(Bailey & Brown, 1967*a*,*b*); 1,4-bis(isopropylamino)-anthraquinone (Brown & Mitchell, 1982); 1-[2-(diethylamino)ethylamino]anthraquinone and the 1,5-bis derivative (Almond *et al.*, 1983); 1-[4-(dimethylamino)phenylamino]-4-hydroxyanthraquinone (Foitzik, Paulus & Haase, 1986*a*); and in 1,5-bis-[4-(pentyloxy)phenylamino]anthraquinone (Foitzik, Paulus & Haase, 1986*b*).

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Structure of 6-Hydroperoxy-5-hydroxythymine Hemihydrate (I) and *trans*-5,6-Dihydroxy-1,3-dimethylthymine (II)*

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Abstract. (I) $C_5H_8N_2O_5.\frac{1}{2}H_2O$, $M_r = 185\cdot1$, triclinic, $P\bar{1}$, $a = 6\cdot125$ (2), $b = 10\cdot849$ (3), $c = 11\cdot674$ (3) Å, $\alpha = 98\cdot21$ (2), $\beta = 91\cdot38$ (2), $\gamma = 94\cdot66$ (2)°, $V = 764\cdot7$ (2) Å^3, $Z = 4$, $D_x = 1\cdot61$ Mg m^{-3}, λ(Cu $K\alpha$) = 1·54178 Å, $\mu = 1\cdot31$ mm^{-1}, $F(000) = 388$, $T = 295$ K, final $R = 0\cdot047$, $wR = 0\cdot061$ for 1952 observed data with $F_o > 3\sigma(F_o)$. (II) $C_7H_{12}N_2O_4$, $M_r = 188\cdot2$, orthorhombic, *Pbca*, $a = 8\cdot265$ (2), $b = 21\cdot128$ (3), $c = 10\cdot587$ (6) Å, $V = 1848\cdot8$ (2) Å^3, $Z = 8$, $D_x = 1\cdot35$ Mg m^{-3}, λ(Cu $K\alpha$) = 1·54178 Å, $\mu = 0\cdot96$ mm^{-1}, $F(000) = 800$, $T = 296$ K, final $R = 0\cdot066$, $wR = 0\cdot067$ for 913 observed data with $F_o > 3\sigma(F_o)$. The thymine ring has a half-chair conformation in both (I) and (II). The hydroxyl group on C(5) is *cis* to the peroxy group in (I) and *trans* to the hydroxyl on C(6) in (II). (I) shows extensive hydrogen bonding (ten bonds ranging from 2·67 to 2·98 Å) involving all possible donors and several close intermolecular approaches which may explain, in part, the rather high density. In (II) only two hydrogen bonds are possible [O(5)···O(4) at 2·82 Å and O(6)···O(2) at 2·70 Å] and its density is that more normally found for molecules of this type.

* 5,6-Dihydro-6-hydroperoxy-5-hydroxy-5-methyl-2,4(1*H*,3*H*)-pyrimidinedione hemihydrate (I) and 5,6-dihydro-5,6-dihydroxy-1,3-dimethyl-2,4(1*H*,3*H*)-pyrimidinedione (II).

Experimental. Crystals of both (I) and (II) supplied by the late S. Y. Wang (formerly of Johns Hopkins University). Crystals used had dimensions 0·20 × 0·25 × 0·15 (I) and 0·30 × 0·25 × 0·08 (II). Automated Nicolet P3F diffractometer with incident-beam monochromator, 20 centered reflections with $8 < 2\theta < 60°$ for (I) and 25 centered reflections with $8 < 2\theta < 70°$ for (II) used for determining lattice parameters. Data corrected for Lorentz and polarization effects, absorption ignored. $[(\sin\theta)/\lambda]_{max} = 0\cdot547$ for both, range of *hkl*: $-6 \le h \le 0$, $-8 \le k \le 11$, $-12 \le l \le 12$ for (I); $-8 \le h \le 8$, $0 \le k \le 22$, $0 \le l \le 11$ for (II). For (I), standards 300, 004, 247 monitored every 60 reflections showed a linear decrease of 10% and were then used to scale the data, θ–2θ scan technique ($2\cdot0 + \Delta\alpha_1\alpha_2$), constant scan rate of 30° min^{-1} (crystals were unstable). 2347 reflections measured, 1999 unique, 1952 considered observed [$|F_o| > 3\sigma(|F_o|)$] and used for refinement. For (II): standards, 400, 0,10,0, 0,0,14 monitored every 60 reflections showed a 2·5% random variation over data collection, θ–2θ mode, same scan width as (I), scan rate a function of count rate (4° min^{-1} minimum, 30° min^{-1} maximum), 3014 reflections measured, 1154 unique, 913 considered observed [$|F_o| > 3\sigma(|F_o|)$] and used for refinement.

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Table 1. *Atomic coordinates and equivalent isotropic displacement parameters* ($Å^2 \times 10^2$) *for molecule* (I)

Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
N(1)	1·0708 (4)	0·1005 (2)	0·1508 (2)	4·0 (1)
C(2)	0·9616 (4)	−0·0042 (2)	0·1747 (2)	3·5 (1)
N(3)	0·8945 (3)	−0·0024 (2)	0·2860 (2)	3·6 (1)
C(4)	0·9338 (3)	0·0927 (2)	0·3748 (2)	2·8 (1)
C(5)	1·1143 (4)	0·1909 (2)	0·3535 (2)	3·1 (1)
C(6)	1·1025 (4)	0·2171 (2)	0·2281 (2)	3·9 (1)
O(2)	0·9182 (3)	−0·0974 (2)	0·1024 (2)	5·0 (1)
C(7)	1·3333 (4)	0·1405 (3)	0·3787 (3)	4·1 (1)
O(4)	0·8435 (3)	0·0929 (2)	0·4659 (1)	3·7 (1)
O(5)	1·1043 (3)	0·3019 (2)	0·4326 (2)	3·6 (1)
O(6)	0·9539 (3)	0·3026 (2)	0·2069 (2)	5·6 (1)
O(8)	0·7442 (3)	0·2521 (2)	0·2340 (2)	5·8 (1)
N(1′)	0·4866 (3)	0·5392 (2)	0·3469 (2)	3·7 (1)
C(2′)	0·3622 (4)	0·6194 (2)	0·4080 (2)	3·1 (1)
N(3′)	0·3105 (3)	0·7221 (2)	0·3575 (2)	3·2 (1)
C(4′)	0·3839 (4)	0·7506 (2)	0·2547 (2)	3·3 (1)
C(5′)	0·4697 (4)	0·6421 (2)	0·1756 (2)	3·2 (1)
C(6′)	0·6036 (4)	0·5652 (2)	0·2474 (2)	3·4 (1)
C(7′)	0·2711 (4)	0·5628 (3)	0·1152 (2)	4·3 (1)
O(2′)	0·2937 (3)	0·6052 (2)	0·5037 (2)	3·8 (1)
O(4′)	0·3730 (3)	0·8526 (2)	0·2261 (2)	5·0 (1)
O(5′)	0·5976 (3)	0·6862 (2)	0·0886 (1)	3·7 (1)
O(6′)	0·8187 (3)	0·6210 (2)	0·2748 (2)	4·2 (1)
O(8′)	0·7988 (3)	0·7397 (2)	0·3470 (2)	4·2 (1)
W	0·6023 (7)	0·1273 (4)	0·0248 (3)	10·0 (2)

Table 2. *Atomic coordinates and equivalent isotropic displacement parameters* ($Å^2 \times 10^2$) *for molecule* (II)

Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
N(1)	0·5970 (4)	0·7067 (2)	0·0093 (3)	5·0 (1)
C(1)	0·5670 (6)	0·7753 (2)	0·0129 (5)	6·4 (2)
C(2)	0·6788 (6)	0·6803 (2)	0·1032 (5)	5·3 (2)
O(2)	0·7190 (5)	0·7076 (2)	0·2005 (3)	7·4 (1)
N(3)	0·7243 (5)	0·6161 (2)	0·0891 (4)	5·9 (2)
C(3)	0·8347 (8)	0·5896 (3)	0·1836 (5)	8·8 (3)
C(4)	0·6655 (6)	0·5770 (2)	0·9954 (5)	5·4 (2)
O(4)	0·7115 (4)	0·5224 (2)	0·9874 (3)	6·9 (1)
C(6)	0·5733 (5)	0·6741 (2)	0·8894 (4)	4·6 (2)
O(6)	0·7124 (4)	0·6775 (2)	0·8127 (3)	5·7 (1)
C(5)	0·5365 (5)	0·6050 (2)	0·9122 (4)	4·8 (2)
O(5)	0·3887 (4)	0·6049 (2)	0·9858 (3)	6·5 (1)
C(7)	0·5157 (6)	0·5691 (2)	0·7902 (4)	6·3 (2)

Table 3. *Bond lengths* (Å) *and angles* (°) *for molecule* (I)

N(1)–C(2)	1·338 (3)	N(1)–C(6)	1·442 (3)
C(2)–N(3)	1·371 (3)	C(2)–O(2)	1·228 (3)
N(3)–C(4)	1·356 (3)	C(4)–C(5)	1·523 (3)
C(4)–O(4)	1·209 (3)	C(5)–C(6)	1·532 (4)
C(5)–C(7)	1·527 (3)	C(5)–O(5)	1·415 (3)
C(6)–O(6)	1·393 (3)	O(6)–O(8)	1·416 (3)
N(1′)–C(2′)	1·343 (3)	N(1′)–C(6′)	1·429 (3)
C(2′)–N(3′)	1·387 (3)	C(2′)–O(2′)	1·229 (3)
N(3′)–C(4′)	1·359 (3)	C(4′)–C(5′)	1·526 (3)
C(4′)–O(4′)	1·207 (3)	C(5′)–C(6′)	1·533 (4)
C(5′)–C(7′)	1·528 (4)	C(5′)–O(5′)	1·410 (3)
C(6′)–O(6′)	1·414 (3)	O(6′)–O(8′)	1·451 (3)
C(6)–N(1)–C(2)	125·5 (2)	N(3)–C(2)–N(1)	116·3 (2)
O(2)–C(2)–N(1)	123·4 (2)	O(2)–C(2)–N(3)	120·3 (2)
C(4)–N(3)–C(2)	126·7 (2)	C(5)–C(4)–N(3)	114·2 (2)
O(4)–C(4)–N(3)	122·0 (2)	O(4)–C(4)–C(5)	123·6 (2)
C(6)–C(5)–C(4)	111·1 (2)	C(7)–C(5)–C(4)	107·4 (2)
C(7)–C(5)–C(6)	109·9 (2)	O(5)–C(5)–C(4)	110·7 (2)
O(5)–C(5)–C(6)	111·2 (2)	O(5)–C(5)–C(7)	106·4 (2)
C(5)–C(6)–N(1)	109·5 (2)	O(6)–C(6)–N(1)	113·4 (2)
O(6)–C(6)–C(5)	114·6 (2)	O(8)–O(6)–C(6)	107·0 (2)
C(6′)–N(1′)–C(2′)	124·2 (2)	N(3′)–C(2′)–N(1′)	116·7 (2)
O(2′)–C(2′)–N(1′)	123·3 (2)	O(2′)–C(2′)–N(3′)	120·0 (2)
C(4′)–N(3′)–C(2′)	125·3 (2)	C(5′)–C(4′)–N(3′)	115·0 (2)
O(4′)–C(4′)–N(3′)	122·4 (2)	O(4′)–C(4′)–C(5′)	122·5 (2)
C(6′)–C(5′)–C(4′)	109·7 (2)	C(7′)–C(5′)–C(4′)	107·4 (2)
C(7′)–C(5′)–C(6′)	110·9 (2)	O(5′)–C(5′)–C(4′)	110·8 (2)
O(5′)–C(5′)–C(6′)	110·6 (2)	O(5′)–C(5′)–C(7′)	107·3 (2)
C(5′)–C(6′)–N(1′)	110·2 (2)	O(6′)–C(6′)–N(1′)	113·3 (2)
O(6′)–C(6′)–C(5′)	112·4 (2)	O(8′)–O(6′)–C(6′)	107·0 (1)

Table 4. *Bond lengths* (Å) *and angles* (°) *for molecule* (II)

N(1)–C(1)	1·471 (5)	N(1)–C(2)	1·326 (5)
N(1)–C(6)	1·457 (5)	C(2)–O(2)	1·227 (5)
C(2)–N(3)	1·415 (6)	N(3)–C(3)	1·465 (5)
N(3)–C(4)	1·379 (6)	C(4)–O(4)	1·216 (5)
C(4)–C(5)	1·505 (6)	C(6)–O(6)	1·409 (5)
C(6)–C(5)	1·511 (6)	C(5)–O(5)	1·449 (5)
C(5)–C(7)	1·508 (6)		
C(2)–N(1)–C(1)	118·8 (4)	C(6)–N(1)–C(1)	117·7 (4)
C(6)–N(1)–C(2)	121·5 (4)	O(2)–C(2)–N(1)	124·7 (5)
N(3)–C(2)–N(1)	117·4 (4)	N(3)–C(2)–O(2)	117·9 (4)
C(3)–N(3)–C(2)	117·3 (4)	C(4)–N(3)–C(2)	123·8 (4)
C(4)–N(3)–C(3)	118·8 (4)	O(4)–C(4)–N(3)	120·5 (4)
C(5)–C(4)–N(3)	115·8 (4)	C(5)–C(4)–O(4)	123·6 (5)
O(6)–C(6)–N(1)	111·6 (4)	C(5)–C(6)–N(1)	110·1 (3)
C(5)–C(6)–O(6)	107·7 (4)	C(6)–C(5)–C(4)	109·4 (4)
O(5)–C(5)–C(4)	106·4 (4)	O(5)–C(5)–C(6)	104·9 (4)
C(7)–C(5)–C(4)	112·6 (4)	C(7)–C(5)–C(6)	111·9 (4)
C(7)–C(5)–O(5)	111·3 (4)		

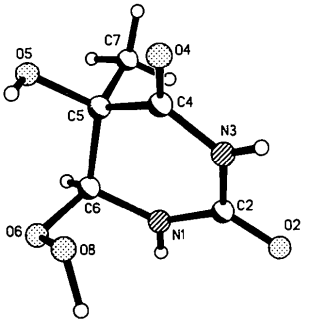

Fig. 1. Diagram of the results of the X-ray analysis of (I). Only one of the two similar molecules in the asymmetric unit is shown.

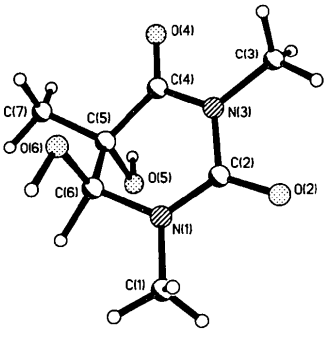

Fig. 2. Diagram of the results of the X-ray analysis of (II).

Both structures solved by direct methods and refined by full-matrix least squares. $\sum w(|F_o| - |F_c|)^2$ minimized where $w = 1/[\sigma^2(|F_o|) + g(|F_o|)^2]$ and $g(F_o)^2$ is included to account for random instrument error (g estimated to be 0·00025). For (I), 262 parameters refined: atom coordinates and anisotropic temperature factors for all non-H atoms, H atoms from difference maps, coordinates refined if involved in a hydrogen bond, otherwise idealized (C—H distance set at 0·96 Å, CH angles set to trigonal or tetrahedral values) then allowed to ride on covalently bonded neighbors, thermal parameters constant. $(\Delta/\sigma)_{max} = 0{\cdot}371$, $R = 0{\cdot}047$, $wR = 0{\cdot}061$, $S = 3{\cdot}1$. For II, 133 parameters refined: same as for (I), $(\Delta/\sigma)_{max} = 0{\cdot}089$, $R = 0{\cdot}066$, $wR = 0{\cdot}069$, $S = 1{\cdot}7$. Final difference Fourier excursions 0·66 and −0·33 for (I); 0·33 and −0·24 for (II). Atomic scattering factors from *International Tables for X-ray Crystallography* (1974). Atom numbering for Tables 1 and 2, which list atom coordinates for (I) and (II), follows that shown in Figs. 1 and 2.* Bond lengths and angles for (I) and (II) are given in Tables 3 and 4, respectively.

Related literature. *cis*-Thymine glycol shows a density similar to that of molecule (I) (Flippen, 1973).

* Lists of structure factors, bond lengths, bond angles, H-atom coordinates, H-bond parameters, torsion angles and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44131 (21 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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O-Acetylisomoschatoline

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Abstract. $C_{20}H_{15}NO_5$, $M_r = 349{\cdot}35$, triclinic, $P\bar{1}$, $a = 7{\cdot}451$ (1), $b = 10{\cdot}458$ (3), $c = 10{\cdot}666$ (4) Å, $\alpha = 97{\cdot}55$ (2), $\beta = 95{\cdot}86$ (2), $\gamma = 100{\cdot}20$ (2)°, $V = 804{\cdot}2$ (2) Å^3, $Z = 2$, $D_x = 1{\cdot}443$ g cm^{-3}, λ(Cu $K\alpha$) = 1·54178 Å, $\mu = 8{\cdot}26$ cm^{-1}, $F(000) = 364$, $T = 298$ K, $R = 0{\cdot}043$ for 1921 unique reflections. The oxoaporphine isomoschatoline (3-hydroxy-1,2-dimethoxy-7*H*-dibenzo[*de,g*]quinolin-7-one) was isolated from *Cleistopholis patens* and converted into the acetyl derivative to aid in assignment of substituents. The acetyl group is located at the C(3) position in the alkaloid. The alkaloid framework is planar, and the plane of the acetoxy moiety is almost perpendicular [96·5 (3)°] to the alkaloid molecular plane.

Experimental. The sample recrystallized from chloroform, space group *Pbca*, contained disordered molecule of solvent, decomposed during data collection, refined only to $R = 0{\cdot}133$. Good transparent light yellow crystal from ethanol, 0·30 × 0·35 × 0·40 mm; Nicolet $R3m/\mu$ update of $P2_1$ diffractometer; data collected in θ:2θ mode ($3 \le 2\theta \le 120°$, 4·00 to 29·3° min^{-1}), graphite-monochromated Cu $K\alpha$ radiation; lattice parameters from a least-squares refinement of 17 reflections ($24{\cdot}36 \le 2\theta \le 46{\cdot}82°$), angles measured by a centering routine; no systematic absences and statistics consistent with space group $P\bar{1}$, Laue symmetry $\bar{1}$; monitored reflections ($1\bar{1}\bar{2}$ and $2\bar{2}\bar{1}$) showed no significant changes in intensities; 2170 independent reflections measured ($0 \le h \le 9$, $-12 \le k \le 12$, $-12 \le l \le 12$), 1921 with $I \ge 2{\cdot}5\sigma(I)$ considered observed; Lorentz–polarization corrections, ψ-scan empirical absorption correction (transmission factors 0·921 to 0·952); structure solved by direct-methods techniques, block-cascade least-squares refinement with H atoms refined isotropically; final $R = 0{\cdot}0431$, $wR = 0{\cdot}0503$, 295 parameters refined, $S = 1{\cdot}48$, $(\Delta/\sigma)_{max} = 0{\cdot}23$; largest peaks in the final difference Fourier map of 0·20 and −0·22 e Å^{-3}, $\sum w(|F_o| - |F_c|)^2$ minimized with $w = [\sigma^2(F_o) + 0{\cdot}0017F_o^2]^{-1}$. All computer programs supplied by Nicolet Instrument Corporation (1986) for Desktop 30 Microeclipse and Nova 4/C configurations; atomic scattering factors and anomalous-dispersion corrections from *International Tables for X-ray Crystallography* (1974). Table 1 lists atomic positional

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