Supporting Information

Real-time broadband proton-homodecoupled CLIP/CLAP-HSQC for automated measurement of heteronuclear one-bond coupling constants

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Experimental Section

Experiments on the modified sugar (methyl α-D-mannopyranoside) and on the PAF protein were performed at 11.7 T (500 MHz for ^{1}H) on a Bruker Avance II spectrometer (Bruker BioSpin GmbH, Rheinstetten, Germany) equipped with a TXI z-gradient probe. For all measurements the nominal temperature was set to 298 K unless indicated otherwise. Spectra were processed with TopSpin 2.1 or 3.0 (Bruker Biospin GmbH, Karlsruhe, Germany).

^{1}H-^{13}C CLIP/CLAP-HSQC spectra were recorded with proton 90° pulses of 13.9 μs and carbon 90° pulses of 15.7 μs. ^{1}H-^{15}N CLIP/CLAP-HSQC spectra were collected with proton 90° pulses of 12.7 μs and nitrogen 90° pulses of 37 μs. Purging gradient pulses, G_1 and G_3 were set to 13% and 27% of maximum gradient strength (50.1 G/cm), and CTP selection gradient pulses, G_5 and G_6 were set to 61 % and 31 % of maximum gradient strength (50.1 G/cm). Other experimental details are given in the figure legends.

Studies on (+)-isopinocampheol were performed at 14.1 T (600 MHz for ^{1}H) on a Bruker Avance III spectrometer (Bruker BioSpin GmbH, Rheinstetten, Germany) equipped with a 5 mm TBI probe equipped with a z-gradient. The samples used in a previous study[1] were employed. Temperature was regulated at 300 K (correction according to ref. [2]).

An experiment using interleaved acquisition for collecting ^{1}H-^{13}C CLIP- and CLAP-HSQC spectra without homodecoupling was used (Bruker pulse program hsqcetgpiajcsp.2), with adiabatic inversion and refocusing pulses on the X-channel. Spectra were collected with 16 dummy scans, a 16 step phase cycle, and a total of 512 accumulations in the indirect dimension, resulting in 128 complex data points in F_1 for each CLIP- and CLAP-HSQC dataset. Spectral widths used were 10.41 ppm in the ^{1}H and 70.0 ppm in the ^{13}C dimension, with the transmitter frequencies placed at 1.5 ppm and 45 ppm, respectively. In the direct dimension, 4096 complex data points were collected, resulting in a nominal FID resolution of 1.53 Hz. Hard 90° pulses of 9.1 μs and 12.75 μs length were used for the proton and carbon channels, respectively. For carbon inversion, 0.5 ms smoothed Chirp pulses sweeping over a frequency band of 60 kHz were used (with adiabaticity factor Q = 5). Carbon refocusing was achieved using a four-Chirp composite pulse of 2 ms duration but otherwise identical settings. Two *z*-field gradient pulses of 80% and 20.1% of the maximum gradient amplitude of 53.5 G/cm, both of smoothed square shape and 1 ms duration and followed by 200 μs recovery delays, were used for coherence selection, including echo-antiecho encoding. INEPT delays were optimized for a coupling constant of 135 Hz and the recovery delay was set to 2.25 s to give an overall experiment duration of 6 h 40 min.

CLIP- and CLAP-HSQC spectra with real-time BIRD decoupling were collected as individual experiments, each of 3 h 20 min duration. To obtain equal measurement durations for the experiments, the relaxation delay was shortened to compensate for the lengthening of the effective acquisition duration due to homodecoupling, resulting in relaxation delays of 1 s for experiments with a FID resolution of 0.76 Hz and 20.48 ms chunk length (8192 complex points in F_2), of 1.95 s for experiments with a FID-resolution of 1.53 Hz and 20.48 ms chunk length, of 1.84 s for experiments with 15.36 ms chunk length or of 1.65 s for experiments with 10.24 ms chunk length (see Table S7).

Spectral widths, transmitter frequencies, resolutions (unless otherwise stated) and pulse lengths were chosen as for the experiments without homodecoupling. INEPT and BIRD delays were both optimized for a coupling constant of 135 Hz. For insertion of homodecoupling blocks, the acquisition was interrupted at regular intervals of 20.48 ms, 15.36 ms or 10.24 ms, corresponding to 32, 43 or 64 interruptions, respectively. All gradient pulses were of half sine shape, with 0.2 ms length if applied during homodecoupling elements, and with 1 ms length elsewhere. All gradient pulses were followed by a 200 µs recovery delay. Gradient amplitudes were G1 = 55 %, G2 = 80 %, G3 = 32 %, G4 = 20.1 %, G5 = 11 % and G6 = 14 % of the maximum gradient amplitude of 53.5 G/cm (for numbering, see Figure 1).

Time domain data for (+)-isopinocampheol were zero-filled once and forward linear prediction with 32 coefficients was used in the indirect dimension, while no linear prediction was used in the direct dimension. Time domain signals were multiplied with π/2-shifted squared cosine function (QSINE, SSB: 2) in both dimensions prior to Fourier transformation.

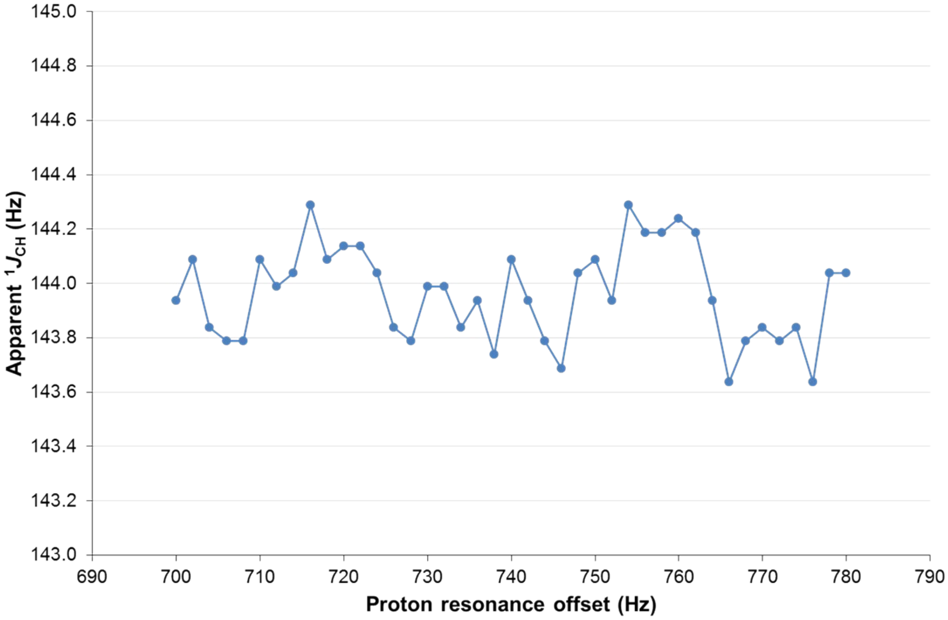

Figure S1A. Apparent ${}^{1}J_{\mathrm{CH}}$ values measured by the phase program optimized real-time BIRD$^{(d)}$ CLAP-HSQC sequence as a function of proton resonance offset. The spectrometer operating (carrier) frequency was varied from 700 Hz up to 780 Hz off-resonance from the CH_3-signal of methyl α-D-mannopyranoside in steps of 2 Hz, to give 40 experiments. The 40 spectra yielded an average value of ${}^{1}J_{\mathrm{CH}}$ = 143.96 Hz with a maximum deviation of 0.42 Hz and a standard deviation (σ_J) of 0.17 Hz.

Figure S1B. Apparent ${}^{1}J_{\mathrm{CH}}$ values measured by the phase program optimized real-time BIRD$^{(d)}$ CLAP-HSQC sequence as a function of proton resonance offset. The spectrometer operating (carrier) frequency was varied from 1900 Hz up to 1980 Hz off-resonance from the CH_3-signal of methyl α-D-mannopyranoside in steps of 2 Hz, to give 40 experiments. The 40 spectra yielded an average value of ${}^{1}J_{\mathrm{CH}}$ = 144.04 Hz with a maximum deviation of 1.05 Hz and a standard deviation (σ_J) of 0.40 Hz.

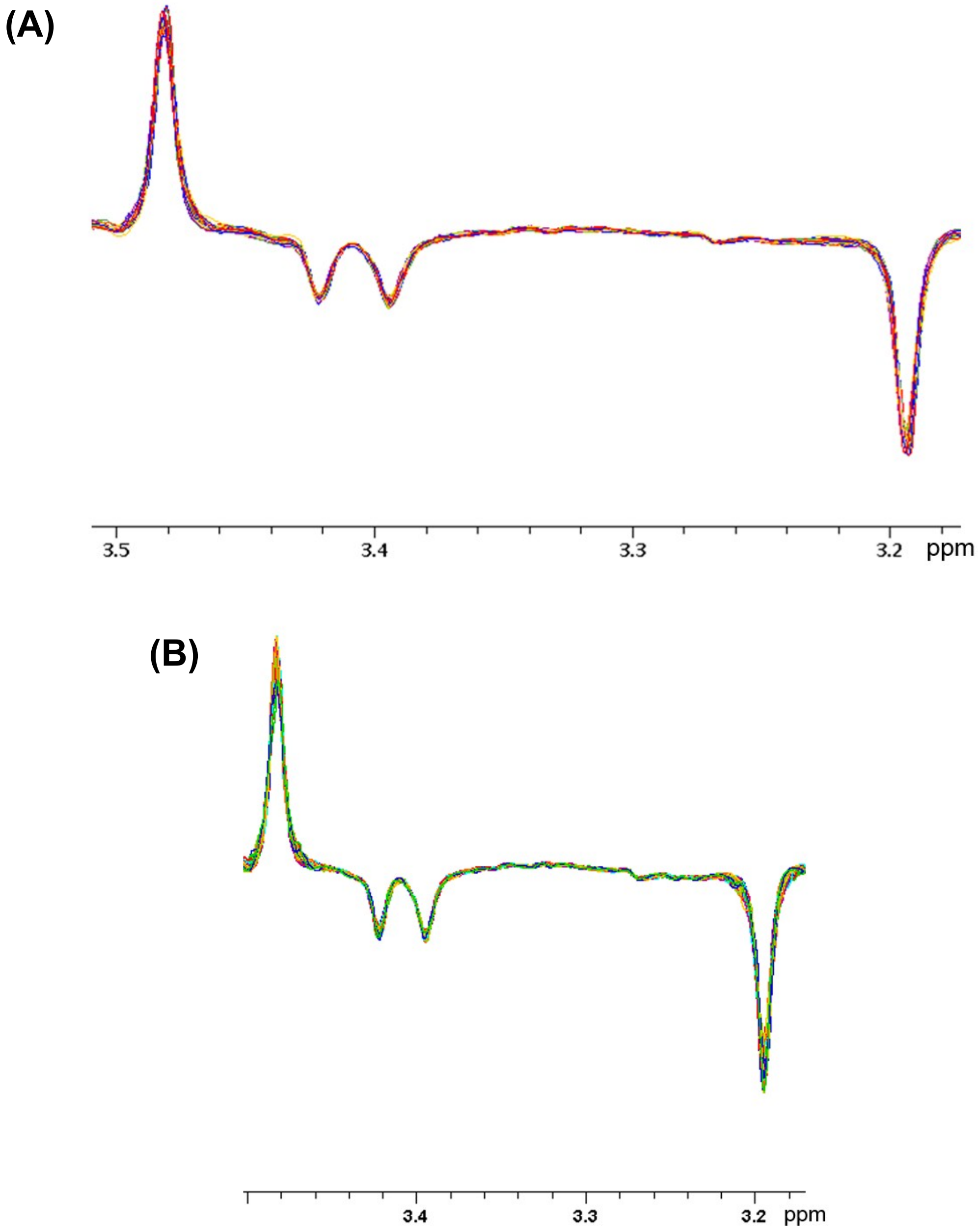

Figure S2. Overlays of 40 spectra of methyl α-D-mannopyranoside (0.345 M in D_2O) recorded with the real-time BIRD$^{(d)}$ CLAP-HSQC pulse sequence of Figure 1 using optimized chunk to chunk and scan to scan phase steps during real-time acquisition of the proton-homodecoupled FID. For testing the offset dependence of the proposed experiment, the proton carrier frequency was varied in two frequency ranges: from 700 to 780 Hz off-resonance from the CH_3-signal of methyl α-D-mannopyranoside (**A**), and from 1900 to 1980 Hz off-resonance (**B**), both in steps of 2 Hz, to give 40 experiments in each case. The 40 spectra in the 700 – 780 Hz offset range (**A**) yielded an average value of $^1J_{CH}$ = 143.96 Hz with a maximum deviation of 0.42 Hz, a standard deviation (σ_J) of 0.17 Hz, and an average value of δ = 3.338 ppm with σ_J = 0.37 ppb. In the 1900 – 1980 Hz offset range (**B**) the 40 spectra yielded an average value of $^1J_{CH}$ = 144.04 Hz with a maximum deviation of 1.05 Hz, σ_J of 0.40 Hz, and $\delta_{av.}$ = 3.338 ppm with σ_J = 0.37 ppb. The following parameters were used in these experiments: spectral width in ^1H dimension = 9.98 ppm, number of complex data points in ^1H dimension = 2048, number of t_1 increments = 2 (spectra shown are processed from the first FID with final digital resolution of 0.3 Hz/point), number of scans = 16, relaxation delay = 1.7 s. Data acquisition in the pure shift experiment was divided into 16 chunks and the duration of each chunk was 25.7 ms.

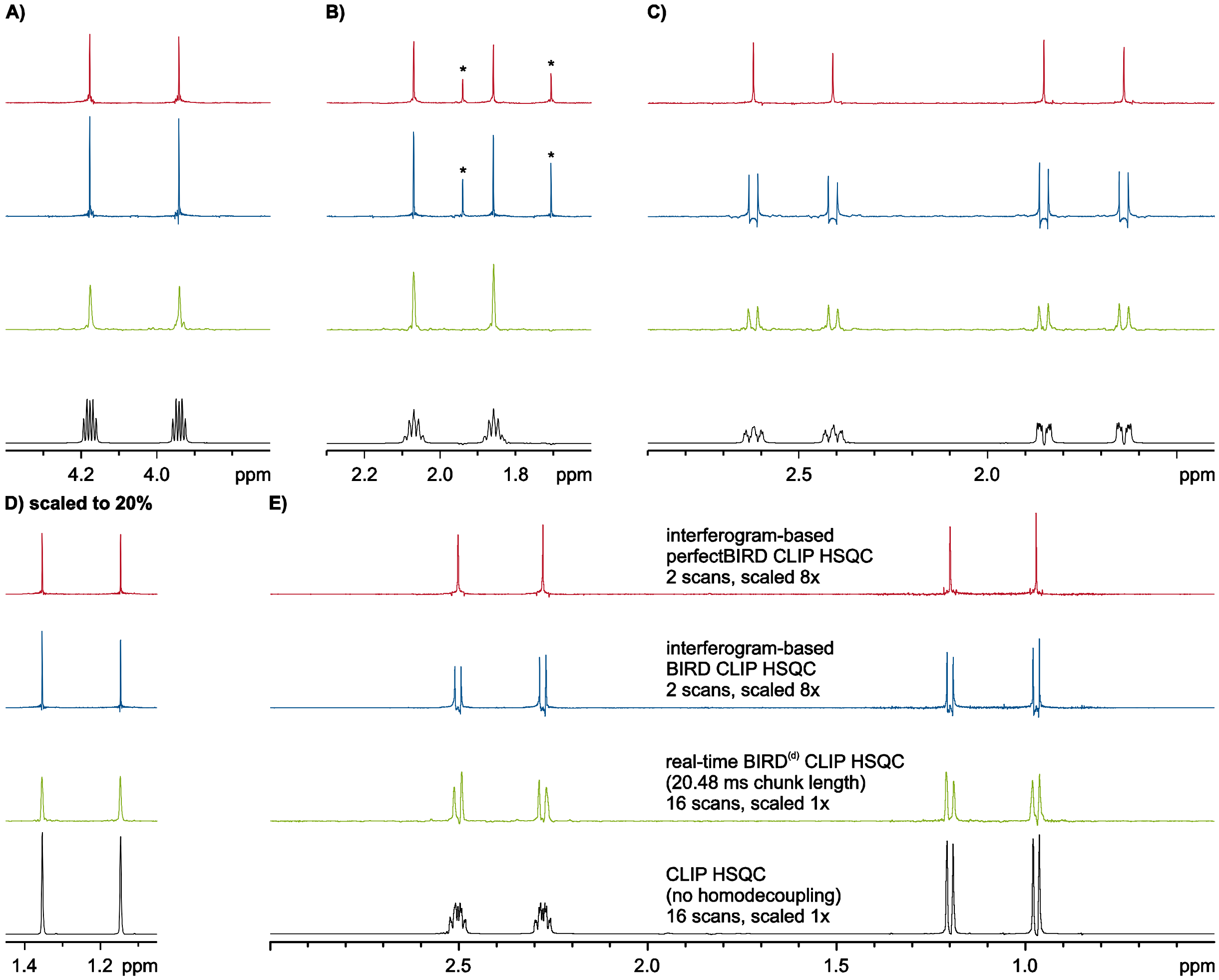

Figure S3. F_2-traces extracted from different CLIP-HSQC experiments with or without homonuclear decoupling in the proton dimension. Spectra were collected on a sample of (+)-isopinocampheol dissolved in CD_2Cl_2. Traces are shown for A) H3, B) H2, C) H4a + H4s, D) H8 and E) H7a + H7s. The numbering is shown in Figure 8 of the main text. Signals marked with an asterisk appear in the spectra obtained with interferogram-based homodecoupling because the lower F_1 resolution of these experiments led to partial overlap of the signals of C1 and C2.

Table S1. Apparent ${}^{1}J_{CH}$ and chemical shift (δ) values obtained from the CH_3-doublet of methyl α-D-mannopyranoside as a function of the phase program combinations (scan to scan phase cycles superimposed on chunk to chunk phase sequences) applied to the homodecoupling elements during real-time acquisition. The average *J* values, average chemical shifts and their standard deviations (STDEV) obtained in the offset dependence experiments described in the main text are listed for all the phase program combinations tested. The abbreviations used in the table are explained underneath the table.

Reference values measured in ^{1}H spectrum: *J* = 144.06 Hz δ = 3.339 ppm

							offset 700-780 Hz *J* (Hz)	offset 700-780 Hz STDEV (Hz)	offset 1900-1980 Hz *J* (Hz)	offset 1900-1980 Hz STDEV (Hz)	offset 700-780 Hz δ (ppm)	offset 700-780 Hz STDEV (ppb)	offset 1900-1980 Hz δ (ppm)	offset 1900-1980 Hz STDEV (ppb)
Chunks: 16	1H	0	1133	2	02	+ q-MLEV4 for every pulse	143.944	0.203	144.011	0.421	3.339	0.958	3.339	0.993
	X		1	0022										
Scans:	1H	0	1133	2	02									
	X		1	0022										
Chunks: 16	1H	0	1133	2	02	+ q-MLEV4 for every pulse	143.951	0.177	144.001	0.401	3.339	0.877	3.338	1.016
	X		1133	0022										
Scans:	1H	0	1133	2	02									
	X		1133	0022										
Chunks: 16	1H	0	1	2	0	+ MLEV4 for every pulse			143.985	0.506			3.338	0.513
	X		1	0										
Scans:	1H	0	1133	2	02									
	X		1	0022										
Chunks: 16	1H	0	1	2	0	+ MLEV8 for every pulse	143.979	0.126	143.939	0.401	3.340	0.558	3.340	0.552
	X		1	0										
Scans:	1H	0	1133	2	02									
	X		1	0022										
Chunks: 8	1H	0	1	2	0	+ MLEV8 for every pulse	143.975	0.106			3.337	0.307		
	X		1	0										
Scans:	1H	0	1133	2	02									
	X		1	0022										
Chunks: 16	1H	0	1	2	0	+ MLEV8 for every pulse	143.958	0.168	143.963	0.429	3.339	0.189	3.339	0.344
	X		1	0										
Scans:	1H	0	1133	2	02									
	X		1133	0022										
Chunks: 16	1H	0	13	2	02	+ d-MLEV8 for every pulse			144.170	0.416			3.337	0.422
	X		0	0										
Scans:	1H	0	1	2	0									
	X		0	0										
Chunks: 16	1H	0	13	2	02	+ d-MLEV8 for every pulse			144.039	0.398			3.338	0.368
	X		13	02										
Scans:	1H	0	13	2	02									
	X		13	02										
Chunks: 16	1H	0	1	2	0	+ MLEV16 for every pulse	143.974	0.136	143.945	0.462	3.338	0.366	3.338	0.464
	X		1	0										
Scans:	1H	0	1133	2	02									
	X		1	0022										

Chunks: 16	1H $(0)_{16}$ $(1)_8(3)_8$ $(2)_{16}$ $(0)_8(2)_8$ + MLEV4 for every pulse X $(1)_8(3)_8$ $(0)_8(2)_8$			144.408	0.494			3.338	0.516
Scans:	1H 0 1 2 0 X 1 0								
Chunks: 16	1H $(0)_{16}$ $(1)_8(3)_8$ $(2)_{16}$ $(0)_8(2)_8$ + MLEV8 for every pulse X $(1)_8(3)_8$ $(0)_8(2)_8$			144.253	0.456			3.338	0.429
Scans:	1H 0 1 2 0 X 1 0								
Chunks: 16	1H 0 13 2 02 + d-XY8 for every pulse X 1 0			144.233	0.333			3.337	0.316
Scans:	1H 0 1 2 0 X 1 0								
Chunks: 16	1H 0 13 2 02 + d-XY8 for every pulse X 1 0	143.964	0.172	144.033	0.373	3.338	0.297	3.338	0.427
Scans:	1H 0 1133 2 02 X 1 0022								
Chunks: 32	1H 0 13 2 02 + d-XY16 for every pulse X 1 0			144.240	0.398			3.338	0.410
Scans:	1H 0 1 2 0 X 1 0								
Chunks: 32	1H 0 13 2 02 + d-XY16 for every pulse X 1 0			144.024	0.501			3.338	0.551
Scans:	1H 0 1133 2 02 X 1 0022								
Chunks: 32	1H 0 1133 2 02 + q-XY8 for every pulse X 1 0			144.097	0.397			3.337	0.520
Scans:	1H 0 1133 2 02 X 1 0022								

In each case a particular MLEV or XY phase sequence is superimposed (as indicated by the plus sign in the table) on the basic phases of the individual pulses of the *J*-refocusing element to form a complex phase sequence from chunk to chunk. The explicit forms of the different MLEV and XY sequences used are as follows:

MLEV4 (0 0 2 2)
MLEV8 (0 0 2 2 2 0 0 2)
MLEV16 (0 0 2 2 2 0 0 2 2 2 0 0 0 2 2 0)
q-MLEV4 (quadrupoled MLEV4) (0 0 0 0 0 0 0 0 2 2 2 2 2 2 2 2)
d-MLEV8 (doubled MLEV8) (0 0 0 0 2 2 2 2 2 2 0 0 0 0 2 2)
XY8 (0 1 0 1 1 0 1 0)
XY16 (0 1 0 1 1 0 1 0 2 3 2 3 3 2 3 2)
d-XY8 (doubled XY8) (0 0 1 1 0 0 1 1 1 1 0 0 1 1 0 0)
d-XY16 (doubled XY16) (0 0 1 1 0 0 1 1 1 1 0 0 1 1 0 0 2 2 3 3 2 2 3 3 3 3 2 2 3 3 2 2)
q-XY8 (quadrupoled XY8) (0 0 0 0 1 1 1 1 0 0 0 0 1 1 1 1 1 1 1 1 0 0 0 0 1 1 1 1 0 0 0 0)

In addition, conventional scan-to-scan phase cycling was used as shown in the table.

Table S2. Optimized phase program

For chunks: d-MLEV8 (doubled MLEV8) (0 0 0 0 2 2 2 2 2 2 0 0 0 0 2 2) is superimposed on all pulses of BIRD$^{(d)}$ and the ^{1}H 180° pulse making the phase sequence from chunk to chunk (16 chunks) as follows:

BIRD$^{(d)}$ ^{1}H-180^{o}

^{1}H ϕ_4=0 ϕ_5=13 ϕ_6=2 ϕ_8=02 superimposed on d-MLEV8 for each pulse
X ϕ_5=13 ϕ_7=02

yields: $\phi_4 = (0)_4\ (2)_6\ (0)_4\ (2)_2$; $\phi_5 = (1\ 3)_2\ (3\ 1)_3\ (1\ 3)_2\ (3\ 1)$; $\phi_6 = (2)_4\ (0)_6\ (2)_4\ (0)_2$; $\phi_7 = \phi_8 = (0\ 2)_2\ (2\ 0)_3\ (0\ 2)_2\ (2\ 0)$.

For scans: 2 step phase cycle for 180^{o} pulses as follows:

^{1}H ϕ_4=0 ϕ_5=13 ϕ_6=2 ϕ_8=02
X ϕ_5=13 ϕ_7=02

transient	half chunk 1					chunk 2					chunk 3					chunk 4				
		1H 90	1H 180	1H 90	1H 180		1H 90	1H 180	1H 90	1H 180		1H 90	1H 180	1H 90	1H 180		1H 90	1H 180	1H 90	1H 180
			13C 180	13C 180				13C 180	13C 180				13C 180	13C 180				13C 180	13C 180	
1	Acq 1/2 Chunk	0	1	2	0	Acquire Chunk	0	3	2	2	Acquire Chunk	0	1	2	0	Acquire Chunk	0	3	2	2
			1	0				3	2				1	0				3	2	
2	Acq 1/2 Chunk	0	3	2	2	Acquire Chunk	0	1	2	0	Acquire Chunk	0	3	2	2	Acquire Chunk	0	1	2	0
			3	2				1	0				3	2				1	0	
3	Acq 1/2 Chunk	0	1	2	0	Acquire Chunk	0	3	2	2	Acquire Chunk	0	1	2	0	Acquire Chunk	0	3	2	2
			1	0				3	2				1	0				3	2	
4	Acq 1/2 Chunk	0	3	2	2	Acquire Chunk	0	1	2	0	Acquire Chunk	0	3	2	2	Acquire Chunk	0	1	2	0
			3	2				1	0				3	2				1	0	

chunk 5					chunk 6					chunk 7					chunk 8				
Acquire Chunk	2	3	0	2	Acquire Chunk	2	1	0	0	Acquire Chunk	2	3	0	2	Acquire Chunk	2	1	0	0
		3	2				1	0				3	2				1	0	
Acquire Chunk	2	1	0	0	Acquire Chunk	2	3	0	2	Acquire Chunk	2	1	0	0	Acquire Chunk	2	3	0	2
		1	0				3	2				1	0				3	2	
Acquire Chunk	2	3	0	2	Acquire Chunk	2	1	0	0	Acquire Chunk	2	3	0	2	Acquire Chunk	2	1	0	0
		3	2				1	0				3	2				1	0	
Acquire Chunk	2	1	0	0	Acquire Chunk	2	3	0	2	Acquire Chunk	2	1	0	0	Acquire Chunk	2	3	0	2
		1	0				3	2				1	0				3	2	

chunk 9					chunk 10					chunk 11					chunk 12				
Acquire Chunk	2	3	0	2	Acquire Chunk	2	1	0	0	Acquire Chunk	0	1	2	0	Acquire Chunk	0	3	2	2
		3	2				1	0				1	0				3	2	
Acquire Chunk	2	1	0	0	Acquire Chunk	2	3	0	2	Acquire Chunk	0	3	2	2	Acquire Chunk	0	1	2	0
		1	0				3	2				3	2				1	0	
Acquire Chunk	2	3	0	2	Acquire Chunk	2	1	0	0	Acquire Chunk	0	1	2	0	Acquire Chunk	0	3	2	2
		3	2				1	0				1	0				3	2	
Acquire Chunk	2	1	0	0	Acquire Chunk	2	3	0	2	Acquire Chunk	0	3	2	2	Acquire Chunk	0	1	2	0
		1	0				3	2				3	2				1	0	

chunk 13					chunk 14					chunk 15					chunk 16					half chunk
Acquire Chunk	0	1	2	0	Acquire Chunk	0	3	2	2	Acquire Chunk	2	3	0	2	Acquire Chunk	2	1	0	0	Acq 1/2 Chunk
		1	0				3	2				3	2				1	0		
Acquire Chunk	0	3	2	2	Acquire Chunk	0	1	2	0	Acquire Chunk	2	1	0	0	Acquire Chunk	2	3	0	2	Acq 1/2 Chunk
		3	2				1	0				1	0				3	2		
Acquire Chunk	0	1	2	0	Acquire Chunk	0	3	2	2	Acquire Chunk	2	3	0	2	Acquire Chunk	2	1	0	0	Acq 1/2 Chunk
		1	0				3	2				3	2				1	0		
Acquire Chunk	0	3	2	2	Acquire Chunk	0	1	2	0	Acquire Chunk	2	1	0	0	Acquire Chunk	2	3	0	2	Acq 1/2 Chunk
		3	2				1	0				1	0				3	2		

Table S3. Dependence of apparent $^1J_{CH}$ values on the duration of BIRD/INEPT delay (2τ, τ) measured for the methyl-doublet of methyl α-D-mannopyranoside with the ^{1}H transmitter on resonance.

Apparent $^1J_{CH}$ (Hz)	BIRD delay (2τ) set to J (Hz)
144.59	125
144.04	135
144.09	145
144.49	155
144.94	165
145.39	175

Table S4. $^1J_{NH}$ values obtained for well-resolved resonances of ^{15}N-labeled PAF protein in 95 % H_2O / 5 % D_2O. Standard deviations (stdev) were evaluated from multiple spectra acquired with each CLIP/CLAP-HSQC variant. (3 CLIP-HSQCs and 3 CLAP-HSQCs for both coupled and real-time decoupled, and 3 CLIP-HSQCs for interferogram-based decoupled.)

A	B	C	D	E	F	G		
	coupled	**stdev**	**rt-decoupl.**	**stdev**	**interf. dec.**	**stdev**	**abs(B-F)**	**abs(D-F)**
PAF	**CLIP/CLAP**		**CLIP/CLAP**		**CLIP/CLAP**			
Residue #	J (Hz)	σ_J (Hz)	J (Hz)	σ_J (Hz)	J (Hz)	σ_J (Hz)	Δ_J (Hz)	Δ_J (Hz)
4	91.34	0.51	92.60	0.09	92.64	0.04	1.30	0.03
5	93.05	0.28	93.38	0.09	93.78	0.09	0.73	0.39
6	91.74	0.30	92.36	0.04	92.58	0.03	0.83	0.22
7	92.75	0.57	93.57	0.28	94.03	0.07	1.28	0.46
8	91.65	0.51	92.56	0.08	92.68	0.06	1.03	0.11
9	92.84	0.11	92.90	0.08	92.96	0.10	0.12	0.06
12	94.59	0.60	94.07	0.33	94.41	0.04	0.18	0.34
13	90.81	0.62	92.05	0.08	92.42	0.04	1.61	0.38
14	91.66	0.41	92.46	0.06	92.56	0.11	0.90	0.10
15	91.76	0.39	92.79	0.08	92.76	0.05	1.01	0.02
16	93.97	0.32	94.57	0.08	94.62	0.04	0.65	0.05

17	91.86	0.27	92.38	0.09	92.59	0.13	0.73	0.21
18	94.03	0.26	94.54	0.09	94.34	0.06	0.31	0.21
19	92.38	0.20	92.36	0.35	92.25	0.13	0.13	0.10
20	91.61	0.30	92.52	0.13	92.21	0.03	0.60	0.31
21	94.19	0.18	94.27	0.17	94.15	0.14	0.04	0.12
22	92.41	0.37	93.55	0.07	93.44	0.04	1.03	0.11
23	93.21	0.06	93.37	0.08	93.29	0.08	0.08	0.08
25	91.59	0.46	92.78	0.10	92.75	0.04	1.16	0.03
26	93.04	0.27	94.11	0.06	93.75	0.03	0.71	0.36
27	94.04	0.07	94.02	0.13	94.16	0.08	0.12	0.14
28	91.62	0.19	91.77	0.11	91.96	0.05	0.34	0.19
30	90.84	0.17	91.08	0.11	91.16	0.02	0.32	0.08
31	92.15	0.09	92.22	0.07	92.36	0.02	0.21	0.15
33	93.20	0.34	93.29	0.25	93.19	0.17	0.01	0.11
34	89.78	0.62	90.74	0.09	91.02	0.05	1.24	0.29
35	92.24	0.55	92.68	0.11	92.60	0.10	0.36	0.09
36	92.80	0.13	92.70	0.12	92.87	0.06	0.07	0.17
37	91.54	0.26	91.44	0.19	91.72	0.11	0.18	0.28
38	92.80	0.11	92.99	0.10	93.30	0.04	0.50	0.32
39	94.34	0.08	94.47	0.11	94.38	0.03	0.04	0.09
40	93.99	0.05	93.83	0.13	93.56	0.02	0.43	0.28
41	94.66	0.23	94.77	0.07	94.75	0.03	0.09	0.02
42	92.97	0.08	93.18	0.06	92.99	0.09	0.02	0.19
43	92.71	0.20	93.20	0.05	93.28	0.05	0.57	0.08
44	92.00	0.53	93.26	0.17	93.24	0.10	1.24	0.03
45	91.21	0.50	92.37	0.12	92.37	0.07	1.16	0.00
46	92.29	0.54	93.36	0.05	93.38	0.09	1.09	0.03
47	94.07	0.12	94.27	0.10	94.25	0.09	0.18	0.02
48	92.97	0.17	93.13	0.10	93.32	0.03	0.35	0.19
49	90.54	0.39	91.33	0.08	91.27	0.02	0.73	0.06
50	94.59	0.17	93.79	0.03	93.86	0.05	0.74	0.07
51	92.84	0.25	93.36	0.05	93.38	0.05	0.55	0.02
52	91.09	0.62	92.44	0.04	92.54	0.07	1.45	0.10
53	92.33	0.52	93.11	0.13	93.38	0.01	1.06	0.27
54	92.63	0.40	93.24	0.11	93.29	0.00	0.66	0.05
55	92.61	0.31	93.26	0.12	93.32	0.04	0.71	0.05
					red:		> 1.0	> 0.35

Table S5. Effect of J_{NH} variation, over the range 66 - 116 Hz in 5 Hz steps, for the real-time BIRD(d) CLIP/CLAP-HSQC on well resolved resonances of ^{15}N-labeled PAF protein in 95% H_2O / 5% D_2O. Values are reported in Hz.

Residue #	J = 66 Hz CLIP/CLAP	J = 71 Hz CLIP/CLAP	J = 76 Hz CLIP/CLAP	J = 81 Hz CLIP/CLAP	J = 86 Hz CLIP/CLAP	J = 91 Hz CLIP/CLAP	J = 96 Hz CLIP/CLAP	J = 101 Hz CLIP/CLAP	J = 106 Hz CLIP/CLAP	J = 111 Hz CLIP/CLAP	J = 116 Hz CLIP/CLAP	average *J*	STDEV	***reference J****	(ref.-aver.) abs.dev.
4	94.07	93.37	93.05	92.82	92.56	92.59	92.58	92.61	92.64	93.04	93.24	92.96	0.47	92.60	0.36
5	92.88	92.97	93.18	93.06	93.34	93.57	93.72	93.78	93.94	94.01	94.00	93.50	0.43	93.38	0.11
6	92.97	92.96	92.92	92.63	92.48	92.42	92.45	92.37	92.28	92.31	92.29	92.55	0.27	92.36	0.19
7	90.80	93.37	94.37	94.44	94.30	94.05	93.95	93.69	93.58	93.45	93.38	93.58	1.00	93.57	0.01
8	93.09	93.20	93.02	92.94	92.88	92.65	92.66	92.54	92.46	92.47	92.39	92.76	0.28	92.56	0.19
9	90.16	91.09	91.89	91.95	92.43	92.87	92.94	92.99	93.01	92.94	92.76	92.28	0.93	92.90	0.62
12	95.73	94.35	94.18	94.11	94.12	94.21	94.28	94.30	94.24	94.24	94.19	94.36	0.46	94.07	0.29
13	92.05	92.22	92.06	91.98	92.02	92.11	92.06	91.98	91.99	92.04	92.03	92.05	0.07	92.05	0.00
14	90.80	91.66	91.83	91.87	92.10	92.39	92.62	92.89	93.05	93.15	93.13	92.32	0.75	92.46	0.14
15	92.80	93.22	92.86	92.93	92.75	92.69	92.63	92.62	92.70	92.60	92.55	92.76	0.19	92.79	0.03
16	93.91	94.05	94.25	94.23	94.31	94.53	94.70	94.82	94.96	95.00	95.02	94.53	0.40	94.57	0.05
17	95.57	93.66	93.11	92.82	92.57	92.54	92.49	92.44	92.38	92.52	92.58	92.97	0.94	92.38	0.59
18	94.35	94.27	94.55	94.41	94.44	94.45	94.60	94.63	94.66	94.62	94.62	94.51	0.13	94.54	0.03
19	91.61	91.03	92.23	91.78	92.36	92.12	91.98	92.46	91.94	92.02	92.77	92.03	0.46	92.36	0.33
20	89.57	90.49	91.26	91.75	92.13	92.29	92.40	92.35	92.12	91.92	91.58	91.62	0.89	92.52	0.90
21	91.04	91.52	92.94	93.32	93.78	94.10	94.20	94.16	94.05	93.88	93.60	93.32	1.09	94.27	0.95
22	96.20	95.15	94.38	93.80	93.57	93.58	93.69	93.78	93.91	94.20	94.37	94.24	0.80	93.55	0.69
23	93.26	92.82	93.08	92.84	93.13	93.22	93.48	93.47	93.59	93.73	93.62	93.29	0.31	93.37	0.08
25	90.53	91.14	92.05	92.15	92.45	92.58	92.86	92.79	92.66	92.52	92.30	92.18	0.73	92.78	0.60
26	96.29	96.17	95.49	94.73	94.36	94.18	94.25	94.26	94.48	94.67	95.06	94.90	0.76	94.11	0.79
27	89.52	91.39	93.48	92.91	93.48	93.92	94.03	94.01	93.94	93.85	93.59	93.10	1.41	94.02	0.92
28	91.31	91.51	91.62	91.55	91.65	91.77	91.96	92.18	92.24	92.30	92.31	91.86	0.36	91.77	0.08
30	88.69	89.82	90.57	90.76	90.90	91.01	90.99	90.89	90.75	90.67	90.47	90.50	0.69	91.08	0.58
31	92.77	92.54	92.36	92.30	92.21	92.41	92.46	92.53	92.62	92.76	92.79	92.52	0.20	92.22	0.31
33	91.15	91.43	92.61	92.72	93.07	93.01	93.47	93.38	93.21	93.13	93.24	92.76	0.77	93.29	0.53
34	90.96	90.92	90.91	90.70	90.77	90.80	90.95	90.99	91.06	91.11	91.22	90.94	0.15	90.74	0.21
35	96.06	94.62	93.72	93.05	92.82	92.78	92.85	93.07	93.26	93.59	93.91	93.61	0.99	92.68	0.93
36	92.72	92.66	92.83	92.64	92.76	92.80	93.01	93.09	93.11	93.18	93.20	92.91	0.21	92.70	0.21
37	90.06	90.00	90.58	90.77	90.95	91.31	91.56	91.46	91.01	90.88	90.46	90.82	0.52	91.44	0.62
38	89.22	90.50	91.74	92.14	92.62	92.81	92.98	93.03	92.84	92.71	92.35	92.09	1.20	92.99	0.90
39	95.88	95.66	95.29	94.87	94.64	94.55	94.45	94.26	94.29	94.20	94.35	94.77	0.59	94.47	0.30
40	97.13	96.20	95.28	94.68	94.16	94.20	93.93	93.95	94.16	94.40	94.81	94.81	1.02	93.83	0.98
41	94.40	94.64	94.78	94.72	94.70	94.85	94.92	94.94	94.93	94.97	94.93	94.80	0.17	94.77	0.02
42	97.28	95.51	94.54	93.79	93.41	93.29	93.24	93.35	93.45	93.72	94.15	94.16	1.24	93.18	0.97
43	95.93	95.19	94.42	93.66	93.45	93.50	93.40	93.56	93.67	94.03	94.26	94.10	0.81	93.20	0.89
44	94.11	93.82	93.78	93.61	93.50	93.54	93.34	93.21	93.26	93.23	93.19	93.51	0.30	93.26	0.24
45	95.47	94.47	93.07	92.51	92.39	92.48	92.63	92.73	92.91	93.04	93.16	93.17	0.95	92.37	0.80
46	94.87	94.13	94.08	93.72	93.54	93.51	93.39	93.32	93.35	93.51	93.74	93.74	0.46	93.36	0.38
47	94.33	94.53	94.55	94.49	94.39	94.31	94.21	94.14	93.97	93.96	93.81	94.25	0.25	94.27	0.03
48	92.20	92.33	92.94	92.90	92.98	93.11	93.30	93.56	93.68	93.76	93.74	93.14	0.54	93.13	0.01
49	91.40	91.34	91.48	91.38	91.37	91.45	91.33	91.37	91.31	91.37	91.35	91.38	0.05	91.33	0.05
50	93.90	94.01	94.04	93.98	93.90	93.91	93.99	94.02	94.01	94.07	94.07	93.99	0.06	93.79	0.20
51	97.05	95.89	94.54	93.63	93.45	93.40	93.56	93.72	93.92	94.26	94.44	94.35	1.15	93.36	0.99
52	92.62	92.62	92.51	92.39	92.38	92.49	92.50	92.49	92.64	92.66	92.63	92.54	0.10	92.44	0.10
53	91.11	92.34	93.00	93.48	93.33	93.28	93.39	93.15	93.13	93.06	94.78	93.09	0.88	93.11	0.02
54	91.16	91.24	91.87	92.35	92.89	93.13	93.08	92.96	92.89	92.59	92.24	92.40	0.71	93.24	0.84
55	95.76	94.92	94.25	93.41	93.31	93.30	93.33	93.51	93.68	93.97	94.19	93.97	0.78	93.26	0.70
												red:	> 1		> 0.9

* Reference *J* is taken as mean of six measurements (3 rt-CLIP and 3 rt-CLAP) with BIRD/INEPT delays corresponding to 91 Hz.

Table S6. RDC-values ($^1D_{\mathrm{NH}}$) obtained with real-time BIRD$^{(d)}$ CLIP-HSQCs for well resolved resonances of ^{15}N-labeled PAF protein in the weakly orienting medium 5% C_8E_5 and octanol in H_2O/D_2O.

PAF residue	anisotropic	RMSD	isotropic	RMSD	RDC*
	$^1T_{NH}$ (Hz)	(Hz)	$^1J_{NH}$ (Hz)	(Hz)	$^1D_{NH}$ (Hz)
1					
2	85.80	0.35	92.04	1.07	-3.12
3					
4	74.69	0.10	92.60	0.09	-8.96
5	78.66	0.62	93.38	0.09	-7.37
6	84.94	0.02	92.36	0.04	-3.71
7	89.07	0.77	93.57	0.28	-2.25
8	106.08	1.36	92.56	0.08	6.76
9	107.66	1.07	92.90	0.08	7.38
10	100.58	1.38	94.39	1.36	3.10
11					
12	106.84	4.69	94.07	0.33	6.39
13	109.70	2.39	92.05	0.08	8.83
14	106.96	0.10	92.46	0.06	7.25
15	91.60	0.73	92.79	0.08	-0.60
16	96.87	3.43	94.57	0.08	1.15
17	88.99	0.47	92.38	0.09	-1.70
18	101.69	1.02	94.54	0.09	3.58
19	99.95	0.65	92.36	0.35	3.80
20	80.84	1.68	92.52	0.13	-5.84
21	99.22	3.97	94.27	0.17	2.48
22					
23	94.99	0.50	93.37	0.08	0.81
24					
25	89.17	1.02	92.78	0.10	-1.81
26	100.22	0.42	94.11	0.06	3.06
27	100.01	0.12	94.02	0.13	3.00
28	109.89	1.03	91.77	0.11	9.06
29					
30	102.46	1.02	91.08	0.11	5.69
31	88.35	0.43	92.22	0.07	-1.94
32	87.67	0.90	92.78	0.79	-2.56
33	104.59	1.40	93.29	0.25	5.65
34	90.06	2.02	90.74	0.09	-0.34
35	76.90	1.51	92.68	0.11	-7.90
36	94.11	1.00	92.70	0.12	0.71
37	94.55	2.90	91.44	0.19	1.55
38	92.89	0.75	92.99	0.10	-0.05
39	95.72	0.37	94.47	0.11	0.63
40	99.15	0.54	93.83	0.13	2.66
41	91.17	0.34	94.77	0.07	-1.80

42	84.21	0.82	93.18	0.06	-4.49
43	76.01	0.65	93.20	0.05	-8.60
44	76.32	0.27	93.26	0.17	-8.47
45	82.99	0.03	92.37	0.12	-4.69
46	78.68	1.25	93.36	0.05	-7.34
47	88.42	0.36	94.27	0.10	-2.93
48	93.86	1.34	93.13	0.10	0.37
49	99.40	0.01	91.33	0.08	4.04
50	81.44	1.93	93.79	0.03	-6.18
51	87.44	0.53	93.36	0.05	-2.96
52	84.83	0.41	92.44	0.04	-3.80
53	77.62	0.13	93.11	0.13	-7.75
54	83.43	0.21	93.24	0.11	-4.91
55	81.88	0.10	93.26	0.12	-5.69

[*] RDC is defined: $^{1}D_{NH} = (^{1}T_{NH} - {}^{1}J_{NH})/2$

Table S7. Scalar one-bond couplings ($^1J_{CH}$) measured for (+)-isopinocampheol in CD_2Cl_2. For comparison, the values reported previously are reproduced here. Standard deviations (σ_J) and maximum deviations (Δ_{max}) reported are calculated with respect to the interferogram perfectBIRD decoupled CLIP-HSQC experiment.

		Values reproduced from ref.[1], ‡			Values for this work (same sample, measurement 20 months later)				
		CLIP	BIRD CLIP	perfectBIRD CLIP	CLIP	rt-BIRD CLIP	rt-BIRD CLIP	rt-BIRD CLIP	rt-BIRD CLIP
NS		2	2	2	16	16	16	16	8
FID-res		0.76 Hz	1.02 Hz	1.02 Hz	1.53 Hz	0.76 Hz	1.53 Hz	1.53 Hz	0.76 Hz
chunk duration		-	15.36 ms	15.36 ms	-	20.48 ms	15.36 ms	10.24 ms	20.48 ms
exp. time		10.5min	7h 8min	9h 26min	3h 20min	3h 20min	3h 20min	3h 20min	1h 40min
C	H		$^1J_{CH}$ / Hz				$^1J_{CH}$ / Hz		
1	1	140.57 ± 0.32	140.56 ± 0.11	140.59 ± 0.11	140.63 ± 0.25	140.47 ± 1.00	141.59 ± 1.50	141.44 ± 0.20	140.38 ± 1.50
2	2	126.67 ± 0.17	126.65 ± 0.11	126.59 ± 0.12	126.72 ± 0.20	126.86 ± 0.30	126.72 ± 0.10	126.13 ± 0.60	126.88 ± 0.40
3	3	141.67 ± 0.11	141.69 ± 0.11	141.71 ± 0.12	141.66 ± 0.10	141.73 ± 0.50	141.87 ± 0.80	142.32 ± 0.80	141.75 ± 1.00
4	4s	126.35 ± 0.18	126.35 ± 0.24	126.34 ± 0.13	126.44 ± 0.25	127.25 ± 0.80	126.72 ± 1.00	127.92 ± 2.00	127.16 ± 0.90
4	4a	127.04 ± 0.12	127.02 ± 0.19	127.07 ± 0.13	126.98 ± 0.20	127.60 ± 0.90	127.11 ± 0.90	128.06 ± 1.40	127.68 ± 0.60
5	5	140.79 ± 0.32	140.72 ± 0.14	140.64 ± 0.23	140.78 ± 0.50	140.11 ± 1.00	139.94 ± 1.60	139.98 ± 3.00	140.05 ± 1.00
7	7s	134.87 ± 0.35	134.81 ± 0.13	134.79 ± 0.17	134.96 ± 0.40	134.79 ± 1.30	135.12 ± 1.00	135.69 ± 0.60	134.79 ± 1.00
7	7a	136.98 ± 0.15	136.94 ± 0.20	136.94 ± 0.12	137.02 ± 0.20	136.43 ± 1.60	136.46 ± 1.40	136.62 ± 0.70	136.51 ± 0.80
8	8	124.56 ± 0.16	124.51 ± 0.12	124.51 ± 0.12	124.56 ± 0.15	124.49 ± 0.30	124.69 ± 0.20	125.03 ± 0.90	124.54 ± 0.20
9	9	123.61 ± 0.11	123.66 ± 0.11	123.70 ± 0.12	123.60 ± 0.10	123.60 ± 0.60	123.80 ± 0.80	123.23 ± 0.50	123.52 ± 0.40
10	10	124.75 ± 0.13	124.69 ± 0.11	124.64 ± 0.15	124.71 ± 0.15	124.58 ± 0.60	125.26 ± 0.50	124.36 ± 0.30	124.53 ± 0.30
δ; Δ_{max} / Hz		0.08; 0.15	0.05; 0.08	reference	0.11; 0.17	0.40; 0.91	0.48; 1.00	0.79; 1.58	0.40; 0.82
		CLAP	BIRD CLAP	perfectBIRD CLAP	CLAP	rt-BIRD CLAP	rt-BIRD CLAP	rt-BIRD CLAP	rt-BIRD CLAP
NS		2	2	2	16	16	16	16	8
FID-res		0.76 Hz	1.02 Hz	1.02 Hz	1.53 Hz	0.76 Hz	1.53 Hz	1.53 Hz	0.76 Hz
chunk duration		-	15.36 ms	15.36 ms	-	20.48 ms	15.36 ms	10.24 ms	20.48 ms
exp. time		10.5min	7h 8min	9h 26min	3h 20min	3h 20min	3h 20min	3h 20min	1h 40min
C	H		$^1J_{CH}$ / Hz				$^1J_{CH}$ / Hz		
1	1	140.54 ± 0.26	140.58 ± 0.15	140.56 ± 0.11	140.49 ± 0.20	140.63 ± 0.70		141.34 ± 0.20	
2	2	126.71 ± 0.16	126.63 ± 0.12	126.56 ± 0.14	126.79 ± 0.20	126.94 ± 0.25		126.03 ± 0.60	
3	3	141.67 ± 0.12	141.71 ± 0.12	141.75 ± 0.13	141.63 ± 0.15	142.11 ± 0.70		141.78 ± 0.70	
4	4s	126.37 ± 0.15	126.32 ± 0.22	126.31 ± 0.18	126.39 ± 0.30	127.19 ± 0.60		127.71 ± 2.00	
4	4a	127.06 ± 0.12	127.06 ± 0.19	127.08 ± 0.16	126.95 ± 0.25	127.58 ± 0.80		128.14 ± 1.20	
5	5	140.73 ± 0.29	140.71 ± 0.24	140.60 ± 0.20	140.81 ± 0.40	140.15 ± 1.00		139.19 ± 2.00	
7	7s	134.85 ± 0.31	134.84 ± 0.11	134.78 ± 0.13	134.91 ± 0.50	134.80 ± 0.80		135.63 ± 0.70	
7	7a	136.99 ± 0.12	137.01 ± 0.22	136.93 ± 0.12	136.99 ± 0.15	136.46 ± 0.70		136.46 ± 0.80	
8	8	124.60 ± 0.14	124.48 ± 0.12	124.47 ± 0.11	124.56 ± 0.10	124.58 ± 0.20		124.64 ± 0.60	
9	9	123.63 ± 0.10	123.69 ± 0.11	123.69 ± 0.11	123.60 ± 0.10	123.61 ± 0.30		123.32 ± 0.40	
10	10	124.74 ± 0.13	124.67 ± 0.11	124.59 ± 0.15	124.75 ± 0.10	124.78 ± 0.40		124.13 ± 0.50	
δ; Δ_{max} / Hz		0.08; 0.12	0.04; 0.07	0.04; 0.05	0.12; 0.20	0.41; 0.85		0.82; 1.45	

‡: *Chem.Commun.*, 2014, **50**, 15702 – Published by The Royal Society of Chemistry

Table S8. One-bond couplings (${}^{1}T_{CH}$) measured for (+)-isopinocampheol weakly aligned in CD_2Cl_2/PBDG (9.05%-wt, $\Delta\nu_Q$ = 110.5 Hz). Values previously reported are scaled by the ratio of the quadrupolar splittings ($T_{i,scaled} = J_i + (T_{i,original} - J_i)*(\Delta\nu_{Q,\ scaled}/\ \Delta\nu_{Q,\ original})$). Standard deviations ($\sigma_J$) and maximum deviations (Δ_{max}) reported are calculated with respect to the interferogram perfectBIRD decoupled CLIP-HSQC experiment.

		Values reproduced from ref.[1] (scaled for comparison), ‡			Values for this work (same sample, measurement 20 months later)				
		CLIP	BIRD CLIP	perfectBIRD CLIP	CLIP	rt-BIRD CLIP	rt-BIRD CLIP	rt-BIRD CLIP	rt-BIRD CLIP
NS		2	2	2	16	16	16	16	8
FID-res		0.76 Hz	3.05 Hz	3.05 Hz	1.53 Hz	1.53 Hz	1.53 Hz	1.53 Hz	0.76 Hz
chunk duration		-	10.24 ms	10.24 ms	-	20.48 ms	15.36 ms	10.24 ms	20.48 ms
exp. time		10.5min	2h 49min	3h 13min	3h 20min	3h 20min	3h 20min	3h 20min	1h 40min
C	H		${}^{1}T_{CH}$ / Hz				${}^{1}T_{CH}$ / Hz		
1	1	161.21 ± 3.51	161.47 ± 0.87	160.72 ± 0.56	162.36 ± 2.80	161.57 ± 0.90	161.85 ± 3.00	162.59 ± 1.20	161.46 ± 0.90
2	2	117.02 ± 1.65	116.74 ± 0.19	116.26 ± 0.48	116.40 ± 1.40	116.37 ± 1.40	116.24 ± 1.50	116.53 ± 2.00	116.33 ± 1.30
3	3	158.76 ± 0.53	158.83 ± 0.35	158.96 ± 0.45	157.83 ± 0.60	157.68 ± 0.80	156.43 ± 0.80	158.07 ± 0.90	157.70 ± 0.80
4	4s	144.51 ± 0.12	144.32 ± 0.39	144.30 ± 0.20	144.65 ± 0.40	144.26 ± 0.80	145.46 ± 2.50	146.03 ± 3.00	144.10 ± 0.80
4	4a	122.42 ± 0.17	122.47 ± 0.46	122.44 ± 0.12	121.70 ± 0.60	121.91 ± 1.00	122.13 ± 2.60	121.61 ± 2.30	122.15 ± 1.00
5	5	138.03 ± 1.00	137.31 ± 0.85	137.03 ± 1.03	139.03 ± 1.40	138.59 ± 0.30	138.94 ± 0.60	138.83 ± 1.20	138.54 ± 0.40
7	7s	133.75 ± 1.82	134.01 ± 2.94	133.76 ± 0.33	133.06 ± 1.90	*133.46 ± 6.00*	*134.04 ± 15.00*	*132.21 ± 7.00*	*133.19 ± 7.00*
7	7a	106.08 ± 1.07	106.17 ± 0.89	106.00 ± 0.80	106.06 ± 0.50	*105.15 ± 5.00*	*104.27 ± 15.00*	*106.31 ± 14.00*	*105.32 ± 5.00*
8	8	131.89 ± 0.49	132.02 ± 0.61	131.85 ± 0.30	132.00 ± 0.80	132.12 ± 1.40	131.50 ± 1.20	132.88 ± 2.80	132.40 ± 1.20
9	9	118.43 ± 0.19	118.58 ± 0.58	118.83 ± 0.48	118.76 ± 0.40	118.89 ± 1.50	118.88 ± 1.80	118.58 ± 0.90	118.88 ± 1.00
10	10	121.19 ± 0.21	120.99 ± 0.62	120.77 ± 0.80	121.16 ± 0.30	120.85 ± 0.80	121.18 ± 1.40	121.08 ± 1.80	120.90 ± 1.00
δ; Δ_{max} / Hz		0.46; 1.01	0.32;0.76	reference	0.93; 2.01	0.74; 1.57	1.22; 2.54	1.17; 1.88	0.72; 1.52
		CLAP	BIRD CLAP	perfectBIRD CLAP	CLAP	rt-BIRD CLAP	rt-BIRD CLAP	rt-BIRD CLAP	rt-BIRD CLAP
NS		2	2	2	16	16	16	16	8
FID-res		0.76 Hz	3.05 Hz	3.05 Hz	1.53 Hz	1.53 Hz	1.53 Hz	1.53 Hz	1.53 Hz
chunk duration		-	10.24 ms	10.24 ms	-	20.48 ms	15.36 ms	10.24 ms	20.48 ms
exp. time		10.5min	2h 49min	3h 13min	3h 20min	3h 20min	3h 20min	3h 20min	1h 40min
C	H		${}^{1}T_{CH}$ / Hz				${}^{1}T_{CH}$ / Hz		
1	1	163.26 ± 1.75	161.21 ± 1.26	161.01 ± 0.46	164.11 ± 2.60	161.06 ± 1.20		162.56 ± 1.00	
2	2	117.00 ± 2.02	116.66 ± 0.24	116.44 ± 0.45	116.41 ± 2.00	116.36 ± 1.20		116.31 ± 1.70	
3	3	158.75 ± 1.12	158.85 ± 0.60	159.01 ± 0.47	155.97 ± 1.20	157.01 ± 1.00		157.39 ± 1.40	
4	4s	144.53 ± 0.13	144.35 ± 0.54	144.34 ± 0.16	144.64 ± 0.50	144.39 ± 0.60		145.64 ± 1.20	
4	4a	122.36 ± 0.41	122.48 ± 0.43	122.48 ± 0.18	121.67 ± 0.50	121.88 ± 0.80		121.32 ± 2.20	
5	5	138.29 ± 1.22	137.35 ± 0.59	137.06 ± 0.67	139.01 ± 1.20	138.60 ± 0.30		138.86 ± 1.10	
7	7s	134.33 ± 3.75	133.97 ± 3.17	133.77 ± 0.26	133.49 ± 2.40	*134.55 ± 7.00*		*132.41 ± 8.00*	
7	7a	106.15 ± 0.31	106.13 ± 2.93	105.97 ± 0.23	106.29 ± 0.40	*106.23 ± 7.00*		*113.94 ± 11.00*	
8	8	132.19 ± 0.38	132.10 ± 0.60	131.95 ± 0.17	131.91 ± 0.90	132.00 ± 1.00		132.55 ± 1.90	
9	9	118.24 ± 0.25	118.66 ± 0.35	118.82 ± 0.34	118.89 ± 0.20	119.28 ± 1.40		117.94 ± 0.80	
10	10	121.25 ± 0.32	121.00 ± 0.14	120.70 ± 0.34	121.36 ± 0.40	121.68 ± 1.00		120.11 ± 2.00	
δ; Δ_{max} / Hz		0.94; 2.54	0.26; 0.50	0.12; 0.29	1.53; 3.40	0.88; 1.96		2.68; 7.95	

‡: *Chem.Commun.*, 2014, **50**, 15702 – Published by The Royal Society of Chemistry

Table S9. One-bond RDCs ($^{1}D_{CH}$) measured for (+)-isopinocampheol weakly aligned in CD_2Cl_2/PBDG (9.05%-wt, $\Delta\nu_Q = 110.5$ Hz). Values previously reported are scaled by the ratio of the quadrupolar splittings ($D_{i,scaled} = D_{i,original}*(\Delta\nu_{Q,\ scaled}/\ \Delta\nu_{Q,\ original})$). Standard deviations ($\sigma_J$) and maximum deviations (Δ_{max}) reported are calculated with respect to the interferogram perfectBIRD decoupled CLIP-HSQC experiment.

		Values reproduced from ref.[1] (scaled for comparison), ‡			Values reported in this work (same sample, measurement 20 months later)				
		CLIP	BIRD CLIP	perfectBIRD CLIP	CLIP	rt-BIRD CLIP	rt-BIRD CLIP	rt-BIRD CLIP	rt-BIRD CLIP
NS		2	2	2	16	16	16	16	8
chunk duration		-	differs for $^{1}J_{CH}$ and $^{1}T_{CH}$ measurement		-	20.48 ms	15.36 ms	10.24 ms	20.48 ms
C	H		$^{1}D_{CH}$ / Hz				$^{1}D_{CH}$ / Hz		
1	1	10.32 ± 3.52	10.45 ± 0.87	10.06 ± 0.56	10.87 ± 2.80	10.55 ± 1.00	10.13 ± 3.00	10.58 ± 1.20	10.54 ± 1.50
2	2	-4.83 ± 1.65	-4.96 ± 0.22	-5.17 ± 0.49	-5.16 ± 1.40	-5.25 ± 1.40	-5.24 ± 1.50	-4.80 ± 2.00	-5.28 ± 1.30
3	3	8.54 ± 0.53	8.57 ± 0.36	8.63 ± 0.46	8.09 ± 0.60	7.98 ± 0.80	7.28 ± 0.80	7.88 ± 0.90	7.97 ± 1.00
4	4s	9.08 ± 0.22	8.99 ± 0.45	8.98 ± 0.24	9.11 ± 0.40	8.51 ± 0.80	9.37 ± 2.50	9.06 ± 3.00	8.47 ± 0.90
4	4a	-2.31 ± 0.21	-2.27 ± 0.49	-2.32 ± 0.17	-2.64 ± 0.60	-2.85 ± 1.00	-2.49 ± 2.60	-3.23 ± 2.30	-2.77 ± 1.00
5	5	-1.38 ± 1.05	-1.70 ± 0.85	-1.81 ± 1.05	-0.88 ± 1.40	-0.76 ± 1.00	-0.50 ± 1.60	-0.57 ± 3.00	-0.76 ± 1.00
7	7s	-0.56 ± 1.85	-0.40 ± 2.94	-0.51 ± 0.37	-0.95 ± 1.90	{ *-0.66 ± 6.00*	*-0.54 ± 15.00*	*-1.74 ± 7.00*	*-0.80 ± 7.00* }
7	7a	-15.45 ± 1.08	-15.38 ± 0.90	-15.47 ± 0.80	-15.48 ± 0.50	{ *-15.64 ± 5.00*	*-16.10 ± 15.00*	*-15.16 ± 14.00*	*-15.60 ± 5.00* }
8	8	3.67 ± 0.51	3.75 ± 0.62	3.67 ± 0.32	3.72 ± 0.80	3.82 ± 1.40	3.41 ± 1.20	3.93 ± 2.80	3.93 ± 1.20
9	9	-2.59 ± 0.22	-2.54 ± 0.59	-2.43 ± 0.49	-2.42 ± 0.40	-2.36 ± 1.50	-2.46 ± 1.80	-2.33 ± 0.90	-2.32 ± 1.00
10	10	-1.78 ± 0.25	-1.85 ± 0.63	-1.94 ± 0.81	-1.78 ± 0.30	-1.87 ± 0.80	-2.04 ± 1.40	-1.64 ± 1.80	-1.82 ± 1.00
δ; Δ_{max} / Hz		0.20; 0.43	0.16; 0.40	reference	0.45; 0.94	0.47; 1.05	0.62; 1.35	0.68; 1.24	0.47; 1.06

		CLAP	BIRD CLAP	perfectBIRD CLAP	CLAP	rt-BIRD CLAP	rt-BIRD CLAP	rt-BIRD CLAP	rt-BIRD CLAP
NS		2	2	2	16	16	16	16	8
chunk duration		-			-	20.48 ms	15.36 ms	10.24 ms	20.48 ms
			differs for $^{1}J_{CH}$ and $^{1}T_{CH}$ measurement						
C	H		$^{1}D_{CH}$ / Hz				$^{1}D_{CH}$ / Hz		
1	1	11.36 ± 1.77	10.32 ± 1.26	10.22 ± 0.47	11.81 ± 2.60	10.22 ± 1.20		10.61 ± 1.00	
2	2	-4.86 ± 2.02	-4.99 ± 0.27	-5.06 ± 0.46	-5.19 ± 2.00	-5.29 ± 1.20		-4.86 ± 1.70	
3	3	8.54 ± 1.12	8.57 ± 0.61	8.63 ± 0.48	7.17 ± 1.20	7.45 ± 1.00		7.80 ± 1.40	
4	4s	9.08 ± 0.20	9.02 ± 0.58	9.02 ± 0.24	9.12 ± 0.50	8.60 ± 0.60		8.97 ± 2.00	
4	4a	-2.35 ± 0.42	-2.29 ± 0.46	-2.30 ± 0.24	-2.64 ± 0.50	-2.85 ± 0.80		-3.41 ± 2.20	
5	5	-1.22 ± 1.25	-1.68 ± 0.64	-1.77 ± 0.70	-0.90 ± 1.20	-0.78 ± 1.00		-0.16 ± 2.00	
7	7s	-0.26 ± 3.76	-0.44 ± 3.16	-0.50 ± 0.29	-0.71 ± 2.40	{ *-0.13 ± 7.00* }		{ *-1.61 ± 8.00* }	
7	7a	-15.42 ± 0.33	-15.44 ± 2.94	-15.48 ± 0.26	-15.35 ± 0.40	{ *-15.12 ± 7.00* }		{ *-11.26 ± 11.00* }	
8	8	3.79 ± 0.40	3.81 ± 0.61	3.74 ± 0.20	3.68 ± 0.90	3.71 ± 1.00		3.96 ± 1.90	
9	9	-2.70 ± 0.27	-2.52 ± 0.36	-2.43 ± 0.35	-2.36 ± 0.20	-2.17 ± 1.40		-2.69 ± 0.80	
10	10	-1.75 ± 0.34	-1.83 ± 0.17	-1.95 ± 0.37	-1.70 ± 0.40	-1.55 ± 1.00		-2.01 ± 2.00	
δ; Δ_{max} / Hz		0.46; 1.30	0.13; 0.26	0.07; 0.16	0.76; 1.75	0.56; 1.18		1.48; 4.22	

‡: *Chem.Commun.*, 2014, **50**, 15702 – Published by The Royal Society of Chemistry

Table S10. Quality factors obtained from an error-weighted fitting of the RDCs reported in Table S9 (including values reported in brackets) to the DFT-derived structural model previously used.[1, 3-5]

	Values reproduced from ref.[1], ‡						Values for this work (same sample, measurement 20 months later)							
	CLIP	CLAP	BIRD CLIP	BIRD CLAP	perfectBIRD CLIP	perfectBIRD CLAP	CLIP	CLAP	rt-BIRD CLIP	rt-BIRD CLAP	rt-BIRD CLIP	rt-BIRD CLIP	rt-BIRD CLAP	rt-BIRD CLIP
NS	2		2		2		16		16		16	16		8
chunk duration	-		differs for $^1J_{CH}$ and $^1T_{CH}$ measurement				-		20.48 ms		15.36 ms	10.24 ms		20.48 ms
RMSD [Hz]	0.159	0.322‡	0.155	0.142	0.139	0.139	0.230	0.258	0.258	0.150	0.414	0.324	1.307	0.275
Q[a]	0.028	0.030	0.040	0.044	0.026	0.021	0.013	0.010	0.035	0.016	0.017	0.027	0.046	0.038

[a]: The error weighted quality factor Q was used as defined in ref.[6] ‡: this value is biased towards higher discrepancies by partial signal overlap occurring due to low F_1-resolution, as has been described previously.[1]

Pulse sequence codes for Bruker spectrometers

Real-time BIRD CLIP-HSQC for use on Avance II systems

```
;ek_ti_hsqcCLIP_f3
;broadband proton-homodecoupled CLIP-HSQC with real-time acquisition on F3 channel
;designed for the measurement of one-bond heteronuclear coupling constants
;avance-version (08/04/15) - Avance II, AQS system, SADC digitizer, RX22 receiver, TopSpin 2.1
;using digital mode acquisition
;using explicit acquisition dwellmode
;using gradients during acquisition for water-suppression
;phase sensitive using Echo/Antiecho-TPPI gradient selection
;without decoupling during acquisition
;16-step phase sequence from chunk to chunk
;4-step phase cycle from scan to scan
;
;This pulse sequence is part of
;István Timári, Lukas Kaltschnee, Mária H. Raics, Felix Roth, Nicholle G. A. Bell, Ralph W. Adams,
;Mathias Nilsson, Dusan Uhrin, Gareth A. Morris, Christina M. Thiele and Katalin E. Kövér,
;"Real-time broadband proton-homodecoupled CLIP/CLAP-HSQC for automated measurement of heteronuclear
;one-bond coupling constants",
;manuscript in preparation
;
;The pulse sequence has been coded for test purposes only and
;may contain errors. It does contain arguments that can lead to
;hardware damages if acquisition parameters are set unfavorably.
;The functionality of the pulse sequence itself may differ depending on
;the hardware as well as the software used to execute it. Functionality
;on differing systems cannot be granted.
;Any use of this pulse sequence on a spectrometer is at your own risk.
;
;By using this pulse sequence, or any modification of it in any published material
;you agree to acknowledge the above-mentioned publication.
;
;Further relevant publications:
;A. Enthart, J. C. Freudenberger, J. Furrer, H. Kessler, B. Luy, J. Magn. Reson. 2008, 192, 314-322.
;
;I. Timári, L. Kaltschnee, A. Kolmer, R. W. Adams, M. Nilsson, C. M. Thiele,
;G. A. Morris, K. E. Kövér, J. Magn. Reson. 2014, 239, 130-138.
;
;L. Paudel, R. W. Adams, P. Kiraly, J. A. Aguilar, M. Foroozandeh, M. J. Cliff,
;M. Nilsson, P. Sándor, J. P. Waltho, and G. A. Morris,
;Angew. Chem. Int. Ed., 2013, 52, 11616-11619.
;
;P. Kiraly, R. W. Adams, L. Paudel, M. Foroozandeh, J. A. Aguilar, I. Timári, M. J. Cliff,
;M. Nilsson, P. Sándor, G. Batta, J. P. Waltho, K. E. Kövér and G. A. Morris,
;J. Biomol. NMR, 2015, 62, 43-52.
;

;$CLASS=HighRes
;$DIM=2D
;$TYPE=
;$SUBTYPE=
;$COMMENT=

#include <Avance.incl>
#include <Grad.incl>
#include <Delay.incl>
#include <De.incl>


define loopcounter count
define delay dwell


"p2=p1*2"
"p22=p21*2"
"d4=1s/(cnst2*4)"
"d11=30m"
"d0=3u"
```

```
"in0=inf1/2"

"l3=(td1/2)"

"d20=(1s/(2*cnst2))-5u"

"DELTA3=d4-p16-d16-5u"
"DELTA=p16+d16+p2+d0*2"


"d19=dw*l12"
"l11=(0.5*decim*l12/2)"
"l13=(l12*decim*0.5)"

"count=(aq/d19)-1"

"dwell=(dw/decim)"


;dwellmode explicit

1 ze
  d11

2 d1 do:f3
  3m pl1:f1

3 d11 pl1:f1
  9m

4 15u rpp18  ;;reset phase pointers before each transient
  15u rpp19
  15u rpp20
  15u rpp21
  15u rpp22
  15u rpp23  ;;reset phase pointers

  15u rp19   ;;reset phase ph19 to original value
  15u rp21   ;;reset phase ph21 to original value
  15u rp22   ;;reset phase ph22 to original value

  15u ip23*2 ;;phase ph23 inverted at every other scan (1H 180)

  15u iu1    ;;l1 = 0 initial value, l1 = 1, 2, 3, or 4 depending on the actual value of NS

  if "l1 <= 4" goto lab1
   15u ru1                     ;l1 'scan counter' dependent - varies between 1 - 4
   15u iu1

  lab1, 15u

  if "l1 >=3" goto lab2
   15u ip19*2
   15u ip21*2
   15u ip22*2

  lab2, 15u ip19*2   ;phase ph19 (BIRD 1H-180), ph21 (BIRD 13C-180) and ph22 (second 13C-180) inverted at
every 3rd and 4th scan
        15u ip21*2
        15u ip22*2

5 (p1 ph1)
  d4 pl3:f3
  (center (p2 ph1) (p22 ph6):f3)
  d4 UNBLKGRAD
  (p1 ph2)
  3u
  p16:gp4
  d16
  (p21 ph3):f3
  d0
  (p2 ph1)
  d0
```

```
  p16:gp1
  d16
  4u
  (p22 ph1):f3
  4u
  DELTA


  (p21 ph5):f3
  p16:gp5
  d16
  (p1 ph7)
  d4
  (center (p2 ph1) (p22 ph1):f3 )
  5u
  p16:gp9*EA
  d16
  DELTA3 BLKGRAD

  (p21 ph4):f3                       ;purging residual antiphase magn.

6 ACQ_START(ph30,ph31)               ;initial half chunk

   5u
   5u                                ;detection without decoupling
   10u  REC_UNBLK
   10u  syrec
7 dwell  DWL_CLK_ON
  dwell  DWL_CLK_OFF
lo to 7 times l11
   10u  REC_BLK
   10u sytra
   5u
   5u UNBLKGRAD

p17:gp12       ;gradient pulse for coherence selection and water suppression during acquisition
d16            ;p17 = max. 500 us
5u
(p1 ph18^)
d20
5u
(center (p2 ph19^) (p22 ph21^):f3)
5u
d20
(ralign (p1 ph20^) (p22 ph22^):f3)
5u
p17:gp12       ;gradient pulse for coherence selection and water suppression during acquisition
d16

60u
p19:gp13      ;gradient pulse for coherence selection and water suppression during acquisition
d16           ;p19 = max. 500 us
10u
(p2 ph23^)
10u
p19:gp13       ;gradient pulse for coherence selection and water suppression during acquisition
d16 BLKGRAD

;;looped chunks
8  5u
   5u
   10u  REC_UNBLK
   10u  syrec
9  dwell  DWL_CLK_ON
   dwell  DWL_CLK_OFF
lo to 9 times l13
   10u  REC_BLK
   10u  sytra
   5u
   5u UNBLKGRAD

p17:gp12       ;gradient pulse for coherence selection and water suppression during acquisition
d16            ;p17 = max. 500 us
```

```
5u
(p1 ph18^)
d20
5u
(center (p2 ph19^) (p22 ph21^):f3)
5u
d20
(ralign (p1 ph20^) (p22 ph22^):f3)
5u
p17:gp12       ;gradient pulse for coherence selection and water suppression during acquisition
d16

60u
p19:gp13      ;gradient pulse for coherence selection and water suppression during acquisition
d16           ;p19 = max. 500 us
10u
(p2 ph23^)
10u
p19:gp13       ;gradient pulse for coherence selection and water suppression during acquisition
d16 BLKGRAD

lo to 8 times count

;;final half chunk

   5u
   5u
   10u  REC_UNBLK
   10u  syrec
10  dwell DWL_CLK_ON
   dwell DWL_CLK_OFF
lo to 10 times l11
   10u  REC_BLK
   10u  sytra
   5u
   5u


50u
    rcyc=2

11 d1 do:f3 wr #0 if #0 zd
   3m igrad EA
   lo to 3 times 2

   d11 id0
   3m ip3*2
   3m ip6*2
   3m ip31*2
   lo to 4 times l3

12  exit

;Phase Cycling
;    1 2   4       8              16                              32
ph1= 0
ph2= 1
ph3= 0 0 2 2
ph5= 0 0 0 0 2 2 2 2
ph6= 0
ph7= 0
ph4= 0

ph8= 0  ;BIRD 1st 1H 90
ph9= 1 3  ;BIRD 1H 180
ph10=2  ;BIRD 2nd 1H 90
ph11=1 3 ;BIRD 13C 180
ph12=0 2 ;BIRDd 13C 180
ph13=0 2 ;1H 180

ph29=0 0 0 0 2 2 2 2 2 2 0 0 0 0 2 2     ;;MLEV-8 cycle doubled!!

ph18=ph8  + ph29  ;phase sequence from chunk to chunk!
```

```
ph19=ph9  + ph29
ph20=ph10 + ph29
ph21=ph11 + ph29
ph22=ph12 + ph29
ph23=ph13 + ph29

ph30=0
ph31=0 0 2 2 2 2 0 0

;pl1 : f1 channel - power level for pulse (default)
;pl2 : f2 channel - power level for pulse (default)
;pl3 : f3 channel - power level for pulse (default)
;pl12: f2 channel - power level for CPD/BB decoupling
;pl16: f3 channel - power level for CPD/BB decoupling
;pl0:  pulse power off [120 dB]
;p1 : f1 channel -  90 degree high power pulse
;p2 : f1 channel - 180 degree high power pulse
;p3 : f2 channel -  90 degree high power pulse
;p4 : f2 channel - 180 degree high power pulse
;p21 : f3 channel -  90 degree high power pulse
;p22 : f3 channel - 180 degree high power pulse
;p16: gradient t1 encoding/purging gradient pulse [1 ms]
;p17: gradient pulse [0.5 ms]
;p19: gradient pulse [0.5 ms]
;d0 : incremented delay (2D)                  [3 usec]
;d1 : relaxation delay; 1-5 * T1
;d4 : 1/(4J)XH
;d20: BIRD delay - 1/(2J)XH
;d11: delay for disk I/O                             [30 msec]
;d16: delay for homospoil/gradient recovery
;l1: = 0
;cnst2: = J(XH)
;inf1: 1/SW(X) = 2 * DW(X)
;in0: 1/(2 * SW(X)) = DW(X)
;nd0: 2
;NS(min): 4
;NS: 8 * n
;DS: >= 16
;td1: number of experiments
;FnMODE: echo-antiecho

;use gradient ratio: gp 1 : gp 9
;                      80 : 20.1    for C-13
;                      80 :  8.1    for N-15

;for z-only gradients:
;gpz4: homospoil 13%
;gpz4: homospoil 27%
;gpz12: ctp 61%
;gpz13: ctp 31%

;use gradient files:
;gpnam1: SINE.100
;gpnam4: SINE.100
;gpnam5: SINE.100
;gpnam9: SINE.100
;gpnam12: SINE.50
;gpnam13: SINE.50
```

Real-time BIRD CLAP-HSQC for use on Avance II systems

```
;ek_ti_hsqcCLAP_f3
;broadband proton-homodecoupled CLAP-HSQC with real-time acquisition on F3 channel
;designed for the measurement of one-bond heteronuclear coupling constants
;avance-version (08/04/15) - Avance II, AQS system, SADC digitizer, RX22 receiver, TopSpin 2.1
;using digital mode acquisition
;using explicit acquisition dwellmode
;using gradients during acquisition for water-suppression
;phase sensitive using Echo/Antiecho-TPPI gradient selection
;without decoupling during acquisition
;16-step phase sequence from chunk to chunk
;4-step phase cycle from scan to scan
;
;This pulse sequence is part of
;István Timári, Lukas Kaltschnee, Mária H. Raics, Felix Roth, Nicholle G. A. Bell, Ralph W. Adams,
;Mathias Nilsson, Dusan Uhrin, Gareth A. Morris, Christina M. Thiele and Katalin E. Kövér,
;"Real-time broadband proton-homodecoupled CLIP/CLAP-HSQC for automated measurement of heteronuclear
;one-bond coupling constants",
;manuscript in preparation
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;The pulse sequence has been coded for test purposes only and
;may contain errors. It does contain arguments that can lead to
;hardware damages if acquisition parameters are set unfavorably.
;The functionality of the pulse sequence itself may differ depending on
;the hardware as well as the software used to execute it. Functionality
;on differing systems cannot be granted.
;Any use of this pulse sequence on a spectrometer is at your own risk.
;
;By using this pulse sequence, or any modification of it in any published material
;you agree to acknowledge the above-mentioned publication.
;
;Further relevant publications:
;A. Enthart, J. C. Freudenberger, J. Furrer, H. Kessler, B. Luy, J. Magn. Reson. 2008, 192, 314-322.
;
;I. Timári, L. Kaltschnee, A. Kolmer, R. W. Adams, M. Nilsson, C. M. Thiele,
;G. A. Morris, K. E. Kövér, J. Magn. Reson. 2014, 239, 130-138.
;
;L. Paudel, R. W. Adams, P. Kiraly, J. A. Aguilar, M. Foroozandeh, M. J. Cliff,
;M. Nilsson, P. Sándor, J. P. Waltho, and G. A. Morris,
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;
;P. Kiraly, R. W. Adams, L. Paudel, M. Foroozandeh, J. A. Aguilar, I. Timári, M. J. Cliff,
;M. Nilsson, P. Sándor, G. Batta, J. P. Waltho, K. E. Kövér and G. A. Morris,
;J. Biomol. NMR, 2015, 62, 43-52.
;

;$CLASS=HighRes
;$DIM=2D
;$TYPE=
;$SUBTYPE=
;$COMMENT=

#include <Avance.incl>
#include <Grad.incl>
#include <Delay.incl>
#include <De.incl>


define loopcounter count
define delay dwell


"p2=p1*2"
"p22=p21*2"
"d4=1s/(cnst2*4)"
"d11=30m"
"d0=3u"

"in0=inf1/2"

"l3=(td1/2)"
```

```
"d20=(1s/(2*cnst2))-5u"

"DELTA3=d4-p16-d16-5u"
"DELTA=p16+d16+p2+d0*2"


"d19=dw*l12"
"l11=(0.5*decim*l12/2)"
"l13=(l12*decim*0.5)"

"count=(aq/d19)-1"

"dwell=(dw/decim)"


;dwellmode explicit

1 ze
  d11

2 d1 do:f3
  3m pl1:f1

3 d11 pl1:f1
  9m

4 15u rpp18  ;;reset phase pointers before each transient
  15u rpp19
  15u rpp20
  15u rpp21
  15u rpp22
  15u rpp23  ;;reset phase pointers

  15u rp19   ;; reset phase ph19 to original value
  15u rp21   ;; reset phase ph21 to original value
  15u rp22   ;; reset phase ph22 to original value

  15u ip23*2 ;; phase ph23 inverted at every other scan (1H 180)

  15u iu1    ;;l1 = 0 initial value, l1 = 1, 2, 3, or 4 depending on the actual value of NS

  if "l1 <= 4" goto lab1
   15u ru1                          ;l1 'scan counter' dependent - varies between 1 - 4
   15u iu1

  lab1, 15u

  if "l1 >=3" goto lab2
   15u ip19*2
   15u ip21*2
   15u ip22*2

  lab2, 15u ip19*2   ;phase ph19 (BIRD 1H-180), ph21 (BIRD 13C-180) and ph22 (second 13C-180) inverted at
every 3rd and 4th scan
        15u ip21*2
        15u ip22*2

5 (p1 ph1)
  d4 pl3:f3
  (center (p2 ph1) (p22 ph6):f3)
  d4 UNBLKGRAD
  (p1 ph2)
  3u
  p16:gp4
  d16
  (p21 ph3):f3
  d0
  (p2 ph1)
  d0
  p16:gp1
  d16
  4u
```

```
  (p22 ph1):f3
  4u
  DELTA


  (p21 ph5):f3
  p16:gp5
  d16
  (p1 ph7)
  d4
  (p2 ph1)
  5u
  p16:gp9*EA
  d16
  DELTA3 BLKGRAD


6 ACQ_START(ph30,ph31)               ;initial half chunk

   5u
   5u                                ;detection without decoupling
   10u  REC_UNBLK
   10u  syrec
7 dwell  DWL_CLK_ON
  dwell  DWL_CLK_OFF
lo to 7 times l11
   10u  REC_BLK
   10u sytra
   5u
   5u UNBLKGRAD

p17:gp12       ;gradient pulse for coherence selection and water suppression during acquisition
d16            ;p17 = max. 500 us
5u
(p1 ph18^)
d20
5u
(center (p2 ph19^) (p22 ph21^):f3)
5u
d20
(ralign (p1 ph20^) (p22 ph22^):f3)
5u
p17:gp12       ;gradient pulse for coherence selection and water suppression during acquisition
d16

60u
p19:gp13      ;gradient pulse for coherence selection and water suppression during acquisition
d16           ;p19 = max. 500 us
10u
(p2 ph23^)
10u
p19:gp13       ;gradient pulse for coherence selection and water suppression during acquisition
d16 BLKGRAD

;;looped chunks
8  5u
   5u
   10u  REC_UNBLK
   10u  syrec
9  dwell  DWL_CLK_ON
   dwell  DWL_CLK_OFF
lo to 9 times l13
   10u  REC_BLK
   10u  sytra
   5u
   5u UNBLKGRAD

p17:gp12       ;gradient pulse for coherence selection and water suppression during acquisition
d16            ;p17 = max. 500 us
5u
(p1 ph18^)
d20
5u
```

```
(center (p2 ph19^) (p22 ph21^):f3)
5u
d20
(ralign (p1 ph20^) (p22 ph22^):f3)
5u
p17:gp12       ;gradient pulse for coherence selection and water suppression during acquisition
d16

60u
p19:gp13      ;gradient pulse for coherence selection and water suppression during acquisition
d16           ;p19 = max. 500 us
10u
(p2 ph23^)
10u
p19:gp13       ;gradient pulse for coherence selection and water suppression during acquisition
d16 BLKGRAD

lo to 8 times count

;;final half chunk

   5u
   5u
   10u  REC_UNBLK
   10u  syrec
10  dwell DWL_CLK_ON
   dwell DWL_CLK_OFF
lo to 10 times l11
   10u  REC_BLK
   10u  sytra
   5u
   5u


50u
    rcyc=2

11 d1 do:f3 wr #0 if #0 zd
   3m igrad EA
   lo to 3 times 2

   d11 id0
   3m ip3*2
   3m ip6*2
   3m ip31*2
   lo to 4 times l3

12  exit

;Phase Cycling
;    1 2   4       8              16                              32
ph1= 0
ph2= 1
ph3= 0 0 2 2
ph5= 0 0 0 0 2 2 2 2
ph6= 0
ph7= 0
ph4= 0

ph8= 0  ;BIRD 1st 1H 90
ph9= 1 3  ;BIRD 1H 180
ph10=2  ;BIRD 2nd 1H 90
ph11=1 3 ;BIRD 13C 180
ph12=0 2 ;BIRDd 13C 180
ph13=0 2 ;1H 180

ph29=0 0 0 0 2 2 2 2 2 2 0 0 0 0 2 2     ;;MLEV-8 cycle doubled!!

ph18=ph8  + ph29  ;phase sequence from chunk to chunk!
ph19=ph9  + ph29
ph20=ph10 + ph29
ph21=ph11 + ph29
ph22=ph12 + ph29
```

```
ph23=ph13 + ph29

ph30=0
ph31=0 0 2 2 2 2 0 0

;pl1 : f1 channel - power level for pulse (default)
;pl2 : f2 channel - power level for pulse (default)
;pl3 : f3 channel - power level for pulse (default)
;pl12: f2 channel - power level for CPD/BB decoupling
;pl16: f3 channel - power level for CPD/BB decoupling
;pl0:  pulse power off [120 dB]
;p1 : f1 channel -  90 degree high power pulse
;p2 : f1 channel - 180 degree high power pulse
;p3 : f2 channel -  90 degree high power pulse
;p4 : f2 channel - 180 degree high power pulse
;p21 : f3 channel -  90 degree high power pulse
;p22 : f3 channel - 180 degree high power pulse
;p16: gradient t1 encoding/purging gradient pulse [1 ms]
;p17: gradient pulse [0.5 ms]
;p19: gradient pulse [0.5 ms]
;d0 : incremented delay (2D)                  [3 usec]
;d1 : relaxation delay; 1-5 * T1
;d4 : 1/(4J)XH
;d20: BIRD delay - 1/(2J)XH
;d11: delay for disk I/O                             [30 msec]
;d16: delay for homospoil/gradient recovery
;l1: = 0
;cnst2: = J(XH)
;inf1: 1/SW(X) = 2 * DW(X)
;in0: 1/(2 * SW(X)) = DW(X)
;nd0: 2
;NS(min): 4
;NS: 8 * n
;DS: >= 16
;td1: number of experiments
;FnMODE: echo-antiecho

;use gradient ratio: gp 1 : gp 9
;                      80 : 20.1    for C-13
;                      80 :  8.1    for N-15

;for z-only gradients:
;gpz4: homospoil 13%
;gpz4: homospoil 27%
;gpz12: ctp 61%
;gpz13: ctp 31%

;use gradient files:
;gpnam1: SINE.100
;gpnam4: SINE.100
;gpnam5: SINE.100
;gpnam9: SINE.100
;gpnam12: SINE.50
;gpnam13: SINE.50
```

Real-time BIRD CLIP-HSQC for use on Avance III systems

```
;ek_it_hsqcCLIP_f3_AV-III.lk;lk 20160510
;broadband proton-homodecoupled CLIP-HSQC with real-time acquisition on F3 channel
;designed for the measurement of one-bond heteronuclear coupling constants
;avance-version, tested on Avance III, AQS DRU-E Z102520/02081 ECL 06.01, HPPR/2, TopSpin 3.2pl6
;using digital mode acquisition
;using explicit acquisition dwellmode
;using gradients during acquisition for water-suppression
;phase sensitive using Echo/Antiecho-TPPI gradient selection
;without decoupling during acquisition
;16-step phase sequence from chunk to chunk
;4-step phase cycle from scan to scan
;
;This pulse sequence is part of
;István Timári, Lukas Kaltschnee, Mária H. Raics, Felix Roth, Nicholle G. A. Bell, Ralph W. Adams,
;Mathias Nilsson, Dusan Uhrin, Gareth A. Morris, Christina M. Thiele and Katalin E. Kövér,
;"Real-time broadband proton-homodecoupled CLIP/CLAP-HSQC for automated measurement of heteronuclear
;one-bond coupling constants",
;manuscript in preparation
;
;The pulse sequence has been coded for test purposes only and
;may contain errors. It does contain arguments that can lead to
;hardware damages if acquisition parameters are set unfavorably.
;The functionality of the pulse sequence itself may differ depending on
;the hardware as well as the software used to execute it. Functionality
;on differing systems cannot be granted.
;Any use of this pulse sequence on a spectrometer is at your own risk.
;
;By using this pulse sequence, or any modification of it in any published material
;you agree to acknowledge the above-mentioned publication.
;
;Further relevant publications:
;A. Enthart, J. C. Freudenberger, J. Furrer, H. Kessler, B. Luy, J. Magn. Reson. 2008, 192, 314-322.
;
;I. Timári, L. Kaltschnee, A. Kolmer, R. W. Adams, M. Nilsson, C. M. Thiele,
;G. A. Morris, K. E. Kövér, J. Magn. Reson. 2014, 239, 130-138.
;
;L. Paudel, R. W. Adams, P. Kiraly, J. A. Aguilar, M. Foroozandeh, M. J. Cliff,
;M. Nilsson, P. Sándor, J. P. Waltho, and G. A. Morris,
;Angew. Chem. Int. Ed., 2013, 52, 11616-11619.
;
;P. Kiraly, R. W. Adams, L. Paudel, M. Foroozandeh, J. A. Aguilar, I. Timári, M. J. Cliff,
;M. Nilsson, P. Sándor, G. Batta, J. P. Waltho, K. E. Kövér and G. A. Morris,
;J. Biomol. NMR, 2015, 62, 43-52.
;


;
;$CLASS=HighRes
;$DIM=2D
;$TYPE=
;$SUBTYPE=
;$COMMENT=

#include <Avance.incl>
#include <Grad.incl>
#include <Delay.incl>
#include <De.incl>


define loopcounter count
;define list<gradient> gl1 = {0.5 0.4 0.675 0.875 0.2 0.575 0.975 0.95 0.825 1.0 0.325 0.225 0.625 0.85
0.45 0.775 0.25 0.75 0.425 0.925 0.525 0.275 0.65 0.3 0.725 0.35 0.55 0.9 0.7 0.475 0.8 0.6 0.375}
       ; added gradient list to vary the strength of the gradients during acquisition dynamically.
Currently not used. lk20150323
define list<gradient> gl1 = {1}           ; Gradient list for BIRDd elements. Not using gradient scaling
via a list.  lk20150323
define list<gradient> gl2 = {1}           ; Gradient list for hard inversion pulses elements. Not using
gradient scaling via a list.  lk20150323

"p2=p1*2"
"p22=p21*2"
"d4=1s/(cnst2*4)"
```

```
"d11=30m"
"d0=3u"
"in0=inf1/2"
"d20=1s/(2*cnst2)"



"l1=0"

"DELTA3=d4-p16-d16-5u"
"DELTA=p16+d16+p2+d0*2"



;Real time control delays
"d19=dw*l12"
"DELTA7=d19/2" ;half chunk time

"count=(aq/d19)-1"

dwellmode explicit

1 ze

  d11

2 d1
  3m pl1:f1

;3 d11 pl1:f1
;  9m

4 6u rpp18  ;;reset phase pointers before each transient
  6u rpp19
  6u rpp20
  6u rpp21
  6u rpp22
  6u rpp23  ;;reset phase pointers

  6u rp19   ;; reset phase ph19 to original value
  6u rp21   ;; reset phase ph21 to original value
  6u rp22   ;; reset phase ph22 to original value

  6u ip23*2 ;; phase ph23 inverted at every other scan (1H 180)


;inversion of ph19, ph21 and ph22 every second scan:
  6u iu1    ;;l1 = 0 initial value, l1 = 1, 2, 3, or 4 depending on the actual value of NS

  if "l1 <= 4" goto lab1
   6u ru1                          ;l1 'scan counter' dependent - varies between 1 - 4
   6u iu1

  lab1, 6u

  if "l1 >=3" goto lab2
   6u ip19*2
   6u ip21*2
   6u ip22*2

  lab2, 6u ip19*2   ;phase ph19 (BIRD 1H-180), ph21 (BIRD 13C-180) and ph22 (second 13C-180) inverted at
every 3rd and 4th scan
        6u ip21*2
        6u ip22*2


; using the same gradient lists for all scans, lk20150323
  rgrad gl1
  rgrad gl2


5 (p1 ph1)
  d4 pl3:f3
```

```
  (center (p2 ph1) (p22 ph6):f3)
  d4 UNBLKGRAD
  (p1 ph2)
  3u
  p16:gp4
  d16
  (p21 ph3):f3
  d0
  (p2 ph1)
  d0
  p16:gp1
  d16
  4u
  (p22 ph1):f3
  4u
  DELTA


  (p21 ph5):f3
  p16:gp5                                     ;purging gradient
  d16
  (p1 ph7)
  d4
  (center (p2 ph1) (p22 ph1):f3 )
  5u
  p16:gp2*EA
  d16
  DELTA3 BLKGRAD

  (p21 ph4):f3                                 ;purging residual antiphase magn.

6 ACQ_START(ph30,ph31) ;takes de

;initial half chunk
  5u
  5u ;cpd2:f2

  10u  REC_UNBLK
  10u  DWL_CLK_ON
  DELTA7
  10u  DWL_CLK_OFF
  10u  REC_BLK
  5u do:f2
  5u UNBLKGRAD

p17:gp12*gl1
d16            ;p17 = max. 500 us
5u
(p1 ph18^)
d20 pl3:f3
2u
(center (p2 ph19^) (p22 ph21^):f3)
2u
d20
(ralign (p1 ph20^) (p22 ph22^):f3)
5u
p17:gp12*gl1
d16
igrad gl1

60u
p19:gp13*gl2
d16           ;p19 = max. 500 us
d31
(p2 ph23^)
d31
p19:gp13*gl2
d16 BLKGRAD
igrad gl2

;looped chunks
8 5u
  5u ;cpd2:f2
```

```
  10u  REC_UNBLK
  10u  DWL_CLK_ON
  d19
  10u  DWL_CLK_OFF
  10u  REC_BLK
  5u do:f2
  5u UNBLKGRAD


p17:gp12*gl1
d16            ;p17 = max. 500 us
5u
(p1 ph18^)
d20 pl3:f3
2u
(center (p2 ph19^) (p22 ph21^):f3)
2u
d20
(ralign (p1 ph20^) (p22 ph22^):f3)
5u
p17:gp12*gl1
d16
igrad gl1

60u
p19:gp13*gl2
d16           ;p19 = max. 500 us
d31
(p2 ph23^)
d31
p19:gp13*gl2
d16 BLKGRAD
igrad gl2


lo to 8 times count

;;final half chunk

  5u
  5u ;cpd2:f2

  10u  REC_UNBLK
  10u  DWL_CLK_ON
  DELTA7
  10u  DWL_CLK_OFF
  10u  REC_BLK
  5u do:f2


50u
    rcyc=2
    d1 mc #0 to 2
              F1EA(calgrad(EA), caldel(d0, +in0) & calph(ph3, +180) & calph(ph6, +180) & calph(ph31,
+180))
7   exit

;Phase Cycling
;    1 2   4       8              16                              32

;phases used in CLIP-HSQC block
ph1= 0
ph2= 1
ph3= 0 0 2 2
ph4= 0
ph5= 0 0 0 0 2 2 2 2
ph6= 0
ph7= 0

;basic phase cycle for decoupling element -> will be modulated with an MLEV8 cycle
ph8= 0  ;BIRD 1st 1H 90
ph9= 1 3 ;BIRD 1H 180
```

```
ph10=2   ;BIRD 2nd 1H 90
ph11=1 3 ;BIRD 13C 180
ph12=0 2 ;BIRDd 13C 180
ph13=0 2 ;1H 180

;MLEV-8 cycle (doubled)
ph29=0 0 0 0 2 2 2 2 2 2 2 0 0 0 0 2 2

;MLEV-8 cycle for BIRDd elements
ph18=ph8  + ph29  ;BIRD with MLEV-8 cycle from chunk to chunk!!
ph19=ph9  + ph29
ph20=ph10 + ph29
ph21=ph11 + ph29
ph22=ph12 + ph29
ph23=ph13 + ph29

;phase cycle for acquisition
ph30=0
ph31=0 0 2 2 2 2 0 0

;pl1 : f1 channel - power level for pulse (default)
;pl2 : f2 channel - power level for pulse (default)
;pl3 : f3 channel - power level for pulse (default)
;pl0:  pulse power off [120 dB]
;p1 : f1 channel -  90 degree high power pulse
;p2 : f1 channel - 180 degree high power pulse
;p3 : f2 channel -  90 degree high power pulse
;p4 : f2 channel - 180 degree high power pulse
;p16: gradient pulse duration (HSQC) [1 ms]
;p17: G-BIRD gradient duration (ACQU) [0.2 ms]
;p19: hard inversion gradient duration (ACQU) [0.2 ms]
;d0 : incremented delay (2D)                  [3 usec]
;d1 : relaxation delay; 1-5 * T1
;d4 : 1/(4J)XH
;d20: BIRD delay - 1/(2J)XH
;d11: delay for disk I/O                             [30 msec]
;d16: delay for homospoil/gradient recovery
;cnst2: = J(XH)
;inf1: 1/SW(X) = 2 * DW(X)
;in0: 1/(2 * SW(X)) = DW(X)

;NS(min): 4
;NS: 8 * n
;DS: >= 16
;td1: number of experiments
;FnMODE: echo-antiecho
;d31: Bird - 180 interpulse delay [10u]

;use gradient ratio: gp 1 : gp 2
;                      80 : 20.1    for C-13
;                      80 :  8.1    for N-15

;for z-only gradients:
;gpz1: 80%
;gpz2: 20.1% for C-13, 8.1% for N-15
;gpz4: 55%
;gpz5: 32%
;gpz12: 11%
;gpz13: 14%

;use gradient files:
;gpnam1: SINE.100
;gpnam2: SINE.100
;gpnam4: SINE.100
;gpnam5: SINE.100
;gpnam12: SINE.50
;gpnam13: SINE.50
```

Real-time BIRD CLAP-HSQC for use on Avance III systems

```
;ek_it_hsqcCLAP_f3_AV-III.lk;lk 20160510
;broadband proton-homodecoupled CLAP-HSQC with real-time acquisition on F3 channel
;designed for the measurement of one-bond heteronuclear coupling constants
;avance-version, tested on Avance III, AQS DRU-E Z102520/02081 ECL 06.01, HPPR/2, TopSpin 3.2pl6
;using digital mode acquisition
;using explicit acquisition dwellmode
;using gradients during acquisition for water-suppression
;phase sensitive using Echo/Antiecho-TPPI gradient selection
;without decoupling during acquisition
;16-step phase sequence from chunk to chunk
;4-step phase cycle from scan to scan
;
;This pulse sequence is part of
;István Timári, Lukas Kaltschnee, Mária H. Raics, Felix Roth, Nicholle G. A. Bell, Ralph W. Adams,
;Mathias Nilsson, Dusan Uhrin, Gareth A. Morris, Christina M. Thiele and Katalin E. Kövér,
;"Real-time broadband proton-homodecoupled CLIP/CLAP-HSQC for automated measurement of heteronuclear
;one-bond coupling constants",
;manuscript in preparation
;
;The pulse sequence has been coded for test purposes only and
;may contain errors. It does contain arguments that can lead to
;hardware damages if acquisition parameters are set unfavorably.
;The functionality of the pulse sequence itself may differ depending on
;the hardware as well as the software used to execute it. Functionality
;on differing systems cannot be granted.
;Any use of this pulse sequence on a spectrometer is at your own risk.
;
;By using this pulse sequence, or any modification of it in any published material
;you agree to acknowledge the above-mentioned publication.
;
;Further relevant publications:
;A. Enthart, J. C. Freudenberger, J. Furrer, H. Kessler, B. Luy, J. Magn. Reson. 2008, 192, 314-322.
;
;I. Timári, L. Kaltschnee, A. Kolmer, R. W. Adams, M. Nilsson, C. M. Thiele,
;G. A. Morris, K. E. Kövér, J. Magn. Reson. 2014, 239, 130-138.
;
;L. Paudel, R. W. Adams, P. Kiraly, J. A. Aguilar, M. Foroozandeh, M. J. Cliff,
;M. Nilsson, P. Sándor, J. P. Waltho, and G. A. Morris,
;Angew. Chem. Int. Ed., 2013, 52, 11616-11619.
;
;P. Kiraly, R. W. Adams, L. Paudel, M. Foroozandeh, J. A. Aguilar, I. Timári, M. J. Cliff,
;M. Nilsson, P. Sándor, G. Batta, J. P. Waltho, K. E. Kövér and G. A. Morris,
;J. Biomol. NMR, 2015, 62, 43-52.
;

;
;$CLASS=HighRes
;$DIM=2D
;$TYPE=
;$SUBTYPE=
;$COMMENT=

#include <Avance.incl>
#include <Grad.incl>
#include <Delay.incl>
#include <De.incl>


define loopcounter count
;define list<gradient> gl1 = {0.5 0.4 0.675 0.875 0.2 0.575 0.975 0.95 0.825 1.0 0.325 0.225 0.625 0.85
0.45 0.775 0.25 0.75 0.425 0.925 0.525 0.275 0.65 0.3 0.725 0.35 0.55 0.9 0.7 0.475 0.8 0.6 0.375}
	; added gradient list to vary the strength of the gradients during acquisition dynamically.
Currently not used. lk20150323
define list<gradient> gl1 = {1}		; Gradient list for BIRDd elements. Not using gradient scaling
via a list.  lk20150323
define list<gradient> gl2 = {1}		; Gradient list for hard inversion pulses elements. Not using
gradient scaling via a list.  lk20150323

"p2=p1*2"
"p22=p21*2"
```

```
"d4=1s/(cnst2*4)"
"d11=30m"
"d0=3u"
"in0=inf1/2"
"d20=1s/(2*cnst2)"

"d7=p16+d16+4u"

"l1=0"

"DELTA3=d4-p16-d16-5u"
"DELTA=p16+d16+p2+d0*2"



;Real time control delays
"d19=dw*l12"
"DELTA7=d19/2" ;half chunk time

"count=(aq/d19)-1"

dwellmode explicit

1 ze

  d11

2 d1
  3m pl1:f1

;3 d11 pl1:f1
;  9m

4 6u rpp18  ;;reset phase pointers before each transient
  6u rpp19
  6u rpp20
  6u rpp21
  6u rpp22
  6u rpp23  ;;reset phase pointers

  6u rp19   ;; reset phase ph19 to original value
  6u rp21   ;; reset phase ph21 to original value
  6u rp22   ;; reset phase ph22 to original value

  6u ip23*2 ;; phase ph23 inverted at every other scan (1H 180)


;inversion of ph19, ph21 and ph22 every second scan:
  6u iu1    ;;l1 = 0 initial value, l1 = 1, 2, 3, or 4 depending on the actual value of NS

  if "l1 <= 4" goto lab1
   6u ru1                          ;l1 'scan counter' dependent - varies between 1 - 4
   6u iu1

  lab1, 6u

  if "l1 >=3" goto lab2
   6u ip19*2
   6u ip21*2
   6u ip22*2

  lab2, 6u ip19*2   ;phase ph19 (BIRD 1H-180), ph21 (BIRD 13C-180) and ph22 (second 13C-180) inverted at
every 3rd and 4th scan
        6u ip21*2
        6u ip22*2


; using the same gradient lists for all scans, lk20150323
  rgrad gl1
  rgrad gl2


5 (p1 ph1)
```

```
  d4 pl3:f3
  (center (p2 ph1) (p22 ph6):f3)
  d4 UNBLKGRAD
  (p1 ph2)
  3u
  p16:gp4
  d16
  (p21 ph3):f3
  d0
  (p2 ph1)
  d0
  p16:gp1
  d16
  4u
  (p22 ph1):f3
  4u
  DELTA


  (p21 ph5):f3
  p16:gp5                                     ;purging gradient
  d16
  (p1 ph7)

  ; CLAP-version
  d7
  (p2 ph1)
  4u
  p16:gp2*EA
  d16 BLKGRAD


6 ACQ_START(ph30,ph31) ;takes de

;initial half chunk
  5u
  5u ;cpd2:f2

  10u  REC_UNBLK
  10u  DWL_CLK_ON
  DELTA7
  10u  DWL_CLK_OFF
  10u  REC_BLK
  5u do:f2
  5u UNBLKGRAD

p17:gp12*gl1
d16            ;p17 = max. 500 us
5u
(p1 ph18^)
d20 pl3:f3
2u
(center (p2 ph19^) (p22 ph21^):f3)
2u
d20
(ralign (p1 ph20^) (p22 ph22^):f3)
5u
p17:gp12*gl1
d16
igrad gl1

60u
p19:gp13*gl2
d16           ;p19 = max. 500 us
d31
(p2 ph23^)
d31
p19:gp13*gl2
d16 BLKGRAD
igrad gl2

;looped chunks
8 5u
```

```
  5u ;cpd2:f2

  10u  REC_UNBLK
  10u  DWL_CLK_ON
  d19
  10u  DWL_CLK_OFF
  10u  REC_BLK
  5u do:f2
  5u UNBLKGRAD


p17:gp12*gl1
d16            ;p17 = max. 500 us
5u
(p1 ph18^)
d20 pl3:f3
2u
(center (p2 ph19^) (p22 ph21^):f3)
2u
d20
(ralign (p1 ph20^) (p22 ph22^):f3)
5u
p17:gp12*gl1
d16
igrad gl1

60u
p19:gp13*gl2
d16           ;p19 = max. 500 us
d31
(p2 ph23^)
d31
p19:gp13*gl2
d16 BLKGRAD
igrad gl2


lo to 8 times count

;;final half chunk

  5u
  5u ;cpd2:f2

  10u  REC_UNBLK
  10u  DWL_CLK_ON
  DELTA7
  10u  DWL_CLK_OFF
  10u  REC_BLK
  5u do:f2


50u
    rcyc=2
    d1 mc #0 to 2
              F1EA(calgrad(EA), caldel(d0, +in0) & calph(ph3, +180) & calph(ph6, +180) & calph(ph31,
+180))
7   exit

;Phase Cycling
;    1 2   4       8              16                              32

;phases used in CLAP-HSQC block
ph1= 0
ph2= 1
ph3= 0 0 2 2

ph5= 0 0 0 0 2 2 2 2
ph6= 0
ph7= 0

;basic phase cycle for decoupling element -> will be modulated with an MLEV8 cycle
ph8= 0   ;BIRD 1st 1H 90
```

```
ph9= 1 3 ;BIRD 1H 180
ph10=2   ;BIRD 2nd 1H 90
ph11=1 3 ;BIRD 13C 180
ph12=0 2 ;BIRDd 13C 180
ph13=0 2 ;1H 180

;MLEV-8 cycle (doubled)
ph29=0 0 0 0 2 2 2 2 2 2 0 0 0 0 2 2

;MLEV-8 cycle for BIRDd elements
ph18=ph8  + ph29  ;BIRD with MLEV-8 cycle from chunk to chunk!!
ph19=ph9  + ph29
ph20=ph10 + ph29
ph21=ph11 + ph29
ph22=ph12 + ph29
ph23=ph13 + ph29

;phase cycle for acquisition
ph30=0
ph31=0 0 2 2 2 2 0 0

;pl1 : f1 channel - power level for pulse (default)
;pl2 : f2 channel - power level for pulse (default)
;pl3 : f3 channel - power level for pulse (default)
;pl0:  pulse power off [120 dB]
;p1 : f1 channel -  90 degree high power pulse
;p2 : f1 channel - 180 degree high power pulse
;p3 : f2 channel -  90 degree high power pulse
;p4 : f2 channel - 180 degree high power pulse
;p16: gradient pulse duration (HSQC) [1 ms]
;p17: G-BIRD gradient duration (ACQU) [0.2 ms]
;p19: hard inversion gradient duration (ACQU) [0.2 ms]
;d0 : incremented delay (2D)                  [3 usec]
;d1 : relaxation delay; 1-5 * T1
;d4 : 1/(4J)XH
;d20: BIRD delay - 1/(2J)XH
;d11: delay for disk I/O                             [30 msec]
;d16: delay for homospoil/gradient recovery
;cnst2: = J(XH)
;inf1: 1/SW(X) = 2 * DW(X)
;in0: 1/(2 * SW(X)) = DW(X)

;NS(min): 4
;NS: 8 * n
;DS: >= 16
;td1: number of experiments
;FnMODE: echo-antiecho
;d31: Bird - 180 interpulse delay [10u]

;use gradient ratio: gp 1 : gp 2
;                      80 : 20.1    for C-13
;                      80 :  8.1    for N-15

;for z-only gradients:
;gpz1: 80%
;gpz2: 20.1% for C-13, 8.1% for N-15
;gpz4: 55%
;gpz5: 32%
;gpz12: 11%
;gpz13: 14%

;use gradient files:
;gpnam1: SINE.100
;gpnam2: SINE.100
;gpnam4: SINE.100
;gpnam5: SINE.100
;gpnam12: SINE.50
;gpnam13: SINE.50
```

References

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