

and the dispersion correction for Cd were taken from *International Tables for X-ray Crystallography* (1959, 1962). All Fourier and refinement calculations were carried out with the XRAY 70 system (Stewart, Kundell & Baldwin, 1970) on the DEC10 computer of the Institut Laue-Langevin. Final positional parameters are listed in Table 1. Interatomic bond lengths and bond angles are given in Table 2; the unit cell is depicted in Fig. 1.*

Discussion. The structure of $\text{Li}_2\text{CdSiO}_4$ is related to the wurzite structure. The metal atoms occupy the cation sites in an ordered way. Of special interest is the ordering of Li atoms in chains parallel to the a axis, with a distance of 3.24 Å between two neighbouring Li

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 32589 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

atoms in a chain. The SiO_4 tetrahedra are less distorted than they are in synthetic (Riekel, 1973) or natural (Birle, Gibbs, Moore & Smith, 1968) olivines. This distortion is also evident in the infrared spectrum of $\text{Li}_2\text{CdSiO}_4$ as the peak attributed to the ν_3 vibration of the SiO_4 tetrahedron is split because of the reduced site symmetry.

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(3-Pyrida-3-[2,6])(6-benza-6-[1,4;2,5])(9-pyrida-9-[2,6])spiro[5.5]undecaphane-1,4,7,10-tetraene

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Abstract. $\text{C}_{24}\text{H}_{16}\text{N}_2$, FW 332.4; monoclinic, $P2_1/c$, $a = 9.233$ (9), $b = 8.801$ (9), $c = 22.29$ (2) Å, $\beta = 107.08$ (5)° at 20°C, $\lambda(\text{Cu } K\alpha_1) = 1.54050$ Å; $V = 1731.3$ Å³; $D_m = 1.29$, $Z = 4$, $D_x = 1.28$ g cm⁻³. The molecule has non-crystallographic twofold rotation symmetry. The pyridine rings are inclined at 75° to the central benzene nucleus, which is distorted to a twist-boat conformation.

Introduction. The crystals supplied were amber in colour and wedge-shaped, with prominent forms {110}, {011} and {001}. The space group was determined from single-crystal photographs (absent reflexions: $h0l$ for l odd; $0k0$ for k odd). The specimen used for intensity measurements was of nominal dimensions $0.23 \times 0.23 \times 0.17$ mm and was mounted about c^* . The intensities of the 2938 independent reflexions within the limiting sphere $2\theta = 130^\circ$ were measured with a four-circle diffractometer, using Ni-filtered Cu $K\alpha$ radiation and a scintillation counter. The θ - 2θ scan

mode was used, with scans of 2° for $2\theta < 100^\circ$, and 3° otherwise. Background counts were accumulated for 10 s at the end of each scan. For 594 reflexions the net count was less than either 60, or 10% of the corresponding background; these were considered to be unobserved, and were not used in the analysis. No corrections were made for absorption ($\mu = 5.9$ cm⁻¹).

The structure was determined uneventfully by symbolic addition procedures. Refinement was by block-diagonal least squares, minimizing $\sum w(\Delta F)^2$. The H atoms were placed in chemically reasonable positions, and were included in the refinement. The 33 most intense reflexions were corrected for extinction in the manner described by Pinnock, Taylor & Lipson (1956), the largest correction being 10% of F_o . The weighting scheme used in the final stages was $w = w_1 w_2$ where $w_1 = F_o/6$ for $F_o < 6$, = 1 otherwise, and $w_2 = 2.5 \sin^2 \theta$ for $\sin^2 \theta < 0.4$, = 1 otherwise. This weighting scheme was effective in ensuring that there was no obvious systematic dependence of the weighted

Table 1. Final atomic coordinates (with e.s.d.'s) ($\times 10^3$ for hydrogen atoms, $\times 10^4$ otherwise)

	x	y	z
C (1)	5058 (3)	1299 (3)	944 (1)
C (2)	5514 (3)	-40 (3)	785 (1)
C (4)	1307 (3)	-2921 (3)	1069 (1)
C (5)	571 (3)	-1739 (3)	1219 (1)
C (7)	-568 (2)	1504 (3)	559 (1)
C (8)	-992 (2)	2655 (3)	864 (1)
C (10)	3794 (3)	3112 (3)	2497 (1)
C (11)	4511 (3)	2012 (3)	2285 (1)
N (1')	3431 (2)	-1591 (2)	883 (1)
C (2')	4799 (3)	-1532 (3)	788 (1)
C (3')	5544 (4)	-2860 (3)	717 (2)
C (4')	4881 (4)	-4224 (3)	764 (2)
C (5')	3508 (4)	-4284 (3)	894 (2)
C (6')	2792 (3)	-2923 (3)	946 (1)
C (1'')	3643 (2)	1400 (2)	1126 (1)
C (2'')	3646 (2)	1083 (2)	1746 (1)
C (3'')	2564 (3)	58 (3)	1803 (1)
C (4'')	1302 (2)	-235 (3)	1303 (1)
C (5'')	974 (2)	861 (2)	820 (1)
C (6'')	2223 (2)	1485 (2)	689 (1)
N (1'')	1360 (2)	3015 (2)	1693 (1)
C (2''')	-108 (2)	3381 (3)	1457 (1)
C (3''')	-777 (3)	4413 (3)	1767 (1)
C (4''')	80 (4)	5049 (3)	2317 (1)
C (5''')	1575 (3)	4636 (3)	2567 (1)
C (6''')	2196 (3)	3589 (3)	2242 (1)
H (1)	565 (3)	219 (3)	93 (1)
H (2)	647 (3)	-8 (3)	66 (1)
H (4)	82 (3)	-389 (4)	99 (1)
H (5)	-47 (3)	-186 (4)	125 (1)
H (7)	-128 (3)	106 (3)	17 (1)
H (8)	-198 (3)	310 (3)	68 (1)
H (10)	433 (3)	371 (3)	285 (1)
H (11)	559 (3)	187 (3)	250 (1)
H (3')	651 (4)	-280 (4)	62 (2)
H (4')	540 (4)	-514 (5)	70 (2)
H (5')	301 (3)	-527 (4)	94 (1)
H (3'')	268 (3)	-49 (3)	219 (1)
H (6'')	212 (3)	199 (3)	28 (1)
H (3''')	-183 (3)	470 (3)	158 (1)
H (4''')	-32 (3)	575 (4)	254 (1)
H (5''')	223 (4)	502 (4)	296 (2)

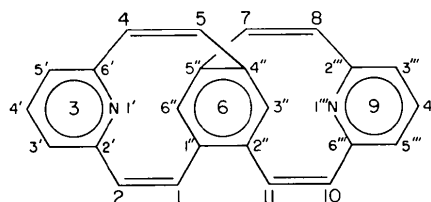
residual on either F_o or θ . On termination of refinement the largest coordinate shift of any non-hydrogen atom was 0.001 Å, or less than 40% of the corresponding e.s.d. The final ΔF synthesis revealed no electron density outside the limits $\pm 0.2 e \text{ \AA}^{-3}$. The final R index (for observed reflexions only) was 0.049, and R_w had the same value. The final atomic coordinates are given in Table 1.*

The computer programs used were those of Ahmed, Hall, Pippy & Huber (1966). Scattering factors were taken from Stewart, Davidson & Simpson (1965) for H, and from Hanson, Herman, Lea & Skillman (1964).

Discussion. The title compound (I) was prepared by I. D. Reingold (1976). It is one of a number of cyclo-

* The following material has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 32609 (19 pp.): atomic thermal-motion parameters (anisotropic for C and N); bond lengths and angles; details of some molecular planes and bond torsion angles; structure factors. Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

phane derivatives designed to force various chemical groupings into close proximity to the π -electron systems of benzene rings (Boekelheide, Galuszko & Szeto, 1974).



(I)

The molecule is found to have non-crystallographic but fairly precise twofold rotation symmetry about an axis through the non-substituted atoms of the benzene nucleus (Fig. 1). It does not deviate greatly from an idealized model having the symmetry 222, with the pyridine rings perpendicular to the benzene nucleus. In the observed structure the essentially planar pyridine rings are bent away from their ideal positions by about 15°. Each N atom lies 2.52 Å from the mean plane of the benzene nucleus and, thus, 5.04 Å from the other N atom. The N...N vector is inclined at 1.75° to the normal to the benzene plane, and passes about 0.10 Å from the centroid of the ring.

The bond lengths and angles given in Fig. 1 have been averaged as though the molecule had 222 symmetry. The greatest observed difference between bond distances related in this way is quite insignificant ($\Delta l = 0.016 \text{ \AA}$, $t = 2.1$). For bond angles, some differences are statistically significant, and doubtless real, but chemically unremarkable ($\Delta\theta = 1.6^\circ$, $t = 5.7$).

There is considerable evidence of strain in the molecular geometry. The benzene nucleus is distorted to a twist-boat conformation. The torsion angles of the bonds [starting with C(1'')-C(2'')] are 38, -19, -19, 38, -19, -19°. (The bonds between adjacent substituted atoms are significantly longer than the

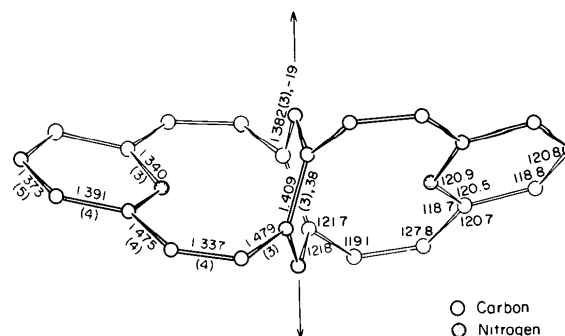


Fig. 1. A view of the molecule, showing mean bond lengths and angles (with e.s.d.'s) and, for the benzene ring, torsion angles. Internal angles of the benzene ring are not shown. They are 115.3 (2)° at substituted atoms, 120.5 (2)° otherwise.

others: $\Delta l = 0.027 \text{ \AA}$, $t = 6.4$.) The bonds to each substituted atom of the benzene nucleus deviate from coplanarity; the substituted atom in each case lies 0.08 to 0.10 Å from the plane of the bonded neighbouring atoms. The bond angles adjacent to the double bonds are markedly unequal. A corresponding inequality is also observed in the similar bridging systems of [2.2]metaparacyclophane-1,9-diene (Hanson, 1971), and [2.2](2,6)pyridinoparacyclophane-1,9-diene (Weaver & Matthews, 1974). In contrast to the angles, all bond lengths are close to their typical values, and show no evidence of strain. Intermolecular distances in the structure are consistent with van der Waals interactions.

The author is grateful to Professor V. Boekelheide for suggesting this problem and for supplying the specimen material.

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A Neutron Diffraction Refinement of *N*-(2-Chloroethyl)-D-gluconamide

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Abstract. C₈H₁₆ClNO₆, triclinic, space group *P*1, $a = 4.807$, $b = 5.093$, $c = 11.888 \text{ \AA}$, $\alpha = 91.25$, $\beta = 84.48$, $\gamma = 94.48^\circ$, $Z = 1$. The linear molecule contains one intramolecular hydrogen bond, N—H···O; the N atom also acts as a donor to the carbonyl O atom of the adjacent molecule, related by the translation along *b*, to form a bifurcated hydrogen bond.

Introduction. The structure of *N*-(2-chloroethyl)-D-gluconamide (I) determined by X-ray diffraction (Satzke & Mackay, 1975) has established the conformation of alkyl glyconamides. The molecule was found to be linear with the extended structure maintained in the crystal by intermolecular hydrogen bonding. Although H atoms bonded to C were located on difference maps, those bonded to N and O could not be

found. This neutron analysis was undertaken to determine accurate atomic coordinates for these six H atoms, and to provide a precise description of the hydrogen bonds.

The crystal was grown from methanol solution by slow evaporation in a desiccator over sulphuric acid, and was an irregular plate of volume 4.6 mm³. It was mounted with [001] along the ϕ axis of a four-circle automatic diffractometer installed at the HIFAR reactor of the Australian Atomic Energy Commission at Lucas Heights, NSW. A set of non-equivalent intensities to a maximum 2θ of 95° were recorded with a neutron beam of wavelength $\lambda = 1.2486 \text{ \AA}$, and a step-scanning technique in the ω - 2θ mode (Elcombe, Cox, Pryor & Moore, 1971). Of the 1000 terms measured, 961 had values significantly greater than background and were considered observed. A reference reflexion, 104, was monitored every 25 reflexions and no significant variation in its intensity was noted. The intensities were corrected for Lorentz and absorption effects. The linear absorption coefficient, $\mu = 2.63 \text{ cm}^{-1}$, was calculated with an incoherent scattering cross-section for H of 40 barns, and values of σ (total scattering plus true absorption) for C, Cl, N, O

