Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry.

SUPPORTING INFORMATION

PS-BEMP as a Basic Catalyst for the Phospha-Michael Addition to Electron-poor alkenes

Giacomo Strappaveccia,[a] Luca Bianchi,[a] Simone Ziarelli,[a] Stefano Santoro*,[a] Daniela Lanari,[b] Ferdinando Pizzo[a] and Luigi Vaccaro*[a]

[a] CEMIN - "Centro di Eccellenza Materiali Innovativi Nanostrutturati" Dipartimento di Chimica, Laboratorio di Chimica Organica, Università di Perugia Via Elce di Sotto, 8 I-06123, Perugia, Italia
[b] Dipartimento di Scienze Farmaceutiche, Università di Perugia, Via del Liceo, 06123 Perugia, Italia.

Fax: (+39) 075 5855560

E-mail: luigi.vaccaro@unipg.it; stefano.santoro@unipg.it

CONTENTS

Experimental Section

Unless otherwise stated, all chemicals were purchased and used without any further purification. GLC analyses were performed by using a Hewlett-Packard HP 5890A equipped with a capillary column DB-35MS (30 m, 0.53 mm), a FID detector and helium as gas carrier. GC-EIMS analyses were carried out by using a Hewlett-Packard HP 6890N Network GC system/5975 Mass Selective Detector equipped with an electron impact ionizer at 70 eV.

All ^{1}H NMR, ^{13}C NMR and ^{31}P NMR spectra were recorded at 400 MHz, 100.6 MHz and 161.9 MHz, respectively, using a Bruker DRX-ADVANCE 400 MHz spectrometer. The deuterated solvent used was $CDCl_3$, and TMS was employed as internal standard. Chemical shifts are reported in ppm and coupling constants in hertz. Elemental analyses were realized by using a FISONS instrument EA 1108 CHN.

Compounds **3a**,[1] **3b-d**,[2] **7a**,[3] **7b**,[4] **7e**,[5] **7g**,[6] **12a**,[7] **12b**,[8] **12c**,[9] **12d**,[9] **13a**[10] and **13b**[1] are known, while compounds **3e**-**f**, **7d**, **7f**, **13c** and **13d** are unknown.

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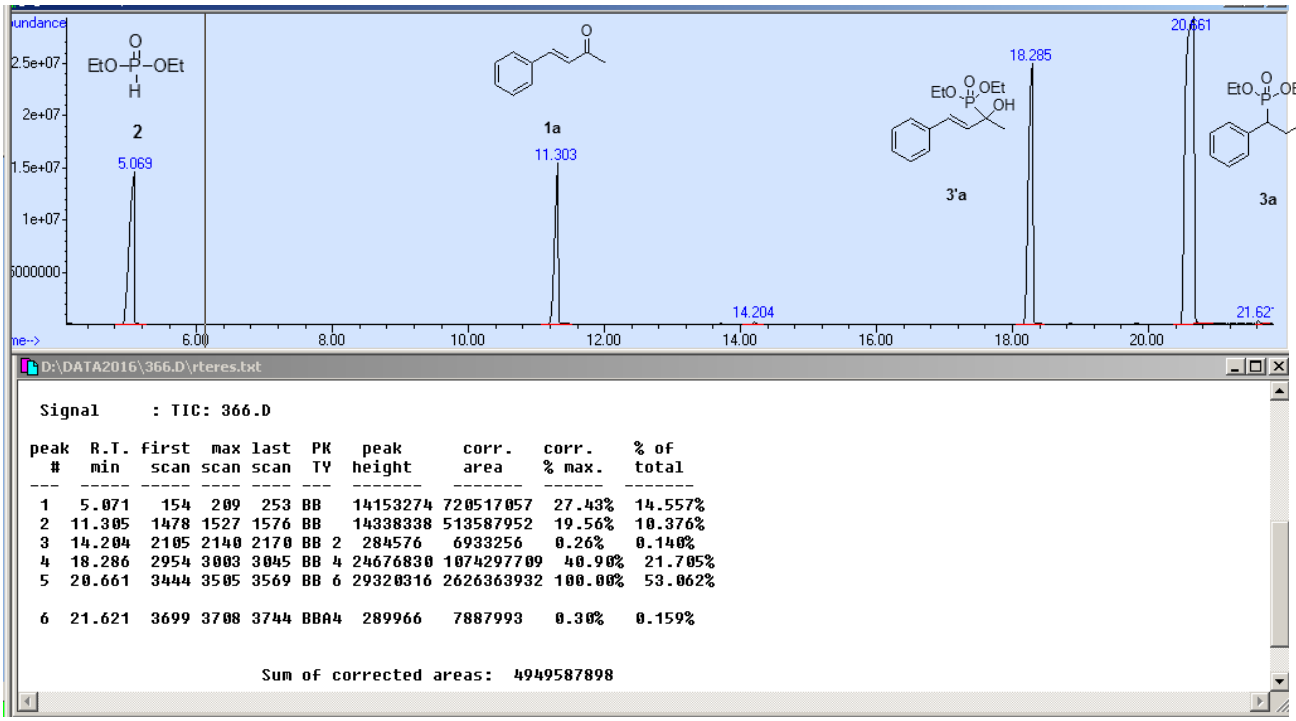
GLC analysis of the reaction mixture of 1 with 2 giving a 70:30 mixture of compounds 3a and 3a' (Table 1 entry 11).



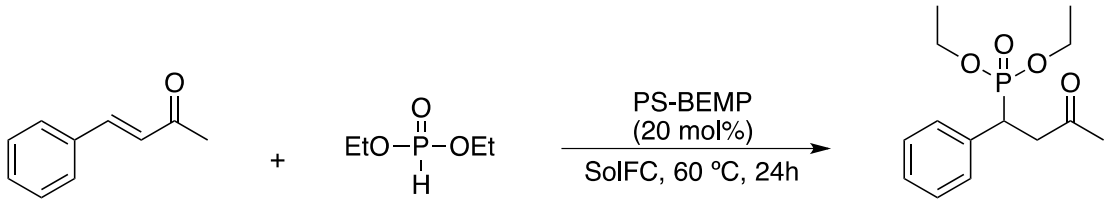
D:\DATA2016\366.D\rteres.txt

Signal : TIC: 366.D

peak #	R.T. min	first scan	max scan	last scan	PK TY	peak height	corr. area	corr. % max.	% of total
1	5.071	154	209	253	BB	14153274	720517057	27.43%	14.557%
2	11.305	1478	1527	1576	BB	14338338	513587952	19.56%	10.376%
3	14.204	2105	2140	2170	BB 2	284576	6933256	0.26%	0.140%
4	18.286	2954	3003	3045	BB 4	24676830	1074297709	40.90%	21.705%
5	20.661	3444	3505	3569	BB 6	29320316	2626363932	100.00%	53.062%
6	21.621	3699	3708	3744	BBA4	289966	7887993	0.30%	0.159%

Sum of corrected areas: 4949587898

Chem. Name	Diethyl (3-oxo-1-phenylbutyl)phosphonate (3a)
Lit. Ref.	*J. Org. Chem.* **1997**, *62*, 2414-2422



1a **2** **3a**

M.W.: 284

METHOD:
In a screw capped vial equipped with a magnetic stirrer PS-BEMP (43 mg, 0.1 mmol, 2.13 mmol/g), (E)-4-phenylbut-3-en-2-one (**1a**) (75 mg, 0.5 mmol), and diethyl phosphite (**2**) (70 mg, 0.065 mL, 0.5 mmol) were consecutively added and the resulting mixture was left under stirring at 60 °C. After 24 hours EtOAc (1 mL) was added and the catalyst was filtered off and the solvent was removed under vacuum. The obtained oil was purified by flash column chromatography on silica gel (petroleum ether/EtOAc 95/5). **3a** was obtained as an oil (114 mg, 80% yield).

Mol Formula	$C_{14}H_{21}O_4P$	m.p.	Oil

Elemental Analysis: Calc.: C: 59.15; H: 7.45; found C: 60.35; H: 7.07

^{1}H NMR 400 MHz $CDCl_3$

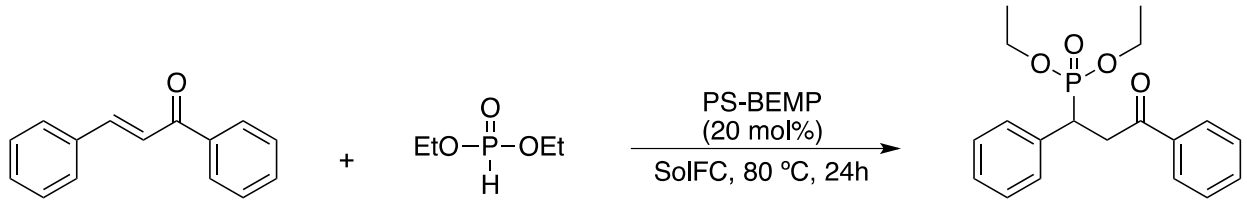
δ value	No. H	Mult.	j value/Hz
1.05	3	*t*	6.9
1.25	3	*t*	6.9
2.05	3	*s*	
3.08 – 3.13	2	*m*	
3.67 – 3.74	2	*m*	
3.83 – 3.88	1	*m*	
4.00 – 4.03	2	*m*	
7.19 – 7.21	1	*m*	
7.25 – 7.28	2	*m*	
7.31 – 7.33	2	*m*	

^{13}C NMR (100.6 MHz, $CDCl_3$) δ : 16.08 (*d*, $j_{P\text{-}C}$= 5.8 Hz); 16.26 (*d*, $j_{P\text{-}C}$= 5.9 Hz); 30.28; 38.85 (*d*, $j_{P\text{-}C}$= 140 Hz); 43.68; 61.92 (*d*, $j_{P\text{-}C}$= 7.1 Hz); 62.76 (*d*, $j_{P\text{-}C}$= 6.8 Hz);127.22; 128.44; 129.04 (*d*, $j_{P\text{-}C}$= 6.4 Hz); 135.71 (*d*, $j_{P\text{-}C}$= 6.8 Hz), 204.78 (*d*, $j_{P\text{-}C}$= 14.4 Hz)

^{31}P NMR (161.9 MHz, $CDCl_3$) δ : 32.11

GC-EIMS (m/z, %): 284 (M^+, 12); 242 (15); 241 (100); 213 (23); 185 (53); 138 (16); 129 (12); 111 (20); 104 (29); 103 (23); 81 (13); 78 (12); 77 (14); 43 (60)

Chem. Name	Diethyl (3-oxo-1,3-diphenylpropyl)phosphonate (3b)
Lit. Ref.	*Heteroat. Chem.* **2013**, *24*, 345-354



1b + **2** → **3b**

M.W.: 346

METHOD:
In a screw capped vial equipped with a magnetic stirrer PS-BEMP (43 mg, 0.1 mmol, 2.13 mmol/g), (E)-chalcone (**1b**) (106 mg, 0.5 mmol), and diethyl phosphite (**2**) (70 mg, 0.065 mL, 0.5 mmol) were consecutively added and the resulting mixture was left under stirring at 80 °C. After 24 hours EtOAc (1 mL) was added and the catalyst was filtered off and the solvent was removed under vacuum. The obtained oil was purified by flash column chromatography on silica gel (petroleum ether/EtOAc 95/5). **3b** was obtained as an oil (142 mg, 82% yield).

Mol Formula	$C_{19}H_{23}O_4P$	m.p.	Oil

Elemental Analysis: Calc.: C: 65.89; H: 6.69; found C: 65.78; H: 6.80

^{1}H NMR 400 MHz $CDCl_3$

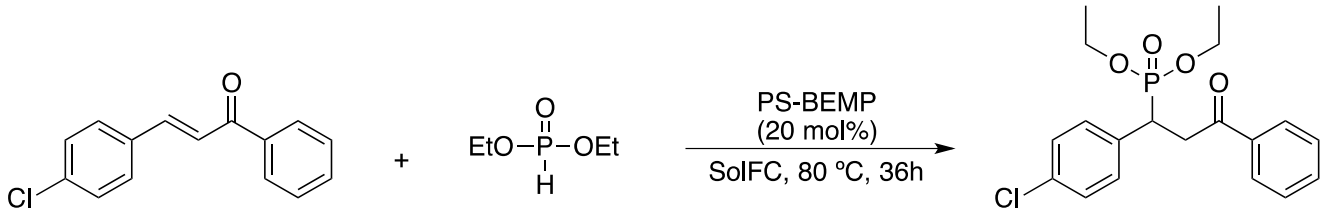
δ value	No. H	Mult.	j value/Hz
1.06	3	*t*	7.0
1.26	3	*t*	7.1
3.61 – 3.80	3	*m*	
3.80 – 4.10	4	*m*	
7.18 – 7.21	1	*m*	
7.26 – 7.29	2	*m*	
7.40 – 7.44	4	*m*	
7.51 – 7.54	1	*m*	
7.92	2	*d*	8.0

^{13}C NMR (100.6 MHz, $CDCl_3$) δ : 16.10 (*d*, $j_{P\text{-}C}$= 5.5 Hz); 16.29 (*d*, $j_{P\text{-}C}$= 5.9 Hz); 38.9 (*d*, $j_{P\text{-}C}$= 140 Hz); 39.01; 61.92 (*d*, $j_{P\text{-}C}$= 7.1 Hz); 62.90 (*d*, $j_{P\text{-}C}$= 6.7 Hz); 127.17; 128.00; 128.41; 128.54; 129.18 (*d*, $j_{P\text{-}C}$= 6.5 Hz), 133.22; 135.87 (*d*, $j_{P\text{-}C}$= 6.7 Hz); 136.46; 196.31 (*d*, $j_{P\text{-}C}$= 14.9 Hz)

^{31}P NMR (161.9 MHz, $CDCl_3$) δ : 32.64

GC-EIMS (m/z, %): 346 (M^+, 8); 242 (13); 241 (99); 213 (15); 185 (36); 105 (100); 104 (30); 103 (21); 81 (14); 78 (15); 77(76); 51 (14)

Chem. Name	Diethyl (1-(4-chlorophenyl)-3-oxo-3-phenylpropyl)phosphonate (3c)
Lit. Ref.	*Heteroat. Chem.* **2013**, *24*, 345-354



1c + **2** → **3c**

M.W.: 380

METHOD:
In a screw capped vial equipped with a magnetic stirrer PS-BEMP (43 mg, 0.1 mmol, 2.13 mmol/g), (E)-3-(4-chlorophenyl)-1-phenylprop-2-en-1-one (**1c**) (121 mg, 0.5 mmol), and diethyl phosphite (**2**) (70 mg, 0.065 mL, 0.5 mmol) were consecutively added and the resulting mixture was left under stirring at 80 °C. After 36 hours EtOAc (1 mL) was added and the catalyst was filtered off and the solvent was removed under vacuum. The obtained oil was purified by flash column chromatography on silica gel (petroleum ether/EtOAc 95/5). **3c** was obtained as an oil (148 mg, 78% yield).

Mol Formula	$C_{19}H_{22}ClO_4P$	m.p.	Oil

Elemental Analysis: Calc.: C: 59.93; H: 5.82; found C: 59.88; H: 5.87

^{1}H NMR 400 MHz $CDCl_3$

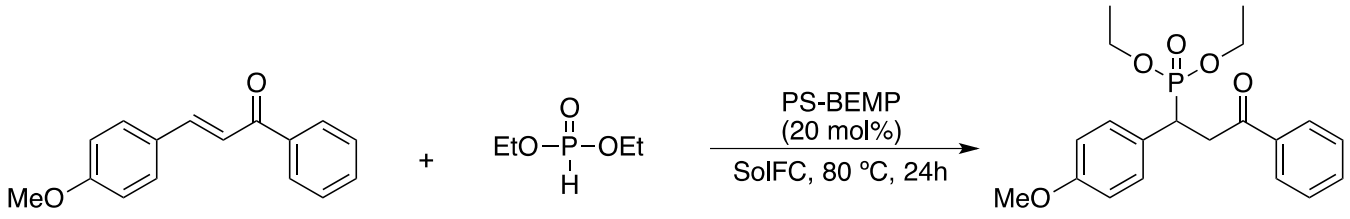
δ value	No. H	Mult.	j value/Hz
1.11	3	*t*	7.0
1.27	3	*t*	7.0
3.59 – 3.82	3	*m*	
3.82 – 4.00	2	*m*	
4.00 – 4.15	2	*m*	
7.24 – 7.26	2	*m*	
7.35 – 7.44	4	*m*	
7.52 – 7.56	1	*m*	
7.91	2	*d*	7.6

^{13}C NMR (100.6 MHz, $CDCl_3$) δ : 16.18 (*d*, $j_{P\text{-}C}$= 5.6 Hz); 16.31 (*d*, $j_{P\text{-}C}$= 5.8 Hz); 38.35 (*d*, $j_{P\text{-}C}$= 141 Hz); 38.92; 62.08 (*d*, $j_{P\text{-}C}$= 7.0 Hz); 62.96 (*d*, $j_{P\text{-}C}$= 6.8 Hz); 127.99; 128.60; 130.49 (*d*, $j_{P\text{-}C}$= 6.5 Hz); 133.03; 133.38; 134.54 (*d*, $j_{P\text{-}C}$= 6.9 Hz); 136.29; 196.06 (*d*, $j_{P\text{-}C}$= 15.4 Hz)

^{31}P NMR (161.9 MHz, $CDCl_3$) δ : 31.96

GC-EIMS (m/z, %): 382 (M^++2 ,2); 380 (M^+, 8); 277 (27); 275 (80); 219 (33); 138 (24); 105 (100); 77 (86)

Chem. Name	Diethyl (1-(4-methoxyphenyl)-3-oxo-3-phenylpropyl)phosphonate (3d)
Lit. Ref.	*Heteroat. Chem.* **2013**, *24*, 345-354



1d **2** **3d**

M.W.: 376

METHOD:
In a screw capped vial equipped with a magnetic stirrer PS-BEMP (43 mg, 0.1 mmol, 2.13 mmol/g), (E)-3-(4-methoxyphenyl)-1-phenylprop-2-en-1-one (**1d**) (119 mg, 0.5 mmol), and diethyl phosphite (**2**) (70 mg, 0.065 mL, 0.5 mmol) were consecutively added and the resulting mixture was left under stirring at 80 °C. After 24 hours EtOAc (1 mL) was added and the catalyst was filtered off and the solvent was removed under vacuum. The obtained oil was purified by flash column chromatography on silica gel (petroleum ether/EtOAc 95/5). **3d** was obtained as an oil (152 mg, 81% yield).

Mol Formula	$C_{20}H_{25}O_5P$	m.p.	Oil

Elemental Analysis: Calc.: C: 63.82; H: 6.69; found C: 63.92; H: 6.60

^{1}H NMR 400 MHz $CDCl_3$

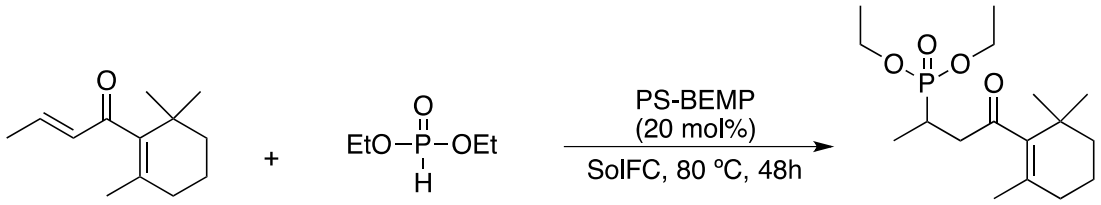
δ value	No. H	Mult.	j value/Hz
1.10	3	*t*	7.0
1.28	3	*t*	7.0
3.58 – 3.76	6	*m*	
3.86 – 3.95	2	*m*	
4.03 – 4.11	2	*m*	
6.82	2	*d*	8.4
7.34 – 7.36	2	*m*	
7.42 – 7.46	2	*m*	
7.53 – 7.56	1	*m*	
7.93	2	*d*	7.6

^{13}C NMR (100.6 MHz, $CDCl_3$) δ : 16.18 (*d*, $j_{P\text{-}C}$= 5.7 Hz); 16.30 (*d*, $j_{P\text{-}C}$= 5.9 Hz); 37.98 (*d*, $j_{P\text{-}C}$= 141 Hz); 39.10; 55.07; 61.86 (*d*, $j_{P\text{-}C}$= 7.2 Hz); 62.88 (*d*, $j_{P\text{-}C}$= 6.9 Hz); 113.84; 127.63 (*d*, $j_{P\text{-}C}$= 6.9 Hz); 128.00; 128.52; 130.16 (*d*, $j_{P\text{-}C}$= 6.5 Hz); 133.20; 136.46; 158.63; 196.44 (*d*, $j_{P\text{-}C}$= 15.3 Hz)

^{31}P NMR (161.9 MHz, $CDCl_3$) δ : 32.93

GC-EIMS (m/z, %): 376 (M^+, 16); 271 (100); 243 (18); 215 (37); 134 (18); 133 (14); 105 (83); 77 (45)

Chem. Name	Diethyl (4-oxo-4-(2,6,6-trimethylcyclohex-1-en-1-yl)butan-2-yl)phosphonate (3e)
Lit. Ref.	/



1e + **2** → **3e**

M.W.: 330

METHOD:
In a screw capped vial equipped with a magnetic stirrer PS-BEMP (43 mg, 0.1 mmol, 2.13 mmol/g), (E)-1-(2,6,6-trimethylcyclohex-1-en-1-yl)but-2-en-1-one (**1e**) (106 mg, 0.114 mL, 0.5 mmol), and diethyl phosphite (**2**) (70 mg, 0.065 mL, 0.5 mmol) were consecutively added and the resulting mixture was left under stirring at 80 °C. After 48 hours EtOAc (1 mL) was added and the catalyst was filtered off and the solvent was removed under vacuum. The obtained oil was purified by flash column chromatography on silica gel (petroleum ether/EtOAc 95/5). **3e** was obtained as an oil (132 mg, 80% yield).

Mol Formula	$C_{17}H_{31}O_4P$	m.p.	Oil

Elemental Analysis: Calc.: C: 61.80; H: 9.46; found C: 61.81; H: 9.40

^{1}H NMR 400 MHz $CDCl_3$

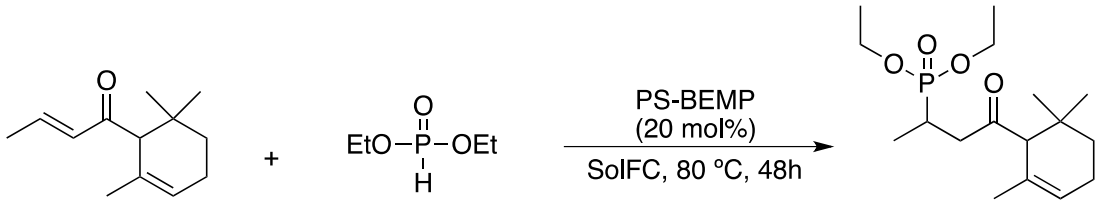
δ value	No. H	Mult.	j value/Hz
0.77	3	*s*	
0.79	3	*s*	
0.90-0.96	3	*m*	
1.04	6	*t*	7.0
1.16-1.17	2	*m*	
1.28	3	*s*	
1.37-1.38	2	*m*	
1.66-1.69	2	*m*	
2.26-2.40	2	*m*	
2.66-2.74	1	*m*	
3.82-3.85	4	*m*	

^{13}C NMR (100.6 MHz, $CDCl_3$) δ : 13.49 (*d*, $j_{P\text{-}C}$= 5.1 Hz); 16.13 (*d*, $j_{P\text{-}C}$= 5.6 Hz); 18.46; 20.34; 24.96 (*d*, $j_{P\text{-}C}$= 144 Hz); 28.33; 30.85; 32.94; 38.52; 45.67; 61.27 (*d*, $j_{P\text{-}C}$= 6.7 Hz); 61.40 (*d*, $j_{P\text{-}C}$= 6.7 Hz); 129.22; 142.17; 207.47 (*d*, $j_{P\text{-}C}$= 15.9 Hz)

^{31}P NMR (161.9 MHz, $CDCl_3$) δ : 38.08

GC-EIMS (m/z, %): 330 (M^+; 15); 207 (63); 180 (15); 179 (50); 151 (100); 123 (17); 81 (24); 69 (53)

Chem. Name	Diethyl (4-oxo-4-(2,6,6-trimethylcyclohex-2-en-1-yl)butan-2-yl)phosphonate (3f)
Lit. Ref.	/



1f **2** **3f**

M.W.: 330

METHOD:
In a screw capped vial equipped with a magnetic stirrer PS-BEMP (43 mg, 0.1 mmol, 2.13 mmol/g), (E)-1-(2,6,6-trimethylcyclohex-2-en-1-yl)but-2-en-1-one (**1f**) (106 mg, 0.114 mL, 0.5 mmol), and diethyl phosphite (**2**) (70 mg, 0.065 mL, 0.5 mmol) were consecutively added and the resulting mixture was left under stirring at 80 °C. After 48 hours EtOAc (1 mL) was added and the catalyst was filtered off and the solvent was removed under vacuum. The obtained oil was purified by flash column chromatography on silica gel (petroleum ether/EtOAc 95/5). **3f** was obtained as an oil (ca. 1:1 diasteroisomeric mixture) (135 mg, 82% yield).

Mol Formula	$C_{17}H_{31}O_4P$	m.p.	Oil

Elemental Analysis: Calc.: C: 61.80; H: 9.46; found C: 61.90; H: 9.50

^{1}H NMR 400 MHz $CDCl_3$

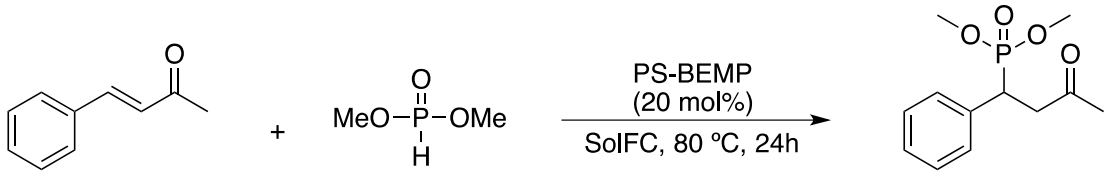
δ value	No. H	Mult.	j value/Hz
0.78	3	*s*	
0.81	3	*s*	
1.00 – 1.08	4	*m*	
1.20	6	*t*	7.0
1.46 – 1.48	3	*m*	
1.59 – 1.68	1	*m*	
1.86 – 2.03	2	*m*	
2.38 – 2.93	4	*m*	
3.98 – 4.02	4	*m*	
5.47	1	*s*	

^{13}C NMR (100.6 MHz, $CDCl_3$) δ : 13.55 (*d, $j_{P\text{-}C}$*= 7.7 Hz); 16.24; 22.44; 23.20; 25.27 (*d, $j_{P\text{-}C}$*= 145.9 Hz); 27.62; 30.49; 32.28; 46.00; 61.68 (*d, $j_{P\text{-}C}$*= 6.9 Hz); 61.81 (*d, $j_{P\text{-}C}$*= 6.5 Hz); 63.00; 123.60; 129.61; 210.10 (*d, $j_{P\text{-}C}$*= 33.00 Hz)

^{31}P NMR (161.9 MHz, $CDCl_3$) δ : 38.08

GC-EIMS (m/z, %): 330 (M^+, 22); 207 (27); 179 (34); 166 (48); 165 (53); 151 (100); 138 (21); 123 (62); 109 (28); 93 (24); 91 (29); 81 (79); 79 (27); 69 (53); 67 (18); 65 (19); 55 (23); 43 (25)

Chem. Name	Dimethyl (3-oxo-1-phenylbutyl)phosphonate (7a)
Lit. Ref.	*Liebigs Ann. Chem.* **1991**, *3*, 229-236



1a + **4** → **7a**

M.W.: 256

METHOD:
In a screw capped vial equipped with a magnetic stirrer PS-BEMP (43 mg, 0.1 mmol, 2.13 mmol/g), (E)-4-phenylbut-3-en-2-one (**1a**) (75 mg, 0.5 mmol), and dimethyl phosphite (**4**) (61 mg, 0.051 mL, 0.5 mmol) were consecutively added and the resulting mixture was left under stirring at 80 °C. After 24 hours EtOAc (1 mL) was added and the catalyst was filtered off and the solvent was removed under vacuum. The obtained oil was purified by flash column chromatography on silica gel (petroleum ether/EtOAc 95/5). **7a** was obtained as an oil (105 mg, 82% yield).

Mol Formula	$C_{12}H_{19}O_4P$	m.p.	Oil

Elemental Analysis: Calc.: C: 56.25; H: 6.69; found C: 57.35; H: 7.00

^{1}H NMR 400 MHz $CDCl_3$

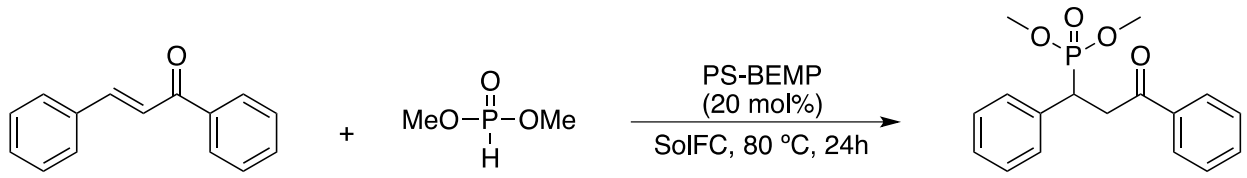
δ value	No. H	Mult.	j value/Hz
2.01	3	*s*	
3.04 – 3.10	2	*m*	
3.40	3	*d*	10.4
3.61	3	*d*	10.4
3.66 – 3.75	1	*m*	
7.15 – 7.18	1	*m*	
7.22 – 7.30	4	*m*	

^{13}C NMR (100.6 MHz, $CDCl_3$) δ : 30.19; 37.23 (*d,* $j_{P\text{-}C}$= 140.0 Hz); 43.52; 52.64 (*d,* $j_{P\text{-}C}$= 7.1 Hz); 53.47 (*d,* $j_{P\text{-}C}$= 6.7 Hz); 127.32 (*d,* $j_{P\text{-}C}$= 2.6 Hz); 128.53; 128.94 (*d,* $j_{P\text{-}C}$= 6.5 Hz); 135.42 (*d,* $j_{P\text{-}C}$= 7.0 Hz); 204.51 (*d,* $j_{P\text{-}C}$= 14.0 Hz)

^{31}P NMR (161.9 MHz, $CDCl_3$) δ : 34.51

GC-EIMS (m/z, %): 256 (M^+, 9); 214 (14); 213 (100); 110 (19); 104 (16); 103 (21); 43 (26)

Chem. Name	Dimethyl (3-oxo-1,3-diphenylpropyl)phosphonate (7b)
Lit. Ref.	*Chem. Eur. J.* **2009**, *15*, 2738-2741



1b + **4** → **7b**

M.W.: 318

METHOD:
In a screw capped vial equipped with a magnetic stirrer PS-BEMP (43 mg, 0.1 mmol, 2.13 mmol/g), (E)-chalcone (**1b**) (106 mg, 0.5 mmol), and dimethyl phosphite (**4**) (61 mg, 0.051 mL, 0.5 mmol) were consecutively added and the resulting mixture was left under stirring at 80 °C. After 24 hours EtOAc (1 mL) was added and the catalyst was filtered off and the solvent was removed under vacuum. The obtained oil was purified by flash column chromatography on silica gel (petroleum ether/EtOAc 95/5). **7b** was obtained as an oil (135 mg, 85% yield).

Mol Formula	$C_{17}H_{19}O_4P$	m.p.	Oil

Elemental Analysis: Calc.: C: 64.15; H: 6.02; found C: 64.38; H: 6.08

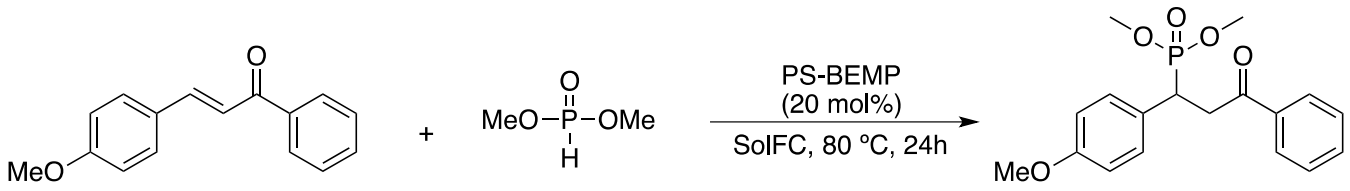
^{1}H NMR 400 MHz $CDCl_3$	δ value	No. H	Mult.	j value/Hz
	3.46	3	*d*	10.4
	3.60 – 3.77	5	*m*	
	3.95 – 4.04	1	*m*	
	7.18 – 7.22	1	*m*	
	7.26 – 7.30	2	*m*	
	7.39 – 7.43	4	*m*	
	7.50 – 7.54	1	*m*	
	7.91	2	*d*	7.6

^{13}C NMR (100.6 MHz, $CDCl_3$) δ : 37.37 (*d,* $j_{P\text{-}C}$= 140.2 Hz); 38.94; 52.66 (*d,* $j_{P\text{-}C}$= 7.2 Hz); 53.61 (*d,* $j_{P\text{-}C}$= 6.8 Hz); 127.33; 127.99; 128.55; 129.10 (*d,* $j_{P\text{-}C}$= 6.5 Hz); 133.26; 135.60 (*d,* $j_{P\text{-}C}$= 6.9 Hz); 136.40; 196.08 (*d,* $j_{P\text{-}C}$= 15.0 Hz)

^{31}P NMR (161.9 MHz, $CDCl_3$) δ : 35.02

GC-EIMS (m/z, %): 318 (M^+, 8); 214 (12); 213 (100); 105 (48); 104 (11); 103 (15); 77 (35)

Chem. Name	Dimethyl (1-(4-methoxyphenyl)-3-oxo-3-phenylpropyl)phosphonate (7d)
Lit. Ref.	/



1d **4** **7d**

M.W.: 348

METHOD:
In a screw capped vial equipped with a magnetic stirrer PS-BEMP (43 mg, 0.1 mmol, 2.13 mmol/g), (E)-3-(4-methoxyphenyl)-1-phenylprop-2-en-1-one (**1d**) (119 mg, 0.5 mmol), and dimethyl phosphite (**4**) (61 mg, 0.051 mL, 0.5 mmol) were consecutively added and the resulting mixture was left under stirring at 80 °C. After 24 hours EtOAc (1 mL) was added and the catalyst was filtered off and the solvent was removed under vacuum. The obtained oil was purified by flash column chromatography on silica gel (petroleum ether/EtOAc 95/5). **7d** was obtained as an oil (144 mg, 83% yield).

Mol Formula	$C_{18}H_{21}O_5P$	m.p.	Oil

Elemental Analysis: Calc.: C: 62.07; H: 6.08; found C: 62.22; H: 6.10

^{1}H NMR 400 MHz $CDCl_3$

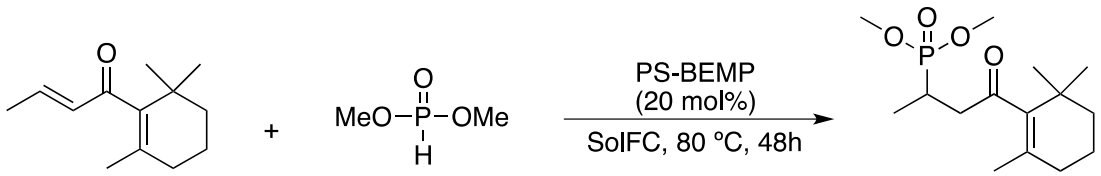
δ value	No. H	Mult.	j value/Hz
3.45	3	*d*	10.4
3.57 – 3.69	8	*m*	
3.87 – 3.96	1	*m*	
6.79	2	*d*	8.4
7.30 – 7.32	2	*m*	
7.36 – 7.40	2	*m*	
7.47 – 7.50	1	*m*	
7.88	2	*d*	7.6

^{13}C NMR (100.6 MHz, $CDCl_3$) δ : 36.47 (*d,* $j_{P\text{-}C}$= 141.1 Hz); 39.02; 52.60 (*d,* $j_{P\text{-}C}$= 7.3 Hz); 53.56 (*d,* $j_{P\text{-}C}$= 6.8 Hz); 55.03; 113.96; 127.36 (*d,* $j_{P\text{-}C}$= 6.9 Hz); 127.95; 128.52; 130.08 (*d,* $j_{P\text{-}C}$= 6.5 Hz); 133.21; 136.42; 158.72; 196.18 (*d,* $j_{P\text{-}C}$= 15.2 Hz)

^{31}P NMR (161.9 MHz, $CDCl_3$) δ : 35.27

GC-EIMS (m/z, %): 348 (M^+, 13); 244 (13); 242 (100); 134 (10); 133 (20); 105 (42); 77 (28)

Chem. Name	Dimethyl (4-oxo-4-(2,6,6-trimethylcyclohex-1-en-1-yl)butan-2-yl)phosphonate (7e)
Lit. Ref.	*Tetrahedron Lett.* **1998**, *39*, 7615-7618



1e **4** **7e**

M.W.: 302

METHOD:
In a screw capped vial equipped with a magnetic stirrer PS-BEMP (43 mg, 0.1 mmol, 2.13 mmol/g), (E)-1-(2,6,6-trimethylcyclohex-1-en-1-yl)but-2-en-1-one (**1e**) (106 mg, 0.114 mL, 0.5 mmol), and dimethyl phosphite (**4**) (61 mg, 0.051 mL, 0.5 mmol) were consecutively added and the resulting mixture was left under stirring at 80 °C. After 48 hours EtOAc (1 mL) was added and the catalyst was filtered off and the solvent was removed under vacuum. The obtained oil was purified by flash column chromatography on silica gel (petroleum ether/EtOAc 95/5). **7e** was obtained as an oil (121 mg, 80% yield).

Mol Formula	$C_{15}H_{27}O_4P$	m.p.	Oil

Elemental Analysis: Calc.: C: 59.59; H: 9.00; found C: 59.69; H: 9.05

^{1}H NMR 400 MHz $CDCl_3$

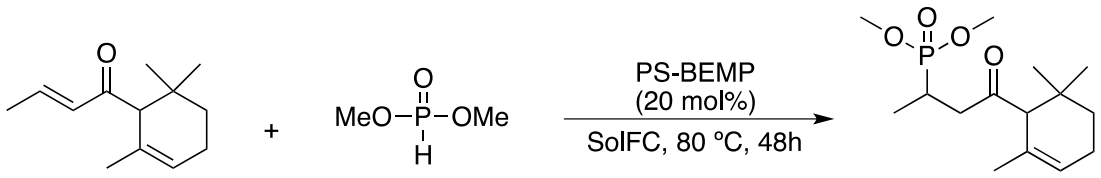
δ value	No. H	Mult.	j value/Hz
0.98	3	*s*	
0.99	3	*s*	
1.12-1.18	3	*m*	
1.35-1.38	2	*m*	
1.49	3	*s*	
1.55-1.61	2	*m*	
1.88	2	*t*	6.8
2.52-2.62	2	*m*	
2.84-2.93	1	*m*	
3.67	3	*d*	3.6
3.69	3	*d*	3.6

^{13}C NMR (100.6 MHz, $CDCl_3$) δ : 14.18 (*d,* $j_{P\text{-}C}$= 5.2 Hz); 19.13; 21.05; 25.18 (*d,* $j_{P\text{-}C}$= 143.6 Hz); 29.04; 31.56; 33.66; 39.21; 46.27; 52.84 (*d,* $j_{P\text{-}C}$= 6.7 Hz); 53.01 (*d,* $j_{P\text{-}C}$= 6.7 Hz); 130.12; 142.80; 208.26 (*d,* $j_{P\text{-}C}$= 15.9 Hz)

^{31}P NMR (161.9 MHz, $CDCl_3$) δ : 32.10

GC-EIMS (m/z, %): 302 (M^+, 21); 179 (100); 165 (55); 151 (89); 138 (24); 123 (59); 110 (31); 109 (51), 95 (19); 93 (32); 91 (25); 81 (66); 79 (50); 69 (68); 55 (26); 43 (22)

Chem. Name	Dimethyl (4-oxo-4-(2,6,6-trimethylcyclohex-2-en-1-yl)butan-2-yl)phosphonate (7f)
Lit. Ref.	/



1f + **4** → **7f**

M.W.: 302

METHOD:
In a screw capped vial equipped with a magnetic stirrer PS-BEMP (43 mg, 0.1 mmol, 2.13 mmol/g), (E)-1-(2,6,6-trimethylcyclohex-2-en-1-yl)but-2-en-1-one (**1f**) (106 mg, 0.114 mL, 0.5 mmol), and dimethyl phosphite (**4**) (61 mg, 0.051 mL, 0.5 mmol) were consecutively added and the resulting mixture was left under stirring at 80 °C. After 48 hours EtOAc (1 mL) was added and the catalyst was filtered off and the solvent was removed under vacuum. The obtained oil was purified by flash column chromatography on silica gel (petroleum ether/EtOAc 95/5). **7f** was obtained as an oil (ca. 1:1 diasteroisomeric mixture) (124 mg, 82% yield).

Mol Formula	$C_{15}H_{27}O_4P$	m.p.	Oil

Elemental Analysis: Calc.: C: 59.59; H: 9.00; found C: 59.50; H: 9.04

^{1}H NMR 400 MHz $CDCl_3$

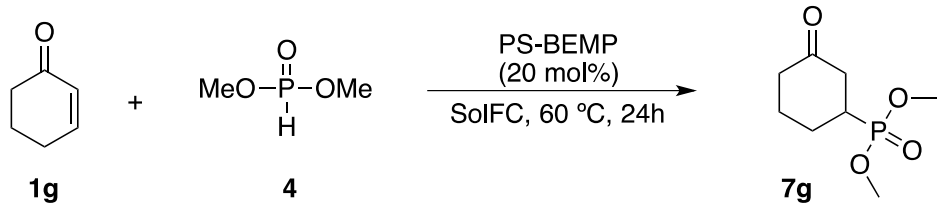
δ value	No. H	Mult.	j value/Hz
0.85 and 0.85	3	*two s*	(diasteroisomeric mixture)
0.88 and 0.89	3	*two s*	(diasteroisomeric mixture)
1.07 – 1.16	4	*m*	
1.54	3	*dd*	8.0; 1.2
2.13	3	*s*	
2.46 – 3.00	4	*m*	
3.69	3	*d*	4.0
3.71	3	*d*	4.0
5.55	1	*s*	

^{13}C NMR (100.6 MHz, $CDCl_3$) δ : 14.14 (*d,* $j_{P\text{-}C}$= 13.0 Hz); 22.97; 23.76; 25.31 (*d,* $j_{P\text{-}C}$= 145.3 Hz); 28.19; 31.08; 32.80; 46.96; 52.90 (*d,* $j_{P\text{-}C}$= 7.1. Hz); 53.04 (*d,* $j_{P\text{-}C}$= 7.0 Hz); 63.64; 124.20; 130.11; 210.68 (*d,* $j_{P\text{-}C}$= 33.2 Hz)

^{31}P NMR (161.9 MHz, $CDCl_3$) δ : 32.1

GC-EIMS (m/z, %): 302 (M^+, 7); 179 (100); 110 (18); 109 (23); 101 (22); 93 (22); 79 (20); 69 (68)

Chem. Name	Dimethyl (3-oxocyclohexyl)phosphonate (7g)
Lit. Ref.	*Tetrahedron Lett.* **1997**, *38*, 3543-3546



1g **4** **7g**

M.W.: 206

METHOD:
In a screw capped vial equipped with a magnetic stirrer PS-BEMP (43 mg, 0.1 mmol, 2.13 mmol/g), cyclohex-2-enone (**1g**) (51 mg, 0.051 mL, 0.5 mmol), and dimethyl phosphite (**4**) (61 mg, 0.051 mL, 0.5 mmol) were consecutively added and the resulting mixture was left under stirring at 60 °C. After 24 hours EtOAc (1 mL) was added and the catalyst was filtered off and the solvent was removed under vacuum. The obtained oil was purified by flash column chromatography on silica gel (petroleum ether/EtOAc 95/5). **7g** was obtained as an oil (79 mg, 80% yield).

Mol Formula	$C_8H_{15}O_4P$	**m.p.**	Oil

Elemental Analysis: Calc.: C: 46.60; H: 7.33; found C: 46.65; H: 7.29

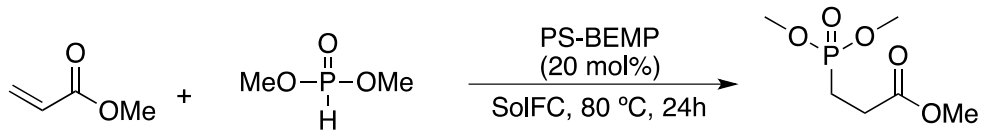
^{1}H NMR 400 MHz $CDCl_3$	**δ value**	**No. H**	**Mult.**	**j value/Hz**
	1.61-1.81	2	*m*	
	2.06-2.54	7	*m*	
	3.72	6	*d*	10.4

^{13}C NMR (100.6 MHz, $CDCl_3$) δ : 24.28 (*d,* $j_{P\text{-}C}$= 4.4 Hz); 25.90; 35.38 (*d,* $j_{P\text{-}C}$= 145.9 Hz); 40.30 (*d,* $j_{P\text{-}C}$= 5.0 Hz); 40.96; 52.74 (*d,* $j_{P\text{-}C}$= 6.9 Hz); 208.52 (*d,* $j_{P\text{-}C}$= 16.7 Hz)

^{31}P NMR (161.9 MHz, $CDCl_3$) δ : 35.27

GC-EIMS (m/z, %): 206 (M^+, 39); 163 (26); 137 (100); 111 (48); 110 (59); 109 (31); 97 (65); 96 (71); 95 (19); 80 (25); 79 (37); 69 (19); 55 (30)

Chem. Name	Methyl 3-(dimethoxyphosphoryl)propanoate (12a)
Lit. Ref.	*Tetrahedron,* **2013***, 69*, 7785-7809; *Magn. Reson. Chem.,* **1995***, 33,* 971-976.



10a **4** **12a**

M.W.: 196

METHOD:
In a screw capped vial equipped with a magnetic stirrer PS-BEMP (43 mg, 0.1 mmol, 2.13 mmol/g), methyl acrylate (**10a**) (86 mg, 0.090 mL, 1 mmol), and dimethyl phosphite (**4**) (61 mg, 0.051 mL, 0.5 mmol) were consecutively added and the resulting mixture was left under stirring at 80 °C. After 24 hours EtOAc (1 mL) was added and the catalyst was filtered off and the solvent was removed under vacuum. The obtained oil was purified by flash column chromatography on silica gel (petroleum ether/EtOAc 95/5). **12a** was obtained as an oil (83 mg, 85% yield).

Mol Formula	$C_6H_{13}O_5P$	**m.p.**	Oil

Elemental Analysis: Calc.: C: 36.74; H: 6.68; found C: 36.78; H: 6.63

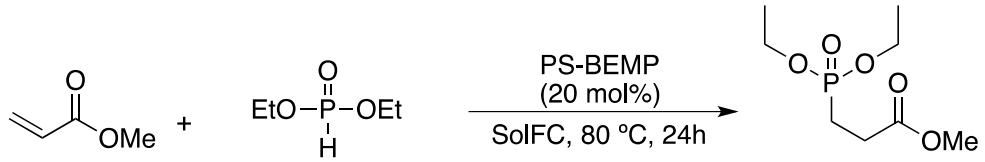
^{1}H NMR 400 MHz $CDCl_3$	**δ value**	**No. H**	**Mult.**	**j value/Hz**
	2.04-2.13	2	*m*	
	2.56-2.63	2	*m*	
	3.60	3	*s*	
	3.73	6	*d*	10.8

^{13}C NMR (100.6 MHz, $CDCl_3$) δ : 19.92 (*d, j_{P-C}*= 144.9 Hz); 27.13 (*d, j_{P-C}*= 3.9 Hz); 52.02; 52.47 (*d, j_{P-C}*= 6.4 Hz); 172.40 (*d, j_{P-C}*= 19.2 Hz)

^{31}P NMR (161.9 MHz, $CDCl_3$) δ : 33.03

GC-EIMS (m/z, %): 196 (M^+,6); 165 (100); 164 (33); 137 (51); 110 (59); 109 (83); 105 (13); 93 (18); 87 (10); 80 (12); 79 (35); 55 (21).

Chem. Name	Methyl 3-(diethoxyphosphoryl)propanoate (12b)
Lit. Ref.	*Green Chem.,* **2010**, *12,* 1171–1174



10a **2** **12b**

M.W.: 224

METHOD:
In a screw capped vial equipped with a magnetic stirrer PS-BEMP (43 mg, 0.1 mmol, 2.13 mmol/g), methyl acrylate (**10a**) (86 mg, 0.090 mL, 1 mmol), and diethyl phosphite (**2**) (70 mg, 0.065 mL, 0.5 mmol) were consecutively added and the resulting mixture was left under stirring at 80 °C. After 24 hours EtOAc (1 mL) was added and the catalyst was filtered off and the solvent was removed under vacuum. The obtained oil was purified by flash column chromatography on silica gel (petroleum ether/EtOAc 95/5). **12b** was obtained as an oil (88 mg, 79% yield).

Mol Formula	$C_8H_{17}O_5P$	**m.p.**	Oil

Elemental Analysis: Calc.: C: 42.86; H: 7.64; found C: 42.90; H: 7.59

^{1}H NMR 400 MHz $CDCl_3$	**δ value**	**No. H**	**Mult.**	**j value/Hz**
	1.30	6	*t*	7.2
	2.01-2.10	2	*m*	
	2.55-2.62	2	*m*	
	3.68	3	*s*	
	4.06-4.11	4	*m*	

^{13}C NMR (100.6 MHz, $CDCl_3$) δ : 16.35 (*d,* $j_{P\text{-}C}$= 5.9 Hz); 20.92 (*d,* $j_{P\text{-}C}$= 143.8 Hz); 27.28 (*d,* $j_{P\text{-}C}$= 3.4 Hz); 51.96; 61.74 (*d,* $j_{P\text{-}C}$= 6.2 Hz); 172.50 (*d,* $j_{P\text{-}C}$= 18.8 Hz)

^{31}P NMR (161.9 MHz, $CDCl_3$) δ : 29.80

GC-EIMS (m/z, %): 224 (M^+, 4);197 (13), 193 (17); 192 (10); 179 (16); 165 (100); 151 (34); 138 (42); 137 (93); 123 (10) 111 (16); 109 (31); 95 (11); 91 (10); 81 (17); 65 (10); 55 (24)

Chem. Name	Methyl 3-(dimethoxyphosphoryl)butanoate (12c)
Lit. Ref.	*Synthesis,* **1999**, 1056-1062

10b **4** **12c**

M.W.: 210

METHOD:
In a screw capped vial equipped with a magnetic stirrer PS-BEMP (43 mg, 0.1 mmol, 2.13 mmol/g), methyl crotonate (**10b**) (100 mg, 0.106 mL, 1 mmol), and dimethyl phosphite (**4**) (61 mg, 0.051 mL, 0.5 mmol) were consecutively added and the resulting mixture was left under stirring at 80 °C. After 24 hours EtOAc (1 mL) was added and the catalyst was filtered off and the solvent was removed under vacuum. The obtained oil was purified by flash column chromatography on silica gel (petroleum ether/EtOAc 95/5). **12c** was obtained as an oil (36 mg, 34% yield).

Mol Formula	$C_7H_{15}O_5P$	**m.p.**	Oil

Elemental Analysis: Calc.: C: 40.01; H: 7.19; found C: 40.22; H: 7.24

^{1}H NMR 400 MHz $CDCl_3$

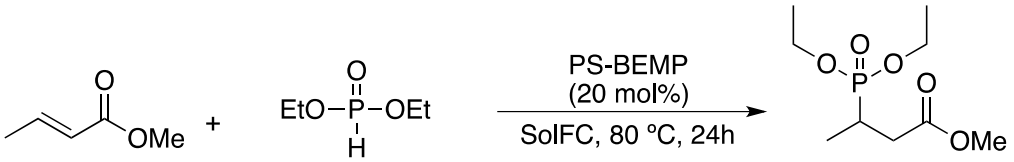
δ value	No. H	Mult.	j value/Hz
1.19	3	*dd*	6.9; 18.2
2.29-2.35	1	*m*	
2.38-2.44	1	*m*	
2.70-2.76	1	*m*	
3.68	3	*s*	
3.74	6	*d*	10.4

^{13}C NMR (100.6 MHz, $CDCl_3$) δ : 13.58 (*d,* $j_{P\text{-}C}$= 4.4 Hz); 27.59 (*d,* $j_{P\text{-}C}$= 144.8 Hz); 35.00; 51.89; 52.65 (*d,* $j_{P\text{-}C}$= 6.8 Hz); 52.78 (*d,* $j_{P\text{-}C}$= 6.8 Hz); 171.95 (*d,* $j_{P\text{-}C}$= 18.9 Hz)

^{31}P NMR (161.9 MHz, $CDCl_3$) δ : 35.43

GC-EIMS (m/z, %): 210 (M^+, 5); 179 (52); 151 (100); 150 (11); 137 (44); 124 (10); 119 (12);110 (64); 109 (54); 101 (13); 93 (10); 80 (14); 79 (30); 69 (23)

Chem. Name	Methyl 3-(diethoxyphosphoryl)butanoate (12d)
Lit. Ref.	*Synthesis,* **1999**, 1056-1062



10b **2** **12d**

M.W.: 238

METHOD:
In a screw capped vial equipped with a magnetic stirrer PS-BEMP (43 mg, 0.1 mmol, 2.13 mmol/g), methyl crotonate (**10b**) (100 mg, 0.106 mL, 1 mmol), and diethyl phosphite (**2**) (70 mg, 0.065 mL, 0.5 mmol) were consecutively added and the resulting mixture was left under stirring at 80 °C. After 24 hours EtOAc (1 mL) was added and the catalyst was filtered off and the solvent was removed under vacuum. The obtained oil was purified by flash column chromatography on silica gel (petroleum ether/EtOAc 95/5). **12d** was obtained as an oil (62 mg, 52% yield).

Mol Formula	$C_9H_{19}O_5P$	**m.p.**	Oil

Elemental Analysis: Calc.: C: 45.38; H: 8.04; found C: 45.12; H: 7.97

^{1}H NMR 400 MHz $CDCl_3$

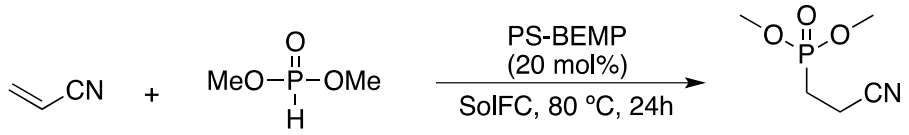
δ value	No. H	Mult.	j value/Hz
1.20	3	*dd*	6.9; 18.2
1.31	6	*t*	7.0
2.26-2.36	2	*m*	
2.72 – 2.79	1	*m*	
3.69	3	*s*	
4.07-4.13	4	*m*	

^{13}C NMR (100.6 MHz, $CDCl_3$) δ : 13.60(*d,* $j_{P\text{-}C}$= 5.2 Hz); 16.40 (*d,* $j_{P\text{-}C}$= 5.8 Hz); 27.59 (*d,* $j_{P\text{-}C}$= 145.0 Hz); 35.12; 51.84; 61.80 (*d,* $j_{P\text{-}C}$= 6.8 Hz); 61.87 (*d,* $j_{P\text{-}C}$= 7.5 Hz); 172.12 (*d,* $j_{P\text{-}C}$= 19.0 Hz)

^{31}P NMR (161.9 MHz, $CDCl_3$) δ : 32.86

GC-EIMS (m/z, %): 238 (M^+, 3); 207 (22); 193 (13); 179 (100); 165 (40); 152 (11); 151 (55); 138 (62); 137 (10); 123 (22); 111 (41); 110 (12); 109 (23); 87 (12); 82 (16); 81 (20); 69 (49); 65 (12); 59 (16)

Chem. Name	Dimethyl (2-cyanoethyl)phosphonate (13a)
Lit. Ref.	*Tetrahedron,* **2003***, 59,* 7901-7906



11a **4** **13a**

M.W.: 163

METHOD:
In a screw capped vial equipped with a magnetic stirrer PS-BEMP (43 mg, 0.1 mmol, 2.13 mmol/g), acrylonitrile (**11a**) (53 mg, 0.065 mL, 1 mmol), and dimethyl phosphite (**4**) (61 mg, 0.051 mL, 0.5 mmol) were consecutively added and the resulting mixture was left under stirring at 80 °C. After 24 hours EtOAc (1 mL) was added and the catalyst was filtered off and the solvent was removed under vacuum. The obtained oil was purified by flash column chromatography on silica gel (petroleum ether/EtOAc 95/5). **13a** was obtained as an oil (73 mg, 90% yield).

Mol Formula	$C_5H_{10}NO_3P$	m.p.	Oil

Elemental Analysis: Calc.: C: 36.82; H: 6.18; N: 8.59; found C: 36.61; H: 6.04; N: 8.48

^{1}H NMR 400 MHz $CDCl_3$	δ value	No. H	Mult.	j value/Hz
	2.04-2.15	2	*m*	
	2.60-2.77	2	*m*	
	3.80	6	*d*	8.5

^{13}C NMR (100.6 MHz, $CDCl_3$) δ : 11.47 (*d,* $j_{P\text{-}C}$= 4.4 Hz); 21.15 (*d,* $j_{P\text{-}C}$= 146.2 Hz); 52.86 (*d,* $j_{P\text{-}C}$= 6.5 Hz); 118.16 (*d,* $j_{P\text{-}C}$= 17.5 Hz)

^{31}P NMR (161.9 MHz, $CDCl_3$) δ : 28.36

GC-EIMS (m/z, %): 163 (M^+, 4); 132 (10); 110 (100); 109 (100); 95 (11); 80 (14); 79 (47); 68 (12); 47 (11)

Chem. Name	Diethyl (2-cyanoethyl)phosphonate (13b)
Lit. Ref.	*J. Org. Chem.*, **1997**, *62*, 2414-2422

11a **2** **13b**

M.W.: 191

METHOD:
In a screw capped vial equipped with a magnetic stirrer PS-BEMP (43 mg, 0.1 mmol, 2.13 mmol/g), acrylonitrile (**11a**) (53 mg, 0.065 mL, 1 mmol), and diethyl phosphite (**2**) (70 mg, 0.065 mL, 0.5 mmol) were consecutively added and the resulting mixture was left under stirring at 80 °C. After 24 hours EtOAc (1 mL) was added and the catalyst was filtered off and the solvent was removed under vacuum. The obtained oil was purified by flash column chromatography on silica gel (petroleum ether/EtOAc 95/5). **13b** was obtained as an oil (85 mg, 89% yield).

Mol Formula	$C_7H_{14}NO_3P$	m.p.	Oil

Elemental Analysis: Calc.: C: 43.98; H: 7.38; N: 7.33; found C: 44.12; H: 7.50; N: 7.54

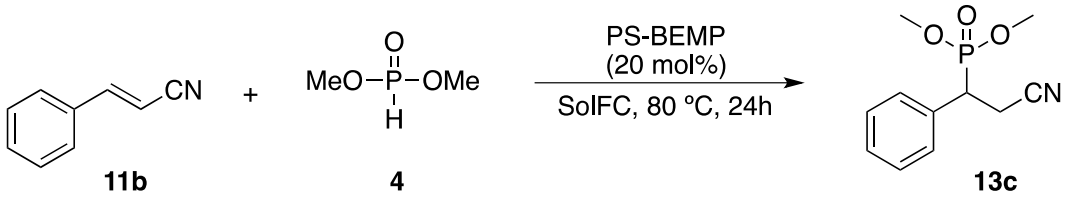
^{1}H NMR 400 MHz $CDCl_3$	δ value	No. H	Mult.	j value/Hz
	1.34	6	*t*	6.8
	1.97-2.06	2	*m*	
	2.60-2.67	2	*m*	
	4.09-4.19	4	*m*	

^{13}C NMR (100.6 MHz, $CDCl_3$) δ : 11.54 (*d, j_{P-C}*= 3.6 Hz); 16.35 (*d, j_{P-C}*= 5.7 Hz); 22.02 (*d, j_{P-C}*= 145.8 Hz); 62.34 (*d, j_{P-C}*= 6.4 Hz); 118.33 (*d, j_{P-C}*= 18.1 Hz)

^{31}P NMR (161.9 MHz, $CDCl_3$) δ : 25.69

GC-EIMS (m/z, %): 191 (M^+, 4); 164 (50); 146 (11); 138 (12); 136 (100); 135 (15); 118 (25); 109 (14); 82 (11); 81 (12); 54 (22)

Chem. Name	Dimethyl (2-cyano-1-phenylethyl)phosphonate (13c)
Lit. Ref.	/



11b + **4** → **13c**

M.W.: 239

METHOD:
In a screw capped vial equipped with a magnetic stirrer PS-BEMP (43 mg, 0.1 mmol, 2.13 mmol/g), cinnamonitrile (**11b**) (129 mg, 0.126 mL, 1 mmol), and dimethyl phosphite (**4**) (61 mg, 0.051 mL, 0.5 mmol) were consecutively added and the resulting mixture was left under stirring at 80 °C. After 24 hours EtOAc (1 mL) was added and the catalyst was filtered off and the solvent was removed under vacuum. The obtained oil was purified by flash column chromatography on silica gel (petroleum ether/EtOAc 70/30). **13c** was obtained as an oil (91 mg, 76% yield).

Mol Formula	$C_{11}H_{14}NO_3P$	m.p.	Oil

Elemental Analysis: Calc.: C: 55.23; H: 5.90; N: 5.86; found C: 55.48; H: 6.02; N: 5.98

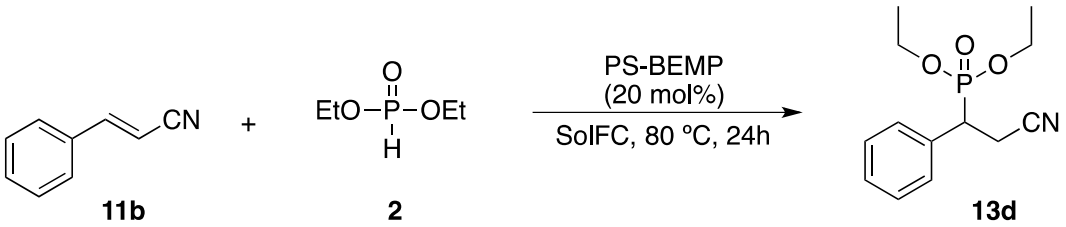
^{1}H NMR 400 MHz $CDCl_3$	δ value	No. H	Mult.	j value/Hz	
	2.94-3.14	2	*m*		
	3.41	1	*ddd*	22.0; 10.4; 5.6	
	3.50	3	*d*	10.6	
	3.74	3	*d*	10.9	
	7.31-7.42	5	*m*		

^{13}C NMR (100.6 MHz, $CDCl_3$) δ : 19.53; 40.47 (*d,* $j_{P\text{-}C}$= 141.9 Hz); 53.07 (*d,* $j_{P\text{-}C}$= 7.2 Hz); 54.05 (*d,* $j_{P\text{-}C}$= 6.9 Hz); 117.23 (*d,* $j_{P\text{-}C}$= 19.6 Hz); 128.55; 128.70 (*d,* $j_{P\text{-}C}$= 6.6 Hz); 129.13; 132.81 (*d,* $j_{P\text{-}C}$= 6.6 Hz)

^{31}P NMR (161.9 MHz, $CDCl_3$) δ : 26.25

GC-EIMS (m/z, %): 239 (M^+, 68); 238 (10); 224 (27); 207 (13); 131 (11); 130 (100); 129 (12); 128 (10); 110 (93); 109 (36); 104 (72); 103 (47); 102 (15); 93 (14); 80 (13); 79 (19); 78 (12); 77 (32)

Chem. Name	Diethyl (2-cyano-1-phenylethyl)phosphonate (13d)
Lit. Ref.	/



11b **2** **13d**

M.W.: 267

METHOD:
In a screw capped vial equipped with a magnetic stirrer PS-BEMP (43 mg, 0.1 mmol, 2.13 mmol/g), cinnamonitrile (**11b**) (129 mg, 0.126 mL, 1 mmol), and diethyl phosphite (**2**) (70 mg, 0.065 mL, 0.5 mmol) were consecutively added and the resulting mixture was left under stirring at 80 °C. After 24 hours EtOAc (1 mL) was added and the catalyst was filtered off and the solvent was removed under vacuum. The obtained oil was purified by flash column chromatography on silica gel (petroleum ether/EtOAc 70/30). **13d** was obtained as an oil (115 mg, 86% yield).

Mol Formula	$C_{13}H_{18}NO_3P$	m.p.	Oil

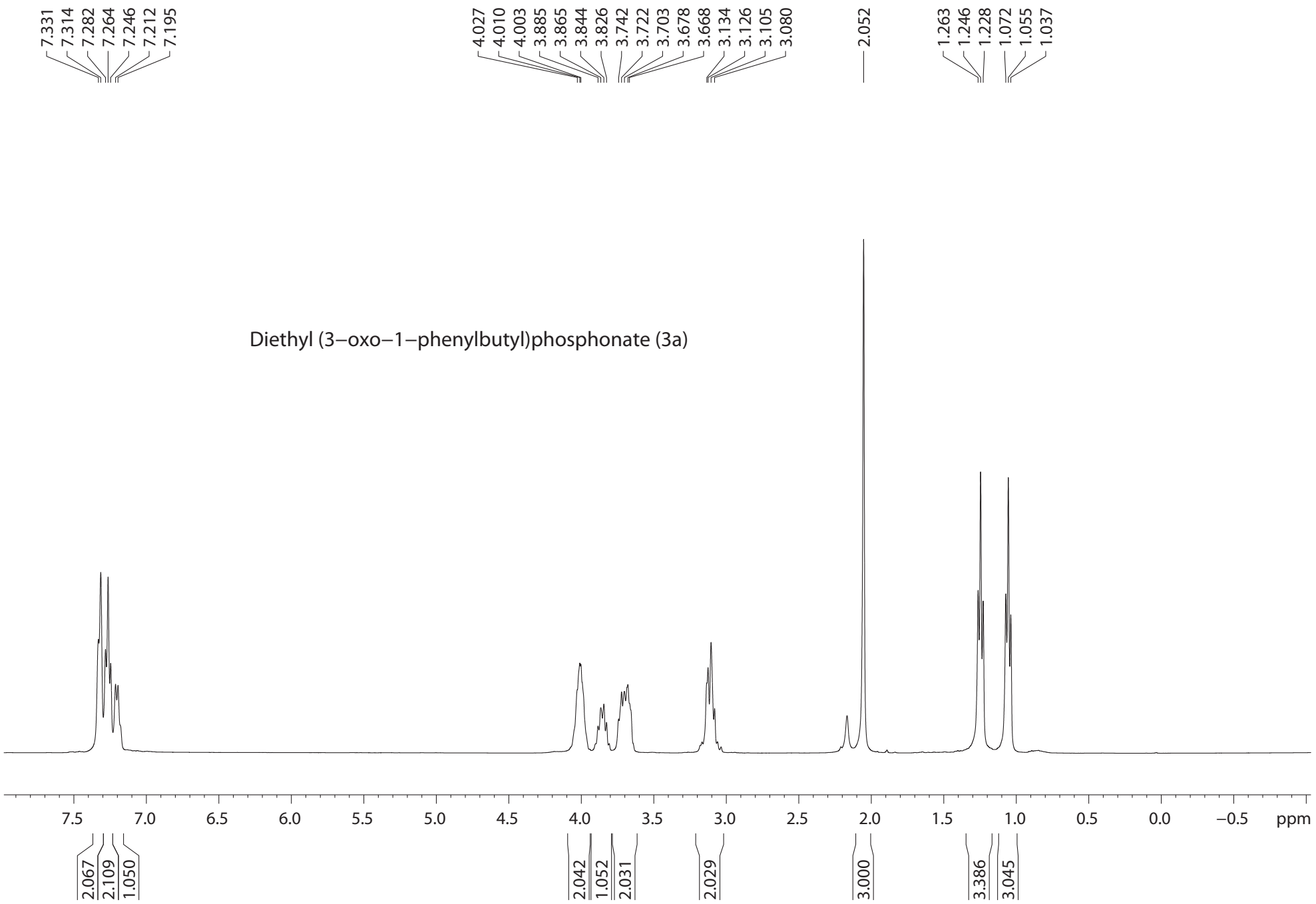
Elemental Analysis: Calc.: C: 58.42; H: 6.79; N: 5.24; found C: 58.61; H: 6.64; N: 5.16

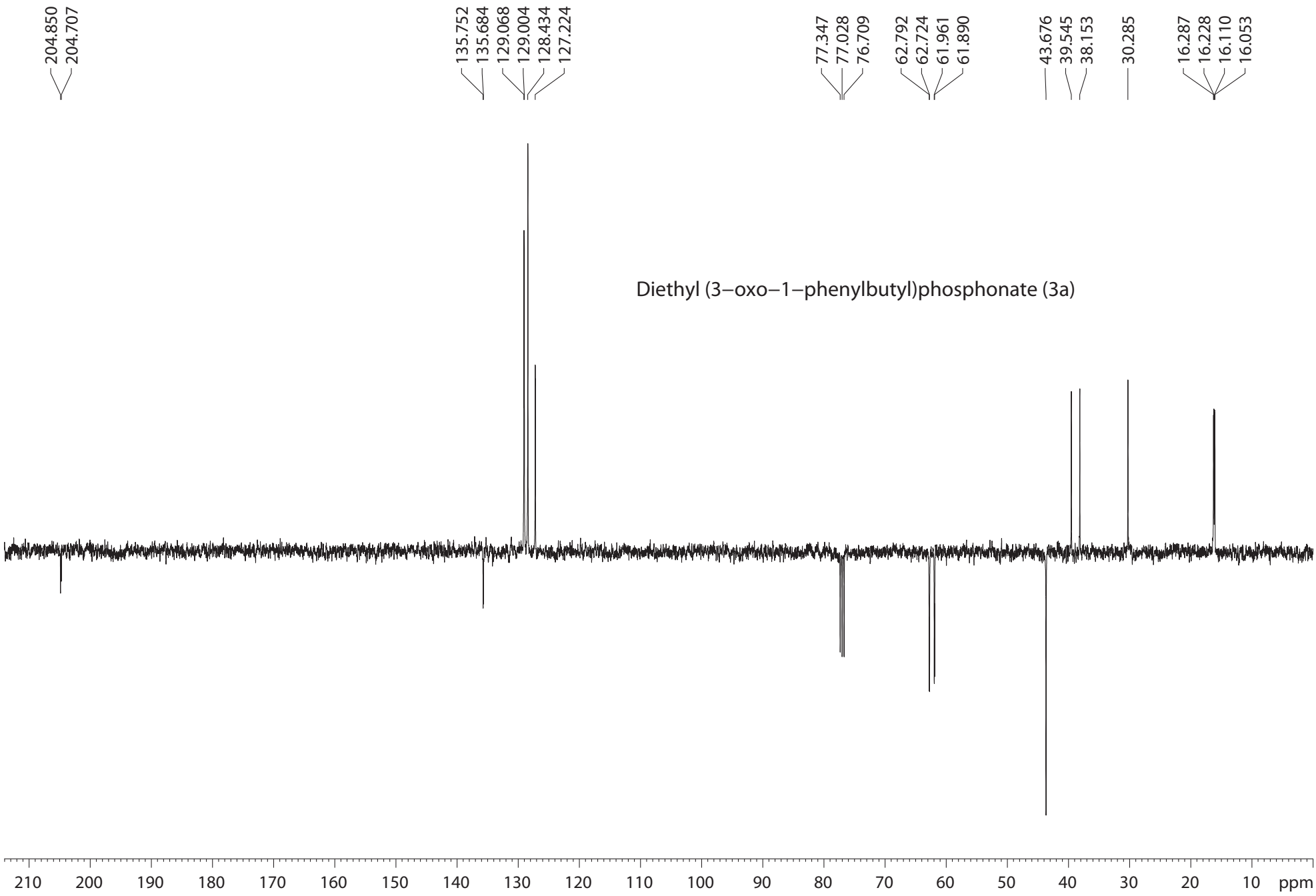
^{1}H NMR 400 MHz $CDCl_3$	δ value	No. H	Mult.	j value/Hz
	1.11	3	*t*	7.2
	1.32	3	*t*	6.8
	2.99-3.10	2	*m*	
	3.37	1	*ddd*	21.6; 10.4; 4.8
	3.72-3.76	1	*m*	
	3.90-3.94	1	*m*	
	4.07-4.11	2	*m*	
	7.32-7.41	5	*m*	

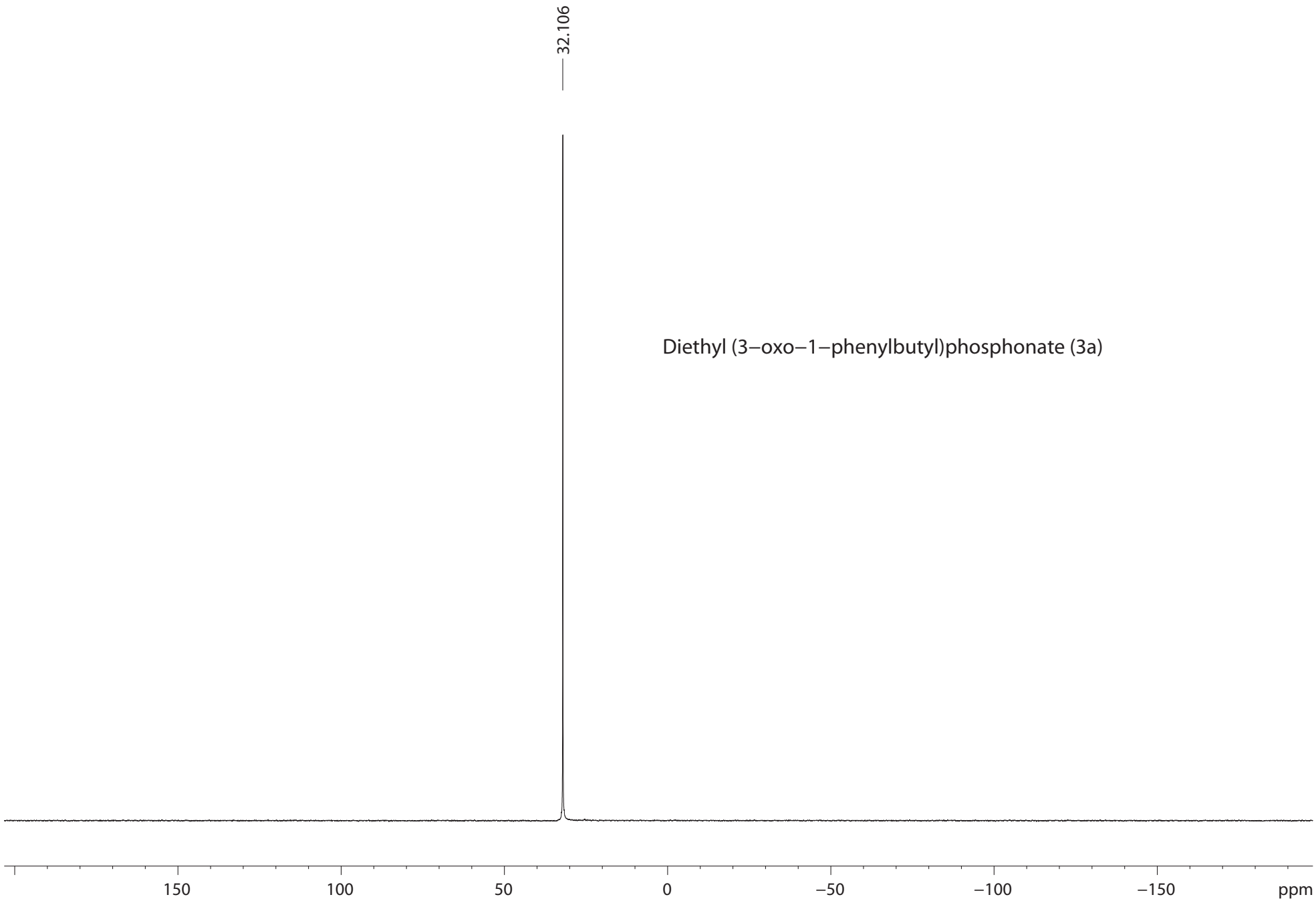
^{13}C NMR (100.6 MHz, $CDCl_3$) δ : 16.13 (*d,* $j_{P\text{-}C}$= 5.6 Hz); 16.32 (*d,* $j_{P\text{-}C}$= 5.9 Hz); 19.60; 40.87 (*d,* $j_{P\text{-}C}$= 141.8 Hz); 62.53 (*d,* $j_{P\text{-}C}$= 5.6 Hz); 63.51 (*d,* $j_{P\text{-}C}$= 6.9 Hz); 117.40 (*d,* $j_{P\text{-}C}$= 19.8 Hz); 128.39; 128.77 (*d,* $j_{P\text{-}C}$= 6.3 Hz); 128.99; 133.11 (*d,* $j_{P\text{-}C}$= 6.6 Hz)

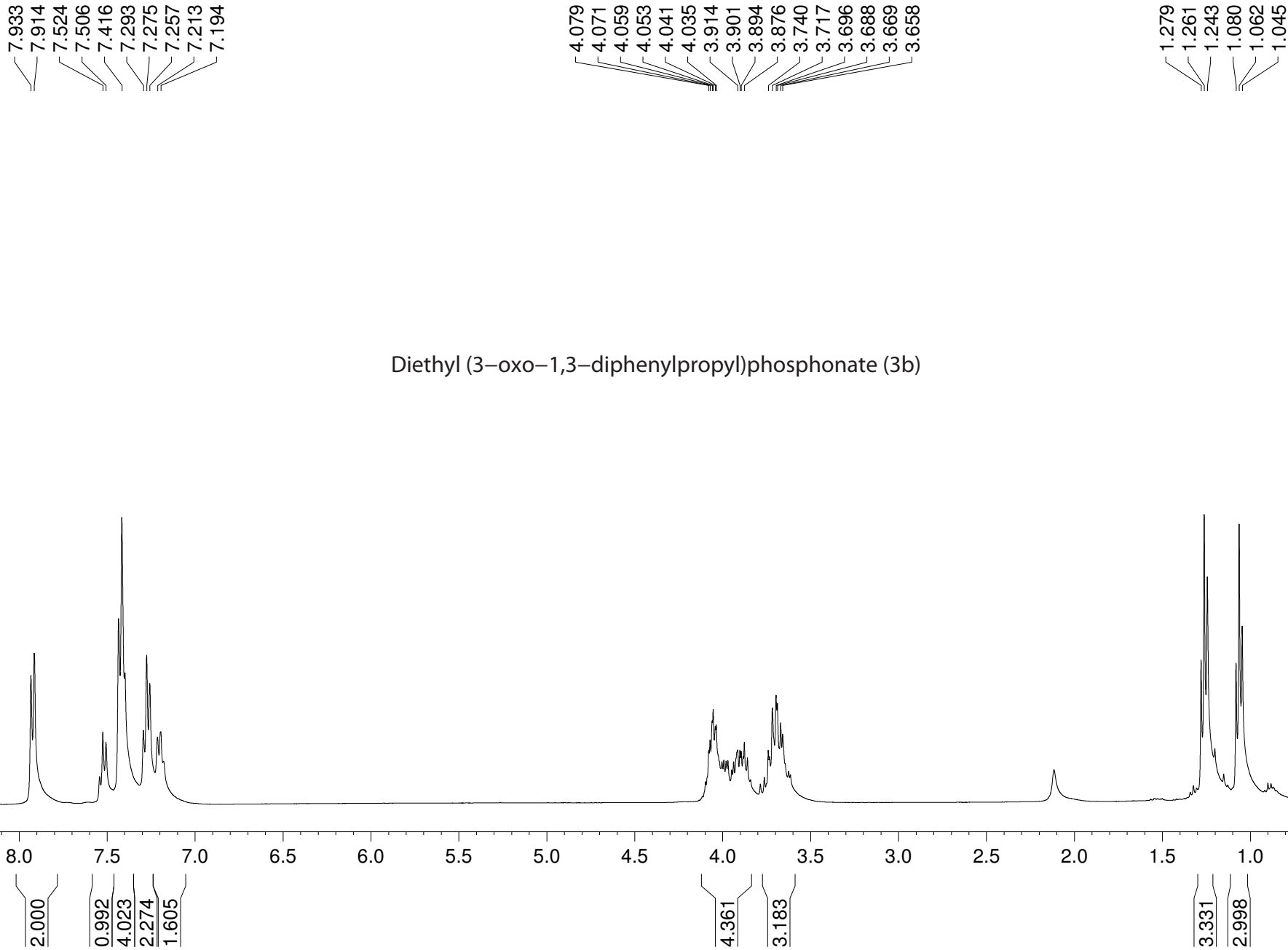
^{31}P NMR (161.9 MHz, $CDCl_3$) δ : 23.90

GC-EIMS (m/z, %): 267 (M^+, 42); 238 (14); 211 (13); 210 (10); 138 (36); 131 8 (10); 130 (65); 129 (10); 111 (20); 109 (21); 105 (11); 104 (100); 103 (41); 102 (10); 91 (14); 81 (15); 77 (24)

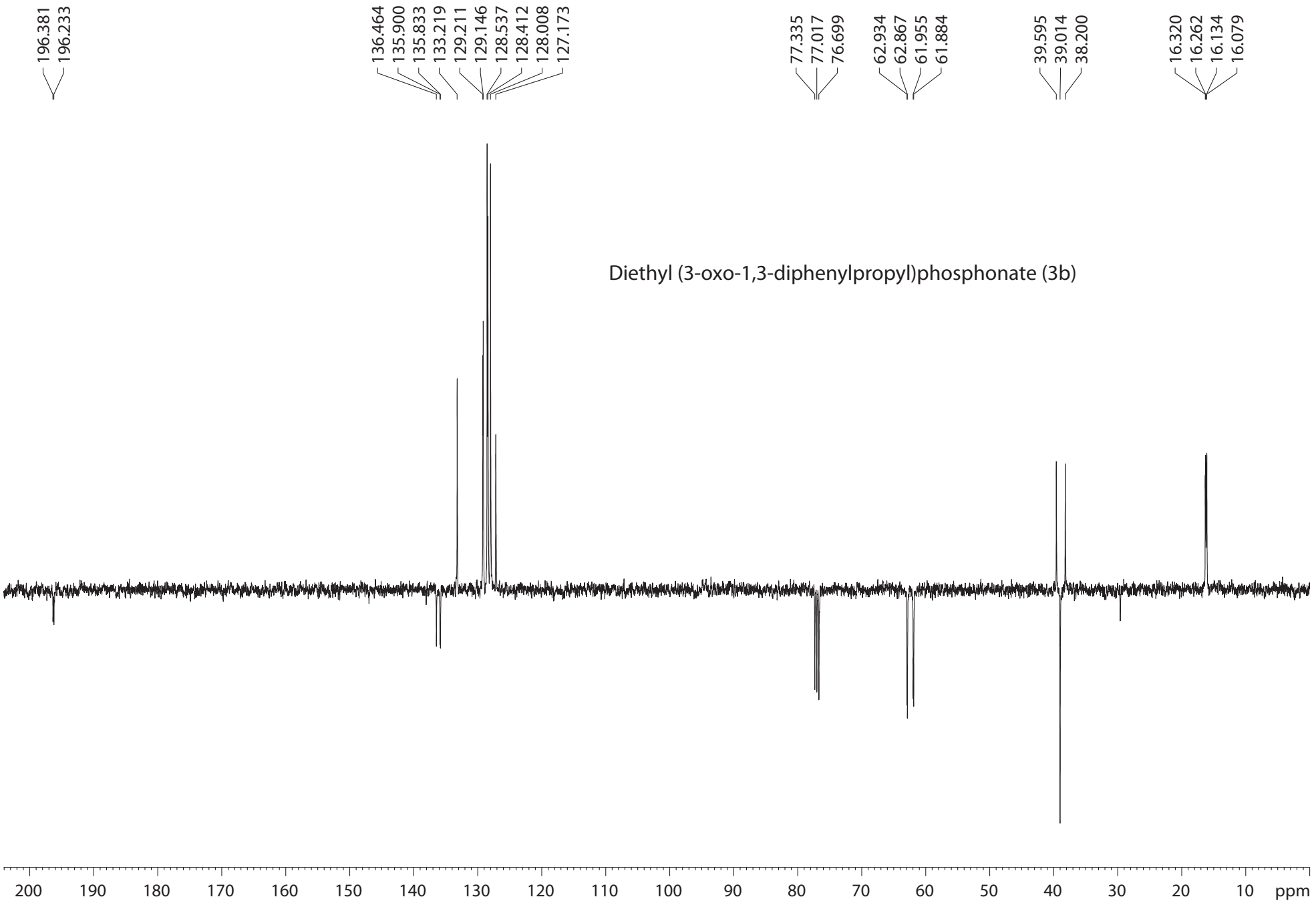

7.331
7.314
7.282
7.264
7.246
7.212
7.195
4.027
4.010
4.003
3.885
3.865
3.844
3.826
3.742
3.722
3.703
3.678
3.668
3.134
3.126
3.105
3.080
2.052
1.263
1.246
1.228
1.072
1.055
1.037
Diethyl (3−oxo−1−phenylbutyl)phosphonate (3a)
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
−0.5
ppm
2.067
2.109
1.050
2.042
1.052
2.031
2.029
3.000
3.386
3.045

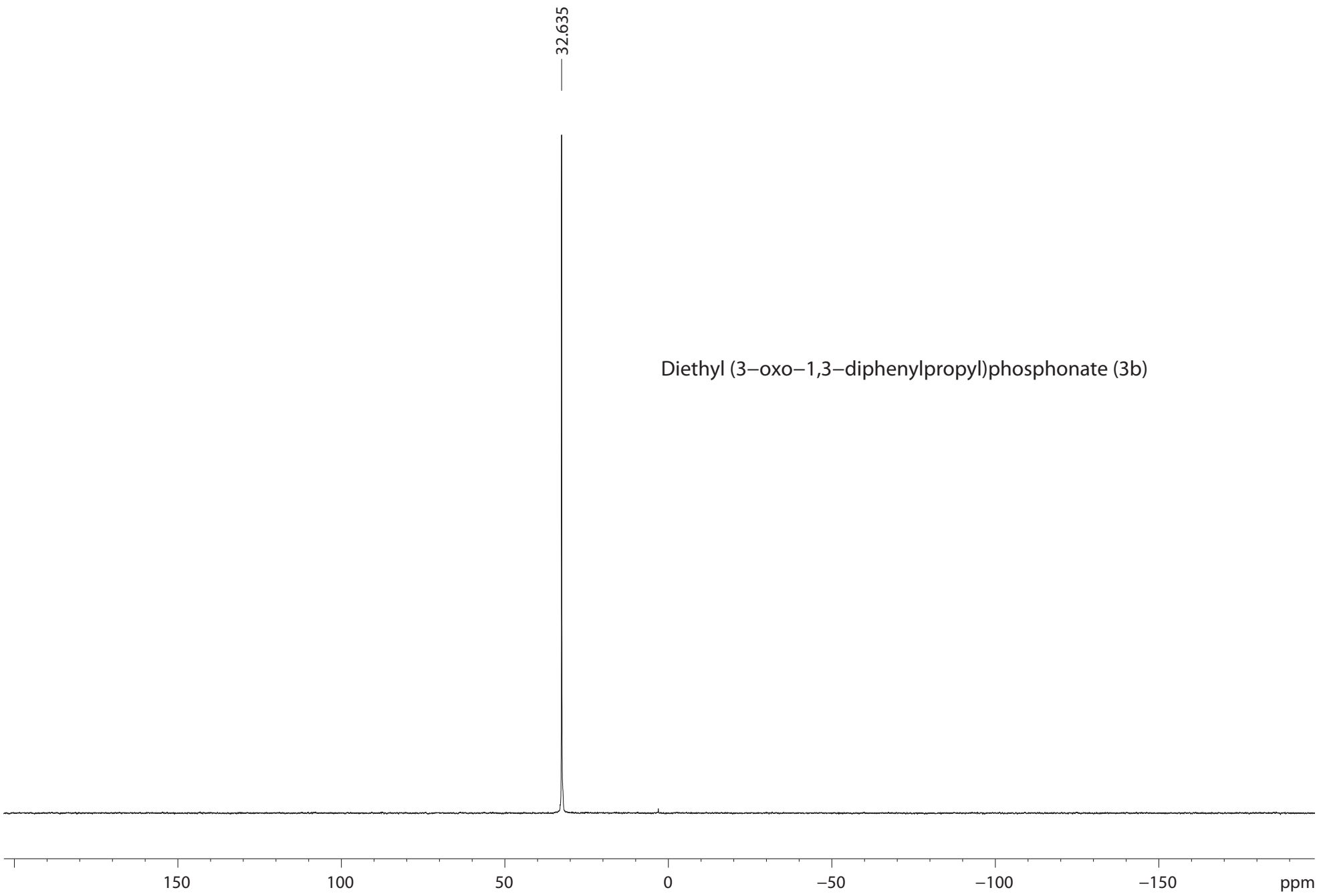

204.850
204.707
135.752
135.684
129.068
129.004
128.434
127.224
77.347
77.028
76.709
62.792
62.724
61.961
61.890
43.676
39.545
38.153
30.285
16.287
16.228
16.110
16.053
Diethyl (3−oxo−1−phenylbutyl)phosphonate (3a)
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

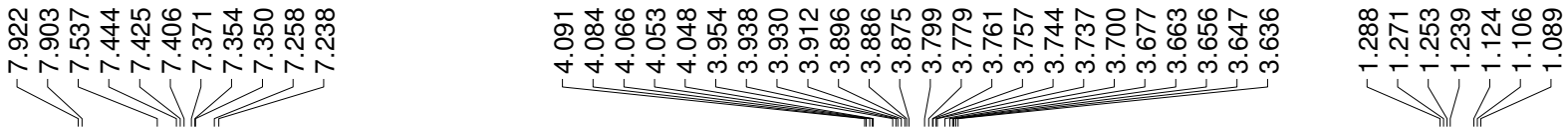

32.106
Diethyl (3−oxo−1−phenylbutyl)phosphonate (3a)
150
100
50
0
−50
−100
−150
ppm



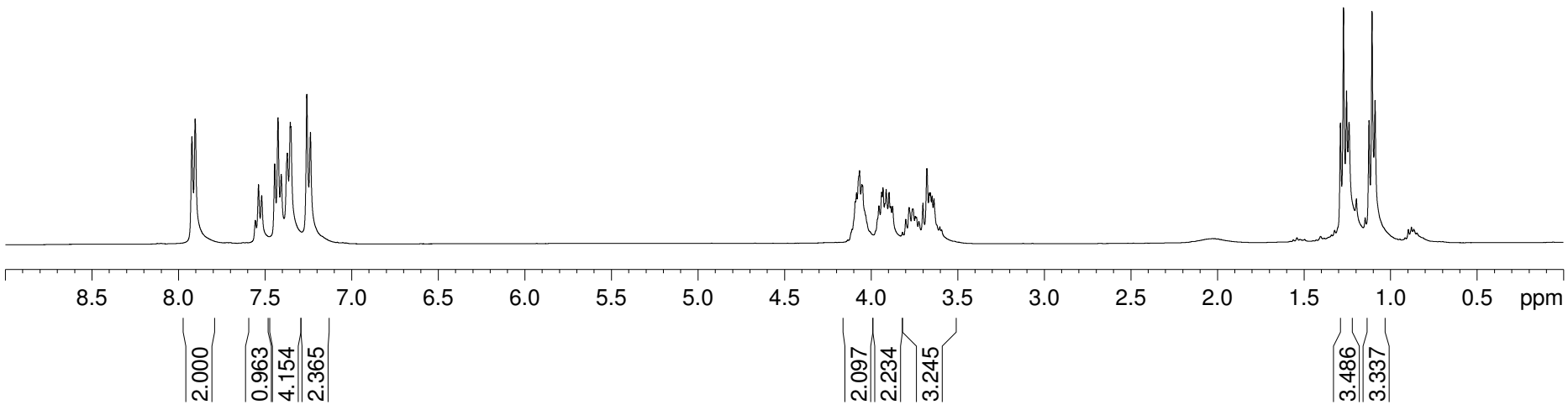
Diethyl (3–oxo–1,3–diphenylpropyl)phosphonate (3b)

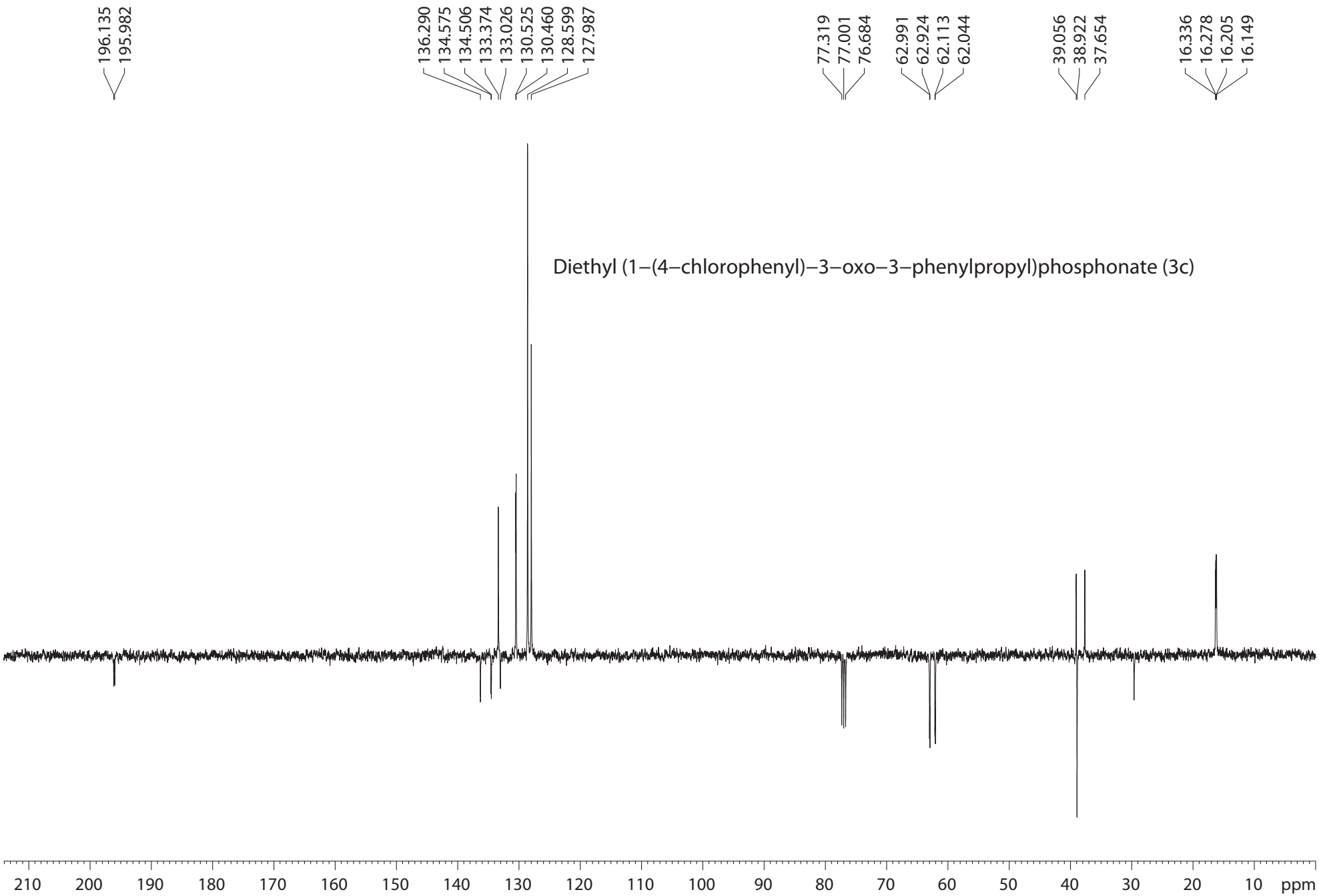

196.381
196.233
136.464
135.900
135.833
133.219
129.211
129.146
128.537
128.412
128.008
127.173
77.335
77.017
76.699
62.934
62.867
61.955
61.884
39.595
39.014
38.200
16.320
16.262
16.134
16.079
Diethyl (3-oxo-1,3-diphenylpropyl)phosphonate (3b)
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

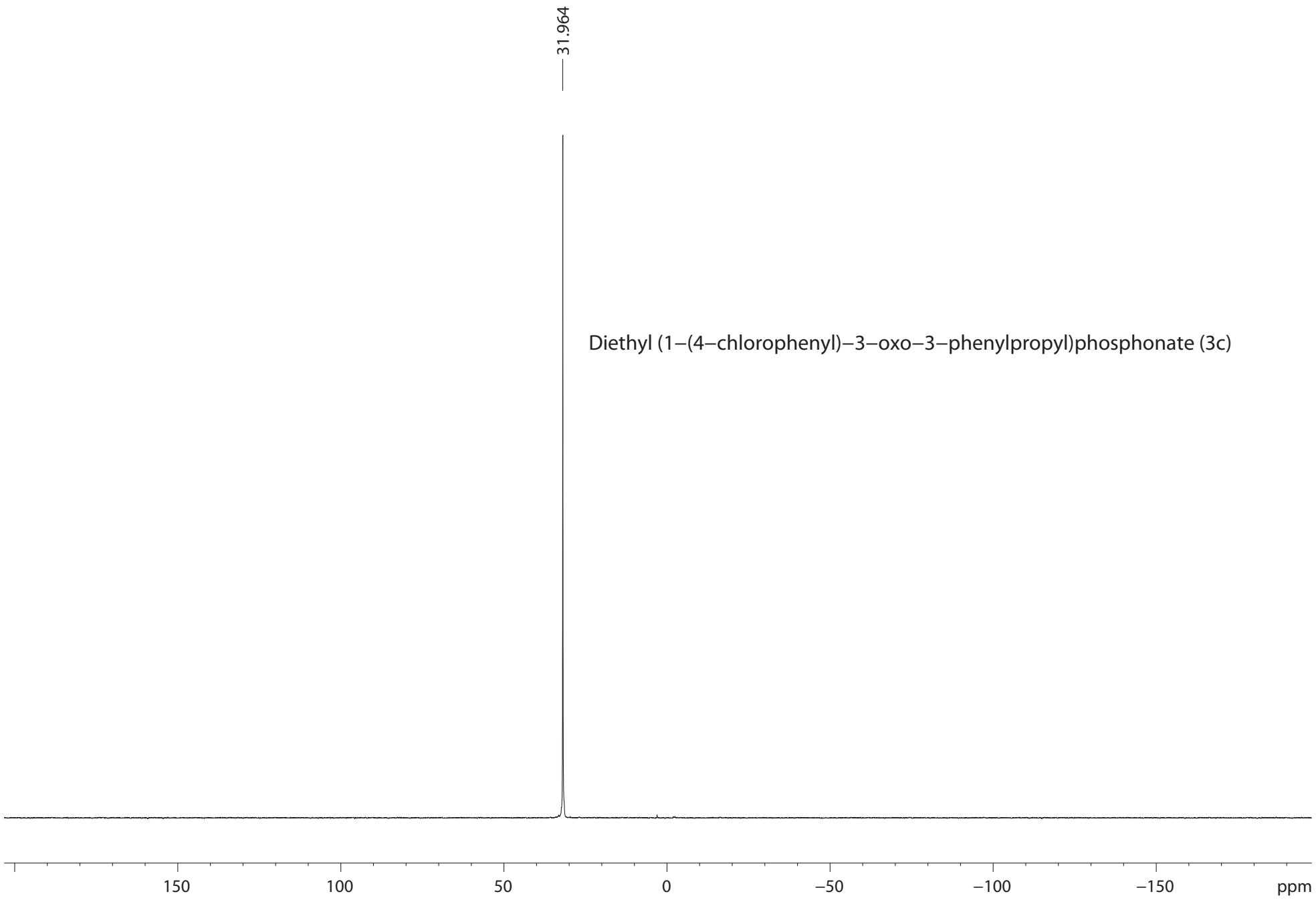

32.635
Diethyl (3–oxo–1,3–diphenylpropyl)phosphonate (3b)
150
100
50
0
–50
–100
–150
ppm

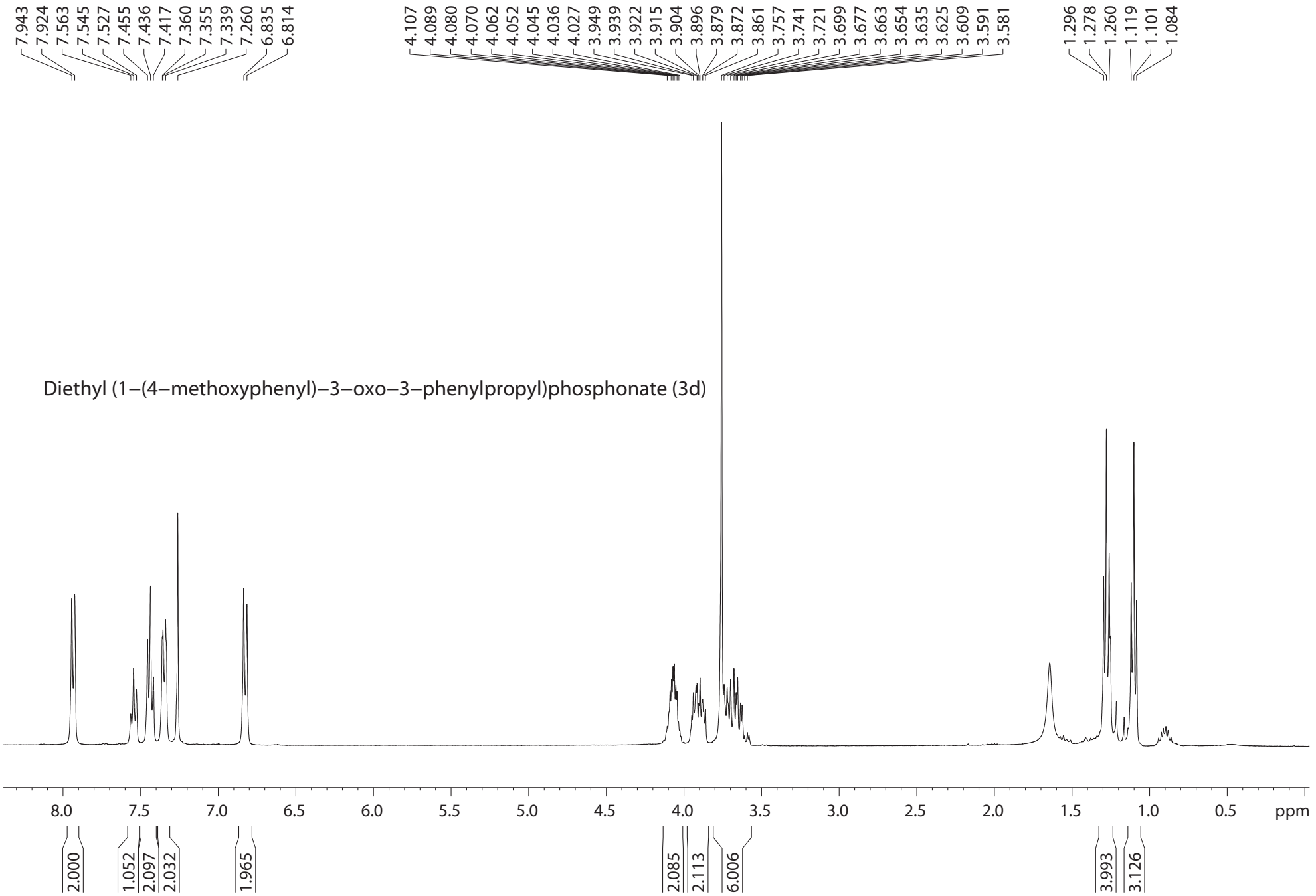


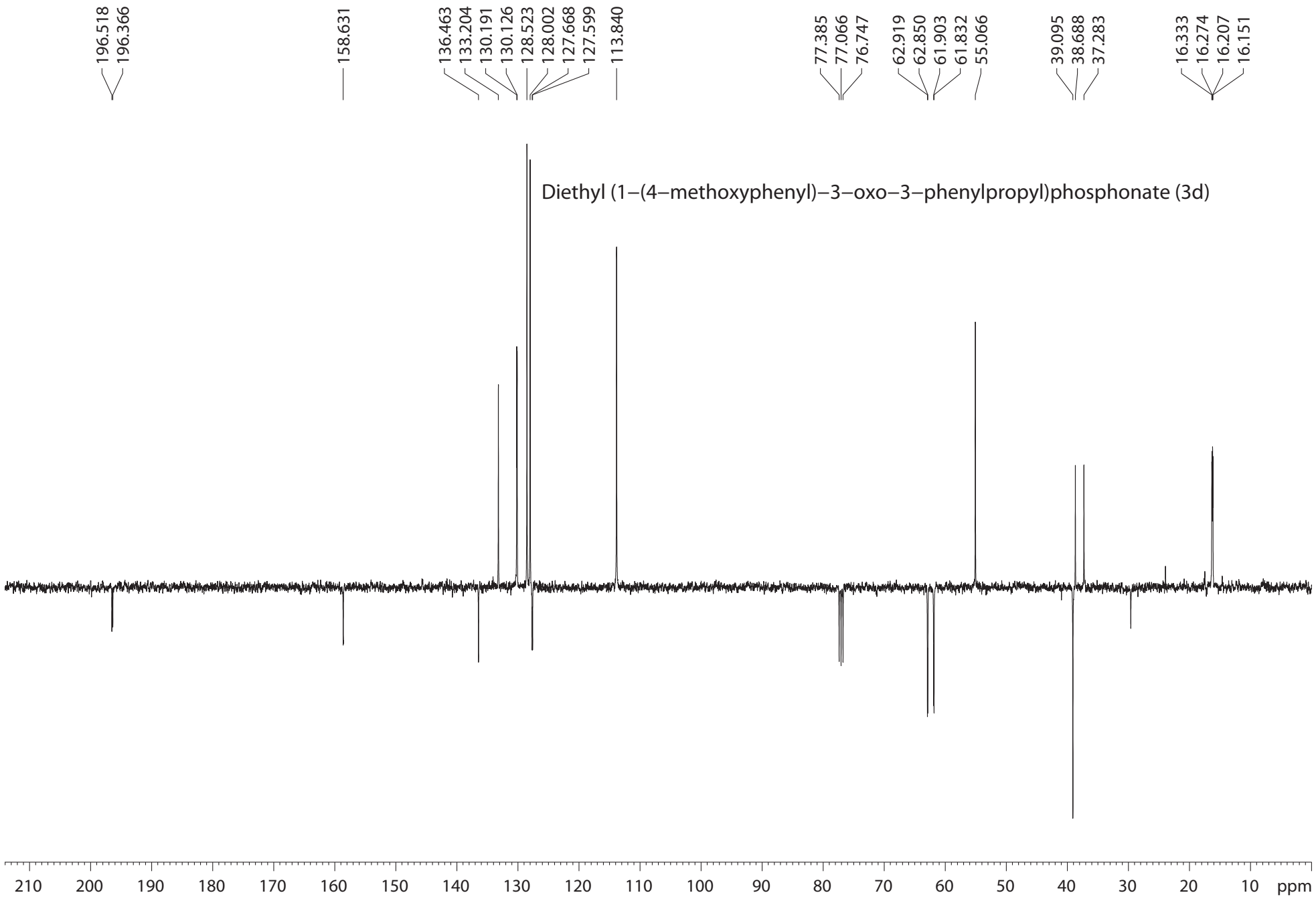
Diethyl (1–(4–chlorophenyl)–3–oxo–3–phenylpropyl)phosphonate (3c)

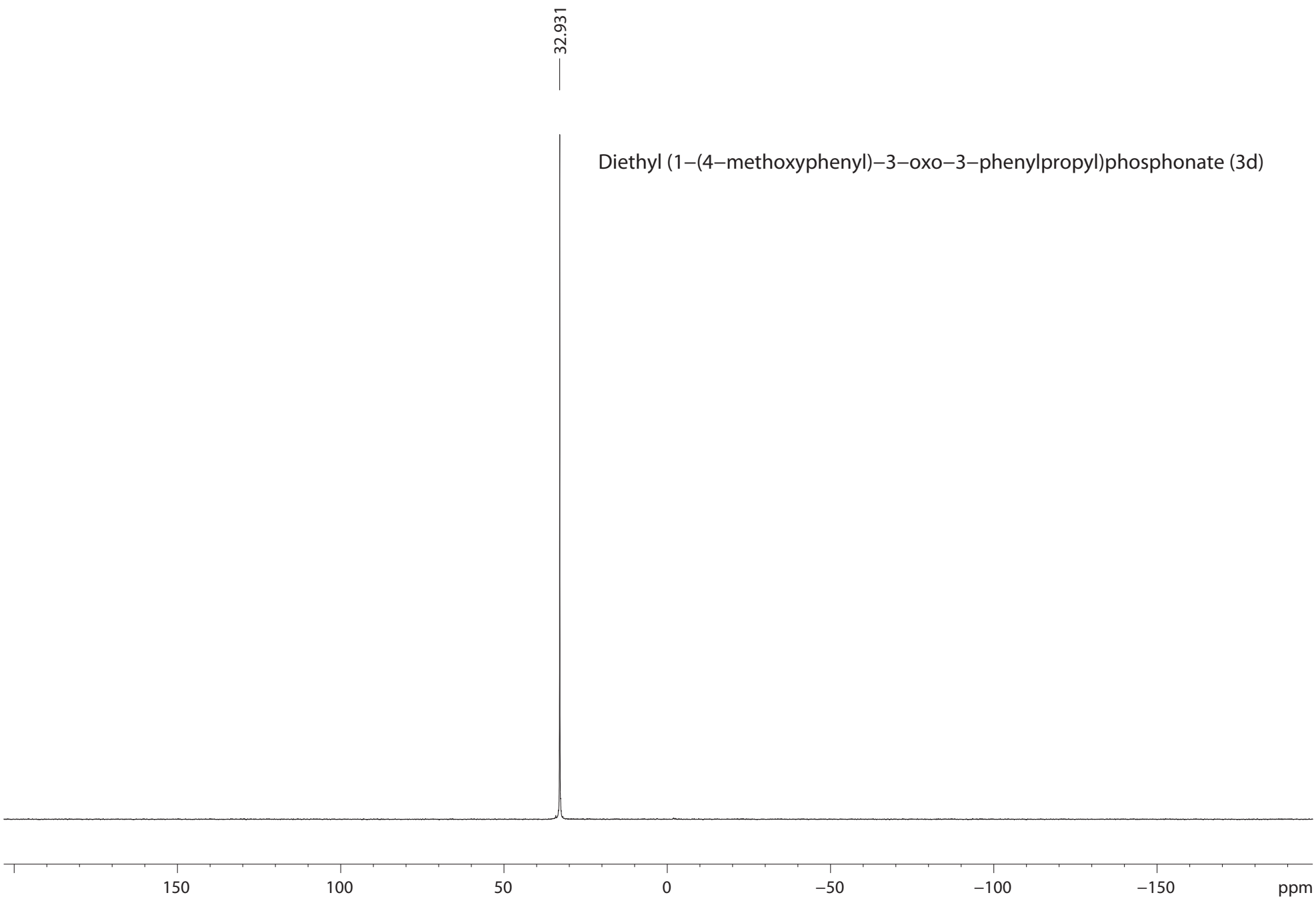


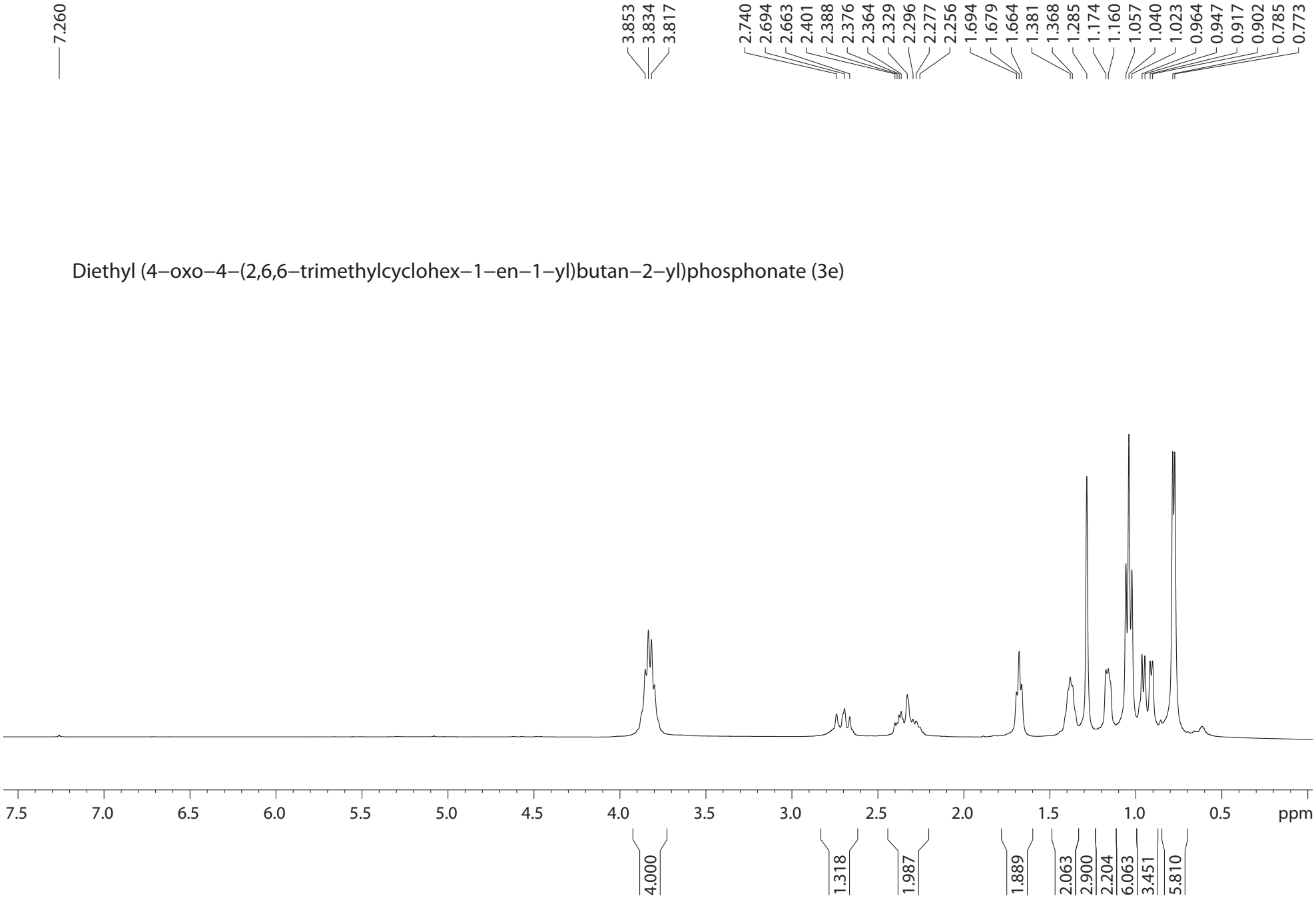

196.135
195.982
136.290
134.575
134.506
133.374
133.026
130.525
130.460
128.599
127.987
77.319
77.001
76.684
62.991
62.924
62.113
62.044
39.056
38.922
37.654
16.336
16.278
16.205
16.149
Diethyl (1−(4−chlorophenyl)−3−oxo−3−phenylpropyl)phosphonate (3c)
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm


31.964
Diethyl (1–(4–chlorophenyl)–3–oxo–3–phenylpropyl)phosphonate (3c)
150
100
50
0
–50
–100
–150
ppm

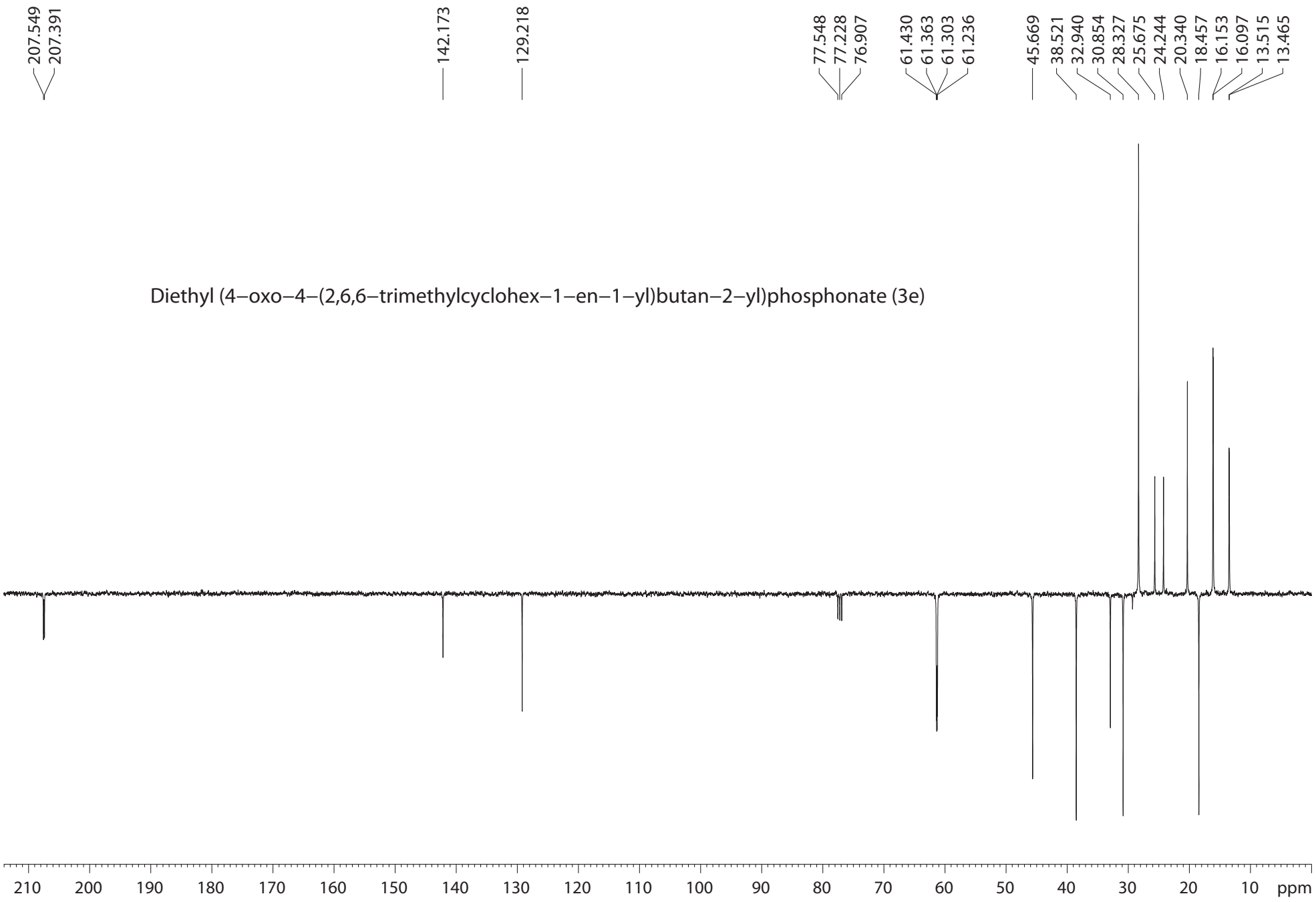

7.943
7.924
7.563
7.545
7.527
7.455
7.436
7.417
7.360
7.355
7.339
7.260
6.835
6.814
4.107
4.089
4.080
4.070
4.062
4.052
4.045
4.036
4.027
3.949
3.939
3.922
3.915
3.904
3.896
3.879
3.872
3.861
3.757
3.741
3.721
3.699
3.677
3.663
3.654
3.635
3.625
3.609
3.591
3.581
1.296
1.278
1.260
1.119
1.101
1.084
Diethyl (1–(4–methoxyphenyl)–3–oxo–3–phenylpropyl)phosphonate (3d)
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
2.000
1.052
2.097
2.032
1.965
2.085
2.113
6.006
3.993
3.126

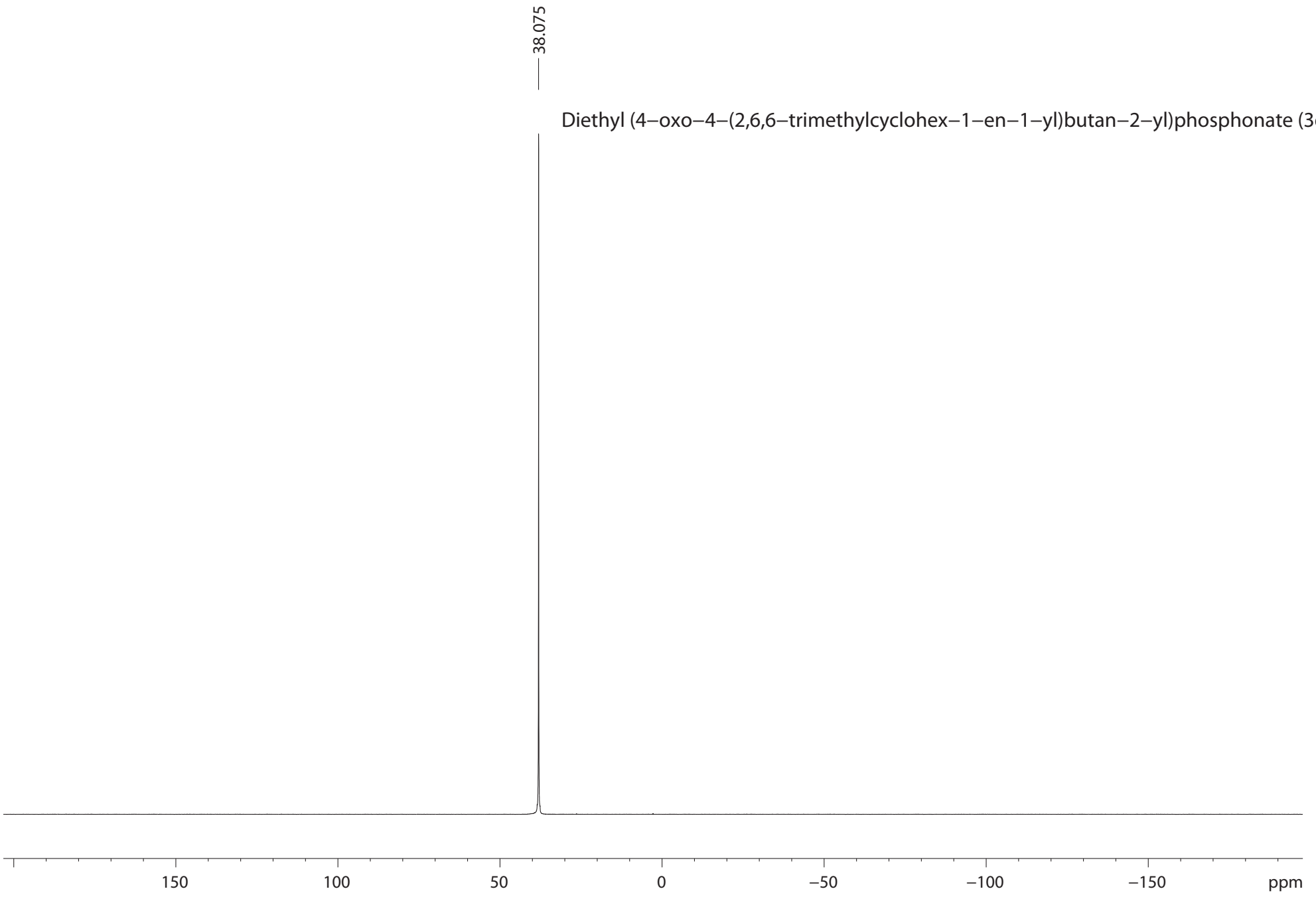

196.518
196.366
158.631
136.463
133.204
130.191
130.126
128.523
128.002
127.668
127.599
113.840
77.385
77.066
76.747
62.919
62.850
61.903
61.832
55.066
39.095
38.688
37.283
16.333
16.274
16.207
16.151
Diethyl (1−(4−methoxyphenyl)−3−oxo−3−phenylpropyl)phosphonate (3d)
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

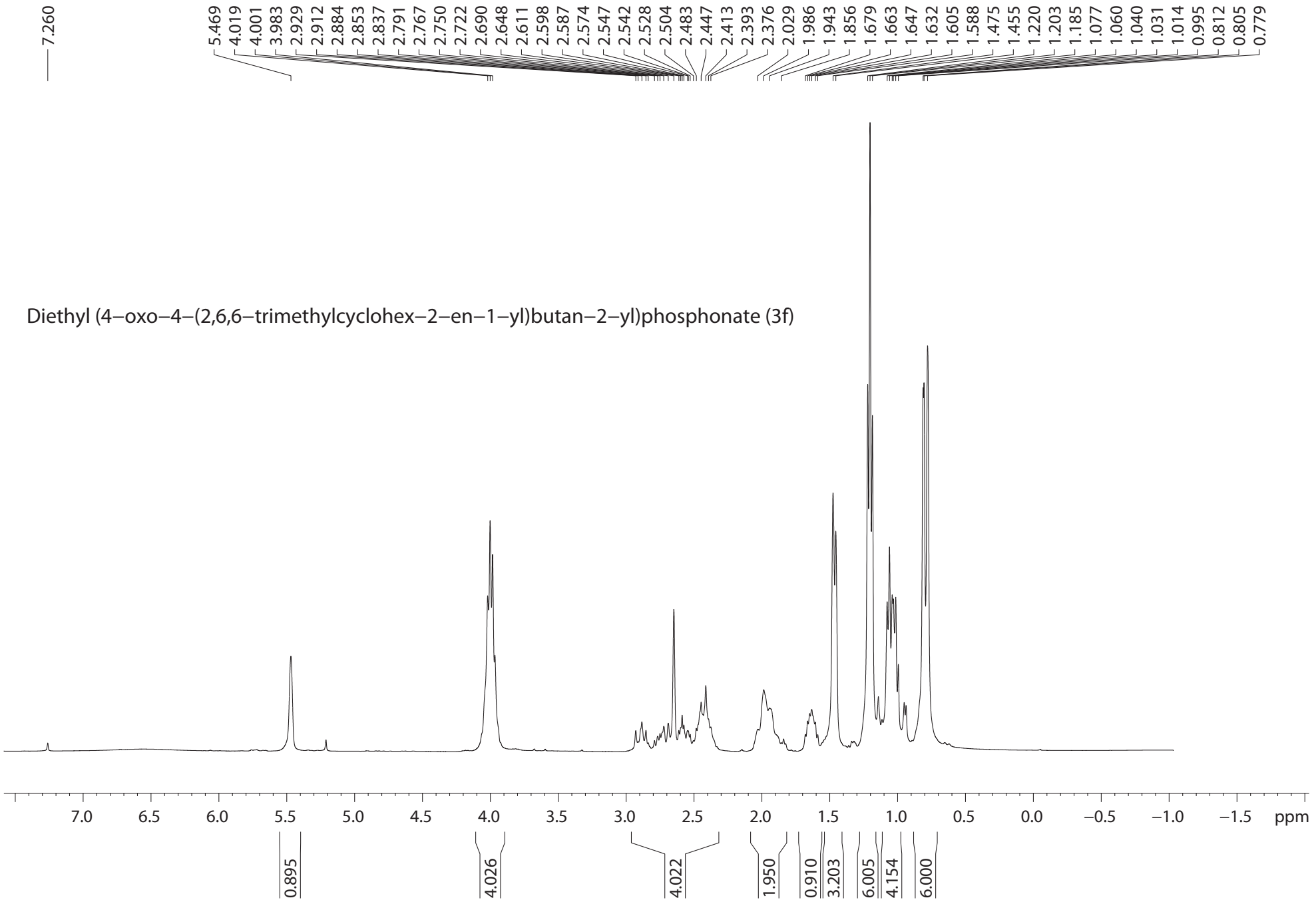

32.931
Diethyl (1–(4–methoxyphenyl)–3–oxo–3–phenylpropyl)phosphonate (3d)
150
100
50
0
–50
–100
–150
ppm

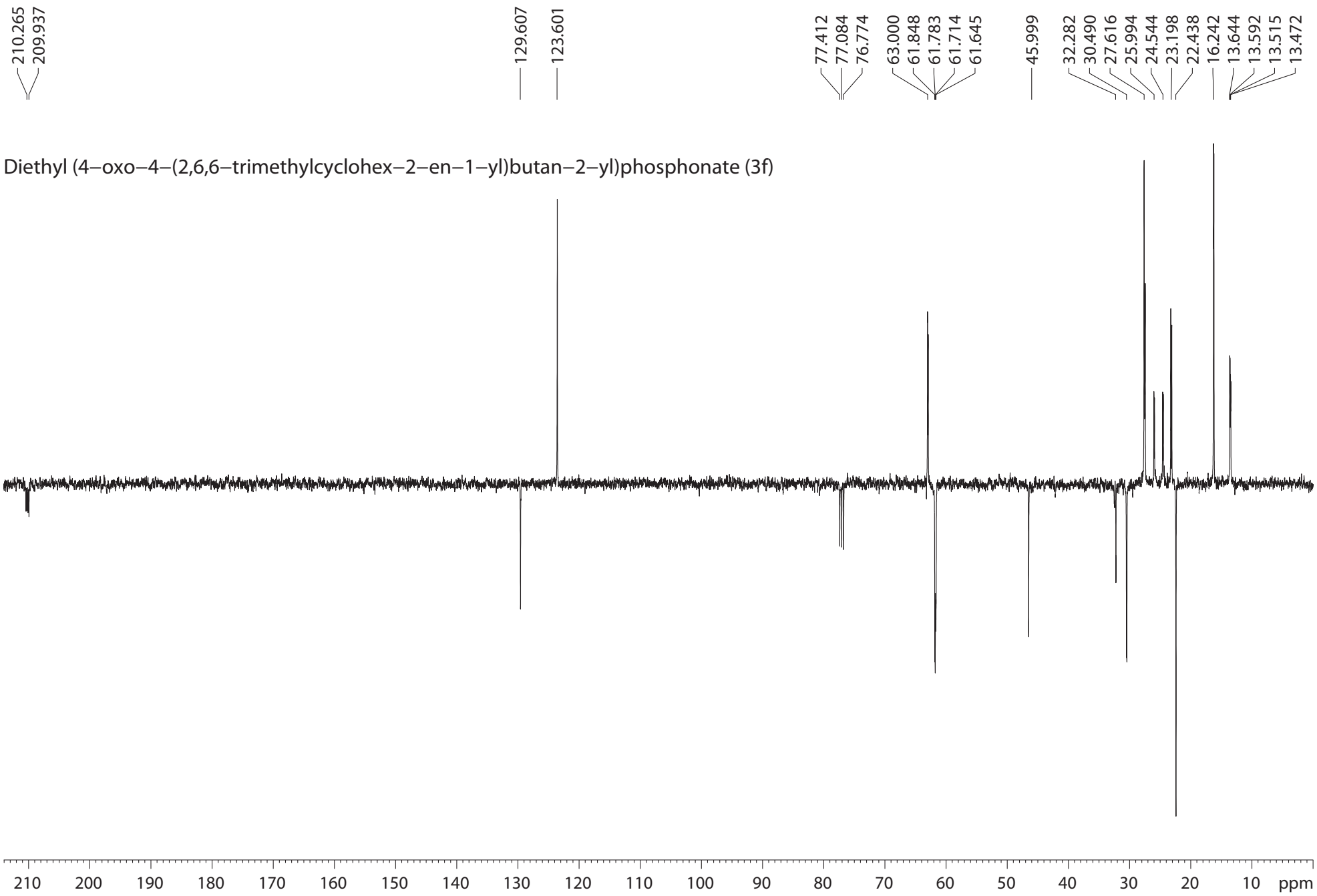


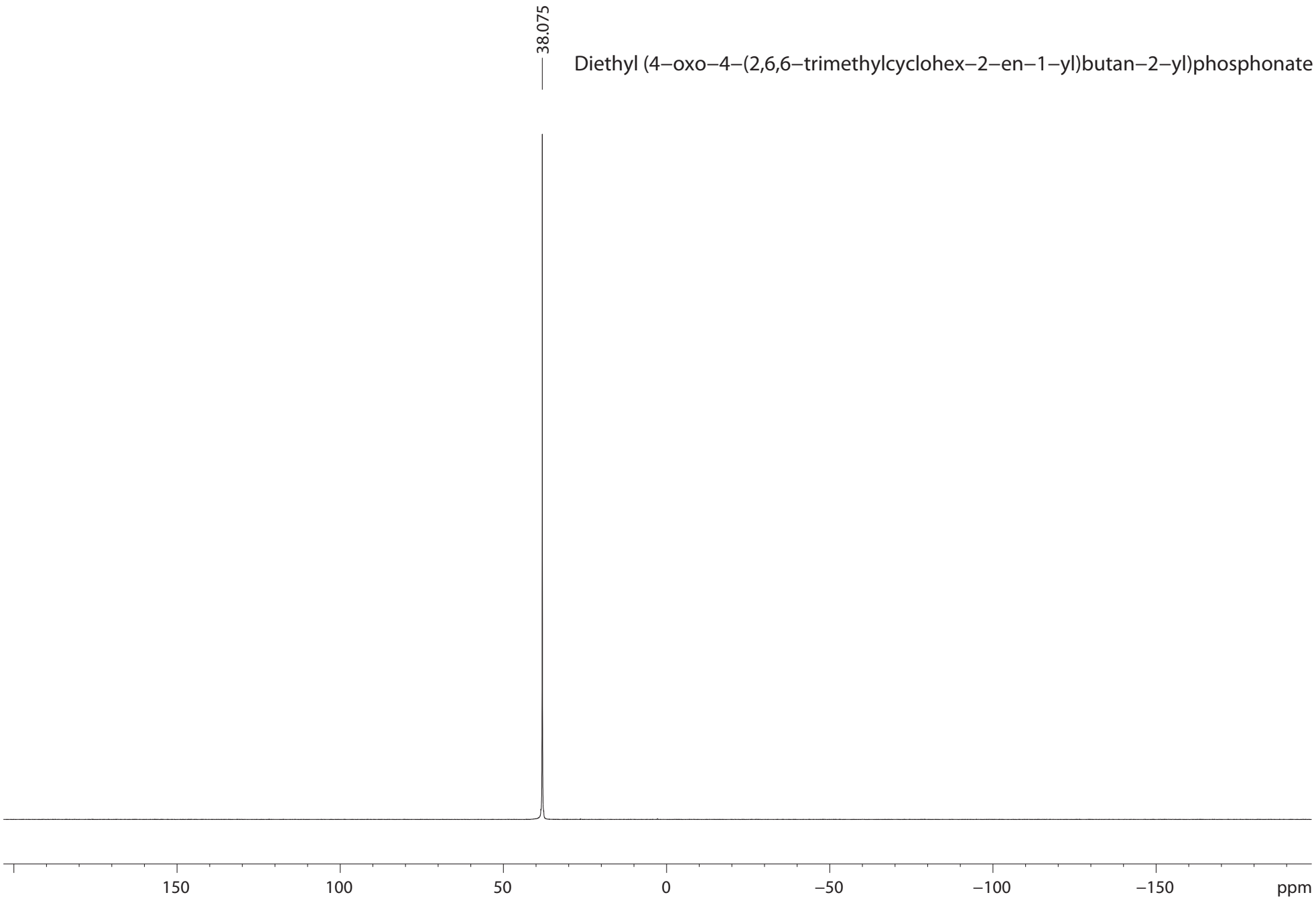
Diethyl (4–oxo–4–(2,6,6–trimethylcyclohex–1–en–1–yl)butan–2–yl)phosphonate (3e)

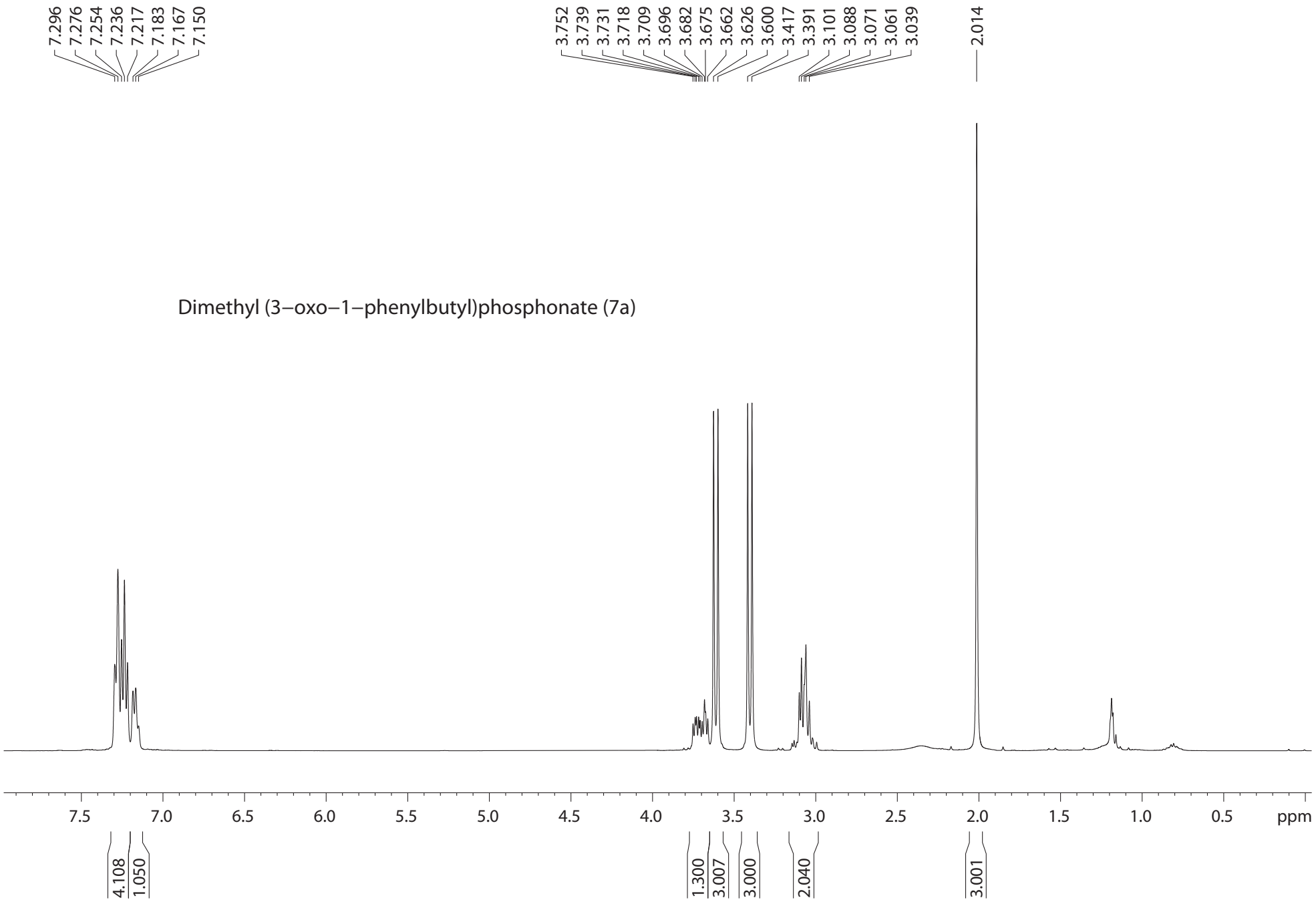

207.549
207.391
142.173
129.218
77.548
77.228
76.907
61.430
61.363
61.303
61.236
45.669
38.521
32.940
30.854
28.327
25.675
24.244
20.340
18.457
16.153
16.097
13.515
13.465
Diethyl (4–oxo–4–(2,6,6–trimethylcyclohex–1–en–1–yl)butan–2–yl)phosphonate (3e)
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

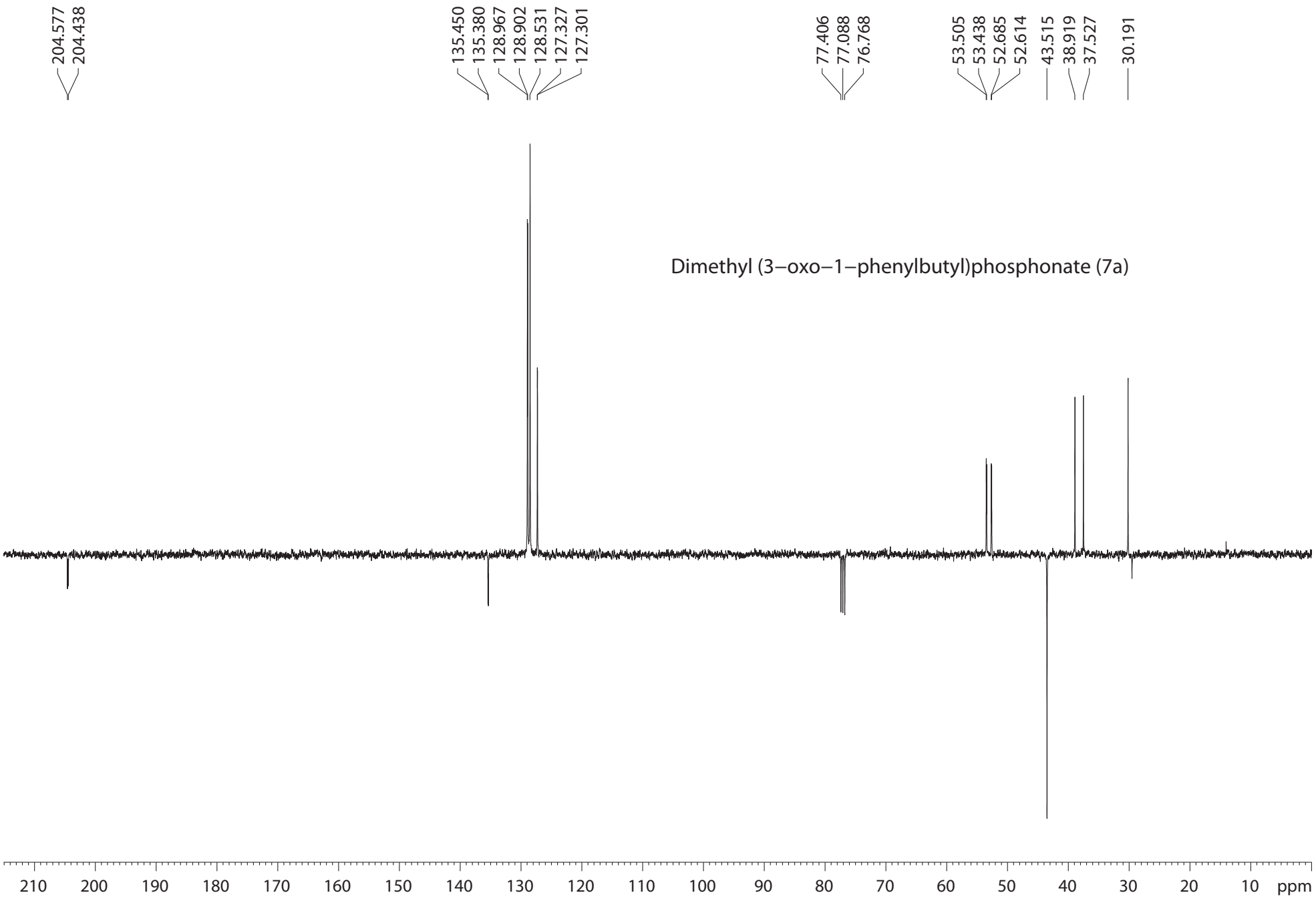

38.075
Diethyl (4–oxo–4–(2,6,6–trimethylcyclohex–1–en–1–yl)butan–2–yl)phosphonate (3e)
150
100
50
0
–50
–100
–150
ppm

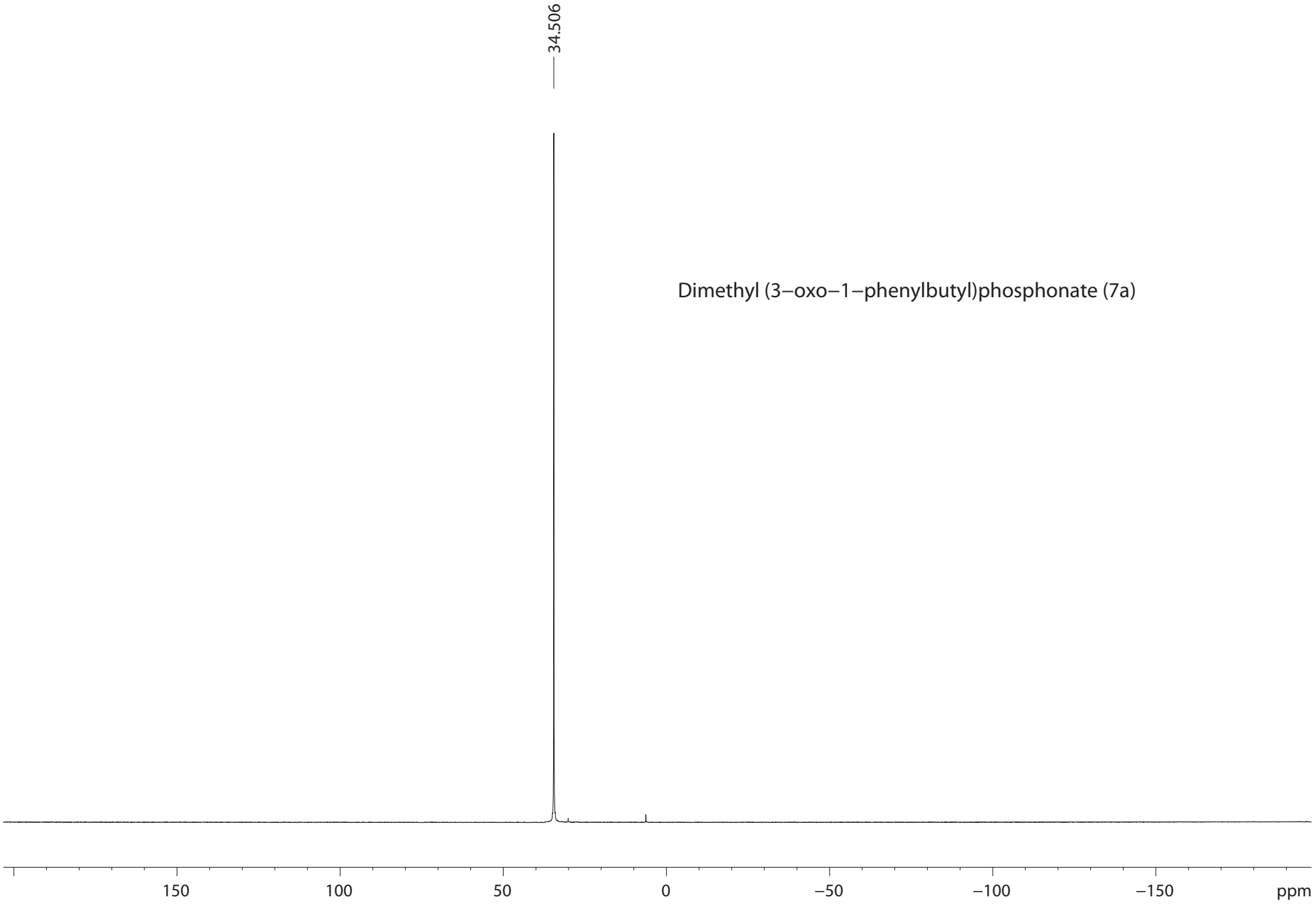

7.260
5.469
4.019
4.001
3.983
2.929
2.912
2.884
2.853
2.837
2.791
2.767
2.750
2.722
2.690
2.648
2.611
2.598
2.587
2.574
2.547
2.542
2.528
2.504
2.483
2.447
2.413
2.393
2.376
2.029
1.986
1.943
1.856
1.679
1.663
1.647
1.632
1.605
1.588
1.475
1.455
1.220
1.203
1.185
1.077
1.060
1.040
1.031
1.014
0.995
0.812
0.805
0.779
Diethyl (4–oxo–4–(2,6,6–trimethylcyclohex–2–en–1–yl)butan–2–yl)phosphonate (3f)
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
–0.5
–1.0
–1.5
ppm
0.895
4.026
4.022
1.950
0.910
3.203
6.005
4.154
6.000

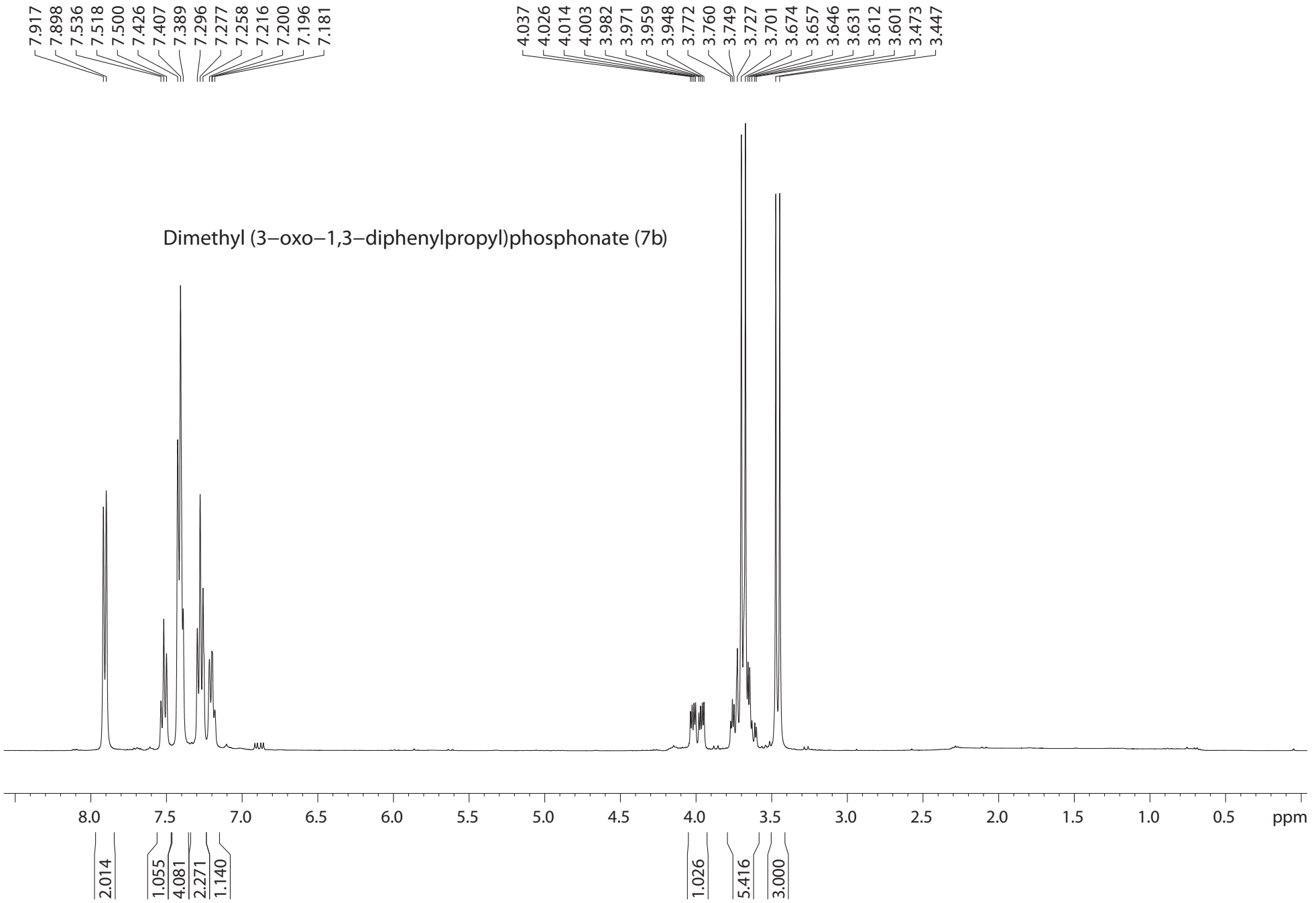

210.265
209.937
129.607
123.601
77.412
77.084
76.774
63.000
61.848
61.783
61.714
61.645
45.999
32.282
30.490
27.616
25.994
24.544
23.198
22.438
16.242
13.644
13.592
13.515
13.472
Diethyl (4–oxo–4–(2,6,6–trimethylcyclohex–2–en–1–yl)butan–2–yl)phosphonate (3f)
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

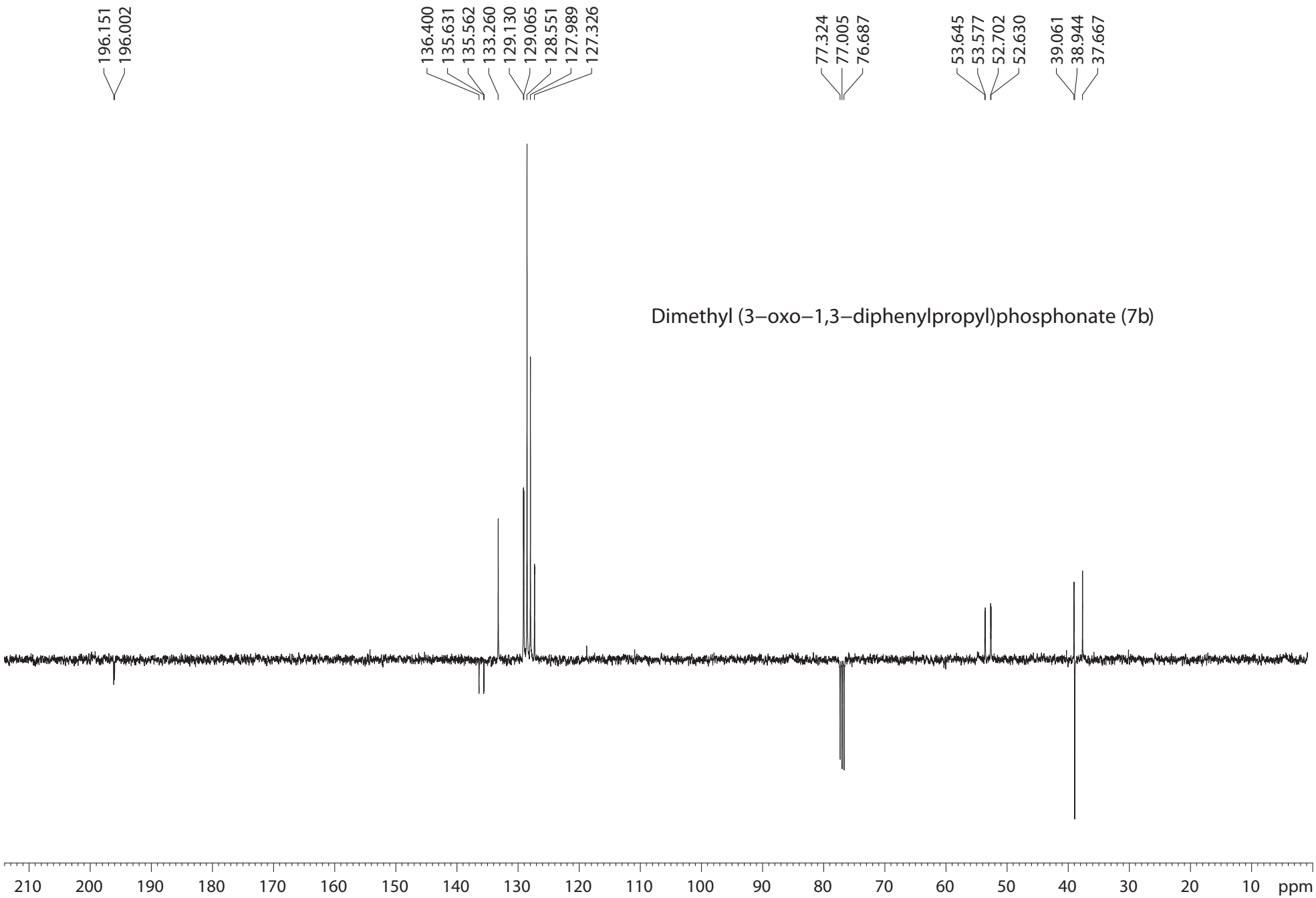

38.075
Diethyl (4–oxo–4–(2,6,6–trimethylcyclohex–2–en–1–yl)butan–2–yl)phosphonate (3f)
150
100
50
0
–50
–100
–150
ppm

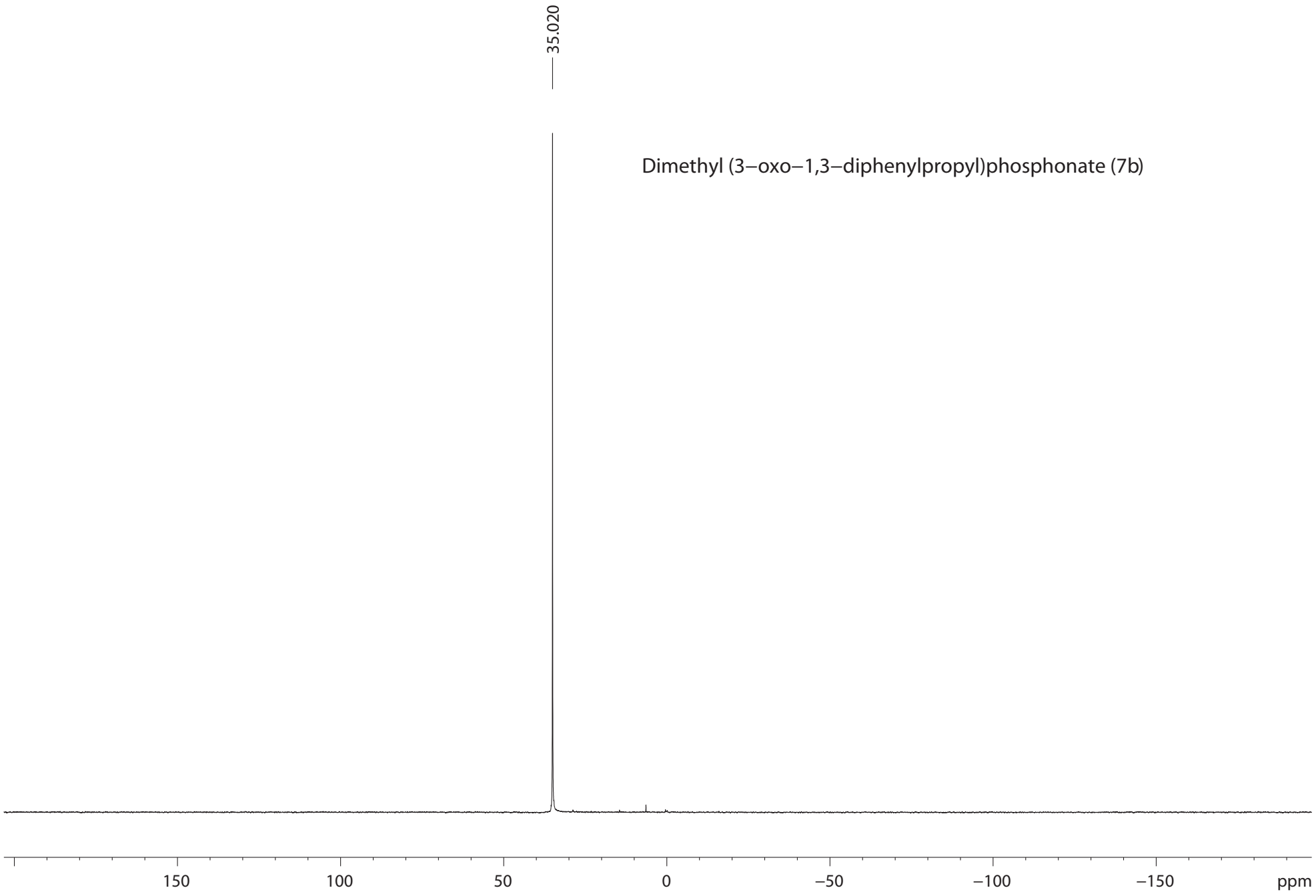

7.296
7.276
7.254
7.236
7.217
7.183
7.167
7.150
3.752
3.739
3.731
3.718
3.709
3.696
3.682
3.675
3.662
3.626
3.600
3.417
3.391
3.101
3.088
3.071
3.061
3.039
2.014
Dimethyl (3−oxo−1−phenylbutyl)phosphonate (7a)
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
4.108
1.050
1.300
3.007
3.000
2.040
3.001

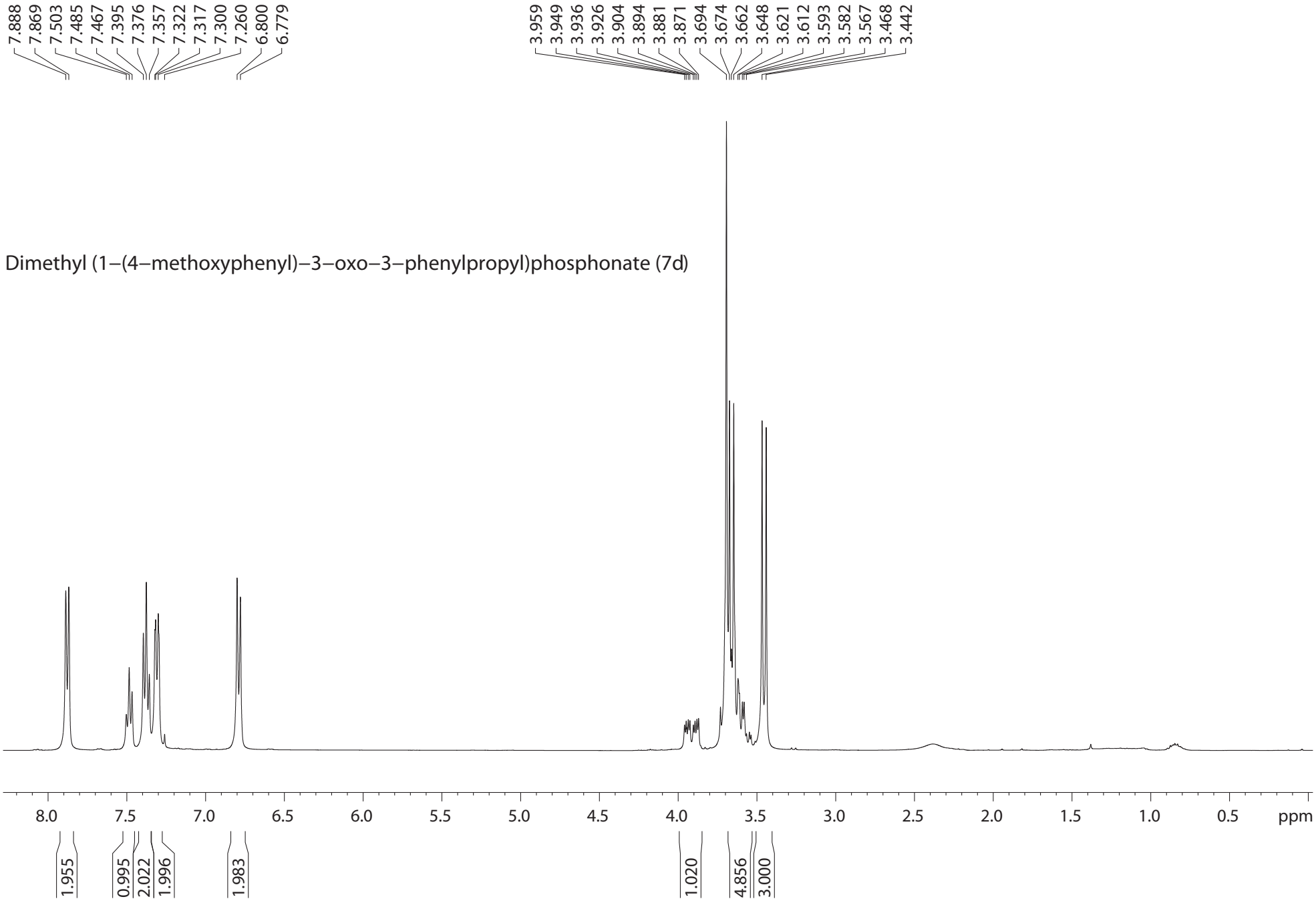

204.577
204.438
135.450
135.380
128.967
128.902
128.531
127.327
127.301
77.406
77.088
76.768
53.505
53.438
52.685
52.614
43.515
38.919
37.527
30.191
Dimethyl (3−oxo−1−phenylbutyl)phosphonate (7a)
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

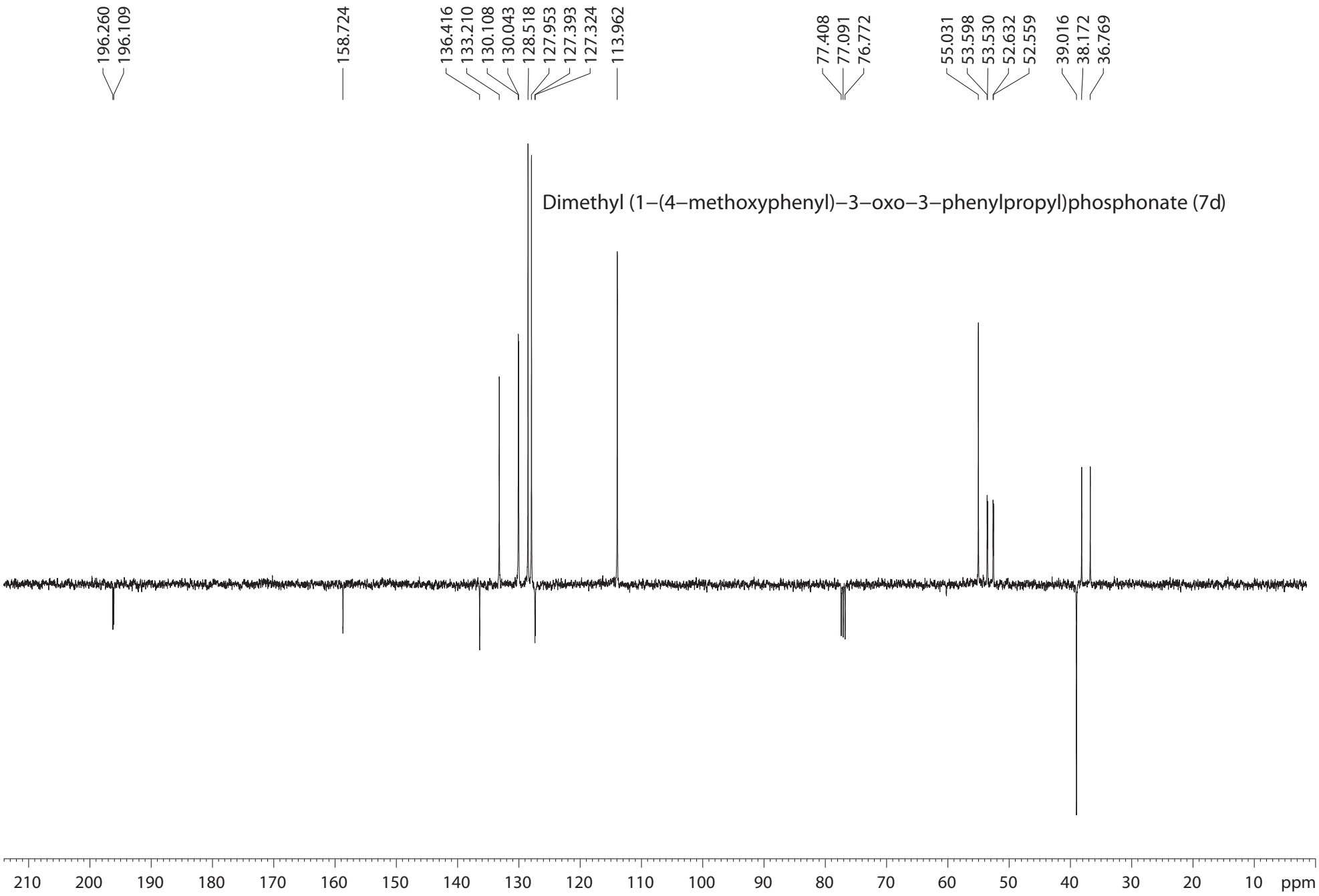

34.506
Dimethyl (3−oxo−1−phenylbutyl)phosphonate (7a)
150
100
50
0
−50
−100
−150
ppm

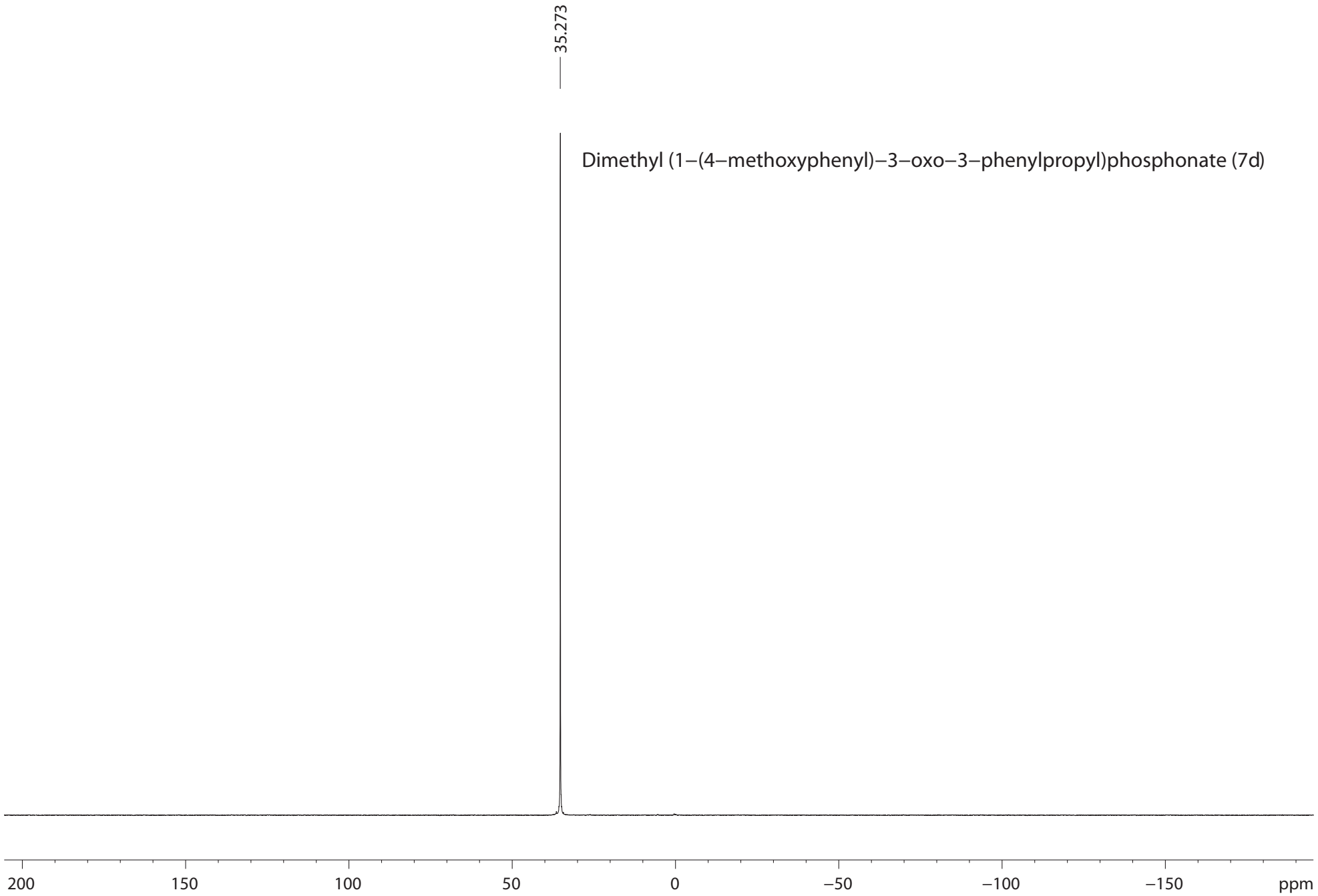

7.917
7.898
7.536
7.518
7.500
7.426
7.407
7.389
7.296
7.277
7.258
7.216
7.200
7.196
7.181
4.037
4.026
4.014
4.003
3.982
3.971
3.959
3.948
3.772
3.760
3.749
3.727
3.701
3.674
3.657
3.646
3.631
3.612
3.601
3.473
3.447
Dimethyl (3–oxo–1,3–diphenylpropyl)phosphonate (7b)
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
2.014
1.055
4.081
2.271
1.140
1.026
5.416
3.000

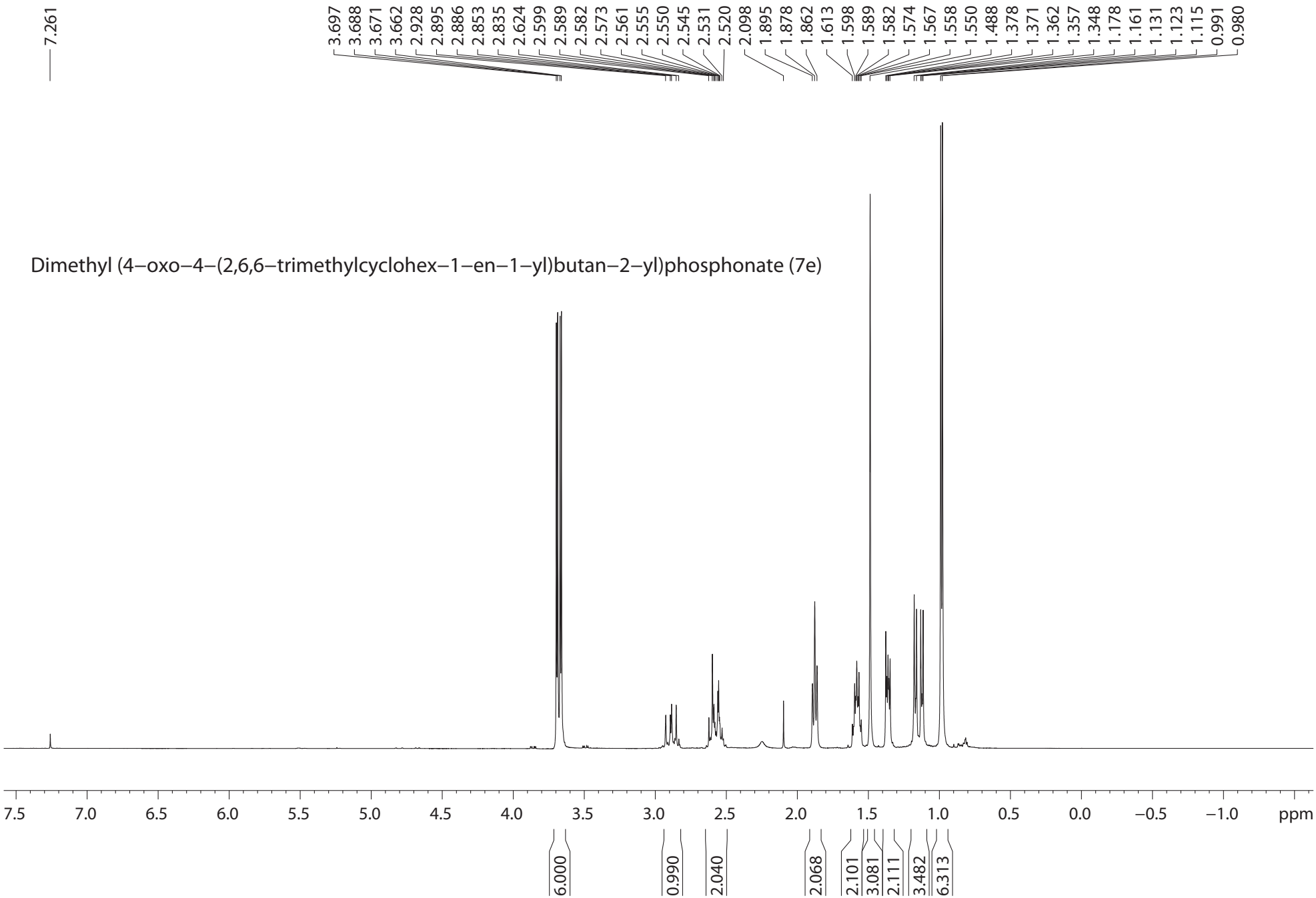

196.151
196.002
136.400
135.631
135.562
133.260
129.130
129.065
128.551
127.989
127.326
77.324
77.005
76.687
53.645
53.577
52.702
52.630
39.061
38.944
37.667
Dimethyl (3–oxo–1,3–diphenylpropyl)phosphonate (7b)
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

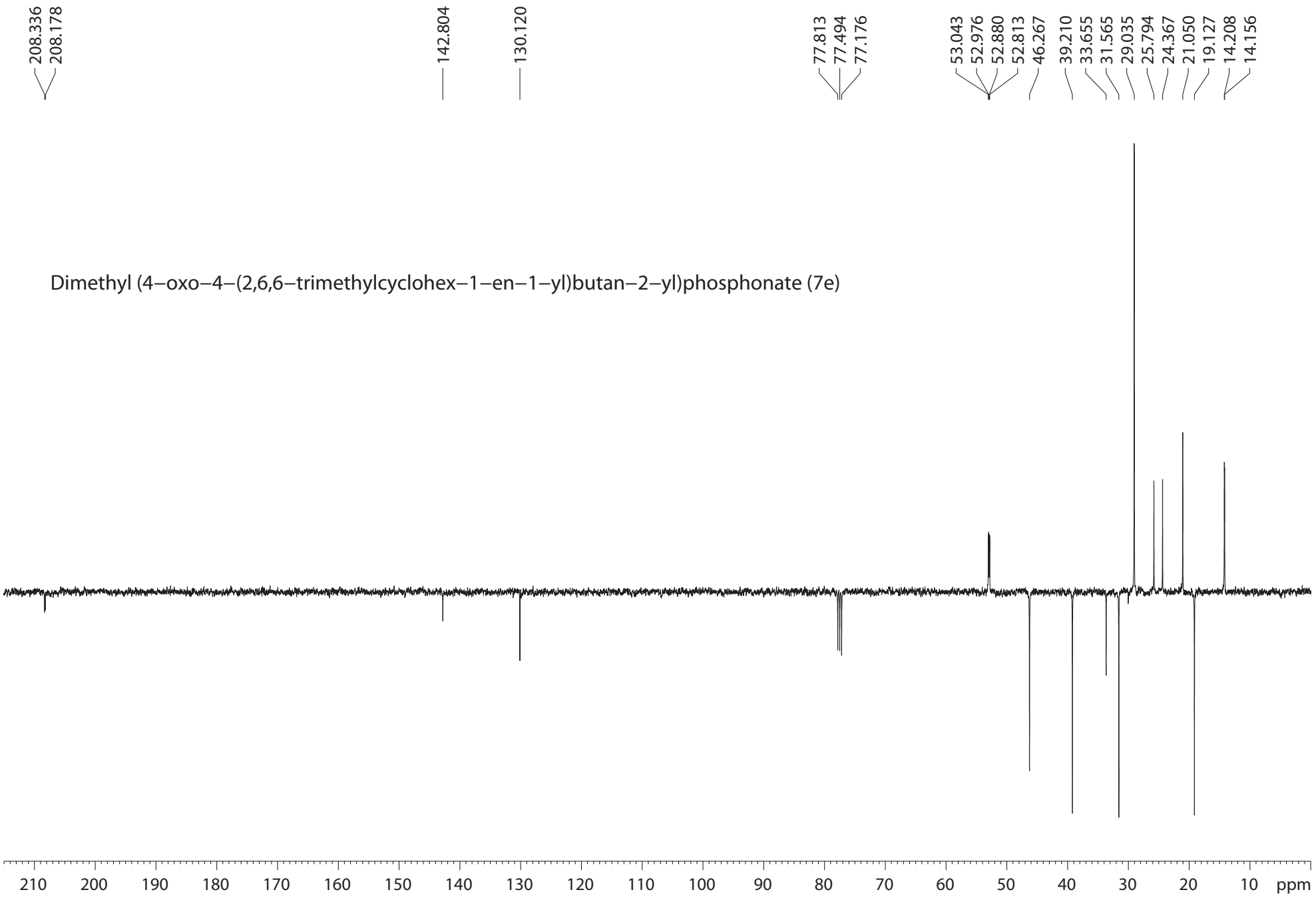

35.020
Dimethyl (3–oxo–1,3–diphenylpropyl)phosphonate (7b)
150
100
50
0
–50
–100
–150
ppm

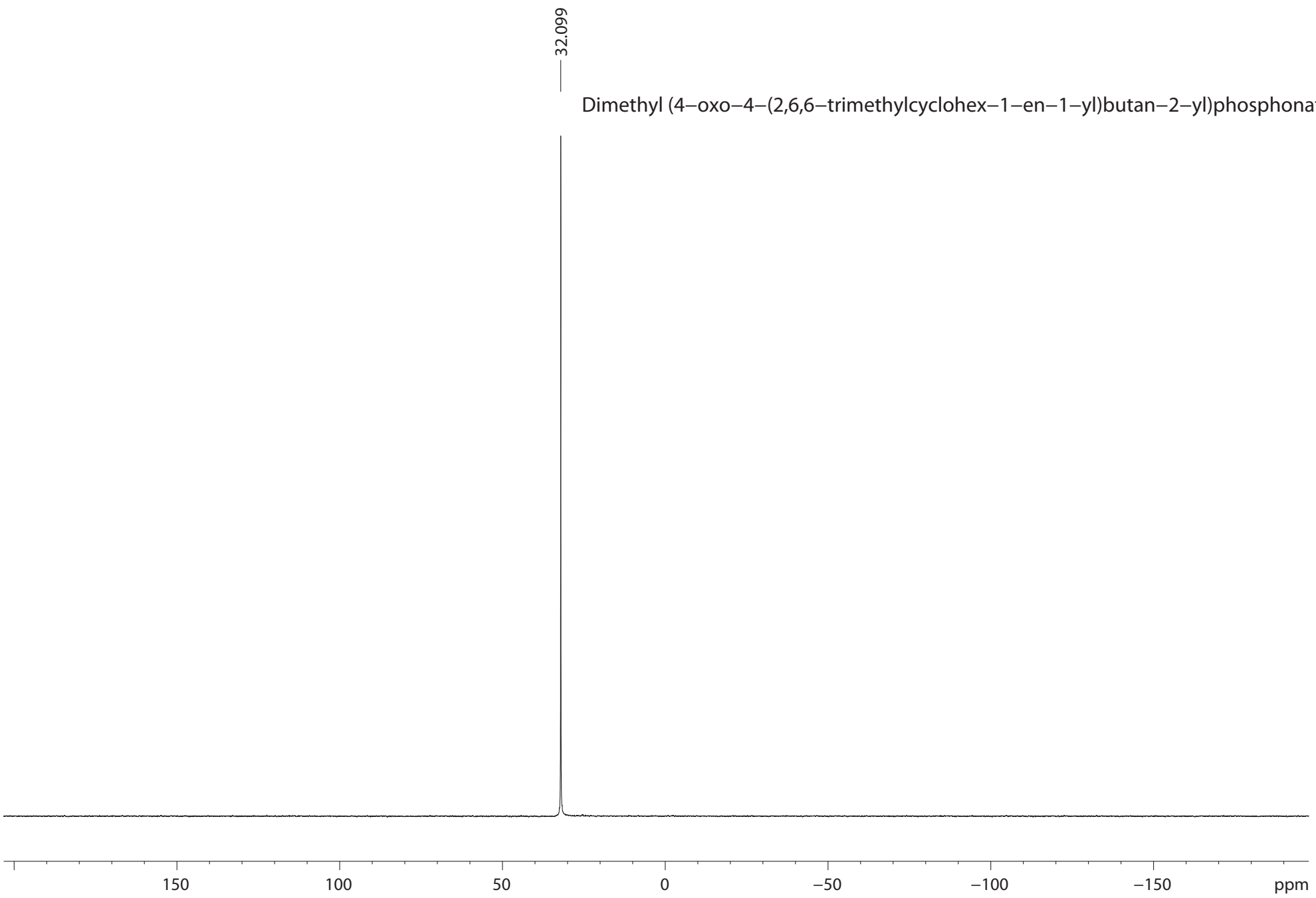

7.888
7.869
7.503
7.485
7.467
7.395
7.376
7.357
7.322
7.317
7.300
7.260
6.800
6.779
3.959
3.949
3.936
3.926
3.904
3.894
3.881
3.871
3.694
3.674
3.662
3.648
3.621
3.612
3.593
3.582
3.567
3.468
3.442
Dimethyl (1−(4−methoxyphenyl)−3−oxo−3−phenylpropyl)phosphonate (7d)
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
1.955
0.995
2.022
1.996
1.983
1.020
4.856
3.000

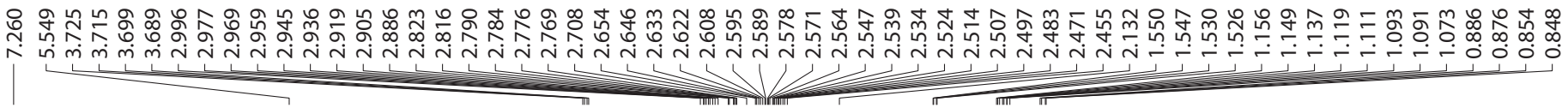

196.260
196.109
158.724
136.416
133.210
130.108
130.043
128.518
127.953
127.393
127.324
113.962
77.408
77.091
76.772
55.031
53.598
53.530
52.632
52.559
39.016
38.172
36.769
Dimethyl (1–(4–methoxyphenyl)–3–oxo–3–phenylpropyl)phosphonate (7d)
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm


35.273
Dimethyl (1–(4–methoxyphenyl)–3–oxo–3–phenylpropyl)phosphonate (7d)
200
150
100
50
0
–50
–100
–150
ppm

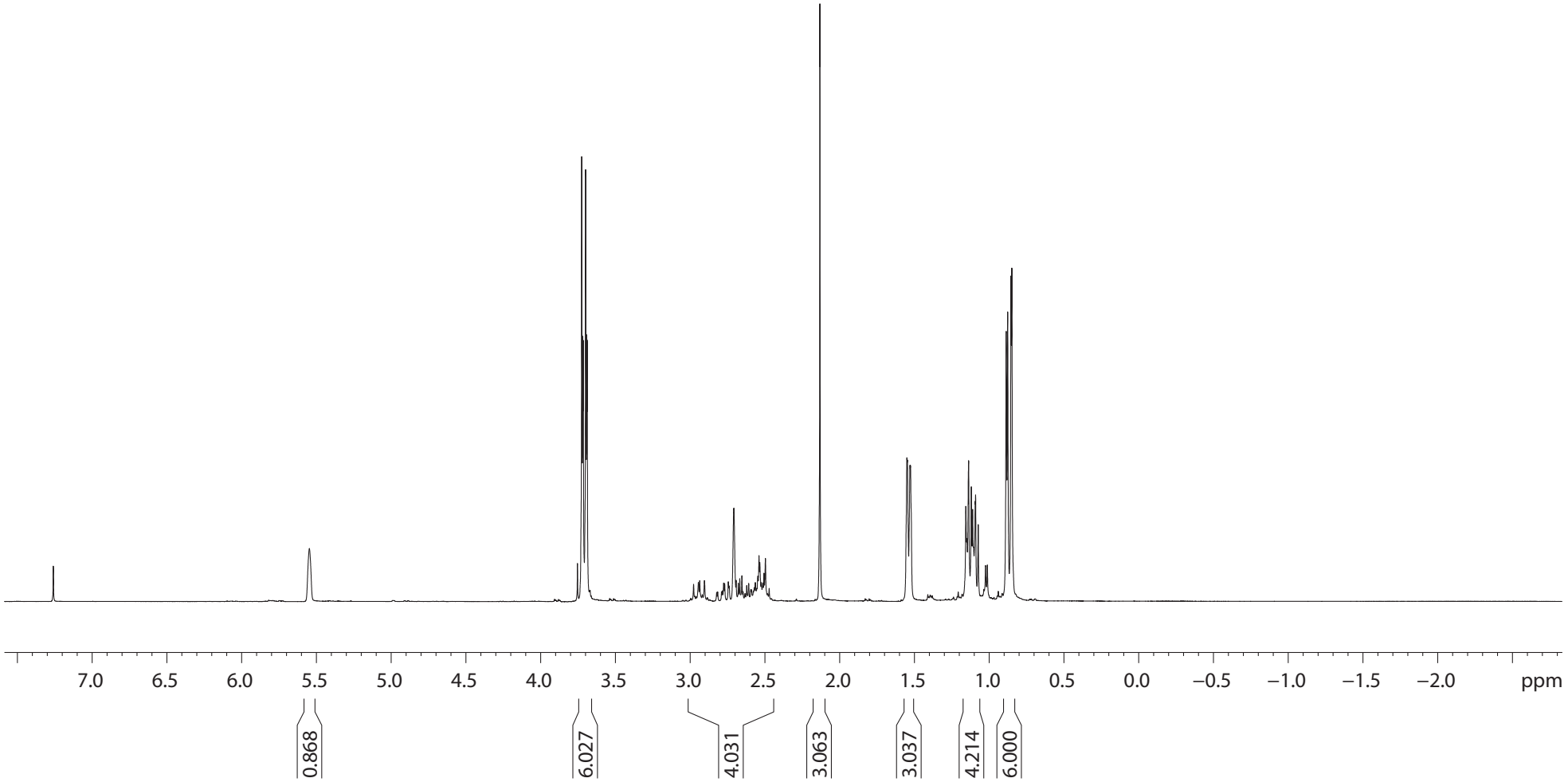

7.261
3.697
3.688
3.671
3.662
2.928
2.895
2.886
2.853
2.835
2.624
2.599
2.589
2.582
2.573
2.561
2.555
2.550
2.545
2.531
2.520
2.098
1.895
1.878
1.862
1.613
1.598
1.589
1.582
1.574
1.567
1.558
1.550
1.488
1.378
1.371
1.362
1.357
1.348
1.178
1.161
1.131
1.123
1.115
0.991
0.980
Dimethyl (4–oxo–4–(2,6,6–trimethylcyclohex–1–en–1–yl)butan–2–yl)phosphonate (7e)
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
–0.5
–1.0
ppm
6.000
0.990
2.040
2.068
2.101
3.081
2.111
3.482
6.313

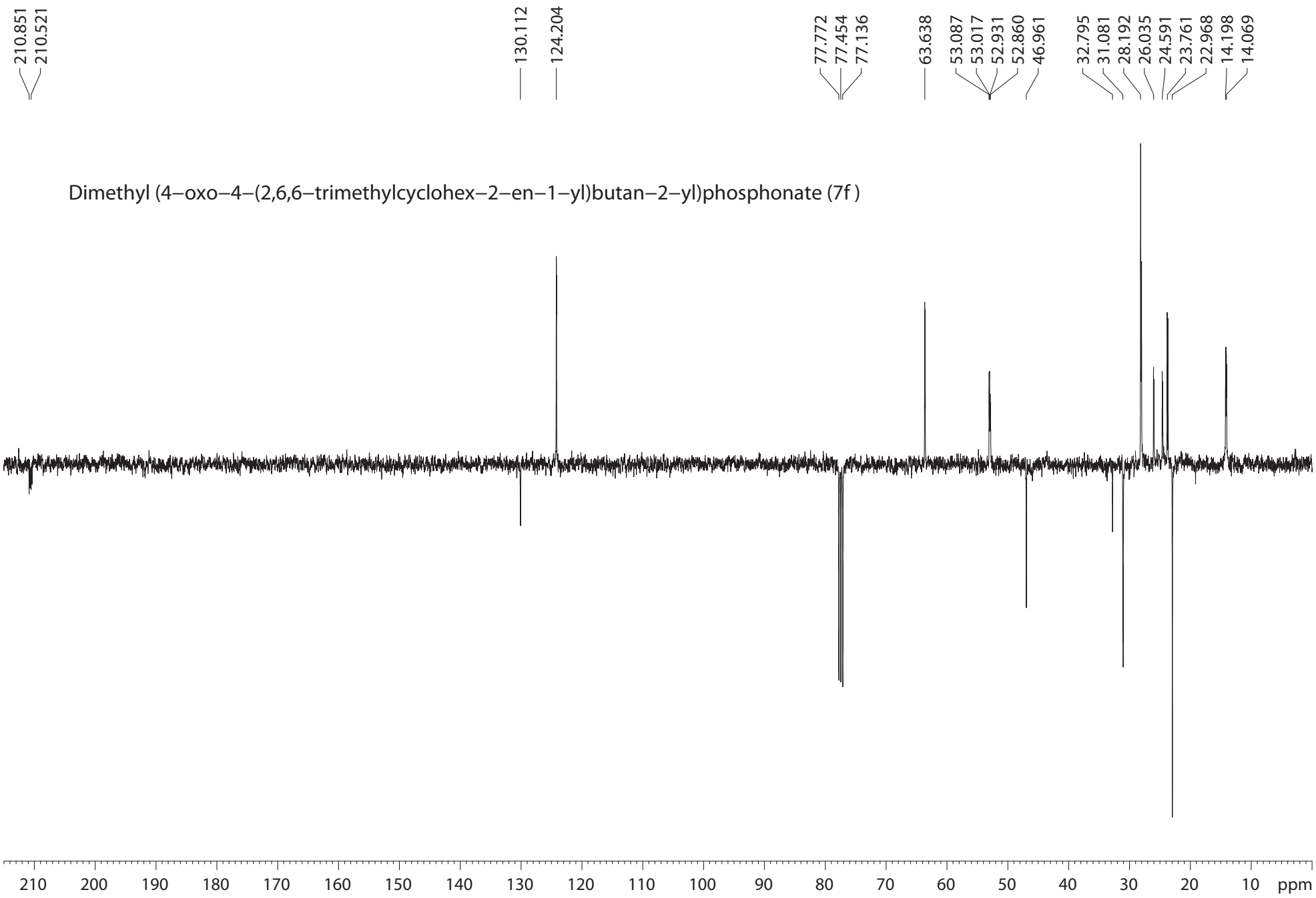

208.336
208.178
142.804
130.120
77.813
77.494
77.176
53.043
52.976
52.880
52.813
46.267
39.210
33.655
31.565
29.035
25.794
24.367
21.050
19.127
14.208
14.156
Dimethyl (4–oxo–4–(2,6,6–trimethylcyclohex–1–en–1–yl)butan–2–yl)phosphonate (7e)
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

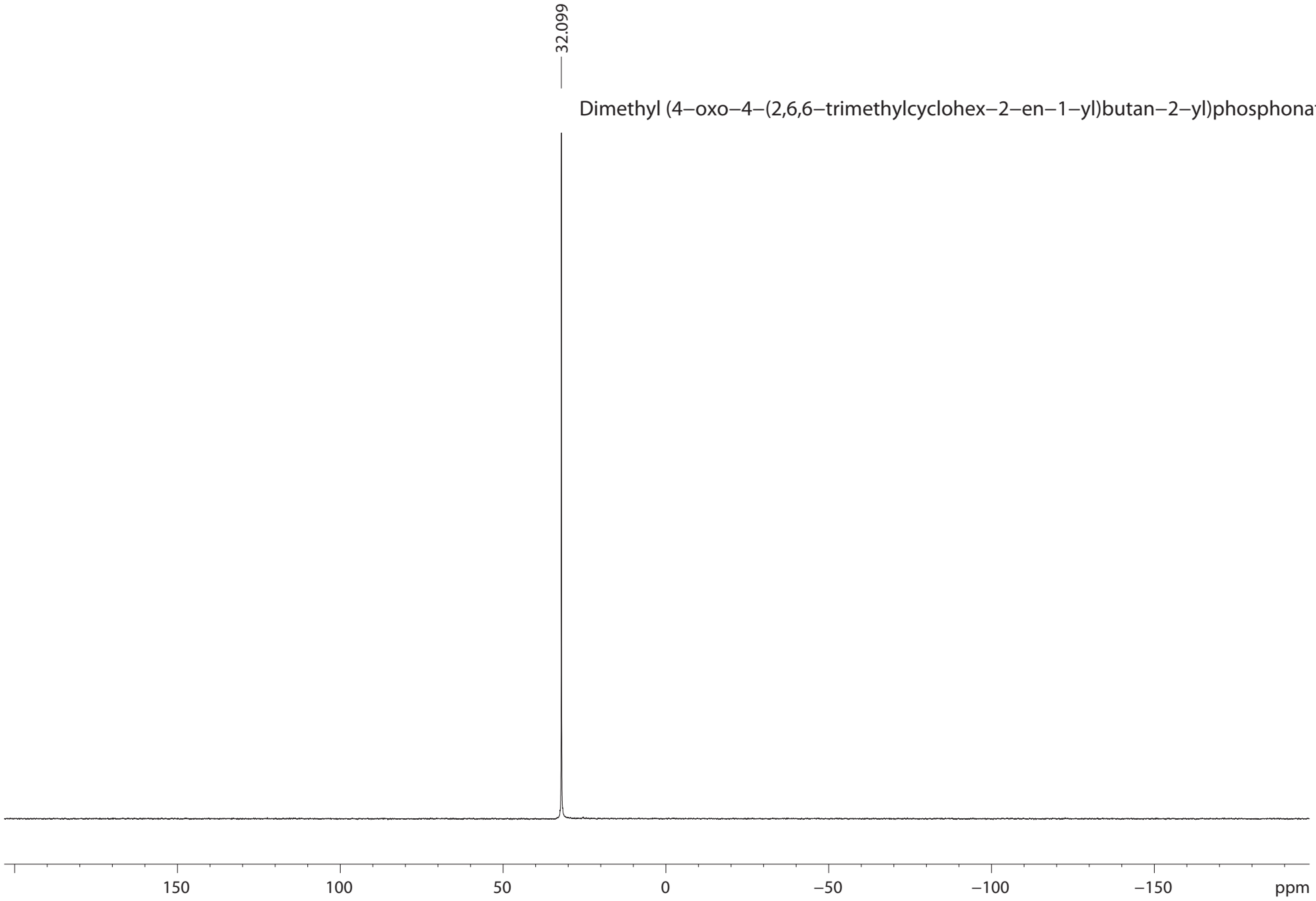

32.099
Dimethyl (4–oxo–4–(2,6,6–trimethylcyclohex–1–en–1–yl)butan–2–yl)phosphonate (7e)
150
100
50
0
–50
–100
–150
ppm

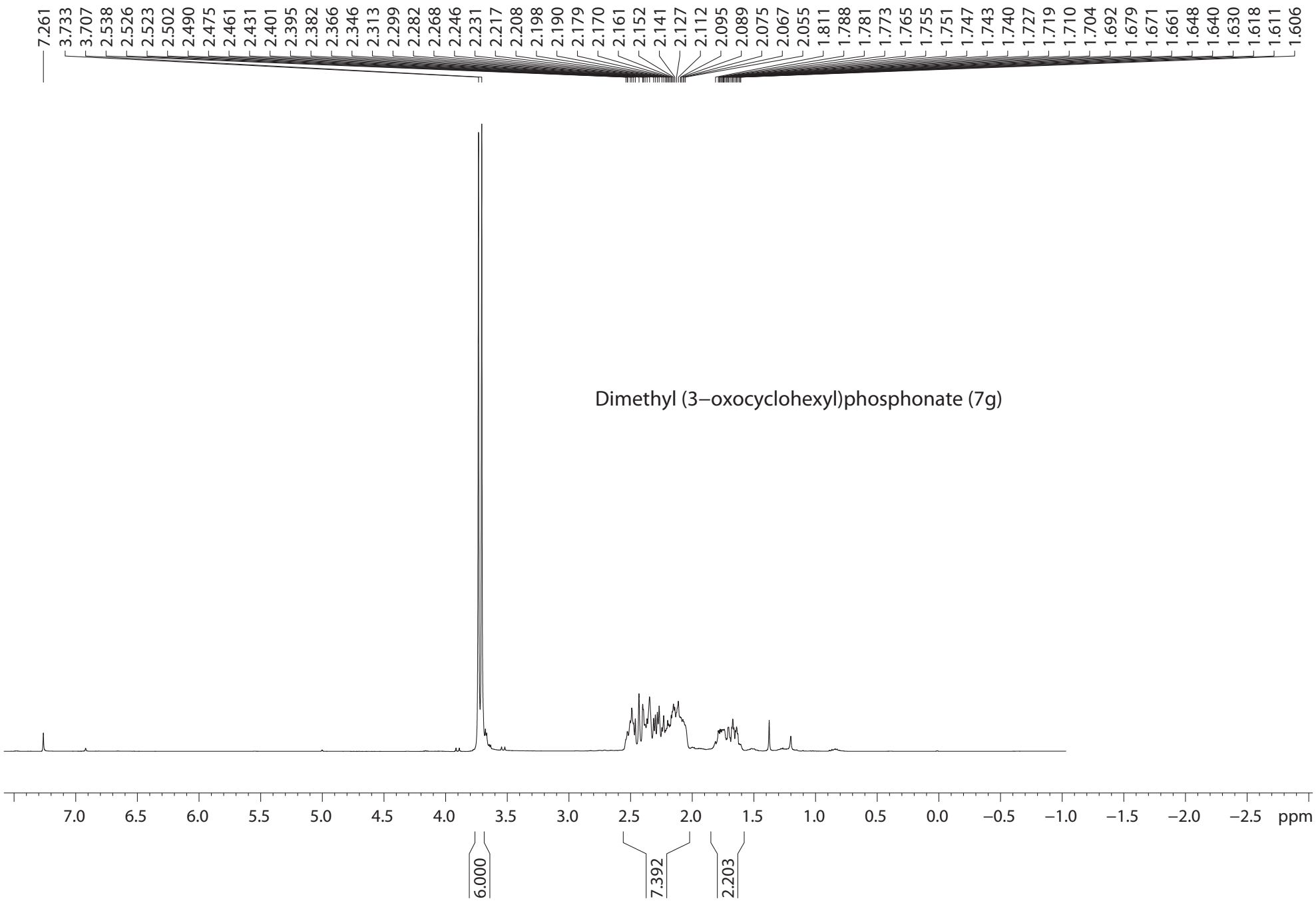


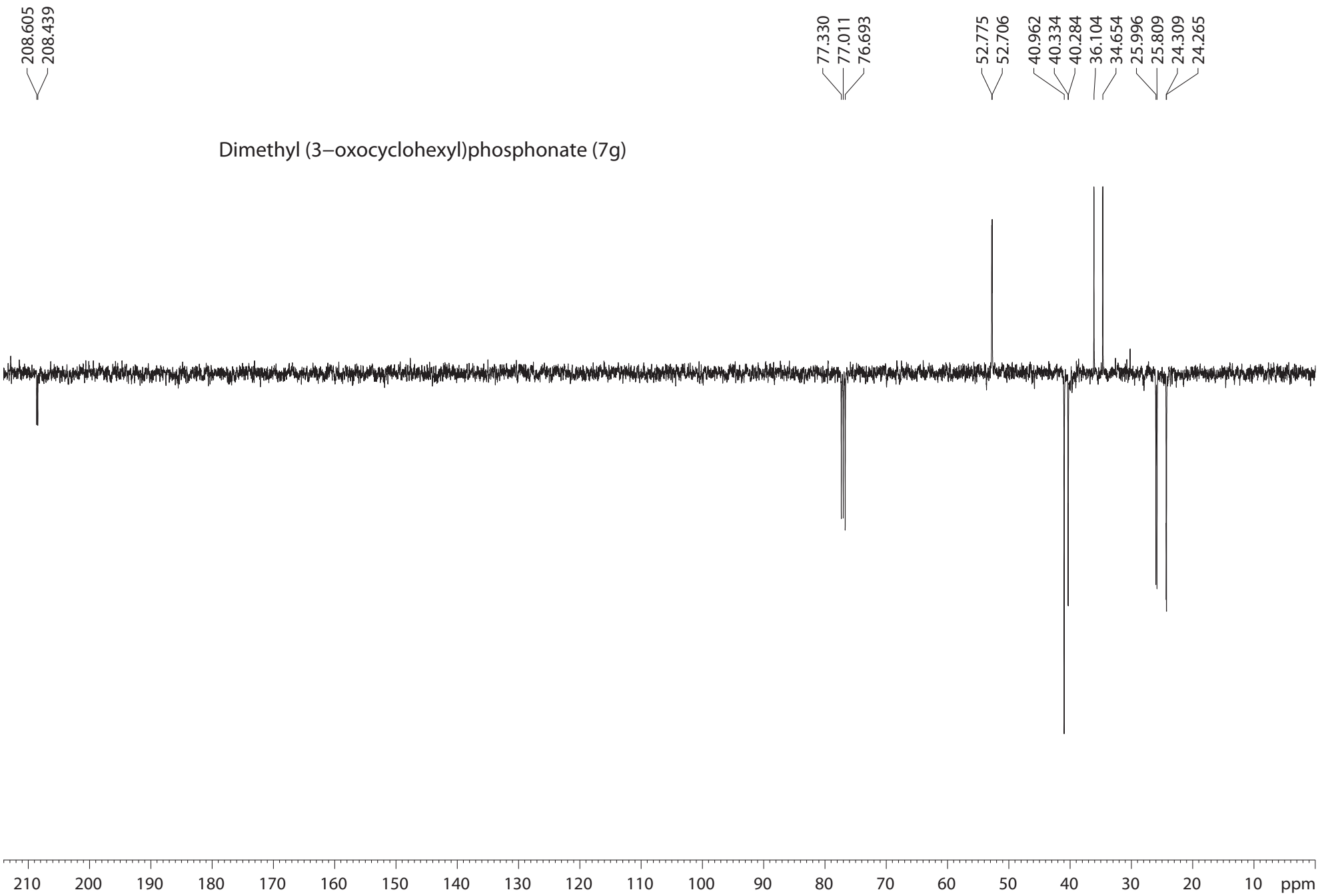
Dimethyl (4−oxo−4−(2,6,6−trimethylcyclohex−2−en−1−yl)butan−2−yl)phosphonate (7f)

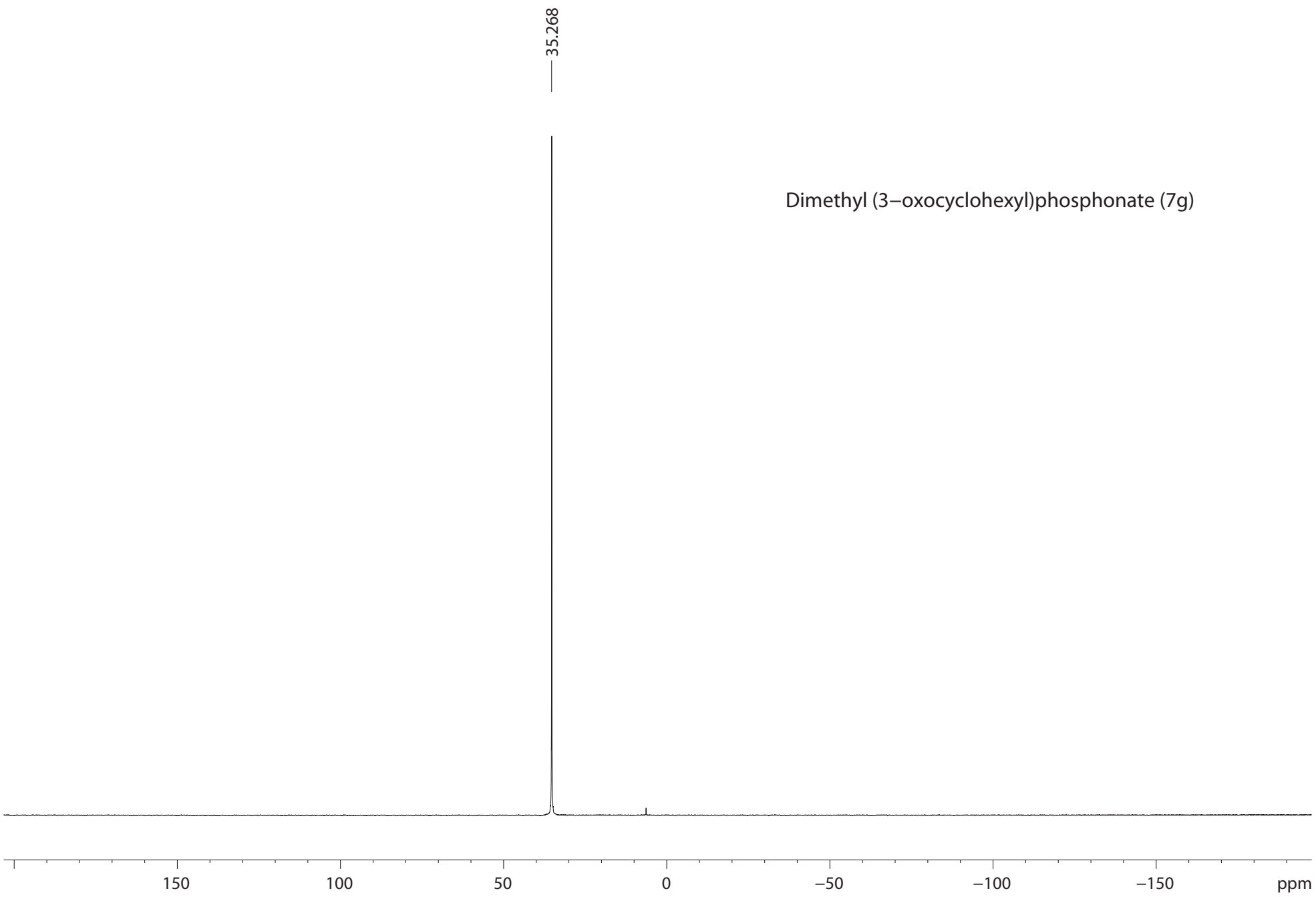


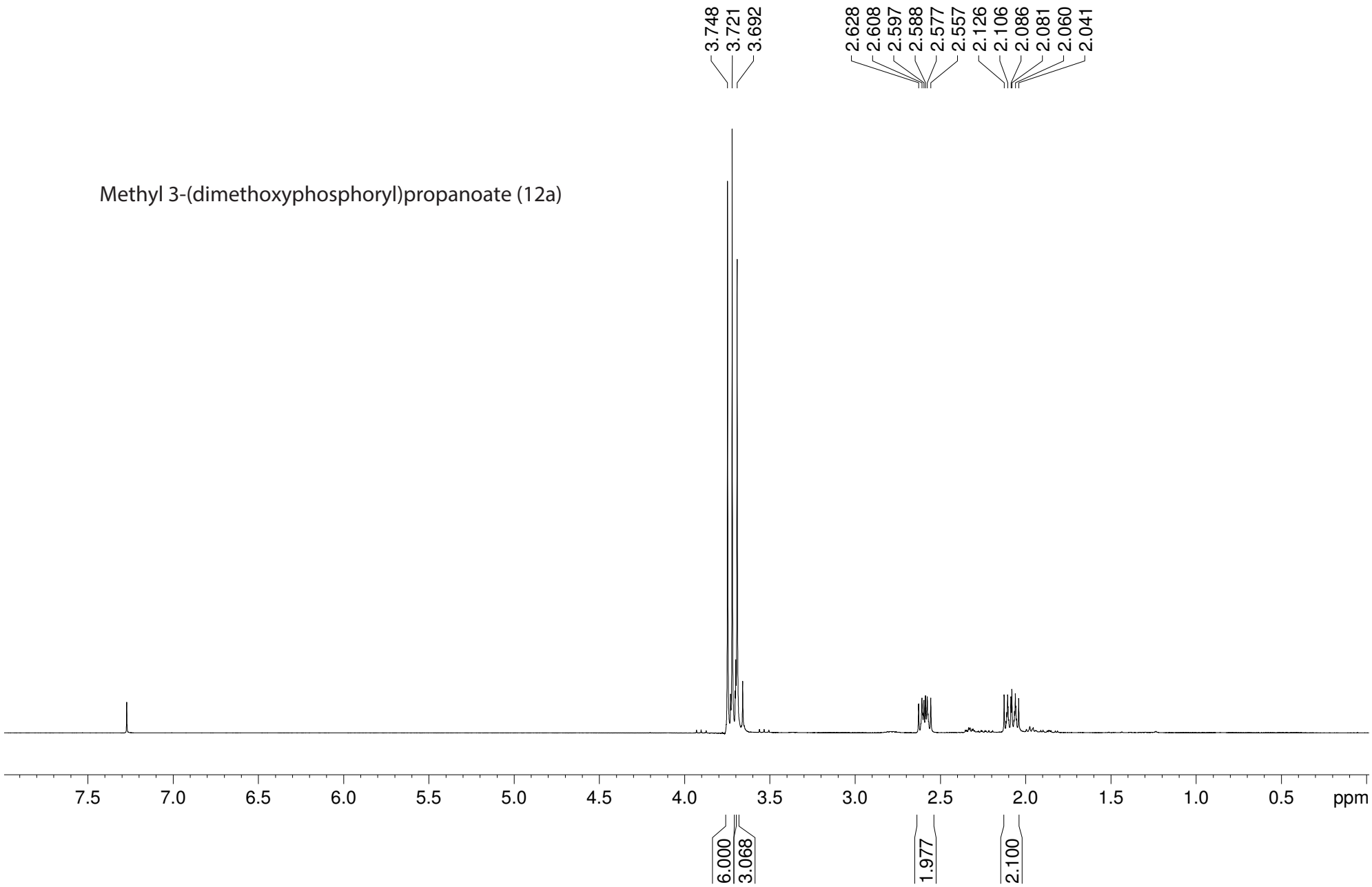

210.851
210.521
130.112
124.204
77.772
77.454
77.136
63.638
53.087
53.017
52.931
52.860
46.961
32.795
31.081
28.192
26.035
24.591
23.761
22.968
14.198
14.069
Dimethyl (4–oxo–4–(2,6,6–trimethylcyclohex–2–en–1–yl)butan–2–yl)phosphonate (7f)
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

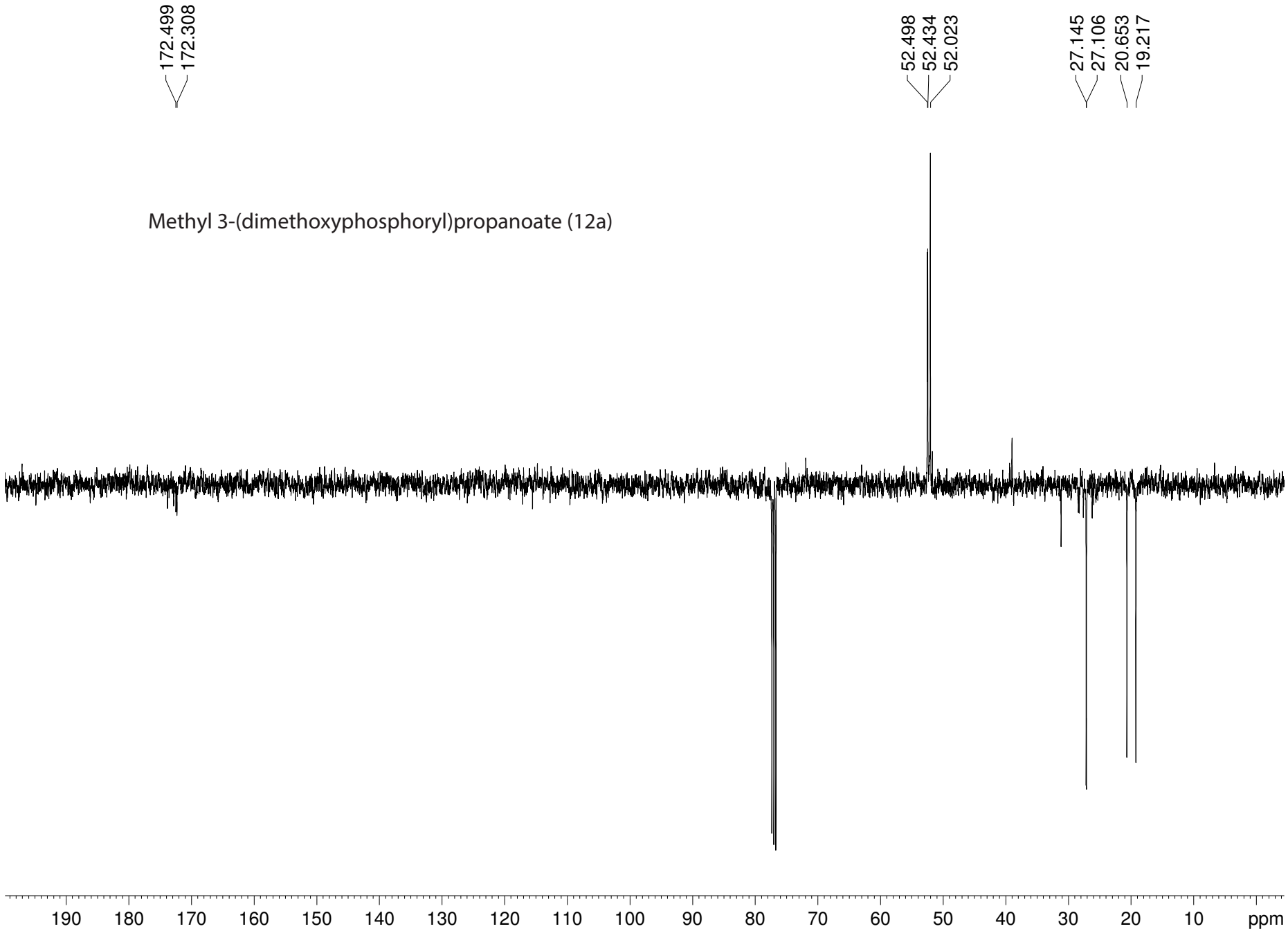

32.099
Dimethyl (4–oxo–4–(2,6,6–trimethylcyclohex–2–en–1–yl)butan–2–yl)phosphonate (7f)
150
100
50
0
–50
–100
–150
ppm

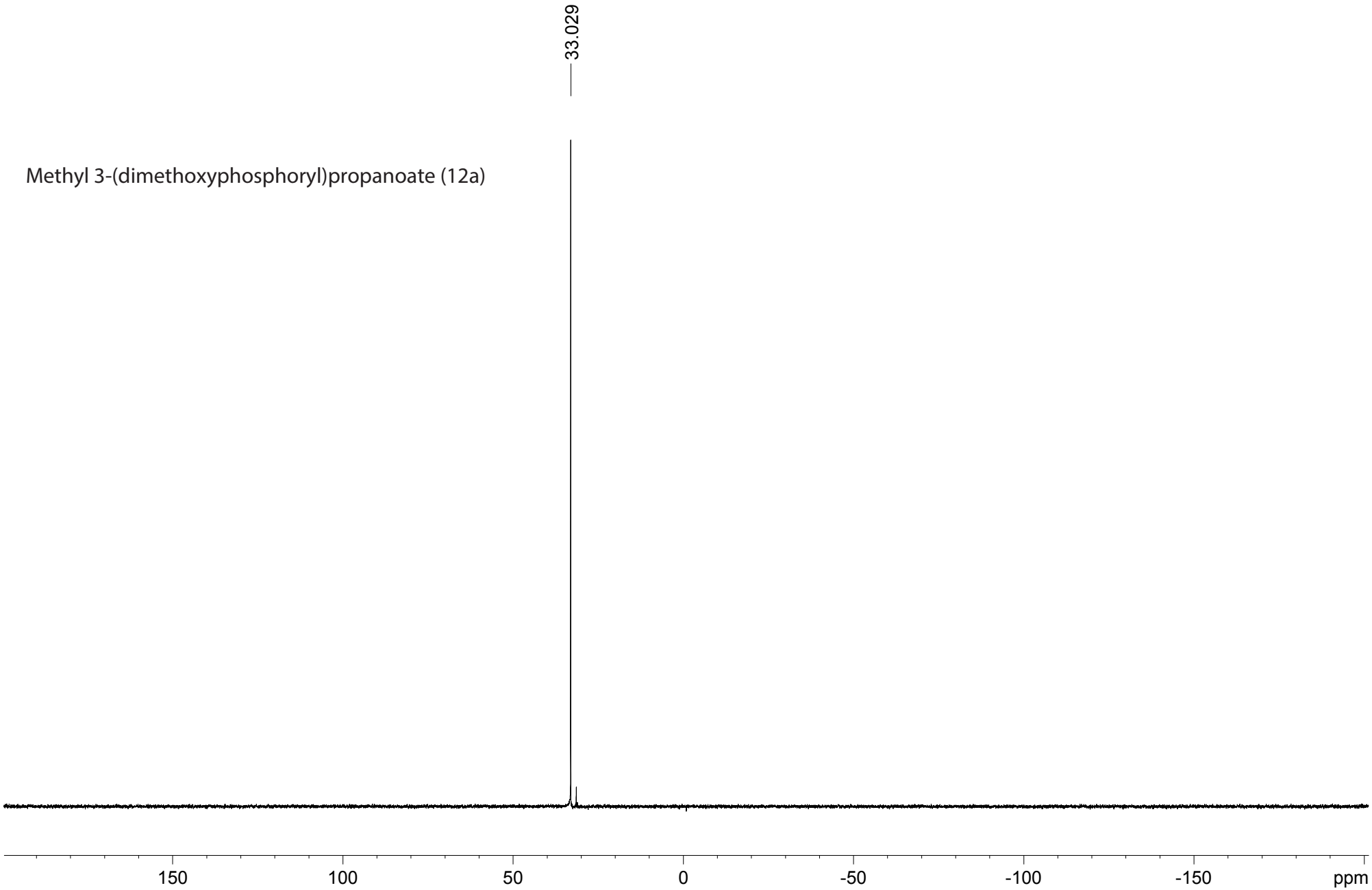

7.261
3.733
3.707
2.538
2.526
2.523
2.502
2.490
2.475
2.461
2.431
2.401
2.395
2.382
2.366
2.346
2.313
2.299
2.282
2.268
2.246
2.231
2.217
2.208
2.198
2.190
2.179
2.170
2.161
2.152
2.141
2.127
2.112
2.095
2.089
2.075
2.067
2.055
1.811
1.788
1.781
1.773
1.765
1.755
1.751
1.747
1.743
1.740
1.727
1.719
1.710
1.704
1.692
1.679
1.671
1.661
1.648
1.640
1.630
1.618
1.611
1.606
Dimethyl (3–oxocyclohexyl)phosphonate (7g)
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
−0.5
−1.0
−1.5
−2.0
−2.5
ppm
6.000
7.392
2.203

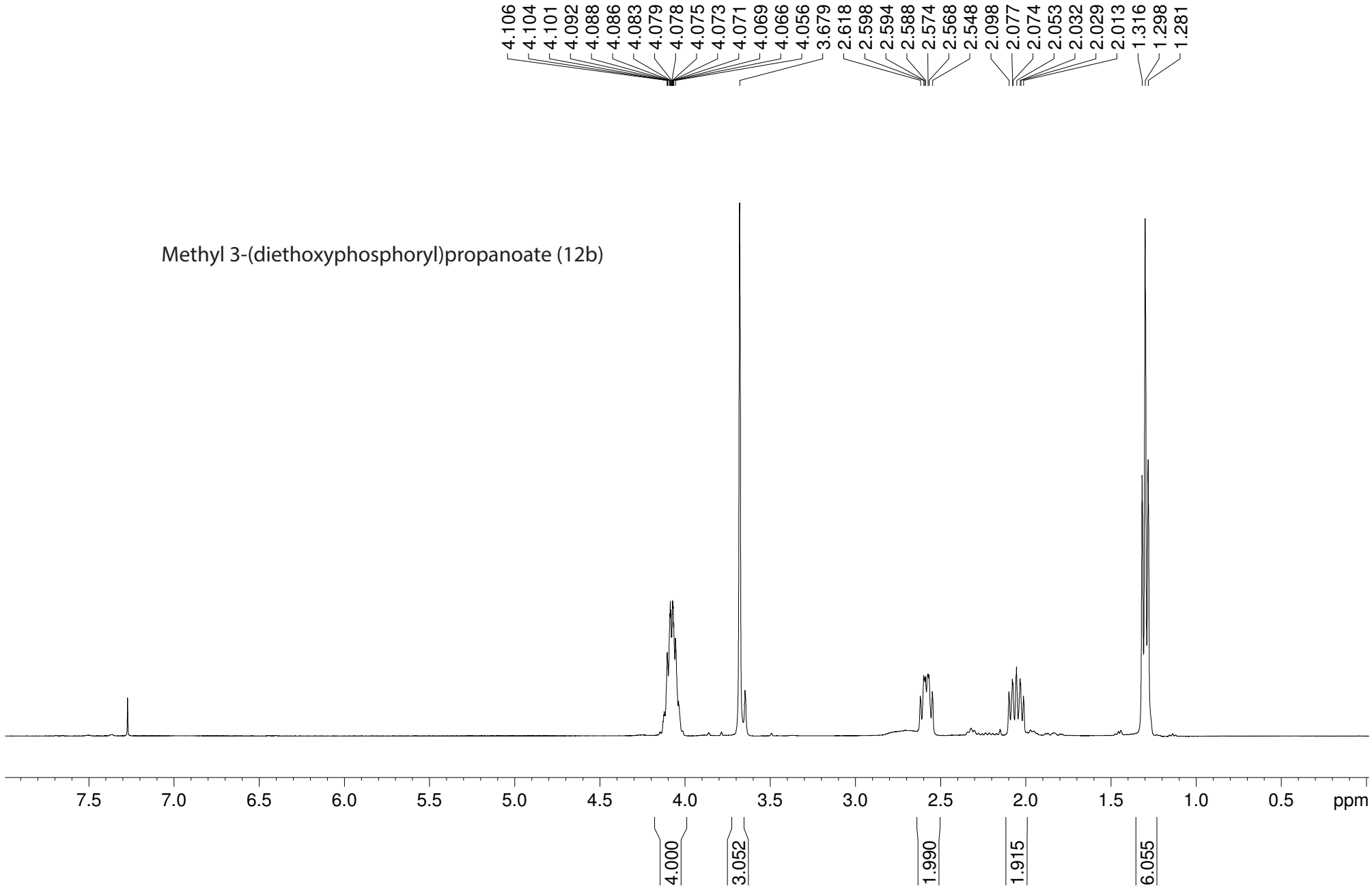

208.605
208.439
77.330
77.011
76.693
52.775
52.706
40.962
40.334
40.284
36.104
34.654
25.996
25.809
24.309
24.265
Dimethyl (3–oxocyclohexyl)phosphonate (7g)
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

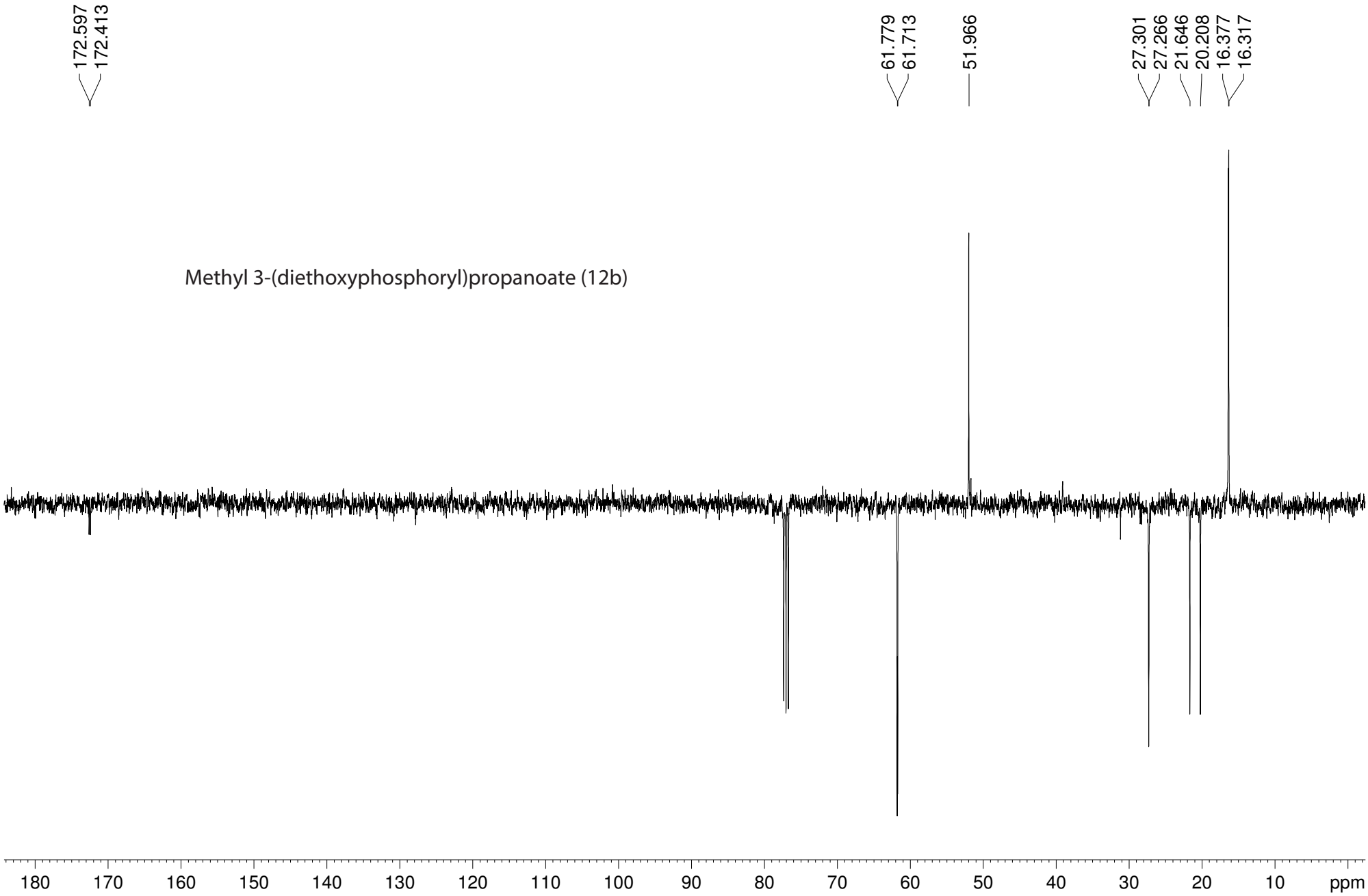

35.268
Dimethyl (3–oxocyclohexyl)phosphonate (7g)
150
100
50
0
–50
–100
–150
ppm


3.748
3.721
3.692
2.628
2.608
2.597
2.588
2.577
2.557
2.126
2.106
2.086
2.081
2.060
2.041
Methyl 3-(dimethoxyphosphoryl)propanoate (12a)
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
6.000
3.068
1.977
2.100


172.499
172.308
52.498
52.434
52.023
27.145
27.106
20.653
19.217
Methyl 3-(dimethoxyphosphoryl)propanoate (12a)
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

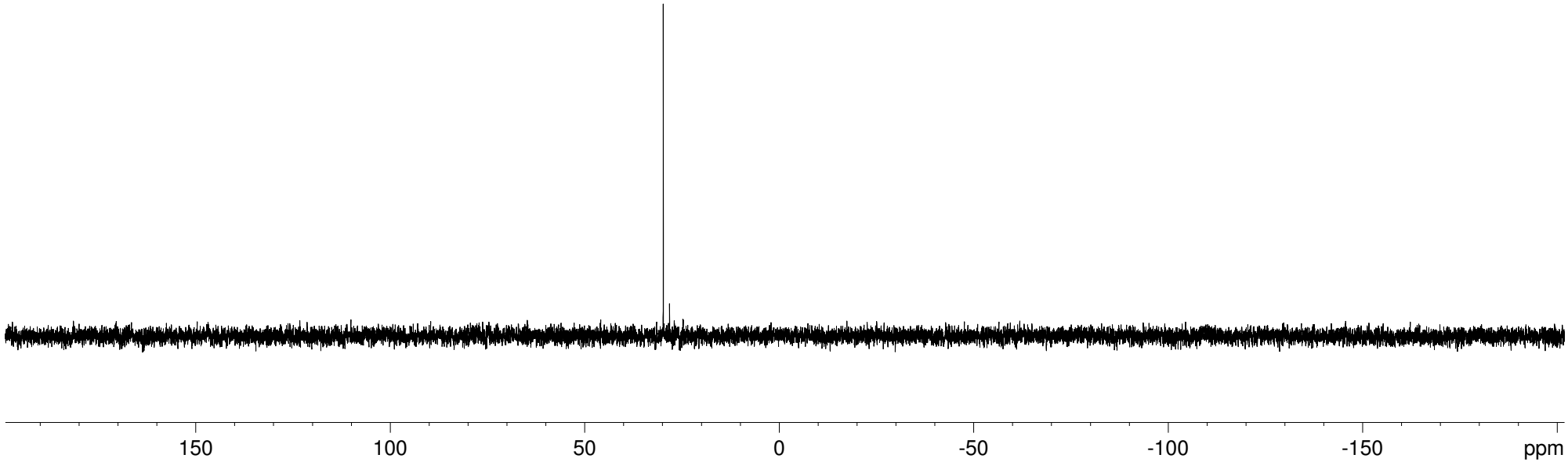

33.029
Methyl 3-(dimethoxyphosphoryl)propanoate (12a)
150
100
50
0
-50
-100
-150
ppm


4.106
4.104
4.101
4.092
4.088
4.086
4.083
4.079
4.078
4.075
4.073
4.071
4.069
4.066
4.056
3.679
2.618
2.598
2.594
2.588
2.574
2.568
2.548
2.098
2.077
2.074
2.053
2.032
2.029
2.013
1.316
1.298
1.281
Methyl 3-(diethoxyphosphoryl)propanoate (12b)
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
4.000
3.052
1.990
1.915
6.055

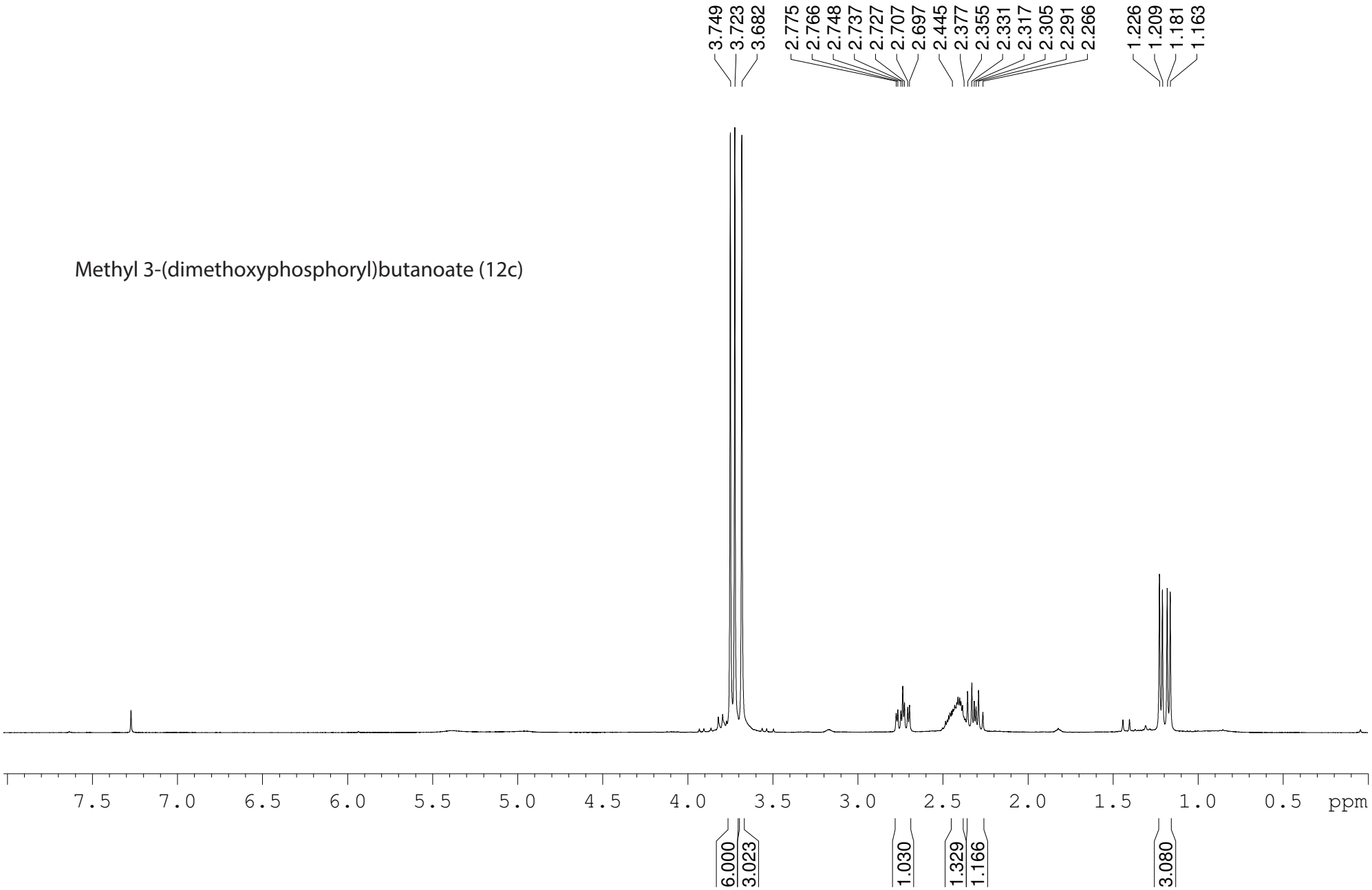

172.597
172.413
61.779
61.713
51.966
27.301
27.266
21.646
20.208
16.377
16.317
Methyl 3-(diethoxyphosphoryl)propanoate (12b)
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

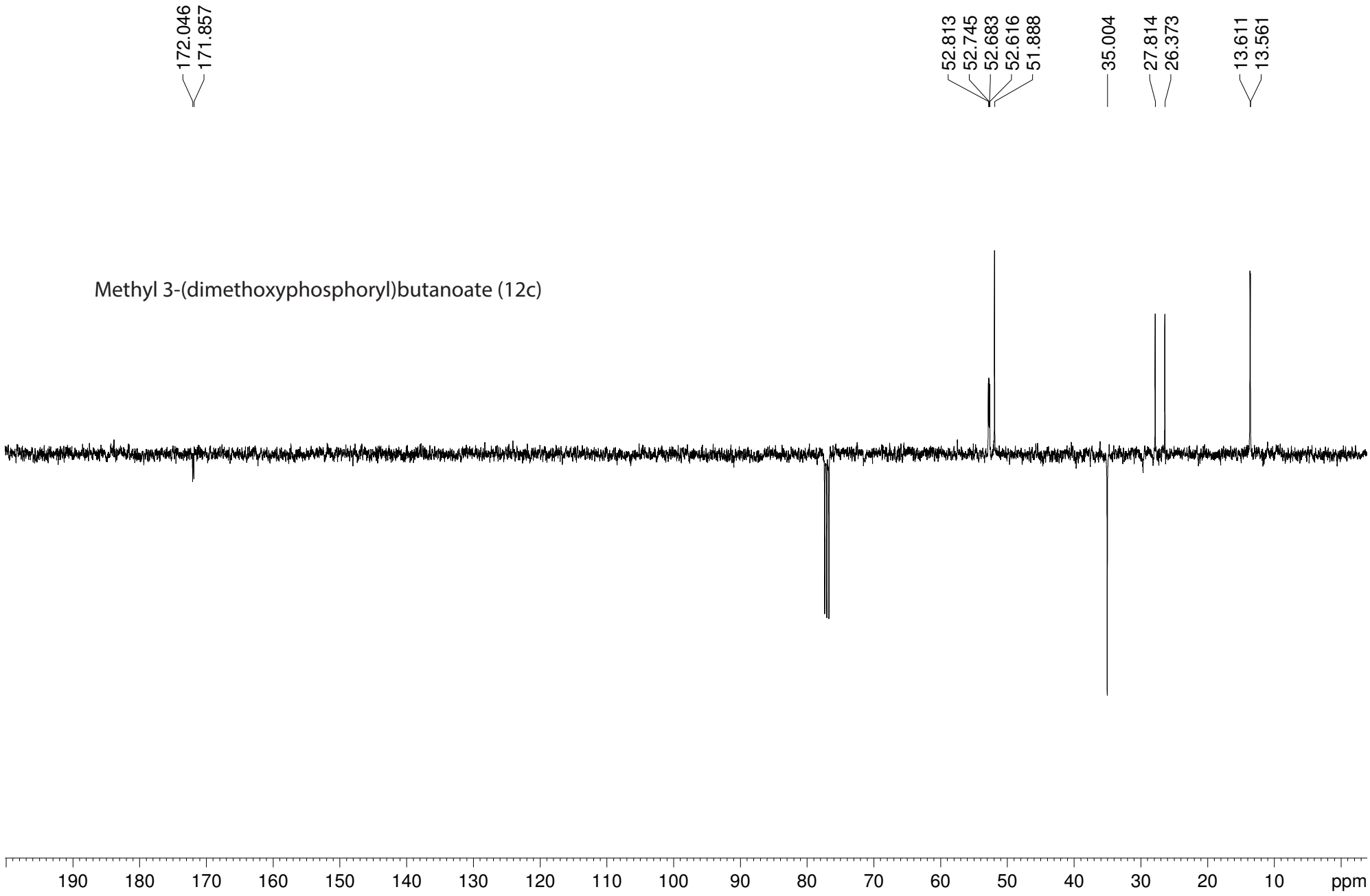
29.801

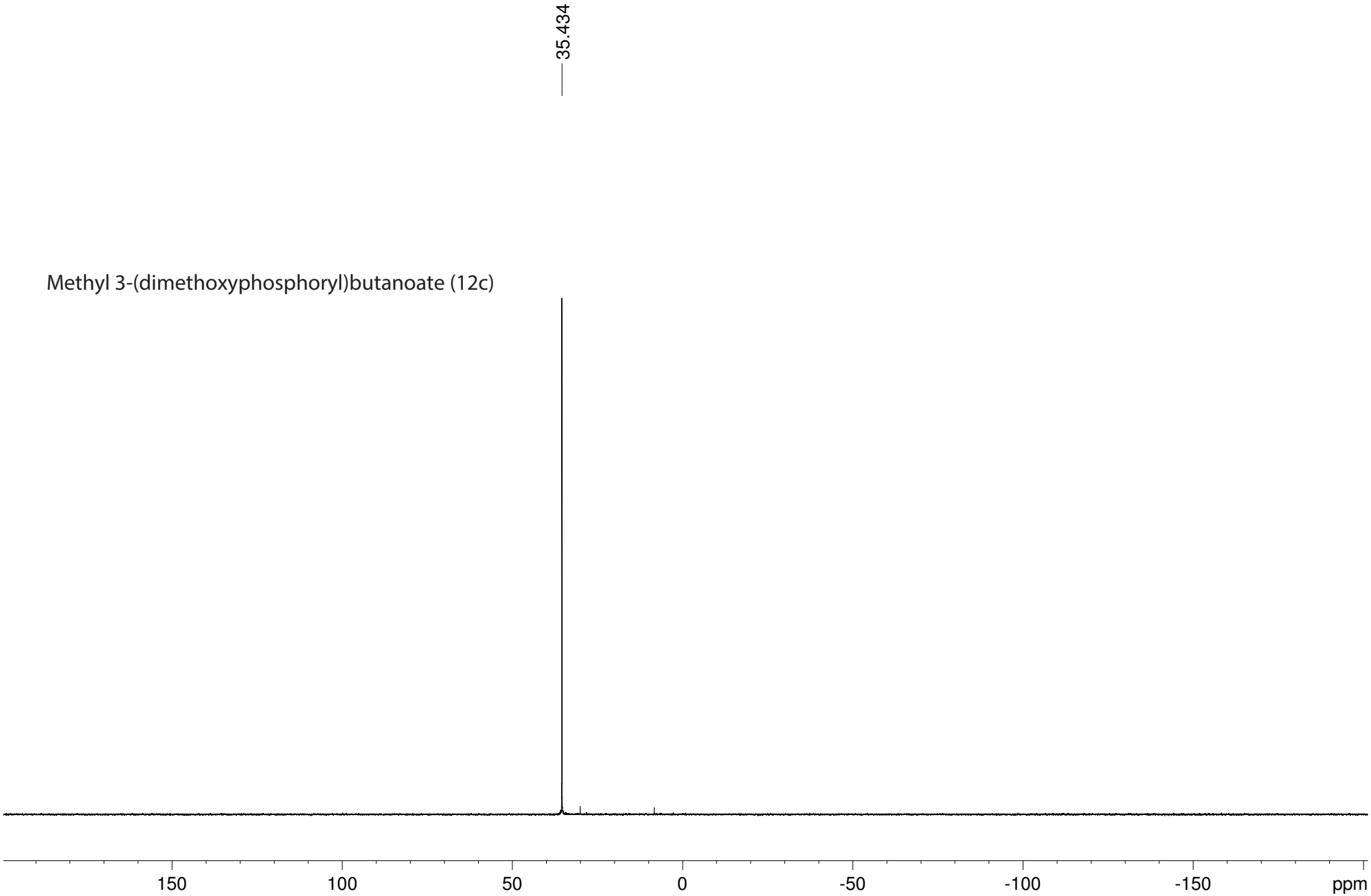
Methyl 3-(diethoxyphosphoryl)propanoate (12b)

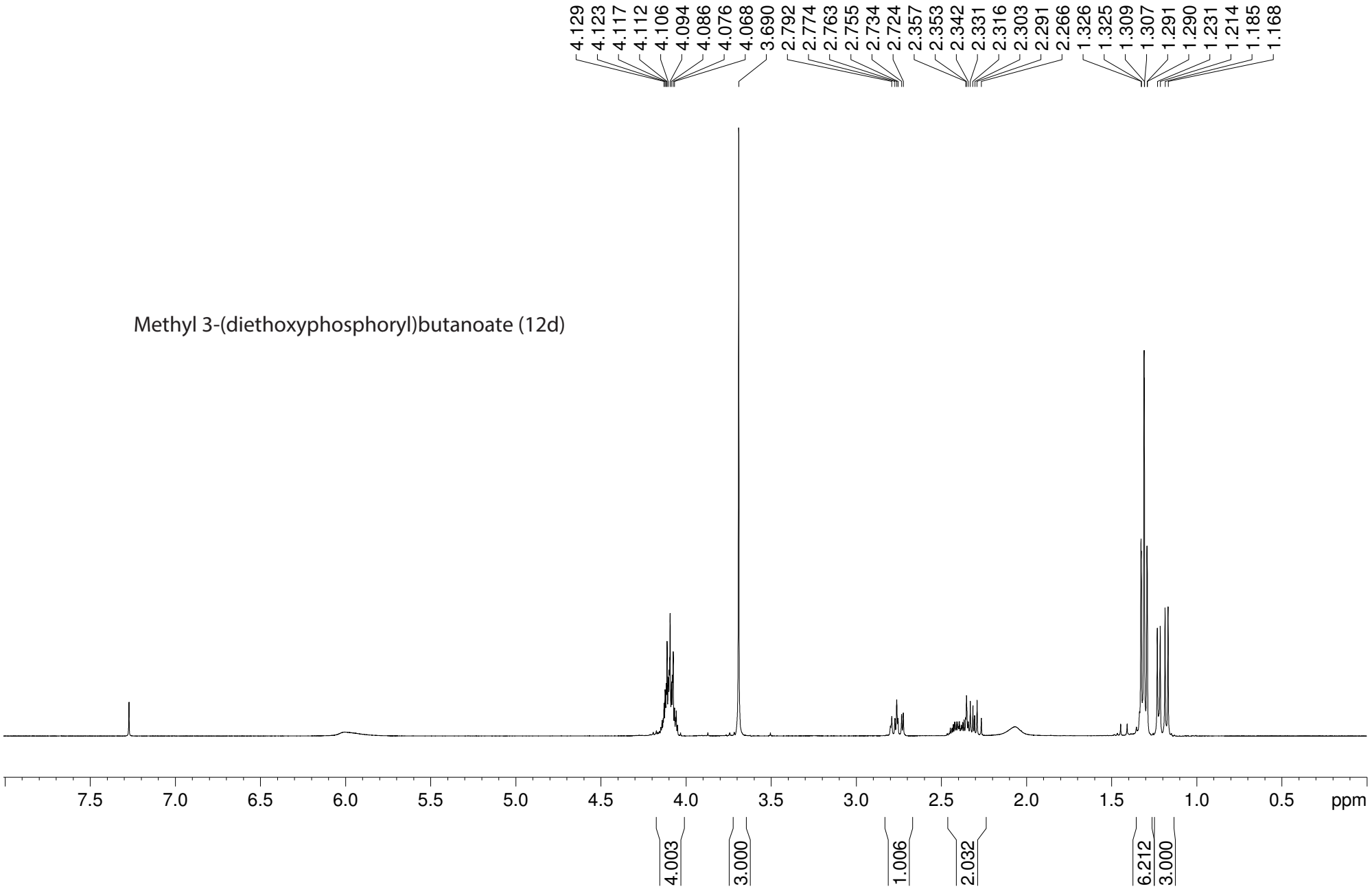


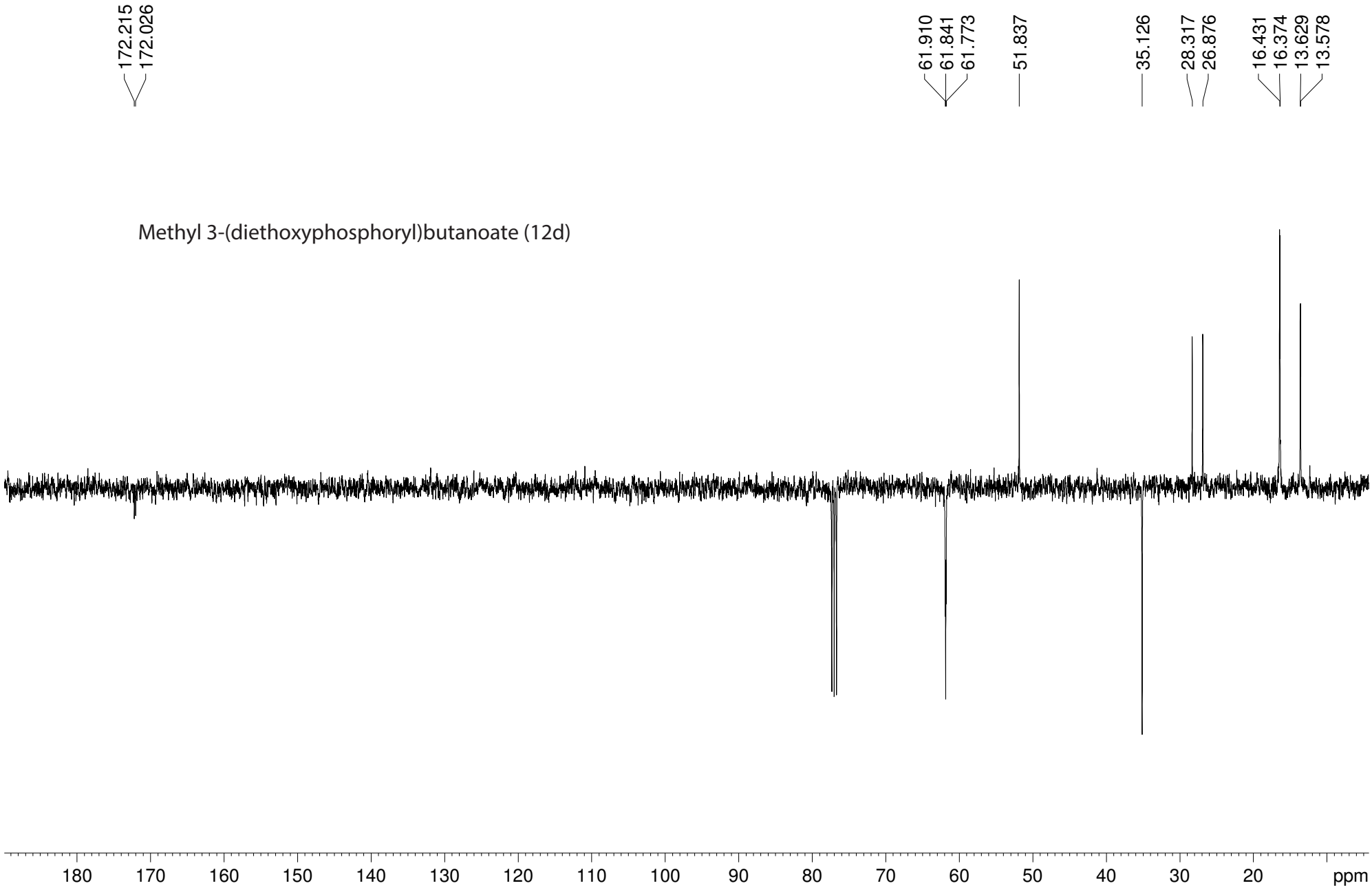
Methyl 3-(dimethoxyphosphoryl)butanoate (12c)

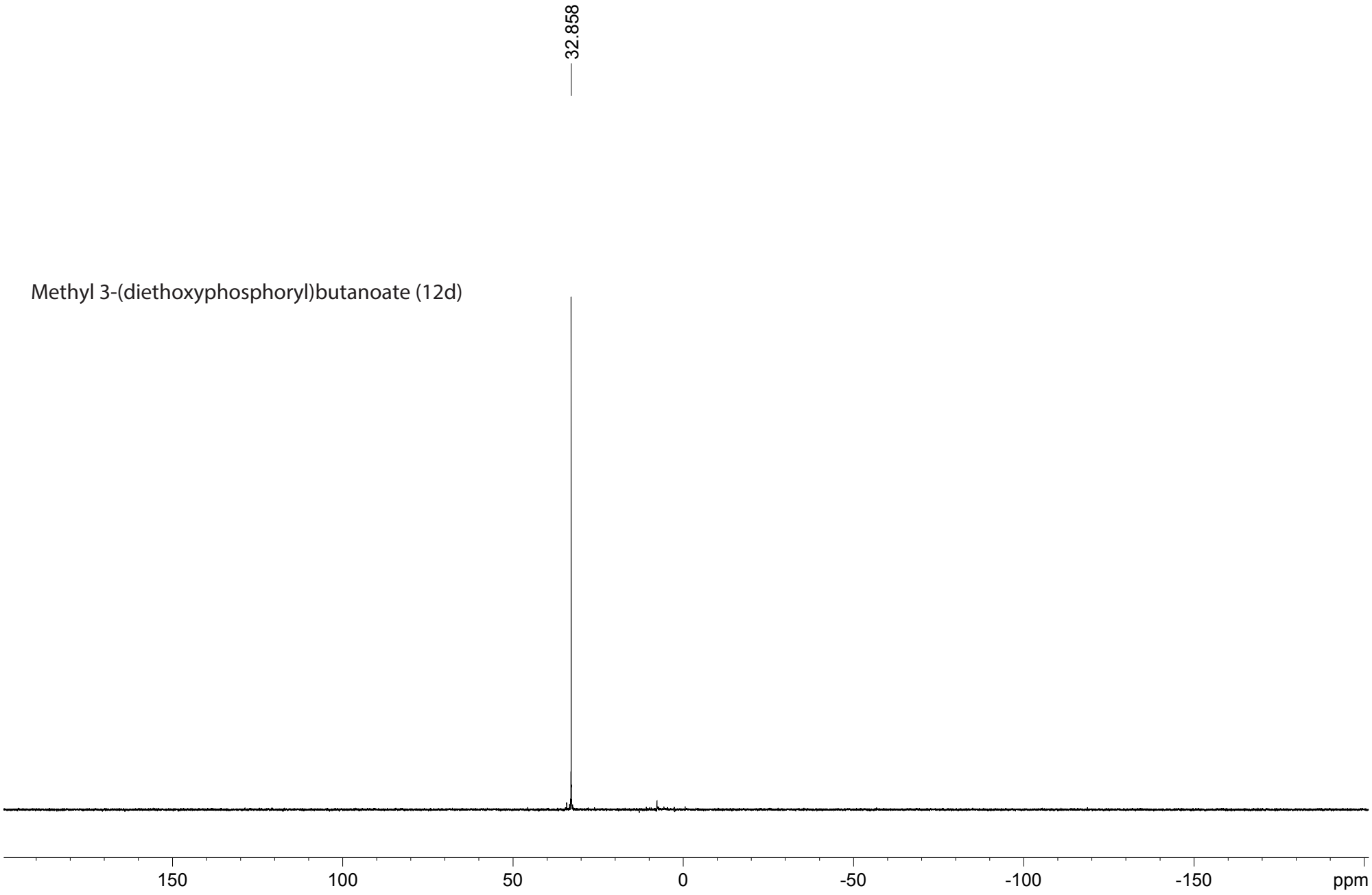


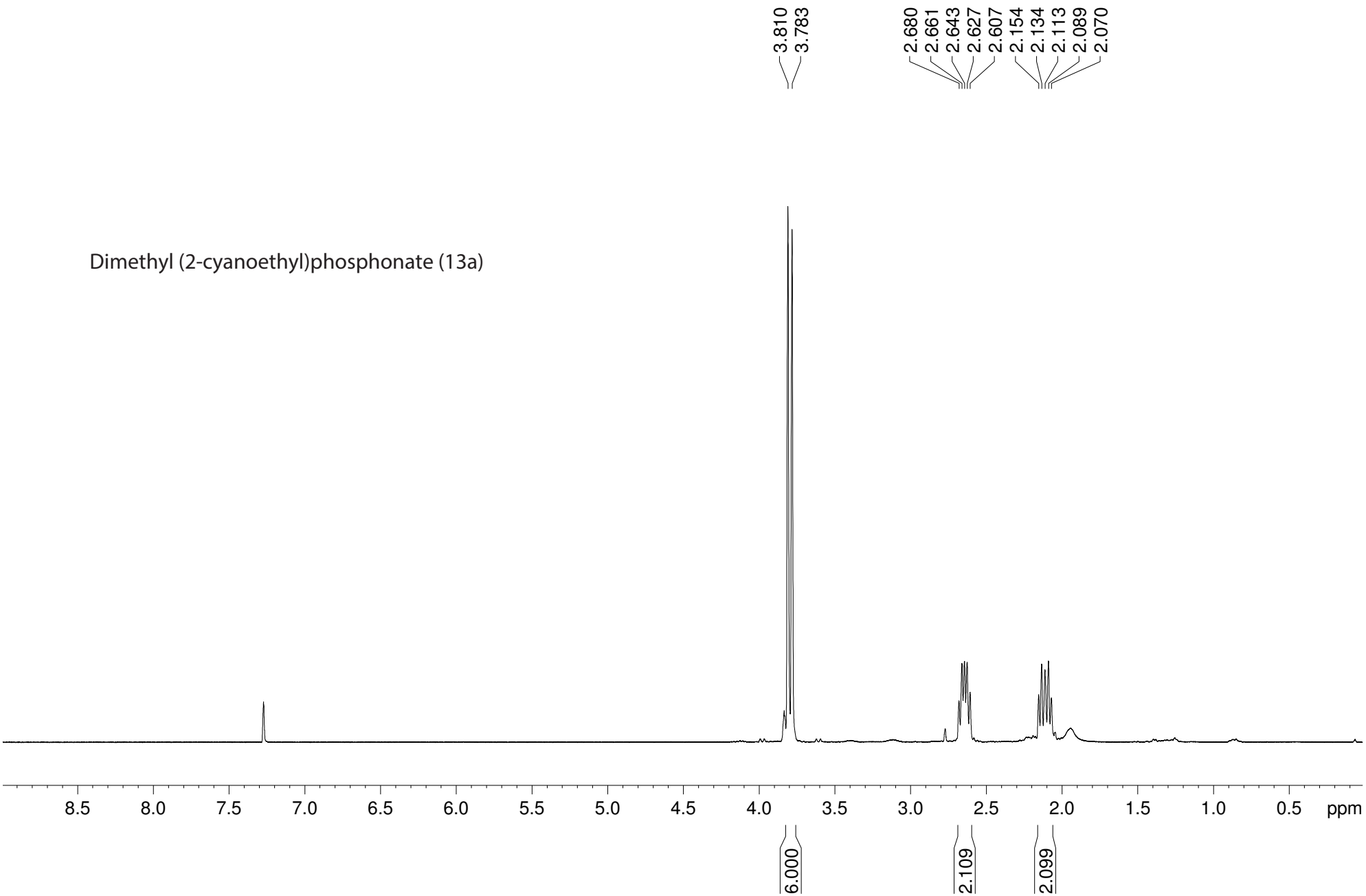

172.046
171.857
52.813
52.745
52.683
52.616
51.888
35.004
27.814
26.373
13.611
13.561
Methyl 3-(dimethoxyphosphoryl)butanoate (12c)
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm


35.434
Methyl 3-(dimethoxyphosphoryl)butanoate (12c)
150
100
50
0
-50
-100
-150
ppm

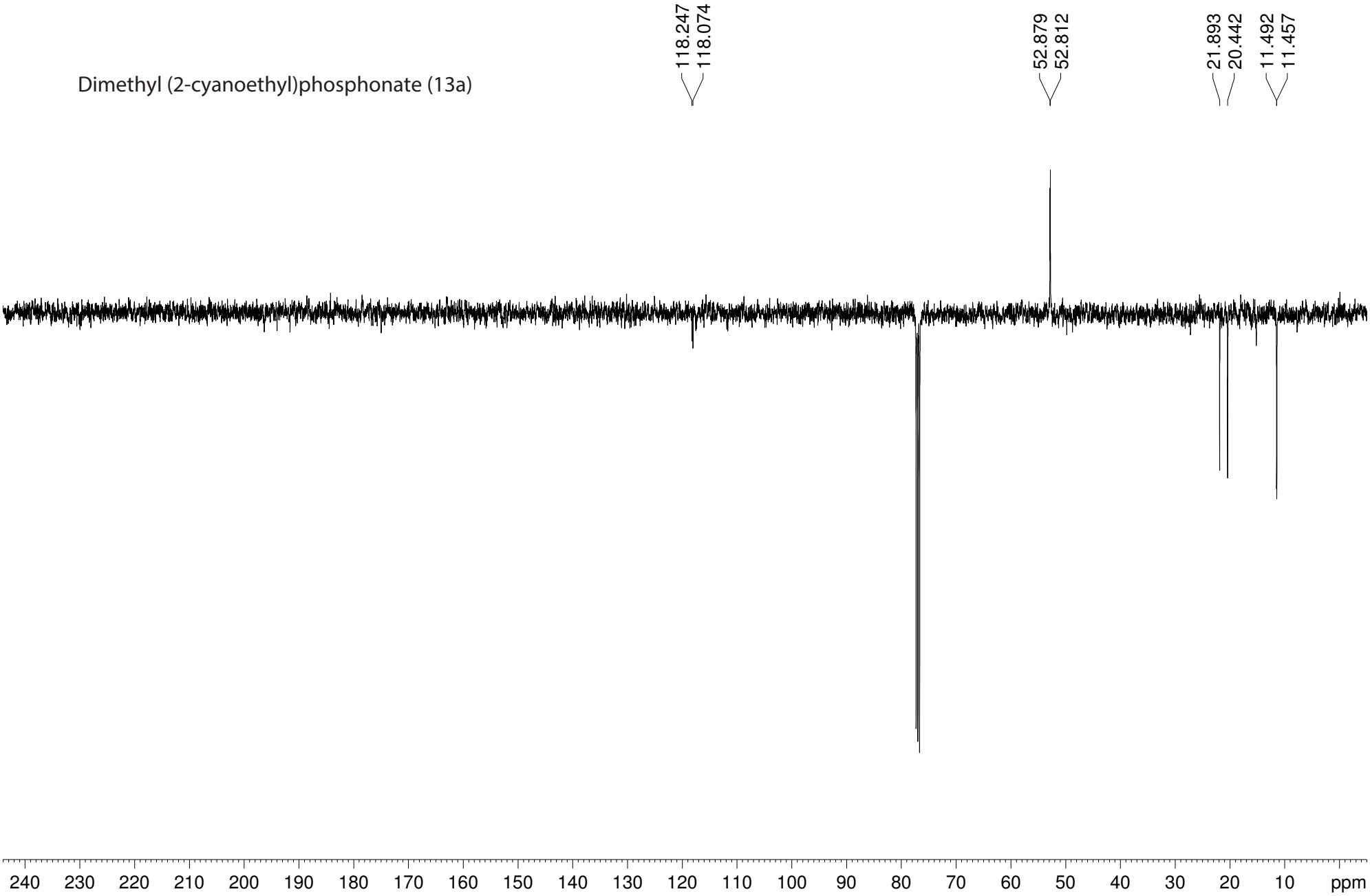

4.129
4.123
4.117
4.112
4.106
4.094
4.086
4.076
4.068
3.690
2.792
2.774
2.763
2.755
2.734
2.724
2.357
2.353
2.342
2.331
2.316
2.303
2.291
2.266
1.326
1.325
1.309
1.307
1.291
1.290
1.231
1.214
1.185
1.168
Methyl 3-(diethoxyphosphoryl)butanoate (12d)
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
4.003
3.000
1.006
2.032
6.212
3.000


172.215
172.026
61.910
61.841
61.773
51.837
35.126
28.317
26.876
16.431
16.374
13.629
13.578
Methyl 3-(diethoxyphosphoryl)butanoate (12d)
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

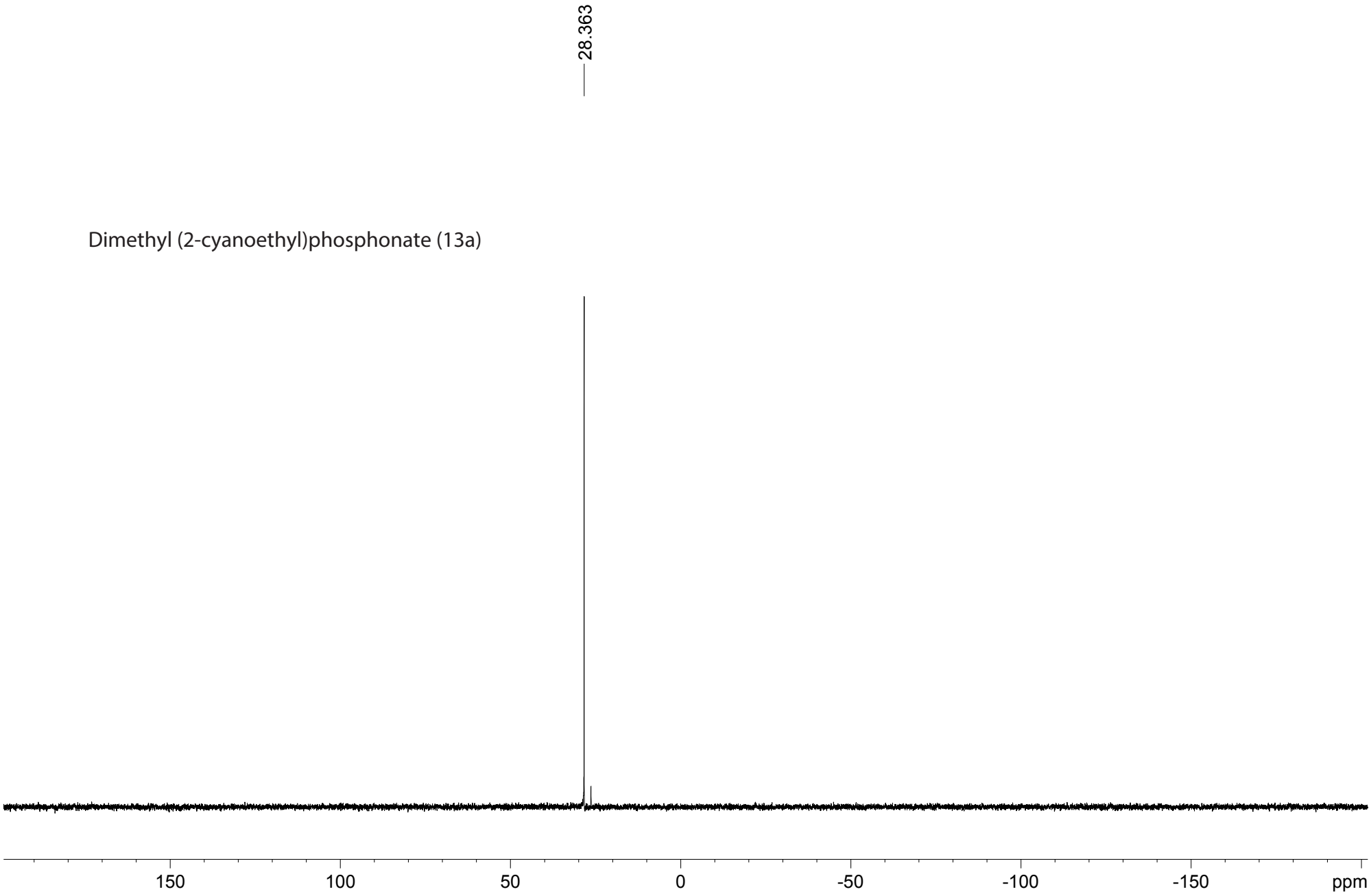

32.858
Methyl 3-(diethoxyphosphoryl)butanoate (12d)
150
100
50
0
-50
-100
-150
ppm

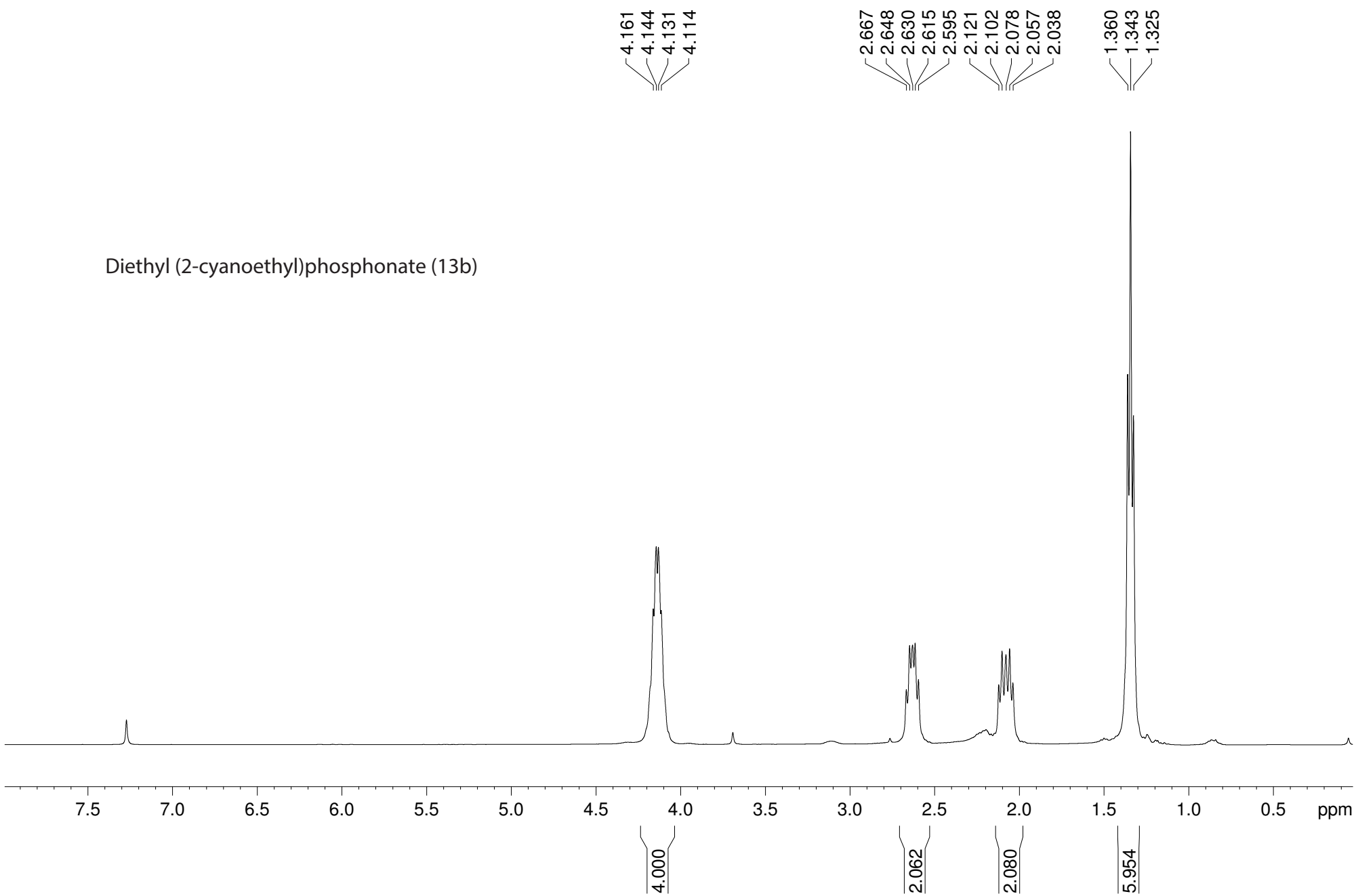

3.810
3.783
2.680
2.661
2.643
2.627
2.607
2.154
2.134
2.113
2.089
2.070
Dimethyl (2-cyanoethyl)phosphonate (13a)
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
6.000
2.109
2.099

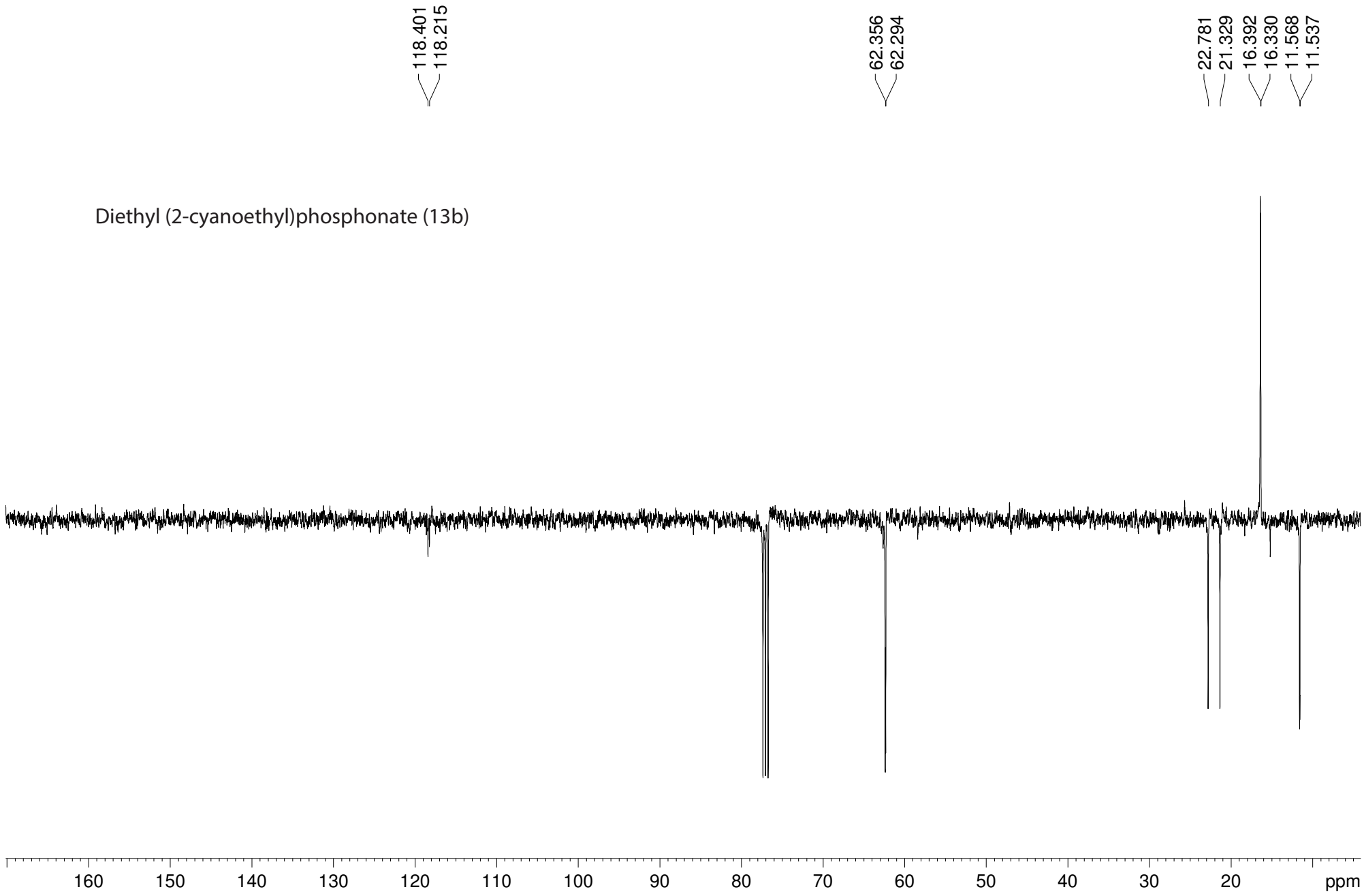
Dimethyl (2-cyanoethyl)phosphonate (13a)



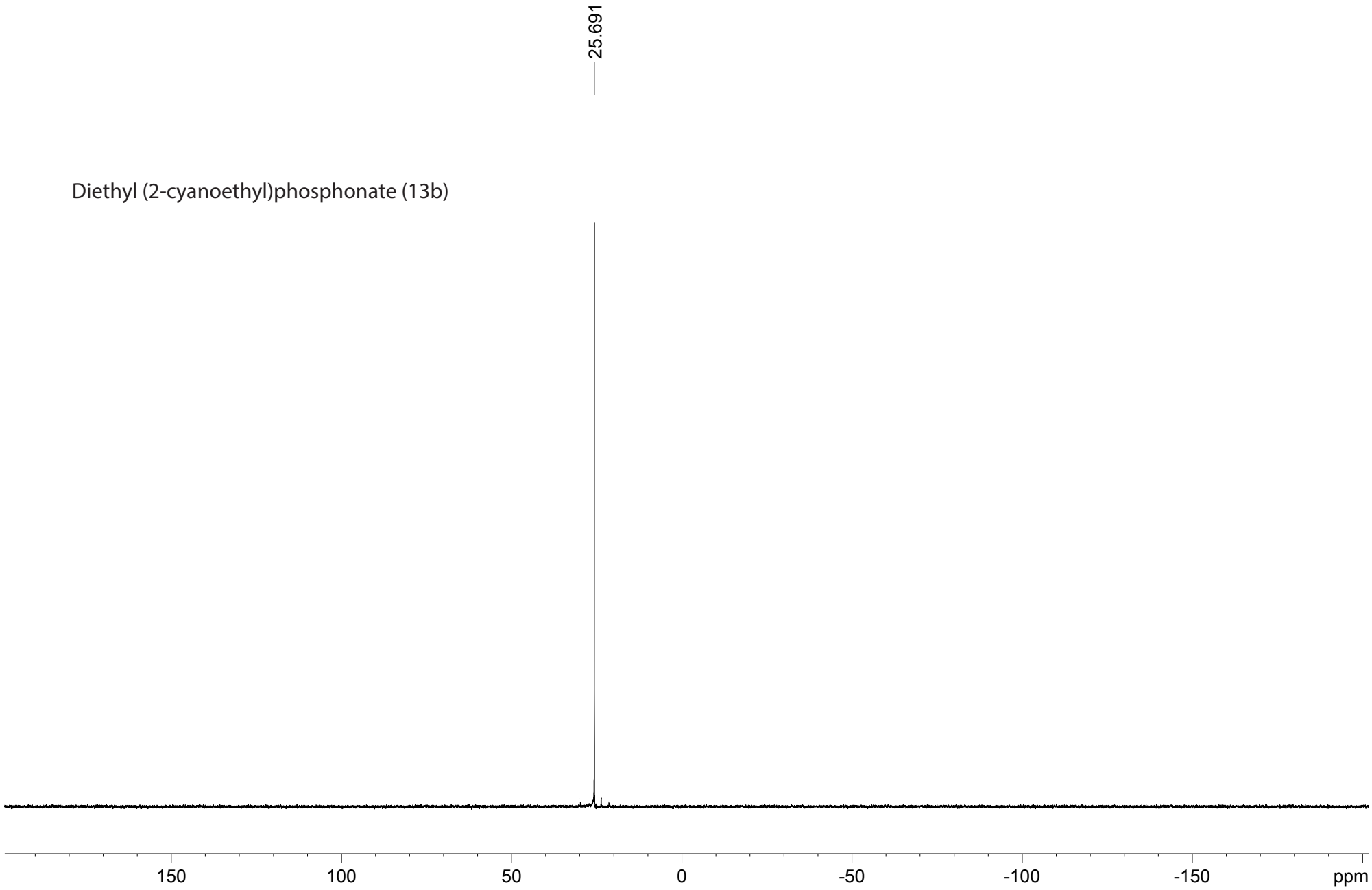
Dimethyl (2-cyanoethyl)phosphonate (13a)

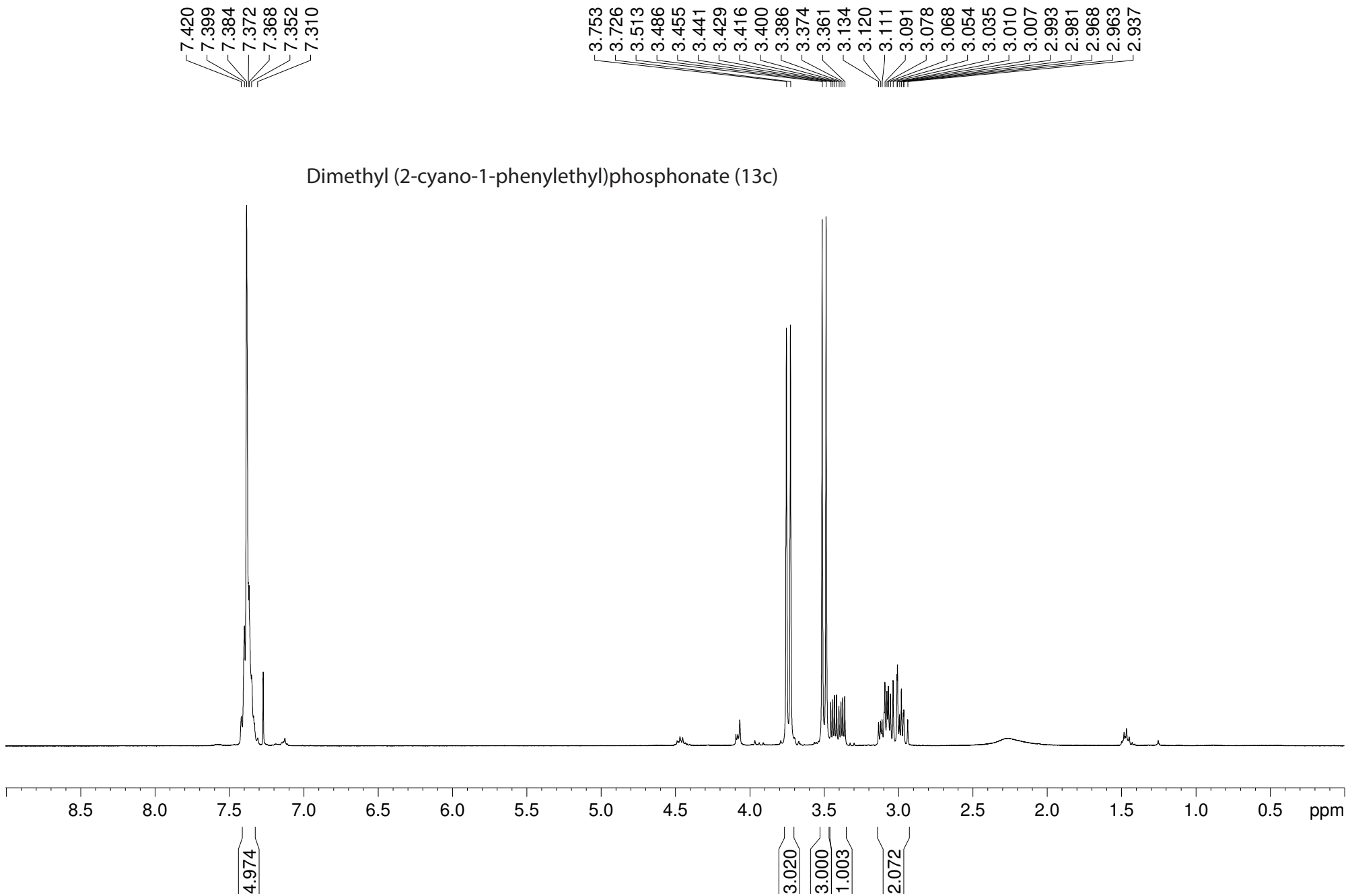


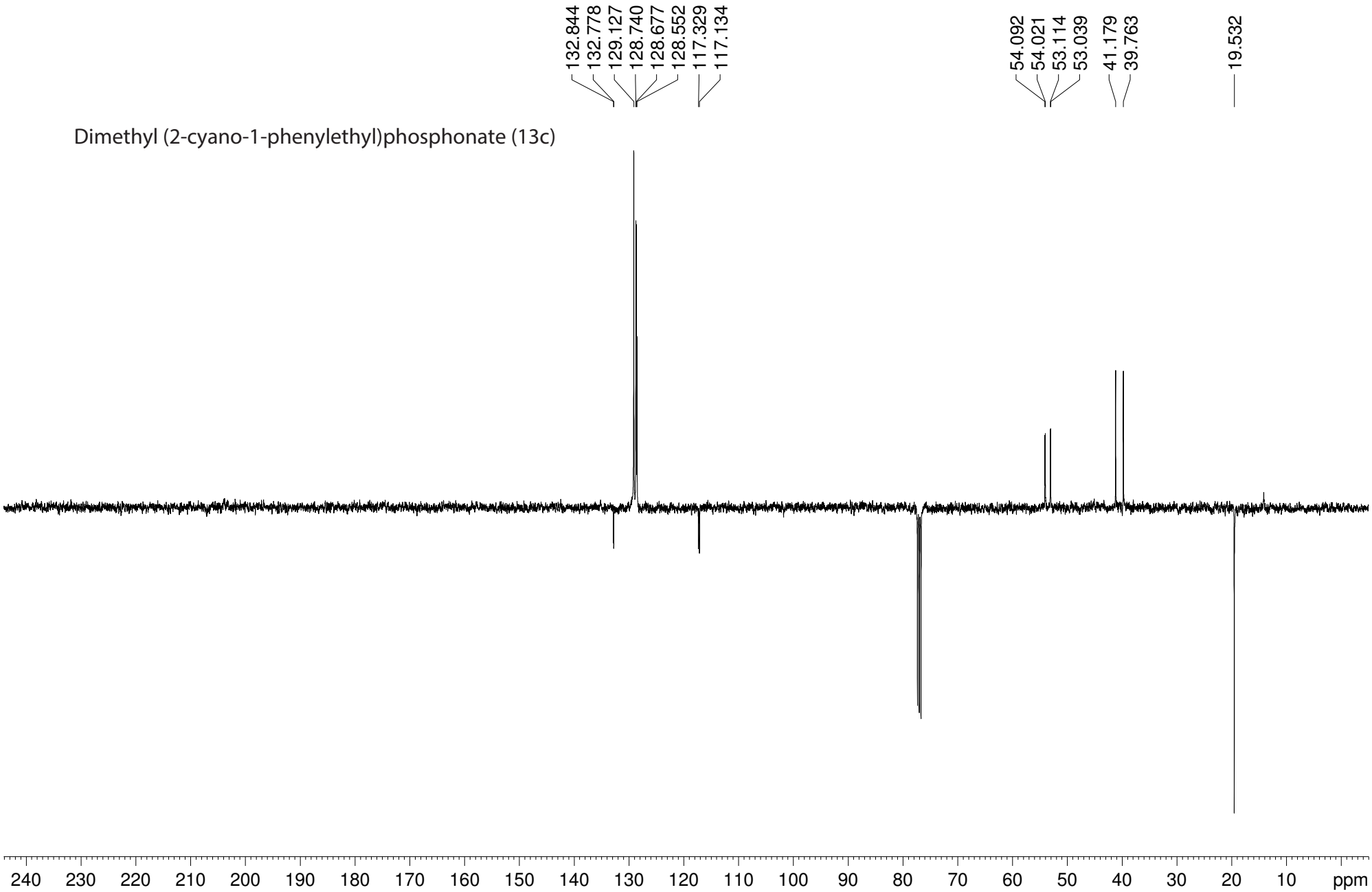

4.161
4.144
4.131
4.114
2.667
2.648
2.630
2.615
2.595
2.121
2.102
2.078
2.057
2.038
1.360
1.343
1.325
Diethyl (2-cyanoethyl)phosphonate (13b)
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
4.000
2.062
2.080
5.954


118.401
118.215
62.356
62.294
22.781
21.329
16.392
16.330
11.568
11.537
Diethyl (2-cyanoethyl)phosphonate (13b)
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

Diethyl (2-cyanoethyl)phosphonate (13b)

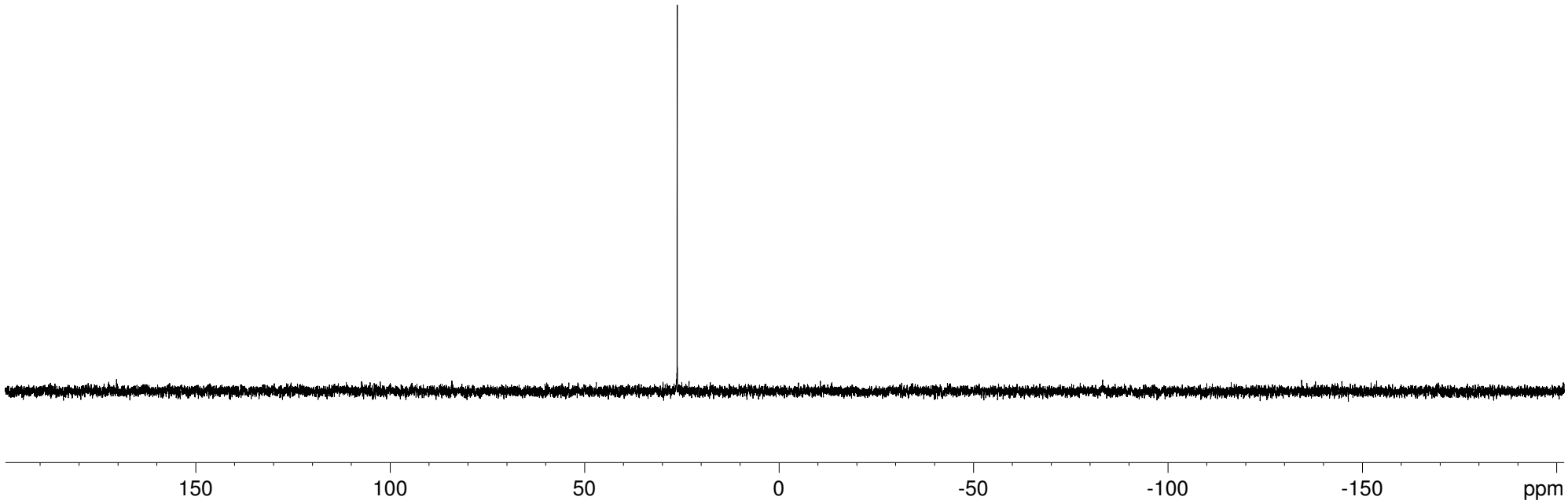


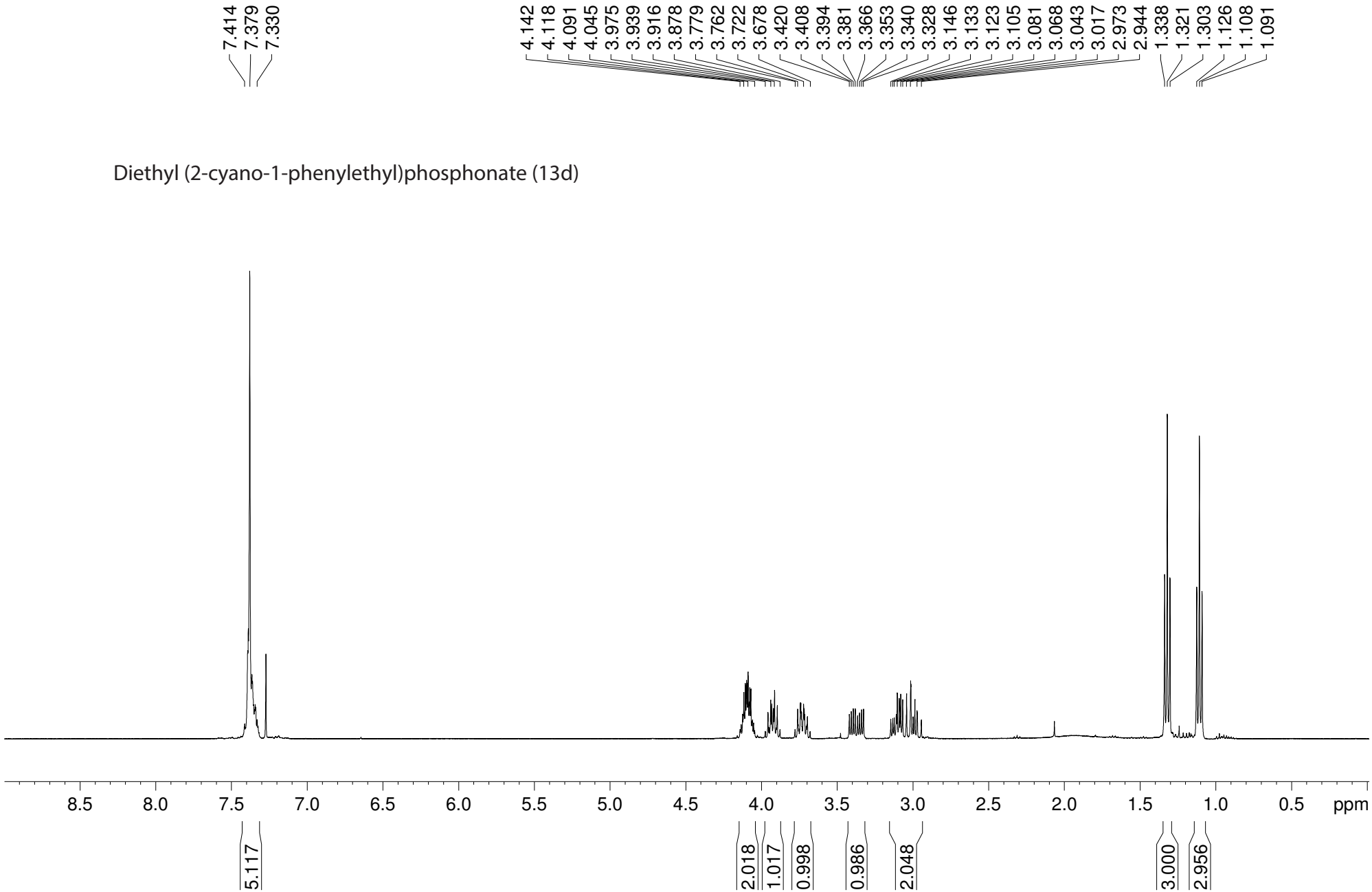

7.420
7.399
7.384
7.372
7.368
7.352
7.310
3.753
3.726
3.513
3.486
3.455
3.441
3.429
3.416
3.400
3.386
3.374
3.361
3.134
3.120
3.111
3.091
3.078
3.068
3.054
3.035
3.010
3.007
2.993
2.981
2.968
2.963
2.937
Dimethyl (2-cyano-1-phenylethyl)phosphonate (13c)
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
4.974
3.020
3.000
1.003
2.072


132.844
132.778
129.127
128.740
128.677
128.552
117.329
117.134
54.092
54.021
53.114
53.039
41.179
39.763
19.532
Dimethyl (2-cyano-1-phenylethyl)phosphonate (13c)
240 230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

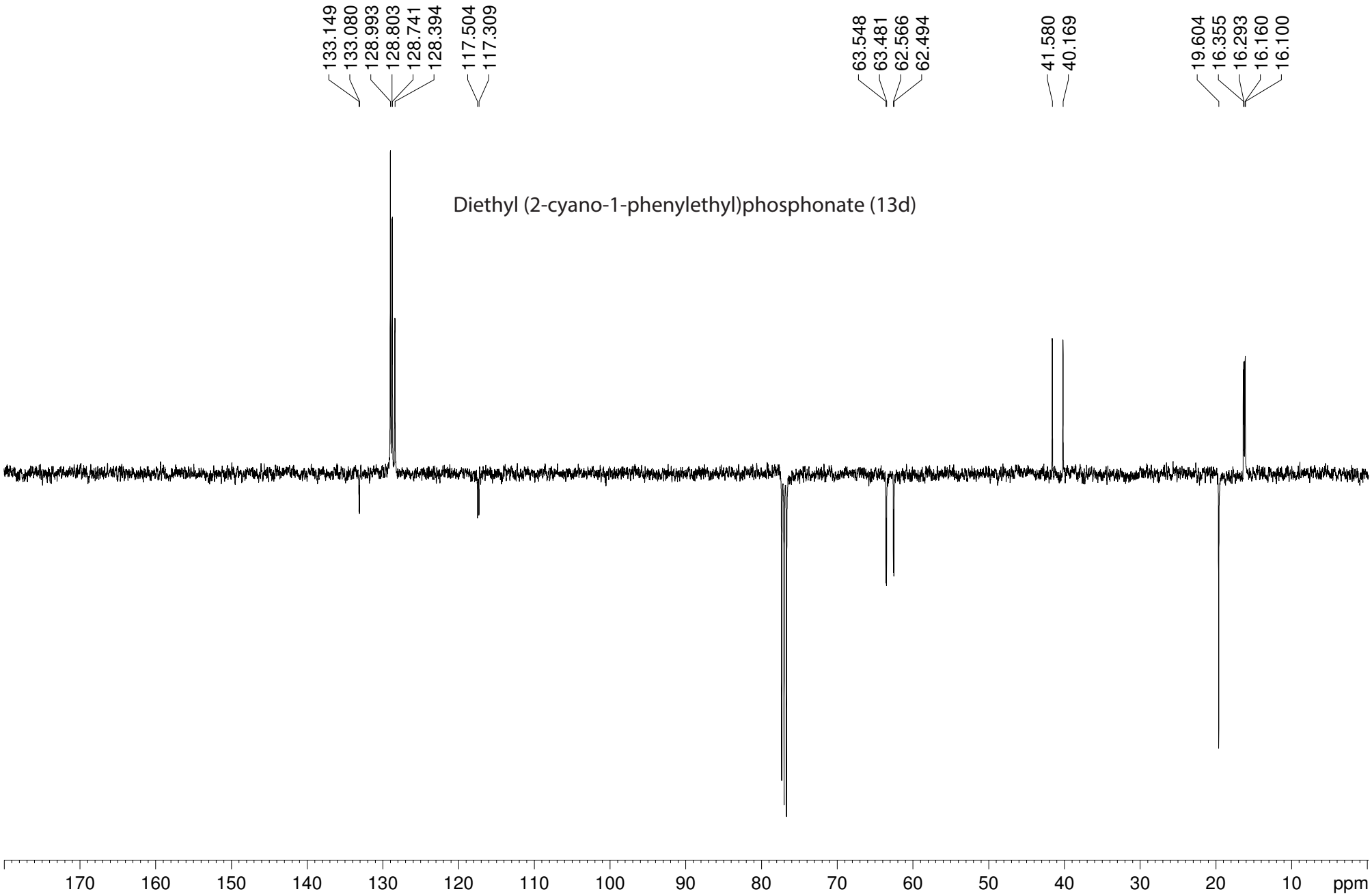
26.252

Dimethyl (2-cyano-1-phenylethyl)phosphonate (13c)





Diethyl (2-cyano-1-phenylethyl)phosphonate (13d)


133.149
133.080
128.993
128.803
128.741
128.394
117.504
117.309
63.548
63.481
62.566
62.494
41.580
40.169
19.604
16.355
16.293
16.160
16.100
Diethyl (2-cyano-1-phenylethyl)phosphonate (13d)
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

Diethyl (2-cyano-1-phenylethyl)phosphonate (13d)