

Stereoselective synthesis of a *cis*-cedrane-8,9-diol as a key intermediate for an amber odorant

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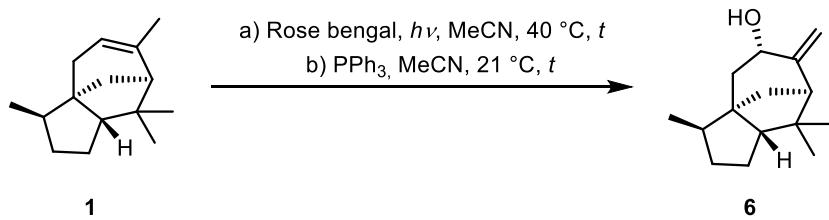
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1. Photooxidation of (-)- α -cedrene (**1**)

Table S1 shows the results of the photooxidation of cedrene to the allyl alcohol **6**.

Procedure for photooxidation of (-)- α -Cedrene (1**):** (-)- α -Cedrene (**1**) (650 mg, 3.18 mmol) and rose bengal (62 mg, 0.064 mmol) were dissolved in CH₃CN (50 mL). The reaction mixture was irradiated with a 900 W mercury lamp in a double-walled glass reactor while O₂ was bubbled through the solution continuously. The cooling medium was set at 40 °C. After 4.5 h the mixture was transferred to a round-bottom flask and Ph₃P (920 mg, 3.51 mmol) was added. After stirring at ambient temperature for 16 h all volatiles were evaporated and the residue was purified via column chromatography (SiO₂, *c*Hex/EtOAc 20:1). The product **6** (302 mg, 1.37 mmol, 43%) was obtained a colourless solid. ¹H NMR (300 MHz, CDCl₃) δ = 0.87 (d, *J* = 7.0 Hz, 3H), 0.94 (s, 3H), 0.97 (s, 3H), 1.14–1.49 (m, 4H), 1.49–1.66 (m, 2H), 1.69–1.95 (m, 3H), 2.08–2.20 (m, 1H), 2.35 (d, *J* = 4.2 Hz, 1H), 4.34 (ddt, *J* = 10.1, 7.6, 2.5 Hz, 1H), 4.75 (ddt, *J* = 2.5, 1.9, 0.6 Hz, 1H), 4.99 (dd, *J* = 2.3, 1.9 Hz, 1H) ppm.

Table S1. Photooxidation of cedrene (**1**) to the corresponding allyl alcohol **6**.



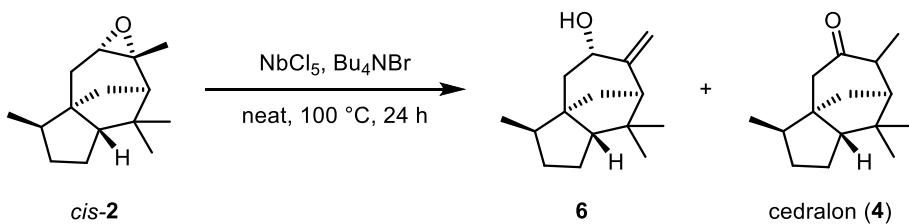
entry	<i>t</i> / h (photooxidation)	<i>t</i> / h (reduction)	yield 6 / %
1	2	3	18
2	4.5	16	43

2. Isomerization of *cis*-cedrene epoxide (*cis*-2)

Table S2 shows studies on the isomerization of *cis*-2 based on a report by Da Silva and co-workers on the niobium pentachloride catalyzed isomerization of monoterpene epoxides.³

NbCl₅ catalyzed isomerization of cedrene epoxide (2). Under Ar a reaction vessel suitable for high pressure reactions was charged with *cis*-cedrene epoxide (*cis*-2, 600 mg, 2.72 mmol), NbCl₅ (4.0 mg, 0.015 mmol) and Bu₄NBr (44 mg, 0.14 mmol). The vessel was sealed, and the mixture was stirred at 100 °C for 24 h without any solvent. A sample for NMR analysis was taken from the crude reaction mixture. The reaction mixture was purified via column chromatography (cHex/EtOAc 20:1). and the product **6** (249 mg, 1.13 mmol, 42%) was obtained as colorless solid. ¹H NMR (300 MHz, CDCl₃) δ = 0.87 (d, *J* = 7.0 Hz, 3H), 0.94 (s, 3H), 0.97 (s, 3H), 1.11–1.52 (m, 4H), 1.48–1.64 (m, 2H), 1.71–1.93 (m, 3H), 2.09–2.20 (m, 1H), 2.35 (d, *J* = 3.9 Hz, 1H), 4.34 (ddt, *J* = 10.1, 7.6, 2.4 Hz, 1H), 4.75 (ddt, *J* = 2.5, 1.9, 0.6 Hz, 1H), 4.99 (dd, *J* = 2.4, 1.9 Hz, 1H) ppm.

Table S2. Isomerization of *cis*-cedrene epoxide (*cis*-2) using NbCl₅.



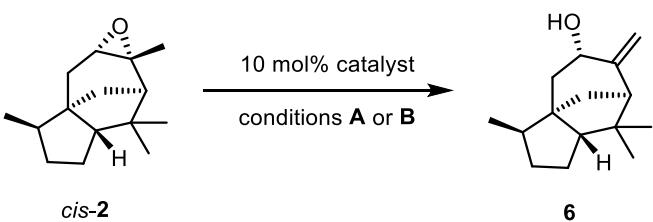
entry	NbCl ₅ / mol%	Bu ₄ NBr / mol%	ratio 6 : 4 ^[a]
1 ^[b]	1	2	1 : 1.2
2 ^[b]	0.5	5	1 : 0.7 (42%) ^[c]
3 ^[d]	0.5	5	1 : 8.9
4	10	–	0 : 1
5	–	5	no conversion

^[a] Product ratio determined via ¹H NMR. ^[b] Chlorinated by-product was observed. ^[c] Isolated yield is given in parentheses. ^[d] 9 mmol of *cis*-cedrene epoxide (*cis*-2) were used as starting material.

Table S3 shows studies on the isomerization of *cis*-2 based on the report by Kolomeyer *et al.* which described the isomerization of limonene oxide to the allylic alcohol carveol in presence of a metal compound and an aminophenol as catalyst system.⁴

Organocatalyzed isomerization of *cis*-cedrene oxide (*cis*-2). In a vial suitable for pressure reactions *cis*-cedrene epoxide (*cis*-2, 220 mg, 1.00 mmol) was dissolved in toluene (5 mL). The organocatalyst (10 mol%, 0.1 mmol) was added, the tube was sealed and stirred at 23 °C for 20 h (conditions A) or at 80 °C for 8.5 h (conditions B). The yield was determined direct from the crude reaction mixture by ¹H NMR spectroscopy.

Table S3. Organocatalyzed isomerization of cedrene oxide *cis*-2.

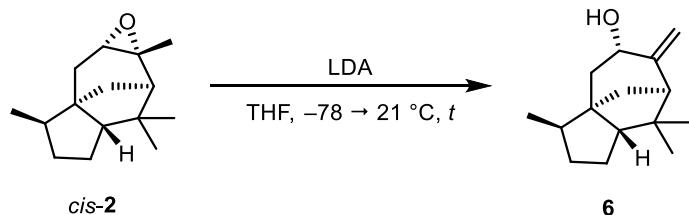


entry	catalyst	conditions A ^[a] yield / % ^[c]	conditions B ^[b] yield / % ^[c]
1		0 (>99)	0 (>99)
2		7 (93)	12 (88)
3 ^[d]	2,2'-bipyridine	— ^[e]	— ^[e]
4		15 (85)	26 (74)
6		33 (67)	traces (>99)
5		26 (74)	traces (>99)

^[a] Conditions A: 1 mmol cedrene oxide *cis*-2, 10 mol% catalyst, toluene (5 mL), 21 °C, 20 h. ^[b] Conditions B: 1 mmol cedrene oxide **2**, 10 mol% catalyst, toluene (5 mL), 80 °C, 8.5 h. ^[c] Determined by ¹H NMR from the reaction mixture. Conversion in % is given in parentheses. ^[d] Addition of 0.5 mmol TfOH. ^[e] Only cedralon (**4**) was obtained.

Table S4 shows studies on the LDA mediated isomerisation of *cis*-2 to 6.

Table S4. Optimization of the isomerization of *cis*-cedrene epoxide (*cis*-2) using LDA.



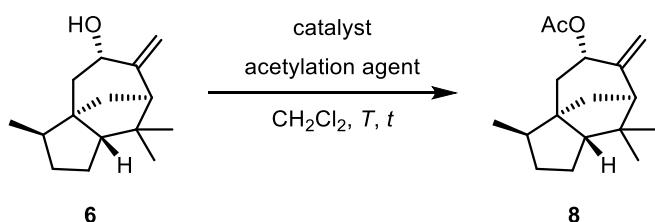
entry	equiv LDA	t / h	<i>cis</i> -2 / mmol	yield 6 / % ^[a]
1	2	24	0.45	72 ^[b]
2	3	28	4.54	39
3	4	26	4.54	64
5	2	48	22.7	89

^[a] Isolated yield. ^[b] Yield was determined via ¹H NMR from the reaction mixture.

3. Optimization of the esterification of β -cedren-9-ol (6)

Table S5 shows the optimization of the esterification of allyl alcohol 6.

Table S5. Esterification of β -cedren-9-ol (6) using different conditions.



entry	acetylation agent	catalyst	T / °C	t / h	yield 8 / %
1	AcCl	NEt ₃	0 to 23	15	n.d. ^[a]
2	Ac ₂ O	DMAP / NEt ₃	23	15	82
3	Ac ₂ O	DMAP	23	18.5	95

^[a] Ratio starting material/product = 1 : 0.7, determined via ¹H NMR integration.

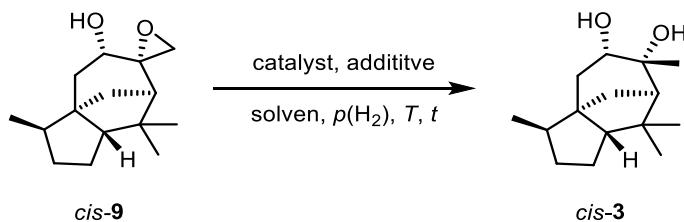
4. Reduction of β -epoxy cedren-9-ol (*cis*-9) using H₂

Table S6 shows the attempts to reduce *cis*-9 with H₂.

Procedure for hydrogenation reactions at 20 bar H₂. Under Ar a glass vial was charged with β -epoxy cedren-9-ol (*cis*-9, 60 mg, 0.25 mmol) and catalyst (1 mol%). Dry solvent (2 mL) was added, and the vial was placed in an autoclave. The reactor was sealed and charged with 20 bar H₂ at 100 °C. After stirring for 24 h, the reactor was cooled to ambient temperature, the pressure was released carefully, and the crude reaction mixture was filtered through SiO₂ using EtOAc as eluent. The yield was determined using ¹H NMR spectroscopy and mesitylene as the internal standard.

Procedure for hydrogenation reactions at 4 bar H₂. Under Ar a glass vial was charged with β -epoxy cedren-9-ol (*cis*-9, 20 mg, 85 mmol or 10 mg, 43 mmol), catalyst (1 mol%) and additive (0.2 equiv, 17 mmol or 8.6 mmol). Dry solvent (2 mL) was added and it was placed in an autoclave. The reactor was sealed and charged with 4 bar H₂ at 23 °C and it was stirred for 4–6 h. Subsequently, pressure was released carefully, and the crude reaction mixture was filtered through SiO₂ using EtOAc as eluent. The yield was determined using ¹H NMR spectroscopy and mesitylene as the internal standard.

Table S6. Reduction of β -epoxy cedren-9-ol (*cis*-9) with H_2 using various catalysts.



entry	catalyst	additive	solvent	$p(\text{H}_2)$ / bar	T / °C	t / h	yield / %
1	[Fe] ^[a]		MeOH	20	100	24	— ^[b]
2	[Fe] ^[a]	TsOH	MeOH	20	100	24	— ^[b]
3	$\text{Co}_2(\text{CO}_2)_8$		MeOH	20	100	24	no conversion
4	Pd/C ^[c]		EtOH	20	100	24	— ^[d]
5	Pd/C ^[c]		EtOH	4	23	4	—
6	Pd/C ^[c]	NaOH	EtOH	4	23	4	—
7	Pd/C ^[c]	Na ₂ CO ₃	EtOH	4	23	4	—
8			EtOH	4	23	4	—
9		Na ₂ CO ₃	EtOH	4	23	4	—
10	Pd/C ^[c]		DMP	4	23	6	—
11	Pd/C ^[c]		EtOAc	4	23	6	—
12	Pd/C ^[e]		EtOH	4	23	5.5	—
13	Pd/C ^[e]	Na ₂ CO ₃	EtOH	4	23	5.5	—
14	Pd/C ^[f]		EtOH	4	23	6	—
15	Pd(OH) ₂ /C		EtOH	4	23	5.5	—
16	Pd(OAc) ₂		EtOH	4	23	6	—
17	Lindlar		EtOH	4	23	6	—
18	Ru/C ^[g]		EtOH	4	23	6	—
19 ^[h]	Pd/C ^[c]		EtOH	4	23	5.5	no conversion

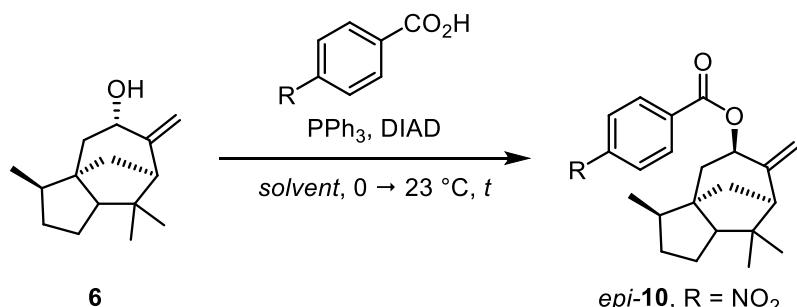
^[a] [Fe] = [FeF(tris(2-diphenylphosphino)phenyl)phosphine]BF₄⁵ ^[b] β -Cedren-9-ol (**6**) was obtained quantitatively. ^[c]

Pd/C (10 wt%) was purchased from abcr. ^[d] Cedralon (**4**) was obtained quantitatively. ^[e] Pd/C (10 wt%) was purchased from Fluka. ^[f] Pd/C (10 wt%) was purchased from Sigma-Aldrich. ^[g] Ru/C with 5 wt% Ru. ^[h] *cis*-Cedrene diol (*cis*-**3**) was used as starting material.

5. Optimization of the Mitsunobu reaction of β -cedren-9-ol (**6**)

Table S7 shows the optimization of the Mitsunobu reaction of allyl alcohol **6**.

Table S7. Optimization of the conversion of β -cedren-9-ol (**6**) to β -cedrenyl *p*-nitrobenzoate (**8**) under Mitsunobu conditions.



entry	acid R (equiv)	equiv PPh_3	equiv DIAD	solvent (v:v)	t / h	yield <i>epi</i> - 10 / %
1	H (4)	4	4	THF	24	—
2	NO_2 (4)	4	4	toluene/THF (3:1)	43	74
3	NO_2 (4)	4	4	toluene/THF (2:1)	96	88
4	NO_2 (4)	4	4	THF	72	64
5	NO_2 (1.3)	1.1	1.1	toluene/THF (5:2)	42	87
6 ^[a]	NO_2 (1.3)	1.1	1.1	toluene/THF (5:2)	42	84

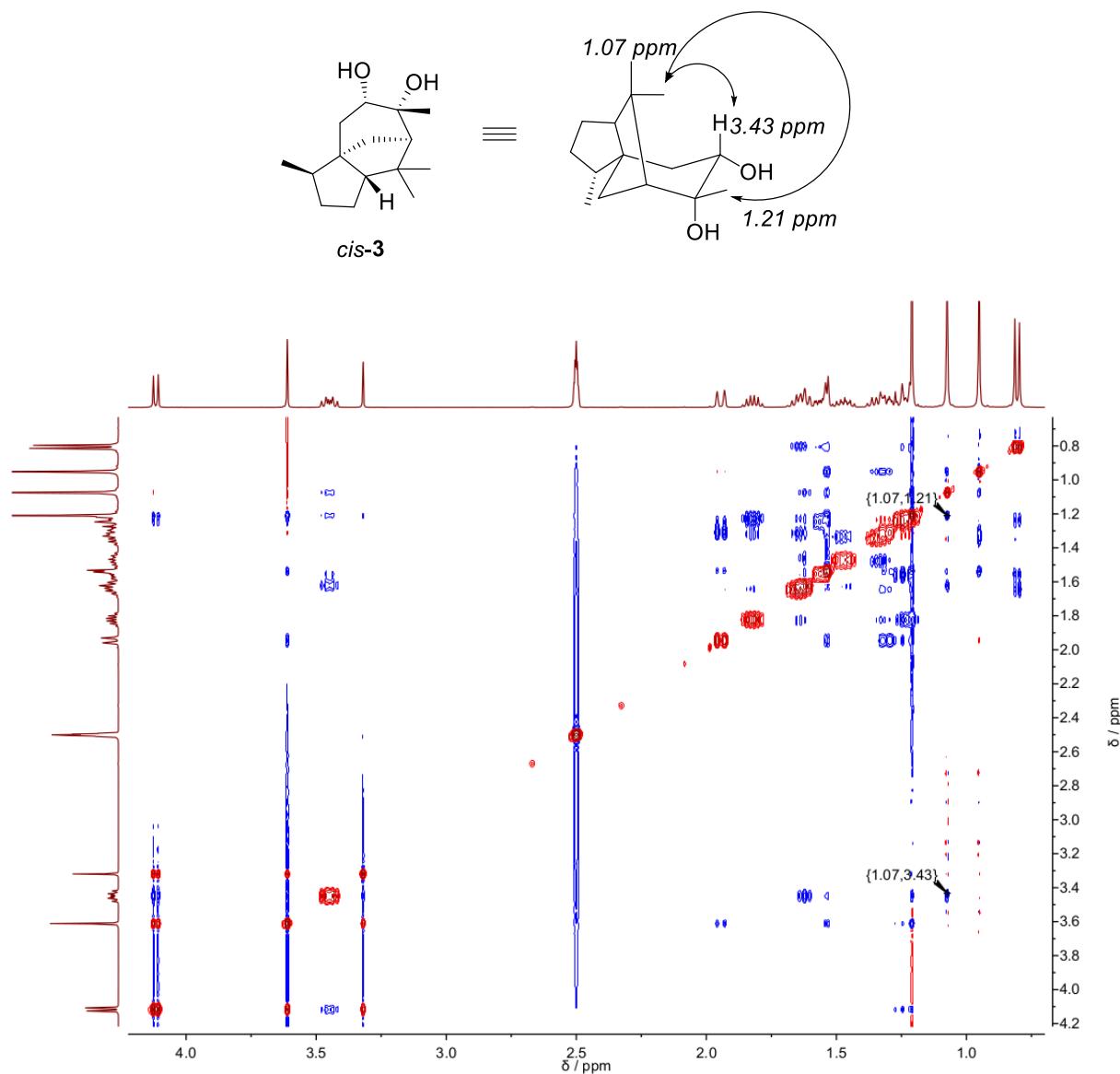
^[a] 20 mmol β -cedren-9-ol (**6**) were used as starting material.

6. Additional procedures

***p*-Nitrobenzoyl chloride.**¹ *p*-Nitro benzoic acid (1 g, 6 mmol) and $SOCl_2$ (2.14 g, 18.0 mmol) were stirred at 80 $^{\circ}C$ for 24 h. The excess of $SOCl_2$ was removed via co-evaporation with toluene (4×3 mL). The resulting solid was dried in vacuo to obtain the product (1.1 g, 6.0 mmol, 99%) as pale yellow solid and used as received without further purification. 1H NMR (300 MHz, $CDCl_3$): δ = 8.24–8.42 (m, 4H) ppm.

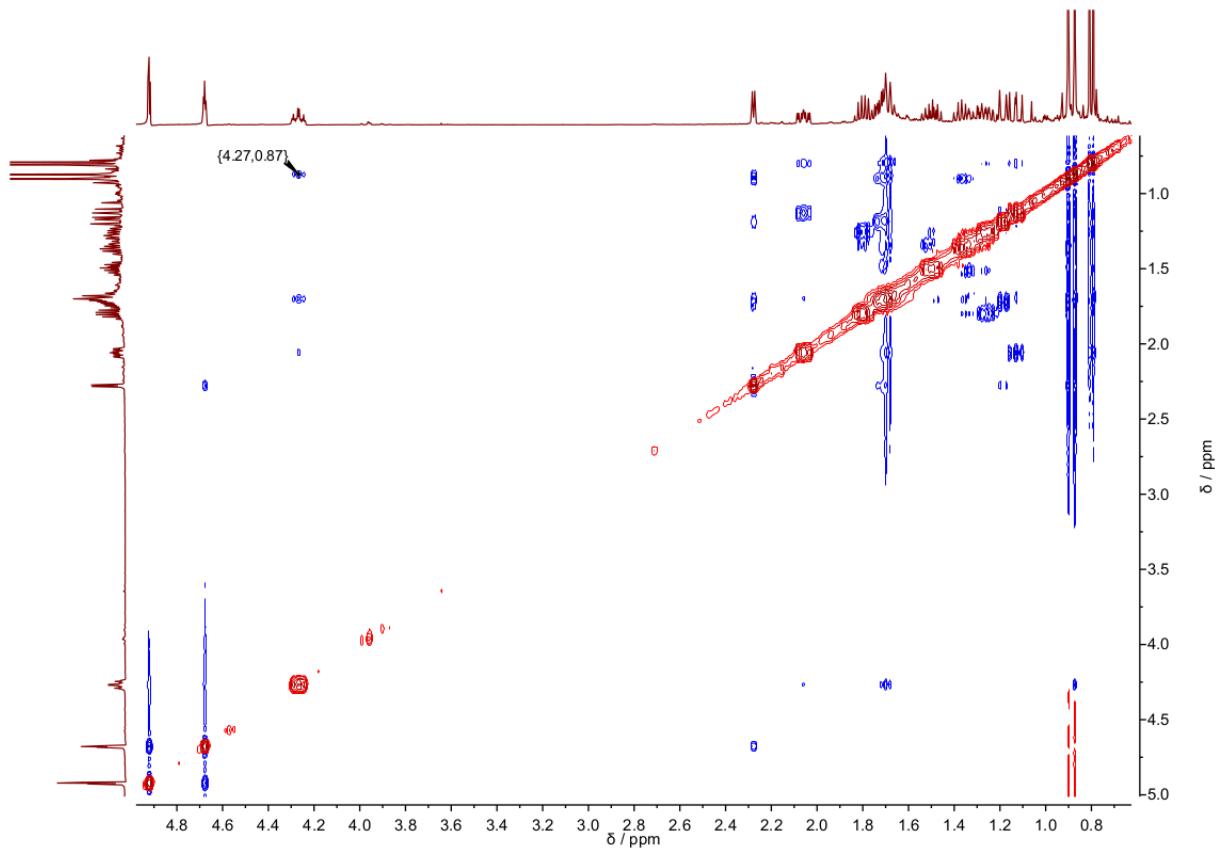
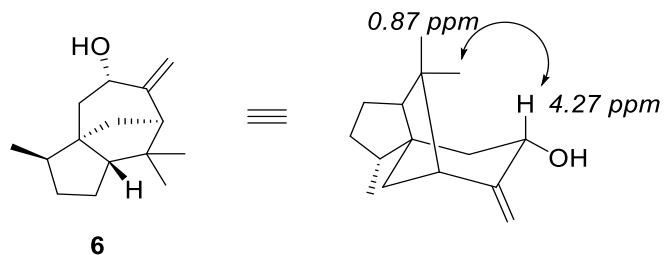
7. Selected NOESY spectra

NOESY spectrum of *cis*-cedrene-8,9-diol (*cis*-3) in DMSO-d₆



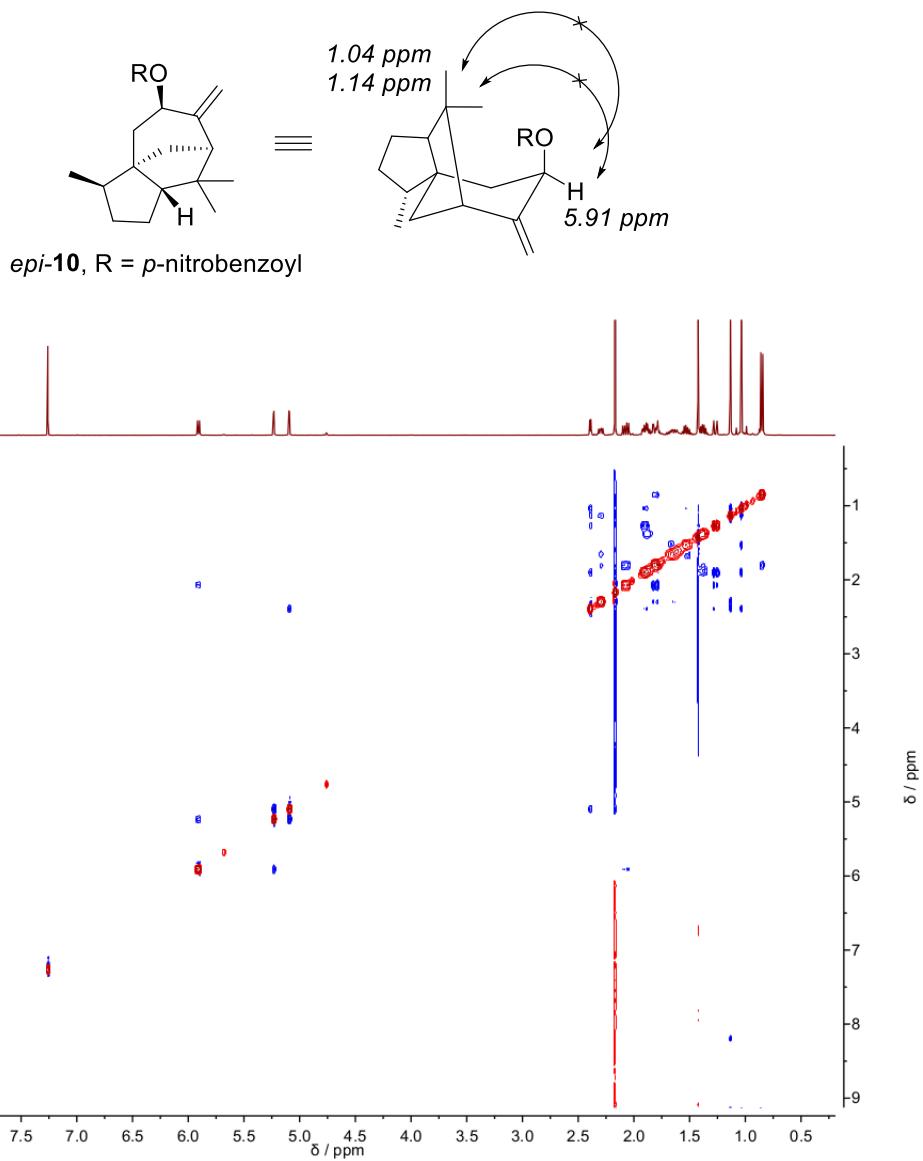
The correlation between the Me-group of the 2-propylidene bridge at 1.07 ppm and the proton at 3.43 ppm on the one hand and the methyl group at 1.21 ppm on the other hand confirmed the stereochemistry of the diol *cis*-3 as shown.

NOESY spectrum of β -cedren-9-ol (**6**) in CDCl_3

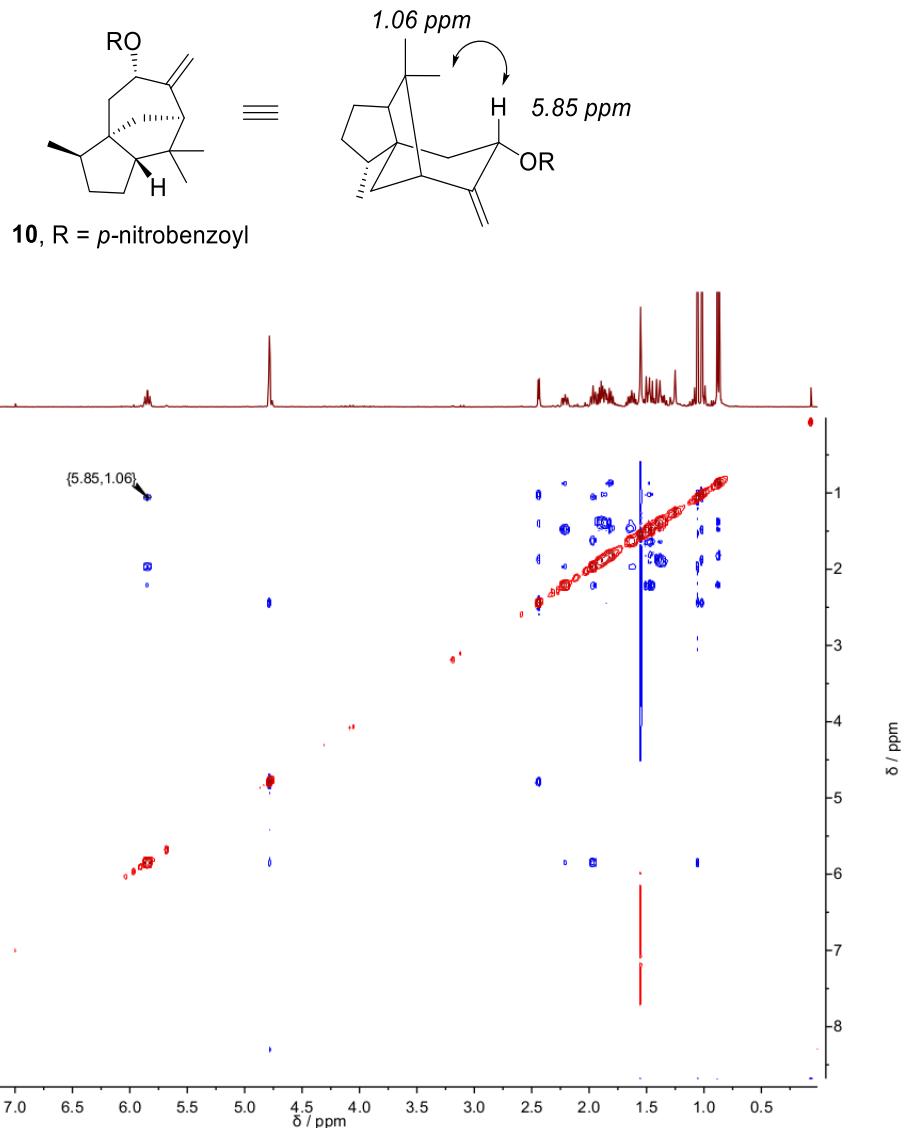


The NOE between the proton at 4.27 ppm and the Me-group of the 2-propylidene bridge confirmed the stereochemistry as shown.

NOESY spectrum of *epi*- β -cedrenyl *p*-nitrobenzoate (*epi*-10) in CDCl_3

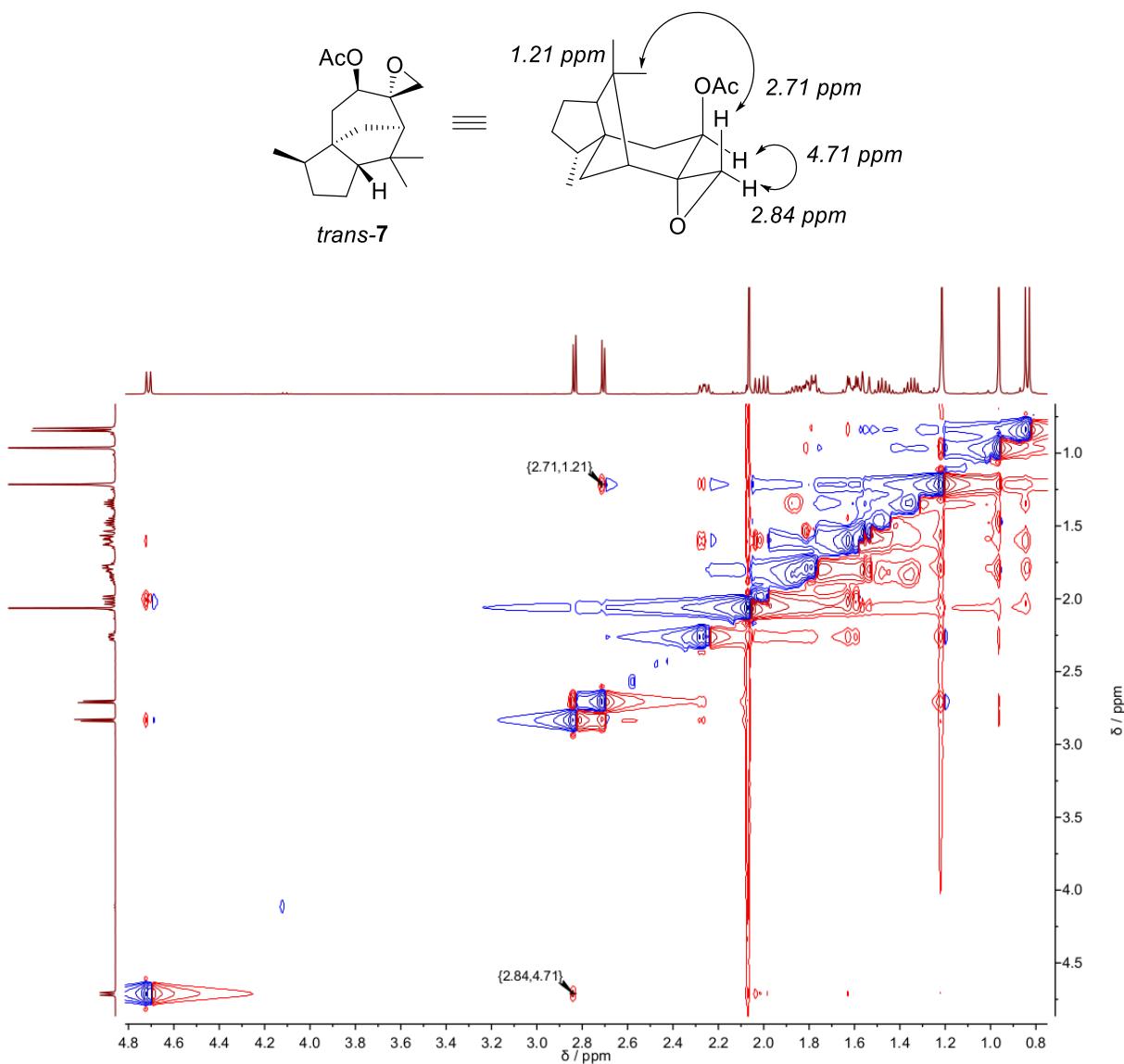


NOESY spectrum β -cedrenyl *p*-nitrobenzoate (**10**) in CDCl_3



In comparison to compound *epi*-**10** the presence of an NOE between the Me-group of the 2-propylidene bridge and the proton at 5.85 ppm confirmed the stereochemistry as shown.

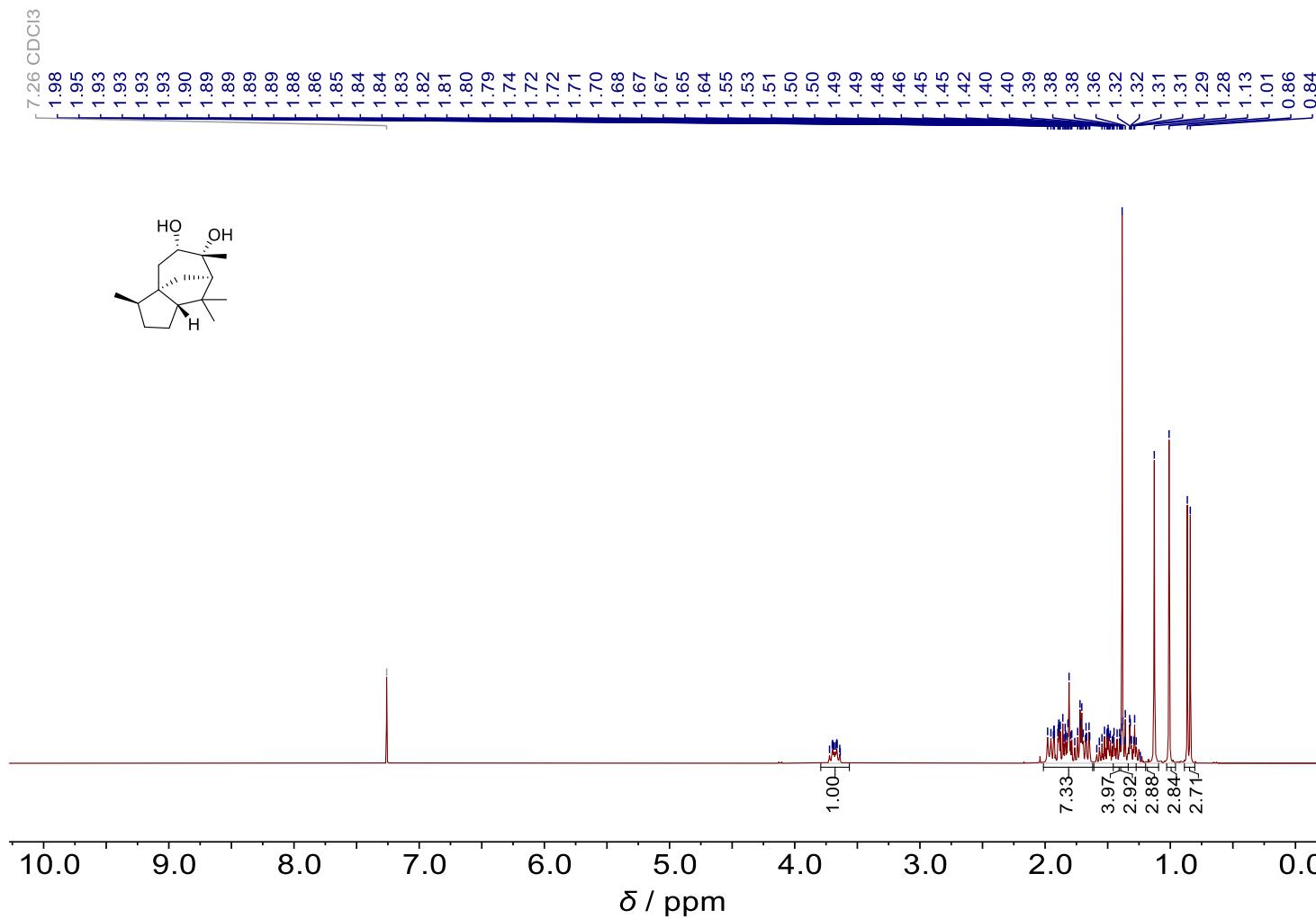
NOESY spectrum of the epoxidized β -cedrenyl acetate *trans*-7 in CDCl_3



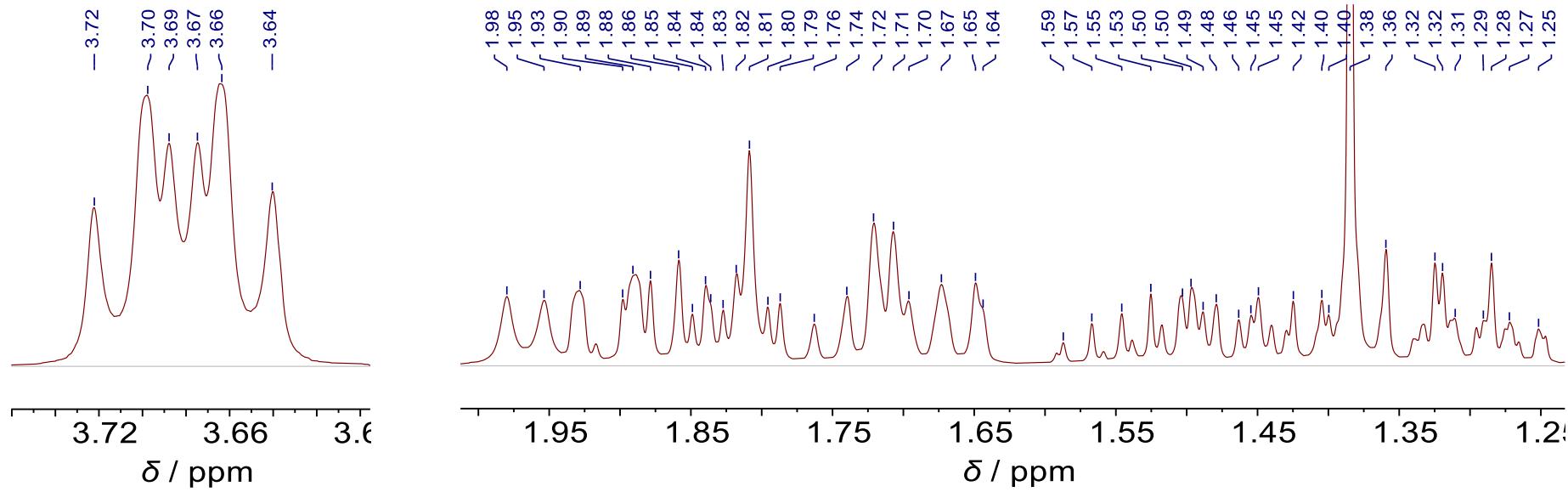
The correlation between the protons of the oxirane ring ($-\text{CH}_2\text{O}-$) at $\delta = 2.71$ ppm and the methyl protons ($-\text{CH}_3$) of the 2-propylidene bridge at $\delta = 1.21$ ppm confirmed that the epoxidation proceeded *trans* with respect to the acetate. Furthermore, a correlation signal between the proton at $\delta = 4.72$ ppm and the propylidene bridge was absent.

8. NMR spectra of synthesized compounds

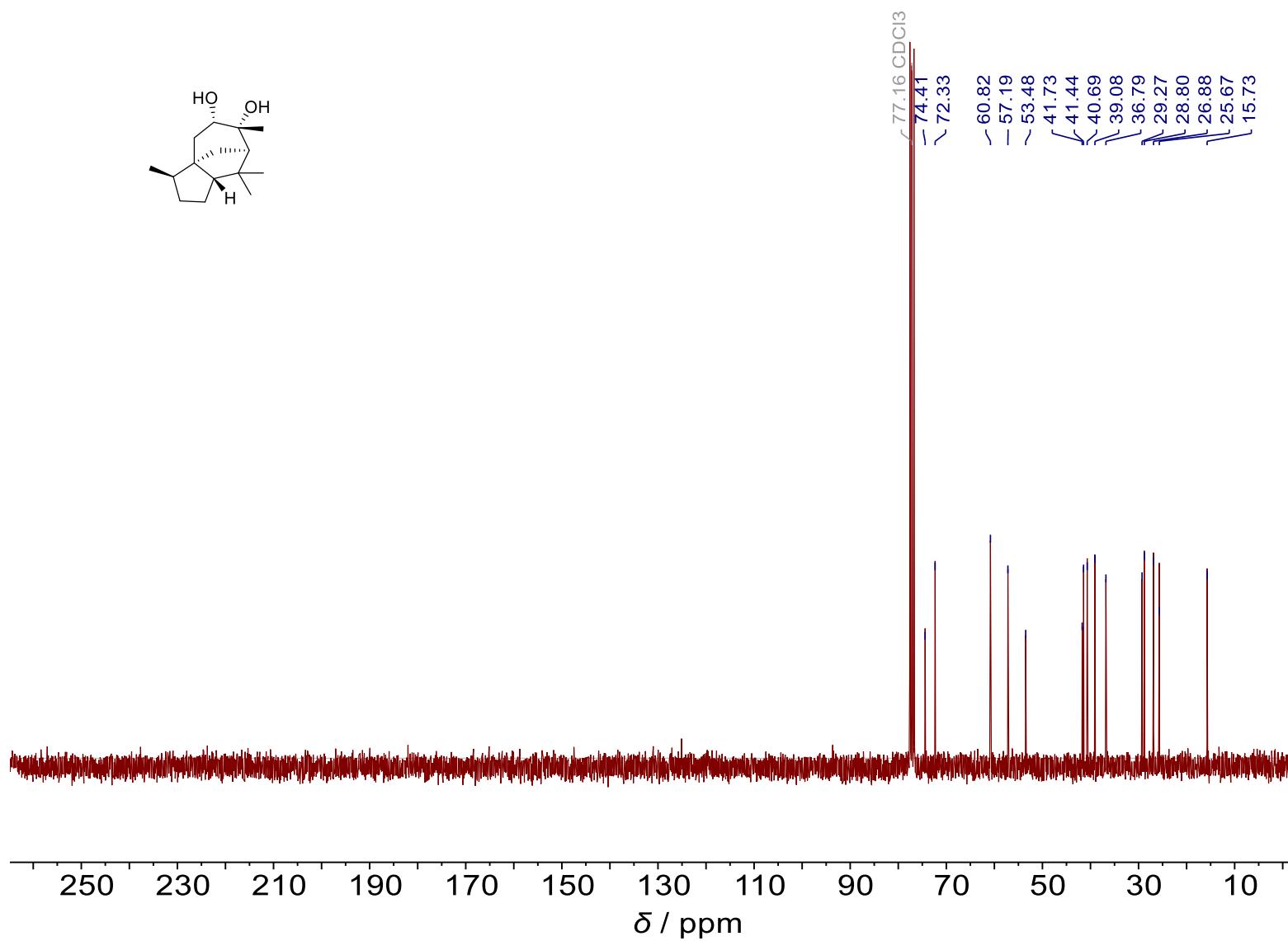
^1H NMR spectrum of *cis*-cedrene-8,9-diol (*cis*-3) in CDCl_3



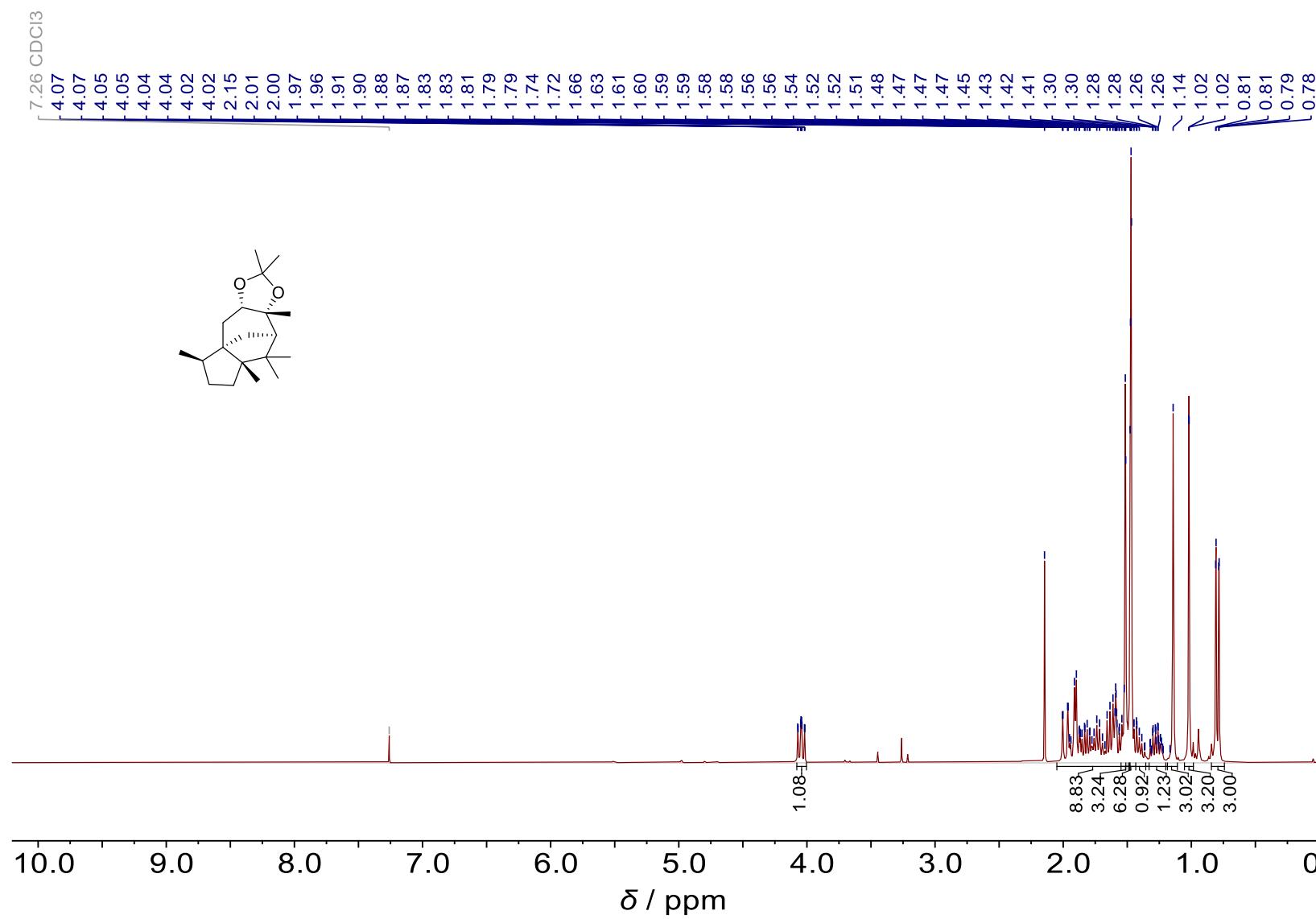
Expansions of the ^1H NMR spectrum of *cis*-cedrene-8,9-diol (*cis*-3) in CDCl_3



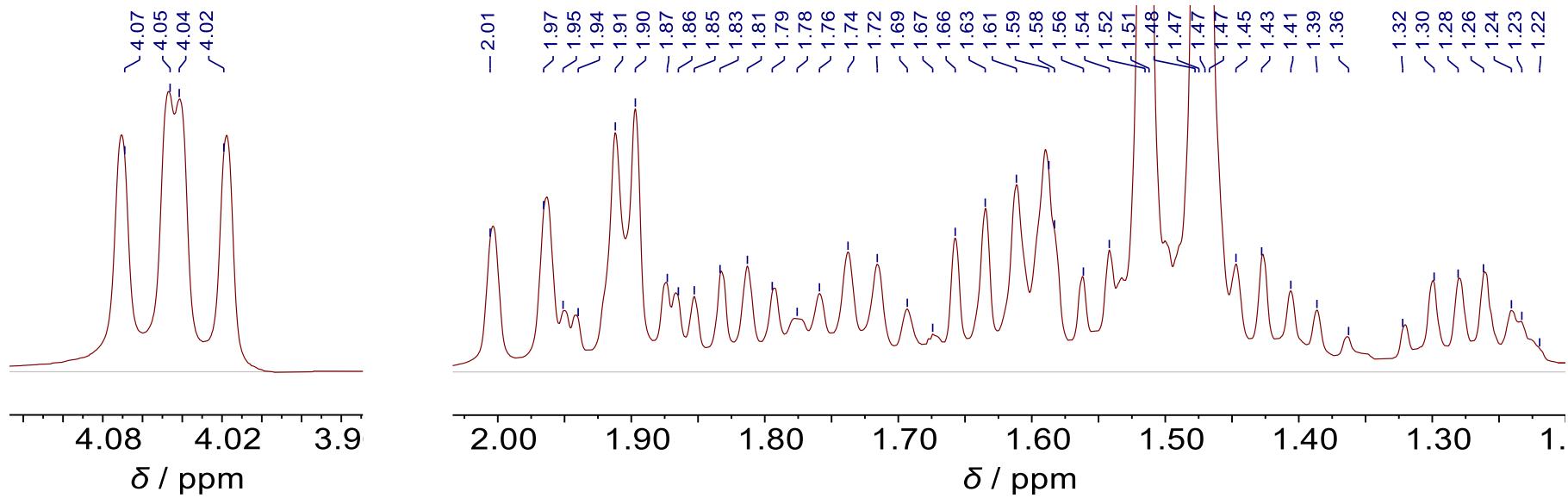
^{13}C NMR spectrum of *cis*-cedrene-8,9-diol (*cis*-3) in CDCl_3



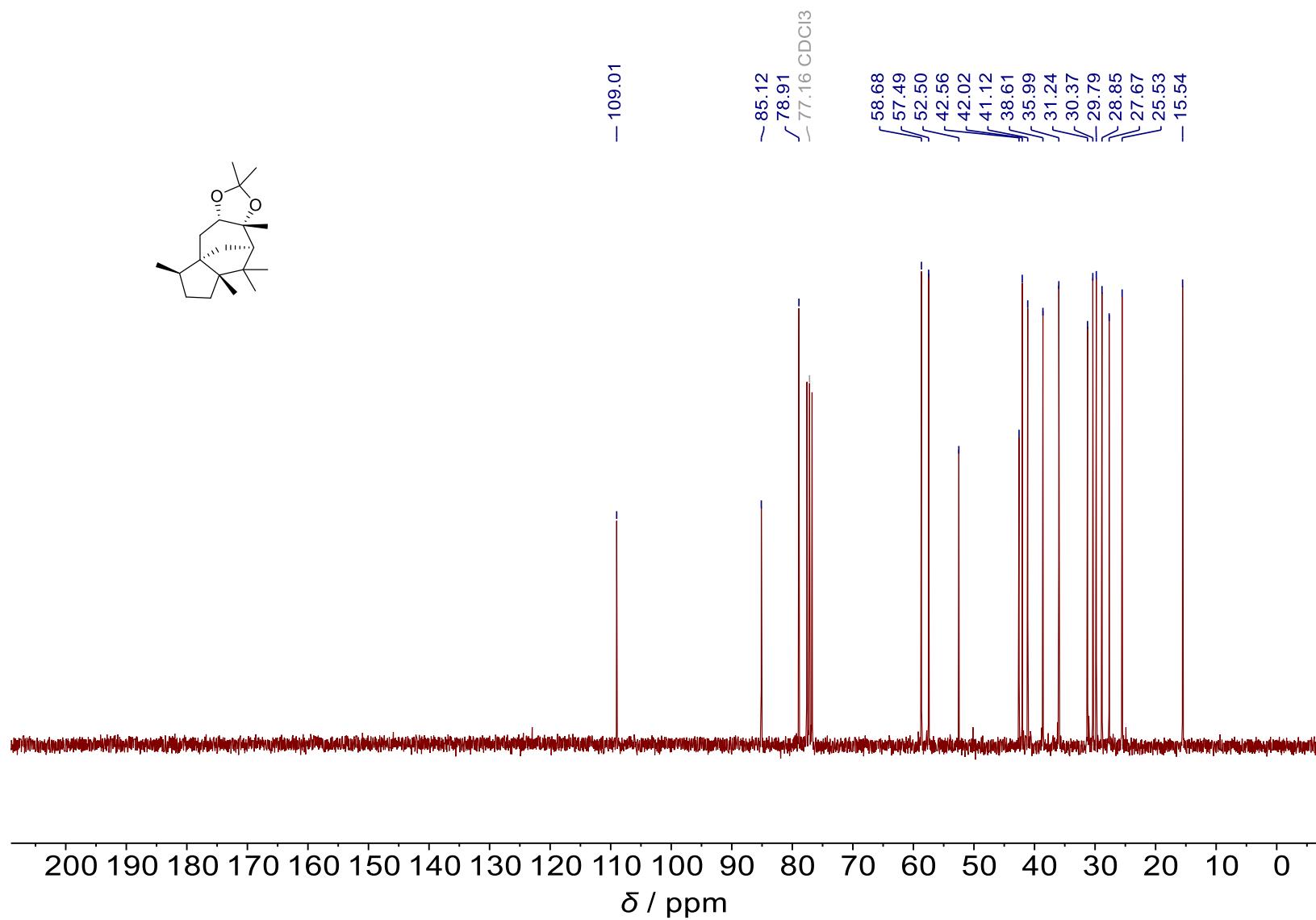
¹H NMR spectrum of *cis*-cedrene acetonide (*cis*-5a) in CDCl₃



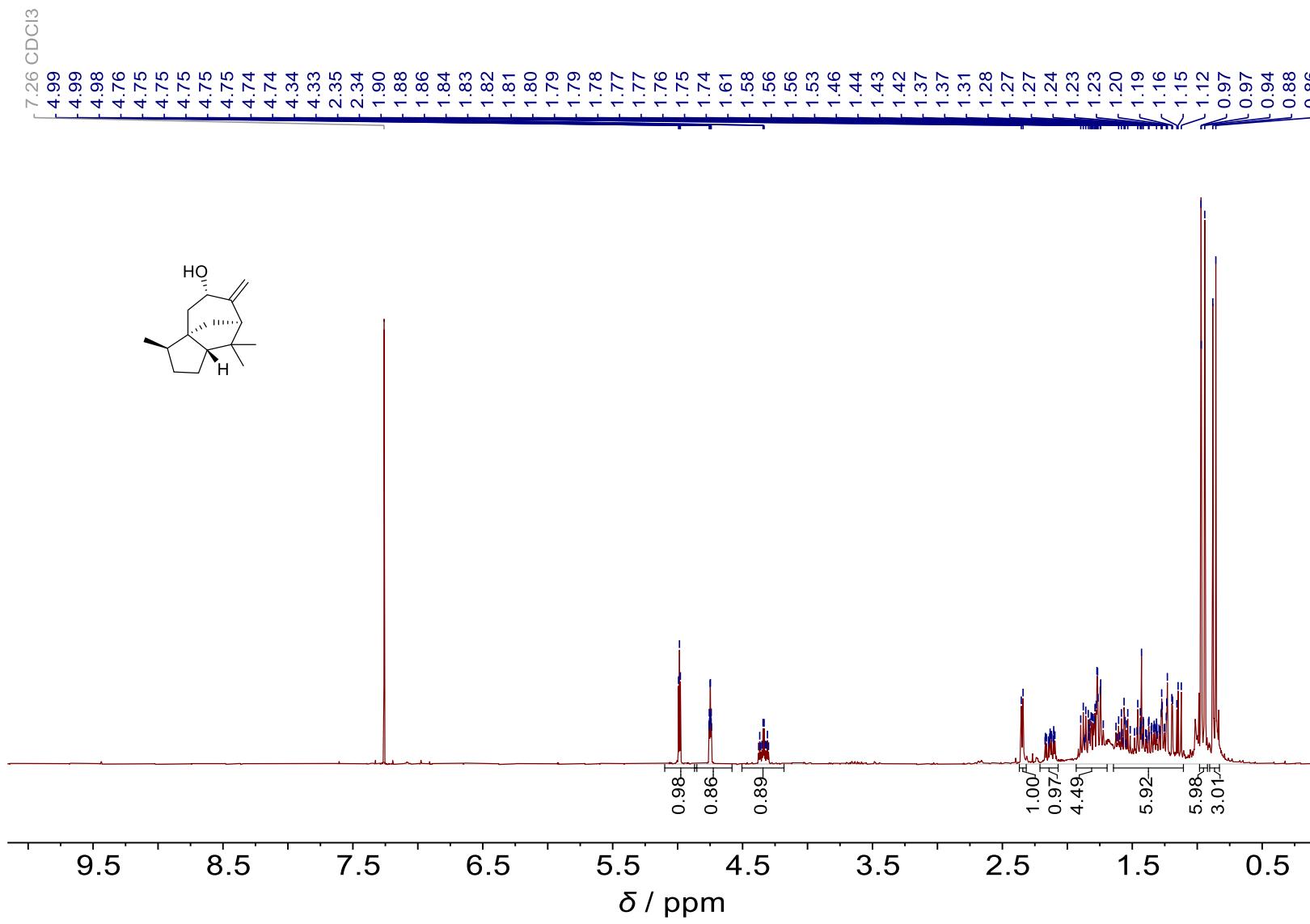
Expansions of the ^1H NMR spectrum of *cis*-cedrene acetonide (*cis*-5a) in CDCl_3



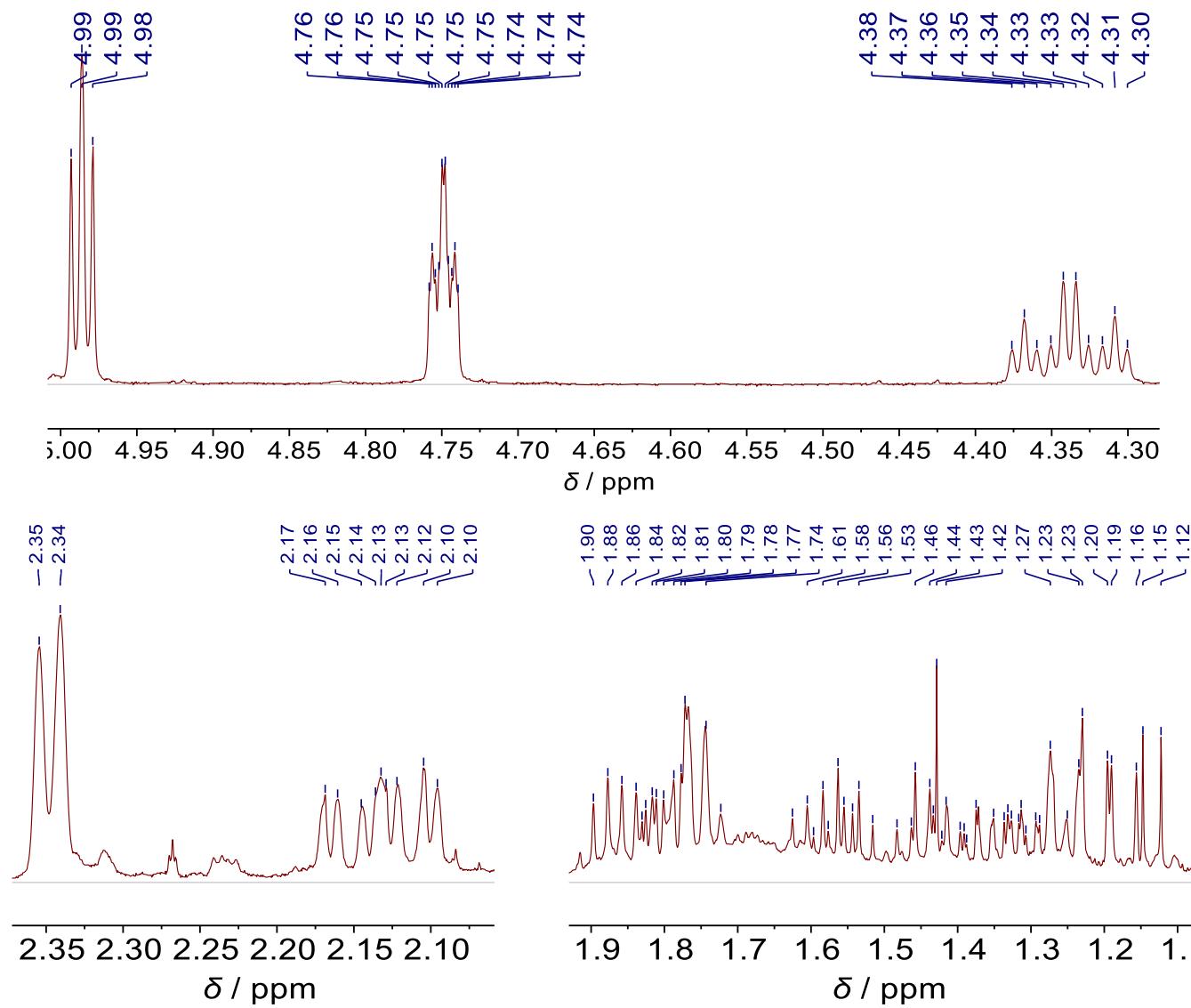
^{13}C NMR spectrum of *cis*-cedrene acetonide (*cis*-5a) in CDCl_3



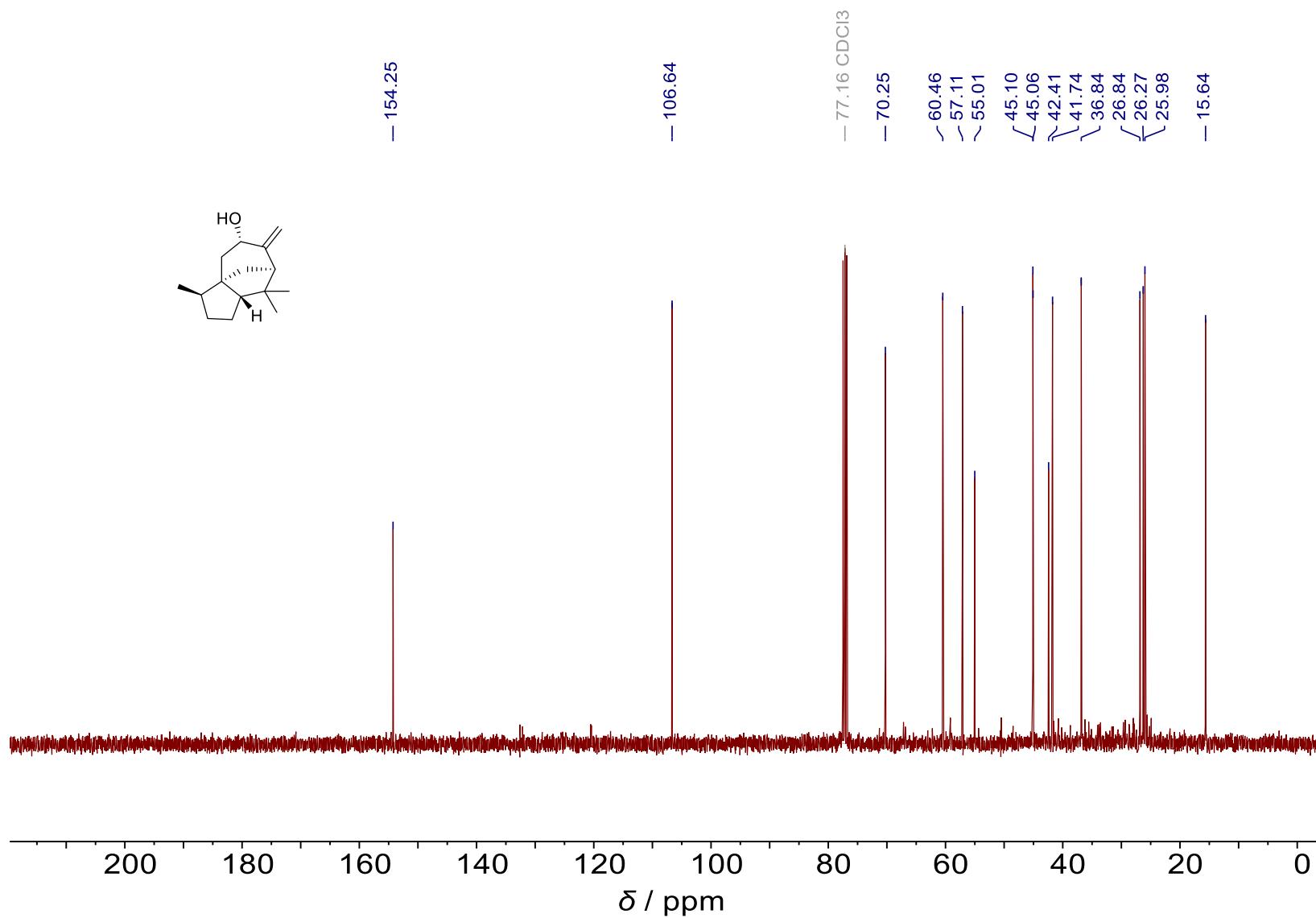
¹H NMR spectrum of β -cedren-9-ol (6) in CDCl_3



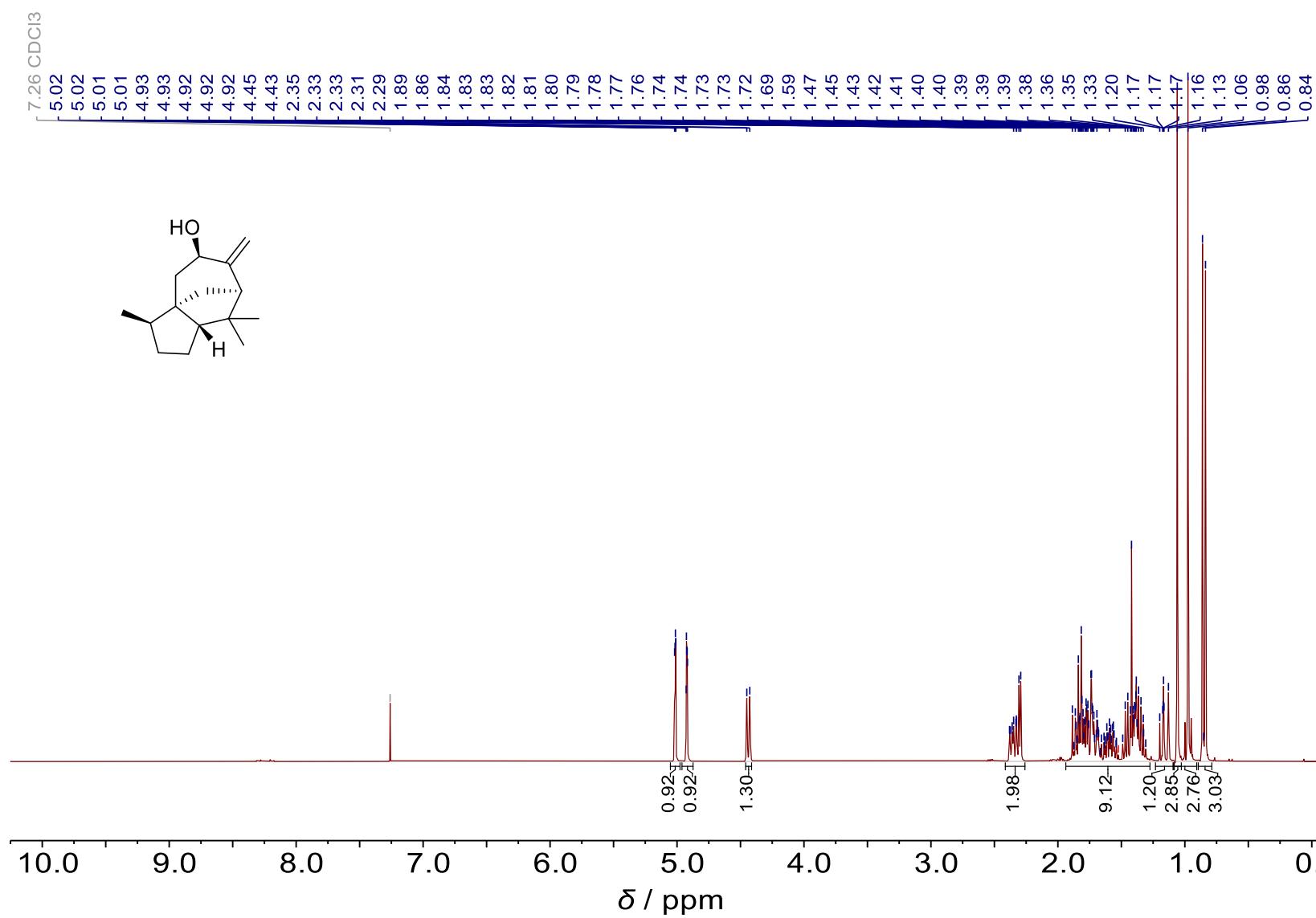
Expansions of the ^1H NMR spectrum of β -cedren-9-ol (**6**) in CDCl_3



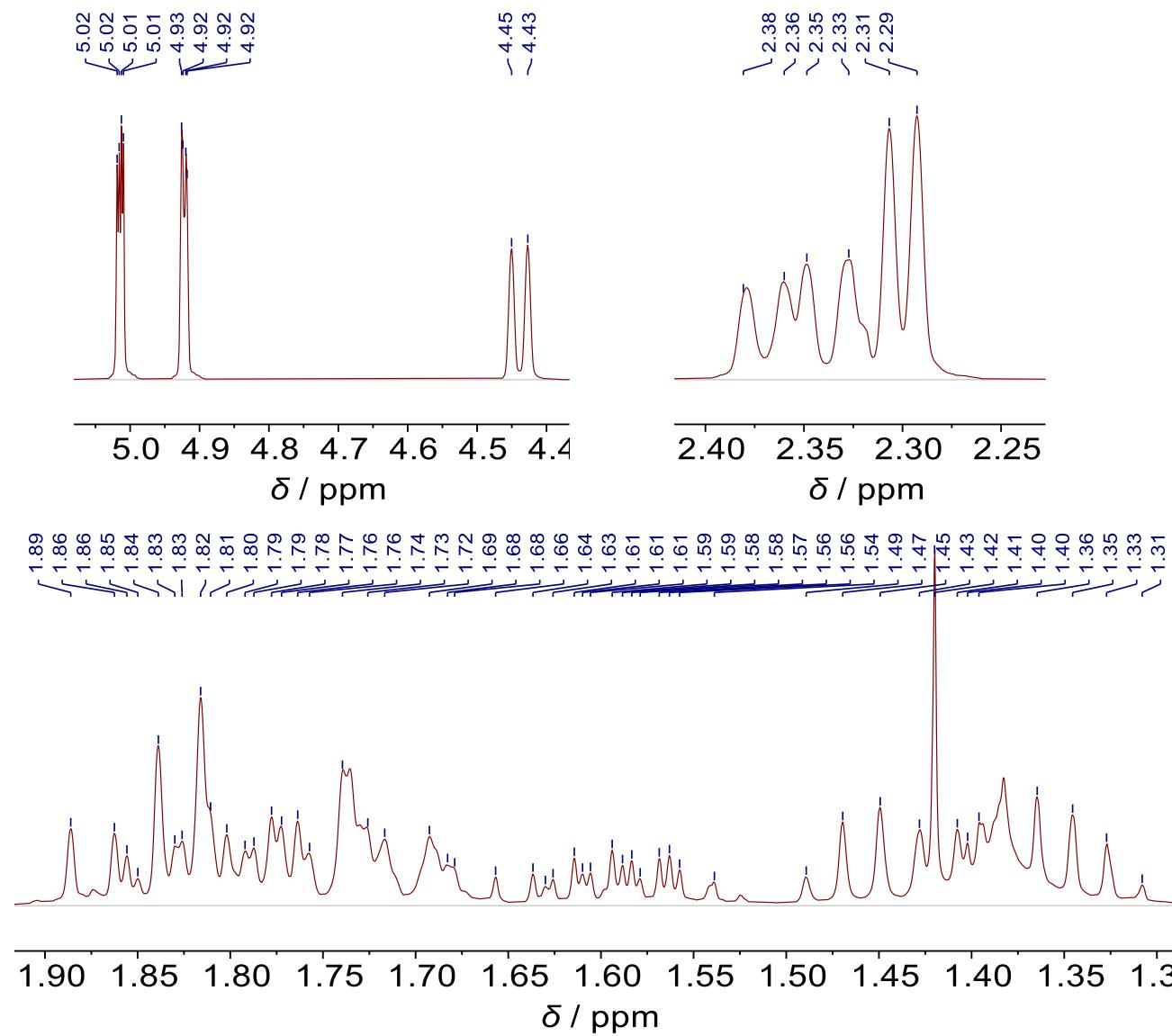
¹³C NMR spectrum of β -cedren-9-ol (6) in CDCl_3



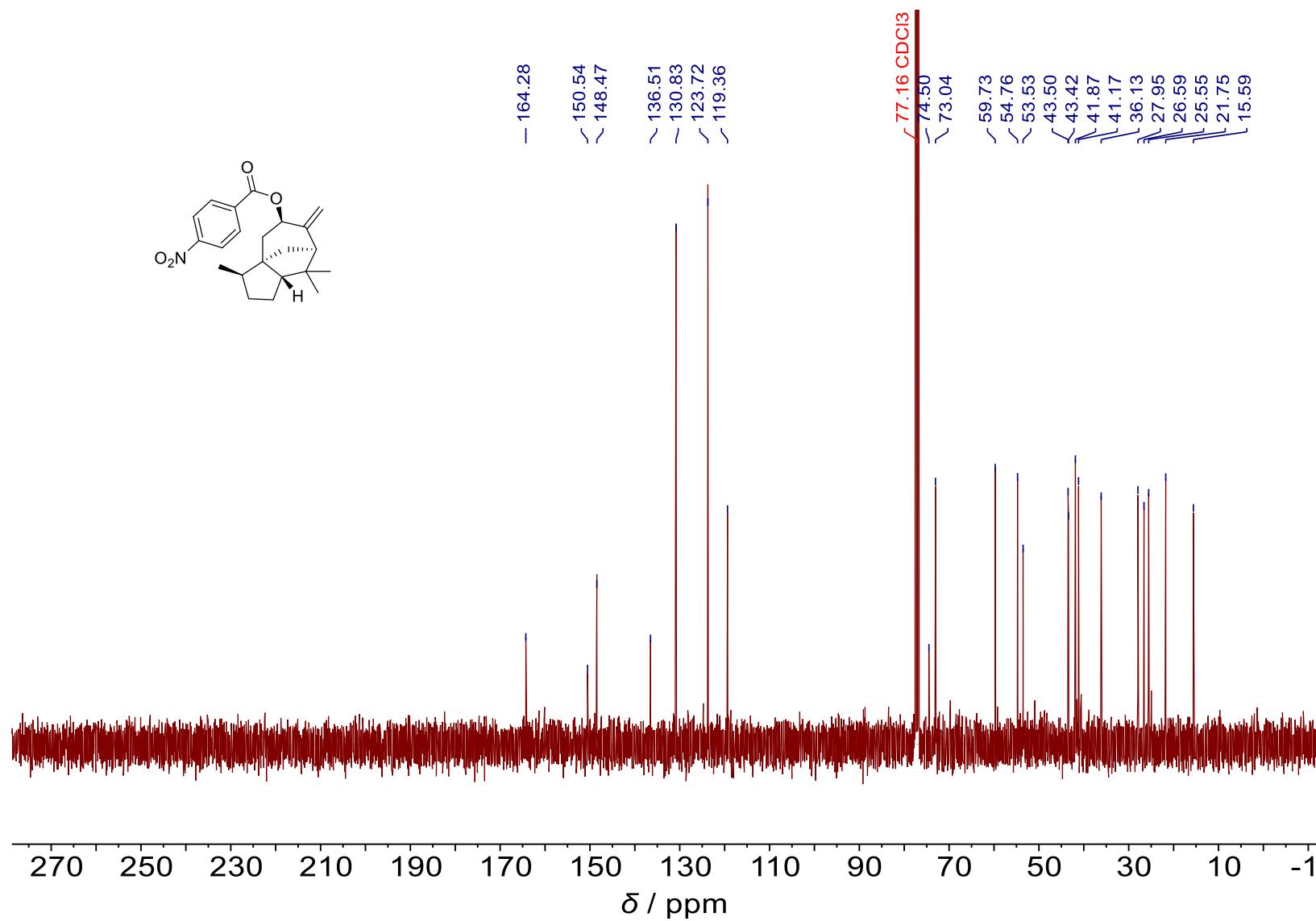
¹H NMR spectrum of *epi*- β -cedren-9-ol (*epi*-6) in CDCl₃



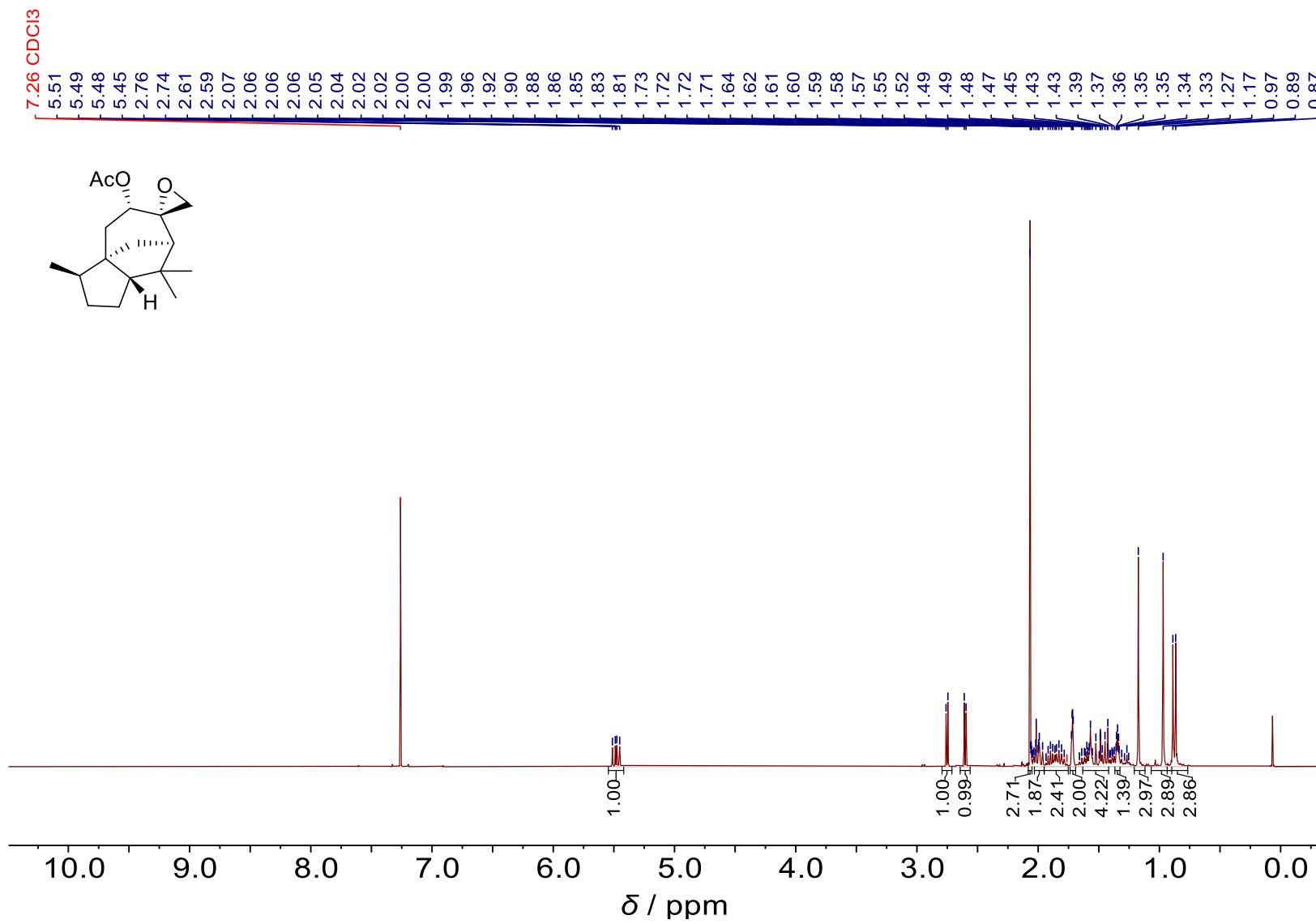
Expansions of the ^1H NMR spectrum of *epi*- β -cedren-9-ol (*epi*-6) in CDCl_3



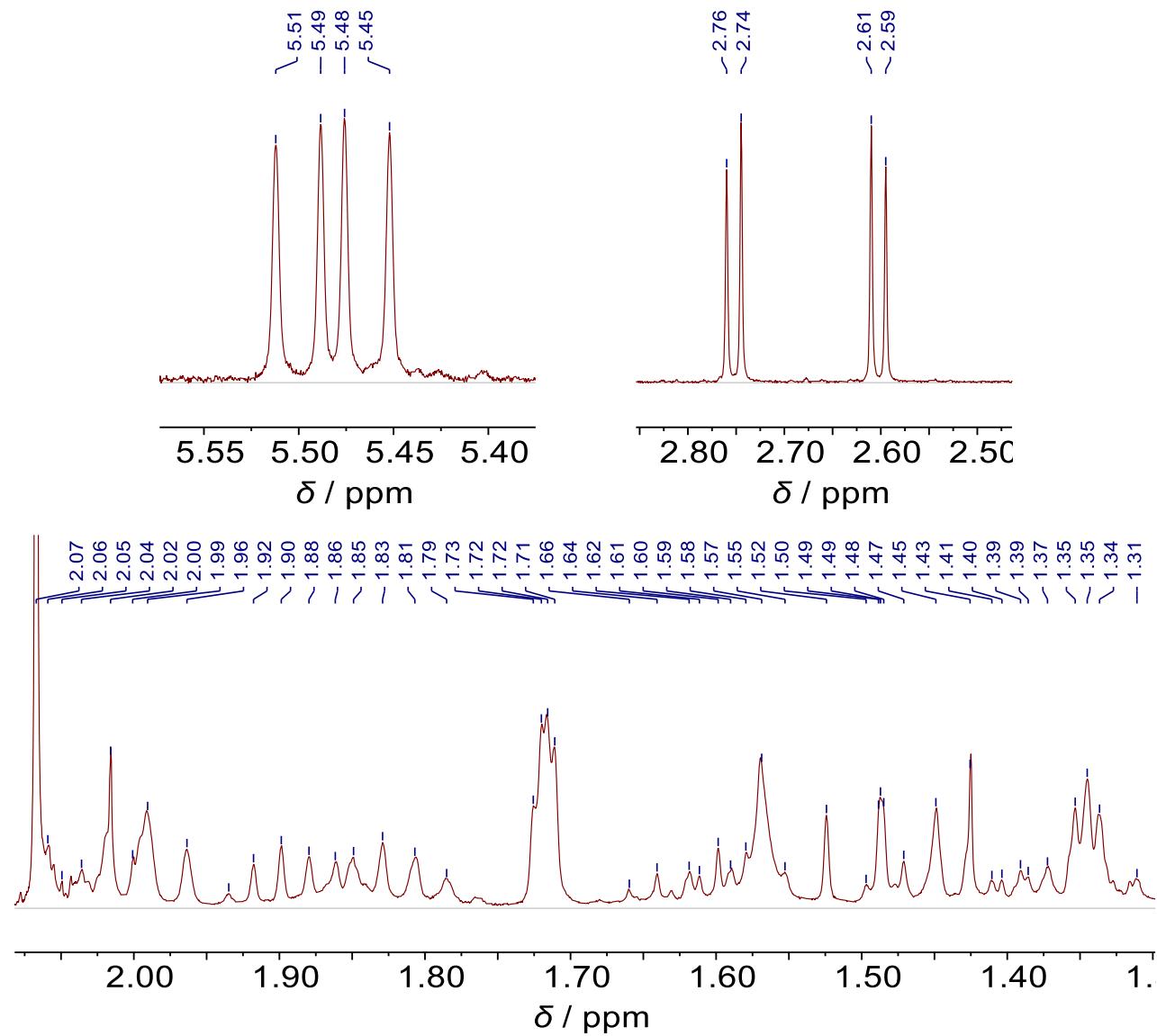
^{13}C NMR spectrum of *epi*- β -cedren-9-ol (*epi*-6) in CDCl_3



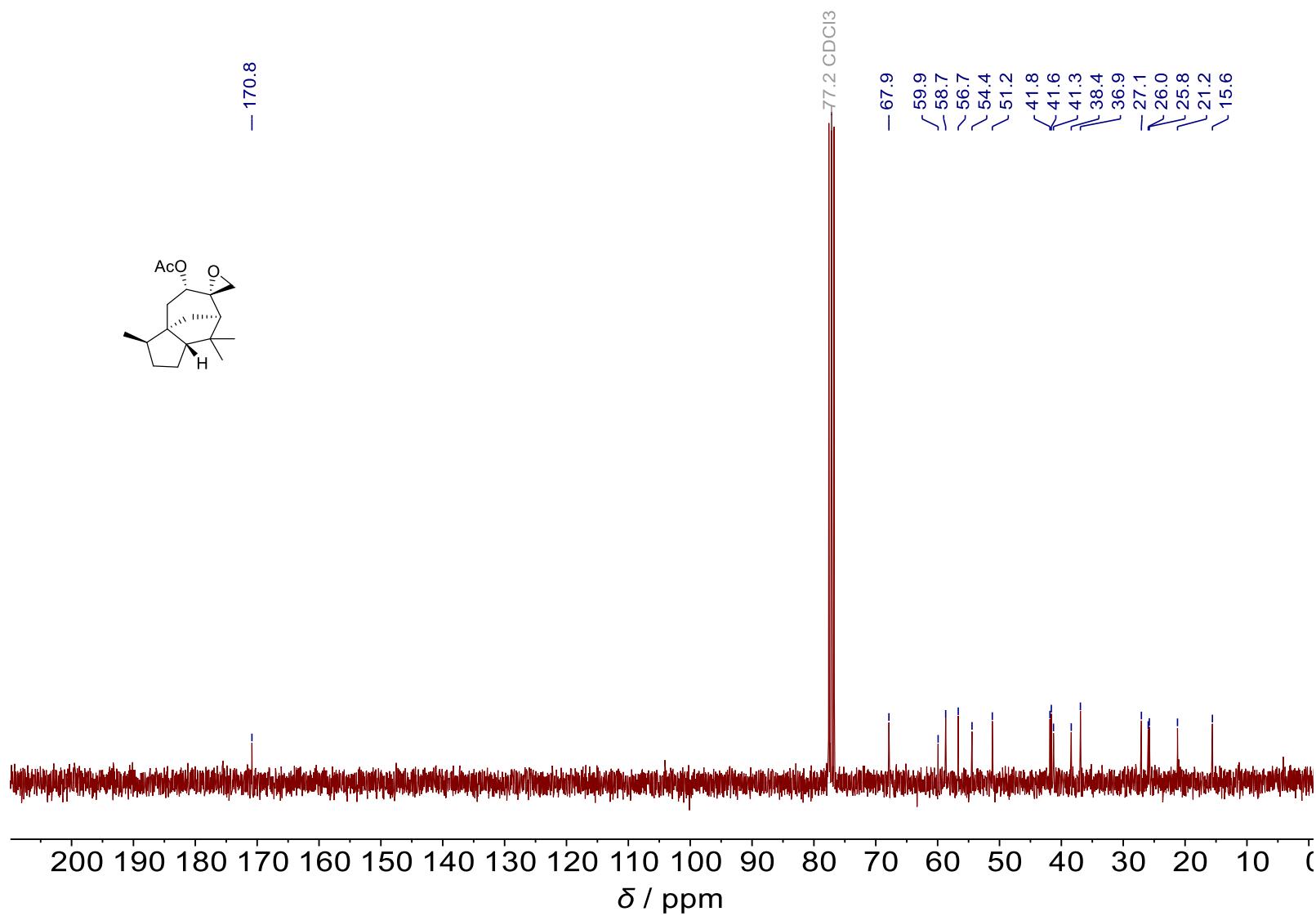
¹H NMR spectrum of β -epoxy cedrenyl acetate (*cis*-7) in CDCl_3



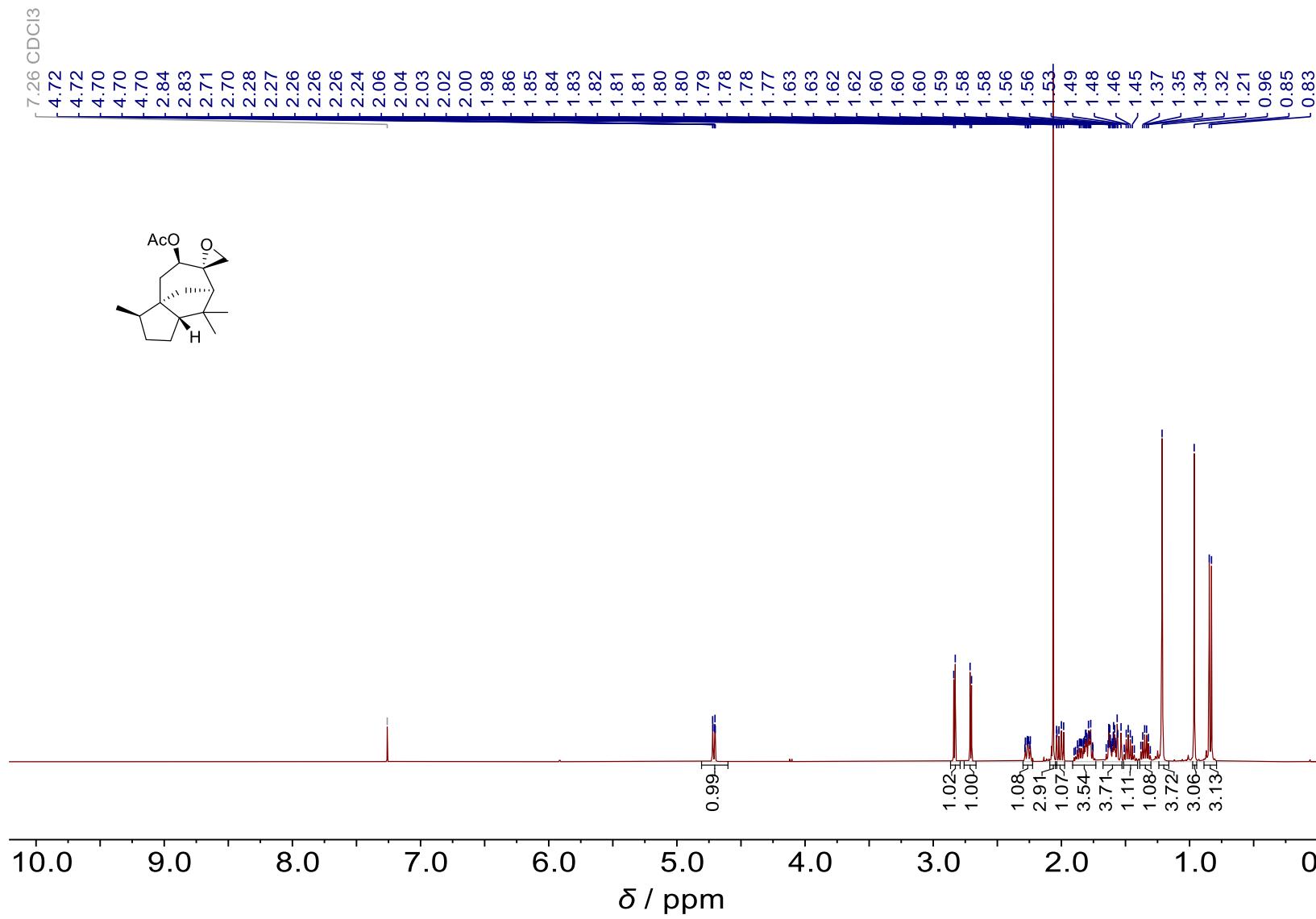
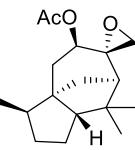
Expansions of the ^1H NMR spectrum of β -epoxy cedrenyl acetate (*cis*-7) in CDCl_3



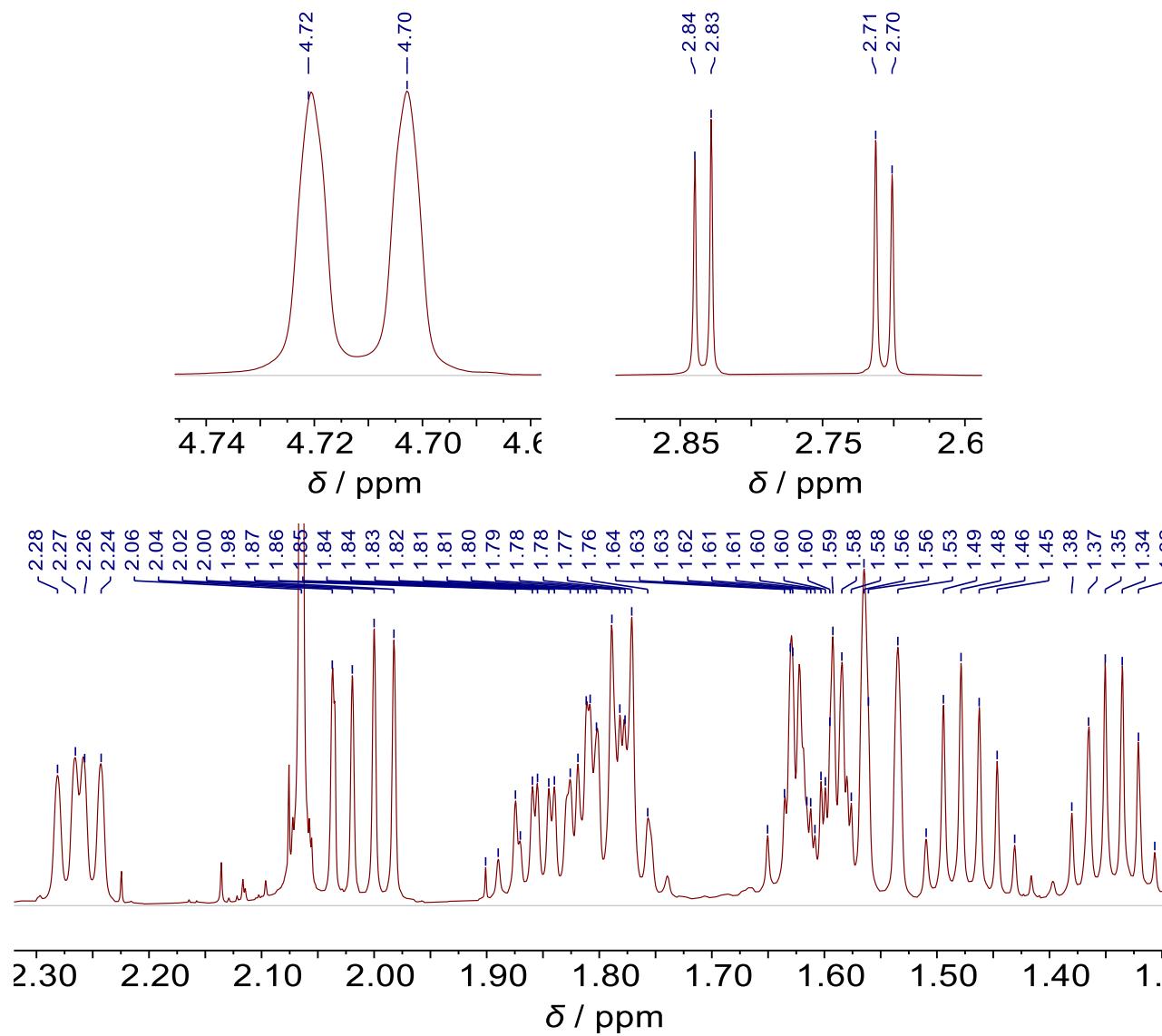
^{13}C NMR spectrum of β -epoxy cedrenyl acetate (*cis*-7) in CDCl_3



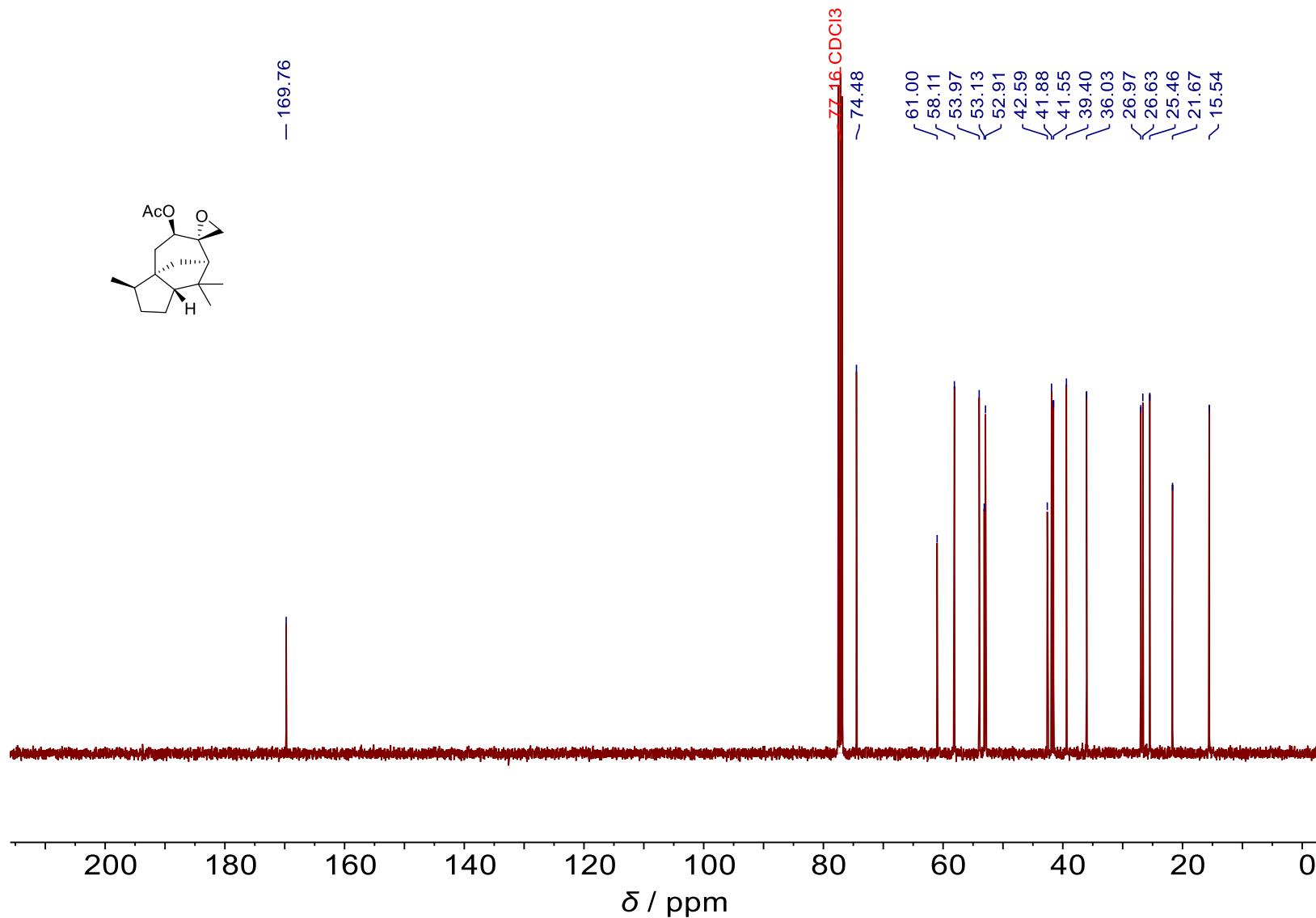
¹H NMR spectrum of the *trans*- β -epoxy cedrenyl acetate *trans*-7 in CDCl₃



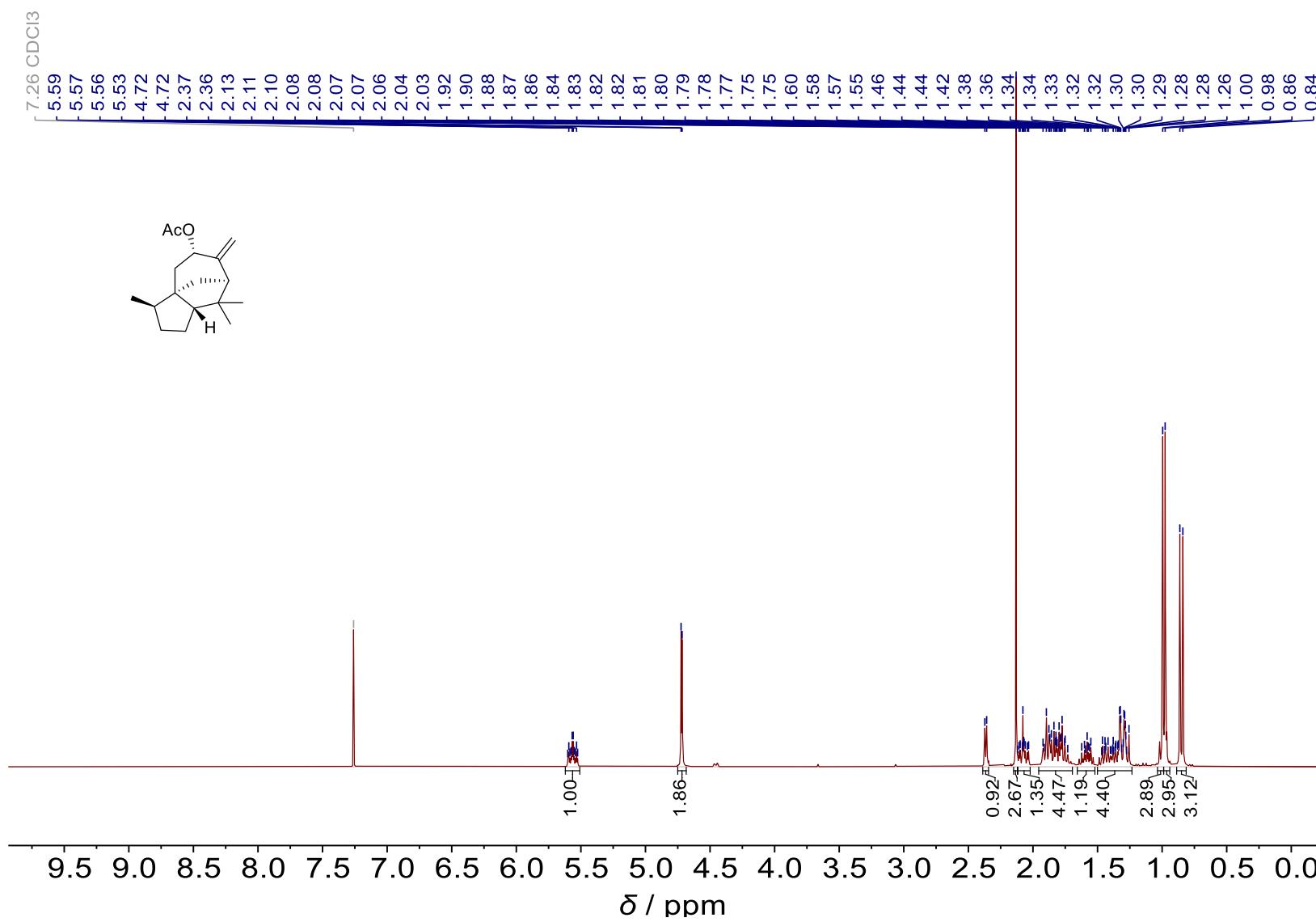
Expansions of the ^1H NMR spectrum of the *trans*- β -epoxy cedrenyl acetate *trans*-7 in CDCl_3



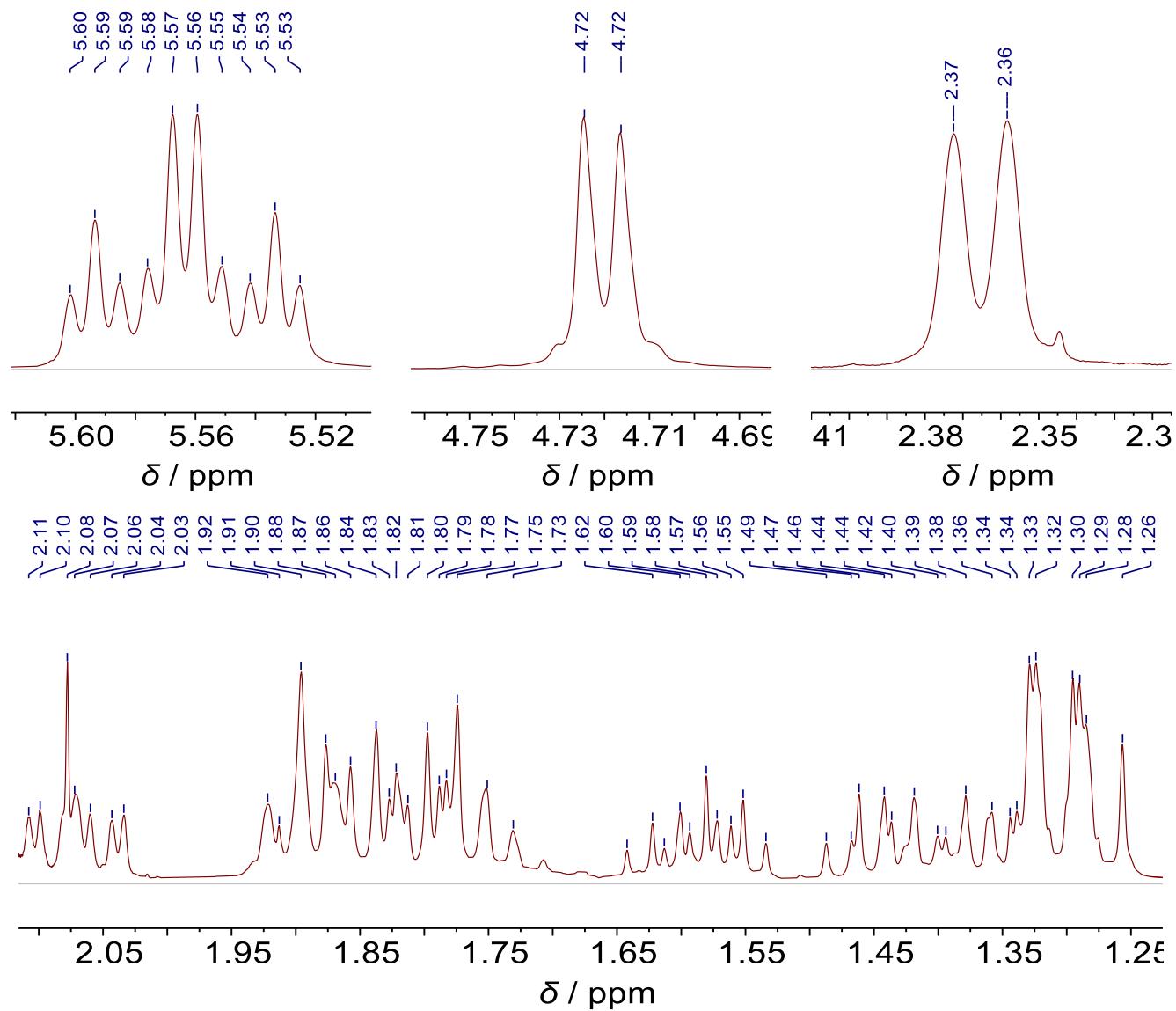
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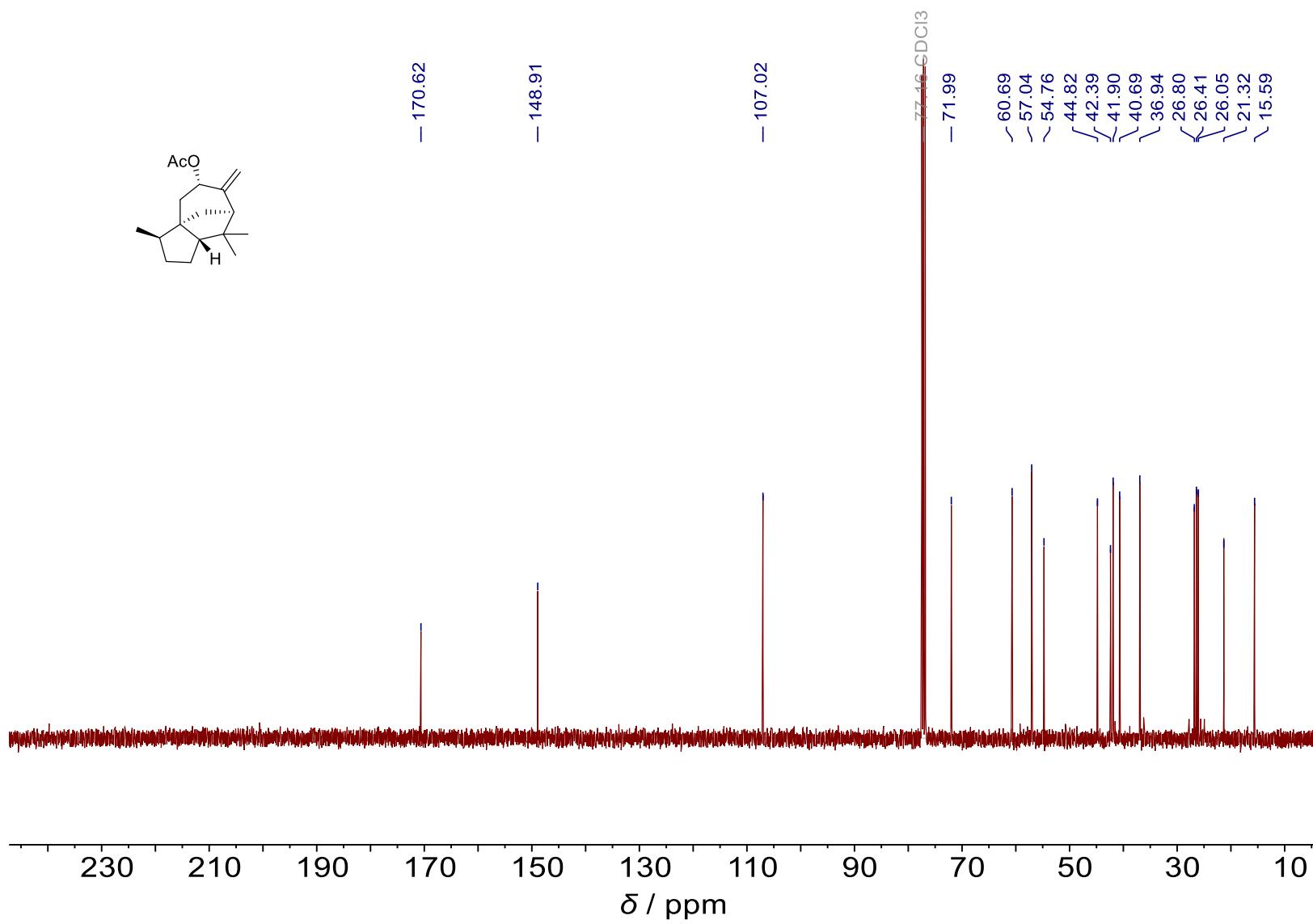
¹H NMR spectrum of β -cedrenyl acetate (8) in CDCl₃



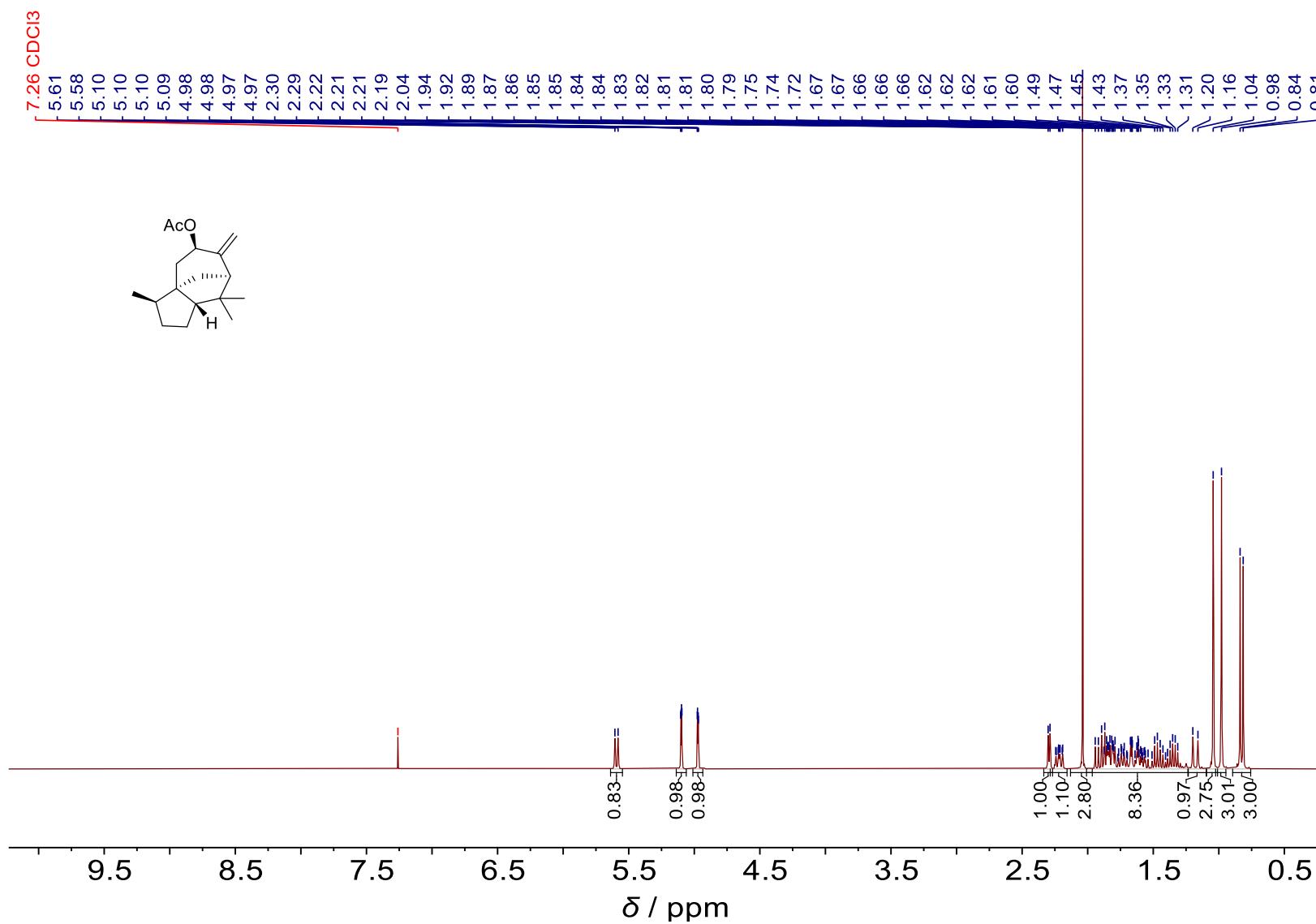
Expansions of the ^1H NMR spectrum of β -cedrenyl acetate (8) in CDCl_3



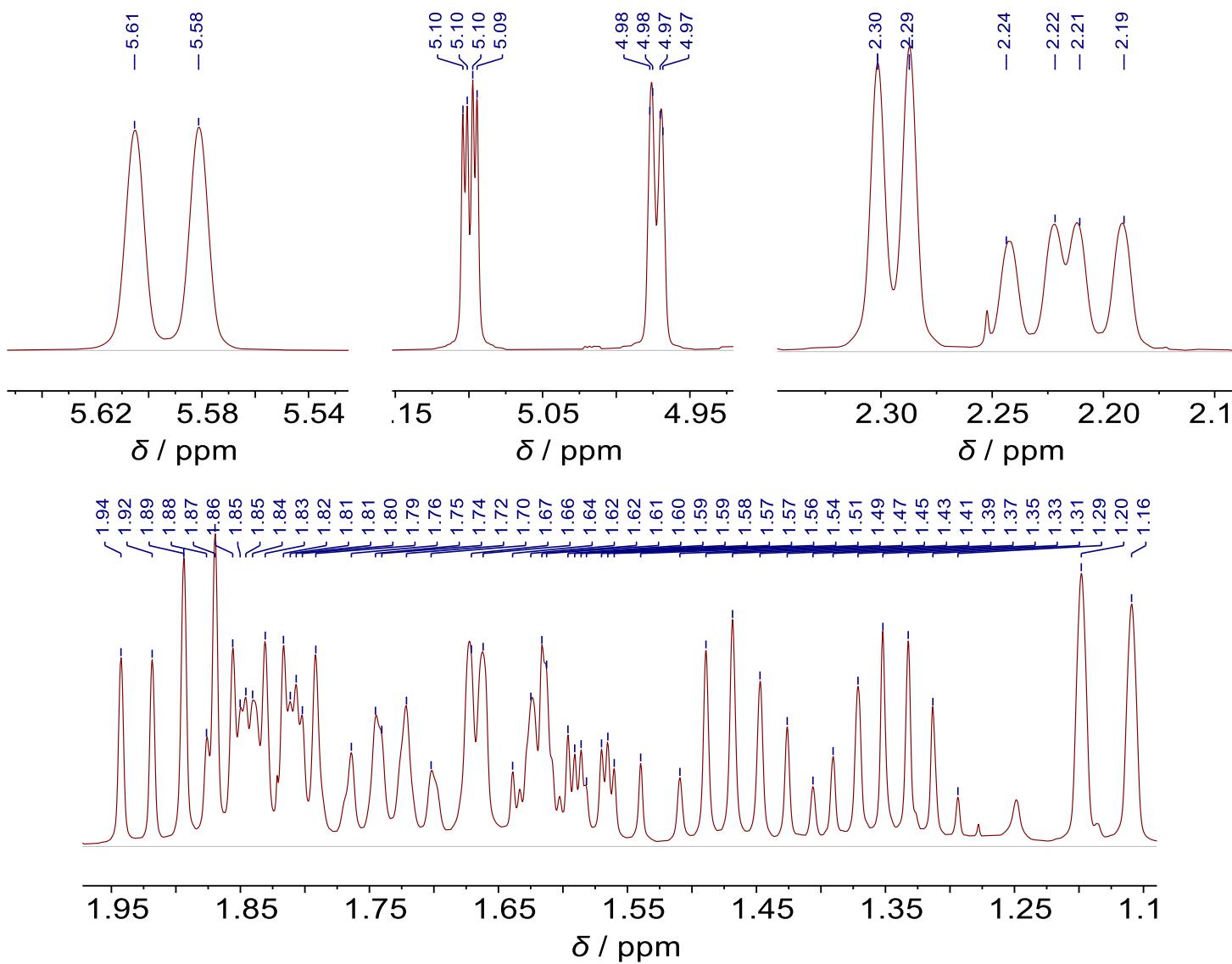
¹³C NMR spectrum of β -cedrenyl acetate (8) in CDCl_3



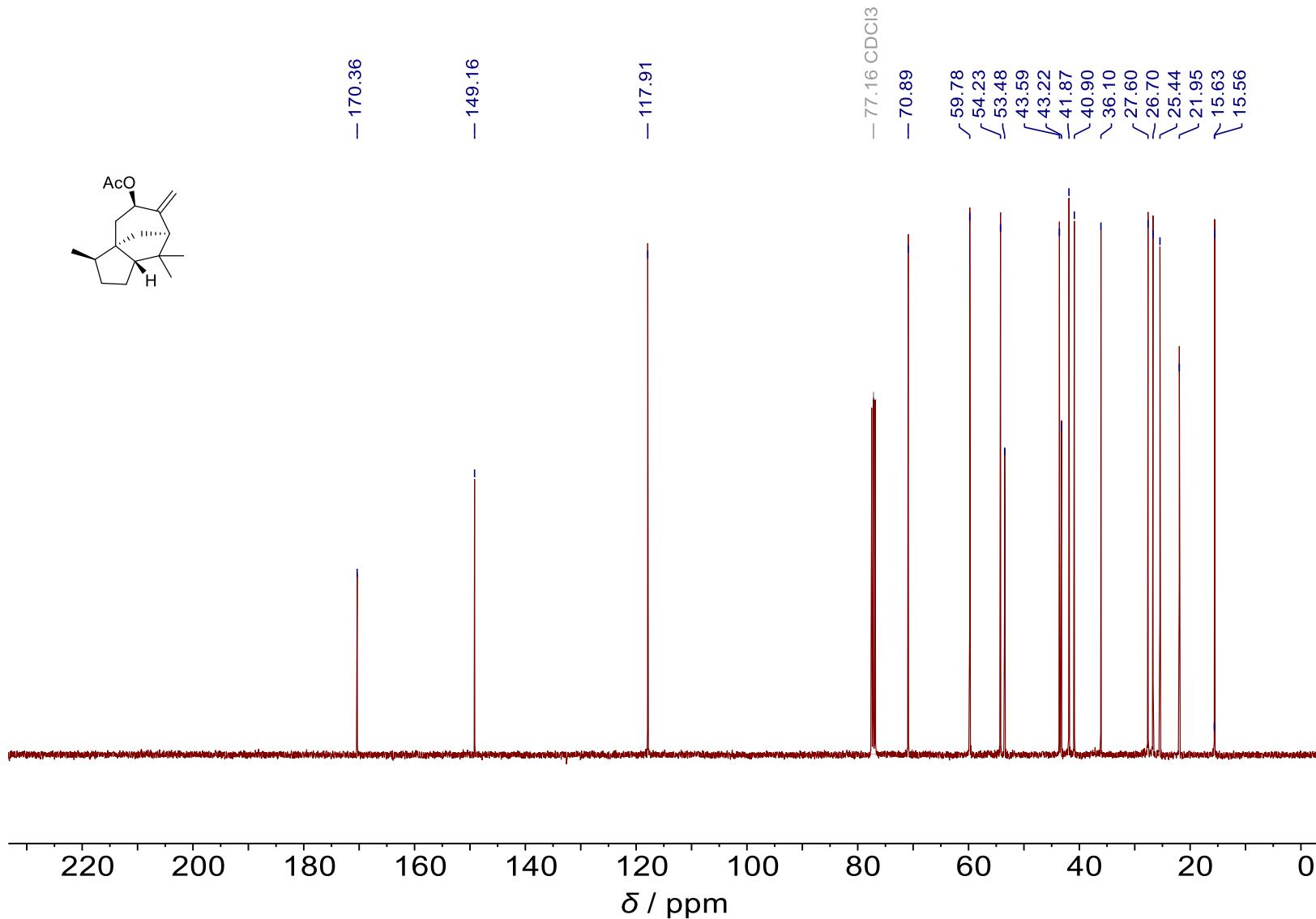
¹H NMR spectrum of *epi*- β -cedrenyl acetate (*epi*-8) in CDCl₃



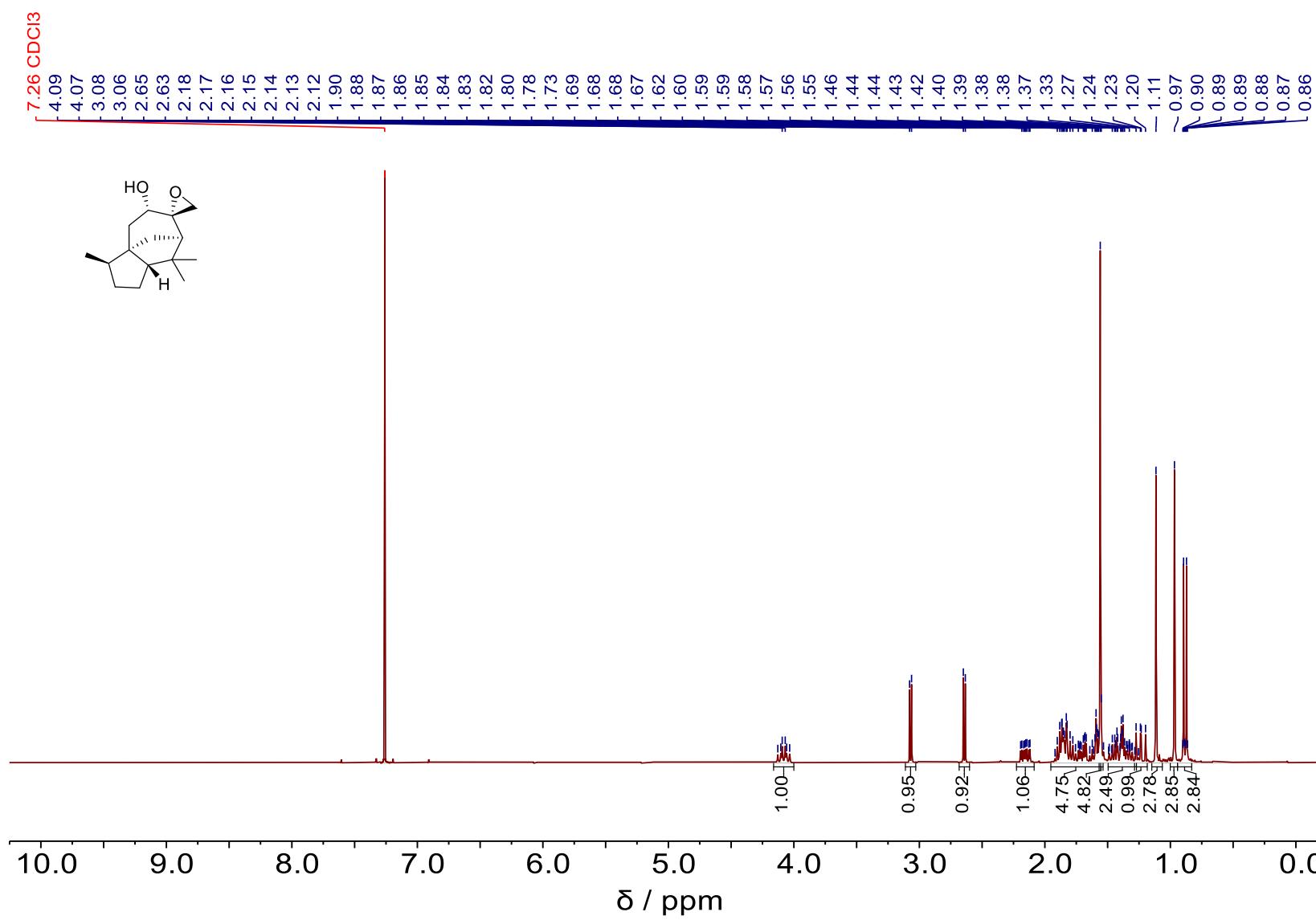
Expansions of the ^1H NMR spectrum of *epi*- β -cedrenyl acetate (*epi*-8) in CDCl_3



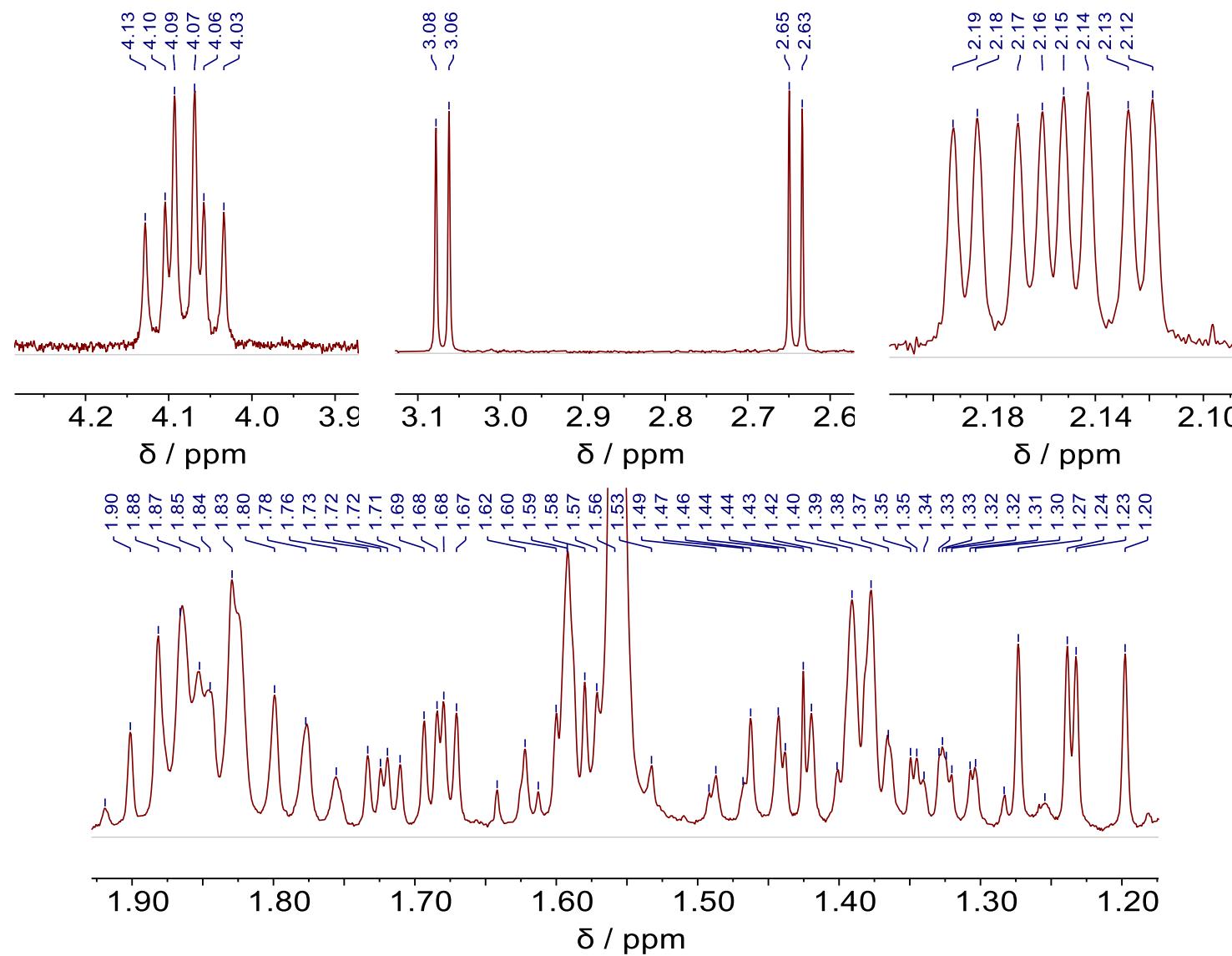
^{13}C NMR spectrum of *epi*- β -cedrenyl acetate (*epi*-8) in CDCl_3



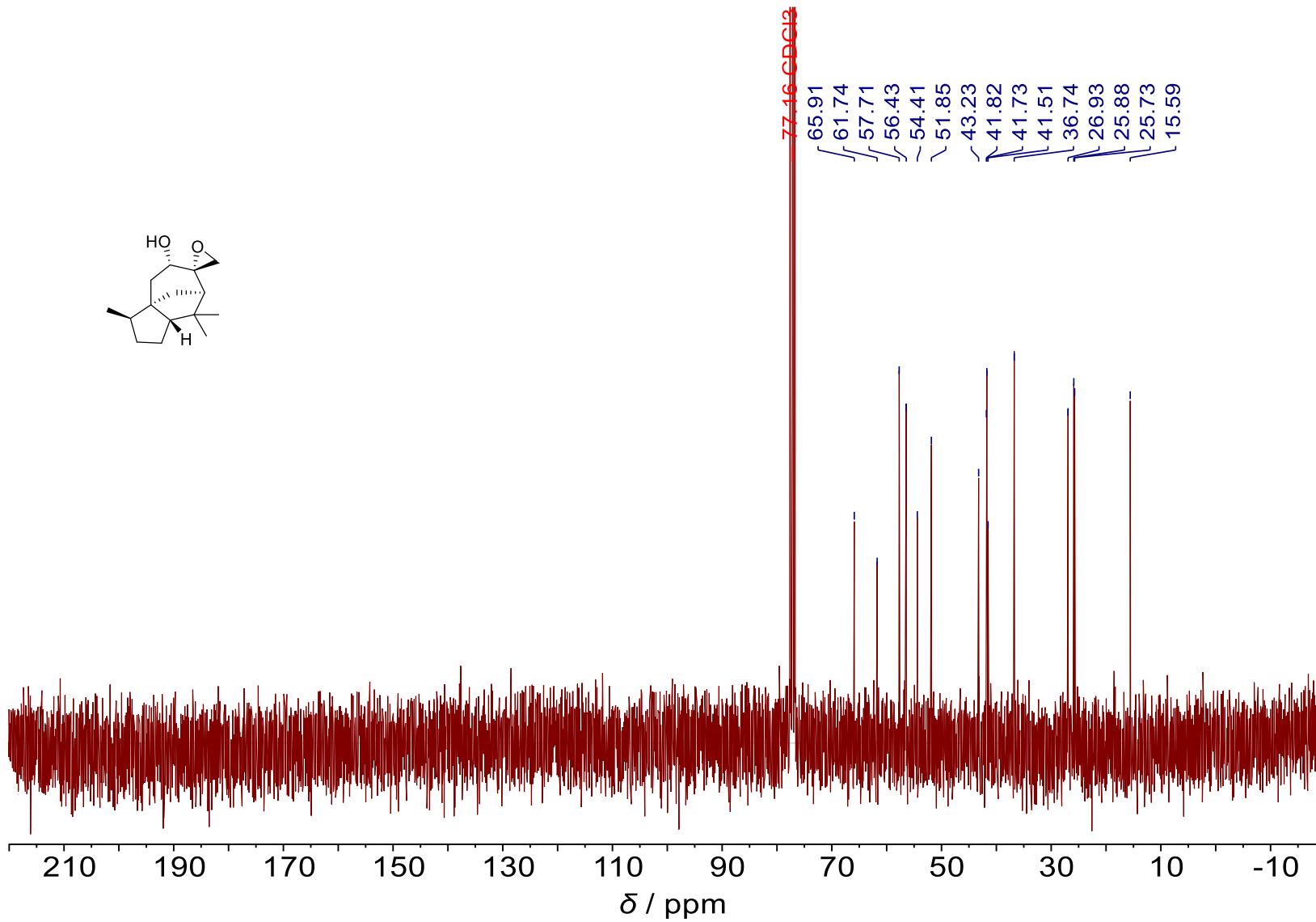
¹H NMR spectrum of β -epoxy cedren-9-ol (*cis*-9) in CDCl₃



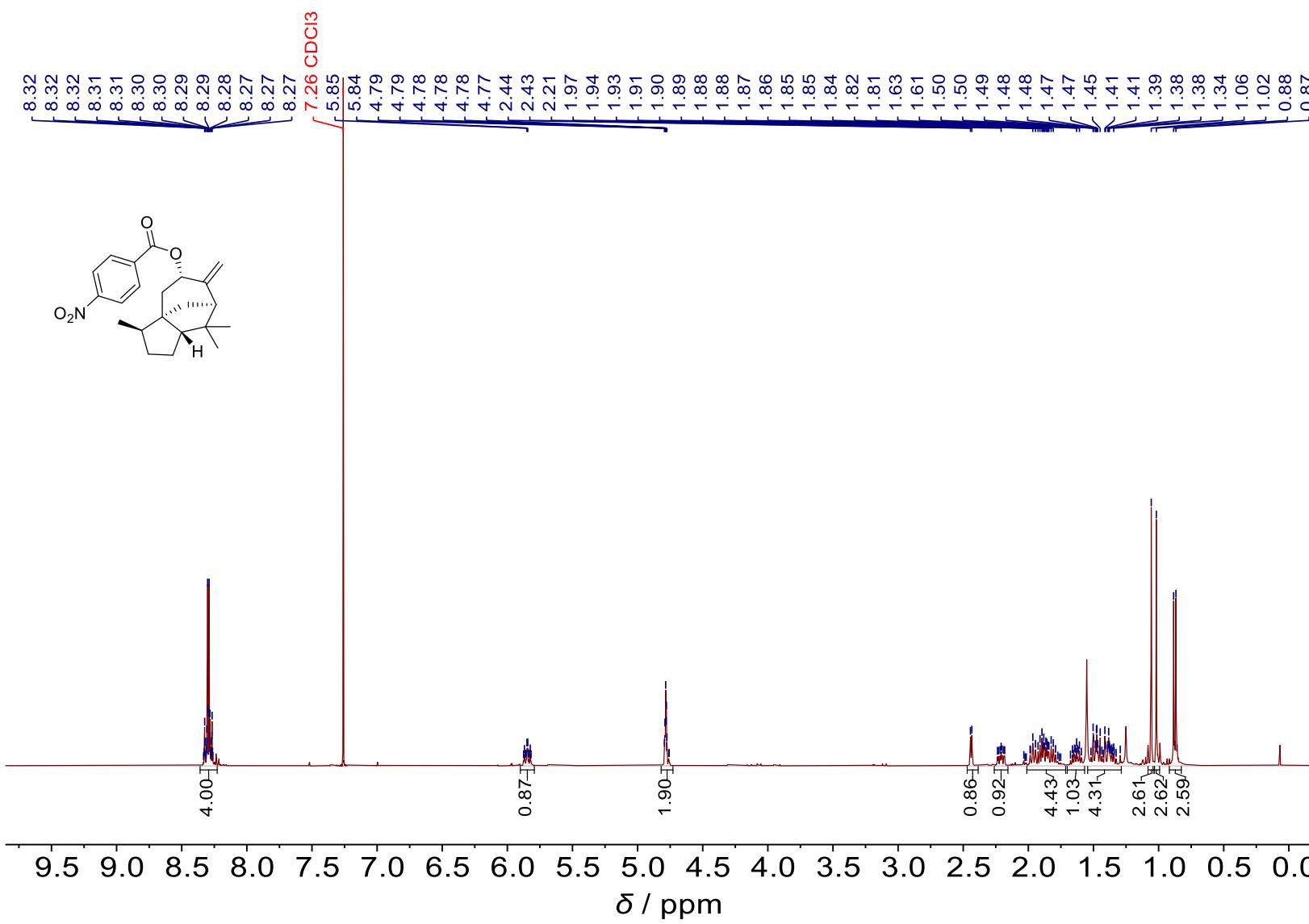
Expansions of the ^1H NMR spectrum of β -epoxy cedren-9-ol (*cis*-9) in CDCl_3



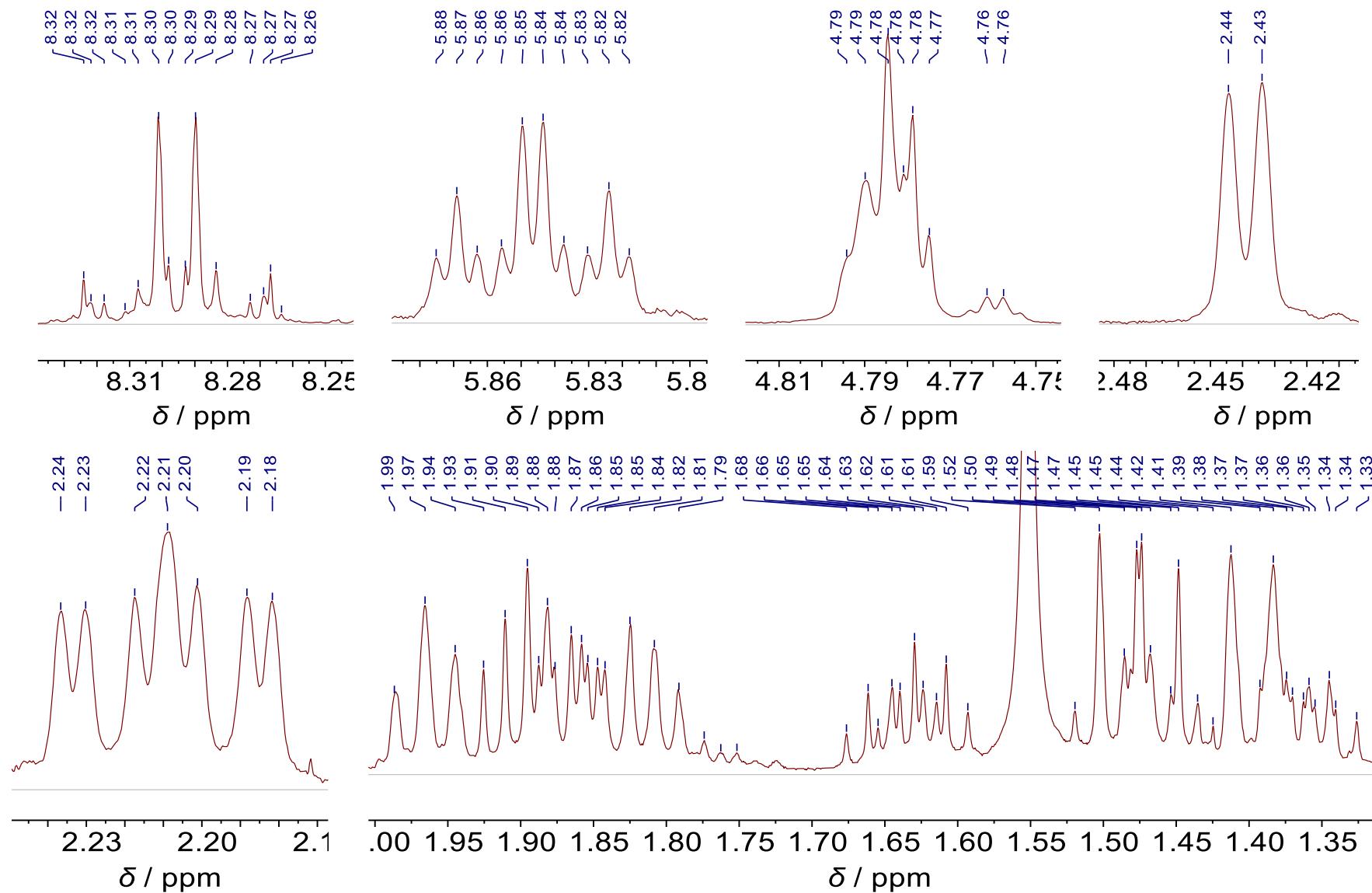
^{13}C NMR spectrum of β -epoxy cedren-9-ol (*cis*-9) in CDCl_3



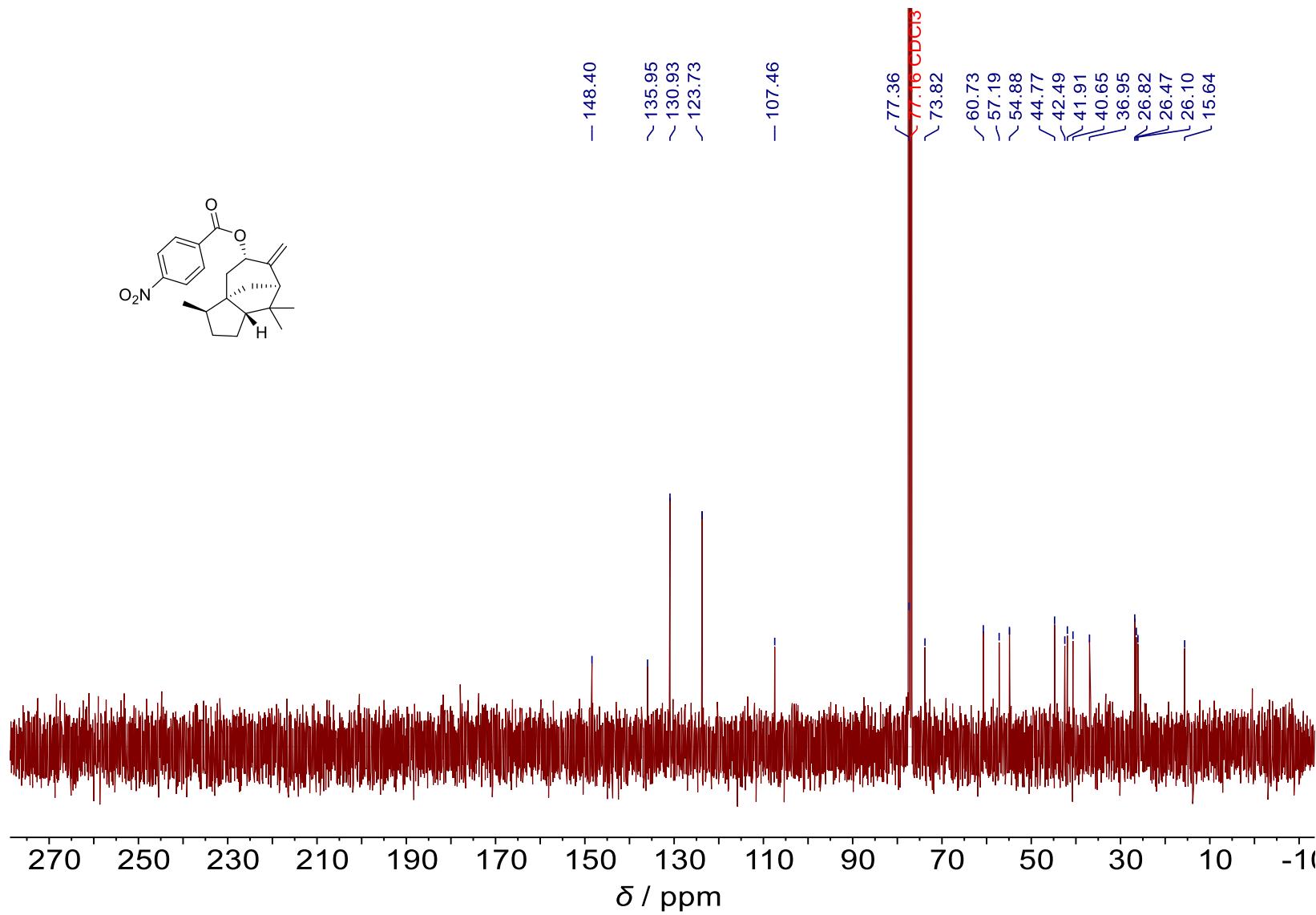
¹H NMR spectrum of β -cedrenyl *p*-nitrobenzoate (**10**) in CDCl₃



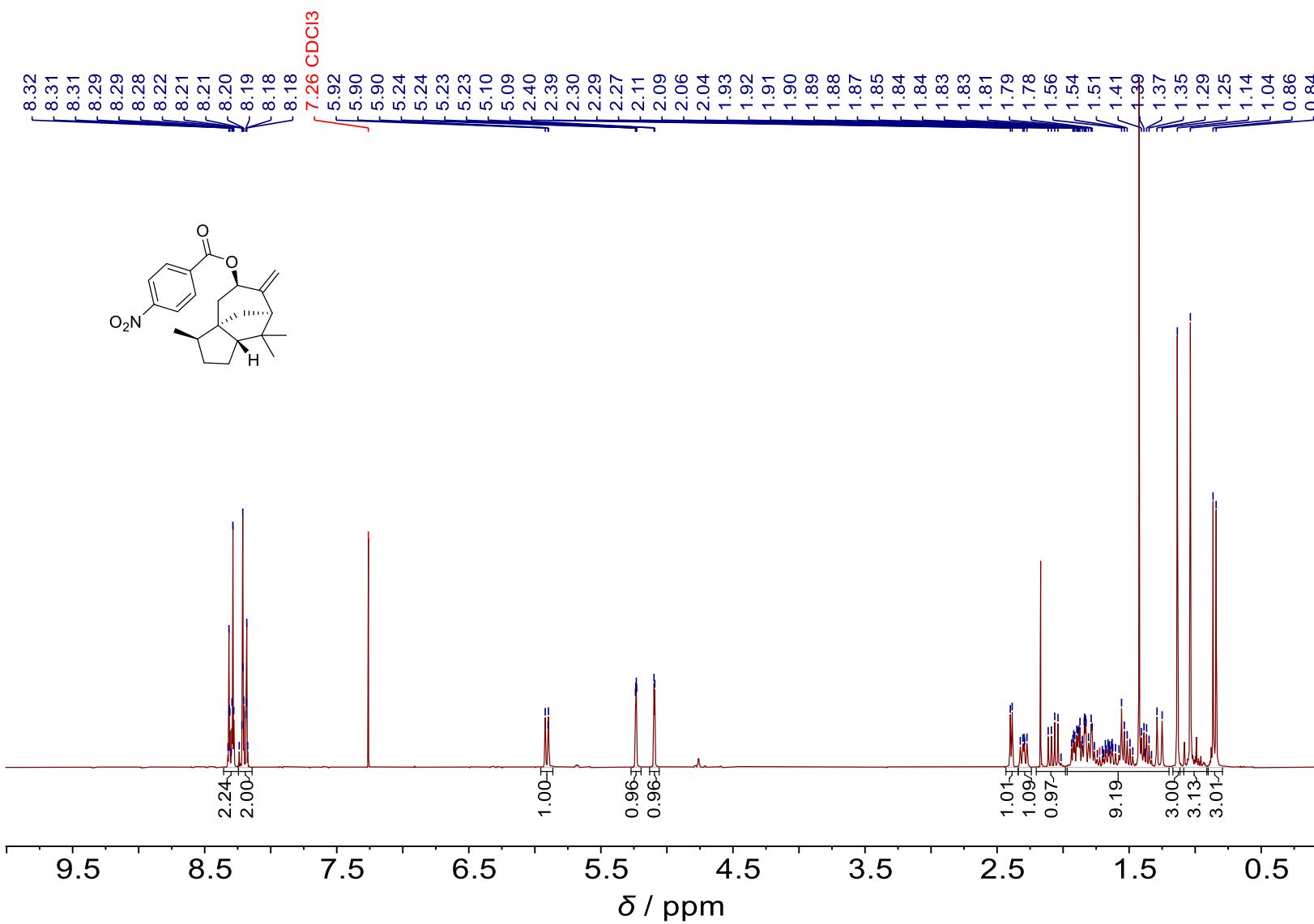
Expansions of the ^1H NMR spectrum of β -cedrenyl *p*-nitrobenzoate (10) in CDCl_3



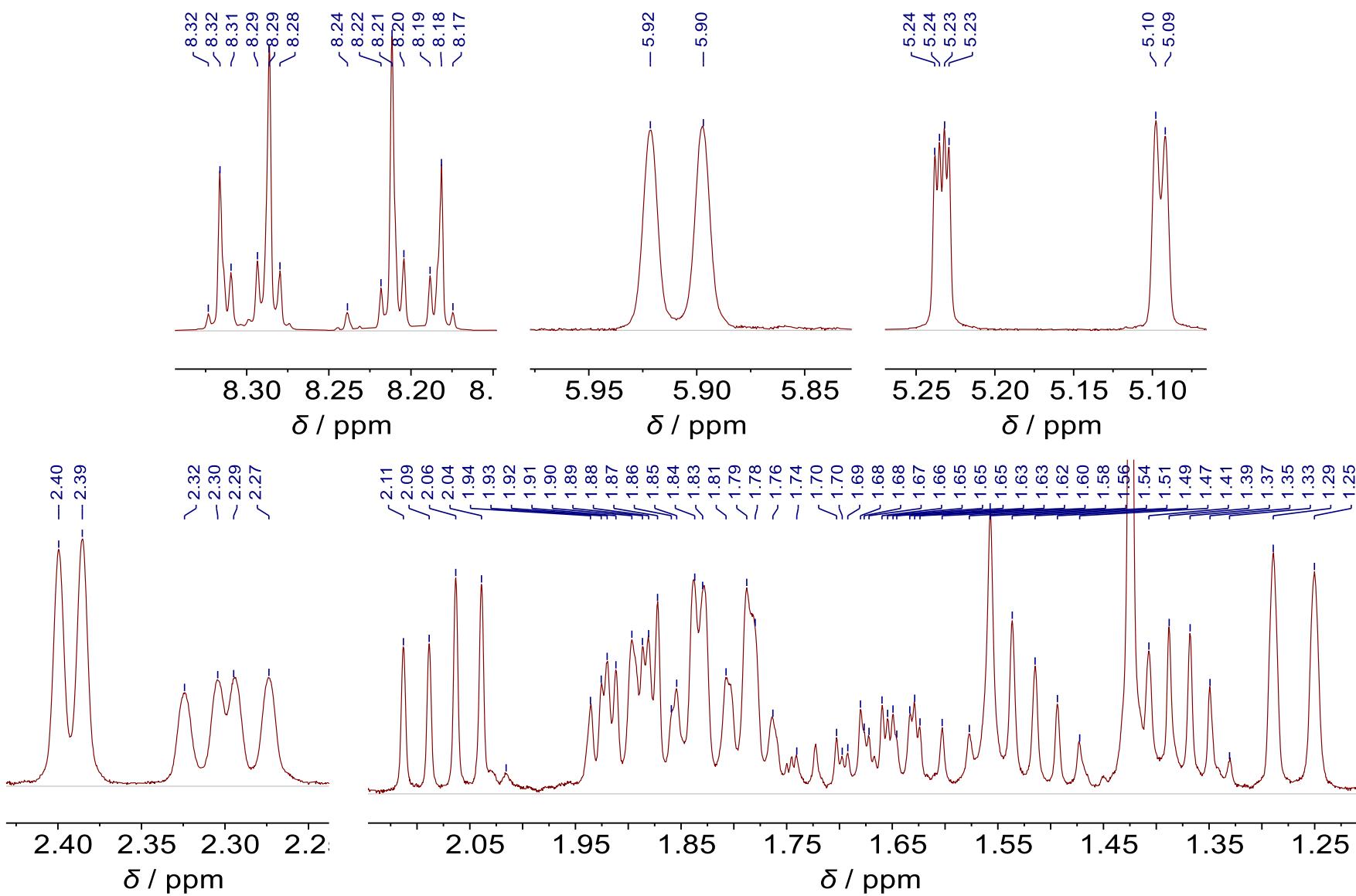
¹³C NMR spectrum of β -cedrenyl *p*-nitrobenzoate (**10**) in CDCl₃



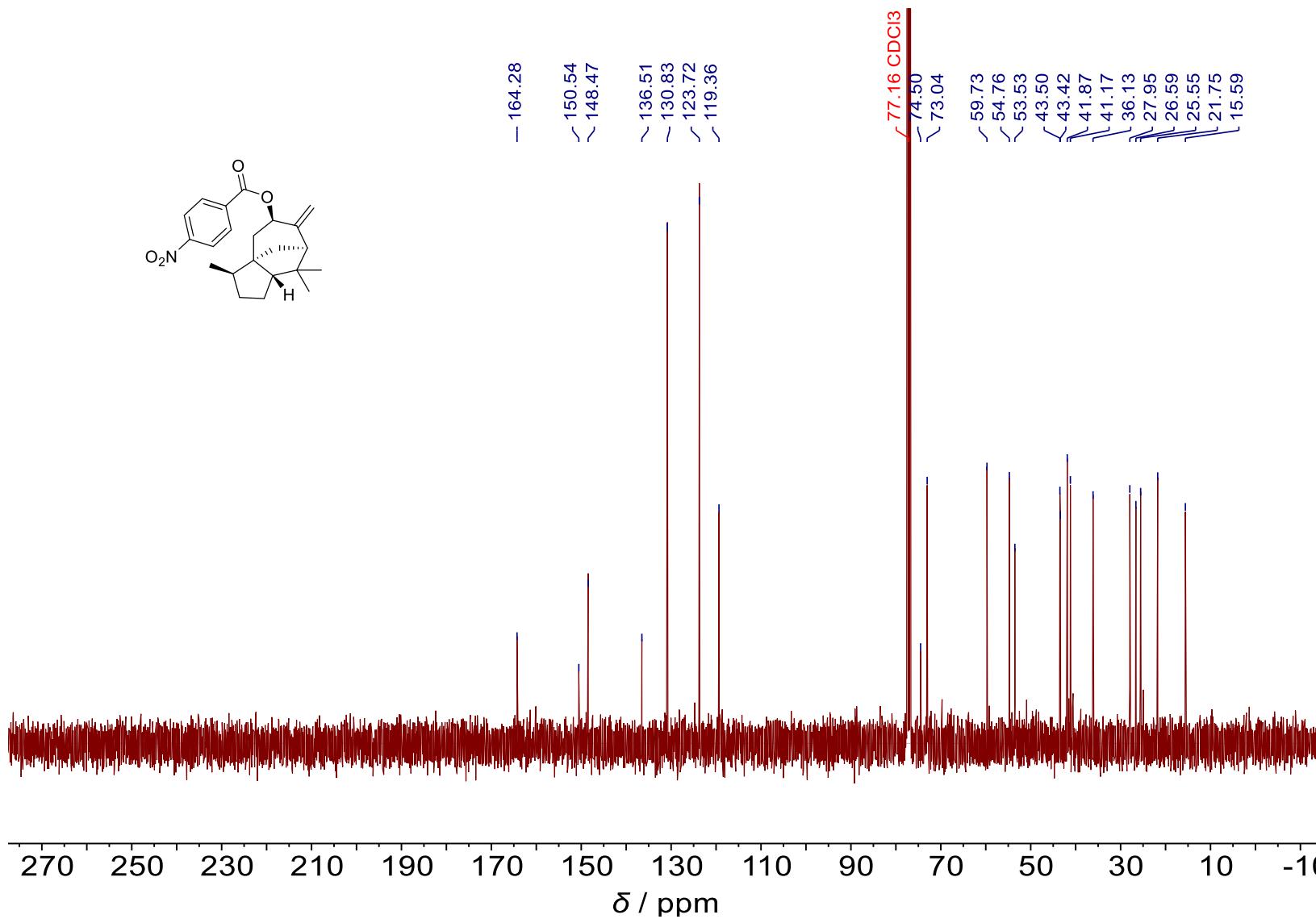
¹H NMR spectrum of *epi*- β -cedrenyl *p*-nitrobenzoate (*epi*-10) in CDCl₃



Expansions of the ^1H NMR spectrum of *epi*- β -cedrenyl *p*-nitrobenzoate (*epi*-10) in CDCl_3



^{13}C NMR spectrum of *epi*- β -cedrenyl *p*-nitrobenzoate (*epi*-10) in CDCl_3



9. X-ray crystal structure analysis of *cis*-3, 6 and *epi*-10

Data were collected on a Burker Kappa APEX II Duo diffractometer. The structures were solved by direct methods (SHELXS-97)⁶ and redefined by full-matrix least-squares procedures on F^2 (SHELXL-2018).⁷ XP (Burker AXS) was used for graphical representations. The crystal structure of *cis*-3 firstly described by Song *et al.* (CCDC 130215, measurement using molybdenum radiation at 293 K)⁸ was re-determined at 150 K using copper radiation in order to prove the absolute configuration (Flack parameter $x = 0.05(5)$). The enantiomers 6 and *epi*-10 have been assigned by reference to unchanging chiral centers at C1, C2, C5, and C7 in the synthetic procedures. CCDC 2023906–2023908 contain the supplementary crystallographic data for this paper. These data were provided free of charge by the joint Cambridge Crystallographic Data Centre and Fachinformationszentrum Karlsruhe Access Structures service www.ccdc.cam.ac.uk/structures.

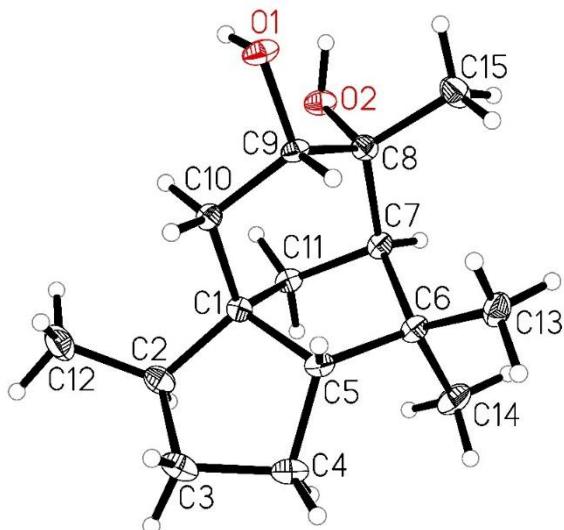


Figure S1. ORTEP representation of *cis*-cedrene-8,9-diol (*cis*-3). Displacement ellipsoids correspond to 30% probability.

Table S8. Crystallographic details for *cis*-cedrene-8,9-diol (*cis*-**3**), β -cedren-9-ol (**6**) and *epi*- β -cedrenyl *p*-nitrobenzoate (*epi*-**10**).

	<i>cis</i> - 3	6	<i>epi</i> - 10
chemical formula	C ₁₅ H ₂₆ O ₂	C ₁₅ H ₂₄ O	C ₂₂ H ₂₇ NO ₄
formula weight	238.36	220.34	369.44
crystal system	trigonal	trigonal	triclinic
<i>a</i> [Å]	14.1629(3)	13.8127(7)	7.2627(4)
<i>b</i> [Å]	14.1629(3)	13.8127(7)	10.8558(7)
<i>c</i> [Å]	5.8443(1)	6.0557(3)	12.9086(8)
α [deg]	90	90	81.804(2)
β [deg]	90	90	78.739(2)
γ [deg]	120	120	84.592(2)
<i>V</i> [Å ³]	1015.24(5)	1000.58(11)	985.64(10)
<i>T</i> [K]	150	150	150
space group	<i>P</i> 3 ₁	<i>P</i> 3 ₁	<i>P</i> 1
<i>Z</i>	3	3	2
μ [mm ⁻¹]	0.583	0.500	0.687
density [g·cm ⁻³]	1.170	1.097	1.245
no. of reflections measured	12030	6561	23596
no. of independent reflections	2350 ($R_{\text{int}} = 0.0240$)	2291 ($R_{\text{int}} = 0.0192$)	6768 ($R_{\text{int}} = 0.0253$)
no. of observed reflections ($I > 2\sigma(I)$)	2332	2264	6679
no. of parameters	167	152	493
R_1 ($I > 2\sigma(I)$)	0.0261	0.0284	0.0307
<i>wR</i> ₂ (all data)	0.0657	0.0754	0.0787
Goodness of fit on F^2	1.104	1.046	1.037
Flack parameter <i>x</i>	0.05(5)	0.05(12)	-0.03(7)

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