Table 3. *Selected torsion angles* (°) *for* (I)

| | Molecule *A* | Molecule *B* |
|---|---|---|
| D-Mannitol chain | | |
| O(1)—C(1)—C(2)—C(3) | −168·6 (10) | −170·6 (11) |
| C(1)—C(2)—C(3)—C(4) | −151·0 (10) | −149·4 (11) |
| C(2)—C(3)—C(4)—C(5) | −141·6 (10) | −141·3 (10) |
| C(3)—C(4)—C(5)—C(6) | −149·8 (10) | −153·5 (10) |
| C(4)—C(5)—C(6)—O(6) | −169·9 (10) | −165·8 (10) |
| 2,4-Dioxane ring | | |
| C(2)—C(3)—C(4)—O(4) | −25·3 (11) | −25·1 (12) |
| C(3)—C(2)—O(2)—C(7) | 68·1 (11) | 69·6 (11) |
| C(2)—O(2)—C(7)—O(4) | −30·9 (12) | −33·1 (12) |
| O(2)—C(2)—C(3)—C(4) | −34·0 (11) | −34·4 (12) |
| C(3)—C(4)—O(4)—C(7) | 64·4 (11) | 64·9 (11) |
| C(4)—O(4)—C(7)—O(2) | −33·2 (12) | −33·3 (11) |
| 3,5-Dioxane ring | | |
| O(3)—C(3)—C(4)—C(5) | −26·6 (11) | −23·7 (12) |
| C(4)—C(3)—O(3)—C(10) | 66·3 (11) | 64·2 (12) |
| C(3)—O(3)—C(10)—O(5) | −34·3 (12) | −32·6 (12) |
| C(3)—C(4)—C(5)—O(5) | −33·6 (11) | −35·8 (11) |
| C(4)—C(5)—O(5)—C(10) | 69·2 (11) | 71·2 (11) |
| C(5)—O(5)—C(10)—O(3) | −33·6 (12) | −34·6 (12) |

Table 4. *Selected intramolecular distances between* O *atoms and intermolecular distances shorter than* 3·5 Å *between the non*-H *atoms for* (I)

| | | | |
|---|---|---|---|
| Intramolecular distances | | | |
| O(1)···O(2) | 2·926 (6) | O(1*B*)···O(2*B*) | 2·896 (8) |
| O(3)···O(4) | 3·128 (7) | O(3*B*)···O(4*B*) | 3·157 (6) |
| O(5)···O(6) | 2·945 (7) | O(5*B*)···O(6*B*) | 2·973 (7) |
| Intermolecular contacts | | | |
| O(1)···O(1*B*) | 2·758 (8) | O(5)···O(6$B^{\mathrm{ii}}$) | 3·427 (7) |
| O(1)···O(6$B^{\mathrm{i}}$) | 2·699 (5) | O(6)···O(1$B^{\mathrm{iii}}$) | 2·644 (7) |
| O(2)···O(1*B*) | 3·485 (9) | O(6)···O(6$B^{\mathrm{ii}}$) | 2·640 (8) |

Symmetry code: (i) $x, y, z+1$; (ii) $x, y-1, z+1$; (iii) $x, y-1, z$.

While it was evident from IR spectra that in the solution there is an intramolecular hydrogen bond enclosing a five-membered ring (Gawrońska, 1988), several patterns of hydrogen bonding were possible in the crystal. The molecular packing in the crystal lattice is shown in Fig. 2. Table 4 lists the shortest intra- and intermolecular contacts of the non-H atoms. Each of the four hydroxyl groups act as a donor and as an acceptor in two intermolecular, and as a donor in one intramolecular, hydrogen bond. Although the H atoms at the hydroxyl groups could not be located, it appears that the five-membered rings observed in solution are still preserved in the crystal. This is deduced from short O(1)···O(2) and O(5)···O(6) intramolecular distances of 2·9 Å in molecules *A* and *B* – see Table 4. The shortest are the intermolecular hydrogen bonds which link the molecules into sheets along (100) and enclose an eight-membered ring, this pattern often being observed in crystals where hydroxyl groups form the hydrogen bonds linking the molecules [*e.g.* in the pentaerythritol crystal (Eilerman & Rudman, 1979)]. The pattern of the hydrogen bonds in (I) is indicated in Figs. 1 and 2. No other intramolecular contacts shorter than 3·40 Å between non-H atoms are present in this structure.

The author is grateful to Professor J. Gawroński and Dr K. Gawrońska of the Chemistry Department, Adam Mickiewicz University, for suggesting this study and providing the crystals.

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# Structure of Photostable 1-(Hydroxyalkyl)-2(1*H*)-pyrazinones

By Yukie Mori, Atsuko Hayakawa and Koko Maeda*

*Department of Chemistry, Faculty of Science, Ochanomizu University, Otsuka, Bunkyo-ku, Tokyo* 112, *Japan*

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**Abstract.** 1-(2-Hydroxyethyl)-5,6-diphenyl-2(1*H*)-pyrazinone (I), $C_{18}H_{16}N_2O_2$, $M_r = 292.32$, orthorhombic, *Pbca*, $a = 13.320$ (4), $b = 31.654$ (15), $c = 6.991$ (3) Å, $V = 2948$ (2) Å$^3$, $Z = 8$, $D_x = 1.317$ g cm$^{-3}$, $\lambda$(Mo $K\alpha$) = 0.71068 Å, $\mu = 0.81$ cm$^{-1}$, $F(000) = 1232$, $T = 294$ K, $R = 0.057$ for

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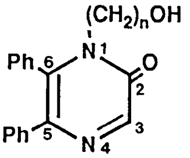
1735 observed reflections. 1-(3-Hydroxypropyl)-5,6-diphenyl-2(1*H*)-pyrazinone (II), $C_{19}H_{18}N_2O_2$, $M_r = 306.35$, triclinic, $P\bar{1}$, $a = 9.939$ (2), $b = 10.043$ (2), $c = 9.603$ (3) Å, $\alpha = 114.19$ (2), $\beta = 96.89$ (2), $\gamma = 106.40$ (2)°, $V = 807.9$ (4) Å$^3$, $Z = 2$, $D_x = 1.259$ g cm$^{-3}$, $\lambda$(Mo $K\alpha$) = 0.71068 Å, $\mu = 0.77$ cm$^{-1}$, $F(000) = 324$, $T = 294$ K, $R = 0.054$ for 2619 observed reflections. In both crystals, neighboring pyrazine rings lie apart from each other, which accounts for the observation that these pyrazinones do not undergo solid-state photodimerization. Crystals of (I) show a characteristic dimeric structure in which two intermolecular hydrogen bonds between the hydroxyl and the carbonyl groups connect two molecules related by an inversion center, while in (II) one intermolecular hydrogen bond between the hydroxyl group and N(4) of the neighbor constructs a chain-like structure along the *a* axis.

**Introduction.** 1-Methyl-5,6-diphenyl-2(1*H*)-pyrazinone was reported to undergo photochemical [4 + 4] cyclodimerization in the solid state (Nishio, Nakajima & Omote, 1980) and the crystal structure shows that two neighboring pyrazinone rings are arranged in a nearly parallel fashion at a distance of 3.5 Å (Kaftory, 1984). During the course of synthetic and photochemical studies for other pyrazinone derivatives, 1-(2-hydroxyethyl)- (I) and 1-(3-hydroxypropyl)-5,6-diphenyl-2(1*H*)-pyrazinone (II) were obtained.



(I) $n = 2$
(II) $n = 3$

The photochemical behaviors of these pyrazinones were investigated in deaerated solutions and in the solid state, but in (I) and (II) neither [4+4] dimerization nor other photochemical reactions occurred. Both (I) and (II), having a hydroxyl group at the end of *N*-alkyl substituent, may form intra- or intermolecular hydrogen bonds and the hydrogen-bonding pattern is considered as an important factor determining molecular packing in the crystal. In connection with our interest in relationship between crystal structure and solid-state photoreactivity the crystal structures of (I) and (II) were determined.

**Experimental.** (I) was synthesized from 5,6-diphenyl-2(1*H*)-pyrazinone and 1,2-dibromoethane in $Me_2SO$ in the presence of $K_2CO_3$ (m.p. 427–428 K). (II) was synthesized by a similar procedure using 1,3-dibromopropane instead of 1,2-dibromoethane (m.p. 364–365 K). Solid-state photolysis of (I) and (II) was carried out in KBr pellets (*ca* 2% *w*/*w*) with a xenon or a high-pressure mercury lamp, but after 10 h of irradiation no photoproducts were observed in TLC or NMR spectra and the starting pyrazinone was recovered.

(I): Colorless column-like crystal obtained from a benzene solution, crystal dimensions 0.5 × 0.2 × 0.1 mm, Rigaku AFC-5 diffractometer, graphite-monochromated Mo $K\alpha$ radiation; cell parameters were refined by least squares on the basis of 16 independent $2\theta$ values ($20 < 2\theta < 26°$); intensity data were collected up to $2\theta = 55°$ ($h\ 0 \rightarrow 17$, $k\ 0 \rightarrow 41$, $l\ 0 \rightarrow 9$) using the $\omega$–$2\theta$ scan technique; scan speed 4° min$^{-1}$ in $\theta$, scan width $(1.0 + 0.35\tan\theta)°$, stationary background counts were accumulated 5 s before and after each scan, three standard reflections (0,18,0, 800, 174) were monitored every 50 reflections, no significant variation in intensities; 3359 unique reflections were measured, 1737 with $|F_o| > 3\sigma(|F_o|)$ were considered as observed and used for structure determination; corrections for Lorentz and polarization effects were made, absorption was ignored; direct methods (*MULTAN*78; Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978) and subsequent difference Fourier map calculations, full-matrix least squares (*SHELX*76; Sheldrick, 1976) with anisotropic thermal parameters for non-H atoms and isotropic ones for H atoms, all H atoms were located on difference Fourier maps, intense reflections with low $2\theta$ angle (040 and 021) seemed to suffer from extinction and were not used in the refinements; $\sum w(|F_o| - |F_c|)^2$ minimized, $w = [\sigma^2(|F_o|) + 0.00032|F_o|^2]^{-1}$; final $R = 0.057$ and $wR = 0.054$ for 1735 observed reflections and 263 refined parameters; $S = 1.49$; $(\Delta/\sigma)_{\max} = 0.05$; final difference map showed $-0.22 < \Delta\rho < 0.21$ e Å$^{-3}$; atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV); calculations were carried out on an IBM 4381-R24 computer at this university.

(II): Data collection and structure determination were carried out by similar methods to those for (I); colorless plate-like crystal obtained from a benzene solution, crystal dimensions 0.55 × 0.3 × 0.2 mm; cell parameters were refined by least squares on the basis of 18 independent $2\theta$ values ($24 < 2\theta < 29°$); $2\theta_{\max} = 55°$ ($h\ -13 \rightarrow 13$, $k\ -13 \rightarrow 13$, $l\ 0 \rightarrow 12$); three standard reflections ($\bar{1}\bar{5}6$, $5\bar{1}3$, $\bar{3}60$) were monitored; 3896 reflections were measured, of which 3680 were unique, $R_{\text{int}} = 0.017$, 2619 with $|F_o| > 3\sigma(|F_o|)$ were considered as observed and used for structure determination; some H atoms were located on the difference Fourier map and other H-atom positions were calculated geometrically and then refined; $w = [\sigma^2(|F_o|) + 0.0021|F_o|^2]^{-1}$; final $R =$

Table 1. *Final atomic coordinates* ($\times 10^4$) *with their e.s.d.'s and equivalent isotropic temperature factors for non-*H *atoms of* (I) *and* (II)

$$B_{\rm eq} = (4/3)\sum_i\sum_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$$

| | $x$ | $y$ | $z$ | $B_{\rm eq}$(Å$^2$) |
|---|---|---|---|---|
| (I) | | | | |
| N(1) | 9217 (2) | 628 (1) | 6641 (3) | 3.3 |
| C(2) | 10143 (3) | 442 (1) | 6310 (5) | 3.8 |
| C(3) | 10746 (3) | 649 (1) | 4906 (5) | 3.9 |
| N(4) | 10501 (2) | 991 (1) | 4017 (4) | 3.7 |
| C(5) | 9587 (2) | 1177 (1) | 4435 (4) | 3.0 |
| C(6) | 8947 (2) | 997 (1) | 5716 (4) | 2.9 |
| O(7) | 10400 (2) | 122 (1) | 7205 (4) | 5.5 |
| C(8) | 9419 (2) | 1588 (1) | 3412 (4) | 3.3 |
| C(9) | 8490 (3) | 1739 (1) | 2814 (5) | 3.9 |
| C(10) | 8405 (3) | 2129 (1) | 1919 (5) | 4.6 |
| C(11) | 9235 (3) | 2368 (1) | 1557 (5) | 4.5 |
| C(12) | 10164 (3) | 2219 (1) | 2083 (5) | 4.5 |
| C(13) | 10256 (3) | 1836 (1) | 3010 (5) | 3.9 |
| C(14) | 7951 (2) | 1186 (1) | 6207 (4) | 2.9 |
| C(15) | 7069 (3) | 1037 (1) | 5397 (5) | 3.7 |
| C(16) | 6174 (3) | 1243 (1) | 5736 (6) | 4.2 |
| C(17) | 6148 (3) | 1595 (1) | 6869 (6) | 4.7 |
| C(18) | 7012 (3) | 1744 (1) | 7739 (5) | 4.2 |
| C(19) | 7910 (3) | 1537 (1) | 7388 (5) | 3.5 |
| C(20) | 8555 (3) | 423 (1) | 8084 (5) | 3.9 |
| C(21) | 8741 (3) | 594 (1) | 10067 (5) | 4.5 |
| O(22) | 8119 (2) | 378 (1) | 11412 (4) | 5.5 |
| (II) | | | | |
| N(1) | 2703 (2) | 1496 (2) | 4187 (2) | 3.2 |
| C(2) | 2388 (2) | 2742 (3) | 5229 (2) | 3.7 |
| C(3) | 862 (2) | 2499 (3) | 4919 (3) | 4.4 |
| N(4) | −162 (2) | 1284 (2) | 3756 (2) | 4.1 |
| C(5) | 207 (2) | 123 (2) | 2695 (2) | 3.2 |
| C(6) | 1634 (2) | 212 (2) | 2904 (2) | 3.0 |
| O(7) | 3342 (2) | 3912 (2) | 6323 (2) | 5.0 |
| C(8) | −1035 (2) | −1196 (2) | 1362 (3) | 3.4 |
| C(9) | −956 (3) | −1875 (3) | −202 (3) | 3.8 |
| C(10) | −2171 (3) | −3066 (3) | −1399 (3) | 4.6 |
| C(11) | −3466 (3) | −3592 (3) | −1055 (4) | 5.2 |
| C(12) | −3549 (3) | −2931 (3) | 483 (4) | 5.2 |
| C(13) | −2351 (2) | −1738 (3) | 1690 (3) | 4.2 |
| C(14) | 2072 (2) | −1019 (2) | 1776 (2) | 3.2 |
| C(15) | 1622 (3) | −2513 (3) | 1651 (3) | 4.4 |
| C(16) | 1933 (3) | −3684 (3) | 494 (4) | 6.0 |
| C(17) | 2662 (3) | −3376 (4) | −526 (4) | 6.7 |
| C(18) | 3110 (3) | −1906 (4) | −404 (3) | 5.8 |
| C(19) | 2821 (3) | −720 (3) | 738 (3) | 4.2 |
| C(20) | 4226 (2) | 1582 (3) | 4597 (3) | 3.5 |
| C(21) | 5273 (2) | 2675 (3) | 4165 (3) | 4.0 |
| C(22) | 6822 (3) | 2809 (3) | 4691 (3) | 4.6 |
| O(23) | 6891 (2) | 1309 (3) | 4009 (3) | 7.4 |

Table 2. *Selected geometrical parameters* (Å, °) *for* (I) *and* (II)

| | (I) | (II) |
|---|---|---|
| N(1)—C(2) | 1.386 (4) | 1.391 (3) |
| N(1)—C(6) | 1.382 (4) | 1.385 (3) |
| N(1)—C(20) | 1.489 (5) | 1.486 (3) |
| C(2)—C(3) | 1.427 (5) | 1.440 (4) |
| C(2)—O(7) | 1.239 (4) | 1.226 (3) |
| C(3)—N(4) | 1.293 (4) | 1.297 (4) |
| N(4)—C(5) | 1.384 (4) | 1.375 (3) |
| C(5)—C(6) | 1.363 (4) | 1.380 (3) |
| C(5)—C(8) | 1.499 (4) | 1.491 (3) |
| C(6)—C(14) | 1.496 (4) | 1.487 (3) |
| C(20)—C(21) | 1.509 (6) | 1.514 (4) |
| C(21)—C(22) | | 1.511 (5) |
| C(21)—O(22) | 1.427 (5) | |
| C(22)—O(23) | | 1.401 (5) |
| | | |
| C(2)—N(1)—C(6) | 120.8 (3) | 121.6 (2) |
| C(2)—N(1)—C(20) | 117.0 (3) | 115.9 (2) |
| C(6)—N(1)—C(20) | 122.1 (3) | 122.4 (2) |
| N(1)—C(2)—C(3) | 114.9 (3) | 113.8 (2) |
| N(1)—C(2)—O(7) | 120.6 (3) | 121.8 (2) |
| C(3)—C(2)—O(7) | 124.5 (3) | 124.3 (2) |
| C(2)—C(3)—N(4) | 124.9 (3) | 125.4 (3) |
| C(3)—N(4)—C(5) | 118.6 (3) | 118.9 (2) |
| N(4)—C(5)—C(6) | 120.7 (3) | 120.6 (2) |
| N(4)—C(5)—C(8) | 113.5 (3) | 115.0 (2) |
| C(6)—C(5)—C(8) | 125.7 (3) | 124.3 (2) |
| N(1)—C(6)—C(5) | 119.9 (3) | 119.4 (2) |
| N(1)—C(6)—C(14) | 117.5 (3) | 118.5 (2) |
| C(5)—C(6)—C(14) | 122.5 (3) | 122.0 (2) |
| N(1)—C(20)—C(21) | 111.7 (3) | 122.9 (2) |
| C(20)—C(21)—C(22) | | 111.4 (3) |
| C(20)—C(21)—O(22) | 109.8 (3) | |
| C(21)—C(22)—O(23) | | 109.0 (3) |
| | | |
| Pyrazine and 5-Ph | 32.2 (1) | 42.0 (1) |
| Pyrazine and 6-Ph | 78.8 (1) | 69.3 (1) |
| | | |
| C(2)—N(1)—C(20)—C(21) | 89.4 (4) | −78·6 (3) |
| N(1)—C(20)—C(21)—O(22) | −178.0 (3) | |
| N(1)—C(20)—C(21)—C(22) | | 176.5 (2) |
| C(20)—C(21)—C(22)—O(23) | | 57.0 (4) |
| | | |
| Hydrogen-bond parameters | | |
| O(22)—O(7′)$^{\rm i}$ | 2.709 (4) | |
| H(22)—O(7′)$^{\rm i}$ | 1.80 (4) | |
| O(23)—N(4′)$^{\rm ii}$ | | 2.975 (4) |
| H(23)—N(4′)$^{\rm ii}$ | | 2.05 (4) |
| O—H$\cdots X$ | 161 (4) | 166 (4) |

Symmetry code: (i) $2 - x$, $-y$, $2 - z$; (ii) $x + 1$, $y$, $z$.

0.054 and $wR = 0.070$ for 2619 observed reflections and 280 refined parameters; $S = 1.50$; $(\Delta/\sigma)_{\max} = 0.06$; $-0.25 < \Delta\rho < 0.19$ e Å$^{-3}$.

**Discussion.** Final atomic coordinates for (I) and (II) are listed in Table 1, and selected structural parameters are summarized in Table 2.* The molecular structures with atomic numbering are shown in Fig. 1. The molecules of (I) and (II) have similar structures and no significant differences are observed between bond distances and angles for (I) and (II). In both molecules the pyrazine ring is almost planar with a maximum deviation of 0.017 Å for (I) or 0.026 Å for (II). The sum of the three bond angles around N(1) is 360°, indicating that N(1) is of planar $sp^2$ character rather than pyramidal $sp^3$. The phenyl rings rotate from the pyrazine ring; in particular, the large dihedral angle between 6-phenyl and pyrazine planes (Table 2) seems due to steric repulsion with the alkyl substituent at the 1-position. These structural features for (I) and (II) are similar to those reported for 1-methyl and 1-ethyl derivatives (Kaftory, 1984). With respect to the conformation of the hydroxyalkyl groups, N(1)—C(20)—C(21)—O(22) in (I) and N(1)—C(20)—C(21)—C(22) in (II) are antiperiplanar, while the C(20)—C(21)—C(22)—O(23) moiety in (II) adopts a *gauche* conformation. This unexpected conformation of the hydroxypropyl

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* Lists of structure factors, anisotropic thermal parameters, full bond lengths and angles including those involving H atoms, least-squares planes, and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54415 (25 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AS0499]

group in (II) may result from the formation of intermolecular hydrogen bond (see below).

Solid-state cyclodimerization reactions have been considered favorable when the reacting double bonds are arranged in a parallel fashion with the distance between the reacting centers about 4 Å (Schmidt, 1971). As is seen in Fig. 2, in (I) and (II) the molecules are not packed in such a parallel fashion and the distances between the possible reacting centers, *i.e.* C(3) and C(6), are too long to undergo [4 + 4] cyclodimerization. Thus, the photostability of the crystals of (I) and (II) can be explained in terms of their molecular packings.

In (I) and (II) intermolecular hydrogen bonding is observed, but, interestingly, the hydrogen-bonding schemes are different. In the case of (I) the OH group forms an O(22)—H(22)···O(7) type hydrogen bond with the carbonyl O atom of the molecule which is related by the inversion center. The two molecules of (I) which are related to each other by the inversion center form a dimeric structure containing a 14-membered ring by two hydrogen bonds, O(22)—H(22)···O(7′) and O(7)···H(22′)—O(22′). Hydrogen-bond parameters are also listed in Table 2. On the other hand, in the crystal of (II) the OH group forms an O—H···N type hydrogen bond with N(4) of the translationally related molecule. As a result of this type of hydrogen bonding, the molecules of (II) are connected along the *a* axis to form an antiparallel chain-like structure.

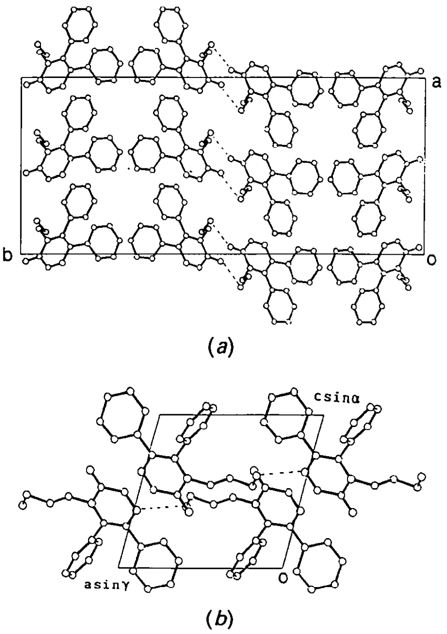

Fig. 2. Crystal structures of (I) and (II): (*a*) viewed along the *c* axis for (I), (*b*) viewed along the *b* axis for (II).

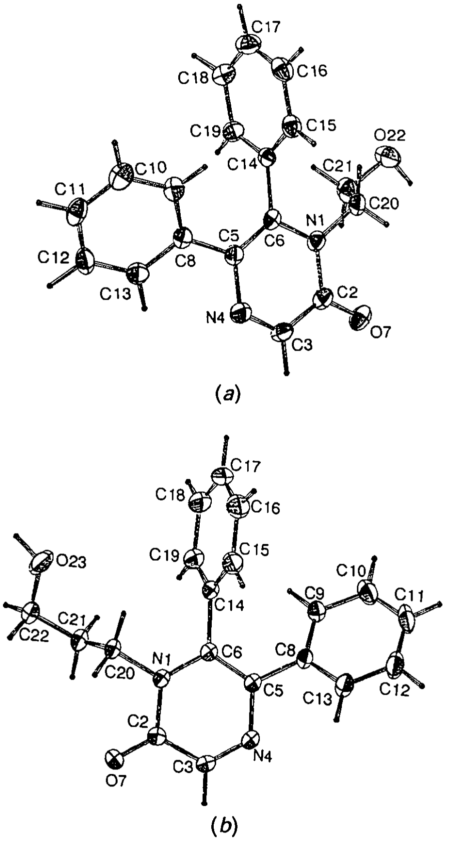

Fig. 1. *ORTEP* (Johnson, 1965) plots of (*a*) (I) and (*b*) (II) with atomic numbering. The anisotropic ellipsoids for non-H atoms enclose 30% probability.

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