$w = 1/[\sigma^2(F_o^2) + (0.0086P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.001$

Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²)*

$U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U^{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$

	x	y	z	$U_{\rm eq}$
I1	0.654070 (8)	0.687482 (7)	−0.006829 (11)	0.02098 (3)
O1	0.75744 (8)	0.90831 (7)	−0.00683 (12)	0.01740 (14)
C1	0.75028 (8)	0.76063 (8)	−0.16952 (15)	0.01362 (15)
C2	0.81343 (9)	0.83352 (10)	−0.0813 (2)	0.0171 (2)

Table 2. *Selected geometric parameters (Å, °)*

I1—C1	2.122 (1)	C1—C1$^{\rm i}$	1.341 (2)
O1—C2	1.418 (2)	C1—C2	1.505 (2)
C1$^{\rm i}$—C1—C2	127.8 (1)	C2—C1—I1	112.1 (1)
C1$^{\rm i}$—C1—I1	120.1 (1)	O1—C2—C1	111.9 (1)
C1$^{\rm i}$—C1—C2—O1	117.9 (2)	I1—C1—C2—O1	−62.2 (1)

Symmetry code: (i) $\frac{3}{2} - x, \frac{3}{2} - y, -\frac{1}{2} - z$.

The asymmetric unit contains half a molecule. All atoms, except for the three H atoms, were refined anisotropically. The two ethylene H atoms were fixed in idealized positions, refining only the C—H distance with $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm C2})$. The hydroxy H atom was localized using the *AFIX* card 147 of *SHELXTL* (Sheldrick, 1994) and $U_{\rm iso}$ was set to $1.2U_{\rm eq}({\rm O1})$ in the subsequent refinement.

Data collection: *SMART* (Siemens, 1995). Cell refinement: *SAINT* (Siemens, 1995). Data reduction: *SAINT*. Program(s) used to solve structure: *SHELXTL*. Program(s) used to refine structure: *SHELXTL*.

The authors are indebted to Hanno Priebe, Oslo, for kindly providing the title compound.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: AB1505). Services for accessing these data are described at the back of the journal.

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Acta Cryst. (1998). C**54**, 557–559

9-(4-Dimethylaminophenyl)-3,3,6,6-tetramethyl-3,4,6,7,9,10-hexahydro-1,8(2*H*,5*H*)-acridinedione

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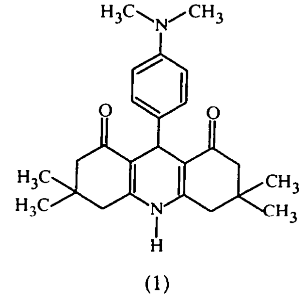
Abstract

In the title compound, $C_{25}H_{32}N_2O_2$, the central ring in the acridine moiety is nearly planar, while the two outer rings adopt half-chair conformations. The phenyl ring is axial with respect to the central pyridine ring.

Comment

Aminoacridinyl derivatives are biologically useful molecules, especially as potential anticancer agents. Acridines have been used as antibacterial agents for wound therapy (Neidle, 1979). Some of them fluoresce well as laser dyes in alcohol solvents (Selladurai *et al.*, 1990).

The bond lengths and angles of the title compound, (1), are similar to those of related acridine derivatives (Selladurai *et al.*, 1989, 1990; Sivaraman *et al.*, 1994, 1996). The O1 atom deviates significantly [−0.256 (2) Å] from the mean plane of ring I (Fig. 1), while the deviation of the O2 atom [−0.036 (5) Å] from the mean plane of ring III is small but significant. Phenyl ring IV is axial with respect to central pyridine ring II. The bonding geometry about each of the two N atoms is planar. Phenyl ring IV is nearly perpendicular to the mean plane of the acridine moiety. The buckling of the



(1)

Acta Crystallographica Section C
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molecule [15.7 (2)°] is large, as is also seen in related acridine derivatives (Clark *et al.*, 1986; Gunasekaran *et al.*, 1996; Hemple *et al.*, 1979).

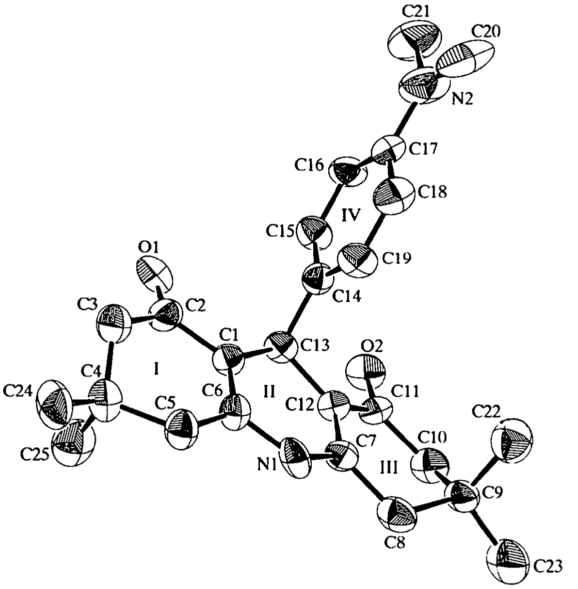

Fig. 1. *ORTEP*II (Johnson, 1976) diagram (50% probability ellipsoids) of the title molecule with the numbering scheme. H atoms have been omitted for clarity.

Experimental

A mixture of dimedone and 4-(dimethylamino)benzaldehyde was refluxed in ethanol in the presence of ammonium hydroxide. The separated solid was filtered, dried and recrystallized from MeOH–$CHCl_3$ (1:2).

Crystal data

$C_{25}H_{32}N_2O_2$
$M_r = 392.53$
Orthorhombic
$Pna2_1$
$a = 14.266\,(2)$ Å
$b = 13.528\,(1)$ Å
$c = 11.747\,(1)$ Å
$V = 2267.1\,(4)$ Å^3
$Z = 4$
$D_x = 1.150$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 24 reflections
$\theta = 9–16°$
$\mu = 0.073$ mm^{-1}
$T = 293\,(2)$ K
Needle
0.36 × 0.19 × 0.14 mm
Pale yellow

Data collection

Siemens $R3m/V$ diffractometer
$\omega/2\theta$ scans
Absorption correction: none
2113 measured reflections
2112 independent reflections
1339 reflections with $I > 2\sigma(I)$

$R_{int} = 0.004$
$\theta_{max} = 25.05°$
$h = 0 \rightarrow 17$
$k = 0 \rightarrow 16$
$l = 0 \rightarrow 13$
2 standard reflections every 200 reflections intensity decay: <1%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.057$
$wR(F^2) = 0.151$
$S = 1.032$
2112 reflections
262 parameters
H atoms riding
$w = 1/[\sigma^2(F_o^2) + (0.0685P)^2 + 0.3592P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.027$

$\Delta\rho_{max} = 0.180$ e Å^{-3}
$\Delta\rho_{min} = -0.213$ e Å^{-3}
Extinction correction: *SHELXL*93 (Sheldrick, 1993)
Extinction coefficient: 0.0058 (16)
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected torsion angles (°)*

C3—C4—C5—C6	−47.2 (7)	C8—C9—C10—C11	50.7 (6)
C13—C1—C6—N1	0.1 (8)	C9—C10—C11—C12	−25.9 (7)
C2—C1—C6—C5	−0.7 (8)	C8—C7—C12—C11	3.6 (8)
C7—N1—C6—C1	−8.8 (8)	N1—C7—C12—C13	6.0 (8)
C4—C5—C6—C1	22.7 (9)	C10—C11—C12—C7	−3.4 (7)
C6—N1—C7—C12	5.7 (8)	C6—C1—C13—C12	9.8 (7)
C12—C7—C8—C9	25.2 (8)	C6—C1—C13—C14	−114.9 (6)
C7—C8—C9—C10	−49.3 (6)	C7—C12—C13—C1	−12.8 (7)

Data collection: *P*3 (Siemens, 1991). Cell refinement: *P*3. Data reduction: *SHELXTL-Plus* (Sheldrick, 1991). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ORTEP*92 (Vickovic, 1994) and *PLUTO* (Motherwell & Clegg, 1978). Software used to prepare material for publication: *SHELXL*93 and *PARST* (Nardelli, 1983).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: VJ1060). Services for accessing these data are described at the back of the journal.

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L-Histidinol Phosphate Tetrahydrate: Redetermination at 110 and 240 K

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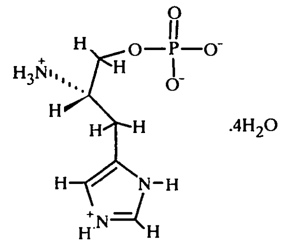
Abstract

In the crystal of L-histidinol phosphate tetrahydrate, $C_6H_{12}N_3O_4P.4H_2O$, at 110 and 240 K, the L-histidinol phosphate residue exists as a zwitterion in which the phosphate group is doubly ionized with the amino and imidazole moieties each accepting a proton from the acid group.

Comment

During the analysis of the geometrical parameters of L-histidinol phosphate tetrahydrate, (I), at ambient temperature (Suga & Okabe, 1997), it was found that the P—O(H) distance does not differ from other P—O(terminal) distances. Since this was rather unexpected (Lis, 1987, and references therein), it was decided to reinvestigate this structure.



(I)

The present studies show that in the crystal of L-histidinol phosphate tetrahydrate at 110 and 240 K, L-histidinol phosphate exists as a zwitterion (Fig. 1) in which the phosphate group is doubly ionized with the amino and imidazole moieties each accepting a proton from the acid group.

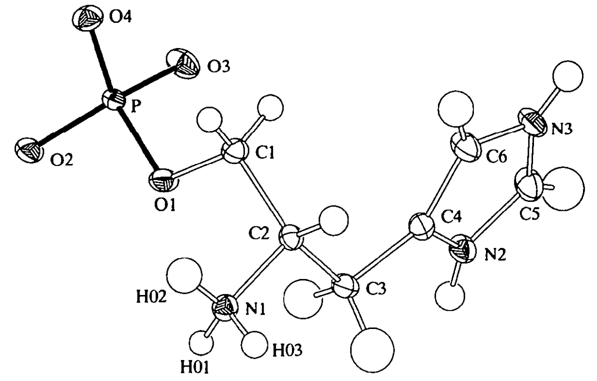


Fig. 1. The structure and numbering scheme of the L-histidinol phosphate residue at 110 K. Displacement ellipsoids are shown at the 50% probability level.

The new atomic coordinates for the heavy atoms do not differ significantly from those of the previous refinement (Suga & Okabe, 1997), although the new s.u.'s are lower. The principal interatomic distances and angles at 110 K do not differ significantly either from those at 240 K or from those of Suga & Okabe (1997). The P—O(terminal) bond lengths may be accepted as normal P—O(terminal) bond lengths in the doubly ionized monoesterified phosphate group (Starynowicz *et al.*, 1986).

The determination of all H-atom positions showed that the hydrogen-bonding pattern postulated by Suga & Okabe (1997) is correct. The crystal structure is stabilized by a three-dimensional network utilizing all O- and N-bonded H atoms. Furthermore, two short C—H···O contacts may be distinguished (Tables 2 and 3).

Experimental

Colourless crystals of the title compound were provided by Suga & Okabe (1997).

(I) at 110 K

Crystal data

$C_6H_{12}N_3O_4P.4H_2O$
$M_r = 293.22$
Monoclinic
$P2_1$
$a = 9.337\,(8)$ Å
$b = 7.597\,(4)$ Å
$c = 9.321\,(6)$ Å
$\beta = 104.46\,(7)°$
$V = 640.2\,(8)$ Å^3
$Z = 2$
$D_x = 1.521$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 44 reflections
$\theta = 8–17°$
$\mu = 0.253$ mm^{-1}
$T = 110\,(1)$ K
Plate
0.40 × 0.30 × 0.18 mm
Colourless