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α -D-Glucose: Further Refinement Based on Neutron-Diffraction Data*

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Abstract

Precise translations were determined for α -D-glucose, C₆H₁₂O₆, (orthorhombic, *P*2₁2₁2₁) as follows: $a = 10.3662$ (9), $b = 14.8506$ (16), $c = 4.9753$ (3) Å at 294–296 K based on $\lambda(\text{Cu } K\alpha_1) = 1.54051$ Å. The structure has been further refined using neutron-diffraction data previously obtained [Brown & Levy (1965). *Science*, **147**, 1038–1039] and the full-matrix least-squares method, with anisotropic extinction corrections. The fit to the observations is much improved [$R(F) = 0.048$; standard deviation of fit = 1.053], and the parameter errors are slightly smaller. Parameter changes are slight and of no chemical significance. The segmented-body model has been used to calculate corrections to the bond lengths and valence angles for the effects of thermal motion. Bond lengths, valence angles, torsion angles, and details of hydrogen bonding are tabulated.

we had attempted to minimize the effects of extinction by the somewhat unsatisfactory method of giving zero weights to the 37 observations of $|F_o|^2$ corresponding to the highest observed intensities. Because of the biochemical importance of α -D-glucose, we have now completed the refinement using our original data and applying anisotropic extinction corrections (Coppens & Hamilton, 1970). Shifts from the old parameters (Brown & Levy, 1965) are small (see below), but there is striking improvement in the fit to the observations, and there are slight decreases in the standard errors of the parameters. We have used the segmented-body model (Johnson, 1970) to analyze the new thermal parameters and thereby provide corrections to the bond lengths and valence angles for the effects of thermal motion. The bond length and angle data are also rendered more precise, and presumably more reliable, by use of the new, more precisely determined cell parameters which we also report here.

Introduction

Thirteen years ago (Brown & Levy, 1965) we reported atomic coordinates and selected derivative structural parameters for α -D-glucose from a fairly precise refinement, based on neutron-diffraction data, of the structure of McDonald & Beevers (1952). The data used in the 1965 work appear to be of high quality by present standards; but there are considerable extinction effects, and the least-squares refinement procedure did not include adjustment of extinction parameters. Instead,

Data and refinement

The revised cell parameters (see *Abstract*) were determined, with the assistance of R. D. Ellison, by the least-squares method from the Bragg angles of 11 high-angle reflections ($138 \leq 2\theta \leq 165^\circ$) from a small crystal on an X-ray diffractometer. In every significant detail the procedures in recording neutron-diffraction data and in preliminary data reduction were as in our work on sucrose (Brown & Levy, 1972). A total of 2168 intensity measurements were made for the 1656 independent reflections with indices all positive or zero to the limit 0.760 \AA^{-1} in $(\sin \theta)/\lambda$ ($\lambda = 1.078 \text{ \AA}$, $\theta_{\text{max}} = 55.0^\circ$). For the absorption corrections (Busing & Levy, 1957) the coefficient was $\mu = 0.28 \text{ mm}^{-1}$; the minimum

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and maximum correction factors were 1.78 and 2.09. After averaging the replicate $|F_o|^2$ data, the variance $\sigma^2(|F_o|^2)$ of each average was corrected empirically by the addition of the term $(0.03|F_o|^2)^2$ (Peterson & Levy, 1957). Except as noted below for the final cycles, each observation $|F_o|^2$ was weighted in the least-squares refinement by the reciprocal of its corrected variance.

Refinement of an isotropic extinction parameter along with a scale factor, the coordinates, and the U_{ij} 's reduced $R(F)$ from 0.099 to 0.061 and also brought $R(F^2)$, $R_w(F^2)$, and σ_1 (standard deviation of fit) down to 0.061, 0.077, and 1.260, respectively, each less than half its starting value. Attempts to refine all six of the type I anisotropic extinction parameters Z'_{ij} (Coppens & Hamilton, 1970) led to a non-positive-definite quadratic form involving the Z'_{ij} 's. However, adjustment of only the three Z'_{ii} axial parameters was successful, leading on convergence to the following final measures of goodness of fit: $R(F) = 0.048$, $R(F^2) = 0.045$, $R_w(F^2) = 0.063$, $\sigma_1 = 1.053$. In the final cycles 68 reflections were given zero weights, two of them because the instrument angles had probably been mis-set during their measurement and the other 66 because we considered their phases undetermined [$|F_c|^2 < \sigma(|F_o|^2)$ and $|F_o|^2 > \sigma(|F_o|^2)$ for each of the 66]. Four other reflections at the $(\sin \theta)/\lambda$ limit were inadvertently omitted. The value of $R(F)$ is 0.034 for the 1416 reflections having $|F_o|^2 > \sigma(|F_o|^2)$.

In the last cycle of refinement no shift was as large as 0.05 times the corresponding standard error, and most shifts were much smaller. In a difference Fourier map the maximum and minimum densities were 0.09 and -0.08 (in units of 10^{-11} mm \AA^{-3}). In comparison, the H atoms had peak densities of -1.51 to -2.28 in an $F_o - F_{\text{heavy}}$ Fourier map; and in a Fourier map the C and O peak densities were respectively 4.89 to 6.04 and 3.58 to 5.00.

Table 1 contains the final atomic coordinates and their standard errors.* The r.m.s. coordinate difference and r.m.s. thermal parameter (U_{ij}) difference between the new and the old parameters are 0.0006 \AA and 0.0005 \AA^2 ; the corresponding r.m.s. Δ/σ values are 0.26 and 0.53. No apparent atom position shifted more than 0.0024 \AA . The three Coppens & Hamilton extinction parameters are: $Z'_{11} = 0.047$ (14), $Z'_{22} = 0.013$ (3), $Z'_{33} = 0.043$ (7), all in units of 10^{-8} rad $^{-2}$, where the reference Cartesian axes 1, 2, and 3 are parallel to **a**, **b**, and **c**, respectively. The largest extinction correction factor on $|F_o|^2$ was 1.448 (for reflection 111). The neutron-scattering amplitudes used are as follows: $b_c = 0.6648$, $b_o = 0.5803$, $b_H = -0.374$, all in units of 10^{-11} mm (Shull, 1972).

* Lists of anisotropic thermal parameters and values of $|F_o|^2$, $|F_o|^2 - |F_c|^2$, $\sigma(|F_o|^2)$, and the extinction factor have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 34050 (12 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Fractional atomic coordinates in α -D-glucose

The numbers in parentheses, corresponding to the least significant digits of the coordinates, give estimated standard errors from the least-squares covariance matrix.

	x	y	z
C(1)	0.33293 (11)	0.60072 (8)	-0.07611 (30)
C(2)	0.41845 (10)	0.67457 (7)	0.04544 (30)
C(3)	0.56108 (10)	0.65142 (7)	0.01522 (28)
C(4)	0.58598 (10)	0.55687 (7)	0.12005 (26)
C(5)	0.49555 (11)	0.48861 (7)	-0.01218 (26)
C(6)	0.51413 (13)	0.39532 (8)	0.10252 (35)
O(1)	0.34712 (17)	0.60069 (12)	-0.35403 (38)
O(2)	0.38796 (13)	0.75980 (9)	-0.06552 (43)
O(3)	0.63340 (13)	0.71538 (9)	0.16320 (47)
O(4)	0.71758 (13)	0.53482 (10)	0.06872 (39)
O(5)	0.36541 (11)	0.51538 (8)	0.03696 (34)
O(6)	0.44580 (17)	0.33111 (10)	-0.05176 (47)
H(1)	0.23261 (23)	0.61234 (20)	-0.01552 (86)
H(2)	0.39576 (28)	0.67766 (20)	0.25996 (67)
H(3)	0.58889 (29)	0.65417 (19)	-0.19946 (65)
H(4)	0.56756 (29)	0.55656 (21)	0.33854 (59)
H(5)	0.51348 (30)	0.48650 (20)	-0.23107 (62)
H(6)	0.61715 (32)	0.37926 (20)	0.10342 (119)
H(7)	0.48050 (46)	0.39457 (24)	0.31145 (82)
H(8)	0.27529 (34)	0.56742 (24)	-0.42998 (84)
H(9)	0.44860 (30)	0.77768 (18)	-0.20321 (83)
H(10)	0.72463 (23)	0.71369 (18)	0.11783 (79)
H(11)	0.75171 (33)	0.50818 (26)	0.23235 (73)
H(12)	0.41270 (32)	0.28609 (18)	0.07009 (92)

Molecular structure and hydrogen bonding

To provide the basis for correction of bond lengths and angles for the effects of thermal motion, we applied the segmented-body model for molecular thermal motion as incorporated in program *ORSBA* (Johnson, 1970). The molecule was treated as having two coupled rigid segments: segment (1) containing the six ring atoms, O(5) and C(1) through C(5); segment (2) containing C(6), O(6), H(6), and H(7). Segment (1) was permitted the full 20-parameter rigid-body description according to Schomaker & Trueblood (1968); segment (2) was permitted the additional motion (with six parameters) of libration about a center at C(5), this being a riding motion superposed on that of segment (1), as described by Johnson (1970). The observed thermal parameters of the two H atoms were first corrected for internal vibration by subtracting components derived from the vibrations of methane as given by Cyvin (1968), namely 0.00601 along and 0.01550 \AA^2 perpendicular to the C-H bonds. The r.m.s. difference between observed and calculated thermal parameters U_{ij} for the ten fitted atoms was 0.00049 \AA^2 .

Bond lengths were corrected (Johnson & Levy, 1974) by use of *ORSBA* by combining as appropriate the corrections for segmented-body motion and for riding motion (Busing & Levy, 1964). For the bonds within the segmented body, only the former kind of motion is involved in making the corrections. For each

Table 2. Bond lengths (Å) (except O–H bonds), valence angles (°), and torsion angles (°) in α -D-glucose

The bond lengths and valence angles (but not the torsion angles) are corrected for thermal motion. The additive corrections included are shown in the adjacent angle brackets, where the numbers given correspond to the least significant digits of the lengths and angles. Estimated standard errors calculated from the full least-squares covariance matrix for the *uncorrected* lengths and angles are given in parentheses. Bonds and angles involving only atoms of the ring are in bold type.

C(1)–C(2) 1.539 $\langle 4 \rangle$ (2)	C(1)–O(5) 1.430 $\langle 3 \rangle$ (2)	C(4)–O(4) 1.430 $\langle 4 \rangle$ (2)	C(4)–H(4) 1.123 $\langle 19 \rangle$ (3)
C(2)–C(3) 1.529 $\langle 4 \rangle$ (2)	C(5)–O(5) 1.431 $\langle 3 \rangle$ (2)	C(6)–O(6) 1.424 $\langle 10 \rangle$ (2)	C(5)–H(5) 1.123 $\langle 18 \rangle$ (3)
C(3)–C(4) 1.523 $\langle 3 \rangle$ (2)	C(1)–O(1) 1.400 $\langle 9 \rangle$ (3)	C(1)–H(1) 1.116 $\langle 19 \rangle$ (3)	C(6)–H(6) 1.121 $\langle 27 \rangle$ (4)
C(4)–C(5) 1.534 $\langle 4 \rangle$ (2)	C(2)–O(2) 1.421 $\langle 4 \rangle$ (2)	C(2)–H(2) 1.112 $\langle 18 \rangle$ (4)	C(6)–H(7) 1.123 $\langle 27 \rangle$ (5)
C(5)–C(6) 1.517 $\langle 6 \rangle$ (2)	C(3)–O(3) 1.425 $\langle 9 \rangle$ (2)	C(3)–H(3) 1.127 $\langle 20 \rangle$ (3)	
C(1)–C(2)–C(3) 111.1 $\langle 0 \rangle$ (1)	O(4)–C(4)–C(3) 108.2 $\langle 1 \rangle$ (1)	H(5)–C(5)–O(5) 109.6 $\langle 0 \rangle$ (2)	
C(2)–C(3)–C(4) 109.8 $\langle 0 \rangle$ (1)	O(4)–C(4)–C(5) 110.9 $\langle 0 \rangle$ (1)	H(1)–C(1)–O(1) 112.0 $\langle 0 \rangle$ (3)	
C(3)–C(4)–C(5) 111.1 $\langle 0 \rangle$ (1)	O(6)–C(6)–C(5) 109.9 $\langle 5 \rangle$ (1)	H(1)–C(1)–O(5) 104.7 $\langle 1 \rangle$ (2)	
C(4)–C(5)–C(6) 111.5 $\langle 0 \rangle$ (1)	H(1)–C(1)–C(2) 109.1 $\langle 0 \rangle$ (2)	H(2)–C(2)–O(2) 107.2 $\langle 0 \rangle$ (2)	
C(5)–O(5)–C(1) 113.7 $\langle 1 \rangle$ (1)	H(2)–C(2)–C(1) 106.9 $\langle 0 \rangle$ (2)	H(3)–C(3)–O(3) 109.8 $\langle 0 \rangle$ (2)	
O(5)–C(1)–C(2) 110.1 $\langle 0 \rangle$ (1)	H(2)–C(2)–C(3) 108.3 $\langle 0 \rangle$ (2)	H(4)–C(4)–O(4) 109.9 $\langle 0 \rangle$ (2)	
O(5)–C(5)–C(4) 108.7 $\langle 0 \rangle$ (1)	H(3)–C(3)–C(2) 109.8 $\langle 0 \rangle$ (2)	H(6)–C(6)–O(6) 110.3 $\langle 2 \rangle$ (3)	
O(5)–C(5)–C(6) 108.1 $\langle 0 \rangle$ (1)	H(3)–C(3)–C(4) 108.7 $\langle 0 \rangle$ (2)	H(7)–C(6)–O(6) 110.7 $\langle 3 \rangle$ (3)	
O(1)–C(1)–O(5) 111.5 $\langle 0 \rangle$ (1)	H(4)–C(4)–C(3) 108.2 $\langle 0 \rangle$ (2)	H(8)–O(1)–C(1) 107.8 $\langle 0 \rangle$ (3)	
O(1)–C(1)–C(2) 109.3 $\langle 0 \rangle$ (1)	H(4)–C(4)–C(5) 108.4 $\langle 0 \rangle$ (2)	H(9)–O(2)–C(2) 112.1 $\langle 0 \rangle$ (2)	
O(2)–C(2)–C(1) 110.9 $\langle 0 \rangle$ (1)	H(5)–C(5)–C(4) 109.9 $\langle 0 \rangle$ (2)	H(10)–O(3)–C(3) 112.1 $\langle 0 \rangle$ (2)	
O(2)–C(2)–C(3) 112.2 $\langle 1 \rangle$ (1)	H(5)–C(5)–C(6) 109.0 $\langle 0 \rangle$ (2)	H(11)–O(4)–C(4) 106.9 $\langle 1 \rangle$ (3)	
O(3)–C(3)–C(2) 108.1 $\langle 0 \rangle$ (1)	H(6)–C(6)–C(5) 109.2 $\langle 2 \rangle$ (2)	H(12)–O(6)–C(6) 107.2 $\langle 5 \rangle$ (3)	
O(3)–C(3)–C(4) 110.6 $\langle 0 \rangle$ (1)	H(7)–C(6)–C(5) 109.1 $\langle 0 \rangle$ (2)	H(6)–C(6)–H(7) 107.6 $\langle 1 \rangle$ (4)	
O(5)–C(1)–C(2)–C(3) 54.1 (2)	O(3)–C(3)–C(4)–O(4) –65.5 (2)	H(6)–C(6)–C(5)–H(5) 72.3 (4)	
O(5)–C(1)–C(2)–H(2) –63.8 (2)	C(3)–C(4)–C(5)–O(5) –57.5 (1)	H(6)–C(6)–C(5)–C(4) –49.2 (3)	
O(1)–C(1)–C(2)–O(2) 56.9 (2)	C(3)–C(4)–C(5)–H(5) 62.4 (2)	H(8)–O(1)–C(1)–O(5) 75.0 (3)	
O(1)–C(1)–C(2)–C(3) –68.7 (2)	O(4)–C(4)–C(5)–C(6) 62.9 (2)	H(8)–O(1)–C(1)–H(1) –42.1 (3)	
H(1)–C(1)–C(2)–H(2) 50.6 (3)	O(4)–C(4)–C(5)–H(5) –58.0 (2)	H(8)–O(1)–C(1)–C(2) –163.1 (3)	
H(1)–C(1)–C(2)–O(2) –65.8 (3)	H(4)–C(4)–C(5)–O(5) 61.3 (2)	H(9)–O(2)–C(2)–C(3) 24.4 (3)	
C(1)–C(2)–C(3)–C(4) –51.3 (1)	H(4)–C(4)–C(5)–C(6) –57.9 (2)	H(9)–O(2)–C(2)–C(1) –100.6 (3)	
C(1)–C(2)–C(3)–H(3) 68.2 (2)	C(4)–C(5)–O(5)–C(1) 62.2 (2)	H(9)–O(2)–C(2)–H(2) 143.2 (3)	
O(2)–C(2)–C(3)–O(3) 63.2 (2)	H(5)–C(5)–O(5)–C(1) –57.8 (2)	H(10)–O(3)–C(3)–C(4) 73.8 (3)	
O(2)–C(2)–C(3)–H(3) –56.6 (2)	C(6)–C(5)–O(5)–C(1) –176.5 (1)	H(10)–O(3)–C(3)–H(3) –46.1 (3)	
H(2)–C(2)–C(3)–C(4) 65.8 (2)	C(5)–O(5)–C(1)–C(2) –60.9 (2)	H(10)–O(3)–C(3)–C(2) –165.9 (3)	
H(2)–C(2)–C(3)–O(3) –54.9 (2)	H(5)–C(5)–O(5)–C(1) 60.6 (2)	H(11)–O(4)–C(4)–H(4) 16.2 (3)	
C(2)–C(3)–C(4)–C(5) 53.3 (1)	C(5)–O(5)–C(1)–H(1) –178.1 (2)	H(11)–O(4)–C(4)–C(5) –103.6 (3)	
C(2)–C(3)–C(4)–H(4) –65.6 (2)	O(6)–C(6)–C(5)–O(5) 70.2 (2)	H(11)–O(4)–C(4)–C(3) 134.3 (3)	
H(3)–C(3)–C(4)–O(4) 55.2 (2)	O(6)–C(6)–C(5)–H(5) –48.8 (2)	H(12)–O(4)–C(4)–H(6) 95.7 (4)	
H(3)–C(3)–C(4)–C(5) –66.9 (2)	H(7)–C(6)–C(5)–C(4) 68.2 (3)	H(12)–O(4)–C(4)–H(7) –23.1 (4)	
O(3)–C(3)–C(4)–H(4) 53.6 (2)	H(7)–C(6)–C(5)–O(5) –51.3 (3)	H(12)–O(4)–C(4)–C(5) –143.8 (3)	

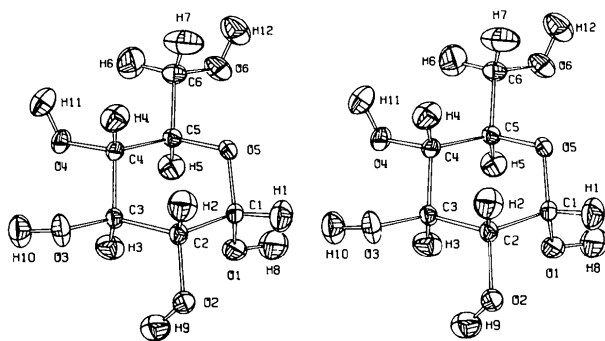


Fig. 1. Stereoscopic drawing of the α -D-glucose molecule, showing the atom numbering. The atoms are represented by their ellipsoids of 50% probability (Johnson, 1976).

external C–O bond on segment (1) a correction is made similarly for the rigid motion of the segment, assumed to apply here for the external O atoms as well

as for the ring atoms. In addition, a correction is made for these bonds for O riding on C, *after* the motions of both C and O are corrected for the segment (1) motion. The O–H and C–H bonds are treated similarly; but specifically for the two H atoms in segment (2) the motion subtracted before the segmented-body fit is put back in before the riding correction is computed. Note that the effect of O riding on C is ignored when the correction to O–H is computed. The corrections to valence angles are computed from the segmented-body motion only.

The corrected bond lengths (except for O–H bonds, for which see Table 3) and corrected valence angles are shown in Table 2. The additive corrections included in the lengths and angles given are shown in angle brackets $\langle \rangle$. Also included in Table 2 are uncorrected torsion angles (Klyne & Prelog, 1960) sufficient to specify (with slight redundancy) a Newman projection for each C–C and C–O bond. The α -D-glucose

Table 3. *The O—H bond lengths and details of the hydrogen bonding*

The meaning of the numbers in angle brackets and parentheses is as in Table 2. Only the O—H bond lengths have been corrected for thermal motion.

Number*	Bond O—H...O	Symmetry transformation	Distances (Å)			Angle (°) O—H...O
			O—H	H...O	O...O	
1	O(3)—H(10)...O(2)†	2,010	0.975 <02> (3)	1.758 (3)	2.708 (2)	164.9 (3)
2	O(6)—H(12)...O(3)†	4,110	0.977 <11> (4)	1.758 (4)	2.714 (3)	169.7 (4)
3	O(4)—H(11)...O(4)†	3,110	0.987 <15> (4)	1.819 (4)	2.777 (1)	167.7 (3)
4	O(2)—H(9)...O(6)†	4,101	0.980 <13> (4)	1.821 (4)	2.778 (3)	170.1 (3)
5	O(1)—H(1)...O(5)†	3,011	0.984 <14> (4)	1.915 (4)	2.849 (2)	160.9 (4)

* Each hydrogen bond can be located in Fig. 2 through use of its identifying number, generally in more than one equivalent position.

† The symmetry transformations generate the coordinates of the acceptor O atoms from the basic coordinates in Table 1. The first digit represents a position equivalent to x, y, z as follows: (2) $\frac{1}{2} + x, \frac{1}{2} - y, \bar{z}$; (3) $\frac{1}{2} - x, y, \frac{1}{2} + z$; (4) $\bar{x}, \frac{1}{2} + y, \frac{1}{2} - z$. The last three digits specify a lattice translation.

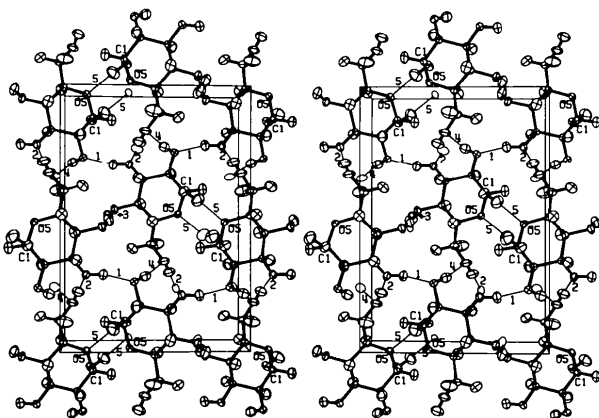


Fig. 2. Stereoscopic drawing showing the molecular packing and hydrogen bonding in the α -D-glucose crystal. The atoms are represented by their 50% probability ellipsoids. The center of the cell outline shown is at $\frac{1}{2}, \frac{1}{2}, 0$. The view direction is along c ; a points from right to left; b points from bottom to top. The molecule of the reference asymmetric unit is the one closest to the center of the cell outline. The numbering of the hydrogen-bonding interactions H...O, represented here by thin lines, is consistent with that in Table 3.

molecule is shown stereoscopically in correct absolute configuration in Fig. 1.

The Cremer & Pople (1975) ring-puckering parameters are $q_2 = 0.0347$ (15), $q_3 = 0.5657$ (14), $Q = 0.5668$ (14) Å, $\theta = 3.51$ (15), $\phi_2 = -36.9$ (2.4)°. The atomic-numbering convention used for their calculation was the same as that used by Cremer & Pople (1975) for the glucopyranoid ring of sucrose.

Details of the geometry of the hydrogen bonds, including the O—H bond lengths, are given in Table 3. Five different kinds of bonds connect the glucose molecules to form a three-dimensional hydrogen-bonded network, shown in the stereoscopic packing drawing of Fig. 2.

The structural data from our previous publication (Brown & Levy, 1965) on α -D-glucose have been used in many discussions in the chemical and crystallographic literature on carbohydrate molecular and

crystal structures, including hydrogen bonding. The data reported here in somewhat more detail are not sufficiently different to furnish any new structural inferences or to require modification of any old ones; consequently, we shall present no additional discussion of them.

The computer programs used in this study and not specifically referenced are the same as those used for the corresponding calculations of Lisensky, Johnson & Levy (1976).

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