Acta Cryst. (1978). B**34**, 2654–2655

2,3,5,6-Tetrahydro-5-phenyl-1*H*-imidazo[1,2-*a*]imidazole (Imafen)

BY P. LAMBERT, G. EVRARD* AND F. DURANT

Groupe de Chimie Physique, Laboratoire de Chimie Moléculaire Structurale, Facultés Universitaires de Namur, 61 rue de Bruxelles, B-5000 Namur, Belgium

(*Received* 22 *February* 1978; *accepted* 21 *March* 1978)

Abstract. $C_{11}H_{13}N_3$, $M_r = 187{\cdot}25$, monoclinic, $P2_1/c$, $a = 6{\cdot}053\ (4)$, $b = 7{\cdot}970\ (4)$, $c = 21{\cdot}528\ (11)$ Å, $\beta = 105{\cdot}18\ (5)°$, $V = 1002{\cdot}3$ Å^3, $D_c = 1{\cdot}24$, $D_m = 1{\cdot}29$ g cm^{-3}, $Z = 4$; $R = 0{\cdot}052$ for 1633 reflexions with $I \geq 2{\cdot}5\sigma$. The conformation of the molecule in the crystal corresponds to that calculated for the isolated molecule.

* To whom correspondence should be addressed.

Table 1. *Experimental conditions*

Crystal dimensions: 0·37 × 0·29 × 0·17 mm
Absences: $h0l$, $l \neq 2n$; $0k0$, $k \neq 2n$; space group $P2_1/c$
Source: graphite-monochromatized Cu $K\bar{\alpha}$ radiation, $\lambda = 1{\cdot}54178$ Å
Scan: ω–2θ
Scan length: $\Delta\theta = 0{\cdot}7 + 0{\cdot}3$ tg θ (°)
Apertures: horizontal = 3·0 + 0·5 tg θ (mm); vertical = 4 mm
$\theta_{min} = 2{\cdot}0°$; $\theta_{max} = 72{\cdot}0°$
Confidence level: $2{\cdot}5\sigma$ with $\sigma^2(I) = S + B + (0{\cdot}03S)^2$, S being the scan width and B the background count
Number of independent reflexions: 2005
Number of accepted reflexions ($\geq 2{\cdot}5\sigma$): 1633
$\mu = 6{\cdot}174$ cm^{-1}; no corrections applied
All observed reflexions given unit weight

Table 2. *Fractional coordinates* (×10^4) *with e.s.d.'s in parentheses*

	x	*y*	*z*
C(1)	3845 (5)	6182 (4)	2383 (2)
C(2)	2011 (6)	5265 (4)	2426 (1)
C(3)	945 (4)	4150 (3)	1946 (1)
C(4)	1750 (4)	3955 (2)	1408 (1)
C(5)	3640 (4)	4880 (4)	1365 (1)
C(6)	4660 (5)	5990 (4)	1861 (2)
C(7)	563 (4)	2831 (3)	857 (1)
C(8)	−1830 (4)	3512 (3)	469 (1)
N(9)	−3273 (4)	2021 (3)	262 (1)
C(10)	−2172 (4)	807 (3)	606 (1)
N(11)	−150 (3)	1209 (2)	1061 (1)
C(12)	1284 (4)	−305 (3)	1172 (1)
C(13)	−572 (5)	−1680 (3)	1035 (1)
N(14)	−2530 (4)	−858 (3)	604 (1)
H(C1)	4750 (41)	7026 (38)	2682 (13)
H(C2)	1414 (43)	5383 (36)	2760 (12)
H(C3)	−337 (41)	3599 (34)	1924 (12)
H(C5)	4797 (43)	4666 (35)	952 (12)
H(C6)	5830 (47)	6536 (39)	1823 (13)
H(C7)	1646 (39)	2630 (32)	524 (11)
H′(C8)	−1614 (41)	4179 (34)	74 (12)
H″(C8)	−2537 (41)	4220 (34)	796 (12)
H′(C12)	2476 (42)	−323 (35)	821 (12)
H″(C12)	2199 (43)	−364 (35)	1679 (12)
H′(C13)	−1108 (44)	−2095 (37)	1467 (13)
H″(C13)	−152 (45)	−2571 (37)	864 (13)
H(N14)	−3986 (44)	−1316 (37)	394 (12)

Introduction. The structure of Imafen was solved as part of a study on the structure–activity relationships of imipramine-like antidepressant drugs. Colourless prismatic crystals were obtained from acetone. The space group was determined by photographic techniques. Cell dimensions and intensities were measured on a Nonius CAD-4 automatic diffractometer. The experimental conditions are given in Table 1.

The structure was solved with *MULTAN* (Germain, Main & Woolfson, 1971). The most probable set of

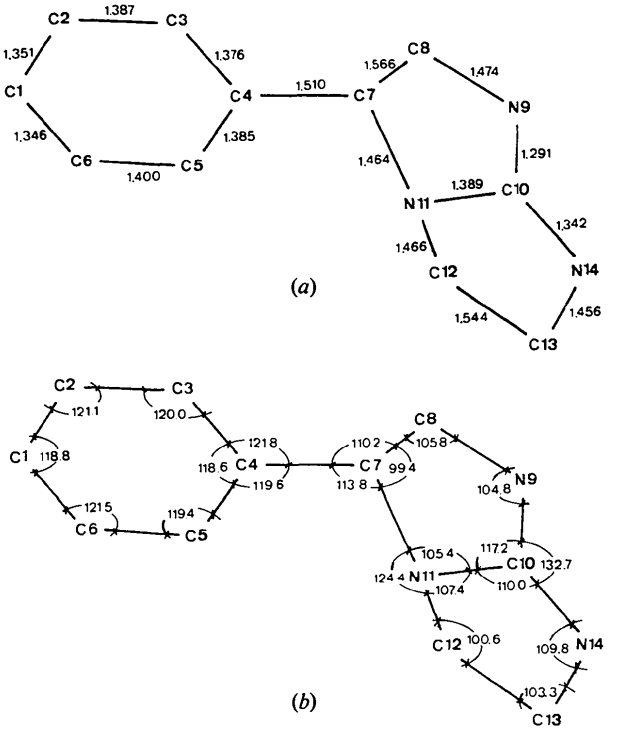

Fig. 1. (*a*) Bond lengths (Å) and (*b*) bond angles (°). The e.s.d.'s for the bond lengths are in the range 0·004–0·007 Å and for the angles 0·3–0·4°.

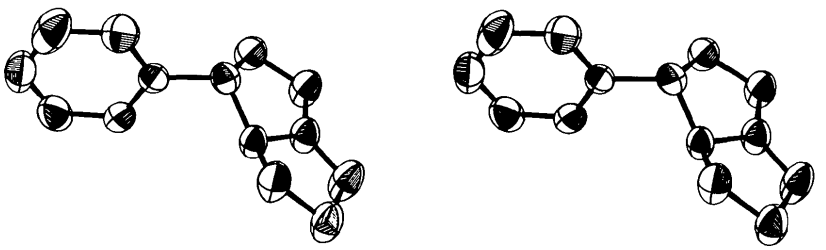

Fig. 2. Stereoscopic view of the molecule with 50% probability thermal ellipsoids (Johnson, 1965).

Table 3. *Bond lengths* (Å) *and angles* (°) *involving* H *atoms*

The e.s.d.'s are in parentheses.

C(1)–H(C1)	0·98 (4)	C(6)–H(C6)	0·86 (4)	C(8)–H″(C8)	1·08 (4)	C(13)–H′(C13)	1·12 (4)
C(2)–H(C2)	0·89 (4)	C(7)–H(C7)	1·10 (3)	C(12)–H′(C12)	1·16 (4)	C(13)–H″(C13)	0·86 (4)
C(3)–H(C3)	0·88 (4)	C(8)–H′(C8)	1·03 (4)	C(12)–H″(C12)	1·08 (4)	N(14)–H(N14)	0·95 (4)
C(5)–H(C5)	1·11 (4)						

C(2)–C(1)–H(C1)	129 (3)	C(4)–C(7)–H(C7)	110 (2)	N(11)–C(12)–H″C(12)	109 (2)
C(6)–C(1)–H(C1)	111 (3)	C(8)–C(7)–H(C7)	109 (2)	C(13)–C(12)–H″(C12)	108 (2)
C(1)–C(2)–H(C2)	120 (3)	N(11)–C(7)–H(C7)	110 (2)	H′(C12)–C(12)–H″(C12)	113 (3)
C(3)–C(2)–H(C2)	118 (3)	C(7)–C(8)–H′(C8)	109 (2)	C(12)–C(13)–H′(C13)	114 (2)
C(2)–C(3)–H(C3)	126 (3)	N(9)–C(8)–H′(C8)	110 (2)	N(4)–C(13)–H′(C13)	108 (2)
C(4)–C(3)–H(C3)	114 (3)	C(7)–C(8)–H″(C8)	108 (2)	C(12)–C(13)–H″(C13)	112 (3)
C(4)–C(5)–H(C5)	121 (2)	N(9)–C(8)–H″(C8)	109 (2)	N(14)–C(13)–H″(C13)	112 (3)
C(6)–C(5)–H(C5)	120 (2)	H′(C8)–C(8)–H″(C8)	115 (3)	H′(C13)–C(13)–H″(C13)	108 (4)
C(1)–C(6)–H(C6)	121 (3)	N(11)–C(12)–H′(C12)	110 (2)	C(10)–N(14)–H(N14)	121 (3)
C(5)–C(6)–H(C6)	117 (3)	C(13)–C(12)–H′(C12)	114 (2)	C(13)–N(14)–H(N14)	129 (3)

signs gave eleven of the fourteen backbone atoms. The three missing ones were readily located on a subsequent Fourier map. Refinement was by full-matrix least squares (Frenz & Okaya, 1975) with scattering factors and anomalous-dispersion coefficients from Cromer & Waber (1974). At the end of the process, all the H atoms were found in a ΔF map and included in the last cycles of refinement. The final positional parameters are given in Table 2.*

Discussion. The atomic numbering and the bond distances and angles are given in Fig. 1. Table 3 gives the bond distances and angles involving H atoms. Fig. 2 shows the conformation of the molecule.

The angle between the two five-membered rings is 13° and the phenyl substituent is nearly perpendicular (99°) to the imidazoimidazole moiety.

Potential-energy calculations, allowing rotation about C(4)–C(7), show that the crystal structure corresponds to the energy minimum of the isolated molecule.

A single hydrogen bond, N(14)–H(14)···N(9) [N(14)–N(9) ($\bar{x}-1, \bar{y}, \bar{z}$) 2·884 (4) Å, H(14)–N(9) ($\bar{x}-1, \bar{y}, \bar{z}$) 1·96 (6) Å], links molecules in the x direction, while cohesion in the remaining two directions is assured by van der Waals forces.

We are grateful to Dr M. H. J. Koch (Janssen Pharmaceutica, B-2340 Beerse, Belgium) who supplied the compound.

* Lists of structure factors and thermal parameters have been deposited with the British Library Lending Division as Supplementary publication No. SUP 33481 (14 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

CROMER, D. T. & WABER, J. T. (1974). In *International Tables for X-ray Crystallography*. Vol. IV. Birmingham: Kynoch Press.

FRENZ, B. & OKAYA, T. (1975). Enraf–Nonius structure determination package. Molecular Structure Corporation, College Station, Texas 77840.

GERMAIN, G., MAIN, P. & WOOLFSON, M. M. (1971). *Acta Cryst.* A**27**, 368–376.

JOHNSON, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee.