N-(1-Deoxy-α-D-tagatopyranos-1-yl)-*N*-methylaniline ("D-Tagatose-*N*-methylaniline")

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Supporting Information

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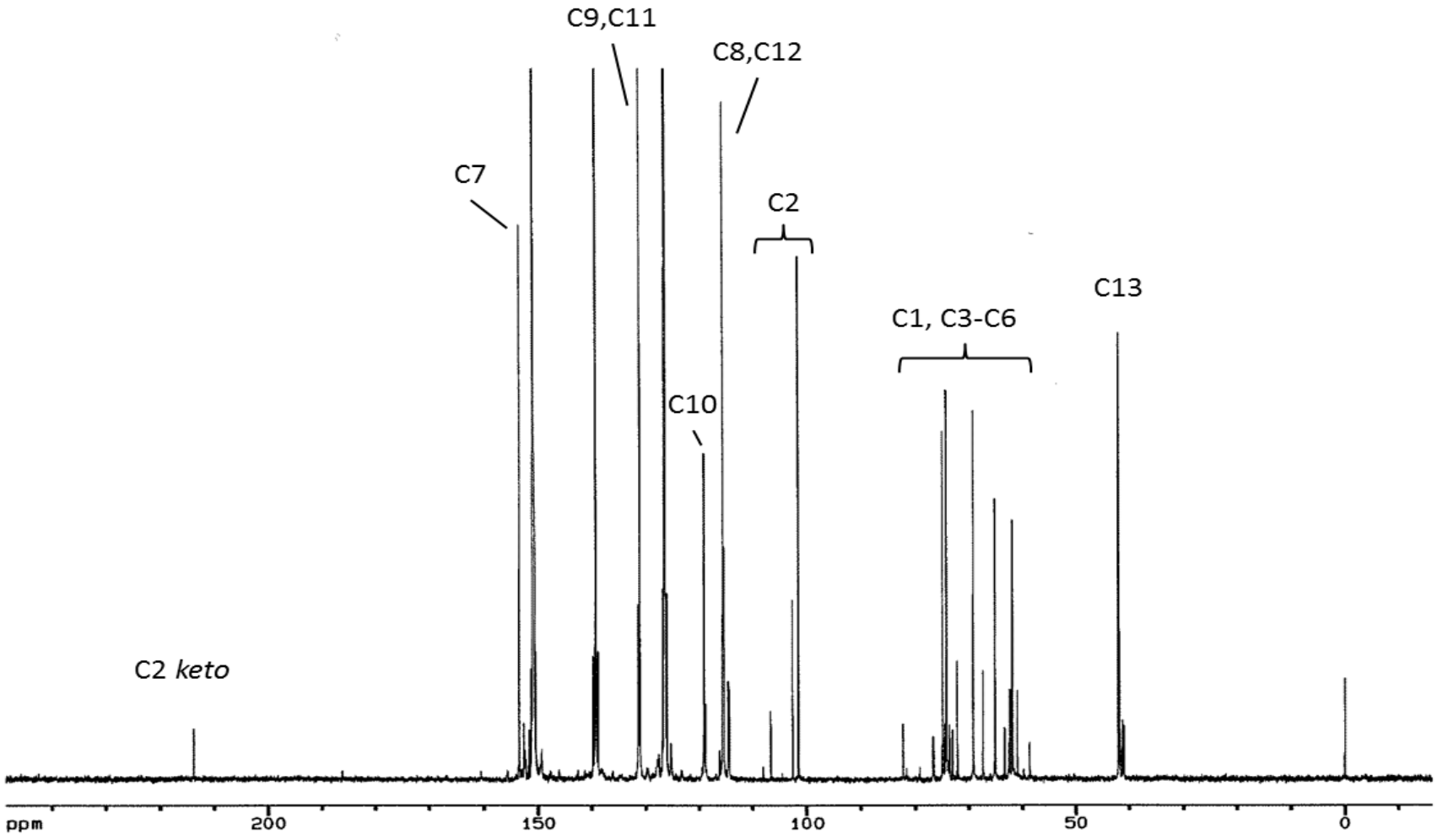

Figure S1. C13 NMR spectrum of D-tagatose-*N*-methylaniline in 1:1 pyridine/D_2O

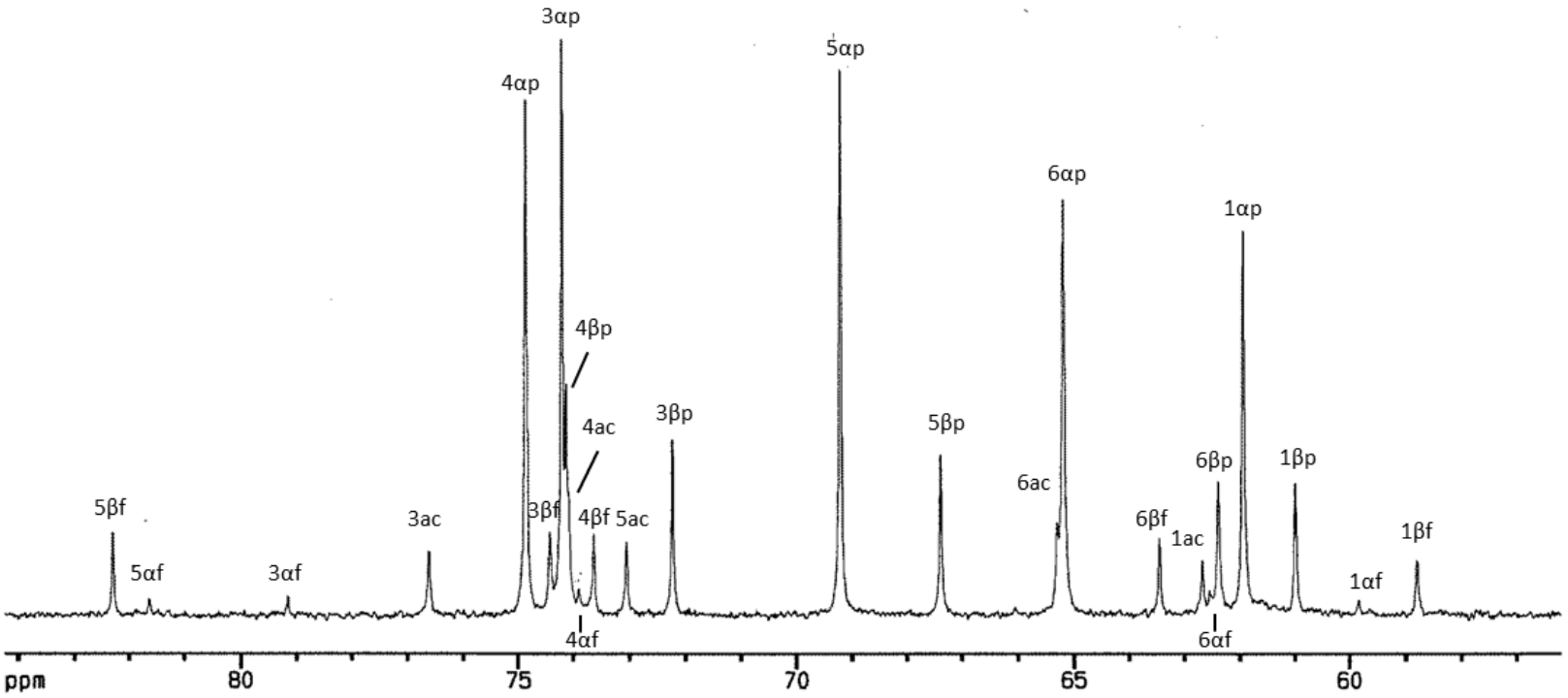

Figure S2. An expanded carbohydrate region of C13 NMR spectrum of D-tagatose-*N*-methylaniline in 1:1 pyridine/D_2O showing resolution of signals for the anomeric/tautomeric forms

Table S1. Crystal data, data collection and structure refinement details.

Crystal Data	
Chemical formula	$C_{13}H_{19}NO_5$
M_r	269.29
Crystal system, space group	Orthorhombic, $P2_12_12_1$
a, b, c (Å)	6.5757 (10), 7.7698 (10), 25.0403 (10)
V (Å^3)	1279.4 (3)
Z	4
F(000)	576
D_{calc}	1.398 Mg·m^{-3}
μ (mm-1)	0.899
Cell parameters from	2205 reflections
2θ range (°)	40.0 – 50.0
Crystal size (mm)	0.40 × 0.25 × 0.15
Data Collection	
Diffractometer	Enraf-Nonius CAD4
Temperature (K)	295
Radiation type	Cu$K\alpha$, λ = 1.54060 Å
Absorption correction	ψ-scans
T_{min}, T_{max}	0.76, 0.87
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	1559, 1559, 1478
R_{int}	0.0000
h, k, l ranges	$0 \rightarrow 8$; $-9 \rightarrow 9$; $-31 \rightarrow 31$
θ_{min}, θ_{max} (°)	3.5, 74.6
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.029, 0.076, 1.07
w	$1/[\sigma 2(F_o^2)+(0.403P)^2+0.177P]$, $P = (F_o^2 + 2F_c^2)/3$
No. of reflections	1559
No. of parameters	189
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$(\Delta/\sigma)_{max}$	0.038
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.13, -0.18
Absolute structure parameter	0.0 (2)

Table S2. Atomic Parameters x, y, z and B or B_{eq} for *N*-(1-deoxy-α-D-tagatopyranos-1-yl)-*N*-methylaniline. E.S.Ds. refer to the last digit printed.

	x	y	z	B or B_{eq}
O2	0.2619 (3)	0.2202 (2)	0.89535(6)	3.69(6)
O3	0.2456 (3)	-0.1229 (2)	0.80052(6)	3.44(6)
O4	0.5051 (3)	0.1209 (2)	0.74387(6)	3.86(6)
O5	0.2138 (3)	0.3777 (2)	0.72880(6)	3.59(6)
O6	-0.0061 (2)	0.1361 (2)	0.84061(6)	3.29(6)
N	-0.0032 (3)	0.0151 (3)	0.95136(7)	3.50(7)
C1	0.1103 (4)	-0.0581 (3)	0.90636(8)	3.23(8)
C2	0.1741 (3)	0.0841 (3)	0.86754(7)	2.61(7)
C3	0.3315 (3)	0.0238 (3)	0.82588(7)	2.63(7)
C4	0.3716 (3)	0.1718 (3)	0.78615(7)	2.68(7)
C5	0.1720 (3)	0.2317 (3)	0.76153(7)	2.74(7)
C6	0.0253 (4)	0.2789 (3)	0.80562(9)	3.40(8)
C7	0.0633 (3)	-0.0191 (3)	1.00349(8)	3.04(7)
C8	0.2699 (4)	-0.0023 (4)	1.01652(9)	3.84(9)
C9	0.3346 (4)	-0.0250 (4)	1.06882(11)	4.61(11)
C10	0.2005 (5)	-0.0640 (4)	1.10903(9)	4.43(11)
C11	-0.0011 (5)	-0.0822 (4)	1.09642(9)	4.19(10)
C12	-0.0688 (4)	-0.0610 (3)	1.04456(9)	3.69(9)
C13	-0.2233 (4)	0.0234 (5)	0.94291(12)	4.54(12)
H(O2)	0.240 (5)	0.229 (4)	0.9263 (13)	4.5
H(O3)	0.354 (5)	-0.192 (4)	0.7845 (11)	4.2
H(O4)	0.607 (5)	0.043 (4)	0.7550 (12)	4.6
H(O5)	0.116 (5)	0.401 (4)	0.7090 (12)	4.4
H1A	0.034 (4)	-0.142 (4)	0.8867 (11)	4.0
H1B	0.233 (5)	-0.103 (4)	0.9180 (11)	4.0
H3	0.452 (4)	-0.015 (4)	0.8438 (10)	3.4
H4	0.418 (4)	0.268 (4)	0.8049 (11)	3.5
H5	0.120 (4)	0.137 (4)	0.7393 (11)	3.5
H6A	-0.108 (5)	0.291 (4)	0.7876 (11)	4.2
H6B	0.086 (5)	0.384 (4)	0.8262 (11)	4.2
H8	0.365 (5)	0.006 (4)	0.9885 (12)	4.6
H9	0.486 (5)	-0.014 (5)	1.0748 (13)	5.4
H10	0.269 (5)	-0.094 (5)	1.1435 (12)	5.2
H11	-0.106 (5)	-0.108 (4)	1.1241 (12)	5.0
H12	-0.220 (5)	-0.066 (4)	1.0357 (11)	4.5
H13A	-0.266 (6)	0.079 (4)	0.9075 (13)	5.3
H13B	-0.268 (5)	0.094 (5)	0.9690 (14)	5.3
H13C	-0.264 (5)	-0.089 (5)	0.9467 (12)	5.3

B_{eq} is the Mean of the Principal Axes of the Displacement Ellipsoid

Table S3. List of u(i,j) or U values *100 for *N*-(1-deoxy-α-D-tagatopyranos-1-yl)-*N*-methylaniline. E.S.Ds. refer to the last digit printed

	u11(U)	u22	u33	u12	u13	u23
O2	6.51(10)	4.22(8)	3.31(7)	-0.76(8)	-0.23(7)	-0.79(6)
O3	5.49(9)	3.48(7)	4.09(7)	0.02(7)	-0.24(7)	-0.69(6)
O4	5.26(8)	5.69(9)	3.70(7)	1.19(9)	1.24(7)	1.04(7)
O5	5.20(8)	4.31(8)	4.13(8)	-0.27(7)	-0.97(7)	1.37(7)
O6	3.96(7)	4.71(8)	3.85(7)	0.07(7)	-0.01(6)	0.68(7)
N	3.55(8)	6.26(11)	3.50(8)	0.60(9)	0.43(7)	0.56(8)
C1	4.78(11)	4.22(11)	3.26(9)	-0.20(10)	0.37(8)	0.36(8)
C2	3.64(9)	3.31(9)	2.97(8)	-0.15(8)	-0.28(7)	-0.07(7)
C3	3.69(9)	3.43(9)	2.87(8)	-0.14(8)	-0.39(7)	0.02(7)
C4	3.47(9)	3.59(10)	3.14(8)	0.10(8)	0.05(7)	0.19(8)
C5	4.11(10)	3.24(9)	3.08(8)	-0.18(9)	-0.61(8)	0.23(8)
C6	4.75(11)	4.31(11)	3.86(9)	1.01(10)	-0.05(9)	0.62(9)
C7	4.14(10)	3.84(10)	3.57(10)	0.03(9)	0.48(8)	0.09(8)
C8	4.06(10)	6.25(14)	4.27(11)	-0.51(11)	0.20(9)	0.72(10)
C9	5.59(14)	6.91(17)	5.01(12)	-0.52(14)	-1.12(11)	0.12(12)
C10	7.55(16)	5.68(14)	3.60(10)	0.04(14)	-0.64(11)	-0.14(10)
C11	6.93(15)	5.40(13)	3.60(10)	0.26(14)	1.53(11)	-0.04(10)
C12	4.79(11)	5.03(12)	4.21(11)	-0.05(11)	0.81(9)	-0.11(10)
C13	3.72(11)	8.33(20)	5.21(13)	-0.04(13)	-0.01(10)	0.69(14)

Anisotropic Displacement Factors are of the form
Temp=-2*Pi*Pi*(h*h*u11*astar*astar+---+2*h*k*u12*astar*bstar+---)

Table S4. Bond distances (Å) and angles (˚) for *N*-(1-deoxy-α-D-tagatopyranos-1-yl)-*N*-methylaniline

O2-C2	1.392(2)	C2-C3	1.543(3)
O3-C3	1.422(2)	C3-C4	1.543(3)
O4-C4	1.431(3)	C4-C5	1.523(3)
O5-C5	1.426(2)	C5-C6	1.511(3)
O6-C2	1.422(2)	C7-C8	1.403(3)
O6-C6	1.429(3)	C7-C12	1.385(3)
N-C1	1.466(3)	C8-C9	1.388(4)
N-C7	1.402(3)	C9-C10	1.372(4)
N-C13	1.464(3)	C10-C11	1.370(4)
C1-C2	1.530(3)	C11-C12	1.383(3)

C2-O6-C6	113.0(2)	O4-C4-C5	108.3(2)
C1-N-C7	118.9(2)	C3-C4-C5	110.0(2)
C1-N-C13	114.2(2)	O5-C5-C4	108.0(2)
C7-N-C13	116.8(2)	O5-C5-C6	110.5(2)
N-C1-C2	110.4(2)	C4-C5-C6	109.2(2)
O2-C2-O6	111.5(2)	O6-C6-C5	110.6(2)
O2-C2-C1	110.2(2)	N-C7-C8	120.1(2)
O2-C2-C3	106.9(2)	N-C7-C12	122.7(2)
O6-C2-C1	106.2(2)	C8-C7-C12	117.2(2)
O6-C2-C3	108.9(2)	C7-C8-C9	120.3(2)
C1-C2-C3	113.3(2)	C8-C9-C10	121.6(2)
O3-C3-C2	106.2(2)	C9-C10-C11	118.4(2)
O3-C3-C4	112.2(2)	C10-C11-C12	121.0(2)
C2-C3-C4	108.9(2)	C7-C12-C11	121.6(2)
O4-C4-C3	112.0(2)		

Table S5. Torsion angles (˚) for *N*-(1-deoxy-α-D-tagatopyranos-1-yl)-*N*-methylaniline

C6	O6	C2	O2	55.3(3)	C6	O6	C2	C1	175.3(4)
C6	O6	C2	C3	-62.4(3)	C2	O6	C6	C5	63.0(3)
C7	N	C1	C2	-125.3(4)	C13	N	C1	C2	90.3(4)
C1	N	C7	C8	46.6(3)	C1	N	C7	C12	-136.8(4)
C13	N	C7	C8	-169.9(5)	C13	N	C7	C12	6.6(3)
N	C1	C2	O2	48.1(2)	N	C1	C2	O6	-72.8(3)
N	C1	C2	C3	167.7(4)	O2	C2	C3	O3	175.9(4)
O2	C2	C3	C4	-63.1(3)	O6	C2	C3	O3	-63.5(3)
O6	C2	C3	C4	57.5(2)	C1	C2	C3	O3	54.4(3)
C1	C2	C3	C4	175.4(4)	O3	C3	C4	O4	-58.4(3)
O3	C3	C4	C5	62.1(3)	C2	C3	C4	O4	-175.6(4)
C2	C3	C4	C5	-55.2(2)	O4	C4	C5	O5	-62.3(3)
O4	C4	C5	C6	177.6(4)	C3	C4	C5	O5	175.0(4)
C3	C4	C5	C6	54.9(3)	O5	C5	C6	O6	-176.1(4)
C4	C5	C6	O6	-57.4(3)	N	C7	C8	C9	175.8(5)
C12	C7	C8	C9	-0.9(3)	N	C7	C12	C11	-175.3(5)
C8	C7	C12	C11	1.3(3)	C7	C8	C9	C10	-0.1(3)
C8	C9	C10	C11	0.7(3)	C9	C10	C11	C12	-0.4(3)
C10	C11	C12	C7	-0.7(3)					