

The synthesis of α,β -unsaturated $18\alpha\text{H},19\beta\text{H}$ -ursane methyl ketones

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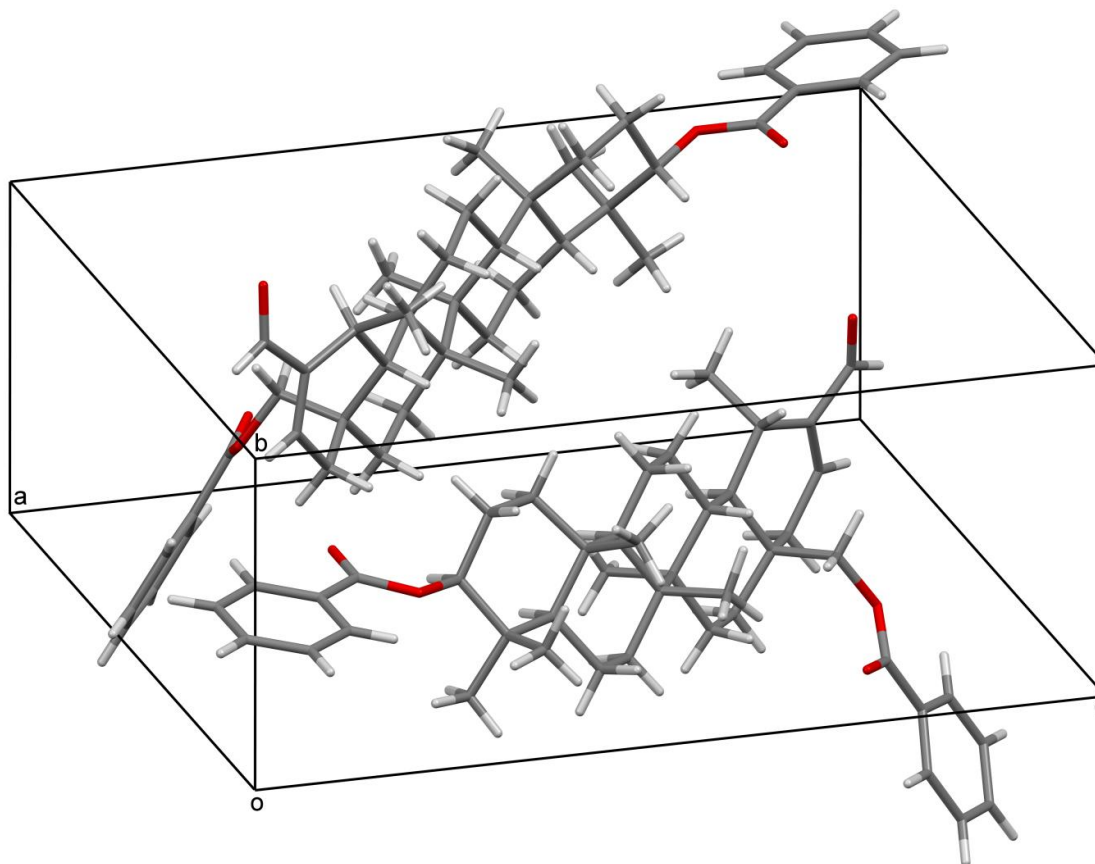
1. X-ray studies of 3 β ,28-dibenzoyloxy-18 α ,19 β H-ursa-20(21)-ene-29-al (4)

Figure S1. Packing of compound 3 β ,28-dibenzoyloxy-18 α ,19 β H-ursa-20(21)-ene-29-al (**4**) in crystalline lattice (CCDC 1914625).

Crystal structure determination

The unit cell parameters and the X-ray diffraction intensities were measured on the Xcalibur Ruby diffractometer. The empirical absorption correction was introduced by the multi-scan method using the SCALE3 ABSPACK algorithm¹. Using the OLEX2², the structure was solved with the Superflip program³ and refined by the full-matrix least-squares method in the anisotropic approximation for all non-hydrogen atoms using the SHELXL⁴ program package. Positions of the hydrogen atoms were calculated from the Fourier synthesis of the electron density and refined using the “riding” model.

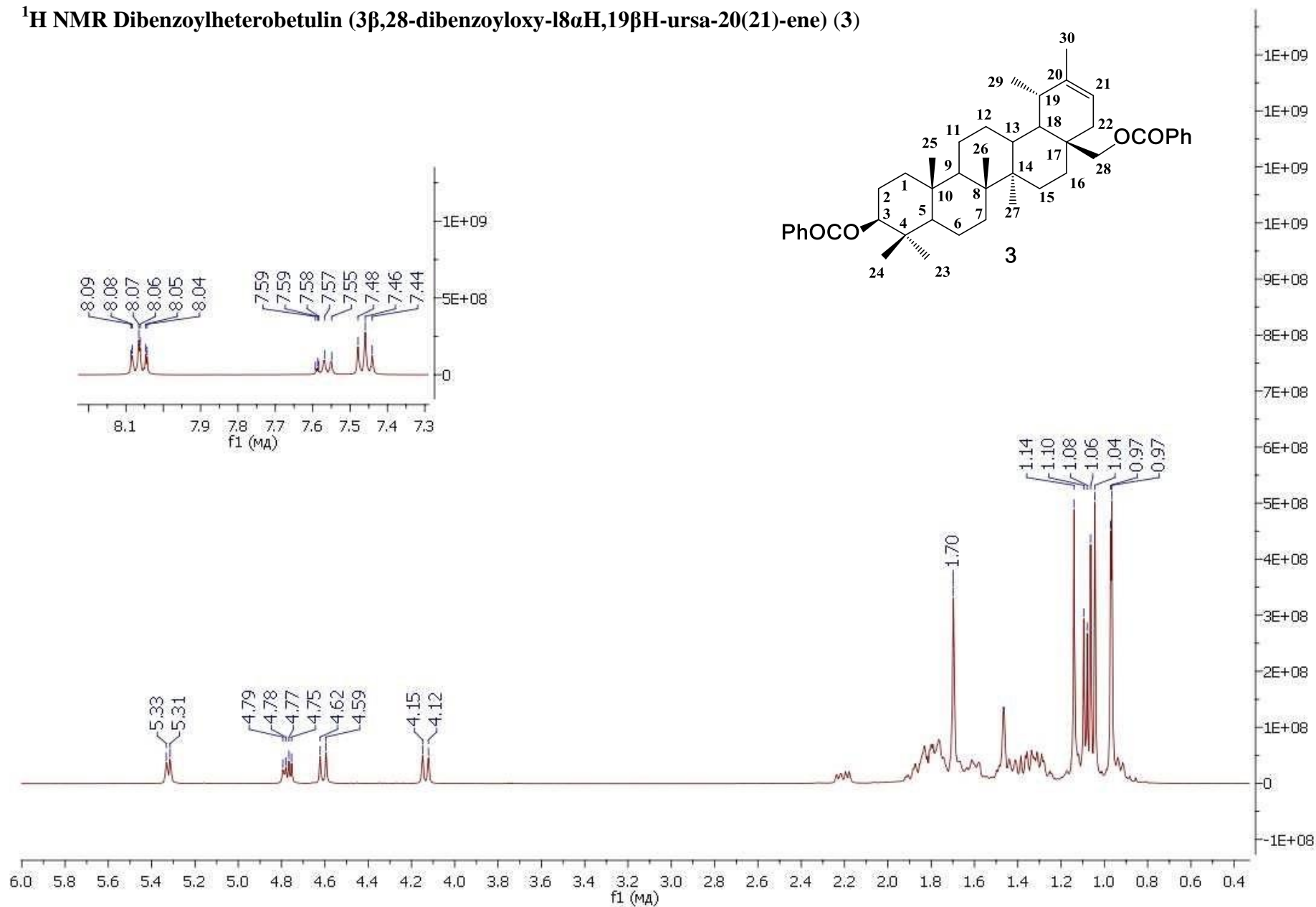
Crystal Data of 3 β ,28-dibenzoyloxy-18 α ,19 β H-ursa-20(21)-ene-29-al (4**).** C₄₄H₅₆O₅, *M* = 664.88, monoclinic, *a* = 13.786(3) Å, *b* = 7.5936(16) Å, *c* = 18.038(4) Å, β = 97.41(2)°, *V* = 1872.6(7) Å³, *T* = 295(2), space group *P*2₁, *Z* = 2, μ (Mo K α) = 0.075 mm⁻¹. The final refinement parameters: *R*₁ = 0.0635, *wR*₂ = 0.1442 [for observed 6068 reflections with *I* > 2 σ (*I*)]; *R*₁ = 0.0937, *wR*₂ = 0.1686 (for all independent 8819 reflections, *R*_{int} = 0.0518), *S* = 1.022.

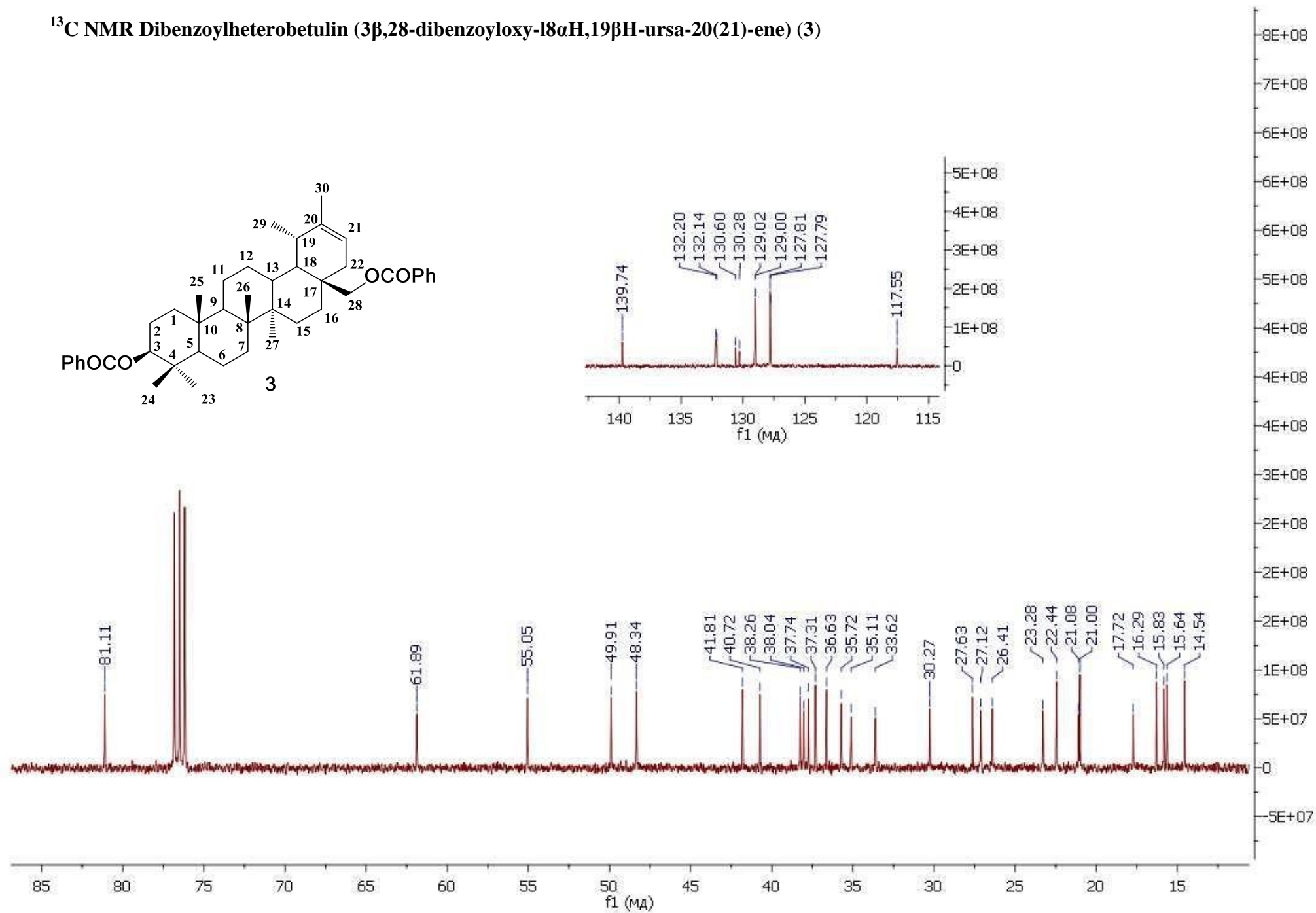
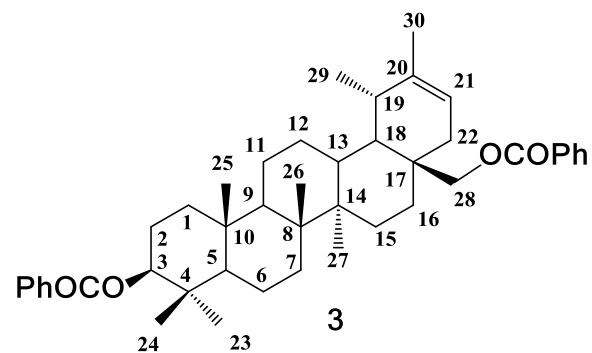
CCDC 1914625 contains the supplementary crystallographic data for this paper. The data can be obtained free of charge from The Cambridge Crystallographic Data Centre via <http://www.ccdc.cam.ac.uk>.

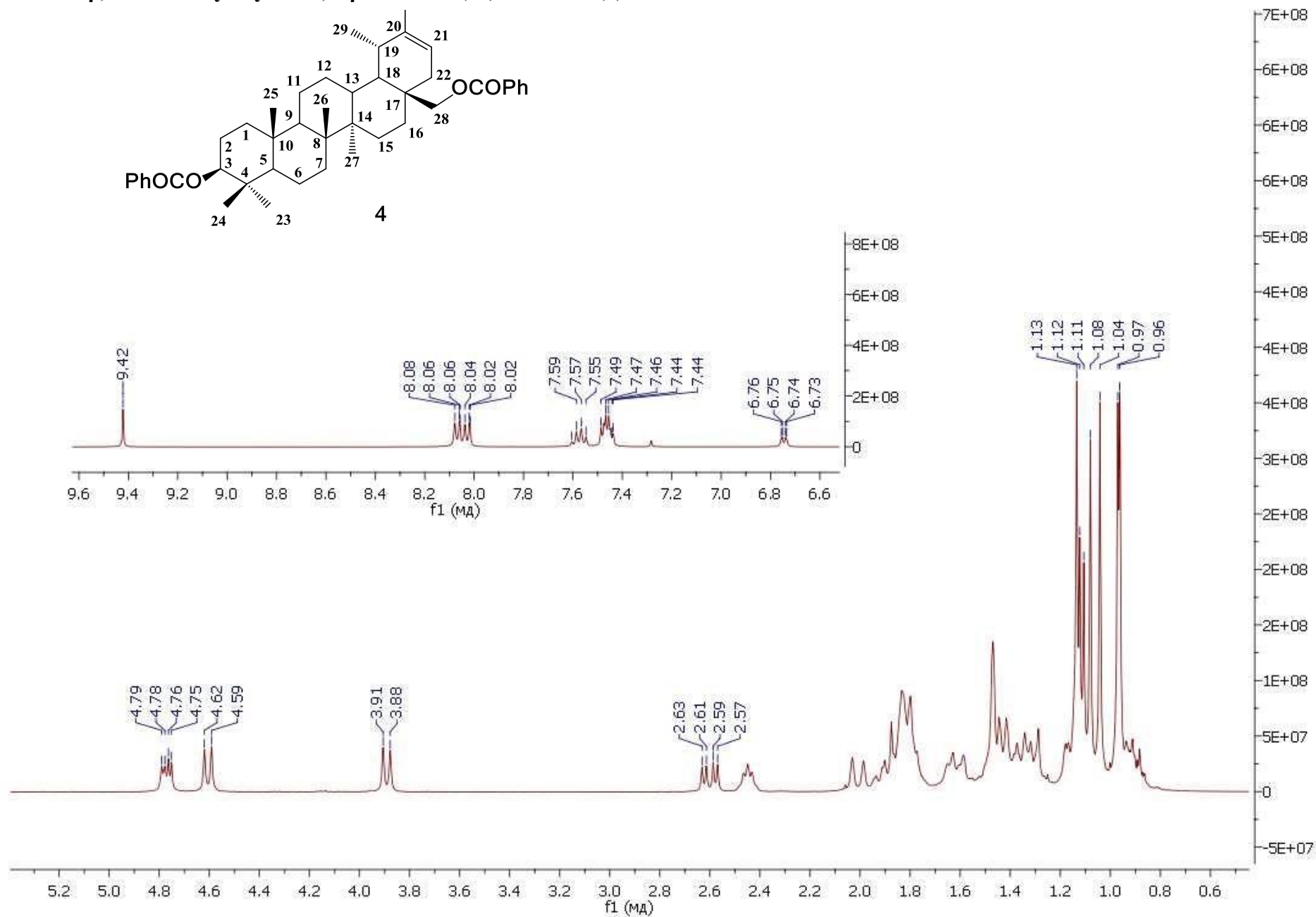
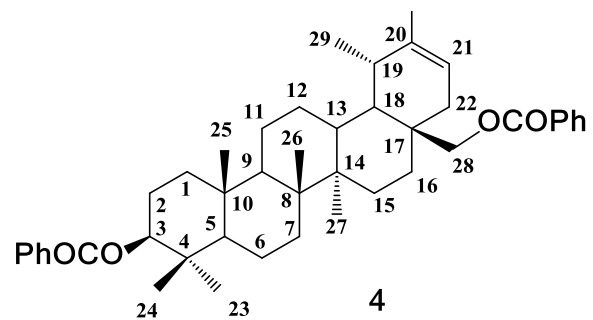
References

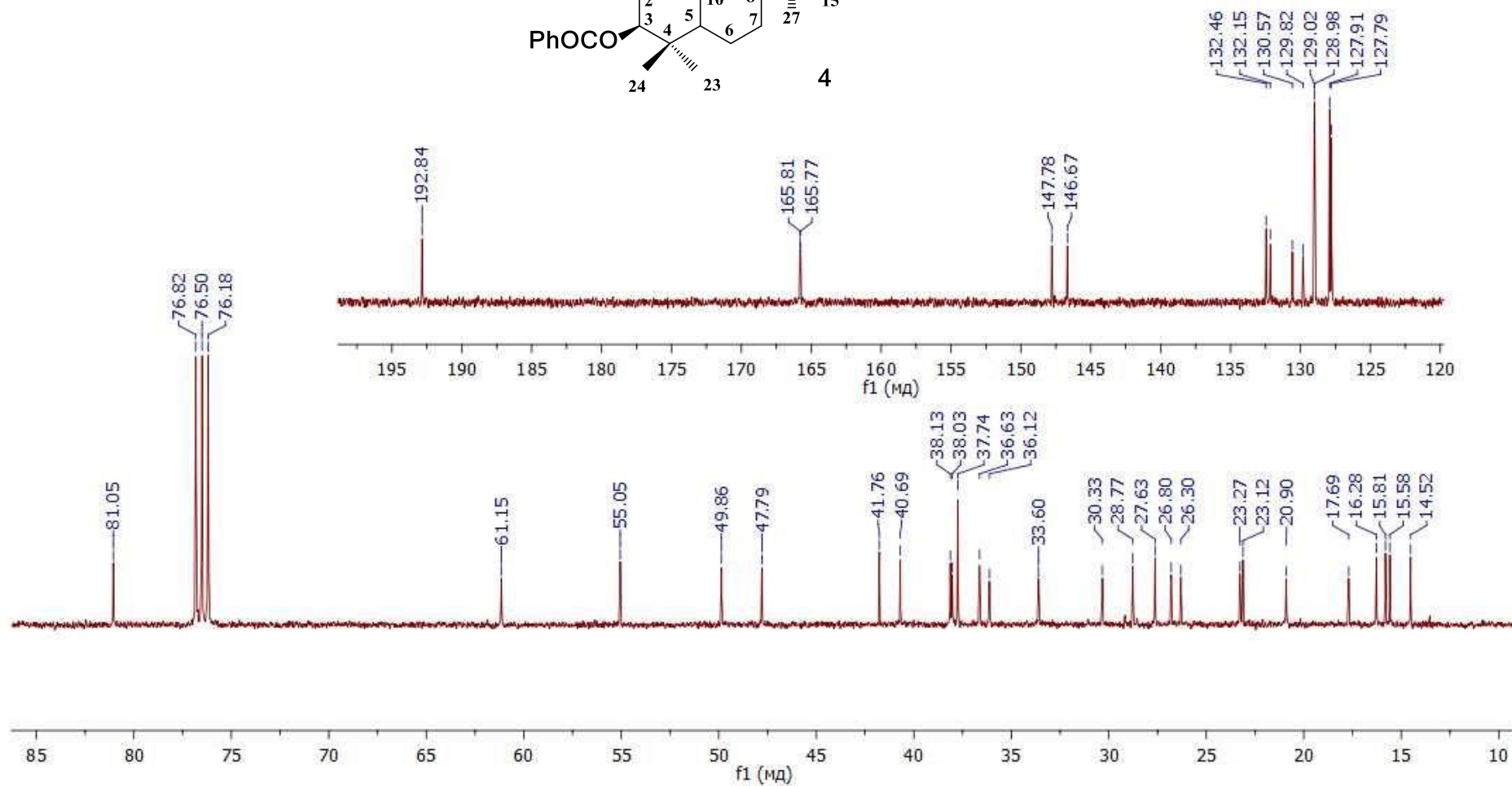
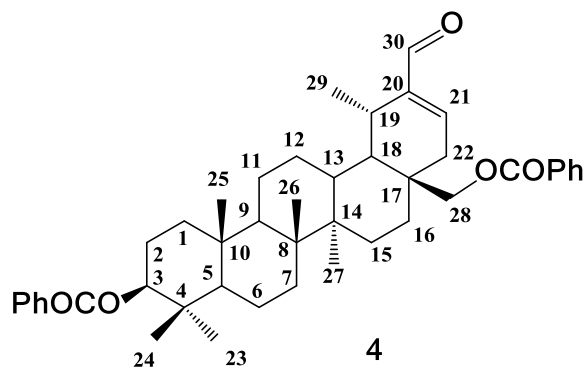
1. CrysAlisPro, Agilent Technologies, Version 1.171.37.33 (release 27-03-2014 CrysAlis171 .NET).

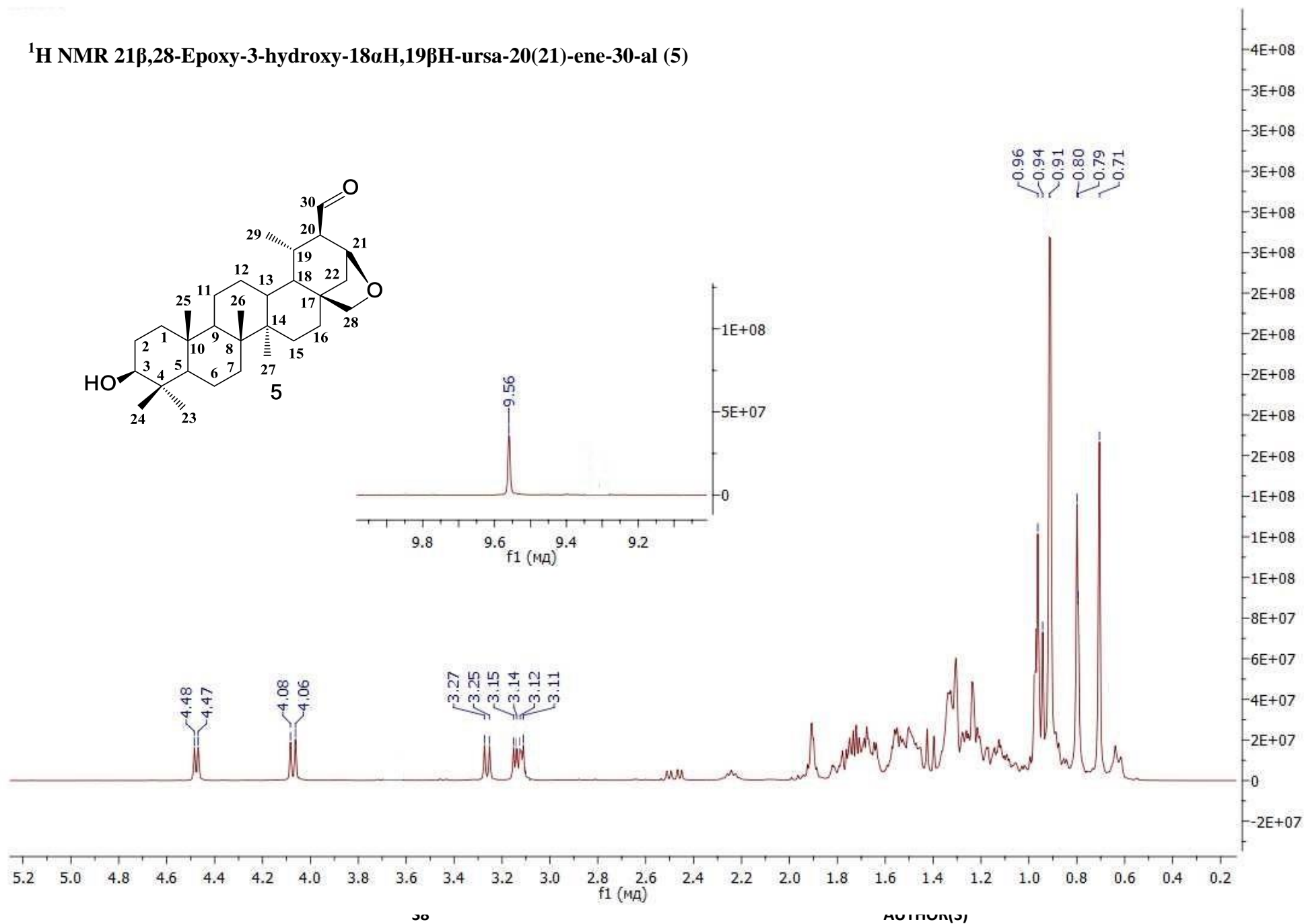
2. Dolomanov, O. V.; Bourhis, L. J.; Gildea, R. J.; Howard, J. A. K.; Puschmann, H. *J. Appl. Cryst.* **2009**, *42*, 339.
<http://dx.doi.org/10.1107/S0021889808042726>
3. Palatinus, L.; Chapuis, G. *J. Appl. Cryst.* **2007**, *40*, 786.
<https://doi.org/10.1107/S0021889807029238>
4. Sheldrick, G. M. *Acta Cryst.* **2015**, *C71*, 3.
<https://doi.org/10.1107/S2053229614024218>

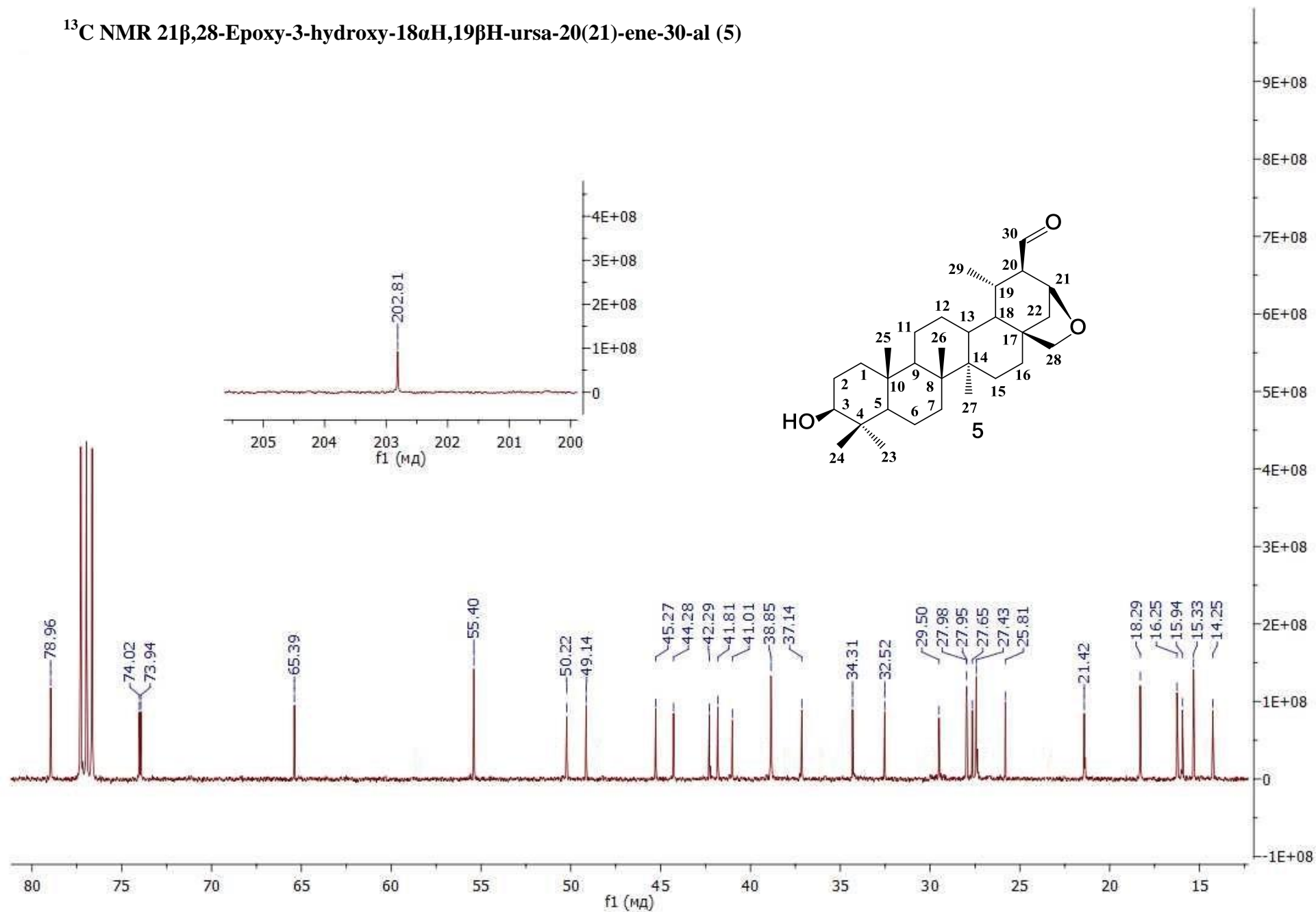
2. ^1H , ^{13}C NMR and 2D spectra ^1H NMR Dibenzoylheterobetulin ($3\beta,28$ -dibenzoyloxy- $18\alpha\text{H},19\beta\text{H}$ -ursa- $20(21)$ -ene) (3)

^{13}C NMR Dibenzoylheterobetulin ($3\beta,28$ -dibenzoyloxy- $18\alpha\text{H},19\beta\text{H}$ -ursa- $20(21)$ -ene) (3)

^1H NMR $3\beta,28$ -Dibenzoyloxy- $18\alpha\text{H},19\beta\text{H}$ -ursa- $20(21)$ -ene- 30 -al (4)

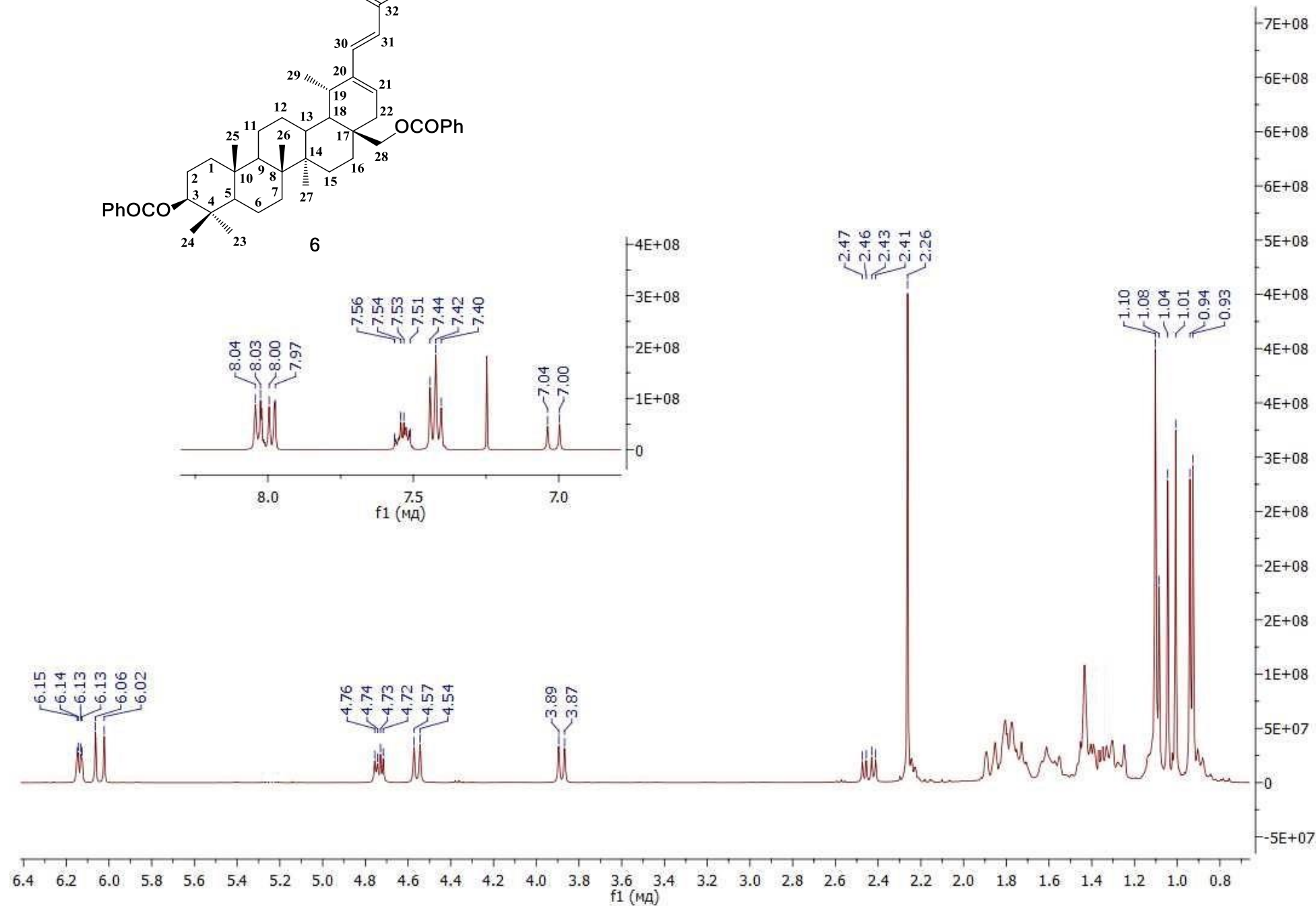
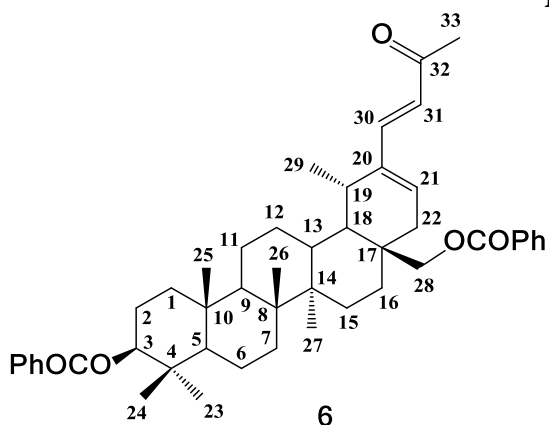
^{13}C NMR $3\beta,28$ -Dibenzoyloxy- $18\alpha\text{H},19\beta\text{H}$ -ursa- $20(21)$ -ene- 30 -al (4)

^1H NMR 21 β ,28-Epoxy-3-hydroxy-18 α H,19 β H-ursa-20(21)-ene-30-al (5)

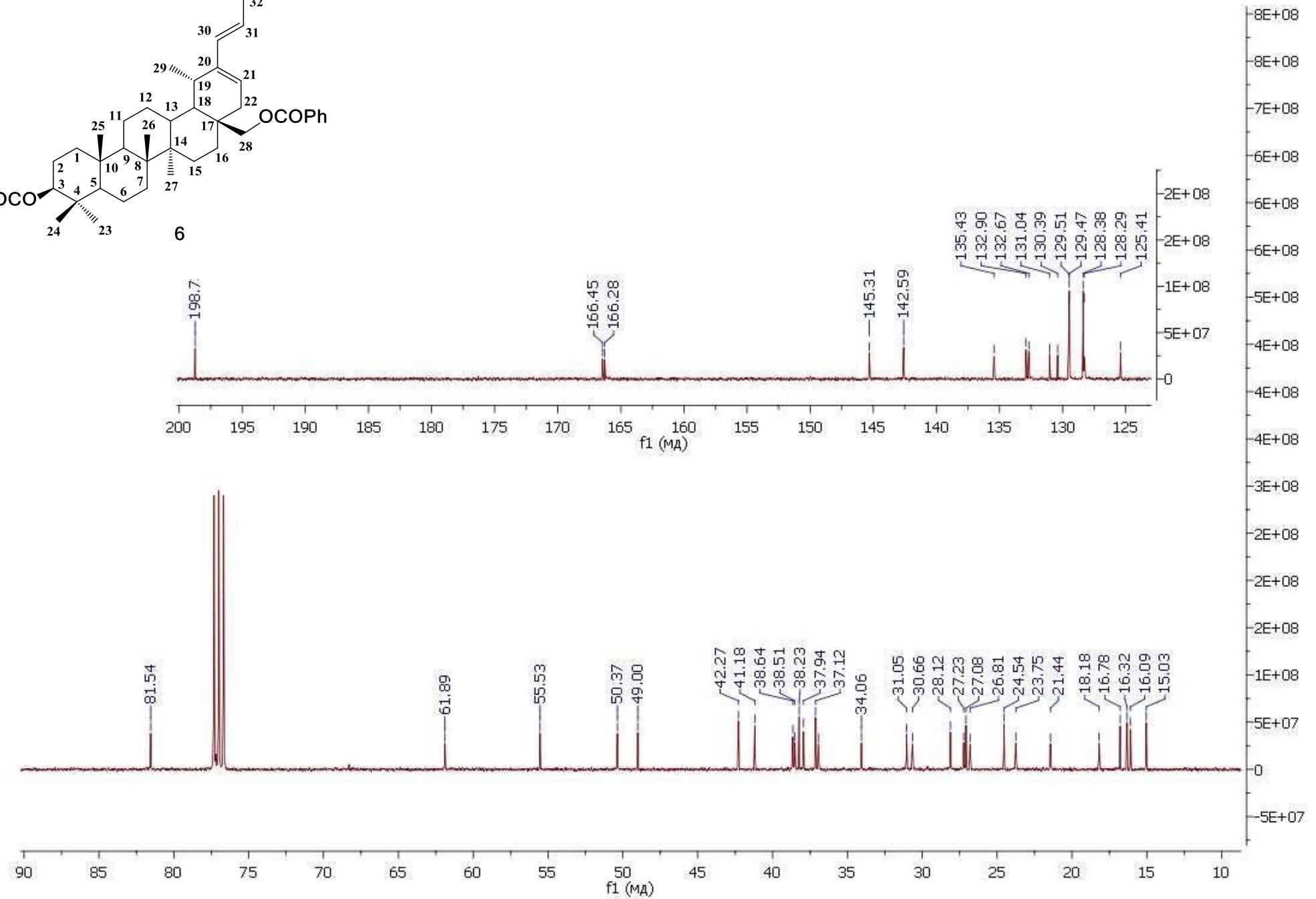
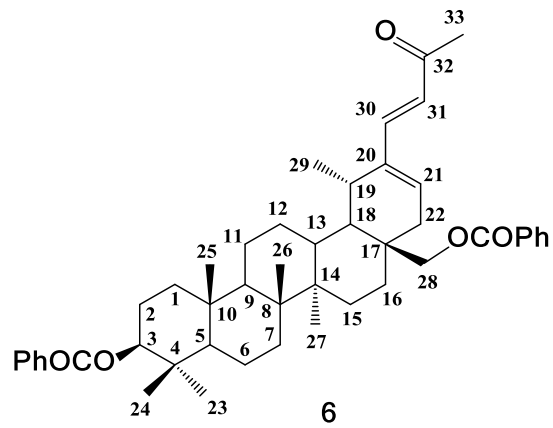
^{13}C NMR 21 β ,28-Epoxy-3-hydroxy-18 α H,19 β H-ursa-20(21)-ene-30-al (5)

General P:

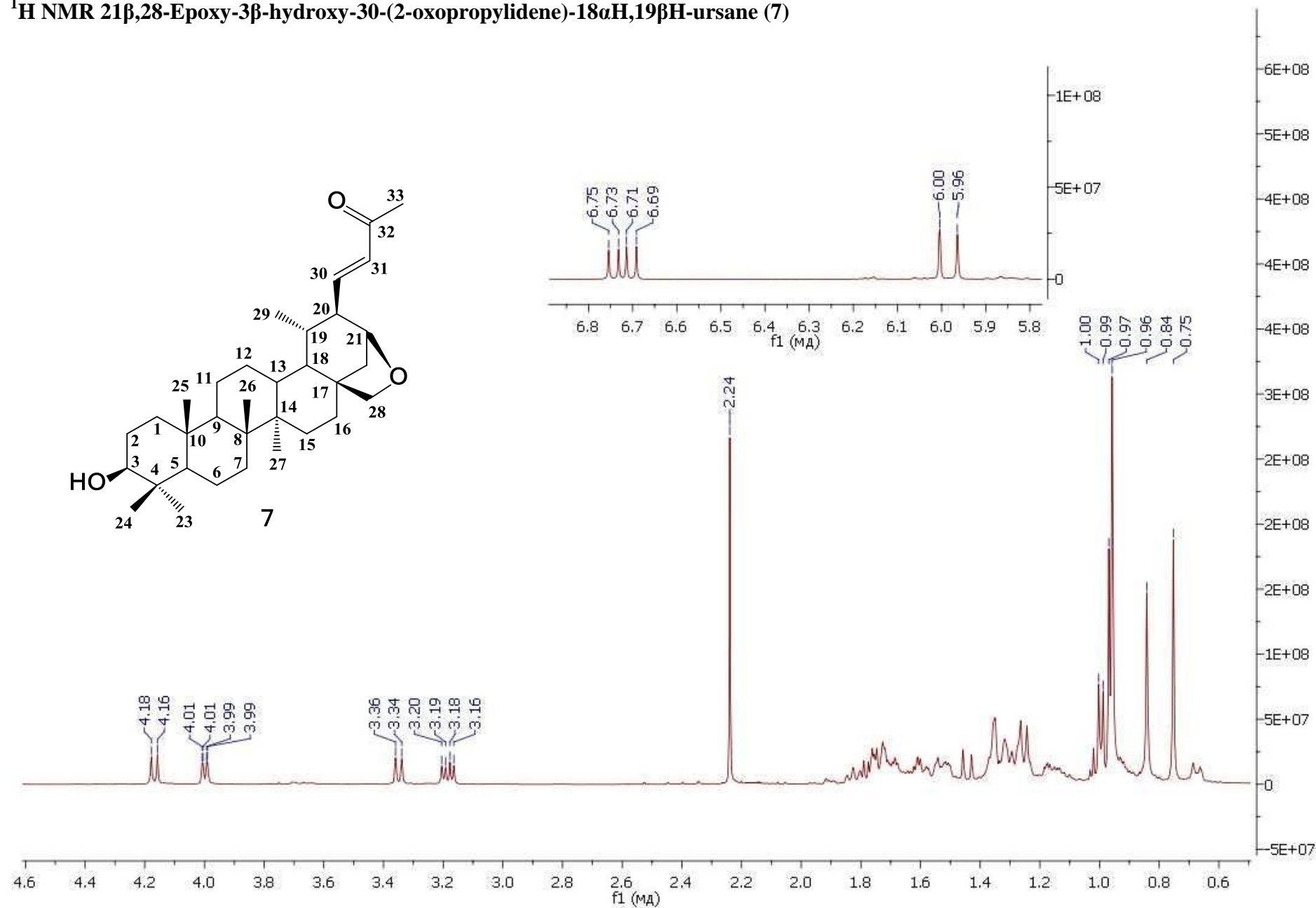
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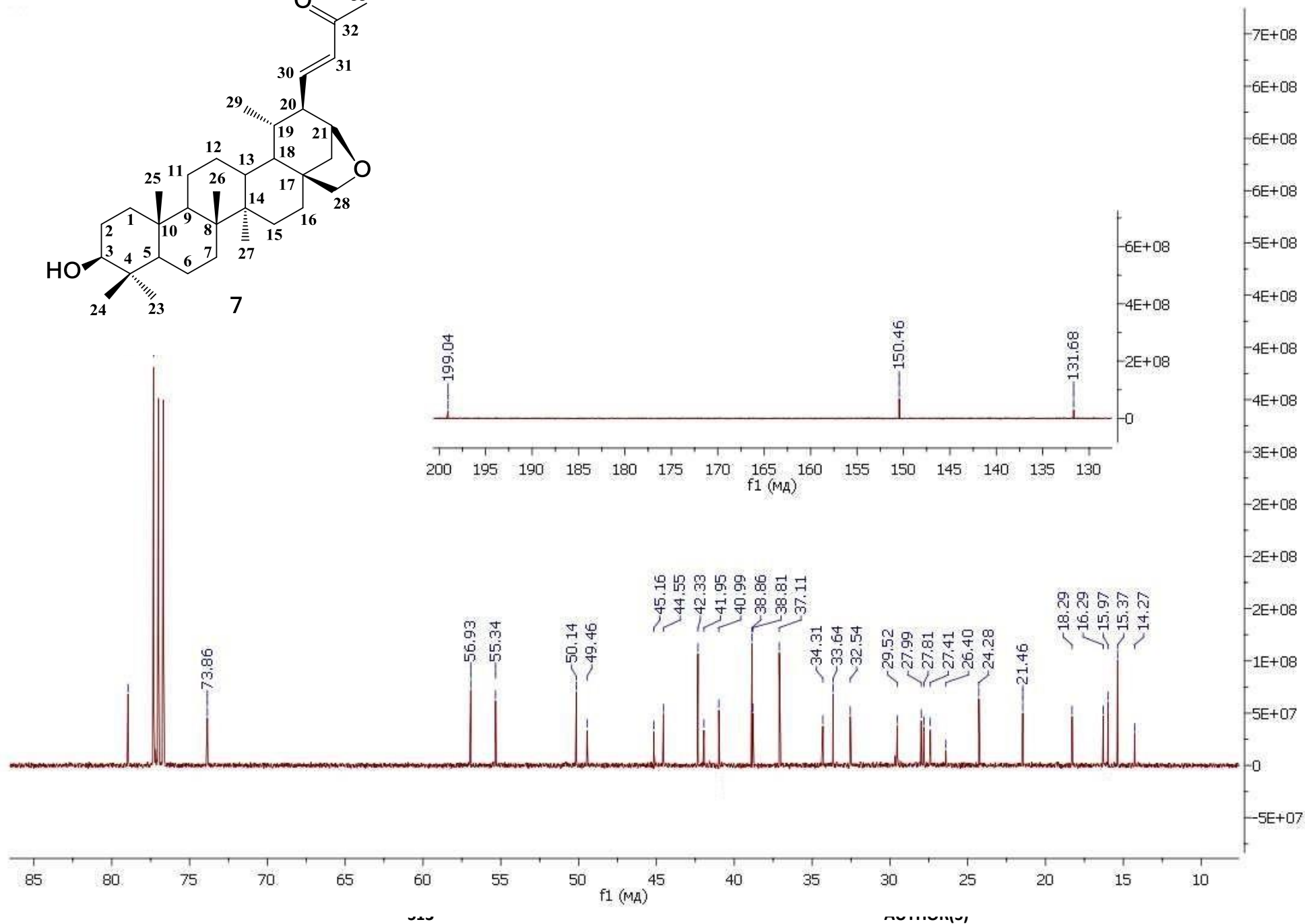
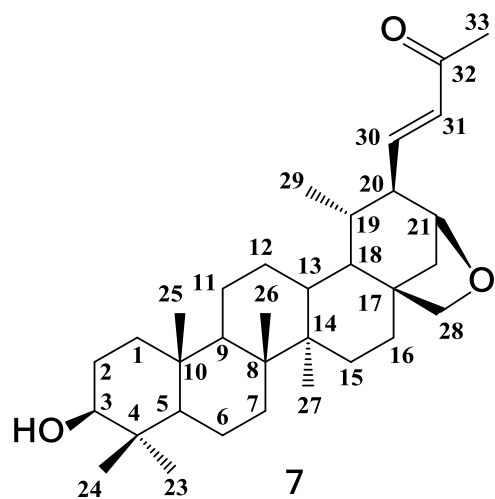
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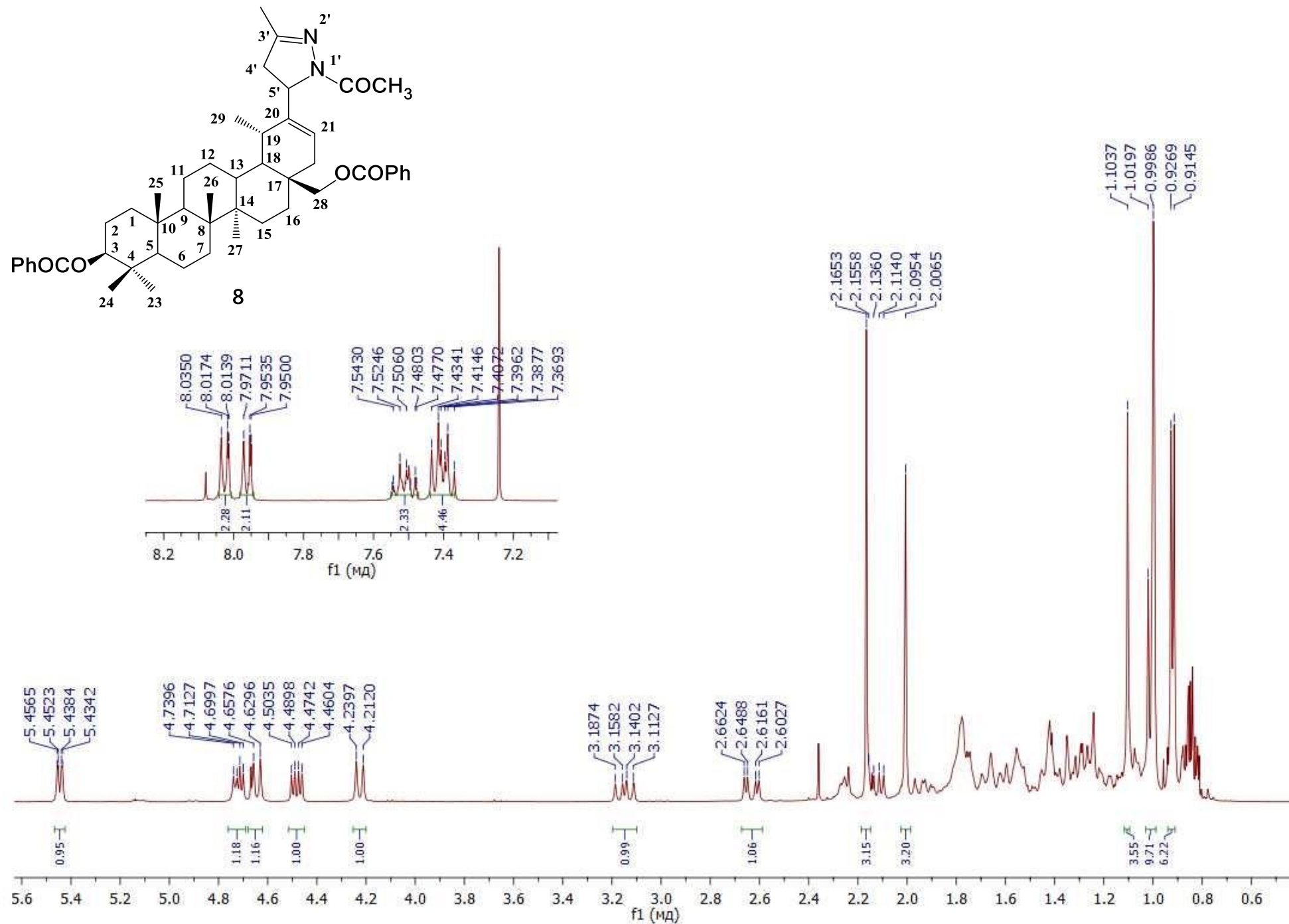
¹H NMR 21β,28-Epoxy-3β-hydroxy-30-(2-oxopropylidene)-18αH,19βH-ursane (7)

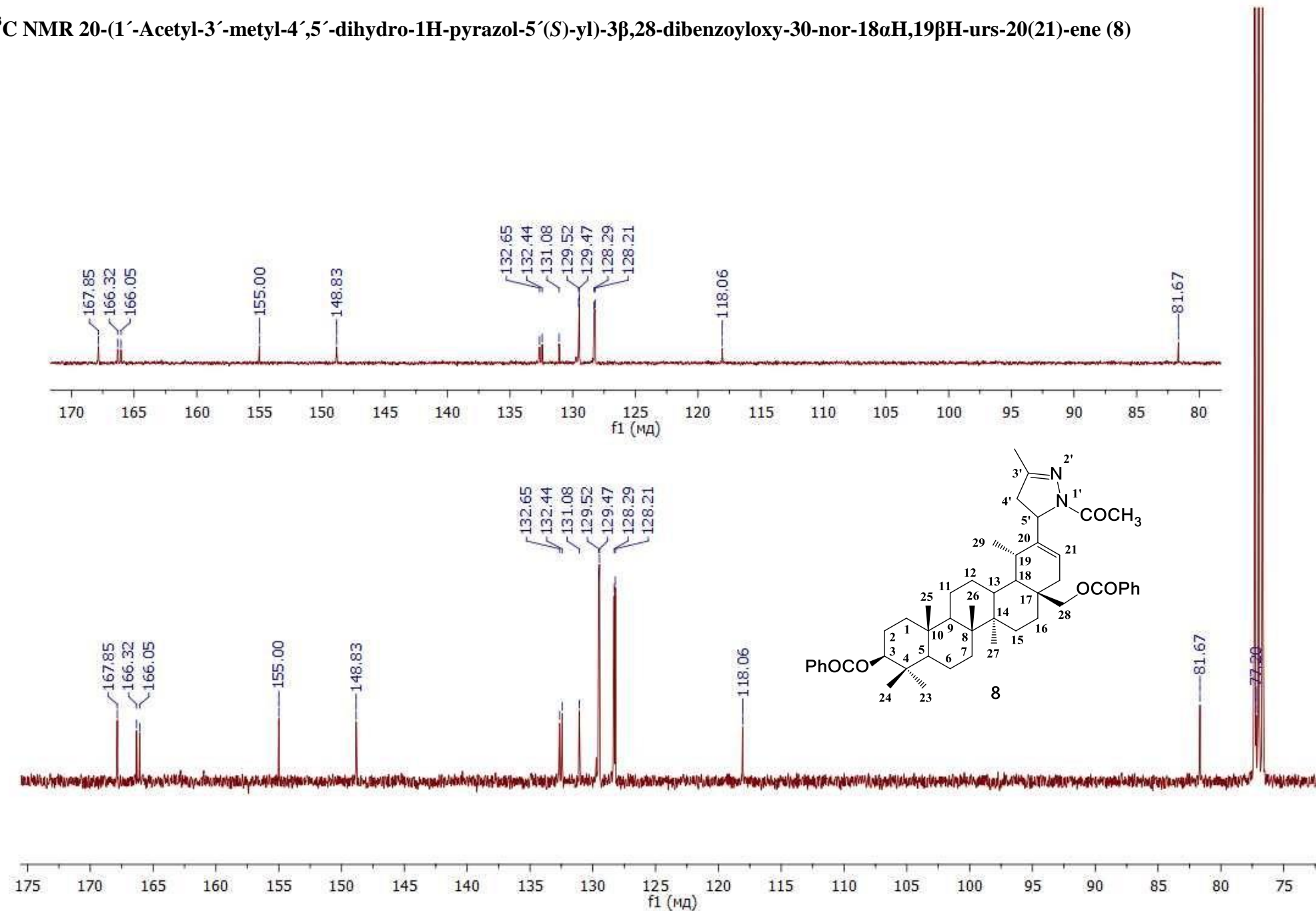


¹³C NMR 21β,28-Epoxy-3β-hydroxy-30-(2-oxopropylidene)-18αH,19βH-ursane (7)

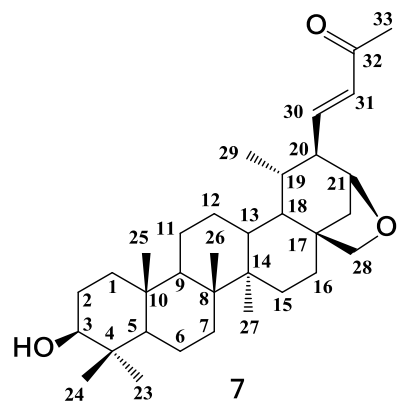


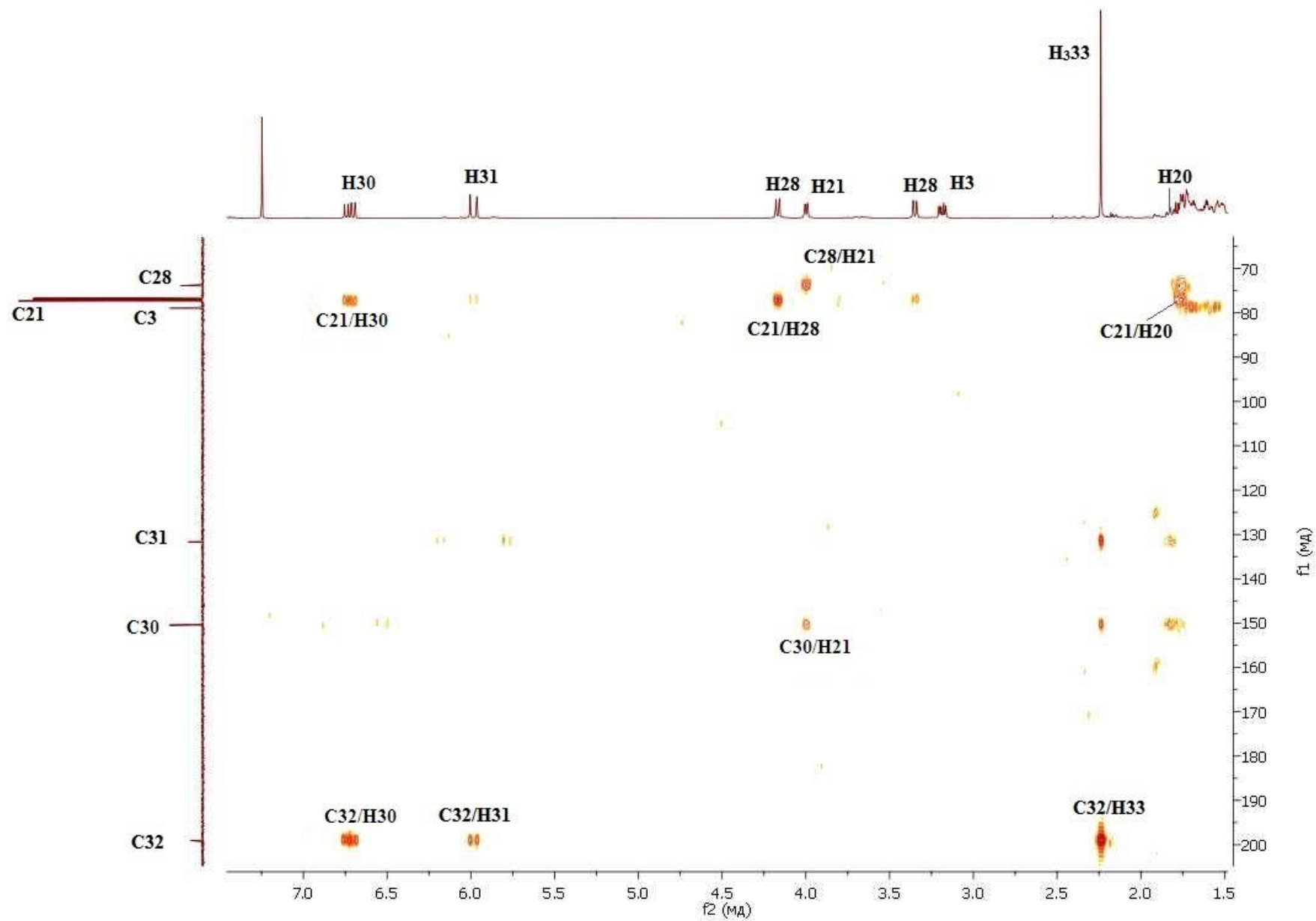
¹H NMR 20-(1'-Acetyl-3'-methyl-4',5'-dihydro-1H-pyrazol-5'-(S)-yl)-3β,28-dibenzoyloxy-30-nor-18αH,19βH-urs-20(21)-ene (8)

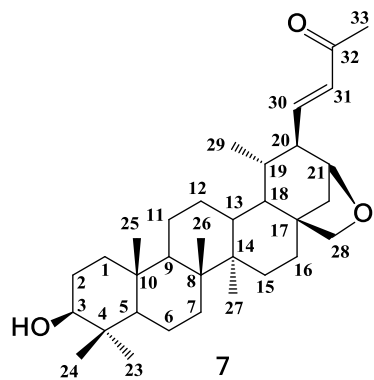


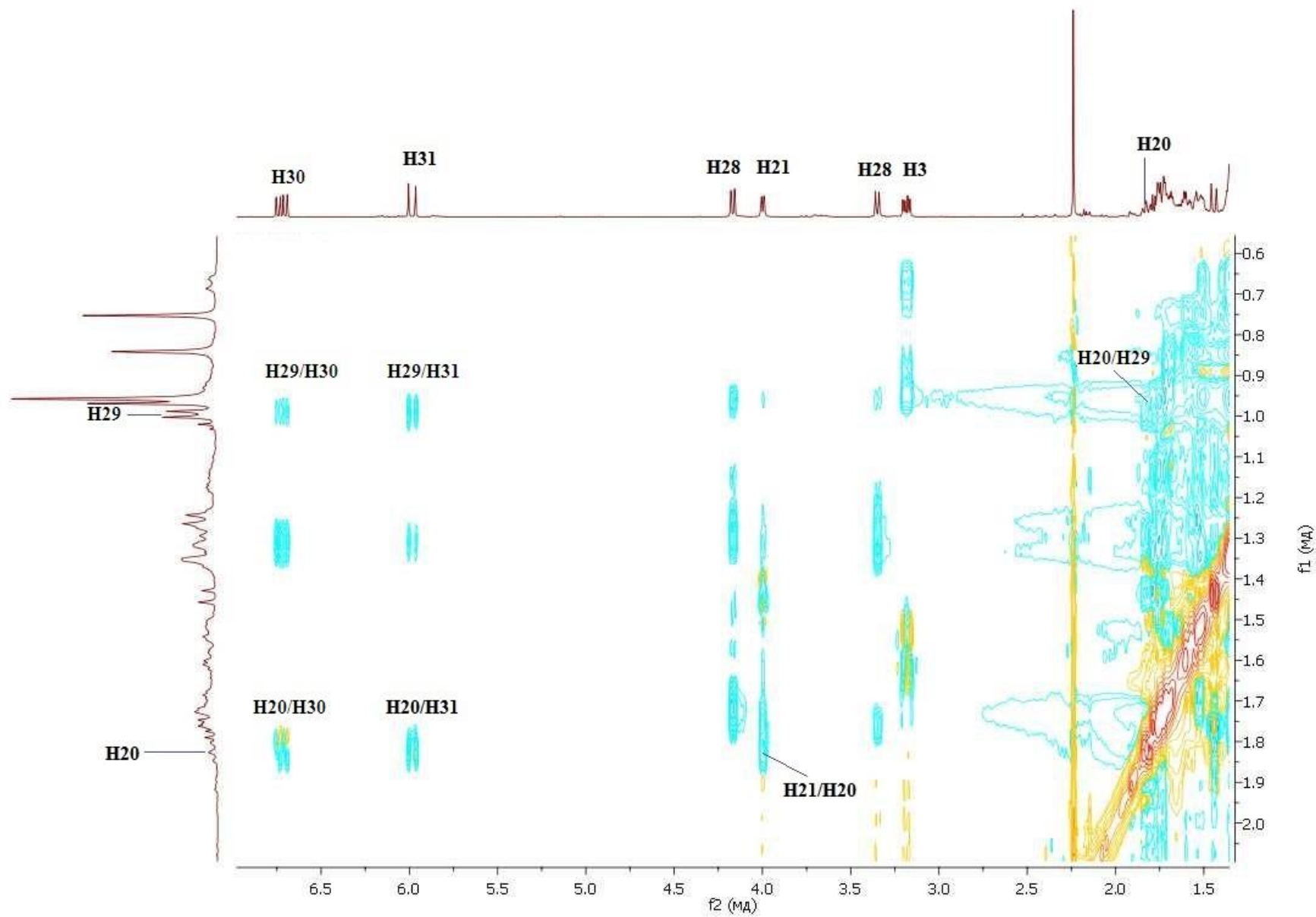
^{13}C NMR 20-(1'-Acetyl-3'-methyl-4',5'-dihydro-1H-pyrazol-5'-(S)-yl)-3 β ,28-dibenzoyloxy-30-nor-18 α H,19 β H-urs-20(21)-ene (8)

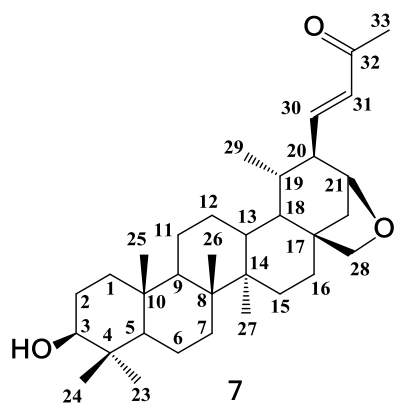
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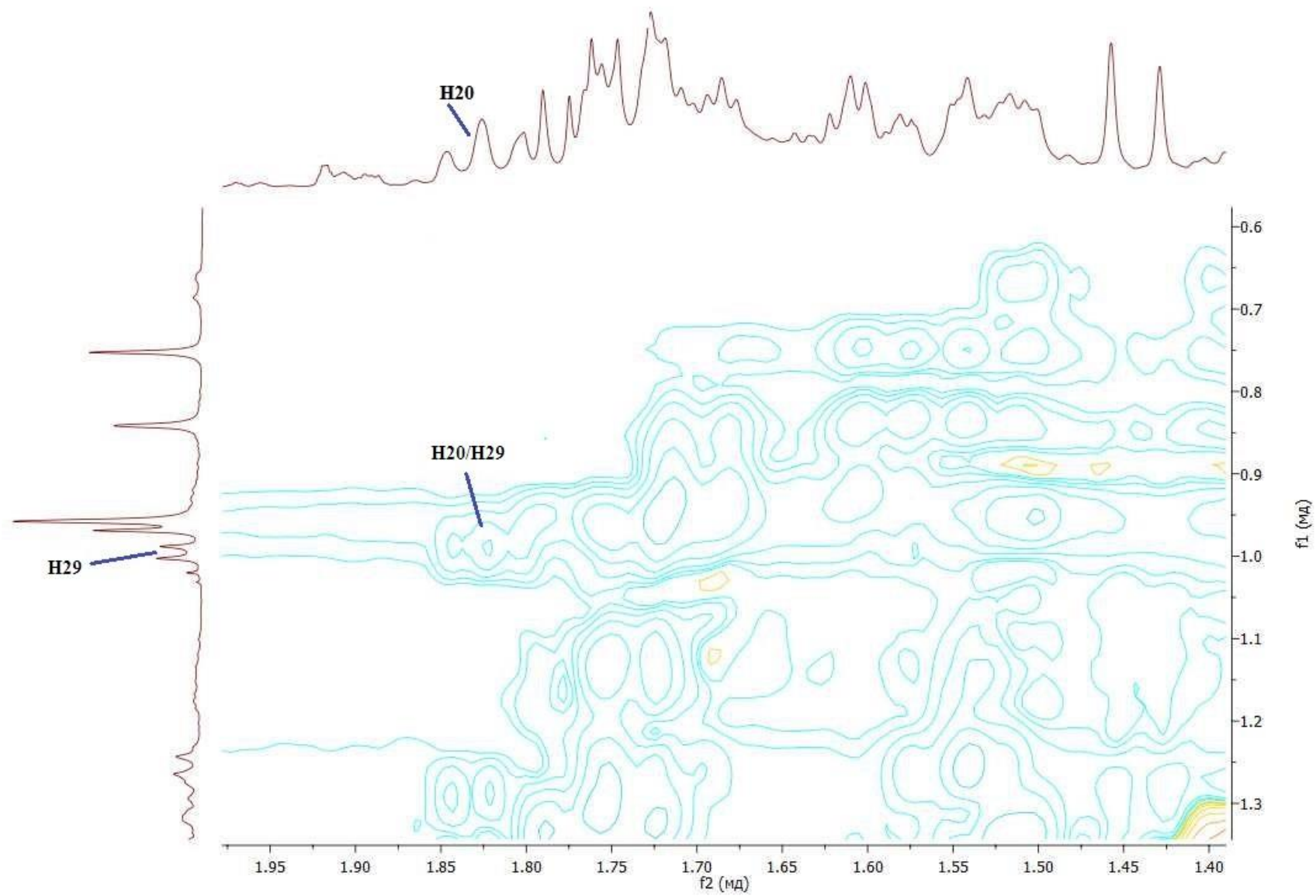
21 β ,28-Epoxy-3 β -hydroxy-30-(2-oxopropylidene)-18 α H,19 β H-ursane (7)



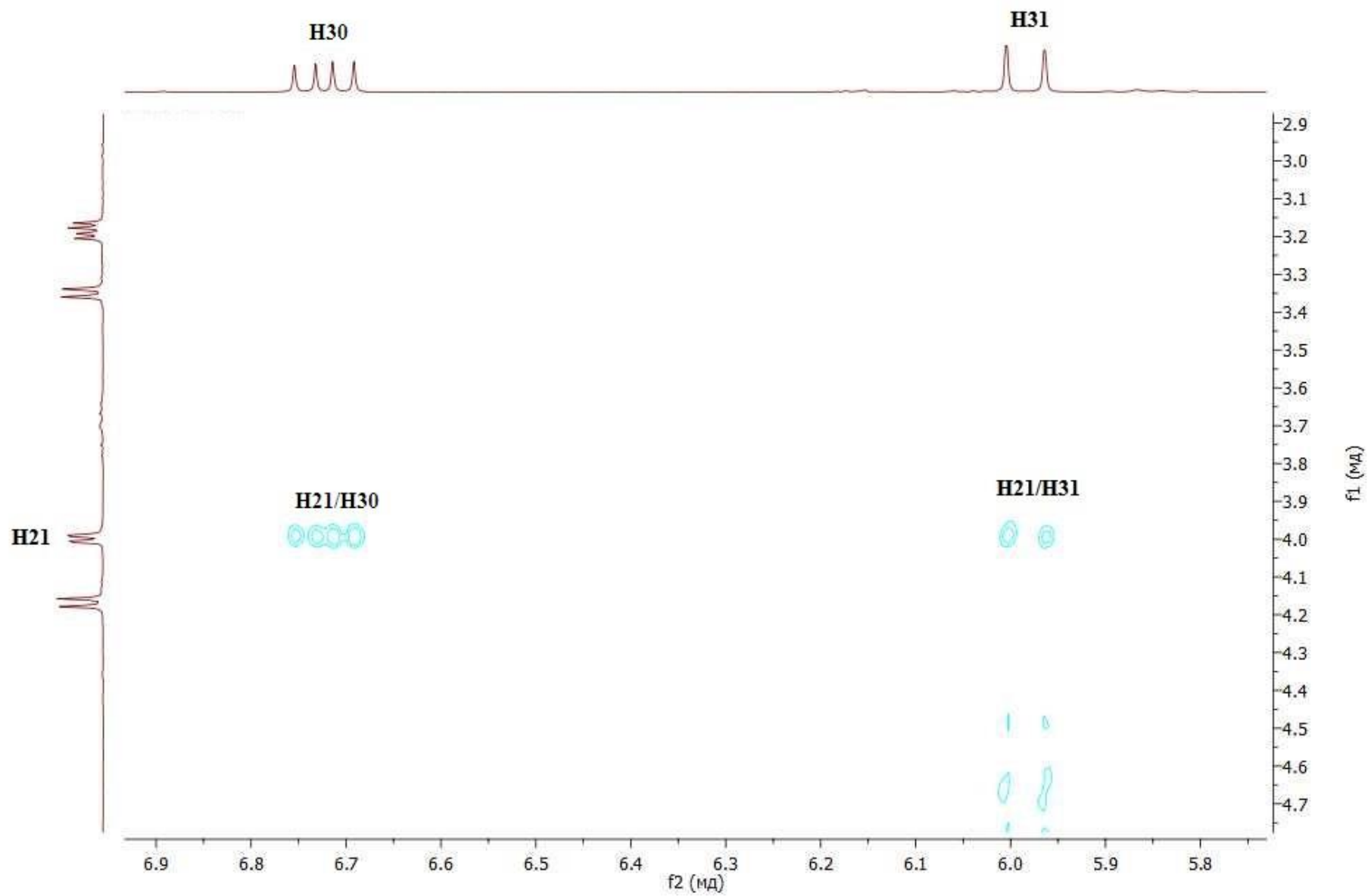
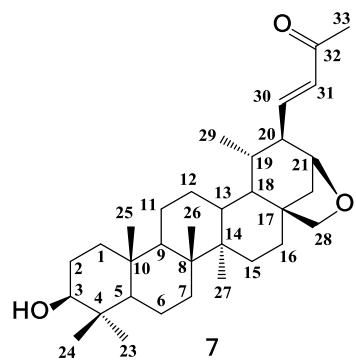
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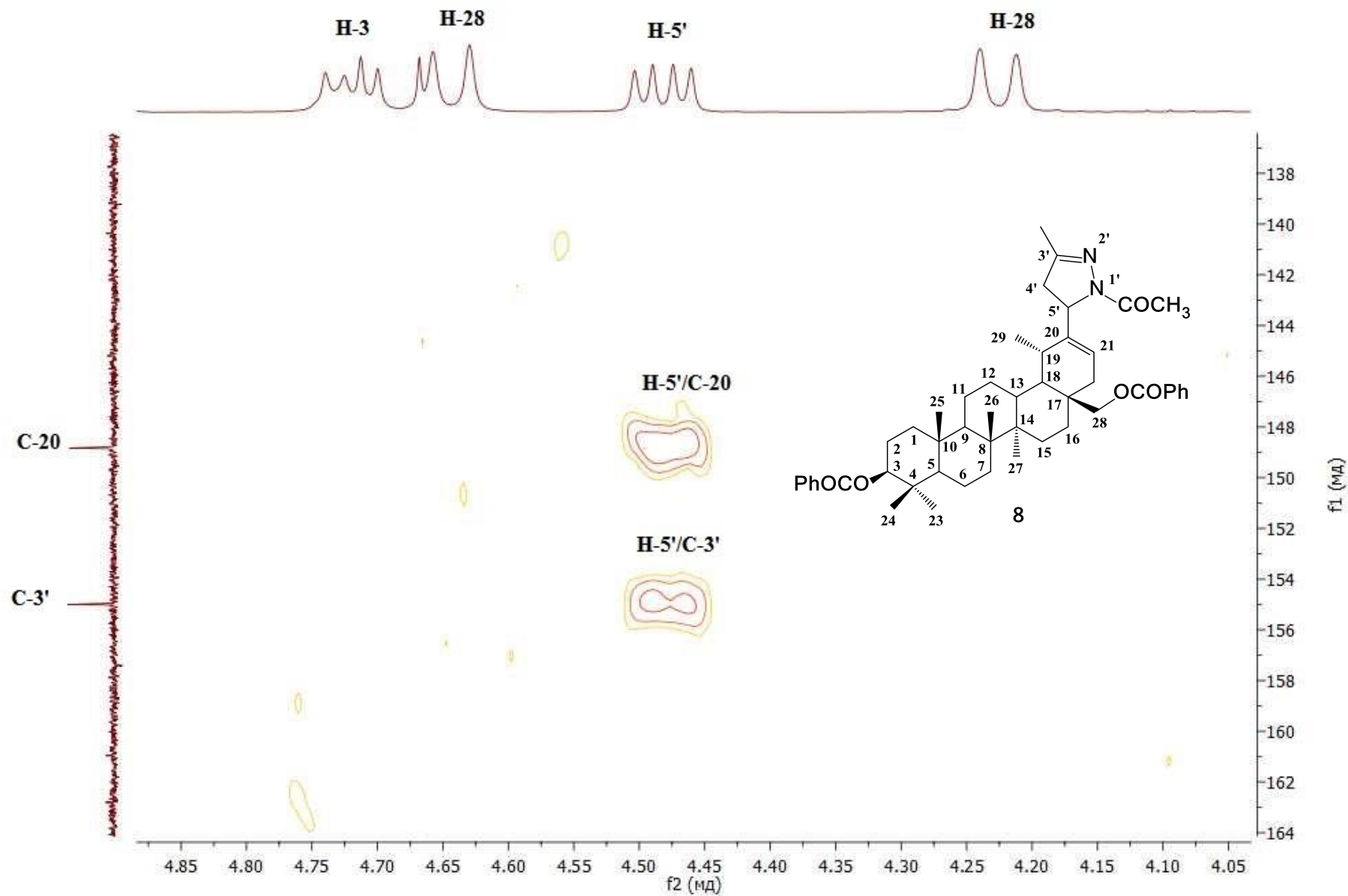
**Fragment 2 of NOESY****21 β ,28-Epoxy-3 β -hydroxy-30-(2-oxopropylidene)-18 α H,19 β H-ursane (7)**



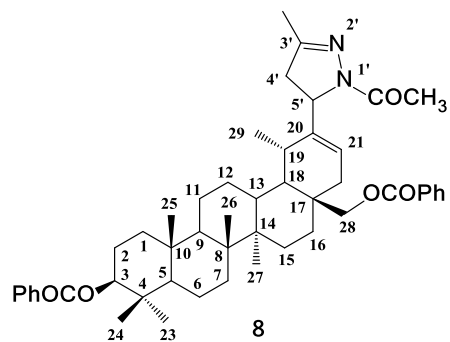
Fragment 3 of NOESY
21 β ,28-Epoxy-3 β -hydroxy-30-(2-oxopropylidene)-18 α H,19 β H-ursane (7)



Fragment 1 of HMBC

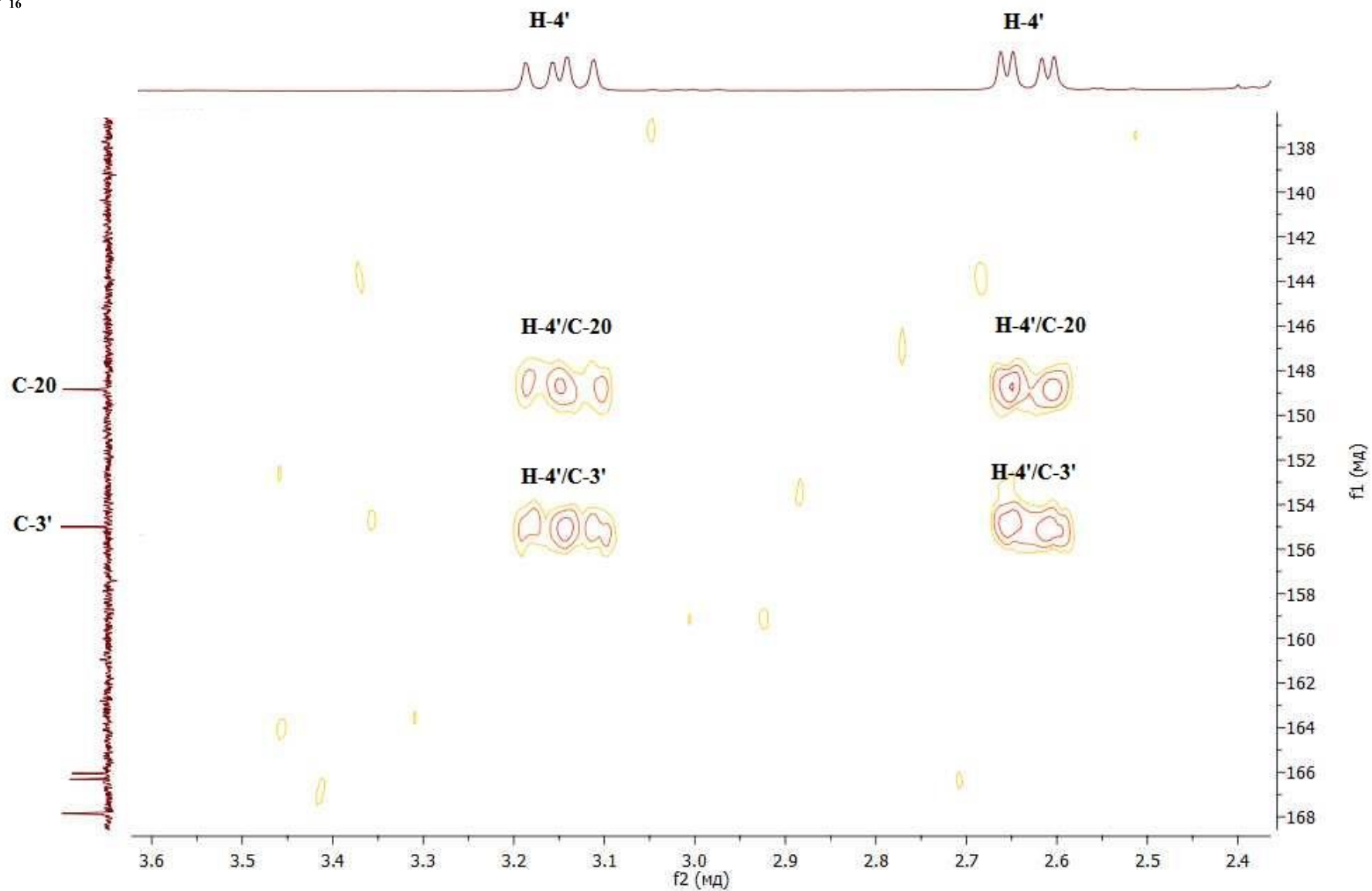
20-(1'-Acetyl-3'-methyl-4',5'-dihydro-1H-pyrazol-5'(S)-yl)-3 β ,28-dibenzoyloxy-30-nor-18 α H,19 β H-urs-20(21)-ene (8)

General Papers

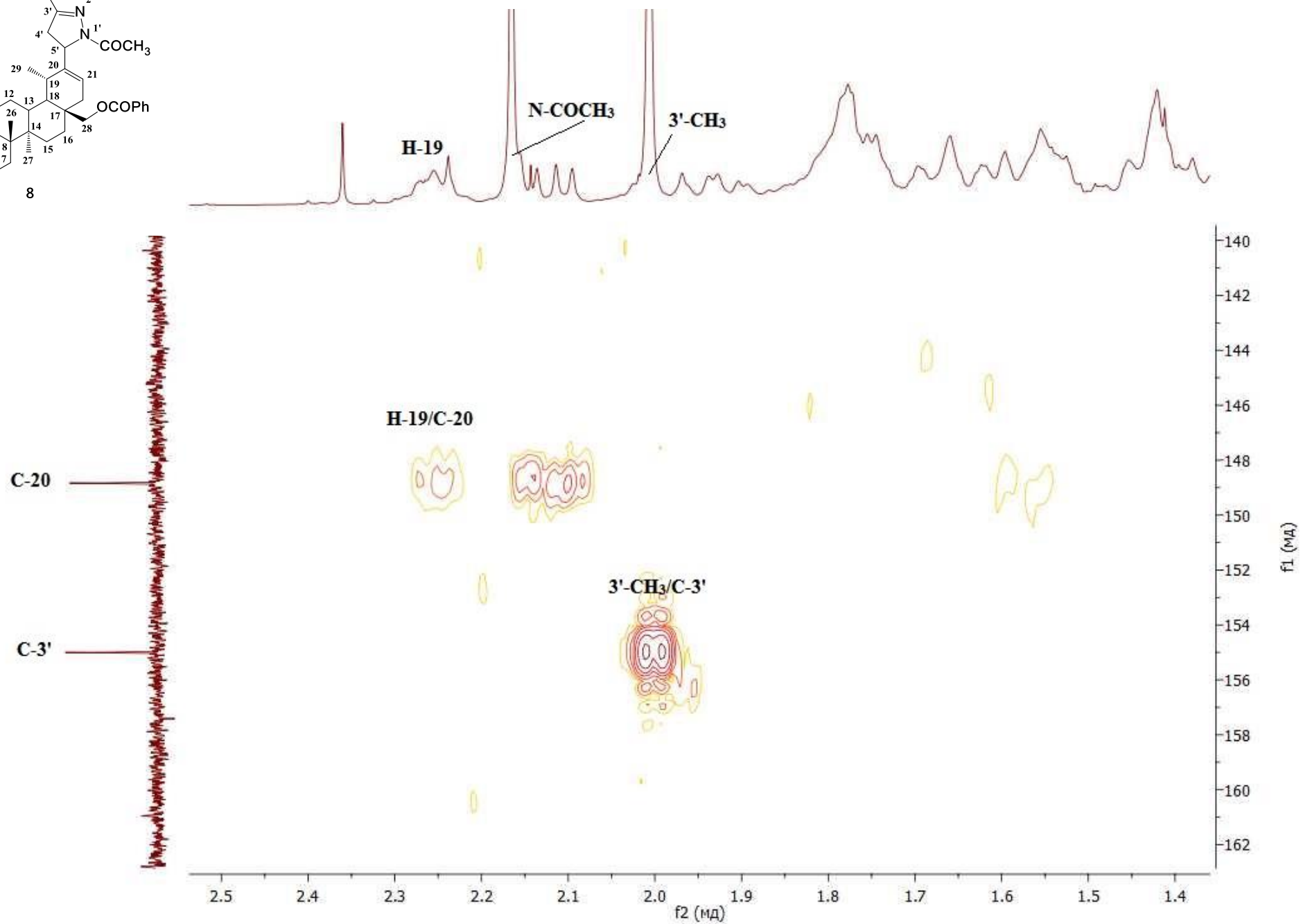
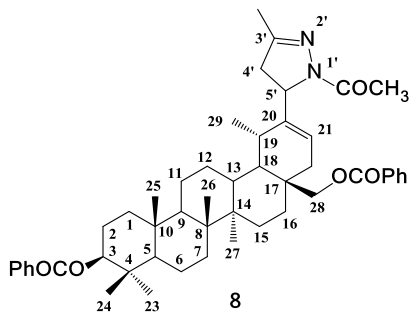


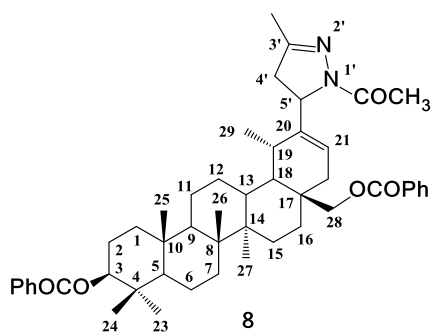
Fragment 2 of HMBC

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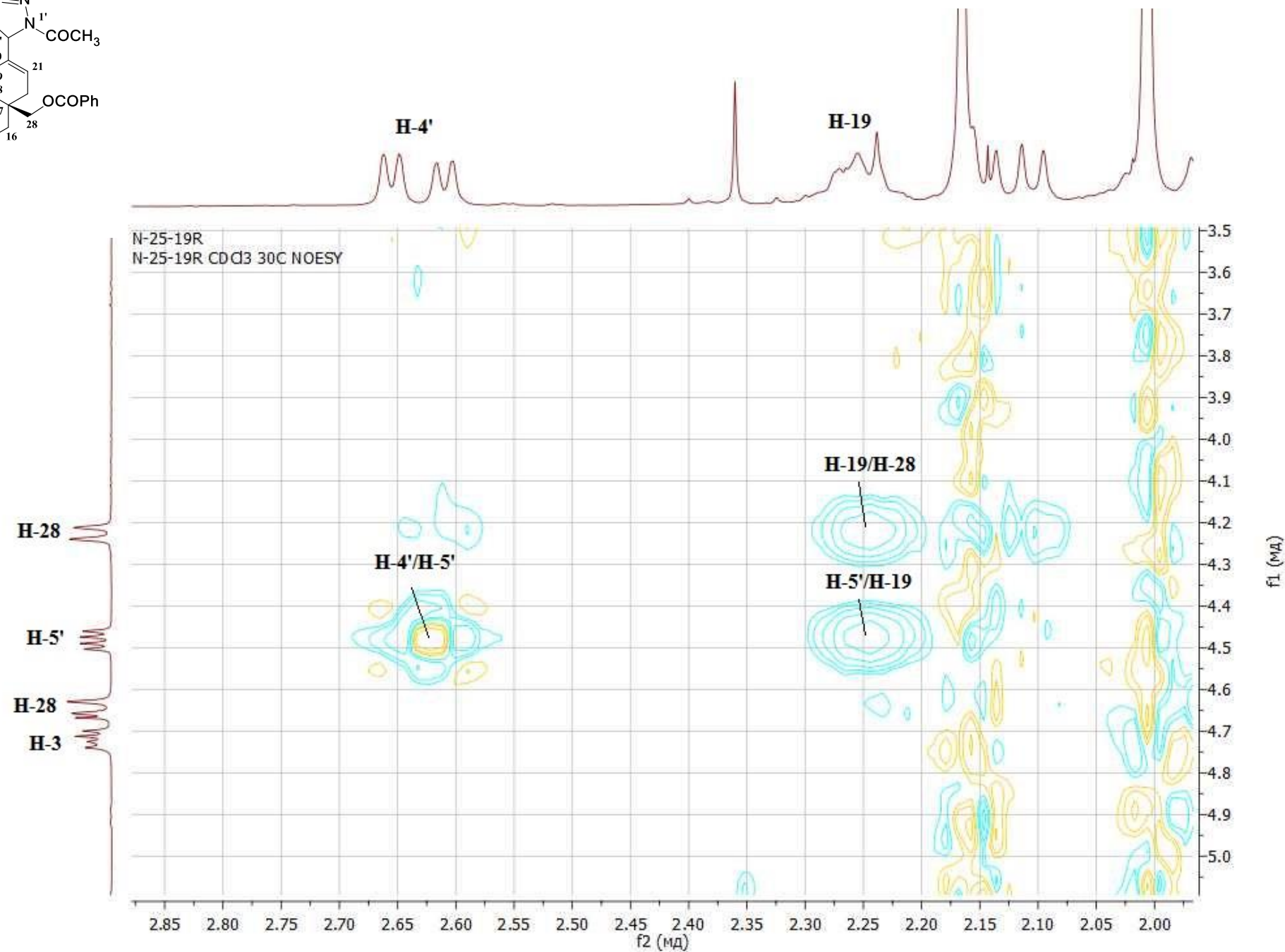


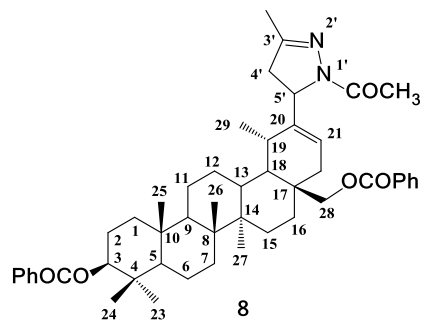
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Fragment 2 of NOESY

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