Solid-State NMR Spectroscopy of Saccharides: Complete ^{1}H Resonance Assignment of β-Maltose from ^{1}H-^{1}H DQ-SQ CRAMPS and ^{1}H (DQ-DUMBO)-^{13}C (SQ) Refocused INEPT 2D Spectra and First-Principles GIPAW Calculations

Supporting Information

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Table S1. Phases used for ^{1}H DQ – ^{13}C INEPT pulse sequence given in Figure 1.

Φ_1	x, y, –x, –y
Φ_2	{x, x, x, x, x, x, x, x}*4 (–x, –x, –x, –x, –x, –x, –x, –x}*4
Φ_3	y, y, y, y, y, y, y, y, –y, –y, –y, –y, –y, –y, –y, –y
Φ_4	x, x, x, x, –x, –x, –x, –x
Φ_5	x, x, x, x, –x, –x, –x, –x
Φ_6	{x, x, x, x}*4 { y, y, y, y,}*4
	{–x, –x, –x, –x }*4 { –y, –y, –y, –y }*4
Φ_7	{x, x, x, x, –x, –x, –x, –x}*2 {y, y, y, y, –y, –y, –y, –y}*2
Φ_R	{x, –x, x, –x}*2 {–x, x, –x, x}*2
	{y, –y, y, –y}*2 {–y, y, –y, y}*2

Table S2. ^{1}H DQ correlations[a] involving the OH and CH_2 ^{1}H nuclei (< 2.9 Å) in β-maltose monohydrate, **1**

		H-H Å	δ_{SQ_1} (^{1}H) /ppm	δ_{SQ_2} (^{1}H) /ppm	δ_{DQ} (^{1}H) /ppm			H-H Å	δ_{SQ_1} (^{1}H) /ppm	δ_{SQ_2} (^{1}H) /ppm	δ_{DQ} (^{1}H) /ppm
OH1'	CH1'	2.134	7.2	4.2	11.4	**OH6'**	OH2'	2.137	6.2	6.2	12.4
	OH6	2.426	7.2	6.2	13.4		WH2	2.144	6.2	6.2	12.4
	OH6	2.468	7.2	6.2	13.4		WH1	2.274	6.2	6.2	12.4
	WH1	2.706	7.2	6.2	13.4		CH6'b	2.294	6.2	3.7	9.9
	CH6b	2.884	7.2	3.7	10.9		CH5'	2.571	6.2	3.7	9.9
OH3	CH3	2.103	6.4	3.7	10.1		OH3	2.785	6.2	6.4	12.6
	WH1	2.338	6.4	6.2	12.6		CH6'a	2.865	6.2	3.7	9.9
	WH2	2.423	6.4	6.2	12.6	**WH1**	WH2	1.591	6.2	6.2	12.4
	WH2	2.470	6.4	6.2	12.6		OH6	2.255	6.2	6.2	12.4
	OH4	2.593	6.4	5.3	11.7		OH6'	2.274	6.2	6.2	12.4
	CH6'b	2.696	6.4	3.7	10.1		OH3	2.338	6.2	6.4	12.6
	OH6'	2.785	6.4	6.2	12.6		CH6a	2.631	6.2	3.7	9.9
OH2'	OH6'	2.137	6.2	6.2	12.4		CH6b	2.670	6.2	3.7	9.9
	CH2'	2.250	6.2	3.0	9.2		OH1'	2.706	6.2	7.2	13.4
	OH4	2.453	6.2	5.3	11.5	**WH2**	WH1	1.591	6.2	6.2	12.4
	CH6'b	2.643	6.2	3.7	9.9		OH6'	2.144	6.2	6.2	12.4
	CH6'b	2.788	6.2	3.7	9.9		OH3	2.423	6.2	6.4	12.6
	CH6'a	2.885	6.2	3.7	9.9		OH3	2.470	6.2	6.4	12.6
OH6	WH1	2.255	6.2	6.2	12.4		CH2	2.686	6.2	3.7	9.9
	CH6a	2.284	6.2	3.7	9.9	**OH3'**	OH4	2.007	5.6	5.3	10.9
	OH1'	2.426	6.2	7.2	13.4		CH3'	2.234	5.6	3.7	9.3
	OH1'	2.468	6.2	7.2	13.4		CH4	2.353	5.6	3.1	8.7
	CH6'a	2.690	6.2	3.7	9.9		OH2	2.457	5.6	5.1	10.7
	CH6b	2.867	6.2	3.7	9.9		CH6b	2.483	5.6	3.7	9.3

		H-H Å	δ_{SQ_1} (^{1}H) /ppm	δ_{SQ_2} (^{1}H) /ppm	δ_{DQ} (^{1}H) /ppm			H-H Å	δ_{SQ_1} (^{1}H) /ppm	δ_{SQ_2} (^{1}H) /ppm	δ_{DQ} (^{1}H) /ppm
OH4	OH3'	2.007	5.3	5.6	10.9	**CH6b**	CH6a	1.771	3.7	3.7	7.4
	CH4	2.323	5.3	3.1	8.4		OH3'	2.483	3.7	5.6	9.3
	OH2'	2.453	5.3	6.2	11.5		CH5	2.485	3.7	3.6	7.3
	OH3	2.593	5.3	6.4	11.7		WH1	2.670	3.7	6.2	9.9
OH2	OH3'	2.457	5.1	5.6	10.7		OH6	2.867	3.7	6.2	9.9
	CH6a	2.497	5.1	3.7	8.8		OH1'	2.884	3.7	7.2	10.9
	CH3	2.630	5.1	3.7	8.8	**CH6'a**	CH5'	2.429	3.7	3.7	7.4
	CH4'	2.802	5.1	3.4	8.5		OH6	2.690	3.7	6.2	9.9
	CH1	2.848	5.1	4.8	9.9		OH6'	2.865	3.7	6.2	9.9
	CH2	2.854	5.1	3.7	8.8		OH2'	2.885	3.7	6.2	9.9
CH6a	CH6b	1.771	3.7	3.7	7.4	**CH6'b**	CH6'a	1.780	3.7	3.7	7.4
	OH6	2.284	3.7	6.2	9.9		OH6'	2.294	3.7	6.2	9.9
	CH4	2.483	3.7	3.1	6.8		CH5'	2.406	3.7	3.7	7.4
	OH2	2.497	3.7	5.1	8.8		OH2'	2.643	3.7	6.2	9.9
	WH1	2.631	3.7	6.2	9.9		OH3	2.696	3.7	6.4	10.1
							OH2'	2.788	3.7	6.2	9.9

[a] The stated ^{1}H chemical shifts are the experimental chemical shifts measured at 298 K bearing gas temperature and reported in Tables 1 to 3.

Figure S1. ^{13}C CP MAS (12.5 kHz) NMR (^{1}H Larmor frequency 500 MHz, Warwick) spectra of β-maltose monohydrate, **1**, doped with $MnCl_2$, whereby the bearing gas temperature is regulated at (a) 348 K, (b) 323 K (c) 298 K (d) 248 K. Chemical shift values are stated in Table 6.