Solid-State NMR Study of the Charge-Transfer Complex between Ubiquinone-8 and Disulfide Bond Generating Membrane Protein DsbB

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Supporting Information

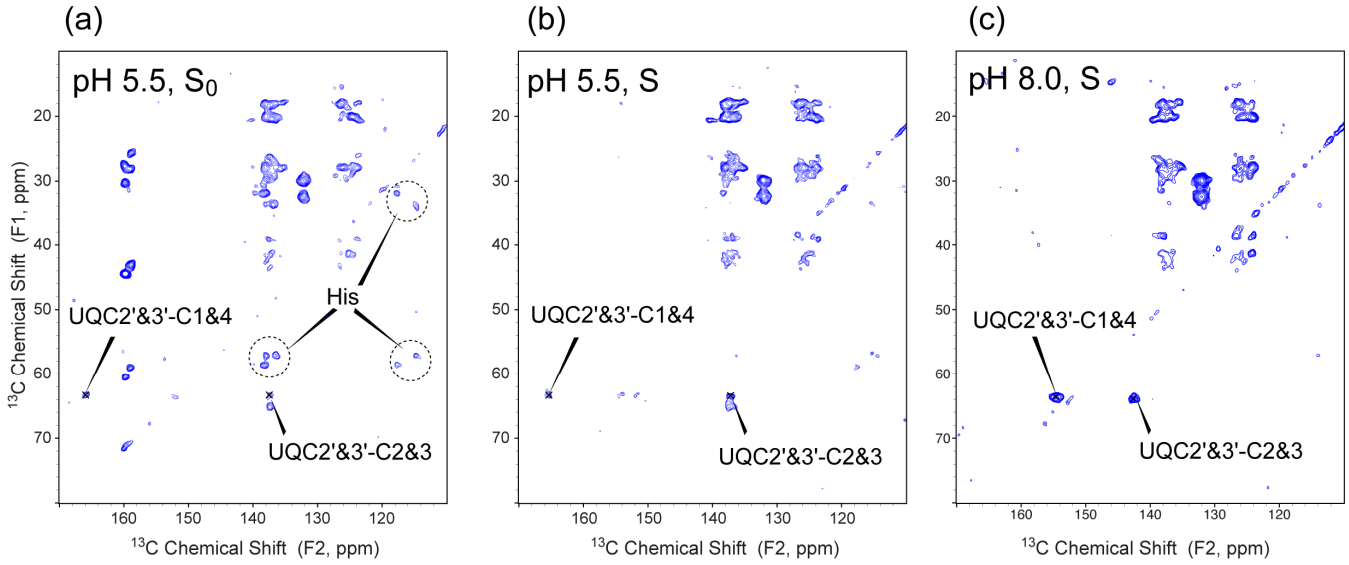

Figure S1. ^{13}C-^{13}C 2D correlation spectra of FLYPW-DsbB(C41S) with 100 ms DARR mixing (B_0 = 14.1 T, T_{sample} = –16.6 °C, 1.5 s pulse delay, maximum t_1 = 10.24 ms, maximum t_2 = 20.48 ms) and 1.8 ms Hahn-echo before acquisition (a) without ^{15}N REDOR pulses (S_0 spectrum) at 5.5, (b) with ^{15}N REDOR pulses (S spectrum) at pH 5.5 and (c) with ^{15}N REDOR pulses (S spectrum) at pH 8.0.

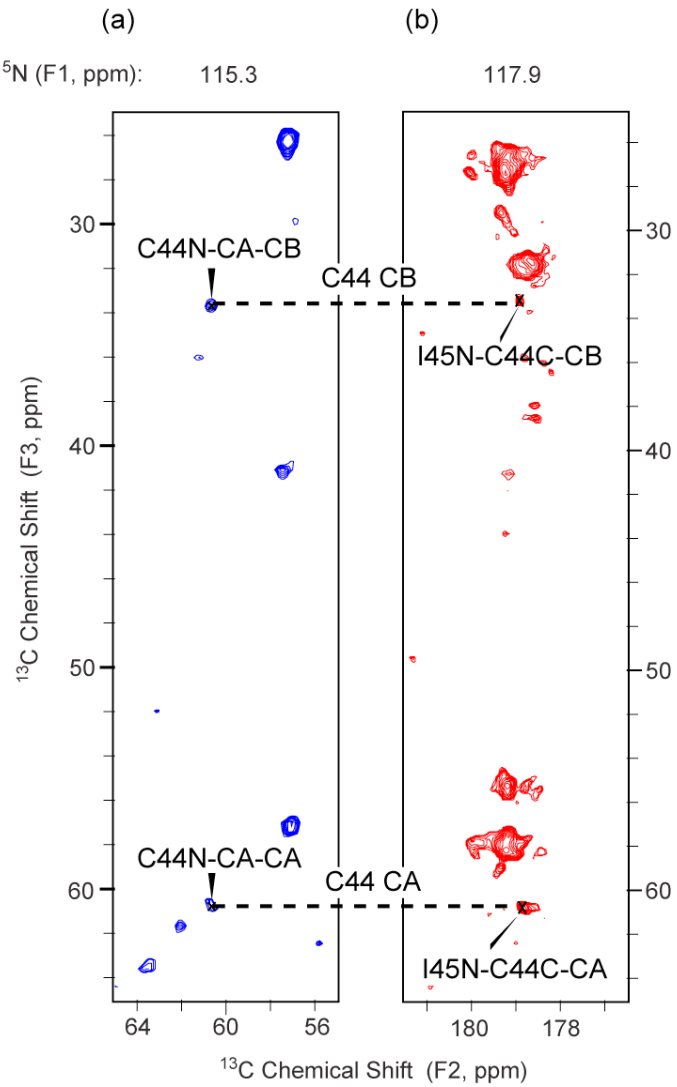

Figure S2. Strip plot of (a) NCACX 3D spectrum and (b) NCOCX 3D spectrum of U-DsbB(C41S) at pH 8.0 with 90 ms DARR mixing under 11.111 kHz MAS (B_0 = 11.8 T, T_{sample} = –12 °C, 1.5 s pulse delay). The assignments of C44 are marked.

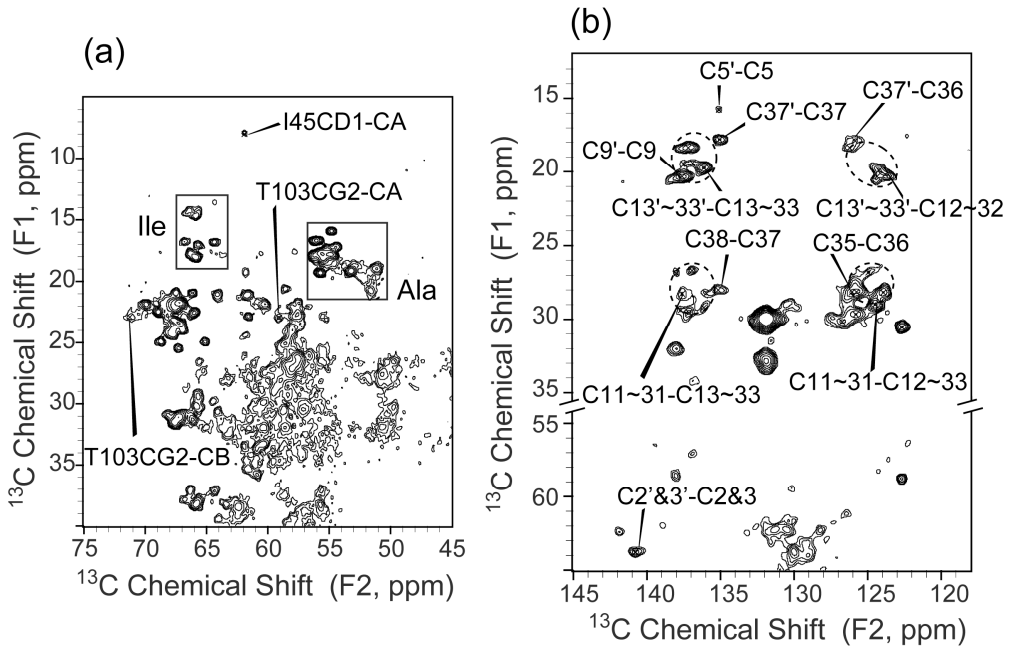

Figure S3. Expansions of (a) Ile, Ala, and other CA-CB correlations and (b) UQ-8 resonances regions from a ^{13}C-^{13}C 2D correlation spectrum of DsbA(C33S)/DsbB* complex at 12.5 kHz MAS with 25 ms DARR mixing (B_0 = 17.6 T, T_{sample} = –12 °C, 1.5 s pulse delay, 8 scans per row, maximum t_1 = 15.36 ms, maximum t_2 = 20.48 ms).

Table S1. ^{13}C chemical shifts of UQ in various states (DSS scale, the solution shifts in TMS scale are converted by +2.0 ppm[1]). Carbonyl chemical shifts in DsbB with significant difference are bolded.

Chemical Shifts (ppm)	UQ-10[2]	Ubiquinol-10[3]	UQ-8 in DsbB(C41S) pH 8.0	UQ-8 in DsbB(C41S) pH 5.5
C=O (1, 4)	185.9, 186.7	141.9, 142.1	**154.7**	**165.5**
C=C (2, 3)	146.4, 146.2	138.9, 138.8	142.4	137.5
C=C (5, 6)	140.8, 143.7	120.0, 124.0	127.4, 135.6	126.8, 137.5
C=C (13~37)	133.6, 136.9	137.3~136.9	139.0~135.2	139.0~135.2
C=C (12~36)	126.2	126.4~125.9	126.6~123.6	126.6~123.6
C=C (9)	139.6	137.3	140.2	140.2
C=C (8)	120.8	124.3	123.7	123.7
CH_3O (2', 3')	63.0	62.8, 62.7	63.6, 64.0	63.4, 65.2
CH_2 (10~34)	41.7	41.8~41.7	43.4~41.6	43.4~41.6
CH_2 (11~35)	28.5	28.8~28.5	30.3~27.3	30.3~27.3
CH_3 (38)	27.7	28.9	28.2	28.2
CH_2 (7)	27.3	27.3	26.6	26.6
CH_3 (9'~37')	18.2	18.0	20.7~17.9	20.7~17.9
CH_3 (5')	13.9	14.0	15.9	15.0

(1) Morcombe, C. R.; Zilm, K. W. *J. Magn. Reson.* **2003**, *162*, 479.

(2) Vanliemt, W. B. S.; Steggerda, W. F.; Esmeijer, R.; Lugtenburg, J. *Recl. Trav. Chim. Pays-Bas* **1994**, *113*, 153.

(3) Afri, M.; Ehrenberg, B.; Talmon, Y.; Schmidt, J.; Cohen, Y.; Frimer, A. A. *Chem. Phys. Lipids* **2004**, *131*, 107.