Special Issue

Supplementary Information

JBCS 30 years

Synthesis of Bis-*Strychnos* Alkaloids (–)-Sungucine, (–)-Isosungucine, and (–)-Strychnogucine B from (–)-Strychnine

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General information

Single crystal X-ray diffraction was performed with a Mo Kα radiation obtained from a sealed molybdenum tube with a monochromator. The structure was solved using direct methods and refined using full-matrix least squares (SHELXTL). Additional experimental and sample details are given in the crystallographic tables.

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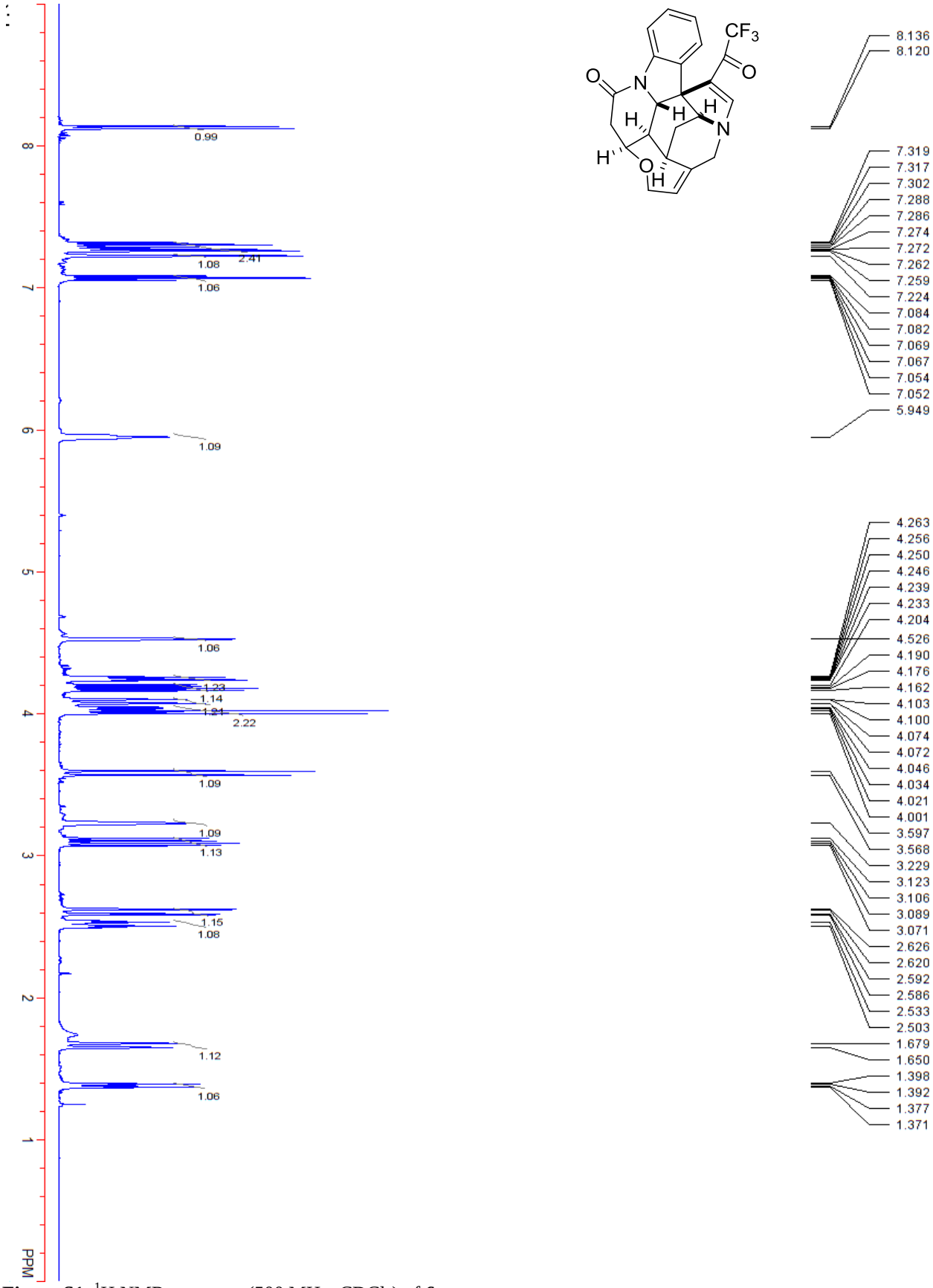

Figure S1. ^{1}H NMR spectrum (500 MHz, $CDCl_3$) of **6**.

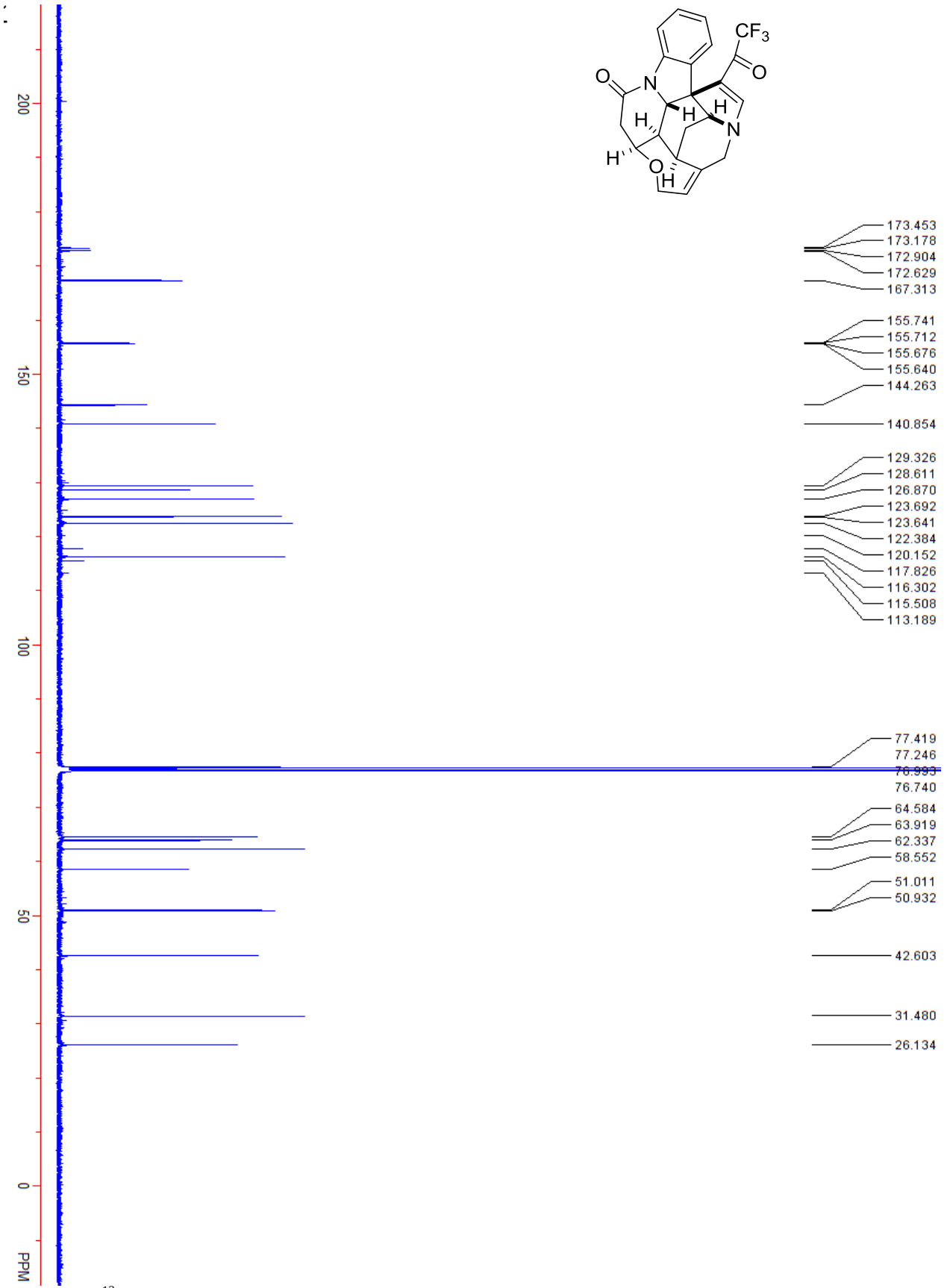

Figure S2. ^{13}C NMR spectrum (125 MHz, $CDCl_3$) of **6**.

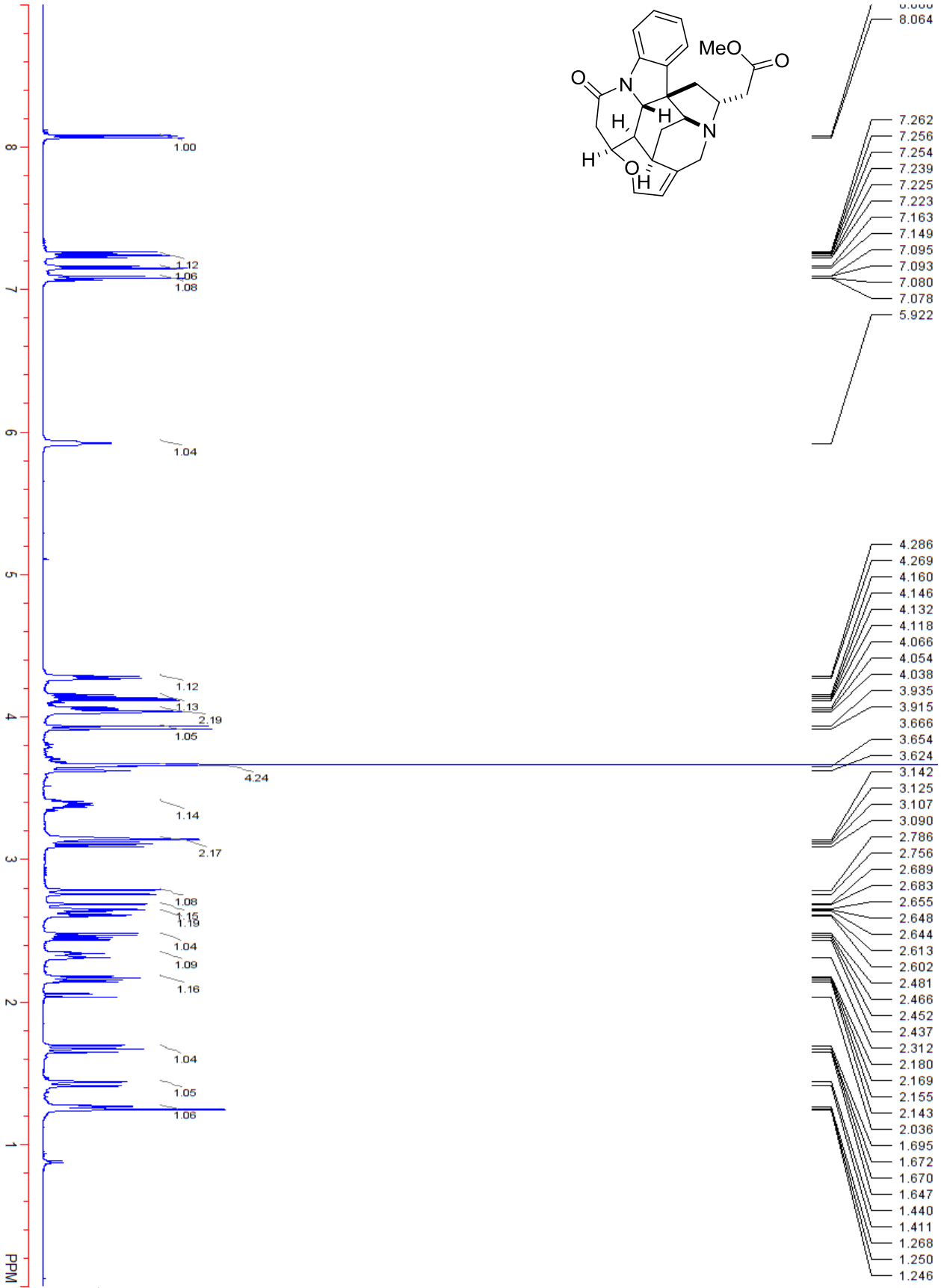

Figure S3. ^{1}H NMR spectrum (500 MHz, $CDCl_3$) of **10**.

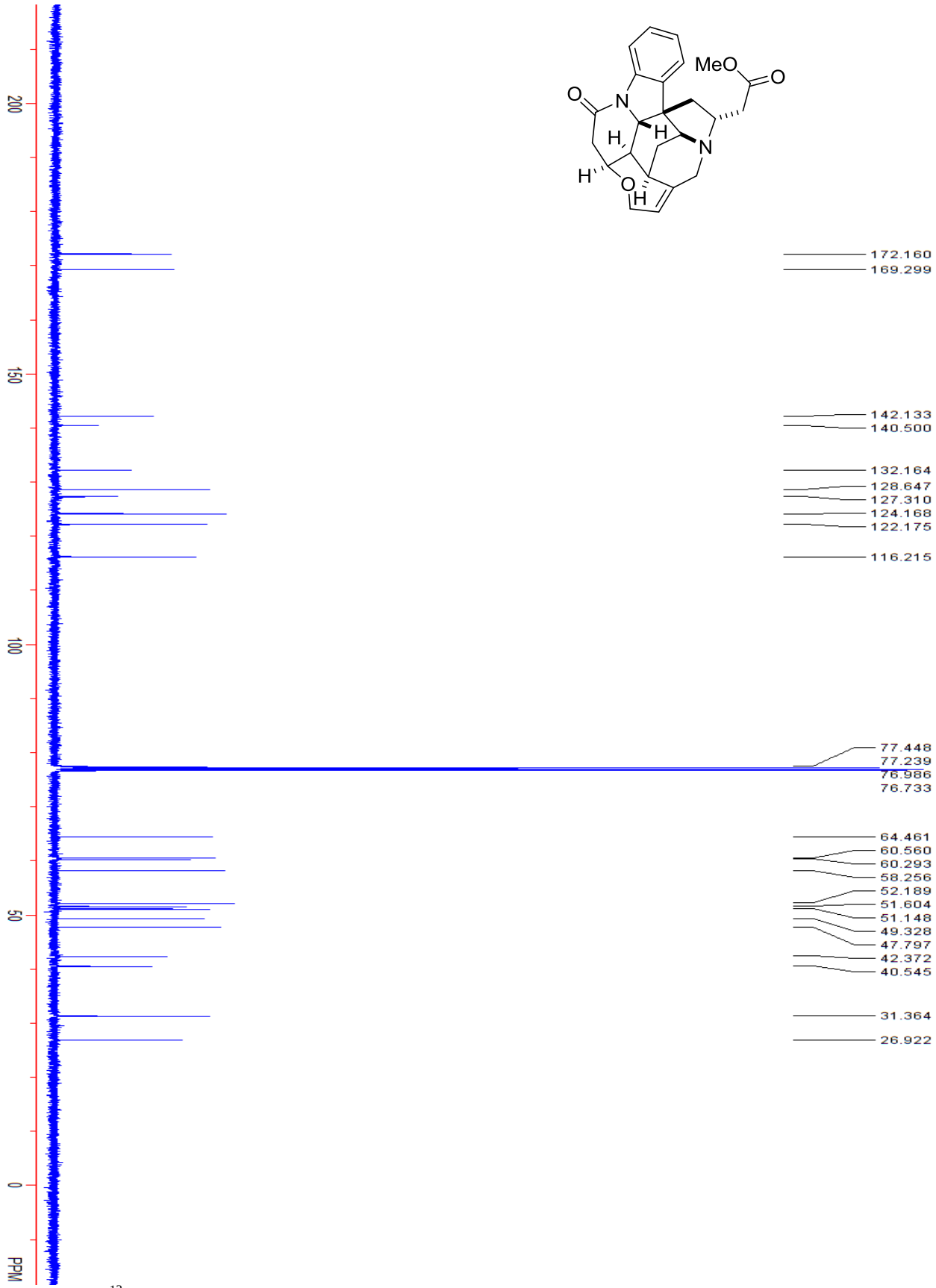

Figure S4. ^{13}C NMR spectrum (125 MHz, $CDCl_3$) of **10**.

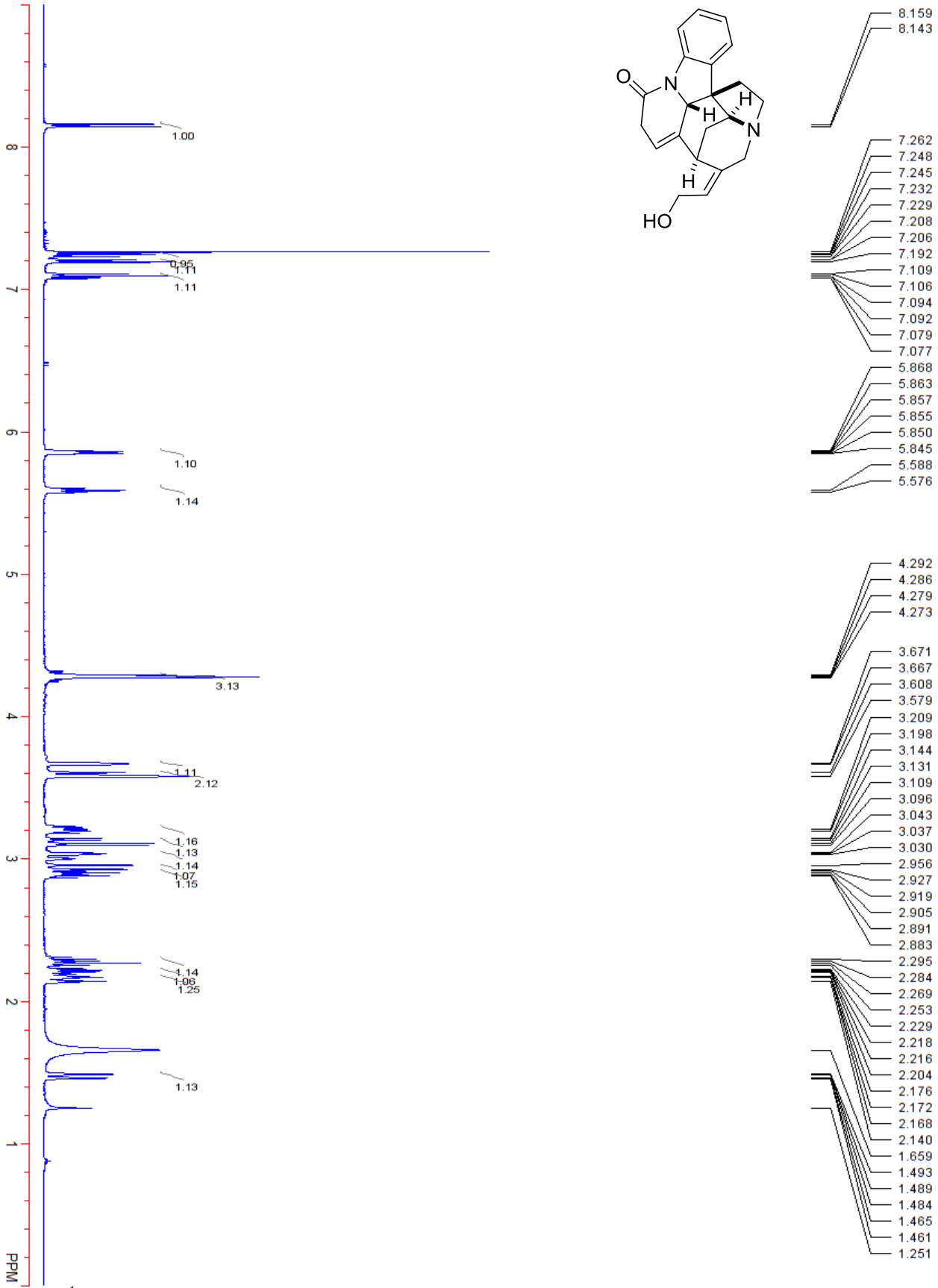

Figure S5. ^{1}H NMR spectrum (500 MHz, $CDCl_3$) of **13**.

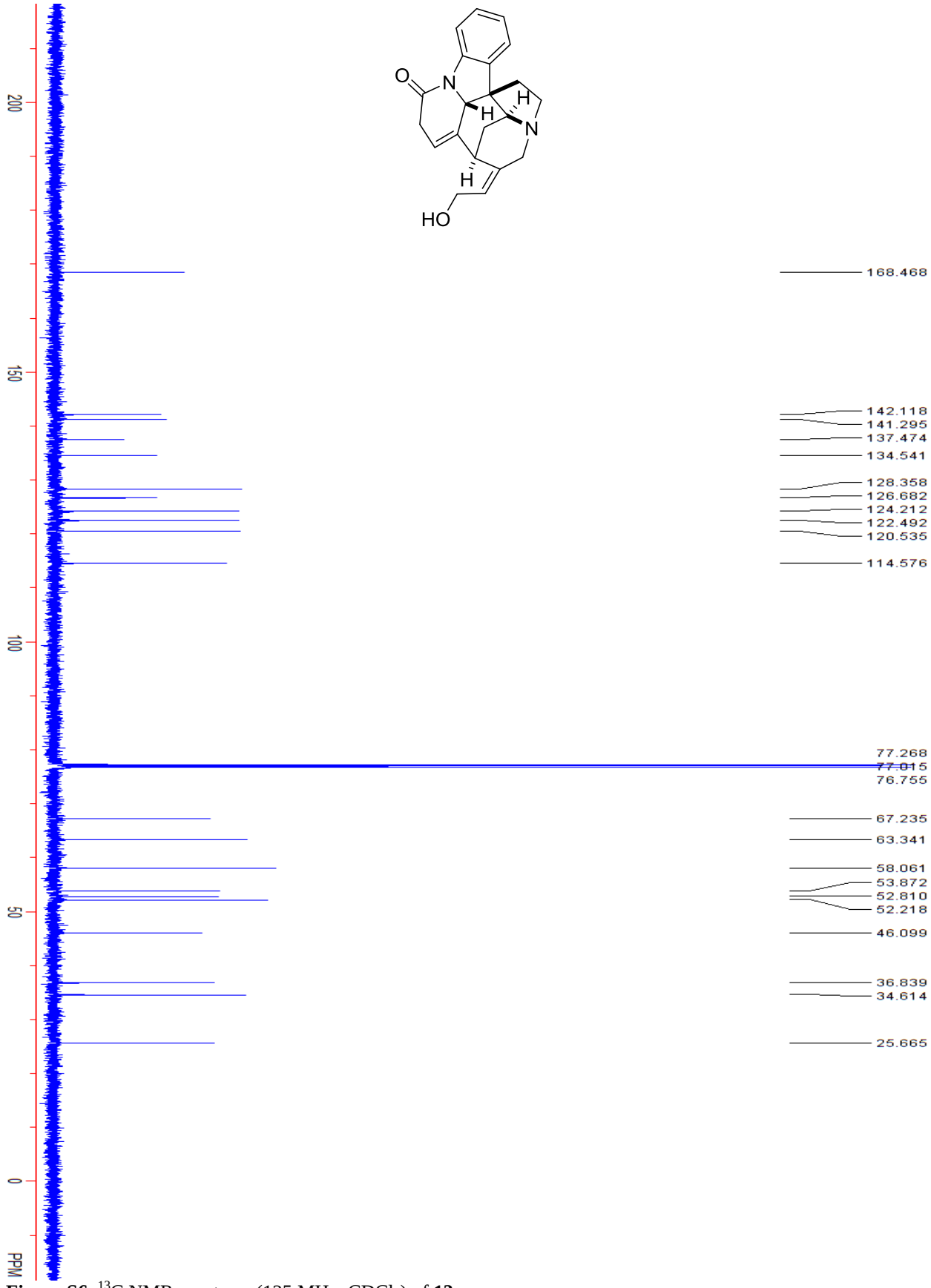

Figure S6. ^{13}C NMR spectrum (125 MHz, $CDCl_3$) of **13**.

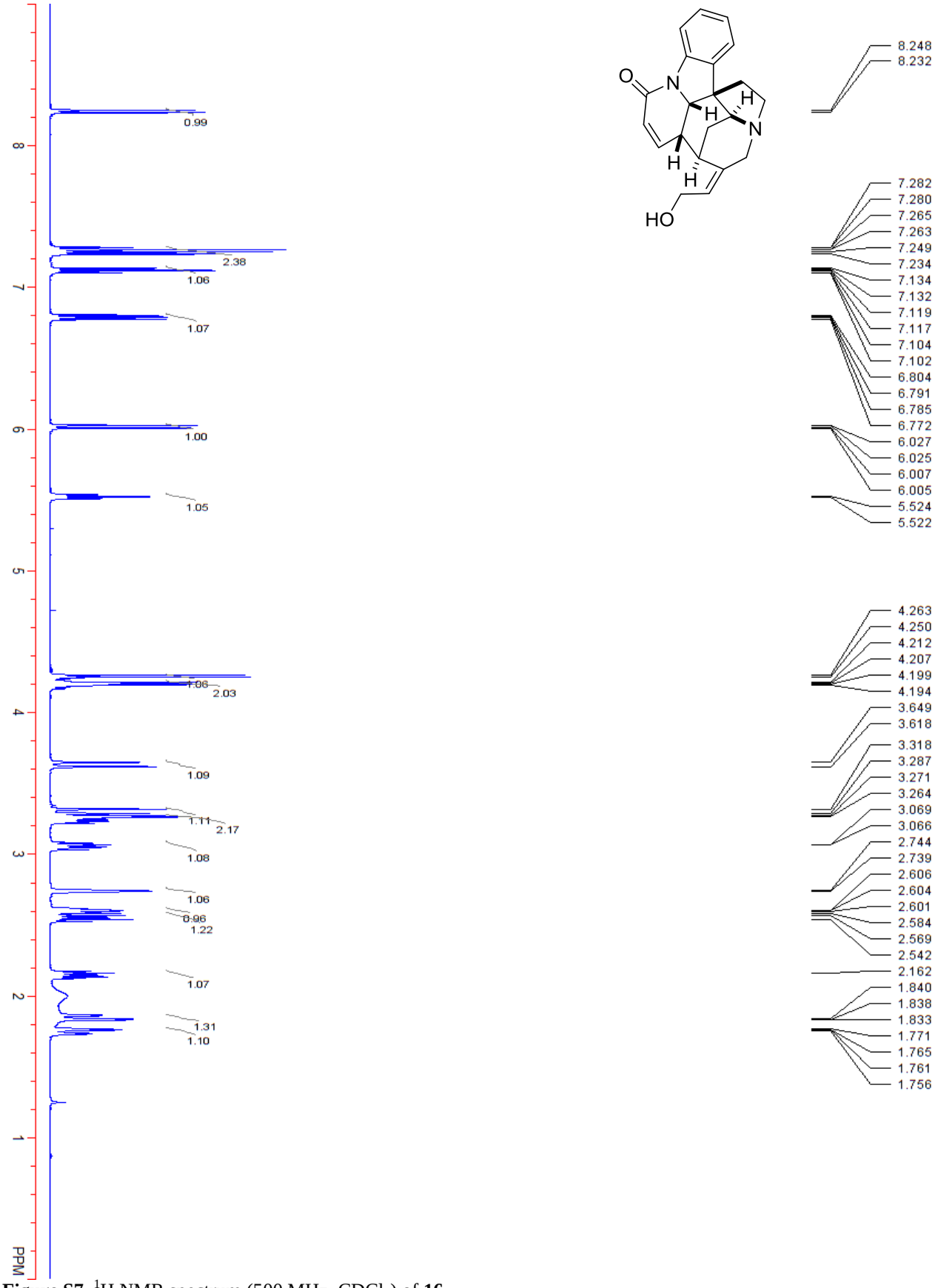

Figure S7. ^{1}H NMR spectrum (500 MHz, $CDCl_3$) of **16**.

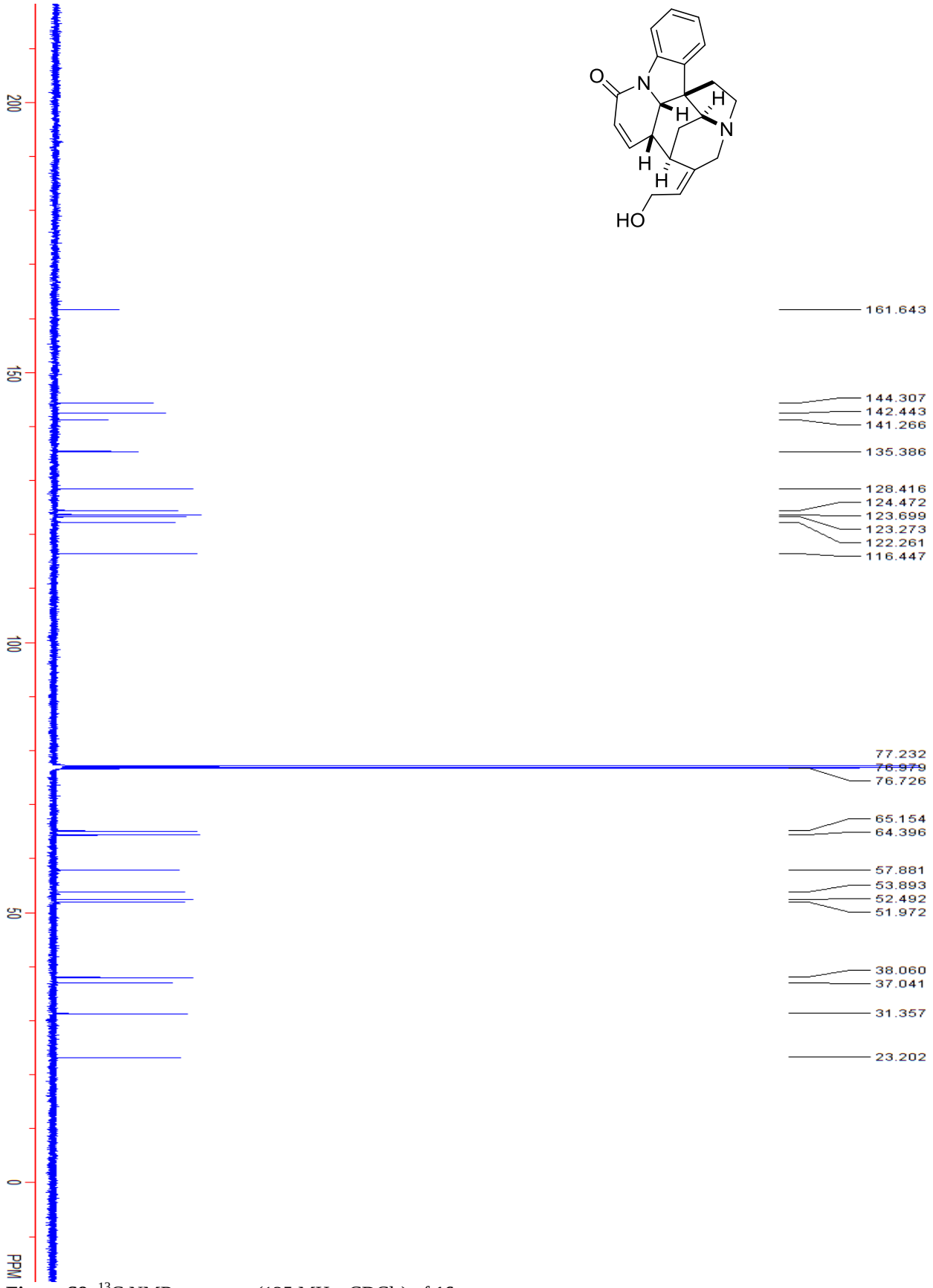

Figure S8. ^{13}C NMR spectrum (125 MHz, $CDCl_3$) of **16**.

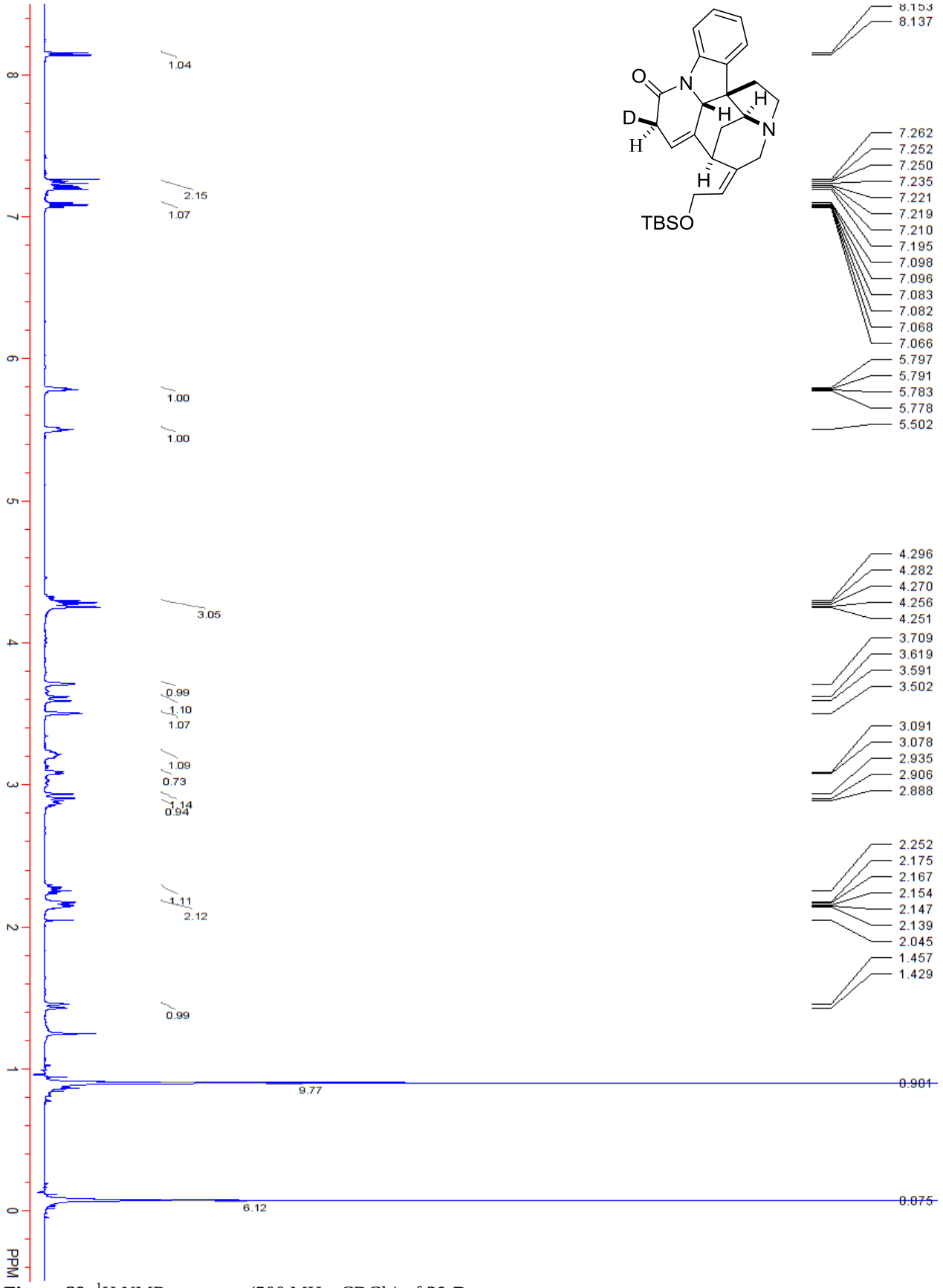

Figure S9. ^{1}H NMR spectrum (500 MHz, $CDCl_3$) of **20-D**.

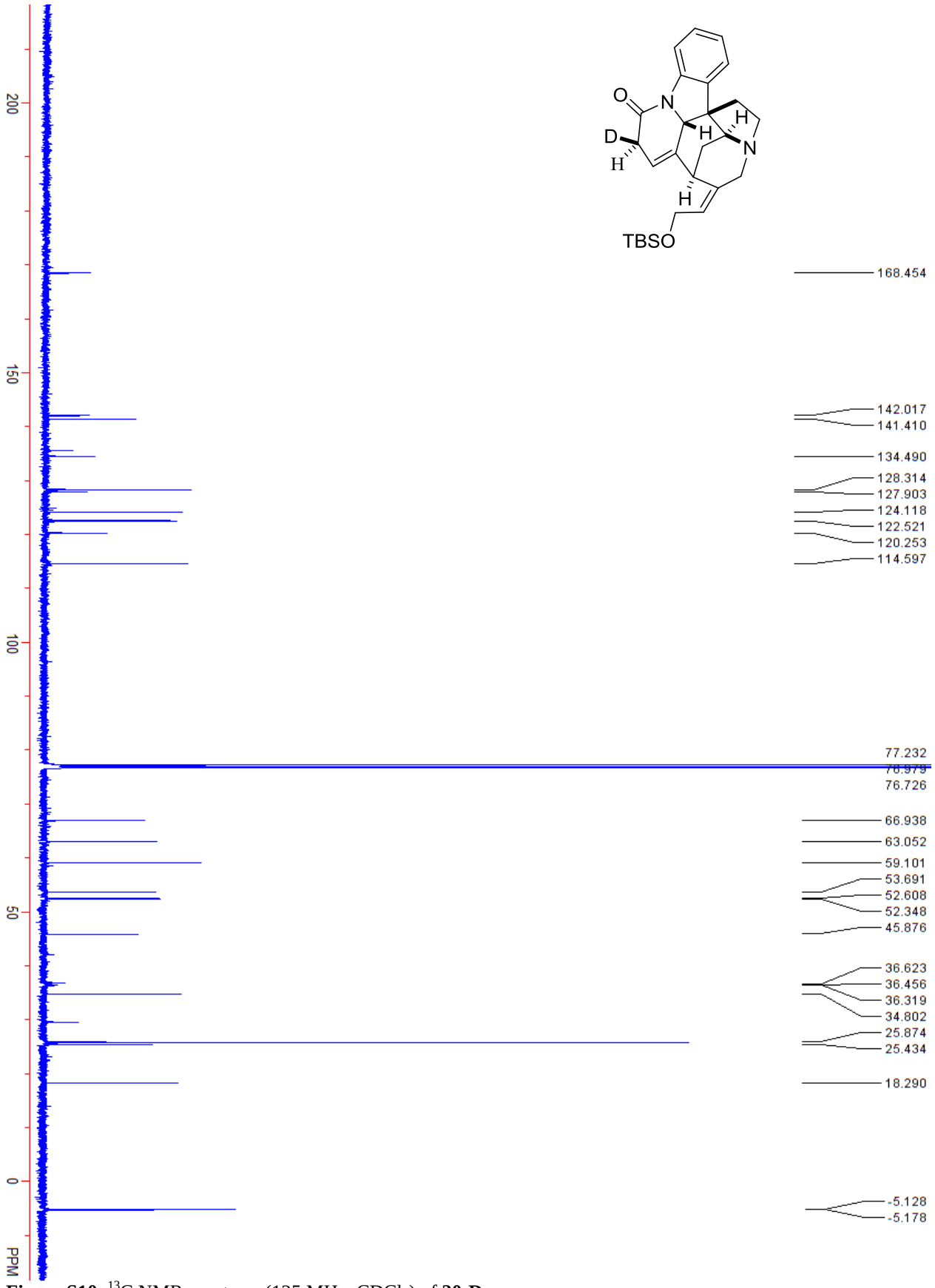

Figure S10. ^{13}C NMR spectrum (125 MHz, $CDCl_3$) of **20-D**.

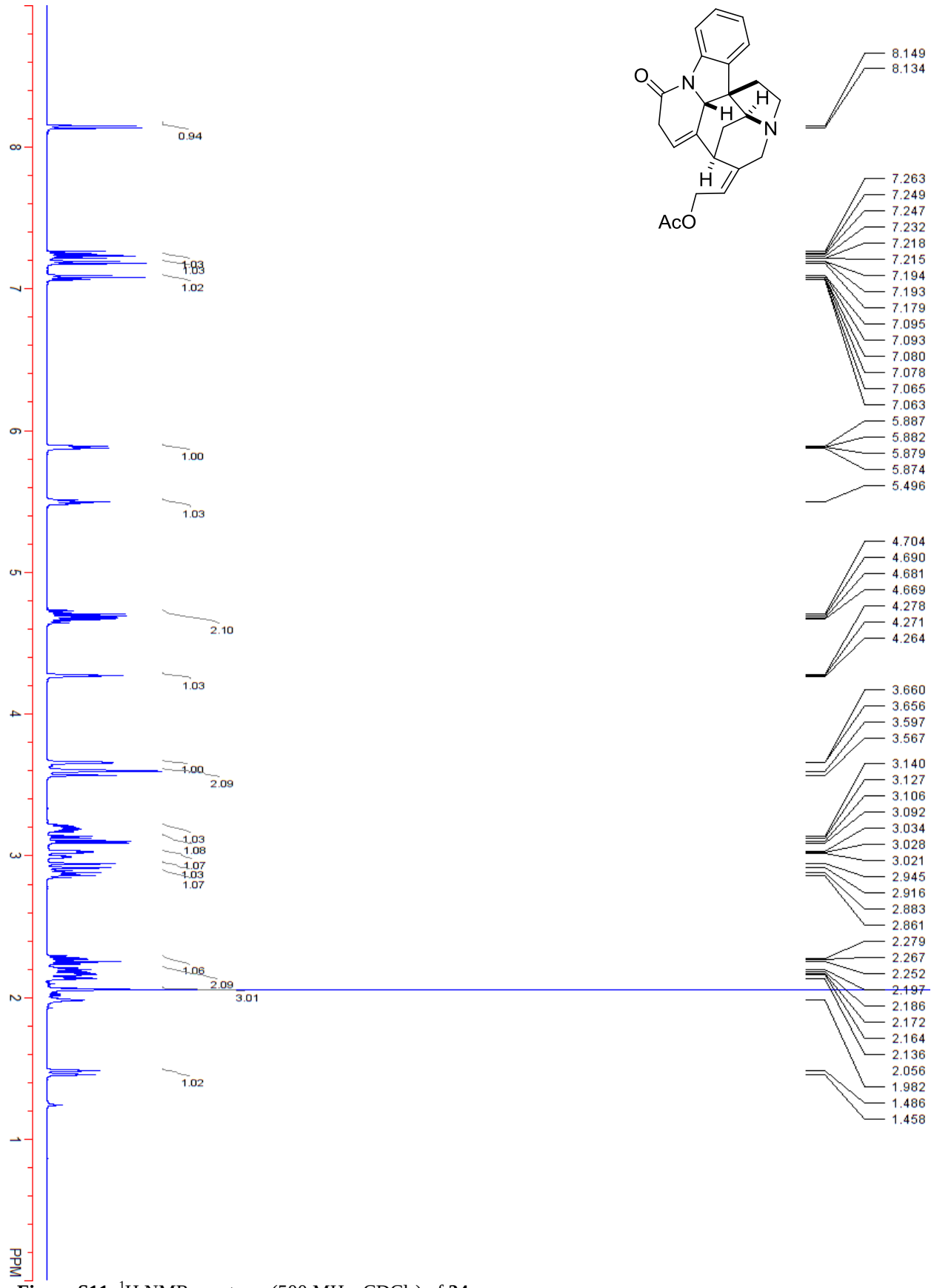

Figure S11. ^{1}H NMR spectrum (500 MHz, $CDCl_3$) of **24**.

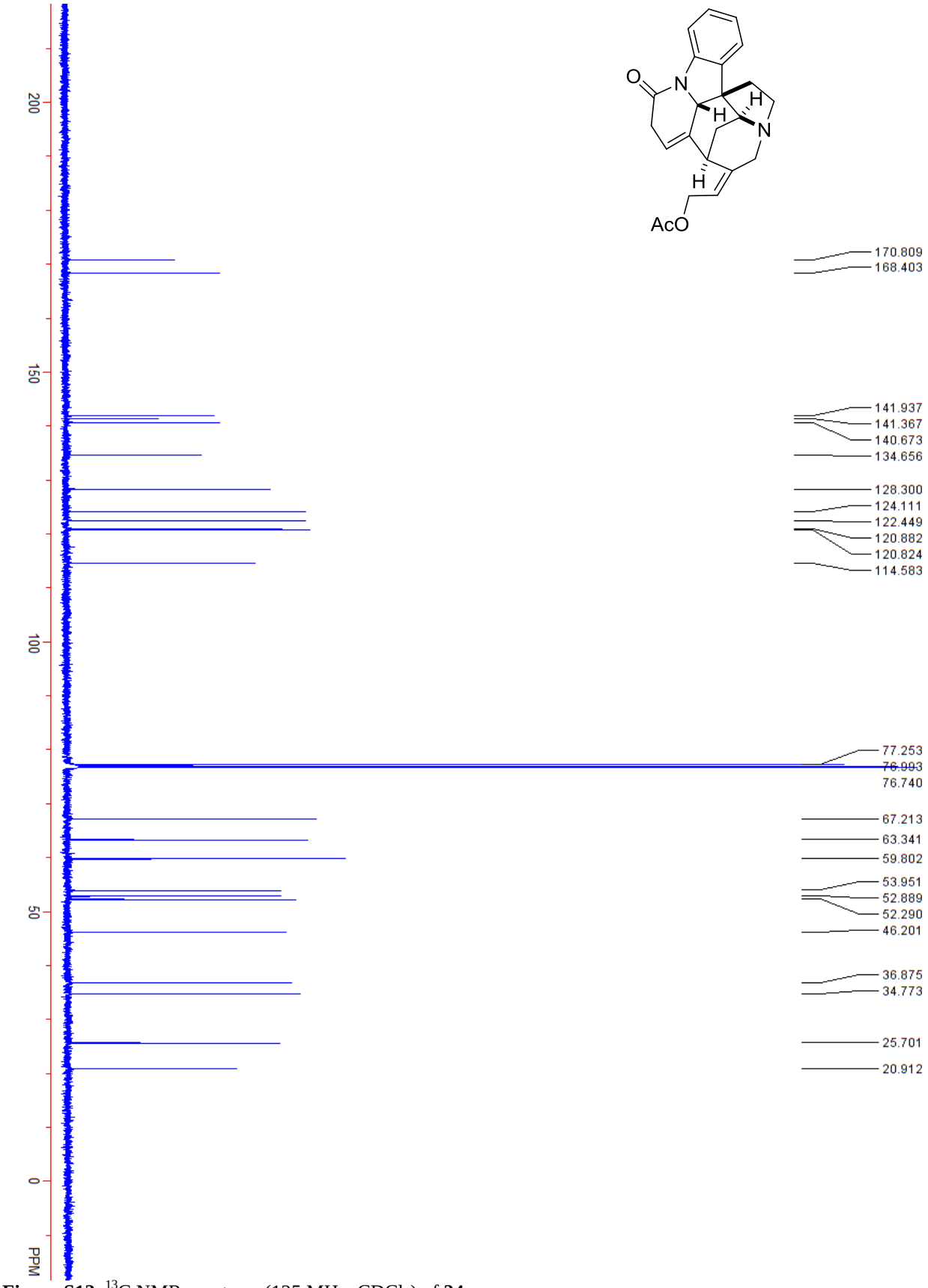

Figure S12. ^{13}C NMR spectrum (125 MHz, $CDCl_3$) of **24**.

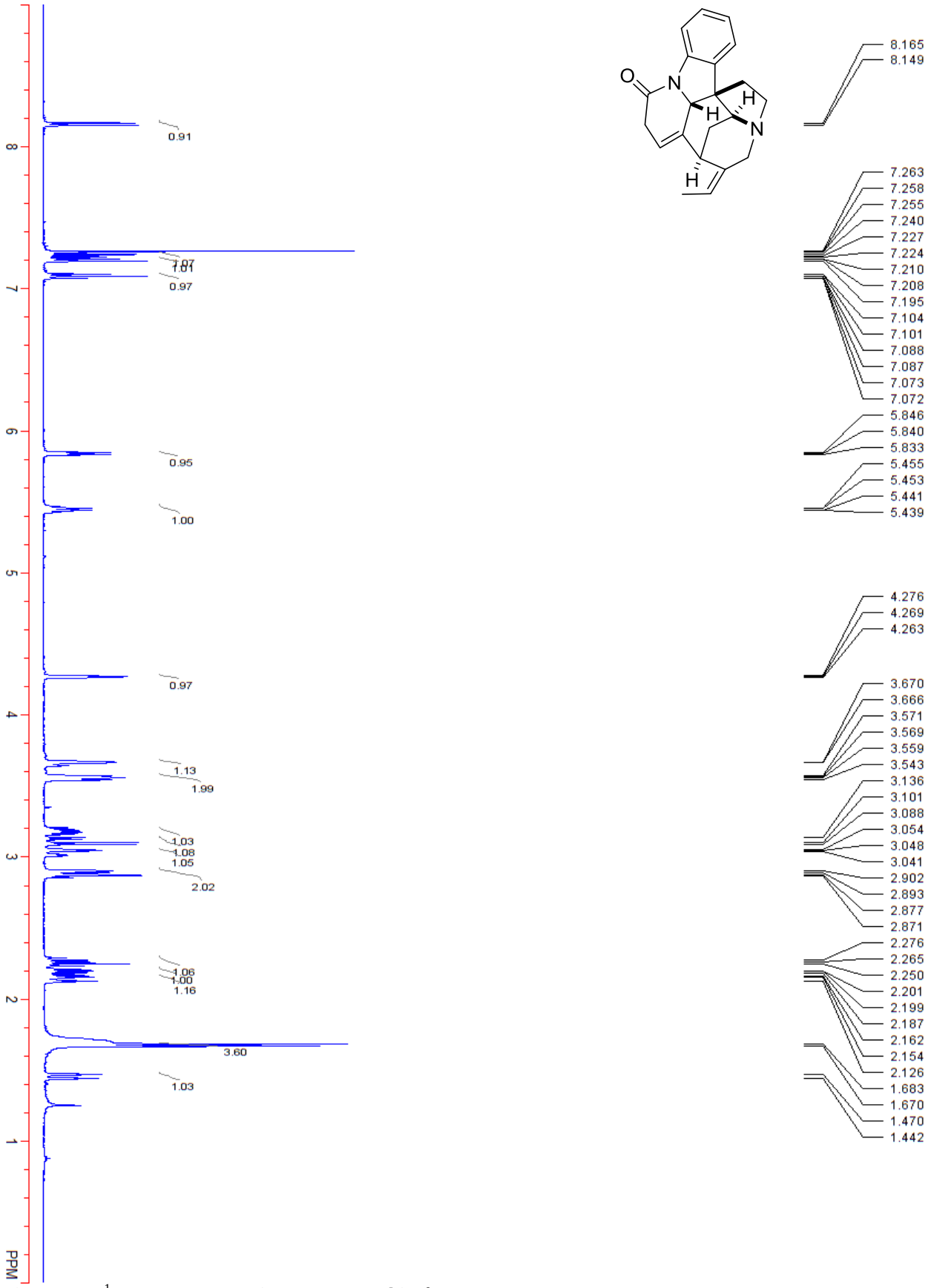

Figure S13. ^{1}H NMR spectrum (500 MHz, $CDCl_3$) of **26**.

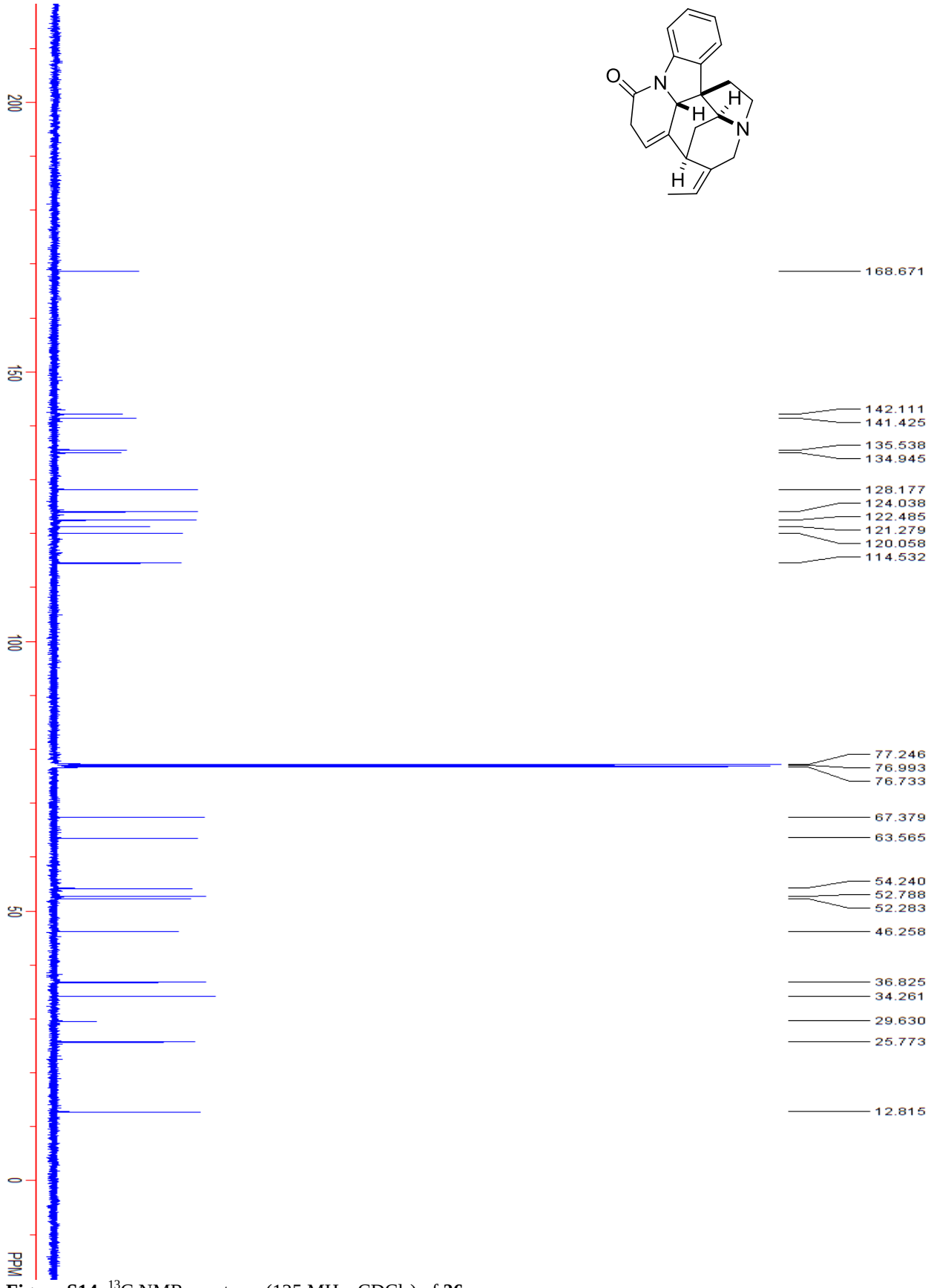

Figure S14. ^{13}C NMR spectrum (125 MHz, $CDCl_3$) of **26**.

Crystal structure report for **6**

A specimen of $C_{23}H_{19}F_3N_2O_3$, approximate dimensions of 0.040 × 0.160 × 0.220 mm, was used for the X-ray crystallographic analysis. The X-ray intensity data were measured.

The integration of the data using a triclinic unit cell yielded a total of 8438 reflections to a maximum θ angle of 28.00° (0.76 Å resolution), of which 7331 were independent (average redundancy 1.151, completeness = 98.8%, R_{int} = 1.30%, R_{sig} = 2.24%) and 7084 (96.63%) were greater than $2\sigma(F^2)$. The final cell constants of *a* = 7.2817(10) Å, *b* = 10.2276(15) Å, *c* = 13.916(2) Å, α = 77.657(2)°, β = 78.862(2)°, γ = 72.059(2)°, volume = 954.1(2) $Å^3$, are based upon the refinement of the XYZ-centroids of reflections above 20 σ(I). The calculated minimum and maximum transmission coefficients (based on crystal size) are 0.6907 and 0.7456.

The structure was solved and refined using the Bruker SHELXTL Software Package, using the space group P 1, with Z = 2 for the formula unit, $C_{23}H_{19}F_3N_2O_3$. The final anisotropic full-matrix least-squares refinement on F^2 with 587 variables converged at R1 = 3.05%, for the observed data and wR2 = 7.83% for all data. The goodness-of-fit was 1.034. The largest peak in the final difference electron density synthesis was 0.321 $eÅ^{-3}$ and the largest hole was –0.214 $eÅ^{-3}$ with an RMS deviation of 0.039 $eÅ^{-3}$. On the basis of the final model, the calculated density was 1.491 g cm^{-3} and F(000), 444 e^-.

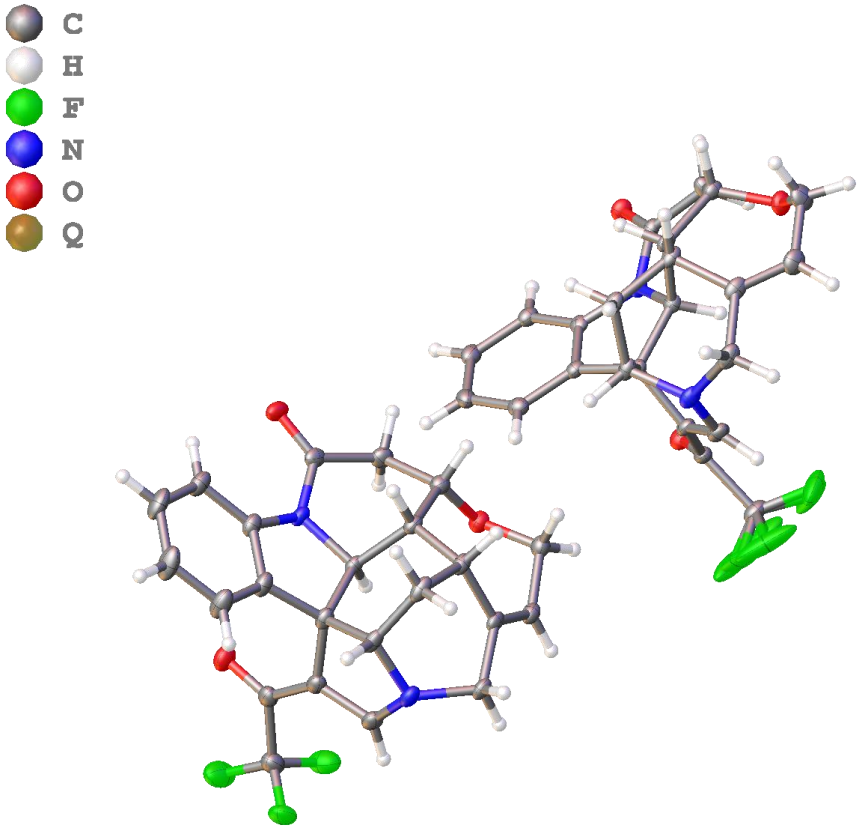

Table S1. Sample and crystal data for 6_1

Identification code	6_1	
Chemical formula	$C_{23}H_{19}F_3N_2O_3$	
Formula weight / (g mol^{-1})	428.40	
Wavelength / Å	0.71073	
Crystal size / mm	0.040 × 0.160 × 0.220	
Crystal system	Triclinic	
Space group	P 1	
Unit cell dimensions	*a* = 7.2817(10) Å	α = 77.657(2)°
	b = 10.2276(15) Å	β = 78.862(2)°
	c = 13.916(2) Å	γ = 72.059(2)°
Volume / $Å^3$	954.1(2)	
Z	2	
Density (calculated) / (g cm^{-3})	1.491	
Absorption coefficient / mm^{-1}	0.119	
F(000)	444	

Table S2. Data collection and structure refinement for 6_1

Theta range for data collection / °	2.12 to 28.00	
Index ranges	$-9 \le h \le 7, -13 \le k \le 13, -18 \le l \le 18$	
Reflections collected	8438	
Independent reflections	7331 [R(int) = 0.0130]	
Max. and min. transmission	0.7456 and 0.6907	
Structure solution technique	direct methods	
Structure solution program	XS (Sheldrick, 2008)	
Refinement method	full-matrix least-squares on F^2	
Refinement program	XL (Sheldrick, 2008)	
Function minimized	$\Sigma\, w(F_o^2 - F_c^2)^2$	
Data / restraints / parameters	7331 / 39 / 587	
Goodness-of-fit on F^2	1.034	
Final R indices	7084 data; $I > 2\sigma(I)$	R1 = 0.0305, wR2 = 0.0772
	all data	R1 = 0.0319, wR2 = 0.0783
Weighting scheme	$w = 1 / [\sigma^2(F_o^2) + (0.0434P)^2 + 0.2604P]$, where $P = (F_o^2 + 2F_c^2) / 3$	
Absolute structure parameter	0.2(2)	
Largest diff. peak and hole / eÅ^{-3}	0.321 and –0.214	
R.M.S. deviation from mean / eÅ^{-3}	0.039	

Table S3. Bond lengths for 6_1

Bond length / Å			
F1-C10	1.324(3)	F2-C10	1.333(3)
F3-C10	1.351(3)	O1-C9	1.227(3)
O2-C18	1.448(3)	O2-C19	1.436(3)
O3-C21	1.220(3)	N1-C1	1.411(3)
N1-C21	1.379(3)	N1-C22	1.488(2)
N2-C11	1.352(3)	N2-C12	1.487(3)
N2-C13	1.477(3)	C1-C2	1.397(3)

C1-C6	1.399(3)	C2-H2	0.95
C2-C3	1.384(4)	C3-H3	0.95
C3-C4	1.397(4)	C4-H4	0.95
C4-C5	1.394(4)	C5-H5	0.95
C5-C6	1.382(3)	C6-C7	1.513(3)
C7-C8	1.537(3)	C7-C12	1.560(3)
C7-C22	1.568(3)	C8-C9	1.427(3)
C8-C11	1.377(3)	C9-C10	1.553(3)
C11-H11	0.95	C12-H12	1.0
C12-C16	1.517(3)	C13-H13A	0.99
C13-H13B	0.99	C13-C14	1.520(3)
C14-C15	1.528(3)	C14-C17	1.326(3)
C15-H15	1.0	C15-C16	1.534(3)
C15-C23	1.542(3)	C16-H16A	0.99
C16-H16B	0.99	C17-H17	0.95
C17-C18	1.509(3)	C18-H18A	0.99
C18-H18B	0.99	C19-H19	1.0
C19-C20	1.552(3)	C19-C23	1.534(3)
C20-H20A	0.99	C20-H20B	0.99
C20-C21	1.523(3)	C22-H22	1.0
C22-C23	1.539(3)	C23-H23	1.0
F4-C33	1.276(4)	F4A-C33	1.390(12)
F5-C33	1.333(3)	F5A-C33	1.359(8)
F6-C33	1.329(4)	F6A-C33	1.221(9)
O4-C44	1.223(3)	O5-C41	1.435(3)
O5-C42	1.425(2)	O6-C32	1.230(3)
N3-C24	1.415(3)	N3-C44	1.368(3)
N3-C45	1.489(2)	N4-C34	1.340(3)
N4-C35	1.492(3)	N4-C36	1.477(3)
C24-C25	1.397(3)	C24-C29	1.398(3)
C25-H25	0.95	C25-C26	1.397(3)

C26-H26	0.95	C26-C27	1.387(3)
C27-H27	0.95	C27-C28	1.402(3)
C28-H28	0.95	C28-C29	1.386(3)
C29-C30	1.515(3)	C30-C31	1.550(3)
C30-C35	1.544(3)	C30-C45	1.572(3)
C31-C32	1.412(3)	C31-C34	1.380(3)
C32-C33	1.558(3)	C34-H34	0.95
C35-H35	1.0	C35-C39	1.515(3)
C36-H36A	0.99	C36-H36B	0.99
C36-C37	1.519(3)	C37-C38	1.533(3)
C37-C40	1.323(3)	C38-H38	1.0
C38-C39	1.537(3)	C38-C46	1.541(3)
C39-H39A	0.99	C39-H39B	0.99
C40-H40	0.95	C40-C41	1.508(3)
C41-H41A	0.99	C41-H41B	0.99
C42-H42	1.0	C42-C43	1.548(3)
C42-C46	1.539(3)	C43-H43A	0.99
C43-H43B	0.99	C43-C44	1.516(3)
C45-H45	1.0	C45-C46	1.531(3)
C46-H46	1.0		

Table S4. Bond angles for 6_1

	Bond angle / °		
C19-O2-C18	114.35(16)	C1-N1-C22	109.61(16)
C21-N1-C1	126.11(18)	C21-N1-C22	118.19(17)
C11-N2-C12	108.13(17)	C11-N2-C13	121.1(2)
C13-N2-C12	117.44(18)	C2-C1-N1	128.8(2)
C2-C1-C6	121.5(2)	C6-C1-N1	109.76(19)
C1-C2-H2	121.1	C3-C2-C1	117.8(2)
C3-C2-H2	121.1	C2-C3-H3	119.2

C2-C3-C4	121.5(2)	C4-C3-H3	119.2
C3-C4-H4	120.1	C5-C4-C3	119.9(2)
C5-C4-H4	120.1	C4-C5-H5	120.2
C6-C5-C4	119.6(2)	C6-C5-H5	120.2
C1-C6-C7	111.0(2)	C5-C6-C1	119.8(2)
C5-C6-C7	129.2(2)	C6-C7-C8	118.73(18)
C6-C7-C12	113.74(18)	C6-C7-C22	102.32(17)
C8-C7-C12	99.62(17)	C8-C7-C22	109.51(18)
C12-C7-C22	113.39(17)	C9-C8-C7	125.09(19)
C11-C8-C7	107.14(19)	C11-C8-C9	126.1(2)
O1-C9-C8	125.3(2)	O1-C9-C10	117.2(2)
C8-C9-C10	117.38(19)	F1-C10-F2	108.05(19)
F1-C10-F3	107.3(2)	F1-C10-C9	111.5(2)
F2-C10-F3	105.9(2)	F2-C10-C9	113.5(2)
F3-C10-C9	110.32(18)	N2-C11-C8	113.2(2)
N2-C11-H11	123.4	C8-C11-H11	123.4
N2-C12-C7	103.42(18)	N2-C12-H12	108.9
N2-C12-C16	110.38(18)	C7-C12-H12	108.9
C16-C12-C7	116.13(18)	C16-C12-H12	108.9
N2-C13-H13A	110.1	N2-C13-H13B	110.1
N2-C13-C14	108.15(16)	H13A-C13-H13B	108.4
C14-C13-H13A	110.1	C14-C13-H13B	110.1
C13-C14-C15	114.91(19)	C17-C14-C13	122.77(19)
C17-C14-C15	122.23(19)	C14-C15-H15	108.8
C14-C15-C16	110.35(18)	C14-C15-C23	114.60(17)
C16-C15-H15	108.8	C16-C15-C23	105.46(17)
C23-C15-H15	108.8	C12-C16-C15	108.38(18)
C12-C16-H16A	110.0	C12-C16-H16B	110.0
C15-C16-H16A	110.0	C15-C16-H16B	110.0
H16A-C16-H16B	108.4	C14-C17-H17	119.0
C14-C17-C18	122.0(2)	C18-C17-H17	119.0

O2-C18-C17	110.89(17)	O2-C18-H18A	109.5
O2-C18-H18B	109.5	C17-C18-H18A	109.5
C17-C18-H18B	109.5	H18A-C18-H18B	108.0
O2-C19-H19	109.3	O2-C19-C20	104.41(17)
O2-C19-C23	114.66(17)	C20-C19-H19	109.3
C23-C19-H19	109.3	C23-C19-C20	109.73(17)
C19-C20-H20A	108.4	C19-C20-H20B	108.4
H20A-C20-H20B	107.5	C21-C20-C19	115.54(18)
C21-C20-H20A	108.4	C21-C20-H20B	108.4
O3-C21-N1	123.1(2)	O3-C21-C20	123.2(2)
N1-C21-C20	113.69(17)	N1-C22-C7	104.76(16)
N1-C22-H22	109.8	N1-C22-C23	104.95(16)
C7-C22-H22	109.8	C23-C22-C7	117.28(17)
C23-C22-H22	109.8	C15-C23-H23	104.9
C19-C23-C15	118.85(17)	C19-C23-C22	107.40(16)
C19-C23-H23	104.9	C22-C23-C15	114.53(16)
C22-C23-H23	104.9	C42-O5-C41	114.63(18)
C24-N3-C45	109.54(16)	C44-N3-C24	125.93(16)
C44-N3-C45	119.20(16)	C34-N4-C35	108.82(17)
C34-N4-C36	122.36(19)	C36-N4-C35	117.40(17)
C25-C24-N3	128.19(19)	C25-C24-C29	121.49(19)
C29-C24-N3	110.26(17)	C24-C25-H25	121.2
C26-C25-C24	117.6(2)	C26-C25-H25	121.2
C25-C26-H26	119.3	C27-C26-C25	121.33(19)
C27-C26-H26	119.3	C26-C27-H27	119.8
C26-C27-C28	120.41(19)	C28-C27-H27	119.8
C27-C28-H28	120.5	C29-C28-C27	119.0(2)
C29-C28-H28	120.5	C24-C29-C30	110.45(17)
C28-C29-C24	120.12(19)	C28-C29-C30	129.42(19)
C29-C30-C31	117.70(17)	C29-C30-C35	115.17(17)
C29-C30-C45	102.35(15)	C31-C30-C45	109.05(16)

C35-C30-C31	99.78(15)	C35-C30-C45	113.14(16)
C32-C31-C30	123.22(18)	C34-C31-C30	106.47(19)
C34-C31-C32	128.2(2)	O6-C32-C31	126.01(19)
O6-C32-C33	115.9(2)	C31-C32-C33	118.07(18)
F4-C33-F5	109.9(3)	F4-C33-F6	108.2(3)
F4-C33-C32	113.3(3)	F4A-C33-C32	116.6(6)
F5-C33-C32	110.19(19)	F5A-C33-F4A	96.2(8)
F5A-C33-C32	110.1(3)	F6-C33-F5	105.0(3)
F6-C33-C32	109.9(2)	F6A-C33-F4A	103.6(10)
F6A-C33-F5A	111.1(10)	F6A-C33-C32	117.2(4)
N4-C34-C31	112.83(19)	N4-C34-H34	123.6
C31-C34-H34	123.6	N4-C35-C30	102.86(16)
N4-C35-H35	109.0	N4-C35-C39	109.94(17)
C30-C35-H35	109.0	C39-C35-C30	116.64(17)
C39-C35-H35	109.0	N4-C36-H36A	110.4
N4-C36-H36B	110.4	N4-C36-C37	106.51(17)
H36A-C36-H36B	108.6	C37-C36-H36A	110.4
C37-C36-H36B	110.4	C36-C37-C38	115.14(19)
C40-C37-C36	121.95(19)	C40-C37-C38	122.8(2)
C37-C38-H38	108.9	C37-C38-C39	109.51(17)
C37-C38-C46	114.88(17)	C39-C38-H38	108.9
C39-C38-C46	105.66(16)	C46-C38-H38	108.9
C35-C39-C38	108.28(17)	C35-C39-H39A	110.0
C35-C39-H39B	110.0	C38-C39-H39A	110.0
C38-C39-H39B	110.0	H39A-C39-H39B	108.4
C37-C40-H40	118.8	C37-C40-C41	122.5(2)
C41-C40-H40	118.8	O5-C41-C40	111.12(18)
O5-C41-H41A	109.4	O5-C41-H41B	109.4
C40-C41-H41A	109.4	C40-C41-H41B	109.4
H41A-C41-H41B	108.0	O5-C42-H42	109.2
O5-C42-C43	104.32(18)	O5-C42-C46	114.29(16)

C43-C42-H42	109.2	C46-C42-H42	109.2
C46-C42-C43	110.35(17)	C42-C43-H43A	107.8
C42-C43-H43B	107.8	H43A-C43-H43B	107.2
C44-C43-C42	117.87(18)	C44-C43-H43A	107.8
C44-C43-H43B	107.8	O4-C44-N3	122.66(19)
O4-C44-C43	122.21(19)	N3-C44-C43	115.11(17)
N3-C45-C30	104.54(15)	N3-C45-H45	109.6
N3-C45-C46	106.15(15)	C30-C45-H45	109.6
C46-C45-C30	117.12(17)	C46-C45-H45	109.6
C38-C46-H46	105.8	C42-C46-C38	118.32(17)
C42-C46-H46	105.8	C45-C46-C38	113.56(16)
C45-C46-C42	106.51(17)	C45-C46-H46	105.8

Crystal structure report for **10**

A specimen of $C_{24.50}H_{27}ClN_2O_4$, approximate dimensions of 0.030 × 0.070 × 0.300 mm, was used for the X-ray crystallographic analysis. The X-ray intensity data were measured.

The integration of the data using an orthorhombic unit cell yielded a total of 8738 reflections to a maximum θ angle of 21.95° (0.95 Å resolution), of which 4595 were independent (average redundancy 1.902, completeness = 96.4%, R_{int} = 4.05%, R_{sig} = 7.10%) and 3648 (79.39%) were greater than $2\sigma(F^2)$. The final cell constants of *a* = 7.188(4) Å, *b* = 22.411(12) Å, *c* = 26.507(15) Å, volume = 4270.(4) $Å^3$, are based upon the refinement of the XYZ-centroids of reflections above 20 σ(I). The calculated minimum and maximum transmission coefficients (based on crystal size) are 0.6411 and 0.7447.

The structure was solved and refined using the Bruker SHELXTL Software Package, using the space group P 21 21 21, with Z = 8 for the formula unit, $C_{24.50}H_{27}ClN_2O_4$. The final anisotropic full-matrix least-squares refinement on F^2 with 568 variables converged at R1 = 4.29%, for the observed data and wR2 = 9.64% for all data. The goodness-of-fit was 1.013. The largest peak in the final difference electron density synthesis was 0.201 $eÅ^{-3}$ and the largest hole was –0.286 $eÅ^{-3}$ with an RMS deviation of 0.049 $eÅ^{-3}$. On the basis of the final model, the calculated density was 1.397 g cm^{-3} and F(000), 1896 e^-.

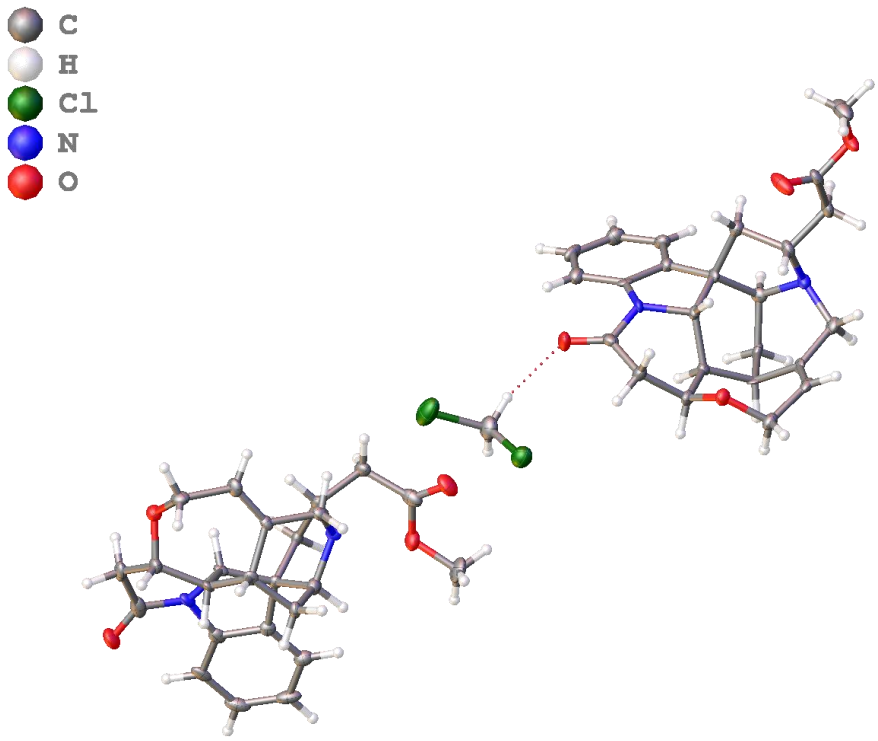

Table S5. Sample and crystal data for 10_1

Identification code	10_1	
Chemical formula	$C_{24.50}H_{27}ClN_2O_4$	
Formula weight / (g mol^{-1})	448.93	
Wavelength / Å	0.71073	
Crystal size / mm	0.030 × 0.070 × 0.300	
Crystal system	orthorhombic	
Space group	P 21 21 21	
Unit cell dimensions	*a* = 7.188(4) Å	α = 90°
	b = 22.411(12) Å	β = 90°
	c = 26.507(15) Å	γ = 90°
Volume / $Å^3$	4270.(4)	
Z	8	
Density (calculated) / (g cm^{-3})	1.397	

Absorption coefficient / mm^{-1}	0.215
F(000)	1896

Table S6. Data collection and structure refinement for 10_1

Theta range for data collection / °	1.19 to 21.95	
Index ranges	$-7 \le h \le 7, -19 \le k \le 23, -17 \le l \le 27$	
Reflections collected	8738	
Independent reflections	4595 [R(int) = 0.0405]	
Max. and min. transmission	0.7447 and 0.6411	
Structure solution technique	direct methods	
Structure solution program	XS (Sheldrick, 2008)	
Refinement method	full-matrix least-squares on F^2	
Refinement program	XL (Sheldrick, 2008)	
Function minimized	$\Sigma\, w(F_o^2 - F_c^2)^2$	
Data / restraints / parameters	4595 / 0 / 568	
Goodness-of-fit on F^2	1.013	
Final R indices	3648 data; $I > 2\sigma(I)$	R1 = 0.0429, wR2 = 0.0882
	all data	R1 = 0.0656, wR2 = 0.0964
Weighting scheme	$w = 1 / [\sigma^2(F_o^2) + (0.0493P)^2]$, where $P = (F_o^2 + 2F_c^2) / 3$	
Absolute structure parameter	–0.1(1)	
Largest diff. peak and hole / $eÅ^{-3}$	0.201 and –0.286	
R.M.S. deviation from mean / $eÅ^{-3}$	0.049	

Table S7. Bond lengths for 10_1

Bond length / Å			
Cl01-C49	1.780(7)	Cl02-C49	1.739(6)
C49-H49A	0.99	C49-H49B	0.99
O00C-C26	1.189(7)	O004-C37	1.441(7)
O004-C38	1.432(7)	O006-C25	1.462(8)

O006-C26	1.335(7)	O007-C42	1.227(7)
N00A-C40	1.494(7)	N00A-C42	1.376(7)
N00A-C43	1.421(8)	N00E-C28	1.474(8)
N00E-C31	1.499(7)	N00E-C35	1.479(8)
C25-H25A	0.98	C25-H25B	0.98
C25-H25C	0.98	C26-C27	1.511(9)
C27-H27A	0.99	C27-H27B	0.99
C27-C28	1.525(8)	C28-H28	1.0
C28-C29	1.541(8)	C29-H29A	0.99
C29-H29B	0.99	C29-C30	1.556(8)
C30-C31	1.559(8)	C30-C40	1.538(8)
C30-C48	1.523(8)	C31-H31	1.0
C31-C32	1.517(8)	C32-H32A	0.99
C32-H32B	0.99	C32-C33	1.535(8)
C33-H33	1.0	C33-C34	1.519(8)
C33-C39	1.526(8)	C34-C35	1.515(8)
C34-C36	1.331(9)	C35-H35A	0.99
C35-H35B	0.99	C36-H36	0.95
C36-C37	1.506(8)	C37-H37A	0.99
C37-H37B	0.99	C38-H38	1.0
C38-C39	1.540(8)	C38-C41	1.554(8)
C39-H39	1.0	C39-C40	1.535(8)
C40-H40	1.0	C41-H41A	0.99
C41-H41B	0.99	C41-C42	1.504(9)
C43-C44	1.402(8)	C43-C48	1.389(8)
C44-H44	0.95	C44-C45	1.375(9)
C45-H45	0.95	C45-C46	1.389(8)
C46-H46	0.95	C46-C47	1.379(8)
C47-H47	0.95	C47-C48	1.379(9)
O00D-C13	1.222(7)	O003-C15	1.433(7)
O003-C16	1.446(7)	O005-C23	1.353(8)

O005-C24	1.453(7)	O009-C23	1.202(7)
N00B-C6	1.422(8)	N00B-C12	1.498(7)
N00B-C13	1.377(7)	N008-C8	1.495(7)
N008-C19	1.470(8)	N008-C20	1.477(8)
C1-H1	0.95	C1-C2	1.386(9)
C1-C6	1.412(9)	C2-H2	0.95
C2-C3	1.391(9)	C3-H3	0.95
C3-C4	1.400(9)	C4-H4	0.95
C4-C5	1.382(9)	C5-C6	1.379(8)
C5-C7	1.506(9)	C7-C8	1.558(8)
C7-C12	1.562(8)	C7-C21	1.538(8)
C8-H8	1.0	C8-C9	1.515(8)
C9-H9A	0.99	C9-H9B	0.99
C9-C10	1.531(8)	C10-H10	1.0
C10-C11	1.532(8)	C10-C18	1.535(8)
C11-H11	1.0	C11-C12	1.529(9)
C11-C15	1.532(8)	C12-H12	1.0
C13-C14	1.517(10)	C14-H14A	0.99
C14-H14B	0.99	C14-C15	1.548(9)
C15-H15	1.0	C16-H16A	0.99
C16-H16B	0.99	C16-C17	1.503(8)
C17-H17	0.95	C17-C18	1.322(9)
C18-C19	1.509(7)	C19-H19A	0.99
C19-H19B	0.99	C20-H20	1.0
C20-C21	1.524(9)	C20-C22	1.544(7)
C21-H21A	0.99	C21-H21B	0.99
C22-H22A	0.99	C22-H22B	0.99
C22-C23	1.519(9)	C24-H24A	0.98
C24-H24B	0.98	C24-H24C	0.98

Table S8. Bond angles for 10_1

Bond angle / °			
Cl01-C49-H49A	109.2	Cl01-C49-H49B	109.2
Cl02-C49-Cl01	112.2(4)	Cl02-C49-H49A	109.2
Cl02-C49-H49B	109.2	H49A-C49-H49B	107.9
C38-O004-C37	114.5(5)	C26-O006-C25	114.5(5)
C42-N00A-C40	117.7(5)	C42-N00A-C43	125.5(5)
C43-N00A-C40	108.8(5)	C28-N00E-C31	109.3(4)
C28-N00E-C35	112.2(5)	C35-N00E-C31	113.4(5)
O006-C25-H25A	109.5	O006-C25-H25B	109.5
O006-C25-H25C	109.5	H25A-C25-H25B	109.5
H25A-C25-H25C	109.5	H25B-C25-H25C	109.5
O00C-C26-O006	123.0(6)	O00C-C26-C27	125.5(5)
O006-C26-C27	111.5(5)	C26-C27-H27A	108.8
C26-C27-H27B	108.8	C26-C27-C28	113.9(5)
H27A-C27-H27B	107.7	C28-C27-H27A	108.8
C28-C27-H27B	108.8	N00E-C28-C27	110.8(4)
N00E-C28-H28	109.2	N00E-C28-C29	103.7(5)
C27-C28-H28	109.2	C27-C28-C29	114.5(5)
C29-C28-H28	109.2	C28-C29-H29A	111.6
C28-C29-H29B	111.6	C28-C29-C30	100.9(5)
H29A-C29-H29B	109.4	C30-C29-H29A	111.6
C30-C29-H29B	111.6	C29-C30-C31	100.2(4)
C40-C30-C29	110.7(5)	C40-C30-C31	114.9(5)
C48-C30-C29	113.5(5)	C48-C30-C31	114.3(5)
C48-C30-C40	103.7(4)	N00E-C31-C30	104.5(5)
N00E-C31-H31	109.0	N00E-C31-C32	111.7(5)
C30-C31-H31	109.0	C32-C31-C30	113.6(4)
C32-C31-H31	109.0	C31-C32-H32A	109.7
C31-C32-H32B	109.7	C31-C32-C33	109.6(5)
H32A-C32-H32B	108.2	C33-C32-H32A	109.7

C33-C32-H32B	109.7	C32-C33-H33	109.1
C34-C33-C32	108.7(5)	C34-C33-H33	109.1
C34-C33-C39	114.1(5)	C39-C33-C32	106.6(5)
C39-C33-H33	109.1	C35-C34-C33	114.9(6)
C36-C34-C33	122.1(5)	C36-C34-C35	122.9(6)
N00E-C35-C34	111.8(5)	N00E-C35-H35A	109.2
N00E-C35-H35B	109.2	C34-C35-H35A	109.2
C34-C35-H35B	109.2	H35A-C35-H35B	107.9
C34-C36-H36	119.3	C34-C36-C37	121.4(6)
C37-C36-H36	119.3	O004-C37-C36	111.1(5)
O004-C37-H37A	109.4	O004-C37-H37B	109.4
C36-C37-H37A	109.4	C36-C37-H37B	109.4
H37A-C37-H37B	108.0	O004-C38-H38	109.1
O004-C38-C39	115.5(4)	O004-C38-C41	103.7(5)
C39-C38-H38	109.1	C39-C38-C41	110.0(5)
C41-C38-H38	109.1	C33-C39-C38	118.7(5)
C33-C39-H39	104.9	C33-C39-C40	114.3(4)
C38-C39-H39	104.9	C40-C39-C38	107.8(5)
C40-C39-H39	104.9	N00A-C40-C30	104.1(5)
N00A-C40-C39	106.0(4)	N00A-C40-H40	109.9
C30-C40-H40	109.9	C39-C40-C30	116.5(5)
C39-C40-H40	109.9	C38-C41-H41A	108.4
C38-C41-H41B	108.4	H41A-C41-H41B	107.4
C42-C41-C38	115.7(5)	C42-C41-H41A	108.4
C42-C41-H41B	108.4	O007-C42-N00A	121.4(6)
O007-C42-C41	123.9(5)	N00A-C42-C41	114.7(5)
C44-C43-N00A	129.3(5)	C48-C43-N00A	110.6(5)
C48-C43-C44	120.1(6)	C43-C44-H44	120.7
C45-C44-C43	118.6(6)	C45-C44-H44	120.7
C44-C45-H45	119.6	C44-C45-C46	120.8(6)
C46-C45-H45	119.6	C45-C46-H46	119.6

C47-C46-C45	120.9(6)	C47-C46-H46	119.6
C46-C47-H47	120.6	C48-C47-C46	118.8(6)
C48-C47-H47	120.6	C43-C48-C30	109.2(5)
C47-C48-C30	129.9(5)	C47-C48-C43	120.9(6)
C15-O003-C16	114.9(5)	C23-O005-C24	116.1(5)
C6-N00B-C12	109.0(4)	C13-N00B-C6	124.8(5)
C13-N00B-C12	118.4(5)	C19-N008-C8	114.0(4)
C19-N008-C20	114.2(5)	C20-N008-C8	108.6(5)
C2-C1-H1	120.9	C2-C1-C6	118.2(6)
C6-C1-H1	120.9	C1-C2-H2	119.5
C1-C2-C3	120.9(6)	C3-C2-H2	119.5
C2-C3-H3	120.0	C2-C3-C4	120.1(7)
C4-C3-H3	120.0	C3-C4-H4	120.3
C5-C4-C3	119.4(6)	C5-C4-H4	120.3
C4-C5-C7	128.4(5)	C6-C5-C4	120.4(6)
C6-C5-C7	110.9(6)	C1-C6-N00B	128.6(5)
C5-C6-N00B	110.4(6)	C5-C6-C1	121.0(6)
C5-C7-C8	116.5(5)	C5-C7-C12	102.7(4)
C5-C7-C21	112.1(5)	C8-C7-C12	113.0(5)
C21-C7-C8	101.0(4)	C21-C7-C12	111.7(5)
N008-C8-C7	105.8(5)	N008-C8-H8	107.8
N008-C8-C9	111.6(5)	C7-C8-H8	107.8
C9-C8-C7	115.6(4)	C9-C8-H8	107.8
C8-C9-H9A	110.2	C8-C9-H9B	110.2
C8-C9-C10	107.6(5)	H9A-C9-H9B	108.5
C10-C9-H9A	110.2	C10-C9-H9B	110.2
C9-C10-H10	108.5	C9-C10-C11	106.5(5)
C9-C10-C18	110.9(4)	C11-C10-H10	108.5
C11-C10-C18	113.8(5)	C18-C10-H10	108.5
C10-C11-H11	104.9	C10-C11-C15	118.7(6)
C12-C11-C10	113.6(4)	C12-C11-H11	104.9

C12-C11-C15	108.4(5)	C15-C11-H11	104.9
N00B-C12-C7	104.2(5)	N00B-C12-C11	106.5(4)
N00B-C12-H12	109.6	C7-C12-H12	109.6
C11-C12-C7	117.1(5)	C11-C12-H12	109.6
O00D-C13-N00B	122.2(7)	O00D-C13-C14	123.7(5)
N00B-C13-C14	114.0(6)	C13-C14-H14A	108.3
C13-C14-H14B	108.3	C13-C14-C15	116.0(5)
H14A-C14-H14B	107.4	C15-C14-H14A	108.3
C15-C14-H14B	108.3	O003-C15-C11	114.8(4)
O003-C15-C14	103.9(5)	O003-C15-H15	109.4
C11-C15-C14	109.8(6)	C11-C15-H15	109.4
C14-C15-H15	109.4	O003-C16-H16A	109.4
O003-C16-H16B	109.4	O003-C16-C17	111.2(5)
H16A-C16-H16B	108.0	C17-C16-H16A	109.4
C17-C16-H16B	109.4	C16-C17-H17	118.7
C18-C17-C16	122.7(6)	C18-C17-H17	118.7
C17-C18-C10	122.3(5)	C17-C18-C19	124.8(6)
C19-C18-C10	112.9(5)	N008-C19-C18	112.8(5)
N008-C19-H19A	109.0	N008-C19-H19B	109.0
C18-C19-H19A	109.0	C18-C19-H19B	109.0
H19A-C19-H19B	107.8	N008-C20-H20	108.6
N008-C20-C21	106.1(5)	N008-C20-C22	109.4(5)
C21-C20-H20	108.6	C21-C20-C22	115.6(5)
C22-C20-H20	108.6	C7-C21-H21A	110.9
C7-C21-H21B	110.9	C20-C21-C7	104.3(5)
C20-C21-H21A	110.9	C20-C21-H21B	110.9
H21A-C21-H21B	108.9	C20-C22-H22A	109.4
C20-C22-H22B	109.4	H22A-C22-H22B	108.0
C23-C22-C20	111.3(5)	C23-C22-H22A	109.4
C23-C22-H22B	109.4	O005-C23-C22	112.3(6)
O009-C23-O005	122.1(7)	O009-C23-C22	125.7(7)

O005-C24-H24A	109.5	O005-C24-H24B	109.5
O005-C24-H24C	109.5	H24A-C24-H24B	109.5
H24A-C24-H24C	109.5	H24B-C24-H24C	109.5

checkCIF/PLATON report

Structure factors have been supplied for datablock(s) 10, 6

THIS REPORT IS FOR GUIDANCE ONLY. IF USED AS PART OF A REVIEW PROCEDURE FOR PUBLICATION, IT SHOULD NOT REPLACE THE EXPERTISE OF AN EXPERIENCED CRYSTALLOGRAPHIC REFEREE.

No syntax errors found. CIF dictionary Interpreting this report

Datablock: 6

```
Bond precision:  C-C = 0.0031 A          Wavelength=0.71073

Cell:          a=7.2817(10)      b=10.2276(15)     c=13.916(2)
               alpha=77.657(2)   beta=78.862(2)    gamma=72.059(2)
Temperature:   100 K

                Calculated                Reported
Volume          954.1(2)                  954.1(2)
Space group     P 1                       P 1
Hall group      P 1                       P 1
Moiety formula  C23 H19 F3 N2 O3          C23 H19 F3 N2 O3
Sum formula     C23 H19 F3 N2 O3          C23 H19 F3 N2 O3
Mr              428.40                    428.40
Dx,g cm-3       1.491                     1.491
Z               2                         2
Mu (mm-1)       0.119                     0.119
F000            444.0                     444.0
F000'           444.27
h,k,lmax        9,13,18                   9,13,18
Nref            9214[ 4607]               7331
Tmin,Tmax       0.977,0.995               0.691,0.746
Tmin'           0.974

Correction method= # Reported T Limits: Tmin=0.691 Tmax=0.746
AbsCorr = MULTI-SCAN

Data completeness= 1.59/0.80      Theta(max)= 28.003

R(reflections)= 0.0305( 7084)     wR2(reflections)= 0.0783( 7331)

S = 1.034                 Npar= 587
```

The following ALERTS were generated. Each ALERT has the format
test-name_ALERT_alert-type_alert-level.
Click on the hyperlinks for more details of the test.

Alert level B

PLAT213_ALERT_2_B	Atom F6A	has ADP max/min Ratio	4.6 prolat

Alert level C

PLAT213_ALERT_2_C	Atom F4	has ADP max/min Ratio	3.5 prolat
PLAT213_ALERT_2_C	Atom F4A	has ADP max/min Ratio	3.5 prolat
PLAT213_ALERT_2_C	Atom F5A	has ADP max/min Ratio	3.3 prolat
PLAT242_ALERT_2_C	Low 'MainMol' Ueq as Compared to Neighbors of		C33 Check
PLAT250_ALERT_2_C	Large U3/U1 Ratio for Average U(i,j) Tensor		2.1 Note
PLAT911_ALERT_3_C	Missing FCF Refl Between Thmin & STh/L=	0.600	4 Report
PLAT915_ALERT_3_C	No Flack x Check Done: Low Friedel Pair Coverage		60 %

Alert level G

PLAT003_ALERT_2_G	Number of Uiso or Uij Restrained non-H Atoms ...		6 Report
PLAT154_ALERT_1_G	The s.u.'s on the Cell Angles are Equal ..(Note)		0.002 Degree
PLAT177_ALERT_4_G	The CIF-Embedded .res File Contains DELU Records		1 Report
PLAT178_ALERT_4_G	The CIF-Embedded .res File Contains SIMU Records		1 Report
PLAT242_ALERT_2_G	Low 'MainMol' Ueq as Compared to Neighbors of		C10 Check
PLAT301_ALERT_3_G	Main Residue Disorder .....(Resd 1)		10% Note
PLAT791_ALERT_4_G	Model has Chirality at C7	(Chiral SPGR)	S Verify
PLAT791_ALERT_4_G	Model has Chirality at C12	(Chiral SPGR)	S Verify
PLAT791_ALERT_4_G	Model has Chirality at C15	(Chiral SPGR)	R Verify
PLAT791_ALERT_4_G	Model has Chirality at C19	(Chiral SPGR)	S Verify
PLAT791_ALERT_4_G	Model has Chirality at C22	(Chiral SPGR)	S Verify
PLAT791_ALERT_4_G	Model has Chirality at C23	(Chiral SPGR)	R Verify
PLAT791_ALERT_4_G	Model has Chirality at C30	(Chiral SPGR)	S Verify
PLAT791_ALERT_4_G	Model has Chirality at C35	(Chiral SPGR)	S Verify
PLAT791_ALERT_4_G	Model has Chirality at C38	(Chiral SPGR)	R Verify
PLAT791_ALERT_4_G	Model has Chirality at C42	(Chiral SPGR)	S Verify
PLAT791_ALERT_4_G	Model has Chirality at C45	(Chiral SPGR)	S Verify
PLAT791_ALERT_4_G	Model has Chirality at C46	(Chiral SPGR)	R Verify
PLAT860_ALERT_3_G	Number of Least-Squares Restraints		39 Note
PLAT910_ALERT_3_G	Missing # of FCF Reflection(s) Below Theta(Min).		1 Note
PLAT912_ALERT_4_G	Missing # of FCF Reflections Above STh/L=	0.600	52 Note
PLAT913_ALERT_3_G	Missing # of Very Strong Reflections in FCF		1 Note
PLAT978_ALERT_2_G	Number C-C Bonds with Positive Residual Density.		18 Info

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23 **ALERT level G** = General information/check it is not something unexpected

1 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
9 ALERT type 2 Indicator that the structure model may be wrong or deficient
6 ALERT type 3 Indicator that the structure quality may be low
15 ALERT type 4 Improvement, methodology, query or suggestion
0 ALERT type 5 Informative message, check

Datablock: 10

Bond precision: C-C = 0.0082 A Wavelength=0.71073

Cell:	a=7.188(4)	b=22.411(12)	c=26.507(15)
	alpha=90	beta=90	gamma=90
Temperature:	100 K		

	Calculated	Reported
Volume	4270(4)	4270(4)
Space group	P 21 21 21	P 21 21 21
Hall group	P 2ac 2ab	P 2ac 2ab
Moiety formula	2(C24 H26 N2 O4), C H2 C12	0.5(C H2 C12), C24 H26 N2 O4
Sum formula	C49 H54 C12 N4 O8	C24.50 H27 C1 N2 O4
Mr	897.86	448.93
Dx,g cm-3	1.397	1.397
Z	4	8
Mu (mm-1)	0.215	0.215
F000	1896.0	1896.0
F000'	1898.00	
h,k,lmax	7,23,27	7,23,27
Nref	5206[2998]	4595
Tmin,Tmax	0.982,0.994	0.641,0.745
Tmin'	0.938	

Correction method= # Reported T Limits: Tmin=0.641 Tmax=0.745
AbsCorr = MULTI-SCAN

Data completeness= 1.53/0.88 Theta(max)= 21.948

R(reflections)= 0.0429(3648) wR2(reflections)= 0.0964(4595)

S = 1.013 Npar= 568

The following ALERTS were generated. Each ALERT has the format
test-name_ALERT_alert-type_alert-level.
Click on the hyperlinks for more details of the test.

Alert level A

THETM01_ALERT_3_A The value of sine(theta_max)/wavelength is less than 0.550
Calculated sin(theta_max)/wavelength = 0.5259

Author Response: These were small, needle-shaped crystals, and due to small size, high resolution data could not be obtained. The resolution is sufficient for structu identification and bond measurement to three significant figures.

Alert level B

PLAT089_ALERT_3_B Poor Data / Parameter Ratio (Zmax < 18) ..... 5.09 Note

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Alert level C

PLAT029_ALERT_3_C	_diffrn_measured_fraction_theta_full value Low .	0.964	Why?
PLAT340_ALERT_3_C	Low Bond Precision on C-C Bonds	0.00823	Ang.
PLAT911_ALERT_3_C	Missing FCF Refl Between Thmin & STh/L= 0.526	109	Report
PLAT915_ALERT_3_C	No Flack x Check Done: Low Friedel Pair Coverage	77	%
PLAT978_ALERT_2_C	Number C-C Bonds with Positive Residual Density.	0	Info

Alert level G

PLAT042_ALERT_1_G	Calc. and Reported MoietyFormula Strings Differ	Please	Check
PLAT045_ALERT_1_G	Calculated and Reported Z Differ by a Factor ...	0.50	Check
PLAT720_ALERT_4_G	Number of Unusual/Non-Standard Labels	14	Note
PLAT791_ALERT_4_G	Model has Chirality at C7 (Chiral SPGR)	R	Verify
PLAT791_ALERT_4_G	Model has Chirality at C8 (Chiral SPGR)	S	Verify
PLAT791_ALERT_4_G	Model has Chirality at C10 (Chiral SPGR)	R	Verify
PLAT791_ALERT_4_G	Model has Chirality at C11 (Chiral SPGR)	R	Verify
PLAT791_ALERT_4_G	Model has Chirality at C12 (Chiral SPGR)	S	Verify
PLAT791_ALERT_4_G	Model has Chirality at C15 (Chiral SPGR)	S	Verify
PLAT791_ALERT_4_G	Model has Chirality at C20 (Chiral SPGR)	R	Verify
PLAT791_ALERT_4_G	Model has Chirality at C28 (Chiral SPGR)	R	Verify
PLAT791_ALERT_4_G	Model has Chirality at C31 (Chiral SPGR)	S	Verify
PLAT791_ALERT_4_G	Model has Chirality at C33 (Chiral SPGR)	R	Verify
PLAT791_ALERT_4_G	Model has Chirality at C38 (Chiral SPGR)	S	Verify
PLAT791_ALERT_4_G	Model has Chirality at C39 (Chiral SPGR)	R	Verify
PLAT791_ALERT_4_G	Model has Chirality at C40 (Chiral SPGR)	S	Verify
PLAT802_ALERT_4_G	CIF Input Record(s) with more than 80 Characters	1	Info
PLAT909_ALERT_3_G	Percentage of I>2sig(I) Data at Theta(Max) Still	56%	Note

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It is advisable to attempt to resolve as many as possible of the alerts in all categories. Often the minor alerts point to easily fixed oversights, errors and omissions in your CIF or refinement strategy, so attention to these fine details can be worthwhile. In order to resolve some of the more serious problems it may be necessary to carry out additional measurements or structure refinements. However, the purpose of your study may justify the reported deviations and the more serious of these should normally be commented upon in the discussion or experimental section of a paper or in the "special_details" fields of the CIF. checkCIF was carefully designed to identify outliers and unusual parameters, but every test has its limitations and alerts that are not important in a particular case may appear. Conversely, the absence of alerts does not guarantee there are no aspects of the results needing attention. It is up to the individual to critically assess their own results and, if necessary, seek expert advice.

Publication of your CIF in IUCr journals

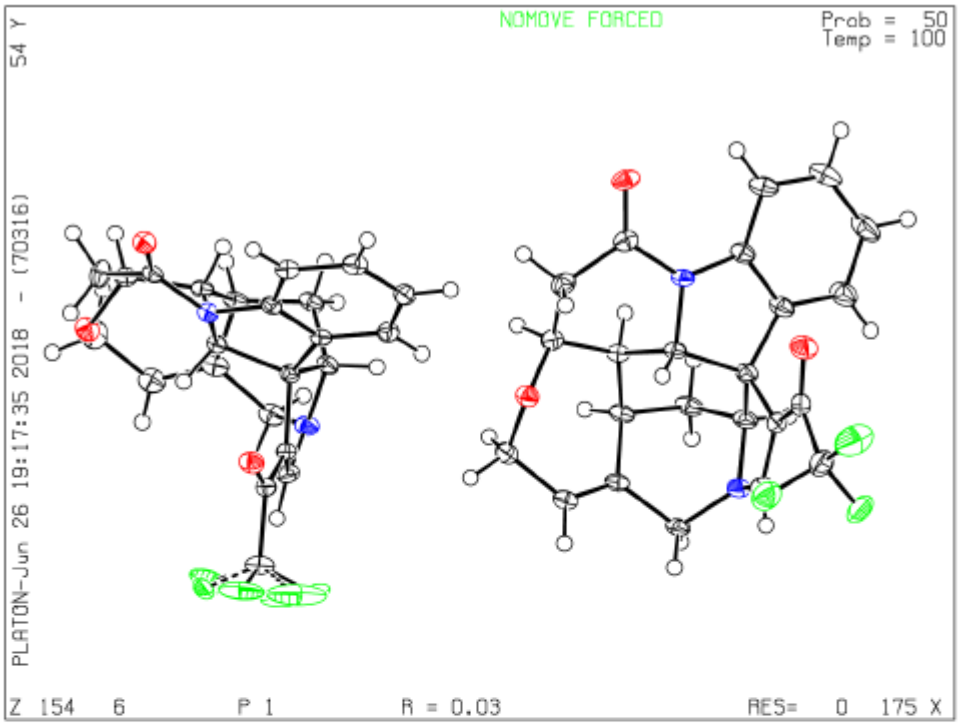
A basic structural check has been run on your CIF. These basic checks will be run on all CIFs submitted for publication in IUCr journals (*Acta Crystallographica*, *Journal of Applied Crystallography*, *Journal of Synchrotron Radiation*); however, if you intend to submit to *Acta Crystallographica Section C* or *E* or *IUCrData*, you should make sure that full publication checks are run on the final version of your CIF prior to submission.

Publication of your CIF in other journals

Please refer to the *Notes for Authors* of the relevant journal for any special instructions relating to CIF submission.

PLATON version of 23/04/2018; check.def file version of 23/04/2018

Datablock 6 - ellipsoid plot



Datablock 10 - ellipsoid plot