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September 2014: The paragraphs above replace the section "Handling and Disposal of Hazardous Chemicals" in the originally published version of this article. The statements above do not supersede any specific hazard caution notes and safety instructions included in the procedure.

Organic Syntheses

N-Carboxylated-2-substituted Indoles and 2,3-Disubstituted-2,3-dihydro-4-quinolones from 2-Alkynylbenzamides

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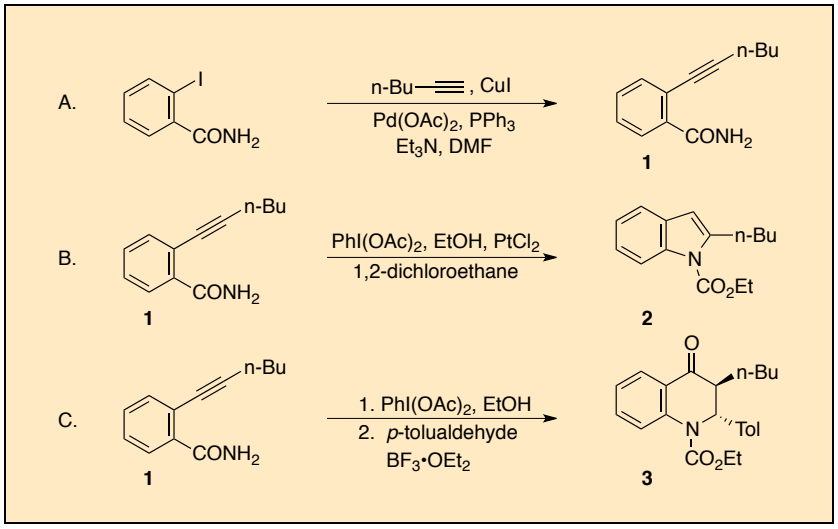
Checked by Hang Chu and Viresh H. Rawal



Procedure

A. *2-(1-Hexynyl)benzamide (**1**).*[2] A 500-mL round-bottomed flask equipped with a Teflon coated magnetic stirring bar (3.8 × 0.9 cm) is charged successively with 2-iodobenzamide (19.8 g, 80 mmol), PPh_3 (419 mg, 1.60 mmol, 0.02 equiv), CuI (152 mg, 0.80 mmol, 0.01 equiv),

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$Pd(OAc)_2$ (180 mg, 0.80 mmol, 0.01 equiv), *N*,*N*-dimethylformamide (DMF, 40 mL, anhydrous), Et_3N (120 mL), and 1-hexyne (12.0 mL, 104 mmol, 1.3 equiv) (Note 1). A glass inlet adapter (24/40) is fitted on the flask, which is then purged by two cycles of evacuation (2-5 sec.) and back-filling with nitrogen. A slight positive pressure of nitrogen is maintained. The flask is then placed in a preheated oil bath (oil bath temperature 60 °C) and allowed to stir for 16 h, during which time the color of the slurry darkens progressively to brown. Completion of the reaction is confirmed by TLC monitoring (hexanes:EtOAc, 2:1, R_f = 0.35), the reaction mixture is diluted with 200 mL of EtOAc and transferred to a 1 L separatory funnel. The organic phase is sequentially washed with saturated NH_4Cl solution (2 x 120 mL) and saturated brine (1 x 100 mL), and the aqueous layer is back-extracted with EtOAc (1 x 100 mL). The combined organic phase is dried over anhydrous Na_2SO_4 (15 g, 10 min), filtered, and concentrated by rotary evaporation (15–20 mmHg, 23 °C) to give a dark orange solid. The solid is purified by column chromatography on silica gel (Note 2) to afford 2-(1-hexynyl)benzamide (**1**) as a yellowish powder (14.4 g, 90%) (Note 3).

B. *Ethyl 2-butyl-1H-indole-1-carboxylate (**2**)*. A 500-mL, round-bottomed flask equipped with a Teflon coated magnetic stirring bar (3.8 × 0.9 cm) is successively charged with 2-(1-hexynyl)benzamide **1** (9.02 g, 45 mmol), $PhI(OAc)_2$ (15.2 g, 47 mmol, 1.04 equiv), 1,2-dichloroethane (150 mL), and ethanol (7.85 mL, 134 mmol). The flask is fitted with a Dimroth condenser topped with a glass inlet adapter (24/40) and purged by two cycles of evacuation (2-5 sec.) and back-filling with nitrogen. The solution is maintained under a slight positive pressure of nitrogen. The reaction flask is placed in an oil bath (90 °C, bath temp.) and allowed to stir for 2 h. During this period, the reaction mixture turns dark red. The oil bath is removed, and the reaction mixture is allowed to cool to room temperature. The condenser is removed and $PtCl_2$ (596 mg, 2.2 mmol, 0.05 equiv) (Note 4) is quickly added in one portion. After replacing the condenser, the mixture is put back in the 90 °C oil bath and stirred for further 3 h (Note 5), during which time the color of the reaction mixture turns dark brown. After completion of the reaction is confirmed by TLC monitoring (hexane:EtOAc, 25:1, R_f = 0.30), the reaction mixture is filtered through a pad of Florisil (20 g). The Florisil filter pad is washed with an additional 40 mL of 1,2-dichloroethane. The filtrate is concentrated by rotary evaporation (15–20 mmHg, 23 °C), and the residual brown oil is purified by column chromatography on silica gel (Note 6) to afford ethyl 2-butyl-1*H*-indole-1-carboxylate **2** (9.59 g, 87 %) as colorless prisms (Note 7).

C. *Ethyl 3-butyl-4-oxo-2-p-tolyl-3,4-dihydroquinoline-1(2H)-carboxylate (**3**).* A 500-mL round-bottomed flask equipped with a Teflon coated magnetic stirring bar (3.8 × 0.9 cm) is charged with 2-(1-hexynyl)benzamide **1** (8.04 g, 40 mmol), $PhI(OAc)_2$ (14.2 g, 44 mmol, 1.1 equiv), 1,2-dichloroethane (100 mL), and ethanol (4.70 mL, 80 mmol). The flask containing the reagents is fitted with a Dimroth condenser topped with a glass inlet adapter (14/20) and purged by two cycles of evacuation (2-5 sec.) and back-filling with nitrogen. The solution is then maintained under a slight positive pressure of nitrogen. The stirred mixture is placed in a preheated oil bath (90 °C, bath temp.) for 2 h, during which period the reaction mixture turns dark red. The oil bath is removed, and the reaction mixture is allowed to cool to room temperature. The condenser is replaced with a rubber septum, into which is inserted a needle connected to a nitrogen line, and a positive nitrogen pressure is maintained. Neat *p*-tolualdehyde (7.1 mL, 60 mmol, 1.5 equiv) (Note 8) is added by syringe, dropwise over 3 min, followed by $BF_3•Et_2O$ (8.25 mL, 40 mmol, 1.0 equiv) (Note 9), which is also added by syringe, dropwise over 5 min. The reaction mixture turns black upon the addition of $BF_3•Et_2O$,[3] and a slight exotherm is observed. The rubber septum is replaced with the Dimroth condenser and the system is maintained under a slight positive pressure of nitrogen. The black reaction mixture is placed in a preheated oil bath (90 °C, bath temp.) and allowed to stir for 24 h. After confirming the completion of the reaction by TLC monitoring (hexanes:EtOAc, 20:1, R_f = 0.30 distinct blue spot under a UV lamp (254 nm)), the reaction mixture is diluted with 100 mL of chloroform and transferred to a 1 L round-bottomed flask. Saturated aqueous $NaHCO_3$ solution (150 mL) is added slowly via a glass funnel and the reaction mixture is stirred at room temperature until no gas evolution is visible. The mixture is then transferred to a 1 L separatory funnel and washed with saturated $NaHCO_3$ solution (2 x 100 mL) (Note 10) and brine (1 x 100 mL). The aqueous layer is back-extracted with chloroform (1 x 150 mL). The combined organic layer is dried over Na_2SO_4 (20 g, approx. 15 min). The drying agent is removed by filtration and the filtrate concentrated by rotary evaporation (15–20 mmHg, 23 °C) to give a brown oil, which is purified by column chromatography on silica gel (Note 11) to afford ethyl 3-butyl-4-oxo-2-*p*-tolyl-3,4-dihydroquinoline-1(2*H*)-carboxylate **3** (*trans*:*cis* = 20:1, based on integral ratio of ^{1}H NMR) as a slightly yellow oil (11.39 g, 78%) (Note 12).

Notes

1. 2-Iodobenzamide (98.0%) was purchased from Tokyo Chemical Industry Co., Ltd. and used as received. $Pd(OAc)_2$ (98.0%), 1-hexyne (98.0%), CuI (99.5%), PPh_3 (97.0%), and Et_3N (99.0%) were purchased from Sigma Aldrich, Co. and used as received. DMF (Fischer Optimum Grade) was dried through a molecular sieves based solvent drying system (Innovative Technologies).
2. Column chromatography was carried out using SILICYCLE SiliaFlash P60 silica gel. A glass column (5 x 40 cm) was slurry-packed with 200 g of silica gel in hexane. The compound was loaded on the column as a solution in a small amount of CH_2Cl_2, and the column was eluted first with hexane, then hexane:EtOAc, 50:1 (ca. 1 L), then hexane:EtOAc, 10:1 (ca. 0.5 L), then hexane:EtOAc, 5:1 (ca. 0.5 L), and then hexane:EtOAc, 2:1 until all the product had eluted. Fractions containing the desired product **1** were combined and concentrated using a rotary evaporator at 30 °C (15–20 mmHg) and dried under high vacuum at 23 °C (5–10 mmHg).
3. The procedure was performed at half-scale in 91% by the checkers. Characterization data for compound **1**: TLC R_f = 0.35 (hexane:EtOAc, 2:1); mp 105–106 °C (slightly orange powder from hexane-EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$) δ: 0.96 (t, *J* = 7.2 Hz, 3 H), 1.45–1.50 (m, 2 H), 1.60–1.64 (m, 2 H), 2.50 (t, *J* = 7.0 Hz, 2 H), 6.15 (br s, 1 H), 7.35–7.42 (m, 2H), 7.48 (td, *J* = 2.4, 5.6 Hz, 1H), 7.69 (br s, 1H), 8.11 (dd, *J* = 2.4, 7.2 Hz, 1 H); ^{13}C NMR ($CDCl_3$, 126 MHz) δ: 13.6, 19.3, 22.1, 30.5, 79.7, 97.8, 121.0, 128.1, 130.3, 131.0, 133.8, 134.0, 168.2. IR ($CHCl_3$, cm^{-1}) 3372, 3181, 2956, 2930, 2871, 1648, 1594, 1489, 1452, 1398, 1124, 817, 759, 634. HRMS (EI): *m*/*z* calcd for $C_{13}H_{15}NO$: 201.1154; found: 201.1151. Anal. Calcd for $C_{l3}H_{15}NO$: C, 77.58; H, 7.51; N, 6.96. Found: C, 77.84; H, 7.51; N, 7.12.[4]
4. $PhI(OAc)_2$ (97.0%) and 1,2-dichloroethane (99.5%) were purchased from Sigma Aldrich, Co. and used as received. Ethanol (99.5%) was purchased from Acros Organic and used as received. $PtCl_2$ (98.0%) was purchased from Strem Chemicals and used as received.
5. The present procedure represents a modification of a previously published procedure.[5]
6. Column chromatography was carried out using SILICYCLE SiliaFlash P60 silica gel. A glass column (5 x 40 cm) was slurry-packed with 200 g of silica gel in hexane. The compound was loaded as a solution in a

small amount of CH_2Cl_2, and eluted first with hexane, then hexane:EtOAc, 50:1 (ca. 0.5 L), and then hexane:EtOAc, 25:1 (ca. 2 L). The fractions containing the desired product **2** were combined and concentrated by rotary evaporation at 30 °C (15–20 mmHg) and dried under high vacuum at 23 °C (5–10 mmHg).

7. The procedure was performed at half-scale in 88% by the checkers. Characterization data for compound **2**: TLC R_f = 0.30 (hexanes:EtOAc, 25:1); mp 34–35 °C (colorless prisms from hexane, extensive drying is required); ^{1}H NMR ($CDCl_3$, 500 MHz) δ: 0.97 (t, *J* = 7.5 Hz, 3 H), 1.45 (sex, *J* = 7.5 Hz, 2H), 1.48 (t, *J* = 7.5 Hz, 3 H), 1.69 (quint, *J* = 7.5 Hz, 2 H), 3.01 (t, *J* = 7.5 Hz, 2H), 4.49 (q, *J* = 7.0 Hz, 2 H), 6.34 (s, 1 H), 7.18–7.23 (m, 2 H), 7.44 (dd, *J* = 7.4, 1.8 Hz, 1 H), 8.09 (d, *J* = 7.4 Hz, 1 H); ^{13}C NMR ($CDCl_3$, 126 MHz) δ: 14.0, 14.4, 22.6, 29.8, 31.1, 63.0, 107.5, 115.7, 119.8, 122.9, 123.4, 129.6, 136.6, 142.6, 152.1. IR ($CHCl_3$, cm^{-1}) 2958, 2871, 1736, 1593, 1568, 1456, 1398, 1378, 1323, 1258, 1211, 1118, 1081, 807, 766, 746. HRMS (EI): *m*/*z* calcd for $C_{15}H_{19}NO_2$: 245.1416; found: 245.1413. Anal. Calcd for $C_{15}H_{19}NO_2$: C, 73.44; H, 7.81; N, 5.71. Found: C, 73.78; H, 7.89; N, 5.87.[6]
8. *p*-Tolualdehyde (98.0%) was purchased from SigmaAldrich, Co. and used as received.
9. BF_3•Et_2O (95.0%) was purchased from Sigma Aldrich, Co. and used as received.
10. **CAUTION**: A large amount of carbon dioxide is generated in this extraction and appropriate care should be taken to release pressure from the funnel.
11. Column chromatography was carried out using SILICYCLE SiliaFlash P60 silica gel. A glass column (6 x 40 cm) was slurry-packed with 300 g of silica gel. The compound was loaded as a solution in a small amount of CH_2Cl_2, and the column eluted with hexane, then hexane:EtOAc, 50:1 (ca. 0.75 L), and then hexane:EtOAc, 30:1 (ca. 1 L). The R_f value of *p*-tolualdehyde is very close to that of the desired product. It is useful to take note of the distinct UV response of the product (i.e. a blue spot). The fractions containing the desired product **3** were combined and concentrated by rotary evaporation at 30 °C (15–20 mmHg) and dried under high vacuum at 23 °C (5–10 mmHg).
12. The procedure was performed at half-scale in 77% by the checkers. Characterization data for compound **3**: TLC R_f = 0.30 (hexanes:EtOAc, 20:1); slightly yellow oil; ^{1}H NMR ($CDCl_3$, 500 MHz) δ: 0.92 (t, *J* = 7.5 Hz, 3 H), 1.34–1.41 (m, 5 H), 1.43–1.50 (m, 1 H), 1.54–1.62 (m, 1 H), 1.71–

1.81 (m, 2 H), 2.22 (s, 3 H), 3.12 (t, J = 6.5 Hz, 1 H), 4.32–4.46 (m, 2 H), 5.98 (s , 1 H), 7.00 (d, J = 7.0 Hz, 2 H), 7.04–7.09 (m, 3 H), 7.46 (t, J = 7.0 Hz, 1 H), 7.89–7.90 (m, 2 H); ^{13}C NMR ($CDCl_3$, 126 MHz) δ: 13.9, 14.5, 20.9, 22.5, 29.3, 29.8, 51.1, 59.7, 62.8, 123.5, 123.8, 126.6, 127.4, 129.3, 134.4, 135.5, 137.1, 141.2, 155.1, 195.8. IR ($CHCl_3$, cm^{-1}) 3036, 3007, 2961, 2932, 1706, 1682, 1601, 1479, 1460, 1396, 1381, 1321, 1298, 1269, 1242, 1196, 1049. MS (EI): m/z = 365 (M^+). HRMS (EI): m/z calcd for $C_{23}H_{27}NO_3$: 365.1991; found: 365.1985. Anal. Calcd for $C_{23}H_{27}NO_3$: C, 75.59; H, 7.45; N, 3.83. Found: C, 75.89; H, 7.36; N, 3.75.

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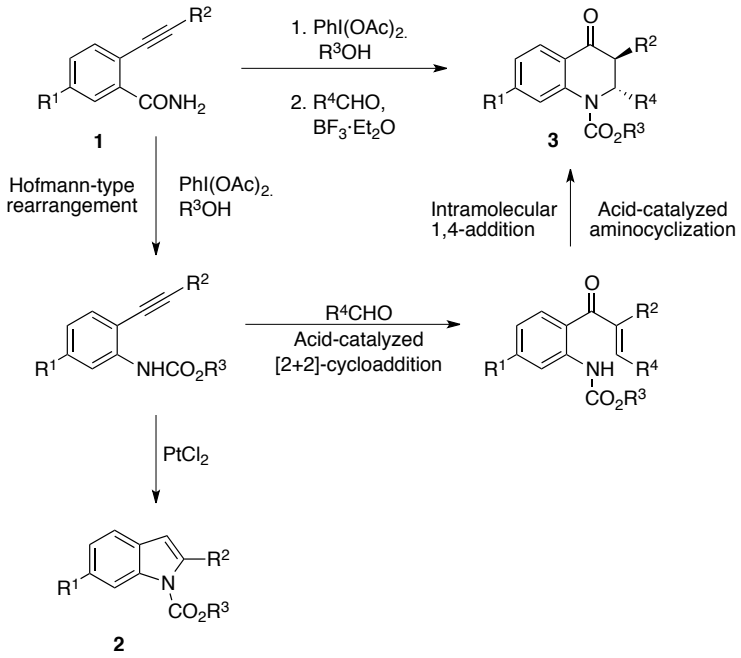
Discussion

Synthesis of heterocyclic compounds has attracted a great deal of attention due to their biological activities. Metal-catalyzed ring closure of 2-alkynylaniline derivatives is one of the most efficient approaches for the construction of benzo-fused *N*-containing heterocyclic compounds;[7] however, one drawback of the method is the air instability of the amines. We became interested in using 2-alkynylbenzamide **1** for heterocycle formation, since alkynylbenzamides can be converted to amine derivatives via a Hofmann-type rearrangement.[8] The strategy described in this

manuscript for the synthesis of indole **2** involves (1) Hofmann-type rearrangement of 2-alkynylbenzamides **1** using hypervalent iodine reagent $PhI(OAc)_2$ followed by a nucleophilic addition of an alcohol to an isocyanate intermediate,[9] and (2) platinum(II)-catalyzed 5-*endo* cyclization of carbamate nitrogen atom toward an alkyne functionality (Scheme 1). Similarly, the strategy for the synthesis of quinolone[10] **3** involves (1) Hofmann-type rearrangement of 2-alkynylbenzamides **1** followed by a nucleophilic addition of an alcohol to an isocyanate intermediate, (2) acid-catalyzed intermolecular [2+2]-cycloaddition between the carbon-carbon triple bonds of carbamates and aldehydes, and (3) acid-catalyzed intramolecular aminocyclization to the α,β-unsaturated ketones.[11]

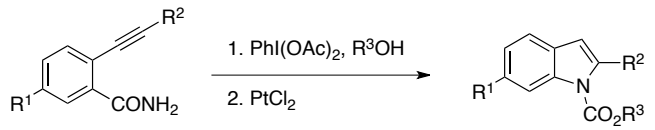
Scheme 1. Heterocycle Formation



The present procedure provides easy access to *N*-carboxylated-2-substituted indoles **2** via a one-pot tandem reaction (Table 1).[5] The electronic nature of the substituents on the aromatic ring does not affect the reaction; in the presence of electron-withdrawing groups or electron-donating groups, the yields of indoles are within the range of 82–91 % (entries 1–3). Alkynylbenzamide, bearing a phenyl group on the acetylene terminus, also

provides the corresponding indole (entry 4). The terminal alkyne is not suitable for this reaction (entry 8).

Table 1. One-pot indole synthesis from benzamide

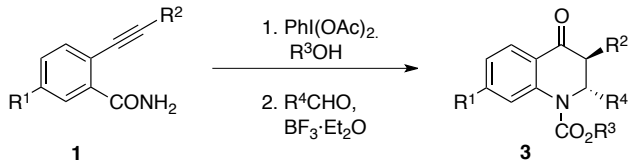


1 **2**

entry	R^1	R^2	R^3	yield (%)
1	F	*n*-Bu	Et	84
2	NO_2	*n*-Bu	Et	91
3	OMe	*n*-Bu	Et	82
4	H	Ph	Et	84
5	H	*p*-Tol	Et	66
6	H	$-(CH_2)_3OTs$	Et	100
7	H	*n*-Bu	Bn	90
8	H	H	Et	33

In 2,3-dihydro-4-quinolone **3** synthesis, moderate yields and high *trans*-selectivities are observed (Table 2).[11] In the presence of either electron-donating or moderately electron-withdrawing substituents R^1 on the aromatic ring of 2-alkynylbenzamides **1**, the yields of desired products range from 72–80% (Table 2, entries 1–3). However, the presence of the strongly electron-withdrawing nitro group on the aromatic ring hinders the reaction (entry 4). For *p*-cyanobenzaldehyde or *p*-nitrobenzaldehyde, a higher temperature of 90 °C is required (entries 10 and 11). The reaction can tolerate aliphatic aldehydes (entries 12 and 13). Terminal alkyne is also suitable for this reaction (entry 14). The use of benzophenone instead of aldehydes fails to give the desired product (entry 15).

Table 2. One-pot 4-quinolone synthesis from benzamide



1 3

entry	R^1	R^2	R^3	R^4	temp (°C)	yield (%) (*trans:cis*)
1	H	*n*-Bu	Et	*p*-Tol	60	78 (20:1)
2	OMe	*n*-Bu	Et	*p*-Tol	60	72 (7:1)
3	F	*n*-Bu	Et	*p*-Tol	60	80 (38:1)
4	NO_2	*n*-Bu	Et	*p*-Tol	60	0
5	H	*n*-Bu	Bn	*p*-Tol	60	73 (8:1)
6	H	*n*-Bu	Me	Ph	60	72 (28:1)
7	OMe	*n*-Bu	Et	Ph	60	52 (11:1)
8	H	Ph	Me	*p*-Tol	60	73 (*trans*)
9	H	Ph	Et	*p*-Tol	60	66 (*trans*)
10	H	*n*-Bu	Et	*p*-CNC_6H_4	90	82 (*trans*)
11	H	*n*-Bu	Et	*p*-$NO_2C_6H_4$	90	80 (*trans*)
12	H	*n*-Bu	Et	hexyl	60	61 (20:1)
13	H	*n*-Bu	Et	cyclohexyl	60	87 (99:1)
14	H	H	Et	*p*-Tol	60	85
15	H	*n*-Bu	Et	(PhCOPh)	90	0

References

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Appendix
Chemical Abstracts Nomenclature (Registry Number)

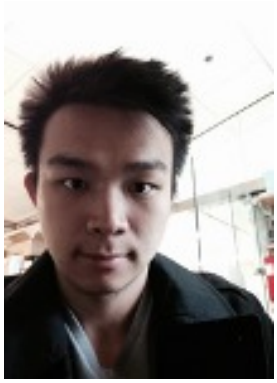
2-(1-Hexynyl)benzamide: Benzamide, 2-(1-hexyn-1-yl)-; (110166-74-0)
Ethyl 2-butyl-1H-indole-1-carboxylate: 1H-Indole-1-carboxylic acid, 2-butyl-, ethyl ester; (221353-60-2)
Ethyl 3-butyl-4-oxo-2-p-tolyl-3,4-dihydroquinoline-1(2H)-carboxylate: 1(2H)-Quinolinecarboxylic acid, 3-butyl-3,4-dihydro-2-(4-methylphenyl)-4-oxo-, ethyl ester, (2*R*,3*S*)-*rel*-; (1337988-00-7)

2-Iodobenzamide; (3930-83-4)
Triphenylphosphine; (603-35-0)
Copper(I) iodide; (7681-65-4)
Palladium(II) acetate; (3375-31-3)
Triethylamine; (121-44-8)
Hexyne; (928-49-4)
(Diacetoxyiodo)benzene; (3240-34-4)
Platinum(II) chloride; (10025-65-7)
p-Tolualdehyde; (104-87-0)
Boron trifluoride diethyl ethereate; (109-63-7)

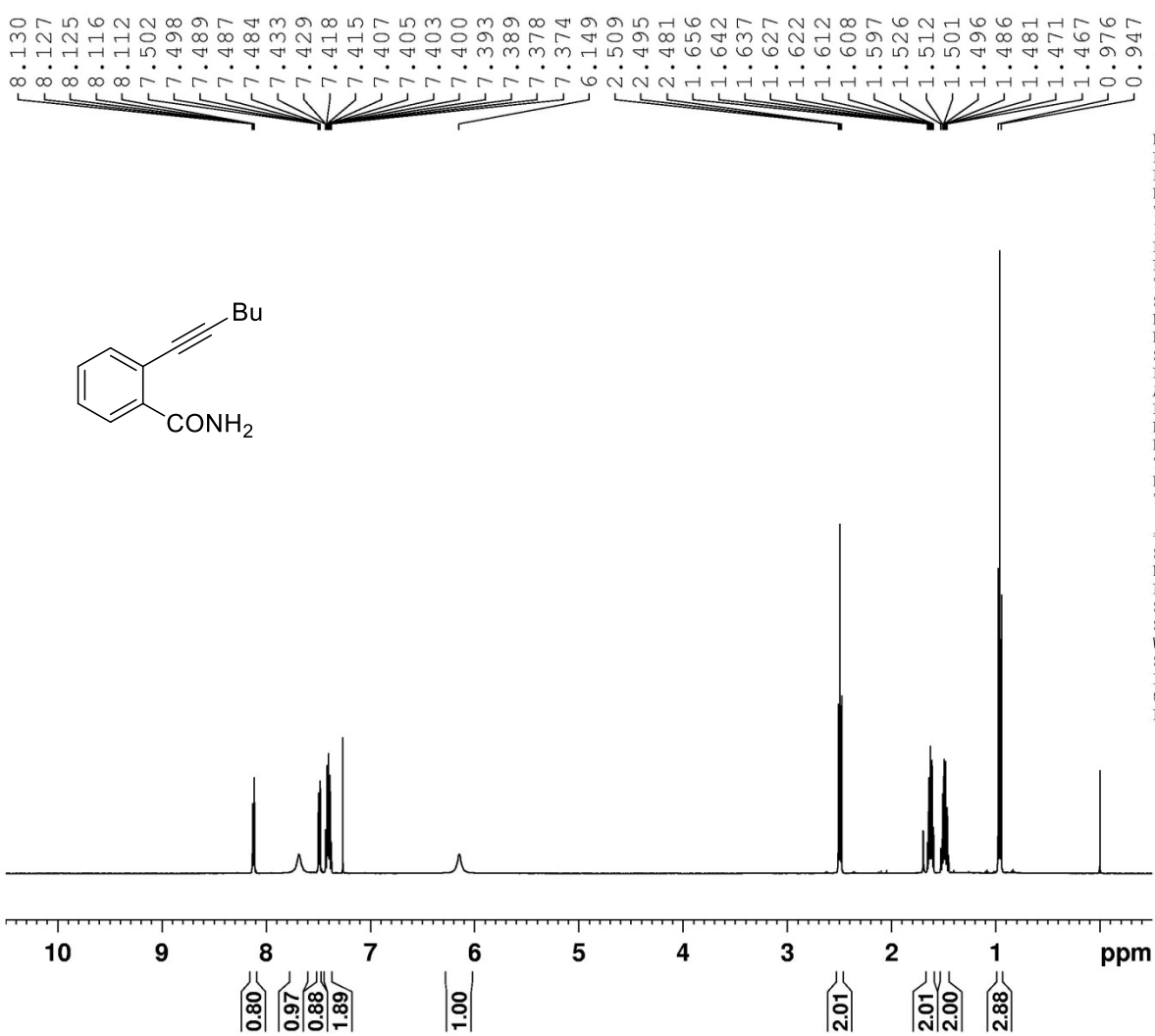
Reiko Yanada received both her B.S. degree (1977) and M.S. degree (1979) from Toyama University (with Eiichi Yoshii) and her Ph.D. degree (1988) from Kyoto University (with Fumio Yoneda). After working at the Dyson Perrins Laboratory of University of Oxford with Professor S. G. Davies, she was promoted to assistant professor, lecturer, and then to associate professor at Kyoto University. She became a professor of organic chemistry at Hiroshima International University in 2006. Her current research interest is in the development of new synthetic organic methodologies utilizing tandem reaction.

Noriko Okamoto obtained her B.S. degree (2004), M.S. degree (2006), and Ph.D. degree (2011) from Hiroshima University (with Kei Takeda). She joined Professor Reiko Yanada's research group at Hiroshima International University in 2008 as an assistant professor. Her current research interest is in the development of new synthetic reactions. She was the recipient of the Chugoku-Shikoku Branch of Pharmaceutical Society of Japan Award for Young Scientists (2012).

Kei Takeda is professor of organic chemistry at Hiroshima University. He was born in 1952 and received both his B.S. degree (1975) and M.S. degree (1977) from Toyama University (with Eiichi Yoshii) and his Ph.D. degree (1980) from the University of Tokyo (with Toshihiko Okamoto). In 1980, he joined the faculty at Toyama Medical and Pharmaceutical University (Prof. Yoshii's group). After working at MIT with Professor Rick L. Danheiser (1988-1989), he was promoted to Lecturer (1989) and then to Associate Professor (1996). He became a professor of Hiroshima University in 2000. His research interests are in the invention of new synthetic reactions and chiral carbanion chemistry. He was the recipient of the Sato Memorial Award (1998) and the 41st Senji Miyata Foundation Award.



Hang Chu was born in China in 1991. He is currently pursuing a joint BS/MS degree in chemistry at the University of Chicago. He began his research in synthetic organic chemistry under the supervision of Dr. Viresh Rawal in 2011. Currently, he is working on developing a diene to achieve "meta"-selective Diels Alder reactions. He plans to continue his studies in organic chemistry – particularly asymmetric catalysis - by pursuing a doctoral degree.



BRUKER

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PROCNO	1
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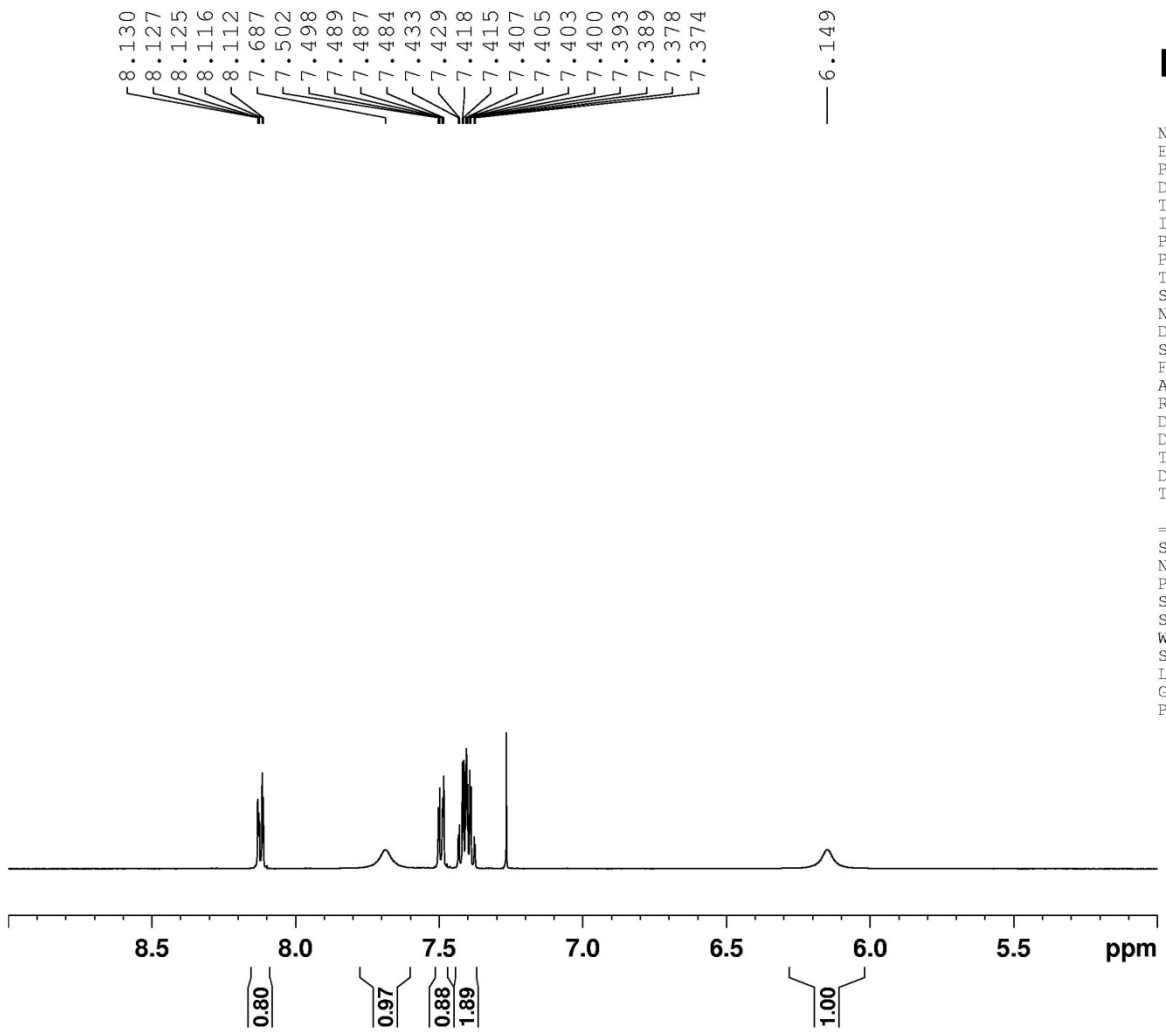
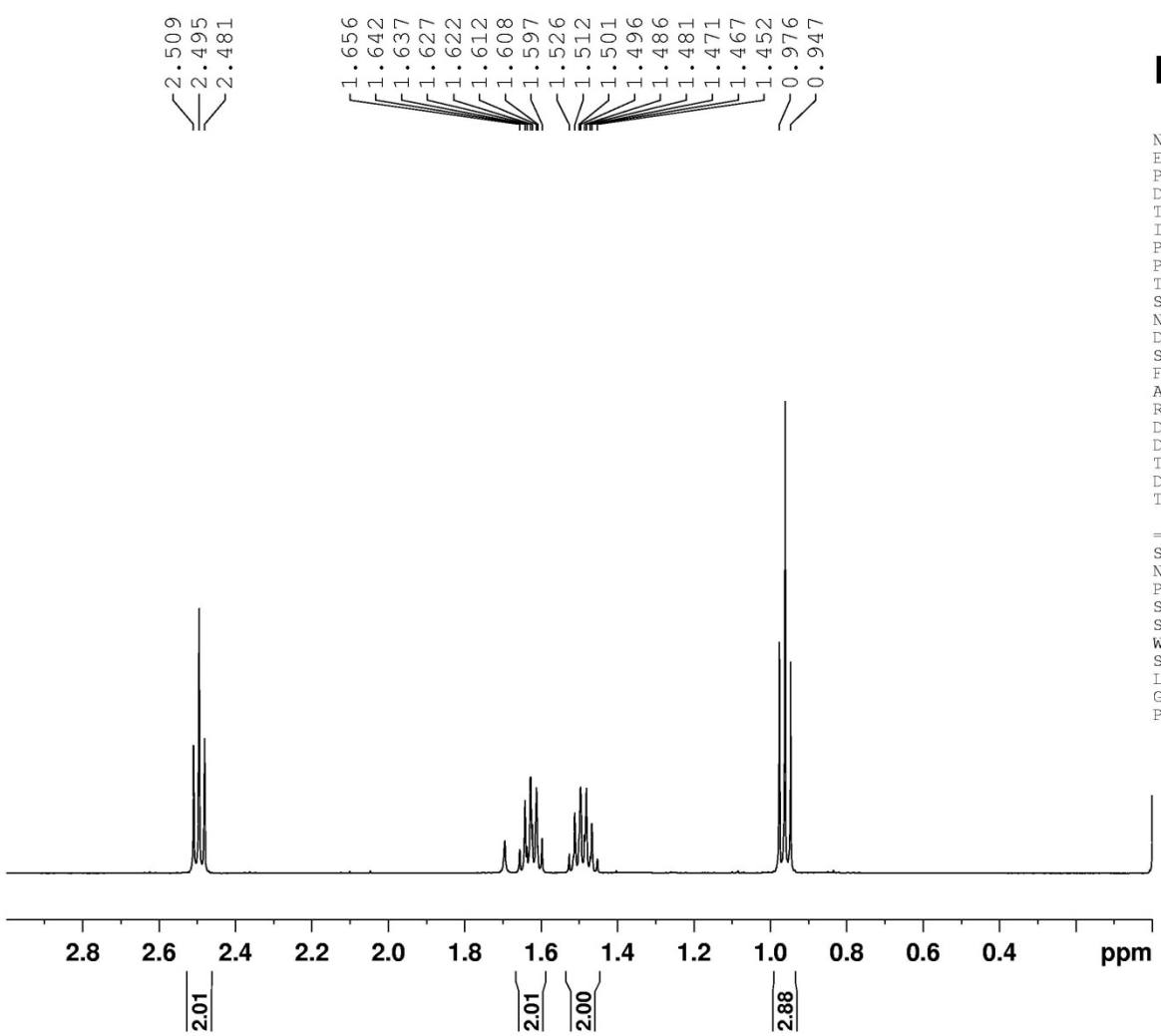
PROBHD	5 mm PATXI 1H/
PULPROG	zg
TD	59998
SOLVENT	CDCl3
NS	16
DS	0
SWH	10000.000 Hz
FIDRES	0.166672 Hz
AQ	2.9999499 sec
RG	87.71
DW	50.000 usec
DE	10.00 usec
TE	295.7 K
D1	3.00000000 sec
TD0	1
======== CHANNEL f1 ========	
SFO1	500.1330885 MHz
NUC1	1H
P1	8.00 usec
SI	65536
SF	500.1300104 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



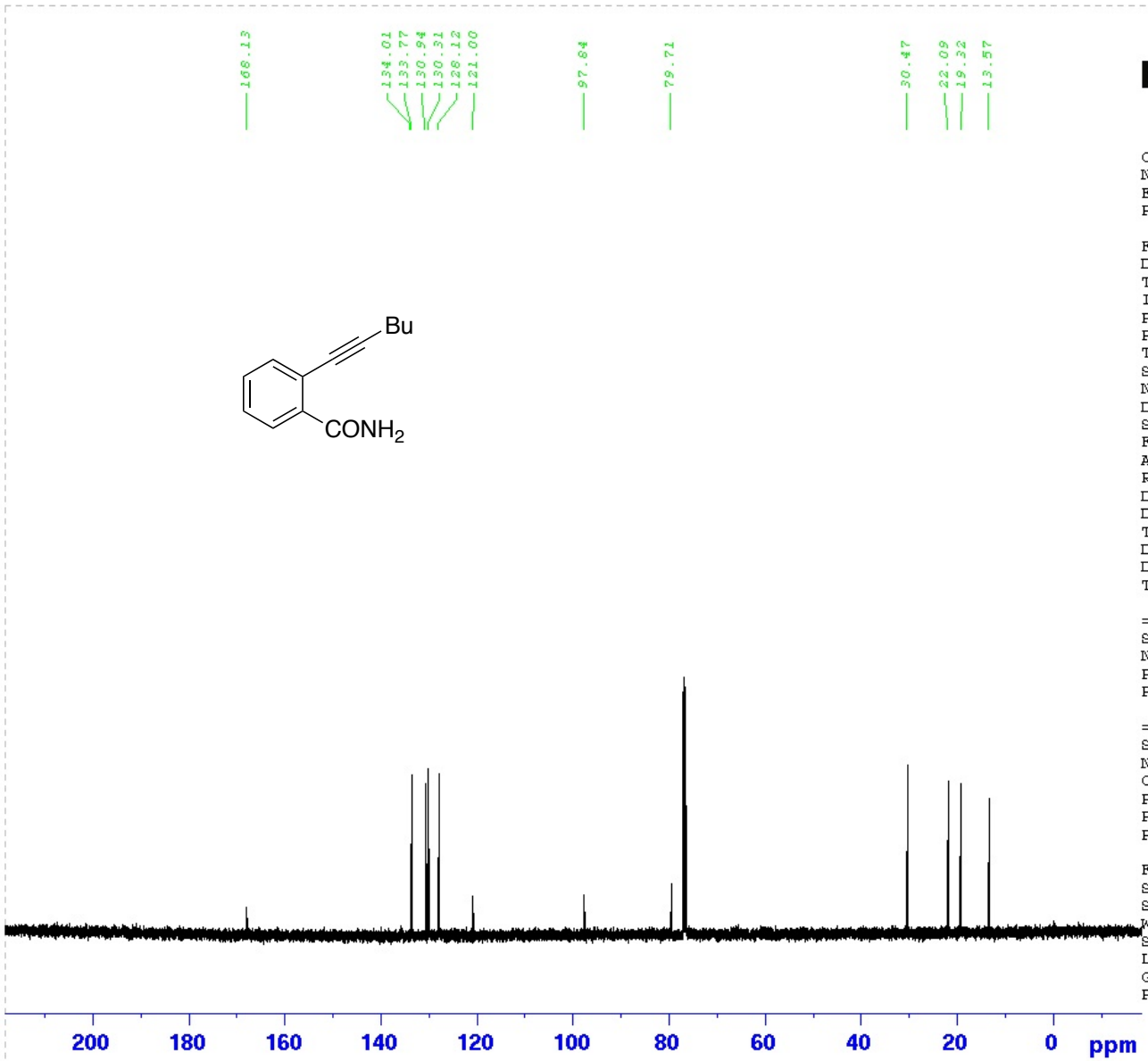
BRUKER

NAME	HC-2-ORGSYN-A-1H
EXPNO	1
PROCNO	1
Date_	20130404
Time	18.55
INSTRUM	spect
PROBHD	5 mm PATXI 1H/
PULPROG	zg
TD	59998
SOLVENT	CDCl3
NS	16
DS	0
SWH	10000.000 Hz
FIDRES	0.166672 Hz
AQ	2.9999499 sec
RG	87.71
DW	50.000 usec
DE	10.00 usec
TE	295.7 K
D1	3.00000000 sec
TD0	1
======== CHANNEL f1 ========	
SFO1	500.1330885 MHz
NUC1	1H
P1	8.00 usec
SI	65536
SF	500.1300104 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



BRUKER

NAME	HC-2-ORGSYN-A-1H
EXPNO	1
PROCNO	1
Date_	20130404
Time	18.55
INSTRUM	spect
PROBHD	5 mm PATXI 1H/
PULPROG	zg
TD	59998
SOLVENT	CDCl3
NS	16
DS	0
SWH	10000.000 Hz
FIDRES	0.166672 Hz
AQ	2.9999499 sec
RG	87.71
DW	50.000 usec
DE	10.00 usec
TE	295.7 K
D1	3.00000000 sec
TD0	1
======== CHANNEL f1 ========	
SFO1	500.1330885 MHz
NUC1	1H
P1	8.00 usec
SI	65536
SF	500.1300104 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



Current Data Parameters
NAME ORGSYN-A-13C
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20131026
Time 13.55
INSTRUM spect
PROBHD 5 mm PATXI 1H/
PULPROG zgdc
TD 178568
SOLVENT CDCl3
NS 256
DS 0
SWH 29761.904 Hz
FIDRES 0.166670 Hz
AQ 2.9999423 sec
RG 196.79
DW 16.800 usec
DE 10.00 usec
TE 296.7 K
D1 3.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 125.7703643 MHz
NUC1 13C
P1 14.00 usec
PLW1 170.00000000 W

======== CHANNEL f2 ========
SFO2 500.1320005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 12.19999981 W
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F2 - Processing parameters
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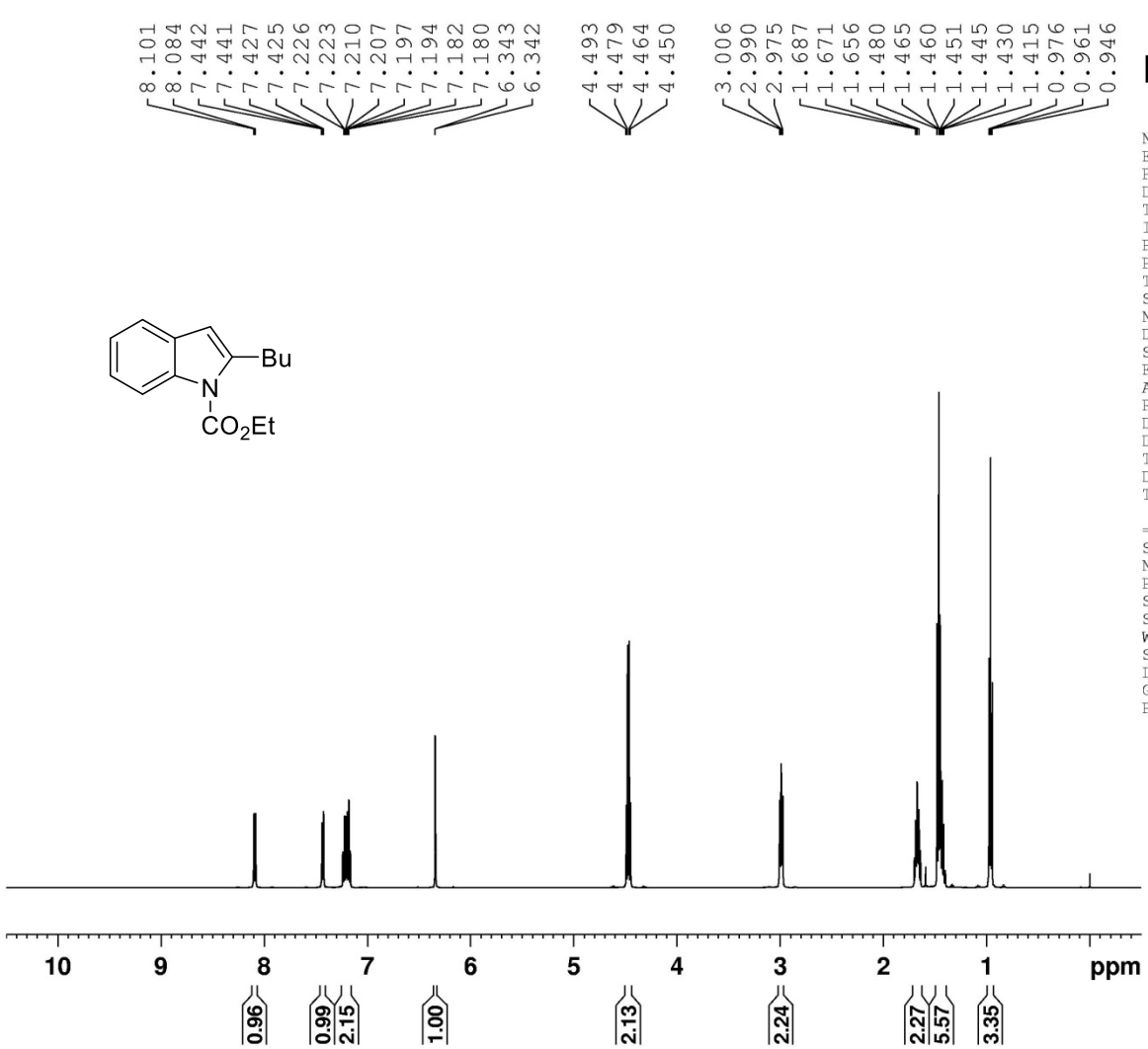

BRUKER

NAME	HC-ORGSYN-B-1H
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PROCNO	1
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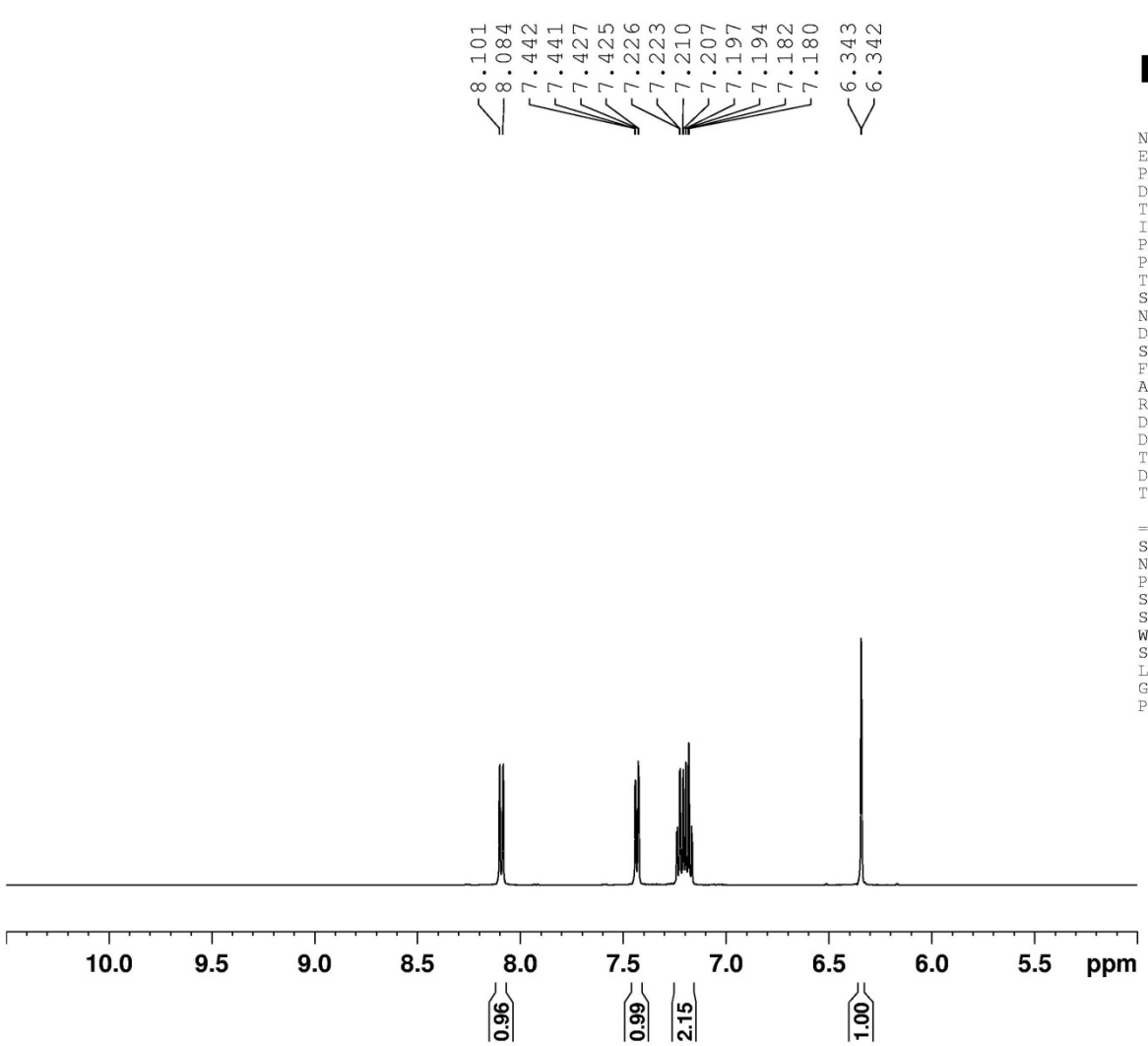
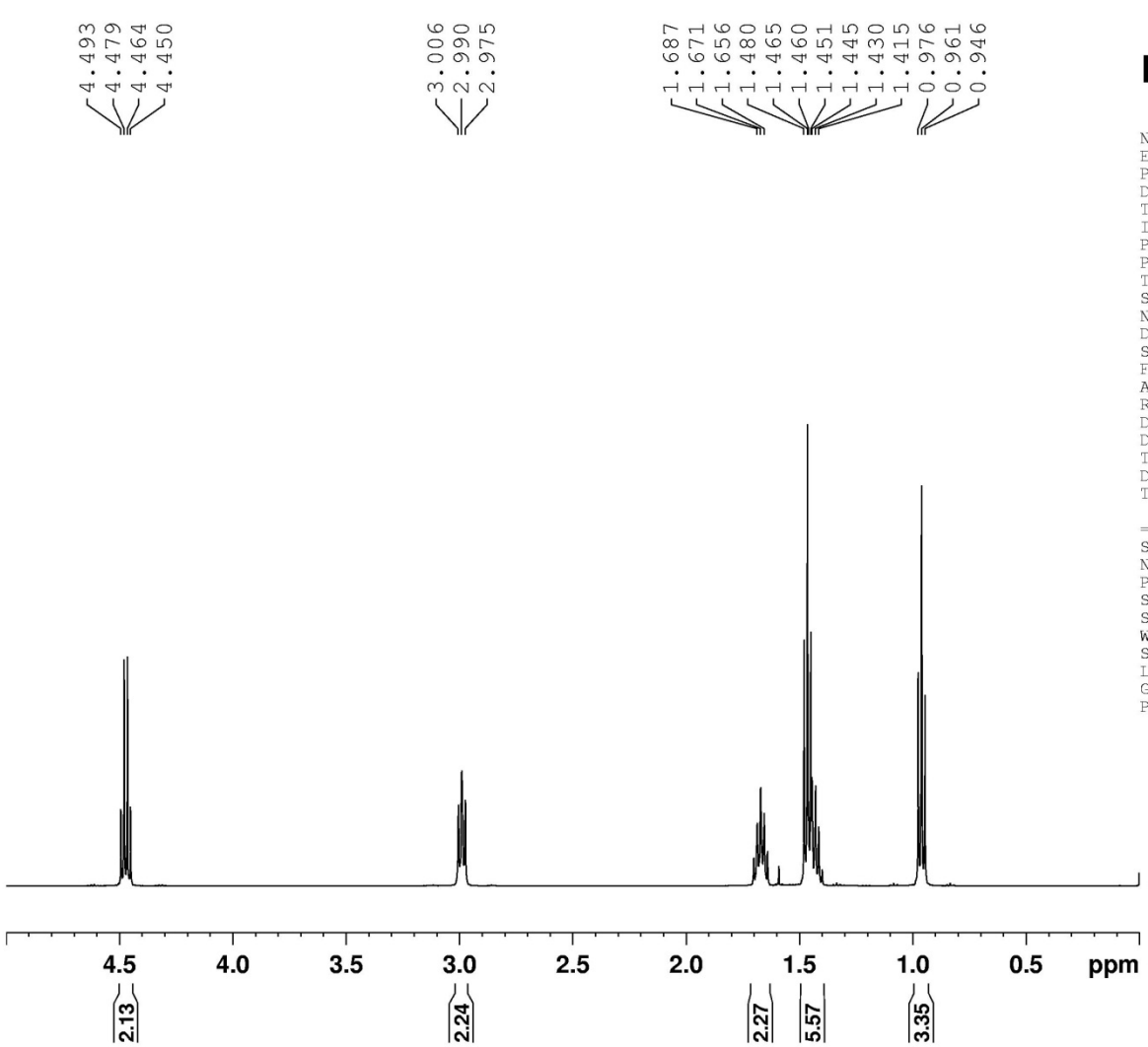
PULPROG	zg
TD	59998
SOLVENT	CDCl3
NS	16
DS	0
SWH	10000.000 Hz
FIDRES	0.166672 Hz
AQ	2.9999499 sec
RG	12.63
DW	50.000 usec
DE	10.00 usec
TE	294.6 K
D1	3.00000000 sec
TD0	1
======== CHANNEL f1 ========	
SFO1	500.1330885 MHz
NUC1	1H
P1	8.00 usec
SI	65536
SF	500.1300378 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



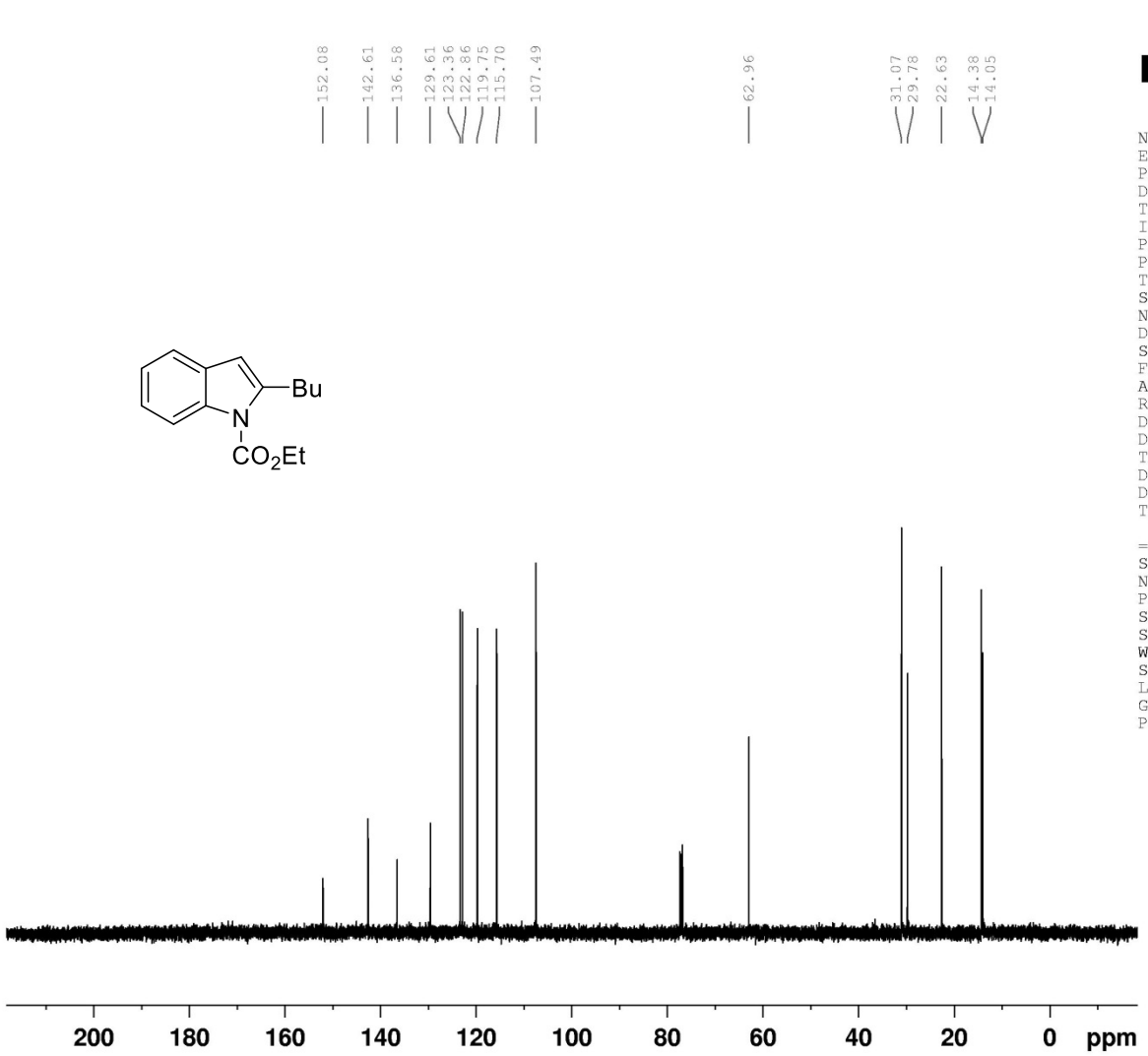
BRUKER

NAME	HC-ORGSYN-B-1H
EXPNO	1
PROCNO	1
Date_	20130611
Time	14.49
INSTRUM	spect
PROBHD	5 mm PATXI 1H/
PULPROG	zg
TD	59998
SOLVENT	CDCl3
NS	16
DS	0
SWH	10000.000 Hz
FIDRES	0.166672 Hz
AQ	2.9999499 sec
RG	12.63
DW	50.000 usec
DE	10.00 usec
TE	294.6 K
D1	3.00000000 sec
TD0	1
======== CHANNEL f1 ========	
SFO1	500.1330885 MHz
NUC1	1H
P1	8.00 usec
SI	65536
SF	500.1300378 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



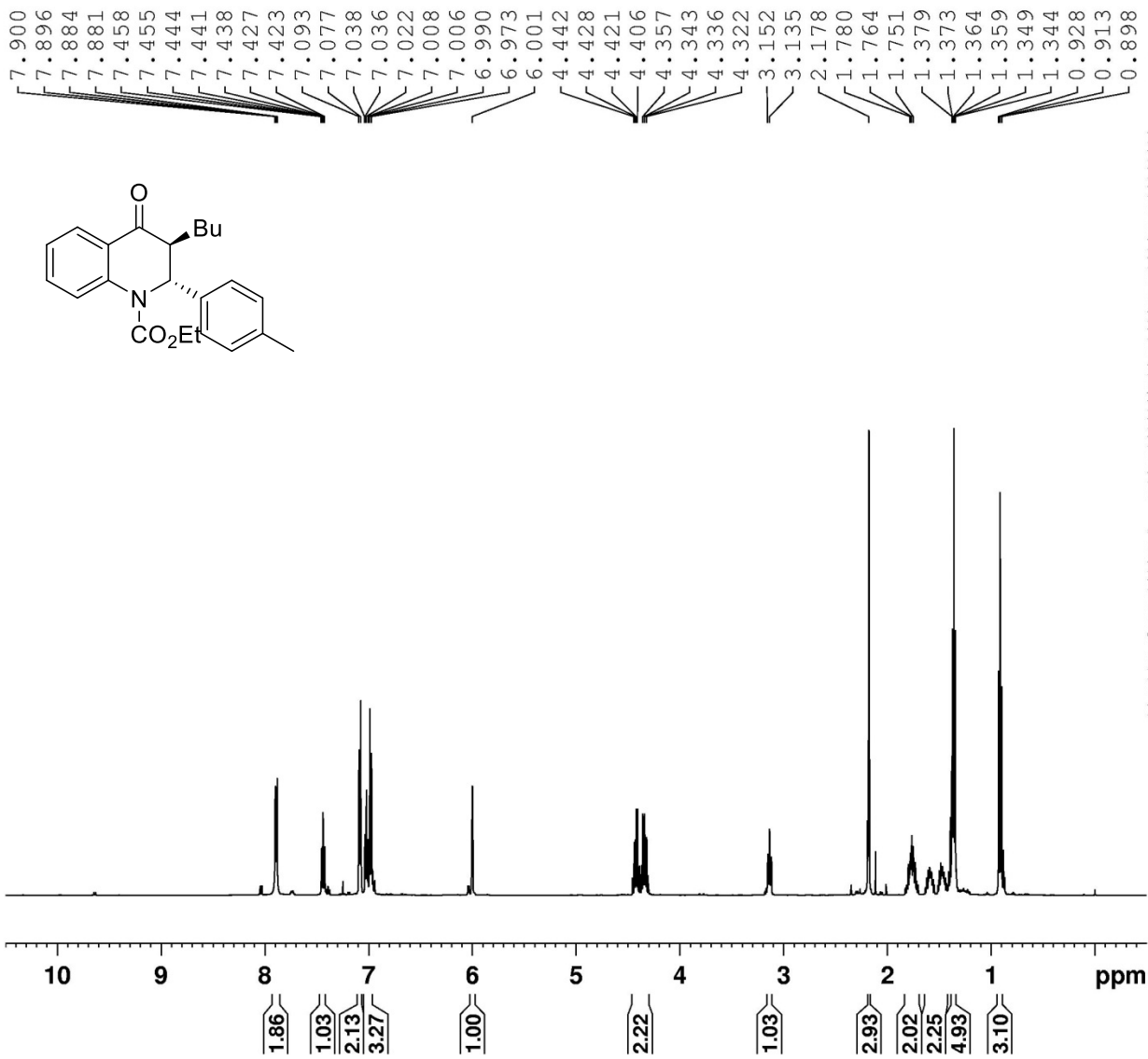
BRUKER

NAME	HC-ORGSYN-B-1H
EXPNO	1
PROCNO	1
Date_	20130611
Time	14.49
INSTRUM	spect
PROBHD	5 mm PATXI 1H/
PULPROG	zg
TD	59998
SOLVENT	CDCl3
NS	16
DS	0
SWH	10000.000 Hz
FIDRES	0.166672 Hz
AQ	2.9999499 sec
RG	12.63
DW	50.000 usec
DE	10.00 usec
TE	294.6 K
D1	3.00000000 sec
TD0	1
======== CHANNEL f1 ========	
SFO1	500.1330885 MHz
NUC1	1H
P1	8.00 usec
SI	65536
SF	500.1300378 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



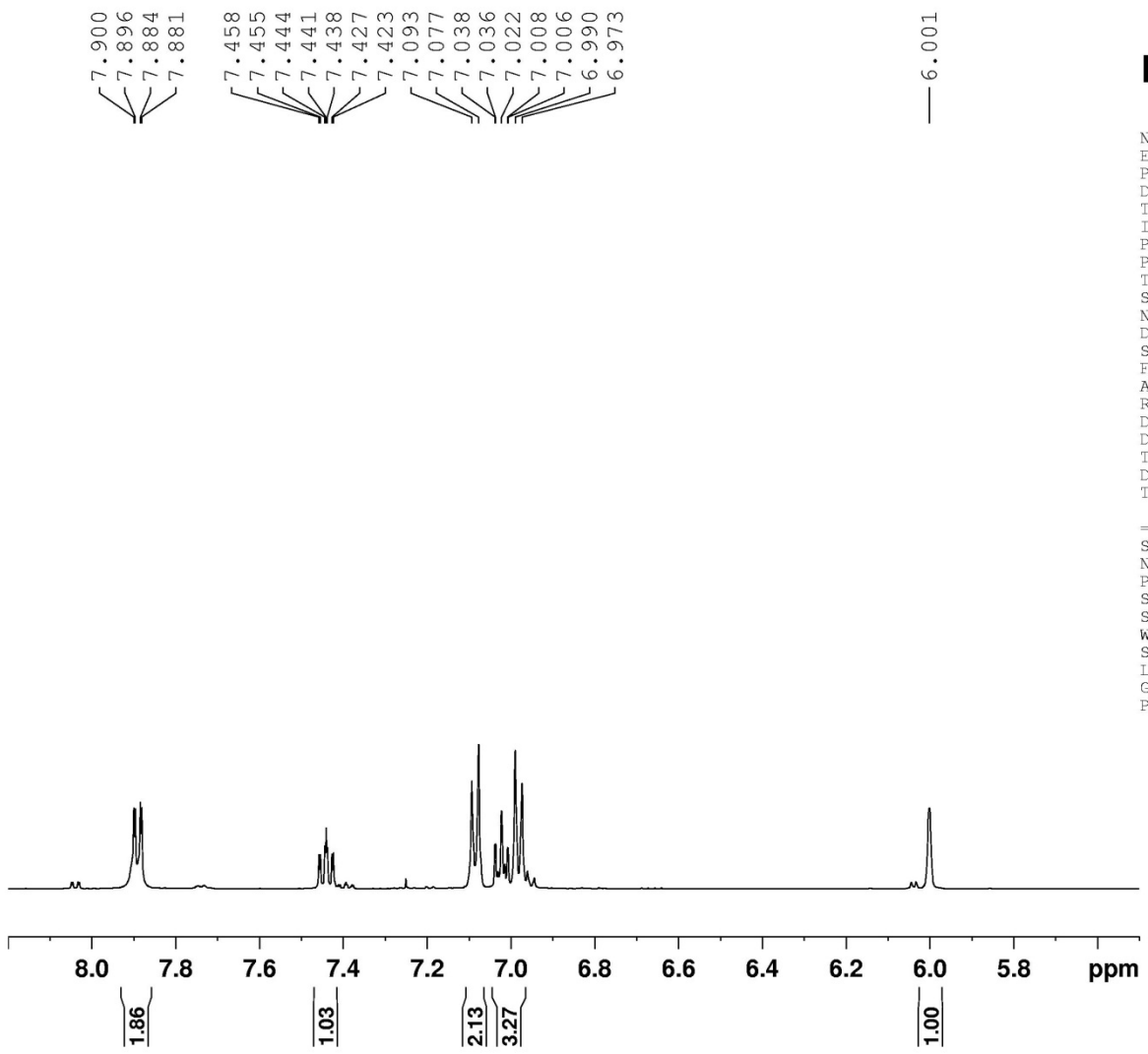
BRUKER

NAME	HC-ORGSYN-B-13C
EXPNO	1
PROCNO	1
Date_	20130611
Time	14.55
INSTRUM	spect
PROBHD	5 mm PATXI 1H/
PULPROG	zgdc
TD	178568
SOLVENT	CDCl3
NS	16
DS	0
SWH	29761.904 Hz
FIDRES	0.166670 Hz
AQ	2.9999924 sec
RG	196.79
DW	16.800 usec
DE	10.00 usec
TE	295.5 K
D1	3.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
SFO1	125.7703643 MHz
NUC1	13C
P1	14.00 usec
SI	131072
SF	125.7577890 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40



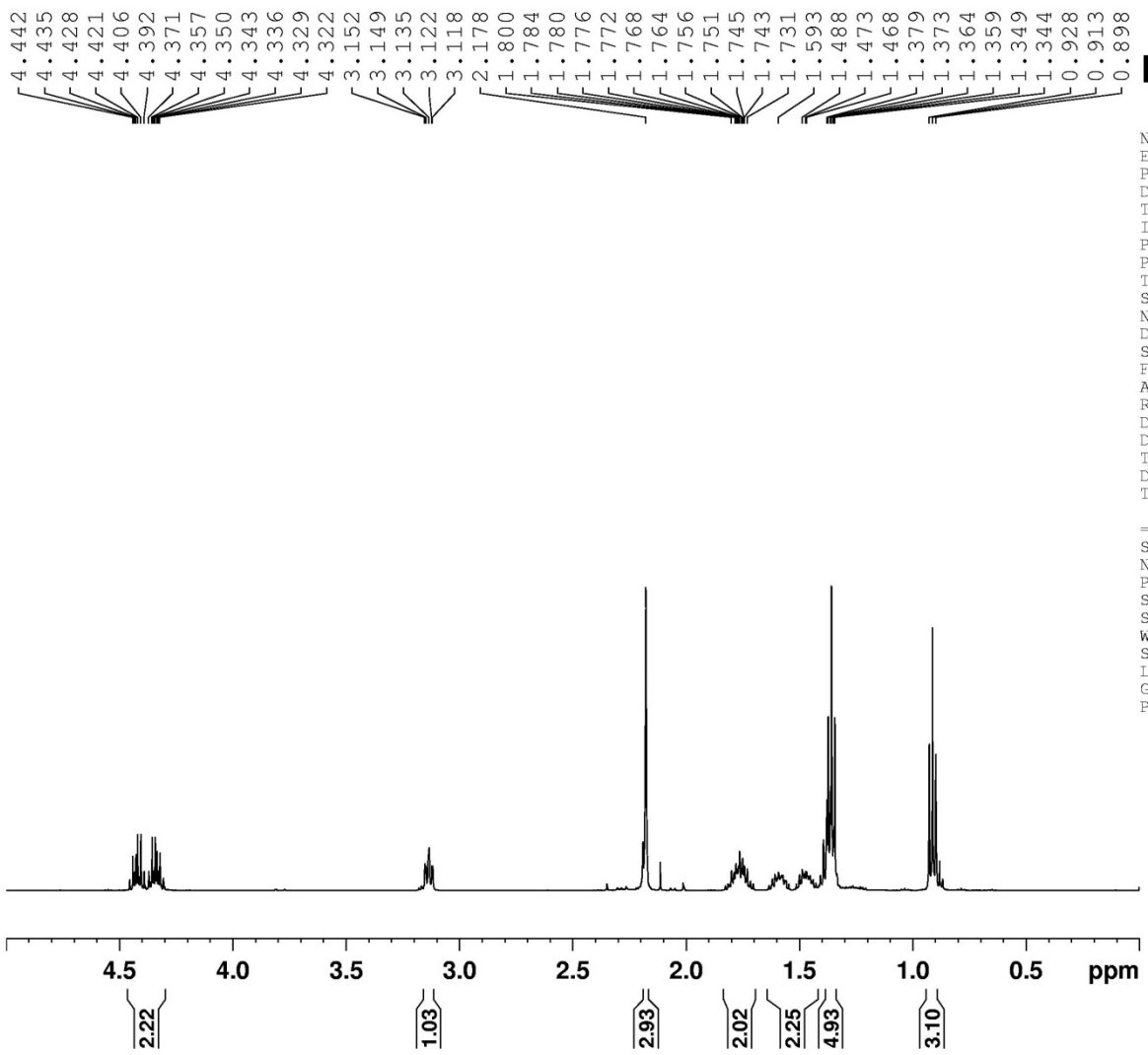
BRUKER

NAME	HC-ORGSYN-C-1H
EXPNO	1
PROCNO	1
Date_	20130522
Time	15.52
INSTRUM	spect
PROBHD	5 mm PATXI 1H/
PULPROG	zg
TD	59998
SOLVENT	CDCl3
NS	16
DS	0
SWH	10000.000 Hz
FIDRES	0.166672 Hz
AQ	2.9999499 sec
RG	5.58
DW	50.000 usec
DE	10.00 usec
TE	294.4 K
D1	4.00000000 sec
TD0	1
======== CHANNEL f1 ========	
SFO1	500.1330885 MHz
NUC1	1H
P1	8.00 usec
SI	65536
SF	500.1300168 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

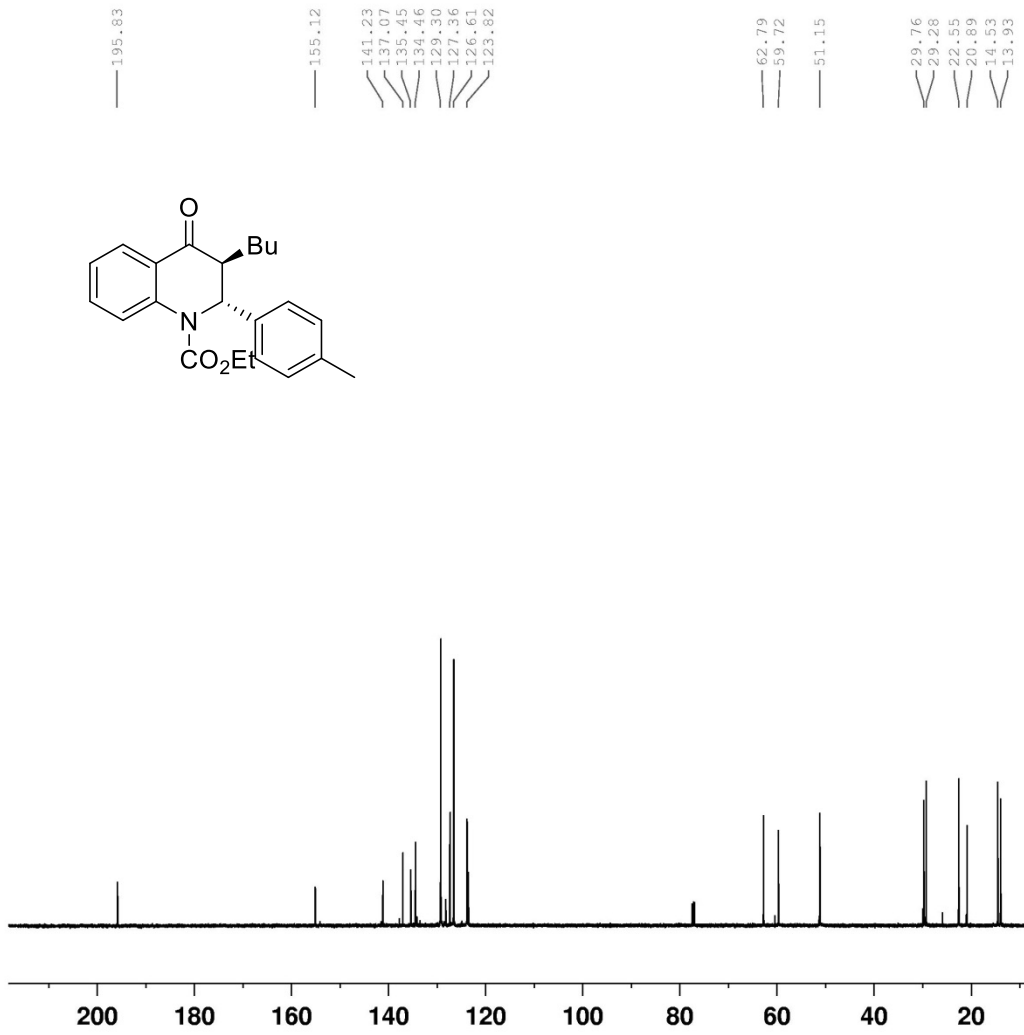


BRUKER

NAME	HC-ORGSYN-C-1H
EXPNO	1
PROCNO	1
Date_	20130522
Time	15.52
INSTRUM	spect
PROBHD	5 mm PATXI 1H/
PULPROG	zg
TD	59998
SOLVENT	CDCl3
NS	16
DS	0
SWH	10000.000 Hz
FIDRES	0.166672 Hz
AQ	2.9999499 sec
RG	5.58
DW	50.000 usec
DE	10.00 usec
TE	294.4 K
D1	4.00000000 sec
TD0	1
======== CHANNEL f1 ========	
SFO1	500.1330885 MHz
NUC1	1H
P1	8.00 usec
SI	65536
SF	500.1300168 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



NAME	HC-ORGSYN-C-1H
EXPNO	1
PROCNO	1
Date_	20130522
Time	15.52
INSTRUM	spect
PROBHD	5 mm PATXI 1H/
PULPROG	zg
TD	59998
SOLVENT	CDCl3
NS	16
DS	0
SWH	10000.000 Hz
FIDRES	0.166672 Hz
AQ	2.9999499 sec
RG	5.58
DW	50.000 usec
DE	10.00 usec
TE	294.4 K
D1	4.00000000 sec
TD0	1
======== CHANNEL f1 ========	
SFO1	500.1330885 MHz
NUC1	1H
P1	8.00 usec
SI	65536
SF	500.1300168 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



BRUKER

NAME	HC-ORGSYN-C-13C
EXPNO	1
PROCNO	1
Date_	20130522
Time	16.04
INSTRUM	spect
PROBHD	5 mm PATXI 1H/
PULPROG	zgdc
TD	178568
SOLVENT	CDCl3
NS	27
DS	0
SWH	29761.904 Hz
FIDRES	0.166670 Hz
AQ	2.9999924 sec
RG	196.79
DW	16.800 usec
DE	10.00 usec
TE	295.3 K
D1	3.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
SFO1	125.7703643 MHz
NUC1	13C
P1	14.00 usec
SI	131072
SF	125.7577890 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40