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Structure of *p*-Aminobenzoic Acid-1,3-Dimethyl-2-imidazolidinone (1/1)

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Abstract. C₇H₇NO₂·C₅H₁₀N₂O, $M_r = 251.3$, monoclinic, $P2_1/c$, $a = 7.965$ (2), $b = 12.559$ (3), $c = 14.250$ (2) Å, $\beta = 115.01$ (2)°, $V = 1291.8$ (5) Å³, $Z = 4$, $D_x = 1.29$ Mg m⁻³, $\text{Mo } K\alpha$, $\lambda = 0.71070$ Å, $\mu = 0.09$ mm⁻¹, $F(000) = 536$, $T = 293$ K, final $R = 0.077$ for 1665 unique reflections. The complex is formed by hydrogen bonding between the carbonyl group of 1,3-dimethyl-2-imidazolidinone and the hydroxy group of *p*-aminobenzoic acid. The units are packed together with hydrogen bonding between amino groups and carbonyl groups of the *p*-aminobenzoic acid moieties. Both constituents of the complex are planar with normal bond lengths and angles.

Introduction. 1,3-Dimethyl-2-imidazolidinone (DMI) is an interesting compound in the pharmaceutical field. It is known to have low toxicity (Lien & Kumler, 1968) and also does not bring about any primary irritation with redness, itching and scaling in contact with rabbit skin (Ueda, 1976). Recently, DMI has been studied as an absorption promoter on percutaneous absorption (Sato, Kobayashi, Nishiu & Tamada, 1983). Therefore, it is important to elucidate the interaction of DMI with

a drug. The X-ray crystal structure analysis of the complex of DMI and *p*-aminobenzoic acid (PABA) has been undertaken to reveal the interaction between the two components.

Experimental. To a solution of 1 g *p*-aminobenzoic acid in 200 ml ethyl ether, 1 ml of 1,3-dimethyl-2-imidazolidinone was added slowly with stirring at 303 K. Solution was kept standing for at least 48 h at room temperature in the dark. Crystals were filtered out, washed with cold ethyl ether and dried under vacuum. Crystal is transparent and colorless column, m.p. 380 K. Analysis: calc. for C₁₂H₁₇N₃O₃: C, 57.11; H, 6.94; N, 16.75%; found: C, 57.37; H, 6.82; N, 16.73%. Electron-impact mass spectrometry: m/z 114 (M^+), 137 (M^+). Single crystal 0.6 × 0.3 × 0.3 mm. Rigaku AFC-5FOS four-circle diffractometer, graphite-monochromated Mo $K\alpha$ radiation. Cell parameters refined by least squares for 20 reflections with $7.25 < \theta < 11.23^\circ$. No corrections for absorption. 3184 independent reflections measured, 2θ 0-54° without $F(000)$; range of hkl : h 0-10, k 0-16, l -18-16; $\omega/2\theta$ scan mode; 1666 reflections with $F \geq 3\sigma(F)$. Linear

Table 1. Fractional atomic coordinates ($\times 10^4$) and equivalent isotropic thermal parameters B_{eq} of non-hydrogen atoms

$$B_{eq} = (4/3) \sum_i \sum_j \beta_{ij} a_i \cdot a_j$$

	x	y	z	$B_{eq}(\text{\AA}^2)$
C(1)	7628 (6)	343 (3)	2727 (3)	2.9
C(2)	7344 (6)	1284 (3)	3241 (3)	3.7
C(3)	6271 (7)	2121 (4)	2662 (3)	3.7
C(4)	5926 (7)	3004 (4)	3118 (3)	3.4
C(5)	6672 (6)	3082 (4)	4203 (3)	3.3
C(6)	7751 (6)	2242 (4)	4798 (3)	3.2
C(7)	8072 (6)	1366 (3)	4324 (3)	3.2
O(1)	8602 (5)	-422 (2)	3341 (2)	4.3
O(2)	6972 (5)	257 (3)	1779 (2)	4.4
N	6377 (6)	3975 (3)	4652 (3)	5.0
O	9248 (5)	-2207 (3)	2630 (3)	5.3
CA	8410 (7)	-2855 (4)	1936 (3)	3.9
N(1)	7658 (6)	-2663 (3)	912 (3)	4.6
CB	6915 (8)	-3614 (5)	317 (4)	5.2
CC	6938 (7)	-4393 (4)	1125 (4)	4.9
N(2)	8133 (6)	-3883 (3)	2090 (3)	4.6
CD	8056 (9)	-1728 (5)	462 (5)	6.0
CE	8397 (9)	-4315 (5)	3070 (5)	6.2

decay (ca 12%) of F values of standard reflections throughout data collection, correction applied. Structure solved by direct methods (*MULTAN70*; Germain, Main & Woolfson, 1970) and refined by block-diagonal least squares; H-atom positions determined from difference map; anisotropic temperature factors for non-hydrogen atoms and isotropic for H atoms refined; $R = 0.0768$, $wR = 0.0768$, $w = 1$, $(\Delta/\sigma)_{\max} = 0.87$ (0.48 for non-H atoms), $S = 0.281$; largest peak in final ΔF map = 0.41 e \AA^{-3} . * Atomic scattering factors from *International Tables for X-ray Crystallography* (1974). R value did not decrease below 0.07 mainly due to considerable radiation decay.

Discussion. The basic structural unit consists of one pair of DMI and PABA, forming an asymmetric unit. Atomic coordinates and equivalent isotropic temperature factors B_{eq} for non-hydrogen atoms are listed in Table 1. The atom numbering scheme and selected bond lengths and angles are shown in Fig. 1. The mean standard deviations of the bond lengths and angles are estimated as: C—C, C—O, N—C = 0.011 \AA , C—C—O, O—C—O, C—C—C, C—C—N, O—C—N, N—C—N, C—N—C = 0.6° , C—H, O—H, N—H = 0.053 \AA , C—C—H, C—O—H, C—N—H, N—C—H, H—N—H, H—C—H = 3.6° . A projection of the structure along b is shown in Fig. 2. The complex owes its formation mainly to a hydrogen bond between the carbonyl group of DMI and the hydroxy group of PABA. In addition,

* Lists of structure factors, anisotropic temperature factors, H-atom parameters, bond lengths and bond angles and least-squares planes have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42626 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

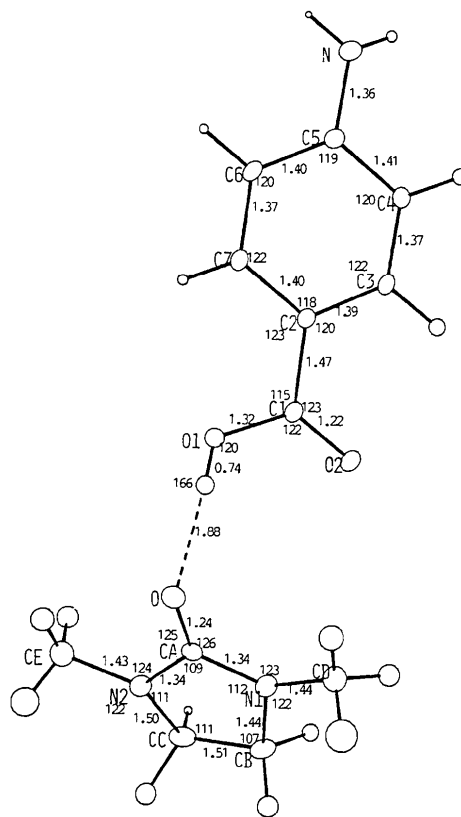


Fig. 1. Selected bond lengths (\AA) and angles ($^\circ$). The thermal ellipsoids of all atoms show 10% probability distribution.

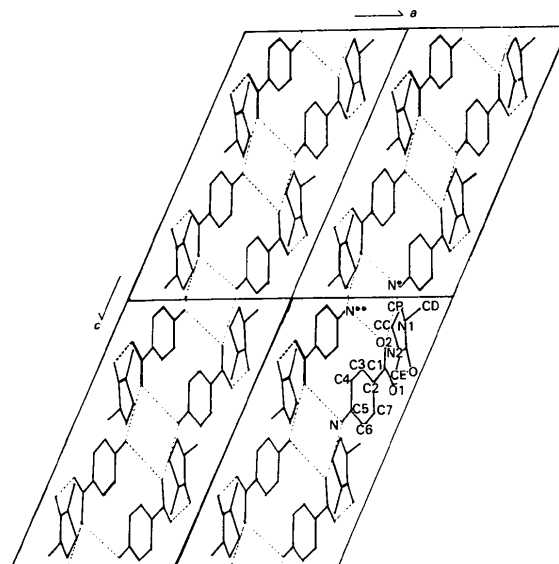


Fig. 2. Projection of the structure of DMI-PABA along b . Hydrogen bonds are illustrated by broken lines. N^* and N^{**} are at $(x, \frac{1}{2} - y, -\frac{1}{2} + z)$ and $(1 - x, -\frac{1}{2} + y, \frac{1}{2} - z)$, respectively.

Table 2. *Hydrogen-bond distances (Å) with standard deviations in parentheses*

O(1)···O	2.60 (1)	H(O1)···O	1.88 (5)
O(2)···N ⁱ	3.02 (1)	O(2)···H(N ^A)	2.25 (4)
O(2)···N ⁱⁱ	3.04 (4)	O(2)···H(N ^B)	2.28 (5)

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

amino groups and carbonyl groups of PABA are hydrogen bonded head-to-tail. Hydrogen-bond distances are listed in Table 2. These show normal values (Pauling, 1960; Sutton, 1958). Other intermolecular distances are quite normal. The shortest intermolecular distance is 3.411 (9) Å for C(1)···N(2)($2 - x, \frac{1}{2} + y, \frac{1}{2} - z$), which is not a hydrogen bond. The interaction mode and the reason why PABA and DMI form a one-to-one complex can be well understood. These results should be useful for understanding the properties of DMI-drug complexes. The least-squares plane of C(2), C(3), C(4), C(5), C(6) and C(7) was calculated. The deviations of the other non-hydrogen atoms of PABA from the plane are 0.034 (8) to 0.087 (9) Å. For DMI, high planarity is also observed. The deviations of CD and CE from the least-squares plane of CA, N(1),

CB, CC and N(2) are 0.180 (11) and 0.165 (11) Å, respectively (Ito, 1982). The sum of the three bond angles around N(1) is 357.2 (18)° and that around N(2) is 356.5 (18)°. Thus, the arrangements of bonds around N(1) and N(2) are quasi-planar.

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(6R*,7S*,10R*,11S*)-6,6a,7,8,9,10,10a,11-Octahydro-6,11-dimethyl-12-phenyl-6,11-imino-7,10-methano-5H-dibenzo[*b,e*]azepine

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Abstract. C₂₃H₂₆N₂, *M_r* = 330.48, triclinic, *P* $\bar{1}$, *a* = 11.276 (4), *b* = 11.510 (4), *c* = 8.369 (4) Å, α = 113.40 (3), β = 99.14 (3), γ = 108.94 (3)°, *V* = 889.6 (6) Å³, *Z* = 2, *D_m* = 1.230, *D_x* = 1.234 g cm⁻³, $\lambda(\text{Cu K}\alpha)$ = 1.54178 Å, μ = 5.57 cm⁻¹, *F*(000) = 356, room temperature (295 K), *R* = 0.040 for 2099 independent observed reflections. The molecule can be described as composed of a seven-membered ring fused to a six-membered saturated ring and a phenyl ring. The six-membered ring is bridged by a methylene group while the seven-membered ring is bridged by an *N*-phenyl nitrogen. The seven-membered ring can be described in terms of a planar moiety folded along an axis through the bridged atoms forming a dihedral angle of 108.4 (3)°. There are a number of intramolecular

H···H contacts less than 2.50 Å, and molecular-mechanics calculations are used to analyze these interactions.

Introduction. The pyramidalization of the C(*sp*²) carbon atoms in *syn*-sesquinorbornenes is well documented (Watson, 1983), and compound (1) with a 22.1 (2)° fold along the C=C bond represents the largest such deviation reported (Watson, Galloy, Grossie, Bartlett & Combs, 1984). It is of interest to prepare the nitrogen analogue of this compound, and synthetic routes involving pyrroles have been investigated. 1,4-Diketones are known to undergo condensation with aniline, in the presence of acids, to give the corresponding *N*-phenyl pyrroles (Wynberg & Klunder, 1969). When 2,3-diacetylnorbornane was treated with aniline in refluxing ethanol for 3.0 d,

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