Supplementary Information

Organocatalyzed epoxidation in the total synthesis of (-)-*trans*-, (+)-*trans*- and (+)-*cis*-disparlures

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General information: All of the chemicals were of a commercial quality and were purified in accordance with accepted procedures. Over anhydrous sodium sulphate, organic extracts were dried. Prior to use, solvents and reagents were purified and dried using conventional techniques. TLC was used to monitor the progress of the reactions using Merck Kieselgel 60 F254 precoated aluminium plates. On silica gel (100–200 mesh), column chromatography was carried out using an n-hexane/ethyl acetate combination. Except where otherwise noted, $CDCl_3$ was used as the recording medium for the 1H and 13C{^{1}H} NMR spectra on a JEOL ECS running at 500 and 126 MHz, respectively. Chemical shifts are provided in ppm with a TMS reference. SCIEX X500R QTOF was used to record mass spectral data (TOF-MS). Enantiomeric purity (ee) was determined by chiral HPLC analysis with a Waters instrument using Chiralpak IG chiral column.

An overview of previously reported enantioselective synthesis (starting only from achiral starting materials) for Disparlure.

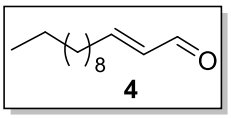
Table S1

Sr. No.	Synthesis	Enantioselective synthesis key step & enantiomeric purity	No. of steps	Overall yield (%)
1.	B. E. Rossiter, T. Katsuki, K. B. Sharpless, *J. Am. Chem. Soc.*, 1981, **103**, 464.	Sharpless asymmetric epoxidation, 91% ee (+)-*cis*-disparlure	5	33
2.	K. Mori, T. Ebata, *Tetrahedron* 1986, **42,** 3471.	Sharpless asymmetric epoxidation, 84.2% ee (+)-*cis*-disparlure	9	12
3.	V. N. Odinokov, V. R. Akhmetova, K. D. Khasanov, A. A. Abduvakhabov, A. V. Kuchin, N. I. Andreeva and G. A. Tolstikov. *Chem. Nat. Compd.*, 1989, **25**, 610.	Sharpless asymmetric epoxidation (+)-*cis*-disparlure	5	1.3
4.	S. Marczak, M. Masnyk, J. Wicha, *Liebigs Ann. Chem.*, 1990, 345.	Sharpless asymmetric epoxidation (+)-*cis*-disparlure	8	14.7
5.	E. Fukusaki, S. Senda, Y. Nakazono, H. Yuasa, T. Omata, *J. Ferment. Bioeng.*, 1992, **73**, 284.	Sharpless asymmetric epoxidation, 52.2% ee (+)-*cis*-disparlure	8	16.5
6.	G.-Q. Lin and C.-M. Zeng, *Acta Chim. Sin.*, 1992, **50**, 78.	Sharpless asymmetric epoxidation, 99.3% ee (+)-*cis*-disparlure	9	28.1
7.	L. H. Li, D. Wang, T. H. Chan, *Tetrahedron Lett.*, 1997, **38**, 101.	Sharpless asymmetric epoxidation, 52% ee (+)-*cis*-disparlure	4	30.4
8.	W. Zhigang, Z. Jianfeng, H. Peiqiang, *Chin. J. Chem.*, 2012, **30**, 23.	Sharpless asymmetric epoxidation, 80% ee (+)-*cis*-disparlure	6	28.7
9.	E. Keinan, S. C. Sinha, A. Sinhabagchi, Z. M. Wang, X. L. Zhang, K. B. Sharpless, *Tetrahedron Lett.*, 1992, **33**, 6411.	Sharpless asymmetric dihydroxylation, (-)-*trans*-disparlure 95% ee	8	50
		(+)-*trans*-disparlure 97% ee	8	51.3
		(+)-*cis*-disparlure 95% ee	8	43
10.	S. Y. Ko, *Tetrahedron Lett.*, 1994, **35**, 3601.	Sharpless asymmetric dihydroxylation, 90% ee (+)-*cis*-disparlure	9	44.5
11.	A. Sinha-Bagchi, S. C. Sinha, E. Keinan, *Tetrahedron Asymmetry*, 1995, **6**, 2889.	Sharpless asymmetric dihydroxylation, 98% ee (+)-*cis*-disparlure	10	29.7
12.	C. X. Zhang, S. J. Da, H. B. Zhang, B. Sun, Y. Li, *Acta Chim. Sinica*, 2007, **65**, 2433.	L-proline-catalyzed aldol reaction (+)-*cis*-disparlure	8	37.8

13	S. G. Kim, *Synthesis*, 2009, **14**, 2418.	Tandem asymmetric organocatalytic aminoxylation-allylation,		
		(-)-*trans*-disparlure dr 4:1 (anti/syn) anti 98% ee	8	40
		(+)-*cis*-disparlure dr 4:1 (anti/syn) anti 99% ee	7	23
14	Y. Garg, A. K. Tiwari, S. K. Pandey, *Tetrahedron Lett.*, 2017, **58**, 3344.	Organocatalytic MacMillan's self-aldol reaction, dr (4:1) syn/anti		
		(+)-*trans*-disparlure	11	20
		(+)-*cis*-disparlure	13	18.1
15	D. W. Klosowski, S. F. Martin, *Org. Lett.*, 2018, **20**, 1269.	Enantioselective iodolactonization 90% ee		
		(+)-*cis*-disparlure	6	33.1
16	G. R. Pinnelli, M. Terrado, N. K. Hillier, D. R. Lance, E. Plettner, *Eur. J. Org. Chem.*, 2019, **40**, 6807.	MacMillan's SOMO-activated α-chlorination, 99% ee		
		(+)-*cis*-disparlure	5	53
17	**This work**	Organocatalyzed asymmetric Jørgensen epoxidation >99% ee		
		(-)-*trans* and (+)-*trans*-disparlure	5	46.5
		(+)-*cis*-disparlure	13	33.7

EXPERIMENTAL SECTION

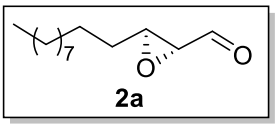
(*E*)-Tridec-2-enal, 4



To a stirred solution of oxalyl chloride (0.79 mL, 8.72 mmol) in dry DCM (15 mL) at –78 °C was added dropwise DMSO (1.28 mL, 18.01 mmol) in CH_2Cl_2 (15 mL) and stirred for 0.5 h. The undecanol **5** (1.0 g, 5.81 mmol) in dry DCM (15 mL) was mixed over 15 min. The reaction mixture was agitated for 2 h at the same temperature then triethyl amine (3.56 mL, 25.56 mmol) was added and stirred for additional 0.5 hours at –60 °C. The organic layer was separated after the reaction mixture was added to 40 mL of sat. $NaHCO_3$ and aqueous layer was extracted with DCM (3 x 20 mL). The organic extract was washed with brine, dried over Na_2SO_4, and concentrated under vacuum, and used in the subsequent step without further purification.

The above aldehyde and (formylmethylene)triphenyl-phosphine (2.11 g, 6.97 mmol) was dissolved in toluene (15.0 mL) and agitated at 70 °C under argon for 14 h. The resultant solution was cooled to room temperature and concentrated in *vacuo* once the reaction was completed (monitored by TLC). The *n*-hexane (20 mL) was used to dilute the residue, and the resulting solution was then filtered through a coarse sintered funnel. The filtrate was concentrated in *vacuo* and purified by column chromatography over silica gel (EtOAc:hexane, 0.5:9.5) to afford the *α,β*-unsaturated aldehyde **4** (934 mg) as a light yellow oil in 82% yield (over two steps). ^{1}H NMR (500 MHz, $CDCl_3$) δ: 9.51 (d, *J* = 7.9 Hz, 1H), 6.85 (dt, *J* = 15.2, 6.8 Hz, 1H), 6.12 (dd, *J* = 15.6, 7.9 Hz, 1H), 2.34 (dd, *J* = 14.0, 6.9 Hz, 2H), 1.54–1.48 (m, 2H), 1.27 (brs, 14H), 0.88 (t, *J* = 6.8 Hz, 3H); ^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ: 194.3, 159.2, 133.1, 32.8, 32.0, 29.7, 29.6, 29.4, 29.4, 29.2, 27.9, 22.8, 14.2. HRMS (ESI$^+$) m/z; $C_{13}H_{24}O$ $[M + H]^+$ calcd. 197.1900; found 197.1901.

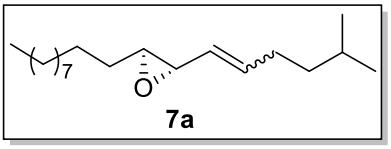
(*2R,3S*)-3-Decyloxirane-2-carbaldehyde, 2a



To a stirred solution of *α,β*-unsaturated aldehyde **4** (392 mg, 2.0 mmol) in $CHCl_3$ (10 mL) at 4 °C was added (*R*)-2-(bis(3,5-bis(trifluoromethyl)-phenyl)((tertbutyldimethyl-silyl)oxy)methyl)pyrrolidine **6d** (127 mg, 0.20 mmol, 10 mol%) followed by 35% aq. H_2O_2 (0.27 mL, 2.40 mmol) addition and stirred the reaction mixture for 9 h. The reaction mixture was diluted with water after completion of the reaction (as monitored by TLC) and then extracted with DCM (3 x 10 mL). The organic layer was washed with brine, dried over Na_2SO_4 (anhyd.), concentrated in *vacuo* and then purified by column chromatography over silica gel (EtOAc:hexane, 1.0:9.0) to afford the (*R,S*)-epoxy aldehyde **2a** (385 mg, 91%) as a colourless oil. $[\alpha]_D^{25}$ +20.10 (*c* 1.0, CH_2Cl_2); ^{1}H NMR (500 MHz, $CDCl_3$) δ: 9.00 (d, *J* = 6.4 Hz, 1H), 3.22 (t, *J* = 4.7 Hz, 1H), 3.12 (d, *J* = 5.4 Hz, 1H), 1.66–1.63 (m, 2H), 1.47–1.43 (m, 2H), 1.25 (brs, 14H), 0.87 (t, *J* = 6.7 Hz, 3H); ^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ: 198.6, 59.2, 56.9, 32.0, 31.3, 29.6, 29.6, 29.5, 29.4, 29.3, 25.8, 22.8, 14.2. HRMS (ESI$^+$) m/z; $C_{13}H_{24}O_2$ $[M + H]^+$ calcd. 213.1849; found 213.1853. The enantiomeric purity was determined by HPLC analysis of the

corresponding 3,5-dinitrobenzoyl ester (chiral column-Chiralpak IG, 4.6 x 250 mm; 30 °C; mobile phase hexane/EtOH, 8.5:1.5; flow rate 1 mL/min; PDA detection at 254 nm): major enantiomer: *t*R = 22.131 min, minor enantiomer: *t*R = 25.170 min.

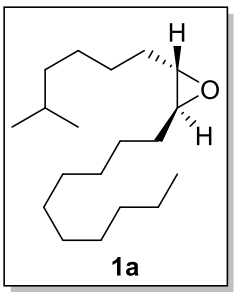
2-Methyl-(*7S,8S*)-epoxyoctadec-5-ene, 7a



7a

To a stirred solution of freshly prepared 4-methylpentyl triphenylphosphonium bromide (389 mg, 0.90 mmol) in dry THF (10 mL) at –78 °C was added *n*-BuLi (0.45 mL, 1.12 mmol, 2.5 M in hexane) and stirred under inert atmosphere for 1 h, during which time the solution became dark orange. Further, (*R,S*)-epoxy aldehyde **2a** (159 mg, 0.75 mmol) in THF (7 mL) was added dropwise, and the reaction was maintained at –78 °C for 4 h before being allowed to warm to room temperature for 7 h. The reaction mixture was then quenched with saturated aq. $NaHCO_3$ solution and extracted with EtOAc (3 x 10 mL). The combined organic extract were washed with brine, dried over anhydrous Na_2SO_4, concentrated in *vacuo* and then purified by column chromatography over silica gel (EtOAc:hexane, 0.2:0.98) to afford the (*S,S*)-epoxy olefin **7a** (170 mg) as a colourless oil in 81% yield. ^{1}H NMR (500 MHz, $CDCl_3$) δ: 5.68 (dt, *J* = 10.9, 7.6 Hz, 1H), 5.03 (dd, *J* = 10.7, 9.3 Hz, 1H), 3.34 (dd, *J* = 8.7, 1.5 Hz, 1H), 2.81 (td, *J* = 5.7, 1.9 Hz, 1H), 2.24–2.16 (m, 2H), 1.60–1.55 (m, 3H), 1.49–1.42 (m, 2H), 1.26 (brs, 16H), 0.91–0.88 (m, 9H); ^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ: 136.6, 127.0, 60.3, 54.6, 38.9, 32.2, 32.0, 29.7, 29.7, 29.6, 29.4, 27.7, 26.0, 25.7, 22.8, 22.6, 14.2. HRMS (ESI$^+$) m/z; $C_{19}H_{36}O$ $[M + H]^+$ calcd. 281.2839; found 281.2842.

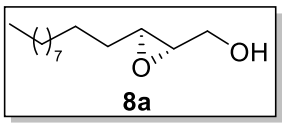
(-)-*trans*-Disparlure, 1a



1a

To a stirred solution degassed benzene (5 mL) and Wilkinson's catalyst $(Ph_3P)_3RhCI$ (0.73 mg, 0.25 mmol) under H_2 at atmospheric pressure was stirred until the initially intense red colour of the solution turned yellow orange (approximately 1.5 h). The transformation of colour showed that Wilkinson's catalyst was successfully reduced. After the colour change, (*S,S*)-epoxy olefin **7a** (100 mg, 0.35 mmol) in benzene (2 mL) was added and the reaction was stirred under H_2 at atmospheric pressure for 3 h. *n*-Hexane (5 mL) was added to precipitate the Rhodium reagent and solid was resuspended. The reaction mixture filtered through a Celite pad, washed with additional EtOAc (10 mL), dried over anhydrous Na_2SO_4, concentrated under reduced pressure and then purified by chromatography over silica gel (EtOAc:hexane, 0.2:0.98) which afforded the (-)-*trans*-disparlure **1a** (77 mg) as a colourless oil in 77% yield. $[\alpha]_D^{25}$ –26.0 (*c* 1.9, CCl_4); [Lit.[1] $[\alpha]_D^{25}$ –25.80 (*c* 1.9, CCl_4)]; ^{1}H NMR (500 MHz, $CDCl_3$) δ: 2.66–2.64 (m, 2H), 1.55 – 1.49 (m, 4H), 1.43–1.39 (m, 3H), 1.26 (brs, 18H), 1.20–1.15 (m, 2H), 0.89–0.86 (m, 9H); ^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ: 59.0, 39.0, 32.3, 32.0, 32.3, 29.8, 29.7, 29.6, 29.4, 28.0, 27.7, 26.4, 26.2, 22.8, 22.7, 14.2. HRMS (ESI$^+$) m/z; $C_{19}H_{38}O$ $[M + H]^+$ calcd. 283.2996; found 283.2994.

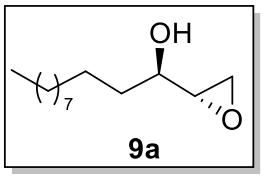
((*2S,3S*)-3-Decyloxiran-2-yl)methanol, 8a



8a

To a stirred solution of (*R,S*)-epoxy aldehyde **2a** (212 mg, 1.0 mmol) in methanol (5 mL) was added $NaBH_4$ (74 mg, 2.0 mmol) at 0 °C, and the reaction was agitated for 0.5 h. With saturated aq. NH_4Cl, the reaction was quenched, and EtOAc (3 x 5 mL) was used to extract it. The organic layer was separated, dried over Na_2SO_4 (anhyd.), concentrated in *vacuo* and then purified by column chromatography over silica gel (EtOAc:hexane, 1.5:8.5) to afford the 2, 3-epoxy alcohol **8a** (209 mg) as a white solid in 98% yield. m.p. 61-63 °C; $[\alpha]_D^{25}$ –42.50 (*c* 2.0, $CHCl_3$); ^{1}H NMR (500 MHz, $CDCl_3$) δ: 3.91 (d, *J* = 12.5 Hz, 1H), 3.62 (d, *J* = 12.4 Hz, 1H), 2.97–2.91 (m, 2H), 1.92 (s, 1H), 1.59–1.55 (m, 2H), 1.48–1.41 (m, 2H), 1.26 (brs, 14H), 0.88 (t, *J* = 7.0 Hz, 3H); ^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ: 61.7, 58.4, 56.0, 31.9, 31.5, 29.5, 29.5, 29.5, 29.4, 29.3, 25.9, 22.6, 14.1. HRMS (ESI^+) m/z; $C_{13}H_{26}O_2$ $[M + H]^+$ calcd. 215.2006; found 215.2009.

(*R*)-1-((*S*)-Oxiran-2-yl)undecan-1-ol, 9a To a stirred solution of 2, 3-epoxy alcohol **8a** (150 mg, 0.70 mmol) in DCM (5 mL) at 0 °C were added triethylamine (0.19 mL, 1.40 mmol) and a catalytic quantity of DMAP followed by slow addition of mesyl chloride (0.08 mL, 1.05 mmol). Following a 2 h reaction time, it was quenched with 10 mL of ice-cold water and extracted with DCM (3 x 5 mL). The mixed organic extracts were treated with ice-cold 1N HCl, saturated aqueous $NaHCO_3$ and brine, dried over anhydrous Na_2SO_4, concentrated in *vacuo*, and then used for the subsequent step without further purification.

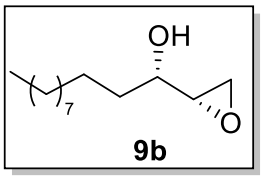


9a

To a stirred solution of the above synthesized 2, 3-epoxy 1-sulfonate ester intermediate in 5 mL of THF (60% aq.) was added 0.05 mL of $HClO_4$ (70% aq.). The reaction mixture was stirrered at room temperature for 12 hours then diluted with EtOAc (5 mL) and washed with water. The organic phase was dried over anhydrous Na_2SO_4, concentrated in *vacuo* to afford diol mesylate derivative, which was used as such for the next step without further purification.

To the above synthesized diol mesylate intermediate in methanol (5 mL) was added K_2CO_3 (144 mg, 1.05 mmol) at 15 °C. The reaction was stirred for 3 h at the same temperature, quenched with water and extracted with EtOAc (3 x 5 mL). The organic layer was washed with brine, dried over anhydrous Na_2SO_4, concentrated in *vacuo* and then purified by column chromatography over silica gel (EtOAc:hexane, 1:9) to furnish the 1, 2-epoxy alcohol **9a** (123 mg) as a colourless oil in 82% yield (over three steps). $[\alpha]_D^{25}$ –47.9 (*c* 0.6, CH_3OH); [Lit.[2] $[\alpha]_D^{25}$ –47.50 (*c* 0.6, CH_3OH)]; ^{1}H NMR (500 MHz, $CDCl_3$) δ: 3.78–3.76 (m, 1H), 2.98–2.91 (m, 1H), 2.75–2.73 (m, 1H), 2.69–2.63 (m, 1H), 1.70 (s, 1H), 1.48–1.41 (m, 2H), 1.26 (brs, 16H), 0.81 (t, *J* = 6.6 Hz, 3H); ^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ: 68.5, 54.6, 43.5, 33.5, 32.0, 29.8, 29.7, 29.6, 29.4, 25.4, 22.8, 14.2. HRMS (ESI^+) m/z; $C_{13}H_{27}O_2$ $[M + H]^+$ calcd. 215.2006; found 215.2012.

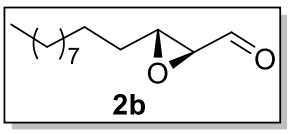
(*S*)-1-((*S*)-Oxiran-2-yl)undecan-1-ol, 9b



To a stirred solution of 1, 2-epoxy alcohol **9a** (100 mg, 0.46 mmol) in 5 mL of toluene were added PPh_3 (361 mg, 1.38 mmol), 4-nitrobenzoic acid (230 mg, 1.38 mmol) and DIAD (278 mg, 1.38 mmol) at 0 ^oC. The reaction mixture was then warm at room temperature and stirred for 2 h. After completion of reaction (monitored by TLC) the reaction was diluted with water and extracted with EtOAc (3 x 5 mL). The organic layer was washed with brine and dried over anhydrous Na_2SO_4 before being concentrated in *vacuo* to afford the required ester intermediate, which was used directly for the next step without any further purification.

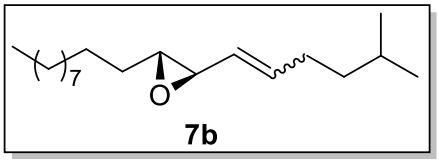
To the above ester intermediate in H_2O:CH_3OH:THF (1:2:3, 4 mL) solution was added $LiOH.H_2O$ (37 mg, 0.90 mmol) and stirred the reaction mixture for 1 h at room temperature. The reaction was quenched with water and extracted with EtOAc (3 x 5 mL). The organic layer was washed with brine, dried over Na_2SO_4 (anhyd.), concentrated in *vacuo* and then purified by column chromatography over silica gel using (EtOAc:hexane, 1:9) as eluent to afford the 1, 2-epoxy alcohol **9b** (92 mg) as a colourless oil in 92% yield (over two steps). $[\alpha]_D^{25}$ –84.0 (*c* 1.0, CH_3OH); [Lit.[2] $[\alpha]_D^{25}$ –83.9 (*c* 1.0, CH_3OH)]; ^1H NMR (500 MHz, $CDCl_3$) δ: 3.46–3.41 (m, 1H), 2.98 (dd, *J* = 7.5, 4.3 Hz, 1H), 2.82 (t, *J* = 4.1 Hz, 1H), 2.73–2.70 (m, 1H), 1.91 (d, *J* = 6.6 Hz, 1H), 1.63–1.58 (m, 2H), 1.49–1.45 (m, 1H), 1.26 (brs, 15H), 0.88 (t, *J* = 7.0 Hz, 3H); $^{13}C\{^1H\}$ NMR (126 MHz, $CDCl_3$) δ: 71.7, 55.4, 45.2, 34.4, 31.9, 29.6, 29.5, 29.3, 25.3, 22.7, 14.1. HRMS (ESI^+) m/z; $C_{13}H_{27}O_2$ $[M + H]^+$ calcd. 215.2006; found 215.2004.

(*2S,3R*)-3-Decyloxirane-2-carbaldehyde, 2b



(*S,R*)-epoxy aldehyde **2b** was prepared from *α,β*-unsaturated aldehyde **4** (196 mg, 1.0 mmol) in presence of (*S*)-2-(bis(3,5-bis(trifluoromethyl)phenyl)((tertbutyldimethylsilyl)oxy)meth-yl) pyrolidine (10 mol%) by employing the method outlined for ((*R,S*)-epoxy aldehyde **2a** (193 mg) as a colourless oil in 91% yield. $[\alpha]_D^{25}$ –20.50 (*c* 1.0, CH_2Cl_2); ^1H NMR (500 MHz, $CDCl_3$) δ: 9.02 (d, *J* = 6.3 Hz, 1H), 3.23 (ddd, *J* = 6.1, 5.1, 2.0 Hz, 1H), 3.13 (dd, *J* = 6.3, 2.0 Hz, 1H), 1.70–1.60 (m, 2H), 1.51–1.42 (m, 2H), 1.26 (brs, 14H), 0.88 (t, *J* = 7.0 Hz, 3H); $^{13}C\{^1H\}$ NMR (126 MHz, $CDCl_3$) δ: 198.6, 59.3, 56.9, 32.0, 31.3, 29.8, 29.6, 29.6, 29.5, 29.4, 29.3, 25.9, 22.8, 14.2. The enantiomeric purity was determined by HPLC analysis of the correspoinding 3,5-dinitrobenzoyl ester (Chiralpak IG chiral column, 4.6 × 250 mm; mobile phase hexane/EtOH, 8.5:1.5; flow rate 1 mL/min; 30 °C; PDA detection at 254 nm): minor enantiomer: *t*R = 24.551 min, major enantiomer: *t*R = 28.439 min.

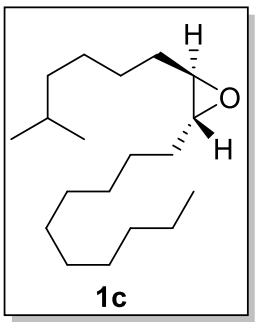
2-Methyl-(*7R,8R*)-epoxyoctadec-5-ene, **7b**



7b

(*R,R*)-epoxy olefin **7b** was prepared from (*S,R*)-epoxy aldehyde **2b** (150 mg, 0.70 mmol) by employing the method outlined for (*S,S*)-epoxy olefin **7a** in 81% yield (160 mg) as a colourless oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ: 5.68 (dt, *J* = 10.9, 7.5 Hz, 1H), 5.05 –5.01 (m, 1H), 3.35–3.33 (m, 1H), 2.81–2.80 (m, 1H), 2.26–2.16 (m, 2H), 1.60–1.55 (m, 3H), 1.47–1.42 (m, 2H), 1.26 (brs, 16H), 0.91–0.86 (m, 9H); ^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ: 136.6, 127.1, 60.3, 54.6, 38.9, 32.2, 32.0, 29.7, 29.5, 29.4, 27.7, 26.0, 25.7, 22.8, 22.6, 14.2.

(+)-*trans*-Disparlure, 1c



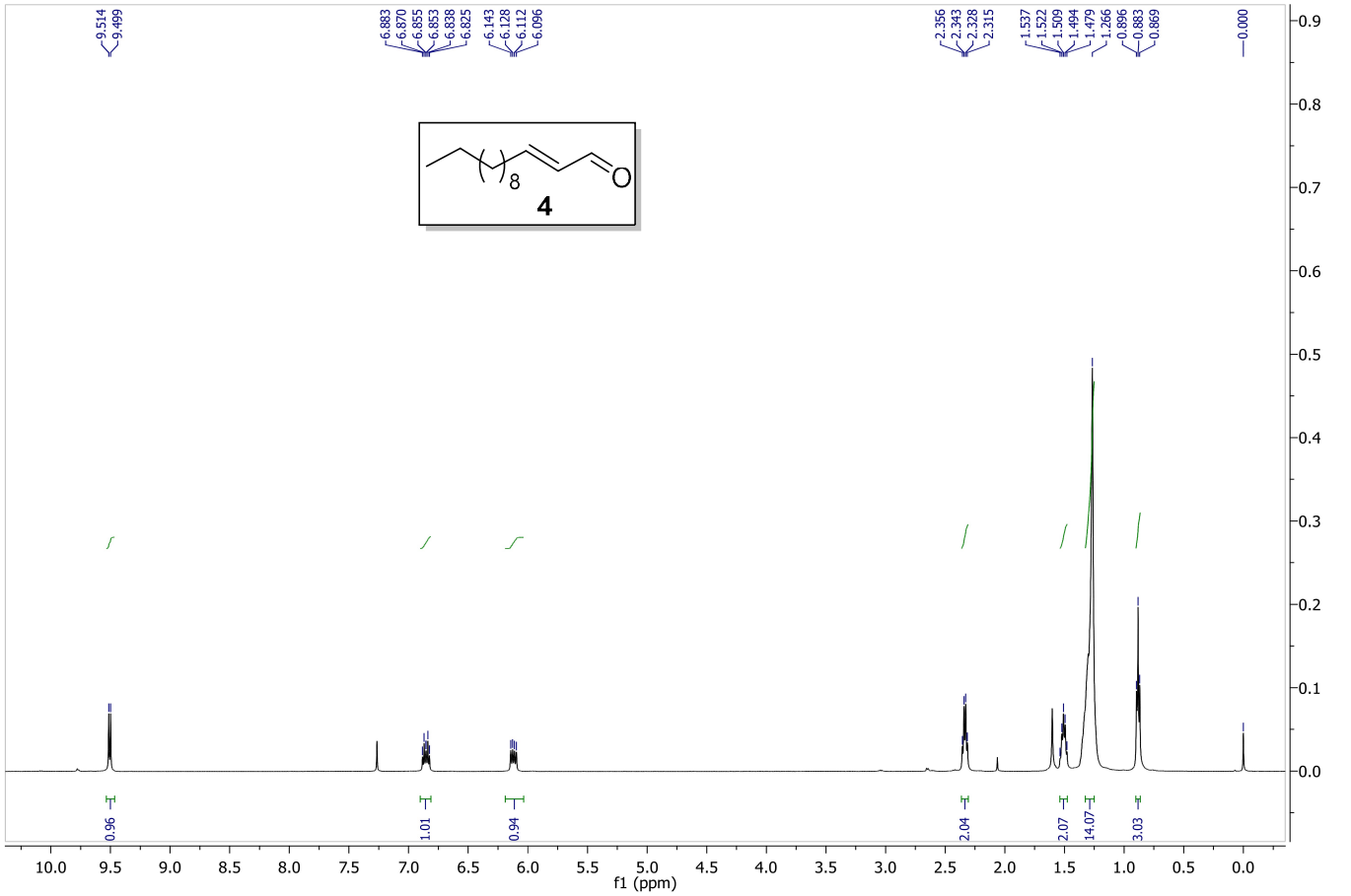
1c

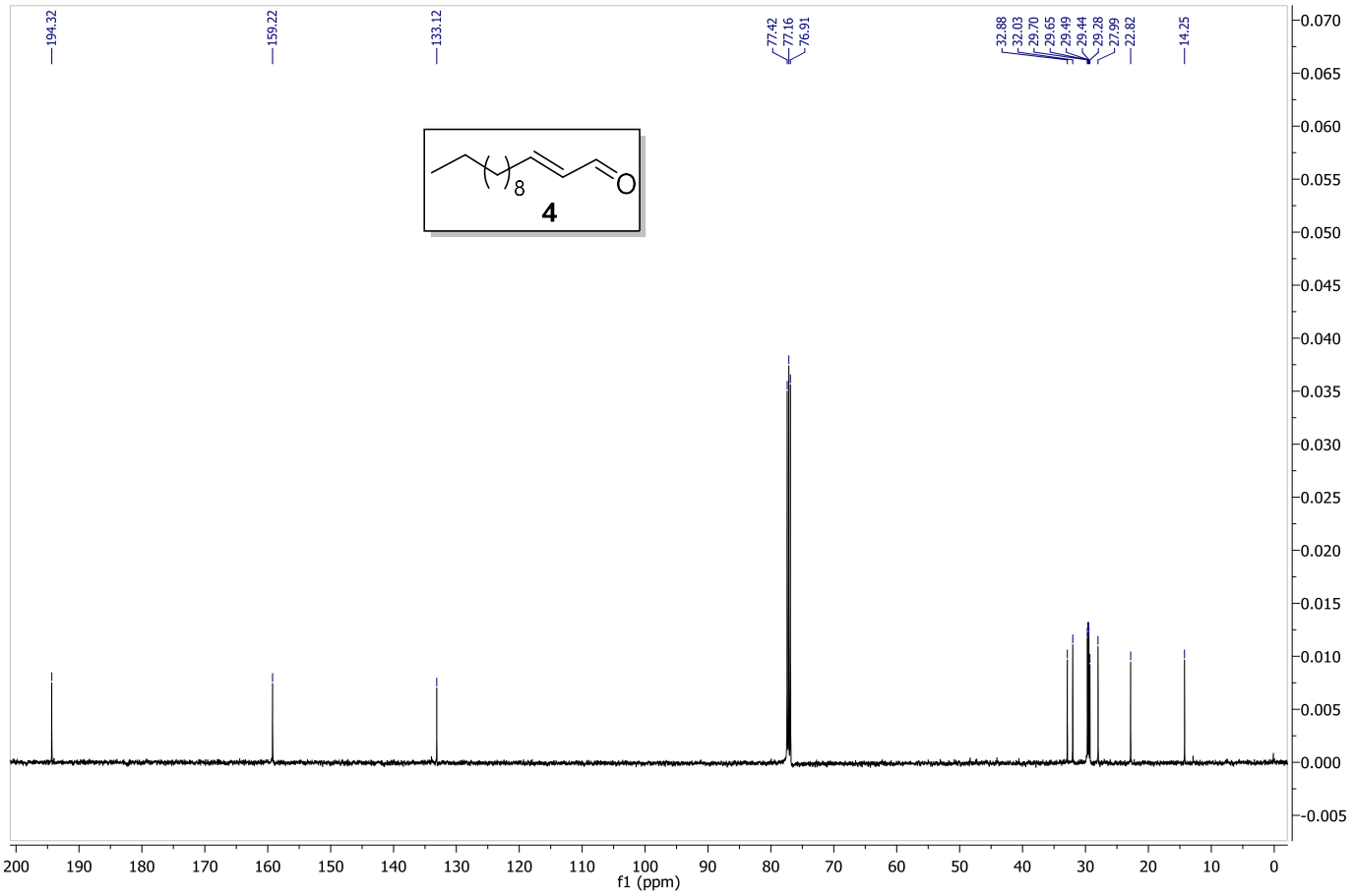
(+)-*trans*-Disparlure **1c** was prepared from (*R,R*)-epoxy olefin **7b** (70 mg, 0.25 mmol) by following the same procedure as described for (-)-*trans*-disparlure **1a** in 77% yield (53 mg) as a colourless oil. $[\alpha]_D^{25}$ +27.70 (*c* 0.5, CCl_4); [Lit.[2] $[\alpha]_D^{25}$ +27.80 (*c* 0.5, CCl_4)];^{1}H NMR (500 MHz, $CDCl_3$) δ: 2.66–2.64 (m, 2H), 1.53–1.50 (m, 4H), 1.43–1.39 (m, 3H), 1.26 (brs, 18H), 1.19–1.15 (m, 2H), 0.89–0.86 (m, 9H); ^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ: 59.0, 39.0, 32.3, 32.3, 32.0, 29.74, 29.71, 29.6, 29,4, 28.0, 27.3, 26.4, 26.2, 22.8, 22.7, 14.2. HRMS (ESI^{+}) m/z; $C_{19}H_{38}O$ $[M + H]^{+}$ calcd. 283.2996; found 283.2991.

References:

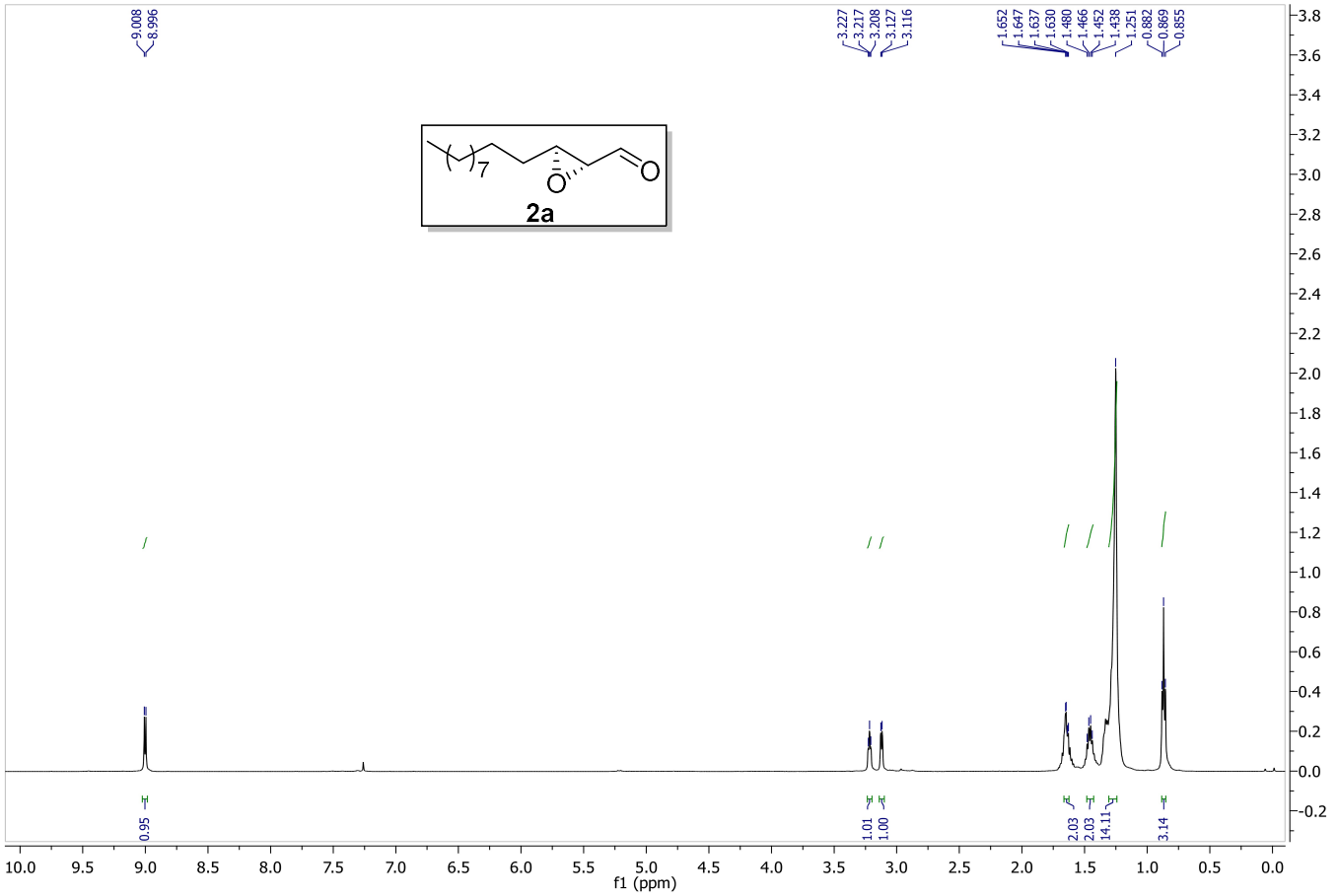
1. S. G. Kim, *Synthesis*, 2009, **14**, 2418.
2. Y. Garg, A. K. Tiwari, S. K. Pandey, *Tetrahedron Lett.,* 2017, **58**, 3344.

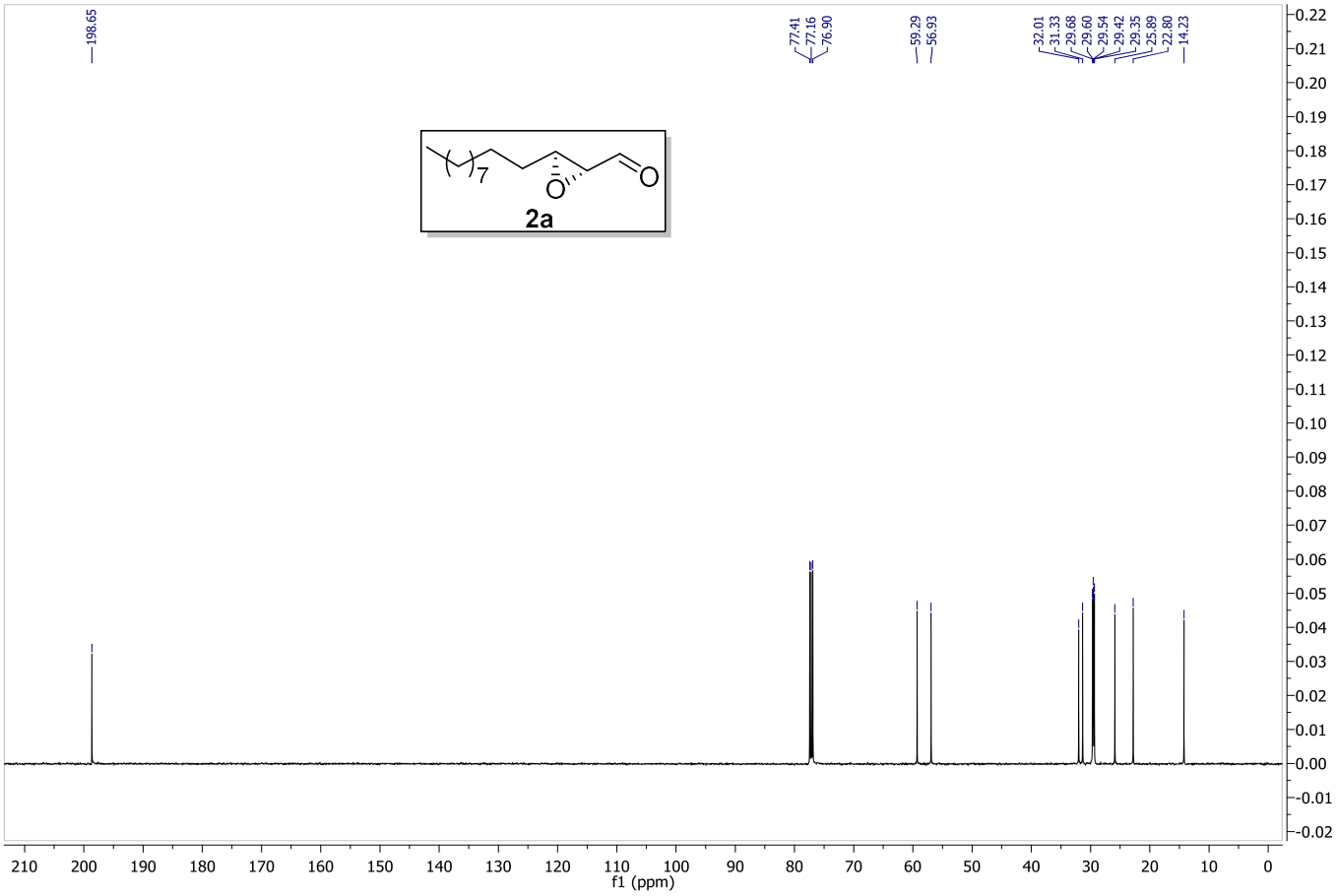
^{1}H NMR (500 MHz, $CDCl_3$) and $^{13}C\{^{1}H\}$ NMR (126 MHz, $CDCl_3$), **4**





^{1}H NMR (500 MHz, $CDCl_3$) and ^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$), **2a**





HPLC Report of Recemic, 2a

Empower™ 3 SOFTWARE

Dis Rec

SAMPLE INFORMATION

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Injection Volume:	20.00 ul	Channel Name:	2998 Ch1 254nm@1.2nm
Run Time:	120.0 Minutes	Proc. Chnl. Descr.:	2998 Ch1 254nm@1.2nm
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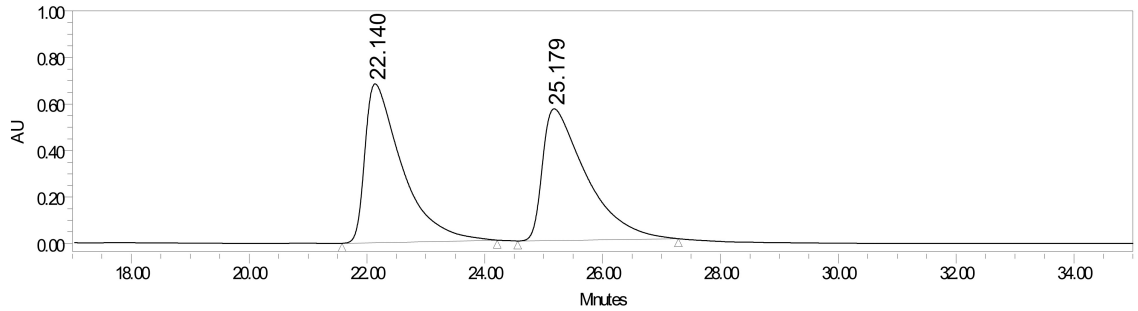
Date Processed:	4/30/2022 1:19:47 PM IST		



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Reported by User: System
Report Method: Dis Rec
Report Method ID: 1693 1693
Page: 1 of 1

Project Name: SKF
Date Printed:
7/4/2022
3:57:40 PM Asia/Calcutta

Empower™ 3 SOFTWARE

Dis Rec

SAMPLE INFORMATION

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Injection #:	2	Processing Method:	dis chiral
Injection Volume:	20.00 ul	Channel Name:	2998 Ch1 254nm@1.2nm
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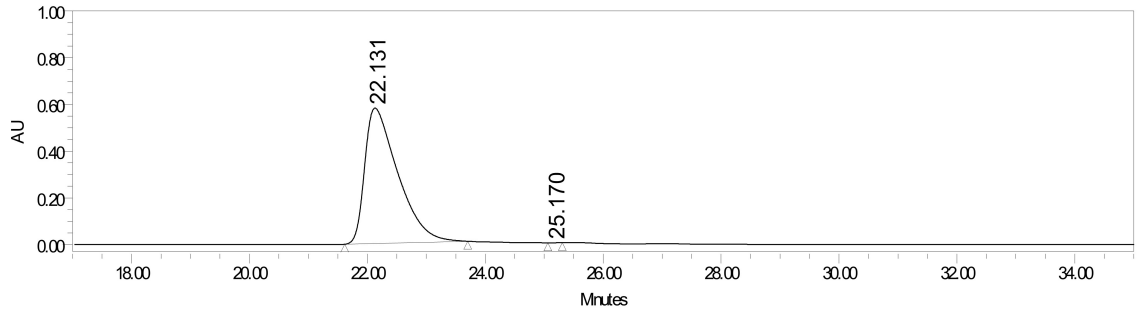

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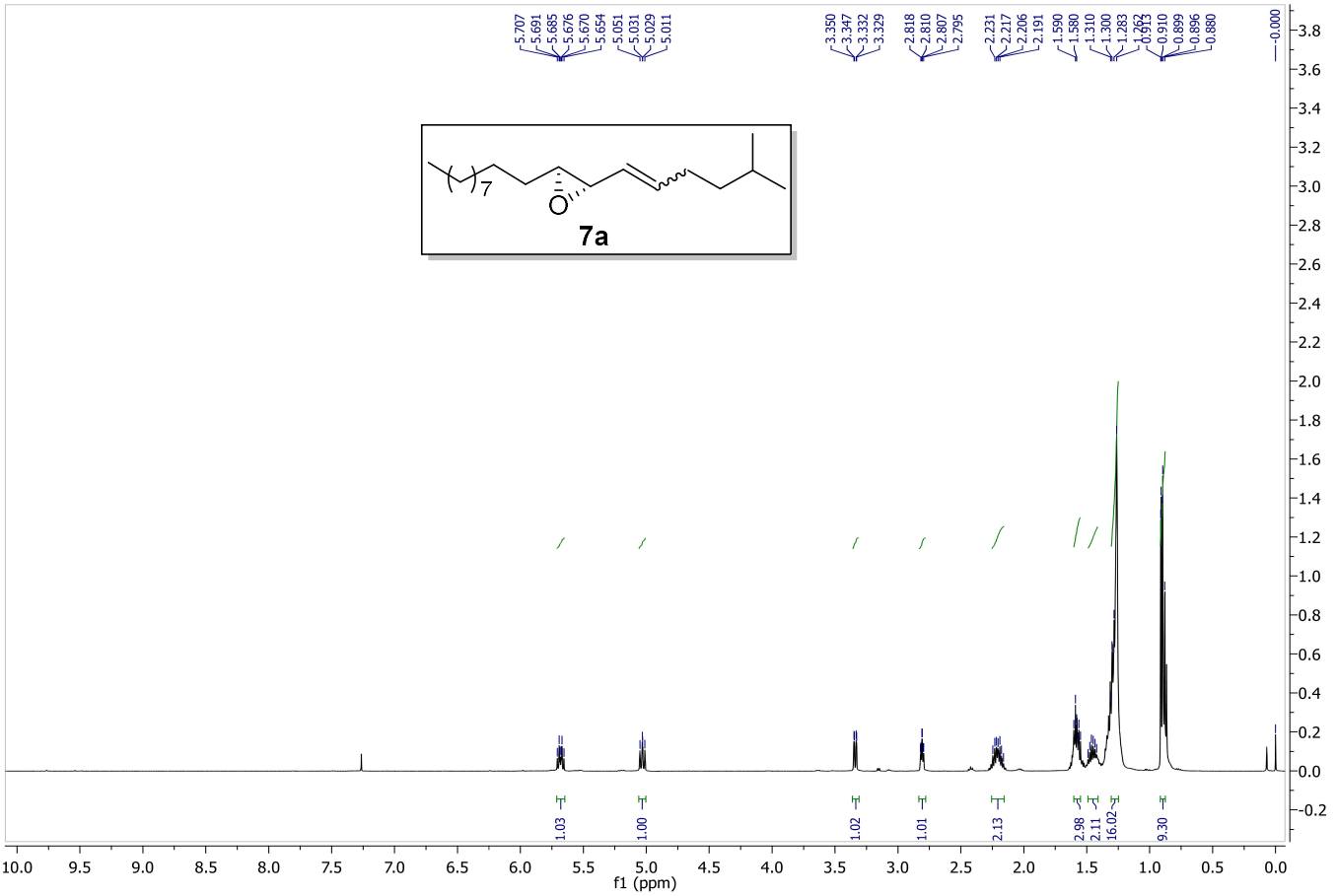
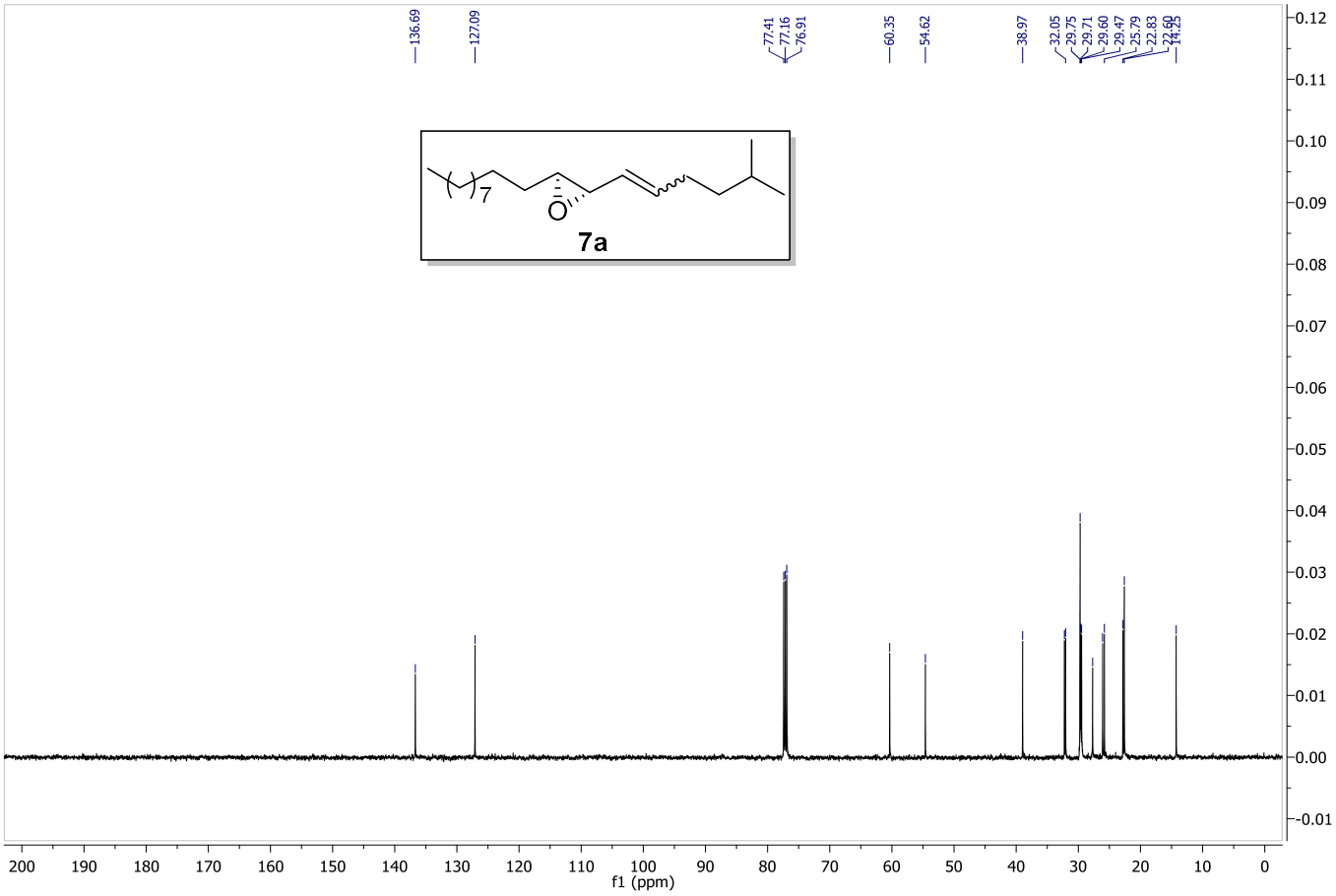
Report Method: Dis Rec
Report Method ID: 1720 1720
Page: 1 of 1

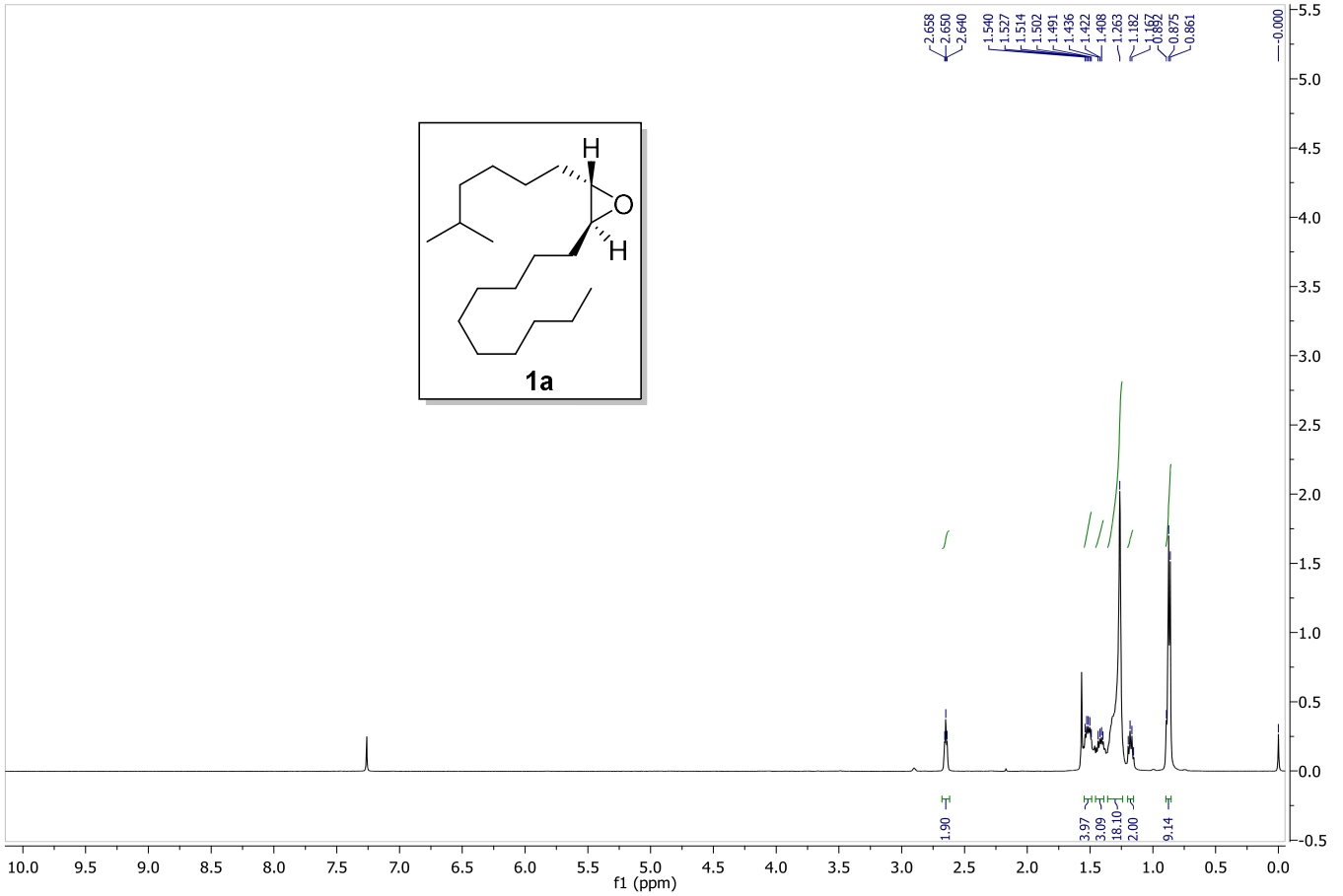
Project Name: SKP
Date Printed:
7/4/2022
4:17:22 PM Asia/Calcutta

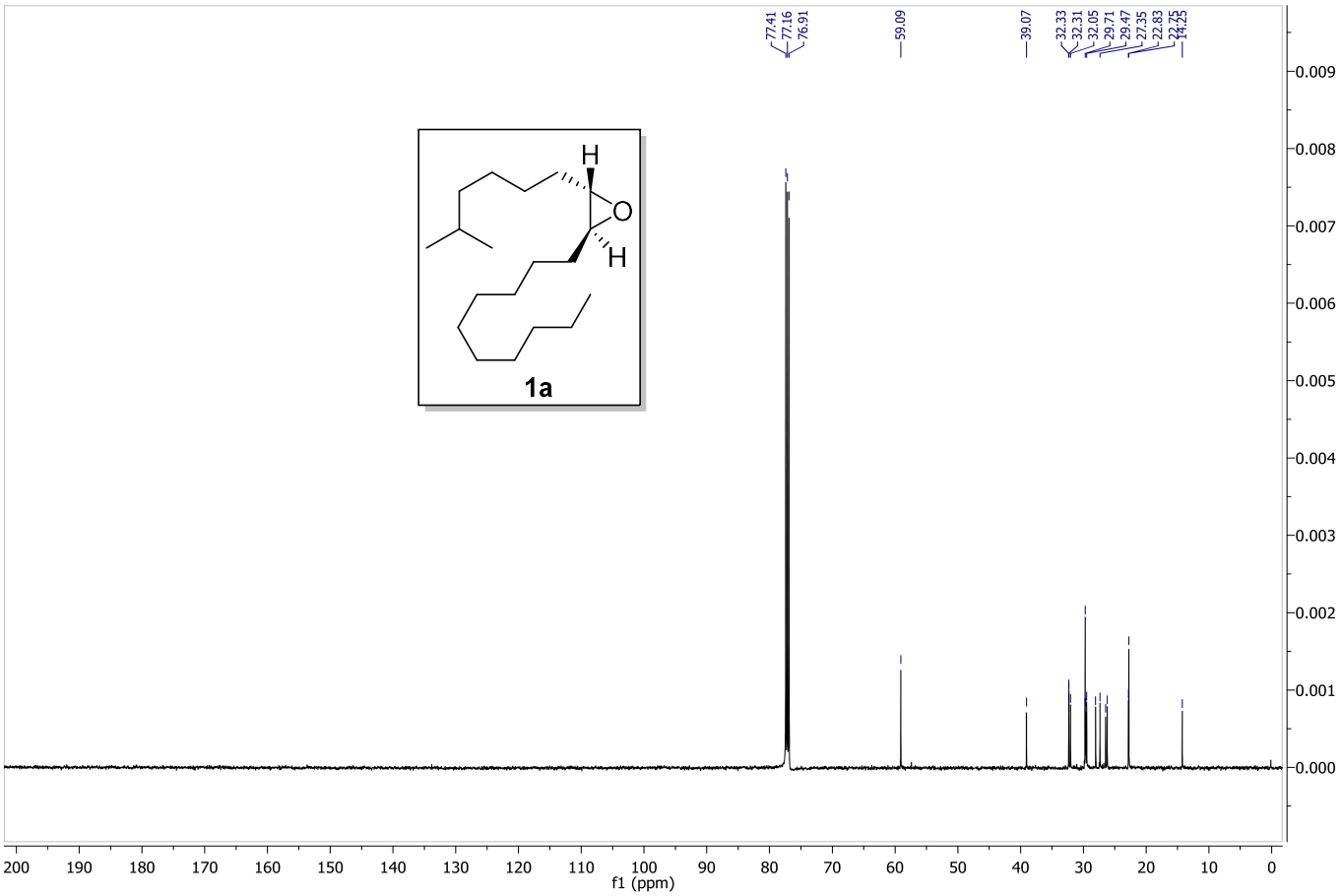
^{1}H NMR (500 MHz, $CDCl_3$) and $^{13}C\{^{1}H\}$ NMR (126 MHz, $CDCl_3$), **7a**



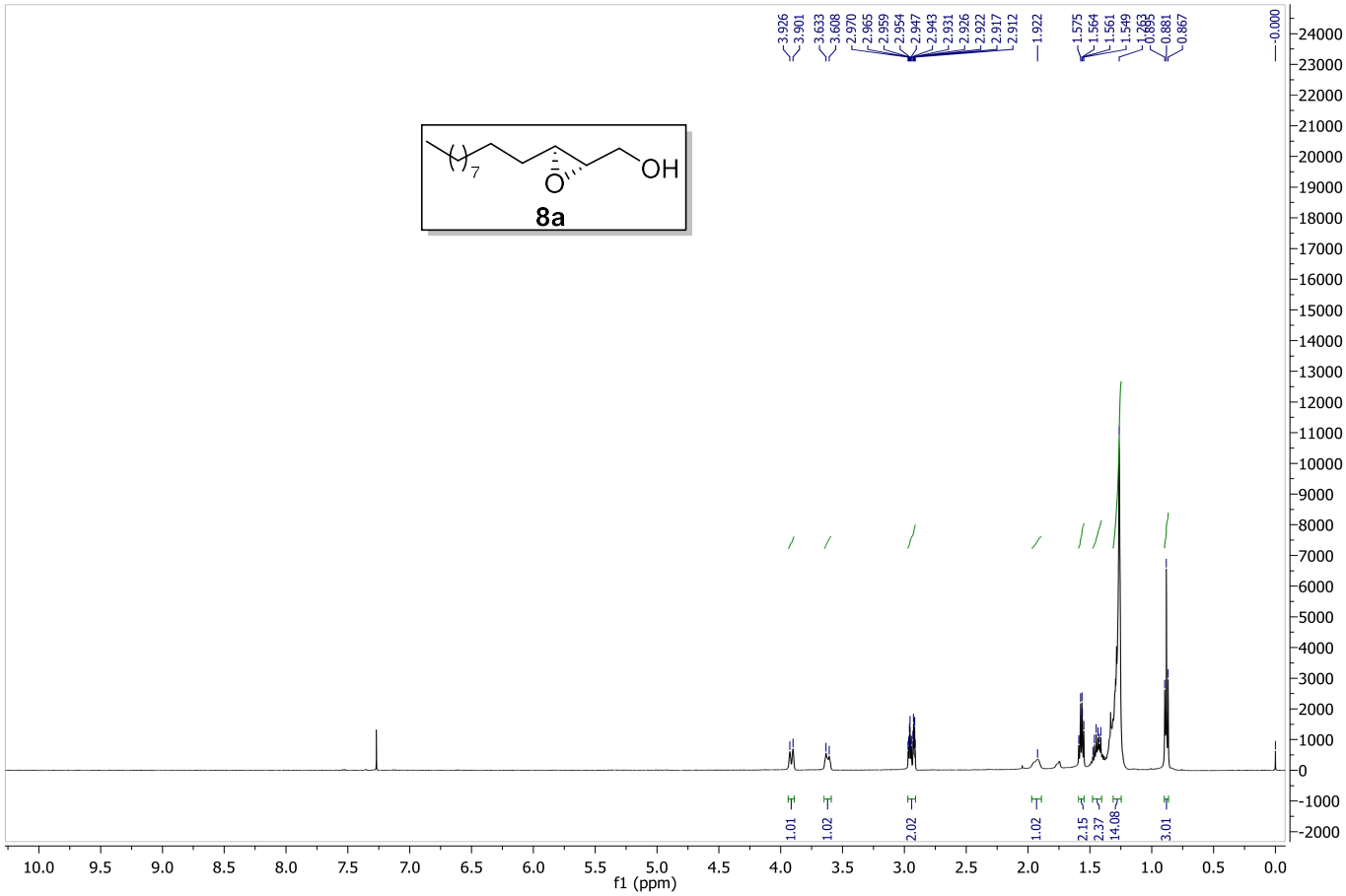


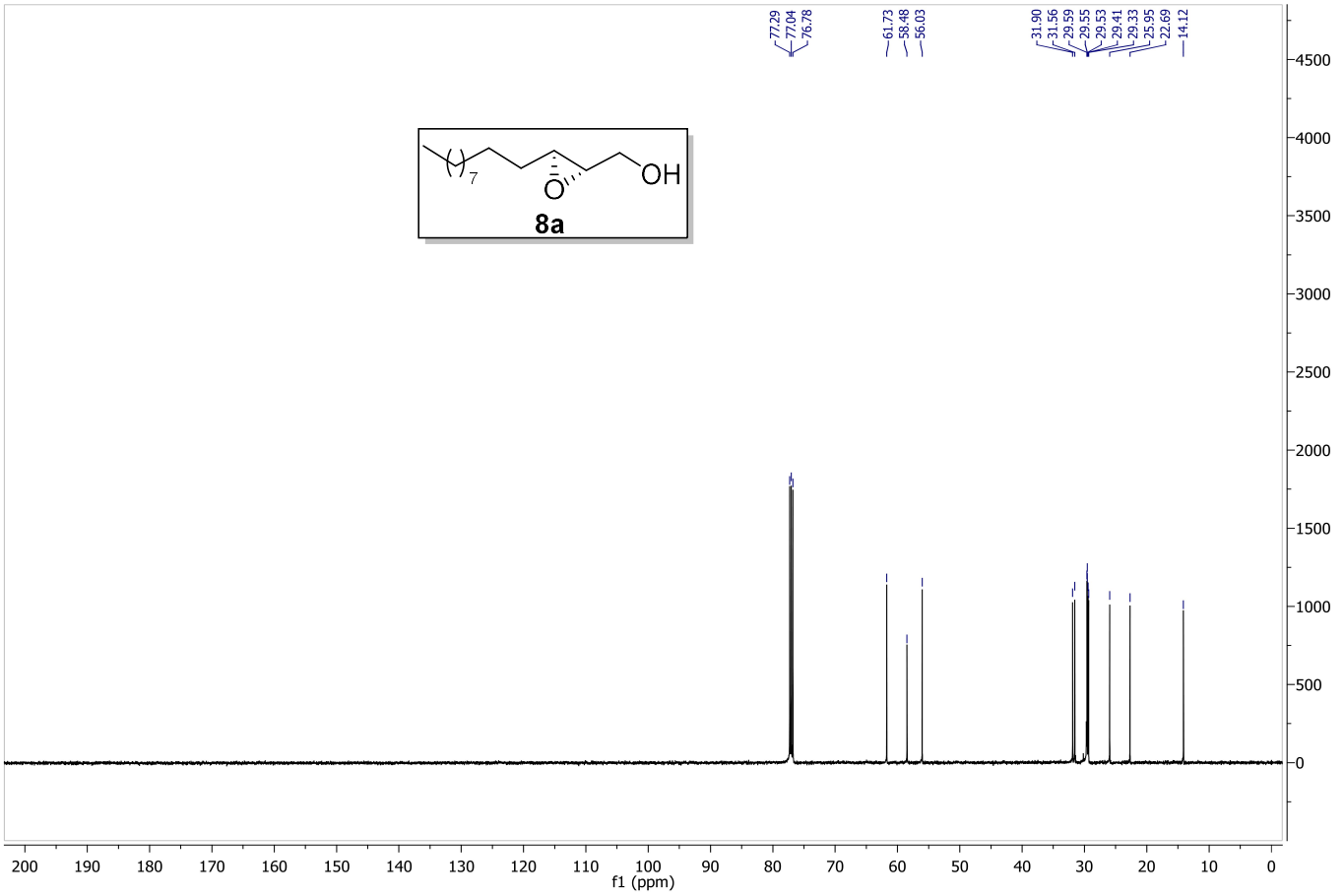
^{1}H NMR (500 MHz, $CDCl_3$) and $^{13}C\{^{1}H\}$ NMR (126 MHz, $CDCl_3$), **1a**



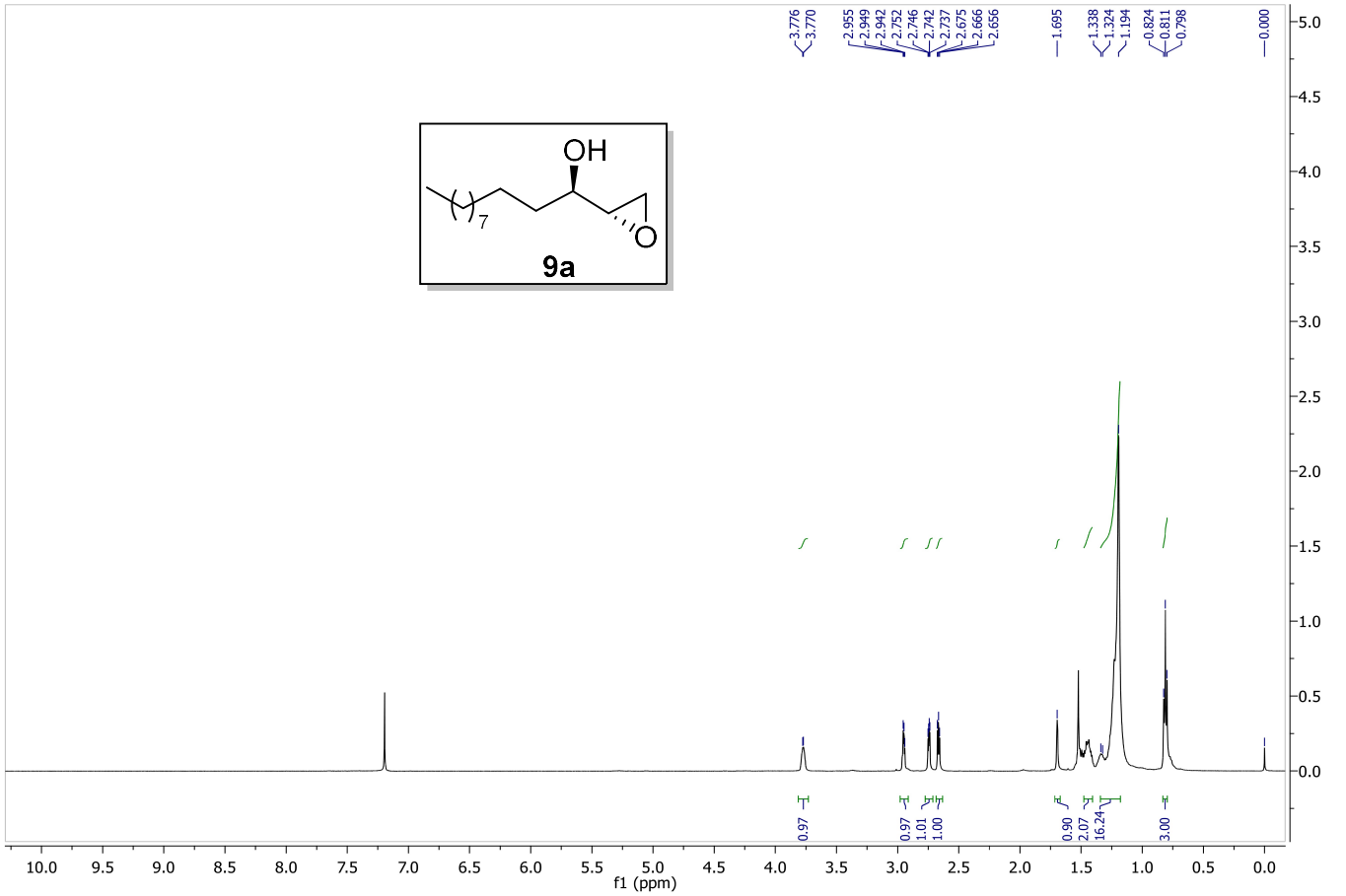


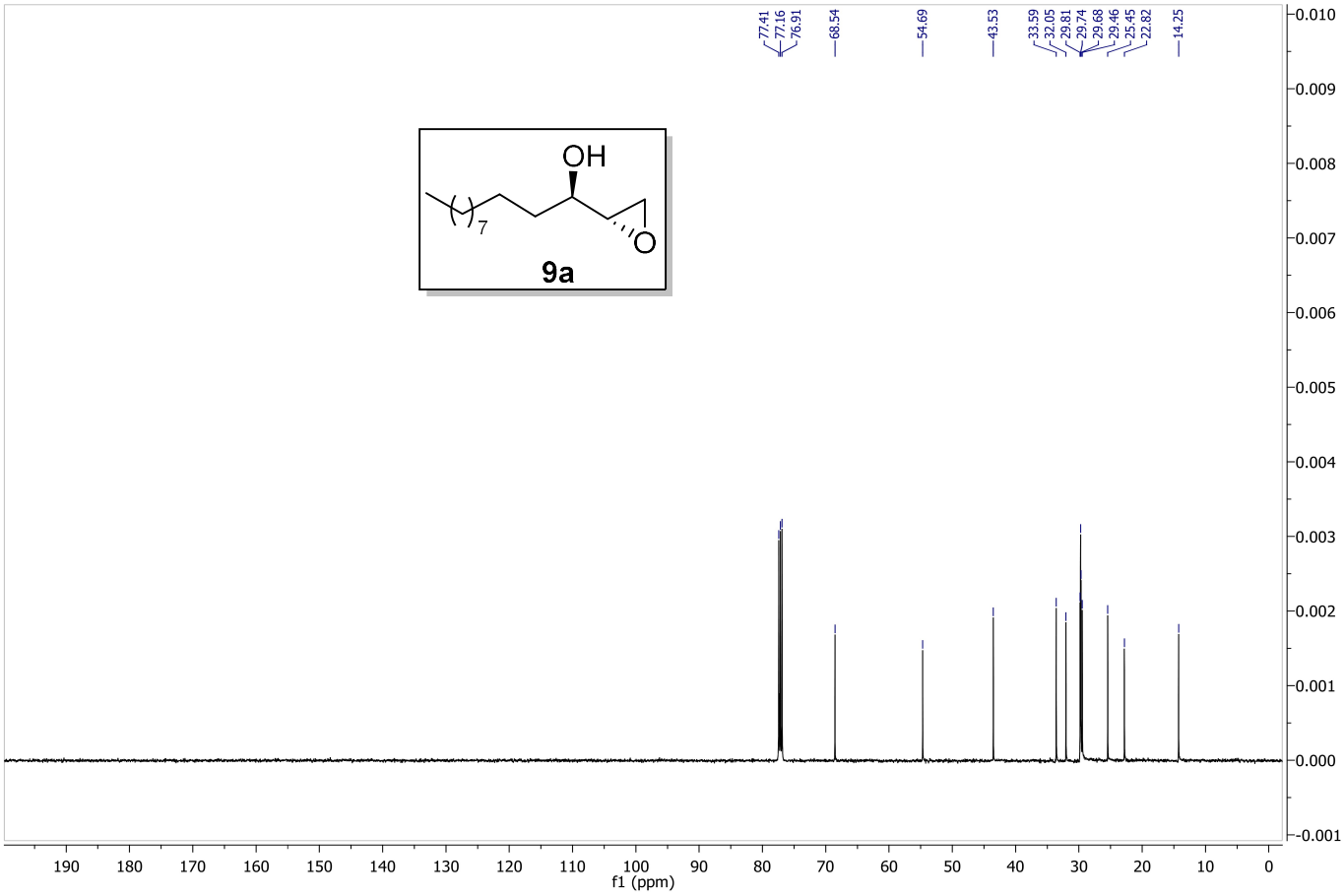
^{1}H NMR (500 MHz, $CDCl_3$) and ^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$), **8a**



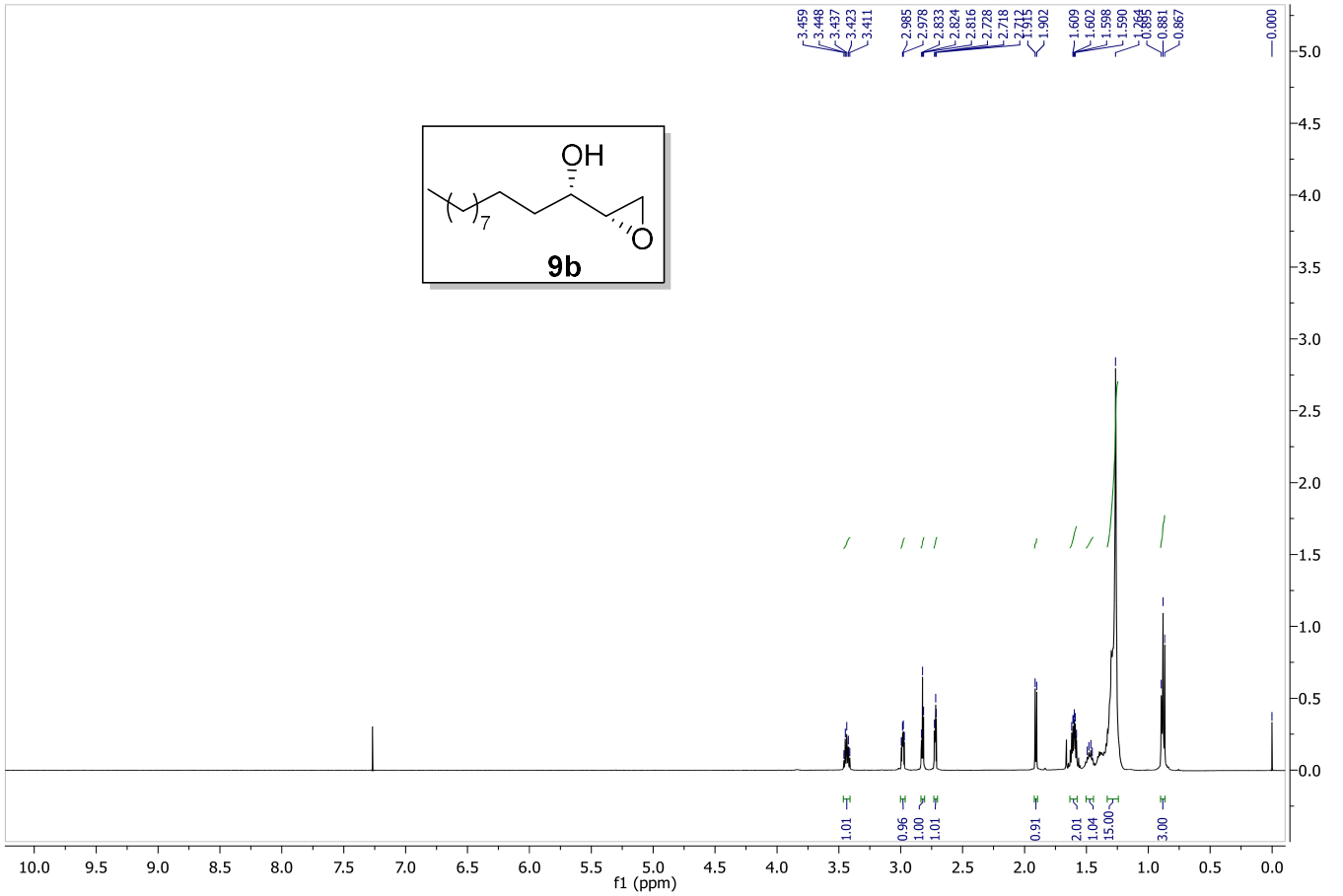


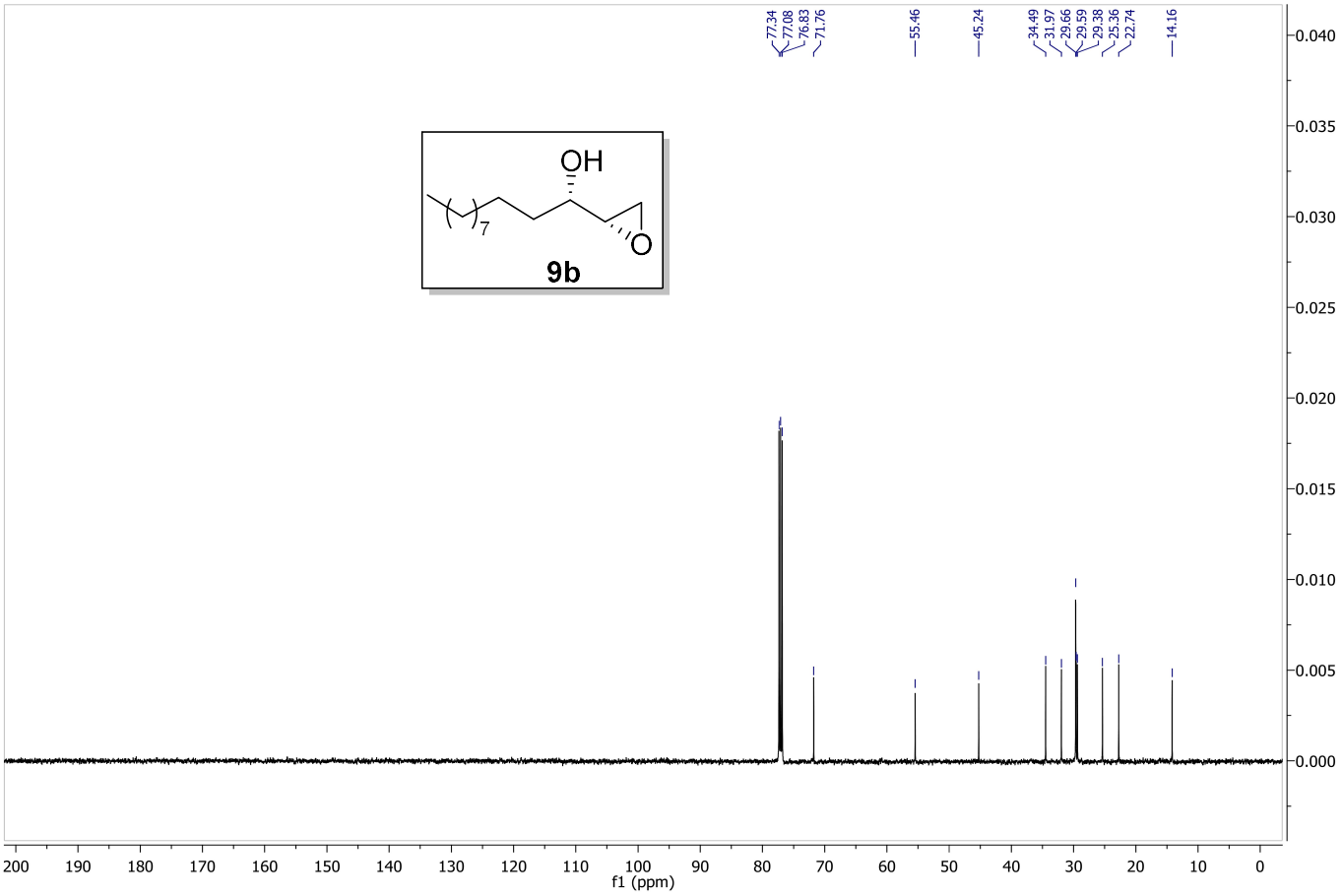
^{1}H NMR (500 MHz, $CDCl_3$) and $^{13}C\{^{1}H\}$ NMR (126 MHz, $CDCl_3$), **9a**



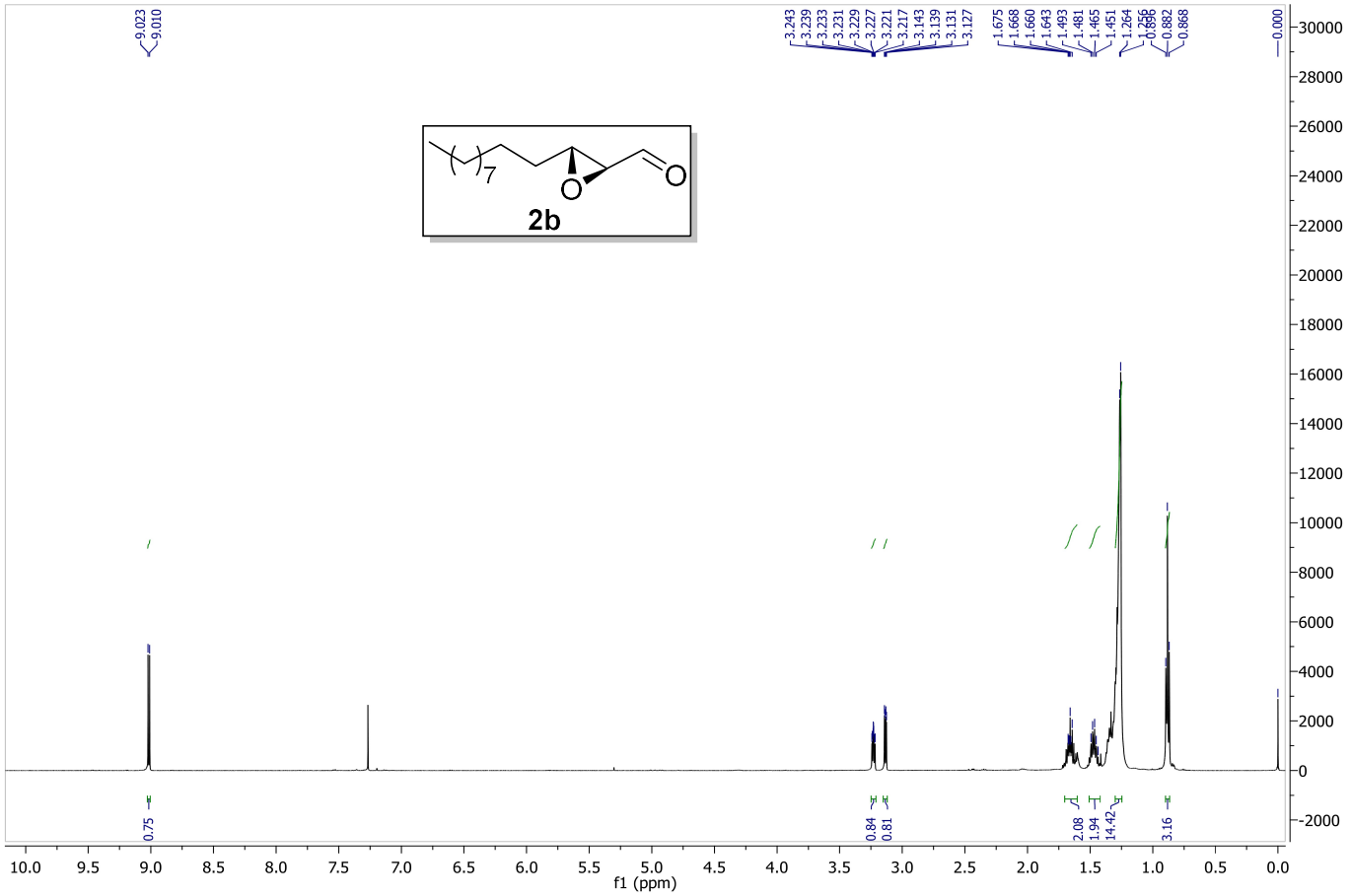


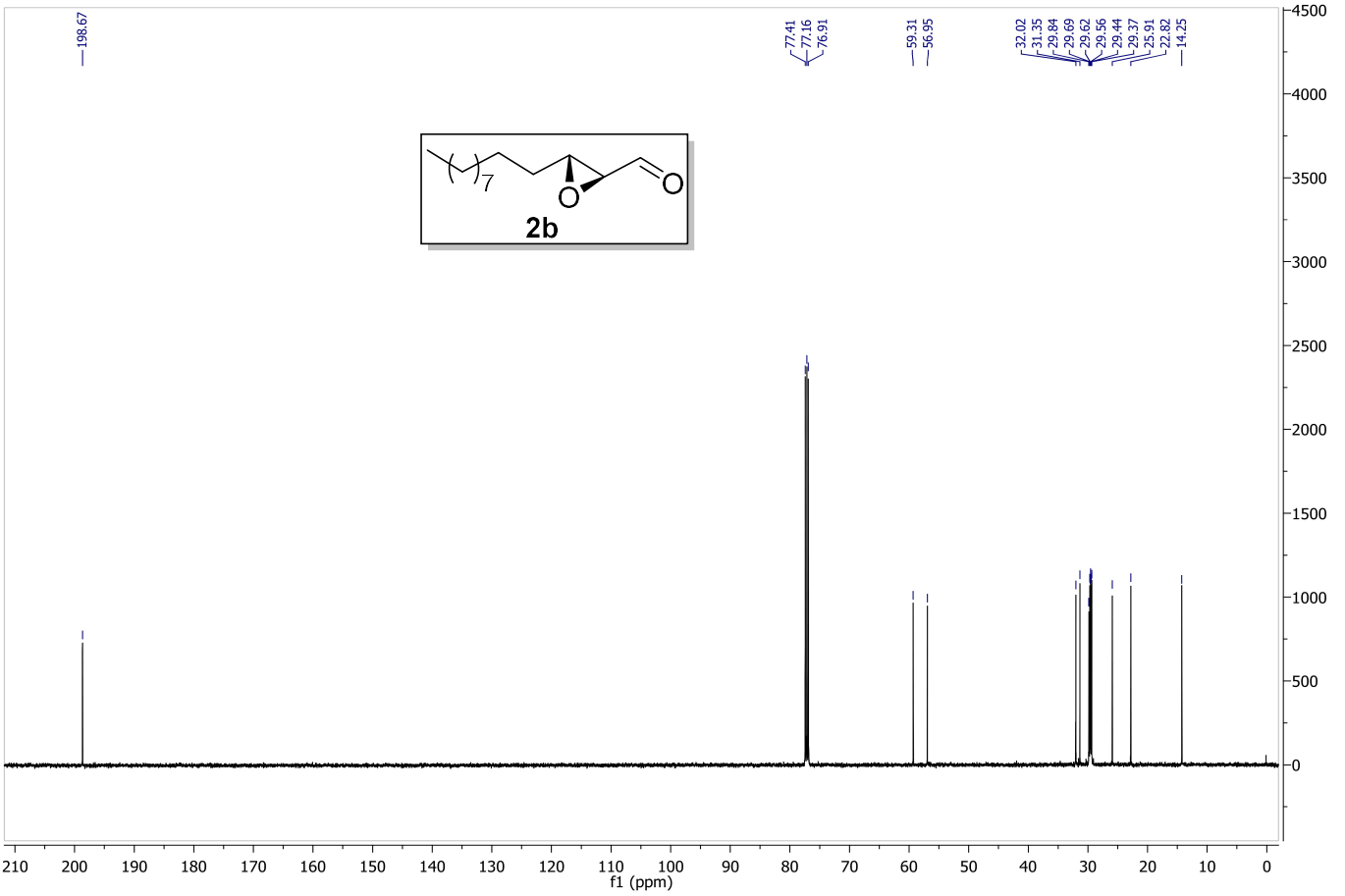
^{1}H NMR (500 MHz, $CDCl_3$) and $^{13}C\{^{1}H\}$ NMR (126 MHz, $CDCl_3$), **9b**





^{1}H NMR (500 MHz, $CDCl_3$) and ^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$), **2b**





Empower™ 3 SOFTWARE

Dls Rec

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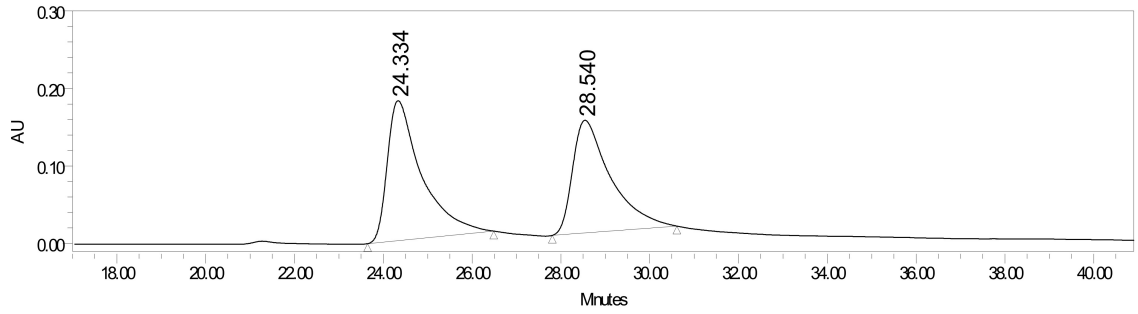

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Reported by User: System
Report Method: Dls Rec
Report Method ID: 1728 1728
Page: 1 of 1

Project Name: SKF
Date Printed:
7/4/2022
4:29:28 PM Asia/Calcutta

Empower™ 3 SOFTWARE

Dis Rec

SAMPLE INFORMATION

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Injection Volume:	20.00 ul	Channel Name:	2998 Ch1 254nm@1.2nm
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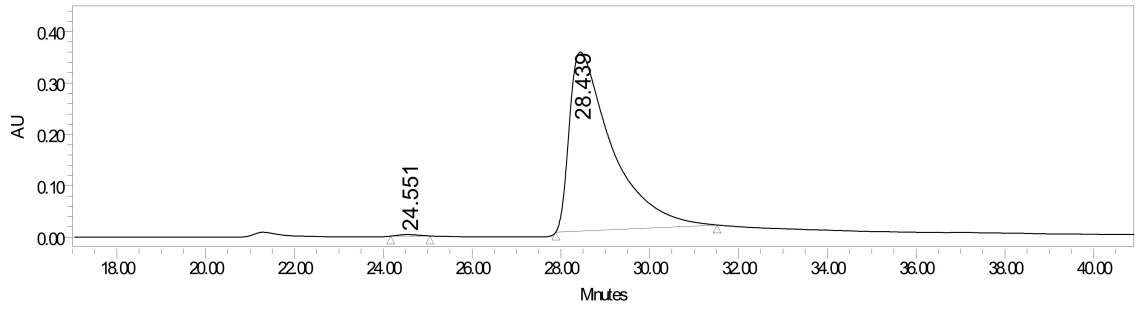

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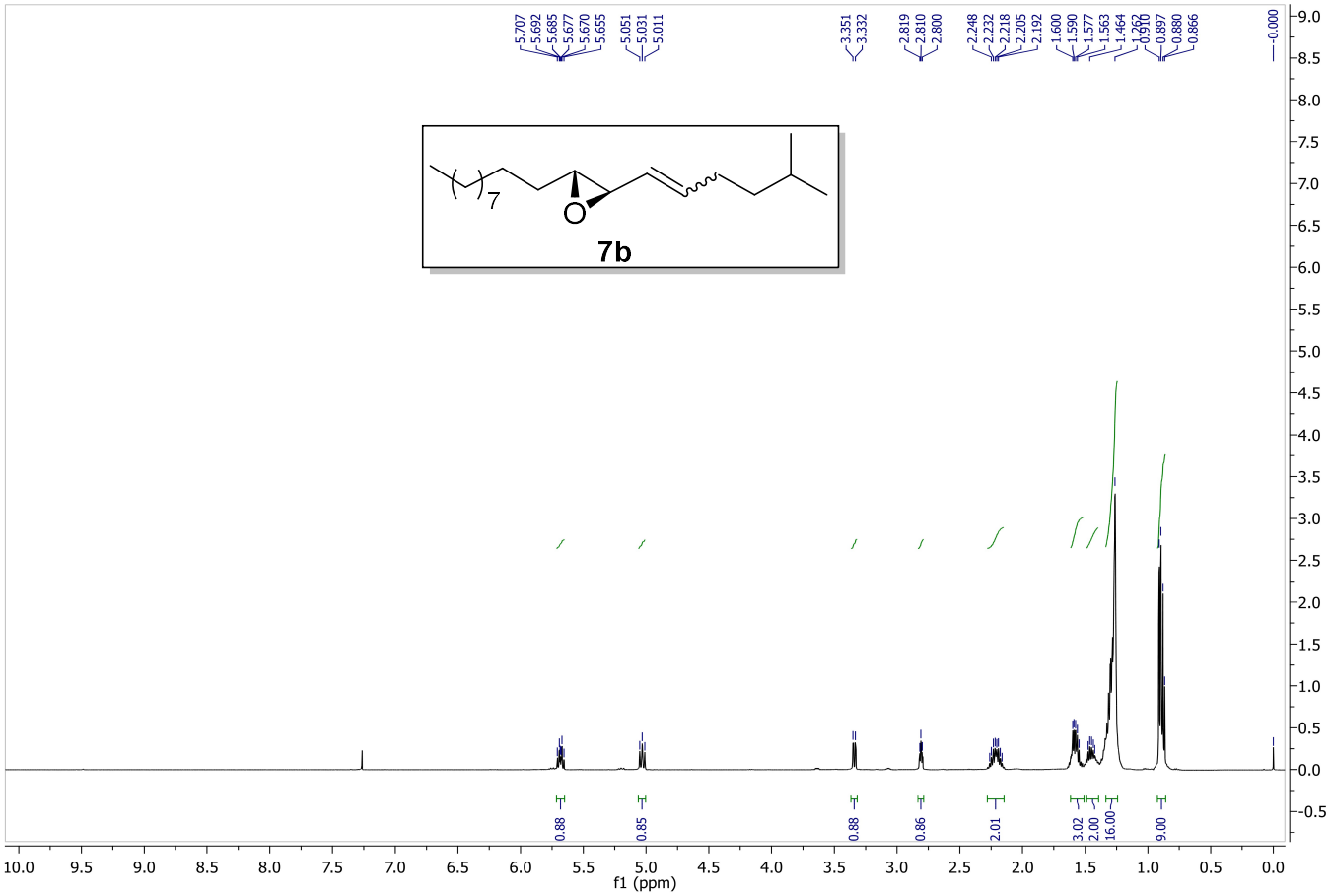
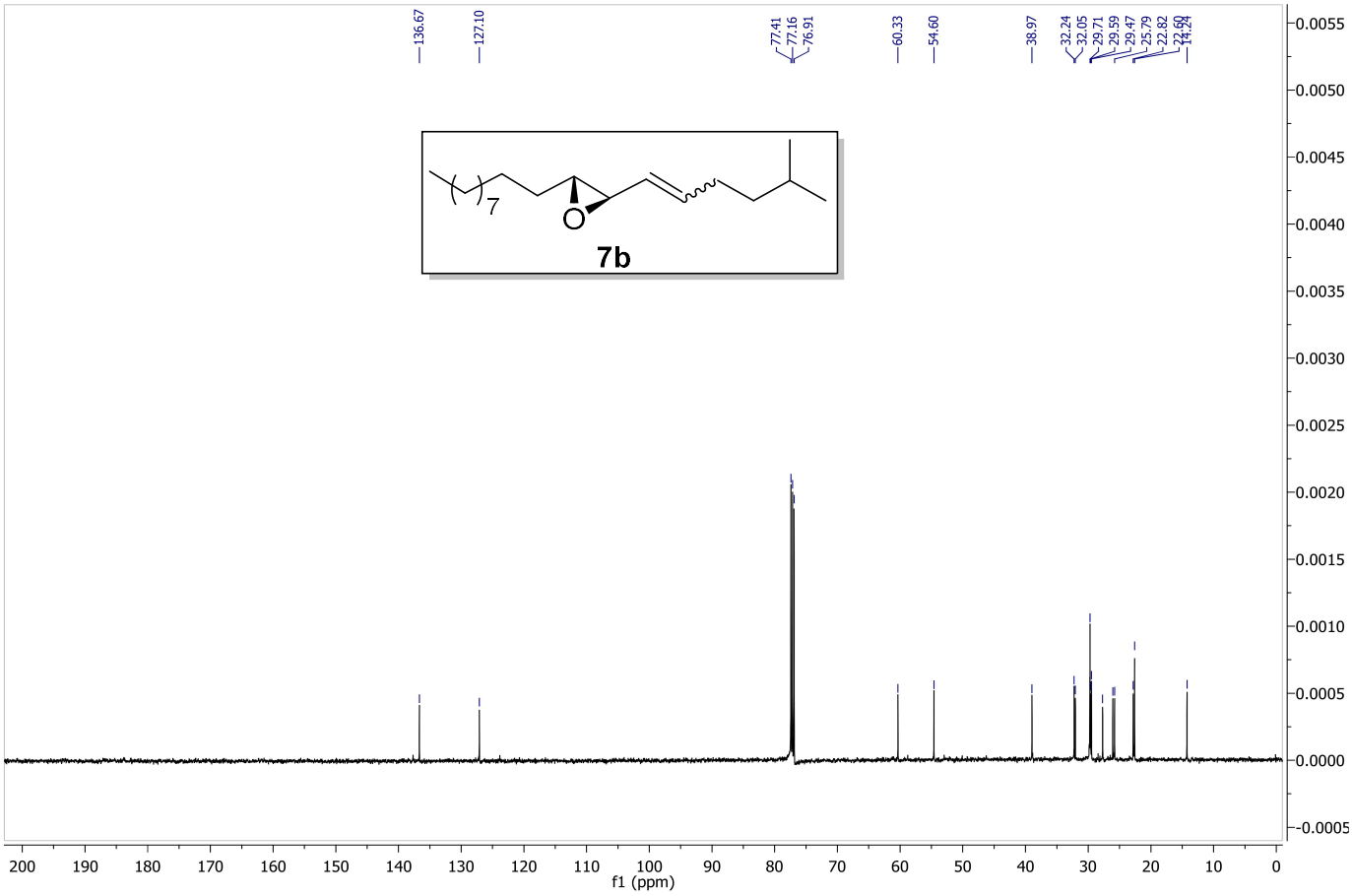
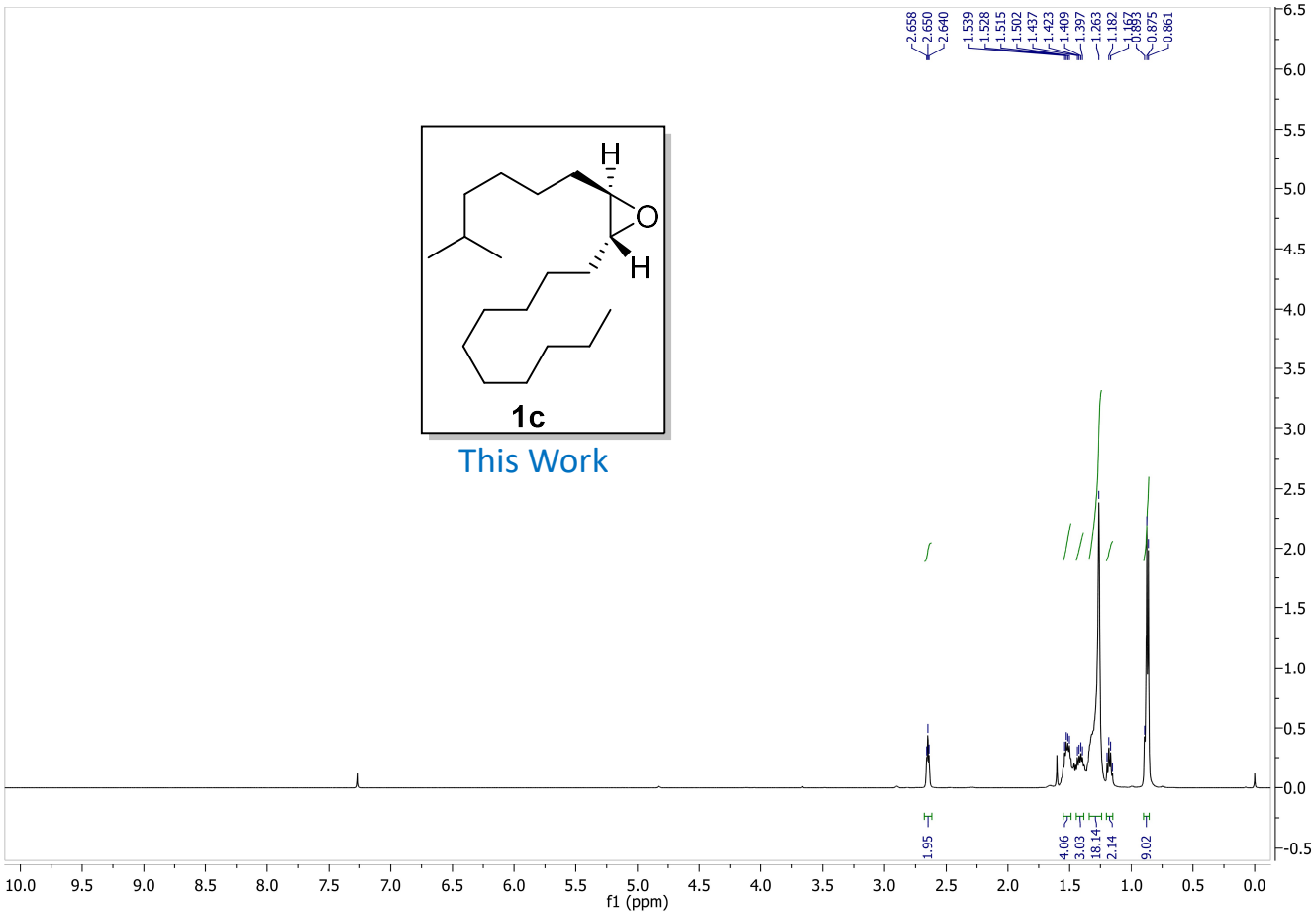
^{1}H NMR (500 MHz, $CDCl_3$) and ^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$), **7b**





^{1}H NMR (500 MHz, $CDCl_3$) of **1c** and known spectra

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PROCNO                   1
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TE                 292.9 K
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TD0                      1
======== CHANNEL f1 ========
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P1                 13.50 usec
PL1                -1.00 dB
PL1W          10.56200695 W
SFO1          400.1324710 MHz
SI                   32768
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SSB                     0
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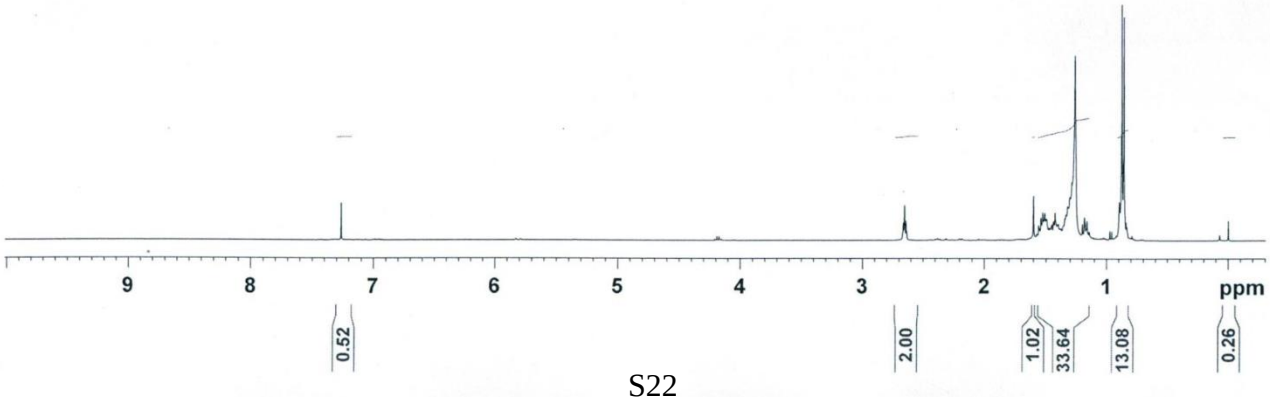
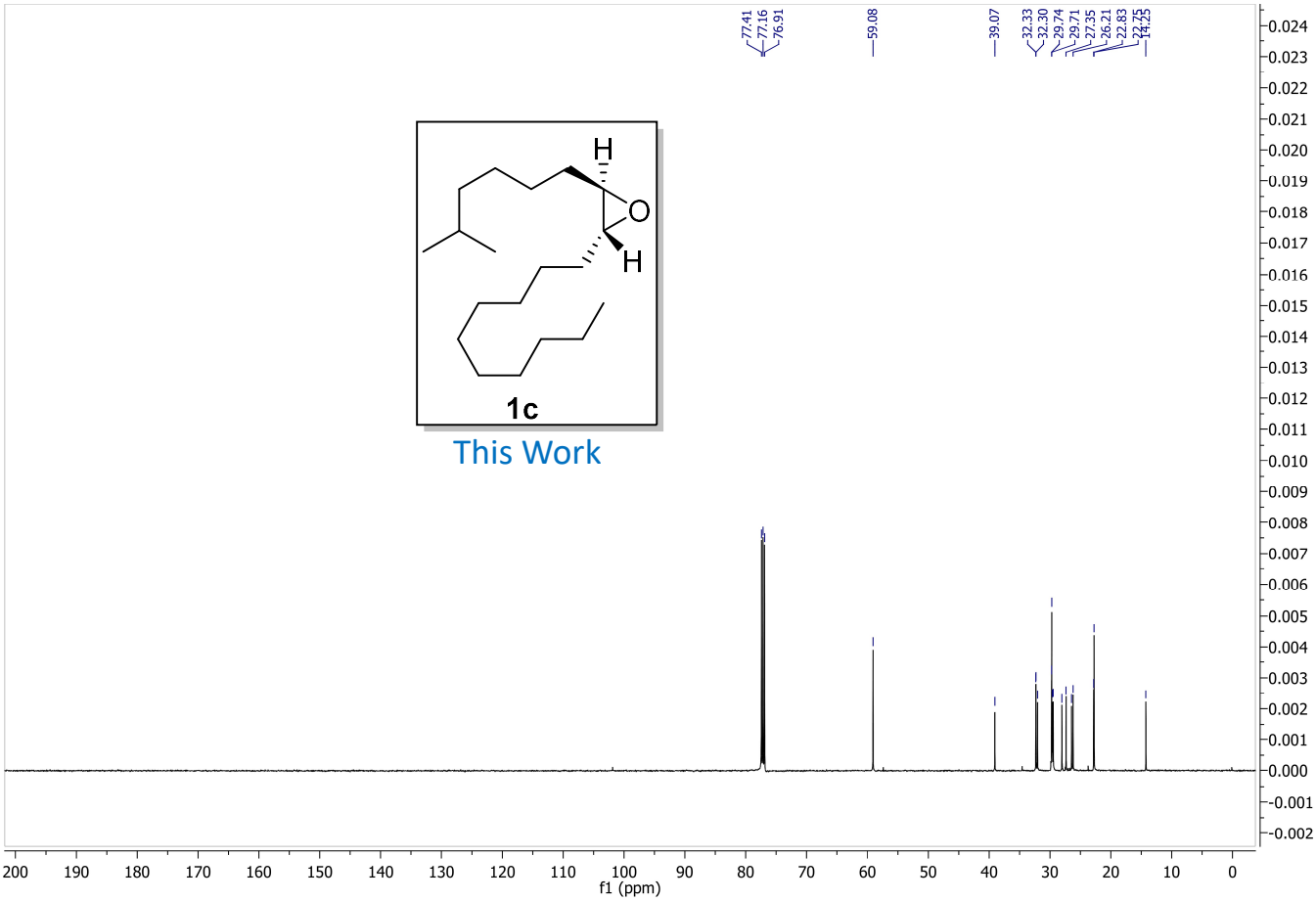
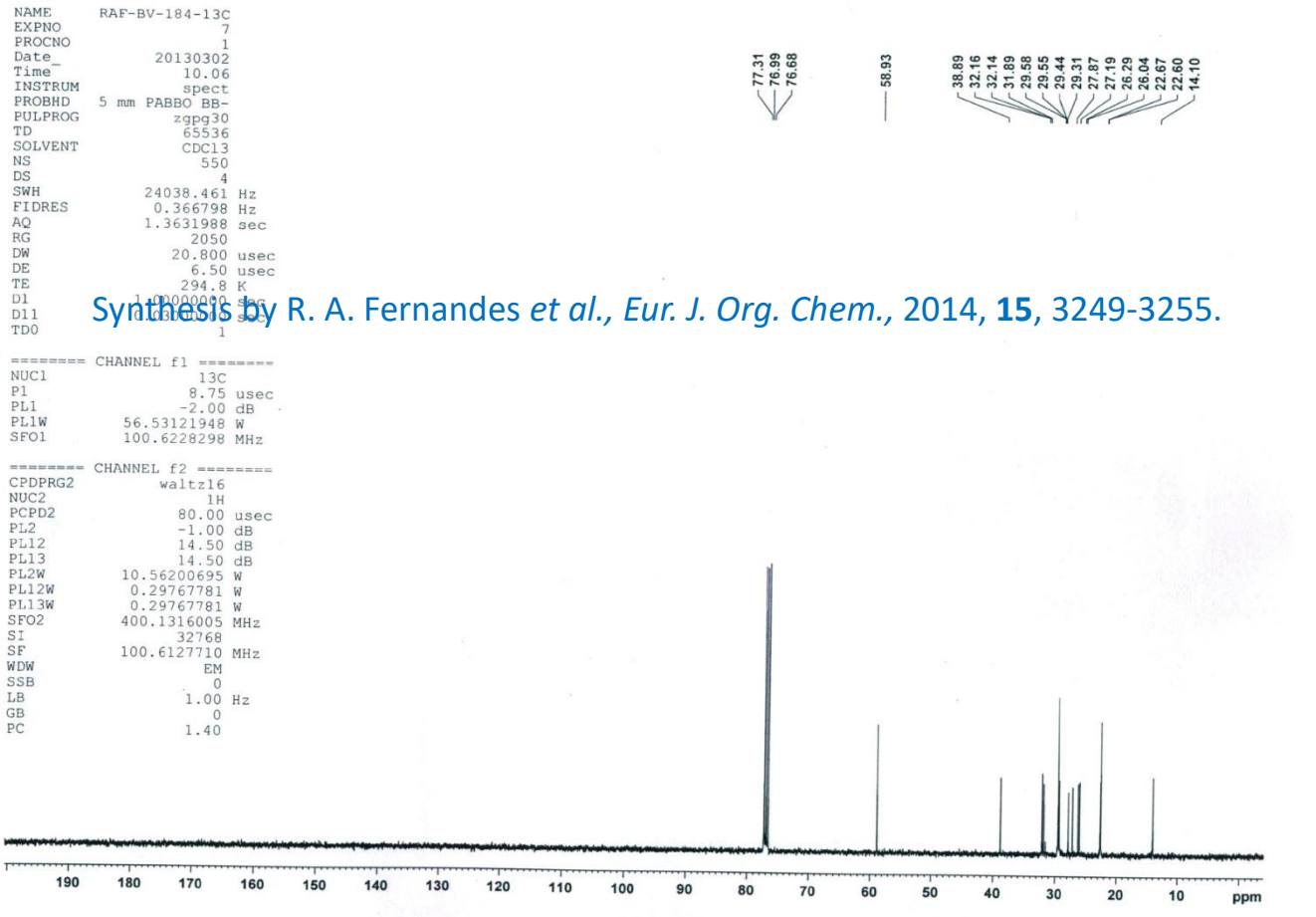
Synthesis by R. A. Fernandes *et al., Eur. J. Org. Chem.,* 2014, **15**, 3249-3255.



$^{13}C\{^{1}H\}$ NMR (126 MHz, $CDCl_3$) of **1c** and known spectra



This Work



Synthesis by R. A. Fernandes *et al., Eur. J. Org. Chem.,* 2014, **15**, 3249-3255.

Tabular comparison of NMR spectra for **1a** with known spectra.

^{1}H NMR spectra for **1a** and known spectra	
This Work	S. G. Kim, *Synthesis*, 2009, **14**, 2418.
^{1}H NMR (500 MHz, $CDCl_3$) δ: 2.66–2.64 (m, 2H) 1.55–1.49 (m, 4H) 1.43–1.39 (m, 3H) 1.26 (brs, 18H) 1.20–1.15 (m, 2H) 0.89–0.86 (m, 9H)	^{1}H NMR (300 MHz, CDCl3) δ: 2.62–2.67 (m, 2 H) 1.17–1.58 (m, 27 H) 0.85–0.91 (m, 9 H)
^{13}C{^{1}H} NMR spectra for **1a** and known spectra	
This Work	S. G. Kim, *Synthesis*, 2009, **14**, 2418.
^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ: 59.0, 39.0, 32.3, 32.0, 32.3, 29.8, 29.7, 29.6, 29.4, 28.0, 27.7, 26.4, 26.2, 22.8, 22.7, 14.2.	^{13}C NMR (75 MHz, CDCl3) δ: 59.1, 39.1, 32.39, 32.36, 32.1, 29.81, 29.77, 29.67, 29.5, 28.1, 27.4, 26.5, 26.3, 22.9, 22.7, 14.3.

Tabular comparison of NMR spectra for **1c** with known spectra.

^{1}H NMR spectra for **1c** and known spectra	
This Work	R. A. Fernandes, *et al.*, *Eur. J. Org. Chem.*, 2014, **15**, 3249.
^{1}H NMR (500 MHz, $CDCl_3$) δ: 2.66–2.64 (m, 2H) 1.53–1.50 (m, 4H) 1.43–1.39 (m, 3H) 1.26 (brs, 18H) 1.19–1.15 (m, 2H) 0.89–0.86 (m, 9H)	^{1}H NMR (400 MHz, $CDCl_3$/TMS): *δ* 2.67–2.64 (m, 2 H) 1.57–1.14 (m, 27 H) 0.97–0.82 (m, 9 H).
^{13}C{^{1}H} NMR spectra for **1c** and known spectra	
This Work	R. A. Fernandes, *et al.*, *Eur. J. Org. Chem.*, 2014, **15**, 3249.
^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ: 59.0, 39.0, 32.3, 32.3, 32.0, 29.74, 29.71, 29.6, 29,4, 28.0, 27.3, 26.4, 26.2, 22.8, 22.7, 14.2.	^{13}C NMR (100 MHz, $CDCl_3$) δ: 58.9, 38.9, 32.2, 32.1, 31.9, 29.6, 29.5, 29.4, 29.3, 27.9, 27.2, 26.3, 26.0, 22.7, 22.6, 14.1.