C'	C—O—C'	C'—O'	O—C'—O'	Reference
1.364 (7)	1.394 (7)	123.5 (4)	1.205 (7)	114.8 (4)	Present work
1.382 (9)	1.385 (9)	122.4 (4)	1.212 (9)	115.9 (4)	Lai & Marsh (1974)
1.366 (2)	1.395 (2)	122.1 (2)	1.204 (3)	116.6 (2)	Shen & Bryan (1975)
1.368 (2)	1.384 (2)	122.1 (2)	1.207 (3)	116.6 (2)	
(b) Hydrogen-bond distances (Å) and angles (°)					
D...A	D—H	D...A	H...A	D—H...A	
O(14)...O(17 ⁱⁱ)	1.00 (10)	2.464 (7)	1.52 (9)	156 (9)	
C(8)...O(13 ⁱⁱⁱ)	0.96 (6)	3.445 (8)	2.49 (6)	172 (6)	
C(5)...O(13 ⁱⁱⁱ)	0.98 (5)	3.315 (8)	2.35 (5)	171 (5)	
C(10)...O(17 ⁱⁱⁱ)	0.94 (7)	3.380 (9)	2.56 (7)	146 (6)	
C(12)...O(14 ^{iv})	0.94 (7)	3.428 (8)	2.56 (7)	153 (6)	

Symmetry code

- (i) x, y, z
 (ii) $x - 1, -y + \frac{1}{2}, z - \frac{1}{2}$
 (iii) $-x + 1, y - \frac{1}{2}, -z + \frac{3}{2}$
 (iv) $x + 1, -y + \frac{1}{2}, z + \frac{1}{2}$

Some of the short C...O intermolecular contacts can also be considered as possible C—H...O hydrogen bonds (Berkovitch-Yellin & Leiserowitz, 1984). Table 3(b) gives the relevant distances and angles.

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SHORT COMMUNICATIONS

Contributions intended for publication under this heading should be expressly so marked; they should not exceed about 1000 words; they should be forwarded in the usual way to the appropriate Co-editor; they will be published as speedily as possible.

Acta Cryst. (1985). **C41**, 804–805

Nouvelle description du tétrafluorure de neptunium hydraté, $\text{Np}_3\text{F}_{12}\cdot\text{H}_2\text{O}$. Par ALAIN COUSSON, *Institut Curie, Section de Physique et Chimie, ERA CNRS 1006, 11 rue Pierre et Marie Curie, 75231 Paris CEDEX 05, France* et MADELEINE GASPERIN, *Laboratoire de Minéralogie–Cristallographie, associé au CNRS, Université Pierre et Marie Curie, 4 place Jussieu, 75230 Paris CEDEX 05, France*

(Reçu le 30 mai 1984, accepté le 4 décembre 1984)

Abstract

The triclinic cell of $\text{Np}_3\text{F}_{12}\cdot\text{H}_2\text{O}$ [Cousson, Abazli, Pagès & Gasperin (1983), *Acta Cryst.* **C39**, 425–427] can be reduced to a monoclinic cell. Relative atomic positions are identical with those of the triclinic cell but the polyhedra surrounding Np are easier to describe. $M_r = 956.99$, monoclinic, Cm , $a = 12.023$ (5), $b = 11.723$ (5), $c = 8.435$ (4) Å, $\beta = 128.77$ (2)°, $V = 926.9$ (5) Å³, $Z = 4$, $D_x = 6.86$ Mg m⁻³, Mo $K\alpha$, $\lambda = 0.7107$ Å, $\mu = 25.8$ mm⁻¹, $F(000) = 1455$, room temperature, $R(F) = 0.045$, $wR = 0.058$, $S = 0.596$ for 1342 independent reflexions.

Introduction

A la suite de la parution de l'article décrivant la structure de $\text{Np}_3\text{F}_{12}\cdot\text{H}_2\text{O}$ (Cousson, Abazli, Pagès & Gasperin, 1983), un de nos confrères (Tillmanns, 1984) nous faisait remarquer

que la maille pouvait être réduite et que les tables de matrices de Niggli (*International Tables for X-ray Crystallography*, 1969) montraient que le composé était descriptible dans une maille monoclinique base centrée C .

Partie expérimentale

Après transformation des indices par la matrice appropriée, la structure a donc été à nouveau affinée dans la maille monoclinique C , dans des conditions identiques à celles du travail antérieur, à ceci près qu'une correction de l'extinction secondaire a depuis été introduite dans le programme d'affinement. De plus, il a semblé préférable de n'affecter que les atomes lourds de coefficients de température anisotropes.

Un premier essai dans le groupe $C2/m$ ne donne pas de résultats satisfaisants: malgré un facteur R de 0,089, les facteurs de température sont inhomogènes et élevés et un calcul de distances montre que l'atome F(14) n'est plus lié.