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Structure and Absolute Configuration of (–)-Phenyl α -(1-Phenylpropylimino)benzyl Ketone

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Abstract. $C_{23}H_{21}NO$, $P2_1$, four molecules per unit cell, two independent in the asymmetric unit, with $a = 19.188$ (2), $b = 9.2573$ (5), $c = 10.739$ (1) Å, $\beta = 100.190$ (8)°, Cu $K\alpha$ radiation. The structure was solved by direct methods and refined by block-diagonal least squares to a final R of 0.037 for 5535 observed reflexions. The enantiomer studied has an R configuration.

Introduction. As a continuation of the study of derivatives of benzyl imines (Fonseca, Martínez-Carrera & García-Blanco, 1979), the molecular structure and absolute configuration of the title compound are described. A colourless, prismatic crystal of $0.10 \times 0.40 \times 0.41$ mm was used to determine the cell parameters and to record the intensity data on a Philips

1100 four-circle diffractometer. Lattice parameters were refined by a least-squares fit of 34 reflexions. 6494 (hkl , $\bar{h}\bar{k}l$) intensities were measured using the $\omega/2\theta$ scan technique to $\theta = 60^\circ$ with Ni-filtered Cu $K\alpha$ radiation; 5535 with intensities greater than $2\sigma(I)$ were used for the structure analysis. No absorption correction was applied. The structure was solved by direct methods with *MULTAN 77* (Main, Lessinger, Woolfson, Germain & Declercq, 1977) on an E map calculated with the 200 largest E values. All non-hydrogen atoms of molecule *A* and 22 of molecule *B* were located. A three-dimensional Fourier synthesis showed the three remaining atoms. The structure was refined by block-diagonal least-squares calculation with anisotropic thermal parameters to an R of 0.083. A difference Fourier map revealed the positions of all H

Table 1. Atomic parameters ($\times 10^4$) and equivalent isotropic temperature factors ($\text{\AA}^2 \times 10^3$) for C₂₃H₂₁NO non-hydrogen atoms with e.s.d's in parentheses

$$U_{eq} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* a_i a_j \cos(a_i a_j)$$

	x	y	z	U _{eq}
C(1A)	-1177 (1)	-943 (2)	-5945 (1)	46 (0.1)
C(2A)	-497 (1)	-1343 (2)	-5392 (2)	54 (1)
C(3A)	-192 (1)	-757 (3)	-4228 (2)	67 (1)
C(4A)	-557 (1)	222 (3)	-3639 (2)	74 (1)
C(5A)	-1235 (1)	608 (3)	-4177 (2)	79 (1)
C(6A)	-1552 (1)	29 (2)	-5326 (2)	62 (1)
C(7A)	-1518 (1)	-1522 (2)	-7185 (1)	45 (0.1)
C(8A)	-1111 (1)	-2603 (2)	-7852 (1)	43 (0.1)
C(9A)	-1327 (1)	-4142 (2)	-7783 (2)	46 (0.1)
C(10A)	-1597 (1)	-4656 (2)	-6750 (2)	60 (1)
C(11A)	-1795 (1)	-6096 (3)	-6708 (3)	76 (1)
C(12A)	-1738 (1)	-7001 (3)	-7690 (3)	78 (1)
C(13A)	-1472 (1)	-6508 (2)	-8718 (2)	74 (1)
C(14A)	-1262 (1)	-5074 (2)	-8766 (2)	61 (1)
C(15A)	-457 (1)	-727 (2)	-8636 (2)	52 (1)
C(16A)	303 (1)	-415 (2)	-8060 (2)	50 (1)
C(17A)	848 (1)	-1373 (2)	-8169 (2)	60 (1)
C(18A)	1543 (1)	-1010 (3)	-7707 (2)	70 (1)
C(19A)	1711 (1)	297 (3)	-7121 (2)	77 (1)
C(20A)	1180 (1)	1240 (3)	-7001 (3)	86 (1)
C(21A)	482 (1)	885 (2)	-7462 (2)	73 (1)
C(22A)	-599 (1)	-409 (3)	-10060 (2)	67 (1)
C(23A)	-1374 (1)	-528 (4)	-10645 (3)	86 (1)
N(A)	-631 (1)	-2256 (2)	-8474 (1)	49 (1)
O(A)	-2110 (1)	-1174 (2)	-7691 (1)	68 (1)
C(1B)	-3797 (1)	-8129 (1)	-112 (1)	51 (1)
C(2B)	-4488 (1)	-7681 (1)	-116 (1)	61 (1)
C(3B)	-4859 (1)	-8248 (1)	761 (1)	74 (1)
C(4B)	-4548 (1)	-9237 (1)	1647 (1)	77 (1)
C(5B)	-3860 (1)	-9659 (1)	1662 (1)	78 (1)
C(6B)	-3485 (1)	-9119 (1)	782 (1)	62 (1)
C(7B)	-3389 (1)	-7594 (1)	-1069 (1)	51 (1)
C(8B)	-3726 (1)	-6484 (1)	-2030 (1)	49 (1)
C(9B)	-3535 (1)	-4944 (1)	-1769 (1)	50 (1)
C(10B)	-3184 (1)	-4496 (1)	-596 (1)	66 (1)
C(11B)	-3006 (1)	-3045 (1)	-384 (1)	83 (1)
C(12B)	-3172 (1)	-2068 (1)	-1343 (1)	81 (1)
C(13B)	-3515 (1)	-2497 (1)	-2501 (1)	73 (1)
C(14B)	-3698 (1)	-3929 (1)	-2725 (1)	60 (1)
C(15B)	-4305 (1)	-8323 (1)	-3386 (1)	62 (1)
C(16B)	-5076 (1)	-8606 (1)	-3374 (1)	60 (1)
C(17B)	-5586 (1)	-7641 (1)	-3906 (1)	79 (1)
C(18B)	-6294 (1)	-7898 (1)	-3885 (1)	103 (1)
C(19B)	-6484 (1)	-9154 (1)	-3316 (1)	120 (1)
C(20B)	-5981 (1)	-10107 (1)	-2797 (1)	125 (1)
C(21B)	-5282 (1)	-9837 (1)	-2826 (1)	94 (1)
C(22B)	-4115 (1)	-8569 (1)	-4702 (1)	86 (1)
C(23B)	-3334 (1)	-8409 (1)	-4722 (1)	118 (1)
N(B)	-4139 (1)	-6810 (1)	-3057 (1)	56 (0.1)
O(B)	-2802 (1)	-8037 (1)	-1123 (1)	74 (1)

atoms. A suitable weighting scheme (Martinez-Ripoll & Cano, 1975) was applied so as to give no trends in $\langle w\Delta^2 \rangle$ vs $|F_o|$ and $\sin\theta/\lambda$. The final R and R_w values were 0.037 and 0.045 respectively. The final atomic parameters are listed in Tables 1 and 2.* A three-dimensional difference map indicates no unaccounted electron density.

* Lists of anisotropic thermal parameters, structure factors, weighting scheme and least-squares planes have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 36892 (44 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. Atomic parameters ($\times 10^3$) and isotropic thermal parameters ($\text{\AA}^2 \times 10^3$) for C₂₃H₂₁NO hydrogen atoms

Thermal parameters are defined as $\exp[-8\pi^2 U(\sin\theta/\lambda)^2]$.

	x	y	z	U
H(2A)	-22 (1)	-200 (3)	-579 (2)	33 (5)
H(3A)	27 (1)	-102 (3)	-388 (2)	53 (6)
H(4A)	-34 (1)	65 (3)	-286 (3)	63 (7)
H(5A)	-155 (2)	124 (5)	-379 (3)	99 (11)
H(6A)	-203 (1)	27 (2)	-574 (2)	38 (5)
H(10A)	-160 (1)	-405 (3)	-599 (2)	52 (6)
H(11A)	-200 (1)	-644 (4)	-603 (3)	64 (8)
H(12A)	-189 (1)	-799 (3)	-760 (2)	49 (6)
H(13A)	-149 (1)	-717 (4)	-946 (3)	66 (7)
H(14A)	-105 (1)	-465 (3)	-950 (2)	52 (6)
H(15A)	-75 (1)	-7 (2)	-819 (2)	33 (5)
H(17A)	72 (1)	-229 (3)	-852 (2)	49 (6)
H(18A)	190 (1)	-160 (3)	-780 (2)	55 (7)
H(19A)	221 (2)	57 (3)	-675 (2)	58 (7)
H(20A)	130 (2)	216 (5)	-653 (3)	93 (10)
H(21A)	12 (2)	148 (4)	-735 (3)	76 (9)
H(22A1)	-42 (1)	56 (3)	-1019 (2)	43 (6)
H(22A2)	-29 (1)	-114 (3)	-1047 (2)	46 (6)
H(23A1)	-150 (1)	-157 (4)	-1054 (3)	67 (8)
H(23A2)	-164 (2)	12 (5)	-1019 (4)	108 (13)
H(23A3)	-144 (2)	-23 (4)	-1153 (3)	75 (8)
H(2B)	-471 (1)	-703 (3)	-75 (3)	52 (7)
H(3B)	-531 (2)	-796 (4)	72 (3)	71 (9)
H(4B)	-480 (2)	-967 (4)	223 (3)	77 (9)
H(5B)	-361 (2)	-1029 (4)	234 (3)	69 (8)
H(6B)	-300 (1)	-947 (2)	79 (2)	29 (5)
H(10B)	-307 (1)	-511 (3)	10 (3)	47 (7)
H(11B)	-280 (2)	-283 (4)	39 (3)	76 (10)
H(12B)	-297 (2)	-117 (5)	-117 (3)	79 (9)
H(13B)	-357 (2)	-182 (4)	-310 (3)	68 (9)
H(14B)	-395 (1)	-428 (3)	-355 (3)	58 (7)
H(15B)	-402 (1)	-902 (3)	-277 (2)	36 (6)
H(17B)	-544 (1)	-679 (4)	-423 (3)	51 (7)
H(18B)	-663 (2)	-717 (4)	-433 (3)	69 (8)
H(19B)	-699 (3)	-936 (7)	-340 (5)	136 (16)
H(20B)	-614 (2)	-1112 (6)	-248 (4)	114 (14)
H(21B)	-493 (2)	-1047 (6)	-246 (4)	104 (13)
H(22B1)	-425 (2)	-948 (5)	-488 (3)	73 (9)
H(22B2)	-441 (1)	-783 (3)	-523 (2)	45 (7)
H(23B1)	-316 (2)	-722 (6)	-446 (4)	108 (13)
H(23B2)	-302 (2)	-909 (5)	-401 (4)	87 (11)
H(23B3)	-319 (2)	-858 (5)	-548 (4)	93 (11)

The absolute configuration has been determined by measuring Bijvoet differences using Cu K α radiation. 98 Bijvoet pairs (Bijvoet, Peerdeman & van Bommel, 1951) with $F_{c,\min} > 0.050$ and $F_{o,\min} > 0.017$ were selected. The anomalous scattering of N, O and C atoms was used and the f' and f'' values were taken from *International Tables for X-ray Crystallography* (1974). The discrepancy indices (Martinez-Ripoll & Fayos, 1980) $R_1 = 0.206$ (0.228) and $R_2 = 0.037$ (0.044) showed that the correct positional parameters correspond to those given in Table 1. The chiral C(15) atoms of the two independent molecules have an R configuration.

Discussion. Fig. 1 shows the atomic numbering and absolute configuration of the compound. An ORTEP (Johnson, 1965) view of the two independent molecules is shown in Fig. 2. Bond lengths and angles (Tables 3

and 4) are similar in the two independent molecules and are in good agreement with those found in the literature. The length C(8)–N is in both cases close to the value estimated by Burke-Laing & Laing (1976). The value of the C(7)–C(8) bond is nearer to a C(sp³)–C(sp³) bond

(1.536 Å) instead of the expected C(sp²)–C(sp²) one (1.482 Å). Torsion angles (Table 5) show the conformation of the molecules: the two molecules are very

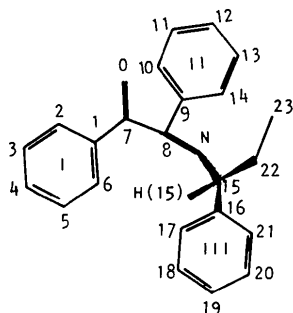


Fig. 1. Scheme of the molecule with atomic numbering and absolute configuration.

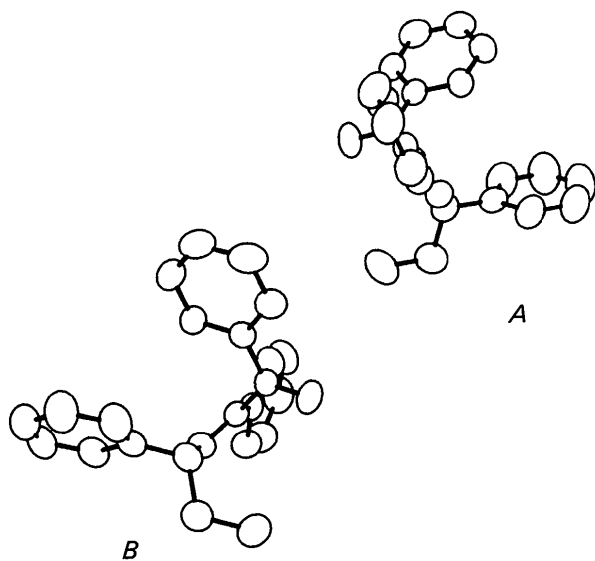


Fig. 2. ORTEP (Johnson, 1965) view of the two independent molecules.

Table 3. Bond distances (Å) with *e.s.d.*'s for non-hydrogen atoms

	A	B		A	B
C(1)–C(2)	1.385 (2)	1.388 (3)	C(11)–C(12)	1.366 (4)	1.365 (4)
C(1)–C(6)	1.392 (3)	1.384 (3)	C(12)–C(13)	1.372 (4)	1.359 (4)
C(1)–C(7)	1.477 (2)	1.483 (3)	C(13)–C(14)	1.391 (3)	1.381 (3)
C(2)–C(3)	1.392 (3)	1.397 (3)	C(15)–C(16)	1.508 (2)	1.505 (3)
C(3)–C(4)	1.367 (3)	1.365 (3)	C(15)–C(22)	1.534 (3)	1.538 (4)
C(4)–C(5)	1.374 (3)	1.374 (4)	C(15)–N	1.473 (2)	1.467 (3)
C(5)–C(6)	1.382 (3)	1.379 (3)	C(16)–C(17)	1.391 (3)	1.373 (3)
C(7)–C(8)	1.524 (2)	1.517 (3)	C(16)–C(21)	1.378 (3)	1.372 (4)
C(7)–O	1.212 (2)	1.210 (2)	C(17)–C(18)	1.379 (3)	1.383 (4)
C(8)–C(9)	1.490 (2)	1.486 (3)	C(18)–C(19)	1.376 (3)	1.392 (6)
C(8)–N	1.270 (2)	1.275 (2)	C(19)–C(20)	1.364 (4)	1.353 (6)
C(9)–C(10)	1.389 (3)	1.383 (3)	C(20)–C(21)	1.384 (3)	1.370 (5)
C(9)–C(14)	1.386 (3)	1.386 (3)	C(22)–C(23)	1.511 (3)	1.510 (5)
C(10)–C(11)	1.389 (3)	1.395 (3)			

Table 4. Bond angles (°) with *e.s.d.*'s for non-hydrogen atoms

	A	B		A	B
C(6)–C(1)–C(7)	118.8 (2)	119.0 (2)	C(10)–C(11)–C(12)	120.2 (2)	119.8 (2)
C(2)–C(1)–C(7)	121.5 (2)	121.3 (2)	C(11)–C(12)–C(13)	120.6 (2)	120.5 (3)
C(2)–C(1)–C(6)	119.7 (2)	119.7 (2)	C(12)–C(13)–C(14)	119.9 (2)	120.4 (2)
C(1)–C(2)–C(3)	119.7 (2)	119.2 (2)	C(9)–C(14)–C(13)	120.0 (2)	120.3 (2)
C(2)–C(3)–C(4)	120.3 (2)	120.5 (2)	C(22)–C(15)–N	107.3 (2)	106.6 (2)
C(3)–C(4)–C(5)	120.1 (2)	120.1 (2)	C(16)–C(15)–N	111.1 (2)	109.7 (2)
C(4)–C(5)–C(6)	120.6 (2)	120.4 (2)	C(16)–C(15)–C(22)	111.2 (2)	112.0 (2)
C(1)–C(6)–C(5)	119.5 (2)	120.1 (2)	C(15)–C(16)–C(21)	120.0 (2)	120.4 (2)
C(1)–C(7)–O	122.5 (2)	122.0 (2)	C(15)–C(16)–C(17)	122.0 (2)	120.8 (2)
C(1)–C(7)–C(8)	118.7 (1)	118.9 (2)	C(17)–C(16)–C(21)	117.8 (2)	118.7 (2)
C(8)–C(7)–O	118.7 (2)	119.0 (2)	C(16)–C(17)–C(18)	120.5 (2)	120.7 (3)
C(7)–C(8)–N	124.1 (2)	123.6 (2)	C(17)–C(18)–C(19)	120.8 (2)	119.0 (3)
C(7)–C(8)–C(9)	115.7 (1)	117.4 (2)	C(18)–C(19)–C(20)	119.1 (2)	120.2 (4)
C(9)–C(8)–N	120.1 (2)	119.0 (2)	C(19)–C(20)–C(21)	120.4 (2)	120.1 (4)
C(8)–C(9)–C(14)	119.6 (2)	119.6 (2)	C(16)–C(21)–C(20)	121.2 (2)	121.2 (3)
C(8)–C(9)–C(10)	120.9 (2)	121.6 (2)	C(15)–C(22)–C(23)	112.9 (2)	113.3 (3)
C(10)–C(9)–C(14)	119.5 (2)	118.8 (2)	C(8)–N–C(15)	120.4 (2)	120.7 (2)
C(9)–C(10)–C(11)	119.8 (2)	120.2 (2)			

Table 5. Torsion angles with *e.s.d.*'s (°)

	A	B
C(2)–C(1)–C(7)–C(8)	1.1 (2)	2.8 (3)
C(6)–C(1)–C(7)–C(8)	–179.1 (2)	–178.7 (2)
C(2)–C(1)–C(7)–O	–179.2 (2)	–175.6 (2)
C(6)–C(1)–C(7)–O	0.6 (3)	2.9 (3)
C(1)–C(7)–C(8)–N	–79.5 (2)	–84.5 (2)
C(1)–C(7)–C(8)–C(9)	102.9 (2)	98.1 (2)
O–C(7)–C(8)–N	100.8 (2)	94.1 (2)
O–C(7)–C(8)–C(9)	–76.9 (2)	–83.4 (2)
C(7)–C(8)–C(9)–C(10)	–30.1 (2)	–12.8 (3)
C(7)–C(8)–C(9)–C(14)	149.5 (2)	165.9 (2)
N–C(8)–C(9)–C(14)	–28.2 (3)	–11.7 (3)
N–C(8)–C(9)–C(10)	152.1 (2)	169.6 (2)
C(7)–C(8)–N–C(15)	–2.7 (3)	–1.2 (3)
C(9)–C(8)–N–C(15)	174.9 (2)	176.2 (2)
C(8)–C(9)–C(10)–C(11)	180.0 (2)	179.4 (2)
C(8)–C(9)–C(14)–C(13)	–179.0 (2)	–179.1 (2)
C(16)–C(15)–N–C(8)	118.9 (2)	113.6 (2)
C(22)–C(15)–N–C(8)	–119.4 (2)	–124.9 (2)
C(22)–C(15)–C(16)–C(17)	–79.2 (2)	–74.6 (3)
C(22)–C(15)–C(16)–C(21)	97.3 (2)	105.5 (3)
N–C(15)–C(16)–C(17)	40.2 (3)	43.6 (3)
N–C(15)–C(16)–C(21)	–143.3 (2)	–136.3 (2)

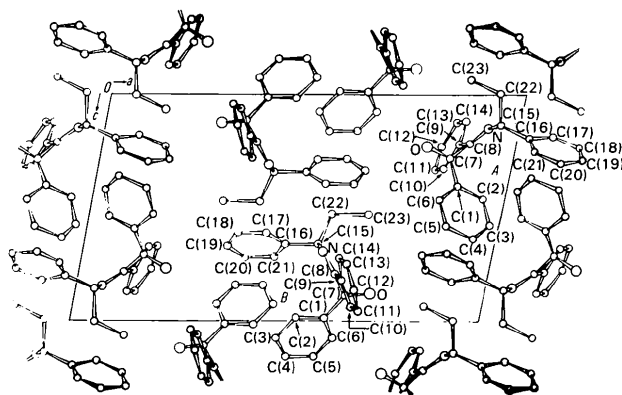


Fig. 3. Packing diagram for the title compound as viewed along the *b* axis.

similar, the only difference being a rotation of approximately 17 (3)° around the bond C(8)–C(9) of ring II. The molecules are not planar. The packing of the molecules is illustrated in Fig. 3. No intermolecular distances are less than the sum of the van der Waals radii of the atoms involved.

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The Structure of 2',3'-O-(Tetraisopropyl-1,3-disiloxanediyl)cytidine at 97 K*

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Abstract. C₂₁H₃₉N₃O₆Si₂, orthorhombic, *P*2₁2₁2₁, *a* = 8.649 (2), *b* = 23.301 (4), *c* = 26.902 (8) Å at 293 K and *a* = 8.630 (4), *b* = 23.082 (5), *c* = 26.301 (9) Å at 97 K; *Z* = 8, *D*_c = 1.23 Mg m⁻³ at 293 K; *R*_w(*R*) = 0.028 (0.045) for 2830 reflexions [2323 with *I* > 2σ(*I*)]. The structure was solved by direct methods. The two independent ribose rings display a C(2')-endo conformation; pseudorotation angles *P* = 160.3 (2) and 163.2 (1)° and φ_{max} = 40.5 (3) and 41.0 (3)°, respectively. Both O(5') atoms are *gauche-gauche* oriented with respect to O(1') and C(3'). The two cytosine bases are *anti* with respect to the sugar rings: χ_{CN} = 39.9 (5) and 55.6 (5)°. The two independent molecules form dimeric units by means of hydrogen bonds N(3A)⋯H–N(4B) [2.992 (5) Å] and N(4A)–H⋯N(3B) [2.967 (5) Å]. The cytosine carbonyl oxygens serve as acceptors in hydrogen bonds O(5'A)–H⋯O(2A) [2.696 (4) Å] and O(5'B)–H⋯O(2B) [2.718 (4) Å] connecting neighbouring molecules in the direction [100].

Introduction. As a follow-up to the structure determination of 3',5'-O-(tetraisopropyl-1,3-disiloxanediyl)-

cytidine (Verdegaal, de Kok, Westerink, van Boom & Romers, 1981) we now report the crystal structure of 2',3'-O-(tetraisopropyl-1,3-disiloxanediyl)cytidine (TIS). The disiloxane group is introduced in order to protect two of the three hydroxyl functions during the synthesis of oligonucleotides (Verdegaal, Jansse, de Rooij & van Boom, 1980). Crystals were grown by evaporation of a solution of TIS in a 1:1 acetonitrile/acetone mixture. A crystal with dimensions 0.35 × 0.15 × 0.15 mm was sealed in a glass capillary and mounted on a Nonius CAD-4 diffractometer. A total of 2830 symmetry-independent reflexions were collected at 97 K, using graphite-monochromated Mo *K*α radiation (λ = 0.71069 Å) and scanning in the range of 2° < θ < 20°. 2323 reflexions had *I* > 2σ(*I*). The intensities were corrected for loss of scattering power (12%) during the exposure period. No absorption correction was applied.

The structure was solved by direct methods using the *MULTAN* 78 program system (Main, 1978). The missing heavy atoms and H atoms were found in Fourier and difference Fourier maps. Full-matrix refinement with anisotropic heavy atoms and isotropic H atoms resulted in *R*_w = [Σ w(*F*_o – |*F*_c|)²/Σ w*F*_o²]^{1/2} = 0.028 and *R* = Σ |*F*_o – |*F*_c||/Σ *F*_o = 0.045 for 2830 reflexions. The weights used are *w* = 1/σ²(*F*_o), the variance σ²(*F*_o) being derived from the measurements.

* Nucleic Acid Constituents. XXII. Part XXI: Mellema, Haasnoot, van Boom & Altona (1981).