Supplementary Material (ESI) for Organic and Biomolecular Chemistry

Biomimetically relevant self-condensations of C_5 units derived from lysine

A contribution from:

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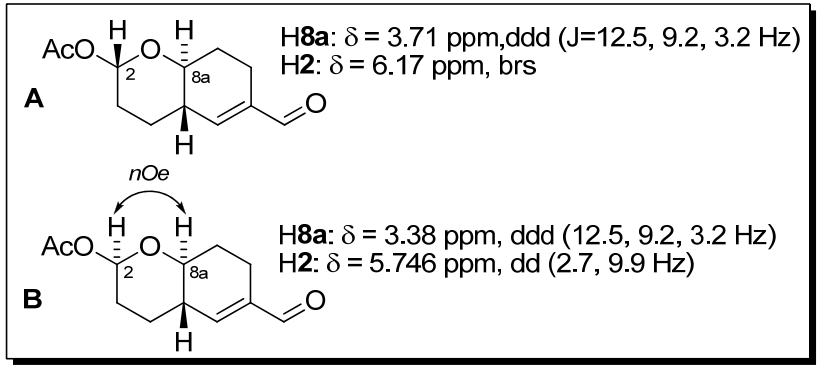
SUPPORTING INFORMATION

Acetylation of **6** into compounds **6A** and **6B:**



[A]:oil, δ_C (100 MHz; $CDCl_3$) 193.0 (HC=O), 169.1 ($CH_3\underline{CO}$), 150.3 (C-5), 140.9 (C-6), 91.7 (C2), 71.7 (C-8a), 40.5 (C-4a), 28.5, 27.2, 24.1, 20.9, 20.8 (CH_3).

[B]:crystals, δ_C (100 MHz; $CDCl_3$) 193.0 (HC=O), 168.9 ($CH_3\underline{CO}$), 149.4 (C-5), 140.9 (C-6), 94.2 (C2), 76.8 (C-8a), 40.2 (C-4a), 30.1, 27.3, 27.2, 20.9, 20.8 (CH_3).

Crystal structure determinations

Single colourless crystals of **8**, **10**, **18** *were recrystallised from cyclohexane, ethylic ether, cyclohexane/ethyl acetate (7:3) respectively, and glued on top of a thin silica rod and* X-ray data were collected on an Enraf-Nonius kappaCCD diffractometer at room temperature using graphite monochromatized Mo-Kα radiation (λ =0.7107 Å).

Each crystal was positioned at 31 mm from the CCD and the Bragg peaks were measured using a φ-and-ω-scan-strategy optimized by the *COLLECT* suite[1] once the cell parameters were derived by *DENZO (*HKL2000 suite)[2] from a preliminar10°- φ-scan. The counting time employed was 10 s (20 s for compound **10**) per degree of oscillation. Data reduction including a multiscan absorption correction was carried out using the Scalepack (HKL2000).[2] The structures were solved by direct methods and by subsequent difference Fourier syntheses and refined by full matrix least squares on F^2 using the SHELX-97 suite.[3] Anisotropic thermal parameters were used for all non-hydrogen atoms whereas hydrogen atoms, located from difference Fourier maps, were refined as a riding model with *U*iso = 1.2*U*eq of the parent atom (1.5 for the O–H hydrogen atoms).

Crystal data of compound **8**, C_{10} H_{12} O_3. Mr = 180.20. Monoclinic, space group *P* 2_1/c, *Z* = 4, *a* = 6.627(1), *b* = 12.282(3), *c* = 10.388(2) Å, β = 96.38(5)°, *V* = 908.7(3)Å^3, ρ(calc)=1.317g.cm^{-3}, μ=0.097mm^{-1}. 13337 reflections were collected and subsequently merged to 1646 unique reflections with an R_{int} of 0.0189. The final refinement of 119 parameters converged to final *R* and w*R* indices *R*1 = 0.073 and w*R*2 = 0.197 for 1166 reflections with $I > 2\sigma(I)$ and *R*1 = 0.096 and w*R*2 = 0.219 for all *hkl* data.

Crystal data of compound **10**. C_{10} H_{16} O_4. Mr = 200.23. Triclinic, space group *P* -1, *Z* = 2, *a* = 6.280(3), *b* = 7.095(3), *c* = 11.413(5) Å , α = 97.531(5), β = 102.847(8), γ = 101.348(6)°, *V* = 477.9(4)Å^3, ρ(calc)=1.391g.cm^{-3}, μ=0.107mm^{-1}. 2432 reflections were collected and subsequently merged to 1098 unique reflections with an R_{int} of 0.0410. The final refinement of 128 parameters converged to final *R* and w*R* indices *R*1 = 0.073 and w*R*2 = 0.184 for 817 reflections with $I > 2\sigma(I)$ and *R*1 = 0.096 and w*R*2 = 0.205 for all *hkl* data.

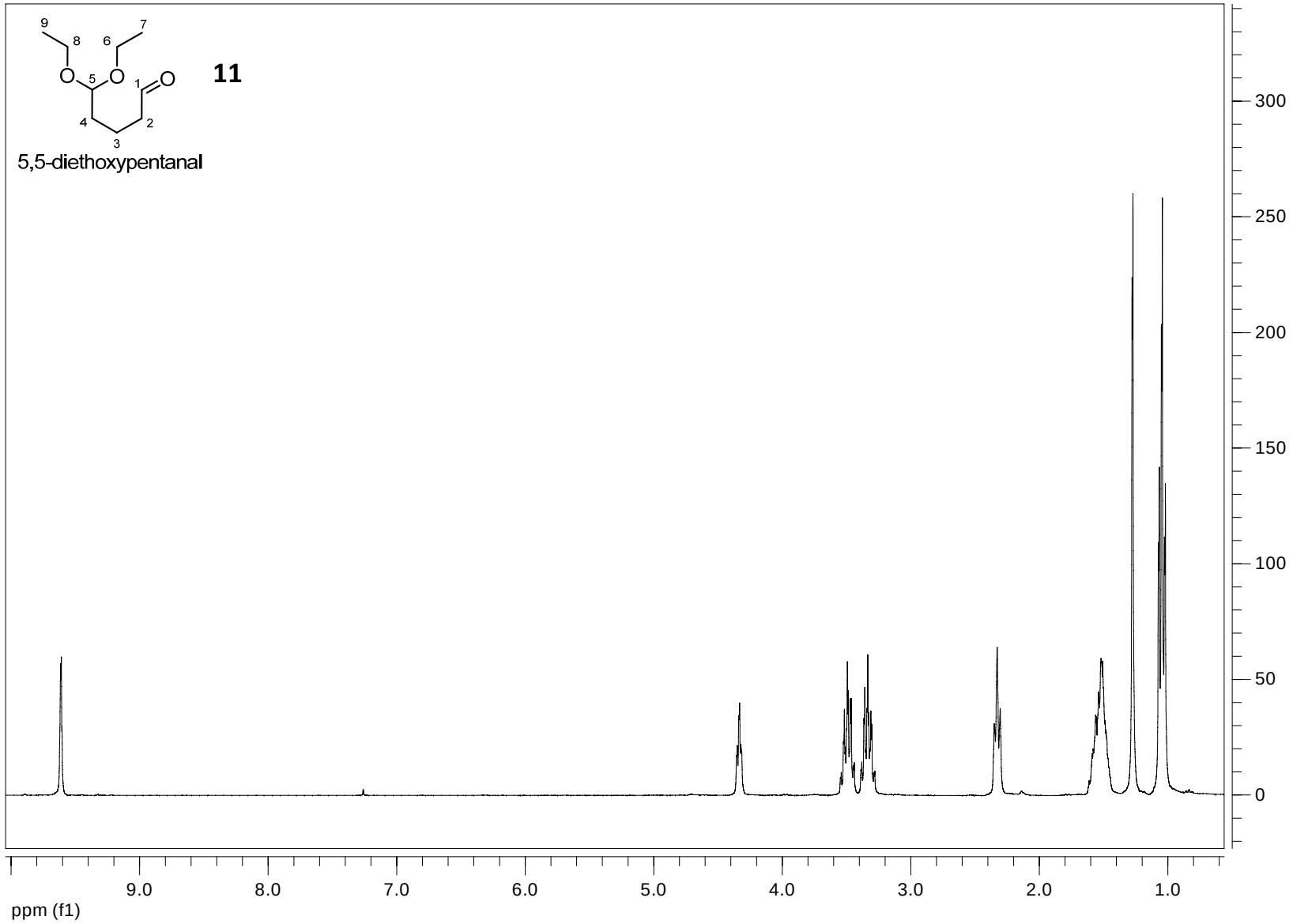
Crystal data of compound **18**. C_{10} H_{12} O_2. Mr = 164.20. Monoclinic, space group *P* 2_1/n, *Z* = 4, *a* = 7.160(4), *b* = 13.956(5), *c* = 8.879(4) Å, β = 101.312(5)°, *V* = 870.0(7)Å^3, ρ(calc)=1.254g.cm^{-3}, μ=0.086mm^{-1}. 11473 reflections were collected and subsequently merged to 1701 unique reflections with an R_{int} of 0.0236. The final refinement of 109 parameters converged to final *R* and w*R* indices *R*1 = 0.047 and w*R*2 = 0.114 for 1204 reflections with $I > 2\sigma(I)$ and *R*1 = 0.071 and w*R*2 = 0.129 for all *hkl* data.

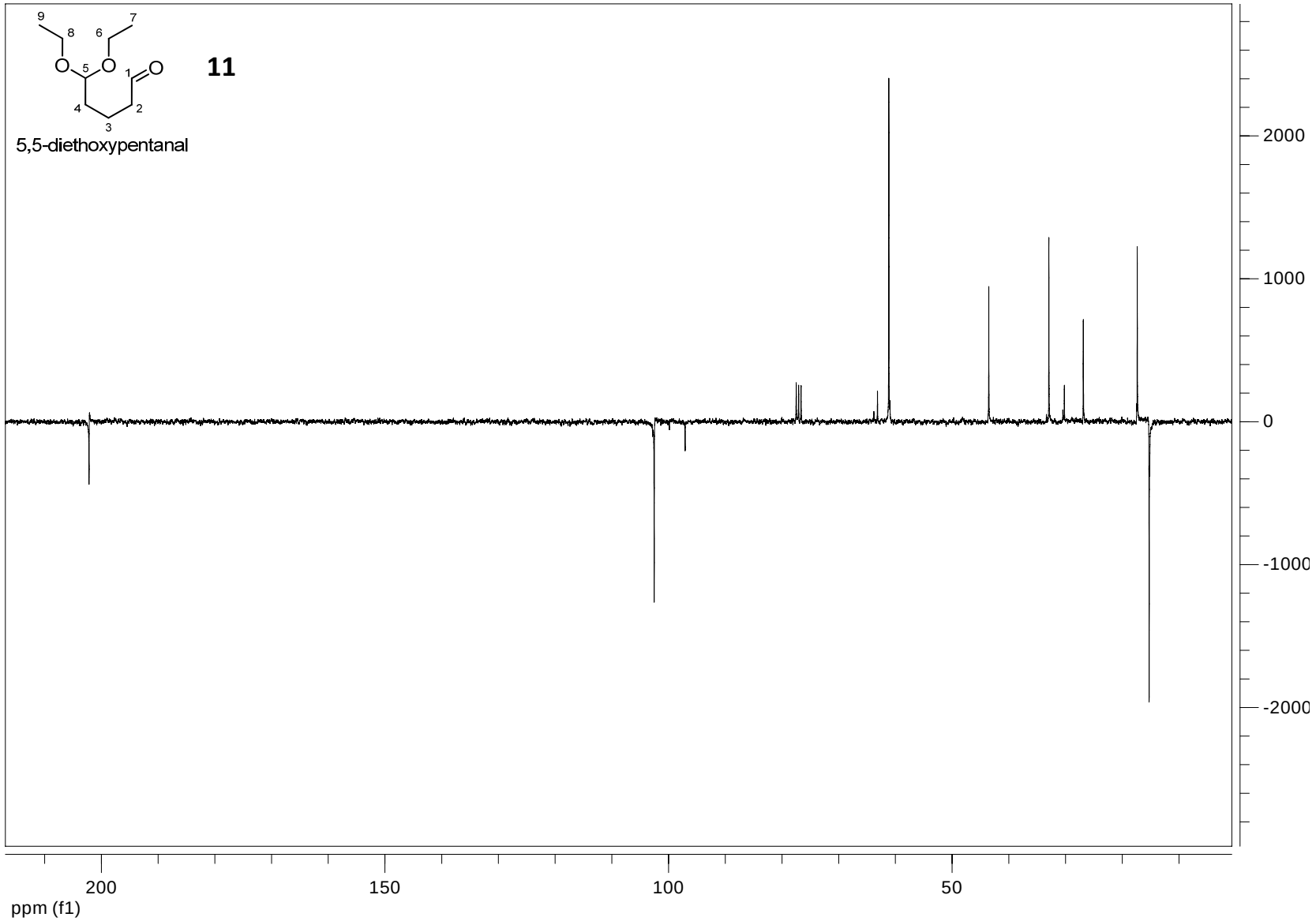
CCDC 753047 (compound **8**), 753048 (compound **10**), and 753049 (compound **18**) contain the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

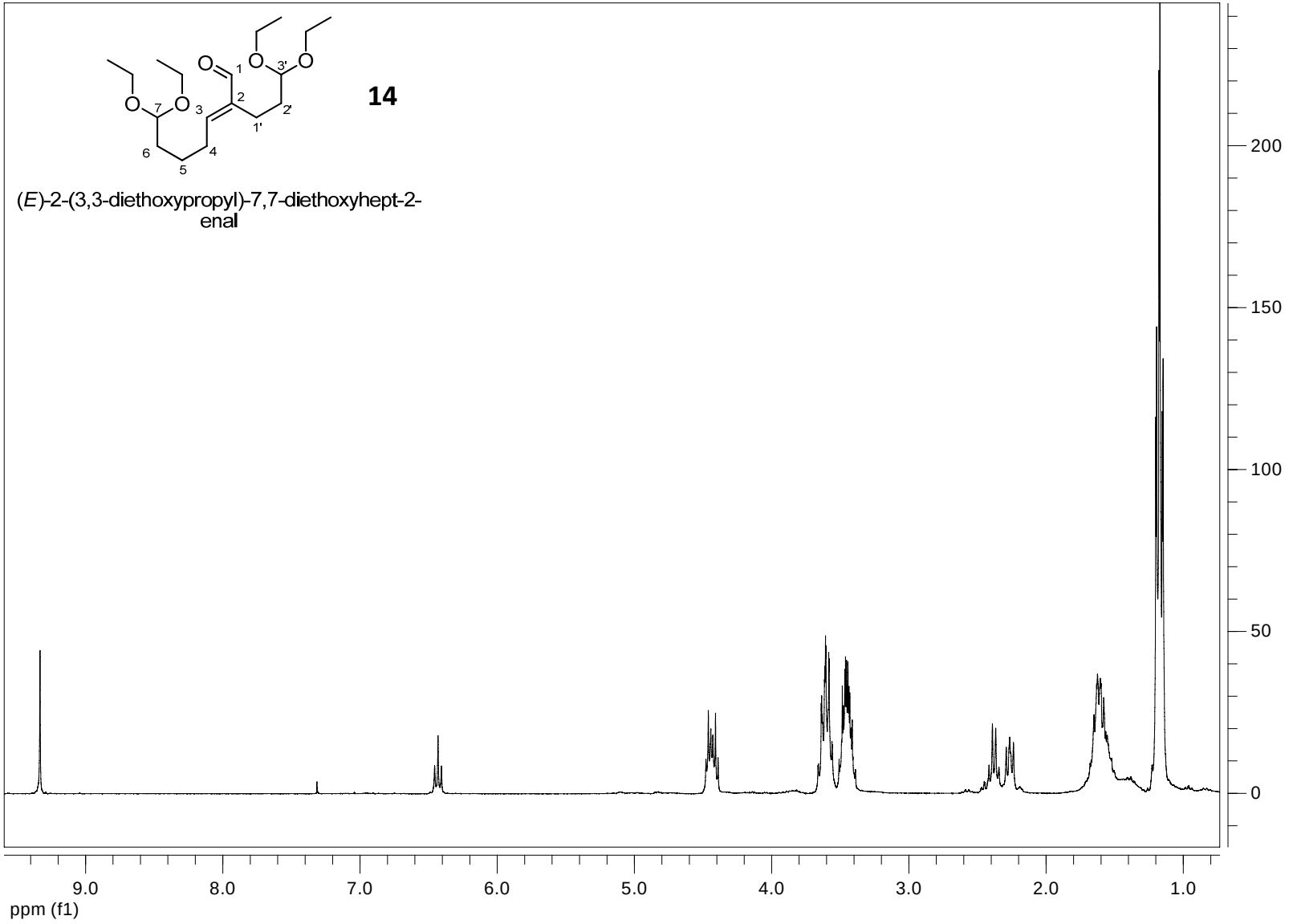
1. Nonius, B. V. *COLLECT*, data collection software, 1999.

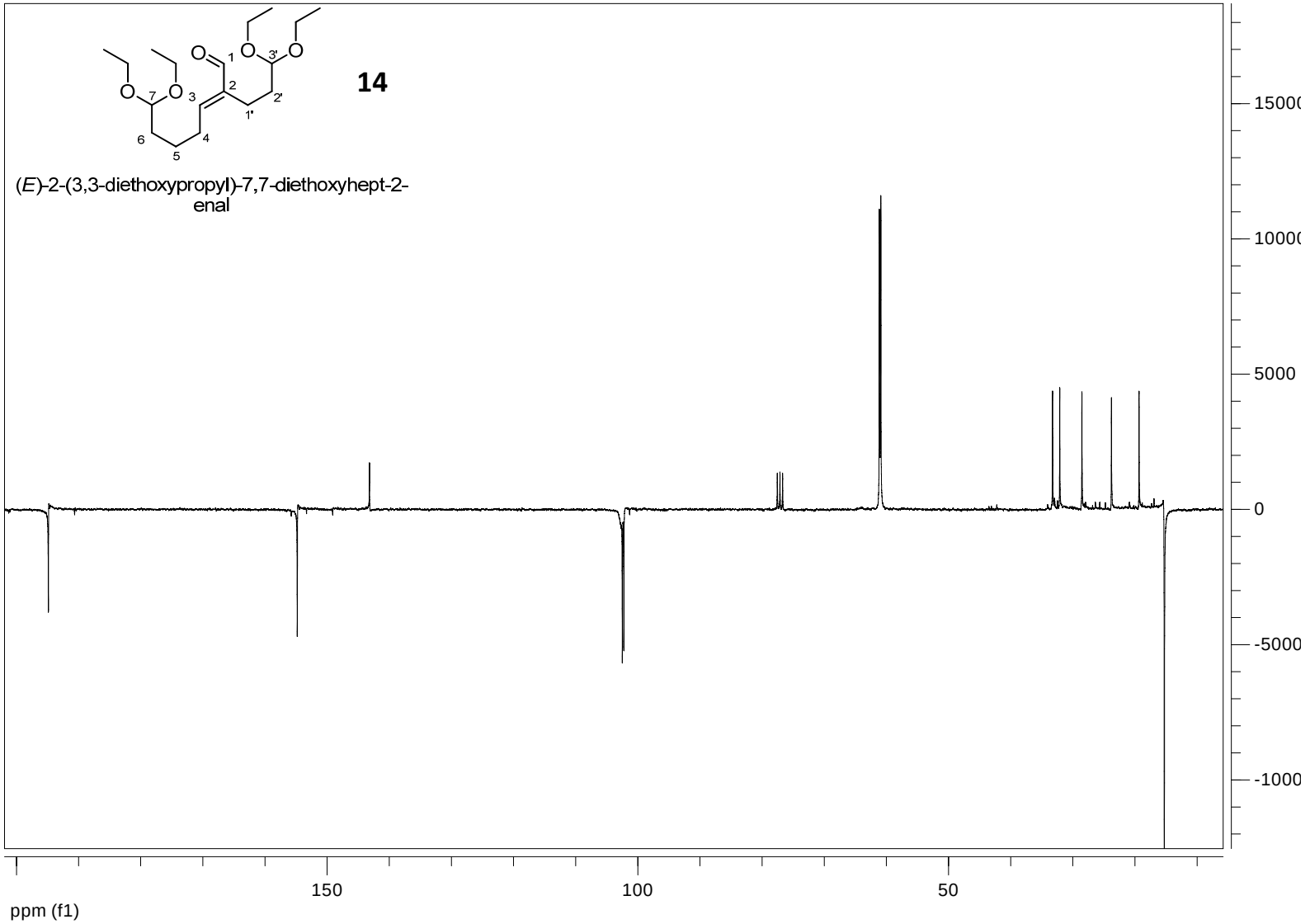
2. Z.Otwinowski, & W. Minor, Methods in Enzymology, Macromolecular Crystallography, part A, edited by C.W. Carter, Jr. & R.M. Sweet, New York: Academic Press, 1997, **276**, 307.

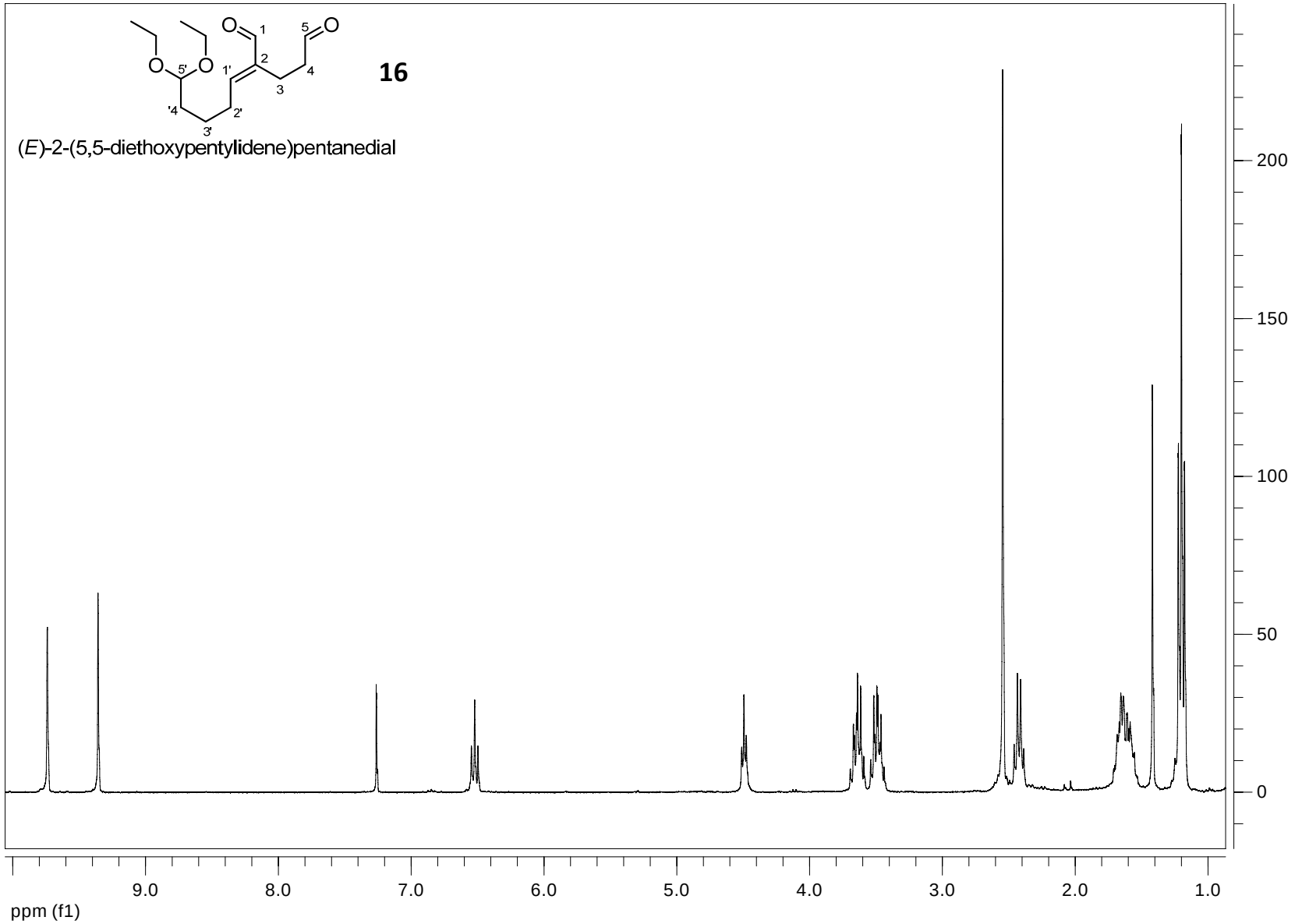
3. G. M. Sheldrick, *Acta Crystallogr., Sect. A: Found. Crystallogr.*, 2008, **64**, 112.

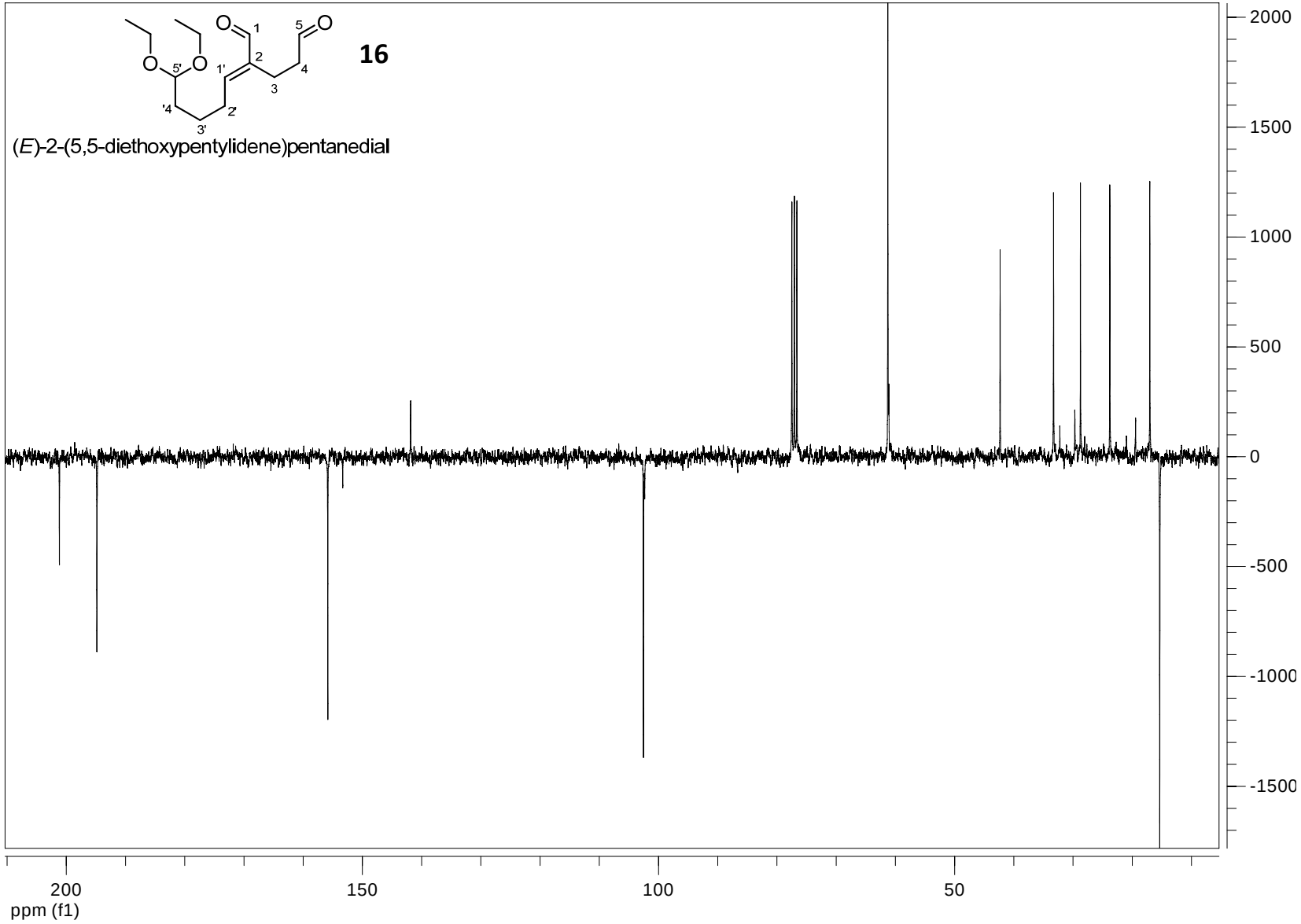


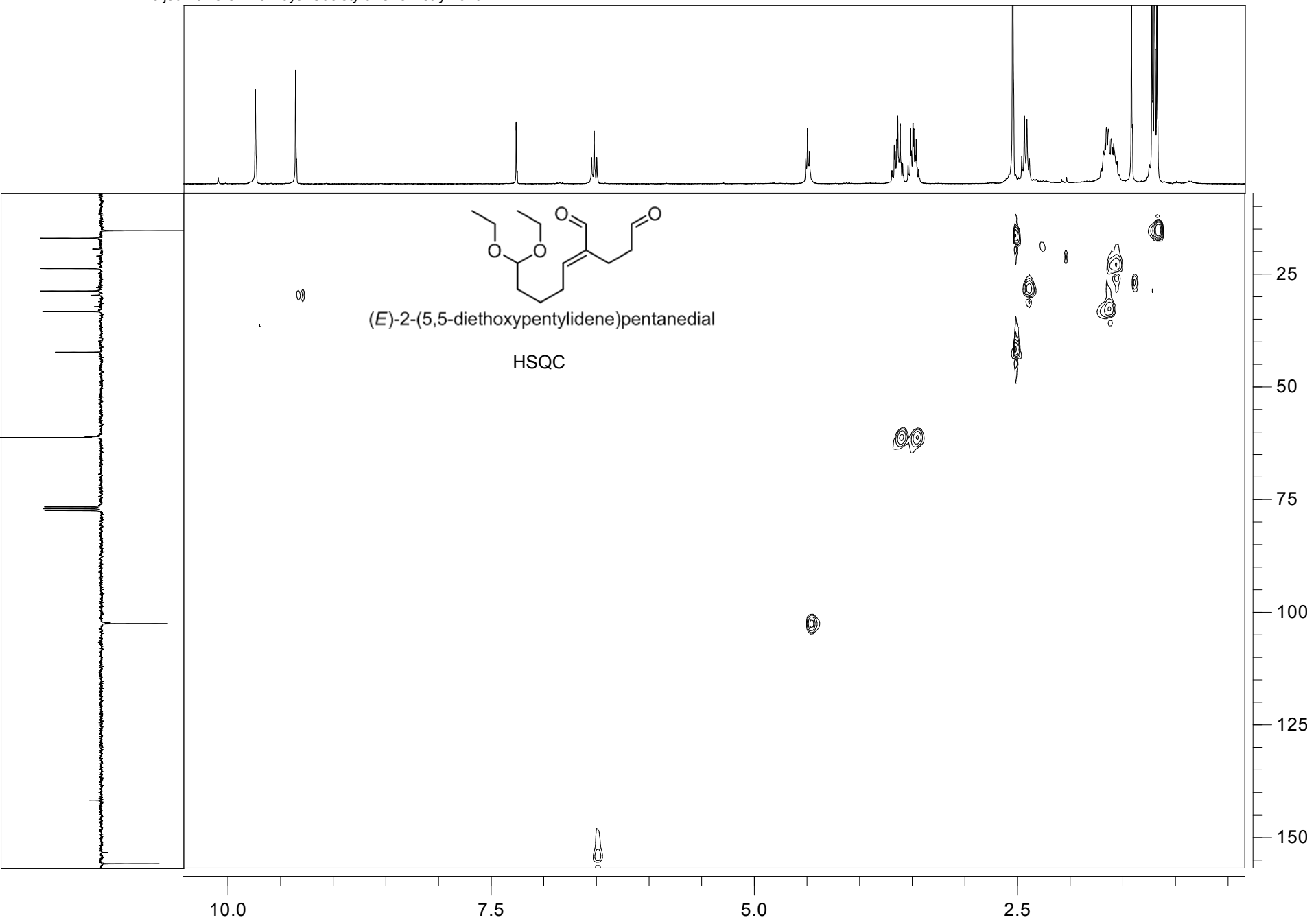

9
8
6
7
O
5
O
1
O
4
2
3
11
5,5-diethoxypentanal
2000
1000
0
-1000
-2000
200
150
100
50
ppm (f1)

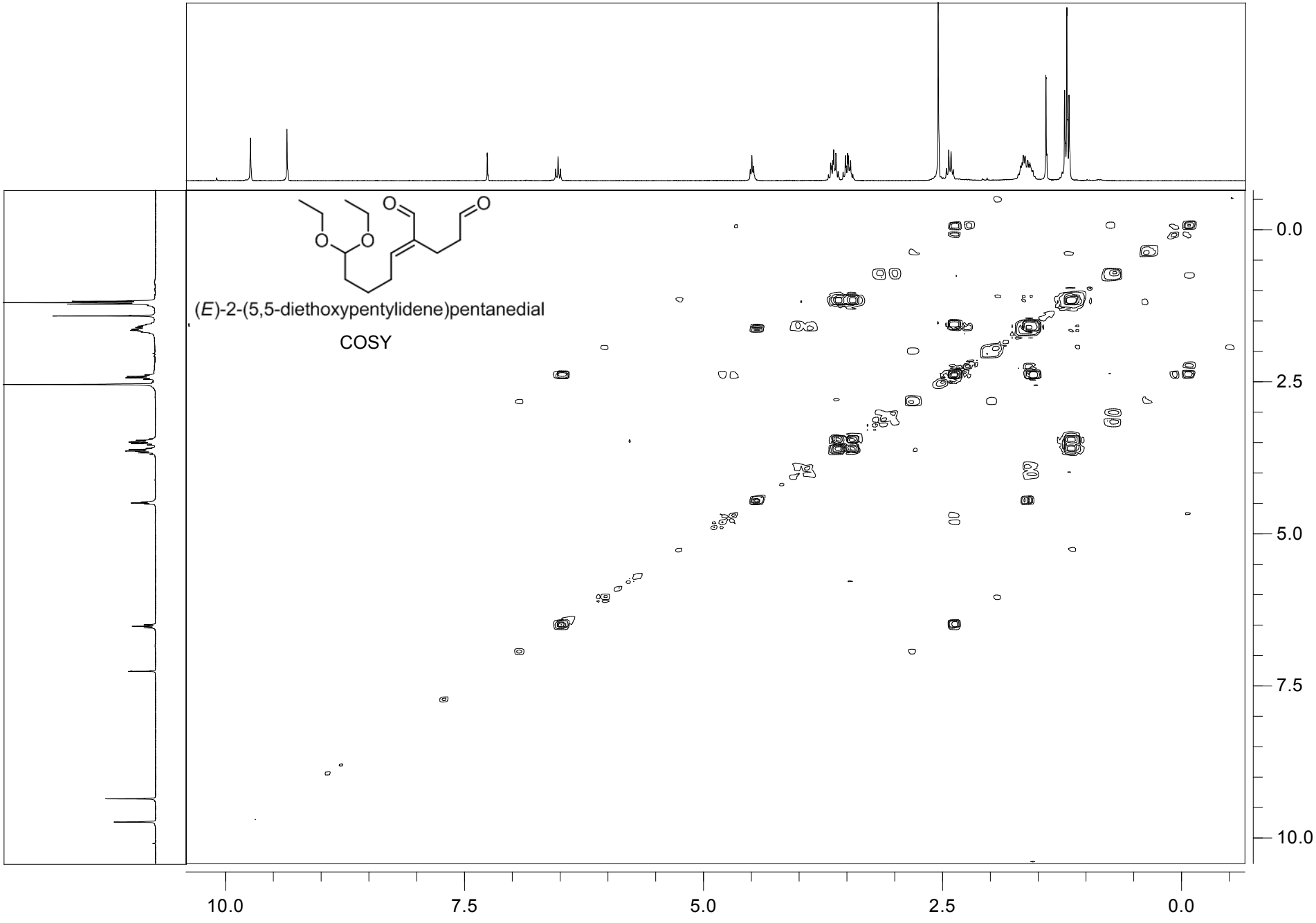


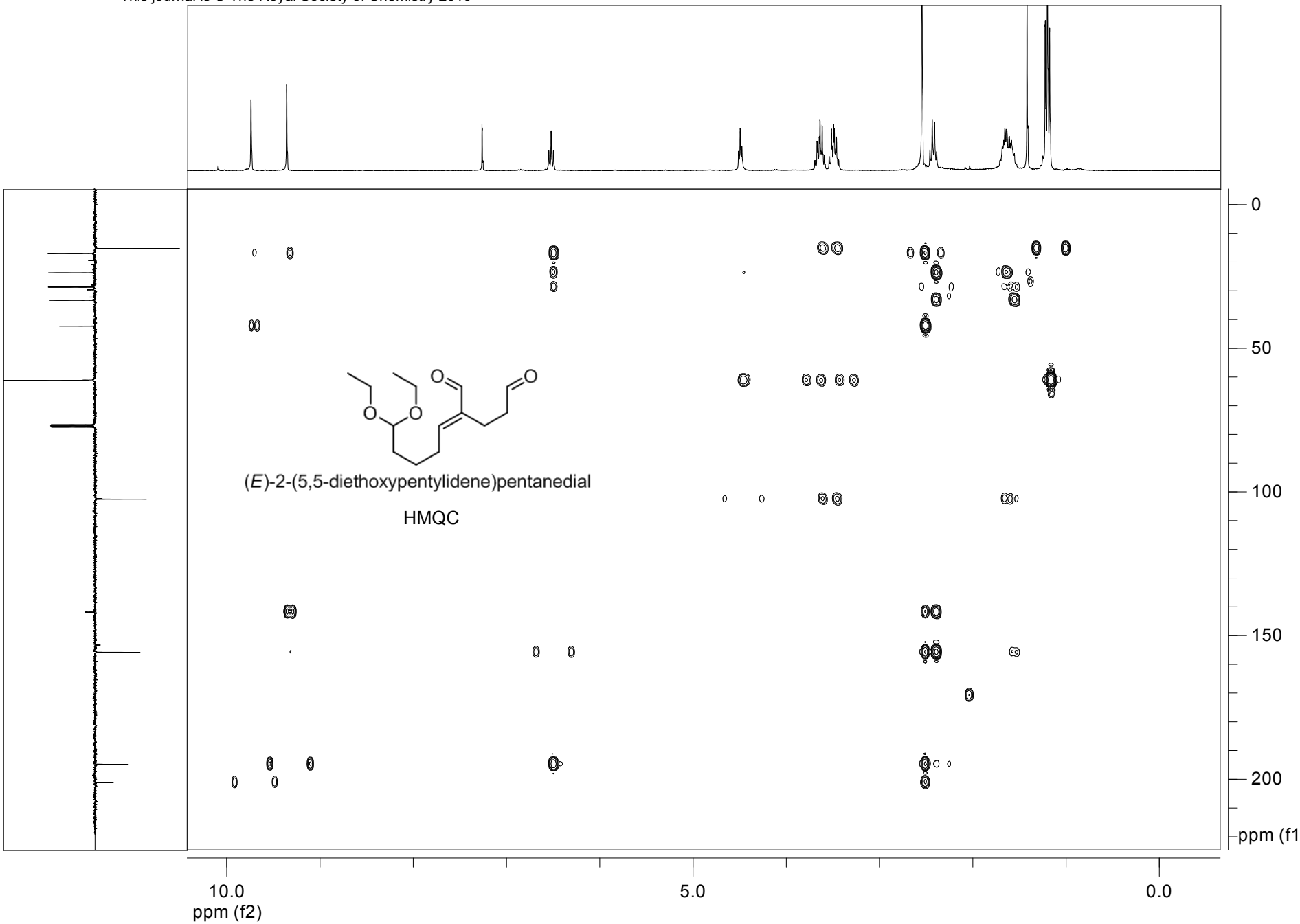


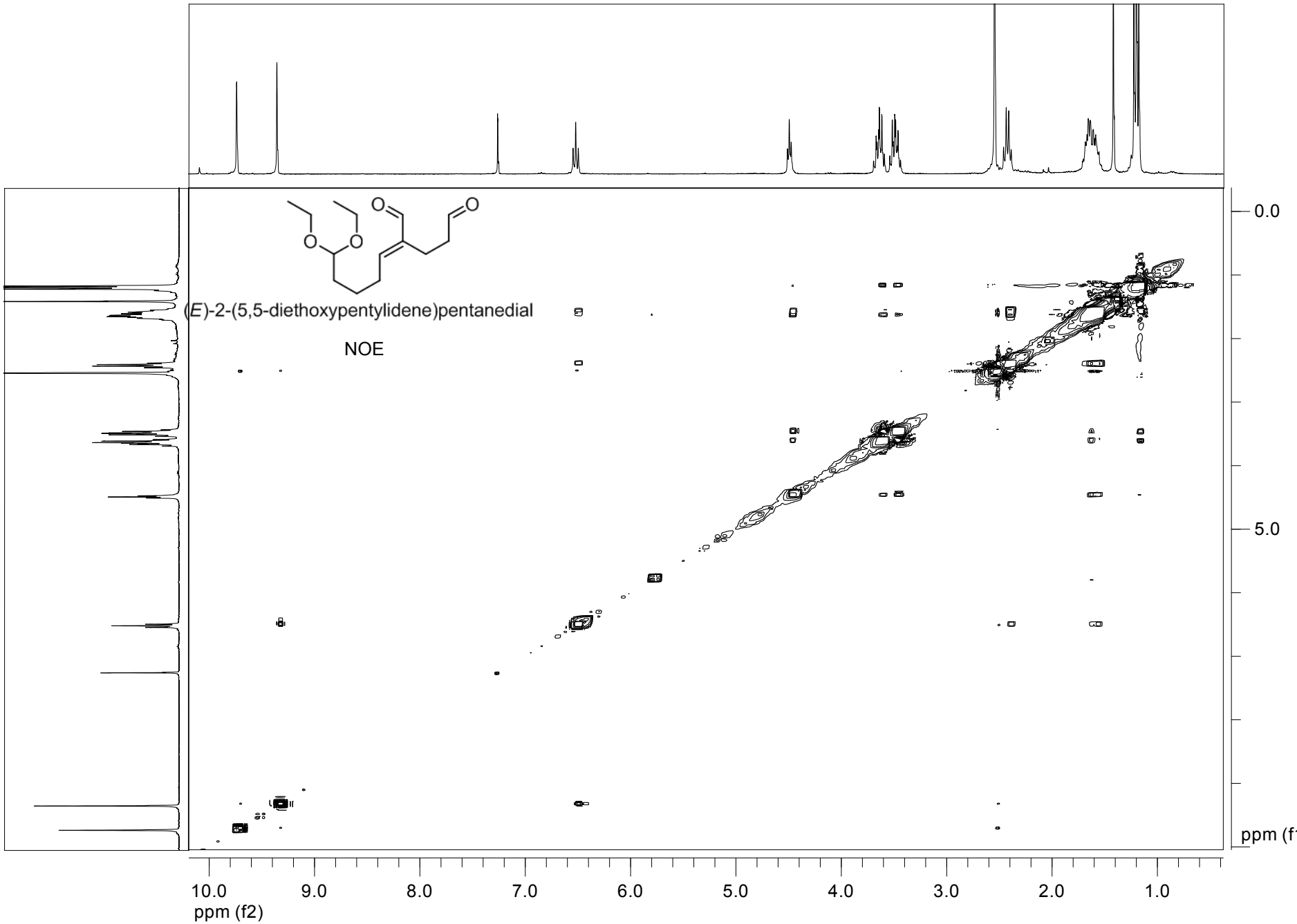


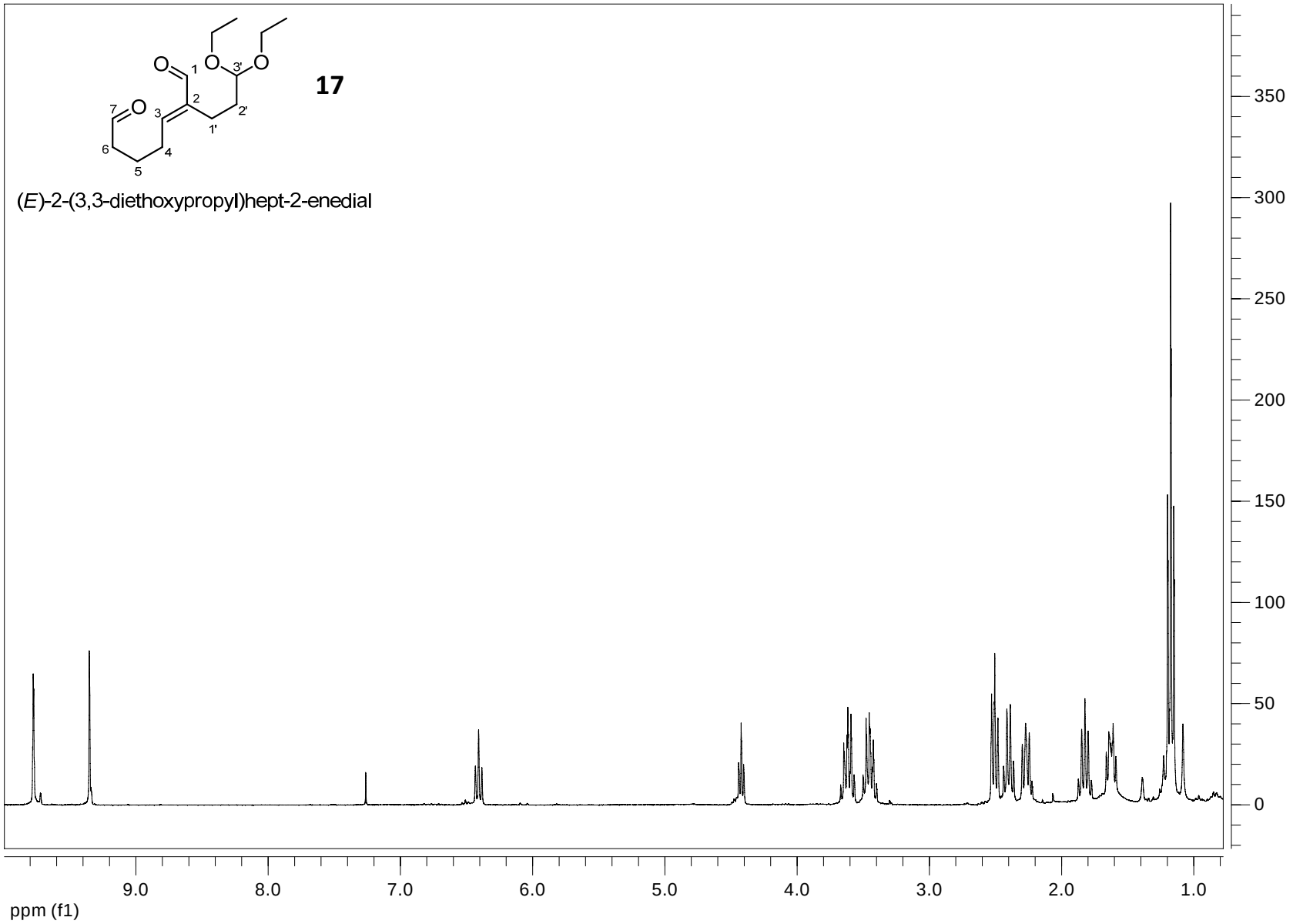


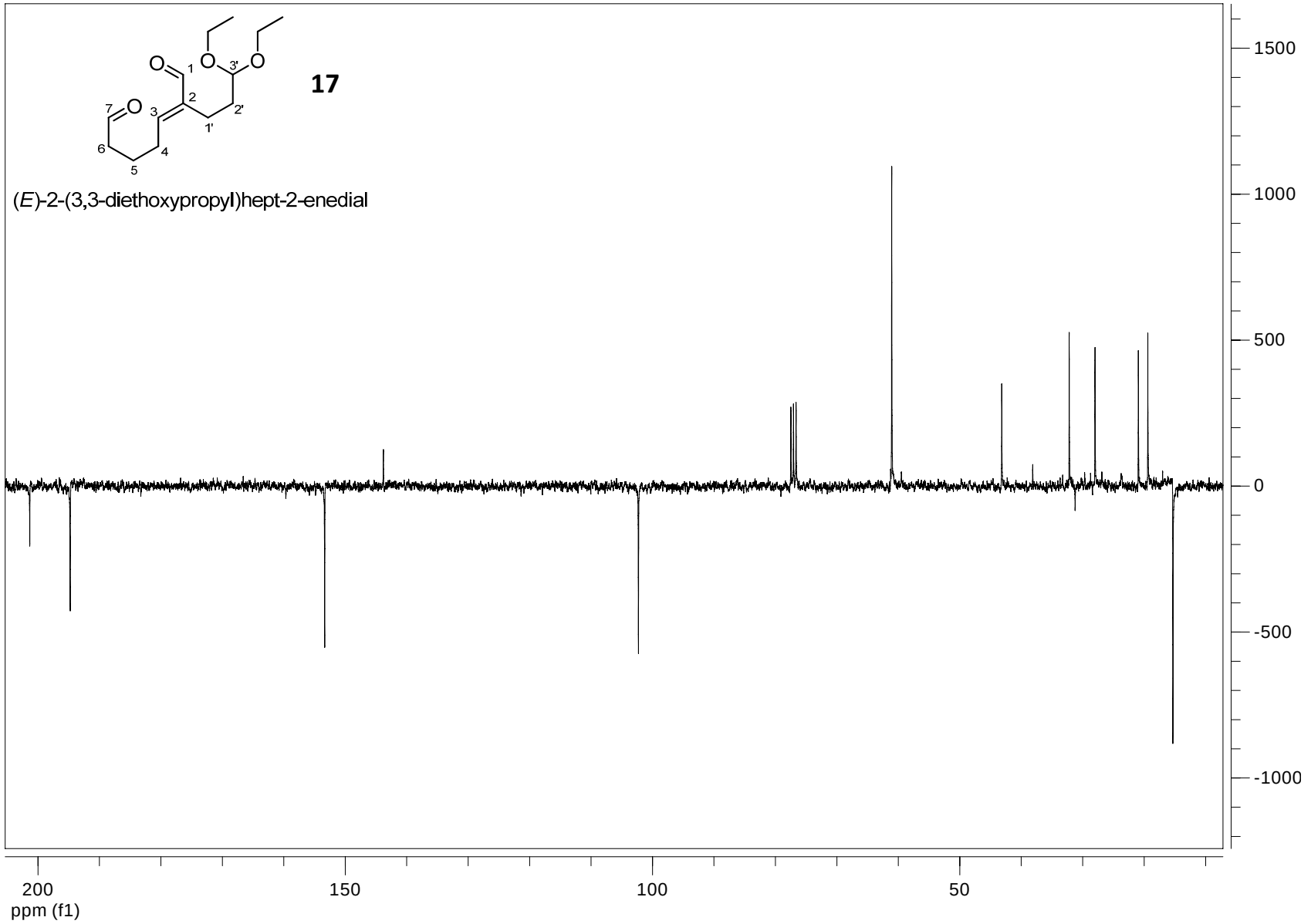


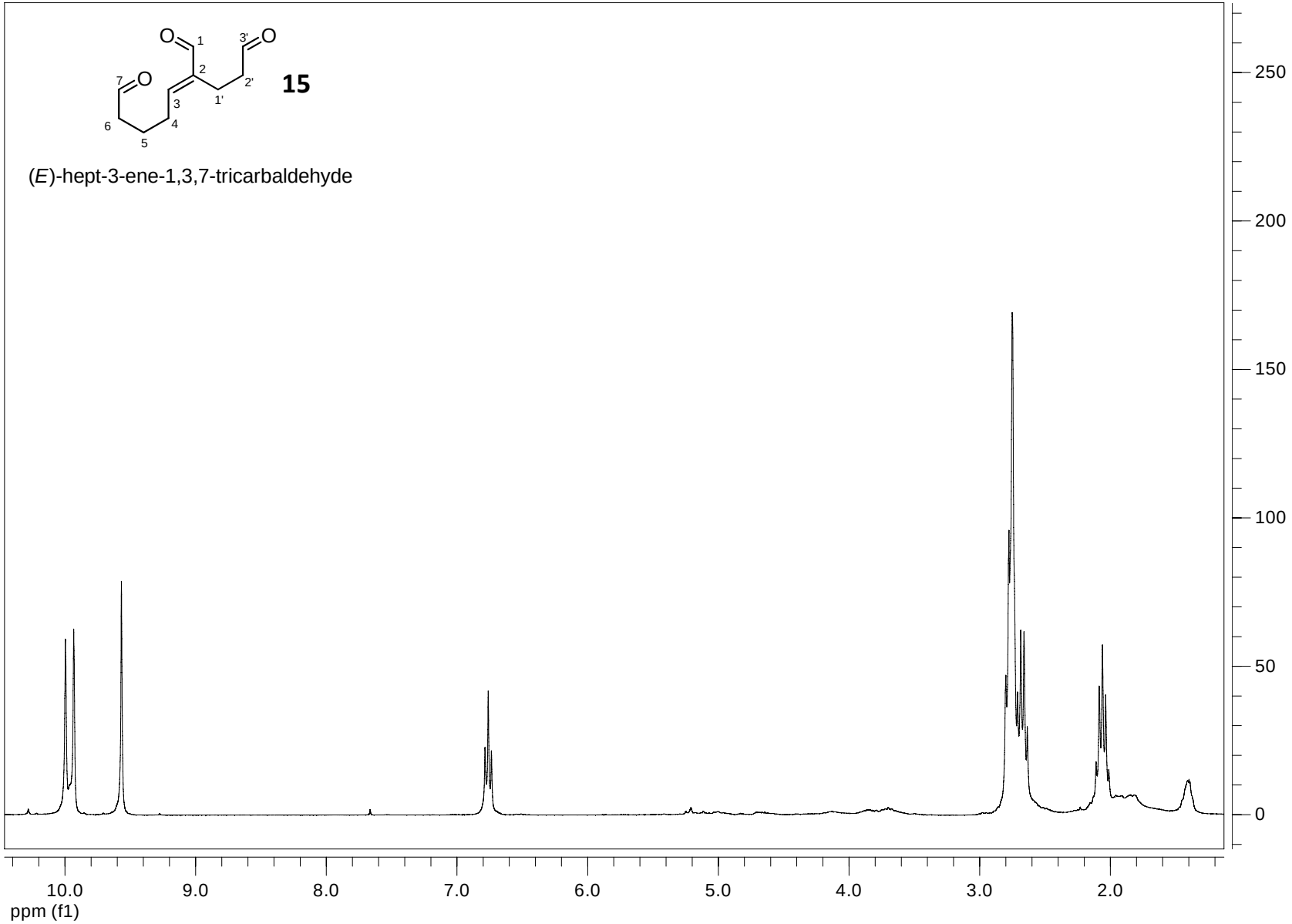


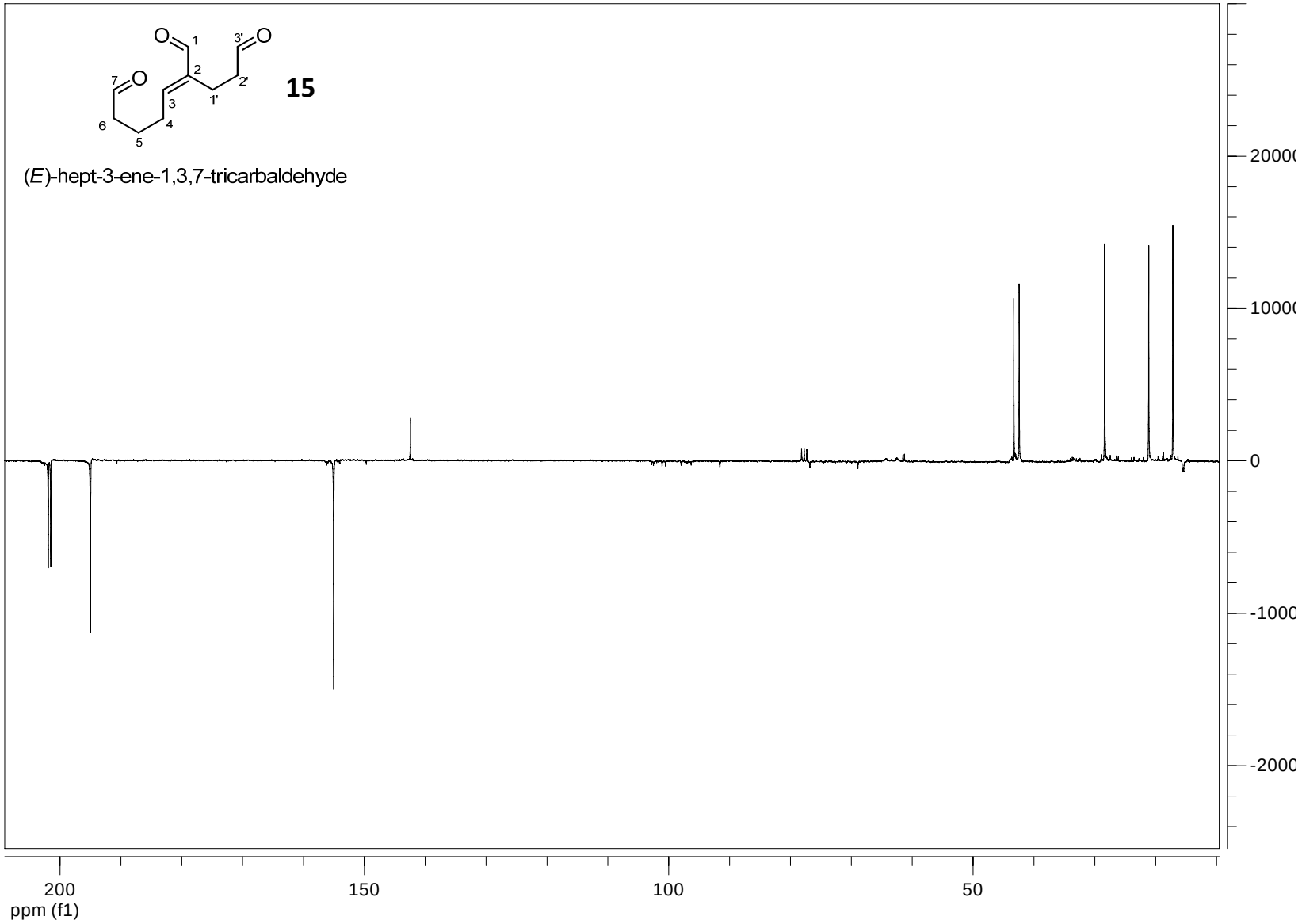


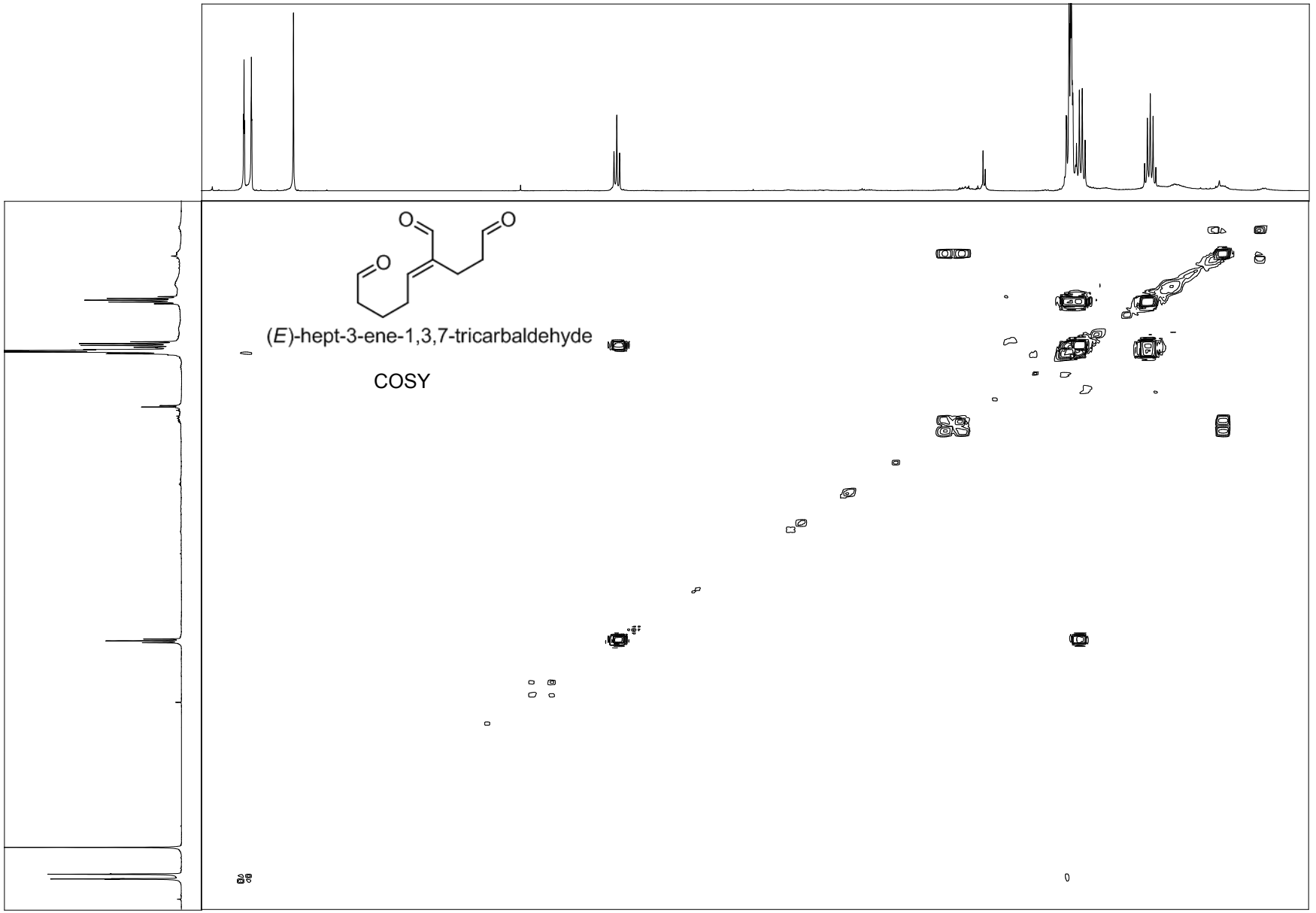


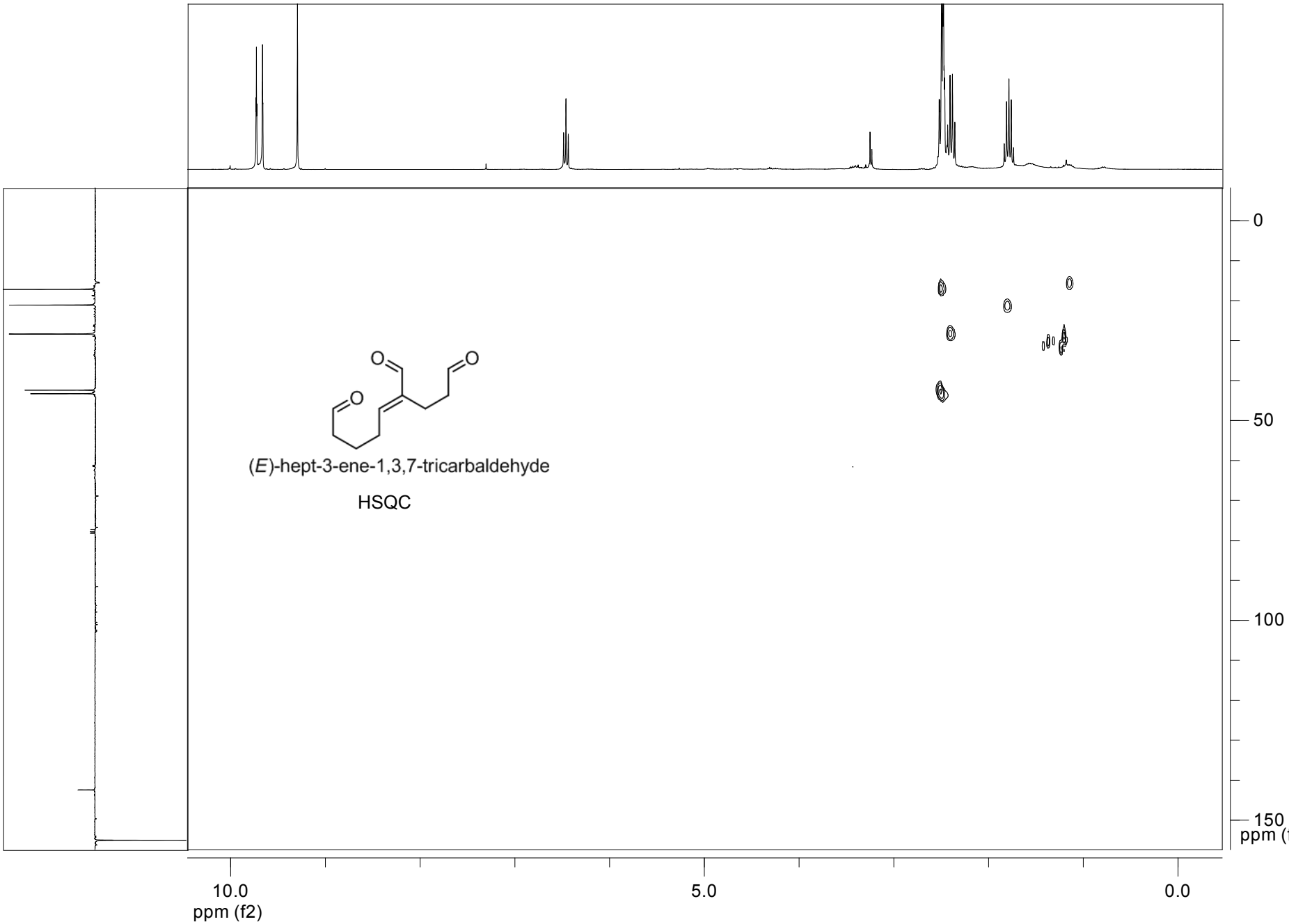


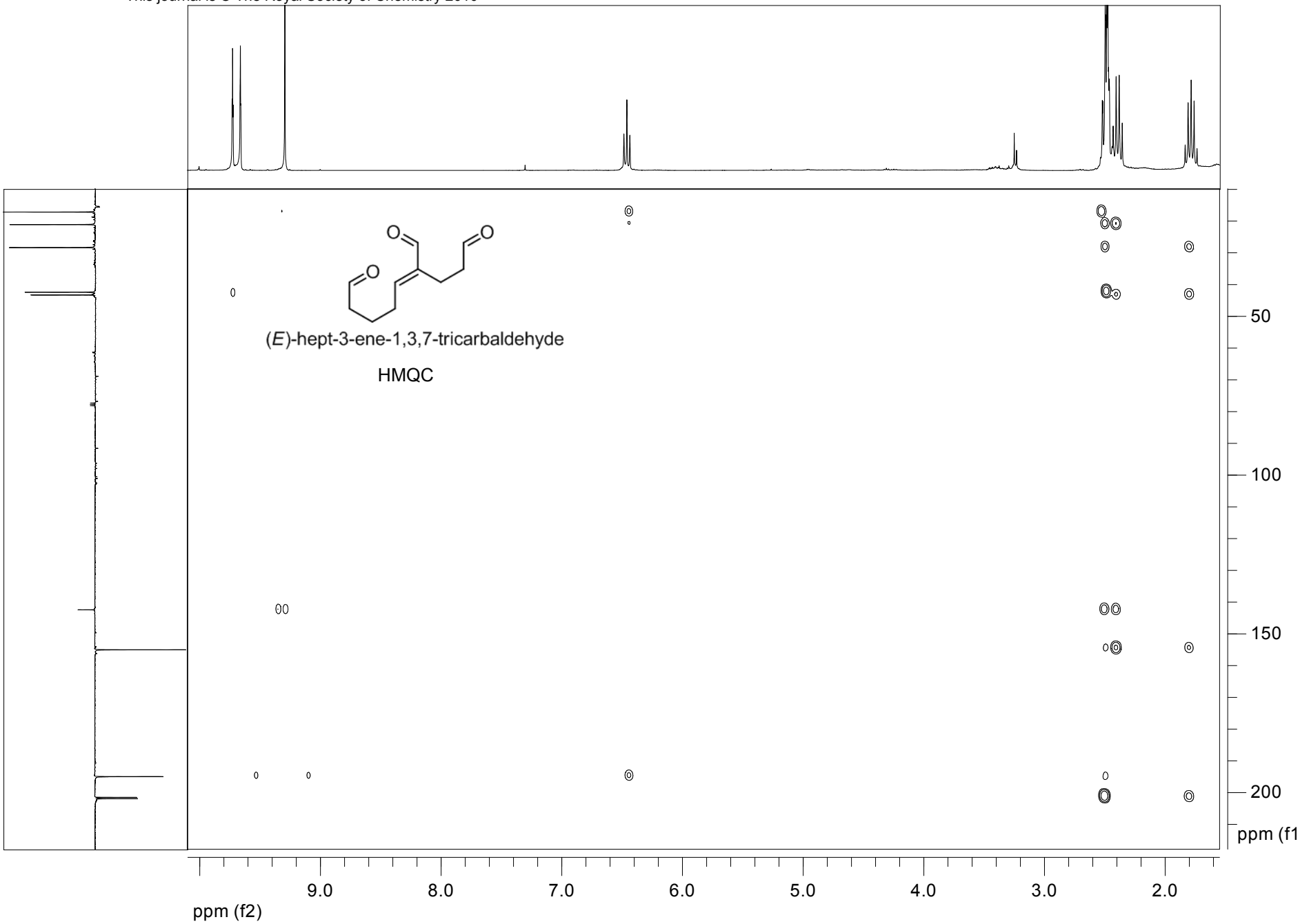


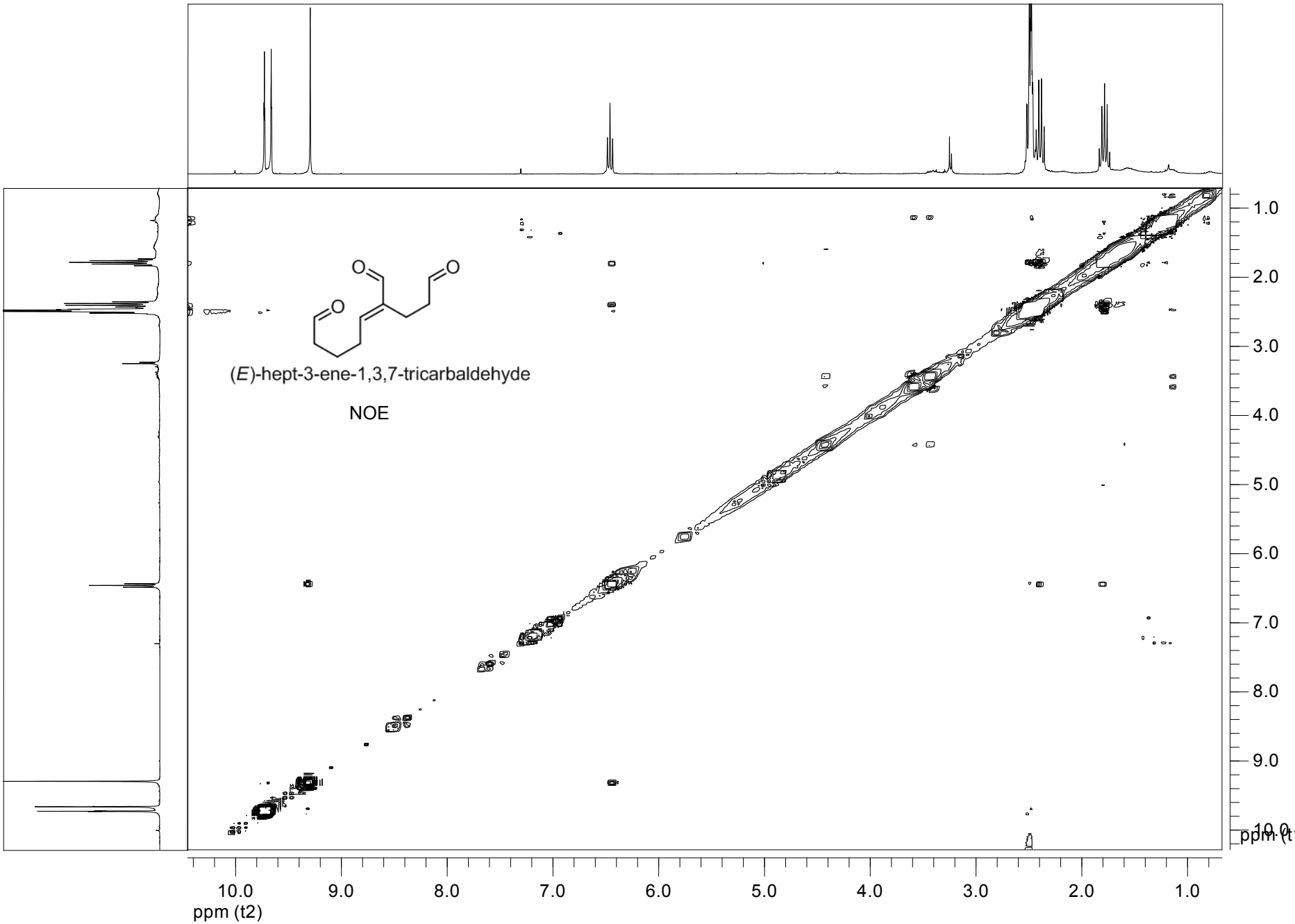


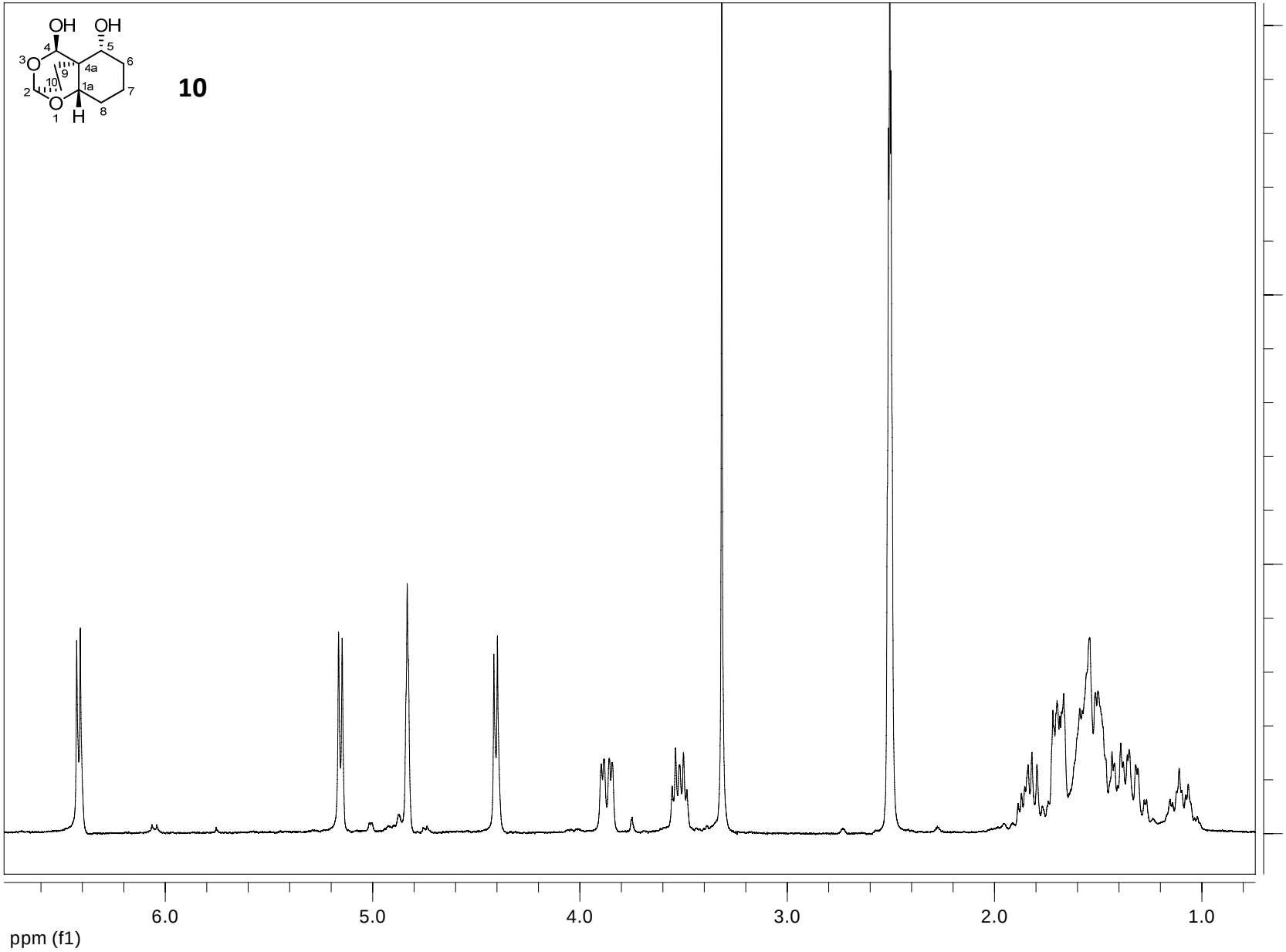


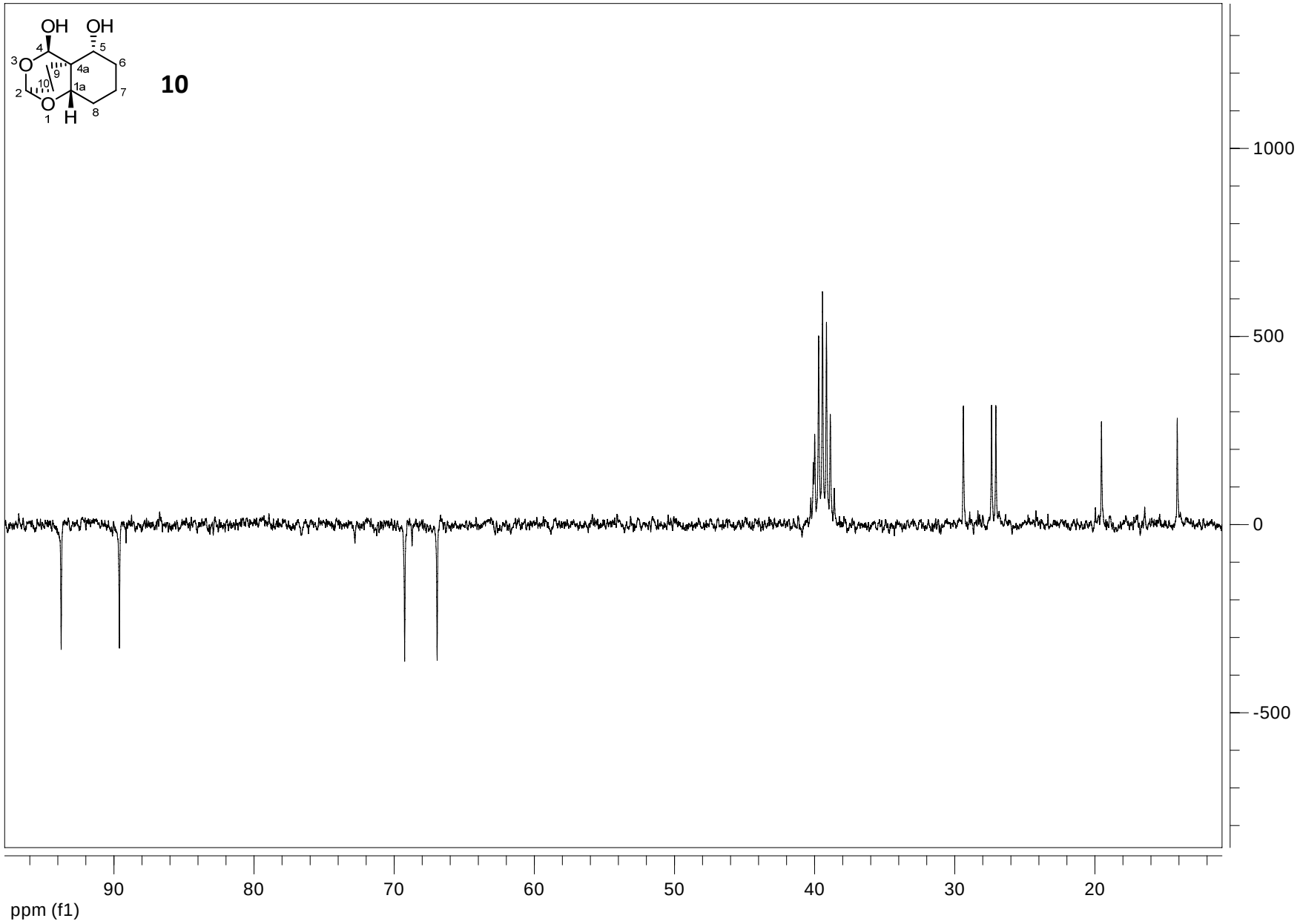


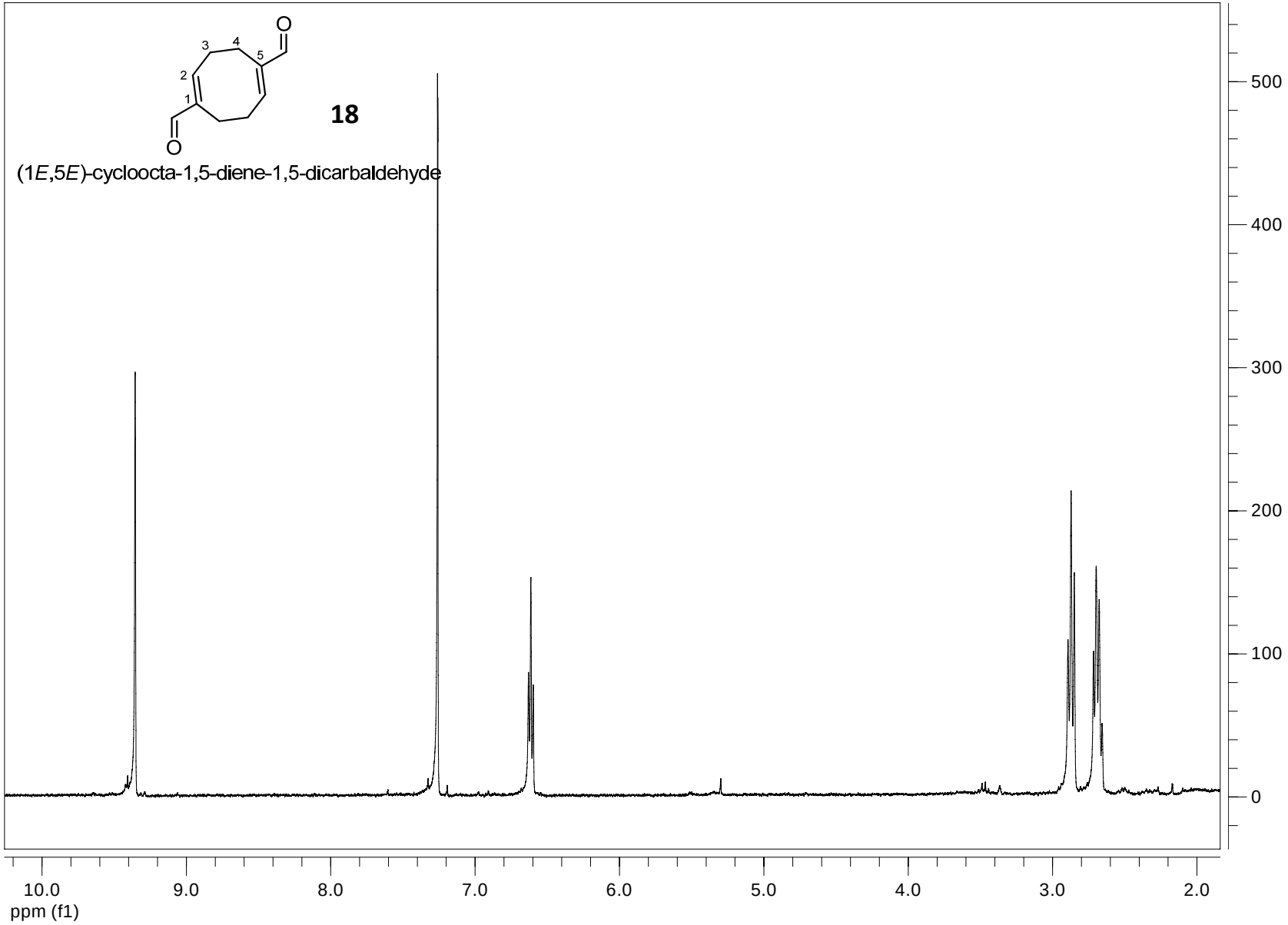


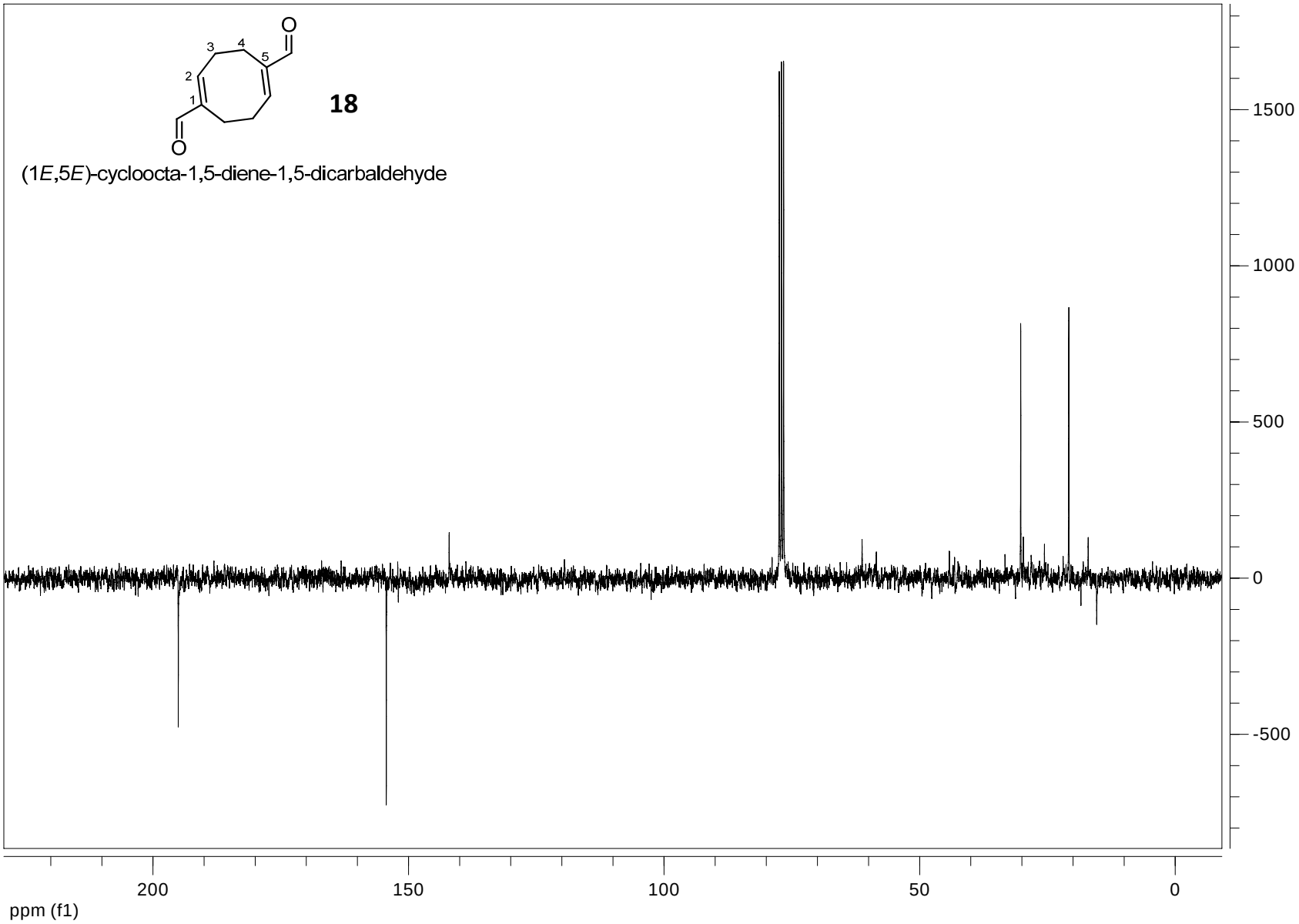


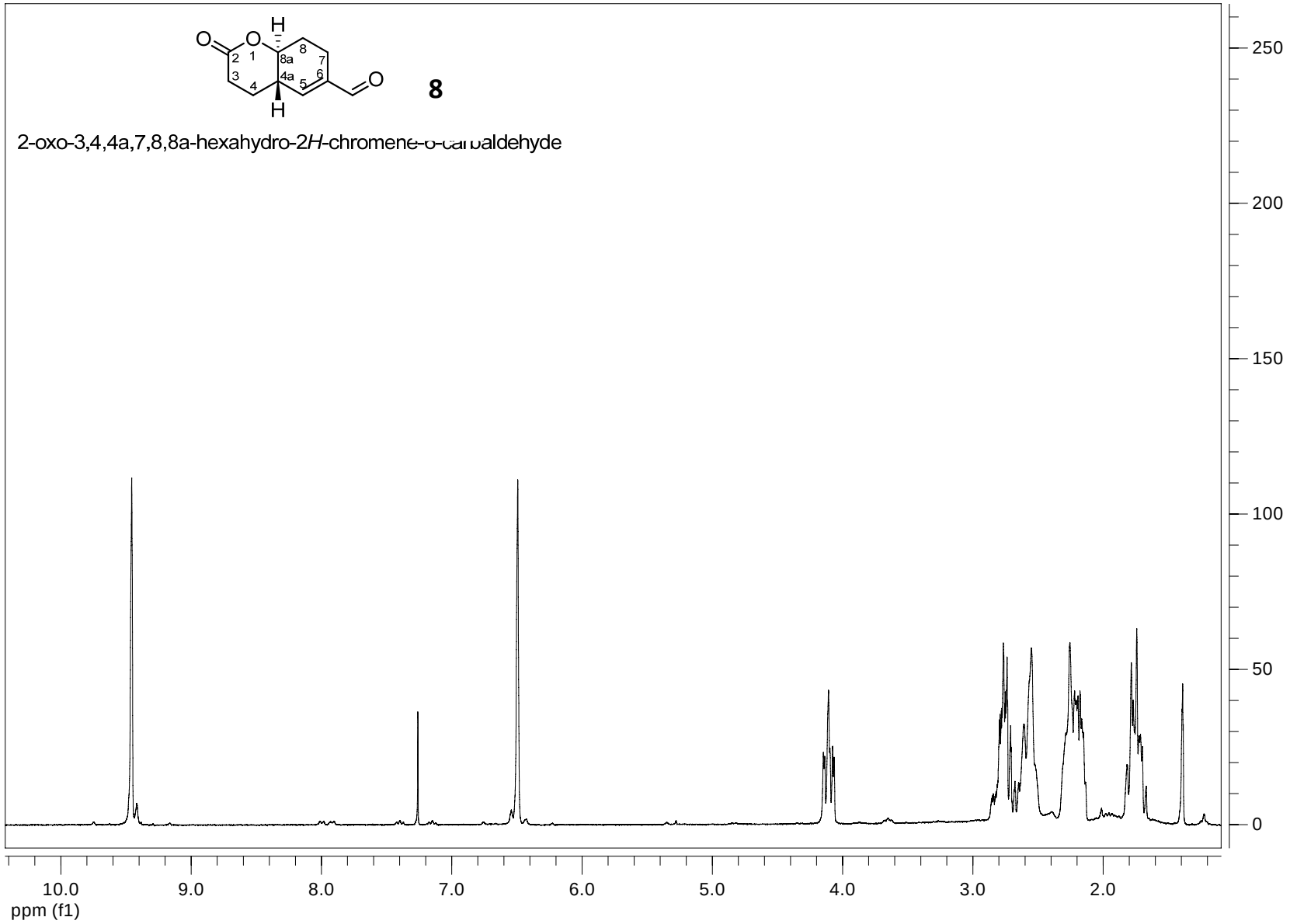





OH
OH
4
5
3
O
6
4a
9
10
1a
2
7
O
H
1
8
10
1000
500
0
-500
90
80
70
60
50
40
30
20
ppm (f1)






8
2-oxo-3,4,4a,7,8,8a-hexahydro-2H-chromene-6-carbaldehyde
250
200
150
100
50
0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
ppm (f1)