Photochemistry of β-γ-Unsaturated Spirolactones

Werner Fudickar, Melanie Metz, Tobias Krüger-Braunert, Alexandra Kelling, Eric Sperlich, Pablo Wessig and Torsten Linker*

Department of Chemistry, University of Potsdam, Karl-Liebknecht-Str. 24-25, 14476 Golm, Germany
* Correspondence: linker@uni-potsdam.de

Supplementary Materials

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1. DFT calculations

General

DFT calculations were performed with the functional MN15 [30] and the basis set 6-31G* [31], using the program package Gaussian 16 [32]. All stationary point were characterized by frequency analysis. Energies were corrected by the zero point vibrational energy (ZPE). Biradicals were calculated with a spin-unrestricted wavefunction and using the keyword "guess=mix" to destroy α-β and spatial symmetries of the initial guess. Excited states were calculated using time-dependent DFT (TD-DFT) [33,34].

Figure S1: Excited state of spirolactone 11a

Geometry of 11a was optimized at MN15/6-31G* level and singlet excited states were calculated with TDDFT at same level.

Excited states and oscillator strength:

Excited State 1: Singlet-A 5.0648 eV **244.80 nm** f=0.0050 <S**2>=0.000

38 -> 41	0.34377
38 -> 43	0.25724
39 -> 41	-0.11933
40 -> 41	**0.49106**
40 -> 43	0.20228

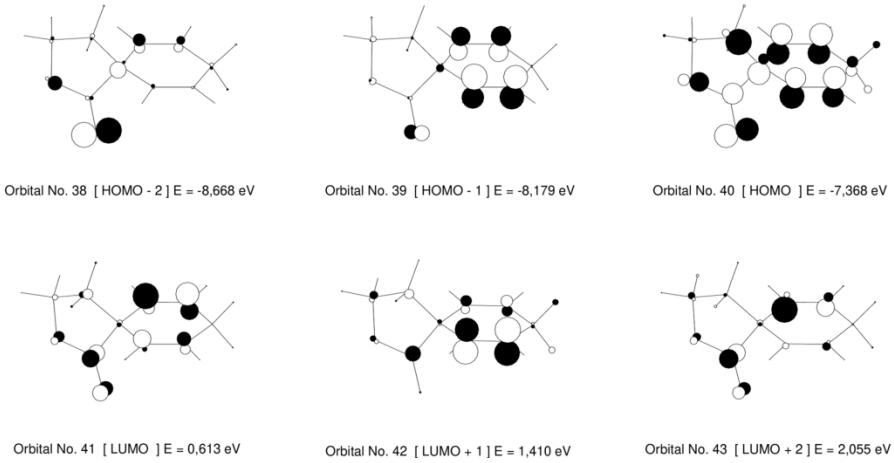
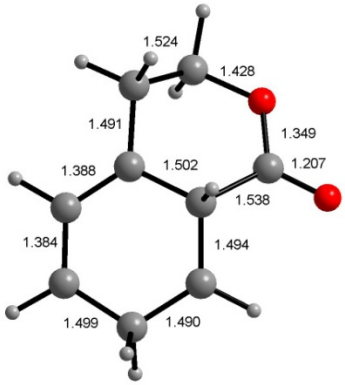
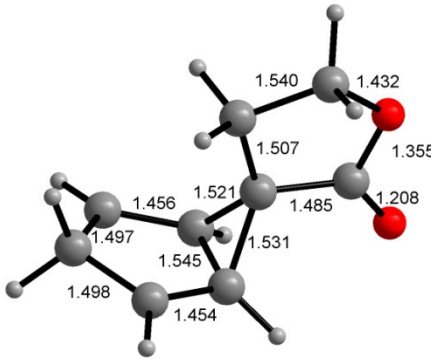


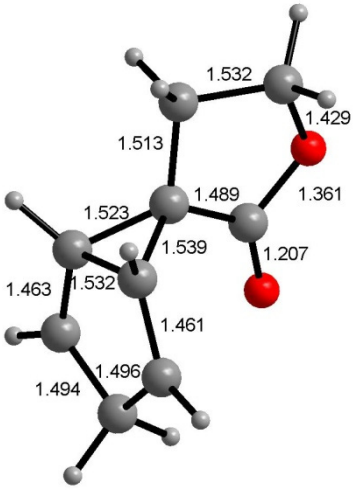
Figure S2: Geometries and energies of BR4, BR2a, BR2b, BR3a, BR3b



BR4
E_{REL} = 0.0 kcal/mol



BR2a
E_{REL} = 9.7 kcal/mol



BR2b
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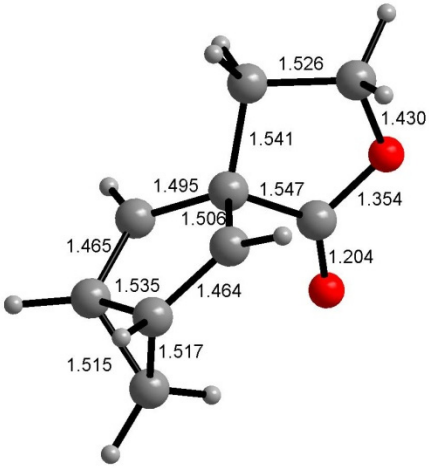

BR3a
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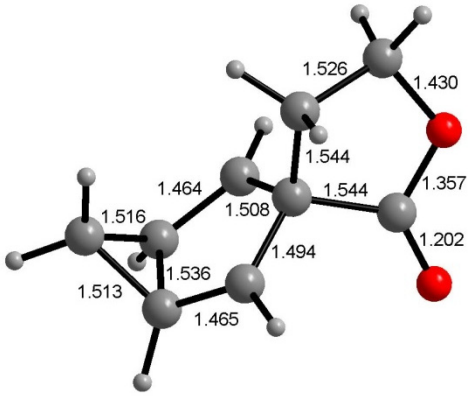

BR3b
E_{REL} = 16.9 kcal/mol

BR4 E = -498,7371415 a.u. ZPE = 106,89214 kcal/mol

C	2.653967	-0.608247	-0.261815

C	1.552862	-1.426673	-0.079005
C	0.300433	-0.947382	0.280626
C	0.070348	0.516048	0.526725
C	1.164468	1.385492	-0.001139
C	2.559759	0.882662	-0.140522
C	-0.917926	-1.790080	0.446297
C	-1.993100	-1.244748	-0.486341
O	-2.262409	0.137476	-0.247846
C	-1.291550	1.031153	0.032494
O	-1.533702	2.211591	-0.028339
H	3.619860	-1.042244	-0.502606
H	1.667420	-2.503260	-0.207871
H	-0.027240	0.655955	1.628675
H	0.944263	2.437698	-0.128783
H	3.182237	1.213279	0.715939
H	3.039222	1.356952	-1.011725
H	-1.296336	-1.739147	1.480110
H	-0.722484	-2.842716	0.211331
H	-2.951163	-1.753204	-0.347767
H	-1.682459	-1.365193	-1.531741

BR2a E = -498,7209952 a.u. ZPE = 106,5157 kcal/mol

C	-2.052976	-0.196463	-1.146336
C	-0.884075	-0.924124	-0.674457
C	0.205648	-0.071091	-0.043265
C	-0.838328	-0.734646	0.858406
C	-1.985124	0.086819	1.210283
C	-2.708074	0.541007	-0.020125
C	0.387104	1.410613	-0.247200
C	1.882322	1.587118	0.075500
O	2.489688	0.295652	-0.038474
C	1.556576	-0.685928	-0.007338
O	1.826458	-1.862434	0.039579
H	-2.467125	-0.278835	-2.141785
H	-0.535741	-1.845163	-1.136803
H	-0.455004	-1.516666	1.509828
H	-2.341622	0.244527	2.218981
H	-2.670079	1.638759	-0.154592
H	-3.783836	0.304039	0.039675
H	-0.245722	2.009307	0.418076
H	0.156427	1.691550	-1.282114
H	2.036308	1.946202	1.099131
H	2.398776	2.260713	-0.612046

BR2b E = -498,7153636 a.u. ZPE = 106,46785 kcal/mol

C	1.775378	-0.143603	1.265725
C	0.756330	-1.056627	0.754789
C	-0.375739	-0.501806	-0.127728
C	0.891972	-1.048498	-0.770859
C	1.994458	-0.139289	-1.084417
C	2.550725	0.486812	0.153063
C	-0.727504	0.944650	-0.113872
O	-2.076411	1.062749	0.022489
C	-2.673303	-0.208906	0.283147
C	-1.685221	-1.250069	-0.252854
H	0.481814	-1.953236	1.310098
H	0.726711	-1.945127	-1.368799
H	2.441354	1.582339	0.142013
H	3.632805	0.286984	0.255890
O	-0.002483	1.905572	-0.196148

H	-3.648556	-0.231846	-0.208028
H	-2.819226	-0.311649	1.365339
H	-1.714078	-2.184156	0.318299
H	-1.898518	-1.485553	-1.303426
H	2.000352	-0.017066	2.316069
H	2.385928	0.016755	-2.080146

BR3a E = -498,7130882 a.u. ZPE = 106,82345 kcal/mol

C	-2.210115	-0.397435	0.651112
C	-0.878622	-0.374092	1.262737
C	0.201681	-0.386788	0.229089
C	-0.538290	-0.685765	-1.048449
C	-1.986441	-0.587630	-0.855289
C	-2.519225	0.726023	-0.316522
C	1.396997	-1.322272	0.493271
C	2.497934	-0.662121	-0.330967
O	2.231848	0.740166	-0.245087
C	0.934811	0.966666	0.069750
O	0.473442	2.074368	0.172728
H	-3.042969	-0.871250	1.165236
H	-0.678338	-0.265003	2.321179
H	-0.044938	-0.907020	-1.987378
H	-2.657510	-1.197016	-1.455629
H	-3.570462	0.932464	-0.503485
H	-1.857907	1.588475	-0.314557
H	1.197715	-2.356808	0.201998
H	1.653710	-1.295921	1.559252
H	2.458702	-0.966733	-1.384695
H	3.507298	-0.836977	0.048567

BR3b E = -498,7096806 a.u. ZPE = 106,57487 kcal/mol

C	-2.213243	0.583032	-0.465937
C	-0.893350	0.552777	-1.101029
C	0.132261	-0.069859	-0.210343
C	-0.597243	-0.271278	1.093719
C	-2.019581	0.051169	0.961939
C	-2.799950	-0.751434	-0.059875
C	1.340767	0.853892	0.053755
O	2.440908	0.089323	0.267936
C	2.136130	-1.299818	0.116209
C	0.860992	-1.331298	-0.721325
H	-2.903515	1.396130	-0.675661
H	-0.638701	1.056364	-2.024095
H	-0.103932	-0.536158	2.020865
H	-2.568718	0.468481	1.802365
H	-3.878276	-0.798768	0.071657
H	-2.343881	-1.655254	-0.459649
O	1.359825	2.054658	0.092147
H	1.980754	-1.742242	1.108450
H	2.994411	-1.783096	-0.356322
H	1.100048	-1.215275	-1.785465
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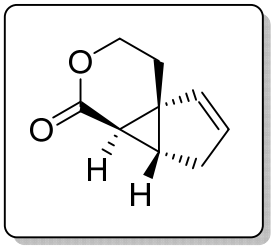
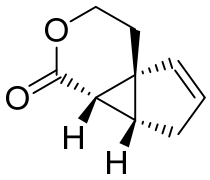
Figure S3: Geometries and energies of P1, P2



P1a



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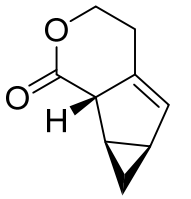
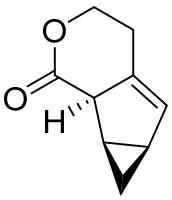


P2a



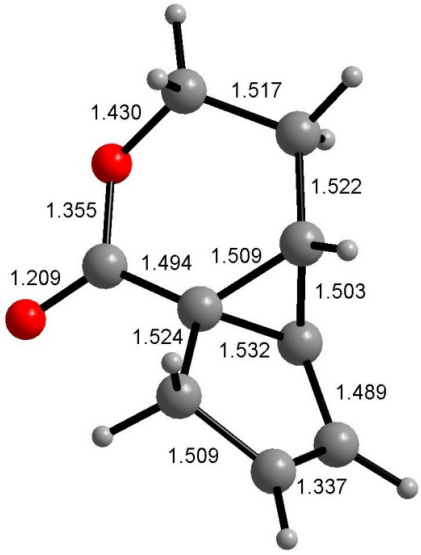
P2b

E_{REL} = 0.0 kcal/mol

E_{REL} = 28.9 kcal/mol

E_{REL} = 3.0 kcal/mol

E_{REL} = 1.7 kcal/mol



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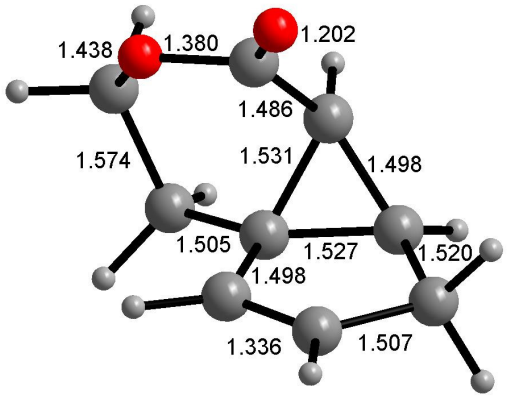

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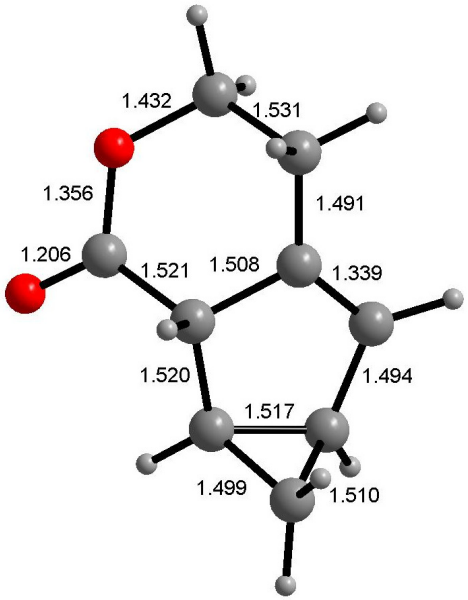

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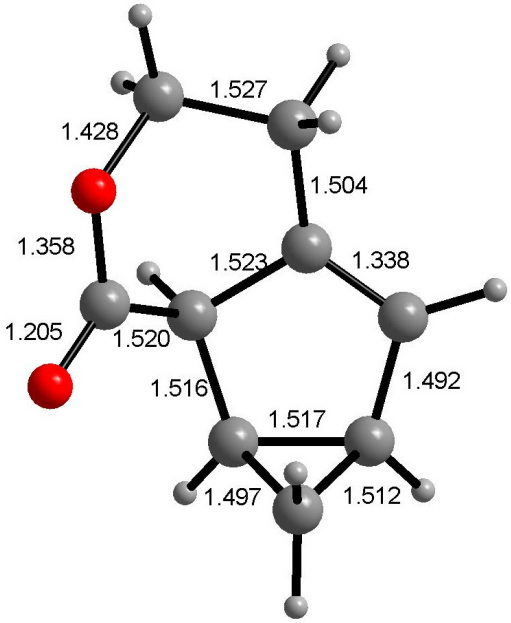


P2b

P1a E = -498,8108113 a.u. ZPE = 110,07418 kcal/mol

```
C          -2.331243   -0.732336   -0.671843
C          -2.686134    0.083939    0.326390
```

C	-0.854210	-0.756875	-0.859310
C	-0.308597	0.217706	0.189715
C	-1.507758	0.826852	0.905415
C	-0.051713	-1.265059	0.305514
H	-0.409032	-0.823203	-1.851097
C	0.800064	1.143821	-0.190790
H	-1.437392	0.721516	1.996486
C	1.385508	-1.700936	0.061273
H	-0.611569	-1.787190	1.081086
H	-3.699130	0.216213	0.696167
C	2.325684	-0.576666	0.453808
O	2.042704	0.611942	-0.288650
O	0.621479	2.311877	-0.445083
H	1.543933	-1.945051	-0.996878
H	1.621201	-2.600442	0.640203
H	3.370117	-0.813112	0.235867
H	2.237715	-0.359015	1.527052
H	-3.007408	-1.343852	-1.262346
H	-1.551502	1.900917	0.682291

P1b E = -498,7640902 a.u. ZPE = 109,68662 kcal/mol

C	-2.331243	-0.732336	-0.671843
C	-2.686134	0.083939	0.326390
C	-0.854210	-0.756875	-0.859310
C	-0.308597	0.217706	0.189715
C	-1.507758	0.826852	0.905415
C	-0.051713	-1.265059	0.305514
H	-0.409032	-0.823203	-1.851097
C	0.800064	1.143821	-0.190790
H	-1.437392	0.721516	1.996486
C	1.385508	-1.700936	0.061273
H	-0.611569	-1.787190	1.081086
H	-3.699130	0.216213	0.696167
C	2.325684	-0.576666	0.453808
O	2.042704	0.611942	-0.288650
O	0.621479	2.311877	-0.445083
H	1.543933	-1.945051	-0.996878
H	1.621201	-2.600442	0.640203
H	3.370117	-0.813112	0.235867
H	2.237715	-0.359015	1.527052
H	-3.007408	-1.343852	-1.262346
H	-1.551502	1.900917	0.682291

P2a E = -498,8059516 a.u. ZPE = 109,98435 kcal/mol

C	-2.331243	-0.732336	-0.671843
C	-2.686134	0.083939	0.326390
C	-0.854210	-0.756875	-0.859310
C	-0.308597	0.217706	0.189715
C	-1.507758	0.826852	0.905415
C	-0.051713	-1.265059	0.305514
H	-0.409032	-0.823203	-1.851097
C	0.800064	1.143821	-0.190790
H	-1.437392	0.721516	1.996486
C	1.385508	-1.700936	0.061273
H	-0.611569	-1.787190	1.081086
H	-3.699130	0.216213	0.696167
C	2.325684	-0.576666	0.453808
O	2.042704	0.611942	-0.288650
O	0.621479	2.311877	-0.445083
H	1.543933	-1.945051	-0.996878
H	1.621201	-2.600442	0.640203

H	3.370117	-0.813112	0.235867
H	2.237715	-0.359015	1.527052
H	-3.007408	-1.343852	-1.262346
H	-1.551502	1.900917	0.682291

P2b E = -498,8080405 a.u. ZPE = 110,07525 kcal/mol

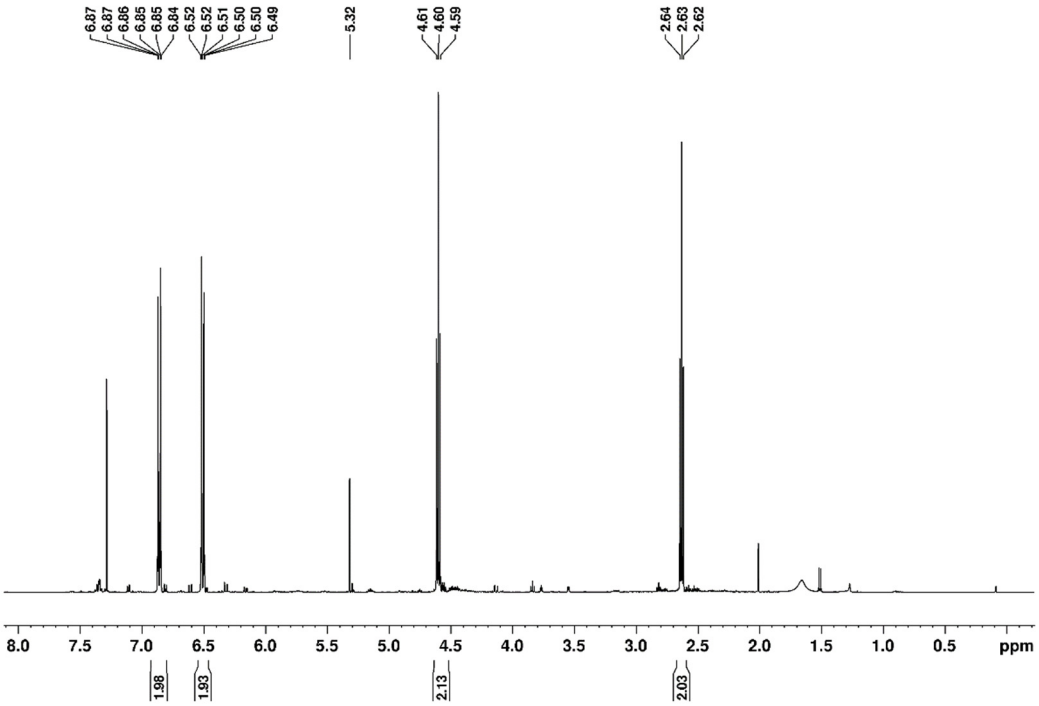
C	-2.331243	-0.732336	-0.671843
C	-2.686134	0.083939	0.326390
C	-0.854210	-0.756875	-0.859310
C	-0.308597	0.217706	0.189715
C	-1.507758	0.826852	0.905415
C	-0.051713	-1.265059	0.305514
H	-0.409032	-0.823203	-1.851097
C	0.800064	1.143821	-0.190790
H	-1.437392	0.721516	1.996486
C	1.385508	-1.700936	0.061273
H	-0.611569	-1.787190	1.081086
H	-3.699130	0.216213	0.696167
C	2.325684	-0.576666	0.453808
O	2.042704	0.611942	-0.288650
O	0.621479	2.311877	-0.445083
H	1.543933	-1.945051	-0.996878
H	1.621201	-2.600442	0.640203
H	3.370117	-0.813112	0.235867
H	2.237715	-0.359015	1.527052
H	-3.007408	-1.343852	-1.262346
H	-1.551502	1.900917	0.682291

2. Photochemical Setup (Figure S4)

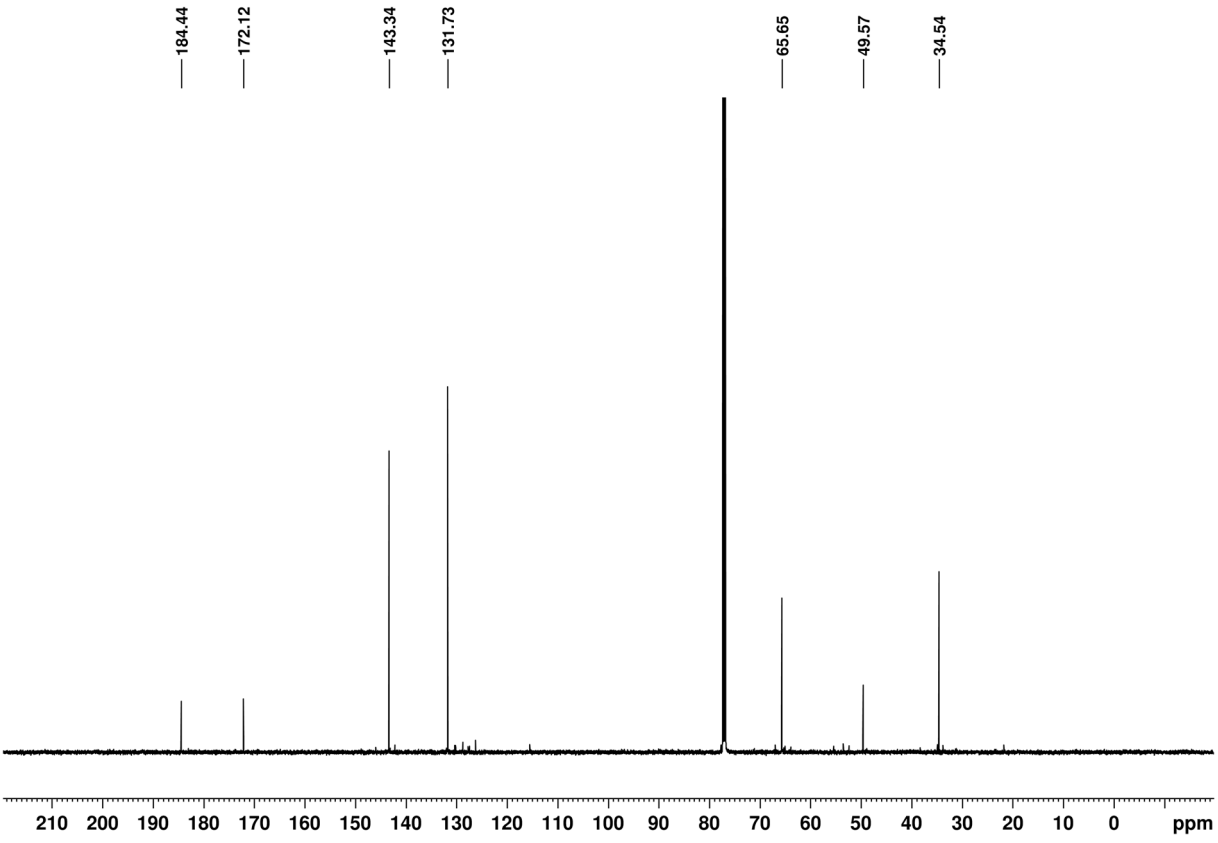
3. NMR Spectra

3.1. Side product 12a

^{1}H NMR spectrum (500 MHz, $CDCl_3$) of 12a

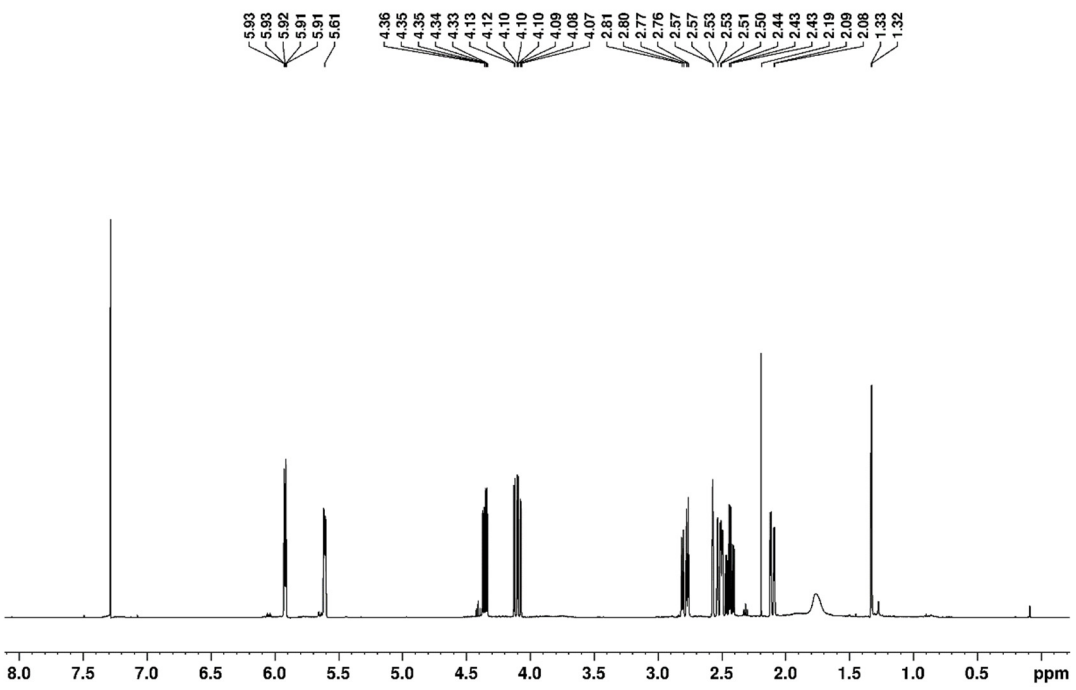


^{13}C NMR spectrum (125 MHz)

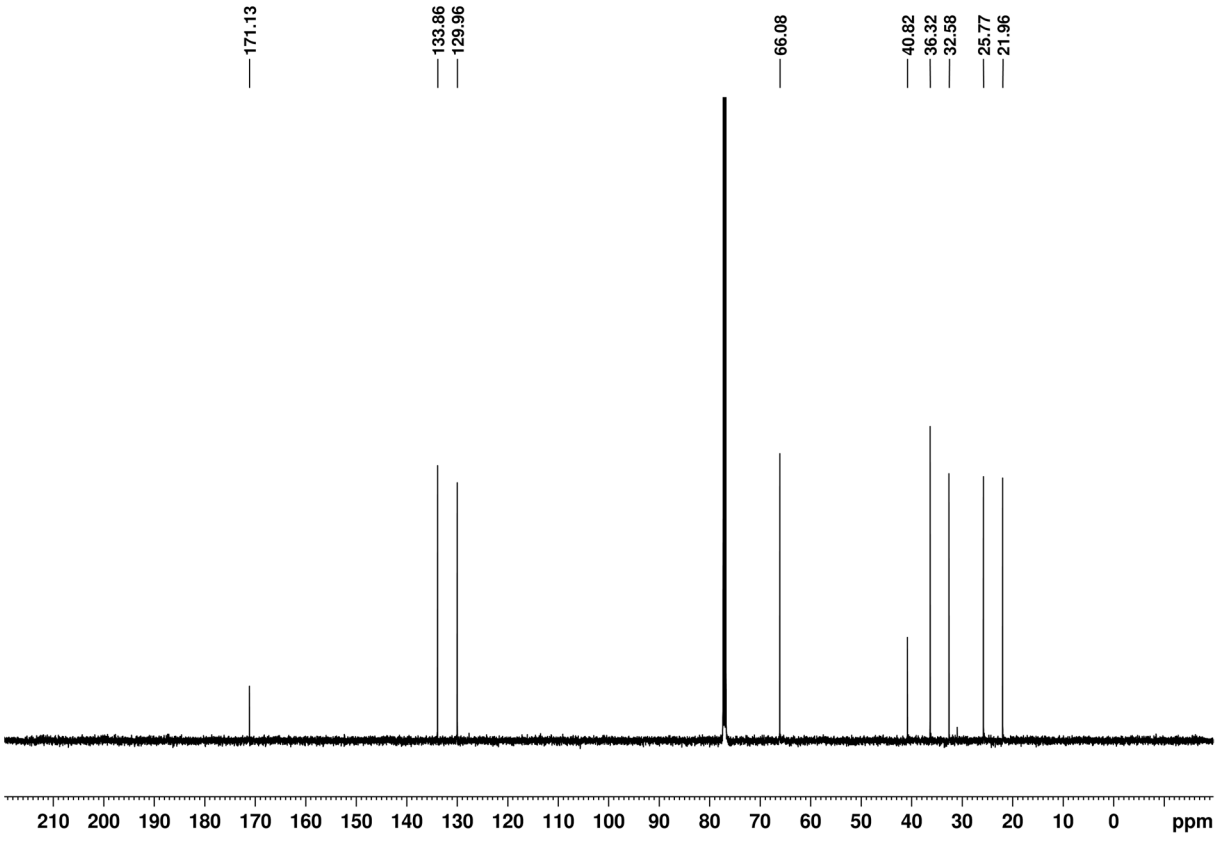


3.2. Oxa-Di-π-Methane Rearrangement Products 13

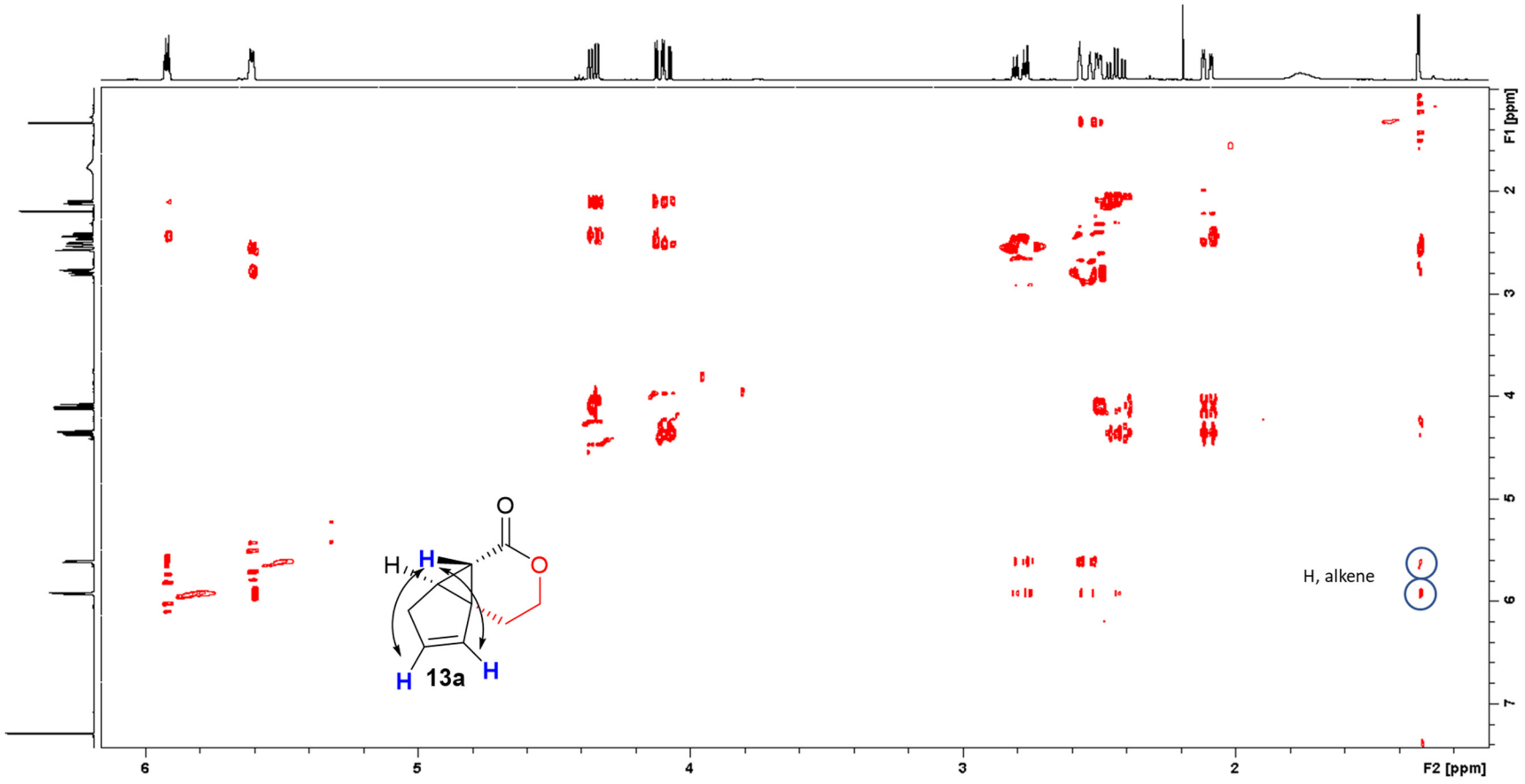
^{1}H NMR spectrum (500 MHz, $CDCl_3$) of 13a



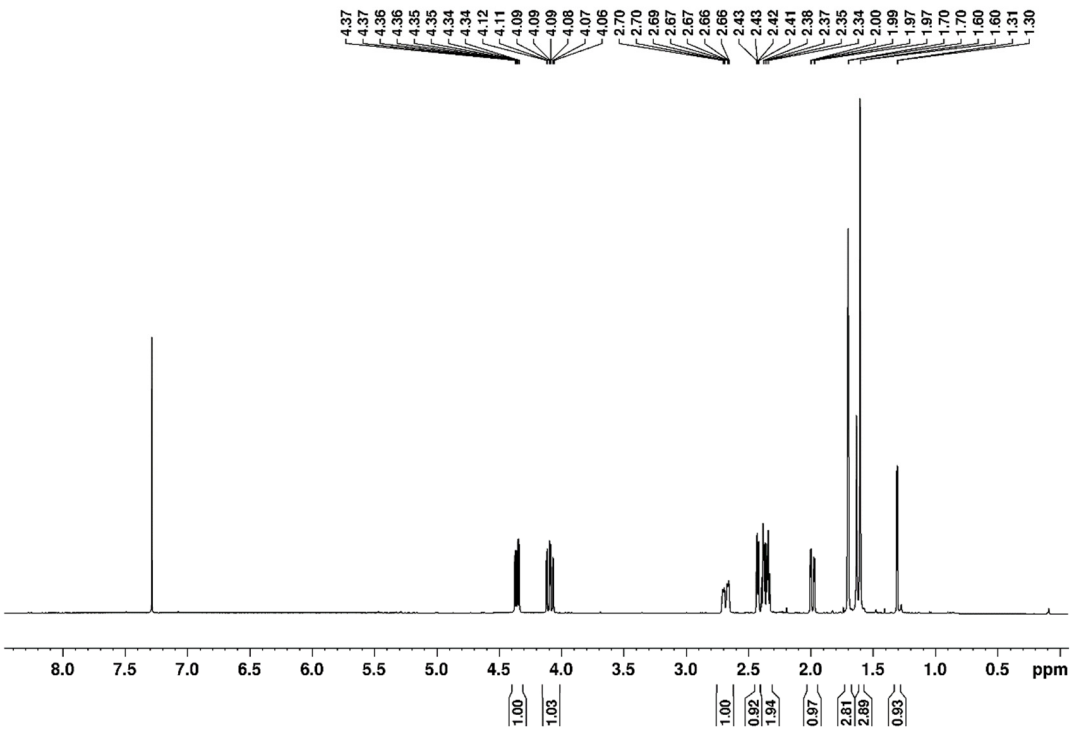
^{13}C NMR spectrum (125 MHz)



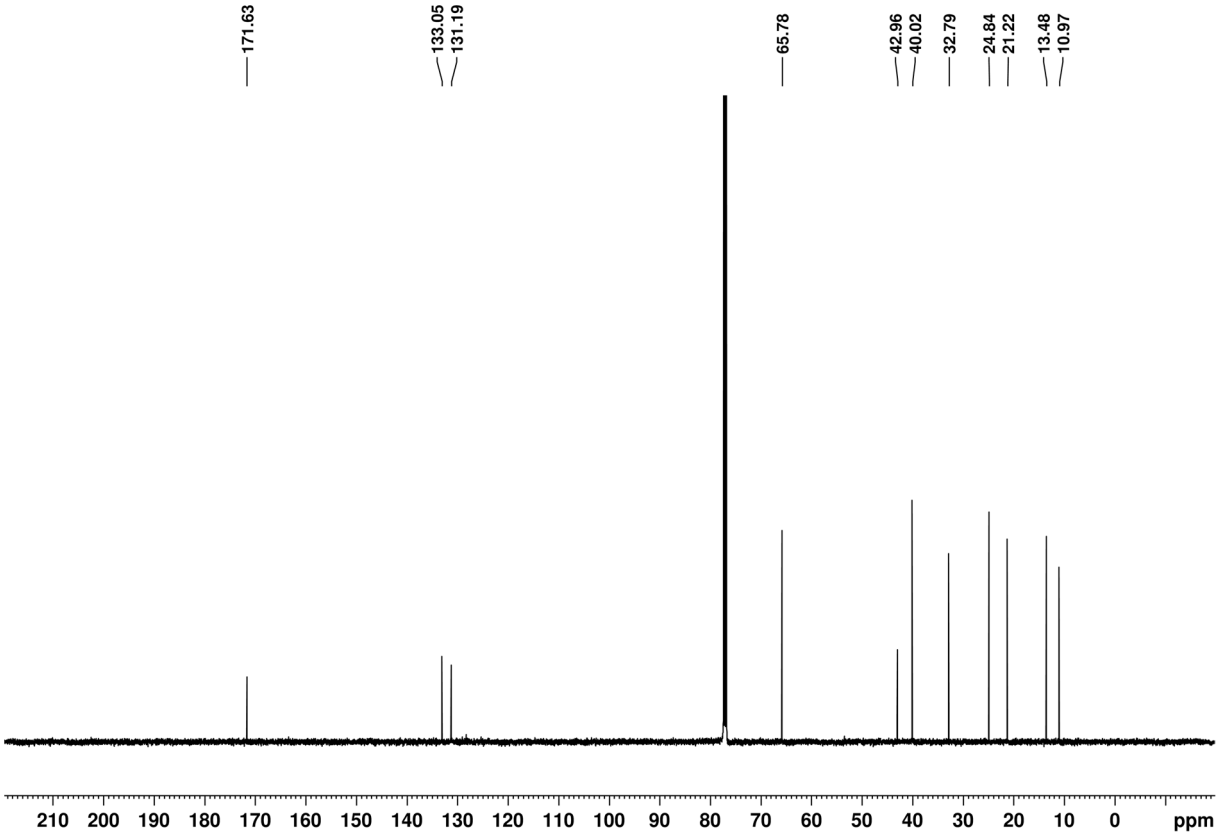
NOESY of 13a



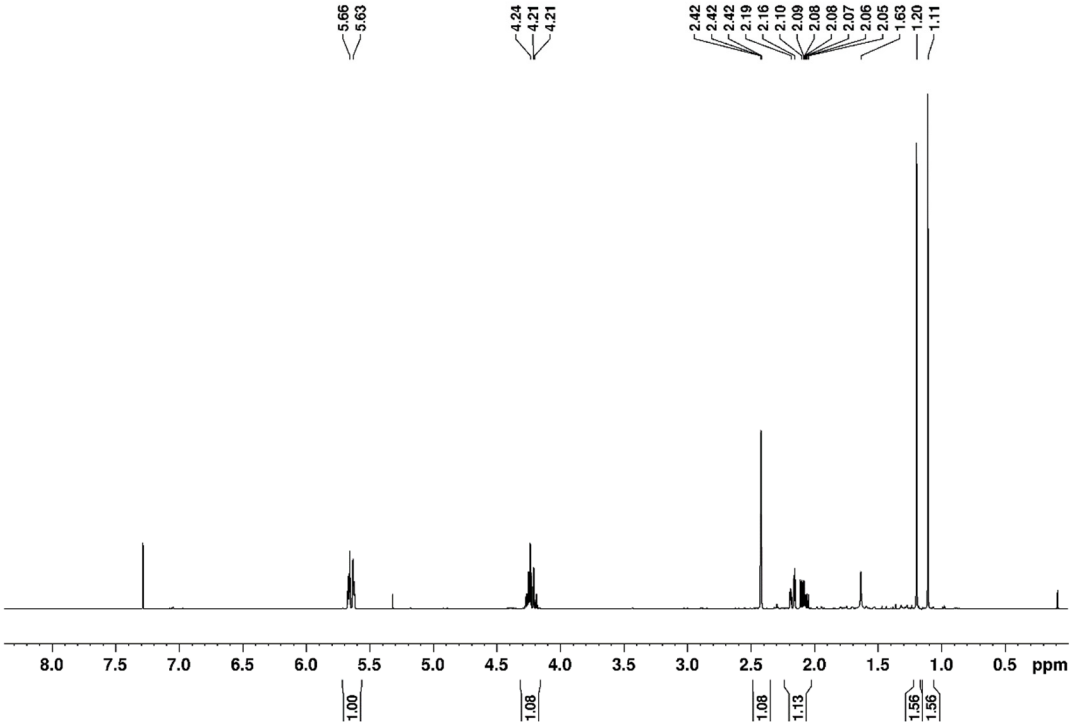
^{1}H NMR spectrum (500 MHz, $CDCl_3$) of 13ba



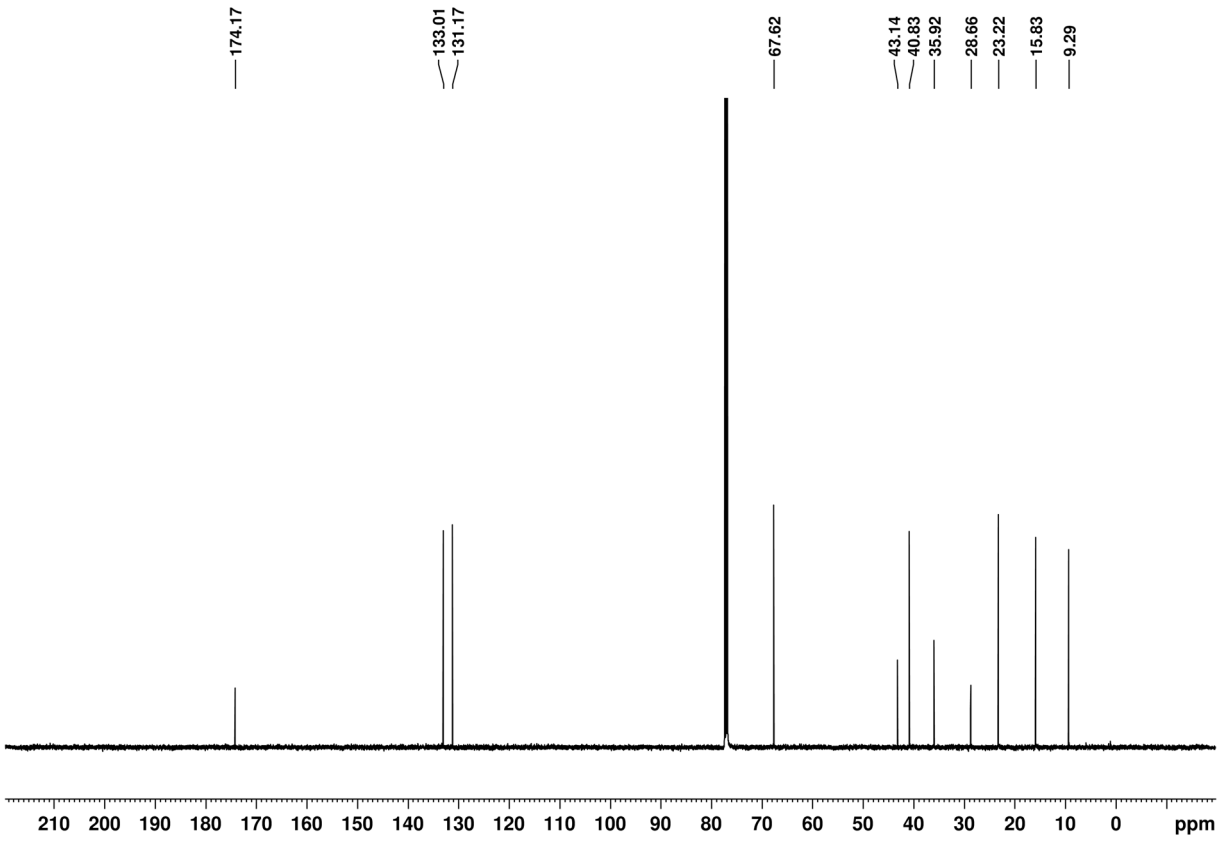
^{13}C NMR spectrum (125 MHz)



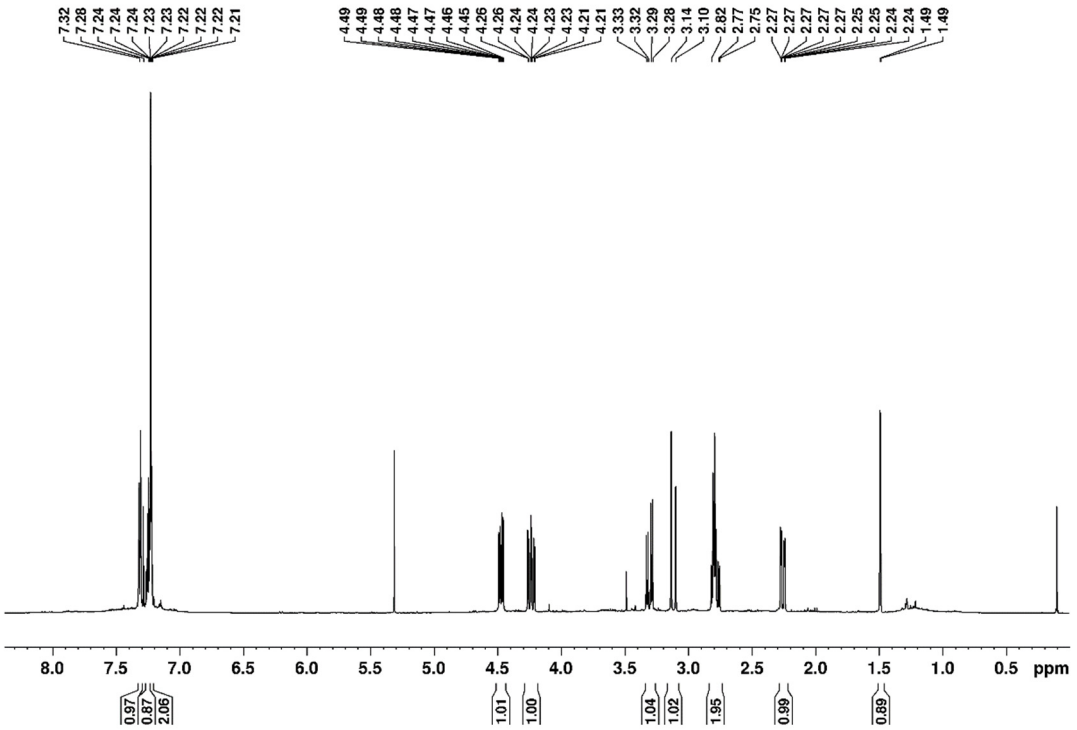
^{1}H NMR spectrum (500 MHz, $CDCl_3$) of 13bb



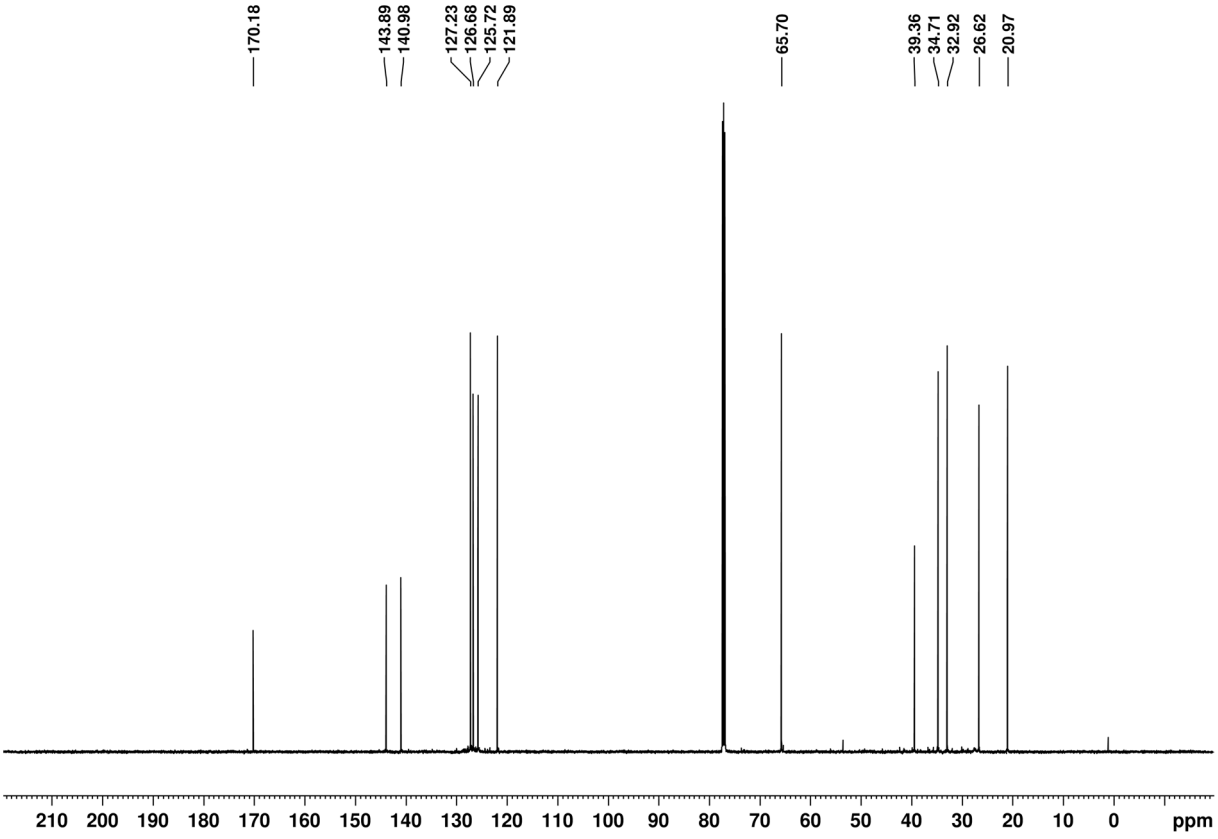
^{13}C NMR spectrum (125 MHz)



^{1}H NMR spectrum (500 MHz, $CDCl_3$) of 13c

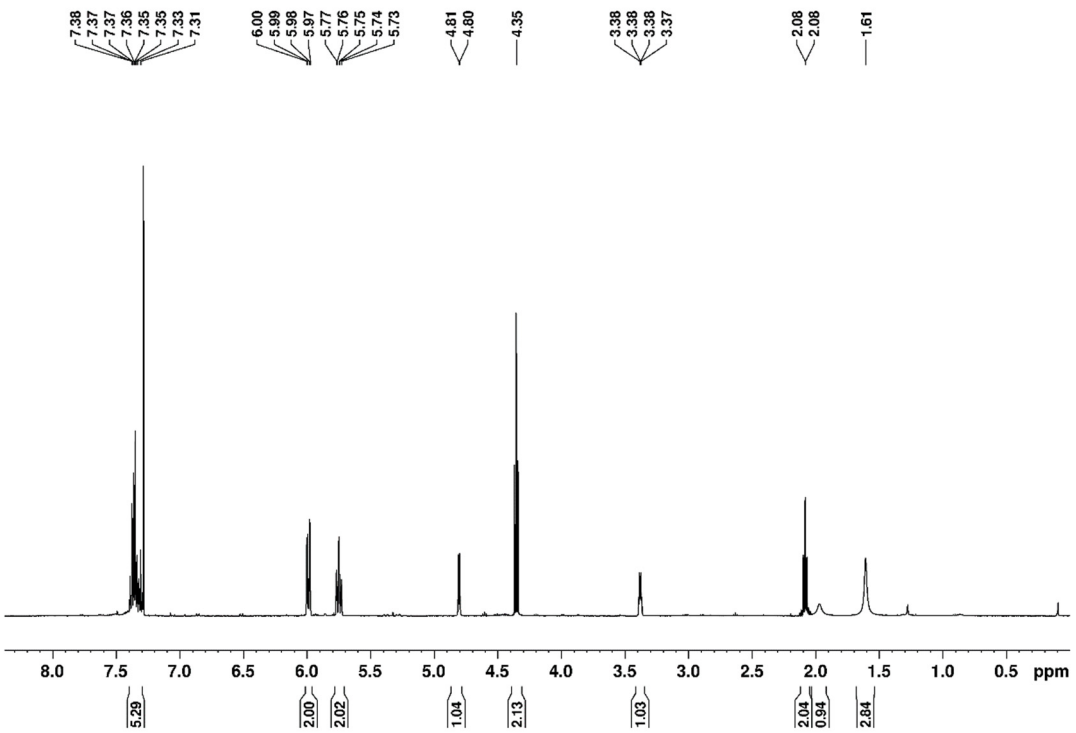


^{13}C NMR spectrum (125 MHz)

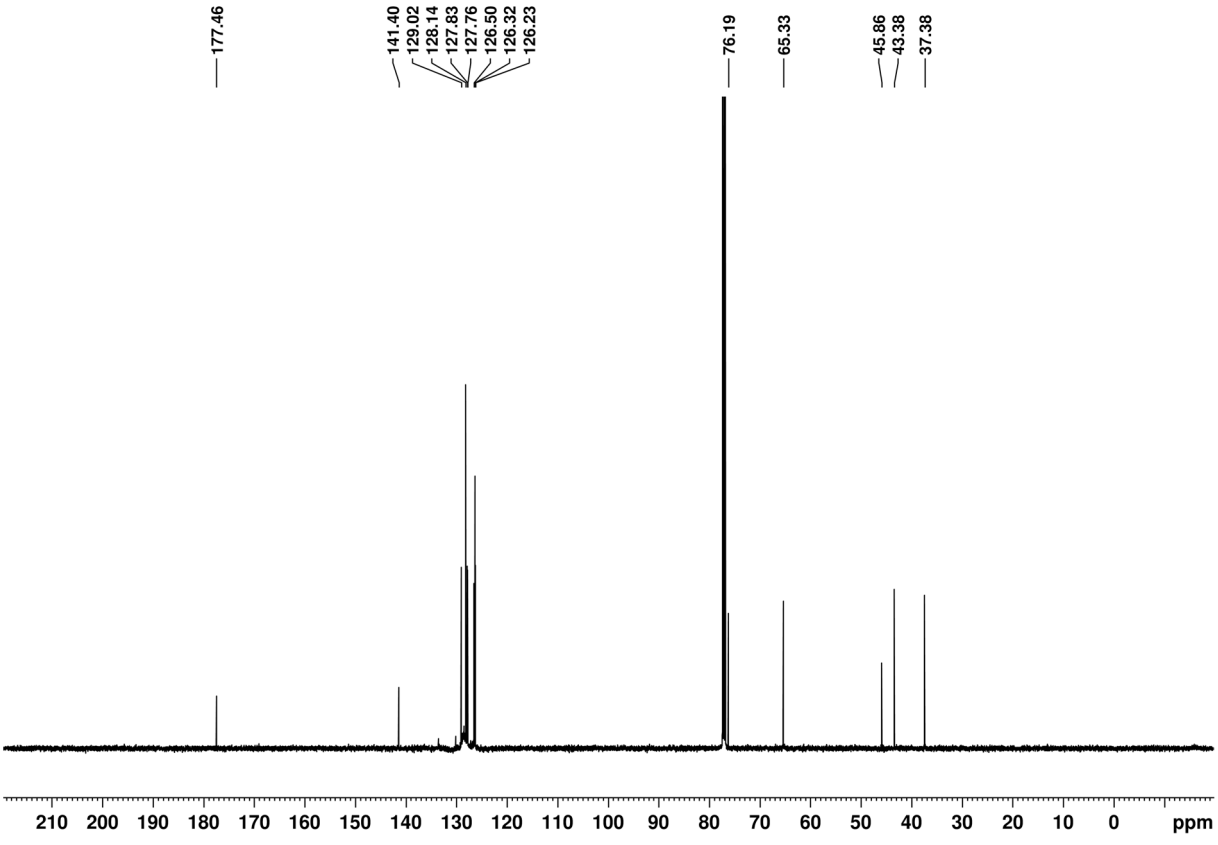


3.3. Products 20 and 26 from Reactions with Benzaldehyde (18)

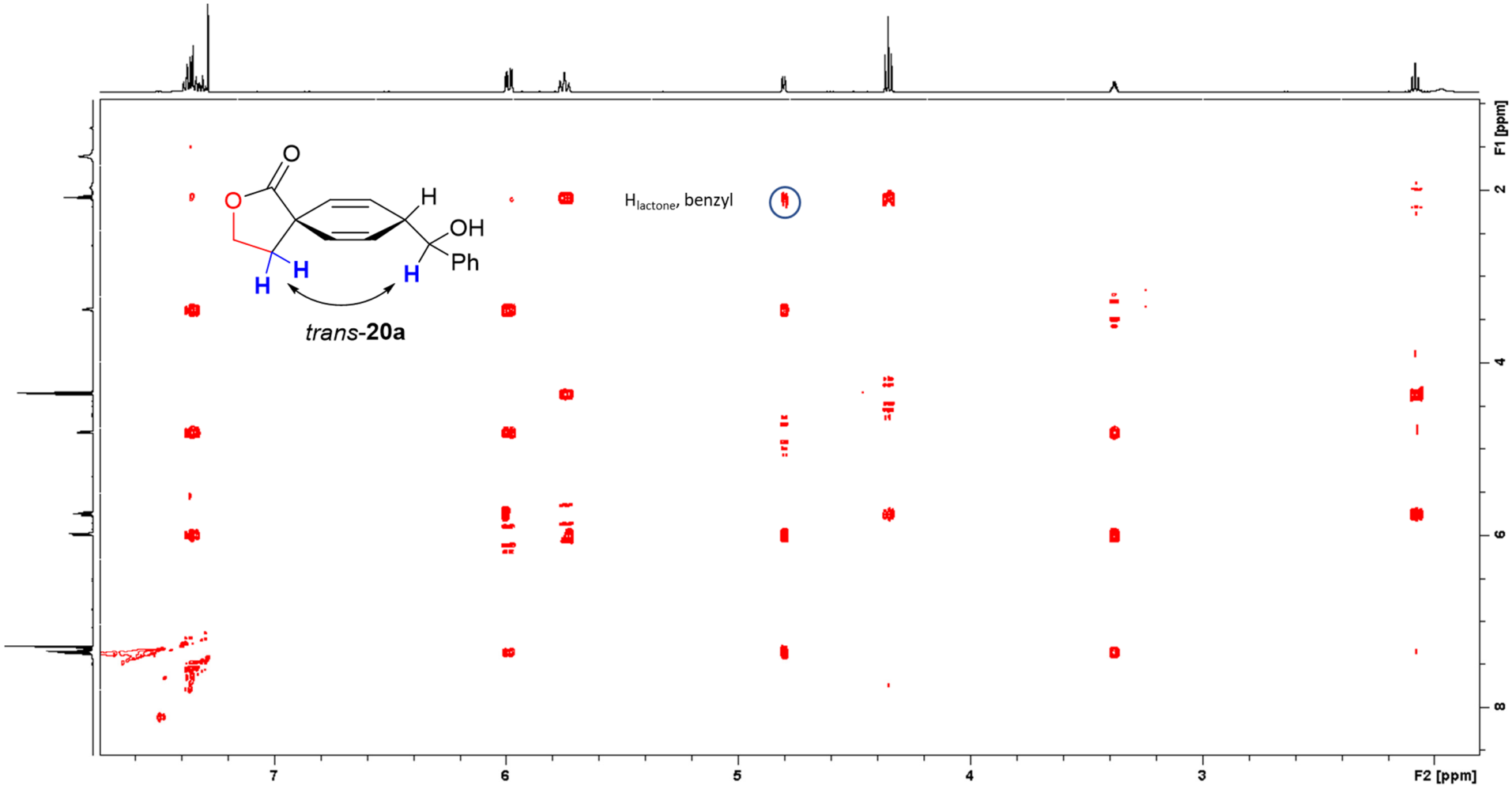
^{1}H NMR spectrum (500 MHz, $CDCl_3$) of *trans*-20a



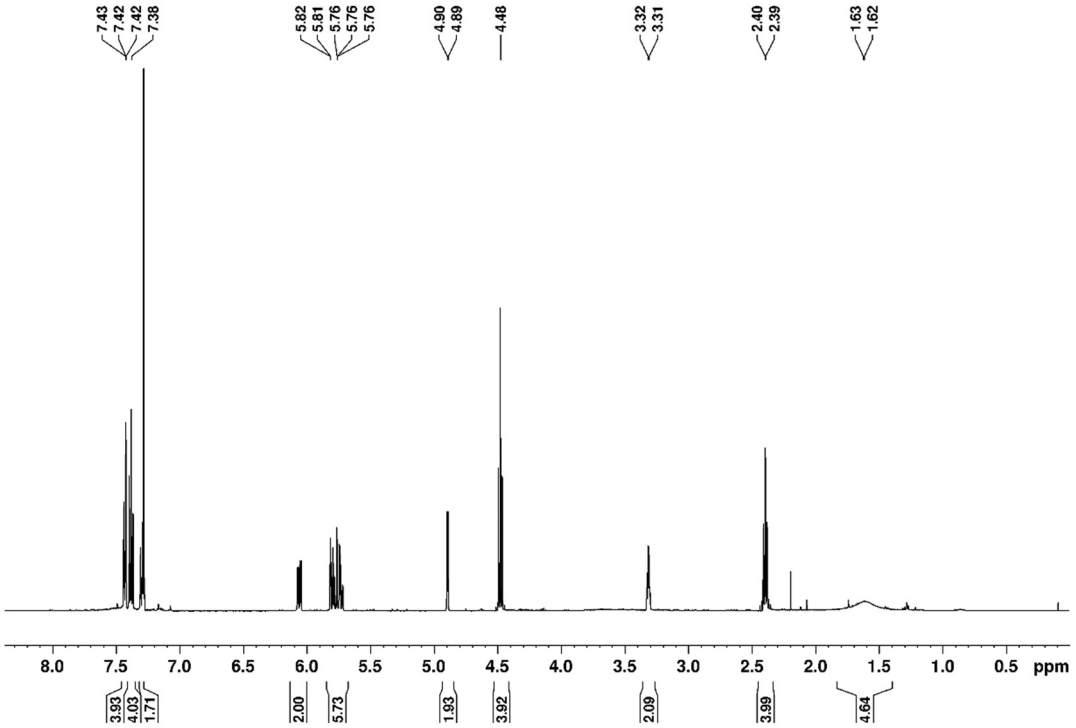
^{13}C NMR spectrum (125 MHz)



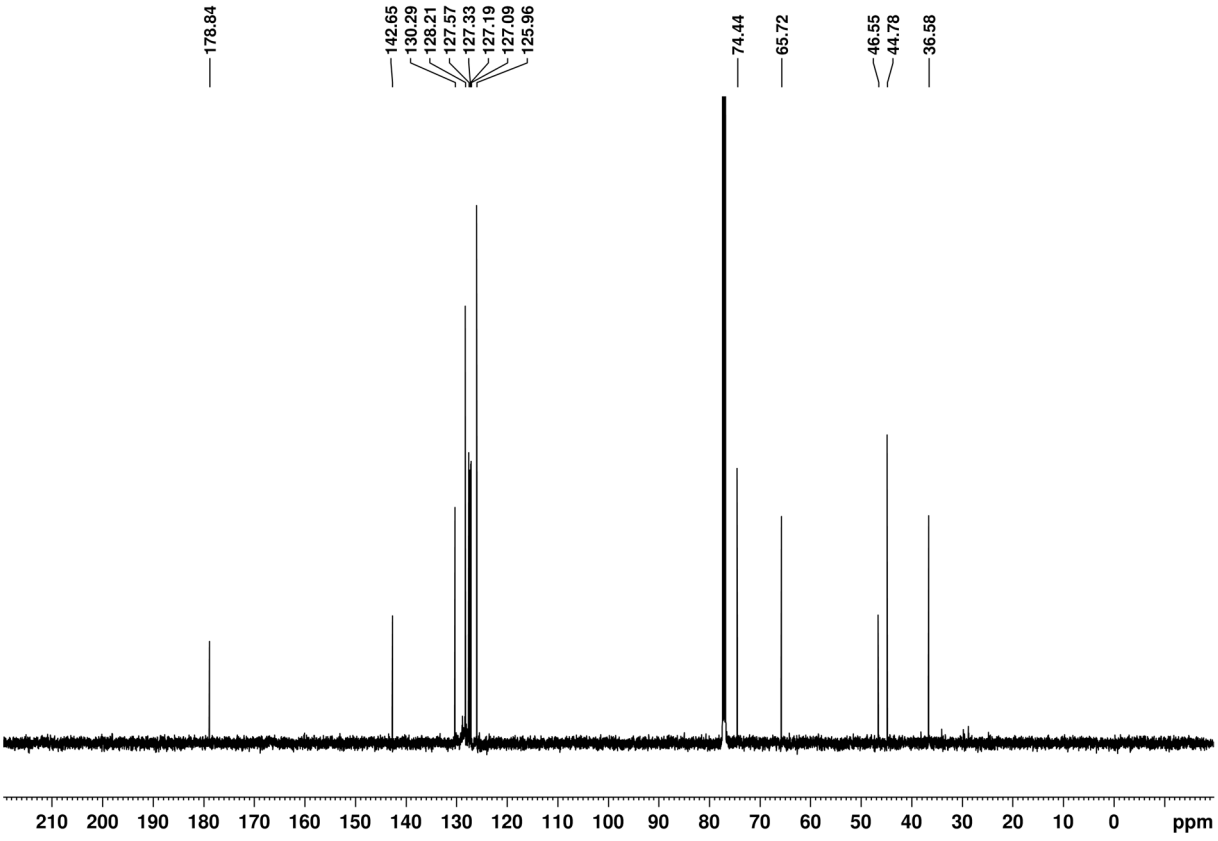
NOESY of *trans*-20a



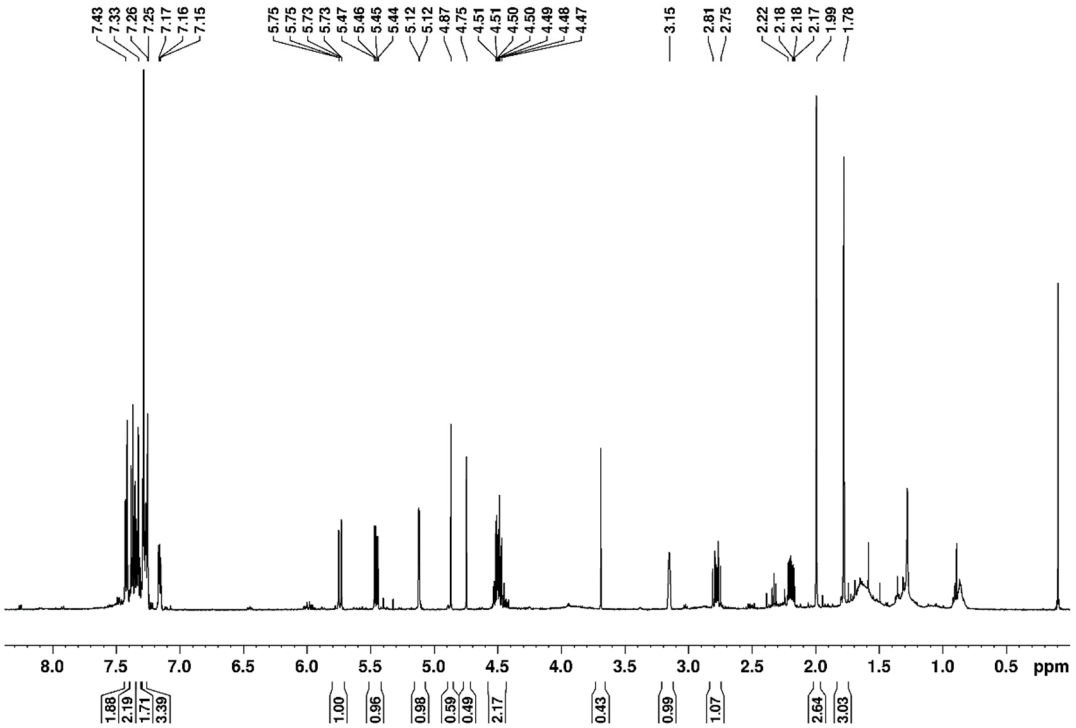
^{1}H NMR spectrum (500 MHz, $CDCl_3$) of *cis*-20a



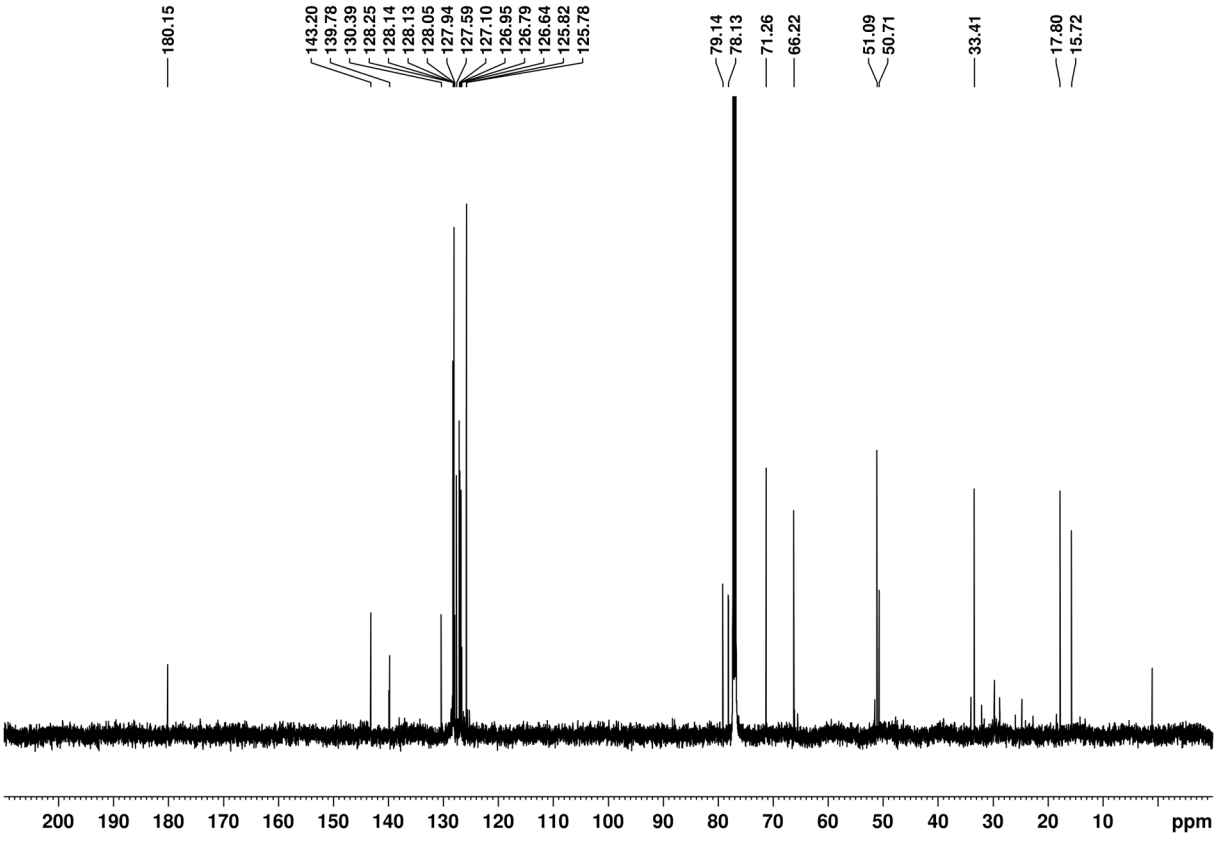
^{13}C NMR spectrum (125 MHz)



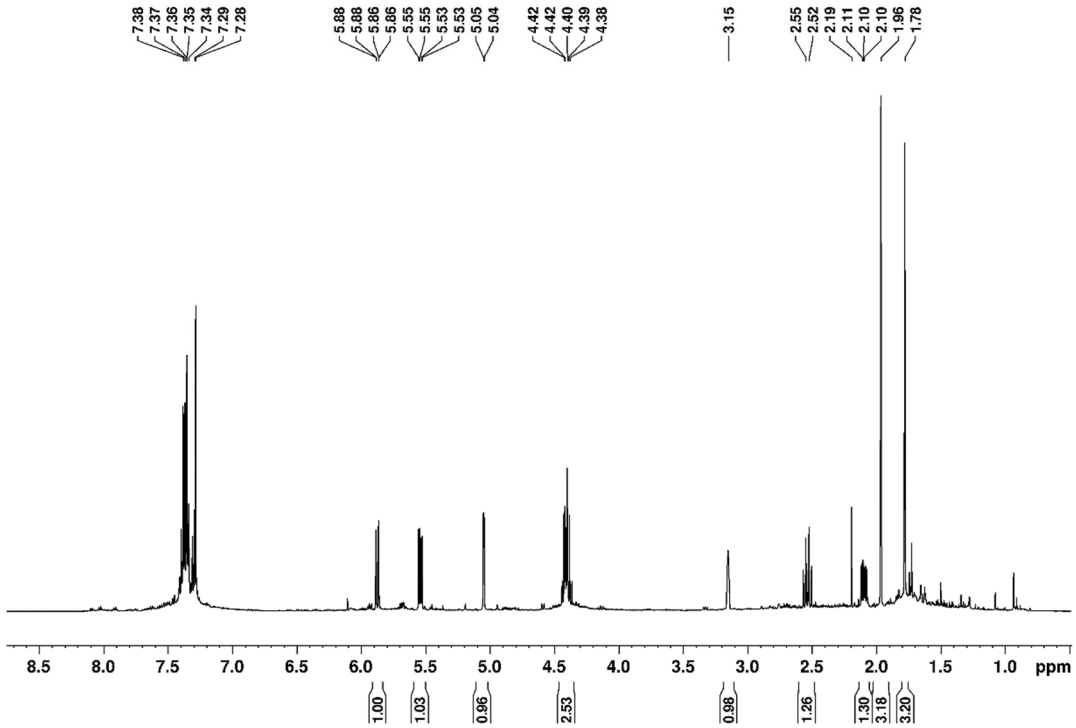
^{1}H NMR spectrum (500 MHz, $CDCl_3$) of *trans*-20b



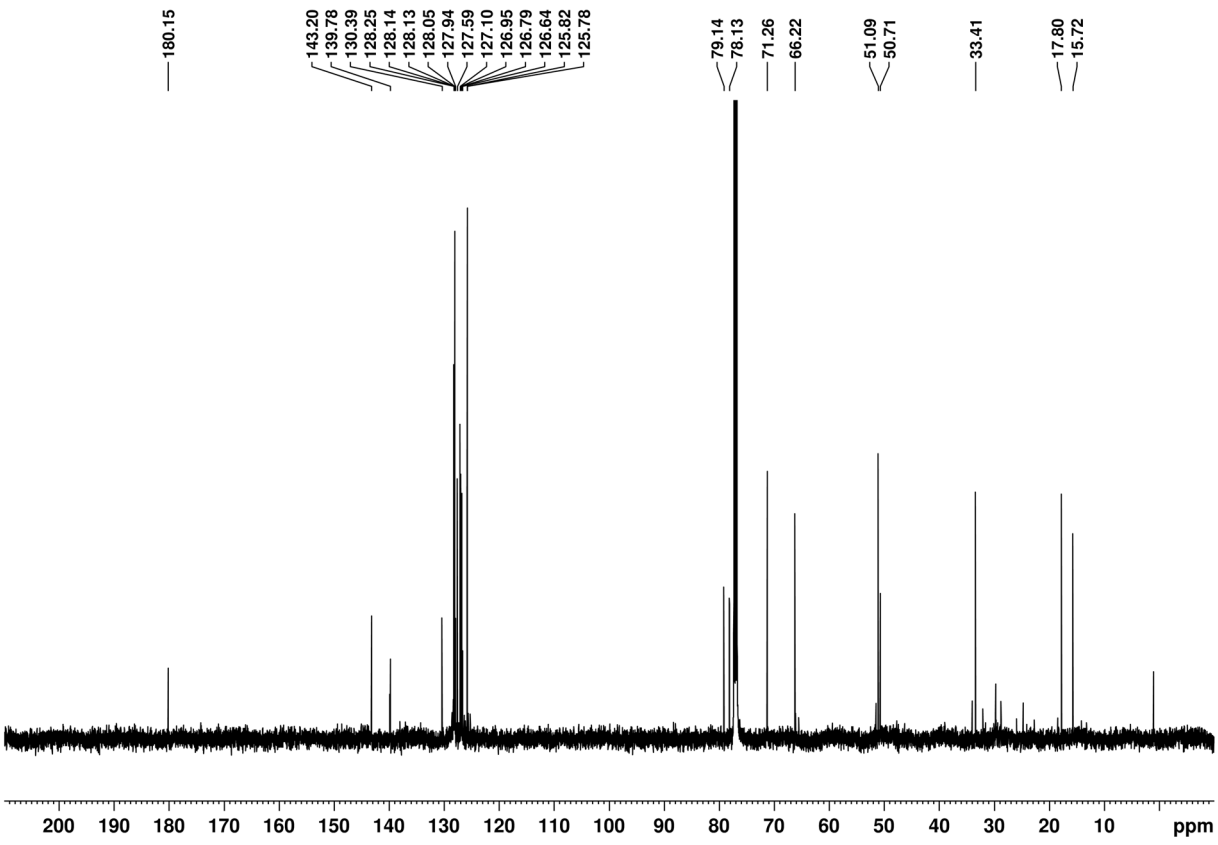
^{13}C NMR spectrum (125 MHz)



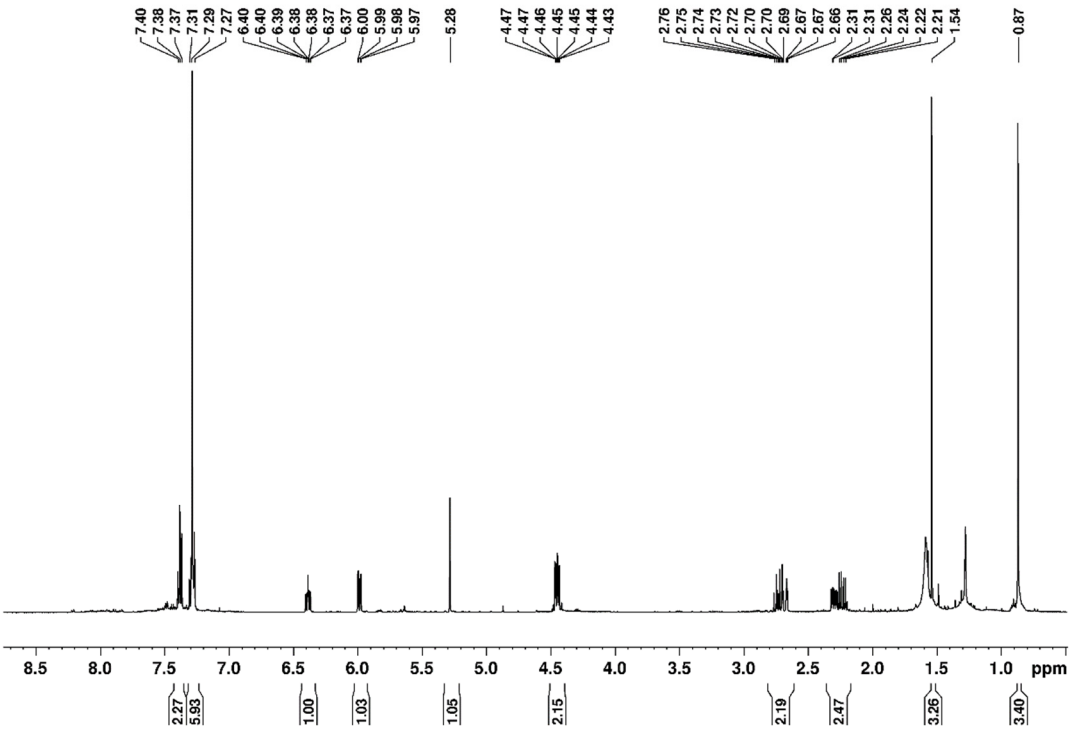
^{1}H NMR spectrum (500 MHz, $CDCl_3$) of *cis*-20b



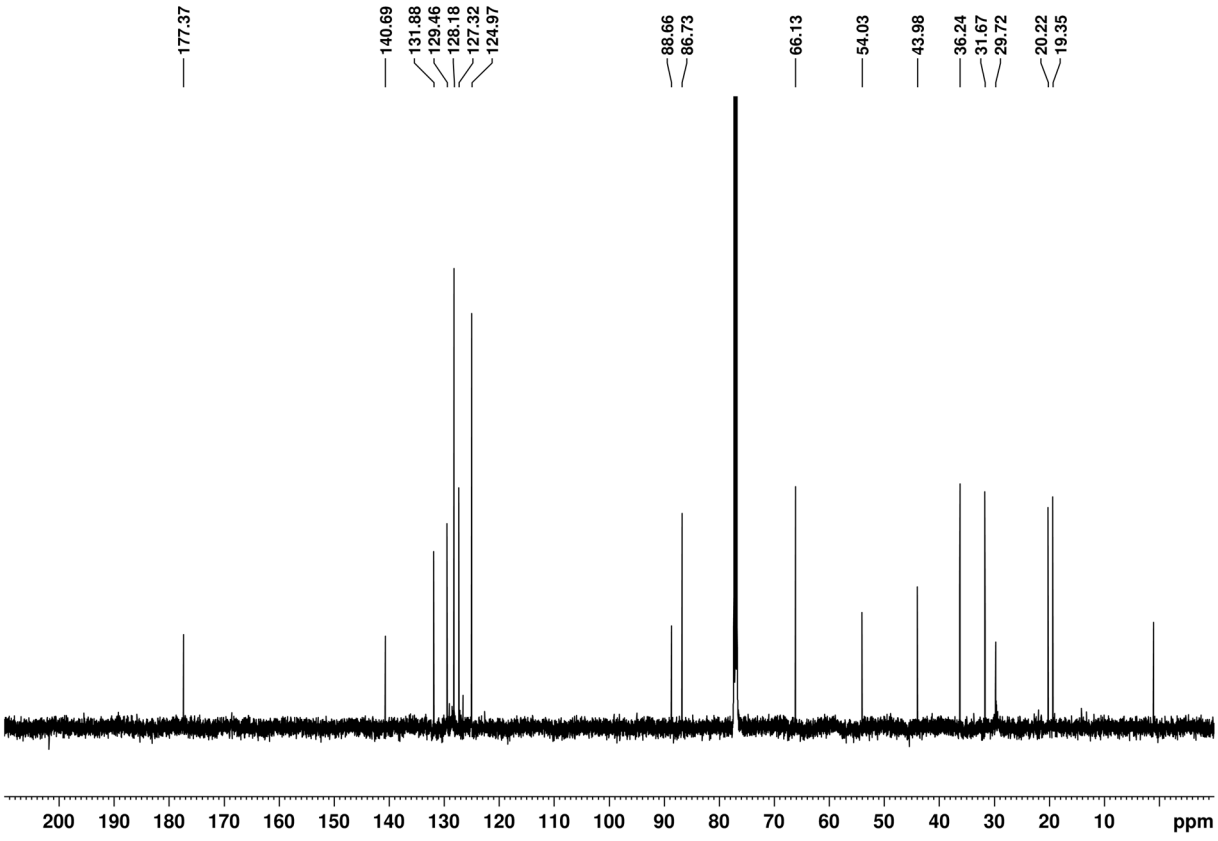
^{13}C NMR spectrum (125 MHz)



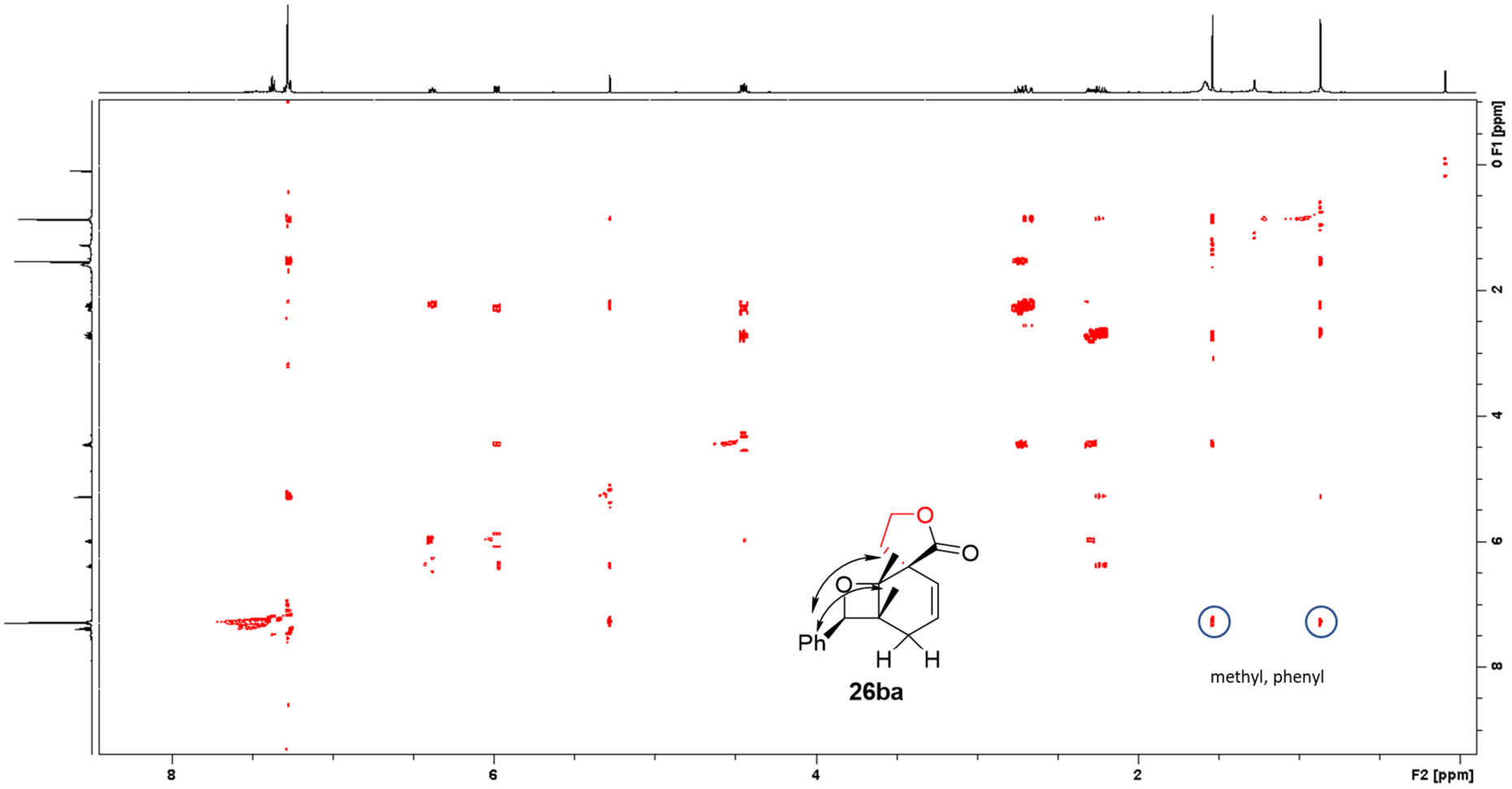
^{1}H NMR spectrum (500 MHz, $CDCl_3$) of 26ba



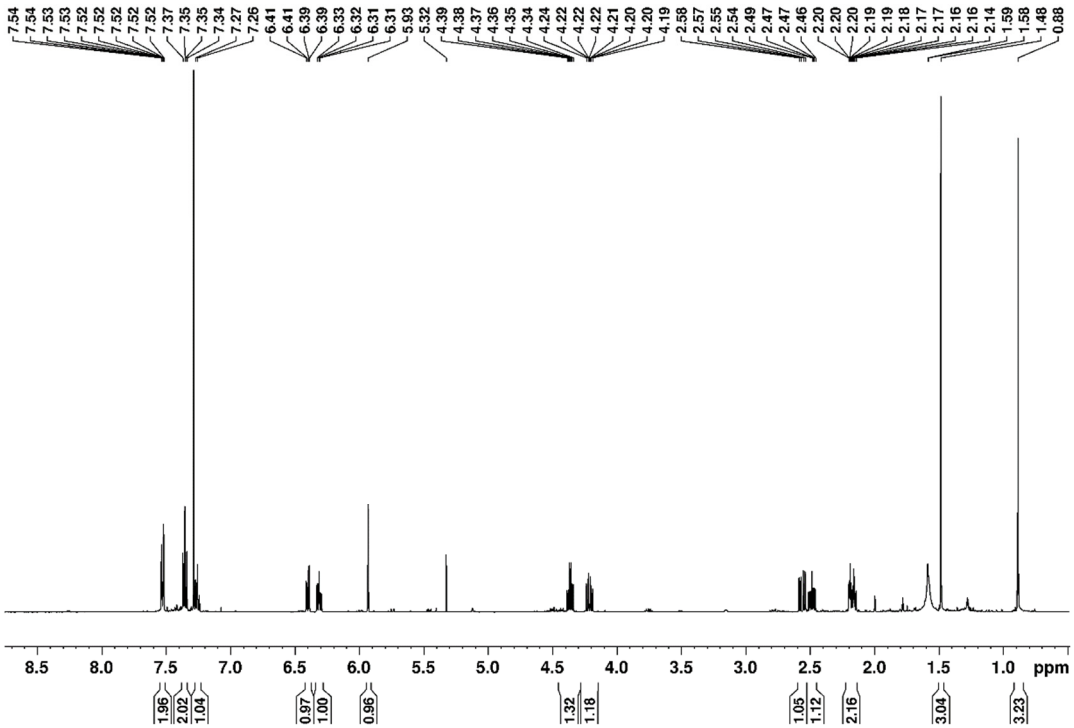
^{13}C NMR spectrum (125 MHz)



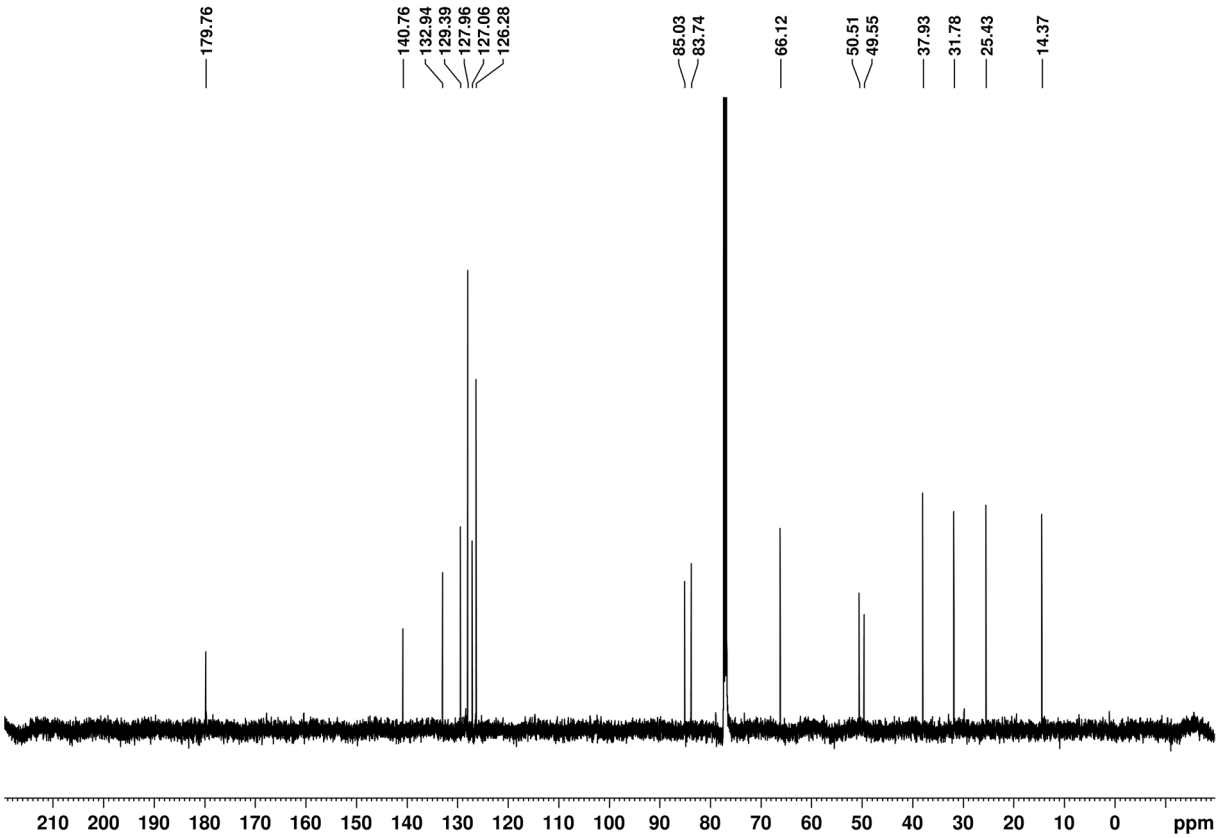
NOESY of 26ba



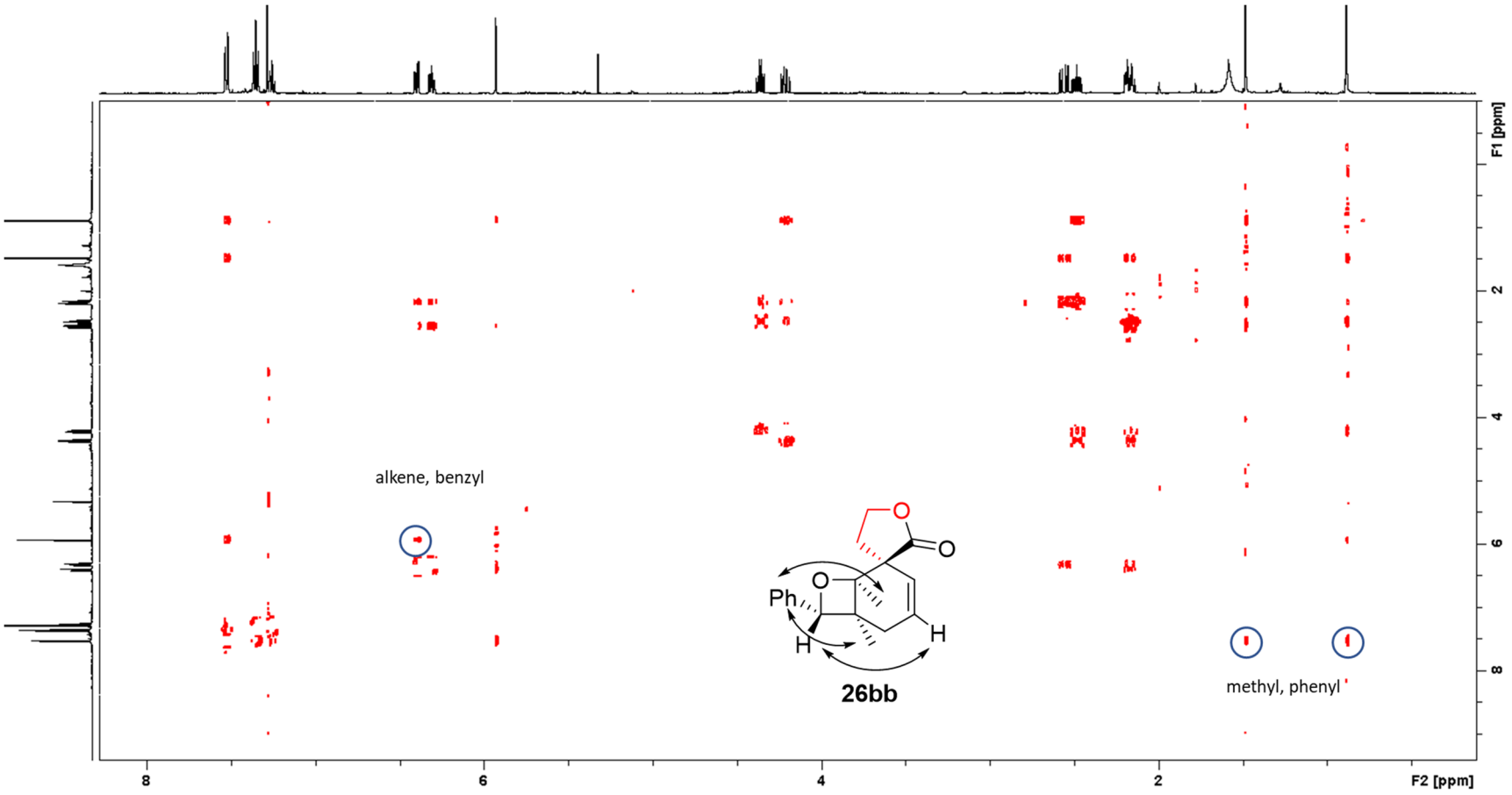
^{1}H NMR spectrum (500 MHz, $CDCl_3$) of 26bb



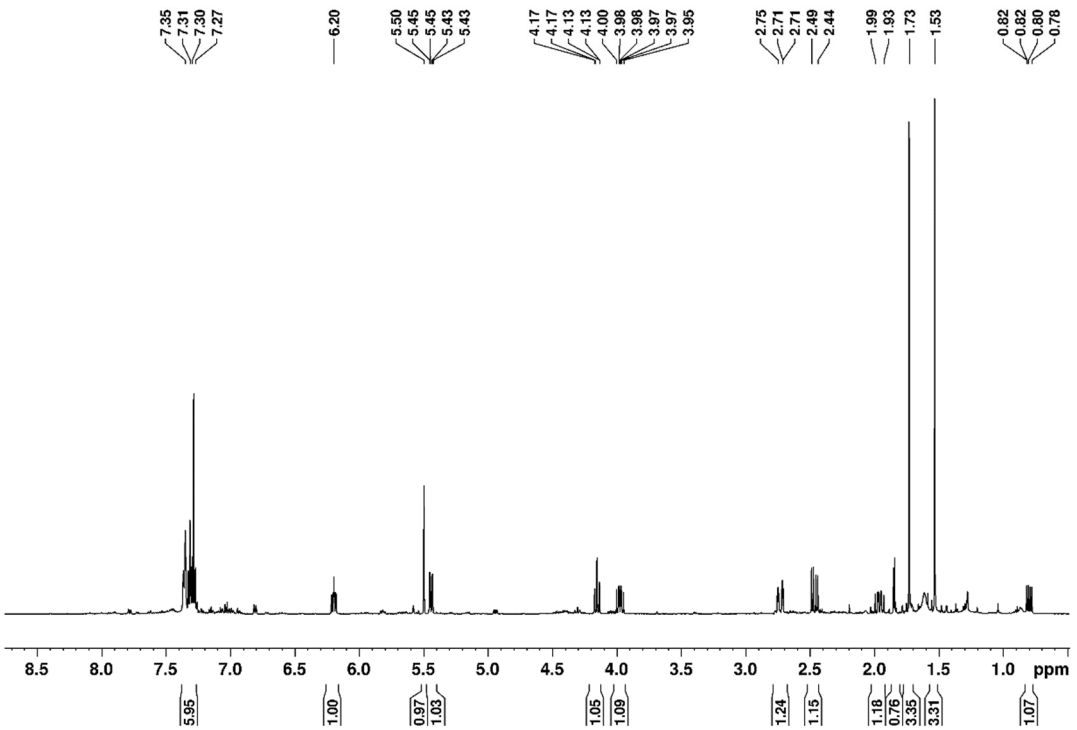
^{13}C NMR spectrum (125 MHz)



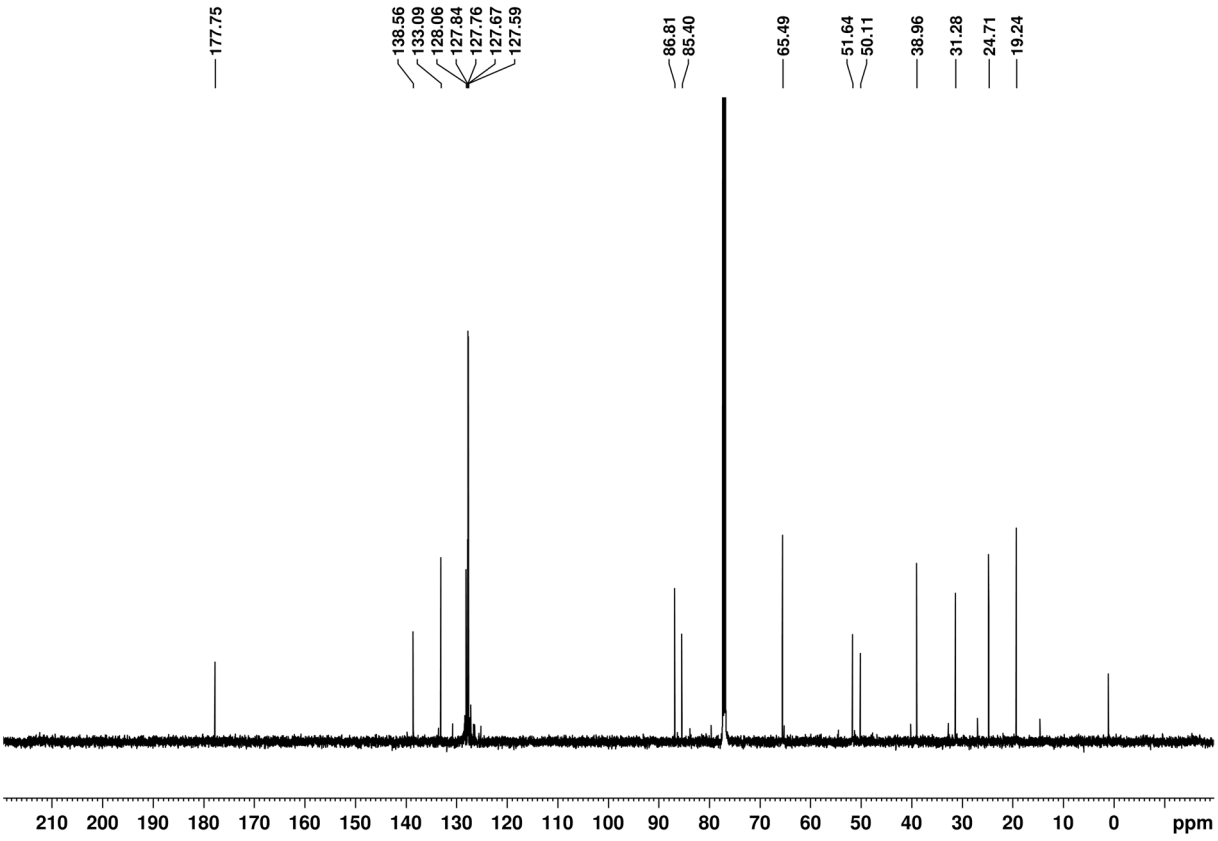
NOESY of 26bb



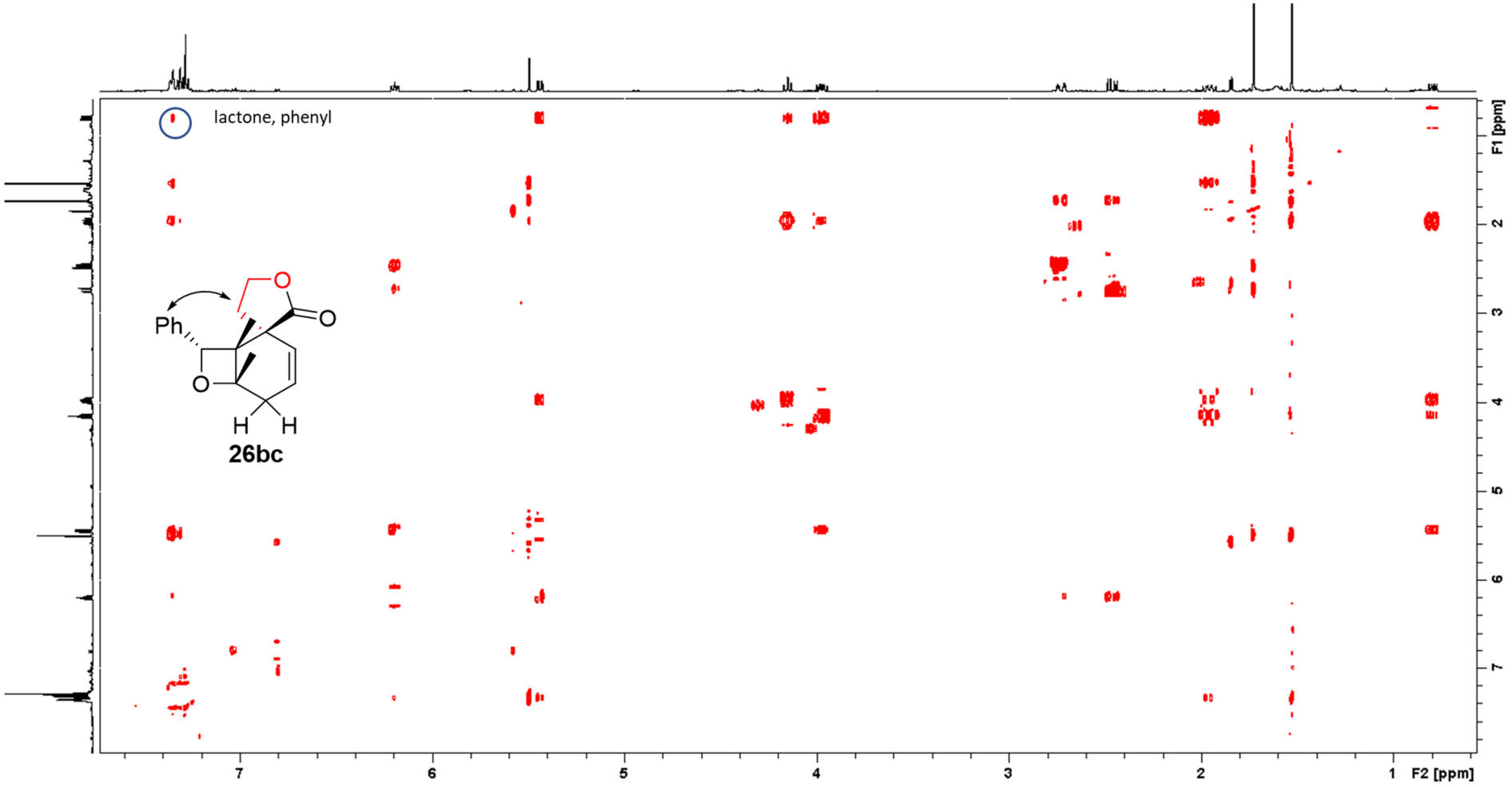
^{1}H NMR spectrum (500 MHz, $CDCl_3$) of 26bc



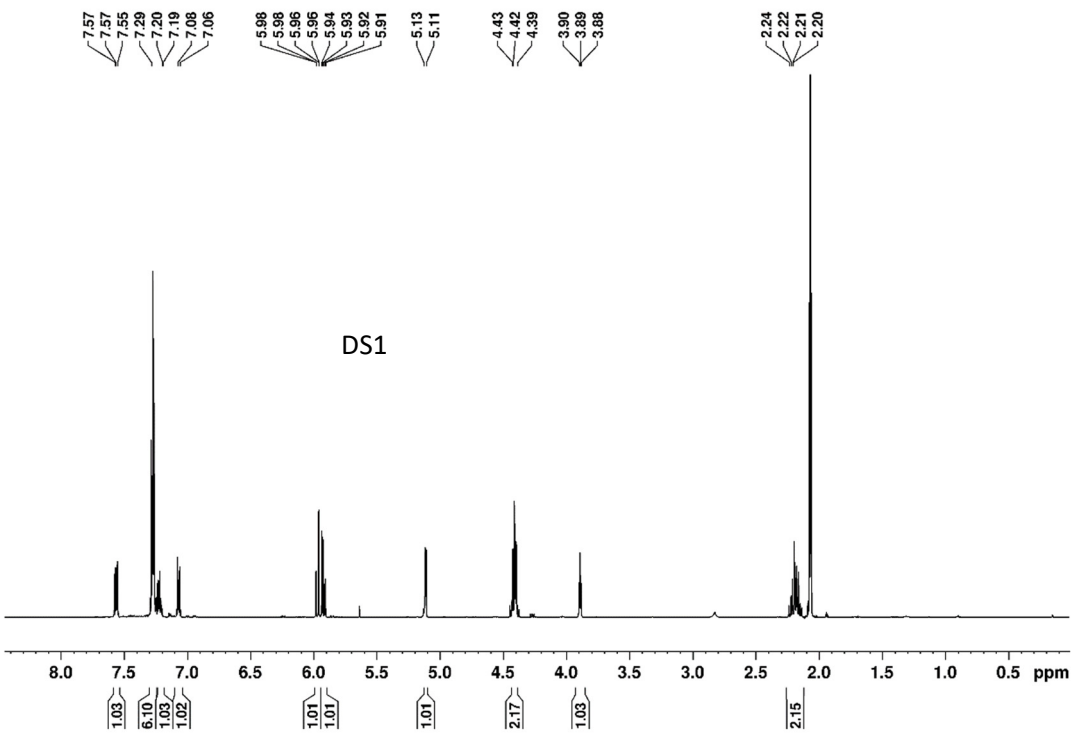
^{13}C NMR spectrum (125 MHz)



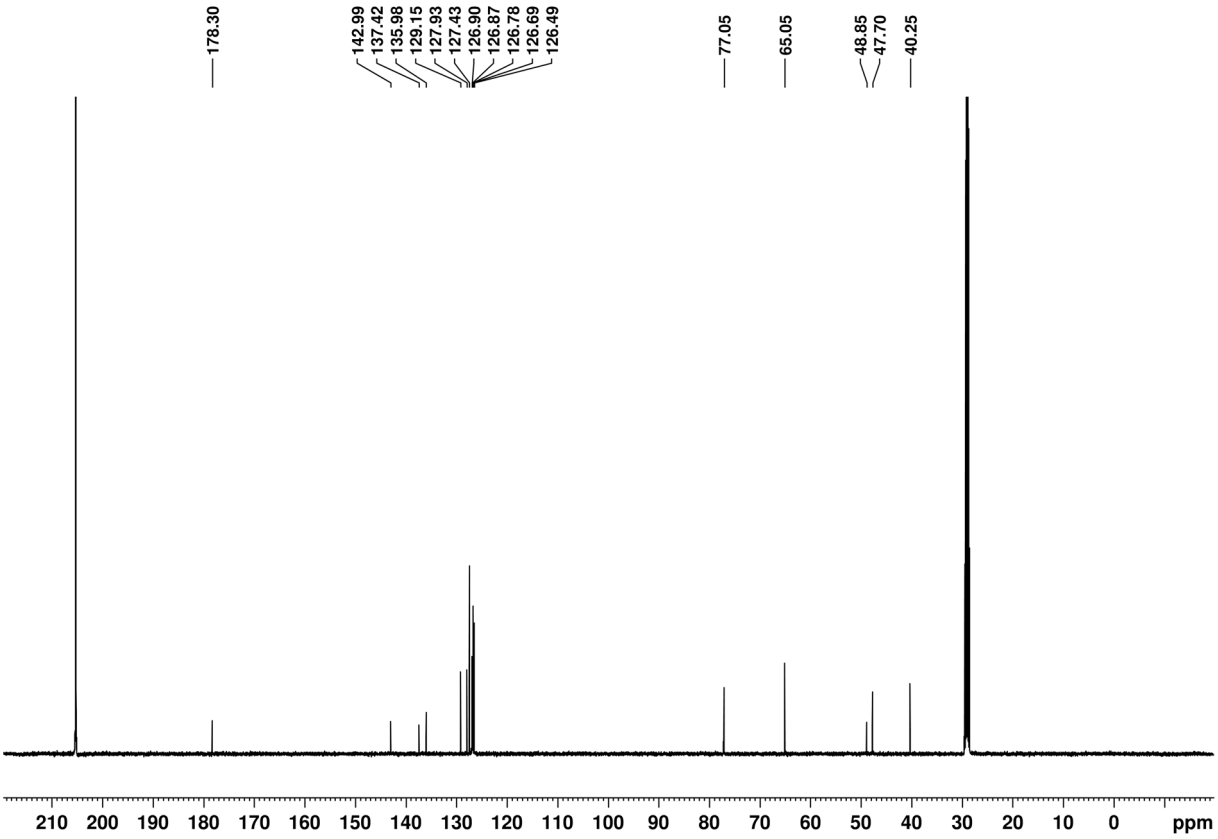
NOESY of 26bc



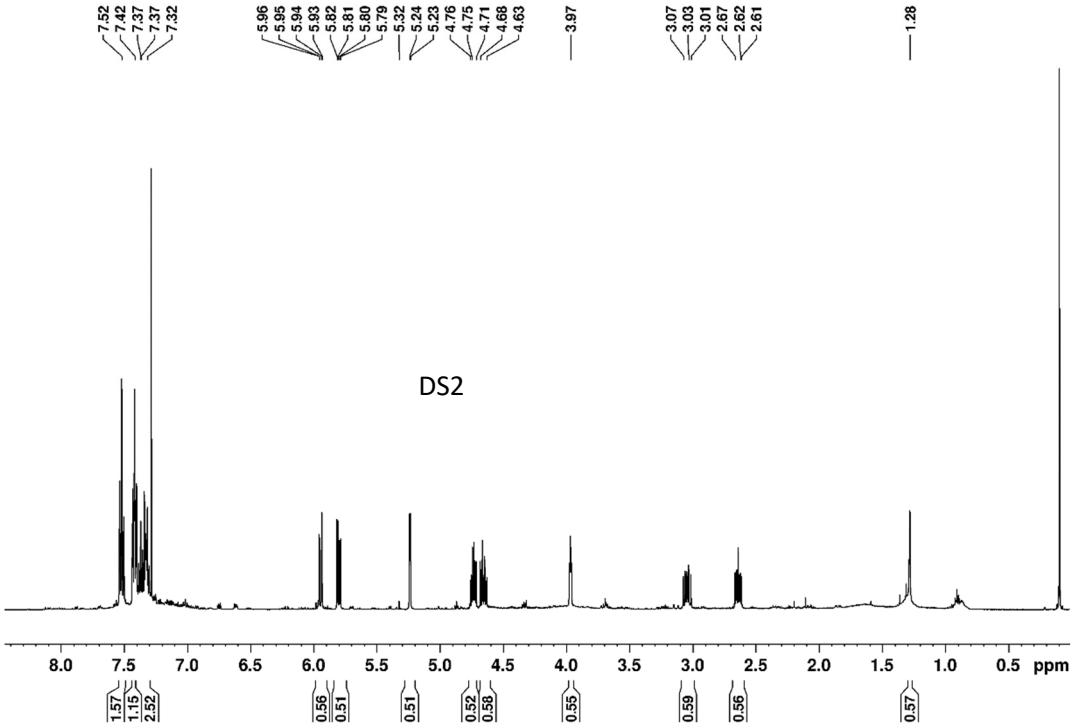
^{1}H NMR spectrum (500 MHz, acetone-d_6) of 20c DS1



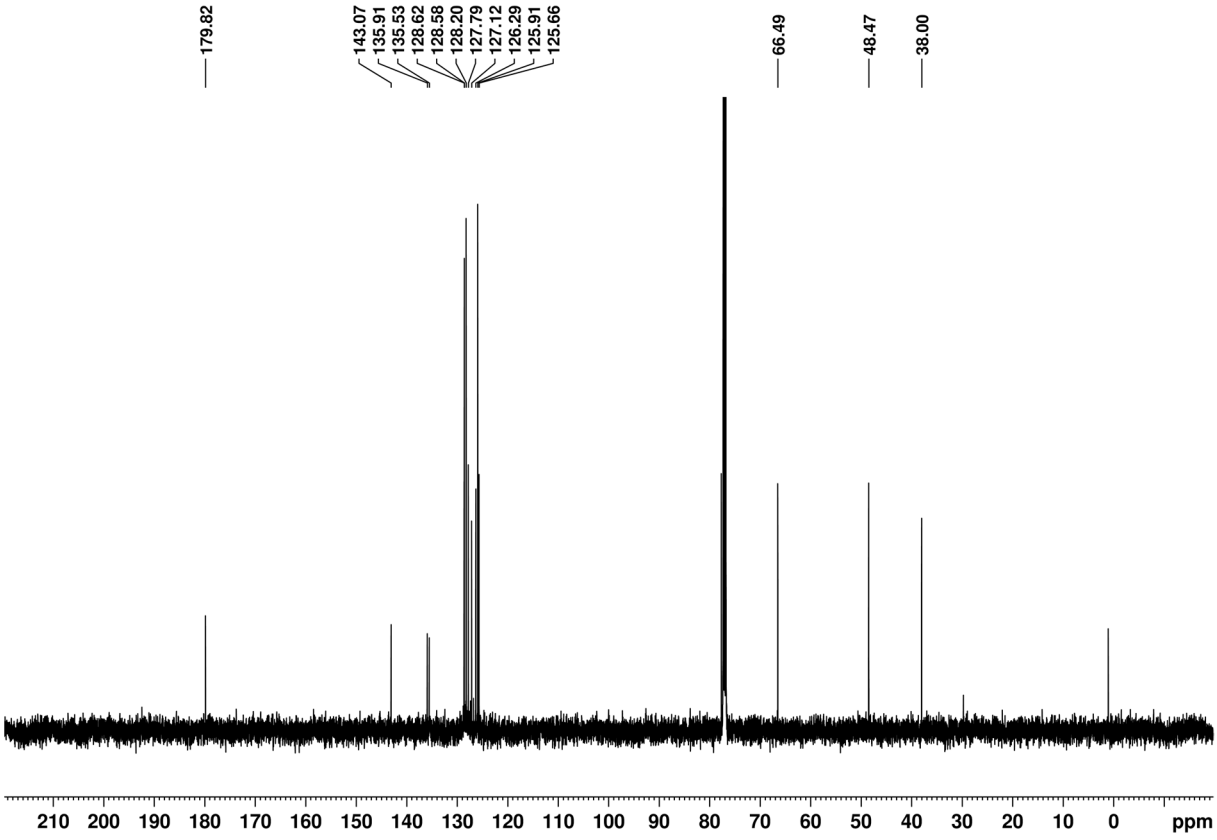
^{13}C NMR spectrum (125 MHz)



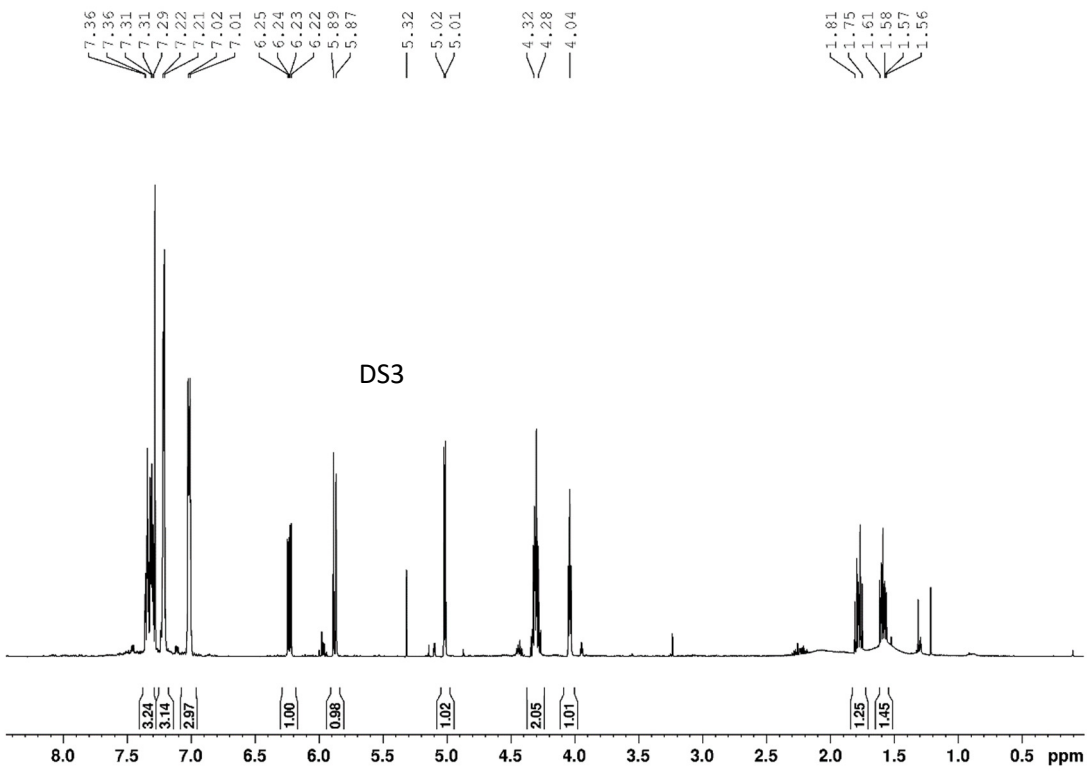
^{1}H NMR spectrum (500 MHz, $CDCl_3$) of 20c DS2



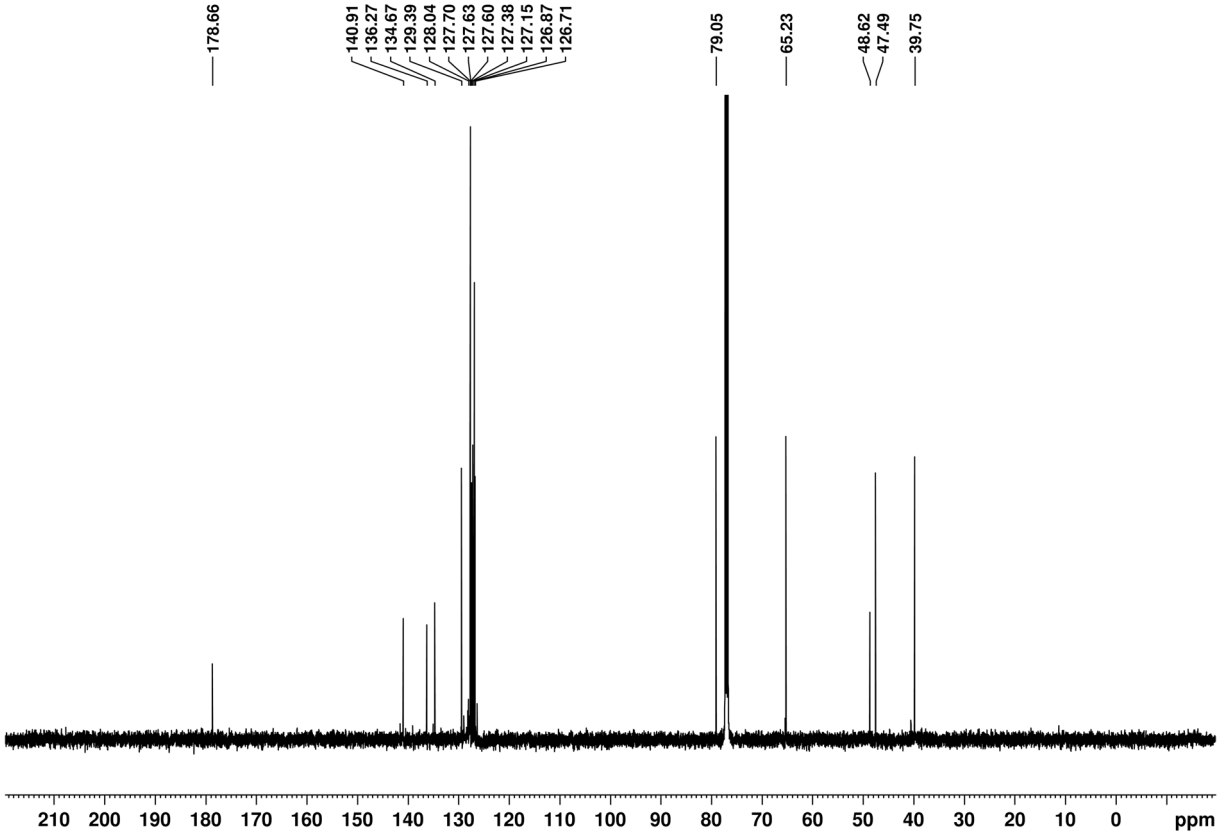
^{13}C NMR spectrum (125 MHz)



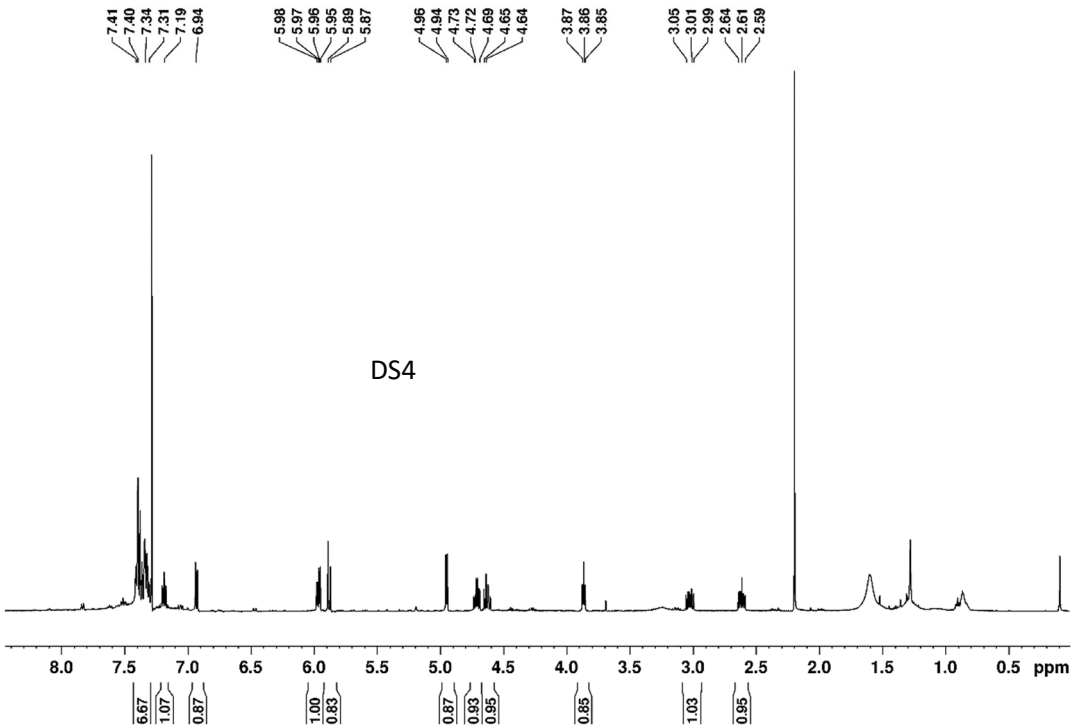
^{1}H NMR spectrum (500 MHz, $CDCl_3$) of 20c DS3



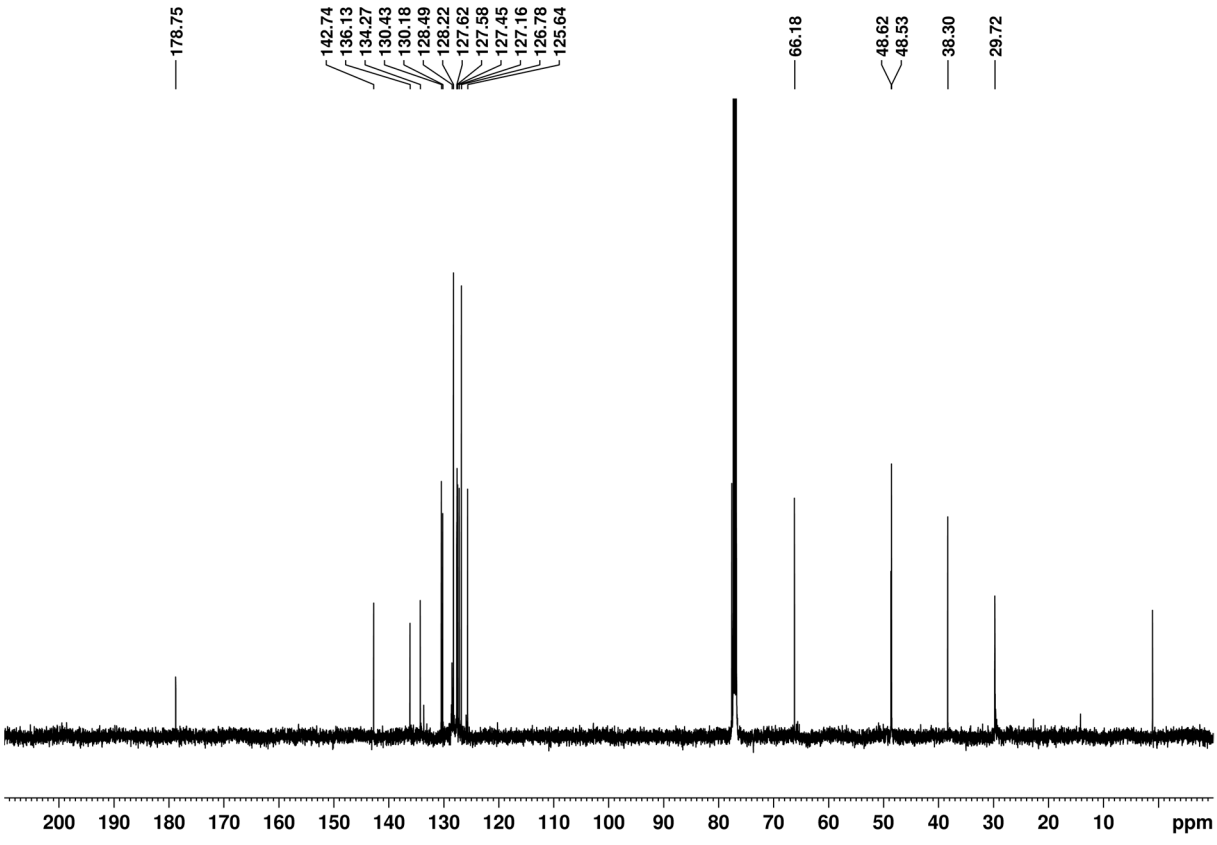
^{13}C NMR spectrum (125 MHz)



^{1}H NMR Spectrum (500 MHz, $CDCl_3$) of 20c DS4



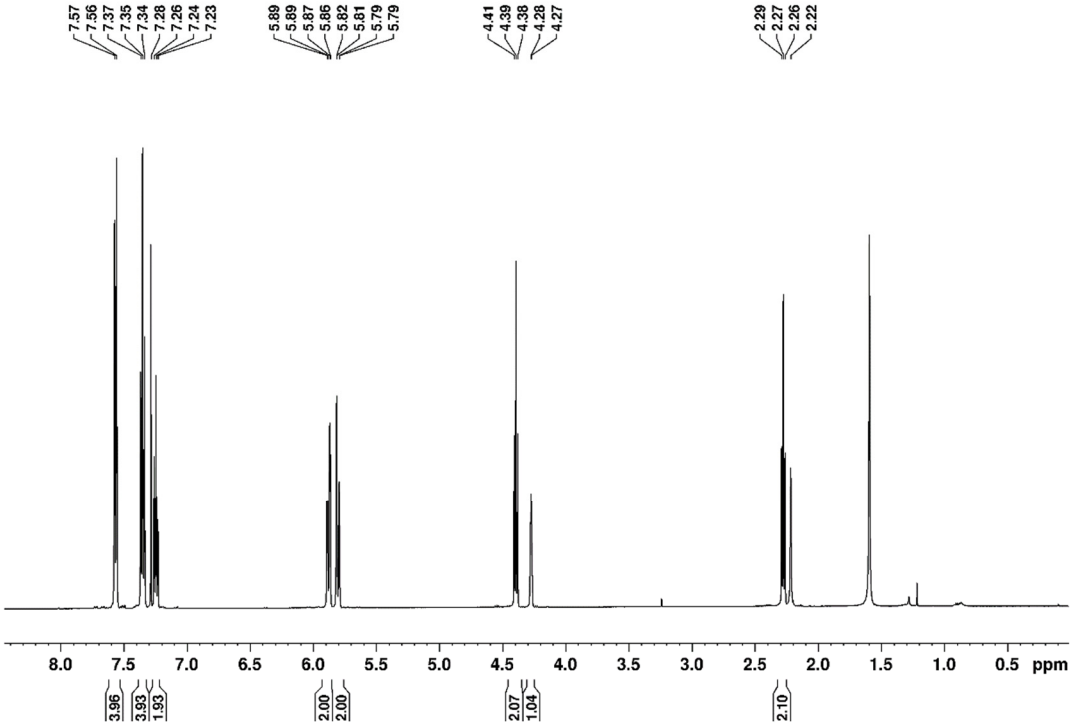
^{13}C NMR spectrum (125 MHz)



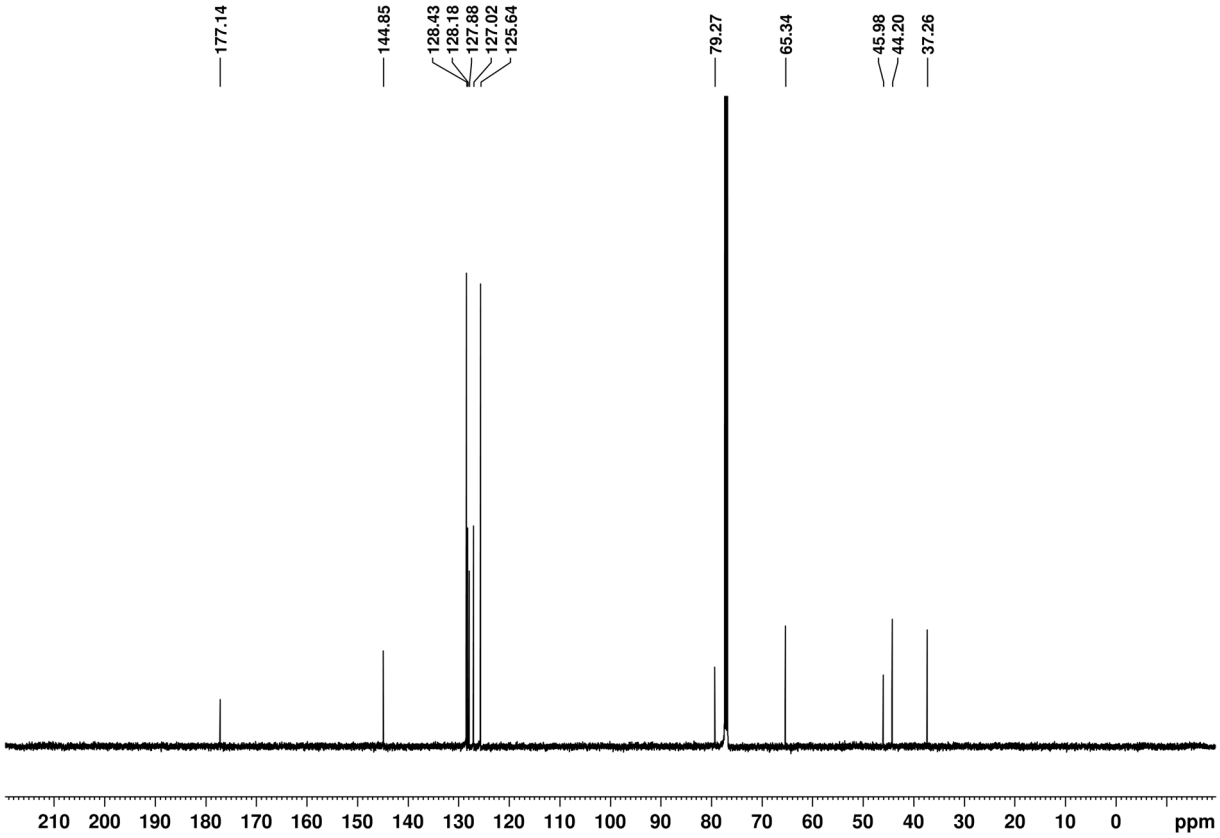
3.4. Products 27 from Reactions with Benzophenone (19)

trans-27a

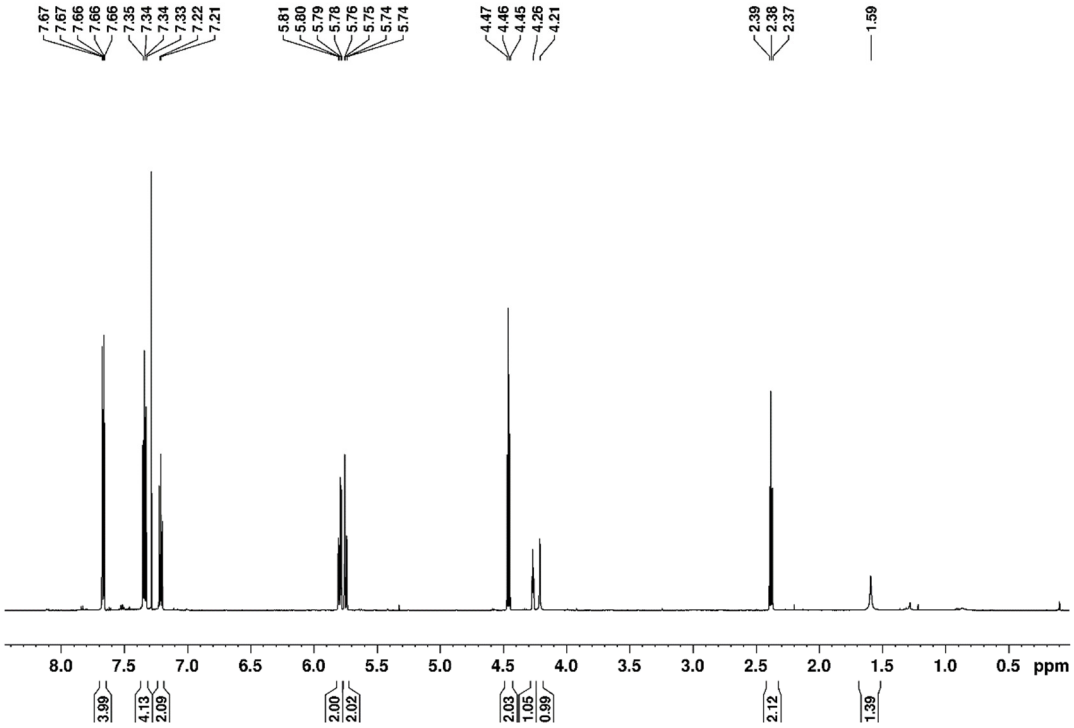
^{1}H NMR spectrum (500 MHz, $CDCl_3$) of *trans*-27a



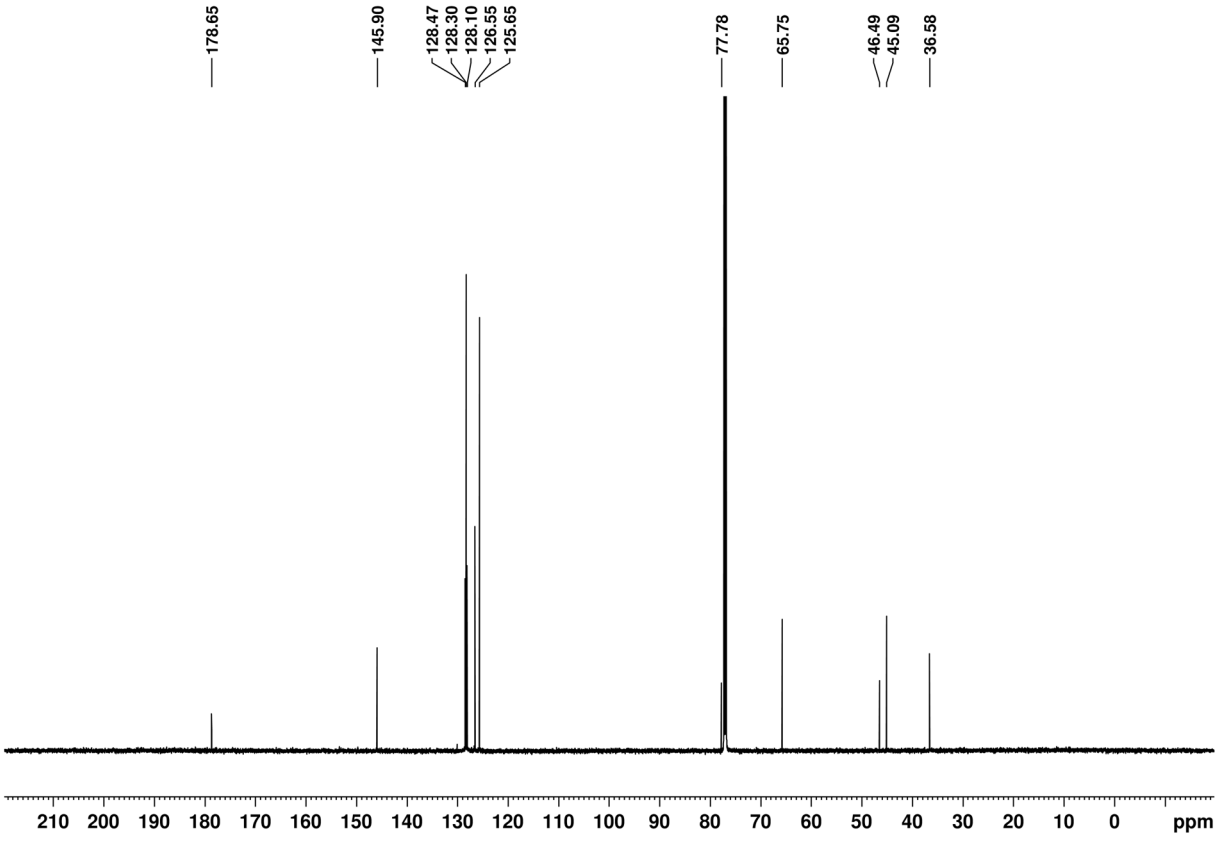
^{13}C NMR spectrum (125 MHz)



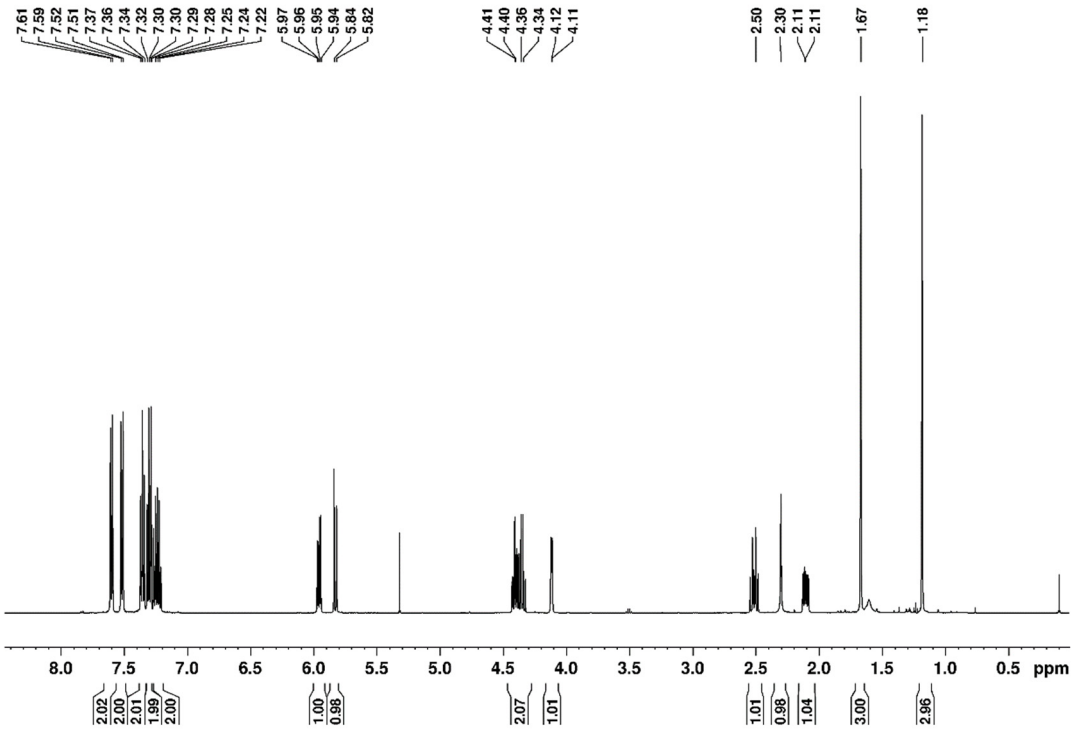
^{1}H NMR spectrum (600 MHz, $CDCl_3$) of *cis*-27a



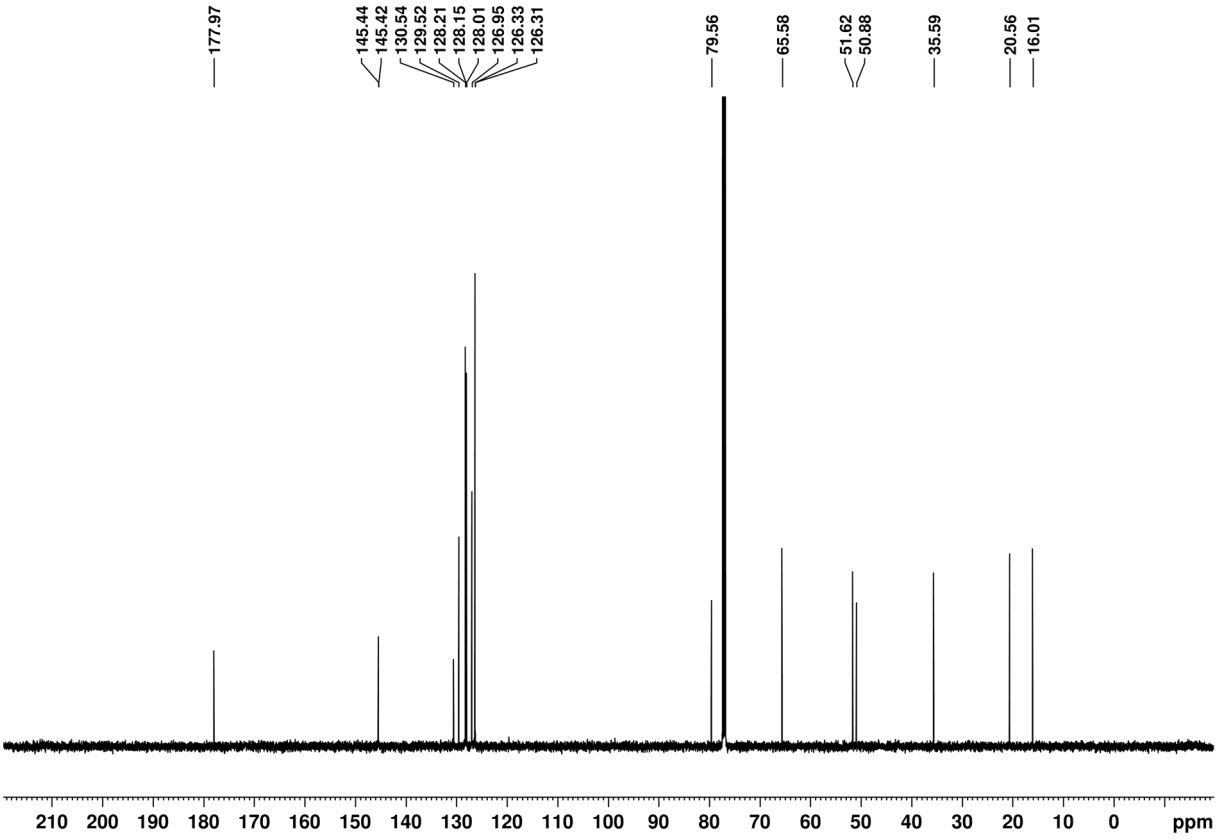
^{13}C NMR spectrum (150 MHz)



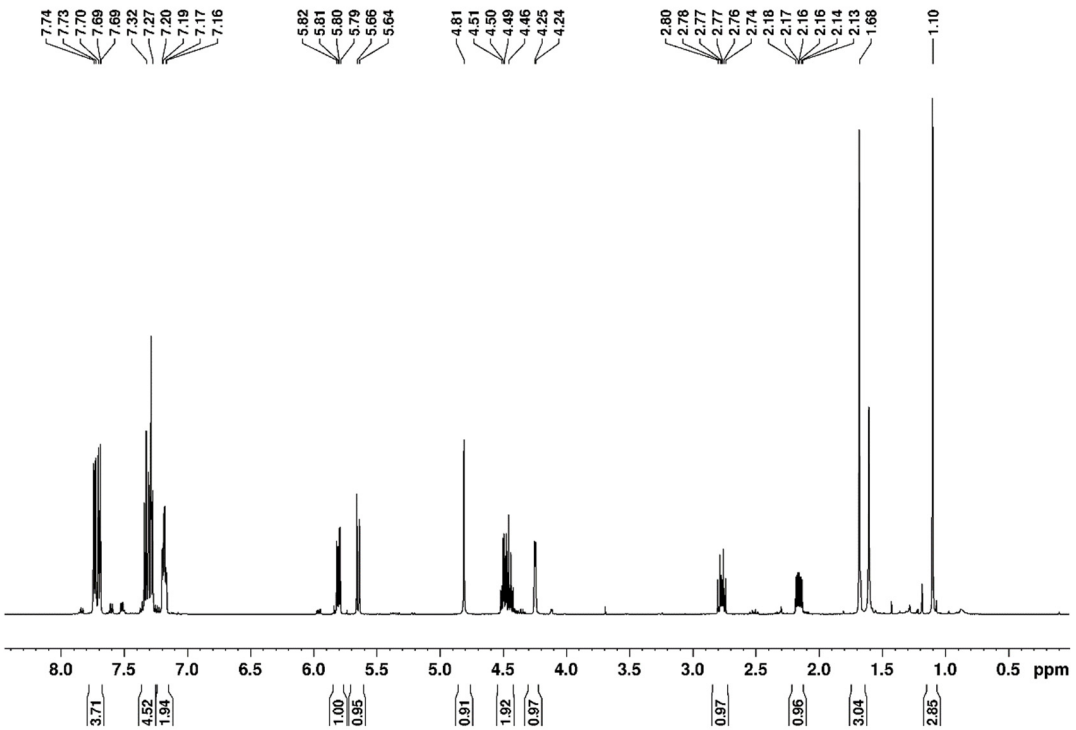
^{1}H NMR spectrum (500 MHz, $CDCl_3$) of *trans*-27b



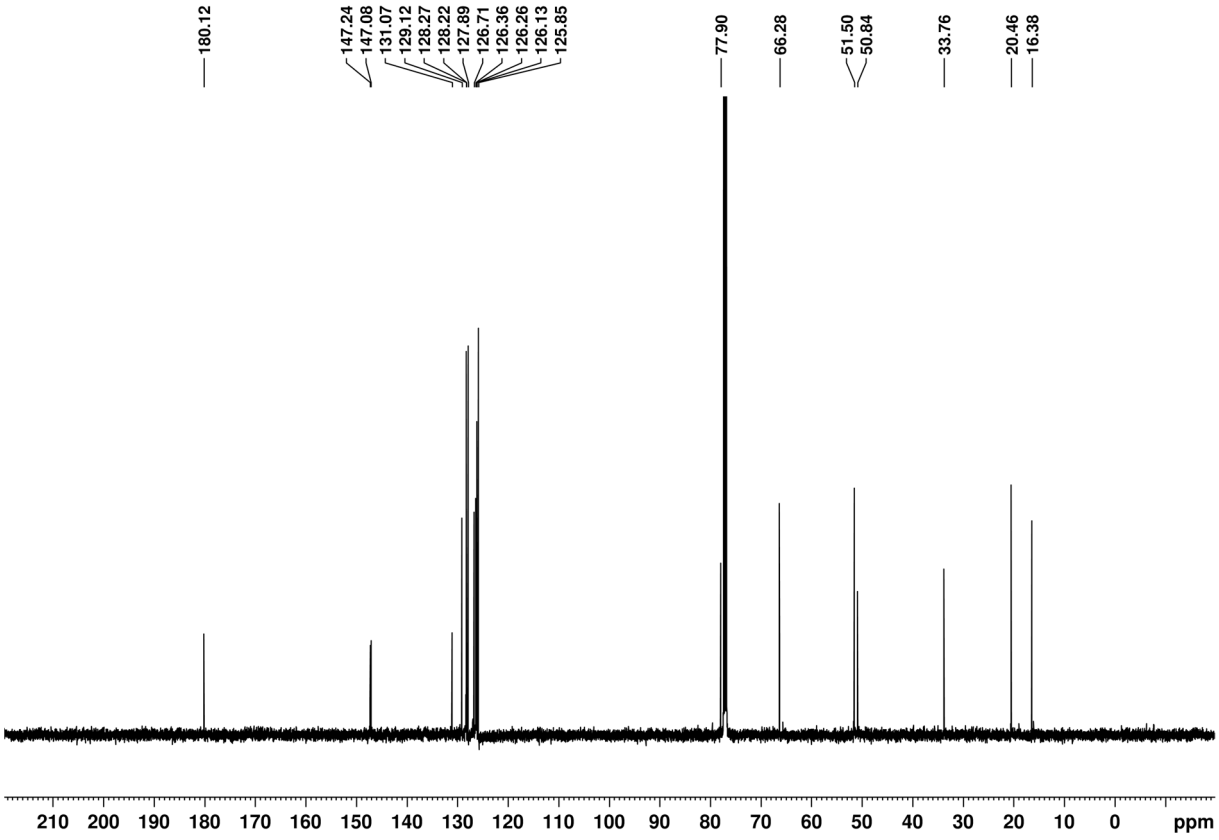
^{13}C NMR spectrum (125 MHz)



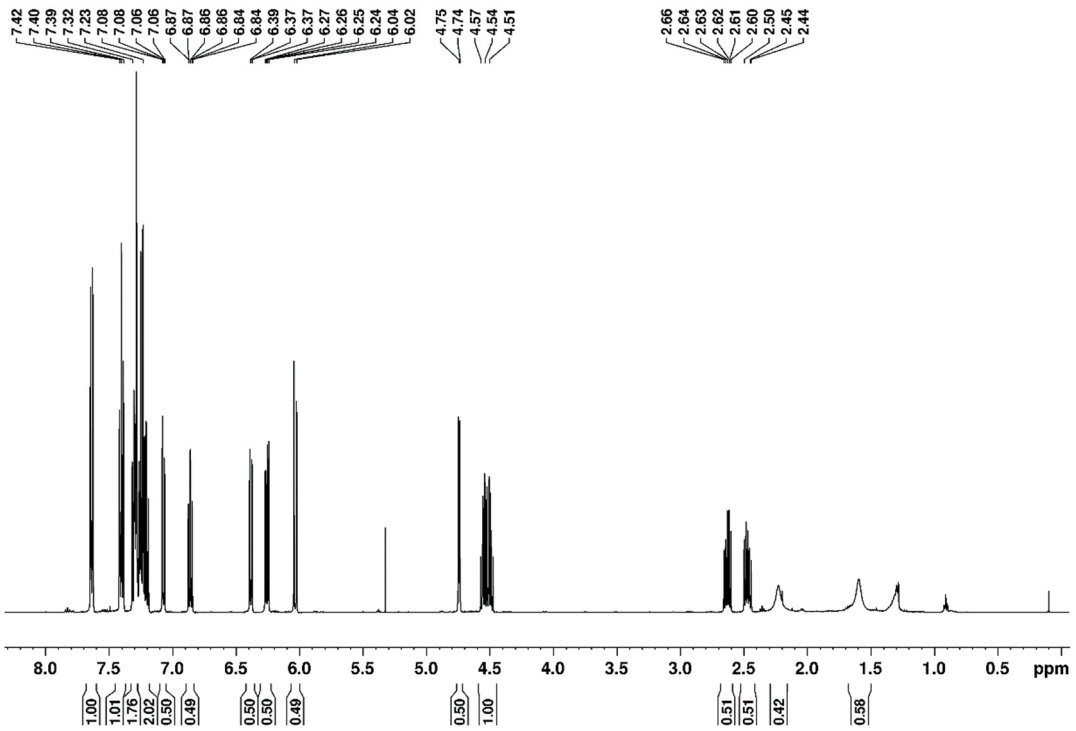
^{1}H NMR spectrum (500 MHz, $CDCl_3$) of *cis*-27b



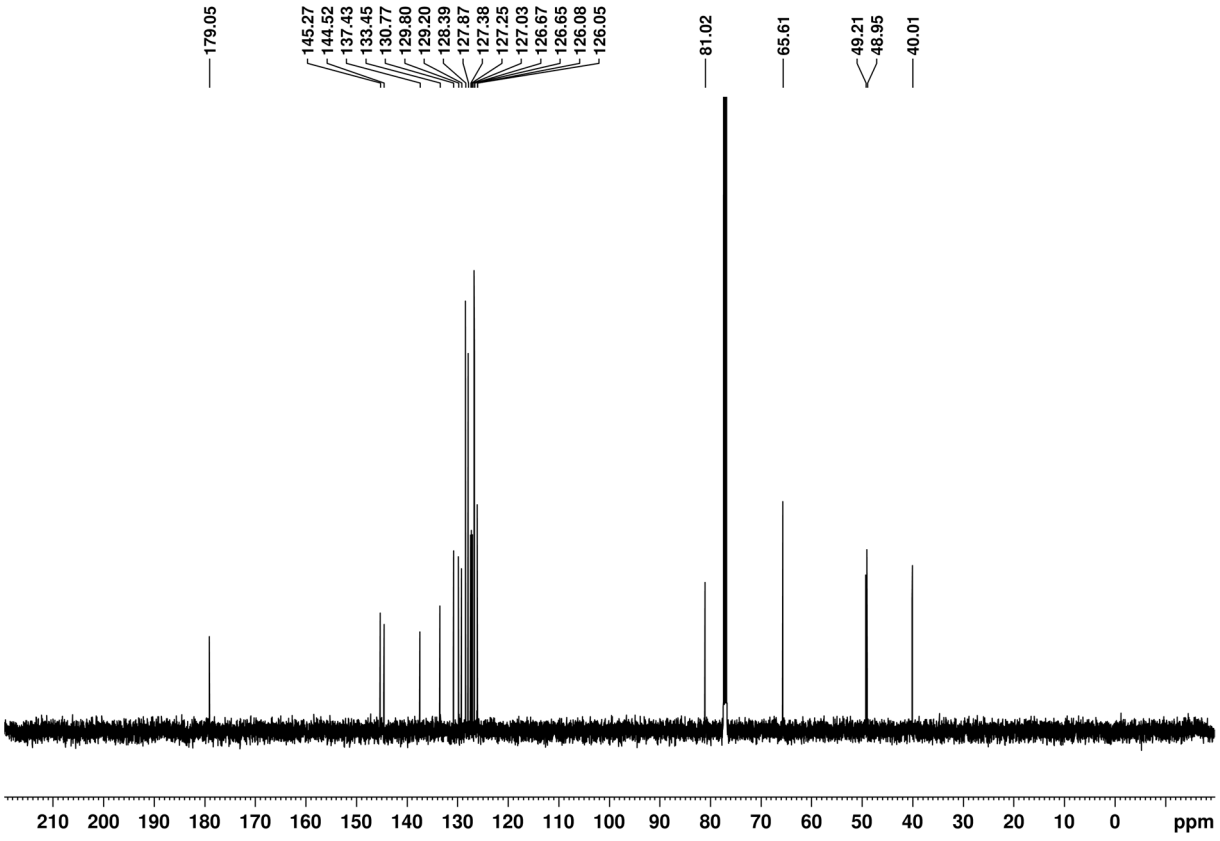
^{13}C NMR spectrum (125 MHz)



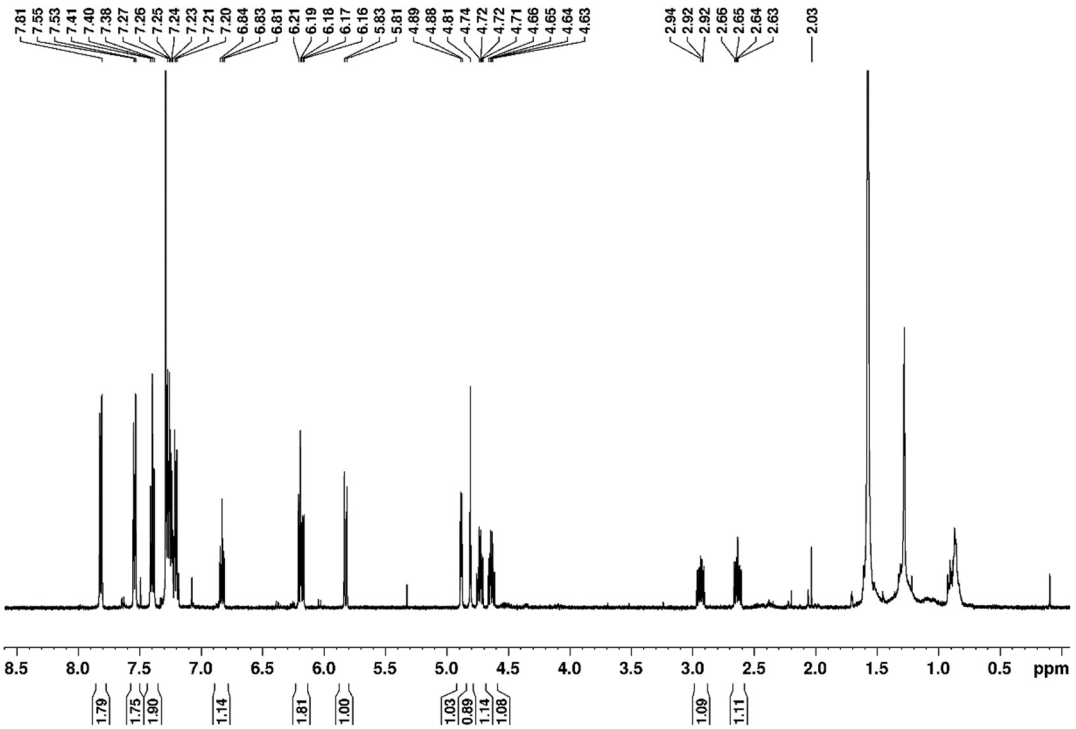
^{1}H NMR spectrum (500 MHz, $CDCl_3$) of *trans*-27c



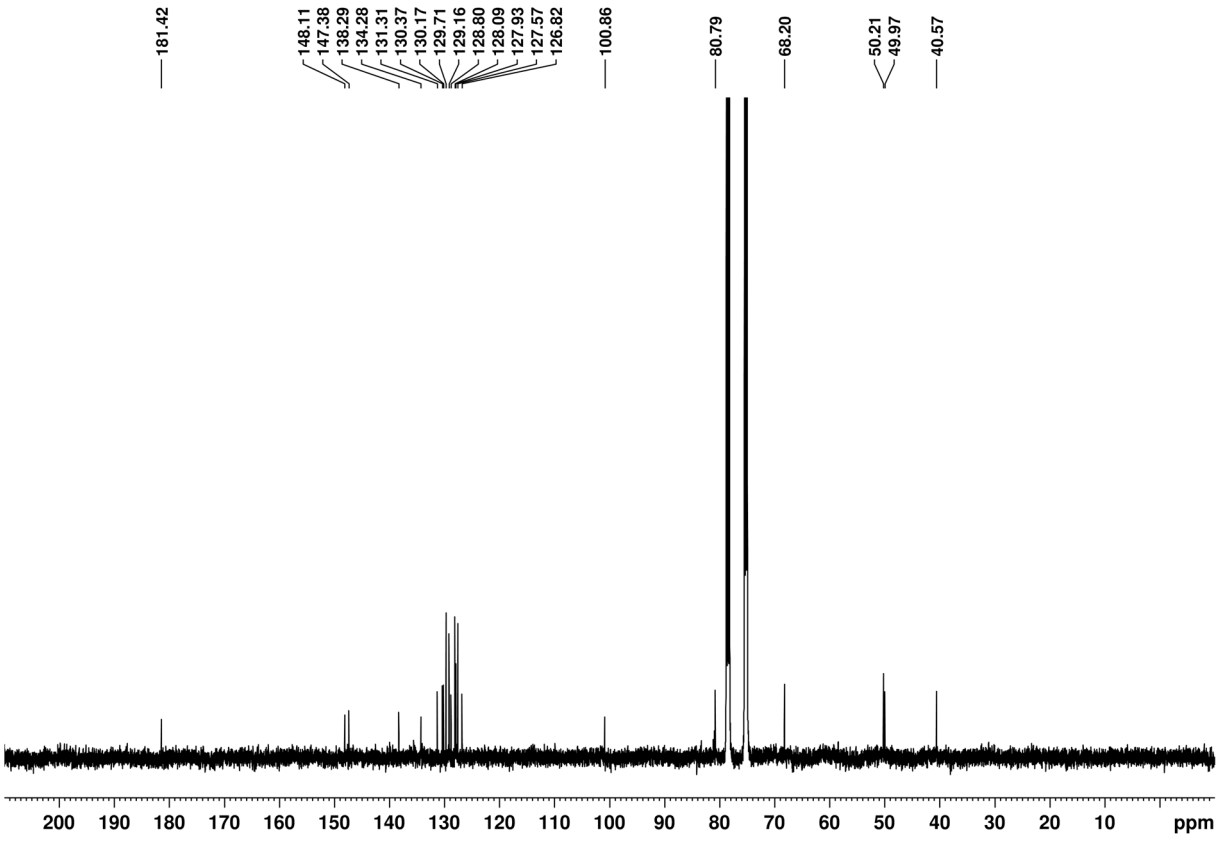
^{13}C NMR spectrum (125 MHz)



^{1}H NMR spectrum (500 MHz, $CDCl_3$ and $C_2D_2Cl_4$) of *cis*-27c



^{13}C NMR spectrum (125 MHz)

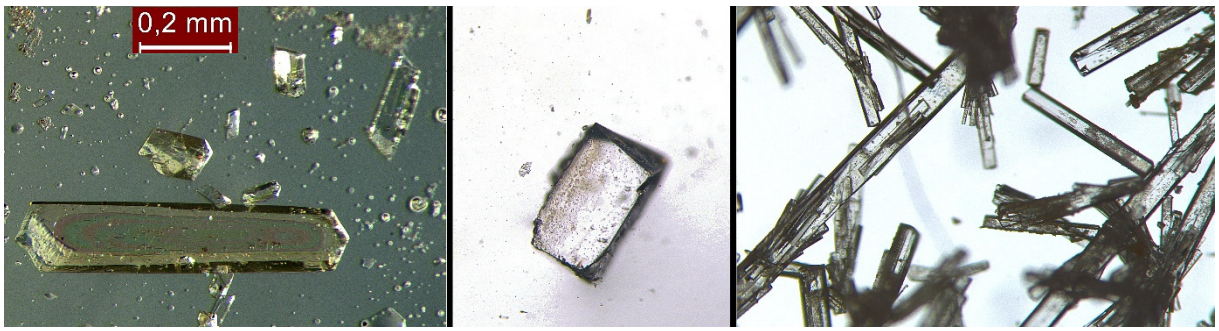


4. X-ray Structure Analyses

General

The crystal structures of the compounds **13c**, *cis*-**27a**, *trans*-**27b** and *trans*-**27c** were determined by single crystal structure analysis. Suitable single crystals were selected using a Leica M205C light microscope and separated with oil. Microscope images show single crystals of the compounds. X-ray crystal structure analysis was performed on a Stadivari diffractometer (Stoe) with monochromated Mo-*K*α radiation (λ = 0.71073 Å). The data correction was performed using the program X-Area [35] The structures were solved by direct methods and refined against F^2 on all data by full-matrix least-squares using the SHELX suite of programs [36,37]. All non-hydrogen atoms were refined anisotropically, the hydrogen atoms were placed on calculated positions. **Table 1** was created using FinalCif [38]. The crystal structures were visualized with Diamond 4 [39]. The data (**13c:** CCCD 2287188, *cis*-**27a**: CCDC 2287189, *trans*-**27b:** CCCD 2287193, *trans*-**27c:** CCCD 2287194) can be obtained free of charge from The Cambridge Crystallographic Data Centre, http://www.ccdc.cam.ac.uk.

Klicken oder tippen Sie hier, um Text einzugeben.



Microscope images of crystals of **13c** (left), *trans*-**27b** (center) and *trans*-**27c** (right).

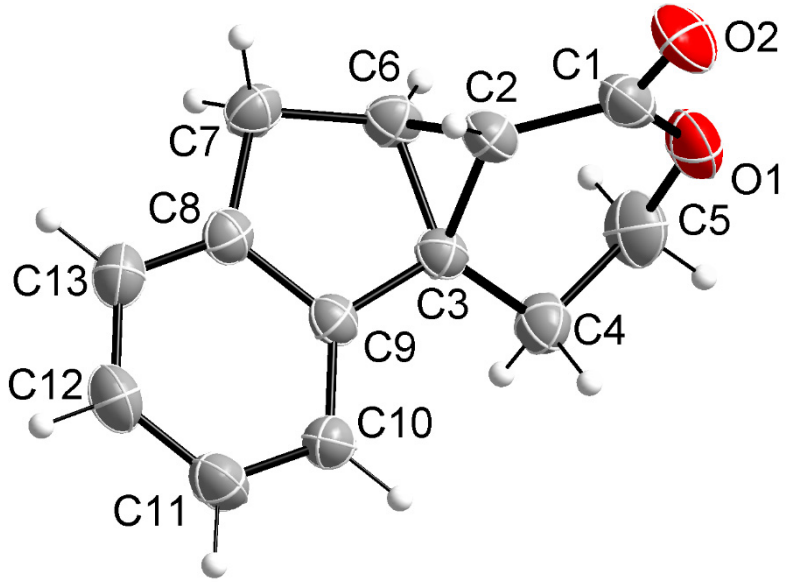
Crystallographic Data

Table 1: Crystal data and structure refinement for 13c, *cis*-27a, *trans*-27b and *trans*-27c.

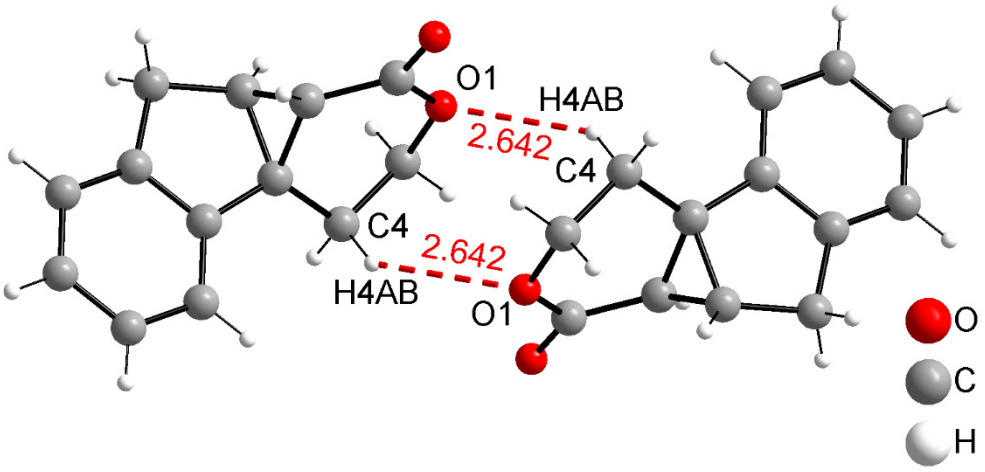
Compound	**13c**	***cis*-27a**	***trans*-27b**	***trans*-27c**
CCDC number	2287188	2287189	2287193	2287194
Empirical formula	$C_{13}H_{12}O_2$	$C_{22}H_{20}O_3$	$C_{24}H_{24}O_3$	$C_{26}H_{22}O_3$
Formula weight	200.23	332.38	360.43	382.43
Temperature [K]	240	210	210	210
Crystal system	monoclinic	triclinic	orthorhombic	orthorhombic
Space group (number)	$P2_1/c$ (14)	$P\overline{1}$ (2)	$P2_12_12_1$ (19)	$P2_12_12_1$ (19)
a [Å]	9.837(2)	9.4469(19)	6.1461(2)	6.2613(4)
b [Å]	11.087(2)	10.192(2)	11.9288(4)	11.8297(7)
c [Å]	9.4485(19)	10.454(2)	25.5496(8)	26.0964(16)
α [°]	90	103.99(3)	90	90
β [°]	106.09(3)	103.50(3)	90	90
γ [°]	90	111.81(3)	90	90
Volume [Å^3]	990.1(4)	846.4(4)	1873.18(11)	1932.9(2)
Z	4	2	4	4
ρ_{calc} [gcm^{-3}]	1.343	1.304	1.278	1.314
μ [mm^{-1}]	0.090	0.085	0.083	0.085
F(000)	424	352	768	808
Crystal size [mm^3]	0.500×0.400×0.200	0.500×0.367×0.100	0.790×0.597×0.500	0.500×0.350×0.250
Crystal colour	yellow	colorless	colorless	colorless
Crystal shape	plate	plate	block	rod
Radiation	Mo K_α (λ=0.71073 Å)	Mo K_α (λ=0.71073 Å)	Mo K_α (λ=0.71073 Å)	Mo K_α (λ=0.71073 Å)
2θ range [°]	5.66 to 65.21 (0.66 Å)	6.55 to 65.67 (0.66 Å)	5.88 to 49.96 (0.84 Å)	7.13 to 50.96 (0.83 Å)
Index ranges	−14 ≤ h ≤ 14 −16 ≤ k ≤ 16 −12 ≤ l ≤ 14	−14 ≤ h ≤ 13 −14 ≤ k ≤ 14 −15 ≤ l ≤ 13	−7 ≤ h ≤ 7 −14 ≤ k ≤ 14 −30 ≤ l ≤ 30	−7 ≤ h ≤ 7 −14 ≤ k ≤ 14 −31 ≤ l ≤ 31
Reflections collected	14834	21537	13355	10068
Independent reflections	R_{int} = 0.0314 R_{sigma} = 0.0356	R_{int} = 0.0456 R_{sigma} = 0.0466	R_{int} = 0.0496 R_{sigma} = 0.0325	R_{int} = 0.0382 R_{sigma} = 0.0428
Completeness to θ = 25.242°	99.9 %	99.4 %	99.6 %	97.3 %
Data / Restraints / Parameters	3380/0/137	5655/0/228	3293/0/250	3487/0/266
Goodness-of-fit on F^2	1.010	1.007	1.026	1.040
Final *R* indexes [I≥2σ(I)]	R_1 = 0.0426 wR_2 = 0.1136	R_1 = 0.0490 wR_2 = 0.1158	R_1 = 0.0415 wR_2 = 0.1058	R_1 = 0.0491 wR_2 = 0.1083
Final *R* indexes [all data]	R_1 = 0.0714 wR_2 = 0.1213	R_1 = 0.0936 wR_2 = 0.1427	R_1 = 0.0469 wR_2 = 0.1103	R_1 = 0.0703 wR_2 = 0.1187
Largest peak/hole [eÅ^{-3}]	0.28/-0.17	0.33/-0.27	0.18/-0.16	0.16/-0.18

Visualizations of the crystal structure and molecule structure

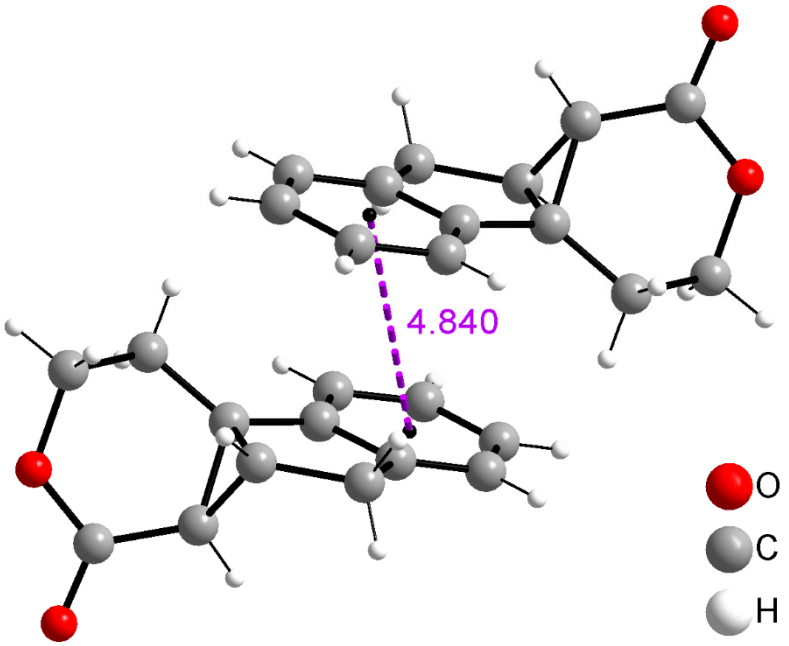
Compound 13c:



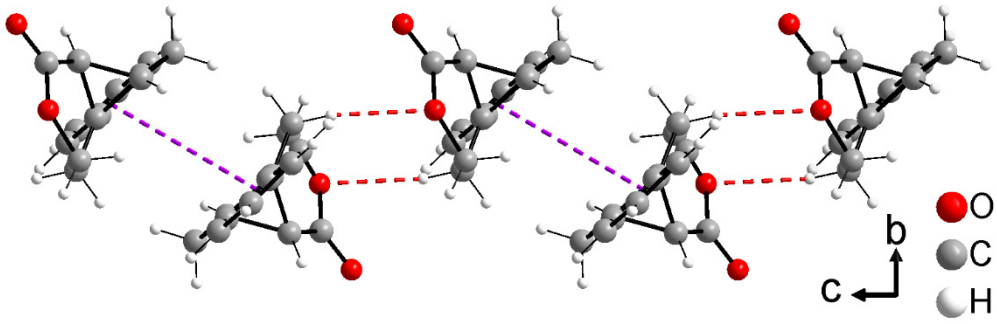
Molecular structure with atom labeling of **13c**. Displacement ellipsoids are shown at the 50% probability level.



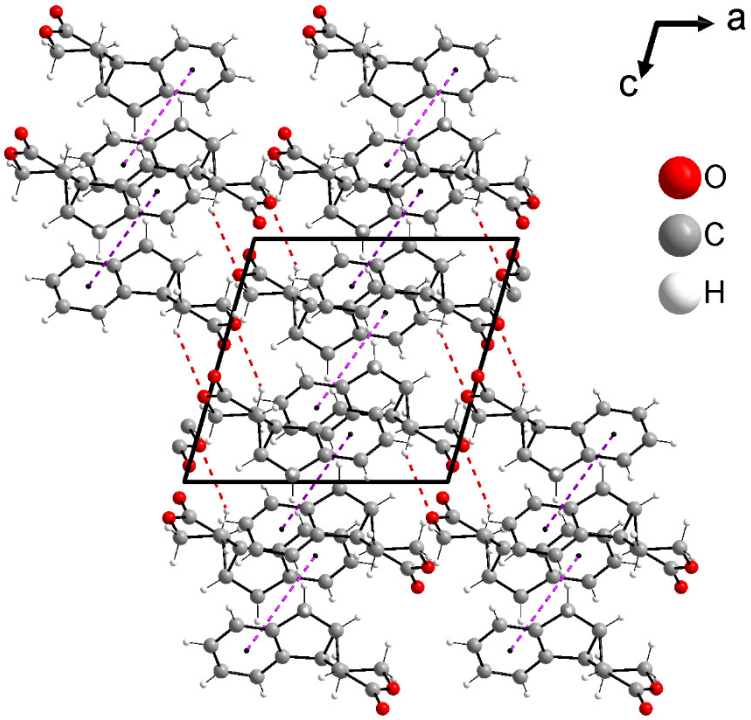
C-H···O hydrogen bonding (red dashed lines) in **13c** forming dimers.



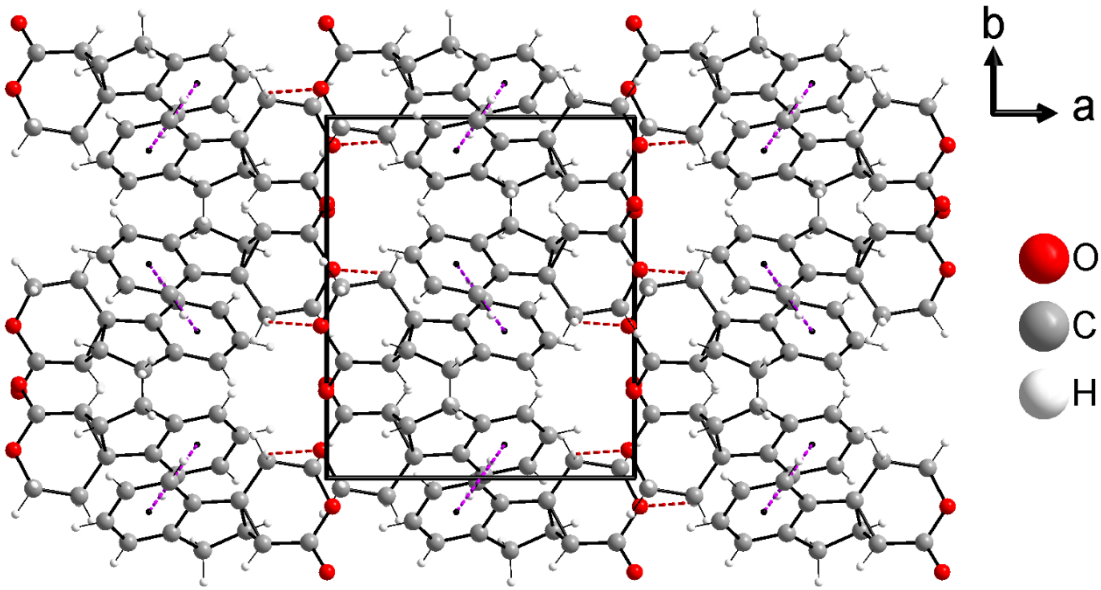
Stacking interactions (purple dotted lines) between the molecules in **13c**.



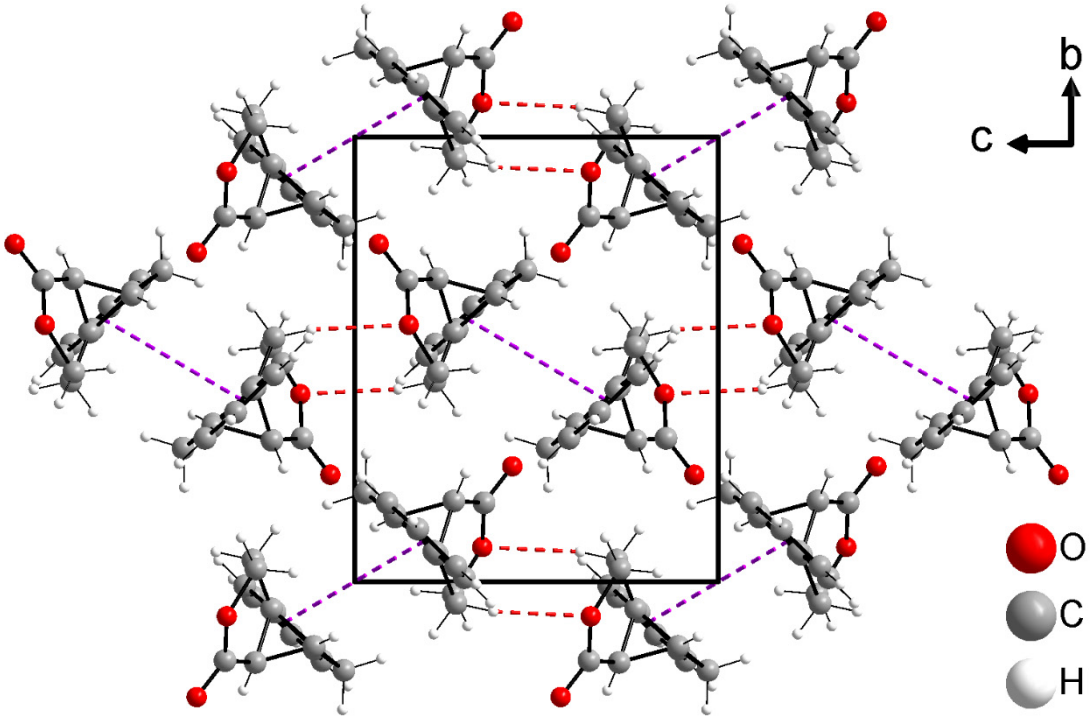
The hydrogen bonds (red dashed lines) and stacking interactions (purple dotted lines) form chains along the crystallographic c axis between the molecules in **13c**.



Cell view of **13c** looking along the crystallographic b axis.

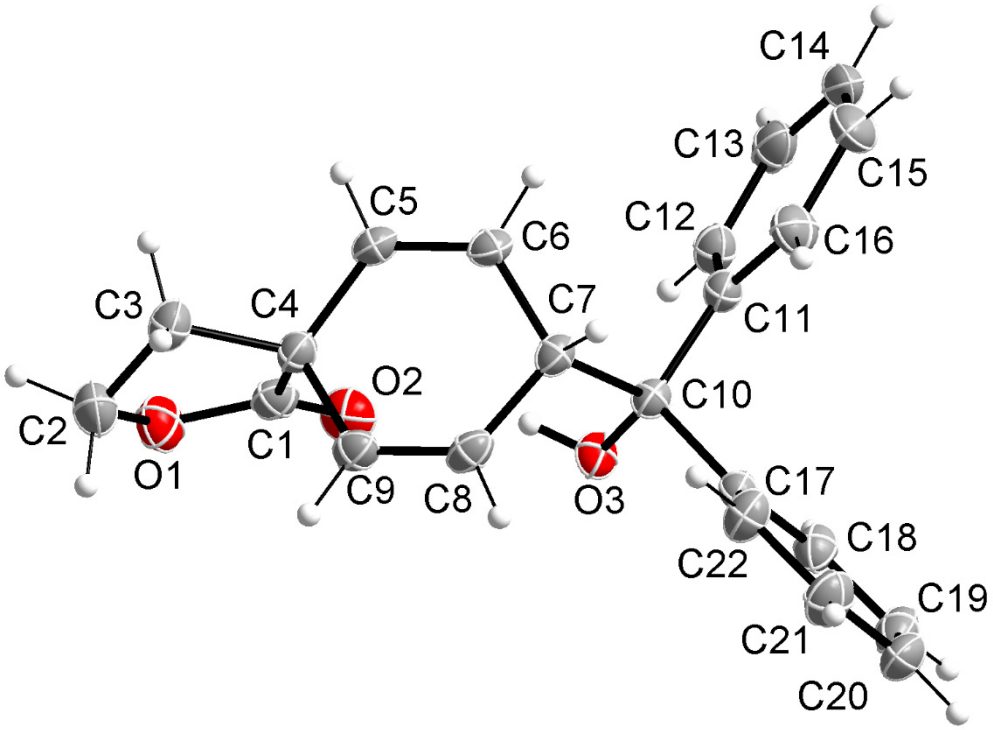


Cell view of **13c** looking along the crystallographic c axis.

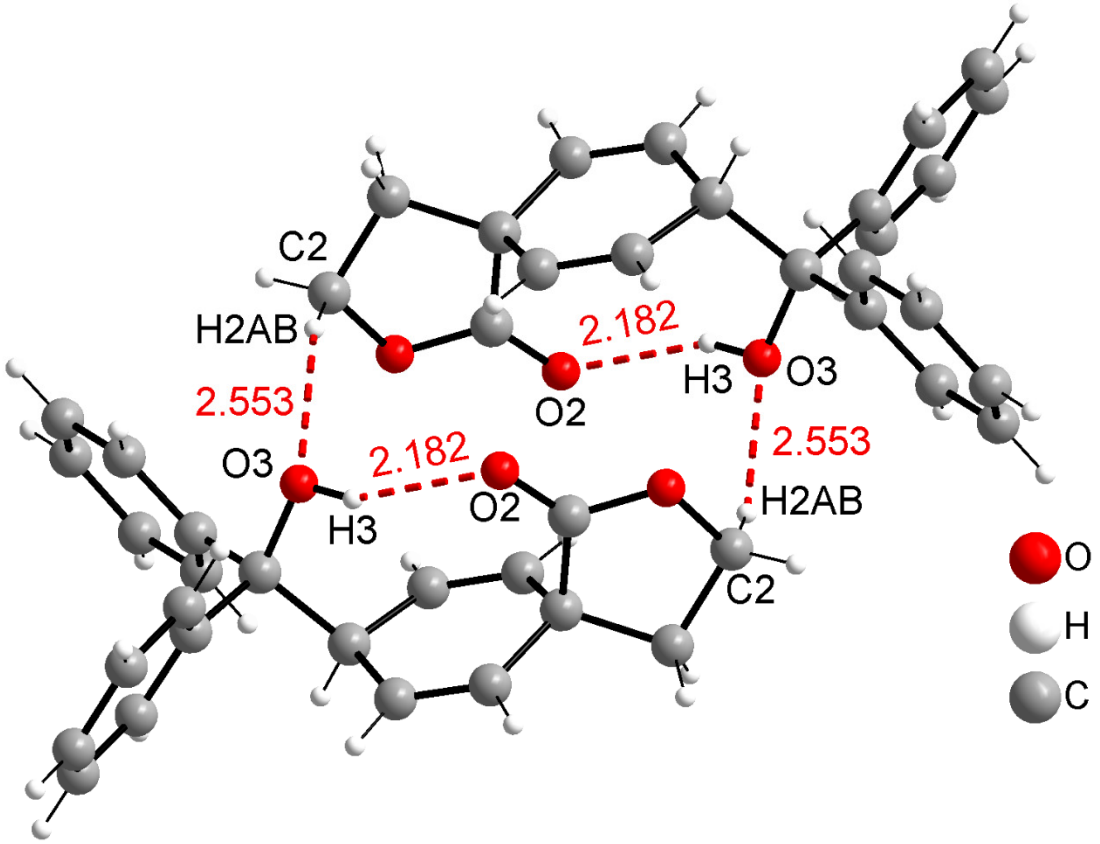


Cell view of **13c** looking along the crystallographic a axis.

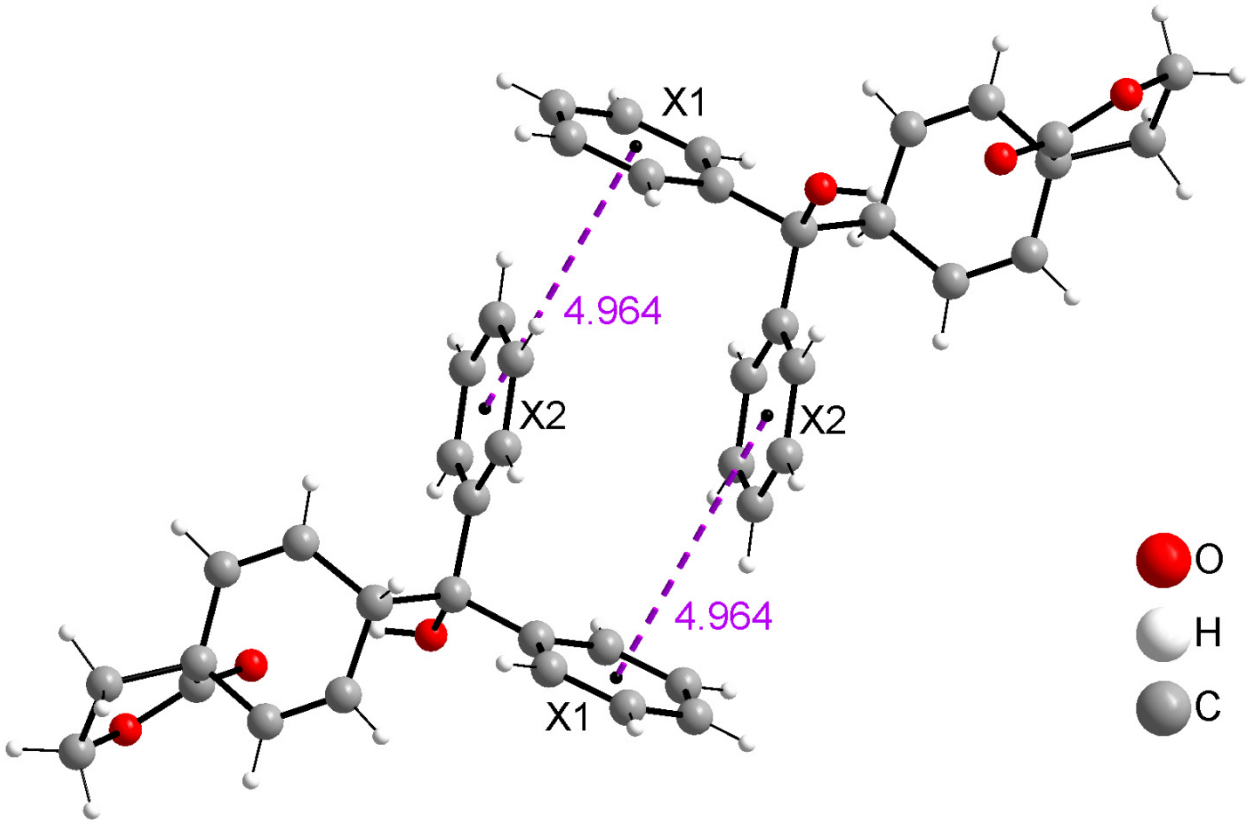
Compound *cis*-27a:



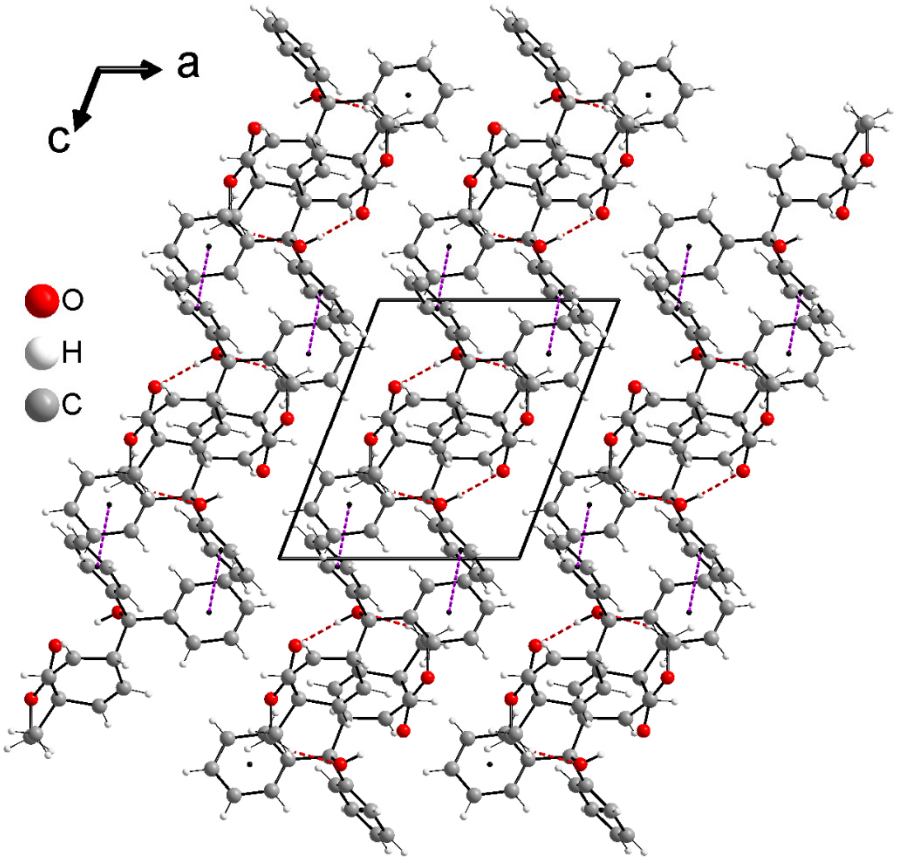
Molecular structure with atom labeling of *cis*-**27a**. Displacement ellipsoids are shown at the 50% probability level.



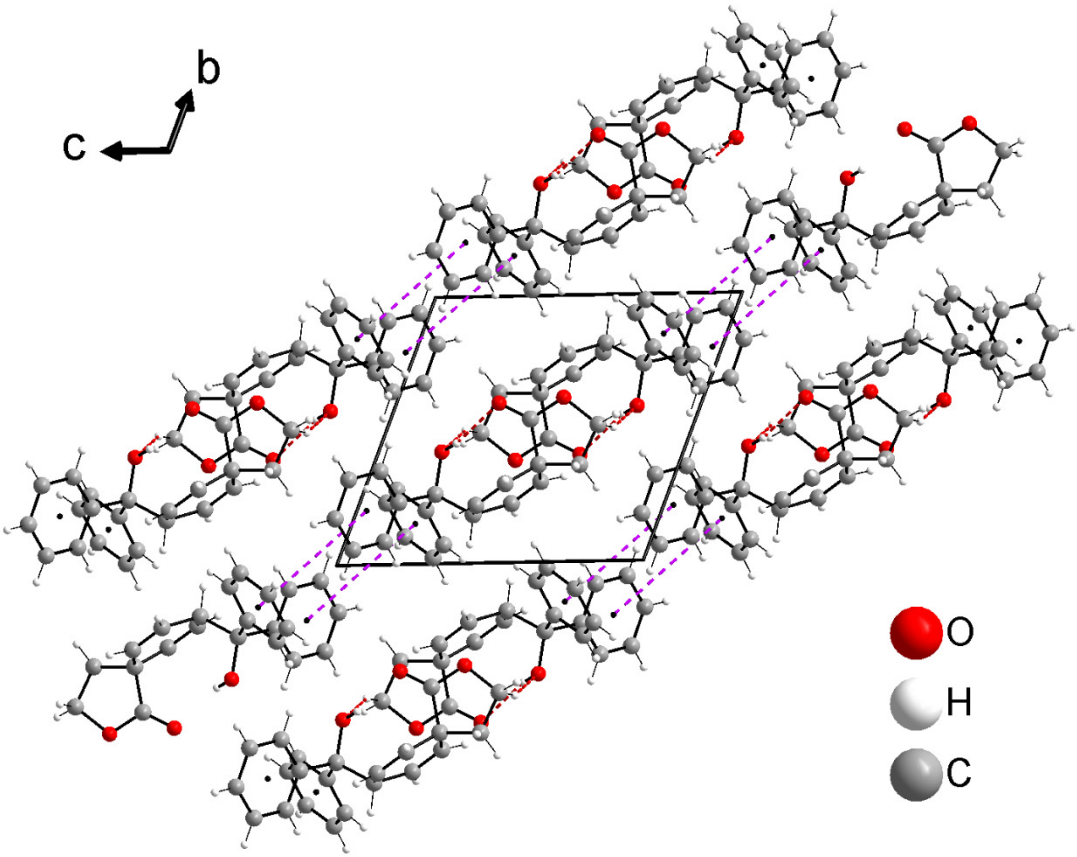
O-H···O and C-H···O hydrogen bonds (red dashed lines) in *cis*-**27a** forming dimers.



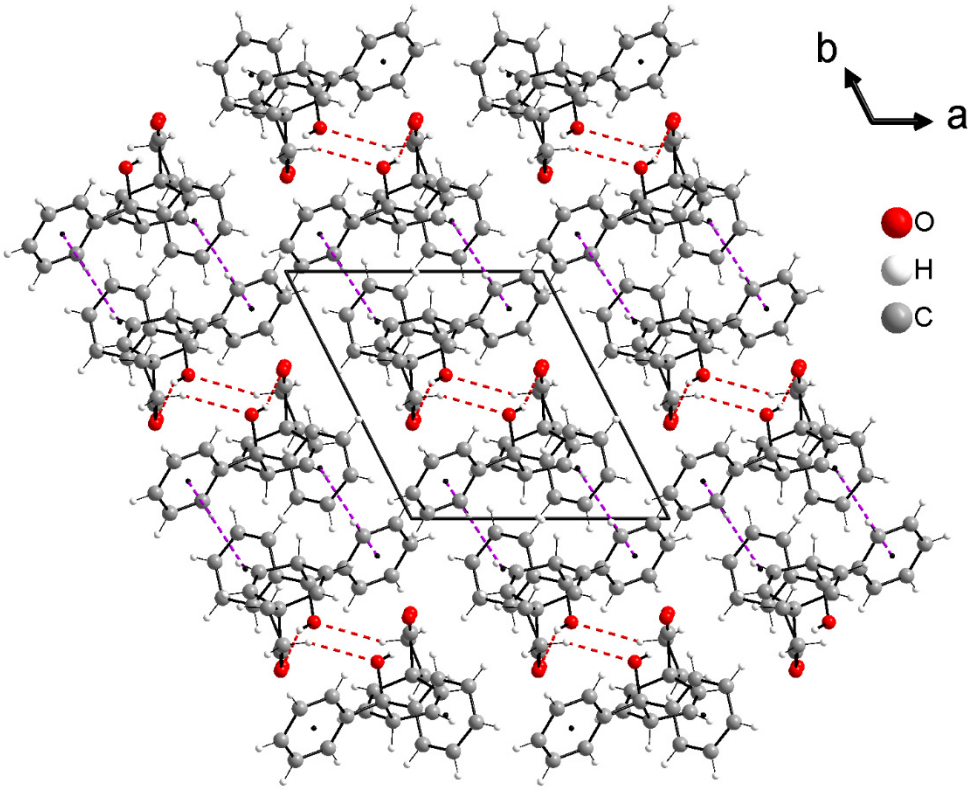
Stacking interactions (purple dotted lines) between the molecules in *cis*-**27a** (X marks the center of the aromatics).



Cell view of *cis*-**27a** looking along the crystallographic b axis.

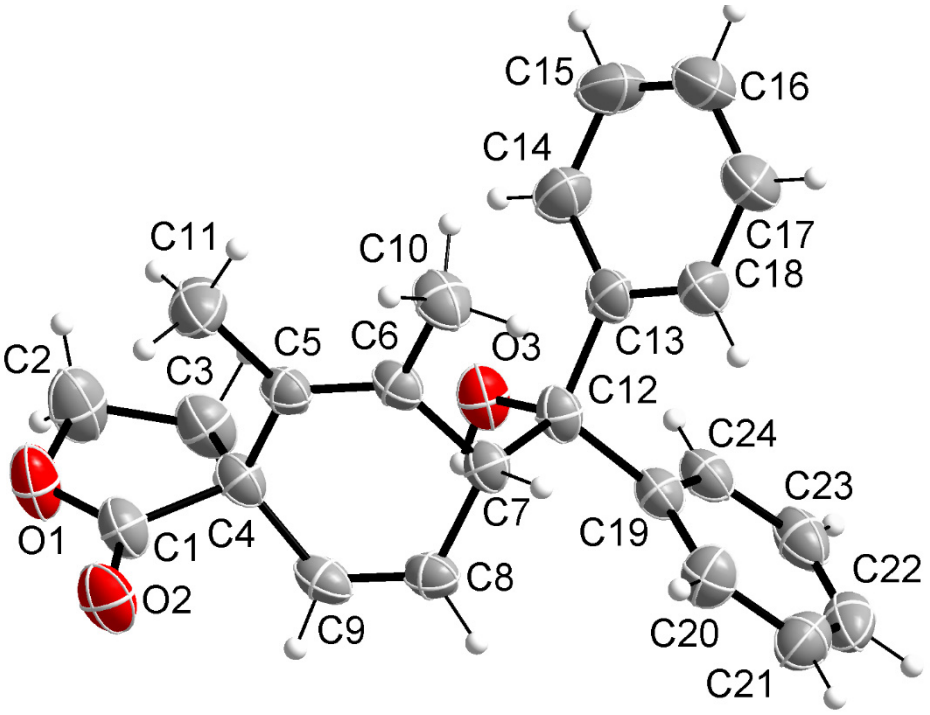


Cell view of *cis*-**27a** looking along the crystallographic a axis.

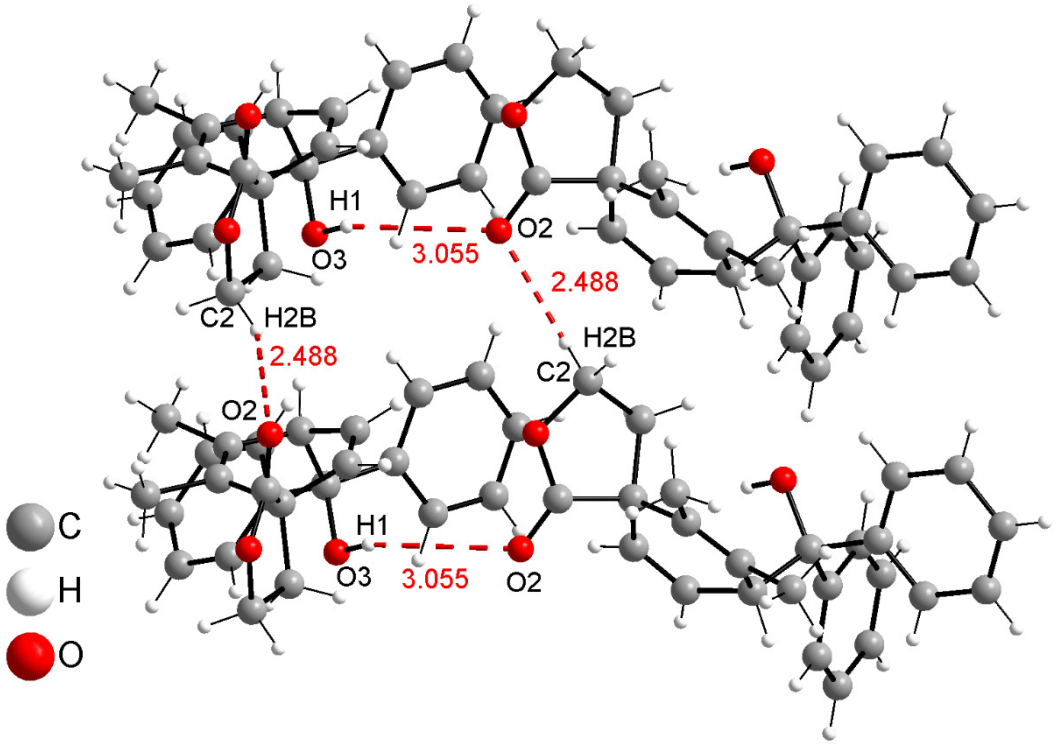


Cell view of *cis*-**27a** looking along the crystallographic c axis.

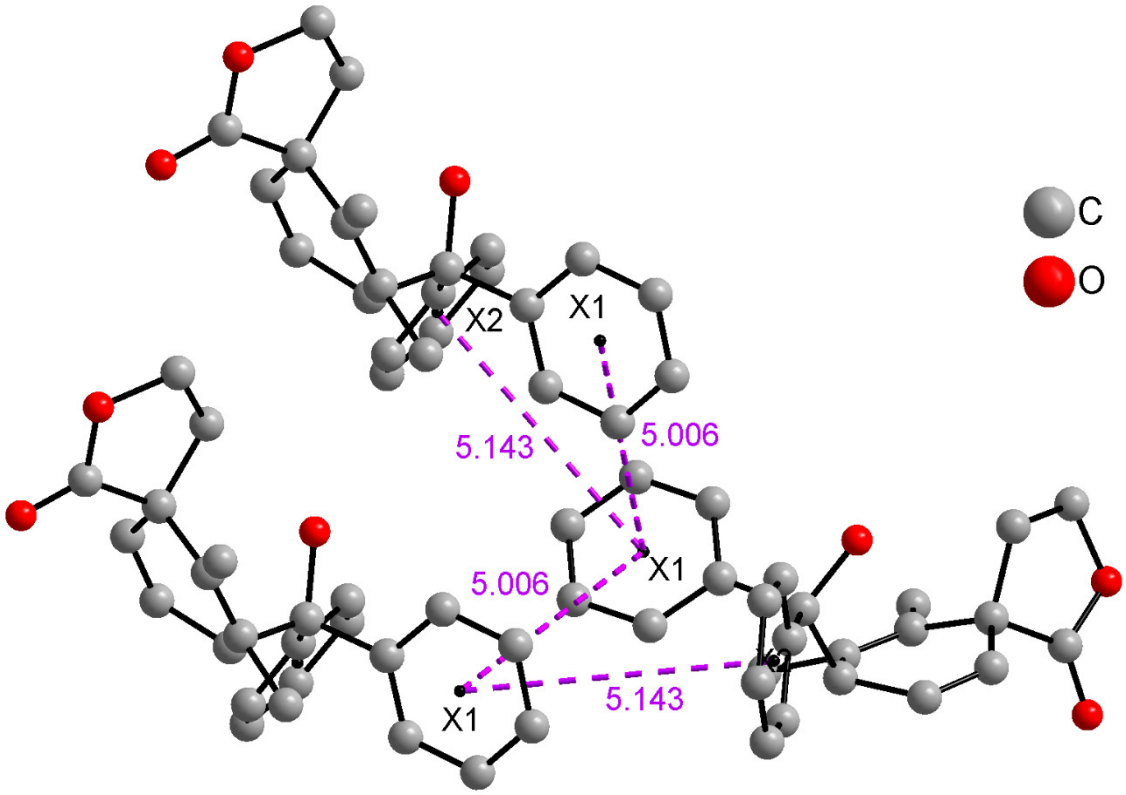
Compound *trans*-27b:



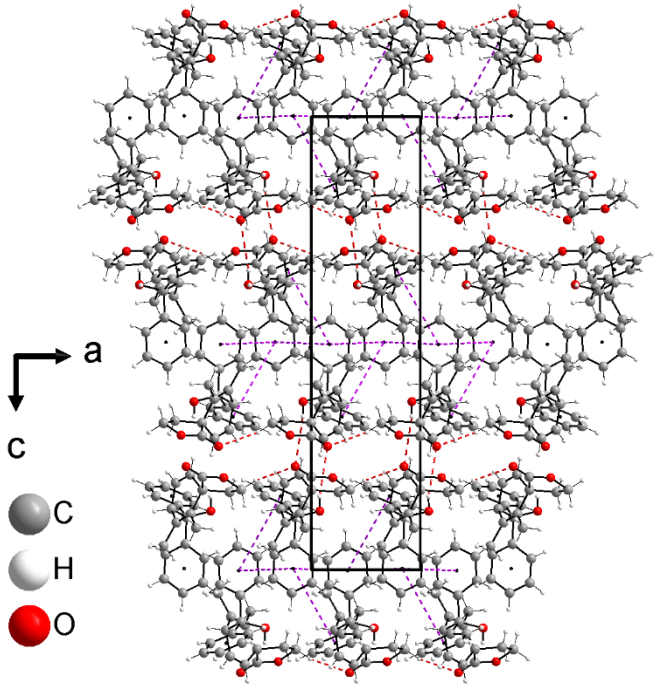
Molecular structure with atom labeling of *trans*-**27b**. Displacement ellipsoids are shown at the 50% probability level.



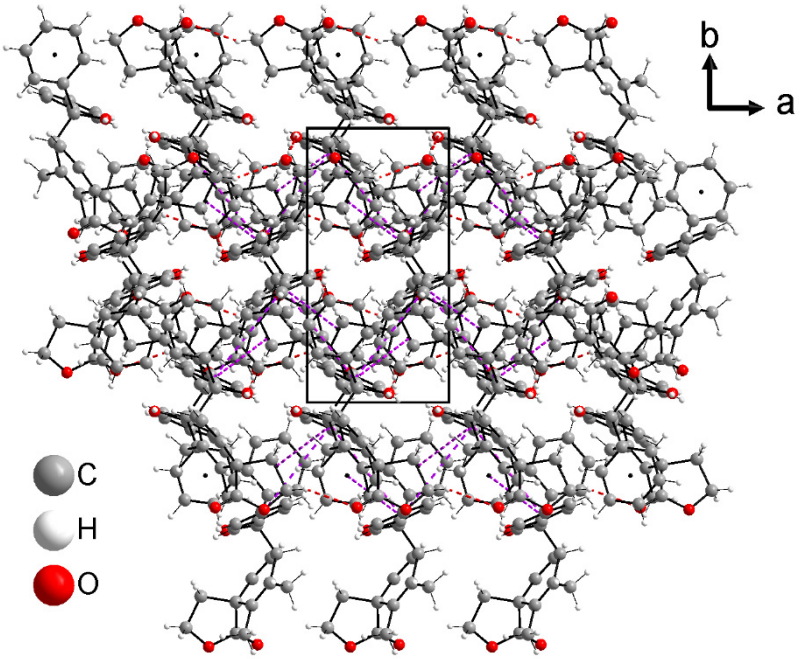
Weak O-H···O and C-H···O hydrogen bonds (red dashed lines) in *trans*-**27b** forming dimers.



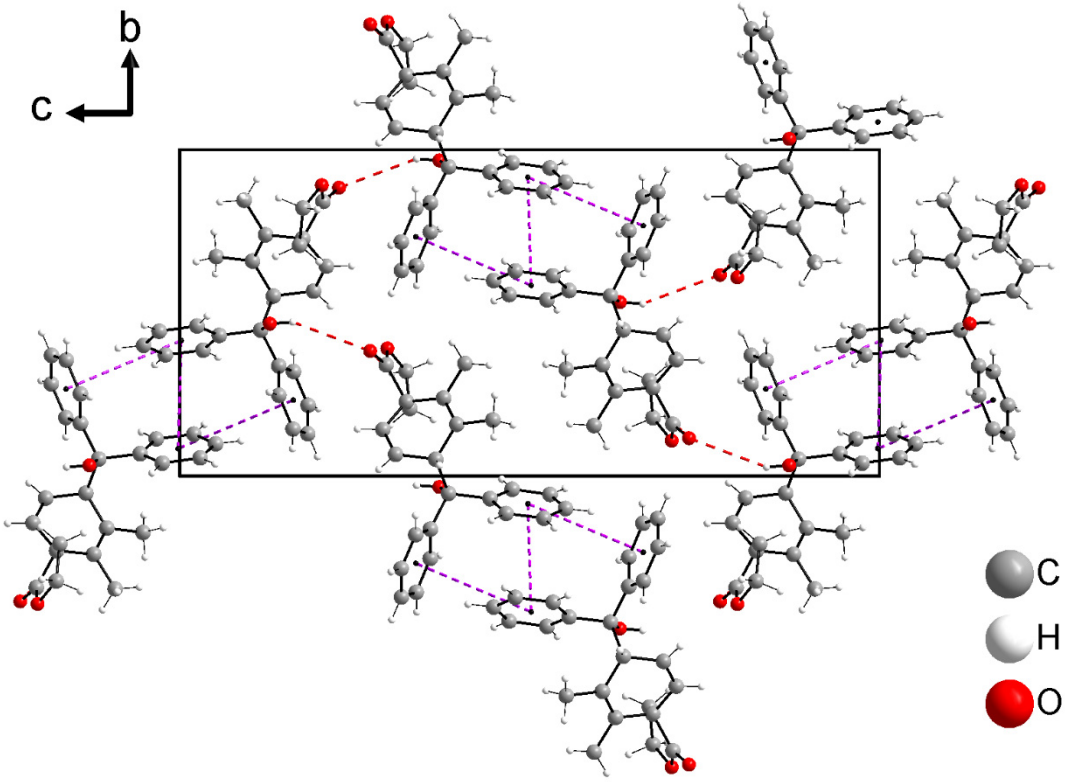
Stacking interactions (purple dotted lines) between the molecules in *trans*-**27b** (X marks the center of the aromatics, hydrogen atoms have been omitted).



Cell view of *trans*-**27b** looking along the crystallographic b axis.

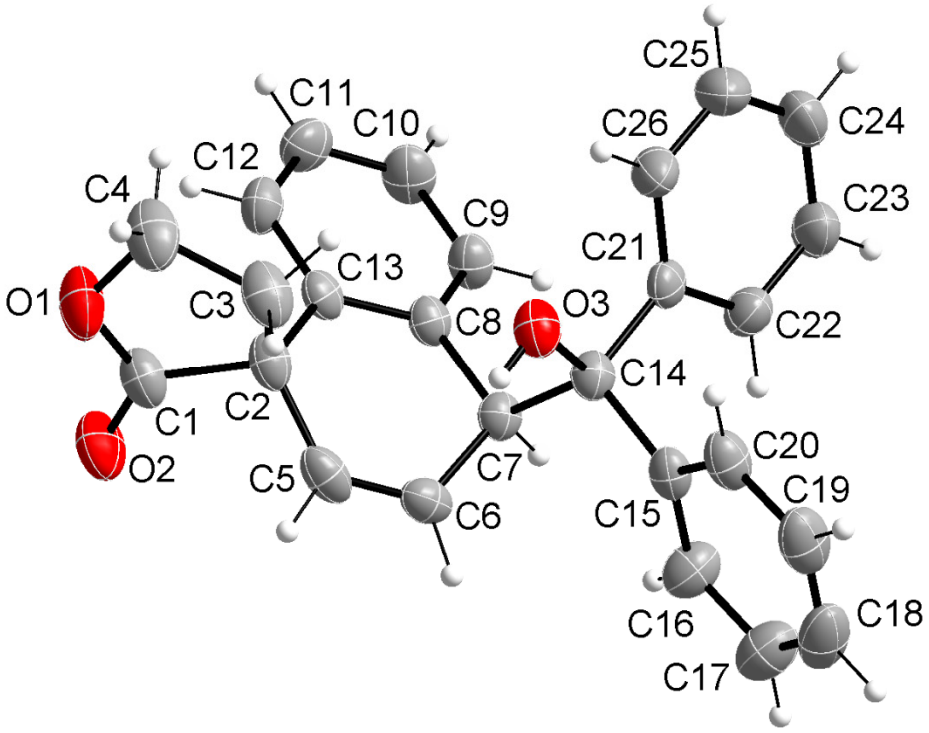


Cell view of *trans*-**27b** looking along the crystallographic c axis.

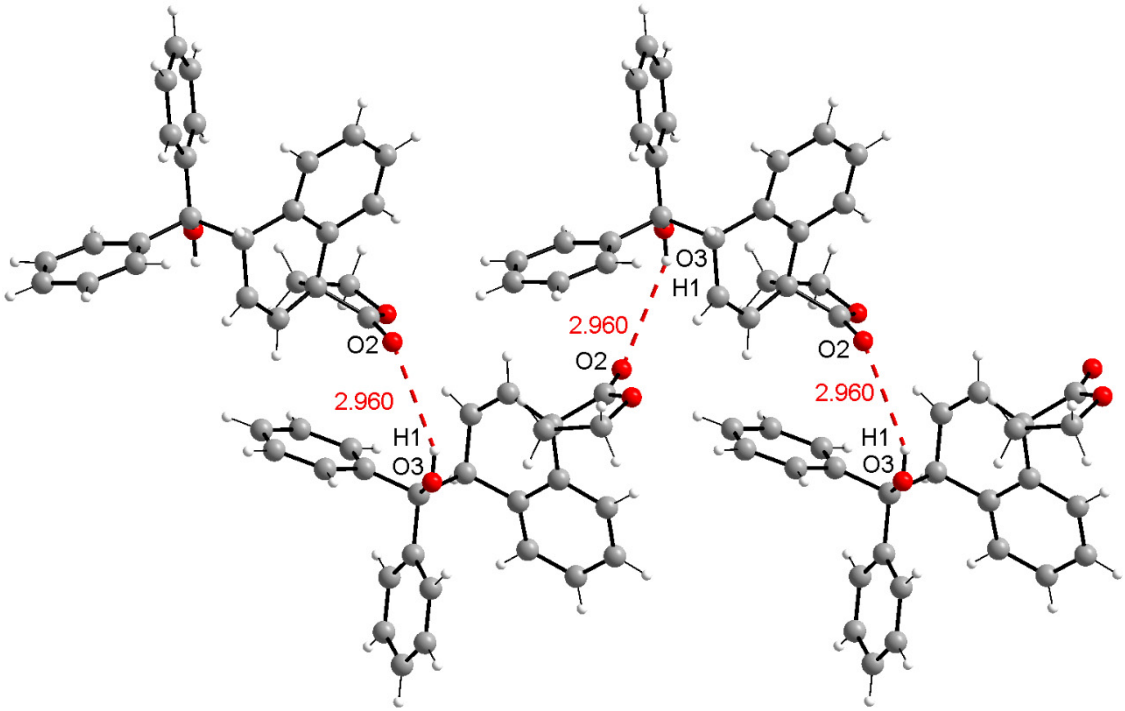


Cell view of *trans*-**27b** looking along the crystallographic a axis.

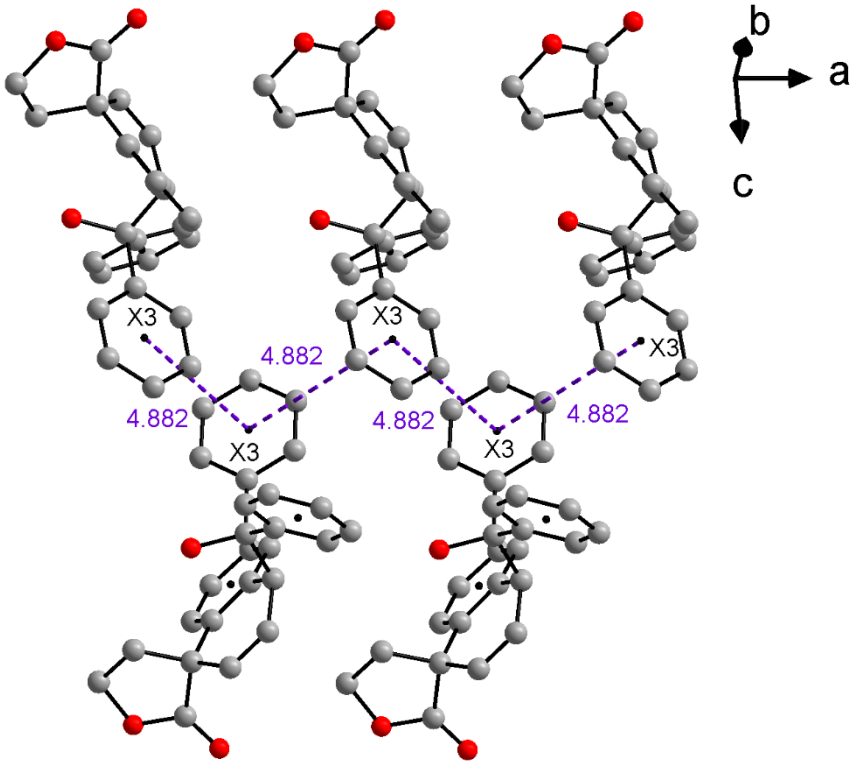
Compound *trans*-27c:



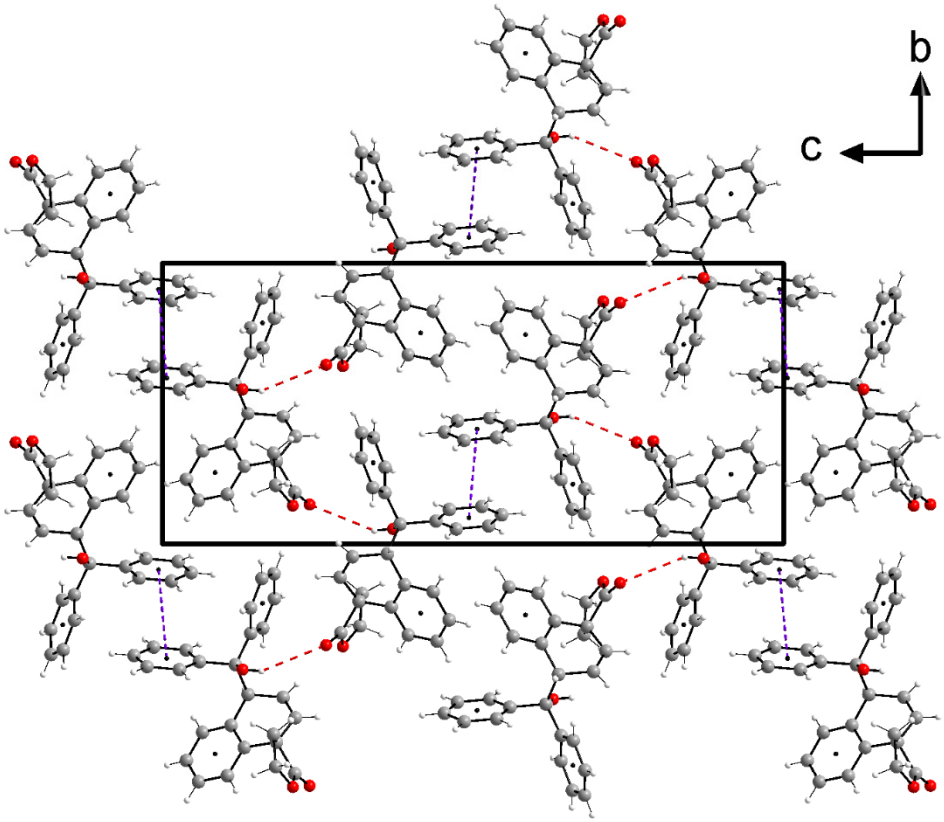
Molecular structure with atom labeling of *trans*-**27c**. Displacement ellipsoids are shown at the 50% probability level.



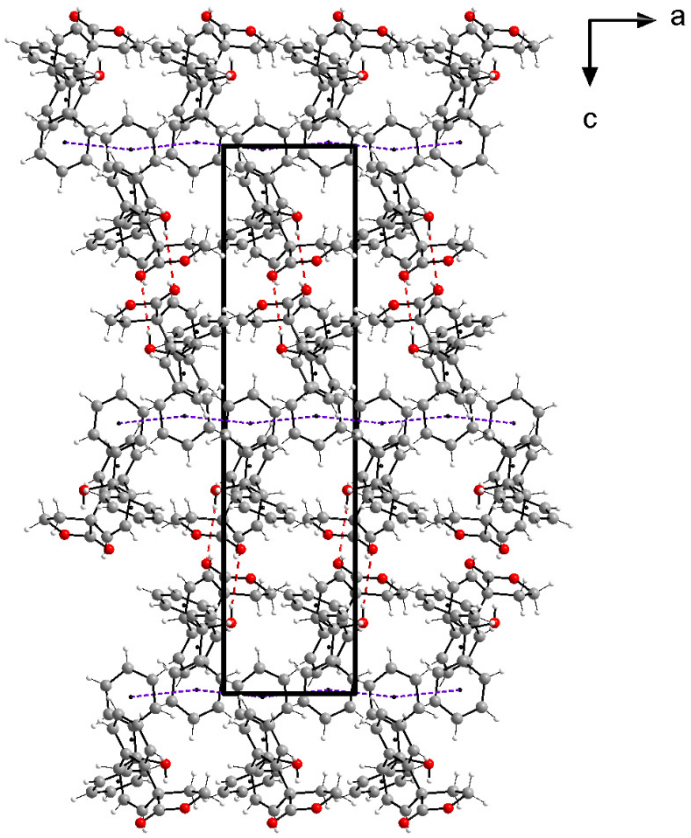
Weak O-H···O hydrogen bonding (red dashed lines) in *trans*-**27c** forming chains.



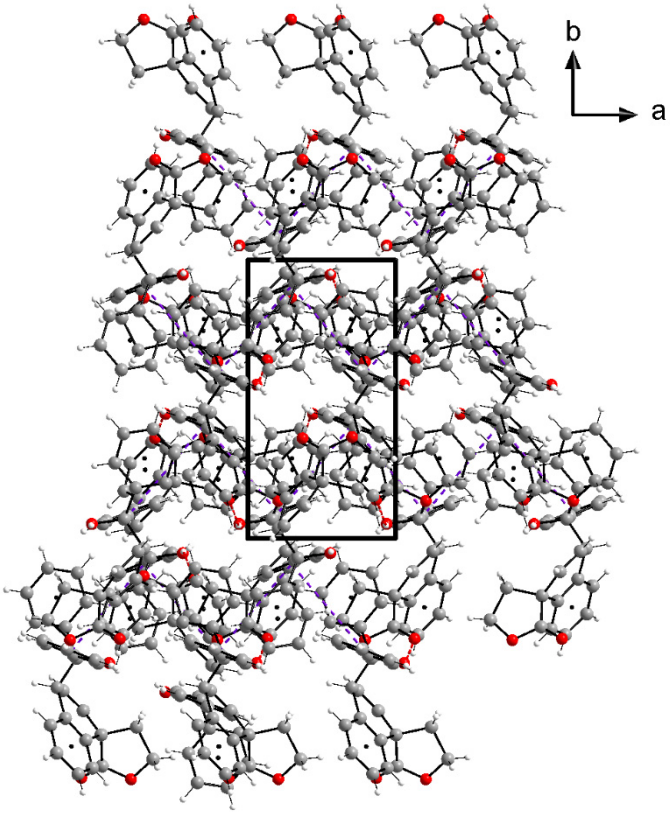
Stacking interactions (purple dotted lines) between the molecules in *trans*-**27c** (X marks the center of the aromatics, hydrogen atoms have been omitted).



Cell view of *trans*-**27c** looking along the crystallographic a axis.



Cell view of *trans*-**27c** looking along the crystallographic b axis.



Cell view of *trans*-**27c** looking along the crystallographic c axis.