values of a chair–boat–chair conformation may be due to the large side groups attached at these sites, there is a striking resemblance between the torsion-angle values for inuolide and scorpioidine (Drew, Hitchman, Mann & Lopes, 1980) where there are no large side groups attached to the corresponding sites. The torsion angles are also comparable to those of 1,3,5,7-tetraoxa-9-aza-8-cyclodecanone (Kobelt & Paulus, 1973), epoxyineupatorolide and ineupatolide (Baruah, Sharma, Thyagarajan, Herz, Govindan & Blount, 1980).

The $\gamma$-lactone ring is *trans*-fused to the ten-membered ring at C(7) and C(8) and assumes an envelope form with C(8) as the flap, about 0·4 Å away from the plane of the other atoms of the lactone ring.

We thank Dr John F. Blount for kindly sending us the data from his paper. We are also thankful to Drs L. M. Pant and T. N. Guru Row for useful discussions during the course of this work. We thank Dr A. P. B. Sinha, Head, Physical Chemistry Division, for encouragement during the course of this work.

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*Acta Cryst.* (1983). C**39**, 464–466

# Structure of Methyl 5,6,7,8,9-Pentaacetoxy-2-amino-4-nitromethyl-D-*glycero*-L-*gluco*-2-nonene-3-carboxylate, $C_{22}H_{32}N_2O_{14}$

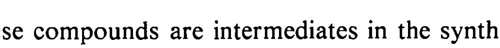
By R. Vega, A. López-Castro and R. Márquez

*Departamento de Optica y Sección de Física del Departamento de Investigaciones Físicas y Químicas del CSIC, Universidad de Sevilla, Spain*

(*Received* 13 *July* 1982; *accepted* 26 *October* 1982)

**Abstract.** $M_r = 548{\cdot}5$, orthorhombic, $P2_12_12_1$, $a = 14{\cdot}270\,(2)$, $b = 18{\cdot}743\,(2)$, $c = 10{\cdot}309\,(1)$ Å, $V = 2757{\cdot}3\,(6)$ Å$^3$, $Z = 4$, $D_x = 1{\cdot}32$, $D_m = 1{\cdot}30$ Mg m$^{-3}$, $\lambda(\text{Cu } K\alpha) = 1{\cdot}542$ Å, $\mu = 0{\cdot}95$ mm$^{-1}$, $F(000) = 1160$, $T = 300$ K. Final $R = 0{\cdot}074$ for 1848 observed reflections. The configurations of the chiral centres C(8), C(7), C(6), C(5) and C(4) are *R*, *S*, *R*, *S* and *S*, respectively, that is D-*glycero*-L-*gluco*. The molecule has an intramolecular hydrogen bond between the N and O atoms of the amino and carbonyl groups respectively, showing a chelate structure. Packing of the molecules is governed by van der Waals contacts.

**Introduction.** Open-chain sugar nitro-olefins [polyacetoxy-1-nitroalk-1-enes, $No_2.HC{=}CH.(CHOAc)_4.CH_2OAc$ and O-protected 2-*C*-glycosyl-1-nitroethylenes, $R.NH.C(CH_3){=}CH.O_2R'$] are attractive as potential starting materials for the synthesis of functionalized, branched-chain sugars and *C*-(polyhydroxyalkyl) and *C*-glycosylheterocyclic compounds derived from them. Gómez-Sánchez, Mancera, Rosado & Bellanato (1980) reported an easy way to obtain 3-(pentaacetoxypentyl)pyrrole derivatives by Michael-type addition of 3-(alkylamino)crotonic esters to pentaacetoxy-1-nitrohept-1-enes and further cyclization of the adducts.



$$\begin{array}{l} R.NH.C(CH_3){=}\underset{\displaystyle |}{C}.COOR' \\ \qquad\quad NO_2.CH_2.C^{*}H.(CHOAc)_4.CH_2OAc \end{array}$$

These compounds are intermediates in the synthesis of 3-(pentaacetoxypentyl)pyrroles, substances which have aroused interest as precursors of *C*-glycosylpyrroles. A new chiral centre is found (marked with an asterisk) and two diastereoisomers could result, although only one has been isolated. The configuration of these derivatives could not be established from chemical and spectroscopic considerations.

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The crystal structure of the title compound ($R$=H, $R'$=$CH_3$) has been determined as a systematic structural investigation of the conformational aspects of this type of compound; one of these ($R$=$C_4H_9$, $R'$=$C_2H_5$) has already been studied (Vega, López-Castro & Márquez, 1981).

**Experimental.** Grey elongated prisms (from ethanol), 0·7 × 0·4 × 0·3 mm, Enraf–Nonius CAD-4 diffractometer, Ni-filtered Cu $K\alpha$, 2630 independent reflections with $\theta<65°$, 1848 with $I>2\sigma(I)$, Lp correction, absorption ignored; direct methods (*MULTAN*, Main, Lessinger, Woolfson, Germain & Declercq, 1977), anisotropic full matrix, H(calculated) isotropic, final $R = R_w = 0{\cdot}074$, $w = 1/\sigma^2(F)$, DCT 2000 terminal of the Centro de Cálculo of Seville University, connected to a Univac 1108 computer; atomic scattering factors taken from *International Tables for X-ray Crystallography* (1974), XRAY system (Stewart, Kundell & Baldwin, 1970) computer programs used.

**Discussion.** The positional and isotropic thermal parameters for non-H atoms are given in Table 1.* Fig. 1. shows a schematic view of the molecule with the atom numbers, bond distances and angles for non-H atoms.

As observed in other analogous compounds (Vega *et al.*, 1981), the main nonene chain has an approximately planar zigzag part comprising C(4), C(5), C(6), C(7), C(8) and C(9) with deviations <0·10 Å from their best plane and a second part, completely planar, involving C(1), C(2), C(3) and C(4) with a largest deviation of 0·001 Å, the two chain sections being joined by C(3)—C(4) (torsion angle −135·6°). The five acetoxy, nitromethyl and methoxycarbonyl groups are planar with deviations less than 0·011, 0·002 and 0·004 Å from the corresponding best plane.

The torsion angles of the acetoxy groups with respect the nonene chain are: C(9)—C(8)—O(81)—C(81) = −91·3 (10), C(8)—C(7)—O(71)—C(71) = 111·2 (10), C(7)—C(6)—O(61)—C(61) = 114·9 (9), C(6)—C(5)—O(51)—C(51) = −124·0 (9) and C(8)—C(9)—O(91)—C(91) = 171·2 (8)°. The methoxycarbonyl and nitromethyl groups are twisted from the chain, the torsion angles being: C(4)—C(3)—C(31)—O(31) = 8·7 (13), C(4)—C(3)—C(31)—O(32) = −174·9 (9) and C(5)—C(4)—C(41)—N(41) = 86·4 (9)°.

The Newman projections corresponding to C—C bonds of the main nonene chain are shown in Fig. 2 where the configuration around the chiral centres can be deduced. According to Klyne & Prelog (1960) rules the conformation is as follows. In the projection along C(2)=C(3), C(1) and N(21) are nearly eclipsed by C(4) and C(31) with C(4) +synperiplanar to C(1) and +antiperiplanar to N(21). With respect to C(3)—C(4), C(2) is eclipsed by HC(4) with C(5) +synclinal to C(31) and −anticlinal to C(2). These peculiarities, as observed in analogous compounds, are consequences of

Table 1. *Final positional coordinates* (×10⁴) *and equivalent isotropic temperature factors for non*-H *atoms* (×10³)

$$U_{\rm eq} = \tfrac{1}{3}\textstyle\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i . \mathbf{a}_j .$$

| | $x$ | $y$ | $z$ | $U_{\rm eq}$(Å$^2$) |
|---|---|---|---|---|
| C(1) | −700 (11) | 4965 (6) | 2562 (14) | 66 |
| C(2) | 210 (9) | 4554 (6) | 2503 (11) | 48 |
| C(3) | 233 (7) | 3819 (5) | 2708 (10) | 35 |
| C(4) | −667 (7) | 3409 (5) | 2990 (9) | 30 |
| C(5) | −550 (6) | 2868 (5) | 4114 (8) | 25 |
| C(6) | −308 (7) | 3224 (5) | 5393 (8) | 25 |
| C(7) | −199 (7) | 2653 (5) | 6513 (9) | 32 |
| C(8) | −73 (7) | 2992 (5) | 7836 (9) | 35 |
| C(9) | 206 (7) | 2440 (6) | 8861 (10) | 43 |
| C(31) | 1096 (8) | 3436 (6) | 2456 (10) | 44 |
| C(32) | 1854 (9) | 2292 (8) | 2002 (16) | 72 |
| C(41) | −979 (7) | 3025 (6) | 1716 (9) | 39 |
| C(51) | −1523 (8) | 1799 (5) | 4038 (11) | 44 |
| C(52) | −2445 (9) | 1498 (6) | 4423 (13) | 57 |
| C(61) | −821 (9) | 4404 (5) | 6018 (11) | 53 |
| C(62) | −1647 (12) | 4820 (5) | 6453 (16) | 82 |
| C(71) | 551 (10) | 1536 (6) | 6046 (13) | 66 |
| C(72) | 1436 (12) | 1209 (7) | 5413 (16) | 82 |
| C(81) | 512 (9) | 4104 (6) | 8602 (12) | 56 |
| C(82) | 1317 (11) | 4634 (7) | 8482 (16) | 74 |
| C(91) | −486 (9) | 1404 (6) | 9772 (11) | 52 |
| C(92) | −1371 (10) | 955 (7) | 9809 (15) | 66 |
| O(31) | 1002 (5) | 2719 (4) | 2318 (8) | 47 |
| O(32) | 1885 (5) | 3727 (5) | 2302 (10) | 64 |
| O(41) | −2524 (7) | 3491 (6) | 1880 (11) | 81 |
| O(42) | −2395 (7) | 2369 (6) | 1457 (10) | 76 |
| O(51) | −1461 (5) | 2515 (3) | 4289 (7) | 34 |
| O(52) | −872 (7) | 1476 (5) | 3564 (10) | 70 |
| O(61) | −1066 (5) | 3705 (4) | 5783 (7) | 37 |
| O(62) | −54 (7) | 4623 (5) | 5862 (10) | 72 |
| O(71) | 623 (5) | 2260 (4) | 6111 (7) | 41 |
| O(72) | −108 (9) | 1248 (5) | 6457 (12) | 92 |
| O(81) | 650 (5) | 3537 (4) | 7810 (7) | 38 |
| O(82) | −187 (7) | 4163 (5) | 9266 (10) | 75 |
| O(91) | −605 (5) | 2000 (4) | 9032 (7) | 47 |
| O(92) | 232 (7) | 1271 (5) | 10291 (11) | 81 |
| N(21) | 997 (8) | 4936 (5) | 2260 (11) | 63 |
| N(41) | −2062 (7) | 2954 (6) | 1683 (9) | 48 |

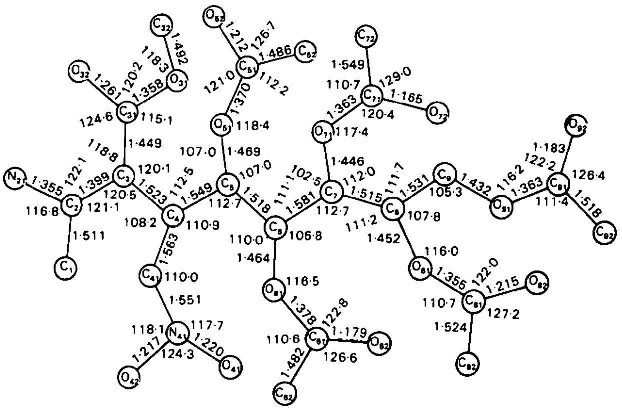

Fig. 1. View of the molecule with atom numbering, bond lengths (Å) and angles (°); e.s.d.'s are 0·014 Å and 0·9°.

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* Lists of structure factors, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38246 (23 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

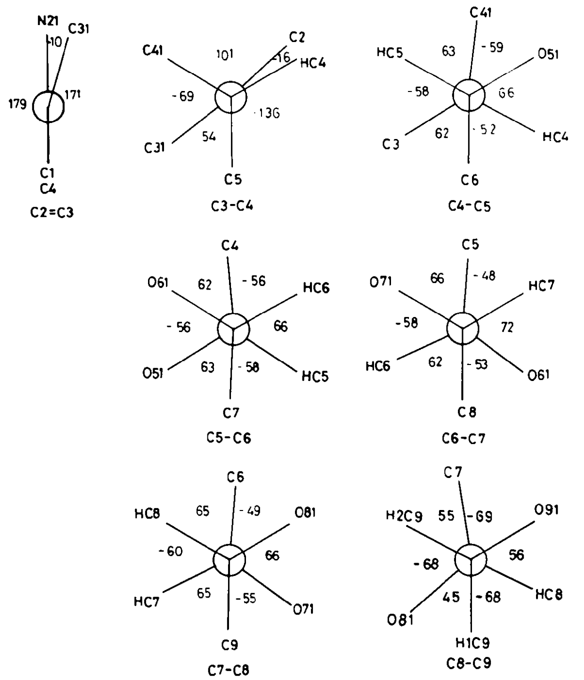

Fig. 2. Some Newman projections. Torsion angles in °; e.s.d.'s are in the range 0·0–0·1°.

the presence of the double bond C(2)=C(3) which necessarily restricts the side-chain conformation. In the C(4)—C(5) bond, C(6) is +synclinal to C(3) and —antiperiplanar to C(41). The conformation about the C(5)—C(6) bond is such that for the two acetoxy groups, O(61) is —synclinal to O(51) and C(7) +antiperiplanar to C(4). A situation with inverted torsions is found around C(7)—C(8), with O(81) +synclinal to O(71) and C(9) —antiperiplanar to C(6). Along C(6)—C(7), O(71) is —antiperiplanar to O(61) and C(8) —antiperiplanar to C(5). In the last conformational situation C(7) adopts a —synclinal orientation to O(91), and O(91) is +antiperiplanar to O(81). Thus, the configurations around the chiral centres C(8), C(7), C(6), C(5) and C(4) are *R*, *S*, *R*, *S* and *S*, respectively, in accordance with the D-*glycero*-L-*gluco* constitution.

The molecule has an intramolecular hydrogen bond between the N and O atoms of the amino and carbonyl groups respectively. The N(21)···O(32) distance is 2·60 (1) Å. This confirms the chelate structure previously observed by IR spectroscopy (Gómez-Sánchez *et al.*, 1980). The packing of the molecules is governed by normal van der Waals contacts.

The authors are grateful to Professor A. Gómez-Sánchez for suggesting the study and supplying the crystal and Professor A. Conde for helpful discussions. They also thank Dr E. Gutierrez Puebla (Inorganic Department, Facultad de Química, Universidad Complutense, Madrid) for collecting data.

The present work is a part of a wider research project supported by the Government through the 'Comisión Asesora de Investigación Científica y Técnica'.

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*Acta Cryst.* (1983). C**39**, 466–470

# Structure des Formes Racémique et Optiquement Active du Radical *N*-Oxyle-*exo*: Diméthyl-4′,4′ Trinor-8,9,10 Spiro[bornane-2:2′-[oxazolidine-1,3]] Oxyle-3′, $C_{11}H_{18}NO_2$

Par Denise Bordeaux, Geneviève Gagnaire et Jeanine Lajzérowicz

*Université Scientifique et Médicale de Grenoble, Laboratoire de Spectrométrie Physique, Boîte Postale n° 68, 38402 Saint-Martin-d'Heres CEDEX, France*

(*Reçu le* 23 *mars* 1982, *accepté le* 28 *octobre* 1982)

**Abstract.** $M_r = 196{\cdot}26$. racemic form: monoclinic, $P2_1/c$, $a = 8{\cdot}571$ (3), $b = 10{\cdot}526$ (3), $c = 11{\cdot}876$ (4) Å, $\beta = 98{\cdot}22$ (2)°, $Z = 4$, $V = 1047{\cdot}9$ Å$^3$, $D_x = 1{\cdot}243$ Mg m$^{-3}$, $\lambda$(Cu $K\alpha$) = 1·5418 Å, $\mu$ =

0108-2701/83/040466-05$01.50