Synthesis

Reviews and Full Papers in Chemical Synthesis

Supporting Information

for DOI: 10.1055/s-0042-1752404



Georg Thieme Verlag KG, Rüdigerstraße 14, 70469 Stuttgart, Germany

Thieme

*Supplementary data for:*

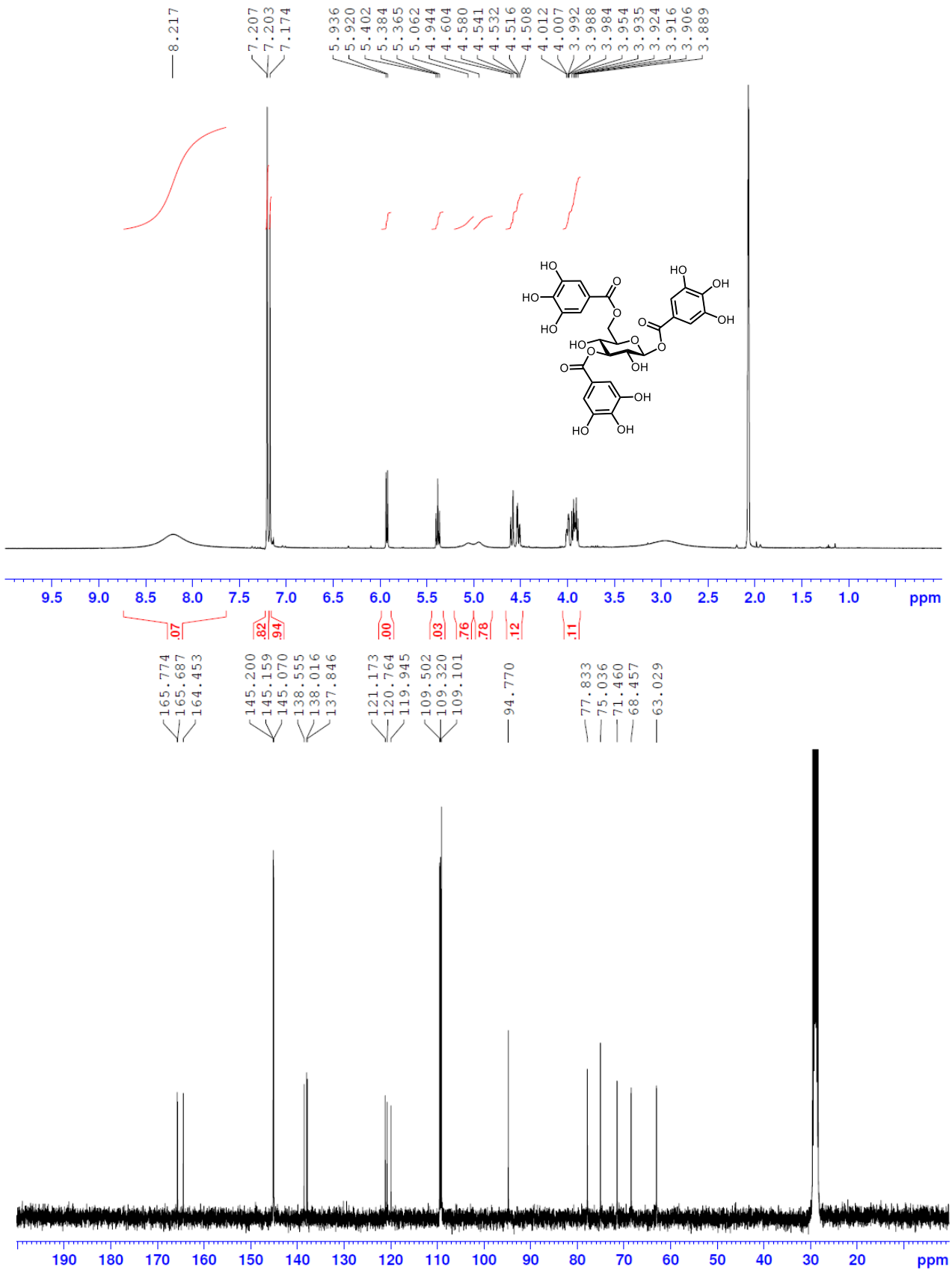
# Improved Total Synthesis of 1,3,6-trigalloyl-β-D-glucose from Glucose

**Yann Pauvert [a], Roger Gaudreault [b] and André B. Charette[a*]**

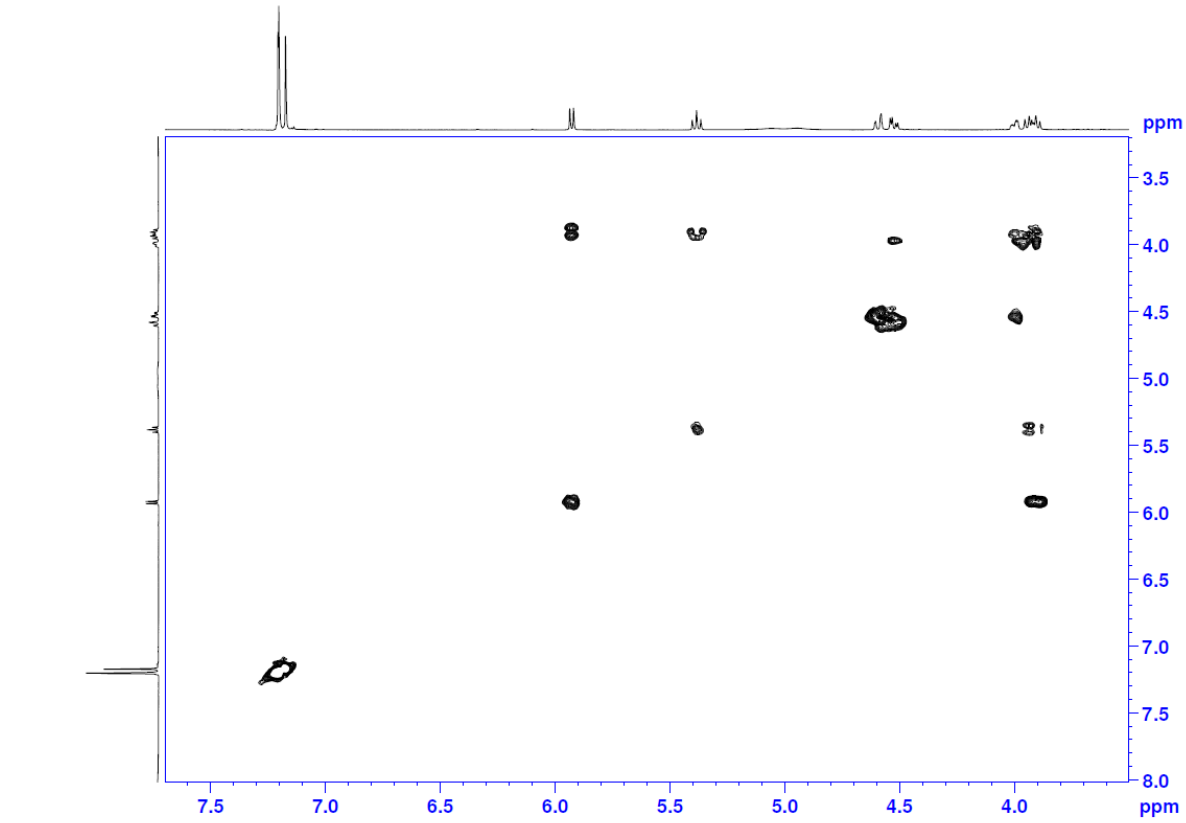
[a] FRQNT-Center in Green Chemistry and Catalysis, Department of Chemistry, Université de Montréal, 1375 av. Thérèse Lavoie-Roux, Montréal, Québec Canada H2V 0B3

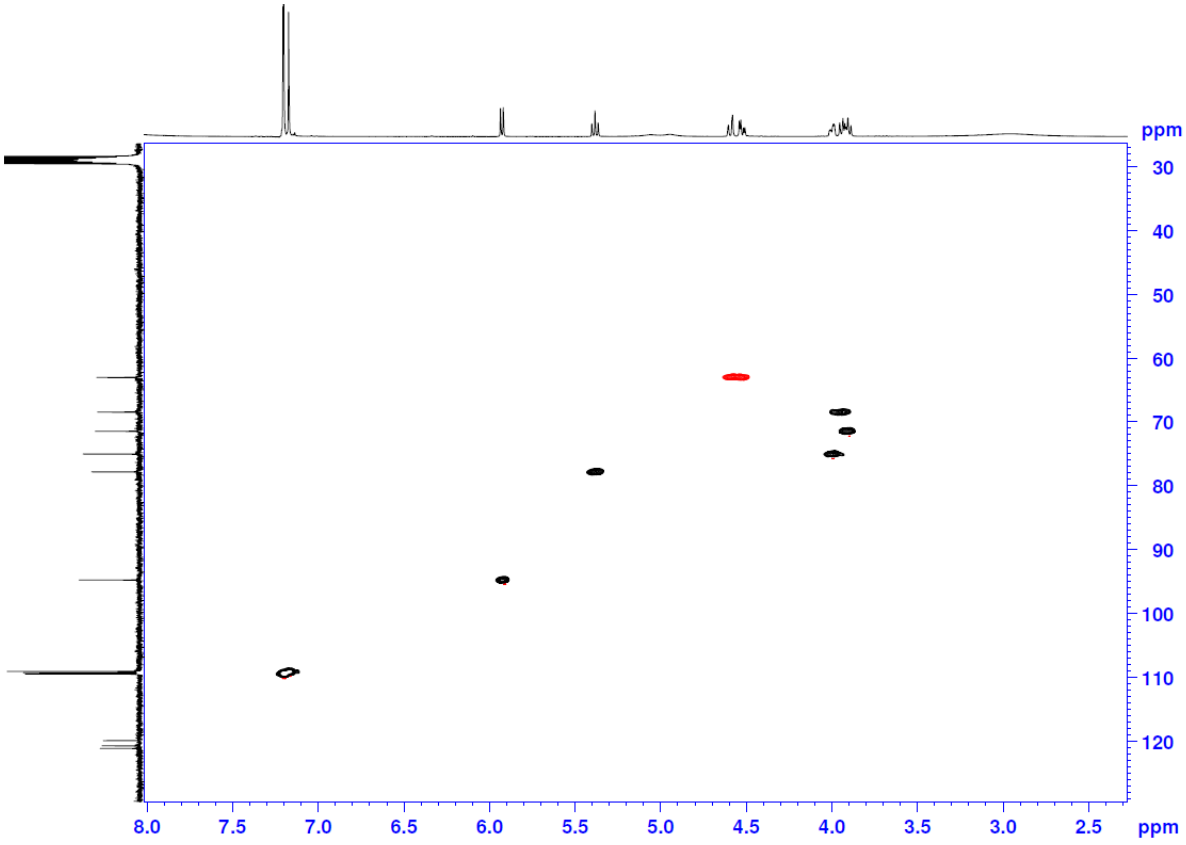
[b] Department of Physics, Université de Montréal, 1375 av. Thérèse Lavoie-Roux, Montréal, Québec Canada H2V 0B3

$^{1}H$ and $^{13}C$ NMR spectra of compound **1** in acetone-$d_6$

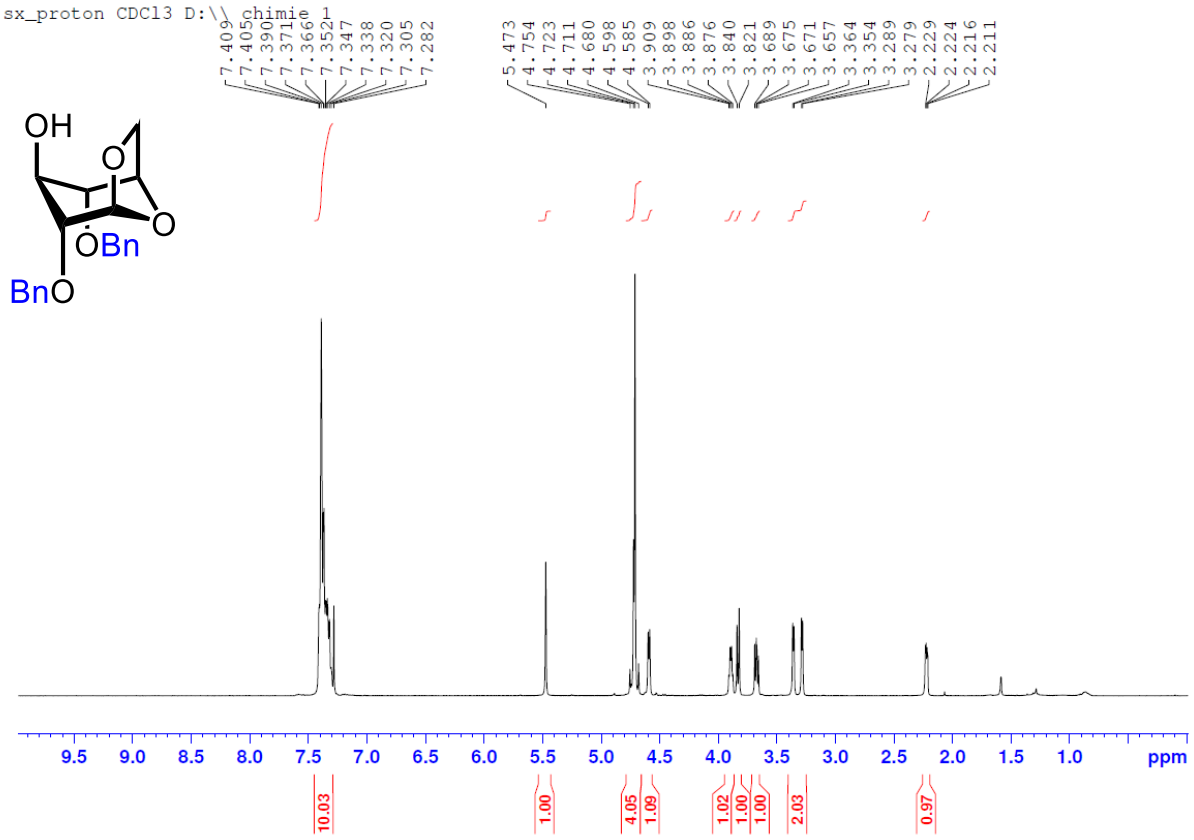


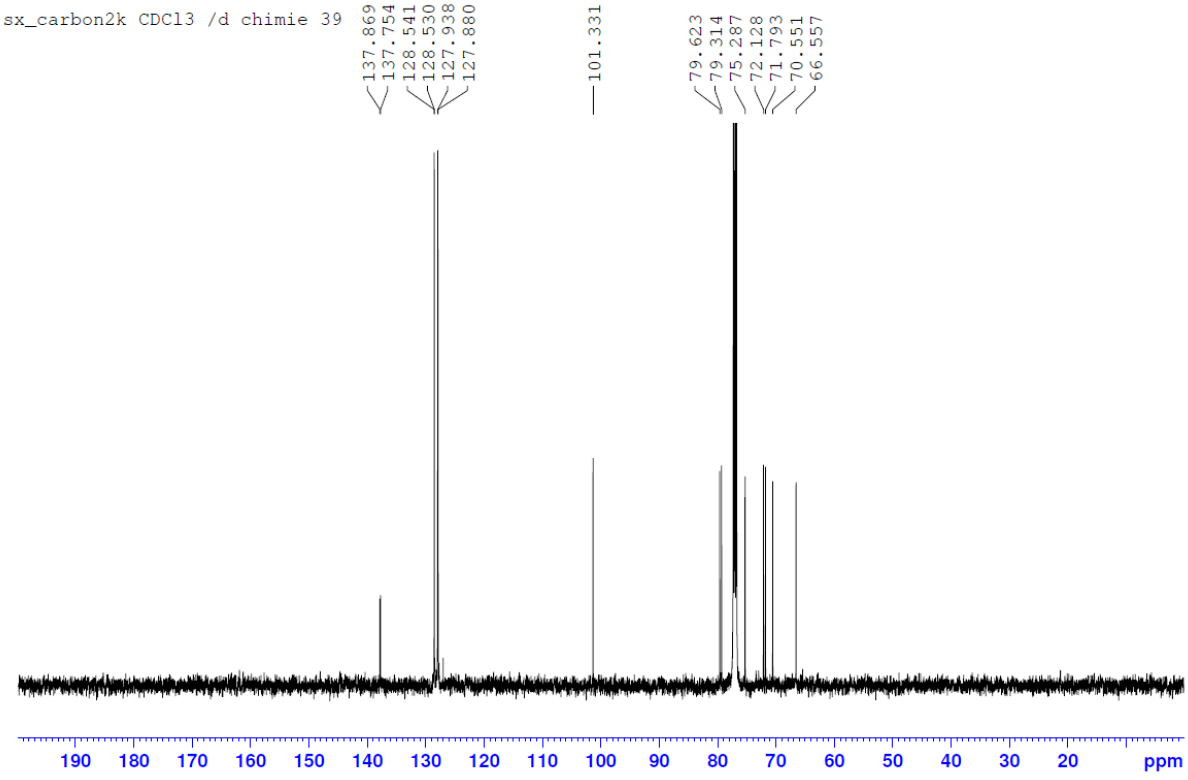
COSY and HSQC NMR spectra of compound **1** in acetone-$d_6$



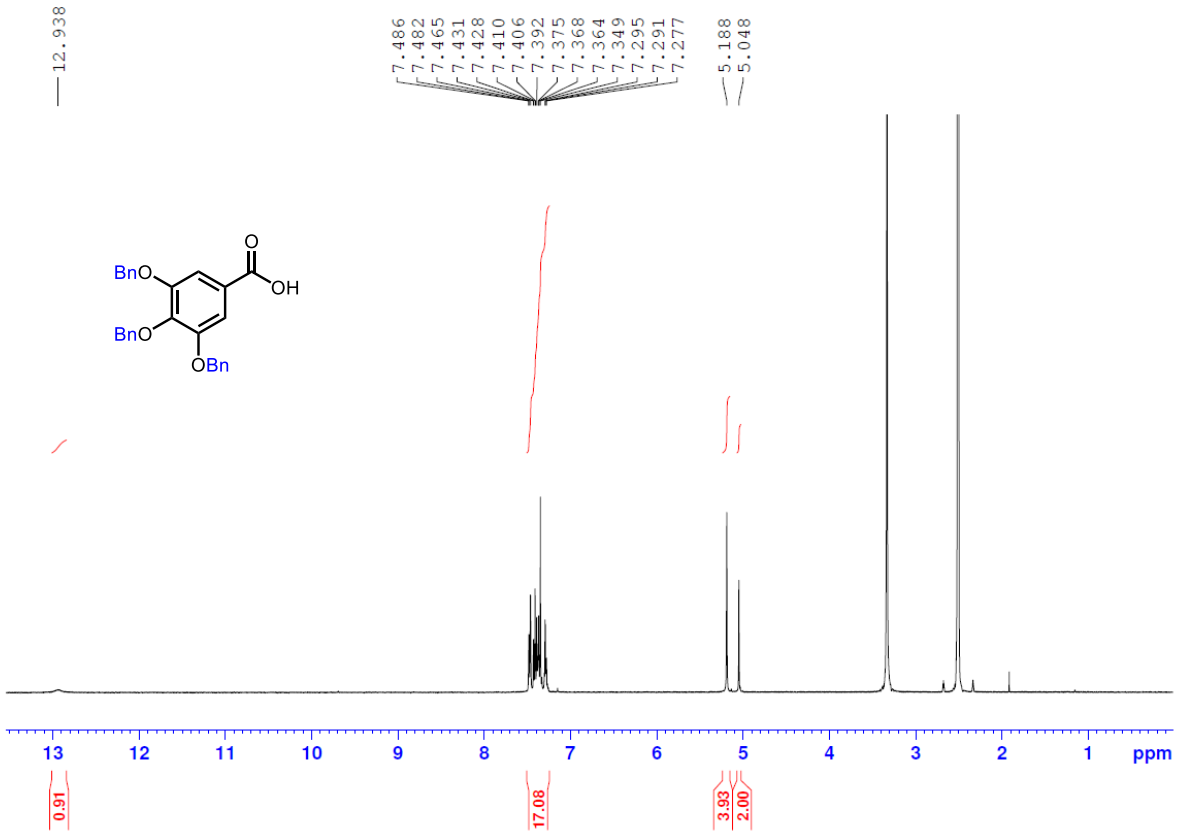



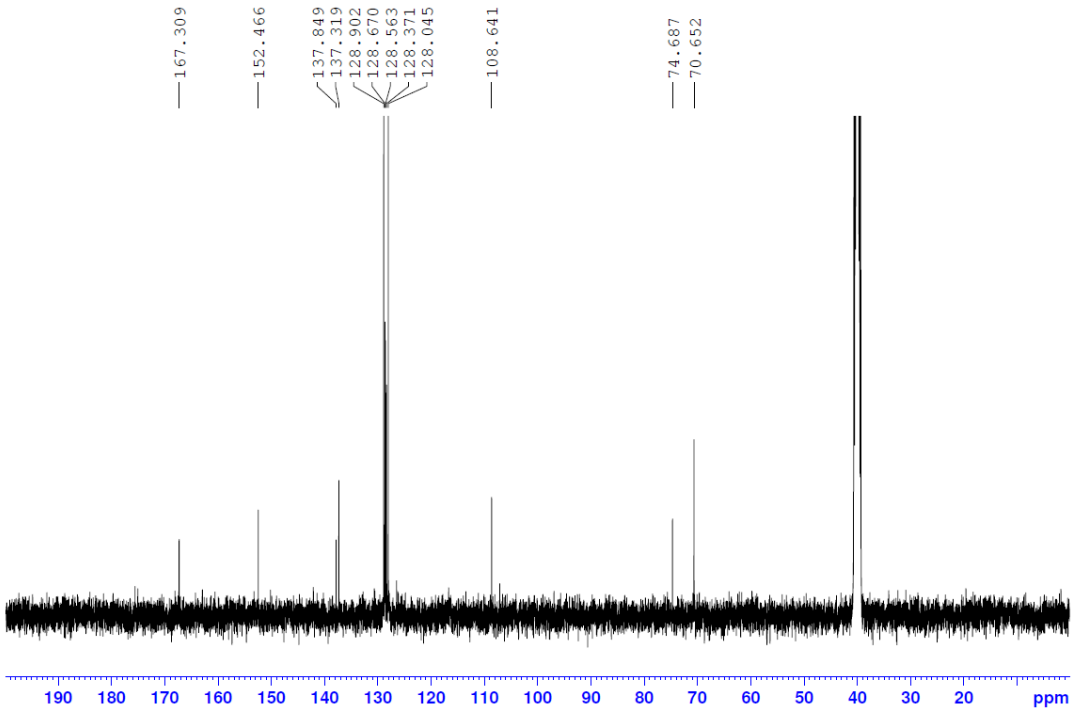
$^{1}H$ and $^{13}C$ NMR spectra of compound **6** in $CDCl_3$



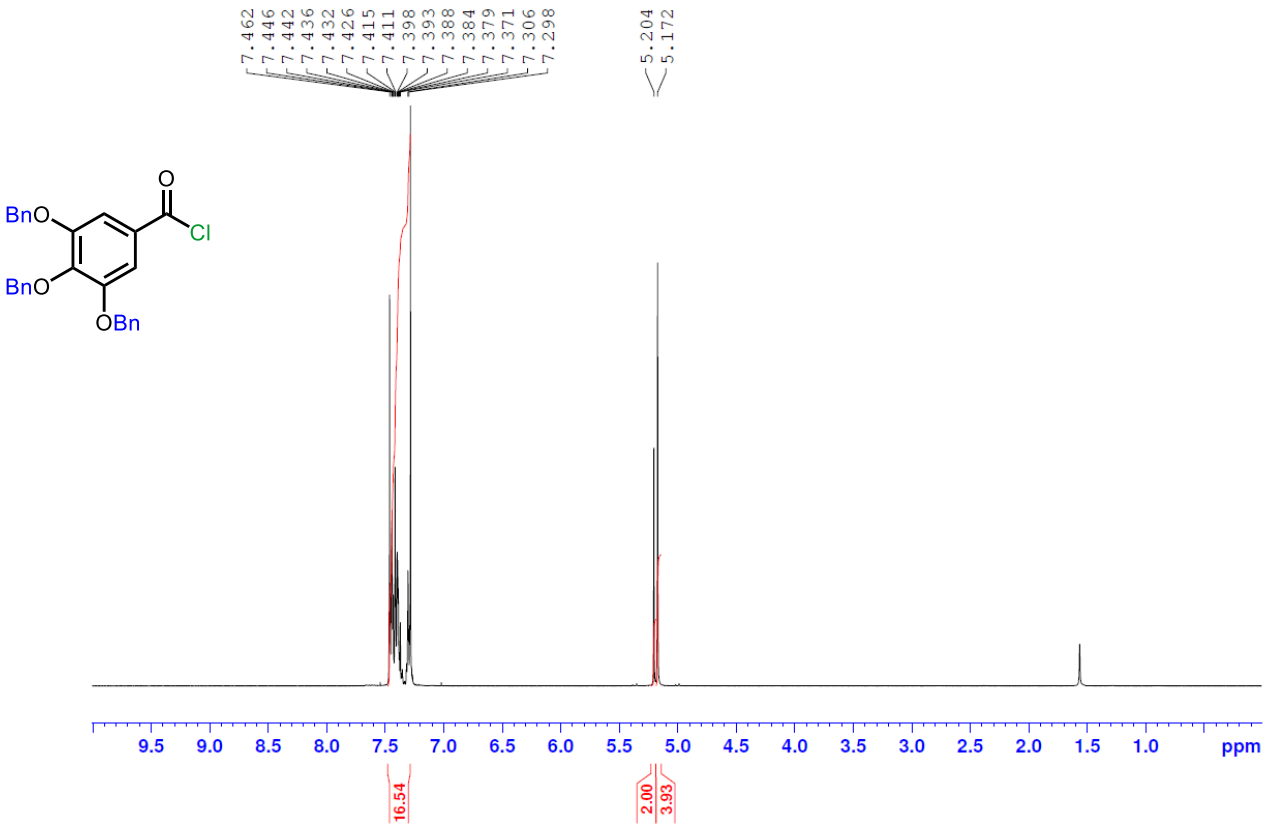



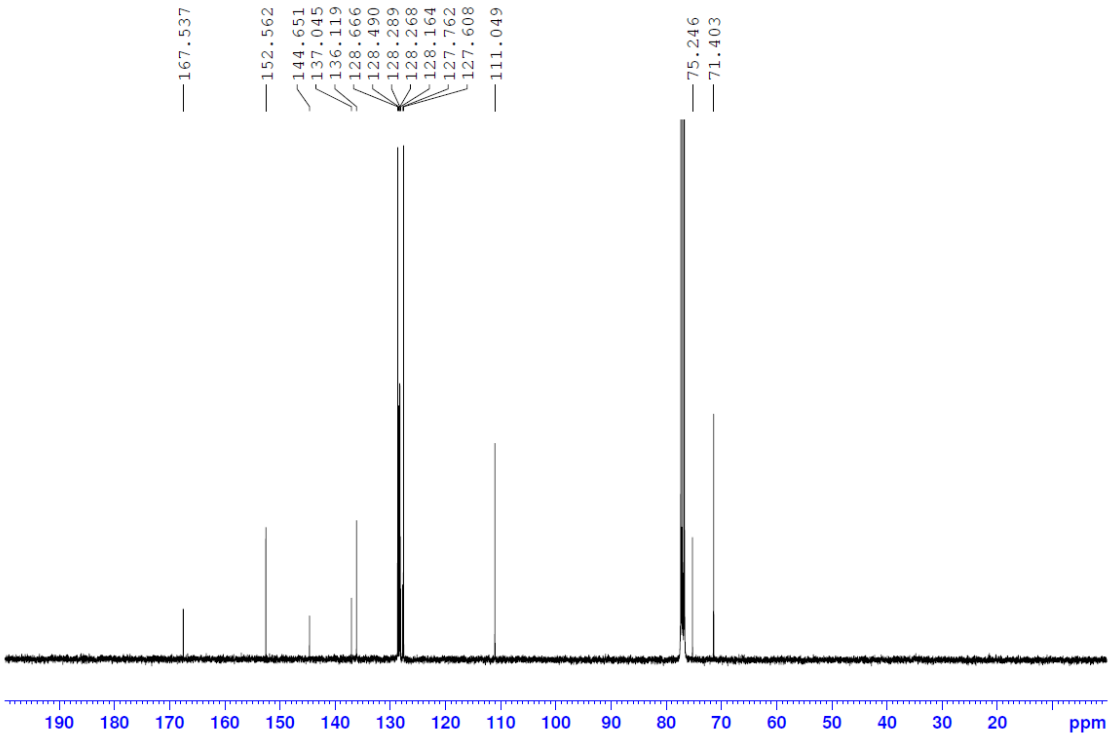
$^{1}H$ and $^{13}C$ NMR spectra of compound **9** in MeOD



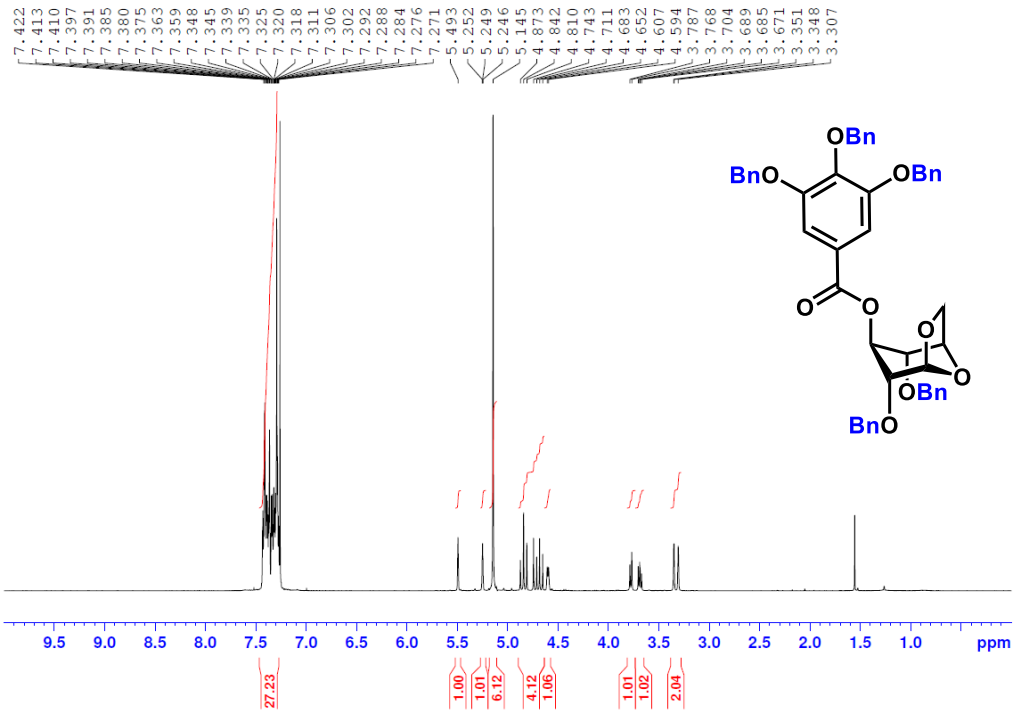



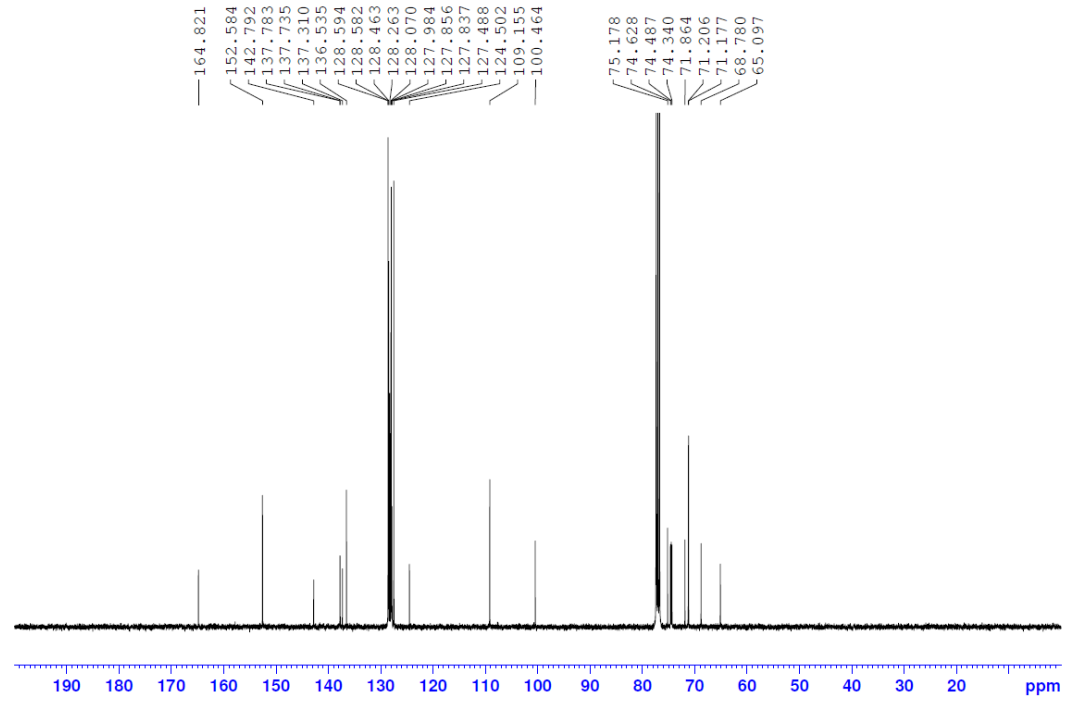
$^{1}H$ and $^{13}C$ NMR spectra of compound **10** in $CDCl_3$



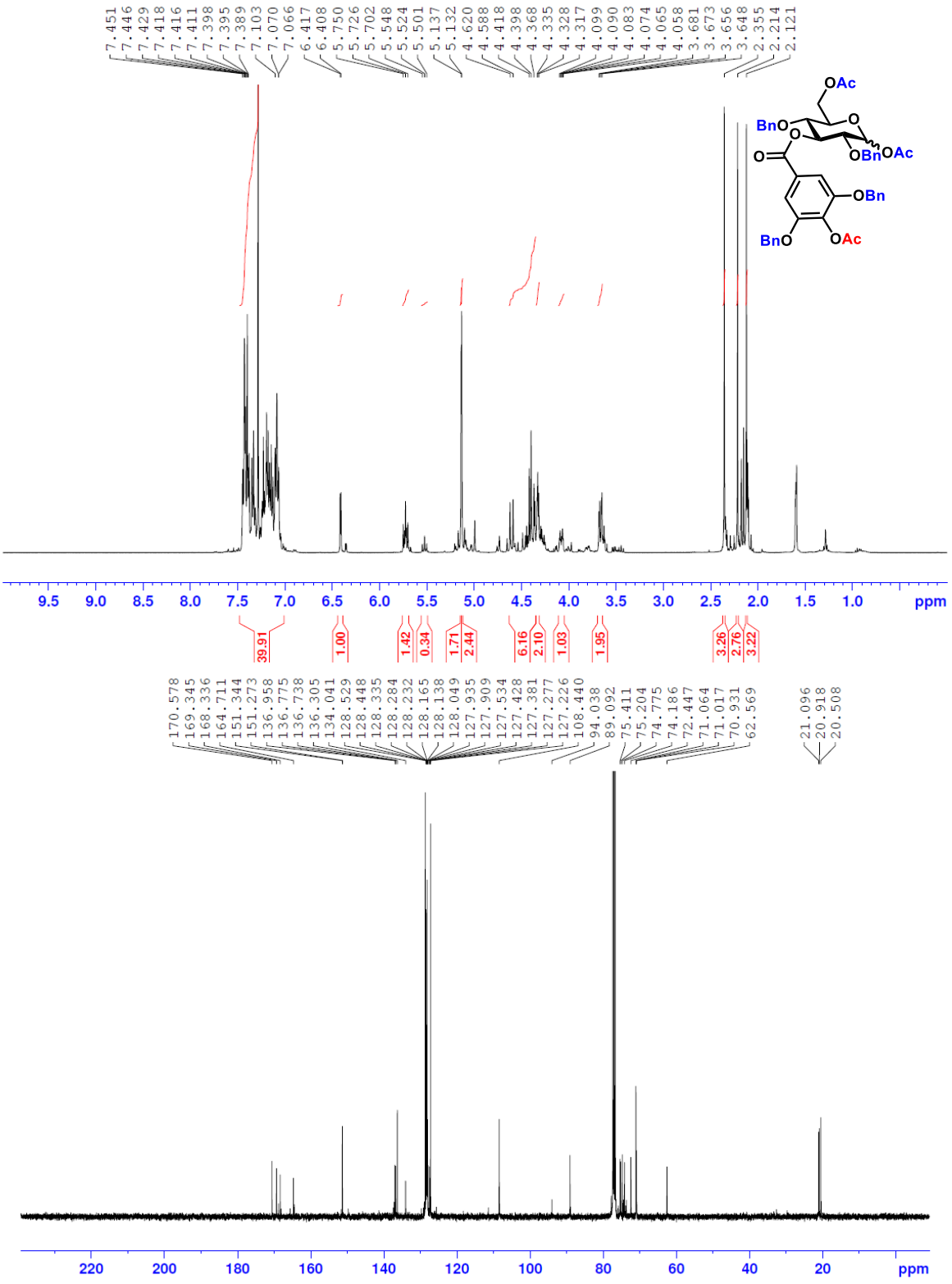



$^{1}H$ and $^{13}C$ NMR spectra of compound **11** in $CDCl_3$

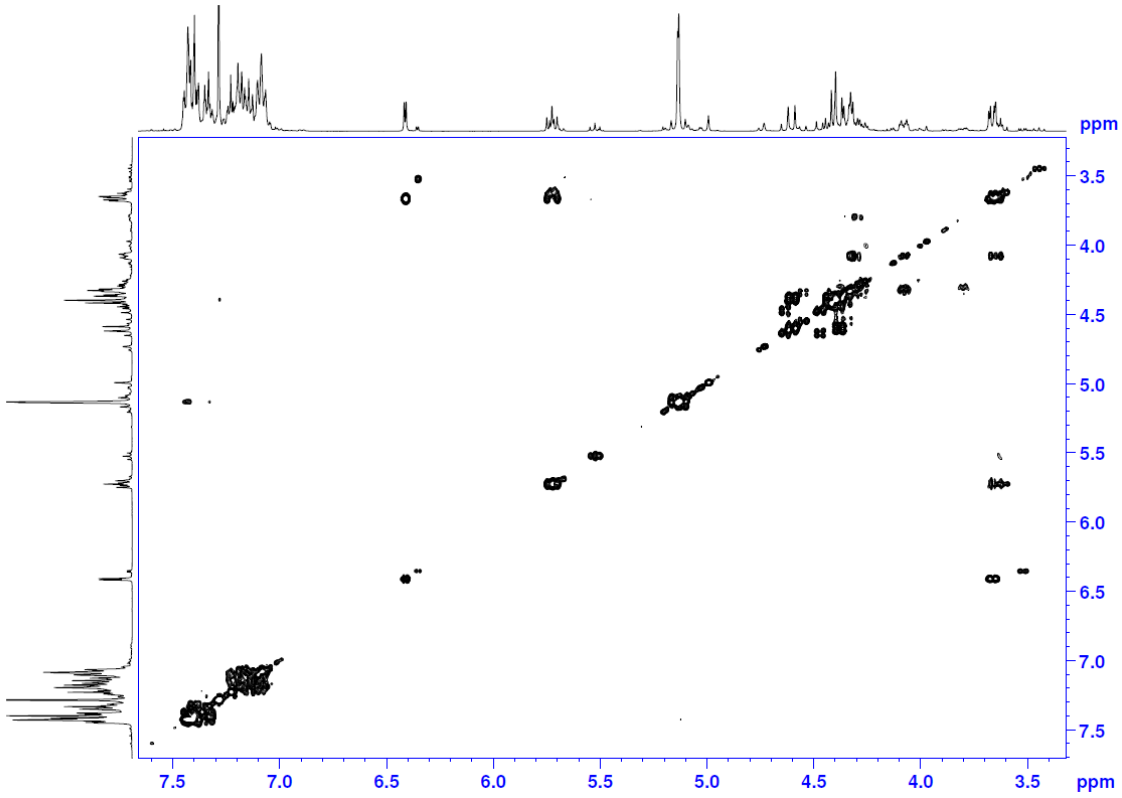


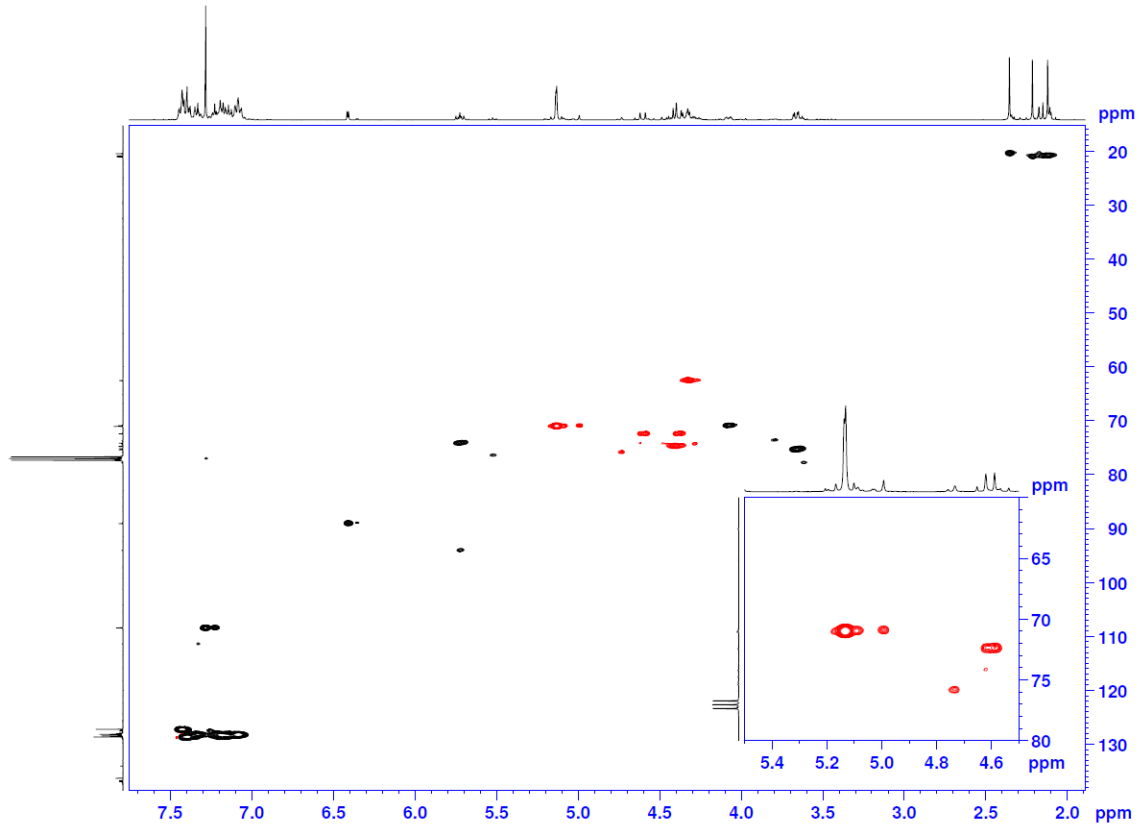



$^{1}H$ and $^{13}C$ NMR spectra of compound **12** in $CDCl_3$

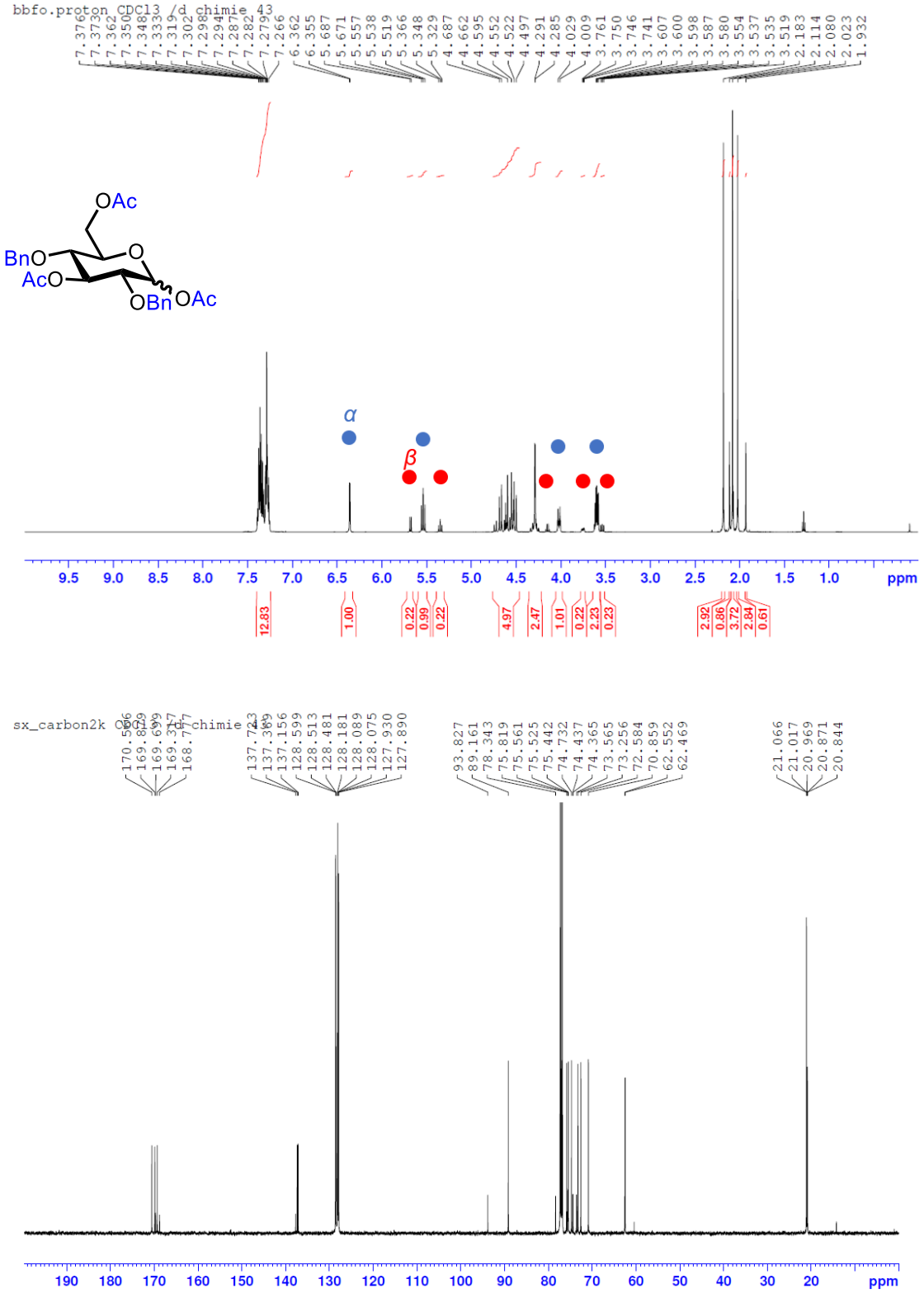


COSY and HSQC NMR spectra of compound **12** in $CDCl_3$

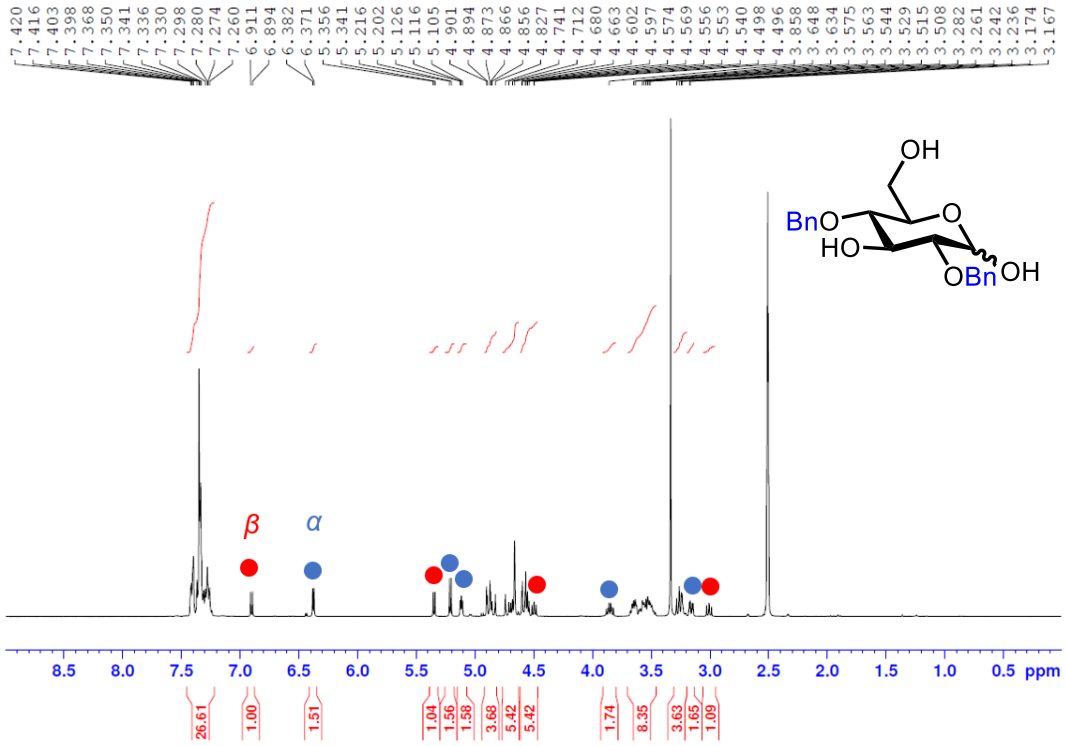


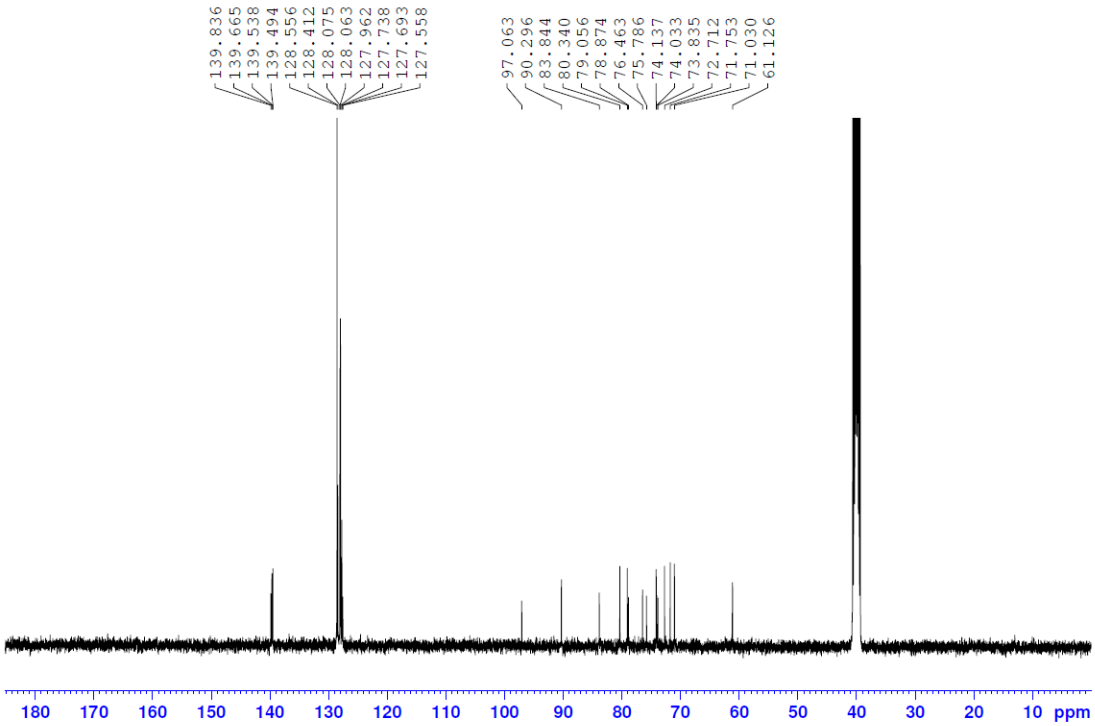



$^{1}H$ and $^{13}C$ NMR spectra of compound **13** in $CDCl_3$

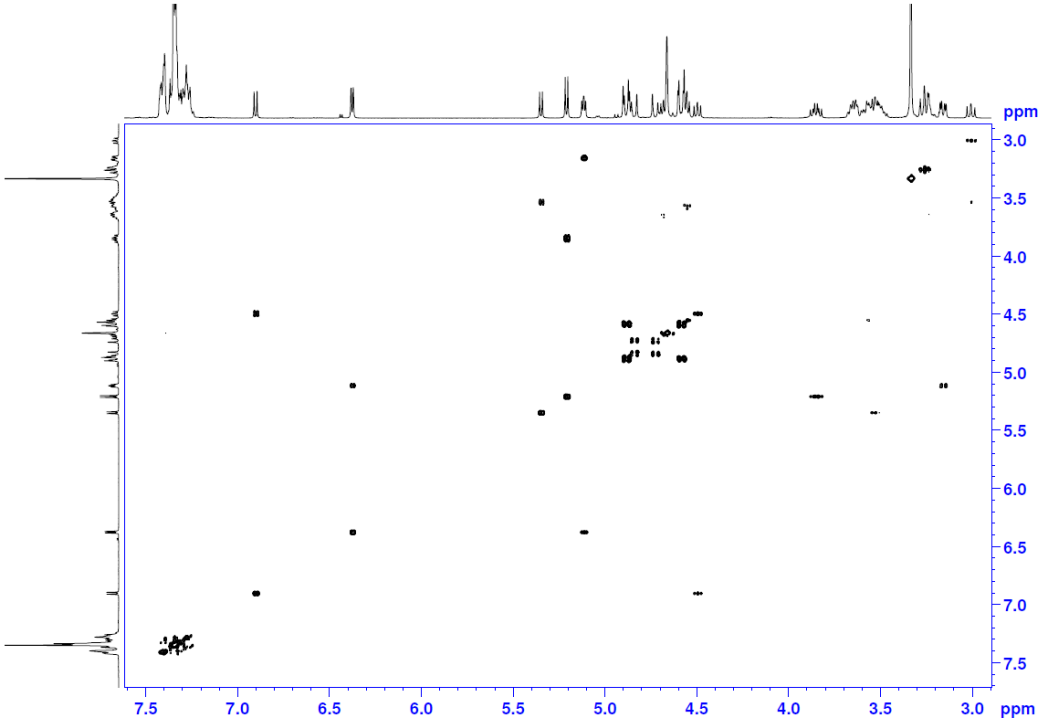


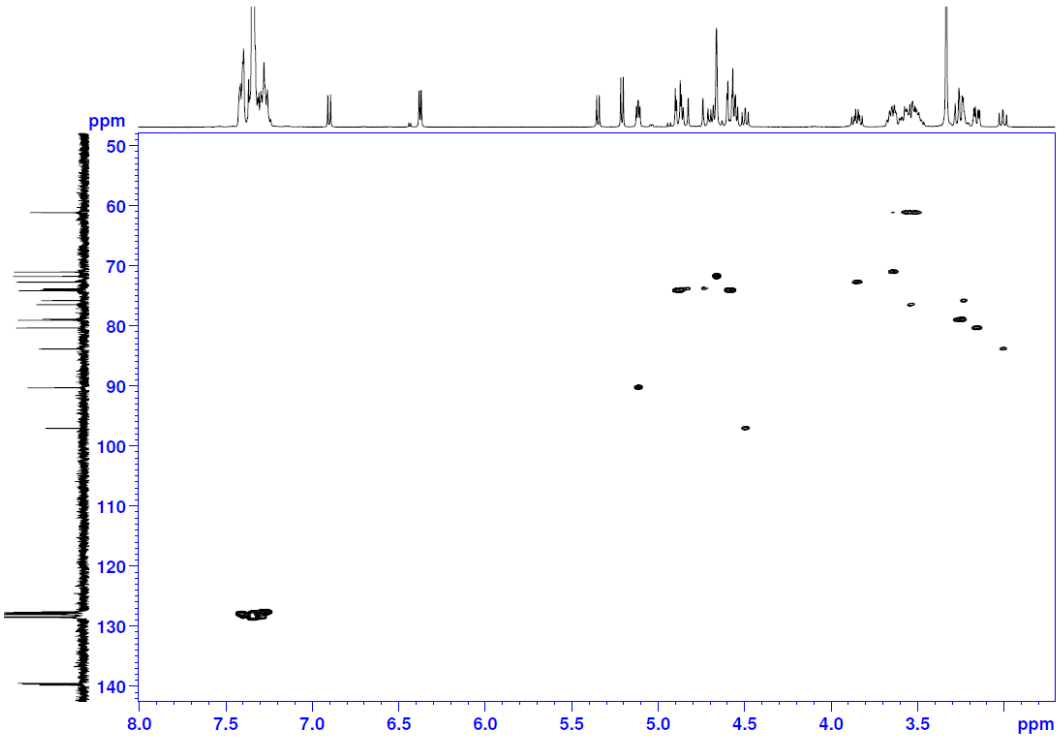
$^{1}H$ and $^{13}C$ NMR spectra of compound **14** in DMSO-$d_6$



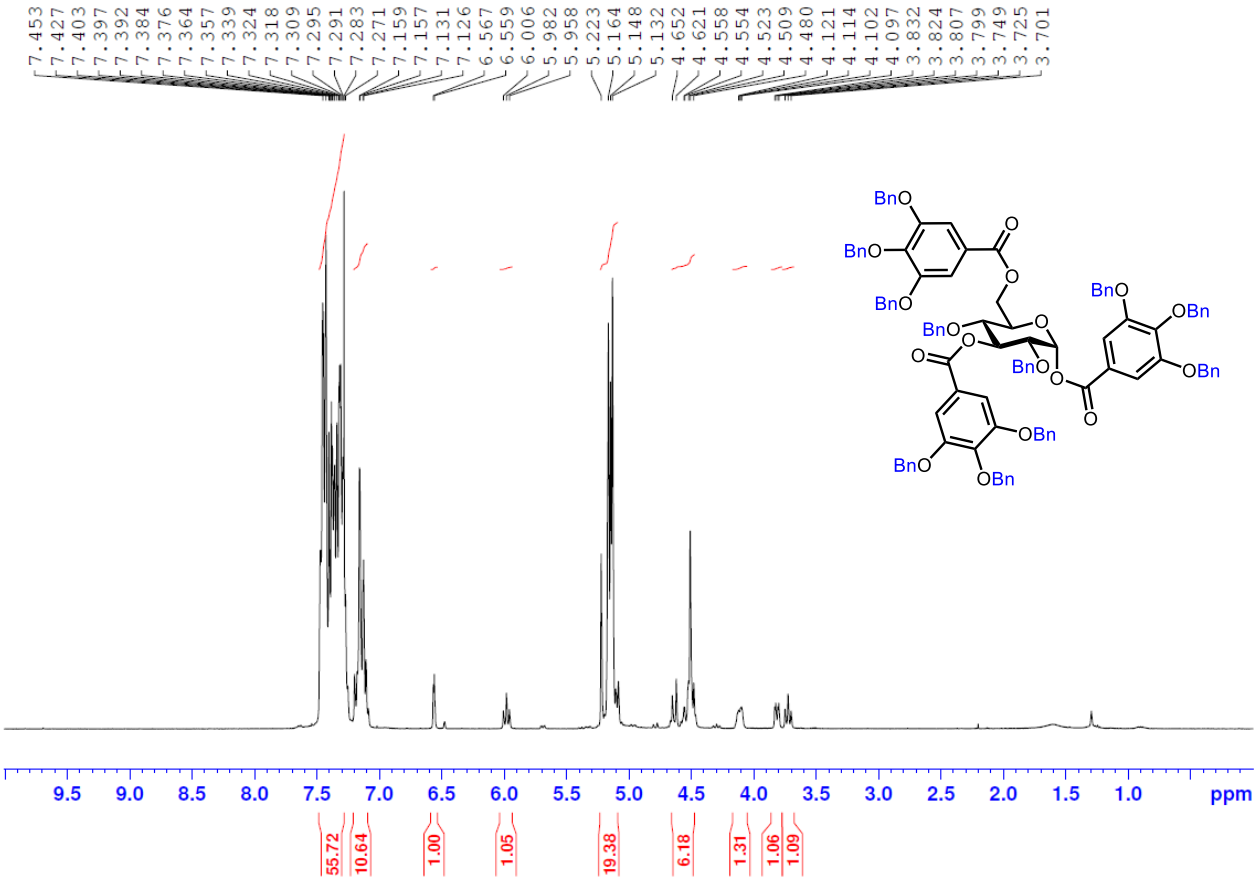



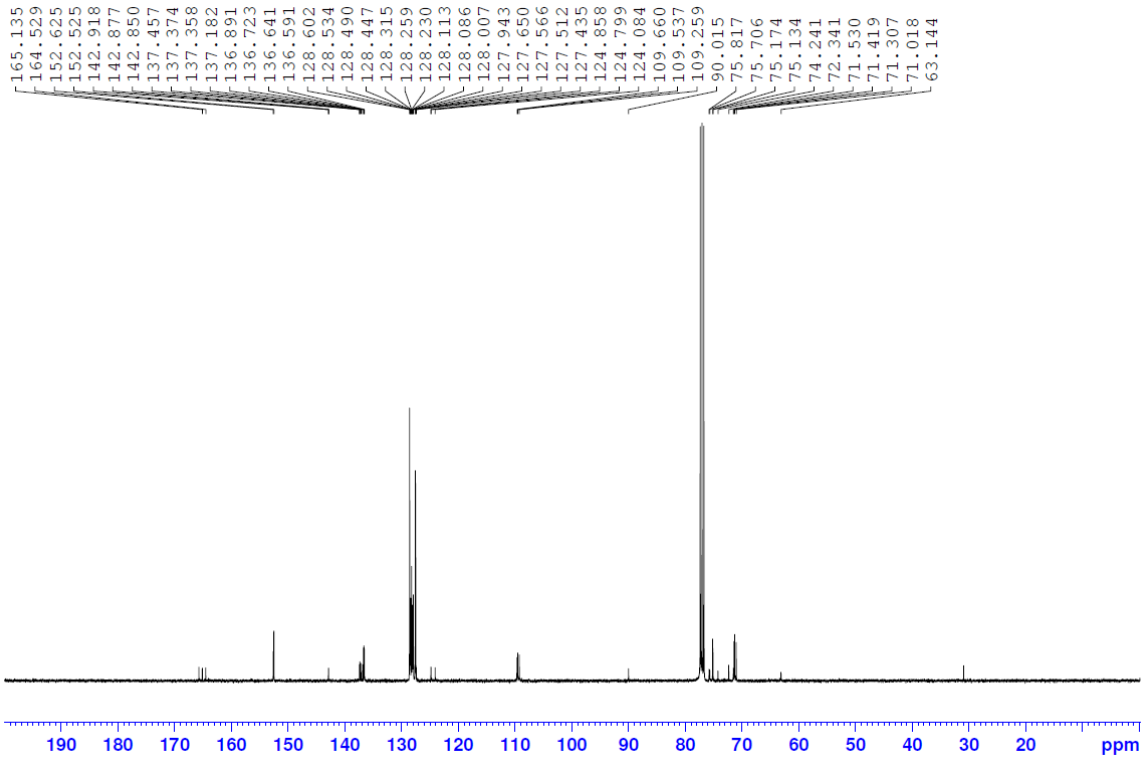
COSY and HSQC NMR spectra of compound **14** in DMSO-$d_6$



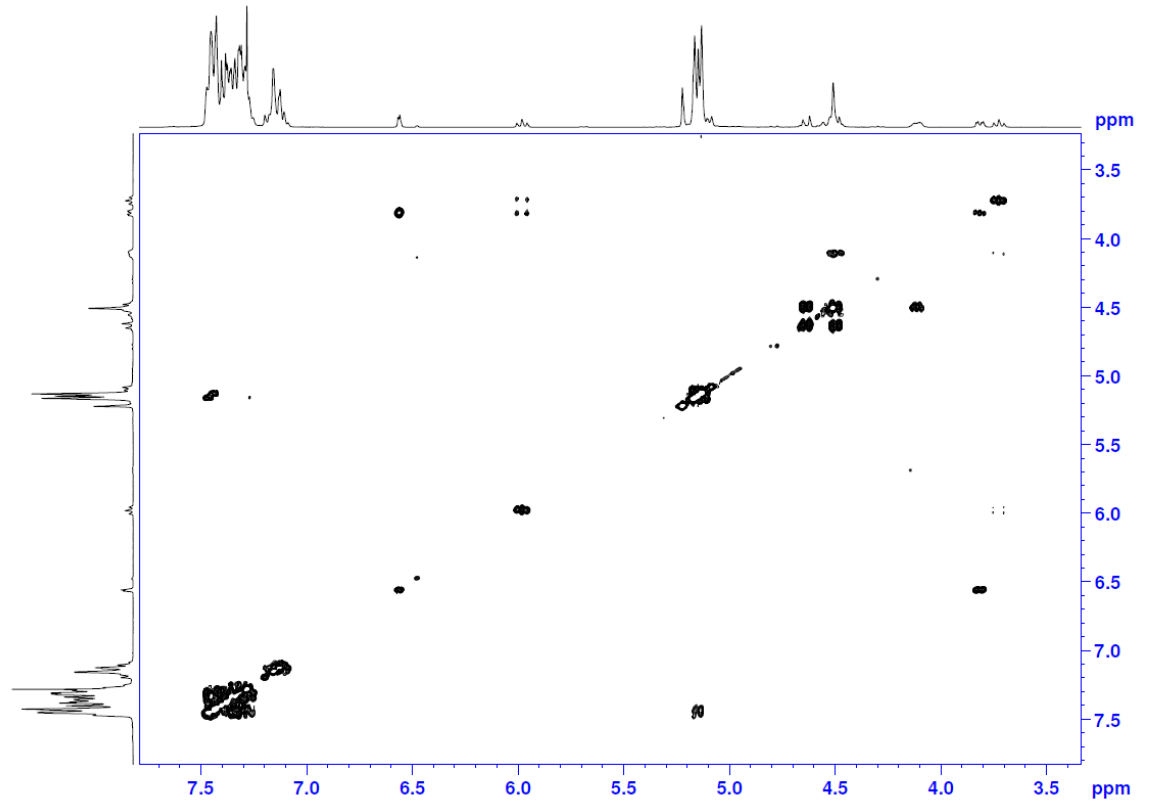



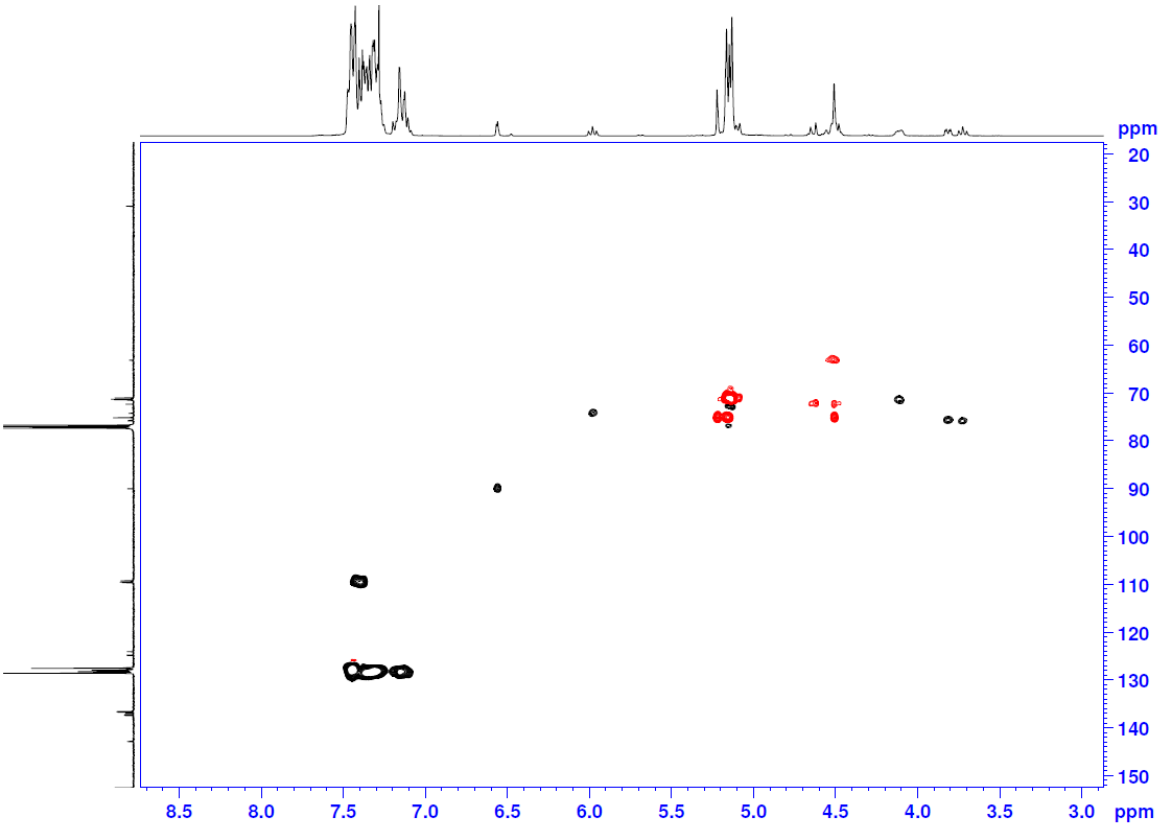
$^{1}H$ and $^{13}C$ NMR spectra of compound **α-16** in $CDCl_3$



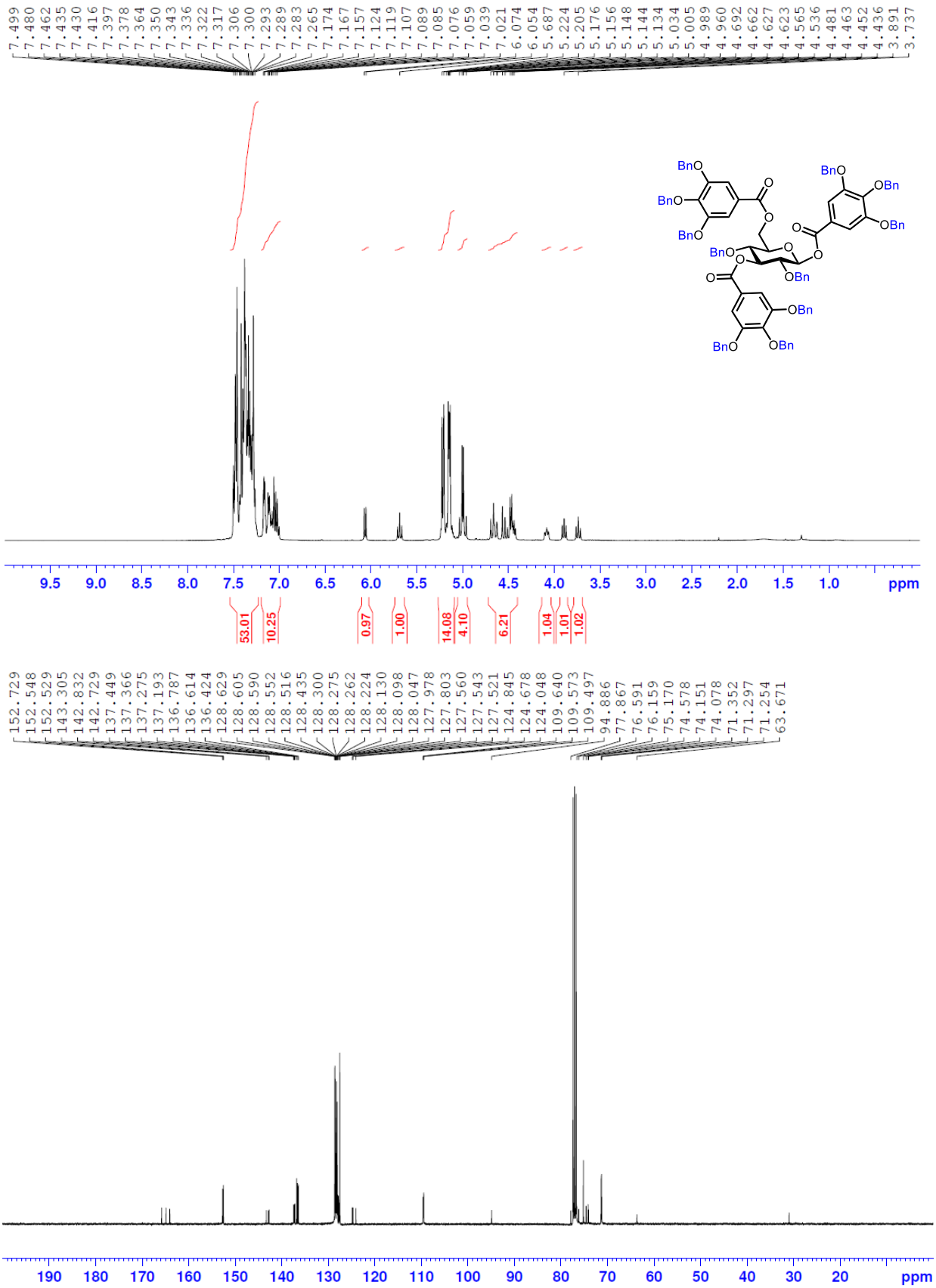



COSY and HSQC NMR spectra of compound **α-16** in $CDCl_3$

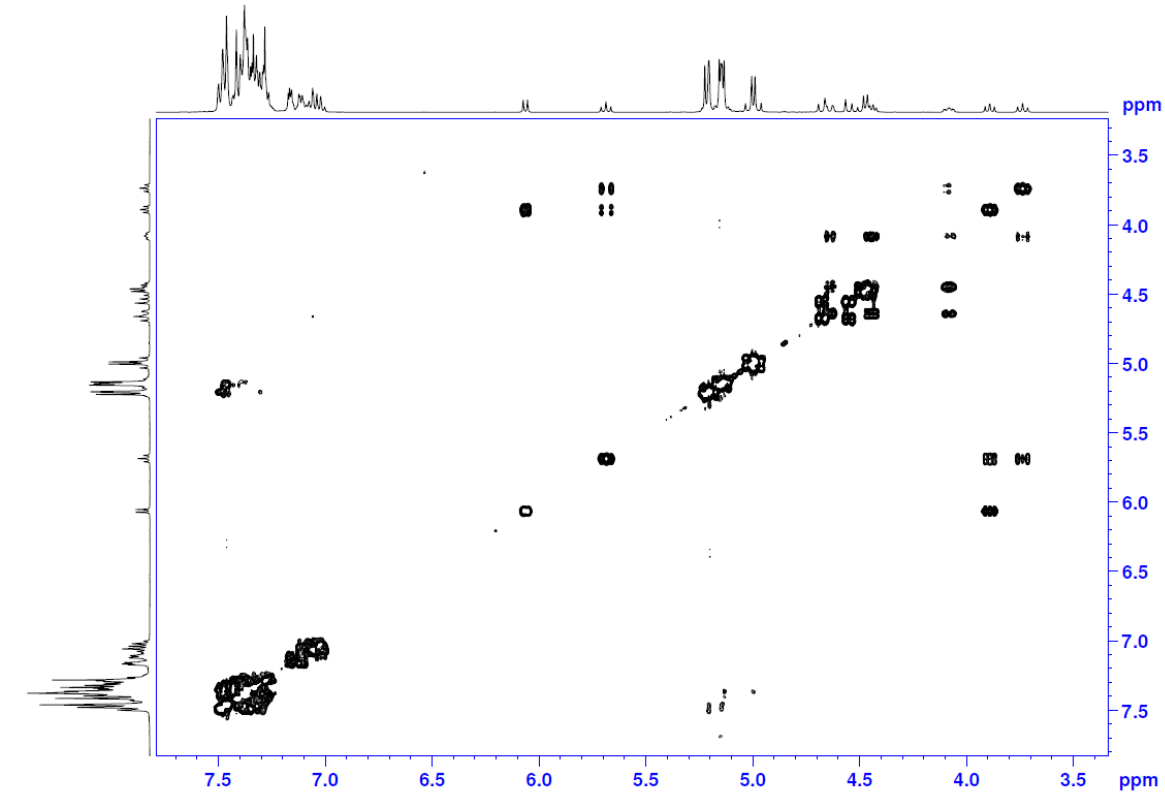


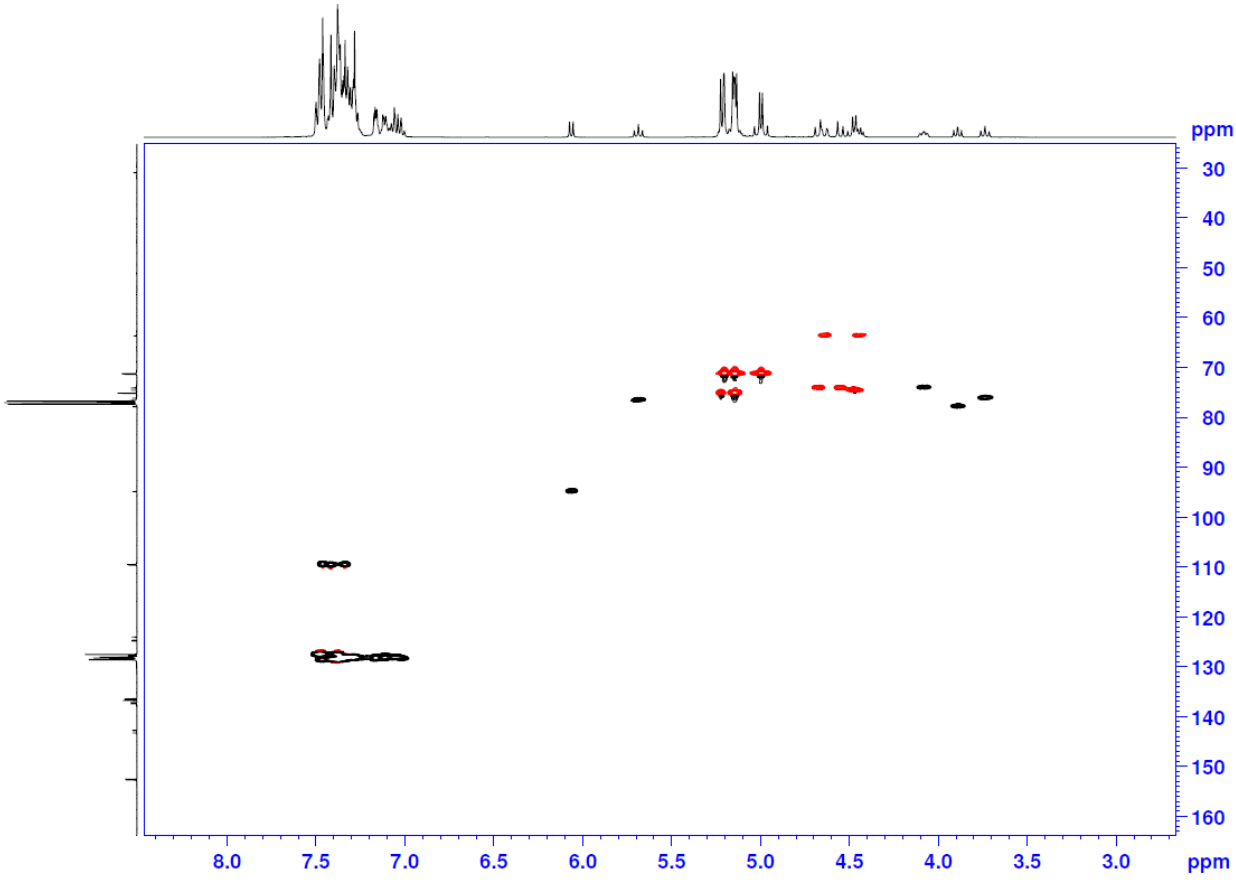



$^{1}H$ and $^{13}C$ NMR spectra of compound **β-16** in $CDCl_3$

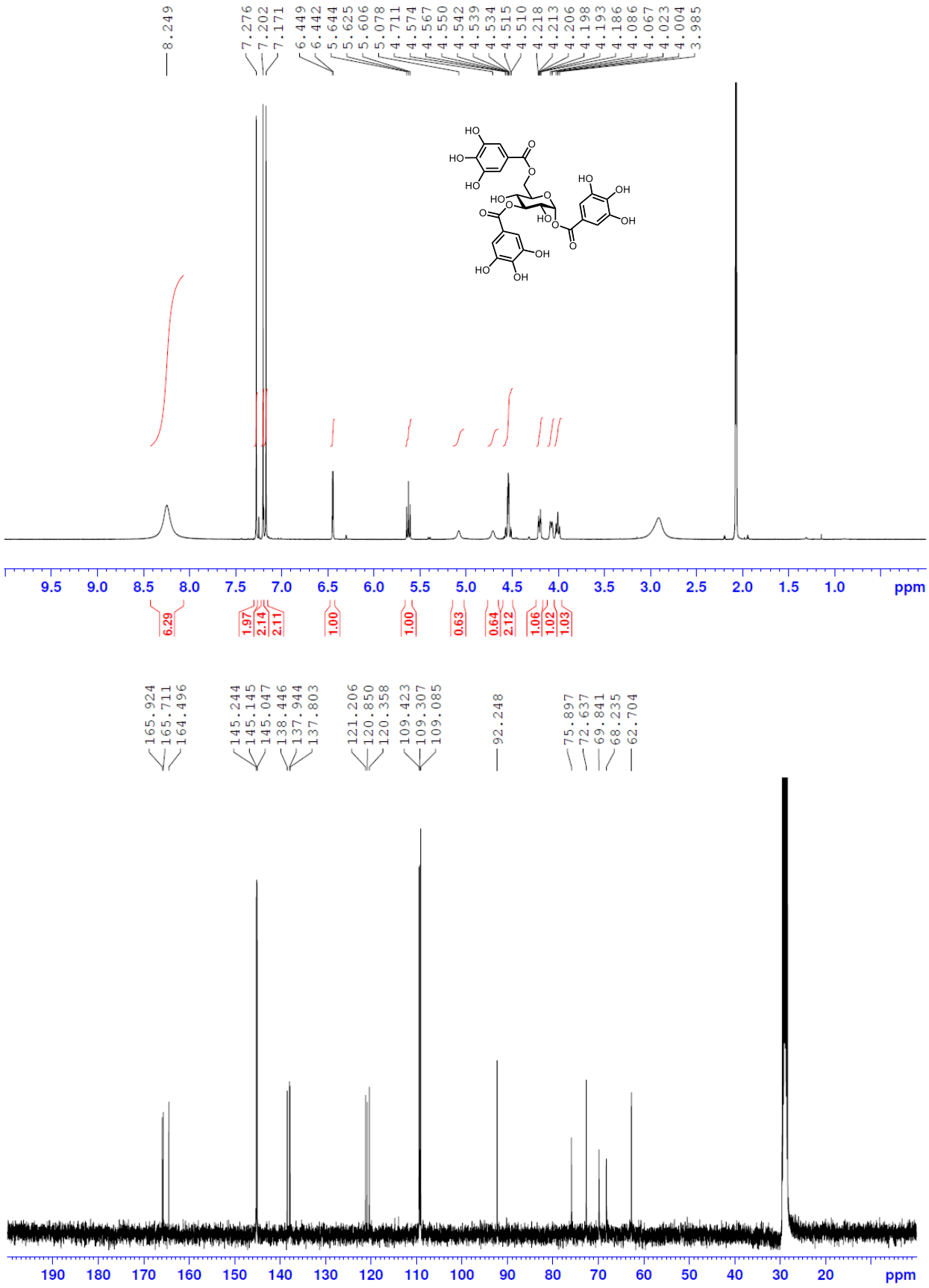


COSY and HSQC NMR spectra of compound **β-16** in $CDCl_3$

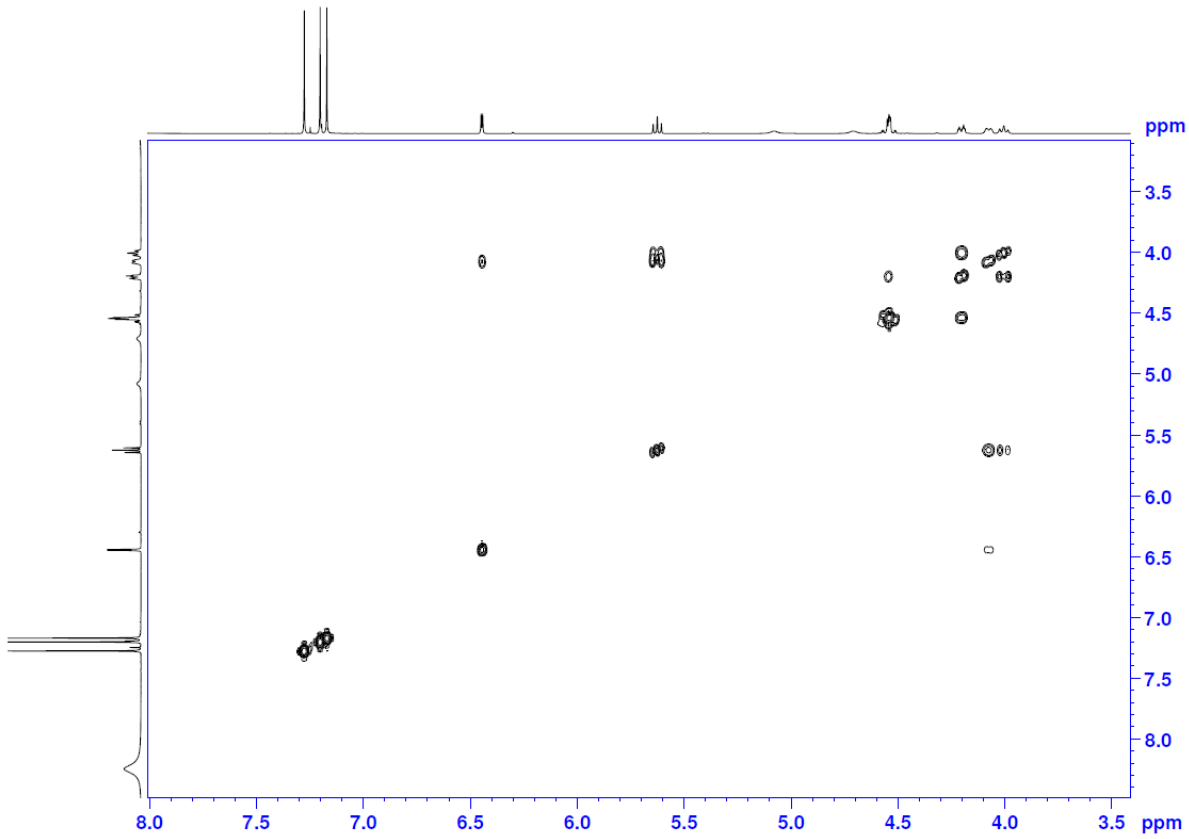


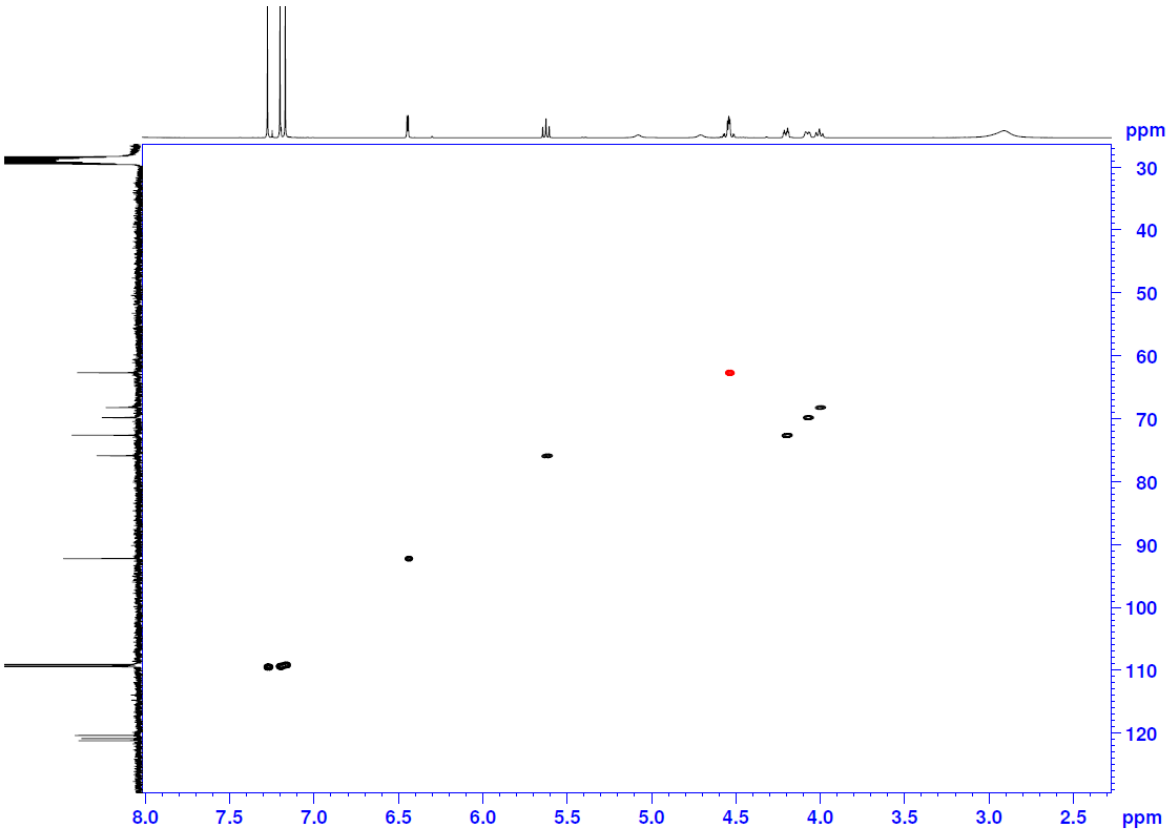



$^{1}H$ and $^{13}C$ NMR spectra of compound **17** in acetone-$d_6$

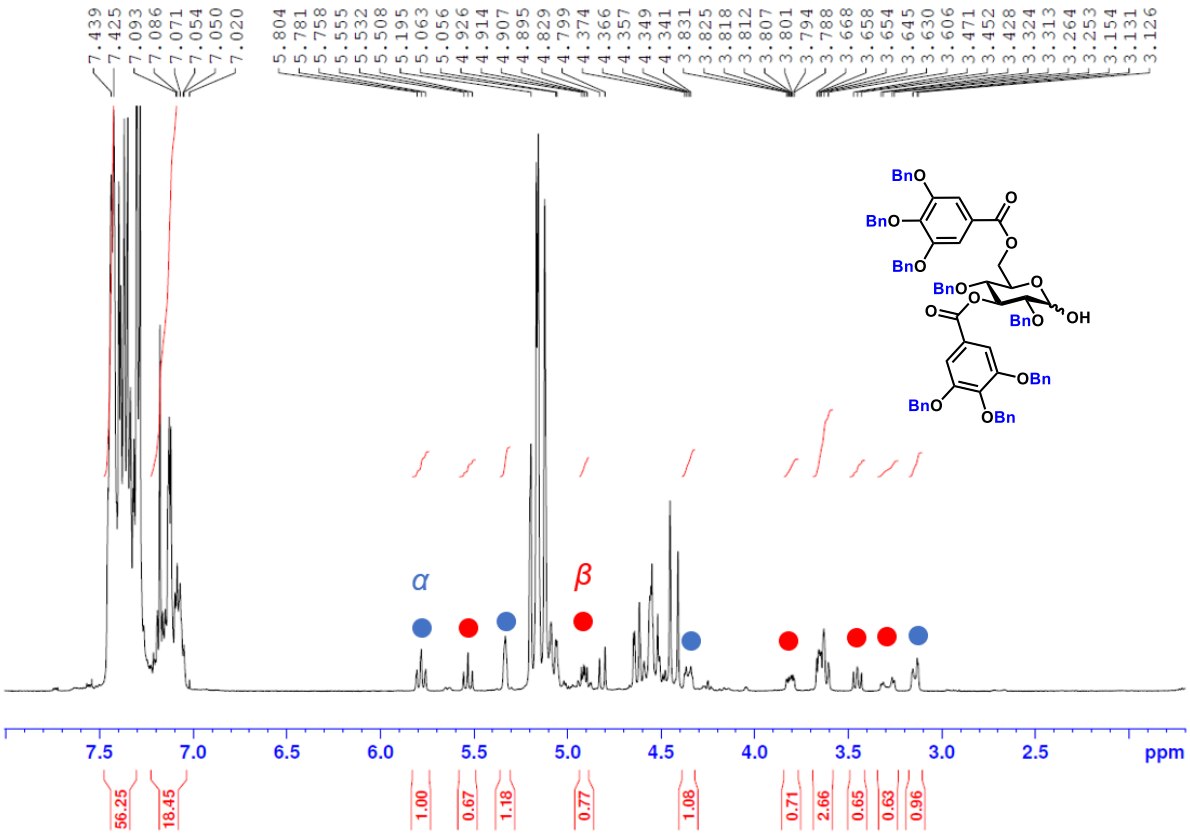


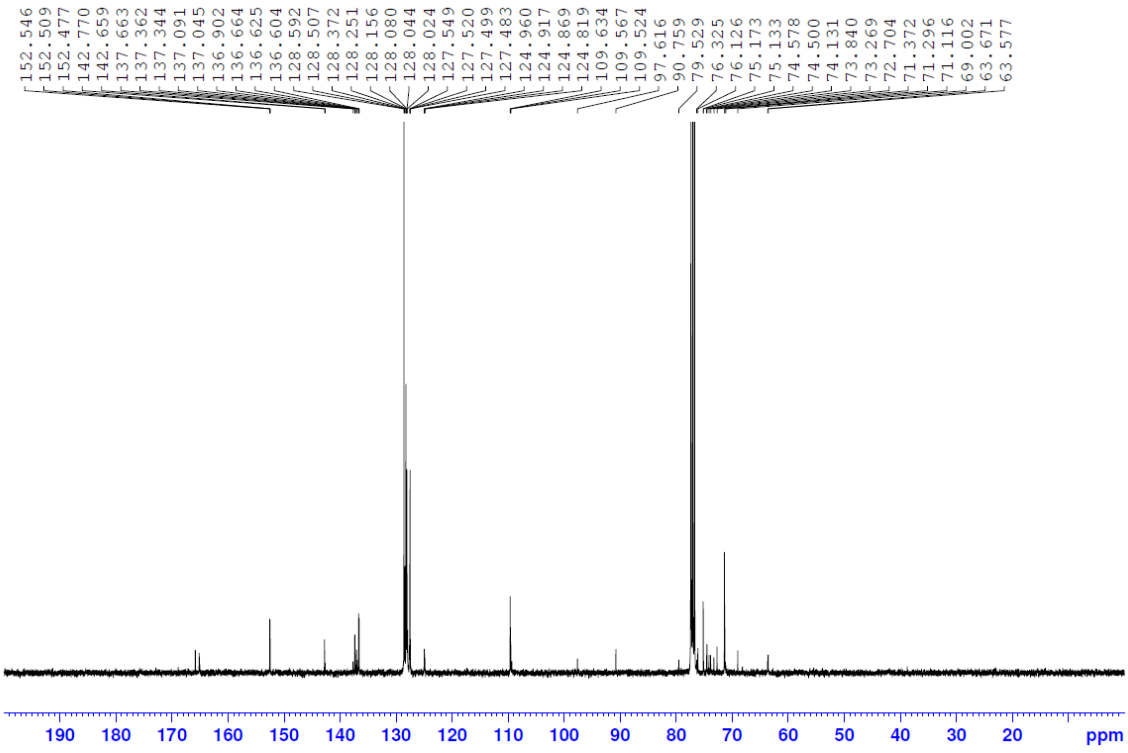
COSY and HSQC NMR spectra of compound **17** in acetone-$d_6$



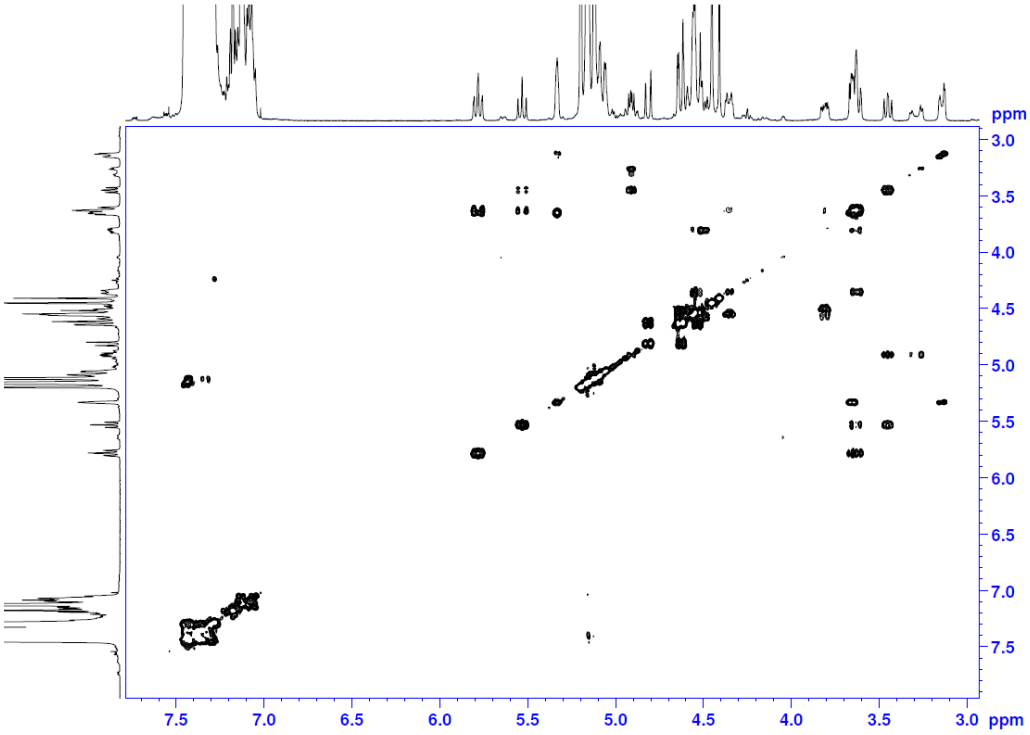



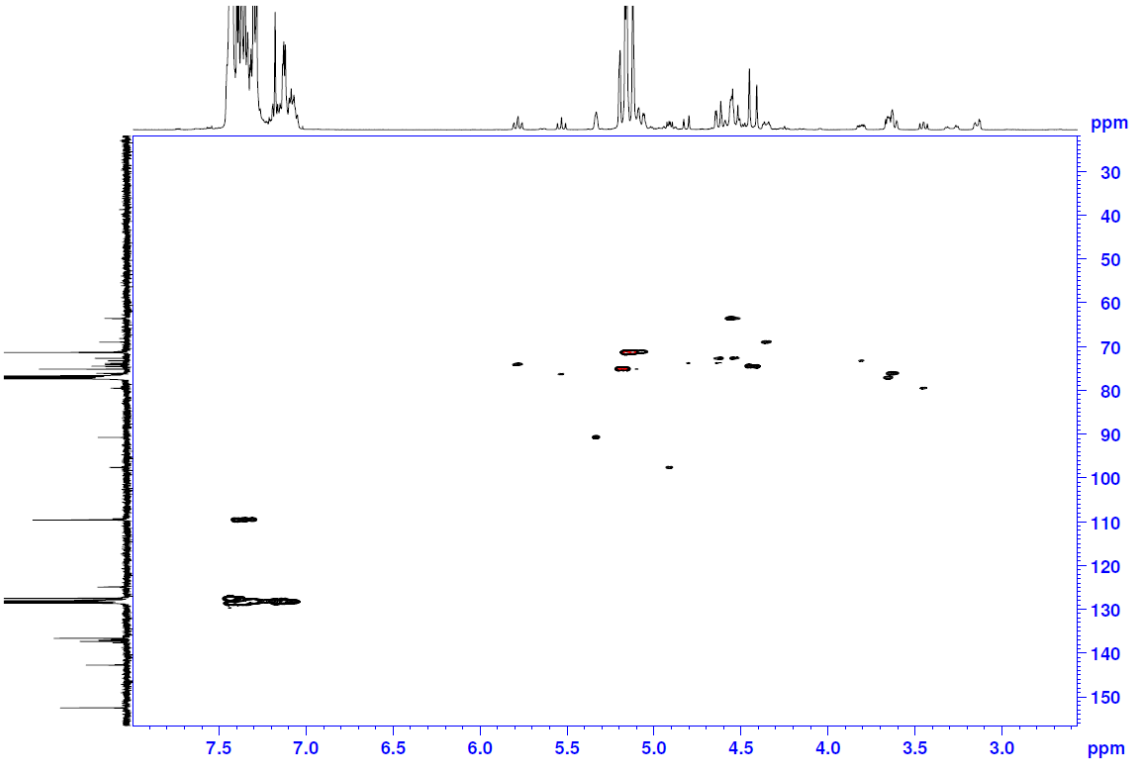
1H and 13C NMR spectra of compound **18** in CDCl3



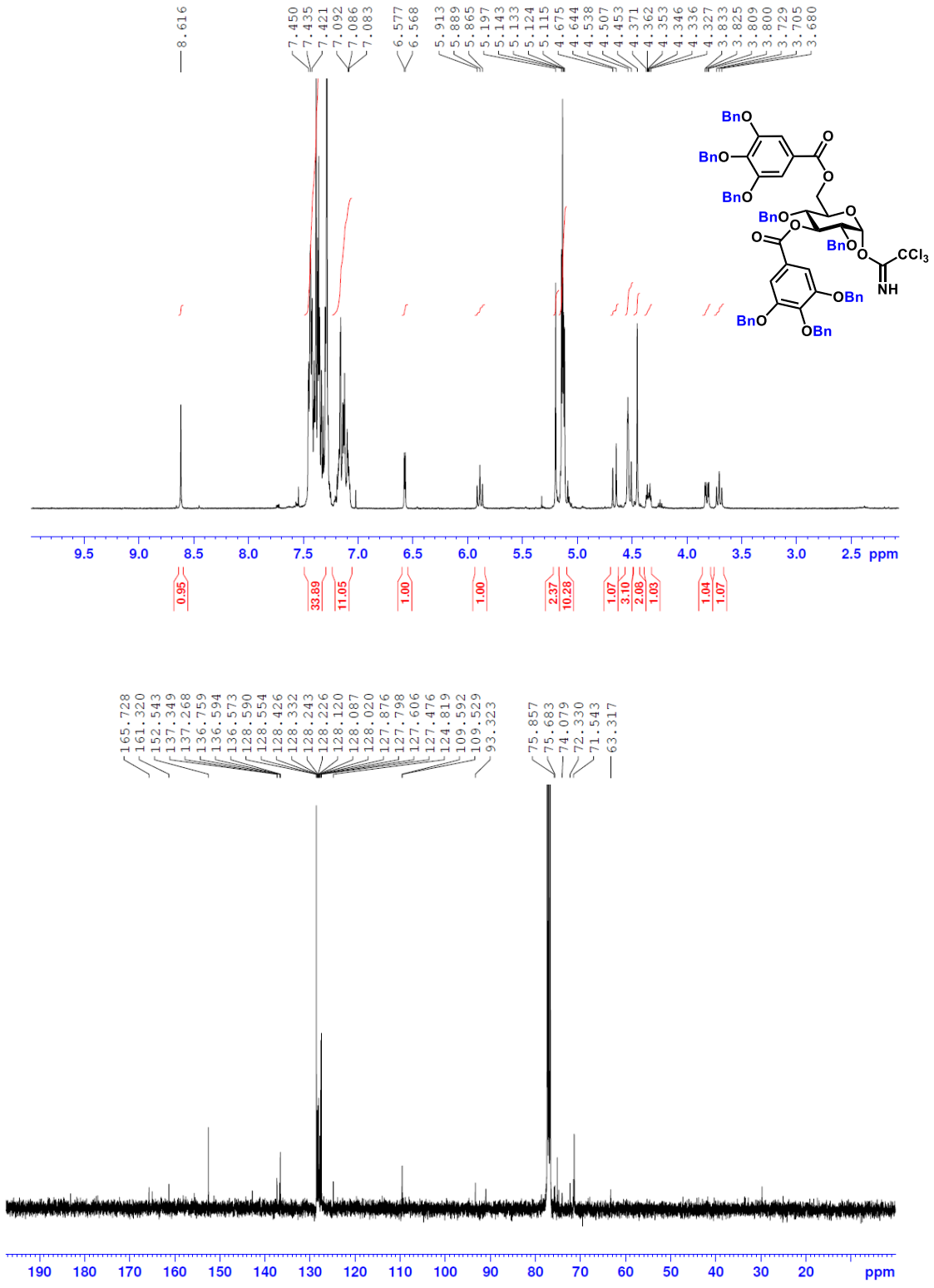



COSY and HSQC NMR spectra of compound **18** in $CDCl_3$

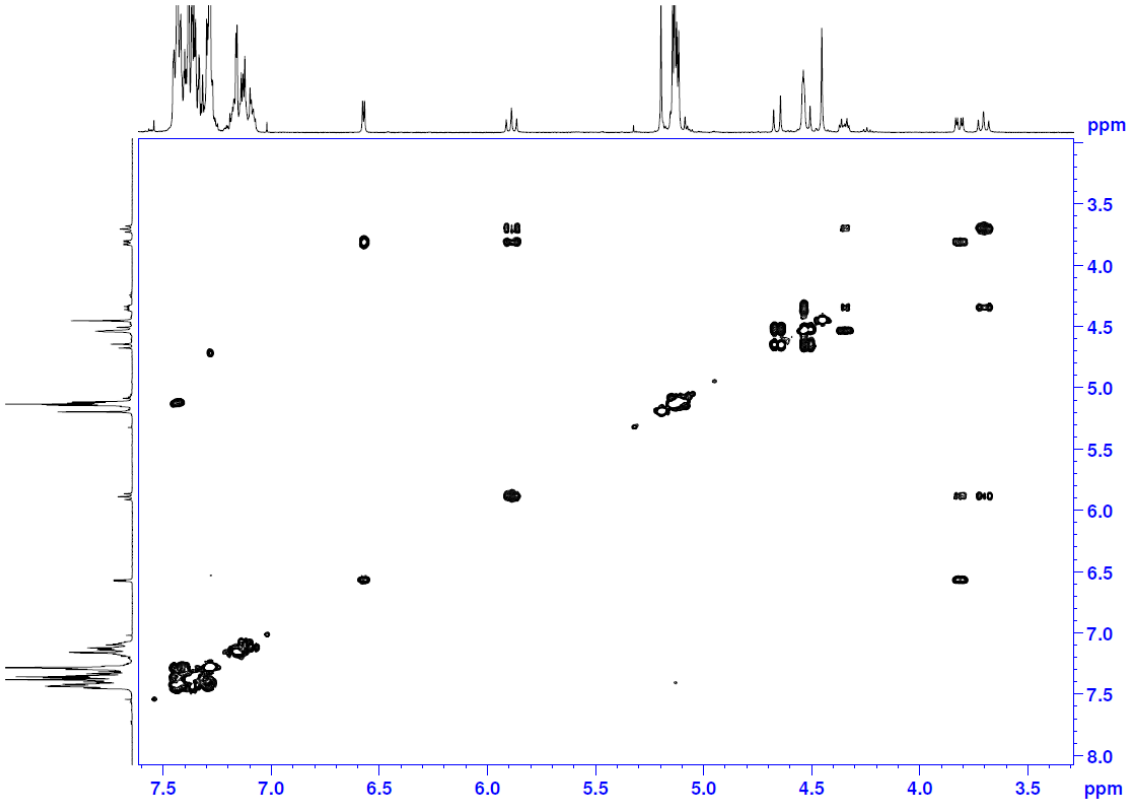


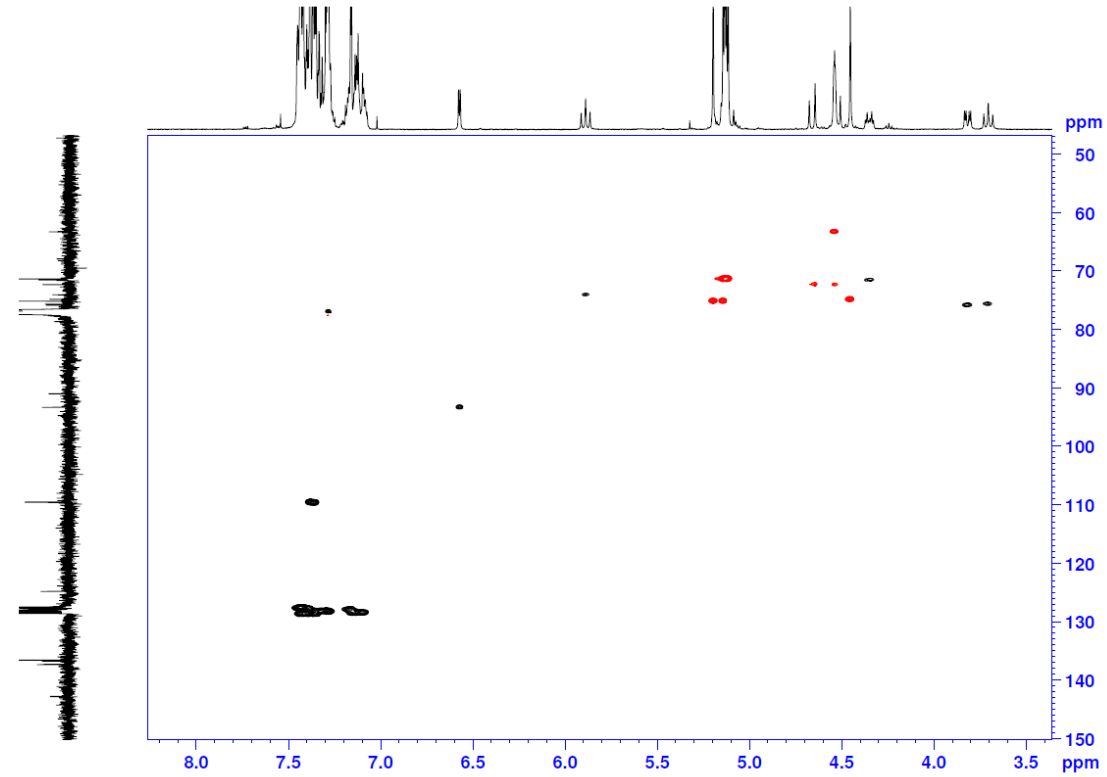



$^{1}H$ and $^{13}C$ NMR spectra of compound **19** in $CDCl_3$

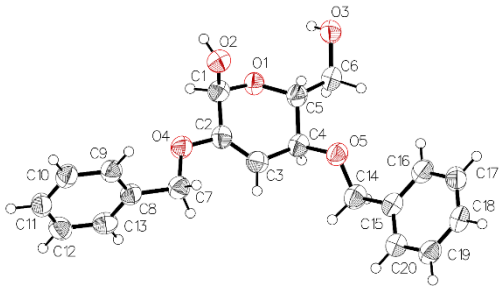


COSY and HSQC NMR spectra of compound **19** in $CDCl_3$



Crystal structure of **α-15**




## Table 1. Crystal data and structure refinement for cha251

| | |
|---|---|
| Empirical formula | $C_{20}H_{22}O_5$ |
| Formula weight | 342.37 |
| Temperature [K] | 100 |
| Crystal system | orthorhombic |
| Space group (number) | $P2_12_12_1$ (19) |
| *a* [Å] | 5.6875(5) |
| *b* [Å] | 14.3227(11) |
| *c* [Å] | 21.3425(18) |
| α [°] | 90 |
| β [°] | 90 |
| γ [°] | 90 |
| Volume [Å$^3$] | 1738.6(3) |
| *Z* | 4 |
| $\rho_{calc}$ [gcm$^{-3}$] | 1.308 |
| $\mu$ [mm$^{-1}$] | 0.767 |
| *F*(000) | 728 |
| Crystal size [mm$^3$] | 0.12×0.17×0.21 |
| Crystal colour | clear light colourless |
| Crystal shape | Fragment |
| Radiation | Cu $K_\alpha$ (λ=1.54178 Å) |
| 2Θ range [°] | 7.43 to 133.22 (0.84 Å) |
| Index ranges | ≤ h ≤<br>≤ k ≤<br>≤ l ≤ |
| Reflections collected | 1811 |
| Independent reflections | 1811<br>$R_{int}$ = 0.0863<br>$R_{sigma}$ = 0.0317 |
| Completeness to θ = 66.612° | 100.0 % |
| Data / Restraints / Parameters | 1811 / 2 / 234 |
| Goodness-of-fit on $F^2$ | 1.108 |
| Final *R* indexes [$I$≥2σ($I$)] | $R_1$ = 0.0531<br>w$R_2$ = 0.1442 |
| Final *R* indexes [all data] | $R_1$ = 0.0566<br>w$R_2$ = 0.1480 |
| Largest peak/hole [eÅ$^{-3}$] | 0.24/−0.26 |
| Flack X parameter | 0.5(4) |

## Table 2. Atomic coordinates and Ueq [Å$^2$] for cha251

| Atom | *x* | *y* | *z* | $U_{eq}$ |
|---|---|---|---|---|
| O1 | 0.6565(5) | 0.32672(17) | 0.50459(12) | 0.0448(6) |
| O2 | 0.8571(5) | 0.2723(2) | 0.41688(14) | 0.0519(7) |
| H2 | 0.941(10) | 0.228(3) | 0.432(3) | 0.09(2) |
| O3 | 0.9359(5) | 0.45052(19) | 0.57778(14) | 0.0510(7) |
| H3 | 0.915(12) | 0.401(3) | 0.600(2) | 0.087(19) |
| O4 | 0.4094(5) | 0.23642(19) | 0.36303(13) | 0.0487(7) |
| O5 | 0.4541(5) | 0.54630(18) | 0.44054(13) | 0.0478(7) |
| C1 | 0.6460(7) | 0.2710(3) | 0.44970(19) | 0.0480(9) |
| H1 | 0.610008 | 0.205126 | 0.462037 | 0.058 |
| C2 | 0.4556(8) | 0.3052(3) | 0.40618(19) | 0.0460(9) |
| C3 | 0.3527(7) | 0.3870(3) | 0.41236(18) | 0.0466(8) |
| H3A | 0.232130 | 0.404559 | 0.383888 | 0.056 |
| C4 | 0.4237(7) | 0.4537(3) | 0.46424(18) | 0.0458(9) |
| H4 | 0.300519 | 0.453865 | 0.497690 | 0.055 |
| C5 | 0.6588(7) | 0.4258(2) | 0.49229(18) | 0.0453(8) |
| H5 | 0.786699 | 0.440199 | 0.461632 | 0.054 |
| C6 | 0.7107(8) | 0.4743(3) | 0.5534(2) | 0.0493(9) |
| H6A | 0.703056 | 0.542715 | 0.546909 | 0.059 |
| H6B | 0.588582 | 0.457287 | 0.584390 | 0.059 |
| C7 | 0.2531(8) | 0.2597(3) | 0.31150(19) | 0.0483(9) |
| H7A | 0.328593 | 0.305385 | 0.283111 | 0.058 |
| H7B | 0.104938 | 0.286976 | 0.327543 | 0.058 |
| C8 | 0.2047(7) | 0.1699(3) | 0.27745(18) | 0.0452(8) |
| C9 | 0.3680(7) | 0.1346(3) | 0.23501(18) | 0.0469(9) |
| H9 | 0.508222 | 0.168367 | 0.226581 | 0.056 |
| C10 | 0.3265(8) | 0.0503(3) | 0.20496(18) | 0.0515(9) |
| H10 | 0.438791 | 0.025874 | 0.176349 | 0.062 |
| C11 | 0.1204(8) | 0.0018(3) | 0.2169(2) | 0.0556(10) |
| H11 | 0.091537 | −0.055834 | 0.196263 | 0.067 |
| C12 | −0.0423(8) | 0.0362(3) | 0.2584(2) | 0.0554(10) |
| H12 | −0.183071 | 0.002537 | 0.266346 | 0.066 |
| C13 | −0.0010(8) | 0.1204(3) | 0.28882(18) | 0.0516(9) |
| H13 | −0.113875 | 0.144157 | 0.317502 | 0.062 |
| C14 | 0.2429(8) | 0.5987(3) | 0.4393(2) | 0.0542(10) |
| H14A | 0.186980 | 0.609836 | 0.482602 | 0.065 |
| H14B | 0.119736 | 0.563646 | 0.416475 | 0.065 |
| C15 | 0.2884(8) | 0.6905(3) | 0.40715(19) | 0.0479(9) |
| C16 | 0.4930(8) | 0.7408(3) | 0.4189(2) | 0.0517(9) |
| H16 | 0.604230 | 0.718307 | 0.448503 | 0.062 |
| C17 | 0.5343(8) | 0.8244(3) | 0.3872(2) | 0.0533(10) |
| H17 | 0.674332 | 0.858508 | 0.395301 | 0.064 |
| C18 | 0.3751(9) | 0.8580(3) | 0.3445(2) | 0.0530(10) |
| H18 | 0.405372 | 0.914800 | 0.322918 | 0.064 |
| C19 | 0.1724(9) | 0.8092(3) | 0.33299(19) | 0.0539(10) |
| H19 | 0.060891 | 0.832853 | 0.303881 | 0.065 |
| C20 | 0.1285(8) | 0.7250(3) | 0.3638(2) | 0.0502(9) |
| H20 | −0.011406 | 0.691227 | 0.355074 | 0.060 |

$U_{eq}$ is defined as 1/3 of the trace of the orthogonalized $U_{ij}$ tensor.

## Table 3. Bond lengths and angles for cha251

| Atom–Atom | Length [Å] |
|---|---|
| O1–C1 | 1.419(5) |
| O1–C5 | 1.443(4) |
| O2–H2 | 0.858(12) |
| O2–C1 | 1.390(5) |
| O3–H3 | 0.853(12) |
| O3–C6 | 1.424(5) |
| O4–C2 | 1.374(5) |
| O4–C7 | 1.453(5) |
| O5–C4 | 1.429(4) |
| O5–C14 | 1.417(5) |
| C1–H1 | 1.0000 |
| C1–C2 | 1.509(6) |
| C2–C3 | 1.316(6) |
| C3–H3A | 0.9500 |
| C3–C4 | 1.517(6) |
| C4–H4 | 1.0000 |
| C4–C5 | 1.519(5) |
| C5–H5 | 1.0000 |
| C5–C6 | 1.507(6) |
| C6–H6A | 0.9900 |
| C6–H6B | 0.9900 |
| C7–H7A | 0.9900 |
| C7–H7B | 0.9900 |
| C7–C8 | 1.502(5) |
| C8–C9 | 1.392(6) |
| C8–C13 | 1.390(6) |
| C9–H9 | 0.9500 |
| C9–C10 | 1.388(6) |
| C10–H10 | 0.9500 |
| C10–C11 | 1.386(7) |
| C11–H11 | 0.9500 |
| C11–C12 | 1.373(7) |
| C12–H12 | 0.9500 |
| C12–C13 | 1.389(6) |
| C13–H13 | 0.9500 |
| C14–H14A | 0.9900 |
| C14–H14B | 0.9900 |
| C14–C15 | 1.506(6) |
| C15–C16 | 1.391(6) |
| C15–C20 | 1.389(6) |
| C16–H16 | 0.9500 |
| C16–C17 | 1.396(6) |
| C17–H17 | 0.9500 |
| C17–C18 | 1.372(6) |
| C18–H18 | 0.9500 |
| C18–C19 | 1.371(7) |
| C19–H19 | 0.9500 |
| C19–C20 | 1.396(6) |
| C20–H20 | 0.9500 |

| Atom–Atom–Atom | Angle [°] |
|---|---|
| C1–O1–C5 | 113.8(3) |
| C1–O2–H2 | 106(5) |
| C6–O3–H3 | 106(5) |
| C2–O4–C7 | 117.4(3) |
| C14–O5–C4 | 113.3(3) |
| O1–C1–H1 | 108.8 |
| O1–C1–C2 | 110.8(3) |
| O2–C1–O1 | 111.8(3) |
| O2–C1–H1 | 108.8 |
| O2–C1–C2 | 107.8(3) |
| C2–C1–H1 | 108.8 |
| O4–C2–C1 | 108.4(3) |
| C3–C2–O4 | 128.3(4) |
| C3–C2–C1 | 123.2(4) |
| C2–C3–H3A | 119.5 |
| C2–C3–C4 | 121.0(4) |
| C4–C3–H3A | 119.5 |
| O5–C4–C3 | 111.0(3) |
| O5–C4–H4 | 109.6 |
| O5–C4–C5 | 106.1(3) |
| C3–C4–H4 | 109.6 |
| C3–C4–C5 | 110.8(3) |
| C5–C4–H4 | 109.6 |
| O1–C5–C4 | 108.8(3) |
| O1–C5–H5 | 109.2 |
| O1–C5–C6 | 107.3(3) |
| C4–C5–H5 | 109.2 |
| C6–C5–C4 | 113.1(3) |
| C6–C5–H5 | 109.2 |
| O3–C6–C5 | 112.5(3) |
| O3–C6–H6A | 109.1 |
| O3–C6–H6B | 109.1 |
| C5–C6–H6A | 109.1 |
| C5–C6–H6B | 109.1 |
| H6A–C6–H6B | 107.8 |
| O4–C7–H7A | 110.5 |
| O4–C7–H7B | 110.5 |
| O4–C7–C8 | 106.4(3) |
| H7A–C7–H7B | 108.6 |
| C8–C7–H7A | 110.5 |
| C8–C7–H7B | 110.5 |
| C9–C8–C7 | 120.2(4) |
| C13–C8–C7 | 120.5(4) |
| C13–C8–C9 | 119.3(4) |
| C8–C9–H9 | 119.9 |
| C10–C9–C8 | 120.2(4) |
| C10–C9–H9 | 119.9 |
| C9–C10–H10 | 120.2 |
| C11–C10–C9 | 119.6(4) |
| C11–C10–H10 | 120.2 |
| C10–C11–H11 | 119.7 |
| C12–C11–C10 | 120.6(4) |
| C12–C11–H11 | 119.7 |
| C11–C12–H12 | 120.0 |
| C11–C12–C13 | 120.0(4) |
| C13–C12–H12 | 120.0 |
| C8–C13–H13 | 119.9 |
| C12–C13–C8 | 120.3(4) |

| | | | |
|---|---|---|---|
| C12–C13–H13 | 119.9 | C16–C17–H17 | 119.6 |
| O5–C14–H14A | 109.9 | C18–C17–C16 | 120.8(4) |
| O5–C14–H14B | 109.9 | C18–C17–H17 | 119.6 |
| O5–C14–C15 | 109.0(3) | C17–C18–H18 | 120.2 |
| H14A–C14–H14B | 108.3 | C19–C18–C17 | 119.7(4) |
| C15–C14–H14A | 109.9 | C19–C18–H18 | 120.2 |
| C15–C14–H14B | 109.9 | C18–C19–H19 | 119.8 |
| C16–C15–C14 | 120.9(4) | C18–C19–C20 | 120.4(4) |
| C20–C15–C14 | 120.1(4) | C20–C19–H19 | 119.8 |
| C20–C15–C16 | 118.9(4) | C15–C20–C19 | 120.3(4) |
| C15–C16–H16 | 120.1 | C15–C20–H20 | 119.8 |
| C15–C16–C17 | 119.8(4) | C19–C20–H20 | 119.8 |
| C17–C16–H16 | 120.1 | | |

## Table 4. Torsion angles for cha251

| Atom–Atom–Atom–Atom | Torsion Angle [°] | | |
|---|---|---|---|
| O1–C1–C2–O4 | −164.0(3) | C4–C5–C6–O3 | −177.5(3) |
| O1–C1–C2–C3 | 14.0(6) | C5–O1–C1–O2 | 72.0(4) |
| O1–C5–C6–O3 | 62.5(4) | C5–O1–C1–C2 | −48.3(4) |
| O2–C1–C2–O4 | 73.3(4) | C7–O4–C2–C1 | −172.1(3) |
| O2–C1–C2–C3 | −108.7(4) | C7–O4–C2–C3 | 10.1(6) |
| O4–C2–C3–C4 | 178.5(4) | C7–C8–C9–C10 | 178.0(4) |
| O4–C7–C8–C9 | −80.2(4) | C7–C8–C13–C12 | −178.4(4) |
| O4–C7–C8–C13 | 98.5(4) | C8–C9–C10–C11 | 0.7(6) |
| O5–C4–C5–O1 | −167.5(3) | C9–C8–C13–C12 | 0.4(6) |
| O5–C4–C5–C6 | 73.3(4) | C9–C10–C11–C12 | −0.3(6) |
| O5–C14–C15–C16 | −41.8(5) | C10–C11–C12–C13 | 0.0(6) |
| O5–C14–C15–C20 | 136.5(4) | C11–C12–C13–C8 | 0.0(6) |
| C1–O1–C5–C4 | 66.6(4) | C13–C8–C9–C10 | −0.7(6) |
| C1–O1–C5–C6 | −170.7(3) | C14–O5–C4–C3 | 86.9(4) |
| C1–C2–C3–C4 | 0.9(6) | C14–O5–C4–C5 | −152.7(3) |
| C2–O4–C7–C8 | −171.8(3) | C14–C15–C16–C17 | 178.2(4) |
| C2–C3–C4–O5 | 133.6(4) | C14–C15–C20–C19 | −178.8(4) |
| C2–C3–C4–C5 | 16.0(5) | C15–C16–C17–C18 | 0.2(6) |
| C3–C4–C5–O1 | −47.0(4) | C16–C15–C20–C19 | −0.4(6) |
| C3–C4–C5–C6 | −166.1(3) | C16–C17–C18–C19 | 0.4(6) |
| C4–O5–C14–C15 | −173.8(3) | C17–C18–C19–C20 | −0.9(6) |
| | | C18–C19–C20–C15 | 1.0(6) |
| | | C20–C15–C16–C17 | −0.1(6) |

Crystal structure of **α-14**

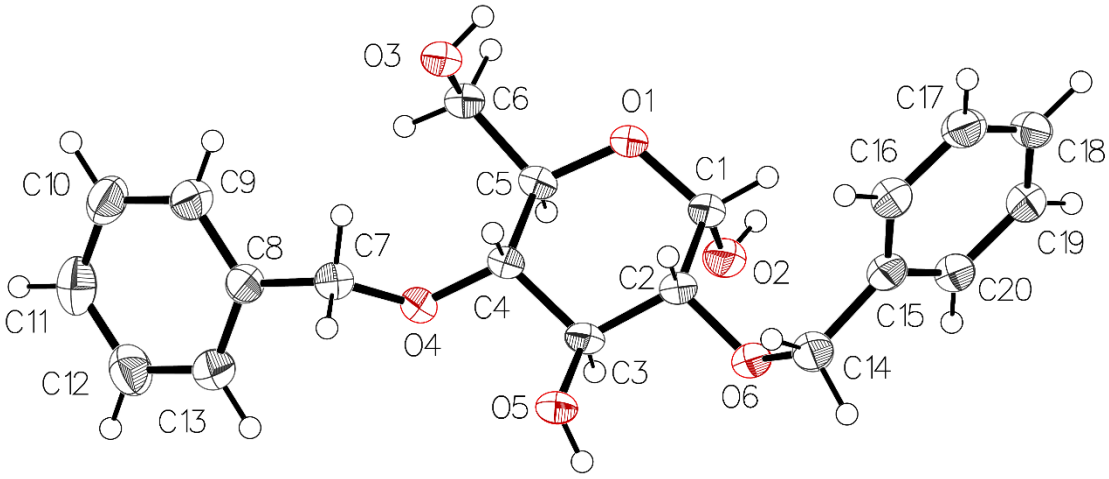



**Table 5. Crystal data and structure refinement for cha253 / ABC-YP-0722**

| | |
|---|---|
| Empirical formula | $C_{20}H_{24}O_6$ |
| Formula weight | 360.39 |
| Temperature [K] | 100 |
| Crystal system | monoclinic |
| Space group (number) | $P2_1$ (4) |
| *a* [Å] | 11.7664(6) |
| *b* [Å] | 6.3363(3) |
| *c* [Å] | 12.0727(6) |
| α [°] | 90 |
| β [°] | 94.878(3) |
| γ [°] | 90 |
| Volume [Å$^3$] | 896.83(8) |
| *Z* | 2 |
| $\rho_{calc}$ [gcm$^{-3}$] | 1.335 |
| $\mu$ [mm$^{-1}$] | 0.811 |
| *F*(000) | 384 |
| Crystal size [mm$^3$] | 0.07×0.1×0.3 |
| Crystal colour | clear light colourless |
| Crystal shape | Needle |

| | |
|---|---|
| Radiation | Cu $K_\alpha$ (λ=1.54178 Å) |
| 2Θ range [°] | 7.35 to 140.16 (0.82 Å) |
| Index ranges | -14 ≤ h ≤ 14<br>-7 ≤ k ≤ 6<br>-14 ≤ l ≤ 14 |
| Reflections collected | 15510 |
| Independent reflections | 3056<br>$R_{int}$ = 0.0386<br>$R_{sigma}$ = 0.0285 |
| Completeness to θ = 67.679° | 100.0 % |
| Data / Restraints / Parameters | 3056 / 1 / 238 |
| Goodness-of-fit on $F^2$ | 1.061 |
| Final *R* indexes [*I*≥2σ(*I*)] | $R_1$ = 0.0351<br>w$R_2$ = 0.0962 |
| Final *R* indexes [all data] | $R_1$ = 0.0367<br>w$R_2$ = 0.0978 |
| Largest peak/hole [eÅ$^{-3}$] | 0.32/−0.19 |
| Flack X parameter | -0.02(11) |

## Table 6. Atomic coordinates and Ueq [Å$^2$] for cha253

| Atom | *x* | *y* | *z* | $U_{eq}$ |
|---|---|---|---|---|
| O1 | 0.21530(14) | 0.7528(3) | 0.53334(14) | 0.0320(4) |
| O2 | 0.05041(16) | 0.5468(3) | 0.54448(16) | 0.0385(4) |
| H2 | 0.004006 | 0.642562 | 0.557545 | 0.058 |
| O3 | 0.44959(14) | 0.8653(3) | 0.60942(14) | 0.0342(4) |
| H3 | 0.424830 | 0.965250 | 0.568469 | 0.051 |
| O4 | 0.39552(13) | 0.3423(3) | 0.67287(13) | 0.0310(4) |
| O5 | 0.30963(15) | 0.1351(3) | 0.47109(14) | 0.0328(4) |
| H5 | 0.266432 | 0.029394 | 0.465968 | 0.049 |
| O6 | 0.10566(15) | 0.2978(3) | 0.36901(14) | 0.0343(4) |
| C1 | 0.1289(2) | 0.6245(4) | 0.4751(2) | 0.0319(5) |
| H1 | 0.088223 | 0.708468 | 0.413763 | 0.038 |
| C2 | 0.1868(2) | 0.4340(4) | 0.42628(19) | 0.0289(5) |
| H2A | 0.241493 | 0.484833 | 0.373114 | 0.035 |
| C3 | 0.2520(2) | 0.3071(4) | 0.51812(19) | 0.0290(5) |
| H3A | 0.197819 | 0.252112 | 0.570719 | 0.035 |
| C4 | 0.33885(19) | 0.4500(4) | 0.57969(18) | 0.0280(5) |
| H4 | 0.396415 | 0.495302 | 0.528147 | 0.034 |
| C5 | 0.2800(2) | 0.6456(4) | 0.62324(19) | 0.0300(5) |
| H5A | 0.226647 | 0.599519 | 0.678747 | 0.036 |
| C6 | 0.3642(2) | 0.8015(4) | 0.6784(2) | 0.0324(5) |
| H6A | 0.322212 | 0.927933 | 0.700593 | 0.039 |
| H6B | 0.401413 | 0.736901 | 0.746833 | 0.039 |
| C7 | 0.51813(19) | 0.3580(4) | 0.67713(19) | 0.0330(5) |
| H7A | 0.539813 | 0.494784 | 0.645343 | 0.040 |
| H7B | 0.548513 | 0.243606 | 0.632214 | 0.040 |
| C8 | 0.56866(19) | 0.3411(4) | 0.7956(2) | 0.0325(5) |
| C9 | 0.6297(2) | 0.5090(5) | 0.8435(2) | 0.0392(6) |
| H9 | 0.637269 | 0.634728 | 0.801909 | 0.047 |
| C10 | 0.6801(2) | 0.4966(5) | 0.9518(2) | 0.0455(7) |
| H10 | 0.722440 | 0.612500 | 0.983516 | 0.055 |
| C11 | 0.6682(2) | 0.3140(6) | 1.0132(2) | 0.0467(7) |
| H11 | 0.702759 | 0.304201 | 1.087065 | 0.056 |
| C12 | 0.6057(2) | 0.1458(6) | 0.9664(2) | 0.0463(7) |
| H12 | 0.596274 | 0.021802 | 1.008867 | 0.056 |
| C13 | 0.5569(2) | 0.1581(5) | 0.8576(2) | 0.0402(6) |
| H13 | 0.515520 | 0.041408 | 0.825453 | 0.048 |
| C14 | 0.0967(2) | 0.3156(5) | 0.2496(2) | 0.0367(5) |
| H14A | 0.057336 | 0.188925 | 0.217296 | 0.044 |
| H14B | 0.174390 | 0.318242 | 0.223989 | 0.044 |
| C15 | 0.0334(2) | 0.5086(4) | 0.20669(19) | 0.0337(5) |
| C16 | 0.0840(2) | 0.6537(4) | 0.1391(2) | 0.0353(5) |
| H16 | 0.161227 | 0.635282 | 0.123681 | 0.042 |
| C17 | 0.0224(2) | 0.8242(5) | 0.0944(2) | 0.0385(5) |
| H17 | 0.057043 | 0.920262 | 0.047105 | 0.046 |
| C18 | −0.0897(2) | 0.8559(4) | 0.1183(2) | 0.0372(5) |
| H18 | −0.131690 | 0.973123 | 0.087583 | 0.045 |
| C19 | −0.1404(2) | 0.7141(4) | 0.1878(2) | 0.0358(5) |
| H19 | −0.216720 | 0.735829 | 0.205481 | 0.043 |
| C20 | −0.0792(2) | 0.5421(4) | 0.2309(2) | 0.0347(5) |
| H20 | −0.114206 | 0.445370 | 0.277660 | 0.042 |

$U_{eq}$ is defined as 1/3 of the trace of the orthogonalized $U_{ij}$ tensor.

## Table 7. Bond lengths and angles for cha253

| Atom–Atom | Length [Å] |
|---|---|
| O1–C1 | 1.438(3) |
| O1–C5 | 1.442(3) |
| O2–H2 | 0.8400 |
| O2–C1 | 1.388(3) |
| O3–H3 | 0.8400 |
| O3–C6 | 1.417(3) |
| O4–C4 | 1.431(3) |
| O4–C7 | 1.443(3) |
| O5–H5 | 0.8400 |
| O5–C3 | 1.426(3) |
| O6–C2 | 1.422(3) |
| O6–C14 | 1.441(3) |
| C1–H1 | 1.0000 |
| C1–C2 | 1.529(3) |
| C2–H2A | 1.0000 |
| C2–C3 | 1.523(3) |
| C3–H3A | 1.0000 |
| C3–C4 | 1.513(3) |
| C4–H4 | 1.0000 |
| C4–C5 | 1.534(3) |
| C5–H5A | 1.0000 |
| C5–C6 | 1.513(3) |
| C6–H6A | 0.9900 |
| C6–H6B | 0.9900 |
| C7–H7A | 0.9900 |
| C7–H7B | 0.9900 |
| C7–C8 | 1.505(3) |
| C8–C9 | 1.383(4) |
| C8–C13 | 1.394(4) |
| C9–H9 | 0.9500 |
| C9–C10 | 1.391(4) |
| C10–H10 | 0.9500 |
| C10–C11 | 1.387(5) |
| C11–H11 | 0.9500 |
| C11–C12 | 1.387(5) |
| C12–H12 | 0.9500 |
| C12–C13 | 1.390(4) |
| C13–H13 | 0.9500 |
| C14–H14A | 0.9900 |
| C14–H14B | 0.9900 |
| C14–C15 | 1.501(4) |
| C15–C16 | 1.396(4) |
| C15–C20 | 1.397(4) |
| C16–H16 | 0.9500 |
| C16–C17 | 1.385(4) |
| C17–H17 | 0.9500 |
| C17–C18 | 1.388(4) |
| C18–H18 | 0.9500 |
| C18–C19 | 1.397(4) |
| C19–H19 | 0.9500 |
| C19–C20 | 1.384(4) |
| C20–H20 | 0.9500 |

| Atom–Atom–Atom | Angle [°] |
|---|---|
| C1–O1–C5 | 114.07(18) |
| C1–O2–H2 | 109.5 |
| C6–O3–H3 | 109.5 |
| C4–O4–C7 | 113.12(18) |
| C3–O5–H5 | 109.5 |
| C2–O6–C14 | 115.34(19) |
| O1–C1–H1 | 109.6 |
| O1–C1–C2 | 108.34(18) |
| O2–C1–O1 | 112.6(2) |
| O2–C1–H1 | 109.6 |
| O2–C1–C2 | 107.1(2) |
| C2–C1–H1 | 109.6 |
| O6–C2–C1 | 111.31(19) |
| O6–C2–H2A | 109.0 |
| O6–C2–C3 | 108.02(19) |
| C1–C2–H2A | 109.0 |
| C3–C2–C1 | 110.47(19) |
| C3–C2–H2A | 109.0 |
| O5–C3–C2 | 109.91(19) |
| O5–C3–H3A | 109.7 |
| O5–C3–C4 | 109.21(18) |
| C2–C3–H3A | 109.7 |
| C4–C3–C2 | 108.60(18) |
| C4–C3–H3A | 109.7 |
| O4–C4–C3 | 110.52(19) |
| O4–C4–H4 | 109.3 |
| O4–C4–C5 | 108.04(18) |
| C3–C4–H4 | 109.3 |
| C3–C4–C5 | 110.27(18) |
| C5–C4–H4 | 109.3 |
| O1–C5–C4 | 110.33(17) |
| O1–C5–H5A | 108.7 |
| O1–C5–C6 | 107.94(19) |
| C4–C5–H5A | 108.7 |
| C6–C5–C4 | 112.35(19) |
| C6–C5–H5A | 108.7 |
| O3–C6–C5 | 113.46(19) |
| O3–C6–H6A | 108.9 |
| O3–C6–H6B | 108.9 |
| C5–C6–H6A | 108.9 |
| C5–C6–H6B | 108.9 |
| H6A–C6–H6B | 107.7 |
| O4–C7–H7A | 109.7 |
| O4–C7–H7B | 109.7 |
| O4–C7–C8 | 110.00(19) |
| H7A–C7–H7B | 108.2 |
| C8–C7–H7A | 109.7 |
| C8–C7–H7B | 109.7 |
| C9–C8–C7 | 119.5(2) |
| C9–C8–C13 | 119.1(2) |
| C13–C8–C7 | 121.4(2) |
| C8–C9–H9 | 119.5 |
| C8–C9–C10 | 121.0(3) |
| C10–C9–H9 | 119.5 |
| C9–C10–H10 | 120.2 |
| C11–C10–C9 | 119.7(3) |
| C11–C10–H10 | 120.2 |
| C10–C11–H11 | 120.1 |
| C10–C11–C12 | 119.8(2) |
| C12–C11–H11 | 120.1 |
| C11–C12–H12 | 119.9 |
| C11–C12–C13 | 120.3(3) |
| C13–C12–H12 | 119.9 |

| | |
|---|---|
| C8–C13–H13 | 119.9 |
| C12–C13–C8 | 120.2(3) |
| C12–C13–H13 | 119.9 |
| O6–C14–H14A | 108.8 |
| O6–C14–H14B | 108.8 |
| O6–C14–C15 | 113.6(2) |
| H14A–C14–H14B | 107.7 |
| C15–C14–H14A | 108.8 |
| C15–C14–H14B | 108.8 |
| C16–C15–C14 | 120.9(2) |
| C20–C15–C14 | 120.2(2) |
| C20–C15–C16 | 118.8(2) |
| C15–C16–H16 | 119.8 |
| C17–C16–C15 | 120.4(2) |
| C17–C16–H16 | 119.8 |
| C16–C17–H17 | 119.8 |
| C16–C17–C18 | 120.4(2) |
| C18–C17–H17 | 119.8 |
| C17–C18–H18 | 120.2 |
| C17–C18–C19 | 119.6(2) |
| C19–C18–H18 | 120.2 |
| C18–C19–H19 | 120.1 |
| C20–C19–C18 | 119.8(2) |
| C20–C19–H19 | 120.1 |
| C15–C20–H20 | 119.6 |
| C19–C20–C15 | 120.9(2) |
| C19–C20–H20 | 119.6 |

## Table 8. Torsion angles for cha253

| Atom–Atom–Atom–Atom | Torsion Angle [°] |
|---|---|
| O1–C1–C2–O6 | 178.94(18) |
| O1–C1–C2–C3 | 58.9(2) |
| O1–C5–C6–O3 | −66.8(2) |
| O2–C1–C2–O6 | 57.2(2) |
| O2–C1–C2–C3 | −62.8(2) |
| O4–C4–C5–O1 | −176.28(17) |
| O4–C4–C5–C6 | 63.2(2) |
| O4–C7–C8–C9 | −117.8(3) |
| O4–C7–C8–C13 | 63.3(3) |
| O5–C3–C4–O4 | −65.1(2) |
| O5–C3–C4–C5 | 175.50(18) |
| O6–C2–C3–O5 | 60.5(2) |
| O6–C2–C3–C4 | 179.92(18) |
| O6–C14–C15–C16 | 123.3(2) |
| O6–C14–C15–C20 | −58.9(3) |
| C1–O1–C5–C4 | 58.7(2) |
| C1–O1–C5–C6 | −178.19(19) |
| C1–C2–C3–O5 | −177.53(19) |
| C1–C2–C3–C4 | −58.1(2) |
| C2–O6–C14–C15 | −75.7(3) |
| C2–C3–C4–O4 | 175.05(18) |
| C2–C3–C4–C5 | 55.7(2) |
| C3–C4–C5–O1 | −55.4(2) |
| C3–C4–C5–C6 | −175.89(19) |
| C4–O4–C7–C8 | 152.5(2) |
| C4–C5–C6–O3 | 55.1(3) |
| C5–O1–C1–O2 | 58.3(3) |
| C5–O1–C1–C2 | −59.9(2) |
| C7–O4–C4–C3 | 129.9(2) |
| C7–O4–C4–C5 | −109.3(2) |
| C7–C8–C9–C10 | −178.3(2) |
| C7–C8–C13–C12 | 179.2(2) |
| C8–C9–C10–C11 | −0.6(4) |
| C9–C8–C13–C12 | 0.3(4) |
| C9–C10–C11–C12 | −0.3(4) |
| C10–C11–C12–C13 | 1.3(4) |
| C11–C12–C13–C8 | −1.2(4) |
| C13–C8–C9–C10 | 0.7(4) |
| C14–O6–C2–C1 | 101.7(2) |
| C14–O6–C2–C3 | −136.8(2) |
| C14–C15–C16–C17 | 176.0(2) |
| C14–C15–C20–C19 | −177.0(2) |
| C15–C16–C17–C18 | 1.5(4) |
| C16–C15–C20–C19 | 0.8(4) |
| C16–C17–C18–C19 | −0.1(4) |
| C17–C18–C19–C20 | −0.9(4) |
| C18–C19–C20–C15 | 0.6(4) |
| C20–C15–C16–C17 | −1.8(4) |