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Note

Crystal structure of an adduct of sarcosine with sucrose

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Sucrose (β-D-fructofuranosyl-α-D-glucopyranoside, $C_{12}H_{22}O_{11}$), a disaccharide, is widely distributed in nature, having been found universally throughout the plant kingdom in fruits, seeds, flowers and roots of plants. The crystal structure of sucrose has been determined precisely using neutron diffraction data, and a detailed analysis has already been carried out [1,2]. Sarcosine (*N*-methylglycine, $CH_3NH_2^+CH_2COO^-$) is an α-amino acid found to be present in several biologically important compounds and is also used in certain cosmetics [3,4]. The crystal structure of sarcosine was earlier elucidated in the authors' laboratory [5]. This study, on the structure elucidation of an adduct of sarsocine with sucrose seems to be the first of its kind involving the interactions of an amino acid with a carbohydrate.

Single crystals of the above adduct were obtained by slow evaporation of an aqueous solution containing stoichiometric amounts of sarcosine and sucrose in 1:1 proportion. Intensity data were collected on an Enraf-Nonius CAD4 diffractometer with graphite monochromated MoK_α radiation using the ω-2θ scan technique up to 50.0° in 2θ giving 1795 unique reflections. Cell parameters were obtained from least-squares refinement of the setting angles of 25 centred reflections in the range $6 < \theta < 12°$. Crystal and experimental data are given in Table 1. The data were corrected for Lorentz

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[1] Tables of atomic coordinates, bond lengths and bond angles have been deposited with the Cambridge Crystallographic Data Centre. These tables may be obtained, on request, from the Director, Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge, CB2 1EZ, UK.

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Table 1
Crystal and experimental data for sarcosine–sucrose monohydrate

Formula	$C_{15}H_{31}NO_{14}$
Formula weight (amu)	449.41
Temperature (°C)	22 ± 1
Wavelength (Å)	0.70930
Crystal system	Monoclinic
Space group	$P2_1$
a (Å)	7.1750(10)
b (Å)	18.898(6)
c (Å)	7.777(4)
β (°)	110.3(2)
Volume (Å^3)	989.0(6)
Z	2
D_c (g cm^{-3})	1.509
μ (cm^{-1})	1.35
$F(000)$	480
Number of unique data	1933
Number of observed data [$I \geq 2\sigma(I)$], N	1795
No of parameters, P	396
R [a]	3.7%
R_w [b]	9.2%
S, goodness-of-fit [c]	1.145
Maximum shift/error, final	0.001
Largest positive peak (e/Å^3)	0.222
Largest negative hole (e/Å^3)	−0.205

[a] $R = \Sigma(|F_o| - |F_c|)/\Sigma|F_o|$.
[b] $R_w = \{\Sigma(F_o^2 - F_c^2)^2/\Sigma w(F_o^2)^2\}^{1/2}$.
[c] $S = [\Sigma w(F_o^2 - F_c^2)^2/(N - P)]^{1/2}$. $w = 1/[\sigma^2(F_o^2) + (0.0554 * P)^2 + 0.34 * P]$ where $P = [\max(F_o^2, 0) + 2F_c^2]/3$.

and polarization effects. No absorption correction was made. The structure was solved by direct methods using the programs SHELX86 [6] and SHELXL93 [7] [1]. All the hydrogen atoms were located from a difference map and included (with isotropic temperature factors) in the refinement. The maximum value of shift/esd was 0.001 in the final cycle. The final value of $R = 0.037$ and $R_w = 0.0978$.

Final positional and equivalent thermal parameters are given in Table 2. A drawing of the molecule illustrating the atom-labelling scheme is shown in Fig. 1. Selected torsion angles are reported in Table 3. The asymmetric unit consists of one sarcosine molecule, one sucrose molecule, and a water molecule. The sarcosine molecule exists in the zwitterionic state as two hydrogen atoms are located at the nitrogen atom. The bond distances in the sucrose molecule of the adduct sarcosine sucrose monohydrate (SSM) are in good agreement with the corresponding values for sucrose. The C–C bond lengths vary from 1.501(6) to 1.537(5) Å [mean 1.517(5) Å]. The C–O bond lengths vary from 1.405(5) to 1.443(5) Å [mean 1.422(5) Å]. The bond distances involving the C–H bonds are in the range 0.708 to 1.030 Å, the average being 0.912 Å. The C–O bond distances

Table 2
Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters (Å^2 $\times 10^3$) of the non-hydrogen atoms

Atom	x	y	z	U(eq) [a]
O-1′	3154(4)	2837(2)	316(5)	38(1)
C-1′	2916(6)	3529(2)	888(6)	29(1)
C-2′	1078(5)	3896(2)	−366(5)	22(1)
C-3′	1010(6)	4033(2)	−2322(5)	24(1)
O-3′	2079(4)	4646(2)	−2431(4)	33(1)
C-4′	−1193(6)	4033(2)	−3393(5)	26(1)
O-4′	−1623(5)	3897(2)	−5274(4)	34(1)
C-5′	−1908(6)	3443(2)	−2439(5)	27(1)
O-2′	−594(4)	3467(2)	−553(3)	26(1)
C-6′	−4049(6)	3522(3)	−2589(7)	34(1)
O-6′	−4760(5)	2917(2)	−1915(5)	42(1)
O-1	975(4)	4568(2)	454(3)	24(1)
C-1	−296(5)	4638(2)	1495(5)	23(1)
C-2	696(5)	5140(2)	3068(5)	25(1)
O-2	2481(5)	4844(2)	4287(4)	34(1)
C-3	960(6)	5871(2)	2358(6)	26(1)
O-3	1683(5)	6353(2)	3839(4)	37(1)
O-4	−595(5)	6718(2)	27(5)	37(1)
C-4	−997(6)	6135(2)	1013(5)	26(1)
C-5	−2087(6)	5575(2)	−414(5)	26(1)
O-5	−2171(4)	4898(2)	413(3)	25(1)
C-6	−4199(7)	5766(3)	−1502(6)	34(1)
O-6	−5489(4)	5737(2)	−439(5)	41(1)
OW	−4334(5)	4975(2)	3183(5)	36(1)
O-1$_s$	−2336(5)	6003(2)	5329(4)	41(1)
O-2$_s$	−4592(5)	6856(2)	4321(5)	50(1)
C-1$_s$	−2879(6)	6627(3)	5085(6)	32(1)
N$_s$	660(5)	6849(2)	6888(6)	34(1)
C-2$_s$	−1241(7)	7181(3)	5818(7)	37(1)
C-3$_s$	2317(8)	7349(3)	7660(8)	45(1)

[a] $U(\text{eq}) = \frac{1}{3}(U_{11} + U_{22} + U_{33})$.

associated with the anomeric carbon atom and the ring oxygen atom of both the furanose [mean 1.430(6) Å] and the pyranose ring [mean 1.422 Å] show a similar trend as observed in the structures of sucrose [1] and sucrose octaacetate [8]. The mean C–O lengths, excluding the ring oxygen and anomeric carbon atoms of furanose and pyranose rings are 1.413(5) and 1.426(6) Å, respectively. The exocyclic C–C bond distances are 1.507(6) and 1.501(6) Å in both the furanose and the pyranose ring. This shortening of the C–C bonds may be attributed to the fact that they involve a primary and a secondary carbon atom, while the ring C–C bonds are all between secondary carbon atoms [9]. The angle involving the bridge oxygen atom O-1 [C-2′–O-1–C-1 = 118.5(3)°] is 4.2° larger than that found in sucrose. The bond angles C-1–O-5–C-5 [113.5(3)°] and O-1–C-1–O-5 [112.2(3)°] agree well with the expected values of 113.8° and 111.5°, respectively, for

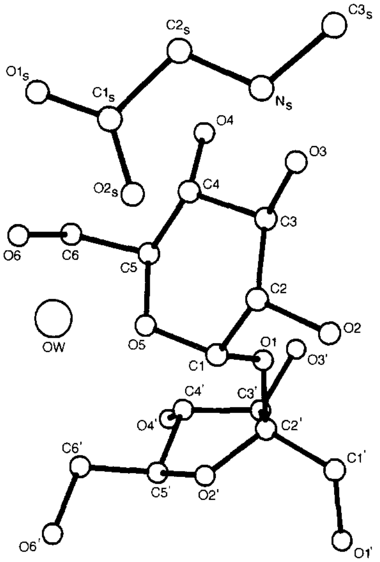

Fig. 1. Structure of the adduct of sarcosine with sucrose, showing the atom labelling scheme.

α-pyranoses [10]. The relative positions of the two rings are defined by the torsion angles O-5–C-1–O-1–C-2′ and O-2′–C-2′–O-1–C-1 whose values are −94.1° and 20.6°, respectively. These values, which are not far from those observed for sucrose [107.8(1)° and −44.8(1)°], indicate that the molecule exhibits an asymmetrical twist about the bridge bonds. The endocyclic torsion angles of the pyranose ring differ from the corresponding angles for sucrose by not more than 9.7° (mean 5.3°) and range from 46.6° to 61.2°. In the case of the furanose ring, the deviations in the endocylic torsion angles differ by not more than 4.9° (mean 3.3°) and range from 11.6° to 38.3°.

The changes in conformation of the sucrose molecule, due to the presence of an amino acid in its surrounding, is negligible in comparison with the conformation in sucrose crystals. The pyranose ring has the usual chair conformation (calculated puckering coordinates [11] are: $\phi = -111.3°$, $\theta = 172.1°$ and $Q = 0.536$). The five-membered furanose ring adopts a twist conformation (with $q = 0.381$ and $\phi = 91.1$), which seems to be the most stable conformation for many compounds containing this ring [12]. The structure is stabilized by an extensive network of hydrogen-bonding interactions that involve the pyranose ring oxygen as well as the carboxyl oxygens and nitrogen of the amino acid. An intramolecular hydrogen bond between the hydroxyl O-2(H) and the glycosidic oxygen O-1 is observed and the angle O-2–H...O-1 is far from linearity (95.1°). The two intramolecular hydrogen bonds O-1′(H)...O-2(H) and O-6′(H)...O-5 found in sucrose are absent in the present structure. The hydroxyl O-4′(H) does not take part in the hydrogen-bonding scheme as observed in the structure of sucrose [1]. The ring oxygen O-5 of the pyranose ring acts as an acceptor of a

Table 3
A selection of torsion angles (°) of sarcosine–sucrose monohydrate

O-1′–C-1′–C-2′–C-3′	−63.6	O-1–C-1–O-5–C-5	−58.1
O-1′–C-1′–C-2′–O-2′	54.8	O-1–C-1–C-2–O-2	−65.3
O-1′–C-1′–C-2′–O-1	176.3	O-1–C-1–C-2–C−3	62.1
C-1′–C-2′–O-1–C-1	−98.6	C-2–C-1–O-5–C-5	61.2
C-1′–C-2′–O-2′–C-5′	−136.6	O-5–C-1–C-2–C-3	59.4
C-1′–C-2′–C-3′–O-3′	−81.9	O-5–C-1–C-2–O-2	173.3
C-1′–C-2′–C-3′–C-4′	151.9	C-1–C-2–C-3–O-3	173.5
O-2′–C-2′–O-1–C-1	20.6	C-1–C-2–C-3–C-4	52.1
C-3′–C-2′–O-1–C-1	136.0	O-2–C-2–C-3–O-3	−60.8
O-1–C-2′–O-2′–C-5′	105.3	O-2–C-2–C-3–C-4	177.8
C-3′–C-2′–O-2′–C-5′	−11.6	C-2–C-3–C-4–C-5	−46.5
O-2′–C-2′–C-3′–C-4′	31.6	C-2–C-3–C-4–O-4	−164.7
O-2′–C-2′–C-3′–O-3′	157.7	O-3–C-3–C-4–C-5	−168.1
O-1–C-2′–C-3′–C-4′	−88.5	O-3–C-3–C-4–O-4	73.7
O-1–C-2′–C-3′–O-3′	37.6	O-4–C-4–C-5–O-5	166.5
C-2′–C-3′–C-4′–O-4′	−158.7	C-3–C-4–C-5–O-5	47.6
C-2′–C-3′–C-4′–C-5′	−38.3	O-4–C-4–C-5–C-6	−73.2
O-3′–C-3′–C-4′–O-4′	78.8	C-3–C-4–C-5–C-6	167.8
O-3′–C-3′–C-4′–C-5′	−160.7	C-4–C-5–O-5–C-1	−55.3
C-3′–C-4′–C-5′–O-2′	32.1	C-4–C-5–C-6–O-6	−70.8
C-3′–C-4′–C-5′–C-6′	153.9	C-6–C-5–O-5–C-1	−179.9
O-4′–C-4′–C-5′–O-2′	152.5	O-5–C-5–C-6–O-6	52.9
O-4′–C-4′–C-5′–C-6′	−85.6		
C-4′–C-5′–O-2′–C-2′	−13.0		
C-4′–C-5′–C-6′–O-6′	171.8	O-1$_s$–C-1$_s$–C-2$_s$–N$_s$	172.8
C-6′–C-5′–O-2′–C-2′	−136.2	O-2$_s$–C-1$_s$–C-2$_s$–N$_s$	−7.1
O-2′–C-5′–C-6′–O-6′	−69.9	C-3$_s$–N$_s$–C-2$_s$–C-1$_s$	−179.4
C-2′–O-1–C-1–C-2	144.9		
C-2′–O-1–C-1–O-5	−94.1		

hydrogen bond from a water molecule, whereas the ring oxygen O-2′ of the furanose ring does not take part in hydrogen bonding.

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