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Supplement of

Dual bilinear rotations

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S1 The COB-variant of the quadruple J -resolved pulse sequence

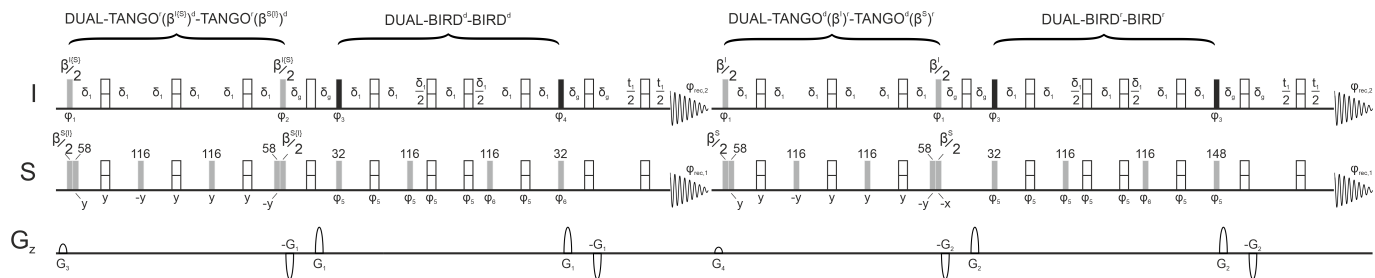


Figure S1. Quadruple J -resolved experiment designed to rapidly acquire four hetero- and homonuclear decoupled spectra for differentiating $I\{S_{2n}\}$, $I\{S_{2n+1}\}$, $S\{I_{2n}\}$, and $S\{I_{2n+1}\}$ employing the coupling compensated bilinear rotations as described in (Woordes et al., 2025) with typically $I = {}^1\text{H}$ and $S = {}^{13}\text{C}$. Black, solid bars describe hard 90° pulses, while gray solid bars stand for hard pulses with flip angles as annotated. Open bars with a dividing central line describe universal rotation 180° pulses. Delays δ_1 correspond to 2.583 ms. The delays with duration δ_g are determined by corresponding gradient durations and necessary gradient recovery delays. Gradients of $250 \mu\text{s}$ duration and a recovery delay of $50 \mu\text{s}$ have been used on our spectrometer with typical strengths of $G_1 = 81\%$, $G_2 = 79\%$, $G_3 = 29\%$, and $G_4 = 19\%$ of the maximum gradient strength of the probehead ($\approx 50 \text{ G/cm}$). Phases of the pulses are all x unless indicated otherwise. A basic phase cycle has been applied with $\varphi_1 = x, -x$, $\varphi_2 = -x, x$, $\varphi_3 = x, -x$, $y, -y$, $\varphi_4 = -x, x, -y, y$, $\varphi_5 = x, y, -x, -y$, $\varphi_6 = -x, -y, x, y$, $\varphi_{rec,1} = x, -x, -x, x$, and $\varphi_{rec,2} = x, -x$. Please note that the sequence requires dual receive capabilities.

S2 Spectra of the COB-variant of the quadrupole J -resolved experiment for glucose

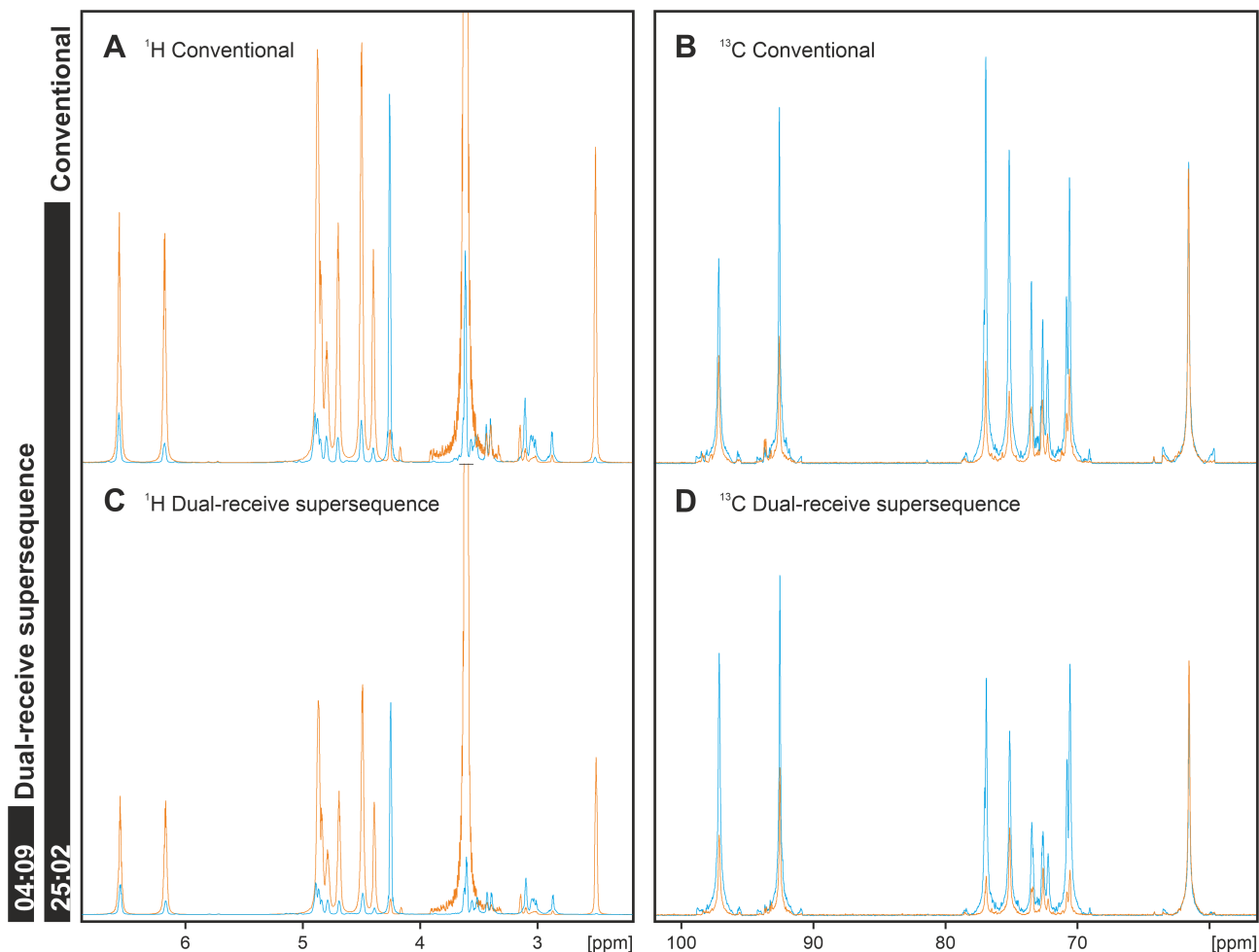


Figure S2. Various fully heteronuclear and homonuclear decoupled ^1H (A, C) and ^{13}C (B, D) 1D spectra on uniformly ^{13}C labeled glucose dissolved in $\text{DMSO-}d_6$. All spectra are obtained via 2D J -resolved experiments either in the conventional matter where all four need to be measured separately (A, B), or in a quadrupole J -resolved experiment as described in Fig. S1. In the conventional case the total experiment time amounts to 25 minutes and 2 seconds, whereas the implementation of dual-receiver with NORD type interleaving reduces the time to just 4 minutes and 9 seconds, as highlighted in the bars on left of the figure. The orange and blue color indicate $I\{S_{2n}\} / S\{I_{2n}\}$, and $I\{S_{2n+1}\} / S\{I_{2n+1}\}$ selection. Assignment can be found in the main text figures 4 and 5. $\beta^I = \beta^{I\{S\}} = \beta^{S\{I\}} = \beta^S = 90^\circ$; ^1H pulses with 90° duration of $9.7 \mu\text{s}$ were irradiated at 4.48 ppm, ^{13}C pulses with 90° duration of $12.0 \mu\text{s}$ at 80.0 ppm; acquisition times were 250 ms in the indirect t_1 dimensions using 64 increments each; direct acquisition times with 4k complex data points were 350 ms for ^1H and 160 ms for ^{13}C . Spectra were zero-filled to $128 \times 8\text{k}$ points. 2D J -resolved type spectra were processed using sine-apodization in the direct dimension and either sine- or exponential-apodization in the indirect dimension for the ^1H and ^{13}C spectra, respectively. Subsequently, the 2D spectra were tilted and the projection along the direct dimension is shown.

S3 2D J -resolved subspectra for glucose

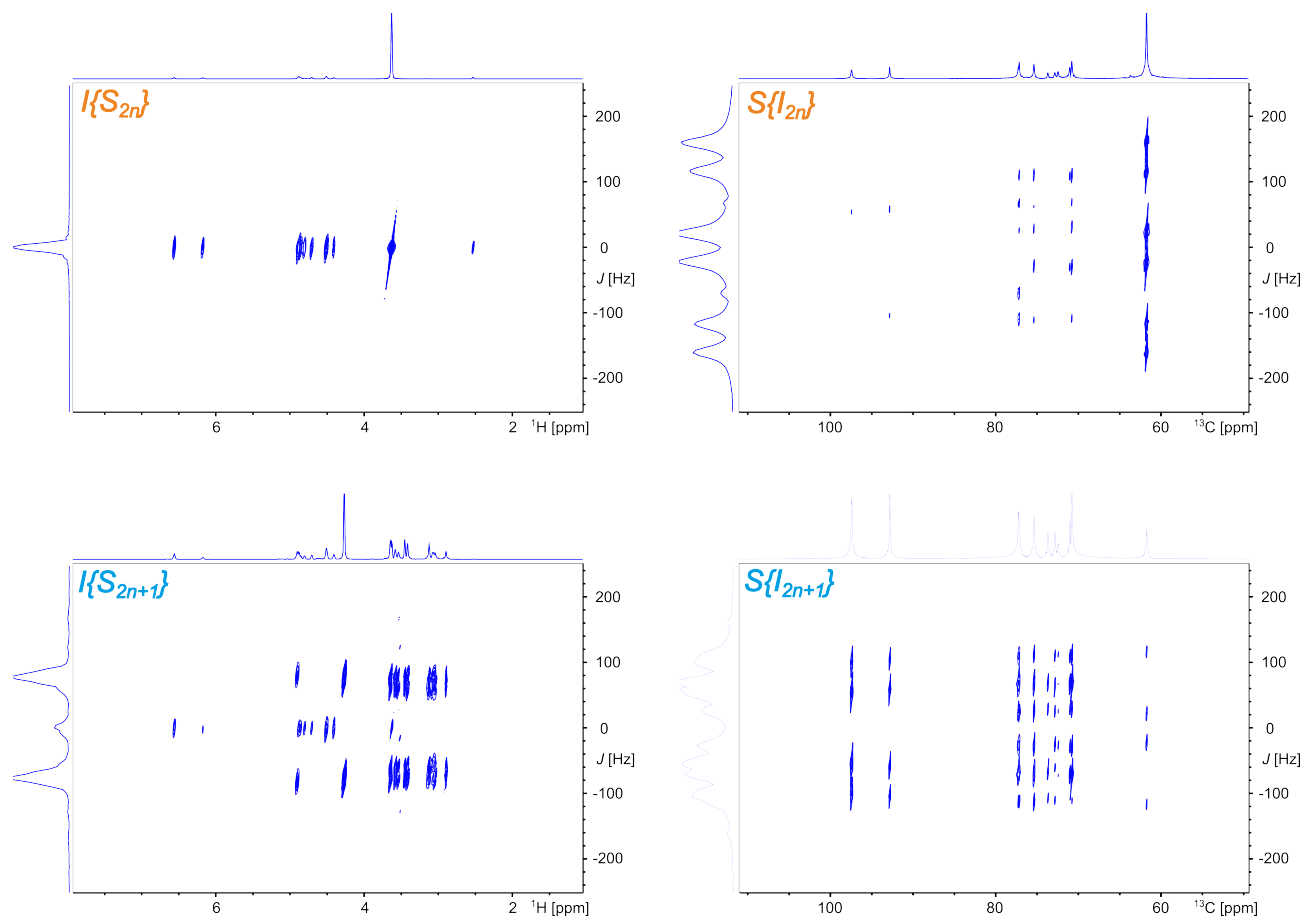


Figure S3. 2D J -subspectra for the projections shown in Figs. 4 and 5 in the main text. top left) non- ^{13}C -bound protons; bottom left) ^{13}C -bound protons; top right) ^{13}C spectrum of carbons with an even number of attached protons; bottom right) ^{13}C spectrum of carbons with an odd number of attached protons.

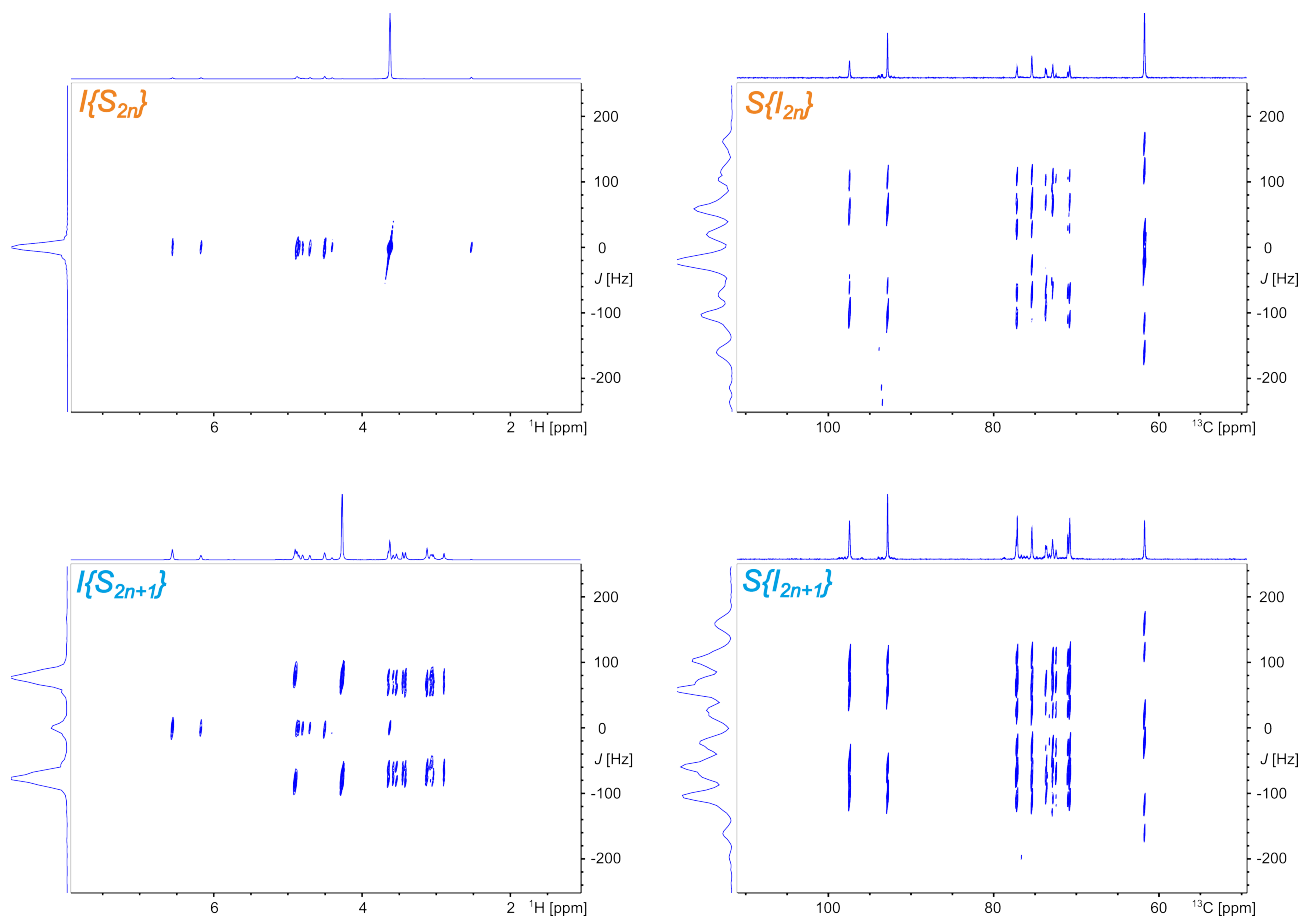


Figure S4. 2D J -subspectra for the COB-enhanced version of D-glucose. top left) non- ^{13}C -bound protons; bottom left) ^{13}C -bound protons; top right) ^{13}C spectrum of carbons with an even number of attached protons; bottom right) ^{13}C spectrum of carbons with an odd number of attached protons.

S4 Proton subspectra of the quadruple J -resolved experiment for mixture

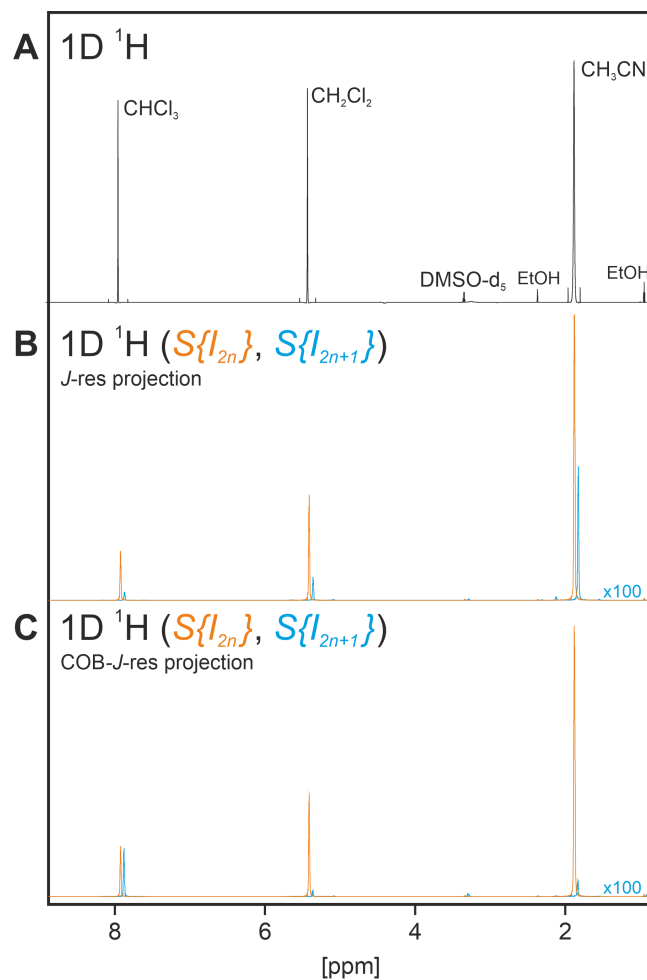


Figure S5. In Fig. 6 of the main text, ^{13}C subspectra of the quadruple J -resolved experiment are shown for a mixture of four small molecule compounds dissolved in DMSO- d_6 . The proton counterparts are summarized here. Please be aware that non- ^{13}C -bound protons form the majority for the natural abundance sample. The opposite experiment leads to a very weak spectrum of natural abundance ^{13}C -bound protons. No additional information is gained, as, of course, all molecules have ^{12}C and ^{13}C bound protons. Signal intensities of the blue spectra may also be compromised by residual ^{12}C bound proton artefacts.

S5 2D J -resolved subspectra for mixture

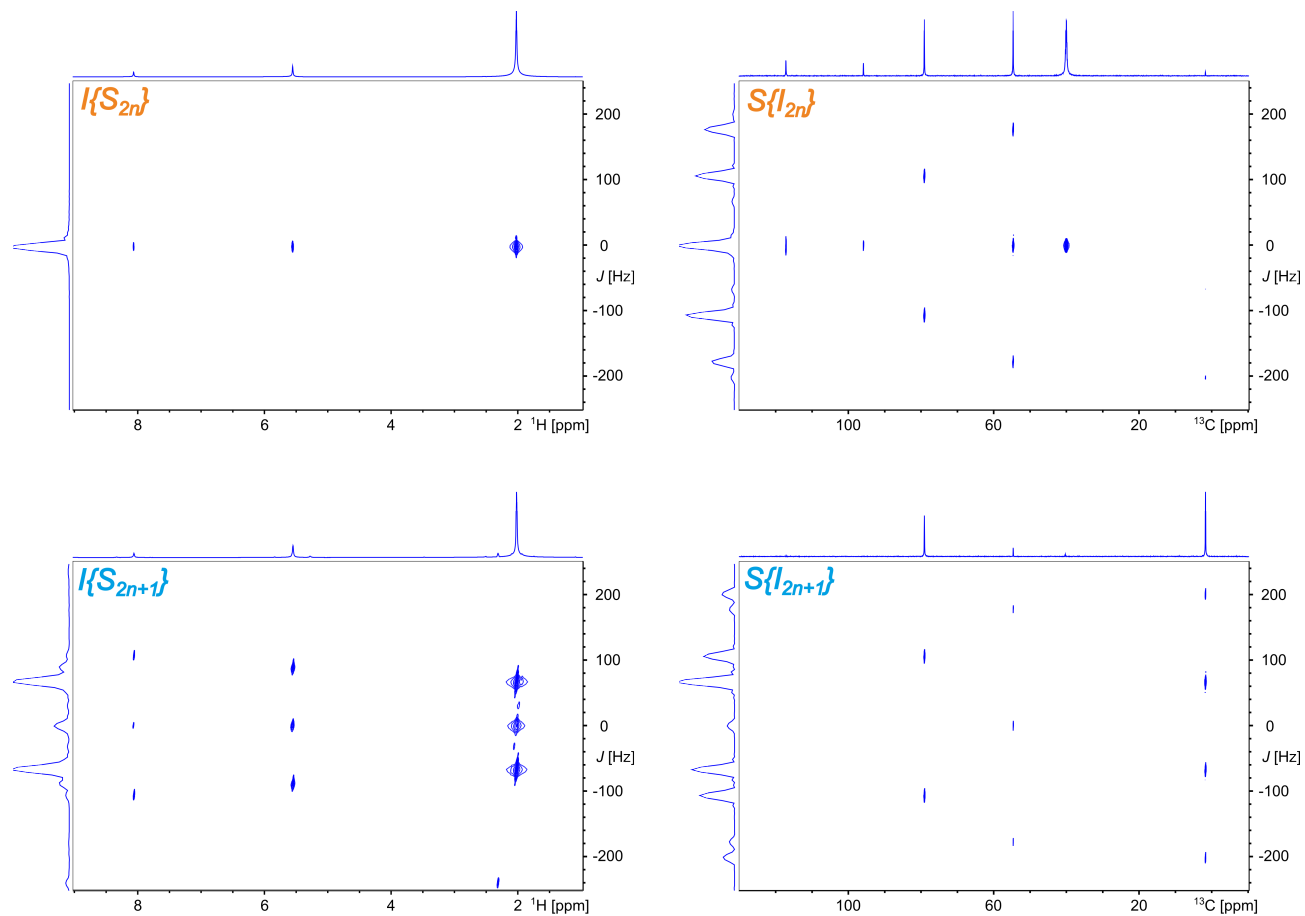


Figure S6. 2D J -subspectra for the projections shown in Fig. 6 and S5. top left) non- ^{13}C -bound protons; bottom left) ^{13}C -bound protons; top right) ^{13}C spectrum of carbons with an even number of attached protons; bottom right) ^{13}C spectrum of carbons with an odd number of attached protons.

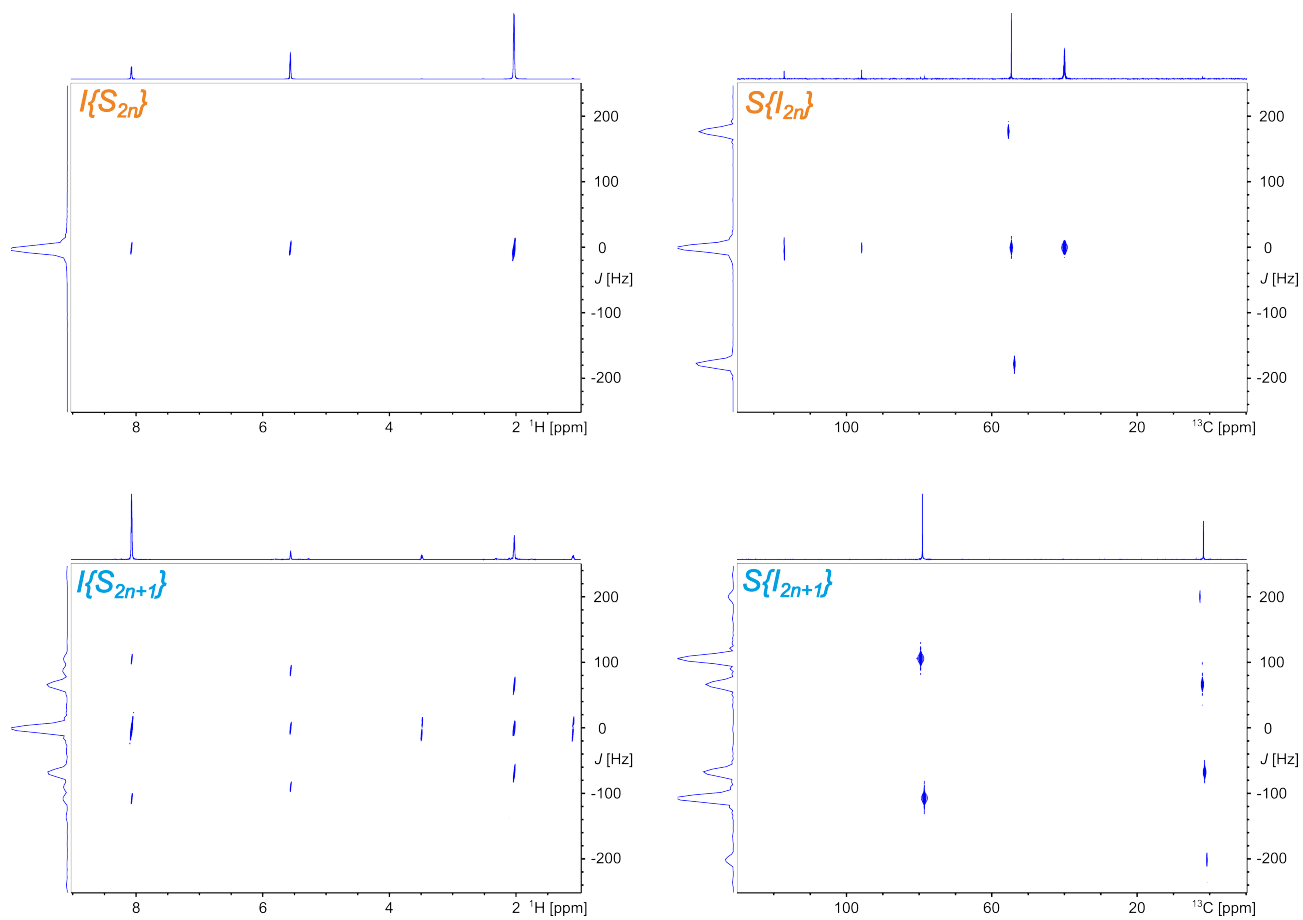


Figure S7. 2D J -subspectra for the COB-enhanced version of the mixture described in the main text. top left) non- ^{13}C -bound protons; bottom left) ^{13}C -bound protons; top right) ^{13}C spectrum of carbons with an even number of attached protons; bottom right) ^{13}C spectrum of carbons with an odd number of attached protons.

S6 Broadband refocusing pulse shape used (^{13}C)

Broadband universal rotation 180° pulse shape (BURBOP-180) for a bandwidth of 10 kHz, an rf-amplitude $\nu_{\text{rf}} = 15$ kHz, B_1 -field compensation of $\pm 10\%$, and a pulse length t_p of $300 \mu\text{s}$ in Bruker Topspin shape format (amplitude, phase). Quality factor of the pulse shape was >0.99999 . Please note that it is *not* J -compensated like the BUBI (Ehni and Luy, 2013) or BUBU (Ehni et al., 2022) pulse sandwiches. The pulses, however, are very short and unwanted effects within the bilinear rotations will be small. To save space, we present it in two-column format.

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S7 Broadband refocusing pulse shape used (^1H)

Broadband universal rotation 180° pulse shape (BURBOP-180) for a bandwidth of 5 kHz, an rf-amplitude $\nu_{\text{rf}} = 20$ kHz, B_1 -field compensation of $\pm 30\%$, and a pulse length t_p of $300 \mu\text{s}$ in Bruker Topspin shape format (amplitude, phase). Quality factor of the pulse shape was >0.99999 . Please note that it is *not* J -compensated like the BUBI (Ehni and Luy, 2013) or BUBU (Ehni et al., 2022) pulse sandwiches. The pulses, however, are very short and unwanted effects within the bilinear rotations will be small. To save space, we present it in two-column format.

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