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Further Respond to Reviewers

(OB-COM-03-2018-000699)

Reviewer 3

The revised manuscript has addressed the main questions properly. It is recommended for publication after a few minor changes.

We are grateful to the reviewer for positive comments and kind suggestions on our revised manuscript. Corresponding changes have been made according to the reviewer's suggestions as bellow:

1, Remove the citation (reference 10) in the sentence "…we would like to report our recent study on the iron-catalysed intermolecular vicinal aminoazidation of alkenes10 (Scheme 1c)." No reference should be cited before "(Scheme 1c)"

We appreciate the reviewer's suggestion, and have remove the cited reference in this sentence.

2, Change the citation 10 to 10a in the sentence "…On the contrary, no matter how much the reaction time was, the yields of 2a remained poor in absence of ligand when changing the iron catalyst to CuCl10 (entry 12),.."

We appreciate the reviewer's suggestion, and have changed the citation 10 to 10a in this sentence.

3, The following paragraph should be revised for clarity. The information is somehow confusing and doesn't fit well the cited references.

"In 2014, Zhang and Studer reported a novel copper-catalysed aminoazidation of styrenes (Scheme 1b),10a which has drawn our attention since organic azides could serve as versatile intermediates to easily transform to other nitrogen-containing functional groups including amines,10, 15 albeit their protocol could only be effective on styrenes and could not react in absence of a ligand. Zhang and Studer also demonstrated that both benzenesulfonyl and azide two different protecting groups of amines, which can be deprotected under different conditions sequentially,10a allowing maximal synthetic flexibility and providing an opportunity to prepare vicinal diamines with two miscellaneously functionalized amino groups. "

Consider the revision as below:

In 2014, Zhang and Studer reported a novel copper-catalysed aminoazidation of styrenes (Scheme 1b),10a which has drawn our attention since organic azides could serve as versatile intermediates to easily transform to other nitrogen-containing functional groups including amines.10, 15 They also demonstrated that both benzenesulfonyl and azide two different protecting groups of amines can be deprotected under different conditions sequentially.10a

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Although it offers maximal synthetic flexibility and an opportunity to prepare vicinal diamines with two miscellaneously functionalized amino groups, this copper-catalysed protocol was only be effective on styrenes and could not react in absence of a ligand.

We appreciate the reviewer's suggestion, and have changed this paragraph as the reviewer suggested, highlighted in yellow background.

4, remove the yellow highlight in the manuscript for the accepted version.

We appreciate the reviewer's kind suggestion. Beside the revised manuscript with yellow highlight to point out the changes we have made, we also provide a DOC file and a PDF file without the yellow highlight for the accepted version.

In summary, we would like to gratefully thank all the editors and reviewers again for the above positive comments and constructive suggestions on our manuscript.

Best regards,

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Journal Name

COMMUNICATION

NFSI-participated Intermolecular Aminoazidation of Alkene through Iron Catalysis†

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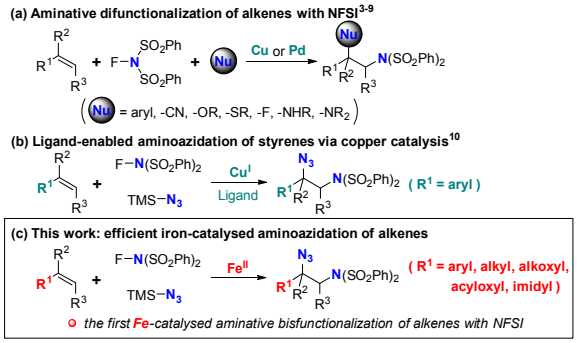
Bowen Lei,[a] Xiaojiao Wang,[a] Lifang Ma,[a] Yan Li,[a] and Ziyuan Li*[a]

An iron-catalysed intermolecular vicinal aminoazidation of alkenes, using *N*-fluorobenzenesulfonimide (NFSI) and trimethylsilyl azide ($TMSN_3$) as the imidating and azidating reagents respectively, is described, which could potentially provide a valuable route toward diverse vicinal diamine derivatives of great significance in medicinal chemistry and organic synthesis. Such iron-catalysed aminative bisfunctionalization of alkene with NFSI has not yet been reported before. Comparing to previously employed copper or palladium catalysts, the iron catalyst, $FeCl_2$, was demonstrated to be a good alternative for its comparable efficiency and broad alkene scope. Preliminary mechanistic study suggested that this iron-catalysed reaction is realized through radical process.

Vicinal diamine is a ubiquitous scaffold of great significance, which widely resides in miscellaneous ligands, organocatalysts, synthetic building blocks, and biologically active natural or artificial molecules.[1] To acquire such significant moiety, many efforts have been spent on transition metal-catalysed 1,2-diamination of alkenes, but the catalysts used in these reactions were generally limited to copper and palladium.[2] Recently, NFSI (*N*-fluorobenzenesulfonimide), which could easily generate an electrophilic disulfonimidyl radical, has been broadly used as an oxidative imidating reagent in transition metal-catalysed or metal-free amination reactions through cascade radical processes. Since Michael group and Liu group disclosed two pioneering works on NFSI-involved diamination[3a] and aminofluorination[3b] of alkene, NFSI has been widely employed as a powerful nitrogen donor in vicinal aminative difunctionalization of alkenes (Scheme 1a)[4-10], such as aminoarylation[4], aminocyanation[5], aminooxygenation[6], amino-thionation[7], aminofluorination[8], diamination[5a, 9], and amino-azidation[10a]. Beside aminative difunctionalization of alkene, Zhang group reported the first two examples on Pd-catalysed oxidative benzylic[11a] and aromatic[11b] C-H aminations with NFSI, followed by the widely applications of this imidating reagent in C-H amination of arenes[12], alkanes[13] and alkenes[14].



Scheme 1 Transition-metal catalyzed aminative functionalization with NFSI

Among these bisfunctionalization of alkenes with NFSI, the diamination and aminoazidation could provide a potential approach toward the above-mentioned significant vicinal diamine moiety. However, all these reactions were Cu-[5a, 9a-b, 10a] or Pd-[9c-f] catalysed, and require assistance of a ligand[5a, 9a,e-f] or an additive[9b-d] to realize the reaction or to guarantee gratifying yields. More importantly, most of these reactions were only effective on active alkenes like styrenes[5a, 9a-b] or allylic ethers[9c]. Michael group reported a series of significant works on 1,2-diamination of inactive alkenes with NFSI,[9d-f] but one of the two amination steps was proceeded intramolecularly, affording aminated *N*-heterocycles only, which limited the diversity of vicinal diamine products. Up to now, no NFSI-participated intermolecular diamination of inactive alkenes has been reported.

In 2014, Zhang and Studer reported a novel copper-catalysed aminoazidation of styrenes (Scheme 1b),[10a] which has drawn our attention since organic azides could serve as versatile intermediates to easily transform to other nitrogen-containing functional groups including amines.[10, 15] They also demonstrated that both benzenesulfonyl and azide group, two different protecting groups of amines, can be deprotected under different conditions sequentially.[10a] Although it offers

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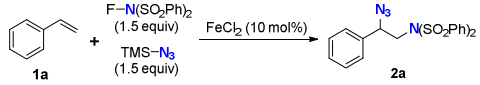
maximal synthetic flexibility and an opportunity to prepare vicinal diamines with two miscellaneously functionalized amino groups, this copper-catalysed protocol was only effective on styrenes and could not react in absence of a ligand. Though limitations were observed, these previous reports have enlightened us to develop new transition metal catalysts with comparable efficiency but broader alkene scope for the NFSI-participated aminative bisfunctionalization. Herein, as an advancement of our previous studies on C-H functionalization[16] including the first iron-catalysed C-H imidation with NFSI[16a], as well as other works on transition metal-catalysed annulations with simple nitrogen donors including azide,[17] we would like to report our recent study on the iron-catalysed intermolecular vicinal aminoazidation of alkenes (Scheme 1c). To our knowledge, the iron-catalysed aminative bisfunctionalization of alkenes with NFSI has not been disclosed previously.

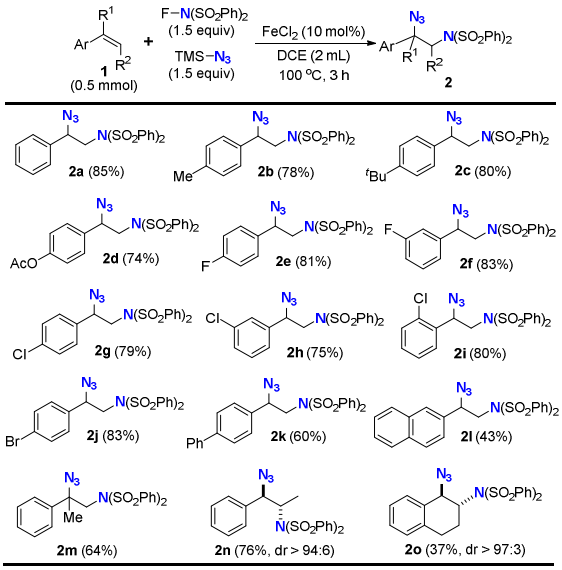
Table 1 Optimization of the reaction conditions[a]



Entry	Solvent (2 ml)	Temperature (°C)	Time (h)	Yield (%)[b]
1	DCE	70	15	44
2	DMF	70	15	trace
3	DME	70	15	0
4	CH_3CN	70	15	35
5	Dioxane	70	15	0
6	Toluene	70	15	trace
7	DCE	100	15	73
8[c]	DCE	100	15	84
9[c, d]	DCE	100	15	78
10[c]	DCE	100	5	(87)
11[c]	**DCE**	**100**	**3**	**85 (90)**
12[c, e]	DCE	100	3	(14)
13[c, f]	DCE	100	3	0

[a]*Reaction conditions: styrene* ***1a*** *(0.5 mmol), NFSI (0.75 mmol), $TMSN_3$ (0.75 mmol), $FeCl_2$ (0.05 mmol) in solvent (2 mL) in air (1 atm).* [b]*Isolated yields of* ***2a****. The yields in the parentheses are determined by NMR using CH_2Br_2 as an internal standard.* [c]*Conducted under N_2 (1 atm).* [d]*With NFSI (1 mmol) and $TMSN_3$ (1 mmol).* [e]*Catalyzed by CuCl (0.05 mmol) instead of $FeCl_2$.* [f]*In the absence of $FeCl_2$.*

Our investigation was commenced with the screening of reaction conditions for this novel iron-catalysed aminoazidation using styrene **1a** as the alkene substrate, and the results are summarized in Table 1. Initial trial with 10 mol% of $FeCl_2$ at 70 °C in air afforded corresponding aminoazidated product **2a** in 44% yield after 15 hours (entry 1). Several commonly used solvents were screened (entries 2-6), and most gave no or only trace product **2a** under the same conditions, except for acetonitrile with 35% yield (entry 4), indicating that DCE is the best solvent. The yield of **2a** was considerably elevated when reaction temperature was increased to 100 °C (entry 7), and could be further promoted to 84% when this aminoazidation was conducted under N_2 (1 atm), suggesting molecular dioxygen is detrimental to this reaction (entry 8). Kinetic experiments were then conducted, in order to investigate whether the relatively long reaction time is necessary.[18] The results showed that the reaction catalysed by $FeCl_2$ was fast, providing **2a** in 90% NMR yield after 3 hours (entry 11), and no ligand or additive is required, indicating that elevating temperature solely is sufficient to promote the efficiency of this iron-catalysed aminoazidation. Prolonging the reaction time could not further promote the yield (entry 10). On the contrary, no matter how much the reaction time was, the yields of **2a** remained poor in absence of ligand when changing the iron catalyst to CuCl[10a] (entry 12), suggesting that, for the copper-catalysed reaction, increasing temperature is less helpful than the employment of an appropriate ligand. In addition, no aminoazidated product was generated when the reaction was conducted without $FeCl_2$ (entry 13). Therefore, the reaction conditions in entry 11 were selected as the optimized conditions for subsequent exploration of alkene scope.



Scheme 2 Fe-catalysed aminoazidation of styrenes with NFSI. Reaction conditions: see **entry 11, Table 1**. Isolated yields.

With the optimized conditions established, a variety of styrene derivatives were investigated for this aminoazidation (Scheme 2, **2a-m**). Generally, all styrenes underwent this Fe-catalysed ligand-free aminoazidation smoothly, affording corresponding products **2b-j** in good yields, and no remarkable differences in yields were observed between electron-rich methyl-, *tert*-butyl- or acetoxyl-substituted products (**2b-d**) and electron-deficient fluoro-, chloro- or bromo-substituted styrenes (**2e-j**). Moreover, steric hindrance on the benzene ring seemed to share little impact on the yield, since the yields of *para*-substituted **2g** and *ortho*-substituted **2i** are comparable. However, conversion of 4-vinylbiphenyl or 2-vinylnaphthalene with larger conjugated system was poorer, and the yields of their aminoazidated products (**2k-l**) were moderate. In addition, a 1,1-disubstituted styrene **1m** also gave corresponding product **2m** in a slightly dropped yield. When internal alkenes, *β*-methylstyrene (**1n**) or 1,2-dihydronaphthalene (**1o**), were conducted under the

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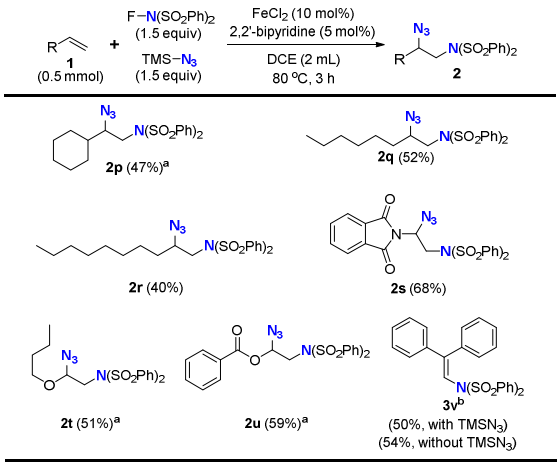
optimized conditions, the yield of aminoazidated product **2n** was well maintained, while the yield of **2o** generated from more rigid substrate **1o** declined remarkably. The diastereoselectivity of **2n** and **2o** could be readily determinated by ^{1}H-NMR spectra of the crude products,[10] and both showed excellent *trans*-selectivity (94:6 and 97:3, respectively), which is comparable to the Cu-catalysed aminoazidation reported by Zhang and Studer.[10]

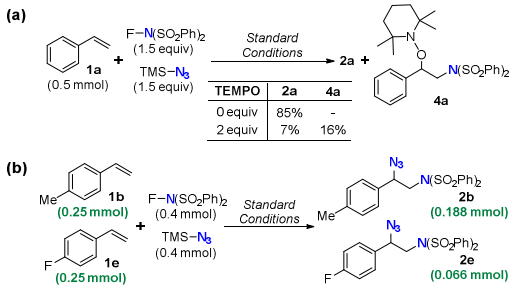


Scheme 3 Fe-catalysed aminoazidation of alkenes beside styrenes. Reaction conditions: alkene **1** (0.5 mmol), NFSI (0.75 mmol), $TMSN_3$ (0.75 mmol), $FeCl_2$ (0.05 mmol) and 2,2'-bipyridine (5 mol%) in DCE (2 mL) under N_2 (1 atm) at 80 °C. Isolated yields. [a]Reaction time was prolonged to 6 h. [b]At 100 °C, without 2,2'-bipyridine.

Subsequently, the substrate scope of this efficient Fe-catalyzed aminoazidation was expanded to other types of alkene beside styrene (Scheme 3, **2p-u**). Assisted by 5 mol% of 2,2'-bipyridine, inactivated alkenes such as 1-octene (**1q**), 1-decene (**1r**) and vinylcyclohexane (**1p**) could provide aminoazidated products **2p-r** in moderate yields at lower temperature, while such aliphatic alkenes did not react under Cu-catalysed conditions.[10] Heteroatom-substituted alkenes including *N*-vinylphthalimide (**1s**), *n*-butyl vinyl ether (**1t**) and vinyl benzoate (**1u**) could also undergo this aminoazidation, affording corresponding products **2s-u** in moderate to good yields. These results suggested that the alkene scope of this Fe-catalysed aminoazidation is broader than previous copper catalysis.[10] Interestingly, the desired aminoazidated product could not be obtained when this reaction was conducted on 1,1-diphenylethylene (**1v**). Instead, an oxidative C-H aminated product **3v** was generated with or without $TMSN_3$, indicating that 1,1-diaryl alkene might undergo hydrogen elimination more easily than azidation after the addition of disulfonimidyl group.

To gain some insights into mechanistic picture preliminarily, some control experiments were then performed.[18] First, the addition of TEMPO (2,2,6,6-tetramethylpiperidine-1-oxyl) severely suppressed this aminoazidation. Meanwhile, an aminooxygenated product **4a** was obtained in 16% yield (Scheme 4a), suggesting that a benzyl radical might probably be formed after the attack of the disulfonimidyl radical generated from NFSI, coinciding with copper-catalysed aminofluorination of styrenes with NFSI studied by Zhang.[8a]

Then a competition experiment was performed to elucidate the electronic preference on the alkene substrate. An equimolecular mixture of styrenes **1b** and **1e** was subjected to insufficient loadings of NFSI and $TMSN_3$, and the ratio between electron-rich aminoazidated products **2b** and electron-deficient product **2e** was approximately 2.85 : 1 (Scheme 4b), indicating that this Fe-catalysed aminoazidation could be categorized as an electrophilic radical addition on alkenes, which is consistent with previously reported copper- or palladium-catalysed aminative bisfunctionalization of alkene with NFSI.



Scheme 4 Control experiments.

Conclusions

To summarize, we have developed an intermolecular vicinal aminoazidation of alkene with NFSI and $TMSN_3$ through iron catalysis. By elevating the reaction temperature, this iron-catalysed aminoazidation on styrene could be achieved in absence of ligand, and the alkene scope of this reaction is broader than previous copper catalysis, suggesting that iron catalysis might probably be a promising alternative for diverse aminative bisfunctionalization of alkenes in addition of copper- or palladium-catalysis. Fully revealing the precise mechanistic picture, as well as further development of other iron-catalysed aminative bisfunctionalization of alkene with NFSI, are undergoing.

Acknowledgements

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18 Please see the Electronic Supplementary Information for the detailed results.

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(R^1 = aryl, alkyl, alkoxyl, acyloxyl, imidyl)

The iron-catalysed intermolecular vicinal aminoazidation of alkene with NFSI is reported, with broader alkene scope comparing to previously reported aminoazidation.

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Electronic Supplementary Information for:

NFSI-participated Intermolecular Aminoazidation of Alkene through Iron Catalysis

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General Remarks

All commercially available compounds were purchased from Sigma-Aldrich, Alfa-Aesar, Acros, J&K Chemicals, Adamas-beta, Accela ChemBio and Aladdin Chemicals. $FeCl_2$ was purchased from Alfa-Aesar (99.99% purity, ultra dry, CAS No. 7758-94-3). $TMSN_3$ was purchased from TCI (>95%, CAS No. 4648-54-8). *N*-Fluorobenzenesulfonimide (NFSI) (98% purity, CAS No. 133745-75-2) and 2,2'-bipyridine (98% purity, CAS No. 366-18-7) were purchased from Accela ChemBio. Unless otherwise noted, materials obtained from commercial suppliers were used without further purification. Products were purified by flash chromatography on silica gel using petroleum ether, ethyl acetate and dichloromethane as the eluents. ^{1}H-NMR spectra were recorded on Bruker AVANCE III-400 spectrometers. Chemical shifts (in ppm) were referenced with TMS in $CDCl_3$ (0 ppm). ^{13}C-NMR spectra were obtained by using the same NMR spectrometers and were calibrated with $CDCl_3$ (δ = 77.00 ppm). High resolution mass spectra were obtained from an Agilent 6520B Q-TOF mass spectrometer with electron spray ionization (ESI) as the ion source.

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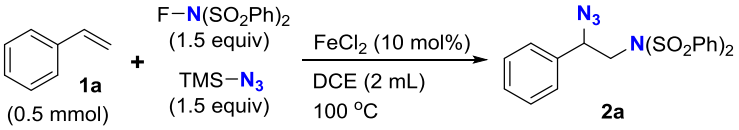
Kinetic Studies



To a reaction tube charged with $FeCl_2$ (6.3 mg, 0.05 mmol) and NFSI (236.5 mg, 0.75 mmol) was added a solution of styrene (**1a**, 57.5 μL, 0.5 mmol), $TMSN_3$ (98.6 μL, 0.75 mmol) in DCE (2 mL) via a syringe under N_2 (1 atm). The reaction mixture was stirred at 100 °C for indicated time in Table S1. After rapidly cooled by ice, the mixture was diluted with ethyl acetate, filtered through a celite pad, and concentrated *in vacuo*. The residue was analyzed with ^{1}H-NMR to determine the yields of **2a** through iron-catalysis, using CH_2Br_2 as an internal standard. Similarly, the yields of **2a** via copper-catalysis, using the same copper catalyst employed by Zhang and Studer,[1] were determined in the same way. Then the yields of **2a** via iron- or copper-catalysis in different times were summarized in Table S1 and Figure S1, accordingly.

Table S1. Detailed Results of Kinetic Studies.

Time (min)	20	40	60	80	100	120	180	300	420
$FeCl_2$	39.8	53.0	64.1	72.7	78.0	82.5	89.8	87.2	85.6
CuCl	9.7	13.7	15.4	16.3	15.8	14.6	13.6	11.8	9.6

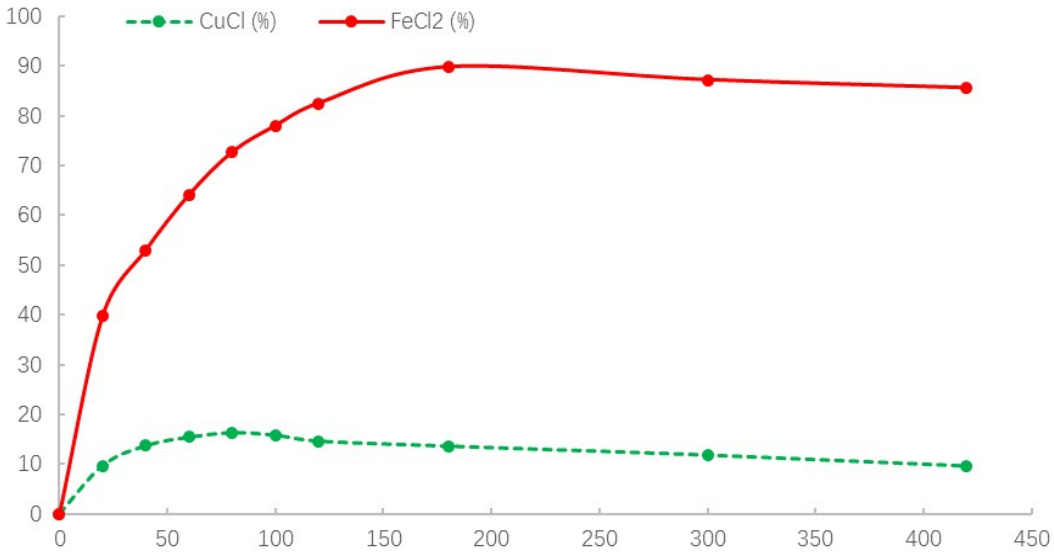


Figure S1. Detailed Results of Kinetic Studies.

From the data in Table S1 and Figure S1, this aminoazidation catalyzed by $FeCl_2$ is remarkably faster than catalyzed by CuCl, which was previously employed by Zhang and Studer,[1] while the yields of **2a** remained very low, no matter how much the reaction time was, when the reaction was catalyzed by CuCl. Theses results demonstrated that the iron catalyst, which could achieve this reaction rapidly without any ligand or additive and provide the product in excellent yield after 3 hours, is a competitive alternative to previously used copper catalyst, which could not effectively realize this reaction in absence of certain ligands.[1]

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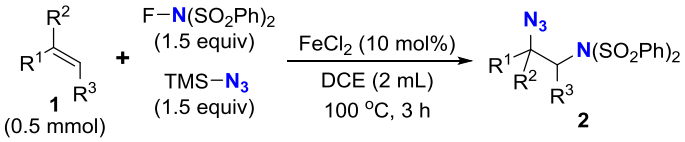
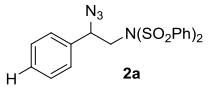
Experimental Procedure and Characterization Data



Typical Procedure: To a reaction tube charged with $FeCl_2$ (6.3 mg, 0.05 mmol) and NFSI (236.5 mg, 0.75 mmol) was added a solution of alkene (**1a-u**, 0.5 mmol), $TMSN_3$ (98.6 μL, 0.75 mmol) in DCE (2 mL) via a syringe under N_2 (1 atm). The reaction mixture was stirred at 100 °C for 3 hours. After rapidly cooling by ice, the mixture was diluted with ethyl acetate, filtered through a celite pad, and concentrated *in vacuo* to give dark residue, which was then purified by flash chromatography using petroleum ether and ethyl acetate as the eluent on silical gel to afford aminoazidated product **2a-u**.

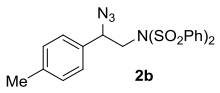
***N*-(2-Azido-2-phenylethyl)-*N*-(phenylsulfonyl)benzenesulfonamide[1] (2a):**



2a

The reaction of 0.5 mmol of styrene (**1a**) with NFSI and $TMSN_3$ afforded 187.2 mg of **2a** (85%) as colorless oil, after flash chromatography on silica gel using petroleum ether and ethyl acetate (15:1, *v*/*v*) as the eluent. **^{1}H NMR ($CDCl_3$, 400 MHz)**: δ = 8.07-8.05 (m, 4H), 7.67-7.63 (m, 2H), 7.56-7.52 (m, 4H), 7.40-7.35 (m, 5H), 5.01 (dd, *J* = 9.6 Hz, 4.2 Hz, 1H), 4.06 (dd, *J* = 15.6 Hz, 9.6 Hz, 1H), 3.72 (dd, *J* = 15.6 Hz, 4.2 Hz, 1H) ppm; **^{13}C NMR ($CDCl_3$, 100 MHz)**: δ = 139.22, 136.44, 134.02, 129.12, 129.02, 128.98, 128.57, 127.19, 65.57, 53.18 ppm. **HRMS *m/z* (ESI)** calcd for $[C_{20}H_{18}N_4O_4S_2+Na]^+$ 465.0662, found 465.0669.

***N*-(2-Azido-2-(p-tolyl)ethyl)-*N*-(phenylsulfonyl)benzenesulfonamide[1] (2b):**



2b

The reaction of 0.5 mmol of *p*-methylstyrene (**1b**) with NFSI and $TMSN_3$ afforded 178.1 mg of **2b** (78%) as white solid (m.p. 133.4-134.8 °C), after flash chromatography on silica gel using petroleum ether and ethyl acetate (15:1, *v*/*v*) as the eluent. **^{1}H NMR ($CDCl_3$, 400 MHz)**: δ = 8.06-8.04 (m, 4H), 7.66-7.62 (m, 2H), 7.55-7.52 (m, 4H), 7.25 (d, *J* = 8.1 Hz, 2H), 7.20 (d, *J* = 8.1 Hz, 2H), 4.98 (dd, *J* = 9.6 Hz, 4.3 Hz, 1H), 4.05 (dd, *J* = 15.6 Hz, 9.6 Hz, 1H), 3.70 (dd, *J* = 15.6 Hz, 4.3 Hz, 1H), 2.36 (s, 3H) ppm; **^{13}C NMR ($CDCl_3$, 100 MHz)**: δ = 139.20, 138.95, 133.98, 133.32, 129.75, 128.94, 128.56, 127.15, 65.32, 53.09, 21.16 ppm. **HRMS *m/z* (ESI)** calcd for $[C_{21}H_{20}N_4O_4S_2+Na]^+$ 479.0818,

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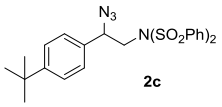
found 479.0827.

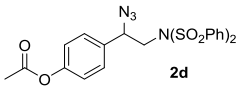
***N*-(2-Azido-2-(4-(*tert*-butyl)phenyl)ethyl)-*N*-(phenylsulfonyl)benzenesulfonamide[1] (2c):**



2c

The reaction of 0.5 mmol of *p*-*tert*-butylstyrene (**1c**) with NFSI and $TMSN_3$ afforded 199.4 mg of **2c** (80%) as light yellow oil, after flash chromatography on silica gel using petroleum ether and ethyl acetate (15:1, *v*/*v*) as the eluent. **^{1}H NMR ($CDCl_3$, 400 MHz)**: δ = 8.08-8.06 (m, 4H), 7.65-7.62 (m, 2H), 7.55-7.51 (m, 4H), 7.42 (d, *J* = 8.3 Hz, 2H), 7.29 (d, *J* = 8.3 Hz, 2H), 4.98 (dd, *J* = 9.8 Hz, 3.9 Hz, 1H), 4.08 (dd, *J* = 15.6 Hz, 9.8 Hz, 1H), 3.71 (dd, *J* = 15.6 Hz, 3.9 Hz, 1H), 1.32 (s, 9H) ppm; **^{13}C NMR ($CDCl_3$, 100 MHz)**: δ = 152.08. 139.25. 133.97, 133.36, 128.93, 128.54, 126.84, 125.96, 65.30, 53.11, 34.61, 31.21 ppm. **HRMS *m/z* (ESI)** calcd for $[C_{24}H_{26}N_4O_4S_2+Na]^+$ 521.1293, found 521.1290; calcd for $[C_{24}H_{26}N_4O_4S_2+K]^+$ 537.1027, found 537.1030.

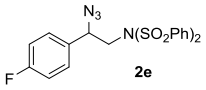
4-(1-Azido-2-(*N*-(phenylsulfonyl)phenylsulfonamido)ethyl)phenyl acetate[1] (2d):



2d

The reaction of 0.5 mmol of 4-vinylphenyl acetate (**1d**) with NFSI and $TMSN_3$ afforded 184.3 mg of **2d** (74%) as colorless oil, after flash chromatography on silica gel using petroleum ether and ethyl acetate (12:1 to 8:1, *v*/*v*) as the eluent. **^{1}H NMR ($CDCl_3$, 400 MHz)**: δ = 8.06-8.04 (m, 4H), 7.66-7.62 (m, 2H), 7.56-7.52 (m, 4H), 7.37 (d, *J* = 8.6 Hz, 2H), 7.12 (d, *J* = 8.6 Hz, 2H), 5.02 (dd, *J* = 9.5 Hz, 4.2 Hz, 1H), 4.03 (dd, *J* = 15.6 Hz, 9.5 Hz, 1H), 3.72 (dd, *J* = 15.6 Hz, 4.2 Hz, 1H), 2.29 (s, 3H) ppm; **^{13}C NMR ($CDCl_3$, 100 MHz)**: δ = 169.12, 150.97, 139.02, 134.00, 133.91, 128.95, 128.47, 128.25, 122.26, 64.99, 53.02, 20.97 ppm. **HRMS *m/z* (ESI)** calcd for $[C_{22}H_{20}N_4O_6S_2+Na]^+$ 523.0716, found 523.0720.

***N*-(2-Azido-2-(4-fluorophenyl)ethyl)-*N*-(phenylsulfonyl)benzenesulfonamide[1] (2e):**



2e

The reaction of 0.5 mmol of *p*-fluorostyrene (**1e**) with NFSI and $TMSN_3$ afforded 185.3 mg of **2e** (81%) as white solid (m.p. 104.7-105.9 ºC), after flash chromatography on silica gel using petroleum ether and ethyl acetate (15:1, *v*/*v*) as the eluent. **^{1}H NMR ($CDCl_3$, 400 MHz)**: δ = 8.06-8.03 (m, 4H), 7.67-7.63 (m, 2H), 7.56-7.52 (m, 4H), 7.34-7.31 (m, 2H), 7.09-7.04 (m, 2H), 5.01 (dd, *J* = 9.3 Hz, 4.5 Hz, 1H), 4.02 (dd, *J* = 15.6 Hz, 9.3 Hz, 1H), 3.71 (dd, *J* = 15.6 Hz, 4.5 Hz, 1H) ppm; **^{13}C NMR ($CDCl_3$, 100 MHz)**: δ = 162.91 (d, *J* = 247.0 Hz), 139.12, 134.06, 132.27 (d, *J* = 3.0 Hz), 129.06, 128.99, 128.51, 116.09 (d, *J* = 21.4 Hz), 64.86, 53.16 ppm. **HRMS *m/z* (ESI)** calcd for $[C_{20}H_{17}FN_4O_4S_2+Na]^+$

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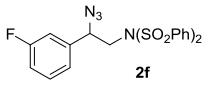
483.0567, found 483.0569.

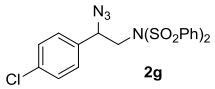
***N*-(2-Azido-2-(3-fluorophenyl)ethyl)-*N*-(phenylsulfonyl)benzenesulfonamide[1] (2f):**



2f

The reaction of 0.5 mmol of *m*-fluorostyrene (**1e**) with NFSI and $TMSN_3$ afforded 190.2 mg of **2f** (83%) as colorless oil, after flash chromatography on silica gel using petroleum ether and ethyl acetate (15:1 to 12:1, *v*/*v*) as the eluent. **^{1}H NMR ($CDCl_3$, 400 MHz)**: δ = 8.07-8.05 (m, 4H), 7.66-7.63 (m, 2H), 7.56-7.52 (m, 4H), 7.38-7.32 (m, 1H), 7.14-7.13 (m, 1H), 7.07-7.02 (m, 2H), 5.01 (dd, *J* = 9.5 Hz, 4.2 Hz, 1H), 4.04 (dd, *J* = 15.6 Hz, 9.5 Hz, 1H), 3.71 (dd, *J* = 15.6 Hz, 4.2 Hz, 1H) ppm; **^{13}C NMR ($CDCl_3$, 100 MHz)**: δ = 162.92 (d, *J* = 246.5 Hz), 139.08, 138.95 (d, *J* = 7.1 Hz), 134.07, 130.74 (d, *J* = 8.1 Hz), 129.00, 128.48, 122.74 (d, *J* = 2.9 Hz), 115.95 (d, *J* = 21.0 Hz), 114.13 (d, *J* = 22.1 Hz), 65.02 (d, *J* = 1.2 Hz), 53.16 ppm. **HRMS *m/z* (ESI)** calcd for $[C_{20}H_{17}FN_4O_4S_2+Na]^+$ 483.0567, found 483.0570.

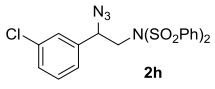
***N*-(2-Azido-2-(4-chlorophenyl)ethyl)-*N*-(phenylsulfonyl)benzenesulfonamide[1] (2g):**



2g

The reaction of 0.5 mmol of *p*-chlorostyrene (**1g**) with NFSI and $TMSN_3$ afforded 188.0 mg of **2g** (79%) as white solid (m.p. 144.8-146.0 °C), after flash chromatography on silica gel using petroleum ether and ethyl acetate (15:1, *v*/*v*) as the eluent. **^{1}H NMR ($CDCl_3$, 400 MHz)**: δ = 8.04-8.01 (m, 4H), 7.66-7.62 (m, 2H), 7.55-7.51 (m, 4H), 7.34 (d, *J* = 8.5 Hz, 2H), 7.28 (d, *J* = 8.5 Hz, 2H), 5.01 (dd, *J* = 9.1 Hz, 4.8 Hz, 1H), 3.99 (dd, *J* = 15.6 Hz, 9.1 Hz, 1H), 3.73 (dd, *J* = 15.6 Hz, 4.8 Hz, 1H) ppm; **^{13}C NMR ($CDCl_3$, 100 MHz)**: δ = 138.99, 134.89, 134.86, 134.04, 129.26, 128.97, 128.58, 128.45, 64.83, 52.98 ppm. **HRMS *m/z* (ESI)** calcd for $[C_{20}H_{17}ClN_4O_4S_2+Na]^+$ 499.0277, found 499.0278; calcd for $[C_{20}H_{17}ClN_4O_4S_2+K]^+$ 515.0011, found 515.0017.

***N*-(2-Azido-2-(3-chlorophenyl)ethyl)-*N*-(phenylsulfonyl)benzenesulfonamide (2h):**



2h

The reaction of 0.5 mmol of *m*-chlorostyrene (**1h**) with NFSI and $TMSN_3$ afforded 177.9 mg of **2h** (75%) as colorless oil, after flash chromatography on silica gel using petroleum ether and ethyl acetate (15:1, *v*/*v*) as the eluent. **^{1}H NMR ($CDCl_3$, 400 MHz)**: δ = 8.07-8.05 (m, 4H), 7.67-7.63 (m, 2H), 7.56-7.52 (m, 4H), 7.33-7.31 (m, 3H), 7.25-7.23 (m, 1H), 4.99 (dd, *J* = 9.4 Hz, 4.3 Hz, 1H), 4.03 (dd, *J* = 15.6 Hz, 9.4 Hz, 1H), 3.71 (dd, *J* = 15.6 Hz, 4.3 Hz, 1H) ppm; **^{13}C NMR ($CDCl_3$, 100 MHz)**: δ = 139.06, 138.51, 134.96, 134.08, 130.39, 129.16, 129.00, 128.48, 127.29, 125.25, 64.99, 53.14 ppm. **HRMS**

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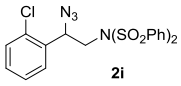
***m/z* (ESI)** calcd for $[C_{20}H_{17}ClN_4O_4S_2+Na]^+$ 499.0272, found 499.0278.

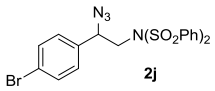
***N*-(2-Azido-2-(2-chlorophenyl)ethyl)-*N*-(phenylsulfonyl)benzenesulfonamide (2i):**



2i

The reaction of 0.5 mmol of *o*-chlorostyrene (**1i**) with NFSI and $TMSN_3$ afforded 189.4 mg of **2i** (80%) as white solid (m.p. 133.1-134.5 °C), after flash chromatography on silica gel using petroleum ether and ethyl acetate (15:1, *v*/*v*) as the eluent. **^{1}H NMR ($CDCl_3$, 400 MHz)**: δ = 8.15-8.14 (m, 4H), 7.69-7.65 (m, 2H), 7.59-7.55 (m, 4H), 7.52-7.49 (m, 1H), 7.40-7.27 (m, 3H), 5.51 (dd, *J* = 10.5 Hz, 3.8 Hz, 1H), 4.05 (dd, *J* = 15.6 Hz, 10.5 Hz, 1H), 3.69 (dd, *J* = 15.6 Hz, 3.8 Hz, 1H) ppm; **^{13}C NMR ($CDCl_3$, 100 MHz)**: δ = 139.17, 134.54, 134.06, 132.92, 130.03, 129.96, 129.02, 128.65, 128.33, 127.70, 61.82, 51.57 ppm. **HRMS *m/z* (ESI)** calcd for $[C_{20}H_{17}ClN_4O_4S_2+Na]^+$ 499.0272, found 499.0276.

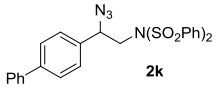
***N*-(2-Azido-2-(4-bromophenyl)ethyl)-*N*-(phenylsulfonyl)benzenesulfonamide[1] (2j):**



2j

The reaction of 0.5 mmol of *p*-bromostyrene (**1j**) with NFSI and $TMSN_3$ afforded 214.5 mg of **2j** (83%) as white solid (m.p. 122.1-123.7 °C), after flash chromatography on silica gel using petroleum ether and ethyl acetate (15:1, *v*/*v*) as the eluent. **^{1}H NMR ($CDCl_3$, 400 MHz)**: δ = 8.03-8.01 (m, 4H), 7.67-7.63 (m, 2H), 7.56-7.49 (m, 6H), 7.22 (d, *J* = 8.4 Hz, 2H), 5.00 (dd, *J* = 9.0 Hz, 4.8 Hz, 1H), 3.98 (dd, *J* = 15.6 Hz, 9.0 Hz, 1H), 3.73 (dd, *J* = 15.6 Hz, 4.8 Hz, 1H) ppm; **^{13}C NMR ($CDCl_3$, 100 MHz)**: δ = 139.01, 135.46, 134.08, 132.27, 129.01, 128.92, 128.51, 123.10, 64.95, 52.93 ppm. **HRMS *m/z* (ESI)** calcd for $[C_{20}H_{17}BrN_4O_4S_2+Na]^+$ 542.9767, found 542.9776.

***N*-(2-([1,1'-Biphenyl]-4-yl)-2-azidoethyl)-*N*-(phenylsulfonyl)benzenesulfonamide[1] (2k):**



2k

The reaction of 0.5 mmol of 4-vinyl-1,1'-biphenyl (**1k**) with NFSI and $TMSN_3$ afforded 153.9 mg of **2k** (60%) as light yellow solid (m.p. 132.0-133.7 °C), after flash chromatography on silica gel using petroleum ether and ethyl acetate (15:1, *v*/*v*) as the eluent. **^{1}H NMR ($CDCl_3$, 400 MHz)**: δ = 8.07-8.05 (m, 4H), 7.65-7.58 (m, 6H), 7.54-7.50 (m, 4H), 7.47-7.35 (m, 5H), 5.07 (dd, *J* = 9.4 Hz, 4.5 Hz, 1H), 4.08 (dd, *J* = 15.6 Hz, 9.4 Hz, 1H), 3.79 (dd, *J* = 15.6 Hz, 4.5 Hz, 1H) ppm; **^{13}C NMR ($CDCl_3$, 100 MHz)**: δ = 141.95, 140.15, 139.17, 135.31, 134.01, 128.97, 128.87, 128.56, 127.75, 127.71, 127.67, 127.03, 65.32, 53.06 ppm. **HRMS *m/z* (ESI)** calcd for $[C_{26}H_{22}N_4O_4S_2+Na]^+$ 541.0975, found 541.0977.

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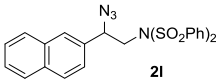
DOI: 10.1039/C8OB00699G

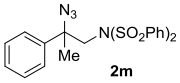
***N*-(2-Azido-2-(naphthalen-2-yl)ethyl)-*N*-(phenylsulfonyl)benzenesulfonamide[1] (2l):**



2l

The reaction of 0.5 mmol of 2-vinylnaphthalene (**1l**) with NFSI and $TMSN_3$ afforded 105.1 mg of **2l** (43%) as light yellow solid (m.p. 125.2-126.8 °C), after flash chromatography on silica gel using petroleum ether and ethyl acetate (15:1, *v*/*v*) as the eluent. **^{1}H NMR ($CDCl_3$, 400 MHz)**: δ = 8.01-7.99 (m, 4H), 7.90-7.79 (m, 4H), 7.60-7.42 (m, 9H), 5.21 (dd, *J* = 8.8 Hz, 5.0 Hz, 1H), 4.10 (dd, *J* = 15.6 Hz, 8.8 Hz, 1H), 3.88 (dd, *J* = 15.6 Hz, 5.0 Hz, 1H) ppm; **^{13}C NMR ($CDCl_3$, 100 MHz)**: δ = 139.03, 133.96, 133.65, 133.42, 133.11, 129.18, 128.88, 128.51, 128.09, 127.74, 127.12, 126.73, 126.66, 124.22, 65.73, 52.94 ppm. **HRMS *m/z* (ESI)** calcd for $[C_{24}H_{20}N_4O_4S_2+Na]^+$ 515.0818, found 515.0825; calcd for $[C_{24}H_{20}N_4O_4S_2+K]^+$ 531.0563, found 531.0566.

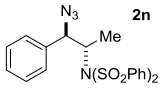
***N*-(2-Azido-2-phenylpropyl)-N-(phenylsulfonyl)benzenesulfonamide[1] (2m):**



2m

The reaction of 0.5 mmol of prop-1-en-2-ylbenzene (**1m**) with NFSI and $TMSN_3$ afforded 144.8 mg of **2m** (64%) as colorless oil, after flash chromatography on silica gel using petroleum ether and ethyl acetate (15:1, *v*/*v*) as the eluent. **^{1}H NMR ($CDCl_3$, 400 MHz)**: δ = 8.11-8.09 (m, 4H), 7.65-7.61 (m, 2H), 7.57-7.53 (m, 4H), 7.44-7.30 (m, 5H), 4.17 (d, *J* = 15.8 Hz, 1H), 3.99 (d, *J* = 15.8 Hz, 1H), 1.67 (s, 3H) ppm; **^{13}C NMR ($CDCl_3$, 100 MHz)**: δ = 141.43, 140.58, 133.73, 128.90, 128.85, 128.43, 128.27, 125.82, 66.92, 58.85, 21.54 ppm. **HRMS *m/z* (ESI)** calcd for $[C_{21}H_{20}N_4O_4S_2+Na]^+$ 479.0818, found 479.0823.

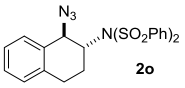
***N*-(1-Azido-1-phenylpropan-2-yl)-*N*-(phenylsulfonyl)benzenesulfonamide[1] (2n):**



2n

The reaction of 0.5 mmol of *(E)*-prop-1-en-1-ylbenzene (**1n**) with NFSI and $TMSN_3$ afforded 173.6 mg of **2n** (76%, dr > 94:6) as colorless oil, after flash chromatography on silica gel using petroleum ether and ethyl acetate (15:1, *v*/*v*) as the eluent. **^{1}H NMR ($CDCl_3$, 400 MHz)**: δ = 7.64-7.61 (m, 4H), 7.57-7.53 (m, 2H), 7.40-7.24 (m, 9H), 5.21 (d, *J* = 9.7 Hz, 1H), 4.37 (dq, *J* = 9.7 Hz, 6.7 Hz, 1H), 1.51 (d, *J* = 6.8, 3H) ppm; **^{13}C NMR ($CDCl_3$, 100 MHz)**: δ = 135.87, 133.69, 129.13, 128.81, 128.73, 128.67, 128.41, 127.56, 69.71, 61.48, 17.94 ppm. **HRMS *m/z* (ESI)** calcd for $[C_{21}H_{20}N_4O_4S_2+Na]^+$ 479.0818, found 479.0818; calcd for $[C_{21}H_{20}N_4O_4S_2+K]^+$ 495.0558, found 495.0559.

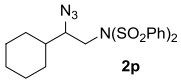
***N*-(1-Azido-1,2,3,4-tetrahydronaphthalen-2-yl)-*N*-(phenylsulfonyl)benzenesulfonamide[1] (2o):**



2o

The reaction of 0.5 mmol of 1,2-dihydronaphthalene (**1o**) with NFSI and $TMSN_3$ afforded 85.6 mg of **2o** (37%, dr > 97:3) as light yellow oil, after flash chromatography on silica gel using petroleum ether and ethyl acetate (15:1, *v*/*v*) as the eluent. **^{1}H NMR ($CDCl_3$, 400 MHz)**: δ = 8.14-8.12 (m, 4H), 7.70-7.66 (m, 2H), 7.60-7.56 (m, 4H), 7.41 (d, *J* = 7.5 Hz, 1H), 7.27-7.20 (m, 2H), 7.08 (d, *J* = 7.2 Hz, 1H), 5.38 (d, *J* = 10.0 Hz, 1H), 4.36-4.29 (m, 1H), 2.82-2.78 (m, 2H), 2.63-2.52 (m, 1H), 1.89-1.83 (m, 1H) ppm; **^{13}C NMR ($CDCl_3$, 100 MHz)**: δ = 135.66, 134.08, 134.00, 129.02, 128.69, 128.56, 128.04, 127.94, 126.83, 64.97, 62.78, 29.99, 28.44 ppm. **HRMS *m/z* (ESI)** calcd for $[C_{22}H_{20}N_4O_4S_2+Na]^+$ 491.0818, found 491.0826.

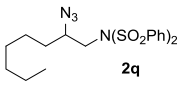
***N*-(2-Azido-2-cyclohexylethyl)-*N*-(phenylsulfonyl)benzenesulfonamide (2p):**



2p

In presence of 2,2'-bipyridine (3.9 mg, 0.025 mmol), the reaction of 0.5 mmol of vinyl cyclohexane (**1p**) with NFSI and $TMSN_3$ at 80°C for 6 hours afforded 104.2 mg of **2p** (47%) as colorless oil, after flash chromatography on silica gel using petroleum ether and ethyl acetate (15:1, *v*/*v*) as the eluent. **^{1}H NMR ($CDCl_3$, 400 MHz)**: δ = 8.12-8.09 (m, 4H), 7.68-7.65 (m, 2H), 7.59-7.55 (m, 4H), 3.89 (dd, *J* = 15.8 Hz, 10.5 Hz, 1H), 3.65-3.61 (m, 2H), 1.77-1.75 (m, 3H), 1.67-1.64 (m, 2H), 1.51-1.43 (m, 1H), 1.26-1.12 (m, 5H) ppm; **^{13}C NMR ($CDCl_3$, 100 MHz)**: δ = 139.33, 133.97, 129.04, 128.40, 67.49, 50.80, 41.10, 29.80, 28.03, 26.00, 25.90, 25.73 ppm. **HRMS *m/z* (ESI)** calcd for $[C_{20}H_{24}N_4O_4S_2+K]^+$ 487.0871, found 487.0880.

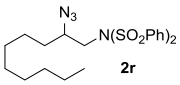
***N*-(2-Azidooctyl)-*N*-(phenylsulfonyl)benzenesulfonamide (2q):**



2q

In presence of 2,2'-bipyridine (3.9 mg, 0.025 mmol), the reaction of 0.5 mmol of 1-octene (**1q**) with NFSI and $TMSN_3$ at 80°C afforded 116.0 mg of **2q** (52%) as colorless oil, after flash chromatography on silica gel using petroleum ether and ethyl acetate (15:1, *v*/*v*) as the eluent. **^{1}H NMR ($CDCl_3$, 400 MHz)**: δ = 8.10-8.08 (m, 4H), 7.68-7.64 (m, 2H), 7.58-7.55 (m, 4H), 3.86 (dd, *J* = 15.1 Hz, 9.2 Hz, 1H), 3.75-3.69 (m, 1H), 3.57 (dd, *J* = 15.1 Hz, 3.9 Hz, 1H), 1.50-1.42 (m, 2H), 1.32-1.23 (m, 8H), 0.89 (t, *J* = 6.9 Hz, 3H) ppm; **^{13}C NMR ($CDCl_3$, 100 MHz)**: δ = 139.40, 134.01, 129.06, 128.41, 62.00, 52.15, 32.29, 31.52, 28.86, 25.80, 22.47, 13.99 ppm. **HRMS *m/z* (ESI)** calcd for $[C_{20}H_{26}N_4O_4S_2+Na]^+$ 473.1288, found 473.1298.

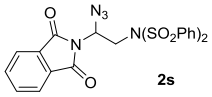
***N*-(2-Azidodecyl)-*N*-(phenylsulfonyl)benzenesulfonamide (2r):**



2r

In presence of 2,2'-bipyridine (3.9 mg, 0.025 mmol), the reaction of 0.5 mmol of 1-decene (**1r**) with NFSI and $TMSN_3$ at 80°C afforded 94.3 mg of **2r** (40%) as colorless oil, after flash chromatography on silica gel using petroleum ether and ethyl acetate (15:1, *v*/*v*) as the eluent. **^{1}H NMR ($CDCl_3$, 400 MHz)**: δ = 8.10-8.08 (m, 4H), 7.68-7.64 (m, 2H), 7.58-7.54 (m, 4H), 3.86 (dd, *J* = 15.1 Hz, 9.3 Hz, 1H), 3.75-3.69 (m, 1H), 3.56 (dd, *J* = 15.1 Hz, 3.9 Hz, 1H), 1.50-1.43 (m, 2H), 1.32-1.26 (m, 12H), 0.89 (t, *J* = 6.9 Hz, 3H) ppm; **^{13}C NMR ($CDCl_3$, 100 MHz)**: δ = 139.31, 133.99, 129.02, 128.35, 61.96, 52.09, 32.23, 31.71, 29.25, 29.15, 29.07, 25.79, 22.56, 14.03 ppm. **HRMS *m/z* (ESI)** calcd for $[C_{22}H_{30}N_4O_4S_2+Na]^+$ 501.1606, found 501.1607; calcd for $[C_{22}H_{30}N_4O_4S_2+Na]^+$ 501.1601, found 501.1607.

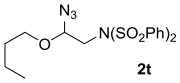
***N*-(2-Azido-2-(1,3-dioxoisoindolin-2-yl)ethyl)-*N*-(phenylsulfonyl)benzenesulfonamide (2s):**



2s

In presence of 2,2'-bipyridine (3.9 mg, 0.025 mmol), the reaction of 0.5 mmol of *N*-vinylphthalimide (**1s**) with NFSI and $TMSN_3$ at 80°C afforded 172.5 mg of **2s** (68%) as white solid (m.p. 177.3-178.6 °C), after flash chromatography on silica gel using petroleum ether and ethyl acetate (15:1 to 3:1, *v*/*v*) as the eluent. **^{1}H NMR ($CDCl_3$, 400 MHz)**: δ = 8.05-8.03 (m, 4H), 7.90-7.88 (m, 2H), 7.77-7.75 (m, 2H), 7.67-7.63 (m, 2H), 7.57-7.53 (m, 4H), 6.04 (dd, *J* = 7.3 Hz, 4.9 Hz, 1H), 4.42-4.30 (m, 2H) ppm; **^{13}C NMR ($CDCl_3$, 100 MHz)**: δ = 166.97, 138.34, 134.60, 134.29, 131.35, 129.14, 128.62, 123.91, 65.30, 47.32 ppm. **HRMS *m/z* (ESI)** calcd for $[C_{22}H_{17}N_5O_6S_2+K]^+$ 550.0252, found 550.0258.

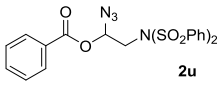
***N*-(2-Azido-2-butoxyethyl)-*N*-(phenylsulfonyl)benzenesulfonamide (2t):**



2t

In presence of 2,2'-bipyridine (3.9 mg, 0.025 mmol), the reaction of 0.5 mmol of 1-(vinyloxy)butane (**1t**) with NFSI and $TMSN_3$ at 80°C for 6 hours afforded 111.2 mg of **2t** (51%) as colorless oil, after flash chromatography on silica gel using petroleum ether and ethyl acetate (15:1, *v*/*v*) as the eluent. **^{1}H NMR ($CDCl_3$, 400 MHz)**: δ = 8.11-8.09 (m, 4H), 7.69-7.65 (m, 2H), 7.59-7.55 (m, 4H), 4.81 (t, *J* = 6.0 Hz, 1H), 3.93 (dd, *J* = 15.7 Hz, 6.1 Hz, 1H), 3.80 (dd, *J* = 15.7 Hz, 5.8 Hz, 1H), 3.67 (dt, *J* = 9.5 Hz, 6.9 Hz, 1H), 3.40 (dt, *J* = 9.5 Hz, 6.6 Hz, 1H), 1.50-1.43 (m, 2H), 1.34-1.26 (m, 2H), 0.90 (t, *J* = 7.3 Hz, 3H) ppm; **^{13}C NMR ($CDCl_3$, 100 MHz)**: δ = 139.17, 134.03, 128.97, 128.55, 90.33, 69.82, 50.54, 31.19, 19.00, 13.73 ppm. **HRMS *m/z* (ESI)** calcd for $[C_{18}H_{22}N_4O_5S_2+Na]^+$ 461.0924, found 461.0930.

1-Azido-2-(*N*-(phenylsulfonyl)phenylsulfonamido)ethyl benzoate (2u):



2u

In presence of 2,2'-bipyridine (3.9 mg, 0.025 mmol), the reaction of 0.5 mmol of vinyl benzoate (**1u**) with NFSI and $TMSN_3$ at 80°C for 6 hours afforded 142.3 mg of **2u** (59%) as colorless oil, after flash chromatography on silica gel using petroleum ether and ethyl acetate (15:1, *v*/*v*) as the eluent. **^{1}H NMR ($CDCl_3$, 400 MHz)**: δ = 8.06-8.02 (m, 6H), 7.62-7.58 (m, 3H), 7.50-7.43 (m, 6H), 6.42 (t, *J* = 6.3 Hz, 1H), 3.80 (dq, *J* = 15.5 Hz, 6.3 Hz, 2H) ppm; **^{13}C NMR ($CDCl_3$, 100 MHz)**: δ = 165.66, 139.11, 134.14, 133.87, 130.09, 129.13, 128.52, 128.35, 128.24, 83.39, 49.56 ppm. **HRMS *m/z* (ESI)** calcd for $[C_{21}H_{18}N_4O_6S_2+K]^+$ 525.0299, found 525.0309.

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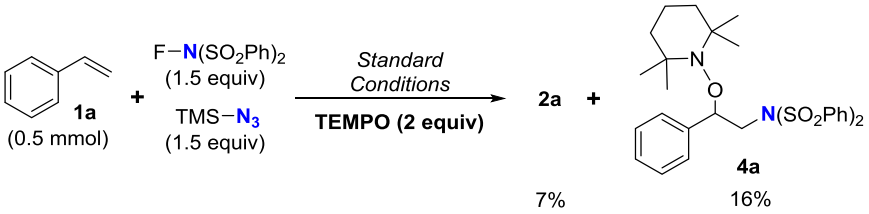
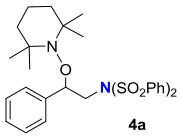
Control Experiments

1) TEMPO-suppressed Control Experiment



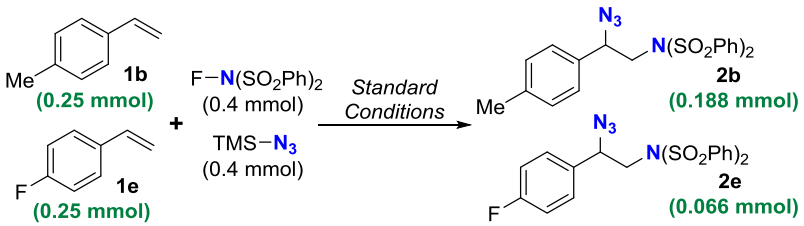
Typical Procedure: To a reaction tube charged with $FeCl_2$ (6.3 mg, 0.05 mmol), NFSI (236.5 mg, 0.75 mmol) and TEMPO (156.3 mg, 1 mmol) was added a solution of styrene (**1a**, 57.5 μL, 0.5 mmol), $TMSN_3$ (98.6 μL, 0.75 mmol) in DCE (2 mL) via a syringe under N_2 (1 atm). The reaction mixture was stirred at 100 °C for 3 hours. After rapidly cooling by ice, the mixture was diluted with ethyl acetate, filtered through a celite pad, and concentrated *in vacuo* to give dark residue, which was then purified by flash chromatography using petroleum ether and ethyl acetate as the eluent on silical gel to afford aminoazidated product **2a** (15.1 mg, 7%) and TEMPO-captured product **4a** (43.4 mg, 16%).

***N*-(2-Phenyl-2-((2,2,6,6-tetramethylpiperidin-1-yl)oxy)ethyl)-*N*-(phenylsulfonyl)benzenesulfonamide[2] (4a):**



The reaction of 0.5 mmol of styrene (1a) under standard conditions, adding 1 mmol of TEMPO afforded 43.4 mg of **4a** (16%) as white solid (m.p. 132.3-133.7 °C), after flash chromatography on silica gel using petroleum ether and ethyl acetate (20:1 to 15:1, *v*/*v*) as the eluent. **^{1}H NMR ($CDCl_3$, 400 MHz)**: δ = 7.60-7.56 (m, 6H), 7.43-7.39 (m, 4H), 7.36-7.30 (m, 5H), 5.23 (dd, *J* = 11.0 Hz, 4.7 Hz, 1H), 4.39 (dd, *J* = 14.9 Hz, 11.0 Hz, 1H), 4.03 (dd, *J* = 14.9 Hz, 4.7 Hz, 1H), 1.51-1.39 (m, 5H), 1.27-1.24 (m, 4H), 1.11-0.97 (m, 6H), 0.88-0.70 (m, 3H) ppm; **^{13}C NMR ($CDCl_3$, 100 MHz)**: δ = 139.90, 138.49, 133.58, 129.28, 128.81, 128.62, 128.12, 127.83, 84.10, 59.95, 50.09, 40.50, 34.90, 34.23, 20.22, 17.11 ppm. **HRMS *m/z* (ESI)** calcd for $[C_{29}H_{36}N_2O_5S_2+H]^+$ 557.2138, found 557.2144.

2) Competition Experiment between Electron-rich/-deficient Styrenes



To a reaction tube charged with $FeCl_2$ (6.3 mg, 0.05 mmol) and NFSI (126.1 mg, 0.4 mmol) was added a solution of *p*-methylstyrene (**1b**, 32.9 μL, 0.25 mmol), *p*-fluorostyrene (**1e**, 29.8 μL, 0.25 mmol), $TMSN_3$ (52.6 μL, 0.4 mmol) in DCE (2 mL) via a syringe under N_2 (1 atm). The reaction mixture was stirred at 100 °C for 3 hours. After rapidly cooling by ice, the mixture was diluted with ethyl acetate, filtered through a celite pad, and concentrated *in vacuo* to give dark residue, which was then purified by flash chromatography using petroleum ether and ethyl acetate as the eluent (15:1, *v*/*v*) on silical gel to afford 116.0 mg of the combined methyl- and fluoro-aminoazidated product **2b** and **2e**. The average ratio of **2b**/**2e** was determined by ^{1}H-NMR as 1 : 0.35, as shown in Figure S2. Herein, this competition reaction afforded 0.188 mmol of **2b** and 0.066 mmol of **2e**.

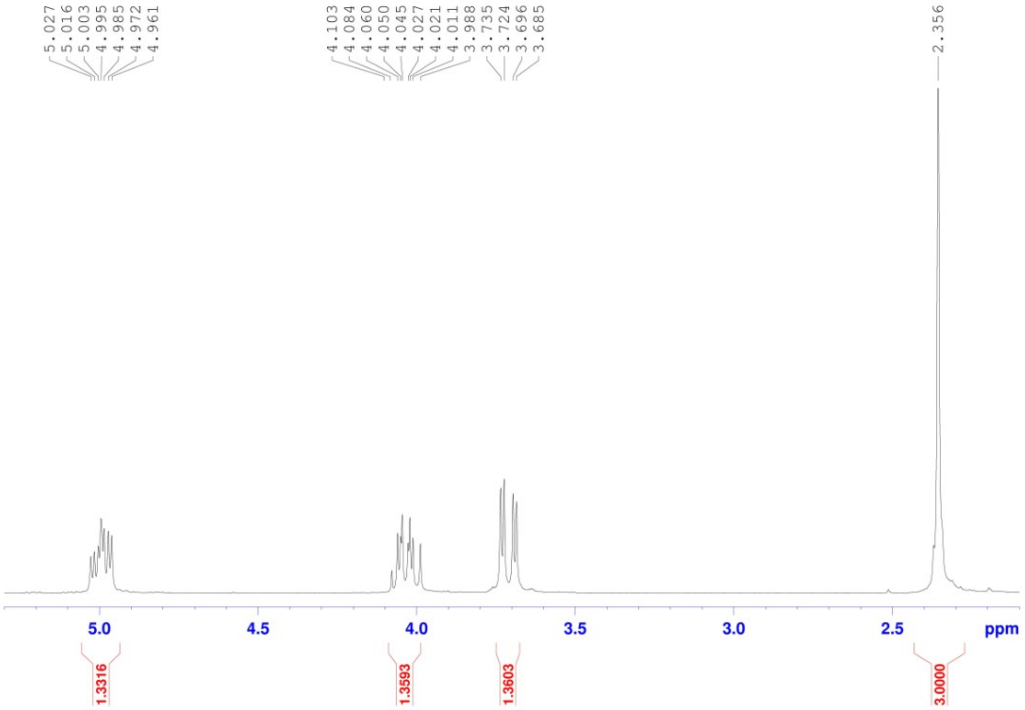
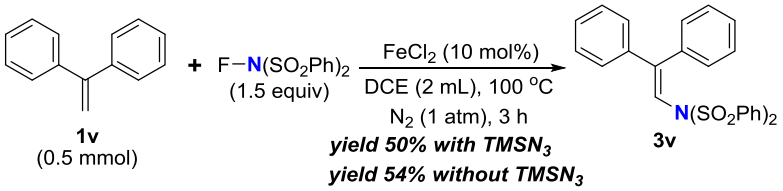


Figure S2. ^{1}H-NMR of the competition experiment between styrenes 1b and 1e.

3) C-H Amination of 1,1-disubstituted Alkenes with NFSI



1v (0.5 mmol)

3v

Typical Procedure: To a reaction tube charged with $FeCl_2$ (6.3 mg, 0.05 mmol) and NFSI (236.5 mg, 0.75 mmol) was added a solution of 1,1-diphenylethylene (**1v**, 88.3 μL, 0.5 mmol) and $TMSN_3$ (98.6 μL, 0.75 mmol, or 0 μL, 0 mmol) in DCE (2 mL) via a syringe under N_2 (1 atm). The reaction mixture was stirred at

100 °C for 3 hours. After rapidly cooling by ice, the mixture was diluted with ethyl acetate, filtered through a celite pad, and concentrated *in vacuo* to give dark residue, which was then purified by flash chromatography using petroleum ether and ethyl acetate (15:1, *v*/*v*) as the eluent on silical gel to afford C-H aminated product **3v** (117.8 mg, 50% with $TMSN_3$, or 128.2 mg, 50% without $TMSN_3$) as white solid (m.p. 170.5-171.9 °C).

***N*-(2,2-Diphenylvinyl)-*N*-(phenylsulfonyl)benzenesulfonamide[3] (3v):**

^{1}H NMR ($CDCl_3$, 400 MHz): δ = 7.71-7.69 (m, 4H), 7.58-7.55 (m, 2H), 7.41-7.36 (m, 4H), 7.34-7.20 (m, 10H), 6.13 (s, 1H) ppm; **^{13}C NMR ($CDCl_3$, 100 MHz)**: δ = 152.25, 139.82, 138.63, 136.73, 133.78, 129.87, 129.07, 128.76, 128.69, 128.64, 128.35, 128.23, 128.13, 116.26 ppm. **HRMS *m/z* (ESI)** calcd for $[C_{26}H_{21}NO_4S_2+Na]^+$ 498.0810, found 498.0818.

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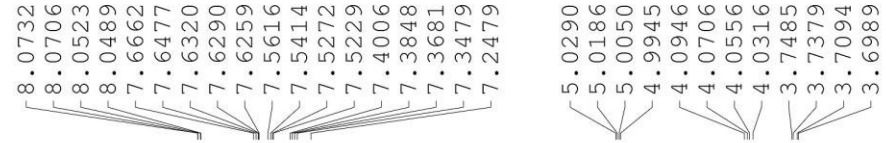
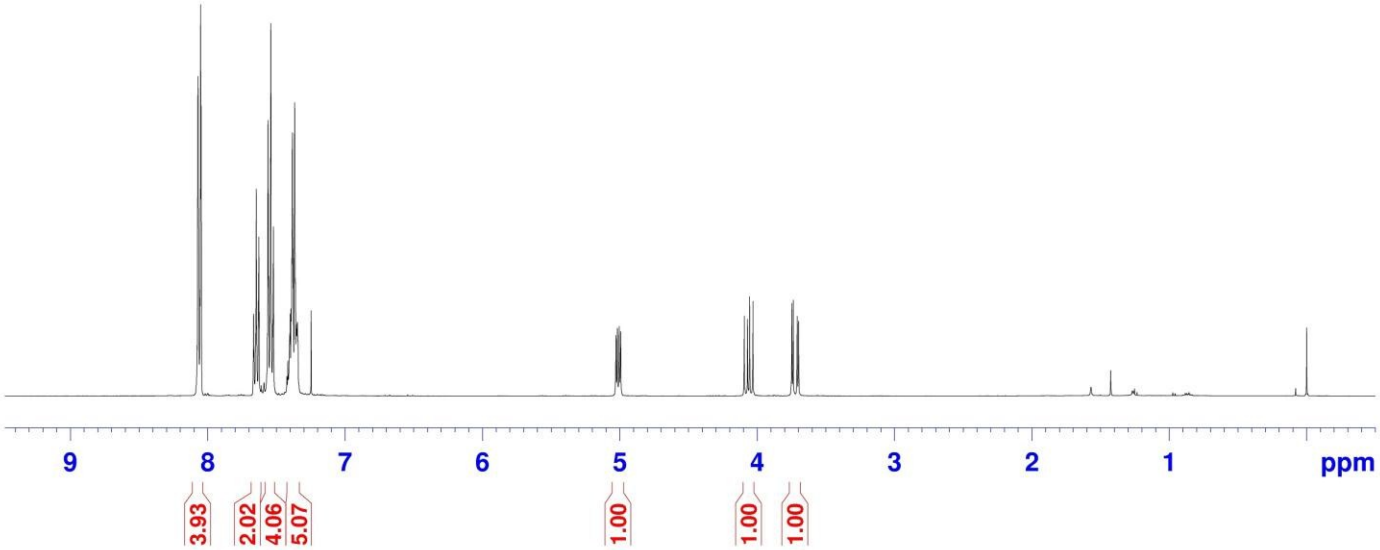
DOI: 10.1039/C8OB00699G

References

[1] Zhang, B.; Studer, A. *Org. Lett.* **2014**, *16*, 1790-1793.
[2] Zhang, H.; Song, Y.; Zhao, J.; Zhang, J.; Zhang, Q. *Angew. Chem. Int. Ed.* **2014**, *53*, 11079-11083.
[3] Weng, S.-S.; Hsieh, K.-Y.; Zeng, Z.-J.; Zhang, J.-W. *Tetrahedron Lett.* **2017**, *58*, 670-673.



2a

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FIDRES          0.122266 Hz
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RG                  51.19
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TE                300.0 K
D1           1.00000000 sec
TD0                     1

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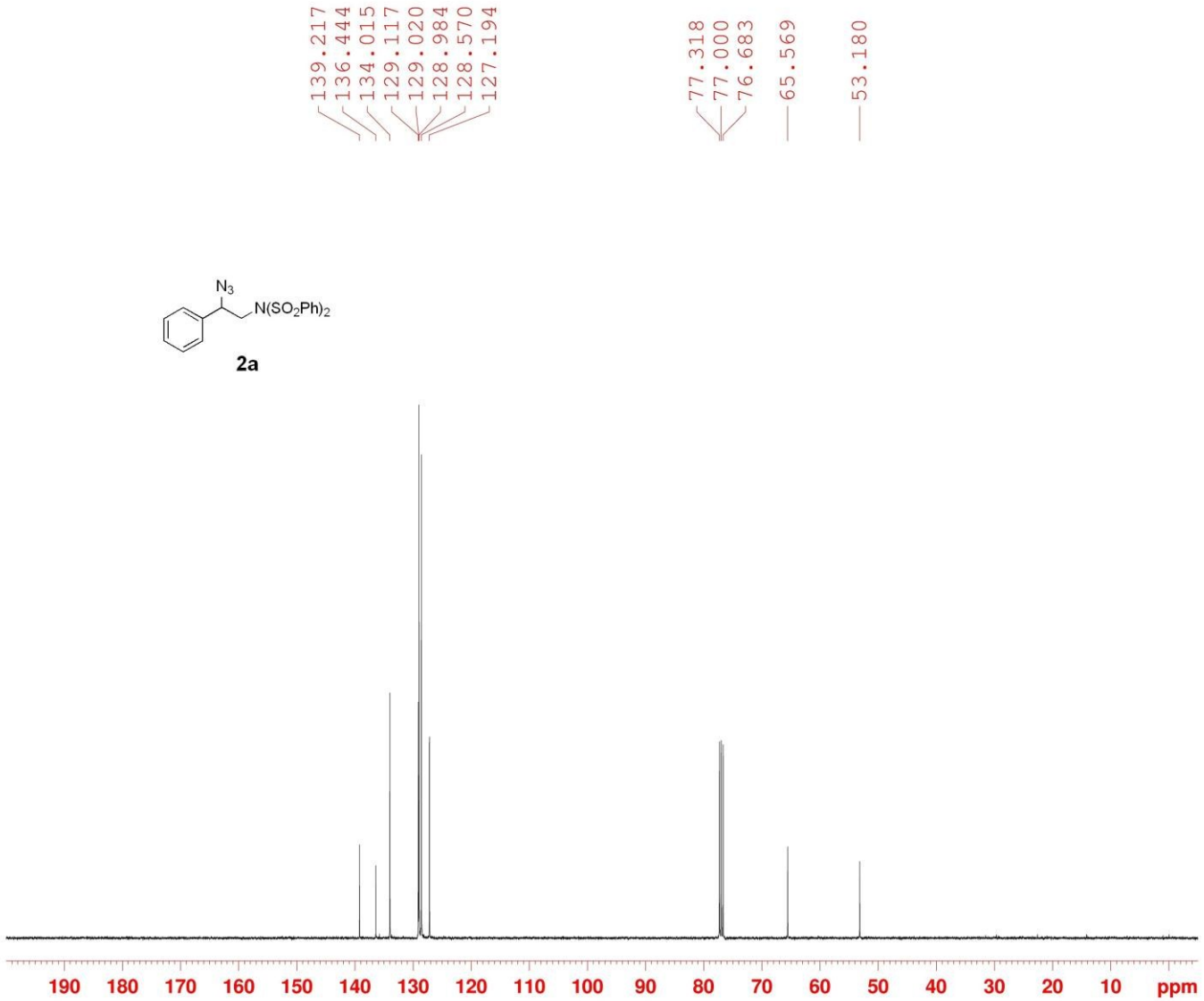

BRUKER

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TE	300.0 K
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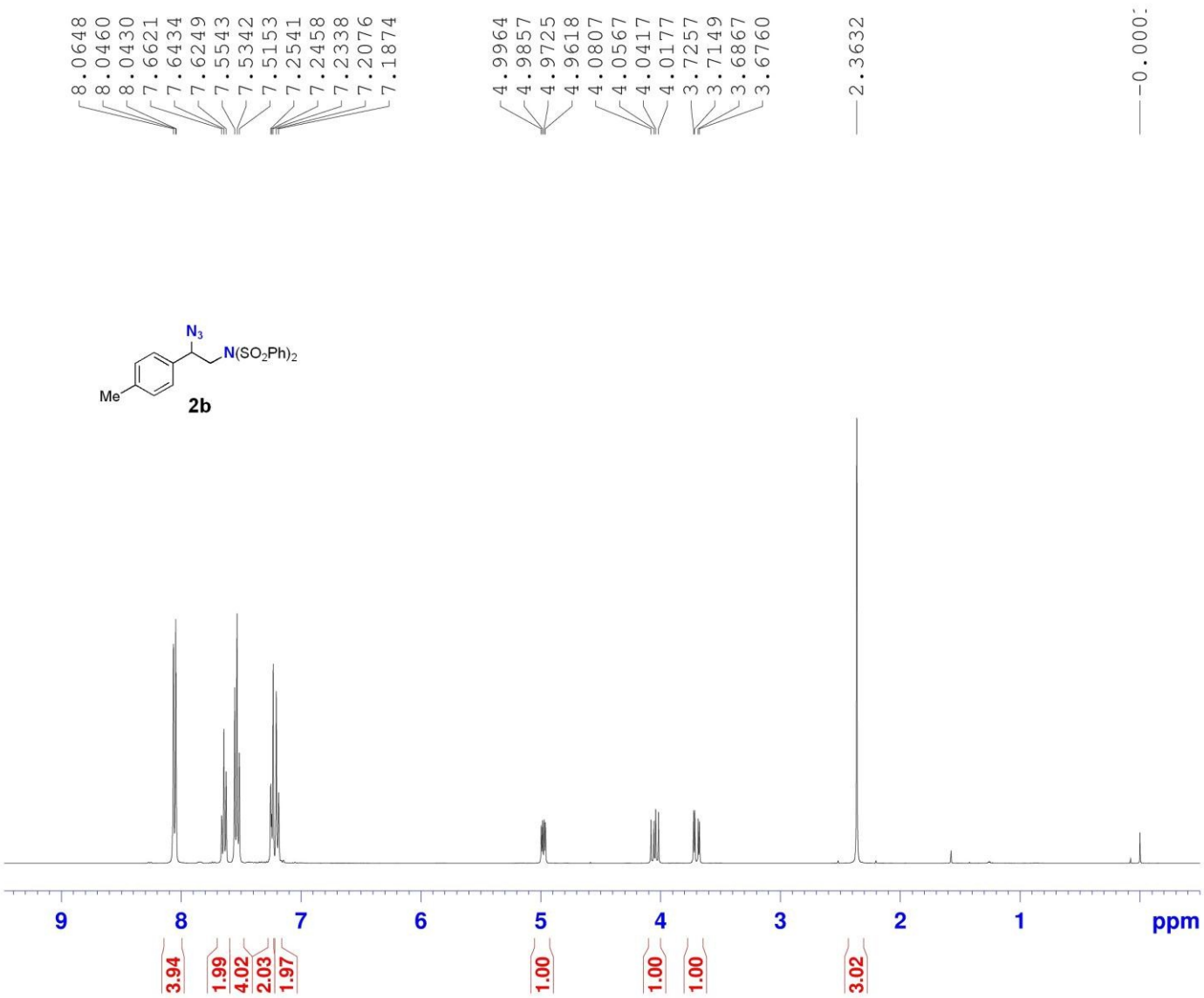

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FIDRES	0.122266 Hz
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RG	41.07
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DE	6.50 usec
TE	296.8 K
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TD0	1
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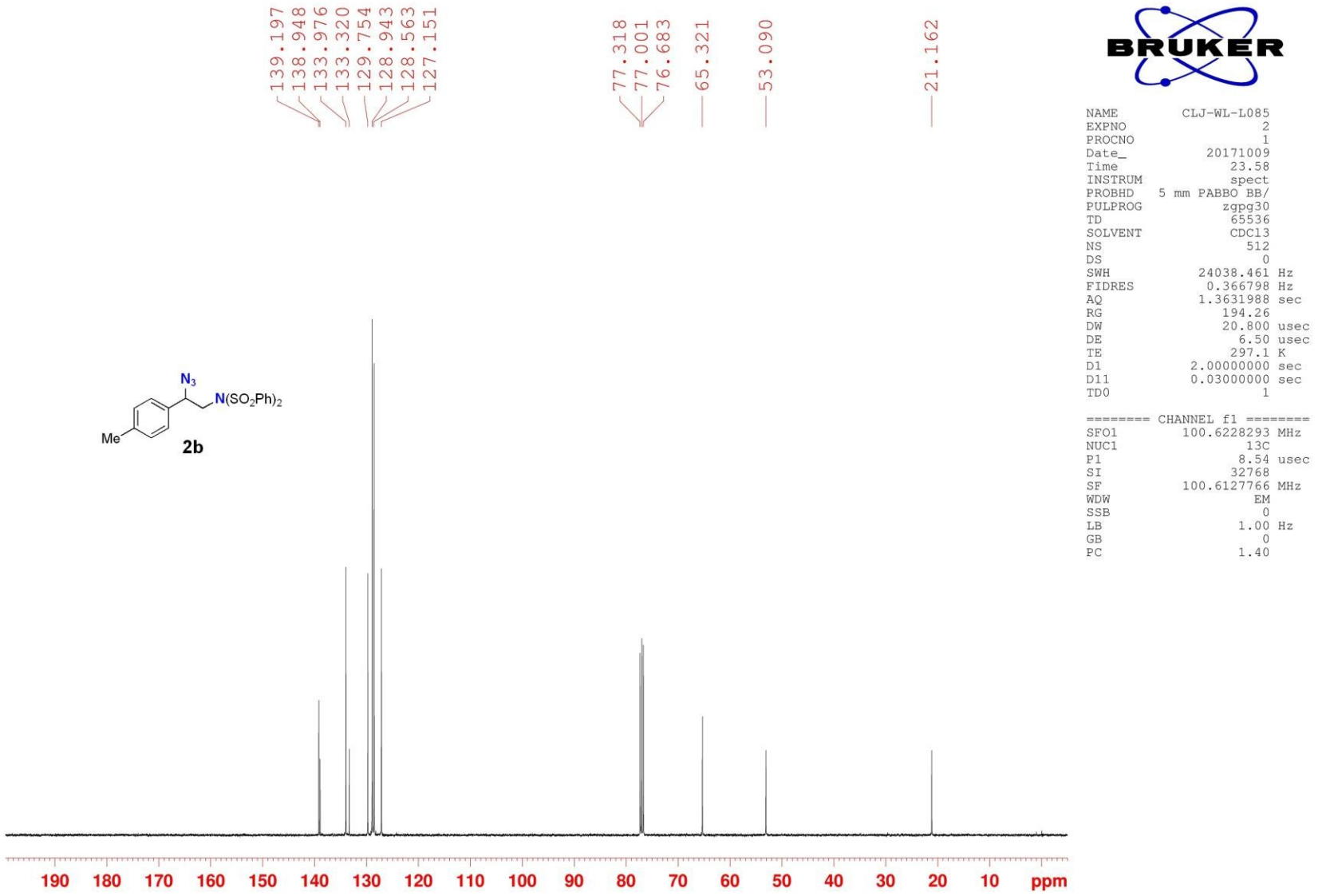

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FIDRES	0.366798 Hz
AQ	1.3631988 sec
RG	194.26
DW	20.800 usec
DE	6.50 usec
TE	297.1 K
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D11	0.03000000 sec
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NUC1	13C
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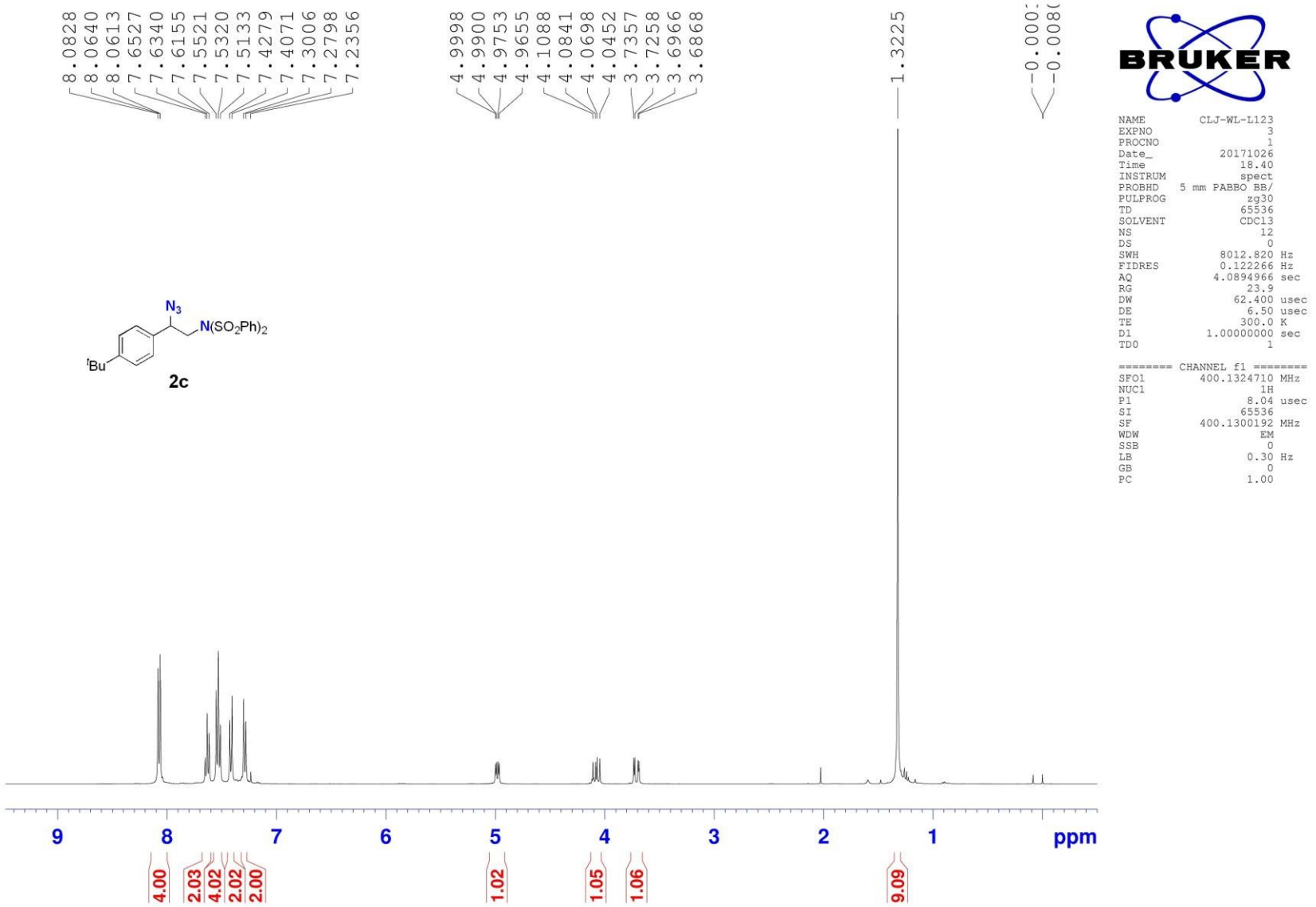
GB	0
PC	1.40



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FIDRES	0.122266 Hz
AQ	4.0894966 sec
RG	23.9
DW	62.400 usec
DE	6.50 usec
TE	300.0 K
D1	1.00000000 sec
TD0	1
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NUC1	1H
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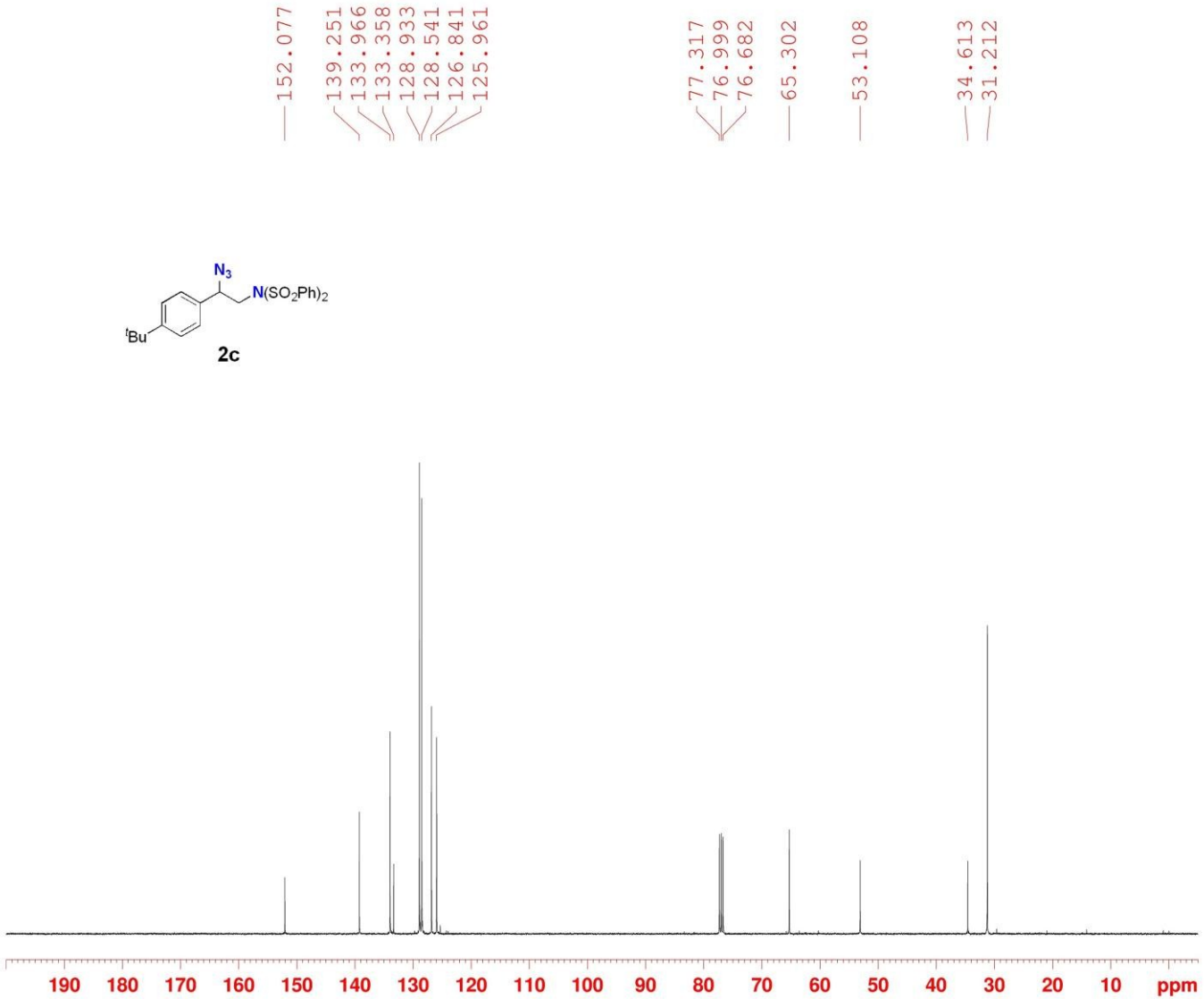

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PROCNO	1
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FIDRES	0.366798 Hz
AQ	1.3631988 sec
RG	194.26
DW	20.800 usec
DE	6.50 usec
TE	300.0 K
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D11	0.03000000 sec
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NUC1	13C
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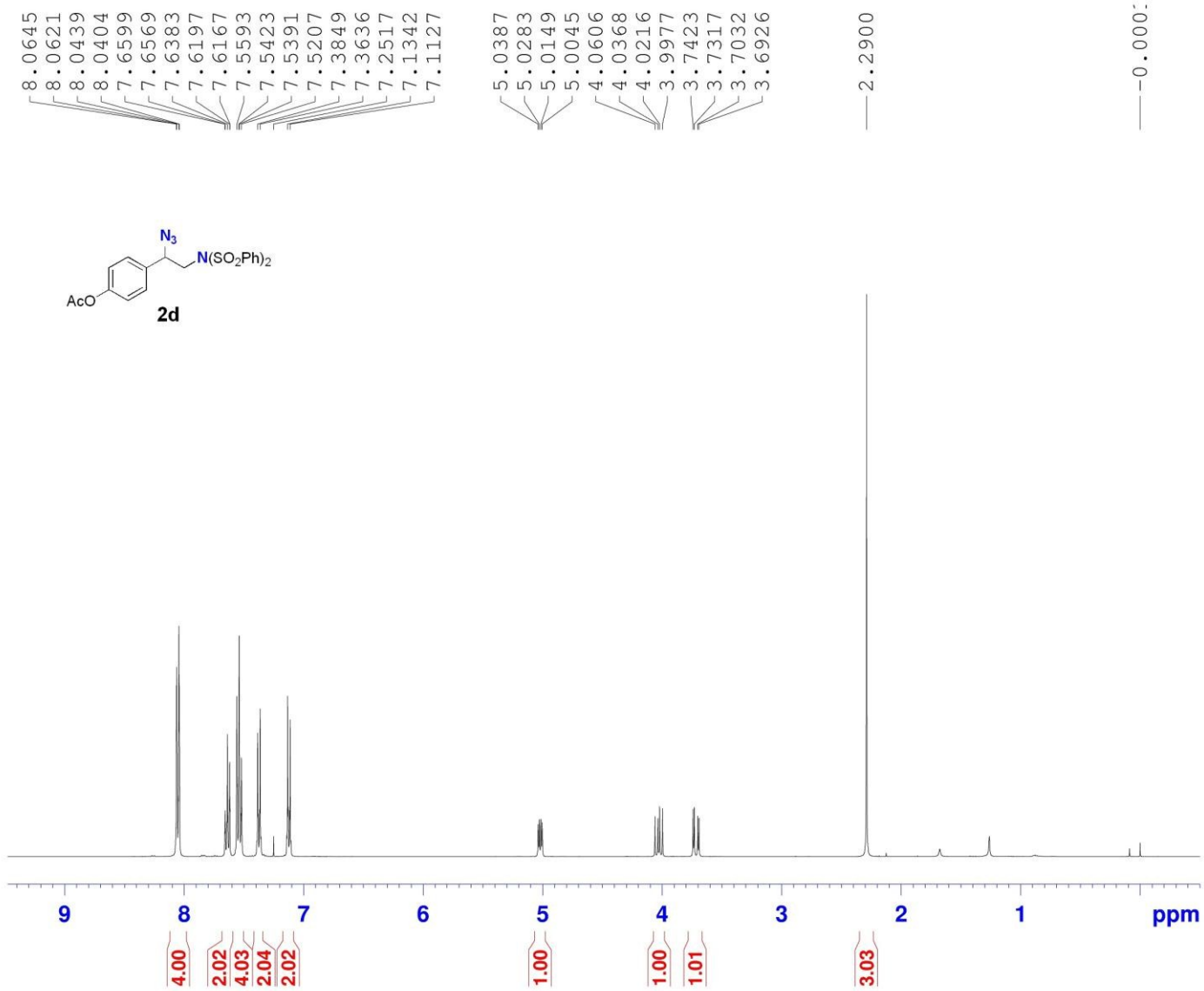

BRUKER

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PROCNO	1
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PULPROG	zg30
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FIDRES	0.122266 Hz
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DE	6.50 usec
TE	300.0 K
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NUC1	1H
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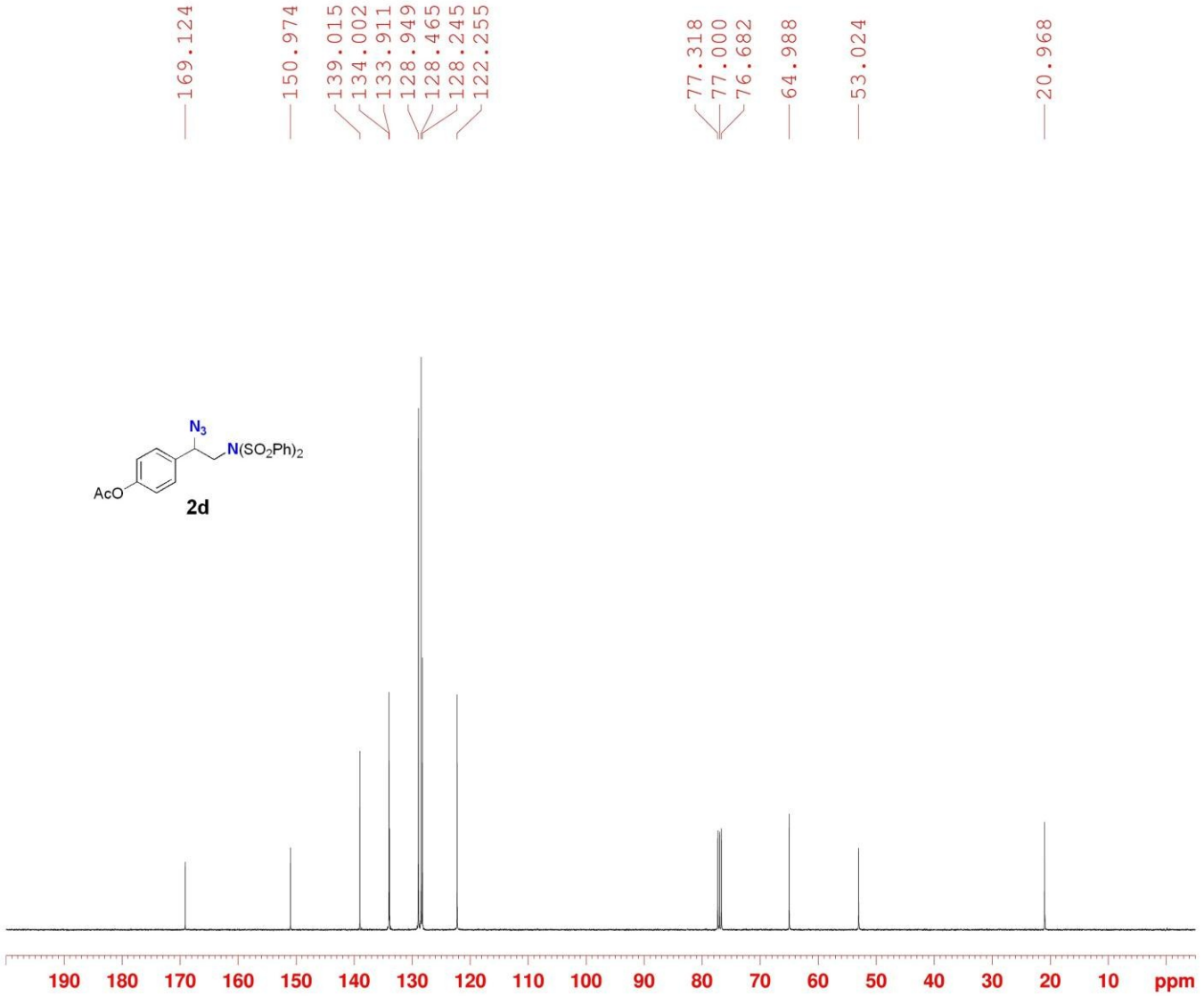

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FIDRES	0.366798 Hz
AQ	1.3631988 sec
RG	194.26
DW	20.800 usec
DE	6.50 usec
TE	300.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
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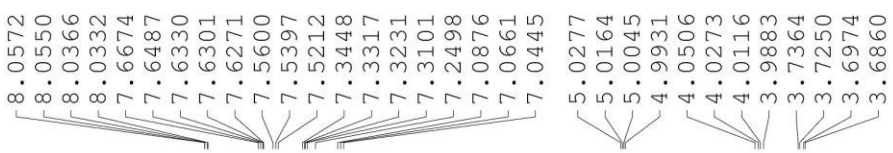

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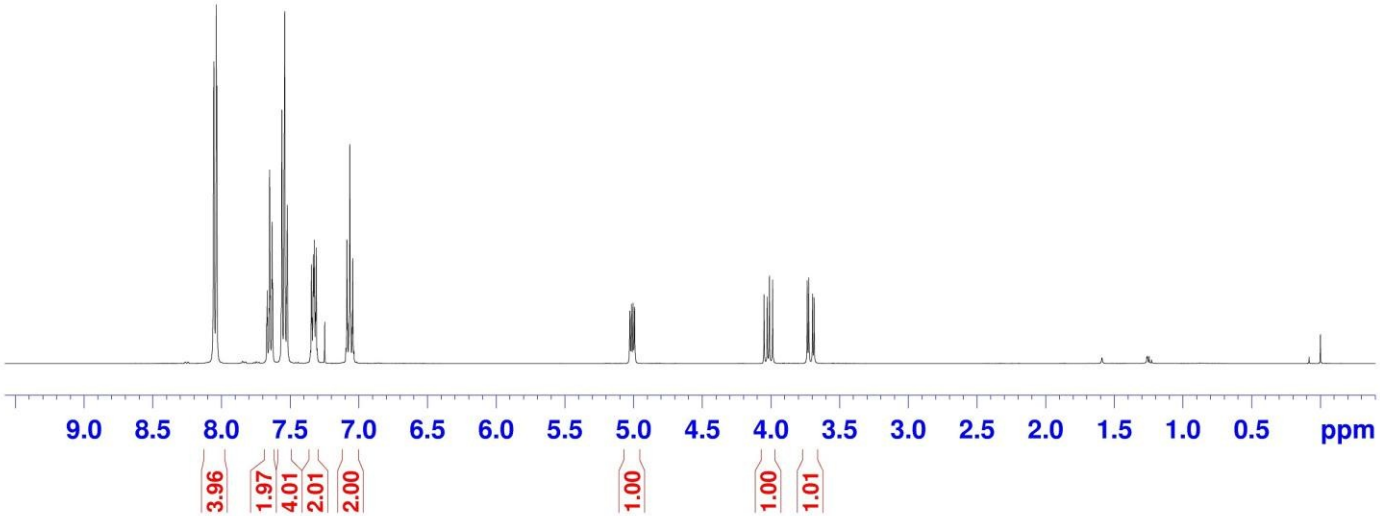


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DS                      0
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FIDRES           0.122266 Hz
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RG                   36.4
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TE                 300.0 K
D1            1.00000000 sec
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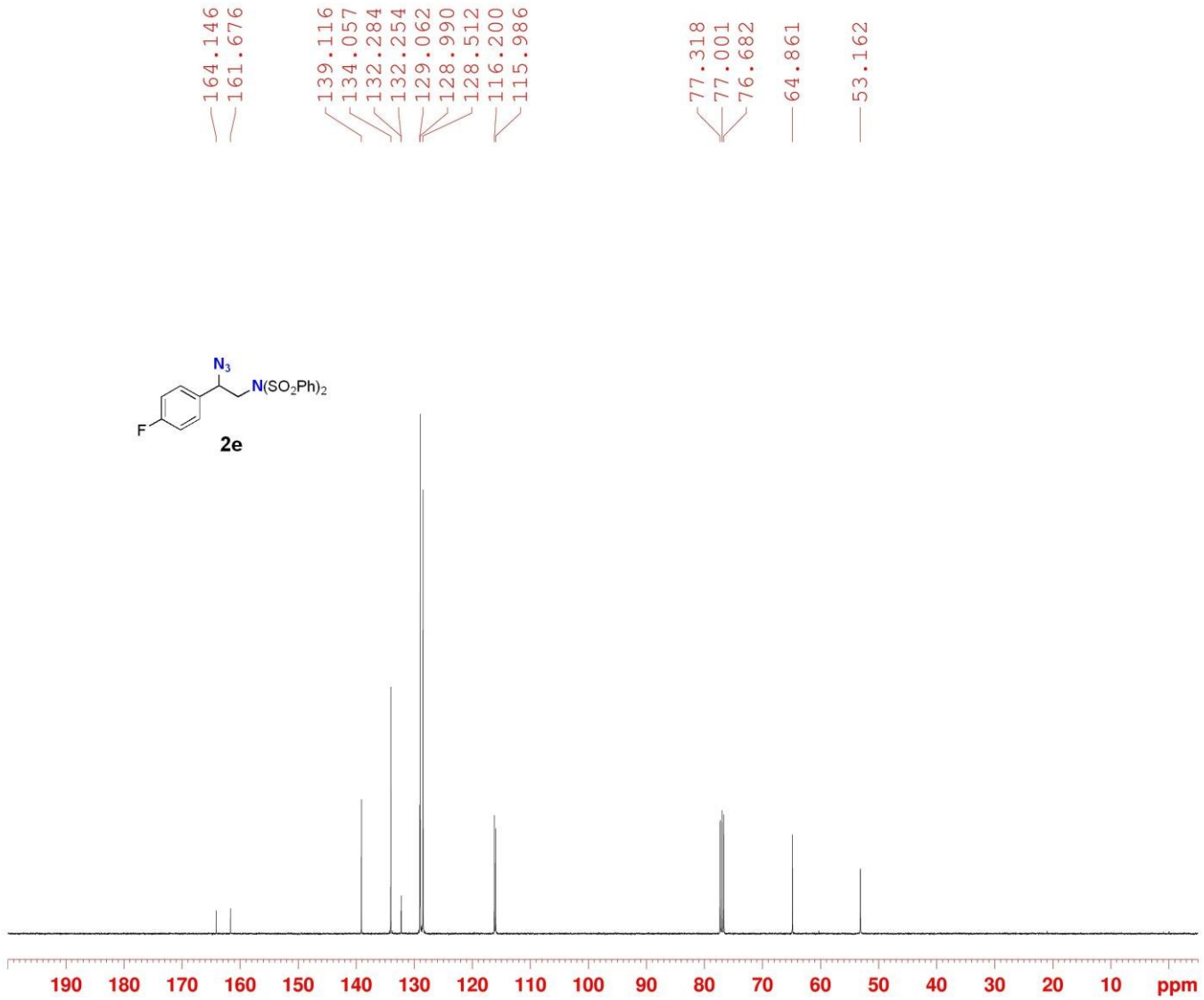

BRUKER

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SOLVENT	CDCl3
NS	512
DS	0
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FIDRES	0.366798 Hz
AQ	1.3631988 sec
RG	194.26
DW	20.800 usec
DE	6.50 usec
TE	300.0 K
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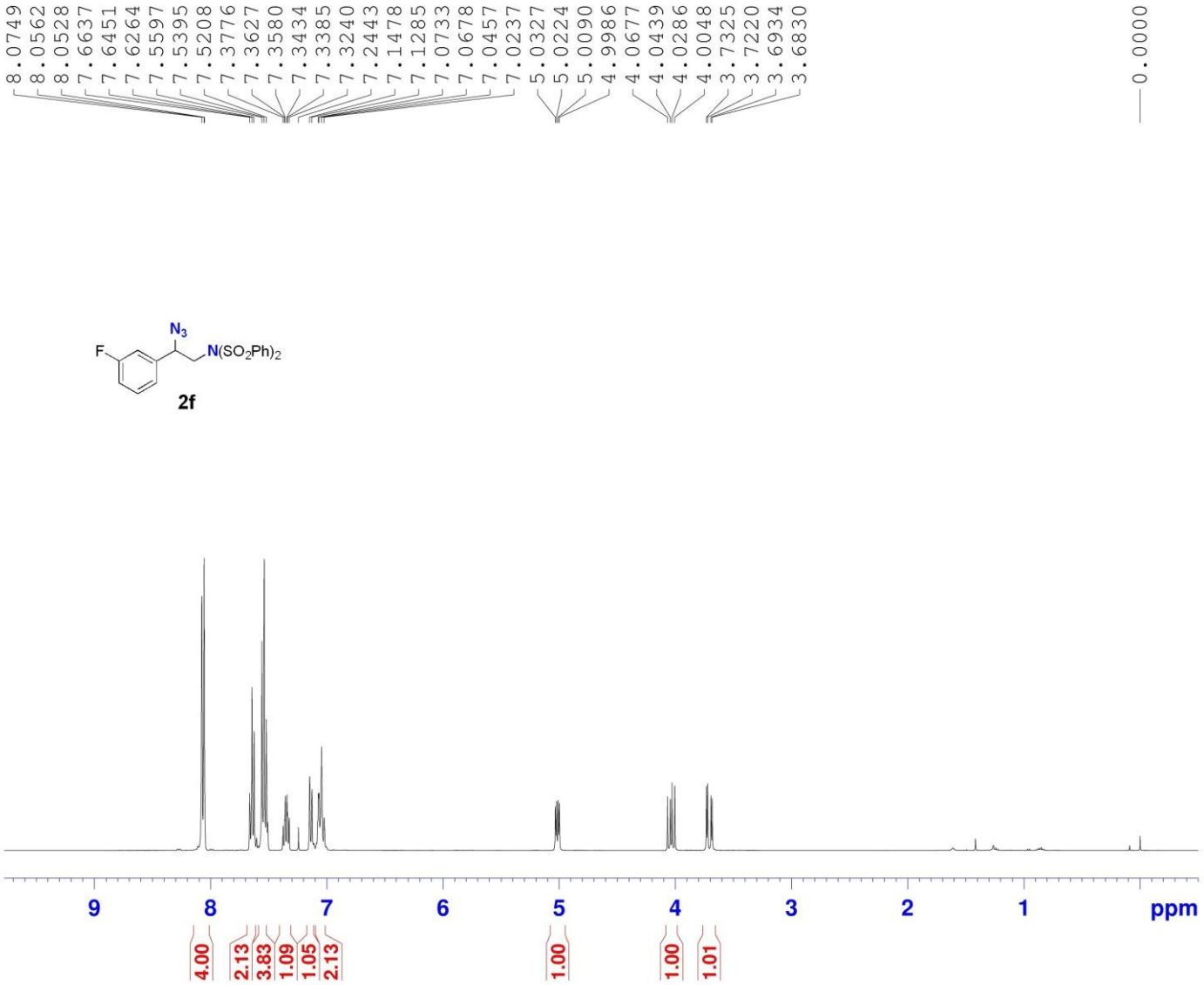


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DS                        0
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FIDRES            0.122266 Hz
AQ                4.0894966 sec
RG                     18.6
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DE                   6.50 usec
TE                  300.0 K
D1              1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
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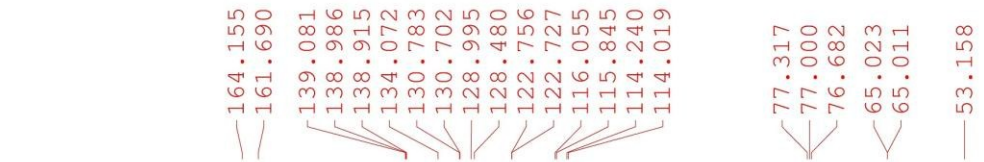

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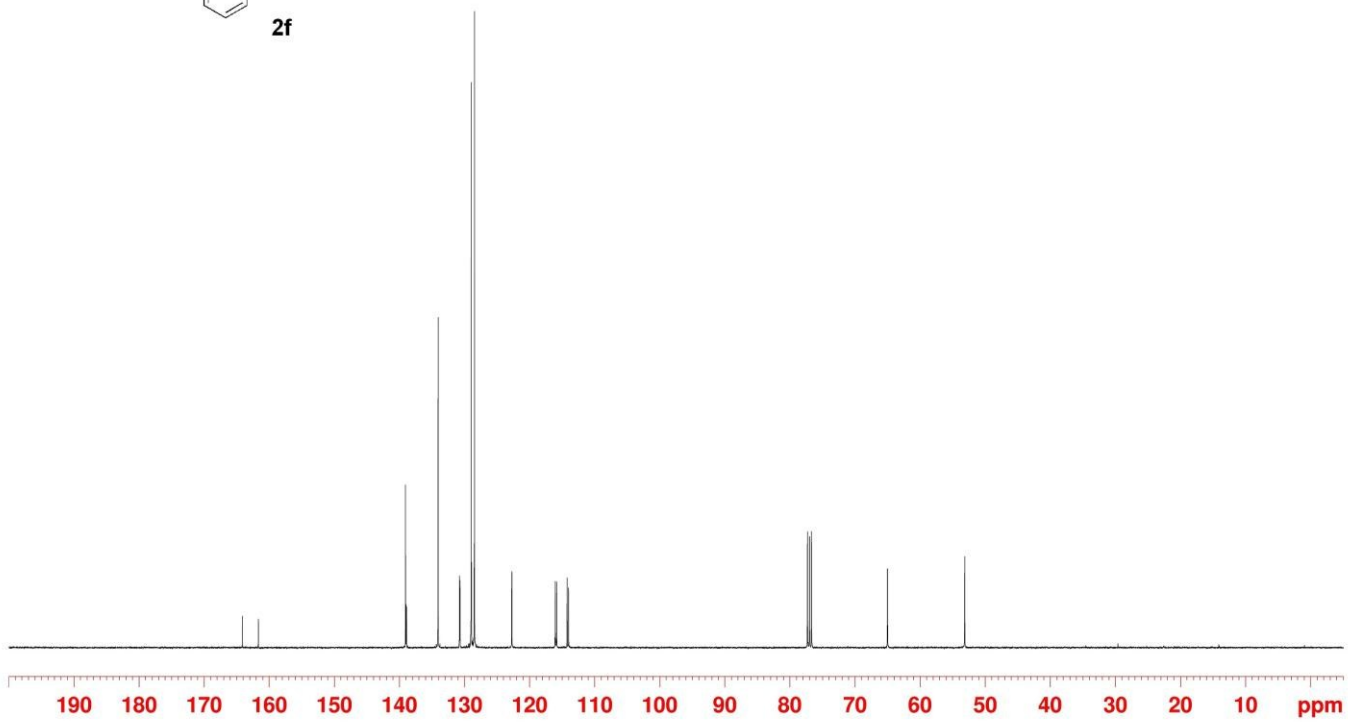

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FIDRES	0.366798 Hz
AQ	1.3631988 sec
RG	194.26
DW	20.800 usec
DE	6.50 usec
TE	300.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
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NUC1	13C
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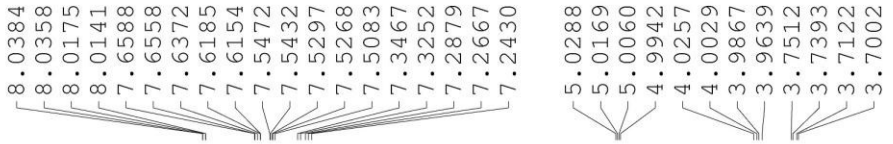

2g

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TD                    65536
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NS                       12
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FIDRES            0.122266 Hz
AQ                4.0894966 sec
RG                    29.13
DW                   62.400 usec
DE                    6.50 usec
TE                   300.0 K
D1              1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
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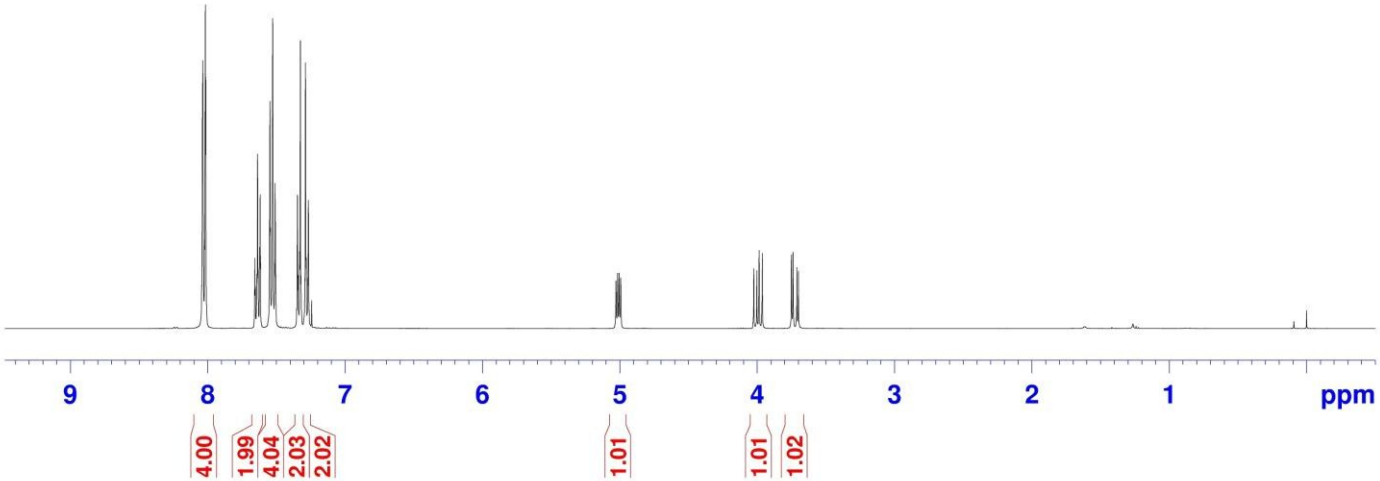

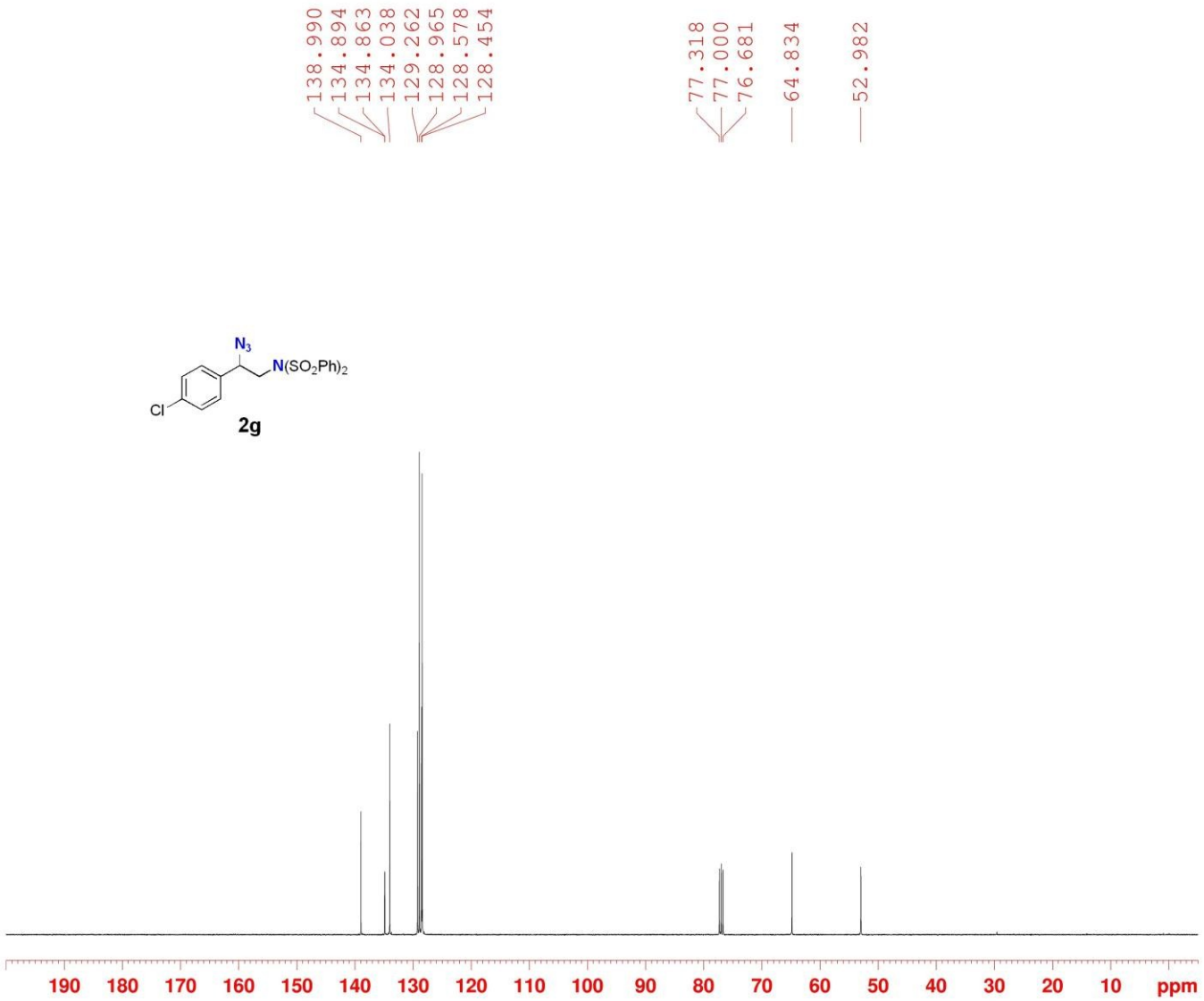

BRUKER

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FIDRES	0.366798 Hz
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RG	194.26
DW	20.800 usec
DE	6.50 usec
TE	300.0 K
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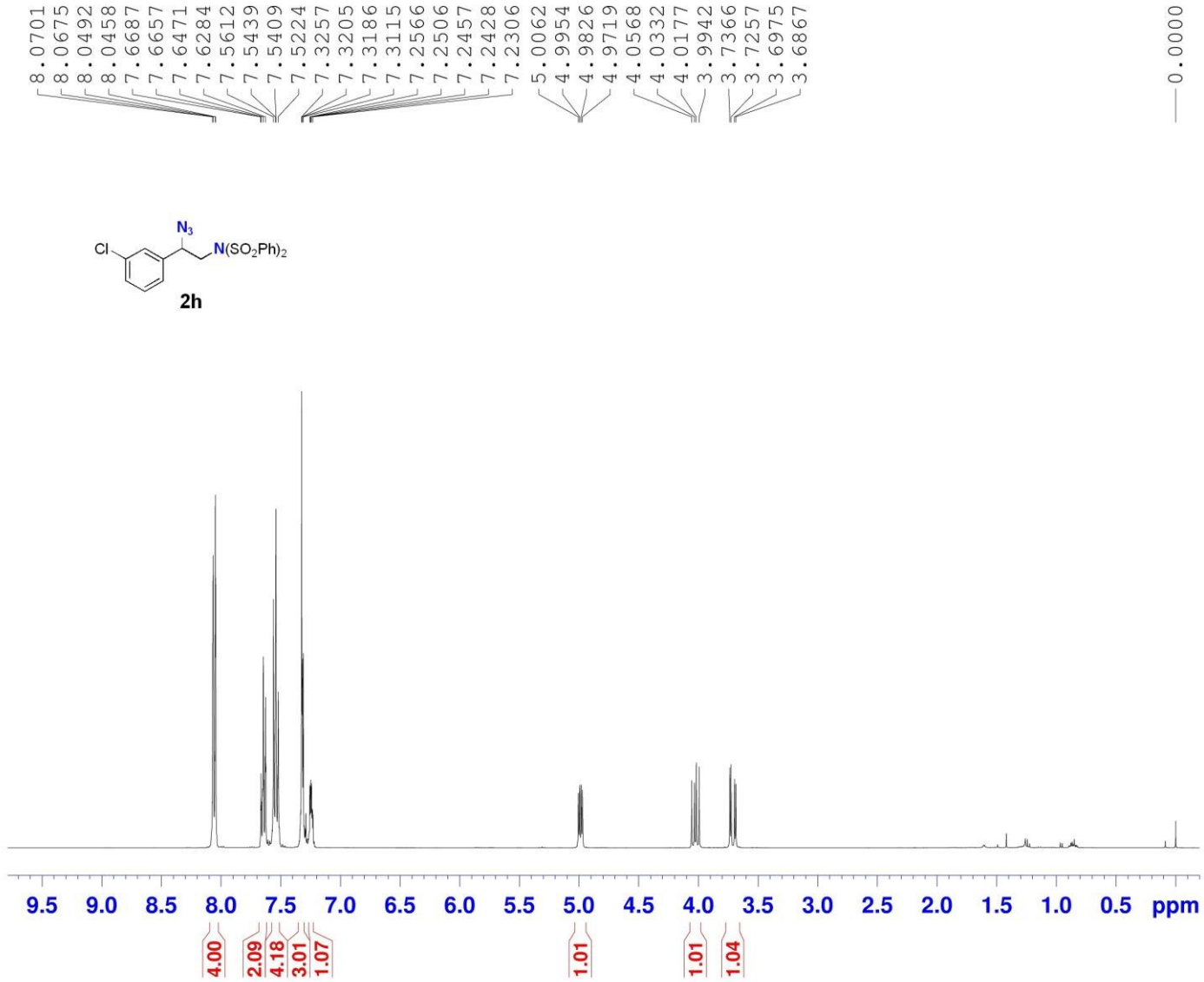

BRUKER

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FIDRES	0.122266 Hz
AQ	4.0894966 sec
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DE	6.50 usec
TE	300.0 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
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NUC1	1H
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SSB	0
LB	0.30 Hz
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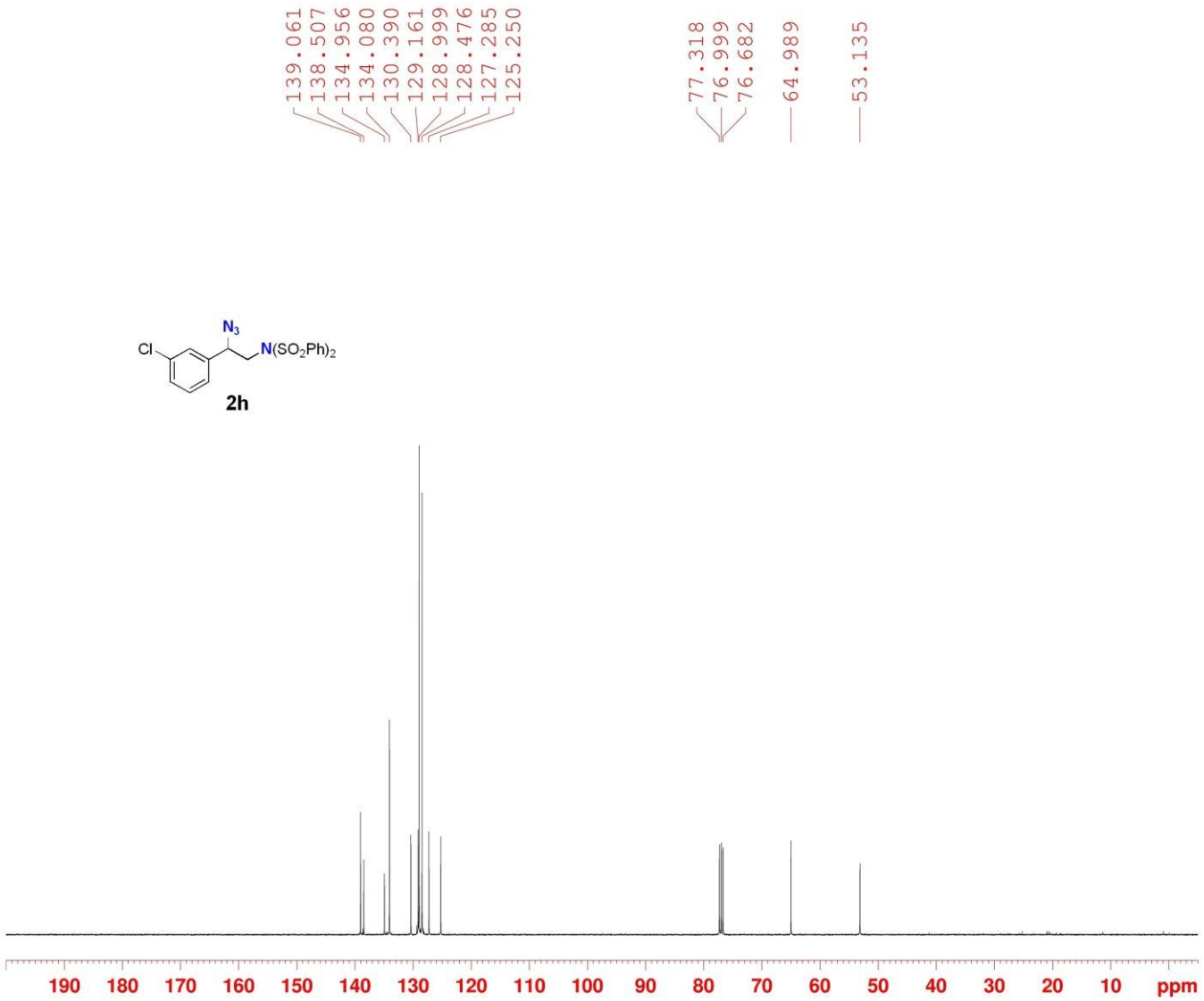

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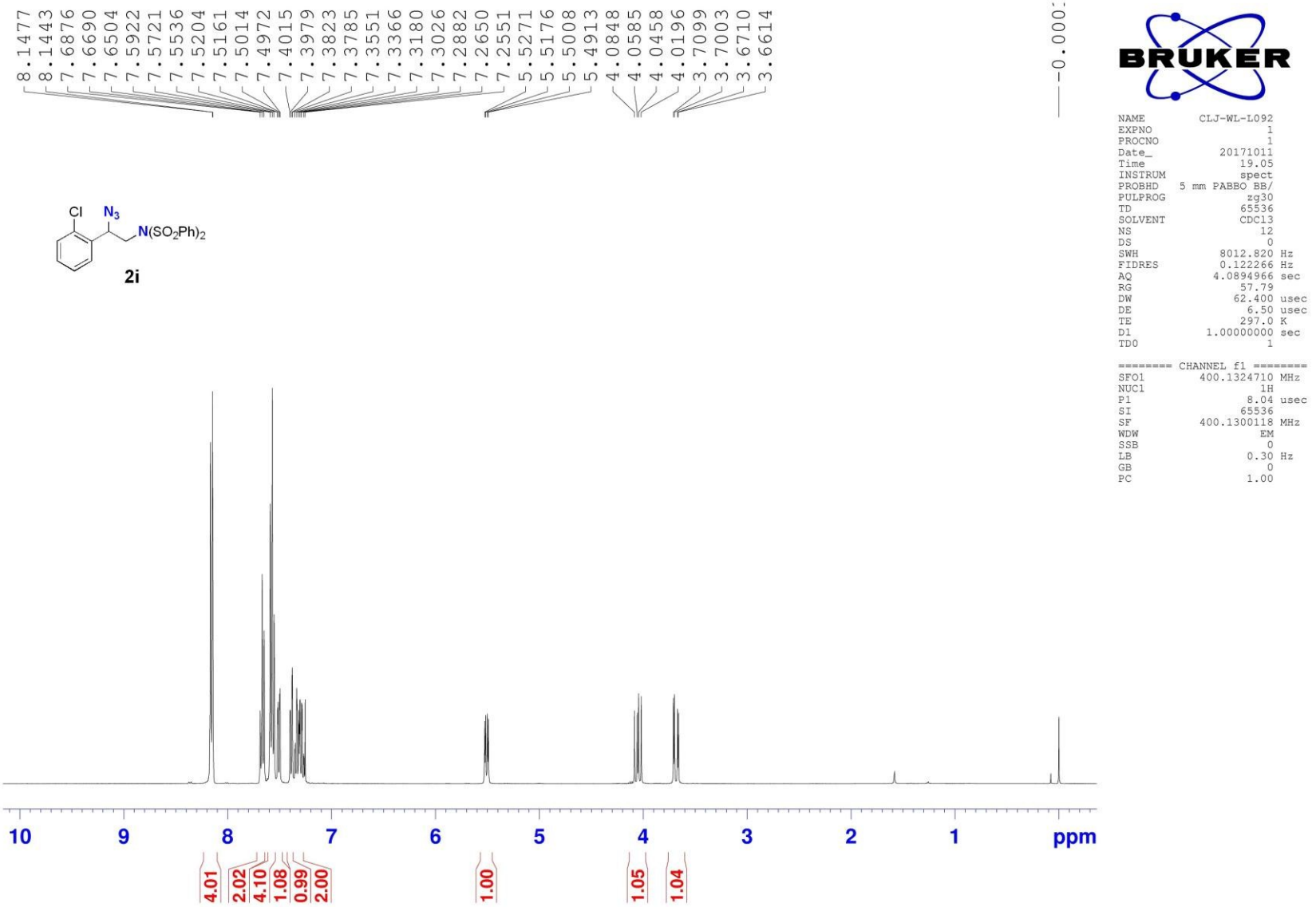
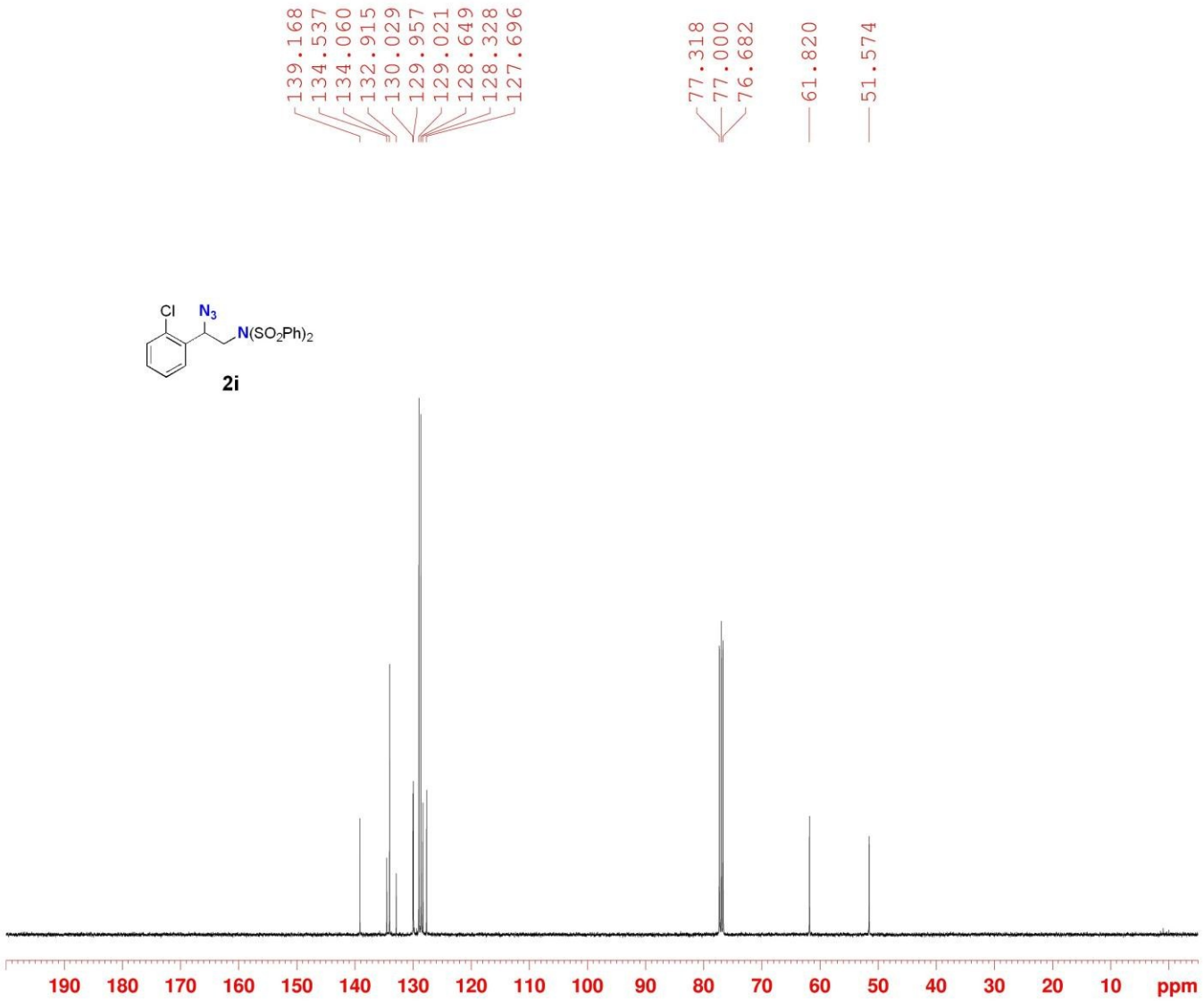
DS	0
SWH	24038.461 Hz
FIDRES	0.366798 Hz
AQ	1.3631988 sec
RG	194.26
DW	20.800 usec
DE	6.50 usec
TE	300.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
SFO1	100.6228293 MHz
NUC1	13C
P1	8.54 usec
SI	32768
SF	100.6127799 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40



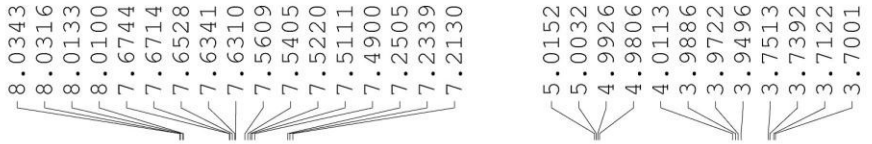
BRUKER

NAME	CLJ-WL-L092
EXPNO	1
PROCNO	1
Date_	20171011
Time	19.05
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	12
DS	0
SWH	8012.820 Hz
FIDRES	0.122266 Hz
AQ	4.0894966 sec
RG	57.79
DW	62.400 usec
DE	6.50 usec
TE	297.0 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
SFO1	400.1324710 MHz
NUC1	1H
P1	8.04 usec
SI	65536
SF	400.1300118 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



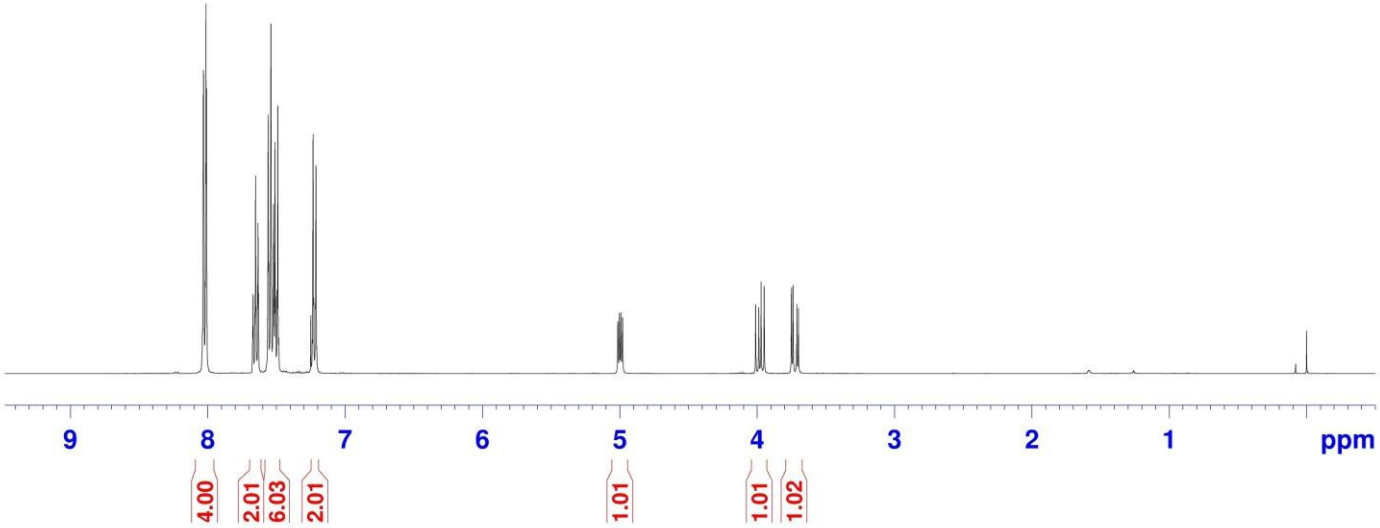
BRUKER

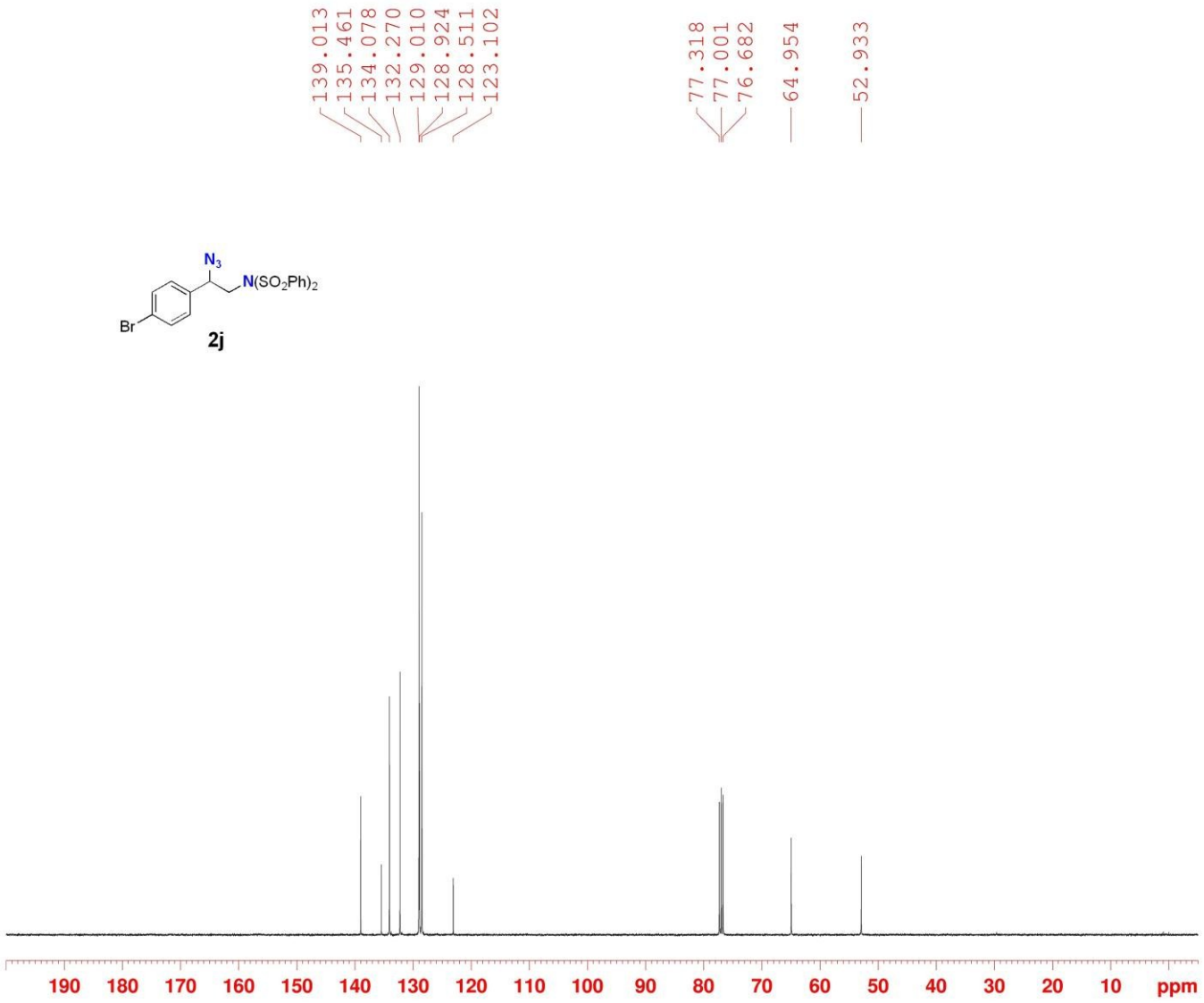
NAME	CLJ-WL-L092
EXPNO	2
PROCNO	1
Date_	20171011
Time	19.36
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	512
DS	0
SWH	24038.461 Hz
FIDRES	0.366798 Hz
AQ	1.3631988 sec
RG	194.26
DW	20.800 usec
DE	6.50 usec
TE	297.3 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
SFO1	100.6228293 MHz
NUC1	13C
P1	8.54 usec
SI	32768
SF	100.6127746 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40



2j

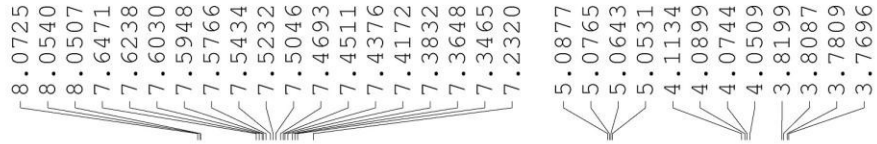

```
NAME            CLJ-WL-LO81
EXPNO                     1
PROCNO                    1
Date_              20170929
Time                  20.45
INSTRUM               spect
PROBHD    5 mm PABBO BB/
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       12
DS                        0
SWH               8012.820 Hz
FIDRES            0.122266 Hz
AQ                4.0894966 sec
RG                    47.53
DW                   62.400 usec
DE                    6.50 usec
TE                   300.0 K
D1             1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
SFO1           400.1324710 MHz
NUC1                     1H
P1                    8.04 usec
SI                    65536
SF             400.1300137 MHz
WDW                      EM
SSB                       0
LB                     0.30 Hz
GB                        0
PC                     1.00
```

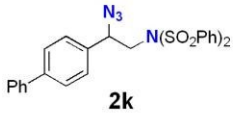




BRUKER

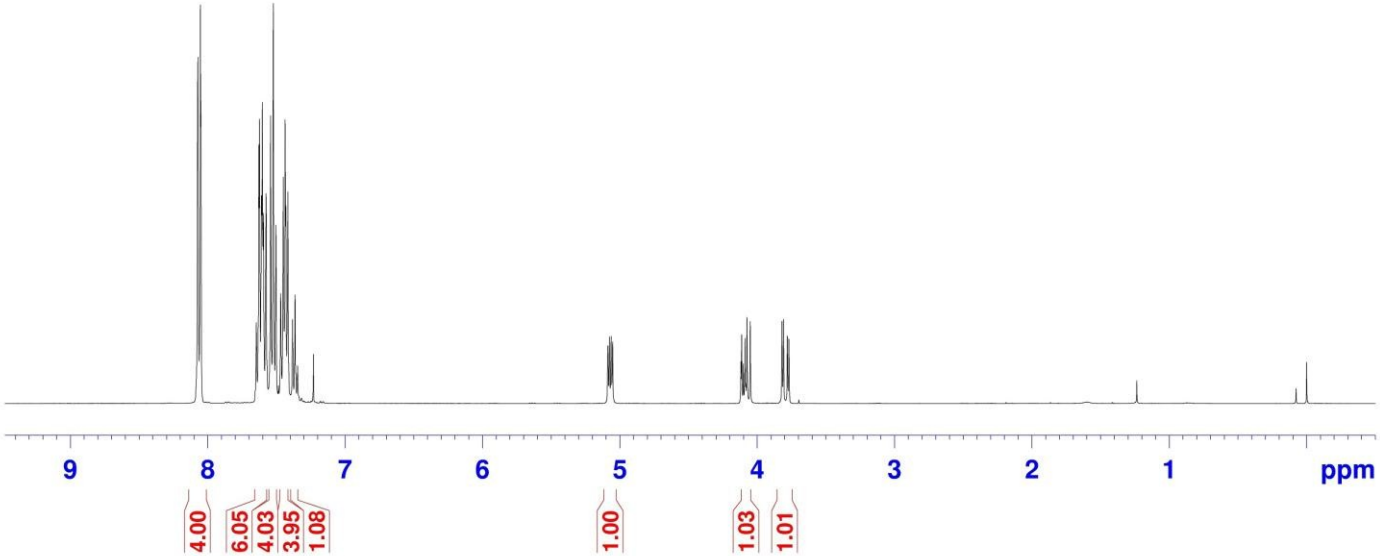
NAME	CLJ-WL-L081
EXPNO	2
PROCNO	1
Date_	20170929
Time	21.16
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	512
DS	0
SWH	24038.461 Hz
FIDRES	0.366798 Hz
AQ	1.3631988 sec
RG	194.26
DW	20.800 usec
DE	6.50 usec
TE	300.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
SFO1	100.6228293 MHz
NUC1	13C
P1	8.54 usec
SI	32768
SF	100.6127767 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

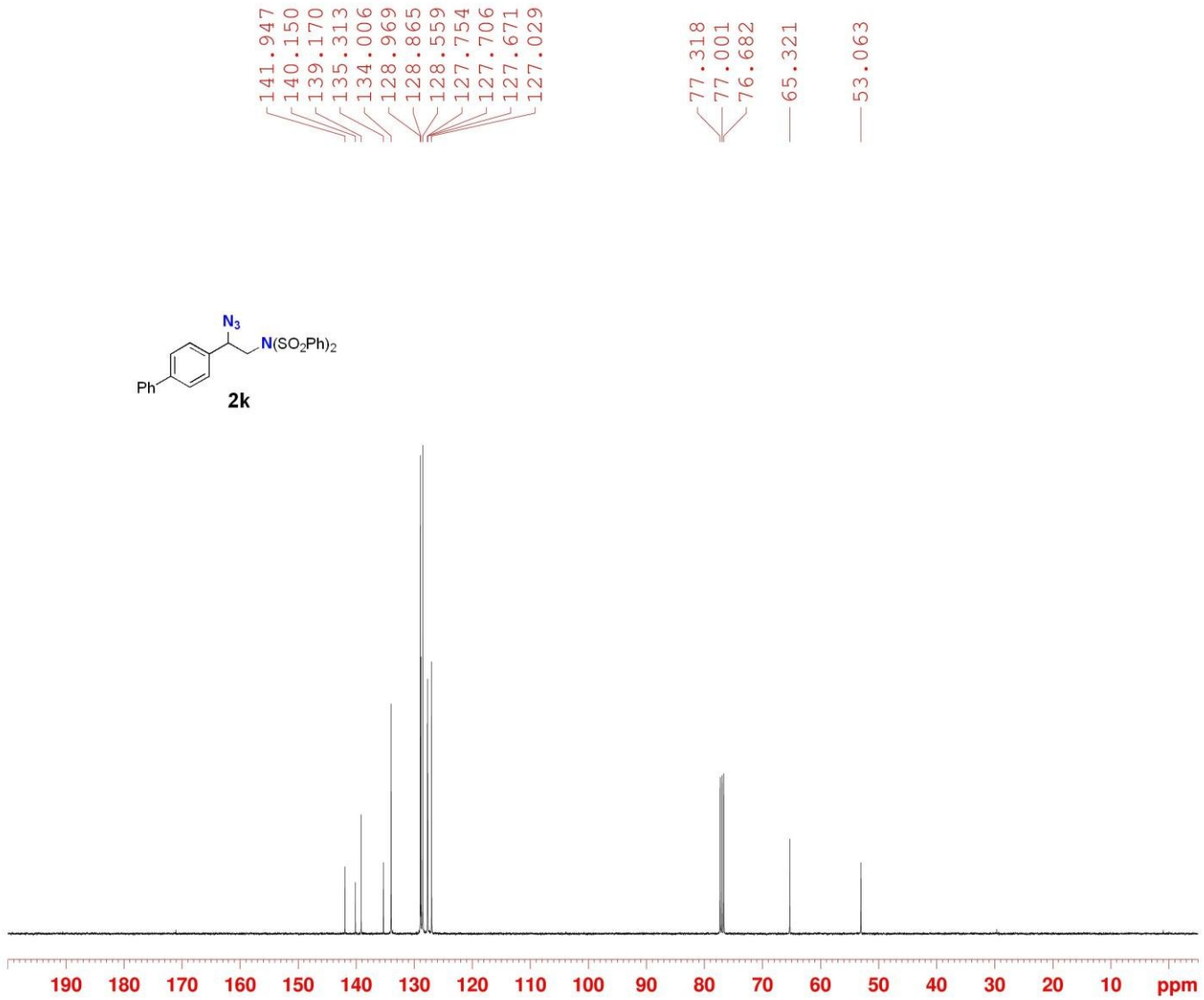




2k

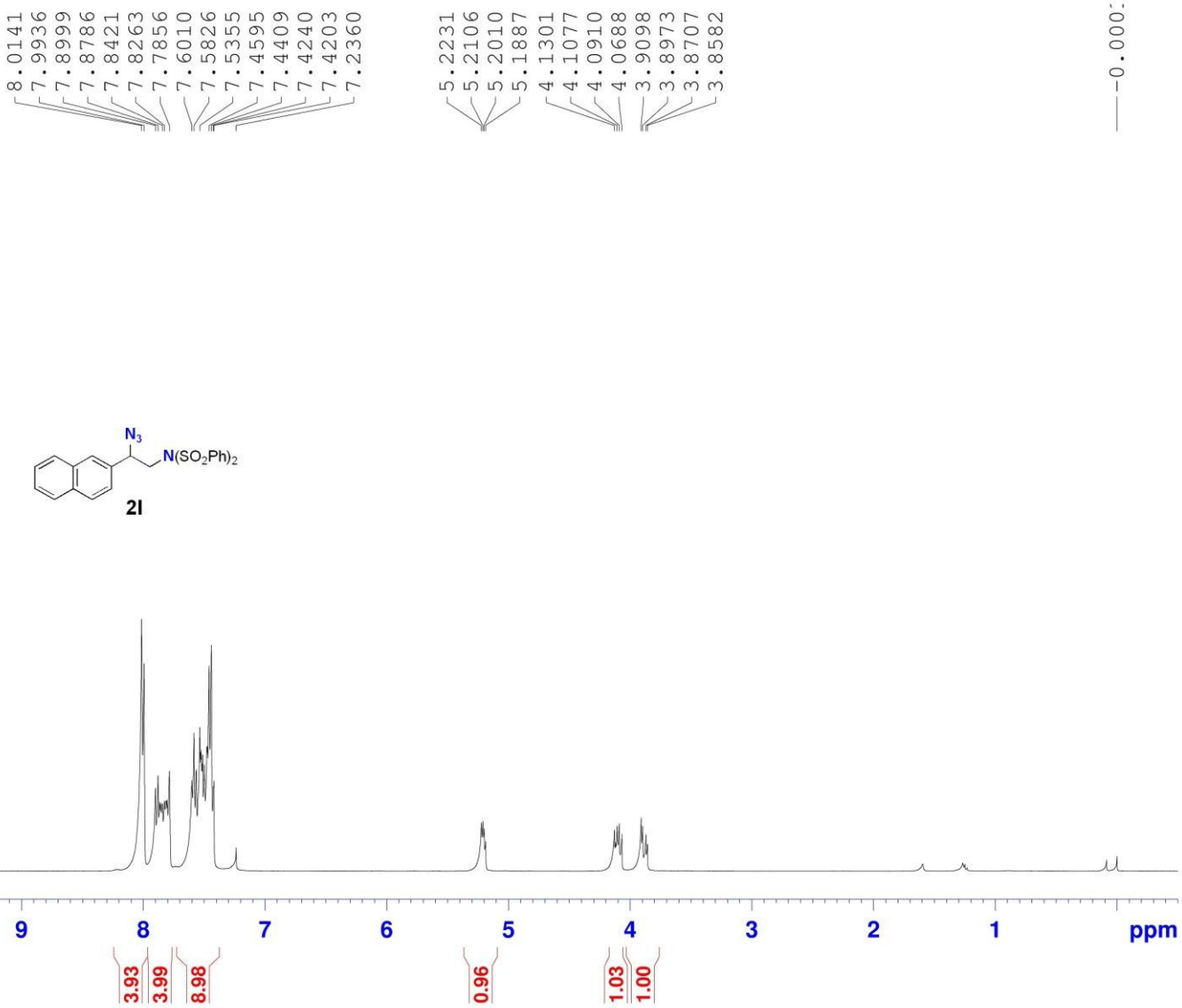

```
NAME          CLJ-WL-L117
EXPNO                   1
PROCNO                  1
Date_            20171023
Time                19.11
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     12
DS                      0
SWH             8012.820 Hz
FIDRES          0.122266 Hz
AQ              4.0894966 sec
RG                   23.9
DW                62.400 usec
DE                 6.50 usec
TE                300.0 K
D1           1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1         400.1324710 MHz
NUC1                   1H
P1                  8.04 usec
SI                  65536
SF           400.1300210 MHz
WDW                    EM
SSB                     0
LB                 0.30 Hz
GB                      0
PC                   1.00
```





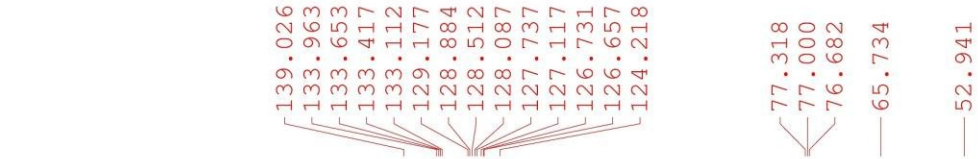
BRUKER

NAME	CLJ-WL-L117
EXPNO	2
PROCNO	1
Date_	20171023
Time	19.42
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	512
DS	0
SWH	24038.461 Hz
FIDRES	0.366798 Hz
AQ	1.3631988 sec
RG	194.26
DW	20.800 usec
DE	6.50 usec
TE	300.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
SFO1	100.6228293 MHz
NUC1	13C
P1	8.54 usec
SI	32768
SF	100.6127780 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

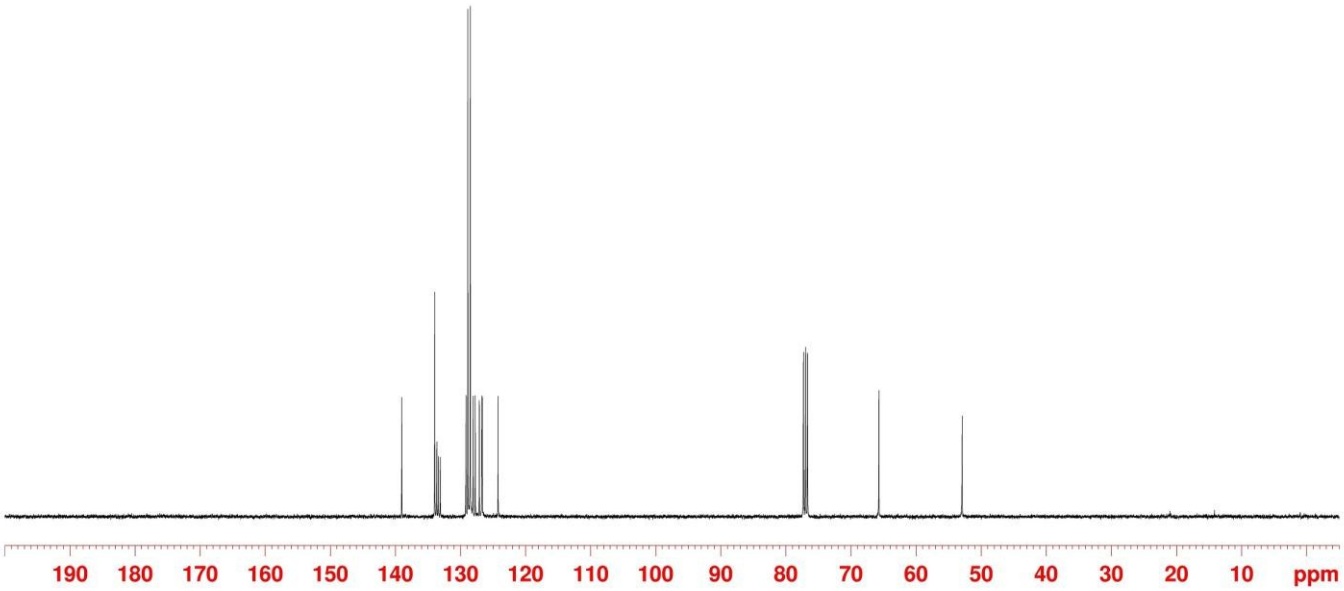


BRUKER

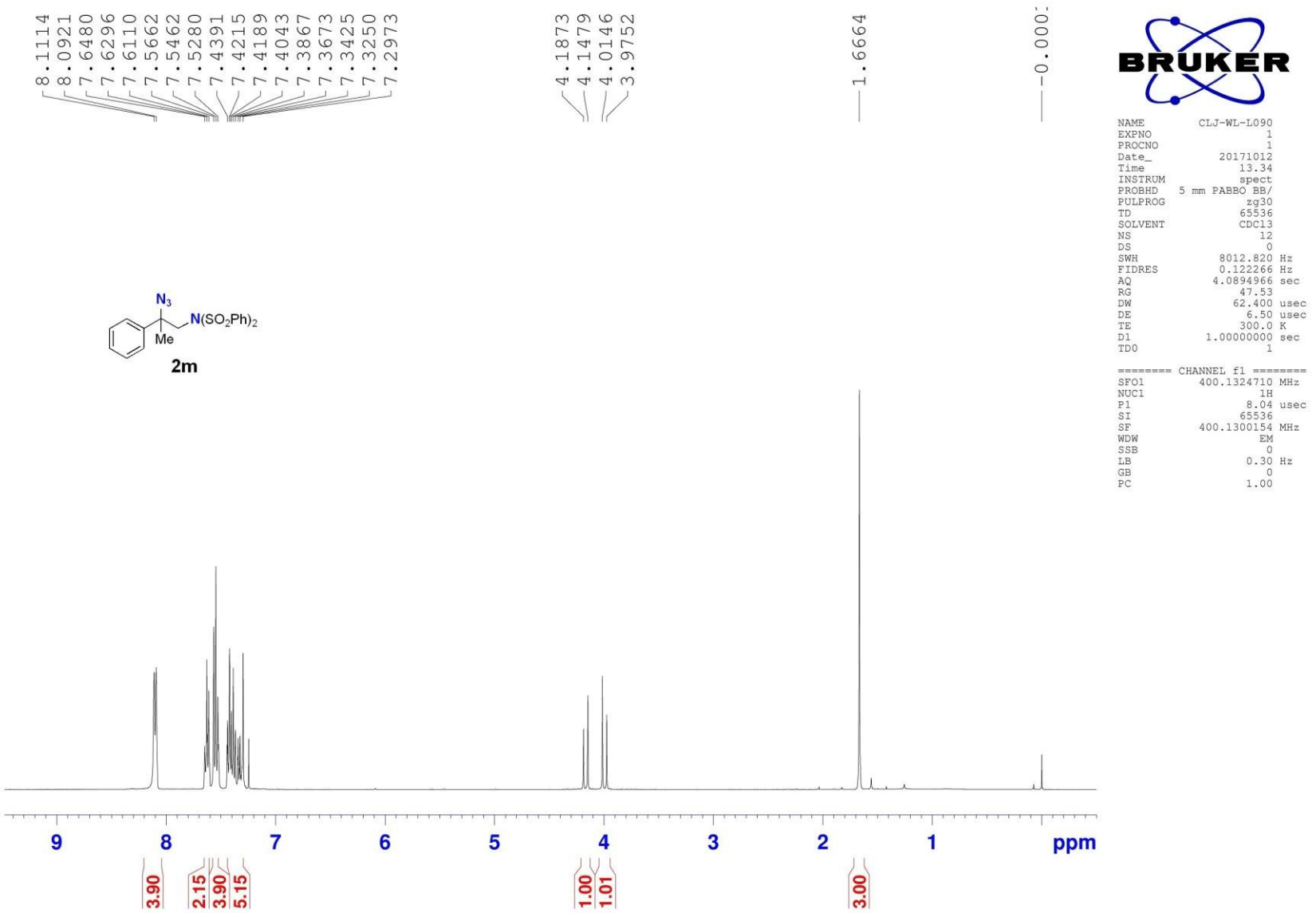
NAME	CLJ-WL-L086
EXPNO	1
PROCNO	1
Date_	20171010
Time	0.04
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	12
DS	0
SWH	8012.820 Hz
FIDRES	0.122266 Hz
AQ	4.0894966 sec
RG	36.4
DW	62.400 usec
DE	6.50 usec
TE	296.8 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
SFO1	400.1324710 MHz
NUC1	1H
P1	8.04 usec
SI	65536
SF	400.1300182 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

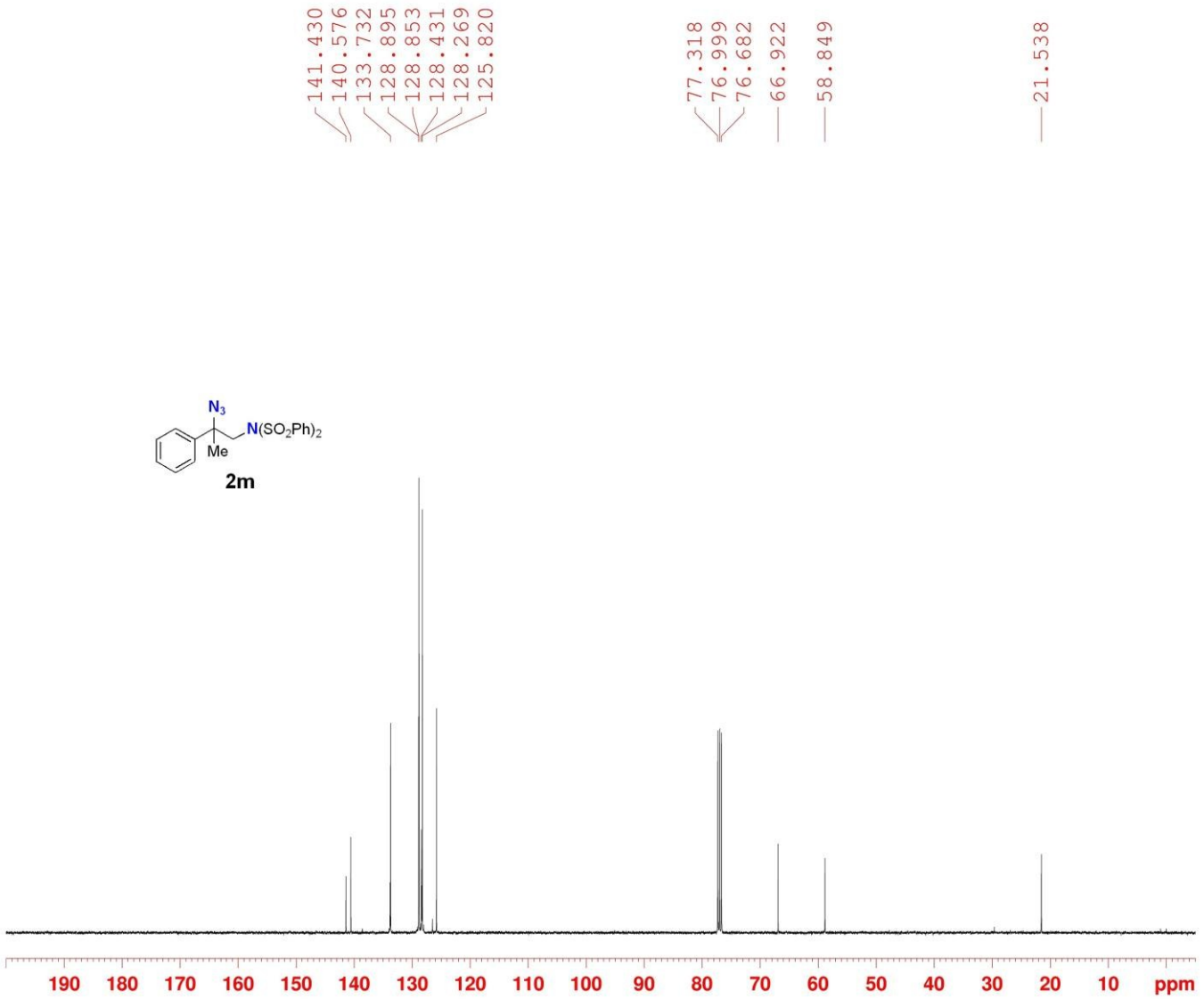


2l



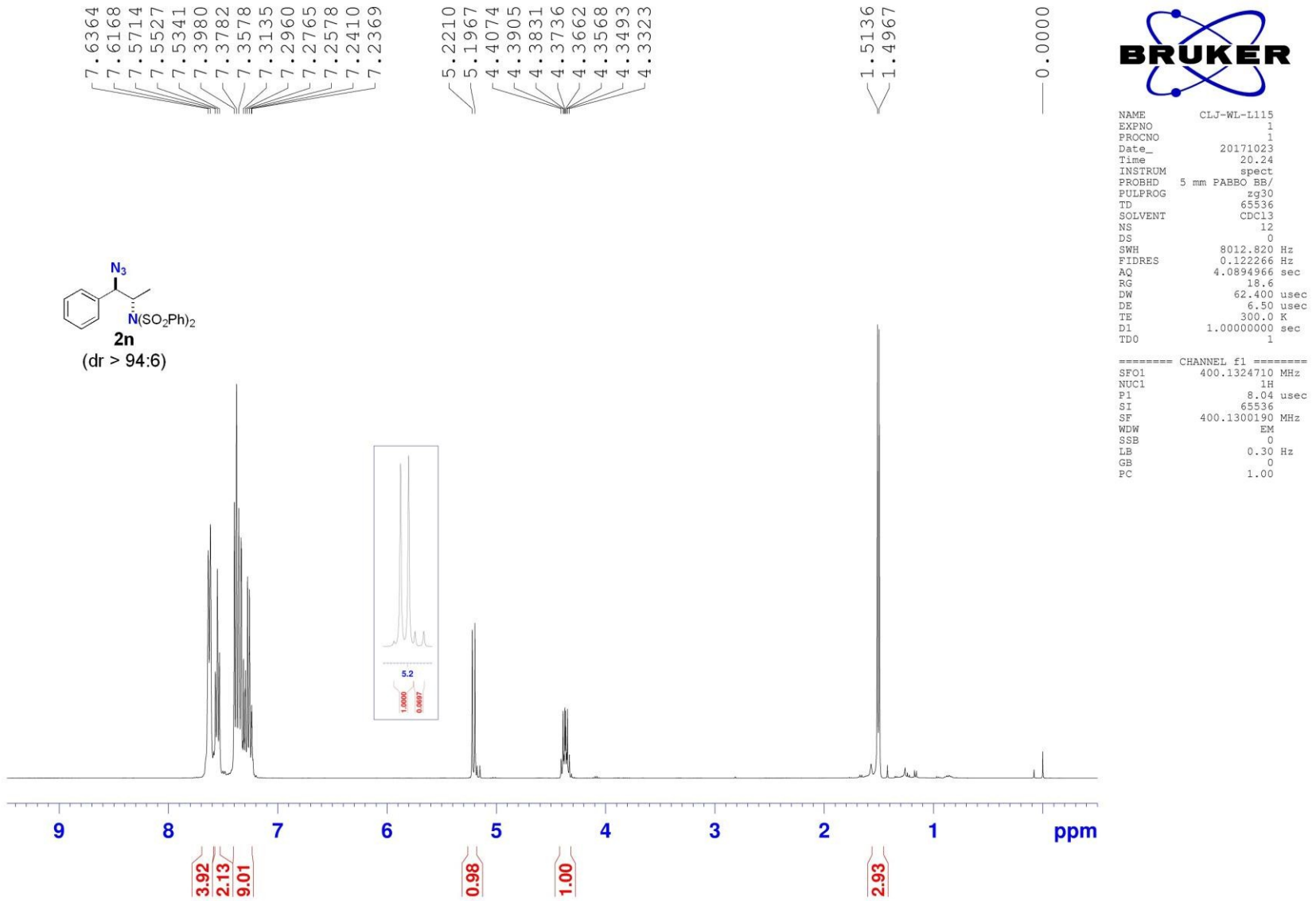
NAME	CLJ-WL-L086
EXPNO	2
PROCNO	1
Date_	20171010
Time	0.35
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	512
DS	0
SWH	24038.461 Hz
FIDRES	0.366798 Hz
AQ	1.3631988 sec
RG	194.26
DW	20.800 usec
DE	6.50 usec
TE	297.1 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
SFO1	100.6228293 MHz
NUC1	13C
P1	8.54 usec
SI	32768
SF	100.6127792 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

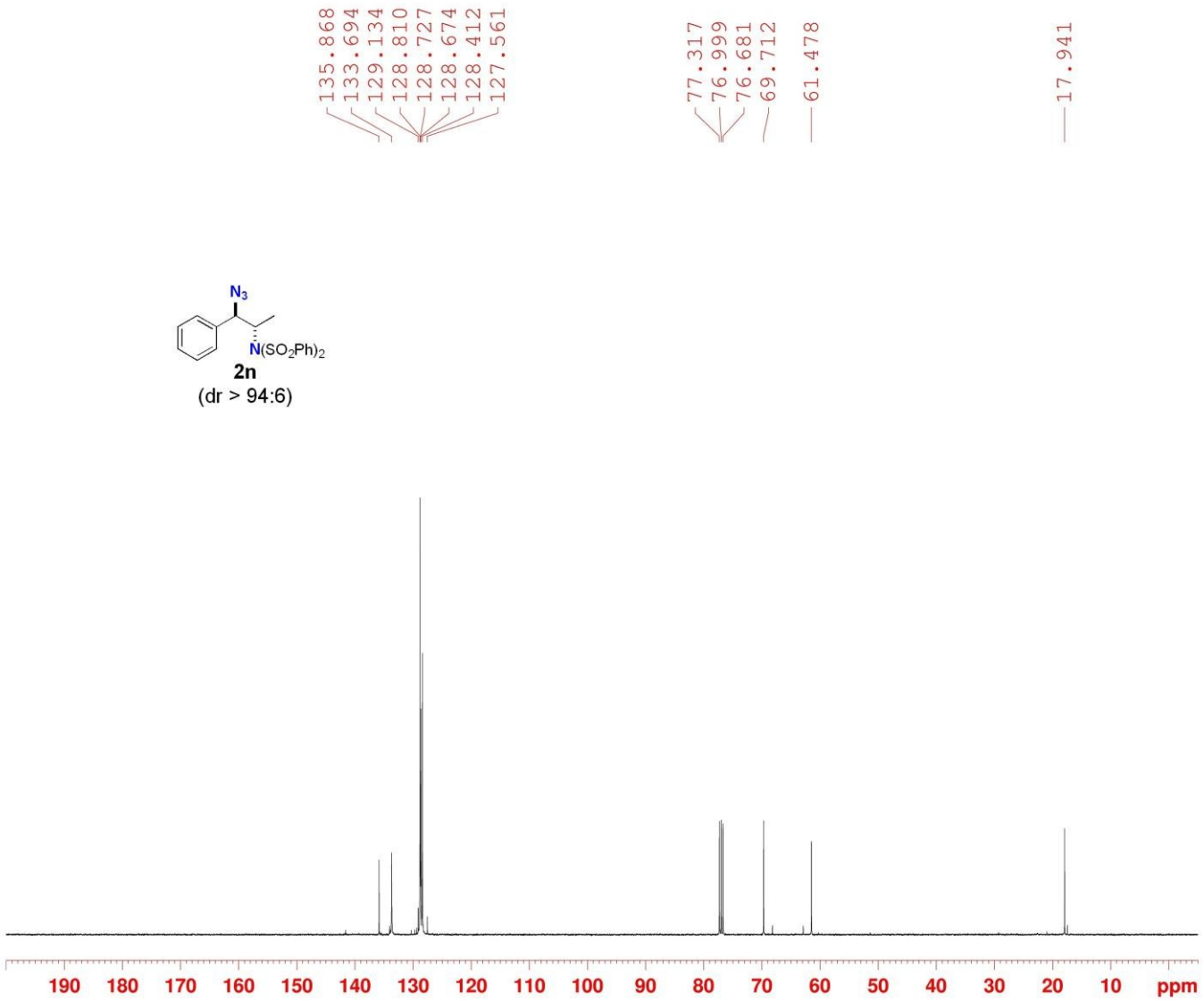




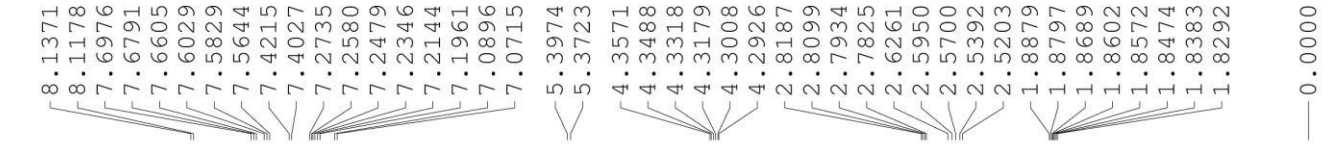
BRUKER

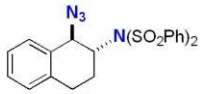
NAME	CLJ-WL-L090
EXPNO	2
PROCNO	1
Date_	20171012
Time	14.04
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	512
DS	0
SWH	24038.461 Hz
FIDRES	0.366798 Hz
AQ	1.3631988 sec
RG	194.26
DW	20.800 usec
DE	6.50 usec
TE	300.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
SFO1	100.6228293 MHz
NUC1	13C
P1	8.54 usec
SI	32768
SF	100.6127748 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40





NAME	CLJ-WL-L115
EXPNO	2
PROCNO	1
Date_	20171023
Time	20.54
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	512
DS	0
SWH	24038.461 Hz
FIDRES	0.366798 Hz
AQ	1.3631988 sec
RG	194.26
DW	20.800 usec
DE	6.50 usec
TE	300.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
SFO1	100.6228293 MHz
NUC1	13C
P1	8.54 usec
SI	32768
SF	100.6127801 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40





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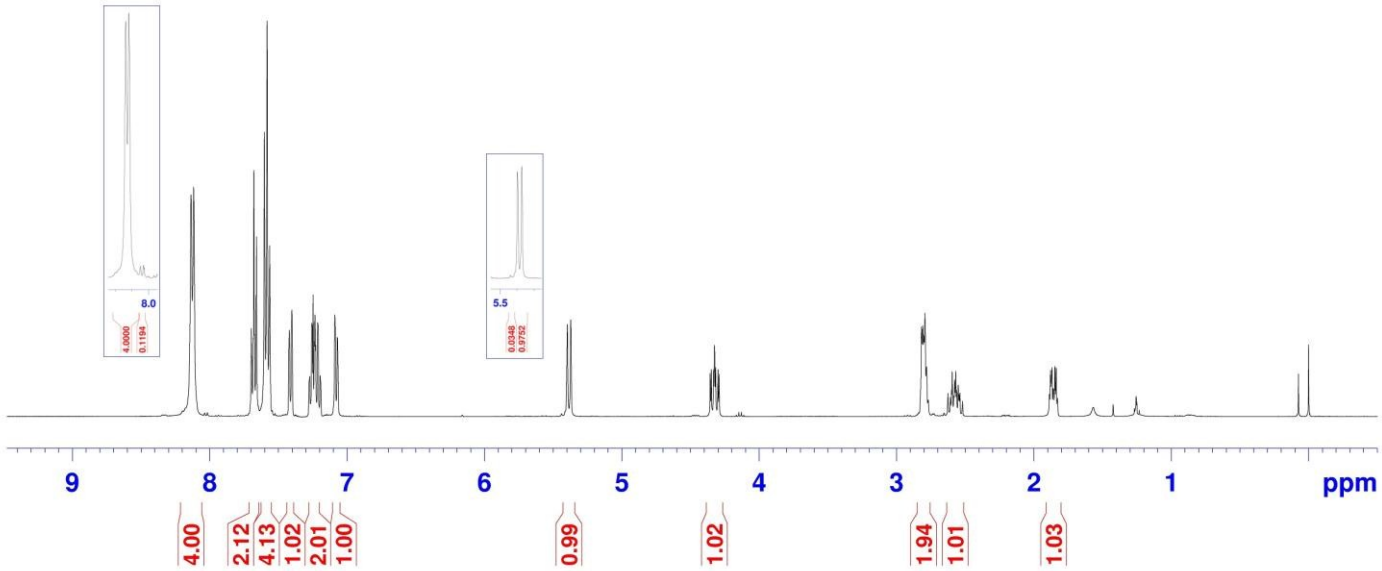
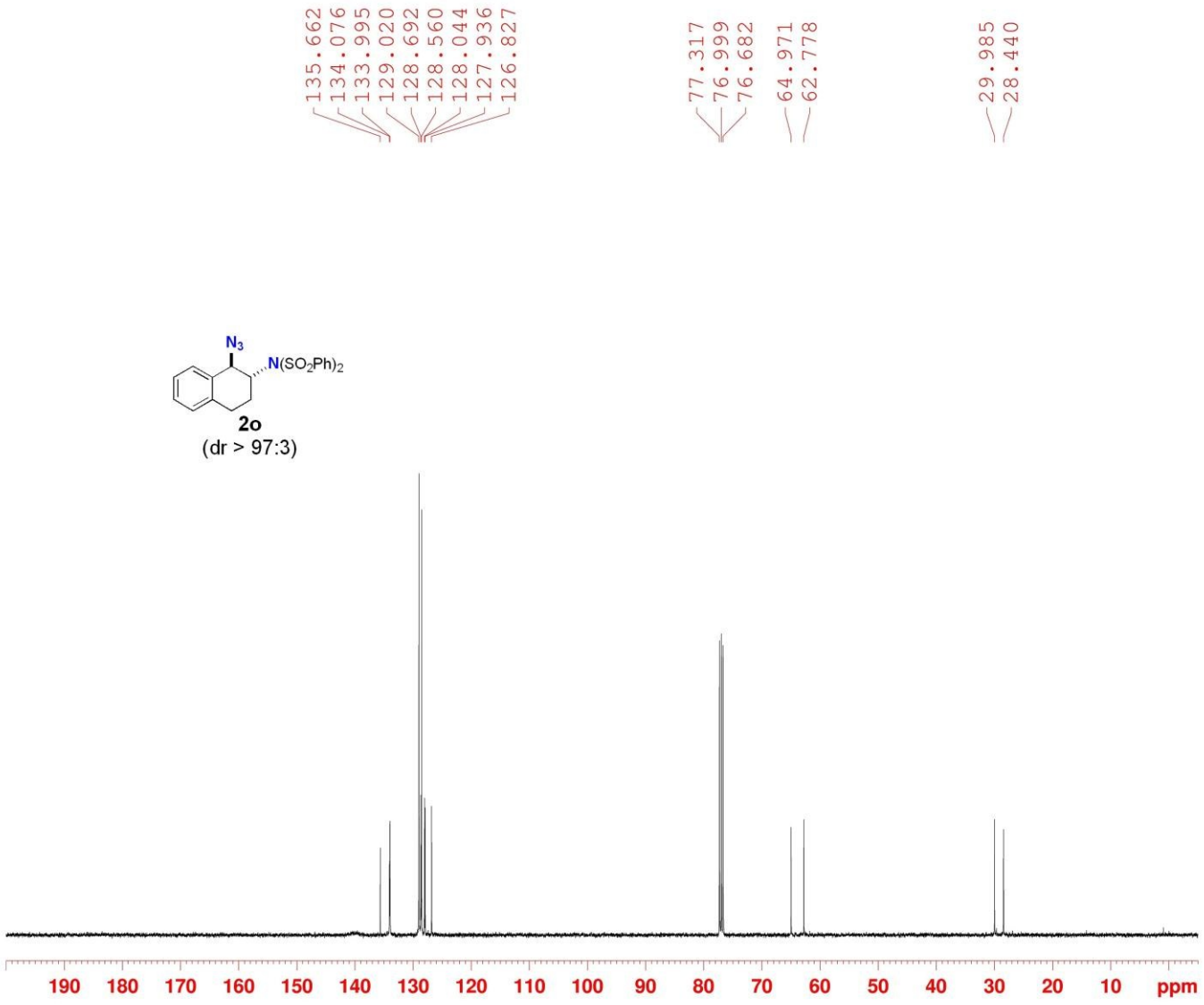
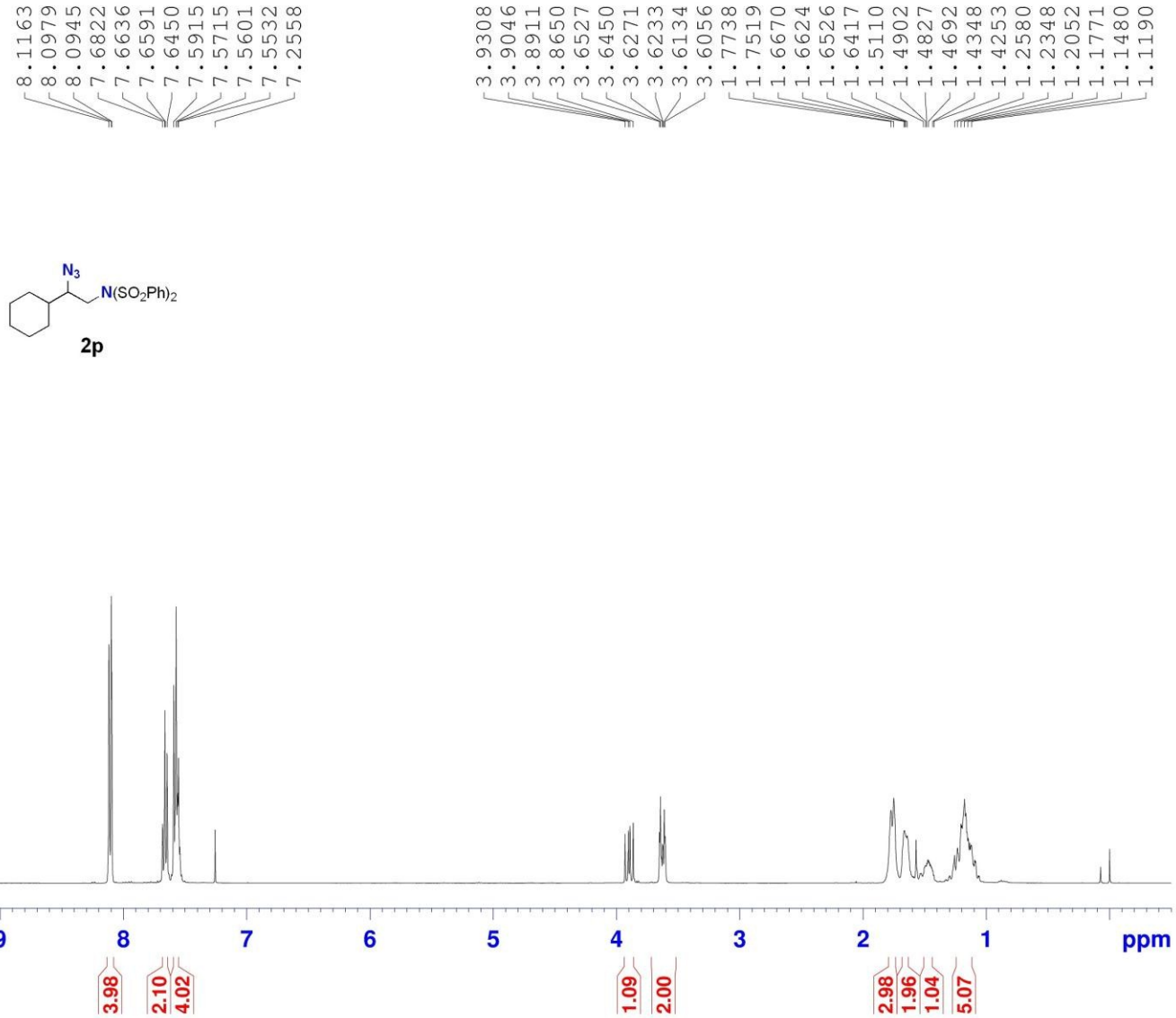


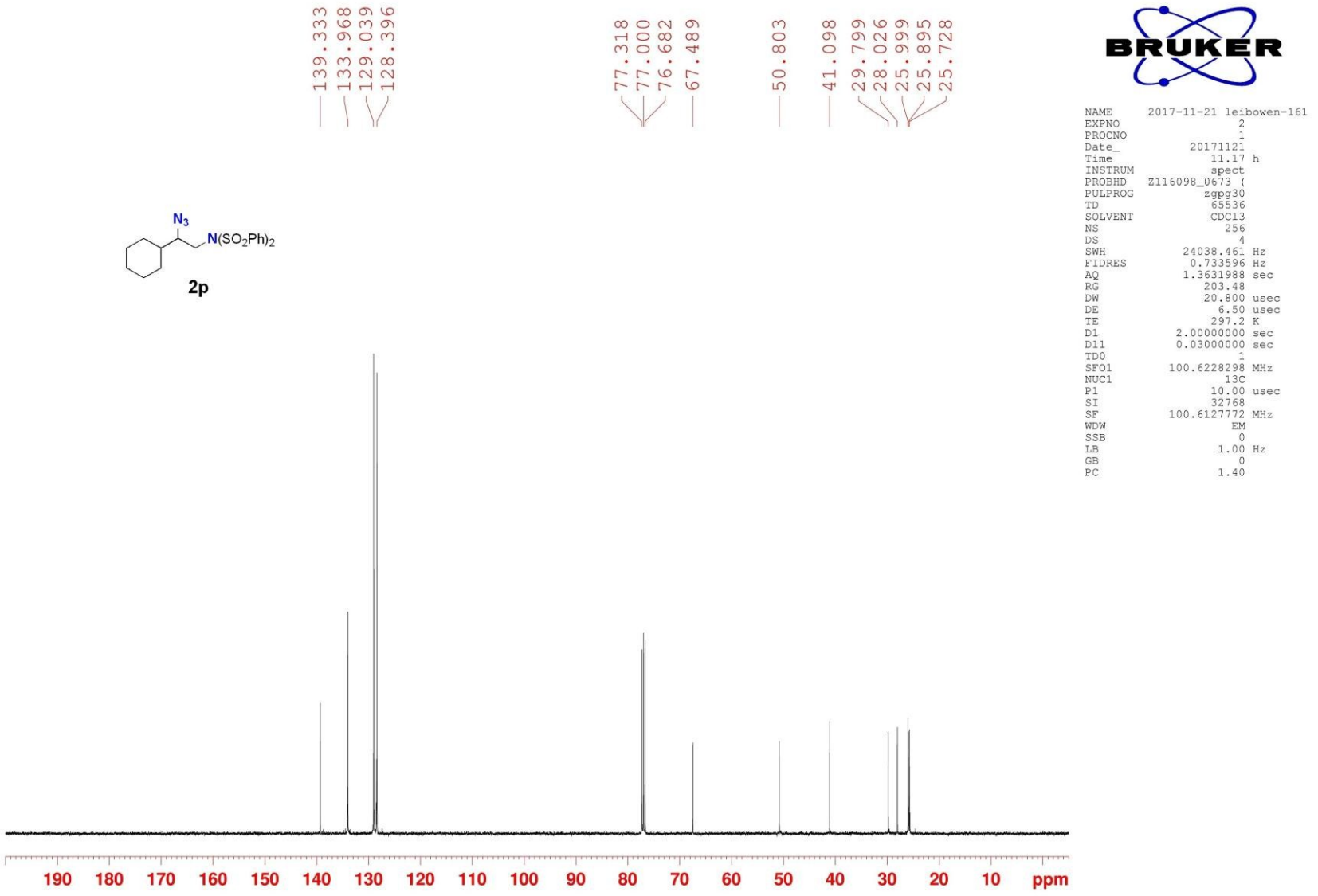

```
NAME            CLJ-WL-L097
EXPNO                     1
PROCNO                    1
Date_              20171018
Time                   2.59
INSTRUM               spect
PROBHD    5 mm PABBO BB/
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       12
DS                        0
SWH                8012.820 Hz
FIDRES             0.122266 Hz
AQ                 4.0894966 sec
RG                    51.19
DW                    62.400 usec
DE                     6.50 usec
TE                    300.0 K
D1                 1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
SFO1           400.1324710 MHz
NUC1                      1H
P1                     8.04 usec
SI                    65536
SF             400.1300146 MHz
WDW                      EM
SSB                       0
LB                     0.30 Hz
GB                        0
PC                     1.00
```

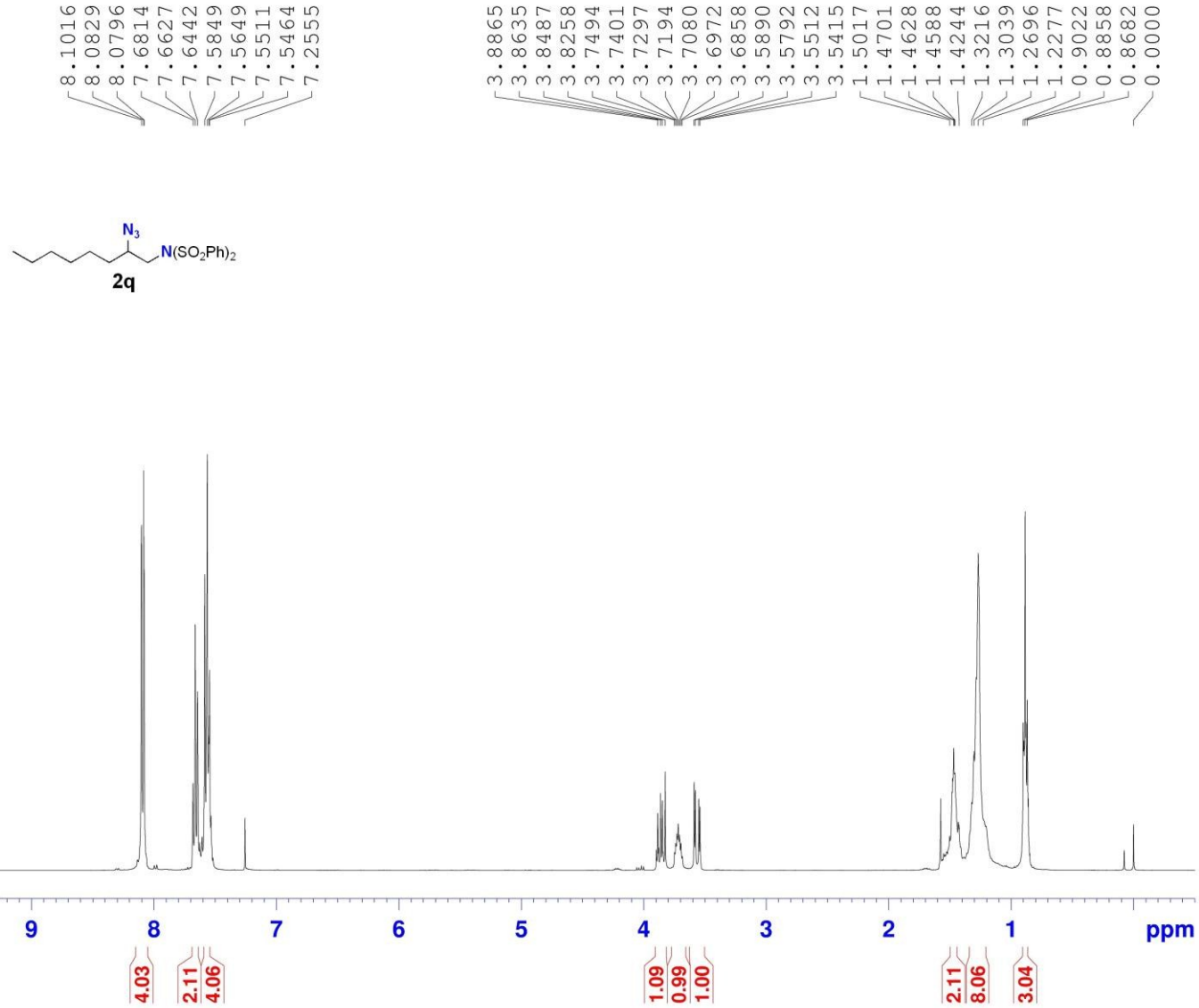


NAME	CLJ-WL-L097
EXPNO	2
PROCNO	1
Date_	20171018
Time	3.29
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	512
DS	0
SWH	24038.461 Hz
FIDRES	0.366798 Hz
AQ	1.3631988 sec
RG	194.26
DW	20.800 usec
DE	6.50 usec
TE	300.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
SFO1	100.6228293 MHz
NUC1	13C
P1	8.54 usec
SI	32768
SF	100.6127741 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40



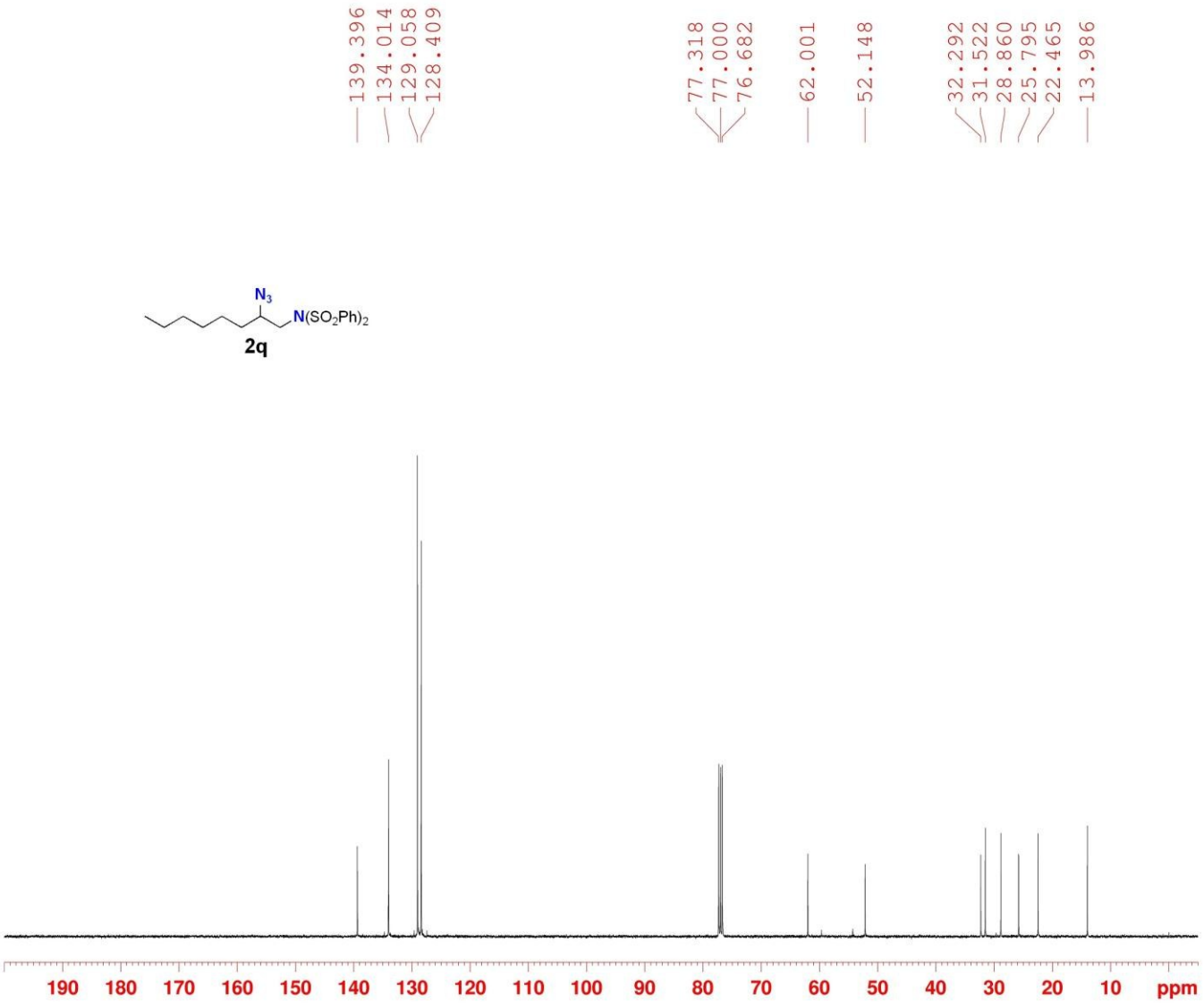


NAME	2017-11-21 leibowen-161
EXPNO	2
PROCNO	1
Date_	20171121
Time	11.17 h
INSTRUM	spect
PROBHD	Z116098_0673 (
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	256
DS	4
SWH	24038.461 Hz
FIDRES	0.733596 Hz
AQ	1.3631988 sec
RG	203.48
DW	20.800 usec
DE	6.50 usec
TE	297.2 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
SFO1	100.6228298 MHz
NUC1	13C
P1	10.00 usec
SI	32768
SF	100.6127772 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40



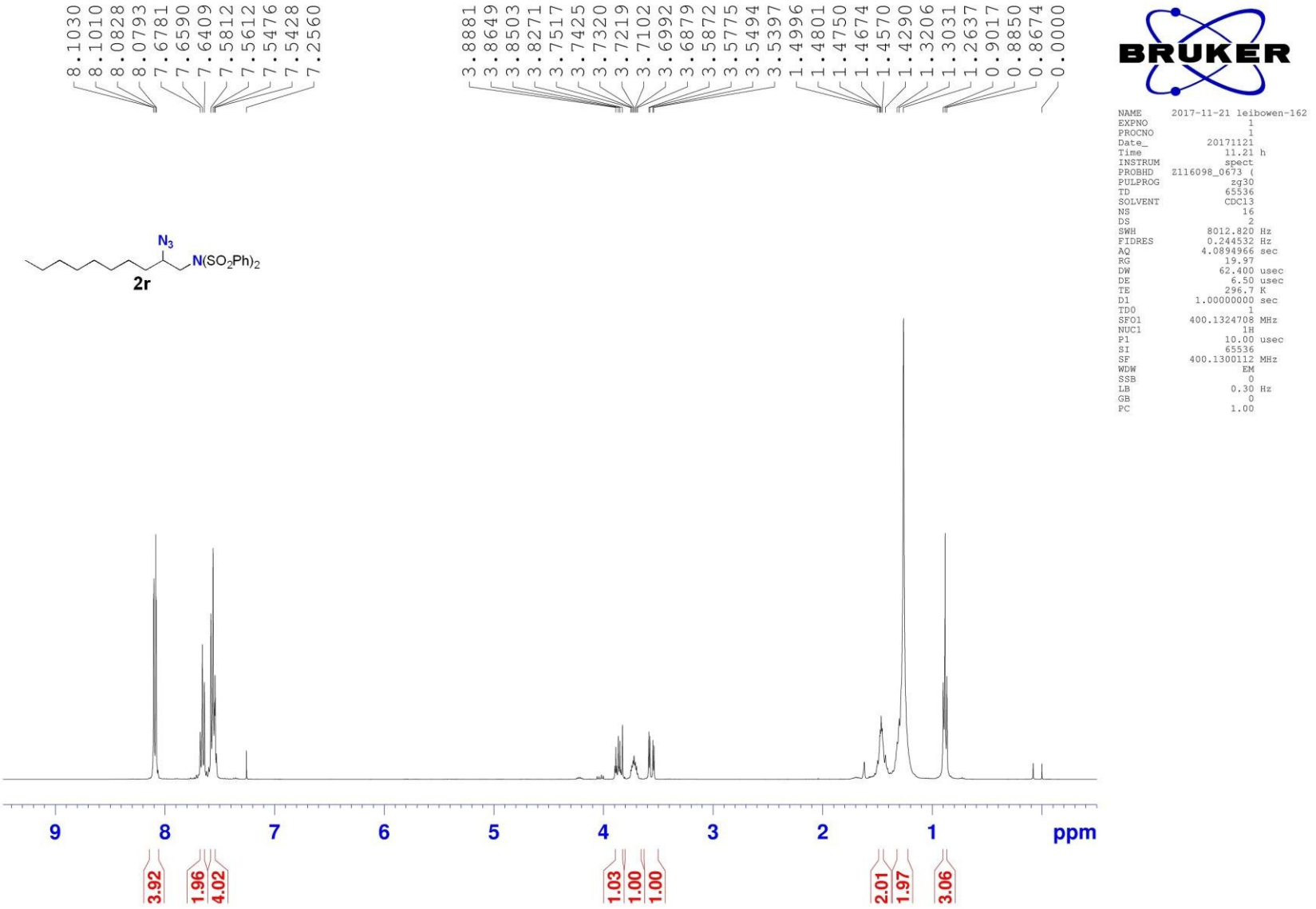
BRUKER

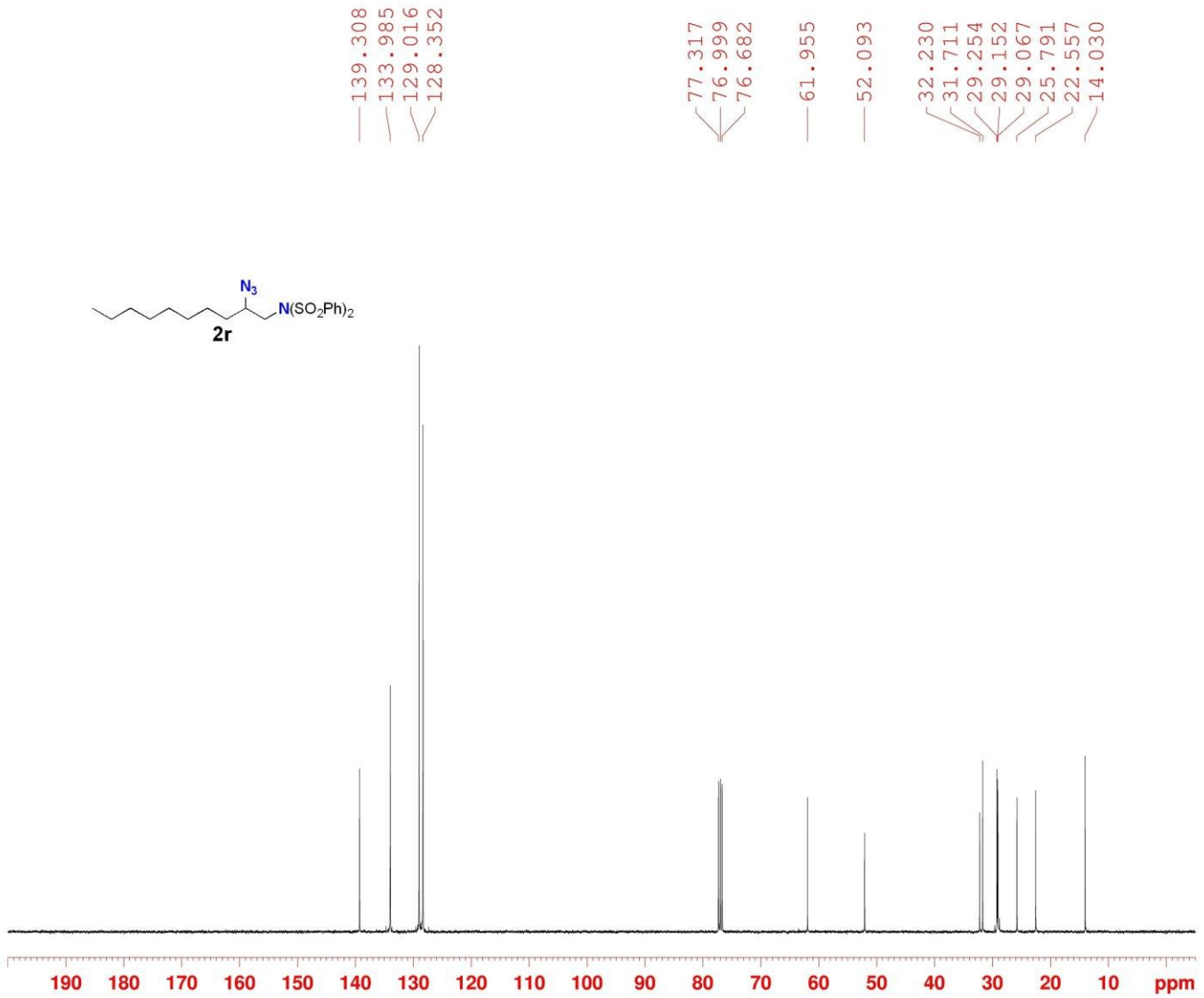
NAME	CLJ-WL-L149
EXPNO	1
PROCNO	1
Date_	20171110
Time	18.55
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	64
DS	0
SWH	8012.820 Hz
FIDRES	0.122266 Hz
AQ	4.0894966 sec
RG	21.11
DW	62.400 usec
DE	6.50 usec
TE	300.0 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
SFO1	400.1324710 MHz
NUC1	1H
P1	8.04 usec
SI	65536
SF	400.1300114 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



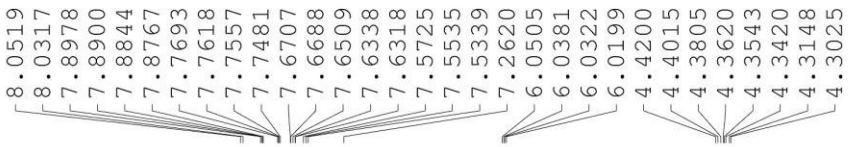
BRUKER

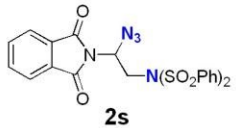
NAME	CLJ-WL-L134
EXPNO	2
PROCNO	1
Date_	20171106
Time	3.20
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	512
DS	0
SWH	24038.461 Hz
FIDRES	0.366798 Hz
AQ	1.3631988 sec
RG	194.26
DW	20.800 usec
DE	6.50 usec
TE	299.1 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
SFO1	100.6228293 MHz
NUC1	13C
P1	8.54 usec
SI	32768
SF	100.6127742 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40





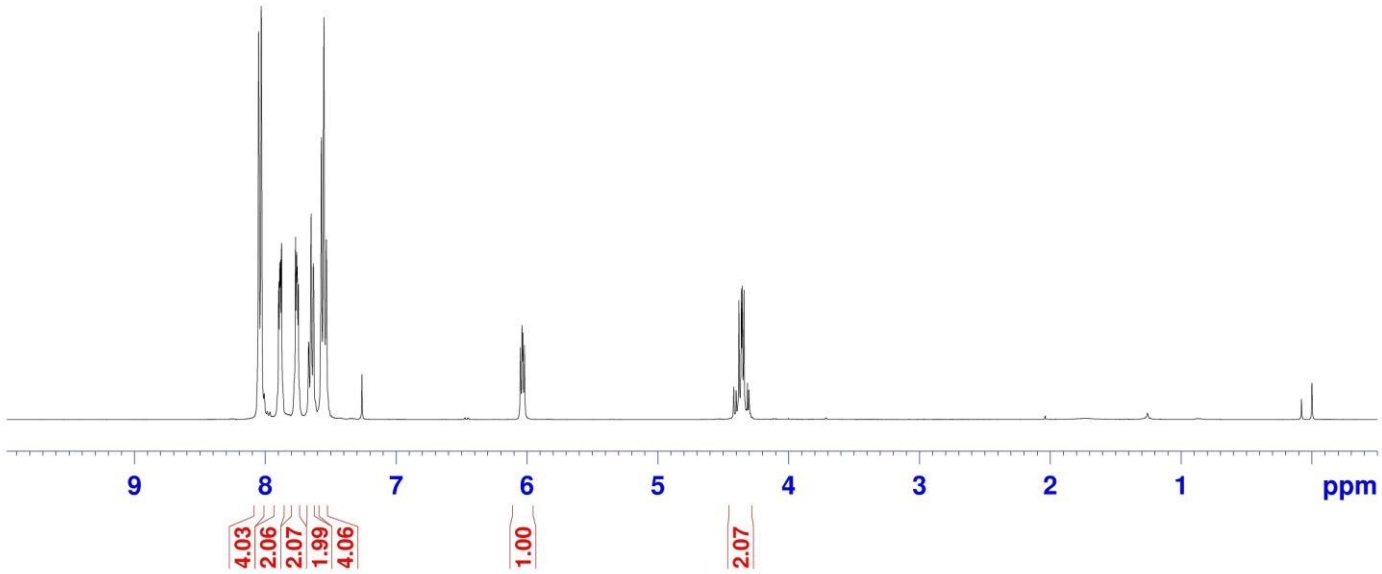
NAME	2017-11-21 leibowen-162
EXPNO	2
PROCNO	1
Date_	20171121
Time	11.37 h
INSTRUM	spect
PROBHD	Z116098_0673 (
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	256
DS	4
SWH	24038.461 Hz
FIDRES	0.733596 Hz
AQ	1.3631988 sec
RG	203.48
DW	20.800 usec
DE	6.50 usec
TE	297.2 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
SFO1	100.6228298 MHz
NUC1	13C
P1	10.00 usec
SI	32768
SF	100.6127798 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

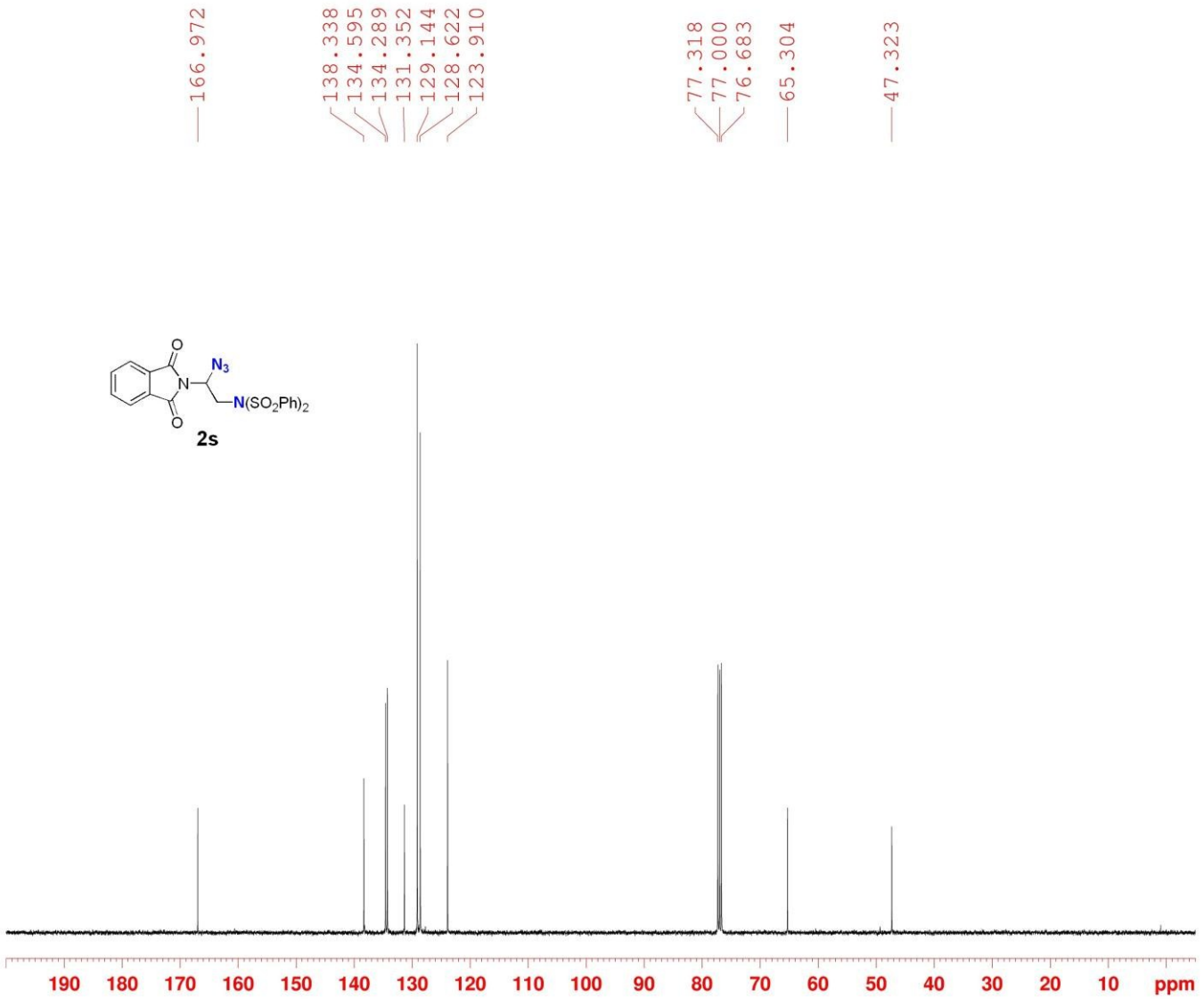




2s

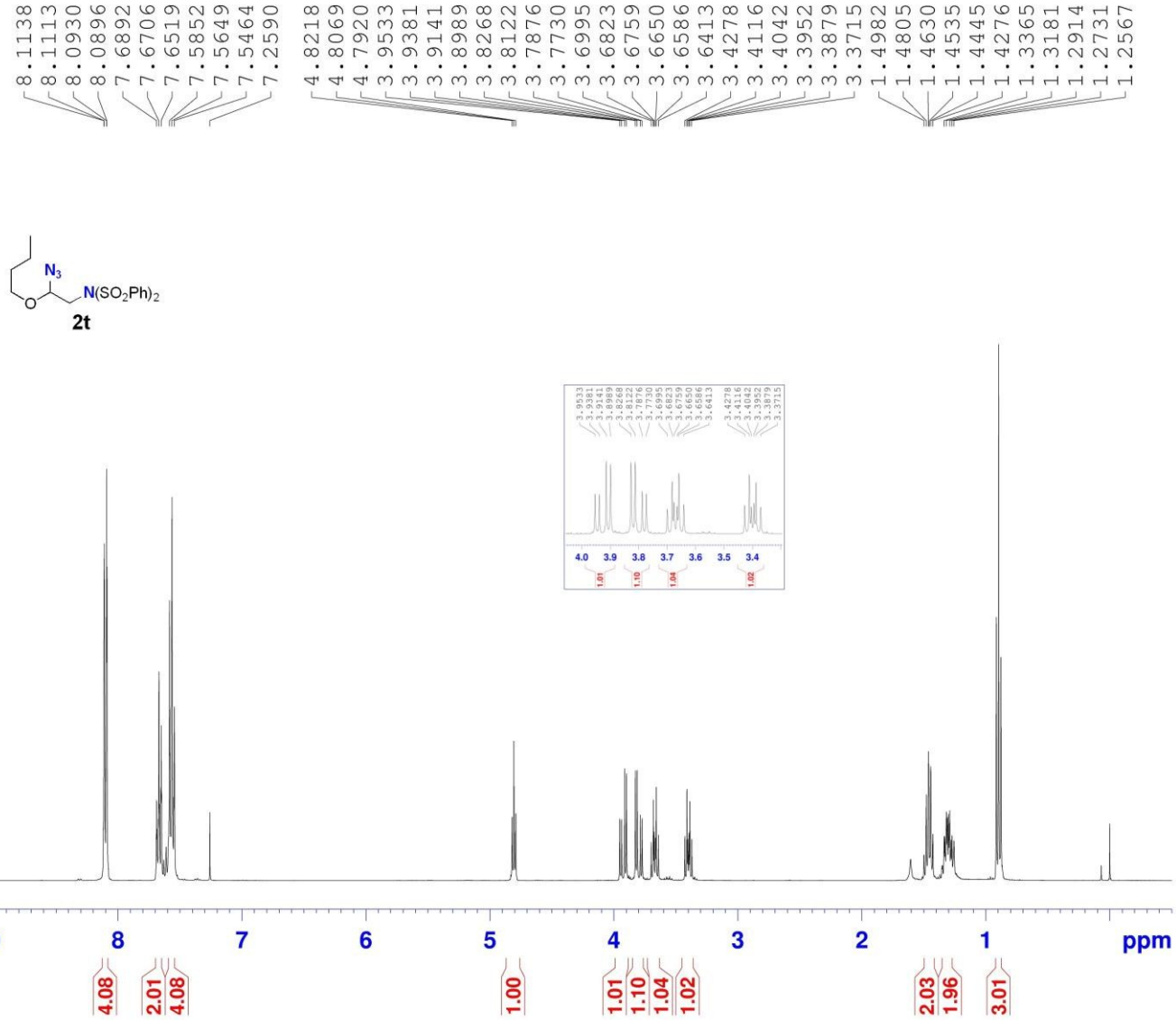
NAME	2017-11-21 leibowen-160
EXPNO	1
PROCNO	1
Date_	20171121
Time	10.41 h
INSTRUM	spect
PROBHD	Z116098_0673 (
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	8012.820 Hz
FIDRES	0.244532 Hz
AQ	4.0894966 sec
RG	51.13
DW	62.400 usec
DE	6.50 usec
TE	296.6 K
D1	1.00000000 sec
TD0	1
SFO1	400.1324708 MHz
NUC1	1H
P1	10.00 usec
SI	65536
SF	400.1300087 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



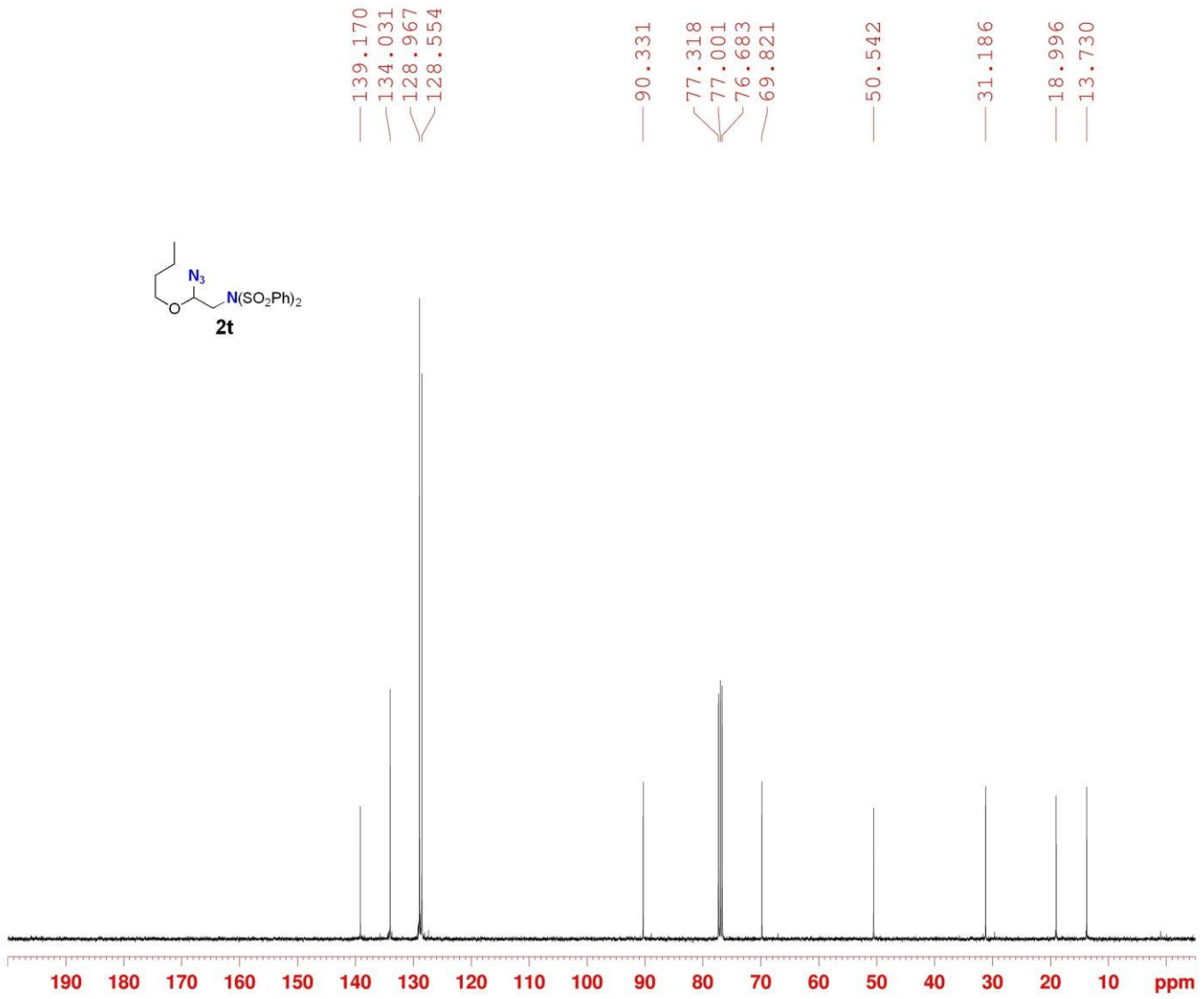


BRUKER

NAME	2017-11-21 leibowen-160
EXPNO	2
PROCNO	1
Date_	20171121
Time	10.57 h
INSTRUM	spect
PROBHD	Z116098_0673 (
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	256
DS	4
SWH	24038.461 Hz
FIDRES	0.733596 Hz
AQ	1.3631988 sec
RG	203.48
DW	20.800 usec
DE	6.50 usec
TE	297.1 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
SFO1	100.6228298 MHz
NUC1	13C
P1	10.00 usec
SI	32768
SF	100.6127776 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

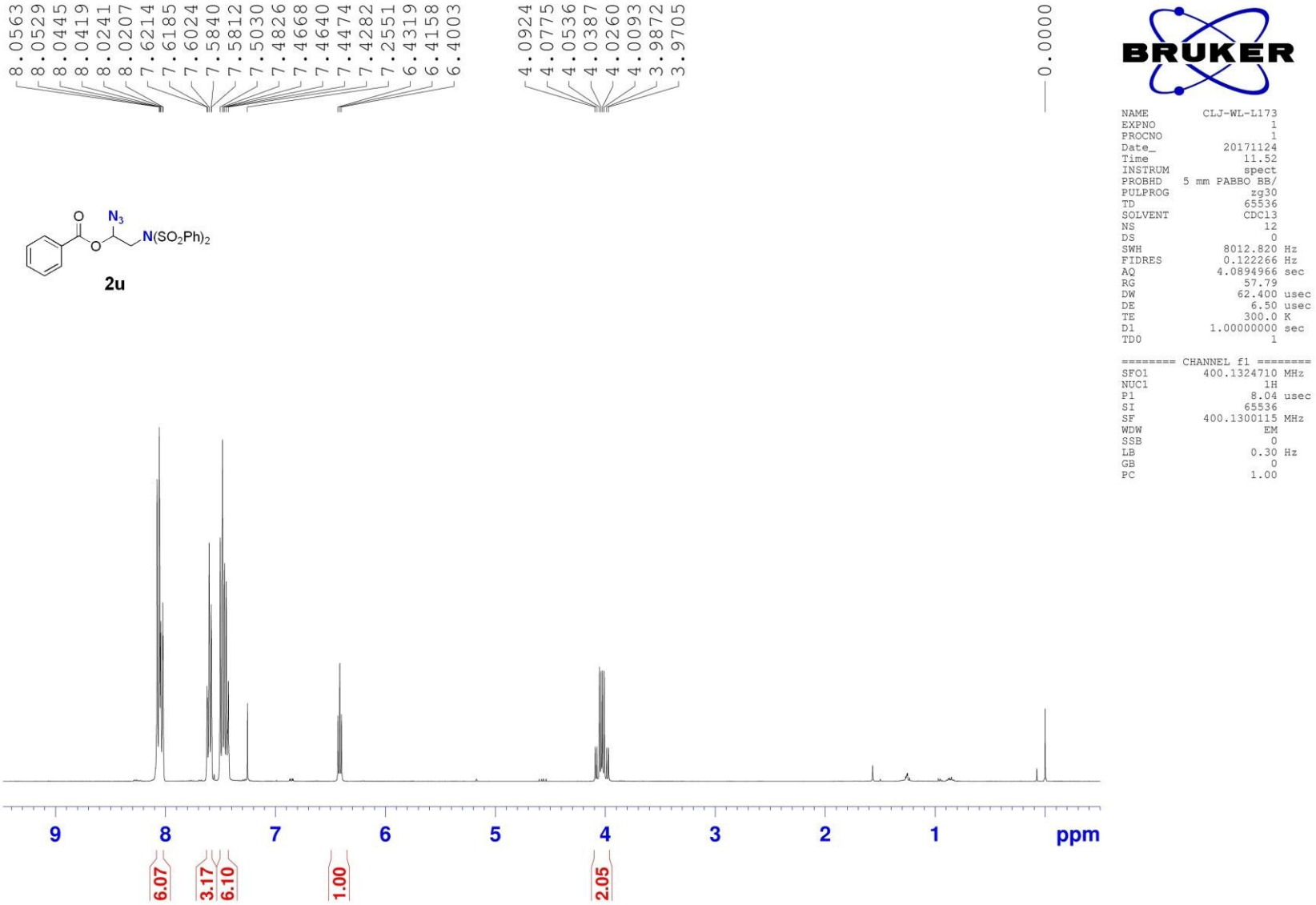


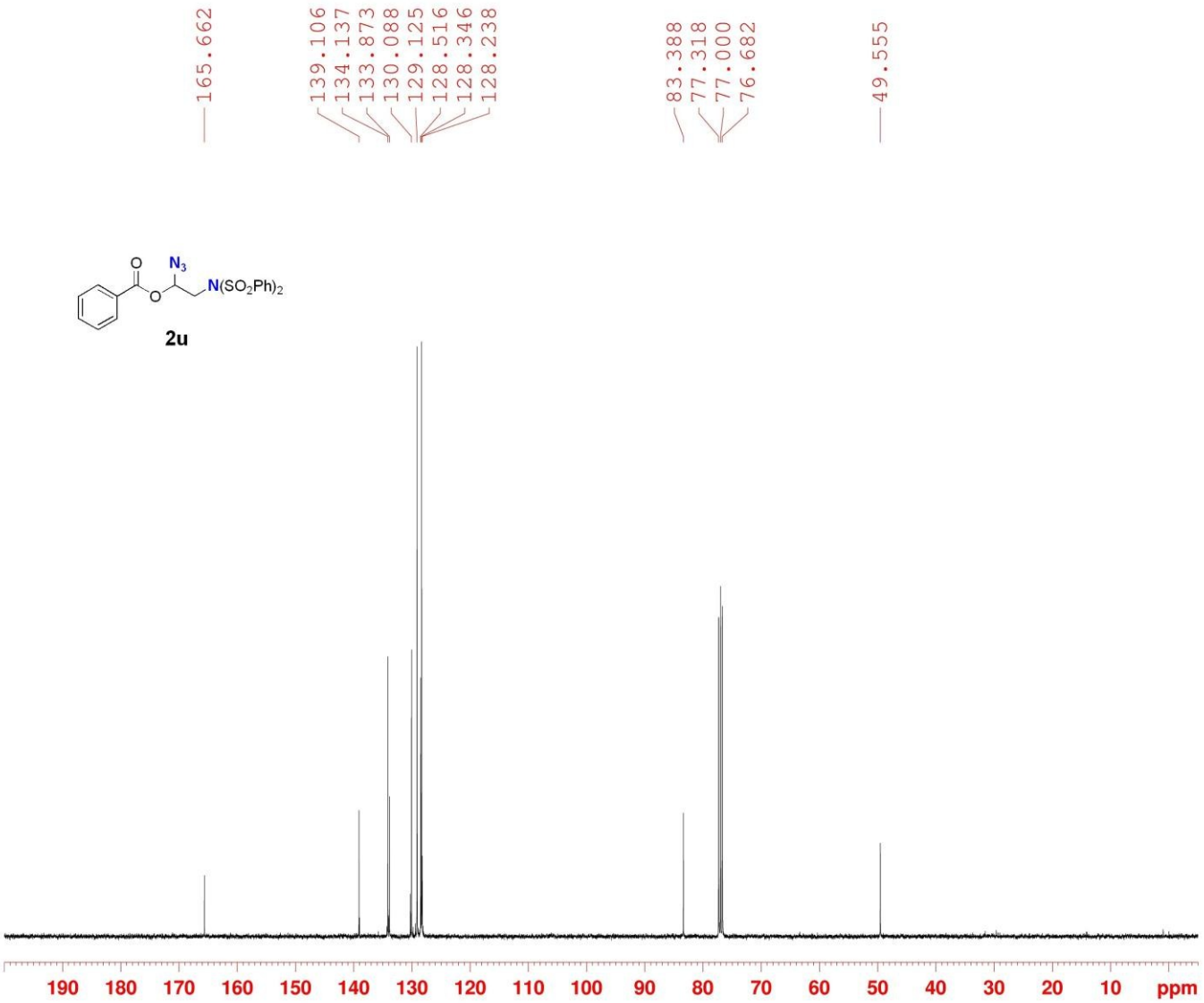
NAME 2017-11-21 leibowen-159
EXPNO 1
PROCNO 1
Date_ 20171121
Time 10.20 h
INSTRUM spect
PROBHD Z116098_0673 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 4.0894966 sec
RG 31.12
DW 62.400 usec
DE 6.50 usec
TE 296.4 K
D1 1.00000000 sec
TD0 1
SFO1 400.1324708 MHz
NUC1 1H
P1 10.00 usec
SI 65536
SF 400.1300101 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



BRUKER

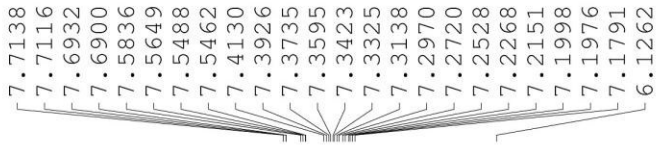
NAME	2017-11-21 leibowen-159
EXPNO	2
PROCNO	1
Date_	20171121
Time	10.36 h
INSTRUM	spect
PROBHD	Z116098_0673 (
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	256
DS	4
SWH	24038.461 Hz
FIDRES	0.733596 Hz
AQ	1.3631988 sec
RG	203.48
DW	20.800 usec
DE	6.50 usec
TE	297.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
SFO1	100.6228298 MHz
NUC1	13C
P1	10.00 usec
SI	32768
SF	100.6127763 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

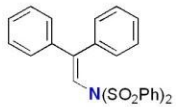




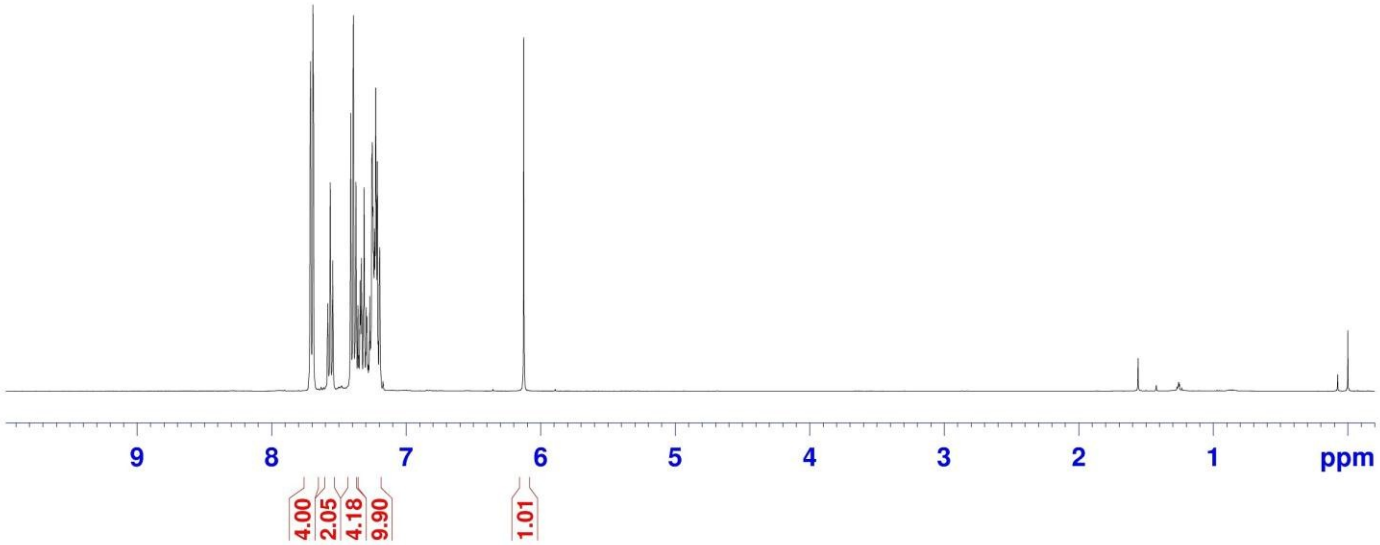
BRUKER

NAME	CLJ-WL-L173
EXPNO	2
PROCNO	1
Date_	20171124
Time	12.22
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	512
DS	0
SWH	24038.461 Hz
FIDRES	0.366798 Hz
AQ	1.3631988 sec
RG	194.26
DW	20.800 usec
DE	6.50 usec
TE	300.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
SFO1	100.6228293 MHz
NUC1	13C
P1	8.54 usec
SI	32768
SF	100.6127731 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40



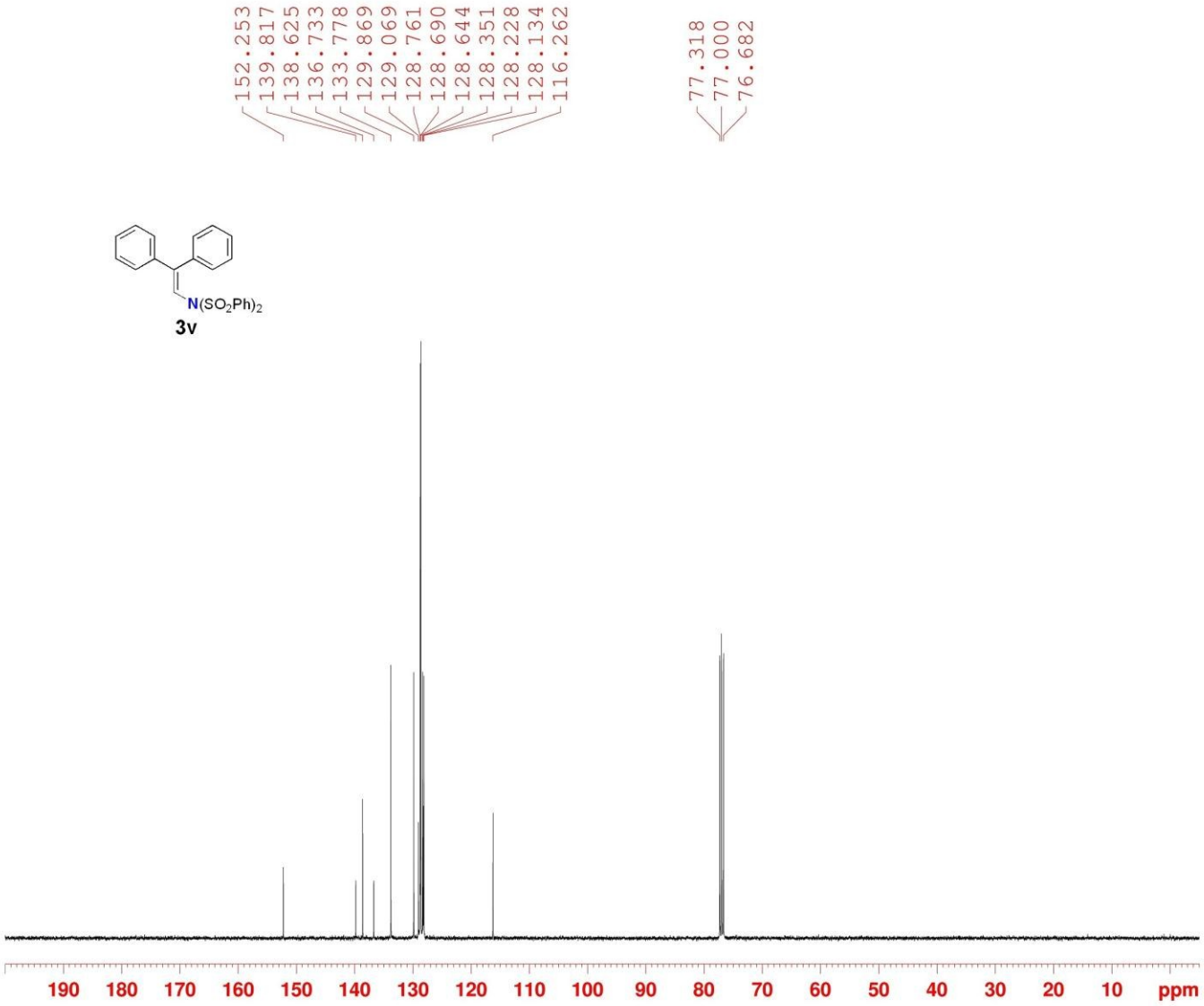


3v



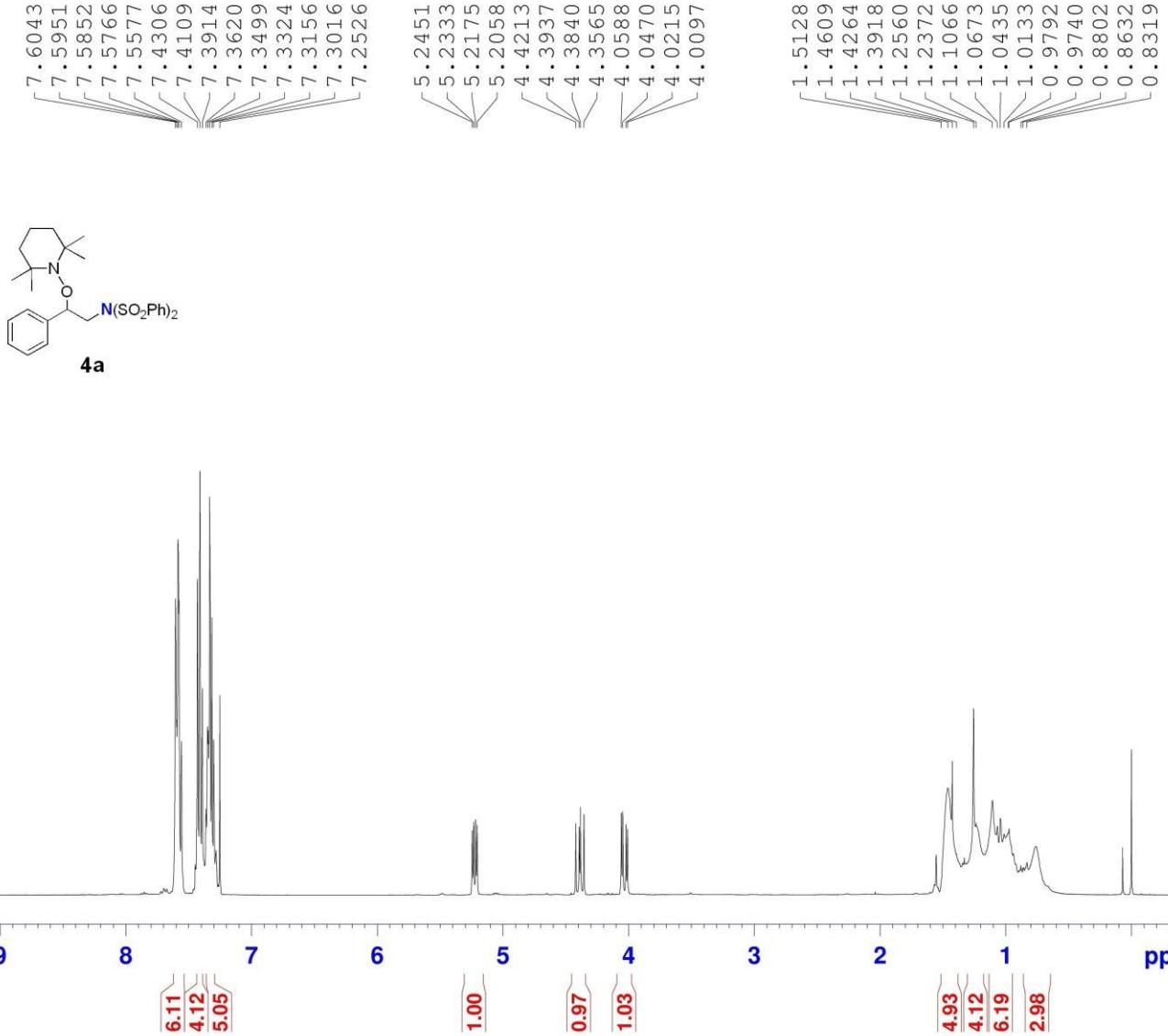

```
NAME          CLJ-WL-L088
EXPNO                   1
PROCNO                  1
Date_            20171014
Time                3.03
INSTRUM            spect
PROBHD    5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     12
DS                      0
SWH             8012.820 Hz
FIDRES          0.122266 Hz
AQ              4.0894966 sec
RG                 51.19
DW                62.400 usec
DE                 6.50 usec
TE                300.0 K
D1             1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1         400.1324710 MHz
NUC1                   1H
P1                  8.04 usec
SI                 65536
SF           400.1300153 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



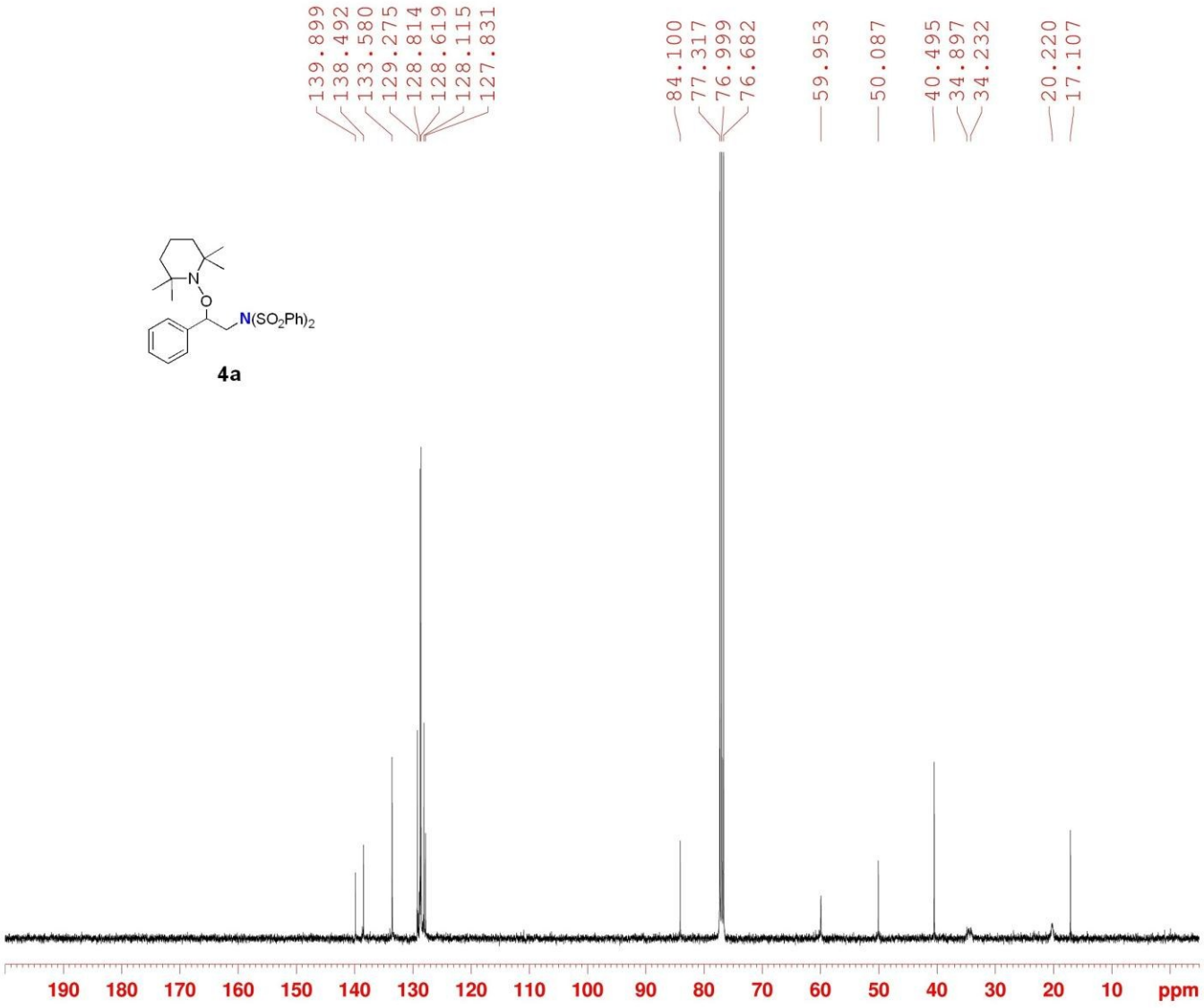
BRUKER

NAME	CLJ-WL-L088
EXPNO	2
PROCNO	1
Date_	20171014
Time	3.33
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	512
DS	0
SWH	24038.461 Hz
FIDRES	0.366798 Hz
AQ	1.3631988 sec
RG	194.26
DW	20.800 usec
DE	6.50 usec
TE	300.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
SFO1	100.6228293 MHz
NUC1	13C
P1	8.54 usec
SI	32768
SF	100.6127739 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40




```
NAME          CLJ-WL-L113
EXPNO                   1
PROCNO                  1
Date_            20171023
Time                21.56
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     12
DS                      0
SWH           8012.820 Hz
FIDRES        0.122266 Hz
AQ            4.0894966 sec
RG                  57.79
DW             62.400 usec
DE              6.50 usec
TE                300.0 K
D1          1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1        400.1324710 MHz
NUC1                   1H
P1                  8.04 usec
SI                  65536
SF          400.1300127 MHz
WDW                    EM
SSB                     0
LB              0.30 Hz
GB                      0
PC                   1.00
```



BRUKER

NAME	CLJ-WL-L133
EXPNO	2
PROCNO	1
Date_	20171106
Time	2.46
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	512
DS	0
SWH	24038.461 Hz
FIDRES	0.366798 Hz
AQ	1.3631988 sec
RG	194.26
DW	20.800 usec
DE	6.50 usec
TE	299.1 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
SFO1	100.6228293 MHz
NUC1	13C
P1	8.54 usec
SI	32768
SF	100.6127771 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40