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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References

Bertz, S. H., Rihs, G. & Woodward, R. B. (1982). *Tetrahedron*, **38**, 63–70.

Chananont, P. & Hamor, T. A. (1981). *Acta Cryst.* B**37**, 1878–1881.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press.

Main, P., Fiske, S. J., Hull, S. E., Lessinger, L., Germain, G., Declercq, J. P. & Woolfson, M. M. (1980). *MULTAN* 80. *A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.

Martínez-Ripoll, M. & Cano, F. H. (1975). *PESOS. A Computer Program for the Automatic Treatment of Weighting Schemes.* Instituto 'Rocasolano', Madrid, Spain.

Nardelli, M. (1980). *PARST7* and *PLALIN*. Instituto di Chimica Generale, Univ. of Parma, Italy.

Owston, P. G. & Rowe, J. M. (1962). *Acta Cryst.* **15**, 231–238.

Sabesan, M. N. & Venkatesan, K. (1971). *Acta Cryst.* B**27**, 986–993.

Stewart, J. M., Kundell, F. A. & Baldwin, J. C. (1970). The XRAY 70 system. Computer Science Center, Univ. of Maryland, College Park, Maryland.

Van Havere, W., Lenstra, A. T. H., Geise, H. J., Van den Berg, G. R. & Benschop, H. P. (1982). *Acta Cryst.* B**38**, 469–472.

Vega, R., López-Castro, A. & Márquez, R. (1981). *Acta Cryst.* B**37**, 1092–1095.

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

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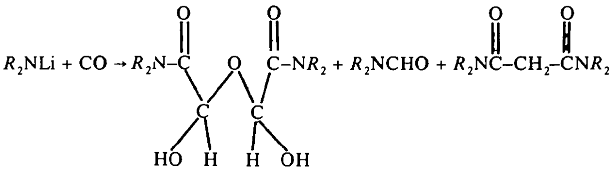
Abstract. $M_r = 501{\cdot}20$, monoclinic, $C2/c$, $a = 14{\cdot}506\,(4)$, $b = 15{\cdot}489\,(7)$, $c = 13{\cdot}875\,(2)$ Å, $\beta = 115{\cdot}90\,(2)^\circ$, $V = 2804\,(2)$ Å^3, $Z = 4$, $D_x = 1{\cdot}19$ Mg m^{-3}, Mo $K\alpha$ ($\lambda = 0{\cdot}71069$ Å), $\mu = 0{\cdot}76$ cm^{-1}, $F(000) = 1080$, room temperature, $R = 0{\cdot}050$ 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)

$R = C_6H_{11}$

Experimental. Prepared as above, recrystallized from hexane, m.p. 388–390 K; IR(Nujol) 3250, 1650, 1110, 1030, 900 cm^{-1}; ^{1}H NMR (100 mHz, $CDCl_3$) δ 5·60 (*d*, 2 exchangeable H), 5·30 (*d*, 2H), 3·76 (*m*, 2H), 2·96 (*m*, 2H), 2·35 (*m*, 4H), 1·85–0·85 (*m*, 36H); ^{13}C (100 mHz, $CDCl_3$) 166·58, 85·76, 56·30, 56·22, 30·88, 30·40, 29·40, 28·86, 26·32, 25·53, 25·28; mass spectrum *m/e* (%relative abundance) 238 (1·8), 208 (20·4), 126 (29·9), 83 (100); 0·7 × 0·6 × 0·6 mm, Syntex $P2_1$, θ:2θ scan, $2\theta_{\max} = 48°$, graphite-monochromated radiation; lattice parameters from least-squares refinement of 15 medium-angle reflections, angles measured by a centering routine associated with the diffractometer, systematic absences $h+k = 2n+1$ for *hkl*; monitored reflection showed no change in intensity greater than $2\sigma(I)$, 2297 independent reflections measured ($-15 \le h \le 15$, $0 \le k \le 17$, $0 \le l \le 16$), 1673 with $I > 3\sigma(I)$, Lorentz and polarization corrections, no absorption; direct methods (*MULTAN* 78: Main, Lessinger, Woolfson, Germain & Declercq, 1978) in *Cc* revealed all nonhydrogen atoms, *C*2/*c* selected as the most probable; hydrogen atoms from difference Fourier synthesis, included but not refined, full-matrix anisotropic least squares gave $R = 0{\cdot}050$, $\omega R = 0{\cdot}064$, $S = 2{\cdot}9$; $\sum\omega(|F_o| - |F_c|)^2$ minimized, $\omega = 1/\sigma^2(F_o)$, largest peak in final difference Fourier 0·3 eÅ^{-3}, average and maximum shift/error 0·04 and 0·20, atomic scattering factors from XRAY 76 (Stewart, Machin, Dickinson, Ammon, Heck & Flack, 1976). Locally written programs were used for data reduction and initial block-diagonal least-squares refinement, *MULTAN* 78 was used for the direct-methods calculations and XRAY 76 was used for all other computations.*

Discussion. Table 1 gives a listing of atomic positional parameters and Table 2 lists interatomic distances and valence angles.

Fig. 1 is an *ORTEP* drawing (Johnson, 1971) of compound (2). The C(4)—C(2)—O(1)—C(2′)—C(4′) fragment exhibits an extended planar conformation and makes an angle of 65 (1)° with the O(5)C(4)N(6)C(7)C(13) planar moiety. The nitrogen atom lies 0·050 (7) Å out of the plane of the three attached atoms and exhibits valence angles of 118·2 (2), 118·3 (2) and 123·1 (3)° which are consistent with a planar *sp*^{2} hybridization. The C(4)—N(6) bond distance is 1·355 (3) Å compared with the C(7)—N(6) and C(13)—N(6) distances of 1·480 (3) and 1·462 (3) Å, which is indicative of conjugation between the lone pair of the nitrogen and the carbonyl functionality. The cyclohexane rings exhibit chair conformations and planes defined by C(8)C(9)-C(11)C(12) and C(14)C(15)C(17)C(18) make an interplanar angle of 58·1 (7)°.

The O(3)C(2)C(4)O(5) grouping exhibits a torsion angle of −7·7 (5)° and an internal hydrogen bond is formed, O(3)—H(3) = 0·81 (3), O(3)···O(5) = 2·575 (3), H(3)···O(5) = 2·11 (3) Å and O(3)—H(3)···O(5) = 116 (3)°. This is consistent with the IR and ^{1}H NMR spectra which indicate strong hydrogen bonding in solution. An additional hydrogen bond is formed in the solid state between O(3) and O(5″) (0·5−*x*,0·5−*y*, 1−*z*) of an adjacent molecule, O(3)···O(5″) = 3·005 (2), H(3)···O(5″) = 2·24 (3) Å

Table 1. *Atomic positional parameters* (×10^4, *hydrogen atoms* ×10^3) *and U values* (Å^2 ×10^3)

	x	*y*	*z*	U_{eq} or U_{iso}*
O(1)	0	2606 (1)	2500	33
C(2)	914 (2)	2123 (1)	2834 (2)	34
O(3)	1134 (2)	1660 (1)	3763 (2)	51
C(4)	1732 (2)	2824 (2)	3055 (2)	35
O(5)	2351 (1)	2933 (1)	3979 (1)	47
N(6)	1681 (1)	3321 (1)	2229 (1)	34
C(7)	2315 (2)	4112 (2)	2478 (2)	37
C(8)	3458 (2)	3919 (2)	2948 (2)	49
C(9)	4082 (2)	4757 (2)	3196 (2)	58
C(10)	3823 (2)	5335 (2)	3919 (2)	61
C(11)	2699 (2)	5560 (2)	3405 (2)	58
C(12)	2051 (2)	4740 (2)	3158 (2)	46
C(13)	949 (2)	3166 (2)	1116 (2)	34
C(14)	1484 (2)	3032 (2)	387 (2)	45
C(15)	713 (2)	2811 (2)	−739 (2)	57
C(16)	−129 (2)	3490 (2)	−1199 (2)	60
C(17)	−641 (2)	3626 (2)	−457 (2)	50
C(18)	132 (2)	3858 (2)	670 (2)	41
H(2)	91 (2)	167 (2)	238 (2)	51†
H(3)	159 (2)	188 (2)	428 (2)	51

* $U_{eq} = \frac{1}{3}\sum_i\sum_j U_{ij}a_i^*a_j^*(\mathbf{a}_i.\mathbf{a}_j)$.
† Hydrogen atom U_{iso} ×10^3.

Table 2. *Interatomic distances* (Å) *and valence angles* (°)

O(1)—C(2)	1·413 (3)	C(8)—C(9)	1·533 (4)
C(2)—O(3)	1·385 (3)	C(9)—C(10)	1·508 (5)
C(2)—C(4)	1·538 (3)	C(10)—C(11)	1·509 (4)
C(4)—O(5)	1·214 (3)	C(11)—C(12)	1·527 (4)
C(4)—N(6)	1·355 (3)	C(13)—C(14)	1·535 (4)
N(6)—C(7)	1·480 (3)	C(13)—C(18)	1·516 (3)
N(6)—C(13)	1·462 (3)	C(14)—C(15)	1·511 (3)
C(7)—C(8)	1·522 (3)	C(15)—C(16)	1·524 (4)
C(7)—C(12)	1·516 (4)	C(16)—C(17)	1·526 (5)
		C(17)—C(18)	1·517 (3)
C(2)—O(1)—C(2′)	116·0 (2)	C(7)—C(8)—C(9)	110·8 (2)
O(1)—C(2)—O(3)	112·4 (2)	C(8)—C(9)—C(10)	110·9 (3)
O(1)—C(2)—C(4)	102·9 (2)	C(9)—C(10)—C(11)	110·8 (2)
O(3)—C(2)—C(4)	109·6 (2)	C(10)—C(11)—C(12)	110·2 (2)
C(2)—C(4)—O(5)	117·3 (2)	C(7)—C(12)—C(11)	112·0 (3)
C(2)—C(4)—N(6)	118·7 (2)	N(6)—C(13)—C(14)	112·1 (2)
O(5)—C(4)—N(6)	123·9 (2)	N(6)—C(13)—C(18)	113·1 (2)
C(4)—N(6)—C(7)	118·2 (2)	C(14)—C(13)—C(18)	110·9 (2)
C(4)—N(6)—C(13)	123·1 (2)	C(13)—C(14)—C(15)	110·7 (2)
C(7)—N(6)—C(13)	118·3 (2)	C(14)—C(15)—C(16)	111·9 (3)
N(6)—C(7)—C(8)	112·8 (2)	C(15)—C(16)—C(17)	110·5 (2)
N(6)—C(7)—C(12)	112·1 (2)	C(16)—C(17)—C(18)	111·7 (2)
C(8)—C(7)—C(12)	112·6 (2)	C(13)—C(18)—C(17)	110·3 (2)

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* Lists of structure factors, thermal parameters, torsion angles and unrefined H-atom coordinates have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38229 (20 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

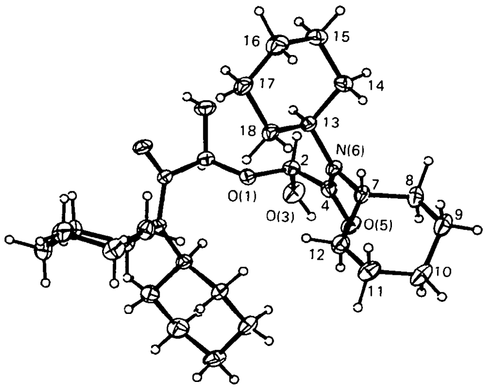

Fig. 1. *ORTEP* drawing of *N,N,N′,N′*-tetracyclohexyloxydiglycolamide hemihydrate. Thermal ellipsoids are drawn at the 35% probability level.

and O(3)—H(3)···O(5″) = 157 (3)°. H(3) may be considered as participating in an asymmetric bifurcated hydrogen bond.

In the solid state there are close intramolecular contacts between O(5)—H(12ax) and O(5)—H(13ax) of 2·515 (2) and 1·736 (2) Å, respectively. If the two cyclohexyl groups were equivalent in solution the ^{13}C spectrum should contain 8 resonances. Of the 11 lines observed the 166·58, 85·76, 56·30, and 56·22 p.p.m. resonances can be assigned to C(4), C(2) and the two cyclohexyl carbons attached to the nitrogen atoms. The remaining 7 resonances indicate there are two additional nonequivalent carbon atoms in each cyclohexyl group, which is consistent with the types of interactions observed in the solid state.

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References

Johnson, C. K. (1971). *ORTEP* II. Report ORNL-3794, revised. Oak Ridge National Laboratory, Tennessee.

Jutzi, P. & Schroder, F. W. (1971). *Angew. Chem. Int. Ed. Engl.* **10**, 339.

Longhi, P., Montagna, R. & Mazzochi, R. (1965). *Chim. Ind. (Milan)*, **47**, 480–486.

Main, P., Lessinger, L., Woolfson, M. M., Germain, G. & Declercq, J. P. (1978). *MULTAN 78. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.

Nudelman, N. S. & Outumuro, P. (1982). *J. Org. Chem.* In the press.

Nudelman, N. S. & Vitale, A. (1981). *J. Org. Chem.* **46**, 4625–4626.

Rautenstrauch, V. & Joyeaux, M. (1979). *Angew. Chem. Int. Ed. Engl.* **18**, 83–85.

Stewart, J. M, Machin, P. A., Dickinson, C., Ammon, H. L., Heck, H. & Flack, H. (1976). The XRAY 76 system. Tech. Rep. TR-446. Computer Science Center, Univ. of Maryland, USA.

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2,3,5,6-Tetramethyl-1,4-dioxane-2,5-diol, $C_8H_{16}O_4$

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Abstract. $M_r = 176·21$, triclinic, $P\bar{1}$, $a = 7·430$ (4), $b = 6·834$ (3), $c = 9·282$ (3) Å, $\alpha = 91·00$ (3), $\beta = 94·50$ (4), $\gamma = 97·14$ (4)°, $V = 466·0$ (4) Å^3, $Z = 2$, $D_x = 1·256$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $F(000) = 192$. Final $R = 0·041$ for 712 observed data. The molecules are centrosymmetric and packed in hydrogen-bonded layers parallel to the (011) plane.

Introduction. Crystals of the title compound, which is a dimer of 3-hydroxy-2-butanone, were formed inadvertently from the hydrolytic decomposition of 2,4,5-trimethyl-1,3,2-dioxaborole. The latter was prepared by heating 3-hydroxy-2-butanone and trimethylborane for 8 h (Longcor, 1983), and was purified by removal of volatiles on a vacuum line at 77 K followed by trap-to-trap distillation. After storage in a sealed container at 273 K for several weeks a crystalline product was observed. A crystallographic investigation of this product was undertaken in the expectation that it was a new, boron-containing compound. The identity of these crystals was proven by solution of the crystal structure. The title compound was subsequently shown to be a hydrolysis product of the borole.

Experimental. Crystal 0·3 × 0·2 × 0·05 mm, random orientation on a Nicolet *P*3 diffractometer, orientation matrix and unit-cell dimensions from 15 carefully centered reflections with $2\theta < 22°$; intensities measured

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