Crystallography (1974). The plot was made with *PLUTO* (Motherwell, 1976). Geometrical calculations were made with *PARST* (Nardelli, 1983). All calculations were made on a IBM 4341 computer.

Discussion. Final positional and thermal parameters are given in Table 1.* Molecular geometry data are collected in Table 2. Fig. 1 shows the nickel coordination and the atomic numbering scheme. The central nickel is pseudo square-planar coordinated by two ibn ligands. The Ni—N average distances, Ni—N(1) 1·907 (5) and Ni—N(2) 1·919 (5) Å, are the usual distances in square-planar coordination, similar to the 1·904 (9) and 1·915 (7) Å found for bis(2-methyl-1,2-propanediamine)nickel(II) diperchlorate (García-Granda *et al.*, 1987). This small difference in Ni—N distances, which is systematically found in both octahedral and square-planar complexes with ibn, appears as a clear effect of the *C*-substitution. The average N(1)—Ni—N(2) bite angle, 86·2 (2)°, is similar to 86·0 (4)° found in the above-mentioned complex. The ligands show an unusual, non-centrosymmetric disposition around the central nickel, being related by a pseudo-mirror plane, which has not been observed before for this ligand. An analysis of torsion angles shows that the distortion of the diamine ligand is similar to that found in the centrosymmetric complexes of this diamine. The average value of the torsion angle N—C—C—N is 47·0 (6)°, which is in good agreement with the values of 48·7 (6) and 46·0 (1)° found by García-Granda & Gómez-Beltrán (1984) in the centrosymmetric octahedral trichloroacetate complex and by García-Granda *et al.* (1987) in the square-planar perchlorate complex respectively.

* Lists of structure amplitudes, anisotropic thermal parameters, H-atom parameters, distances and angles involving H atoms, hydrogen-bond distances and angles, least-squares-planes data and principal torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52491 (25 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

García-Granda, S., Beurskens, P. T., Behm, H. J. J. & Gómez-Beltrán, F. (1987). *Acta Cryst.* C**43**, 236–238.

García-Granda, S. & Gómez-Beltrán, F. (1984). *Acta Cryst.* C**40**, 949–951.

Grant, D. F. & Gabe, E. J. (1978). *J. Appl. Cryst.* **11**, 114–120.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

Lehmann, M. S. & Larsen, F. K. (1974). *Acta Cryst.* A**30**, 580–584.

Motherwell, W. D. S. (1976). *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.* A**24**, 351–359.

Sheldrick, G. M. (1976). *SHELX*. Program for crystal structure determination. Univ. of Cambridge, England.

Sheldrick, G. M. (1985). *SHELXS86, Crystallographic Computing* 3, edited by G. M. Sheldrick, C. Krüger & R. Goddard, pp. 175–189. Oxford Univ. Press.

Walker, N. & Stuart, D. (1983). *Acta Cryst.* A**39**, 158–166.

Acta Cryst. (1990). C**46**, 600–604

Molecular Structures of Caracemide,* an Inhibitor of Ribonucleotide Reductase, and of one of its Degradation Products

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(*Received* 12 *May* 1989; *accepted* 11 *July* 1989)

Abstract. *N*-Acetyl-*N*,*O*-di(methylcarbamoyl)hydroxylamine, $C_6H_{11}N_3O_4$ (CAR), $M_r = 189{\cdot}18$, monoclinic, $P2_1/c$, $a = 5{\cdot}012$ (1), $b = 11{\cdot}989$ (2), $c = 14{\cdot}375$ (2) Å, $\beta = 93{\cdot}10$ (2)°, $V = 862{\cdot}5$ (2) Å^3 at 105 K, $Z = 4$, $D_x = 1{\cdot}457$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 0{\cdot}115$ mm^{-1}, $F(000) = 400$, $T = 105$ K. Final $R = 0{\cdot}034$ for 1351 unique observed reflections. *N*-Acetyl-*O*-methylcarbamoylhydroxylamine, $C_4H_8N_2O_3$ (DECAR), $M_r = 132{\cdot}12$, orthorhombic, *Pbca*, $a = 8{\cdot}975$ (2), $b = 9{\cdot}748$ (1), $c = 14{\cdot}2243$ (8) Å, $V = 1244{\cdot}4$ (5) Å^3 at 105 K, $Z = 8$, $D_x = 1{\cdot}410$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 0{\cdot}113$ mm^{-1}, $F(000) = 560$, $T = 105$ K. Final $R = 0{\cdot}034$ for 1837 unique observed reflections. Both molecules consist of two roughly planar parts with dihedral angles of 84·19 (4) and 74·87 (3)° between the planes, for CAR and DECAR, respectively. One

* *Chemical Abstracts* name: *N*-[(methylamino)carbonyl]-*N*-{[(methylamino)carbonyl]oxy}acetamide.

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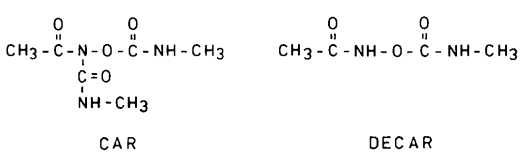
of the planar parts of CAR is stabilized by an intramolecular hydrogen bond, and in both crystal structures the molecules are connected by NH···O bonds in addition to van der Waals forces.

Introduction. Caracemide (CAR) is a new antitumour agent undergoing clinical trials (Newman *et al.*, 1986; Pazdur, Chabot & Baker, 1987; Raber, Adams, Kavanagh, Legha, Dimery & Krakoff, 1987). CAR has been shown to be an inhibitor of the enzyme ribonucleotide reductase from Novikoff ascites tumour cells, but it has been proposed that it inhibits the enzyme by a mechanism different from that of hydroxyurea (Newman *et al.*, 1986). Hydroxyurea and related compounds inhibit ribonucleotide reductase of *E. coli* by destroying the free-radical group present in the smaller subunit, protein B2, of the enzyme (Kjøller Larsen, Sjöberg & Thelander, 1985).



CAR has been shown to be rather unstable in aqueous solution, and the possibility that the cytotoxic effect of CAR is (partly) due to one of its degradation products has been considered (Newman *et al.*, 1986; Lee, Lin & Wang, 1986). The degradation product *N*-acetyl-*O*-methylcarbamoylhydroxylamine (DECAR) also inhibits ribonucleotide reductase of Novikoff ascites tumour cells, but the effect is only about a quarter of that of CAR (Newman *et al.*, 1986).

CAR and DECAR have recently been tested for inhibitory action on highly purified ribonucleotide reductase of *E. coli*, and have been shown to inhibit the enzyme by interacting specifically with the larger subunit protein B1, whereas no effect on the smaller subunit protein B2 was observed (Karlsson, 1989). These findings are in full agreement with the results of Newman *et al.* (1986), *e.g.* the lack of cross resistance of CAR with hydroxyurea, and the fact that CAR, in contrast to hydroxyurea, is not an *S*-phase specific agent. The effect on protein B1 is initiated immediately, and also in this enzyme system CAR is found to be much more inhibitive than its degradation product DECAR. The cytotoxic effect of CAR by inhibiting the enzyme ribonucleotide reductase therefore seems to be caused by CAR rather than by a degradation product. Further studies on the mode of interaction of CAR with protein B1 are in progress (by M. Karlsson).

CAR has some neurological and psychiatric side effects, which probably are due to inhibitory effects on other enzymes, *e.g.* acetylcholinesterase (Newman *et al.*, 1986; McKinney, Pfenning & Richelson, 1986), and choline acetyltransferase (Ho *et al.*, 1988).

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters* (Å^2) *with e.s.d.'s in parentheses*

$B_{eq} = (4/3)\sum_i\sum_j \beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$

	x	y	z	B_{eq}
CAR				
C1	1·0901 (3)	0·0957 (2)	0·6880 (1)	1·54 (3)
C2	0·9315 (3)	0·1004 (1)	0·5964 (1)	1·18 (3)
O2	0·7362 (2)	0·0421 (1)	0·57715 (8)	1·54 (2)
N3	1·0217 (3)	0·1754 (1)	0·53266 (9)	1·21 (2)
O4	1·2506 (2)	0·2357 (1)	0·55928 (8)	1·23 (2)
C5	1·1881 (3)	0·3452 (1)	0·5852 (1)	1·04 (3)
O5	0·9620 (2)	0·3751 (1)	0·59829 (9)	1·60 (2)
N6	1·4102 (3)	0·4044 (1)	0·5944 (1)	1·13 (2)
C7	1·4048 (3)	0·5207 (1)	0·6223 (1)	1·39 (3)
C8	0·9233 (3)	0·2011 (1)	0·4394 (1)	1·16 (3)
O8	1·0404 (2)	0·2696 (1)	0·39468 (8)	1·53 (2)
N9	0·7038 (3)	0·1451 (1)	0·4095 (1)	1·30 (2)
C10	0·5842 (3)	0·1691 (2)	0·3174 (1)	1·55 (3)
DECAR				
C1	0·8782 (1)	−0·05857 (9)	0·14158 (7)	1·45 (1)
C2	0·80074 (9)	0·05692 (8)	0·19088 (6)	1·06 (1)
O2	0·66989 (7)	0·08809 (7)	0·17553 (5)	1·34 (1)
N3	0·88531 (8)	0·12415 (8)	0·25373 (6)	1·45 (1)
O4	0·82518 (7)	0·23867 (7)	0·29846 (5)	1·39 (1)
C5	0·72063 (9)	0·20364 (8)	0·36696 (6)	1·10 (1)
O5	0·69832 (8)	0·08648 (6)	0·39217 (5)	1·41 (1)
N6	0·65312 (9)	0·31740 (7)	0·39633 (6)	1·39 (1)
C7	0·5299 (1)	0·31383 (9)	0·46279 (7)	1·53 (1)

Experimental. CAR and DECAR were prepared as described by Lee *et al.* (1986). Crystals were obtained by slow cooling; for CAR from a solution in methylene chloride and heptane, and for DECAR from a solution in ethanol. The crystals are colourless needles with *a* as the needle axis in both cases, m.p. 389–391 K for CAR and 385–387 K for DECAR. Crystals of CAR 0·08 × 0·15 × 0·50 mm and of DECAR 0·10 × 0·30 × 0·40 mm were chosen for the data collection on an Enraf–Nonius CAD-4 diffractometer equipped with graphite monochromator and Nonius low-temperature device. Temperature was kept constant within ±0·5 K from an estimated value of 105 (5) K. Cell dimensions were determined by least-squares fit of angular settings for 18 reflections (θ range 15·39–19·17°) in the case of CAR, and for 20 reflections (θ range 16·92–20·44°) in the case of DECAR. The intensities were measured by the ω–2θ scan method for $\theta \le 28°$ for CAR ($0 \le h \le 6$, $0 \le k \le 15$, $-18 \le l \le 18$) and for $\theta \le 35°$ for DECAR ($0 \le h \le 14$, $-15 \le k \le 15$, $0 \le l \le 22$). Three standard reflections measured every 100 reflections showed no significant variations. Intensities of 2425 reflections were measured for CAR, 2085 being unique ($R_{int} = 0·054$). Of these 1351 reflections with $I \ge 3·0\sigma(I)$ were considered observed. Intensities for 3542 reflections were measured for DECAR, 2736 being unique ($R_{int} = 0·016$). Of these 1837

reflections with $I \geq 3{\cdot}0\sigma(I)$ were considered observed. No absorption corrections were made. The structures were solved by direct methods using *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980). Full-matrix least-squares refinements of positional parameters for all atoms, with anisotropic temperature factors for non-H atoms and isotropic temperature factors for H-atoms. All H atoms were located in difference Fourier maps. The quantity minimized is $\sum w(|F_o| - k|F_c|)^2$, where $w = 4F^2[\sigma^2(F^2) + (pF^2)^2]^{-1}$ and $p =$ 0·05 for CAR and 0·06 for DECAR. The average and maximum values of Δ/σ in final refinement cycles are 0·01 and 0·03 for CAR and 0·02 and 0·16 for DECAR. The final error indicators are $R =$ 0·034, $wR = 0{\cdot}041$ for CAR, and $R = 0{\cdot}034$, $wR =$ 0·044 for DECAR. The fluctuations in the final difference syntheses are $\pm 0{\cdot}3$ e Å^{-3} in both structures. Scattering factors for the atoms as implemented in the *SDP* program package (Frenz, 1982), which was used for all calculations.

Discussion. The final atomic parameters of CAR and DECAR are listed in Table 1.* Bond lengths and angles are given in Table 2. The atomic numbering schemes and molecular conformations are shown in Fig. 1. The molecular packing arrangements are shown in Fig. 2.

The bond lengths and angles of CAR and DECAR seem to be quite normal, and good agreement is found between corresponding bonds and angles of the two compounds (*cf.* Table 2). The intramolecular distance O5···O8 in CAR is 3·232 (2) Å and the corresponding distance O5···O2 of DECAR is 3·092 (1) Å.

CAR. The molecule is composed of two nearly planar parts almost perpendicular to each other. The first plane (plane 1) is defined by C1, C2, O2, N3, O4, C8, O8, N9 and C10 with average deviation from the least-squares plane of 0·014 (2) Å. The second plane (plane 2) is defined by N3, O4, C5, O5, N6 and C7 with average deviation of 0·055 (2) Å. The dihedral angles between the two planes is 84·19 (4)°. The amide H atoms, H9 and H6, are located close to the planes defined above, the distances to the planes being 0·04 (2) and 0·00 (2) Å, respectively. One of the H atoms of the C1 and C10 methyl groups is also near to plane 1, the distances being 0·14 (2) and 0·03 (2) Å for H11 and H101, respectively.

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52407 (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), *bond angles* (°), *selected torsion angles* (°), *and hydrogen-bond geometry* (Å,°) *with e.s.d.'s in parentheses*

CAR

C1—C2	1·502 (2)	C5—N6	1·321 (2)
C2—O2	1·223 (2)	N6—C7	1·452 (2)
C2—N3	1·378 (2)	N3—C8	1·437 (2)
N3—O4	1·392 (2)	C8—O8	1·213 (2)
O4—C5	1·404 (2)	C8—N9	1·339 (2)
C5—O5	1·213 (2)	N9—C10	1·453 (2)

C1—C2—N3	115·5 (1)	O4—C5—N6	109·3 (1)
C1—C2—O2	123·8 (2)	O5—C5—N6	127·9 (2)
O2—C2—N3	120·7 (1)	C5—N6—C7	121·0 (1)
C2—N3—O4	116·8 (1)	N3—C8—O8	119·2 (1)
C2—N3—C8	130·7 (1)	N3—C8—N9	115·2 (1)
O4—N3—C8	112·4 (1)	O8—C8—N9	125·6 (2)
N3—O4—C5	111·5 (1)	C8—N9—C10	118·9 (1)
O4—C5—O5	122·8 (1)		

C1—C2—N3—O4	1·5 (2)	C1—C2—N3—C8	179·2 (2)
C2—N3—O4—C5	−102·7 (2)	C2—N3—C8—N9	1·8 (2)
N3—O4—C5—O5	12·2 (2)	N3—C8—N9—C10	177·5 (1)
N3—O4—C5—N6	−169·4 (1)	C8—N3—O4—C5	79·1 (2)
O4—C5—N6—C7	−179·0 (1)		

Hydrogen bonding

A—H···*B*	*A*···*B*	H···*B*	∠*A*—H···*B*	Symmetry code for *B*
N9—H9···O2	2·705 (2)	2·01 (2)	132 (2)	*x*, *y*, *z*
N9—H9···O2	3·160 (2)	2·48 (2)	132 (2)	1−*x*, −*y*, 1−*z*
N6—H6···O5	2·785 (2)	2·01 (2)	145 (2)	*x*+1, *y*, *z*

DECAR

C1—C2	1·497 (1)	O4—C5	1·395 (1)
C2—O2	1·233 (1)	C5—O5	1·214 (1)
C2—N3	1·343 (1)	C5—N6	1·331 (1)
N3—O4	1·394 (1)	N6—C7	1·455 (1)

C1—C2—N3	114·60 (7)	O4—C5—N6	108·75 (7)
C1—C2—O2	122·99 (8)	O4—C5—O5	123·20 (7)
O2—C2—N3	122·40 (8)	O5—C5—N6	128·04 (8)
C2—N3—O4	118·41 (7)	C5—N6—C7	122·00 (7)
N3—O4—C5	112·53 (6)		

C1—C2—N3—O4	175·93 (7)	N3—O4—C5—N6	−170·98 (7)
C2—N3—O4—C5	74·51 (10)	O4—C5—N6—C7	175·23 (8)
N3—O4—C5—O5	8·25 (11)		

Hydrogen bonding

A—H···*B*	*A*···*B*	H···*B*	∠*A*—H···*B*	Symmetry code for *B*
N3—H3···O2	2·767 (1)	1·92 (2)	170 (1)	*x*−0·5, *y*, 0·5−*z*
N6—H6···O5	2·943 (1)	2·07 (2)	156 (1)	0·5−*x*, 0·5+*y*, *z*

Hydroxyurea analogs have to be nearly planar molecules with a functional group which can react with the free-radical group of protein B2, in order to be potent inhibitors of ribonucleotide reductase (Kjøller Larsen *et al.*, 1982). CAR does not fulfil these requirements in accordance with the fact that the inhibitory mechanism of CAR is different (see *Introduction*).

The molecular conformation of CAR is stabilized by an intramolecular H bond, N9—H9···O2 (see Table 2). H9 also approaches O2 of a neighbouring molecule at a distance of 2·48 Å (Table 2 and Fig. 2). The crystal structure of CAR is further stabilized by

the hydrogen bond N6—H6···O5, which connects the molecules along **a**, forming infinite rows of dimers.

DECAR. This molecule also consists of two nearly planar parts, in this case with a dihedral angle of 74·87 (3)° between the two planes. Plane 1 is defined by C1, C2, O2, N3 and O4 with an average deviation from the least-squares plane of 0·019 (1) Å. Plane 2 is defined by O4, C5, O5, N6 and C7 with an average deviation of 0·020 (1) Å. The H atoms H11 and H3 are situated near plane 1, the deviations being 0·09 (1) and −0·05 (1) Å, respectively. The amide H atom H6 is situated 0·17 (2) Å from plane 2.

The conformation of N—O—C=O (N3—O4—C5—O5) is *sp* (synperiplanar) in both DECAR and CAR (*cf.* Table 2), whereas an *ap* (antiperiplanar) conformation was found about the corresponding O—C bond of *O*-carbamoylhydroxylamine, $H_2NO(OC)NH_2$ (Larsen, 1968). The conformation of DECAR differs from that of CAR by the fact that C1—C2—N3—O4 has an *ap* conformation in DECAR and an *sp* conformation in CAR. The overall shapes of the two molecules are still similar, as the atom sequence C1 to C7 of DECAR is roughly superimposable with the sequences N9 to N3 and N3 to C7 of CAR (see Fig. 3). DECAR inhibits ribonucleotide reductase in the same way as CAR, *i.e.* by interaction with protein B1. Whether the conformations observed in the crystalline state are the 'active conformations' of the molecules is of course an open question.

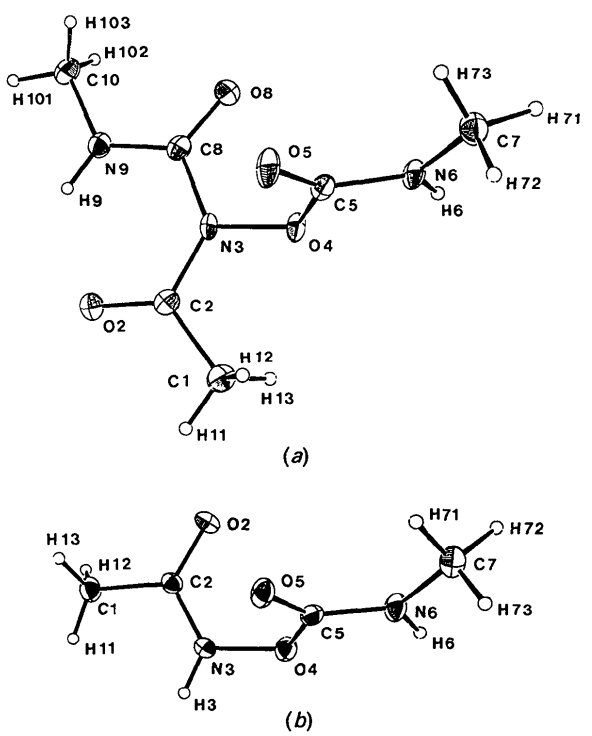


Fig. 1. Molecular structures of (*a*) CAR and (*b*) DECAR (Johnson, 1976) with thermal ellipsoids at the 50% probability level for non-H atoms.

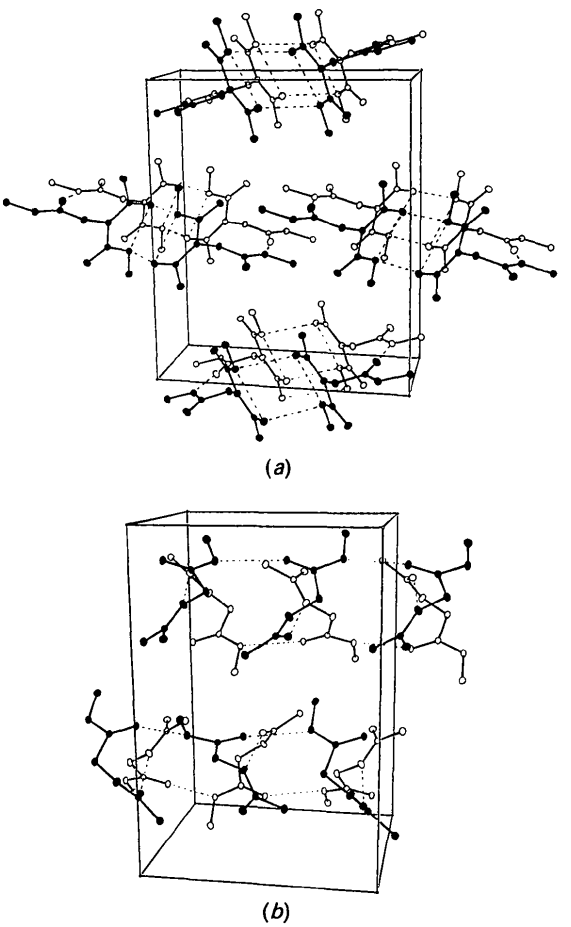


Fig. 2. Crystal packing diagrams for (*a*) CAR and (*b*) DECAR (*b* horizontal and *c* vertical in both cases). Hydrogen bonds are indicated by dashed lines.

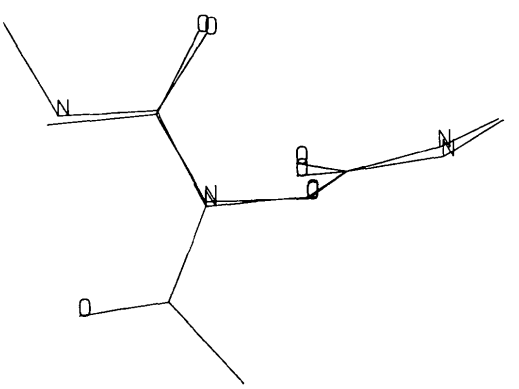


Fig. 3. The molecular skeletons of CAR and DECAR superimposed, using the program *SYBYL* (Tripos Associates Inc., St. Louis, Missouri, USA).

The crystal structure of DECAR is stabilized by two hydrogen bonds, N3—H3···O2 and N6—H6···O5, which connect the molecules along **a** and **b**, respectively (Table 2 and Fig. 2). No other short intermolecular distances were found.

The author thanks Mr Flemming Hansen for collecting the X-ray data. The diffractometer and an X-ray generator were acquired by means of grants 11-1837, 11-2360 and 11-3531 from the Danish Natural Science Research Council.

References

FRENZ, B. A. (1982). *SDP Users Guide*. Enraf–Nonius, Delft, The Netherlands.

HO, B. T., TANSEY, L. W., FEIFFER, R. NEWMAN, R. A., FARQUHAR, D., FIELDS, W. S. & KRAKOFF, I. H. (1988). *J. Neurosci. Res.* **19**, 119–121.

JOHNSON, C. K. (1976). *ORTEPII*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

KARLSSON, M. (1989). Private communication.

KJØLLER LARSEN, I., SJÖBERG, B.-M. & THELANDER, L. (1982). *Eur. J. Biochem.* **125**, 75–81.

LARSEN, I. K. (1968). *Acta Chem. Scand.* **22**, 843–853.

LEE, M.-S., LIN, D. P. & WANG, C. Y. (1986). *Mutat. Res.* **172**, 199–209.

MCKINNEY, M., PFENNING, M. & RICHELSON, E. (1986). *Biochem. Pharmacol.* **35**, 2615–2622.

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.

NEWMAN, R. A., FARQUHAR, D., LU, K., MEYN, R., MOORE, E. C., MASSIA, S., KORP, J. D., WRIGHT, J. A. & MCKINNEY, M. (1986). *Biochem. Pharmacol.* **35**, 2781–2787.

PAZDUR, R., CHABOT, G. G. & BAKER, L. H. (1987). *Invest. New Drugs*, **5**, 365–371.

RABER, M. N., ADAMS, F., KAVANAGH, J., LEGHA, S., DIMERY, I. & KRAKOFF, I. (1987). *Cancer Treat. Rep.* **71** 349–352.

Acta Cryst. (1990). C**46**, 604–606

Structure of Galactitol Hexa(*p*-chlorobenzoate)

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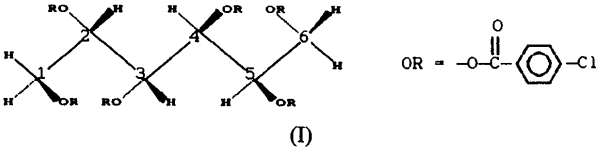
(*Received* 28 *March* 1989; *accepted* 13 *June* 1989)

Abstract. Galactitol hexa(*p*-chlorobenzoate) (I), $C_{48}H_{32}Cl_6O_{12}$, $M_r = 1013{\cdot}492$, monoclinic, $P2_1/n$, $a = 7{\cdot}863$ (2), $b = 23{\cdot}880$ (7), $c = 12{\cdot}345$ (5) Å, $\beta = 96{\cdot}97$ (3)°, $V = 2300$ (1) Å^3, $D_x = 1{\cdot}46$ g cm^{-3} for $Z = 2$, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 4{\cdot}4$ cm^{-1}, $F(000) = 1036$, $T = 295$ K, $R = 0{\cdot}047$ for 1267 unique observed reflections with $|F_o| > 3\sigma(|F_o|)$ and 298 refined parameters. The molecule, which is a *meso* form, has crystallographically induced $\bar{1}$ symmetry. It consists of a planar zigzag carbon chain with terminal *p*-chlorobenzoate groups in *gauche* conformation. The orientation of terminal groups is different from that observed in the parent molecule, galactitol.

Introduction. There has been considerable interest in the stereochemistry of acyclic polyol molecules and their derivatives. Solid state conformations have been extensively studied for many years by X-ray crystallography. Application of CD spectroscopy for studying conformations of *p*-chlorobenzoylated polyols in solution has stimulated our X-ray investigations of this group of polyol derivatives (Gawroński & Gawrońska, 1990). We hope to be able to establish some rules which govern the stereostructure of *p*-chlorobenzoylated polyols. The first in a series is the hexa(*p*-chlorobenzoate) derivative of galactitol. Although this particular compound, being a *meso* form, cannot be used in chiroptical studies, it still serves as a model for deriving contributions to the Cotton effects from the *p*-chlorobenzoate chromophores in various positions and orientations.



(I)

Experimental. Single crystals suitable for X-ray study were grown by slow evaporation from dioxane solutions; crystal *ca* 0·3 × 0·4 × 0·5 mm was selected for data collection. Syntex $P2_1$ diffractometer with graphite-monochromated Mo $K\alpha$ radiation. Accurate cell constants were from setting angles of 15 reflections; ω–2θ scans, variable scan speed. Two standard reflections remeasured every 100 reflections showed no change in intensity greater than $3{\cdot}6\sigma(I)$. 3128 reflections measured, $\theta \leq 22{\cdot}5°$, only 1267 observed $[|F_o| > 3\sigma(|F_o|)]$ due to poor scattering

0108-2701/90/040604-03$03.00