Form 1 are close to a and b respectively, of Form 2, and the direction of the c axis of Form 1 corresponds to that of $[\bar{1}02]$ of Form 2.

The O···O distance of the O···H···O hydrogen bond in Form 2 is significantly shorter than that in Form 1. The hydrogen bis(p-chlorobenzoate) anion in Form 2 has symmetric environments, while the same anion in Form 1 has asymmetric environments. Such a shortening of the O···O distance in symmetric environments has been seen for the same hydrogen bis(p-methylbenzoate) anion in environments of different symmetry (Misaki, Kashino & Haisa, 1989b). This fact is an indication that symmetric environments around the anion are suitable for decreasing the O···O distance. Observations of the asymmetric O—H···O hydrogen bonds show that the O—H bond lengthens as the O···O distance decreases (Misaki, Kashino & Haisa, 1986). Thus, it can be estimated that the O—H length in Form 2 is longer than the 1·12 (6) Å in Form 1. This means that the position of the H atom is within 0·1 Å of the center of the hydrogen bond. This is the reason why the peak of the H atom in a difference Fourier map appeared at the center of the hydrogen bond. The spacing between the planes of the O—C—O groups participating in the hydrogen bond is 0·121 (9) Å and the KKM effect was not observed (Misaki, Kashino & Haisa, 1989a).

References

ASHIDA, T. (1973). *HBLS and DAPH. The Universal Crystallographic Computing System – Osaka.* The Computation Center, Osaka Univ., Japan.

FUJII, S. (1979). *MOLCON. The Universal Crystallographic Computing System – Osaka.* The Computation Center, Osaka Univ., Japan.

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

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

MAIN, P., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1978). *MULTAN78. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.

MISAKI, S., KASHINO, S. & HAISA, M. (1986). *Bull. Chem. Soc. Jpn*, **59**, 1059–1065.

MISAKI, S., KASHINO, S. & HAISA, M. (1989a). *Acta Cryst.* C45, 62–65.

MISAKI, S., KASHINO, S. & HAISA, M. (1989b). *Acta Cryst.* C45, 917–921.

SAKURAI, T. (1967). *RSSFR-5. The Universal Crystallographic Computing System* (I). The Crystallographic Society of Japan, Tokyo, Japan.

TAKUSAGAWA, F. (1982). *CRLS.* Tech. Rep. ICR-1982-0001-0002-001. The Institute for Cancer Research, Philadelphia, USA.

Structures of (E)-9-Styrylacridine and (Z)-9-(2,5-Dimethylstyryl)acridine

BY PAOLO SGARABOTTO* AND FRANCO UGOZZOLI

Istituto di Strutturistica Chimica, Università degli Studi di Parma, Centro di Studio per la Strutturistica Diffrattometrica del CNR, Viale delle Scienze, I-43100 Parma, Italy

AND SALVATORE SORRISO

Dipartimento di Chimica, Università di Perugia, I-06100 Perugia, Italy

Abstract. (E)-9-Styrylacridine, $C_{21}H_{15}N$, $M_r = 281\cdot4$, triclinic, $P\bar{1}$, $a = 12\cdot363$ (4), $b = 8\cdot386$ (3), $c = 8\cdot482$ (3) Å, $\alpha = 77\cdot0$ (1), $\beta = 121\cdot0$ (1), $\gamma = 96\cdot9$ (1)°, $V = 734\cdot5$ (9) Å^3, $Z = 2$, $D_x = 1\cdot27$ g cm^{-3}, Cu $K\alpha$, $\lambda = 1\cdot5418$ Å, $\mu = 5\cdot3$ cm^{-1}, $F(000) = 296$, $T = 293$ K, final conventional $R = 0\cdot038$ for 1346 symmetry-independent observed reflections. (Z)-9-(2,5-Dimethylstyryl)acridine, $C_{23}H_{19}N$, $M_r = 309\cdot4$, triclinic, $P\bar{1}$, $a = 11\cdot905$ (4), $b = 8\cdot955$ (3), $c = 8\cdot429$ (3) Å, $\alpha = 99\cdot5$ (1), $\beta = 73\cdot3$ (1), $\gamma = 92\cdot8$ (1)°, $V = 848\cdot9$ (7) Å^3, $Z = 2$, $D_x = 1\cdot21$ g cm^{-3}, Cu $K\alpha$, $\lambda = 1\cdot5418$ Å, $\mu = 5\cdot0$ cm^{-1}, $F(000) = 328$, $T = 293$ K, final conventional $R = 0\cdot052$ for 2423 symmetry-independent observed reflections. The conformational geometry of the acridine moiety is similar in the two compounds and is characterized by the presence of an approximate mirror plane along the N(1)—C(8) line and of a good degree of planarity for the three-condensed-rings system.

---

* To whom correspondence should be addressed.

Table 1. *Fractional atomic coordinates* (× 10⁴) *and equivalent isotropic thermal parameters* (× 10⁴ Å²) *for non-H atoms with e.s.d.'s in parentheses*

$$U_{eq} = \tfrac{1}{3}\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*(\mathbf{a}_i.\mathbf{a}_j).$$

	x	y	z	U_{eq}
(E)-9-Styrylacridine				
N(1)	995 (2)	2381 (2)	5189 (3)	460 (9)
C(2)	579 (2)	2530 (3)	6338 (3)	426 (11)
C(3)	−273 (2)	1319 (3)	6548 (3)	519 (12)
C(4)	−702 (3)	1389 (3)	7702 (4)	582 (12)
C(5)	−318 (2)	2656 (3)	8736 (4)	560 (14)
C(6)	494 (2)	3837 (3)	8569 (3)	483 (11)
C(7)	968 (2)	3827 (3)	7370 (3)	396 (9)
C(8)	1827 (2)	5010 (2)	7176 (3)	415 (10)
C(9)	2268 (2)	4857 (2)	5985 (3)	422 (11)
C(10)	3097 (2)	6001 (3)	5618 (3)	520 (13)
C(11)	3475 (2)	5805 (3)	4428 (4)	592 (12)
C(12)	3042 (2)	4461 (3)	3503 (4)	589 (15)
C(13)	2230 (2)	3357 (3)	3776 (3)	535 (12)
C(14)	1812 (2)	3508 (3)	5018 (3)	431 (10)
C(15)	2190 (2)	6395 (3)	8171 (3)	441 (11)
C(16)	3359 (2)	6839 (3)	9327 (3)	458 (12)
C(17)	3707 (2)	8202 (2)	10341 (3)	418 (10)
C(18)	4948 (2)	8347 (3)	11769 (3)	506 (11)
C(19)	5296 (3)	9580 (3)	12789 (4)	604 (13)
C(20)	4412 (3)	10693 (3)	12381 (4)	606 (15)
C(21)	3200 (3)	10585 (3)	10947 (4)	599 (13)
C(22)	2845 (2)	9361 (3)	9936 (4)	527 (14)
(Z)-9-(2,5-Dimethylstyryl)acridine				
N(1)	4090 (1)	4502 (1)	7885 (2)	485 (5)
C(2)	3828 (1)	3772 (2)	6516 (2)	452 (5)
C(3)	4012 (2)	2178 (2)	6104 (2)	564 (6)
C(4)	3776 (2)	1370 (2)	4734 (3)	624 (8)
C(5)	3312 (2)	2095 (2)	3682 (3)	645 (7)
C(6)	3125 (2)	3615 (2)	4021 (2)	556 (6)
C(7)	3380 (1)	4510 (2)	5438 (2)	440 (5)
C(8)	3195 (1)	6087 (2)	5845 (2)	428 (5)
C(9)	3443 (1)	6854 (2)	7307 (2)	425 (6)
C(10)	3285 (1)	8448 (2)	7846 (2)	504 (7)
C(11)	3562 (2)	9138 (2)	9242 (2)	541 (6)
C(12)	4008 (2)	8298 (2)	10205 (2)	570 (7)
C(13)	4178 (2)	6781 (2)	9740 (2)	537 (7)
C(14)	3902 (1)	6006 (2)	8276 (2)	442 (5)
C(15)	2842 (2)	6917 (2)	4681 (2)	502 (6)
C(16)	1846 (2)	7637 (2)	4952 (2)	548 (6)
C(17)	799 (1)	7637 (2)	6424 (2)	534 (7)
C(18)	224 (2)	8994 (2)	7264 (3)	648 (8)
C(19)	−763 (2)	8889 (3)	8613 (3)	825 (10)
C(20)	−1182 (2)	7538 (3)	9103 (3)	817 (10)
C(21)	−628 (2)	6175 (3)	8271 (3)	682 (7)
C(22)	368 (2)	6262 (2)	6928 (2)	586 (7)
C(23)	659 (3)	10497 (3)	6795 (4)	842 (12)
C(24)	−1080 (3)	4683 (4)	8796 (4)	935 (13)

Introduction.

The acridines are of considerable biological interest because of their mutagenic and antitumor properties. On the other hand, in general it is well known that the biological activity is influenced, to a large extent, by the molecular dynamics.

In this paper we report the crystal and molecular structure of two styrylacridines, *trans-* and *cis-* substituted, undertaken to establish their conformation and to provide an unequivocal starting point for the interpretation of dielectric relaxation measurements which can correlate the molecular dynamics with the observed biological properties.

Experimental.

Crystals of compounds (E) and (Z) pale-brown prisms elongated on [001]. Specimen dimensions 0·18 × 0·16 × 0·51 and 0·20 × 0·15 × 0·45 mm respectively. Simens AED diffractometer on line to a General Automation Jumbo 220 microcomputer, Ni-filtered Cu *Kα* radiation, *θ–2θ* scan, scan width from (*θ* − 0·60) to (*θ* + 0·60 + 0·142tg*θ*)°; scan speed 3·0° min⁻¹. Cell dimensions based on 26 (*θ*, *χ*, *φ*)$_{hkl}$ (20 ≤ *θ* ≤ 30°) measured intensities. (E) 2592 (−15 ≤ *h* ≤ 12, −9 ≤ *k* ≤ 10, 0 ≤ *l* ≤ 8); (Z) 3388 (−13 ≤ *h* ≤ 14, −10 ≤ *k* ≤ 10, 0 ≤ *l* ≤ 9) symmetry-independent reflections (3 ≤ *θ* ≤ 70°) collected at *T* = 293 K with a modified version (Belletti, Ugozzoli, Cantoni & Pasquinelli, 1979) of the Lehmann & Larsen (1974) procedure. One standard reflection every 50, no significant variation. Corrections for Lorentz and polarization, no correction for absorption effects. Structures solved by direct methods (*MULTAN;* Main, Woolfson & Germain, 1971). (E) 1346, (Z) 2423 symmetry-independent observed reflections [*I* > 2σ(*I*)], in full-matrix least-squares refinement (E) 259, (Z) 289 parameters; all H atoms located in the difference-Fourier map refined isotropically. $\Sigma w(F_o - F_c)^2$ minimized with $w = [\sigma^2(F_o) + 0\cdot005F_o^2]^{-1}$; *SHELX76* system (Sheldrick, 1976); maximum Δ/σ 0·21 for N, 0·10 for C, 0·40 for H. $\Delta\rho_{max}$ and $\Delta\rho_{min}$ (E) 0·18, −0·20; (Z) 0·21, −0·24 e Å⁻³. Final refinement converged to *R* and *wR* (E) 0·038, 0·038; (Z) 0·052, 0·059. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974).

The calculations were carried out on the Gould 32/77 computer of the Centro di Studio per la Strutturistica Diffrattometrica del CNR, Parma, and on the Cray X-MP/12 computer of Consorzio per la Gestione del Centro di Calcolo Interuniversitario dell'Italia Nord-Orientale, Casalecchio, Bologna. Bibliographic searches were carried out using the Cambridge Crystallographic Data Files through the Servizio Italiano di Diffusione dei Dati Cristallografici, Parma.

Discussion.

Final atomic coordinates and equivalent isotropic thermal parameters are given in Table 1.* The arbitrary numbering scheme used in the crystal analyses is shown in Figs. 1 and 2, which represent a perspective view of (E)-9-styryl- and (Z)-9-(2,5-dimethylstyryl)acridine, respectively. Bond distances and angles are reported in Table 2.

The acridine nucleus presents a conformation in good agreement with that found for acridine (Phillips, 1956; Phillips, Ahmed & Barnes, 1960) and related compounds reported in the literature, so that the localized ethylenic double bond [C(15)=C(16) 1·328 (3) and 1·321 (3) Å in (E) and (Z) 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 51946 (30 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

seems not to influence at all the geometry of the three-condensed-rings system: in particular the acridine moiety shows experimental mirror symmetry along the N(1)—C(8) line as expected for simple acridines (Jones & Neidle, 1975, and references therein); in fact the differences between equivalent bond distances and angles are not significant within the limits of error for both compounds. Also the shortening of the bonds C(3)—C(4), C(5)—C(6) and their mirror equivalents [average value 1·356 (2) and 1·357 (2) Å in the (E) and (Z) compounds respectively] (increased double-bond character) and the lengthening of all the other C—C bonds [average value 1·423 (2) and 1·426 (1) Å in the (E) and (Z) compounds respectively] (increased single-bond character) is a situation evident for unsubstituted acridines. The significant overall nonplanarity of the
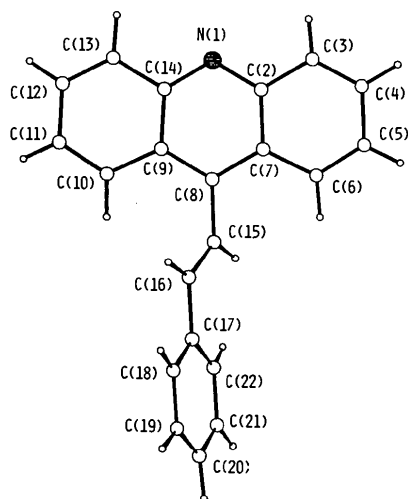

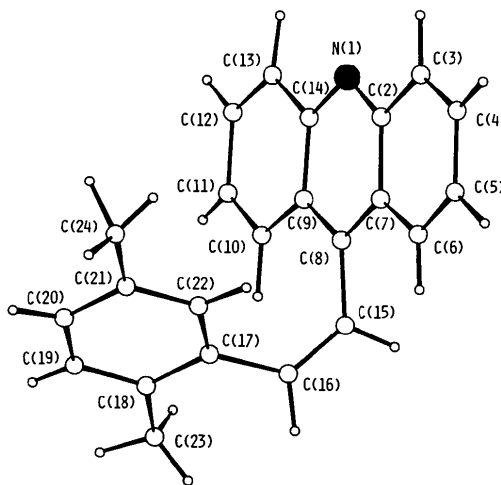Fig. 1. Perspective view of (E)-9-styrylacridine with the atomic numbering scheme.

Fig. 2. Perspective view of (Z)-9-(2,5-dimethylstyryl)acridine with the atomic numbering scheme.

Table 2. *Bond distances* (Å), *bond angles* (°) *and selected torsion angles* (°) *with e.s.d.'s in parentheses*

	(E)	(Z)		(E)	(Z)
N(1)—C(2)	1·348 (5)	1·339 (3)	C(11)—C(12)	1·412 (4)	1·412 (3)
N(1)—C(14)	1·340 (4)	1·347 (2)	C(12)—C(13)	1·356 (4)	1·359 (3)
C(2)—C(3)	1·424 (4)	1·425 (3)	C(13)—C(14)	1·425 (5)	1·428 (3)
C(2)—C(7)	1·432 (4)	1·435 (3)	C(15)—C(16)	1·328 (3)	1·321 (3)
C(3)—C(4)	1·346 (5)	1·352 (3)	C(16)—C(17)	1·475 (4)	1·485 (3)
C(4)—C(5)	1·412 (5)	1·421 (4)	C(17)—C(18)	1·394 (3)	1·402 (3)
C(5)—C(6)	1·361 (4)	1·360 (3)	C(17)—C(22)	1·386 (4)	1·395 (3)
C(6)—C(7)	1·413 (5)	1·425 (3)	C(18)—C(19)	1·384 (4)	1·393 (3)
C(7)—C(8)	1·412 (3)	1·410 (3)	C(18)—C(23)		1·492 (4)
C(8)—C(9)	1·406 (4)	1·410 (3)	C(19)—C(20)	1·377 (5)	1·368 (4)
C(8)—C(15)	1·479 (4)	1·483 (3)	C(20)—C(21)	1·368 (4)	1·395 (4)
C(9)—C(10)	1·423 (4)	1·429 (3)	C(21)—C(22)	1·372 (5)	1·394 (3)
C(9)—C(14)	1·435 (4)	1·430 (3)	C(21)—C(24)		1·503 (5)
C(10)—C(11)	1·358 (5)	1·356 (3)			
C(2)—N(1)—C(14)	117·8 (4)	118·8 (3)	C(11)—C(12)—C(13)	120·3 (4)	120·5 (2)
N(1)—C(2)—C(3)	118·0 (4)	118·1 (2)	C(12)—C(13)—C(14)	121·1 (4)	120·6 (3)
N(1)—C(2)—C(7)	123·2 (5)	123·6 (3)	N(1)—C(14)—C(9)	123·9 (4)	123·5 (2)
C(3)—C(2)—C(7)	118·9 (4)	118·3 (2)	N(1)—C(14)—C(13)	117·4 (4)	117·7 (3)
C(2)—C(3)—C(4)	120·6 (4)	121·4 (3)	C(9)—C(14)—C(13)	118·7 (4)	118·9 (3)
C(3)—C(4)—C(5)	121·0 (5)	120·4 (3)	C(8)—C(15)—C(16)	126·2 (5)	127·5 (2)
C(4)—C(5)—C(6)	120·1 (4)	120·4 (3)	C(15)—C(16)—C(17)	125·6 (5)	126·4 (3)
C(5)—C(6)—C(7)	121·2 (4)	120·9 (3)	C(16)—C(17)—C(18)	119·5 (4)	120·6 (3)
C(2)—C(7)—C(6)	118·2 (4)	118·6 (3)	C(16)—C(17)—C(22)	122·6 (4)	119·2 (2)
C(2)—C(7)—C(8)	118·6 (4)	118·4 (2)	C(18)—C(17)—C(22)	117·9 (4)	120·2 (2)
C(6)—C(7)—C(8)	123·2 (4)	123·0 (2)	C(17)—C(18)—C(19)	120·9 (4)	116·9 (3)
C(7)—C(8)—C(9)	118·5 (4)	118·2 (3)	C(17)—C(18)—C(23)		122·4 (3)
C(7)—C(8)—C(15)	118·9 (4)	120·1 (2)	C(19)—C(18)—C(23)		120·7 (3)
C(9)—C(8)—C(15)	122·5 (4)	121·5 (3)	C(18)—C(19)—C(20)	119·8 (4)	122·7 (3)
C(8)—C(9)—C(10)	123·9 (4)	123·1 (2)	C(19)—C(20)—C(21)	119·6 (4)	121·2 (3)
C(8)—C(9)—C(14)	118·1 (4)	118·6 (3)	C(20)—C(21)—C(22)	121·0 (5)	116·8 (3)
C(10)—C(9)—C(14)	118·0 (4)	118·3 (2)	C(20)—C(21)—C(24)		121·8 (3)
C(9)—C(10)—C(11)	121·3 (4)	120·8 (3)	C(22)—C(21)—C(24)		121·5 (3)
C(10)—C(11)—C(12)	120·6 (5)	120·8 (3)	C(17)—C(22)—C(21)	120·8 (4)	122·2 (3)

	(E)	(Z)
C(8)—C(15)—C(16)—C(17)	−178·6 (4)	−7·1 (6)

Table 3. *Analysis of the planarity*

(a) Distances (Å × 10³) of relevant atoms from the mean plane with standard deviations in parentheses; starred atoms were not used to define the plane

	(E)	(Z)
Plane A: N(1), C(2)–C(14)		
N(1)	1 (4)	8 (4)
C(2)	15 (4)	2 (4)
C(3)	10 (4)	−6 (4)
C(4)	−1 (5)	−2 (4)
C(5)	−12 (5)	−16 (4)
C(6)	−5 (4)	−3 (4)
C(7)	13 (4)	13 (4)
C(8)	−2 (4)	19 (4)
C(9)	−22 (4)	−7 (4)
C(10)	13 (4)	−9 (4)
C(11)	15 (5)	−4 (4)
C(12)	−3 (5)	−4 (4)
C(13)	−7 (4)	3 (4)
C(14)	−16 (4)	2 (4)
C(15)*	57 (4)	161 (4)
Plane B: C(8), C(15)–C(17)		
C(8)	−7 (4)	−11 (4)
C(15)	7 (4)	27 (4)
C(16)	7 (4)	−27 (4)
C(17)	−7 (4)	11 (4)
Plane C: C(17)–C(22)		
C(17)	−12 (5)	−4 (4)
C(18)	9 (5)	7 (4)
C(19)	2 (5)	−4 (4)
C(20)	−11 (5)	−1 (4)
C(21)	7 (5)	3 (4)
C(22)	6 (5)	0 (4)
C(16)*	−52 (5)	31 (4)

(b) Angles (°) between planes

A–B	55·5 (3)	108·2 (1)
A–C	67·1 (1)	106·3 (1)
B–C	11·8 (3)	51·8 (1)

acridine moiety, Table 3, somewhat less pronounced in the (Z) derivative, is of the same order as found in other acridines considered to be planar: for both compounds the best planes of the two outer rings are roughly parallel to one another and also parallel to the central ring in (Z) while in (E) they are rotated with respect to the best plane of the central ring by $1\cdot4\,(1)°$.

As far as the geometry of the ethylenic C(15)=C(16) bond is concerned, the distortion about the double bond is given in terms of the *trans* torsion angle in the (E) and the *cis* torsion angle in the (Z) derivative, reported in Table 2. The puckering value for the *cis*, greater than that of the *trans* compound, reflects the mutual steric interaction of the xylyl and the acridinyl groups.

The packing is consistent with van der Waals interactions.

References

BELLETTI, D., UGOZZOLI, F., CANTONI, A. & PASQUINELLI, G. (1979). *Gestione on line di Diffrattometro a Cristallo Singolo Siemens AED con Sistema General Automation Jumbo 220.* Internal Report 1–3/79. Centro di Studio per la Strutturistica Diffrattometrica del CNR, Parma, Italy.
International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)
JONES, A. & NEIDLE, S. (1975). *Acta Cryst.* B31, 1324–1333.
LEHMANN, M. S. & LARSEN, F. K. (1974). *Acta Cryst.* A30, 580–589.
MAIN, P., WOOLFSON, M. M. & GERMAIN, G. (1971). *MULTAN. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.
PHILLIPS, D. C. (1956). *Acta Cryst.* 9, 237–250.
PHILLIPS, D. C., AHMED, F. R. & BARNES, W. H. (1960). *Acta Cryst.* 13, 365–377.
SHELDRICK, G. M. (1976). *SHELX76.* Program for crystal structure determination. Univ. of Cambridge, England.

Structure of 7,14,15,17-Tetraoxa-10-azapentacyclo[10.2.1.1²,¹¹.1⁵,⁸.0³,⁹]heptadeca-3,9-dien-2-ol, a Pentacyclic Alcohol Prepared from Levoglucosenone

BY V. KETTMANN

Department of Analytical Chemistry, Faculty of Pharmacy, Comenius University, Odbojárov 10, CS-83232 Bratislava, Czechoslovakia

AND S. BYSTRICKÝ, T. STICZAY AND M. KOÓŠ

Institute of Chemistry, Centre for Chemical Research, Slovak Academy of Sciences, Dúbravská cesta, CS-84238 Bratislava, Czechoslovakia

(*Received* 27 *January* 1988; *accepted* 17 *February* 1989)

Abstract. $C_{12}H_{13}NO_5$, $M_r = 251\cdot2$, orthorhombic, $P2_12_12_1$, $a = 9\cdot294\,(3)$, $b = 10\cdot714\,(3)$, $c = 10\cdot846\,(3)$ Å, $V = 1080\cdot0\,(6)$ Å^3, $Z = 4$, $D_m = 1\cdot55$, $D_x = 1\cdot55$ Mg m^{-3}, λ(Cu $K\alpha$) = $1\cdot54178$ Å, $\mu = 1\cdot04$ mm^{-1}, $F(000) = 528$, $T = 293$ K, $R = 0\cdot039$ for 800 unique observed reflections. The crystal structure consists of helical chains of the title molecule, the principal intrachain interaction being hydrogen bonding between the hydroxyl and imine groups. The structure previously assigned on the basis of chemical and spectroscopic evidence is confirmed. The conformation of the pyranose ring is $^1C_4$ distorted towards a mixture of E_0 and $^1H_0$, and that of the anhydro rings is between ^0E and $^0T_4$. The inherent rigidity of the pentacyclic skeleton of the title alcohol forces N=C and C=C bonds of the α,β-unsaturated imine moiety, —N=C—C=C—, to

be coplanar but there is no crystallographic evidence for any conjugation within this moiety.

Introduction. The title compound (III) was prepared by reaction of glycoside precursor (I), 1,6-anhydro-3,4-dideoxy-β-D-*glycero*-hex-3-enopyranos-2-ulose (levoglucosenone) (Bhaté & Horton, 1983), with ammonium carbonate in refluxing 95% ethanol. Spectroscopic and analytical data (Bystrický, Sticzay, Koóš, Kettmann, 1986) were consistent with the structure (III), *i.e.* the reaction was found to proceed in a different way from that observed in pyrones, which, under the same reaction conditions, prefer substitution of the ring oxygen by nitrogen to give pyridones (Kaiser, Work, Wolfe & Hauser, 1967). The carbohydrate product (III) must thus have arisen by a novel sequential reaction, the