The Identification and Crystal Structure of Imenine, a New Ketoaporphine Alkaloid*

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The structure of imenine, an alkaloid isolated from *Abuta imene*, has been solved by iterative application of the Sayre relationship to three-dimensional X-ray diffraction data. Imenine crystallizes in space group PI with unit-cell dimensions a=9.087 (9), b=8.359 (9), c=11.660 (12) Å, $\alpha=111.68$ (1), $\beta=88.59$ (1), $\gamma=101.17$ (2)°, $\varrho_{calc}=1.453$ for Z=2, $\varrho_{exp}=1.458$ g.cm⁻³. The alkaloid is shown to be C₂₀H₁₇NO₅, 1,2,3,4-tetramethoxydibenzo[*de,g*]quinazolin-7-one, the first ketoaporphine to have substitution in the heterocyclic ring.

Introduction

The aporphines comprise a group of over 90 alkaloids whose basic ring system is the 4H-dibenzo[de,g]quinoline structure. In 1960 a subgroup of the aporphines was discovered which contained an oxygen bonded to the 7 position and was called 'keto' or 'oxo' aporphine. Since that time 12 naturally occurring members of this subgroup have been found whose members are distinguished by their oxygenation patterns. This area is discussed thoroughly by Cava & Venkateswarlu (1968).

The thirteenth member of this subgroup has been isolated by Cava & co-workers (Glick, Cook, Cava, Srivanasan, Kunitomo & da Rocha, 1969) from the Brazilian vine *Abuta imene* (Menispermaceae). The vine was chosen for study because several related species were known to be rich in alkaloids, and it was hoped that *Abuta imene* would contain new, unknown alkaloids.

Extractions made from the vine did produce several highly colored alkaloids, the most abundant of which was imenine. Unsuccessful characterization of the structure of imenine was attempted by nuclear magnetic resonance, infrared and ultraviolet spectroscopy, mass spectroscopy and chemical methods several months before crystallographic methods were employed. The above studies indicated the compound was aromatic, probably contained nitrogen-methyl groups and methoxy groups, and had a molecular weight of about 533.

Experimental

A large orange-yellow crystal of imenine was selected from those provided by Drs Cava and Srinivasan and cut to a final size of $0.34 \times 0.19 \times 0.19$ mm. Silastic medical adhesive was used to hold the crystal to a pulled glass fiber. Following preliminary film work the crystal was mounted on a Picker four-angle automatic diffractometer. After the crystal was centered with X-rays, 17 reflections were maximized for Mo $K\alpha_1$ radiation using narrow rear slits and used to calculate the triclinic lattice constants* a=9.087(9), b=8.359(9), c=11.660(12) Å, $\alpha=111.68(1), \beta=88.59(1), \gamma=101.17$ (2)°. The experimental density of 1.454 (8) g.cm⁻³ as measured by flotation was in excellent agreement with the final calculated density of 1.453 g.cm⁻³ based on two molecules of formula C₂₀H₁₇NO₅ per unit cell.

The diffraction data were collected automatically using Mo $K\alpha_1$ radiation filtered through 2.8 mils of Zr foil with the pulse height analyser set to accept 90% of the incoming radiation. The moving-counter moving-crystal scan technique was used with a drive speed of 1° per minute and a take off angle of 2.0°. The intensities were obtained by a continuous scan of 1.6° (0.75° before and 0.85° after the maximum) through the peak (DATA) with stationary backgrounds of 15 seconds at both ends of the scan (BK1 and BK2). The reflection data were collected in the hemisphere $+h, \pm k, \pm l$ out to a 2 θ of 45°.

A total of 2160 reflections were measured of which 1554 had net intensities above 2 standard deviations. Five standard reflections, which were checked after every one hundred data, indicated no systematic change in intensity.

* The programs used in the solving of this structure include local versions of the following programs:

^{*} A preliminary account of the structure analysis was presented at the Eighth International Congress of Crystallography, Buffalo, New York, August 1969.

[†] Based in part on a thesis submitted to the Graduate School of Wayne State University in partial fulfillment of the requirements for the degree of Doctor of Philosophy,

PICK2 – J. A. Ibers' least-squares program for lattice constants and orientation angles based on W. C. Hamilton's *MODE1*.

DACOR – a program for Lp corrections and calculation of intensity standard deviations and normalized structure factors. FORDAP – A. Zalkin's Fourier program.

ORFLS and ORFFE – W. Busing, K. Martin & H. Levy's full-matrix least-squares program and function and error program.

ORTEP – C. K. Johnson's program for drawing thermal ellipsoids of crystal structures.

REL – A program for Phase Determination by Reiterative Application for Sayre's Equation, R. E. Long, Ph. D. Thesis (part III), University of California, Los Angeles, 1965,

The raw data were corrected for Lorentz and polarization factors and background. Standard deviations for the intensities were calculated using the formula $[(DATA + 3.2 BK1 + 3.2 BK2 - 0.03^{2}(DATA - 3.2 BK1$ -3.2 BK2)²]^{1/2} (Corfield, Doedens & Ibers, 1967). Absorption of the crystal was not taken into account.

Solution to structure

Mass spectroscopic results, unit-cell data, and the distribution of normalized structure factors (22.1% of E values >1.0, 5.2% > 2.0, 1.7% > 3.0) pointed toward one molecule of molecular weight 702 in a

Table 1. Comparison of observed and calculated structure factors (×10) for imenine

Table 1 (cont.)

l 91 79 2 41 49 3 97 104 4 442 460 5 73 75 6 34 48 7 21 27 H= 5,K= 3 L FO FC	-1 25 28 0 69 58 H 6.K8 L F0 FC 2 26 27 6 32 16 H 6.K -7 L F0 FC	H= 6,K= -3 L FO FC -6 22 18 -5 59 56 -4 63 55 -2 32 35 -1 76 73 D 77 78 2 67 85 4 53 53	-5 52 53 -4 35 36 -3 41 41 -2 62 62 -1 70 70 0 40 34 1 53 51 3 122 123 4 295 299 5 34 36	-2 45 48 -1 46 41 0 72 67 2 58 55 3 36 29 4 32 34 H= 6,K= 4 L FO FC -7 26 13	$\begin{array}{cccccccccccccccccccccccccccccccccccc$	6 58 67 7 26 30 8 33 27 H= 7,K= 0 L FO FC -7 90 95 -6 160 160 -5 75 75 -4 26 28	-2 60 63 -1 132 146 1 27 36 4 30 23 HW 7,K- 4 L FO FC -6 23 19 -5 41 34 -4 42 32	1 27 26 2 23 31 3 223 214 4 262 246 5 21 37 6 21 1 H+ 8.K* -1 L FO FC -6 197 201 -5 87 86	He 9,K= -5 L FO FC 3 23 17 He 9,K= -4 L FO FC 3 167 151 4 72 60
-8 63 64 -6 244 251 -5 332 343 -4 110 108 -3 22 14 -1 120 112 1 64 59 2 80 93	0 24 27 4 72 69 5 32 25 7 25 15 Here 6,X= -6 L FO FC -2 24 24	5 38 41 7 37 35 8 47 57 9 151 152 H= 6+K= -2 L FO FC -7 44 47	7 29 28 H= 6,K= 1 L FO FC -9 29 24 -8 29 31 -6 182 196 -5 93 101	-6 35 22 -4 35 41 -2 80 81 -1 34 36 0 52 48 2 22 24 3 35 39	6 30 32 8 43 52 H= 7,K= -3 L FD FC -4 42 41 -2 76 86 -1 175 182	-3 37 28 -1 44 43 0 21 28 1 65 68 3 163 173 4 323 337 5 65 59	-3 36 35 -2 46 44 H= 7,K= 5 L F0 FC -4 32 23 -2 34 22	-4 37 42 -2 78 75 1 29 26 3 72 67 5 58 54 6 30 31 H= 8,K= 0	H= 9,K= -3 L FO FC 1 22 12 2 33 32 3 201 97 4 34 37 H= 9,K= -2
5 110 101 5 42 47 4 32 29 H= 5,K= 4 L FO FC -9 21 25 -8 44 56	0 .52 43 1 22 4 3 114 113 4 68 63 5 41 47 6 23 25 8 29 32	-6 29 29 -3 53 56 -2 38 42 -1 182 181 0 200 193 1 42 45 2 49 46 3 68 62	-4 62 55 -3 39 44 -2 80 85 -1 111 101 0 78 78 1 71 55 2 141 147 3 255 261	Hu 6.Ku 5 L FO FC -5 28 28 -4 54 48 -2 45 41 -1 126 131 0 23 30	0 23 20 1 30 32 2 48 52 3 24 13 5 47 51 7 37 31 8 101 106	H= 7.K= 1 1. FO FC -8 62 58 -7 34 39 -6 206 213 -5 197 204 -3 68 66 -2 56 62	H= 8,K= -6 L FD FC 2 23 12 H= 8,K= -5 L FD FC -2 61 56 0 32 30	C FC FC -6 145 144 -5 24 14 -4 28 30 -3 23 16 -1 50 46 0 67 73 1 51 43	L FN FC -4 28 30 -2 26 27 0 59 57 2 53 51 3 92 100 4 69 78
-6 289 299 -5 85 87 -4 81 74 -3 21 20 -2 33 32 -1 44 52 1 101 101 4 64 60	H* 8,K* -5 L FO FC -3 38 32 -1 43 46 0 21 17 1 95 99 2 59 59 3 62 49	4 73 73 5 49 56 6 49 50 7 20 24 8 63 77 H= 6,K= -1 L FO FC	4 30 41 5 63 63 6 23 24 7 28 23 H= 6,K= 2 L FD FC -8 46 43	H= 6,K= 6 L F0 FC -4 35 35 H= 7,K= -7 L F0 FC 0 24 22 4 73 72	H= 7,X= -2 L F0 FC -5 32 33 -4 22 16 -2 77 75 -1 76 84 1 24 23 2 37 45	-1 61 63 0 68 70 2 32 40 3 54 49 4 28 29 5 65 53 6 32 27	1 31 26 3 35 33 H= 8,K= -4 L F0 FC -2 33 34 -1 73 74 0 45 45	3 78 89 5 31 23 H= 8,K= 1 L FO FC -6 50 46 -5 27 27 -2 57 66	H= 9, K= -1 L FO FC -2 46 46 0 22 26 1 22 23 2 45 51 3 37 33
3 37 26 H= 5.K= 5 L F0 FC -9 50 38 -5 32 36 -4 46 45 -3 26 20 -2 45 49	4 74 74 5 27 17 6 44 41 9 32 33 He 6,K= -4 L FD FC -5 46 40 ~4 20 28	-9 28 23 -7 34 44 -4 39 40 -3 83 74 -2 70 71 -1 253 256 0 150 142 1 84 85 2 68 65	-7 84 89 -6 295 307 -3 31 32 -1 71 72 0 42 48 1 59 59 2 56 61 3 38 52 4 215 230	6 23 23 He 7,K= -6 L FO FC -1 40 45 1 42 42 3 51 50 4 50 44 5 51 45	3 44 45 4 70 76 5 52 58 7 31 21 H= 7.K= -1 L FD FC -7 30 31	HT 7,K 2 L F0 FC -8 25 20 -7 39 21 -6 101 107 -5 39 37 -4 40 38 -3 36 34	5 49 46 H# 8.K# -3 L FO FC -1 22 19 O 55 52 1 57 57 2 65 68	-1 95 101 0 31 38 3 40 38 H= 8.Ks 2 L FO FC -6. 53 47 -5 61 54	H= 9,K= 0 L FD FC -4 39 44 -3 48 49 -2 56 56 -1 96 92 0 30 23 1 37 40
-1 67 67 0 44 64 1 38 33 H= 5, x= 6 L F0 FC -6 30 22 -5 70 66 -3 47 45 -2 44 51	-2 27 34 -1 76 79 1 37 33 3 73 75 4 73 70 5 37 35 6 76 79 7 58 53 8 54 59 9 90 90	3 32 24 4 97 96 6 30 21 7 26 17 8 68 72 9 29 26 H= 6,K= 0 L FO FC	5 32 41 6 26 20 H= 6+K= 3 L FO- FC -9 39 33 -8 58 61 -6 70 69 -5 180 179	6 26 23 7 21 3 H 7,K= -5 L F0 FC -6 38 29 0 79 81 1 28 28 2 50 51	-4 45 39 -3 31 33 -2 38 48 -1 67 69 0 45 48 1 79 83 3 220 241 4 115 112 5 106 105	2 26 19 4 26 12 5 82 68 H= 7.K= 3 L FD FC -5 62 56 -4 61 65 -3 22 22	4 175 171 5 55 48 6 39 51 H= 8,K= -2 L FO FC -5 77 79 -2, 49 48 0 22 26	-2 82 88 -1 40 38 0 51 49 1 38 46 3 27 29 He 8,5* 3 L FO FC -1 90 89	H 9.K 1 L FD FC -4 27 24 -2 74 76 -1 81 80 1 24 21 2 23 19

centrosymmetric unit-cell. The 197 reflections whose E values were greater than 1.3 were iteratively phased by application of the modified Sayre equation (Sayre, 1952). The map resulting from the internally most consistent set of phases contained well formed sixmembered rings which were not consistent with the original hypothesis and did not appear to correspond to a chemically plausible structure. The structure was solved by placing fragments apparent in the original

E-map in an acentric unit-cell and using resulting discrepancy factors, electron densities, and thermal parameters to validate these positions. After considerable difficulty, the structure was found to comprise two molecules (M.W. 352) per unit cell related to one another by a center of inversion located at 0.97, 0.72, 0.70. After the coordinates were transformed to a centric unit-cell with the inversion center at the origin, the location of all hydrogen atoms and recognition

Table 2(a). Final coordinates $(\times 10^4)$ and thermal parameters $(\times 10^4$ for anisotropic parameters only) for nonhydrogen atoms of imenine

Standard deviations estimated by least-squares are given in parentheses for least significant digit.

			x	У	Z		В		
		C(1)	6593 (4)	3161 (5)	2505 (4	l) 3·€	5 (8)		
		C(2)	6009 (4)	2664 (5)	3484 (4	Ú 3·6	7 (8)		
		C(3)	4558 (4)	1818 (5)	3428 (3	s) 3·5	8 (8)		
		C(3a)	3646 (4)	1308 (5)	2334 (3	ý <u>3</u> .5	3 (8)		
		C(3 <i>b</i>)	4240 (4)	1786 (5)	1348 (3	s) 3·4	6 (8)		
		C(4)	2162 (4)	328 (5)	2151 (4	kj 3∙9	3 (8)		
		C(5)	1388 (5)	-120(6)	1047 (4	i) 4·6	8 (9)		
		N(6)	1948 (4)	277 (4)	100 (3	5) 4.6	i9 (8)		
		C(6a)	3346 (4)	1205 (5)	237 (4	i) 3·8	32 (8)		
		C(7)	3884 (5)	1587 (5)	- 845 (4	4.0	07 (9)		
		C(7a)	5367 (4)	2743 (5)	- 683 (4	4) 3-8	3 (8)		
		C(8)	5830 (5)	3254 (6)	-1668 (4	l) 4·7	9 (10)		
		C(9)	7140 (5)	4424 (6)	-1557 (5	5) 5.2	4 (10)		
		C(10)	7998 (5)	5081 (6)	-476 (4	4.8	9 (10)		
		C(11)	7564 (5)	4573 (5)	505 (4	4.3	5 (9)	-	
		C(11a)	6236 (4)	3377 (5)	418 (3	3) 3.5	6 (8)		
		C(11b)	5730 (4)	2811 (5)	1448 (3	3) 3.4	5 (8)		
	x	у	Z	β_{11}	β_{22}	β ₃₃	β_{12}	β_{13}	β_{23}
C(12)	9199 (6)	3205 (10)	2701 (7)	111 (8)	328 (16)	171 (9)	8 (9)	-6(7)	110 (10)
C(13)	7210 (8)	1873 (8)	4925 (6)	219 (11)	241 (13)	111 (6)	40 (9)	-20 (7)	82 (8)
C(14)	3413 (9)	2781 (8)	5318 (6)	294 (13)	226 (13)	113 (6)	85 (11)	68 (8)	62 (8)
C(15)	140 (6)	-1260 (9)	2855 (7)	113 (8)	315 (16)	183 (9)	-15 (9)	5 (7)	134 (11)
O(1)	8020 (3)	4102 (4)	2683 (3)	117 (4)	213 (6)	124 (4)	-29(4)	-19(3)	79 (4)
O(2)	6928 (3)	3193 (3)	4523 (2)	166 (5)	176 (6)	93 (3)	-4(4)	-28(3)	54 (3)
O(3)	4036 (3)	1452 (3)	4431 (3)	168 (5)	167 (6)	95 (3)	18 (4)	18 (3)	61 (4)
O(4)	1572 (3)	-134 (4)	3079 (3)	139 (5)	244 (7)	125 (4)	-13(5)	10 (3)	82 (4)
O(5)	3151 (3)	1003 (4)	-1829(3)	168 (5)	256 (7)	93 (3)	9 (5)	-28(3)	48 (4)

Table 2(b). Final coordinates $(\times 10^3)$, thermal parameters, and bond distances for hydrogen atoms of imenine

Standard deviations given in parentheses are for least significant figures. Estimated standard deviations for thermal parameters are 1.2 Å².

	x	у ү	Z	В	D
H(C5)	973 (5)	86 (5)	911 (4)	2∙89 Ų	1·06 (4) Å
H(C8)	511 (4)	267 (5)	754 (4)	2.02	1.05 (4)
H(C9)	729 (6)	469 (7)	772 (5)	6.98	0.95 (6)
H(C10)	895 (5)	599 (6)	970 (4)	4.07	1.01 (5)
H(C11)	818 (4)	506 (5)	125 (4)	1.89	0.95 (4)
H(C12)	-5(7)	356 (8)	234 (6)	6.45	0.86 (6)
H(C12')	882 (9)	201 (12)	253 (8)	11.37	0.96 (9)
H(C12'')	954 (9)	359 (11)	355 (9)	7.38	0·96 (9)
H(C13)	652 (6)	158 (8)	544 (5)	5.29	0·92 (6)
H(C13′)	817 (7)	232 (8)	541 (6)	5.61	0.98 (7)
H(C13'')	714 (6)	75 (8)	422 (6)	6.11	1.00 (6)
H(C14)	306 (6)	246 (6)	597 (5)	4.17	0.93 (5)
H(C14′)	226 (6)	316 (7)	491 (5)	4.63	1.01 (6)
H(C14'')	418 (7)	395 (9)	564 (6)	7.76	1.04 (7)
H(C15)	-9 (6)	853 (7)	359 (6)	5.85	0.95 (6)
H(C15')	-68 (6)	910 (6)	256 (5)	4.11	0.97 (6)
H(C15'')	25 (6)	764 (8)	219 (5)	4.42	0.99 (6)

of nitrogen and oxygen atoms proceeded smoothly. The final full-matrix least-squares refinement* on all positional and isotropic thermal parameters of the ring atoms and the hydrogen atoms and all the positional and anisotropic thermal parameters on the nonring atoms yielded an unweighted discrepancy factor $(\sum ||F_o| - F_c||/\sum |F_o|)$ of 0.062 and a weighted discrepancy factor $\{[\sum w(|F_o| - |F_c|)^2/\sum wF_o^2]^{1/2}\}$ of 0.075.

A comparison between the calculated and observed structure amplitudes is given in Table 1 and the final positional and thermal parameters are in Table 2.

An analysis of the original phases produced with the Sayre equation showed the best set generated

* Scattering factors were obtained from International Tables for X-ray Crystallography, 1968, p. 210.

contained only 62% correct phases and the set used to begin the structure solution contained only 44% correct phases. The apparent reason for the difficulty is that the structure contains a pseudo-center of inversion for 10 of the 26 atoms. The original phasing corresponded to the pseudocenter at the origin; thus the map contained real and image molecules superimposed on one another as in Fig. 1.

Description of the structure

Imenine, pictured in Fig. 2 with bond distances and angles, is shown to be 1, 2, 3, 4-tetramethoxydibenzo-[de, g]quinazolin-7-one. An unusual feature of this compound is the bonding of a methoxy group to the heterocyclic B ring. Substitution in this ring has not



Fig. 1. The imenine molecule and its superimposed image created by the pseudo-center of inversion.



Fig. 2. The structure of imenine with bond distances and angles. Estimated standard deviations are 0.005-0.006 Å and 0.3-0.5° respectively.

been observed in the 12 previously isolated ketoaporphines, while virtually every other available position has had oxygenated functions attached.

Examination of the carbon-carbon bond distances reveals a relatively large range from 1.366 to 1.483 Å. This variation can partially be explained by drawing the common resonance structures for imenine (Fig. 3). None of these structures contain a double bond between C(11a)-C(11b) or around C(7) thus suggesting that these bonds should be somewhat longer. The average carbon-carbon distance in rings A, B and C is 1.399 Å while the C(11a)-C(11b),C(7)-C(6a) and C(7)-C(7a) bonds are 1.483, 1.465 and 1.478 Å respectively. The resonance structures also indicate that the π electrons are not completely delocalized over the entire structure but can be viewed as an aromatic two-ring system (A and B) and a benzene ring (D)





Fig. 4. Packing diagram of imenine showing thermal ellipsoids for non-ring atoms.

separated by the center non aromatic ring (C). The lack of complete delocalization is also manifested in the non-planarity of the rings. The structure is V-shaped with ring B making a 9° dihedral angle with ring D.



Fig. 3. Simple resonance structures of imenine,

The bond angles of $120(\pm 4)^{\circ}$ indicate that the ring carbon atoms are sp^2 hybridized. The angles about the methoxy groups average 116° .

A packing diagram (Fig. 4) shows the two molecules positioned in parallel planes spaced about 4.4 Å apart. Least-squares planes calculated through the strucutre indicate that the substituted oxygen atoms along with C(15) are positioned near the plane of the ring (+0.04Å) while the remaining non-ring carbon atoms are above and below the planes at distances from 1.2 to 1.4 Å.

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The Crystal Structure of a Proposed Fusidic Acid Intermediate

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The structure of a proposed intermediate in the synthesis of the abnormal steroid fusidic acid was determined by means of X-ray analysis. The compound, $C_{23}H_{27}O_3Br$, forms triclinic crystals, space group *P*T, with cell dimensions a=8.681, b=7.361, c=16.077 Å, $\alpha=90.85$, $\beta=99.44$, $\gamma=84.97^{\circ}$; there are two molecules in the cell. The structure was refined to an *R* index of 6.3% for 1979 non-zero reflections measured on a diffractometer. The results of the analysis revealed that the proposed intermediate has a structure incompatible for conversion to the desired steroid.

Introduction

A proposed intermediate (Ia) in the synthesis of the abnormal steroid fusidic acid (II) was prepared in the laboratories of Professor W. G. Dauben at the University of California at Berkeley (Fig. 1). Subsequent conversion of the intermediate was not a trivial undertaking, and conventional methods of organic structure analysis were inconclusive. Therefore, an X-ray analysis was undertaken to establish the structure of the intermediate with certainty.

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Fig. 1. Proposed synthesis.

Experimental

A suitable heavy-atom derivative (Ib) was prepared by reacting the intermediate with 4-bromobenzoyl chloride in pyridine. The resulting reaction product was washed with sodium bicarbonate and was purified by two crystallizations from ethanol. Thin-layer chromatography (silica gel), nuclear magnetic resonance, and infrared and mass spectra revealed that the desired 4-bromobenzoate derivative had been formed in high purity.

Crystals suitable for an X-ray analysis were grown from acetone by slow evaporation. The plate-like crystals were cut to a size of $0.2 \times 0.2 \times 0.1$ mm. A precession camera survey of the crystal indicated that it belonged to a triclinic space group, P1 or P1. Sodium-chloride calibrated, zero level, precession photographs established approximate cell dimensions. More accurate cell dimensions were obtained with a General Electric-Datex diffractometer. Unit-cell dimensions measured on the diffractometer with nickelfiltered copper radiation were as follows:

$a = 8.681 \pm 0.002 \text{ Å}$	$\alpha = 90.85 \pm 0.04^{\circ}$
$b = 7.361 \pm 0.002$	$\beta = 99.44 \pm 0.05$
$c = 16.077 \pm 0.003$	$\gamma = 84.97 \pm 0.02$