

for the sequence N18A—C17A—C16A—C15A—C20A—C19A. For the sequence N18B—C17B—C16B—C15B—C20B—C19B a phase angle $\theta_2 = 4.2(14)^\circ$, $\varphi = 179(22)^\circ$ and $Q = 0.556(14)$ are obtained.

Intermolecular hydrogen bonds (Taylor & Kennard, 1982) are formed between the pyridazinyl hydrogen H7A of molecule A and the pyridazinyl nitrogen N23B of molecule B: $H7A \cdots N23B^i = 2.58(3) \text{ \AA}$. Hydrogen bonds are also found between the pyridazinyl hydrogen H7B of molecule B and the pyridazinyl nitrogen N23A of molecule A: $H7B \cdots N23A^{ii} = 2.62(3) \text{ \AA}$ [symmetry codes: (i) $-x, -y-1, -z+1$; (ii) $-x-1, -y, -z+1$]. No other contacts shorter than the sums of the van der Waals radii are observed.

We thank Dr P. A. J. Janssen (Janssen Pharmaceutica, B-2340 Beerse, Belgium) for providing the R77975 sample. The authors also wish to thank Mr J.-P. Vancuyck for his help with the preparation of the figures.

References

- ANDRIES, K., DEWINDT, B., DE BRABANDER, M., STOKBROEKX, R. & JANSSEN, P. A. J. (1988). *Arch. Virol.* **101**, 155–167.
- ANDRIES, K., STOKBROEKX, R., DEWINDT, B., SNOEKS, J., WILLEBRORDS, R. & JANSSEN, P. A. J. (1989). 29th Interscient. Conf. Antimicrob. Agents Chemother (17–20 September, Houston), Abstract 1327.
- CREMER, D. & POPE, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.
- FRENZ, B. A. (1985). *Enraf-Nonius SDP-Plus Structure Determination Package*. Version 3.0. Enraf-Nonius, Delft, The Netherlands.
- MAIN, P., FISKE, S. J., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1980). *MULTAN80. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.
- MAIN, P., FISKE, S. J., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1982). *MULTAN11/82. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.
- MOTHERWELL, W. D. S. & CLEGG, W. (1978). *PLUTO*. Program for plotting molecular and crystal structures. Univ of Cambridge, England.
- NARDELLI, M. (1983). *Comput. Chem.* **7**, 95–98.
- NORTH, A. C. T., PHILLIPS, D. C. & MATHEWS, F. S. (1968). *Acta Cryst.* **A24**, 351–359.
- STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.
- Stoe & Co. (1985). *REDU4*. Data reduction program. Stoe & Co., Darmstadt, Germany.
- TAYLOR, R. & KENNARD, O. (1982). *J. Am. Chem. Soc.* **104**, 5063–5070.

Acta Cryst. (1991). **C47**, 1520–1523

Structure of β -D-Lyxopyrano[1,2-d]oxazolidin-2-one

BY JÜRGEN KOPF*

Institute of Inorganic and Applied Chemistry, University of Hamburg, Martin-Luther-King-Pl. 6, D-2000 Hamburg 13, Germany

JÓZSEF KOVÁCS

Central Research Institute for Chemistry, Hungarian Academy of Sciences, H-1525 Budapest, Hungary

AND PETER KÖLL

Department of Chemistry, Organic Chemistry, University of Oldenburg, Carl-von-Ossietzky-Str. 9–11, D-2900 Oldenburg, Germany

(Received 21 September 1990; accepted 26 November 1990)

Abstract. C₆H₉NO₅, $M_r = 175.14$, monoclinic, $P2_1$, $a = 5.526(1)$, $b = 9.543(1)$, $c = 13.769(2) \text{ \AA}$, $\beta = 93.74(1)^\circ$, $V = 724.6(2) \text{ \AA}^3$, $Z = 4$, $D_x = 1.60 \text{ g cm}^{-3}$, $\lambda(\text{Cu } K\alpha_1) = 1.54051 \text{ \AA}$, $\mu = 11.8 \text{ cm}^{-1}$, $F(000) = 368$, $T = 293 \text{ K}$, $R = 0.031$ for 1575 observed reflexions. There are two independent

molecules of the title compound in the asymmetric unit. The puckering parameters show that the pyranoid ring is significantly flattened at C(11) and C(21), respectively, in both molecules.

Introduction. Treatment of aldoses with potassium cyanate in the presence of sodium dihydrogenphosphate or ammonium chloride gives 1,2-*cis*-cyclic car-

* Author to whom all correspondence should be addressed.

bamates of glycosylamines of in most cases furanoid structure (Kovács, Pintér, Lending & Köll, 1991). Only D-mannose and D-lyxose prefer to form pyranoid derivatives. Due to difficulties in establishing the structure of these compounds unambiguously from their ¹H NMR spectra, the title compound derived from D-lyxose was subjected to X-ray analysis. To our knowledge this is the first structure determination of such a *cis*-fused carbohydrate carbamate, though the structure of a comparable thiocarbamate (Briard, Roques, Gosselin, Imbach, Montero, Declercq & Germain, 1982) and the structures of two analogous *trans*-fused compounds (Cano, Foces-Foces, Jiménez-Barbero, Alemany, Bernabé & Martín-Lomas, 1987) have been published.

Experimental. Crystals of the title compound were obtained by slow evaporation from ethanol solution. A colorless crystal of dimensions 0.4 × 0.3 × 0.3 mm was used for data collection on an Enraf-Nonius CAD-4 diffractometer with graphite-monochromated Cu K α radiation. The cell parameters were determined by least-squares refinement of the setting angles of 25 reflexions within $78.2 \leq 2\theta \leq 95.5^\circ$. Intensity data were measured by $\theta/2\theta$ scans ($4.5 \leq 2\theta \leq 153^\circ$, $0 \leq h \leq 6$, $0 \leq k \leq 12$, $-17 \leq l \leq 17$). There was no significant decay in the intensity for three standards monitored after every 2 h. 1830 data were measured, of which 1594 were symmetry independent ($R_{\text{int}} = 0.0198$). 1575 intensities were considered observed [$|F_o| > 3\sigma(F_o)$]. The data were corrected for Lorentz and polarization but not for absorption effects.

The structure was solved by direct methods. All H atoms were localized in theoretical positions (sp^2 hybridization for the two N atoms and sp^3 hybridization for all other C atoms) except for H130, H140, H230 and H240 which were taken from a difference map. The structure was refined by full-matrix least-squares procedures on F , assigning anisotropic thermal displacement parameters to all non-H atoms. The refinements converged at $R = 0.031$ and $wR = 0.035$ with 289 parameters including scale factor. The ratio of observations to number of variables is 5.5. The function minimized was $\sum w(|F_o| - |F_c|)^2$ with weights $w = 1/\sigma^2(|F_o|)$. Maximum shift/e.s.d. was 0.37 in the final cycle; maximum and minimum heights in final $\Delta\rho$ map 0.23 and $-0.15 \text{ e } \text{\AA}^{-3}$. The complex neutral-atom scattering factors were taken from *SHELX76* (Sheldrick, 1976). Programs used were *SHELXS86* (Sheldrick, 1986), *SHELX76* and *PLATON88* (Spek, 1982) on MicroVAX II and VAX 3200 computers.

Discussion. Final atomic parameters are given in Table 1, bond distances and angles are reported in

Table 1. *Final fractional coordinates with equivalent isotropic thermal parameters (\AA^2) and of H atoms with general isotropic thermal parameters (\AA^2)*

E.s.d.'s are given in parentheses.

$$U_{\text{eq}} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* a_i a_j$$

Molecule (I)	x	y	z	U_{eq}
O(11)	0.8491 (4)	0.19085 (0)	0.2667 (1)	0.0351 (5)
O(12)	0.3906 (3)	0.3302 (3)	0.3121 (1)	0.0317 (5)
O(13)	0.4427 (4)	0.2416 (3)	0.5071 (1)	0.0377 (6)
O(14)	0.7697 (4)	0.0066 (3)	0.4958 (1)	0.0407 (6)
O(16)	0.2522 (4)	0.3856 (3)	0.1607 (1)	0.0422 (6)
N(11)	0.6597 (4)	0.3906 (3)	0.2088 (1)	0.0344 (6)
C(11)	0.8029 (5)	0.3334 (3)	0.2896 (1)	0.0302 (7)
C(12)	0.6215 (5)	0.3485 (3)	0.3675 (1)	0.0274 (6)
C(13)	0.6510 (5)	0.2452 (3)	0.4511 (1)	0.0289 (6)
C(14)	0.7079 (5)	0.0992 (3)	0.4162 (1)	0.0304 (7)
C(15)	0.9180 (5)	0.1080 (3)	0.3504 (1)	0.0361 (8)
C(16)	0.4207 (5)	0.3708 (3)	0.2196 (1)	0.0304 (7)
H(11)	0.948 (6)	0.388 (3)	0.310 (2)	0.034 (8)
H(12)	0.632 (6)	0.444 (4)	0.396 (2)	0.042 (9)
H(13)	0.797 (5)	0.282 (3)	0.492 (1)	0.027 (6)
H(14)	0.575 (5)	0.061 (3)	0.380 (1)	0.024 (6)
H(111)	0.708 (6)	0.417 (4)	0.150 (2)	0.042 (9)
H(130)	0.364 (9)	0.324 (6)	0.504 (4)	0.10 (1)
H(140)	0.868 (7)	0.057 (4)	0.542 (2)	0.06 (1)
H(151)	0.953 (5)	0.008 (4)	0.330 (2)	0.040 (8)
H(152)	1.062 (7)	0.143 (4)	0.384 (2)	0.043 (9)
Molecule (II)				
O(21)	0.5264 (4)	0.4078 (3)	0.7830 (1)	0.0439 (7)
O(22)	0.9404 (3)	0.2070 (3)	0.8100 (1)	0.0346 (5)
O(23)	1.0117 (5)	0.2703 (3)	0.9993 (1)	0.0560 (7)
O(24)	0.7469 (4)	0.5303 (3)	1.0251 (1)	0.0359 (5)
O(26)	0.9915 (4)	0.1574 (3)	0.6536 (1)	0.0470 (7)
N(21)	0.6136 (4)	0.1992 (3)	0.7091 (1)	0.0356 (6)
C(21)	0.5308 (5)	0.2602 (3)	0.7961 (1)	0.0341 (8)
C(22)	0.7322 (5)	0.2122 (3)	0.8684 (1)	0.0291 (7)
C(23)	0.7790 (5)	0.3051 (3)	0.9565 (1)	0.0309 (7)
C(24)	0.7526 (5)	0.4593 (3)	0.9340 (1)	0.0303 (7)
C(25)	0.5164 (7)	0.4813 (4)	0.8738 (2)	0.0437 (9)
C(26)	0.8578 (5)	0.1863 (3)	0.7168 (1)	0.0339 (7)
H(21)	0.356 (6)	0.225 (4)	0.812 (2)	0.041 (8)
H(22)	0.702 (5)	0.126 (3)	0.888 (2)	0.030 (7)
H(23)	0.663 (7)	0.284 (4)	0.998 (3)	0.05 (1)
H(24)	0.905 (6)	0.487 (4)	0.891 (2)	0.06 (1)
H(211)	0.542 (6)	0.203 (4)	0.646 (2)	0.038 (9)
H(230)	1.052 (9)	0.312 (5)	1.049 (3)	0.09 (1)
H(240)	0.831 (8)	0.601 (5)	1.019 (3)	0.06 (1)
H(251)	0.485 (7)	0.581 (4)	0.860 (3)	0.06 (1)
H(252)	0.369 (6)	0.444 (4)	0.912 (2)	0.05 (1)

Table 2.* Fig. 1 shows a view of molecule (I) and illustrates the atom-numbering scheme chosen. The atom numbers of molecule (II) follow the same scheme, but the atom numbers all start with the digit 2. The corresponding distances and angles of the two independent molecules compare very well; the average deviations are 0.005 (4) \AA and 0.8 (5) $^\circ$, respectively.

The title compound contains four 'stereogenic centers' at the C atoms C(11), C(12), C(13) and C(14). Both independent molecules display the same absolute D-configuration at C(14) and C(24), respectively, which is unambiguous, because the title com-

* Tables of structure factors and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53787 (17 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. Bond lengths (Å) and angles (°)

	Molecule (I)	Molecule (II)
O(11)—C(11)	1.423 (3)	1.420 (5)
O(11)—C(15)	1.429 (3)	1.438 (4)
O(12)—C(12)	1.454 (3)	1.447 (3)
O(12)—C(16)	1.351 (3)	1.349 (3)
O(13)—C(13)	1.428 (3)	1.418 (4)
O(14)—C(14)	1.432 (3)	1.428 (3)
O(16)—C(16)	1.203 (3)	1.209 (3)
N(11)—C(11)	1.431 (3)	1.433 (3)
N(11)—C(16)	1.352 (4)	1.352 (4)
C(11)—C(12)	1.522 (4)	1.515 (4)
C(12)—C(13)	1.515 (4)	1.512 (4)
C(13)—C(14)	1.513 (4)	1.509 (4)
C(14)—C(15)	1.521 (4)	1.514 (4)
O(13)—H(130)	0.90 (6)	0.81 (4)
O(14)—H(140)	0.94 (3)	0.83 (5)
N(11)—H(111)	0.90 (3)	0.93 (3)
C(11)—H(11)	0.98 (3)	1.06 (3)
C(12)—H(12)	0.99 (4)	0.88 (3)
C(13)—H(13)	1.02 (3)	0.91 (4)
C(14)—H(14)	0.93 (3)	1.09 (3)
C(15)—H(151)	1.02 (4)	0.98 (4)
C(15)—H(152)	0.95 (4)	1.06 (3)
C(11)—O(11)—C(15)	113.1 (2)	112.0 (2)
C(12)—O(12)—C(16)	107.6 (2)	107.5 (2)
C(11)—N(11)—C(16)	110.8 (2)	110.1 (2)
O(11)—C(11)—N(11)	106.9 (2)	107.5 (2)
O(11)—C(11)—C(12)	112.5 (2)	112.9 (2)
N(11)—C(11)—C(12)	99.0 (2)	99.5 (2)
O(12)—C(12)—C(11)	102.4 (2)	102.8 (2)
O(12)—C(12)—C(13)	111.6 (2)	111.4 (2)
C(11)—C(12)—C(13)	115.5 (2)	115.5 (2)
O(13)—C(13)—C(12)	112.1 (2)	107.4 (2)
O(13)—C(13)—C(14)	109.9 (2)	112.7 (2)
C(12)—C(13)—C(14)	111.9 (2)	113.4 (2)
O(14)—C(14)—C(13)	111.7 (2)	106.8 (2)
O(14)—C(14)—C(15)	109.7 (2)	110.3 (2)
C(13)—C(14)—C(15)	108.7 (2)	108.4 (2)
O(11)—C(15)—C(14)	109.7 (2)	109.1 (3)
O(12)—C(16)—O(16)	122.1 (2)	122.2 (2)
O(12)—C(16)—N(11)	109.0 (2)	109.5 (2)
O(16)—C(16)—N(11)	128.9 (2)	128.2 (2)
C(13)—O(13)—H(130)	111 (3)	116 (4)
C(14)—O(14)—H(140)	107 (2)	105 (3)
C(11)—N(11)—H(111)	128 (2)	129 (2)
C(16)—N(11)—H(111)	119 (2)	116 (2)
O(11)—C(11)—H(11)	115 (1)	109 (2)
N(11)—C(11)—H(11)	115 (1)	113 (1)
C(12)—C(11)—H(11)	108 (1)	114 (1)
O(12)—C(12)—H(12)	110 (1)	108 (1)
C(11)—C(12)—H(12)	110 (1)	110 (1)
C(13)—C(12)—H(12)	107 (1)	109 (1)
O(13)—C(13)—H(13)	110 (1)	110 (3)
C(12)—C(13)—H(13)	104 (1)	107 (3)
C(14)—C(13)—H(13)	109 (1)	106 (2)
O(14)—C(14)—H(14)	108 (1)	115 (1)
C(13)—C(14)—H(14)	111 (1)	106 (2)
C(15)—C(14)—H(14)	108 (1)	110 (1)
O(11)—C(15)—H(151)	110 (1)	109 (2)
O(11)—C(15)—H(152)	112 (2)	110 (1)
C(14)—C(15)—H(151)	106 (1)	112 (2)
C(14)—C(15)—H(152)	112 (1)	110 (1)
H(151)—C(15)—H(152)	107 (3)	107 (3)

Table 3. Puckering parameters (Cremer & Pople, 1975) (Å, °)

Five-membered ring			
	q_2	φ_2	
Molecule (I)			
O(12)—C(12)—C(11)—N(11)—C(16)	0.320 (3)	55.4 (5)	
Molecule (II)			
O(22)—C(22)—C(21)—N(21)—C(26)	0.307 (3)	56.4 (5)	
Six-membered ring			
	Q	θ	φ
Molecule (I)			
O(11)—C(11)—C(12)—C(13)—C(14)—C(15)	0.536 (3)	16.5 (3)	293.2 (10)
Molecule (II)			
O(21)—C(21)—C(22)—C(23)—C(24)—C(25)	0.538 (3)	20.1 (3)	301.9 (9)

Table 4. Geometry of the hydrogen bonds (Å, °)

D—H...A	D—H	D...A	H...A	D—H...A	Symmetry operation*
N(11)—H(111)...O(24)	0.90 (3)	2.926 (3)	2.05 (3)	162 (3)	$x, y, -1+z$
O(13)—H(130)...O(14)	0.90 (6)	2.787 (4)	1.89 (6)	173 (5)	$1-x, \frac{1}{2}+y, 1-z$
O(14)—H(140)...O(26)	0.94 (3)	2.818 (3)	1.90 (3)	165 (3)	x, y, z
N(21)—H(211)...O(13)	0.93 (3)	2.907 (3)	1.99 (3)	168 (3)	x, y, z
O(23)—H(230)...O(16)	0.81 (4)	2.745 (3)	1.97 (4)	161 (5)	$1+x, y, 1+z$
O(24)—H(240)...O(23)	0.83 (5)	2.683 (4)	1.86 (5)	174 (4)	$2-x, \frac{1}{2}+y, 2-z$

* The symmetry operation is performed on the acceptor atoms. D indicates donor and A acceptor.

pound has been synthesized from enantiomerically pure D-lyxose.

Table 3 gives the puckering parameters (Cremer & Pople, 1975). As also indicated by the ring torsion angles, the pyranoid ring is significantly flattened at C(11) and C(21) in both independent molecules. The θ and φ values for the six-membered rings indicate a conformation between the perfect 4C_1 chair (ideal value: $\theta = 0^\circ$) and the envelope E_5 (ideal values: $\theta = 45^\circ$, $\varphi = 300^\circ$). The φ_2 values close to 54° for the five-membered rings indicate a rather pure 2T_1 conformation (Köll, John & Kopf, 1982).

The conformational distortion of the pyranoid ring is also shown by the unusually small torsion angles of $35(3)^\circ$ for H(11)—C(11)—C(12)—H(12) and of $-47(3)^\circ$ for H(12)—C(12)—C(13)—H(13) which are in good agreement with the corresponding $J_{11,12}$ and $J_{12,13}$ coupling constants from the ${}^1\text{H}$ NMR spectrum of the title compound (Kovács, Pintér, Lendering & Köll, 1990).

As in all carbohydrates with free hydroxyl groups the molecules (I) and (II) are interlinked in the crystal by a complicated hydrogen-bonding network. N—H...A bonds are also included in this network. Only the strongest interactions are summarized in Table 4. In these hydrogen bonds both N—H as well as all O—H bonds act as donors and O(13), O(14), O(23), O(24) as well as O(16) and O(26) (carbonyl oxygens) act as acceptors. Oxygen atoms O(11), O(12), O(21) and O(22) are not involved in this network of hydrogen bonds.

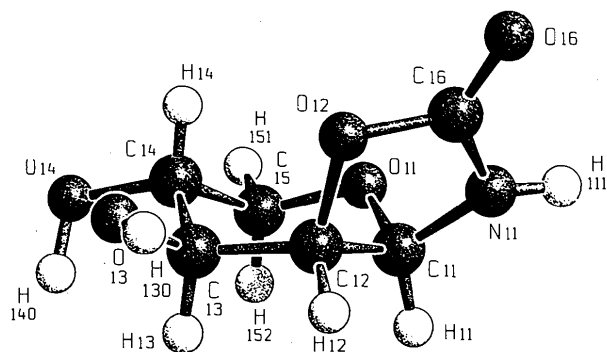


Fig. 1. SCHAKAL88 drawing (Keller, 1986) of molecule (I) of β -D-lyxopyrano[1,2-*d*]oxazolidin-2-one.

We gratefully acknowledge financial support from the Deutsche Forschungsgemeinschaft and the Fonds der Chemischen Industrie.

References

- BRIARD, P., ROQUES R., GOSSELIN, G., IMBACH, J. L., MONTERO, J. L., DECLERCQ, J.-P. & GERMAIN, G. (1982). *Acta Cryst.* **B38**, 1027–1030.
- CANO, F. H., FOCES-FOCES, C., JIMÉNEZ-BARBERO, J., ALEMANY, A., BERNABÉ, M. & MARTÍN-LOMAS, M. (1987). *J. Org. Chem.* **52**, 3367–3372.
- CREMER, D. & POPLE, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.
- KELLER, E. (1986). *Chem. Unserer Zeit*, **20**, 178–181.
- KÖLL, P., JOHN, H.-G. & KOPF, J. (1982). *Justus Liebigs Ann. Chem.* pp. 626–638.
- KOVÁCS, J., PINTÉR, I., LENDERING, U. & KÖLL, P. (1991). *Carbohydr. Res.* In the press.
- SHELDRIK, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.
- SHELDRIK, G. M. (1986). *SHELXS86*. Programs for the solution of crystal structures. Univ. of Göttingen, Germany.
- SPEK, A.L. (1982). *PLATON88*. In *Computational Crystallography*, edited by D. SAYRE, p. 528. Oxford Univ. Press.

Acta Cryst. (1991). **C47**, 1523–1525

Structure of 1-Acetyl-7,8-dimethoxy-2,3,4-*ij*-isoquinolin-2(1*H*)-one

By JONATHAN D. STREET, OWEN S. MILLS AND JOHN A. JOULE*

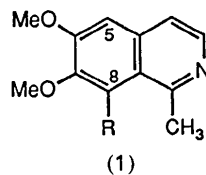
Department of Chemistry, Victoria University of Manchester, Manchester M13 9PL, England

(Received 4 October 1990; accepted 17 December 1990)

Abstract. $C_{14}H_{12}N_2O_4$, $M_r = 272.3$, monoclinic, $P2_1/c$, $a = 10.385$ (2), $b = 16.419$ (3), $c = 7.539$ (5) Å, $\beta = 106.73$ (4)°, $V = 1231.0$ (9) Å³, $Z = 4$, $D_x = 1.47$ Mg m⁻³, $\lambda(Mo K\alpha) = 0.71069$ Å, $\mu = 1.19$ cm⁻¹, $F(000) = 568$, $T = 295$ K, $R = 5.9\%$ for 1363 observed [$I/\sigma(I) \geq 3$] reflexions. The title tricyclic is essentially flat, the 12 atoms of the ring system lying within 0.05 Å of their least-squares best plane. The two carbonyl groups of the imide functional group are oriented *anti*, with the substituent planar acetyl group making a dihedral angle of 18° with the five-membered ring. This close planarity forces the adjacent methoxy methyl out of the plane to the extent of 80°; the other methoxy lies essentially in the plane of the molecule. The structure of this tricyclic imide confirms that nitration of 6,7-dimethoxy-1-methylisoquinoline takes place at the more hindered benzene ring carbon, C8.

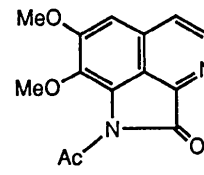
Introduction. In acid solution isoquinolines typically undergo electrophilic substitution *via* the isoquinolinium cation (Brown & Harcourt, 1960); nitration occurs at C5 and C8 with the former predominating to the extent of 9:1 (Dewar & Maitlis, 1957). Using 6,7-dimethoxy-1-methylisoquinoline (1*a*) (Spath & Polgar, 1929) as starting material and requiring from it an intermediate with a nitrogen substituent at C8, we initially assumed that the intrinsic isoquinoline 5 > 8 positional electrophilic substitution tendency would, if anything, be enhanced, in the undesired sense, by the presence of the 1-methyl group and

therefore that direct nitration of (1*a*) would lead predominantly to a 5-nitro derivative. Accordingly we explored the possibility of reversibly blocking the 5-position and indeed bromination did give (mainly) the 5-bromo derivative, which *via* subsequent 8-nitration and hydrogenolytic removal of halogen allowed entry to the desired series. However, we subsequently found that low temperature direct nitration of (1*a*) gave (mainly) a mononitro derivative (1*b*) (Balczewski, Street, Mallon & Joule, 1990) which did not have the predicted 5-nitro structure. It was shown to be the 8-nitro isomer by spectroscopic means, and by a chemical sequence which culminated in a tricyclic imide, shown by the X-ray crystallographic study described herein to have structure (2). One other compound with this ring structure has been reported (Kubo, Saito, Nakahara & Iwata, 1982).



(1)

R a H b NO₂ c NH₂ d NHAc



(2)

Experimental. Nitration of 6,7-dimethoxy-1-methylisoquinoline (1*a*) with fuming nitric acid at 223 K gave a mononitro derivative which was proved to be (1*b*). Thus, reduction of 6,7-dimethoxy-1-methyl-8-nitroisoquinoline (1*b*) gave the corresponding amine (1*c*) which, after *N*-acetylation

* Author to whom correspondence should be addressed.