Supporting Information

Reductive amination of tertiary anilines and aldehydes

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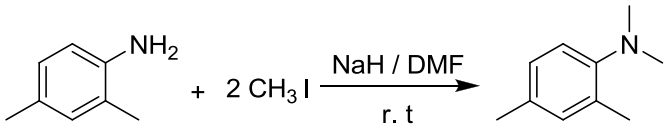
Ⅰ. General information

All reagents were purchased from commercial sources and used without further treatment, unless otherwise indicated. 1, 2-dichloroethane (DCE) was dried over CaH_2 and distilled. The ^1H NMR spectra were recorded at 400 or 500 MHz in $CDCl_3$ and the ^{13}C NMR spectra were recorded at 100 or 125 MHz in $CDCl_3$ with TMS as internal standard. Melting points were obtained with a micro melting point XT4A Beijing Keyi electrooptic apparatus and were uncorrected. High resolution mass spectra were recorded on Bruck microtof. All reactions were monitored by TLC with Taizhou GF254 silica gel coated plates. Flash column chromatography was carried out using 300-400 mesh silica gel at increased pressure.

Ⅱ. Synthesis procedure

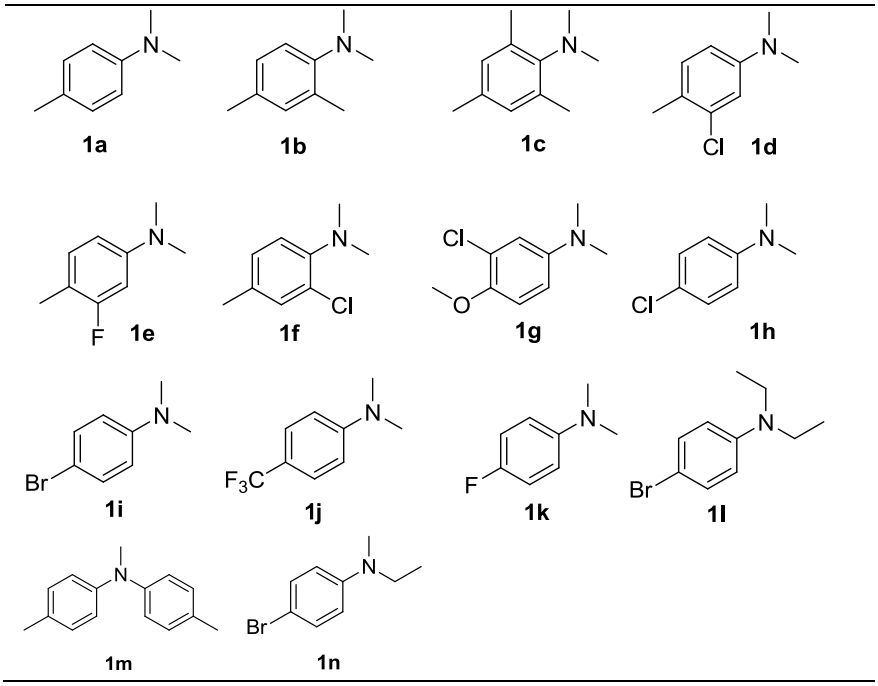
(i) General procedure for the preparation of *N*,*N*-dimethylanilines

1a was purchased from J & K Chemical Limited.
Substrates **1b**–**n** were prepared through the reaction of corresponding anilines and alkyl halide in anhydrous DMF at room temperature (**1b as an example**).

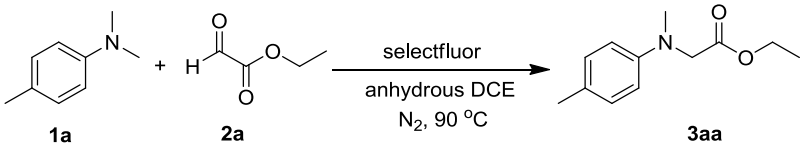


1 b

To the well stirred anhydrous DMF (25 mL), cooling by ice-water, added 2,4-dimethylaniline (1.41 g, 10 mmol), NaH (70%) (0.82 g, 24 mmol) and CH_3I (1.49 mL, 24 mmol) . The reaction mixture was stirred at room temperature for 24 h (monitored by TLC) before it was slowly poured into water (80 mL). Extracted with CH_2Cl_2 (4×10 mL), then the organic phase was washed with water (3×20 mL), the solvent was removed under reduced pressure, and the residue was purified by column chromatography (eluent: diethyl ether/petroleum ether = 1/50) afforded the product **1b**.



(ii) General procedure for the Selectfluor-mediated reductive amination (3aa as an example)

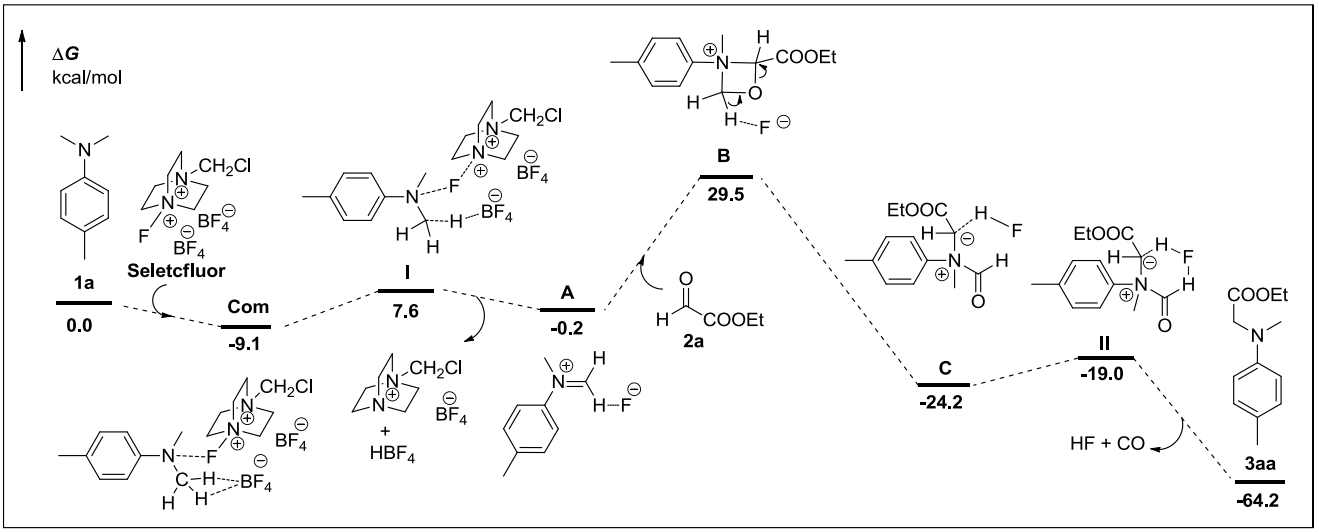


To a solution of *N*,*N*-dimethyl-*p*-toluidine (**1a**) (54.0 mg, 0.4 mmol) in anhydrous DCE (3.0 mL) was added ethyl 2-oxoacetate (**2a**)(0.33 ml, 1.6 mmol) and Selectfluor (283 mg, 0.8 mmol) under nitrogen atmosphere at room temperature. The mixture was stirred at 90 °C for 9.0 h (monitored by TLC), extracted with dichloromethane (5×3 mL), and dried over anhydrous Na_2SO_4. The solvent was removed under reduced pressure, and the residue was purified by a shot flash silica gel column chromatography (eluent: diethyl ether/petroleum ether = 1/40) to give compound **3aa** as a colorless oil (65 mg, 79%).

Ⅲ. DFT study

All calculations were carried out by using Gaussian 09 program package[1] at the B3LYP/6-31G(d) level of theory. Frequency calculations at the same theoretical level were perform to verify the stationary points to be real minima with zero imaginary frequency or tranisiton states with only one imaginary frequency, and also to provide free energies at 289.15 K. The NBO analysis was performed as implemented in the Gaussian 09 code.

Through DFT calculations, the possible mechanism and corresponding Gibbs energy profiles is depicted in Scheme 1. Initially, the interaction between **1a** and Selectfluor forms complex **Com** exothermically, then the F^+ reagent nucleophililic attacks the nitrogen centre of **1a**, and F^+ is reduced to F^-. Simultaneously, the counter anion BF_4^- of Selectfluor abstracts the proton from α-carbon to the nitrogen atom in an intramolecular fashion with the Gibbs energy barrier ($\Delta\Delta G‡$) 16.7 kcal/mol, forming an iminium ion **A**. This redox process was confirmed by the NBO analysis that **1a**, severving as reductive agent, transferred 1.16e to Selectflour in transition state **I**. In the next step, a key nucleophilic attack of the oxygen atom on the carbonyl group of **2a** to **A** takes place via a four-membered transition state **B**, providing an ammonium ylide **C** with the relative Gibbs energy (ΔG) of -24.2 kcal/mol. This ring forming process is the rate-limiting step, and the calculated ΔG value (29.5 kcal/mol) of **B** suggests that the reaction can proceed under the condition of heating, which agrees well with the experimental temperature (90 °C). The calculation result showed that the formation of **C** via **B** from **A** is exothermic by approximately 24.0 kcal/mol. Finally, the proton transfer via a six-membered transitional state **II** (-19.0 kcal/mol) results in the reductive amination product **3aa**. This proposed mechanism show qualitative accordance with our labelling experiments.



Scheme 1 Gibbs energy profiles for the plausible mechanism at the B3LYP/6-31G(d) level. The relative energy is given in kcal/mol relative to the formation of **3aa**.

1a

Electronic and zero-point energy: -405.330738 a.u.
Enthalpy: -405.319258 a.u.
Free energy: -405.367666 a.u.

C	-1.46767000	-1.19134800	-0.03460800
C	-0.07583900	-1.20346000	-0.07116900
C	0.66200700	-0.00000900	-0.10139800
C	-0.07580900	1.20342400	-0.07038900
C	-1.46765600	1.19129800	-0.03385600
C	-2.20108400	-0.00001900	-0.01376900
H	-1.99527600	-2.14333700	-0.01825000
H	0.43310600	-2.16008600	-0.08136700
H	0.43312700	2.16006100	-0.07980500
H	-1.99524500	2.14328400	-0.01684700
C	-3.71023900	-0.00004400	0.06150800
H	-4.06753200	-0.00094700	1.10090700
H	-4.13607000	0.88536600	-0.42396400
H	-4.13613600	-0.88457600	-0.42551100
N	2.05623900	-0.00006700	-0.17790500
C	2.77088100	1.24133000	0.06251800
H	3.84275200	1.06300500	-0.04888000
H	2.48808900	2.00360500	-0.67268400
H	2.59215300	1.65734900	1.06840400
C	2.77078500	-1.24128900	0.06387300
H	3.84278400	-1.06275300	-0.04587500
H	2.59054500	-1.65700100	1.06959400
H	2.48933700	-2.00388800	-0.67153400

Selectflour

Electronic and zero-point energy: -1793.191537 a.u.
Enthalpy: -1793.169191 a.u.
Free energy: -1793.244218 a.u.

N	-0.28933600	0.51018200	0.94266900
C	1.09424900	0.22453600	1.49590200
H	1.82156800	0.82206200	0.94786800
H	1.09353700	0.49620300	2.55342900
C	-1.28801500	-0.38203400	1.67251700
H	-1.16149800	-0.16958600	2.73621700
H	-2.29001100	-0.10374600	1.33751000
C	-0.24786100	0.22716600	-0.56135200
H	-1.20637900	0.50523000	-1.00247600
H	0.57208800	0.82775300	-0.95517500
C	-1.00196400	-1.86307700	1.35718800
H	-0.82109200	-2.46031700	2.25176000
H	-1.80161300	-2.28035500	0.74716400
C	1.40500700	-1.28415500	1.29967700
H	2.29843500	-1.42004700	0.68990700
H	1.48144700	-1.82722100	2.24266500
C	0.05712700	-1.26844200	-0.79699100
H	-0.79175300	-1.77280500	-1.25627800
H	0.99872100	-1.40332100	-1.33244200
N	0.25487900	-1.89864800	0.55274700
F	0.57053300	-3.25099200	0.35015100
C	-0.76425800	1.93431700	1.19204000
H	-1.75731100	1.99679200	0.74297300
H	-0.81130100	2.06778400	2.27322600
Cl	0.31006100	3.16288800	0.51153700
B	-3.67546700	0.01623300	-0.87364400
F	-4.97477300	-0.22548700	-1.19469600
F	-3.57519700	0.80404800	0.33005500
F	-2.95819500	0.68772600	-1.87195600
F	-2.97430300	-1.21639900	-0.60141600
B	3.59379700	0.10997400	-0.84828400
F	2.58989200	1.11323900	-1.01740700
F	3.87654600	0.03536900	0.53829500
F	4.70101200	0.36602400	-1.59740300
F	2.99203600	-1.14565800	-1.21169500

Com

Electronic and zero-point energy: -2198.530345 a.u.
Enthalpy: -2198.496325 a.u.
Free energy: -2198.600780 a.u.

C	-6.28892500	-0.52926600	-1.27865500
C	-5.26788900	0.34139300	-0.90433100
C	-4.54638500	0.14751500	0.29369600
C	-4.93403500	-0.94492800	1.10082000
C	-5.95763800	-1.80257100	0.70719200
C	-6.65726800	-1.62578800	-0.49220200
H	-6.82064600	-0.33674300	-2.20879700
H	-5.04769700	1.18518900	-1.54746500
H	-4.44522400	-1.12713700	2.05065700
H	-6.22357900	-2.62914500	1.36358600
C	-7.74837200	-2.57943100	-0.91978600
H	-7.34509100	-3.44237600	-1.46819900
H	-8.29641400	-2.97575000	-0.05744900
H	-8.47339700	-2.08919300	-1.57887400
N	-3.48329000	0.98360700	0.65273900
C	-3.00862400	0.96009500	2.03082400
H	-2.16352800	1.64411100	2.12253900
H	-3.78345300	1.25348100	2.75771600
H	-2.65791300	-0.04214500	2.30193100
C	-3.32640500	2.25257700	-0.04663500
H	-2.43145000	2.75278300	0.32512300
H	-3.19135600	2.08586300	-1.12190500
H	-4.19068800	2.92483200	0.08096300
N	2.67735500	-0.06931000	-1.01861800
C	1.91625600	1.10770700	-1.60085000
H	2.23780300	2.01647300	-1.09383700
H	2.14367400	1.16174200	-2.66736800
C	2.12406200	-1.34859600	-1.64372900
H	2.19230700	-1.20670900	-2.72444300
H	2.75573100	-2.17976700	-1.32024600
C	2.48144100	-0.03329900	0.49869400
H	3.07048900	-0.83325800	0.94954900
H	2.82260300	0.94840900	0.82200400
C	0.66775500	-1.56980500	-1.19106100
H	-0.01876400	-1.70368400	-2.02798300
H	0.61136000	-2.39798200	-0.48663600
C	0.40189800	0.87992700	-1.35844700
H	-0.03665900	1.72782700	-0.83456800
H	-0.14977000	0.66072300	-2.27371800
C	0.97970700	-0.17696500	0.82451900
H	0.78890400	-1.08595900	1.39353800
H	0.57434900	0.72147500	1.29329000
N	0.24881000	-0.32727100	-0.47891400
F	-1.12052000	-0.44703100	-0.18939800
C	4.16038900	-0.06420300	-1.35172000
H	4.58117200	-0.94323700	-0.85975600
H	4.24360100	-0.14224300	-2.43608700
Cl	4.98899800	1.40813200	-0.82207000
B	3.54077900	-3.29114600	0.91002700
F	3.77231600	-4.56094200	1.33774500
F	4.11247100	-3.06556300	-0.39338700
F	4.05494100	-2.30289100	1.76300100
F	2.12634500	-3.04048600	0.77539400
B	0.83779100	3.54629200	0.67386500
F	2.09165800	2.89431800	0.86723300
F	0.66170700	3.71493400	-0.71797500
F	0.75429700	4.71402100	1.36647700
F	-0.17856200	2.60954200	1.10265900

I

Imaginary frequency: -844.7255 cm**-1
Electronic and zero-point energy: -2198.502874 a.u.
Enthalpy: -2198.468328 a.u.
Free energy: -2198.574196 a.u.

C	-4.55526100	2.62324600	0.77869300

C	-3.87238300	1.41844900	0.87128500
C	-4.06540000	0.44811500	-0.11932300
C	-4.94997700	0.67965500	-1.17607100
C	-5.62853500	1.89595900	-1.24508900
C	-5.44192600	2.88991600	-0.27874600
H	-4.38400200	3.38202800	1.53764500
H	-3.15319400	1.23826000	1.65803000
H	-5.14117800	-0.08577600	-1.91972300
H	-6.31893300	2.06614900	-2.06644300
C	-6.15232300	4.21696200	-0.37050000
H	-5.45097800	5.01516600	-0.64447300
H	-6.94538800	4.19821400	-1.12344200
H	-6.59864100	4.49656700	0.59031400
N	-3.43516200	-0.86275000	-0.01145900
C	-3.06960800	-1.52127800	-1.14257300
H	-1.66415700	-1.75046000	-0.98824800
H	-3.29585200	-2.58456500	-1.16434300
H	-3.11994900	-0.94704700	-2.06091300
C	-3.89268700	-1.71140500	1.12485100
H	-3.17304400	-2.51915300	1.24337800
H	-3.93654100	-1.10031300	2.02152100
H	-4.88656100	-2.09006600	0.86822500
N	2.86249900	-0.48492300	0.55672500
C	2.18074900	-1.61974900	1.29585700
H	1.79173000	-2.32313000	0.56036300
H	2.95798600	-2.11758200	1.88344300
C	3.56778800	0.38626000	1.60170800
H	4.33541800	-0.25184000	2.04910600
H	4.03282500	1.20872500	1.06299500
C	1.78215800	0.31627100	-0.17695100
H	2.27389700	1.02536500	-0.84479000
H	1.22760100	-0.42110800	-0.75862500
C	2.51084200	0.86465900	2.63145700
H	2.71622200	0.44730700	3.62302900
H	2.55878700	1.95419000	2.69252200
C	1.05991700	-0.99239500	2.18560200
H	0.07512400	-1.25537700	1.79327200
H	1.14020800	-1.38721300	3.20378900
C	0.89394200	1.01090300	0.88434400
H	1.11455000	2.07985000	0.88932000
H	-0.15952000	0.84737500	0.64814100
N	1.15944400	0.47040600	2.22772500
F	-1.93340200	-0.38207600	0.83576400
C	3.93767600	-0.93662200	-0.39343100
H	4.37731300	-0.03414300	-0.82374600
H	4.66984300	-1.50233300	0.18271700
Cl	3.33434500	-1.99937100	-1.69678500
B	3.98901700	2.98741500	-0.84106900
F	4.61062800	4.17046800	-1.15011800
F	4.95643000	1.94112200	-0.65527100
F	3.10094100	2.56712300	-1.85069600
F	3.25647400	3.09435400	0.37808500
B	-0.67022000	-3.69504500	-0.37066200
F	0.63082100	-4.05959200	-0.41603200
F	-1.19269400	-3.52843500	0.88450800
F	-1.51160600	-4.29822600	-1.25080000
F	-0.61300500	-2.14189500	-0.93618000

A

Electronic and zero-point energy: -504.483429 a.u.
Enthalpy: -504.470974 a.u.
Free energy: -504.521463 a.u.

C	-1.56996600	1.17755800	-0.07027200
C	-0.18389600	1.05611800	-0.10557000
C	0.36804000	-0.22326500	-0.04320400
C	-0.43284700	-1.36107100	0.05156500
C	-1.82008400	-1.21125100	0.08541400
C	-2.41038300	0.05655100	0.02460500
H	-2.01293900	2.16959500	-0.12731500

H	0.54185700	1.86494000	-0.20692700
H	0.01664500	-2.34837200	0.11216100
H	-2.44872500	-2.09485900	0.16309600
C	-3.91244400	0.21877100	0.04078400
H	-4.28899000	0.52066800	-0.94510300
H	-4.41514200	-0.71408600	0.31479000
H	-4.22261100	0.99206200	0.75295100
N	1.81781300	-0.39797100	-0.04726900
C	2.36480000	-1.09077400	-0.99061300
H	3.41599400	-1.34277200	-0.92229900
H	1.79798500	-1.30536000	-1.88703800
C	2.52914600	-0.19117600	1.23476100
H	3.56847800	0.03700300	1.01435500
H	2.08601400	0.66332400	1.73119000
H	2.43045800	-1.10769400	1.83185700
F	2.35689900	1.65139800	-0.31840700

B

Imaginary frequency: -351.5309 cm**-1
Electronic and zero-point energy: -886.038128 a.u.
Enthalpy: -886.017646 a.u.
Free energy: -886.089390 a.u.

C	-3.91388100	0.56680400	-0.45992700
C	-2.62943200	1.11007400	-0.40513000
C	-1.57906800	0.37087600	0.14700700
C	-1.83628500	-0.91377200	0.64329500
C	-3.12274000	-1.44345200	0.58473300
C	-4.18532800	-0.71574100	0.02990100
H	-4.71982400	1.15813300	-0.88810400
H	-2.45615900	2.11700700	-0.77198500
H	-1.01868700	-1.50179100	1.05182600
H	-3.30215300	-2.44449300	0.96990500
C	-5.57094600	-1.31021200	-0.06127500
H	-5.68083200	-1.92412600	-0.96512300
H	-5.78720700	-1.95601600	0.79645800
H	-6.33917000	-0.53124500	-0.10134400
N	-0.24317400	0.91332100	0.26448200
C	0.34451900	1.79108200	-0.82843800
H	1.22053000	2.90153700	-0.16794200
H	-0.41773200	2.08577000	-1.55559500
C	0.00462500	1.46165100	1.61978600
H	1.02232600	1.84414500	1.66675300
H	-0.68165200	2.29673500	1.81728200
H	-0.17359700	0.67715900	2.36022500
F	1.76739300	3.49331700	0.47482800
C	0.92619100	-0.28626200	-0.93610100
O	1.31419300	0.85758300	-1.43263100
C	1.92887200	-1.24363700	-0.37178900
O	1.59602100	-2.35603400	-0.00475500
O	3.15167600	-0.72090600	-0.29345800
C	4.17962800	-1.57873300	0.26878500
H	3.86859800	-1.86952500	1.27723000
H	4.24065600	-2.48820900	-0.33749800
C	5.47510100	-0.79228700	0.26686000
H	5.37924100	0.11860000	0.86527300
H	6.27732600	-1.40526600	0.69170200
H	5.75822300	-0.50812500	-0.75118300
H	-0.01685300	-0.74687900	-1.22218100

C

Electronic and zero-point energy: -886.126079 a.u.
Enthalpy: -886.105848 a.u.
Free energy: -886.175014 a.u.

C	3.09767300	-1.39532500	0.35122900
C	1.78680000	-0.92960000	0.39229400
C	1.47226300	0.28553700	-0.22914300
C	2.47092200	1.02092800	-0.86688100
C	3.77646800	0.52692800	-0.90239700
C	4.11712900	-0.68526300	-0.29761500

H	3.32651800	-2.34062100	0.83724400
H	1.02491100	-1.51216900	0.89790100
H	2.26550700	1.96754500	-1.34791100
H	4.54045300	1.10688700	-1.41352200
C	5.52851500	-1.21935000	-0.33931100
H	5.57213800	-2.18454300	-0.85886700
H	6.20326700	-0.53041600	-0.85614300
H	5.92161100	-1.38145400	0.67148300
N	0.06250900	0.80263100	-0.14365000
C	-0.13440900	1.19860200	1.36797100
H	-0.86360800	-1.31807300	0.80669000
H	-0.40941900	0.29929700	1.93121000
C	-0.15384000	2.03735300	-0.97652000
H	-1.19610500	2.31972300	-0.85244700
H	0.48236100	2.84439600	-0.61508400
H	0.06625700	1.78731100	-2.01408800
F	-0.81819300	-1.96968300	1.59172300
C	-0.93543100	-0.28768300	-0.43467800
O	0.14420100	2.28567400	1.75398300
C	-2.26692000	0.16212800	-0.14288900
O	-2.51353600	1.09875800	0.63338800
O	-3.23346100	-0.59742300	-0.70233700
C	-4.58035800	-0.35154800	-0.24229800
H	-4.61285400	-0.47214500	0.84529100
H	-4.85352800	0.68433500	-0.46917300
C	-5.48537500	-1.34370200	-0.94885400
H	-5.19570700	-2.37220500	-0.71139100
H	-6.52284500	-1.19375700	-0.63015900
H	-5.43608100	-1.21399600	-2.03500100
H	-0.72494400	-0.79147500	-1.37170300

II

Imaginary frequency: -404.8901 cm**-1
Electronic and zero-point energy: -886.118678 a.u.
Enthalpy: -886.098997 a.u.
Free energy: -886.166661 a.u.

C	3.16077800	-0.89260200	1.03582700
C	1.89038700	-0.33114400	0.94791800
C	1.46527400	0.20067000	-0.27717500
C	2.30657400	0.16343800	-1.38962600
C	3.57604400	-0.40727400	-1.27271400
C	4.02809900	-0.94441600	-0.06459200
H	3.48413700	-1.29915200	1.99140800
H	1.21890500	-0.27272600	1.80939700
H	2.00489300	0.55760000	-2.35157000
H	4.22111300	-0.43152500	-2.14734900
C	5.39945800	-1.56494100	0.05630000
H	5.97857300	-1.09593900	0.86063200
H	5.33163800	-2.63438600	0.29145100
H	5.96988300	-1.46283900	-0.87196100
N	0.09639700	0.79694500	-0.35083900
C	0.06023800	2.05573900	0.75476100
H	-0.80901900	-0.29895900	1.17665000
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H	-0.25413900	0.60297400	-2.43037000
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C	-2.35947900	0.03520300	-0.23444300
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H	-4.75669700	-0.57582800	-1.21071900
C	-5.07833000	-2.38187300	-0.03323600
H	-4.79930600	-2.78368700	0.94574200

H	-6.17132800	-2.33482100	-0.08881300
H	-4.72355100	-3.07280300	-0.80482300
H	-0.61290200	-1.21131600	-0.41732300

2a

Electronic and zero-point energy: -381.592786 a.u.
Enthalpy: -381.584100 a.u.
Free energy: -381.625581 a.u.

C	-1.78730500	-0.73951900	-0.00026000
H	-1.48531700	-1.80566400	-0.00096200
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C	-0.62606000	0.26673300	-0.00013400
O	-0.75592300	1.46672400	-0.00018600
O	0.53954000	-0.40298200	0.00003500
C	1.74075000	0.41179600	0.00017800
H	1.71779100	1.05733800	-0.88348500
H	1.71782900	1.05703900	0.88406500
C	2.93234500	-0.52507000	0.00000600
H	2.92830800	-1.16467300	-0.88821100
H	3.85931200	0.05829300	0.00010700
H	2.92832100	-1.16501000	0.88798000

3aa

Electronic and zero-point energy: -672.448875 a.u.
Enthalpy: -672.431936 a.u.
Free energy: -672.495303 a.u.

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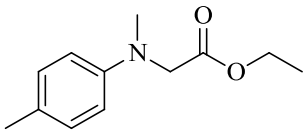
C	1.14644600	0.08217400	-1.10550000
C	0.81656500	0.89035800	0.00471200
C	1.72415800	0.90080300	1.08315700
C	2.90388200	0.16078700	1.03476900
C	3.24047400	-0.63038200	-0.06758700
H	2.54553100	-1.25513300	-2.00941500
H	0.48132300	0.01018200	-1.95847200
H	1.52006700	1.49287100	1.96732400
H	3.57889000	0.20313600	1.88742500
C	4.50940400	-1.45001100	-0.10134300
H	4.31102900	-2.51622500	0.07456800
H	5.01323100	-1.37470000	-1.07243500
H	5.21707200	-1.12024400	0.66683600
N	-0.34707400	1.66841200	0.03033600
H	-0.89679900	1.60099400	-1.99991600
C	-0.77762900	2.25388400	1.28981600
H	-1.70526100	2.80680100	1.12558900
H	-0.96194800	1.49818300	2.06749200
H	-0.03198000	2.96472600	1.66520300
C	-1.33735900	1.48863500	-1.00459400
C	-2.10221500	0.15681100	-1.01245200
O	-2.55327800	-0.35127700	-2.01608600
O	-2.24649700	-0.34712500	0.22695000
C	-2.95725900	-1.60548200	0.32040100
H	-3.96754400	-1.46776600	-0.07849500
H	-2.44838900	-2.33997300	-0.31133200
C	-2.96767300	-2.01623400	1.78032000
H	-3.47557300	-1.26622000	2.39532700
H	-3.49599800	-2.96918700	1.89374600
H	-1.94765200	-2.13974800	2.15758900
H	-2.08499700	2.28456400	-0.91137700

Reference

1. M. J. Frisch, G. W. Trucks, H. B. Schlegel, G. E. Scuseria, M. A. Robb, J. R. Cheeseman, G. Scalmani, V. Barone, B. Mennucci, G. A. Petersson, H. Nakatsuji, M. Caricato, X. Li, H. P. Hratchian, A. F. Izmaylov J., Bloino, G. Zheng, J. L. Sonnenberg, M. Hada, M. Ehara, K. Toyota, R. Fukuda, J. Hasegawa, M. Ishida, T. Nakajima, Y. Honda, O. Kitao, H. Nakai, T. Vreven, J. A. Montgomery, Jr., J. E. Peralta, F. Ogliaro, M. Bearpark, J. J. Heyd, E. Brothers, K. N. Kudin, V. N. Staroverov, R. Kobayashi, J. Normand, K. Raghavachari, A. Rendell, J. C. Burant, S. S. Iyengar, J. Tomasi, M. Cossi, N. Rega, J. M. Millam, M. Klene, J. E. Knox, J. B. Cross, V. Bakken, C. Adamo, J.

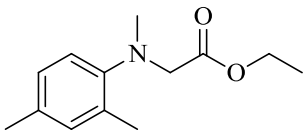
Jaramillo, R. Gomperts, R. E. Stratmann, O. Yazyev, A. J. Austin, R. Cammi, C. Pomelli, J. W. Ochterski, R. L. Martin, K. Morokuma, V. G. Zakrzewski, G. A. Voth, P. Salvador, J. J. Dannenberg, S. Dapprich, A. D. Daniels, O. Farkas, J. B. Foresman, J. V. Ortiz, J. Cioslowski and D. J. Fox, Gaussian, Inc., Wallingford CT, 2009.

IV. Spectra data for the products



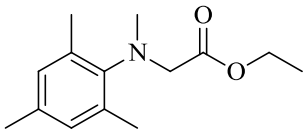
ethyl 2-(methyl(*p*-tolyl)amino)acetate 3aa

Colorless oil. ^{1}H NMR (500 MHz; $CDCl_3$): δ = 1.24 (t, J = 7.0 Hz, 3H), 2.24 (s, 3H), 3.03 (s, 3H), 4.02 (s, 2H), 4.16 (q, J = 7.0 Hz, 2H), 6.62 (d, J = 8.5 Hz, 2H), 7.04 (d, J = 8.5 Hz, 2H). ^{13}C NMR (125 MHz; $CDCl_3$): δ = 14.2, 20.2, 39.6, 54.7, 60.7, 112.6, 126.5, 129.7, 146.8, 171.1. HRMS (ESI-TOF) Calcd for $C_{12}H_{17}NO_2$, $[M+H]^+$ 208.1332; Found 208.1338.



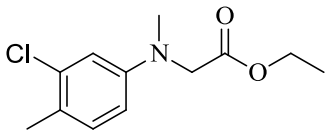
ethyl 2-((2,4-dimethylphenyl)(methyl)amino)acetate 3ba

Colorless oil. ^{1}H NMR (500 MHz; $CDCl_3$): δ = 1.25 (t, J = 7.0 Hz, 3H), 2.26 (s, 3H), 2.28 (s, 3H), 2.83 (s, 3H), 3.68 (s, 2H), 4.16 (q, J = 7.0 Hz, 2H), 6.95 (d, J = 8.0 Hz, 1H), 6.98 (s, 1H), 7.01 (d, J = 8.0 Hz, 1H). ^{13}C NMR (125 MHz; $CDCl_3$): δ = 14.0, 18.0, 20.5, 41.5, 57.4, 60.3, 120.0, 126.6, 131.7, 131.9, 132.3, 148.0, 170.9. HRMS (ESI-TOF) Calcd for $C_{13}H_{19}NO_2$, $[M+H]^+$ 222.1489; Found 222.1490.



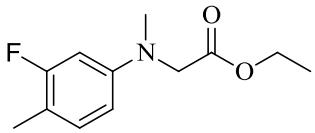
ethyl 2-(mesityl(methyl)amino)acetate 3ca

Colorless oil. ^{1}H NMR (500 MHz; $CDCl_3$): δ = 1.27 (t, J = 7.0 Hz, 3H), 2.23 (s, 3H), 2.30 (s, 6H), 2.83 (s, 3H), 3.74 (s, 2H), 4.17 (q, J = 7.0 Hz, 2H), 6.82 (s, 2H). ^{13}C NMR (125 MHz; $CDCl_3$): δ = 14.2, 18.8, 20.7, 40.9, 57.7, 60.4, 129.3, 134.8, 137.0, 146.4, 172.2. HRMS (ESI-TOF) Calcd for $C_{14}H_{21}NO_2$, $[M+H]^+$ 236.1645; Found 236.1649.



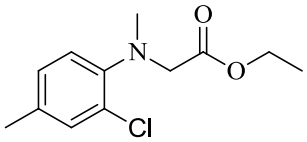
ethyl 2-((3-chloro-4-methylphenyl)(methyl)amino)acetate 3da

Colorless oil. ^{1}H NMR (500 MHz; $CDCl_3$): δ = 1.24 (t, J = 7.0 Hz, 3H), 2.25 (s, 3H), 3.02 (s, 3H), 4.01 (s, 2H), 4.17 (q, J = 7.0 Hz, 2H), 6.48 (dd, J_1 = 2.5 Hz, J_2 = 8.5 Hz, 1H). 6.68 (d, J = 3.0 Hz, 1H), 7.04 (d, J = 8.5 Hz, 1H). ^{13}C NMR (125 MHz; $CDCl_3$): δ = 14.2, 18.8, 39.6, 54.4, 60.9, 110.9, 113.0, 124.2, 131.2, 134.9, 148.1, 170.7. HRMS (ESI-TOF) Calcd for $C_{12}H_{16}ClNO_2$, $[M+H]^+$ 242.0942; Found 242.0949.



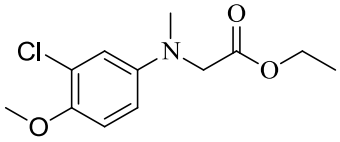
ethyl 2-((3-fluoro-4-methylphenyl)(methyl)amino)acetate 3ea

Colorless oil. ^{1}H NMR (500 MHz; $CDCl_3$): δ = 1.25 (t, J = 7.0 Hz, 3H), 2.15 (d, J = 1.0 Hz, 3H), 3.03 (s, 3H), 4.01 (s, 2H), 4.17 (q, J = 7.0 Hz, 2H), 6.35-6.38 (m, 2H), 7.00 (t, J = 8.5 Hz, 1H). ^{13}C NMR (125 MHz; $CDCl_3$): δ = 13.5, 13.5, 14.2, 39.6, 54.4, 60.9, 99.5, 99.7, 107.7, 107.7, 112.6, 112.8, 131.5, 131.6, 148.6, 148.7, 161.0, 163.0, 170.7. HRMS (ESI-TOF) Calcd for $C_{12}H_{16}FNO_2$, $[M+H]^+$ 226.1238; Found 226.1242.



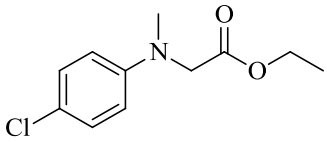
ethyl 2-((2-chloro-4-methylphenyl)(methyl)amino)acetate 3fa

Colorless oil. ^{1}H NMR (500 MHz; $CDCl_3$): δ = 1.24 (t, J = 7.0 Hz, 3H), 2.26 (s, 3H), 2.95 (s, 3H), 3.94 (s, 2H), 4.15 (q, J = 7.0 Hz, 2H), 7.00 (dd, J_1 = 1.5 Hz, J_2 = 8.0 Hz, 1H). 7.10 (d, J = 8.5 Hz, 1H), 7.15 (d, J = 1.0 Hz, 1H). ^{13}C NMR (125 MHz; $CDCl_3$): δ = 14.2, 20.3, 40.7, 56.2, 60.4, 121.8, 127.3, 127.7, 130.8, 133.3, 145.4, 170.7. HRMS (ESI-TOF) Calcd for $C_{12}H_{16}ClNO_2$, $[M+H]^+$ 242.0942; Found 242.0947.



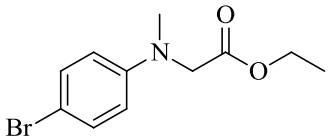
ethyl 2-((3-chloro-4-methoxyphenyl)(methyl)amino)acetate 3ga

Colorless oil. ^{1}H NMR (500 MHz; $CDCl_3$): δ = 1.25 (t, J = 7.0 Hz, 3H), 3.00 (s, 3H), 3.82 (s, 3H), 3.99 (s, 2H), 4.17 (q, J = 7.0 Hz, 2H), 6.55 (dd, J_1 = 3.0 Hz, J_2 = 9.0 Hz, 1H). 6.75 (d, J = 3.0 Hz, 1H), 6.84 (d, J = 9.0 Hz, 1H). ^{13}C NMR (125 MHz; $CDCl_3$): δ = 14.2, 39.8, 54.8, 56.8, 60.9, 111.7, 113.8, 114.9, 123.2, 144.0, 147.2, 170.7. HRMS (ESI-TOF) Calcd for $C_{12}H_{16}ClNO_3$, $[M+H]^+$ 258.0891; Found 258.0898.



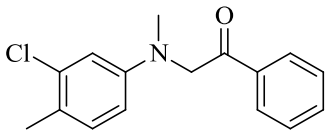
ethyl 2-((4-chlorophenyl)(methyl)amino)acetate 3ha

Colorless oil. ^{1}H NMR (500 MHz; $CDCl_3$): δ = 1.24 (t, J = 7.0 Hz, 3H), 3.04 (s, 3H), 4.03 (s, 2H), 4.17 (q, J = 7.0 Hz, 2H), 6.60 (d, J = 9.0 Hz, 2H), 7.17 (dd, J_1 = 1.5 Hz, J_2 = 6.5 Hz, 2H). ^{13}C NMR (125 MHz; $CDCl_3$): δ = 14.1, 39.6, 54.4, 60.9, 113.4, 122.2, 128.9, 147.5, 170.5. HRMS (ESI-TOF) Calcd for $C_{11}H_{14}ClNO_2$, $[M+H]^+$ 228.0786; Found 228.0785.



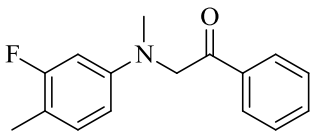
ethyl 2-((4-bromophenyl)(methyl)amino)acetate 3ia

Colorless oil. ^{1}H NMR (500 MHz; $CDCl_3$): δ = 1.24 (t, J = 7.0 Hz, 3H), 3.04 (s, 3H), 4.03 (s, 2H), 4.17 (q, J = 7.0 Hz, 2H), 6.55 (dd, J_1 = 2.0 Hz, J_2 = 7.0 Hz, 2H), 7.30 (dd, J_1 = 2.0 Hz, J_2 = 6.5 Hz, 2H). ^{13}C NMR (125 MHz; $CDCl_3$): δ = 14.1, 39.6, 54.3, 60.9, 109.3, 113.8, 131.8, 147.8, 170.5. HRMS (ESI-TOF) Calcd for $C_{11}H_{14}BrNO_2$, $[M+H]^+$ 272.0281; Found 272.0286.



2-((3-chloro-4-methylphenyl)(methyl)amino)-1-phenylethanone 3db

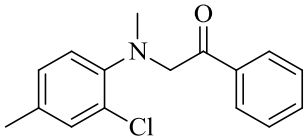
Yellow solid, m.p. 50-51 ºC. ^{1}H NMR (500 MHz; $CDCl_3$): δ = 2.24 (s, 3H), 3.06 (s, 3H), 4.75 (s, 2H), 6.47 (dd, J_1 = 3.0 Hz, J_2 = 8.5 Hz, 1H), 6.69 (d, J = 2.5 Hz, 1H). 7.02 (d, J = 8.5 Hz, 1H), 7.50 (t, J = 7.5 Hz, 2H), 7.61-7.64 (m, 1H), 7.97-7.99 (m, 2H). ^{13}C NMR (125 MHz; $CDCl_3$): δ = 18.8, 39.6, 58.8, 110.8, 112.8, 123.8, 127.8, 128.8, 131.2, 133.6, 134.9, 135.2, 148.4, 196.0. HRMS (ESI-TOF) Calcd for $C_{16}H_{16}ClNO$, $[M+H]^+$ 274.0993; Found 274.0999.



2-((3-fluoro-4-methylphenyl)(methyl)amino)-1-phenylethanone 3eb

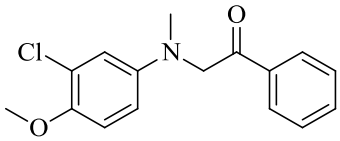
White solid, m.p. 134-135 ºC. ^{1}H NMR (500 MHz; $CDCl_3$): δ = 2.13 (s, 3H), 3.06 (s, 3H), 4.74 (s, 2H), 6.33 (t, J = 5.0 Hz, 2H), 6.96 (t, J = 8.5 Hz, 1H). 7.49 (t, J = 7.5 Hz, 2H), 7.61 (t, J = 7.0 Hz, 1H), 7.97 (d, J = 8.5 Hz, 2H). ^{13}C NMR (125 MHz; $CDCl_3$): δ = 13.4, 13.5, 39.6, 58.8, 99.3, 99.5, 107.6, 107.6, 112.3, 112.5, 127.7, 128.8, 131.5, 131.6, 133.6,

135.2, 148.9, 149.0, 161.1, 163.0, 196.1. HRMS (ESI-TOF) Calcd for $C_{16}H_{16}FNO$, $[M+H]^+$ 258.1289; Found 258.1294.



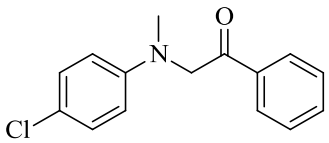
2-((2-chloro-4-methylphenyl)(methyl)amino)-1-phenylethanone 3fb

Yellow oil. ^{1}H NMR (500 MHz; $CDCl_3$): δ = 2.26 (s, 3H), 2.97 (s, 3H), 4.60 (s, 2H), 7.02 (dd, J_1 = 1.5 Hz, J_2 = 8.0 Hz, 1H), 7.16 (d, J = 8.5 Hz, 2H). 7.44 (t, J = 7.5 Hz, 2H), 7.56 (t, J = 7.5 Hz, 1H), 7.95 (s, 1H), 7.96 (d, J = 7.5 Hz, 1H). ^{13}C NMR (125 MHz; $CDCl_3$): δ = 20.4, 41.2, 61.3, 121.7, 127.4, 127.9, 127.9, 128.6, 130.9, 133.3, 135.6, 146.1, 196.8. HRMS (ESI-TOF) Calcd for $C_{16}H_{16}ClNO$, $[M+H]^+$ 274.0993; Found 274.0998.



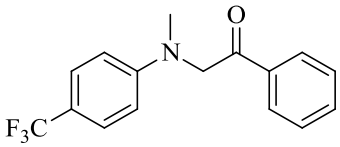
2-((3-chloro-4-methoxyphenyl)(methyl)amino)-1-phenylethanone 3gb

Yellow solid, m.p. 77-78 ºC. ^{1}H NMR (500 MHz; $CDCl_3$): δ = 3.05 (s, 3H), 3.81 (s, 3H), 4.72 (s, 2H), 6.53 (dd, J_1 = 3.0 Hz, J_2 = 9.0 Hz, 1H), 6.75 (d, J = 3.5 Hz, 1H). 6.82 (d, J = 9.0 Hz, 1H), 7.50 (t, J = 7.5 Hz, 2H), 7.62 (t, J = 7.0 Hz, 1H), 7.97 (d, J = 8.0 Hz, 2H). ^{13}C NMR (125 MHz; $CDCl_3$): δ = 39.9, 56.9, 59.3, 111.6, 114.0, 114.8, 123.4, 127.8, 128.8, 133.6, 135.3, 144.5, 147.1, 196.2. HRMS (ESI-TOF) Calcd for $C_{16}H_{16}ClNO_2$, $[M+H]^+$ 290.0942; Found 290.0953.



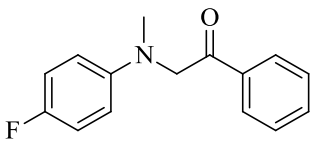
2-((4-chlorophenyl)(methyl)amino)-1-phenylethanone 3hb

Yellow solid, m.p. 99-100 ºC. ^{1}H NMR (500 MHz; $CDCl_3$): δ = 3.06 (s, 3H), 4.74 (s, 2H), 6.56 (d, J = 8.5 Hz, 2H). 7.12 (d, J = 8.5 Hz, 2H), 7.49 (t, J = 7.5 Hz, 2H), 7.60 (t, J = 7.0 Hz, 1H), 7.96 (d, J = 8.0 Hz, 2H). ^{13}C NMR (125 MHz; $CDCl_3$): δ = 39.7, 58.8, 113.3, 121.8, 127.7, 128.8, 128.9, 133.7, 135.2, 147.8, 195.9. HRMS (ESI-TOF) Calcd for $C_{15}H_{14}ClNO$, $[M+H]^+$ 260.0837; Found 260.0844.



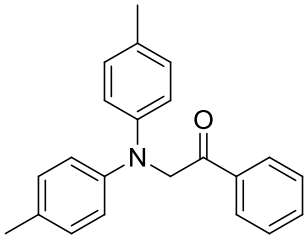
2-(methyl(4-(trifluoromethyl)phenyl)amino)-1-phenylethanone 3jb

White solid, m.p. 106-107 ºC. ^{1}H NMR (500 MHz; $CDCl_3$): δ = 3.14 (s, 3H), 4.84 (s, 2H), 6.64 (d, J = 9.0 Hz, 2H), 7.42 (d, J = 9.0 Hz, 2H). 7.51 (t, J = 7.5 Hz, 2H), 7.62 (d, J = 8.0 Hz, 1H), 7.98 (d, J = 7.5 Hz, 2H). ^{13}C NMR (125 MHz; $CDCl_3$): δ = 39.6, 58.4, 111.2, 118.2, 118.4, 123.9, 126.1, 126.4, 126.5, 126.5, 127.7, 128.9, 133.9, 135.0, 151.3, 195.3. HRMS (ESI-TOF) Calcd for $C_{16}H_{14}F_3NO$, $[M+H]^+$ 294.1100; Found 294.1103.



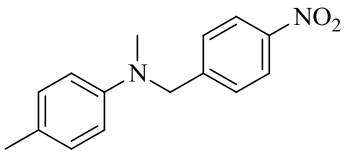
2-((4-fluorophenyl)(methyl)amino)-1-phenylethanone 3kb

White solid, m.p. 122 ºC. ^{1}H NMR (500 MHz; $CDCl_3$): δ = 3.08 (s, 3H), 4.75 (s, 2H), 6.61 (dd, J_1 = 4.0 Hz, J_2 = 9.0 Hz, 2H). 6.91 (t, J = 8.5 Hz, 2H), 7.50 (t, J = 7.5 Hz, 2H), 7.62 (t, J = 7.5 Hz, 1H), 7.98 (d, J = 7.5 Hz, 2H). ^{13}C NMR (125 MHz; $CDCl_3$): δ = 40.1, 59.4, 113.3, 113.4, 115.5, 115.6, 127.8, 128.8, 133.6, 135.3, 145.8, 154.7, 156.6, 196.4. HRMS (ESI-TOF) Calcd for $C_{15}H_{14}FNO$, $[M+H]^+$ 244.1132; Found 244.1138.



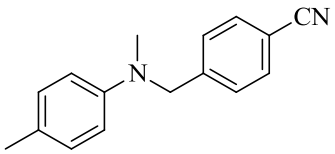
2-(di-*p*-tolylamino)-1-phenylethanone 3mb

Yellow solid, m.p. 122-123 ºC. ^{1}H NMR (400 MHz; $CDCl_3$): δ = 2.27 (s, 6H), 5.12 (s, 2H), 6.89 (dd, J_1 = 3.0 Hz, J_2 = 8.5 Hz, 4H). 7.04 (d, J = 10.0 Hz, 4H), 7.45-7.49 (m, 2H), 7.56-7.60 (m, 1H), 7.96-7.99 (m, 2H). ^{13}C NMR (100 MHz; $CDCl_3$): δ = 20.6, 58.9, 120.6, 127.8, 128.7, 129.8, 130.9, 133.4, 135.4, 145.5, 196.0. HRMS (ESI-TOF) Calcd for $C_{22}H_{22}NO$, $[M+H]^+$ 316.1696; Found 316.1704.



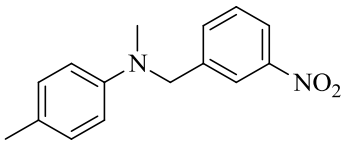
***N*,4-dimethyl-*N*-(4-nitrobenzyl)aniline 3ac**

Yellow solid, m.p. 55-56 ºC. ^{1}H NMR (500 MHz; $CDCl_3$): δ = 2.25 (s, 3H), 3.02 (s, 3H), 4.57 (s, 2H), 6.63 (d, J = 9.0 Hz, 2H). 7.04 (d, J = 8.0 Hz, 2H), 7.40 (d, J = 9.0 Hz, 2H), 8.17 (d, J = 8.5 Hz, 2H). ^{13}C NMR (125 MHz; $CDCl_3$): δ = 20.2, 39.1, 56.8, 112.7, 123.8, 126.6, 127.4, 129.8, 147.0, 147.0, 147.3. HRMS (ESI-TOF) Calcd for $C_{15}H_{16}N_2O_2$, $[M+H]^+$ 257.1285; Found 257.1289.



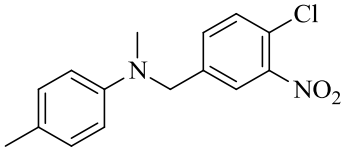
4-((methyl(*p*-tolyl)amino)methyl)benzonitrile 3ad

White solid, m.p. 79-80 ºC. ^{1}H NMR (500 MHz; $CDCl_3$): δ = 2.25 (s, 3H), 3.00 (s, 3H), 4.53 (s, 2H), 6.62 (d, J = 8.5 Hz, 2H). 7.04 (d, J = 9.0 Hz, 2H), 7.34 (d, J = 8.0 Hz, 2H), 7.60 (d, J = 8.0 Hz, 2H). ^{13}C NMR (125 MHz; $CDCl_3$): δ = 20.2, 39.0, 56.9, 110.6, 112.7, 118.9, 126.5, 127.4, 129.8, 132.4, 145.2, 147.1. HRMS (ESI-TOF) Calcd for $C_{16}H_{16}N_2$, $[M+H]^+$ 237.1386; Found 237.1388.



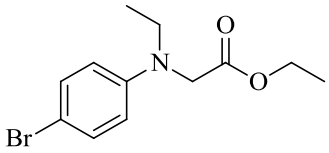
***N*,4-dimethyl-*N*-(3-nitrobenzyl)aniline 3ae**

Yellow oil. ^{1}H NMR (500 MHz; $CDCl_3$): δ = 2.25 (s, 3H), 3.01 (s, 3H), 4.55 (s, 2H), 6.66 (d, J = 9.0 Hz, 2H). 7.04 (d, J = 8.5 Hz, 2H), 7.47 (t, J = 7.5 Hz, 1H), 7.57 (dd, J_1 = 0.5 Hz, J_2 = 8.0 Hz, 1H), 8.11 (t, J = 8.5 Hz, 2H). ^{13}C NMR (125 MHz; $CDCl_3$): δ = 20.2, 39.0, 56.8, 112.9, 121.7, 122.0, 126.7, 129.5, 129.8, 132.9, 141.8, 147.2, 148.5. HRMS (ESI-TOF) Calcd for $C_{15}H_{16}N_2O_2$, $[M+H]^+$ 257.1285; Found 257.1288.



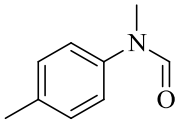
***N*-(4-chloro-3-nitrobenzyl)-*N*,4-dimethylaniline 3af**

Yellow oil. ^{1}H NMR (500 MHz; $CDCl_3$): δ = 2.26 (s, 3H), 2.99 (s, 3H), 4.49 (s, 2H), 6.63 (d, J = 8.5 Hz, 2H). 7.04 (d, J = 8.0 Hz, 2H), 7.39 (d, J = 8.0 Hz, 1H), 7.47 (d, J = 8.5 Hz, 1H), 7.75 (s, 1H). ^{13}C NMR (125 MHz; $CDCl_3$): δ = 20.2, 39.0, 56.3, 113.0, 123.7, 125.1, 127.0, 129.8, 131.3, 131.9, 140.3, 147.0. HRMS (ESI-TOF) Calcd for $C_{15}H_{15}ClN_2O_2$, $[M+H]^+$ 291.0895; Found 291.0899.



ethyl 2-((4-bromophenyl)(ethyl)amino)acetate 3la

Colorless oil. ^{1}H NMR (500 MHz; $CDCl_3$): δ = 1.20 (t, J = 7.0 Hz, 3H), 1.26 (t, J = 7.0 Hz, 3H), 3.43 (q, J = 7.0 Hz, 2H), 3.98 (s, 2H), 4.19 (q, J = 7.0 Hz, 2H), 6.51 (dd, J_1 = 2.0 Hz, J_2 = 7.0 Hz,, 2H), 7.28 (dd, J_1 = 2.0 Hz, J_2 = 7.0 Hz,, 2H). ^{13}C NMR (125 MHz; $CDCl_3$): δ = 12.2, 14.1, 46.2, 52.2, 61.0, 108.7, 113.6, 131.8, 146.7, 170.9. HRMS (ESI-TOF) Calcd for $C_{12}H_{16}BrNO_2$, $[M+H]^+$ 286.0437; Found 286.0432.

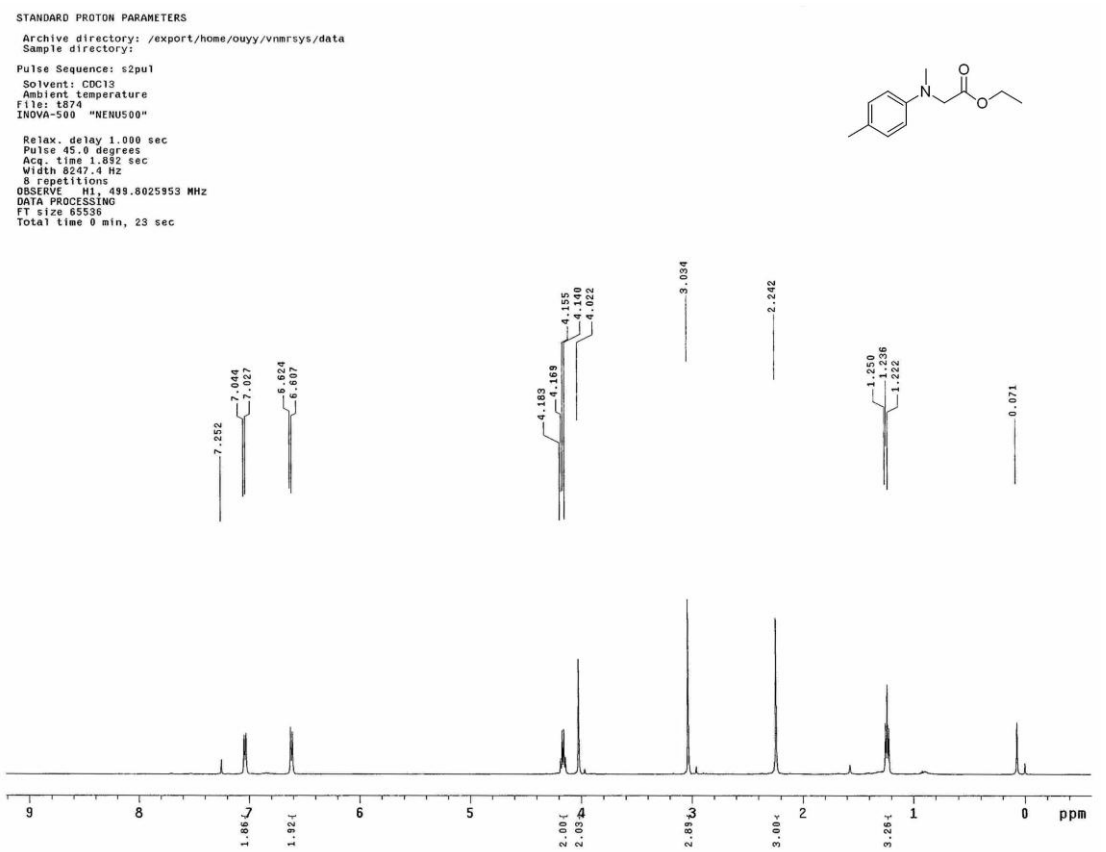


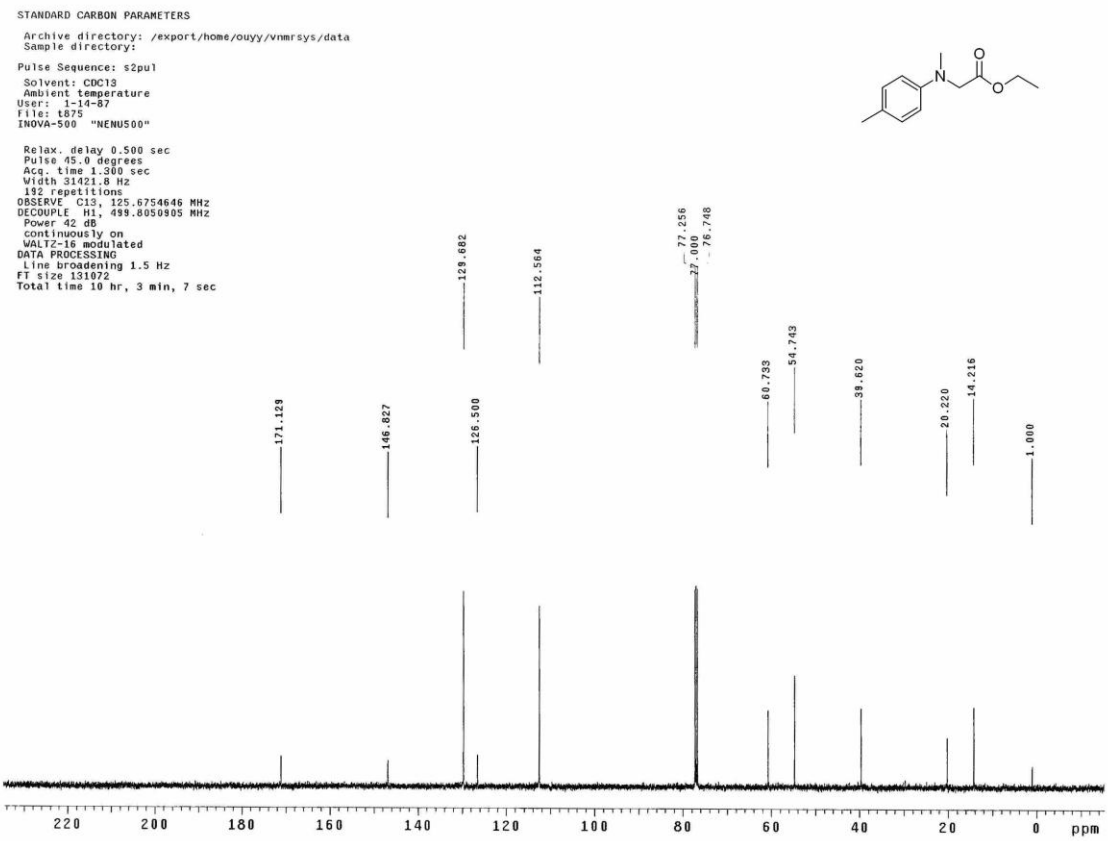
***N*-methyl-*N*-(*p*-tolyl)formamide 4**

Colorless oil. ^{1}H NMR (500 MHz; $CDCl_3$): δ = 2.37 (s, 3H), 3.30 (s, 3H), 7.06 (d, J = 8.5 Hz, 2H), 7.21 (d, J = 8.0 Hz, 2H), 8.42 (s, 1H). ^{13}C NMR (125 MHz; $CDCl_3$): δ = 20.8, 32.2, 122.5, 130.1, 136.3, 139.6, 162.3. HRMS (ESI-TOF) Calcd for $C_9H_{11}NO$, $[M+H]^+$ 150.0913; Found 150.0918.

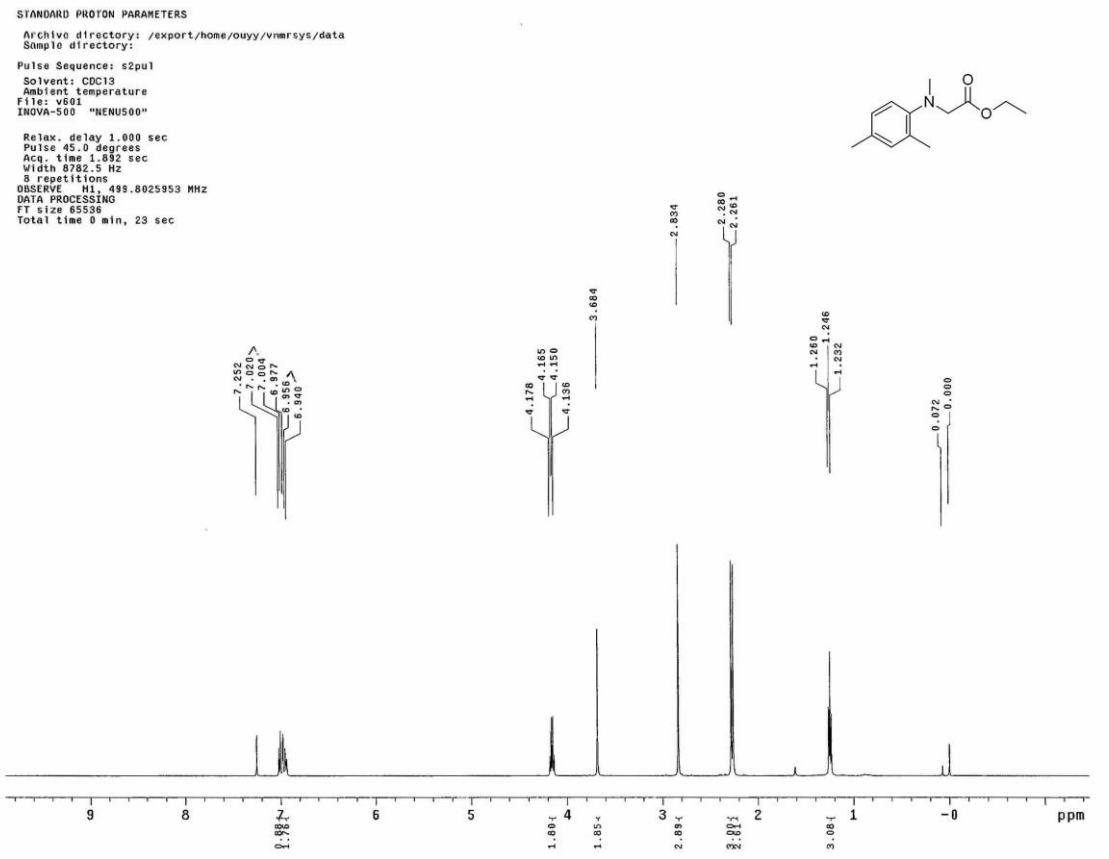
V. ^{1}H NMR and ^{13}C NMR spectra for the products

Product 3aa





Product 3ba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Ambient temperature
File: v601
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 8782.5 Hz
8 repetitions
OBSERVE H1, 499.8025953 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
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7.020
7.004
6.977
6.956
6.940
4.178
4.165
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1.246
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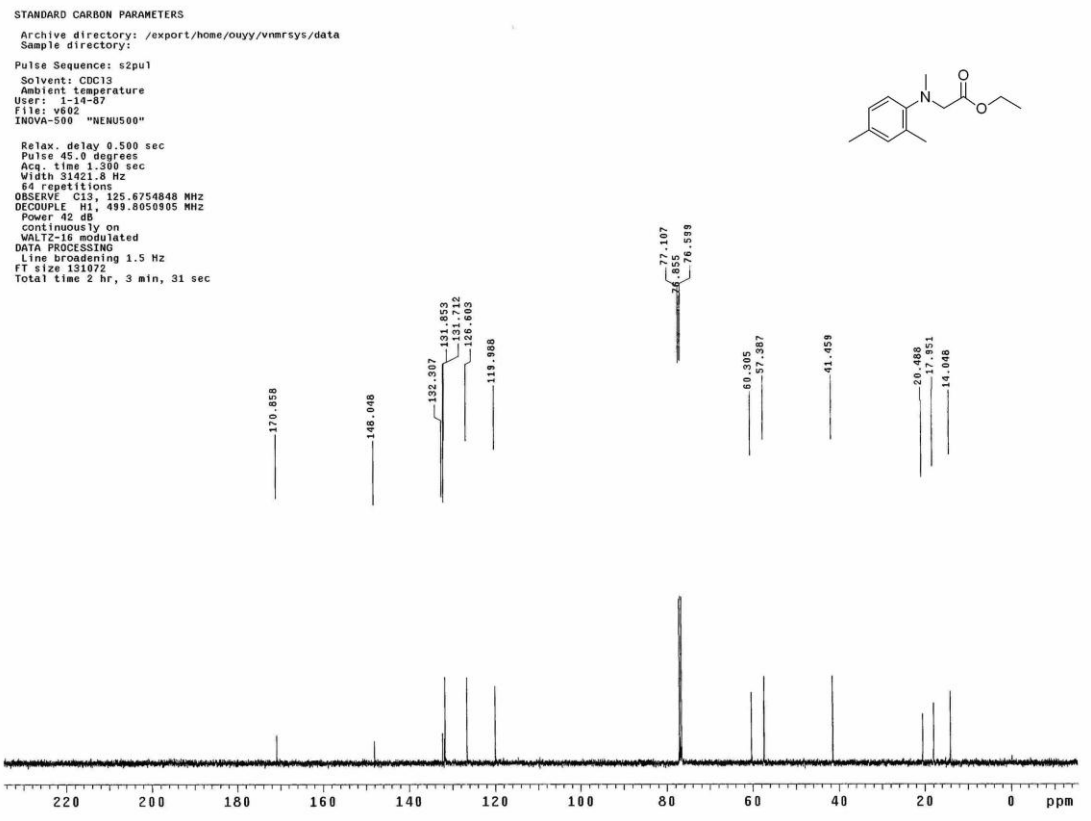
0.000
ppm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Sample directory:
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Solvent: CDCl3
Ambient temperature
User: 1-14-87
File: v602
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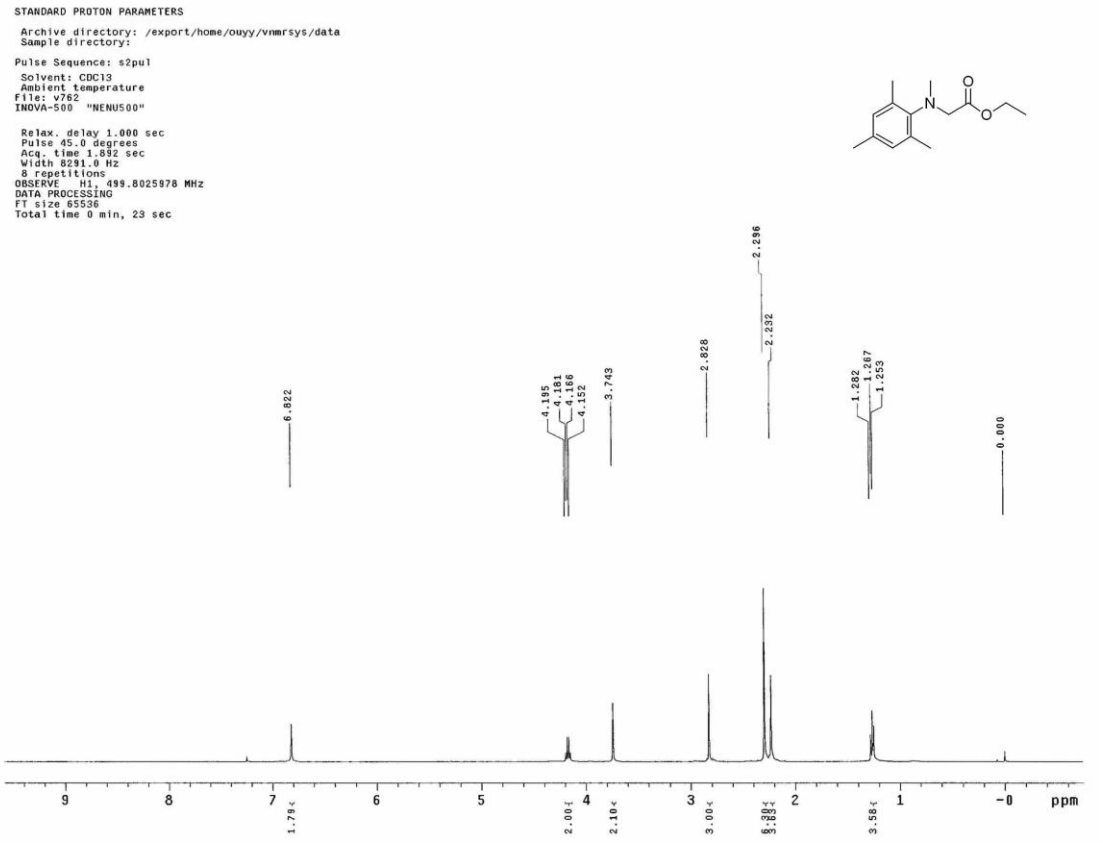
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
64 repetitions
OBSERVE C13, 125.6754848 MHz
DECOUPLE H1, 499.8050905 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 2 hr, 3 min, 31 sec
170.858
148.048
132.307
131.853
131.712
126.603
119.988
77.107
76.855
76.599
60.305
57.387
41.459
20.488
17.951
14.048
ppm

Product 3ca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Sample directory:
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Solvent: CDCl3
Ambient temperature
File: v762
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 8291.0 Hz
8 repetitions
OBSERVE H1, 499.8025978 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
6.822
4.195
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4.152
3.743
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2.232
1.282
1.267
1.253
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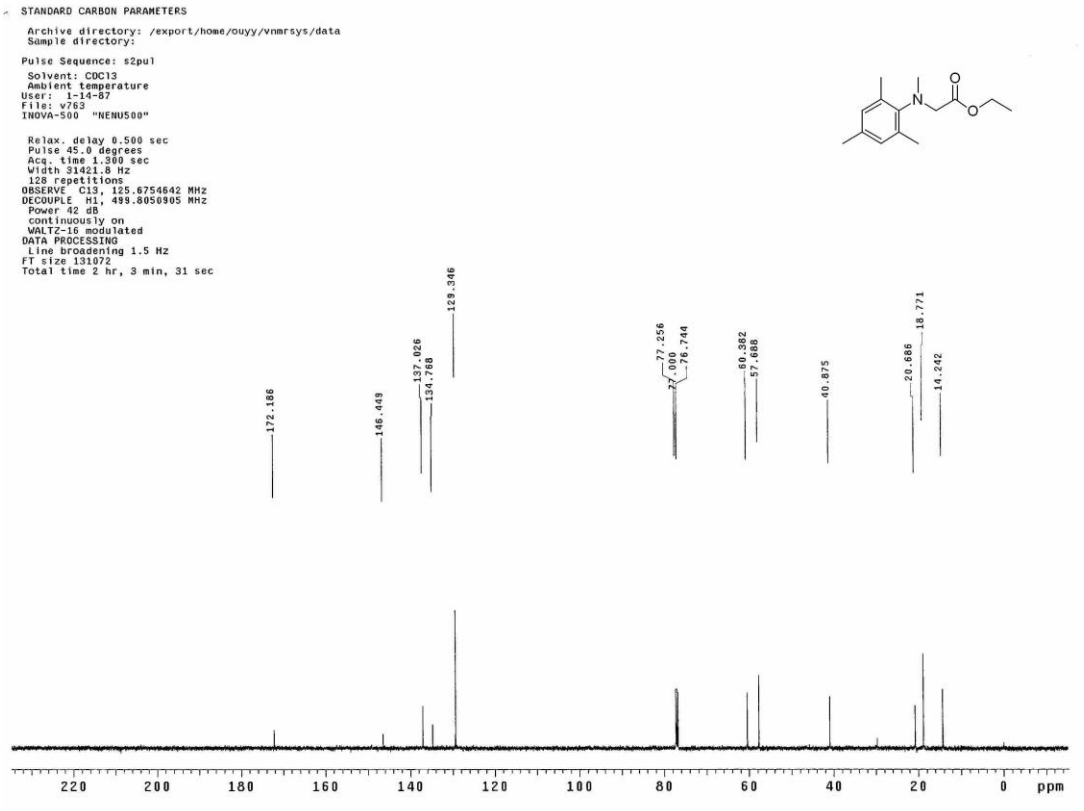
ppm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Sample directory:
Pulse Sequence: s2pul
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Ambient temperature
User: 1-14-87
File: v763
INOVA-500 "NENU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
128 repetitions
OBSERVE C13, 125.6754642 MHz
DECOUPLE H1, 499.8050905 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 2 hr, 3 min, 31 sec
172.186
146.449
137.026
134.768
129.346
77.256
77.000
76.744
60.882
57.688
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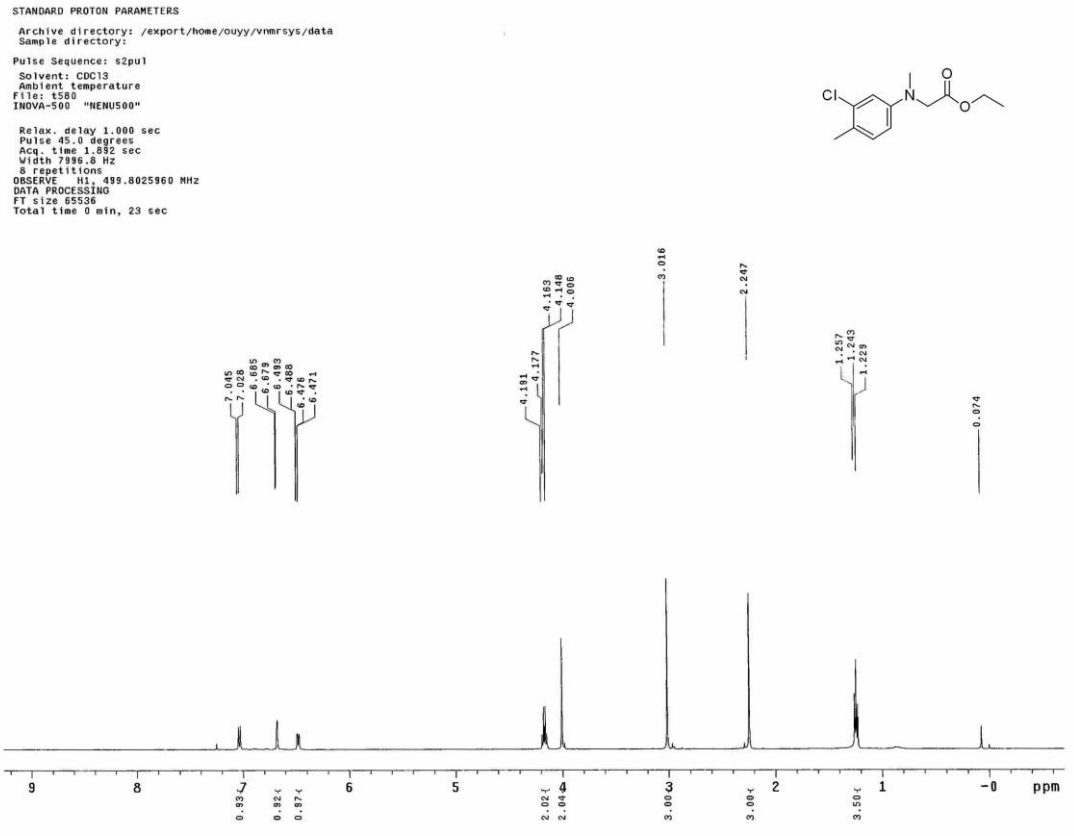
20.686
18.771
14.242
ppm

Product 3da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Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: t580
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 7996.8 Hz
8 repetitions
OBSERVE H1, 499.8025960 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
Cl
N
O
O
7.045
7.028
6.685
6.679
6.493
6.488
6.476
6.471
4.191
4.177
4.163
4.148
4.006
3.016
2.247
1.257
1.243
1.229
0.074
9
8
7
6
5
4
3
2
1
-0
ppm
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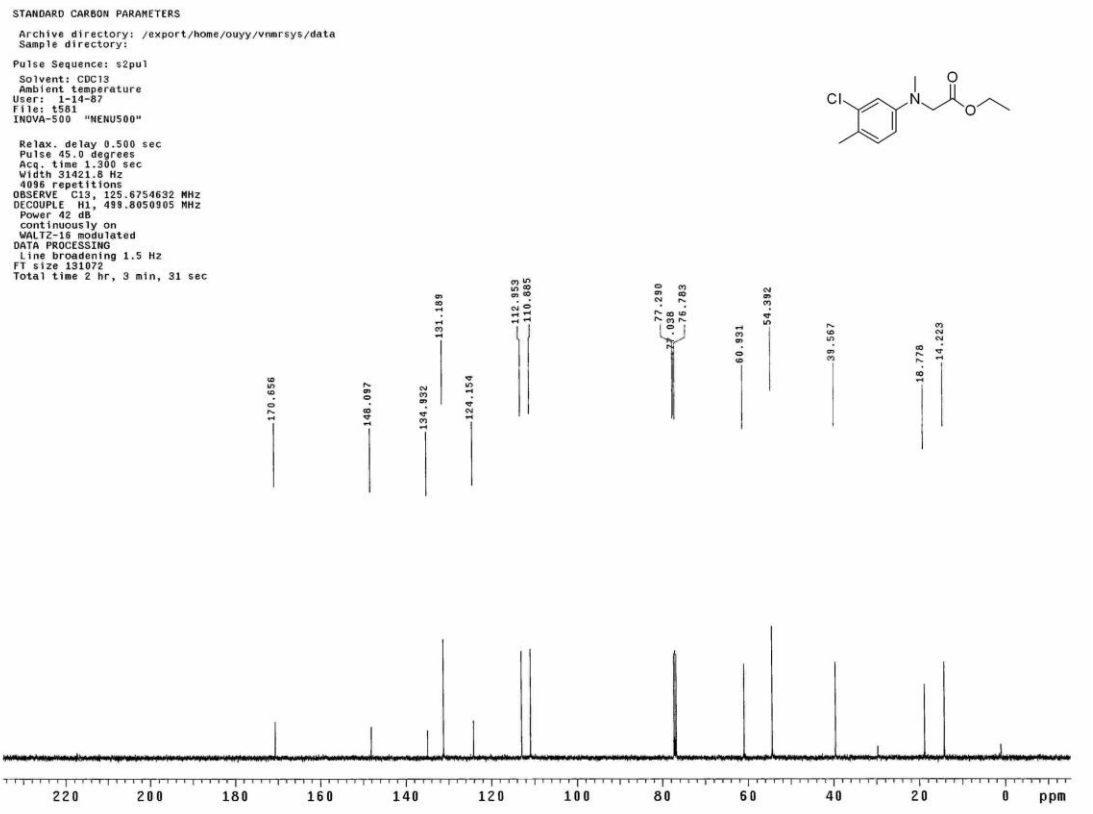
0.82
0.97
2.02
2.04
3.00
3.00
3.50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Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
File: t581
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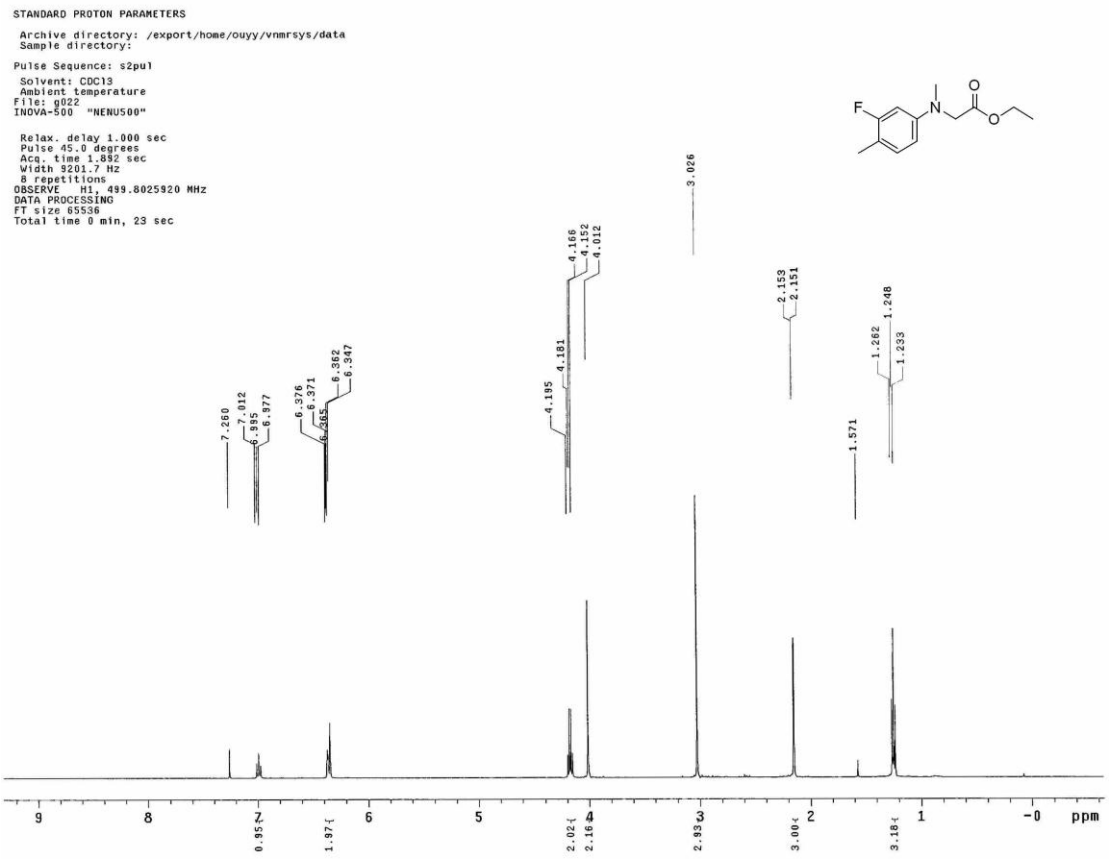
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
4096 repetitions
OBSERVE C13, 125.6754632 MHz
DECOUPLE H1, 499.8050905 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 2 hr, 3 min, 31 sec
Cl
N
O
O
170.656
148.097
134.932
131.189
124.154
112.953
110.885
77.290
77.038
76.783
60.931
54.392
39.567
18.778
14.223
220
200
180
160
140
120
100
80
60
40
20
0
ppm

Product 3ea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Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: g022
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 9201.7 Hz
8 repetitions
OBSERVE H1, 499.8025920 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
7.260
7.012
6.995
6.977
6.376
6.371
6.362
6.347
4.195
4.181
4.166
4.152
4.012
3.026
2.153
2.151
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1.248
1.233
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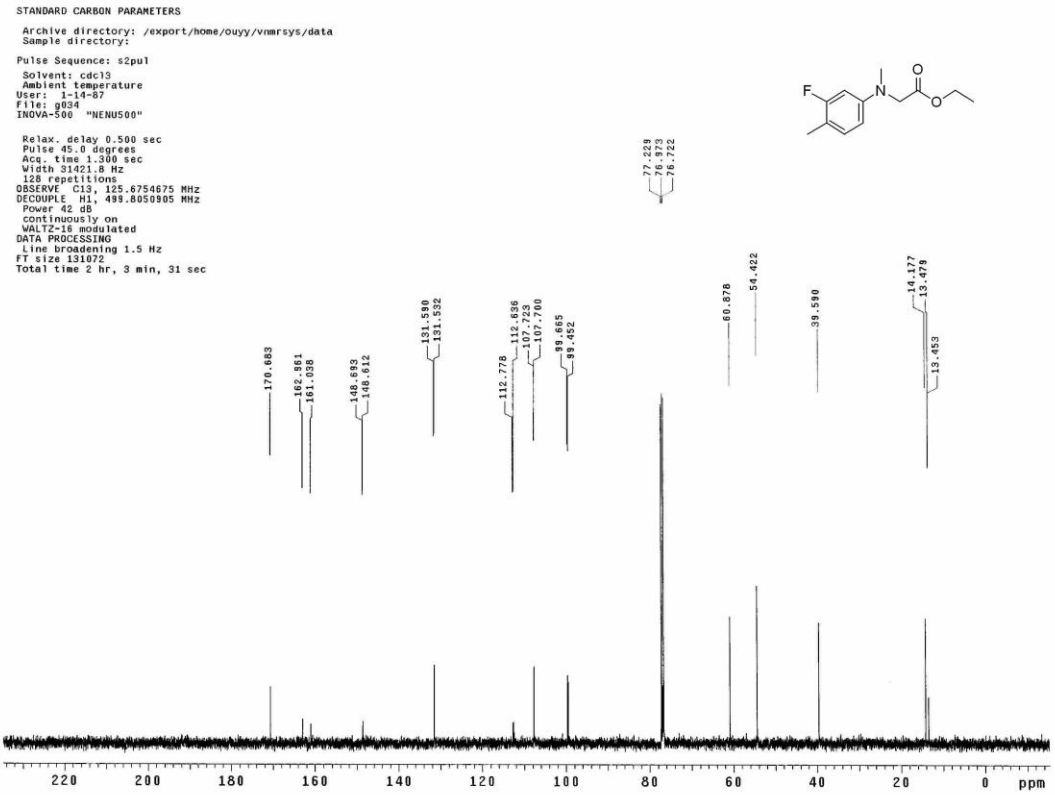
2.16
2.93
3.00
3.18
ppm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Sample directory:
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Solvent: cdcl3
Ambient temperature
User: 1-14-87
File: g034
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Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
128 repetitions
OBSERVE C13, 125.6754675 MHz
DECOUPLE H1, 499.8050905 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 2 hr, 3 min, 31 sec
170.683
162.861
161.038
148.693
148.612
131.590
131.532
112.778
112.636
107.723
107.700
99.665
99.452
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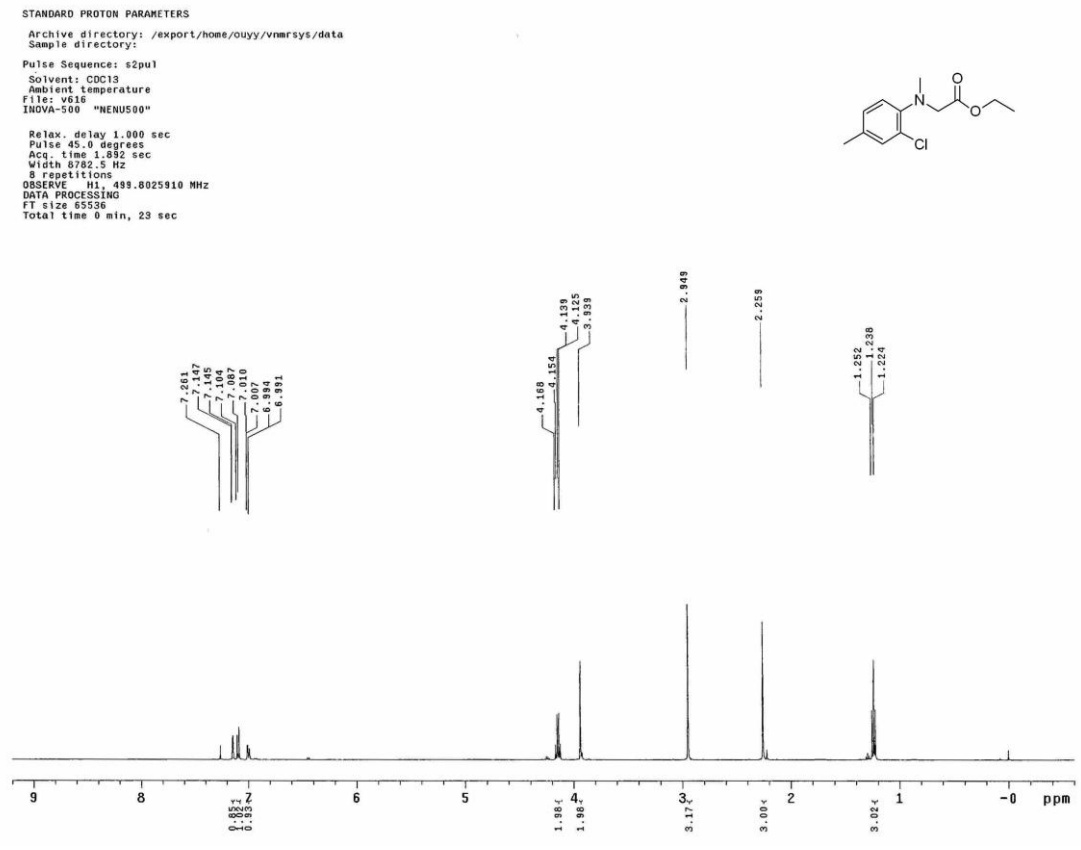
76.973
76.722
60.878
54.422
39.590
14.177
13.479
13.453
ppm

Product 3fa


STANDARD PROTON PARAMETERS
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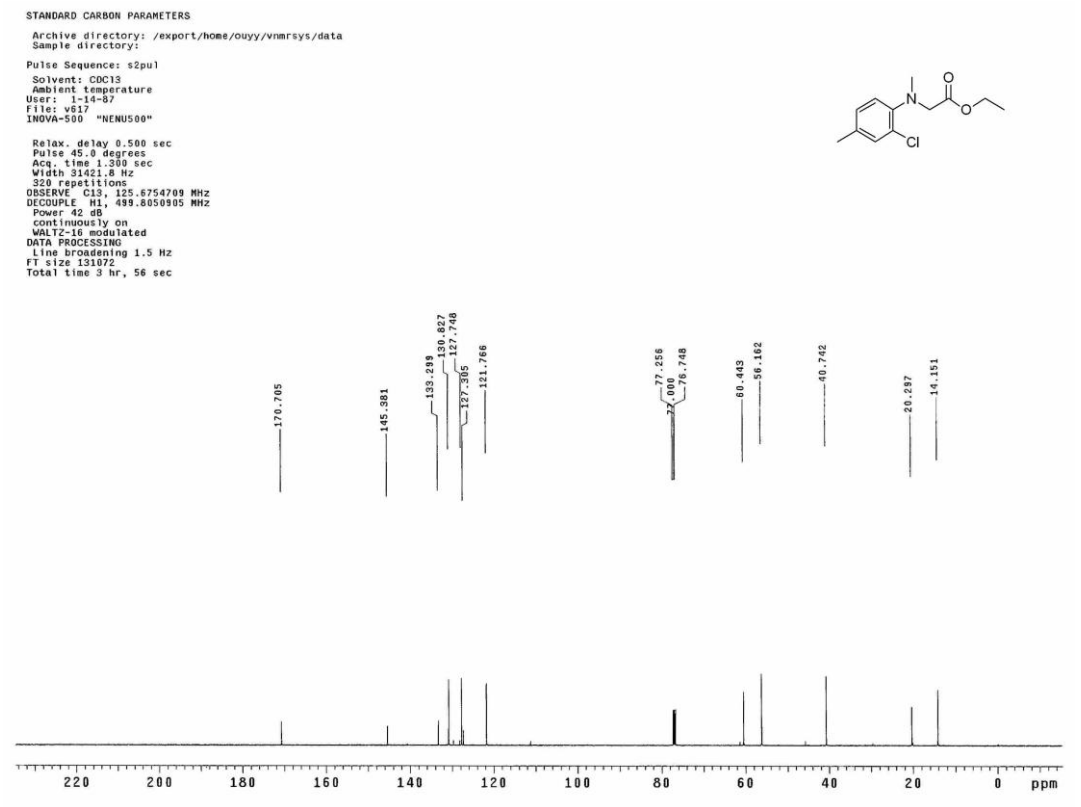
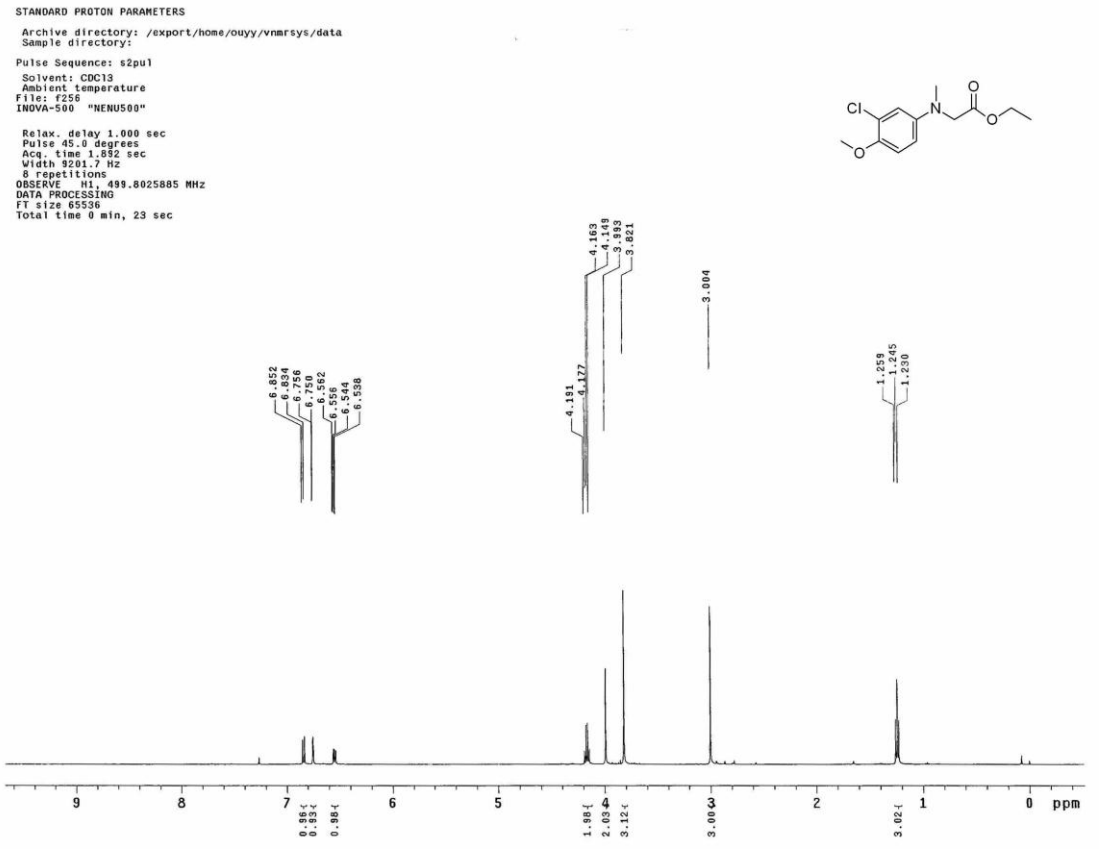
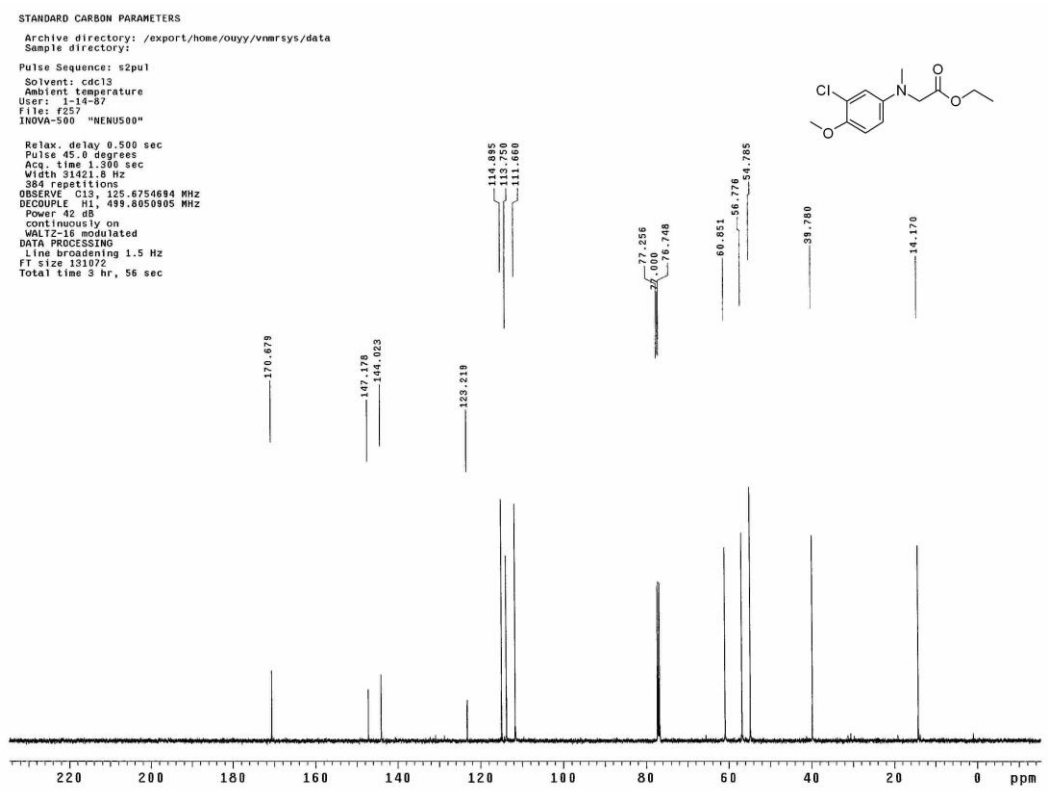
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: v616
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 8782.5 Hz
8 repetitions
OBSERVE H1, 499.8025910 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
7.261
7.147
7.145
7.104
7.087
7.010
7.007
6.994
6.991
4.168
4.154
4.139
4.125
3.939
2.949
2.259
1.252
1.238
1.224
Cl
9
8
7
6
5
4
3
2
1
-0
ppm

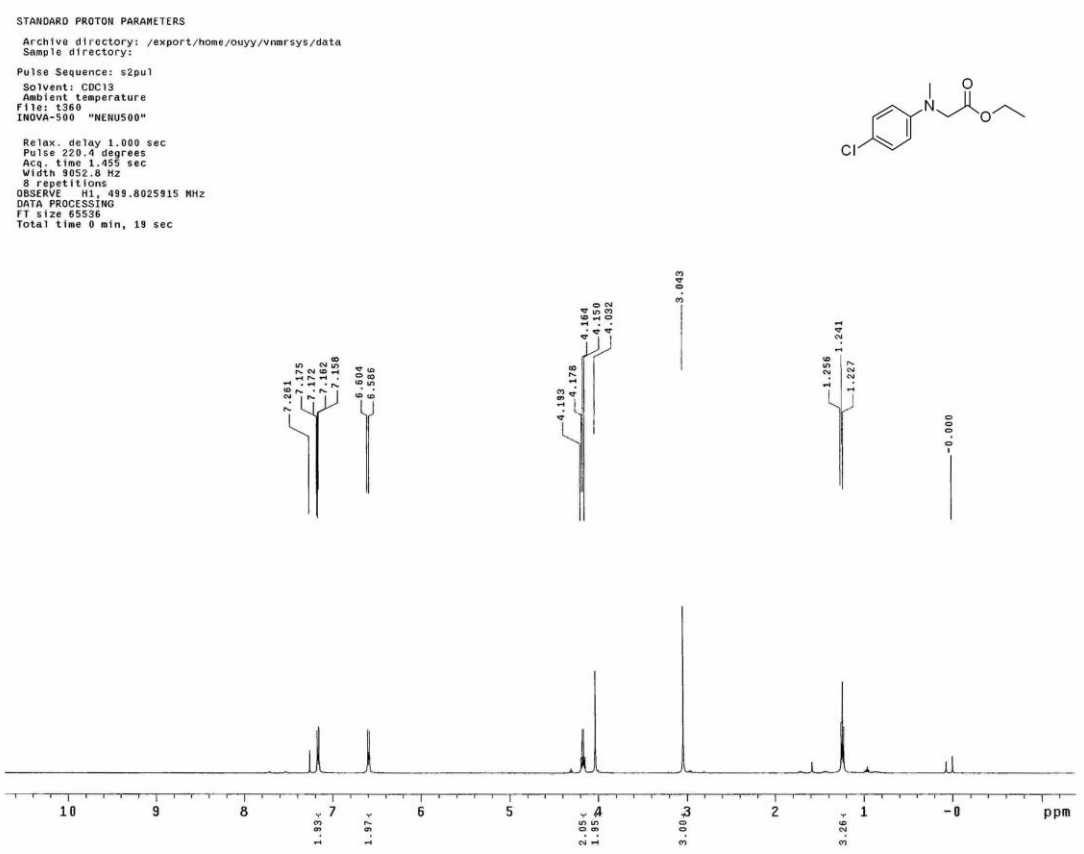

STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
File: v617
INOVA-500 "NENU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
320 repetitions
OBSERVE C13, 125.6754709 MHz
DECOUPLE H1, 499.8050905 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 3 hr, 56 sec
170.705
145.381
133.209
130.827
127.748
127.305
121.766
77.256
77.000
76.748
60.443
56.162
40.742
20.297
14.151
Cl
220
200
180
160
140
120
100
80
60
40
20
0
ppm

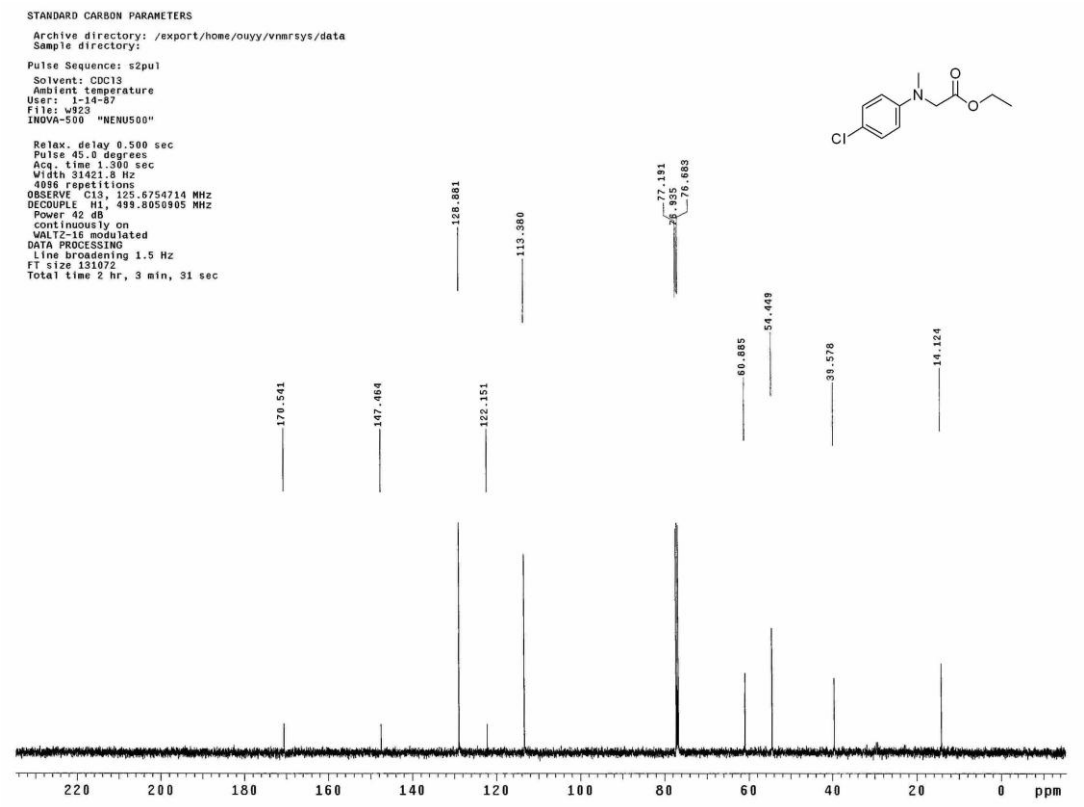
Product 3ga


STANDARD PROTON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: f256
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.882 sec
Width 9201.7 Hz
8 repetitions
OBSERVE H1, 499.8025885 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
6.852
6.834
6.756
6.750
6.562
6.556
6.544
6.538
4.191
4.177
4.163
4.149
3.893
3.821
3.004
1.259
1.245
1.230
0.88
0.93
0.98
1.98
2.03
3.12
3.00
3.02
ppm

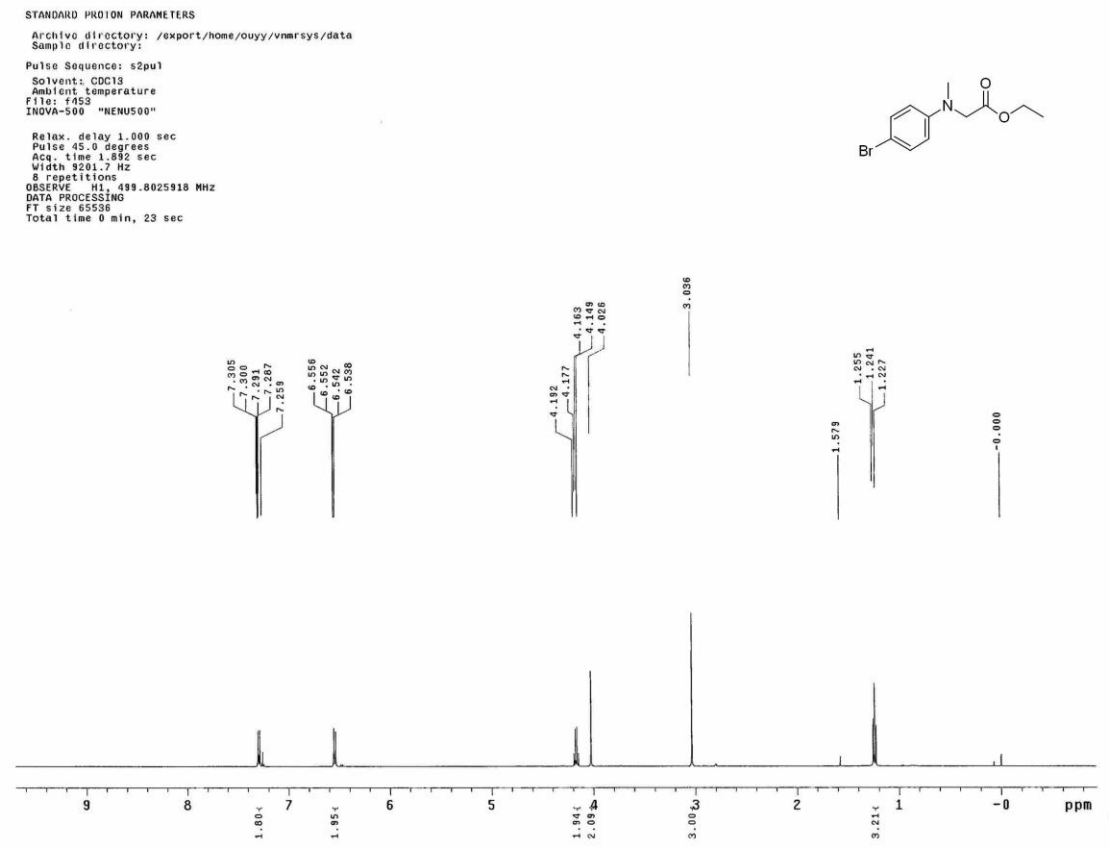

STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
User: 1-14-87
File: f257
INOVA-500 "NENU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
384 repetitions
OBSERVE C13, 125.6754694 MHz
DECOUPLE H1, 499.8050905 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 3 hr, 56 sec
170.679
147.178
144.023
123.219
114.895
113.750
111.660
77.256
77.000
76.748
60.851
56.776
54.765
39.760
14.170
220
200
180
160
140
120
100
80
60
40
20
0
ppm

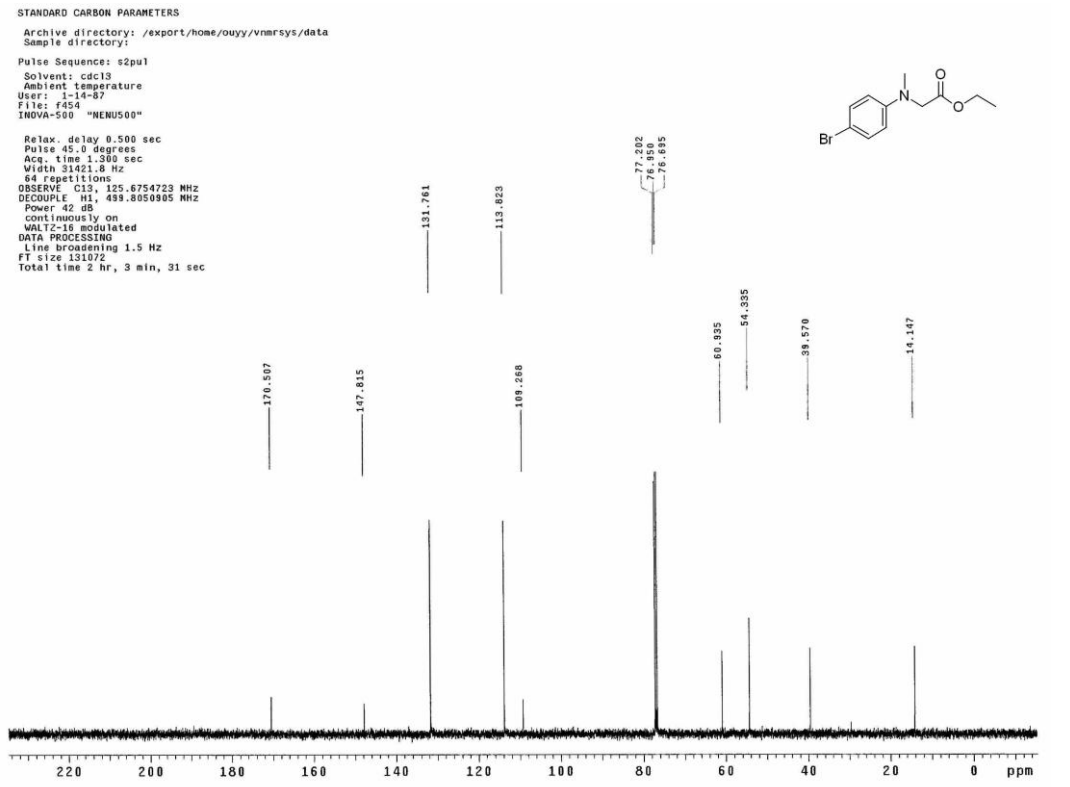
Product 3ha


STANDARD PROTON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: t360
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 220.4 degrees
Acq. time 1.455 sec
Width 9052.8 Hz
8 repetitions
OBSERVE H1, 499.8025915 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 19 sec
7.261
7.175
7.172
7.162
7.158
6.604
6.586
4.193
4.178
4.164
4.150
4.032
3.043
1.256
1.241
1.227
-0.000
Cl
1.83
1.97
2.05
1.95
3.00
3.26
10
9
8
7
6
5
4
3
2
1
-0
ppm

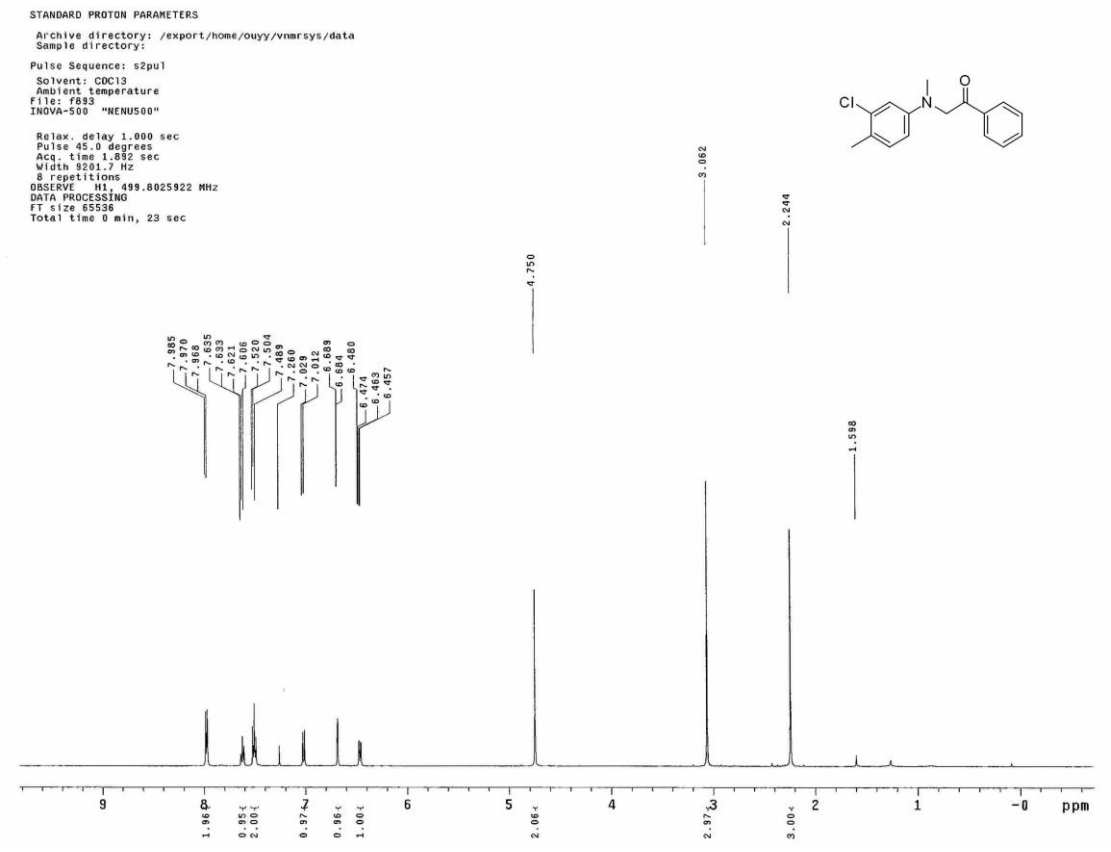

STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
File: W923
INOVA-500 "NENU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
4096 repetitions
OBSERVE C13, 125.6754714 MHz
DECOUPLE H1, 499.8050905 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 2 hr, 3 min, 31 sec
170.541
147.464
128.881
122.151
113.380
77.191
76.683
60.885
54.449
39.578
14.124
Cl
220
200
180
160
140
120
100
80
60
40
20
0
ppm

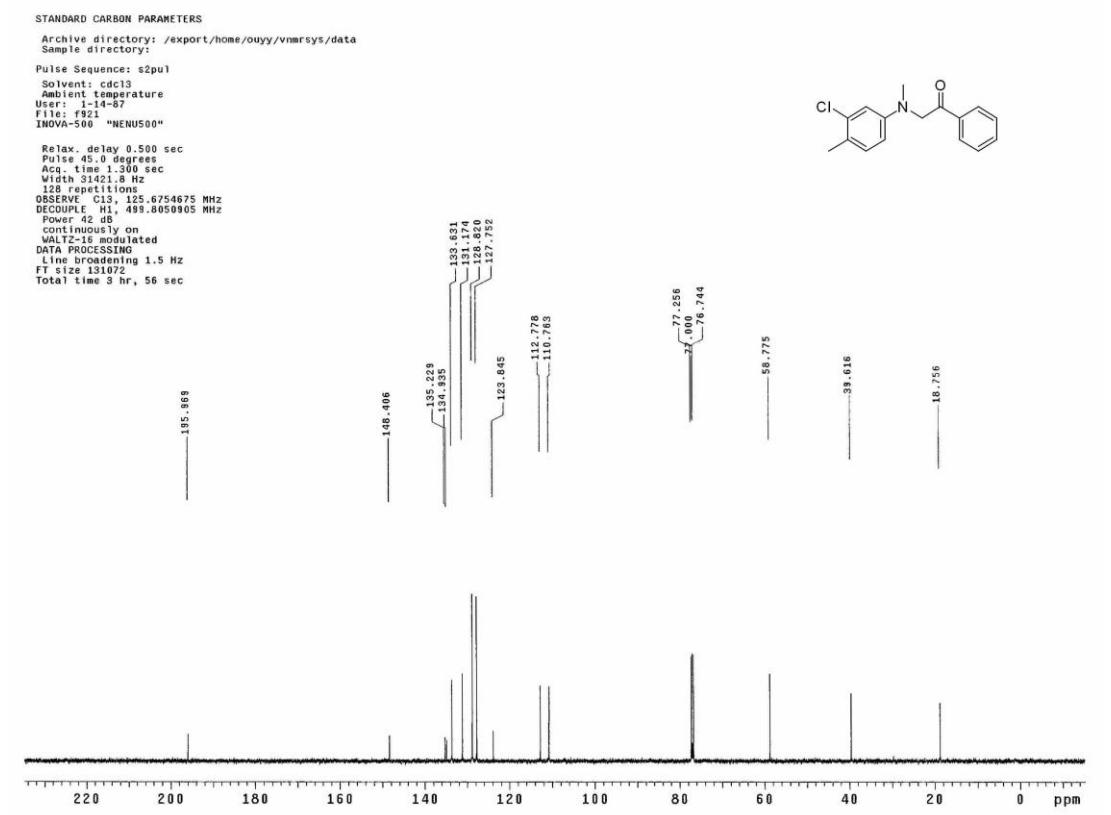
Product 3ia


STANDARD PROTON PARAMETERS
Archivo directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: f453
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 9201.7 Hz
8 repetitions
OBSERVE H1, 499.8025918 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
Br
7.305
7.300
7.291
7.287
7.259
6.556
6.552
6.542
6.538
4.192
4.177
4.163
4.149
4.026
3.036
1.579
1.255
1.241
1.227
-0.000
ppm

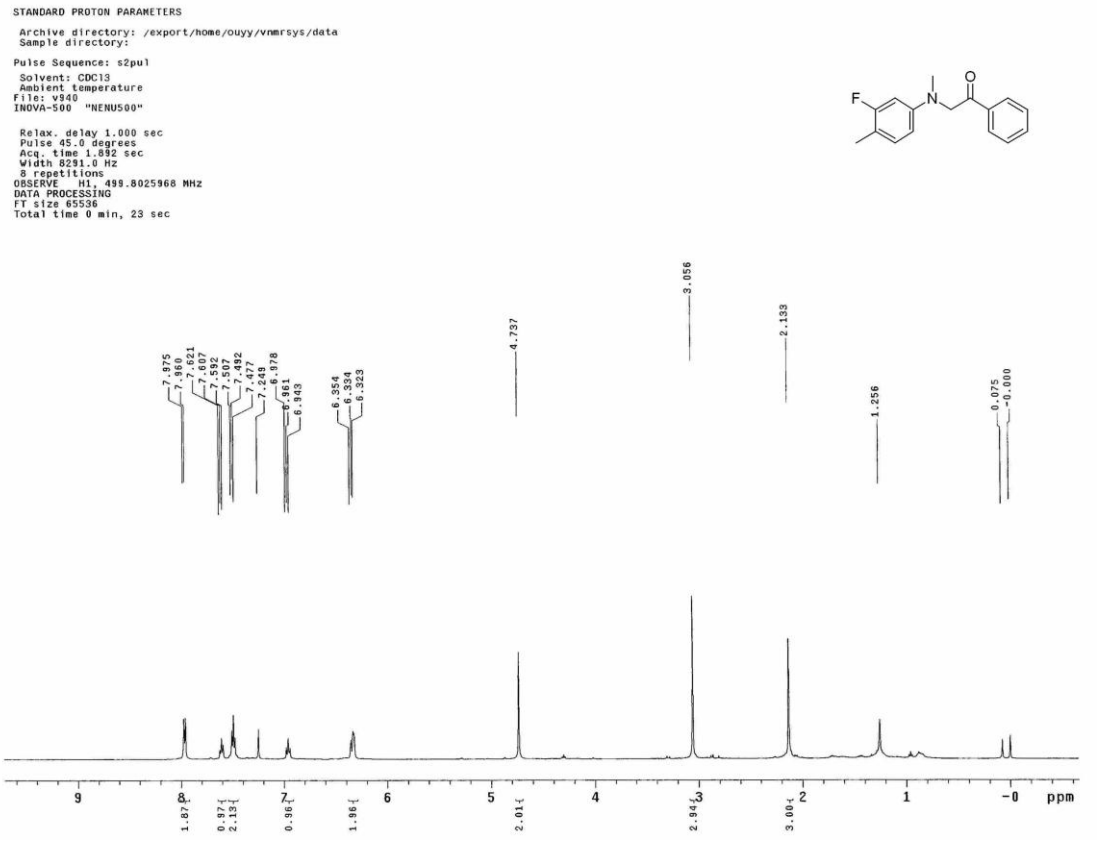

STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
User: 1-14-87
File: f454
INOVA-500 "NENU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
64 repetitions
OBSERVE C13, 125.6754723 MHz
DECOUPLE H1, 499.8050905 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 2 hr, 3 min, 31 sec
Br
170.507
147.815
131.761
113.823
109.268
77.202
76.950
76.695
60.935
54.335
39.570
14.147
ppm

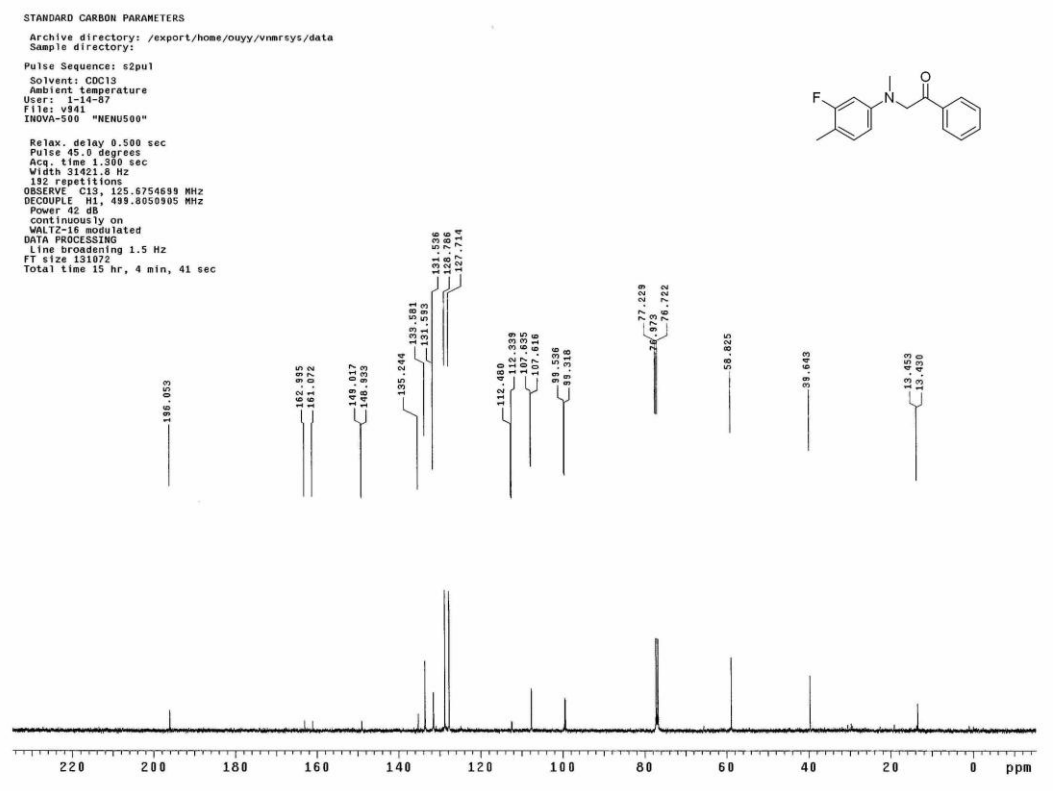
Product 3db


STANDARD PROTON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: f893
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 8201.7 Hz
8 repetitions
OBSERVE H1, 499.8025922 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
Cl
N
O
3.062
2.244
4.750
1.598
7.985
7.970
7.968
7.635
7.633
7.621
7.606
7.520
7.304
7.289
7.260
7.029
7.012
6.689
6.684
6.480
6.474
6.463
6.457
9
8
7
6
5
4
3
2
1
-0
ppm
1.96
0.95
2.00
0.97
0.96
1.00
2.06
2.97
3.00

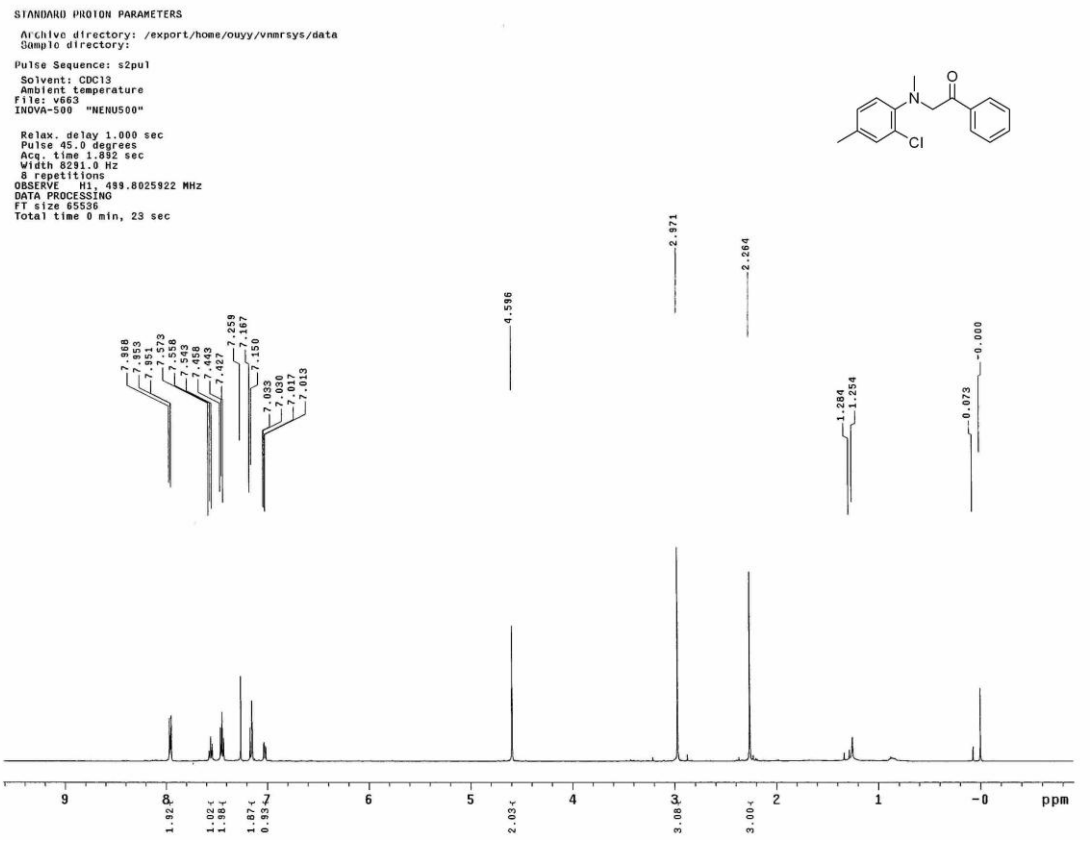

STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
User: 1-14-87
File: f921
INOVA-500 "NENU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
128 repetitions
OBSERVE C13, 125.6754675 MHz
DECOUPLE H1, 499.8050905 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 3 hr, 56 sec
Cl
N
O
195.869
148.406
135.229
134.935
133.631
131.174
128.820
127.752
123.645
112.778
110.763
77.256
77.000
76.744
58.775
39.616
18.756
220
200
180
160
140
120
100
80
60
40
20
0
ppm

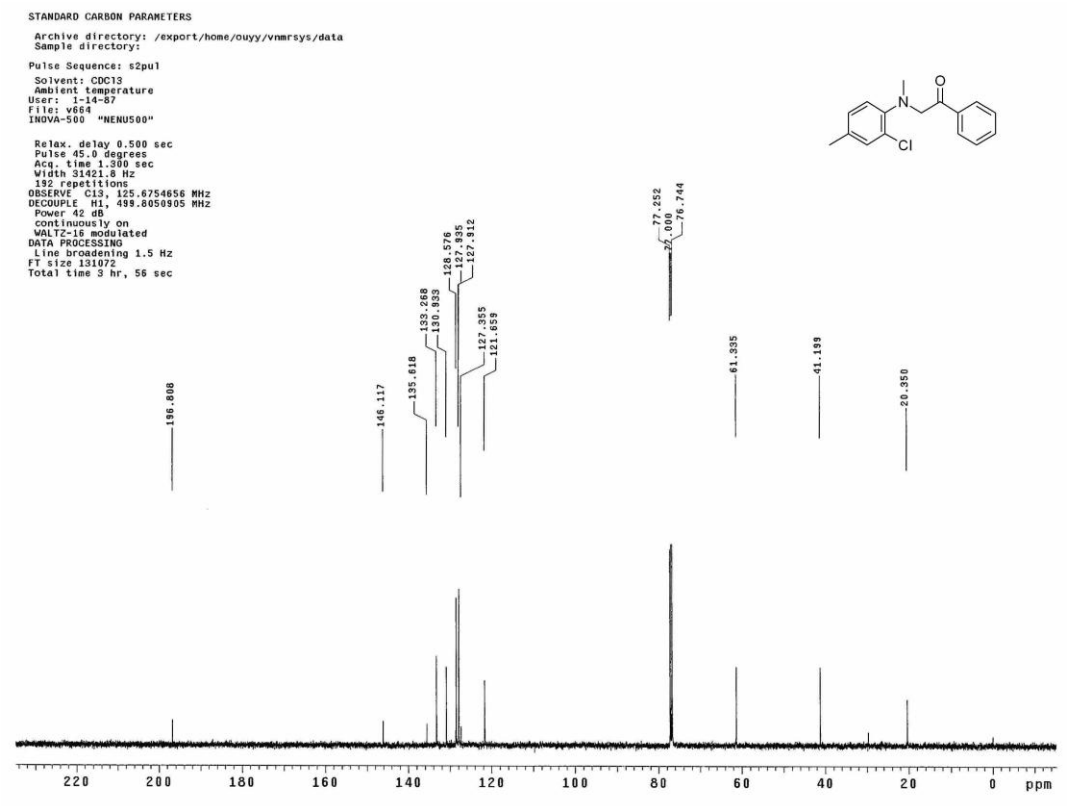
Product 3eb


STANDARD PROTON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: v940
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 8291.0 Hz
8 repetitions
OBSERVE H1, 499.8025968 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
F
N
O
7.975
7.960
7.621
7.607
7.592
7.507
7.492
7.477
7.249
6.978
6.961
6.943
6.354
6.334
6.323
4.737
3.056
2.133
1.256
0.075
0.000
9
8
7
6
5
4
3
2
1
-0
ppm
1.87
0.97
2.13
0.96
1.96
2.01
2.94
3.00

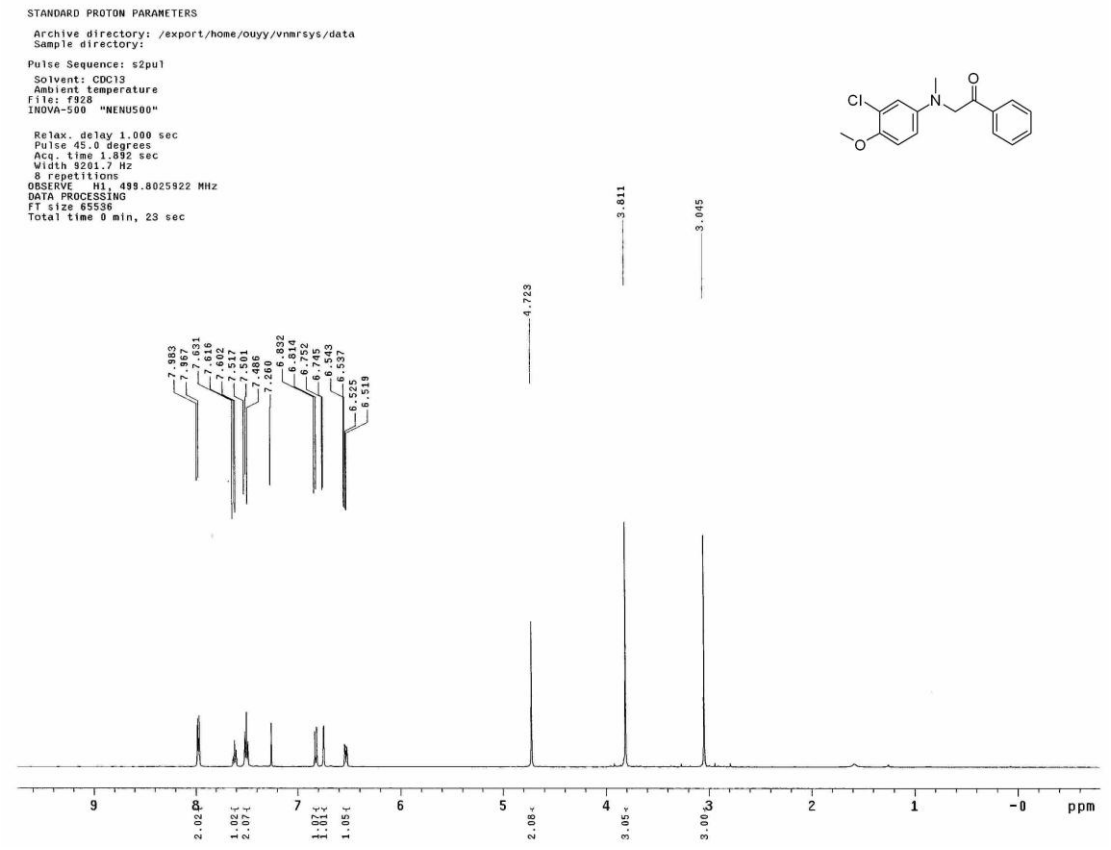

STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
File: v941
INOVA-500 "NENU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
192 repetitions
OBSERVE C13, 125.6754699 MHz
DECOUPLE H1, 499.8050305 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 15 hr, 4 min, 41 sec
F
N
O
186.053
162.995
161.072
149.017
148.933
135.244
133.581
131.593
131.536
128.766
127.714
112.460
112.339
107.635
107.616
89.536
89.318
77.229
76.975
76.722
58.825
39.643
13.453
13.430
220
200
180
160
140
120
100
80
60
40
20
0
ppm

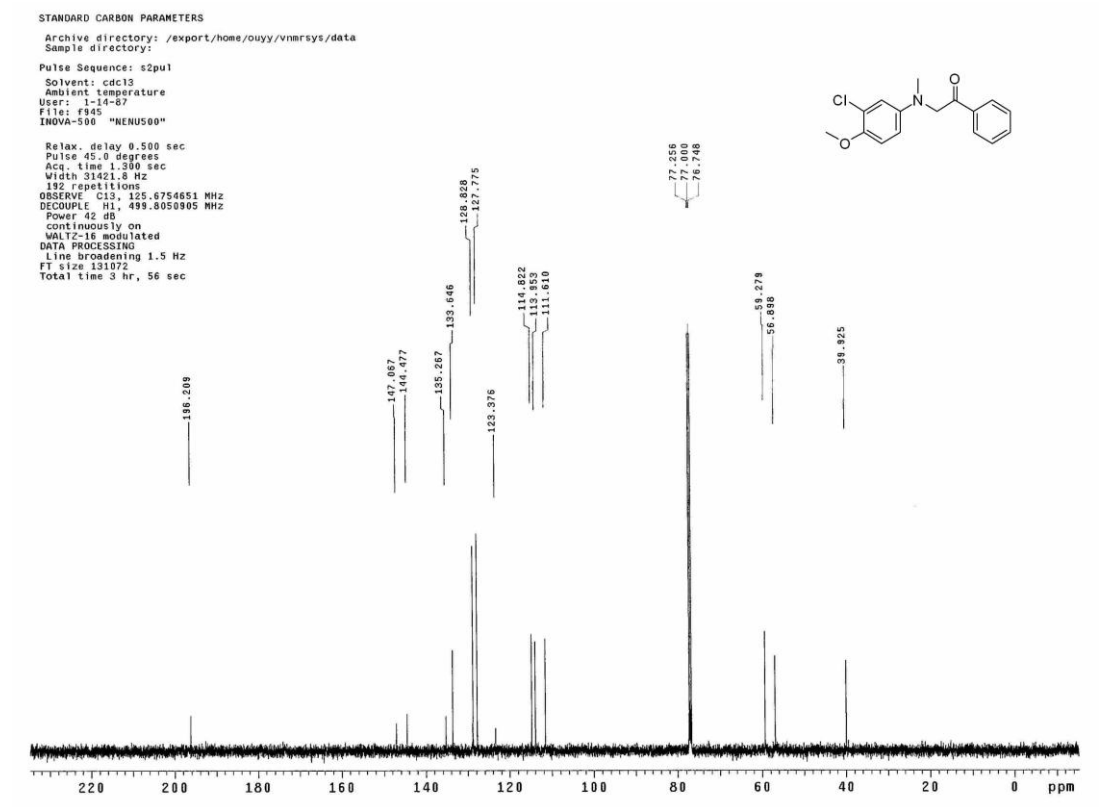
Product 3fb


STANDARD PROTON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: v663
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 8291.0 Hz
8 repetitions
OBSERVE H1, 499.8025922 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
Cl
7.968
7.953
7.951
7.573
7.556
7.543
7.458
7.443
7.427
7.259
7.167
7.150
7.033
7.030
7.017
7.013
4.586
2.971
2.264
1.284
1.254
0.073
-0.000
ppm

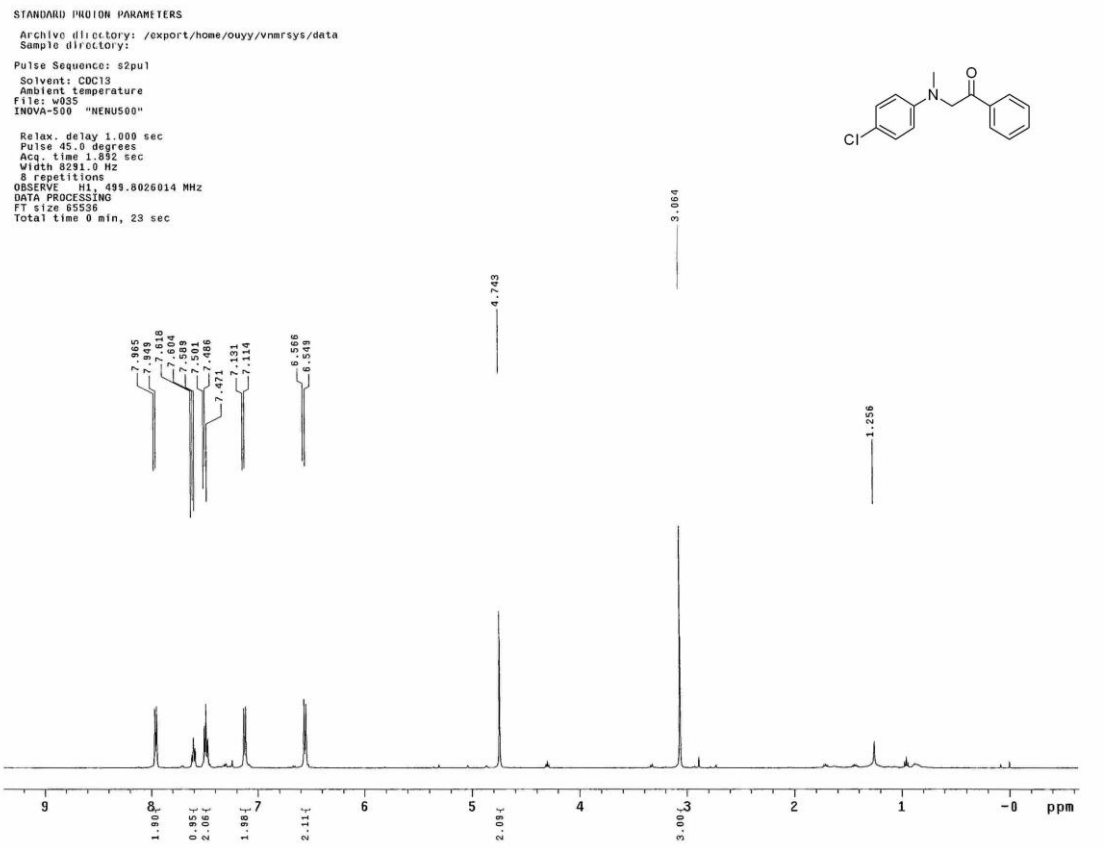

STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
File: v664
INOVA-500 "NENU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
192 repetitions
OBSERVE C13, 125.6754656 MHz
DECOUPLE H1, 499.8050905 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 3 hr, 56 sec
Cl
196.808
146.117
135.618
133.268
130.933
128.576
127.935
127.912
127.355
121.659
77.252
77.000
76.744
61.335
41.199
20.350
ppm

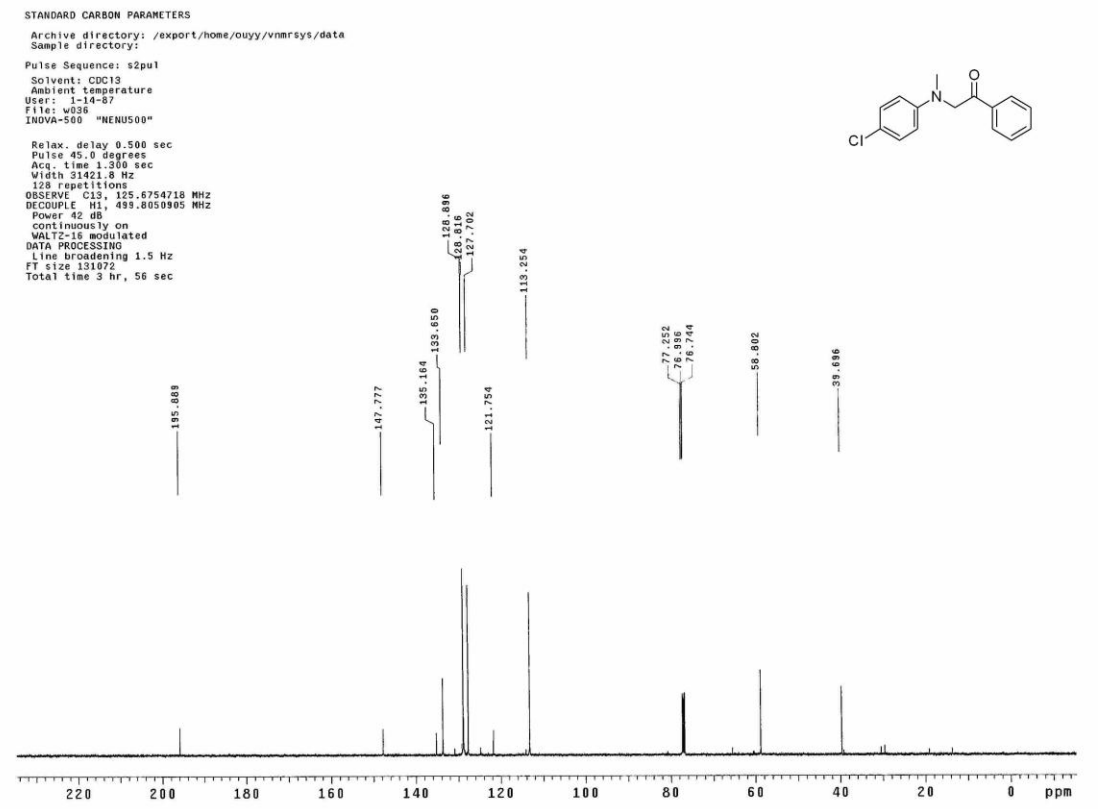
Product 3gb


STANDARD PROTON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: f928
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 9201.7 Hz
8 repetitions
OBSERVE H1, 499.8025922 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
Cl
N
O
O
7.983
7.967
7.631
7.616
7.602
7.517
7.501
7.486
7.260
6.832
6.814
6.752
6.745
6.543
6.537
6.525
6.519
4.723
3.611
3.045
9
8
7
6
5
4
3
2
1
-0
ppm
2.02
1.02
2.07
1.07
1.01
1.05
2.08
3.05
3.00

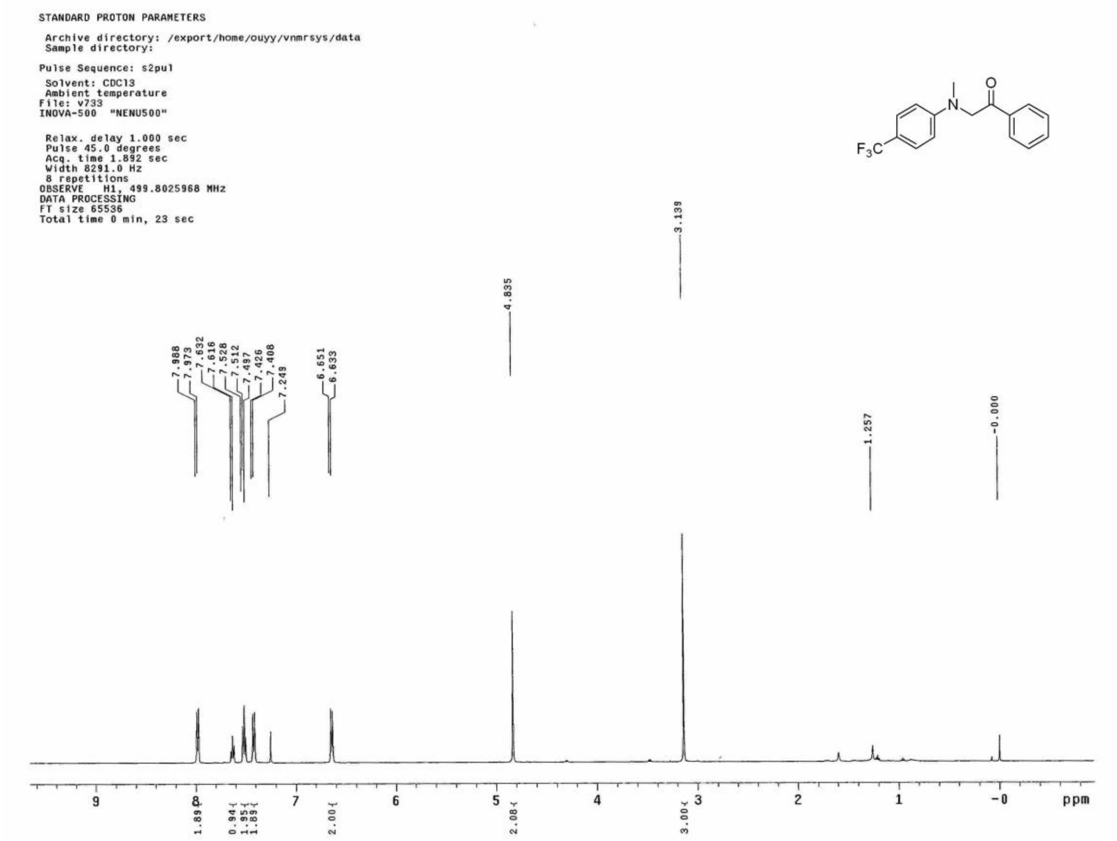

STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
User: 1-14-87
File: f945
INOVA-500 "NENU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
192 repetitions
OBSERVE C13, 125.6754651 MHz
DECOUPLE H1, 499.8050905 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 3 hr, 56 sec
Cl
N
O
O
196.209
147.067
144.477
135.267
133.646
128.828
127.775
123.376
114.822
113.953
111.610
77.256
77.000
76.748
59.279
56.898
39.925
220
200
180
160
140
120
100
80
60
40
20
0
ppm

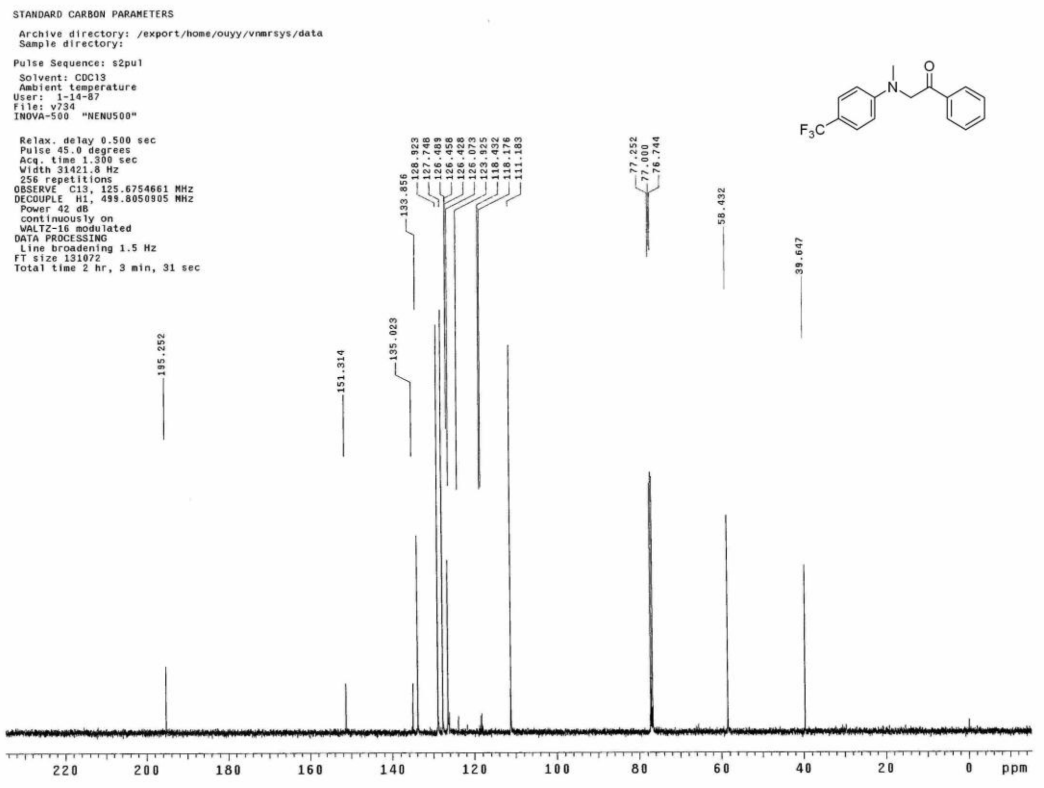
Product 3hb


STANDARD PROTON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: w035
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 8291.0 Hz
8 repetitions
OBSERVE H1, 499.8026014 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
7.965
7.949
7.618
7.604
7.588
7.501
7.486
7.471
7.131
7.114
6.566
6.549
4.743
3.064
1.256
ppm

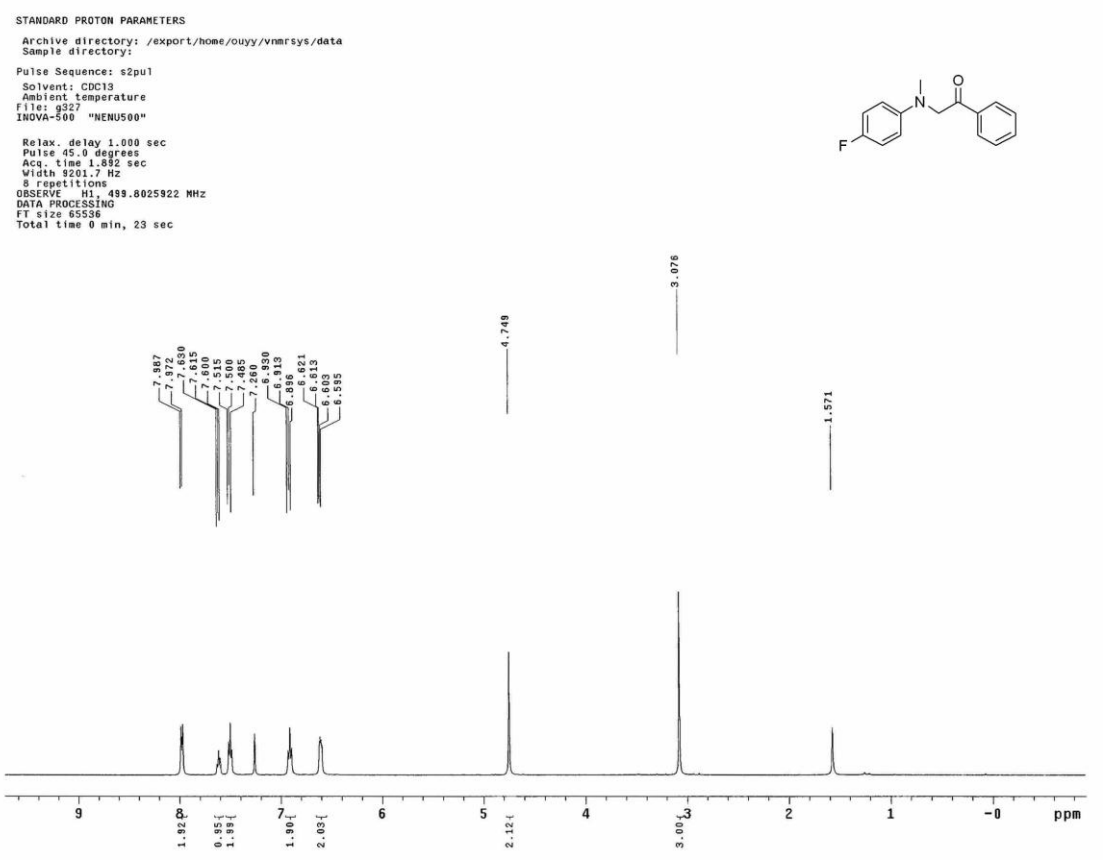

STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
File: w036
INOVA-500 "NENU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
128 repetitions
OBSERVE C13, 125.6754718 MHz
DECOUPLE H1, 499.8050905 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 3 hr, 56 sec
195.889
147.777
135.164
133.650
128.896
128.816
127.702
121.754
113.254
77.252
76.998
76.744
58.802
39.696
ppm

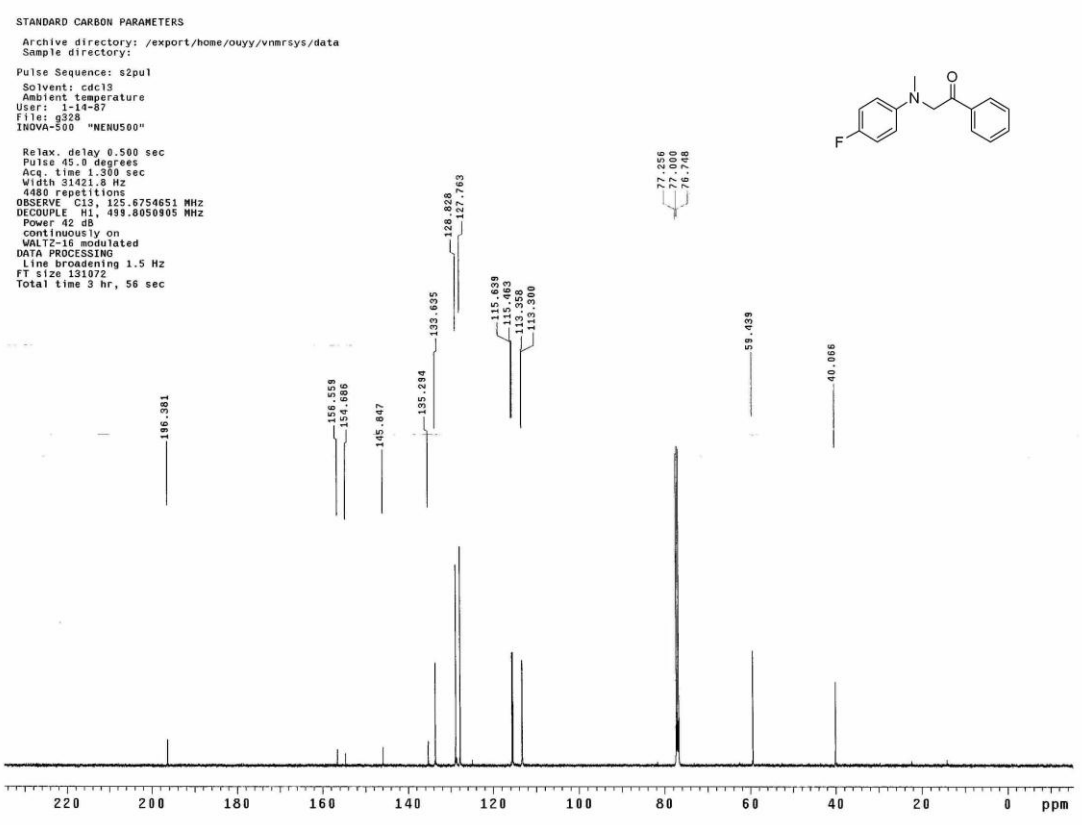
Product 3jb



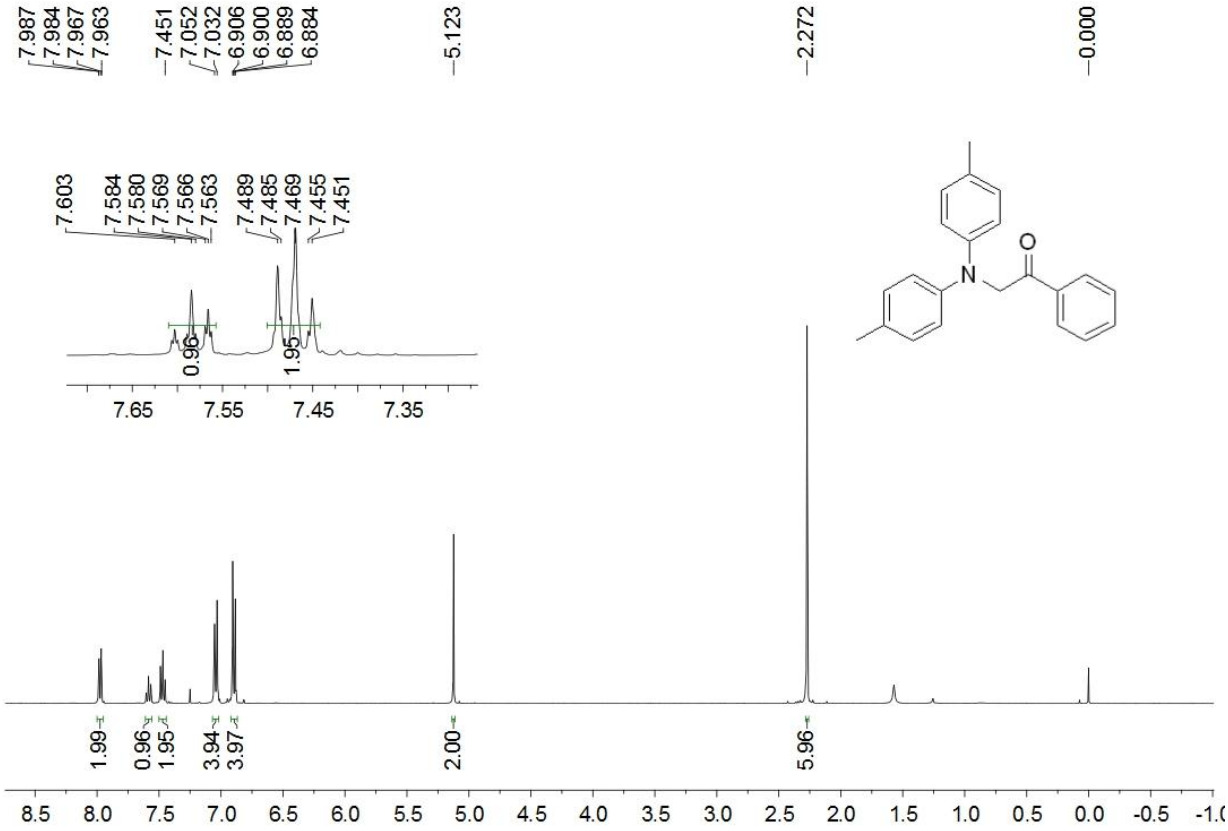


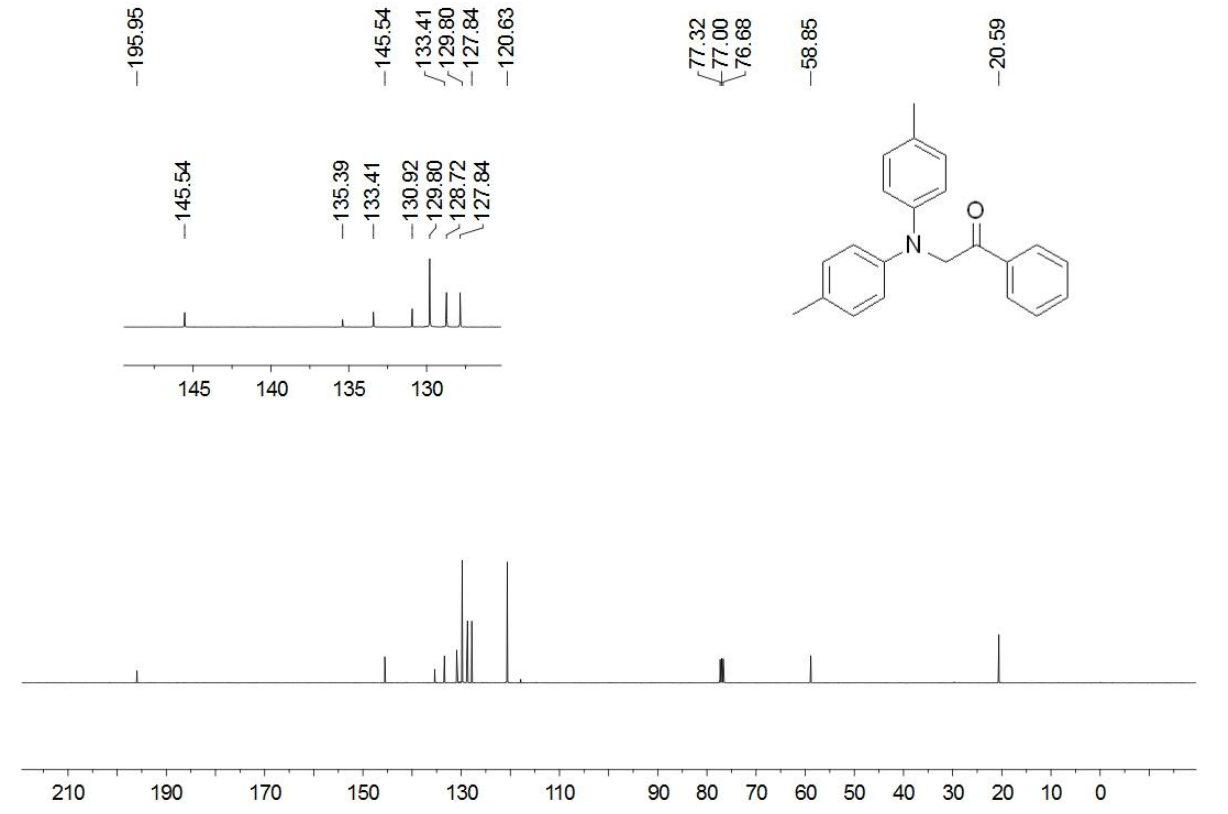
Product 3kb


STANDARD PROTON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: g327
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 9201.7 Hz
8 repetitions
OBSERVE H1, 499.8025922 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
7.987
7.972
7.630
7.615
7.600
7.515
7.500
7.485
7.240
6.930
6.913
6.896
6.621
6.613
6.603
6.595
4.749
3.076
1.571
1.02
0.95
1.99
1.90
2.03
2.12
3.00
9
8
7
6
5
4
3
2
1
-0
ppm

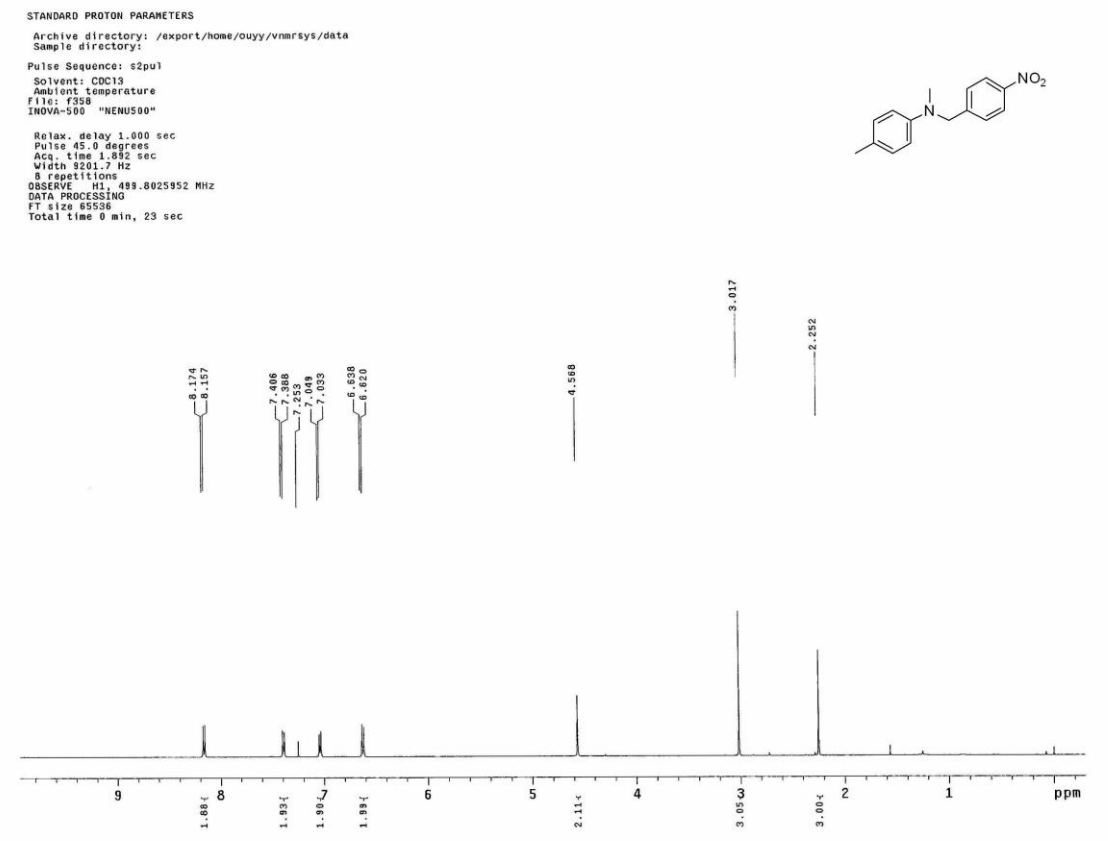

STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
User: 1-14-87
File: g328
INOVA-500 "NENU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
4480 repetitions
OBSERVE C13, 125.6754651 MHz
DECOUPLE H1, 499.8050905 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 3 hr, 56 sec
196.381
156.559
154.686
145.847
135.294
133.635
128.828
127.763
115.639
115.463
113.358
113.300
77.256
77.000
76.748
59.439
40.066
220
200
180
160
140
120
100
80
60
40
20
0
ppm

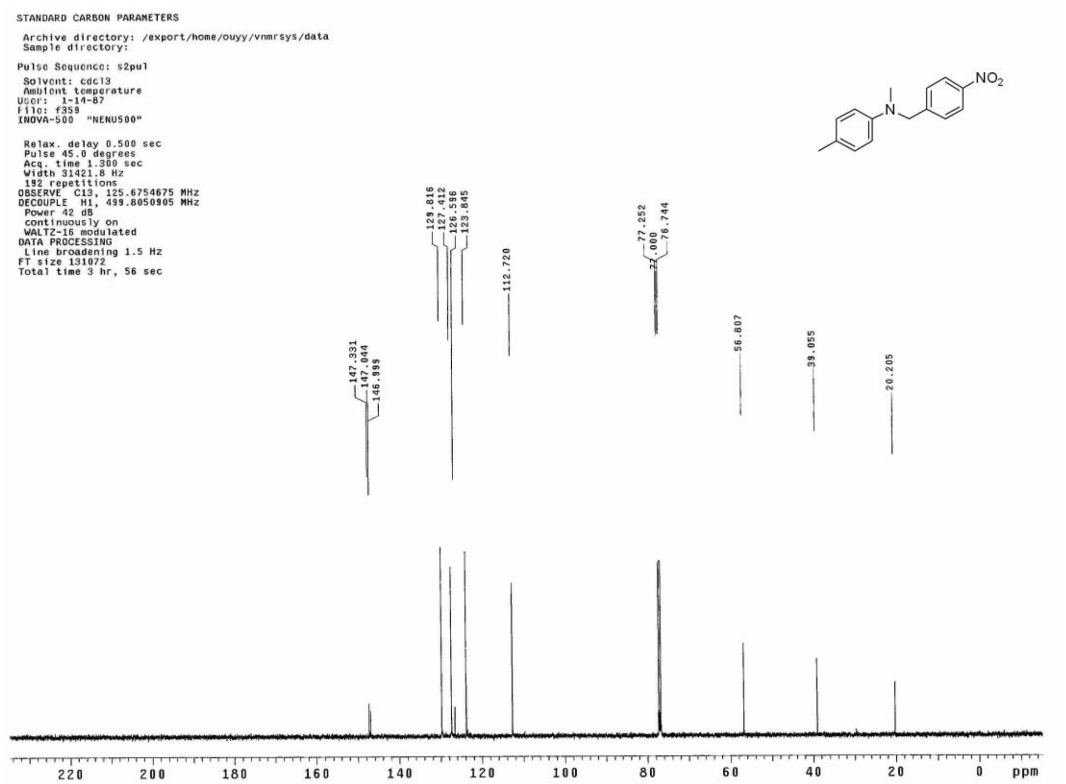
Product 3mb


7.987
7.984
7.967
7.963
7.451
7.052
7.032
6.906
6.900
6.889
6.884
5.123
2.272
0.000
7.603
7.584
7.580
7.569
7.566
7.563
7.489
7.485
7.469
7.455
7.451
0.96
1.95
7.65
7.55
7.45
7.35
O
N
1.99
0.96
1.95
3.94
3.97
2.00
5.96
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5

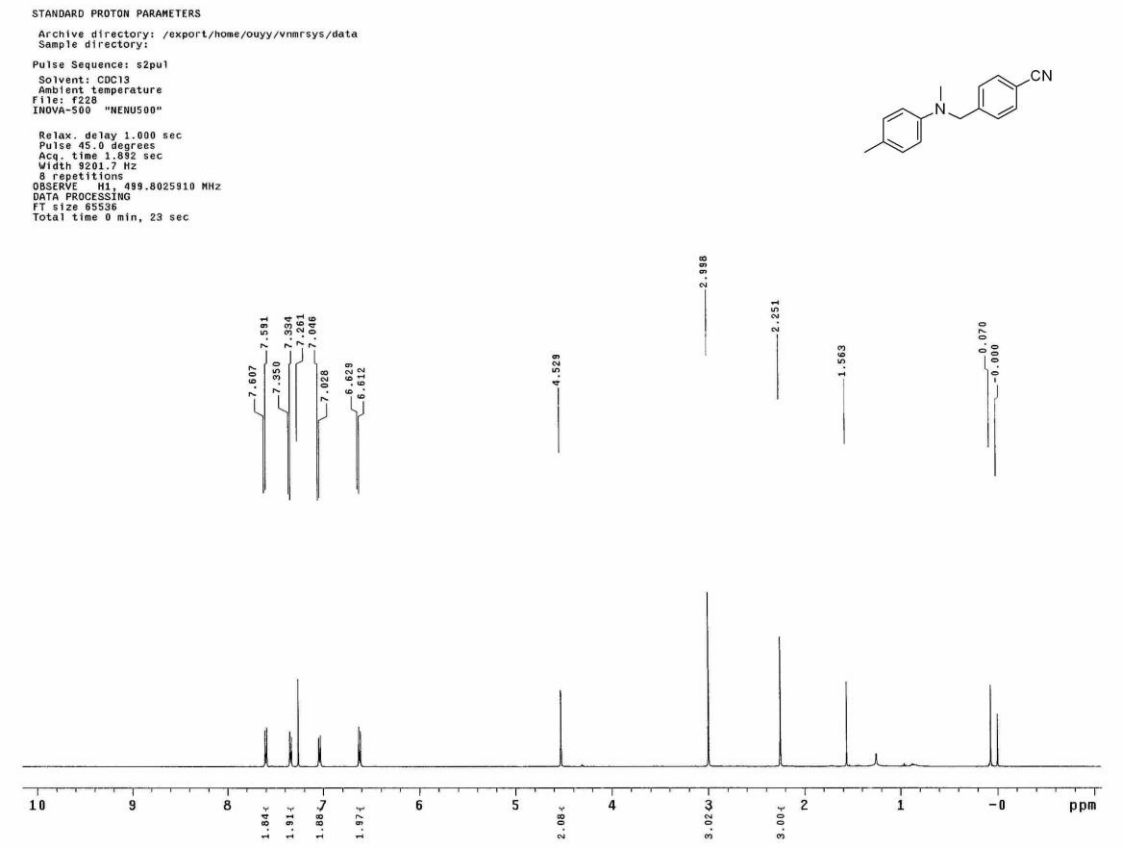

195.95
145.54
133.41
129.80
127.84
120.63
77.32
77.00
76.68
58.85
20.59
145.54
135.39
133.41
130.92
129.80
128.72
127.84
145
140
135
130
O
N
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0

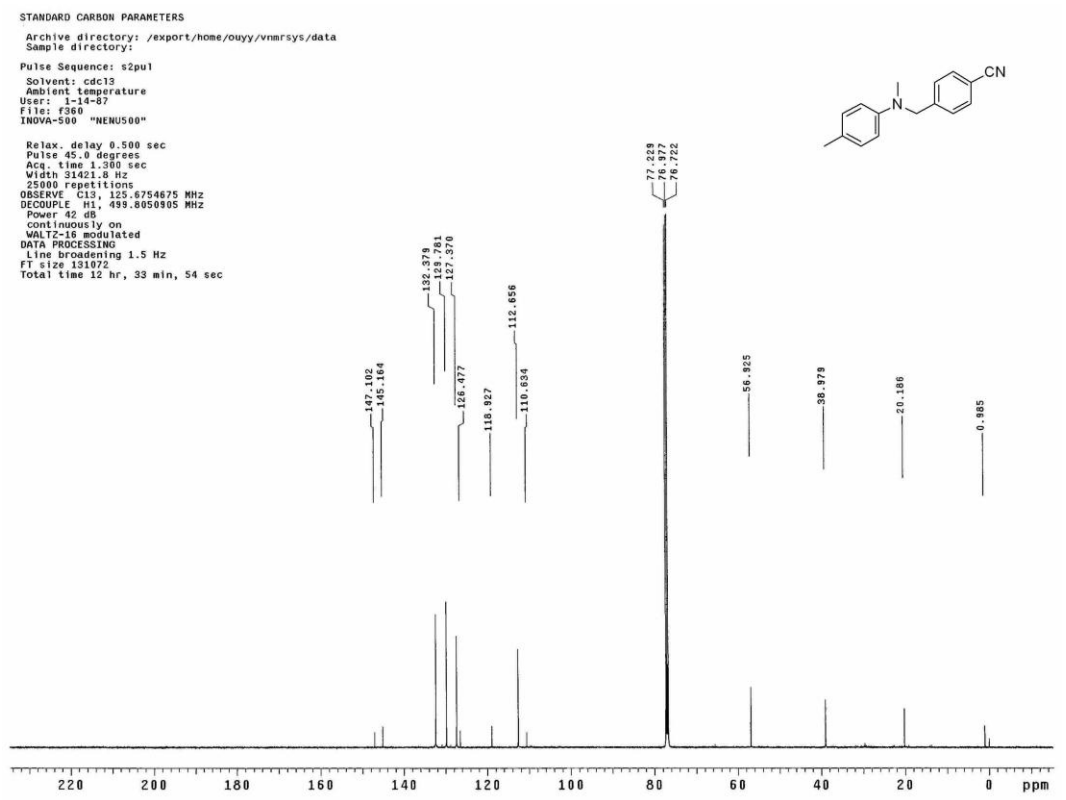
Product 3ac


STANDARD PROTON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: f358
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 9201.7 Hz
8 repetitions
OBSERVE H1, 499.8025952 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
NO2
N
8.174
8.157
7.406
7.388
7.253
7.049
7.033
6.638
6.620
4.568
3.017
2.252
ppm

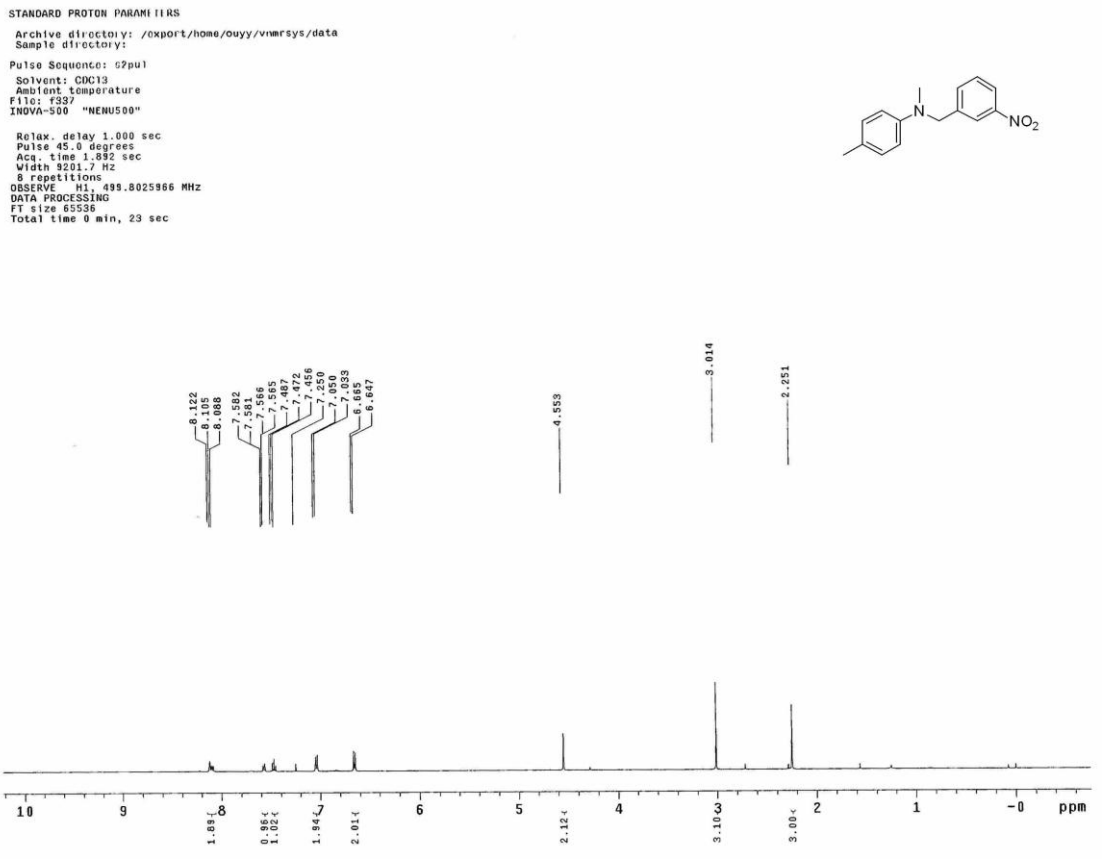

STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
User: 1-14-87
File: f359
INOVA-500 "NENU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
192 repetitions
OBSERVE C13, 125.6754675 MHz
DECOUPLE H1, 499.8050905 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 3 hr, 56 sec
NO2
N
147.331
147.044
146.999
129.816
127.412
126.596
123.845
112.720
77.252
77.000
76.744
56.807
39.055
20.205
ppm

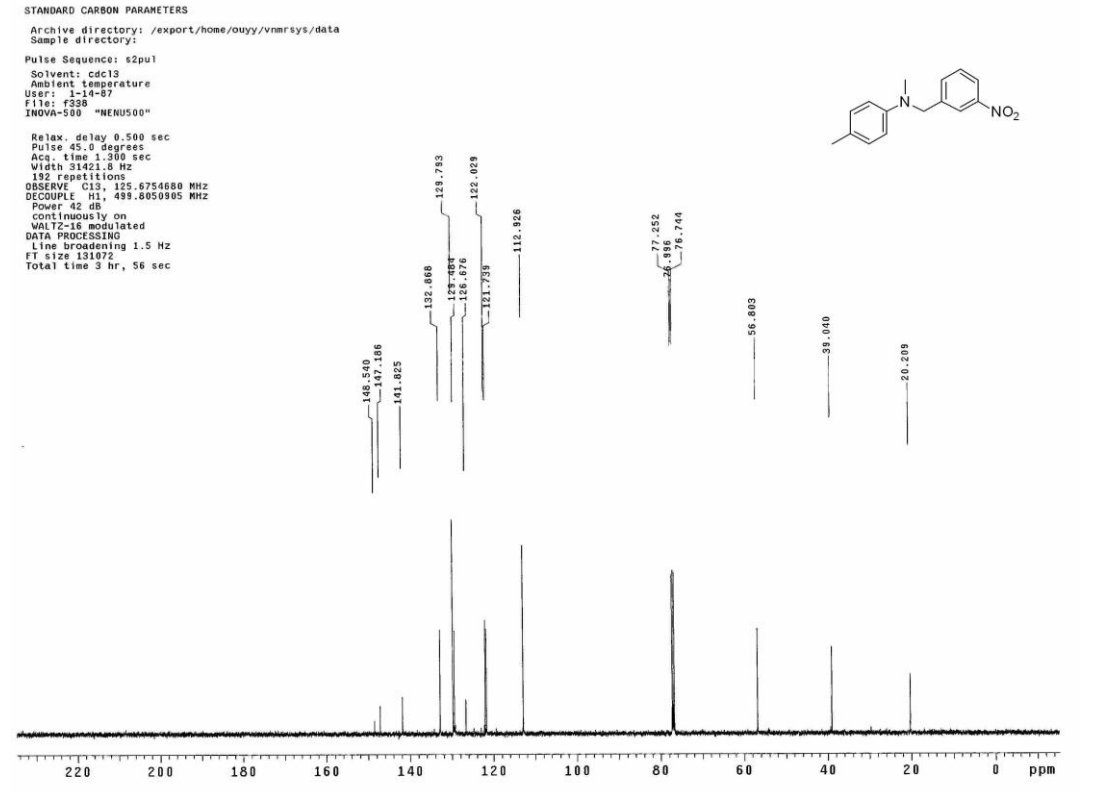
Product 3ad


STANDARD PROTON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: f228
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 9201.7 Hz
8 repetitions
OBSERVE H1, 499.8025910 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
CN
7.607
7.591
7.350
7.334
7.261
7.046
7.028
6.629
6.612
4.529
2.998
2.251
1.563
0.070
-0.000
10
9
8
7
6
5
4
3
2
1
-0
ppm
1.84
1.91
1.88
1.97
2.08
3.02
3.00


STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
User: 1-14-87
File: f360
INOVA-500 "NENU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
25000 repetitions
OBSERVE C13, 125.6754675 MHz
DECOUPLE H1, 499.8050905 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 12 hr, 33 min, 54 sec
CN
147.102
145.164
132.379
129.781
127.370
126.477
118.927
112.656
110.634
77.289
77.077
76.722
56.925
30.979
20.186
0.985
220
200
180
160
140
120
100
80
60
40
20
0
ppm

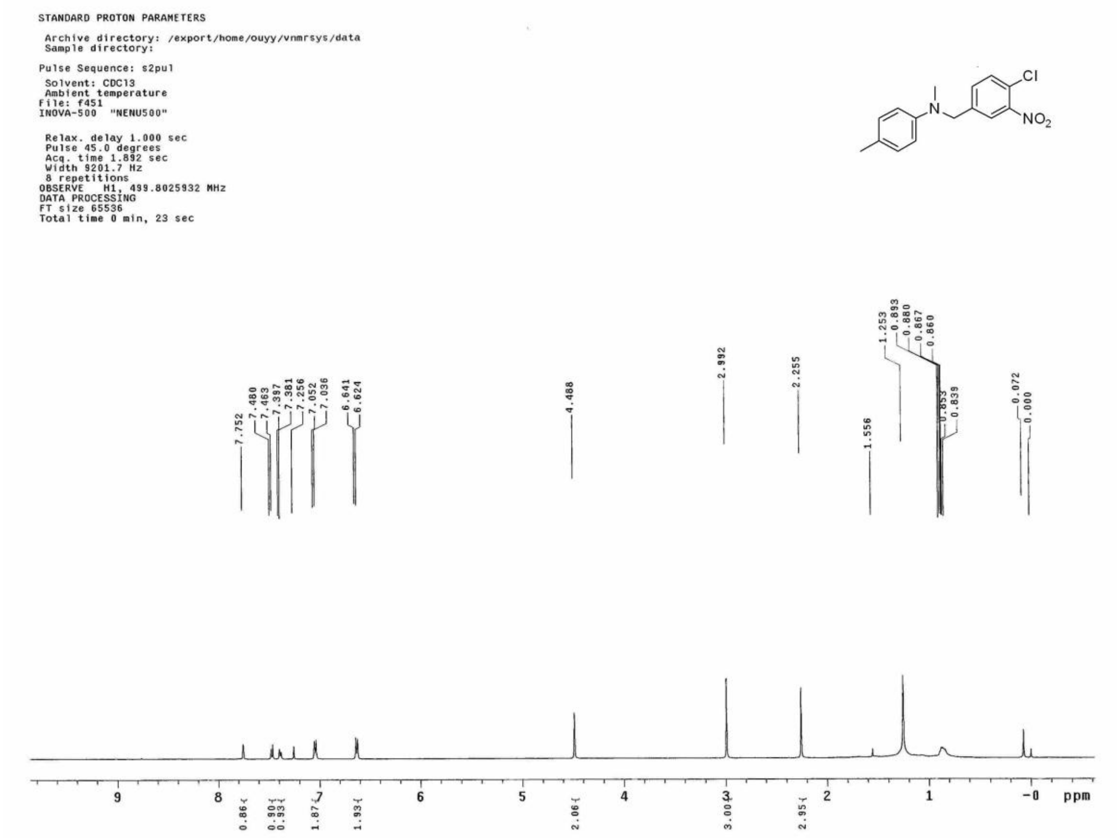
Product 3ae

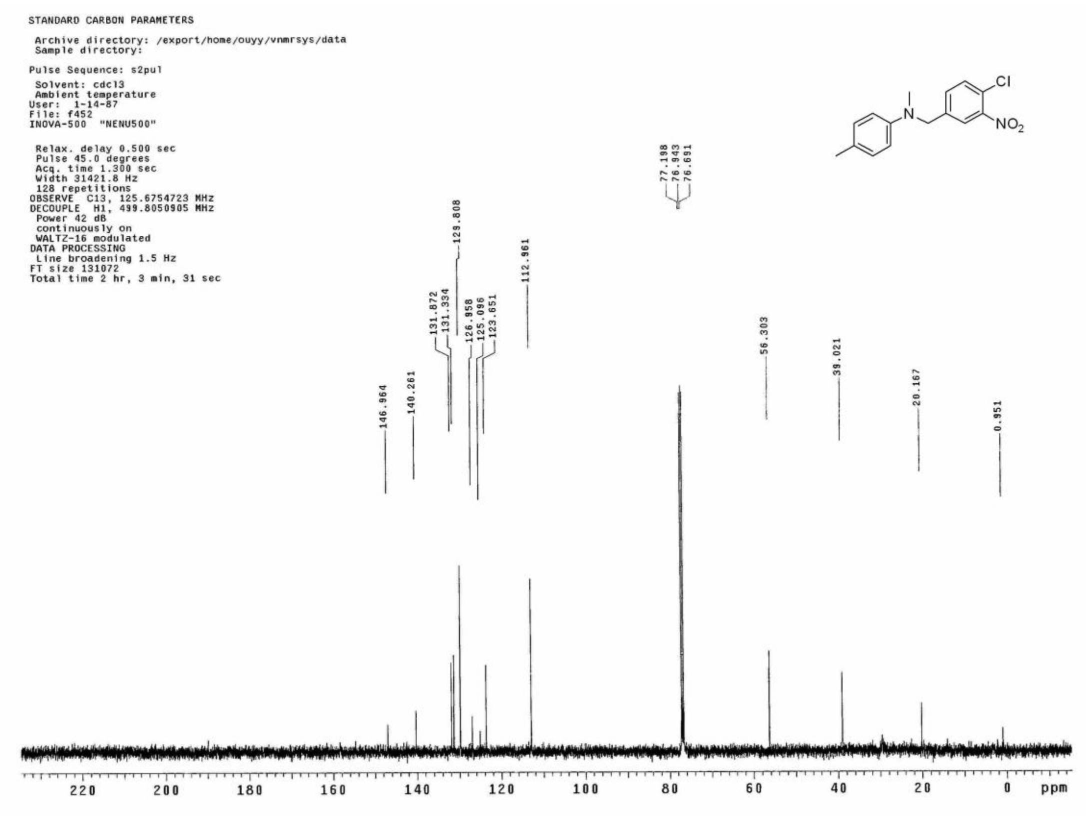

STANDARD PROTON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: f337
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 9201.7 Hz
8 repetitions
OBSERVE H1, 499.8025866 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
NO2
8.122
8.105
8.088
7.582
7.581
7.566
7.565
7.467
7.272
7.456
7.250
7.050
7.033
6.685
6.647
4.553
3.014
2.251
ppm


STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
User: 1-14-87
File: f338
INOVA-500 "NENU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
192 repetitions
OBSERVE C13, 125.6754680 MHz
DECOUPLE H1, 499.8050905 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 3 hr, 56 sec
NO2
148.540
147.186
141.825
132.868
129.793
127.484
126.676
122.029
121.739
112.926
77.252
76.996
76.744
56.803
39.040
20.209
ppm

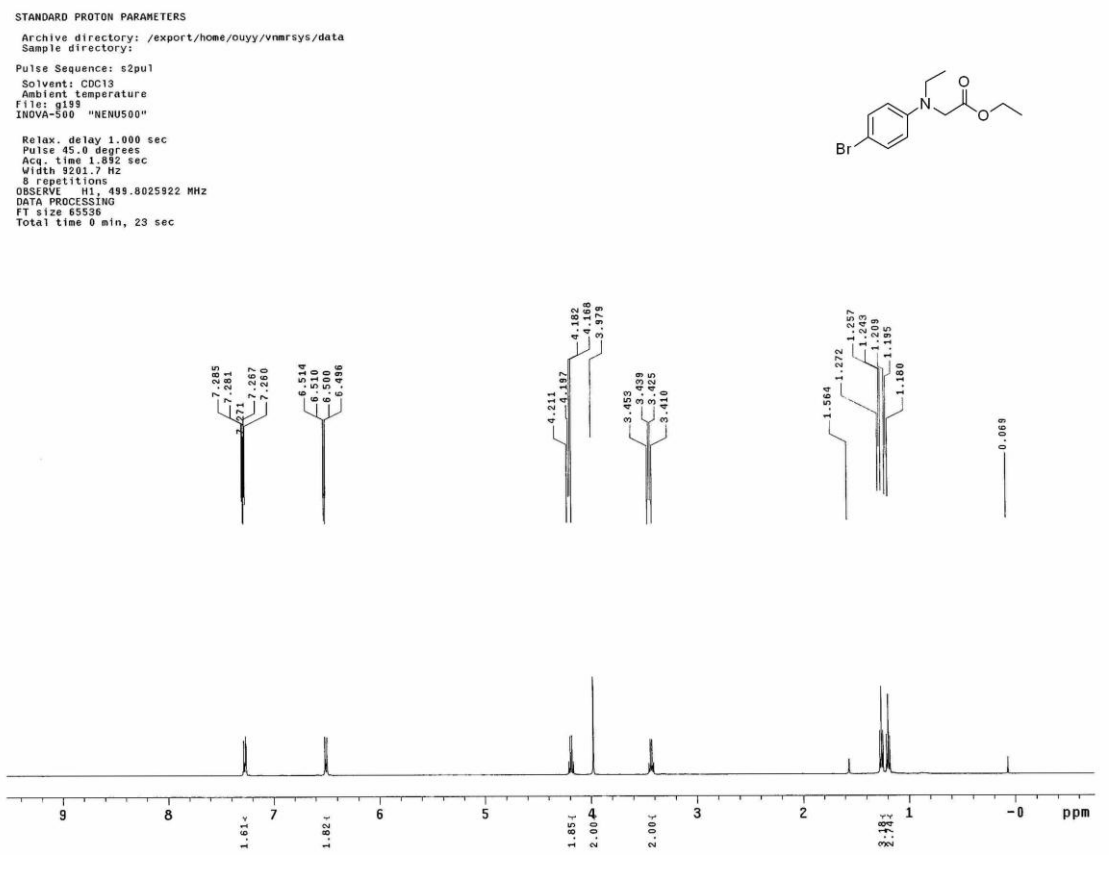
Electronic Supplementary Material (ESI) for Chemical Communications

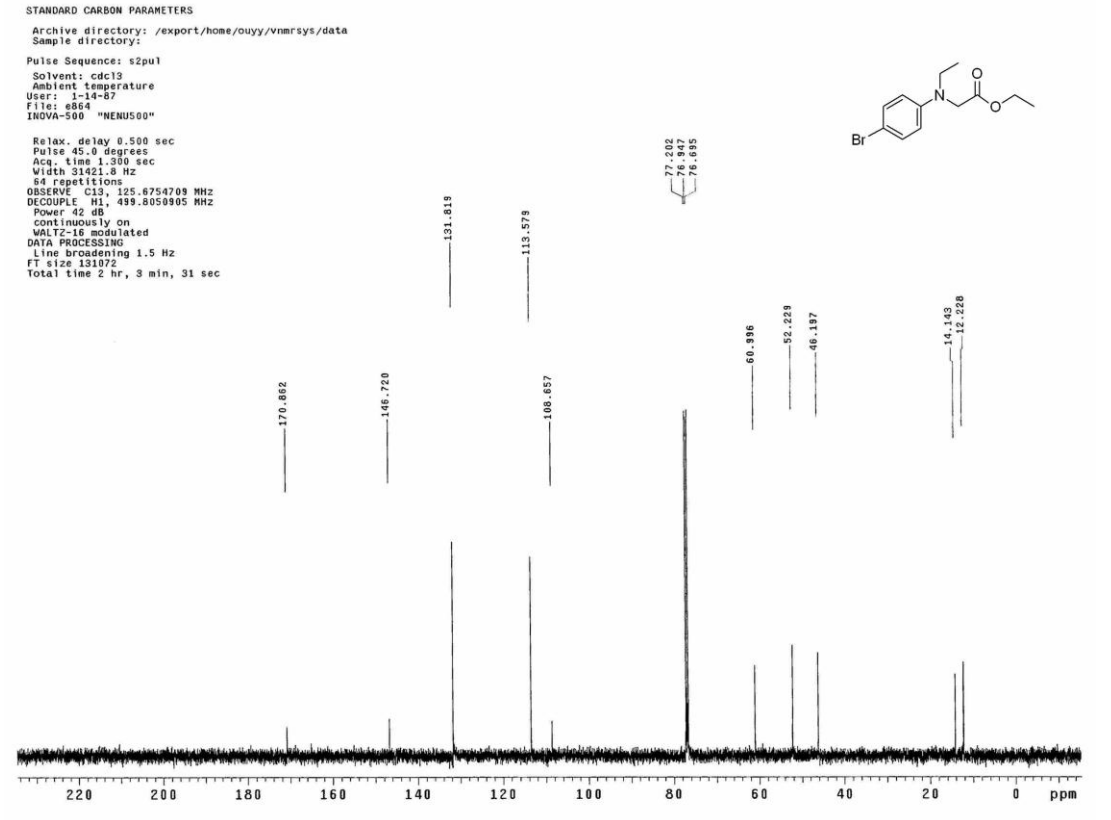
Product 3af



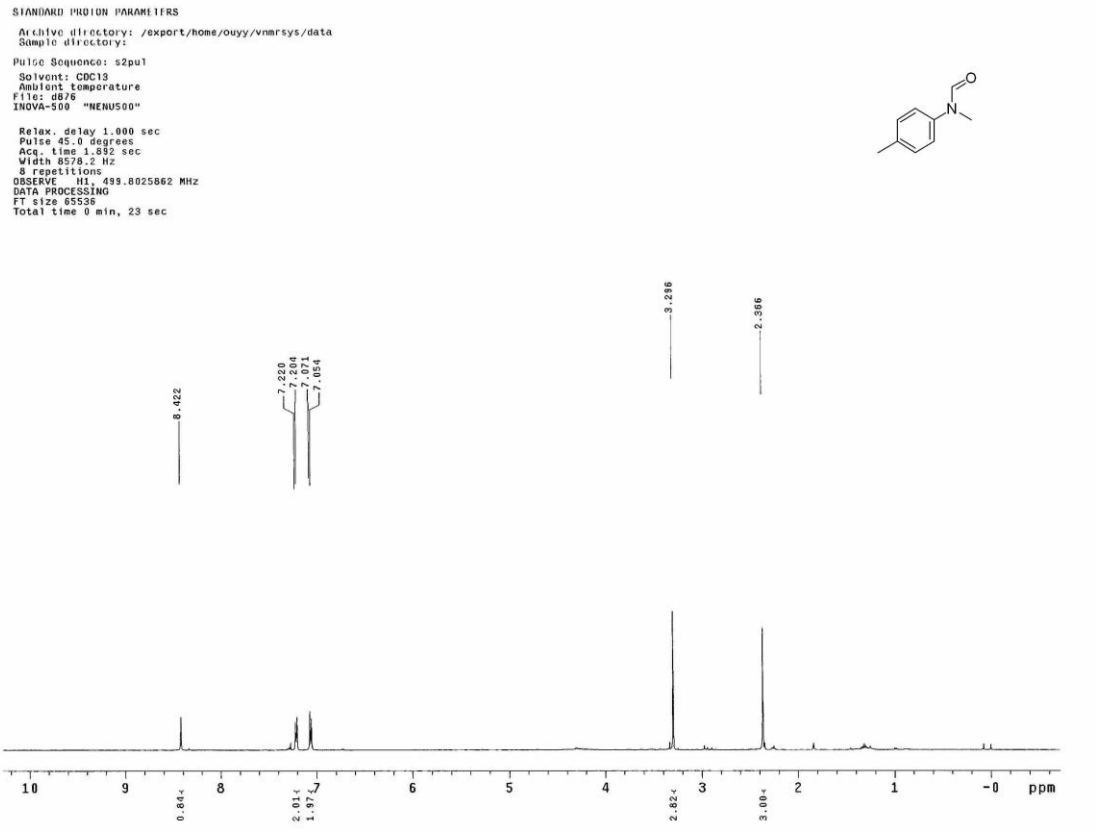


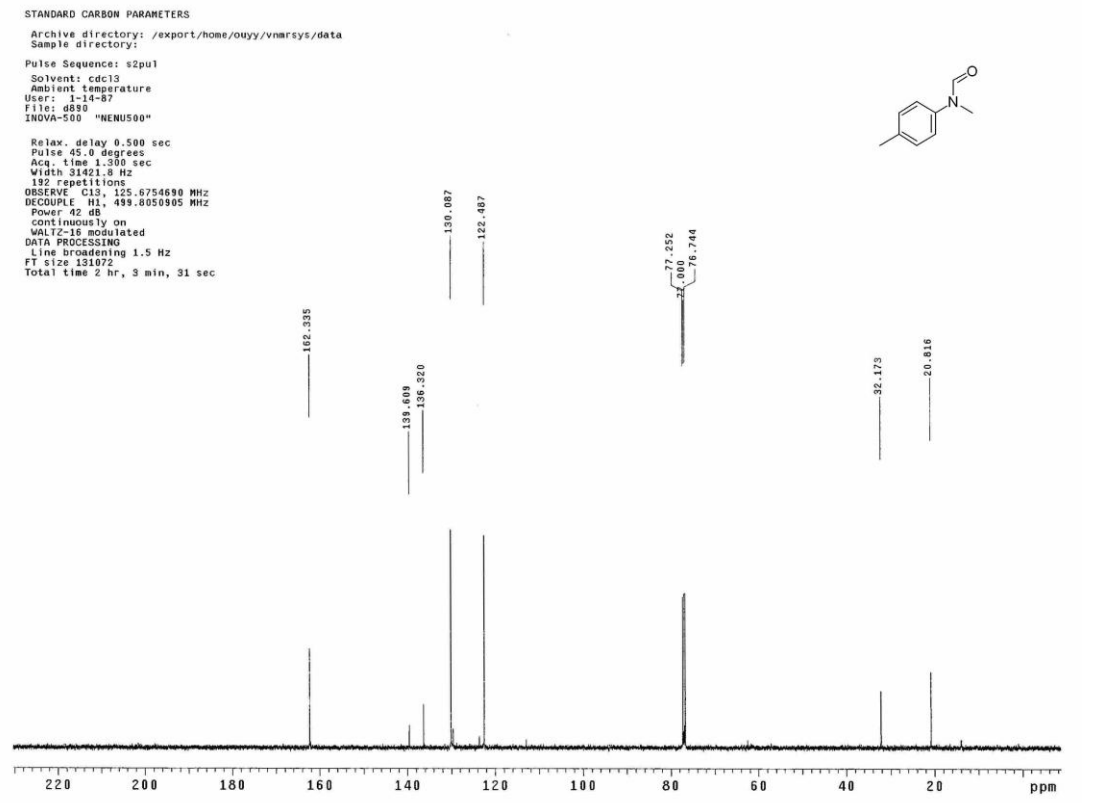
Product 3la


STANDARD PROTON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: g199
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 9201.7 Hz
8 repetitions
OBSERVE H1, 499.8025922 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
Br
ppm

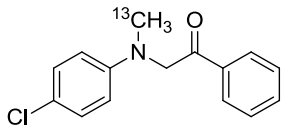

STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
User: 1-14-87
File: e864
INOVA-500 "NENU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
64 repetitions
OBSERVE C13, 125.6754709 MHz
DECOUPLE H1, 499.8050905 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 2 hr, 3 min, 31 sec
Br
ppm

Product 4


STANDARD PROTON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: d876
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 8578.2 Hz
8 repetitions
OBSERVE H1, 499.8025862 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
8.422
7.220
7.204
7.071
7.054
3.286
2.366
10
9
8
7
6
5
4
3
2
1
-0
ppm
0.84
2.01
1.97
2.82
3.00


STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
User: 1-14-87
File: d880
INOVA-500 "NENU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
192 repetitions
OBSERVE C13, 125.6754690 MHz
DECOUPLE H1, 499.8050905 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 2 hr, 3 min, 31 sec
162.335
139.609
136.320
130.087
122.487
77.252
77.000
76.744
32.173
20.816
220
200
180
160
140
120
100
80
60
40
20
ppm

Ⅵ. The HRMS spectrum of [$^{13}C_1$]-3hb



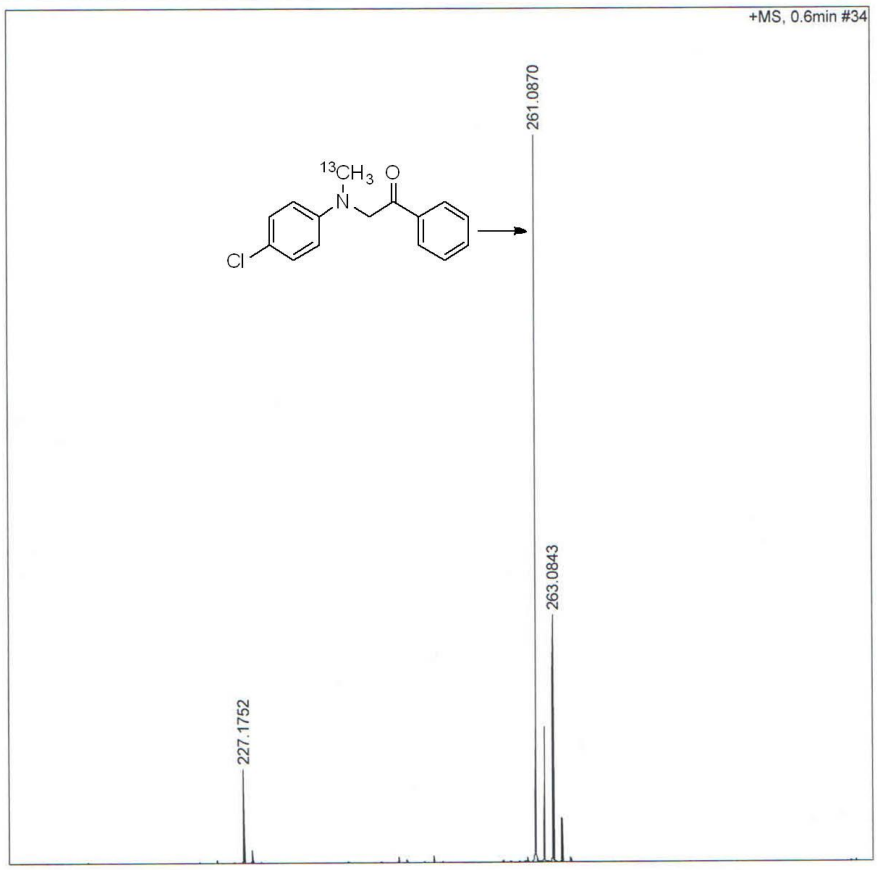
HRMS (ESI-TOF) Calcd for $C_{14}{}^{13}CH_{14}ClNO$, $[M+H]^+$ 261.0870 ; Found 261.0870.

Analysis Info

Analysis Name	D:\Data\user\B2012\1030\B-a206_18_01_3452.d	Acquisition Date	10/30/2012 12:45:10 PM
Method	Sample 5 min.m	Operator	
Sample Name	B-a206	Instrument / Ser#	micrOTOF 10328
Comment			

Acquisition Parameter

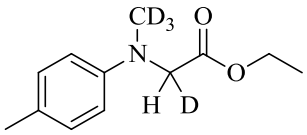
Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.5 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	8.0 l/min
Scan End	1000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste



Meas. m/z	#	Formula	Score	m/z	err [ppm]	Mean err [ppm]	mSigma	rdb	e⁻ Conf	N-Rule
261.0870	1	C 14 H 15 Cl Ct N O	100.00	261.0870	-0.0	0.1	22.1	8.5	even	-

Bruker Compass DataAnalysis 4.0 printed: 10/31/2012 2:24:11 PM Page 1 of 1

Ⅶ. The ^{1}H NMR spectrum of [D_4]-3aa



^{1}H NMR (500 MHz; $CDCl_3$): δ = 1.24 (t, J = 7.0 Hz, 3H), 2.24 (s, 3H), 4.00 (d, J = 2.0 Hz, 1H), 4.16 (q, J = 7.0 Hz, 2H), 6.61 (d, J = 8.5 Hz, 2H), 7.04 (d, J = 8.5 Hz, 2H).