Application of Polysaccharide-Based Chiral HPLC Columns for Separation of Non-Enantiomeric Isomeric Mixtures of Organometallic Compounds

Masamichi Ogasawara,[*,†] Yuuki Enomoto,[†] Mizuho Uryu,[†,‡] Xicheng Yang,[‡] Ayami Kataoka,[†] and Atsushi Ohnishi[*,§]

[†]*Department of Natural Science, Graduate School of Science and Technology and Research Cluster on "Innovative Chemical Sensing", Tokushima University, Tokushima 770-8506, Japan* [‡]*Graduate School of Life Science, Hokkaido University, Kita-ku, Sapporo 001-0021, Japan* [§]*Daicel Corporation, CPI Company, Life Sciences Development Center, Arai 944-8550, Japan*

Supporting Information

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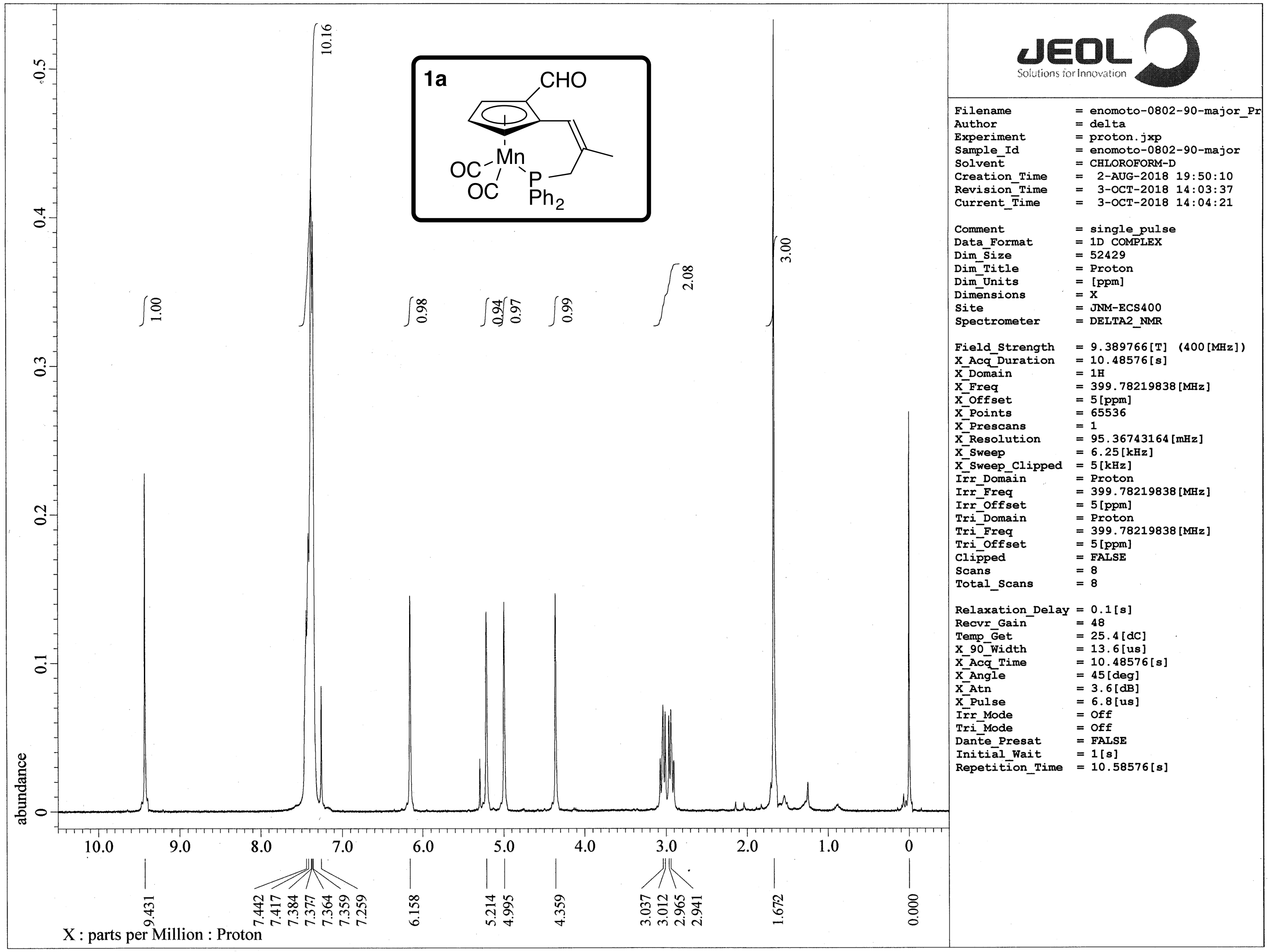


Figure S1. ^{1}H-NMR Spectrum of **1a** in $CDCl_3$ at 400 MHz.

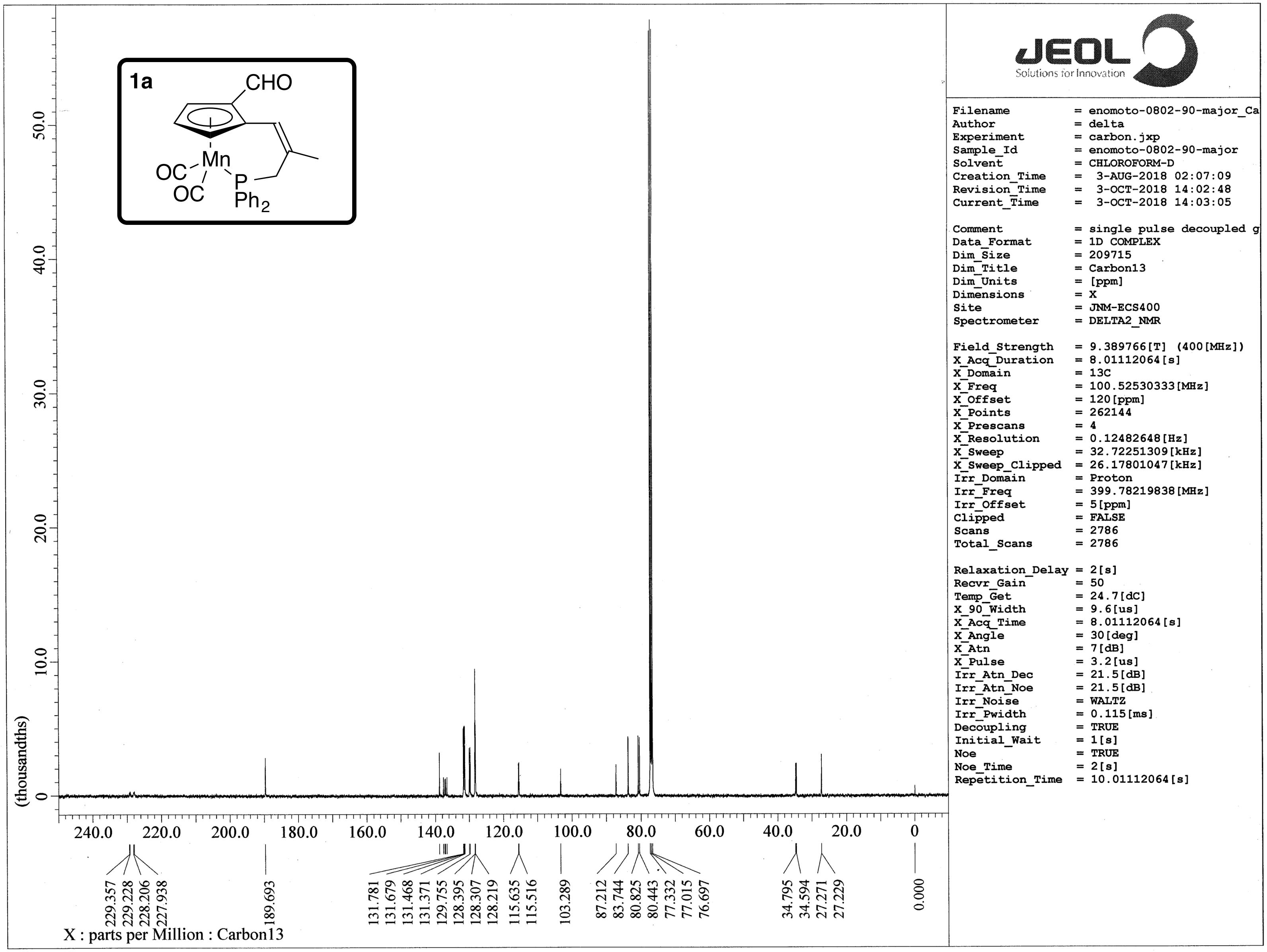


Figure S2. ^{13}C-NMR Spectrum of **1a** in $CDCl_3$ at 101 MHz.

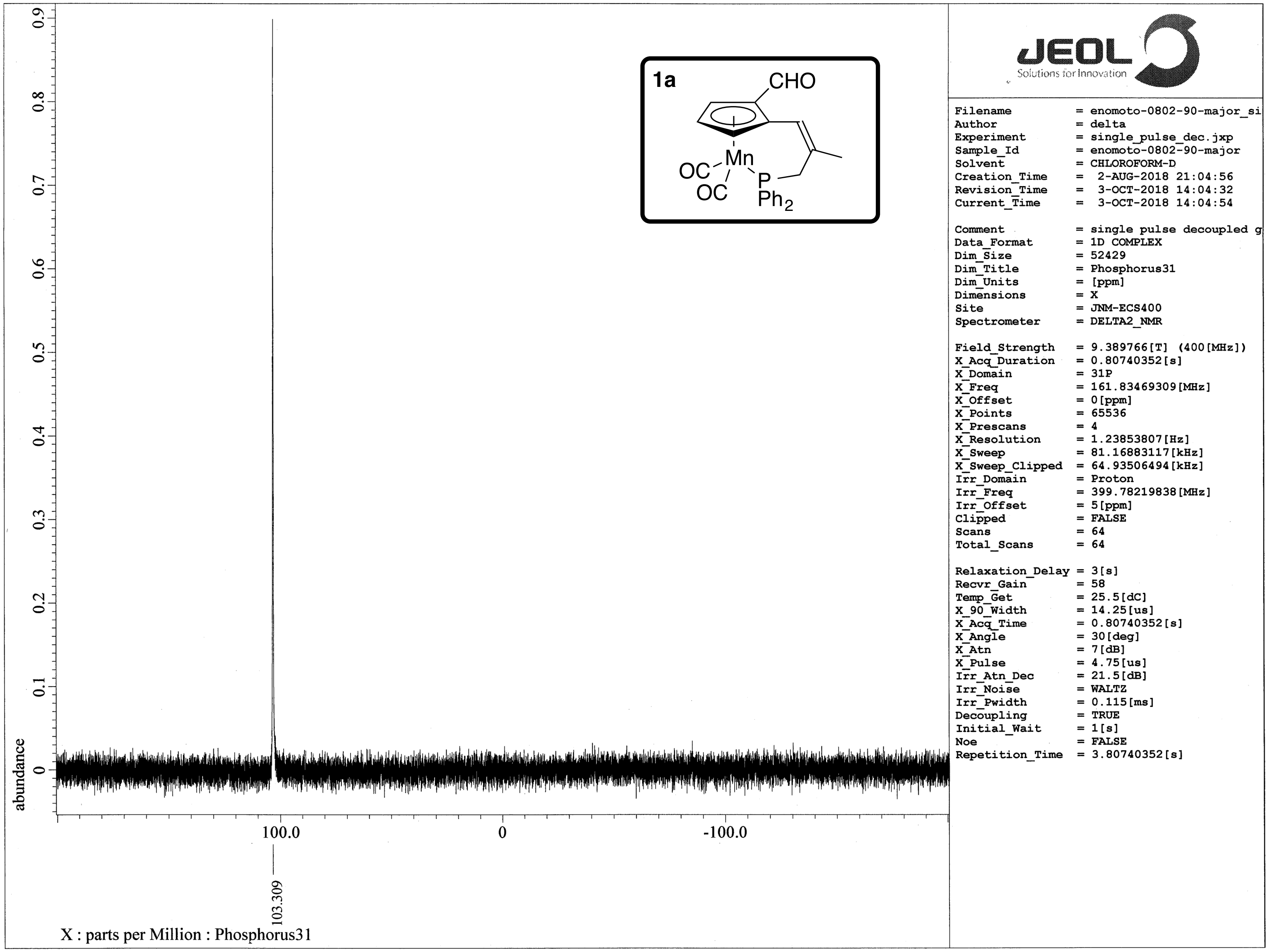


Figure S3. ^{31}P-NMR Spectrum of **1a** in $CDCl_3$ at 162 MHz.

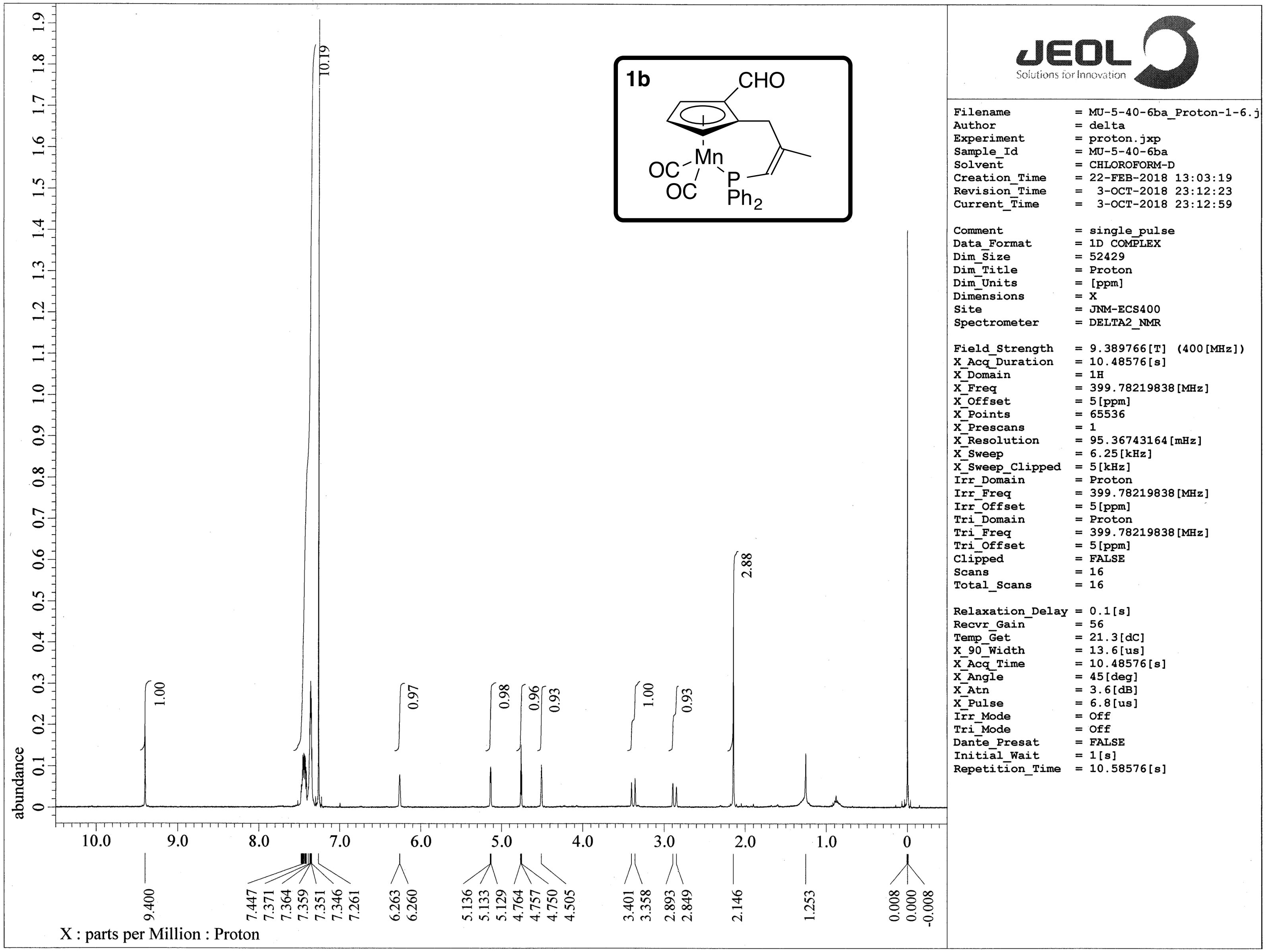


Figure S4. ^{1}H-NMR Spectrum of **1b** in $CDCl_3$ at 400 MHz.

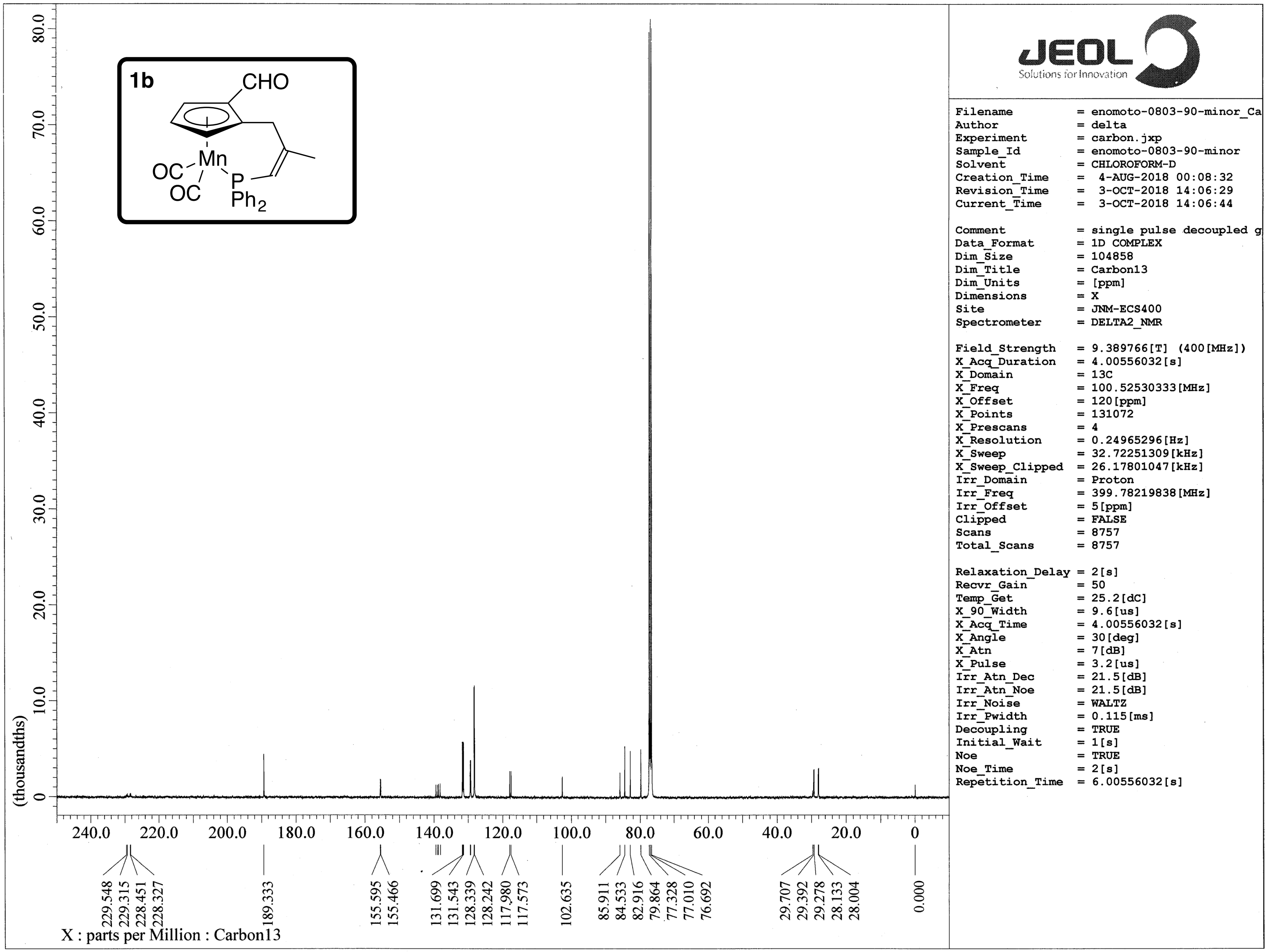


Figure S5. ^{13}C-NMR Spectrum of **1b** in $CDCl_3$ at 101 MHz.

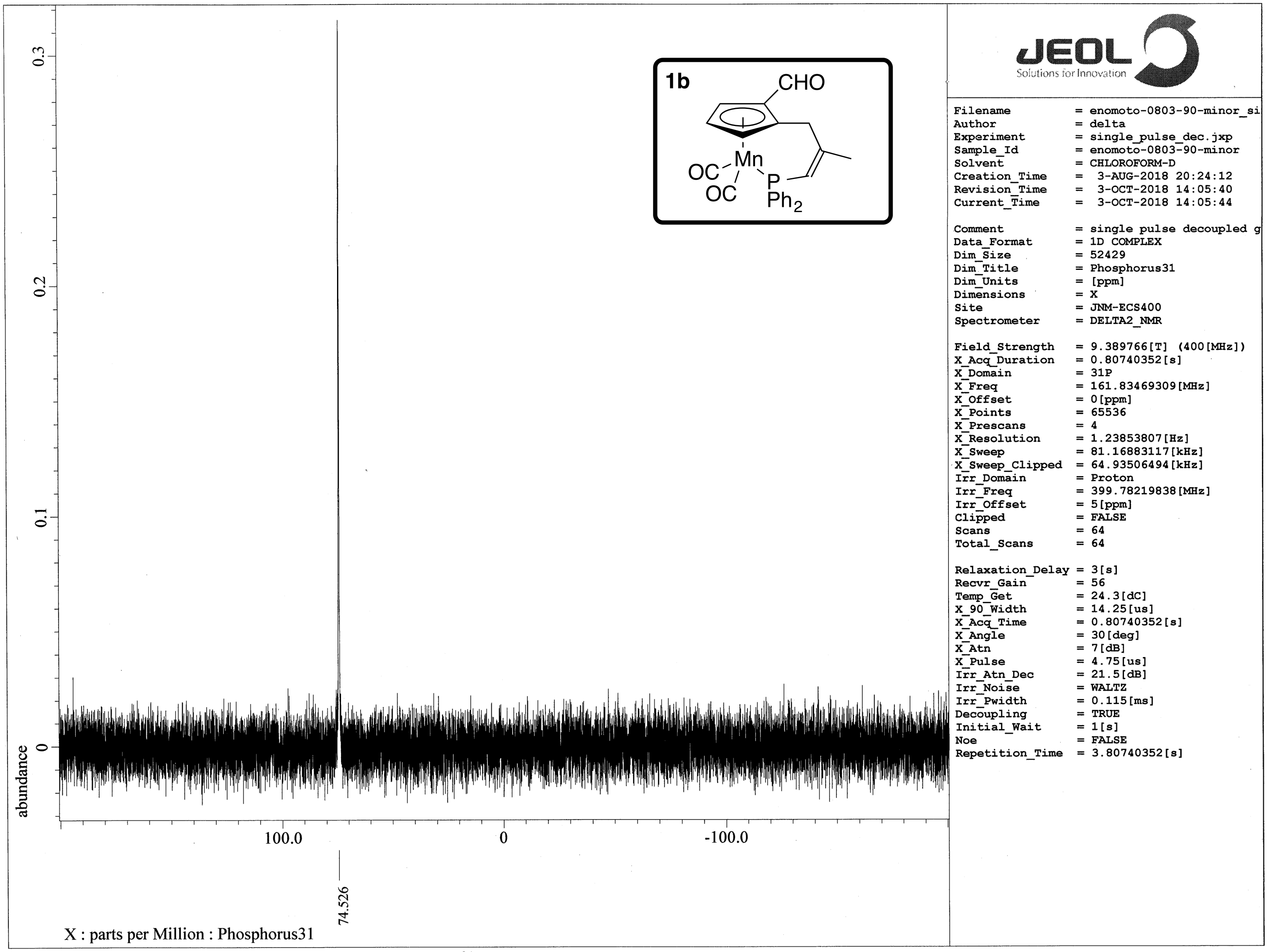


Figure S6. ^{31}P-NMR Spectrum of **1b** in $CDCl_3$ at 162 MHz.

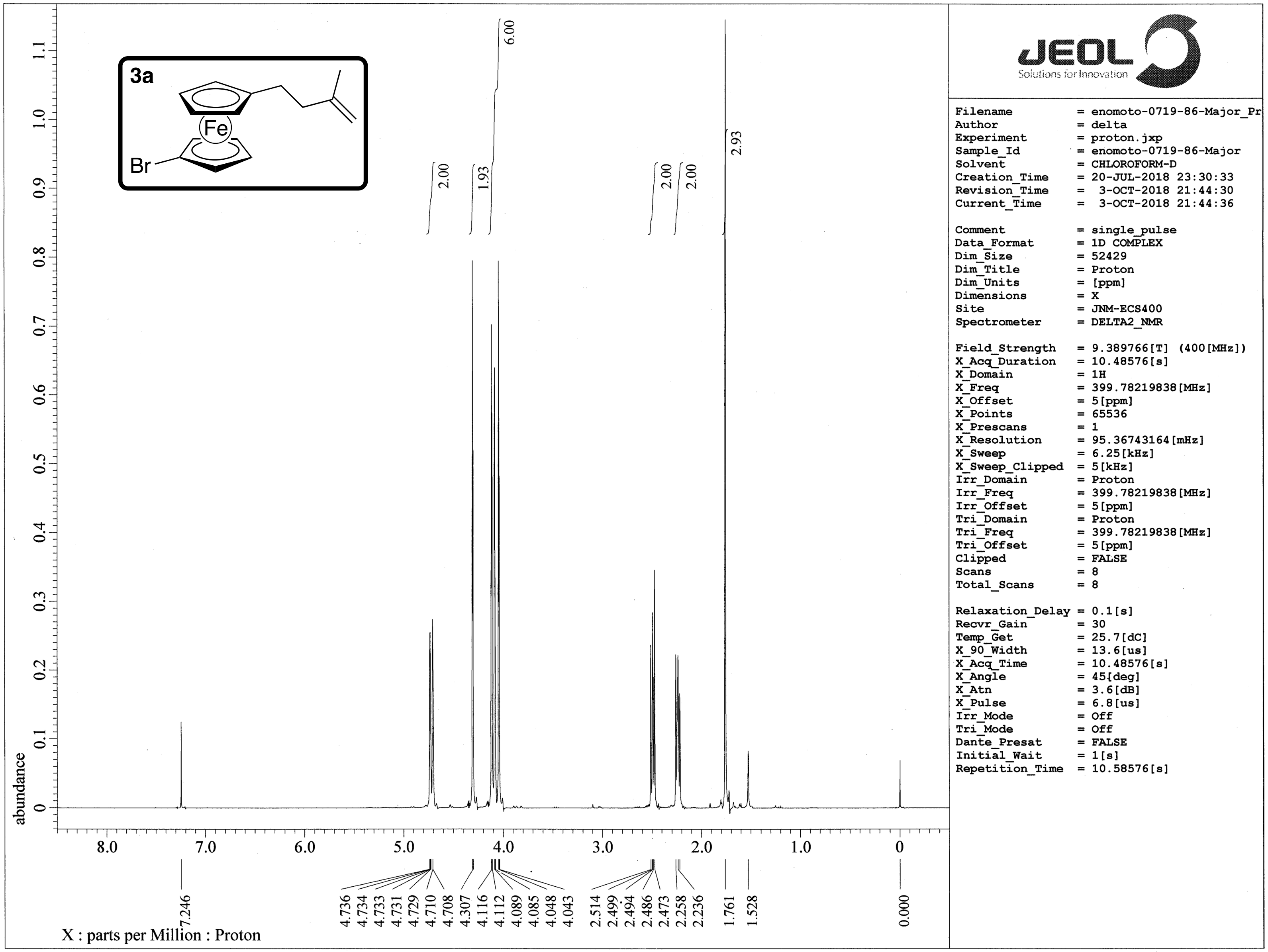


Figure S7. ^{1}H-NMR Spectrum of **3a** in $CDCl_3$ at 400 MHz.

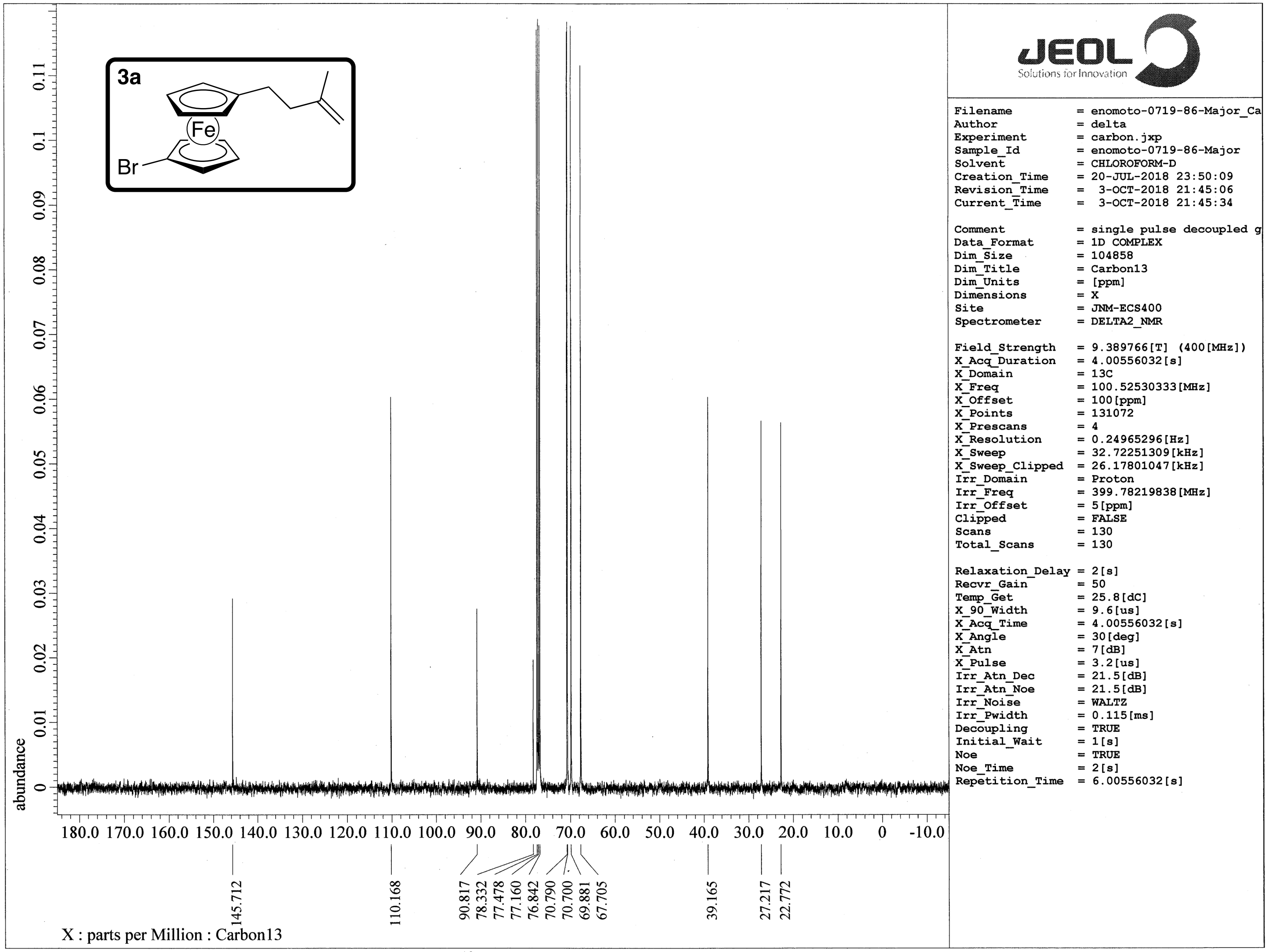


Figure S8. ^{13}C-NMR Spectrum of **3a** in $CDCl_3$ at 101 MHz.

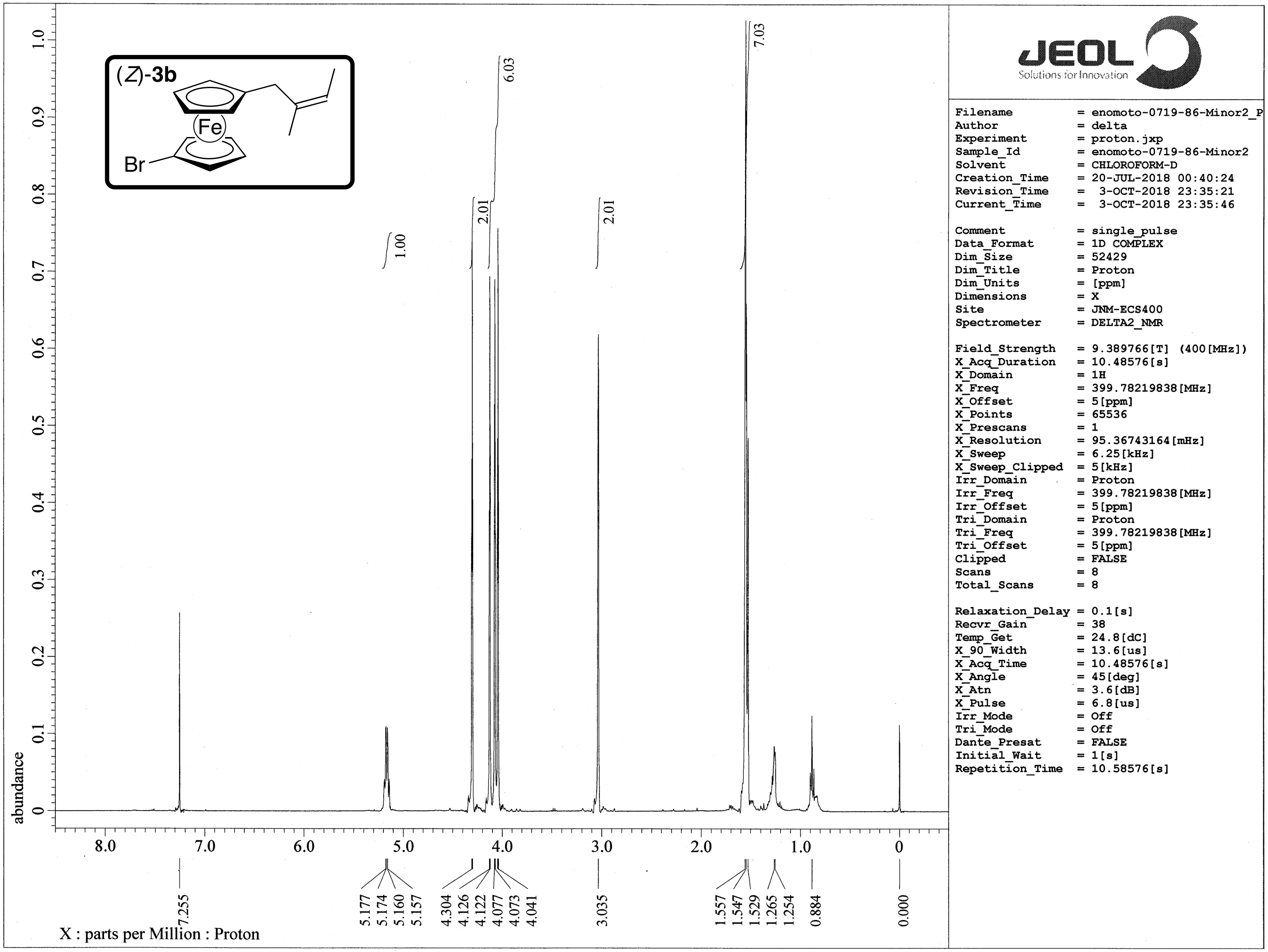


Figure S9. ^{1}H-NMR Spectrum of (*Z*)-**3b** in $CDCl_3$ at 400 MHz.

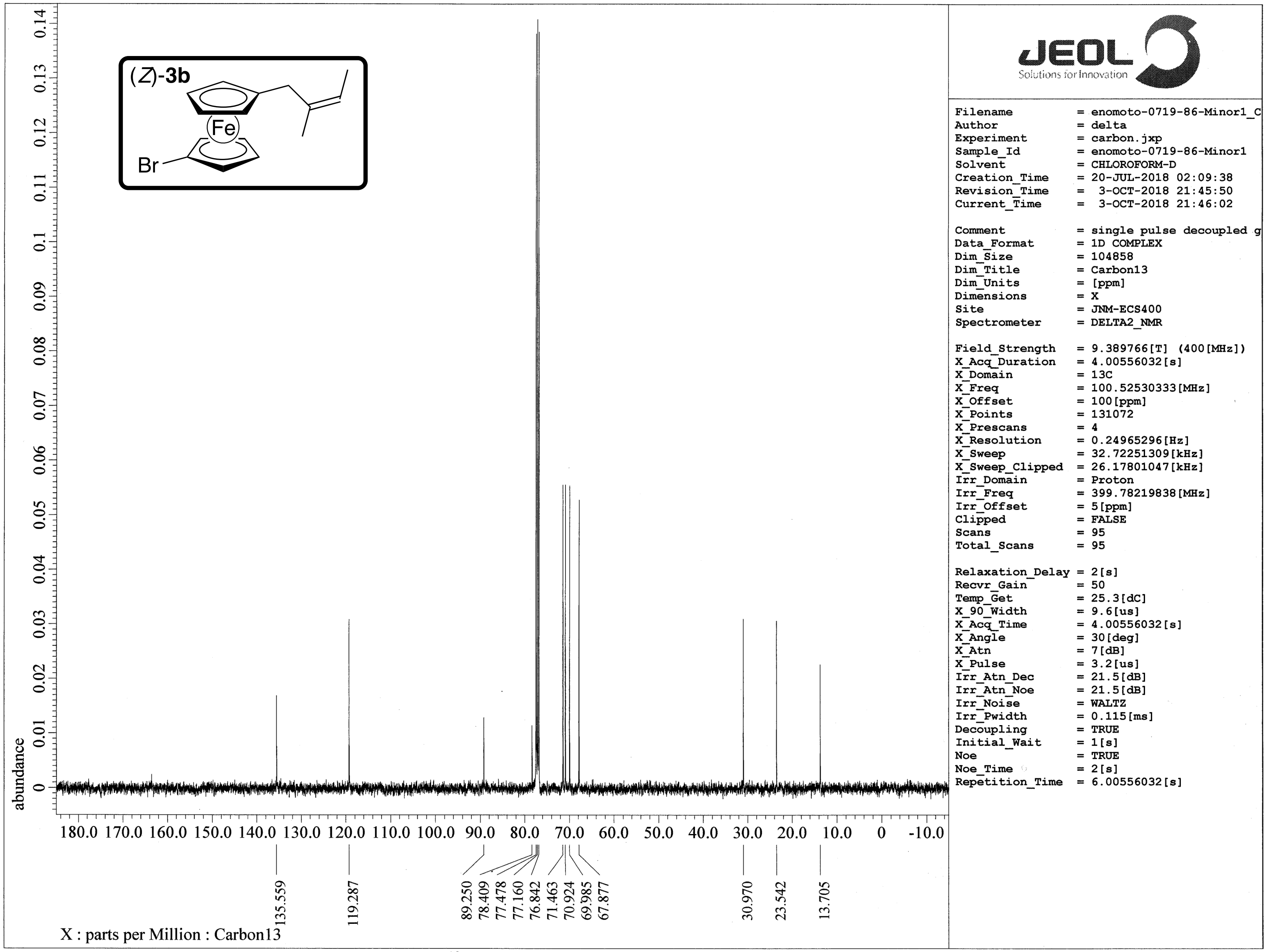


Figure S10. ^{13}C-NMR Spectrum of (*Z*)-**3b** in $CDCl_3$ at 101 MHz.

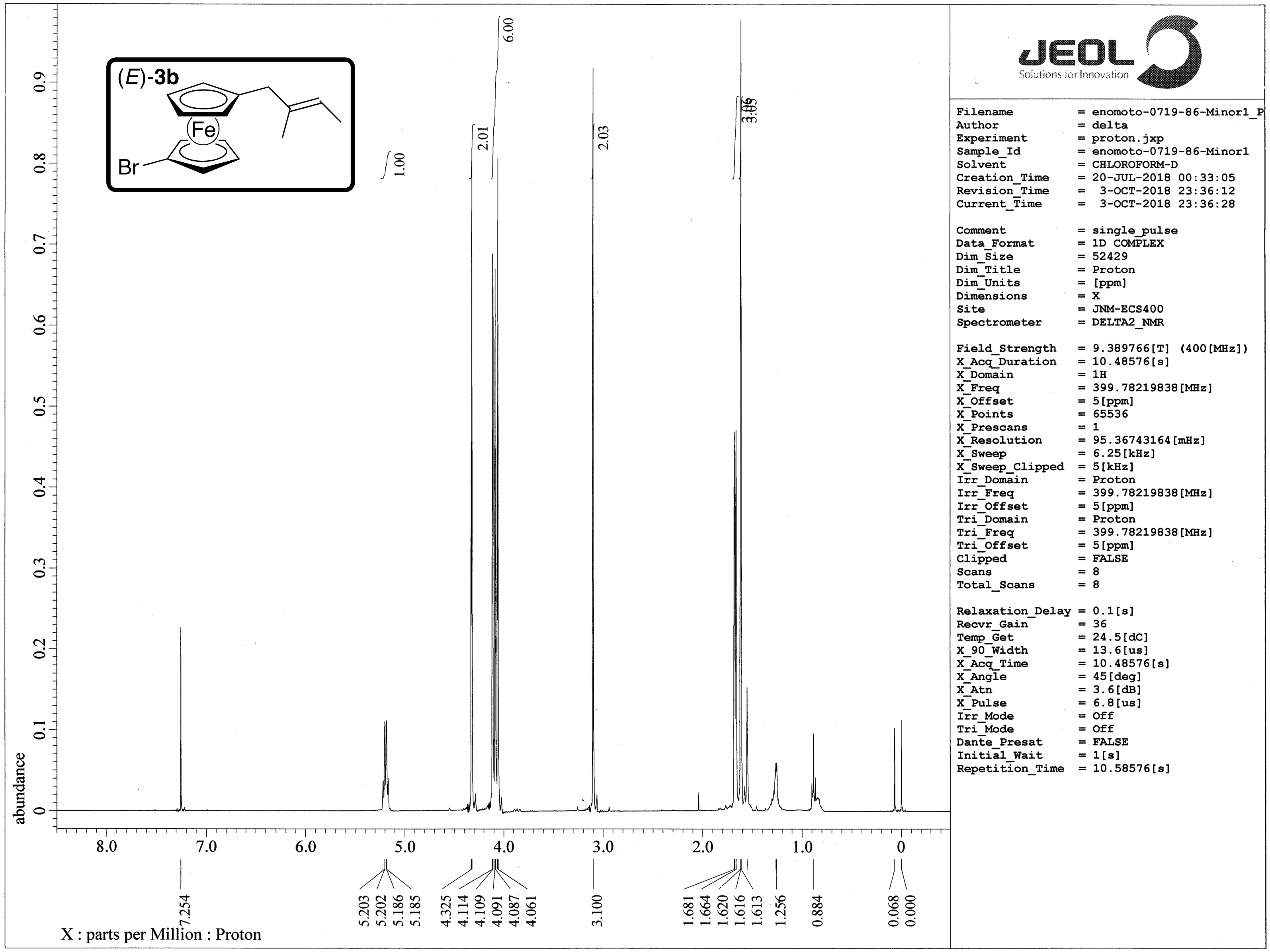


Figure S11. ^{1}H-NMR Spectrum of (*E*)-**3b** in $CDCl_3$ at 400 MHz.

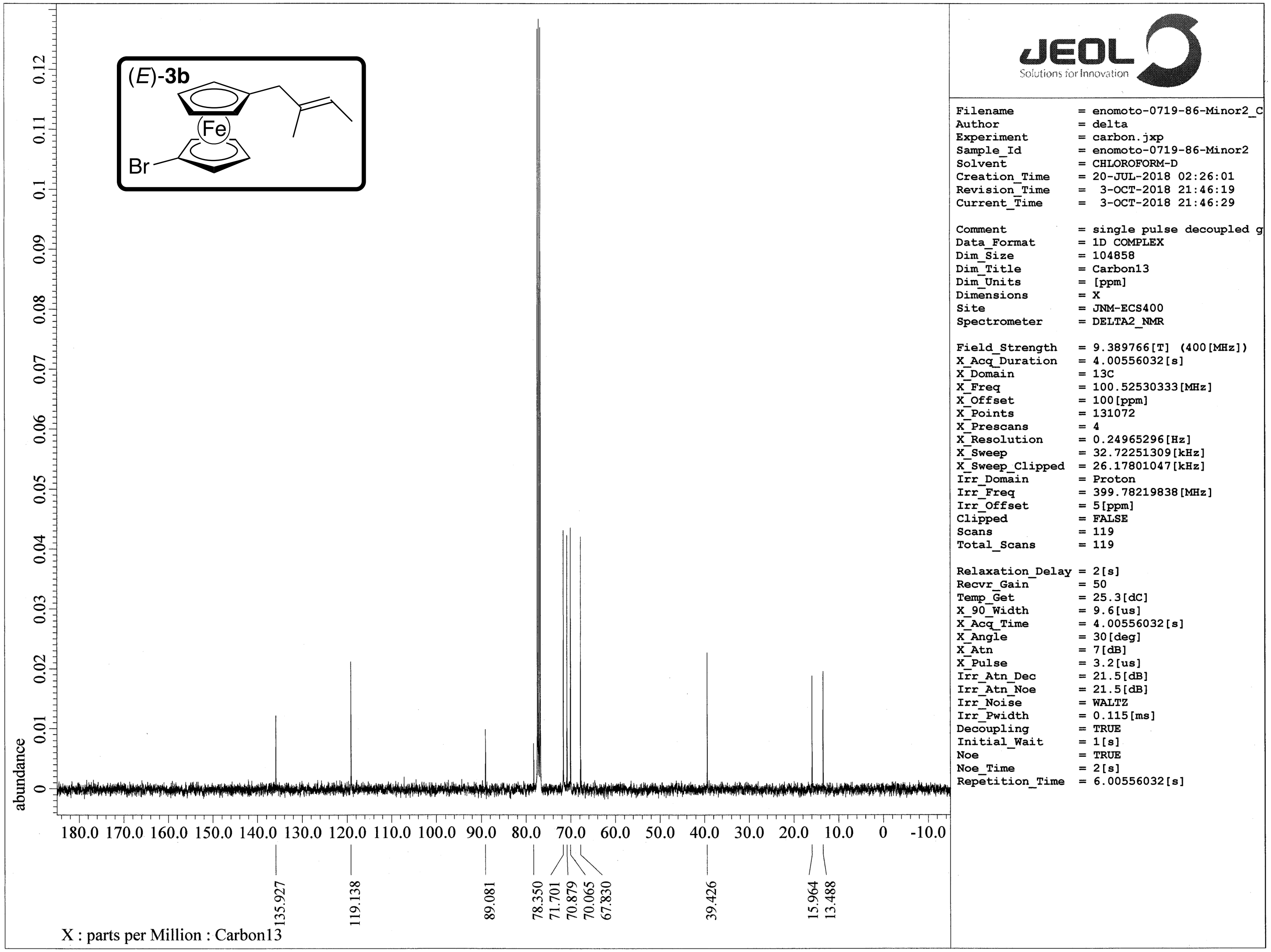


Figure S12. ^{13}C-NMR Spectrum of (*E*)-**3b** in $CDCl_3$ at 101 MHz.

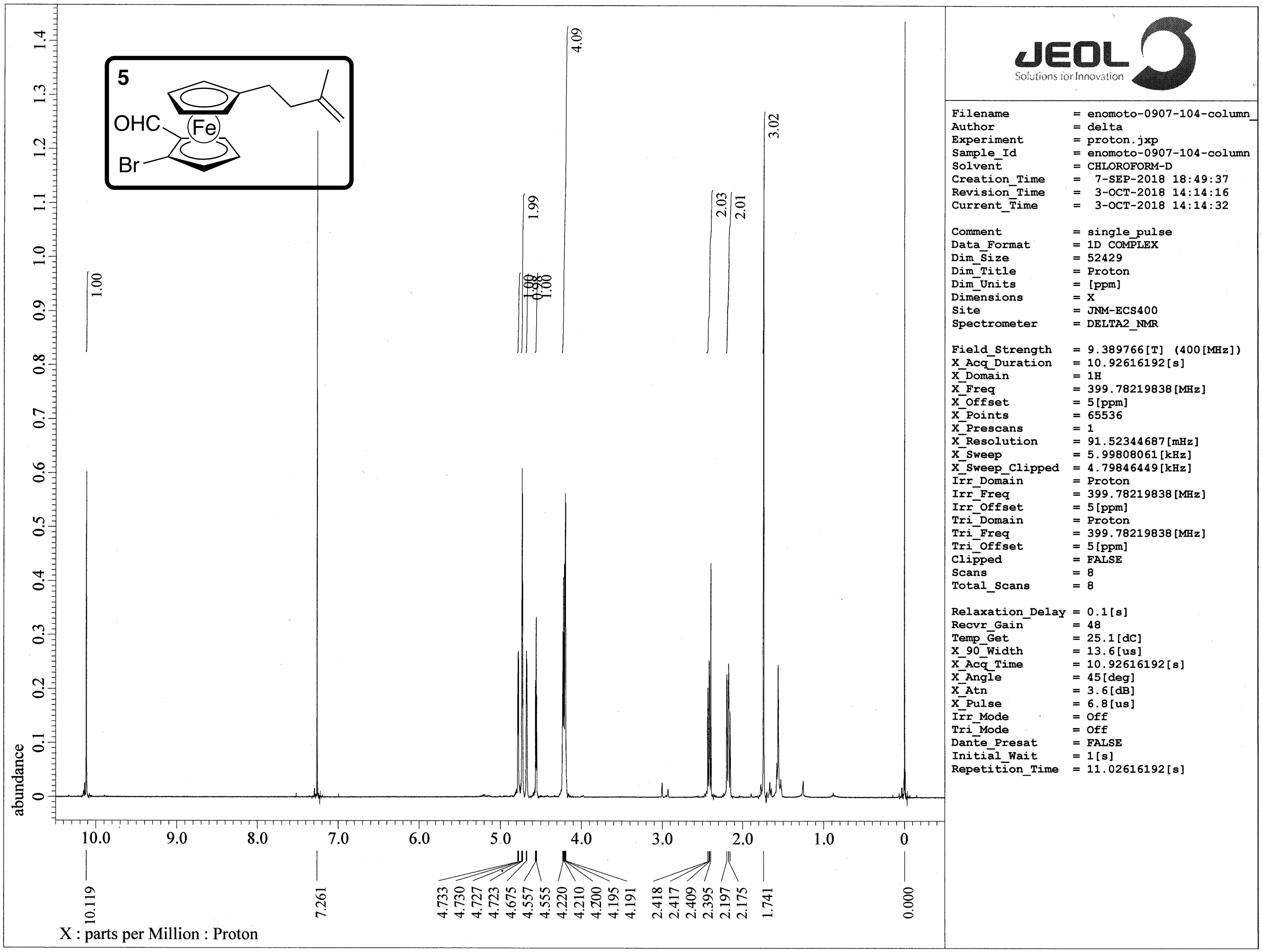


Figure S13. ^{1}H-NMR Spectrum of **5** in $CDCl_3$ at 400 MHz.

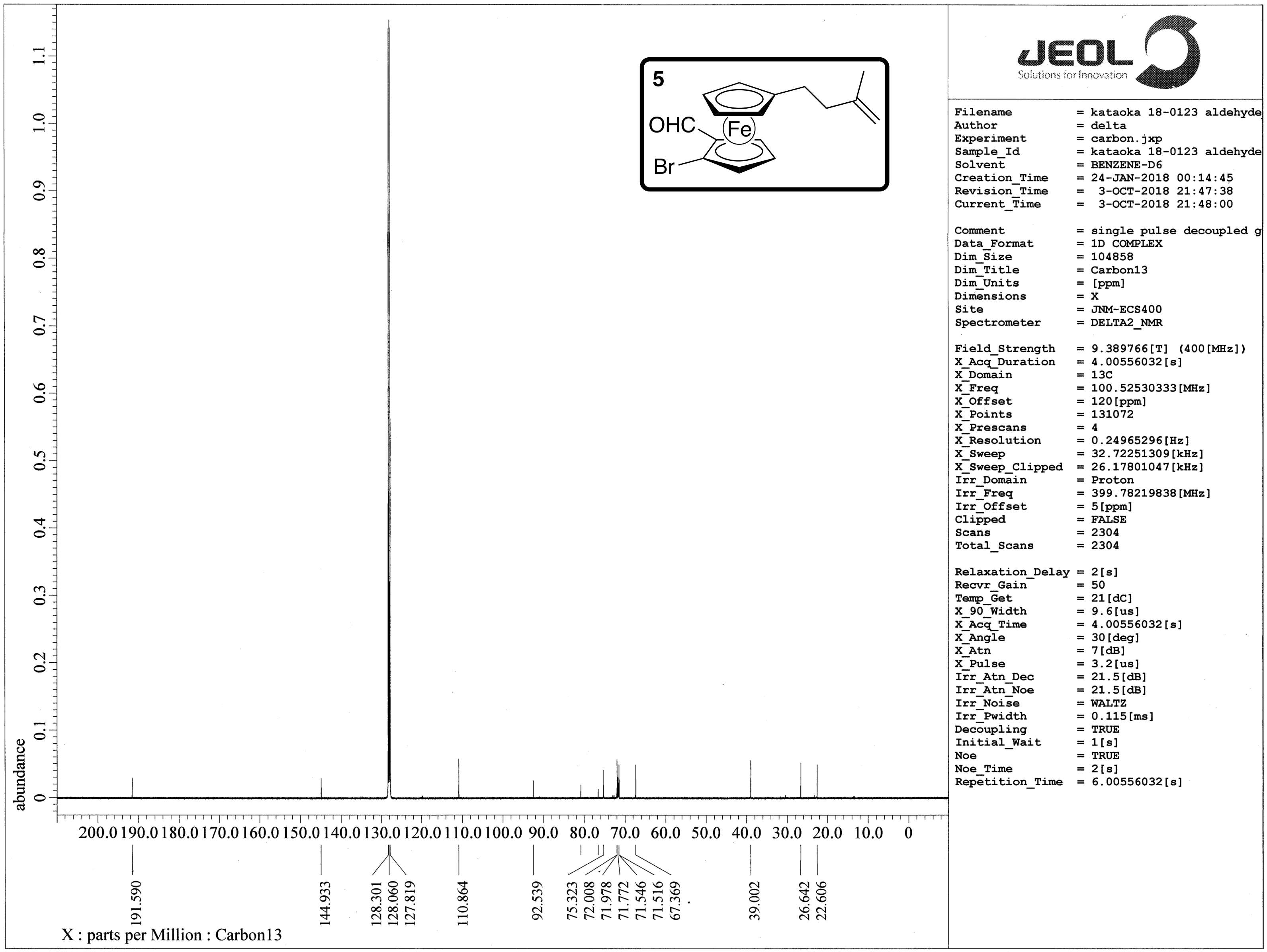


Figure S14. ^{13}C-NMR Spectrum of **5** in $CDCl_3$ at 100 MHz.

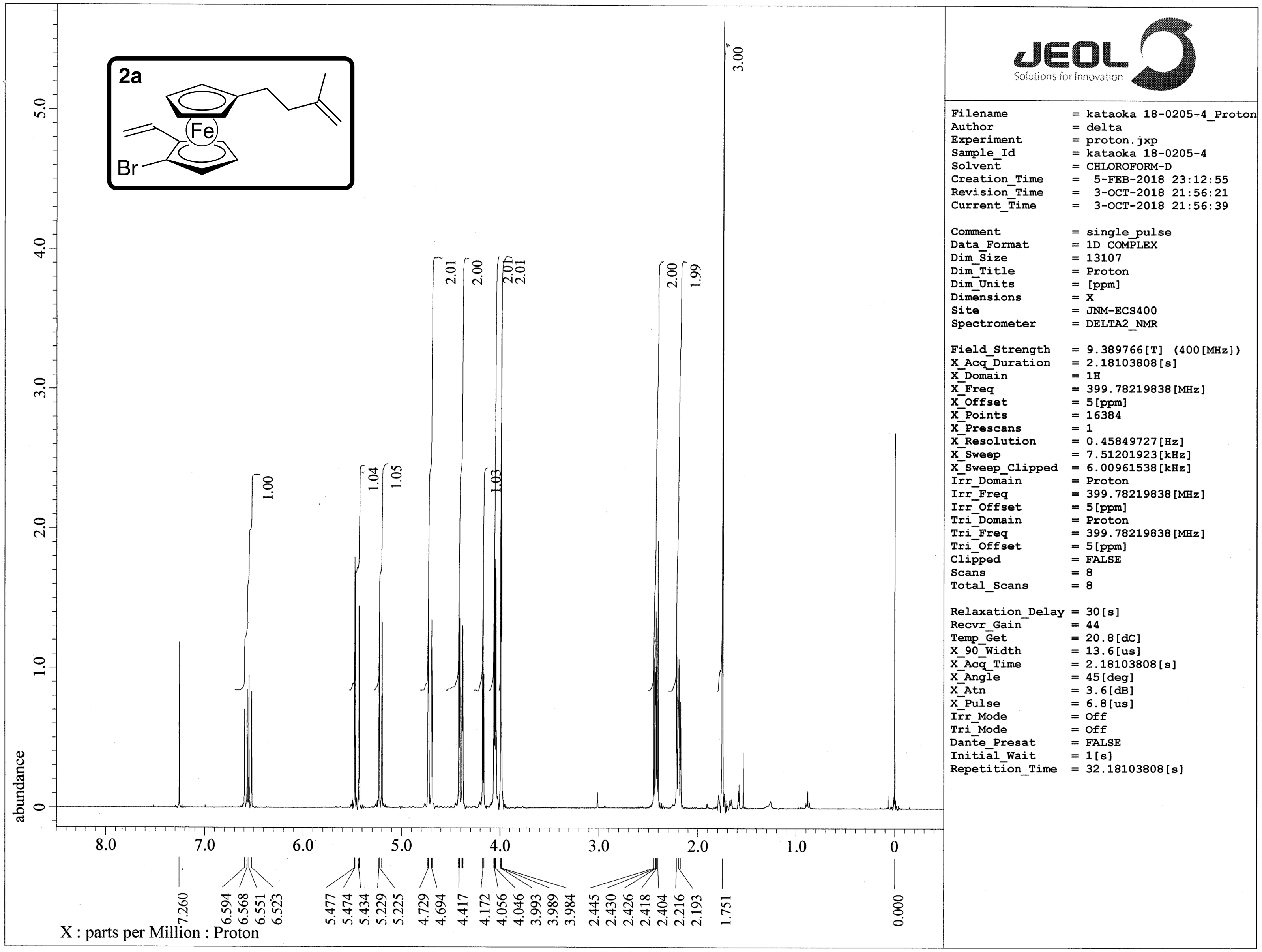


Figure S15. [1]H-NMR Spectrum of **2a** in $CDCl_3$ at 400 MHz.

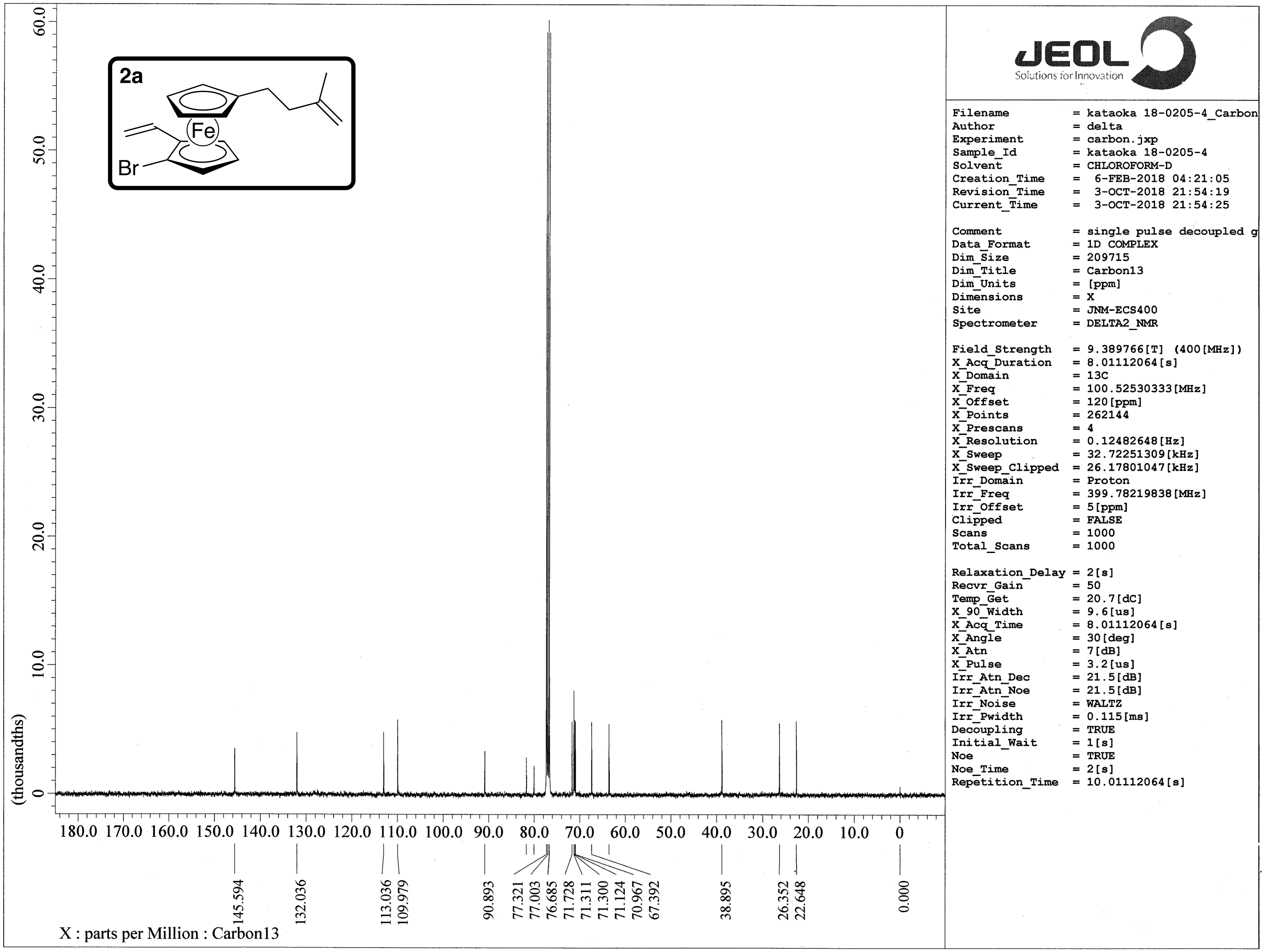


Figure S16. ^{13}C-NMR Spectrum of **2a** in $CDCl_3$ at 101 MHz.

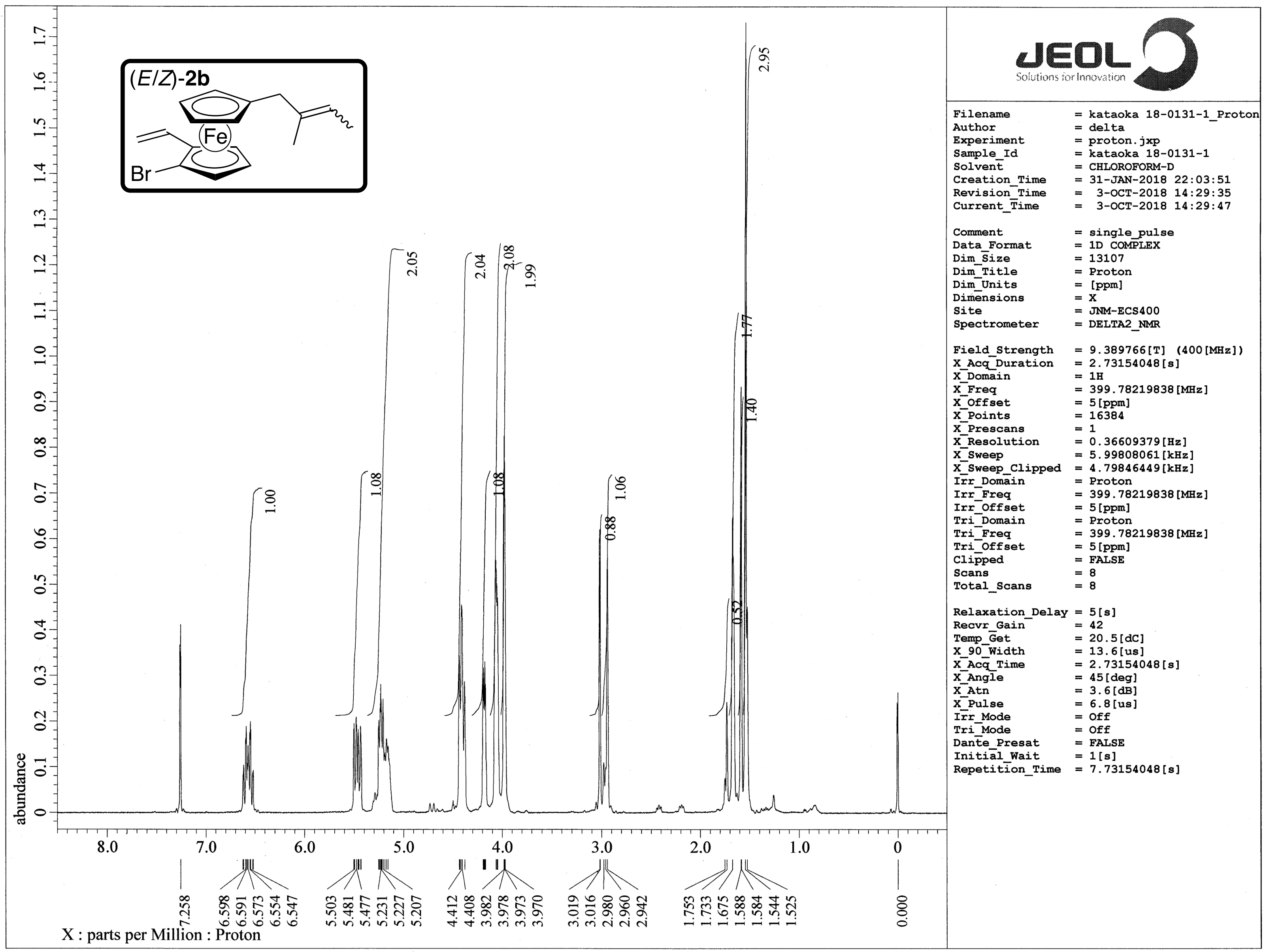


Figure S17. ^{1}H-NMR Spectrum of (*Z*)- and (*E*)-**2b** in $CDCl_3$ at 400 MHz.

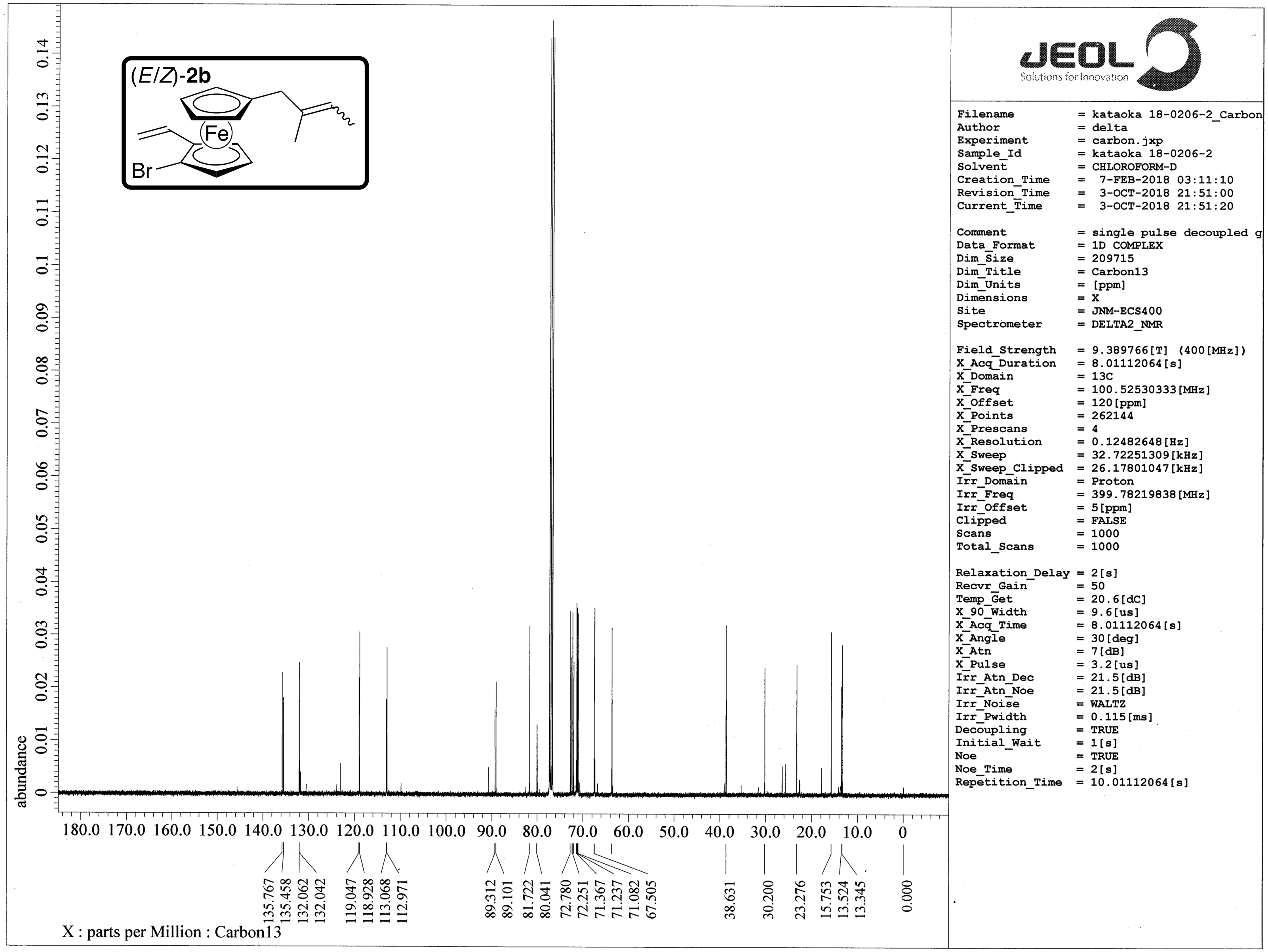
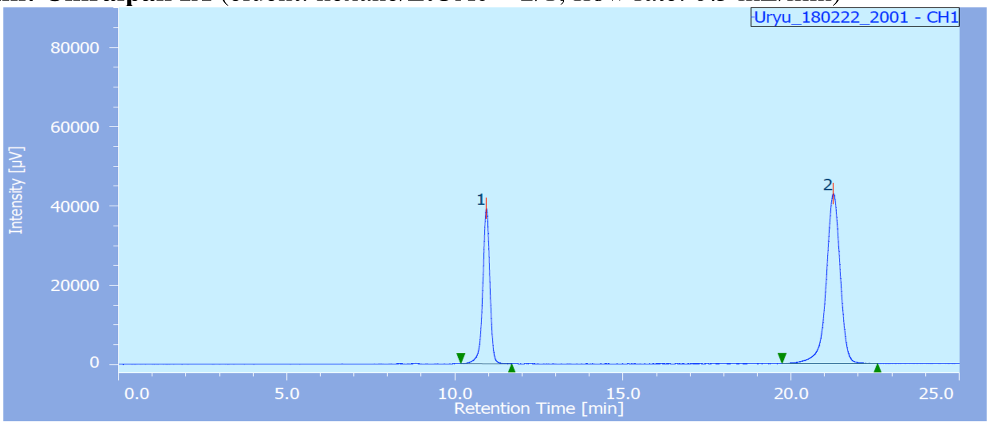


Figure S18. ^{13}C-NMR Spectrum of (*Z*)- and (*E*)-**2b** in $CDCl_3$ at 101 MHz.

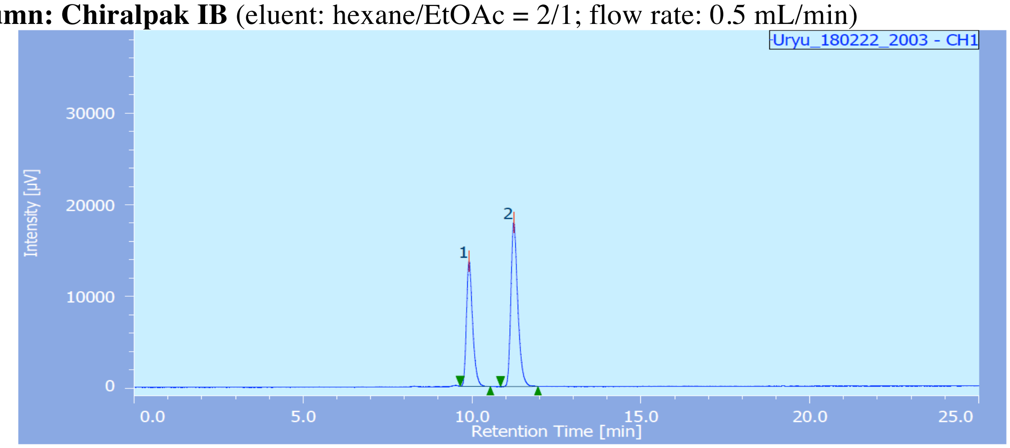
Figure S19. HPLC Traces for the Mixture of (*R*)-**1a** and (*R*)-**1b** (Figure 2).

CSP column: Chiralpak IA (eluent: hexane/EtOAc = 2/1; flow rate: 0.5 mL/min)



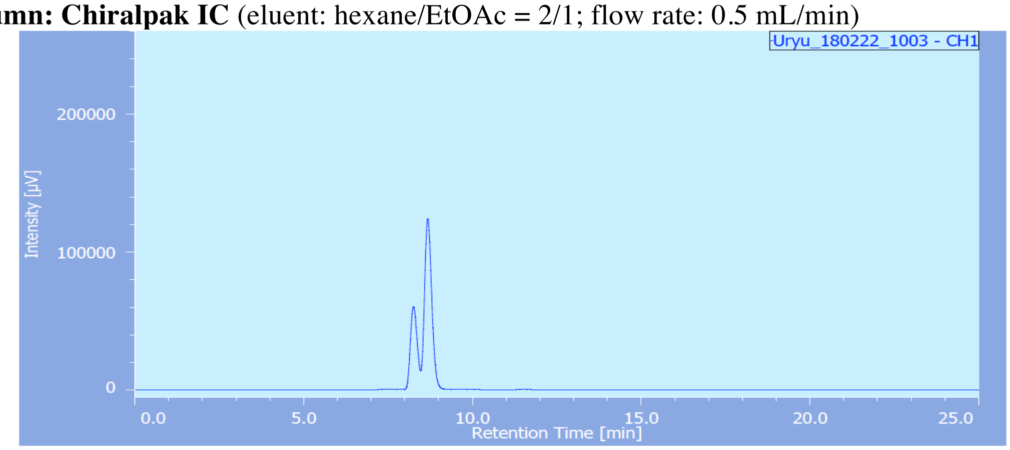
#	peak name	CH	tR [min]	area [µV•sec]	height [µV]	area %	height %	NTP	resolution	symmetry coefficient
1	(*R*)-**1b**	1	10.942	572997	39144	31.660	47.757	14254	19.007	0.906
2	(*R*)-**1a**	1	21.258	1236875	42821	68.340	52.243	13876	N/A	0.906

CSP column: Chiralpak IB (eluent: hexane/EtOAc = 2/1; flow rate: 0.5 mL/min)



#	peak name	CH	tR [min]	area [µV•sec]	height [µV]	area %	height %	NTP	resolution	symmetry coefficient
1	(*R*)-**1b**	1	9.908	173099	13663	40.458	43.282	15052	3.881	1.374
2	(*R*)-**1a**	1	11.233	254746	17905	59.542	56.718	15449	N/A	1.370

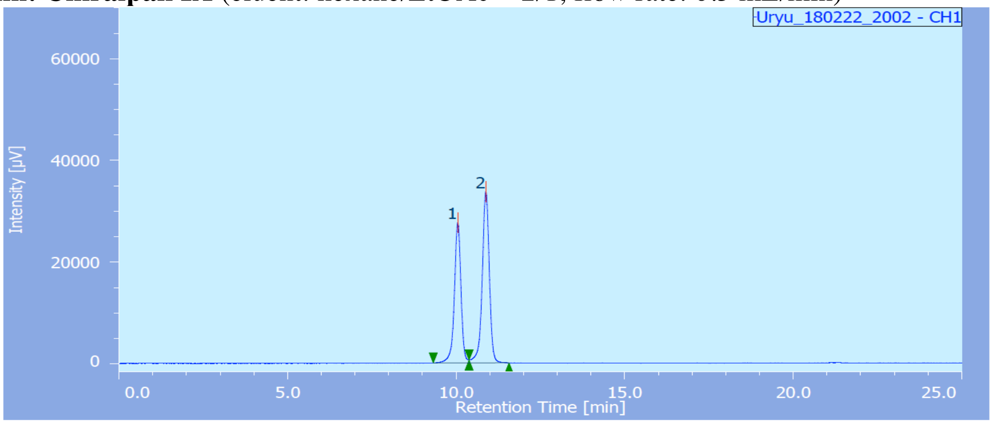
CSP column: Chiralpak IC (eluent: hexane/EtOAc = 2/1; flow rate: 0.5 mL/min)



#	peak name	CH	tR [min]	area [µV•sec]	height [µV]	area %	height %	NTP	resolution	symmetry coefficient
1	(*R*)-**1b**	1	8.258	845399	59985	31.276	32.642	7266	1.069	N/A
2	(*R*)-**1a**	1	8.675	1857651	123783	68.724	67.358	7757	N/A	N/A

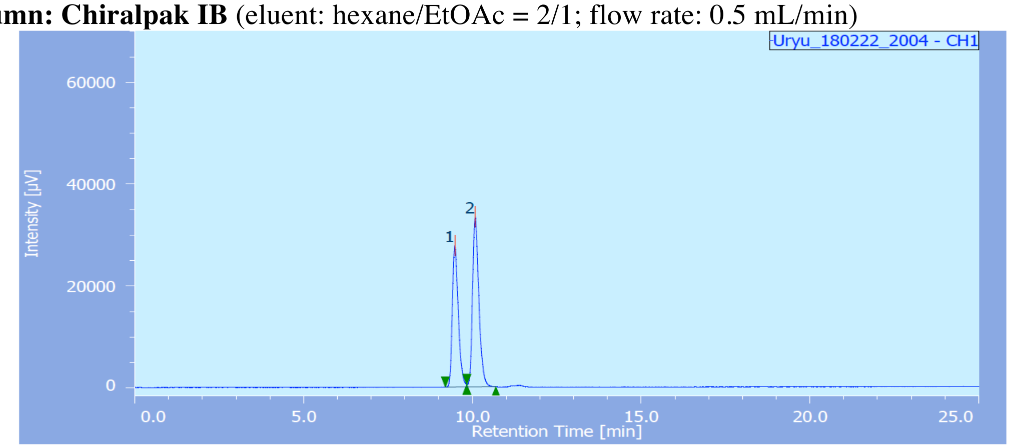
Figure S20. HPLC Traces for the Mixture of (*S*)-**1a** and (*S*)-**1b** (Figure 3).

CSP column: Chiralpak IA (eluent: hexane/EtOAc = 2/1; flow rate: 0.5 mL/min)



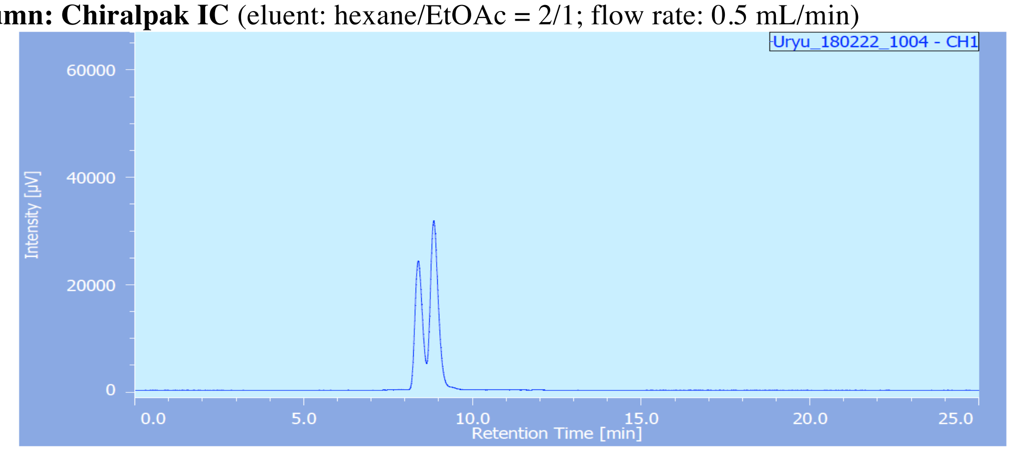
#	peak name	CH	tR [min]	area [µV•sec]	height [µV]	area %	height %	NTP	resolution	symmetry coefficient
1	(*S*)-**1b**	1	10.050	377482	27583	43.235	45.041	14069	2.366	0.908
2	(*S*)-**1a**	1	10.883	495620	33657	56.765	54.959	14035	N/A	0.910

CSP column: Chiralpak IB (eluent: hexane/EtOAc = 2/1; flow rate: 0.5 mL/min)



#	peak name	CH	tR [min]	area [µV•sec]	height [µV]	area %	height %	NTP	resolution	symmetry coefficient
1	(*S*)-**1b**	1	9.483	337403	27755	43.600	45.420	14837	1.867	1.313
2	(*S*)-**1a**	1	10.083	436465	33352	56.400	54.580	14676	N/A	1.318

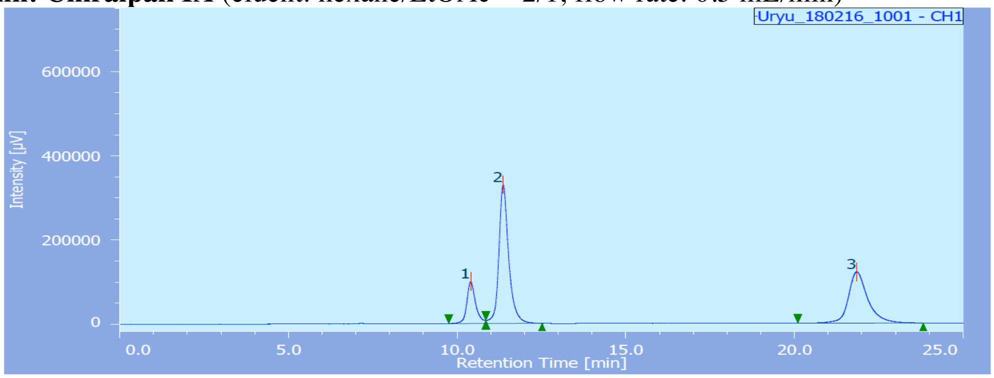
CSP column: Chiralpak IC (eluent: hexane/EtOAc = 2/1; flow rate: 0.5 mL/min)



#	peak name	CH	tR [min]	area [µV•sec]	height [µV]	area %	height %	NTP	resolution	symmetry coefficient
1	(*S*)-**1b**	1	8.400	370758	23977	41.212	43.234	6391	1.083	N/A
2	(*S*)-**1a**	1	8.858	528883	31481	58.788	56.766	6854	N/A	N/A

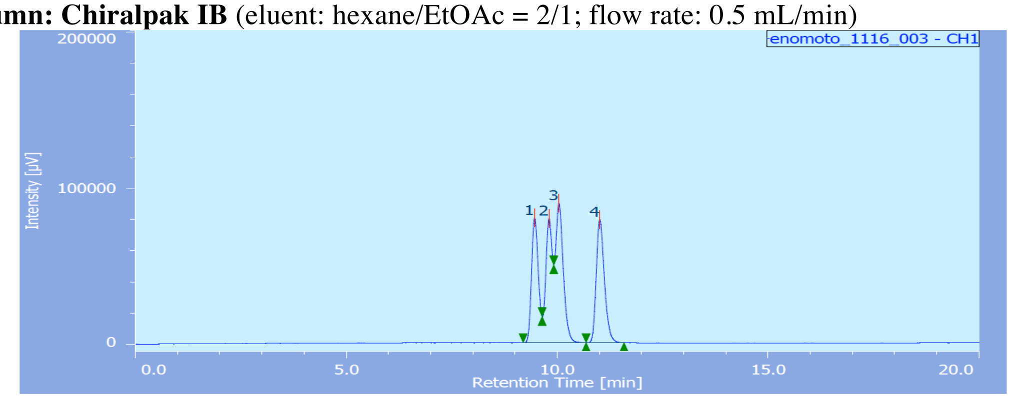
Figure S21. HPLC Traces for the Mixture of *rac*-**1a** and *rac*-**1b**.

CSP column: Chiralpak IA (eluent: hexane/EtOAc = 2/1; flow rate: 0.5 mL/min)



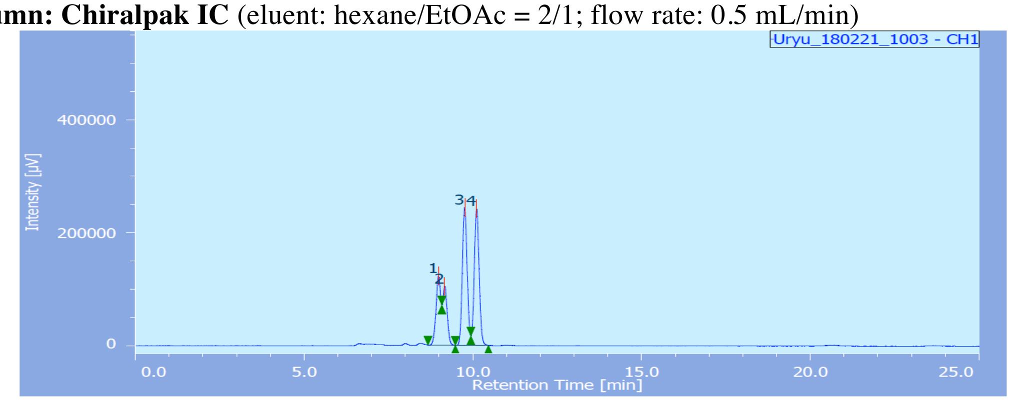
#	peak name	CH	tR [min]	area [μV•sec]	height [μV]	area %	height %	NTP	resolution	symmetry coefficient
1	(*S*)-**1b**	1	10.400	1798535	99183	13.474	18.026	8994	2.099	N/A
2	(*S*)-**1a**/(*R*)-**1b**	1	11.367	6676683	329260	50.020	59.841	8787	14.610	1.220
3	(*R*)-**1a**	1	21.850	4872773	121781	36.506	22.133	8396	N/A	1.261

CSP column: Chiralpak IB (eluent: hexane/EtOAc = 2/1; flow rate: 0.5 mL/min)



#	peak name	CH	tR [min]	area [μV•sec]	height [μV]	area %	height %	NTP	resolution	symmetry coefficient
1	(*S*)-**1b**	1	9.467	900980	79740	22.533	24.366	15498	N/A	N/A
2	(*R*)-**1b**	1	9.808	874040	79498	21.859	24.292	N/A	N/A	N/A
3	(*S*)-**1a**	1	10.042	1165921	89209	29.159	27.260	N/A	N/A	N/A
4	(*R*)-**1a**	1	11.008	1057509	78810	26.448	24.082	16192	N/A	1.281

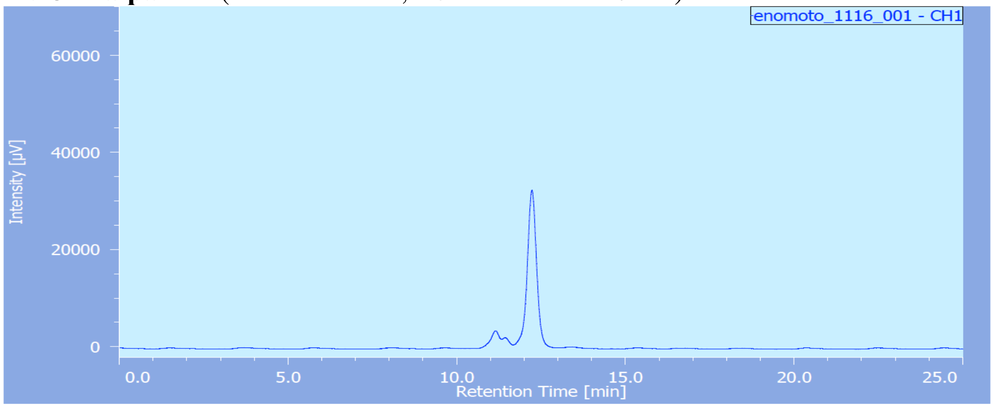
CSP column: Chiralpak IC (eluent: hexane/EtOAc = 2/1; flow rate: 0.5 mL/min)



#	peak name	CH	tR [min]	area [μV•sec]	height [μV]	area %	height %	NTP	resolution	symmetry coefficient
1	(*R*)-**1b**	1	8.983	1202429	122315	17.025	17.191	N/A	N/A	N/A
2	(*S*)-**1b**	1	9.167	988313	103970	13.993	14.613	N/A	N/A	N/A
3	(*R*)-**1a**	1	9.758	2434074	243775	34.463	34.261	21572	1.323	N/A
4	(*S*)-**1a**	1	10.108	2438068	241456	34.519	33.935	23337	N/A	N/A

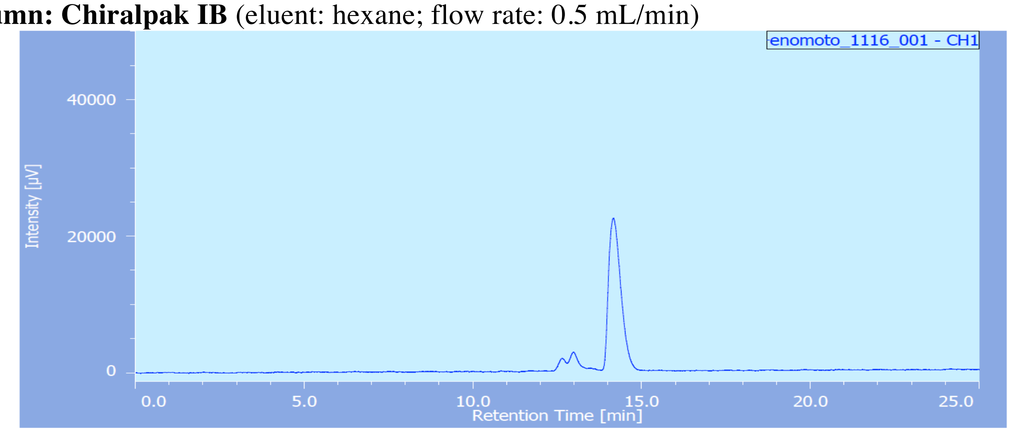
Figure S22. HPLC Trace for the Mixture of *rac*-**2a** and *rac*-**2b** (Figure 4).

CSP column: Chiralpak IB (eluent: hexane; flow rate: 0.5 mL/min)



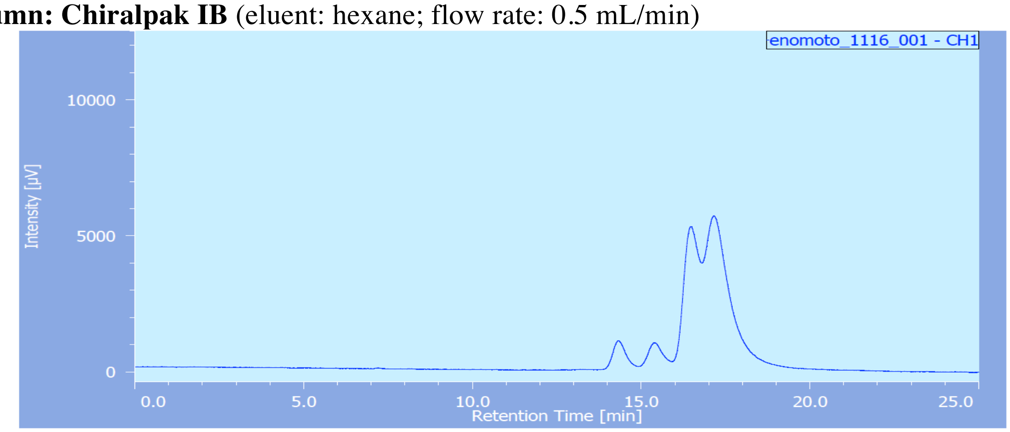
#	peak name	CH	tR [min]	area [µV•sec]	height [µV]	area %	height %	NTP	resolution	symmetry coefficient
1	*rac*-**2b**	1	11.150	100312	3602	13.617	9.984	2717	1.605	N/A
2	*rac*-**2a**	1	12.225	636362	32475	86.383	90.016	10066	N/A	0.939

CSP column: Chiralpak IB (eluent: hexane; flow rate: 0.5 mL/min)



#	peak name	CH	tR [min]	area [µV•sec]	height [µV]	area %	height %	NTP	resolution	symmetry coefficient
1	*rac*-**2b**	1	12.983	85043	2762	13.051	11.041	3040	1.449	1.135
2	*rac*-**2a**	1	14.158	566568	22251	86.949	88.959	6851	N/A	1.555

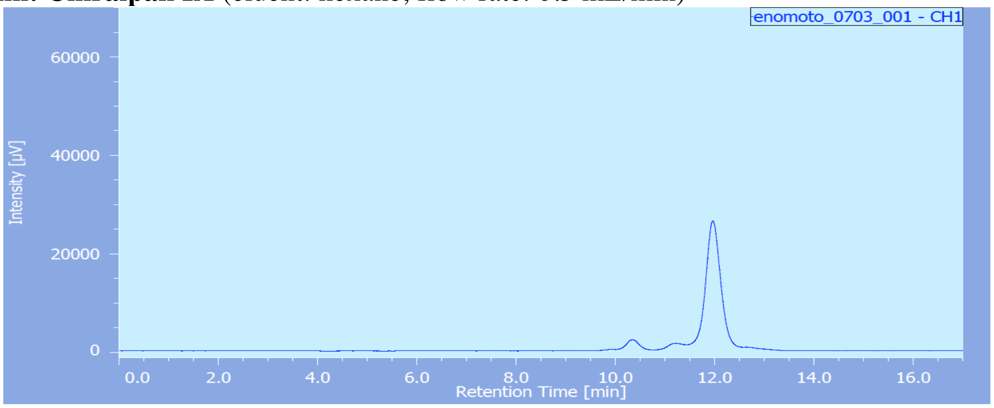
CSP column: Chiralpak IB (eluent: hexane; flow rate: 0.5 mL/min)



#	peak name	CH	tR [min]	area [µV•sec]	height [µV]	area %	height %	NTP	resolution	symmetry coefficient
1	(*R* or *S*)-**2b**	1	14.325	29886	1063	5.656	8.219	6333	1.341	N/A
2	(*S* or *R*)-**2b**	1	15.392	32970	989	6.239	7.647	4944	N/A	N/A
3	(*R* or *S*)-**2a**	1	16.467	173220	5247	32.780	40.564	N/A	N/A	N/A
4	(*S* or *R*)-**2a**	1	17.150	292351	5636	55.325	43.570	N/A	N/A	N/A

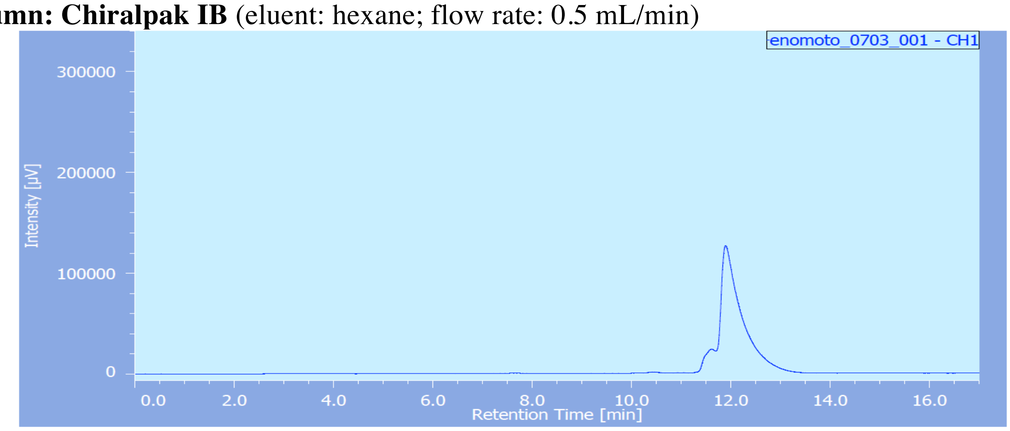
Figure S23. HPLC Traces for the Mixture of **3a**, (*Z*)-**3b**, and (*E*)-**3b** (Figure 5).

CSP column: Chiralpak IA (eluent: hexane; flow rate: 0.5 mL/min)



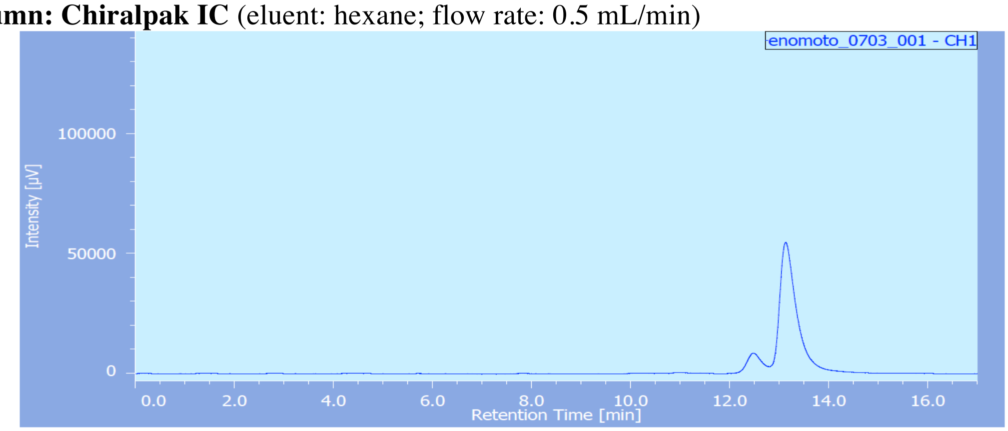
#	peak name	CH	tR [min]	area [µV•sec]	height [µV]	area %	height %	NTP	resolution	symmetry coefficient
1	(*Z*)-**3b**	1	10.350	39868	2120	6.517	7.273	7910	N/A	0.767
2	(*E*)-**3b**	1	11.217	27608	1188	4.513	4.077	N/A	N/A	N/A
3	**3a**	1	11.967	544273	25841	88.970	88.650	8048	N/A	0979

CSP column: Chiralpak IB (eluent: hexane; flow rate: 0.5 mL/min)



#	peak name	CH	tR [min]	area [µV•sec]	height [µV]	area %	height %	NTP	resolution	symmetry coefficient
1	(*Z*/*E*)-**3b**	1	11.608	357831	23652	8.908	15.785	N/A	N/A	N/A
2	**3a**	1	11.900	3658989	126187	91.092	84.215	5283	N/A	N/A

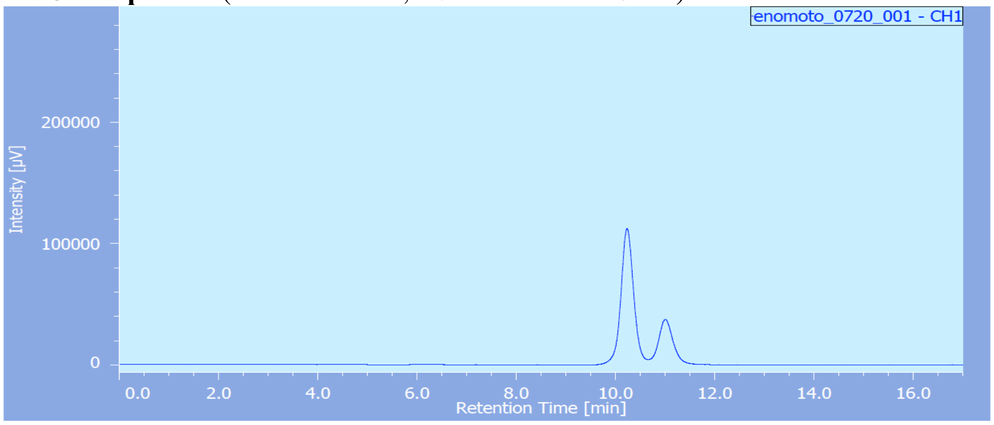
CSP column: Chiralpak IC (eluent: hexane; flow rate: 0.5 mL/min)



#	peak name	CH	tR [min]	area [µV•sec]	height [µV]	area %	height %	NTP	resolution	symmetry coefficient
1	(*Z*/*E*)-**3b**	1	12.483	177748	8439	11.852	13.404	7772	1.136	N/A
2	**3a**	1	13.133	1322018	54523	88.148	86.596	8168	N/A	N/A

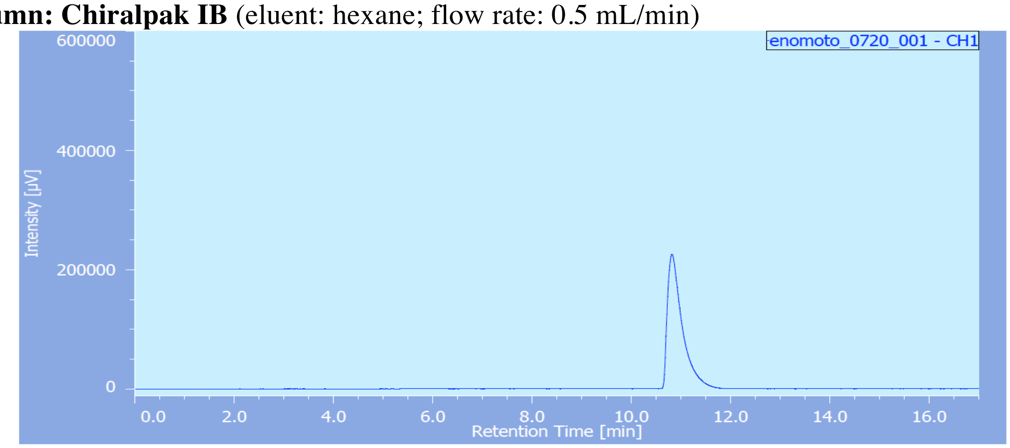
Figure S24. HPLC Trace for the Mixture of (Z)-**3b** and (E)-**3b** (Figure 6).

CSP column: Chiralpak IA (eluent: hexane; flow rate: 0.5 mL/min)



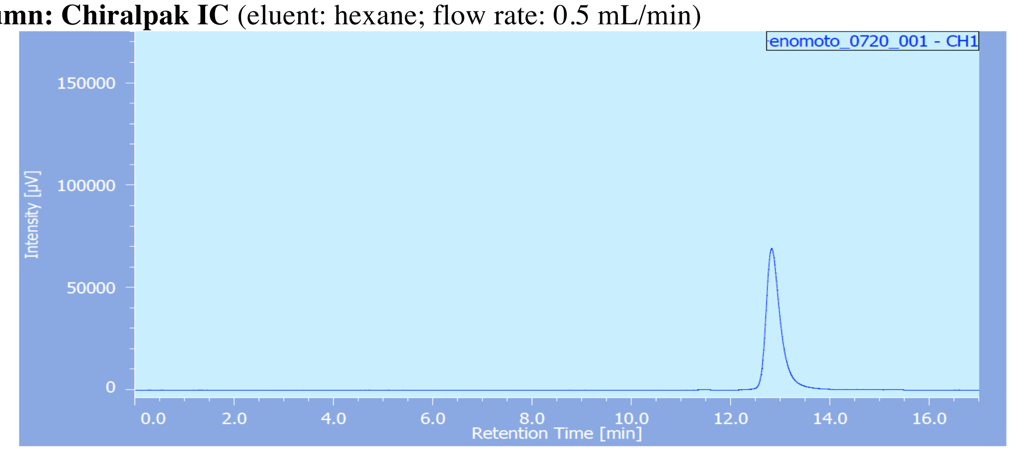
#	peak name	CH	tR [min]	area [µV•sec]	height [µV]	area %	height %	NTP	resolution	symmetry coefficient
1	(*Z*)-**3b**	1	10.233	2024334	112256	72.415	75.103	8230	1.602	1.048
2	(*E*)-**3b**	1	11.008	771138	37213	27.585	24.897	7206	N/A	N/A

CSP column: Chiralpak IB (eluent: hexane; flow rate: 0.5 mL/min)



#	peak name	CH	tR [min]	area [µV•sec]	height [µV]	area %	height %	NTP	resolution	symmetry coefficient
1	(*Z*/*E*)-**3b**	1	10.817	4663763	225321	100.000	100.000	7257	N/A	2.431

CSP column: Chiralpak IC (eluent: hexane; flow rate: 0.5 mL/min)



#	peak name	CH	tR [min]	area [µV•sec]	height [µV]	area %	height %	NTP	resolution	symmetry coefficient
1	(*Z*/*E*)-**3b**	1	12.825	1380399	68628	100.000	100.000	10715	N/A	1.551