Electronic Supplementary Information

Ortho-directed Functionalization of Arenes using Magnesate Bases

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General Information

THF was distilled from benzophenone/Na and used immediately. Water content of the solvent was estimated by the modified Karl Fisher method (less than 60 ppm water). IR spectra were obtained as potassium bromide pellets with a Perkin Elmer Paragon 500 spectrometer. Absorption bands are given in cm^{-1}. ^{1}H NMR, ^{13}C NMR and ^{19}F NMR spectra were recorded in $CDCl_3$ with a Bruker Avance 300 spectrometer (^{1}H at 300MHz, ^{13}C at 75.4MHz and ^{19}F at 282.5MHz). Chemical shift δ were reported in ppm relative to the residual solvent peak (^{1}H, δ=7.26 or ^{13}C, δ=77.16). Melting points (°C) were measured on a Kofler hot-stage (± 2°C) and are uncorrected. Elemental analyses were performed on a Carlo Erba 1106 apparatus or CE Instrument EA 1110 and measurement accuracy is around ± 0.4% on carbon. Column chromatography was performed using silica gel (mesh size 60-80μM). Commercially available (4,4'dimethyloxazolin-2-yl)benzene **1a**, 1,4-dimethoxybenzene **1m** and anisole **1l** were used without further purification.

Preparation of benzene derivatives 1b-k

Synthesis of oxazolinylbenzenes **1b-c**:[1a]

Zinc chloride (300 mg, 2 mmol) was placed in a 250 mL round bottom flask, melted three times under high pressure and allowed to cool to room temperature under N_2 before a solution of 4-chlorobenzonitrile (20 mmol) (respectively 4-methoxybenzonitrile) and 2-amino-2-methylpropane-1-ol (2.87 mL, 30 mmol) in dry chlorobenzene (40 mL) was added. The resulting mixture was refluxed for 48h. The volatiles were removed under vacuum and water (30 mL) was added. The aqueous layer was extracted with CH_2Cl_2 and the combined organic extracts were washed with water, brine, dried ($MgSO_4$) and concentrated in vacuum. The crude product was purified by flash chromatography on silica gel to give 4-chloro-2'-oxazolinylbenzene **1b (**3.9g, 92%) and **1c** (3.4g, 83% yields). All analyses are in accordance with those described in literature.[1b]

Synthesis of *N*-*tert*-butylbenzamides **1d-f** and *N*-cumylbenzamides **1g-i**:[2a]

A solution of benzoyl chloride (10 mmol) (respectively 4-chlorobenzoylchloride, 4-methoxybenzoyl chloride) in THF (30 mL) was added dropwise at 0°C to a solution of *tert*-butylamine (20 mmol) or cumylamine[2b] in THF (30 mL). After stirring 18h, aq.K_2CO_3 (2 mL, 2M) was added. The mixture was extracted with Et_2O (20 mL) and the combined organic extracts were washed water and dried ($MgSO_4$). After filtration, solvents were removed in vacuum to afford crude *N*-*tert*-butylbenzamide **1d-f** and *N*-cumylbenzamide **1g-i** which was purified by flash chromatography on silica gel using EtOAc and petroleum ether as eluents to give *N*-*tert*-butylbenzamide **1d** (1.5g, 86%), 4-chloro-*N*-*tert*-butylbenzamide **1e** (1.9g**,** 90%), 4-methoxy-*N*-*tert*-butylbenzamide **1f** (1.8g**,** 86%), *N*-cumylbenzamide **1g** (1.8g, 76%), 4-

[1] (a) G. K. Jnaneshwara, V. H. Deshpande, M. Lalithambika, T. Ravindranathan, A. V. Bedekar, Tetrahedron Lett., 1998, **39**, 459-462. (b) A. R. Katritzky, C. Cai, K. Suzuki, S. K. Singh, J. Org. Chem., 2004, **69**, 811-814.
[2] (a) Y. Uchida, Y. Kobayashi, S. Kozuka, Bull.Chem. Soc. Jpn., 1981, **54**, 1781-1786. (b) C.Metallinos, S. Nerdinger, V. Snieckus, Org. Lett., 1999, **1**, 1183-1186.

chloro-*N*-cumylbenzamide **1h (**2.2g**,** 81%), 4-methoxy-*N*-cumylbenzamide **1f** (2.3g, 84%) as white solids. All analyses of products **1d**,[3a] **1e,**[3b] **1f,**[3c] **1g**,[3d] **1i**[3e] are in accordance with those reported in literature data. All analysis of the new compound **1h** is given in the characterization data section.

Synthesis of *N*-pivaloylaniline **1k**[4]

A solution of trimethylacetylchloride (6.97 mL, 50 mmol) in CH_2Cl_2 (30 mL) was added dropwise at 0°C to a solution of aniline (4.55 mL, 50 mmol) and triethylamine (8.7 mL, 60 mmol). The resulting mixture was stirred overnight at room temperature. The solvents are removed under vacuum and the crude product was purified by chromatography on silica gel using EtOAc and petroleum ether as eluents to give pivaloylaminobenzene **1k** (8.6g, 97%) as a white solid. All analyses of product **1k** is given in the characterization data section.[4]

General Experimental Procedures of Magnesation-Functionalization Reactions

General procedure for preparation of lithium butylmagnesate bases (procedure 1):

To a suspension of magnesium turning (0.0486g, 2.0 mmol) in THF (10 mL) was added 1,2-dibromoethane (0.17 mL, 2.0 mmol) under N_2. The mixture was refluxed for 1h to afford a magnesium dibromide ($MgBr_{2)}$ as a colourless solution. After cooling at 0°C, the *n*-butyllithium (6 mmol or 8 mmol respectively, 2.5 M in hexanes) was added dropwise and the resulted mixture was stirred at the same temperature for 1 h to give a solution of Bu_3MgLi and Bu_4MgLi_2 (0.2 M) respectively.

General procedure for magnesation of benzene derivatives 1a-m (procedure 2):

Benzene derivatives **1a-c** (4.8 mmol), **1d-i** (3.6 mmol), **1k** (2mmol) and **1l-m** (6.4 mmol) were added dropwise at room temperature to a selected freshly prepared solution of Bu_3MgLi or Bu_4MgLi_2 (see Table 1) (2 mmol) in THF following the above procedure 1. The resulted mixture was stirred at room temperature or at reflux (see Table 1) over a 2h period before the subsequent introduction of the adequate reagents for electrophilic trapping or cross-coupling reactions (The magnesation was previously evaluated by carrying out D_2O-trapping experiments followed by ^1H NMR analysis of the crude of the deprotonation).

[3] (a) L. Zhang, S. Su, H. Wu, S. Wang, Tetrahedron, 2009, **65**, 10022-10024. (b) M. Achmatowicz, O.R. Thiel, P. Wheeler, C. Bernard, J. Huang, R.D. Larsen, M.M. Paul, J. Org. Chem., 2009, **74**, 795-809. (c) J.C., Baum, J.E., Milne, J.A. Murry, O.R. Thiel, J. Org. Chem. 2009, **74**, 2207-2209. (d) K. Kondo, E. Sekimoto, J. Nakao, Y. Murakami, Tetrahedron, 2000, **56**, 5843-5856. (e) J. Clayden, C. J. Menet, K. Tchabanenko, Tetrahedron, 2002, **58**, 4727-4733.

[4] J. P. Bezombes, P.B. Hitchock, M.F. Lappert, P. J. Merle, J. Chem. Soc. Dalton Trans., 2001, 816-821.

General procedure for iodination of benzene derivatives 1a-m (procedure 3):

To a solution of arylmagnesate in THF (10 mL) under N_2, prepared by treatment of **1a-m** with a butylmagnesate base following the above general magnesation procedure 2, was added a solution of I_2 (6 or 8 mmol using Bu_3MgLi or Bu_4MgLi_2 respectively) in THF (10 mL) at room temperature. After a 2h stirring period, satd. aq.NH_4Cl (1 mL) and satd.aq.$Na_2S_2O_3$ (5 mL) solutions were successively added. The mixture was the extracted with Et_2O (10 mL) and the combined organic phase were washed with water (10 mL) and brine (10 mL), dried ($MgSO_4$) and evaporated under vacuum to give crude iodoaromatics **2a-m**. Procedure of purification and all analysis are reported in the next 'characterization data products' section.

General procedure for alkylation of benzene derivatives 1a-m (procedure 4):

To a solution of arylmagnesate in THF (10 mL) under N_2, prepared by treatment of **1a-m** with a butylmagnesate base following the above general magnesation procedure 2, was added dropwise propylene oxide (6 mmol or 8 mmol respectively) at room temperature. After 2h stirring at room temperature, sadt.aq.NH_4Cl was added (1mL) and MgSO4 was added. After filtration, the solvent was removed under vacuum to give crude hydroxymethylaromatics **3a-e**. Procedure of purification and all analysis are reported in the next 'characterization data products' section

General procedure for (hetero)arylation and vinylation of benzene derivatives 1a-m (procedure 5):

To a solution of arylmagnesate in THF (10 mL) under N_2, prepared by treatment of **1a-m** with a butylmagnesate base following the above general magnesation procedure 2, cross-coupling partner (2-bromopyridine, 3-iodopyridine, 3-bromoquinoline and 1-methylbromoethene) (6 mmol or 8 mmol respectively) and [1,1'-Bis(diphenylphosphino)ferrocene]palladium (II) chloride (5 mol%) were added. After refluxing for 18h., sadt.aq.NH_4Cl (1 mL) was then added and the resulted mixture was filtered on Celite washed with Et_2O (40 mL). The solution was dried over $MgSO_4$, and after filtration solvents were removed to give crude arylated and vinylated aromatics **4a-d**, **5a-d**, **6a-b**, **7a-d**. Procedure of purification and all analysis are reported in the next 'characterization data products' section

General procedure for oxydation of benzene derivatives 1a-m (procedure 6):

Dried oxygene (O_2 gas passed through a drying system consisting on a sulphuric acid buller followed by a cartridge of KOH and ends with a silica guard) was bubbled without flow control at room temperature for 10 min into a solution arylmagnesate in THF (10 mL) prepared by treatment of **1a-m** with a butylmagnesate base following the above general magnesation procedure 2. satd.aq.$Na_2S_2O_3$ (0.5 mL) and satd. aq.NH_4Cl (0.5 mL) solutions were successively added and after 5 min stirring, $MgSO_4$ was added. After filtration, the

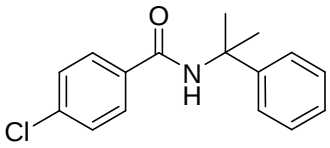
solvent was removed under vacuum to give crude alcohols **8a-d**. Procedure of purification and all analysis are reported in the next 'characterization data products' section.

General procedure for fluorination of benzene derivatives 1a-m (procedure 7):

To a solution of arylmagnesate in THF (10 mL) under N_2, prepared by treatment of **1a-m** with a butylmagnesate base following the above general magnesation procedure 2, was added dropwise at room temperature a solution of *N*-fluorobenzenesulfonimide (NFSi) (6 mmol or 8 mmol respectively) in THF (6 mL). After 30 min stirring, sadt.aq.NH_4Cl (1 mL) was added. The mixture was extracted with Et_2O (20 mL) and the combined organic phases were washed with aq.NaOH (20mL of 0.5 M solution), water (20mL) and brine (20 mL), dried over $MgSO_4$. After filtration, the solvent was removed under vacuum to give crude fluoroaromatics **9a-e**. Procedure of purification and all analysis are reported in the next 'characterization data products' section.

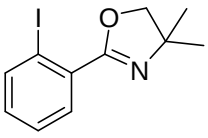
Characterization Data of Products

4-Chloro-*N*-cumylbenzamide **(1h)**



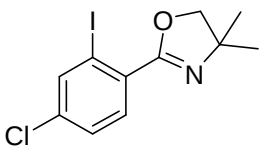
Crude **1h** was prepared according the above reported general synthesis of benzamides and purified by column chromatography on silica gel (AcOEt/PE 2:8, Rf = 0.4) to give a white solid (mp = 198-199°C). ^{1}H NMR ($CDCl_3$) δ 7.72-7.67 (m, 2H), 7.46-7.33 (m, 6H), 7.28-7.23 (m, 1H), 6.35 (br s, 1H), 1.82 (s, 6H); ^{13}C NMR ($CDCl_3$) δ 165.5, 146.7, 137.7, 133.8, 128.9, 128.7, 128.4, 127.0, 124.8, 56.6, 29.21; IR (KBr) ν 3277, 3060, 2978, 2364, 1634, 1594, 1534, 1486, 1196, 1013, 848, 762, 695; Anal. calcd. for $C_{16}H_{16}ClNO$: C, 70.20; H, 5.89; N, 5.12. Found: C, 70.42; H, 5.91; N, 4.95.

2-Iodo-2'-oxazolinylbenzene (2a)



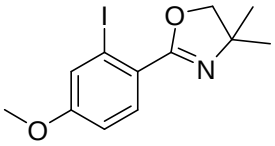
Crude **2a** was prepared according to the above procedure 3 using Bu_3MgLi as base and purified by flash chromatography to give an oil (1.0g, 75%). All analyses are in accordance with those reported in literature.[5a]

4-Chloro-2-iodo-2'-oxazolinylbenzene (2b)



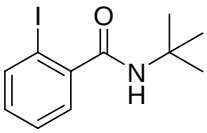
Crude **2b** was prepared according to the general procedure 3 using Bu_3MgLi as base and purified by flash chromatography (1.1g, 68%). All analyses are in accordance with those reported in literature.[5b]

[5] (a) J-. J. Li, R. Giri, J-. Q. Yu, *Tetrahedron*, 2008, **64**, 6979-6987. (b) J. Takaya, K. Sangu, N. Iwasawa, *Angew. Chem. Int. Ed.*, 2009, **48**, 7090-7093. (c) U. Ladziata, A. Y. Koposov, K. Y. Lo, J. Willging, V. N. Nemykin, V. V. Zhdankin, Angew. Chem. Int. Ed., 2005, 44, 7127-7131.(d) Y. Yamagushi, Y. Matsubara, T. Ochi, T. Wakamiya, Z. I. Yoshida, J. Am. Chem. Soc., 2008, **130**, 13867-13869.



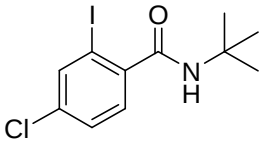
2-Iodo-4-methoxy-2'-oxazolinylbenzene (2c)

Crude **2c** was prepared according to the general procedure 3 using Bu_3MgLi as base and purified by flash chromatography (1.1g, 71%). All analyses are in accordance with those reported in literature.[5b]



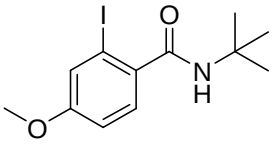
2-Iodo-*N*-*tert*-butylbenzamide (2d)

Crude **2d** was prepared according to the general procedure 3 using Bu_4MgLi_2 as base and purified by flash chromatography (AcOEt/PE 1:9, Rf = 0.11) as a white solid (0.9g, 82%), mp 124-125°C. ^{1}H NMR ($CDCl_3$) δ 7.84-7.82 (m, 1H), 7.40-7.33 (m, 2H), 7.09-7.04 (m, 1H), 5.52 (br s, 1H) 1.49 (s, 9H); ^{13}C NMR ($CDCl_3$) δ 168.8, 143.3, 139.7, 130.8, 128.2, 128.1, 92.5, 52.3, 28.8; IR (KBr) ν 3243, 3070, 2971, 1639, 1588, 1555, 1331, 1224, 1014, 943, 881, 749, 719, 677, 637; Anal. calcd. for $C_{11}H_{14}INO$: C, 43.58; H, 4.66; N, 4.62. Found : C, 43.60; H, 4.60 ; N, 4.65.



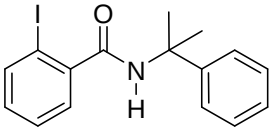
4-Chloro-2-iodo-*N*-*tert*-butylbenzamide (2e)

Crude **2e** was prepared according to the general procedure 3 using Bu_4MgLi_2 as base and purified by flash chromatography (AcOEt/PE 2:8, Rf = 0.46) as a white solid (0.9g, 76%), mp= 142-143 °C. ^{1}H NMR ($CDCl_3$) δ 7.83 (d, J=1.8Hz, 1H), 7.36-7.29 (m, 2H), 5.52 (br s, 1H), 1.48 (s, 9H). ^{13}C NMR ($CDCl_3$) δ 167.9, 141.6, 139.0, 135.6, 128.8, 128.4, 92.7, 52.4, 28.7; IR (KBr) ν 3255, 2964, 1637, 1553, 1365, 1224, 1095, 822, 572; Anal. calcd. for $C_{11}H_{13}ClINO$: C, 39.14; H, 3.88; N, 4.15. Found : C, 39.23 ; H, 3.67 ; N, 4.15 .



2-Iodo-4-methoxy-*N*-*tert*-butylbenzamide (2f)

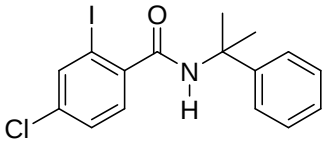
Crude **2f** was prepared according to the general procedure 3 using Bu_4MgLi_2 as base and purified by flash chromatography (AcOEt/PE 2:8, Rf = 0.3) as a white solid (1g, 84%), mp = 82-83 °C. ^{1}H NMR ($CDCl_3$) δ 7.35 (s, 1H), 7.33 (dd, J=8.4Hz, J=2.7Hz, 1H), 6.87 (dd, J=8.7Hz, J=2.4Hz, 1H), 5.56 (br s, 1H), 3.79 (s, 3H), 1.47 (s, 9H). ^{13}C NMR ($CDCl_3$) δ 168.5, 160.3, 135.6, 129.2, 124.9, 114.0, 92.9, 55.6, 52.1, 28.8 IR (KBr) ν 3275, 2967, 1633, 1594, 1485, 1295, 1223, 1026, 887, 837, 820, 685; Anal. calcd. for $C_{12}H_{16}INO_2$: C, 43.26; H, 4.84; N, 4.20. Found : C, 43.34 ; H, 4.78 ; N, 4.23 .



2-Iodo-*N*-cumylbenzamide (2g)

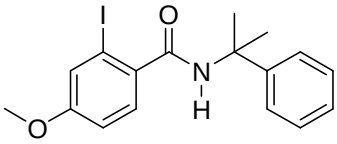
Crude **2g** was prepared according to the general procedure 3 using Bu_4MgLi_2 as base and purified by flash chromatography (AcOEt/PE 2:8, Rf = 0.2) as a orange solid (1.2g, 88%), mp= 122-123°C. ^{1}H NMR ($CDCl_3$) δ 7.86-7.83

(m, 1H), 7.54-7.51 (m, 2H), 7.43-7.34 (m, 4H), 7.29-7.23 (m, 1H), 7.10-7.05 (m, 1H), 6.04 (br s, 1H), 1.85 (s, 6H); ^{13}C NMR ($CDCl_3$) δ 168.2, 146.6, 142.8, 139.9, 131.0, 128.6, 128.4, 128.2, 127.0, 125.0, 92.4, 56.3, 28.9; IR (KBr) ν 3312, 3059, 2987, 2933, 1649, 1583, 1526, 1309, 1194, 1017, 906, 751, 694, 561; Anal. calcd. for $C_{16}H_{16}INO$: C, 52.62; H, 4.42; N, 3.84. Found : C, 52.62; H, 4.52; N, 3.65.



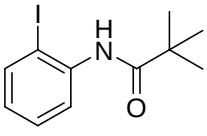
4-Chloro-2-iodo-*N*-cumylbenzamide (2h)

Crude **2h** was prepared according to the general procedure 3 using Bu_4MgLi_2 as base and purified by flash chromatography (AcOEt/PE 2:8, Rf = 0.4) as a white solid (1.2g, 82%), mp = 132-133 °C. ^{1}H NMR ($CDCl_3$) δ 7.85-7.84 (m, 1H), 7.52-7.48 (m, 2H), 7.39-7.34 (m, 4H), 7.29-7.24 (m, 1H), 6.01 (br s, 1H), 1.85 (s, 6H); ^{13}C NMR ($CDCl_3$) δ 167.3, 146.4, 141.1, 139.1, 135.7, 129.0, 128.5, 128.4, 126.9, 125.0, 92.6, 56.9, 28.8; IR (KBr) ν 3230, 3058, 2976, 1640, 1577, 1552, 1322, 1097, 1029, 767, 703; Anal. calcd. for $C_{16}H_{15}ClINO$: C, 48.08; H, 3.78; N, 3.50. Found : C, 48.12; H, 3.53; N, 3.73.



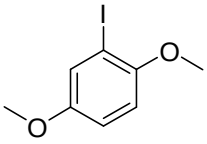
2-Iodo-4-methoxy-*N*-cumylbenzamide (2i)

Crude **2h** was prepared according to the general procedure 3 using Bu_4MgLi_2 as base and purified by flash chromatography (AcOEt/PE 2:8, Rf = 0.2) as a white solid (1.2g, 87%), mp = 121-122 °C. ^{1}H NMR ($CDCl_3$) δ 7.53-7.50 (m, 2H), 7.39-7.23 (m, 5H), 6.90-6.87 (m, 1H), 6.14 (br s, 1H), 3.80 (s, 3H), 1.84 (s, 6H); ^{13}C NMR ($CDCl_3$) δ 167.9, 160.6, 146.7, 135.1, 129.6, 128.5, 126.9, 125.2, 125.0, 114.1, 92.9, 56.8, 55.7, 29.0 IR (KBr) ν 3230, 3071, 2973, 1639, 1597, 1490, 1326, 1235, 1034, 817, 761, 701, 549; Anal. calcd. for $C_{17}H_{18}INO_2$: C, 51.66; H, 4.59; N, 3.54. Found : C, 51.76; H, 4.48; N, 3.33.



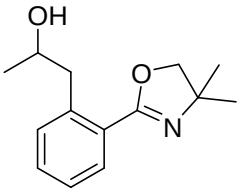
2-Iodo-pivaloylaminobenzene (2k)

Crude **2f** was prepared according to the general procedure 3 using Bu_4MgLi_2 as base and purified by flash chromatography flash chromatography (AcOEt/PE 2:8, Rf = 0.4) as yellow solid (0.4g, 70%). All analyses are in accordance with those reported in literature.[5c]



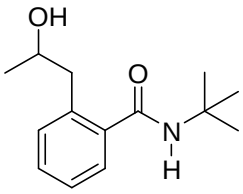
1,4-Dimethoxy-2-Iodobenzene (2m)

Crude **2m** was prepared according to the general procedure 3 using Bu_4MgLi_2 as base and purified by flash chromatography flash chromatography (AcOEt/PE 2:8, Rf = 0.7) flash chromatography (1.5g, 91%). All analyses are in accordance with those reported in literature. [5d]



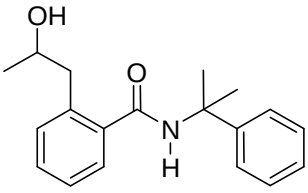
2-(2'-Hydroxypropyl)-2'-oxazolinylbenzene (3a)

Crude **3a** was prepared according to the general procedure 4 using Bu_3MgLi as base and purified by flash chromatography (AcOEt/PE 6:4, Rf = 0.4) as an pale yellow oil (0.6g, 55%). ^{1}H NMR ($CDCl_3$) δ 7.75-7.72 (m, 1H), 7.43-7.37 (m, 1H), 7.28-7.23 (m, 2H), 6.37 (br s, 1H), 4.12 (s, 1H), 4.11 (s, 1H), 4.1-4.04 (m,1H), 3.16-3.02 (m, 2H), 1.41 (s, 3H), 1.40 (s, 3H), 1.30 (d, J=6.3Hz, 3H); ^{13}C NMR ($CDCl_3$) δ 162.9, 140.0, 131.6, 130.7, 129.4, 127.3, 126.0, 78.9, 69.4, 67.8, 42.4, 28.5, 28.2, 24.4; IR (KBr) ν 3288, 2966, 2928, 2870, 1645, 1355, 1313, 1059, 744; Anal. calcd. for $C_{14}H_{19}NO_2$: C, 72.07; H, 8.21; N, 6.00. Found : C, 71.97; H, 8.12 ; N, 6.13 .



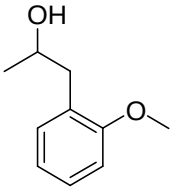
2-(2'Hydroxypropyl)-*N*-*tert*-butylbenzamide (3b)

Crude **3b** was prepared according to the general procedure 4 using Bu_4MgLi_2 as base and purified by flash chromatography flash chromatography (AcOEt/PE 3:7, Rf = 0.2) as a white solid (0.4g, 42%), mp = 99-100°C. ^{1}H NMR ($CDCl_3$) δ 7.40-7.34 (m, 2H), 7.26-7.20 (m, 2H), 6.19 (br s, 1H), 4.31 (d, *J*=4.8Hz, 1H), 4.08-3.97 (m,1H), 2.90-2.77 (m, 2H), 1.46 (s, 9H), 1.31 (d, *J*=6.3Hz, 3H); ^{13}C NMR ($CDCl_3$) δ 170.1, 138.0, 137.3, 130.8, 129.9, 127.3, 126.3, 69.0, 52.0, 42.1, 28.8, 24.3; IR (KBr) ν 3200, 3072, 2963, 2926, 1632, 1598, 1580, 1554, 1451, 1427, 1365, 1331, 1226, 1125, 745; Anal. calcd. for $C_{14}H_{21}NO_2$: C, 71.46; H, 8.99; N, 5.95. Found : C, 71.46; H, 9.03; N, 5.96.



2-(2'Hydroxypropyl)-*N*-cumylbenzamide (3c)

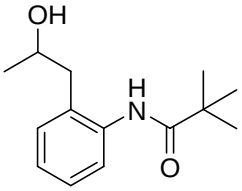
Crude **3c** was prepared according to the general procedure 4 using Bu_4MgLi_2 as base and purified by flash chromatography flash chromatography (AcOEt/PE 1:1, Rf = 0.4) as a white solid (0.7g, 61%), mp= 115-116°C. ^{1}H NMR ($CDCl_3$) δ 7.49-7.46 (m, 3H), 7.42-7.33 (m, 3H), 7.28-7.24 (m, 3H), 6.69 (br s, 1H), 4.00-3.96 (m, 2H), 2.90-2.77 (m, 2H), 1.83 (s, 3H), 1.80 (s, 3H) 1.26 (d, *J*=6.0Hz, 3H); ^{13}C NMR ($CDCl_3$) δ 169.6, 146.7, 137.5, 130.9, 130.1, 128.4, 127.4, 126.7, 126.4, 124.9, 69.1, 56.5, 42.1, 29.4, 29.0, 24.2; IR (KBr) ν 3345, 3059, 2965, 2927, 1610, 1413, 1309, 1194, 1017, 906, 751, 694, 561; Anal. calcd. for $C_{19}H_{23}NO_2$: C, 76.73; H, 7.80; N, 4.71. Found : C,76.70; H, 7.85; N, 3.69.



2-(2'-Hydroxypropyl)-anisole (3d)

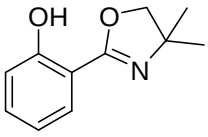
Crude **3d** was prepared according to the general procedure 4 using Bu_4MgLi_2 as base and purified by flash chromatography (AcOEt/PE 2:8, Rf = 0.3) as limpid oil (0.6g, 57%). All analyses are in accordance with those reported in

literature. [6]



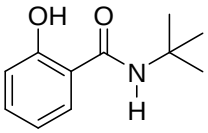
2-(2'-Hydroxypropyl)-pivaloylaminobenzene (3e)

Crude **3e** was prepared according to the general procedure 4 using Bu_4MgLi_2 as base and purified by flash chromatography flash chromatography (AcOEt/PE 2:8, Rf = 0.2) as a white solid (0.2g, 50%), mp 101°C. ^{1}H NMR ($CDCl_3$) 9.03 (br s, 1H), 7.82-7.79 (m, 1H), 7.26-7.21 (m, 1H), 7.13-7.04 (m, 2H), 4.18-4.10 (m, 1H), 2.80-2.63 (m, 2H), 2.10 (br s, 1H), 1.31 (s, 9H), 1.28 (s, 3H); ^{13}C NMR ($CDCl_3$) δ 177.6, 137.1, 131.1, 131.0, 126.9, 124.6, 124.5, 70.0, 40.8, 39.6, 27.8, 23.9; IR (KBr) ν3326, 2961, 2926, 2867, 1638, 1552, 1310, 1201, 1132, 1070, 799, 733, 678; Anal. calcd. for $C_{14}H_{21}NO_2$: C, 71.46; H, 8.99; N, 5.95. Found : C, 71.39 ; H, 8.99 ; N, 6.03.



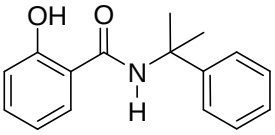
2-Hydroxy-2'-oxazolinylbenzene (8a)

Crude **8a** was prepared according to the general procedure 6 using Bu_3MgLi as base and purified by flash chromatography (AcOEt/PE 1:9, Rf = 0.5) as limpid oil (0.5g, 55%). All analyses are in accordance with those reported in literature.[7a]



2-Hydroxy-*N*-*tert*-butylbenzamide (8b)

Crude **8b** was prepared according to the general procedure 6 using Bu_4MgLi_2 as base and purified by flash chromatography (AcOEt/PE 1:9, Rf = 0.3) as a white solid (0.3g, 37%), mp= 82-83°C. ^{1}H NMR ($CDCl_3$) δ 12.50 (br s, 1H), 7.39-7.33 (m, 1H), 7.27 (dd, *J*=8.1Hz, *J*=1.5Hz, 1H), 6.96 (dd, *J*=8.4Hz, *J*=1.2Hz, 1H), 6.84-6.79 (m, 1H), 6.10 (br s, 1H), 1.48 (s, 9H); ^{13}C NMR ($CDCl_3$) δ 169.9, 161.7, 133.9, 125.4, 118.7, 118.5, 115.2, 52.2, 28.9; IR (KBr) ν 3343, 2969, 2722, 1606, 1573, 1500, 1457, 1390, 1364, 1325, 1231, 1157, 891, 754, 648; Anal. calcd. for $C_{11}H_{15}NO_2$: C, 68.37; H, 7.82; N, 7.25. Found : C, 68.13; H, 7.80; N, 7.17.



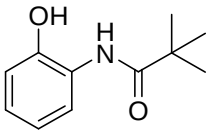
2-Hydroxy-*N*-cumylbenzamide (8c)

Crude **8c** was prepared according to the general procedure 6 using Bu_4MgLi_2 as base and purified by flash chromatography (AcOEt/PE 2:8, Rf = 0.3) as a white solid (0.2g, 23%), mp= 181-182°C. ^{1}H NMR ($CDCl_3$) δ 12.21 (br s, 1H), 7.46-7.34 (m, 6H), 7.30-7.24 (m, 1H), 6.96 (dd, *J*=7.8Hz, *J*=1.2Hz, 1H), 6.86 (ddd, *J*=8.4Hz, *J*=7.5Hz, *J*=12Hz, 1H), 6.57 (br s, 1H), 1.82 (s, 6H); ^{13}C NMR ($CDCl_3$) δ 169.4, 161.8, 146.3, 134.2, 128.7, 127.1, 125.5, 124.6, 118.8, 118.7, 114.8, 56.7, 29.4; IR (KBr) ν

[6] B. Erdelyi, A. Szabo, G. Seres, L. Birincsik, J. Ivanics, G. Szatzker, L. Poppe, Tetrahedron : Asym., 2006, **17**, 268-274.

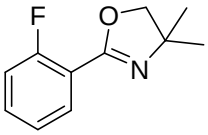
[7] (a) D.J. Berg, C. Zhou, T. Barclay, X. Fei, S. Feng, K. A. Ogilvie., R. A. Gossage, B. Twamley, M. Wood, Can. J. Chem., 2005, 449. (b) W.-M Dai, Y. K. Cheung, K. W.Tang, P. Y. Choi, S. L. Chung, Tetrahedron, 1995), **51**, 12263-12276.

3336, 3086, 2961, 2712, 1629, 1604, 1568, 1495, 1452, 1384, 1363, 1322, 1240, 1208, 1095, 757, 698; Anal. calcd. for $C_{16}H_{17}NO_2$: C, 75.27; H, 6.71; N, 5.49. Found : C, 75.18; H, 6.69; N, 5.14.



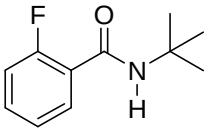
2-Hydroxypivaloylaminobenzene (8e)

Crude **8e** was prepared according to the general procedure 6 using Bu_4MgLi_2 as base and purified by flash chromatography (AcOEt/PE 1:9, Rf = 0.1) as a white solid (0.2g, 42%). All analyses are in accordance with those reported in literature [7b]



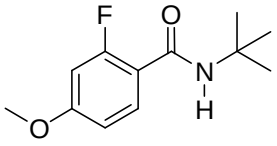
1-Fluoro-2-(4,4'-dimethyloxazolin-2-yl)benzene (9a)

Crude **9a** was prepared according to the general procedure 7 using Bu_3MgLi as base and purified by flash chromatography (AcOEt/PE 1:9, Rf = 0.1) as limpid oil (0.4g, 43%). All analyses are in accordance with those reported in literature. [8a]



2-Fluoro-*N*-*tert*-butylbenzamide (9b)

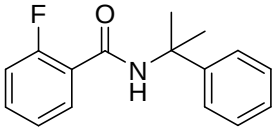
Crude **9b** was prepared according to the general procedure 7 using Bu_4MgLi_2 as base and purified by flash chromatography (AcOEt/PE 5:95, Rf = 0.2) as a yellow solid (0.4g, 59%), mp= 34-35°C. ^{1}H NMR ($CDCl_3$) δ 8.01 (ddd, *J*=9.6Hz, *J*=7.8Hz, *J*=1.8Hz, 1H), 7.43-7.36 (m, 1H), 7.20 (ddd, *J*=8.4Hz, *J*=7.8Hz, *J*=0.9Hz, 1H), 7.05 (ddd, *J*=12.3Hz, *J*=8.4Hz, *J*=0.9Hz, 1H), 6.58 (br s, 1H), 1.45 (s, 9H); ^{13}C NMR ($CDCl_3$) δ 162.3 (*J*=3.0Hz), 160.4 (*J*=245.8Hz), 132.9 (*J*=9.0Hz), 131.8 (*J*=2.3Hz), 124.7 (*J*=3.8Hz), 122.4 (*J*=12.1Hz), 115.9 (*J*=24.9Hz), 51.8, 28.9; ^{19}F NMR ($CDCl_3$) δ -113; IR (KBr) ν 3277, 2970, 1639, 1615, 1538, 1487, 1449, 1364, 1324, 1227, 754; Anal. calcd. for $C_{11}H_{14}FNO$: C, 67.67; H, 7.23; N, 7.17. Found : C, 67.62; H, 7.17; N, 7.06.



2-Fluoro-4-methoxy-*N*-*tert*-butylbenzamide (9c)

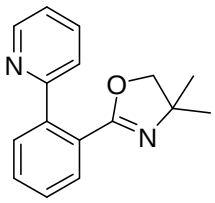
Crude **9c** was prepared according to the general procedure 7 using Bu_4MgLi_2 as base and purified by flash chromatography (AcOEt/PE 2:8, Rf = 0.2) as an yellow oil (0.5g, 65%). ^{1}H NMR ($CDCl_3$) δ 8.04-7.98 (m, 1H), 6.71 (dd, *J*=8.7Hz, *J*=2.4Hz, 1H), 6.59 (dd, *J*=14.1Hz, *J*=2.4Hz, 1H), 6.50 (br s, 1H), 3.83 (s, 3H), 1.46 (s, 9H); ^{13}C NMR ($CDCl_3$) δ 163.0 (*J*=6Hz), 162.2 (*J*=3.8Hz), 161.5 (*J*=263.9Hz), 132.9 (*J*=4.5Hz), 114.6 (*J*=12.1Hz), 110.5 (*J*=2.3Hz), 101.5 (*J*=29.4Hz), 55.8, 51.6, 28.9; ^{19}F NMR ($CDCl_3$) δ -111; IR (KBr) ν 3466, 2967, 1665, 1621, 1535, 1500, 1455, 1281, 1268, 1221, 1155, 1104; Anal. calcd. for $C_{12}H_{16}FNO_2$: C, 63.98; H, 7.16; N, 6.22. Found : C, 64.05; H, 7.13; N, 6.34.

[8] S. L., Sedinkin, N. P. Rath, E. B. Bauer, *J. Organomet. Chem.*, 2008, **693**, 3081-3091.



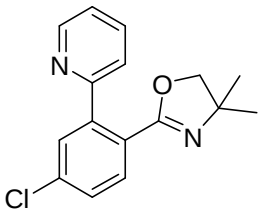
2-Fluoro-*N*-cumylbenzamide (9d)

Crude **9d** was prepared according to the general procedure 7 using Bu_4MgLi_2 as base and purified by flash chromatography (AcOEt/PE 1:9, Rf = 0.2) as a pale yellow solid (0.6g, 69%), mp= 49-50°C. ^{1}H NMR ($CDCl_3$) δ 8.02 (ddd, *J*=9.6Hz, *J*=7.8Hz, *J*=1.8Hz, 1H), 7.50-7.42 (m, 3H), 7.38-7.32 (m, 2H), 7.27-7.21 (m, 2H), 7.13 (ddd, *J*=11.4Hz, *J*=8.4Hz, *J*=0.9Hz, 1H), 7.09 (br s, 1H), 1.82 (s, 6H); ^{13}C NMR ($CDCl_3$) δ 161.9 (*J*=3.8Hz), 160.4 (*J*=245.8Hz), 146.7, 133.1 (*J*=9.0Hz), 131.9 (*J*=2.3Hz), 128.4, 126.6, 124.7 (*J*=3.8Hz), 124.7, 121.9 (*J*=12.1Hz), 115.9 (*J*=24.9Hz), 56.3, 29.3; ^{19}F NMR ($CDCl_3$) δ -116; IR (KBr) ν 3462, 3311, 3061, 2976, 1667, 1651, 1614, 1520, 1480, 1450, 1302, 757, 698; Anal. calcd. for $C_{16}H_{16}FNO$: C, 74.69; H, 6.27; N, 5.44. Found : C, 70.64; H, 6.25; N, 4.51.



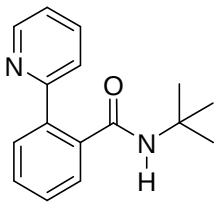
1-(4,4'-dimethyloxazolin-2-yl)-2-(pyridin-2-yl)benzene (4a)

Crude **4a** was prepared according to the general procedure 5 using Bu_3MgLi as base and purified by flash chromatography (AcOEt/PE 7:3, Rf = 0.2) as a red oil (0.5g, 40%). ^{1}H NMR ($CDCl_3$) δ 8.65 (d, *J*=4.8Hz, 1H), 7.78 (dd, *J*=7.8 Hz, *J*=1.5Hz, 1H), 7.71 (ddd, *J*=9.6Hz, *J*=7.8Hz, *J*=1.8Hz, 1H), 7.59 (dd, *J*=7.8Hz, *J*=1.5Hz, 1H), 7.52 (ddd, *J*=8.7Hz, *J*=7.2Hz, *J*=1.2Hz, 1H), 7.48–7.40 (m, 2H), 7.26–7.23 (m, 1H), 3.83 (s, 2H), 1.30 (s, 6H); ^{13}C NMR ($CDCl_3$) δ 163.1, 158.2, 148.6, 140.1, 135.6, 130.1, 129.8, 129.4, 127.8, 127.6, 122.7, 121.6, 79.0, 67.1, 27.6; IR (KBr) ν 2966, 1656, 1587, 1463, 1426, 1312, 1037, 750; Anal. calcd. for $C_{16}H_{16}N_2O$: C, 76.16; H, 6.39; N, 11.1. Found : C, 75.97; H, 6.35; N, 11.00.



1-Chloro-4-(4,4'-dimethyloxazolin-2-yl)-5-(pyridine-2-yl)benzene (4b)

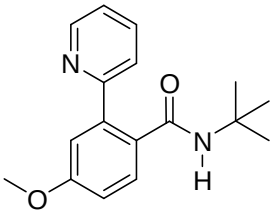
Crude **4b** was prepared according to the general procedure 5 using Bu_3MgLi as base and purified by flash chromatography (AcOEt/PE 1:1, Rf = 0.2) as an yellow solid (0.7g, 53%), mp = 94-95°C. ^{1}H NMR ($CDCl_3$) δ 8.67-8.64 (m, 1H), 7.76-7.70 (m, 2H), 7.59 (d, *J*=2.1Hz, 1H), 7.46-7.39 (m, 2H), 7.29–7.24 (m, 1H), 3.83 (s, 2H), 1.30 (s, 6H); ^{13}C NMR ($CDCl_3$) δ 157.2, 149.1, 142.1, 136.3, 135.9, 131.5, 129.9, 128.1, 126.5, 126.4, 123.0, 122.3, 79.4, 67.6, 27.9; IR (KBr) ν 2978, 2965, 2925, 1651, 1588, 1466, 1427, 1398, 1348, 1311, 1102, 1072, 1034, 842, 793, 756; Anal. calcd. for $C_{16}H_{15}ClN_2O$: C, 67.02; H, 5.29; N, 9.77. Found : C, 67.05; H, 5.27; N, 9.83.



2-(pyridin-2-yl)-*N*-*tert*-butylbenzamide (4c)

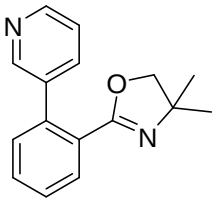
Crude **4c** was prepared according to the general procedure 5 using Bu_4MgLi_2 as base and purified by flash chromatography (AcOEt/PE 4:6, Rf = 0.2) as a white solid (0.3g, 36%), mp = 94-95°C. ^{1}H NMR ($CDCl_3$) δ 8.67-8.65 (m, 1H), 7.77-7.71 (m, 1H), 7.64-7.61 (m, 1H), 7.53-7.40 (m,

4H), 7.29 (dd, *J*=5.1Hz, *J*=1.2Hz, 1H), 5.67 (br s, 1H), 1.21 (s, 9H); ^{13}C NMR ($CDCl_3$) δ 169.0, 158.7, 149.2, 138.5, 137.6, 136.6, 129.8, 129.7, 128.7, 128.4, 124.3, 122.5, 51.65, 28.5; IR (KBr) 3306, 3047, 2962, 2924, 1638, 1590, 1546, 755; Anal. calcd. for $C_{16}H_{18}N_2O$: C, 75.56 ; H, 7.13 ; N, 11.01. Found : C, 75.56 ; H, 7.10 ; N, 11.06 .



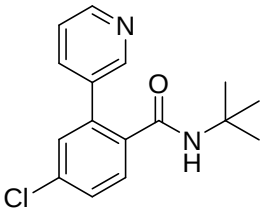
4-Methoxy-2-(pyridin-2-yl)-*N*-*tert*-butylbenzamide (4d)

Crude **4d** was prepared according to the general procedure 5 using Bu_4MgLi_2 as base and purified by flash chromatography (AcOEt/PE 1:1, Rf = 0.2) as an yellow solid (0.4g, 41%), mp = 99-100°C. ^{1}H NMR ($CDCl_3$) δ 8.66 (m, 1H), 7.74 (ddd, *J*=7.5Hz, *J*=7.5Hz, *J*=1.5Hz, 1H), 7.61 (d, *J*=8.4Hz, 1H), 7.47 (d, *J*=7.8Hz, 1H), 7.30 (ddd, *J*=7.5Hz, *J*=4.8Hz, *J*=0.9Hz, 1H), 6.99 (d, *J*=2.7Hz, 1H), 6.96 (dd, *J*=8.4Hz, *J*=2.7Hz, 1H), 5.61 (br s, 1H), 3.85 (s, 3H), 1.17 (s, 9H); ^{13}C NMR ($CDCl_3$) δ 168.3, 160.2, 158.5, 149.0, 140.0, 136.4, 130.2, 129.9, 124.1, 122.4, 114.6 114.1, 55.3, 51.2, 28.2; IR (KBr) 3315, 1642, 1542, 1479, 1319, 1293, 1216, 1180, 1034; Anal. calcd. for $C_{17}H_{20}N_2O_2$: C, 71.81 ; H, 7.09 ; N, 9.85. Found : C, 71.55 ; H, 6.91 ; N, 9.69.



1-(4,4'-dimethyloxazolin-2-yl)-2-(pyridin-3-yl)benzene (5a)

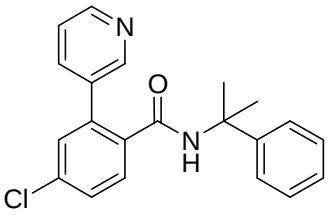
Crude **5a** was prepared according to the general procedure 5 using Bu_3MgLi as base and purified by flash chromatography (AcOEt/PE 6:4, Rf = 0.2) as an yellow solid (0.6g, 52%), mp = 96-97°C. ^{1}H NMR ($CDCl_3$) δ 8.60 (d, *J*=1.8Hz, 1H), 8.56 (dd, *J*=4.8Hz, *J*=1.5Hz, 1H), 7.80 (dd, *J*=7.5Hz, *J*=1.2Hz, 1H), 7.68 (ddd, *J*=7.8Hz, *J*=5.7Hz, *J*=1.8Hz, 1H), 7.51 (ddd, *J*=9Hz, *J*=7.5Hz, *J*=1.5Hz, 1H), 7.41 (ddd, *J*=9Hz, *J*=7.5Hz, *J*=1.5Hz, 1H), 7.36–7.28 (m, 2H), 3.82 (s, 2H), 1.26 (s, 6H); ^{13}C NMR ($CDCl_3$) δ 163.1, 149.3, 148.4, 138.2, 137.0, 135.7, 130.8, 130.5, 130.4, 128.1, 128.0, 122.9, 79.4, 67.8, 28.1; IR (KBr) ν 2961, 2926, 2885, 1651, 1591, 1473, 1437, 1413, 1348, 1300, 1068, 1039, 1028, 968, 816, 776, 698; Anal. calcd. for $C_{16}H_{16}N_2O$: C, 76.16; H, 6.39; N, 11.1. Found : C, 76.10; H, 6.50; N, 11.87.



4-chloro-2-(pyridin-3-yl)-*N*-*tert*-butylbenzamide (5b)

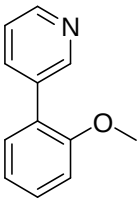
Crude **5b** was prepared according to the general procedure 5 using Bu_4MgLi_2 as base and purified by flash chromatography (AcOEt/PE 1:1, Rf = 0.3) as an yellow solid (0.5g, 52%ρ), mp = 172-173°C. ^{1}H NMR ($CDCl_3$) δ 8.67-8.65 (m, 2H), 7.78-7.75 (m, 1H), 7.59(d, 8.4Hz, 1H), 7.42 (dd, J=8.1Hz, J=2.1Hz, 1H), 7.37-7.35 (m, 2H), 5.08 (br s, 1H), 1.18 (s, 9H); ^{13}C NMR ($CDCl_3$) δ 167.3, 149.3, 149.1, 137.5, 136.2, 135.9, 135.8, 134.9, 130.0 (2C), 128.4, 123.3, 52.0, 28.4. IR (KBr) 3220, 3055, 1633, 1590, 1563, 1474, 1455, 1411, 1362, 1324,

1219, 1097, 1014, 848, 708 ; Anal. calcd. for $C_{16}H_{17}ClN_2O$: C, 66.55 ; H, 5.93 ; N, 9.7. Found : C, 66.13 ;H, 6.01 ;N, 9.65 .



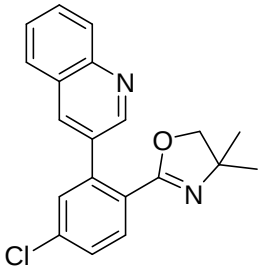
4-Chloro-2-(pyridin-3-yl)- *N*-cumylbenzamide (5c)

Crude **5c** was prepared according to the general procedure 5 using Bu_4MgLi_2 as base and purified by flash chromatography (AcOEt/Pe 3:7, Rf = 0.1) as an yellow solid (0.7g, 58%), mp= 181-182°C. ^{1}H NMR ($CDCl_3$) δ 8.70 (m, 1H), 7.77 (d, J=7.8Hz, 1H), 7.61 (d, J=8.1Hz, 1H), 7.42 (ddd, J=10.5Hz, J=8.4Hz, J=2.1Hz, 1H), 7.36-7.35 (m, 2H), 7.29-7.20 (m, 4H), 7.11-7.08 (m, 2H), 5.54 (br s, 1H), 1.56 (s, 6H); ^{13}C NMR ($CDCl_3$) δ 167.0, 149.3, 149.2, 146.3, 137.6, 136.4, 136.0, 135.6, 130.1, 130.0, 128.5, 128.4, 126.9, 124.6, 56.7, 28.2; IR (KBr) 3285, 1643, 1542, 1314, 764, 701, 558; Anal. calcd. for $C_{21}H_{19}ClN_2O$: C, 71.89; H, 5.46 ; N, 7.98. Found : C, 72.00; H, 5.43; N, 7.96.



2-(3'-pyridyl)-anisole (5d)

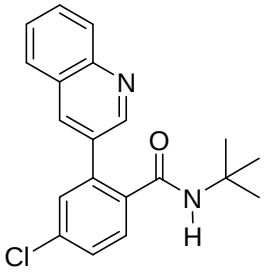
Crude **5b** was prepared according to the general procedure 5 using Bu_4MgLi_2 as base and purified by flash chromatography (AcOEt/PE 2:8, Rf = 0.2) as a limpid oil (0.6g, 51%). All analyses are in accordance with those reported in literature.[9a]



4-Chloro-1-(4,4'-dimethyloxazolin-2-yl)-2-(quinolin-3-yl)benzene (6a)

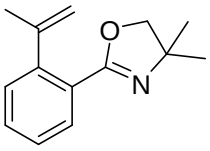
Crude **6a** was prepared according to the general procedure 5 using Bu_3MgLi as base and purified by flash chromatography (AcOEt/PE 1:1, Rf = 0.4) as a white solid (1g, 62%), mp = 92-93°C. ^{1}H NMR ($CDCl_3$) δ 8.86 (d, *J*=2.1Hz, 1H), 8.14-8.11 (m, 2H), 7.85-7.81 (m, 2H), 7.76–7.70 (m, 1H), 7.59-7.54 (m, 1H), 7.46–7.41 (m, 2H), 3.79 (s, 2H), 1.24 (s, 6H); ^{13}C NMR ($CDCl_3$) δ 162.1, 150.6, 147.3, 140.0, 136.9, 134.5, 133.2, 132.0, 130.9, 129.8, 129.4, 128.2, 128.1, 127.7, 127.1, 126.6, 79.5, 67.9, 28.1; IR (KBr) 2964, 1667, 1305, 1099, 1037, 898, 745; Anal. calcd. for $C_{20}H_{17}ClN_2O$: C, 71.32; H, 5.09; N, 8.32. Found : C, 70.97; H, 5.11; N, 8.33.

[9] (a) A.-S. Rebstock, F. Mongin,; F. Trecourt, G. Queguiner, Org. Biomol. Chem., 2003, **1**, 3064-3068. (b) S.R. Wilson, D.T. Mao, H. N. Khatri, Synthetic Commun., 1980, **10**, 17-23. (c) H. Zhi, A. K. Yudin, Org. Lett., 2006, **8**, 5829-5832. (d) A. Fryszkowska, K. Fisher, J.M. Gardiner, J.M. Stephens, J. Org. Chem. 2008, **73**, 3295-4298



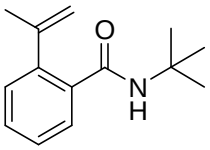
4-Chloro-2-(quinolin-3-yl)-*N*-*tert*-butylbenzamide (6b)

Crude **6b**was prepared according to the general procedure 5 using Bu_4MgLi_2 as base and purified by flash chromatography (AcOEt/PE 3:7, Rf = 0.2) as an yellow solid (0.5g, 42%), mp = 171-172°C. ^{1}H NMR ($CDCl_3$) δ 8.95 (d, *J*=2.4Hz, 1H), 8.24 (d, *J*=2.1Hz, 1H), 8.16 (d, *J*=8.4Hz, 1H), 7.85 (d, *J*=8.4Hz, 1H), 7.80-7.75 (m, 1H), 7.65–7.58 (m, 2H), 7.47–7.43 (m, 2H), 5.18 (br s, 1H), 1.12 (s, 9H); ^{13}C NMR ($CDCl_3$) δ 167.5, 150.0, 147.3, 137.5 136.0, 135.9, 135.3, 131.9, 130.2, 130.1, 130.0, 129.2, 128.3, 128.0, 127.4, 127.4, 51.9, 28.3; IR (KBr) 3246, 2967, 1664, 1593, 1547, 1455, 1310, 1227, 759 ; Anal. calcd. for $C_{20}H_{19}ClN_2O$: C, 70.90; H, 5.65; N, 8.27. Found : C, 70.89; H, 5.67; N, 8.25 .



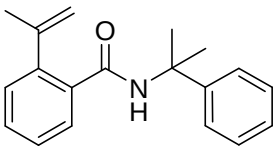
1-(4,4'-dimethyloxazolin-2-yl)-2-propenyl-benzene (7a)

Crude **7a** was prepared according to the general procedure 5 using Bu_4MgLi_2 as base and was purified by flash chromatography to give a mixture of **7a** and the starting material (3:2, 66%). All analyses are in accordance with those reported in literature.[9b]



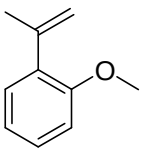
2-Propenyl-*N*-*tert*-butylbenzamide (7b)

Crude **7b** was prepared according to the general procedure 5 using Bu_4MgLi_2 as base and purified by flash chromatography (AcOEt/PE 3:7, Rf = 0.18) as an white solid (0.5g, 67%), mp = 77-78°C. All analyses are in accordance with those reported in literature.[9c]



2-Propenyl-*N*-cumylbenzamide (7c)

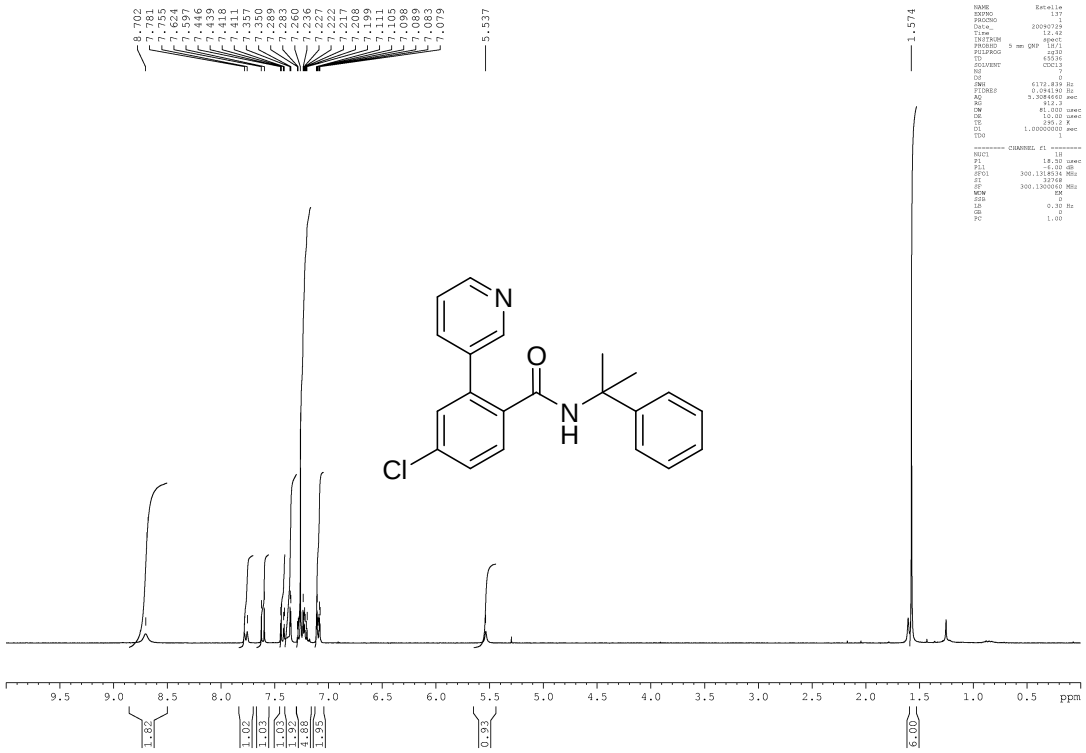
Crude **7c** was prepared according to the general procedure 5 using Bu_4MgLi_2 as base and purified by flash chromatography (AcOEt/PE 2:8, Rf = 0.4) as a white solid (0.7, 67%), mp = 100-101°C. ^{1}H NMR ($CDCl_3$) δ 7.67-7.64 (m, 1H), 7.46-7.43 (m, 2H), 7.40-7.28 (m, 4H), 7.25-7.18 (m, 3H), 6.48 (br s, 1H), 5.23 (s, 1H), 5.09 (s, 1H), 2.12 (s, 3H), 1.80 (s, 6H); ^{13}C NMR ($CDCl_3$) δ 167.9, 146.7, 146.5, 141.7, 134.8, 130.1, 128.8, 128.6, 128.4, 127.5, 126.8, 124.9, 115.9, 56.4, 28.5, 24.8; IR (KBr) 3275, 2972, 1644, 1532, 1316, 890, 765, 699; Anal. calcd. for $C_{19}H_{21}NO$: C, 81.68 ; H, 7.58 ; N, 5.01. Found : C, 81.70; H, 7.54; N, 5.03.

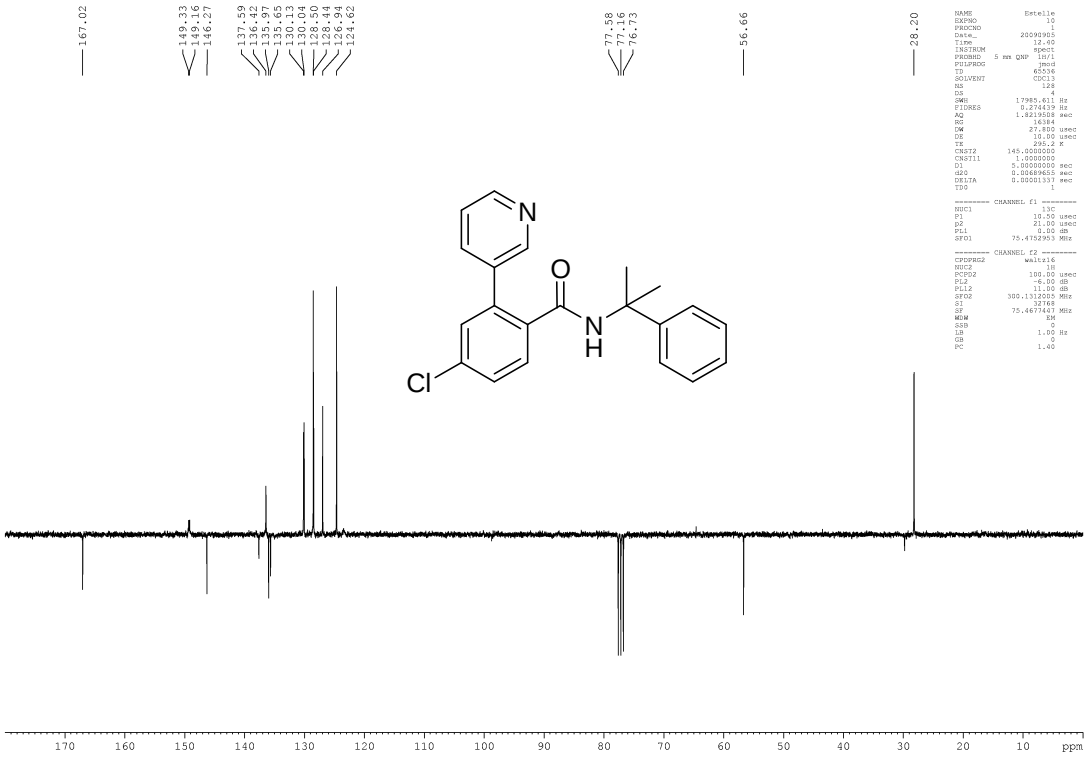


2-Propenyl-anisole 7d

Crude **7d** was prepared according to the general procedure 5 using Bu_4MgLi_2 as base and purified by flash chromatography (AcOEt/PE 3:7, Rf = 0.18) as a limpid oil (0.6g, 66%). All analyses are in accordance with those reported in literature. [9d]

^{1}H, ^{13}C and ^{19}F NMR Spectra of Product



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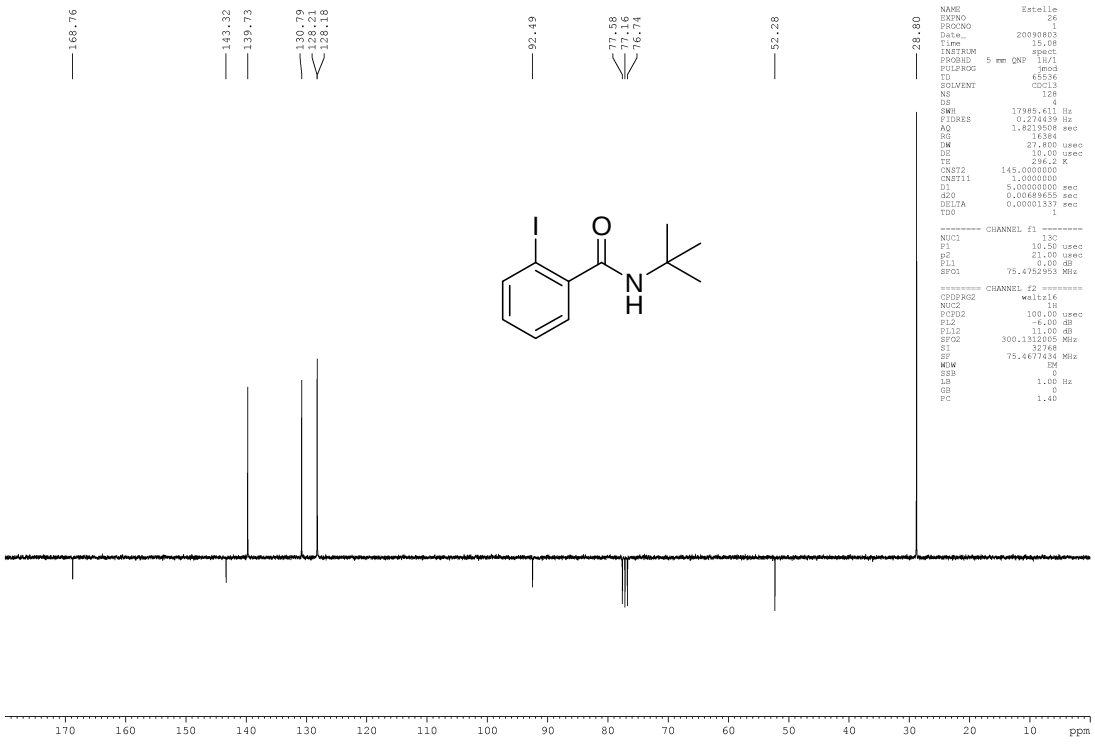
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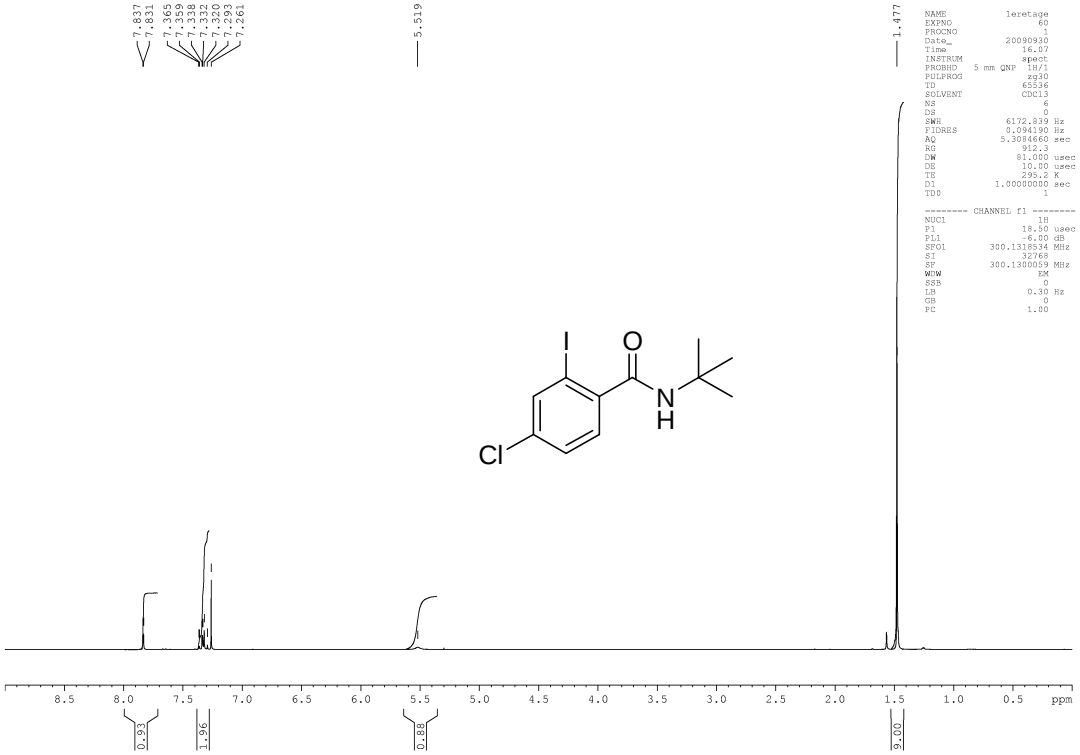
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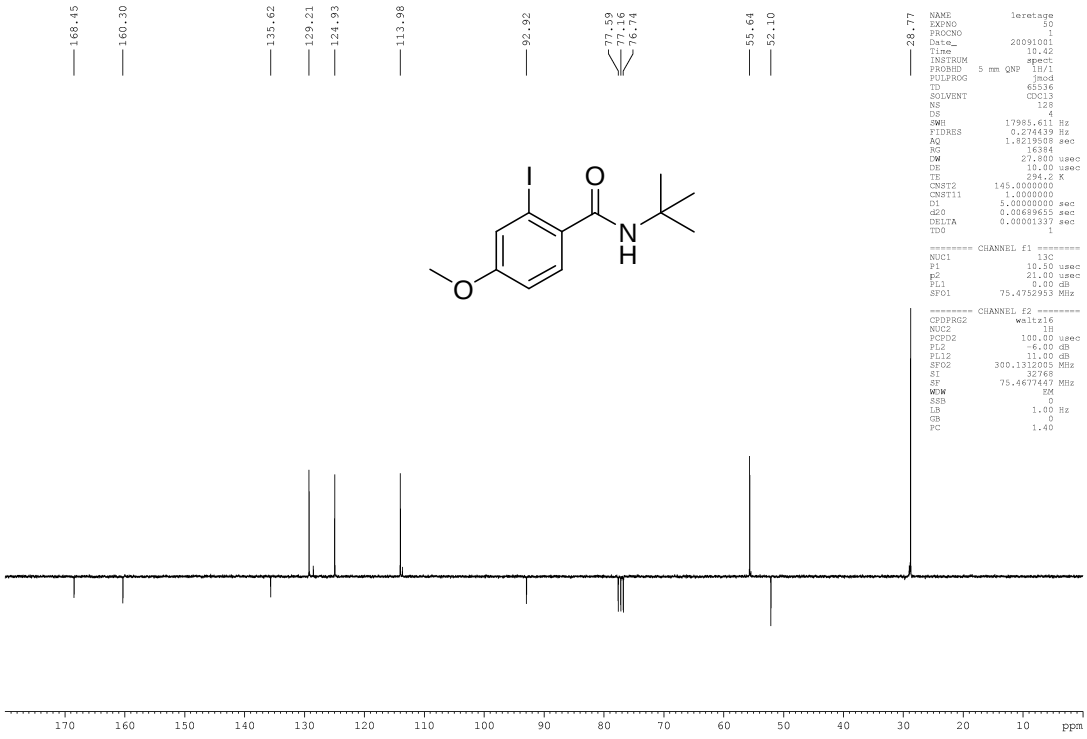
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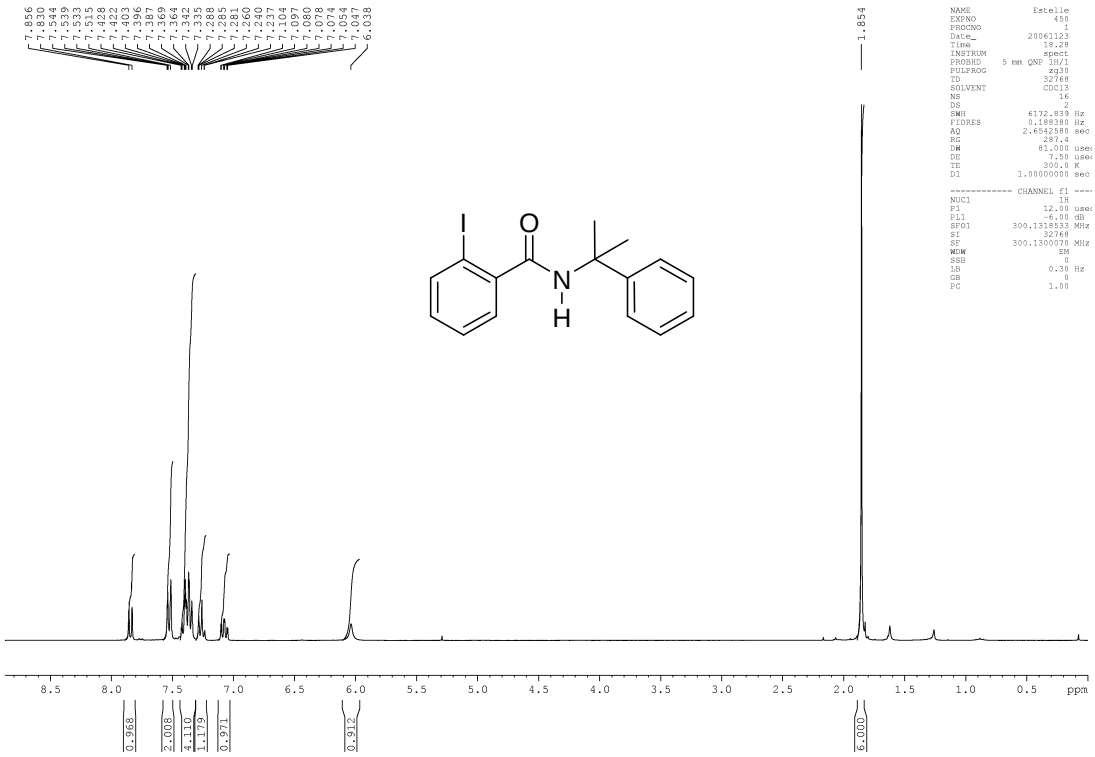
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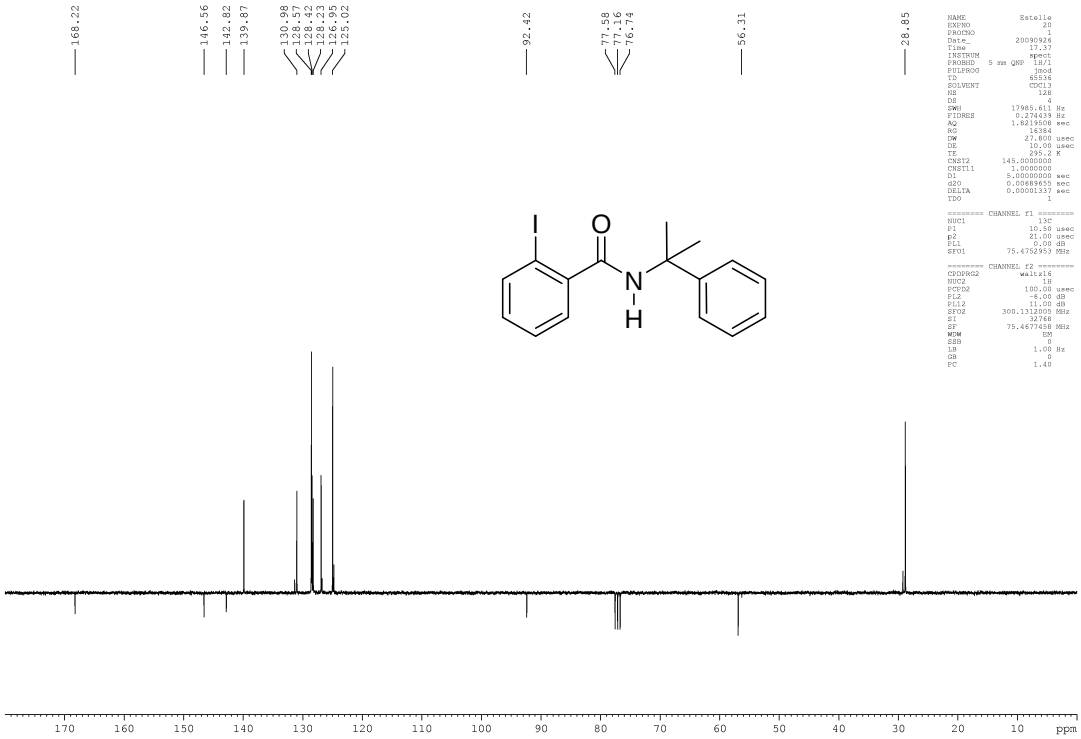
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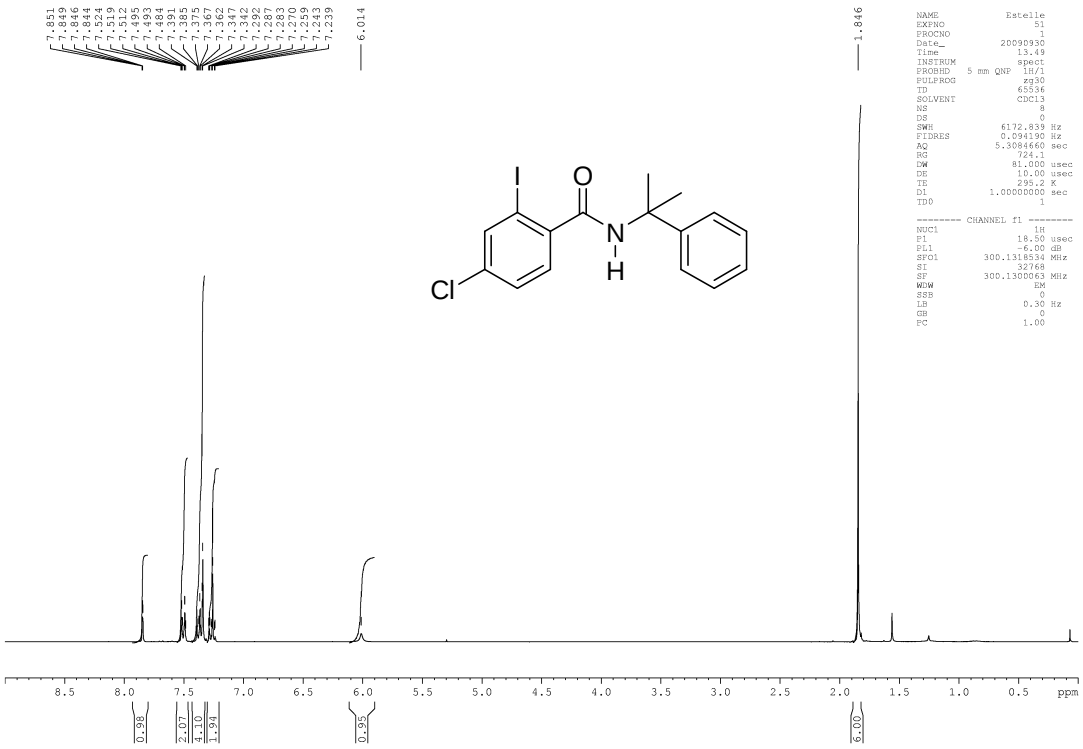


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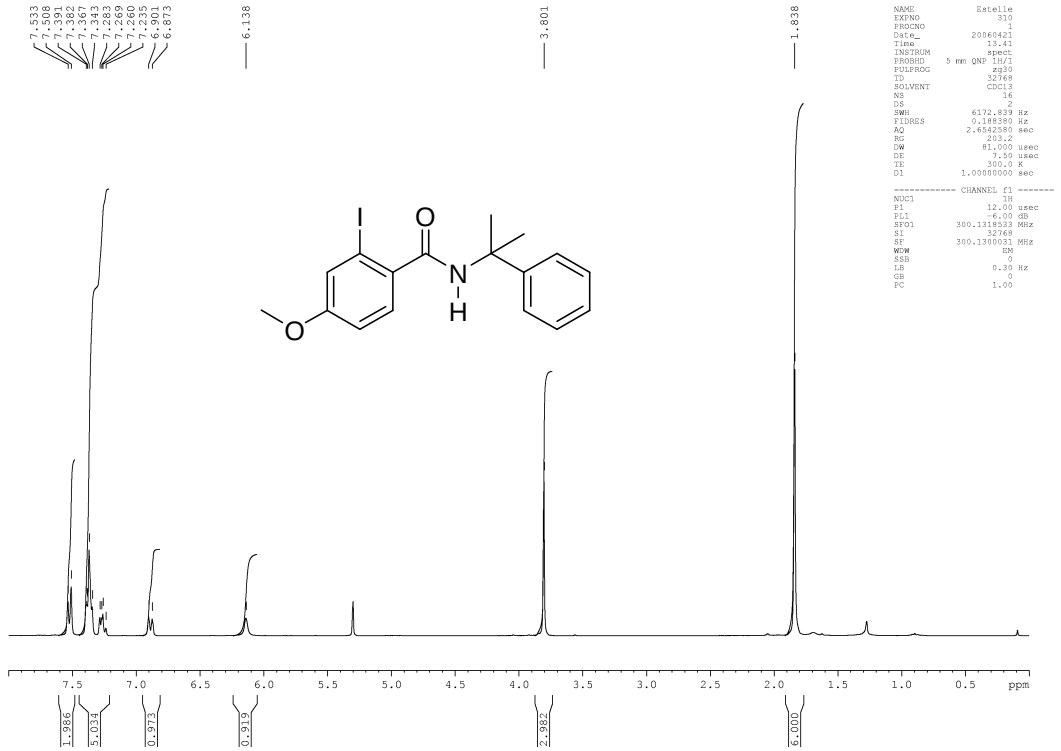


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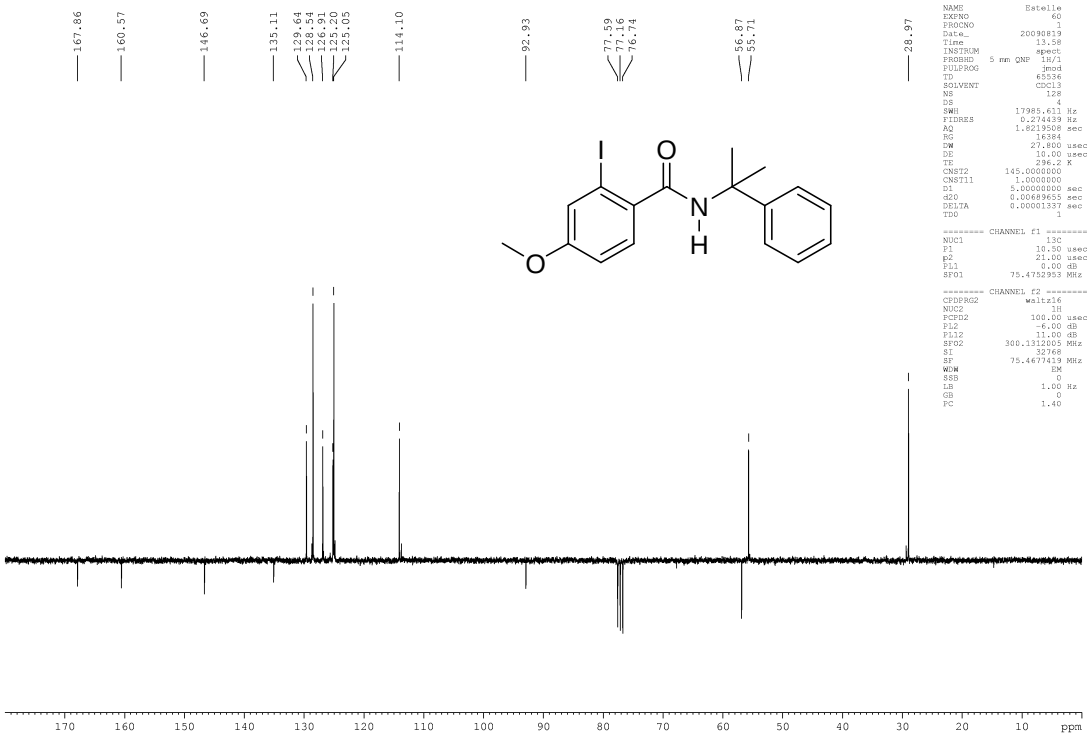
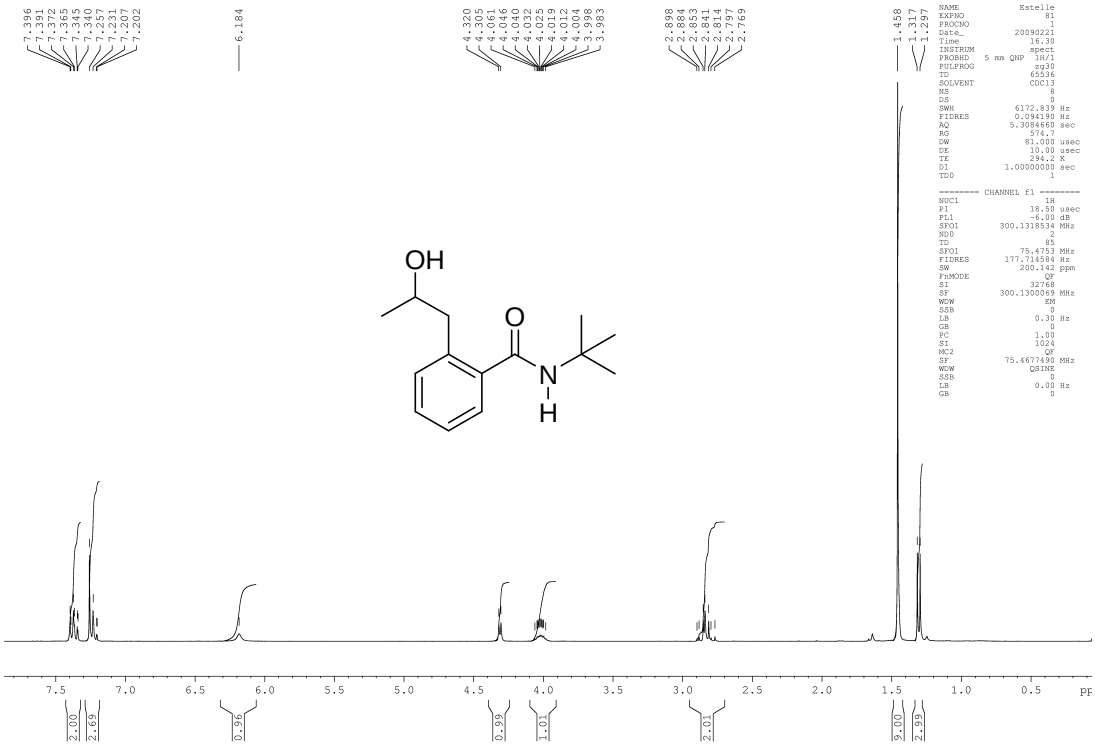
NUC1                  1H
P1                 12.00 usec
PL1                -6.00 dB
SFO1          300.1318533 MHz
SI                 32768
SF            300.1300031 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

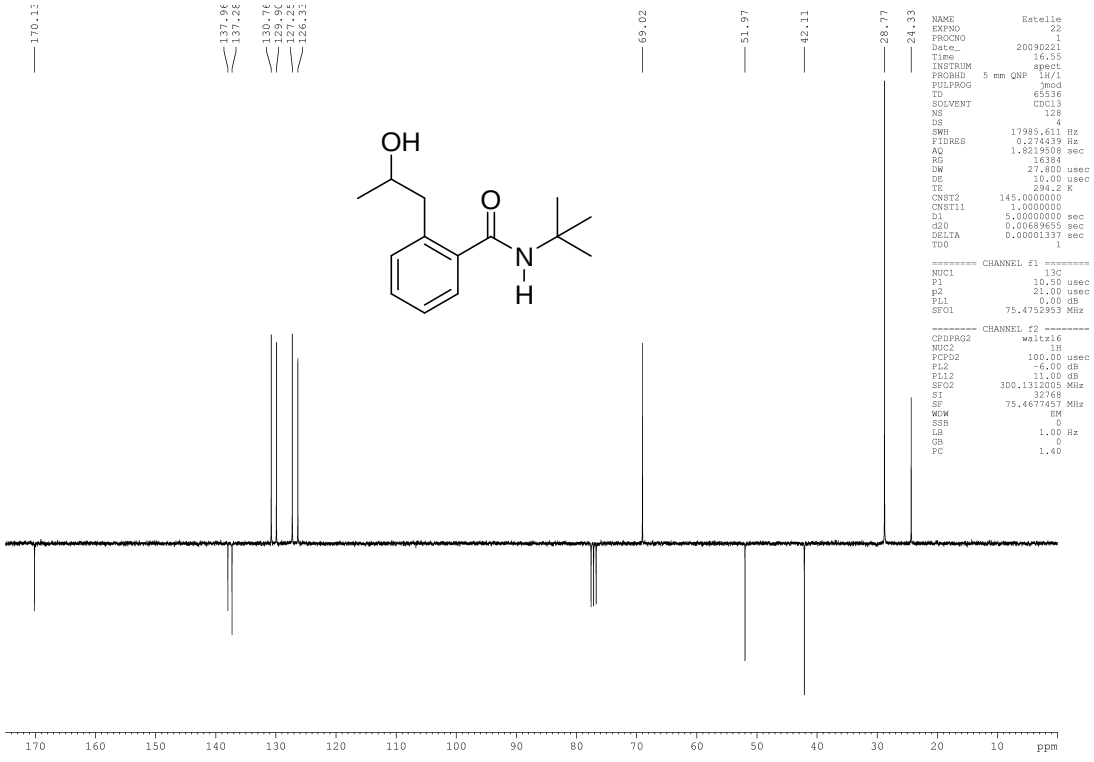


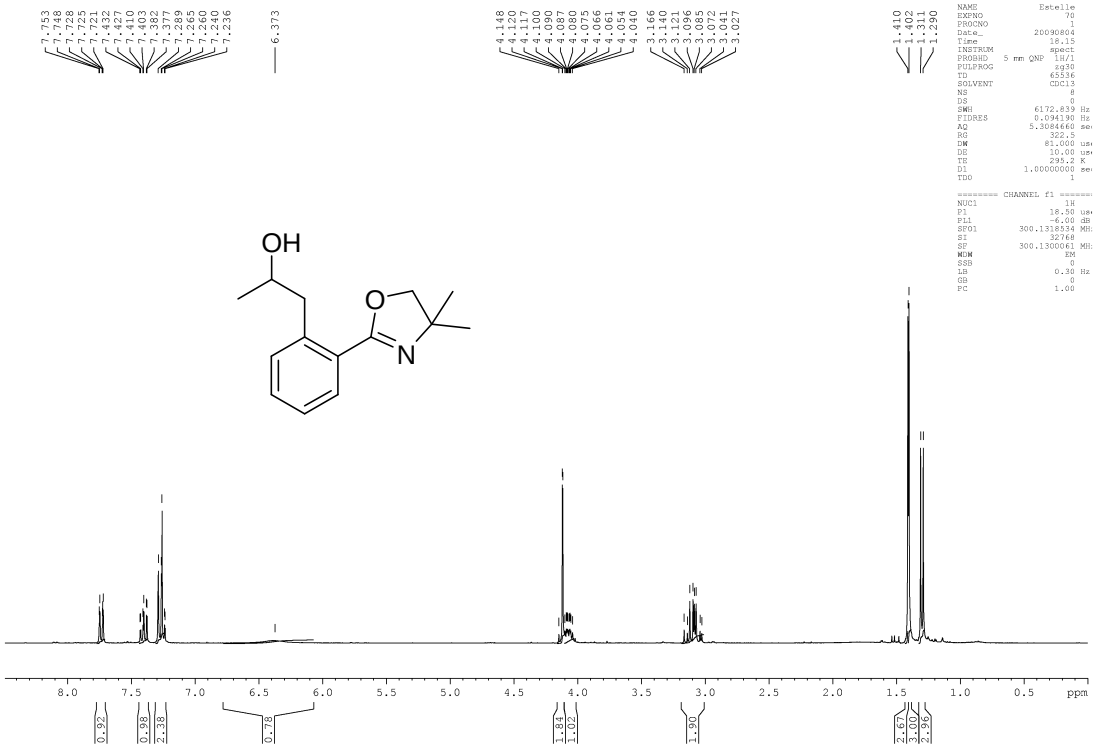

```
NAME              Estelle
EXPNO                  60
PROCNO                  1
Date_            20090819
Time                13.58
INSTRUM             spect
PROBHD   5 mm QNP 1H/1
PULPROG             zgpg
TD                 65536
SOLVENT            CDCl3
NS                   128
DS                     4
SWH             17985.611 Hz
FIDRES          0.274439 Hz
AQ              1.8219508 sec
RG                 16384
DW                 27.800 usec
DE                 10.00 usec
TE                296.2 K
CNST2          145.0000000
CNST11           1.0000000
D1               5.00000000 sec
d20              0.00689655 sec
DELTA            0.00001337 sec
TD0                    1

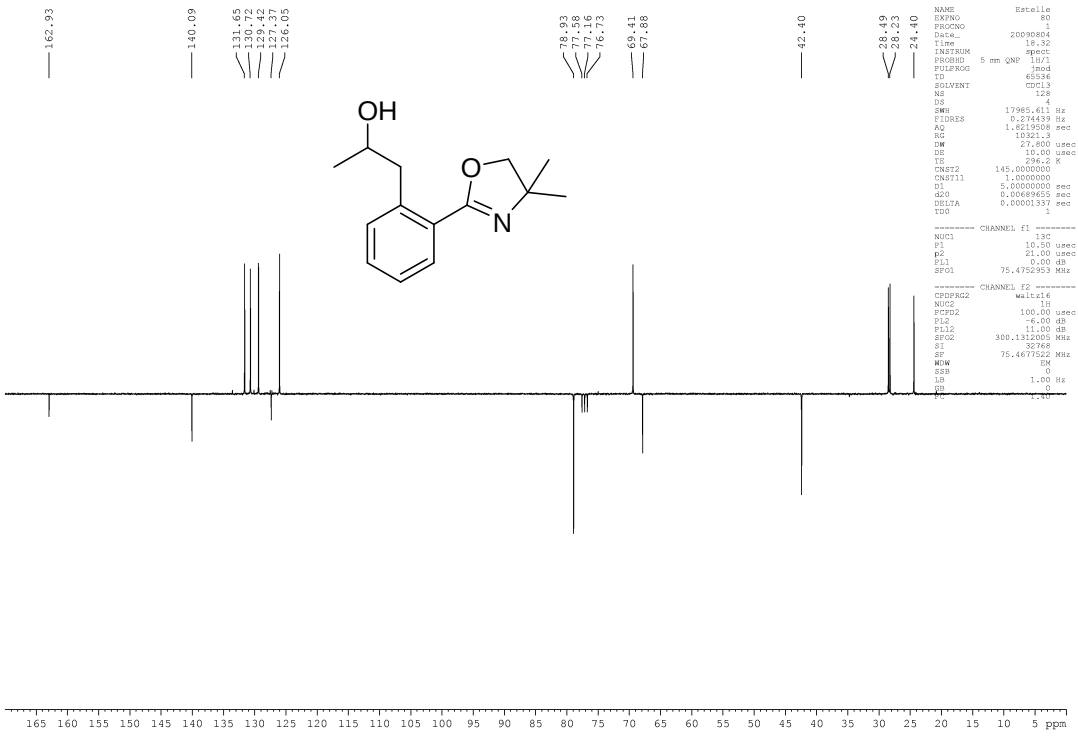
======== CHANNEL f1 ========
NUC1                  13C
P1                 10.50 usec
p2                 21.00 usec
PL1                0.00 dB
SFO1          75.4752903 MHz

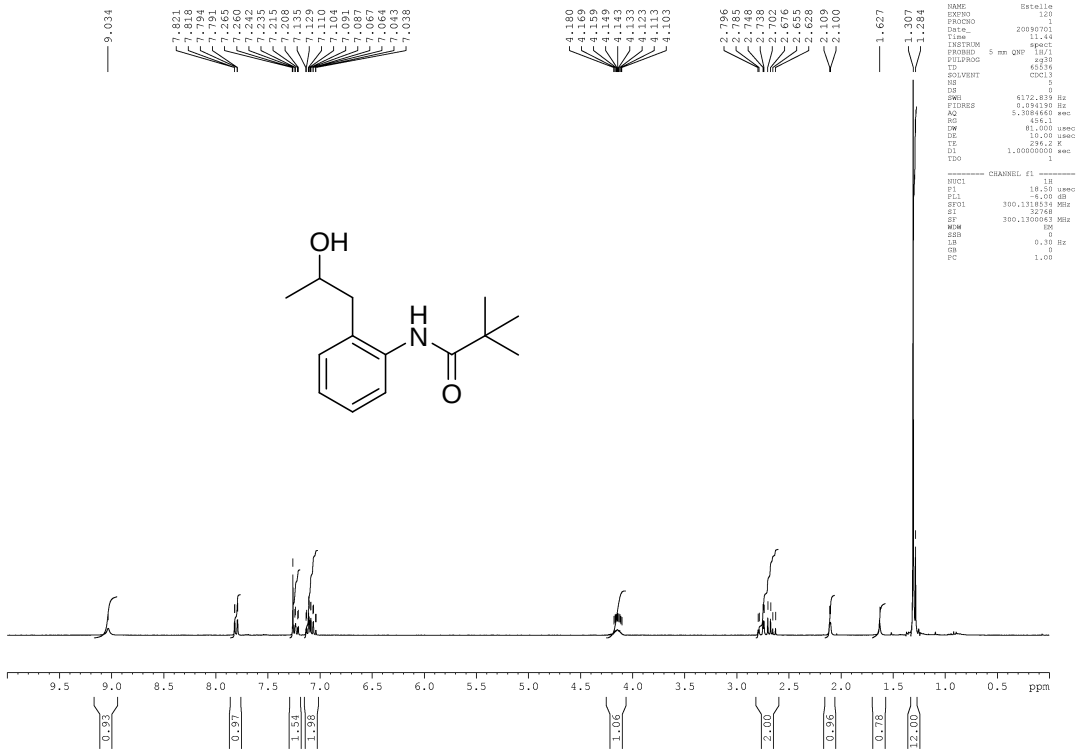
======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                  1H
PCPD2             100.00 usec
PL2                -6.00 dB
PL12               11.00 dB
SFO2          300.1312005 MHz
SI                 32768
SF            75.4677419 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

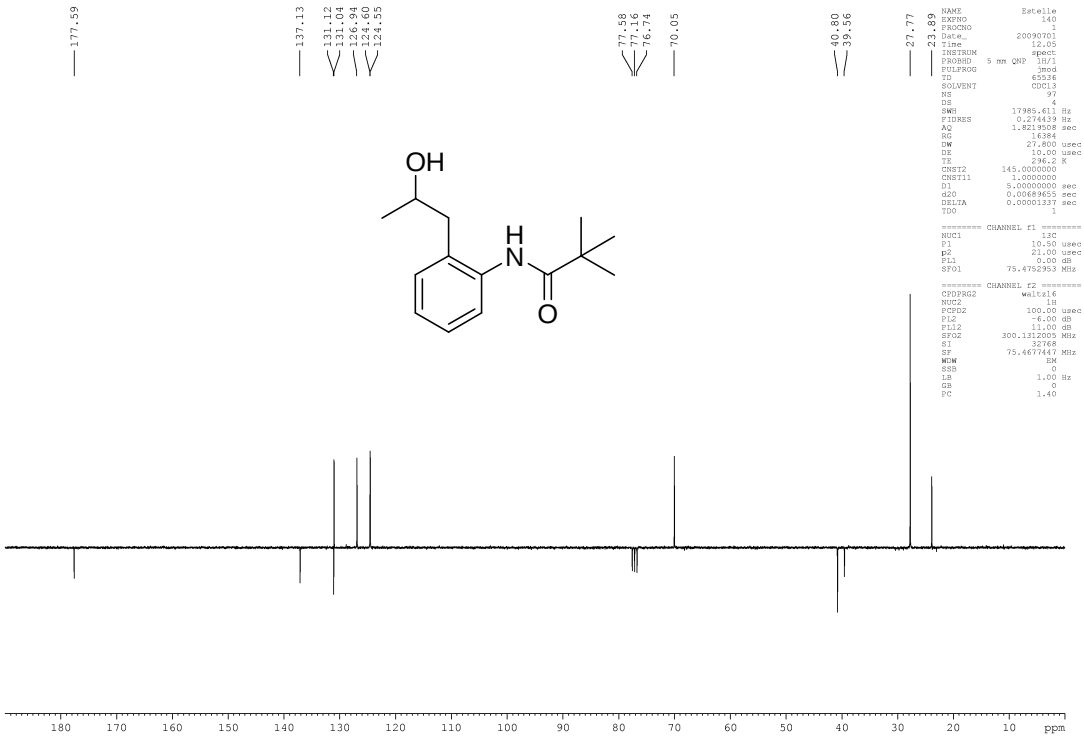

7.396
7.391
7.372
7.365
7.345
7.340
7.257
7.231
7.207
7.202
6.184
4.320
4.305
4.061
4.046
4.040
4.032
4.025
4.019
4.012
4.004
3.998
3.983
2.808
2.804
2.783
2.781
2.814
2.797
2.769
1.458
1.317
1.297
OH
O
N
H
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
2.00
2.69
0.96
0.99
1.01
2.01
9.00
2.99

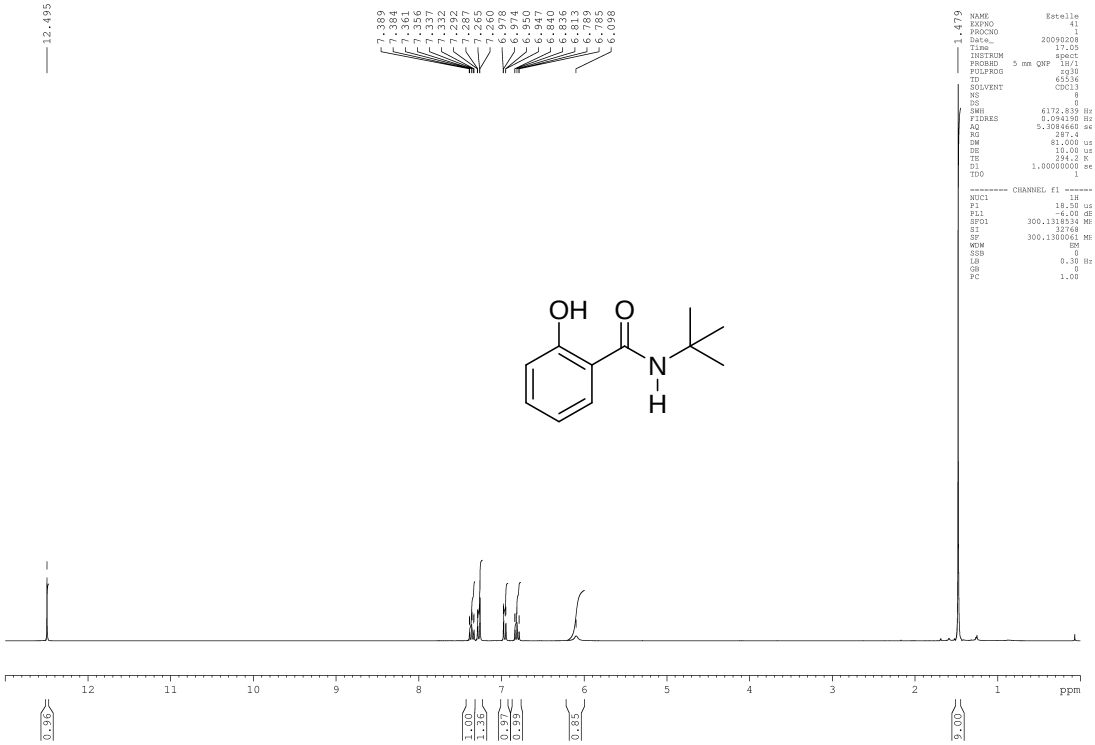

170.11
137.96
137.28
130.76
129.96
127.25
126.33
69.02
51.97
42.11
28.77
24.33
OH
O
N
H
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

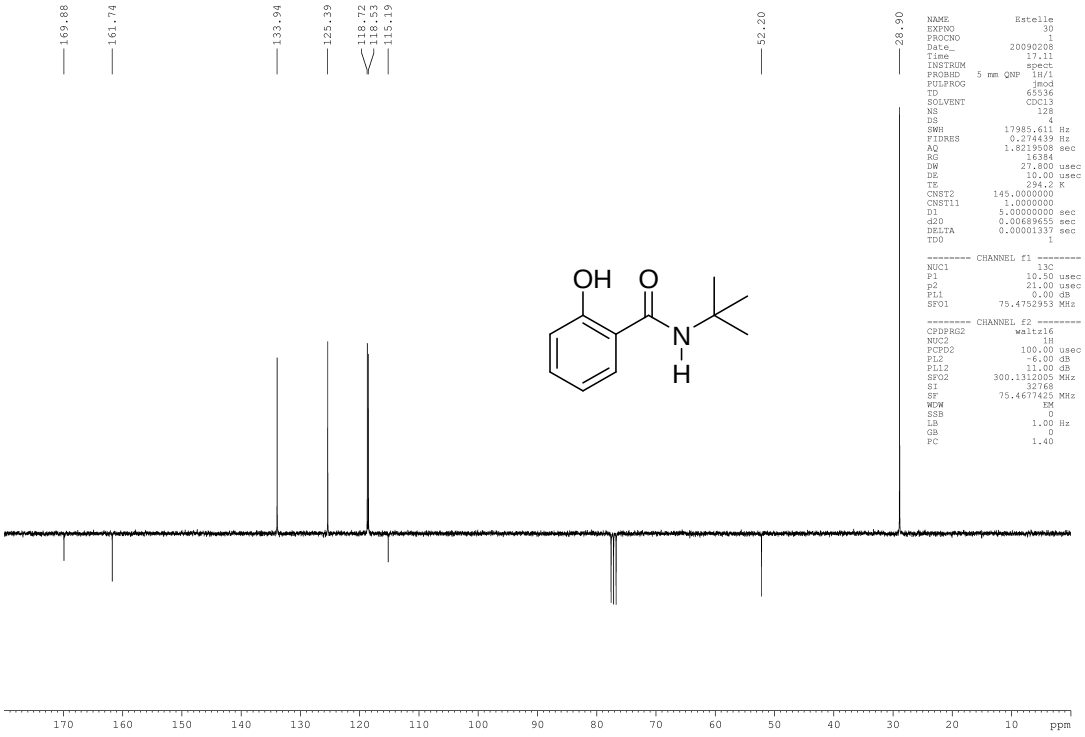

7.753
7.748
7.728
7.725
7.721
7.432
7.427
7.410
7.403
7.382
7.377
7.289
7.265
7.260
7.240
7.236
6.373
4.148
4.120
4.117
4.100
4.090
4.087
4.080
4.075
4.066
4.061
4.054
4.040
3.166
3.140
3.121
3.096
3.085
3.072
3.041
3.027
1.410
1.402
1.311
1.290
OH
O
N
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
0.92
0.98
2.38
0.78
1.84
1.02
1.90
2.67
3.00
2.96

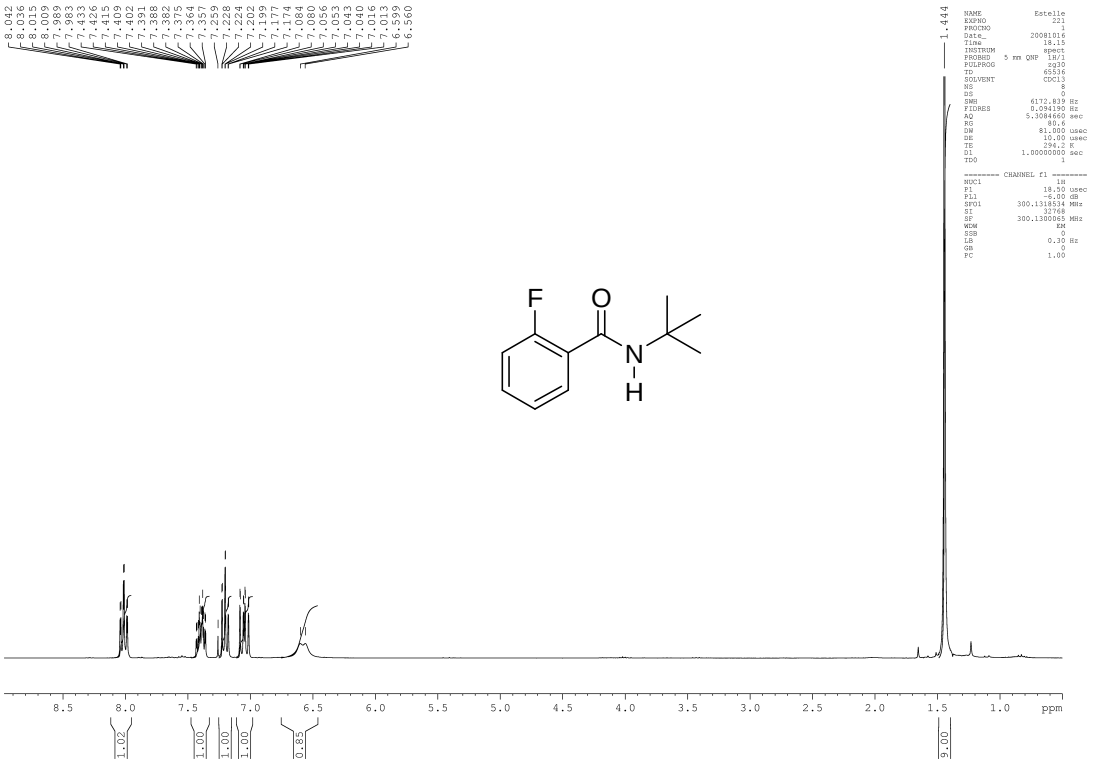

162.93
140.09
131.65
130.72
129.42
129.37
126.05
78.93
77.58
77.16
76.73
69.41
67.88
42.40
28.49
28.23
24.40
OH
O
N
165 160 155 150 145 140 135 130 125 120 115 110 105 100 95 90 85 80 75 70 65 60 55 50 45 40 35 30 25 20 15 10 5 ppm

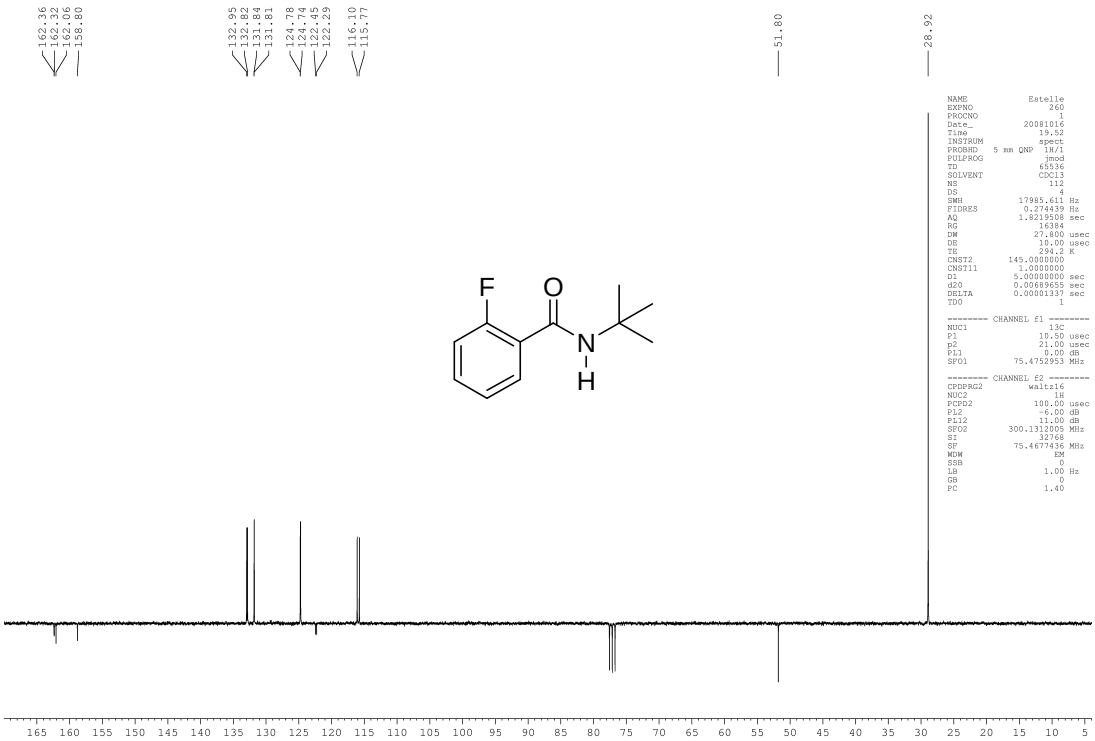

9.034
7.821
7.818
7.794
7.791
7.265
7.260
7.242
7.235
7.215
7.208
7.135
7.129
7.110
7.104
7.091
7.087
7.067
7.064
7.043
7.038
4.180
4.169
4.159
4.149
4.143
4.133
4.123
4.113
4.103
2.796
2.785
2.748
2.738
2.702
2.676
2.655
2.628
2.109
2.100
1.627
1.307
1.284
OH
H
N
O
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
0.93
0.97
1.54
1.98
1.06
2.00
0.96
0.78
12.00

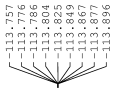

177.59
137.13
131.12
131.04
126.94
124.60
124.55
77.58
77.16
76.74
70.05
40.80
39.56
27.77
23.89
OH
H
N
O
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

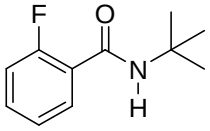

12.495
7.389
7.384
7.361
7.356
7.337
7.332
7.322
7.287
7.265
7.260
6.978
6.974
6.950
6.947
6.840
6.836
6.813
6.789
6.785
6.098
1.479
OH
O
N
H
ppm
0.96
1.00
1.36
0.97
0.99
0.85
9.00


169.88
161.74
133.94
125.39
118.72
118.53
115.19
52.20
28.90
OH
O
N
H
ppm


8.042
8.036
8.015
8.009
7.989
7.983
7.433
7.426
7.415
7.409
7.402
7.391
7.388
7.382
7.375
7.364
7.259
7.228
7.224
7.202
7.199
7.177
7.174
7.084
7.080
7.056
7.053
7.043
7.040
7.016
7.013
6.599
6.560
1.444
F
O
N
H
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
ppm
1.02
1.00
1.00
1.00
0.85
9.00


162.36
162.32
162.06
158.80
132.95
132.82
131.84
131.81
124.78
124.74
122.45
122.29
116.10
115.77
51.80
28.92
F
O
N
H
165
160
155
150
145
140
135
130
125
120
115
110
105
100
95
90
85
80
75
70
65
60
55
50
45
40
35
30
25
20
15
10
5



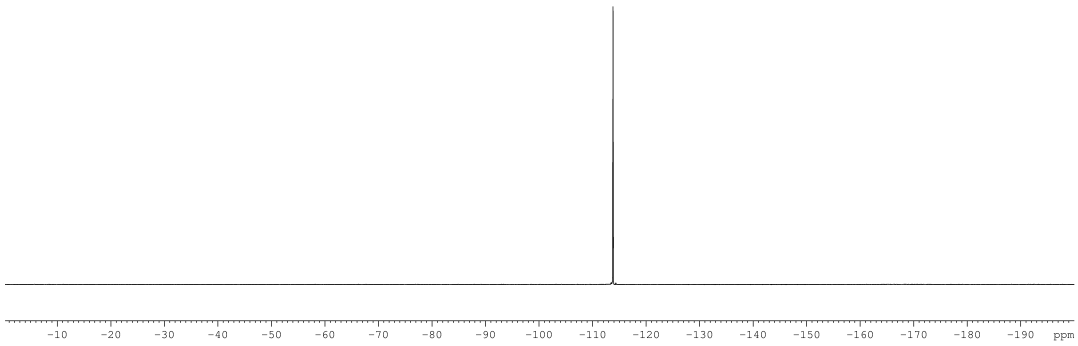


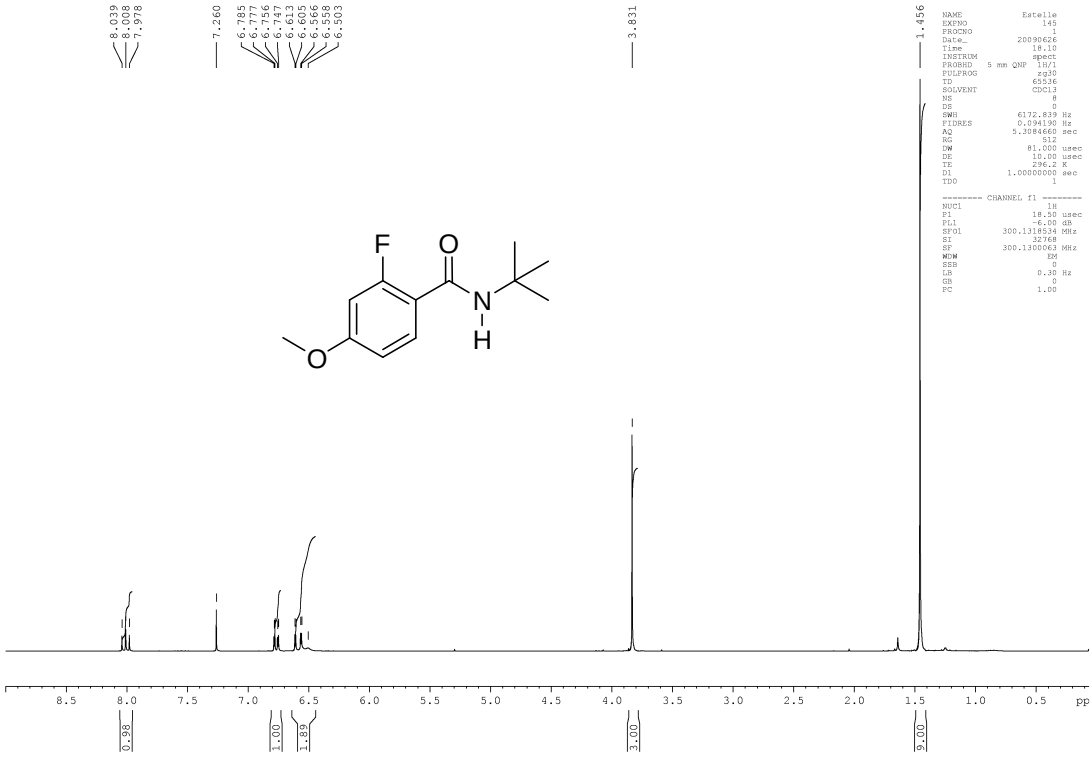

```
Current Data Parameters
NAME            Apr06-2009
EXPNO                  210
PROCNO                   1

F2 - Acquisition Parameters
Date_           20090406
Time                12.03
INSTRUM             spect
PROBHD    5 mm QNP  1H/1
PULPROG             zgflqn
TD                  131072
SOLVENT             CDCl3
NS                      16
DS                       4
SWH             67567.570 Hz
FIDRES          0.515500 Hz
AQ              0.9699828 sec
RG                   574.7
DW                7.400 usec
DE                10.00 usec
TE                295.2 K
D1           1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                   19F
P1                10.90 usec
PL1               -6.00 dB
SFO1         282.3761148 MHz

F2 - Processing parameters
SI                   65536
SF          282.4043550 MHz
WDW                     EM
SSB                      0
LB                0.30 Hz
GB                       0
PC                1.00
```

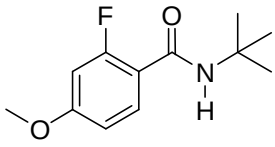


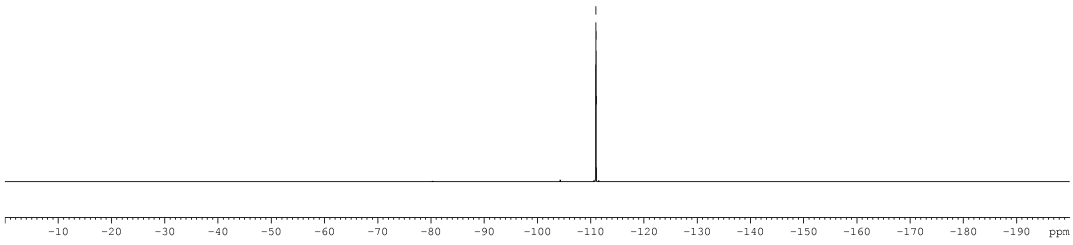

8.039
8.008
7.978
7.260
6.785
6.777
6.756
6.747
6.613
6.605
6.566
6.558
6.503
3.831
1.456
F
O
N
H
O
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
0.98
1.00
1.89
3.00
9.00

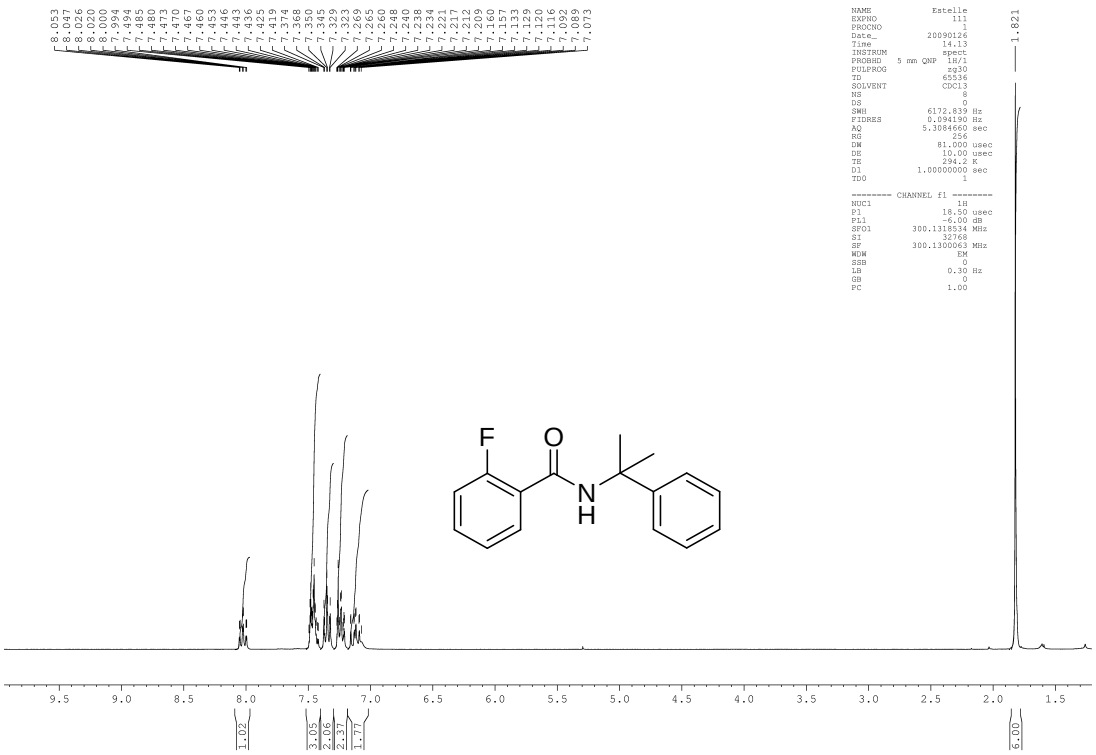

163.25
163.09
162.31
162.27
162.22
159.76
132.95
132.89
114.73
114.57
110.49
110.46
101.65
101.26
77.58
77.16
76.73
55.76
51.56
28.96
F
O
N
H
O
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm


```
NAME                 Estelle
EXPNO                    146
PROCNO                     1
Date_             20090626
Time              18.15
INSTRUM           spect
PROBHD    5 mm QNP  1H/1
PULPROG           zgflqn
TD                  131072
SOLVENT           CDCl3
NS                      16
DS                       4
SWH               67567.570 Hz
FIDRES            0.515500 Hz
AQ                0.9699828 sec
RG                   645.1
DW                 7.400 usec
DE                10.00 usec
TE                 296.2 K
D1                1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                   19F
P1                 10.90 usec
PL1                -6.00 dB
SFO1          282.3761148 MHz
SI                   65536
SF            282.4043550 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00
```



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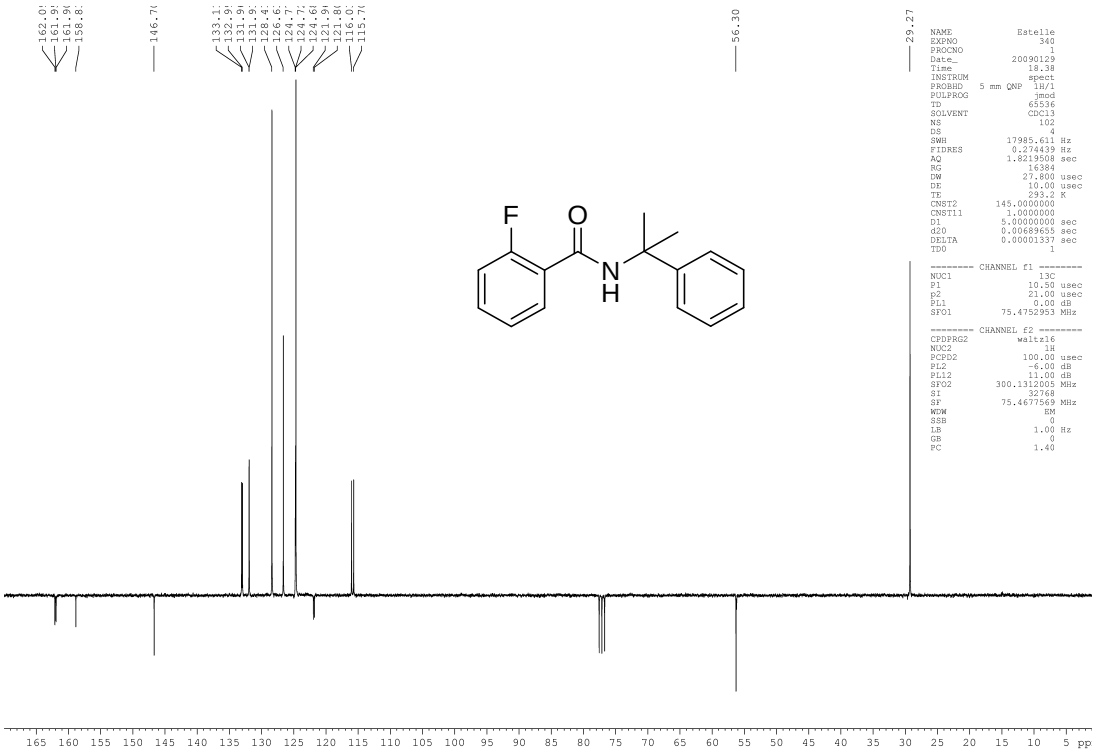
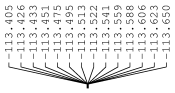
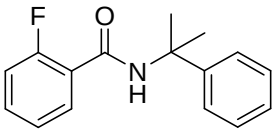
8.047
8.026
8.020
8.000
7.994
7.494
7.485
7.480
7.473
7.470
7.467
7.460
7.453
7.446
7.443
7.436
7.425
7.419
7.374
7.368
7.350
7.345
7.329
7.323
7.265
7.260
7.248
7.240
7.238
7.234
7.221
7.217
7.212
7.209
7.160
7.157
7.133
7.129
7.120
7.116
7.092
7.089
7.073
1.821
F
O
N
H
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.02
3.05
2.06
2.37
1.77
6.00


162.0
161.9
161.9
158.8
146.7
133.1
132.9
131.9
131.9
128.4
126.6
124.7
124.7
124.6
121.8
116.0
115.7
56.30
29.27
F
O
N
H
165
160
155
150
145
140
135
130
125
120
115
110
105
100
95
90
85
80
75
70
65
60
55
50
45
40
35
30
25
20
15
10
5 ppm



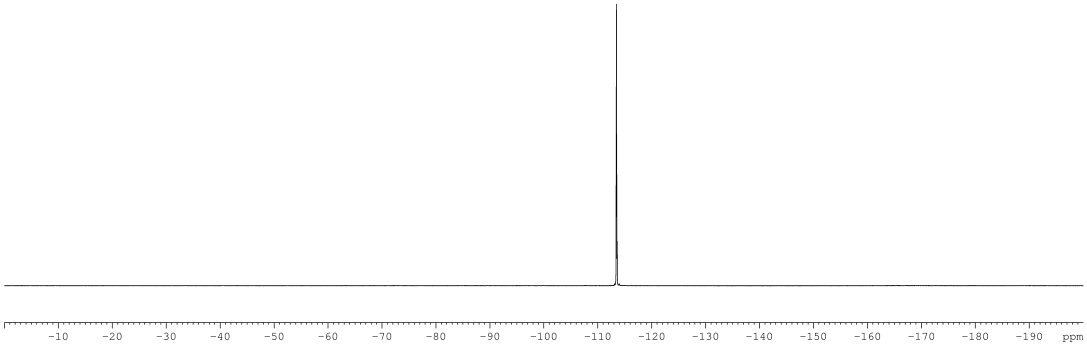


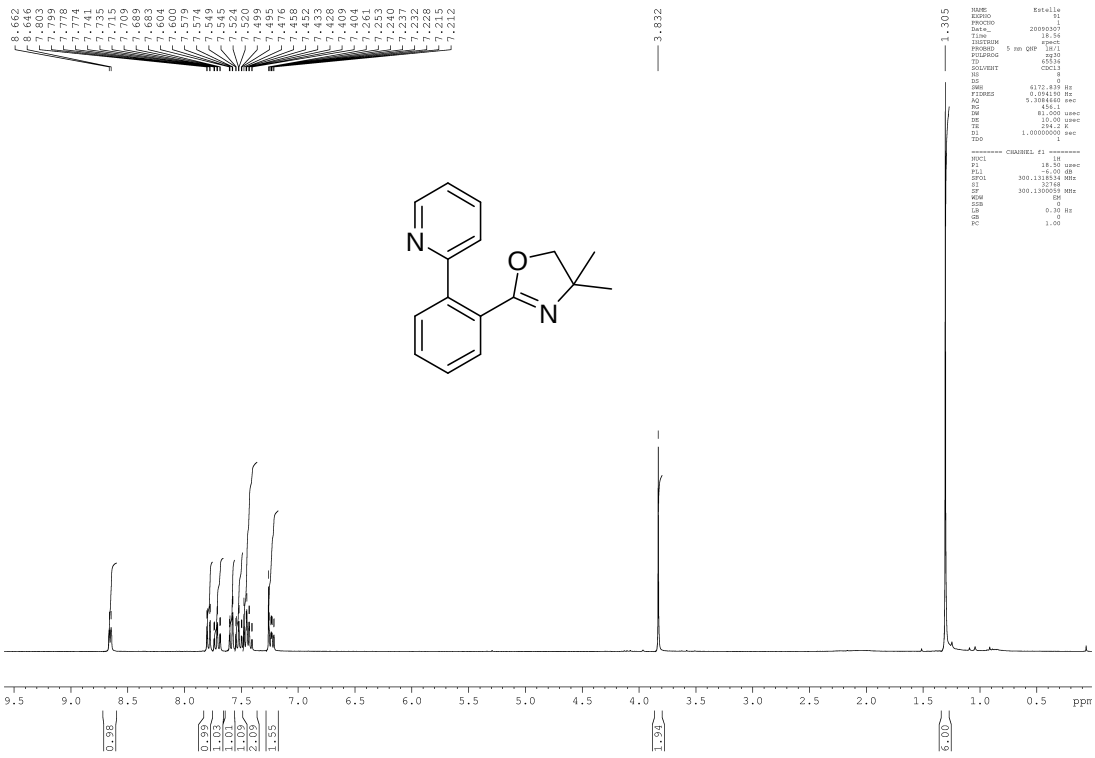

```
Current Data Parameters
NAME                  Feb13-2009
EXPNO                        210
PROCNO                         1

F2 - Acquisition Parameters
Date_                   20090213
Time                       13.44
INSTRUM                    spect
PROBHD    5 mm QNP  1H/1
PULPROG                  zgflqn
TD                       131072
SOLVENT                    CDCl3
NS                            16
DS                             4
SWH                    67567.570 Hz
FIDRES                 0.515500 Hz
AQ                     0.9699828 sec
RG                         456.1
DW                        7.400 usec
DE                       10.00 usec
TE                        294.2 K
D1                  1.00000000 sec
TD0                            1

======== CHANNEL f1 ========
NUC1                         19F
P1                         10.90 usec
PL1                        -6.00 dB
SFO1                282.3761148 MHz

F2 - Processing parameters
SI                         65536
SF                  282.4043550 MHz
WDW                           EM
SSB                            0
LB                          0.30 Hz
GB                             0
PC                          1.00
```

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8.646
7.803
7.799
7.778
7.774
7.741
7.735
7.715
7.709
7.689
7.683
7.604
7.600
7.579
7.574
7.549
7.545
7.524
7.469
7.449
7.495
7.476
7.458
7.452
7.433
7.428
7.409
7.404
7.261
7.253
7.240
7.237
7.232
7.228
7.215
7.212
3.832
1.305
N
O
N
9.5
9.0
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
ppm
0.98
0.99
1.03
1.03
1.09
2.09
1.55
1.94
6.00


163.10
158.18
148.55
140.14
135.55
130.07
129.75
129.35
128.78
127.65
122.70
121.61
78.97
67.14
27.60
N
O
N
165
160
155
150
145
140
135
130
125
120
115
110
105
100
95
90
85
80
75
70
65
60
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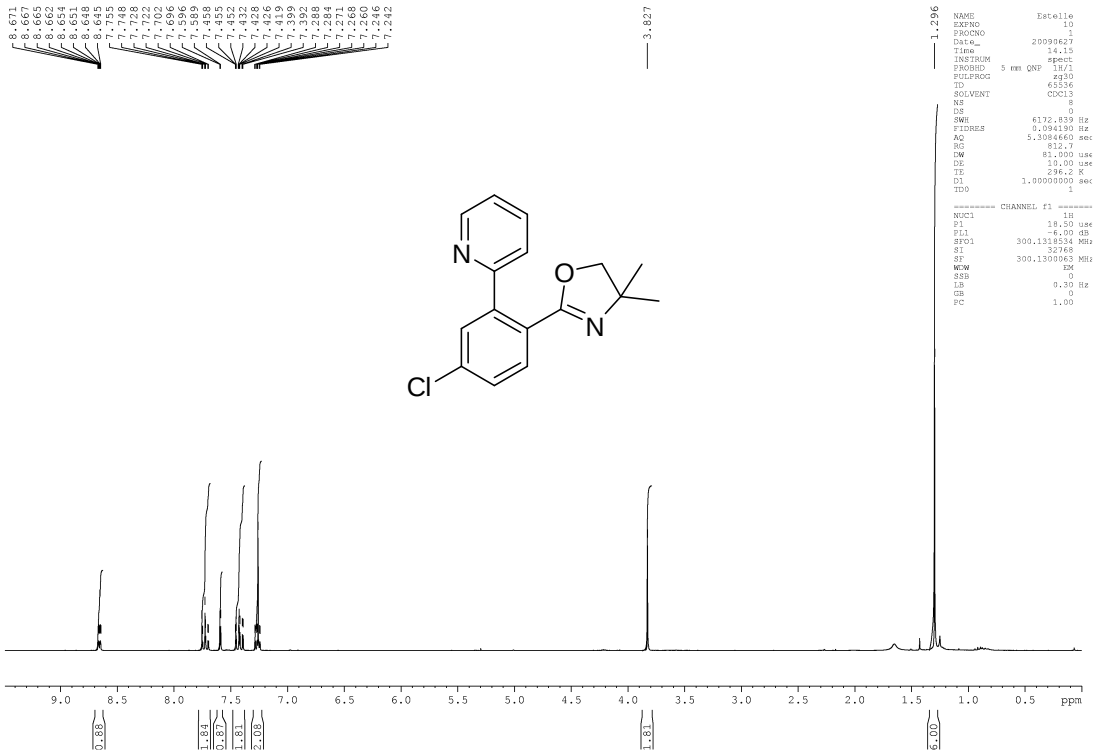
50
45
40
35
30
25
20
15
10
5


8.671
8.667
8.665
8.662
8.654
8.651
8.648
8.645
7.755
7.748
7.728
7.722
7.702
7.696
7.596
7.589
7.458
7.455
7.452
7.432
7.428
7.426
7.419
7.399
7.392
7.388
7.284
7.271
7.268
7.260
7.246
7.242
3.827
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N
O
N
Cl
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7.5
7.0
6.5
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3.5
3.0
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1.0
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0.87
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1.81
6.00


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136.30
135.93
131.53
129.86
128.12
126.41
123.04
122.34
79.36
77.59
77.16
76.74
67.60
27.88
N
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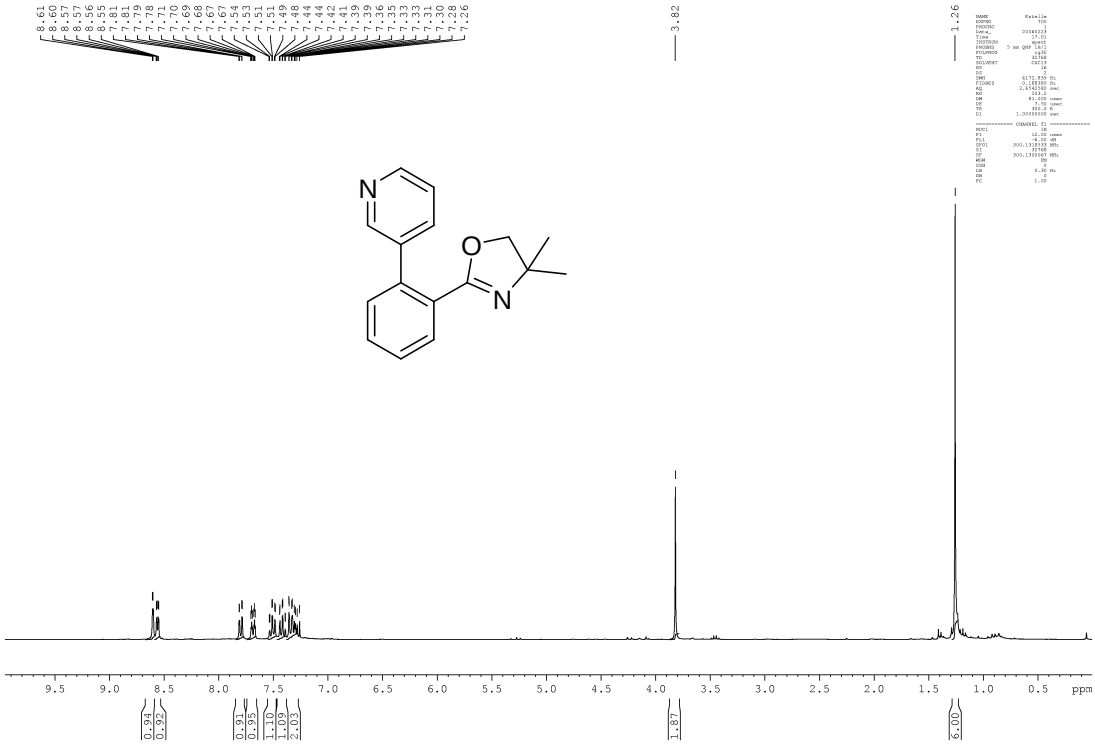
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7.68
7.67
7.67
7.54
7.53
7.51
7.51
7.49
7.48
7.44
7.44
7.42
7.41
7.39
7.39
7.36
7.35
7.33
7.33
7.31
7.30
7.28
7.26
3.82
1.26
N
O
N
9.5
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8.0
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7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
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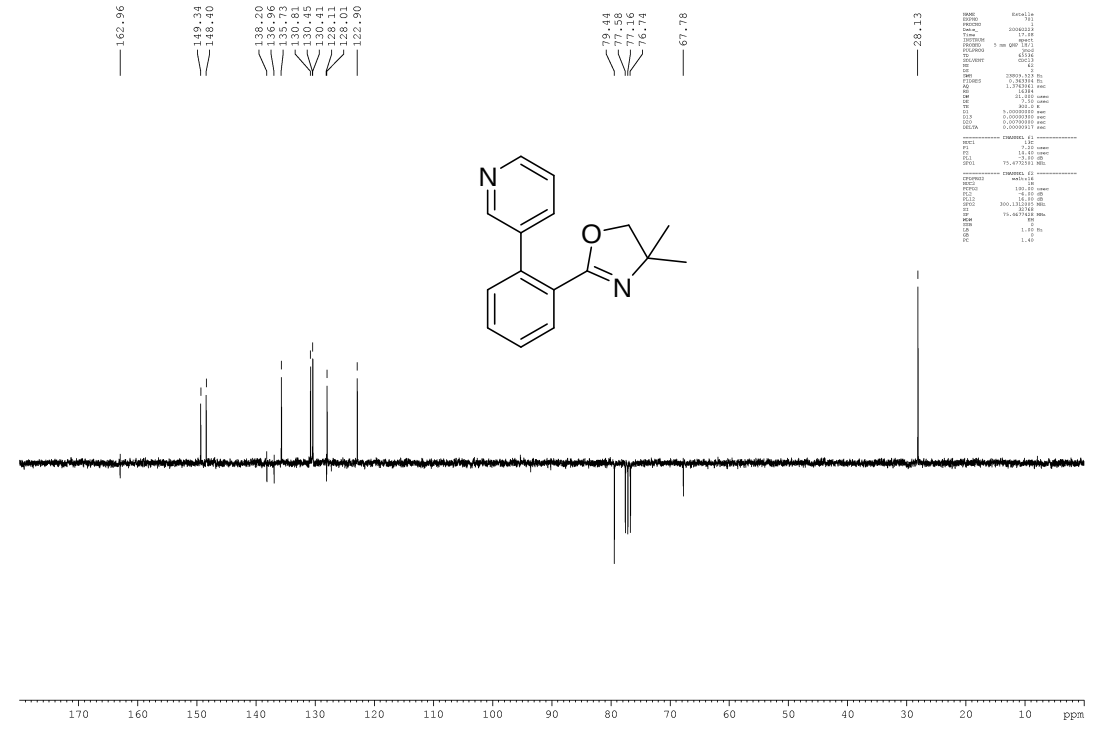
6.00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130.45
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128.11
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122.90
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77.58
77.16
76.74
67.78
28.13
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150
140
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70
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20
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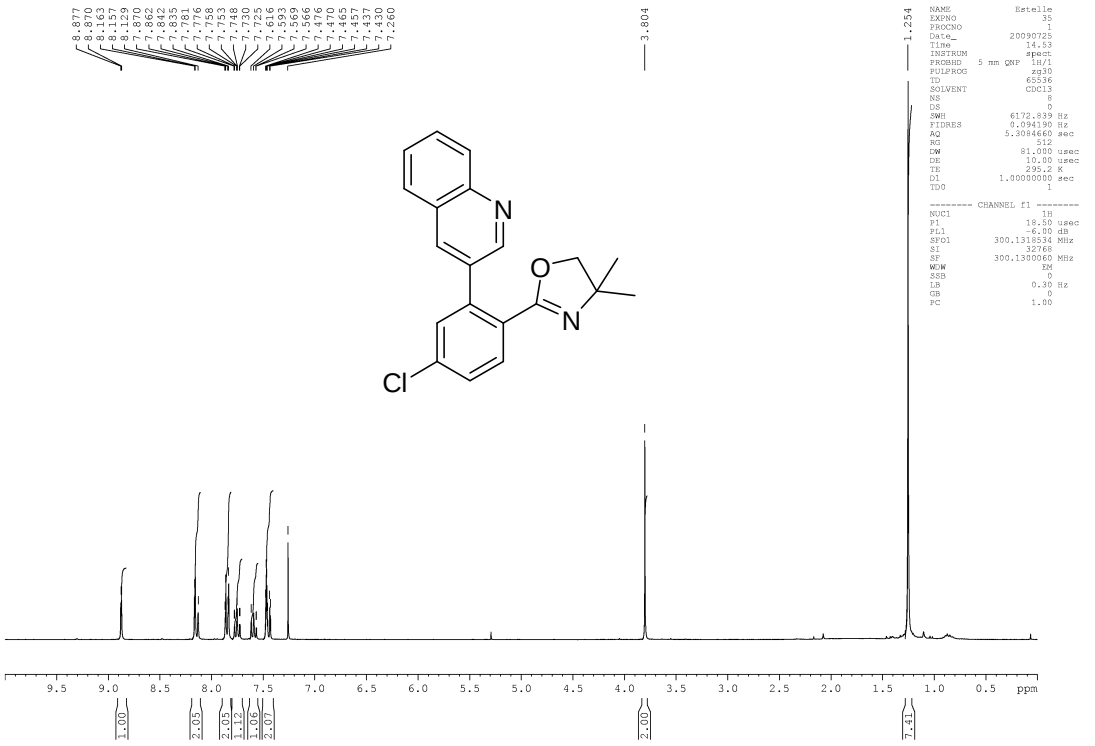
ppm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7.753
7.748
7.720
7.616
7.593
7.569
7.566
7.476
7.470
7.465
7.457
7.437
7.430
7.260
3.804
1.254
N
O
N
Cl
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
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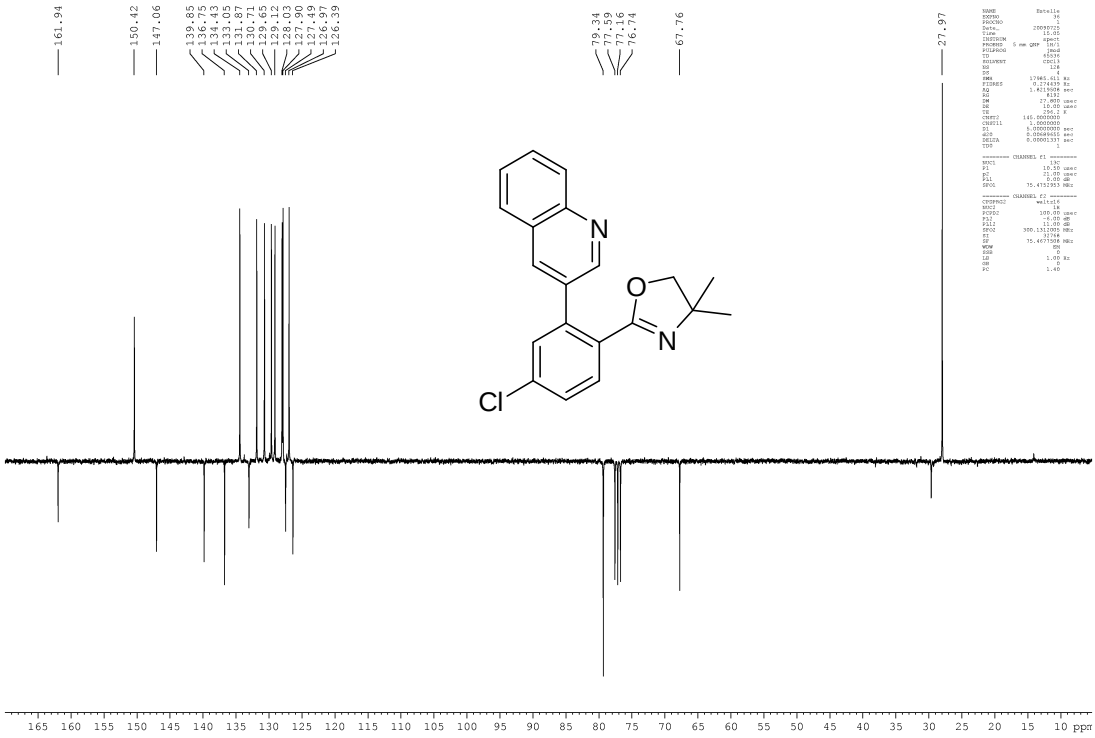
2.05
1.12
1.06
2.07
2.00
7.41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147.06
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134.43
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131.87
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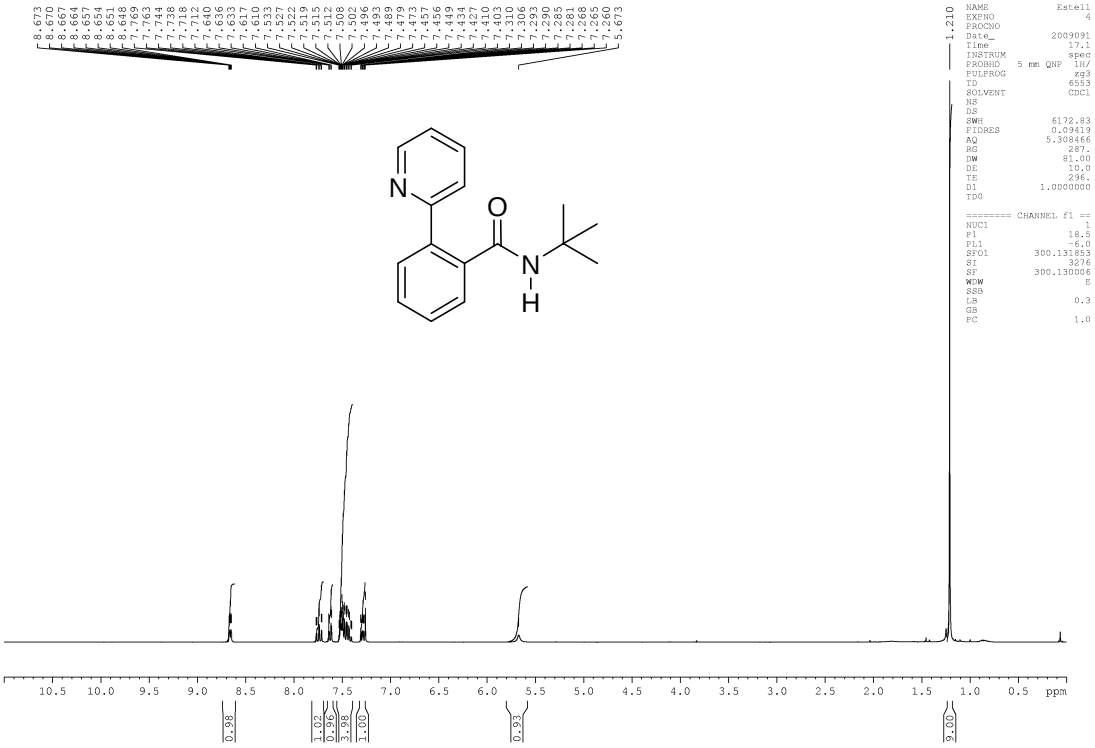
129.65
128.12
128.03
127.90
127.49
126.97
126.39
79.34
77.59
77.16
76.74
67.76
27.97
N
O
N
Cl
165 160 155 150 145 140 135 130 125 120 115 110 105 100 95 90 85 80 75 70 65 60 55 50 45 40 35 30 25 20 15 10 ppm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8.648
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7.763
7.744
7.738
7.718
7.712
7.640
7.636
7.633
7.617
7.610
7.533
7.527
7.522
7.519
7.515
7.508
7.502
7.496
7.493
7.489
7.479
7.473
7.457
7.456
7.449
7.434
7.427
7.410
7.403
7.310
7.306
7.293
7.290
7.285
7.281
7.268
7.265
5.673
1.210
N
O
N
H
NAME Estell
EXPNO 4
PROCNO 1
Date_ 20090921
Time 17.1
INSTRUM spec
PROBHD 5 mm QNP 1H/
PULPROG zg3
TD 6553
SOLVENT CDCl
NS
DS
SWH 6172.83
FIDRES 0.09419
AQ 5.308466
RG 287.
DW 81.00
DE 10.0
TE 296.
D1 1.0000000
TD0
======== CHANNEL f1 ==
NUC1 1
P1 18.5
PL1 -6.0
SFO1 300.131853
SI 3276
SF 300.130006
WDW E
SSB
LB 0.3
GB
PC 1.0
10.5 10.0 9.5 9.0 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 ppm
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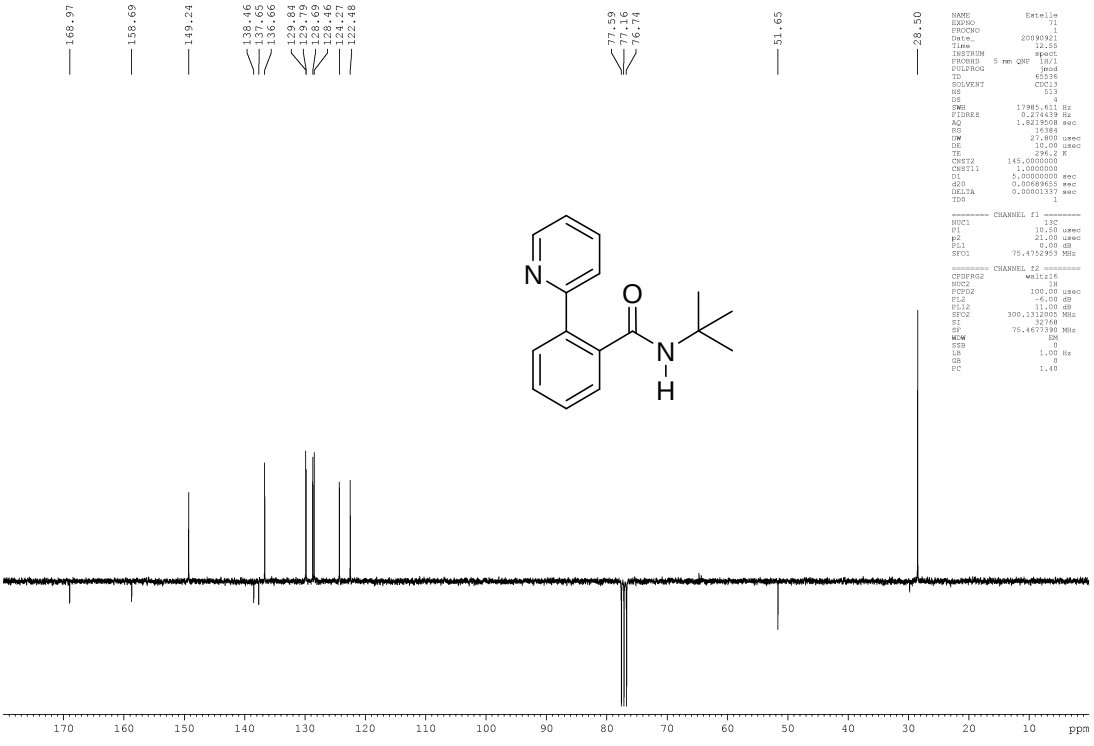
0.96
3.98
1.00
0.93
9.00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136.66
129.84
129.79
128.69
128.46
128.37
122.48
77.59
77.16
76.74
51.65
28.50
N
O
N
H
NAME Estelle
EXPNO 71
PROCNO 1
Date_ 20090921
Time 12.58
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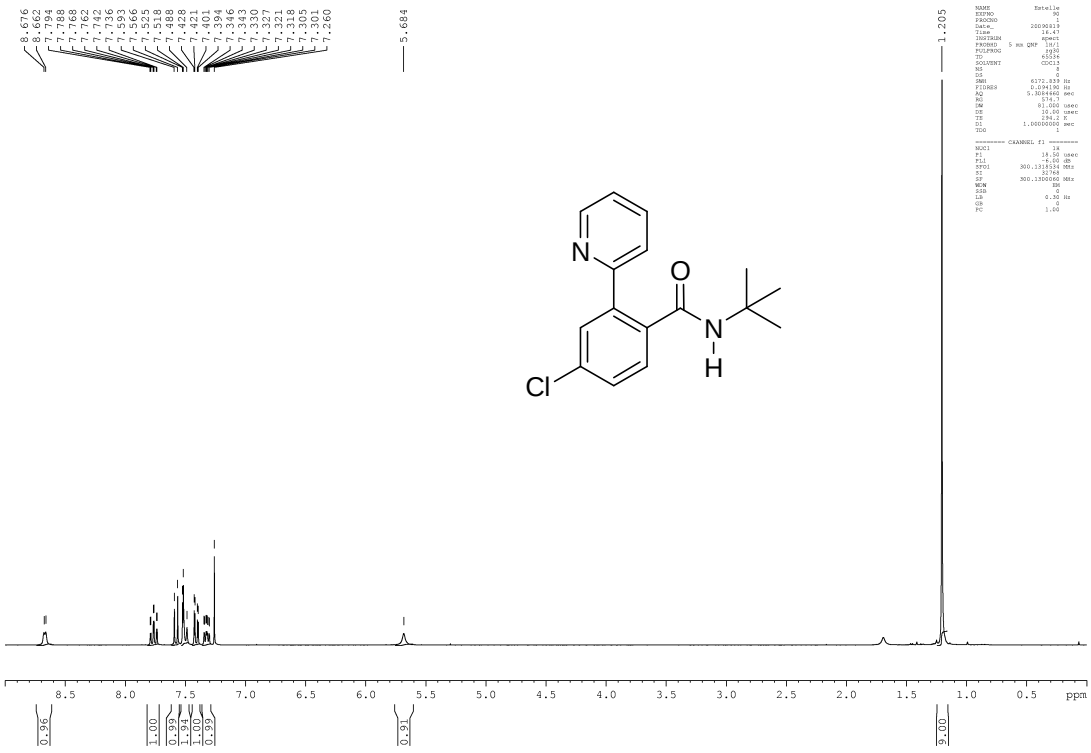
PULPROG mod
TD 65536
SOLVENT CDCl3
NS 513
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 16384
DW 27.800 usec
DE 10.00 usec
TE 296.2 K
CNST2 145.0000000
CNST11 1.0000000
D1 5.00000000 sec
d20 0.00689655 sec
DELTA 0.00001337 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.50 usec
p2 21.00 usec
PL1 0.00 dB
SFO1 75.475295 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -6.00 dB
PL12 11.00 dB
SFO2 300.1312005 MHz
SI 32768
SF 75.4677390 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm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7.566
7.525
7.518
7.488
7.428
7.421
7.401
7.394
7.366
7.346
7.330
7.327
7.321
7.318
7.305
7.301
7.260
5.684
1.205
N
O
N
H
Cl
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
ppm
0.96
1.00
0.99
1.94
1.00
0.99
0.91
9.00


167.82
157.16
149.29
139.94
136.74
135.88
135.52
129.96
129.77
128.62
124.11
122.88
77.58
77.16
76.74
51.70
28.37
N
O
N
H
Cl
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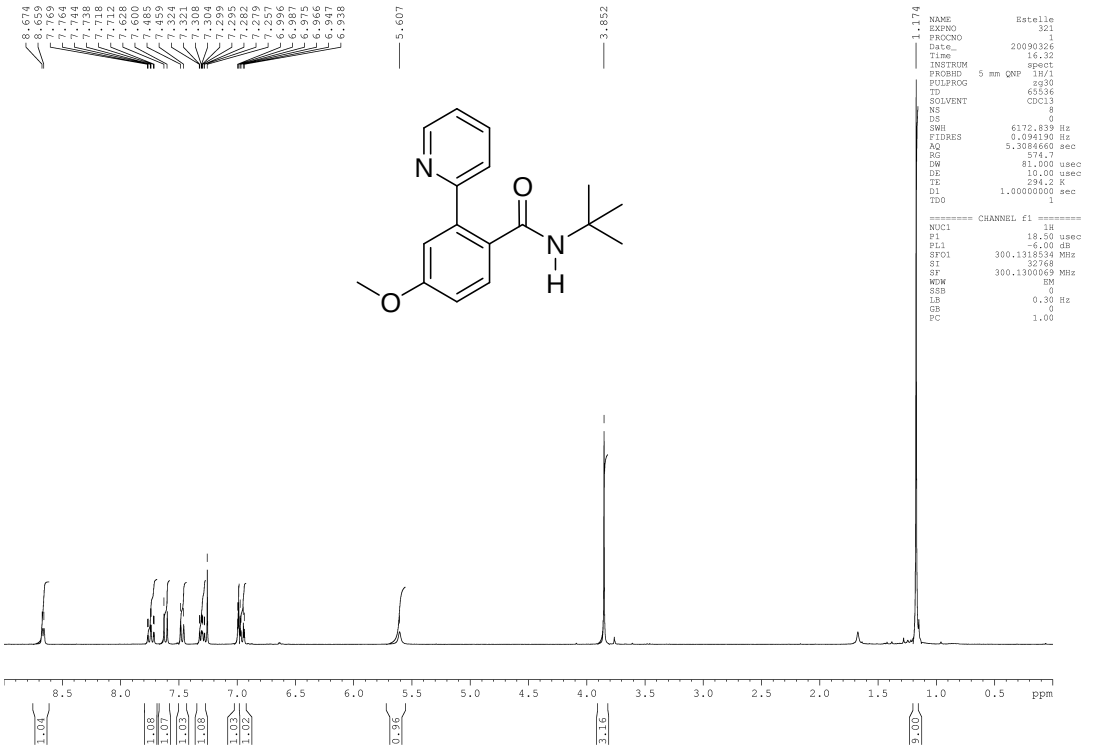
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm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7.712
7.628
7.600
7.485
7.459
7.324
7.321
7.308
7.304
7.299
7.295
7.282
7.279
7.257
6.996
6.987
6.975
6.966
6.957
6.938
5.607
3.852
1.174
N
O
N
H
O
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
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1.08
1.07
1.03
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1.03
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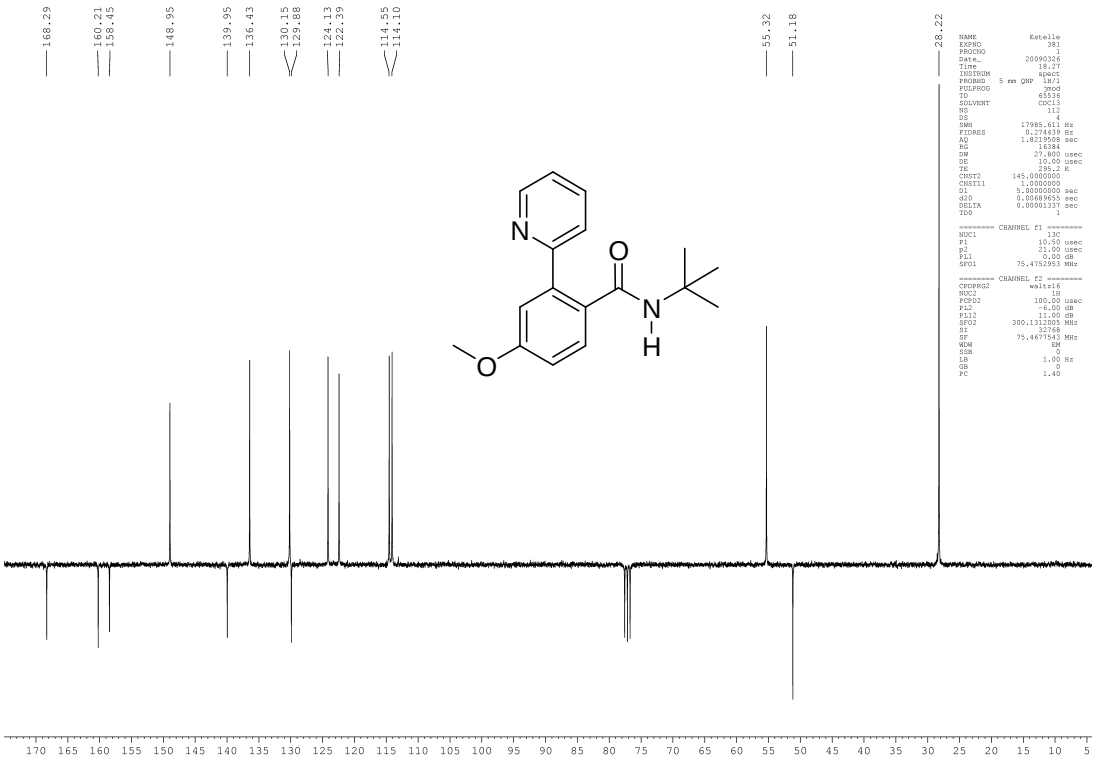
9.00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130.15
129.88
124.13
122.39
114.55
114.10
55.32
51.18
28.22
N
O
N
H
O
170
165
160
155
150
145
140
135
130
125
120
115
110
105
100
95
90
85
80
75
70
65
60
55
50
45
40
35
30
25
20
15
10
5


8.668
8.662
8.652
8.645
7.785
7.779
7.772
7.759
7.753
7.731
7.706
7.688
7.580
7.437
7.430
7.410
7.403
7.392
7.390
7.376
7.374
7.366
7.364
7.352
7.346
7.260
5.077
1.183
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N
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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
ppm
2.01
0.99
1.00
1.01
2.03
0.92
9.00


167.32
149.35
149.13
137.47
136.23
135.95
135.86
134.43
134.01
130.00
128.43
123.31
77.58
77.16
76.73
51.96
28.40
N
O
N
H
Cl
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150
140
130
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80
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60
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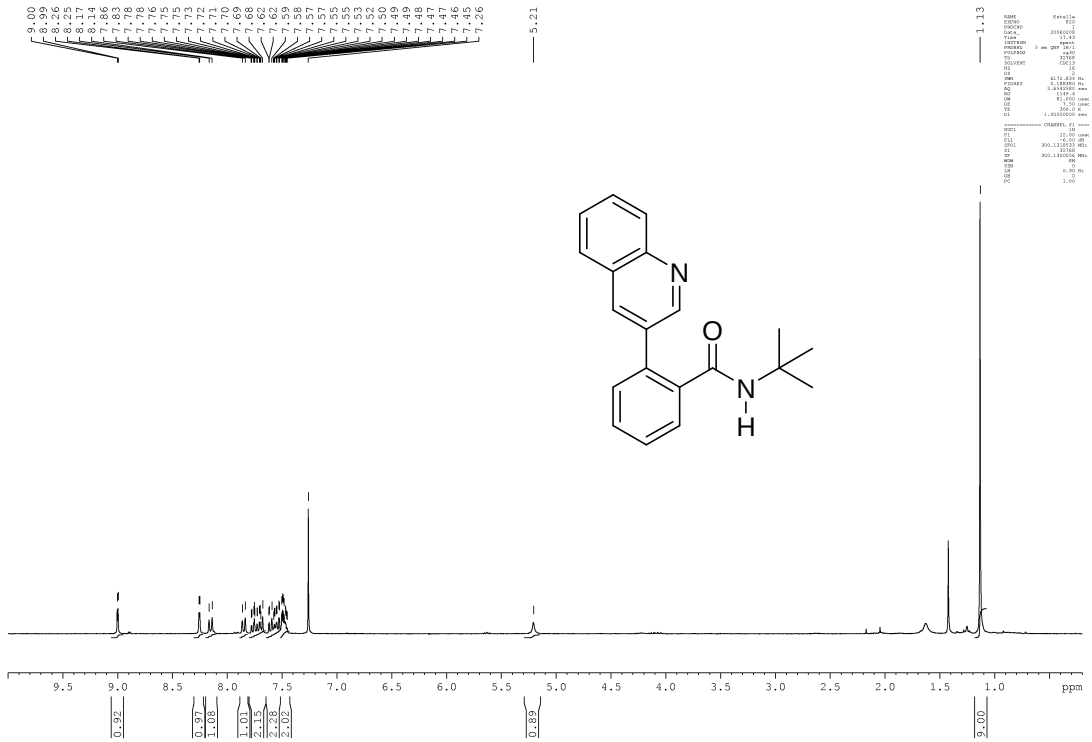
40
30
20
10
ppm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7.83
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7.78
7.76
7.75
7.75
7.73
7.72
7.71
7.70
7.69
7.68
7.62
7.62
7.59
7.58
7.57
7.57
7.55
7.55
7.53
7.52
7.50
7.49
7.49
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7.47
7.47
7.46
7.45
7.45
7.26
5.21
1.13
N
O
N
H
9.5
9.0
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
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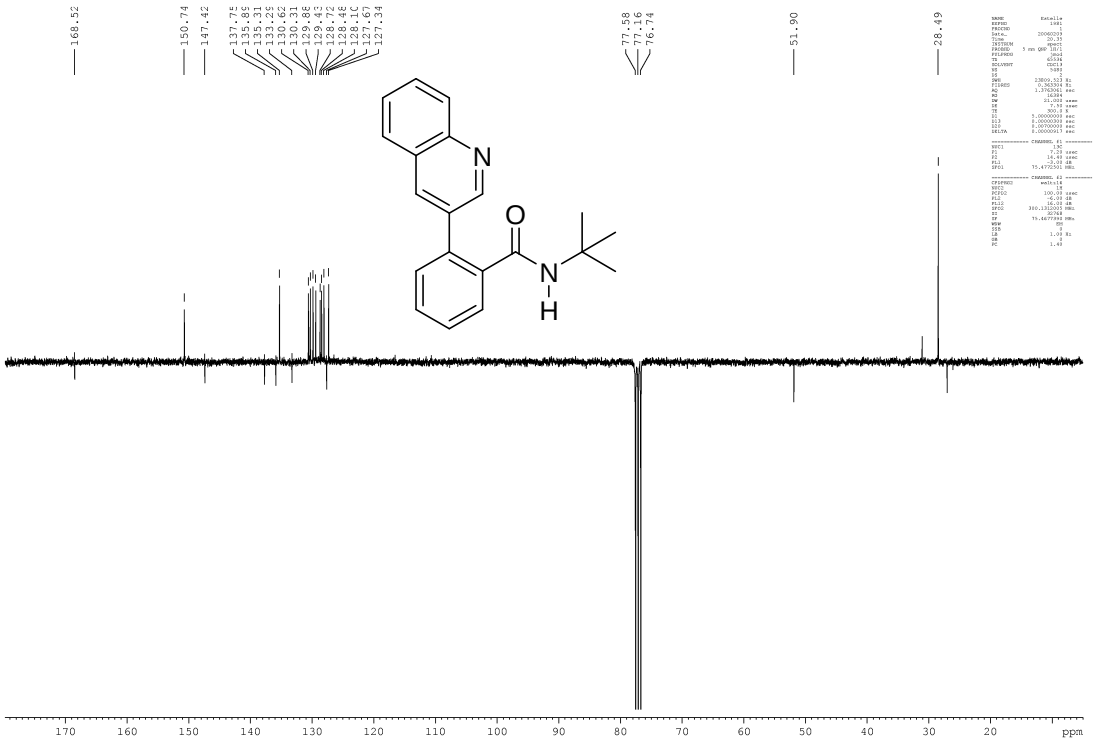
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130.62
130.31
129.86
129.42
128.46
128.16
127.67
127.34
77.58
77.16
76.74
51.90
28.49
N
O
N
H
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160
150
140
130
120
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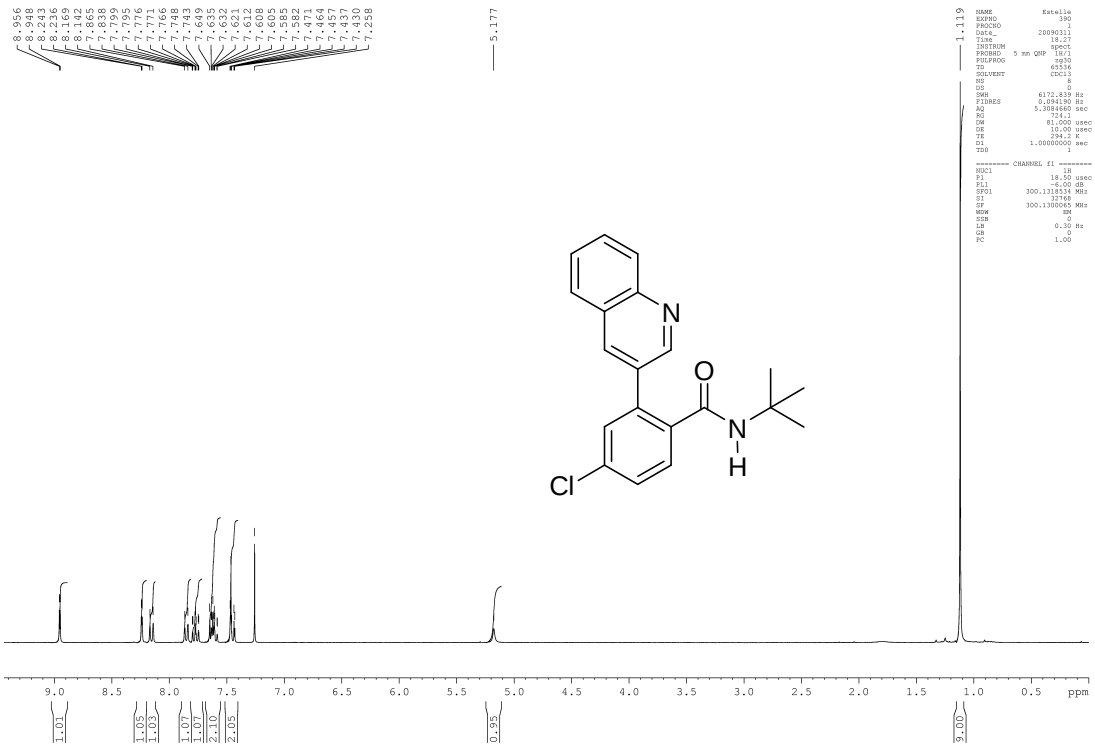
80
70
60
50
40
30
20
ppm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7.748
7.743
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7.635
7.632
7.621
7.612
7.608
7.605
7.585
7.582
7.471
7.464
7.457
7.437
7.430
7.258
5.177
1.119
N
O
N
H
Cl
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8.5
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
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ppm
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1.07
1.07
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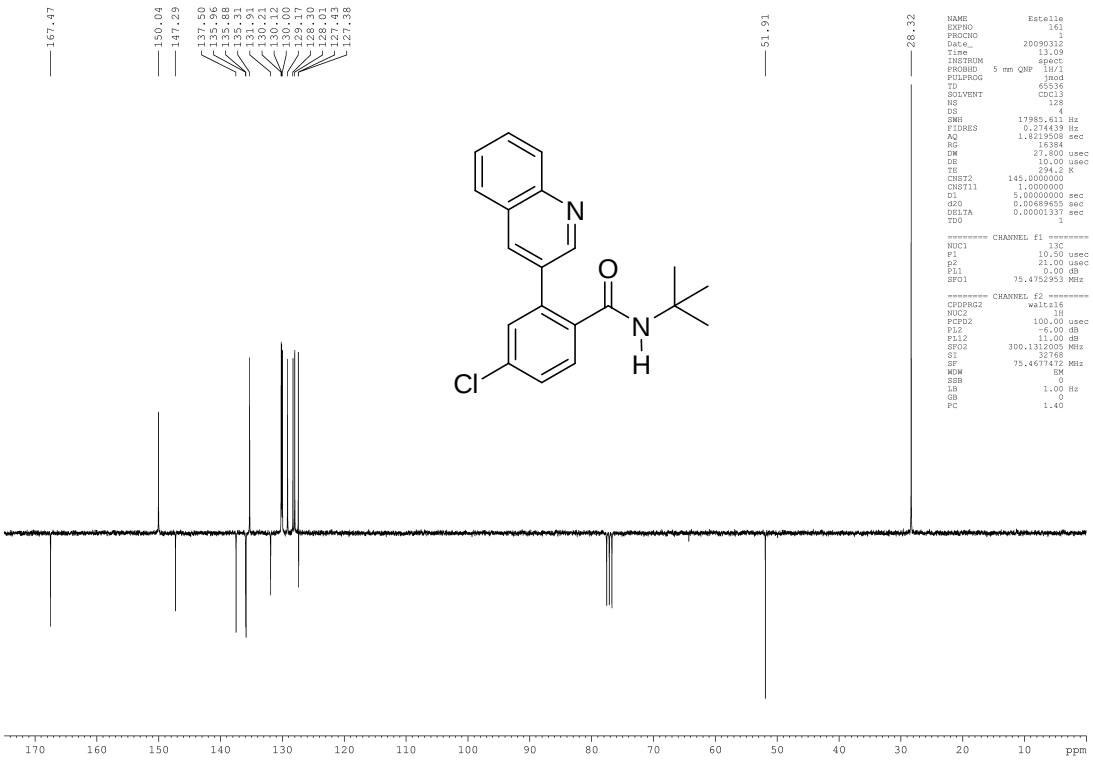
9.00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131.91
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130.12
130.00
129.17
128.30
128.01
127.43
127.38
51.91
28.32
N
O
N
H
Cl
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150
140
130
120
110
100
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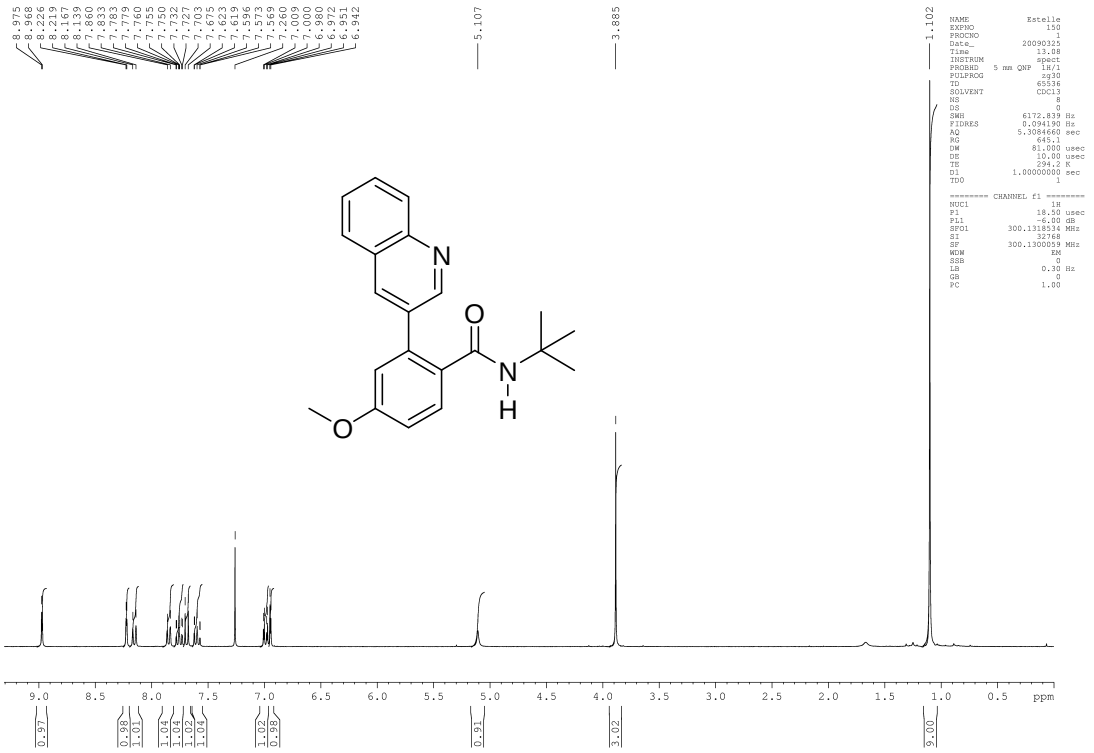
80
70
60
50
40
30
20
10
ppm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7.750
7.732
7.727
7.703
7.675
7.623
7.619
7.596
7.573
7.569
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7.009
7.000
6.980
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6.951
6.942
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3.885
1.102
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O
N
H
O
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8.0
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7.0
6.5
6.0
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5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
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0.98
1.01
1.04
1.04
1.02
1.04
1.04
1.02
0.98
0.91
3.02
9.00


168.03
160.65
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147.26
137.62
135.03
133.31
130.54
130.55
129.76
129.22
127.93
127.49
127.20
115.94
113.34
55.52
51.53
28.35
N
O
N
H
O
170
165
160
155
150
145
140
135
130
125
120
115
110
105
100
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70
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35
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5
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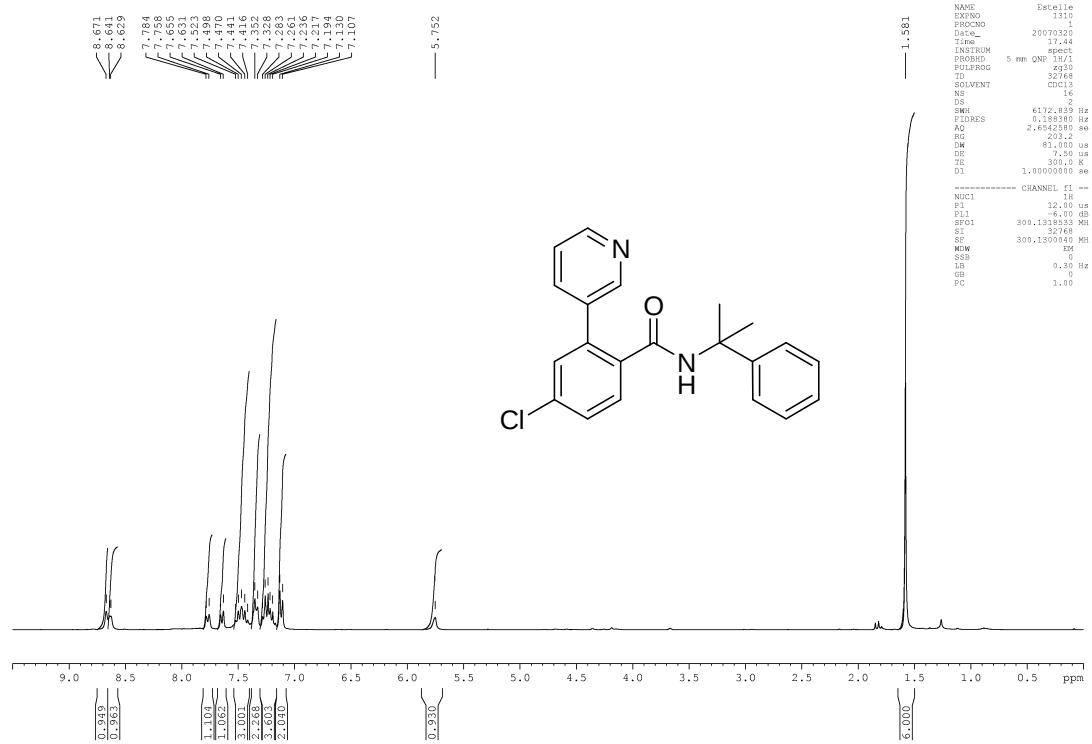
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7.217
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7.107
5.752
1.581
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O
N
H
Cl
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8.0
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7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
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2.5
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1.5
1.0
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ppm
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6.000


168.06
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148.90
146.43
137.26
136.54
136.14
135.82
130.30
130.17
128.56
128.49
128.42
126.88
124.65
123.32
77.59
77.16
76.74
56.56
28.24
N
O
N
H
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140
130
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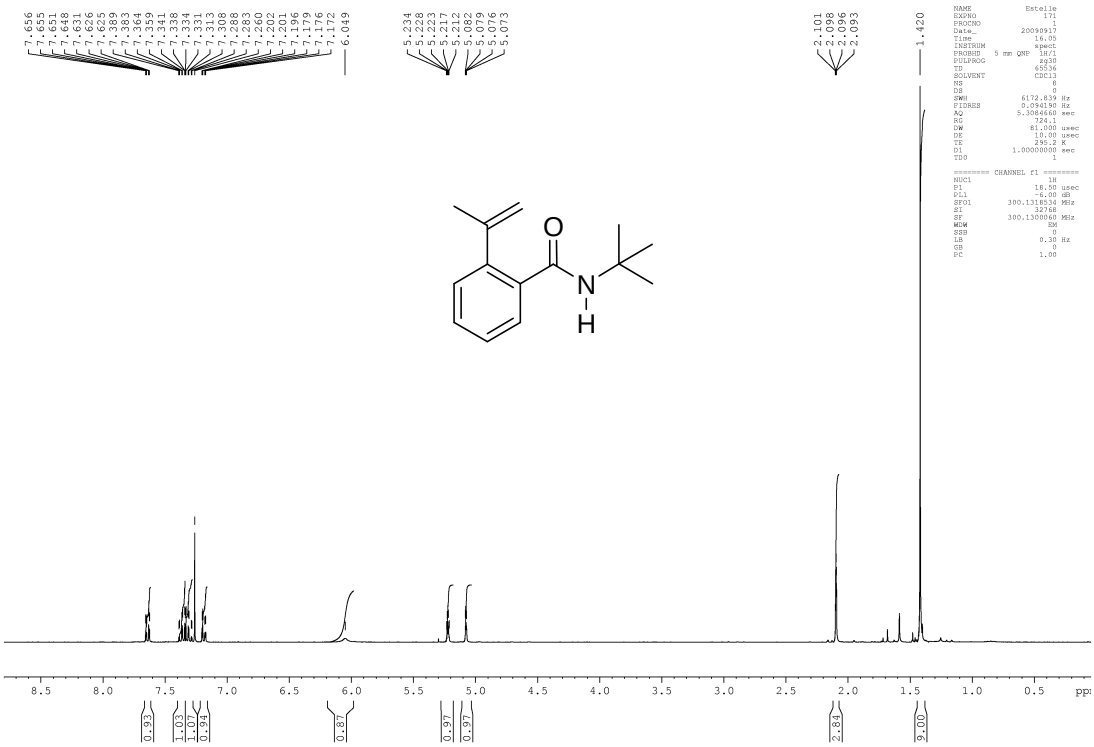
20
10
ppm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7.364
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7.341
7.338
7.334
7.331
7.313
7.308
7.288
7.283
7.260
7.202
7.201
7.196
7.179
7.176
7.172
6.049
5.234
5.228
5.223
5.217
5.212
5.082
5.079
5.076
5.073
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2.098
2.096
2.093
1.420
O
N
H
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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
ppm
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1.03
1.07
0.94
0.87
0.97
0.97
2.84
9.00


168.30
146.80
141.59
135.22
130.03
128.85
128.53
127.54
115.73
77.58
77.16
76.74
51.66
28.55
24.59
O
N
H
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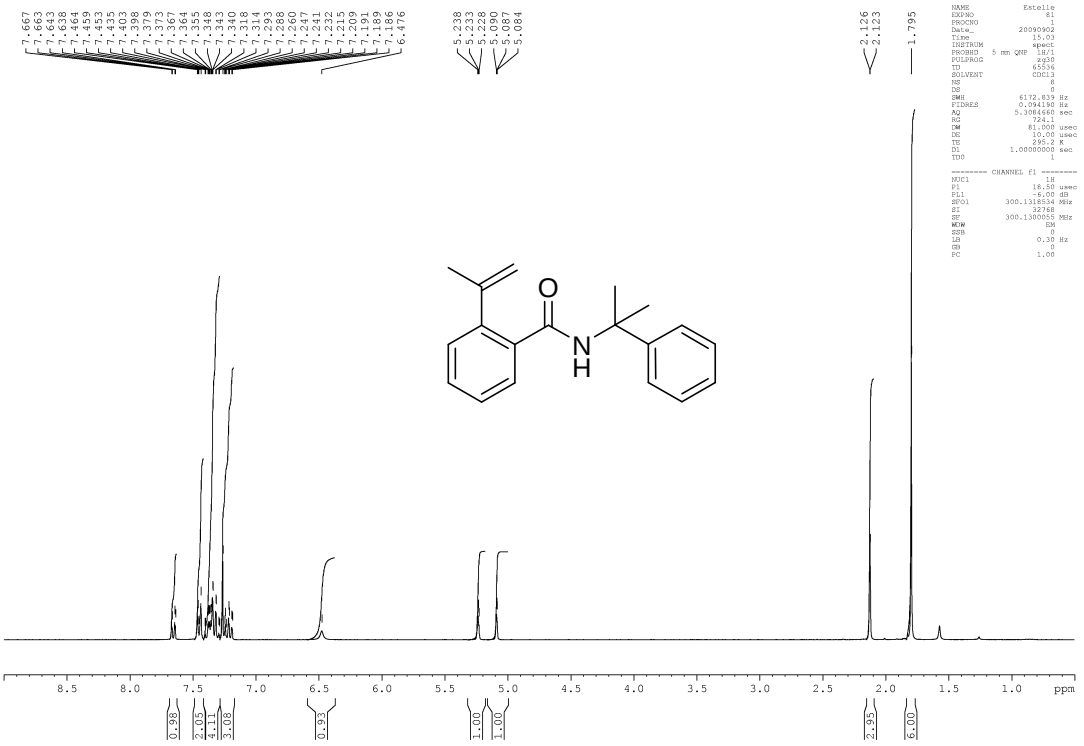
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O
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H
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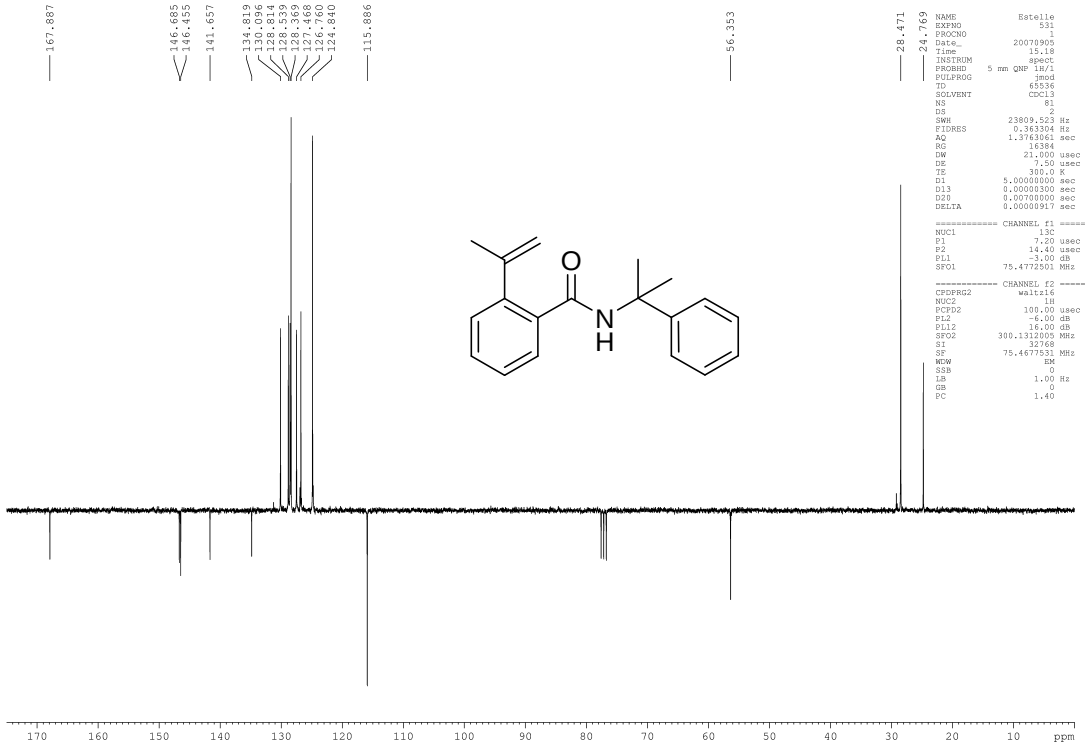
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O
N
H
170
160
150
140
130
120
110
100
90
80
70
60
50
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30
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10
ppm

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