Electronic Supporting Information for

Synthesis, Crystal Structure and Hydrolysis of Novel Isomeric Cage (P–C/P–O)-Phosphoranes on the basis of 4,4,5,5-Tetramethyl-2-(2-oxo-1,2-diphenylethoxy)-1,3,2-dioxaphospholane and Hexafluoroacetone

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General information:

All the manipulations were performed in argon vessels. All the solvents were obtained anhydrous according tostandard methods. NMR experiments were carried out with Bruker spectrometer AVANCE-400 (400.1 MHz (^{1}H), 162.0 МГц, (^{31}P), 100.6 MHz (^{13}C)) or Bruker spectrometer AVANCE-600 (600.0 MHz (^{1}H), 242.9 МГц, (^{31}P), 150.9 MHz (^{13}C)). Chemical shifts are reported in the δ (ppm) scale relative to the residual ^{1}H and ^{13}C signals of $CHCl_3$ or DMSO, or the external standard – H_3PO_4 (^{31}P). IR spectra were measured with Bruker Vector-22 spectrometer as suspensions in nujol or KBr pellets. Melting points were measured with a Stuart digital SMP10 apparatus and uncorrected. Elemental analyses for C, H and N were performed using a EuroVector 2000 CHNS-3 analyzer, Italy.

The X-ray diffraction data were collected on a Bruker AXS Smart Apex II CCD diffractometer in the ω- scan modes using graphite monochromated MoK_α (λ 0.71073 Å) radiation. The crystal data, data collection, and the refinement parameters are given in Table 1. Data were corrected for the absorption effect using SADABS program.[1] The structure was solved by direct method and refined by the full matrix least-squares using SHELX[2] and WinGX[3] programs. All non-hydrogen atoms were refined anisotropically. All hydrogen atoms were inserted at calculated positions and refined as riding atoms except the hydrogen atoms of OH groups which were located from difference maps and refined isotropically. Data collection: images were indexed, integrated, and scaled using the APEX2 data reduction package.[4] Figures were made, molecular structures and conformations were analyzed by PLATON.[5] Crystallographic data (excluding structure factors) for the structure reported in this paper have been deposited with the Cambridge Crystallographic Data Centre as supplementary publication numbers CCDC 1406191. Copies of the data can be obtained, free of charge, on application to CCDC, 12 Union Road, Cambridge CB2 1EZ, UK, (fax: +44-(0)1223-336033 or e-mail: deposit@ccdc.cam.ac.uk).

References:

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4. C. F. Macrae, I. J. Bruno, J. A. Chisholm, P. R. Edgington, P. McCabe, E. Pidcock, L. Rodriguez-Monge, R. Taylor, J. van de Streek and P. A. Wood, *J. Appl. Cryst*. **2008**, *41*, 466-470, DOI 10.1107/S0021889807067908.

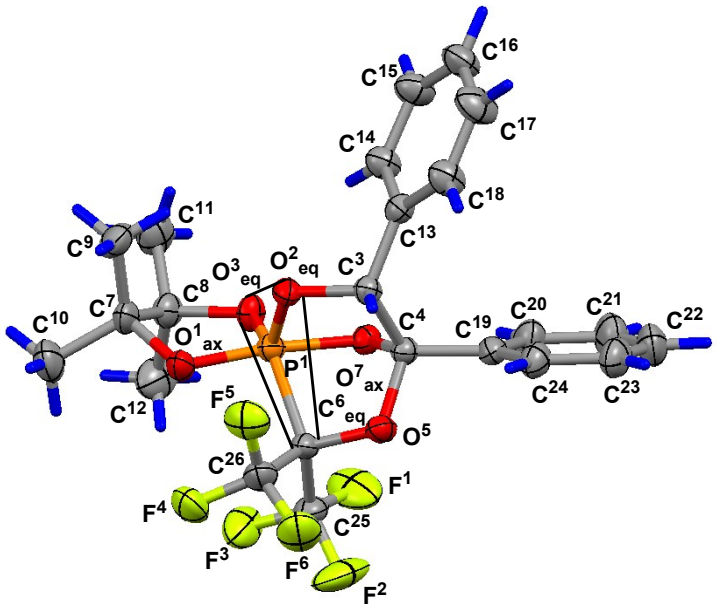


Table S1 – Bond Distances (Angstrom) for molecule **13**: k51_fin P 21 R = 0.07

Bond	*d*	Bond	*d*	Bond	*d*
P^1–O^1	1.613(5)	O^3–C^8	1.483(10)	C^6–C^{25}	1.540(12)
P^1–O^2	1.619(5)	O^5–C^4	1.482(8)	C^6–C^{26}	1.516(11)
P^1–O^3	1.583(5)	O^5–C^6	1.422(9)	C^7–C^8	1.529(12)
P^1–O^7	1.711(5)	O^7–C^4	1.386(8)	C^7–C^9	1.533(14)
P^1–C^6	1.930(7)	C^3–C^4	1.549(10)	C^7–C^{10}	1.527(15)
O^1–C^7	1.475(9)	C^3–C^{13}	1.515(10)	C^8–C^{11}	1.525(12)
O^2–C^3	1.446(8)	C^4–C^{19}	1.503(10)	C^8–C^{12}	1.523(13)

Table S2 – Bond Angles (Degrees) for molecule **13**: k51_fin P 21 R = 0.07

Bond angle	φ	Bond angle	φ	Bond angle	φ
O^1–P^1–O^2	96.9(3)	O^5–C^4–O^7	105.2(5)	F^4–C^{26}–C^6	114.0(7)
O^1–P^1–O^3	92.7(3)	O^5–C^4–C^3	108.2(5)	F^5–C^{26}–F^6	106.1(7)
O^1–P^1–O^7	172.4(3)	O^5–C^4–C^{19}	107.3(5)	F^5–C^{26}–C^6	112.4(7)
O^1–P^1–C^6	92.8(3)	O^7–C^4–C^3	103.7(5)	F^6–C^{26}–C^6	111.9(7)
O^2–P^1–O^3	120.9(3)	O^7–C^4–C^{19}	115.3(6)	O^5–C^6–C^{26}	105.6(6)
O^2–P^1–O^7	90.3(2)	C^3–C^4–C^{19}	116.4(6)	C^{25}–C^6–C^{26}	109.9(7)
O^2–P^1–C^6	100.7(3)	P^1–C^6–O^5	102.7(5)	O^1–C^7–C^8	102.3(6)
O^3–P^1–O^7	85.7(2)	P^1–C^6–C^{25}	113.4(5)	O^1–C^7–C^9	107.7(7)
O^3–P^1–C^6	136.9(3)	P^1–C^6–C^{26}	115.9(5)	O^1–C^7–C^{10}	106.3(7)
O^7–P^1–C^6	83.3(3)	O^5–C^6–C^{25}	108.7(6)	C^8–C^7–C^9	114.5(7)
P^1–O^1–C^7	113.3(4)	F^1–C^{25}–F^2	105.8(8)	C^8–C^7–C^{10}	115.6(8)
P^1–O^2–C^3	113.2(4)	F^1–C^{25}–F^3	106.2(9)	C^9–C^7–C^{10}	109.7(8)
P^1–O^3–C^8	116.5(4)	F^1–C^{25}–C^6	111.8(8)	O^3–C^8–C^7	102.9(6)
C^4–O^5–C^6	109.9(5)	F^2–C^{25}–F^3	106.7(8)	O^3–C^8–C^{11}	106.1(7)
P^1–O^7–C^4	102.0(4)	F^2–C^{25}–C^6	112.1(7)	O^3–C^8–C^{12}	105.4(7)
O^2–C^3–C^4	102.2(5)	F^3–C^{25}–C^6	113.7(8)	C^7–C^8–C^{11}	115.3(7)
O^2–C^3–C^{13}	110.1(5)	F^4–C^{26}–F^5	105.4(7)	C^7–C^8–C^{12}	115.7(8)
C^4–C^3–C^{13}	115.5(6)	F^4–C^{26}–F^6	106.5(7)	C^{11}–C^8–C^{12}	110.2(8)

Table S3 – Torsion Angles (Degrees) for molecule **13**: k51_fin P 21 R = 0.07

Torsion angle	τ	Torsion angle	τ	Torsion angle	τ
O^2–P^1–O^1–C^7	98.6(5)	O^2–P^1–C^6–C^{26}	62.0(6)	C^4–O^5–C^6–C^{25}	110.5(6)
O^3–P^1–O^1–C^7	–23.0(6)	O^3–P^1–C^6–O^5	113.0(5)	C^4–O^5–C^6–C^{26}	–131.6(6)
C^6–P^1–O^1–C^7	–160.2(6)	O^3–P^1–C^6–C^{25}	–4.1(8)	P^1–O^7–C^4–O^5	59.5(5)
O^1–P^1–O^2–C^3	154.3(4)	O^3–P^1–C^6–C^{26}	–132.6(6)	P^1–O^7–C^4–C^3	–54.1(5)
O^3–P^1–O^2–C^3	–108.4(5)	O^7–P^1–C^6–O^5	36.6(4)	P^1–O^7–C^4–C^{19}	177.5(5)
O^7–P^1–O^2–C^3	–23.1(4)	O^7–P^1–C^6–C^{25}	–80.5(6)	O^2–C^3–C^4–O^5	–73.5(6)
C^6–P^1–O^2–C^3	60.1(5)	O^7–P^1–C^6–C^{26}	151.0(6)	O^2–C^3–C^4–O^7	37.9(6)
O^1–P^1–O^3–C^8	0.6(5)	P^1–O^1–C^7–C^8	36.4(8)	O^2–C^3–C^4–C^{19}	165.6(6)
O^2–P^1–O^3–C^8	–99.1(5)	P^1–O^1–C^7–C^9	–84.6(7)	C^{13}–C^3–C^4–O^5	167.0(5)
O^7–P^1–O^3–C^8	173.1(5)	P^1–O^1–C^7–C^{10}	158.0(6)	C^{13}–C^3–C^4–O^7	–81.6(7)
C^6–P^1–O^3–C^8	97.7(6)	P^1–O^2–C^3–C^4	–4.2(6)	C^{13}–C^3–C^4–C^{19}	46.2(8)
O^2–P^1–O^7–C^4	46.1(4)	P^1–O^2–C^3–C^{13}	119.0(5)	O^2–C^3–C^{13}–C^{14}	–43.2(9)
O^3–P^1–O^7–C^4	167.1(4)	P^1–O^3–C^8–C^7	19.7(8)	O^2–C^3–C^{13}–C^{18}	137.0(7)
C^6–P^1–O^7–C^4	–54.7(4)	P^1–O^3–C^8–C^{11}	141.2(6)	C^4–C^3–C^{13}–C^{14}	71.9(9)
O^1–P^1–C^6–O^5	–150.1(4)	P^1–O^3–C^8–C^{12}	–101.9(8)	C^4–C^3–C^{13}–C^{18}	–108.0(8)
O^1–P^1–C^6–C^{25}	92.9(6)	C^6–O^5–C^4–O^7	–29.4(7)	O^5–C^4–C^{19}–C^{20}	117.8(8)
O^1–P^1–C^6–C^{26}	–35.6(6)	C^6–O^5–C^4–C^3	81.0(6)	O^5–C^4–C^{19}–C^{24}	–64.4(8)
O^2–P^1–C^6–O^5	–52.5(5)	C^6–O^5–C^4–C^{19}	–152.7(6)	O^7–C^4–C^{19}–C^{20}	1(1)

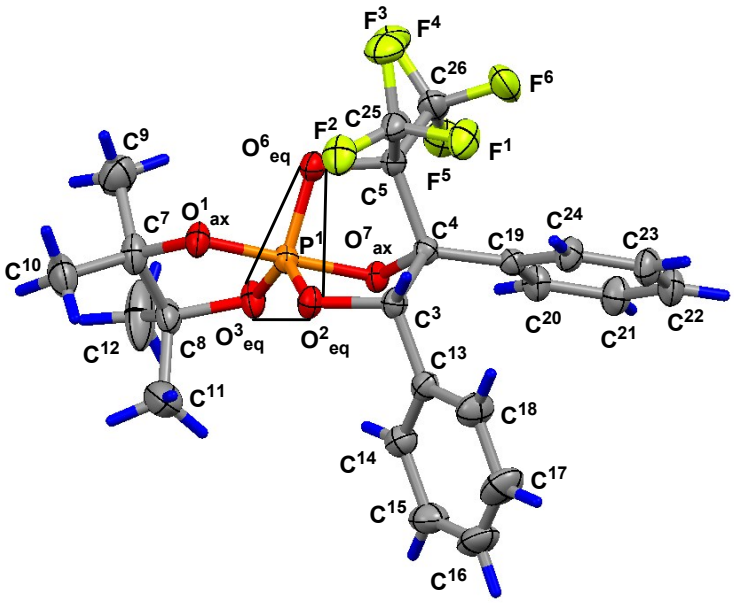


Table S4 – Bond Distances (Angstrom) for molecule **14**: k53n_fin1 P b c a R = 0.05

Bond	*d*	Bond	*d*	Bond	*d*
P^1–O^1	1.625(2)	O^3–C^8	1.464(4)	C^8–C^{12}	1.544(7)
P^1–O^2	1.606(2)	O^6–C^5	1.421(4)	C^{13}–C^{14}	1.380(5)
P^1–O^3	1.582(2)	O^7–C^4	1.411(3)	C^{13}–C^{18}	1.382(5)
P^1–O^6	1.647(2)	C^3–C^4	1.571(4)	C^{14}–C^{15}	1.396(5)
P^1–O^7	1.700(2)	C^3–C^{13}	1.509(4)	C^{15}–C^{16}	1.352(7)
F^1–C^{25}	1.344(4)	C^4–C^5	1.616(4)	C^{16}–C^{17}	1.367(7)
F^2–C^{25}	1.315(4)	C^4–C^{19}	1.508(4)	C^{17}–C^{18}	1.384(6)
F^3–C^{25}	1.335(4)	C^5–C^{25}	1.547(5)	C^{19}–C^{20}	1.385(4)
F^4–C^{26}	1.329(4)	C^5–C^{26}	1.536(5)	C^{19}–C^{24}	1.398(5)
F^5–C^{26}	1.328(4)	C^7–C^8	1.530(5)	C^{20}–C^{21}	1.383(5)
F^6–C^{26}	1.337(4)	C^7–C^9	1.547(6)	C^{21}–C^{22}	1.376(5)
O^1–C^7	1.436(4)	C^7–C^{10}	1.500(5)	C^{22}–C^{23}	1.366(5)
O^2–C^3	1.450(3)	C^8–C^{11}	1.488(6)	C^{23}–C^{24}	1.375(5)

Table S5 – Bond Angles (Degrees) for molecule **14**: k53n_fin1 P b c a R = 0.05

Bond angle	φ	Bond angle	φ	Bond angle	φ
O^1–P^1–O^2	92.1(1)	O^7–C^4–C^3	100.4(2)	F^4–C^{26}–C^5	111.9(3)
O^1–P^1–O^3	92.4(1)	O^7–C^4–C^5	99.0(2)	F^5–C^{26}–F^6	107.6(3)
O^1–P^1–O^6	91.6(1)	O^7–C^4–C^{19}	112.4(2)	C^4–C^5–C^{26}	112.6(2)
O^1–P^1–O^7	177.5(1)	C^3–C^4–C^5	110.6(2)	C^{25}–C^5–C^{26}	110.0(3)
O^2–P^1–O^3	126.4(1)	C^3–C^4–C^{19}	114.4(2)	O^1–C^7–C^8	103.3(3)
O^2–P^1–O^6	105.8(1)	C^5–C^4–C^{19}	117.6(2)	O^1–C^7–C^9	106.9(3)
O^2–P^1–O^7	90.4(1)	O^6–C^5–C^4	104.6(2)	O^1–C^7–C^{10}	108.0(3)
O^3–P^1–O^6	127.4(1)	O^6–C^5–C^{25}	105.5(2)	C^8–C^7–C^9	112.3(3)
O^3–P^1–O^7	86.1(1)	O^6–C^5–C^{26}	107.4(2)	C^8–C^7–C^{10}	117.8(3)
O^6–P^1–O^7	87.8(1)	C^4–C^5–C^{25}	116.0(2)	C^9–C^7–C^{10}	107.9(3)
P^1–O^1–C^7	116.0(2)	F^1–C^{25}–F^2	107.1(3)	O^3–C^8–C^7	103.4(3)
P^1–O^2–C^3	112.7(2)	F^1–C^{25}–F^3	106.2(3)	O^3–C^8–C^{11}	108.3(3)

P^1–O^3–C^8	115.7(2)	F^1–C^{25}–C^5	111.7(3)	O^3–C^8–C^{12}	103.6(3)
P^1–O^6–C^5	110.5(2)	F^2–C^{25}–F^3	107.1(3)	C^7–C^8–C^{11}	117.2(4)
P^1–O^7–C^4	101.5(2)	F^2–C^{25}–C^5	112.0(3)	C^7–C^8–C^{12}	111.0(3)
O^2–C^3–C^4	102.5(2)	F^3–C^{25}–C^5	112.4(3)	F^5–C^{26}–C^5	111.8(3)
O^2–C^3–C^{13}	110.2(2)	F^4–C^{26}–F^5	106.3(3)	F^6–C^{26}–C^5	111.1(3)
C^4–C^3–C^{13}	115.0(2)	F^4–C^{26}–F^6	107.9(3)	C^{11}–C^8–C^{12}	112.0(4)

Table S6 – Torsion Angles (Degrees) for molecule **14**: k53n_fin1 P b c a R = 0.05

Torsion angle	τ	Torsion angle	τ	Torsion angle	τ
O^2–P^1–O^1–C^7	–135.6(2)	P^1–O^6–C^5–C^4	7.3(2)	O^7–C^4–C^{19}–C^{24}	–167.3(3)
O^3–P^1–O^1–C^7	–9.0(2)	P^1–O^6–C^5–C^{25}	–115.5(2)	C^3–C^4–C^{19}–C^{20}	123.7(3)
O^6–P^1–O^1–C^7	118.5(2)	P^1–O^6–C^5–C^{26}	127.2(2)	C^3–C^4–C^{19}–C^{24}	–53.6(4)
O^1–P^1–O^2–C^3	–156.1(2)	P^1–O^7–C^4–C^3	56.8(2)	C^5–C^4–C^{19}–C^{20}	–103.9(3)
O^3–P^1–O^2–C^3	109.3(2)	P^1–O^7–C^4–C^5	–56.3(3)	C^5–C^4–C^{19}–C^{24}	78.8(3)
O^6–P^1–O^2–C^3	–63.9(2)	P^1–O^7–C^4–C^{19}	178.8(2)	O^6–C^5–C^{25}–F^1	161.4(2)
O^7–P^1–O^2–C^3	23.9(2)	O^2–C^3–C^4–O^7	–40.0(2)	O^6–C^5–C^{25}–F^2	41.3(3)
O^1–P^1–O^3–C^8	–12.0(2)	O^2–C^3–C^4–C^5	63.8(3)	O^6–C^5–C^{25}–F^3	–79.3(3)
O^2–P^1–O^3–C^8	82.4(2)	O^2–C^3–C^4–C^{19}	–160.6(2)	C^4–C^5–C^{25}–F^2	–74.0(3)
O^6–P^1–O^3–C^8	–105.9(2)	C^{13}–C^3–C^4–O^7	79.7(3)	C^4–C^5–C^{25}–F^3	165.5(3)
O^7–P^1–O^3–C^8	170.0(2)	C^{13}–C^3–C^4–C^5	–176.6(2)	C^{26}–C^5–C^{25}–F^1	–83.1(3)
O^1–P^1–O^6–C^5	146.8(2)	C^{13}–C^3–C^4–C^{19}	–40.9(3)	C^{26}–C^5–C^{25}–F^2	156.8(3)
O^2–P^1–O^6–C^5	54.1(2)	O^2–C^3–C^{13}–C^{14}	44.4(4)	C^{26}–C^5–C^{25}–F^3	36.2(4)
O^3–P^1–O^6–C^5	–119.0(2)	O^2–C^3–C^{13}–C^{18}	–132.7(3)	O^6–C^5–C^{26}–F^4	45.2(3)
O^7–P^1–O^6–C^5	–35.7(2)	C^4–C^3–C^{13}–C^{14}	–70.9(4)	O^6–C^5–C^{26}–F^5	–73.9(3)
O^2–P^1–O^7–C^4	–49.3(2)	C^4–C^3–C^{13}–C^{18}	112.0(3)	O^6–C^5–C^{26}–F^6	165.9(2)
O^3–P^1–O^7–C^4	–175.7(2)	O^7–C^4–C^5–O^6	31.6(2)	C^4–C^5–C^{26}–F^4	159.9(3)
O^6–P^1–O^7–C^4	56.6(2)	O^7–C^4–C^5–C^{25}	147.4(2)	C^4–C^5–C^{26}–F^5	40.8(3)
P^1–O^1–C^7–C^8	24.9(3)	O^7–C^4–C^5–C^{26}	–84.7(3)	C^4–C^5–C^{26}–F^6	–79.4(3)
P^1–O^1–C^7–C^9	–93.7(3)	C^3–C^4–C^5–O^6	–73.1(2)	C^{25}–C^5–C^{26}–F^4	–69.1(3)
P^1–O^1–C^7–C^{10}	150.4(3)	C^3–C^4–C^5–C^{25}	42.6(3)	C^{25}–C^5–C^{26}–F^5	171.8(3)
P^1–O^2–C^3–C^4	5.6(2)	C^3–C^4–C^5–C^{26}	170.5(2)	C^{25}–C^5–C^{26}–F^6	51.6(3)
P^1–O^2–C^3–C^{13}	–117.3(2)	C^{19}–C^4–C^5–O^6	152.8(2)	O^1–C^7–C^8–O^3	–29.6(3)
P^1–O^3–C^8–C^7	27.1(3)	C^{19}–C^4–C^5–C^{25}	–91.5(3)	O^1–C^7–C^8–C^{11}	–148.7(3)
P^1–O^3–C^8–C^{11}	152.1(3)	C^{19}–C^4–C^5–C^{26}	36.5(3)	O^1–C^7–C^8–C^{12}	80.9(4)
P^1–O^3–C^8–C^{12}	–88.8(3)	O^7–C^4–C^{19}–C^{20}	10.0(4)	C^9–C^7–C^8–O^3	85.2(4)

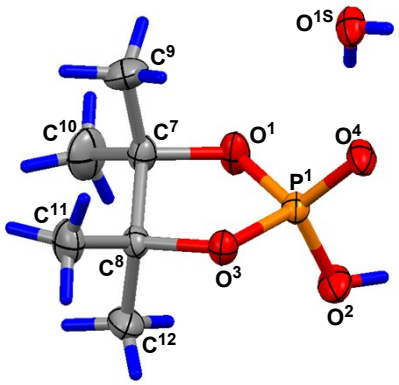


Table S7 – Bond Distances (Angstrom) for molecule **15**: k60_fin2 P 21 21 21 R = 0.05

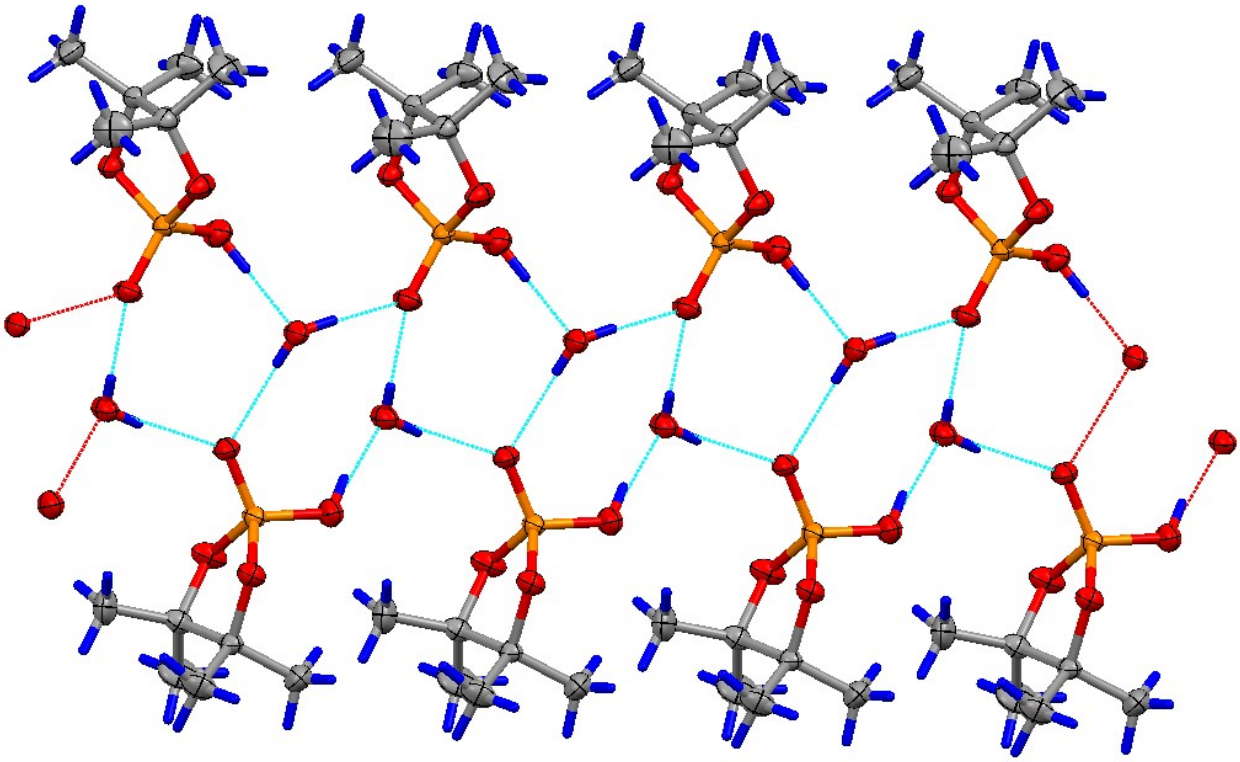
Bond	*d*	Bond	*d*	Bond	*d*
$P^1–O^1$	1.568(6)	$O^3–C^8$	1.489(8)	$C^7–C^9$	1.504(12)
$P^1–O^2$	1.533(7)	$O^2–H^2$	0.86(7)	$C^7–C^{10}$	1.533(11)
$P^1–O^3$	1.572(6)	$O^1S–H^1S$	0.72(9)	$C^8–C^{11}$	1.510(11)
$P^1–O^4$	1.477(6)	$O^1S–H^2S$	0.83(10)	$C^8–C^{12}$	1.512(11)
$O^1–C^7$	1.477(9)	$C^7–C^8$	1.557(9)		

Table S8 – Bond Angles (Degrees) for molecule **15**: k60_fin2 P 21 21 21 R = 0.05

Bond angle	φ	Bond angle	φ	Bond angle	φ
$O^1–P^1–O^2$	108.5(3)	$P^1–O^3–C^8$	110.0(4)	$O^3–C^8–C^{11}$	106.5(5)
$O^1–P^1–O^3$	98.6(2)	$O^1–C^7–C^9$	109.0(6)	$O^3–C^8–C^{12}$	107.0(5)
$O^1–P^1–O^4$	115.7(3)	$O^1–C^7–C^8$	102.7(5)	$C^7–C^8–C^{12}$	116.0(6)
$O^2–P^1–O^3$	107.2(3)	$C^8–C^7–C^{10}$	111.9(6)	$C^{11}–C^8–C^{12}$	110.1(6)
$O^2–P^1–O^4$	113.5(3)	$O^1–C^7–C^{10}$	106.2(6)	$C^7–C^8–C^{11}$	114.1(6)
$O^3–P^1–O^4$	112.1(3)	$C^8–C^7–C^9$	114.4(6)	$O^3–C^8–C^7$	102.2(4)
$P^1–O^1–C^7$	112.1(4)	$C^9–C^7–C^{10}$	111.9(6)		

Table S9 – Torsion Angles (Degrees) for molecule **15**: k60_fin2 P 21 21 21 R = 0.05

Torsion angle	τ	Torsion angle	τ	Torsion angle	τ
$O^2–P^1–O^1–C^7$	117.6(5)	$P^1–O^1–C^7–C^9$	94.7(6)	$O^1–C^7–C^8–C^{12}$	–79.0(6)
$O^3–P^1–O^1–C^7$	6.1(5)	$P^1–O^1–C^7–C^{10}$	–144.6(5)	$C^9–C^7–C^8–O^3$	–81.0(6)
$O^4–P^1–O^1–C^7$	–113.5(5)	$P^1–O^3–C^8–C^7$	–35.4(6)	$C^9–C^7–C^8–C^{11}$	33.6(8)
$O^1–P^1–O^3–C^8$	18.8(4)	$P^1–O^3–C^8–C^{11}$	–155.4(4)	$C^9–C^7–C^8–C^{12}$	163.1(6)
$O^2–P^1–O^3–C^8$	–93.7(4)	$P^1–O^3–C^8–C^{12}$	86.9(5)	$C^{10}–C^7–C^8–O^3$	150.4(6)
$O^4–P^1–O^3–C^8$	141.1(4)	$O^1–C^7–C^8–O^3$	36.9(6)	$C^{10}–C^7–C^8–C^{11}$	–95.0(8)
$P^1–O^1–C^7–C^8$	–27.0(6)	$O^1–C^7–C^8–C^{11}$	151.5(6)	$C^{10}–C^7–C^8–C^{12}$	34.5(8)



H-bonding in a crystal of compound **15**.

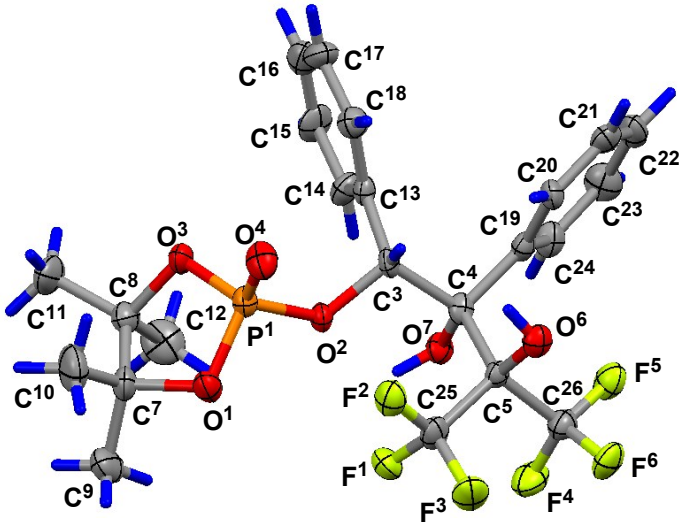


Table S10 – Bond Distances (Angstrom) for molecule **16**: shelx P –1 R = 0.09

Bond	*d*	Bond	*d*	Bond	*d*
P^1–O^1	1.582(6)	O^6–C^5	1.385(10)	C^8–C^{11}	1.514(15)
P^1–O^2	1.581(6)	O^7–C^4	1.423(9)	C^8–C^{12}	1.544(15)
P^1–O^3	1.583(7)	O^6–H^6	0.66(6)	C^{13}–C^{18}	1.377(12)
P^1–O^4	1.460(6)	O^7–H^7	0.87(8)	C^{13}–C^{14}	1.384(13)
F^1–C^{25}	1.334(12)	C^3–C^4	1.573(11)	C^{14}–C^{15}	1.375(14)
F^2–C^{25}	1.338(12)	C^3–C^{13}	1.501(12)	C^{15}–C^{16}	1.360(16)
F^3–C^{25}	1.352(12)	C^4–C^{19}	1.542(10)	C^{16}–C^{17}	1.362(17)
F^4–C^{26}	1.327(10)	C^4–C^5	1.636(12)	C^{17}–C^{18}	1.418(16)
F^5–C^{26}	1.331(11)	C^5–C^{25}	1.566(12)	C^{19}–C^{24}	1.403(12)
F^6–C^{26}	1.335(11)	C^5–C^{26}	1.586(11)	C^{19}–C^{20}	1.377(11)
O^1–C^7	1.501(11)	C^7–C^9	1.527(15)	C^{20}–C^{21}	1.376(12)
O^2–C^3	1.478(9)	C^7–C^8	1.536(13)	C^{21}–C^{22}	1.394(14)
O^3–C^8	1.494(10)	C^7–C^{10}	1.518(16)	C^{22}–C^{23}	1.369(15)

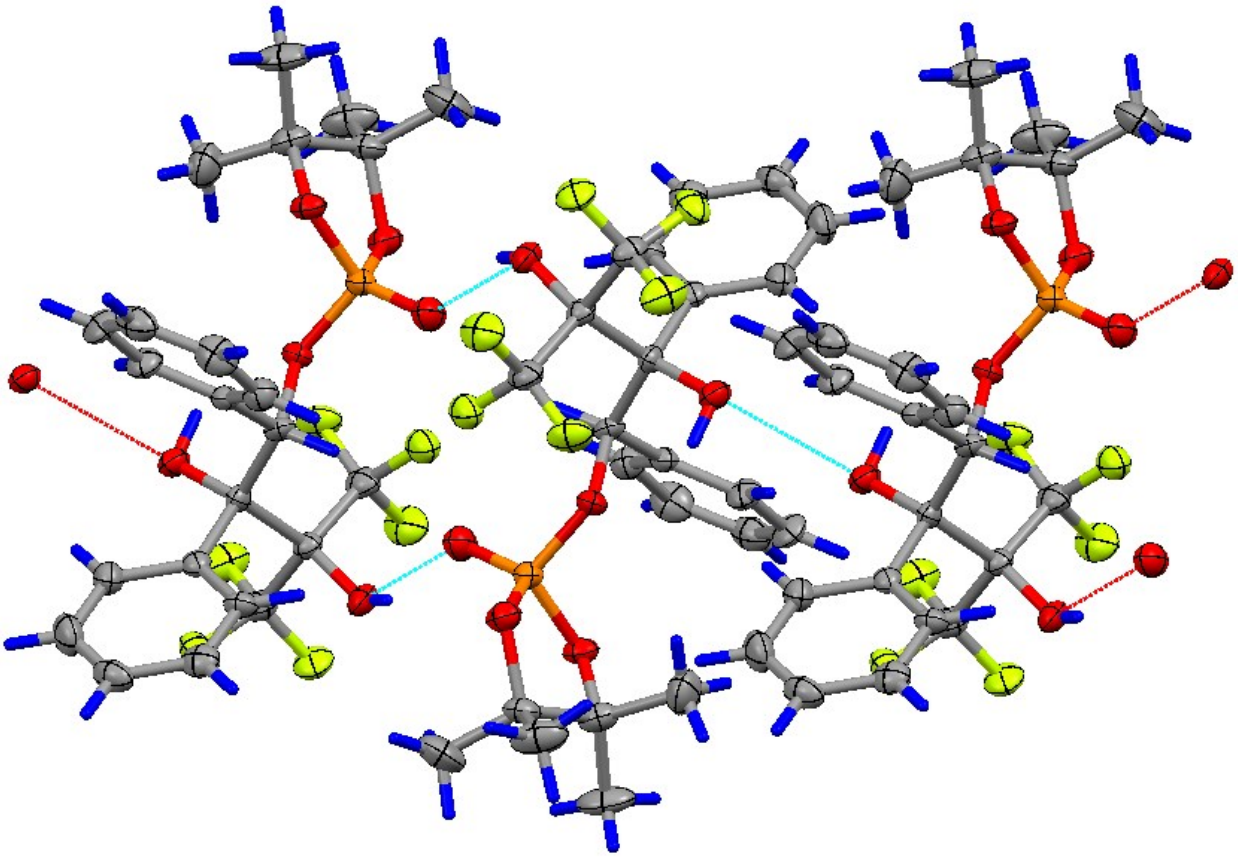
Table S11 – Bond Angles (Degrees) for molecule **16**: shelx P –1 R = 0.09

Bond angle	φ	Bond angle	φ	Bond angle	φ
O^1–P^1–O^2	101.2(3)	C^5–C^4–C^{19}	110.2(6)	F^4–C^{26}–C^5	111.6(7)
O^1–P^1–O^3	99.6(3)	C^3–C^4–C^5	113.0(6)	F^5–C^{26}–C^5	113.0(7)
O^1–P^1–O^4	118.9(4)	O^7–C^4–C^3	111.0(6)	F^6–C^{26}–C^5	110.4(7)
O^2–P^1–O^3	109.6(3)	O^6–C^5–C^{26}	103.5(6)	F^5–C^{26}–F^6	106.1(7)
O^2–P^1–O^4	112.7(3)	C^4–C^5–C^{25}	113.8(6)	F^4–C^{26}–F^5	107.4(7)
O^3–P^1–O^4	113.4(4)	C^4–C^5–C^{26}	109.5(6)	F^1–C^{25}–F^3	107.1(8)
P^1–O^1–C^7	109.6(5)	C^{25}–C^5–C^{26}	107.2(7)	F^1–C^{25}–F^2	108.7(7)
P^1–O^2–C^3	121.0(4)	O^6–C^5–C^{25}	108.2(7)	C^8–C^7–C^{10}	114.7(9)
P^1–O^3–C^8	110.3(5)	O^6–C^5–C^4	114.0(6)	C^9–C^7–C^{10}	109.4(8)
C^5–O^6–H^6	139(6)	O^1–C^7–C^9	106.1(8)	O^1–C^7–C^{10}	107.1(8)
O^2–C^3–C^{13}	106.8(6)	O^1–C^7–C^8	103.6(6)	C^8–C^7–C^9	115.1(8)
O^2–C^3–C^4	106.4(6)	F^2–C^{25}–C^5	110.3(7)	O^3–C^8–C^{12}	105.9(7)
C^4–C^3–C^{13}	115.7(6)	F^1–C^{25}–C^5	114.6(8)	O^3–C^8–C^7	103.4(7)
O^7–C^4–C^5	108.0(6)	F^2–C^{25}–F^3	105.7(8)	O^3–C^8–C^{11}	106.3(8)

O^7–C^4–C^{19}	108.3(6)	F^3–C^{25}–C^5	110.0(7)	C^7–C^8–C^{12}	113.3(9)
C^3–C^4–C^{19}	106.2(6)	F^4–C^{26}–F^6	108.1(7)	C^7–C^8–C^{11}	115.4(8)

Table S12 – Torsion Angles (Degrees) for molecule **16**: shelx P –1 R = 0.09

Torsion angle	τ	Torsion angle	τ	Torsion angle	τ
O^2–P^1–O^1–C^7	–125.3(5)	C^{13}–C^3–C^4–C^{19}	45.2(9)	C^4–C^5–C^{25}–F^1	47.2(10)
O^3–P^1–O^1–C^7	–12.9(5)	O^2–C^3–C^{13}–C^{14}	–61.9(9)	C^4–C^5–C^{25}–F^2	–75.8(9)
O^4–P^1–O^1–C^7	110.7(6)	O^2–C^3–C^{13}–C^{18}	110.2(8)	C^{26}–C^5–C^{25}–F^1	–74.0(9)
O^1–P^1–O^2–C^3	–159.1(6)	C^4–C^3–C^{13}–C^{14}	56.3(10)	C^{26}–C^5–C^{25}–F^2	163.0(7)
O^3–P^1–O^2–C^3	96.4(6)	C^4–C^3–C^{13}–C^{18}	–131.6(8)	O^6–C^5–C^{26}–F^4	159.7(7)
O^4–P^1–O^2–C^3	–31.0(7)	O^7–C^4–C^5–O^6	165.2(6)	O^6–C^5–C^{26}–F^5	–79.1(8)
O^1–P^1–O^3–C^8	–11.1(5)	O^7–C^4–C^5–C^{25}	–70.1(8)	O^6–C^5–C^{26}–F^6	39.5(9)
O^2–P^1–O^3–C^8	94.6(5)	O^7–C^4–C^5–C^{26}	49.9(8)	C^4–C^5–C^{26}–F^4	–78.4(8)
O^4–P^1–O^3–C^8	–138.5(5)	C^3–C^4–C^5–O^6	–71.6(8)	C^4–C^5–C^{26}–F^5	42.8(9)
P^1–O^1–C^7–C^8	31.4(7)	C^3–C^4–C^5–C^{25}	53.1(8)	C^4–C^5–C^{26}–F^6	161.4(6)
P^1–O^1–C^7–C^9	153.0(6)	C^3–C^4–C^5–C^{26}	173.0(6)	C^{25}–C^5–C^{26}–F^4	45.5(10)
P^1–O^1–C^7–C^{10}	–90.2(8)	C^{19}–C^4–C^5–O^6	47.0(8)	C^{25}–C^5–C^{26}–F^5	166.6(7)
P^1–O^2–C^3–C^4	163.2(5)	C^{19}–C^4–C^5–C^{25}	171.8(6)	C^{25}–C^5–C^{26}–F^6	–74.8(8)
P^1–O^2–C^3–C^{13}	–72.6(7)	C^{19}–C^4–C^5–C^{26}	–68.3(7)	O^1–C^7–C^8–O^3	–37.0(8)
P^1–O^3–C^8–C^7	30.2(7)	O^7–C^4–C^{19}–C^{20}	175.3(8)	O^1–C^7–C^8–C^{11}	–152.5(8)
P^1–O^3–C^8–C^{11}	152.1(7)	O^7–C^4–C^{19}–C^{24}	–1.5(10)	O^1–C^7–C^8–C^{12}	77.2(9)
P^1–O^3–C^8–C^{12}	–89.3(8)	C^3–C^4–C^{19}–C^{20}	56.0(10)	C^9–C^7–C^8–O^3	–152.3(8)
O^2–C^3–C^4–O^7	46.1(8)	C^3–C^4–C^{19}–C^{24}	–120.8(8)	C^9–C^7–C^8–C^{11}	92.1(11)
O^2–C^3–C^4–C^5	–75.4(7)	C^5–C^4–C^{19}–C^{20}	–66.7(9)	C^9–C^7–C^8–C^{12}	–38.1(11)
O^2–C^3–C^4–C^{19}	163.6(6)	C^5–C^4–C^{19}–C^{24}	116.5(8)	C^{10}–C^7–C^8–O^3	79.4(9)
C^{13}–C^3–C^4–O^7	–72.3(8)	O^6–C^5–C^{25}–F^1	175.0(7)	C^{10}–C^7–C^8–C^{11}	–36.2(12)
C^{13}–C^3–C^4–C^5	166.2(6)	O^6–C^5–C^{25}–F^2	52.0(9)	C^{10}–C^7–C^8–C^{12}	–166.4(9)



H-bonding in a crystal of compound **16**.

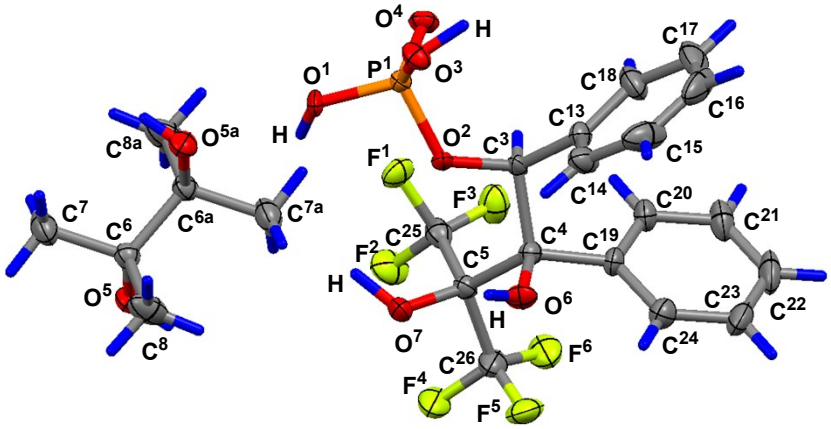


Table S13 – Bond Distances (Angstrom) for solvate **18** of molecule **17** with pinacol: k66_f2 P 21/c R = 0.08

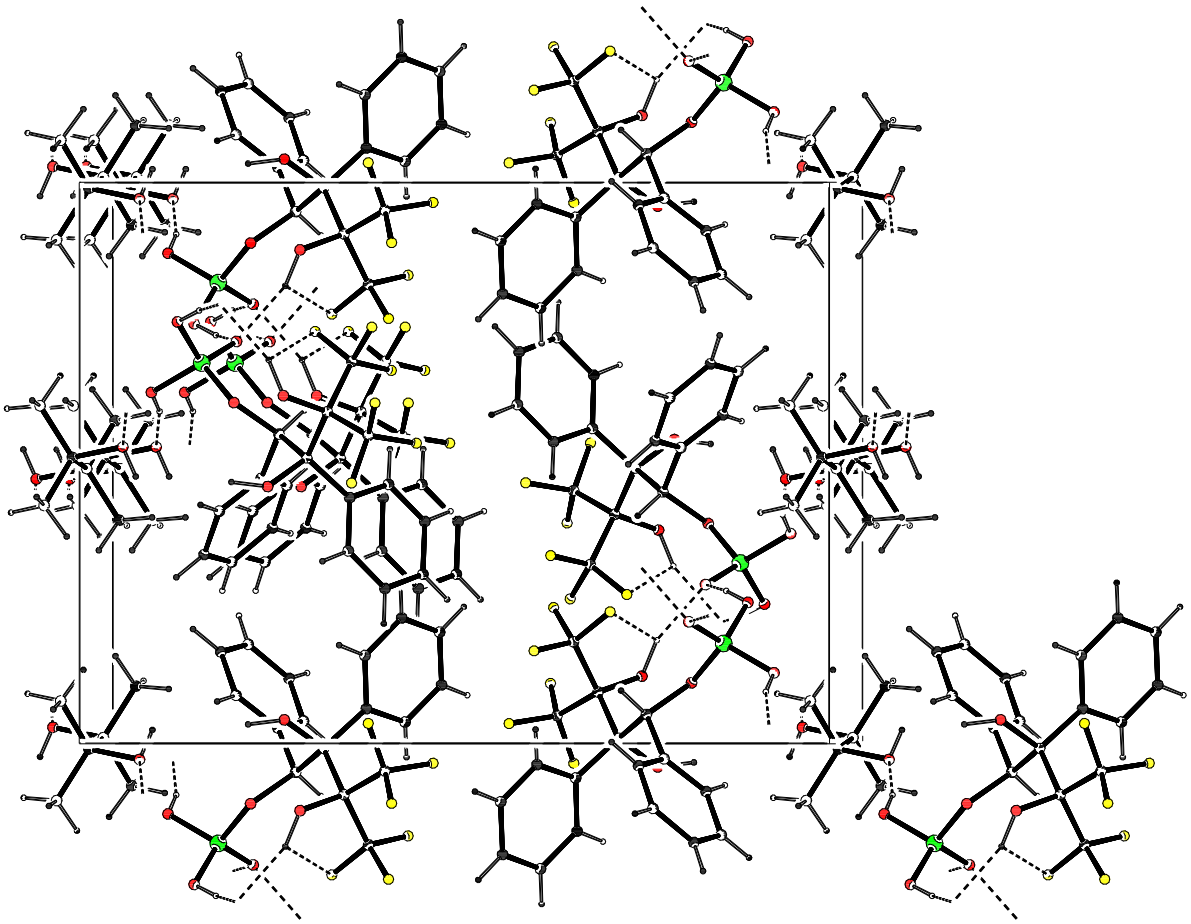
Bond	*d*	Bond	*d*	Bond	*d*
P^1–O^1	1.535(5)	O^6–C^4	1.416(7)	C^{19}–C^{24}	1.395(9)
P^1–O^2	1.588(4)	O^7–C^5	1.421(8)	C^{20}–C^{21}	1.380(11)
P^1–O^3	1.551(4)	O^5–C^6	1.462(8)	C^{22}–C^{23}	1.373(13)
P^1–O^4	1.489(4)	C^3–C^{13}	1.530(8)	C^6–C^8	1.540(9)
F^1–C^{25}	1.348(8)	C^3–C^4	1.573(8)	C^6–C^{6a}	1.565(9)
F^2–C^{25}	1.347(8)	C^4–C^{19}	1.528(9)	C^6–C^7	1.527(9)
F^3–C^{25}	1.322(8)	C^4–C^5	1.635(8)	C^5–C^{26}	1.554(10)
F^4–C^{26}	1.341(9)	C^5–C^{25}	1.533(9)	C^{15}–C^{16}	1.370(15)
F^5–C^{26}	1.349(9)	C^{13}–C^{14}	1.372(9)	C^{17}–C^{18}	1.399(12)
F^6–C^{26}	1.334(10)	C^{13}–C^{18}	1.371(9)	C^{19}–C^{20}	1.389(10)
O^2–C^3	1.447(7)	C^{16}–C^{17}	1.380(14)	C^{23}–C^{24}	1.396(11)

Table S14 – Bond Angles (Degrees) for solvate **18** of molecule **17** with pinacol: k66_f2 P 21/c R = 0.08

Bond angle	φ	Bond angle	φ	Bond angle	φ
O^1–P^1–O^2	103.4(2)	O^6–C^4–C^3	107.9(4)	O^5–C^6–C^7	104.7(5)
O^1–P^1–O^3	102.6(3)	C^3–C^4–C^5	113.0(4)	C^{6a}–C^6–C^7	112.4(5)
O^1–P^1–O^4	116.9(3)	C^3–C^4–C^{19}	107.9(5)	C^{6a}–C^6–C^8	112.2(5)
O^2–P^1–O^3	109.1(2)	O^6–C^4–C^{19}	108.7(5)	C^7–C^6–C^8	111.0(5)
O^2–P^1–O^4	111.3(2)	C^5–C^4–C^{19}	115.5(5)	F^1–C^{25}–C^5	112.2(6)
O^3–P^1–O^4	112.8(2)	O^7–C^5–C^{26}	104.0(5)	F^2–C^{25}–F^3	105.7(6)
P^1–O^2–C^3	122.5(3)	O^7–C^5–C^4	108.8(4)	F^2–C^{25}–C^5	112.6(5)
C^4–C^{19}–C^{20}	122.7(5)	O^7–C^5–C^{25}	108.5(5)	F^1–C^{25}–F^3	106.8(5)
C^{20}–C^{19}–C^{24}	117.4(6)	C^{25}–C^5–C^{26}	108.3(5)	F^1–C^{25}–F^2	103.6(5)
C^4–C^{19}–C^{24}	119.8(6)	C^4–C^5–C^{25}	115.9(5)	F^3–C^{25}–C^5	115.1(6)
C^4–C^3–C^{13}	111.6(5)	C^4–C^5–C^{26}	110.7(5)	F^6–C^{26}–C^5	112.4(6)
O^2–C^3–C^4	106.8(4)	C^{14}–C^{13}–C^{18}	119.8(6)	F^4–C^{26}–F^5	105.7(6)
O^2–C^3–C^{13}	111.6(4)	O^5–C^6–C^8	109.1(5)	F^4–C^{26}–C^5	111.6(6)
O^6–C^4–C^5	103.6(4)	O^5–C^6–C^{6a}	107.0(4)	F^5–C^{26}–F^6	107.7(6)

Table S15 – Torsion Angles (Degrees) for solvate **18** of molecule **17** with pinacol: k66_f2 P 21/c R = 0.08

Torsion angle	τ	Torsion angle	τ	Torsion angle	τ
O^{1}–P^{1}–O^{2}–C^{3}	155.2(4)	O^{6}–C^{4}–C^{5}–C^{25}	156.0(5)	O^{7}–C^{5}–C^{25}–F^{3}	176.2(5)
O^{3}–P^{1}–O^{2}–C^{3}	–96.2(4)	O^{6}–C^{4}–C^{5}–C^{26}	–80.1(6)	C^{4}–C^{5}–C^{25}–F^{2}	174.8(5)
O^{4}–P^{1}–O^{2}–C^{3}	29.0(5)	C^{3}–C^{4}–C^{5}–O^{7}	–82.9(6)	C^{4}–C^{5}–C^{25}–F^{3}	53.5(7)
P^{1}–O^{2}–C^{3}–C^{4}	–168.4(3)	C^{3}–C^{4}–C^{5}–C^{25}	39.6(7)	C^{26}–C^{5}–C^{25}–F^{1}	166.0(5)
P^{1}–O^{2}–C^{3}–C^{13}	69.4(5)	C^{3}–C^{4}–C^{5}–C^{26}	163.4(5)	C^{26}–C^{5}–C^{25}–F^{3}	–71.6(7)
O^{2}–C^{3}–C^{4}–O^{6}	–55.0(5)	C^{19}–C^{4}–C^{5}–O^{7}	152.2(5)	O^{7}–C^{5}–C^{26}–F^{5}	–70.9(7)
O^{2}–C^{3}–C^{4}–C^{5}	58.8(6)	C^{19}–C^{4}–C^{5}–C^{25}	–85.3(7)	O^{7}–C^{5}–C^{26}–F^{6}	168.0(6)
O^{2}–C^{3}–C^{4}–C^{19}	–172.3(4)	C^{19}–C^{4}–C^{5}–C^{26}	38.6(7)	C^{4}–C^{5}–C^{26}–F^{5}	45.8(8)
C^{13}–C^{3}–C^{4}–O^{6}	67.2(6)	O^{6}–C^{4}–C^{19}–C^{20}	–168.2(6)	C^{25}–C^{5}–C^{26}–F^{4}	–68.1(7)
C^{13}–C^{3}–C^{4}–C^{5}	–178.9(4)	O^{6}–C^{4}–C^{19}–C^{24}	6.5(8)	C^{25}–C^{5}–C^{26}–F^{6}	52.8(8)
C^{13}–C^{3}–C^{4}–C^{19}	–50.1(6)	C^{3}–C^{4}–C^{19}–C^{20}	–51.5(8)	O^{5}–C^{6}–C^{6a}–O^{5a}	180.0(5)
O^{2}–C^{3}–C^{13}–C^{14}	48.8(7)	C^{3}–C^{4}–C^{19}–C^{24}	123.2(6)	O^{5}–C^{6}–C^{6a}–C^{8a}	60.4(6)
O^{2}–C^{3}–C^{13}–C^{18}	–130.8(6)	C^{5}–C^{4}–C^{19}–C^{20}	75.9(8)	C^{7}–C^{6}–C^{6a}–O^{5a}	65.6(6)
C^{4}–C^{3}–C^{13}–C^{14}	–70.6(7)	C^{5}–C^{4}–C^{19}–C^{24}	–109.3(7)	C^{7}–C^{6}–C^{6a}–C^{7a}	–180.0(5)
C^{4}–C^{3}–C^{13}–C^{18}	109.8(6)	O^{7}–C^{5}–C^{25}–F^{1}	53.8(7)	C^{8}–C^{6}–C^{6a}–O^{5a}	–60.4(6)
O^{6}–C^{4}–C^{5}–O^{7}	33.5(6)	O^{7}–C^{5}–C^{25}–F^{2}	–62.5(7)	C^{8}–C^{6}–C^{6a}–C^{8a}	180.0(5)



H-bonding in a crystal of solvate **18** of molecule **17** with pinacol, view along the 0*z* axis.

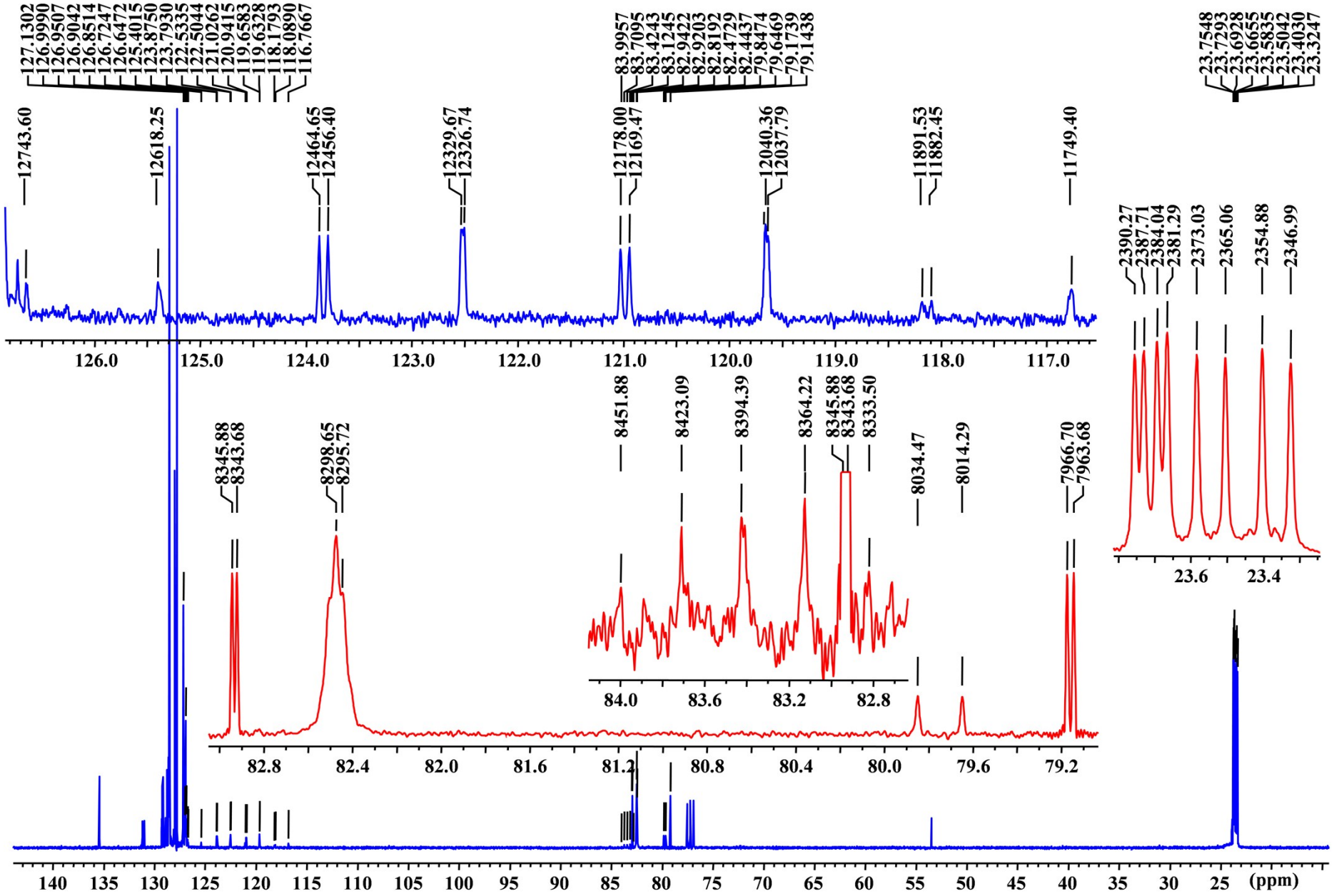


Figure 1. Full ^{13}C-{^{1}H} NMR spectrum (100.6 MHz, $CDCl_3/CH_2Cl_2$, 25°C) of phosphorane **14**.

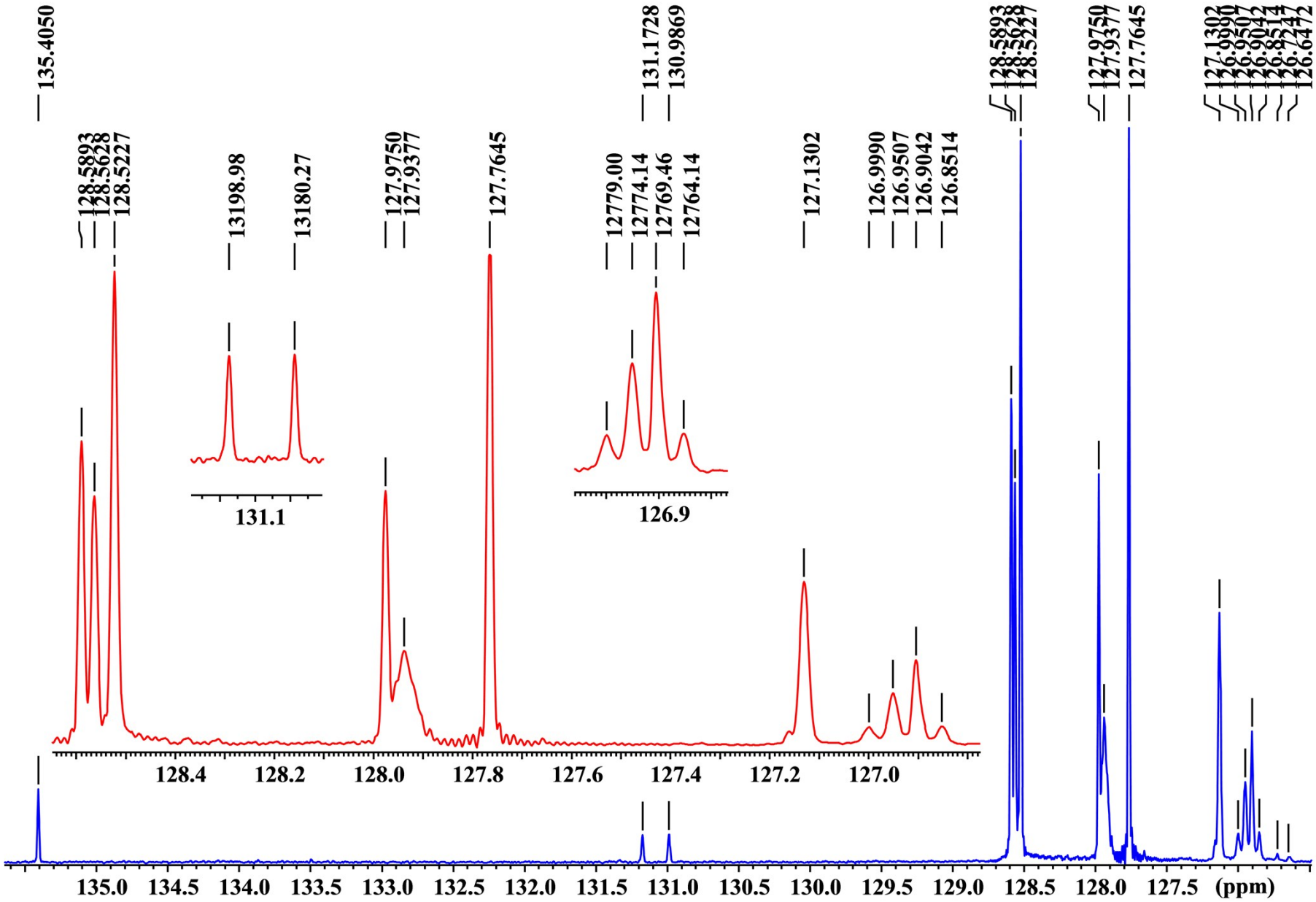


Figure 2. The fragment of ^{13}C-{^{1}H} NMR spectrum (100.6 MHz, $CDCl_3/CH_2Cl_2$, 25°C) of phosphorane **14** (the low-field region is shown).

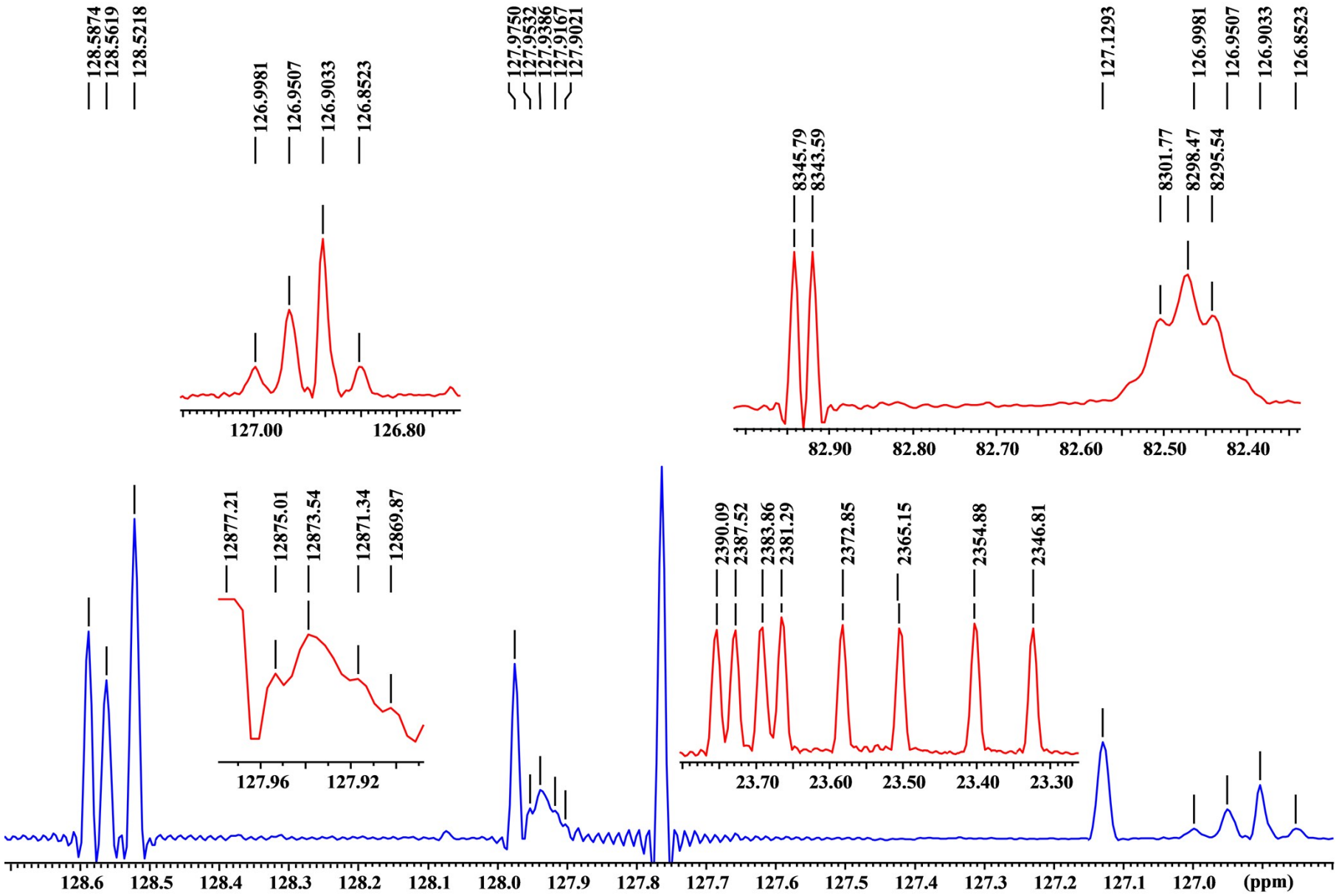


Figure 3. The fragments of ^{13}C-{^{1}H} NMR spectrum (100.6 MHz, $CDCl_3/CH_2Cl_2$, 25°C) of phosphorane **14** (the aromatic carbons and CH_3 groups regions are shown).

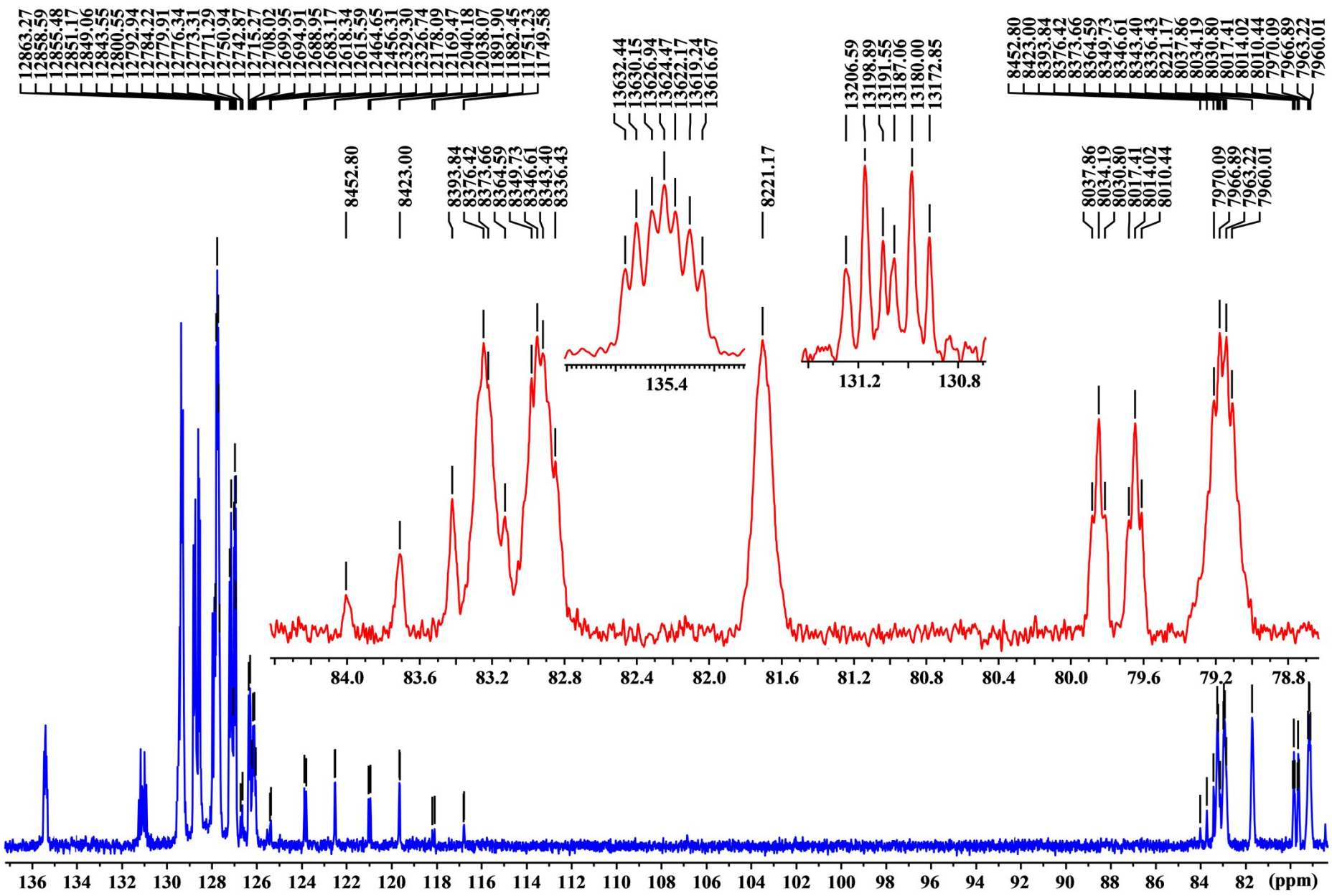


Figure 4. The fragments of ^{13}C NMR spectrum (100.6 MHz, $CDCl_3/CH_2Cl_2$, 25°C) of phosphorane **14**.

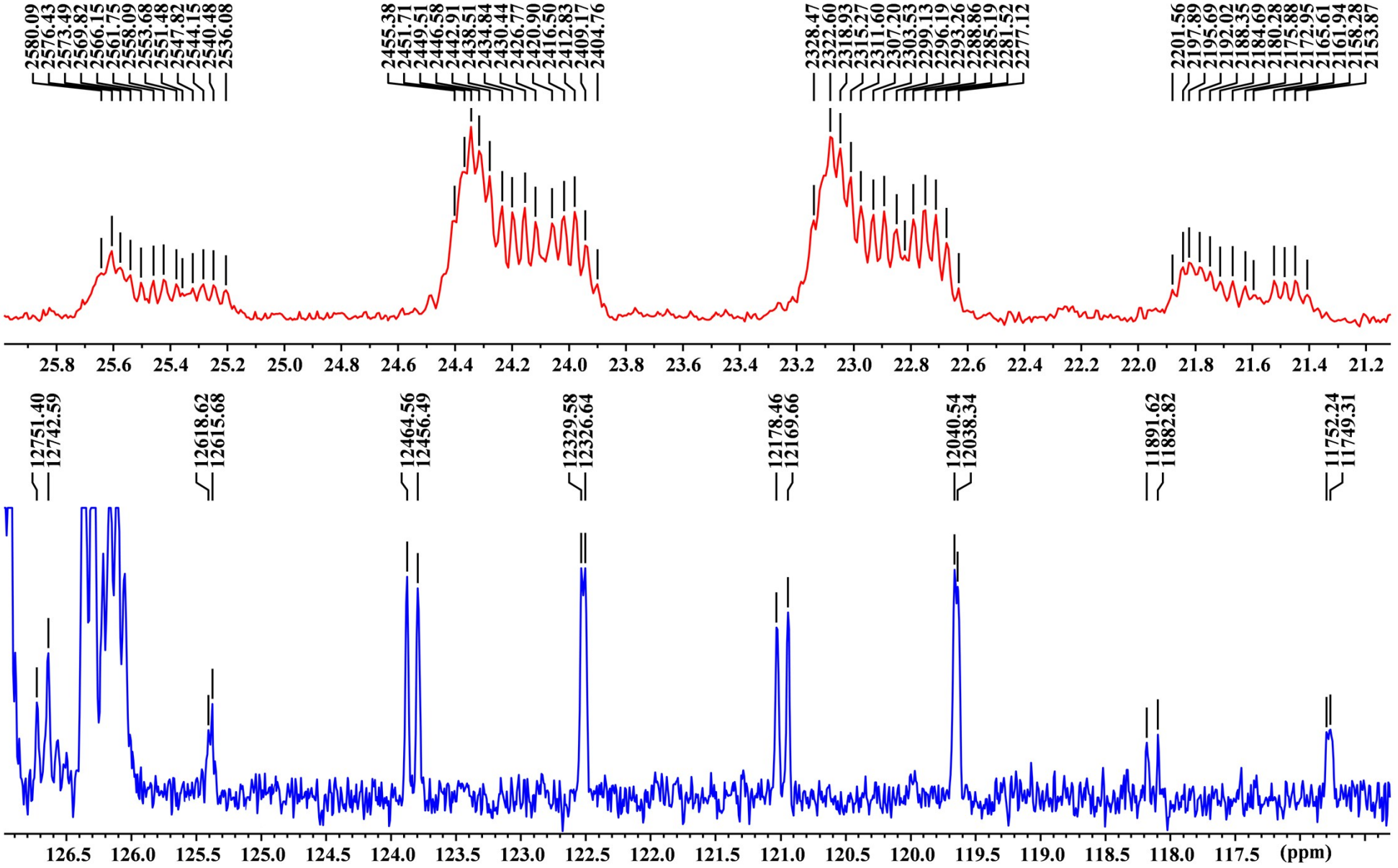


Figure 5. The fragments of ^{13}C NMR spectrum (100.6 MHz, $CDCl_3/CH_2Cl_2$, 25°C) of phosphorane **14** (the CF_3 and CH_3 groups regions are shown).

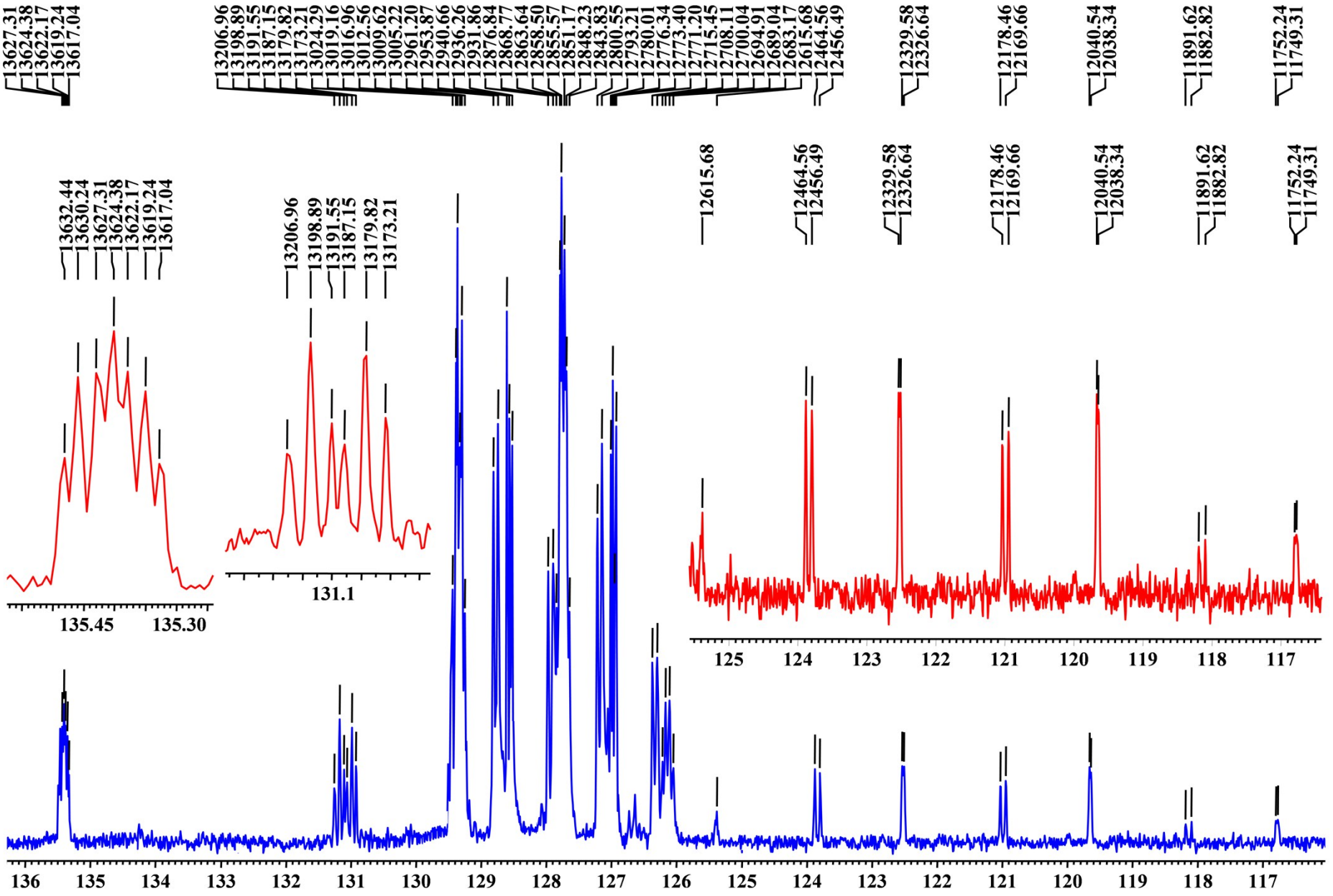


Figure 6. The low-field region of ^{13}C NMR spectrum (100.6 MHz, $CDCl_3/CH_2Cl_2$, 25°C) of phosphorane **14**.

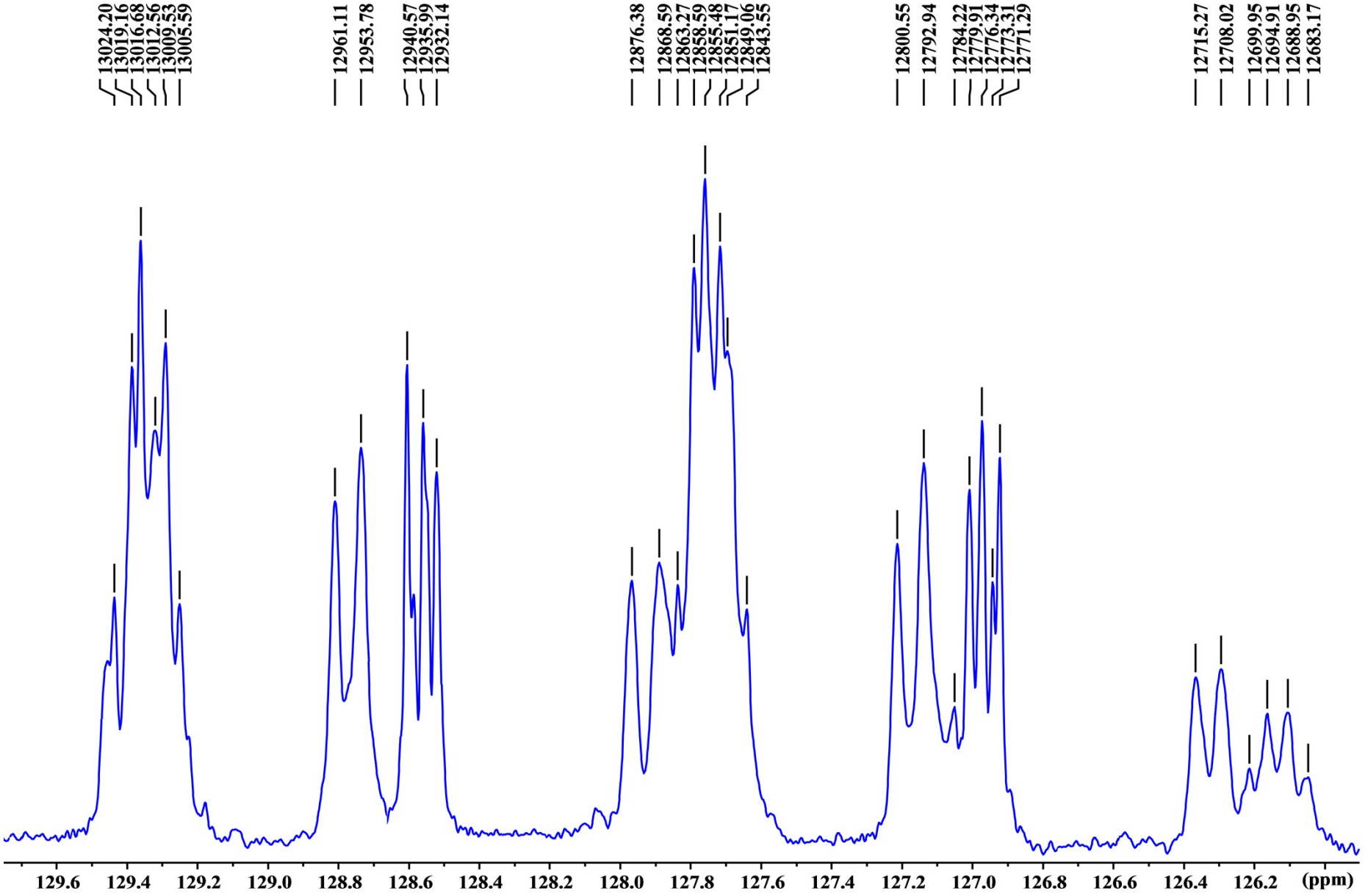


Figure 7. The aromatic carbons region of ^{13}C NMR NMR spectrum (100.6 MHz, $CDCl_3/CH_2Cl_2$, 25°C) of phosphorane **14**.

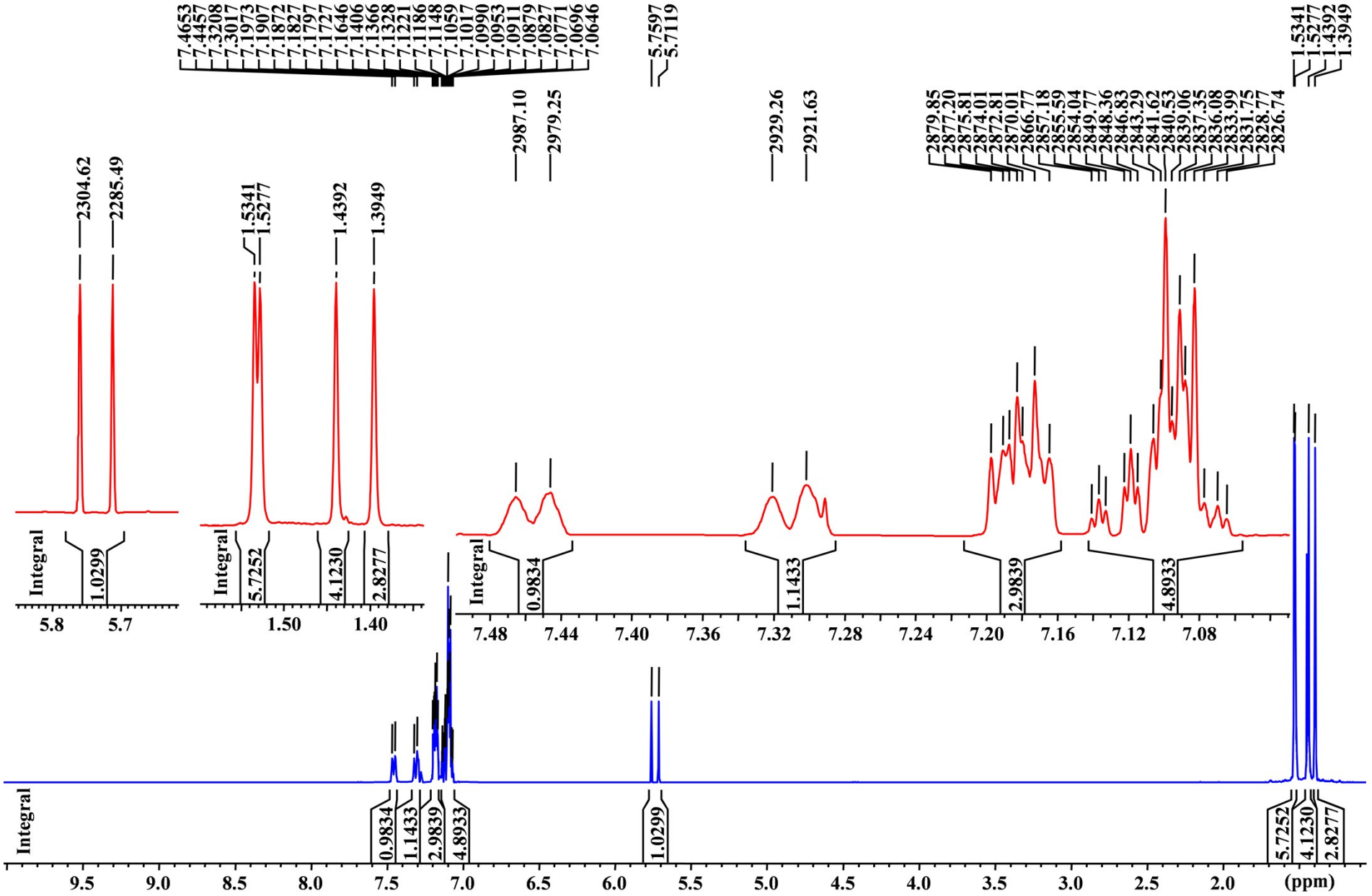


Figure 8. ^{1}H NMR spectrum (400 MHz, $CDCl_3$, 25°C) of phosphorane **14**.

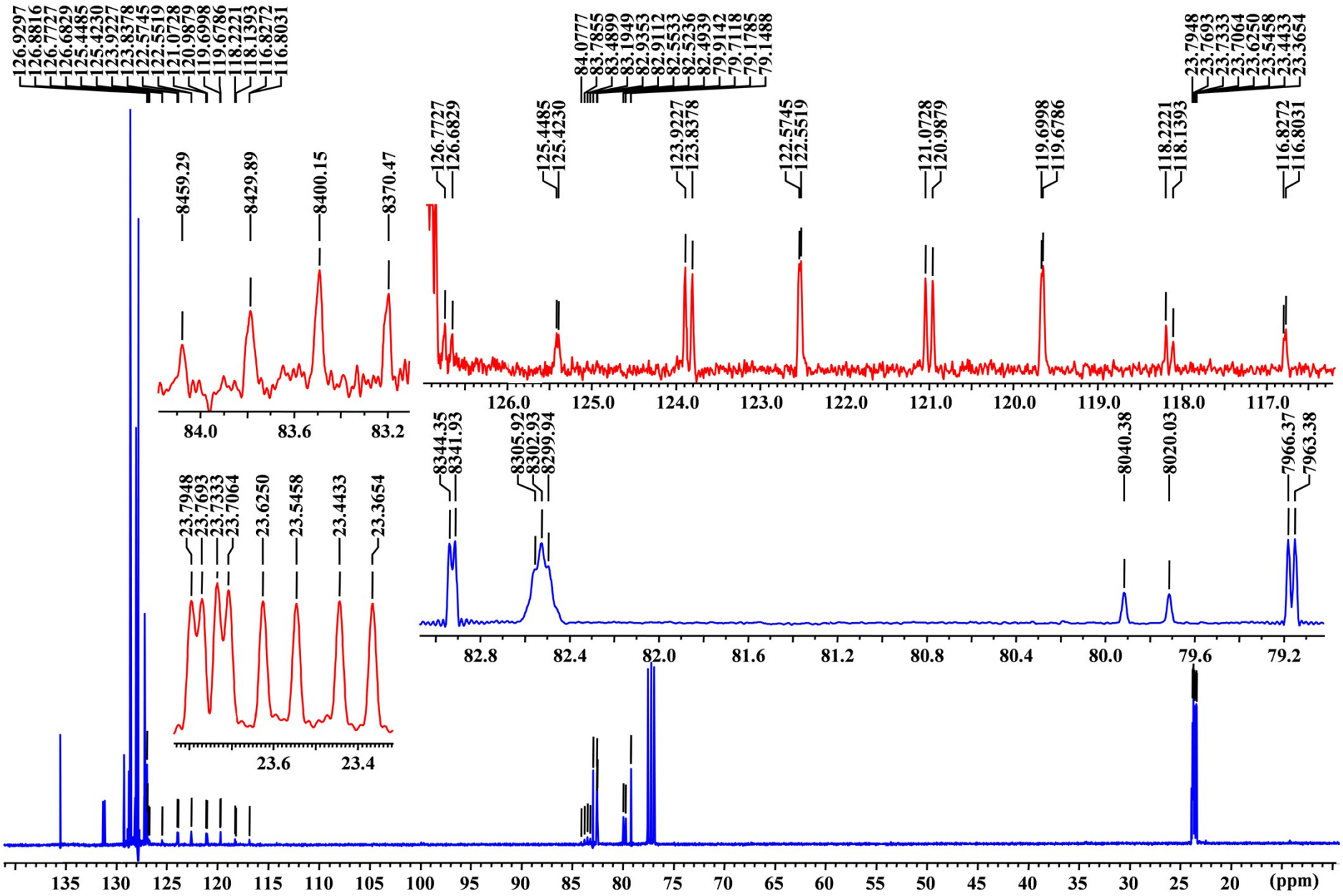


Figure 9. Full ^{13}C-{^{1}H} NMR spectrum (400 MHz, $CDCl_3$, 25°C) of phosphorane **14**.

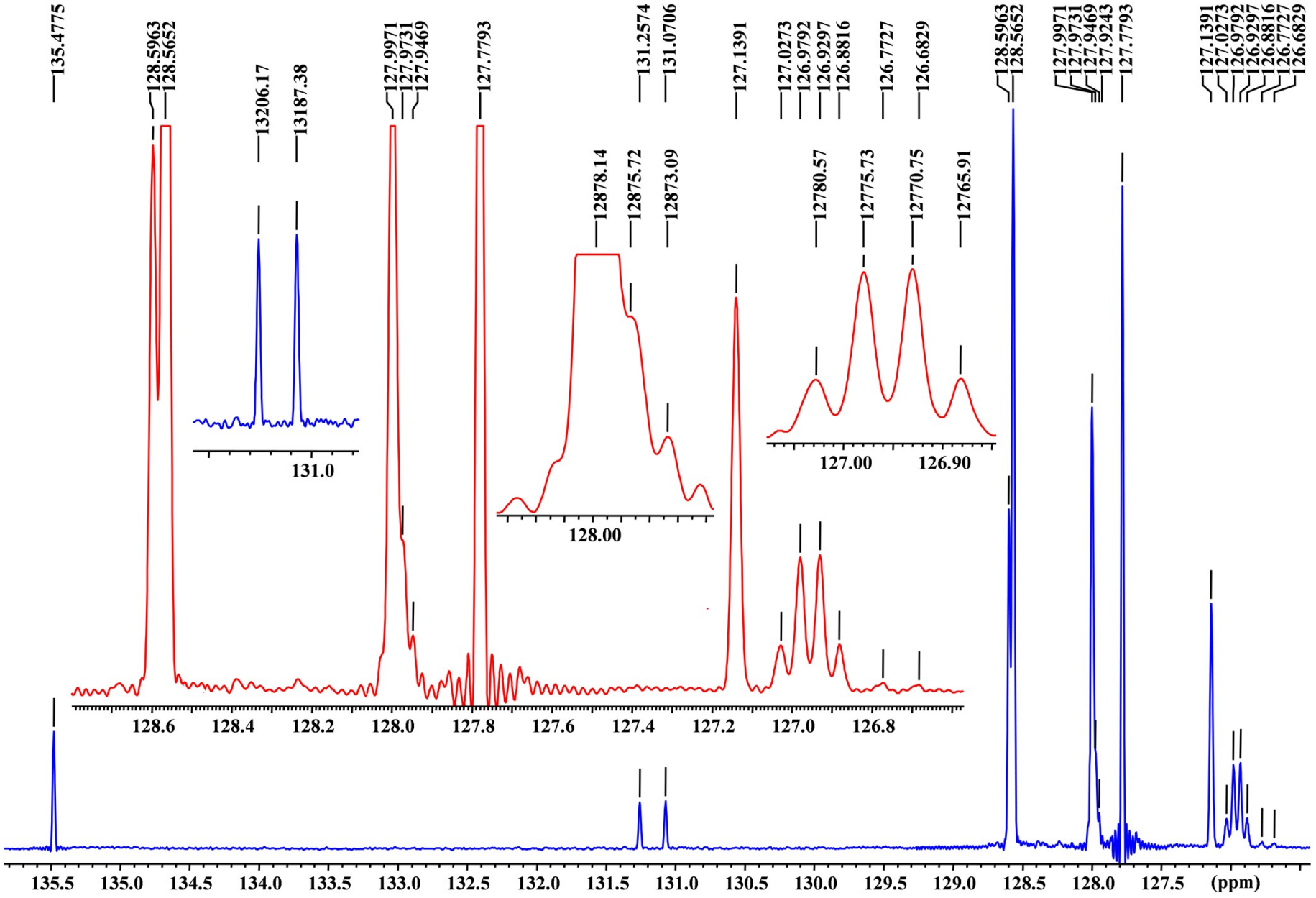


Figure 10. The low-field region of ^{13}C-{^{1}H} NMR spectrum (100.6 MHz, $CDCl_3/CH_2Cl_2$, 25°C) of phosphorane **14**.

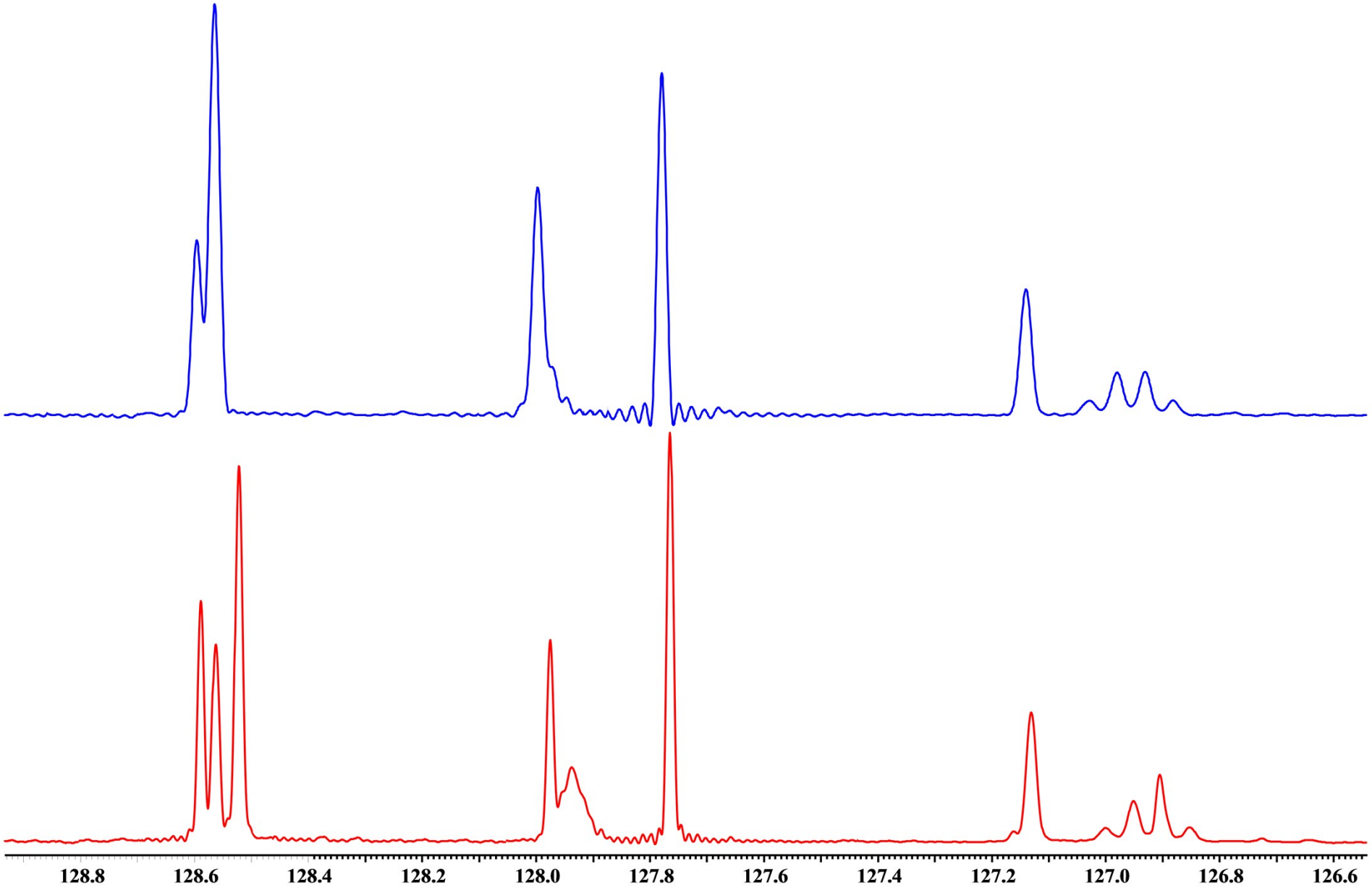


Figure 11. The aromatic carbons region of ^{13}C-{^{1}H} NMR spectra (100.6 MHz, $CDCl_3$, $CDCl_3/CH_2Cl_2$, 25°C) of phosphorane **14**.

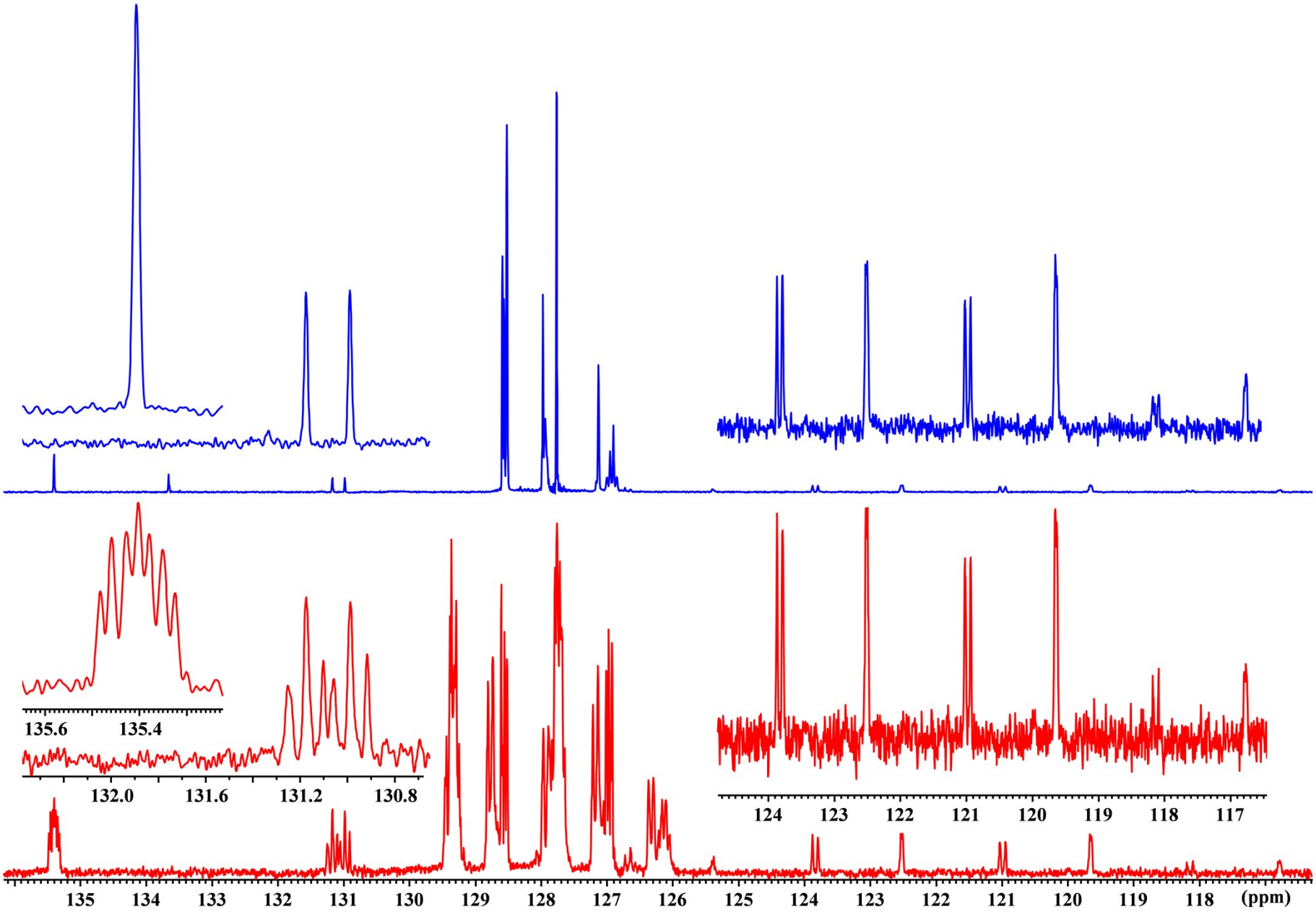


Figure 12. The low-field region of ^{13}C-{^{1}H} and ^{13}C NMR spectra (100.6 MHz, $CDCl_3$, 25°C) of phosphorane **14**.

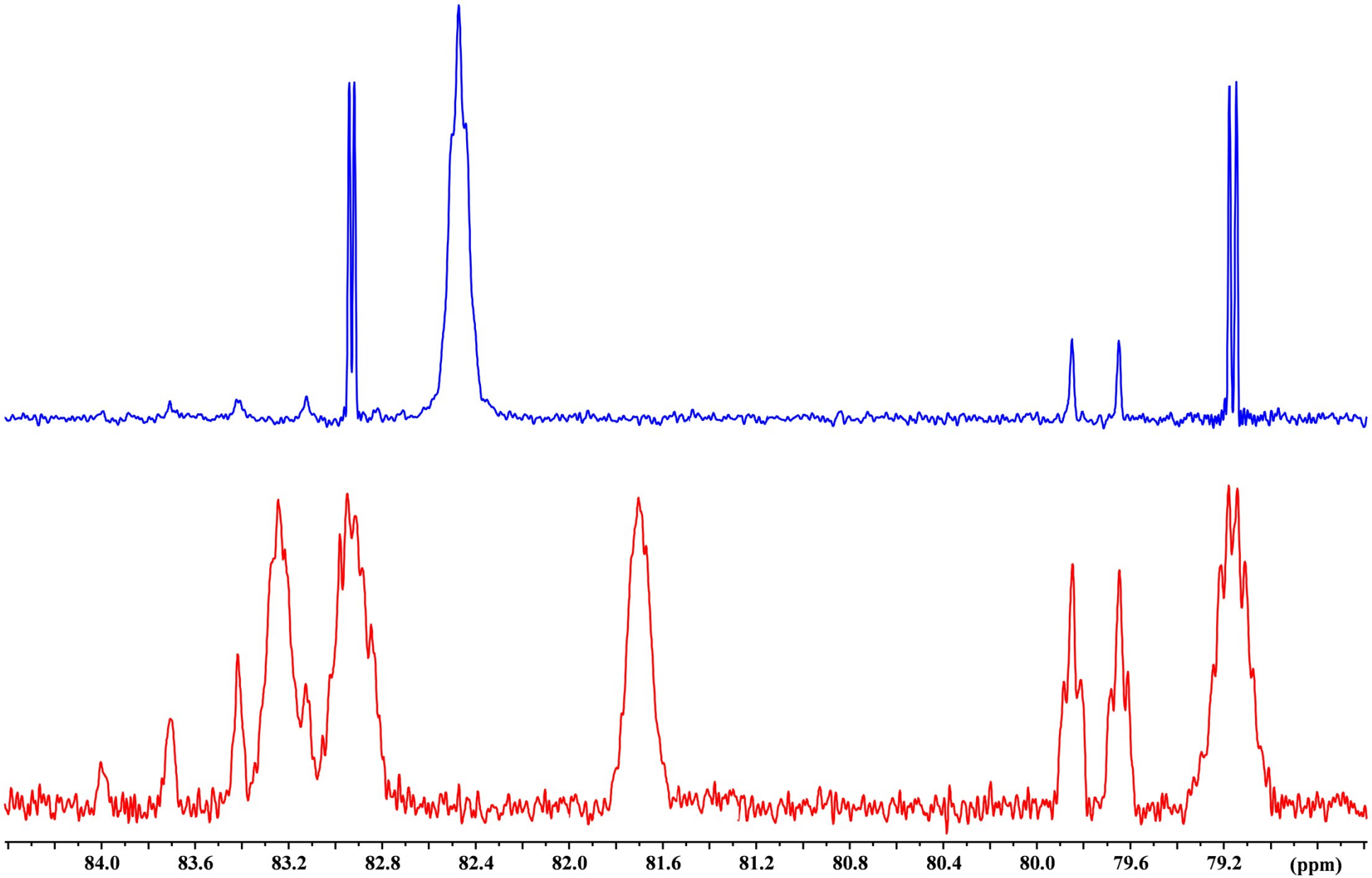


Figure 13. The fragments of ^{13}C-{^{1}H} and ^{13}C NMR spectra (100.6 MHz, $CDCl_3$, 25°C) of phosphorane **14** (the 78-84 ppm field is shown).

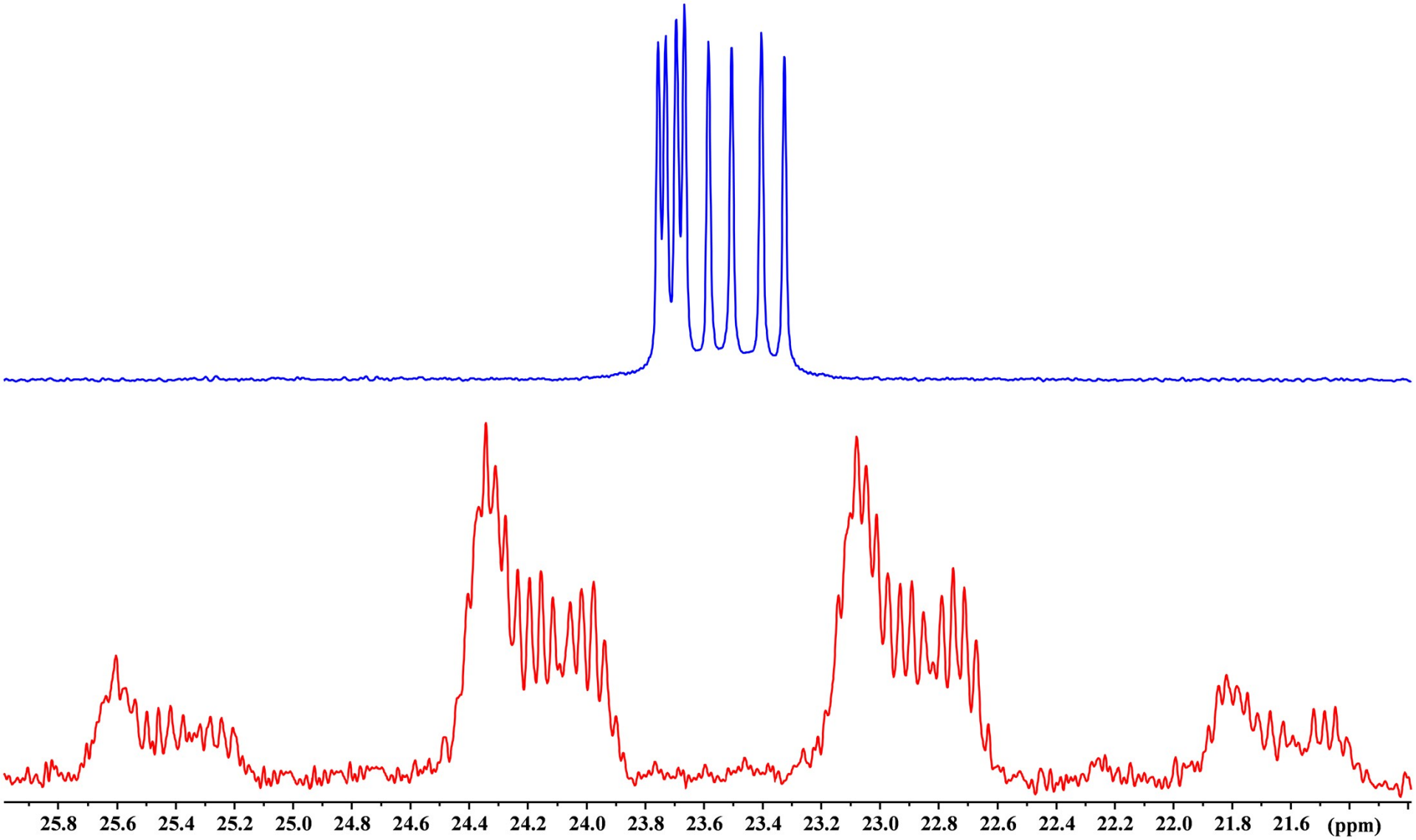


Figure 14. The high-field region of ^{13}C-{^{1}H} and ^{13}C NMR spectra (100.6 MHz, $CDCl_3$, 25°C) of phosphorane **14**.

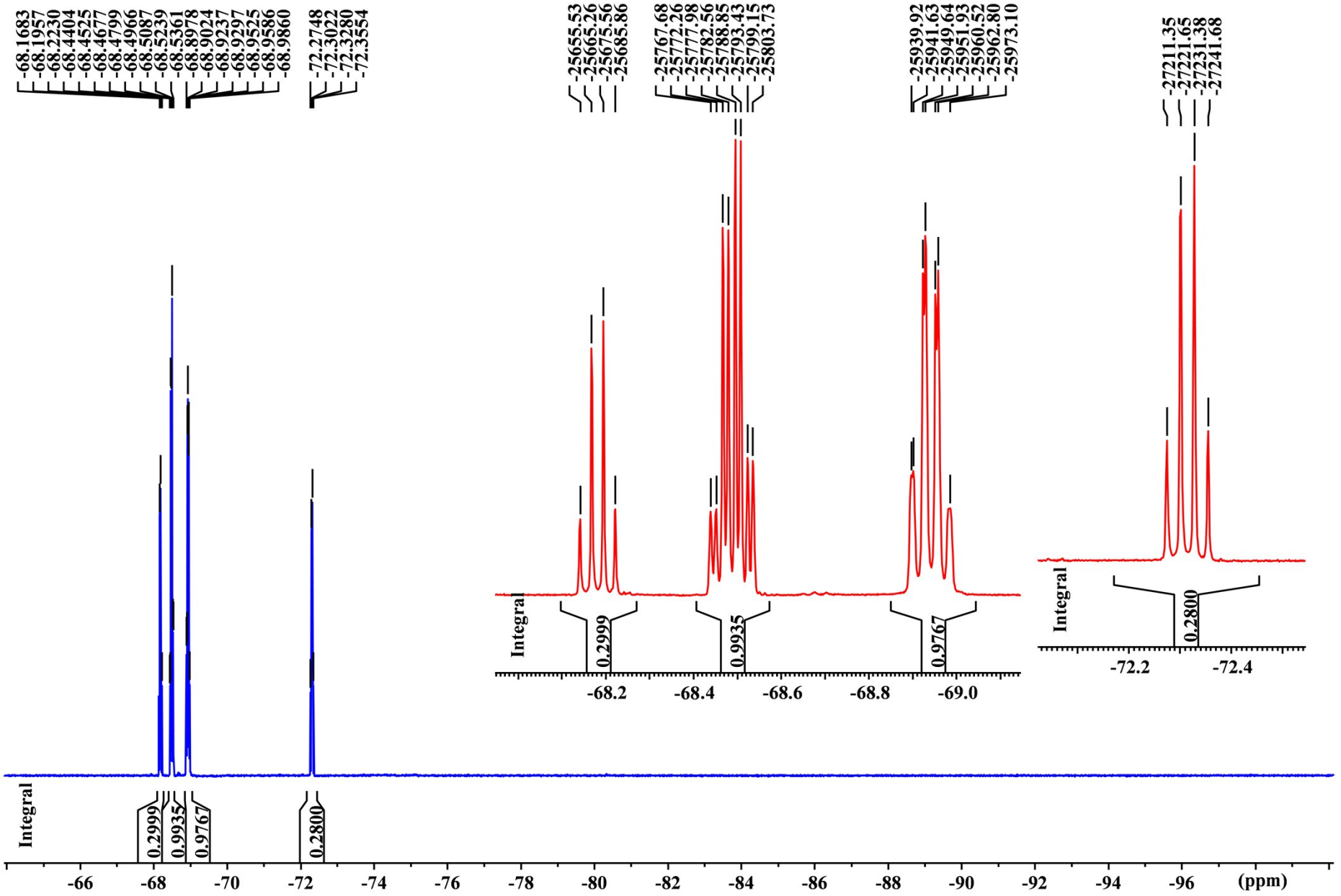


Figure 15. ^{19}F NMR spectrum (376.4 MHz, $CDCl_3$, 25°C) of phosphoranes **13, 14** mixture after ^{13}C experiments for compound **13**.

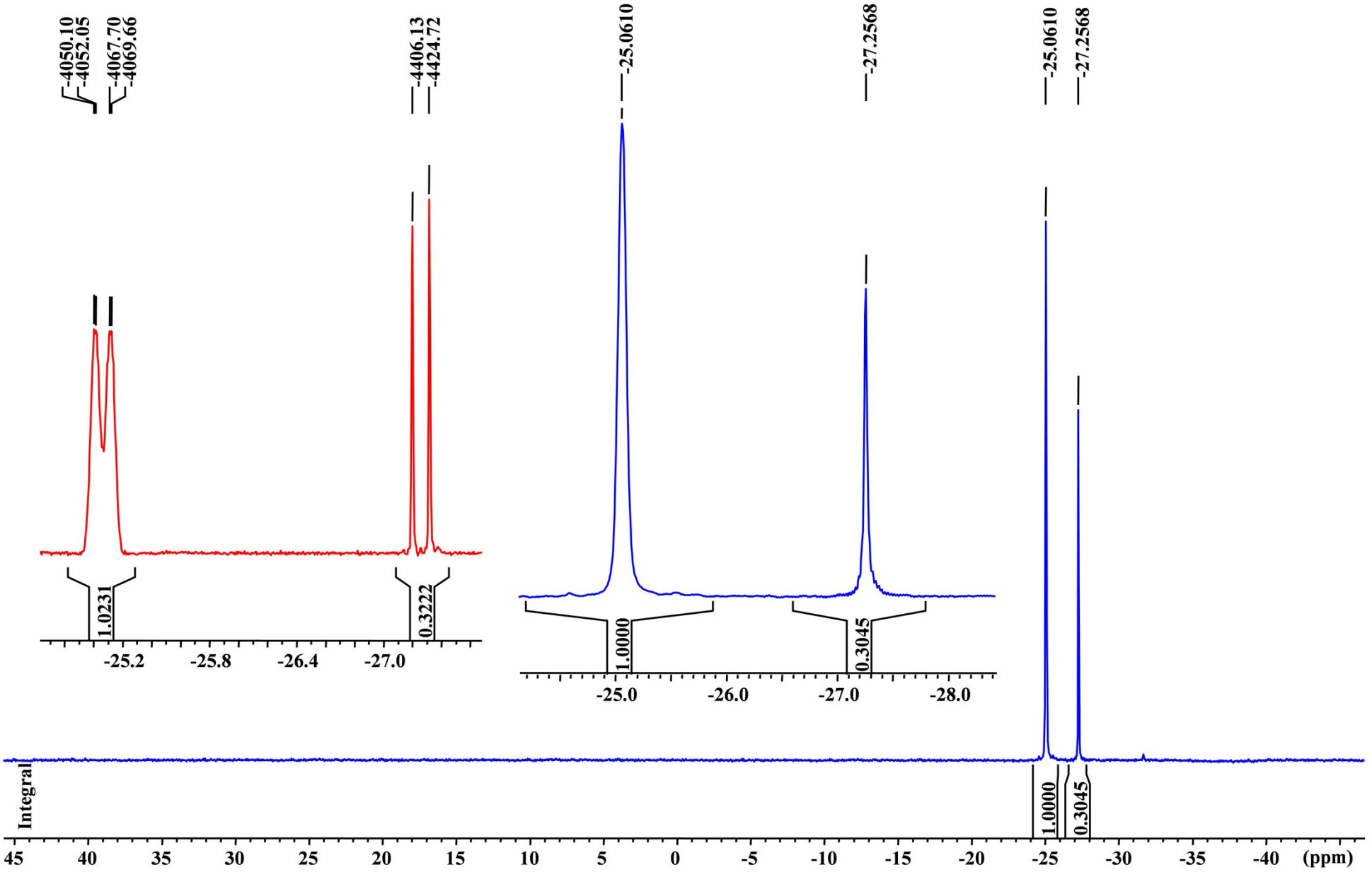


Figure 16. ^{31}P and ^{31}P-{^{1}H} NMR spectra (162.0 MHz, $CDCl_3$, 25°C) of phosphoranes **13, 14** mixture after ^{13}C experiments for compound **13**.

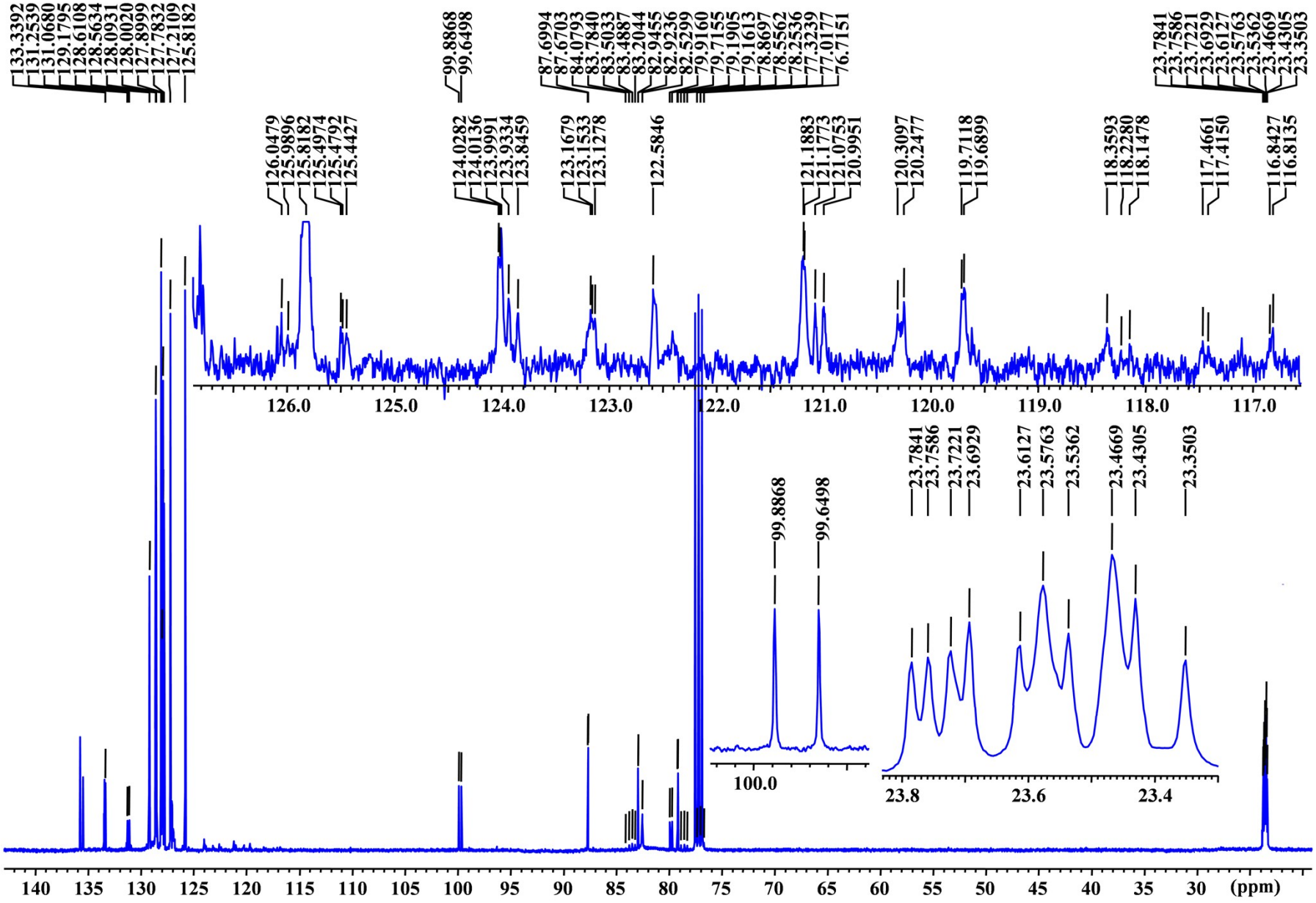


Figure 17. ^{13}C-{^{1}H} NMR spectrum (100.6 MHz, $CDCl_3$, 25°C) of phosphoranes **13, 14** mixture.

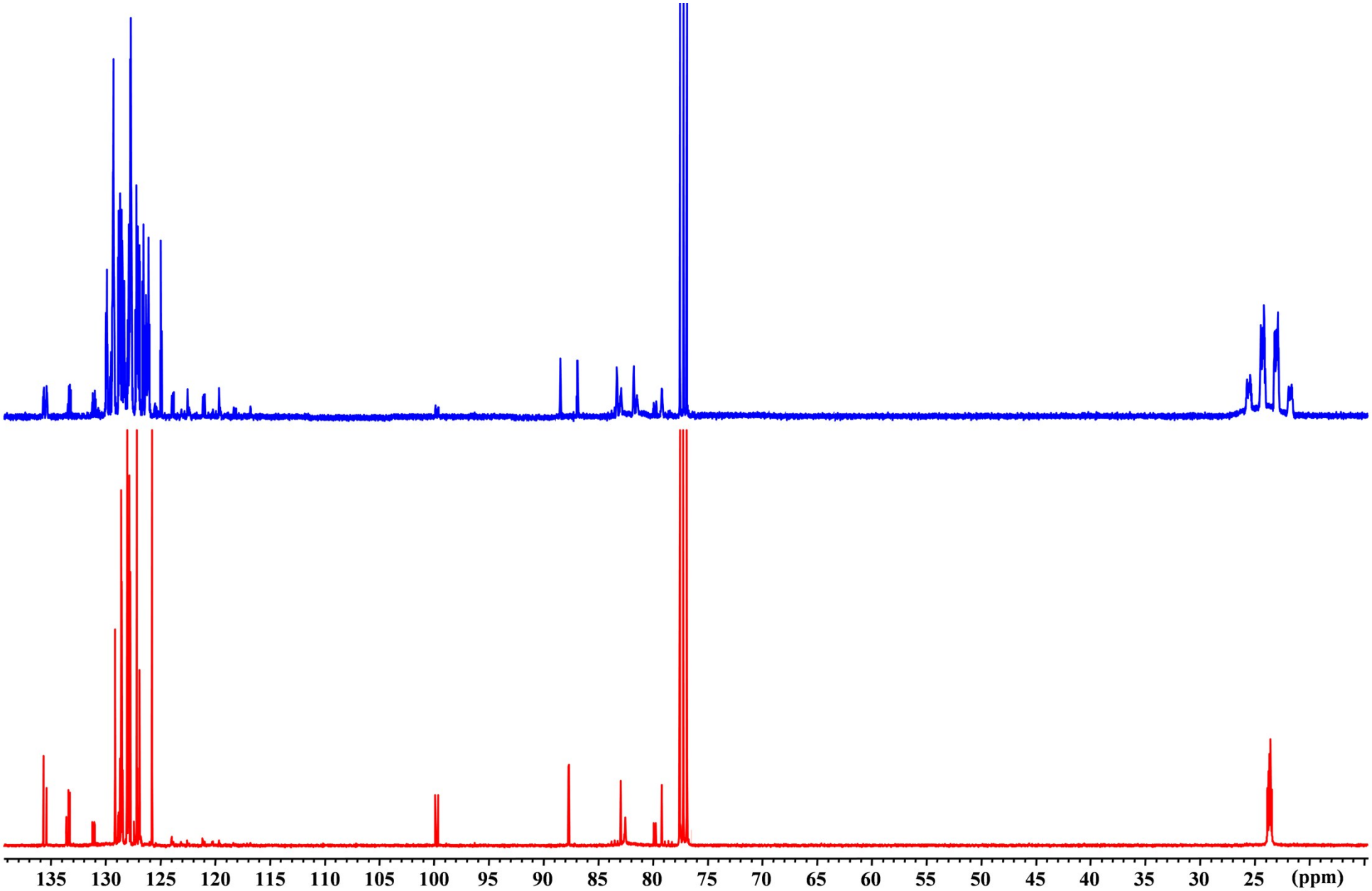


Figure 18. ^{13}C-{^{1}H} and ^{13}C NMR spectra (100.6 MHz, $CDCl_3$, 25°C) of phosphoranes **13**, **14** mixture.

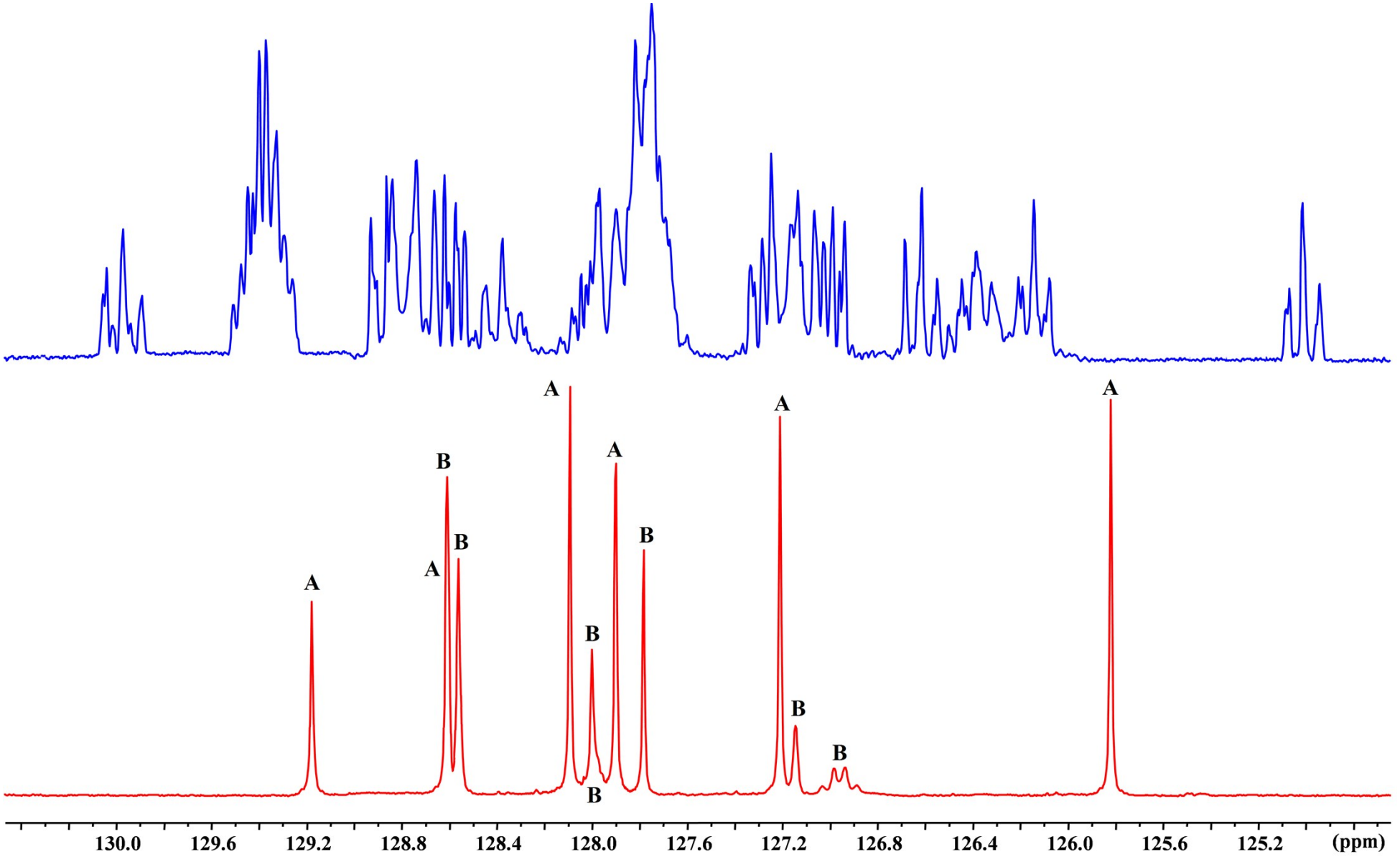


Figure 19. ^{13}C-{^{1}H} and ^{13}C NMR spectra (100.6 MHz, $CDCl_3$, 25°C) of phosphoranes **13, 14** mixture (**13 – A**, **14 – B**).

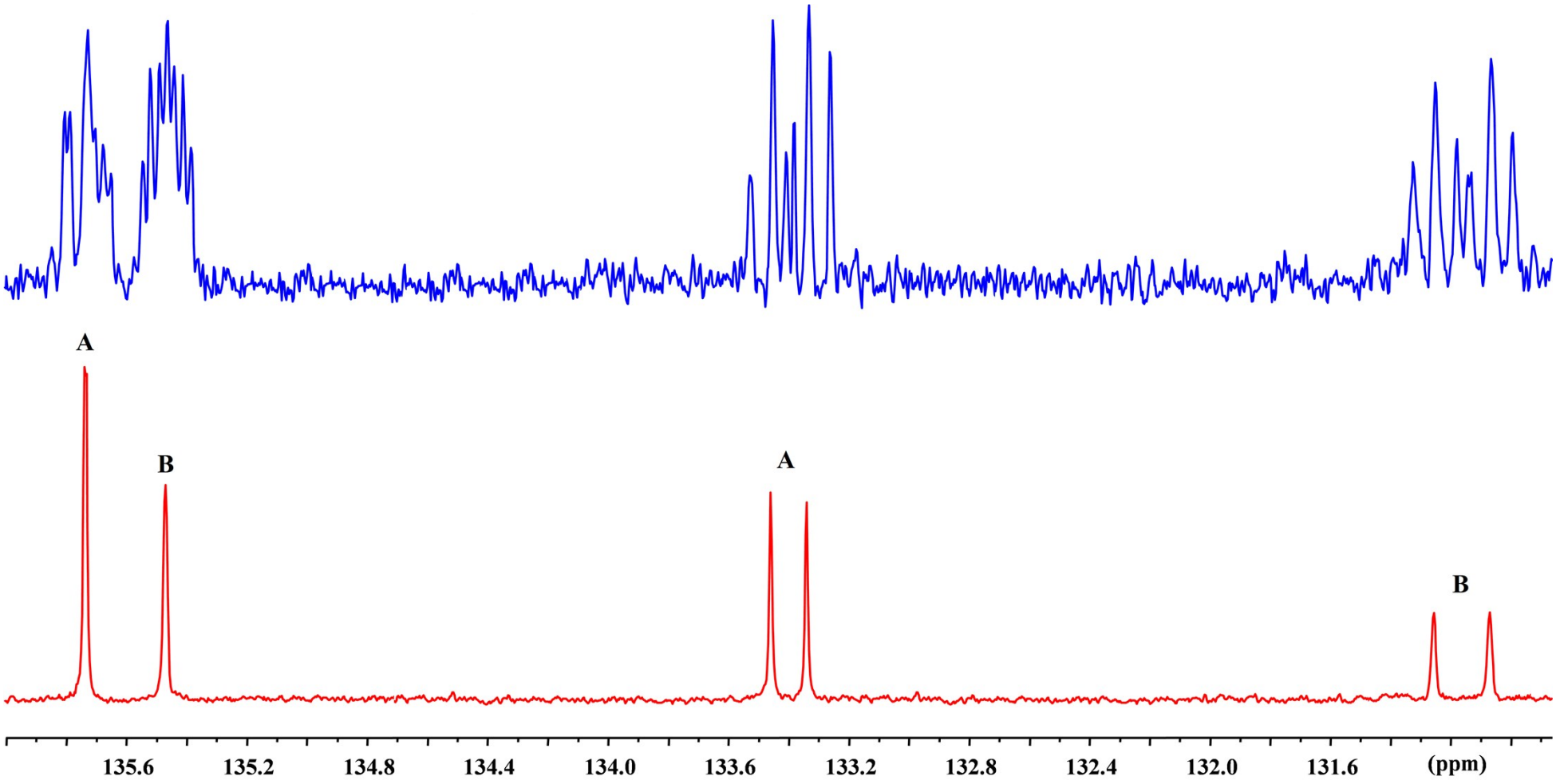


Figure 20. The low-field region of ^{13}C-{^{1}H} and ^{13}C NMR spectra (100.6 MHz, $CDCl_3$, 25°C) of phosphoranes **13, 14** mixture (**13 – A**, **14 – B**).

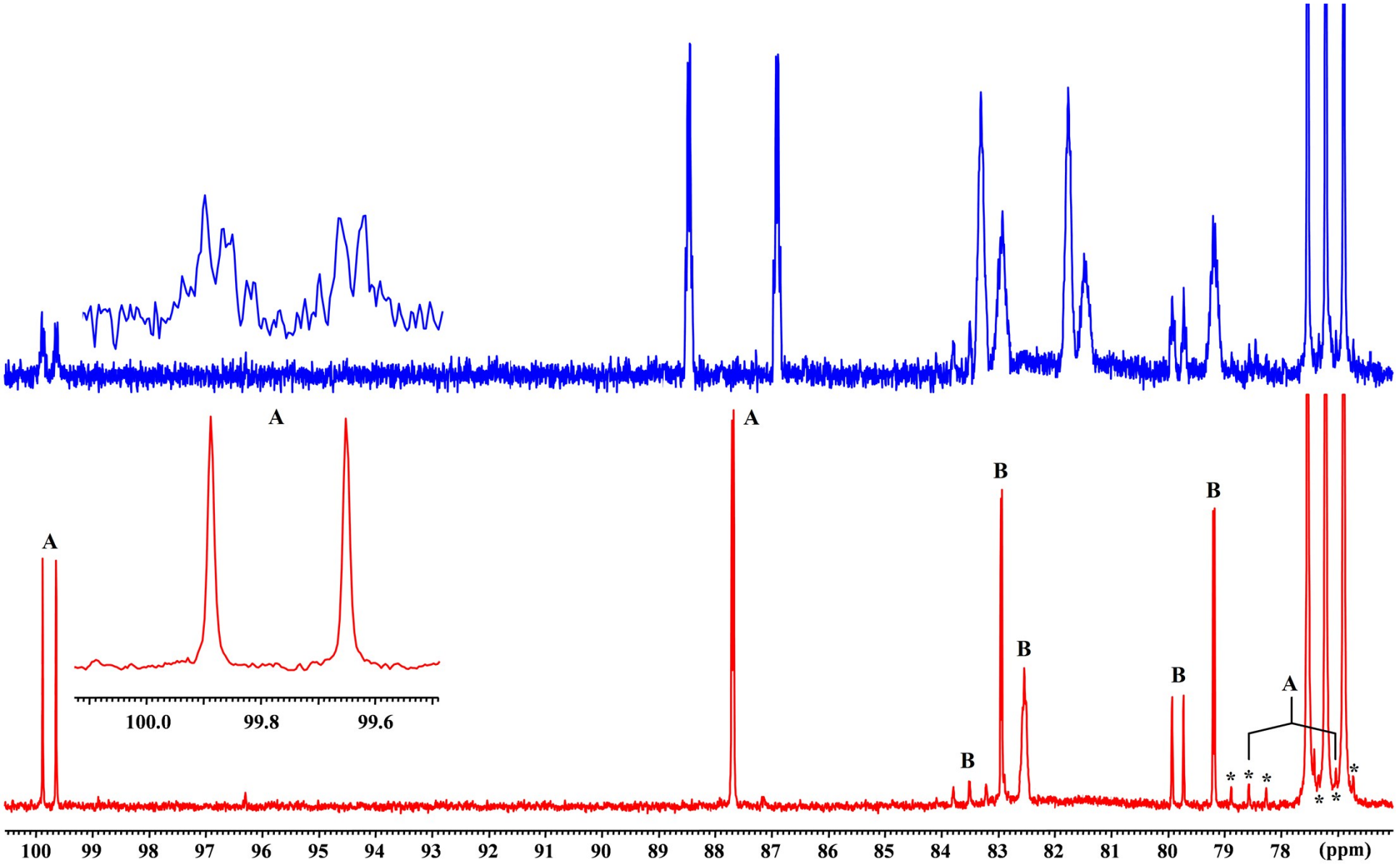


Figure 21. The fragments of ^{13}C-{^{1}H} and ^{13}C NMR spectra (100.6 MHz, $CDCl_3$, 25°C) of phosphoranes **13, 14** mixture (the 76-100 ppm field is shown) (**13 – A**, **14 – B**).

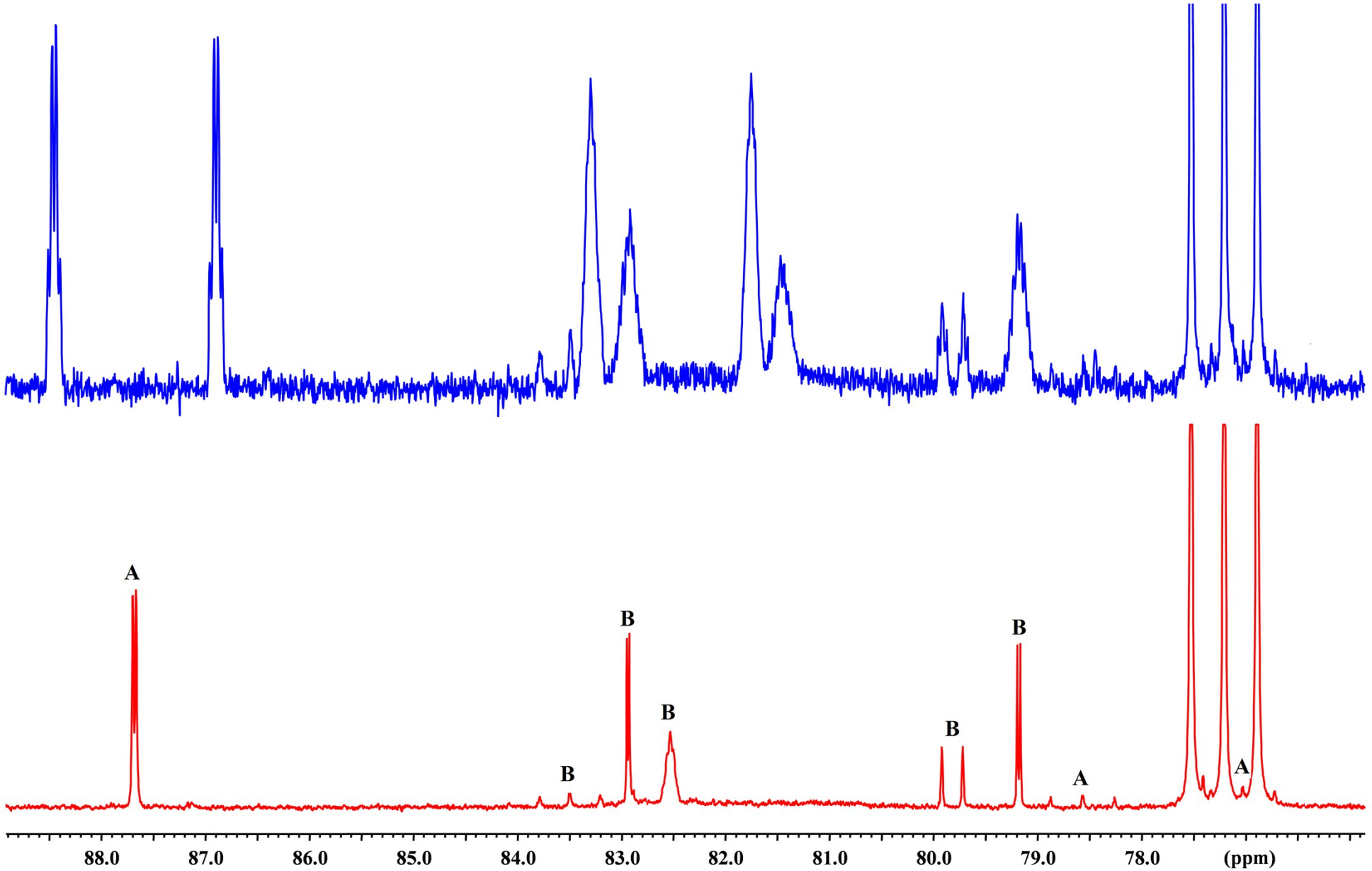


Figure 22. The fragments of ^{13}C-{^{1}H} and ^{13}C NMR spectra (100.6 MHz, $CDCl_3$, 25°C) of phosphoranes **13, 14** mixture (the 76-89 ppm field is shown) (**13 – A**, **14 – B**).

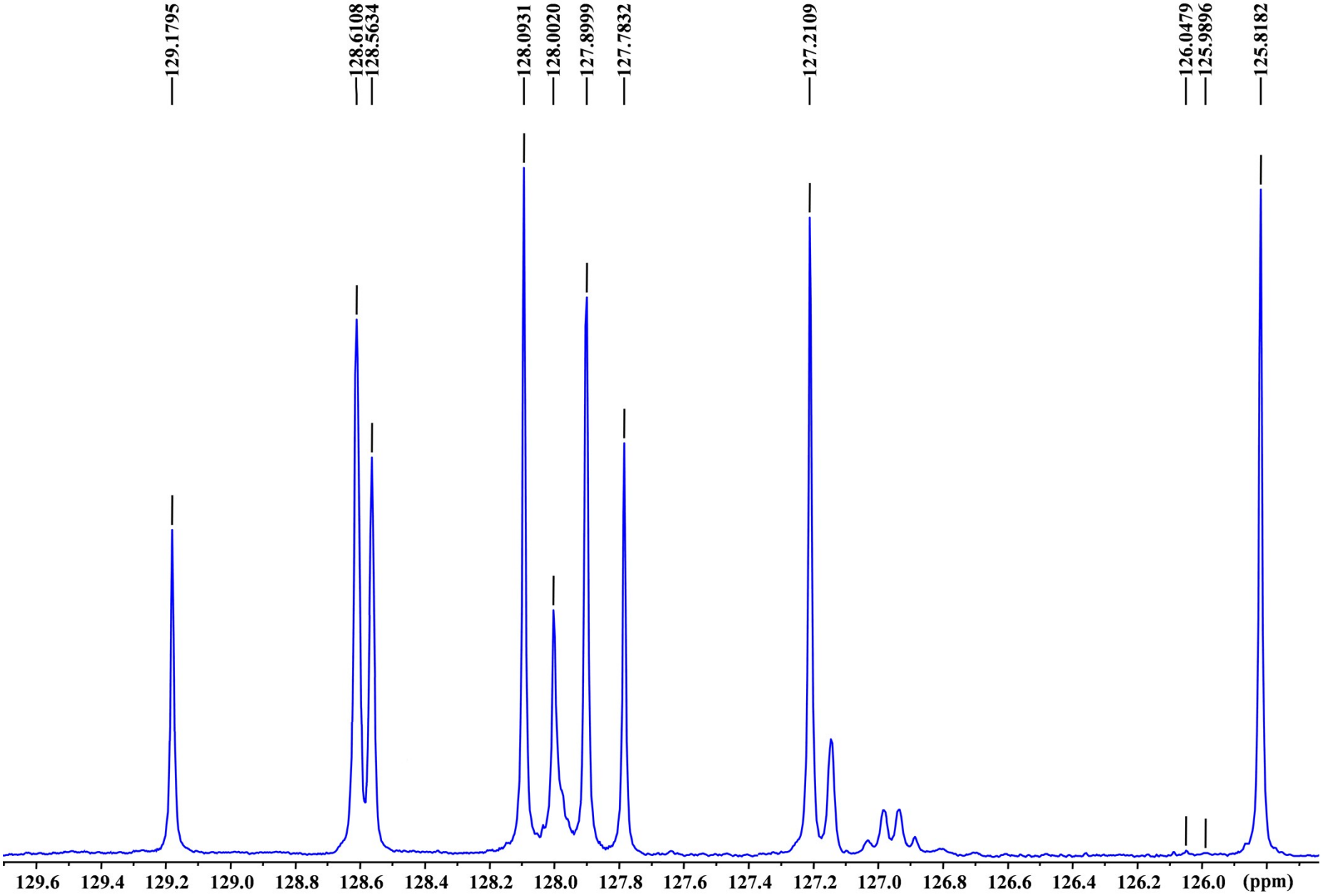


Figure 23. The aromatic carbons region of ^{13}C-{^{1}H} NMR spectrum (100.6 MHz, $CDCl_3$, 25°C) of phosphoranes **13, 14** mixture.

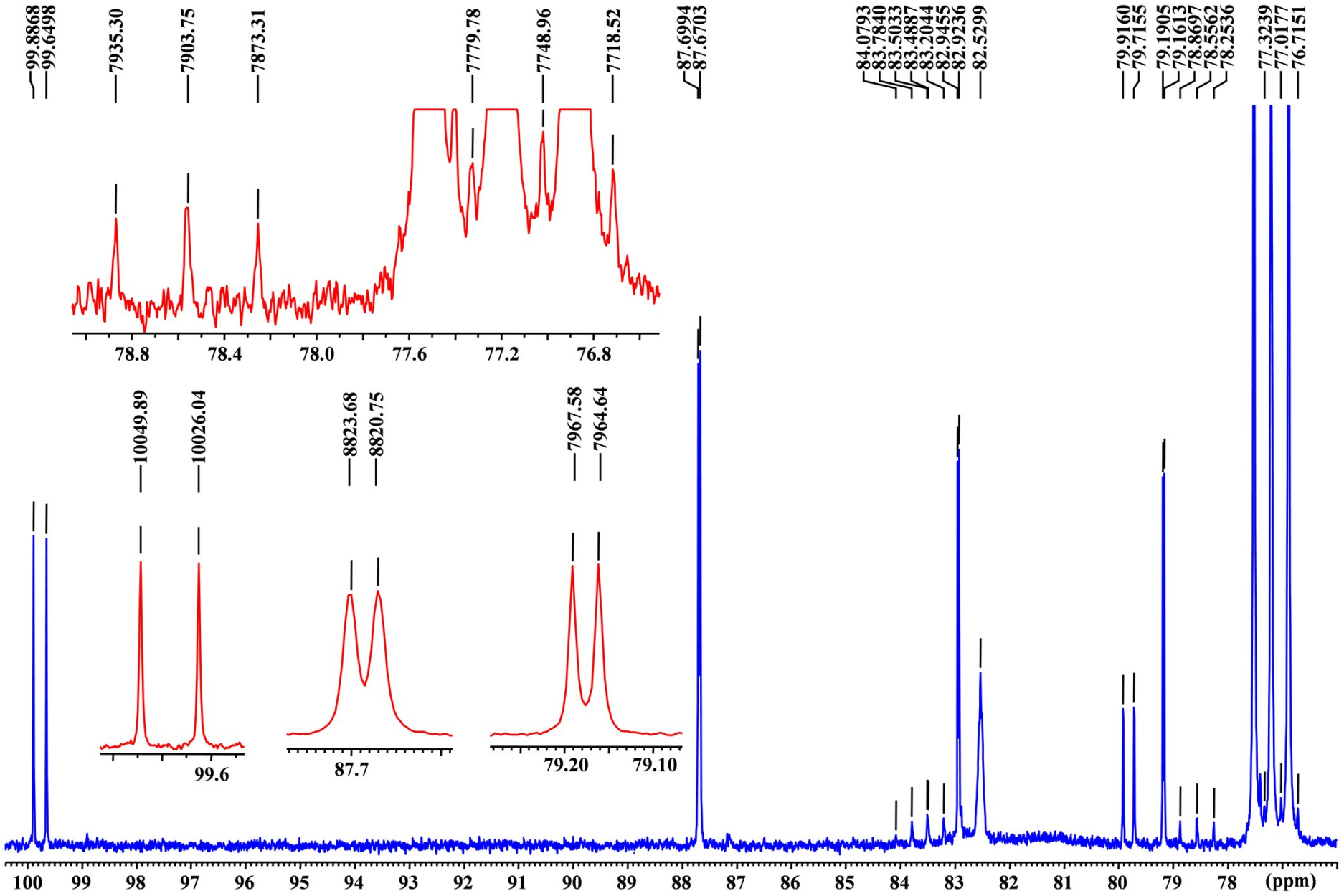


Figure 24. The fragment of ^{13}C-{^{1}H} NMR spectrum (100.6 MHz, $CDCl_3$, 25°C) of phosphoranes **13, 14** mixture (the 76-100 ppm field is shown).

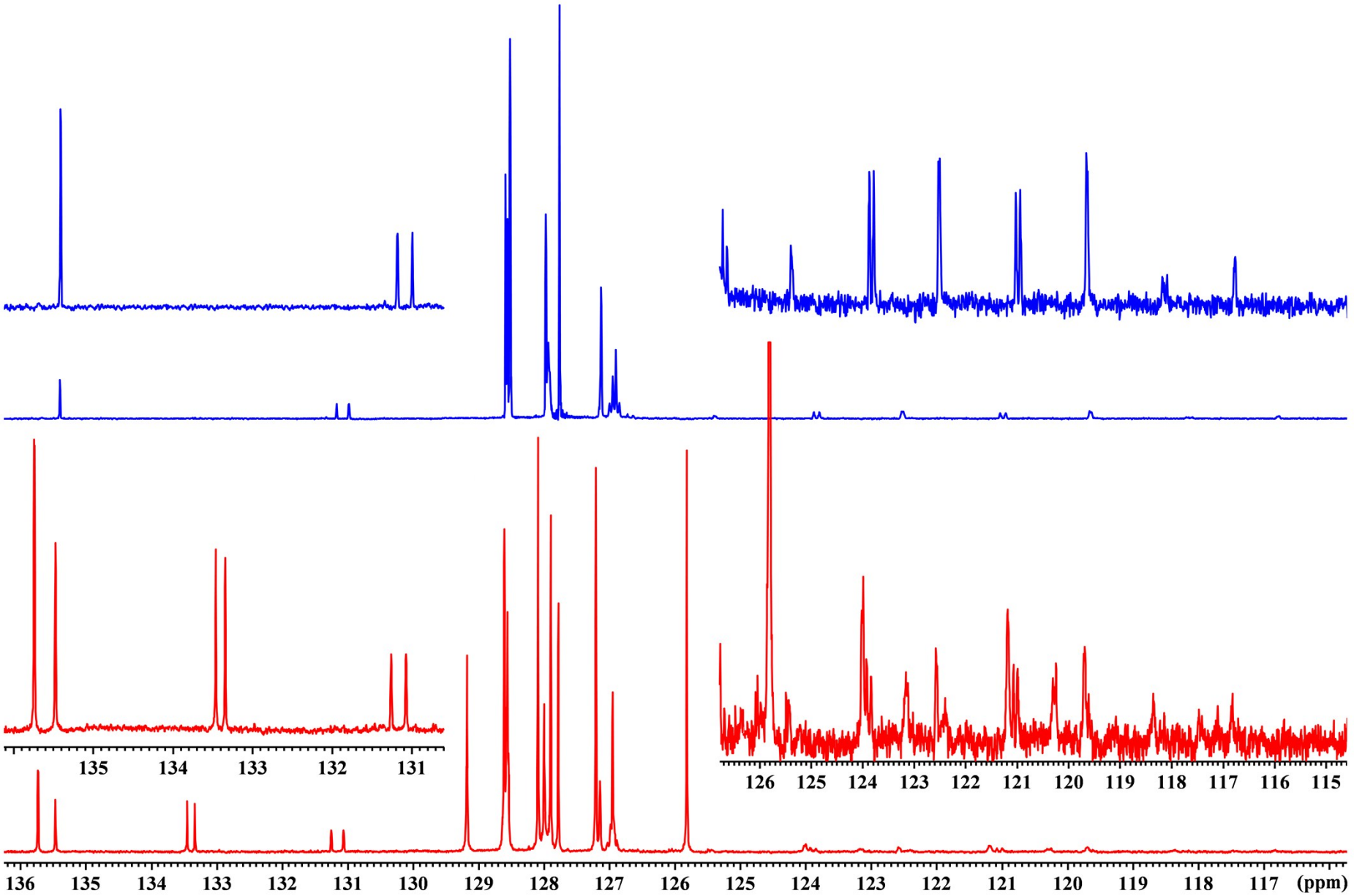


Figure 25. A comparison of ^{13}C-{^{1}H} NMR spectrum (100.6 MHz, $CDCl_3$, 25°C) of phosphorane **14** and compounds **13, 14** mixture (the low-field region is shown).

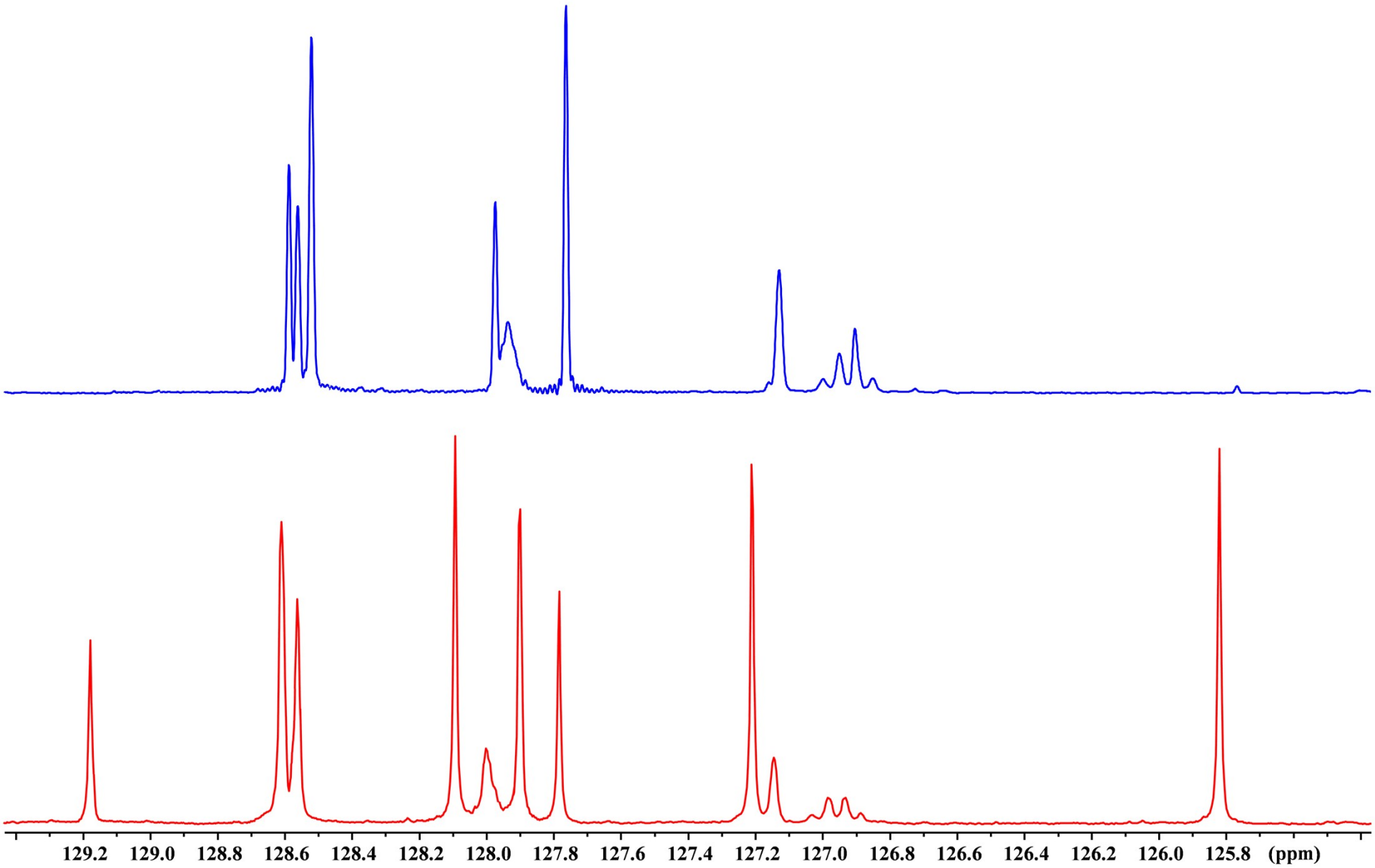


Figure 26. A comparison of ^{13}C-{^{1}H} NMR spectrum (100.6 MHz, $CDCl_3$, 25°C) of phosphorane **14** and compounds **13, 14** mixture (the aromatic carbons region is shown).

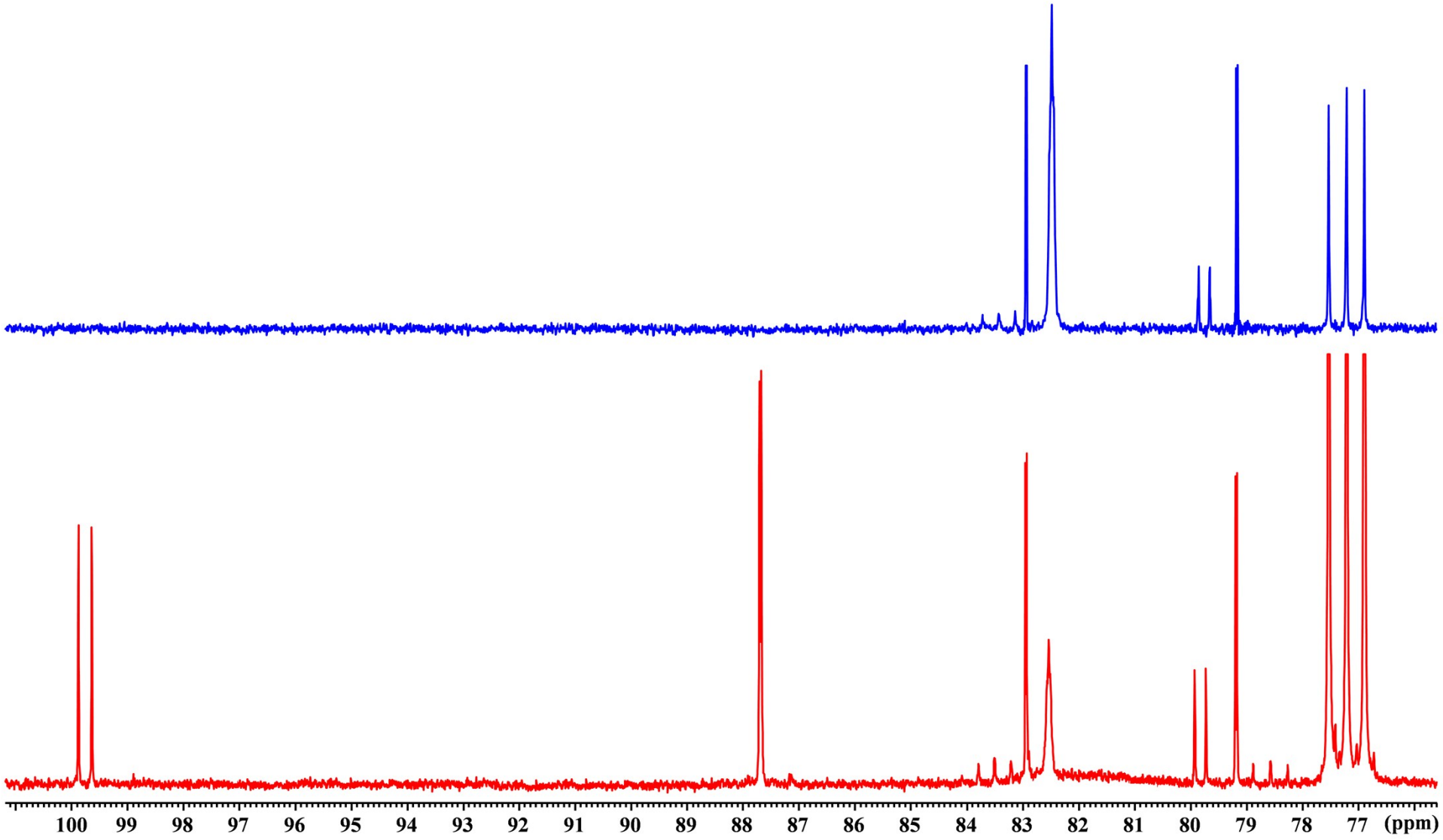


Figure 27. A comparison of ^{13}C-{^{1}H} NMR spectrum (100.6 MHz, $CDCl_3$, 25°C) of phosphorane **14** and compounds **13, 14** mixture (the 76-101 ppm region is shown).

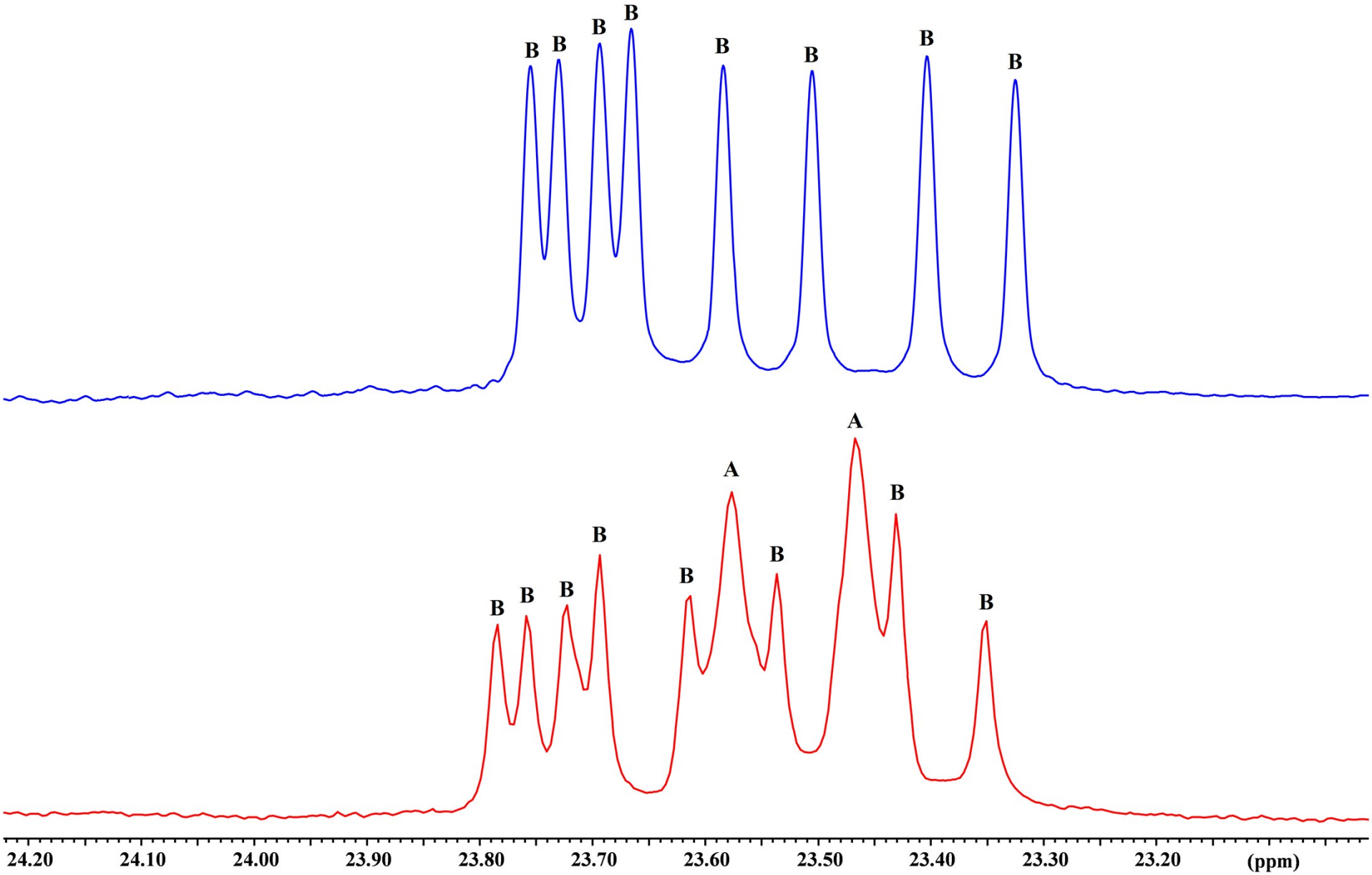


Figure 28. A comparison of ^{13}C-{^{1}H} NMR spectrum (100.6 MHz, $CDCl_3$, 25°C) of phosphorane **14** and compounds **13, 14** mixture (the high-field region is shown) (**13 – A**, **14 – B**).

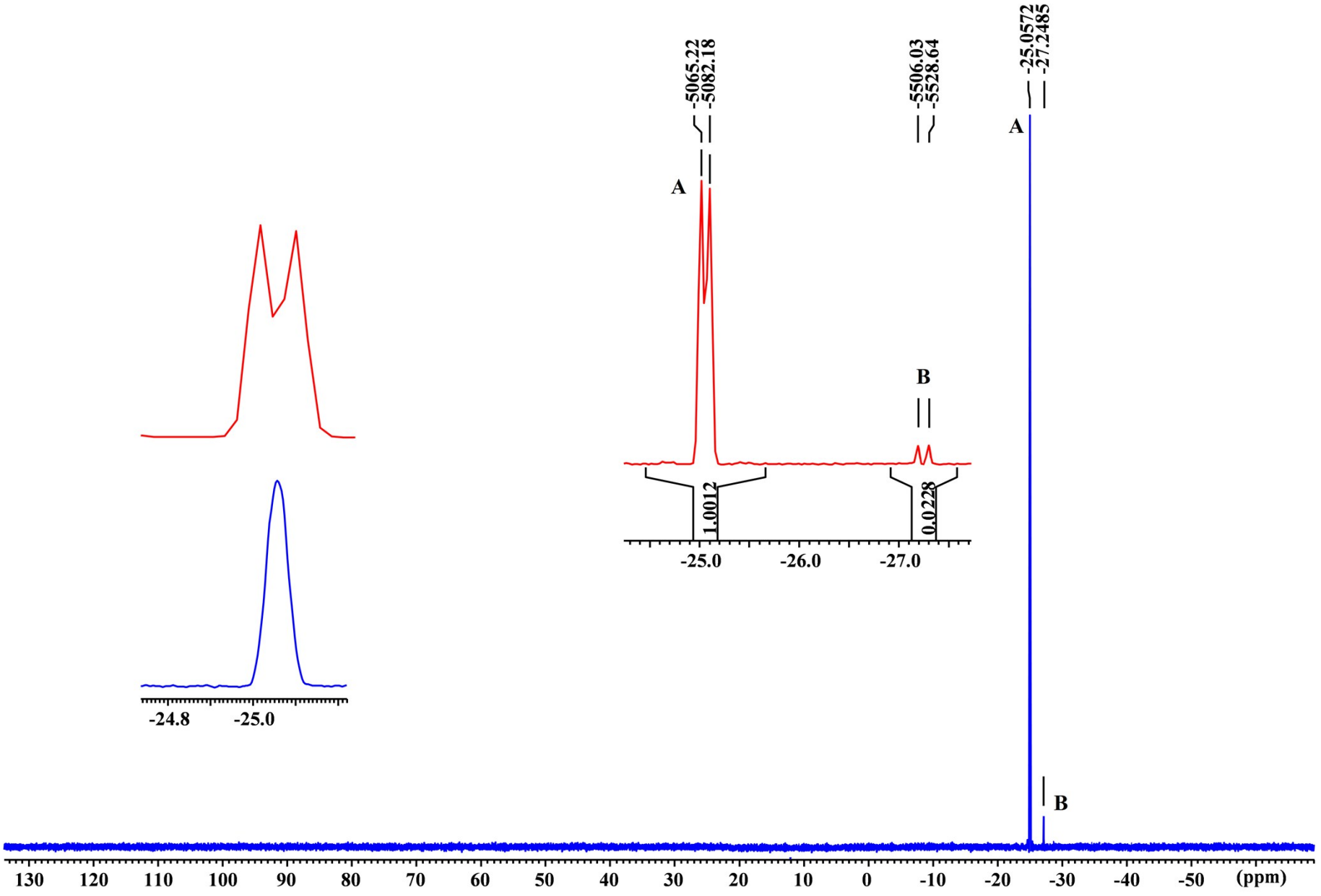


Figure 29. ^{31}P and ^{31}P-{^{1}H} NMR spectra (162.0 MHz, $CDCl_3$, 25°C) of phosphoranes **13** before ^{13}C experiments (**13 – A**, **14 – B**).

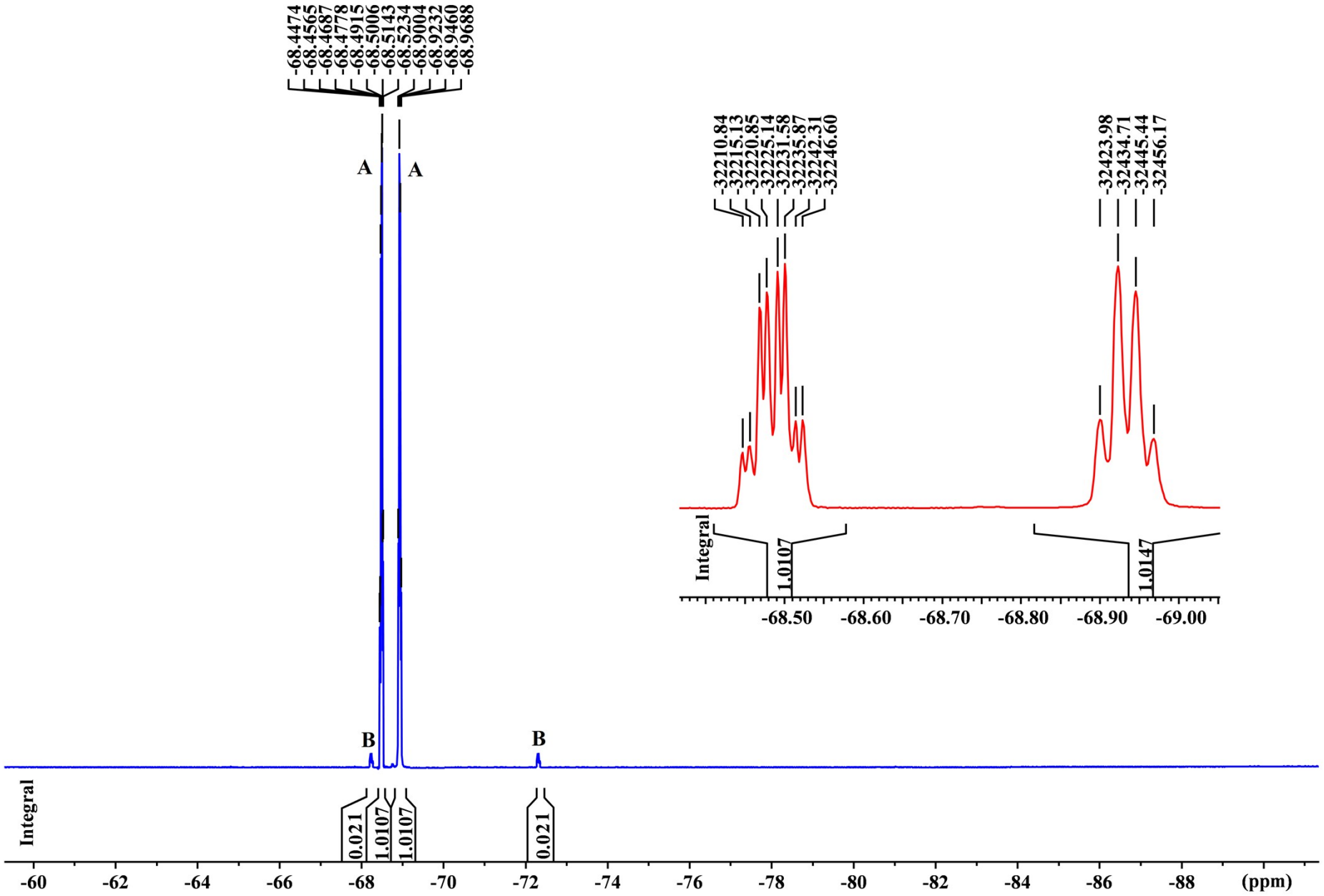


Figure 30. ^{19}F NMR spectrum (376.4 MHz, $CDCl_3$, 25°C) of phosphoranes **13** before ^{13}C experiments (**13 – A**, **14 – B**).

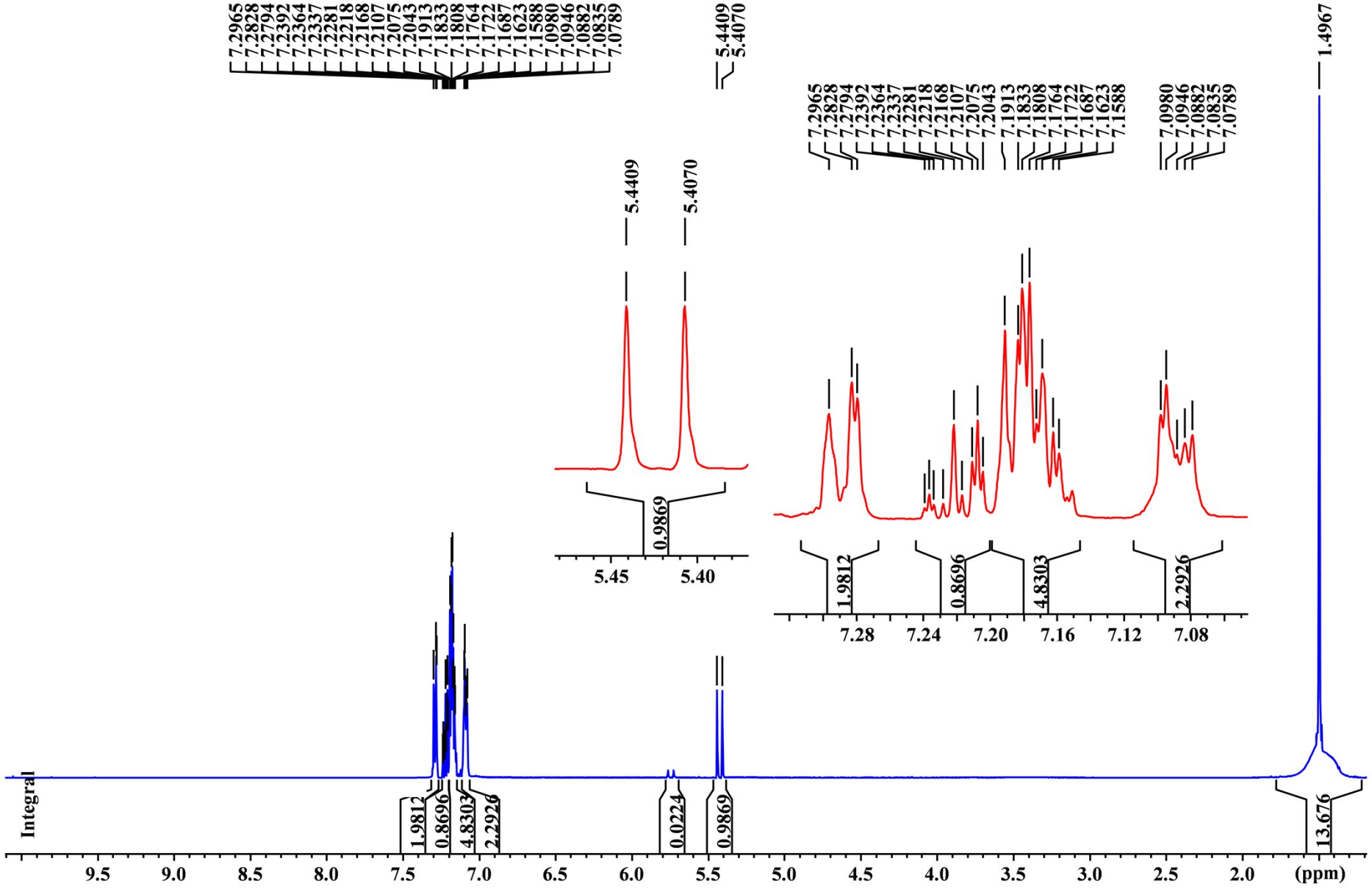


Figure 31. ^{1}H NMR spectrum (400.0 MHz, $CDCl_3$, 25°C) of phosphoranes **13** before ^{13}C experiments.

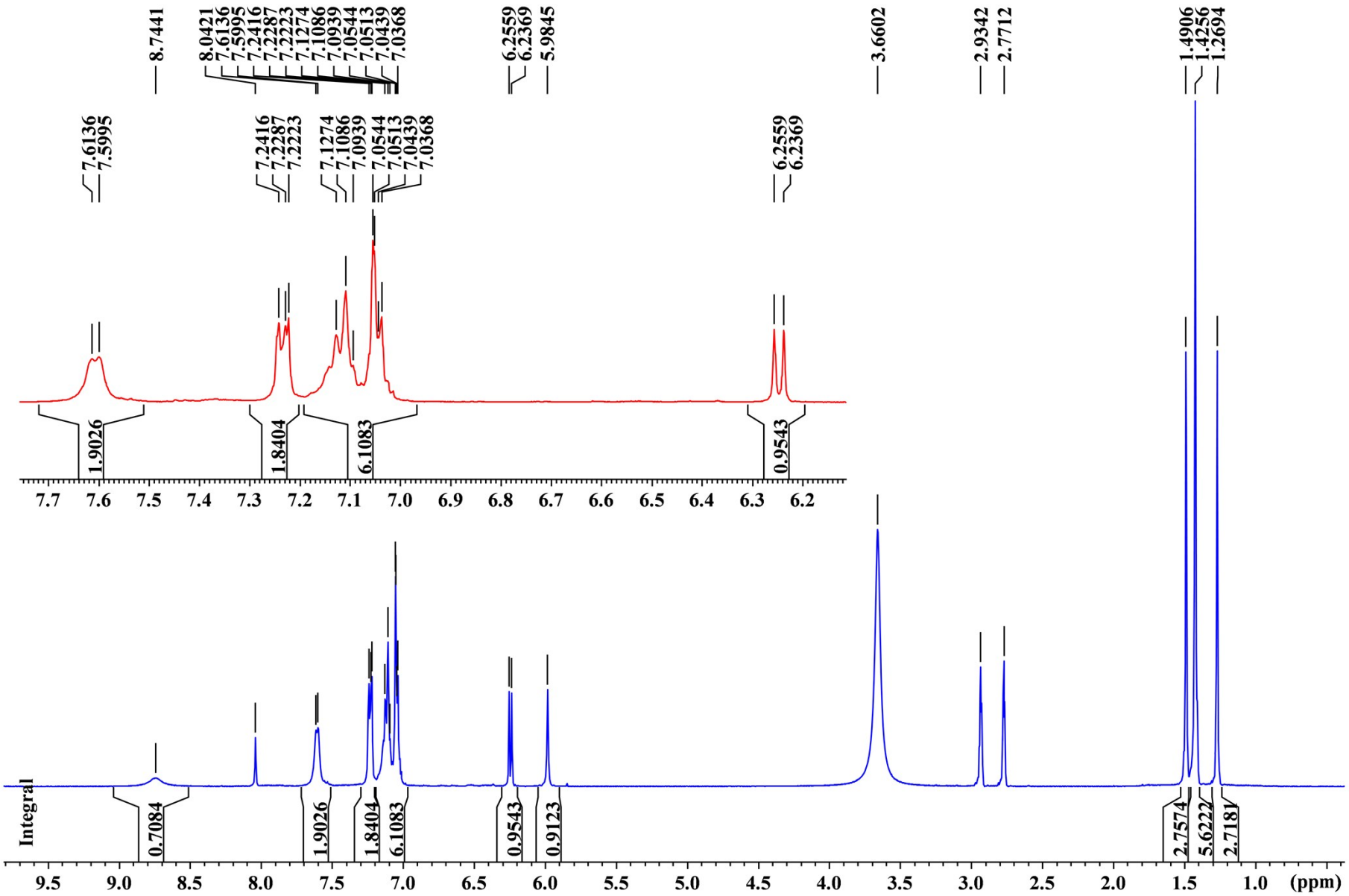


Figure .32 ^{1}H spectrum (400 MHz, DMF-d_7, 25°C) of phospholane **16**.

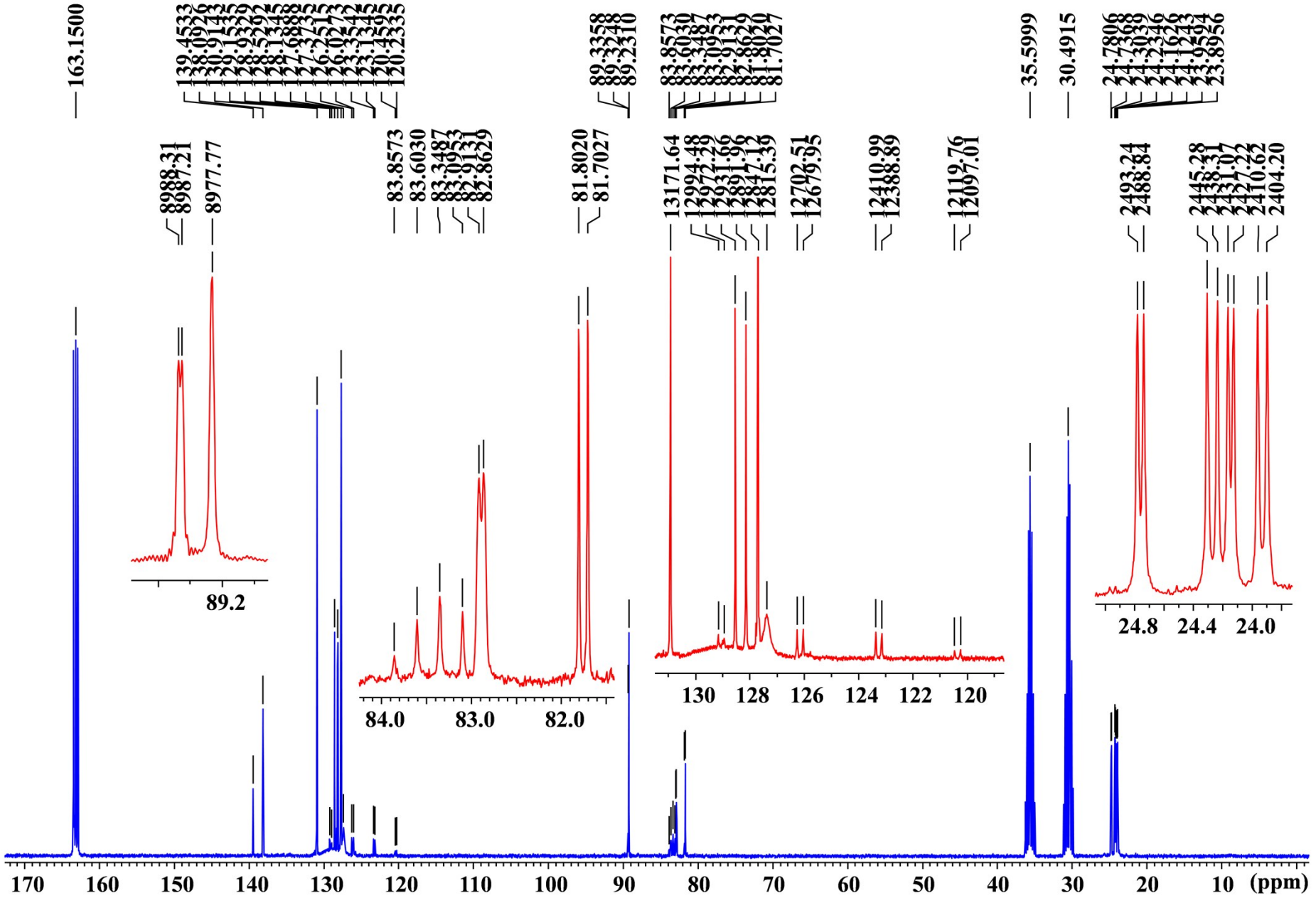


Figure 33. Full ^{13}C-{^{1}H} spectrum (100.6 MHz, DMF-d_7, 25°C) of phospholane **16**.

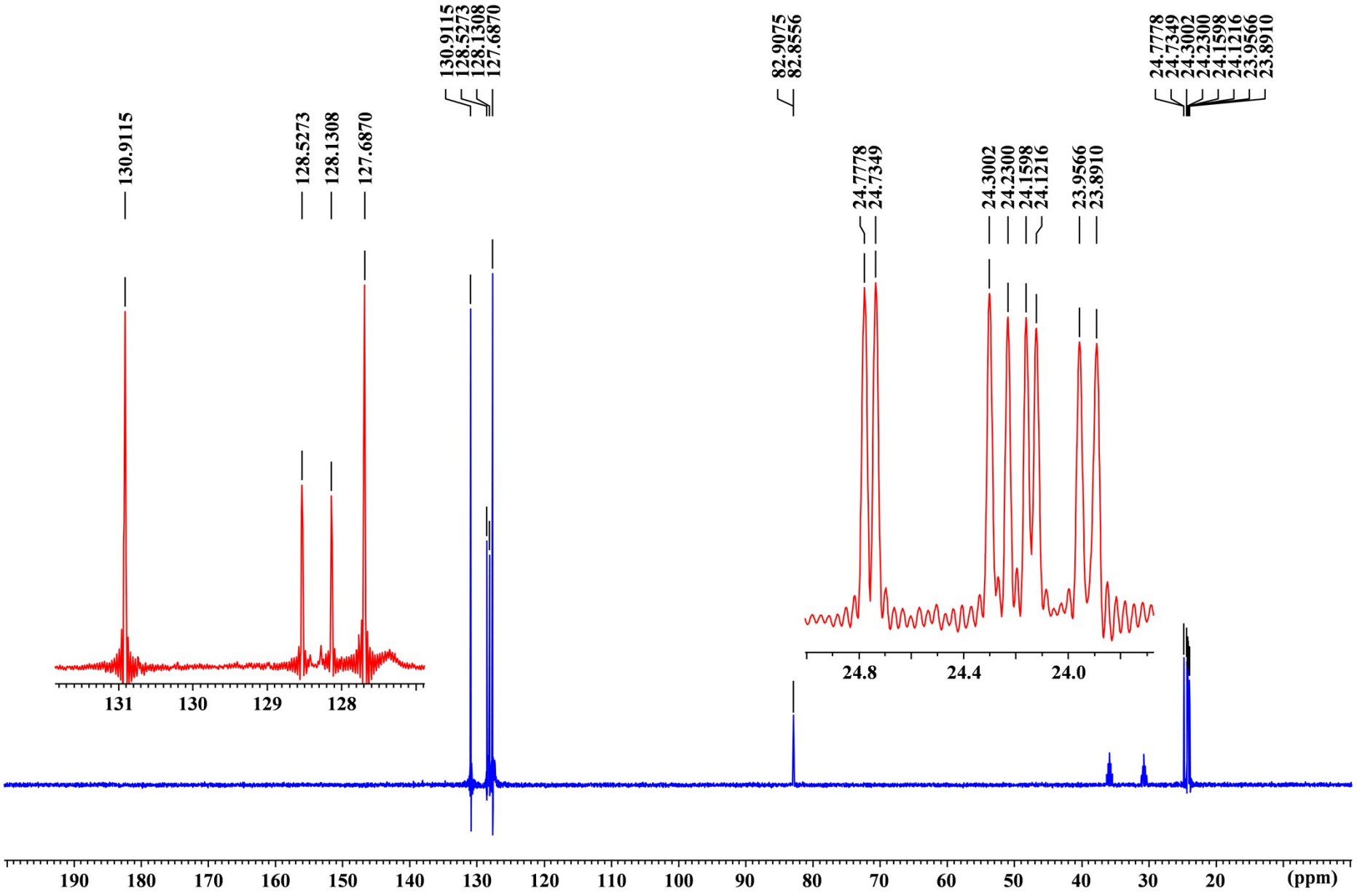


Figure 34. ^{13}C-{^{1}H} and ^{13}C-{^{1}H} dept spectrum (100.6 MHz, DMF-d_7, 25°C) of phospholane **16**.

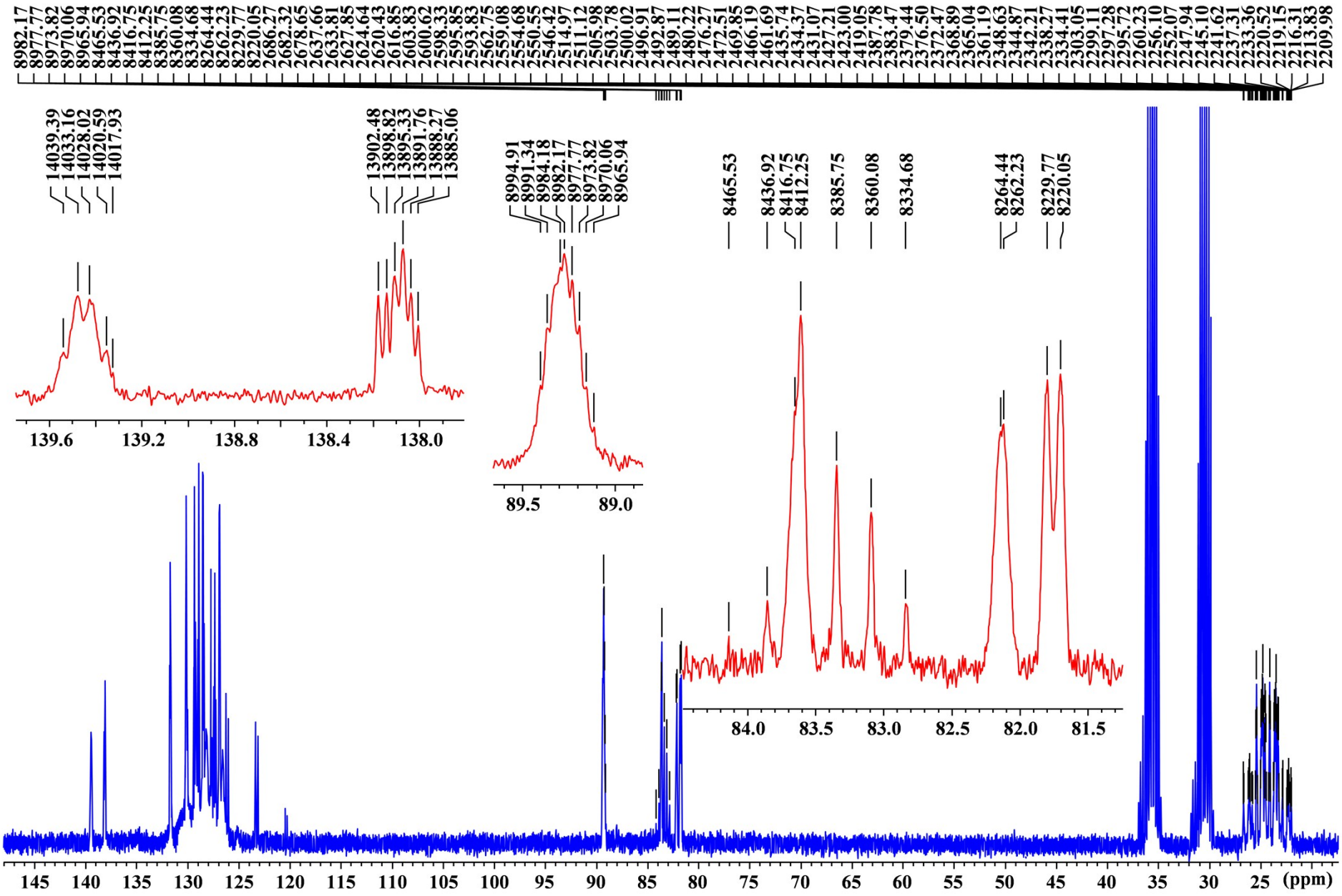


Figure 35. Full ^{13}C spectrum (100.6 MHz, DMF-d_7, 25°C) of phospholane **16**.

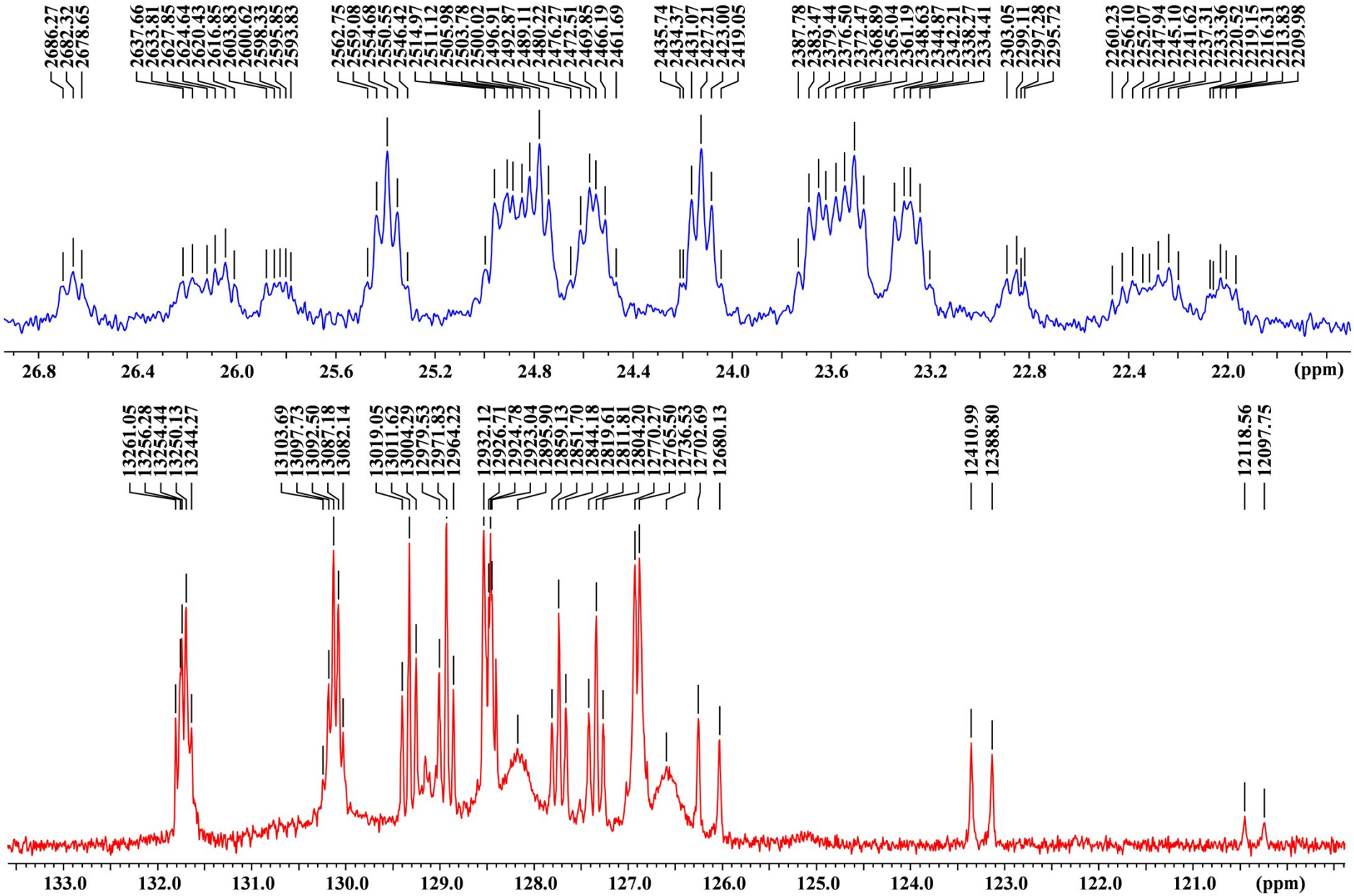


Figure 36. Low-field and high-field fragments of ^{13}C spectrum (100.6 MHz, DMF-d_7, 25°C) of phospholane **16**.

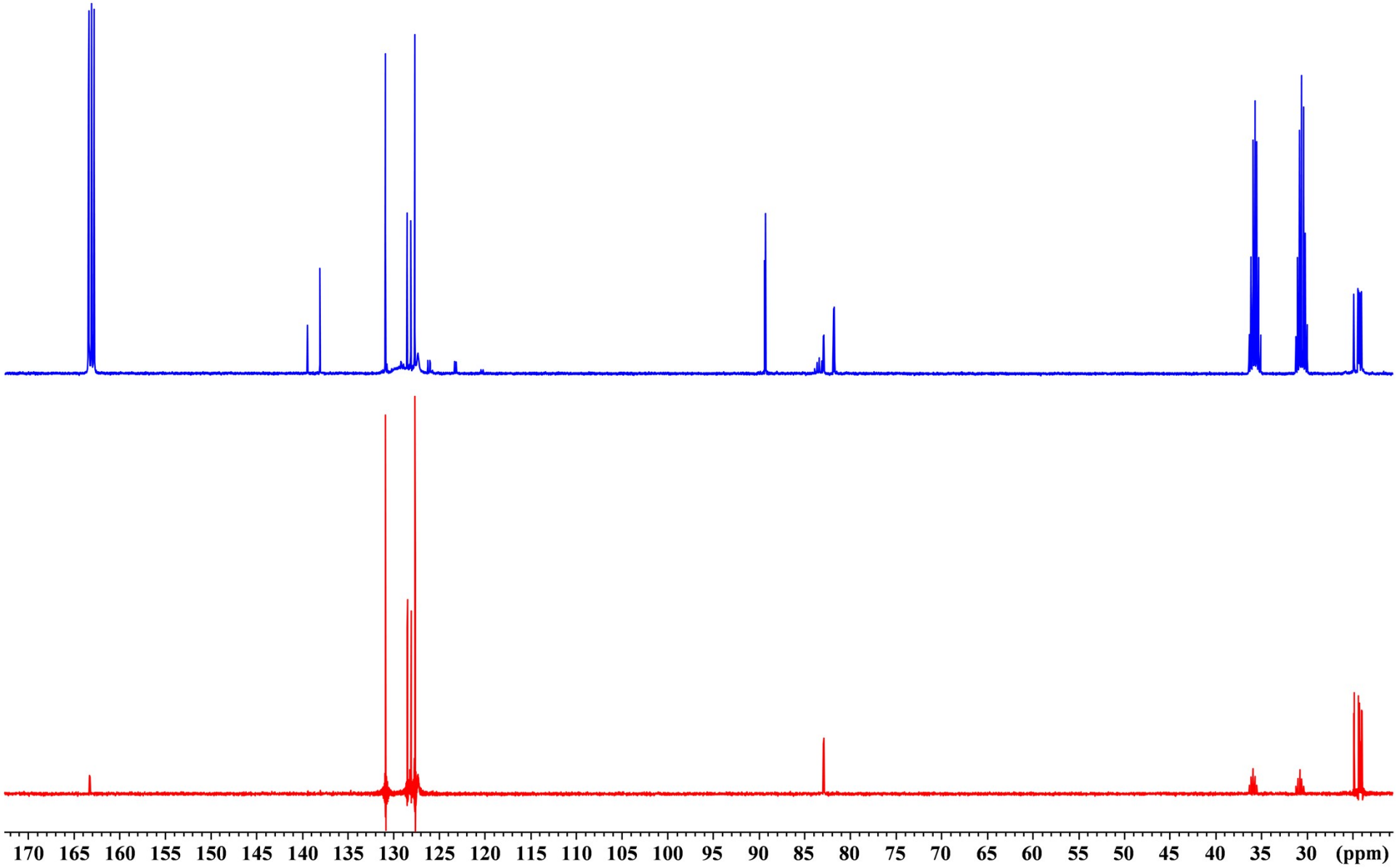


Figure 37. A comparison of ^{13}C-{^{1}H} and ^{13}C-{^{1}H} dept spectra (100.6 MHz, DMF-d_7, 25°C) of phospholane **16**.

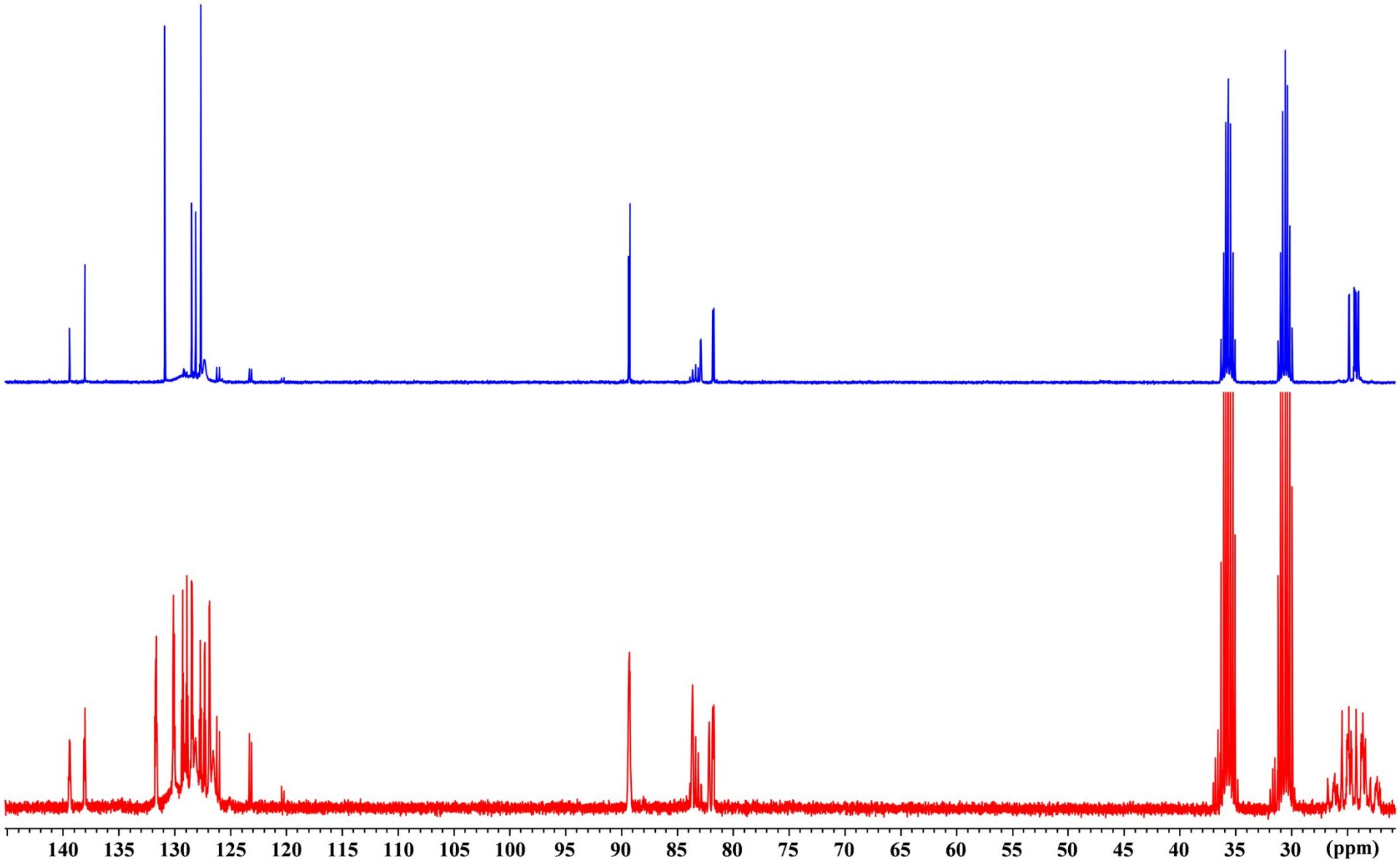


Figure 38. A comparison of ^{13}C and ^{13}C-{^{1}H} spectra (100.6 MHz, DMF-d_7, 25°C) of phospholane **16**.

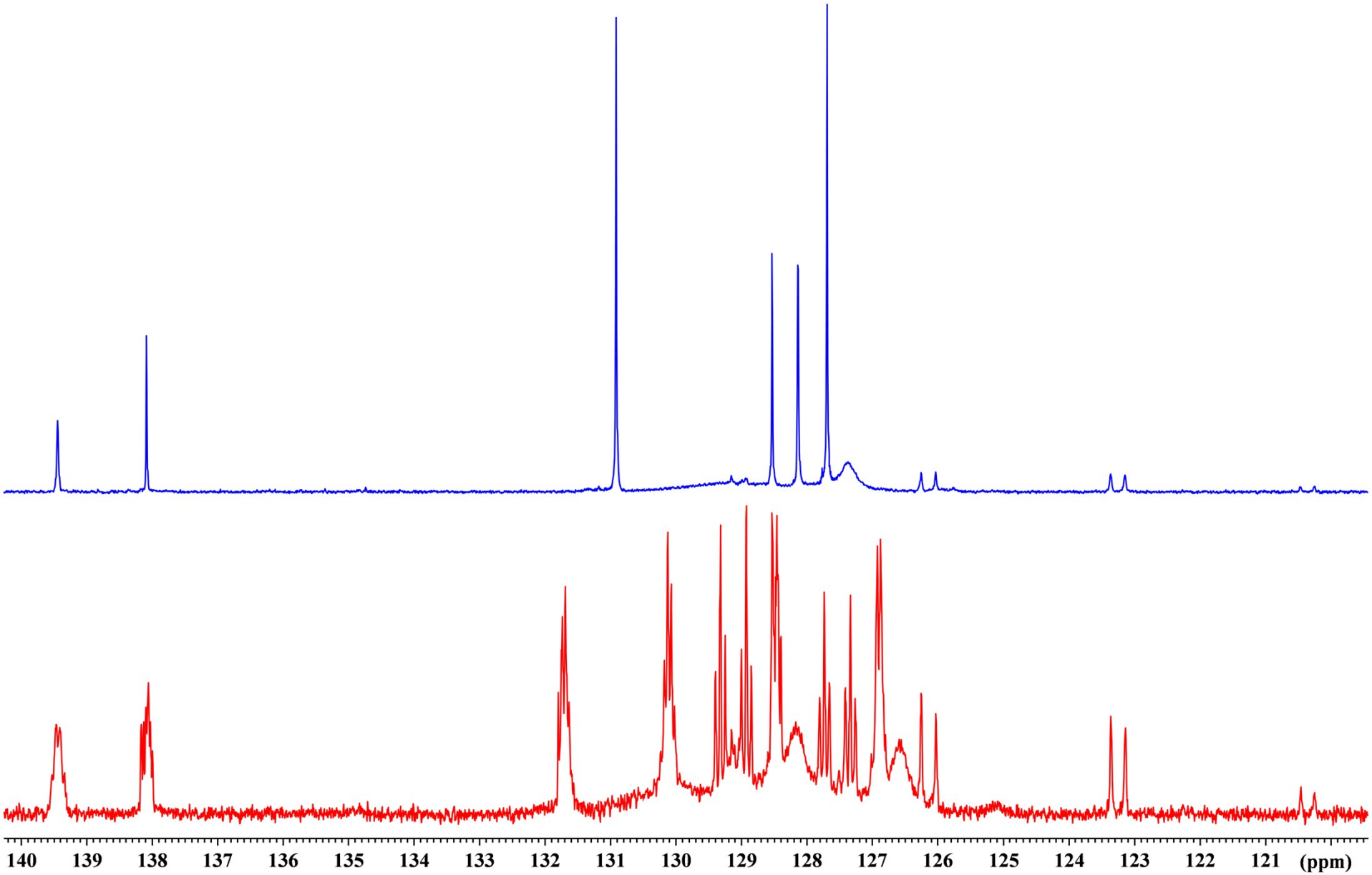


Figure 39. Low-field fragments of ^{13}C and ^{13}C-{^{1}H} spectra (100.6 MHz, DMF-d_7, 25°C) of phospholane **16**.

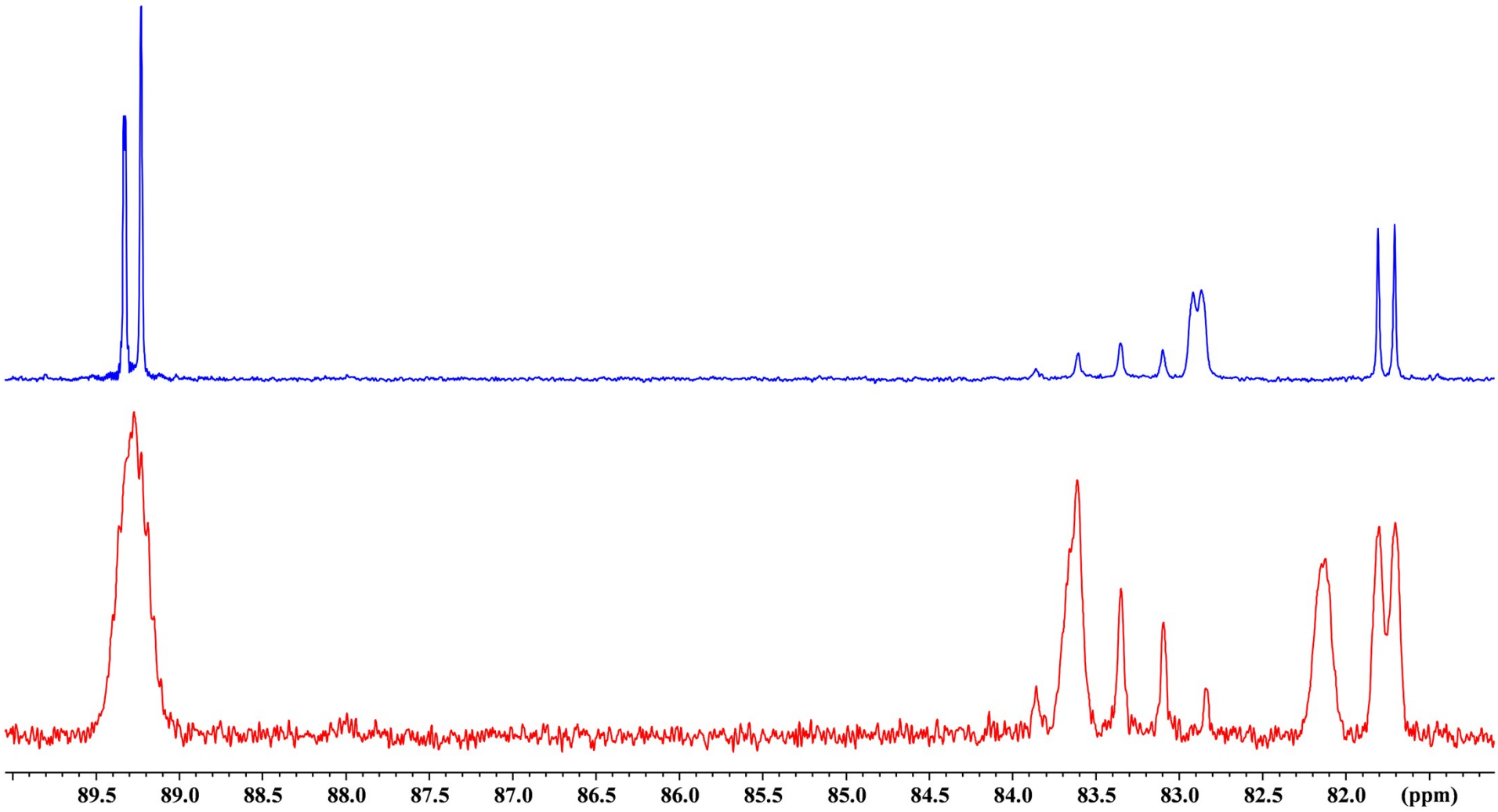


Figure 40. High-field fragments of ^{13}C and ^{13}C-{^{1}H} spectra (100.6 MHz, DMF-d_7, 25°C) of phospholane **16** (the 81-90 ppm field is shown).

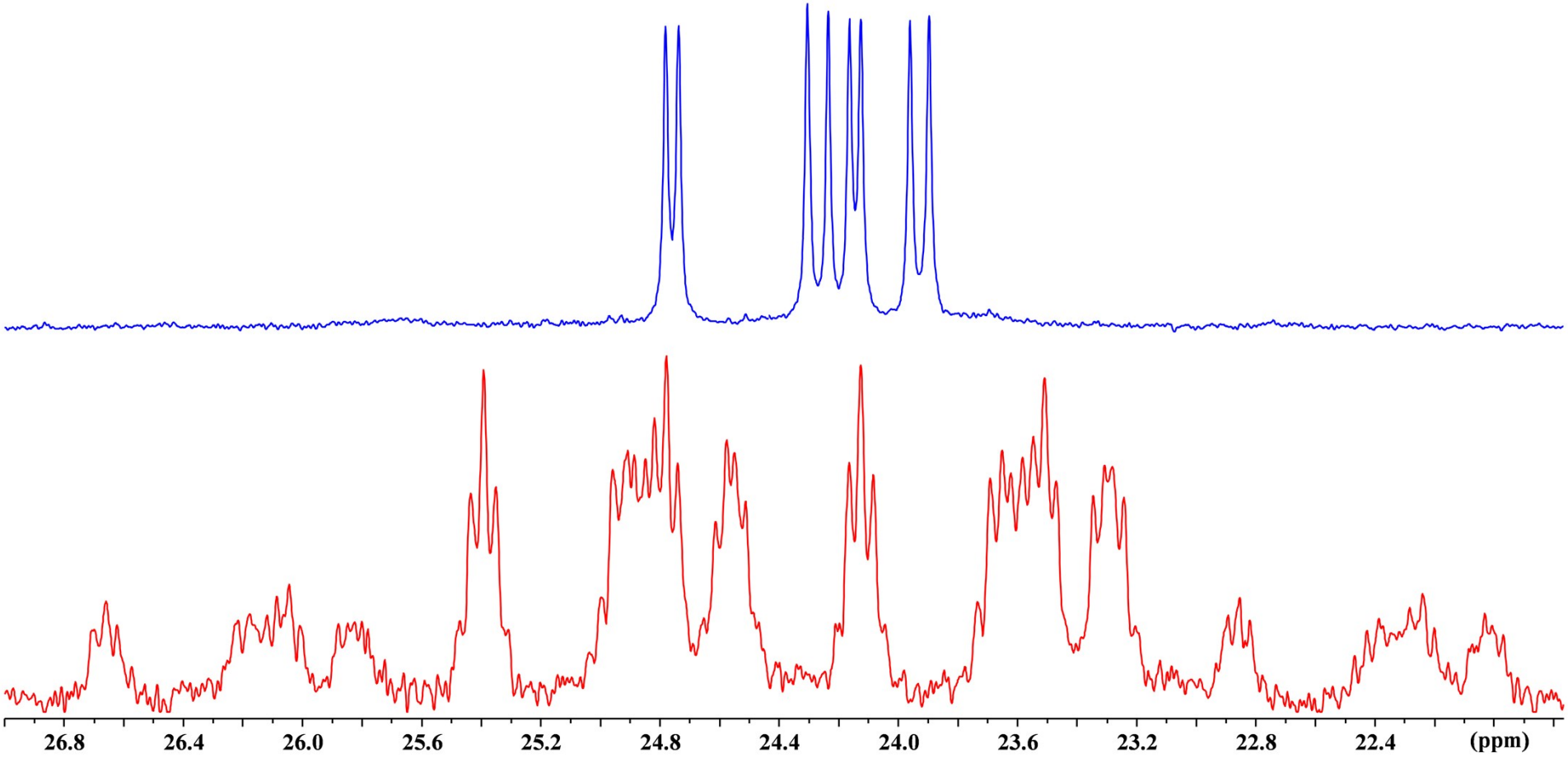


Figure 41. High-field fragments of ^{13}C and ^{13}C-{^{1}H} spectra (100.6 MHz, DMF-d_7, 25°C) of phospholane **16** (the methyl groups region is shown).

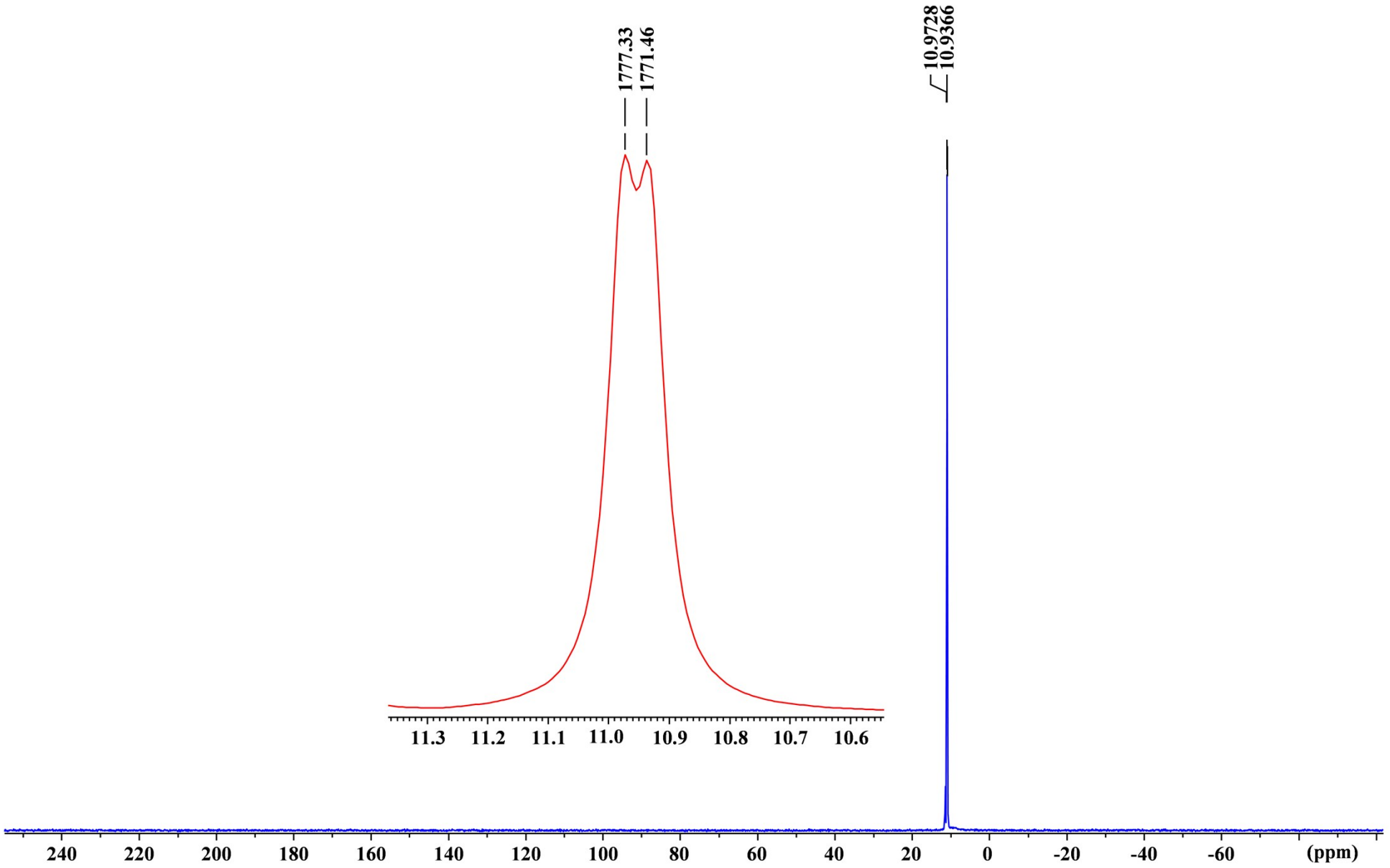


Figure 42. ^{31}P and ^{31}P-{^{1}H} NMR spectra (162.0 MHz, DMF-d_7, 25°C) of phospholane **16**.

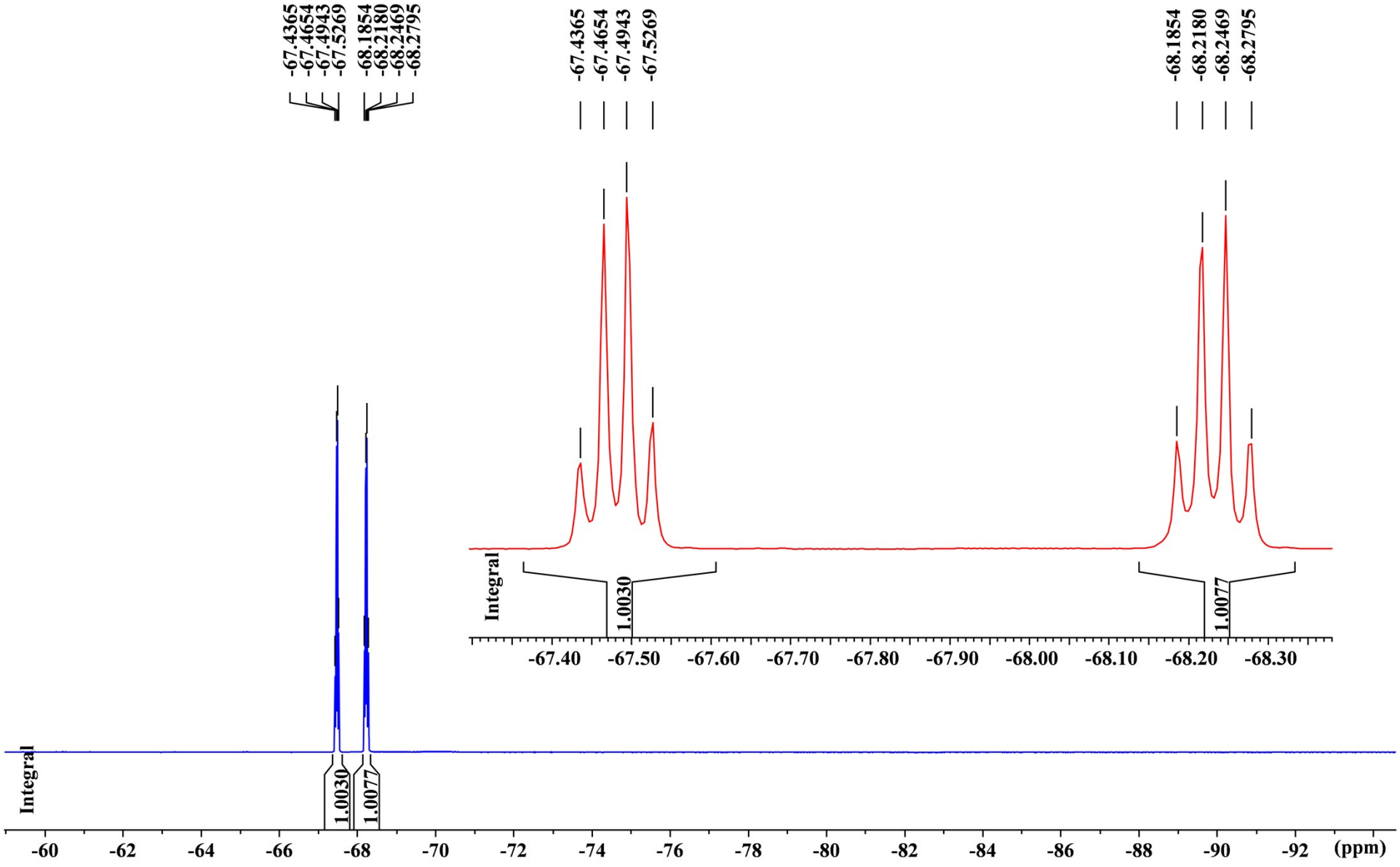


Figure 43. ^{19}F NMR spectrum (376.4 MHz, DMF-d_7, 25°C) of phospholane **16**.

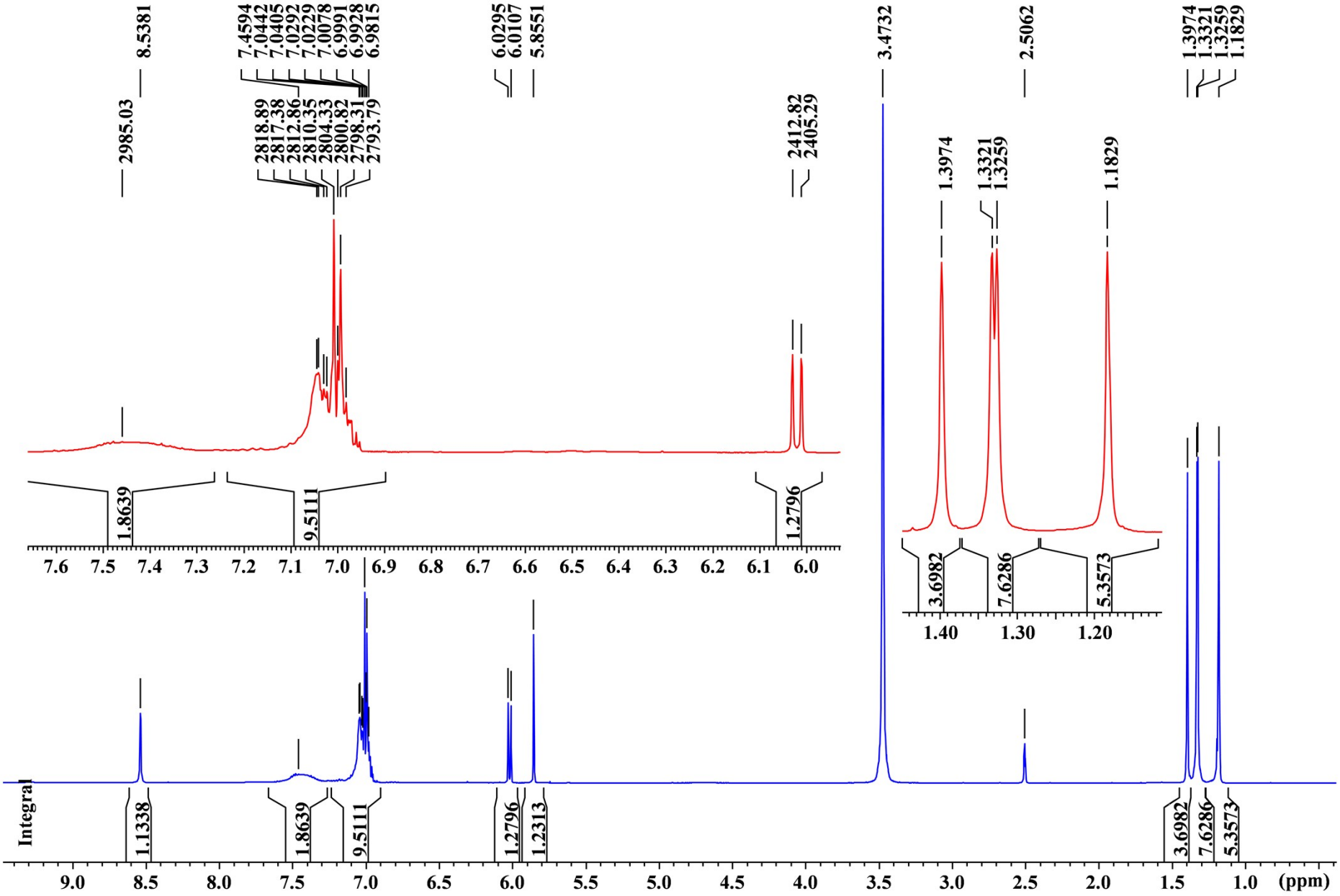


Figure 44. ^{1}H NMR spectrum (400 MHz, DMSO-d_6, 25°C) of phospholane **16**.

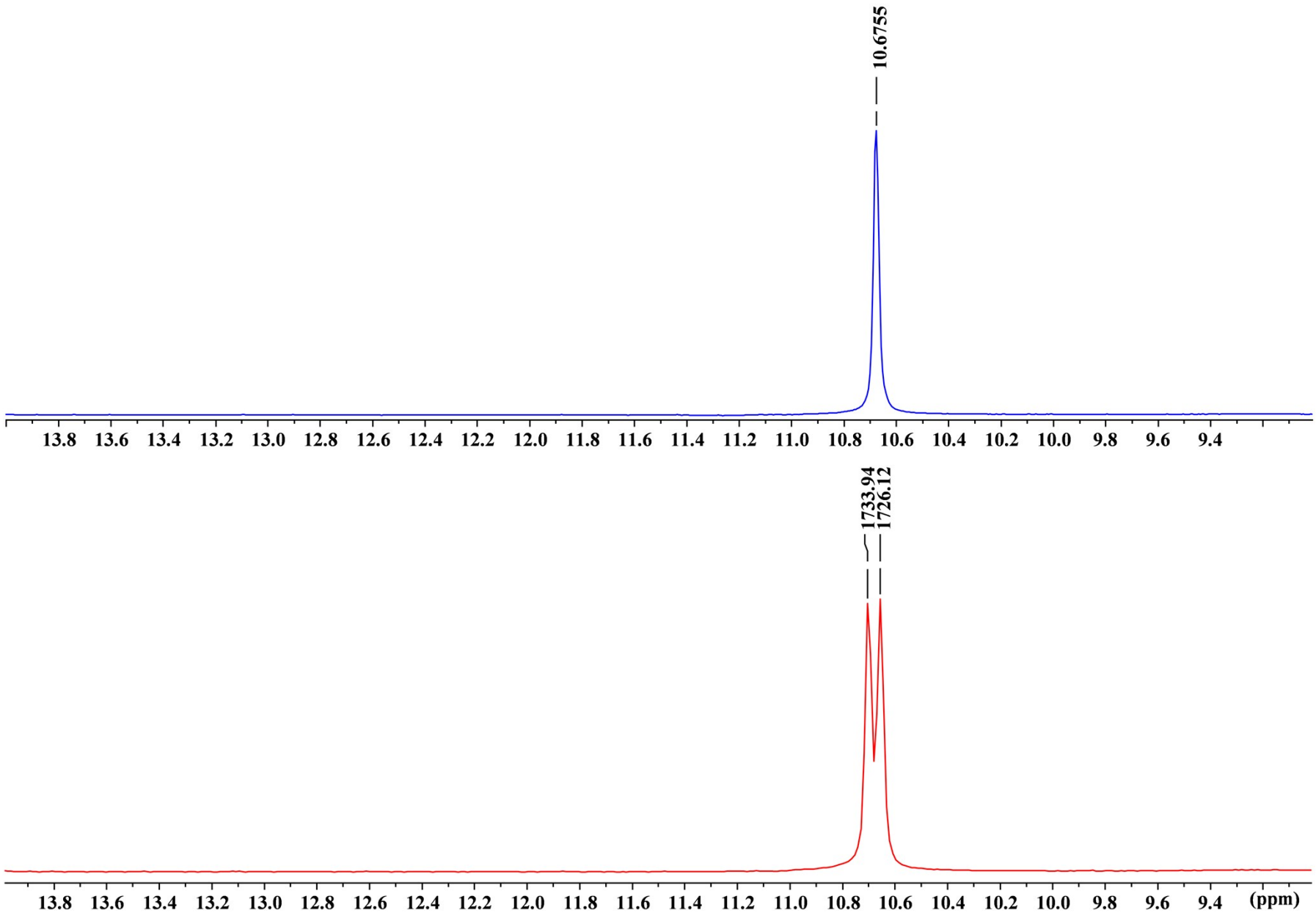


Figure 45. ^{31}P and ^{31}P-{^{1}H} NMR spectra (162.0 MHz, DMSO-d_6, 25°C) of phospholane **16**.

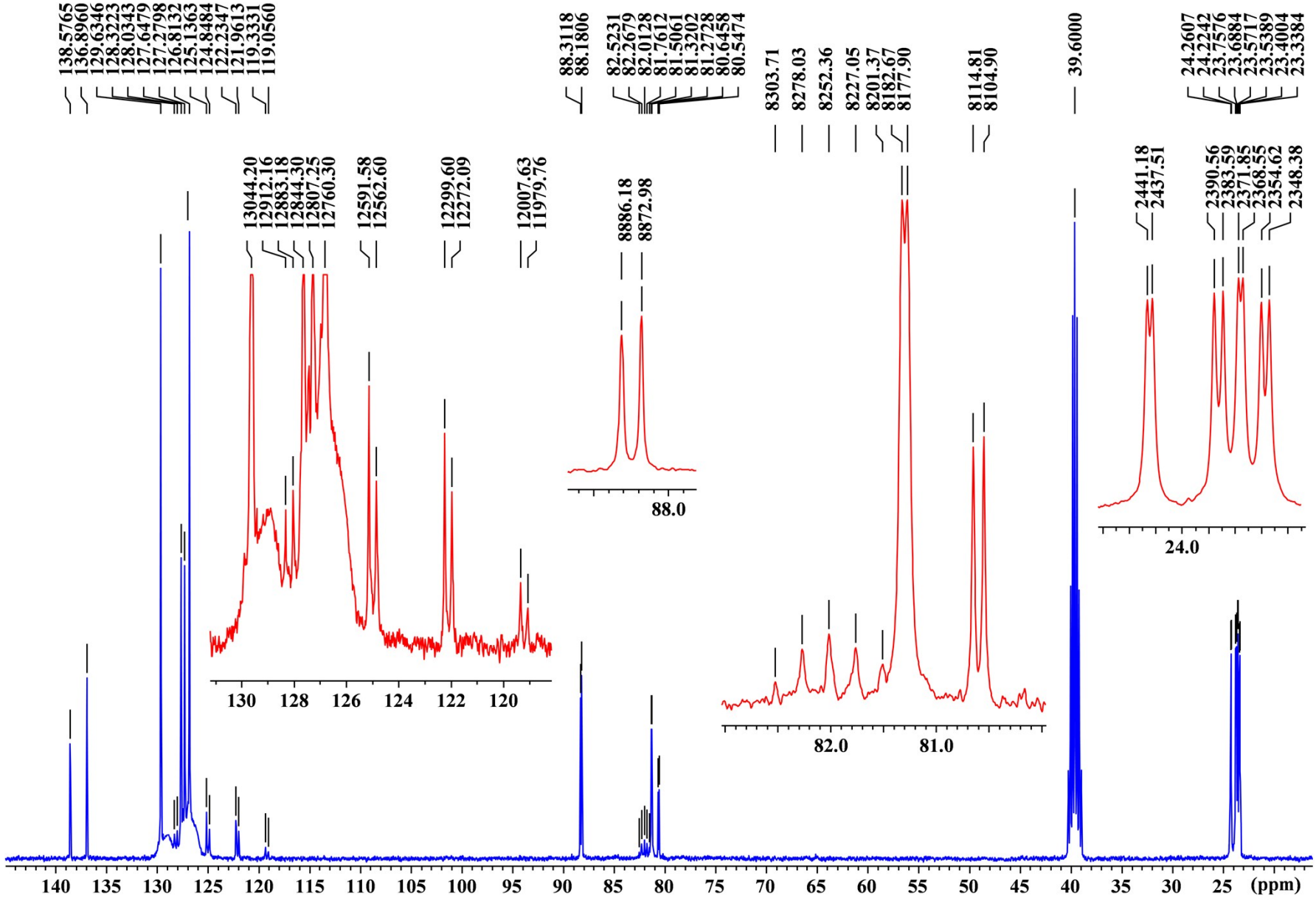


Figure 46. ^{13}C NMR spectrum (100.6 MHz, DMSO-d_6, 25°C) of phospholane **16**.

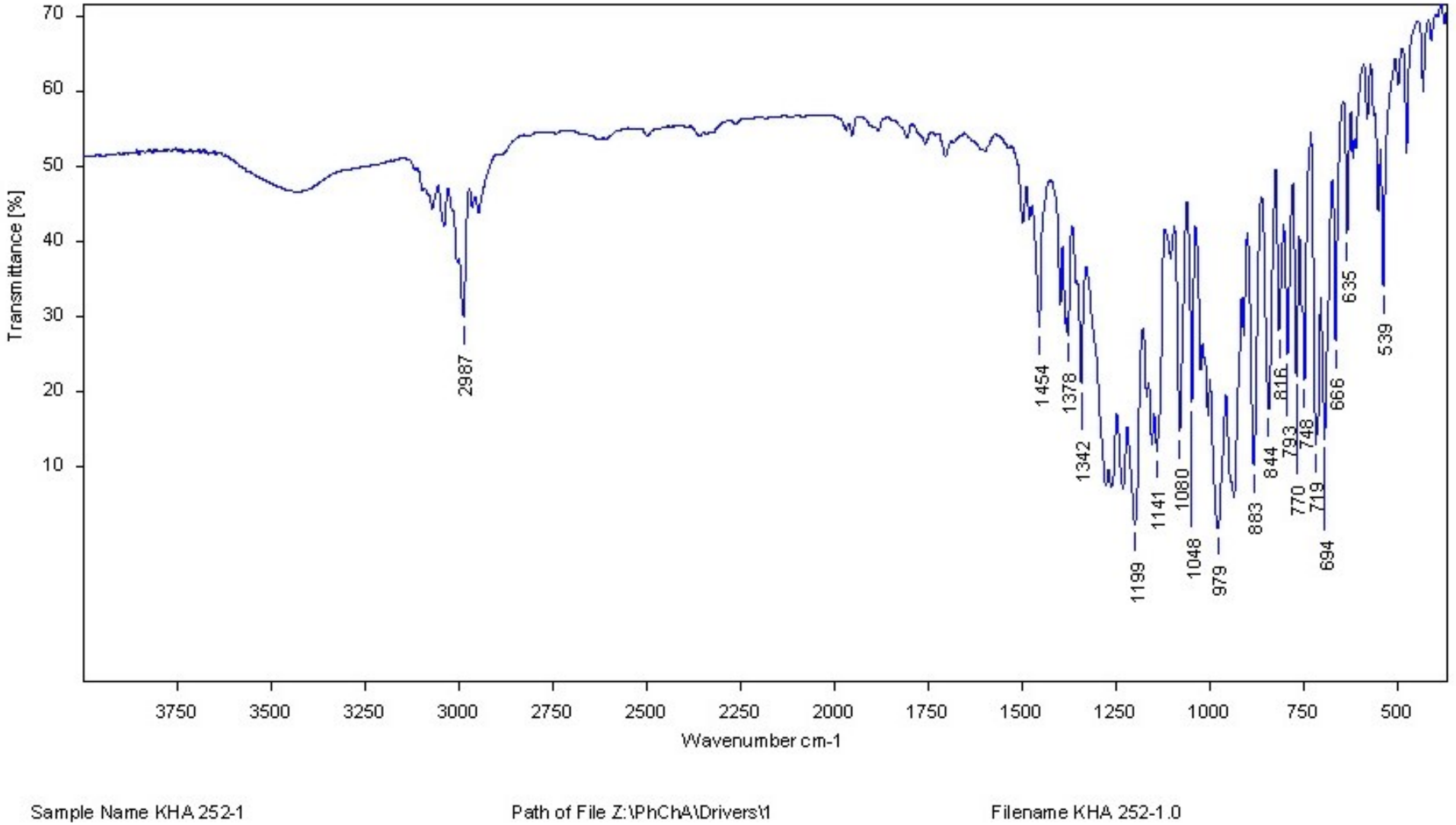


Figure 47. IR spectrum of phosphorane **13** (KBr pellet).

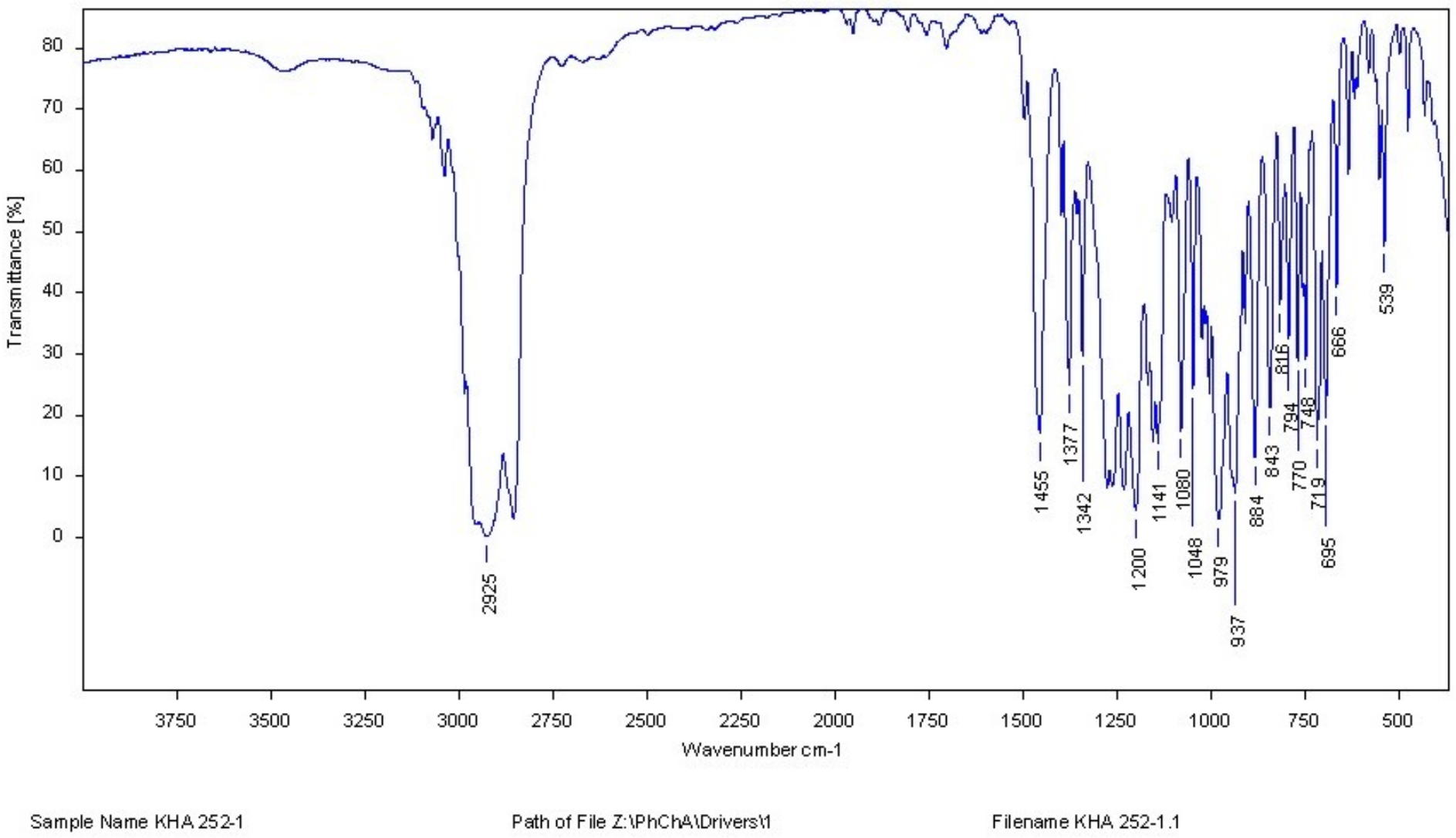


Figure 48. IR spectrum of phosphorane **13** (nujol).

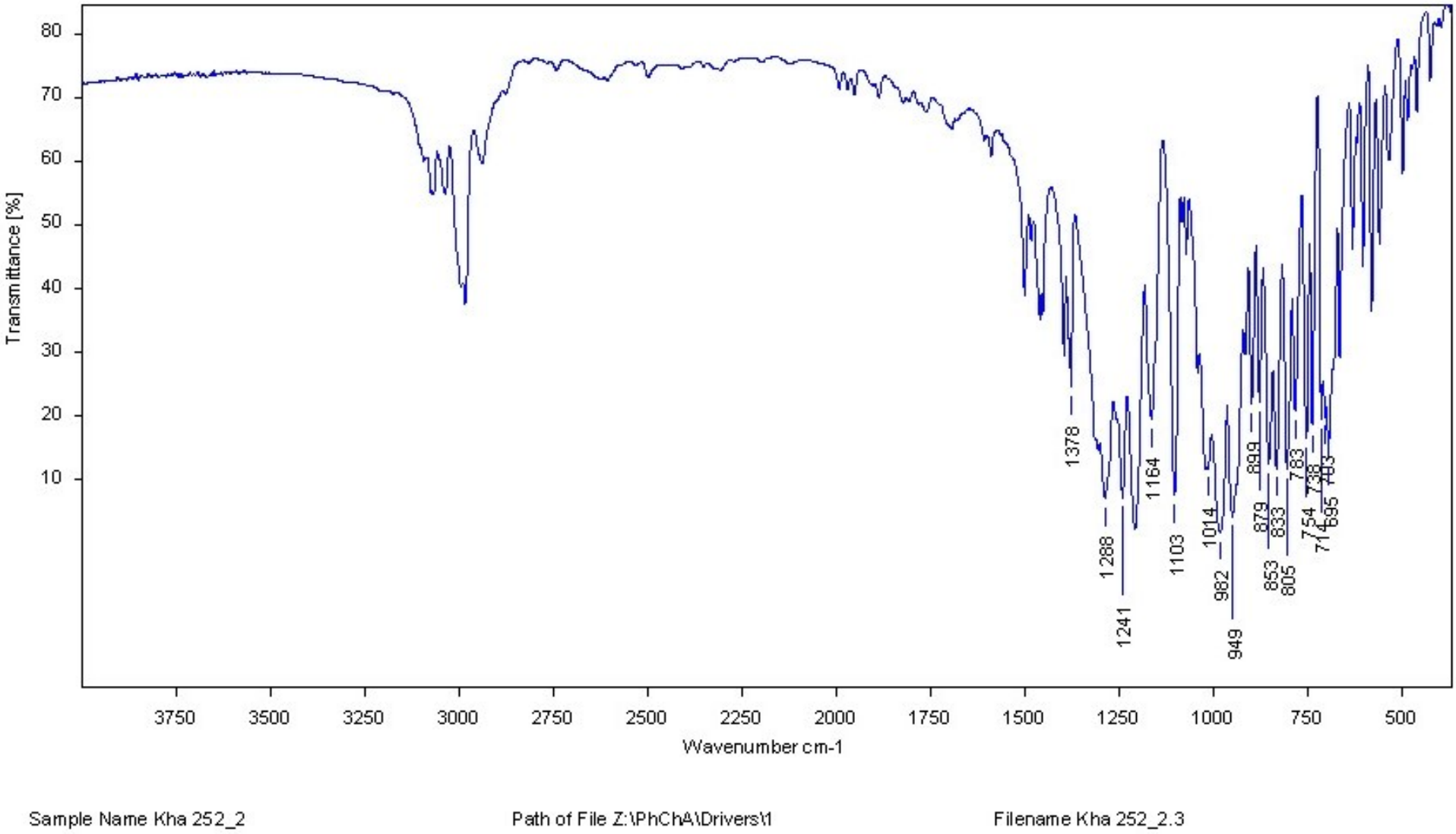


Figure 49. IR spectrum of phosphorane **14** (KBr pellet).

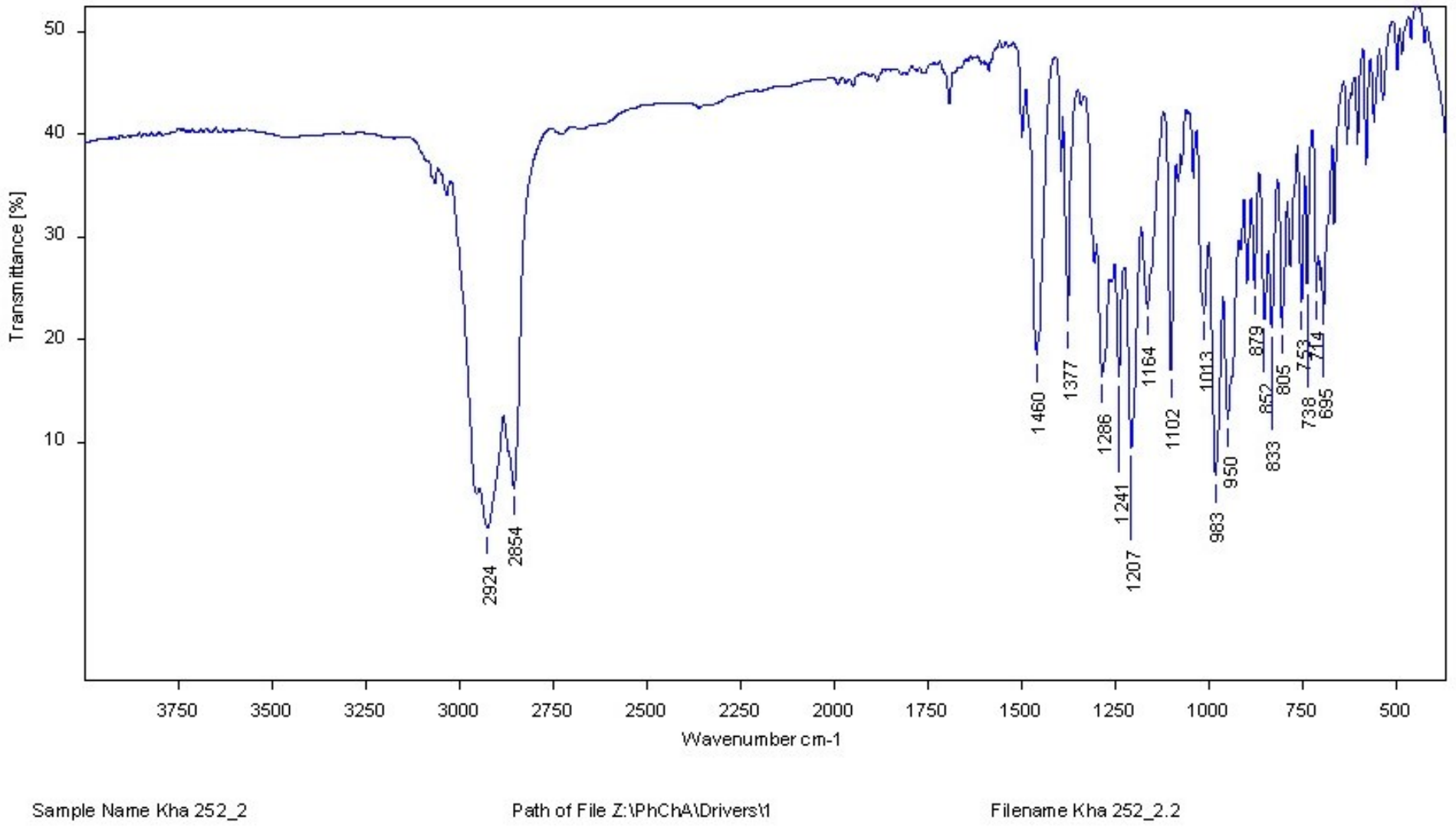


Figure 50. IR spectrum of phosphorane **14** (nujol).

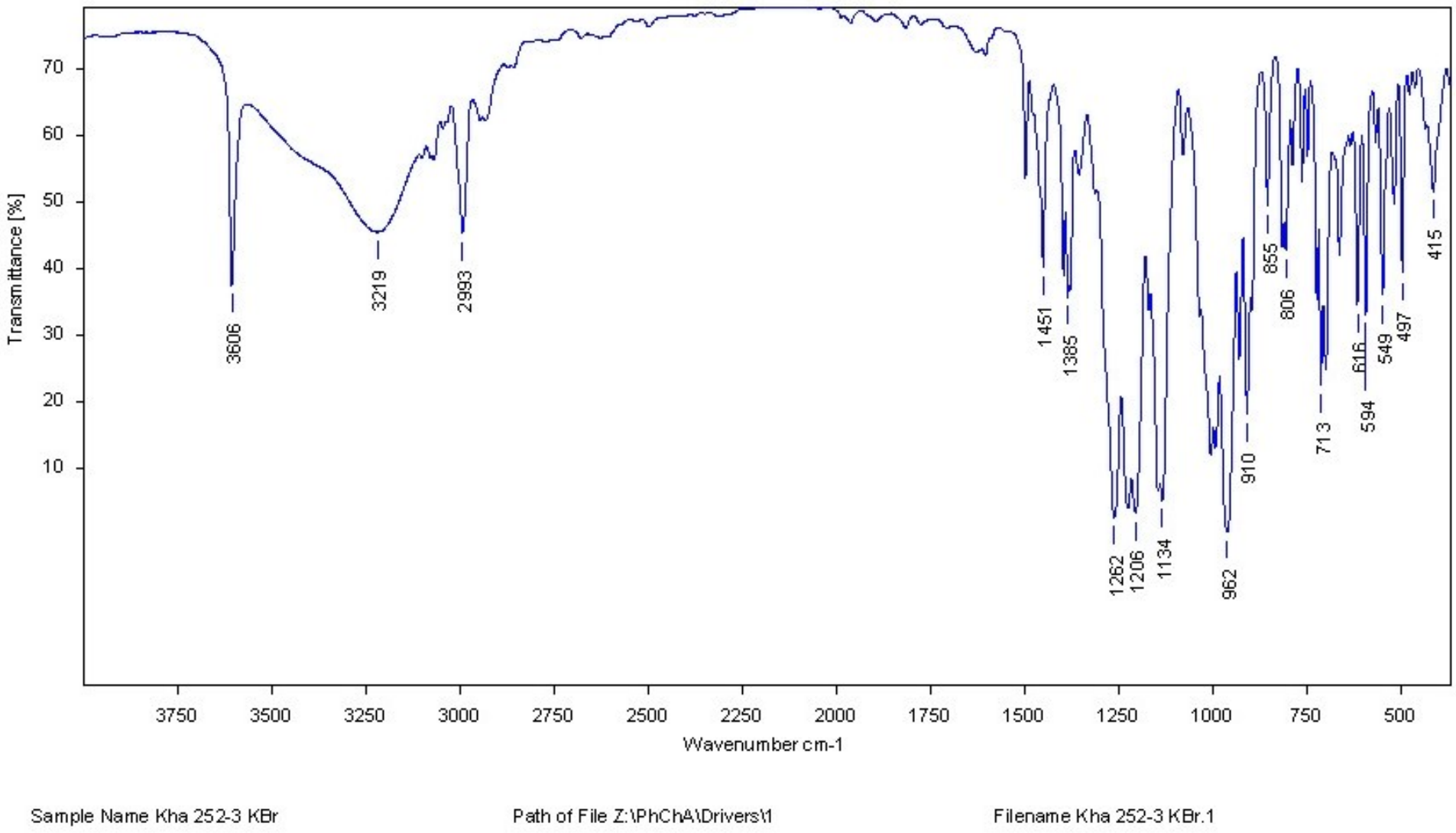


Figure 51. IR spectrum of phospholane **16** (KBr pellet).

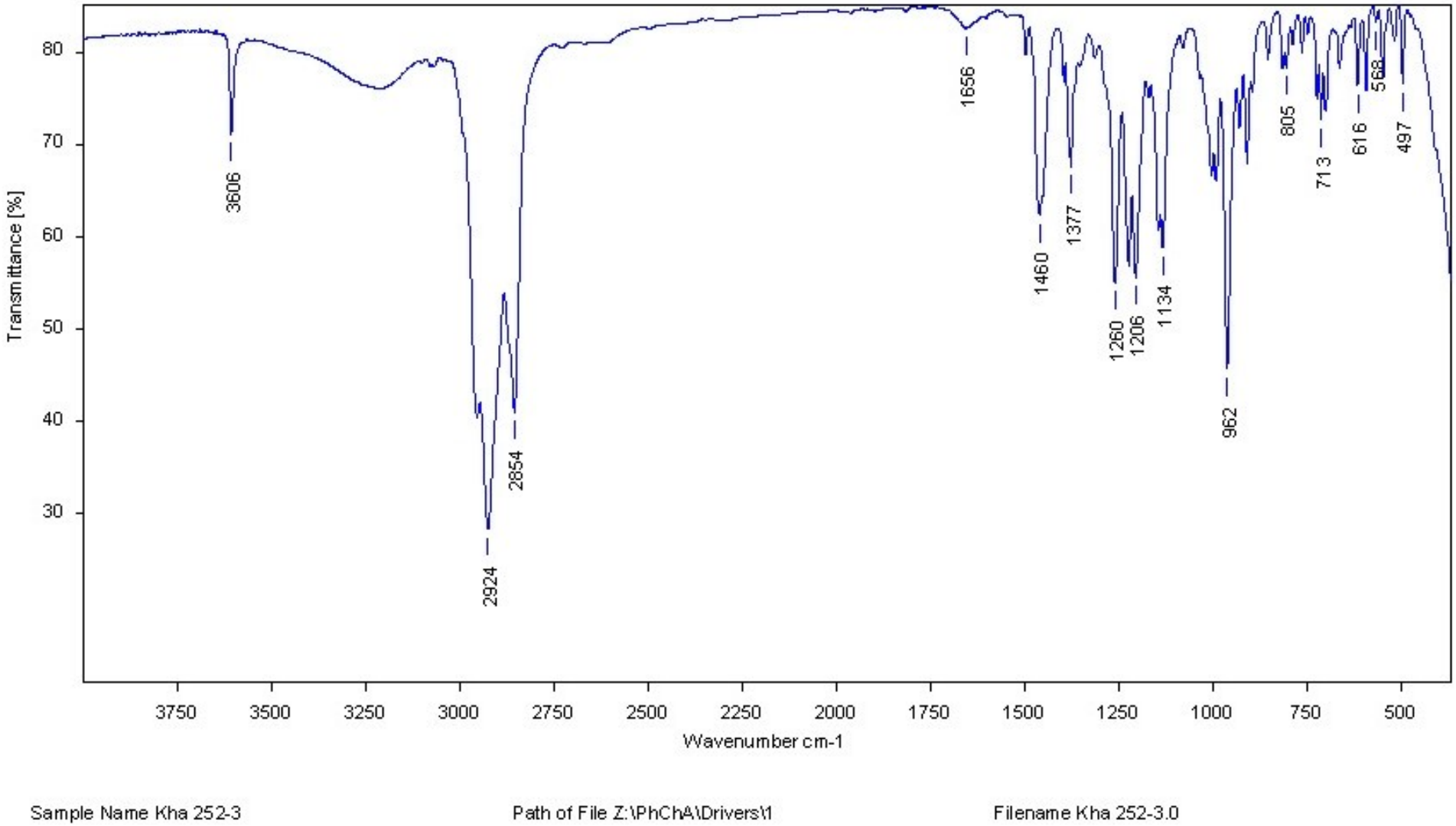


Figure 52. IR spectrum of phospholane **16** (nujol).