Electronic Supplementary Material (ESI) for RSC Advances

Iodine Mediated Reactions of Quinones and *N*-substituted Amino Esters to 2-Substituted benzo[*f*]isoindole-4,9-diones

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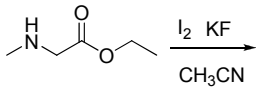
1. The GC-MS Spectra of the reaction

a) A mixture of sarcosine ethyl ester (**2a**, 2.0 mmol, 0.234 g, 2.0 equiv.), KF (2.0 mmol, 0.116 g, 2.0 equiv.), Iodine (1 mmol, 0.254 g, 1.0 equiv.) in CH_3CN (5.0 mL), was stirred at room temperature for 1h. Then the mixture detected by GC-MS. The following two intermediates were captured.



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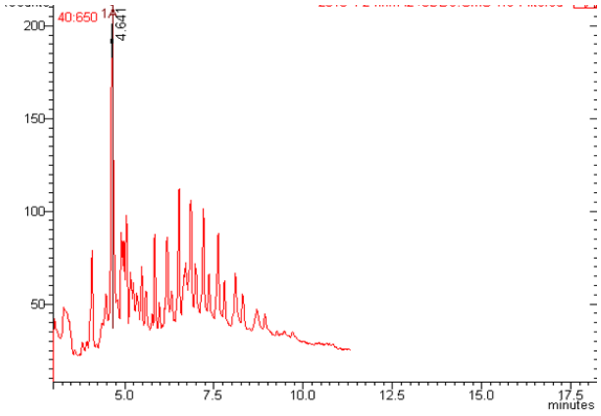
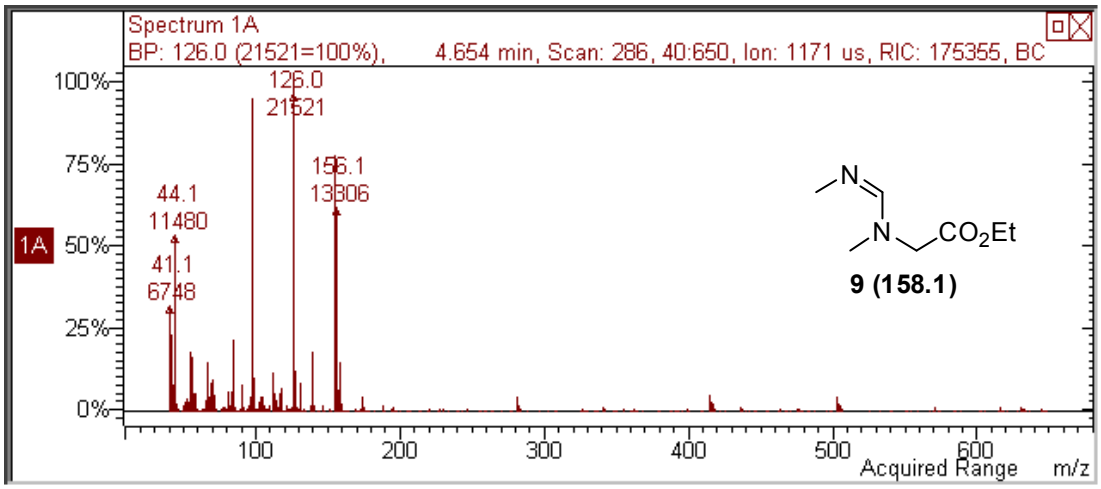
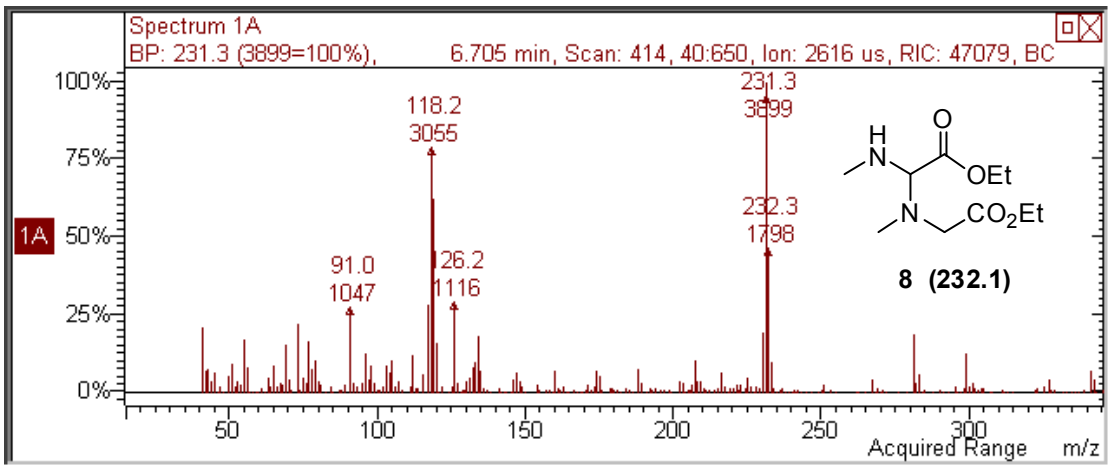
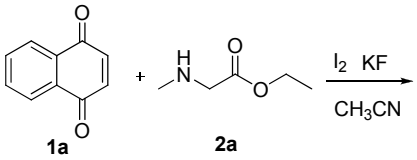
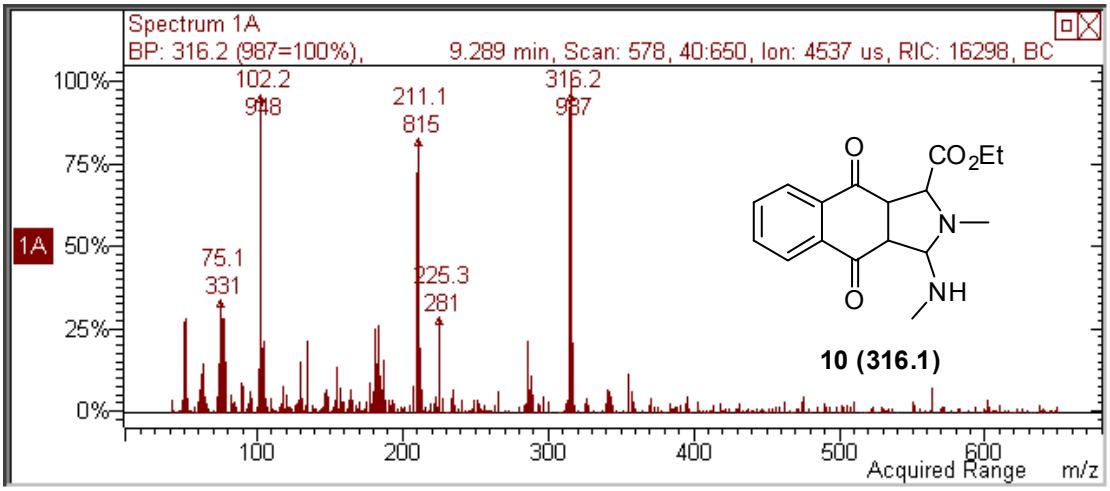






b) A mixture of 1,4-naphthoquinone (1a, 1.0 mmol, 0.158 g, 1.0 equiv.), sarcosine ethyl ester (**2a**, 2.0 mmol, 0.234 g, 2.0 equiv.), KF (2.0 mmol, 0.116 g, 2.0 equiv.), Iodine (0.5 mmol, 0.127g, 0.5 equiv.) in CH_3CN (5.0 mL), was stirred at refluxing temperature for 2 h. Then, the mixture detected by GC-MS and the following intermediate was captured.





2. NMR Spectra

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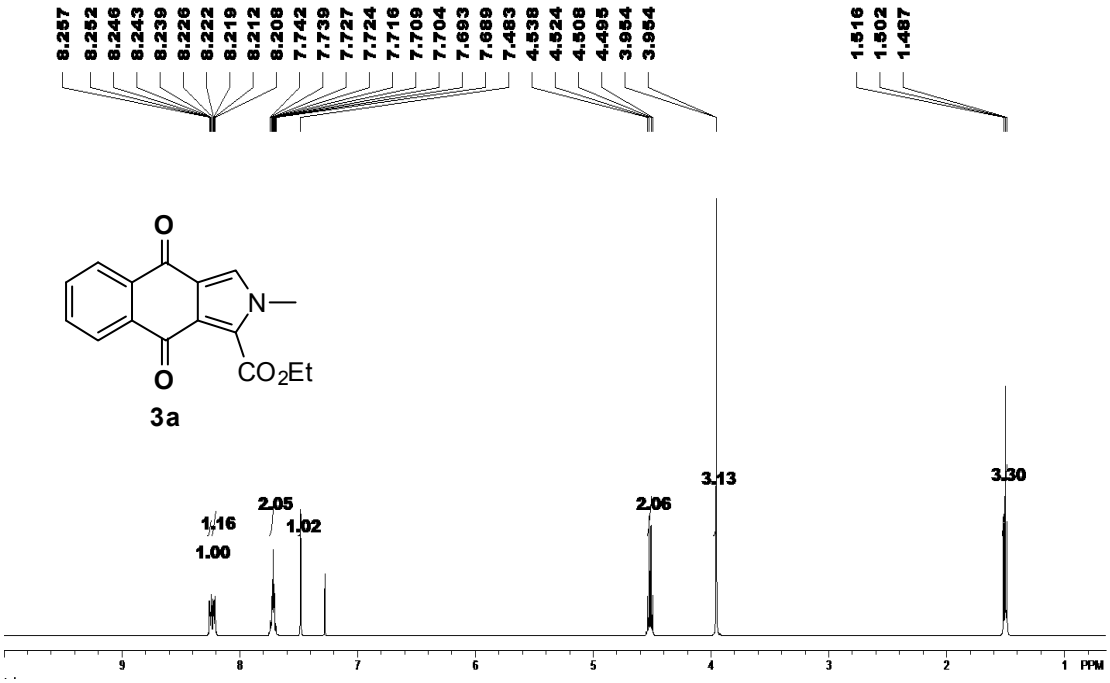

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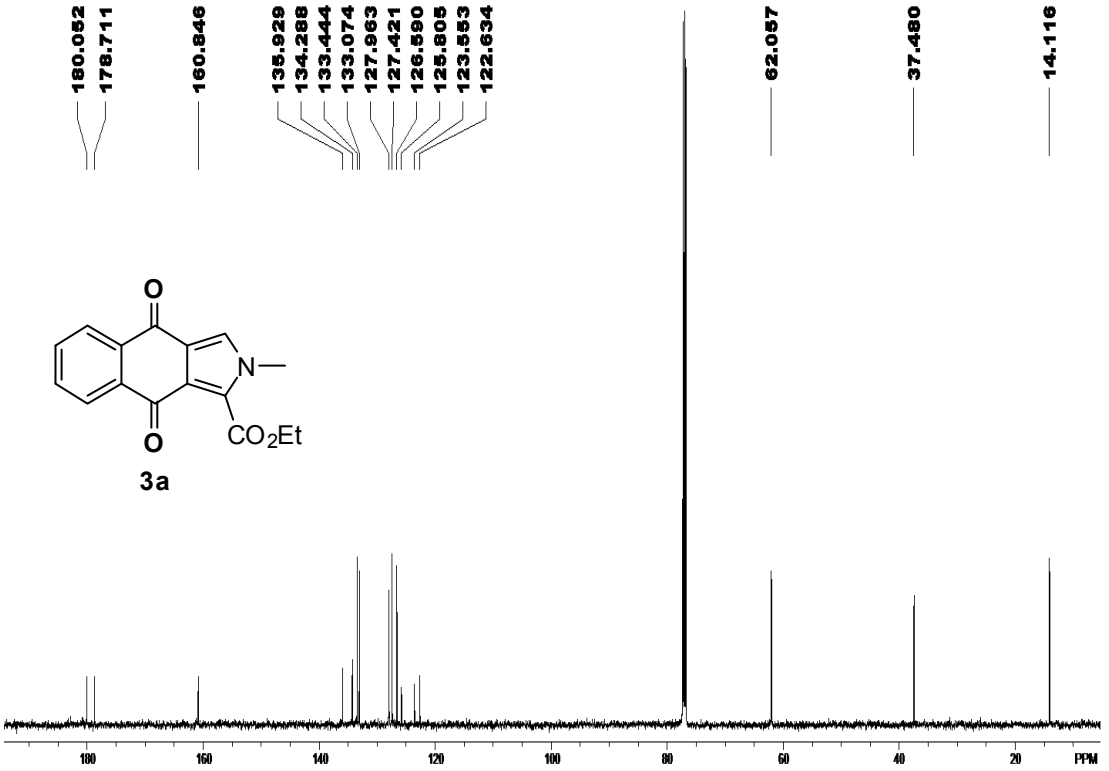

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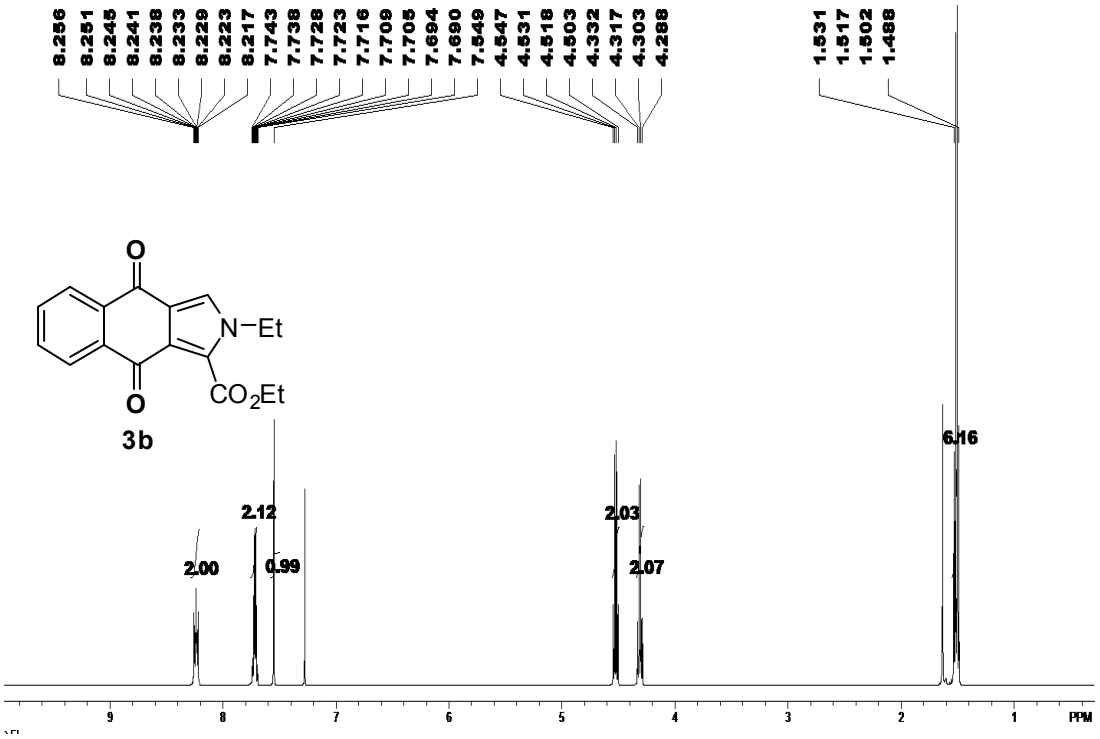

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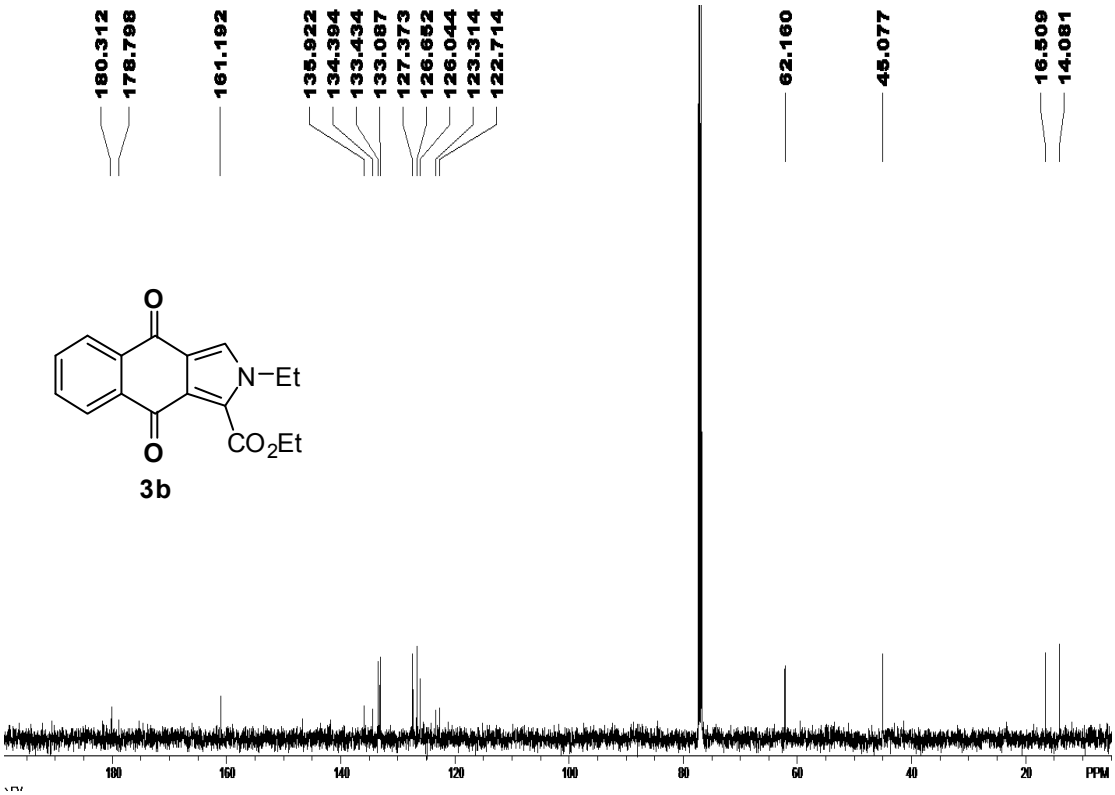

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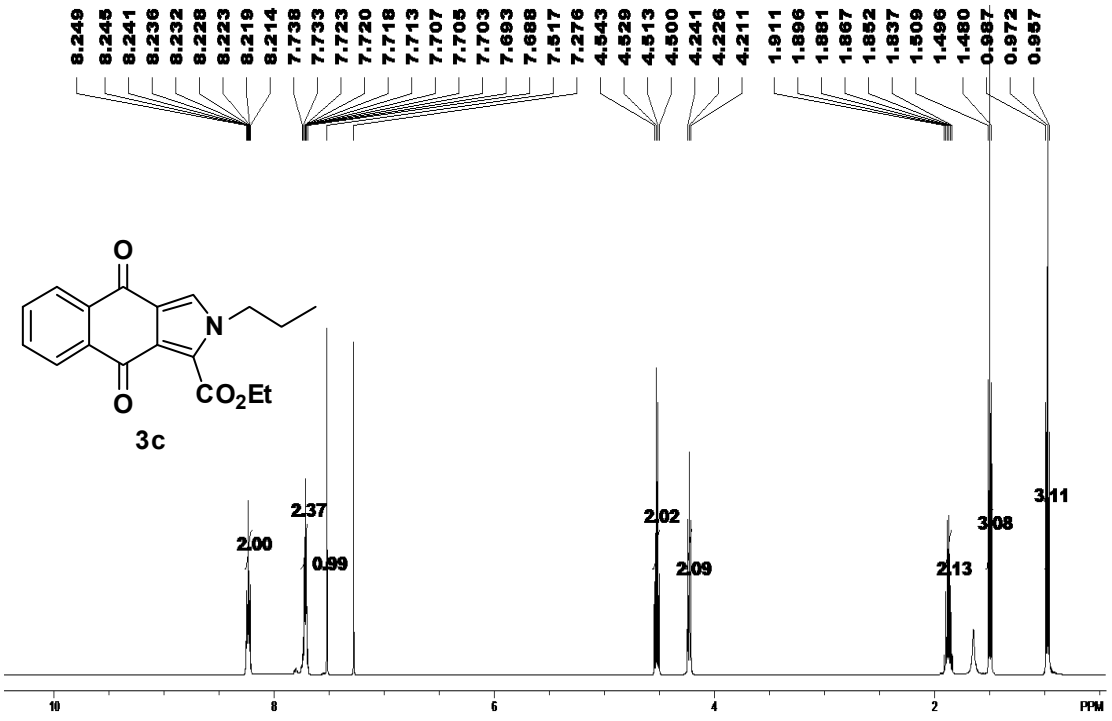

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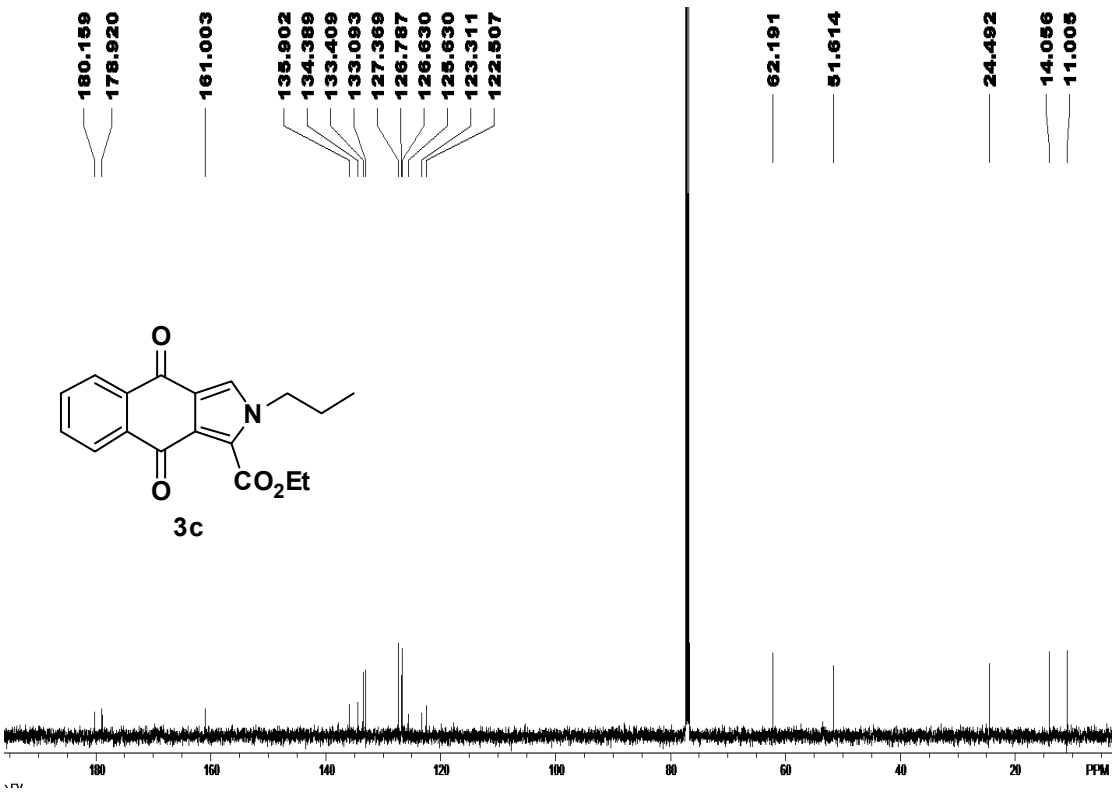

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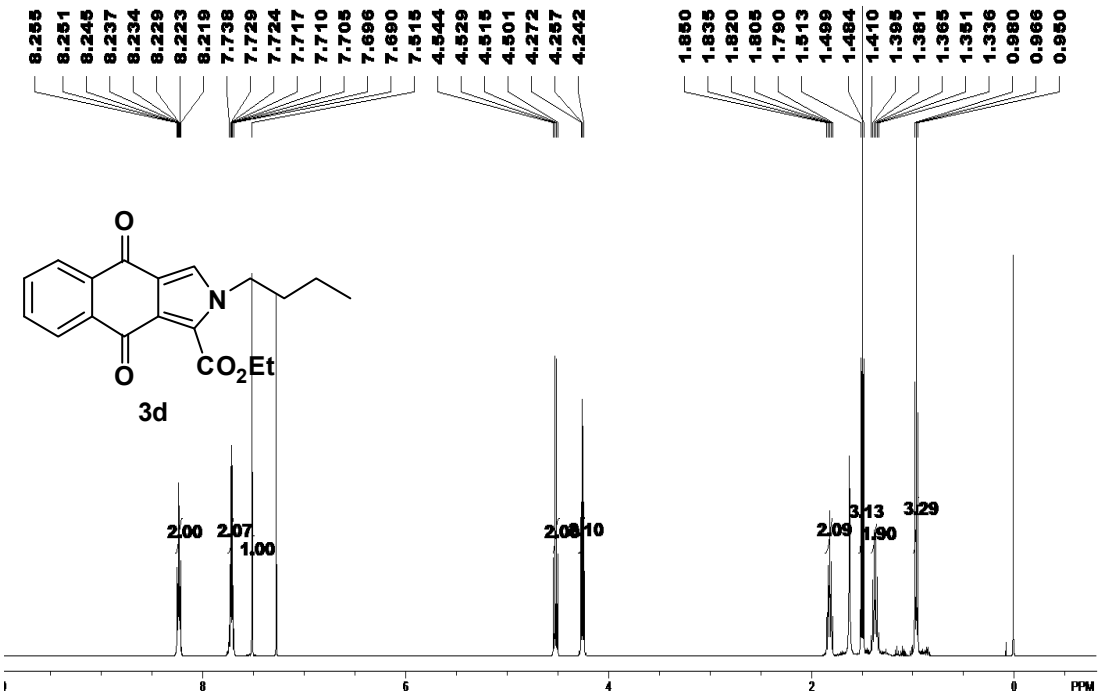

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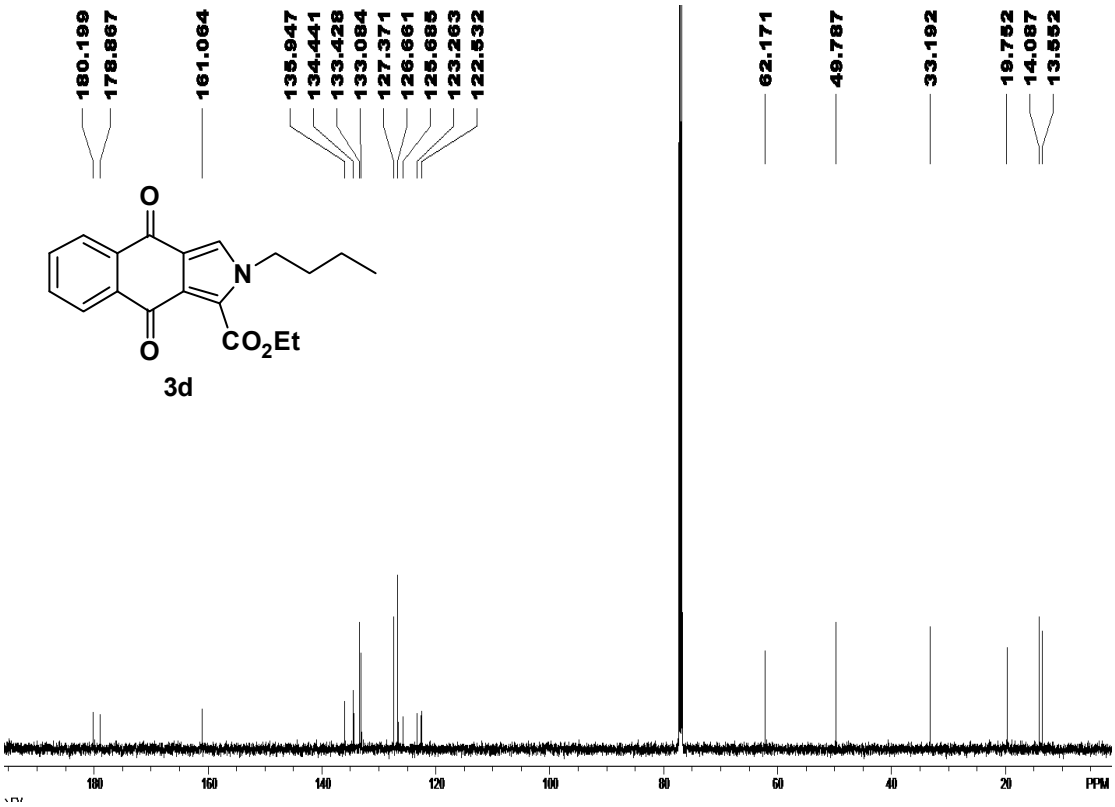

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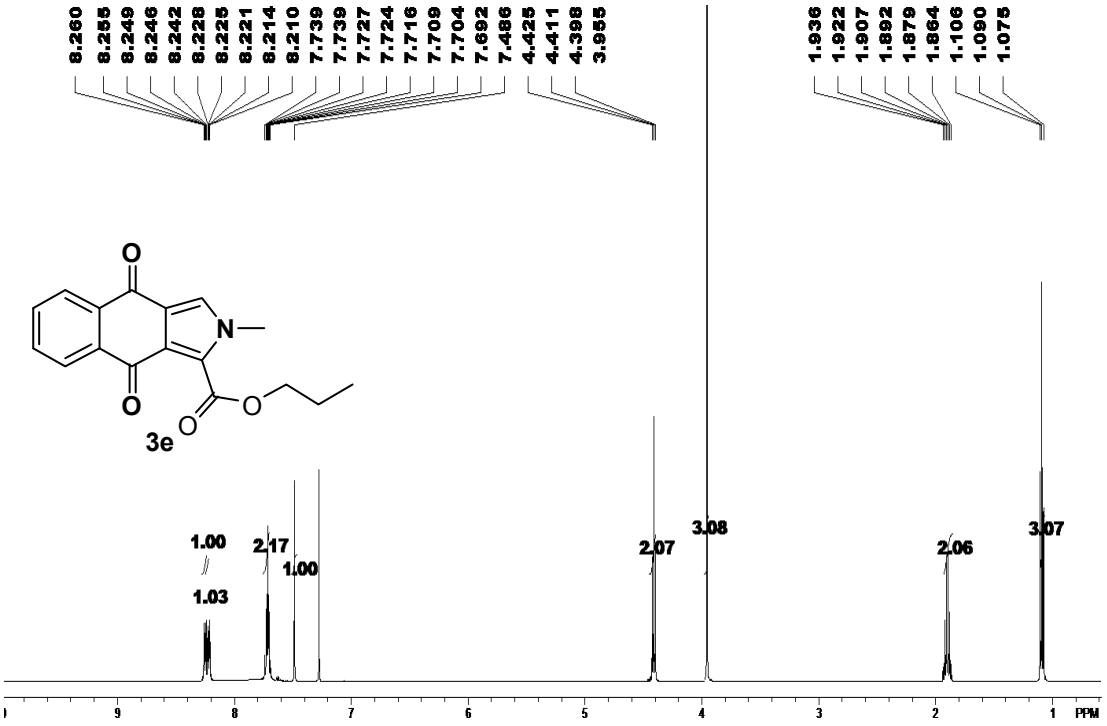

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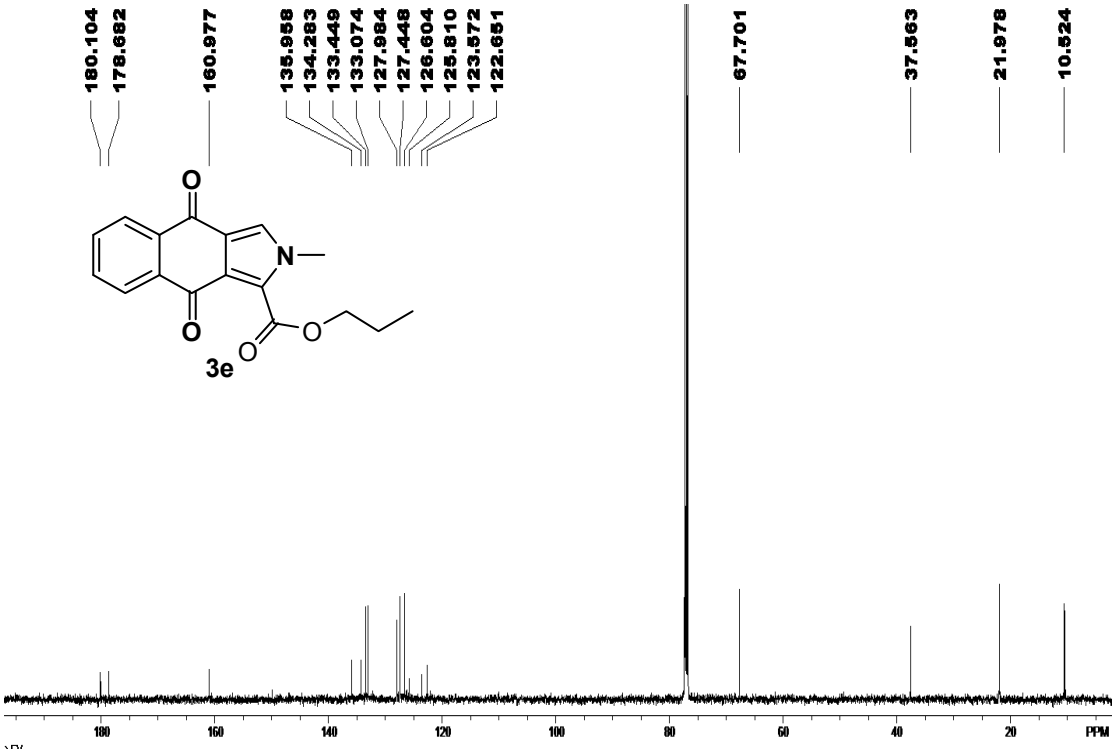

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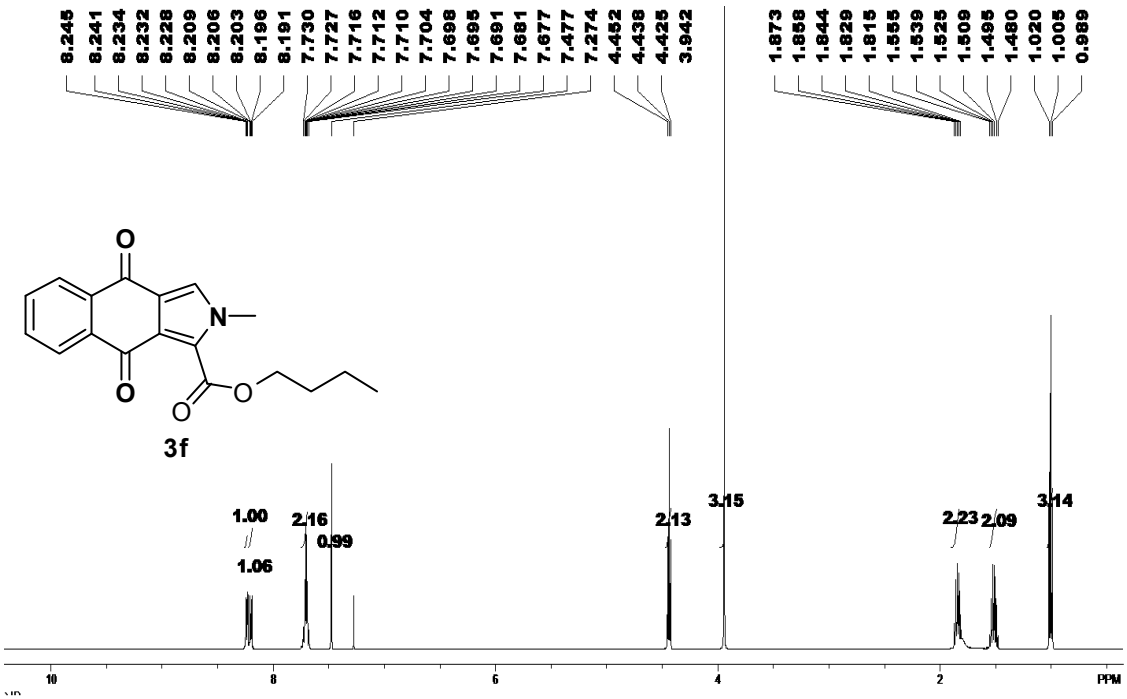

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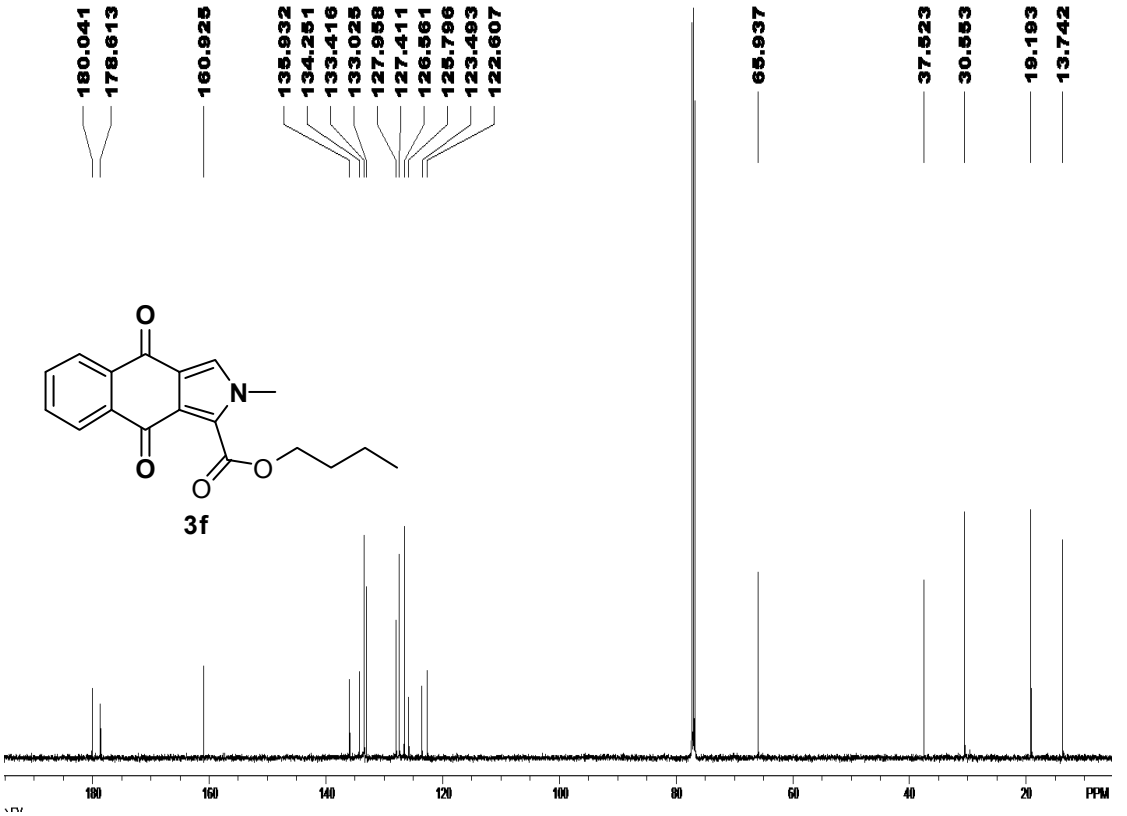

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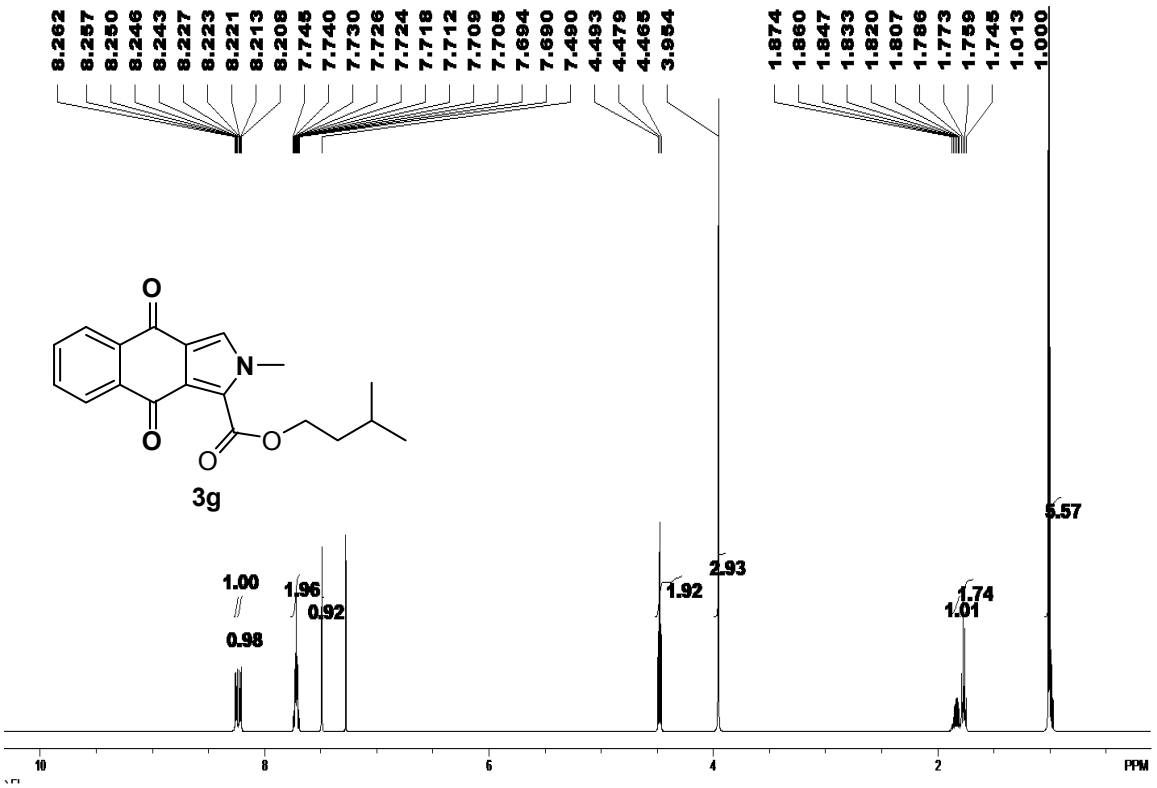

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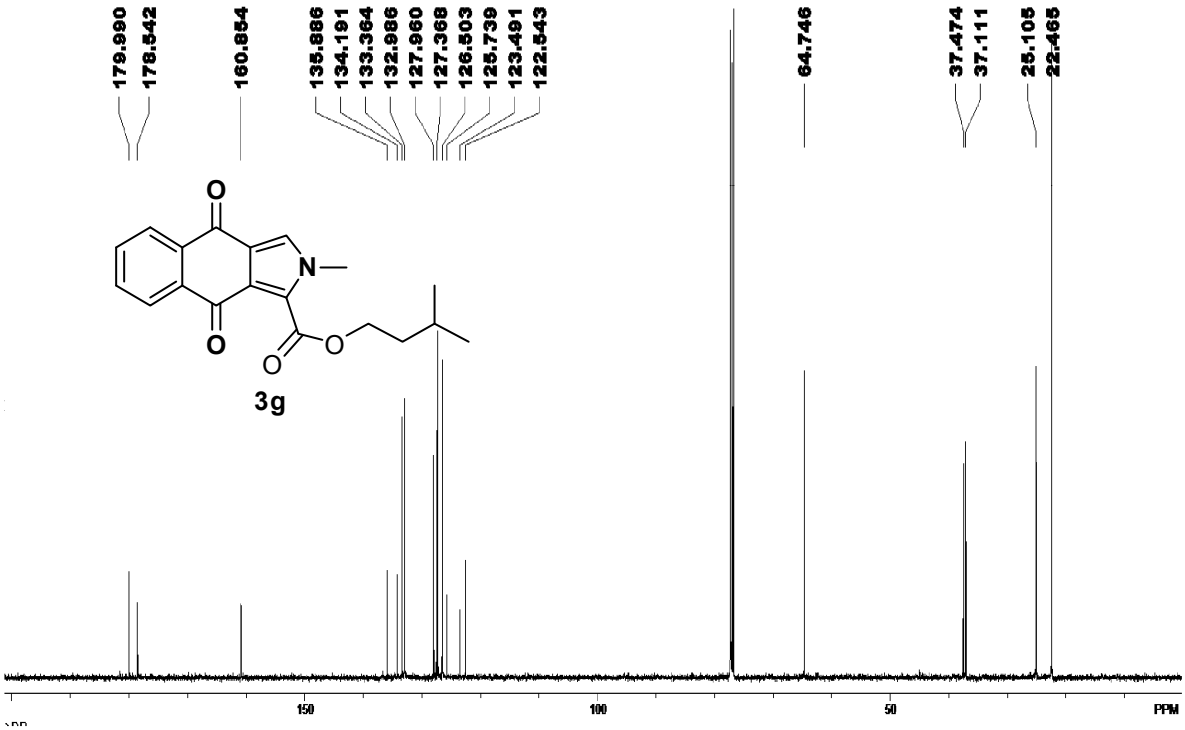

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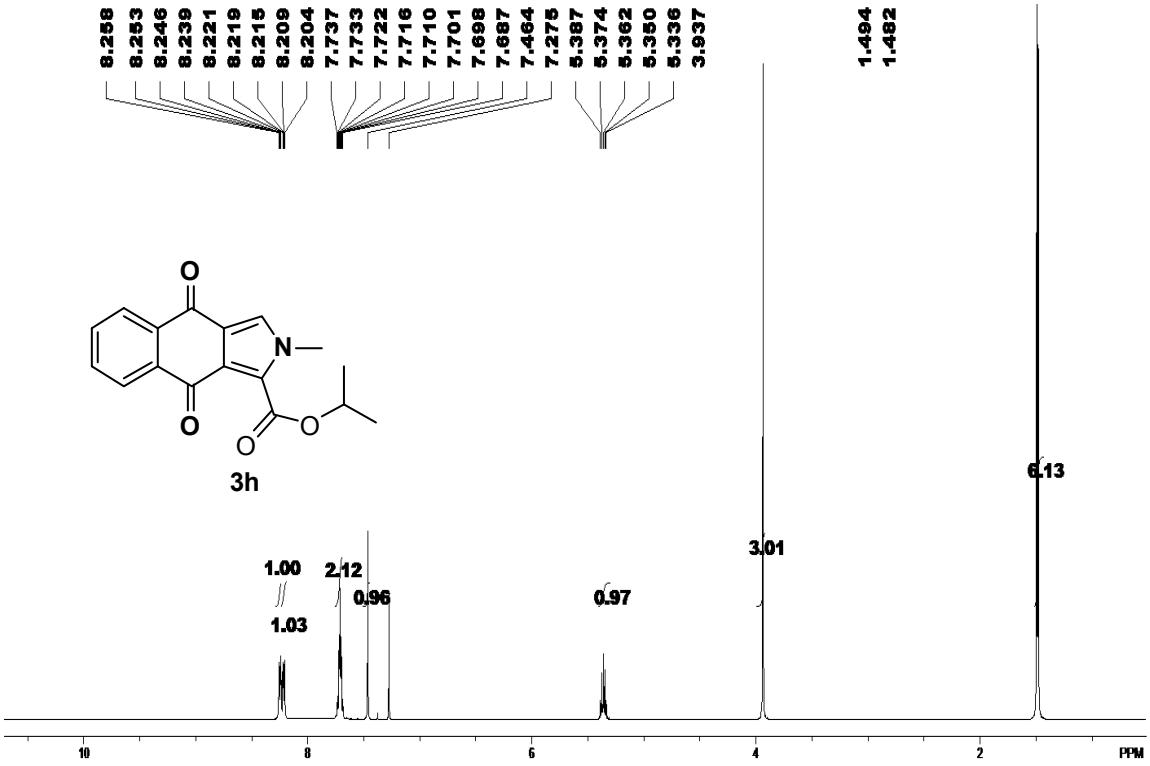

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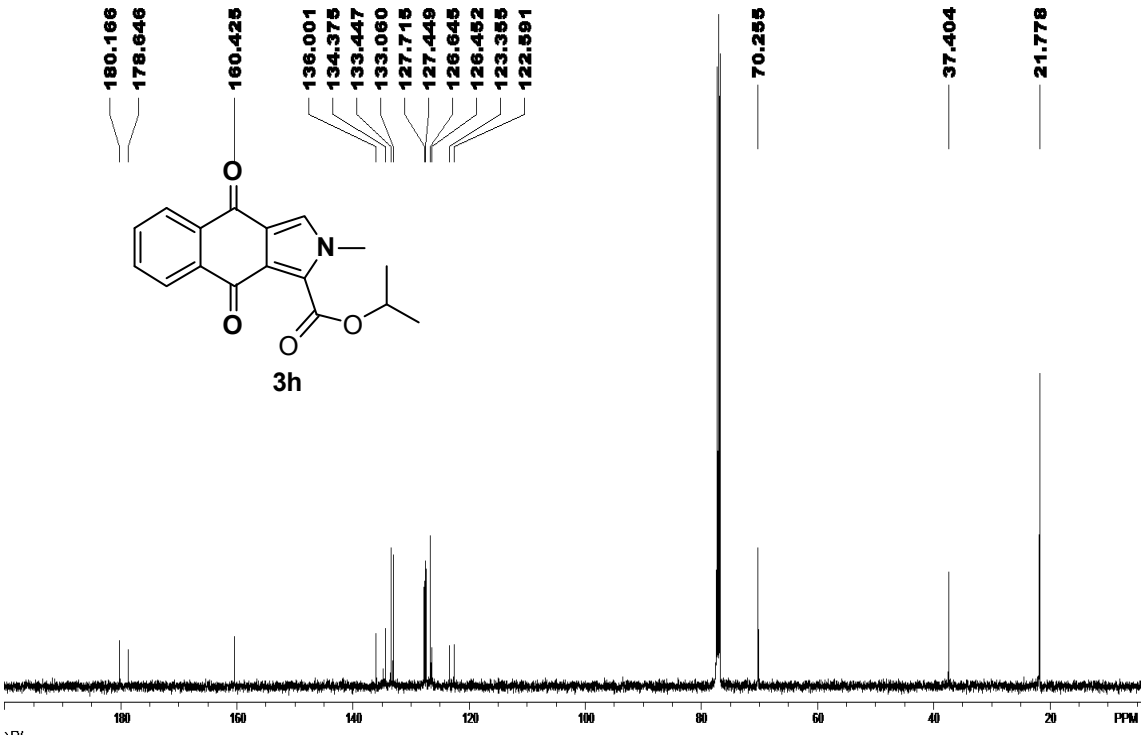

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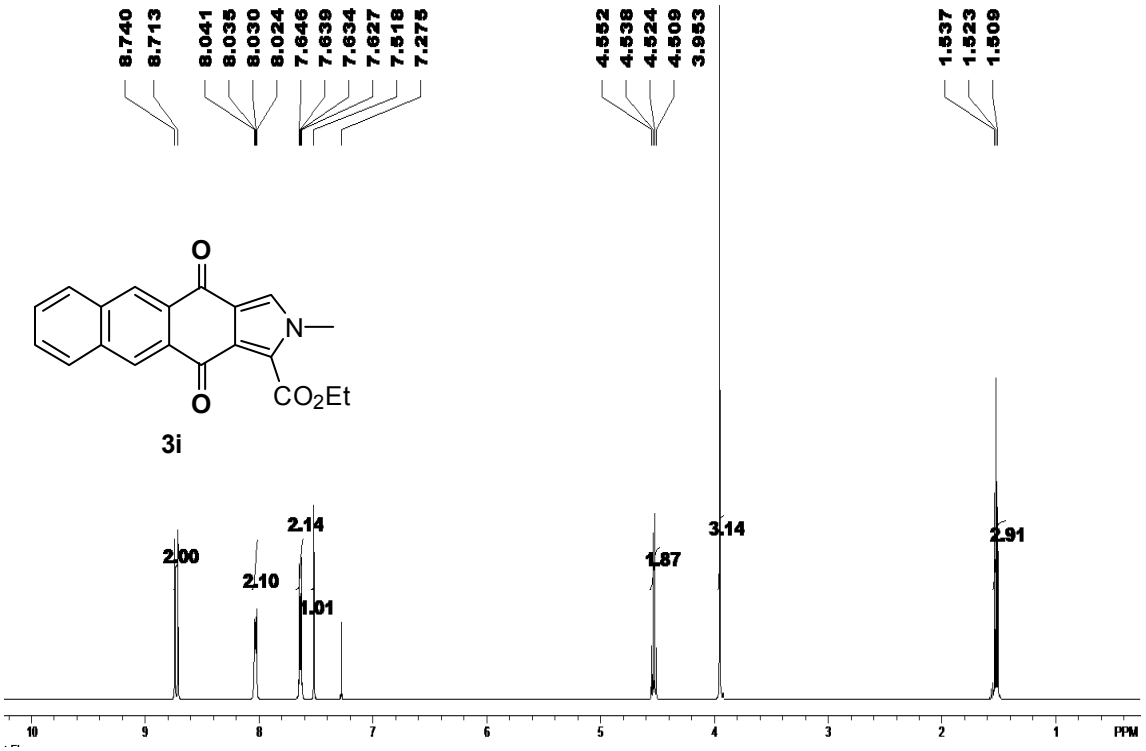

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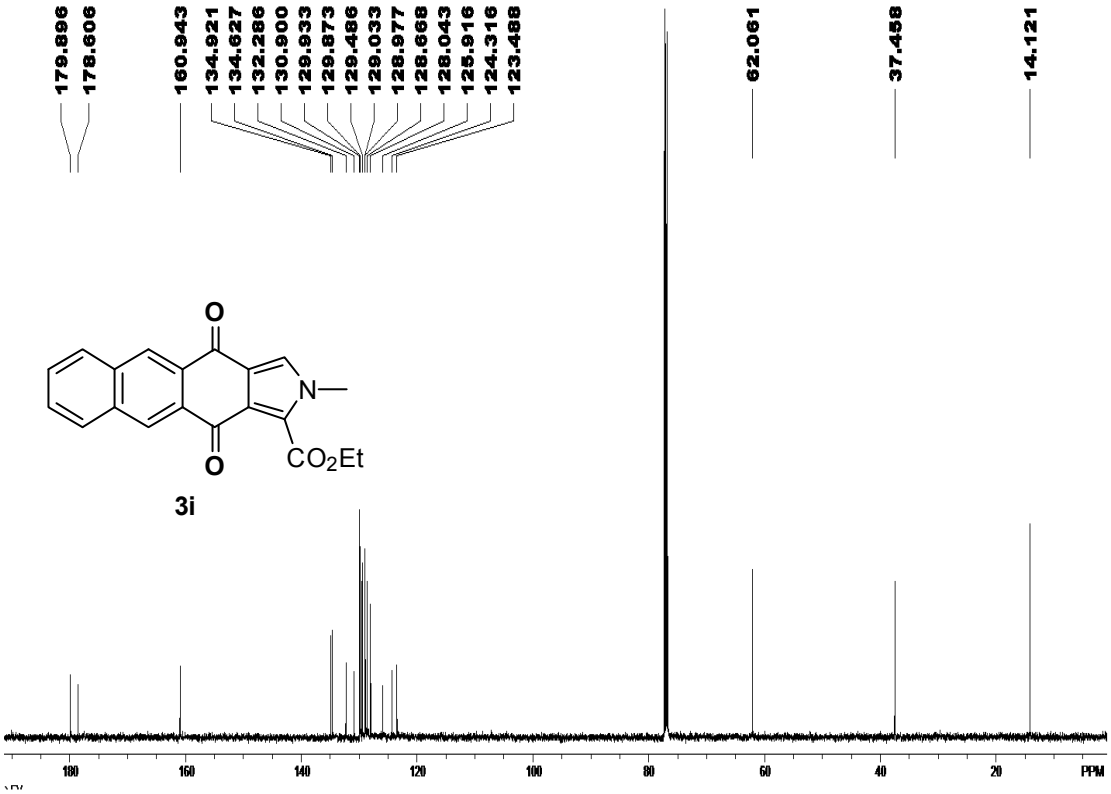

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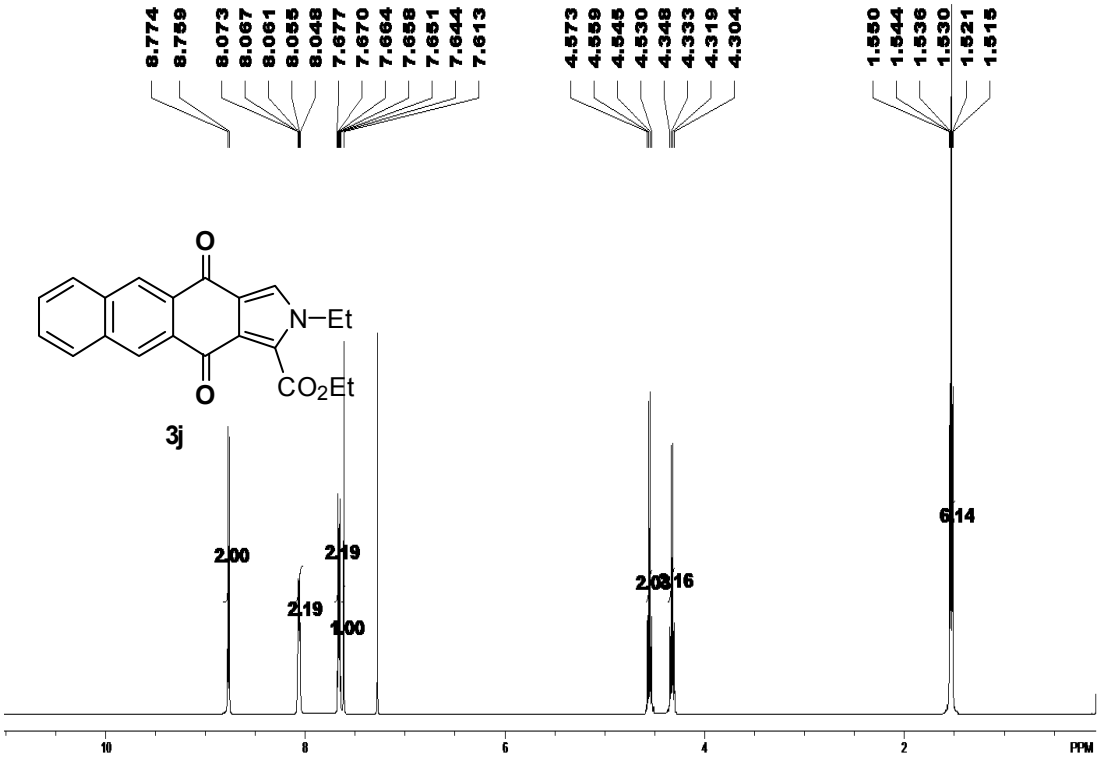

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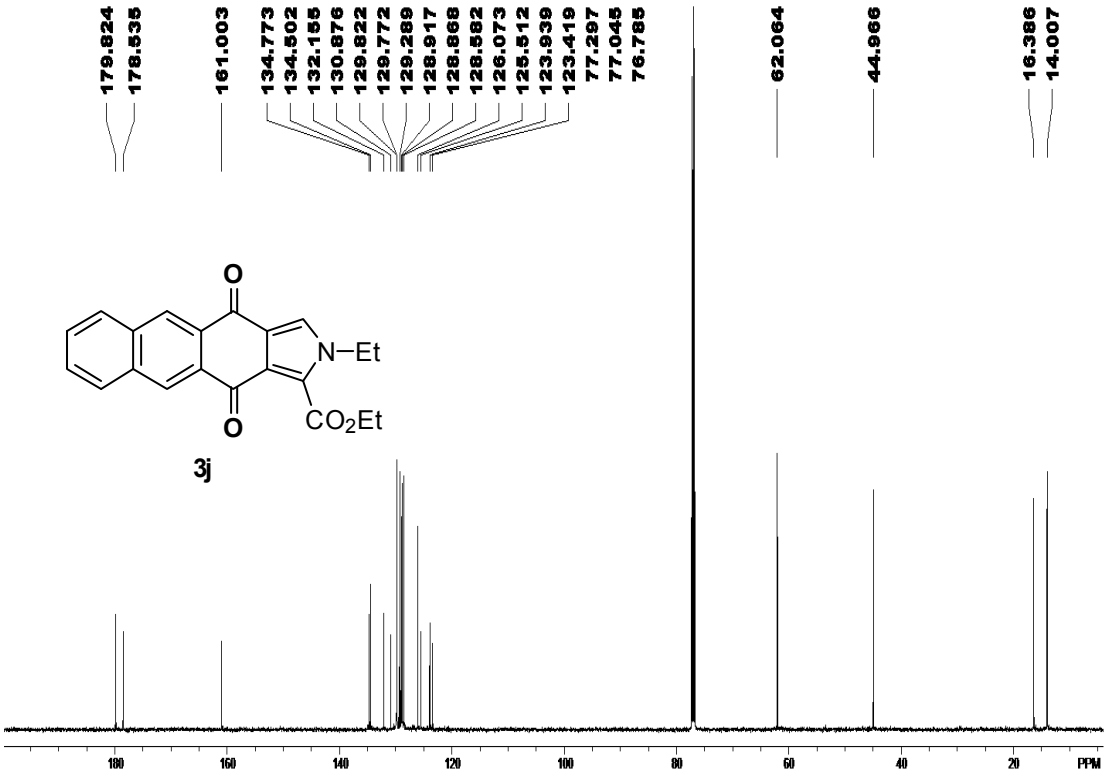

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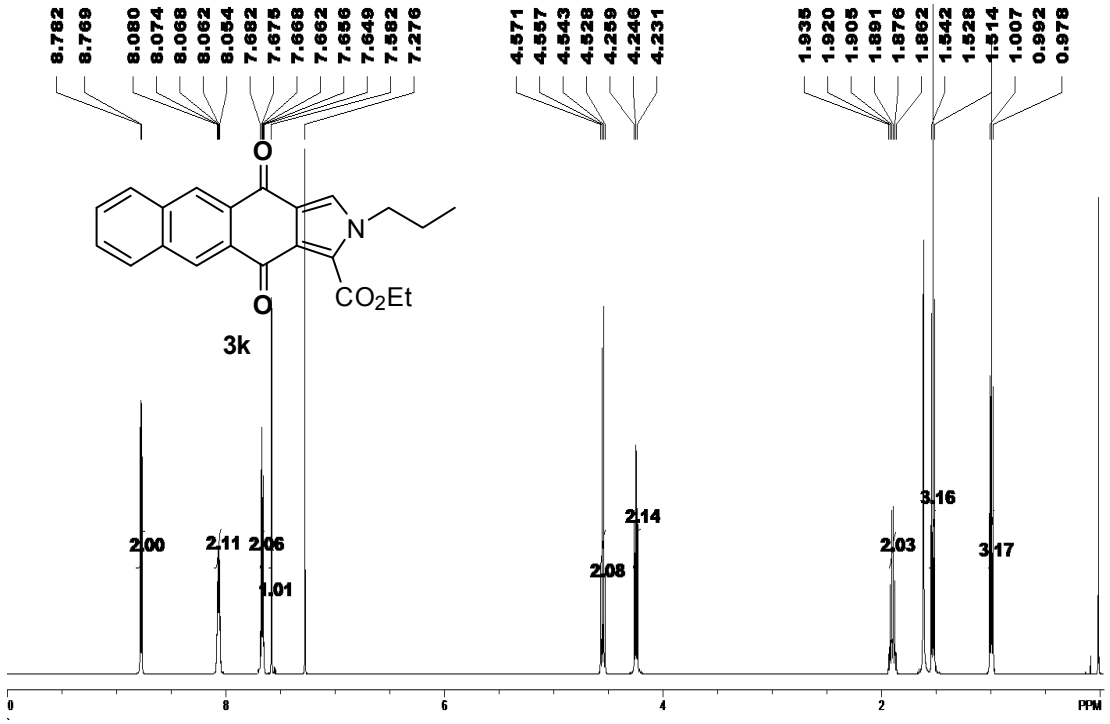

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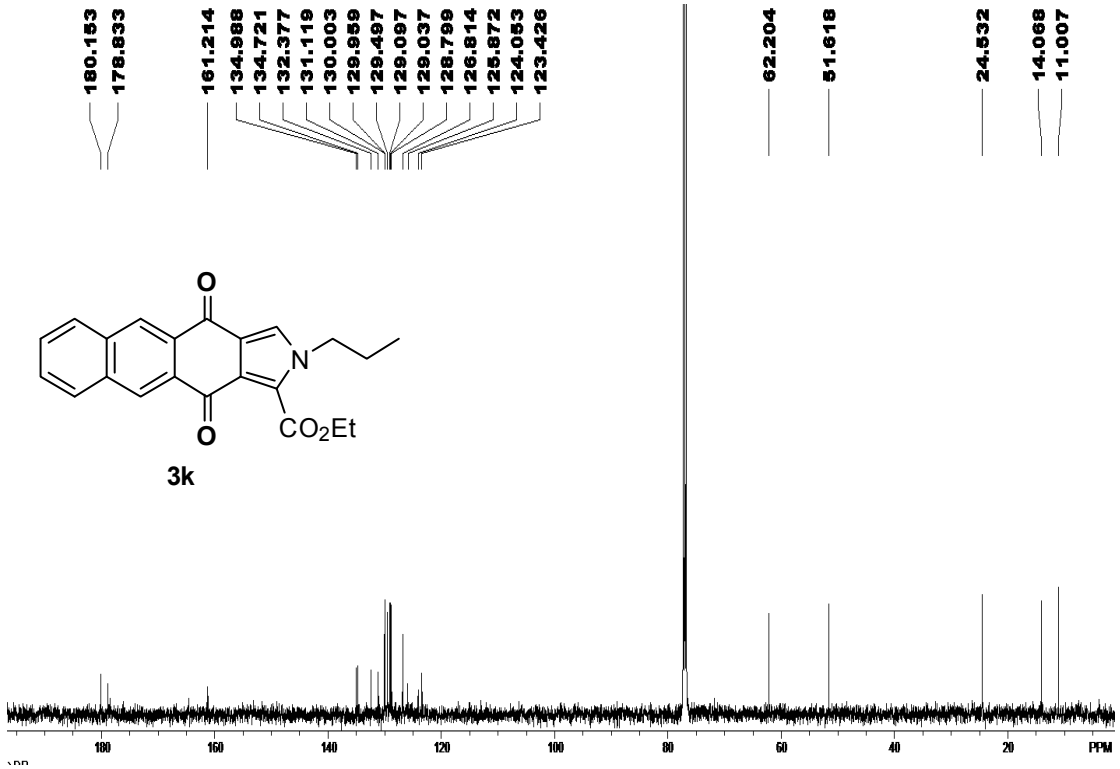

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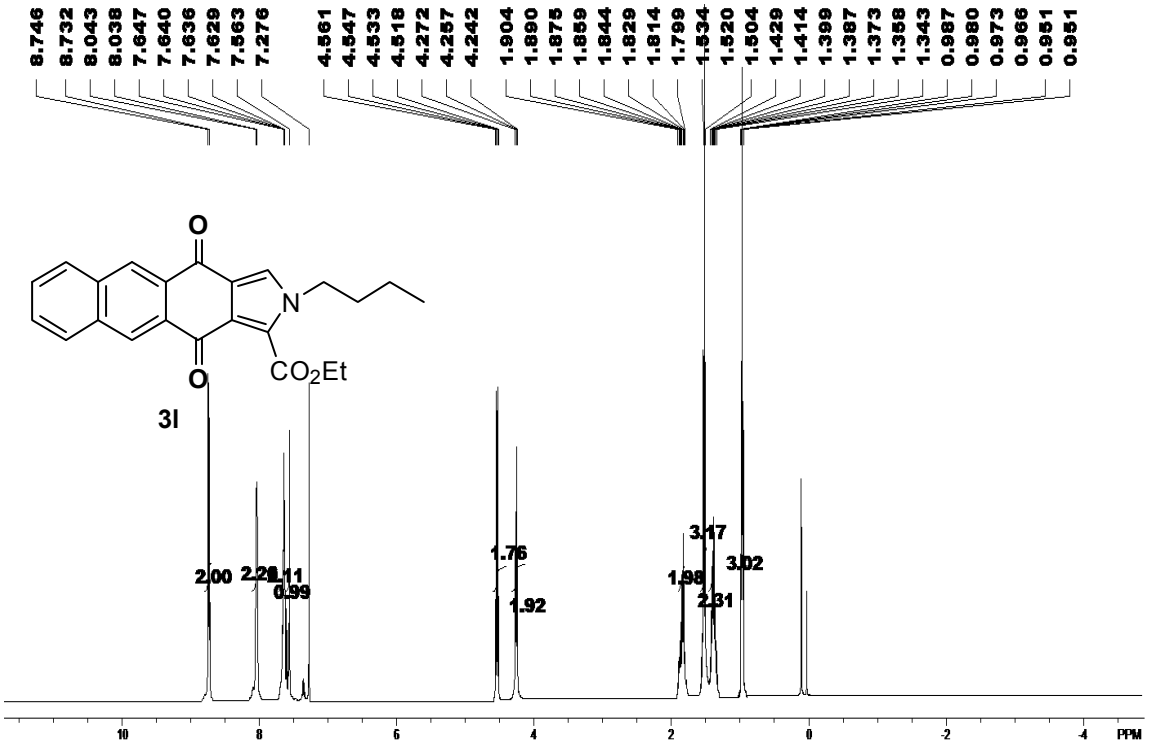

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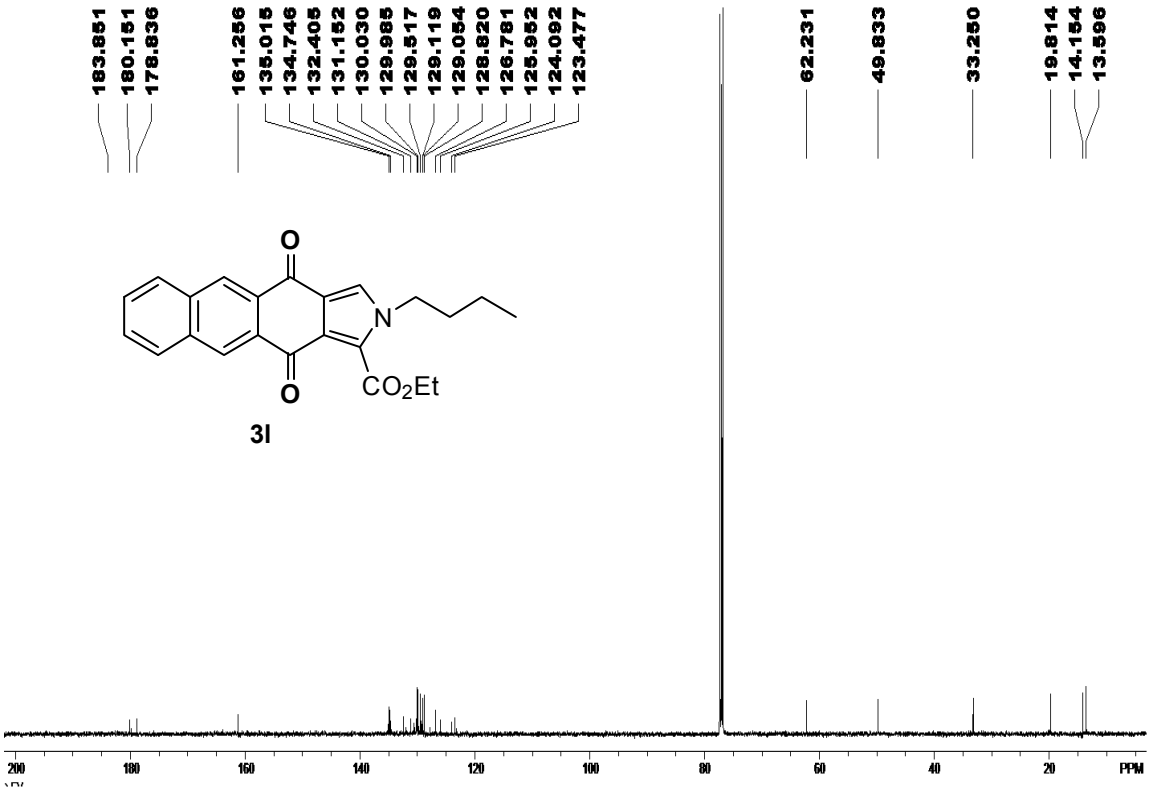

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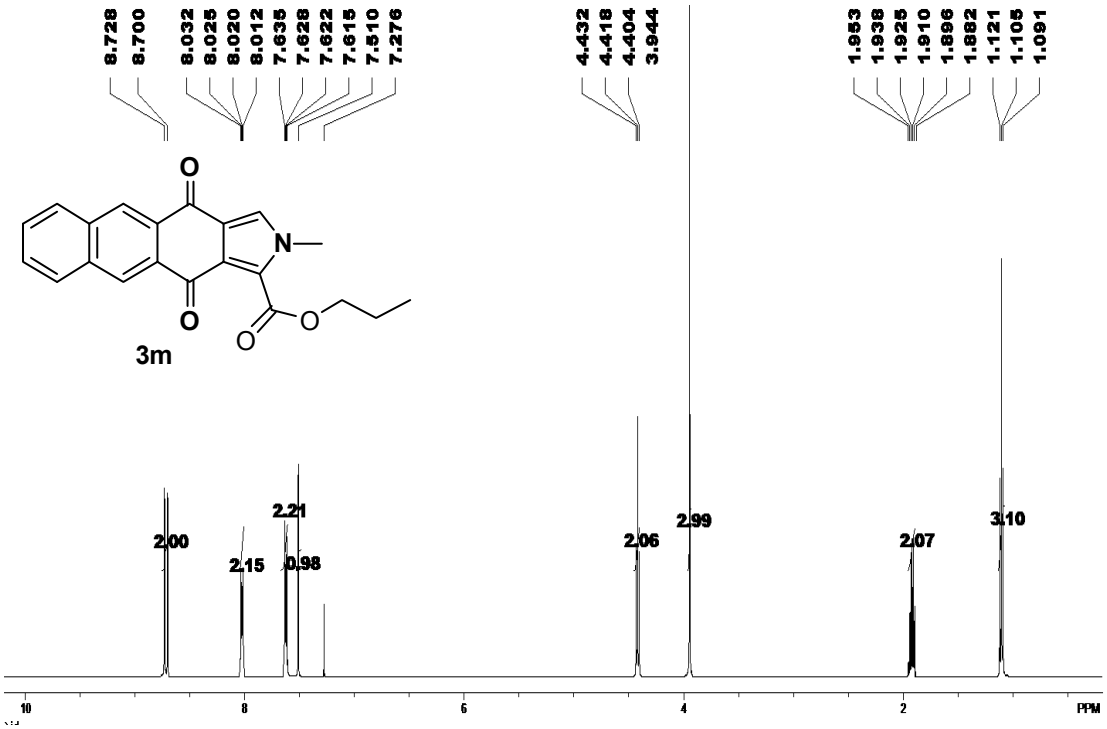

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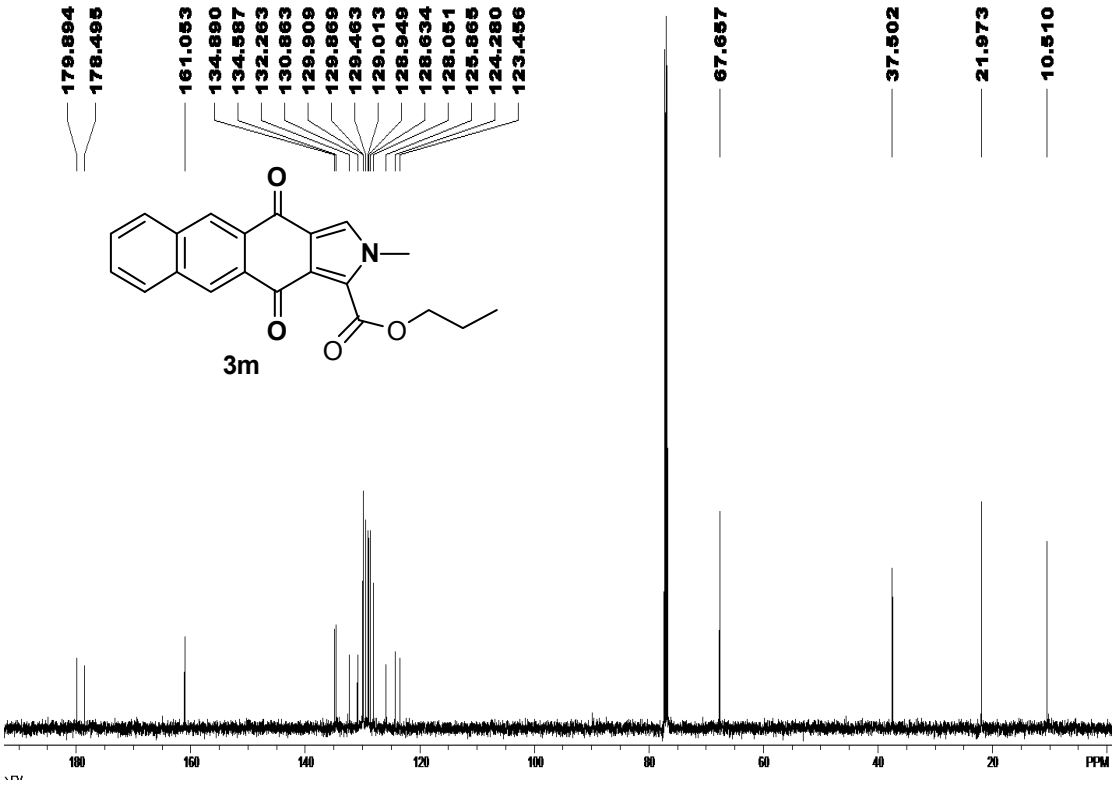

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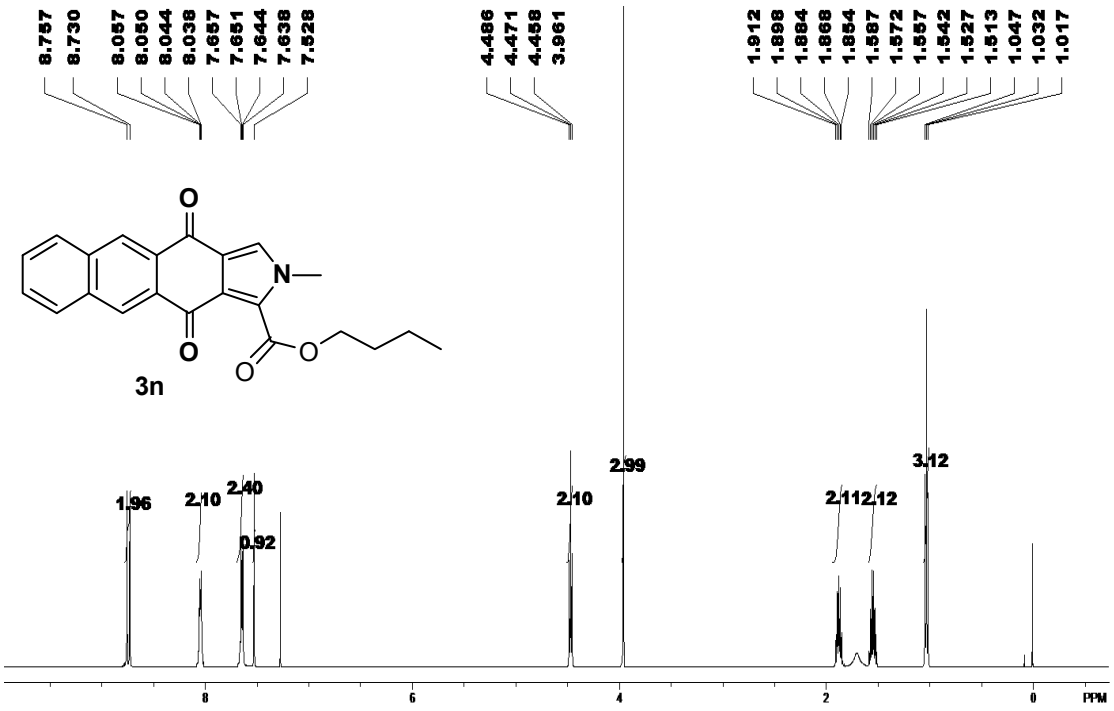

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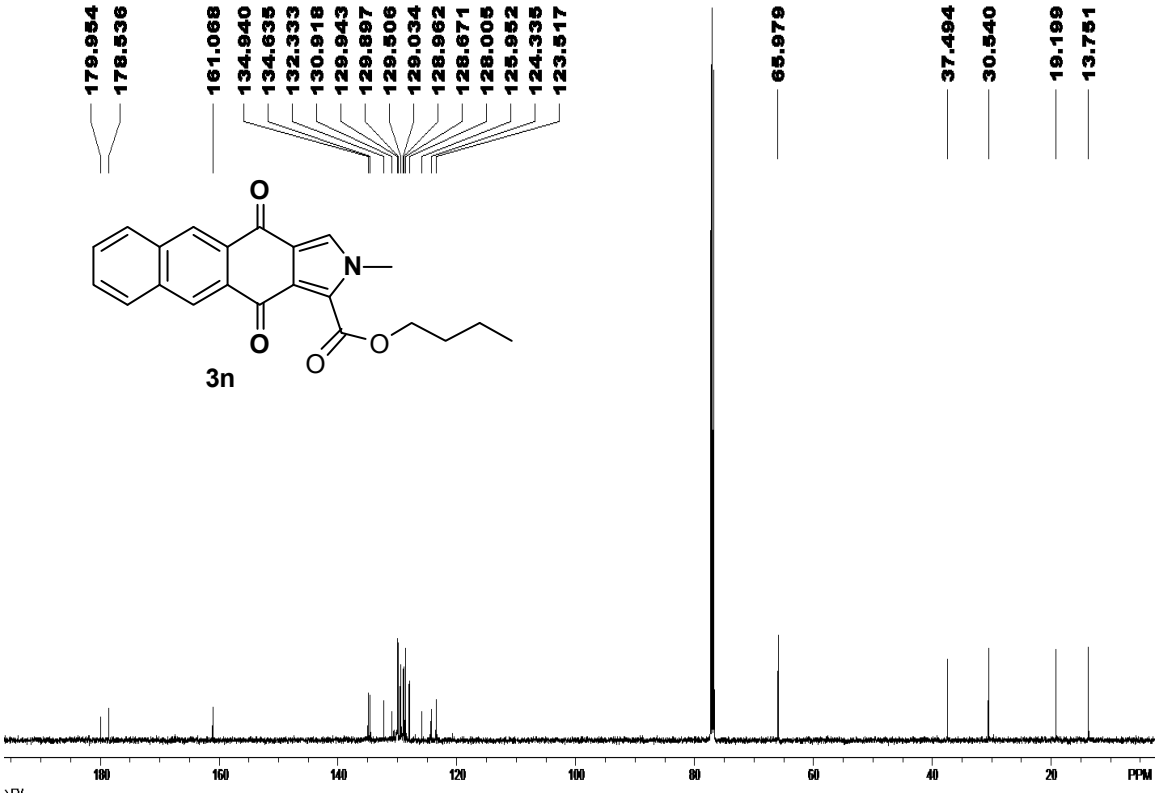

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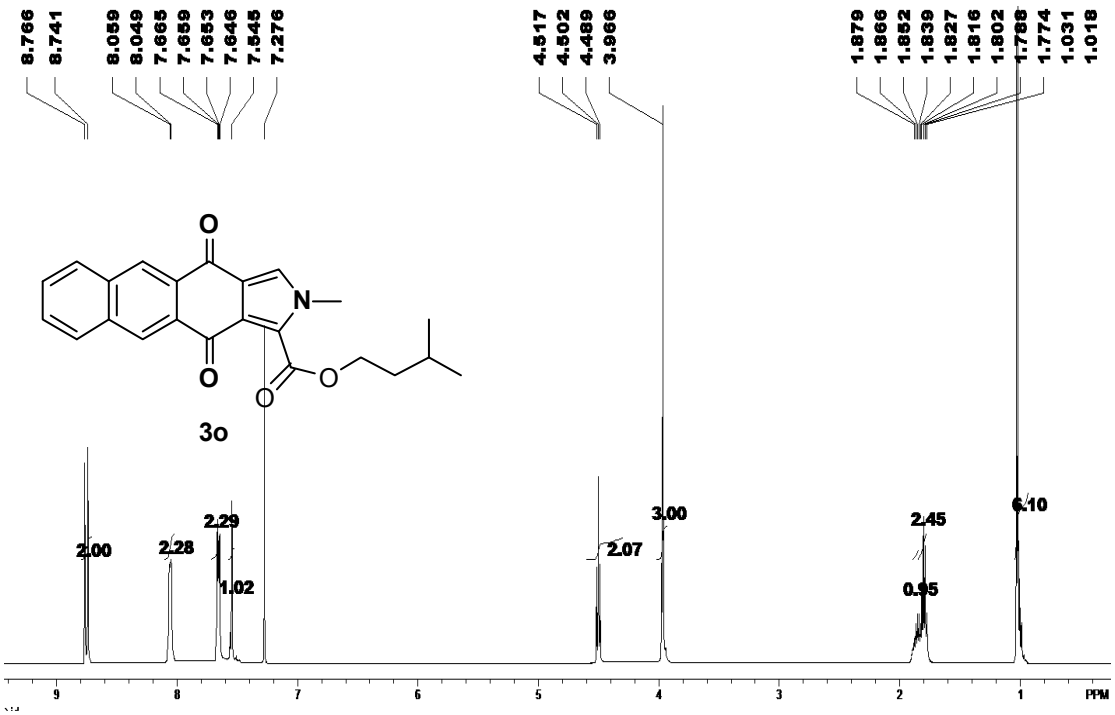

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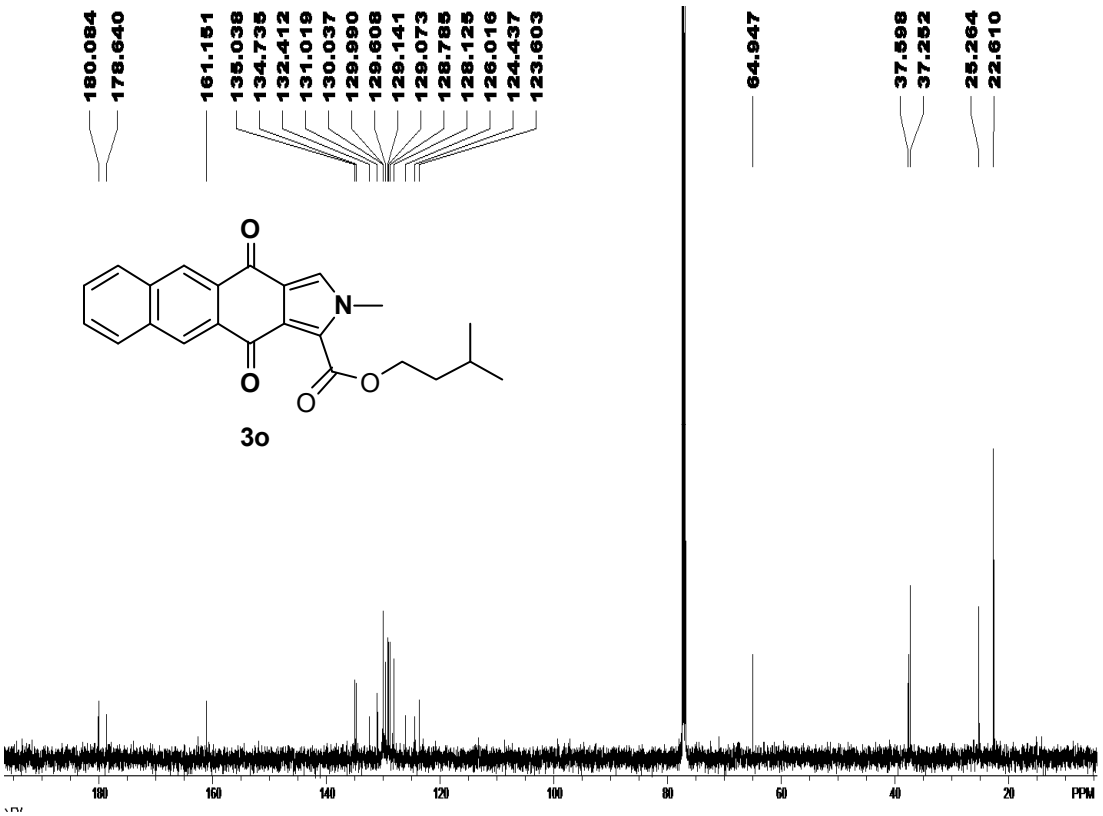

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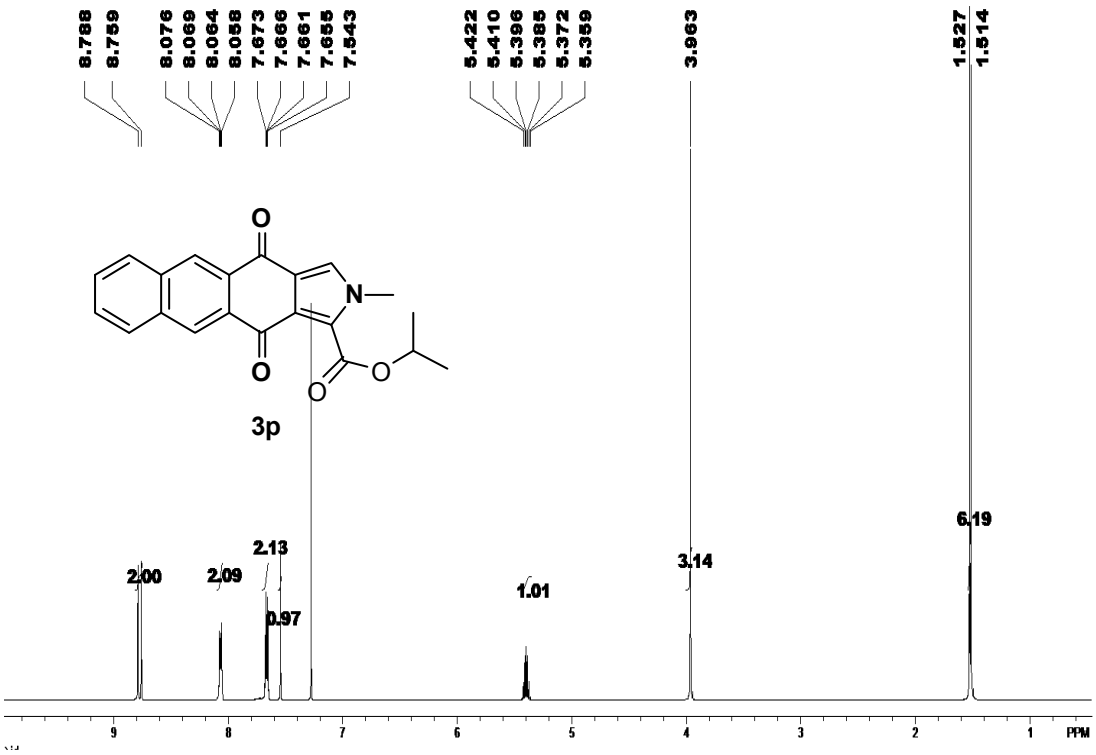

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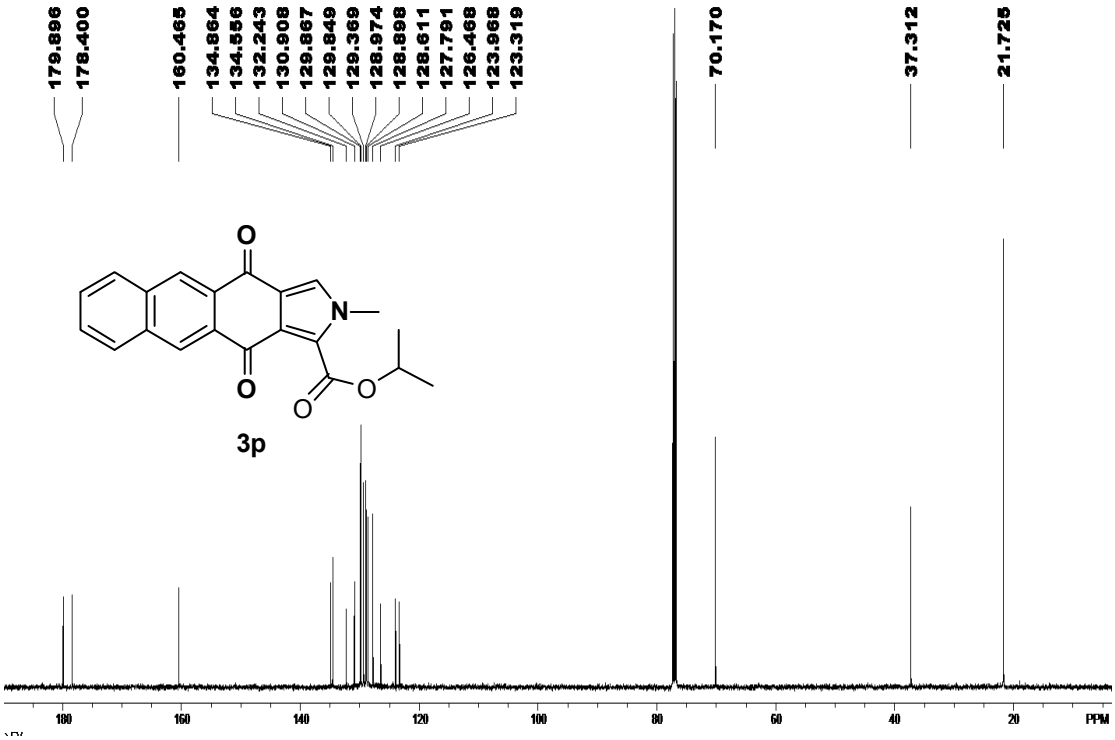

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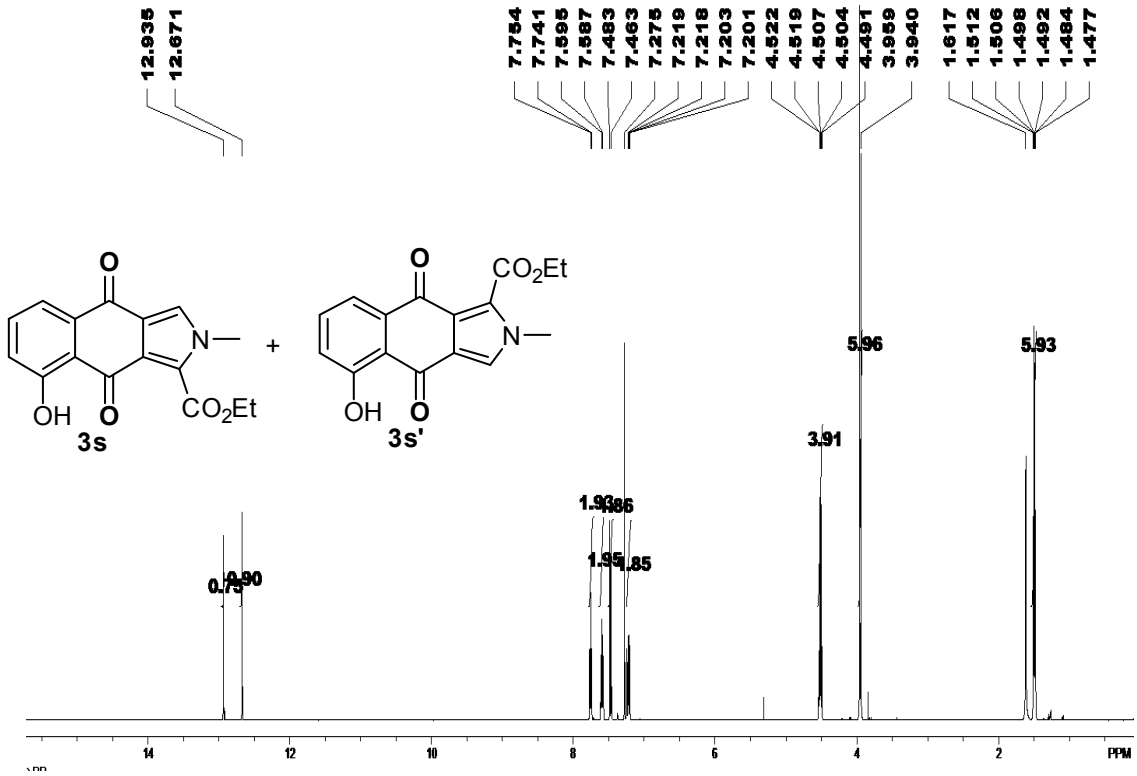

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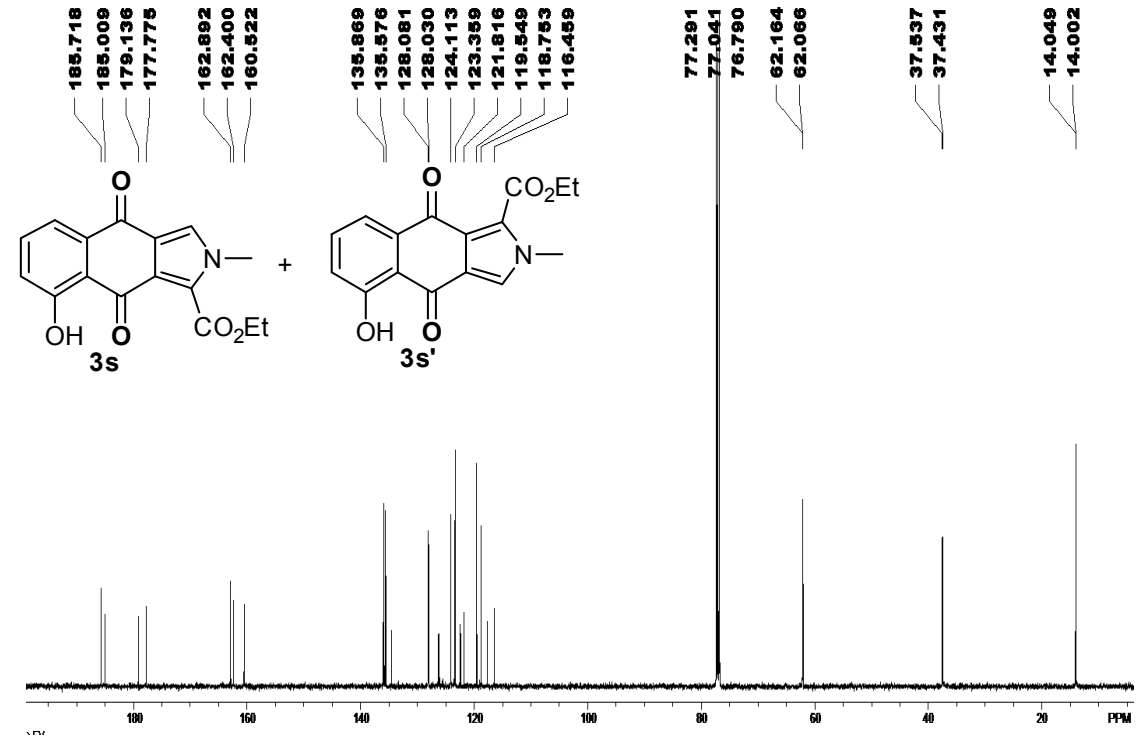

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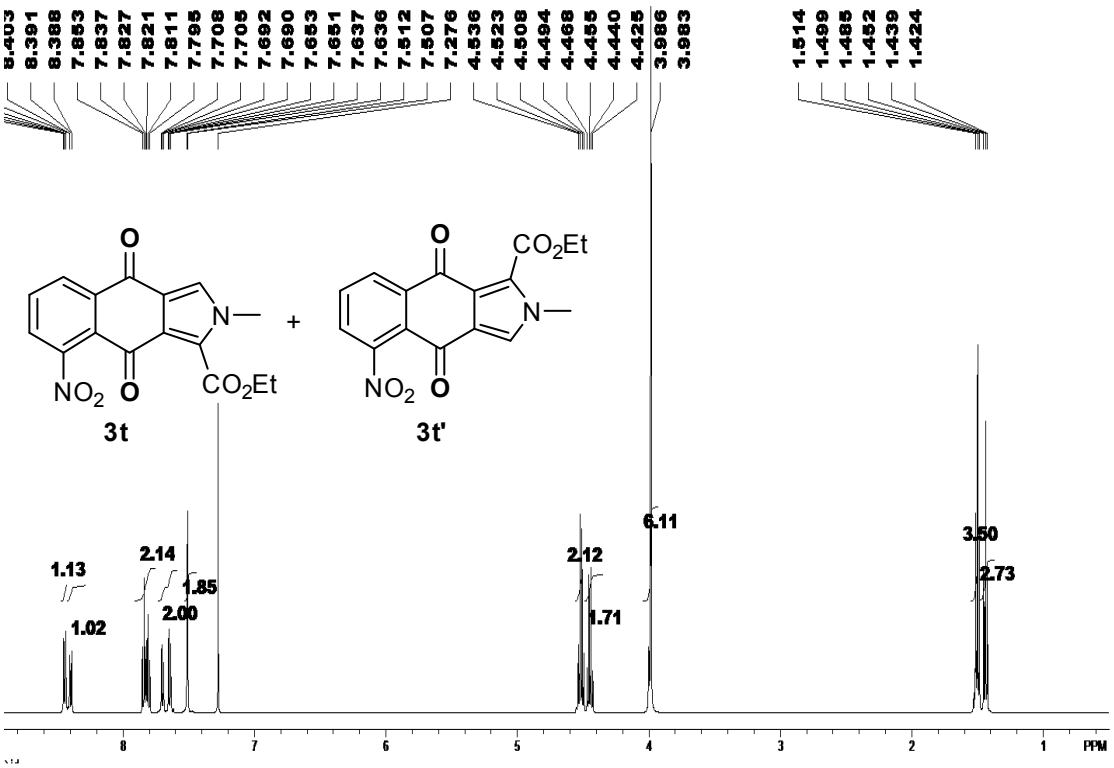

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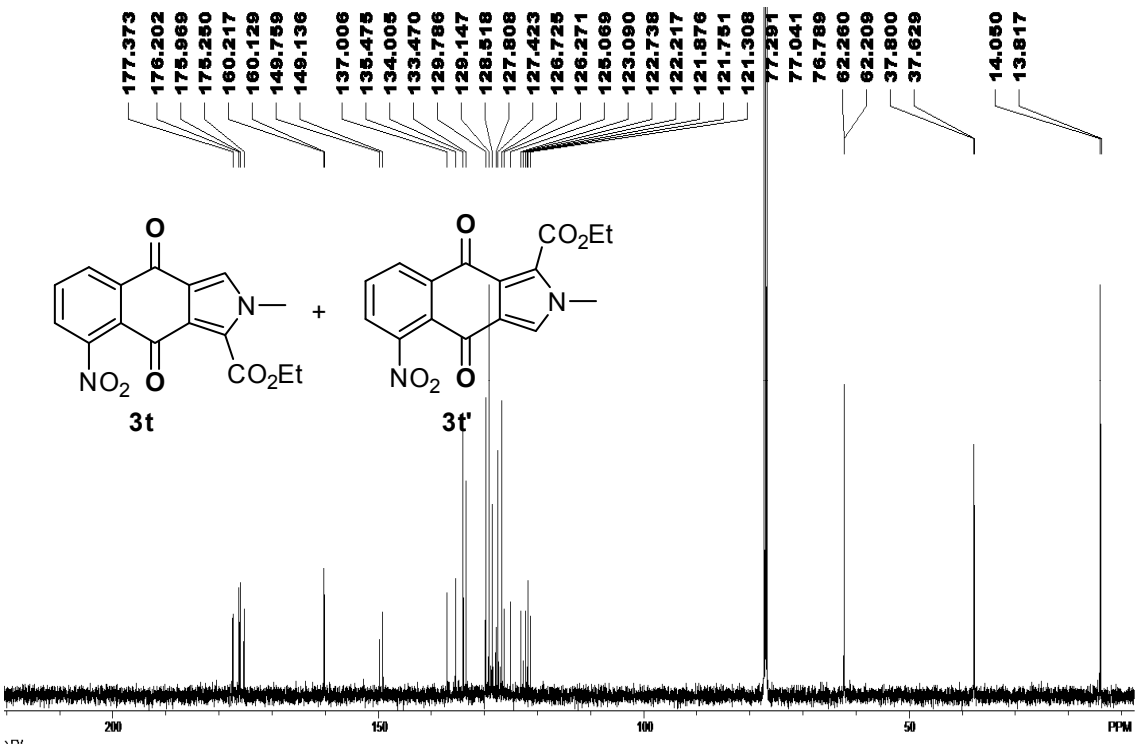

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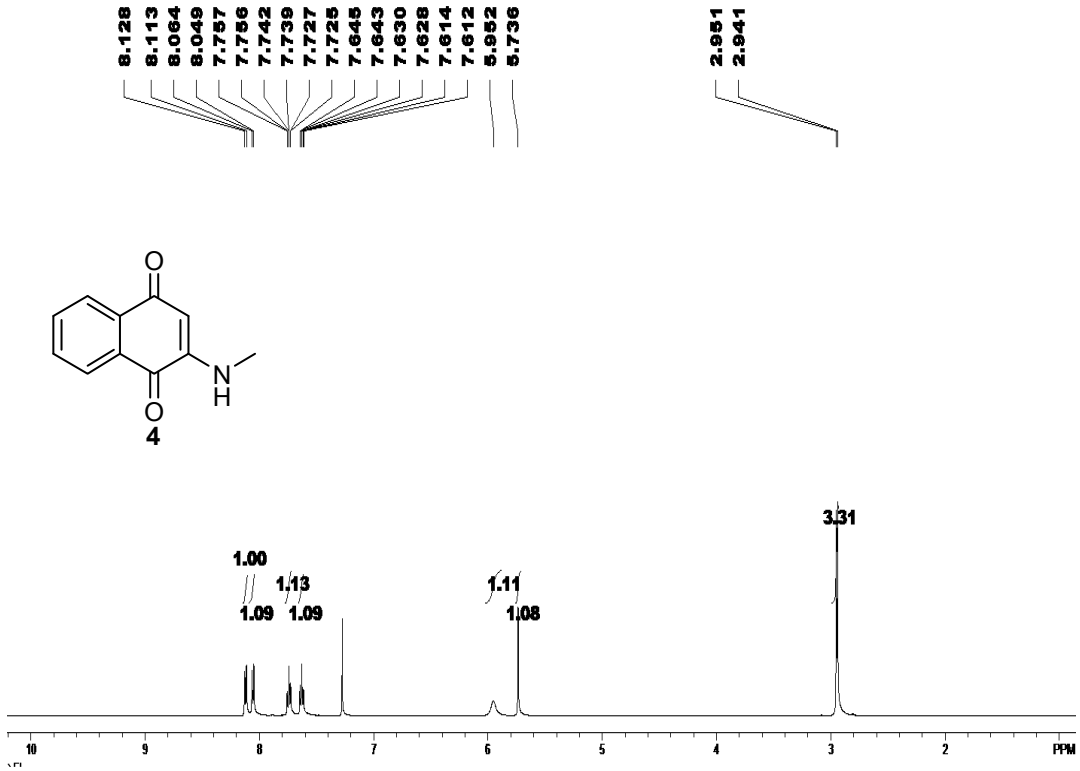

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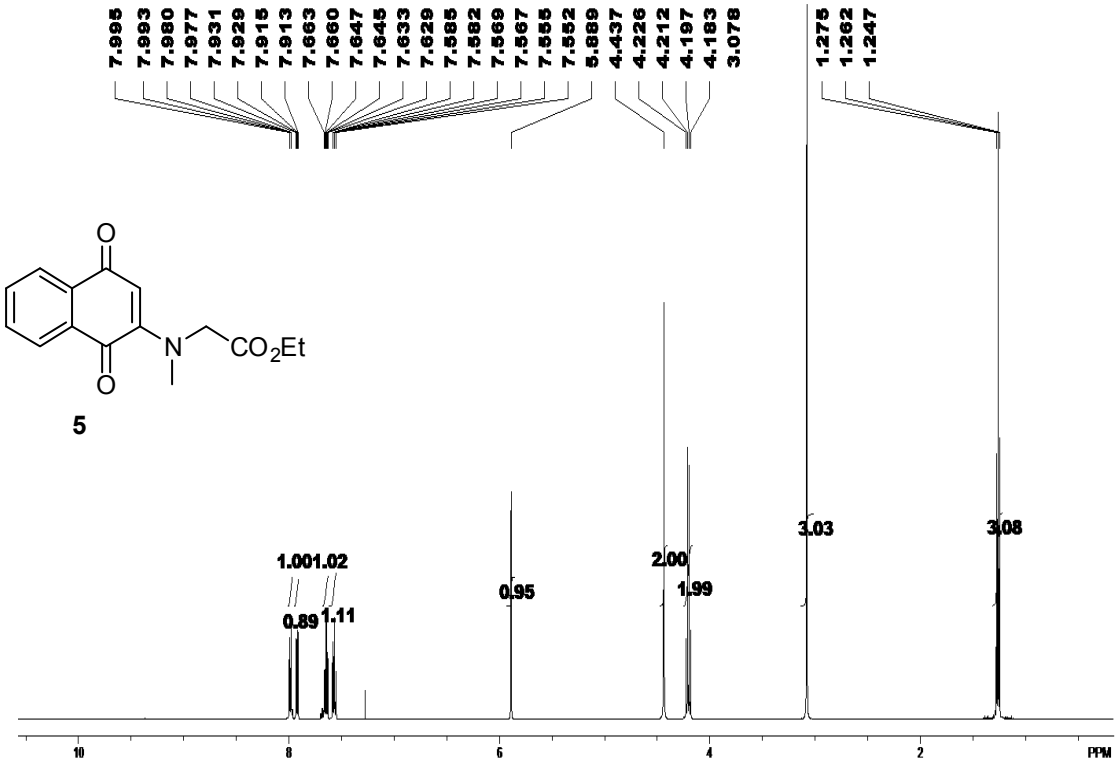

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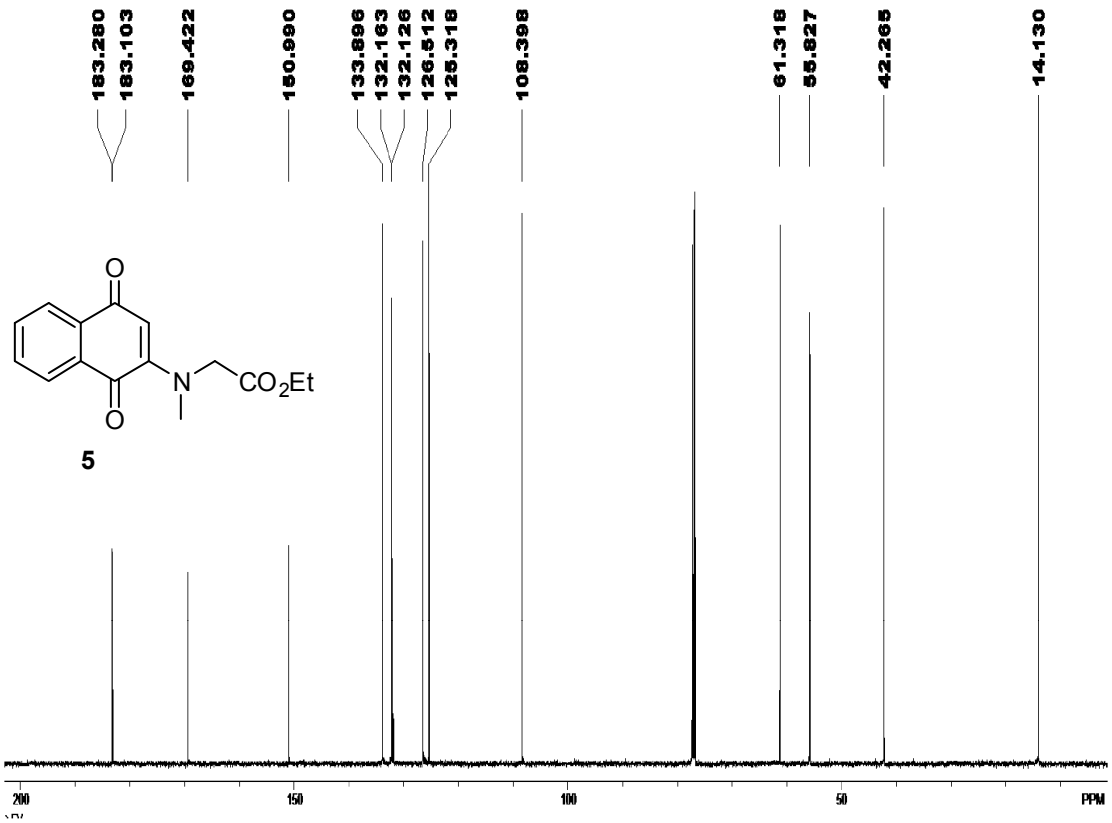
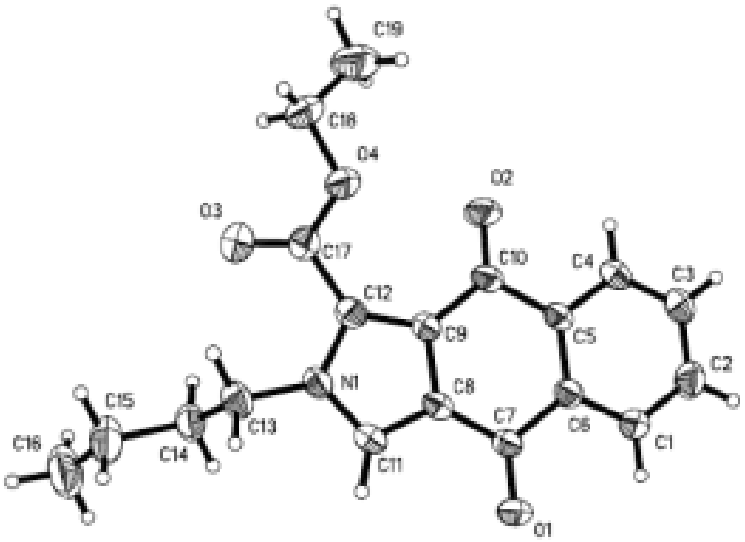


5

3. X ray Crystal Structure of **3d**



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_diffrn_reflns_limit_k_min -5

_diffrn_reflns_limit_k_max 5

_diffrn_reflns_limit_l_min -31

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_diffrn_reflns_limit_l_max          31
_diffrn_reflns_theta_min            2.91
_diffrn_reflns_theta_max            25.00
_reflns_number_total                2916
_reflns_number_gt                   1753
_reflns_threshold_expression        >2sigma(I)

_computing_data_collection          ?
_computing_cell_refinement          ?
_computing_data_reduction           ?
_computing_structure_solution       'SHELXS-97 (Sheldrick, 1990)'
_computing_structure_refinement     'SHELXL-97 (Sheldrick, 1997)'
_computing_molecular_graphics       ?
_computing_publication_material     ?

_refine_special_details
;
 Refinement of F^2^ against ALL reflections.  The weighted R-factor wR and
 goodness of fit S are based on F^2^, conventional R-factors R are based
 on F, with F set to zero for negative F^2^. The threshold expression of
 F^2^ > 2sigma(F^2^) is used only for calculating R-factors(gt) etc. and is
 not relevant to the choice of reflections for refinement.  R-factors based
 on F^2^ are statistically about twice as large as those based on F, and R-
 factors based on ALL data will be even larger.
;
```

```
_refine_ls_structure_factor_coef    Fsqd
_refine_ls_matrix_type              full
_refine_ls_weighting_scheme         calc
_refine_ls_weighting_details
 'calc w=1/[\s^2^(Fo^2^)+(0.0947P)^2^+0.1957P] where P=(Fo^2^+2Fc^2^)/3'
_atom_sites_solution_primary        direct
_atom_sites_solution_secondary      difmap
_atom_sites_solution_hydrogens      geom
_refine_ls_hydrogen_treatment       constr
_refine_ls_extinction_method        none
_refine_ls_extinction_coef          ?
_refine_ls_number_reflns            2916
_refine_ls_number_parameters        219
_refine_ls_number_restraints        0
_refine_ls_R_factor_all             0.1024
_refine_ls_R_factor_gt              0.0580
_refine_ls_wR_factor_ref            0.1851
_refine_ls_wR_factor_gt             0.1556
_refine_ls_goodness_of_fit_ref      1.052
_refine_ls_restrained_S_all         1.052
_refine_ls_shift/su_max             0.000
_refine_ls_shift/su_mean            0.000

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_atom_site_fract_y

_atom_site_fract_z

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_atom_site_symmetry_multiplicity

_atom_site_calc_flag

_atom_site_refinement_flags

_atom_site_disorder_assembly

_atom_site_disorder_group

O1 O 0.56301(14) 0.3727(5) 0.08504(8) 0.0747(7) Uani 1 1 d . . .

O2 O 0.92688(16) 0.8530(6) 0.15289(9) 0.0993(9) Uani 1 1 d . . .

O3 O 0.8480(3) 1.2726(10) -0.00848(12) 0.1586(17) Uani 1 1 d . . .

O4 O 0.92435(18) 1.2229(7) 0.07486(10) 0.1093(10) Uani 1 1 d . . .

N1 N 0.68171(15) 0.9815(5) 0.00345(8) 0.0500(6) Uani 1 1 d . . .

C1 C 0.6914(2) 0.2291(7) 0.18658(12) 0.0611(8) Uani 1 1 d . . .

H1 H 0.6297 0.1413 0.1752 0.073 Uiso 1 1 calc R . .

C2 C 0.7570(2) 0.1672(8) 0.23648(13) 0.0728(9) Uani 1 1 d . . .

H2 H 0.7392 0.0405 0.2588 0.087 Uiso 1 1 calc R . .

C3 C 0.8490(2) 0.2948(7) 0.25289(12) 0.0698(9) Uani 1 1 d . . .

H3 H 0.8934 0.2533 0.2863 0.084 Uiso 1 1 calc R . .

C4 C 0.8751(2) 0.4822(7) 0.22017(11) 0.0608(8) Uani 1 1 d . . .

H4 H 0.9375 0.5664 0.2316 0.073 Uiso 1 1 calc R . .

C5 C 0.81054(18) 0.5490(6) 0.17031(10) 0.0480(7) Uani 1 1 d . . .

C6 C 0.71703(18) 0.4200(6) 0.15359(10) 0.0483(7) Uani 1 1 d . . .

C7 C 0.64465(18) 0.4856(6) 0.10051(10) 0.0495(7) Uani 1 1 d . . .

C8 C 0.67691(17) 0.6919(6) 0.06862(10) 0.0467(6) Uani 1 1 d . . .

C9 C 0.77142(17) 0.8219(6) 0.08407(10) 0.0458(6) Uani 1 1 d . . .

C10 C 0.84445(19) 0.7534(6) 0.13624(11) 0.0548(7) Uani 1 1 d . . .

C11 C 0.6245(2) 0.7963(6) 0.01907(11) 0.0518(7) Uani 1 1 d . . .

H11 H 0.5600 0.7467 -0.0005 0.062 Uiso 1 1 calc R . .

C12 C 0.77324(18) 1.0011(6) 0.04248(10) 0.0508(7) Uani 1 1 d . . .

C13 C 0.6450(2) 1.1341(7) -0.04772(11) 0.0613(8) Uani 1 1 d . . .

H13A H 0.6821 1.3121 -0.0454 0.074 Uiso 1 1 calc R . .

H13B H 0.5764 1.1858 -0.0546 0.074 Uiso 1 1 calc R . .

C14 C 0.6531(2) 0.9594(7) -0.09288(11) 0.0680(9) Uani 1 1 d . . .

H14A H 0.6161 0.7811 -0.0954 0.082 Uiso 1 1 calc R . .

H14B H 0.7217 0.9089 -0.0863 0.082 Uiso 1 1 calc R . .

C15 C 0.6141(3) 1.1218(8) -0.14536(13) 0.0964(13) Uani 1 1 d . . .

H15A H 0.5495 1.1990 -0.1487 0.116 Uiso 1 1 calc R . .

H15B H 0.6574 1.2848 -0.1442 0.116 Uiso 1 1 calc R . .

C16 C 0.6059(4) 0.9524(11) -0.19215(15) 0.1326(18) Uani 1 1 d . . .

H16A H 0.6697 0.8786 -0.1899 0.199 Uiso 1 1 calc R . .

H16B H 0.5812 1.0723 -0.2231 0.199 Uiso 1 1 calc R . .

H16C H 0.5614 0.7938 -0.1946 0.199 Uiso 1 1 calc R . .

C17 C 0.8513(2) 1.1778(7) 0.03321(13) 0.0635(8) Uani 1 1 d . . .

C18 C 1.0106(3) 1.3772(11) 0.07086(17) 0.1206(17) Uani 1 1 d . . .

H18A H 1.0242 1.3165 0.0392 0.145 Uiso 1 1 calc R . .

H18B H 0.9981 1.5840 0.0685 0.145 Uiso 1 1 calc R . .

C19 C 1.0927(3) 1.3142(11) 0.11652(19) 0.1202(16) Uani 1 1 d . . .

H19A H 1.0747 1.3436 0.1477 0.180 Uiso 1 1 calc R . .

H19B H 1.1465 1.4399 0.1176 0.180 Uiso 1 1 calc R . .

H19C H 1.1123 1.1164 0.1152 0.180 Uiso 1 1 calc R . .

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_atom_site_aniso_U_23

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O1 0.0466(11) 0.0990(17) 0.0659(13) 0.0138(12) 0.0016(10) -0.0224(11)

O2 0.0570(13) 0.137(2) 0.0770(16) 0.0399(15) -0.0136(11) -0.0394(14)

O3 0.139(3) 0.244(4) 0.075(2) 0.045(2) 0.0114(17) -0.100(3)

O4 0.0731(15) 0.156(3) 0.0783(17) 0.0388(16) -0.0021(13) -0.0543(16)

N1 0.0521(13) 0.0510(13) 0.0416(12) 0.0026(11) 0.0082(10) 0.0020(11)

C1 0.0520(16) 0.075(2) 0.0540(17) 0.0065(15) 0.0140(14) -0.0060(15)

C2 0.075(2) 0.085(2) 0.060(2) 0.0194(17) 0.0241(17) 0.0028(18)

C3 0.066(2) 0.085(2) 0.0479(17) 0.0137(16) 0.0056(15) 0.0036(17)

C4 0.0500(16) 0.075(2) 0.0465(16) 0.0032(16) 0.0017(13) -0.0006(15)

C5 0.0435(14) 0.0539(16) 0.0414(14) -0.0015(13) 0.0070(11) 0.0016(12)

C6 0.0442(14) 0.0529(16) 0.0454(15) -0.0014(13) 0.0115(11) 0.0006(12)

C7 0.0411(14) 0.0567(16) 0.0451(15) -0.0020(13) 0.0065(12) -0.0021(13)

C8 0.0404(14) 0.0510(15) 0.0434(15) -0.0029(12) 0.0064(11) 0.0032(12)

C9 0.0430(14) 0.0483(15) 0.0416(15) -0.0015(12) 0.0082(11) -0.0008(12)

C10 0.0417(15) 0.0625(18) 0.0513(16) 0.0022(14) 0.0035(13) -0.0051(13)

C11 0.0470(15) 0.0556(16) 0.0450(15) -0.0036(13) 0.0047(12) 0.0010(13)

C12 0.0522(15) 0.0491(15) 0.0480(16) -0.0044(13) 0.0121(12) 0.0002(13)

C13 0.0704(19) 0.0558(18) 0.0502(17) 0.0089(14) 0.0095(14) 0.0086(14)

C14 0.088(2) 0.065(2) 0.0479(18) 0.0061(15) 0.0176(15) -0.0056(17)

C15 0.153(4) 0.075(2) 0.061(2) 0.0076(19) 0.034(2) -0.016(2)

C16 0.184(5) 0.137(4) 0.062(3) 0.017(3) 0.021(3) -0.015(4)

C17 0.070(2) 0.0615(19) 0.0561(19) 0.0053(16) 0.0173(16) -0.0101(16)

C18 0.085(3) 0.158(4) 0.106(3) 0.032(3) 0.014(2) -0.062(3)

C19 0.070(3) 0.143(4) 0.137(4) 0.000(3) 0.019(3) -0.030(3)

_geom_special_details

;

All esds (except the esd in the dihedral angle between two l.s. planes)

are estimated using the full covariance matrix. The cell esds are taken

into account individually in the estimation of esds in distances, angles

and torsion angles; correlations between esds in cell parameters are only

used when they are defined by crystal symmetry. An approximate (isotropic)

treatment of cell esds is used for estimating esds involving l.s. planes.

;

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O1 C7 1.223(3) . ?

O2 C10 1.209(3) . ?

O3 C17 1.183(4) . ?

O4 C17 1.269(4) . ?

O4 C18 1.463(4) . ?

N1 C11 1.344(3) . ?

N1 C12 1.385(3) . ?

N1 C13 1.473(3) . ?

C1 C6 1.380(4) . ?

C1 C2 1.385(4) . ?

C1 H1 0.9300 . ?

C2 C3 1.379(4) . ?

C2 H2 0.9300 . ?

C3 C4 1.368(4) . ?

C3 H3 0.9300 . ?

C4 C5 1.384(3) . ?

C4 H4 0.9300 . ?

C5 C6 1.400(4) . ?

C5 C10 1.500(4) . ?

C6 C7 1.487(3) . ?

C7 C8 1.452(4) . ?

C8 C11 1.376(3) . ?

C8 C9 1.415(3) . ?

C9 C12 1.393(4) . ?

C9 C10 1.476(3) . ?

C11 H11 0.9300 . ?

C12 C17 1.472(4) . ?

C13 C14 1.488(4) . ?

C13 H13A 0.9700 . ?

C13 H13B 0.9700 . ?

C14 C15 1.524(4) . ?

C14 H14A 0.9700 . ?

C14 H14B 0.9700 . ?

C15 C16 1.447(6) . ?

C15 H15A 0.9700 . ?

C15 H15B 0.9700 . ?

C16 H16A 0.9600 . ?

C16 H16B 0.9600 . ?

C16 H16C 0.9600 . ?

C18 C19 1.419(5) . ?

C18 H18A 0.9700 . ?

C18 H18B 0.9700 . ?

C19 H19A 0.9600 . ?

C19 H19B 0.9600 . ?

C19 H19C 0.9600 . ?

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C17 O4 C18 119.3(3) . . ?

C11 N1 C12 109.4(2) . . ?

C11 N1 C13 121.7(2) . . ?

C12 N1 C13 128.9(2) . . ?

C6 C1 C2 120.5(3) . . ?

C6 C1 H1 119.8 . . ?

C2 C1 H1 119.8 . . ?

C3 C2 C1 119.6(3) . . ?

C3 C2 H2 120.2 . . ?

C1 C2 H2 120.2 . . ?

C4 C3 C2 120.2(3) . . ?

C4 C3 H3 119.9 . . ?

C2 C3 H3 119.9 . . ?

C3 C4 C5 121.3(3) . . ?

C3 C4 H4 119.4 . . ?

C5 C4 H4 119.4 . . ?

C4 C5 C6 118.6(3) . . ?

C4 C5 C10 118.5(2) . . ?

C6 C5 C10 122.9(2) . . ?

C1 C6 C5 119.8(2) . . ?

C1 C6 C7 119.4(2) . . ?

C5 C6 C7 120.8(2) . . ?

O1 C7 C8 122.5(2) . . ?

O1 C7 C6 121.7(2) . . ?

C8 C7 C6 115.8(2) . . ?

C11 C8 C9 107.4(2) . . ?

C11 C8 C7 128.0(2) . . ?

C9 C8 C7 124.6(2) . . ?

C12 C9 C8 106.7(2) . . ?

C12 C9 C10 133.4(2) . . ?

C8 C9 C10 119.9(2) . . ?

O2 C10 C9 124.7(3) . . ?

O2 C10 C5 119.4(2) . . ?

C9 C10 C5 115.9(2) . . ?

N1 C11 C8 109.0(2) . . ?

N1 C11 H11 125.5 . . ?

C8 C11 H11 125.5 . . ?

N1 C12 C9 107.4(2) . . ?

N1 C12 C17 119.7(2) . . ?

C9 C12 C17 132.8(2) . . ?

N1 C13 C14 113.2(2) . . ?

N1 C13 H13A 108.9 . . ?

C14 C13 H13A 108.9 . . ?

N1 C13 H13B 108.9 . . ?

C14 C13 H13B 108.9 . . ?

H13A C13 H13B 107.8 . . ?

C13 C14 C15 112.1(3) . . ?

C13 C14 H14A 109.2 . . ?

C15 C14 H14A 109.2 . . ?

C13 C14 H14B 109.2 . . ?

C15 C14 H14B 109.2 . . ?

H14A C14 H14B 107.9 . . ?

C16 C15 C14 115.4(3) . . ?

C16 C15 H15A 108.4 . . ?

C14 C15 H15A 108.4 . . ?

C16 C15 H15B 108.4 . . ?

C14 C15 H15B 108.4 . . ?

H15A C15 H15B 107.5 . . ?

C15 C16 H16A 109.5 . . ?

C15 C16 H16B 109.5 . . ?

H16A C16 H16B 109.5 . . ?

C15 C16 H16C 109.5 . . ?

H16A C16 H16C 109.5 . . ?

H16B C16 H16C 109.5 . . ?

O3 C17 O4 122.0(3) . . ?

O3 C17 C12 124.5(3) . . ?

O4 C17 C12 113.4(3) . . ?

C19 C18 O4 108.7(3) . . ?

C19 C18 H18A 110.0 . . ?

O4 C18 H18A 110.0 . . ?

C19 C18 H18B 110.0 . . ?

O4 C18 H18B 110.0 . . ?

H18A C18 H18B 108.3 . . ?

C18 C19 H19A 109.5 . . ?

C18 C19 H19B 109.5 . . ?

H19A C19 H19B 109.5 . . ?

C18 C19 H19C 109.5 . . ?

H19A C19 H19C 109.5 . . ?

H19B C19 H19C 109.5 . . ?

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C6 C1 C2 C3 -0.9(5) . . . . ?

C1 C2 C3 C4 0.3(5) . . . . ?

C2 C3 C4 C5 0.2(5) . . . . ?

C3 C4 C5 C6 -0.1(4) . . . . ?

C3 C4 C5 C10 -179.2(3) . . . . ?

C2 C1 C6 C5 1.0(4) . . . . ?

C2 C1 C6 C7 -179.2(3) . . . . ?

C4 C5 C6 C1 -0.5(4) . . . . ?

C10 C5 C6 C1 178.6(3) . . . . ?

C4 C5 C6 C7 179.7(2) . . . . ?

C10 C5 C6 C7 -1.3(4) . . . . ?

C1 C6 C7 O1 -0.6(4) . . . . ?

C5 C6 C7 O1 179.3(3) . . . . ?

C1 C6 C7 C8 179.1(2) . . . . ?

C5 C6 C7 C8 -1.0(4) . . . . ?

O1 C7 C8 C11 1.5(4) . . . . ?

C6 C7 C8 C11 -178.2(3) . . . . ?

O1 C7 C8 C9 -178.1(3) . . . . ?

C6 C7 C8 C9 2.2(4) . . . . ?

C11 C8 C9 C12 -0.6(3) . . . . ?

C7 C8 C9 C12 179.1(2) . . . . ?

C11 C8 C9 C10 179.3(2) . . . . ?

C7 C8 C9 C10 -1.0(4) . . . . ?

C12 C9 C10 O2 -0.2(5) . . . . ?

C8 C9 C10 O2 179.9(3) . . . . ?

C12 C9 C10 C5 178.6(3) . . . . ?

C8 C9 C10 C5 -1.3(4) . . . . ?

C4 C5 C10 O2 0.4(4) . . . . ?

C6 C5 C10 O2 -178.7(3) . . . . ?

C4 C5 C10 C9 -178.5(3) . . . . ?

C6 C5 C10 C9 2.4(4) . . . . ?

C12 N1 C11 C8 0.3(3) . . . . ?

C13 N1 C11 C8 -178.6(2) . . . . ?

C9 C8 C11 N1 0.2(3) . . . . ?

C7 C8 C11 N1 -179.5(2) . . . . ?

C11 N1 C12 C9 -0.7(3) . . . . ?

C13 N1 C12 C9 178.1(2) . . . . ?

C11 N1 C12 C17 176.2(2) . . . . ?

C13 N1 C12 C17 -5.0(4) . . . . ?

C8 C9 C12 N1 0.7(3) . . . . ?

C10 C9 C12 N1 -179.2(3) . . . . ?

C8 C9 C12 C17 -175.6(3) . . . . ?

C10 C9 C12 C17 4.5(5) . . . . ?

C11 N1 C13 C14 -84.1(3) . . . . ?

C12 N1 C13 C14 97.3(3) . . . . ?

N1 C13 C14 C15 179.7(3) . . . . ?

C13 C14 C15 C16 -171.3(4) . . . . ?

C18 O4 C17 O3 -4.9(6) . . . . ?

C18 O4 C17 C12 175.5(4) . . . . ?

N1 C12 C17 O3 -12.2(5) . . . . ?

C9 C12 C17 O3 163.8(4) . . . . ?

N1 C12 C17 O4 167.4(3) . . . . ?

C9 C12 C17 O4 -16.6(5) . . . . ?

C17 O4 C18 C19 -159.8(4) . . . . ?

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