

Supporting Information

5,8-Di-*tert*-butyl-2-hydroxy-1H-benzo[de]isoquinoline-1,3(2H)-dione – a new lipophilic N-oxyl radical precursor

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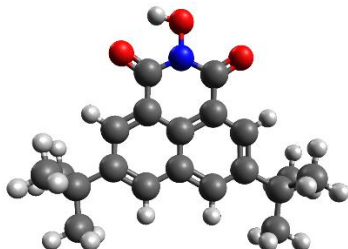
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Optimized geometries of 5,8-Di-*tert*-butyl-N-hydroxynaphthalimide 4, NHNI 6, NHPI and corresponding N-oxyl radicals used for calculation of NO–H BDE values

5,8-Di-*tert*-butyl-N-hydroxynaphthalimide 4



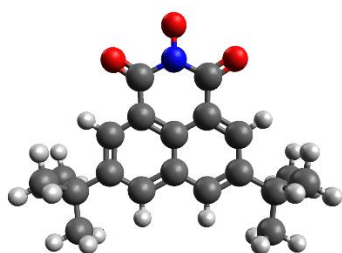
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Total Enthalpy ... -1102.11455307 Eh

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C	2.74785645793316	1.24934702328175	-0.00078610596575
C	2.12696047767468	0.04595277724106	-0.00020857359291
C	0.78645512160895	0.01302967973193	0.00086818734300
C	0.10256689618163	1.17580156868300	0.00132171072664
C	0.73428068330728	2.31890816409070	0.00075255757638
H	3.82537082004315	-1.07964031637251	-0.00161607789480
H	3.76759730985735	1.25301260804135	-0.00163499428066
C	2.80666610587212	-1.12499539244905	-0.00072968528388
C	0.15449194578739	-1.17886047875565	0.00142218775776
H	0.17730182680597	3.17330948723097	0.00110656052209
C	0.84412014242484	-2.28757895283768	0.00094598635072
C	2.19687499281234	-2.28326189235852	-0.00016292698565
H	0.32814151561731	-3.16732625243534	0.00140473145567
C	-1.25712980505608	-1.23755216493426	0.00250084007724
C	-1.29959636568279	1.14447523115159	0.00240131768480
N	-1.85146811682003	-0.04974580203600	0.00284646699120
O	-1.99288561890066	2.07982296241633	0.00281678234723
O	-1.86874568374767	-2.21309483387429	0.00309810426415
O	-3.15511266323654	-0.06234511271622	0.00382679583562
H	-3.36519215958182	0.83174525607857	0.00398223039531
C	2.89897101389466	-3.55624400424759	-0.00057441273181
C	2.51917607514047	-4.31337544351814	-1.18975322717815
H	3.00152541133906	-5.22038332441727	-1.19952493538328
H	1.50949266053772	-4.49527218574056	-1.21785668157842

H	2.77418614471648	-3.80579740350621	-2.04561323999082
C	4.34277572468109	-3.38525897065309	-0.00355186019917
H	4.66322358134319	-2.87865669246062	-0.83773775026743
H	4.66662770336571	-2.87760547101237	0.82867178267940
H	4.79778308502573	-4.30556422301541	-0.00380530972839
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H	3.00315923176845	-5.21999251718465	1.19892667783213
H	2.78540353013970	-3.80401355567587	2.04523385497223
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H	1.29514008185109	4.55522994266177	-1.21930814398985
H	2.75102428346849	5.35023664072372	-1.20021423317839
H	2.59169316817376	3.92623090475108	-2.04596794272172
C	2.31609220145340	4.42032353391764	1.19064043952303
H	2.75281267996409	5.35016696946609	1.19867812351794
H	1.29872940575155	4.55192528347826	1.22403483594809
H	2.60031143998007	3.92563356018541	2.04482233254831
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H	4.52111573527124	3.08872871737889	-0.83709617312025
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5,8-Di-*tert*-butylnaphthalimide-N-oxyl radical **5**



of imaginary frequencies = 0

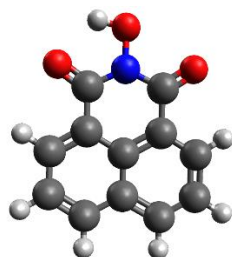
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C	2.12402255318230	0.04455300840341	-0.00034381632823
C	0.78181684269063	0.01222035525373	0.00068714616896
C	0.09982678122752	1.17659175316781	0.00121069180261
C	0.73909147461174	2.31702530598100	0.00072229336492

H	3.82420930476356	-1.07566960067240	-0.00171458870225
H	3.76839508518862	1.24531834003062	-0.00173996928424
C	2.80576000386131	-1.12474820483033	-0.00082460466941
C	0.15661436540249	-1.18361119649886	0.00119363603747
H	0.18396776212172	3.17246120339120	0.00119370274069
C	0.84998928725781	-2.29200775479456	0.00079819888969
C	2.20106716816911	-2.28586882091090	-0.00024817923012
H	0.33659775509890	-3.17312533960378	0.00128062048762
C	-1.24779428986807	-1.25501980911806	0.00205324631493
C	-1.30632526444913	1.18058619196557	0.00239147467542
N	-1.90483416734729	-0.05227068103523	0.00263996688981
O	-1.95626628335815	2.12397703359441	0.00268554184139
O	-1.85185390447216	-2.22839190816302	0.00270438299886
O	-3.11182466803010	-0.08125764906806	0.00345310016694
C	2.90721494262267	-3.55660101417008	-0.00044661582916
C	2.52920786182262	-4.31535139656869	-1.18914955757830
H	3.01512747912426	-5.22038144286438	-1.19903061313424
H	1.52028533598957	-4.50187472700707	-1.21613252514363
H	2.78122439306593	-3.80735081685934	-2.04562611265282
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H	4.66900678569291	-2.87335444239862	-0.83865339972881
H	4.67309424457223	-2.87154121478751	0.82782660209801
H	4.80860164802050	-4.29911699499352	-0.00404563392414
C	2.53461477839793	-4.31236045653161	1.19182947854329
H	3.01645112758859	-5.21960511820324	1.19930279279401
H	2.79524836279752	-3.80443795188832	2.04577705512476
H	1.52503050720613	-4.49401952148049	1.22640113857437
C	2.73275504796215	3.67923940302146	-0.00068864180984
C	2.31899026267088	4.41834951817469	-1.18986293390779
H	1.30212384906410	4.55486458404206	-1.21783237059490
H	2.75975184622987	5.34621209296178	-1.19943430911962
H	2.59662740794206	3.92328338041457	-2.04599475978322
C	2.32347534499629	4.41651473257990	1.19116023508618
H	2.76146445839462	5.34571453167858	1.19887311398528
H	1.30634877055719	4.54977927491841	1.22485145058152
H	2.60739789393356	3.92175454280952	2.04541056824288
C	4.18276970429814	3.57304524097179	-0.00359261191016
H	4.52939177360468	3.08069332386569	0.82865765649422

H	4.52602293528574	3.08164972868586	-0.83780807828577
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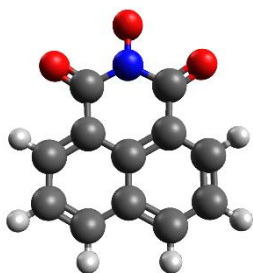
N-hydroxynaphthalimide (NHNI) **6**



of imaginary frequencies = 0

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C	-2.66295146713983	-1.24600378380903	0.00003456918638
C	-2.04454375043187	-0.04376618231837	0.00001838488691
C	-0.69790061664642	-0.01112032493525	-0.00005872360607
C	-0.00757665569635	-1.17227712151369	-0.00002872931065
C	-0.63477284906014	-2.32049875210877	-0.00000369189860
H	-3.74189489072925	1.10397751185715	0.00016158027997
H	-2.45716635720429	-3.25679090331341	0.00004444461928
H	-3.68394174177090	-1.27167045032240	0.00007244959962
C	-2.72088019809950	1.12665983453014	0.00008500063675
C	-0.05864403185450	1.17875897737772	-0.00014247040157
H	-0.08609205229856	-3.18055501953134	0.00003388364858
C	-0.74322312100470	2.29346115314246	-0.00004933545008
C	-2.08659998131145	2.26691546239455	0.00006182124995
H	-0.23533304096879	3.17820067848662	-0.00012952904381
H	-2.60699145658925	3.14415990319759	0.00010447909001
C	1.35327772629037	1.23672692985556	-0.00048486114892
C	1.39487045728944	-1.14122112029636	0.00011229680297
N	1.94884218515542	0.05090027520243	-0.00008894859588
O	2.08456013625666	-2.07863131123654	0.00003426877498
O	1.96175898709693	2.21377626055290	0.00003894195725
O	3.25174498416945	0.06366417658641	0.00012748959280
H	3.46336021227294	-0.83003038728755	0.00046414776461

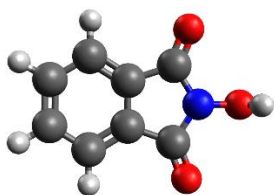
Naphthalimide-N-oxyl radical 7



of imaginary frequencies = 0

Total Enthalpy	...	-770.92899426 Eh	
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C	2.01339664798491	0.00002164017471	0.0000638885606
C	0.66477767685392	0.00000593669781	0.00003189152792
C	0.00384064188795	1.17848121388982	-0.00008727265579
C	0.66540475172570	2.30879639857807	0.00004626190965
H	3.68505159707157	-1.18231010598346	-0.00004398305032
H	2.50937324915302	3.20015146961055	0.00025342787250
H	3.68502405973457	1.18239222818570	0.00024875899952
C	2.66382529534879	-1.18453313824786	-0.00002223653455
C	0.00386778358357	-1.17848449093910	0.00011466018270
H	0.13902752697714	3.18248849314029	-0.00007798717379
C	0.66545827112180	-2.30878425287485	0.00000302156819
C	2.00750676062866	-2.31233213247524	-0.00007787870084
H	0.13910129504926	-3.18248861623357	0.00009725319353
H	2.50944761113515	-3.20009649365832	-0.00011595976323
C	-1.40241200806306	-1.21543491763909	0.00065953754760
C	-1.40243969253037	1.21539954063789	-0.00069405156184
N	-2.03171964513329	-0.00002505770714	-0.00005407123250
O	-2.02590172597555	2.17607771225270	0.00006886127859
O	-2.02585128447288	-2.17612789508785	-0.00024246040795
O	-3.23919272090716	-0.00003854060201	-0.00011835733339

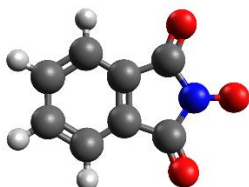
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of imaginary frequencies = 0

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C	1.85359240141242	2.17216842933538 0.01432536288944
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C	2.10326993785393	-0.12724612830112 -0.01144602726811
C	0.79118584924337	-0.22247259696655 -0.01672397204387
C	0.01813456499833	0.85538491344674 -0.00813746551209
C	0.52900722257321	2.06758606816420 0.00583406024455
H	-0.07308957216605	2.89092818993735 0.01189070566987
H	2.28573929380166	3.09650577285031 0.02803044746224
H	3.64124141131395	1.20667937437961 0.01326136335081
H	2.69003568055043	-0.96157772621332 -0.01842678226816
C	-0.03670405412794	-1.37850467722257 -0.03604754751781
C	-1.34238432063545	0.44210635644949 -0.02124706062408
O	-2.30248534129122	1.06596396762097 -0.01131550466097
N	-1.27661153192580	-0.88895610701442 -0.06675916753675
O	0.24632506696265	-2.48799658466631 -0.04017583538759
O	-2.31914764306685	-1.63785295565897 0.07263273250752
H	-2.62086173951278	-1.84466002825284 -0.75994701729414

Phthalimide-N-oxyl radical (PINO)



Total Enthalpy	... -610.42885137 Eh	
O	2.08057905987686	-1.79622231951448 0.24374276332005
C	0.22269678948874	-1.04745617940831 1.30657206537090
C	0.13329486796723	-1.57615596955380 -0.89735315832076
C	-1.08720094900599	-0.82765952035736 0.82301513255755
C	-1.13943400143164	-1.13670141054996 -0.46712826848105
C	-2.25664313926320	-1.01865827608948 -1.15696318986418
C	-2.15045086473329	-0.39022181448424 1.46807985911311
C	-3.27701968721630	-0.26975899511613 0.77898502394791
H	-4.11976046295580	0.06574370979163 1.24596213300260
C	-3.32924719683336	-0.57885904702196 -0.51302064543173
H	-4.21113053376931	-0.47496803506115 -1.01533163991754
O	0.49371143869239	-1.92684222615317 -1.91973167038741
O	0.66712045530394	-0.90230390103554 2.34558642721612

H	-2.28724004595632	-1.25822702440706	-2.14772339033918
H	-2.10101037498259	-0.15561992018913	2.45926482369670
N	0.91894564481865	-1.49876407084985	0.21939673451690

H₂O

of imaginary frequencies = 0

Total Enthalpy ... -79.16317555 Eh

O	-1.93291642291460	0.93371873437967	-0.00833279856100
H	-1.02847829569868	0.94006259685901	-0.03464893506657
H	-2.20887528138672	1.13566866876132	-0.84607826637243

Hydroxyl radical

of imaginary frequencies = 0

Total Enthalpy ... -78.29067201 Eh

O	-1.94799237405106	0.93768387191607	-0.02478131096353
H	-2.25329762594894	1.14005612808393	-0.86427868903647

Spectral data of the synthesized compounds 2-4

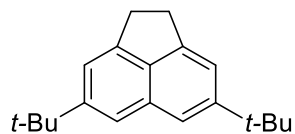
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3.43

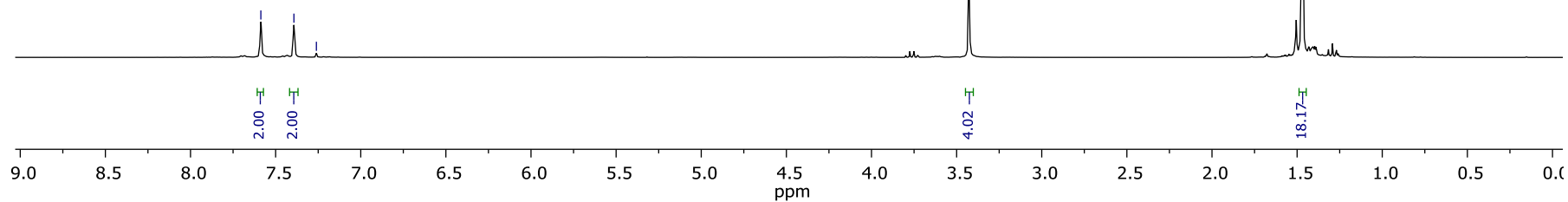
1.47

¹H NMR (300.13 MHz, CDCl₃)

4,7-di-tert-butyl-1,2-dihydroacenaphthylene 2



Parameter	Value
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2 Solvent	CDCl ₃
3 Temperature	298.0
4 Pulse Sequence	zg
5 Number of Scans	1
6 Receiver Gain	32.0
7 Relaxation Delay	0.1000
8 Pulse Width	14.7000
9 Acquisition Time	2.7724
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11 Spectral Width	5882.4
12 Lowest Frequency	-1094.2
13 Nucleus	¹ H
14 Acquired Size	16308
15 Spectral Size	65536



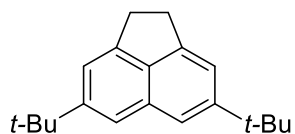
— 151.42
— 145.21
— 136.42
— 130.85
117.60
117.53

77.58
77.16
76.74

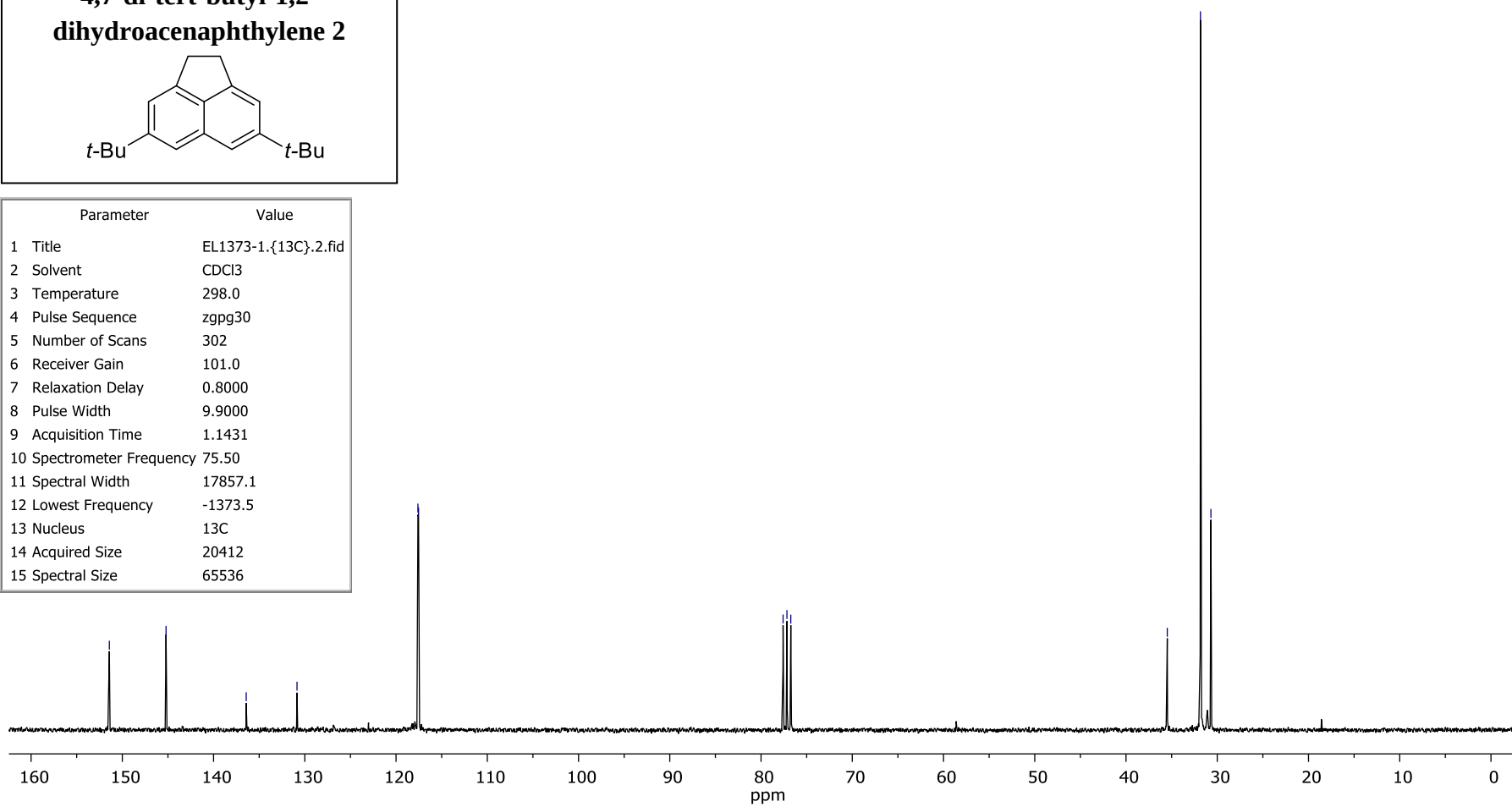
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¹³C NMR (75.48 MHz, CDCl₃)

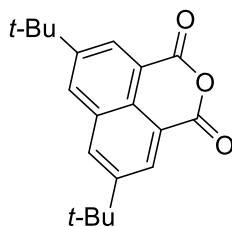
4,7-di-tert-butyl-1,2-dihydroacenaphthylene 2



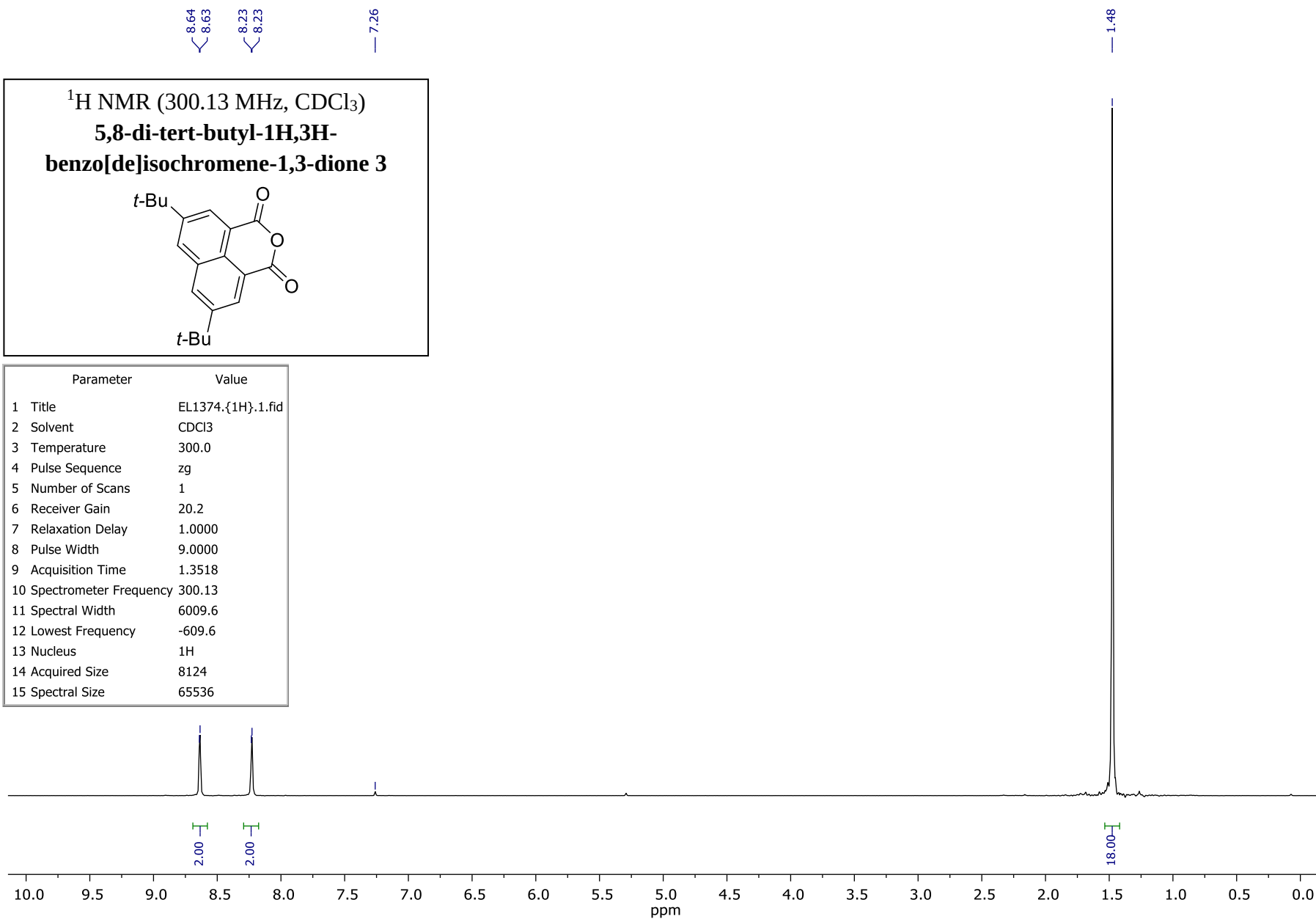
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6 Receiver Gain	101.0
7 Relaxation Delay	0.8000
8 Pulse Width	9.9000
9 Acquisition Time	1.1431
10 Spectrometer Frequency	75.50
11 Spectral Width	17857.1
12 Lowest Frequency	-1373.5
13 Nucleus	¹³ C
14 Acquired Size	20412
15 Spectral Size	65536



¹H NMR (300.13 MHz, CDCl₃)
5,8-di-tert-butyl-1H,3H-benzo[de]isochromene-1,3-dione 3



Parameter	Value
1 Title	EL1374.{1H}.1.fid
2 Solvent	CDCl3
3 Temperature	300.0
4 Pulse Sequence	zg
5 Number of Scans	1
6 Receiver Gain	20.2
7 Relaxation Delay	1.0000
8 Pulse Width	9.0000
9 Acquisition Time	1.3518
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13 Nucleus	1H
14 Acquired Size	8124
15 Spectral Size	65536

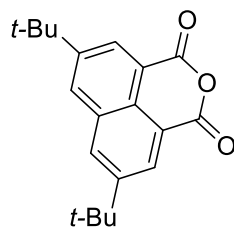


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151.10
132.30
131.48
130.81
127.17
118.37

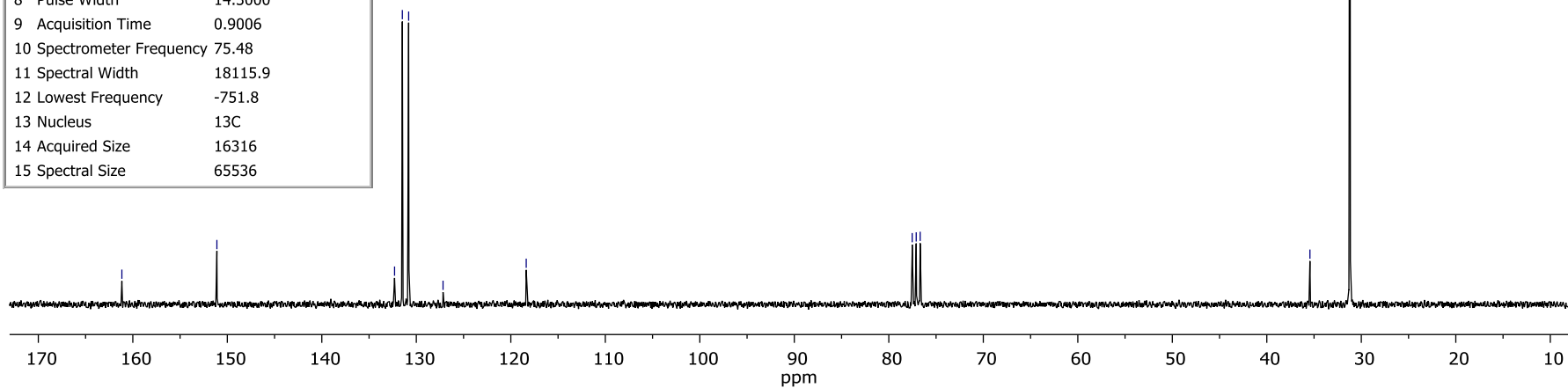
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35.44
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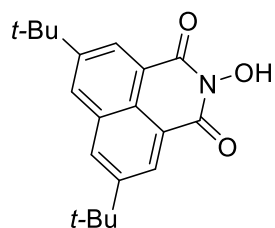
¹³C NMR (75.48 MHz, CDCl₃)
5,8-di-tert-butyl-1H,3H-benzo[de]isochromene-1,3-dione 3



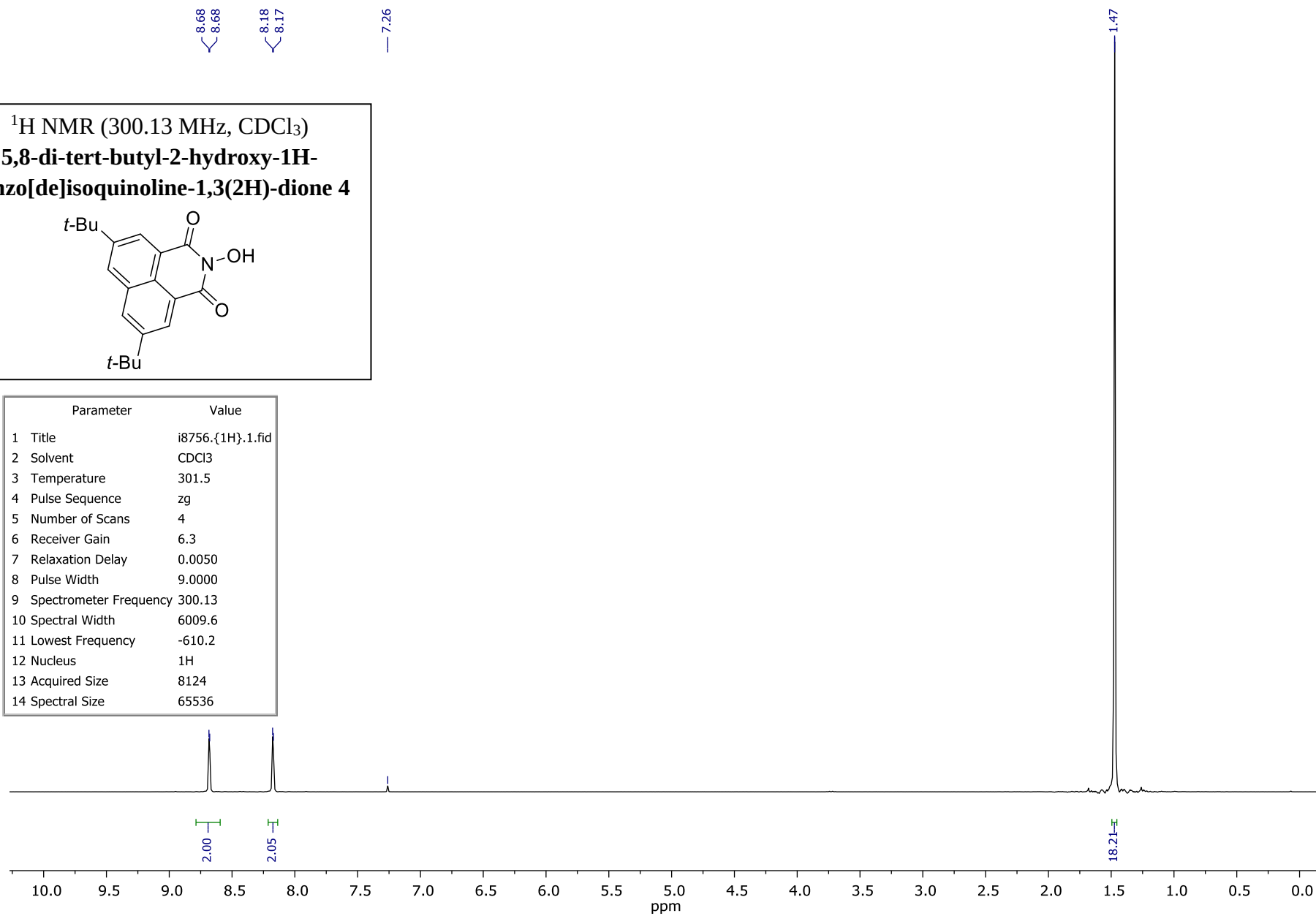
Parameter	Value
1 Title	EL1374.{ ¹³ C}.130.fid
2 Solvent	CDCl ₃
3 Temperature	300.0
4 Pulse Sequence	zgpg60base
5 Number of Scans	254
6 Receiver Gain	3250.0
7 Relaxation Delay	1.0000
8 Pulse Width	14.3000
9 Acquisition Time	0.9006
10 Spectrometer Frequency	75.48
11 Spectral Width	18115.9
12 Lowest Frequency	-751.8
13 Nucleus	¹³ C
14 Acquired Size	16316
15 Spectral Size	65536



¹H NMR (300.13 MHz, CDCl₃)
5,8-di-tert-butyl-2-hydroxy-1H-benzo[de]isoquinoline-1,3(2H)-dione 4



Parameter	Value
1 Title	i8756.{1H}.1.fid
2 Solvent	CDCl ₃
3 Temperature	301.5
4 Pulse Sequence	zg
5 Number of Scans	4
6 Receiver Gain	6.3
7 Relaxation Delay	0.0050
8 Pulse Width	9.0000
9 Spectrometer Frequency	300.13
10 Spectral Width	6009.6
11 Lowest Frequency	-610.2
12 Nucleus	¹ H
13 Acquired Size	8124
14 Spectral Size	65536

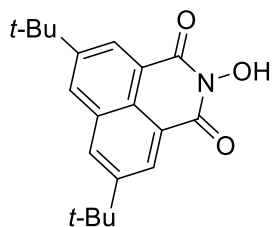


— 159.97 — 150.76
 132.27 130.57 130.14
 — 123.34 — 121.07

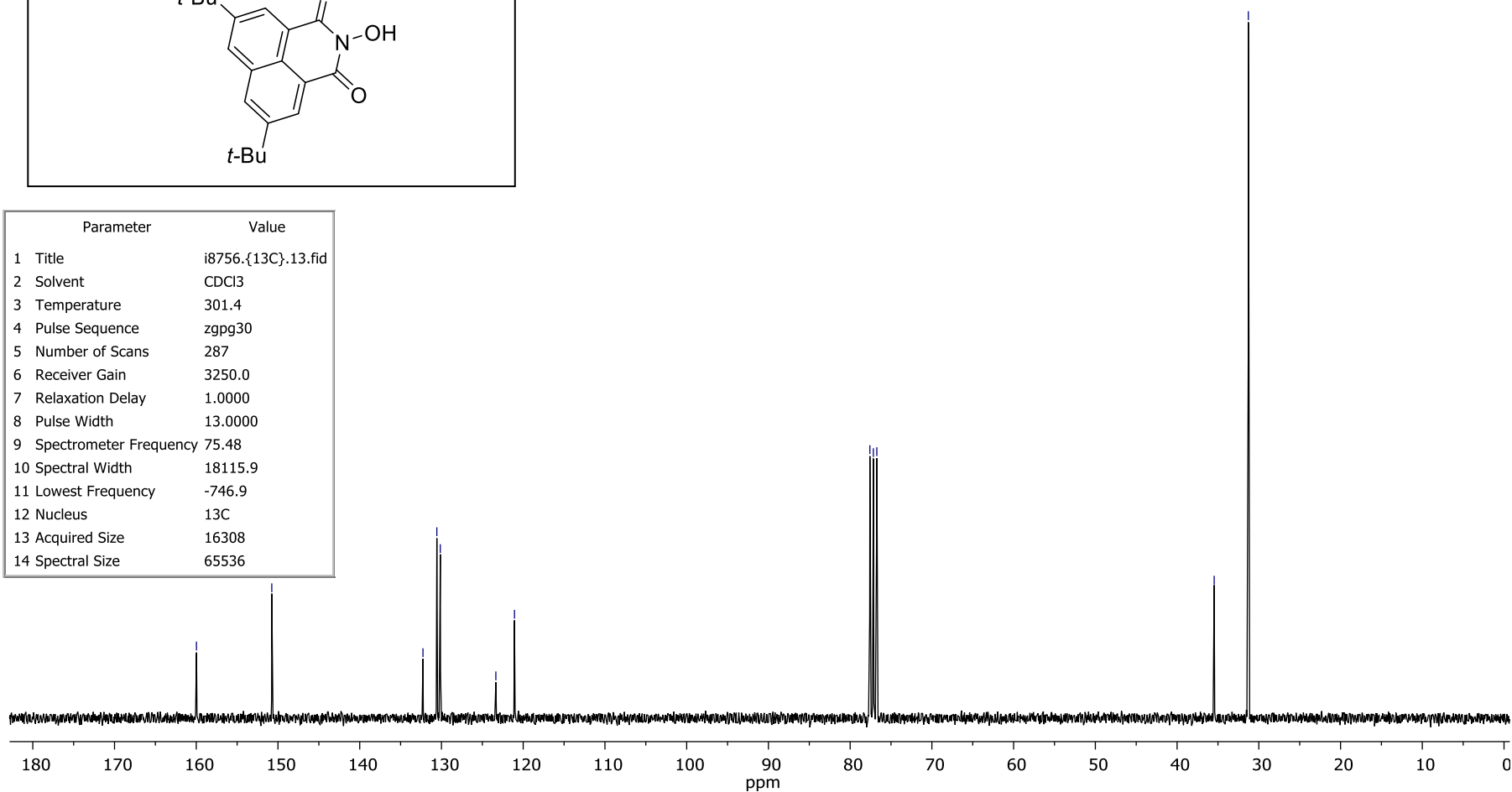
77.58
 77.16
 76.74

— 35.46 — 31.28

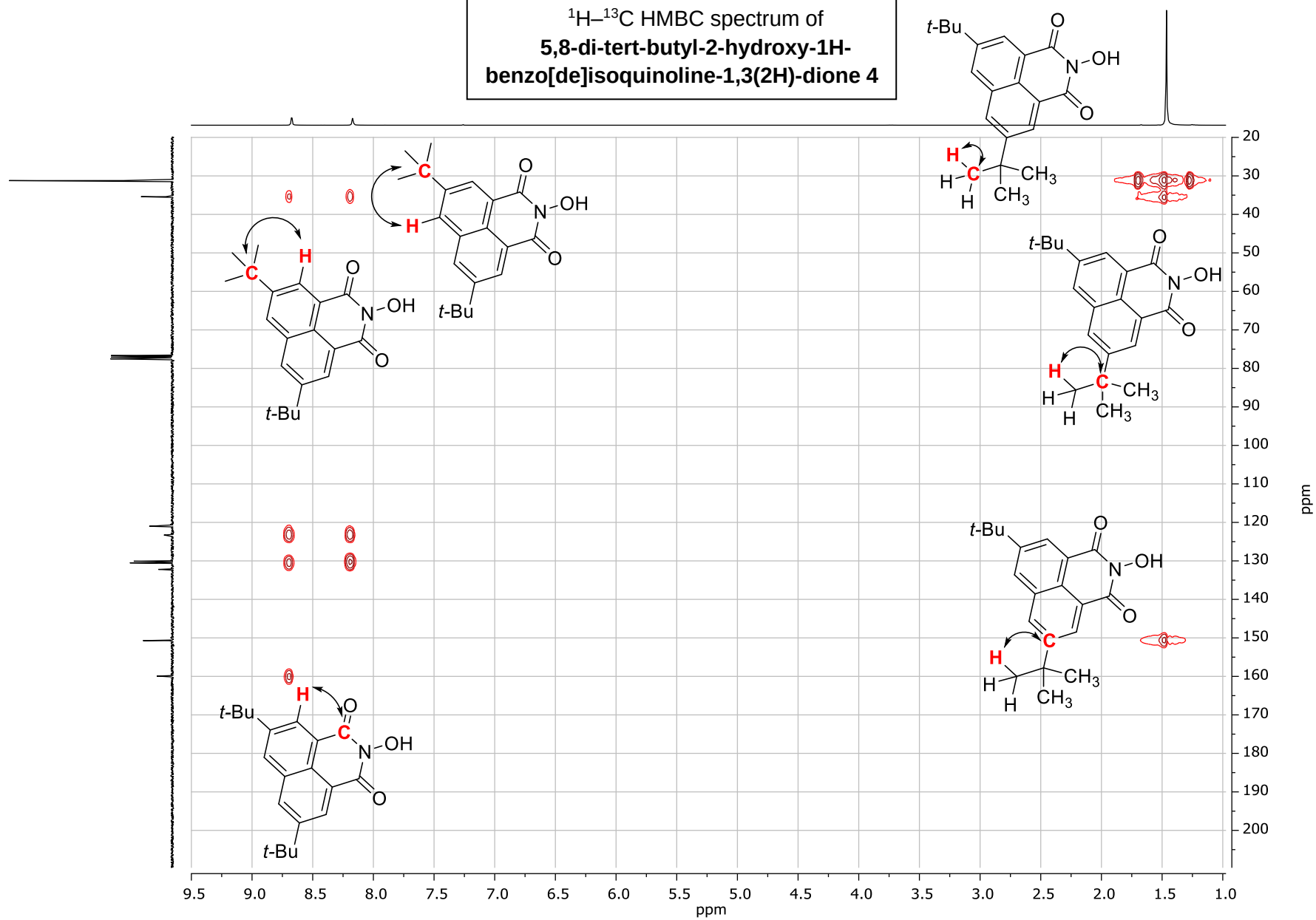
¹³C NMR (75.48 MHz, CDCl₃)
5,8-di-tert-butyl-2-hydroxy-1H-benzo[de]isoquinoline-1,3(2H)-dione 4



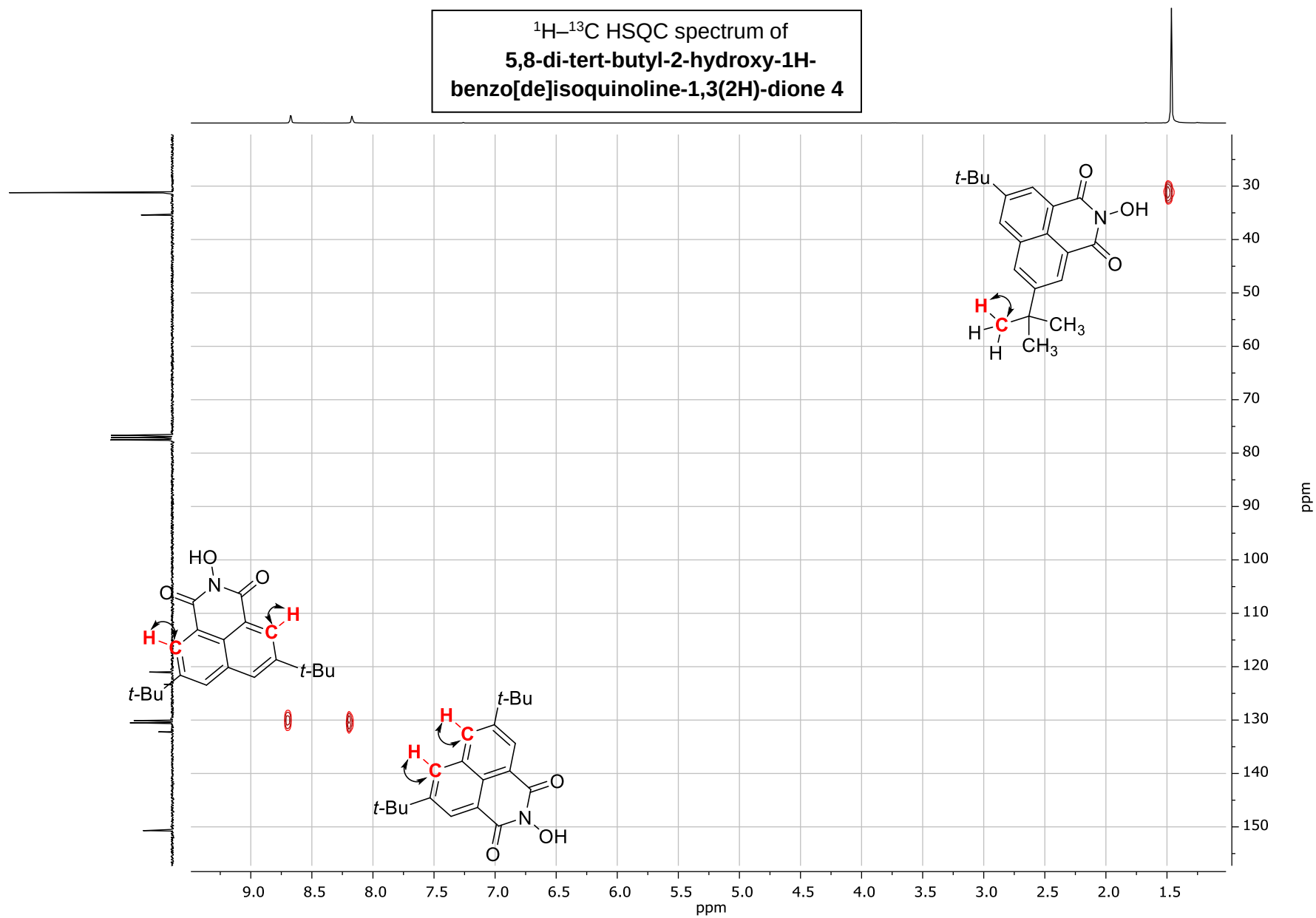
Parameter	Value
1 Title	i8756.{13C}.13.fid
2 Solvent	CDCl ₃
3 Temperature	301.4
4 Pulse Sequence	zgpg30
5 Number of Scans	287
6 Receiver Gain	3250.0
7 Relaxation Delay	1.0000
8 Pulse Width	13.0000
9 Spectrometer Frequency	75.48
10 Spectral Width	18115.9
11 Lowest Frequency	-746.9
12 Nucleus	¹³ C
13 Acquired Size	16308
14 Spectral Size	65536

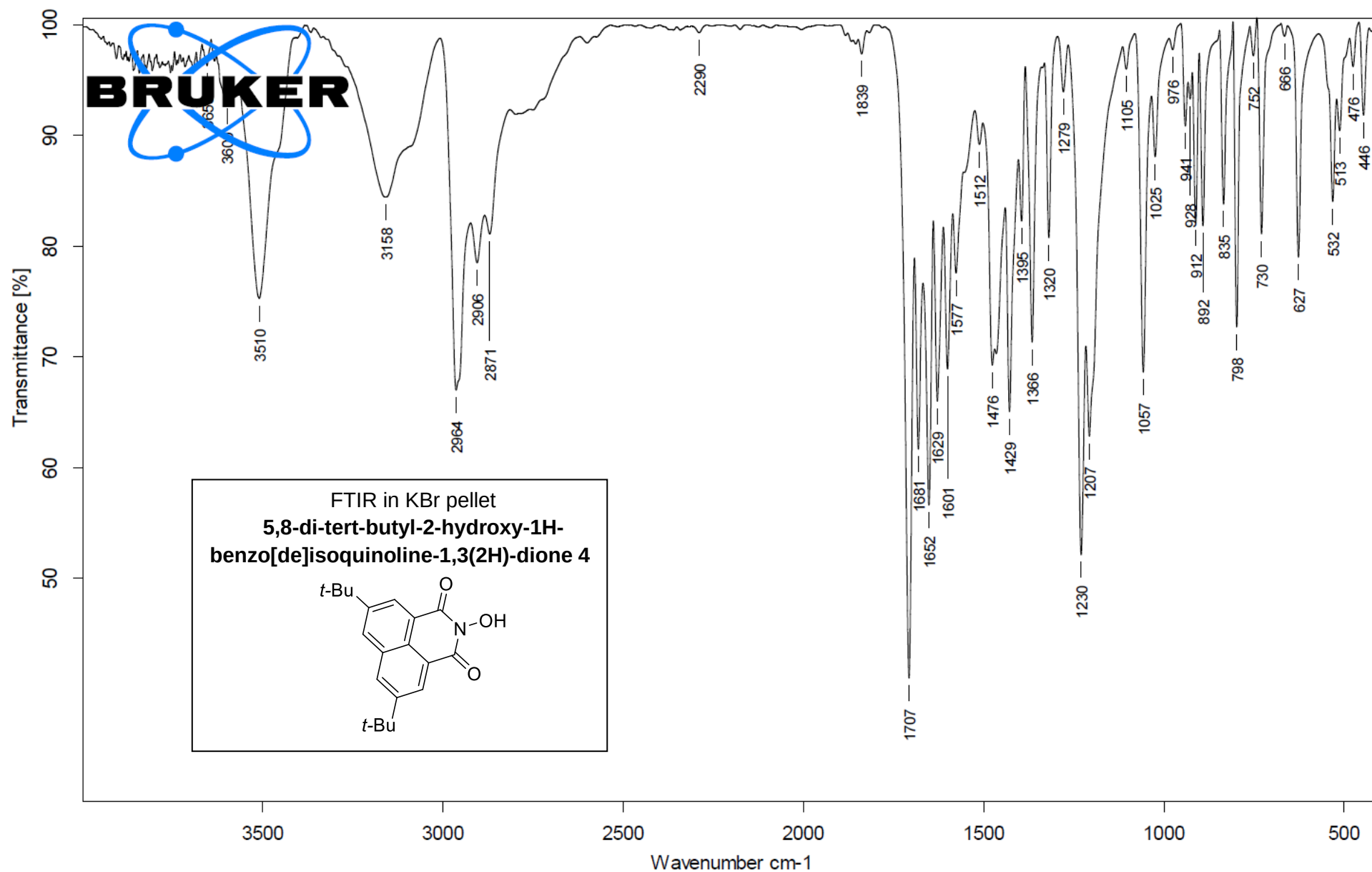


^1H - ^{13}C HMBC spectrum of
5,8-di-tert-butyl-2-hydroxy-1H-
benzo[de]isoquinoline-1,3(2H)-dione 4



^1H - ^{13}C HSQC spectrum of
5,8-di-tert-butyl-2-hydroxy-1H-
benzo[de]isoquinoline-1,3(2H)-dione 4





Display Report

Analysis Info

Analysis Name D:\Data\Chizhov\Miscellaneous\Terent'ev\Lopat'eva\II-1060_&clblow.d
 Method tune_low_1550.m
 Sample Name /TERN LL-1060or1039
 Comment CH3CN 100 %, dil. 200, calibrant added

Acquisition Date 25.08.2022 14:15:38

Operator BDAL@DE
 Instrument / Ser# maXis 43

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	1550 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Source

