with 1,4,8,11-tetrathiacyclotetradecane [in its $Cu(ClO_4)_2$ complex] (Glick *et al.*, 1976). Strained torsion angles of 90 to 110° may be attributed to the small ring size [*cf.* '12-crown-4' (Groth, 1978)] as compared to the large diameter of the S atoms, whilst the pyridine moiety does not necessarily induce conformational irregularities (Maverick, Grossenbacher & Trueblood, 1979; Weber, 1982).

Ca 16% occupied and hence less precisely determined minor-component positions may indicate a relatively small energy difference between the major and minor conformation. In the latter, the coplanarity of the four heteroatoms (within $\pm$ 0·09 Å) and the angle N(1)···S(7)′/S(4)′···S(10)′ = 89·8 (7)° meet two conditions required for transition-metal complexes with regular coordination polyhedra. From the N(1)···S(7)′ distance of 4·42 (1) Å as compared to the sum of the van der Waals radii of N and S = 3·35 Å (*Handbook of Chemistry and Physics*, 1970), cations with $r = 0{\cdot}5$ to *ca* 0·7 Å should be suitable guest species. The same procedure, applied to S(4)′···S(10)′ = 6·51 (2) Å, however, results in $r = 1{\cdot}4$ to *ca* 1·6 Å. Therefore it might be assumed that (*a*) the ligand has to adopt a third conformation on coordinating (which seems rather doubtful in view of the restricted flexibility of the small macrocycle) or (*b*) that not all heteroatoms will serve as electron donors; the latter might explain some unusual stoicheiometric ligand:cation ratios (Weber & Vögtle, 1976).

Pyridine nuclei related by inversion at $\frac{1}{2}$,0,0, are 3·59 (2) Å apart; this distance and an overlap of *ca* 15% will probably not allow appreciable $\pi\cdots\pi$ interactions.

Table 3. *Torsion angles* (°)

C(12)–N(1)–C(2)–C(3)	−179·0 (2)		
C(12)–N(1)–C(2)–C(15)	−1·2 (3)		
C(2)–N(1)–C(12)–C(11)	178·4 (2)		
C(2)–N(1)–C(12)–C(13)	−0·4 (3)		
N(1)–C(2)–C(3)–S(4)	109·2 (2)	N(1)–C(2)–C(3)–S(4)′	99·9 (3)
C(15)–C(2)–C(3)–S(4)	−68·7 (3)	C(15)–C(2)–C(3)–S(4)′	−78·0 (4)
N(1)–C(2)–C(15)–C(14)	1·6 (3)		
C(3)–C(2)–C(15)–C(14)	179·4 (2)		
C(2)–C(3)–S(4)–C(5)	−51·4 (2)	C(2)–C(3)–S(4)′–C(5)′	−69·9 (7)
C(3)–S(4)–C(5)–C(6)	−68·0 (3)	C(3)–S(4)′–C(5)′–C(6)′	99·7 (11)
S(4)–C(5)–C(6)–S(7)	176·0 (2)	S(4)′–C(5)′–C(6)′–S(7)′	−176·7 (9)
C(5)–C(6)–S(7)–C(8)	−90·8 (3)	C(5)′–C(6)′–S(7)′–C(8)′	112·2 (12)
C(6)–S(7)–C(8)–C(9)	92·2 (3)	C(6)′–S(7)′–C(8)′–C(9)′	−103·1 (11)
S(7)–C(8)–C(9)–S(10)	−177·7 (2)	S(7)′–C(8)′–C(9)′–S(10)′	178·9 (8)
C(8)–C(9)–S(10)–C(11)	67·5 (2)	C(8)′–C(9)′–S(10)′–C(11)′	−110·1 (10)
C(9)–S(10)–C(11)–C(12)	49·3 (2)	C(9)′–S(10)′–C(11)–C(12)	66·4 (5)
S(10)–C(11)–C(12)–C(13)	73·3 (3)	S(10)′–C(11)–C(12)–C(13)	84·0 (3)
S(10)–C(11)–C(12)–N(1)	−105·5 (2)	S(10)′–C(11)–C(12)–N(1)	−94·7 (3)
N(1)–C(12)–C(13)–C(14)	1·5 (3)		
C(11)–C(12)–C(13)–C(14)	−177·2 (2)		
C(12)–C(13)–C(14)–C(15)	−1·0 (4)		
C(13)–C(14)–C(15)–C(2)	−0·5 (4)		

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Acta Cryst. (1983). C**39**, 391–393

Ethyl [1,2,4]Triazolo[1,5-*a*]pyridine-2-carboxylate, $C_9H_9N_3O_2$

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Abstract. $M_r = 191{\cdot}189$, monoclinic, $P2_1/c$, $a = 9{\cdot}853$ (3), $b = 6{\cdot}232$ (3), $c = 16{\cdot}020$ (3) Å, $\beta = 106{\cdot}45$ (2)°, $V = 943{\cdot}4$ (6) Å^3, $Z = 4$, $D_x = 1{\cdot}316$, $D_m = 1{\cdot}34$ Mg m^{-3}, μ(Cu $K\alpha$) = 0·7833 mm^{-1}, $\lambda = 1{\cdot}5418$ Å, $F(000) = 400$. Final $R = 0{\cdot}056$ for 1350 observed reflections. The molecules, which are practically planar, are held together only by van der Waals interactions.

Introduction. The structure of the title compound has been determined and refined to characterize unambiguously the conformation of the molecule and the positions of the N atoms.

Experimental. Samples provided by Drs C. Avendaño and E. Gomez-Molinero from the Departamento de Química Orgánica de la Facultad de Farmacia de la

0108-2701/83/030391-03$01.50

Universidad Complutense de Madrid, Spain; D_m measured by flotation, white, elongated prisms, $0{\cdot}3 \times 0{\cdot}4 \times 0{\cdot}2$ mm, Philips PW-1100 automatic diffractometer, Ni-filtered Cu $K\alpha$, 1748 $\pm hkl$ (max. hkl range, 11,7,18) with $\theta < 76°$, 1601 independent, 1350 with $I > 2\sigma(I)$, Lp correction, absorption ignored, 25 reflections used for measuring lattice parameters; direct methods *MULTAN* 80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980), anisotropic full matrix, H (from ΔF synthesis) isotropic, weighting scheme $w = K/|f(F_o)|^2$ (Martínez-Ripoll & Cano, 1975) with $f(F_o) = 0{\cdot}31 + 0{\cdot}01|F_o|$, $K = 0{\cdot}56$ for all values of $|F_o|$, final $R = 0{\cdot}056$ and $R_w = 0{\cdot}057$; ratio of LS shift-to-error mean $= 0{\cdot}06$, final difference Fourier max. $= 0{\cdot}23$ e Å^{-3}, scattering factors from *International Tables for X-ray Crystallography* (1974), Univac 1108 computer, programs from XRAY 70 system (Stewart, Kundell & Baldwin, 1970) and *PARST7–PLALIN* (Nardelli, 1980).

Discussion. The atomic coordinates and isotropic thermal parameters are given in Table 1.*

The atomic labelling, bond lengths and angles for the non-H atoms are shown in Fig. 1. The bond lengths in the two rings are shorter than sp^3–sp^3 bonds and larger than sp^2–sp^2 double bonds. The two fused rings are coplanar as shown by the torsion angles (Table 2) in agreement with an aromatic delocalization of the π electrons in them. Consequently several canonical formulae are possible, *A*, *B* and *C* being the predominant forms.

Table 1. *Atomic coordinates and isotropic thermal parameters (for non-*H *atoms* $\times 10^3$, *for* H $\times 10^2$)

$$U_{\rm eq} = \tfrac{1}{3}\textstyle\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$
$$U = \exp[-8\pi^2 U(\sin\theta/\lambda)^2].$$

	x	y	z	$U_{\rm eq}/U$(Å^2)
N(1)	0·2381 (2)	0·4178 (3)	0·3273 (1)	44 (1)
N(2)	0·1525 (2)	0·2605 (3)	0·3424 (1)	45 (1)
C(3)	0·2435 (2)	0·1396 (4)	0·4000 (1)	40 (1)
N(4)	0·3791 (2)	0·2041 (4)	0·4233 (1)	46 (1)
C(5)	0·3739 (2)	0·3831 (4)	0·3761 (1)	39 (1)
C(6)	0·4809 (3)	0·5267 (5)	0·3712 (2)	55 (1)
C(7)	0·4437 (3)	0·6958 (5)	0·3156 (2)	61 (1)
C(8)	0·3048 (4)	0·7246 (5)	0·2661 (2)	65 (1)
C(9)	0·1997 (3)	0·5887 (5)	0·2709 (2)	57 (1)
C(10)	0·2010 (3)	−0·0579 (4)	0·4379 (1)	44 (1)
O(11)	0·2834 (3)	−0·1798 (4)	0·4926 (1)	77 (1)
O(12)	0·0612 (2)	−0·0916 (3)	0·4074 (1)	50 (1)
C(13)	0·0075 (3)	−0·2765 (5)	0·4424 (2)	54 (1)
C(14)	−0·1459 (4)	−0·2961 (7)	0·3955 (2)	70 (1)
H(61)	0·572 (4)	0·506 (6)	0·403 (2)	5 (2)
H(71)	0·518 (5)	0·793 (7)	0·309 (2)	6 (2)
H(81)	0·289 (4)	0·843 (7)	0·228 (2)	6 (2)
H(91)	0·104 (4)	0·603 (6)	06241 (2)	5 (2)
H(131)	0·022 (4)	−0·244 (6)	0·508 (2)	5 (2)
H(132)	0·065 (4)	−0·408 (6)	0·433 (2)	5 (2)
H(141)	−0·170 (4)	−0·319 (7)	0·330 (3)	7 (2)
H(142)	−0·190 (4)	−0·161 (8)	0·408 (3)	7 (2)
H(143)	−0·181 (4)	−0·431 (7)	0·419 (3)	7 (2)

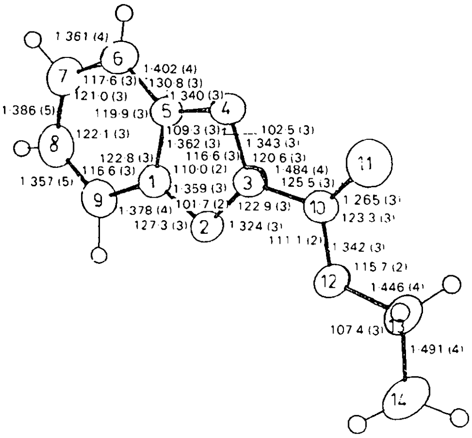
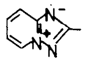


Fig. 1. Atomic numbering and bond lengths (Å) and angles (°).

Table 2. *Torsion angles* (°)

C(5)–N(1)–C(9)–C(8)	−0·3 (5)	N(4)–C(3)–C(10)–O(11)	−0·4 (5)
N(2)–N(1)–C(9)–C(8)	−179·3 (3)	N(4)–C(3)–C(10)–O(12)	178·9 (2)
N(2)–N(1)–C(5)–C(6)	−179·7 (3)	C(10)–C(3)–N(4)–C(5)	180·0 (2)
C(9)–N(1)–C(5)–N(4)	−179·0 (3)	C(3)–N(4)–C(5)–N(1)	−0·1 (3)
N(2)–N(1)–C(5)–N(4)	0·1 (3)	C(3)–N(4)–C(5)–C(6)	179·7 (3)
C(5)–N(1)–N(2)–C(3)	−0·1 (3)	N(4)–C(5)–C(6)–C(7)	179·1 (3)
C(9)–N(1)–N(2)–C(3)	179·0 (3)	N(1)–C(5)–C(6)–C(7)	−1·1 (5)
C(9)–N(1)–C(5)–C(6)	1·1 (4)	C(5)–C(6)–C(7)–C(8)	0·3 (5)
N(1)–N(2)–C(3)–N(4)	0·1 (3)	C(6)–C(7)–C(8)–C(9)	0·6 (6)
N(1)–N(2)–C(3)–C(10)	−179·9 (3)	C(7)–C(8)–C(9)–N(1)	−0·6 (5)
N(2)–C(3)–C(10)–O(11)	179·6 (3)	C(3)–C(10)–O(12)–C(13)	−178·0 (2)
N(2)–C(3)–C(10)–O(12)	−1·2 (4)	O(11)–C(10)–O(12)–C(13)	1·2 (4)
N(2)–C(3)–N(4)–C(5)	0·0 (3)	C(10)–O(12)–C(13)–C(14)	−175·4 (3)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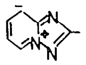
(*A*)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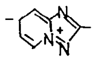
(*B*)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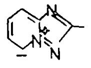
(*C*)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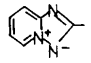
(*D*)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(*E*)

In the hexagonal ring the distances and angles are in agreement with those found by Van Havere, Lenstra, Geise, Van den Berg & Benschop (1982) in aromatic pyridine rings forming part of different molecules. The distances in the pentagonal ring are also similar to those given by Sabesan & Venkatesan (1971) and Owston & Rowe (1962). The ethoxycarbonyl radical attached to C(3) is nearly planar, with a torsion angle C(13)–C(10)–O(12)–C(13) of 178·(2)°, slightly larger than the value 174° typical for esters (Chananont & Hamor, 1981). Moreover, the ethoxycarbonyl group is coplanar with the annular system as can be seen from the torsion angles involving the C(13)–C(10) bond. Bond lengths and angles in that group are in good

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* Lists of structure factors, anisotropic thermal parameters and a projection of the structure along **a** have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38243 (13 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

agreement with those found in the literature (Vega, López-Castro & Márquez, 1981; Bertz, Rihs & Woodward, 1982).

There are no intermolecular contacts shorter than those required for van der Waals interactions.

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Acta Cryst. (1983). C**39**, 393–395

N,N,N′,N′-Tetracyclohexyloxydiglycolamide Hemihydrate, $C_{28}H_{48}N_2O_5.\frac{1}{2}H_2O$

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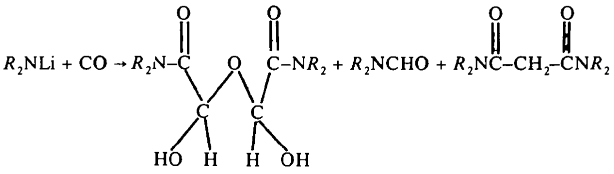
Abstract. $M_r = 501\cdot20$, monoclinic, $C2/c$, $a = 14\cdot506\ (4)$, $b = 15\cdot489\ (7)$, $c = 13\cdot875\ (2)$ Å, $\beta = 115\cdot90\ (2)°$, $V = 2804\ (2)$ Å^3, $Z = 4$, $D_x = 1\cdot19$ Mg m^{-3}, Mo $K\alpha$ ($\lambda = 0\cdot71069$ Å), $\mu = 0\cdot76$ cm^{-1}, $F(000) = 1080$, room temperature, $R = 0\cdot050$ for 1673 unique reflections with intensities greater than $3\sigma(I)$. The amide nitrogen is trigonal planar with a C—N bond distance of 1·355 (3) Å. There is an intramolecular hydrogen bond involving the carbonyl and the hydroxyl group on an adjacent carbon atom.

Introduction. Recently, it was shown that the migratory insertion reaction of carbon monoxide into C—Li bonds could be used to produce useful intermediates in organic syntheses (Nudelman & Vitale, 1981; Nudelman & Outumuro, 1982). Insertion of carbon monoxide into N—Li bonds has not been as well studied (Longhi, Montagna & Mazzochi, 1965; Jutzi & Schroder, 1971; Rautenstrauch & Joyeaux, 1979), and the results have been contradictory. The reaction of lithium dicyclohexylamide (1) with carbon monoxide led to the formation in high yields (85%) of *N,N,N′,N′*-tetracyclohexyl oxyglycolamide hemihydrate (2) in a one-pot reaction. The conventional preparation of oxyglycolamides involves a multistep synthesis. Compounds (3) and (4) are also produced in the reaction, but reaction conditions can be adjusted to maximize the production of (2).



(1) (2) (3) (4)

$R = C_6H_{11}$