Further Refinement of the Structure of Sucrose Based on Neutron-Diffraction Data*

By George M. Brown and Henri A. Levy

Chemistry Division, Oak Ridge National Laboratory, Oak Ridge, Tennessee 37830, U.S.A.

The structure of sucrose, $C_{12}H_{22}O_{11}$, has been further refined using the neutron-diffraction data of Brown & Levy [*Science* (1963). **141**, 921-923] and the full-matrix least-squares method, with anisotropic extinction corrections. The final discrepancy index $R(F)$ is 0·033 and the standard deviation of fit is 0·997. Parameters and structural details reported earlier are confirmed at a higher level of precision. Standard errors of bond lengths are as follows: C–C and C–O, 0·0012 to 0·0019 Å; C–H, 0·0022 to 0·0033 Å; O–H, 0·0028 to 0·0042 Å.

Introduction

In 1963 we published a preliminary report (Brown & Levy, 1963) on a refinement based on neutron-diffraction data of the structure of sucrose ($C_{12}H_{22}O_{11}$) as reported by Beevers, McDonald, Robertson & Stern (1952). The structure at a later stage of refinement was described at the Rome I.U.Cr. meeting in 1963 (see also the brief description in Brown & Levy, 1964*a*). Parameters corresponding to this later stage have been made available on request to a number of interested persons, and comparisons have been made of structural features of sucrose with those of other sugar molecules whose structures have been well determined in the intervening time (see for example: Sundaralingam, 1965, 1968; Berman, Chu & Jeffrey, 1967; Fries, Rao & Sundaralingam, 1971; Strahs, 1970; Rohrer, 1972).

Because of the scale of the least-squares computational problem (involving the usual nine parameters for each of 45 atoms) relative to the limited capability of the computer used in 1963, the structure parameters had to be refined in blocks and the refinement was not continued to complete convergence. Further, the available least-squares program had no provision for correcting the observations for effects of extinction, as is commonly done today. We had attempted to minimize extinction effects (Brown & Levy, 1964*a*, *b*) by using data from the smaller two of three crystal specimens for the stronger reflections and data from the largest crystal for the weaker reflections which make up the bulk of the data. However, although we were confident that the 1963 atomic coordinates were reliable, we have been concerned lately about possible errors in the thermal parameters arising as residual effects of extinction. Knowing of a study in progress (see the accompanying paper by Hanson, Sieker & Jensen, 1973) using our neutron parameters in conjunction with new X-ray data to compare thermal parameters for the hydrogen atoms and to study the

distribution of bonding electron density in the molecule, we recently resumed work on sucrose and have now completed refinement of the parameters using our original neutron data and the full-matrix least-squares method, with Type 1 anisotropic extinction corrections (Coppens & Hamilton, 1970).

Data and refinement

Revised cell parameters determined* by the method of least squares from measurements of the Bragg angles of 14 high-angle reflections are given in Table 1. Most details of the collection of intensity data have been given previously (Brown & Levy, 1963, 1964*a*, *b*). The neutron wavelength was 1·078 Å. The experimental value 2·60 cm^{-1} was used for the absorption coefficient. In Table 2 the weight of each of the three crystals is given, along with the range of the correction factors for absorption.

For the final refinement the data set consisted of 2813 values of $|F(hkl)|_o^2$ from crystal A,† 1002 values

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Table 1. *Crystal data for sucrose at* $22·5 \pm 1·5°C$

Formula	$C_{12}H_{22}O_{11}$
Formula weight	342·30
Space group	$P2_1$
a	10·8633 (5) Å
b	8·7050 (4)
c	7·7585 (4)
β	102·945 (6)°
Cell volume	715·04 Å³
Z	2
d(calc).	1·590 g cm^{-3}
d(exp.)	1·58 *
[λ(Cu $K\alpha_1$) taken to be	1·54051 Å]

* *Structure Reports* (1952)

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from crystal B, and 145 values from crystal C, with a standard error, $\sigma(|F|_o^2)$, for each. The standard error was computed as

$$\sigma(|F|_o^2) = \{[\sigma\,(|F|_o^2)]^2 + (0{\cdot}03\,|F|_o^2)^2\}^{1/2},$$

where $[\sigma_s(|F|_o^2)]^2$ is the purely statistical variance and the term $(0{\cdot}03\,|F|_o^2)^2$ is an empirical variance correction. The 169 $|F(hkl)|_o^2$ values equal to or less than their standard errors were included with the others as observations. For every reflection the reciprocal of the

Table 2. *Weights, ranges of absorption corrections, maximum extinction corrections, and extinction parameters* Z'_{ij} *of the three sucrose crystals used*

The mean mosaic spread $\langle \eta \rangle$ associated with each Z'_{ii} parameter is also given.

	Crystal A	Crystal B	Crystal C
Weight (mg)	96·3†	10·8	5·1
Range of	2·144 to	1·423 to	1·292 to
abs. corr.	2·466	1·657	1·465
Max. ext. corr.	2·965	1·164	1·112
$Z'_{11}, \langle \eta_1 \rangle$	2·73 (21), 3·5″	0·075 (18), 21·2″	0·059 (58), 23·9″
$Z'_{22}, \langle \eta_2 \rangle$	0·22 (7), 12·5″	0·016 (12), 46·7″	0·055 (43), 24·7″
$Z'_{33}, \langle \eta_3 \rangle$	2·87 (10), 3·4″	0·0002 (6), 392·3″	0·005 (7), 84·5″
Z'_{12}	−0·14 (9)	−0·009 (10)	0·013 (40)
Z'_{13}	0·00 (14)	0·001 (6)	−0·020 (38)
Z'_{23}	0·00 (7)	0·000 (5)	−0·000 (18)

* The components Z'_{ij} are defined by Coppens & Hamilton (1970). However, they are here referred not to the axis system normally used by Coppens & Hamilton but to the φ-axis system of Cartesian coordinates of Busing & Levy (1967).
† Incorrectly said to be ~ 80 mg in previous publications.

Table 3. *Coordinates and thermal parameters, with standard errors, for the crystal structure of sucrose, all times* 10^5

The anisotropic temperature factor has the form $\exp\,(-\beta_{11}h^2 - \beta_{22}k^2 - \beta_{33}l^2 - 2\beta_{12}hk - 2\beta_{13}hl - 2\beta_{23}kl)$.

ATOM	X	Y	Z	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
C (1)	29961(8)	35792(0)	48487(12)	274(6)	376 (9)	584(12)	6(6)	94(7)	4(9)
C (2)	31253(9)	47474 (15)	63600 (12)	304(6)	498(10)	641(13)	−44(7)	73(7)	−64(9)
C (3)	28545(9)	63673(15)	56447(13)	321(7)	437(10)	965(15)	−18(7)	196(8)	−72(10)
C (4)	37404 (10)	67095(15)	44198(14)	400(7)	403(10)	1003(15)	−22(7)	243(9)	6(11)
C (5)	35925(9)	55107(16)	29529(13)	314(7)	525 (11)	756(14)	−23(7)	133(8)	64(10)
C (6)	45754 (11)	57083(17)	18455(14)	525(8)	749(14)	858(15)	−133(9)	307(9)	−2(12)
C' (1)	10301(10)	13110(15)	54380(12)	463(8)	559(11)	517(12)	−71(8)	152(8)	104(9)
C' (2)	12446(8)	19262(14)	36895(10)	307(6)	362(9)	426(11)	−6(6)	101(6)	32(8)
C' (3)	718(8)	19075(15)	21485(11)	288(6)	430(9)	521(11)	−32(6)	106(7)	19(9)
C' (4)	6478(9)	16653(15)	5476(11)	354(6)	474(10)	464(11)	−39(7)	107(7)	−2(9)
C' (5)	17635(9)	6133(15)	12864(12)	393(7)	440(10)	565(12)	27(7)	152(7)	−62(9)
C' (6)	28927(10)	8194(17)	4668(14)	482(8)	795(14)	814(15)	81(9)	303(9)	−49(12)
O (1)	17143(9)	34630(15)	39165(13)	272(7)	340(10)	611(13)	−5(7)	86(8)	18(10)
O (2)	22954(12)	43550(19)	74766(16)	488(10)	749(15)	668(16)	−67(10)	206(10)	−40(13)
O (3)	30801(13)	74770(19)	70279(21)	577(12)	656 (16)	1650(27)	−213(11)	507(15)	−516(17)
O (4)	34880(18)	81412(20)	35631(24)	1042(17)	478(15)	1629(29)	151(13)	593(19)	271(18)
O (5)	37719(10)	39878(16)	36864(15)	316(8)	442(12)	837(16)	−1(8)	224(9)	−20(11)
O (6)	58144(13)	54525(21)	28621(20)	384(9)	878(19)	1425(24)	−118(11)	379(12)	−190(17)
O' (1)	3017(12)	23548(20)	62119(16)	503(9)	859(16)	690(16)	−75(11)	314(10)	−26(13)
O' (2)	21205(10)	9445(16)	31572(13)	400(8)	472(12)	540(13)	124(8)	78(8)	−17(10)
O' (3)	−7367(11)	31776(17)	20452(16)	337(8)	602(13)	856(17)	109(9)	126(10)	64(13)
O' (4)	−2123(12)	9734(19)	−8904(14)	502(10)	836(16)	541(15)	−143(10)	39(10)	−62(12)
O' (6)	32644(13)	23799(21)	4035(17)	553(11)	931(18)	852(19)	−125(12)	259(11)	36(15)
H (C1)	33468(20)	24508(27)	53880(29)	509(16)	610(22)	1155(33)	74(16)	112(18)	159(22)
H (C2)	41158(21)	469.9(33)	71166(31)	440(16)	1125(32)	1123(36)	−21(19)	−65(19)	−86(28)
H (C3)	18707(20)	64476(32)	48969(34)	412(15)	947(30)	1749(44)	59(18)	146(21)	46(30)
H (C4)	47171(21)	66808(34)	52180(33)	502(17)	1118(33)	1462(39)	−183(20)	228(20)	−153(31)
H (C5)	26384(21)	56129(35)	20931(32)	492(16)	1135(34)	1347(38)	11(21)	−29(20)	149(30)
H (C6)	45305(32)	68728(40)	13445(42)	1101(31)	1212(41)	1993(58)	−11(31)	607(35)	652(42)
H' (C6)	43723(32)	49265(46)	7189(39)	1023(30)	1939(57)	1272(43)	−442(35)	454(29)	−556(40)
H(C'1)	5199(30)	2238(32)	52157(36)	1129(30)	800(30)	1425(43)	−345(25)	436(30)	80(29)
H' (C'1)	19473(24)	10908(36)	63300(31)	732(20)	1337(38)	975(32)	145(24)	102(21)	360(30)
H (C'3)	−4946(21)	8843(27)	22896(28)	556(17)	728(25)	1116(32)	−181(17)	212(19)	25(23)
H(C'4)	9840(22)	27720(27)	1551(29)	635(18)	747(25)	1101(33)	−58(18)	273(20)	231(24)
H (C'5)	14653(25)	−5988(29)	11325(32)	846(22)	610(23)	1204(35)	−74(19)	270(23)	−154(23)
H (C'6)	36724(30)	1189(42)	11947(48)	743(24)	1541(49)	2281(66)	462(29)	466(33)	491(46)
H' (C'6)	26505(31)	3991(41)	−8870(36)	1114(29)	1451(43)	1227(39)	−116(32)	580(29)	−464(36)
H (O2)	27193(26)	37235(36)	84664(31)	845(23)	1253(37)	969(34)	−77(26)	172(23)	185(31)
H (O3)	23175(29)	76597(36)	74255(44)	888(27)	1047(34)	2232(60)	−253(25)	832(34)	−598(38)
H (O4)	34066(58)	89130(40)	43250 (60)	3073(88)	695(35)	2880(91)	367(47)	1812(78)	149(47)
H (O6)	60152(31)	43834(39)	28663(46)	890(28)	1052(39)	2121(62)	26(27)	340(33)	248(39)
H (O'1)	8454(27)	32466(32)	65416(35)	863(24)	905(30)	1489(42)	−138(23)	508(26)	−209(30)
H (O'3)	−3100(24)	40891(30)	17613(35)	716(21)	677(26)	1589(43)	85(19)	327(24)	159(28)
H (O'4)	−1672(25)	15381(34)	−19611(27)	849(23)	1109(33)	729(30)	−4(23)	45(21)	47(25)
H (O'6)	34766(29)	27804(37)	16026(35)	909(26)	1261(39)	1234(40)	−211(26)	287(27)	−193(32)

corrected variance was used as the least-squares weight. The data set for the largest crystal was essentially complete to the value 0·764 Å^{-1} in $(\sin\theta)/\lambda$. The data

for the three different crystals were not averaged because the effects of extinction were different for the three.

Table 4. *Structure factors*

See text for explanation

Table 4 (cont.)

The values used for the coherent neutron scattering amplitudes, b's, were from the compilation of the Neutron Diffraction Commission (1969); namely, 0·665, 0·577, and −0·372 (in units of 10^{-12} cm) for carbon, oxygen, and hydrogen atoms respectively. In the full-matrix refinement the following parameters, 425 in number, were adjusted: a scale parameter and six anisotropic extinction parameters for each crystal; three coordinates and six anisotropic thermal parameters for each atom except C(1), for which y was held constant.

At convergence in the least-squares refinement the values of the usual measures of goodness of fit were as follows: 0·033 for $R(F)$, the usual discrepancy index; 0·037 for $R(F^2)$, the corresponding index computed on the $|F|^2$ values; 0·050 for the weighted index computed on $|F|^2$; 0·997 for the standard deviation of fit. These measures of goodness of fit indicate a significantly better fit of parameters to the data than had been attained without the inclusion of the extinction parameters. A final difference map showed maximum positive and negative excursions of 0·02 in units of 10^{12} cm^{-2}. For comparison, in the corresponding Fourier map the height of the highest positive peak, that of atom C(1), was 6·45 units and the depth of the deepest 'hole', corresponding to atom H(C1), was 2·50 units. The

extinction parameters Z'_{ij} and the maximum correction factors for extinction are included in Table 2, and the final coordinates and thermal parameters are given in Table 3. In Table 4 under the headings FO and FC are the values of $100|F|_o$ and $100|F_c|$; under SG are the values $100\sigma(|F|_o)$ if $|F|_o^2 \geq \sigma(|F|_o^2)$ but otherwise the values $-100\sigma(|F|_o^2|$; under EXT are the values of the transmission factor for extinction (given modulo 1·000, times 1000). The scaled* values of $|F|_o$ and of the standard errors include the corrections for extinction. The data for crystals B and C are so marked; the other data are for crystal A.

Discussion

As Table 2 shows, the extinction was extraordinarily anisotropic for crystal A, somewhat anisotropic for B,

and essentially isotropic for C. Since none of the Z'_{ij} parameters for $i \neq j$ are significantly different from zero, the axial directions of the reference Cartesian system are the principal axes. For all three crystals axis 3 corresponds to the **b** direction in the crystal. For A the axis of *greatest* mosaic spread is along the c axis of the crystal. In contrast, for crystal B the *smallest* mosaic spread is associated with the direction of **c**. The fact that crystal B had been repeatedly dipped in liquid nitrogen, whereas A had not, may account for the difference.

All bond lengths and valence angles for the sucrose molecule are reported in Fig. 1,† along with the torsion angles (Klyne & Prelog, 1960) of greatest interest. Included in the Figure are the standard errors of all these molecular parameters as calculated from the

* The scale factors of 100 are appropriate for $|F|_o$ and $|F|_c$ in units of 10^{-12} cm.

† In Figs. 1 and 2 the parentheses in the atom designations given in Table 3 are omitted for simplicity.
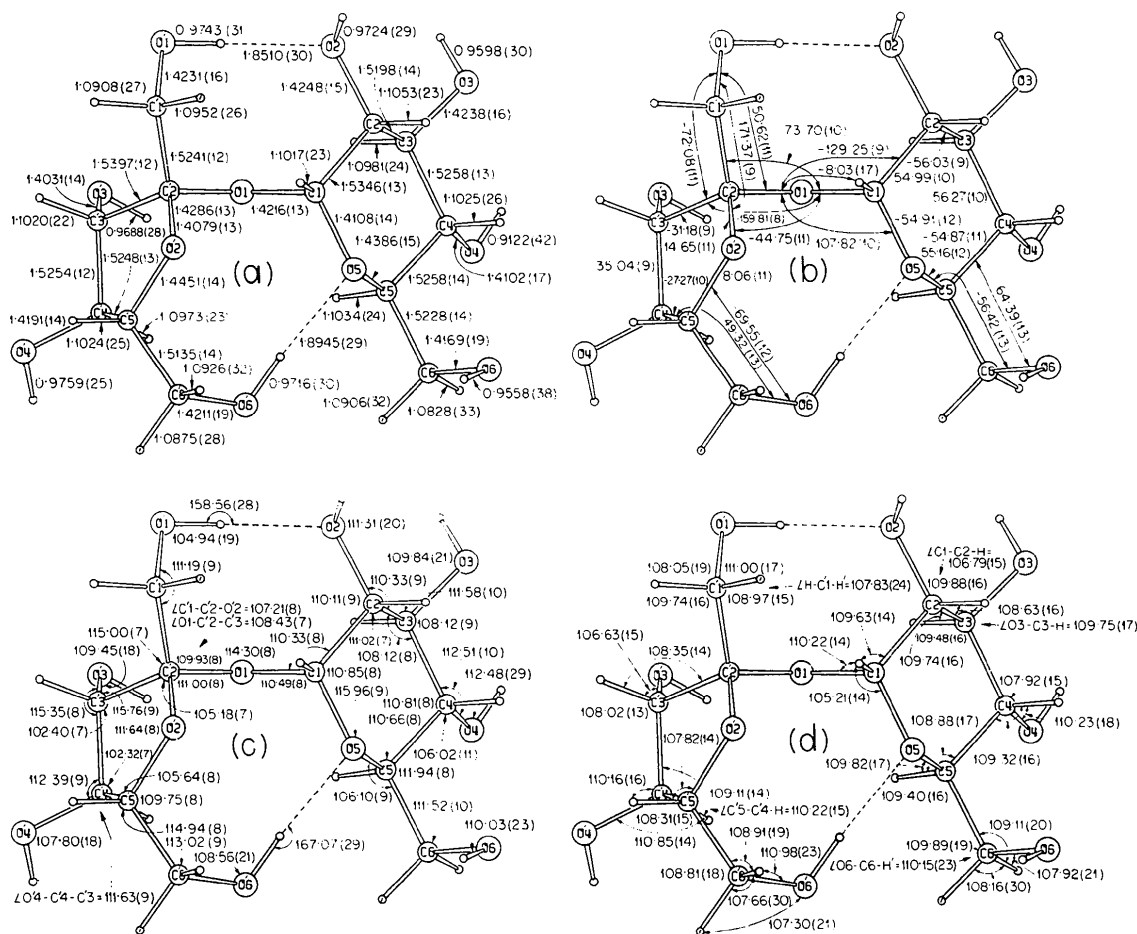

Fig. 1. The structure of the sucrose molecule: (a) bond lengths (Å); (b) selected torsion angles (°); (c) valence angles (°) not involving C–H bonds; (d) valence angles involving C–H bonds. All lengths and angles are uncorrected for effects of thermal motion. Numbers in parentheses specify the standard errors of the corresponding quantities computed from the full least-squares covariance matrix. The two intramolecular hydrogen bonds are indicated. The atom labels correspond to those of Table 1, except that the parentheses have been omitted. Torsion angles involving ring bonds only are written adjacent to the ring bonds; each of the other torsion angles is defined by a double-headed arrow pointing to two bonds at opposite ends of a given bond.

complete least-squares covariance matrix. Fig. 2†
shows a different view of the molecule which displays
more clearly the shape of the furanose ring. Fig. 3
shows a stereoscopic view in which the atoms are
represented by their ellispoids of 50% probability
(Johnson, 1970).

No lengthy discussion of the molecular structure is
warranted here, since the refinement essentially con-
firms at a higher level of precision details already
discussed in our preliminary paper (Brown & Levy,
1963) or by other authors to whom we supplied param-
eters (see Introduction). Discussion will be limited,
therefore, to a few features not previously considered
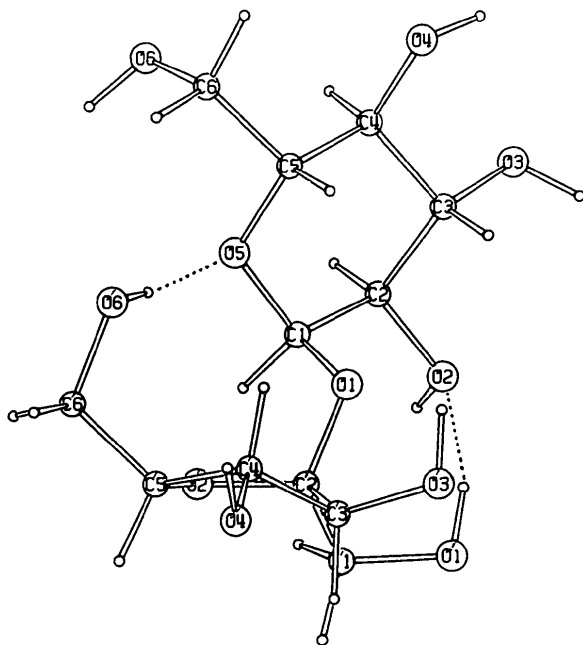in detail.

Fig. 2. The sucrose molecule viewed along a line in the plane
of C'(2), O'(2), and C'(5), perpendicular to bond C'(2)–O'(2).

Contrary to what is usually found for α-pyranosides
(Eliel, 1969; de Hoog, Buys, Altona & Havinga, 1969),
the conformation about the C(1)–O(1) bond does not
correspond to the A1 conformer, in which the O(1)–
C'(2) bond would be +syn-clinal (gauche) to C(1)–O(5)
and anti-periplanar (trans) to C(1)–C(2). In fact (see
Fig. 1) the bond O(1)–C'(2) is +anti-clinal to C(1)–O(5)
and nearly eclipsed with respect to C(1)–H(C1). In
this conformation there is no 'rabbit-ear' interaction
between O(1) and O(5) (Eliel, 1969), just as there is
none in A1. The conformation about the corresponding
bond in the sucrosyl portion of planteose (dihydrate,
Rohrer, 1972) is almost identical to that in sucrose,
whereas it is closer to the ideal A1 conformation in the
sucrosyl portions of raffinose (pentahydrate, Berman,
1970) and 1-kestose (Jeffrey & Park, 1972).

This determination, like the neutron determinations
of α-D-glucose (Brown & Levy, 1965) and D-glucitol
(Park, Jeffrey & Hamilton, 1971), is more nearly
complete than most structure determinations of carbo-
hydrates or related molecules in the sense that the
hydrogen atoms have been located accurately. The
valence geometry of each atom is therefore completely
described, and a firm basis for the desired under-
standing of bonding in the molecule is provided.

No one of the three H–C–H angles differs significant-
ly from the average of 107·9°, which is almost identical
with the value 107·8° for the single H–C–H angle in
α-D-glucose. Other H–C–H angles from recent precise
neutron-diffraction studies are: 108·2 (6) and 107·3 (6)°
in glycylgycine monohydrochloride monohydrate
(Koetzle & Hamilton, 1972); 106·7 (12) and 105·2 (13)°
in L-glutamic acid hydrochloride (Sequeira, Rajagopal
& Chidambaram, 1972); 108·7 (7) and 109·3 (6)° in
the α form of D-glucitol (Park, Jeffrey & Hamilton,
1971). The departure of most of these angles from the
tetrahedral value is consistent with the known struc-
tures of simple hydrocarbons, in which H–C–H angles
are generally smaller than they would be for carbon
atoms in exact sp^3 hybridization (or, in the case of

Table 5. *Description of the hydrogen bonds in sucrose*

O–H⋯O	Symmetry Transformations*	O–H	Distances (Å) H⋯O	O⋯O	Angle O–H⋯O (°)
Intramolecular					
O'(1)–H⋯O(2)	1,000	0·974	1·851	2·781	158·6
O'(6)–H⋯O(5)	1,000	0·972	1·895	2·850	167·1
Intermolecular					
O(2)–H⋯O'(6)	1,001	0·972	1·892	2·855	170·2
O(3)–H⋯O'(3)	2,001	0·959	1·907	2·862	172·8
O(6)–H⋯O(3)	2,1T1	0·956	1·921	2·848	162·9
O'(3)–H⋯O'(4)	2,000	0·969	1·908	2·864	168·5
O'(4)–H⋯O'(1)	1,00T	0·976	1·760	2·716	165·4
Non-participating OH					
O'(2)	1,010		2·309	2·838	116·6
O(4)–H O(3)	1,000	0·912	2·534	2·879	103·0
O(6)	2,101		2·539	3·373	152·1

* The symmetry transformations generate the coordinates of the acceptor oxygen atoms from the basic coordinates in Table 3.
The first digit represents either (1) the position x,y,z or (2) the position $-x, y+\frac{1}{2}, -z$. The last three digits specify a lattice trans-
lation.

terminal methylene groups, smaller than for carbon atoms in exact sp^2 hybridization). The average angle H–C–H for tetrahedral carbon atoms in ethane (Kuchitsu, 1968), propane (Lide, 1960a), isobutane (Lide, 1960b), propylene (Lide & Christensen, 1961; Hirota & Morino, 1966), and isobutylene (Scharpen & Laurie, 1963) is about 107·5°. Theoretical calculations on ethane have yielded 107·7° (Frost & Rouse, 1968) and 107·8° (Newton, Latham, Hehre & Pople, 1970).

The angles H–C–C in sucrose vary more widely than the H–C–H angles, from 106·8 to 110·2°, averaging 109·1°; similarly, the angles H–C–O range from 105·2 to 111·0°, averaging 109·1°. The angles H(C1)–C(1)–O(5) and H(C5)–C(5)–O(5), 105·2 and 109·8°, are identical within experimental error with the corresponding angles in α-D-glucose.

The general appearance of the ellipsoids in the stereoscopic drawing of Fig. 3 suggests that all the apparent C–H and O–H bond lengths, that is, those calculated from the coordinates without correction, are significantly shorter than the actual mean interatomic separations because of the effects of thermal motion (Cruickshank, 1956). The average apparent length is 1·090 Å for the six C–H bonds of the CH_2OH groups, 1·102 Å for the other eight C–H bonds, and 1·097 Å overall. After correction according to the 'riding' model (Busing & Levy, 1964), these averages become 1·122, 1·121, and 1·121 Å respectively. Closely similar averages have been calculated for α-D-glucose (Brown & Levy, 1965). The riding model is an approximation which must result in overcorrections here, since the carbon atoms are not the centers of motion for the hydrogens. It seems clear, however, that the typical C–H mean separation must be about 1·110 Å. The mean separation in gaseous methane has been reported from an electron diffraction study (Bartell, Kuchitsu & deNeui) to be $1·106_8 \pm 0·001$ Å.

The average of the uncorrected O–H bond lengths excluding the extremely short O(4)–H(O4) length is 0·968 Å, and the average of the lengths corrected by the riding model is 0·989 Å (cf. 0·986 Å in α-D-glucose).

The O(4)–H(O4) length is in a category by itself as a consequence of the unusually large thermal motion of atom H(O4), which is minimally involved in hydrogen bonding, if at all (see below). For H(O4) the principal axis of greatest thermal motion is at 102° to the O(4)–H(O4) bond direction; the associated r.m.s. displacement is 0·432 (6) Å, to be compared, for example, with displacements of 0·43 to 0·56 Å found for the hydrogen atoms in the methyl groups of copper acetate monohydrate (Chidambaram & Brown, 1972), which of course are not hydrogen bonded. The apparent O(4)–H(O4) distance of 0·912 (4) is corrected to the value 0·989 (5) Å by use of the riding model. Though the exact agreement to three decimal places between this corrected value and the average of those of the other OH bond lengths is fortuitous, the agreement is nevertheless a striking demonstration of basic understanding of the effects of thermal motion on apparent bond lengths.

The distance and angle parameters describing the hydrogen bonds are given in Table 5. A figure showing the hydrogen bonds was published previously (Brown & Levy, 1963). As the table and figure show, there are seven crystallographically distinct hydrogen bonds, excluding the weak interactions of H(O4). These seven are all in the normal ranges of O · · · O distance and of angularity. They involve the ring oxygen O(5) as an acceptor and each OH group except O(4)–H(O4) and O(6)–H(O6) as both donor and acceptor; they involve O(6)–H(O6) as a donor only and O(4)–H(O4) and ring oxygen O′(2) not at all.

Whether or not one considers even the shortest of the interactions H(O4) · · · O in Table 5, an indication of a hydrogen bond is a matter of definition: basically a matter first of what is taken as the normal van der Waals's distance H · · · O and then what is taken as the minimum significant contraction. Although the H(O4) · · · O′(2) [1,010] distance is 0·09 Å less than the distance 2·4 Å that one might take as minimum for the van der Waals distance, the energy effect must be very small, as is evidenced by the large vibrational motion of H(O4).

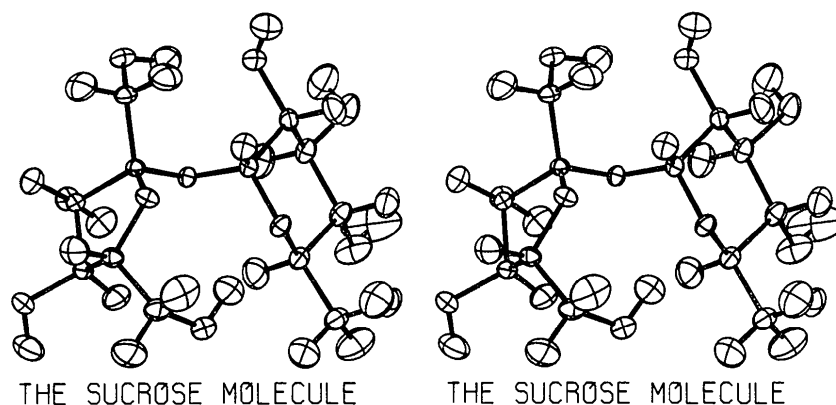THE SUCROSE MOLECULE   THE SUCROSE MOLECULE

Fig. 3. Stereoscopic view of the sucrose molecule showing the 50% probability ellipsoids. Atoms may be identified by reference to Fig. 1, in which the molecular orientation is only slightly different.

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Sucrose: X-ray Refinement and Comparison with Neutron Refinement

BY JONATHAN C. HANSON, LARRY C. SIEKER AND LYLE H. JENSEN

Department of Biological Structure, University of Washington, Seattle, Washington 98195, U.S.A.

The structure of sucrose has been refined with Mo $K\alpha$ diffractometer data collected from two crystals to a $\sin \theta/\lambda$ limit of 0·81 Å^{-1}. 3280 reflections were measured of which 3098 were greater than 2σ. The structure has been refined with low-angle data ($\sin \theta/\lambda < 0·64$ Å^{-1}), high-angle data ($\sin \theta/\lambda > 0·64$ Å^{-1}) and all the data, and the results compared with the very precise neutron-diffraction results of Brown & Levy. The final R indices are 0·025, 0·029 and 0·029 respectively for the X-ray data. The C–O bond lengths from refinements of low-angle and all data are longer and thermal parameters greater than those from the neutron data. The thermal parameters for the nonhydrogen atoms obtained from the high-angle refinement, however, are not significantly different from the neutron values. Use of bonded hydrogen atom scattering factors of Stewart, Davidson & Simpson [*J. Chem. Phys.* (1965). **42**, 3175–3187] led to thermal parameters for hydrogen bonded to oxygen and for hydrogen bonded to carbon which were respectively 0·3(3) Å^2 greater and 1·9(1) Å^2 less than those from the neutron results. Residual electron-density maps based on neutron coordinates and X-ray data showed the expected bond and lone pair features much more clearly than maps based on the coordinates obtained from refinement of high $\sin \theta$ X-ray data.

Introduction

The structure of sucrose was solved by Beevers, McDonald, Robertson & Stern (1952), and subsequently Brown & Levy (1963) carried out a highly precise refinement using neutron-diffraction data (see accompanying paper by Brown & Levy, 1973). This report describes a refinement based on X-ray data collected on