Electronic Supplementary Material (ESI) for ChemComm.

Enantiodiscrimination by Matrix-Assisted DOSY NMR

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Supplemental Information

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Experimental details

Compounds **1-11** and **22** were obtained commercially and were used without further purification. The phenylethanol derivatives (compounds **12-21**) were obtained by reducing the carbonyl of the correspondent acetophenone using 1.2 mols of $LiAlH_4$ and 1 mol of acetophenone in THF, at 0 ºC. The reactions were monitored by TLC and were quenched with water when all the reagent was consumed. The crude product was extracted, dried and no further purification was necessary.

NMR compounds were prepared using 8.0 µmol of analyte and 41 µmol of matrixes **1-4** or 8.0 µmol of **5** in 300 µL of $CDCl_3$ using 3 mm NMR tubes. All DOSY measurements were carried out non-spinning on an 11.74 Tesla Bruker AVANCE-III spectrometer, equipped with a 5mm BBO SmartProbe, with a z-gradient coil producing a nominal maximum gradient of 50 G cm^{-1}, operating at 499.87 and 470.29 MHz for ^{1}H and ^{19}F, respectively.

The pulse sequence chosen to measure diffusion coefficients was the Oneshot45 for ^{1}H and ^{19}F nuclei. The data were acquired with 32 gradient amplitudes (TD 2), 32K points (TD 1), 32 scans (NS) and 32 dummy scans (DS). The delay for gradient recovery (d16) and duration of the gradient purge pulse (p19) were fixed at 0.2 and 0.6 ms, respectively. The diffusion delay Δ (d20) and the diffusion-encoding pulse duration δ (p30) were optimized for each compound. The experiments were carried out at 25 ºC unless stated otherwise.

Diffusion coefficients were obtained using the GNAT[1] software, with two zero fillings and reference deconvolution[2] and DOSY maps were plotted using the software Origin 8.1 The errors in Tables S1-S42 are the standard errors estimated in the fitting procedure.

ΔD of diastereoisomeric complexes.

Table S1: difference in diffusion values (ΔD) of the diastereoisomeric complexes.

Entry	Analyte:CSA	**ΔD[a]**	**Error[a]**
1	**6/7:1**	0.22	0.21
2	**6/7:2**	0.28	0.24
3	**8/9:1**	0.20	0.16
4	**8/9:2**	0.20	0.17
5	**8/9:5**	0.57	0.08
6	**10:1**	0.19	0.10
7	**10:2**	0.20	0.10
8	**11:1**	0.23	0.09
9	**11:2**	0.20	0.10
10	**12:1**	0.82	0.17
11	**12:2**	0.95	0.17
12	**13:1**	0.34	0.16
13	**13:2**	0.35	0.12
14	**14:1**	0.55	0.19
15	**14:2**	0.55	0.08
16	**15:1**	0.74	0.40
17	**15:2**	0.59	0.15
18	**16:1**	0.0	0.0
19	**16:2**	0.0	0.0
20	**16:3**	0.0	0.0
21	**16:4**	0.0	0.0
22	**17:3**	0.15	0.18
23	**17:4**	0.21	0.19
24	**18:1**	0.45	0.12
25	**18:2**	0.24	0.10
26	**18:3**	0.29	0.16
27	**18:4**	0.28	0.15
28	**19:1**	0.0	0.0
29	**19:2**	0.0	0.0
30	**19:3**	0.0	0.0
31	**19:4**	0.0	0.0
32	**19:5** ($^{19}F\{^{1}H\}$)	1.56	0.33
33	**19:5** (^{1}H)	0.21	0.14
34	**20:1** ($^{19}F\{^{1}H\}$)	1.03	0.32
35	**20:1** (^{1}H)	0.36	0.19
36	**20:2** ($^{19}F\{^{1}H\}$)	1.54	0.19
37	**20:2** (^{1}H)	0.54	0.13
38	**20:5** ($^{19}F\{^{1}H\}$)	0.43	0.08
39	**20:5** (^{1}H)	0.06	0.17
40	**21:1** ($^{19}F\{^{1}H\}$)	1.47	0.17
41	**21:1** (^{1}H)	0.15	0.18
42	**21:2** ($^{19}F\{^{1}H\}$)	1.16	0.13
43	**21:2** (^{1}H)	0.17	0.12
44	**21:5** ($^{19}F\{^{1}H\}$)	0.91	0.13
45	**21:5** (^{1}H)	0.45	0.14
46	**22:1**	0.30	0.04
47	**22:2**	0.32	0.03
48	**22:3**	1.23	0.10
49	**22:4**	1.22	0.14
50	**22:5**	0.22	0.09

[a] $\times 10^{-10}$ m^2s^{-1}.

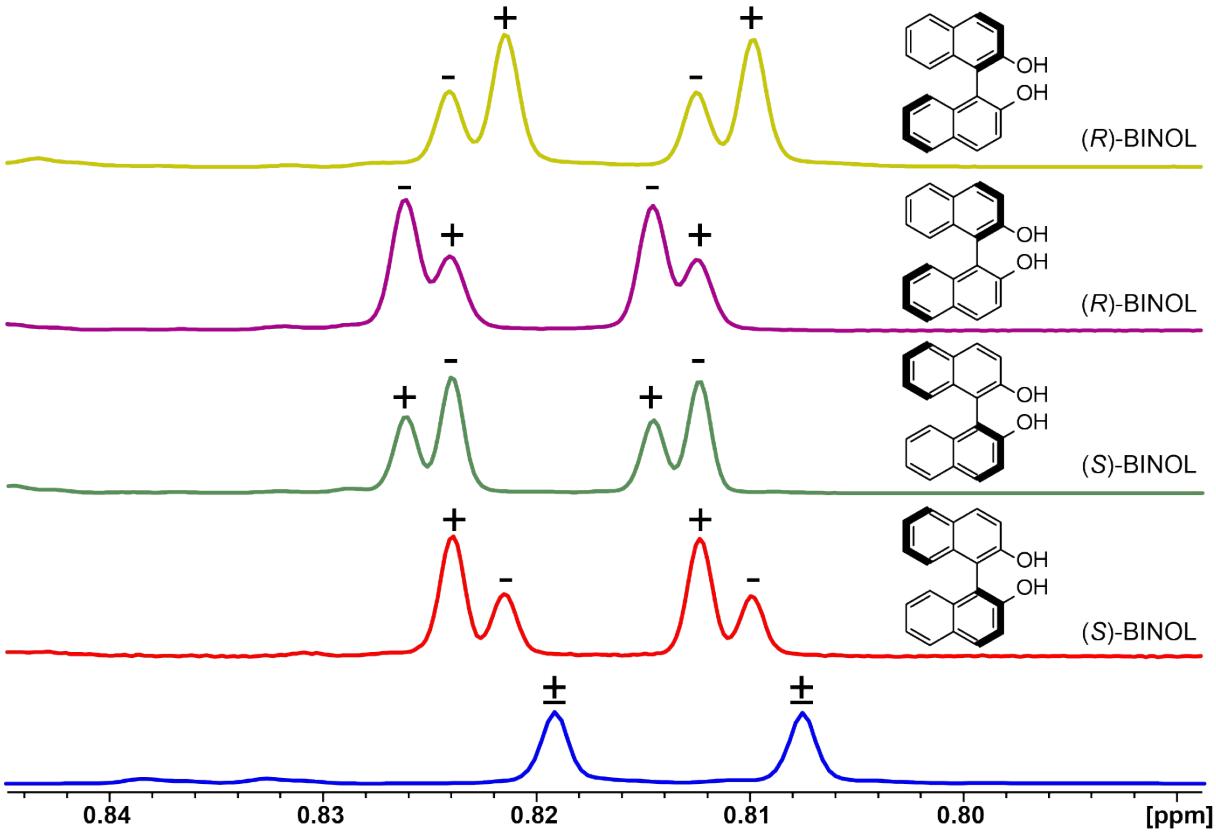


Figure S1: 500 MHz ^{1}H NMR of (+) and (-)-menthol and (*R*) or (*S*)-BINOL as CSA. From bottom to top: racemic menthol (blue); excess of (+)-menthol and (*S*)-BINOL as CSA (red); excess of (-)-menthol and (*S*)-BINOL as CSA (green); excess of (-)-menthol and (*R*)-BINOL as CSA (purple); excess of (+)-menthol and (*R*)-BINOL as CSA (yellow)

The following image is an example of how the difference between diffusion coefficients was calculated for signals with multiplicity higher than 1.

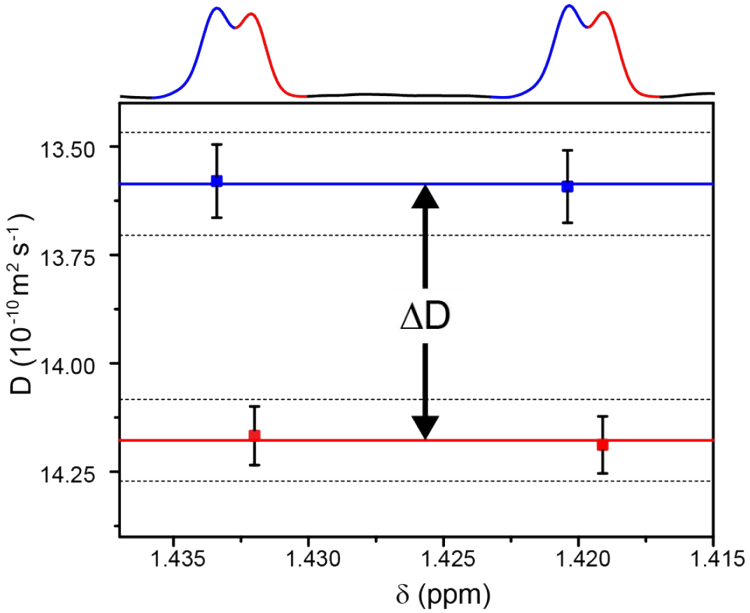


Figure S2: 500 MHz ^{1}H MAD projections with the least attenuated 1D spectrum shown at the top, for 2'-NO_2-1-phenylethanol and (*S*)-BINOL

In Fig. S2 each diastereoisomeric complex presents one doublet, represented in red or blue and each transition of the doublet exhibits its own D (colored squares). The average of each doublet is expressed by the straight line of the same color and the dot line is the respective error of the fitting procedure. The ΔD is then calculated by the difference between both straight lines following the ensuing formula:

$$\bar{D} = \frac{D_1 + D_2}{2} \pm \sqrt{error_1^2 + error_2^2} = \frac{13.58 + 13.59}{2} \pm \sqrt{0.08^2 + 0.08^2}$$

$$\bar{D} = 13.59 \pm 0.12$$

$$\bar{D} = \frac{D_1 + D_2}{2} \pm \sqrt{error_1^2 + error_2^2} = \frac{14.17 + 14.19}{2} \pm \sqrt{0.07^2 + 0.07^2}$$

$$\bar{D} = 14.18 \pm 0.09$$

$$\Delta D = \bar{D} - \bar{D} \pm \sqrt{error^2 + error^2}$$

$$\bar{\Delta D} = 14.18 - 13.59 \pm \sqrt{0.12^2 + 0.09^2}$$

$$\bar{\Delta D} = 0.59 \pm 0.15$$

500 MHz ^{1}H MAD projections and measured diffusion coefficients of diastereoisomeric complexes.

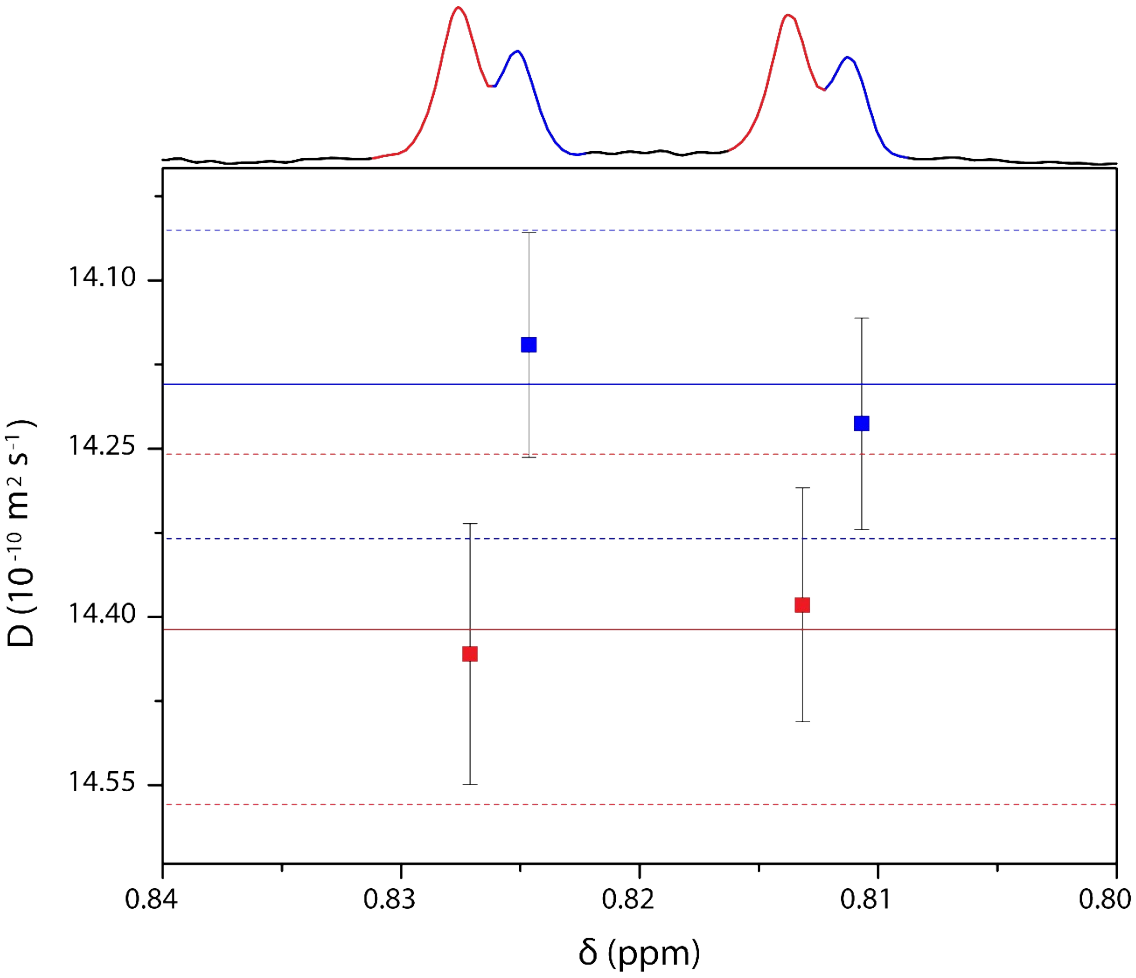


Figure S3: 500 MHz ^{1}H MAD projections, with the least attenuated 1D spectrum shown at the top, for compounds **6** and **7** with matrix **1**

Table S1: Measured ^{1}H diffusion coefficients of compounds **6** and **7** with matrix **1**

Chemical Shift	Exp Amplitude	Fit. Amplitude	Error	Diffusion coefficient[a]	Error[a]
0.81069	1.83774	1.88250	0.00703	14.20115	0.09163
0.81317	2.69824	2.75841	0.00947	14.29452	0.08462
0.82464	1.90680	1.95299	0.00735	14.16563	0.09220
0.82711	2.60667	2.67378	0.01149	14.35075	0.10610

[a] × 10^{-10} m^2s^{-1}.

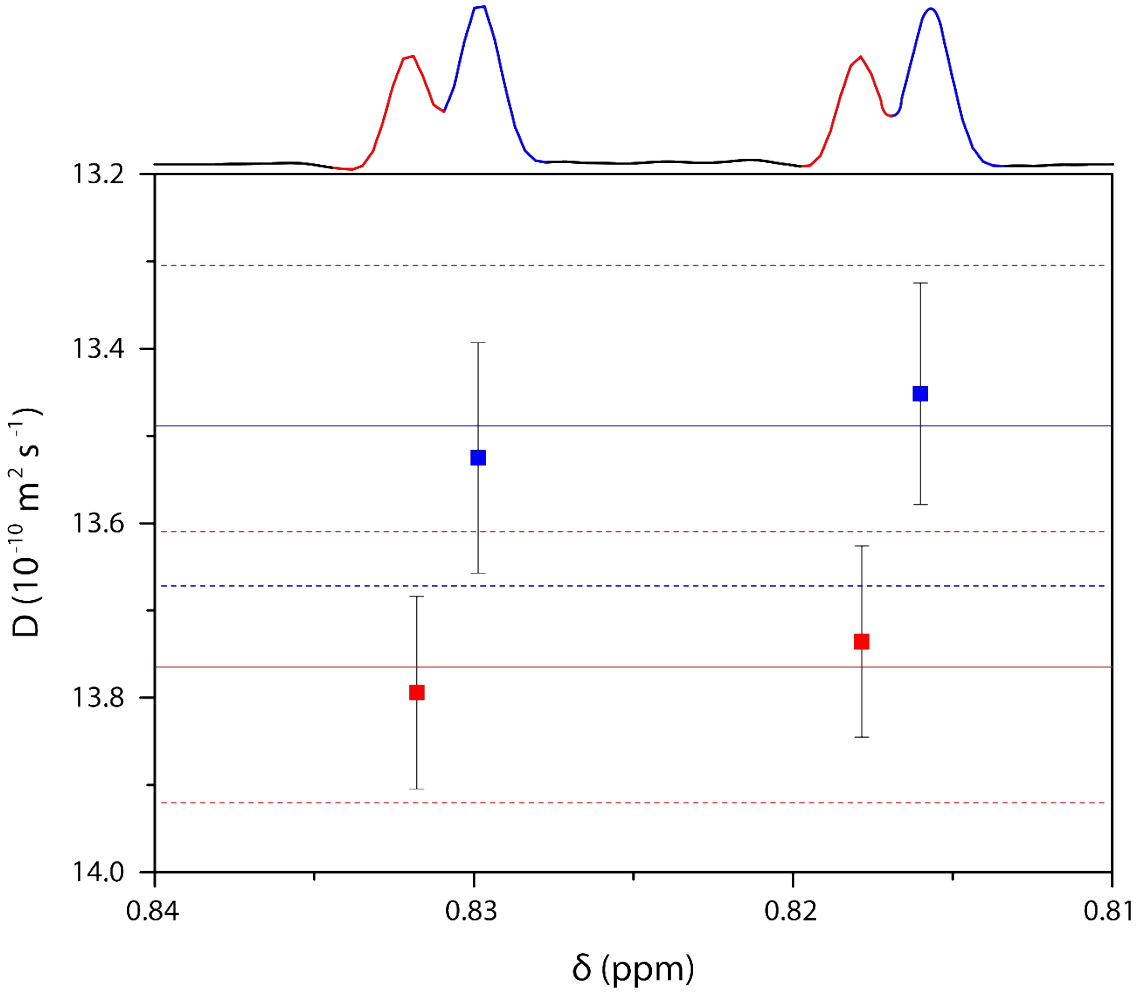


Figure S4: 500 MHz ^{1}H MAD projections, with the least attenuated 1D spectrum shown at the top, for compounds **6** and **7** with matrix **2**

Table S2: Measured ^{1}H diffusion coefficients of compounds **6** and **7** with matrix **2**

Chemical Shift	Exp Amplitude	Fit. Amplitude	Error	Diffusion coefficient[a]	Error[a]
0.81601	3.16413	3.25539	0.01739	13.45170	0.12705
0.81785	1.89178	1.94027	0.00884	13.73564	0.10960
0.82987	3.08017	3.18431	0.01766	13.52534	0.13231
0.83179	1.63426	1.68135	0.00769	13.79421	0.11037

[a] × 10^{-10} m^2s^{-1}.

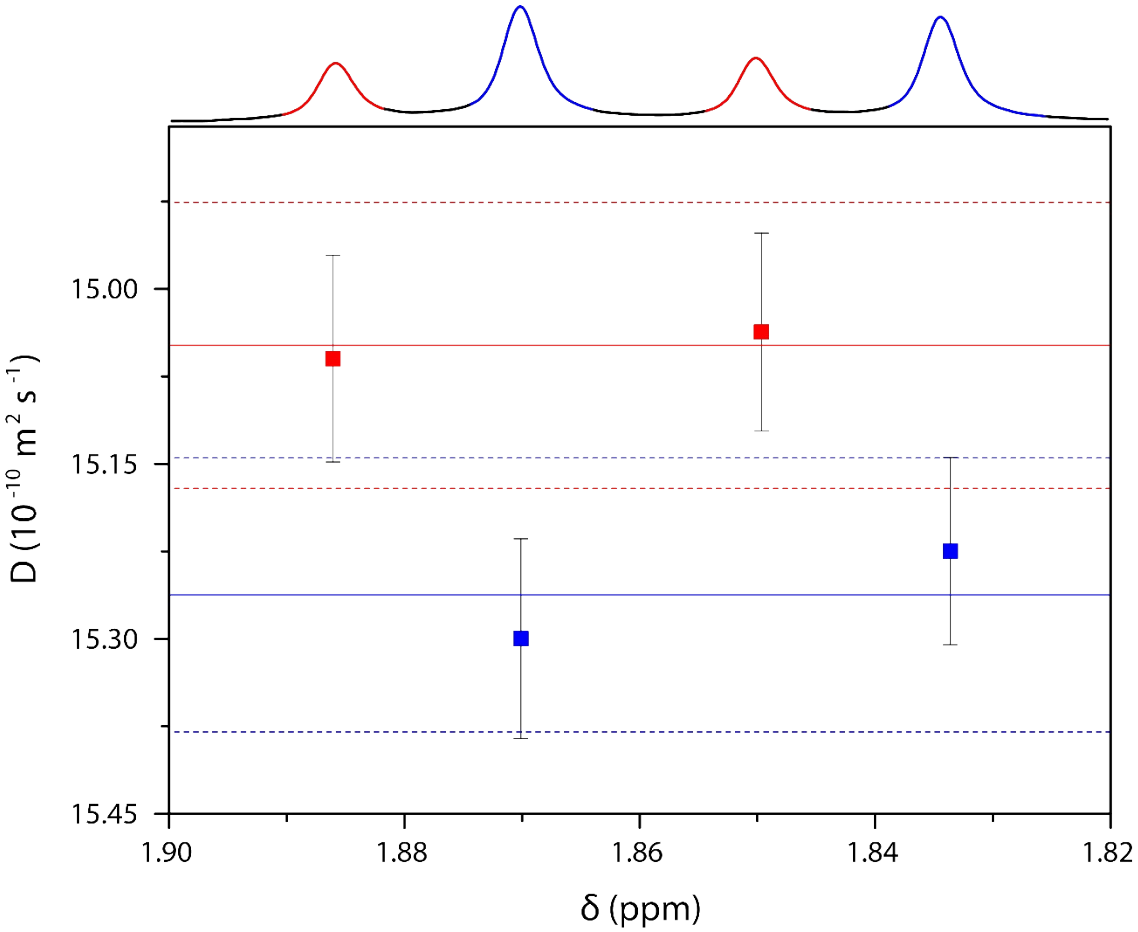


Figure S5: 500 MHz ^{1}H MAD projections, with the least attenuated 1D spectrum shown at the top, for compounds **8** and **9** with matrix **1**

Table S3: Measured ^{1}H diffusion coefficients of compounds **8** and **9** with matrix **1**

Chemical Shift	Exp Amplitude	Fit. Amplitude	Error	Diffusion coefficient[a]	Error[a]
1.83360	1.15353	1.17542	0.00311	15.18982	0.07498
1.84965	0.72405	0.74075	0.00208	15.01451	0.07918
1.87010	1.28234	1.30945	0.00369	15.25986	0.07987
1.88606	0.65486	0.67133	0.00197	15.03576	0.08269

[a] × 10^{-10} m^2s^{-1}.

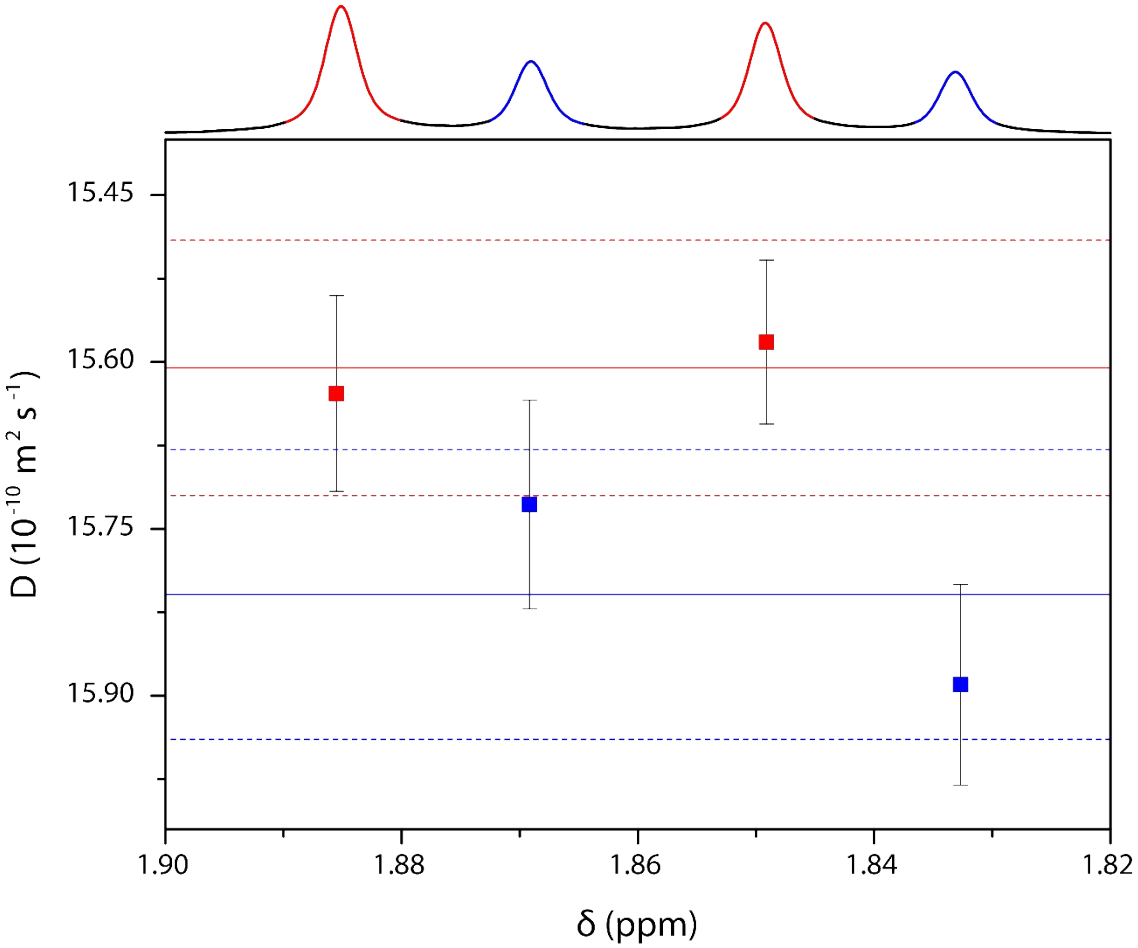


Figure S6: 500 MHz ^{1}H MAD projections, with the least attenuated 1D spectrum shown at the top, for compounds **8** and **9** with matrix **2**

Table S4: Measured ^{1}H diffusion coefficients of compounds **8** and **9** with matrix **2**

Chemical Shift	Exp Amplitude	Fit. Amplitude	Error	Diffusion coefficient[a]	Error[a]
1.83268	0.56628	0.57852	0.00180	15.89014	0.09020
1.84910	1.09474	1.11299	0.00286	15.58205	0.07361
1.86918	0.65354	0.66838	0.00218	15.72805	0.09379
1.88551	1.00919	1.02785	0.00315	15.62822	0.08799

[a] × 10^{-10} m^2s^{-1}.

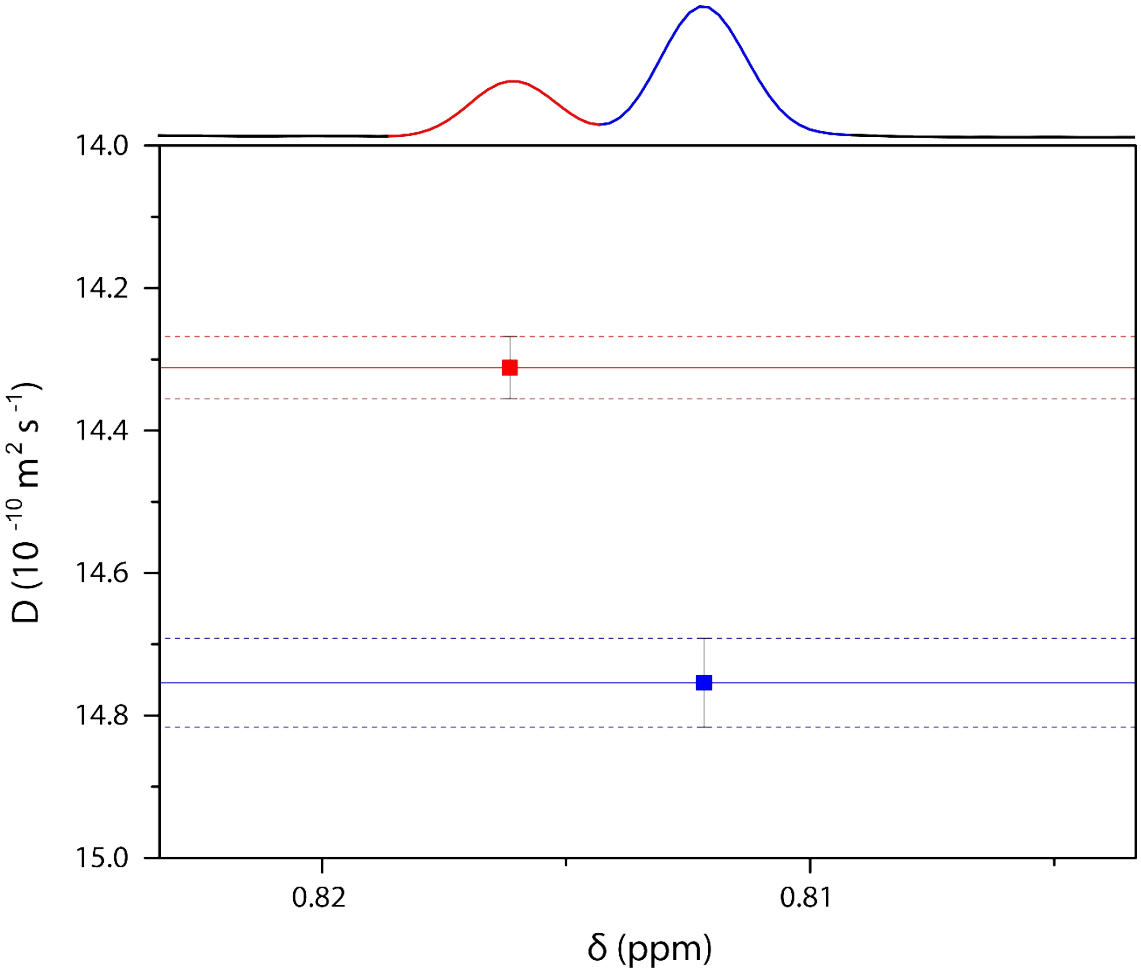


Figure S7: 500 MHz ^{1}H MAD projections, with the least attenuated 1D spectrum shown at the top, for compounds **8** and **9** with matrix **5**

Table S5: Measured ^{1}H diffusion coefficients of compounds **8** and **9** with matrix **5**

Chemical Shift	Exp Amplitude	Fit. Amplitude	Error	Diffusion coefficient[a]	Error[a]
0.81326	36.34134	36.85960	0.09496	14.84569	0.06495
0.81923	18.71638	18.91266	0.03158	14.27944	0.04111

[a] × 10^{-10} m^2s^{-1}.

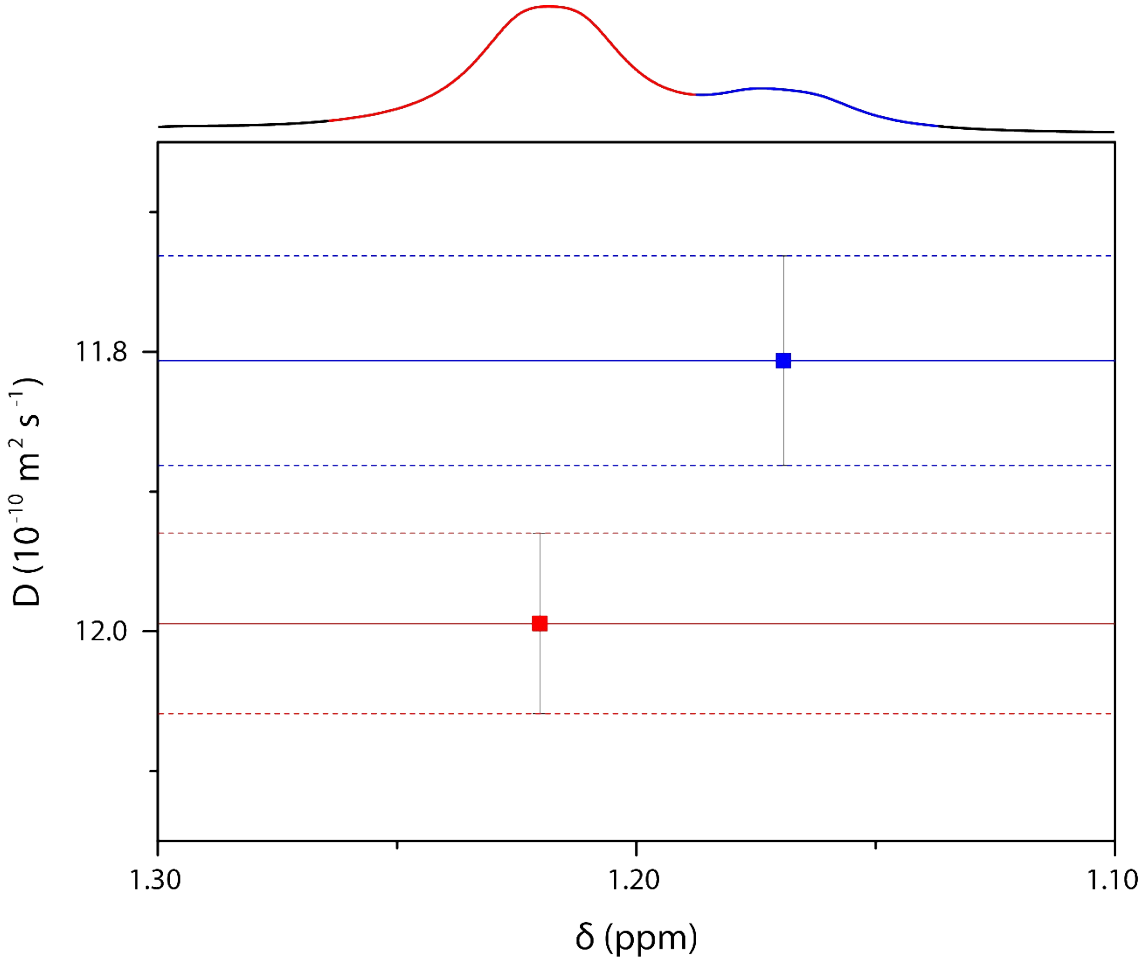


Figure S8: 500 MHz 1H MAD projections, with the least attenuated 1D spectrum shown at the top, for compound **10** with matrix **1**

Table S6: Measured ^{1}H diffusion coefficients of compound **10** with matrix **1**

Chemical Shift	Exp Amplitude	Fit. Amplitude	Error	Diffusion coefficient[a]	Error[a]
1.16924	5.65285	5.72963	0.01935	11.80636	0.07495
1.22015	18.56703	18.84099	0.05437	11.99430	0.06456

[a] × 10^{-10} m^2s^{-1}.

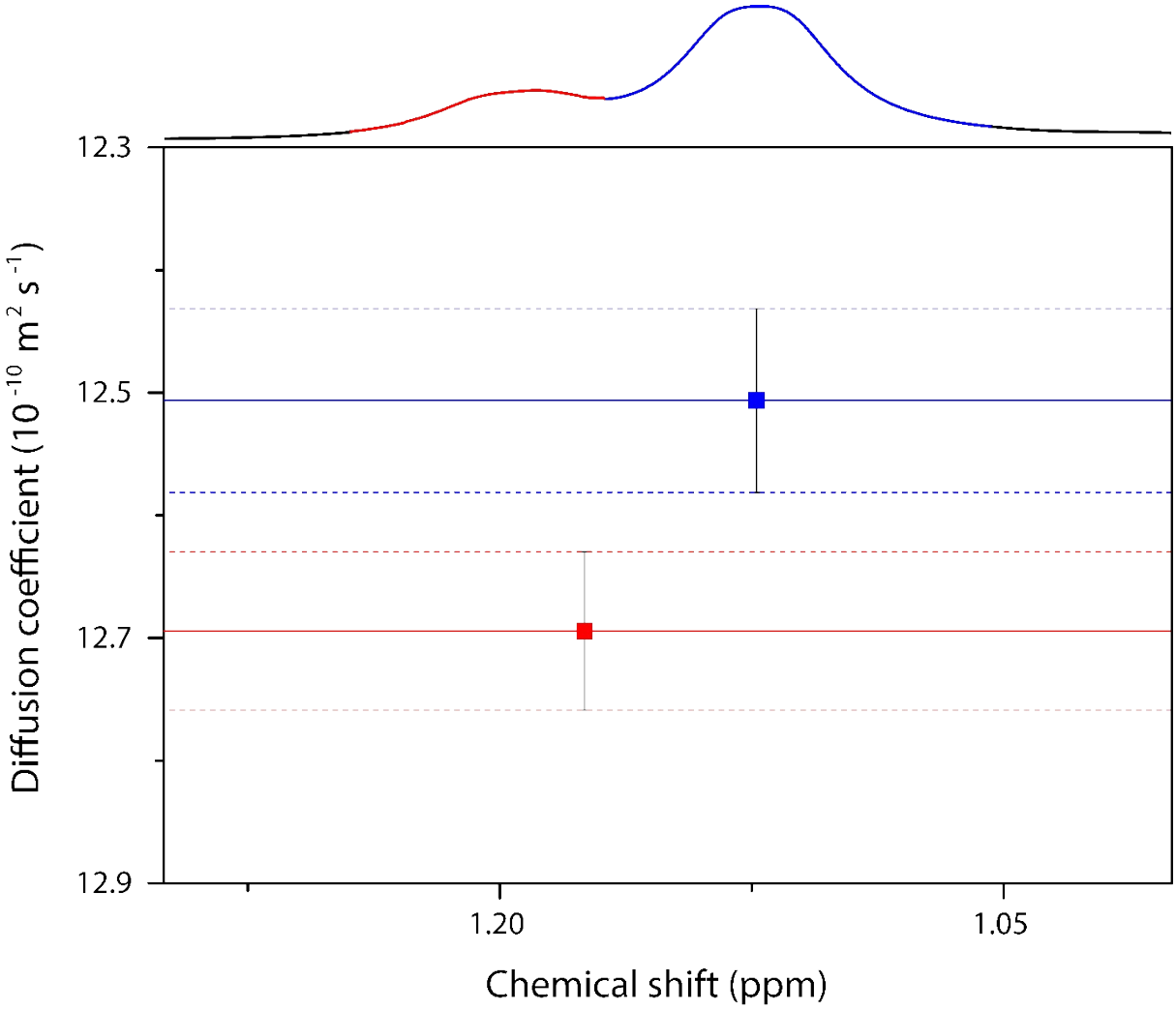


Figure S9: 500 MHz ^{1}H MAD projections, with the least attenuated 1D spectrum shown at the top, for compound **10** with matrix **2**

Table S7: Measured ^{1}H diffusion coefficients of compound **10** with matrix **2**

Chemical Shift	Exp Amplitude	Fit. Amplitude	Error	Diffusion coefficient[a]	Error[a]
1.16924	5.65285	5.72963	0.01935	12.50254	0.06941
1.22015	18.56703	18.84099	0.05437	12.69810	0.07012

[a] × 10^{-10} m^2s^{-1}.

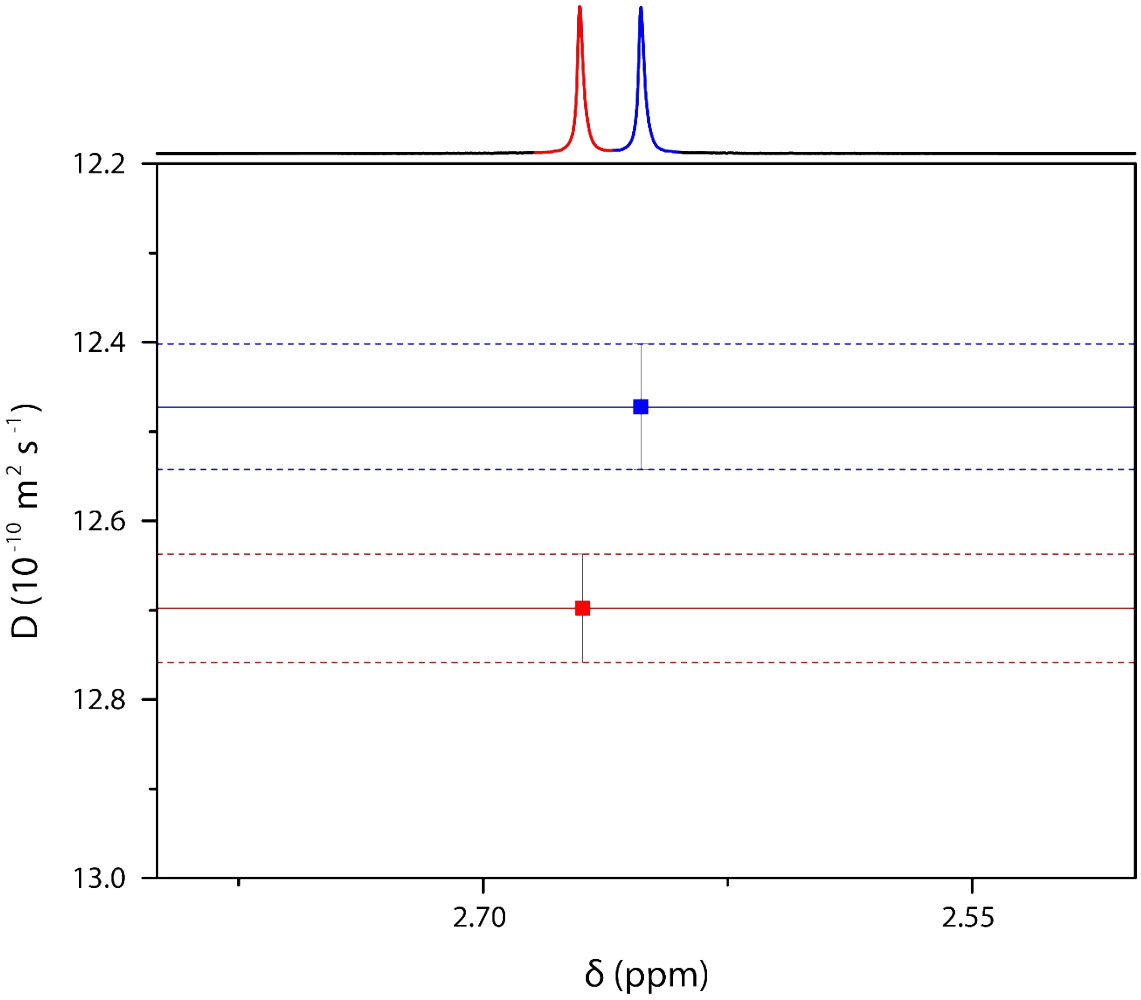


Figure S10: 500 MHz ^{1}H MAD projections, with the least attenuated 1D spectrum shown at the top, for compound **11** with matrix **1**

Table S8: Measured ^{1}H diffusion coefficients of compound **11** with matrix **1**

Chemical Shift	Exp Amplitude	Fit. Amplitude	Error	Diffusion coefficient[a]	Error[a]
2.68855	7.78752	7.90098	0.02082	12.69798	0.06071
2.70882	7.77404	7.92039	0.02442	12.47229	0.07036

[a] × 10^{-10} m^2s^{-1}.

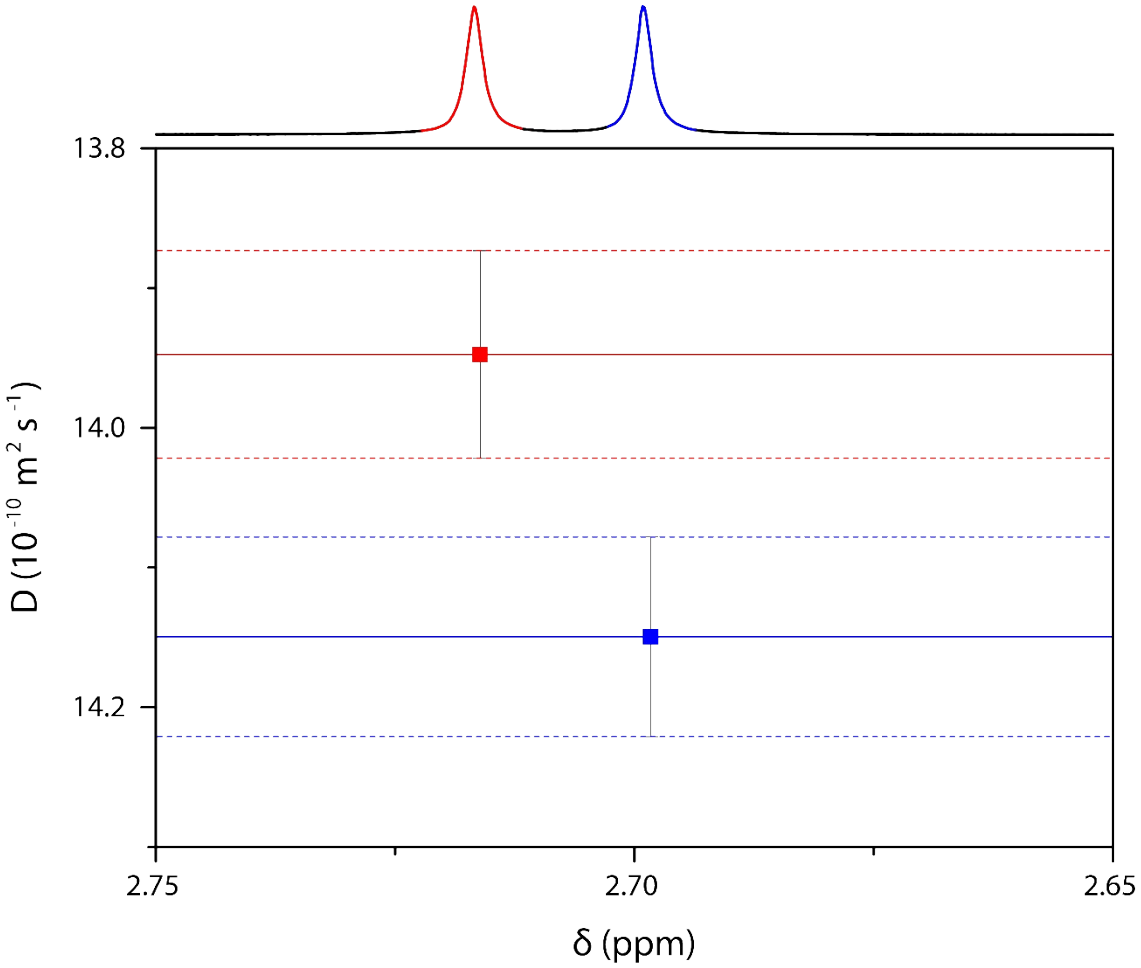


Figure S11: 500 MHz ^{1}H MAD projections, with the least attenuated 1D spectrum shown at the top, for compound **11** with matrix **2**

Table S9: Measured ^{1}H diffusion coefficients of compound **11** with matrix **2**

Chemical Shift	Exp Amplitude	Fit. Amplitude	Error	Diffusion coefficient[a]	Error[a]
2.69830	5.11227	5.19924	0.01443	14.14955	0.07146
2.71610	5.15640	5.24320	0.01524	13.94748	0.07426

[a] × 10^{-10} m^2s^{-1}.

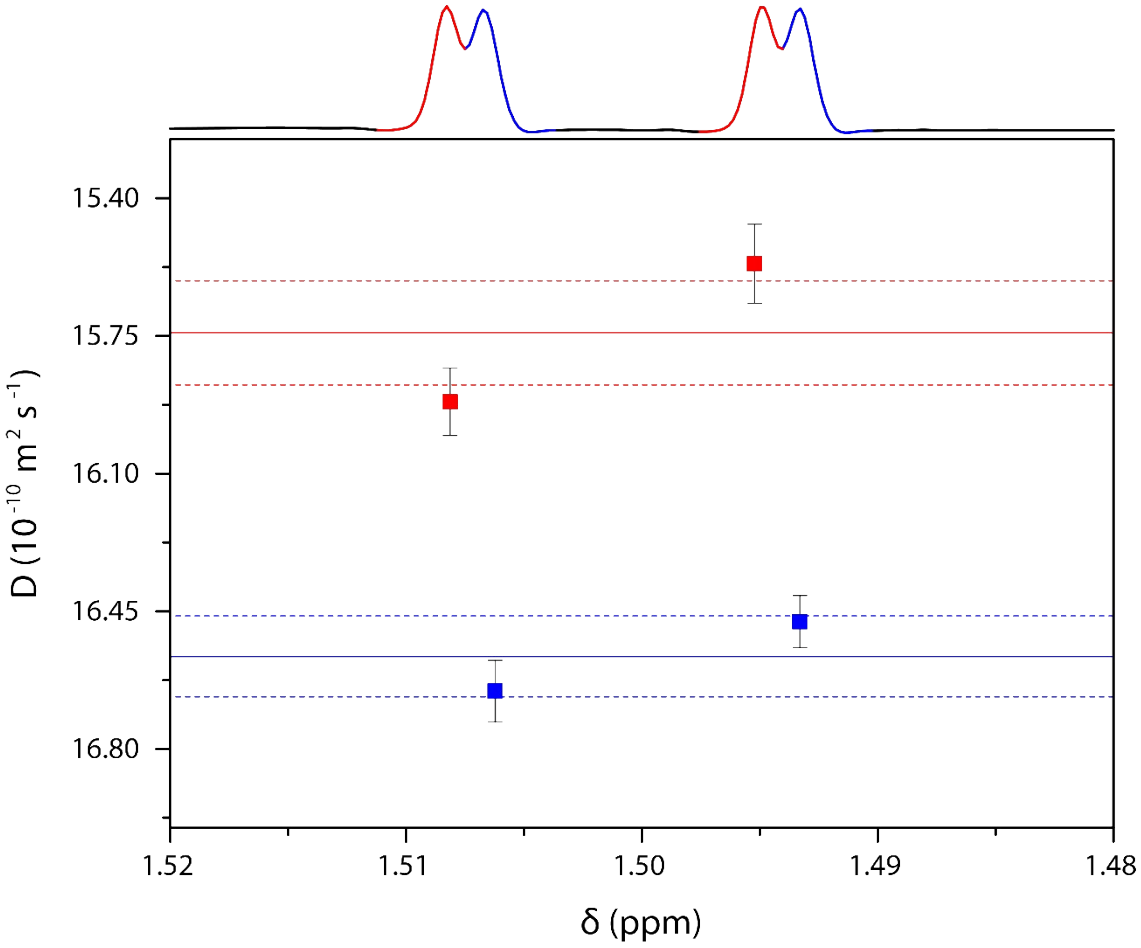


Figure S12: 500 MHz ^{1}H MAD projections, with the least attenuated 1D spectrum shown at the top, for compound **12** with matrix **1**

Table S10: Measured ^{1}H diffusion coefficients of compound **12** with matrix **1**

Chemical Shift	Exp Amplitude	Fit. Amplitude	Error	Diffusion coefficient[a]	Error[a]
1.49330	2.40316	2.42239	0.00546	16.47628	0.06654
1.49522	2.68906	2.75857	0.00970	15.56655	0.10076
1.50622	2.58960	2.59309	0.00683	16.65316	0.07823
1.50814	2.88342	2.93713	0.00867	15.91765	0.08562

[a] × 10^{-10} m^2s^{-1}.

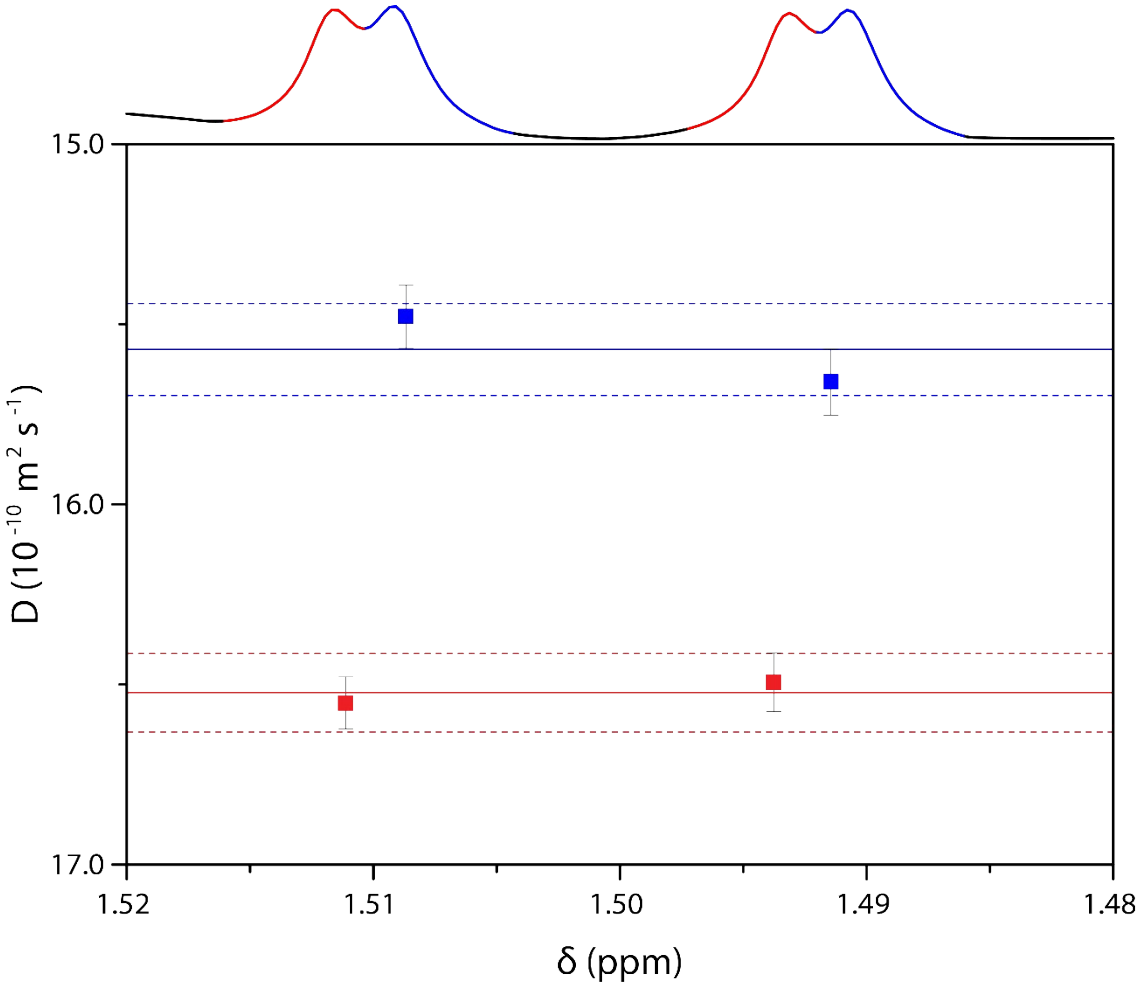


Figure S13: 500 MHz ^{1}H MAD projections, with the least attenuated 1D spectrum shown at the top, for compound **12** with matrix **2**

Table S11: Measured ^{1}H diffusion coefficients of compound **12** with matrix **2**

Chemical Shift	Exp Amplitude	Fit. Amplitude	Error	Diffusion coefficient[a]	Error[a]
1.49358	2.26943	2.30307	0.00742	15.66057	0.09257
1.49532	2.32781	2.36063	0.00650	16.49386	0.08139
1.50651	2.26684	2.29921	0.00706	15.47889	0.08775
1.50834	2.38837	2.42235	0.00592	16.55122	0.07236

[a] × 10^{-10} m^2s^{-1}.

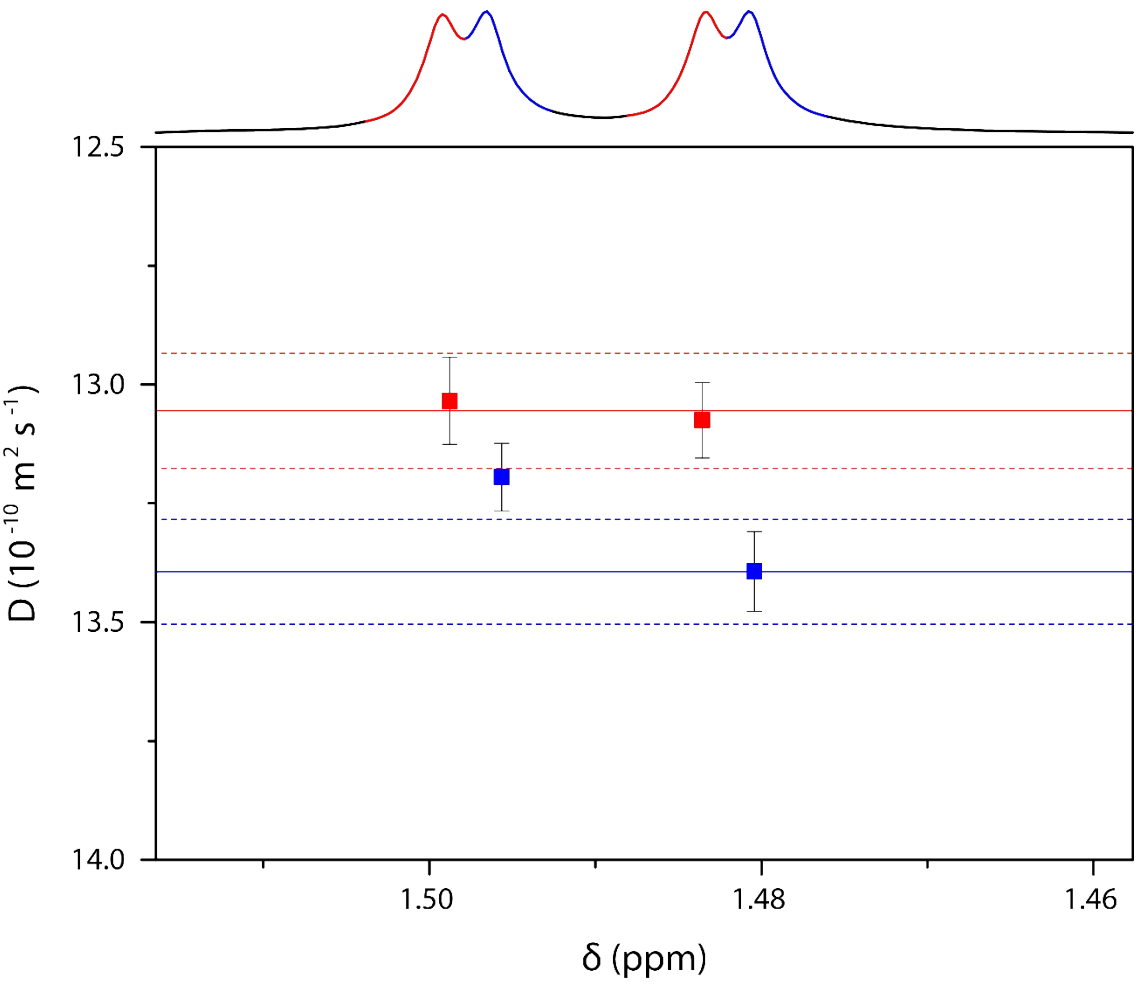


Figure S14: 500 MHz ^{1}H MAD projections, with the least attenuated 1D spectrum shown at the top, for compound **13** with matrix **1**

Table S12: Measured ^{1}H diffusion coefficients of compound **13** with matrix **1**

Chemical Shift	Exp Amplitude	Fit. Amplitude	Error	Diffusion coefficient[a]	Error[a]
1.47936	4.20744	4.22713	0.01482	13.39325	0.08389
1.48202	3.62022	3.66103	0.01240	13.07517	0.07929
1.49229	4.19327	4.22670	0.01282	13.19483	0.07137
1.49495	4.34861	4.38867	0.01720	13.03435	0.09161

[a] × 10^{-10} m^2s^{-1}.

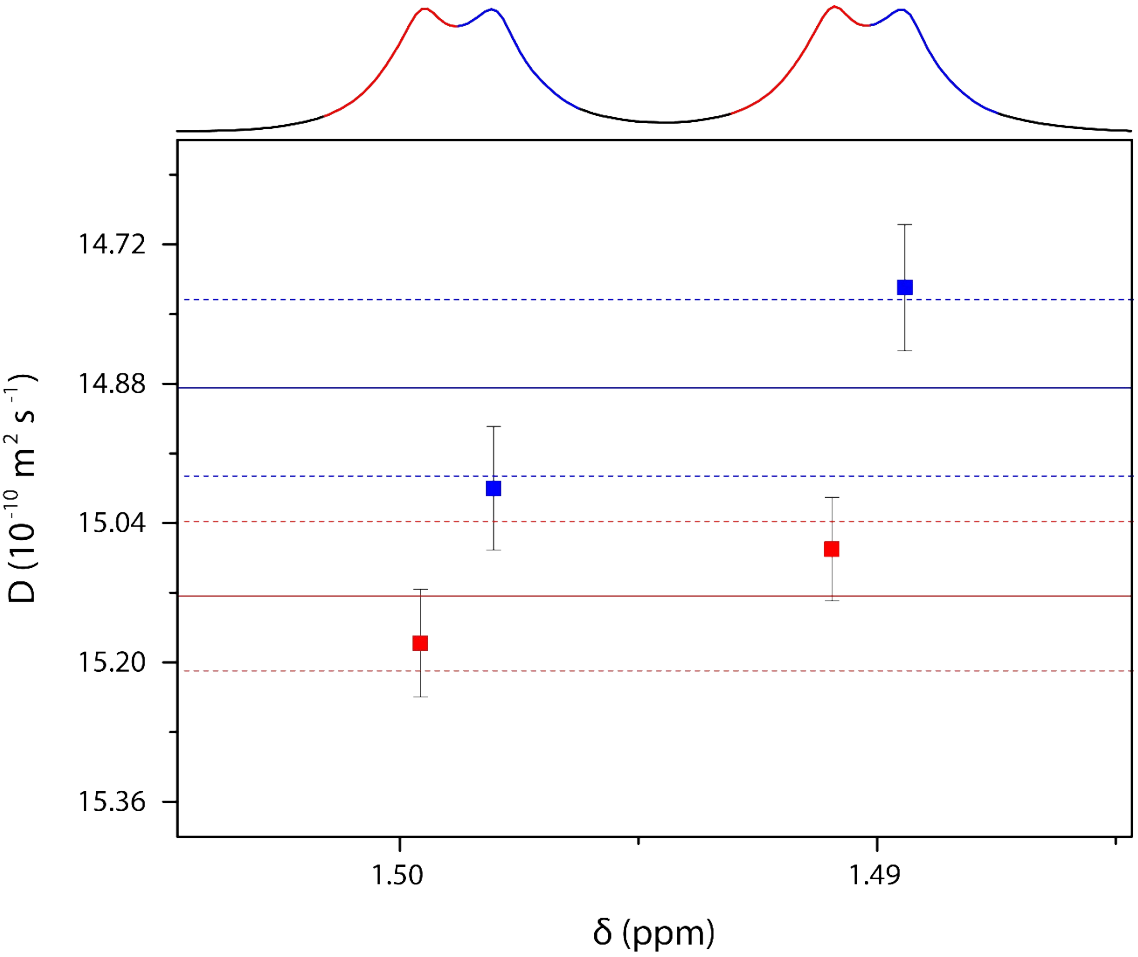


Figure S15: 500 MHz ^{1}H MAD projections, with the least attenuated 1D spectrum shown at the top, for compound **13** with matrix **2**

Table S13: Measured ^{1}H diffusion coefficients of compound **13** with matrix **2**

Chemical Shift	Exp Amplitude	Fit. Amplitude	Error	Diffusion coefficient[a]	Error[a]
1.48064	4.62294	4.72908	0.01513	13.55113	0.07639
1.48321	4.72615	4.81238	0.01336	13.74725	0.06682
1.49358	4.75583	4.85238	0.01158	13.80693	0.05760
1.49614	4.79185	4.84910	0.00708	14.30571	0.03601

[a] × 10^{-10} m^2s^{-1}.

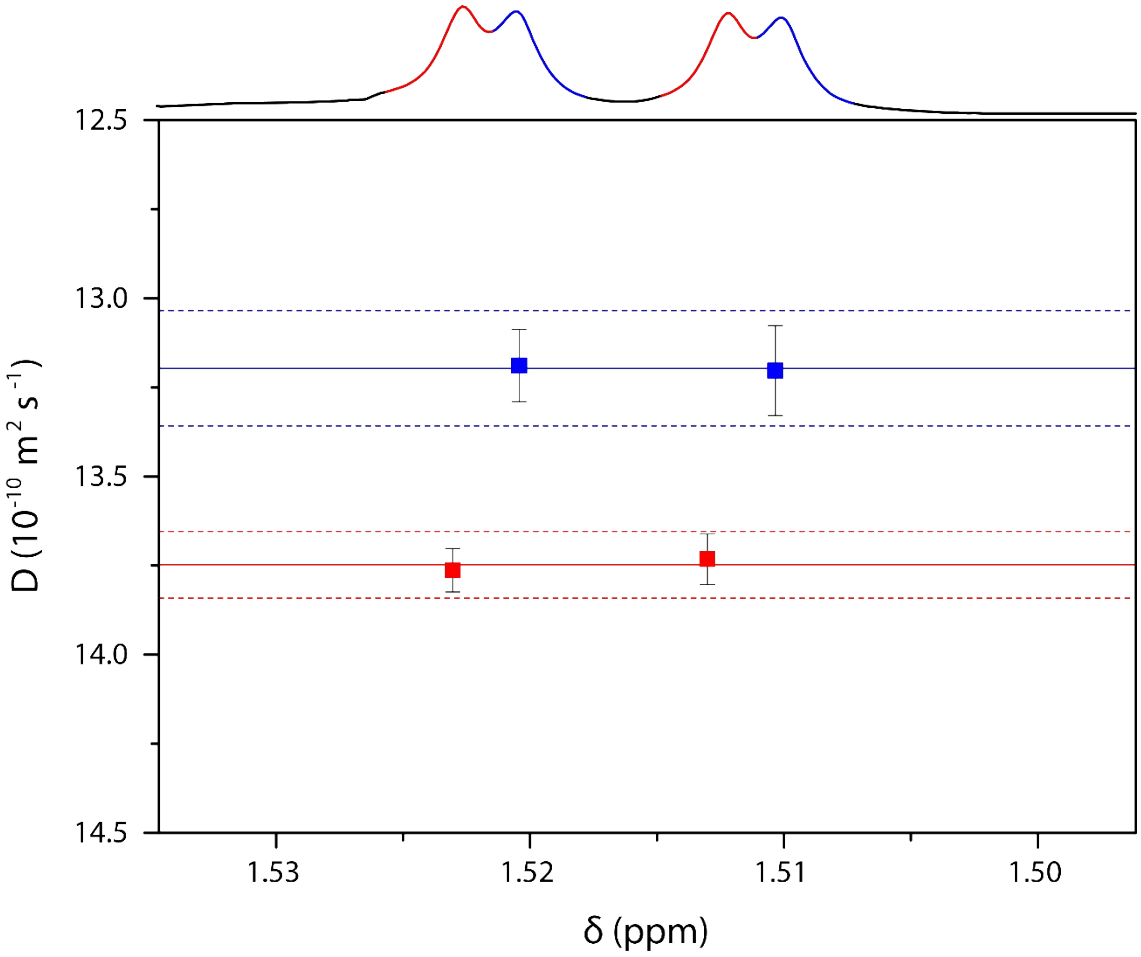


Figure S16: 500 MHz ^{1}H MAD projections, with the least attenuated 1D spectrum shown at the top, for compound **14** with matrix **1**

Table S14: Measured ^{1}H diffusion coefficients of compound **14** with matrix **1**

Chemical Shift	Exp Amplitude	Fit. Amplitude	Error	Diffusion coefficient[a]	Error[a]
1.50844	1.42912	1.47091	0.00788	13.20300	0.12615
1.51192	1.12779	1.14599	0.00338	13.73167	0.07088
1.52155	1.18386	1.20932	0.00520	13.18910	0.10105
1.52495	1.31235	1.32754	0.00336	13.76311	0.06089

[a] × 10^{-10} m^2s^{-1}.

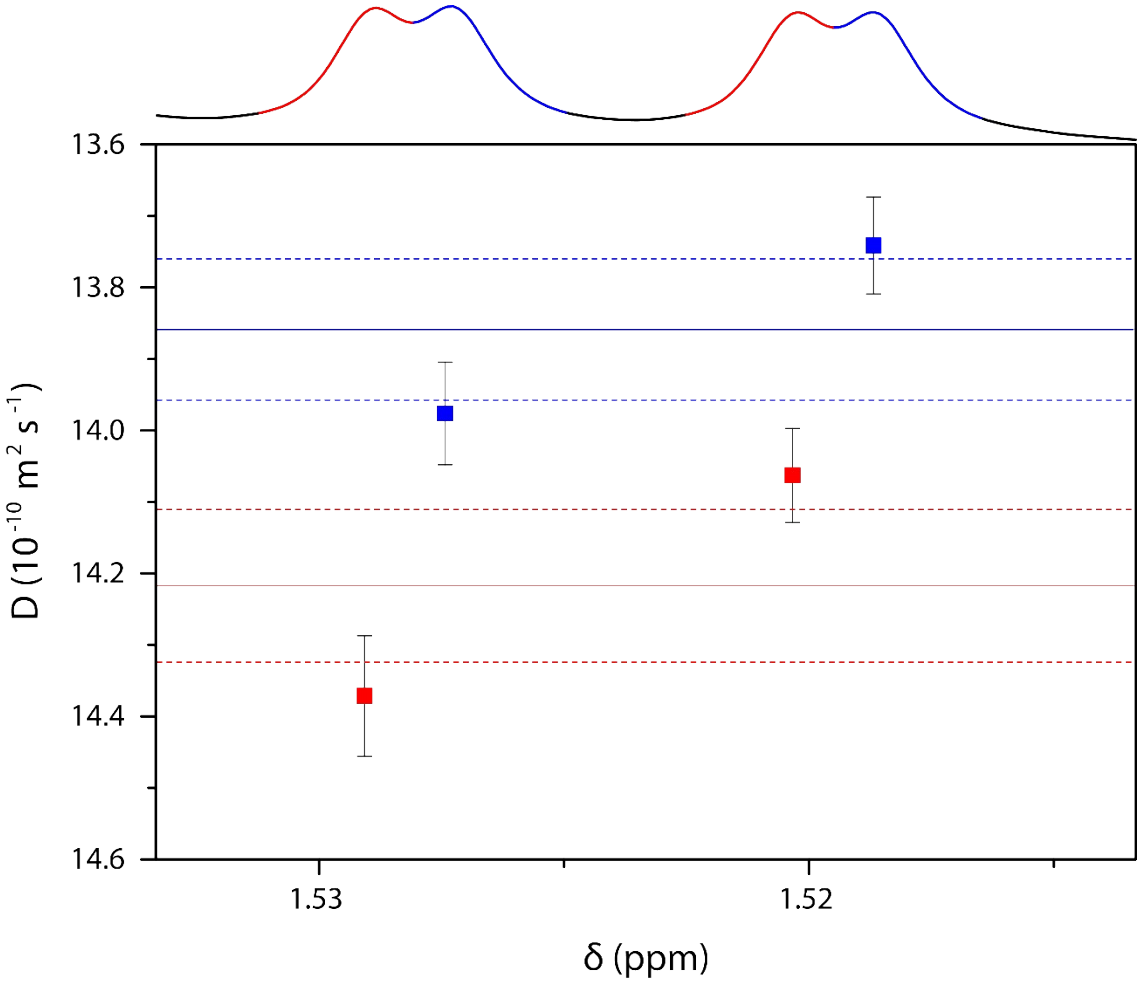


Figure S17: 500 MHz ^{1}H MAD projections, with the least attenuated 1D spectrum shown at the top, for compound **14** with matrix **2**

Table S15: Measured ^{1}H diffusion coefficients of compound **14** with matrix **2**

Chemical Shift	Exp Amplitude	Fit. Amplitude	Error	Diffusion coefficient[a]	Error[a]
1.51302	7.30044	7.39424	0.01390	13.97072	0.04568
1.51550	6.71414	6.80521	0.01145	14.04935	0.04102
1.52614	7.66127	7.70830	0.00901	14.24562	0.02875
1.52861	8.44555	8.41592	0.01807	15.27327	0.05512

[a] × 10^{-10} m^2s^{-1}.

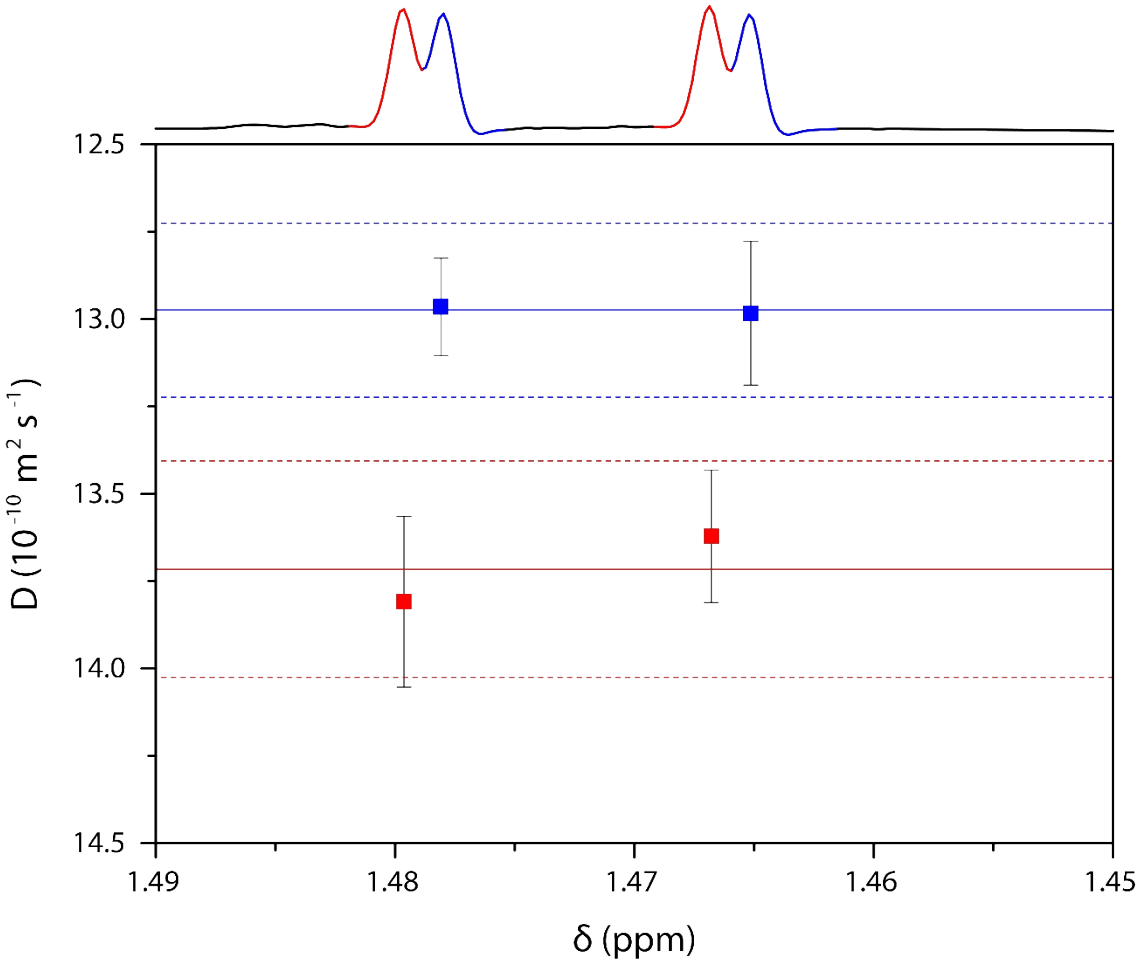


Figure S18: 500 MHz ^{1}H MAD projections, with the least attenuated 1D spectrum shown at the top, for compound **15** with matrix **1**

Table S16: Measured ^{1}H diffusion coefficients of compound **15** with matrix **1**

Chemical Shift	Exp Amplitude	Fit. Amplitude	Error	Diffusion coefficient[a]	Error[a]
1.46514	2.56758	2.50407	0.02093	12.98333	0.20595
1.46679	2.44109	2.54833	0.01915	13.62187	0.18966
1.47808	2.48768	2.46420	0.01397	12.96501	0.13958
1.47963	1.80214	1.91122	0.01839	13.80908	0.24459

[a] × 10^{-10} m^2s^{-1}.

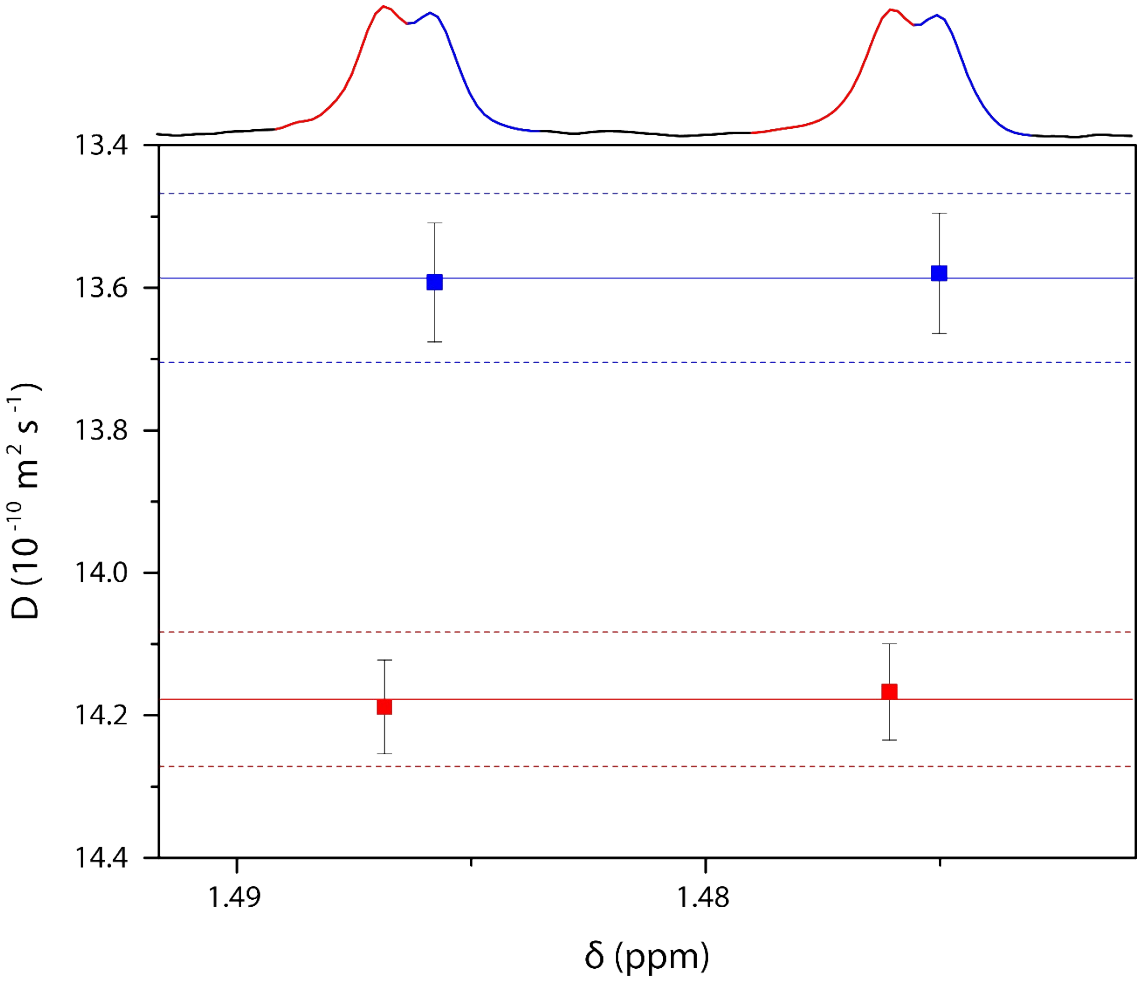


Figure S19: 500 MHz ^{1}H MAD projections, with the least attenuated 1D spectrum shown at the top, for compound **15** with matrix **2**

Table S17: Measured ^{1}H diffusion coefficients of compound **15** with matrix **2**

Chemical Shift	Exp Amplitude	Fit. Amplitude	Error	Diffusion coefficient[a]	Error[a]
1.47002	2.92785	2.99342	0.01003	13.57971	0.08442
1.47130	2.61045	2.65072	0.00696	14.16703	0.06760
1.48294	2.82113	2.88786	0.00958	13.59247	0.08363
1.48422	2.68615	2.73388	0.00697	14.18804	0.06572

[a] × 10^{-10} m^2s^{-1}.

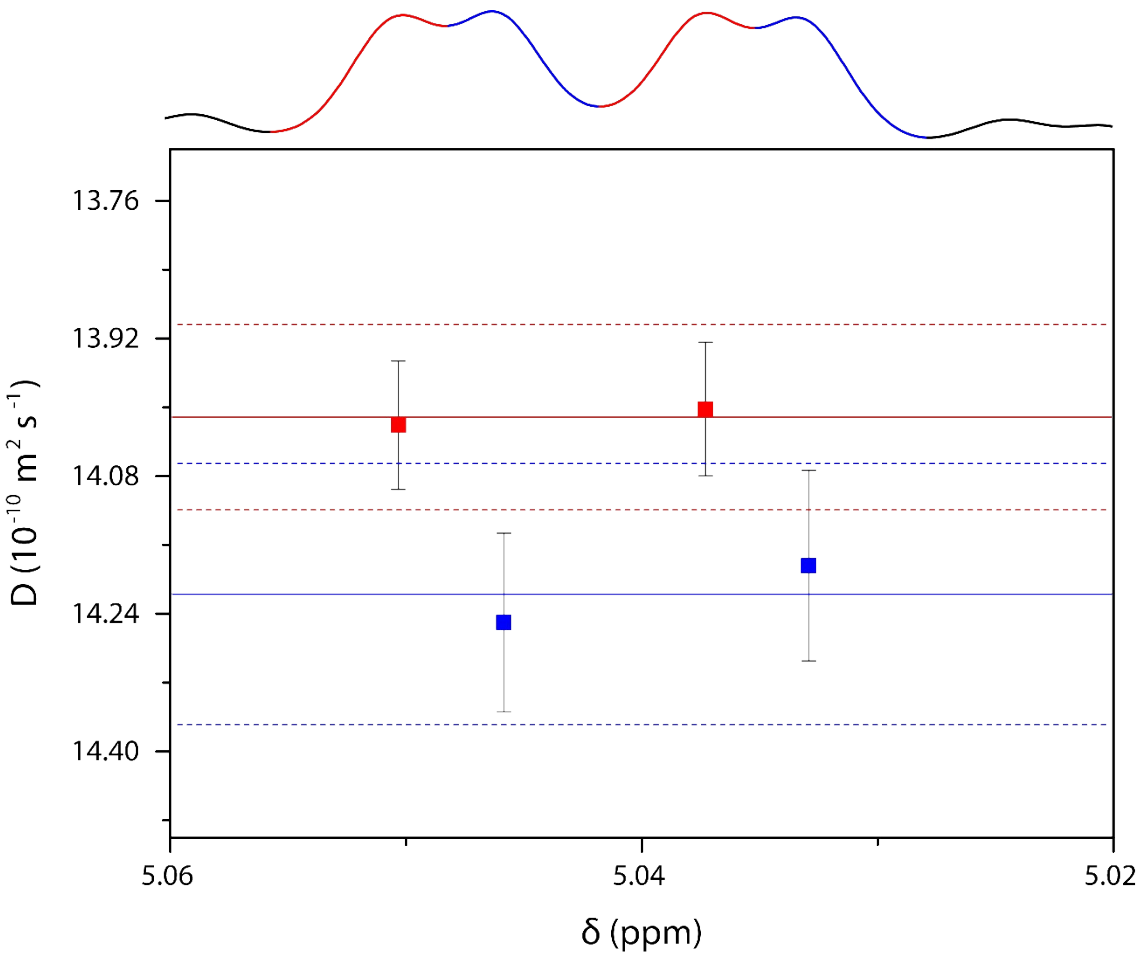


Figure S20: 500 MHz ^{1}H MAD projections, with the least attenuated 1D spectrum shown at the top, for compound **17** with matrix **3**

Table S18: Measured ^{1}H diffusion coefficients of compound **17** with matrix **3**

Chemical Shift	Exp Amplitude	Fit. Amplitude	Error	Diffusion coefficient[a]	Error[a]
5.02000	0.29319	0.30384	0.00236	14.15614	0.10343
5.02449	0.34037	0.35240	0.00243	14.02536	0.07427
5.03292	0.89992	0.92067	0.00348	14.20879	0.09666
5.03730	0.96625	0.98478	0.00269	14.02303	0.07265

[a] × 10^{-10} m^2s^{-1}.

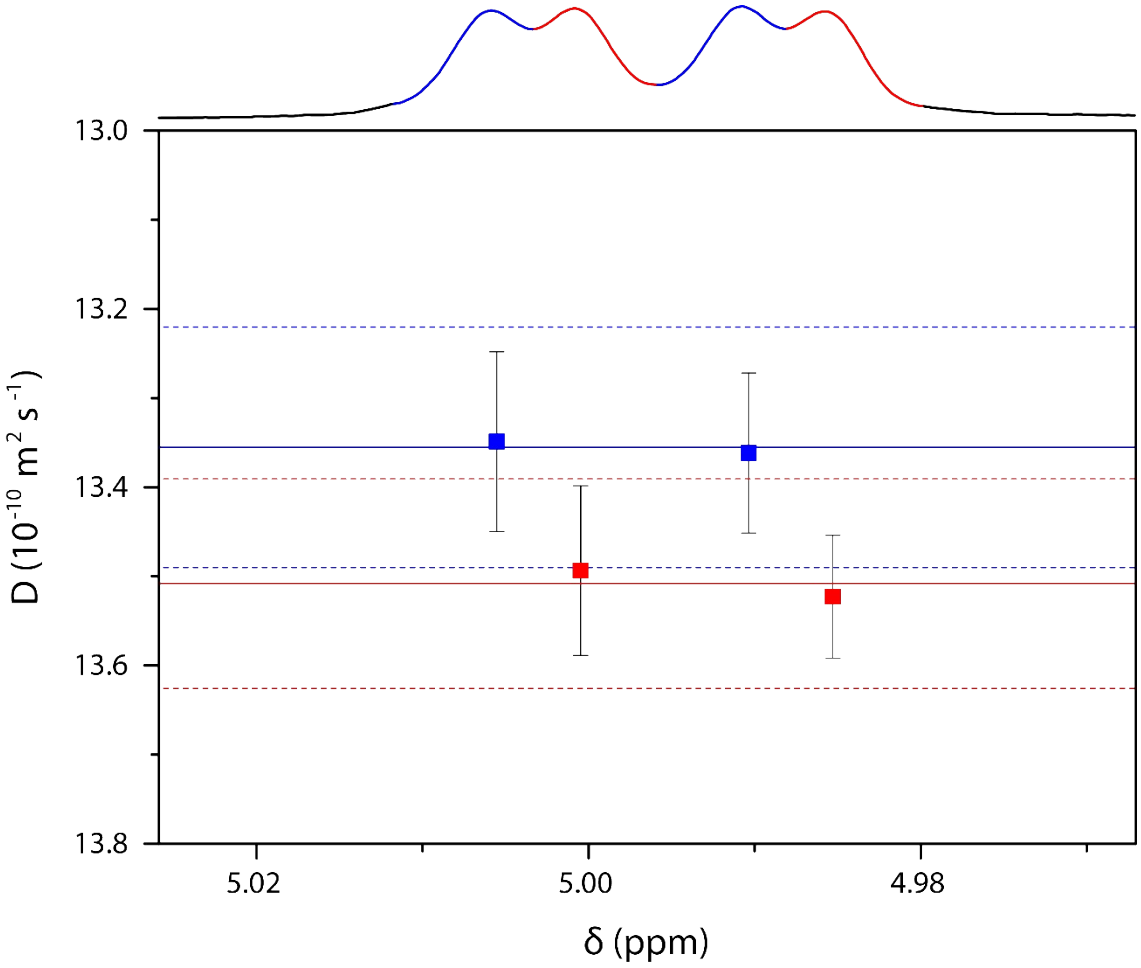


Figure S21: 500 MHz ^{1}H MAD projections, with the least attenuated 1D spectrum shown at the top, for compound **17** with matrix **4**

Table S19: Measured ^{1}H diffusion coefficients of compound **17** with matrix **4**

Chemical Shift	Exp Amplitude	Fit. Amplitude	Error	Diffusion coefficient[a]	Error[a]
5.03181	1.04642	1.05878	0.00414	13.34881	0.10062
5.03649	1.16329	1.18485	0.00435	13.49348	0.09511
5.04475	1.13104	1.15614	0.00403	13.36159	0.08983
5.04942	1.22586	1.24517	0.00331	13.52275	0.06893

[a] × 10^{-10} m^2s^{-1}.

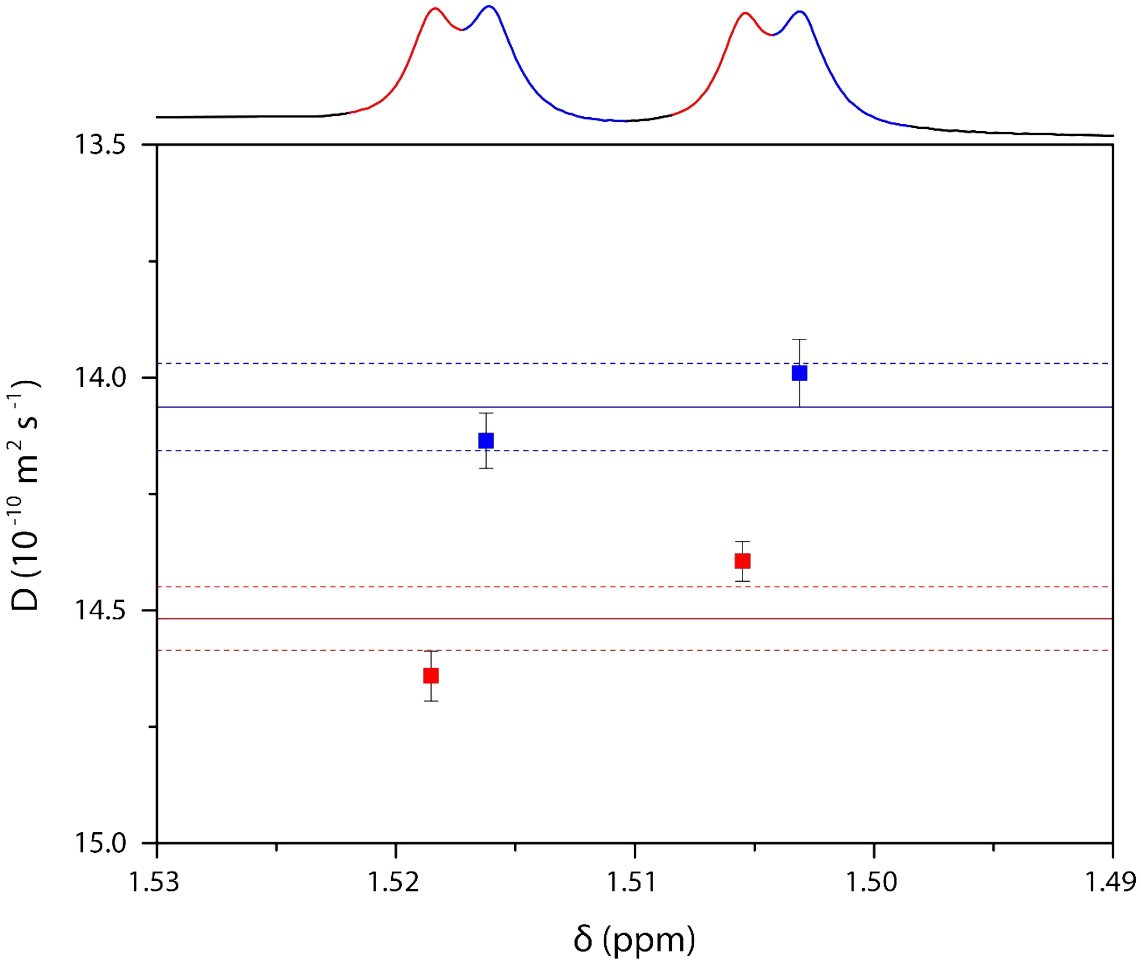


Figure S22: 500 MHz ^{1}H MAD projections, with the least attenuated 1D spectrum shown at the top, for compound **18** with matrix **1**

Table S20: Measured ^{1}H diffusion coefficients of compound **18** with matrix **1**

Chemical Shift	Exp Amplitude	Fit. Amplitude	Error	Diffusion coefficient[a]	Error[a]
1.50312	4.96584	4.98740	0.01414	13.99049	0.07254
1.50550	4.21720	4.25686	0.00698	14.39462	0.04261
1.51623	4.83496	4.88606	0.01125	14.13577	0.05924
1.51853	3.49139	3.52643	0.00720	14.64094	0.05351

[a] × 10^{-10} m^2s^{-1}.

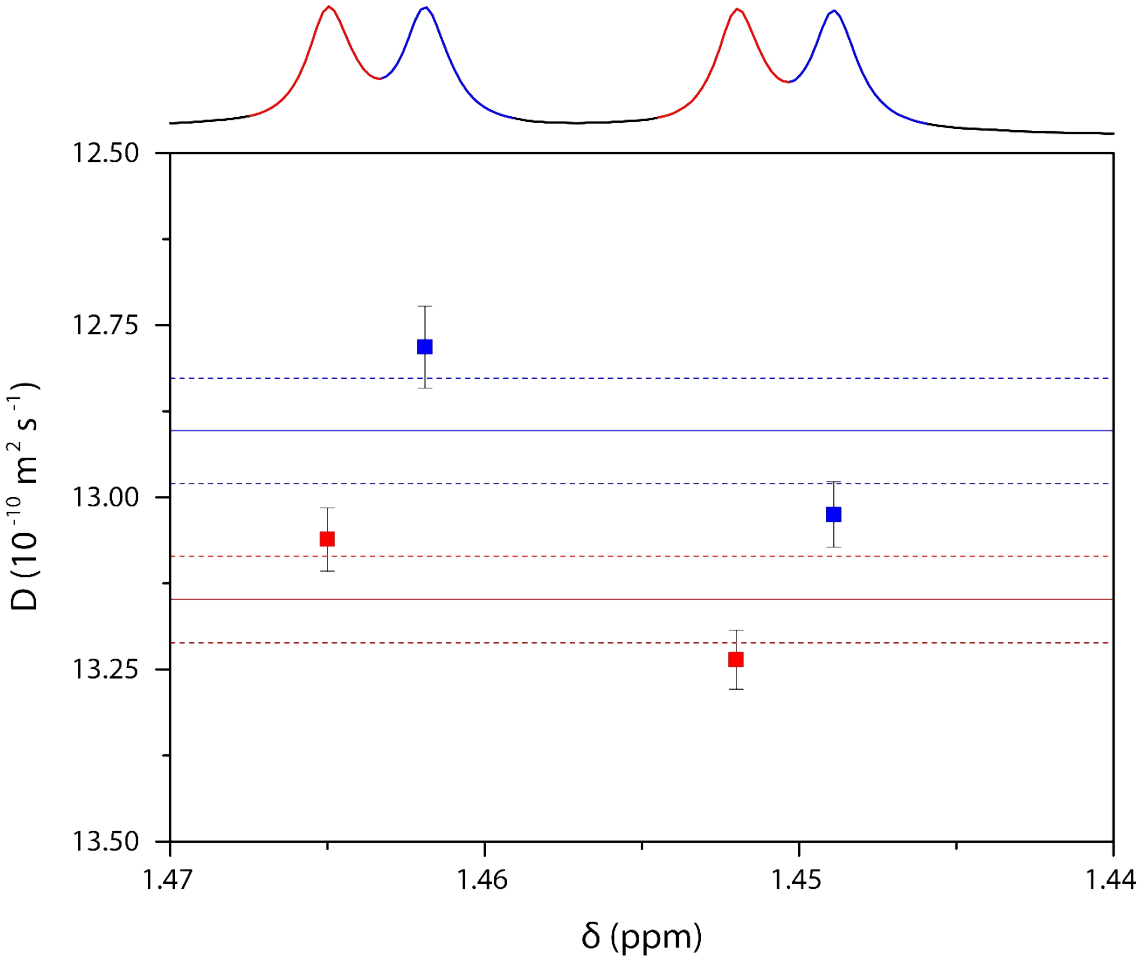


Figure S23: 500 MHz ^{1}H MAD projections, with the least attenuated 1D spectrum shown at the top, for compound **18** with matrix **2**

Table S21: Measured ^{1}H diffusion coefficients of compound **18** with matrix **2**

Chemical Shift	Exp Amplitude	Fit. Amplitude	Error	Diffusion coefficient[a]	Error[a]
1.49514	1.89598	1.91188	0.00376	13.06111	0.04606
1.49825	1.83147	1.85980	0.00479	12.78197	0.05953
1.50816	1.86166	1.87271	0.00342	13.23589	0.04299
1.51128	1.97308	1.99480	0.00407	13.02503	0.04763

[a] × 10^{-10} m^2s^{-1}.

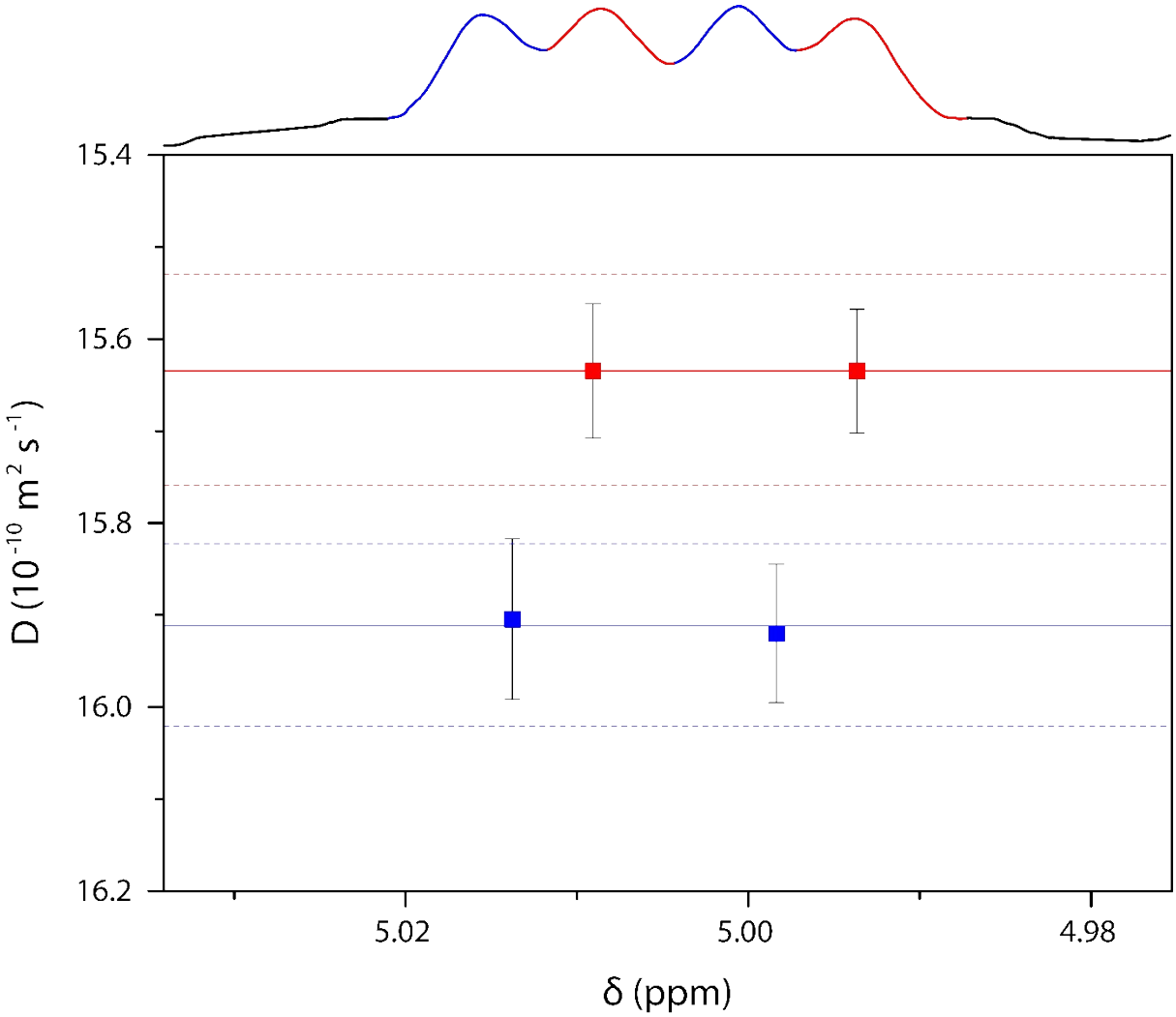


Figure S24: 500 MHz ^{1}H MAD projections, with the least attenuated 1D spectrum shown at the top, for compound **18** with matrix **3**

Table S 22: Measured ^{1}H diffusion coefficients of compound **18** with matrix **4**

Chemical Shift	Exp Amplitude	Fit. Amplitude	Error	Diffusion coefficient[a]	Error[a]
4.99012	0.85416	0.85691	0.00080	15.63215	0.08469
4.99527	0.56147	0.59321	0.00056	15.91352	0.07513
5.09654	0.86215	0.82146	0.00038	15.64598	0.08417
5.14235	0.62314	0.64785	0.00090	15.94984	0.07411

[a] × 10^{-10} m^2s^{-1}.

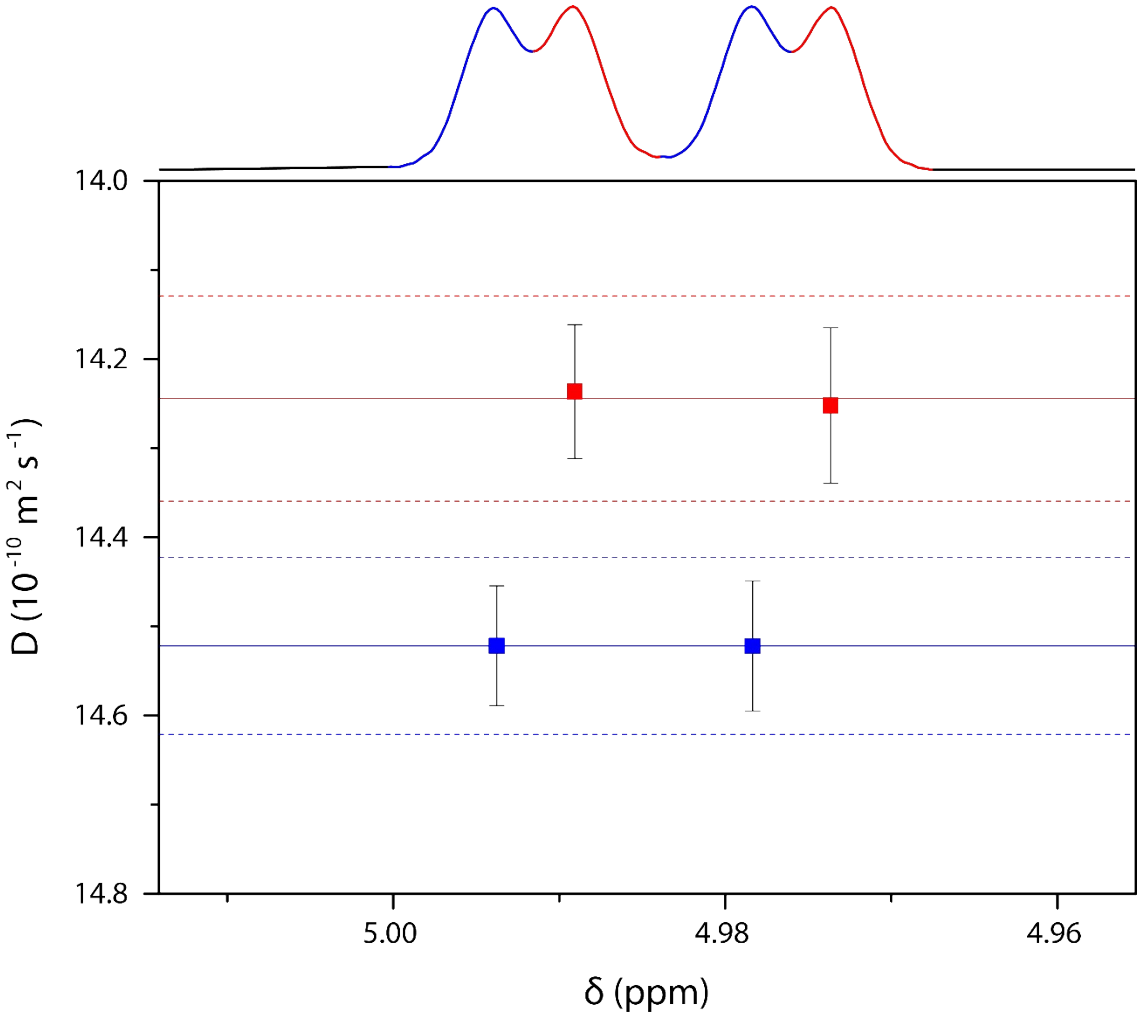


Figure S25: 500 MHz ^{1}H MAD projections, with the least attenuated 1D spectrum shown at the top, for compound **18** with matrix **4**

Table S23: Measured ^{1}H diffusion coefficients of compound **18** with matrix **4**

Chemical Shift	Exp Amplitude	Fit. Amplitude	Error	Diffusion coefficient[a]	Error[a]
4.97581	0.24624	0.25257	0.00099	14.22176	0.0672
4.97856	0.24452	0.24952	0.00072	14.53654	0.07512
4.98752	0.82929	0.84509	0.00228	14.21197	0.07307
4.99523	0.80300	0.81862	0.00250	14.52152	0.08733

[a] × 10^{-10} m^2s^{-1}.

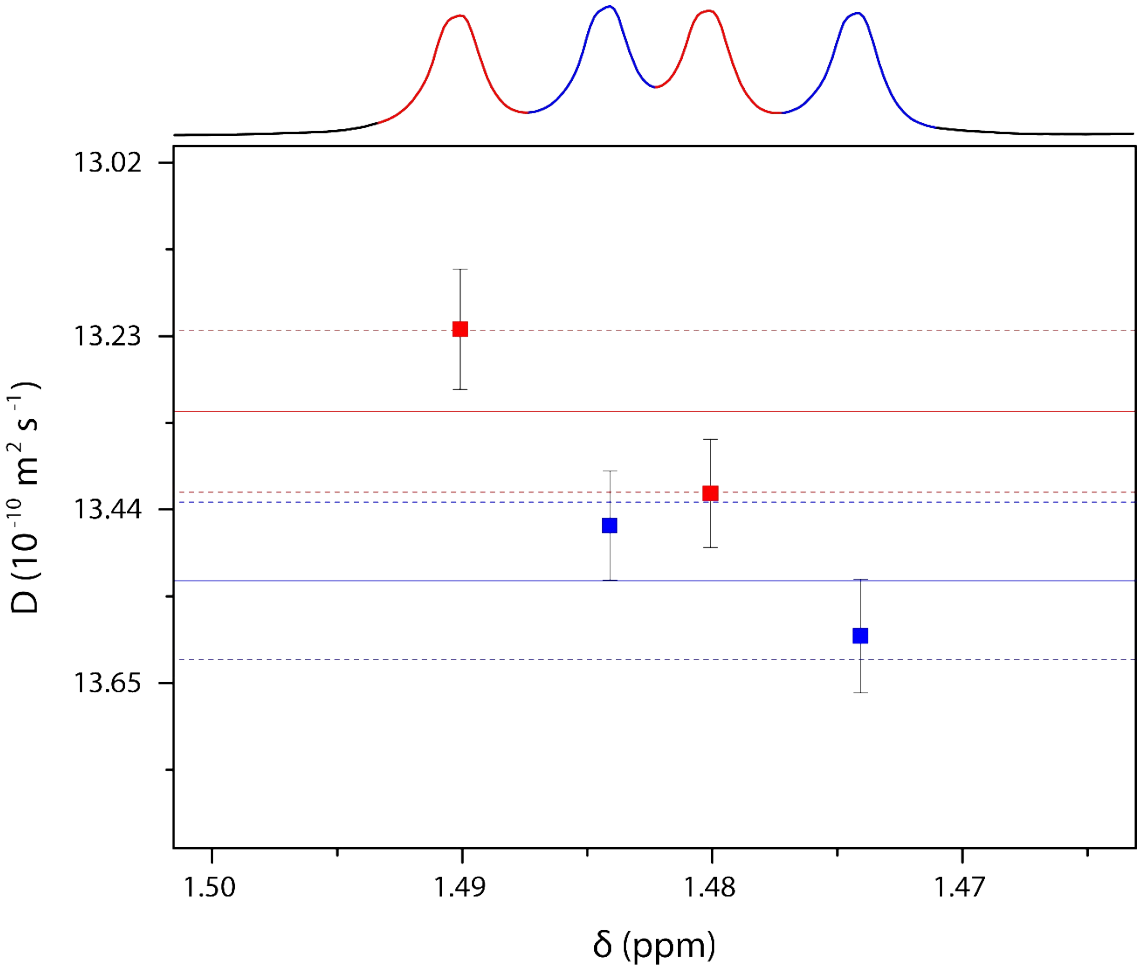


Figure S26: 500 MHz ^{1}H MAD projections, with the least attenuated 1D spectrum shown at the top, for compound **19** with matrix **5**

Table S24: Measured ^{1}H diffusion coefficients of compound **19** with matrix **5**

Chemical Shift	Exp Amplitude	Fit. Amplitude	Error	Diffusion coefficient[a]	Error[a]
1.47429	23.16102	23.57981	0.06774	13.59298	0.06873
1.48209	22.52145	22.92692	0.06327	13.42034	0.06554
1.48732	22.71900	23.13304	0.06423	13.45929	0.06605
1.49511	22.96126	23.32449	0.07205	13.22179	0.07276

[a] × 10^{-10} m^2s^{-1}.

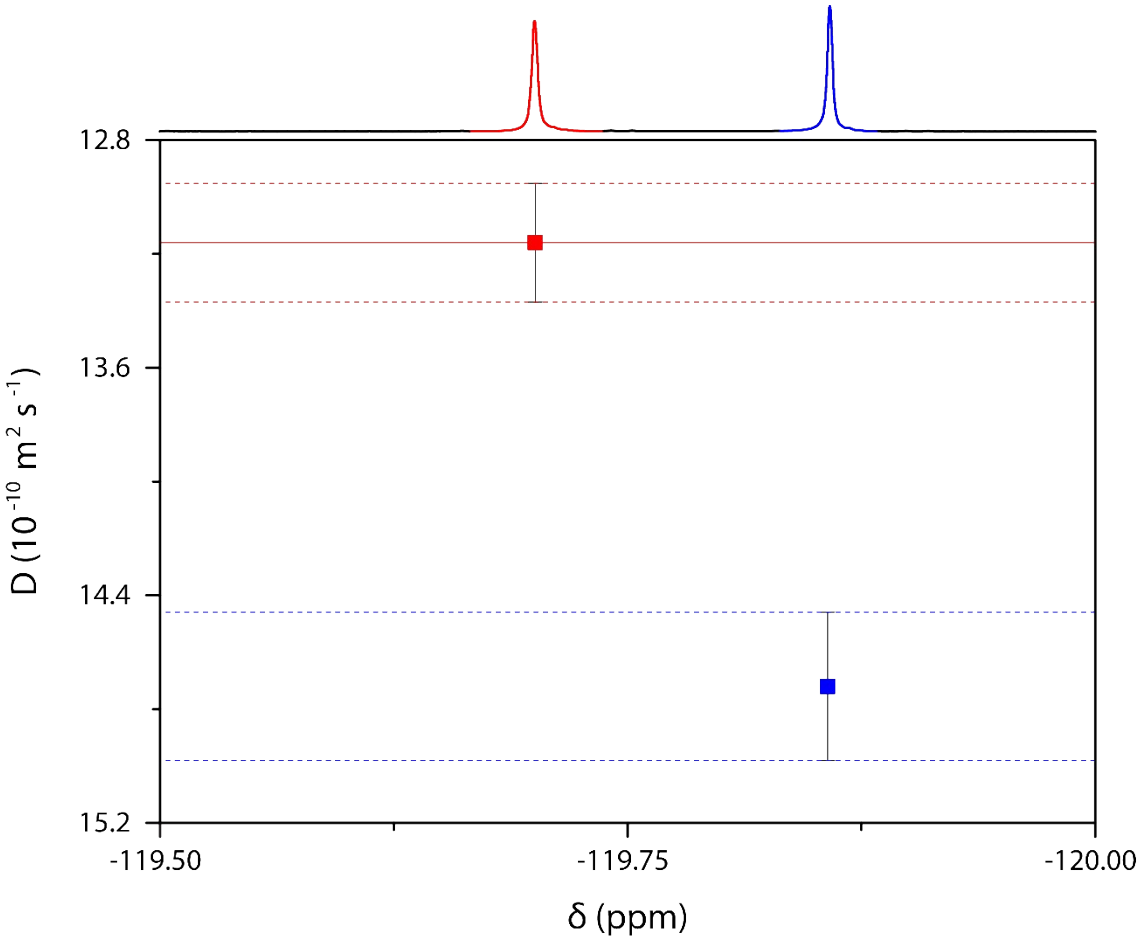


Figure S27: 470 MHz ^{19}F MAD projections, with the least attenuated 1D spectrum shown at the top, for compound **19** with matrix **5**

Table S 25: Measured ^{19}F diffusion coefficients of compound **19** with matrix **5**

Chemical Shift	Exp Amplitude	Fit. Amplitude	Error	Diffusion coefficient[a]	Error[a]
-119.85697	1069.24432	1057.99064	10.94702	14.72032	0.26089
-119.70061	1088.98827	1094.16931	9.66519	13.16042	0.20880

[a] × 10^{-10} m^2s^{-1}.

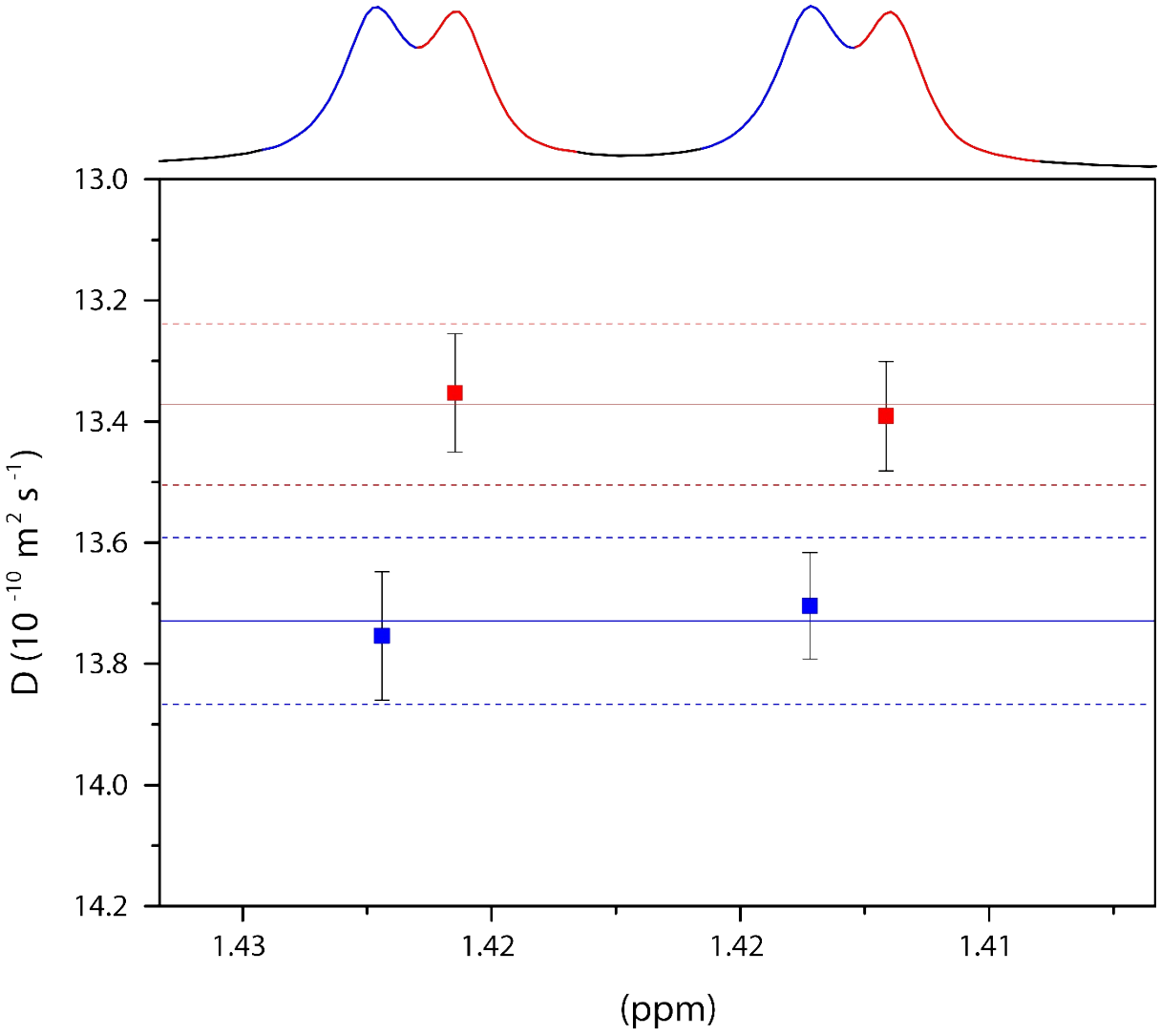


Figure S28: 500 MHz ^{1}H MAD projections, with the least attenuated 1D spectrum shown at the top, for compound **20** with matrix **1**

Table S26: Measured ^{1}H diffusion coefficients of compound **20** with matrix **1**

Chemical Shift	Exp Amplitude	Fit. Amplitude	Error	Diffusion coefficient[a]	Error[a]
1.46848	2.24693	2.29562	0.01031	13.75392	0.10579
1.46998	1.90696	1.94642	0.00814	13.35288	0.09773
1.48142	2.22216	2.25608	0.00836	13.70422	0.08786
1.48291	1.91028	1.93584	0.00741	13.59103	0.09031

[a] × 10^{-10} m^2s^{-1}.

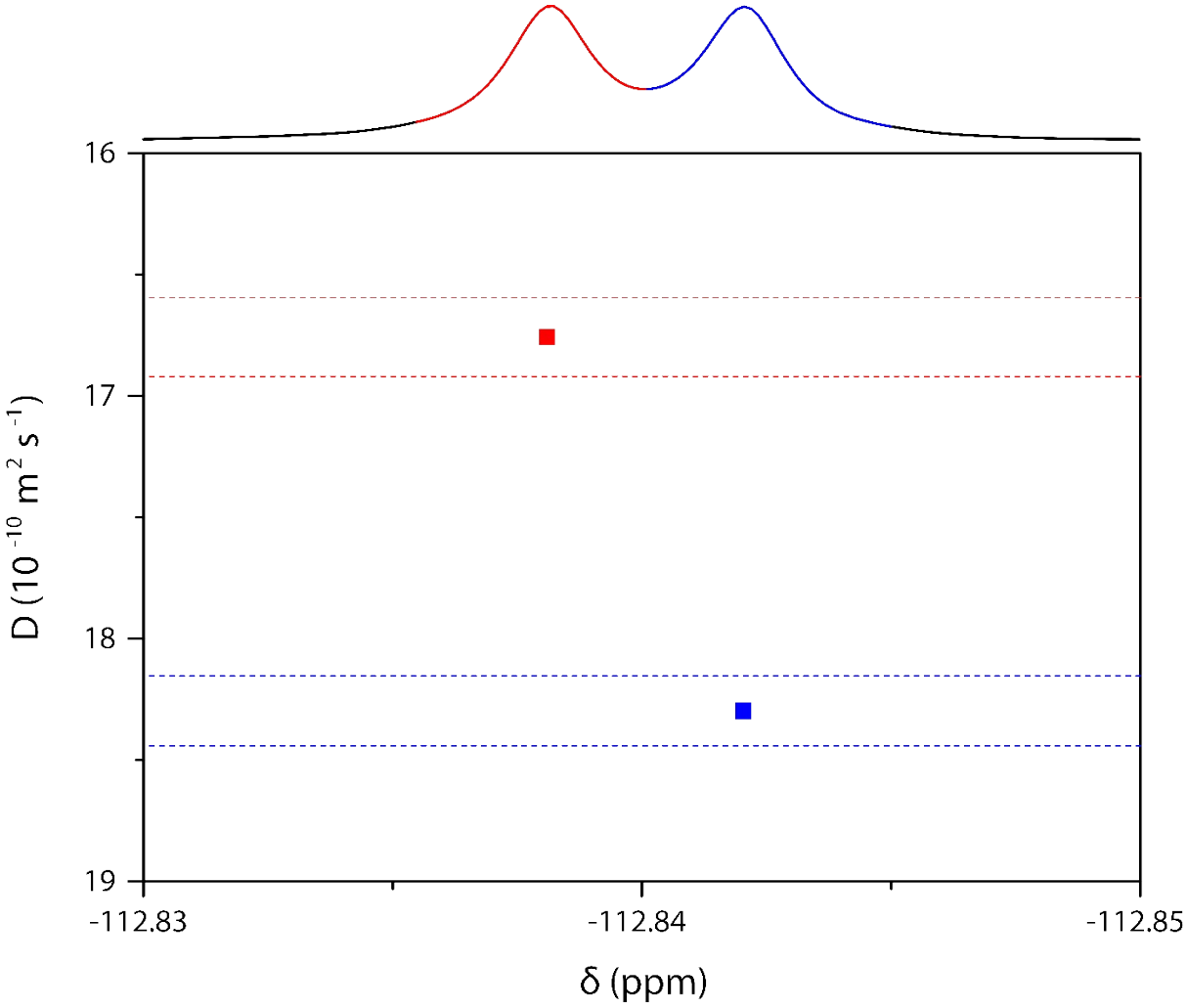


Figure S 29: 470 MHz ^{19}F MAD projections, with the least attenuated 1D spectrum shown at the top, for compound **20** with matrix **1**

Table S27: Measured ^{19}F diffusion coefficients of compound **20** with matrix **1**

Chemical Shift	Exp Amplitude	Fit. Amplitude	Error	Diffusion coefficient[a]	Error[a]
-112.84259	58.65415	51.23681	0.30214	18.01147	0.19821
-112.83985	56.36541	54.65418	0.56898	16.97952	0.24896

[a] × 10^{-10} m^2s^{-1}.

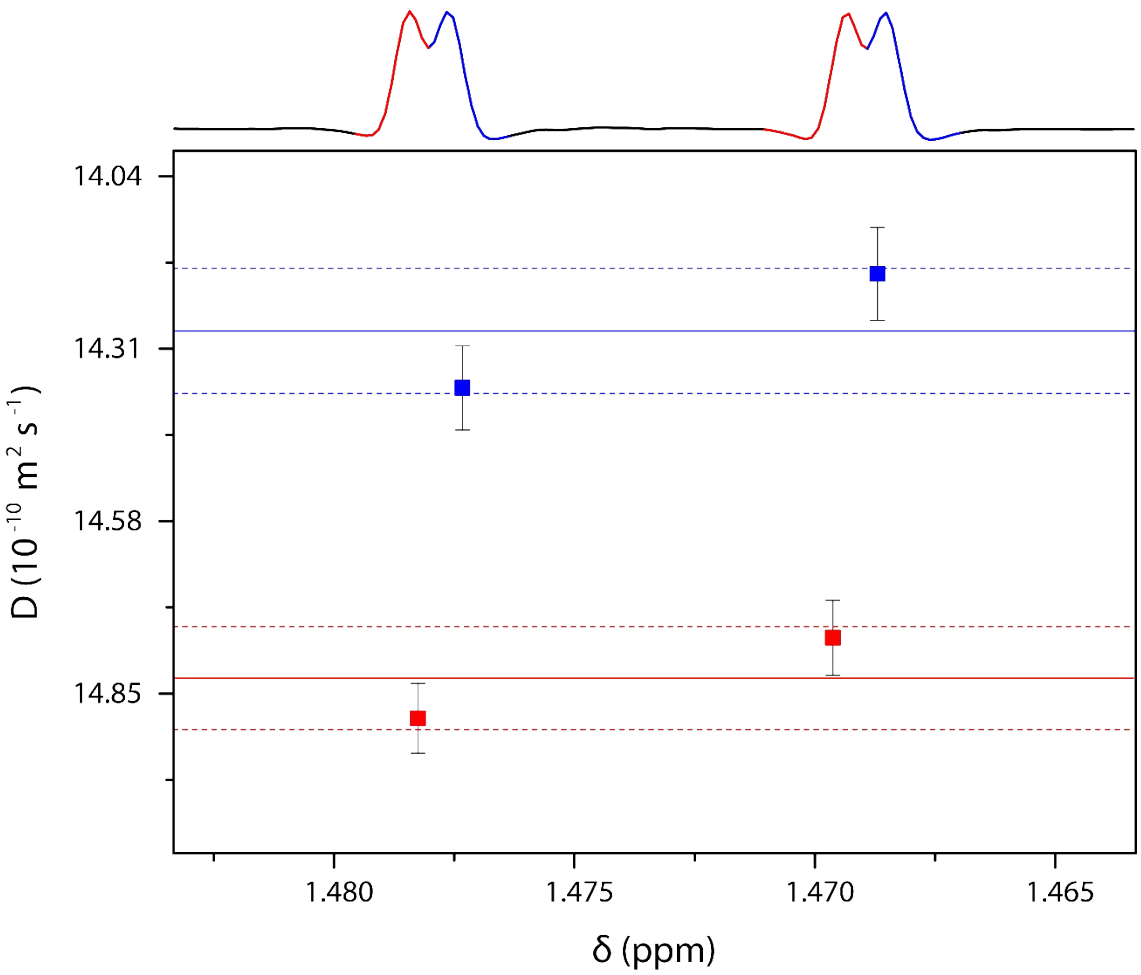


Figure S30: 500 MHz ^{1}H MAD projections, with the least attenuated 1D spectrum shown at the top, for compound **20** with matrix **2**

Table S28: Measured ^{1}H diffusion coefficients of compound **20** with matrix **2**

Chemical Shift	Exp Amplitude	Fit. Amplitude	Error	Diffusion coefficient[a]	Error[a]
1.46805	1.77473	1.81001	0.00544	14.19300	0.07283
1.46944	1.40784	1.42935	0.00339	14.76259	0.05880
1.48099	1.64515	1.67421	0.00453	14.37115	0.06606
1.48238	1.43106	1.45034	0.00318	14.88882	0.05470

[a] × 10^{-10} m^2s^{-1}.

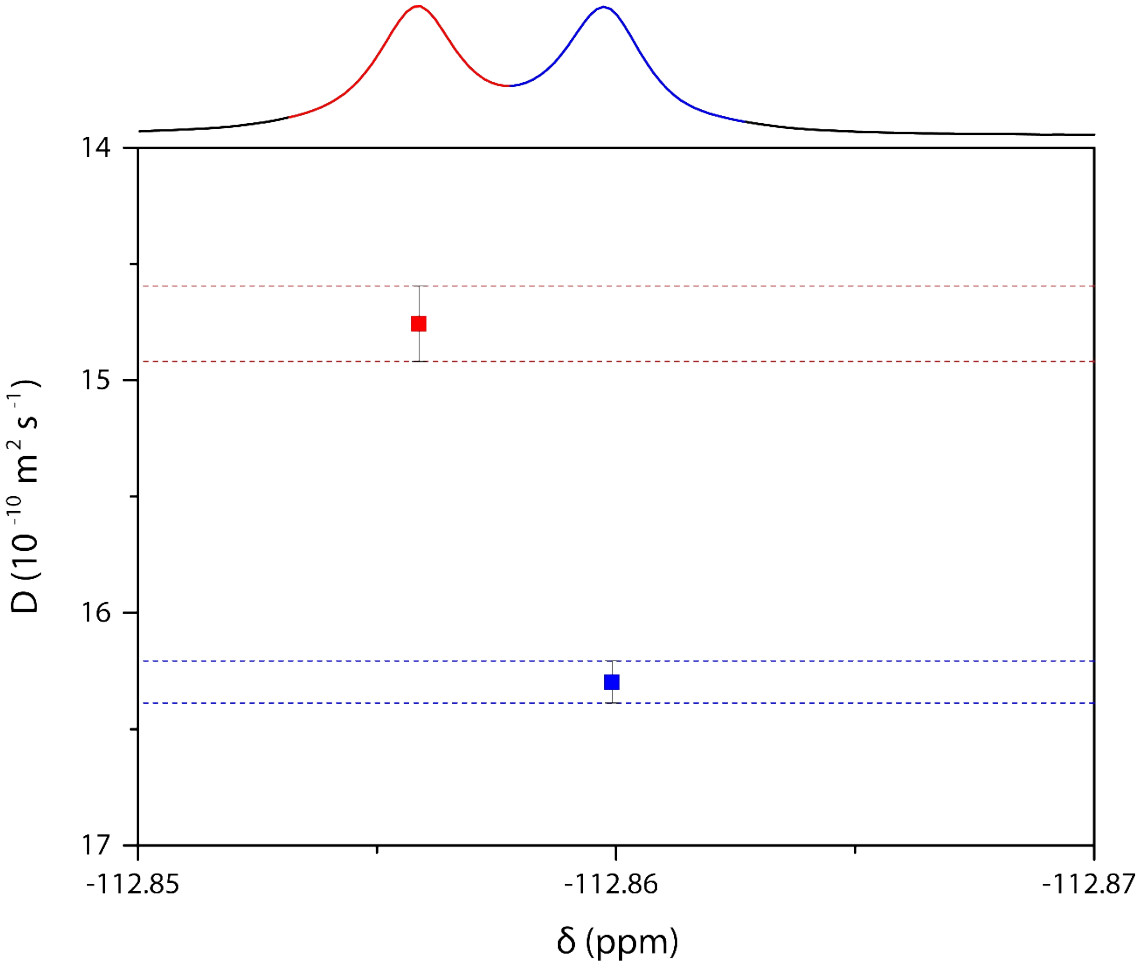


Figure S31: 470 MHz ^{19}F MAD projections, with the least attenuated 1D spectrum shown at the top, for compound **20** with matrix **2**

Table S29: Measured ^{19}F diffusion coefficients of compound **20** with matrix **2**

Chemical Shift	Exp Amplitude	Fit. Amplitude	Error	Diffusion coefficient[a]	Error[a]
-112.85992	57.22152	57.49906	0.16705	16.29726	0.09054
-112.85588	55.73456	57.28784	0.31248	14.75697	0.16252

[a] × 10^{-10} m^2s^{-1}.

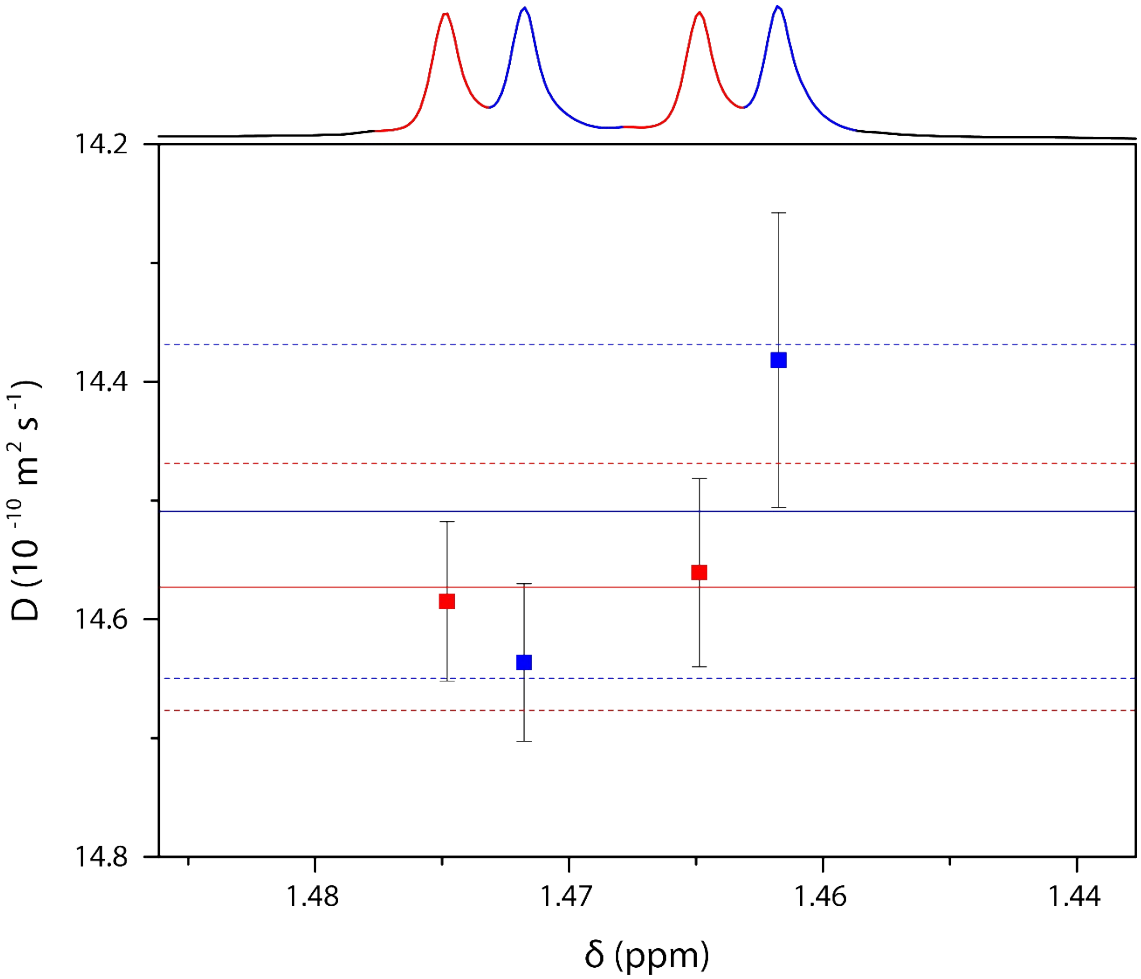


Figure S32: 500 MHz ^{1}H MAD projections, with the least attenuated 1D spectrum shown at the top, for compound **20** with matrix **5**

Table S30: Measured ^{1}H diffusion coefficients of compound **20** with matrix **5**

Chemical Shift	Exp Amplitude	Fit. Amplitude	Error	Diffusion coefficient[a]	Error[a]
1.45827	40.41234	40.97098	0.19588	14.38181	0.12414
1.46233	38.41639	39.16120	0.11879	14.56064	0.07929
1.47130	40.48803	41.22221	0.10428	14.63634	0.06631
1.47525	38.38826	39.32021	0.10092	14.58485	0.06716

[a] × 10^{-10} m^2s^{-1}.

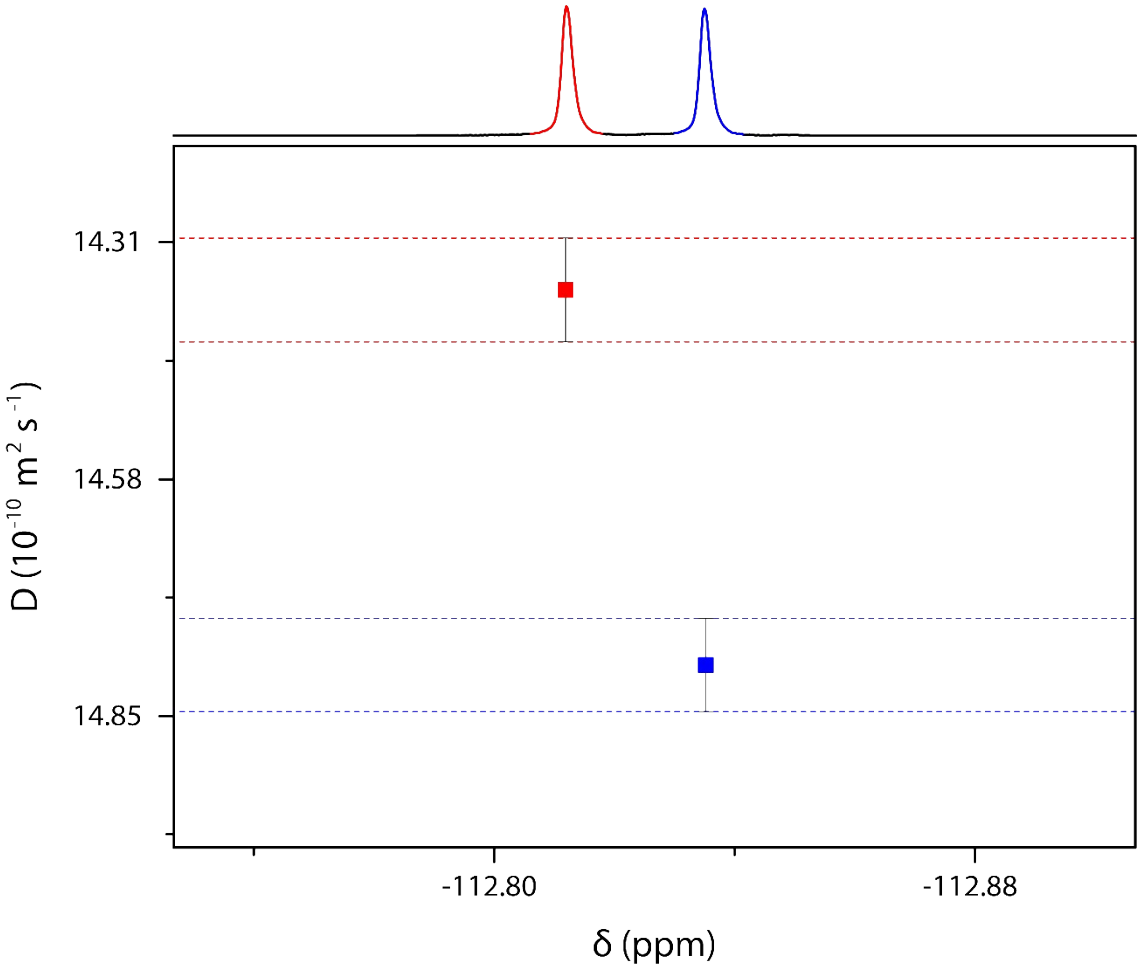


Figure S33: 500 MHz ^{1}H MAD projections, with the least attenuated 1D spectrum shown at the top, for compound **20** with matrix **5**

Table S31: Measured ^{1}H diffusion coefficients of compound **20** with matrix **5**

Chemical Shift	Exp Amplitude	Fit. Amplitude	Error	Diffusion coefficient[a]	Error[a]
-112.83303	189.09478	191.04179	0.30596	14.79189	0.05326
-112.81115	218.87327	221.62622	0.39854	14.36425	0.05918

[a] × 10^{-10} m^2s^{-1}.

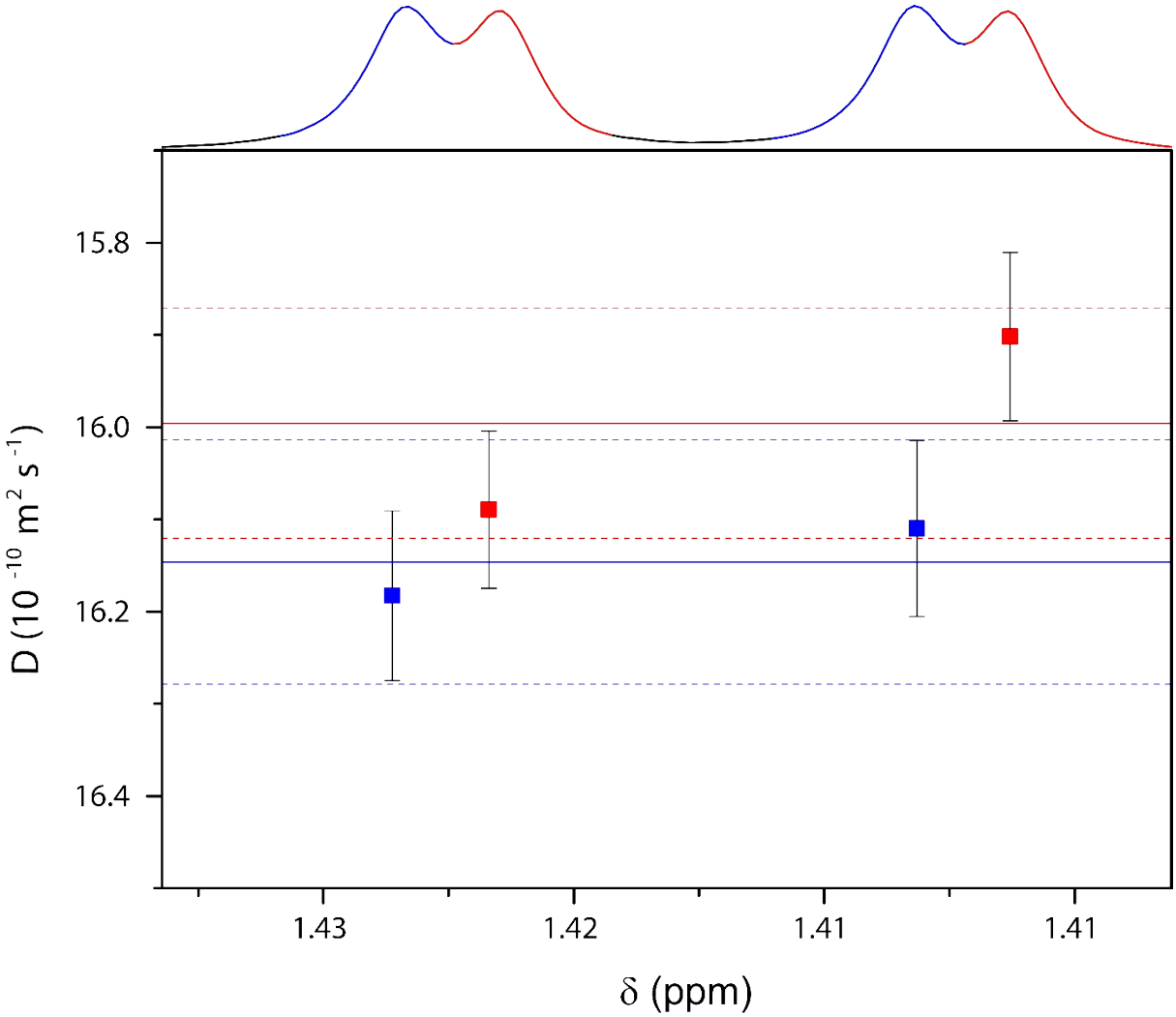
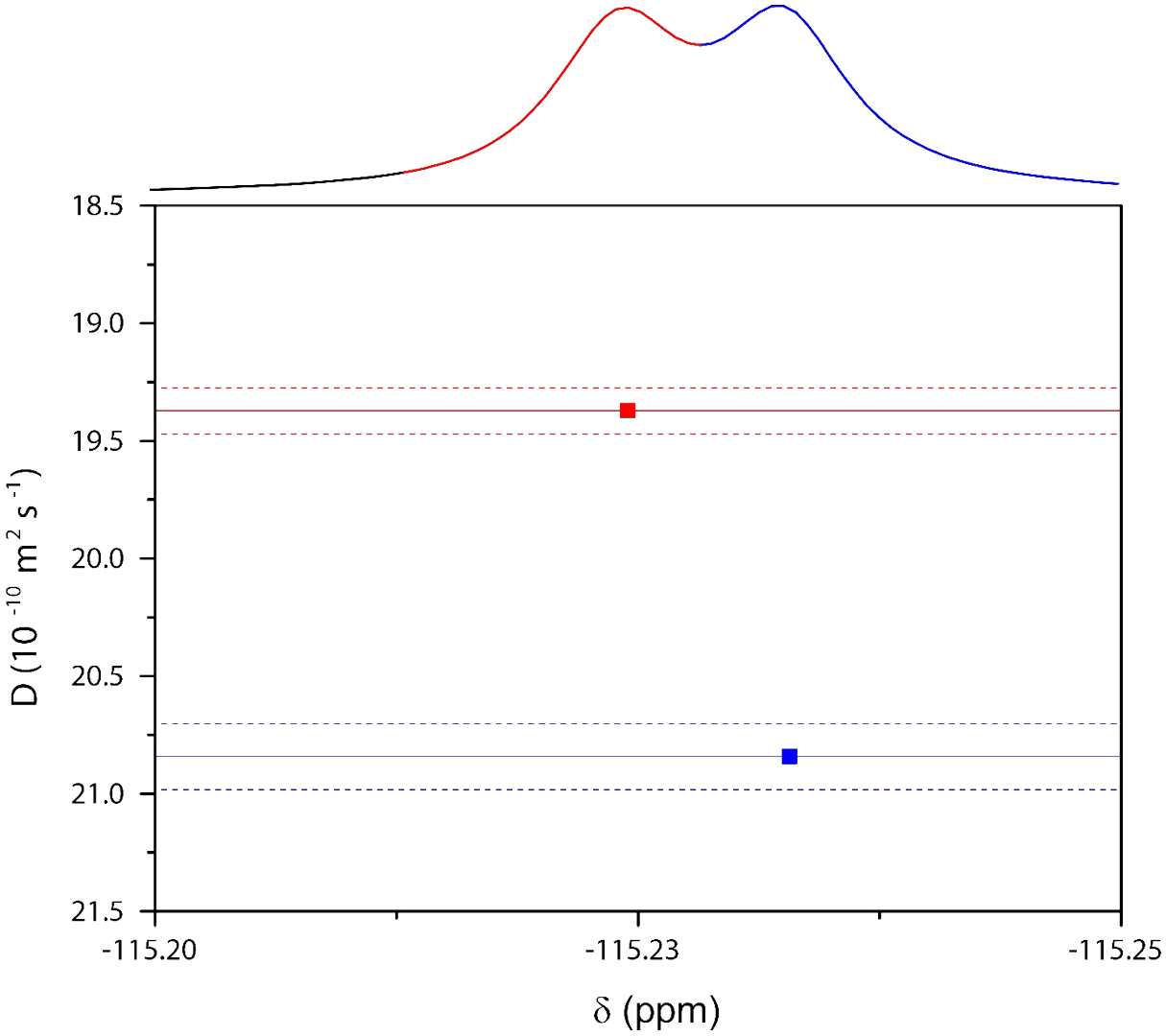


Figure S34: 500 MHz ^{1}H MAD projections, with the least attenuated 1D spectrum shown at the top, for compound **21** with matrix **1**

Table S32: Measured ^{1}H diffusion coefficients of compound **21** with matrix **1**

Chemical Shift	Exp Amplitude	Fit. Amplitude	Error	Diffusion coefficient[a]	Error[a]
1.40901	1.88287	1.92494	0.00638	16.18304	0.09207
1.41135	2.11322	2.15707	0.00663	16.08970	0.08509
1.42191	1.97652	2.02557	0.00703	16.10982	0.09538
1.42427	2.15047	2.20201	0.00731	15.90186	0.09124

[a] × 10^{-10} m^2s^{-1}.



FigureS 35: 470 MHz ^{19}F MAD projections, with the least attenuated 1D spectrum shown at the top, for compound **21** with matrix **1**

Table S33: Measured ^{19}F diffusion coefficients of compound **21** with matrix **1**

Chemical Shift	Exp Amplitude	Fit. Amplitude	Error	Diffusion coefficient[a]	Error[a]
-115.23285	34.29999	35.20581	0.14512	20.84247	0.14069
-115.22447	35.93416	36.62742	0.10968	19.37249	0.09739

[a] × 10^{-10} m^2s^{-1}.

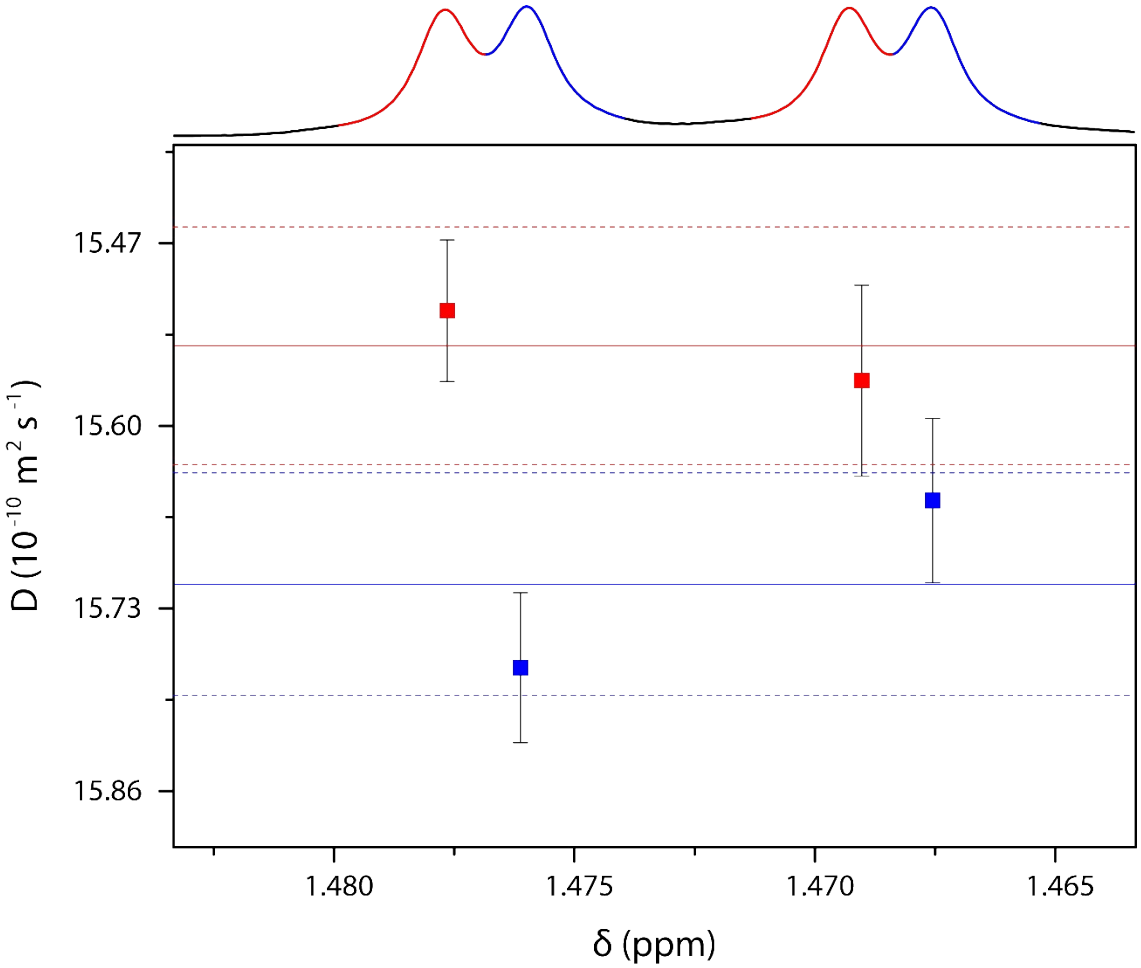


Figure S36: 500 MHz ^{1}H MAD projections, with the least attenuated 1D spectrum shown at the top, for compound **21** with matrix **2**

Table S34: Measured ^{1}H diffusion coefficients of compound **21** with matrix **2**

Chemical Shift	Exp Amplitude	Fit. Amplitude	Error	Diffusion coefficient[a]	Error[a]
1.46633	3.22707	3.26156	0.00664	15.65318	0.05849
1.46854	3.13290	3.18615	0.00755	15.56786	0.06795
1.47918	3.31760	3.35346	0.00621	15.77217	0.05345
1.48147	2.90653	2.94859	0.00519	15.51803	0.05035

[a] × 10^{-10} m^2s^{-1}.

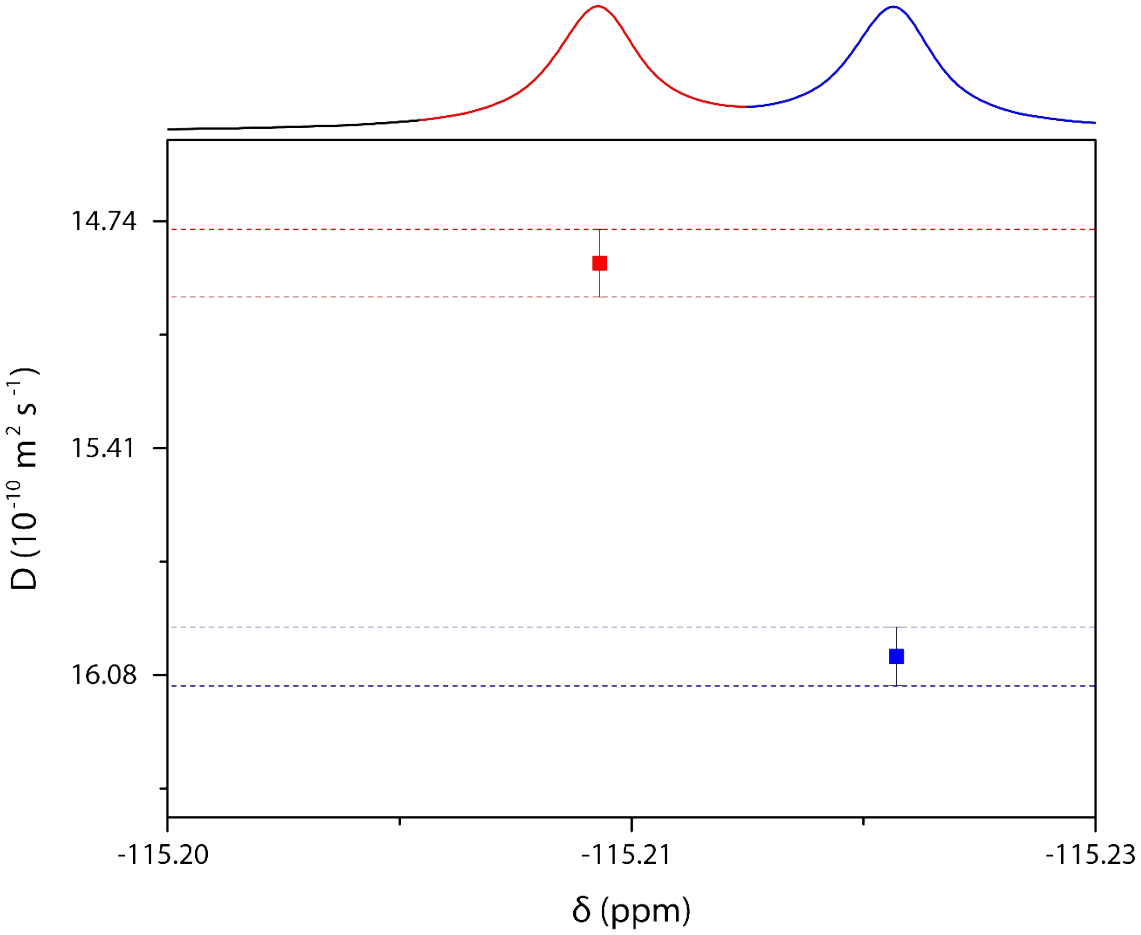


Figure S37: 470 MHz ^{19}F MAD projections, with the least attenuated 1D spectrum shown at the top, for compound **21** with matrix **2**

Table S35: Measured ^{19}F diffusion coefficients of compound **21** with matrix **2**

Chemical Shift	Exp Amplitude	Fit. Amplitude	Error	Diffusion coefficient[a]	Error[a]
-115.22357	35.59858	36.31540	0.10180	16.02499	0.08667
-115.21397	33.48251	34.34681	0.11432	14.86335	0.09948

[a] × 10^{-10} m^2s^{-1}.

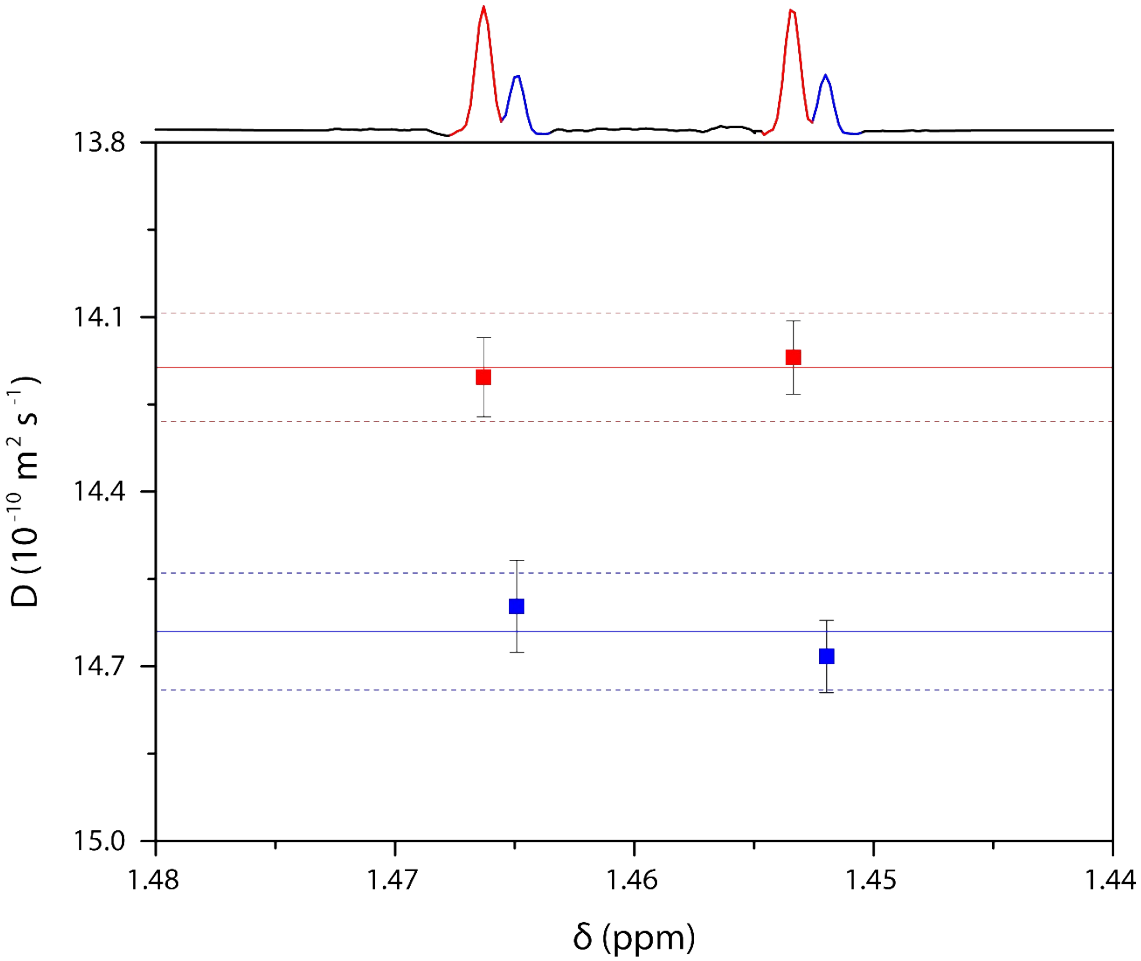


Figure S38: 500 MHz ^{1}H MAD projections, with the least attenuated 1D spectrum shown at the top, for compound **21** with matrix **5**

Table S36: Measured ^{1}H diffusion coefficients of compound **21** with matrix **5**

Chemical Shift	Exp Amplitude	Fit. Amplitude	Error	Diffusion coefficient[a]	Error[a]
1.45197	1.81497	1.84154	0.00507	14.71635	0.06781
1.45336	3.11113	3.16720	0.00879	14.22127	0.06691
1.46491	1.99712	2.02657	0.00726	14.65934	0.08811
1.46630	3.17046	3.22030	0.00913	14.17420	0.06822

[a] × 10^{-10} m^2s^{-1}.

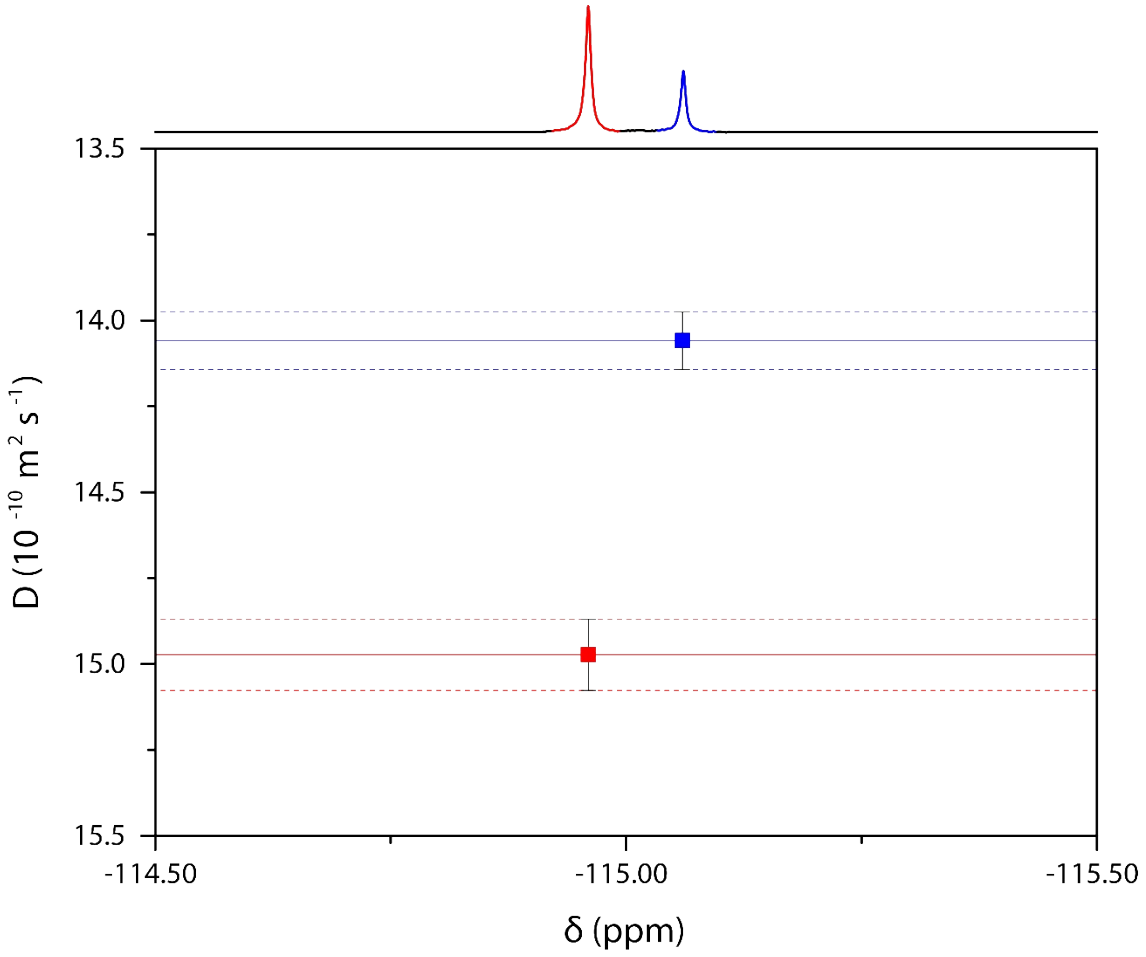


Figure S39: 470 MHz ^{19}F MAD projections, with the least attenuated 1D spectrum shown at the top, for compound **21** with matrix **5**

Table S37: Measured ^{19}F diffusion coefficients of compound **21** with matrix **5**

Chemical Shift	Exp Amplitude	Fit. Amplitude	Error	Diffusion coefficient[a]	Error[a]
-115.05398	64.67351	66.32904	0.26369	14.97297	0.10378
-114.95899	155.33808	158.92908	0.53061	14.05888	0.08411

[a] × 10^{-10} m^2s^{-1}.

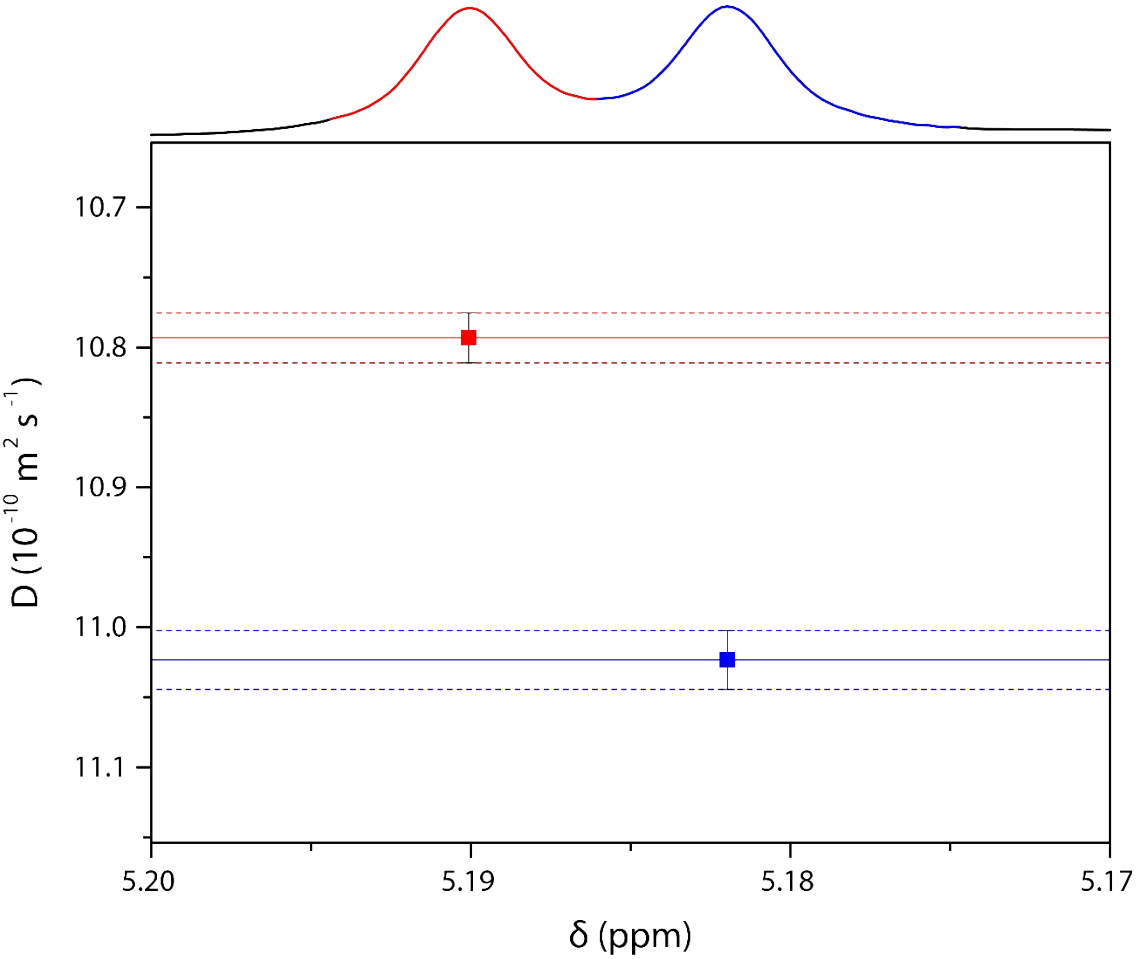


Figure S40: 500 MHz ^{1}H MAD projections, with the least attenuated 1D spectrum shown at the top, for compound **22** with matrix **1**

Table S38: Measured ^{1}H diffusion coefficients of compound **22** with matrix **1**

Chemical Shift	Exp Amplitude	Fit. Amplitude	Error	Diffusion coefficient[a]	Error[a]
5.18197	3.46637	3.46083	0.00459	11.08037	0.02732
5.19007	3.38298	3.38872	0.00387	10.78122	0.02320

[a] × 10^{-10} m^2s^{-1}.

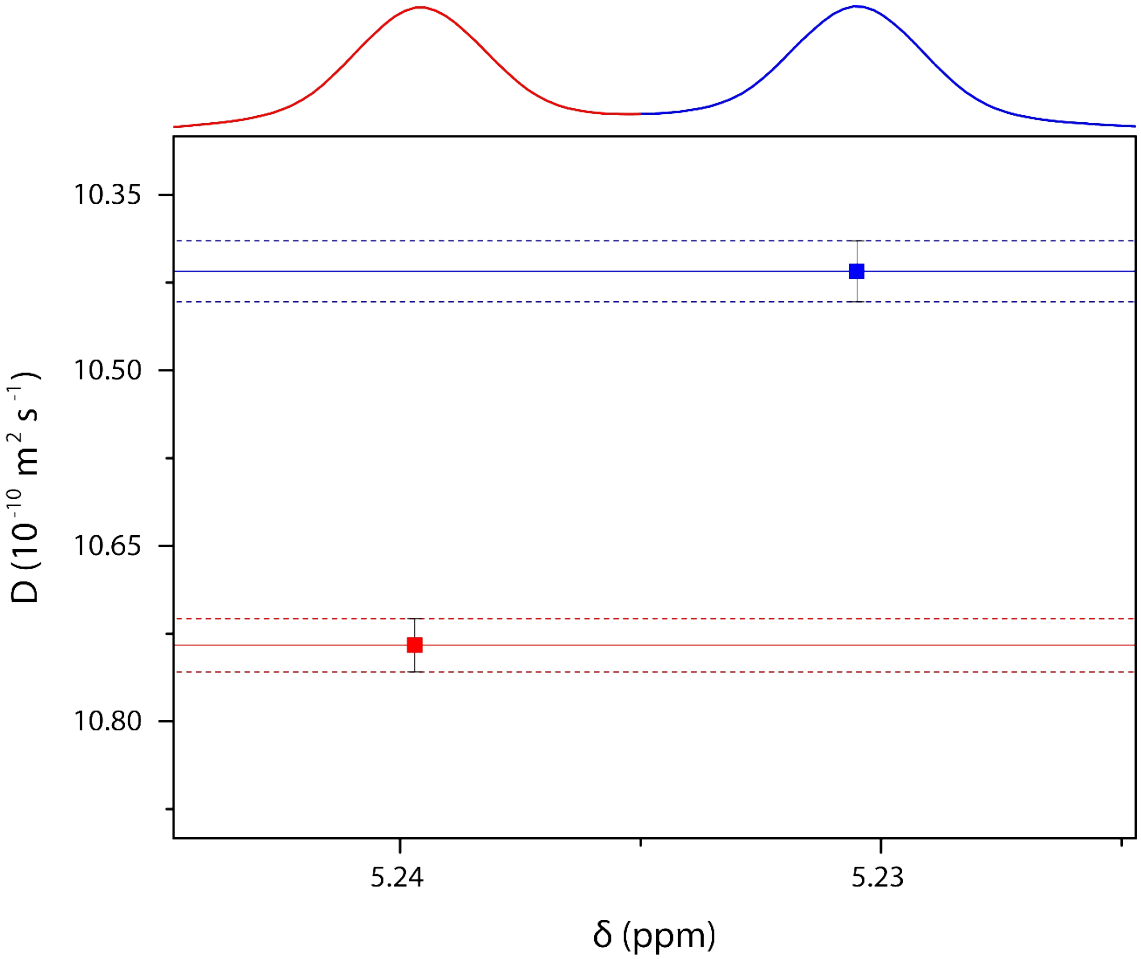


Figure S41: 500 MHz ^{1}H MAD projections, with the least attenuated 1D spectrum shown at the top, for **22** with matrix **2**

Table S39: Measured ^{1}H diffusion coefficients of compound **22** with matrix **2**

Chemical Shift	Exp Amplitude	Fit. Amplitude	Error	Diffusion coefficient[a]	Error[a]
5.22792	2.95133	2.96586	0.00437	10.41542	0.02602
5.23574	2.86011	2.87325	0.00375	10.73493	0.02281

[a] × 10^{-10} m^2s^{-1}.

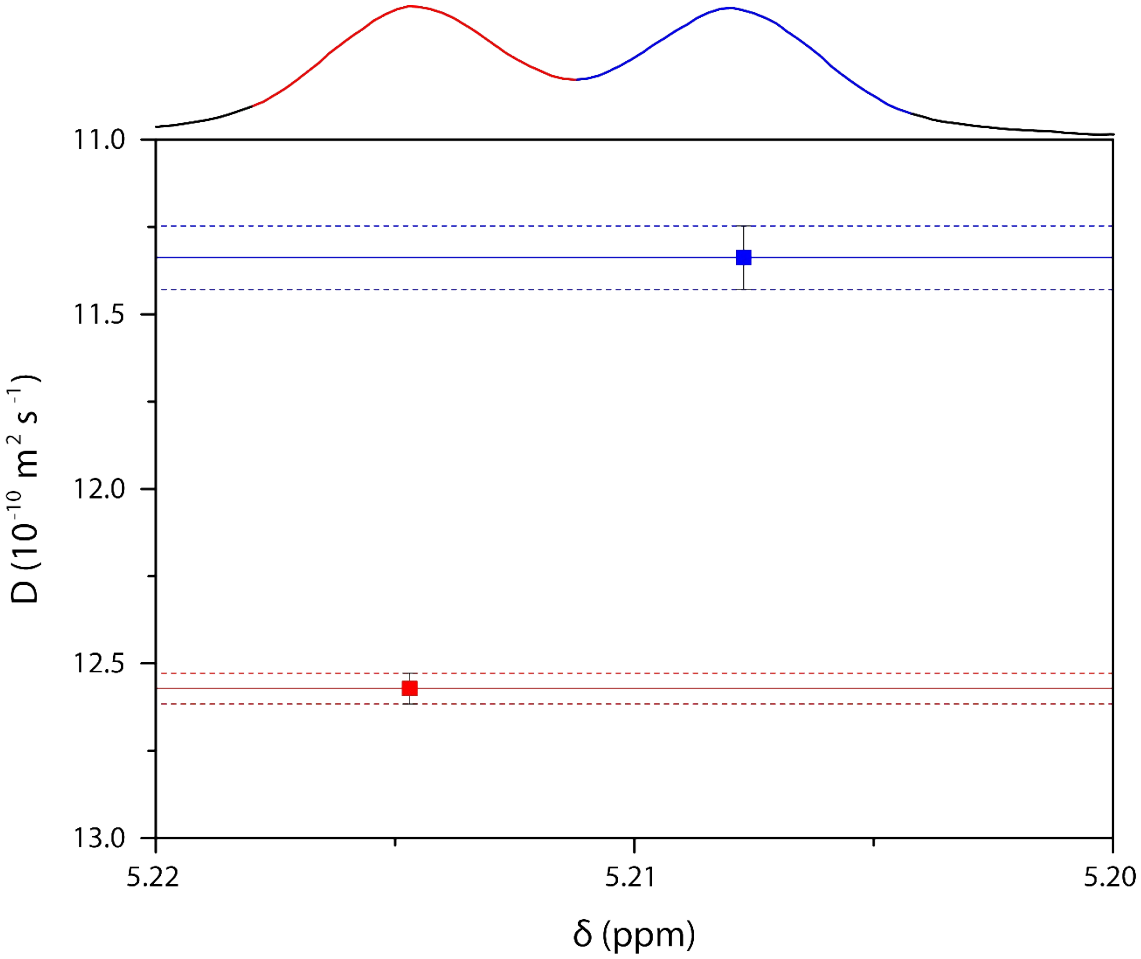


Figure S42: 500 MHz 1H MAD projections, with the least attenuated 1D spectrum shown at the top, for compound **22** with matrix **3**

Table S40: Measured 1H diffusion coefficients of compound **22** with matrix **3**

Chemical Shift	Exp Amplitude	Fit. Amplitude	Error	Diffusion coefficient[a]	Error[a]
5.20879	3.03221	3.12006	0.01300	11.33795	0.09072
5.21482	2.96480	3.01126	0.00579	12.57174	0.04410

[a] × 10^{-10} m^2s^{-1}.

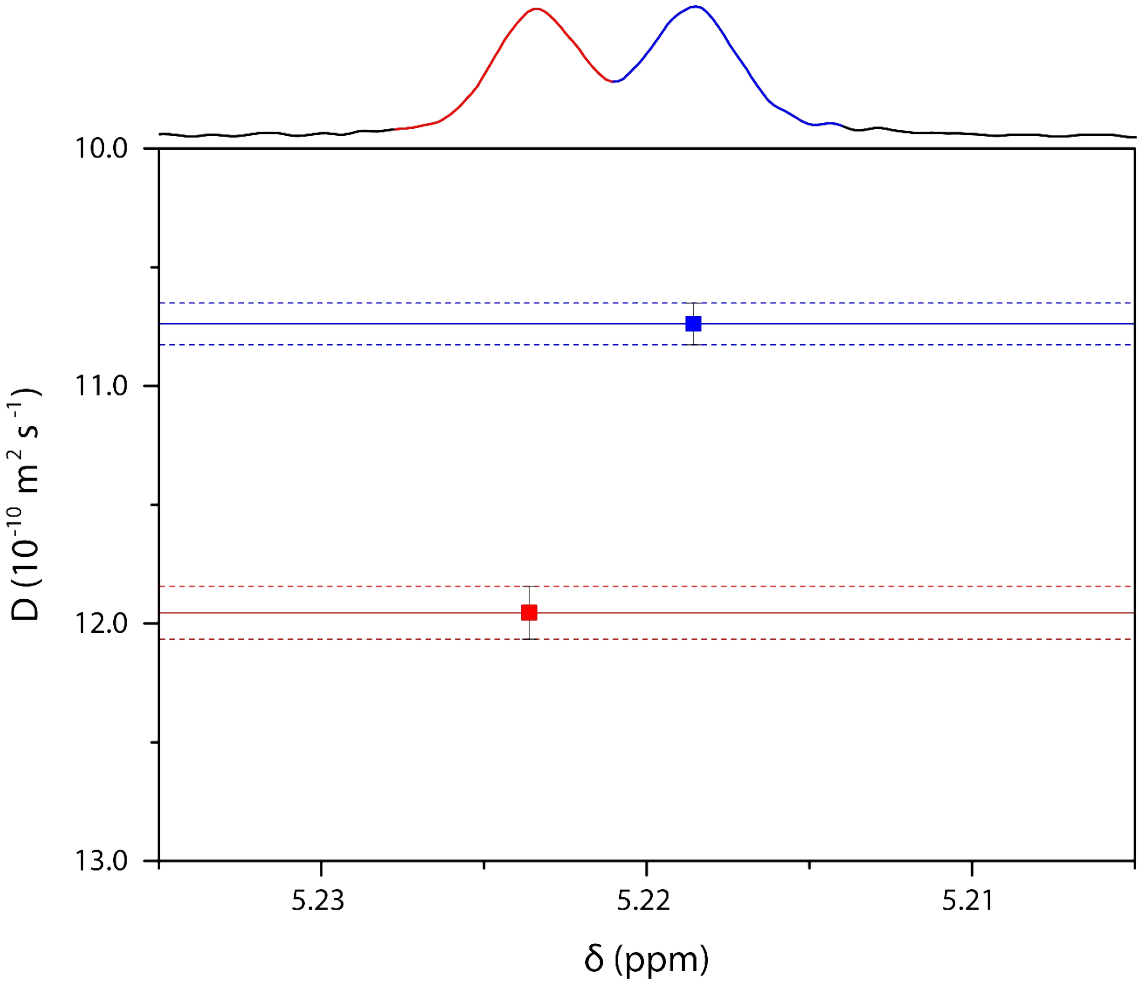


Figure S43: 500 MHz 1H MAD projections, with the least attenuated 1D spectrum shown at the top, for compound **22** with matrix **4**

Table S41: Measured 1H diffusion coefficients of compound **22** with matrix **4**

Chemical Shift	Exp Amplitude	Fit. Amplitude	Error	Diffusion coefficient[a]	Error[a]
5.21856	5.82146	5.93012	0.02728	10.73906	0.08795
5.22361	5.90230	6.05418	0.03315	11.95481	0.11146

[a] × 10^{-10} m^2s^{-1}.

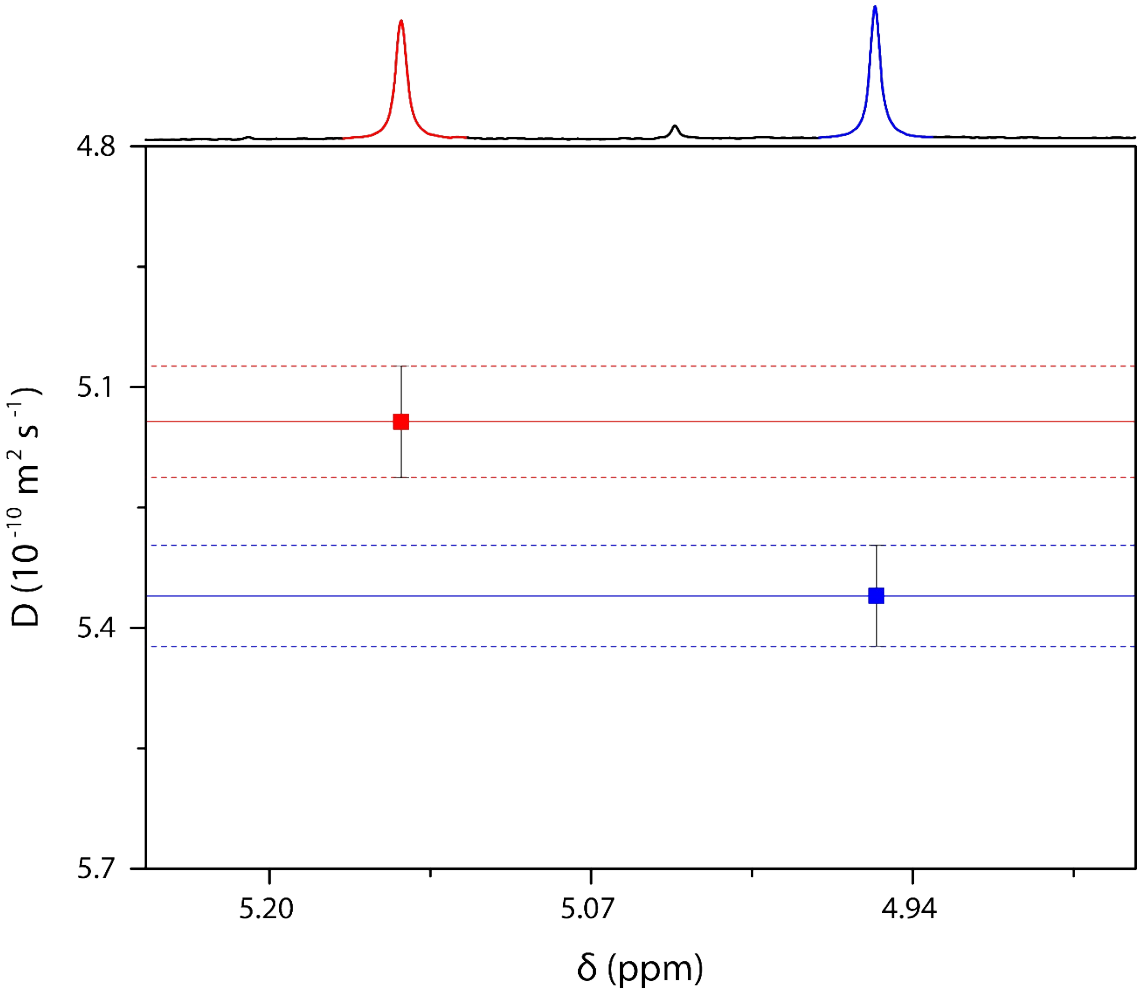


Figure S44: 500 MHz ^{1}H MAD projections, with the least attenuated 1D spectrum shown at the top, for compound **22** with matrix **5**

Table S42: Measured ^{1}H diffusion coefficients of compound **22** with matrix **5**

Chemical Shift	Exp Amplitude	Fit. Amplitude	Error	Diffusion coefficient[a]	Error[a]
4.95465	4.26278	4.42193	0.02680	5.36005	0.06298
5.14675	4.10159	4.22652	0.02880	5.14321	0.06949

[a] × 10^{-10} m^2s^{-1}.

References

1 L. Castañar, G. D. Poggetto, A. A. Colbourne, G. A. Morris, and M. Nilsson, *Magnetic Resonance in Chemistry*, 2018, **56**, 546.

2 G. A. Morris, *Journal of Magnetic Resonance*, 1988, **80**, 547.