been reported by Ahmed, Singh & Barnes (1969), where the lengths of the P–*R* ligands have been found to be influenced by the electronegativity of all the substituents on the phosphazene ring.

(3) In each P–N–P island in *E*, the P–N bond nearest the P atom with an excess of chlorine substituents is 0·03–0·06 Å shorter than the other P–N bond of the same island. This indicates that the electron density in the P–N–P islands is unequally shared between the two P–N bonds, is drawn nearer to the P atom which carries more Cl atoms, and thus pulls the N atom with it closer to that phosphorus.

(4) Despite the observed effects discussed under (1), (2), and (3), the net bonding-electron-density in each P–N–P $\pi$-bond centre remains unchanged. No significant change is observed in the mean of the two P–N bonds of each P–N–P island in the five compounds under consideration.

(5) Comparison of the cyclic angles of the trimeric compounds *A* and *D* to those of the tetrameric compounds *B* and *C* shows that the main difference is in the P–N–P angles, but that the N–P–N angles are maintained close to 120°. This supports the directional nature of the $d_\pi$ orbitals of the P atoms, as described by Dewar, Lucken & Whitehead (1960). However, a substantial reduction of the cyclic N–P–N angles to about 113° appears to be possible as observed in the unit $\equiv$P(NMe$_2$)$_2$ of *E*. The two P–N bonds forming the sides of this angle are the longest (1·606 and 1·608 Å) of any in the rings under consideration. Therefore, this small angle is consistent with an expected lower repulsion between the two elongated bonds which would have lower electron-density distribution.

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# The Crystal and Molecular Structure of 5-Brosyl-3-deoxy-3-*C*-(*R*)-(ethoxycarbonylformamido)methyl-1,2-*O*-isopropylidene-α-D-ribofuranose

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The crystal structure of 5-brosyl-3-deoxy-3-*C*-(ethoxycarbonylformamido)methyl-1,2-*O*-isopropylidene-α-D-ribofuranose has been determined by three-dimensional Patterson and Fourier techniques. The crystals are monoclinic with space group $P2_1$. The unit cell of dimensions $a=10{\cdot}381$, $b=9{\cdot}142$, $c=11{\cdot}850$ Å and $\beta=99{\cdot}92°$ contains two formula units. The final $R$ index for 1474 independent non-zero reflexions was 0·055. The carbon atom on C3 was shown to have *R*-stereochemistry. As a result of intermolecular hydrogen bonding between the N and O atoms of the NHCO-functional group, a continuous chain of these arrangements is observed about a twofold screw axis.

## Introduction

Several investigators have shown interest in the synthesis of various branched-chain carbohydrates (Overend, 1963). There is also a general interest in the amino sugars, partly because they occur as basic constituents of many antibiotics (Fox, Watanabe & Bloch, 1966).

The method by which α-(formylamino)acrylic esters can be prepared from α-metalated isocyanoacetic esters and carbonyl compounds (Schöllkopf, Gerhard

& Schröder, 1969) has been applied to the synthesis of branched-chain carbohydrates from uloses (Brink, Coetzer, Jordaan & Lourens, to be published). Thus 3-deoxy-3-*C*-(ethoxycarbonylformamido)methylene-1,2-*O*-isopropylidene-5-trityl-α-D-ribofuranose was prepared from 1,2-*O*-isopropylidene-5-*O*-trityl-α-D-*erythro*-pentafuranos-3-ulose and ethyl isocyanatosodioacetate. Hydrogenation of the unsaturated carbohydrate derivative and reaction of the purified product with brosyl chloride gave 5-brosyl-3-deoxy-3-*C*-(ethoxycarbonylformamido)methyl-1,2-*O*-isopropylidene-α-D-ribofuranose.

Although chemical and spectroscopic methods served to establish most of the structural features of the brosylate the configuration of the substituent on C-3 [C(14) according to the atomic numbering used in the X-ray analysis as described below] was not known.

## Experimental

Colourless needles of the brosylate were grown from its saturated solution in a 1:1 mixture of acetone and hexane. A single crystal with dimensions of 0·12 × 0·12 × 0·30 mm was selected for the X-ray intensity measurements. Weissenberg, precession and oscillation photographs showed the crystal to be monoclinic with space group $P2_1$ (systematic absence of reflexions $0k0$ with $k$ odd). The crystal data are

Molecular formula: $C_{19}H_{24}NO_9SBr$
M.W. 522·39
$a = 10{\cdot}381\ (3)$ Å
$b = 9{\cdot}142\ (3)$
$c = 11{\cdot}850\ (3)$
$\beta = 99{\cdot}92\ (2)^\circ$
$D_m = 1{\cdot}55$ g.cm$^{-3}$
$D_x = 1{\cdot}56$ (by flotation in a mixture of bromoform and methanol)
$Z = 2$

Unit-cell dimensions were calculated from the accurate spot positions measured on a Hilger & Watts four-circle automatic diffractometer using a least-squares procedure (Busing & Levy, 1967). Intensities for 1879 independent reflexions with $\theta < 25^\circ$ were collected on the four-circle diffractometer with crystal monochromated Mo $K\alpha$ radiation and applying the $\omega$–$2\theta$ scanning technique. Of these intensities 1474 were greater than $3\sigma(I)$, where $\sigma(I)$ is given by $[I_o + I_b]^{1/2}$. $I_o$ is the total number of counts during the peak scan and $I_b$ the number of counts for the background intensity.

The strong 020 reflexion was taken as a reference standard and measured after each set of ten reflexions. A maximum fluctuation of about 3% in the value of the reference intensity was observed during data collection.

Table 1. *Final atomic parameters* (× $10^4$) (*fractional coordinates and anisotropic temperature factors*)

Thermal parameters are of the form $T = \exp\,[-(\beta_{11}h^2 + \beta_{22}k^2 + \beta_{33}l^2 + 2\beta_{12}hk + 2\beta_{13}hl + 2\beta_{23}kl)^2 \times 10^{-4}]$.

(Standard deviations are given in parentheses).

| | $x$ | $y$ | $z$ | $\beta_{11}$ | $\beta_{22}$ | $\beta_{33}$ | $\beta_{12}$ | $\beta_{13}$ | $\beta_{23}$ |
|---|---|---|---|---|---|---|---|---|---|
| Br | 4528 (1) | 10000 | 930 (1) | 128 (2) | 298 (4) | 127 (1) | 16 (3) | 18 (1) | 49 (3) |
| k | 10643 (3) | 10162 (5) | 773 (2) | 118 (3) | 92 (4) | 65 (2) | 10 (4) | 1 (2) | 19 (3) |
| O(1) | 10835 (8) | 9887 (14) | −372 (6) | 159 (10) | 181 (16) | 70 (6) | 42 (13) | 20 (6) | 37 (10) |
| O(2) | 11236 (9) | 8761 (9) | 1464 (8) | 126 (12) | 87 (12) | 77 (8) | 21 (10) | 19 (8) | 4 (7) |
| O(3) | 11181 (11) | 11455 (11) | 1331 (9) | 163 (15) | 117 (15) | 96 (9) | −21 (12) | −27 (9) | 20 (9) |
| O(4) | 10450 (7) | 6567 (10) | 2914 (7) | 67 (8) | 125 (13) | 90 (7) | 16 (9) | −1 (6) | 26 (8) |
| O(5) | 10581 (7) | 4608 (10) | 4141 (6) | 82 (8) | 153 (15) | 90 (6) | 21 (9) | 16 (6) | 28 (8) |
| O(6) | 12717 (6) | 4402 (8) | 4097 (6) | 63 (7) | 103 (11) | 78 (6) | −13 (7) | −4 (5) | 28 (7) |
| O(7) | 14616 (8) | 4706 (13) | 1911 (8) | 98 (9) | 227 (20) | 128 (9) | −13 (12) | 8 (8) | −98 (13) |
| O(8) | 16219 (8) | 6038 (11) | 2941 (8) | 79 (10) | 182 (17) | 124 (9) | −3 (10) | 14 (7) | −47 (10) |
| O(9) | 14471 (8) | 9283 (10) | 4390 (7) | 138 (11) | 82 (12) | 96 (8) | −7 (9) | −17 (7) | −11 (8) |
| N | 14497 (8) | 6799 (10) | 4324 (7) | 71 (10) | 80 (12) | 48 (7) | −8 (9) | 3 (6) | −12 (7) |
| C(1) | 6340 (11) | 9981 (20) | 894 (8) | 126 (14) | 161 (22) | 51 (8) | −7 (21) | 6 (8) | 29 (15) |
| C(2) | 6990 (16) | 8720 (18) | 830 (15) | 121 (22) | 133 (24) | 114 (17) | −10 (19) | −2 (15) | 9 (16) |
| C(3) | 8337 (15) | 8717 (16) | 814 (14) | 111 (19) | 109 (20) | 96 (15) | −5 (16) | −0 (13) | −4 (13) |
| C(4) | 8964 (10) | 10061 (16) | 859 (8) | 114 (12) | 88 (15) | 48 (7) | 9 (17) | −6 (7) | −4 (12) |
| C(5) | 8307 (20) | 11361 (16) | 894 (14) | 190 (28) | 98 (20) | 88 (14) | 2 (18) | −14 (15) | −3 (13) |
| C(6) | 6934 (18) | 11347 (19) | 919 (15) | 127 (23) | 174 (28) | 103 (16) | 13 (20) | 9 (15) | 8 (17) |
| C(7) | 11499 (11) | 8867 (14) | 2716 (9) | 108 (14) | 109 (17) | 53 (9) | 25 (13) | −8 (8) | 11 (10) |
| C(8) | 11678 )10) | 7336 (13) | 3157 (9) | 64 (12) | 99 (15) | 57 (9) | −10 (11) | −8 (8) | 3 (9) |
| C(9) | 10703 (9) | 5065 (17) | 3035 (8) | 69 (10) | 118 (16) | 62 (8) | −2 (16) | −3 (7) | 10 (13) |
| C(10) | 11733 (11) | 3848 (15) | 4647 (10) | 77 (13) | 130 (30) | 87 (11) | −1 (14) | −7 (9) | 19 (12) |
| C(11) | 12038 (13) | 4270 (18) | 5923 (10) | 149 (17) | 225 (27) | 59 (9) | 5 (18) | −5 (10) | −13 (14) |
| C(12) | 11582 (13) | 2212 (15) | 4450 (11) | 145 (17) | 98 (18) | 95 (12) | −37 (15) | 13 (12) | 12 (12) |
| C(13) | 12163 (9) | 4808 (14) | 2958 (8) | 54 (10) | 119 (18) | 50 (7) | −25 (12) | −10 (7) | 8 (10) |
| C(14) | 12608 (9) | 6342 (12) | 2651 (8) | 49 (10) | 83 (14) | 55 (8) | 6 (11) | 2 (7) | 6 (9) |
| C(15) | 14085 (9) | 6751 (13) | 3067 (8) | 52 (10) | 99 (15) | 51 (8) | 0 (10) | 6 (7) | −2 (9) |
| C(16) | 14947 (11) | 5684 (14) | 2565 (10) | 81 (14) | 133 (19) | 64 (10) | −24 (13) | 4 (9) | 5 (11) |
| C(17) | 17212 (10) | 5113 (22) | 2536 (11) | 76 (12) | 189 (23) | 137 (13) | 21 (20) | 41 (10) | −39 (20) |
| C(18) | 17496 (12) | 3760 (18) | 3285 (12) | 104 (15) | 175 (24) | 111 (13) | 20 (17) | 25 (12) | 15 (15) |
| C(19) | 14705 (10) | 8076 (15) | 4849 (10) | 58 (13) | 116 (19) | 89 (11) | −12 (13) | 2 (9) | −12 (13) |

Background corrections were carried out by making slow scans of intensity against $\theta$. These scans were taken parallel to central lattice rows, but shifted from the lattice rows far enough in $\varphi$ in order not to overlap with the tails of diffraction peaks. The usual corrections were made for Lorentz and polarization factors. Absorption corrections were obviated on account of the low value for $\mu R$ ($\sim$0·10).

## Structure refinement

The fractional coordinates for the heavy atom (Br) were readily deduced from a three-dimensional Patterson map. All the remaining atomic positions (excluding hydrogen) were found on a subsequent three-dimensional Fourier map phased by the bromine atom.

The trial structure was refined using the full-matrix least-squares program *ORFLS* (Busing, Martin & Levy, 1962) which minimizes the function $\sum\omega(|F_o|-|kF_c|)^2$. Unit weights were assigned to the 1474 observed reflexions while the others (unobserved) were given zero weight. Refinement with individual isotropic thermal parameters for all the atoms gave an $R$ index $[R=(\sum||F_o|-|F_c||)/\sum|F_o|]$ of 0·105. The $R$ value was reduced to 0·055 on refinement with individual anisotropic thermal parameters for all the atoms. The form factors used were those of Hansen, Herman, Lea & Skillman (1964).

A list of the refined positional and thermal parameters appears in Table 1. The crystallographic program *ORFFE* of Busing, Martin & Levy (1964) was used to calculate the values for the interatomic distances and bond angles listed in Tables 2 and 3. Observed and calculated structure factors are given in Table 4.

Table 2. *Bond distances* (Å)

(Standard deviations in parentheses.)

| | | | |
|---|---|---|---|
| C=O | | $sp^2$C–$sp^3$C | |
| C(16)–O(7) | 1·19 (1) Å | C(15)–C(16) | 1·51 (2) Å |
| C(19)–O(9) | 1·23 (1) | | |
| $sp^2$C–O | | C–C | |
| C(16)–O(8) | 1·36 (1) | C(7)—C(8) | 1·49 (2) |
| | | C(8)—C(14) | 1·52 (1) |
| $sp^3$C–O | | C(9)—C(13) | 1·55 (1) |
| C(7)—O(2) | 1·46 (1) | C(10)–C(11) | 1·54 (2) |
| C(8)—O(4) | 1·44 (1) | C(10)–C(12) | 1·52 (2) |
| C(9)—O(4) | 1·40 (2) | C(13)–C(14) | 1·54 (2) |
| C(9)—O(5) | 1·40 (1) | C(14)–C(15) | 1·57 (1) |
| C(10)–O(5) | 1·42 (1) | C(17)–C(18) | 1·52 (2) |
| C(10)–O(6) | 1·40 (1) | | |
| C(13)–O(6) | 1·42 (1) | | |
| C(17)–O(8) | 1·48 (2) | C(19)–N | 1·32 (1) |
| | | C(15)–N | 1·48 (1) |
| C=C(arom) | | | |
| C(1)–C(2) | 1·35 (2) | S–O(1) | 1·43 (1) |
| C(2)–C(3) | 1·40 (2) | S–O(2) | 1·58 (1) |
| C(3)–C(4) | 1·39 (2) | S–O(3) | 1·42 (1) |
| C(4)–C(5) | 1·37 (2) | S–C(4) | 1·77 (1) |
| C(5)–C(6) | 1·43 (3) | | |
| C(6)–C(1) | 1·39 (2) | C(1)–Br | 1·89 (1) |

## Discussion

A stereoscopic drawing of the molecule is shown in Fig. 1 while the structural formula (I) gives the atomic numbering used. It is interesting to note that the position in the unit cell of the *p*-bromophenylsulphonyl group is almost invariant with respect to the choice of enantiomeric form. This is vizualized by considering a mirror plane ($x$, 1·0, $z$) passing through the atoms Br, S, C(1), C(4), and O(1) to within 0·16 Å of their respective centres and reflecting the atoms C(2), C(3) and O(2) into C(5), C(6) and O(3), respectively (Table 1).

Table 3. *Bond angles*

(Standard deviations in parentheses.)

| | | | |
|---|---|---|---|
| O(1)—S——O(2) | 104·0 (6)° | C(7)—C(8)—C(14) | 118·1 (9)° |
| O(1)—k——O(3) | 118·8 (7) | O(4)—C(8)—C(14) | 103·2 (8) |
| O(1)—k——C(4) | 110·2 (5) | O(4)—C(9)—O(5) | 110·0 (10) |
| O(2)—S——O(3) | 110·2 (4) | O(4)—C(9)—C(13) | 108·0 (9) |
| O(2)—S——C(4) | 103·2 (6) | O(5)—C(9)—C(13) | 104·9 (8) |
| O(3)—S——C(4) | 109·3 (7) | O(5)—C(10)–C(12) | 111·1 (10) |
| S——O(2)–C(7) | 117·2 (7) | O(5)—C(10)–C(11) | 107·9 (10) |
| C(8)—O(4)–C(9) | 108·3 (8) | O(5)—C(10)–O(6) | 104·8 (9) |
| C(9)—O(5)–C(10) | 109·5 (8) | O(6)—C(10)–C(11) | 108·9 (10) |
| C(10)–O(6)–C(13) | 109·2 (7) | O(6)—C(10)–C(12) | 110·5 (11) |
| C(16)–O(8)–C(17) | 116·8 (9) | C(11)–C(10)–C(12) | 113·3 (12) |
| C(15)–N——C(19) | 119·7 (9) | O(6)—C(13)–C(9) | 102·9 (7) |
| | | O(6)—C(13)–C(14) | 111·9 (8) |
| Br——C(1)–C(2) | 121·4 (12) | C(9)—C(13)–C(14) | 102·2 (9) |
| Br——C(1)–C(6) | 115·5 (12) | C(13)–C(14)–C(8) | 102·4 (8) |
| C(2)—C(1)–C(6) | 123·0 (11) | C(13)–C(14)–C(15) | 117·3 (8) |
| C(1)—C(2)–C(3) | 120·9 (14) | C(8)—C(14)–C(15) | 112·6 (8) |
| C(2)—C(3)–C(4) | 117·4 (13) | C(14)–C(15)–N | 114·8 (7) |
| C(3)—C(4)–C(5) | 122·4 (10) | C(14)–C(15)–C(16) | 109·5 (8) |
| C(3)—C(4)–C(5) | 120·3 (10) | N——C(15)–C(16) | 109·1 (8) |
| C(5)—C(4)–S | 117·1 (12) | C(15)–C(16)–O(7) | 127·9 (10) |
| C(4)—C(5)–C(6) | 119·6 (14) | C(15)–C(16)–O(8) | 108·9 (10) |
| C(1)—C(6)–C(5) | 116·6 (14) | O(7)—C(16)–O(8) | 123·1 (12) |
| O(2)—C(7)–C(8) | 106·4 (9) | O(8)—C(17)–C(18) | 110·6 (10) |
| C(7)—C(8)–O(4) | 109·6 (8) | O(9)—C(19)–N | 125·3 (10) |

Table 4. *Observed and calculated structure factors*

The columns are $k$, $l$, $F_{\text{obs}}\times 10$, $F_{\text{calc}}\times 10$.

| | | | |
|---|---|---|---|
| H = 0 | | | |
| 0 | 1 | 449 | 545 |
| 0 | 2 | 246 | 266 |
| 0 | 3 | 96 | 69 |
| 0 | 4 | 460 | 449 |
| 0 | 5 | 1088 | 1077 |
| 0 | 6 | 902 | 884 |
| 0 | 7 | 130 | 101 |
| 0 | 8 | 333 | 339 |
| 0 | 9 | 44 | 5 |
| 0 | 10 | 210 | 216 |
| 0 | 11 | 132 | 126 |
| 0 | 12 | 141 | 141 |
| 0 | 13 | 72 | 69 |
| 1 | 1 | 477 | 467 |
| 1 | 2 | 1622 | 1670 |
| 1 | 3 | 1573 | 1525 |
| 1 | 4 | 569 | 541 |
| 1 | 5 | 140 | 153 |
| 1 | 6 | 216 | 211 |
| 1 | 7 | 445 | 446 |
| 1 | 8 | 551 | 537 |
| 1 | 9 | 209 | 207 |
| 1 | 10 | 192 | 196 |
| 1 | 11 | 113 | 112 |
| 1 | 13 | 50 | 54 |
| 2 | 0 | 1463 | 1526 |
| 2 | 1 | 919 | 915 |
| 2 | 2 | 481 | 538 |
| 2 | 3 | 156 | 200 |
| 2 | 4 | 360 | 375 |
| 2 | 5 | 459 | 449 |
| 2 | 6 | 647 | 645 |
| 2 | 7 | 228 | 247 |
| 2 | 8 | 144 | 136 |
| 2 | 9 | 37 | 58 |
| 2 | 10 | 142 | 162 |
| 2 | 11 | 130 | 131 |
| 2 | 12 | 128 | 115 |
| 3 | 1 | 152 | 141 |
| 3 | 2 | 134 | 166 |
| 3 | 3 | 415 | 420 |
| 3 | 4 | 493 | 495 |
| 3 | 5 | 227 | 244 |
| 3 | 6 | 174 | 155 |
| 3 | 7 | 168 | 170 |
| 3 | 8 | 302 | 312 |
| 3 | 9 | 271 | 259 |
| 3 | 10 | 133 | 133 |
| 3 | 11 | 101 | 91 |
| 3 | 12 | 59 | 46 |
| 3 | 13 | 46 | 44 |
| 4 | 0 | 596 | 572 |
| 4 | 1 | 502 | 486 |
| 4 | 2 | 341 | 334 |
| 4 | 3 | 96 | 92 |
| 4 | 4 | 165 | 163 |
| 4 | 5 | 231 | 217 |
| 4 | 6 | 265 | 254 |
| 4 | 7 | 314 | 323 |
| 4 | 8 | 247 | 253 |
| 4 | 10 | 132 | 124 |
| 4 | 11 | 67 | 67 |
| 4 | 12 | 82 | 71 |
| 5 | 1 | 190 | 197 |
| 5 | 2 | 297 | 281 |
| 5 | 3 | 273 | 283 |
| 5 | 4 | 226 | 216 |
| 5 | 5 | 137 | 141 |
| 5 | 7 | 180 | 175 |
| 5 | 8 | 275 | 269 |
| 5 | 9 | 96 | 99 |
| 5 | 10 | 156 | 142 |
| 6 | 0 | 413 | 439 |
| 6 | 1 | 401 | 417 |
| 6 | 2 | 92 | 90 |
| 6 | 4 | 131 | 129 |
| 6 | 5 | 330 | 330 |
| 6 | 6 | 147 | 152 |
| 6 | 7 | 116 | 109 |
| 6 | 8 | 94 | 92 |
| 6 | 9 | 64 | 65 |
| 6 | 10 | 95 | 80 |
| 6 | 11 | 78 | 74 |
| 7 | 1 | 155 | 167 |
| 7 | 2 | 363 | 375 |
| 7 | 3 | 295 | 296 |
| 7 | 4 | 170 | 190 |
| 7 | 6 | 42 | 38 |
| 7 | 7 | 179 | 183 |
| 7 | 8 | 146 | 137 |
| 7 | 9 | 110 | 104 |
| 8 | 0 | 283 | 268 |
| 8 | 1 | 304 | 306 |
| 8 | 3 | 41 | 39 |
| 8 | 4 | 73 | 77 |
| 8 | 5 | 257 | 252 |
| 8 | 6 | 95 | 85 |
| 8 | 7 | 77 | 83 |
| 9 | 1 | 95 | 94 |
| 9 | 2 | 106 | 90 |
| 9 | 3 | 170 | 175 |
| 9 | 4 | 51 | 46 |
| 9 | 5 | 75 | 73 |
| 9 | 7 | 78 | 67 |
| 10 | 0 | 48 | 47 |
| 10 | 2 | 52 | 60 |
| 10 | 5 | 60 | 55 |
| H = 1 | | | |
| 0 | -12 | 70 | 56 |
| 0 | -11 | 84 | 91 |
| 0 | -10 | 94 | 83 |
| 0 | -9 | 181 | 187 |
| 0 | -8 | 91 | 89 |
| 0 | -7 | 193 | 196 |
| 0 | -6 | 167 | 141 |
| 0 | -5 | 145 | 153 |
| 0 | -4 | 327 | 287 |
| 0 | -3 | 287 | 329 |
| 0 | -2 | 398 | 379 |
| 0 | -1 | 408 | 474 |
| 0 | 0 | 139 | 174 |
| 0 | 1 | 348 | 372 |
| 0 | 2 | 319 | 301 |
| 0 | 3 | 456 | 366 |
| 0 | 4 | 351 | 313 |
| 0 | 5 | 75 | 77 |
| 0 | 6 | 499 | 492 |
| 0 | 7 | 54 | 67 |
| 0 | 8 | 148 | 162 |
| 0 | 9 | 133 | 159 |
| 0 | 10 | 63 | 79 |
| 0 | 11 | 101 | 96 |
| 0 | 13 | 80 | 100 |
| 1 | -11 | 109 | 115 |
| 1 | -10 | 45 | 47 |
| 1 | -9 | 40 | 42 |
| 1 | -8 | 71 | 60 |
| 1 | -7 | 140 | 122 |
| 1 | -6 | 35 | 12 |
| 1 | -5 | 58 | 59 |
| 1 | -4 | 343 | 306 |
| 1 | -3 | 675 | 695 |
| 1 | -2 | 549 | 500 |
| 1 | -1 | 272 | 316 |
| 1 | 0 | 228 | 218 |
| 1 | 1 | 348 | 332 |
| 1 | 2 | 255 | 210 |
| 1 | 3 | 561 | 571 |
| 1 | 4 | 258 | 254 |
| 1 | 5 | 424 | 400 |
| 1 | 6 | 296 | 307 |
| 1 | 7 | 38 | 19 |
| 1 | 8 | 166 | 161 |
| 1 | 9 | 82 | 80 |
| 1 | 10 | 161 | 180 |
| 1 | 11 | 83 | 100 |
| 1 | 12 | 52 | 67 |
| 2 | -11 | 99 | 89 |
| 2 | -10 | 117 | 120 |
| 2 | -9 | 228 | 226 |
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| 2 | 7 | 174 | 157 |
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| 3 | 8 | 77 | 75 |
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| 5 | 5 | 188 | 189 |
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| 6 | 7 | 128 | 117 |
| 6 | 8 | 66 | 66 |
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| 0 | 7 | 195 | 190 |
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| 2 | 7 | 253 | 238 |
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| 2 | 11 | 132 | 122 |
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| 4 | -5 | 178 | 168 |
| 4 | -4 | 229 | 246 |
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| 4 | -1 | 287 | 294 |
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| 4 | 5 | 332 | 345 |
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| 5 | -8 | 49 | 59 |
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| 5 | 7 | 177 | 183 |
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| 9 | 1 | 126 | 117 |
| 9 | 2 | 59 | 64 |
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| 0 | 10 | 47 | 36 |
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| 1 | -5 | 295 | 299 |
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| 2 | -11 | 136 | 135 |
| 2 | -10 | 159 | 157 |
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| 2 | -6 | 71 | 72 |
| 2 | -5 | 241 | 243 |
| 2 | -4 | 276 | 276 |
| 2 | -3 | 377 | 373 |
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| 2 | -1 | 180 | 177 |
| 2 | 0 | 276 | 299 |
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| 2 | 3 | 254 | 230 |
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| 2 | 6 | 96 | 108 |
| 2 | 7 | 198 | 198 |
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| 2 | 9 | 126 | 118 |
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| 3 | -12 | 56 | 43 |
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| 3 | -1 | 98 | 105 |
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| 3 | 2 | 325 | 317 |
| 3 | 3 | 174 | 167 |
| 3 | 4 | 126 | 131 |
| 3 | 5 | 220 | 221 |
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| 3 | 8 | 115 | 122 |
| 3 | 9 | 75 | 80 |
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| 4 | -8 | 161 | 152 |
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| 4 | 1 | 150 | 162 |
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| 4 | 3 | 182 | 188 |
| 4 | 4 | 133 | 134 |
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| 5 | -8 | 119 | 123 |
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| 5 | -1 | 208 | 218 |
| 5 | 1 | 340 | 355 |
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| 5 | 4 | 55 | 66 |
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| 5 | 8 | 94 | 100 |
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| 6 | -1 | 222 | 223 |
| 6 | 0 | 111 | 115 |
| 6 | 1 | 268 | 270 |
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| 6 | 3 | 53 | 43 |
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| 6 | 6 | 88 | 99 |
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| 7 | 5 | 108 | 102 |
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| 8 | -6 | 62 | 62 |
| 8 | -4 | 146 | 151 |
| 8 | -3 | 97 | 98 |
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| 8 | -1 | 91 | 87 |
| 8 | 1 | 106 | 106 |
| 8 | 2 | 107 | 108 |
| 8 | 3 | 70 | 60 |
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| 8 | 5 | 75 | 74 |
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| 1 | -5 | 277 | 287 |
| 1 | -4 | 226 | 249 |
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| 1 | 3 | 122 | 143 |
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| 2 | 2 | 387 | 375 |
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| 2 | 6 | 103 | 97 |
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| 3 | 4 | 292 | 295 |
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| 4 | -1 | 152 | 136 |
| 4 | 0 | 126 | 148 |
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| 5 | 4 | 117 | 119 |
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| 5 | 8 | 53 | 49 |
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| 8 | -2 | 83 | 80 |
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| 8 | 4 | 84 | 79 |
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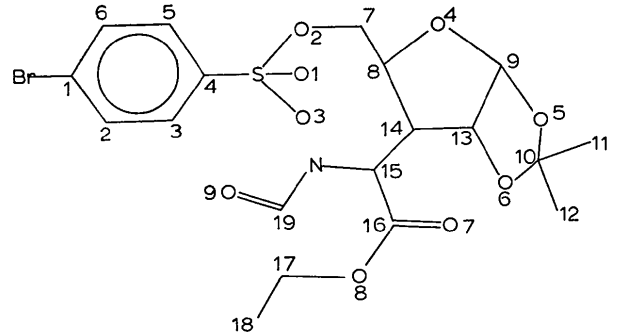
| 0 | -5 | 296 | 306 |
| 0 | -4 | 399 | 374 |
| 0 | -3 | 775 | 788 |
| 0 | -2 | 158 | 167 |
| 0 | -1 | 252 | 230 |
| 0 | 0 | 323 | 317 |
| 0 | 1 | 893 | 848 |
| 0 | 2 | 401 | 376 |
| 0 | 3 | 379 | 375 |
| 0 | 4 | 153 | 168 |
| 0 | 5 | 287 | 288 |
| 0 | 6 | 215 | 225 |
| 0 | 7 | 238 | 264 |
| 0 | 8 | 58 | 44 |
| 0 | 9 | 308 | 310 |
| 0 | 10 | 154 | 144 |
| 0 | -12 | 50 | 55 |
| 1 | -11 | 133 | 135 |
| 1 | -10 | 80 | 65 |
| 1 | -9 | 104 | 113 |
| 1 | -8 | 160 | 158 |
| 1 | -7 | 351 | 364 |
| 1 | -6 | 428 | 444 |
| 1 | -5 | 522 | 527 |
| 1 | -4 | 51 | 63 |
| 1 | -2 | 701 | 697 |
| 1 | -1 | 699 | 675 |
| 1 | 0 | 351 | 354 |
| 1 | 1 | 494 | 510 |
| 1 | 2 | 280 | 280 |
| 1 | 3 | 139 | 150 |
| 1 | 4 | 415 | 416 |
| 1 | 5 | 284 | 289 |
| 1 | 6 | 130 | 116 |
| 1 | 7 | 345 | 354 |
| 1 | 9 | 132 | 148 |
| 1 | 10 | 66 | 73 |
| 1 | 11 | 87 | 90 |
| 2 | -13 | 47 | 29 |
| 2 | -11 | 48 | 45 |
| 2 | -10 | 55 | 61 |
| 2 | -9 | 258 | 257 |
| 2 | -8 | 133 | 127 |
| 2 | -7 | 117 | 120 |
| 2 | -6 | 83 | 84 |
| 2 | -5 | 106 | 117 |
| 2 | -4 | 261 | 281 |
| 2 | -3 | 426 | 455 |
| 2 | -2 | 301 | 311 |
| 2 | -1 | 328 | 325 |
| 2 | 0 | 258 | 262 |
| 2 | 1 | 561 | 563 |
| 2 | 2 | 286 | 291 |
| 2 | 3 | 555 | 572 |
| 2 | 4 | 259 | 253 |
| 2 | 5 | 72 | 67 |
| 2 | 6 | 237 | 245 |
| 2 | 7 | 131 | 145 |
| 2 | 8 | 196 | 205 |
| 2 | 9 | 203 | 200 |
| 2 | 10 | 78 | 78 |
| 3 | -12 | 72 | 63 |
| 3 | -11 | 88 | 80 |
| 3 | -10 | 50 | 61 |
| 3 | -9 | 90 | 78 |
| 3 | -8 | 116 | 111 |
| 3 | -7 | 104 | 107 |
| 3 | -6 | 269 | 270 |
| 3 | -5 | 316 | 328 |
| 3 | -4 | 120 | 133 |
| 3 | -2 | 114 | 124 |
| 3 | -1 | 230 | 221 |
| 3 | 0 | 449 | 441 |
| 3 | 1 | 545 | 515 |
| 3 | 2 | 72 | 80 |
| 3 | 3 | 220 | 221 |
| 3 | 4 | 188 | 171 |
| 3 | 5 | 290 | 286 |
| 3 | 6 | 330 | 327 |
| 3 | 8 | 51 | 59 |
| 3 | 10 | 66 | 61 |
| 3 | 11 | 109 | 106 |
| 4 | -10 | 81 | 82 |
| 4 | -9 | 120 | 124 |
| 4 | -8 | 93 | 97 |
| 4 | -7 | 59 | 34 |
| 4 | -4 | 75 | 70 |
| 4 | -3 | 276 | 266 |
| 4 | -2 | 305 | 303 |
| 4 | -1 | 173 | 176 |
| 4 | 0 | 138 | 143 |
| 4 | 1 | 198 | 196 |
| 4 | 2 | 387 | 376 |
| 4 | 3 | 349 | 348 |
| 4 | 4 | 236 | 227 |
| 4 | 5 | 166 | 166 |
| 4 | 6 | 84 | 89 |
| 4 | 7 | 122 | 122 |
| 4 | 8 | 172 | 160 |
| 4 | 9 | 113 | 113 |
| 5 | -10 | 55 | 58 |
| 5 | -9 | 92 | 105 |
| 5 | -7 | 155 | 170 |
| 5 | -6 | 144 | 148 |
| 5 | -5 | 189 | 190 |
| 5 | -4 | 83 | 71 |
| 5 | -3 | 151 | 164 |
| 5 | -2 | 75 | 78 |
| 5 | -1 | 290 | 299 |
| 5 | 0 | 296 | 306 |
| 5 | 1 | 294 | 275 |
| 5 | 2 | 82 | 88 |
| 5 | 3 | 89 | 86 |
| 5 | 4 | 96 | 112 |
| 5 | 5 | 197 | 200 |
| 5 | 6 | 153 | 154 |
| 5 | 7 | 92 | 90 |
| 5 | 8 | 67 | 77 |
| 5 | 10 | 80 | 83 |
| 6 | -10 | 45 | 52 |
| 6 | -9 | 87 | 102 |
| 6 | -8 | 79 | 91 |
| 6 | -6 | 88 | 94 |
| 6 | -5 | 87 | 99 |
| 6 | -4 | 182 | 197 |
| 6 | -3 | 153 | 155 |
| 6 | -2 | 193 | 185 |
| 6 | -1 | 237 | 232 |
| 6 | 0 | 93 | 105 |
| 6 | 1 | 224 | 232 |
| 6 | 2 | 180 | 192 |
| 6 | 3 | 126 | 129 |
| 6 | 4 | 141 | 144 |
| 6 | 5 | 98 | 99 |
| 6 | 6 | 94 | 90 |
| 6 | 7 | 46 | 43 |
| 6 | 8 | 89 | 89 |
| 6 | 9 | 87 | 99 |
| 7 | -7 | 153 | 157 |
| 7 | -6 | 162 | 156 |
| 7 | -5 | 46 | 32 |
| 7 | -4 | 104 | 105 |
| 7 | -3 | 69 | 88 |
| 7 | -2 | 169 | 174 |
| 7 | -1 | 181 | 189 |
| 7 | 0 | 86 | 98 |
| 7 | 1 | 68 | 62 |
| 7 | 2 | 97 | 87 |
| 7 | 3 | 108 | 107 |
| 7 | 4 | 88 | 90 |
| 7 | 6 | 100 | 104 |
| 7 | 7 | 73 | 71 |
| 8 | -7 | 61 | 50 |
| 8 | -5 | 72 | 78 |
| 8 | -4 | 177 | 173 |
| 8 | -2 | 124 | 128 |
| 8 | -1 | 61 | 63 |
| 8 | 0 | 52 | 65 |
| 8 | 1 | 89 | 83 |
| 8 | 2 | 137 | 145 |
| 8 | 4 | 104 | 97 |
| 9 | -6 | 52 | 52 |
| 9 | -2 | 79 | 83 |
| 9 | 0 | 102 | 100 |
| 9 | 3 | 48 | 60 |
| H = 6 | | | |
| 0 | -12 | 75 | 75 |
| 0 | -10 | 70 | 62 |
| 0 | -8 | 154 | 152 |
| 0 | -7 | 73 | 69 |
| 0 | -6 | 204 | 204 |
| 0 | -4 | 227 | 237 |
| 0 | -3 | 497 | 507 |
| 0 | -2 | 245 | 240 |
| 0 | -1 | 294 | 307 |
| 0 | 0 | 248 | 260 |
| 0 | 1 | 153 | 154 |
| 0 | 2 | 205 | 195 |
| 0 | 3 | 166 | 152 |
| 0 | 4 | 72 | 75 |
| 0 | 5 | 47 | 14 |
| 0 | 7 | 96 | 118 |
| 0 | 8 | 55 | 46 |
| 0 | 9 | 45 | 44 |
| 1 | -11 | 54 | 53 |
| 1 | -10 | 133 | 134 |
| 1 | -9 | 60 | 56 |
| 1 | -8 | 201 | 197 |
| 1 | -7 | 94 | 105 |
| 1 | -6 | 135 | 143 |
| 1 | -5 | 237 | 237 |
| 1 | -4 | 173 | 157 |
| 1 | -3 | 138 | 142 |
| 1 | -2 | 170 | 176 |
| 1 | -1 | 219 | 213 |
| 1 | 0 | 358 | 363 |
| 1 | 1 | 109 | 112 |
| 1 | 2 | 229 | 248 |
| 1 | 3 | 52 | 54 |
| 1 | 4 | 115 | 126 |
| 1 | 5 | 174 | 186 |
| 1 | 6 | 81 | 94 |
| 1 | 7 | 49 | 56 |
| 1 | 8 | 80 | 78 |
| 2 | -13 | 72 | 62 |
| 2 | -12 | 76 | 68 |
| 2 | -11 | 64 | 47 |
| 2 | -10 | 41 | 40 |
| 2 | -9 | 78 | 75 |
| 2 | -8 | 194 | 194 |
| 2 | -7 | 140 | 138 |
| 2 | -6 | 130 | 140 |
| 2 | -5 | 176 | 165 |
| 2 | -4 | 117 | 109 |
| 2 | -3 | 271 | 249 |
| 2 | -2 | 371 | 383 |
| 2 | -1 | 183 | 186 |
| 2 | 0 | 337 | 331 |
| 2 | 1 | 64 | 77 |
| 2 | 2 | 70 | 65 |
| 2 | 3 | 91 | 91 |
| 2 | 4 | 126 | 121 |
| 2 | 5 | 95 | 74 |
| 2 | 6 | 94 | 82 |
| 2 | 7 | 44 | 39 |
| 2 | 9 | 77 | 63 |
| 2 | 10 | 44 | 37 |
| 3 | -11 | 110 | 95 |
| 3 | -10 | 141 | 135 |
| 3 | -9 | 169 | 172 |
| 3 | -8 | 92 | 101 |
| 3 | -7 | 55 | 50 |
| 3 | -6 | 188 | 185 |
| 3 | -5 | 170 | 179 |
| 3 | -4 | 237 | 237 |
| 3 | -3 | 109 | 111 |
| 3 | -2 | 56 | 58 |
| 3 | -1 | 38 | 38 |
| 3 | 0 | 194 | 188 |
| 3 | 1 | 205 | 207 |
| 3 | 2 | 220 | 201 |
| 3 | 3 | 127 | 136 |
| 3 | 4 | 58 | 64 |
| 3 | 5 | 60 | 67 |
| 3 | 6 | 194 | 200 |
| 3 | 7 | 129 | 120 |
| 3 | 8 | 111 | 121 |
| 4 | -12 | 66 | 61 |
| 4 | -11 | 109 | 110 |
| 4 | -9 | 102 | 104 |
| 4 | -8 | 174 | 175 |
| 4 | -7 | 127 | 128 |
| 4 | -6 | 134 | 135 |
| 4 | -5 | 136 | 129 |
| 4 | -4 | 96 | 88 |
| 4 | -3 | 212 | 208 |
| 4 | -2 | 287 | 291 |
| 4 | -1 | 155 | 140 |
| 4 | 0 | 206 | 208 |
| 4 | 1 | 221 | 222 |
| 4 | 3 | 170 | 166 |
| 4 | 4 | 133 | 132 |
| 4 | 5 | 84 | 81 |
| 4 | 6 | 175 | 173 |
| 4 | 7 | 84 | 78 |
| 4 | 8 | 65 | 57 |
| 5 | -11 | 48 | 48 |
| 5 | -10 | 136 | 131 |
| 5 | -9 | 101 | 93 |
| 5 | -8 | 88 | 79 |
| 5 | -7 | 65 | 66 |
| 5 | -6 | 85 | 103 |
| 5 | -5 | 157 | 141 |
| 5 | -4 | 122 | 105 |
| 5 | -3 | 80 | 92 |
| 5 | -2 | 170 | 167 |
| 5 | -1 | 106 | 106 |
| 5 | 0 | 134 | 130 |
| 5 | 1 | 110 | 108 |
| 5 | 2 | 101 | 95 |
| 5 | 3 | 129 | 128 |
| 5 | 4 | 137 | 127 |
| 5 | 5 | 168 | 174 |
| 5 | 7 | 89 | 92 |
| 5 | 8 | 51 | 55 |
| 6 | -10 | 66 | 55 |
| 6 | -9 | 47 | 48 |
| 6 | -8 | 116 | 113 |
| 6 | -7 | 78 | 77 |
| 6 | -6 | 42 | 29 |
| 6 | -5 | 154 | 166 |
| 6 | -4 | 73 | 77 |
| 6 | -3 | 127 | 136 |
| 6 | -2 | 106 | 95 |
| 6 | -1 | 41 | 47 |
| 6 | 0 | 208 | 191 |
| 6 | 1 | 143 | 132 |
| 6 | 2 | 139 | 146 |
| 6 | 3 | 40 | 26 |
| 6 | 5 | 60 | 52 |
| 6 | 6 | 73 | 74 |
| 6 | 7 | 73 | 58 |
| 7 | -9 | 51 | 32 |
| 7 | -8 | 67 | 65 |
| 7 | -7 | 80 | 81 |
| 7 | -5 | 63 | 61 |
| 7 | -4 | 60 | 48 |
| 7 | -3 | 114 | 100 |
| 7 | -2 | 179 | 161 |
| 7 | -1 | 82 | 82 |
| 7 | 0 | 69 | 60 |
| 7 | 2 | 47 | 67 |
| 7 | 3 | 118 | 117 |
| 7 | 4 | 64 | 68 |
| 7 | 5 | 69 | 61 |
| 7 | 6 | 51 | 65 |
| 7 | 7 | 46 | 36 |
| 8 | -7 | 46 | 44 |
| 8 | -5 | 104 | 97 |
| 8 | -4 | 77 | 69 |
| 8 | -2 | 46 | 52 |
| 8 | -1 | 47 | 46 |
| 8 | 1 | 57 | 54 |
| 8 | 2 | 80 | 70 |
| 9 | -3 | 99 | 90 |
| 9 | 1 | 44 | 24 |
| H = 7 | | | |
| 0 | -10 | 65 | 59 |
| 0 | -9 | 45 | 34 |
| 0 | -8 | 82 | 89 |
| 0 | -7 | 50 | 52 |
| 0 | -6 | 131 | 146 |
| 0 | -5 | 147 | 150 |
| 0 | -4 | 39 | 28 |
| 0 | -3 | 51 | 36 |
| 0 | -2 | 69 | 72 |
| 0 | -1 | 258 | 266 |
| 0 | 0 | 84 | 84 |
| 0 | 1 | 221 | 227 |
| 0 | 3 | 288 | 290 |
| 0 | 4 | 169 | 175 |
| 0 | 5 | 127 | 119 |
| 0 | 6 | 43 | 49 |
| 0 | 8 | 135 | 136 |
| 0 | 9 | 123 | 137 |
| 1 | -7 | 127 | 129 |
| 1 | -6 | 89 | 80 |
| 1 | -5 | 156 | 168 |
| 1 | -4 | 204 | 214 |
| 1 | -2 | 121 | 118 |
| 1 | -1 | 90 | 83 |
| 1 | 0 | 94 | 89 |
| 1 | 1 | 275 | 273 |
| 1 | 2 | 155 | 159 |
| 1 | 3 | 44 | 41 |
| 1 | 4 | 48 | 47 |
| 1 | 5 | 128 | 130 |
| 1 | 6 | 235 | 249 |
| 1 | 7 | 66 | 72 |
| 1 | 10 | 52 | 55 |
| 2 | -10 | 61 | 69 |
| 2 | -9 | 62 | 53 |
| 2 | -8 | 54 | 57 |
| 2 | -7 | 136 | 134 |
| 2 | -5 | 91 | 96 |
| 2 | -4 | 125 | 135 |
| 2 | -3 | 88 | 84 |
| 2 | -2 | 295 | 294 |
| 2 | -1 | 177 | 184 |
| 2 | 0 | 165 | 160 |
| 2 | 1 | 153 | 156 |
| 2 | 2 | 255 | 268 |
| 2 | 3 | 282 | 281 |
| 2 | 4 | 165 | 155 |
| 2 | 5 | 55 | 53 |
| 2 | 7 | 50 | 68 |
| 2 | 8 | 153 | 165 |
| 2 | 9 | 98 | 96 |
| 3 | -9 | 74 | 72 |
| 3 | -8 | 62 | 77 |
| 3 | -7 | 105 | 101 |
| 3 | -6 | 141 | 137 |
| 3 | -5 | 206 | 212 |
| 3 | -4 | 191 | 204 |
| 3 | -2 | 228 | 224 |
| 3 | -1 | 202 | 193 |
| 3 | 0 | 297 | 294 |
| 3 | 1 | 198 | 202 |
| 3 | 2 | 62 | 63 |
| 3 | 3 | 98 | 96 |
| 3 | 4 | 181 | 188 |
| 3 | 5 | 189 | 198 |
| 3 | 6 | 187 | 196 |
| 3 | 7 | 45 | 47 |
| 3 | 9 | 49 | 49 |
| 4 | -8 | 46 | 40 |
| 4 | -7 | 99 | 102 |
| 4 | -5 | 76 | 74 |
| 4 | -4 | 135 | 154 |
| 4 | -3 | 190 | 195 |
| 4 | -2 | 235 | 239 |
| 4 | -1 | 99 | 90 |
| 4 | 0 | 192 | 188 |
| 4 | 1 | 205 | 209 |
| 4 | 2 | 300 | 296 |
| 4 | 3 | 224 | 224 |
| 4 | 4 | 105 | 117 |
| 4 | 5 | 76 | 91 |
| 4 | 6 | 77 | 81 |
| 4 | 7 | 70 | 86 |
| 4 | 8 | 86 | 101 |
| 5 | -9 | 43 | 25 |
| 5 | -7 | 96 | 98 |
| 5 | -6 | 58 | 76 |
| 5 | -5 | 142 | 138 |
| 5 | -4 | 70 | 64 |
| 5 | -3 | 39 | 35 |
| 5 | -2 | 139 | 143 |
| 5 | -1 | 154 | 150 |
| 5 | 0 | 195 | 176 |
| 5 | 1 | 76 | 65 |
| 5 | 2 | 47 | 38 |
| 5 | 3 | 92 | 103 |
| 5 | 4 | 97 | 114 |
| 5 | 5 | 182 | 180 |
| 5 | 6 | 63 | 58 |
| 5 | 7 | 68 | 64 |
| 5 | 8 | 54 | 54 |
| 6 | -7 | 52 | 45 |
| 6 | -5 | 45 | 58 |
| 6 | -3 | 81 | 83 |
| 6 | -2 | 65 | 63 |
| 6 | 0 | 52 | 53 |
| 6 | 1 | 73 | 71 |
| 6 | 2 | 117 | 112 |
| 6 | 3 | 123 | 131 |
| 6 | 6 | 70 | 68 |
| 6 | 7 | 63 | 69 |
| 7 | -3 | 84 | 81 |
| 7 | -1 | 64 | 61 |
| 7 | 1 | 59 | 67 |
| 7 | 2 | 45 | 53 |
| 7 | 3 | 59 | 51 |
| 7 | 5 | 67 | 76 |
| 8 | -4 | 56 | 70 |
| 8 | -1 | 60 | 50 |
| 8 | 2 | 49 | 50 |
| H = 8 | | | |
| 0 | -10 | 52 | 44 |
| 0 | -8 | 260 | 259 |
| 0 | -7 | 165 | 168 |
| 0 | -6 | 196 | 199 |
| 0 | -5 | 103 | 95 |
| 0 | -4 | 69 | 83 |
| 0 | -3 | 101 | 86 |
| 0 | -2 | 467 | 470 |
| 0 | -1 | 154 | 154 |
| 0 | 0 | 256 | 261 |
| 0 | 3 | 78 | 86 |
| 0 | 4 | 117 | 122 |
| 0 | 5 | 93 | 95 |
| 0 | 6 | 111 | 118 |
| 0 | 9 | 45 | 47 |
| 1 | -11 | 46 | 40 |
| 1 | -10 | 127 | 118 |
| 1 | -9 | 105 | 105 |
| 1 | -8 | 124 | 107 |
| 1 | -7 | 82 | 98 |
| 1 | -6 | 84 | 80 |
| 1 | -5 | 217 | 207 |
| 1 | -4 | 306 | 298 |
| 1 | -3 | 197 | 207 |
| 1 | -2 | 205 | 209 |
| 1 | -1 | 68 | 56 |
| 1 | 0 | 152 | 142 |
| 1 | 1 | 188 | 183 |
| 1 | 2 | 284 | 297 |
| 1 | 3 | 128 | 128 |
| 1 | 4 | 65 | 52 |
| 1 | 5 | 52 | 46 |
| 1 | 6 | 56 | 60 |
| 1 | 7 | 111 | 122 |
| 1 | 8 | 81 | 67 |
| 2 | -10 | 43 | 33 |
| 2 | -9 | 77 | 77 |
| 2 | -8 | 108 | 93 |
| 2 | -7 | 105 | 101 |
| 2 | -6 | 202 | 206 |
| 2 | -5 | 96 | 88 |
| 2 | -4 | 138 | 145 |
| 2 | -3 | 96 | 91 |
| 2 | -2 | 195 | 182 |
| 2 | -1 | 296 | 294 |
| 2 | 0 | 190 | 188 |
| 2 | 1 | 48 | 43 |
| 2 | 2 | 43 | 40 |
| 2 | 3 | 58 | 53 |
| 2 | 4 | 182 | 178 |
| 2 | 5 | 70 | 64 |
| 2 | 7 | 81 | 78 |
| 3 | -9 | 69 | 72 |
| 3 | -8 | 61 | 61 |
| 3 | -6 | 53 | 65 |
| 3 | -5 | 40 | 51 |
| 3 | -4 | 139 | 128 |
| 3 | -3 | 168 | 166 |
| 3 | -2 | 46 | 45 |
| 3 | -1 | 48 | 53 |
| 3 | 0 | 58 | 61 |
| 3 | 1 | 160 | 151 |
| 3 | 2 | 219 | 226 |
| 3 | 3 | 127 | 126 |
| 3 | 4 | 73 | 73 |
| 3 | 5 | 95 | 94 |
| 3 | 6 | 87 | 97 |
| 4 | -8 | 61 | 54 |
| 4 | -7 | 52 | 61 |
| 4 | -6 | 92 | 89 |
| 4 | -5 | 77 | 80 |
| 4 | -2 | 70 | 60 |
| 4 | -1 | 224 | 230 |
| 4 | 0 | 223 | 218 |
| 4 | 2 | 82 | 75 |
| 4 | 3 | 91 | 93 |
| 4 | 4 | 85 | 91 |
| 4 | 5 | 106 | 94 |
| 5 | -5 | 50 | 52 |
| 5 | -4 | 42 | 44 |
| 5 | -3 | 99 | 90 |
| 5 | -2 | 124 | 119 |
| 5 | -1 | 83 | 88 |
| 5 | 1 | 44 | 48 |
| 5 | 2 | 140 | 138 |
| 5 | 3 | 77 | 87 |
| 6 | -5 | 70 | 70 |
| 6 | -3 | 84 | 93 |
| 6 | -1 | 107 | 95 |
| 6 | 0 | 128 | 118 |
| H = 9 | | | |
| 0 | -7 | 106 | 117 |
| 0 | -5 | 65 | 58 |
| 0 | -4 | 185 | 187 |
| 0 | -3 | 62 | 55 |
| 0 | -2 | 214 | 211 |
| 0 | -1 | 219 | 210 |
| 0 | 0 | 83 | 77 |
| 0 | 1 | 53 | 57 |
| 0 | 3 | 181 | 186 |
| 0 | 4 | 56 | 54 |
| 1 | -7 | 116 | 111 |
| 1 | -6 | 97 | 96 |
| 1 | -5 | 168 | 157 |
| 1 | -4 | 176 | 174 |
| 1 | -3 | 162 | 164 |
| 1 | -1 | 161 | 164 |
| 1 | 0 | 83 | 91 |
| 1 | 1 | 168 | 164 |
| 1 | 3 | 119 | 125 |
| 1 | 4 | 88 | 90 |
| 2 | -7 | 69 | 62 |
| 2 | -5 | 142 | 146 |
| 2 | -4 | 74 | 75 |
| 2 | -3 | 62 | 61 |
| 2 | -2 | 114 | 116 |
| 2 | -1 | 120 | 114 |
| 2 | 0 | 67 | 69 |
| 2 | 1 | 58 | 61 |
| 2 | 2 | 93 | 108 |
| 2 | 3 | 44 | 41 |
| 2 | 4 | 51 | 57 |
| 3 | -7 | 129 | 120 |
| 3 | -6 | 58 | 60 |
| 3 | -5 | 69 | 63 |
| 3 | -4 | 41 | 47 |
| 3 | -3 | 147 | 151 |
| 3 | -2 | 52 | 64 |
| 3 | 0 | 50 | 49 |
| 3 | 2 | 47 | 49 |
| 3 | 3 | 186 | 180 |
| 4 | -5 | 103 | 99 |
| 4 | -1 | 52 | 61 |
| 4 | 0 | 83 | 62 |
| 4 | 1 | 100 | 83 |
| 5 | -4 | 51 | 52 |
| 5 | -3 | 58 | 60 |
| 5 | -2 | 70 | 67 |
| 5 | 0 | 43 | 52 |
| H = 10 | | | |
| 0 | -6 | 67 | 89 |
| 0 | -5 | 41 | 36 |
| 0 | -4 | 105 | 109 |
| 0 | -3 | 70 | 64 |
| 0 | -2 | 57 | 33 |
| 0 | -1 | 50 | 37 |
| 0 | 0 | 206 | 210 |
| 0 | 2 | 75 | 76 |
| 0 | 3 | 84 | 92 |
| 1 | -6 | 47 | 42 |
| 1 | -5 | 135 | 127 |
| 1 | -4 | 96 | 85 |
| 1 | -2 | 140 | 132 |
| 1 | 1 | 107 | 96 |
| 1 | 2 | 101 | 101 |
| 2 | -6 | 60 | 53 |
| 2 | -5 | 56 | 64 |
| 2 | -4 | 97 | 107 |
| 2 | -3 | 46 | 43 |
| 2 | -2 | 119 | 104 |
| 2 | -1 | 127 | 120 |
| 2 | 0 | 104 | 100 |
| 2 | 1 | 69 | 78 |
| 2 | 2 | 49 | 54 |
| 3 | -5 | 83 | 83 |
| 3 | -4 | 113 | 104 |
| 3 | -3 | 87 | 87 |
| 3 | -2 | 84 | 88 |
| 3 | 0 | 99 | 97 |
| 3 | 1 | 155 | 157 |



(I)

However, since the absolute configuration of the sugar skeleton is known to be α-D from the chemical work, the correct enantiomorph can be assigned unambiguously. Fig. 1 shows that the brosylate is therefore 5-brosyl-3-deoxy-3-*C*-(*R*)-(ethylcarboxy-formamido)methyl-1,2-*O*-isopropylidene-α-D-ribofuranose and the unsaturated carbohydrate derivative from which it was prepared must have *E*-stereochemistry (*IUPAC Tentative Rules for Nomenclature of Organic Chemistry*, 1970).

Hydrogen bonding is indicated between N and O(8) on account of their separation distance of 3·05 (2) Å (Pimentel & McClellan, 1960). The -NHCO- group is placed in the unit cell in such a way that a continuous column of these arrangements results about the two-fold screw axis at $x=\frac{1}{2}$ and $z=\frac{1}{2}$. This is clearly seen on the stereoscopic drawing (Fig. 2) showing the contents of the unit cell.

The mean aromatic C=C bond length of 1·39 (2) Å shows good agreement with the value of 1·395 Å quoted for this distance. The least-squares plane passing through the six benzene carbon atoms is given by equation (1) in Table 5. From Table 5 is seen that all six atoms are coplanar to within 0·01 Å. The two substituent atoms of the benzene ring, *viz*. Br and S, show insignificant deviations of 0·02 and 0·06 Å, respectively, from this plane. Typical separation distances of 1·89 Å and 1·77 Å for the respective atom pairs Br–C(1) and S–C(4) were observed (Hirai, Okuda & Nozoe, 1969).

Tetrahedral geometry about the sulphur atom is demonstrated by the six bond angles C(4)–S–O(1) (110·2°), C(4)–S–O(2) (103·2°), C(4)–S–O(3) (109·3°), O(1)–S–O(2) (104·0°), O(1)–S–O(3) (118·8°) and O(2)–S–O(3) (110·2°). The respective S–O bond lengths of 1·42 [S–O(1), S–O(3)] and 1·59 Å [S–O(2)] compare

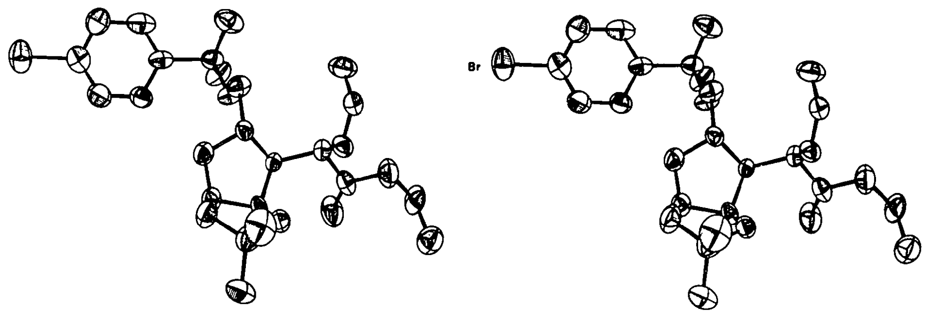


Fig. 1. Stereoscopic drawing of the molecule of 5-brosyl-3-deoxy-3-C-(*R*)-(ethylcarboxy-formamido)methyl-1,2-*O*-isopropylidene-α-D-ribofuranose.

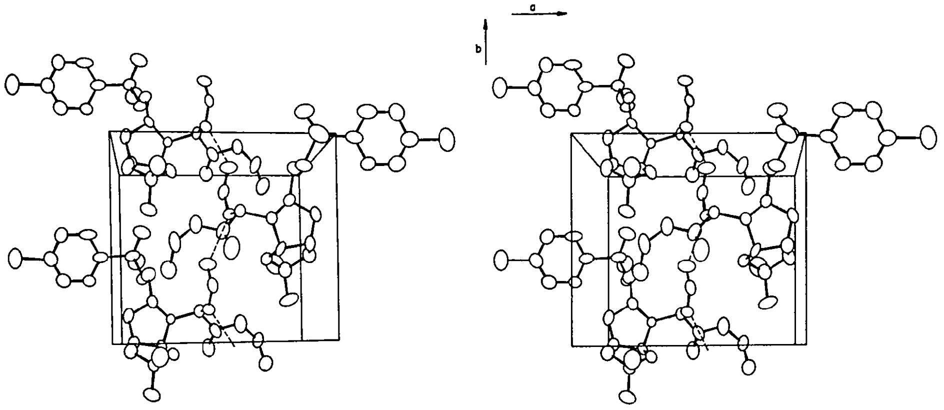


Fig. 2. Stereoscopic diagram showing the molecular packing in the unit cell viewed along the *c* axis.

Table 5. *Equations for least-squares planes*

| | | |
|---|---|---|
| C(1), C(2), C(3), C(4), C(5), C(6) | $0·0166x-0·0404y+0·9990z=0·7706$ | 0·01 (1) |
| C(16), O(8), O(9), C(15) | $-0·1285x-0·6146y+0·7783z=-2·8031$ | 0·01 (2) |

favourably with the corresponding values of 1·44 (2) and 1·56 (2) reported by Hirai *et al.* (1969) for these distances.

The least-squares plane through the carbonyl carbon atom C(16) and its three bonded neighbours O(8), O(9) and C(15) is given by equation (2) in Table 5. All the individual atomic deviations are smaller than 0·02 Å making the arrangement planar. The two C=O bond lengths of 1·23 Å [C(19)–O(9)] and 1·19 Å [C(16)–O(7)] fall within the accepted range for this distance (Sutton, 1965).

As expected, the $sp^2$-carbon–oxygen bond length [C(16)–O(8)] of 1·36 Å is slightly smaller than the average value of 1·43 (1) Å in the case of the $sp^3$C–oxygen bonds C(8)–O(4), C(9)–O(4), C(9)–O(5), C(10)–O(5), C(10)–O(6), C(13)–O(6), C(17)–O(8) and C(7)–O(2) (McPhail & Sim, 1966). The mean $sp^3$C–$sp^3$C distance of 1·53 (2) Å agrees satisfactorily with the value of 1·545 Å in diamond.

The chain pattern previously mentioned in which separate molecules are linked together through hydrogen bonding also gives rise to the shortest contact distance observed, *viz.* 3·05 Å between N and O(9) whose parent molecules are related by the twofold screw axis at $a=\frac{1}{2}$ and $c=\frac{1}{2}$.

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# The Crystal Structure of α-$CF_4$

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The crystal structure of tetrafluoromethane in a low-temperature phase has been studied with polycrystalline specimens. A monoclinic tetramolecular cell has been found which has the following parameters at 10°K: $a=8·435\pm0·005$, $b=4·320\pm0·002$, $c=8·369\pm0·005$ Å, $\beta=119·40\pm0·05°$; the unit-cell volume and X-ray density are $V=265·7\pm0·5$ Å$^3$ and $\varrho=2·119\pm0·005$ g.cm$^{-3}$. The calculation of the structure factors and their comparison with the experimental intensities of reflexions have been carried out on the basis of the packing model of $CF_4$ molecules in a lattice. The space group $P2_1/c$ has been found.

## Introduction

The crystal structures of most of the carbon tetrahalides have not been determined unambiguously, thus complicating studies and understanding of the physical properties of this class of materials. Reliable investigation of the structure of these crystals is impeded by the lack of single crystals and the requirement for performing experiments at low temperatures. Crystals grown in a high-temperature phase are usually destroyed (Rudman & Post, 1966) because of a large volume jump at a phase transition. Therefore, the data on the structures of some carbon tetrahalides have either been obtained recently ($CCl_4$: Rudman & Post,