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Electronic Supplementary Information

for

The Ortho Effect in Directed C-H Activation

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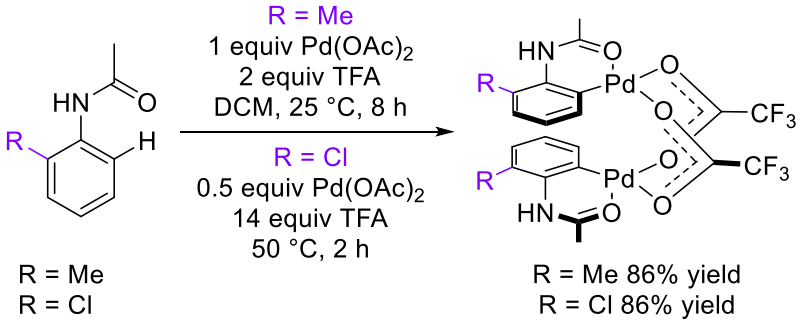
Content

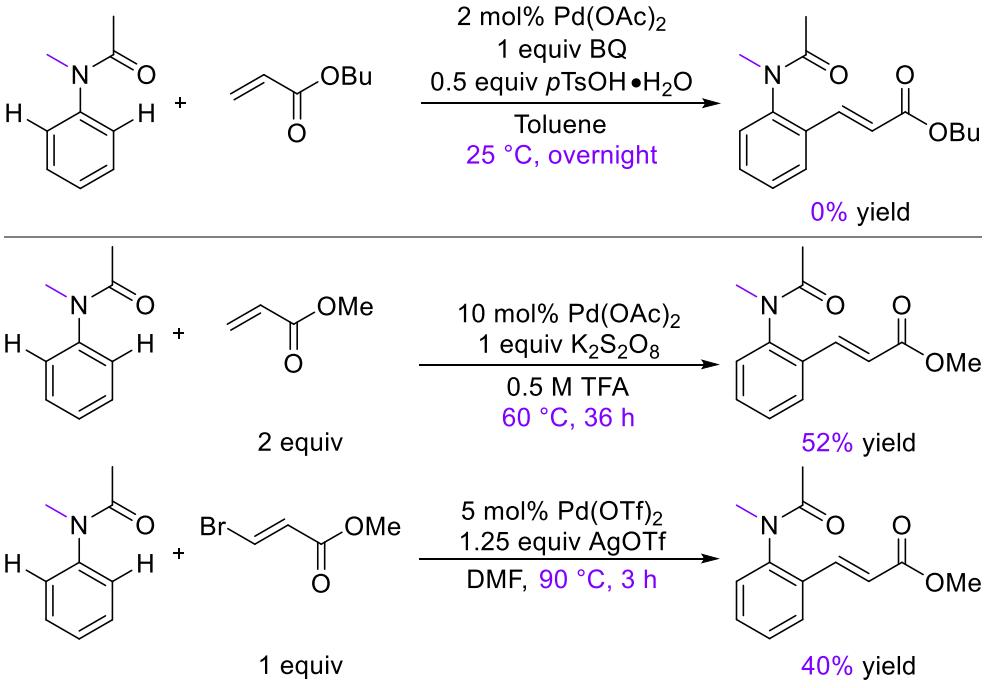
Examples for "Ortho Effect" from Literature

The cited references of this chapter displayed only in the main article.



SI Scheme 1. Palladation of 2-methyl, 2-chloro acetanilide.

As it is described by Horino and coworkers[A7] 2-methyl and 2-chloro acetanilide yielded trifluoroacetate bridged Pd complexes in 86% yield in a palladation reaction in the presence of TFA additive (**Scheme 1**).[A8,A9] The reactions of the *ortho*-substituted anilide substrates were carried out under mild conditions (temperature 25-50 °C) and yielded very stable palladium complexes. This finding reveals the absence of steric effect of the *ortho* substituents of the anilide on this C-H activation reaction. Later on these substrates were successfully functionalized in *ortho* position in various transformations such as alkylation, alkenylation and arylation.[A10]

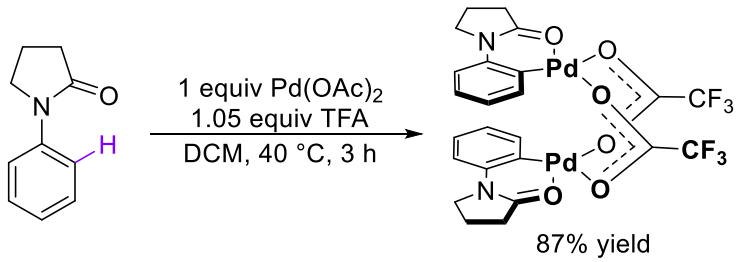


SI Scheme 2. C-H alkenylation reactions of *N*-methyl acetanilide.

As a second example, *N*-methyl acetanilide owns a very important part of this case-study (**Scheme 2**). Analyzing the available literature examples, it can be concluded that the directed palladium catalyzed C-H activation reactions did not take place at 25 °C on this substrate [A11] but at elevated temperature (60-90 °C)[A12,13] the coupling could be completed under the appropriate conditions. Alkynylation of *N*-methyl acetanilides at 70 °C in 15 hours is also reported.[A14] This suggests that the C-H activation of *N*-methyl acetanilide is possible, but requires higher energy than simple acetanilide or 2-methylacetanilide.

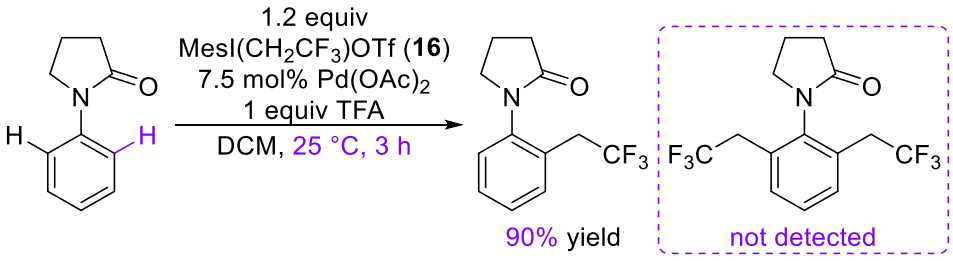
The decreased reactivity of the *N*-methylated substrate could be the consequence of steric properties. However, when any type of ortho substituent is present in the *ortho* position of *N*-methyl acetanilide there are no precedent either for successful palladium catalyzed C-H functionalization or synthesis of Pd complexes, supposedly due to the steric conflict between the *N*-Me group and the *ortho* substituent.

We could observe a similar behavior in case of 1-phenylpyrrolidin-2-one. This substrate could be easily cyclopalladated with $Pd(OAc)_2$ in the presence of trifluoroacetic acid (TFA) in dichloromethane (DCM) solvent at 40 °C, and provide the appropriate stable Pd-complex in 87% yield after 3 hours (**Scheme 3**).[A15]



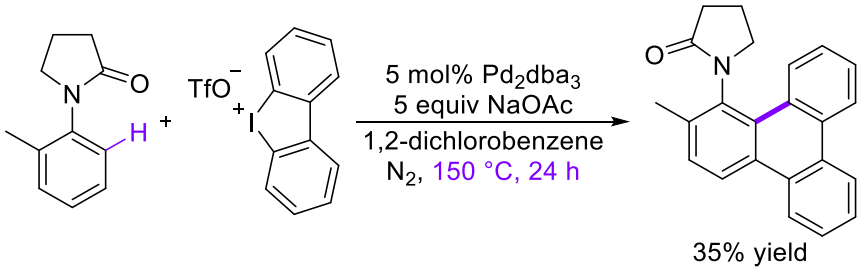
SI Scheme. 3. Palladation reaction of *N*-phenyl pyrrolidine-2-one.

When a catalytic transformation was performed with 7.5 mol% $Pd(OAc)_2$ 1 equivalent TFA, in DCM at 25 °C, the 2,2,2-trifluoroetylation of 1-phenylpyrrolidin-2-one gave the mono-trifluoroethylated product exclusively in 90% yield, whereas the disubstituted bis-trifluoroethylated product was not seen. (**Scheme 4**).[A16] To understand the selectivity we note that after the successful *ortho* C-H alkylation reaction there is a steric clash between the trifluoroethyl group and the pyrrolidinone DG yielding unfavorable conformation for the second *ortho* C-H bond functionalization at 25 °C. The deflection from the ideal coplanar geometry can be also a benefit for a chemical synthesis, because in certain cases, the selective mono-substitution is the required objective of a synthetic sequence.



SI Scheme 4. Mono-selective trifluoroethylation reaction of *N*-phenyl pyrrolidine-2-one.

However, with the investment of thermal energy, C-H activation reaction of 1-(*o*-tolyl)pyrrolidin-2-one could be achieved. In this example the *ortho* substituted substrate were arylated using 5 mol% of Pd_2dba_3, superstoichiometric NaOAc and 1,2-dichlorobenzene at 150 °C for 24 hours and yielded the desired compound in 35% (**Scheme 5**).[A17]



SI Scheme 5. Harsh reaction conditions to achieve sterically hindered C-H activation reaction.

Computational Details

Additional details of the calculations:

In order to see how the rotational energy contributes to the barrier of the C-H activation we calculated the free energy profiles for the formation of the dimer palladacycles of selected substrates. We have employed typical reaction conditions: $Pd(OAc)_2$ catalyst (in its dimer form), CH_2Cl_2 solvent and 25°C temperature in all cases. For the optimizations and TS calculations we have used the ωB97XD functional[1] and the 6-31G* basis set for the main group atoms. The energies of the optimized structures have been recalculated by using the M06 functional[2] with the larger 6-311++G** basis set together with the SMD solvent model.[3] For Pd we used the LANL2DZ basis set augmented with a set of diffuse functions of s, p and d types and two sets of polarization functions of f-type taken from the aug-cc-PVDZ-PP basis set.[4] The inner shells of Pd have not been included into the calculations, instead their interactions have been represented by the LANL2DZ effective core potentials.[5] Vibrational calculations were performed to obtain free energy corrections and also to verify the optimized structures and TS structures. Additional IRC and optimization calculations have been carried out to see that the calculated TSs connect the respective initial and product states. The ideal gas - rigid rotor - harmonic oscillator approximations were applied to estimate the Gibbs free energy contributions. Earlier studies have already shown that the catalytically active form of $Pd(OAc)_2$ is its dimeric form.[6] Two reaction steps have been calculated: the first is when the catalyst reacts with the first substrate and a second step is when the dimeric form of the Pd-complex is formed. The mechanistic picture obtained from the calculations is very similar to that of Ref. [6]. We note that with these calculations we wished to probe our hypothesis that the initial steric conflicts present in the substrates are incorporated into the barriers of the C-H activation and for a meaningful comparison we followed the same mechanistic pathway for each substrate. In contrast, performing full, substrate-dependent refinements of the specific routes (including e.g. specific experimental conditions) is beyond the scope of the study. We obtained that both C-H activation steps (i.e. the stepwise activation of two substrates by the dimeric form of $Pd(OAc)_2$) are exergonic process. The barrier heights are therefore referenced to the preceeding intermediate states. For the first C-H activation this is the $Pd_2(OAc)_4$ + isolated substrate state. For the second step this is the Pd-complex featuring a single activated substrate. The results are summarized in SI Figure 1.

[1] Chai, J.-D.; Head-Gordon, M. Long-Range Corrected Hybrid Density Functionals with Damped Atom–Atom Dispersion Corrections. *Phys. Chem. Chem. Phys.* **2008**, *10*, 6615–6620.

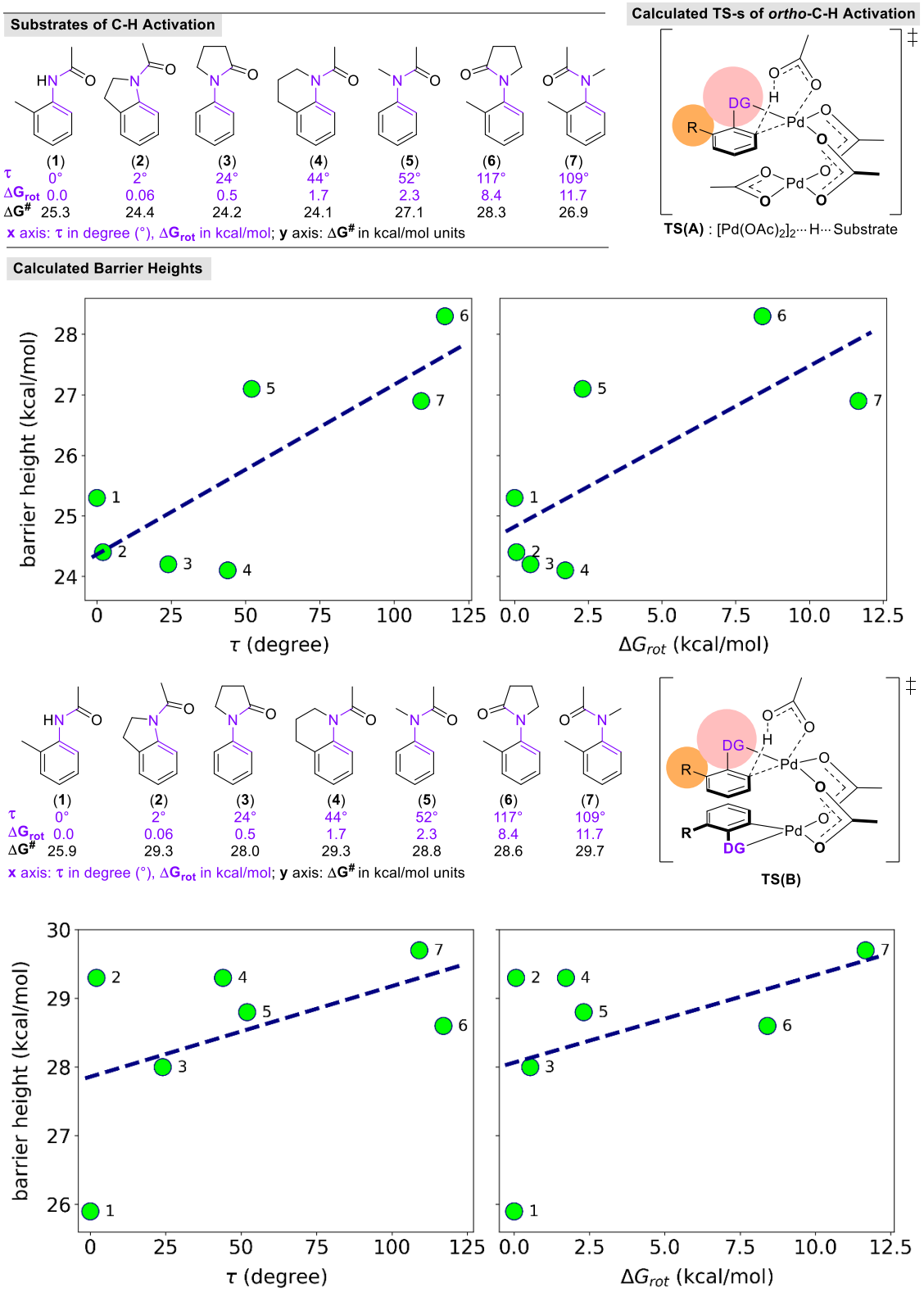
[2] Zhao, Y.; Truhlar, D. G. The M06 Suite of Density Functionals for Main Group Thermochemistry, Thermochemical Kinetics, Noncovalent Interactions, Excited States, and Transition Elements: Two New Functionals and Systematic Testing of Four M06 Functionals and 12 Other Functionals. *Theor. Chem. Acc.* **2008**, *119*, 525–525.

[3] Marenich, A. V.; Cramer, C. J.; Truhlar, D. G. Universal Solvation Model Based on Solute Electron Density and on a Continuum Model of the Solvent Defined by the Bulk Dielectric Constant and Atomic Surface Tensions. *J. Phys. Chem. B* **2009**, *113*, 6378–6396.

[4] **(a)** Pritchard, B. P.; Altarawy, D.; Didier, B.; Gibson, T. D.; Windus, T. L. New Basis Set Exchange: An Open, Up-to-Date Resource for the Molecular Sciences Community. *J. Chem. Inf. Model.* **2019**, *59*, 4814–4820. **(b)** Feller, D. The Role of Databases in Support of Computational Chemistry Calculations. J. Comput. Chem. **1996**, *17*, 1571–1586. **(c)** Schuchardt, K. L.; Didier, B. T.; Elsethagen, T.; Sun, L.; Gurumoorthi, V.; Chase, J.; Li, J.; Windus, T. L. Basis Set Exchange:0.167em A Community Database for Computational Sciences. *J. Chem. Inf. Model.* **2007**, *47*, 1045–1052.

[5] Hay, P. J.; Wadt, W. R. Ab Initio Effective Core Potentials for Molecular Calculations. Potentials for K to Au Including the Outermost Core Orbitals. *J. Chem. Phys.* **1985**, *82*, 299–310.

[6] Haines, B. E.; Berry, J. F.; Yu, J.-Q.; Musaev, D. G. Factors Controlling Stability and Reactivity of Dimeric Pd(II) Complexes in C–H Functionalization Catalysis. *ACS Catal.* **2016**, *6*, 829–839.



SI Figure 1. Barrier height of *ortho*-C-H activation of aromatic amides by $Pd(OAc)_2$ as a function of torsional angle t and DG_{rot} for the two steps leading to the formation of dimer palladacomplex. The numbering of the substrates helps to identify the data-pairs on the graphs. The calculated TS structure is shown schematically at the upper right corner. The blue trend lines are only guides to the eyes, as there is no chemical basis for a linear relationship in any case. Note that here we invoke DG_{rot} to make the actual energy comparisons.

A few notes are in order here. First, a perfect correlation can be expected if the variation of the activation barrier is solely due to the variation of the value of $DG^{\#}_{rot}$ (see Figure 2 in the article). This is however very difficult to achieve for a given set of substrates and the scattering of the points can be attributed to this issue. We also note that different substrates may form different intermediates under the given conditions which can contribute to an additional stabilization of some of the free energy levels. In this series of calculations we have followed reaction pathways defined by analogous structures, hence the barriers are not necessarily the optimal ones. An additional note is that these standardized calculations are at least an order of magnitude more expensive than the computation of the equilibrium torsional angle and the rotational barriers of the substrates alone. This implies that substrate-specific calculations including the actual experimental conditions would be even more demanding, stressing the advantage of our model.

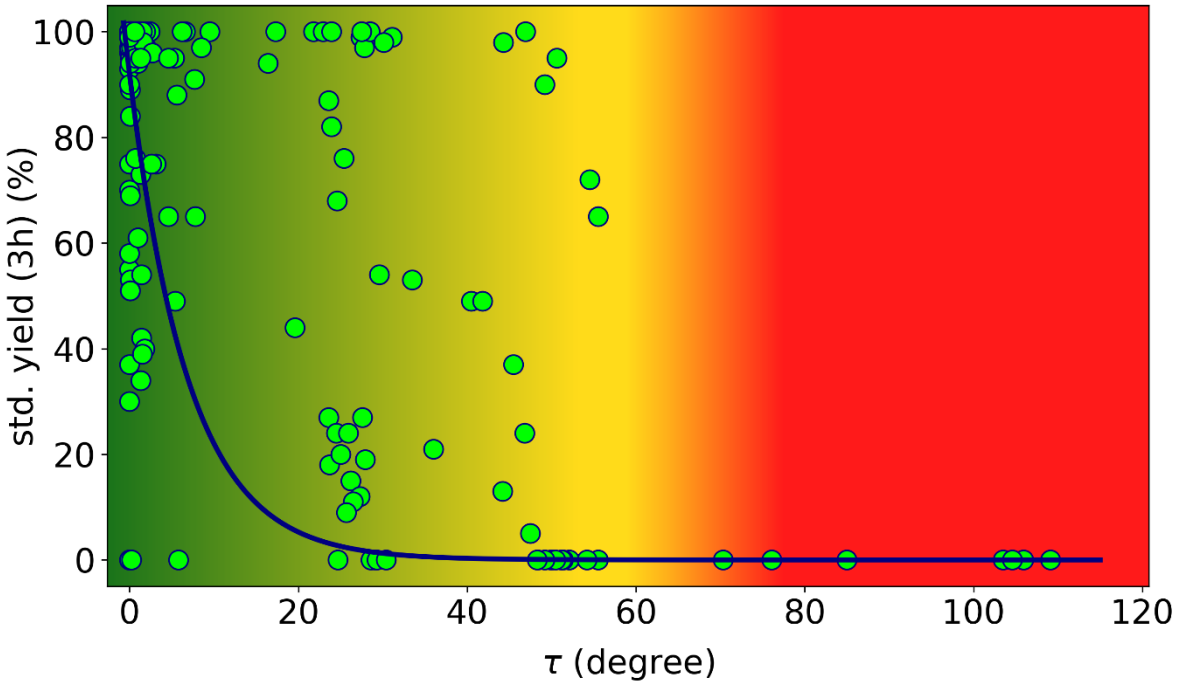
Concept of standardizing:

In order to put all the available experimental and computational data in equal footing we can use standardized yields and standardized barriers to express the efficiency of the reactions. In the study we use standardized barriers but we used the experimental yields. In ideal cases standardized yields would be the proper quantities to employ. The standardized yield is the yield obtained within (or projected to) a well-defined time frame (e.g. within 3 hours). However, using standardized yields implies the following assumptions: **I.)** the reactions follow the same kinetics with respect to the substrate (presumably first order); **II.)** the reaction time available for a reaction is entirely necessary to reach the given yield (e.g. the experimental yield could not have been obtained in shorter time frame[7]); **III.)** the reactions are kinetically controlled and the C-H activation is the rate determining step. With these assumptions in mind one can correlate the yields of equal reaction times (standardized yields) with quantities proportional to the energetics of the nonplanarity. To derive standardized yields a reasonable reaction time frame has to be chosen (e.g. 3 hours). The assumed first-order kinetics gives the yield $p(t_1)$ at the experimentally selected t_1 time as $p(t_1) = 1- \exp(-kt_1)$, where k is the reaction rate constant. It follows that the yield $p(t_2)$ at the standardized t_2 time can be calculated as

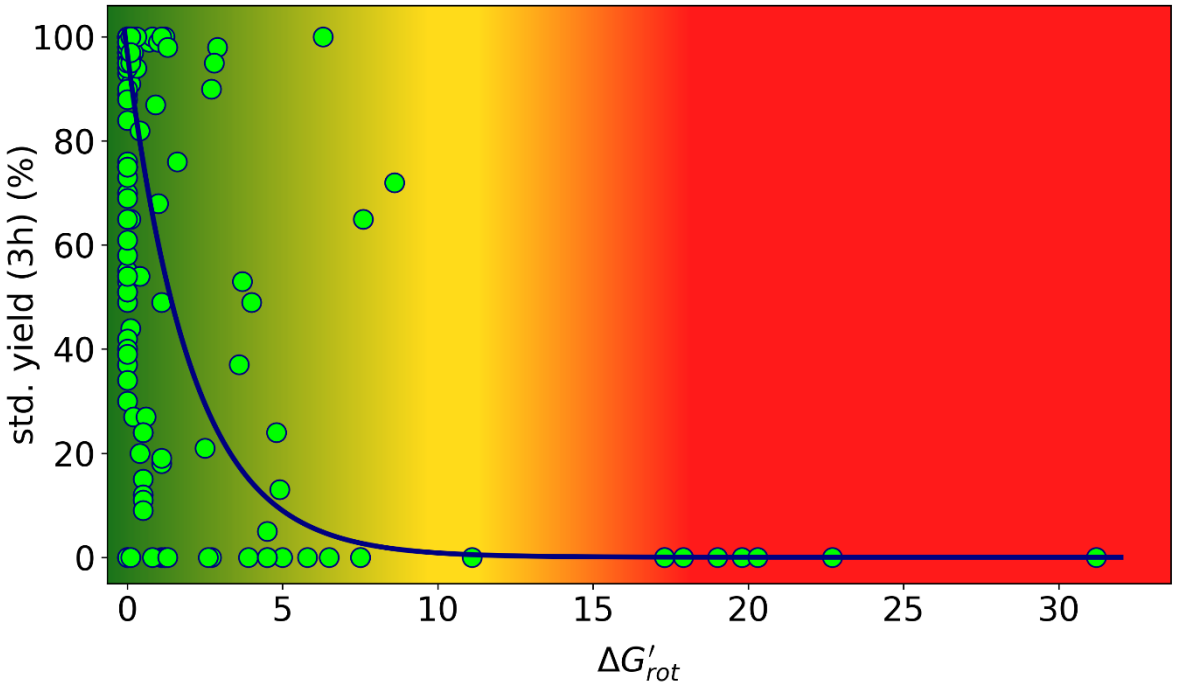
$$p(t_2) = 1 - \exp\left(\frac{t_2}{t_1} \cdot \ln(1 - p(t_2))\right) \qquad (2)$$

using the available t_1 and $p(t_1)$ values and setting t_2 to the standardized time (e.g. 3 hours). Note that the value of the actual rate constants is not necessary to calculate the standardized yield as seen in Eq. 2. However, as assumptions I-III are quite strong and in most of cases they are not satisfied, we decided to use the non-standardized, original yields and the results obtained employing standardized yields are shown here.

[7] The reaction time is most often defined by the initial optimization studies. This implies that the reaction time optimal for a given substrate is not necessarily optimal for another substrate.



SI Figure 2. Variation of the standardized experimental yield (projected to reaction time of 3 hours) as a function of the torsional angle τ of the directing groups. The blue line is a simple exponential curve and serves as guide to the eyes. The background color gradient is a qualitative measure of the success of the reactions, for explanation see Figure. 4 in the article.

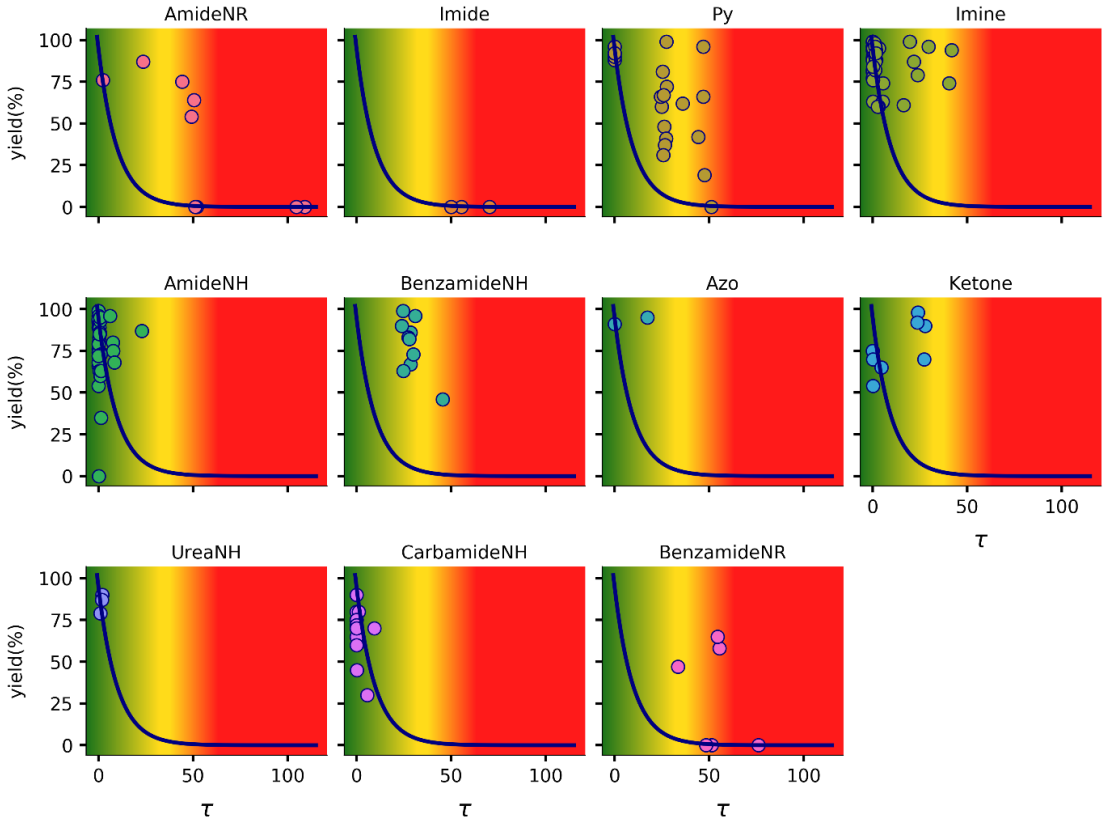


SI Figure 3. Variation of the standardized experimental yield (projected to reaction time of 3 hours) as a function of the standardized conformational energy (taking into account the actual experimental temperature) of the directing groups. The blue line is a simple exponential curve and serves as guide to the eyes. The background color gradient is a qualitative measure of the success of the reactions, for explanation see Figure. 4 in the article.

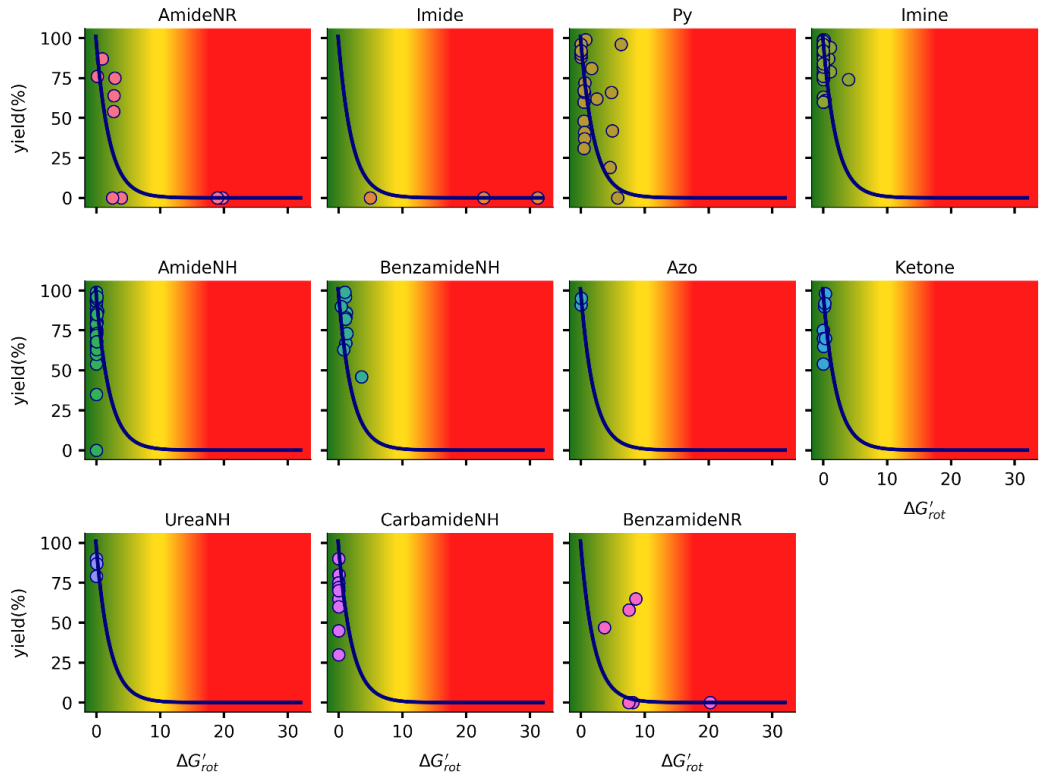
We wish to emphasize again, that the reaction times given in the literature most often do not correspond to the exact time necessary for a given yield, because these times are mostly the results of an optimalization protocol followed in the given publications. Indeed, a reaction time optimal for a given substrate may not necessarily be optimal for another one, and in fact it can be either shorter or longer for that substrate. Therefore we cannot expect accurate correlations with either the experimental or the standardized yields.

Correlation of the experimental yields and calculated parameters for individual directing groups

As we mentioned in the article the graphs based on the full data sets hide important features characteristic to the individual directing groups. To reveal these features, we decomposed the data and plotted the individual distributions in **SI Figures 4 and 5**. Clearly, in several cases the number of data points are too small to obtain statistically meaningful trends. However we have to keep in mind that **I.)** the number of available experiments where the Pd-activated intermediates have been reported is limited; **II.)** as we mentioned earlier, results with lower yields less likely get published; **III.)** often the Pd-intermediate cannot be isolated although its presence can be safely postulated from the successful overall yield of the corresponding coupling reactions.



SI Figure 4. Variation of the experimental yield as a function of the torsional angle τ of the directing groups. The continuous blue line representing a hypothetical trend in each plot is in fact only a guide for the eyes.



SI Figure 5. Variation of the experimental yield as a function of the standardized conformational energy ($\Delta G_{rot}/RT$, ($\Delta G'_{rot}$) for the different directing groups. The continuous blue line representing a hypothetical trend in each plot is in fact only a guide for the eyes.

Details of Syntheses

General conditions

Analytical thin-layer chromatography (TLC) was performed on Merck DC pre coated TLC plates with 0.25 mm Kieselgel 60 F_{254}. Visualization was performed with a 254 nm UV lamp.

The ^{1}H, ^{13}C, and ^{19}F NMR spectra were recorded on a Bruker Avance-250, Agilent (Varian) VNMRS-400 and VNMR-600 spectrometers in CD_2Cl_2 and DMSO-d_6. Measurements were performed on indirect detection Z-gradient probes. Direct detection probe was used for extremely poor soluble samples Chemical shifts are expressed in parts per million (δ). The^{1}H and ^{13}C chemical shifts are referenced to the residual solvent signals; for ^{19}F chemical shifts, $CFCl_3$internal standard is used. Coupling constants (J) are reported in hertz (Hz). Splitting patterns are designated as s (singlet), bs (broad singlet), d (doublet), t (triplet), q (quartet), and m (multiplet). The structure determination is based on one- (^{1}H, ^{13}C, ^{19}F NMR, selective 1d-NOESY) and two-dimensional (^{1}H-^{13}C-gHSQCAD, ^{1}H-^{13}C-gHMBCAD, ^{1}H-zqTOCSY) NMR experiments. The amide rotamers were identified by ^{1}H and ^{19}F two dimensional NOESY experiments. For NMR spectra see the Supporting Information – NMR Spectra.

Conversions determined on Gas chromatography Agilent 5890 Gas Chromatograph (30 m × 0.25 mm column with 0.25 μm HP-5MS coating, He carrier gas) with FID detector and low resolution mass spectrometry was obtained on an Agilent 6890N Gas Chromatograph (30 m × 0.25 mm column with 0.25 μm HP-5MS coating, He carrier gas) and Agilent 5973 Mass Spectrometer (Ion source: EI+, 70eV, 230 ºC interface 300 ºC).

IR spectra were obtained in dichloromethane solutions on a Mettler Toledo ReactIR™ 15, AgX DiComp probe, 6 mm x 1.5 m Fiber (Silver Halide), MCT detector. The in-situ reactions were followed with following setup: sampling interval: 15 sec., 2500-650 cm^{-1} (resolution 8 cm^{-1}) Scan option: AutoSelect; Gain: 1x. Data were processed by Mettler Toledo iC IR™.

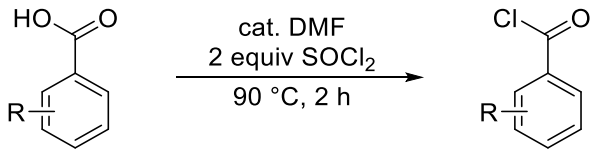
All melting points were measured on Büchi 501 apparatus and are uncorrected.

High-resolution mass spectra were acquired on an Agilent 6230 time-of-flight mass spectrometer equipped with a Jet Stream electrospray ion source in positive ion mode. Injections of 0.1-0.3 μl were directed to the mass spectrometer at a flow rate 0.5 ml/min (70% acetonitrile-water mixture, 0.1 % formic acid), using an Agilent 1260 Infinity HPLC system. Jet Stream parameters: drying gas (N_2) flow and temperature: 10.0 l/min and 325 °C, respectively; nebulizer gas (N_2) pressure: 10 psi; capillary voltage: 4000V; sheath gas flow and temperature: 325 °C and 7.5 l/min; TOFMS parameters: fragmentor voltage: 120 V; skimmer potential: 120V; OCT 1 RF Vpp:750 V. Full-scan mass spectra were acquired over the m/z range 100-2500 at an acquisition rate of 250 ms/spectrum and processed by Agilent MassHunter B.03.01 software. Pd-complexes: 10 V cone voltage, calculated for ^{108}Pd isotope.

Preparation of starting materials

Unless otherwise indicated, all starting materials were obtained from commercial suppliers, and were used without further purification.

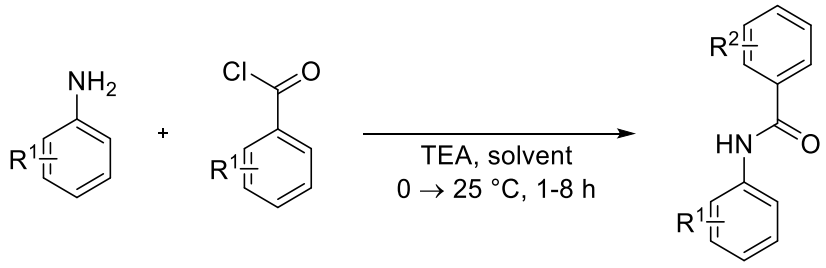
Preparation of benzoyl chlorides



SI Scheme 6.

A 100 mL two-neck round-bottom flask was charged with *aromatic carboxylic acid* (15-40 mmol scale). The flask equipped with a stirring bar, a reflux condenser and a calcium chloride (anh.) guard tube. 2 equivalents of *thionyl chloride* was added in one portion. The reaction mixture was stirred for 1 minute and 0.1 mL of *N,N-Dimethylformamide* was added. The mixture heated to 90 °C, and stirred for 2 hours. The reaction takes place with intensive evolution of hydrochloric acid, and the reaction mass becomes liquid. After the reaction the yellow liquid was allowed to cool to room temperature, the condenser was removed, and the flask was connected to a vacuum system with a trap-tube and the remained thionyl chloride distilled off. The residual aroyl chloride was used without further purification.

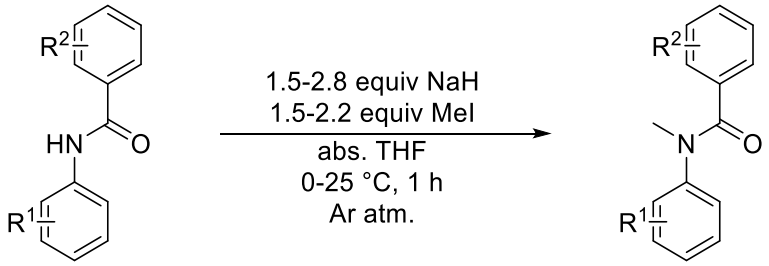
Preparation of amide substrates



SI Scheme 7.

A 100 mL round-bottom flask was equipped with stirring bar and charged with *aryl amine* (15 mmol), 40 mL of *abs. ethyl acetate* was added and the solution cooled to 0 °C. *Triethyl amine* (2.2 mL, 1.05 equiv. 15.75 mmol) added and the solution cooled to 0 °C. Subsequently, a solution of *aroyl chloride* (1.1 equiv. 16.5 mmol in 15 mL *solvent)* added to the amine solution in small portions and allowed to warm up to room temperature. The reaction mixture stirred until all the starting materials reacted (1-4 hours), monitored by TLC. The precipitated white triethyl amine hydrochloride was filtered off and washed.

The reaction mixture extracted with 1M HCl solution, twice with distilled water, then with sat. $NaHCO_3$ solution and once with brine. The organic phase solution was dried over $MgSO_4$ and the solvent evaporated in rotary evaporator. The crude product was purified by crystallization.

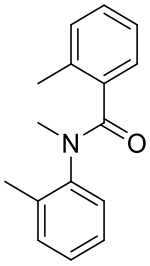


SI Scheme 8.

A flame-dried 50 mL three-neck round-bottom flask was equipped with stirring bar and charged with *N-arylbenzamide* (6.5 mmol) and sealed with septum. The flask was inertized via evacuating and refilling with argon gas (repeat 3-times), then 20 mL absolute tetrahydrofuran was added as solvent. The flask connected to a gas bubbler and argon-line. The solution was cooled to 0 °C by ice-water bath. Under argon atmosphere sodium-hydride (60 m/m% in mineral oil) was added in a minute by small portions, which resulted intensive hydrogen evolution. The reaction mixture was stirred for 30 minutes, it became off-white slurry. After this methyl iodide was added to the solution, and the ice-water bath was removed to allow the mixture to warm up to room temperature. The reaction mixture stirred until all the starting materials reacted (1-4 hours), monitored by TLC.

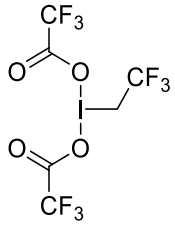
The reaction mixture was filtrated trough Celite, washed with abs. tetrahydrofuran and the solution evaporated to dryness. The oily residual dissolved in ethyl acetate and extracted sequentially with distilled water, 1M HCl solution, distilled water, sat. $NaHCO_3$ solution and finally with sat. NaCl solution. The organic phase solution was separated and dried over $MgSO_4$. The solvent evaporated in rotary evaporator and the crude product was purified by column chromatography using silica gel and hexane : ethyl acetate eluent.

***N*,2-dimethyl-*N*-(*o*-tolyl)benzamide (15)**



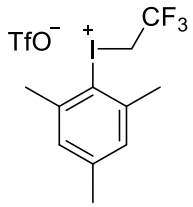
The general procedure was followed using 1 equiv *2-methyl-N-(o-tolyl)benzamide* (1,126 g , 5 mmol) and 2.2 equiv NaH (440 mg, 11 mmol) and 2.2 MeI (0.68 ml d=2.28 g/cm^3, 11 mmol). White solid 1.069 g (4.47 mmol, 89%), **MP**: 89-92 °C. **^{1}H NMR** (250 MHz, DMSO) 2 rotamers (R1 and R2) in R1:R2=2:1 ratio : δ 7.74 – 6.67 (m, 16H R1+R2), 3.26 (s, 3H R1), 2.99 (s, 3H R2), 2.38 (s, 3H R2), 2.34 (s, 3H R1), 2.29 (s, 3H R2), 2.24 (s, 3H R1). **^{13}C{H} NMR** (63 MHz, DMSO) R1+R2 δ 170.12, 169.98, 142.80, 142.27, 137.22, 136.69, 135.52, 135.11, 134.87, 133.93, 131.26, 131.06, 130.65, 130.42, 129.13, 128.72, 128.68, 127.94, 127.88, 127.71, 127.35, 126.96, 126.57, 126.28, 126.02, 124.94, 39.43, 36.51, 19.73, 19.03, 17.76. **HRMS** calculated for $C_{16}H_{18}NO$ $[M+H]^+$ 240.1388 found: 240.1389. **MS** (EI, 70 eV) m/z (%): 239(12), 208(5), 120(11), 119(100), 91(51), 65(18), **IR** (ATR): *ν* = 2942, 2927, 2910, 2871, 2858, 2537, 1639, 1495, 1437, 1422, 1376, 1264, 897, 736, 705 cm^{-1}.

(2,2,2-Trifluoroethyl)-λ^3-iodanediyl bis(2,2,2-trifluoroacetate)



As described:[8] Into a 200 mL round-bottom flask *2,2,2-trifluoroacetic anhydride* (72 mL, 520 mmol) and *trifluoroacetic acid* (620 µL, 10 mol%) was measured and the mixture was cooled to 0 °C. *H_2O_2* (8 mL, 50% aqueous solution, 118 mmol) was added dropwise and the mixture was stirred for 5 minutes. *2,2,2-trifluoroiodoethane* (8 mL, 80 mmol) was added, then the reaction was allowed to warm up to room temperature and it was stirred for 20 hours. The volatile was evaporated under reduced pressure and white oil was obtained, which crystallized in refrigerator and gave the white solid in quantitative yield. **^{1}H NMR** (250 MHz, $CDCl_3$) δ = 4.93 (q, J = 10.0 Hz, 2H). **^{19}F NMR** (235 MHz, $CDCl_3$) δ = -63.2, -73.4.

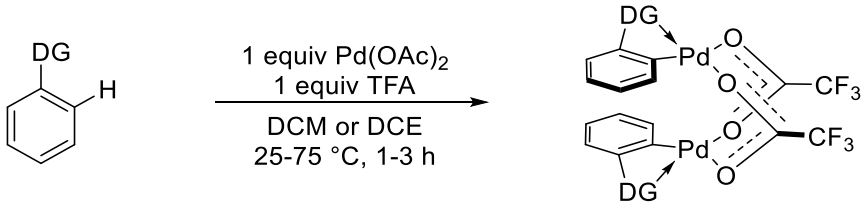
2,2,2-Trifluoroethyl(mesityl)iodonium trifluoromethanesulfonate



As described:[9,10] In a 100 mL round-bottom flask *(2,2,2-trifluoroethyl)-λ^3-iodanediyl bis(2,2,2-trifluoroacetate)* (7.847 g, 18 mmol) was dissolved in *dichloromethane* (30 mL), then the solution was cooled to 0 °C and *mesitylene* (3.88 mL, 28 mmol) was added. Then *trifluoromethanesulfonic acid* (1.6 mL, 18 mmol) was added dropwise. The reaction mixture turned to a dark red solution and it was kept at 0 °C for 24 hours. The solvent was evaporated under reduced pressure, then diethyl ether was added. White crystals precipitated from the mixture. The product was filtered off and washed with diethyl ether.
White solid 8.15 g (17 mmol, 92%). **MP:** 114 °C. **^{1}H NMR** (250 MHz, DMSO) δ = 6.95 (s, 2H), 5.01 (q, J = 7.5 Hz, 2H), 2.35 (s, 6H), 2.19 (s, 3H). **^{13}C{H} NMR** (63 MHz, DMSO) δ = 141.5, 137.2, 128.0, 104.3, 59.3 (q, J = 34.0), 29.2, 20.3. **^{19}F NMR** (235 MHz, $CDCl^3$) δ = -61.9, -78.6. **HRMS** calculated for $C_{11}H_{13}F_3I$ $[M]^+$ 329.0009; found 329.0014. **IR** (ATR) 2036, 1454, 1395, 1279, 1227, 1160, 1130, 1059, 1022, 854, 839 cm^{-1}.

[8] Umemoto, T.; Gotoh, Y. Synthesis, Properties, and Reactivity of (1H,1H-Perfluoroalkyl)- and (1H-Perfluoro-1-alkenyl)aryliodonium Triflates and Their Analogs. *Bull. Chem. Soc. Jpn.* **1987**, *60*, 3307–3313.
[9] Tolnai, G. L.; Székely, A.; Makó, Z.; Gáti, T.; Daru, J.; Bihari, T.; Stirling, A.; Novák, Z. Efficient direct 2,2,2-trifluoroethylation of indoles via C–H functionalization. *Chem. Commun.* **2015**, *51*, 4488–4491.
[10] Kovács, S.; Tóth, B. L.; Borsik, G.; Bihari, T.; May, N. V.; Stirling, A.; Novák, Z. Direct *ortho*-Trifluoroethylation of Aromatic Ureas by Palladium Catalyzed C-H activation: A Missing Piece of Aromatic Substitutions. *Adv. Synth. Catal.* **2016**, *359*, 527–532.

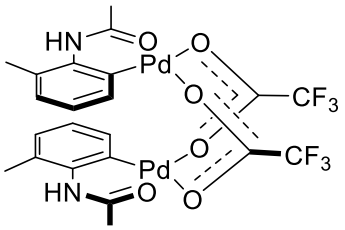
Synthesis of the palladium-complexes



SI Scheme 9.

General procedure: A 4 mL screw-cap vial charged with *palladium(II) acetate* (1 equiv, 112.5 mg, 0.5 mmol), then *aromatic amide* (1 equiv, 0.5 mmol) and equipped with a stirring bar. 0.5 mL *dichloromethane* was added as solvent. *Trifluoroacetic acid* (1 equiv, 0.5 mmol) was added instantly and the mixture was stirred at 40 °C for 3 hours. resulting a brown solution or slurry. The reaction mixture evaporated in rotary evaporator, and the brownish crude product scoured in 1 mL pentane : dichloromethane = 1:1. The (mainly yellow) solid filtered off and washed with eluent. The product was dried overnight under reduced pressure (35 °C).

Bis(2-acetamidophenyl)bis[*μ*-(trifluoroacetato)]-dipalladium(II) (1-Pd)

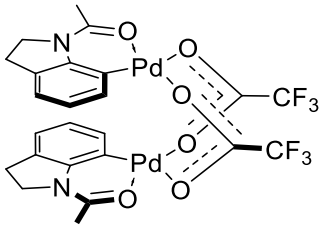


As described:[12] A 7 mL screw-cap vial charged with *palladium(II) acetate* (449 mg, 2 mmol), then *N-(o-tolyl)acetamide* (**3**, 298.2 mg, 2 mmol) and equipped with a stirring bar. 4 mL *dichloromethane* was added as solvent. *Trifluoroacetic acid* (154 μL, 2 mmol) was added instantly and the mixture was stirred at 40 °C for 3 hours. Yellow solid precipitated from the brown solution. The reaction mixture evaporated in rotary evaporator, and the brownish crude product scoured in 1 mL hexane : dichloromethane = 1:1. The yellowish solid filtered off and washed with dichloromethane. The product was dried overnight under reduced pressure (35 °C).

As described:[11,12] Yellow solid 683 mg (0.93 mmol, 93%). **MP**: 170-203 °C (dec.).**^{1}H NMR** (250 MHz, DMSO-d6) δ 10.9 (s, 1H), 7.3 (s, 1H), 7.0 (d, J = 6.8 Hz, 1H), 6.9 (t, J = 7.4 Hz, 1H), 2.4 (s, 3H), 2.3 (s, 3H). **^{13}C{H} NMR** (63 MHz, DMSO-d6) δ 171.7, 159.2 (d, J = 33.5 Hz), 132.1, 130.7, 128.5, 126.0, 125.2, 21.3, 18.9. **^{19}F NMR** (235 MHz, DMSO-d6) δ -74.1. **IR** 1659, 1611, 1560, 1543, 1447, 1410, 1376, 1328, 1193, 1149, 850, 768, 731 cm^{-1}.

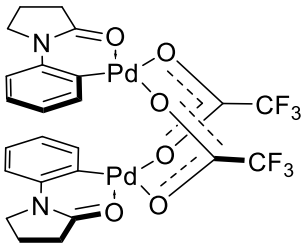
[11] Yang, F.; Song, F.; Li, W.; Lan, J.; You, J. Palladium-catalyzed C–H activation of anilides at room temperature: *ortho*-arylation and acetoxylation. *RSC Adv.* **2013**, *3*, 9649.
[12] Tóth, B. L.; Kovács, S.; Sályi, G.; Novák, Z. Mild and Efficient Palladium-Catalyzed Direct Trifluoroethylation of Aromatic Systems by C-H Activation. *Angew. Chem. Int. Ed.* **2016**, *55*, 1988–1992.

Bis(1-acetylindolin-7-yl)bis[μ-(trifluoroacetato)]-dipalladium(II) (2-Pd)



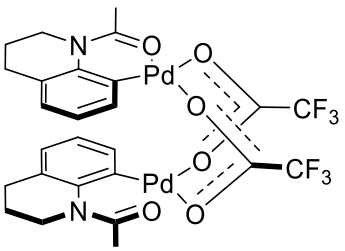
The general procedure was followed using 1 equiv of *N-acetylyindoline* (**22**, 81 mg, 0.5 mmol). Yellow solid, 145 mg (0.191 mmol, 77%), **MP**: 233< °C. **^{1}H NMR** (400 MHz, 25 °C, CD_2Cl_2) δ(ppm): 6.97 (1H, m, H5), 6.84 (2H, m, H4 and H6), 4.02-3.40 (2H, br, H9), 3.40-3.02 (2H, br, H8), 1.67 (3H, s, H11). **^{13}C{^{1}H} NMR** (100 MHz, 25 °C, CD_2Cl_2) δ(ppm): 164.1 (C10), 134.7 (C2), 131.3 (C4), 128.4 (C3), 124.6 (C6), 121.7 (C5), 112.9 (C1), 49.4 (C9), 29.2 (C8), 20.2 (C10). **^{19}F NMR** (376 MHz, 25 °C, CD_2Cl_2) δ(ppm): -75.0. **IR** (ATR): ν = 1663, 1637, 1195, 1169, 1150, 850, 790, 757, 729, 712, 693, 680, 667, 662, 654 cm^{-1}. **HRMS** calculated for $C_{12}H_{13}N_2OPd$ $[M+CH_3CN]^+$ 307.0063 found: 307.0061.

Bis(2-(2-oxopyrrolidin-1-yl)phenyl)bis[μ-(trifluoroacetato)]-dipalladium(II) (3-Pd)



As described:[13] The general procedure was followed using 1 equiv of *N-acetylindoline* (**14**, 80.6 mg, 0.5 mmol). As described[Hiba! A könyvjelző nem létezik.]: yellow solid, 159 mg (0.21 mmol, 84%), **M P**: 238-246 °C (decomp.). **^{1}H NMR** (250 MHz, DMSO-d_6) δ 7.53 (s, 1H), 7.22 (t, J = 7.3 Hz, 1H), 7.04 (t, J = 9.8 Hz, 2H), 4.15 (t, J = 7.1 Hz, 2H), 2.87 (t, J = 8.0 Hz, 2H), 2.25 (p, J = 7.5 Hz, 2H). **^{13}C{H} NMR** (63 MHz, DMSO) δ 135.29, 133.68, 126.58, 125.47, 116.08, 50.88, 32.48, 18.74. **^{19}F NMR** (235 MHz, DMSO-d_6) δ -74.05. **IR** (ATR): ν = 1666, 1626, 1451, 1201, 1147, 853, 789, 757, 730 cm^{-1}.

Bis(1-acetyl-1,2,3,4-tetrahydroquinolin-8-yl)bis[μ-(trifluoroacetato)]-dipalladium(II) (4-Pd)

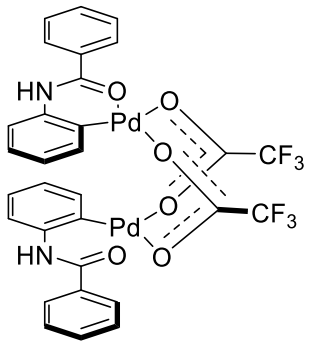


The general procedure was followed using 1 equiv of *N-acetyl-1,2,3,4-tetrahydroquinoline* (**23**, 88 mg, 0.5 mmol). Pale yellow solid, 147 mg (0.188 mmol, 75%), **MP**: 225-227 °C. Two conformers in 60:40 ratio. **^{1}H NMR** (400 MHz, 25 °C, CD_2Cl_2) δ(ppm): 6.89 (1H, dd, J = 7.3, 1.6 Hz, H10), 6.85 (1H, t, J = 7.4 Hz, H11), 6.75 (1H, dd, J = 7.7, 1.7 Hz, H12), 6.72 (1H, dd,

[13] Yeung, C. S.; Zhao, X.; Borduas, N.; Dong, V. M. Pd-catalyzed *ortho*-arylation of phenylacetamides, benzamides, and anilides with simple arenes using sodium persulfate. *Chem. Sci.* **2010**, *1*, 331.

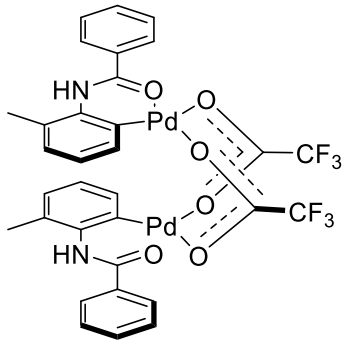
J = 7.3, 1.2 Hz, H10'), 6.52 (1H, t, *J* = 7.6 Hz, H11'), 6.09 (1H, dd, *J* = 7.9, 1.3 Hz, H12'), 3.50 (2H, s, H6), 3.45-2.93 (2H, br, H6'), 2.79 (2H, t, *J* = 6.4 Hz, H4), 2.62 (2H, br, H4'), 2.34 (2H, s H13'), 2.02 (3H, s, H13), 1.91 (2H, q, *J* = 6.1 Hz, H5), 1.80-1.60 (2H, br, H5'). **^{13}C{^{1}H} NMR** (100 MHz, 25 °C, CD_2Cl_2) δ(ppm): 171.7 (C7'), 170.7 (C7), 132.3 (C12'), 132.2 (C12), 131.6 (C2'), 131.1 (C2), 127.1 (C10), 126.8 (C3), 126.7 (C3'), 126.6 (C10'), 124.8 (C11 and C11'), 121.3 (C9'), 120.5 (C9), 49.9 (C6), 49.5 (C6'), 28.3 (C4), 27.8 (C4'), 23.2 (C5), 23.1 (C5'), 22.9 (C13), 22.6 (C13'). **^{19}F NMR** (376 MHz, 25 °C, CD_2Cl_2) δ(ppm): -74.5, -75.0, 75.1. **IR** (ATR): *v* = 1670, 1598, 1562, 1447, 1432, 1266, 1202, 1150, 850, 787, 772, 734, 705, 667, 662 cm^{-1}. **HRMS** calculated for $C_{13}H_{15}N_2OPd$ $[M+CH_3CN]^+$ 321.0219 found: 321.0222. **XRD**: See below.

Bis(2-benzamidophenyl)bis[*μ*-(trifluoroacetato)]-dipalladium(II) (8-Pd)



The general procedure was followed using 1 equiv of *N-phenylbenzamide* (**24**, 80.6 mg, 0.5 mmol). Yellow solid, 174 mg (0.209 mmol, 84%), **MP**: 216-217 °C. Two conformers in 88:12 ratio. **^{1}H NMR** (400 MHz, 25 °C, CD_2Cl_2) δ(ppm): 8.24 (1H, s, H8), 7.55 (1H, tt, *J* = 7.3, 1.3 Hz, H16), 7.39 – 7.30 (2H, m, H15 and H17), 7.31 – 7.23 (2H, m, H14 and H18), 7.00 (1H, dd, *J* = 8.0, 1.3 Hz, H2), 6.69 (1H, td, *J* = 7.4, 1.3 Hz, H6), 6.57 (1H, ddd, *J* = 8.4, 7.2, 1.5 Hz, H1), 6.46 (1H, dd, *J* = 7.7, 1.5 Hz, H5). **^{13}C{^{1}H} NMR** (100 MHz, 25 °C, CD_2Cl_2) δ(ppm): 162.8 (C10), 133.8 (C16), 133.7 (C2), 130.5 (C4), 129.3(C15 and C17), 129.1 (C13), 127.6 (C14 and C18), 126.2 (C6), 124.5 (C1), 116.0 (C3 and C5). **^{19}F NMR** (376 MHz, 25 °C, CD_2Cl_2) δ(ppm): -74.3, -74.9, -75.0. **IR** (ATR): *v* = 1665, 1605, 1579, 1558, 1538, 1495, 1461, 1197, 1150, 854, 789, 751, 733, 699, 656 cm^{-1}. **HRMS** calculated for $C_{15}H_{13}N_2OPd$ $[M+CH_3CN]^+$ 343.0063 found: 343.0056. **XRD**: See below.

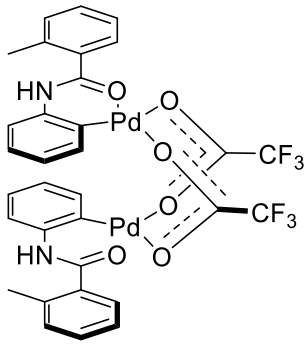
Bis(2-benzamido-3-methylphenyl)bis[*μ*-(trifluoroacetato)]-dipalladium(II) (9-Pd)



The general procedure was followed using 1 equiv of *N-(o-tolyl)benzamide* (**25**, 106 mg, 0.5 mmol). Yellow solid, 143 mg (0.166 mmol, 67%), **MP**: 214-215 °C. Two conformers in 83:17 ratio. **^{1}H NMR** (400 MHz, 25 °C, CD_2Cl_2) δ(ppm): 8.23 (1H, s, H8), 7.65 – 7.54 (1H, *J* = 7.3, 1.3 Hz, H16), 7.39 – 7.30 (2H, m, H15 and H17), 7.25-7.20 (2H, m, H14 and H18)), 6.87 (1H,

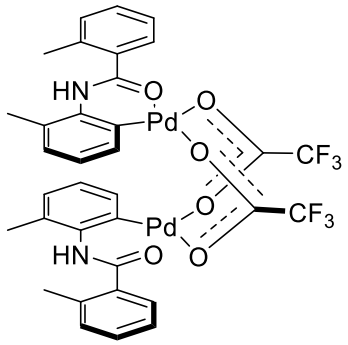
dd, *J* = 7.9, 1.6 Hz, H2), 6.58 (1H, dd, *J* = 7.0, 2.1 Hz, H6), 6.52 (1H, dd, *J* = 7.9, 7.0 Hz H1), 2.05 (3H, s, H19). **^{13}C{^{1}H} NMR** (100 MHz, 25 °C, CD_2Cl_2) δ(ppm): 162.8 (C10), 133.8 (C16), 133.7 (C2), 130.5 (C4), 129.3(C15 and C17), 129.1 (C13), 127.6 (C14 and C18), 126.2 (C6), 124.5 (C1), 116.0 (C3 and C5). **^{19}F NMR** (376 MHz, 25 °C, CD_2Cl_2) δ(ppm): -74.4, -74.9, -75.0. **IR** (ATR): *v* = 1668, 1603, 1575, 1557, 1536, 1493, 1447, 1197, 1150, 854, 789, 770, 733, 721, 697 cm^{-1}. **HRMS** calculated for $C_{16}H_{15}N_2OPd$ $[M+CH_3CN]^+$ 357.0219 found: 357.0227.

Bis(2-(2-methylbenzamido)phenyl)bis[*μ*-(trifluoroacetato)]-dipalladium(II) (10-Pd)



The general procedure was followed using 1 equiv of *2-methyl-N-phenylbenzamide* (**26**, 106 mg, 0.5 mmol). Pale brown solid, 182 mg (0.211 mmol, 85%), **MP**: 210-213 °C. Two conformers in 78:22 ratio. **^{1}H NMR** (400 MHz, 25 °C, CD_2Cl_2) δ(ppm): δ 8.23 (1H, s, H10), 7.40 (1H, td, *J* = 7.6, 1.4 Hz, H4), 7.20 (1H, d, *J* = 7.7 Hz, H3), 7.14 (1H, t, *J* = 7.6 Hz, H5), 7.01 (1H, dd, *J* = 8.2, 1.3 Hz, H13), 6.97 (1H, td, *J* = 7.4, 1.3 Hz, H15), 6.85 (1H, dd, *J* = 7.8, 1.4 Hz, H6), 6.70 (1H, ddd, *J* = 8.3, 7.2, 1.4 Hz, H14), 6.60 (1H, dd, *J* = 7.7, 1.5 Hz, H16), 2.12 (3H, s, H10). **^{13}C{^{1}H} NMR** (100 MHz, 25 °C, CD_2Cl_2) δ(ppm): 164.5 (C7), 138.6 (C2), 134.1 (C13), 132.6 (C4), 132.3 (C3), 130.7 (C11), 130.3 (C1), 128.0 (C6), 126.6 (C5), 126.4 (C15), 124.4 (C14), 116.1 (C16), 115.7 (C12), 20.7 (C9). **^{19}F NMR** (376 MHz, 25 °C, CD_2Cl_2) δ(ppm): -74.4, -74.9, -75.0. **IR** (ATR): *v* = 2917, 1670, 1611, 1596, 1557, 1534, 1489, 1460, 1199, 1152, 854, 789, 753, 733, 675 cm^{-1}. **HRMS** calculated for $C_{16}H_{15}N_2OPd$ $[M+CH_3CN]^+$ 357.0219 found: 357.0214.

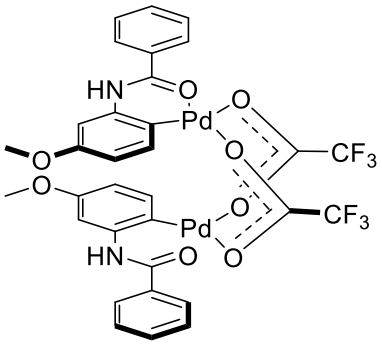
Bis(3-methyl-2-(2-methylbenzamido)phenyl)bis[*μ*-(trifluoroacetato)]-dipalladium(II) (11-Pd)



The general procedure was followed using 1 equiv of *2-methyl-N-(o-tolyl)benzamide* (**27**, 112.5 mg, 0.5 mmol). Pale brown solid, 140 mg (0.158 mmol, 63%), **MP**: 205-206 °C. Two conformers in 78:22 ratio. **^{1}H NMR** (400 MHz, 25 °C, DMSO-*d6*) δ(ppm): δ 11.21 (3H, s, H8), 7.74 (1H, dd, *J* = 8.2, 1.4 Hz, H15), 7.54 (1H, td, *J* = 7.5, 1.4 Hz, H13), 7.47 – 7.34 (3H, m, H3, H12 and H14), 7.07 (1H, d, *J* = 7.2 Hz, H5), 7.00 (1H, t, *J* = 7.6 Hz, H4), 2.53 (3H, s, H18), 2.43 (3H, s, H7). **^{13}C{^{1}H} NMR** (100 MHz, 25 °C, DMSO-*d6*) δ(ppm): 169.0 (C9), 137.2 (C11), 132.3(C3),

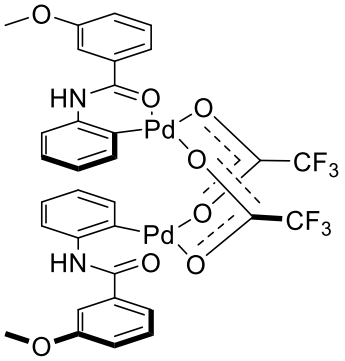
132.1 (C1), 131.8 (C10), 131.6 (C12), 131.0 (C13), 129.5 (C15), 128.6 (C5), 128.2 (C6), 126.0 (14), 125.8 (C4), 117.6 (C2), 19.9 (C18), 18.9 (C7). **IR** (ATR): *v* = 2910, 1655, 1612, 1598, 1571, 1532, 1491, 1447, 1325, 1195, 1156, 856, 787, 770, 734 cm^{-1}. **HRMS** calculated for $C_{17}H_{17}N_2OPd$ $[M+CH_3CN]^+$ 371.0376 found: 371.0376. **XRD**: See below.

Bis(2-benzamido-4-methoxyphenyl)bis[*μ*-(trifluoroacetato)]-dipalladium(II) (40)



The general procedure was followed using 1 equiv of *N-(3-methoxyphenyl)benzamide* (114 mg, 0.5 mmol) and was stirred in ice-cold bath at 0 °C for 3 hours (At 40 °C only oily black tar was formed). Yellow solid, 63 mg (0.071 mmol, 28%), **MP**: 147-148 °C. Two conformers in 86:14 ratio. **^{1}H NMR** (400 MHz, 25 °C, CD_2Cl_2) δ(ppm): δ 8.36 (1H, s, H7), 7.57 (1H, t, *J* = 7.7 Hz, H12), 7.35 (2H, t, *J* = 7.7 Hz, H11 and H13), 7.31 (2H, d, *J* = 7.6 Hz, H10 and H14), 6.85 (1H, d, *J* = 8.8 Hz, H3), 6.17 (1H, dd, *J* = 8.8, 2.7 Hz, H4), 6.07 (1H, d, *J* = 2.7 Hz, H6), 3.43 (3H, s, H17). **^{13}C{^{1}H} NMR** (100 MHz, 25 °C, CD_2Cl_2) δ(ppm): 163.1 (C8), 158.6 (C5), 133.9 (C3), 133.7 (C12), 133.0 (C1), 131.0 (C9), 129.2 (C11 and C13), 128.2 (C10 and C14), 109.9 (C4), 105.6 (C2), 102.1 (C6), 55.5 (C17). **^{19}F NMR** (376 MHz, 25 °C, CD_2Cl_2) δ(ppm): -74.3, 74.9, 75.0. **IR** (ATR): *v* = 1674, 1609, 1570, 1536, 1495, 1467, 1275, 1234, 1202, 1173, 1152, 852, 733, 707, 697 cm^{-1}. **HRMS** calculated for $C_{16}H_{15}N_2O_2Pd$ $[M+CH_3CN]^+$ 373.0168 found: 373.0172.

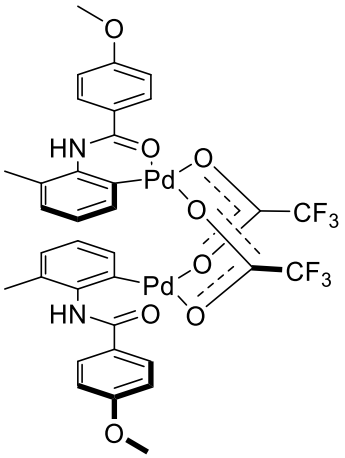
Bis(2-(3-methoxybenzamido)phenyl)bis[*μ*-(trifluoroacetato)]-dipalladium(II) (41)



The general procedure was followed using 1 equiv of *3-methoxy-N-phenylbenzamide* (114 mg, 0.5 mmol). Yellow solid, 158 mg (0.177 mmol, 71%), **MP**: 213-217 °C. Two conformers in 86:14 ratio. **^{1}H NMR** (400 MHz, 25 °C, CD_2Cl_2) δ(ppm): 8.25 (1H, s, H11), 7.25 (1H, t, *J* = 8.0 Hz, H5), 7.08 (1H, ddd, *J* = 8.3, 2.6, 0.9 Hz, H4), 7.00 (1H, dd, *J* = 8.0, 1.3 Hz, H14), 6.88 (1H, ddd, *J* = 7.7, 1.9, 0.9 Hz, H6), 6.82 (1H, t, *J* = 2.1 Hz, H2), 6.67 (1H, td, *J* = 7.4, 1.3 Hz, H16), 6.55 (1H, ddd, *J* = 8.3, 7.2, 1.5 Hz, H15), 6.46 (1H, dd, *J* = 7.7, 1.5 Hz, H17), 3.79 (3H, s, H10). **^{13}C{^{1}H} NMR** (100 MHz, 25 °C, CD_2Cl_2) δ(ppm): 162.5 (C7), 160.4 (C3), 133.6 (C14), 130.5 (C12), 130.2 (C5), 130.1 (C1), 126.1 (C16), 124.4 (C15), 120.1 (C4), 118.9 (C6), 116.0 (C13), 116.0 (C17), 113.4 (C2), 55.9 (C10). **^{19}F NMR** (376 MHz, 25 °C, CD_2Cl_2) δ(ppm):

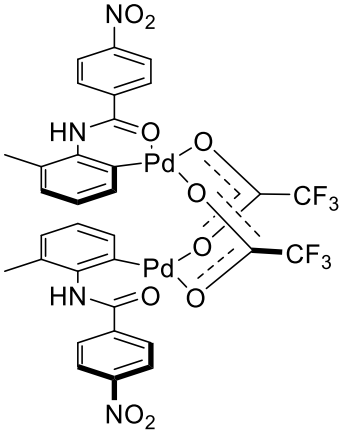
-74.3, -74.9, -75.0. **IR** (ATR): *v* = 1668, 1585, 1560, 1538, 1489, 1461, 1434, 1258, 1242, 1199, 1150, 1042, 790, 751, 733 cm^{-1}. **HRMS** calculated for $C_{16}H_{15}N_2O_2Pd$ $[M+CH_3CN]^+$ 373.0168 found: 373.0176.

Bis(2-(4-methoxybenzamido)-3-methylphenyl)bis[*μ*-(trifluoroacetato)]-dipalladium(II) (42)



The general procedure was followed using 1 equiv of *4-methoxy-N-(o-tolyl)benzamide* (121 mg, 0.5 mmol). Yellow solid, 144 mg (0.157 mmol, 63%), **MP**: 230-231 °C. Two conformers in 74:26 ratio. **^{1}H NMR** (400 MHz, 25 °C, CD_2Cl_2) δ(ppm): 8.05 (1H, s, H8), 8.03 (1H, s, H8'), 7.42 (2H, d, *J* = 8.8 Hz, H14' and H18'), 7.20 (2H, d, *J* = 8.8 Hz, H14 and H18)), 6.84 (2H, d, *J* = 7.2 Hz, H15 and H17), 6.75 (1H, d, *J* = 7.2 Hz, H6'), 6.70 (2H, d, *J* = 8.5 Hz, H15' and H17'), 6.57 (1H, d, *J* = 7.1 Hz, H6), 6.50 (1H, t, *J* = 7.6 Hz, H1), 6.42 (1H, t, *J* = 7.6 Hz, H1'), 6.27 (1H, d, *J* = 8.0 Hz, H2'), 3.87 (3H, s, H21), 3.78 (3H, s, H21'), 2.16 (3H, s, H19'), 2.05 (3H, s, H19). **^{13}C{^{1}H} NMR** (100 MHz, 25 °C, CD_2Cl_2) δ(ppm): 164.4 (C16), 164.3 (C16'), 162.3 (C10), 162.2 C10'), 132.2 (C2'), 131.8 (C2), 129.4 (C14, C14', C18 and C18'), 129.3 (C4'), 129.0 (C4), 128.0 (C6), 127.8 (C6'), 123.9 (C1), 123.5 (C1'), 122.3 (C5'), 122.2 (C5), 121.3 (C13), 121.1 (C13'), 116.8 (C3'), 116.6 (C3), 114.9 (C15' and C17'), 114.8 (C15 and C17), 56.3 (C21), 56.1 (C21'), 18.0 (C19 and C19'). **^{19}F NMR** (376 MHz, 25 °C, CD_2Cl_2) δ(ppm): -74.4, -74.9, -75.0. **IR** (ATR): *v* = 1734, 1508, 1447, 1374, 1266, 1247, 1204, 1180, 1148, 1046, 1029, 897, 733, 705, 660 cm^{-1}. **HRMS** calculated for $C_{17}H_{17}N_2O_2Pd$ $[M+CH_3CN]^+$ 387.0325 found: 387.0311.

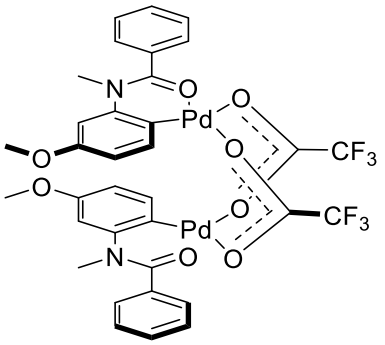
Bis(3-methyl-2-(4-nitrobenzamido)phenyl)bis[*μ*-(trifluoroacetato)]-dipalladium(II) (43)



The general procedure was followed using 1 equiv of *4-nitro-N-(o-tolyl)benzamide* (128 mg, 0.5 mmol).Yellow solid, 161 mg (0.170 mmol, 68%), **MP**: 231-232 °C. **^{1}H NMR** (400 MHz,

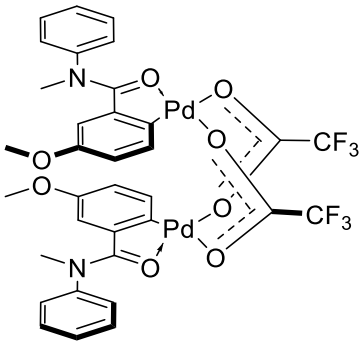
25 °C, CD_2Cl_2) δ(ppm): 8.34 (1H, s, H8), 8.21 (2H, d, *J* = 8.3 Hz, H, H14 and H18), 7.44 (2H, d, *J* = 8.3 Hz, H15 and H17), 6.92 (1H, d, *J* = 8.0 Hz, H2), 6.65 (1H, d, *J* = 7.2 Hz, H6), 6.55 (1H, t, *J* = 7.7 Hz, H1), 2.20 (3H, s, H19). **$^{13}C\{^1H\}$ NMR** (100 MHz, 25 °C, CD_2Cl_2) δ(ppm): 151.0, 134.4, 132.3 (C2), 128.7 (C15 and C17), 128.3 (C6), 125.6, 125.5, 124.7 (C14 and C18), 122.7, 18.2 (C19). Due to the insolubility, only the CHs have been assigned. **^{19}F NMR** (376 MHz, 25 °C, CD_2Cl_2) δ(ppm): -74.9. **IR** (ATR): *ν* = 1668, 1598, 1525, 1447, 1348, 1320, 1199, 1156, 1107, 856, 733, 716, 707, 667, 662 cm^{-1}. **HRMS** calculated for $C_{16}H_{14}N_3O_3Pd$ $[M+CH_3CN]^+$ 402.0070 found: 402.0056.

Bis(4-methoxy-2-(*N*-methylbenzamido)phenyl)bis[*μ*-(trifluoroacetato)]-dipalladium(II) (44)



The general procedure was followed using 1 equiv of *N-(3-methoxyphenyl)-N-methylbenzamide* (121 mg, 0.5 mmol). Brown solid, 125 mg (0.136 mmol, 54%), **MP**: 184-189 °C. Two conformers in 74:26 ratio. 1H NMR (400 MHz, 25 °C, CD2Cl2) δ(ppm): 7.41 (1H, t, J = 7.5 Hz, H12), 7.29 (2H, d, J = 7.6 Hz, H10 and H14), 7.15 (2Ht, J = 7.7 Hz, H11 and H13), 6.39 (1H, dd, J = 8.7, 2.6 Hz, H4), 6.33 (1H, d, J = 2.6 Hz, H6), 6.22 (1H, d, J = 8.7 Hz, H3), 3.74 (3H, s, H17), 3.04 (3H, s, H18). 13C{1H} NMR (100 MHz, 25 °C, CD2Cl2) δ(ppm): 172.0 (C8), 158.9 (C5), 137.4 (C2), 134.0 (C3), 132.7 (C12), 131.9 (C9), 129.6 (c10 and C14), 128.9 (C11 and C13), 110.6 (C2 and C4), 103.7 (C6), 56.1 (C17), 41.2 (C18). 19F NMR (376 MHz, 25 °C, CD2Cl2) δ(ppm): -74.3, 74.9, 75.1. **IR** (ATR): *ν* = 1665, 1586, 1575, 1536, 1465, 1447, 1411, 1195, 1150, 1066, 794, 733, 720, 708, 699 cm^{-1}. **HRMS** calculated for $C_{17}H_{17}N_2O_2Pd$ $[M+CH_3CN]^+$ 387.0325 found: 387.0315. **XRD**: See below.

Bis(4-methoxy-2-(methyl(phenyl)carbamoyl)phenyl)bis[*μ*-(trifluoroacetato)]-dipalladium(II) (45)

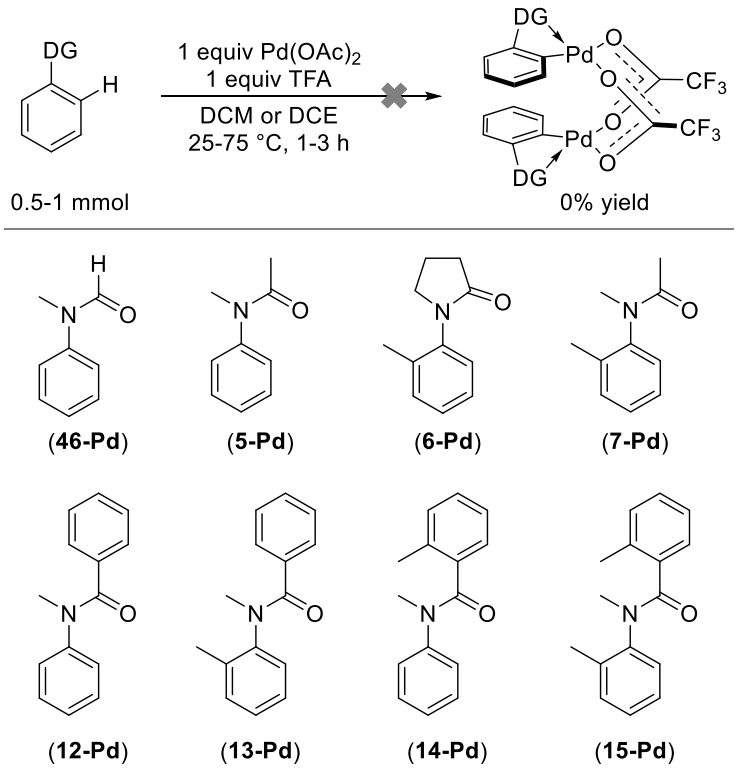


Described.[14] The general procedure was followed using 1 equiv of *3-methoxy-N-methyl-N-phenylbenzamide* (121 mg, 0.5 mmol). Yellow solid, 134 mg (0.141 mmol, 57%), **MP**: 192-195 °C. Two conformers in 64:36 ratio**. ^1H NMR** (400 MHz, 25 °C, CD_2Cl_2) δ(ppm): 7.65-7.26(5H, m,

[14] Borduas, N.; Lough, A. J.; Dong, V. M. Cyclopalladation of *N*-phenylbenzamides: Synthesis and structure of bimetallic palladium(II)-complexes. *Inorg. Chim. Acta* **2011**, *369*, 247–252.

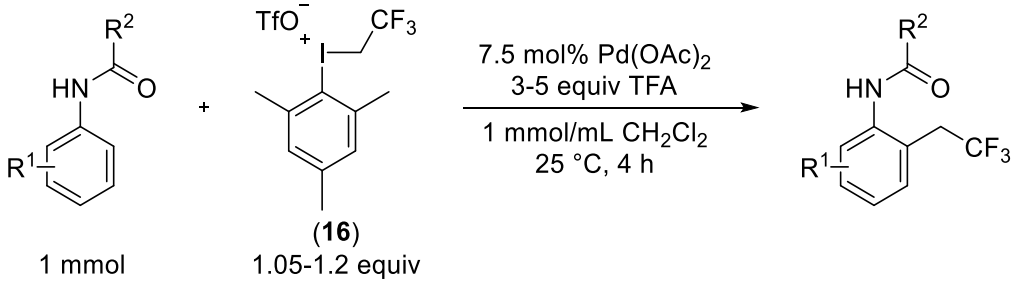
H14, H15, H16, H17 and H18), 6.77 (1H, d, J = 8.7 Hz, H3), 6.69 (1H, dd, J = 8.7, 2.8 Hz, H4), 5.22 (1H, d, J = 2.8 Hz, H6),3.28 (3H, s, H11) 3.04 (3H, s, H19). **^{13}C{^{1}H} NMR** (100 MHz, 25 °C, CD_2Cl_2) δ(ppm): 177.8 (C7), 156.4 (C5), 142.0 (C13), 138.5 (C1), 133.1 (C3), 131.1 (C15 and C17), 130.1 (C16), 126.9 (C14 and C18), 119.6 (C4), 114.4 (C6), 56.5 (C11), 40.7 (C19). **^{19}F NMR** (376 MHz, 25 °C, CD_2Cl_2) δ(ppm): -74.3, 74.9, 75.0. **IR** (ATR): *ν* = 1667, 1536, 1495, 1465, 1447, 1435, 1232, 1202, 1161, 1132, 1027, 850, 759, 731, 699 cm^{-1}. **HRMS** calculated for $C_{17}H_{17}N_2O_2Pd$ $[M+CH_3CN]^+$ 387.0325 found: 387.0322. **XRD**: See below.

Unsuccessful attempts for Pd-complex isolation



SI Scheme 10.

Synthesis of the *ortho* trifluorethylated acetanilides and aromatic amides



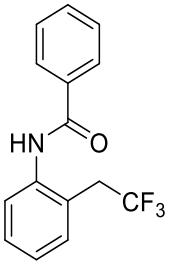
SI Scheme 11.

As previously described[12]: A 4 mL screw-cap vial charged with *palladium(II) acetate* (0.075 mmol, 7.5 mol%, 16.8 mg), the *amide derivatives* (1 mmol) and *mesityl(2,2,2-trifluoroethyl)iodonium trifluoromethanesulfonate* (**16**, 1.05-1.2 equiv.)[15] then equipped with a stirring bar. 1 mL

[15] 1.05 equiv. iodonium salt used if starting material was substituted 2,6-H acetanilide, to avoid the formation of bis(trifluoroethylated) product.

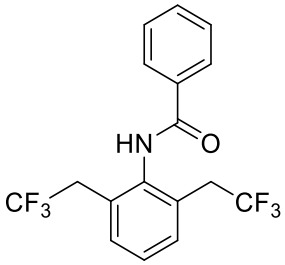
dichloromethane was added as solvent. The *trifluoroacetic acid* (3-5 equiv.)[16] was added dropwise instantly and the mixture was stirred at room temperature for 1.5-3 hours. After the appropriate time mixture was diluted with 25 mL ethyl-acetate and extracted with 2×10 mL cc. $NaHCO_3$ solution and with 10 mL brine. The organic phase solution was dried over $MgSO_4$ and evaporated on to Celite in rotary evaporator. The crude product was purified by gradient flash-column chromatography (hexane : ethyl acetate eluent). The product was dried overnight under reduced pressure (25 °C).

***N*-(2-(2,2,2-trifluoroethyl)phenyl)benzamide (8m)**



The general procedure was followed using 1 equiv *N-phenylbenzamide* (**24**, 394 mg, 2 mmol), 3 equiv TFA (464 µl, 6 mmol) and 1.05 equiv iodonium salt **16** (1014 mg, 2.1 mmol). White solid, 484 mg (1.73 mmol, 87%). **MP:** 178-179 °C. **^{1}H NMR** (400 MHz, 25 °C, DMSO-*d6*) δ(ppm): δ 10.11 (1H, s, H7), 7.96 (2H, dd, *J* = 8.0, 1.2 Hz, H10 and H14), 7.61 (1H, tt, *J* = 7.4, 1.3 Hz, H12), 7.56 – 7.52 (2H, m, H11 and H13), 7.44 (1H, d, *J* = 7.3 Hz, H3), 7.43 – 7.39 (2H, m, H5 and H6), 7.31 (1H, ddd, *J* = 7.5, 6.3, 2.5 Hz, H4), 3.74 (2H, q, *J* = 11.5 Hz, H15). **^{13}C{^{1}H} NMR** (100 MHz, 25 °C, DMSO-*d6*) δ(ppm): 165.7 (C8), 137.3 (C1), 134.3 (C9), 131.7 (C12), 131.6 (C3), 128.6 (C5), 128.4 (C11 and C13), 127.7 (C6), 127.7 (q, *J* = 275 Hz, C16), 127.6 (C10 and C14), 126.3, (C2 and C4) 34.9 (q, *J* = 29 Hz, C15). **^{19}F NMR** (376 MHz, 25 °C, DMSO-*d6*) δ(ppm): -63.6 (t, *J* = 11.5 Hz). **HRMS** calculated for $C_{15}H_{13}NOF_3$ $[M+H]^+$ 280.0949 found: 280.0944. **MS** (EI, 70 eV) m/z (%): 280(3), 279(16), 106(8), 105(100), 77(42). **IR** (ATR) *v* = 1642, 1521, 1448, 1363, 1307, 1297, 1251, 1242, 1130, 1109, 1066, 762, 723, 692, 654 cm^{-1}.

***N*-(2,6-bis(2,2,2-trifluoroethyl)phenyl)benzamide (8b)**

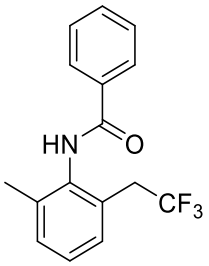


The general procedure was followed using 1 equiv *N-(2-(2,2,2-trifluoroethyl)phenyl)-benzamide* (**8m**, 285 mg ,1 mmol), 3 equiv TFA (232 µl, 3 mmol) and 1.05 equiv iodonium salt **16** (507 mg, 1.05 mmol). White solid, 294 mg (0.814 mmol, 81%). **MP:** 212-218 °C. **^{1}H NMR** (400 MHz, 25 °C, DMSO-*d6*) δ(ppm): δ 10.15 (1H, s, H10), 7.96 (2H, dd, *J* = 7.9, 1.4 Hz, H13 and H17), 7.62 (1H, tt, *J* = 7.3, 1.3 Hz, H15), 7.56 (2H, d, *J* = 7.4 Hz, H14 and H16), 7.50 (2H, 5, *J* = 7.7 Hz, H3 and H5), 7.41 (1H, t, J = 7.7 Hz, H4), 3.68 (4H, br, H7 and H18). **^{13}C{^{1}H} NMR** (100 MHz, 25 °C, DMSO-*d6*) δ(ppm): 165.7 (C11), 137.0 (C1), 134.0 (C12), 131.8 (C3 and C5), 131.7 (C15), 129.3 (q, *J* = 2.9 Hz, C2 and C6), 128.5 (C14 and C16), 127.4 (C14 and C16), 127.4 (C4), 126.3 (q, *J* = 276 Hz, C8 and C19), 35.1 (q, *J* = 29 Hz, (C7 and C18). **^{19}F**

[16] 3 or 5 equiv. of trifluoroacetic acid was used to complete dissolution and ensure the homogeneity. In certain cases, the starting amides and in particular the trifluoroethylated products were not well soluble in DCM.

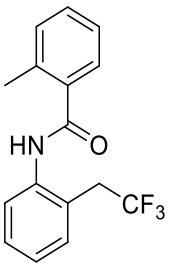
NMR (376 MHz, 25 °C, DMSO-*d6*) δ(ppm): -63.5 (t, *J* = 11.5 Hz). **HRMS** calculated for $C_{17}H_{14}NOF_6$ $[M+H]^+$ 362.0980 found: 362.0985. **MS** (EI, 70 eV) m/z (%): 361(9), 106(7), 105(100), 77(33). **IR** (ATR) *v* = 1642, 1516, 1489, 1361, 1277, 1253, 1238, 1210, 1137, 1109, 1091, 1066, 790, 744, 692 cm^{-1}.

***N*-(2-methyl-6-(2,2,2-trifluoroethyl)phenyl)benzamide (9m)**



As described[12]: The general procedure was followed using 1 equiv *N-(o-tolyl)benzamide* (**25**, 211 mg, 1 mmol) 3 equiv TFA (232 µl, 3 mmol) and 1.2 equiv iodonium salt **16** (580 mg, 1.2 mmol) and stirred for 3 hours. The product recrystallized from aceton-hexan, the solid collected then dried. White solid 239 mg (0.80 mmol, 71%). Rf = 0.38 (hexane: ethyl acetate = 5:2). **MP**: 184-185 °C. **^{1}H NMR** (400 MHz, 25 °C, DMSO-*d6*) δ(ppm): δ 9.92 (1H, s, H8), 7.98 (2H, dd, *J* = 7.3, 1.5 Hz, H11 and H15), 7.60 (1H, tt, *J* = 7.3, 1.5 Hz, H13), 7.54 (2H, t, *J* = 7.3 Hz, H12 and H14), 7.32 (1H, dd, *J* = 7.0, 1.3 Hz, H5), 7.30 (1H, dd, *J* = 6.9, 1.3 Hz, H3), 7.27 (1H, t, *J* = 7.0 Hz, H4), 3.61 (2H, q, *J* = 11.6 Hz, H16), 2.22 (3H, s, H7). **^{13}C{^{1}H} NMR** (100 MHz, 25 °C, DMSO-*d6*) δ(ppm): 165.4 (C9), 136.6 (C6), 136.2 (C1), 134.2 (C10), 131.6 (C13), 130.3 (C5), 129.0 (C3), 128.5 (12 and C14), 128.4 (q, *J* = 2.7 Hz, C2), 127.5 (C11 and C15), 127.0 (C4), 126.2 (q, *J* = 270 Hz, C17), 35.0 (q, *J* = 29 Hz, H16), 18.2 C7). **^{19}F NMR** (376 MHz, 25 °C, DMSO-*d6*) δ(ppm): -63.5 (t, *J* = 11.5 Hz).**MS** (EI, 70 eV): m/z (%): 338(20), 188(26), 151(8), 150(100), 120(11), 104(36), 92(15), 76(17). **HRMS** calculated for $C_{16}H_{14}N_2O_3F_3$ $[M+H]^+$ 339.0957 found: 339.0959. **IR** (ATR) *v* = 1686, 1652, 1607, 1559, 1529, 1507, 1484, 1350, 1294, 1242, 1137, 1108, 1078, 858, 656 cm^{-1}.

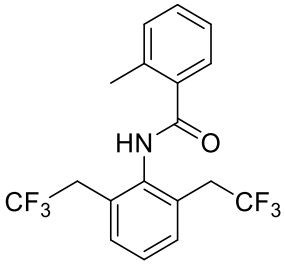
2-methyl-*N*-(2-(2,2,2-trifluoroethyl)phenyl)benzamide (10m)



The general procedure was followed using 1 equiv *2-methyl-N-phenylbenzamide* (**26**, 423 mg, 2 mmol), 3 equiv TFA (464 µl, 6 mmol) and 1.05 equiv iodonium salt **16** (1014 mg, 2.1 mmol). White solid, 494 mg (1.68 mmol, 84%). **MP:** 205-210 °C. **^{1}H NMR** (400 MHz, 25 °C, DMSO-*d6*) δ(ppm): δ 10.01(1H, s, H10), 7.51 (1H, d, *J* = 7.7 Hz, H6), 7.49-7.37 (4H, m, H16, H4, H13 and H15), 7.34-7.30 (2H, m, H5 and H3), 7.28 (1H, dt, *J* = 7.4, 1.3 Hz, H14), 3.77 (2H, q, J = 11.5 Hz, H17), 2.40 (3H, s, H9). **^{13}C{^{1}H} NMR** (100 MHz, 25 °C, DMSO-*d6*) δ(ppm): 168.2 (H7), 137.1 (C11), 136.9 (C1), 135.3 (C2), 131.8 (C13), 130.5 (C3), 129.6 (C15), 128.6 (C4), 127.2 (C6), 127.0 (C16), 126.5 (q, *J* = 276 Hz, C18), 126.1 (C14), 125.6 (C5), 125.5 (q, *J* = 3 Hz, C12), 34.7 (q, J = 29 Hz, H17), 19.0 (C9). **^{19}F NMR** (376 MHz, 25 °C, DMSO-*d6*) δ(ppm):

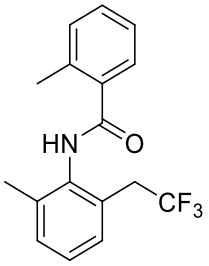
-63.7 (t, *J* = 11.5 Hz). **HRMS** calculated for $C_{16}H_{15}NOF_3$ $[M+H]^+$ 294.1106 found: 294.1104. **MS** (EI, 70 eV) m/z (%): 294(2), 293(13), 120(9), 119(100), 91(35), 65(10). **IR** (ATR) *v* = 1652, 1523, 1454, 1447, 1363, 1307, 1251, 1133, 1120, 1068, 755, 738, 721, 701, 656 cm^{-1}.

***N*-(2,6-bis(2,2,2-trifluoroethyl)phenyl)-2-methylbenzamide (10b)**



The general procedure was followed using 1 equiv *2-methyl-N-(2-(2,2,2-trifluoroethyl)phenyl)benzamide* (**10m**, 293 mg, 1 mmol), 3 equiv TFA (232 µl, 3 mmol) and 1.05 equiv iodonium salt **16** (507 mg, 1.05 mmol). White solid, 267 mg (0.711 mmol, 71%). **MP:** 230-232 °C. **^{1}H NMR** (400 MHz, 25 °C, DMSO-*d6*) δ(ppm): δ 10.14 (1H, s, H10), 7.49 (2H, d, *J* = 7.5 Hz, H3 and H5), 7.45 (1H, dd, *J* = 7.2, 1.3 Hz, H17), 7.41 (1H, m, H15), 7.37 (1H, m, H16), 7.33 (1H, d, *J* = 7.6 Hz, H14), 3.68 (4H, br, H7 and H18), 2.41 (3H, s, H24). **^{13}C{^{1}H} NMR** (100 MHz, 25 °C, DMSO-*d6*) δ(ppm): 168.0 (C11), 136.8 (C1), 136.2 (C12), 135.6 (C13), 131.9 (C3 and C5), 130.8 (C14), 130.3 (C14), 129.9 (C15), 128.9 (q, *J* = 2.9 Hz, C2 and C6), 127.3 (C4), 126.8 (C17), 126.4 (q, *J* = 276 Hz, C8 and C19), 125.7 (C16), 35.1 (q, *J* = 29 Hz, (C7 and C18), 19.0 (C24). **^{19}F NMR** (376 MHz, 25 °C, DMSO-*d6*) δ(ppm): -63.6 (t, *J* = 11.5 Hz). **HRMS** calculated for $C_{18}H_{16}NOF_6$ $[M+H]^+$ 376.1136 found: 376.1129. **MS** (EI, 70 eV) m/z (%):375(4), 120(8), 119(100), 91(31), 65(8) . **IR** (ATR) *v* = 1652, 1514, 1445, 1361, 1277, 1251, 1234, 1210, 1137, 1115, 1089, 1068, 790, 744, 658 cm^{-1}.

2-methyl-*N*-(2-methyl-6-(2,2,2-trifluoroethyl)phenyl)benzamide (11m)

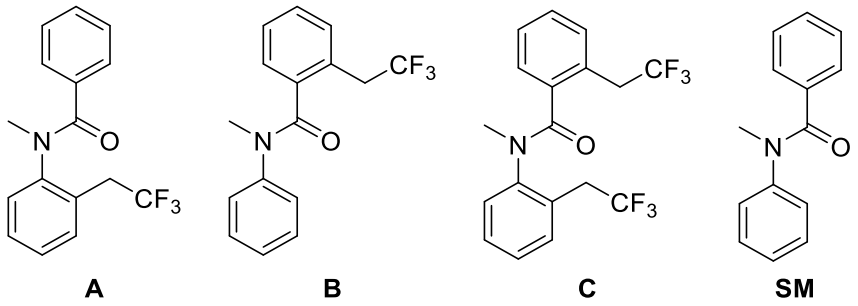


The general procedure was followed using 1 equiv *2-methyl-N-(o-tolyl)benzamide* (**27**, 225mg, 1 mmol), 3 equiv TFA (232 µl, 3 mmol) and 1.2 equiv iodonium salt **16** (580 mg, 1.2 mmol). White solid, 269 mg (0.875 mmol, 88%). **MP:** 178-183 °C. **^{1}H NMR** (400 MHz, 25 °C, DMSO-*d6*) δ(ppm): δ 9.86 (1H, s, H8), 7.50 (1H, dd, *J* = 7.7, 1.5 Hz, H15), 7.40 (1H, td, *J* = 7.4, 1.4 Hz, H13), 7.36-7.27 (4H, m, H3, H5, H12 and H15), 7.25 (1H, t, *J* = 7.3 Hz, H4), 3.67 (2H, q, *J* = 11.5 Hz, H16), 2.43 (3H, s, H20), 2.29 (3H, s, H7).**^{13}C{^{1}H} NMR** (100 MHz, 25 °C, DMSO-*d6*) δ(ppm): 167.9 (C9), 136.7 (C10), 136.4 (C6), 136.0 (C1), 135.4 (C11), 130.6 (C12), 130.4 (C5), 129.6 (C13), 129.1 (C3), 128.1 (q, *J* = 2.9 Hz, C2), 127.0 (C15), 126.9 (C4), 126.1 (q, *J* = 267 Hz, C17),125.7 (C14), 35.0 (q, *J* = 29 Hz, C16), 19.2 (C20), 18.4 (C7). **^{19}F NMR** (376 MHz, 25 °C, DMSO-*d6*) δ(ppm): -63.5 (t, *J* = 11.4 Hz). **HRMS** calculated for $C_{17}H_{17}NOF_3$ $[M+H]^+$ 308.1262 found: 308.1263. **MS** (EI, 70 eV) m/z (%): 307(11), 120(9). 119(100), 118(2), 91(33), 65(9). **IR** (ATR) *v* = 1652, 1516, 1469, 1443, 1365, 1299, 1281, 1264, 1243, 1130, 1107, 1076, 781, 744, 707 cm^{-1}.

***N*-methyl-*N*-(2-(2,2,2-trifluoroethyl)phenyl)benzamide (12a)**

***N*-methyl-*N*-phenyl-2-(2,2,2-trifluoroethyl)benzamide (12b)**

***N*-methyl-2-(2,2,2-trifluoroethyl)-*N*-(2-(2,2,2-trifluoroethyl)phenyl)benzamide (12c)**



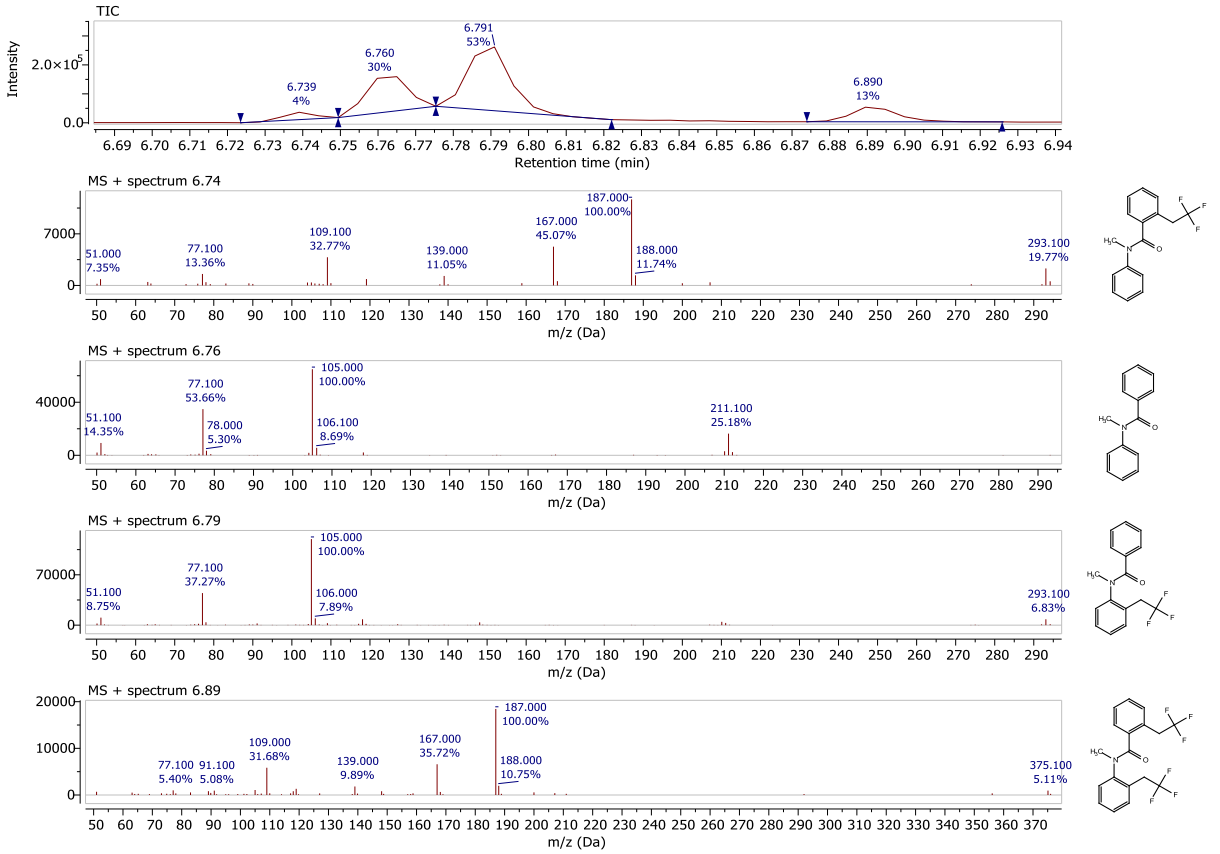
The general procedure was followed using 1 equiv *N-methyl-N-phenylbenzamide* (**28**), 5 equiv. TFA and 1.2 equiv. iodonium salt **16** (580 mg, 1.2 mmol). 71% of conversion was determined by GCMS (see on chromatogram). Two unseparable mixtures were isolated: P1: yellow solid 33 mg, P2 white solid, 171 mg. P1 contains 7% **A**, 60% **B**, 33 % **C**, and P2 contains 72% **A**, 0 % **B**, 21% **C** and 7% **SM**). However, the full characterisation was not possible by NMR measurements, the ratio of the **A**, **B**, **C** and their rotamers was determined by the integration of ^{1}H (methyl region) and ^{19}F single pulse experiments. The position of -CH_2-CF_3 was determined by ^{1}H-^{13}C HMBC measurements.

A (two rotamers in 73-27 ratio): **^{1}H NMR** (400 MHz, 25 °C, DMSO-*d6*) δ(ppm): δ 3.59-3.45 (2H, m, H9 and H9'), 3.37 (3H, s, H8), 3.29 (3H, s, H8'). **^{13}C{^{1}H} NMR** (100 MHz, 25 °C, DMSO-*d6*) δ(ppm): 169.5 (C12 and C12'), 144.6 (C1), 143.9 (C1'), 136.3 (C16'), 135.9 (C16), 37.9 (C8), 37.8 (C8'), 33.8 (q, *J* = 30 Hz, C9 and C9'). **^{19}F NMR** (376 MHz, 25 °C, DMSO-*d6*) δ(ppm): -62.6 (t, *J* = 11.5 Hz, CF_3), -63.2 (t, *J* = 11.5 Hz, CF_3'). **MS** (EI, 70 eV) m/z (%): 293(6), 210(5), 106(8), 105(100), 77(37),51(9).

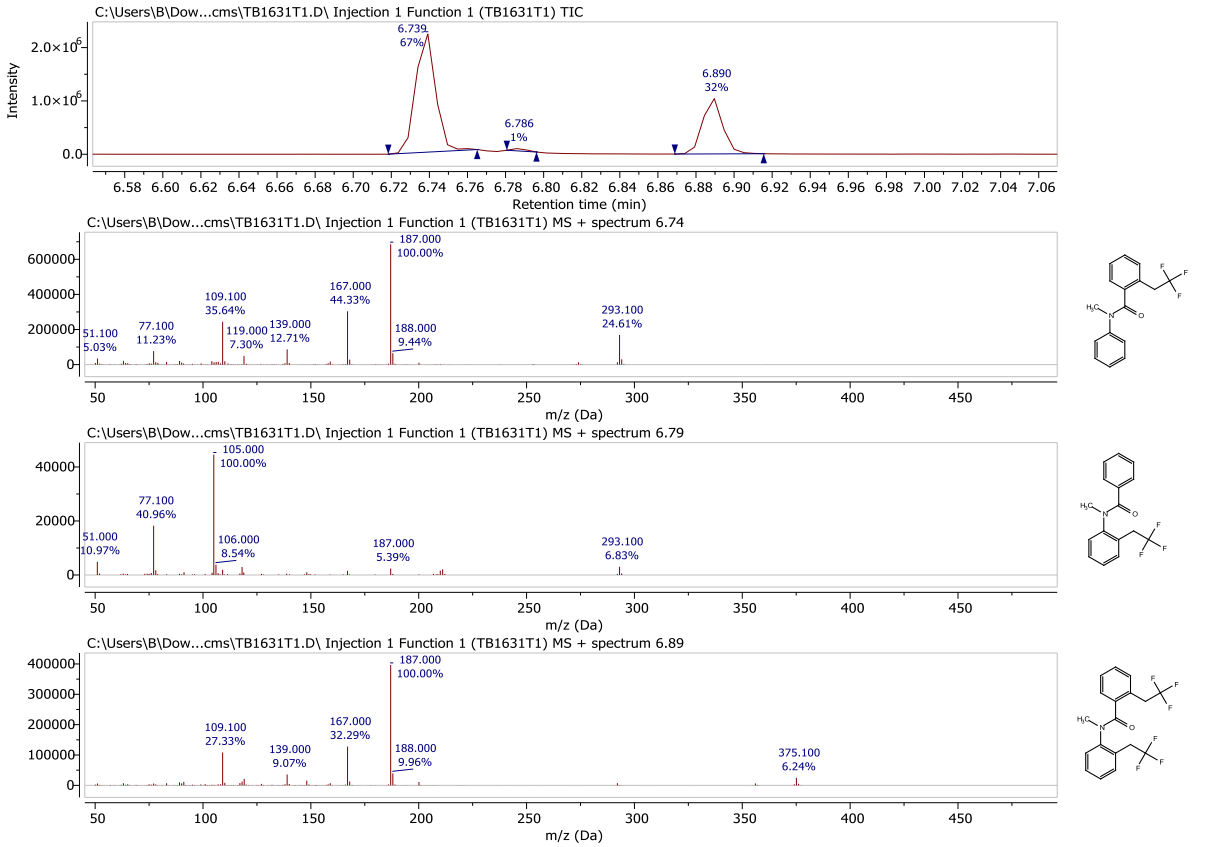
B (two rotamers in 85-15 ratio): **^{1}H NMR** (400 MHz, 25 °C, DMSO-*d6*) δ(ppm): δ 3.76, (2H, q, J = 11.5 Hz), H17 and H17'), 3.44-3.27 (3H, br, H8 and H8'). **^{13}C{^{1}H} NMR** (100 MHz, 25 °C, DMSO-*d6*) δ(ppm): 168.4 (C9 and C9'), 143.7 (C1 and C1'), 137.0 (C16), 136.4 (C16'), 37.2 (C8 and C8'), 35.4 (g, *J* = 30 Hz, C9 and C9'). **^{19}F NMR** (376 MHz, 25 °C, DMSO-*d6*) δ(ppm): -62.7 (br, CF_3), -63.9 (t, *J* = 11.5 Hz, CF_3'). **MS** (EI, 70 eV) m/z (%):294 (4), 293(3), 188(9), 187(100), 167(44), 139(13), 119(7), 109(36), 77(11), 51(5).

C (two rotamers in 70-30 % ratio): **^{1}H NMR** (400 MHz, 25 °C, DMSO-*d6*) δ(ppm): δ 3.72 (4H, m, H9, H9', H22 and H22'), 3.29 (3H, s, H8'), 3.10 (3H, s, H8). **^{13}C{^{1}H} NMR** (100 MHz, 25 °C, DMSO-*d6*) δ(ppm): 169.9 (C12), 168.8 (C12'), 143.4 (C1'), 142.8 (C1), 136.9 (C16), 136.3 (C16'), 40.3 (C8), 37.5 (C8'), 35.3 (q, *J* = 30 Hz, C9 and C9'), 34.2 (q, *J* = 30 Hz, C22 and C22'). **^{19}F NMR** (376 MHz, 25 °C, DMSO-*d6*) δ(ppm): -62.1 (t, *J* = 11.5 Hz, CF_3), -62.4 (t, *J* = 11.5 Hz, CF_3'),63.5 (br, CF_3), -64.2(t, *J* = 11.5 Hz, CF_3'). **MS** (EI, 70 eV) m/z (%): 375(7), 188(10), 187(100), 167(32), 139(9), 109(26).

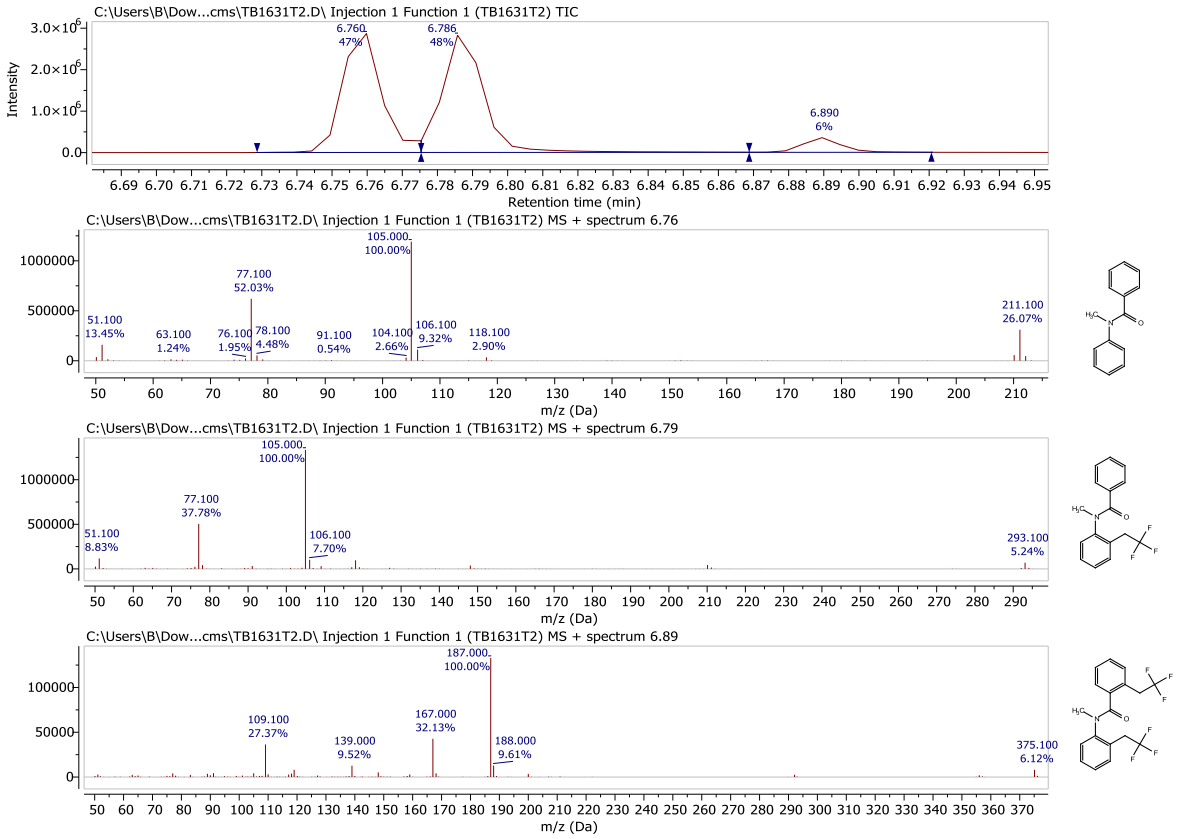
GCMS Chromatogram of Extractum of 12



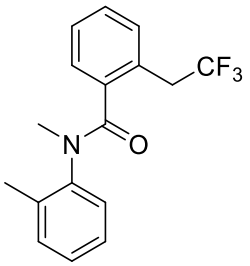
GCMS Chromatogram of 12p1



GCMS Chromatogram of 12p2



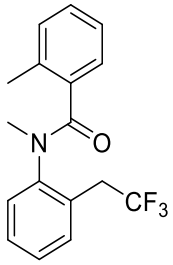
***N*-methyl-*N*-(*o*-tolyl)-2-(2,2,2-trifluoroethyl)benzamide (13m)**



The general procedure was followed using 1 equiv *N-methyl-N-(o-tolyl)benzamide* (**29**, 225 mg, 1 mmol), 5 equiv TFA (387 µl, 5 mmol) and 1.2 equiv iodonium salt **16** (580 mg, 1.2 mmol). Colourless oil, 141 mg (0.459 mmol, 46%). Two rotamers in 53:47 ratio. **^{1}H NMR** (400 MHz, 25 °C, D6-DMSO) δ(ppm): δ 7.62 (1H, m, H4), 7.57-7.50 (3H, m, H3, H4, H18), 7.36-7.26 (5H, m, H3',H6, H15, H16 and H17), 7.23 (1H, dd, *J* = 7.5, 1.4 Hz, H4'), 7.20 (1H, d, *J* = 7.3 Hz, H15'), 7.08 (1H, td, *J* = 7.3, 1.6 Hz, H16'), 7.04 (1H, td, *J* = 7.6, 1.3 Hz, H5'), 6.98 (1H, td, *J* = 7.0, 1.2 Hz, H17'), 6.95 (1H, dd, *J* = 7.5, 1.2 Hz, H18'), 6.93 (1H, dd, *J* = 7.3, 1.2 Hz, H6'), 3.95(1H, dq, 15.1, 11.4 Hz, H9'a), 3.86-3.71 (2H, m, H9), 3.70 (1H, dq, 15.1, 11.4 Hz, H9'b), 3.26 (3H, s, H21'), 3.06 (3H, s, H21), 2.30 (3H, s, H22'), 2.27 (3H, s, H22). **^{13}C NMR** (151 MHz, D6-DMSO) δ 168.7(C7), 168.6 (C7'), 142.4 (C13), 141.7 (C13'), 137.4 (C1), 136.6 (C1'), 135.1 (C14), 134.4 (C14'), 132.1 (C3), 131.1 (C15'), 130.8 (C3'), 130.8 (C15), 129.4 (C4), 129.0 (C4'), 128.2 (C2), 128.2 (C2', C2 and C18'), 127.6 (C16 and H16')), 127.3 (H6'), 127.2 (C6), 127.1 (C5 and H5'), 126.9 n(C17), 126.7 (C17'), 126.1 (q, *J* = 270 Hz, C10 and

C10')) 40.0 (C21), 36.5 C21'), 35.6 (q, *J* = 270 Hz, C9'), 35.3 (q, *J* = 270 Hz, C9), 17.5 (C22'), 17.0 (C22). **^{19}F NMR** (376 MHz, 25 °C, D6-DMSO) δ(ppm): -62.4 (t, *J* = 11.5 Hz), -63.9 (t, *J* = 11.5 Hz). **HRMS** calculated for $C_{17}H_{17}NOF_3$ $[M+H]^+$ 308.1262 found: 308.1263. **MS** (EI, 70 eV) m/z (%): 308(4), 307(19), 306(2), 200(11), 188(10), 187(100), 168(4), 167(39), 138(11), 119(6), 109(31), 91(9), 65(6). **IR** (ATR) *v* = 1639, 1495, 1435, 1361, 1256, 1133, 1092, 1070, 1055, 785, 762, 749, 725, 699, 660 cm^{-1}.

***N*,2-dimethyl-*N*-(2-(2,2,2-trifluoroethyl)phenyl)benzamide (14m)**



The general procedure was followed using 1 equiv *N,2-dimethyl-N-phenylbenzamide* (**30**, 225 mg, 1 mmol), 5 equiv TFA (387 µl, 5 mmol) and 1.2 equiv iodonium salt **16** (580 mg, 1.2 mmol). White solid, 106 mg (0.341 mmol, 34%). **MP:** 112-116 °C. Two rotamers in 59:41 ratio**. ^{1}H NMR** (400 MHz, 25 °C, D6-DMSO) δ(ppm): δ 7.54-7.48 (3H, m,H4', H5' and H6'), 7.43 (1H, m, H13), 7.39-7.26 (6H, m, H5, H6, H11, H11', H12, H14), 7.24 (1H, td, *J* = 7.4, 1.8 Hz, H3'), 7.22 (1H, td, *J* = 7.4, 1.8 Hz, H3'), 7.07 (1H, td, *J* = 7.3, 1.6 Hz, H4), 7.05 (1H, td, *J* = 7.7, 1.3 Hz, H12'), 6.93 (1H, td, *J* = 7.5, 1.3 Hz, H14'), 6.87 (1H, td, *J* = 7.6, 1.8 Hz, H13'), 3.78(4H, m, H15 and H15'), 3.29 (3H, s, H17'), 3.03 (3H, s, H17), 2.35 (3H, s, H19), 2.30 (3H, s, H17). **^{13}C NMR** (151 MHz, 25 °C D6-DMSO) δ 170.2 (C8), 169.9 (C8'), 143.3 (C1'), 142.9 (C1), 136.4 (C10), 135.9 (C10'), 134.5 (C9'), 133.4 (C9), 131.6, 130.8, 130.4, 130.1, 129.7, 129.1, 129.0, 129.0, 128.5, 128.1, 127.9, 127.8, 127.8, 127.6, 127.1, 127.0, 126.3, 126.0, 125.5, 125.3, 125.2, 124.6, 40.0 (C17), 37.1 (C17'), 33.7 (q, *J* = 29.1 Hz, C15), 33.7(q, *J* = 29.1 Hz, C15'), 19.8 (C19), 18.1 (C19'). **^{19}F NMR** (376 MHz, 25 °C, D6-DMSO) δ(ppm): -62.4 (t, *J* = 11.5 Hz), -63.3 (t, *J* = 11.5 Hz). **HRMS** calculated for $C_{17}H_{17}NOF_3$ $[M+H]^+$ 308.1262 found: 308.1261. **MS** (EI, 70 eV) m/z (%): 307(5), 224(1), 148(2), 120(9), 119(100), 118(3), 91(34), 65(10). **IR** (ATR) *v* = 1709, 1646, 1435, 1422, 1361, 1253, 1223, 1200, 1137, 1094, 761, 746, 731, 690, 662 cm^{-1}.

X-Ray Crystallography

Crystal data, data collection and structure refinement details are summarized in SI Table 1.

	(4-Pd)	(8-Pd)	(44)	(61)
Crystal data				
Chemical formula	$C_{26}H_{24}F_6N_2O_6Pd_2$	$C_{30}H_{20}F_6N_2O_6Pd_2 \cdot C_3H_6O$	$C_{34}H_{26}F_6N_2O_8Pd_2$	$C_{17}H_{14}F_3NO_4Pd$
M_r	787.27	889.36	917.37	459.69
Crystal system, space group	Monoclinic, $P2_1/n$	Monoclinic, $P2_1/c$	Monoclinic, $C2/c$	Monoclinic, $C2/c$
Temperature (K)	295	294	295	295
a, *b*, *c* (Å)	17.9432 (6), 15.8649 (5), 21.6563 (7)	19.2535 (7), 8.0799 (4), 21.9245 (10)	32.598 (4), 12.5843 (11), 22.685 (3)	20.9754 (8), 13.1973 (6), 15.3039 (11)
a, b, g (°)	90, 114.273 (1), 90	90, 93.765 (2), 90	90, 128.447 (3), 90	90, 125.008 (1), 90
V (Å^3)	5619.8 (3)	3403.4 (3)	7288.2 (13)	3469.9 (3)
Z	8	4	8	8
Radiation type	Mo *K*a	Mo *K*a	Mo *K*a	Mo *K*a
m (mm^{-1})	1.36	1.14	1.07	1.12
Crystal size (mm)	0.46 × 0.18 × 0.15	0.26 × 0.20 × 0.17	0.34 × 0.16 × 0.06	0.46 × 0.14 × 0.06
Data collection				
Diffractometer	Bruker D8 VENTURE	Bruker D8 VENTURE	Bruker D8 VENTURE	Bruker D8 VENTURE
Absorption correction	Multi-scan *SADABS2016*/2 - Bruker AXS area detector scaling and absorption correction	Multi-scan *SADABS2016*/2 - Bruker AXS area detector scaling and absorption correction	Multi-scan *SADABS2016*/2 - Bruker AXS area detector scaling and absorption correction	Multi-scan *SADABS2016*/2 - Bruker AXS area detector scaling and absorption correction
T_{min}, T_{max}	0.72, 0.82	0.71, 0.83	0.85, 0.94	0.526, 0.745
No. of measured, independent and observed [$I > 2s(I)$] reflections	39605, 11018, 7945	38088, 6682, 5757	40549, 6671, 4487	48632, 3279, 2460
R_{int}	0.035	0.037	0.077	0.124
$(\sin q/l)_{max}$ (Å^{-1})	0.617	0.618	0.604	0.611
Refinement				
$R[F^2 > 2s(F^2)]$, $wR(F^2)$, S	0.052, 0.134, 1.06	0.051, 0.132, 1.12	0.068, 0.224, 1.05	0.050, 0.095, 1.04
No. of reflections	11018	6682	6671	3279
No. of parameters	762	459	472	238
No. of restraints	0	2	0	0
H-atom treatment	H-atom parameters	H atoms treated by a mixture of	H-atom parameters	H-atom parameters

	constrained	independent and constrained refinement	constrained	constrained
	$w = 1/[s^2(F_o^2) + (0.0484P)^2 + 14.3859P]$ where $P = (F_o^2 + 2F_c^2)/3$	$w = 1/[s^2(F_o^2) + (0.0424P)^2 + 15.6605P]$ where $P = (F_o^2 + 2F_c^2)/3$	$w = 1/[s^2(F_o^2) + (0.1122P)^2 + 63.548P]$ where $P = (F_o^2 + 2F_c^2)/3$	$w = 1/[s^2(F_o^2) + (0.0478P)^2 + 3.3885P]$ where $P = (F_o^2 + 2F_c^2)/3$
$D\rho_{max}$, $D\rho_{min}$ (e Å^{-3})	1.38, -0.86	1.18, -0.95	1.59, -0.99	0.74, -0.49
Absolute structure	–	–	–	–
Absolute structure parameter	–	–	–	–

	(9-Pd)	(11-Pd)
Crystal data		
Chemical formula	$C_{32}H_{24}F_6N_2O_6Pd_2 \cdot C_3H_6O$	$C_{34}H_{28}F_6N_2O_6Pd_2 \cdot C_3H_6O$
M_r	917.41	945.46
Crystal system, space group	Orthorhombic, *Fdd*2	Orthorhombic, *Fdd*2
Temperature (K)	298	299
a, *b*, *c* (Å)	37.712 (6), 13.024 (2), 15.254 (3)	37.337 (11), 13.419 (4), 15.197 (5)
a, b, g (°)	90, 90, 90	90, 90, 90
V (Å^3)	7492 (2)	7614 (4)
Z	8	8
Radiation type	Mo *K*a	Mo *K*a
m (mm^{-1})	1.04	1.02
Crystal size (mm)	0.30 × 0.09 × 0.03	0.67 × 0.08 × 0.05
Data collection		
Diffractometer	Bruker D8 VENTURE	Bruker D8 VENTURE
Absorption correction	Multi-scan *SADABS2016*/2 - Bruker AXS area detector scaling and absorption correction	Multi-scan *SADABS2016*/2 - Bruker AXS area detector scaling and absorption correction
T_{min}, T_{max}	0.74, 0.97	0.65, 0.95
No. of measured, independent and observed [$I > 2s(I)$] reflections	16999, 3350, 2121	49149, 3648, 2153
R_{int}	0.081	0.230
$(\sin q/l)_{max}$ (Å^{-1})	0.603	0.612
Refinement		

$R[F^2 > 2s(F^2)]$, $wR(F^2)$, S	0.051, 0.138, 1.10	0.056, 0.173, 1.02
No. of reflections	3350	3648
No. of parameters	241	251
No. of restraints	4	2
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement	H atoms treated by a mixture of independent and constrained refinement
	$w = 1/[s^2(F_o^2) + (0.0627P)^2 + 3.5573P]$ where $P = (F_o^2 + 2F_c^2)/3$	$w = 1/[s^2(F_o^2) + (0.0708P)^2 + 58.4521P]$ where $P = (F_o^2 + 2F_c^2)/3$
$D\rho_{max}$, $D\rho_{min}$ (e Å^{-3})	0.47, -0.52	0.87, -0.67
Absolute structure	Flack x determined using 747 quotients [(I+)-(I-)]/[(I+)+(I-)] (Parsons, Flack and Wagner, Acta Cryst. B69 (2013) 249-259).	Flack x determined using 778 quotients [(I+)-(I-)]/[(I+)+(I-)] (Parsons, Flack and Wagner, Acta Cryst. B69 (2013) 249-259).
Absolute structure parameter	0.00 (3)	-0.02 (4)

Computer programs: Bruker Instrument Service vV6.2.6, *APEX3* v2017.3-0 (Bruker AXS), *SAINT* V8.38A (Bruker AXS Inc., 2017), SHELXT 2014/5 (Sheldrick, 2014), *SHELXL2018*/3 (Sheldrick, 2018), *SHELXL2016*/6 (Sheldrick, 2016), *Mercury*, *WinGX*, *publCIF*.

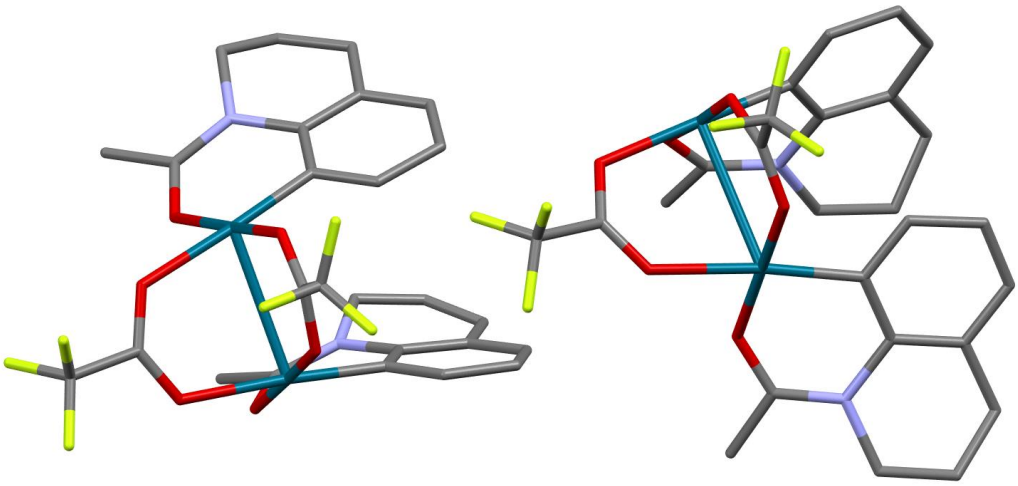
References

Document origin: Westrip, S. P. publCIF: software for editing, validating and formatting crystallographic information files. *J. Apply. Cryst.* **2010**, *43*, 920-925.

Computing details

For all compounds, data collection: Bruker Instrument Service vV6.2.6; cell refinement: *APEX3* v2017.3-0 (Bruker AXS); data reduction: *SAINT* V8.38A (Bruker AXS Inc., 2017); program(s) used to solve structure: SHELXT 2014/5 (Sheldrick, 2014). Program(s) used to refine structure: *SHELXL2018*/3 (Sheldrick, 2018) for 4-Pd, 8-Pd, 44; *SHELXL2016*/6 (Sheldrick, 2016) for 61, 9-Pd, 11-Pd. For all compounds, molecular graphics: *Mercury*; software used to prepare material for publication: *WinGX*, *publCIF*.

(4-Pd)



Crystal data

$C_{26}H_{24}F_6N_2O_6Pd_2$	$F(000)$ = 3104
M_r = 787.27	D_x = 1.861 Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation, l = 0.71073 Å
a = 17.9432 (6) Å	Cell parameters from 251 reflections
b = 15.8649 (5) Å	q = 3.1–20.8°
c = 21.6563 (7) Å	m = 1.36 mm^{-1}
b = 114.273 (1)°	T = 295 K
V = 5619.8 (3) Å^3	Prism, colourless
Z = 8	0.46 × 0.18 × 0.15 mm

Data collection

Bruker D8 VENTURE diffractometer	11018 independent reflections
Radiation source: microfocus sealed tube, INCOATEC ImS 3.0	7945 reflections with $I > 2s(I)$
Multilayer mirror INCOATEC monochromator	R_{int} = 0.035
Detector resolution: 7.3910 pixels mm^{-1}	q_{max} = 26.0°, q_{min} = 2.3°

w and p scan	h = -22→18
Absorption correction: multi-scan *SADABS2016*/2 - Bruker AXS area detector scaling and absorption correction	k = -19→19
T_{min} = 0.72, T_{max} = 0.82	l = -26→26
39605 measured reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2s(F^2)]$ = 0.052	H-atom parameters constrained
$wR(F^2)$ = 0.134	$w = 1/[s^2(F_o^2) + (0.0484P)^2 + 14.3859P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.06	$(D/s)_{max}$ = 0.001
11018 reflections	$D\rho_{max}$ = 1.38 e Å^{-3}
762 parameters	$D\rho_{min}$ = -0.86 e Å^{-3}
0 restraints	Extinction correction: *SHELXL2018*/3 (Sheldrick 2018)
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.00011 (8)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2) for (4-Pd)

	x	y	z	U_{iso}*/U_{eq}
C1	0.3364 (4)	0.3168 (4)	0.4369 (3)	0.0562 (15)
C2	0.4983 (4)	0.2934 (4)	0.3896 (3)	0.0630 (16)
C6	0.9973 (4)	0.2849 (4)	0.4071 (4)	0.0689 (18)
C7	0.8295 (4)	0.3257 (4)	0.4449 (4)	0.0648 (17)
C10	0.3435 (5)	0.3666 (5)	0.4984 (4)	0.076 (2)
C20	0.5787 (4)	0.3383 (5)	0.4281 (4)	0.075 (2)
C30	0.2802 (4)	0.3272 (4)	0.1506 (3)	0.0677 (17)
C31	0.1616 (5)	0.3945 (6)	0.0670 (4)	0.095 (3)
H31A	0.192856	0.444016	0.066149	0.114*

H31B	0.161863	0.356368	0.032136	0.114*
C32	0.0788 (6)	0.4197 (6)	0.0507 (4)	0.108 (3)
H32A	0.052464	0.433321	0.002816	0.13*
H32B	0.079084	0.470292	0.075937	0.13*
C33	0.0311 (5)	0.3538 (7)	0.0661 (4)	0.101 (3)
H33A	0.025929	0.304846	0.037836	0.121*
H33B	-0.023292	0.374656	0.056714	0.121*
C33A	0.0736 (4)	0.3295 (5)	0.1397 (4)	0.0739 (19)
C34	0.0276 (5)	0.3072 (5)	0.1744 (5)	0.091 (2)
H34	-0.029085	0.305671	0.151665	0.109*
C35	0.0635 (5)	0.2872 (6)	0.2422 (5)	0.092 (2)
H35	0.031899	0.270624	0.264983	0.111*
C36	0.1503 (5)	0.2923 (5)	0.2766 (4)	0.081 (2)
H36	0.175575	0.280639	0.322736	0.097*
C37	0.1960 (4)	0.3141 (4)	0.2428 (3)	0.0601 (15)
C37A	0.1572 (4)	0.3325 (4)	0.1731 (3)	0.0587 (16)
C39	0.3193 (5)	0.3358 (6)	0.1027 (4)	0.100 (3)
H39A	0.370065	0.305519	0.11979	0.15*
H39B	0.329387	0.394301	0.097809	0.15*
H39C	0.283685	0.313176	0.059513	0.15*
C40	0.3389 (6)	0.0666 (4)	0.2591 (4)	0.078 (2)
C41	0.2039 (7)	0.0675 (7)	0.1665 (4)	0.117 (3)
H41A	0.222521	0.105468	0.140629	0.141*
H41B	0.208769	0.010317	0.152597	0.141*
C42	0.1207 (7)	0.0846 (8)	0.1515 (5)	0.128 (4)
H42A	0.087969	0.069131	0.104752	0.154*
H42B	0.114431	0.144849	0.155532	0.154*
C43	0.0879 (6)	0.0406 (6)	0.1950 (5)	0.116 (3)
H43A	0.08781	-0.019736	0.187793	0.139*
H43B	0.032047	0.058436	0.183514	0.139*
C43A	0.1404 (5)	0.0610 (5)	0.2682 (5)	0.087 (2)
C44	0.1079 (6)	0.0625 (6)	0.3153 (6)	0.109 (3)
H44	0.053491	0.047347	0.302302	0.13*
C45	0.1533 (7)	0.0858 (6)	0.3810 (5)	0.112 (3)
H45	0.131269	0.082883	0.413075	0.135*
C46	0.2332 (5)	0.1141 (6)	0.3991 (4)	0.091 (2)
H46	0.263099	0.133725	0.442904	0.109*
C47	0.2675 (4)	0.1137 (4)	0.3551 (3)	0.0636 (17)
C47A	0.2215 (4)	0.0851 (4)	0.2877 (4)	0.0673 (18)
C49	0.3691 (7)	0.0337 (6)	0.2089 (5)	0.121 (4)

H49A	0.426469	0.021545	0.231439	0.182*
H49B	0.360257	0.075243	0.174366	0.182*
H49C	0.339987	-0.016926	0.188596	0.182*
C60	1.0797 (5)	0.3305 (5)	0.4344 (4)	0.0734 (19)
C70	0.8278 (6)	0.3950 (6)	0.4920 (4)	0.097 (3)
C81	0.8616 (7)	-0.1373 (5)	0.4324 (6)	0.129 (4)
H81A	0.906284	-0.16435	0.425805	0.155*
H81B	0.873133	-0.14106	0.480182	0.155*
C82	0.7904 (7)	-0.1805 (5)	0.3958 (5)	0.116 (4)
H82A	0.79317	-0.234063	0.418283	0.139*
H82B	0.789422	-0.19285	0.351657	0.139*
C84	0.6459 (6)	-0.0036 (6)	0.3850 (5)	0.099 (3)
H84	0.598089	-0.03374	0.375938	0.119*
C83	0.7168 (5)	-0.0467 (5)	0.3924 (4)	0.079 (2)
C83A	0.7133 (6)	-0.1415 (5)	0.3853 (6)	0.113 (3)
H83A	0.69429	-0.164881	0.417654	0.135*
H83B	0.673432	-0.155927	0.340307	0.135*
C85	0.6453 (5)	0.0813 (6)	0.3907 (4)	0.090 (2)
H85	0.597543	0.109499	0.385299	0.108*
C86	0.7211 (6)	0.1279 (5)	0.4056 (4)	0.084 (2)
H86	0.721952	0.18629	0.410024	0.1*
C87	0.7877 (4)	0.0876 (4)	0.4127 (3)	0.0675 (17)
C87A	0.7860 (5)	-0.0024 (4)	0.4052 (3)	0.0636 (17)
C80	0.9214 (5)	-0.0169 (5)	0.4030 (3)	0.075 (2)
C89	0.9889 (5)	-0.0710 (5)	0.4028 (4)	0.096 (3)
H89A	1.018494	-0.041413	0.381324	0.144*
H89B	0.966601	-0.122012	0.3784	0.144*
H89C	1.025191	-0.084408	0.448585	0.144*
C90	0.8042 (5)	0.1355 (5)	0.2227 (4)	0.0746 (19)
C91	0.7024 (6)	0.0259 (5)	0.2073 (5)	0.111 (3)
H91A	0.727977	-0.00003	0.18031	0.134*
H91B	0.720252	-0.004976	0.249591	0.134*
C92	0.6210 (8)	0.0175 (9)	0.1742 (13)	0.318 (16)
H92A	0.61196	-0.001054	0.129051	0.382*
H92B	0.606987	-0.030047	0.195528	0.382*
C93	0.5636 (6)	0.0741 (7)	0.1652 (6)	0.129 (4)
H93A	0.52364	0.050241	0.179728	0.155*
H93B	0.535793	0.08645	0.117221	0.155*
C94	0.5406 (5)	0.2104 (6)	0.2112 (5)	0.098 (3)
H94	0.485027	0.197961	0.191295	0.117*

C93A	0.5951 (5)	0.1552 (5)	0.2028 (4)	0.084 (2)
C95	0.5660 (5)	0.2822 (6)	0.2476 (4)	0.089 (2)
H95	0.528507	0.31832	0.253279	0.107*
C96	0.6503 (5)	0.3015 (5)	0.2769 (4)	0.077 (2)
H96	0.668087	0.351697	0.300544	0.093*
C97	0.7054 (4)	0.2475 (4)	0.2709 (3)	0.0634 (16)
C97A	0.6763 (4)	0.1721 (4)	0.2327 (3)	0.0628 (16)
C99	0.8506 (6)	0.0755 (6)	0.1984 (5)	0.114 (3)
H99A	0.89525	0.104584	0.194313	0.171*
H99B	0.814851	0.053294	0.155062	0.171*
H99C	0.871408	0.030131	0.230249	0.171*
N30	0.2036 (3)	0.3523 (3)	0.1343 (2)	0.0614 (13)
N40	0.2583 (4)	0.0775 (3)	0.2413 (3)	0.0743 (16)
N70	0.8589 (4)	-0.0464 (4)	0.4134 (3)	0.0728 (15)
N90	0.7326 (4)	0.1146 (4)	0.2227 (3)	0.0708 (15)
O1	0.3081 (3)	0.3551 (3)	0.3829 (2)	0.0711 (12)
O3	0.4452 (3)	0.3346 (3)	0.3457 (2)	0.0791 (14)
O2	0.3626 (3)	0.2436 (3)	0.4508 (2)	0.0698 (12)
O4	0.4943 (3)	0.2206 (3)	0.4085 (2)	0.0761 (13)
O5	0.9921 (3)	0.2275 (3)	0.4426 (3)	0.0919 (16)
O6	0.9471 (3)	0.3115 (3)	0.3531 (3)	0.0837 (14)
O7	0.8501 (3)	0.2565 (3)	0.4711 (2)	0.0801 (14)
O8	0.8109 (3)	0.3497 (3)	0.3861 (2)	0.0746 (13)
O30	0.3236 (3)	0.2935 (3)	0.2066 (2)	0.0780 (13)
O40	0.3914 (3)	0.0820 (3)	0.3167 (3)	0.0797 (13)
O80	0.9284 (3)	0.0589 (3)	0.3918 (3)	0.0926 (16)
O90	0.8360 (3)	0.2055 (3)	0.2410 (2)	0.0771 (13)
F11	0.3288 (6)	0.3258 (5)	0.5418 (4)	0.194 (4)
F12	0.4114 (4)	0.3956 (6)	0.5301 (4)	0.221 (5)
F13	0.2960 (6)	0.4288 (5)	0.4853 (4)	0.209 (4)
F21	0.5906 (3)	0.4011 (4)	0.3932 (3)	0.139 (2)
F22	0.6415 (3)	0.2919 (4)	0.4420 (4)	0.162 (3)
F23	0.5805 (4)	0.3714 (6)	0.4816 (3)	0.192 (4)
F61	1.1398 (3)	0.2794 (4)	0.4375 (3)	0.1240 (18)
F62	1.1000 (4)	0.3566 (4)	0.4966 (3)	0.141 (2)
F63	1.0834 (3)	0.3949 (3)	0.3992 (3)	0.132 (2)
F71	0.8167 (6)	0.3706 (4)	0.5431 (4)	0.194 (4)
F72	0.8968 (4)	0.4350 (4)	0.5152 (4)	0.178 (3)
F73	0.7749 (5)	0.4545 (4)	0.4619 (3)	0.162 (3)
Pd1	0.31360 (3)	0.31739 (3)	0.29334 (2)	0.05695 (15)

Pd2	0.37479 (3)	0.16288 (3)	0.38047 (2)	0.06001 (16)
Pd6	0.88286 (3)	0.15557 (3)	0.42586 (3)	0.06615 (17)
Pd7	0.82028 (3)	0.27495 (3)	0.31190 (2)	0.06109 (16)

Atomic displacement parameters (Å^2) for (4-Pd)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.053 (4)	0.060 (4)	0.052 (3)	-0.003 (3)	0.018 (3)	-0.011 (3)
C2	0.058 (4)	0.069 (4)	0.054 (4)	0.007 (3)	0.015 (3)	-0.002 (3)
C6	0.075 (5)	0.049 (4)	0.076 (5)	-0.002 (3)	0.024 (4)	-0.007 (3)
C7	0.074 (5)	0.053 (4)	0.068 (4)	-0.014 (3)	0.029 (4)	-0.015 (3)
C10	0.091 (6)	0.070 (5)	0.062 (4)	-0.001 (4)	0.026 (4)	-0.011 (4)
C20	0.059 (4)	0.074 (5)	0.079 (5)	0.005 (4)	0.014 (4)	-0.010 (4)
C30	0.069 (5)	0.072 (4)	0.059 (4)	0.003 (3)	0.022 (3)	-0.002 (3)
C31	0.102 (7)	0.097 (6)	0.064 (5)	0.007 (5)	0.012 (4)	0.024 (4)
C32	0.105 (7)	0.105 (7)	0.073 (5)	0.024 (6)	-0.005 (5)	0.010 (5)
C33	0.060 (5)	0.140 (8)	0.069 (5)	0.020 (5)	-0.008 (4)	-0.023 (5)
C33A	0.062 (4)	0.082 (5)	0.071 (4)	0.004 (4)	0.020 (4)	-0.021 (4)
C34	0.056 (5)	0.102 (6)	0.104 (7)	0.002 (4)	0.022 (5)	-0.026 (5)
C35	0.072 (5)	0.109 (7)	0.108 (7)	-0.001 (5)	0.050 (5)	-0.010 (5)
C36	0.097 (6)	0.087 (5)	0.067 (4)	0.020 (4)	0.041 (4)	0.002 (4)
C37	0.057 (4)	0.054 (3)	0.070 (4)	0.005 (3)	0.027 (3)	-0.001 (3)
C37A	0.061 (4)	0.046 (3)	0.054 (3)	0.003 (3)	0.008 (3)	-0.010 (3)
C39	0.105 (7)	0.128 (7)	0.087 (6)	0.011 (5)	0.060 (5)	0.014 (5)
C40	0.124 (7)	0.050 (4)	0.078 (5)	-0.004 (4)	0.059 (5)	-0.007 (4)
C41	0.147 (10)	0.125 (8)	0.071 (6)	-0.038 (7)	0.036 (6)	-0.030 (5)
C42	0.127 (9)	0.151 (10)	0.081 (6)	-0.002 (8)	0.017 (6)	-0.021 (6)
C43	0.122 (8)	0.089 (6)	0.113 (8)	-0.031 (6)	0.024 (6)	-0.028 (6)
C43A	0.101 (6)	0.061 (4)	0.105 (6)	-0.020 (4)	0.048 (5)	-0.014 (4)
C44	0.109 (7)	0.108 (7)	0.120 (8)	-0.035 (6)	0.059 (7)	-0.013 (6)
C45	0.139 (9)	0.111 (7)	0.120 (8)	-0.020 (6)	0.087 (8)	-0.003 (6)
C46	0.095 (6)	0.109 (6)	0.076 (5)	-0.016 (5)	0.042 (5)	-0.027 (5)
C47	0.091 (5)	0.047 (3)	0.055 (4)	-0.005 (3)	0.032 (3)	0.001 (3)
C47A	0.088 (5)	0.044 (3)	0.077 (4)	-0.013 (3)	0.041 (4)	-0.005 (3)
C49	0.186 (11)	0.092 (6)	0.133 (8)	-0.004 (6)	0.113 (8)	-0.014 (6)
C60	0.069 (5)	0.069 (5)	0.077 (5)	-0.006 (4)	0.025 (4)	-0.005 (4)
C70	0.114 (7)	0.094 (6)	0.071 (5)	-0.009 (6)	0.025 (5)	-0.027 (5)
C81	0.125 (9)	0.064 (5)	0.188 (11)	0.015 (6)	0.055 (8)	0.026 (6)
C82	0.143 (9)	0.047 (4)	0.121 (8)	-0.011 (5)	0.017 (7)	-0.002 (5)
C84	0.093 (7)	0.098 (7)	0.116 (7)	-0.020 (5)	0.052 (6)	-0.011 (5)
C83	0.085 (5)	0.071 (5)	0.089 (5)	-0.004 (4)	0.044 (4)	-0.006 (4)

C83A	0.121 (8)	0.079 (6)	0.152 (9)	-0.028 (6)	0.071 (7)	-0.027 (6)
C85	0.085 (6)	0.092 (6)	0.101 (6)	0.010 (5)	0.047 (5)	0.001 (5)
C86	0.136 (8)	0.056 (4)	0.080 (5)	0.025 (5)	0.066 (5)	0.006 (4)
C87	0.081 (5)	0.061 (4)	0.057 (4)	0.001 (3)	0.024 (3)	0.013 (3)
C87A	0.086 (5)	0.052 (4)	0.065 (4)	0.005 (3)	0.042 (4)	0.003 (3)
C80	0.089 (6)	0.072 (5)	0.061 (4)	0.008 (4)	0.027 (4)	0.005 (3)
C89	0.094 (6)	0.098 (6)	0.110 (6)	0.032 (5)	0.057 (5)	0.012 (5)
C90	0.079 (5)	0.076 (5)	0.062 (4)	0.017 (4)	0.022 (4)	-0.002 (4)
C91	0.137 (9)	0.065 (5)	0.105 (7)	-0.005 (5)	0.023 (6)	-0.022 (5)
C92	0.090 (9)	0.129 (11)	0.65 (4)	-0.033 (8)	0.060 (15)	-0.205 (19)
C93	0.093 (7)	0.112 (8)	0.155 (10)	-0.032 (6)	0.022 (7)	-0.037 (7)
C94	0.063 (5)	0.102 (7)	0.118 (7)	0.003 (5)	0.028 (5)	0.008 (6)
C93A	0.082 (6)	0.085 (5)	0.078 (5)	-0.004 (4)	0.026 (4)	-0.004 (4)
C95	0.068 (5)	0.103 (6)	0.097 (6)	0.025 (5)	0.035 (4)	0.008 (5)
C96	0.106 (6)	0.066 (4)	0.062 (4)	0.006 (4)	0.036 (4)	0.000 (3)
C97	0.067 (4)	0.065 (4)	0.055 (4)	0.007 (3)	0.022 (3)	0.012 (3)
C97A	0.072 (4)	0.059 (4)	0.046 (3)	0.002 (3)	0.012 (3)	0.000 (3)
C99	0.118 (7)	0.119 (7)	0.123 (7)	0.025 (6)	0.067 (6)	-0.028 (6)
N30	0.064 (3)	0.062 (3)	0.047 (3)	0.002 (3)	0.012 (2)	0.001 (2)
N40	0.106 (5)	0.057 (3)	0.066 (4)	-0.007 (3)	0.042 (4)	-0.011 (3)
N70	0.081 (4)	0.065 (3)	0.079 (4)	-0.010 (3)	0.039 (3)	0.001 (3)
N90	0.086 (4)	0.067 (4)	0.055 (3)	0.004 (3)	0.024 (3)	-0.004 (3)
O1	0.092 (3)	0.058 (3)	0.051 (3)	0.015 (2)	0.016 (2)	0.000 (2)
O3	0.063 (3)	0.076 (3)	0.077 (3)	-0.001 (2)	0.008 (2)	0.016 (3)
O2	0.092 (3)	0.057 (3)	0.047 (2)	0.005 (2)	0.014 (2)	-0.003 (2)
O4	0.067 (3)	0.064 (3)	0.083 (3)	0.006 (2)	0.016 (2)	0.008 (3)
O5	0.074 (3)	0.072 (3)	0.106 (4)	-0.013 (3)	0.012 (3)	0.017 (3)
O6	0.077 (3)	0.083 (3)	0.077 (3)	-0.007 (3)	0.017 (3)	0.006 (3)
O7	0.111 (4)	0.066 (3)	0.064 (3)	-0.007 (3)	0.037 (3)	0.003 (2)
O8	0.099 (4)	0.055 (3)	0.061 (3)	-0.003 (2)	0.023 (3)	-0.005 (2)
O30	0.068 (3)	0.103 (4)	0.059 (3)	0.019 (3)	0.022 (2)	0.007 (3)
O40	0.101 (4)	0.064 (3)	0.082 (3)	0.007 (3)	0.045 (3)	-0.003 (3)
O80	0.105 (4)	0.062 (3)	0.124 (5)	-0.001 (3)	0.061 (4)	0.006 (3)
O90	0.081 (3)	0.081 (3)	0.068 (3)	0.002 (3)	0.028 (3)	-0.006 (3)
F11	0.368 (12)	0.149 (6)	0.126 (5)	-0.060 (7)	0.162 (7)	-0.043 (4)
F12	0.128 (5)	0.330 (11)	0.204 (7)	-0.091 (7)	0.065 (5)	-0.207 (8)
F13	0.300 (11)	0.187 (7)	0.129 (5)	0.122 (7)	0.077 (6)	-0.037 (5)
F21	0.103 (4)	0.129 (5)	0.151 (5)	-0.040 (3)	0.016 (4)	0.011 (4)
F22	0.062 (3)	0.126 (5)	0.243 (8)	0.015 (3)	0.006 (4)	-0.020 (5)
F23	0.149 (6)	0.302 (10)	0.137 (5)	-0.112 (6)	0.072 (4)	-0.145 (6)

F61	0.083 (3)	0.112 (4)	0.175 (6)	0.004 (3)	0.052 (4)	-0.004 (4)
F62	0.144 (5)	0.166 (5)	0.117 (4)	-0.073 (4)	0.059 (4)	-0.069 (4)
F63	0.113 (4)	0.102 (4)	0.150 (5)	-0.036 (3)	0.022 (3)	0.028 (4)
F71	0.379 (13)	0.128 (5)	0.127 (5)	-0.023 (6)	0.157 (7)	-0.041 (4)
F72	0.150 (6)	0.153 (6)	0.188 (7)	-0.046 (5)	0.027 (5)	-0.109 (5)
F73	0.197 (7)	0.134 (5)	0.139 (5)	0.063 (5)	0.053 (5)	-0.041 (4)
Pd1	0.0563 (3)	0.0558 (3)	0.0479 (3)	0.0046 (2)	0.0105 (2)	0.0066 (2)
Pd2	0.0743 (3)	0.0463 (3)	0.0531 (3)	0.0060 (2)	0.0199 (2)	0.0028 (2)
Pd6	0.0732 (4)	0.0468 (3)	0.0686 (3)	-0.0043 (2)	0.0193 (3)	0.0079 (2)
Pd7	0.0679 (3)	0.0529 (3)	0.0526 (3)	-0.0002 (2)	0.0148 (2)	0.0004 (2)

Geometric parameters (Å, º) for (4-Pd)

C1—O1	1.228 (7)	C60—F62	1.309 (9)
C1—O2	1.241 (7)	C60—F61	1.329 (8)
C1—C10	1.508 (9)	C70—F71	1.263 (10)
C2—O3	1.222 (7)	C70—F72	1.296 (11)
C2—O4	1.238 (8)	C70—F73	1.308 (10)
C2—C20	1.513 (10)	C81—C82	1.378 (12)
C6—O5	1.220 (8)	C81—N70	1.494 (10)
C6—O6	1.222 (8)	C81—H81A	0.97
C6—C60	1.530 (10)	C81—H81B	0.97
C7—O7	1.222 (8)	C82—C83A	1.445 (12)
C7—O8	1.237 (8)	C82—H82A	0.97
C7—C70	1.508 (10)	C82—H82B	0.97
C10—F12	1.216 (9)	C84—C85	1.352 (11)
C10—F11	1.255 (9)	C84—C83	1.394 (11)
C10—F13	1.258 (9)	C84—H84	0.93
C20—F23	1.259 (9)	C83—C87A	1.352 (10)
C20—F22	1.275 (8)	C83—C83A	1.511 (11)
C20—F21	1.321 (9)	C83A—H83A	0.97
C30—O30	1.260 (8)	C83A—H83B	0.97
C30—N30	1.332 (8)	C85—C86	1.464 (11)
C30—C39	1.477 (10)	C85—H85	0.93
C31—C32	1.434 (11)	C86—C87	1.308 (10)
C31—N30	1.496 (8)	C86—H86	0.93
C31—H31A	0.97	C87—C87A	1.436 (9)
C31—H31B	0.97	C87—Pd6	1.939 (7)
C32—C33	1.475 (12)	C87A—N70	1.428 (9)
C32—H32A	0.97	C80—O80	1.243 (8)
C32—H32B	0.97	C80—N70	1.318 (9)

C33—C33A	1.506 (11)	C80—C89	1.485 (10)
C33—H33A	0.97	C89—H89A	0.96
C33—H33B	0.97	C89—H89B	0.96
C33A—C37A	1.372 (9)	C89—H89C	0.96
C33A—C34	1.373 (11)	C90—O90	1.236 (9)
C34—C35	1.376 (12)	C90—N90	1.327 (9)
C34—H34	0.93	C90—C99	1.496 (10)
C35—C36	1.426 (11)	C91—C92	1.344 (14)
C35—H35	0.93	C91—N90	1.495 (10)
C36—C37	1.351 (9)	C91—H91A	0.97
C36—H36	0.93	C91—H91B	0.97
C37—C37A	1.407 (9)	C92—C93	1.319 (15)
C37—Pd1	1.938 (6)	C92—H92A	0.97
C37A—N30	1.439 (8)	C92—H92B	0.97
C39—H39A	0.96	C93—C93A	1.503 (12)
C39—H39B	0.96	C93—H93A	0.97
C39—H39C	0.96	C93—H93B	0.97
C40—O40	1.239 (9)	C94—C95	1.354 (12)
C40—N40	1.346 (10)	C94—C93A	1.378 (11)
C40—C49	1.494 (10)	C94—H94	0.93
C41—C42	1.419 (13)	C93A—C97A	1.357 (10)
C41—N40	1.517 (10)	C95—C96	1.412 (11)
C41—H41A	0.97	C95—H95	0.93
C41—H41B	0.97	C96—C97	1.354 (9)
C42—C43	1.475 (14)	C96—H96	0.93
C42—H42A	0.97	C97—C97A	1.426 (9)
C42—H42B	0.97	C97—Pd7	1.930 (7)
C43—C43A	1.510 (12)	C97A—N90	1.441 (9)
C43—H43A	0.97	C99—H99A	0.96
C43—H43B	0.97	C99—H99B	0.96
C43A—C44	1.366 (11)	C99—H99C	0.96
C43A—C47A	1.393 (10)	O1—Pd1	2.071 (4)
C44—C45	1.369 (12)	O3—Pd1	2.176 (5)
C44—H44	0.93	O2—Pd2	2.069 (4)
C45—C46	1.396 (11)	O4—Pd2	2.176 (5)
C45—H45	0.93	O5—Pd6	2.166 (5)
C46—C47	1.330 (9)	O6—Pd7	2.154 (5)
C46—H46	0.93	O7—Pd6	2.084 (5)
C47—C47A	1.424 (9)	O8—Pd7	2.059 (5)
C47—Pd2	1.936 (7)	O30—Pd1	1.995 (5)

C47A—N40	1.417 (9)	O40—Pd2	1.996 (5)
C49—H49A	0.96	O80—Pd6	2.015 (5)
C49—H49B	0.96	O90—Pd7	2.002 (5)
C49—H49C	0.96	Pd1—Pd2	3.0119 (6)
C60—F63	1.293 (8)	Pd6—Pd7	2.9427 (7)
O1—C1—O2	131.3 (6)	C83—C84—H84	119.4
O1—C1—C10	115.7 (6)	C87A—C83—C84	119.1 (7)
O2—C1—C10	113.0 (6)	C87A—C83—C83A	122.2 (8)
O3—C2—O4	128.5 (7)	C84—C83—C83A	118.6 (8)
O3—C2—C20	116.0 (7)	C82—C83A—C83	114.4 (8)
O4—C2—C20	115.4 (6)	C82—C83A—H83A	108.7
O5—C6—O6	130.1 (7)	C83—C83A—H83A	108.7
O5—C6—C60	115.0 (7)	C82—C83A—H83B	108.7
O6—C6—C60	115.0 (7)	C83—C83A—H83B	108.7
O7—C7—O8	131.1 (6)	H83A—C83A—H83B	107.6
O7—C7—C70	115.2 (7)	C84—C85—C86	118.8 (8)
O8—C7—C70	113.6 (7)	C84—C85—H85	120.6
F12—C10—F11	104.1 (9)	C86—C85—H85	120.6
F12—C10—F13	105.1 (9)	C87—C86—C85	120.0 (7)
F11—C10—F13	104.0 (9)	C87—C86—H86	120.0
F12—C10—C1	113.4 (7)	C85—C86—H86	120.0
F11—C10—C1	115.0 (7)	C86—C87—C87A	119.9 (7)
F13—C10—C1	114.1 (7)	C86—C87—Pd6	116.8 (6)
F23—C20—F22	109.7 (8)	C87A—C87—Pd6	123.1 (6)
F23—C20—F21	105.2 (8)	C83—C87A—N70	119.3 (6)
F22—C20—F21	103.6 (8)	C83—C87A—C87	120.9 (7)
F23—C20—C2	111.3 (7)	N70—C87A—C87	119.7 (6)
F22—C20—C2	114.1 (7)	O80—C80—N70	122.5 (7)
F21—C20—C2	112.4 (6)	O80—C80—C89	114.3 (7)
O30—C30—N30	122.9 (6)	N70—C80—C89	123.2 (7)
O30—C30—C39	115.6 (7)	C80—C89—H89A	109.5
N30—C30—C39	121.5 (7)	C80—C89—H89B	109.5
C32—C31—N30	114.9 (7)	H89A—C89—H89B	109.5
C32—C31—H31A	108.5	C80—C89—H89C	109.5
N30—C31—H31A	108.5	H89A—C89—H89C	109.5
C32—C31—H31B	108.5	H89B—C89—H89C	109.5
N30—C31—H31B	108.5	O90—C90—N90	123.0 (7)
H31A—C31—H31B	107.5	O90—C90—C99	115.7 (8)
C31—C32—C33	112.6 (7)	N90—C90—C99	121.3 (8)

C31—C32—H32A	109.1	C92—C91—N90	115.2 (9)
C33—C32—H32A	109.1	C92—C91—H91A	108.5
C31—C32—H32B	109.1	N90—C91—H91A	108.5
C33—C32—H32B	109.1	C92—C91—H91B	108.5
H32A—C32—H32B	107.8	N90—C91—H91B	108.5
C32—C33—C33A	109.5 (7)	H91A—C91—H91B	107.5
C32—C33—H33A	109.8	C93—C92—C91	128.6 (10)
C33A—C33—H33A	109.8	C93—C92—H92A	105.1
C32—C33—H33B	109.8	C91—C92—H92A	105.1
C33A—C33—H33B	109.8	C93—C92—H92B	105.1
H33A—C33—H33B	108.2	C91—C92—H92B	105.1
C37A—C33A—C34	119.5 (7)	H92A—C92—H92B	105.9
C37A—C33A—C33	121.2 (7)	C92—C93—C93A	113.9 (9)
C34—C33A—C33	119.2 (7)	C92—C93—H93A	108.8
C33A—C34—C35	121.3 (7)	C93A—C93—H93A	108.8
C33A—C34—H34	119.3	C92—C93—H93B	108.8
C35—C34—H34	119.3	C93A—C93—H93B	108.8
C34—C35—C36	118.5 (8)	H93A—C93—H93B	107.7
C34—C35—H35	120.7	C95—C94—C93A	121.6 (8)
C36—C35—H35	120.7	C95—C94—H94	119.2
C37—C36—C35	120.4 (7)	C93A—C94—H94	119.2
C37—C36—H36	119.8	C97A—C93A—C94	119.5 (8)
C35—C36—H36	119.8	C97A—C93A—C93	121.2 (8)
C36—C37—C37A	119.4 (6)	C94—C93A—C93	119.0 (8)
C36—C37—Pd1	117.6 (5)	C94—C95—C96	119.1 (7)
C37A—C37—Pd1	123.0 (5)	C94—C95—H95	120.4
C33A—C37A—C37	120.7 (7)	C96—C95—H95	120.4
C33A—C37A—N30	117.9 (6)	C97—C96—C95	120.6 (7)
C37—C37A—N30	121.4 (6)	C97—C96—H96	119.7
C30—C39—H39A	109.5	C95—C96—H96	119.7
C30—C39—H39B	109.5	C96—C97—C97A	118.6 (7)
H39A—C39—H39B	109.5	C96—C97—Pd7	119.8 (6)
C30—C39—H39C	109.5	C97A—C97—Pd7	121.5 (5)
H39A—C39—H39C	109.5	C93A—C97A—C97	120.5 (7)
H39B—C39—H39C	109.5	C93A—C97A—N90	119.0 (6)
O40—C40—N40	123.0 (7)	C97—C97A—N90	120.5 (6)
O40—C40—C49	116.7 (9)	C90—C99—H99A	109.5
N40—C40—C49	120.3 (8)	C90—C99—H99B	109.5
C42—C41—N40	112.2 (8)	H99A—C99—H99B	109.5
C42—C41—H41A	109.2	C90—C99—H99C	109.5

N40—C41—H41A	109.2	H99A—C99—H99C	109.5
C42—C41—H41B	109.2	H99B—C99—H99C	109.5
N40—C41—H41B	109.2	C30—N30—C37A	124.2 (5)
H41A—C41—H41B	107.9	C30—N30—C31	116.3 (6)
C41—C42—C43	115.0 (10)	C37A—N30—C31	119.0 (6)
C41—C42—H42A	108.5	C40—N40—C47A	124.5 (6)
C43—C42—H42A	108.5	C40—N40—C41	115.9 (7)
C41—C42—H42B	108.5	C47A—N40—C41	118.9 (7)
C43—C42—H42B	108.5	C80—N70—C87A	127.3 (6)
H42A—C42—H42B	107.5	C80—N70—C81	117.1 (7)
C42—C43—C43A	109.3 (8)	C87A—N70—C81	115.5 (6)
C42—C43—H43A	109.8	C90—N90—C97A	125.4 (6)
C43A—C43—H43A	109.8	C90—N90—C91	119.8 (7)
C42—C43—H43B	109.8	C97A—N90—C91	114.7 (7)
C43A—C43—H43B	109.8	C1—O1—Pd1	126.0 (4)
H43A—C43—H43B	108.3	C2—O3—Pd1	132.1 (5)
C44—C43A—C47A	118.9 (8)	C1—O2—Pd2	123.0 (4)
C44—C43A—C43	121.0 (9)	C2—O4—Pd2	119.0 (4)
C47A—C43A—C43	120.0 (8)	C6—O5—Pd6	125.9 (5)
C43A—C44—C45	121.8 (9)	C6—O6—Pd7	122.8 (5)
C43A—C44—H44	119.1	C7—O7—Pd6	124.2 (5)
C45—C44—H44	119.1	C7—O8—Pd7	123.5 (4)
C44—C45—C46	118.8 (8)	C30—O30—Pd1	124.5 (4)
C44—C45—H45	120.6	C40—O40—Pd2	123.8 (5)
C46—C45—H45	120.6	C80—O80—Pd6	125.2 (5)
C47—C46—C45	121.4 (8)	C90—O90—Pd7	123.2 (5)
C47—C46—H46	119.3	C37—Pd1—O30	87.8 (2)
C45—C46—H46	119.3	C37—Pd1—O1	94.4 (2)
C46—C47—C47A	119.5 (7)	O30—Pd1—O1	173.8 (2)
C46—C47—Pd2	119.6 (5)	C37—Pd1—O3	173.8 (2)
C47A—C47—Pd2	120.6 (5)	O30—Pd1—O3	90.9 (2)
C43A—C47A—N40	119.8 (7)	O1—Pd1—O3	86.3 (2)
C43A—C47A—C47	119.4 (7)	C37—Pd1—Pd2	111.36 (19)
N40—C47A—C47	120.8 (6)	O30—Pd1—Pd2	106.54 (15)
C40—C49—H49A	109.5	O1—Pd1—Pd2	78.12 (12)
C40—C49—H49B	109.5	O3—Pd1—Pd2	74.79 (12)
H49A—C49—H49B	109.5	C47—Pd2—O40	87.6 (2)
C40—C49—H49C	109.5	C47—Pd2—O2	93.7 (2)
H49A—C49—H49C	109.5	O40—Pd2—O2	177.0 (2)
H49B—C49—H49C	109.5	C47—Pd2—O4	178.9 (2)

F63—C60—F62	107.4 (7)	O40—Pd2—O4	93.1 (2)
F63—C60—F61	106.0 (7)	O2—Pd2—O4	85.65 (19)
F62—C60—F61	105.3 (7)	C47—Pd2—Pd1	94.95 (18)
F63—C60—C6	114.9 (7)	O40—Pd2—Pd1	102.09 (14)
F62—C60—C6	111.2 (7)	O2—Pd2—Pd1	80.53 (12)
F61—C60—C6	111.5 (6)	O4—Pd2—Pd1	84.04 (12)
F71—C70—F72	106.4 (8)	C87—Pd6—O80	89.3 (3)
F71—C70—F73	108.3 (10)	C87—Pd6—O7	95.4 (3)
F72—C70—F73	102.8 (9)	O80—Pd6—O7	172.9 (2)
F71—C70—C7	115.1 (8)	C87—Pd6—O5	177.8 (2)
F72—C70—C7	109.7 (8)	O80—Pd6—O5	89.3 (2)
F73—C70—C7	113.6 (7)	O7—Pd6—O5	85.8 (2)
C82—C81—N70	113.8 (8)	C87—Pd6—Pd7	103.02 (18)
C82—C81—H81A	108.8	O80—Pd6—Pd7	104.78 (16)
N70—C81—H81A	108.8	O7—Pd6—Pd7	79.40 (13)
C82—C81—H81B	108.8	O5—Pd6—Pd7	78.96 (14)
N70—C81—H81B	108.8	C97—Pd7—O90	89.1 (2)
H81A—C81—H81B	107.7	C97—Pd7—O8	94.3 (2)
C81—C82—C83A	118.6 (9)	O90—Pd7—O8	176.5 (2)
C81—C82—H82A	107.7	C97—Pd7—O6	176.5 (2)
C83A—C82—H82A	107.7	O90—Pd7—O6	90.5 (2)
C81—C82—H82B	107.7	O8—Pd7—O6	86.1 (2)
C83A—C82—H82B	107.7	C97—Pd7—Pd6	101.98 (18)
H82A—C82—H82B	107.1	O90—Pd7—Pd6	99.41 (14)
C85—C84—C83	121.3 (8)	O8—Pd7—Pd6	80.90 (13)
C85—C84—H84	119.4	O6—Pd7—Pd6	81.49 (14)
O1—C1—C10—F12	-97.3 (10)	C95—C94—C93A—C97A	1.2 (14)
O2—C1—C10—F12	81.2 (10)	C95—C94—C93A—C93	176.5 (9)
O1—C1—C10—F11	143.1 (8)	C92—C93—C93A—C97A	11 (2)
O2—C1—C10—F11	-38.5 (11)	C92—C93—C93A—C94	-164.2 (16)
O1—C1—C10—F13	23.0 (11)	C93A—C94—C95—C96	0.9 (14)
O2—C1—C10—F13	-158.6 (8)	C94—C95—C96—C97	-2.5 (12)
O3—C2—C20—F23	93.4 (9)	C95—C96—C97—C97A	1.9 (10)
O4—C2—C20—F23	-84.1 (9)	C95—C96—C97—Pd7	-179.6 (6)

O3—C2—C20—F22	-141.7 (8)	C94—C93A—C97A—C97	-1.8 (11)
O4—C2—C20—F22	40.7 (10)	C93—C93A—C97A—C97	-177.0 (8)
O3—C2—C20—F21	-24.2 (10)	C94—C93A—C97A—N90	-178.9 (7)
O4—C2—C20—F21	158.3 (7)	C93—C93A—C97A—N90	5.8 (12)
N30—C31—C32—C33	-44.8 (11)	C96—C97—C97A—C93A	0.3 (10)
C31—C32—C33—C33A	56.2 (10)	Pd7—C97—C97A—C93A	-178.2 (5)
C32—C33—C33A—C37A	-31.5 (10)	C96—C97—C97A—N90	177.4 (6)
C32—C33—C33A—C34	145.8 (8)	Pd7—C97—C97A—N90	-1.1 (8)
C37A—C33A—C34—C35	-0.5 (12)	O30—C30—N30—C37A	-10.1 (10)
C33—C33A—C34—C35	-177.9 (8)	C39—C30—N30—C37A	168.5 (6)
C33A—C34—C35—C36	1.8 (13)	O30—C30—N30—C31	177.5 (7)
C34—C35—C36—C37	-1.9 (12)	C39—C30—N30—C31	-3.9 (10)
C35—C36—C37—C37A	0.6 (11)	C33A—C37A—N30—C30	-153.0 (6)
C35—C36—C37—Pd1	-178.8 (6)	C37—C37A—N30—C30	24.9 (9)
C34—C33A—C37A—C37	-0.8 (10)	C33A—C37A—N30—C31	19.2 (9)
C33—C33A—C37A—C37	176.5 (7)	C37—C37A—N30—C31	-162.9 (6)
C34—C33A—C37A—N30	177.2 (6)	C32—C31—N30—C30	179.2 (7)
C33—C33A—C37A—N30	-5.6 (10)	C32—C31—N30—C37A	6.4 (10)
C36—C37—C37A—C33A	0.7 (9)	O40—C40—N40—C47A	-17.8 (11)
Pd1—C37—C37A—C33A	-179.9 (5)	C49—C40—N40—C47A	161.5 (7)
C36—C37—C37A—N30	-177.2 (6)	O40—C40—N40—C41	171.6 (7)
Pd1—C37—C37A—N30	2.3 (8)	C49—C40—N40—C41	-9.0 (10)
N40—C41—C42—C43	-49.8 (13)	C43A—C47A—N40—C40	-152.9 (7)
C41—C42—C43—C43A	55.8 (12)	C47—C47A—N40—C40	24.3 (10)

C42—C43—C43A—C44	150.2 (10)	C43A—C47A—N40—C41	17.4 (10)
C42—C43—C43A—C47A	-24.7 (12)	C47—C47A—N40—C41	-165.4 (7)
C47A—C43A—C44—C45	-1.1 (14)	C42—C41—N40—C40	-176.7 (9)
C43—C43A—C44—C45	-175.9 (9)	C42—C41—N40—C47A	12.2 (12)
C43A—C44—C45—C46	4.4 (16)	O80—C80—N70—C87A	8.8 (12)
C44—C45—C46—C47	-4.4 (15)	C89—C80—N70—C87A	-170.4 (7)
C45—C46—C47—C47A	1.0 (12)	O80—C80—N70—C81	-174.7 (8)
C45—C46—C47—Pd2	174.5 (7)	C89—C80—N70—C81	6.1 (11)
C44—C43A—C47A—N40	174.9 (7)	C83—C87A—N70—C80	154.8 (7)
C43—C43A—C47A—N40	-10.2 (11)	C87—C87A—N70—C80	-27.2 (10)
C44—C43A—C47A—C47	-2.3 (11)	C83—C87A—N70—C81	-21.7 (10)
C43—C43A—C47A—C47	172.6 (7)	C87—C87A—N70—C81	156.3 (8)
C46—C47—C47A—C43A	2.3 (10)	C82—C81—N70—C80	-132.3 (10)
Pd2—C47—C47A—C43A	-171.1 (5)	C82—C81—N70—C87A	44.5 (13)
C46—C47—C47A—N40	-174.9 (7)	O90—C90—N90—C97A	10.5 (11)
Pd2—C47—C47A—N40	11.7 (8)	C99—C90—N90—C97A	-167.7 (7)
O5—C6—C60—F63	-175.6 (7)	O90—C90—N90—C91	-173.2 (7)
O6—C6—C60—F63	4.7 (10)	C99—C90—N90—C91	8.6 (11)
O5—C6—C60—F62	-53.3 (9)	C93A—C97A—N90—C90	150.4 (7)
O6—C6—C60—F62	126.9 (8)	C97—C97A—N90—C90	-26.8 (9)
O5—C6—C60—F61	63.9 (9)	C93A—C97A—N90—C91	-26.1 (9)
O6—C6—C60—F61	-115.9 (8)	C97—C97A—N90—C91	156.8 (6)
O7—C7—C70—F71	-25.9 (12)	C92—C91—N90—C90	-146.5 (15)
O8—C7—C70—F71	155.3 (9)	C92—C91—N90—C97A	30.2 (16)

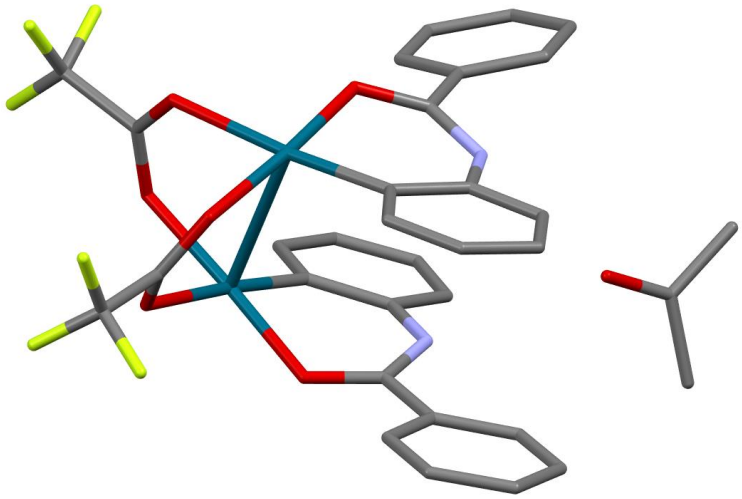
O7—C7—C70—F72	94.0 (10)	O2—C1—O1—Pd1	-12.9 (11)
O8—C7—C70—F72	-84.8 (10)	C10—C1—O1—Pd1	165.1 (5)
O7—C7—C70—F73	-151.6 (8)	O4—C2—O3—Pd1	14.8 (12)
O8—C7—C70—F73	29.6 (12)	C20—C2—O3—Pd1	-162.3 (5)
N70—C81—C82—C83A	-44.7 (16)	O1—C1—O2—Pd2	5.6 (10)
C85—C84—C83—C87A	-0.4 (13)	C10—C1—O2—Pd2	-172.5 (5)
C85—C84—C83—C83A	-179.9 (9)	O3—C2—O4—Pd2	-15.6 (10)
C81—C82—C83A—C83	21.9 (15)	C20—C2—O4—Pd2	161.6 (5)
C87A—C83—C83A—C82	2.2 (14)	O6—C6—O5—Pd6	-5.9 (12)
C84—C83—C83A—C82	-178.2 (9)	C60—C6—O5—Pd6	174.4 (5)
C83—C84—C85—C86	-0.6 (13)	O5—C6—O6—Pd7	11.6 (11)
C84—C85—C86—C87	0.4 (12)	C60—C6—O6—Pd7	-168.7 (5)
C85—C86—C87—C87A	0.6 (11)	O8—C7—O7—Pd6	12.5 (11)
C85—C86—C87—Pd6	175.7 (5)	C70—C7—O7—Pd6	-166.0 (5)
C84—C83—C87A—N70	179.4 (7)	O7—C7—O8—Pd7	-6.0 (11)
C83A—C83—C87A—N70	-1.0 (12)	C70—C7—O8—Pd7	172.5 (5)
C84—C83—C87A—C87	1.4 (11)	N30—C30—O30—Pd1	-30.1 (10)
C83A—C83—C87A—C87	-179.0 (8)	C39—C30—O30—Pd1	151.2 (6)
C86—C87—C87A—C83	-1.6 (10)	N40—C40—O40—Pd2	-24.2 (10)
Pd6—C87—C87A—C83	-176.3 (5)	C49—C40—O40—Pd2	156.4 (5)
C86—C87—C87A—N70	-179.6 (6)	N70—C80—O80—Pd6	28.8 (10)
Pd6—C87—C87A—N70	5.7 (9)	C89—C80—O80—Pd6	-151.9 (5)
N90—C91—C92—C93	-14 (3)	N90—C90—O90—Pd7	31.0 (10)
C91—C92—C93—C93A	-6 (3)	C99—C90—O90—Pd7	-150.7 (6)

Hydrogen-bond geometry (Å, º) for (4-Pd)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C41—H41*B*···O1^{i}	0.97	2.56	3.514 (11)	168
C92—H92*B*···O6^{ii}	0.97	2.74	3.454 (13)	131

Symmetry codes: (i) -*x*+1/2, *y*-1/2, -*z*+1/2; (ii) -*x*+3/2, *y*-1/2, -*z*+1/2.

(8-Pd)



Crystal data

$C_{30}H_{20}F_6N_2O_6Pd_2 \cdot C_3H_6O$	*F*(000) = 1760
M_r = 889.36	D_x = 1.736 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo *K*a radiation, l = 0.71073 Å
a = 19.2535 (7) Å	Cell parameters from 387 reflections
b = 8.0799 (4) Å	q = 2.7–27.7°
c = 21.9245 (10) Å	m = 1.14 mm^{-1}
b = 93.765 (2)°	*T* = 294 K
V = 3403.4 (3) Å^3	Prism, yellow
Z = 4	0.26 × 0.20 × 0.17 mm

Data collection

Bruker D8 VENTURE diffractometer	6682 independent reflections
Radiation source: microfocus sealed tube, INCOATEC ImS 3.0	5757 reflections with *I* > 2s(*I*)
Multilayer mirror INCOATEC monochromator	R_{int} = 0.037
Detector resolution: 7.3910 pixels mm^{-1}	q_{max} = 26.1°, q_{min} = 2.2°
w and p scan	*h* = -23→23
Absorption correction: multi-scan *SADABS2016/2* - Bruker AXS area detector	*k* = -9→9

scaling and absorption correction	
T_{min} = 0.71, T_{max} = 0.83	l = -27→27
38088 measured reflections	

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2s(F^2)]$ = 0.051	Hydrogen site location: mixed
$wR(F^2)$ = 0.132	H atoms treated by a mixture of independent and constrained refinement
S = 1.12	$w = 1/[s^2(F_o^2) + (0.0424P)^2 + 15.6605P]$ where $P = (F_o^2 + 2F_c^2)/3$
6682 reflections	$(D/s)_{max}$ = 0.001
459 parameters	$D\rho_{max}$ = 1.18 e Å^{-3}
2 restraints	$D\rho_{min}$ = -0.95 e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2) for (8-Pd)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.3402 (3)	0.3733 (9)	0.1185 (3)	0.0489 (14)
C2	0.1566 (3)	0.5108 (9)	0.1086 (3)	0.0553 (16)
C10	0.3966 (4)	0.3303 (11)	0.0754 (4)	0.074 (2)
C20	0.1020 (7)	0.5351 (14)	0.0548 (6)	0.114 (4)
C30	0.1546 (3)	0.3254 (7)	0.3063 (3)	0.0398 (12)
C31	0.0938 (3)	0.4031 (8)	0.3332 (3)	0.0475 (14)
C32	0.0775 (4)	0.3675 (11)	0.3923 (4)	0.076 (2)
H32	0.104124	0.29185	0.415714	0.091*
C33	0.0213 (5)	0.4447 (15)	0.4166 (5)	0.103 (3)
H33	0.009865	0.419705	0.456084	0.123*
C34	-0.0168 (5)	0.5562 (14)	0.3831 (5)	0.096 (3)
H34	-0.053892	0.609074	0.400029	0.115*
C35	-0.0017 (4)	0.5925 (10)	0.3247 (5)	0.076 (2)

H35	-0.028306	0.66959	0.302023	0.092*
C36	0.0538 (3)	0.5138 (9)	0.2991 (3)	0.0562 (16)
H36	0.063588	0.536352	0.258926	0.067*
C41	0.2693 (3)	0.1953 (7)	0.3284 (3)	0.0409 (12)
C42	0.2881 (3)	0.1740 (7)	0.2686 (3)	0.0405 (12)
C43	0.3469 (3)	0.0788 (8)	0.2600 (3)	0.0532 (15)
H43	0.36008	0.059477	0.220598	0.064*
C44	0.3860 (3)	0.0121 (8)	0.3095 (4)	0.0586 (17)
H44	0.425178	-0.050823	0.302822	0.07*
C45	0.3676 (3)	0.0379 (8)	0.3678 (3)	0.0587 (17)
H45	0.394399	-0.005621	0.400773	0.07*
C46	0.3086 (3)	0.1293 (8)	0.3772 (3)	0.0539 (15)
H46	0.295352	0.146418	0.416769	0.065*
C50	0.2503 (3)	0.5275 (9)	0.5064 (3)	0.0572 (16)
C51	0.2977 (5)	0.6517 (15)	0.5325 (5)	0.117 (4)
H51A	0.336001	0.664691	0.506957	0.175*
H51B	0.314964	0.617441	0.5726	0.175*
H51C	0.273636	0.755162	0.535214	0.175*
C52	0.1938 (5)	0.4683 (13)	0.5439 (4)	0.095 (3)
H52A	0.179811	0.359157	0.530949	0.142*
H52B	0.154795	0.542053	0.53894	0.142*
H52C	0.210438	0.465449	0.586175	0.142*
C80	0.3519 (3)	0.5818 (7)	0.3047 (3)	0.0390 (12)
C81	0.4125 (3)	0.4944 (7)	0.3336 (3)	0.0435 (13)
C82	0.4240 (3)	0.4744 (10)	0.3964 (3)	0.0611 (18)
H82	0.393409	0.522284	0.422356	0.073*
C83	0.4797 (4)	0.3852 (11)	0.4206 (4)	0.073 (2)
H83	0.486587	0.37204	0.462673	0.088*
C84	0.5257 (3)	0.3148 (9)	0.3823 (4)	0.0655 (19)
H84	0.563554	0.25408	0.398536	0.079*
C85	0.5153 (3)	0.3347 (9)	0.3204 (4)	0.0648 (19)
H85	0.546447	0.288267	0.294641	0.078*
C86	0.4593 (3)	0.4225 (9)	0.2962 (3)	0.0592 (17)
H86	0.452614	0.434164	0.254029	0.071*
C91	0.2386 (3)	0.7137 (7)	0.3178 (3)	0.0394 (12)
C92	0.2165 (3)	0.7365 (7)	0.2565 (3)	0.0415 (12)
C93	0.1580 (3)	0.8356 (8)	0.2435 (3)	0.0530 (16)
H93	0.14288	0.855409	0.203035	0.064*
C94	0.1223 (3)	0.9043 (8)	0.2896 (4)	0.0585 (17)
H94	0.08362	0.970303	0.279851	0.07*

C95	0.1429 (3)	0.8772 (8)	0.3489 (4)	0.0612 (18)
H95	0.11729	0.921068	0.379595	0.073*
C96	0.2014 (3)	0.7852 (8)	0.3640 (3)	0.0499 (14)
H96	0.216458	0.770318	0.40486	0.06*
H30	0.210 (4)	0.314 (9)	0.3806 (13)	0.06*
H80	0.300 (4)	0.594 (9)	0.3750 (13)	0.06*
N30	0.2085 (2)	0.2855 (6)	0.3430 (2)	0.0427 (11)
N80	0.2983 (2)	0.6212 (6)	0.3375 (2)	0.0409 (10)
O1	0.3144 (2)	0.2509 (6)	0.1433 (2)	0.0550 (11)
O2	0.3276 (2)	0.5212 (6)	0.12295 (19)	0.0544 (11)
O3	0.1815 (2)	0.6427 (6)	0.1295 (2)	0.0587 (11)
O5	0.1672 (2)	0.3666 (6)	0.1223 (2)	0.0554 (11)
O30	0.15136 (19)	0.3032 (5)	0.24941 (18)	0.0439 (9)
O50	0.2561 (2)	0.4736 (6)	0.4549 (2)	0.0629 (12)
O80	0.35206 (19)	0.6139 (5)	0.24863 (19)	0.0462 (9)
F11	0.4063 (5)	0.4382 (10)	0.0352 (4)	0.183 (4)
F12	0.4561 (3)	0.3207 (16)	0.1046 (4)	0.197 (5)
F13	0.3886 (4)	0.1934 (10)	0.0482 (4)	0.176 (4)
F21	0.0995 (5)	0.6716 (9)	0.0299 (4)	0.184 (4)
F22	0.0402 (5)	0.527 (2)	0.0753 (6)	0.265 (8)
F23	0.0962 (8)	0.4191 (12)	0.0192 (4)	0.251 (7)
Pd1	0.23361 (2)	0.27341 (5)	0.19976 (2)	0.04028 (13)
Pd2	0.26633 (2)	0.63196 (6)	0.19247 (2)	0.04238 (14)

Atomic displacement parameters ($Å^2$) for (8-Pd)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.050 (3)	0.060 (4)	0.037 (3)	0.002 (3)	0.006 (3)	0.003 (3)
C2	0.057 (4)	0.064 (4)	0.044 (3)	0.005 (3)	-0.005 (3)	0.002 (3)
C10	0.073 (5)	0.079 (6)	0.073 (5)	-0.005 (4)	0.034 (4)	-0.013 (4)
C20	0.130 (10)	0.077 (7)	0.124 (9)	0.004 (6)	-0.081 (8)	0.000 (7)
C30	0.035 (3)	0.033 (3)	0.051 (3)	-0.005 (2)	0.003 (2)	0.000 (2)
C31	0.039 (3)	0.045 (3)	0.059 (4)	-0.010 (3)	0.009 (3)	-0.012 (3)
C32	0.072 (5)	0.090 (6)	0.068 (5)	0.011 (4)	0.022 (4)	0.003 (4)
C33	0.079 (6)	0.139 (10)	0.095 (7)	0.004 (6)	0.042 (5)	-0.028 (7)
C34	0.055 (5)	0.112 (8)	0.125 (9)	-0.002 (5)	0.030 (5)	-0.047 (7)
C35	0.045 (4)	0.064 (5)	0.120 (7)	0.006 (3)	0.001 (4)	-0.023 (5)
C36	0.042 (3)	0.057 (4)	0.068 (4)	-0.001 (3)	-0.001 (3)	-0.006 (3)
C41	0.035 (3)	0.035 (3)	0.052 (3)	-0.003 (2)	-0.003 (2)	0.000 (2)
C42	0.038 (3)	0.032 (3)	0.051 (3)	-0.003 (2)	0.003 (2)	0.002 (2)
C43	0.046 (3)	0.048 (4)	0.066 (4)	-0.003 (3)	0.008 (3)	-0.003 (3)

C44	0.039 (3)	0.050 (4)	0.086 (5)	0.007 (3)	-0.003 (3)	0.003 (4)
C45	0.049 (4)	0.053 (4)	0.072 (5)	0.001 (3)	-0.016 (3)	0.010 (3)
C46	0.053 (4)	0.057 (4)	0.050 (4)	-0.002 (3)	-0.007 (3)	-0.003 (3)
C50	0.054 (4)	0.065 (4)	0.051 (4)	0.014 (3)	-0.005 (3)	-0.001 (3)
C51	0.082 (6)	0.151 (10)	0.119 (8)	-0.010 (6)	0.013 (6)	-0.087 (8)
C52	0.120 (8)	0.097 (7)	0.069 (5)	0.003 (6)	0.027 (5)	0.017 (5)
C80	0.033 (3)	0.038 (3)	0.046 (3)	-0.008 (2)	0.006 (2)	-0.004 (2)
C81	0.031 (3)	0.045 (3)	0.055 (3)	-0.006 (2)	0.004 (2)	-0.003 (3)
C82	0.043 (3)	0.086 (5)	0.053 (4)	0.008 (3)	-0.001 (3)	-0.004 (4)
C83	0.056 (4)	0.099 (6)	0.063 (4)	0.000 (4)	-0.009 (3)	0.010 (4)
C84	0.042 (3)	0.061 (4)	0.092 (6)	0.004 (3)	-0.004 (4)	0.011 (4)
C85	0.042 (3)	0.068 (5)	0.086 (5)	0.012 (3)	0.011 (3)	0.000 (4)
C86	0.044 (3)	0.073 (5)	0.062 (4)	0.005 (3)	0.013 (3)	-0.002 (4)
C91	0.033 (3)	0.029 (3)	0.056 (3)	-0.001 (2)	0.005 (2)	-0.004 (2)
C92	0.036 (3)	0.031 (3)	0.058 (3)	-0.005 (2)	0.002 (2)	-0.002 (2)
C93	0.042 (3)	0.039 (3)	0.077 (4)	0.000 (3)	-0.006 (3)	0.001 (3)
C94	0.042 (3)	0.043 (3)	0.089 (5)	0.006 (3)	-0.003 (3)	-0.005 (3)
C95	0.050 (4)	0.047 (4)	0.089 (5)	0.004 (3)	0.018 (4)	-0.014 (4)
C96	0.047 (3)	0.048 (3)	0.056 (4)	0.001 (3)	0.012 (3)	-0.009 (3)
N30	0.040 (2)	0.043 (3)	0.044 (3)	-0.001 (2)	0.001 (2)	-0.003 (2)
N80	0.039 (2)	0.041 (3)	0.043 (3)	0.000 (2)	0.004 (2)	-0.002 (2)
O1	0.062 (3)	0.052 (3)	0.053 (3)	0.006 (2)	0.015 (2)	-0.002 (2)
O2	0.062 (3)	0.055 (3)	0.047 (2)	-0.003 (2)	0.013 (2)	0.004 (2)
O3	0.064 (3)	0.053 (3)	0.058 (3)	0.010 (2)	-0.010 (2)	0.001 (2)
O5	0.062 (3)	0.052 (3)	0.050 (2)	0.006 (2)	-0.010 (2)	0.004 (2)
O30	0.0362 (19)	0.047 (2)	0.049 (2)	-0.0009 (17)	0.0008 (17)	-0.0017 (18)
O50	0.064 (3)	0.071 (3)	0.054 (3)	-0.001 (2)	0.003 (2)	-0.017 (2)
O80	0.0349 (19)	0.052 (2)	0.052 (2)	-0.0024 (18)	0.0089 (17)	0.003 (2)
F11	0.261 (10)	0.148 (7)	0.159 (7)	0.052 (7)	0.154 (7)	0.045 (5)
F12	0.066 (4)	0.348 (14)	0.180 (8)	0.037 (6)	0.032 (4)	-0.060 (9)
F13	0.188 (7)	0.147 (6)	0.211 (8)	-0.062 (5)	0.143 (7)	-0.108 (6)
F21	0.237 (9)	0.115 (5)	0.180 (7)	-0.038 (6)	-0.146 (7)	0.068 (5)
F22	0.127 (7)	0.354 (19)	0.297 (15)	-0.024 (10)	-0.121 (9)	0.099 (14)
F23	0.393 (16)	0.161 (8)	0.168 (8)	0.123 (9)	-0.208 (10)	-0.076 (7)
Pd1	0.0410 (2)	0.0368 (2)	0.0432 (2)	0.00025 (18)	0.00342 (18)	0.00000 (18)
Pd2	0.0432 (2)	0.0387 (2)	0.0452 (3)	-0.00093 (18)	0.00289 (18)	0.00233 (19)

Geometric parameters (Å, º) for (8-Pd)

C1—O2	1.225 (8)	C51—H51A	0.96

C1—O1	1.248 (8)	C51—H51B	0.96
C1—C10	1.524 (9)	C51—H51C	0.96
C2—O5	1.217 (8)	C52—H52A	0.96
C2—O3	1.244 (8)	C52—H52B	0.96
C2—C20	1.540 (11)	C52—H52C	0.96
C10—F13	1.262 (10)	C80—O80	1.257 (7)
C10—F11	1.262 (10)	C80—N80	1.335 (7)
C10—F12	1.278 (11)	C80—C81	1.471 (8)
C20—F23	1.221 (13)	C81—C86	1.384 (8)
C20—F21	1.230 (12)	C81—C82	1.390 (9)
C20—F22	1.301 (18)	C82—C83	1.370 (10)
C30—O30	1.258 (7)	C82—H82	0.93
C30—N30	1.310 (7)	C83—C84	1.381 (11)
C30—C31	1.483 (8)	C83—H83	0.93
C31—C36	1.371 (9)	C84—C85	1.368 (11)
C31—C32	1.385 (10)	C84—H84	0.93
C32—C33	1.384 (11)	C85—C86	1.368 (9)
C32—H32	0.93	C85—H85	0.93
C33—C34	1.349 (15)	C86—H86	0.93
C33—H33	0.93	C91—C92	1.396 (8)
C34—C35	1.364 (13)	C91—C96	1.402 (8)
C34—H34	0.93	C91—N80	1.415 (7)
C35—C36	1.392 (10)	C92—C93	1.396 (8)
C35—H35	0.93	C92—Pd2	1.945 (6)
C36—H36	0.93	C93—C94	1.375 (10)
C41—C46	1.377 (8)	C93—H93	0.93
C41—C42	1.393 (8)	C94—C95	1.352 (10)
C41—N30	1.433 (7)	C94—H94	0.93
C42—C43	1.391 (8)	C95—C96	1.372 (9)
C42—Pd1	1.954 (6)	C95—H95	0.93
C43—C44	1.388 (9)	C96—H96	0.93
C43—H43	0.93	N30—H30	0.86 (2)
C44—C45	1.364 (10)	N80—H80	0.85 (2)
C44—H44	0.93	O1—Pd1	2.058 (4)
C45—C46	1.382 (9)	O2—Pd2	2.180 (4)
C45—H45	0.93	O3—Pd2	2.069 (4)
C46—H46	0.93	O5—Pd1	2.191 (4)
C50—O50	1.221 (8)	O30—Pd1	1.994 (4)
C50—C51	1.449 (11)	O80—Pd2	1.999 (4)
C50—C52	1.485 (11)	Pd1—Pd2	2.9713 (6)

O2—C1—O1	130.6 (6)	O80—C80—N80	122.0 (5)
O2—C1—C10	115.1 (6)	O80—C80—C81	117.8 (5)
O1—C1—C10	114.2 (6)	N80—C80—C81	120.2 (5)
O5—C2—O3	132.4 (6)	C86—C81—C82	118.1 (6)
O5—C2—C20	114.0 (7)	C86—C81—C80	118.4 (6)
O3—C2—C20	113.6 (7)	C82—C81—C80	123.5 (5)
F13—C10—F11	107.1 (9)	C83—C82—C81	120.9 (7)
F13—C10—F12	105.1 (10)	C83—C82—H82	119.5
F11—C10—F12	102.8 (9)	C81—C82—H82	119.5
F13—C10—C1	115.0 (7)	C82—C83—C84	119.9 (7)
F11—C10—C1	114.8 (8)	C82—C83—H83	120.1
F12—C10—C1	110.8 (7)	C84—C83—H83	120.1
F23—C20—F21	113.9 (13)	C85—C84—C83	119.7 (7)
F23—C20—F22	97.6 (13)	C85—C84—H84	120.1
F21—C20—F22	100.9 (12)	C83—C84—H84	120.1
F23—C20—C2	114.9 (10)	C84—C85—C86	120.4 (7)
F21—C20—C2	117.4 (9)	C84—C85—H85	119.8
F22—C20—C2	108.8 (12)	C86—C85—H85	119.8
O30—C30—N30	124.0 (5)	C85—C86—C81	121.0 (7)
O30—C30—C31	117.7 (5)	C85—C86—H86	119.5
N30—C30—C31	118.3 (5)	C81—C86—H86	119.5
C36—C31—C32	119.5 (6)	C92—C91—C96	120.3 (5)
C36—C31—C30	119.3 (6)	C92—C91—N80	123.6 (5)
C32—C31—C30	121.2 (6)	C96—C91—N80	116.1 (5)
C33—C32—C31	119.8 (9)	C91—C92—C93	117.5 (6)
C33—C32—H32	120.1	C91—C92—Pd2	120.3 (4)
C31—C32—H32	120.1	C93—C92—Pd2	122.1 (5)
C34—C33—C32	120.2 (9)	C94—C93—C92	121.1 (7)
C34—C33—H33	119.9	C94—C93—H93	119.4
C32—C33—H33	119.9	C92—C93—H93	119.4
C33—C34—C35	120.8 (8)	C95—C94—C93	120.8 (6)
C33—C34—H34	119.6	C95—C94—H94	119.6
C35—C34—H34	119.6	C93—C94—H94	119.6
C34—C35—C36	119.9 (9)	C94—C95—C96	120.3 (6)
C34—C35—H35	120.1	C94—C95—H95	119.9
C36—C35—H35	120.1	C96—C95—H95	119.9
C31—C36—C35	119.8 (7)	C95—C96—C91	119.9 (6)
C31—C36—H36	120.1	C95—C96—H96	120.1
C35—C36—H36	120.1	C91—C96—H96	120.1

C46—C41—C42	121.3 (5)	C30—N30—C41	127.9 (5)
C46—C41—N30	116.0 (5)	C30—N30—H30	120 (5)
C42—C41—N30	122.7 (5)	C41—N30—H30	112 (5)
C43—C42—C41	117.4 (6)	C80—N80—C91	127.1 (5)
C43—C42—Pd1	121.5 (5)	C80—N80—H80	119 (5)
C41—C42—Pd1	121.1 (4)	C91—N80—H80	114 (5)
C44—C43—C42	120.8 (6)	C1—O1—Pd1	122.1 (4)
C44—C43—H43	119.6	C1—O2—Pd2	125.2 (4)
C42—C43—H43	119.6	C2—O3—Pd2	118.6 (4)
C45—C44—C43	120.8 (6)	C2—O5—Pd1	126.9 (4)
C45—C44—H44	119.6	C30—O30—Pd1	124.7 (3)
C43—C44—H44	119.6	C80—O80—Pd2	124.3 (3)
C44—C45—C46	119.2 (6)	C42—Pd1—O30	91.9 (2)
C44—C45—H45	120.4	C42—Pd1—O1	92.3 (2)
C46—C45—H45	120.4	O30—Pd1—O1	175.84 (17)
C41—C46—C45	120.4 (6)	C42—Pd1—O5	175.3 (2)
C41—C46—H46	119.8	O30—Pd1—O5	86.41 (17)
C45—C46—H46	119.8	O1—Pd1—O5	89.45 (18)
O50—C50—C51	121.2 (7)	C42—Pd1—Pd2	109.71 (16)
O50—C50—C52	120.8 (7)	O30—Pd1—Pd2	95.19 (12)
C51—C50—C52	118.0 (7)	O1—Pd1—Pd2	83.25 (13)
C50—C51—H51A	109.5	O5—Pd1—Pd2	74.82 (12)
C50—C51—H51B	109.5	C92—Pd2—O80	90.8 (2)
H51A—C51—H51B	109.5	C92—Pd2—O3	93.3 (2)
C50—C51—H51C	109.5	O80—Pd2—O3	175.89 (17)
H51A—C51—H51C	109.5	C92—Pd2—O2	176.7 (2)
H51B—C51—H51C	109.5	O80—Pd2—O2	86.44 (17)
C50—C52—H52A	109.5	O3—Pd2—O2	89.46 (18)
C50—C52—H52B	109.5	C92—Pd2—Pd1	105.57 (16)
H52A—C52—H52B	109.5	O80—Pd2—Pd1	93.73 (12)
C50—C52—H52C	109.5	O3—Pd2—Pd1	85.18 (13)
H52A—C52—H52C	109.5	O2—Pd2—Pd1	76.33 (12)
H52B—C52—H52C	109.5		
O2—C1—C10—F13	-148.0 (9)	C86—C81—C82—C83	-0.5 (11)
O1—C1—C10—F13	31.9 (12)	C80—C81—C82—C83	176.9 (7)
O2—C1—C10—F11	-23.0 (12)	C81—C82—C83—C84	0.5 (12)
O1—C1—C10—F11	156.9 (9)	C82—C83—C84—	0.0 (12)

		C85	
O2—C1—C10—F12	93.0 (10)	C83—C84—C85—C86	-0.6 (12)
O1—C1—C10—F12	-87.1 (10)	C84—C85—C86—C81	0.7 (12)
O5—C2—C20—F23	-26.4 (19)	C82—C81—C86—C85	-0.1 (10)
O3—C2—C20—F23	153.3 (14)	C80—C81—C86—C85	-177.7 (6)
O5—C2—C20—F21	-164.5 (13)	C96—C91—C92—C93	1.7 (8)
O3—C2—C20—F21	15.2 (18)	N80—C91—C92—C93	-177.8 (5)
O5—C2—C20—F22	81.8 (13)	C96—C91—C92—Pd2	-178.1 (4)
O3—C2—C20—F22	-98.5 (12)	N80—C91—C92—Pd2	2.4 (7)
O30—C30—C31—C36	-30.5 (8)	C91—C92—C93—C94	-1.7 (9)
N30—C30—C31—C36	148.9 (6)	Pd2—C92—C93—C94	178.0 (5)
O30—C30—C31—C32	150.5 (6)	C92—C93—C94—C95	-0.3 (10)
N30—C30—C31—C32	-30.1 (9)	C93—C94—C95—C96	2.4 (10)
C36—C31—C32—C33	-0.5 (12)	C94—C95—C96—C91	-2.5 (10)
C30—C31—C32—C33	178.6 (8)	C92—C91—C96—C95	0.4 (9)
C31—C32—C33—C34	-1.0 (15)	N80—C91—C96—C95	179.9 (6)
C32—C33—C34—C35	1.2 (16)	O30—C30—N30—C41	-7.4 (9)
C33—C34—C35—C36	0.0 (14)	C31—C30—N30—C41	173.2 (5)
C32—C31—C36—C35	1.7 (10)	C46—C41—N30—C30	-162.2 (6)
C30—C31—C36—C35	-177.4 (6)	C42—C41—N30—C30	17.6 (9)
C34—C35—C36—C31	-1.5 (11)	O80—C80—N80—C91	-5.9 (9)
C46—C41—C42—C43	2.2 (8)	C81—C80—N80—C91	175.6 (5)
N30—C41—C42—C43	-177.6 (5)	C92—C91—N80—C80	19.9 (9)
C46—C41—C42—Pd1	-178.2 (5)	C96—C91—N80—C80	-159.6 (6)
N30—C41—C42—	2.0 (7)	O2—C1—O1—Pd1	3.3 (10)

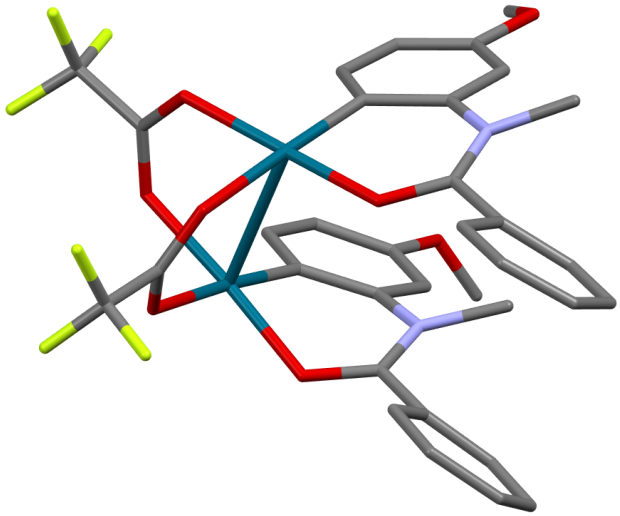
Pd1			
C41—C42—C43—C44	-1.9 (9)	C10—C1—O1—Pd1	-176.5 (5)
Pd1—C42—C43—C44	178.5 (5)	O1—C1—O2—Pd2	12.9 (10)
C42—C43—C44—C45	0.3 (10)	C10—C1—O2—Pd2	-167.2 (5)
C43—C44—C45—C46	1.0 (10)	O5—C2—O3—Pd2	9.2 (11)
C42—C41—C46—C45	-0.9 (9)	C20—C2—O3—Pd2	-170.4 (8)
N30—C41—C46—C45	178.9 (6)	O3—C2—O5—Pd1	5.6 (12)
C44—C45—C46—C41	-0.7 (10)	C20—C2—O5—Pd1	-174.8 (8)
O80—C80—C81—C86	-13.0 (8)	N30—C30—O30—Pd1	-20.6 (8)
N80—C80—C81—C86	165.5 (6)	C31—C30—O30—Pd1	158.8 (4)
O80—C80—C81—C82	169.6 (6)	N80—C80—O80—Pd2	-27.9 (7)
N80—C80—C81—C82	-11.9 (9)	C81—C80—O80—Pd2	150.6 (4)

Hydrogen-bond geometry (Å, º) for (8-Pd)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C52—H52*B*···F21^{i}	0.96	2.55	3.433 (13)	153
N30—H30···O50	0.86 (2)	2.22 (4)	2.980 (7)	149 (6)
N80—H80···O50	0.85 (2)	2.21 (4)	2.997 (7)	153 (6)

Symmetry code: (i) *x*, -*y*+3/2, *z*+1/2.

(44)



Crystal data

$C_{34}H_{26}F_6N_2O_8Pd_2$	$F(000)$ = 3632
M_r = 917.37	D_x = 1.672 Mg m^{-3}
Monoclinic, $C2/c$	Mo $K\alpha$ radiation, l = 0.71073 Å
a = 32.598 (4) Å	Cell parameters from 108 reflections
b = 12.5843 (11) Å	q = 3.4–17.9°
c = 22.685 (3) Å	m = 1.07 mm^{-1}
b = 128.447 (3)°	T = 295 K
V = 7288.2 (13) Å^3	Plate, orange
Z = 8	0.34 × 0.16 × 0.06 mm

Data collection

Bruker D8 VENTURE diffractometer	6671 independent reflections
Radiation source: microfocus sealed tube, INCOATEC ImS 3.0	4487 reflections with $I > 2s(I)$
Multilayer mirror INCOATEC monochromator	R_{int} = 0.077
Detector resolution: 7.3910 pixels mm^{-1}	q_{max} = 25.4°, q_{min} = 2.5°
w and p scan	h = -39→31
Absorption correction: multi-scan *SADABS2016*/2 - Bruker AXS area detector scaling and absorption correction	k = -15→15
T_{min} = 0.85, T_{max} = 0.94	l = -27→27
40549 measured reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier

	map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2s(F^2)] = 0.068$	H-atom parameters constrained
$wR(F^2) = 0.224$	$w = 1/[s^2(F_o^2) + (0.1122P)^2 + 63.548P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.05$	$(D/s)_{max} = 0.001$
6671 reflections	$D\rho_{max} = 1.59$ e Å^{-3}
472 parameters	$D\rho_{min} = -0.99$ e Å^{-3}
0 restraints	Extinction correction: *SHELXL2018*/3 (Sheldrick 2018)
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.00120 (15)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2) for (44)

	x	*y*	*z*	U_{iso}*/U_{eq}
C10	0.2841 (4)	0.2099 (7)	0.2827 (5)	0.062 (2)
H10A	0.248055	0.196321	0.25983	0.093*
H10B	0.306191	0.168218	0.32765	0.093*
H10C	0.290607	0.191197	0.248108	0.093*
C11	0.3502 (3)	0.3529 (7)	0.3574 (5)	0.052 (2)
C12	0.3857 (4)	0.2907 (8)	0.3577 (6)	0.069 (3)
H12	0.37371	0.238116	0.321506	0.083*
C13	0.4387 (4)	0.3075 (9)	0.4119 (6)	0.070 (3)
C14	0.4569 (4)	0.3842 (9)	0.4646 (6)	0.076 (3)
H14	0.492741	0.394493	0.501479	0.091*
C15	0.4214 (4)	0.4466 (8)	0.4624 (5)	0.067 (3)
H15	0.433839	0.500205	0.498079	0.081*
C16	0.3669 (3)	0.4325 (7)	0.4083 (5)	0.052 (2)
C17	0.4728 (11)	0.231 (3)	0.3618 (17)	0.28 (2)
H17A	0.445376	0.259113	0.315444	0.336*
H17B	0.498187	0.189315	0.365864	0.336*
C20	0.2574 (3)	0.3955 (7)	0.2711 (5)	0.052 (2)
C21	0.2036 (4)	0.3704 (7)	0.2064 (5)	0.057 (2)

C22	0.1637 (4)	0.4058 (10)	0.2075 (6)	0.083 (3)
H22	0.172273	0.442406	0.249482	0.1*
C23	0.1110 (5)	0.3881 (14)	0.1472 (9)	0.118 (5)
H23	0.084447	0.412826	0.148001	0.141*
C24	0.0998 (5)	0.3320 (13)	0.0862 (9)	0.114 (5)
H24	0.06501	0.317297	0.046191	0.137*
C25	0.1377 (5)	0.2975 (10)	0.0823 (7)	0.091 (4)
H25	0.128861	0.261584	0.040041	0.11*
C26	0.1908 (4)	0.3177 (8)	0.1442 (5)	0.068 (3)
H26	0.217166	0.29491	0.142435	0.081*
C40	0.3704 (4)	0.5397 (7)	0.5665 (5)	0.055 (2)
C41	0.4144 (5)	0.5817 (10)	0.6448 (7)	0.082 (3)
C60	0.2398 (5)	0.0233 (8)	0.3946 (7)	0.086 (3)
H60A	0.202336	0.01678	0.359452	0.129*
H60B	0.252445	-0.009065	0.441507	0.129*
H60C	0.255043	-0.011691	0.374799	0.129*
C61	0.3074 (4)	0.1659 (8)	0.4593 (6)	0.069 (3)
C62	0.3431 (5)	0.0842 (11)	0.4742 (9)	0.100 (4)
H62	0.330187	0.017806	0.452104	0.12*
C63	0.3954 (5)	0.1015 (11)	0.5200 (9)	0.113 (5)
C64	0.4152 (5)	0.1932 (10)	0.5535 (8)	0.098 (4)
H64	0.451225	0.204382	0.584967	0.117*
C65	0.3811 (5)	0.2742 (10)	0.5411 (7)	0.090 (4)
H65	0.396134	0.338845	0.565029	0.108*
C66	0.3260 (4)	0.2667 (8)	0.4957 (6)	0.071 (3)
C67	0.4785 (7)	0.039 (2)	0.559 (2)	0.29 (2)
H67A	0.489681	0.109742	0.567374	0.344*
H67B	0.501149	-0.014851	0.567706	0.344*
C70	0.2158 (4)	0.2096 (7)	0.3703 (5)	0.057 (2)
C71	0.1603 (4)	0.1799 (8)	0.3066 (5)	0.067 (3)
C72	0.1214 (5)	0.2285 (12)	0.3036 (7)	0.092 (4)
H72	0.129899	0.277736	0.34029	0.111*
C73	0.0701 (6)	0.2046 (15)	0.2469 (10)	0.122 (5)
H73	0.044121	0.231711	0.248202	0.147*
C74	0.0566 (6)	0.1420 (14)	0.1888 (9)	0.108 (4)
H74	0.021392	0.130539	0.148986	0.13*
C75	0.0929 (6)	0.0968 (13)	0.1880 (8)	0.108 (5)
H75	0.083406	0.049947	0.149722	0.13*
C76	0.1458 (6)	0.1203 (11)	0.2455 (8)	0.098 (4)
H76	0.171238	0.0955	0.242344	0.117*

C90	0.2326 (4)	0.6044 (8)	0.4119 (5)	0.063 (2)
C91	0.1907 (6)	0.6868 (11)	0.3918 (8)	0.096 (4)
N1	0.2956 (3)	0.3242 (5)	0.3018 (4)	0.0500 (16)
N51	0.2548 (3)	0.1404 (6)	0.4074 (5)	0.0610 (19)
O1	0.2655 (2)	0.4898 (4)	0.2961 (3)	0.0561 (15)
O13	0.4756 (4)	0.2468 (10)	0.4162 (6)	0.123 (4)
O41	0.3715 (3)	0.5711 (6)	0.5161 (4)	0.0683 (17)
O42	0.3412 (3)	0.4748 (6)	0.5647 (4)	0.0695 (18)
O51	0.2227 (3)	0.3085 (6)	0.3835 (4)	0.083 (2)
O63	0.4273 (4)	0.0150 (9)	0.5321 (10)	0.190 (8)
O91	0.2602 (3)	0.6302 (5)	0.3944 (4)	0.0701 (18)
O92	0.2304 (3)	0.5212 (5)	0.4396 (4)	0.0683 (17)
F41	0.4029 (4)	0.5821 (8)	0.6898 (4)	0.137 (3)
F42	0.4252 (3)	0.6811 (7)	0.6413 (5)	0.131 (3)
F43	0.4551 (3)	0.5278 (10)	0.6728 (5)	0.175 (5)
F91	0.2023 (5)	0.7812 (8)	0.3848 (10)	0.214 (7)
F92	0.1816 (5)	0.6920 (11)	0.4380 (7)	0.195 (6)
F93	0.1476 (4)	0.6697 (10)	0.3275 (7)	0.188 (6)
Pd1	0.31746 (3)	0.52541 (5)	0.40513 (4)	0.0542 (3)
Pd2	0.28169 (3)	0.38788 (6)	0.47067 (4)	0.0610 (3)

Atomic displacement parameters ($Å^2$) for (44)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C10	0.068 (6)	0.042 (5)	0.061 (6)	-0.002 (4)	0.033 (5)	-0.002 (4)
C11	0.043 (5)	0.059 (5)	0.041 (4)	0.000 (4)	0.020 (4)	-0.005 (4)
C12	0.065 (6)	0.076 (6)	0.059 (6)	0.010 (5)	0.034 (5)	-0.005 (5)
C13	0.049 (6)	0.087 (7)	0.065 (6)	0.011 (5)	0.031 (5)	0.000 (5)
C14	0.043 (5)	0.100 (8)	0.070 (7)	-0.005 (5)	0.027 (5)	-0.006 (6)
C15	0.064 (6)	0.070 (6)	0.054 (5)	-0.021 (5)	0.030 (5)	-0.016 (5)
C16	0.049 (5)	0.056 (5)	0.051 (5)	-0.008 (4)	0.032 (4)	-0.003 (4)
C17	0.26 (3)	0.44 (5)	0.23 (3)	0.24 (3)	0.19 (3)	0.14 (3)
C20	0.056 (5)	0.059 (5)	0.045 (5)	0.004 (4)	0.034 (4)	0.008 (4)
C21	0.049 (5)	0.059 (5)	0.050 (5)	-0.003 (4)	0.024 (4)	0.009 (4)
C22	0.066 (7)	0.114 (10)	0.061 (6)	0.007 (6)	0.034 (6)	0.002 (6)
C23	0.058 (8)	0.174 (16)	0.103 (11)	0.008 (9)	0.041 (8)	0.008 (11)
C24	0.057 (8)	0.118 (11)	0.098 (11)	-0.004 (8)	0.013 (8)	0.007 (9)
C25	0.079 (8)	0.083 (8)	0.062 (7)	-0.010 (6)	0.019 (6)	-0.010 (6)
C26	0.064 (6)	0.059 (6)	0.056 (6)	0.000 (5)	0.025 (5)	-0.001 (4)
C40	0.051 (5)	0.056 (5)	0.049 (5)	-0.001 (4)	0.026 (4)	-0.010 (4)
C41	0.074 (8)	0.079 (8)	0.064 (7)	-0.011 (6)	0.029 (6)	-0.020 (6)

C60	0.098 (9)	0.059 (6)	0.093 (9)	0.002 (6)	0.055 (8)	-0.004 (6)
C61	0.076 (7)	0.062 (6)	0.084 (7)	0.014 (5)	0.057 (6)	0.021 (5)
C62	0.075 (8)	0.079 (8)	0.137 (12)	0.003 (6)	0.061 (9)	-0.002 (8)
C63	0.072 (9)	0.080 (9)	0.123 (12)	0.012 (7)	0.029 (8)	-0.009 (8)
C64	0.063 (7)	0.078 (8)	0.129 (11)	0.005 (6)	0.048 (8)	0.006 (8)
C65	0.084 (8)	0.072 (7)	0.086 (8)	0.006 (6)	0.038 (7)	-0.005 (6)
C66	0.045 (5)	0.069 (6)	0.057 (6)	-0.006 (4)	0.011 (5)	0.016 (5)
C67	0.084 (14)	0.16 (2)	0.48 (6)	-0.013 (13)	0.10 (2)	-0.09 (3)
C70	0.065 (6)	0.051 (5)	0.059 (5)	-0.013 (4)	0.041 (5)	-0.001 (4)
C71	0.076 (7)	0.074 (6)	0.058 (6)	-0.021 (5)	0.045 (5)	-0.013 (5)
C72	0.063 (7)	0.140 (12)	0.067 (7)	0.009 (7)	0.037 (6)	0.003 (7)
C73	0.075 (10)	0.166 (16)	0.111 (12)	0.025 (10)	0.051 (9)	0.025 (12)
C74	0.077 (9)	0.132 (12)	0.100 (11)	-0.009 (9)	0.047 (9)	-0.007 (9)
C75	0.078 (9)	0.133 (12)	0.089 (10)	-0.016 (8)	0.040 (8)	-0.028 (8)
C76	0.101 (10)	0.106 (10)	0.095 (9)	0.009 (8)	0.065 (9)	-0.004 (7)
C90	0.061 (6)	0.056 (6)	0.051 (5)	0.005 (5)	0.024 (5)	-0.007 (4)
C91	0.097 (10)	0.088 (9)	0.081 (8)	0.033 (7)	0.045 (8)	0.002 (7)
N1	0.049 (4)	0.042 (4)	0.049 (4)	-0.001 (3)	0.026 (3)	-0.001 (3)
N51	0.076 (6)	0.053 (4)	0.065 (5)	0.000 (4)	0.049 (5)	-0.001 (4)
O1	0.060 (4)	0.046 (3)	0.051 (3)	0.009 (3)	0.029 (3)	0.000 (3)
O13	0.081 (6)	0.180 (10)	0.098 (7)	0.051 (6)	0.050 (6)	-0.004 (7)
O41	0.071 (4)	0.072 (4)	0.055 (4)	-0.011 (3)	0.036 (4)	-0.016 (3)
O42	0.065 (4)	0.080 (5)	0.048 (4)	-0.007 (4)	0.028 (3)	-0.009 (3)
O51	0.059 (4)	0.066 (4)	0.069 (4)	0.001 (3)	0.014 (4)	0.006 (3)
O63	0.087 (7)	0.117 (8)	0.31 (2)	0.007 (6)	0.099 (11)	-0.060 (10)
O91	0.088 (5)	0.056 (4)	0.083 (5)	0.009 (3)	0.061 (5)	0.007 (3)
O92	0.066 (4)	0.063 (4)	0.078 (5)	0.002 (3)	0.046 (4)	0.003 (3)
F41	0.155 (8)	0.176 (9)	0.073 (5)	-0.030 (7)	0.068 (6)	-0.042 (5)
F42	0.119 (6)	0.124 (7)	0.097 (6)	-0.054 (5)	0.042 (5)	-0.047 (5)
F43	0.078 (5)	0.206 (11)	0.108 (7)	0.045 (6)	-0.008 (5)	-0.053 (7)
F91	0.184 (11)	0.079 (6)	0.40 (2)	0.061 (7)	0.192 (14)	0.041 (9)
F92	0.224 (12)	0.239 (14)	0.184 (11)	0.145 (11)	0.157 (10)	0.055 (9)
F93	0.101 (7)	0.192 (11)	0.144 (9)	0.067 (7)	0.014 (7)	-0.025 (8)
Pd1	0.0627 (5)	0.0494 (4)	0.0518 (4)	-0.0030 (3)	0.0363 (4)	-0.0046 (3)
Pd2	0.0648 (5)	0.0569 (5)	0.0486 (4)	-0.0086 (3)	0.0289 (4)	0.0016 (3)

Geometric parameters (Å, º) for (44)

C10—N1	1.482 (10)	C61—N51	1.385 (13)
C10—H10A	0.96	C61—C66	1.426 (15)
C10—H10B	0.96	C61—C62	1.427 (15)

C10—H10C	0.96	C62—C63	1.353 (18)
C11—C16	1.360 (12)	C62—H62	0.93
C11—C12	1.394 (13)	C63—C64	1.310 (17)
C11—N1	1.446 (11)	C63—O63	1.409 (16)
C12—C13	1.378 (14)	C64—C65	1.402 (16)
C12—H12	0.93	C64—H64	0.93
C13—C14	1.353 (15)	C65—C66	1.414 (15)
C13—O13	1.377 (13)	C65—H65	0.93
C14—C15	1.374 (15)	C66—Pd2	1.927 (11)
C14—H14	0.93	C67—O63	1.41 (2)
C15—C16	1.408 (13)	C67—H67A	0.93
C15—H15	0.93	C67—H67B	0.93
C16—Pd1	1.957 (9)	C70—O51	1.267 (11)
C17—O13	1.20 (3)	C70—N51	1.325 (12)
C17—H17A	0.93	C70—C71	1.500 (14)
C17—H17B	0.93	C71—C72	1.372 (15)
C20—O1	1.270 (10)	C71—C76	1.374 (16)
C20—N1	1.328 (11)	C72—C73	1.366 (19)
C20—C21	1.459 (12)	C72—H72	0.93
C21—C26	1.370 (14)	C73—C74	1.35 (2)
C21—C22	1.390 (15)	C73—H73	0.93
C22—C23	1.396 (18)	C74—C75	1.32 (2)
C22—H22	0.93	C74—H74	0.93
C23—C24	1.39 (2)	C75—C76	1.402 (19)
C23—H23	0.93	C75—H75	0.93
C24—C25	1.36 (2)	C76—H76	0.93
C24—H24	0.93	C90—O91	1.231 (12)
C25—C26	1.418 (15)	C90—O92	1.246 (12)
C25—H25	0.93	C90—C91	1.541 (15)
C26—H26	0.93	C91—F92	1.254 (16)
C40—O41	1.230 (11)	C91—F93	1.265 (16)
C40—O42	1.236 (11)	C91—F91	1.287 (18)
C40—C41	1.524 (14)	O1—Pd1	1.994 (6)
C41—F43	1.255 (14)	O41—Pd1	2.062 (6)
C41—F41	1.288 (15)	O42—Pd2	2.089 (7)
C41—F42	1.315 (14)	O51—Pd2	1.969 (7)
C60—N51	1.523 (13)	O91—Pd1	2.173 (7)
C60—H60A	0.96	O92—Pd2	2.155 (7)
C60—H60B	0.96	Pd1—Pd2	2.9533 (10)
C60—H60C	0.96		

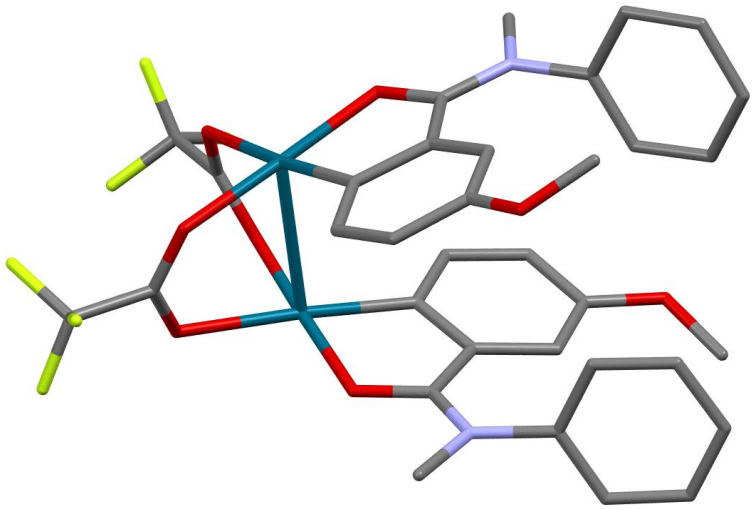
N1—C10—H10A	109.5	C65—C64—H64	120.6
N1—C10—H10B	109.5	C64—C65—C66	126.0 (11)
H10A—C10—H10B	109.5	C64—C65—H65	117.0
N1—C10—H10C	109.5	C66—C65—H65	117.0
H10A—C10—H10C	109.5	C65—C66—C61	111.8 (10)
H10B—C10—H10C	109.5	C65—C66—Pd2	122.8 (8)
C16—C11—C12	121.0 (8)	C61—C66—Pd2	124.2 (7)
C16—C11—N1	123.4 (8)	O63—C67—H67A	120.0
C12—C11—N1	115.6 (8)	O63—C67—H67B	120.0
C13—C12—C11	119.6 (9)	H67A—C67—H67B	120.0
C13—C12—H12	120.2	O51—C70—N51	122.4 (9)
C11—C12—H12	120.2	O51—C70—C71	113.8 (9)
C14—C13—O13	116.6 (10)	N51—C70—C71	123.7 (8)
C14—C13—C12	121.2 (10)	C72—C71—C76	117.8 (11)
O13—C13—C12	122.2 (10)	C72—C71—C70	117.4 (9)
C13—C14—C15	118.6 (9)	C76—C71—C70	124.0 (11)
C13—C14—H14	120.7	C73—C72—C71	120.0 (14)
C15—C14—H14	120.7	C73—C72—H72	120.0
C14—C15—C16	122.4 (9)	C71—C72—H72	120.0
C14—C15—H15	118.8	C74—C73—C72	120.8 (15)
C16—C15—H15	118.8	C74—C73—H73	119.6
C11—C16—C15	117.2 (9)	C72—C73—H73	119.6
C11—C16—Pd1	121.5 (7)	C75—C74—C73	120.8 (14)
C15—C16—Pd1	121.3 (7)	C75—C74—H74	119.6
O13—C17—H17A	120.0	C73—C74—H74	119.6
O13—C17—H17B	120.0	C74—C75—C76	119.3 (14)
H17A—C17—H17B	120.0	C74—C75—H75	120.3
O1—C20—N1	122.0 (8)	C76—C75—H75	120.3
O1—C20—C21	116.0 (8)	C71—C76—C75	120.4 (13)
N1—C20—C21	122.0 (8)	C71—C76—H76	119.8
C26—C21—C22	119.0 (9)	C75—C76—H76	119.8
C26—C21—C20	123.1 (9)	O91—C90—O92	131.9 (9)
C22—C21—C20	117.8 (9)	O91—C90—C91	114.7 (10)
C21—C22—C23	121.7 (12)	O92—C90—C91	113.3 (11)
C21—C22—H22	119.1	F92—C91—F93	107.7 (15)
C23—C22—H22	119.1	F92—C91—F91	107.2 (14)
C24—C23—C22	117.3 (14)	F93—C91—F91	102.4 (14)
C24—C23—H23	121.3	F92—C91—C90	114.5 (12)
C22—C23—H23	121.3	F93—C91—C90	111.7 (11)

C25—C24—C23	122.8 (12)	F91—C91—C90	112.5 (13)
C25—C24—H24	118.6	C20—N1—C11	122.2 (7)
C23—C24—H24	118.6	C20—N1—C10	121.2 (7)
C24—C25—C26	118.5 (12)	C11—N1—C10	116.6 (7)
C24—C25—H25	120.7	C70—N51—C61	125.4 (8)
C26—C25—H25	120.7	C70—N51—C60	116.5 (9)
C21—C26—C25	120.5 (11)	C61—N51—C60	117.9 (9)
C21—C26—H26	119.7	C20—O1—Pd1	123.0 (5)
C25—C26—H26	119.7	C17—O13—C13	121.4 (15)
O41—C40—O42	130.3 (9)	C40—O41—Pd1	123.3 (6)
O41—C40—C41	115.0 (9)	C40—O42—Pd2	126.0 (6)
O42—C40—C41	114.5 (9)	C70—O51—Pd2	130.9 (6)
F43—C41—F41	109.7 (13)	C63—O63—C67	116.6 (15)
F43—C41—F42	107.9 (12)	C90—O91—Pd1	123.6 (6)
F41—C41—F42	105.3 (11)	C90—O92—Pd2	122.8 (7)
F43—C41—C40	109.6 (10)	C16—Pd1—O1	87.5 (3)
F41—C41—C40	113.5 (11)	C16—Pd1—O41	93.6 (3)
F42—C41—C40	110.7 (10)	O1—Pd1—O41	176.6 (3)
N51—C60—H60A	109.5	C16—Pd1—O91	176.5 (3)
N51—C60—H60B	109.5	O1—Pd1—O91	89.4 (3)
H60A—C60—H60B	109.5	O41—Pd1—O91	89.4 (3)
N51—C60—H60C	109.5	C16—Pd1—Pd2	101.9 (2)
H60A—C60—H60C	109.5	O1—Pd1—Pd2	101.31 (18)
H60B—C60—H60C	109.5	O41—Pd1—Pd2	81.6 (2)
N51—C61—C66	123.7 (9)	O91—Pd1—Pd2	80.28 (19)
N51—C61—C62	115.5 (10)	C66—Pd2—O51	88.8 (4)
C66—C61—C62	120.9 (11)	C66—Pd2—O42	93.7 (3)
C63—C62—C61	121.2 (13)	O51—Pd2—O42	176.5 (3)
C63—C62—H62	119.4	C66—Pd2—O92	178.3 (4)
C61—C62—H62	119.4	O51—Pd2—O92	89.5 (3)
C64—C63—C62	121.3 (13)	O42—Pd2—O92	88.0 (3)
C64—C63—O63	122.0 (13)	C66—Pd2—Pd1	98.8 (3)
C62—C63—O63	116.7 (12)	O51—Pd2—Pd1	103.7 (2)
C63—C64—C65	118.7 (12)	O42—Pd2—Pd1	78.5 (2)
C63—C64—H64	120.6	O92—Pd2—Pd1	81.29 (19)
C16—C11—C12—C13	2.2 (15)	N51—C70—C71—C76	-50.7 (15)
N1—C11—C12—C13	-175.6 (9)	C76—C71—C72—C73	9.6 (19)
C11—C12—C13—	-0.7 (17)	C70—C71—C72—	-180.0 (12)

C14		C73	
C11—C12—C13—O13	178.3 (11)	C71—C72—C73—C74	-7 (2)
O13—C13—C14—C15	-180.0 (11)	C72—C73—C74—C75	4 (3)
C12—C13—C14—C15	-0.9 (18)	C73—C74—C75—C76	-5 (3)
C13—C14—C15—C16	1.0 (17)	C72—C71—C76—C75	-9.8 (19)
C12—C11—C16—C15	-2.1 (14)	C70—C71—C76—C75	-179.5 (12)
N1—C11—C16—C15	175.6 (8)	C74—C75—C76—C71	7 (2)
C12—C11—C16—Pd1	176.6 (7)	O91—C90—C91—F92	-145.4 (14)
N1—C11—C16—Pd1	-5.7 (12)	O92—C90—C91—F92	38.0 (17)
C14—C15—C16—C11	0.4 (15)	O91—C90—C91—F93	91.8 (16)
C14—C15—C16—Pd1	-178.3 (8)	O92—C90—C91—F93	-84.8 (16)
O1—C20—C21—C26	133.6 (9)	O91—C90—C91—F91	-22.7 (17)
N1—C20—C21—C26	-46.3 (13)	O92—C90—C91—F91	160.7 (13)
O1—C20—C21—C22	-43.7 (12)	O1—C20—N1—C11	-10.4 (13)
N1—C20—C21—C22	136.5 (10)	C21—C20—N1—C11	169.5 (8)
C26—C21—C22—C23	0.8 (18)	O1—C20—N1—C10	167.3 (8)
C20—C21—C22—C23	178.2 (12)	C21—C20—N1—C10	-12.9 (12)
C21—C22—C23—C24	1 (2)	C16—C11—N1—C20	32.7 (13)
C22—C23—C24—C25	-2 (2)	C12—C11—N1—C20	-149.5 (9)
C23—C24—C25—C26	2 (2)	C16—C11—N1—C10	-145.0 (9)
C22—C21—C26—C25	-1.3 (15)	C12—C11—N1—C10	32.8 (11)
C20—C21—C26—C25	-178.6 (9)	O51—C70—N51—C61	-5.9 (15)
C24—C25—C26—C21	0.2 (18)	C71—C70—N51—C61	170.7 (9)
O41—C40—C41—F43	-81.4 (14)	O51—C70—N51—C60	170.5 (9)
O42—C40—C41—F43	94.7 (14)	C71—C70—N51—C60	-12.9 (13)
O41—C40—C41—	155.6 (11)	C66—C61—N51—	18.4 (15)

F41		C70	
O42—C40—C41—F41	-28.3 (14)	C62—C61—N51—C70	-160.8 (10)
O41—C40—C41—F42	37.5 (14)	C66—C61—N51—C60	-157.9 (10)
O42—C40—C41—F42	-146.5 (10)	C62—C61—N51—C60	22.9 (14)
N51—C61—C62—C63	175.5 (14)	N1—C20—O1—Pd1	-34.9 (11)
C66—C61—C62—C63	-4 (2)	C21—C20—O1—Pd1	145.3 (6)
C61—C62—C63—C64	2 (3)	C14—C13—O13—C17	-132 (3)
C61—C62—C63—O63	178.8 (15)	C12—C13—O13—C17	49 (3)
C62—C63—C64—C65	0 (3)	O42—C40—O41—Pd1	3.8 (15)
O63—C63—C64—C65	-177.3 (17)	C41—C40—O41—Pd1	179.1 (7)
C63—C64—C65—C66	1 (2)	O41—C40—O42—Pd2	2.2 (15)
C64—C65—C66—C61	-2.8 (19)	C41—C40—O42—Pd2	-173.2 (7)
C64—C65—C66—Pd2	-171.0 (11)	N51—C70—O51—Pd2	-17.5 (15)
N51—C61—C66—C65	-175.2 (10)	C71—C70—O51—Pd2	165.5 (7)
C62—C61—C66—C65	4.0 (16)	C64—C63—O63—C67	-22 (3)
N51—C61—C66—Pd2	-7.2 (15)	C62—C63—O63—C67	160 (2)
C62—C61—C66—Pd2	172.0 (10)	O92—C90—O91—Pd1	2.0 (15)
O51—C70—C71—C72	-43.5 (13)	C91—C90—O91—Pd1	-173.8 (7)
N51—C70—C71—C72	139.5 (11)	O91—C90—O92—Pd2	2.2 (15)
O51—C70—C71—C76	126.2 (12)	C91—C90—O92—Pd2	178.0 (7)

(45)



Crystal data

$C_{17}H_{14}F_3NO_4Pd$	$F(000)$ = 1824
M_r = 459.69	D_x = 1.760 Mg m^{-3}
Monoclinic, *C*2/*c*	Mo *K*a radiation, l = 0.71073 Å
a = 20.9754 (8) Å	Cell parameters from 154 reflections
b = 13.1973 (6) Å	q = 5.1–25.4°
c = 15.3039 (11) Å	m = 1.12 mm^{-1}
b = 125.008 (1)°	*T* = 295 K
V = 3469.9 (3) Å^3	Rod, yellow
Z = 8	0.46 × 0.14 × 0.06 mm

Data collection

Bruker D8 VENTURE diffractometer	3279 independent reflections
Radiation source: microfocus sealed tube, INCOATEC ImS 3.0	2460 reflections with $I > 2s(I)$
Multilayer mirror INCOATEC monochromator	R_{int} = 0.124
Detector resolution: 7.3910 pixels mm^{-1}	q_{max} = 25.7°, q_{min} = 2.7°
w and p scan	*h* = -25→25
Absorption correction: multi-scan *SADABS2016*/2 - Bruker AXS area detector scaling and absorption correction	*k* = -16→16
T_{min} = 0.526, T_{max} = 0.745	*l* = -18→18
48632 measured reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier

	map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2s(F^2)] = 0.050$	H-atom parameters constrained
$wR(F^2) = 0.095$	$w = 1/[s^2(F_o^2) + (0.0478P)^2 + 3.3885P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.04$	$(D/s)_{max} = 0.001$
3279 reflections	$D\rho_{max} = 0.74$ e Å^{-3}
238 parameters	$D\rho_{min} = -0.49$ e Å^{-3}
0 restraints	Extinction correction: *SHELXL2018*/3 (Sheldrick 2018)
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.00060 (16)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2) for (45)

	x	*y*	*z*	U_{iso}*/U_{eq}
C10	0.6987 (2)	0.4153 (4)	0.5549 (4)	0.0934 (17)
H10A	0.730892	0.364076	0.606277	0.14*
H10B	0.702896	0.476841	0.591279	0.14*
H10C	0.715461	0.42726	0.509148	0.14*
C11	0.47658 (18)	0.4304 (3)	0.3708 (3)	0.0438 (8)
C12	0.4382 (2)	0.3401 (3)	0.3613 (3)	0.0486 (9)
H12	0.466518	0.283651	0.401175	0.058*
C13	0.3582 (2)	0.3349 (3)	0.2926 (3)	0.0523 (9)
C14	0.3163 (2)	0.4190 (3)	0.2332 (3)	0.0669 (12)
H14	0.262489	0.414662	0.185516	0.08*
C15	0.3532 (2)	0.5083 (3)	0.2439 (4)	0.0666 (12)
H15	0.324083	0.564543	0.204329	0.08*
C16	0.43362 (19)	0.5159 (3)	0.3132 (3)	0.0488 (9)
C17	0.3546 (3)	0.1669 (4)	0.3467 (4)	0.0790 (14)
H17A	0.317563	0.114973	0.331821	0.119*
H17B	0.380397	0.18899	0.419221	0.119*
H17C	0.392317	0.14037	0.336094	0.119*
C20	0.56112 (19)	0.4498 (3)	0.4368 (3)	0.0488 (9)

C21	0.60475 (19)	0.2734 (3)	0.4813 (3)	0.0516 (9)
C22	0.6063 (2)	0.2213 (4)	0.5603 (3)	0.0632 (11)
H22	0.616635	0.254944	0.620539	0.076*
C23	0.5925 (3)	0.1188 (4)	0.5489 (5)	0.0808 (15)
H23	0.592016	0.083192	0.600963	0.097*
C24	0.5796 (3)	0.0692 (4)	0.4628 (5)	0.0894 (16)
H24	0.5695	1.2e-05	0.455336	0.107*
C25	0.5814 (3)	0.1207 (4)	0.3869 (5)	0.0919 (17)
H25	0.574383	0.085999	0.329025	0.11*
C26	0.5936 (3)	0.2238 (4)	0.3957 (4)	0.0717 (12)
H26	0.594292	0.259179	0.343642	0.086*
C40	0.3914 (2)	0.7720 (3)	0.1627 (3)	0.0510 (9)
C41	0.3256 (3)	0.8508 (3)	0.1162 (4)	0.0674 (12)
O1	0.58231 (13)	0.54143 (19)	0.4432 (2)	0.0563 (7)
O3	0.31554 (14)	0.2498 (2)	0.2772 (2)	0.0703 (8)
O41	0.40201 (15)	0.7275 (2)	0.2413 (2)	0.0629 (7)
O42	0.57543 (15)	0.7631 (2)	0.3822 (2)	0.0597 (7)
F41	0.3493 (2)	0.9293 (2)	0.1809 (3)	0.1132 (11)
F42	0.30512 (19)	0.8883 (2)	0.0250 (3)	0.1037 (10)
F43	0.26368 (17)	0.8155 (3)	0.1053 (3)	0.1223 (13)
Pd1	0.49581 (2)	0.63621 (2)	0.34122 (2)	0.05007 (15)
N1	0.61716 (16)	0.3810 (2)	0.4905 (3)	0.0580 (9)

Atomic displacement parameters (Å^2) for (45)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C10	0.044 (2)	0.083 (4)	0.102 (4)	-0.002 (2)	0.012 (2)	0.017 (3)
C11	0.0379 (16)	0.051 (2)	0.0419 (19)	0.0021 (14)	0.0223 (15)	0.0032 (16)
C12	0.0480 (19)	0.049 (2)	0.048 (2)	0.0050 (15)	0.0272 (17)	0.0075 (17)
C13	0.0428 (18)	0.057 (2)	0.054 (2)	-0.0059 (16)	0.0265 (18)	-0.0001 (19)
C14	0.0383 (18)	0.080 (3)	0.070 (3)	0.0007 (19)	0.024 (2)	0.013 (2)
C15	0.046 (2)	0.064 (3)	0.081 (3)	0.0119 (18)	0.031 (2)	0.026 (2)
C16	0.0396 (17)	0.057 (2)	0.050 (2)	0.0007 (15)	0.0255 (17)	0.0055 (18)
C17	0.072 (3)	0.067 (3)	0.081 (3)	-0.018 (2)	0.034 (3)	0.015 (3)
C20	0.0428 (17)	0.054 (2)	0.043 (2)	0.0016 (16)	0.0206 (17)	0.0066 (18)
C21	0.0386 (17)	0.052 (2)	0.054 (2)	0.0098 (15)	0.0210 (17)	0.010 (2)
C22	0.057 (2)	0.077 (3)	0.053 (3)	0.012 (2)	0.030 (2)	0.016 (2)
C23	0.072 (3)	0.072 (4)	0.092 (4)	0.015 (2)	0.043 (3)	0.038 (3)
C24	0.083 (3)	0.054 (3)	0.110 (5)	0.011 (2)	0.043 (3)	0.007 (3)
C25	0.098 (4)	0.084 (4)	0.084 (4)	0.020 (3)	0.046 (3)	-0.007 (3)
C26	0.076 (3)	0.077 (3)	0.068 (3)	0.011 (2)	0.044 (3)	0.010 (3)

C40	0.054 (2)	0.036 (2)	0.063 (3)	0.0006 (16)	0.034 (2)	0.0044 (19)
C41	0.069 (3)	0.049 (3)	0.084 (3)	0.012 (2)	0.043 (3)	0.013 (2)
O1	0.0455 (13)	0.0485 (16)	0.0614 (17)	-0.0029 (11)	0.0228 (13)	0.0076 (13)
O3	0.0503 (15)	0.0692 (19)	0.077 (2)	-0.0156 (13)	0.0279 (15)	0.0032 (16)
O41	0.0651 (16)	0.0585 (18)	0.077 (2)	0.0191 (13)	0.0476 (16)	0.0232 (16)
O42	0.0682 (16)	0.0514 (16)	0.0660 (18)	-0.0071 (13)	0.0424 (15)	-0.0062 (14)
F41	0.143 (3)	0.064 (2)	0.117 (3)	0.0358 (18)	0.065 (2)	0.0016 (18)
F42	0.112 (2)	0.098 (2)	0.101 (2)	0.0505 (18)	0.062 (2)	0.0479 (19)
F43	0.0812 (19)	0.106 (2)	0.197 (4)	0.0348 (17)	0.089 (2)	0.058 (2)
Pd1	0.05009 (19)	0.0472 (2)	0.0562 (2)	0.00347 (12)	0.03244 (16)	0.00813 (14)
N1	0.0382 (15)	0.054 (2)	0.061 (2)	0.0024 (13)	0.0162 (15)	0.0096 (16)

Geometric parameters (Å, º) for (45)

C10—N1	1.472 (5)	C21—C26	1.360 (6)
C10—H10A	0.96	C21—C22	1.374 (6)
C10—H10B	0.96	C21—N1	1.436 (5)
C10—H10C	0.96	C22—C23	1.373 (6)
C11—C16	1.397 (5)	C22—H22	0.93
C11—C12	1.399 (5)	C23—C24	1.353 (7)
C11—C20	1.475 (4)	C23—H23	0.93
C12—C13	1.378 (5)	C24—C25	1.365 (8)
C12—H12	0.93	C24—H24	0.93
C13—O3	1.370 (4)	C25—C26	1.377 (7)
C13—C14	1.383 (6)	C25—H25	0.93
C14—C15	1.368 (6)	C26—H26	0.93
C14—H14	0.93	C40—O42^{i}	1.234 (5)
C15—C16	1.389 (5)	C40—O41	1.238 (5)
C15—H15	0.93	C40—C41	1.538 (5)
C16—Pd1	1.940 (4)	C41—F43	1.297 (5)
C17—O3	1.414 (5)	C41—F42	1.300 (6)
C17—H17A	0.96	C41—F41	1.318 (5)
C17—H17B	0.96	O1—Pd1	2.011 (2)
C17—H17C	0.96	O41—Pd1	2.051 (3)
C20—O1	1.273 (4)	O42—Pd1	2.188 (3)
C20—N1	1.331 (4)	Pd1—Pd1^{i}	2.8963 (6)
N1—C10—H10A	109.5	C24—C23—C22	120.7 (5)
N1—C10—H10B	109.5	C24—C23—H23	119.7
H10A—C10—H10B	109.5	C22—C23—H23	119.7
N1—C10—H10C	109.5	C23—C24—C25	120.1 (5)

H10A—C10—H10C	109.5	C23—C24—H24	120.0
H10B—C10—H10C	109.5	C25—C24—H24	120.0
C16—C11—C12	119.9 (3)	C24—C25—C26	120.2 (5)
C16—C11—C20	112.3 (3)	C24—C25—H25	119.9
C12—C11—C20	127.8 (3)	C26—C25—H25	119.9
C13—C12—C11	119.9 (3)	C21—C26—C25	119.3 (5)
C13—C12—H12	120.1	C21—C26—H26	120.4
C11—C12—H12	120.1	C25—C26—H26	120.4
O3—C13—C12	124.1 (4)	O42^{i}—C40—O41	130.6 (4)
O3—C13—C14	116.0 (3)	O42^{i}—C40—C41	116.7 (4)
C12—C13—C14	119.9 (4)	O41—C40—C41	112.6 (4)
C15—C14—C13	120.7 (4)	F43—C41—F42	108.0 (4)
C15—C14—H14	119.6	F43—C41—F41	107.3 (4)
C13—C14—H14	119.6	F42—C41—F41	104.9 (4)
C14—C15—C16	120.6 (4)	F43—C41—C40	112.5 (3)
C14—C15—H15	119.7	F42—C41—C40	114.2 (4)
C16—C15—H15	119.7	F41—C41—C40	109.4 (4)
C15—C16—C11	119.0 (3)	C20—O1—Pd1	114.3 (2)
C15—C16—Pd1	126.6 (3)	C13—O3—C17	117.8 (3)
C11—C16—Pd1	114.4 (2)	C40—O41—Pd1	124.4 (2)
O3—C17—H17A	109.5	C40^{i}—O42—Pd1	119.7 (2)
O3—C17—H17B	109.5	C16—Pd1—O1	81.69 (12)
H17A—C17—H17B	109.5	C16—Pd1—O41	94.58 (13)
O3—C17—H17C	109.5	O1—Pd1—O41	175.09 (10)
H17A—C17—H17C	109.5	C16—Pd1—O42	174.71 (13)
H17B—C17—H17C	109.5	O1—Pd1—O42	93.05 (10)
O1—C20—N1	117.1 (3)	O41—Pd1—O42	90.70 (11)
O1—C20—C11	116.6 (3)	C16—Pd1—Pd1^{i}	101.08 (11)
N1—C20—C11	126.4 (3)	O1—Pd1—Pd1^{i}	102.78 (8)
C26—C21—C22	120.8 (4)	O41—Pd1—Pd1^{i}	81.02 (8)
C26—C21—N1	119.4 (4)	O42—Pd1—Pd1^{i}	79.52 (7)
C22—C21—N1	119.8 (4)	C20—N1—C21	124.6 (3)
C23—C22—C21	119.0 (5)	C20—N1—C10	118.6 (3)
C23—C22—H22	120.5	C21—N1—C10	116.4 (3)
C21—C22—H22	120.5		
C16—C11—C12—C13	2.0 (6)	N1—C21—C26—C25	-179.7 (4)
C20—C11—C12—C13	-177.9 (4)	C24—C25—C26—C21	0.8 (8)
C11—C12—C13—O3	179.4 (4)	O42^{i}—C40—C41—	133.3 (4)

		F43	
C11—C12—C13—C14	0.2 (6)	O41—C40—C41—F43	-46.8 (6)
O3—C13—C14—C15	178.9 (4)	O42^{i}—C40—C41—F42	9.7 (6)
C12—C13—C14—C15	-1.8 (7)	O41—C40—C41—F42	-170.4 (4)
C13—C14—C15—C16	1.2 (7)	O42^{i}—C40—C41—F41	-107.6 (5)
C14—C15—C16—C11	1.0 (7)	O41—C40—C41—F41	72.3 (5)
C14—C15—C16—Pd1	-178.7 (3)	N1—C20—O1—Pd1	170.3 (3)
C12—C11—C16—C15	-2.6 (6)	C11—C20—O1—Pd1	-10.0 (4)
C20—C11—C16—C15	177.4 (4)	C12—C13—O3—C17	9.4 (6)
C12—C11—C16—Pd1	177.1 (3)	C14—C13—O3—C17	-171.4 (4)
C20—C11—C16—Pd1	-2.9 (4)	O42^{i}—C40—O41—Pd1	11.2 (6)
C16—C11—C20—O1	8.7 (5)	C41—C40—O41—Pd1	-168.6 (3)
C12—C11—C20—O1	-171.4 (4)	O1—C20—N1—C21	-171.3 (4)
C16—C11—C20—N1	-171.7 (4)	C11—C20—N1—C21	9.0 (6)
C12—C11—C20—N1	8.3 (6)	O1—C20—N1—C10	1.5 (6)
C26—C21—C22—C23	-3.3 (6)	C11—C20—N1—C10	-178.2 (4)
N1—C21—C22—C23	178.5 (3)	C26—C21—N1—C20	78.1 (5)
C21—C22—C23—C24	1.8 (7)	C22—C21—N1—C20	-103.7 (5)
C22—C23—C24—C25	1.0 (8)	C26—C21—N1—C10	-94.8 (5)
C23—C24—C25—C26	-2.3 (8)	C22—C21—N1—C10	83.4 (5)
C22—C21—C26—C25	2.1 (6)		

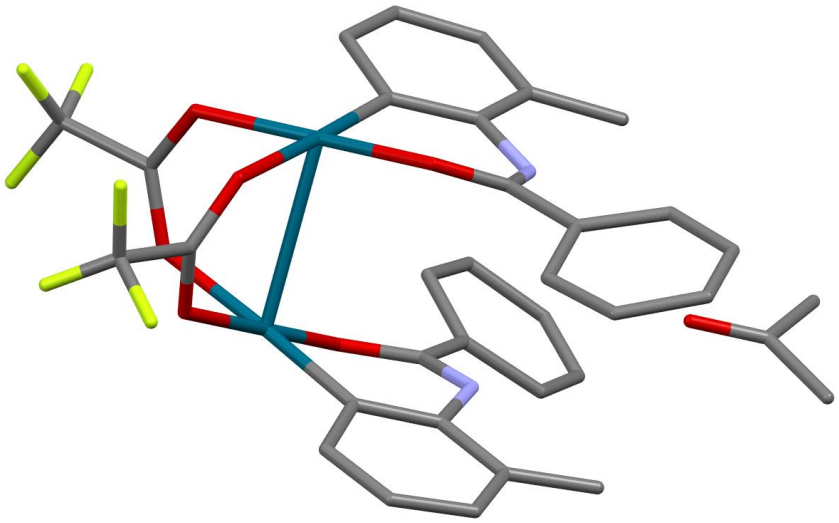
Symmetry code: (i) -*x*+1, *y*, -*z*+1/2.

Hydrogen-bond geometry (Å, º) for (45)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C10—H10*A*···O3^{ii}	0.96	2.63	3.572 (6)	167
C22—H22···O41^{iii}	0.93	2.37	3.212 (5)	150

Symmetry codes: (ii) *x*+1/2, -*y*+1/2, *z*+1/2; (iii) -*x*+1, -*y*+1, -*z*+1.

(9-Pd)



Crystal data

$C_{32}H_{24}F_6N_2O_6Pd_2 \cdot C_3H_6O$	D_x = 1.627 Mg m^{-3}
M_r = 917.41	Mo *K*a radiation, l = 0.71073 Å
Orthorhombic, *Fdd*2	Cell parameters from 314 reflections
a = 37.712 (6) Å	q = 2.1–32.4°
b = 13.024 (2) Å	m = 1.04 mm^{-1}
c = 15.254 (3) Å	*T* = 298 K
V = 7492 (2) Å^3	Needle, colourless
Z = 8	0.30 × 0.09 × 0.03 mm
F(000) = 3648	

Data collection

Bruker D8 VENTURE diffractometer	3350 independent reflections
Radiation source: microfocus sealed tube, INCOATEC ImS 3.0	2121 reflections with *I* > 2s(*I*)
Multilayer mirror INCOATEC monochromator	R_{int} = 0.081
Detector resolution: 7.3910 pixels mm^{-1}	q_{max} = 25.4°, q_{min} = 2.9°
w and p scan	*h* = -41→45
Absorption correction: multi-scan *SADABS2016*/2 - Bruker AXS area detector scaling and absorption correction	*k* = -15→14
T_{min} = 0.74, T_{max} = 0.97	*l* = -18→18
16999 measured reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: mixed
$R[F^2 > 2s(F^2)] = 0.051$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.138$	$w = 1/[s^2(F_o^2) + (0.0627P)^2 + 3.5573P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.10$	$(D/s)_{max} = 0.001$
3350 reflections	$D\rho_{max} = 0.47$ e Å^{-3}
241 parameters	$D\rho_{min} = -0.52$ e Å^{-3}
4 restraints	Absolute structure: Flack x determined using 747 quotients [(I+)-(I-)]/[(I+)+(I-)] (Parsons, Flack and Wagner, Acta Cryst. B69 (2013) 249-259).
Primary atom site location: structure-invariant direct methods	Absolute structure parameter: 0.00 (3)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2) for (9-Pd)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.5415 (3)	0.4031 (10)	0.3332 (9)	0.059 (3)
C11	0.4816 (3)	0.3256 (10)	0.3272 (11)	0.072 (4)
C12	0.4576 (4)	0.2803 (13)	0.3849 (13)	0.096 (5)
C13	0.4293 (5)	0.2263 (15)	0.3464 (17)	0.108 (6)
H13	0.412819	0.194824	0.382783	0.13*
C14	0.4252 (5)	0.2182 (17)	0.260 (2)	0.138 (8)
H14	0.405814	0.182654	0.237773	0.166*
C15	0.4494 (5)	0.2619 (14)	0.2023 (15)	0.112 (6)
H15	0.447145	0.252232	0.142117	0.134*
C16	0.4787 (3)	0.3238 (10)	0.2381 (10)	0.068 (4)
C17	0.4603 (5)	0.2876 (15)	0.4786 (13)	0.108 (6)
H17A	0.450211	0.227379	0.504894	0.163*
H17B	0.447807	0.347313	0.498555	0.163*
H17C	0.484856	0.292885	0.495028	0.163*

C21	0.5710 (3)	0.4366 (10)	0.3886 (10)	0.066 (4)
C22	0.5745 (5)	0.4006 (14)	0.4746 (11)	0.104 (6)
H22	0.557775	0.355723	0.497599	0.124*
C23	0.6028 (7)	0.432 (2)	0.5249 (16)	0.147 (9)
H23	0.604956	0.407179	0.581996	0.176*
C24	0.6262 (7)	0.4932 (19)	0.4947 (16)	0.134 (8)
H24	0.645043	0.512045	0.530533	0.161*
C25	0.6244 (4)	0.5335 (13)	0.410 (2)	0.115 (6)
H25	0.641789	0.578158	0.389655	0.138*
C26	0.5958 (4)	0.5054 (14)	0.3555 (12)	0.092 (5)
H26	0.593577	0.532143	0.299241	0.11*
C40	0.4553 (4)	0.4514 (17)	0.0351 (10)	0.081 (4)
C41	0.4318 (6)	0.419 (2)	-0.0449 (15)	0.124 (8)
C50	0.5	0.5	0.638 (2)	0.167 (17)
C51	0.5267 (12)	0.556 (3)	0.691 (2)	0.32 (3)
H51A	0.548223	0.516977	0.693379	0.475*
H51B	0.517753	0.566723	0.748907	0.475*
H51C	0.531461	0.621588	0.663941	0.475*
H1	0.511 (8)	0.37 (3)	0.421 (5)	0.475*
N1	0.5109 (3)	0.3771 (8)	0.3688 (8)	0.066 (3)
O10	0.5471 (2)	0.3985 (7)	0.2521 (7)	0.067 (3)
O41	0.4762 (3)	0.3835 (9)	0.0555 (7)	0.088 (3)
O42	0.4517 (2)	0.5393 (10)	0.0667 (6)	0.084 (3)
O50	0.5	0.5	0.5600 (16)	0.149 (7)
F41	0.4473 (5)	0.375 (2)	-0.1013 (13)	0.257 (13)
F42	0.4077 (4)	0.3521 (11)	-0.0163 (12)	0.181 (6)
F43	0.4125 (4)	0.4941 (10)	-0.0694 (11)	0.192 (8)
Pd1	0.51097 (3)	0.39084 (8)	0.15793 (9)	0.0717 (4)

Atomic displacement parameters (Å^2) for (9-Pd)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.054 (8)	0.057 (8)	0.066 (9)	0.014 (6)	-0.004 (7)	0.005 (6)
C11	0.063 (9)	0.053 (8)	0.101 (11)	0.004 (6)	0.001 (8)	0.013 (7)
C12	0.079 (10)	0.080 (11)	0.129 (17)	-0.005 (8)	-0.007 (11)	0.030 (10)
C13	0.076 (12)	0.099 (15)	0.151 (19)	-0.019 (10)	-0.011 (12)	0.016 (12)
C14	0.083 (13)	0.130 (17)	0.20 (3)	-0.056 (12)	-0.041 (16)	0.003 (18)
C15	0.080 (11)	0.124 (16)	0.132 (15)	-0.010 (11)	-0.042 (10)	-0.015 (12)
C16	0.056 (8)	0.064 (9)	0.085 (10)	-0.001 (6)	-0.017 (7)	0.005 (7)
C17	0.094 (12)	0.103 (14)	0.128 (16)	-0.010 (10)	0.002 (11)	0.049 (12)
C21	0.056 (7)	0.057 (8)	0.085 (12)	0.005 (6)	-0.003 (8)	0.004 (7)

C22	0.108 (13)	0.112 (14)	0.091 (12)	-0.020 (11)	-0.042 (11)	0.019 (10)
C23	0.142 (19)	0.17 (2)	0.130 (19)	-0.045 (18)	-0.052 (16)	0.016 (16)
C24	0.136 (19)	0.15 (2)	0.121 (17)	-0.024 (15)	-0.055 (14)	-0.006 (17)
C25	0.082 (10)	0.089 (12)	0.176 (19)	-0.021 (8)	0.008 (16)	-0.028 (16)
C26	0.051 (8)	0.113 (15)	0.111 (12)	-0.010 (9)	0.001 (8)	-0.016 (9)
C40	0.063 (9)	0.114 (14)	0.067 (10)	0.000 (9)	-0.009 (8)	0.002 (10)
C41	0.087 (14)	0.18 (3)	0.100 (15)	-0.009 (16)	-0.006 (12)	0.032 (16)
C50	0.27 (6)	0.16 (4)	0.07 (3)	0.06 (3)	0	0
C51	0.60 (10)	0.18 (4)	0.17 (4)	0.05 (5)	-0.12 (4)	-0.02 (2)
N1	0.043 (6)	0.073 (8)	0.081 (7)	-0.002 (5)	-0.003 (5)	0.001 (5)
O10	0.043 (5)	0.083 (7)	0.074 (6)	0.009 (4)	-0.011 (4)	-0.007 (5)
O41	0.082 (7)	0.105 (10)	0.078 (7)	0.014 (6)	-0.016 (6)	-0.022 (6)
O42	0.067 (6)	0.111 (10)	0.075 (7)	0.020 (6)	0.003 (5)	0.007 (6)
O50	0.21 (2)	0.110 (16)	0.13 (2)	-0.004 (15)	0	0
F41	0.163 (14)	0.47 (4)	0.133 (12)	0.067 (18)	-0.050 (13)	-0.129 (19)
F42	0.147 (12)	0.162 (12)	0.233 (16)	-0.019 (9)	-0.090 (12)	0.026 (11)
F43	0.217 (13)	0.127 (11)	0.232 (19)	-0.035 (10)	-0.144 (14)	0.070 (10)
Pd1	0.0643 (6)	0.0737 (6)	0.0772 (6)	0.0074 (6)	-0.0111 (5)	-0.0061 (6)

Geometric parameters (Å, º) for (9-Pd)

C1—O10	1.257 (16)	C23—H23	0.93
C1—N1	1.321 (16)	C24—C25	1.40 (3)
C1—C21	1.463 (18)	C24—H24	0.93
C11—C16	1.36 (2)	C25—C26	1.41 (2)
C11—C12	1.39 (2)	C25—H25	0.93
C11—N1	1.438 (18)	C26—H26	0.93
C12—C13	1.41 (3)	C40—O41	1.22 (2)
C12—C17	1.44 (3)	C40—O42	1.25 (2)
C13—C14	1.33 (3)	C40—C41	1.57 (3)
C13—H13	0.93	C41—F41	1.19 (3)
C14—C15	1.39 (3)	C41—F43	1.28 (3)
C14—H14	0.93	C41—F42	1.33 (3)
C15—C16	1.47 (2)	C50—O50	1.19 (3)
C15—H15	0.93	C50—C51^{i}	1.48 (2)
C16—Pd1	1.933 (14)	C50—C51	1.48 (2)
C17—H17A	0.96	C51—H51A	0.96
C17—H17B	0.96	C51—H51B	0.96
C17—H17C	0.96	C51—H51C	0.96
C21—C26	1.39 (2)	N1—H1	0.80 (3)
C21—C22	1.40 (2)	O10—Pd1	1.981 (9)

C22—C23	1.37 (3)	O41—Pd1	2.042 (11)
C22—H22	0.93	O42—Pd1^{i}	2.178 (11)
C23—C24	1.28 (3)	Pd1—Pd1^{i}	2.961 (2)
O10—C1—N1	122.5 (12)	C24—C25—H25	120.5
O10—C1—C21	117.1 (12)	C26—C25—H25	120.5
N1—C1—C21	120.3 (12)	C21—C26—C25	117.9 (18)
C16—C11—C12	124.8 (14)	C21—C26—H26	121.0
C16—C11—N1	120.7 (12)	C25—C26—H26	121.0
C12—C11—N1	114.6 (14)	O41—C40—O42	129.4 (14)
C11—C12—C13	116.1 (17)	O41—C40—C41	111.6 (19)
C11—C12—C17	123.7 (17)	O42—C40—C41	119.0 (18)
C13—C12—C17	120.2 (17)	F41—C41—F43	116 (2)
C14—C13—C12	122.8 (19)	F41—C41—F42	105 (3)
C14—C13—H13	118.6	F43—C41—F42	101.9 (18)
C12—C13—H13	118.6	F41—C41—C40	114.6 (19)
C13—C14—C15	121.2 (18)	F43—C41—C40	110 (2)
C13—C14—H14	119.4	F42—C41—C40	107.9 (17)
C15—C14—H14	119.4	O50—C50—C51^{i}	122.9 (19)
C14—C15—C16	118.8 (19)	O50—C50—C51	122.9 (19)
C14—C15—H15	120.6	C51^{i}—C50—C51	114 (4)
C16—C15—H15	120.6	C50—C51—H51A	109.5
C11—C16—C15	116.1 (15)	C50—C51—H51B	109.5
C11—C16—Pd1	124.9 (10)	H51A—C51—H51B	109.5
C15—C16—Pd1	119.0 (13)	C50—C51—H51C	109.5
C12—C17—H17A	109.5	H51A—C51—H51C	109.5
C12—C17—H17B	109.5	H51B—C51—H51C	109.5
H17A—C17—H17B	109.5	C1—N1—C11	127.6 (12)
C12—C17—H17C	109.5	C1—N1—H1	115 (10)
H17A—C17—H17C	109.5	C11—N1—H1	113 (4)
H17B—C17—H17C	109.5	C1—O10—Pd1	127.0 (8)
C26—C21—C22	119.5 (15)	C40—O41—Pd1	125.0 (11)
C26—C21—C1	119.6 (13)	C40—O42—Pd1^{i}	124.0 (9)
C22—C21—C1	120.9 (14)	C16—Pd1—O10	89.8 (5)
C23—C22—C21	120.0 (19)	C16—Pd1—O41	93.4 (5)
C23—C22—H22	120.0	O10—Pd1—O41	176.5 (5)
C21—C22—H22	120.0	C16—Pd1—O42^{i}	177.8 (5)
C24—C23—C22	121 (2)	O10—Pd1—O42^{i}	89.9 (4)
C24—C23—H23	119.4	O41—Pd1—O42^{i}	86.8 (4)
C22—C23—H23	119.4	C16—Pd1—Pd1^{i}	105.0 (4)

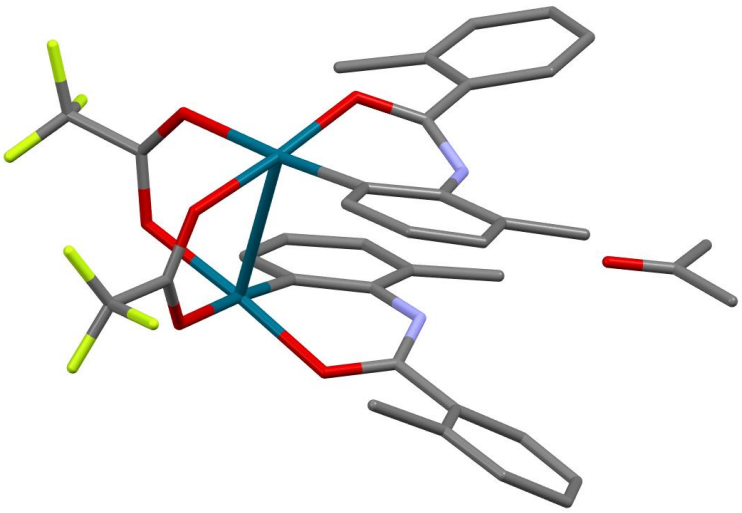
C23—C24—C25	122 (2)	O10—Pd1—Pd1^{i}	98.2 (3)
C23—C24—H24	118.8	O41—Pd1—Pd1^{i}	82.3 (3)
C25—C24—H24	118.8	O42^{i}—Pd1—Pd1^{i}	77.2 (3)
C24—C25—C26	119.0 (18)		

Symmetry code: (i) -*x*+1, -*y*+1, *z*.

Hydrogen-bond geometry (Å, º) for (9-Pd)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O50	0.80 (3)	2.8 (4)	3.35 (2)	130 (40)

(11-Pd)



Crystal data

$C_{34}H_{28}F_6N_2O_6Pd_2 \cdot C_3H_6O$	D_x = 1.650 Mg m^{-3}
M_r = 945.46	Mo *K*a radiation, l = 0.71073 Å
Orthorhombic, *Fdd*2	Cell parameters from 125 reflections
a = 37.337 (11) Å	q = 2.6–14.3°
b = 13.419 (4) Å	m = 1.02 mm^{-1}

c = 15.197 (5) Å	T = 299 K
V = 7614 (4) Å^3	Needle, yellow
Z = 8	0.67 × 0.08 × 0.05 mm
F(000) = 3776	

Data collection

Bruker D8 VENTURE diffractometer	3648 independent reflections
Radiation source: microfocus sealed tube, INCOATEC ImS 3.0	2153 reflections with $I > 2s(I)$
Multilayer mirror INCOATEC monochromator	R_{int} = 0.230
Detector resolution: 7.3910 pixels mm^{-1}	q_{max} = 25.8°, q_{min} = 2.6°
w and p scan	h = -45→45
Absorption correction: multi-scan *SADABS2016*/2 - Bruker AXS area detector scaling and absorption correction	k = -16→16
T_{min} = 0.65, T_{max} = 0.95	l = -18→18
49149 measured reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: mixed
$R[F^2 > 2s(F^2)]$ = 0.056	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.173	$w = 1/[s^2(F_o^2) + (0.0708P)^2 + 58.4521P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.02	$(D/s)_{max}$ = 0.001
3648 reflections	$D\rho_{max}$ = 0.87 e Å^{-3}
251 parameters	$D\rho_{min}$ = -0.67 e Å^{-3}
2 restraints	Absolute structure: Flack x determined using 778 quotients [(I+)-(I-)]/[(I+)+(I-)] (Parsons, Flack and Wagner, Acta Cryst. B69 (2013) 249-259).
Primary atom site location: structure-invariant direct methods	Absolute structure parameter: -0.02 (4)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2) for (11-Pd)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.7044 (5)	0.7978 (16)	0.2459 (12)	0.070 (5)
C10	0.6802 (8)	0.831 (2)	0.1720 (19)	0.109 (8)
C11	0.7346 (5)	0.9254 (13)	0.5356 (11)	0.065 (4)
C12	0.7110 (5)	0.9747 (14)	0.5919 (12)	0.070 (5)
C13	0.6849 (6)	1.0349 (17)	0.5549 (17)	0.088 (6)
H13	0.668289	1.06599	0.591366	0.105*
C14	0.6829 (6)	1.0494 (16)	0.467 (2)	0.097 (7)
H14	0.665654	1.092575	0.444802	0.117*
C15	0.7064 (5)	1.0007 (13)	0.4084 (15)	0.084 (6)
H15	0.705102	1.011621	0.348072	0.101*
C16	0.7324 (4)	0.9335 (13)	0.4454 (13)	0.064 (5)
C17	0.7127 (6)	0.9603 (18)	0.6896 (15)	0.095 (7)
H17A	0.698313	1.009975	0.718085	0.142*
H17B	0.737105	0.966515	0.708852	0.142*
H17C	0.703889	0.895237	0.70432	0.142*
C20	0.7937 (4)	0.8459 (12)	0.5406 (10)	0.054 (4)
C21	0.8228 (4)	0.8147 (12)	0.5986 (15)	0.066 (5)
C22	0.8503 (5)	0.7515 (14)	0.5690 (13)	0.071 (5)
C23	0.8769 (6)	0.7280 (14)	0.6279 (17)	0.091 (6)
H23	0.894658	0.683297	0.611085	0.109*
C24	0.8781 (8)	0.768 (2)	0.7104 (19)	0.141 (12)
H24	0.897159	0.75406	0.747742	0.169*
C25	0.8517 (8)	0.829 (2)	0.7371 (17)	0.133 (10)
H25	0.852081	0.852907	0.794459	0.159*
C26	0.8247 (6)	0.8554 (15)	0.6838 (13)	0.083 (6)
H26	0.807351	0.900327	0.702886	0.1*
C27	0.8516 (5)	0.7049 (14)	0.4777 (15)	0.086 (6)
H27A	0.86761	0.648973	0.478254	0.129*
H27B	0.859929	0.753416	0.436059	0.129*
H27C	0.82804	0.683019	0.461338	0.129*
C50	0.75	0.75	0.843 (3)	0.21 (4)
C51	0.7780 (11)	0.694 (2)	0.893 (2)	0.20 (2)
H51A	0.781883	0.725767	0.948561	0.302*
H51B	0.799955	0.694166	0.85973	0.302*
H51C	0.770225	0.626817	0.901795	0.302*
N1	0.7623 (5)	0.8679 (13)	0.5753 (9)	0.056 (3)

O1	0.8002 (3)	0.8554 (8)	0.4592 (8)	0.061 (3)
O1A	0.7266 (4)	0.8663 (10)	0.2648 (9)	0.080 (4)
O3	0.7993 (3)	0.7834 (11)	0.2772 (9)	0.081 (4)
O50	0.75	0.75	0.766 (2)	0.138 (9)
F11	0.6555 (6)	0.8975 (14)	0.2039 (16)	0.176 (8)
F12	0.6938 (5)	0.883 (2)	0.1147 (16)	0.200 (10)
F13	0.6601 (5)	0.7630 (12)	0.1413 (13)	0.169 (9)
Pd1	0.76276 (4)	0.85904 (10)	0.36573 (13)	0.0638 (4)
H1	0.762 (6)	0.867 (17)	0.632 (2)	0.096*

Atomic displacement parameters (Å^2) for (11-Pd)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.076 (13)	0.083 (13)	0.050 (10)	-0.012 (11)	-0.010 (9)	-0.010 (10)
C10	0.11 (2)	0.13 (2)	0.083 (18)	-0.01 (2)	-0.019 (16)	0.038 (16)
C11	0.064 (12)	0.072 (11)	0.057 (10)	-0.005 (9)	0.000 (9)	-0.005 (9)
C12	0.061 (11)	0.082 (12)	0.069 (13)	0.003 (10)	-0.001 (9)	-0.024 (9)
C13	0.063 (14)	0.106 (17)	0.094 (17)	0.016 (12)	-0.008 (12)	-0.017 (13)
C14	0.069 (14)	0.084 (14)	0.14 (2)	0.018 (11)	0.001 (15)	0.012 (15)
C15	0.075 (13)	0.076 (13)	0.100 (16)	-0.006 (11)	-0.015 (12)	0.004 (11)
C16	0.042 (9)	0.067 (10)	0.082 (13)	-0.005 (8)	-0.003 (9)	0.007 (9)
C17	0.093 (16)	0.106 (16)	0.085 (16)	0.014 (13)	-0.004 (13)	-0.022 (13)
C20	0.049 (10)	0.070 (10)	0.043 (9)	-0.011 (8)	0.003 (8)	-0.005 (8)
C21	0.049 (9)	0.057 (9)	0.092 (17)	-0.003 (8)	0.011 (10)	-0.004 (9)
C22	0.056 (11)	0.071 (12)	0.085 (13)	-0.005 (9)	-0.004 (10)	0.001 (10)
C23	0.091 (14)	0.085 (12)	0.097 (16)	0.026 (11)	-0.012 (14)	0.001 (13)
C24	0.14 (2)	0.17 (3)	0.11 (2)	0.08 (2)	-0.085 (19)	-0.04 (2)
C25	0.13 (2)	0.17 (3)	0.094 (19)	0.04 (2)	-0.048 (17)	-0.035 (18)
C26	0.093 (15)	0.089 (13)	0.069 (13)	0.017 (12)	-0.026 (12)	-0.009 (11)
C27	0.079 (14)	0.074 (12)	0.105 (16)	0.007 (10)	0.013 (13)	-0.017 (12)
C50	0.48 (12)	0.07 (2)	0.08 (4)	0.04 (4)	0	0
C51	0.34 (6)	0.12 (3)	0.14 (4)	-0.09 (3)	-0.07 (3)	0.01 (2)
N1	0.046 (8)	0.071 (9)	0.052 (7)	0.003 (7)	-0.003 (7)	-0.002 (7)
O1	0.047 (7)	0.074 (7)	0.062 (8)	-0.008 (5)	0.002 (6)	-0.002 (6)
O1A	0.078 (10)	0.098 (11)	0.063 (9)	-0.015 (8)	-0.008 (7)	0.009 (7)
O3	0.067 (9)	0.103 (11)	0.072 (9)	-0.014 (8)	-0.003 (7)	-0.015 (8)
O50	0.18 (3)	0.13 (2)	0.10 (2)	0.009 (19)	0	0
F11	0.147 (15)	0.170 (15)	0.21 (2)	0.048 (14)	-0.085 (16)	-0.023 (16)
F12	0.148 (15)	0.33 (3)	0.117 (12)	-0.038 (18)	-0.050 (16)	0.101 (18)
F13	0.212 (17)	0.116 (11)	0.18 (2)	-0.004 (12)	-0.133 (17)	-0.008 (11)
Pd1	0.0586 (7)	0.0803 (7)	0.0524 (6)	-0.0068 (8)	-0.0016 (7)	0.0004 (8)

Geometric parameters (Å, º) for (11-Pd)

C1—O3^{i}	1.20 (2)	C21—C26	1.41 (3)
C1—O1A	1.27 (2)	C22—C23	1.37 (3)
C1—C10	1.51 (3)	C22—C27	1.52 (3)
C10—F12	1.23 (3)	C23—C24	1.37 (3)
C10—F13	1.27 (3)	C23—H23	0.93
C10—F11	1.37 (3)	C24—C25	1.34 (3)
C11—C16	1.38 (3)	C24—H24	0.93
C11—C12	1.40 (2)	C25—C26	1.34 (3)
C11—N1	1.42 (2)	C25—H25	0.93
C12—C13	1.38 (3)	C26—H26	0.93
C12—C17	1.50 (3)	C27—H27A	0.96
C13—C14	1.35 (3)	C27—H27B	0.96
C13—H13	0.93	C27—H27C	0.96
C14—C15	1.41 (3)	C50—O50	1.17 (4)
C14—H14	0.93	C50—C51^{i}	1.49 (4)
C15—C16	1.44 (2)	C50—C51	1.49 (4)
C15—H15	0.93	C51—H51A	0.96
C16—Pd1	1.936 (18)	C51—H51B	0.96
C17—H17A	0.96	C51—H51C	0.96
C17—H17B	0.96	N1—H1	0.87 (3)
C17—H17C	0.96	O1—Pd1	1.993 (12)
C20—O1	1.27 (2)	O1A—Pd1	2.045 (14)
C20—N1	1.32 (2)	O3—Pd1	2.168 (14)
C20—C21	1.46 (3)	Pd1—Pd1^{i}	3.078 (3)
C21—C22	1.40 (2)		
O3^{i}—C1—O1A	130.1 (18)	C24—C23—H23	119.0
O3^{i}—C1—C10	120 (2)	C22—C23—H23	119.0
O1A—C1—C10	110 (2)	C25—C24—C23	120 (2)
F12—C10—F13	113 (3)	C25—C24—H24	120.2
F12—C10—F11	99 (3)	C23—C24—H24	120.2
F13—C10—F11	102 (2)	C24—C25—C26	122 (2)
F12—C10—C1	117 (2)	C24—C25—H25	118.9
F13—C10—C1	115 (2)	C26—C25—H25	118.9
F11—C10—C1	109 (2)	C25—C26—C21	119 (2)
C16—C11—C12	122.3 (18)	C25—C26—H26	120.4
C16—C11—N1	120.6 (16)	C21—C26—H26	120.4
C12—C11—N1	117.1 (15)	C22—C27—H27A	109.5

C13—C12—C11	118.2 (18)	C22—C27—H27B	109.5
C13—C12—C17	120.6 (19)	H27A—C27—H27B	109.5
C11—C12—C17	121.2 (19)	C22—C27—H27C	109.5
C14—C13—C12	122 (2)	H27A—C27—H27C	109.5
C14—C13—H13	119.2	H27B—C27—H27C	109.5
C12—C13—H13	119.2	O50—C50—C51^{i}	121 (2)
C13—C14—C15	122 (2)	O50—C50—C51	121 (2)
C13—C14—H14	119.2	C51^{i}—C50—C51	119 (4)
C15—C14—H14	119.2	C50—C51—H51A	109.5
C14—C15—C16	118 (2)	C50—C51—H51B	109.5
C14—C15—H15	121.2	H51A—C51—H51B	109.5
C16—C15—H15	121.2	C50—C51—H51C	109.5
C11—C16—C15	118.6 (18)	H51A—C51—H51C	109.5
C11—C16—Pd1	123.1 (14)	H51B—C51—H51C	109.5
C15—C16—Pd1	118.3 (15)	C20—N1—C11	126.6 (15)
C12—C17—H17A	109.5	C20—N1—H1	114 (10)
C12—C17—H17B	109.5	C11—N1—H1	115 (10)
H17A—C17—H17B	109.5	C20—O1—Pd1	124.4 (11)
C12—C17—H17C	109.5	C1—O1A—Pd1	124.4 (12)
H17A—C17—H17C	109.5	C1^{i}—O3—Pd1	126.9 (13)
H17B—C17—H17C	109.5	C16—Pd1—O1	88.7 (6)
O1—C20—N1	122.4 (15)	C16—Pd1—O1A	93.3 (7)
O1—C20—C21	118.5 (15)	O1—Pd1—O1A	176.6 (6)
N1—C20—C21	119.0 (15)	C16—Pd1—O3	176.3 (6)
C22—C21—C26	119.6 (19)	O1—Pd1—O3	89.4 (5)
C22—C21—C20	121.6 (18)	O1A—Pd1—O3	88.4 (5)
C26—C21—C20	118.6 (16)	C16—Pd1—Pd1^{i}	108.0 (5)
C23—C22—C21	117.2 (19)	O1—Pd1—Pd1^{i}	101.2 (3)
C23—C22—C27	118.5 (18)	O1A—Pd1—Pd1^{i}	80.9 (4)
C21—C22—C27	124.2 (18)	O3—Pd1—Pd1^{i}	75.5 (4)
C24—C23—C22	122 (2)		
O3^{i}—C1—C10—F12	-144 (3)	O1—C20—C21—C26	143.7 (18)
O1A—C1—C10—F12	36 (4)	N1—C20—C21—C26	-33 (3)
O3^{i}—C1—C10—F13	-8 (4)	C26—C21—C22—C23	4 (3)
O1A—C1—C10—F13	172 (2)	C20—C21—C22—C23	178.6 (17)
O3^{i}—C1—C10—F11	105 (3)	C26—C21—C22—C27	-178.7 (19)
O1A—C1—C10—F11	-75 (3)	C20—C21—C22—	-4 (3)

		C27	
C16—C11—C12—C13	-1 (3)	C21—C22—C23—C24	-4 (3)
N1—C11—C12—C13	177.0 (17)	C27—C22—C23—C24	179 (3)
C16—C11—C12—C17	176.7 (19)	C22—C23—C24—C25	4 (5)
N1—C11—C12—C17	-5 (3)	C23—C24—C25—C26	-3 (5)
C11—C12—C13—C14	-3 (3)	C24—C25—C26—C21	4 (4)
C17—C12—C13—C14	180 (2)	C22—C21—C26—C25	-4 (3)
C12—C13—C14—C15	3 (4)	C20—C21—C26—C25	-179 (2)
C13—C14—C15—C16	1 (3)	O1—C20—N1—C11	-16 (3)
C12—C11—C16—C15	4 (3)	C21—C20—N1—C11	160.6 (17)
N1—C11—C16—C15	-173.6 (16)	C16—C11—N1—C20	24 (3)
C12—C11—C16—Pd1	-175.6 (14)	C12—C11—N1—C20	-153.8 (18)
N1—C11—C16—Pd1	6 (2)	N1—C20—O1—Pd1	-22 (2)
C14—C15—C16—C11	-4 (2)	C21—C20—O1—Pd1	161.8 (11)
C14—C15—C16—Pd1	175.9 (14)	O3^{i}—C1—O1A—Pd1	-4 (3)
O1—C20—C21—C22	-31 (2)	C10—C1—O1A—Pd1	176.0 (16)
N1—C20—C21—C22	152.0 (18)		

Symmetry code: (i) -*x*+3/2, -*y*+3/2, *z*.

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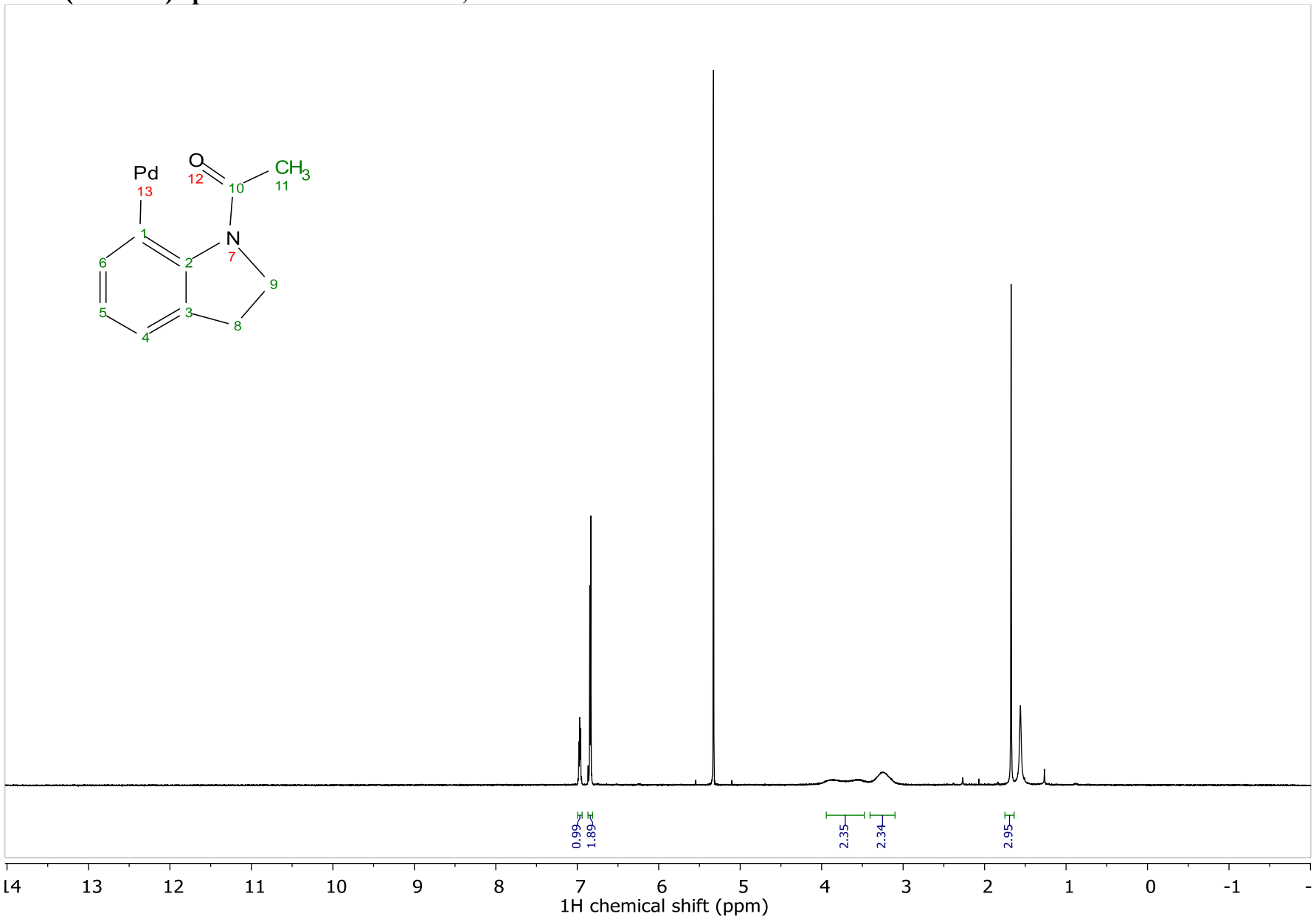
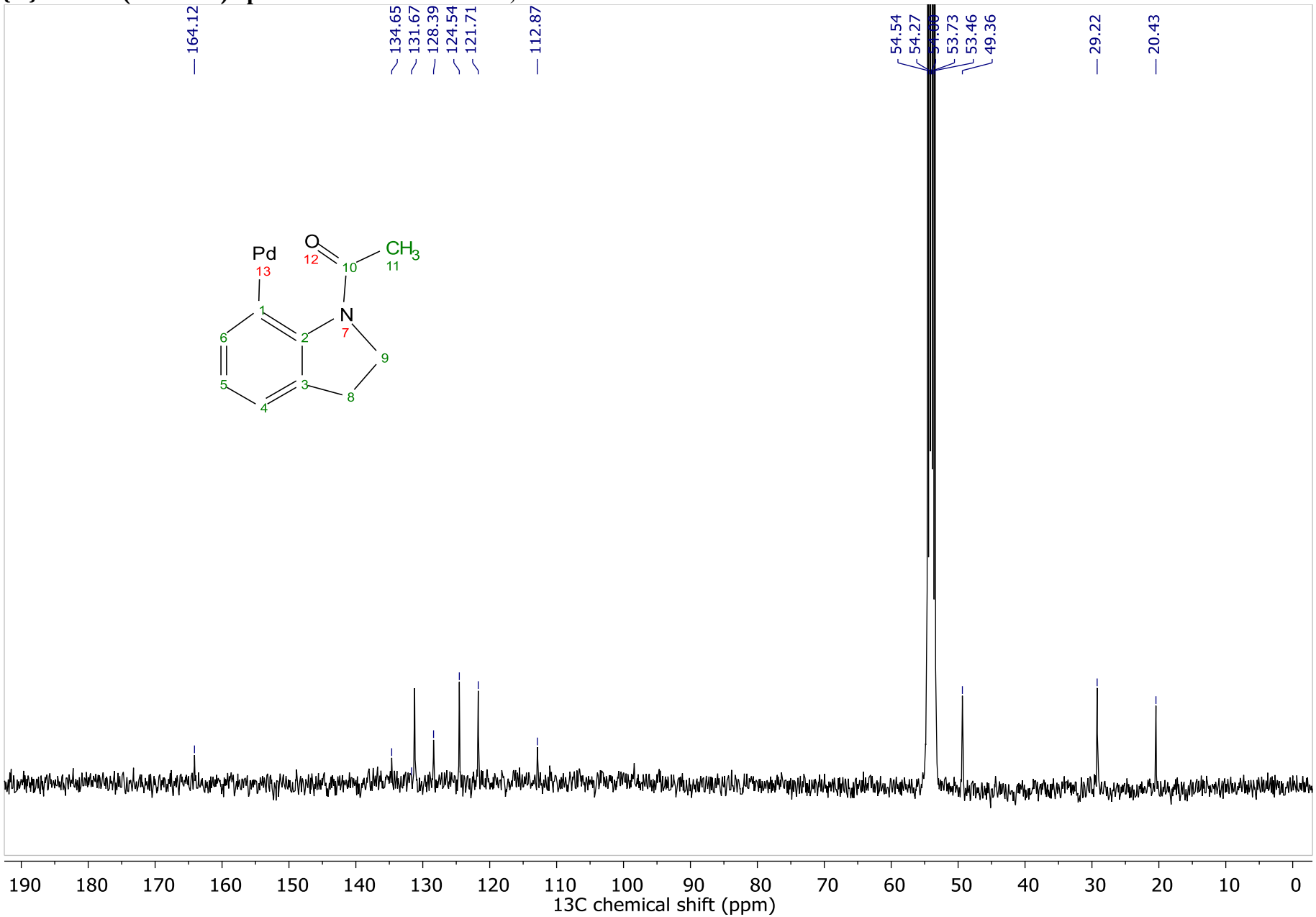
NMR Spectra

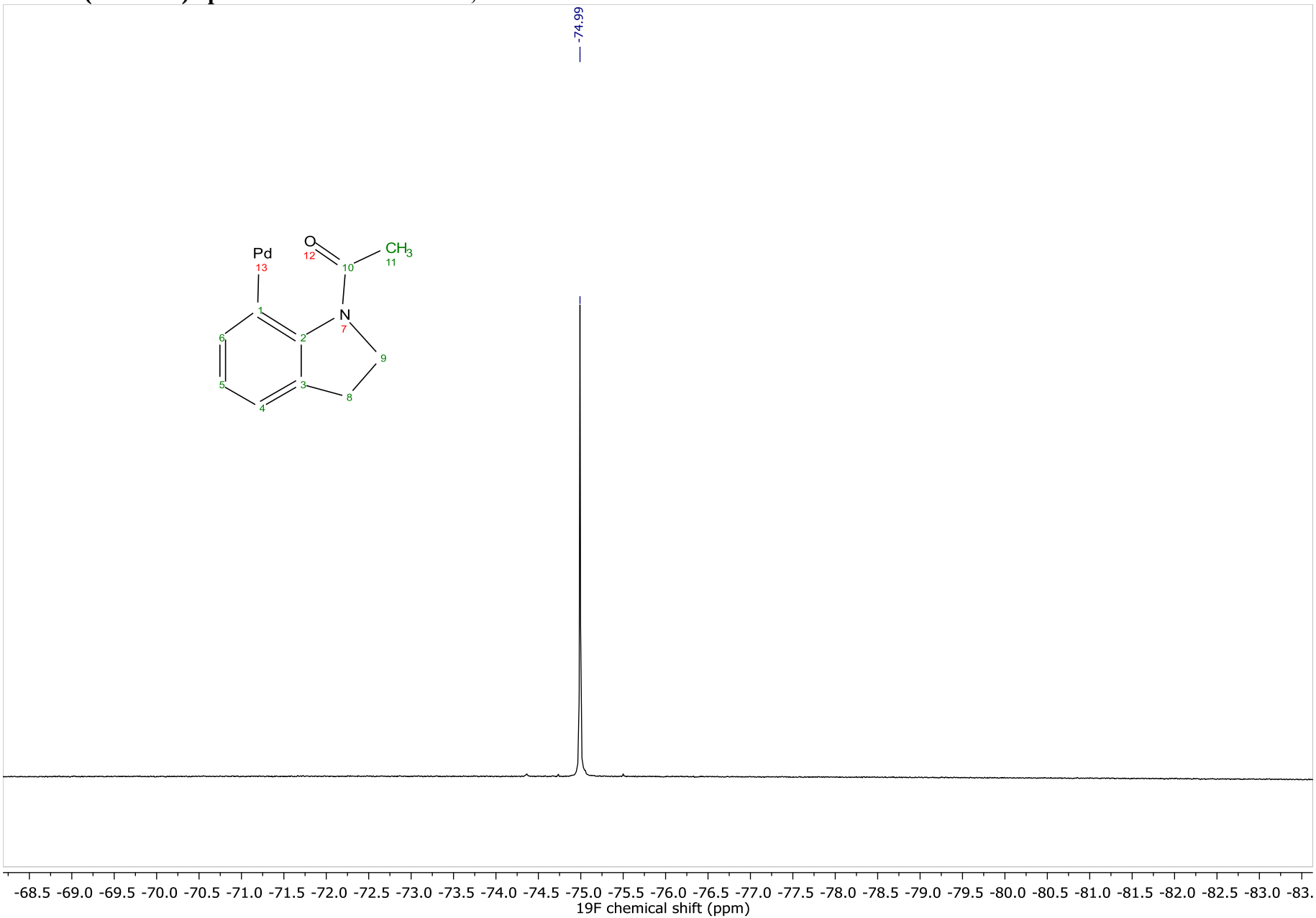
^{1}H-NMR (400 MHz) Spectrum of 2-Pd in CD_2Cl_2, 25 °C



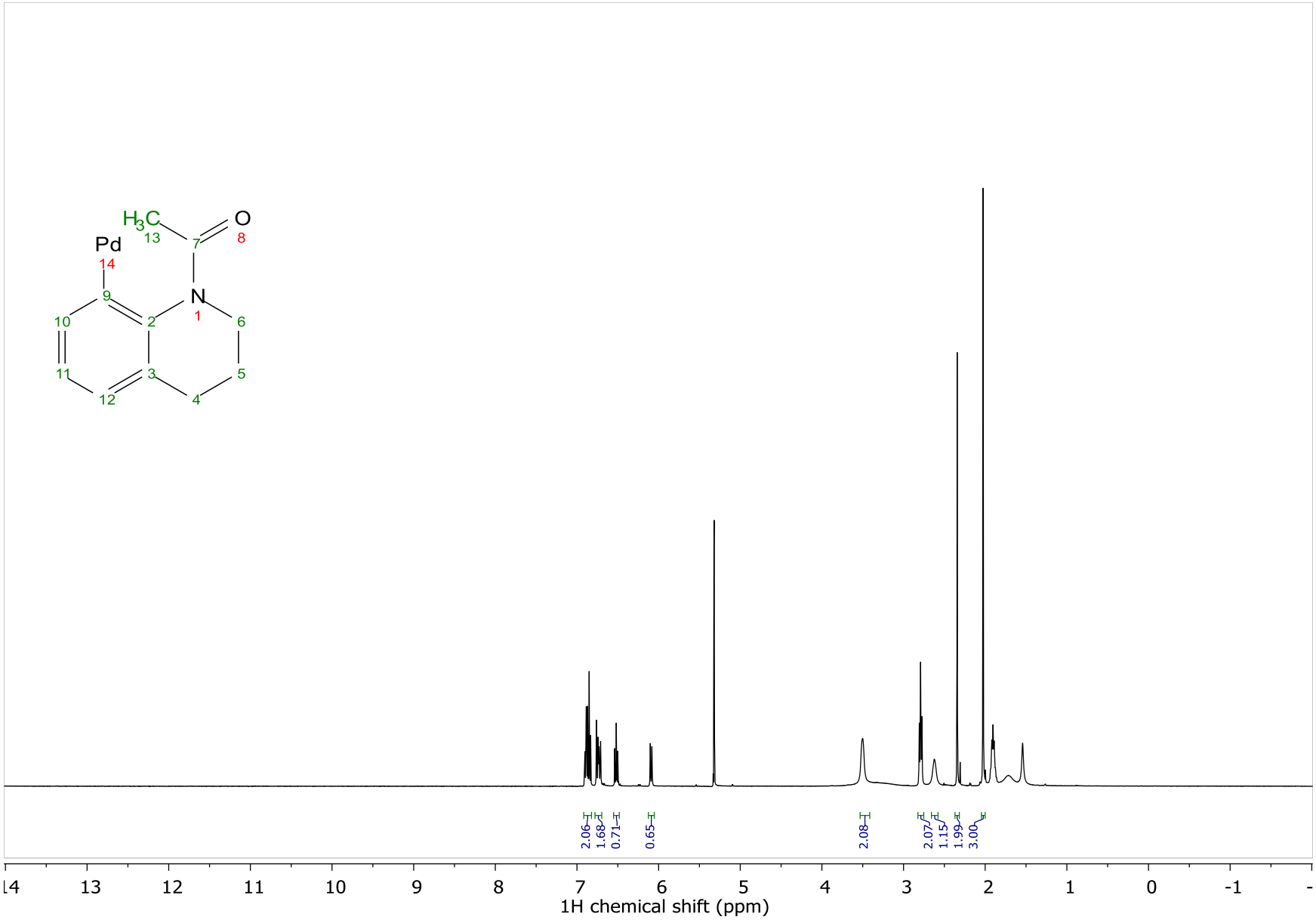
^{13}\{H\}C-NMR (100 MHz) Spectrum of 2-Pd in CD_2Cl_2, 25 °C



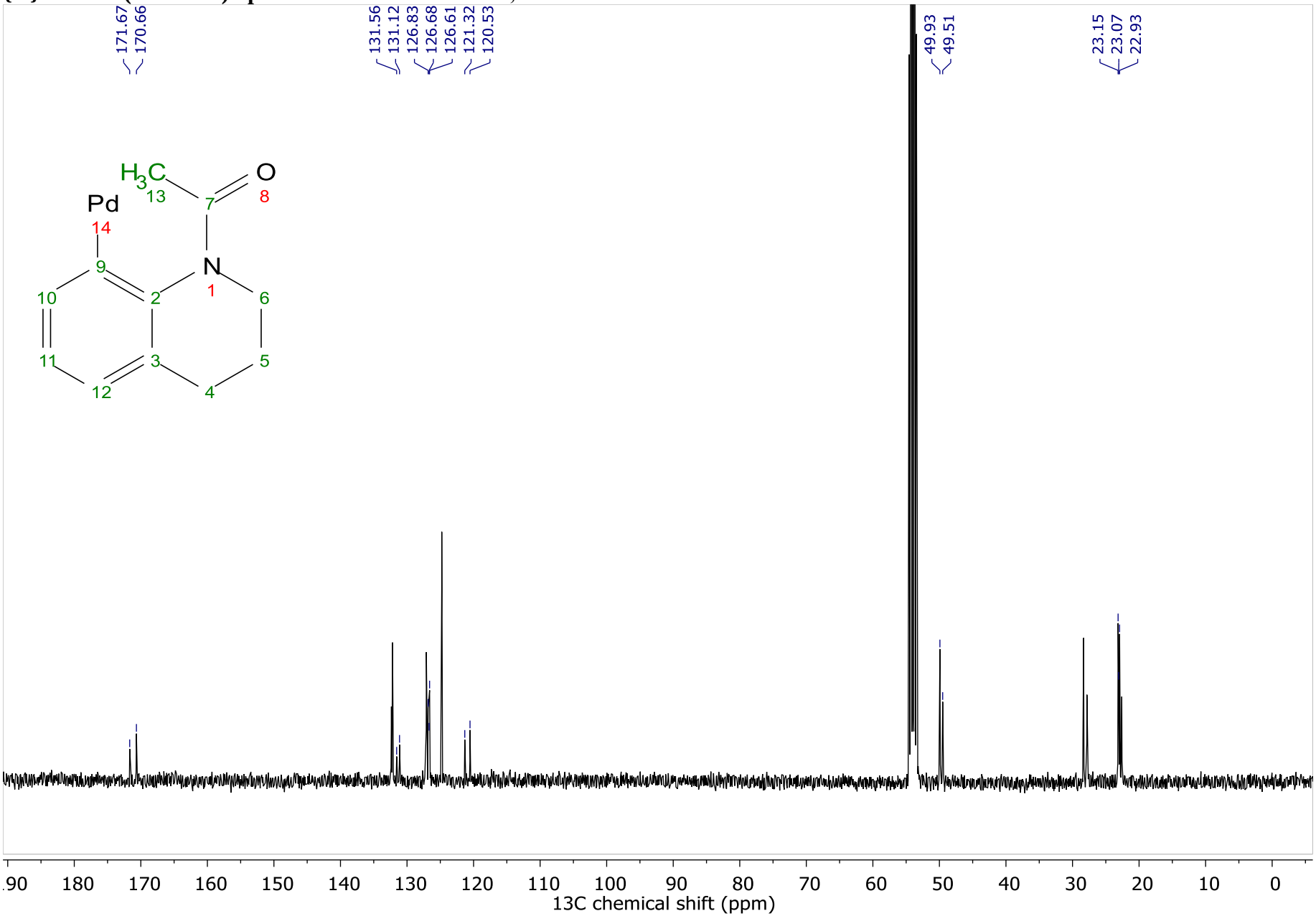
^{19}F-NMR (376 MHz) Spectrum of 2-Pd in CD_2Cl_2, 25 °C



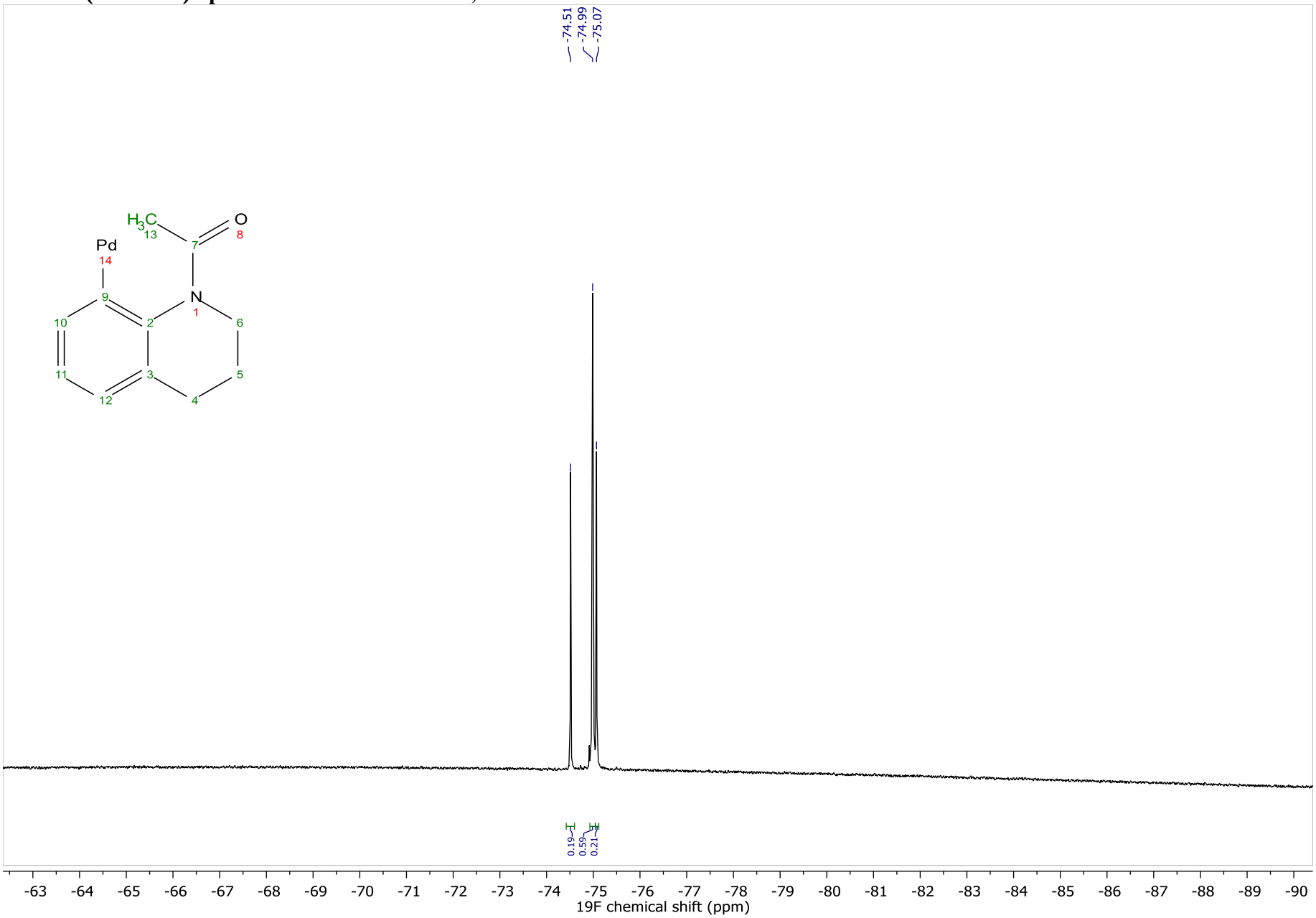
^{1}H-NMR (400 MHz) Spectrum of 4-Pd in CD_2Cl_2, 25 °C



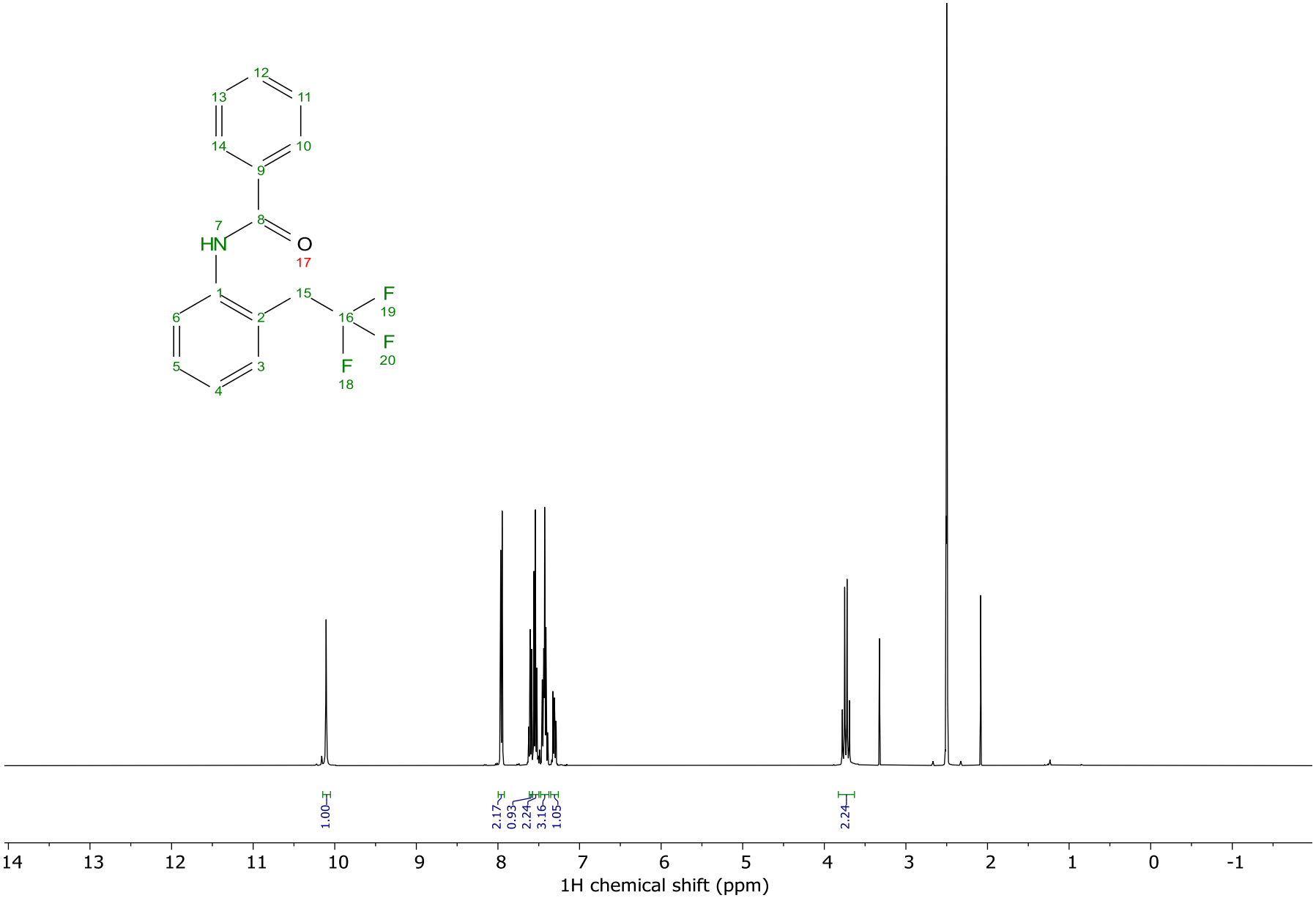
^{13}\{H\}C-NMR (100 MHz) Spectrum of 4-Pd in CD_2Cl_2, 25 °C



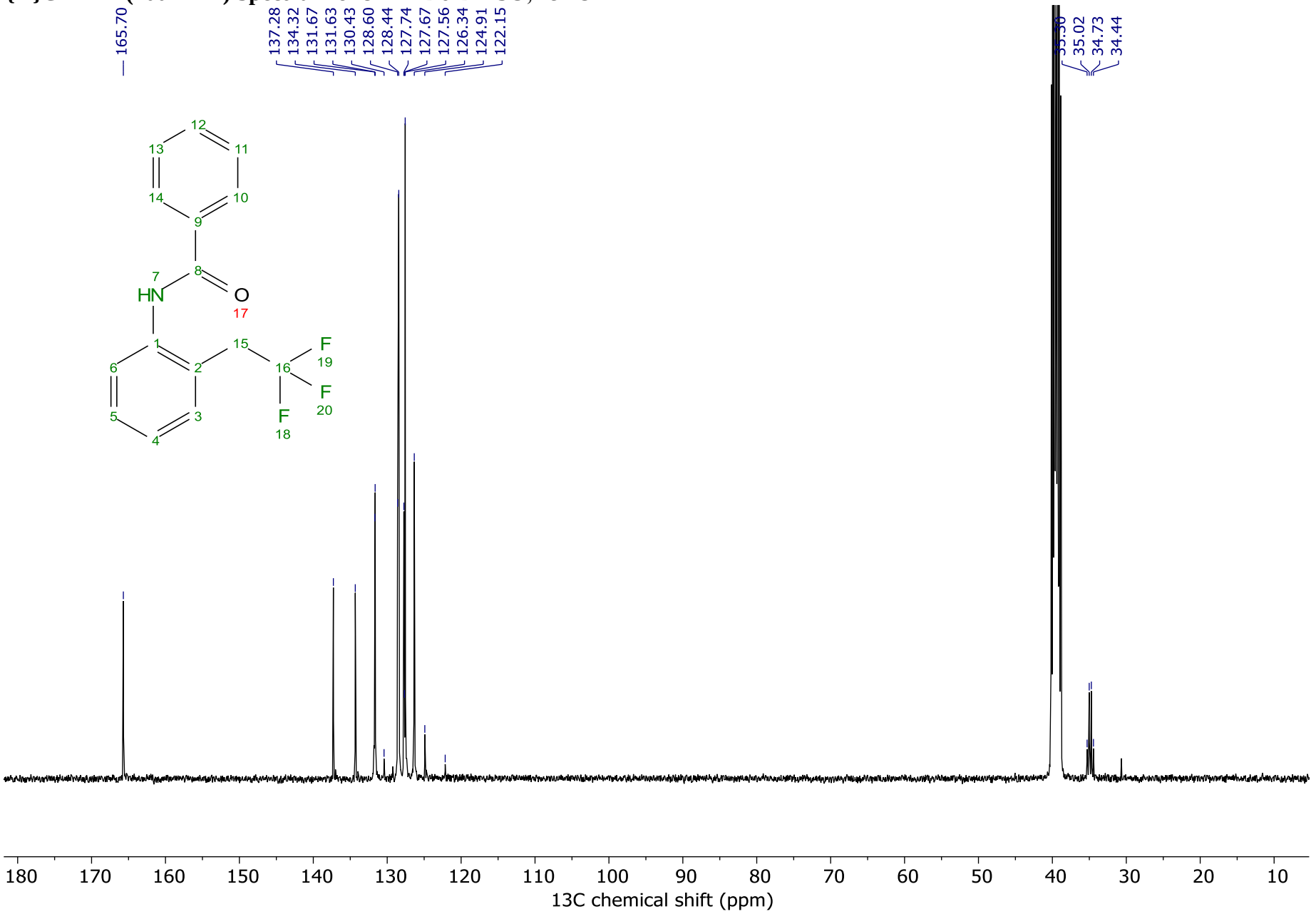
^{19}F-NMR (376 MHz) Spectrum of 4-Pd in CD_2Cl_2, 25 °C



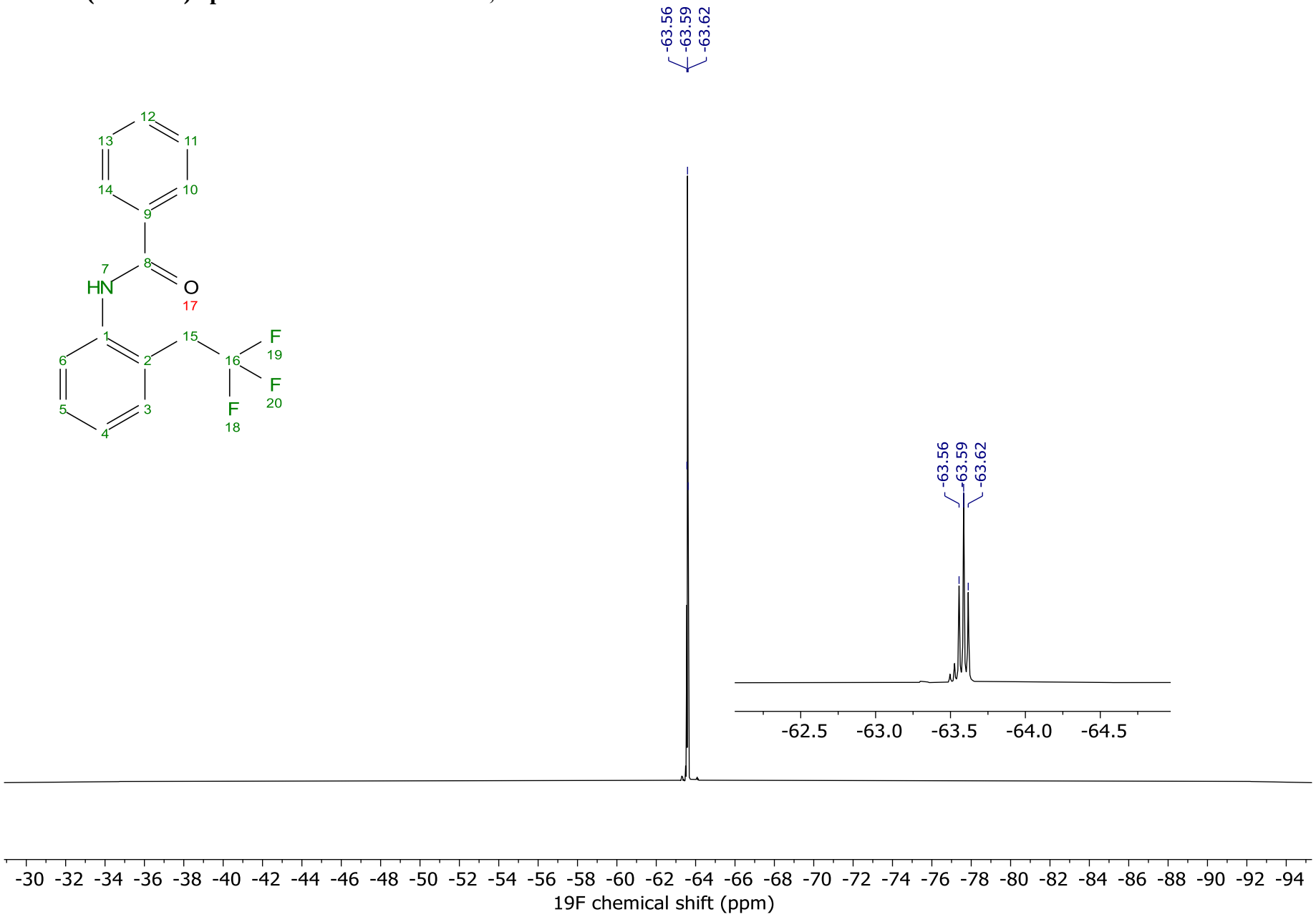
[1]H-NMR (400 MHz) Spectrum of 8m in D6-DMSO, 25 °C



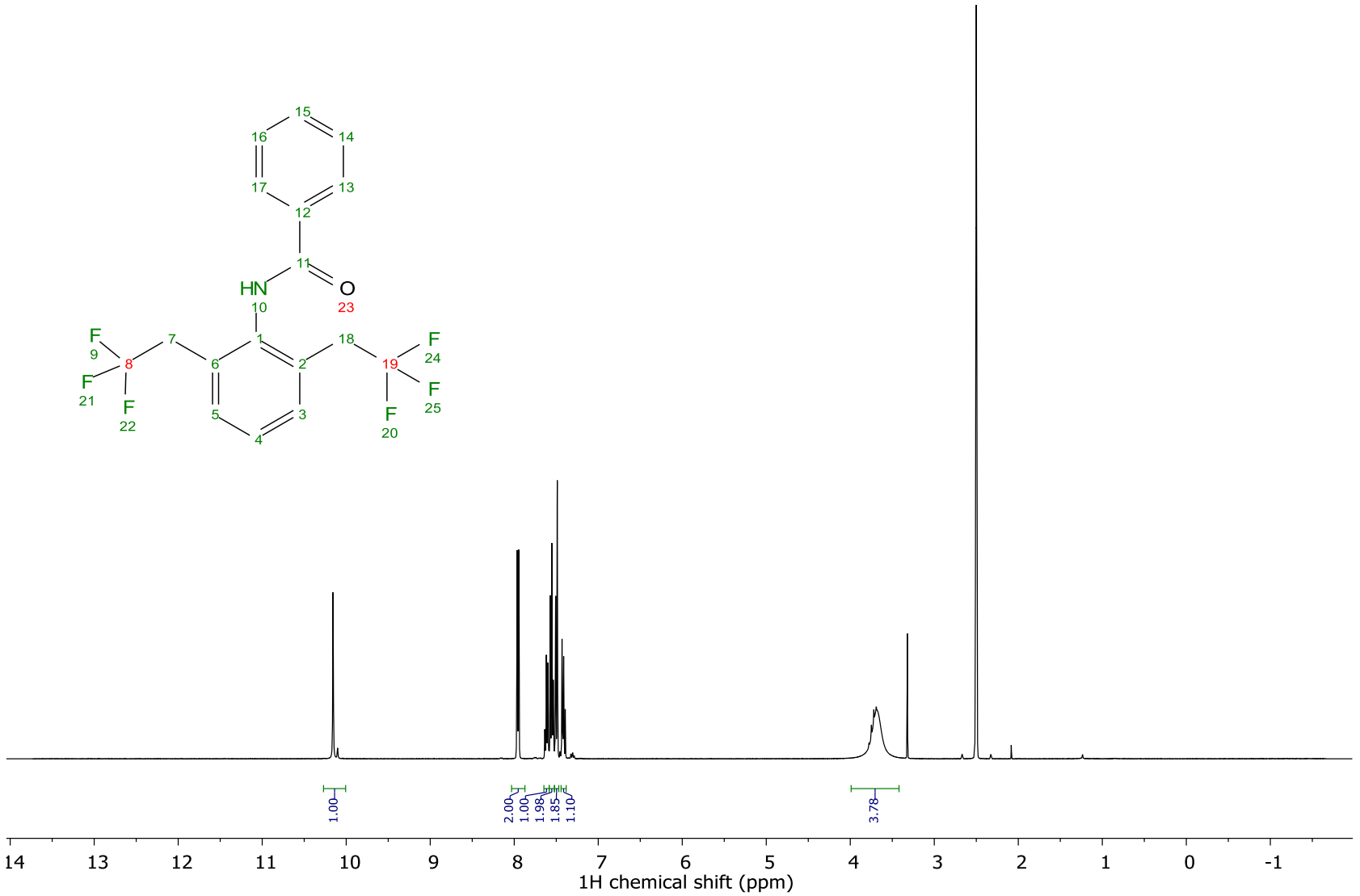
^{13}H}C-NMR (100 MHz) Spectrum of 8m in D6-DMSO, 25 °C



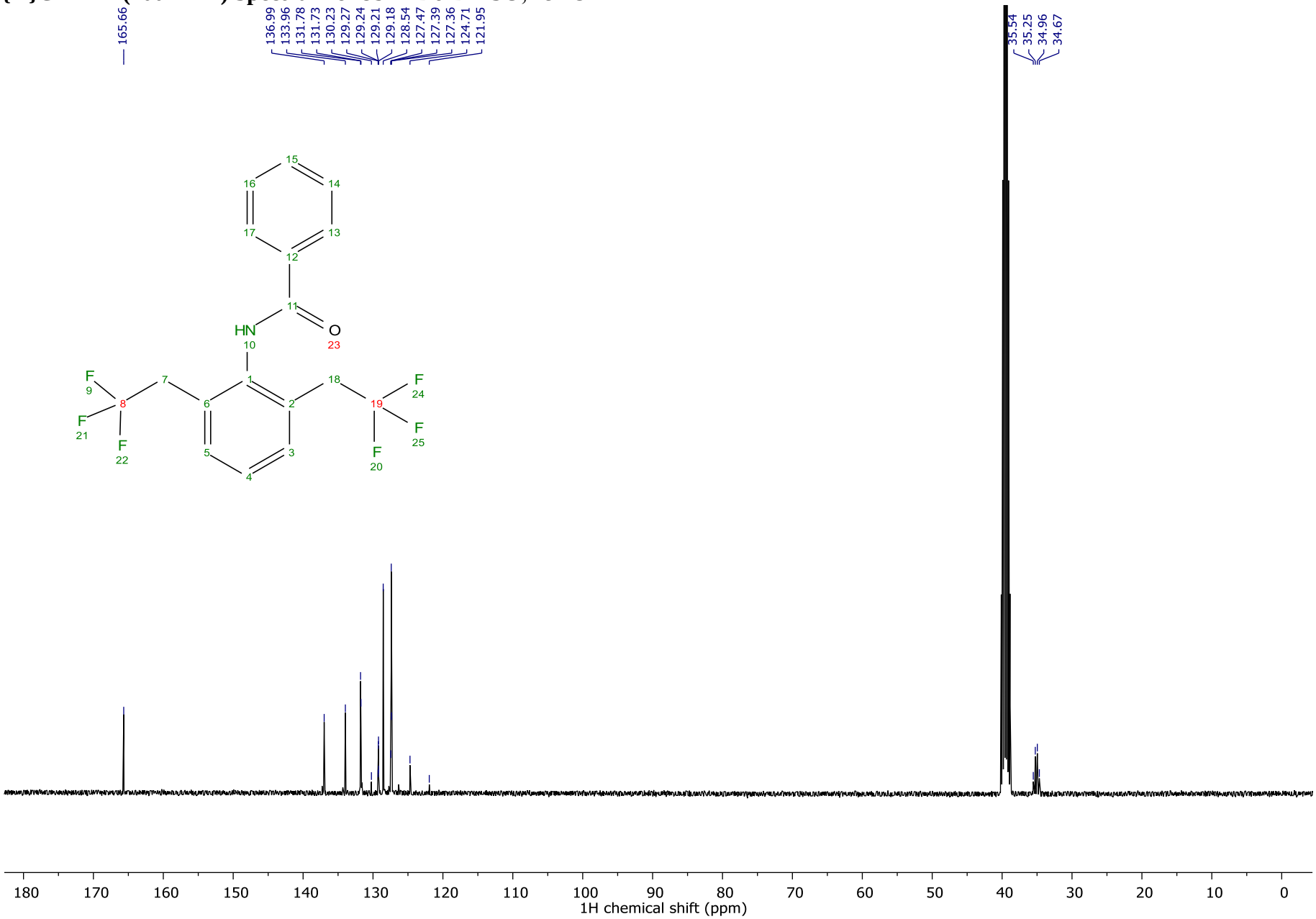
^{19}F-NMR (376 MHz) Spectrum of 8m in D6-DMSO, 25 °C



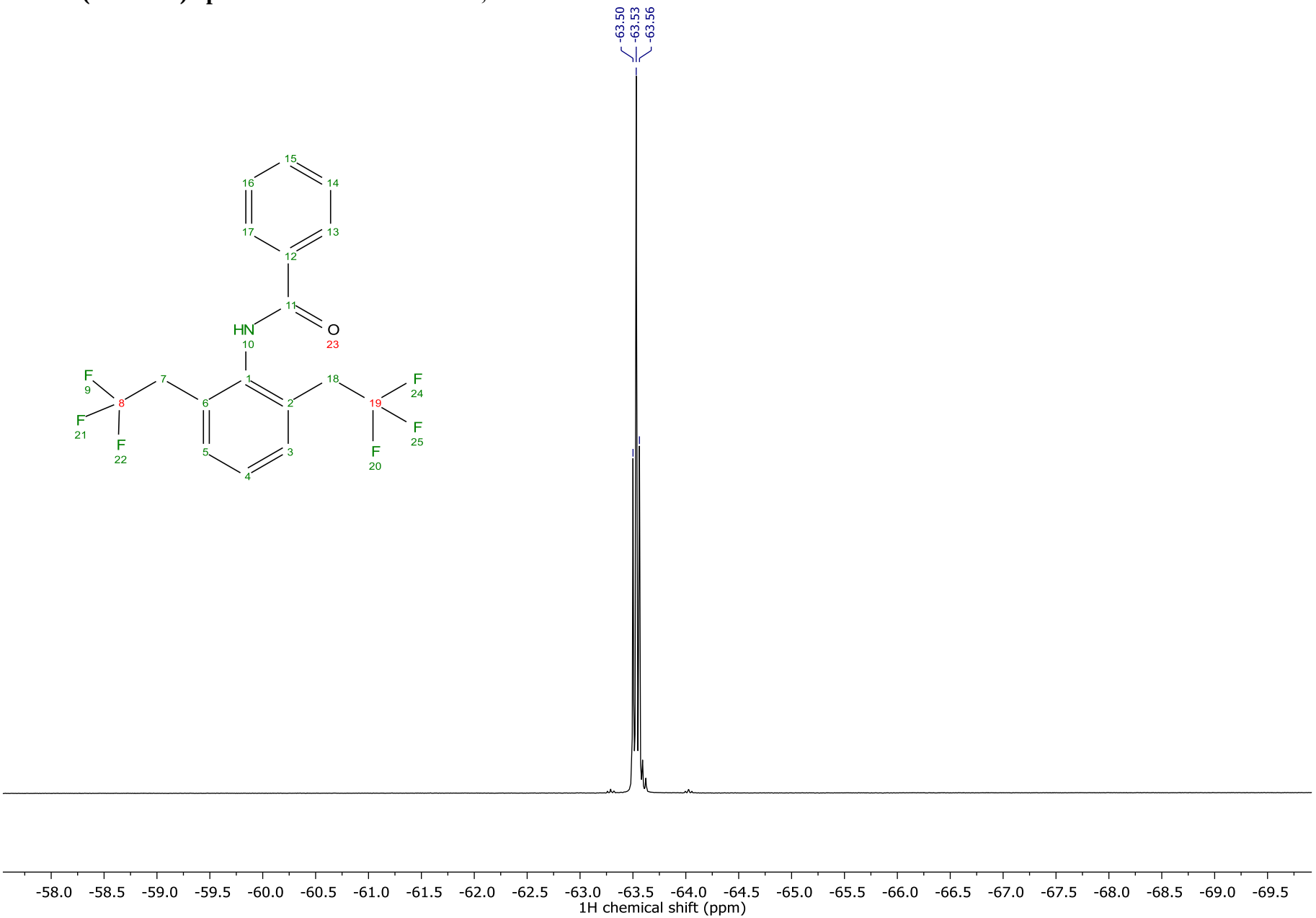
[1]H-NMR (400 MHz) Spectrum of 8b in D6-DMSO, 25 °C



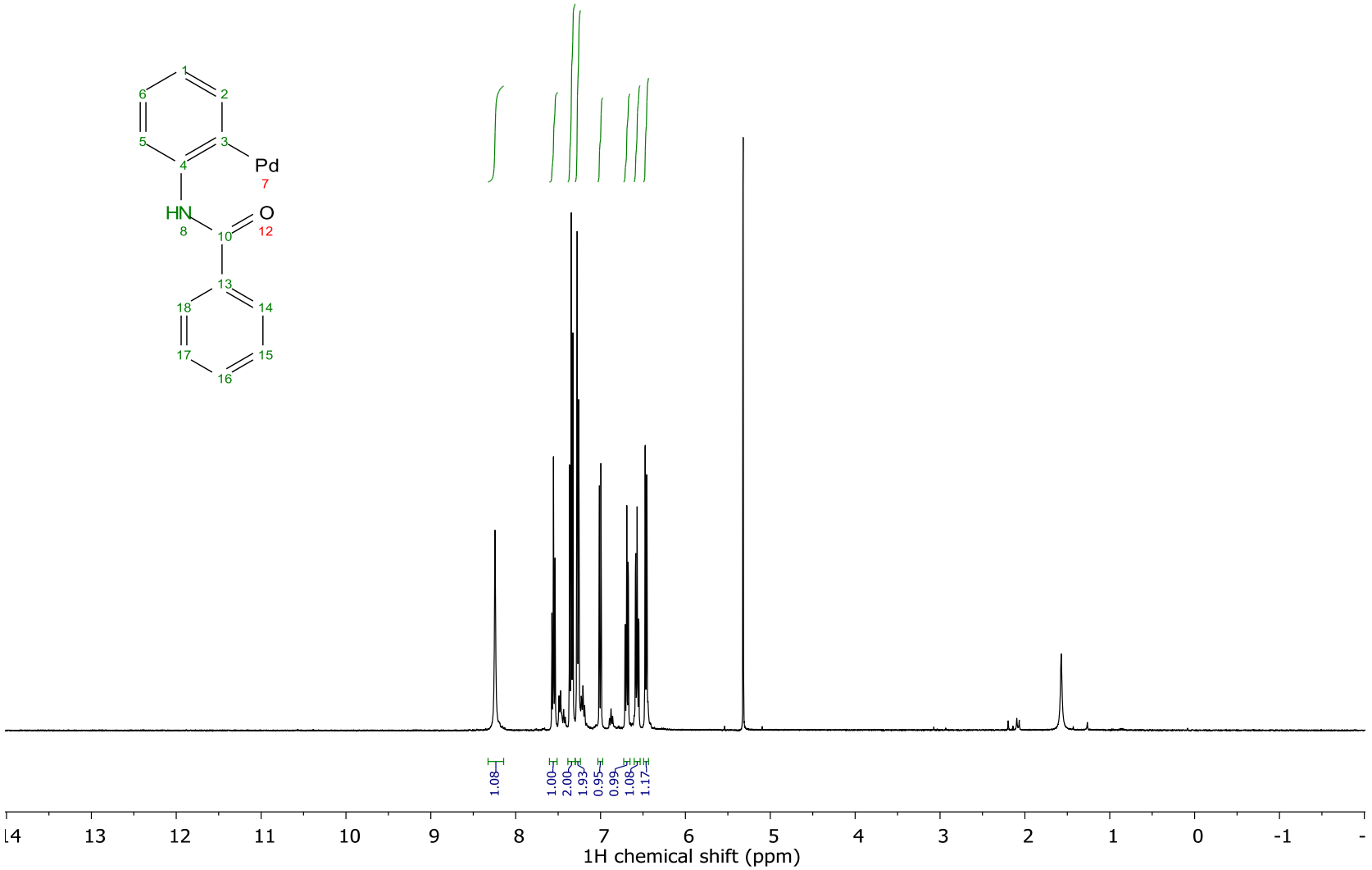
^{13}H}C-NMR (100 MHz) Spectrum of 8b in D6-DMSO, 25 °C



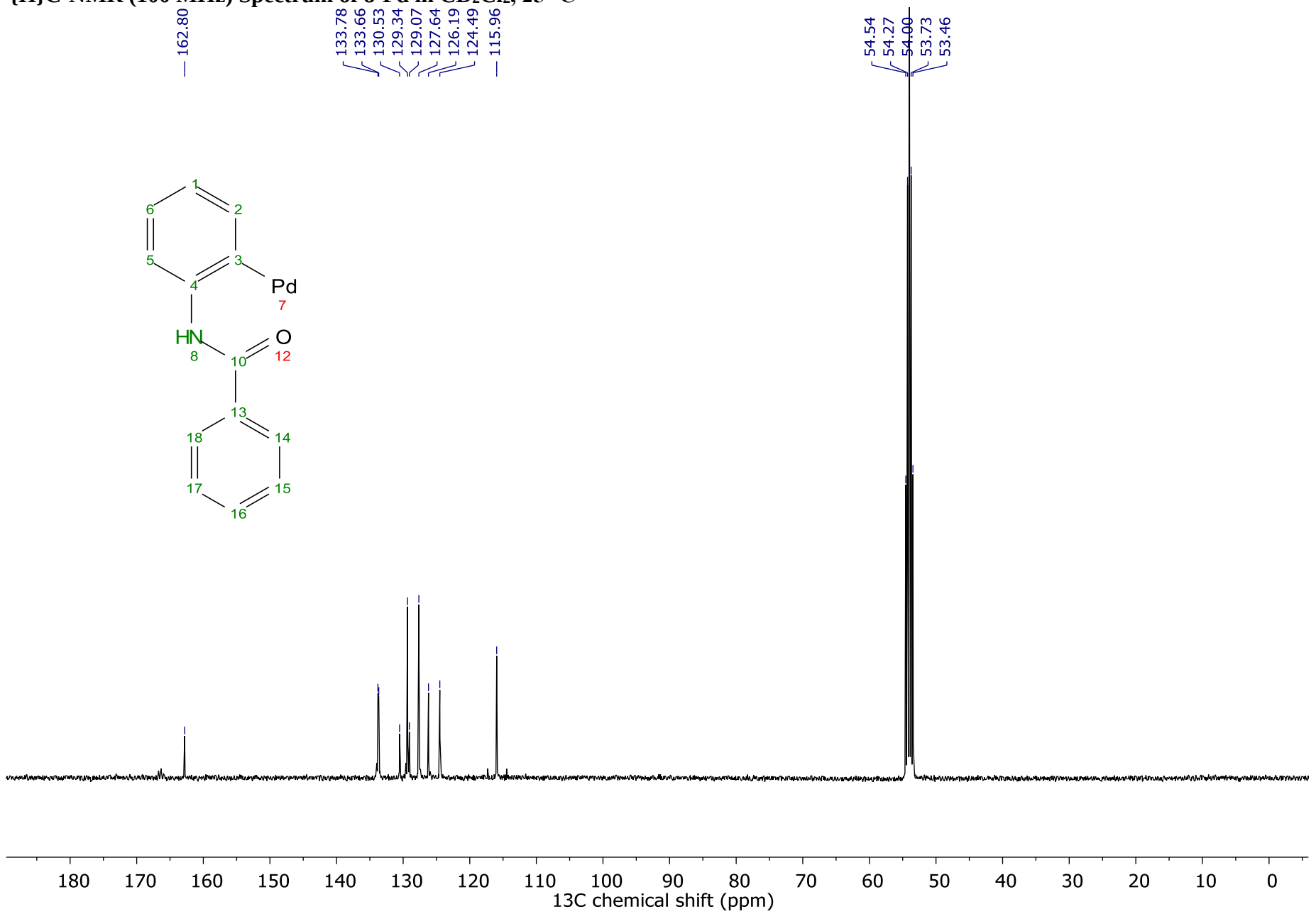
^{19}F-NMR (376 MHz) Spectrum of 8b in D6-DMSO, 25 °C



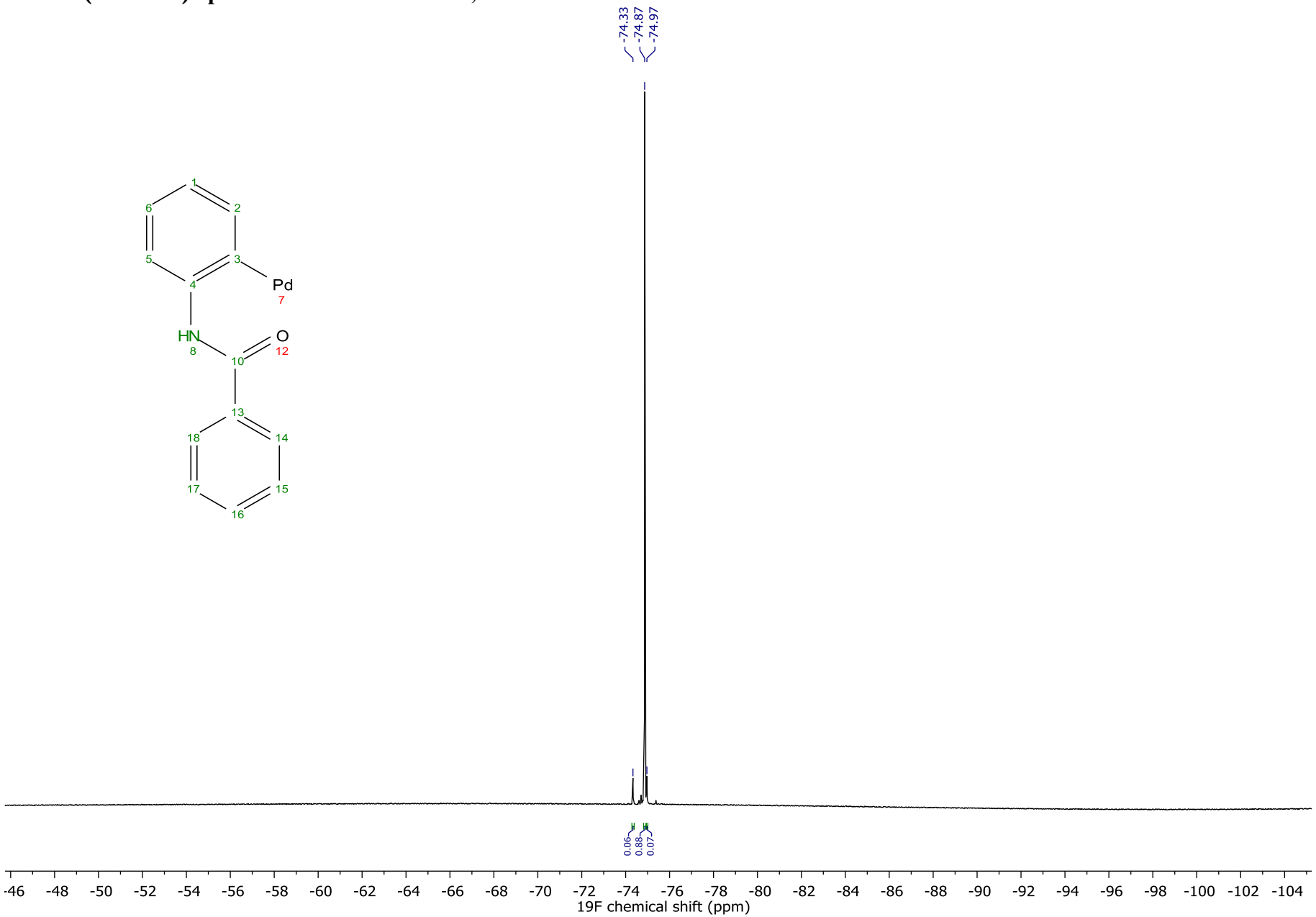
^{1}H-NMR (400 MHz) Spectrum of 8-Pd in CD_2Cl_2, 25 °C



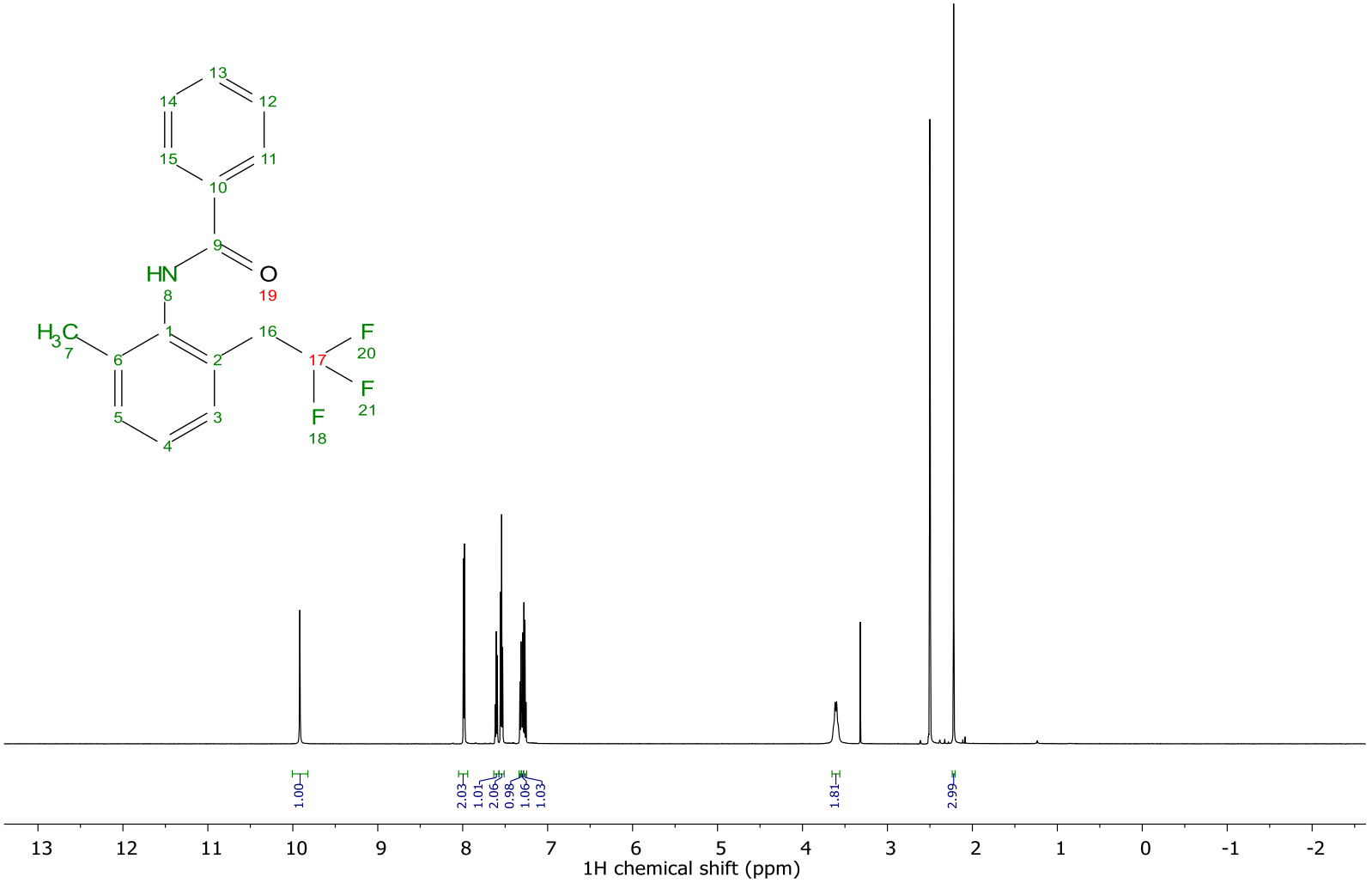
^{13}\{H\}C-NMR (100 MHz) Spectrum of 8-Pd in CD_2Cl_2, 25 °C



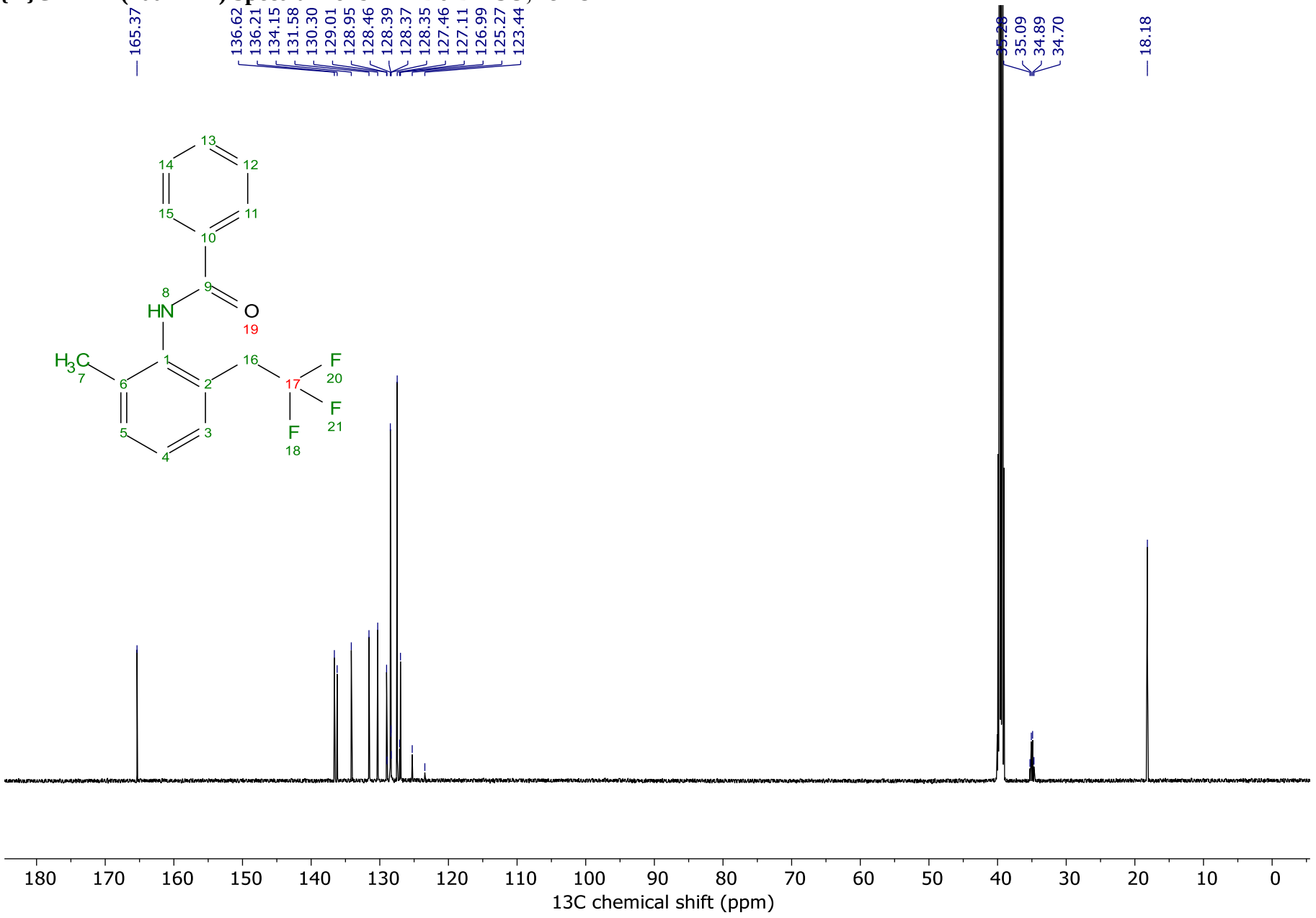
^{19}F-NMR (376 MHz) Spectrum of 8-Pd in CD_2Cl_2, 25 °C



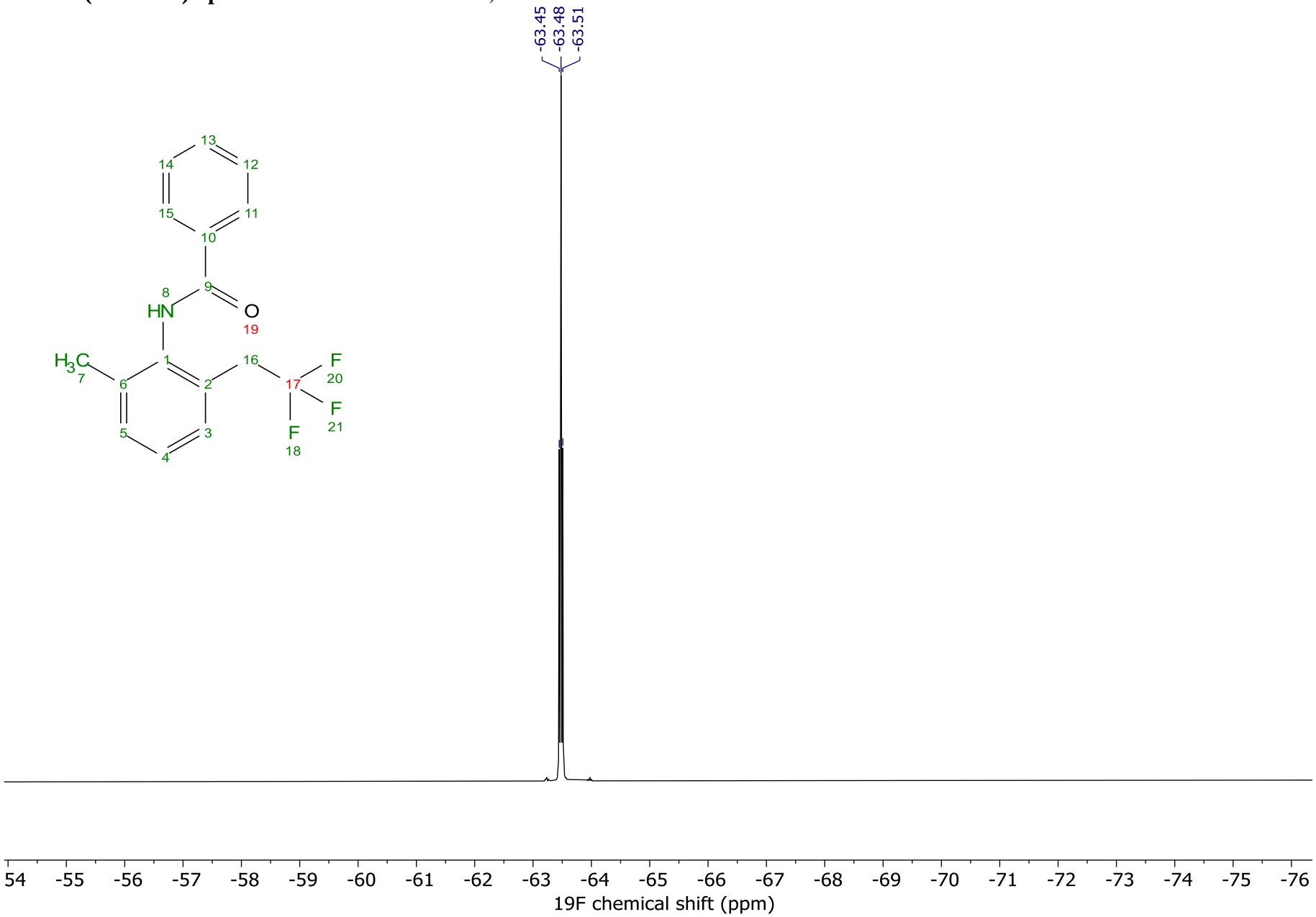
[1]H-NMR (400 MHz) Spectrum of 9m in D6-DMSO, 25 °C



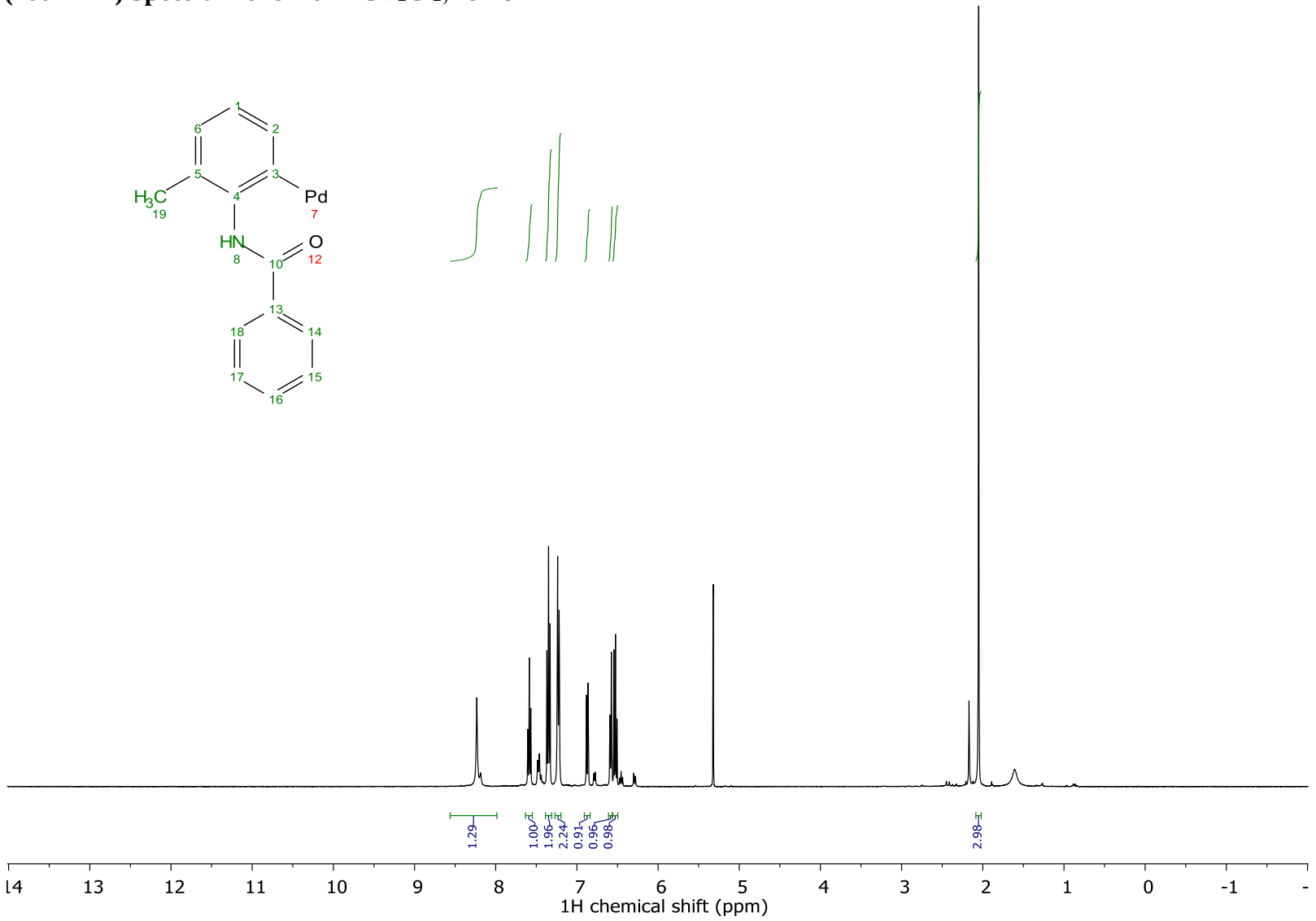
^{13}H}C-NMR (100 MHz) Spectrum of 9m in D6-DMSO, 25 °C



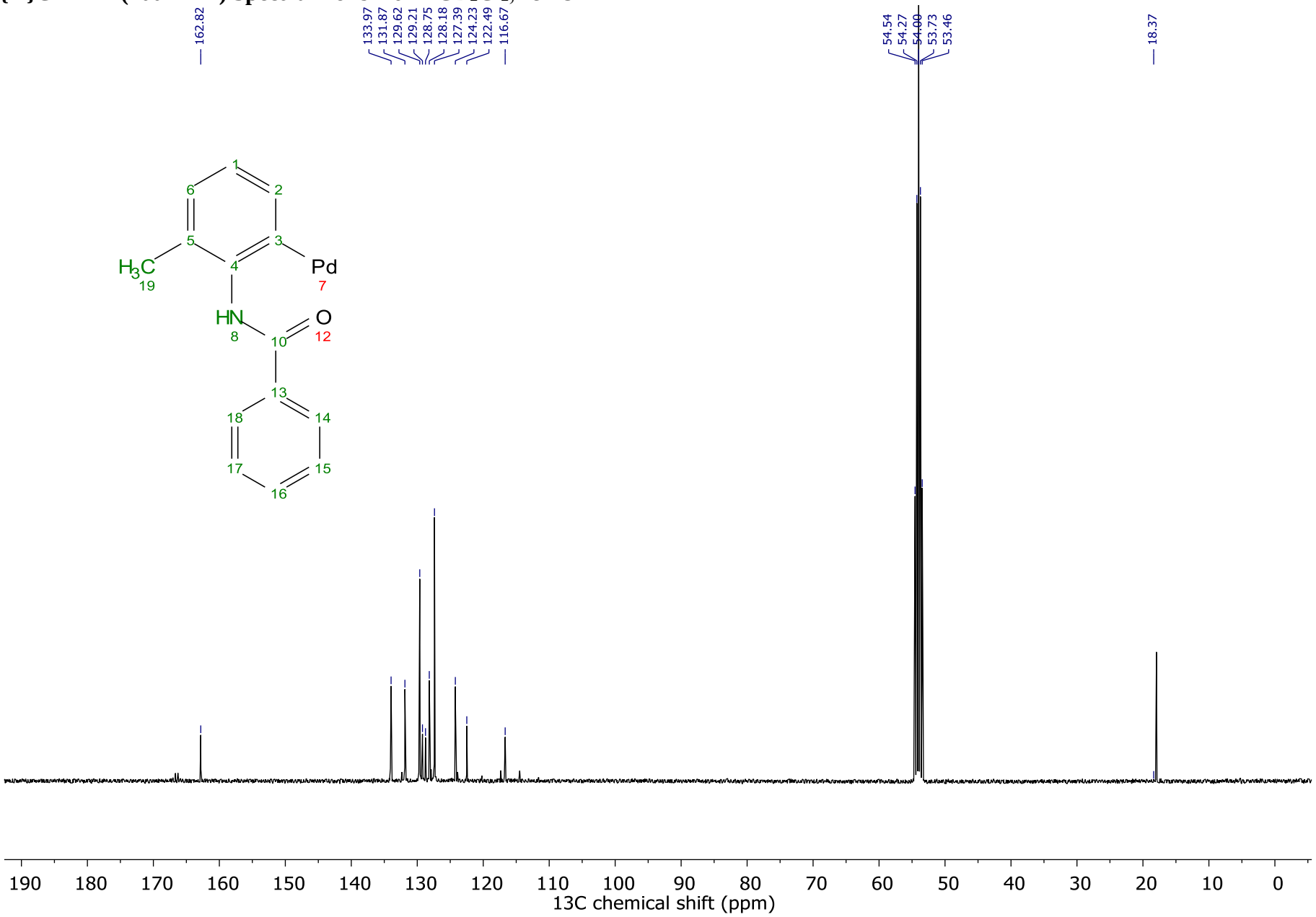
^{19}F-NMR (376 MHz) Spectrum of 9m in D6-DMSO, 25 °C



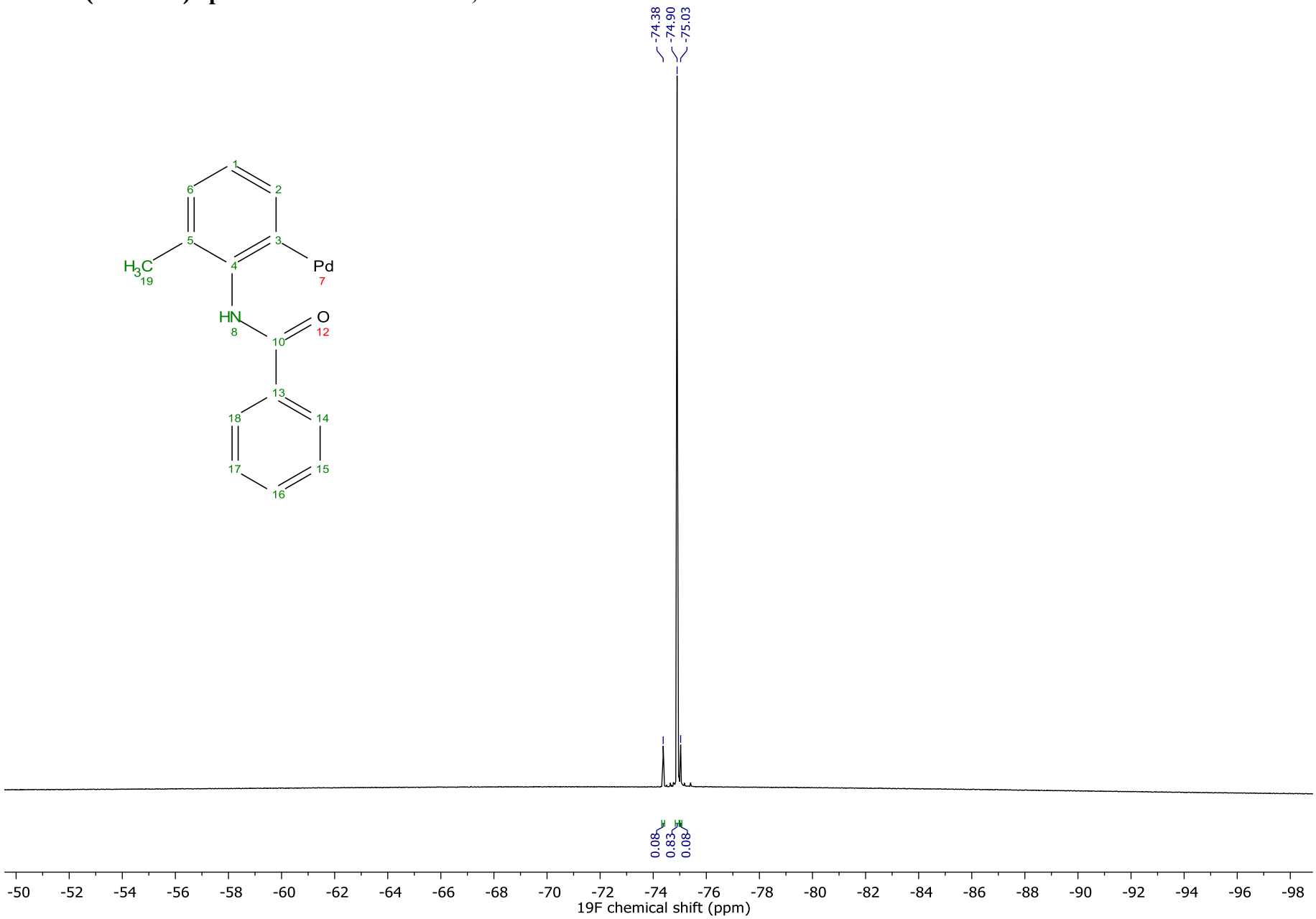
^{1}H-NMR (400 MHz) Spectrum of 9-Pd in CD_2Cl_2, 25 °C



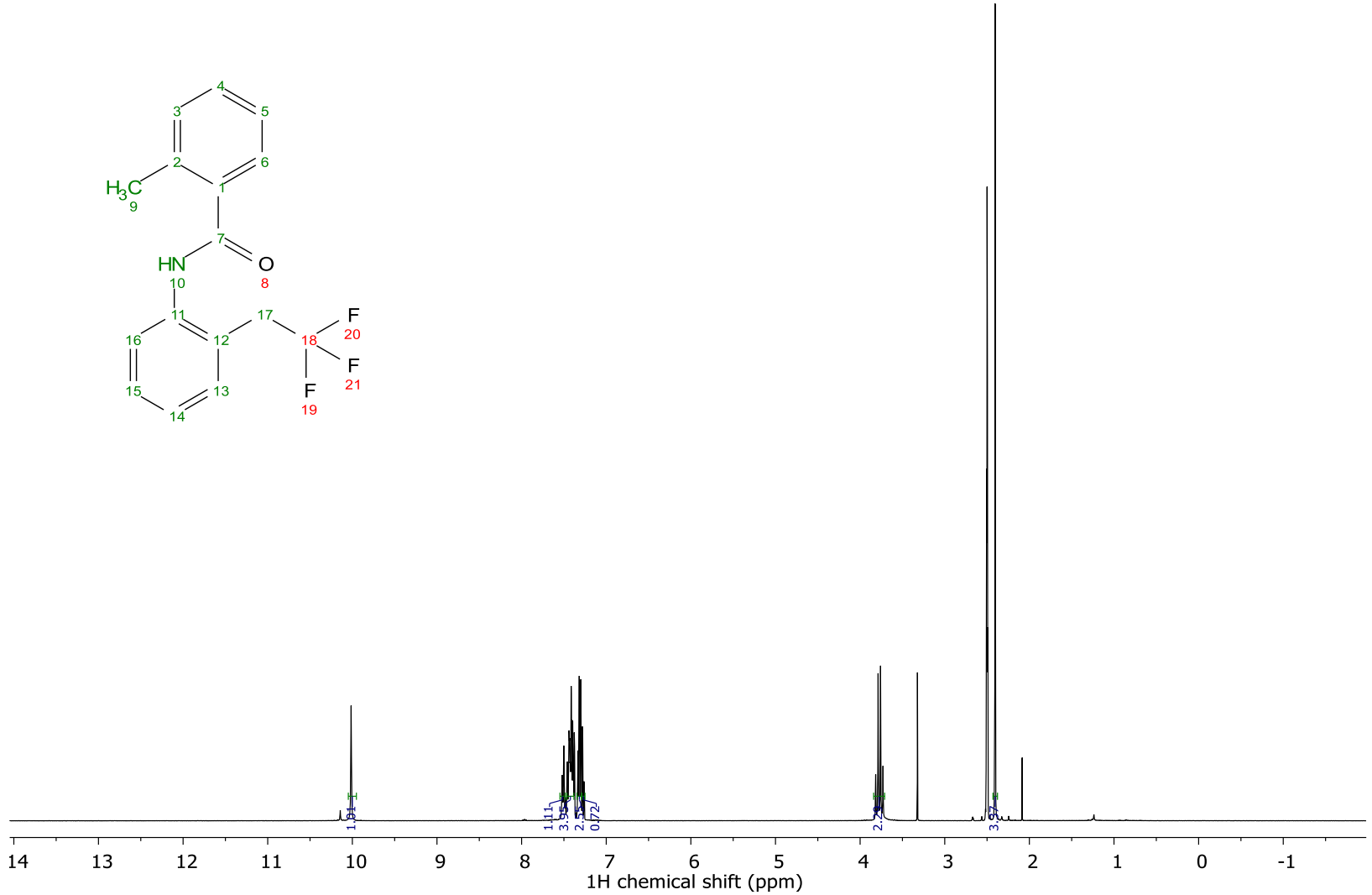
^{13}\{H\}C-NMR (100 MHz) Spectrum of 9-Pd in CD_2Cl_2, 25 °C



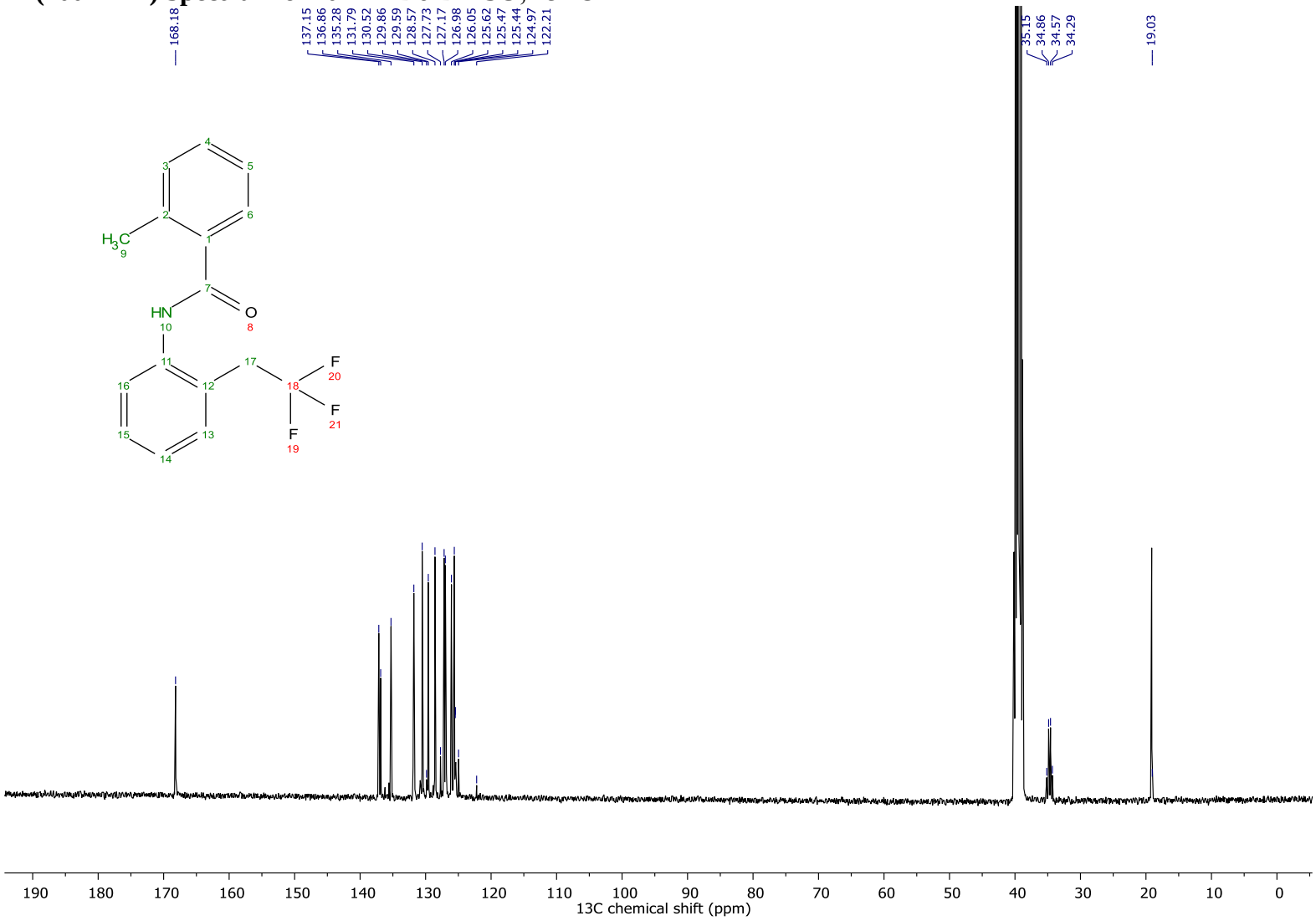
^{19}F-NMR (376 MHz) Spectrum of 9-Pd in CD_2Cl_2, 25 °C



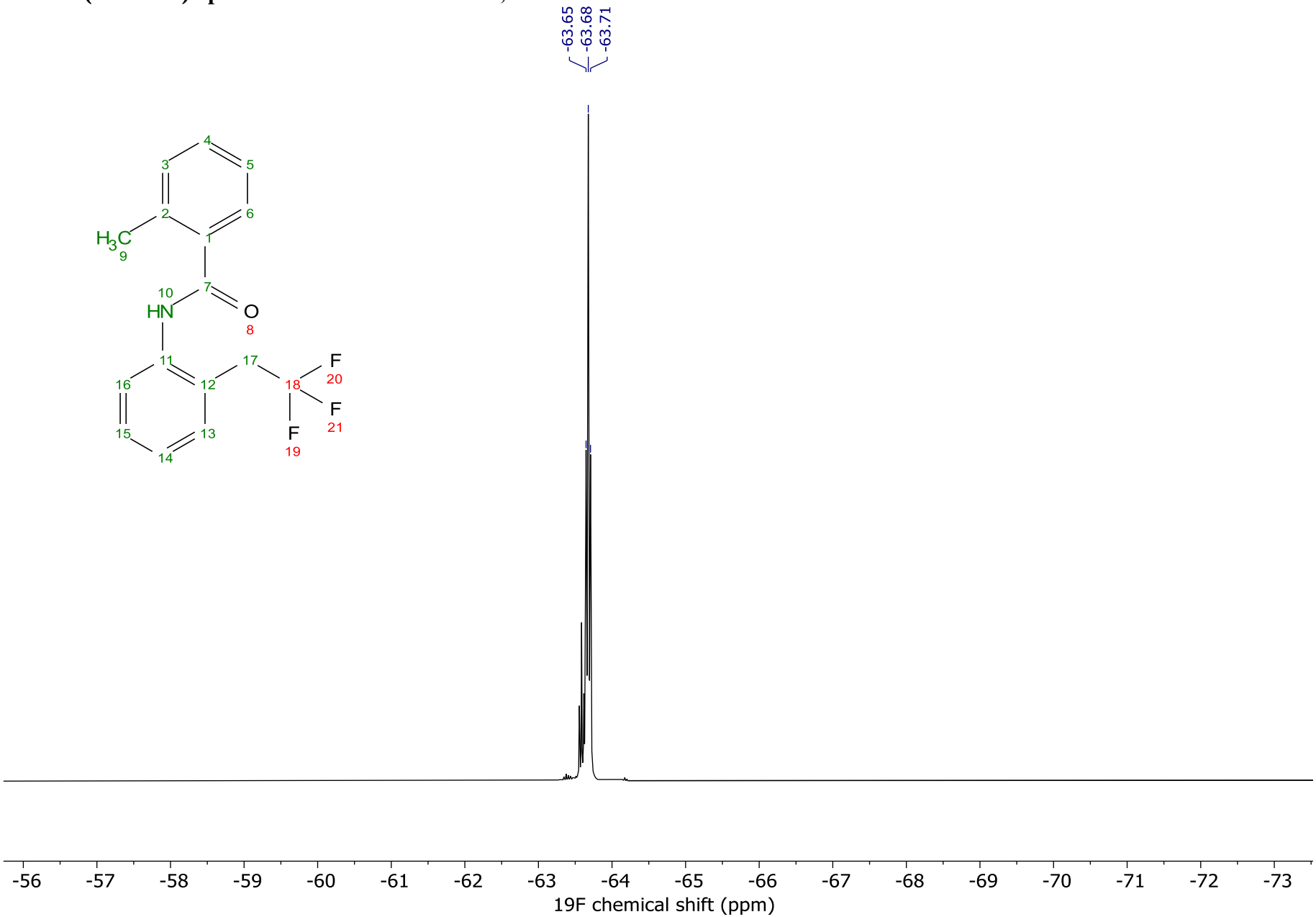
[1]H-NMR (400 MHz) Spectrum of 10m in D6-DMSO, 25 °C



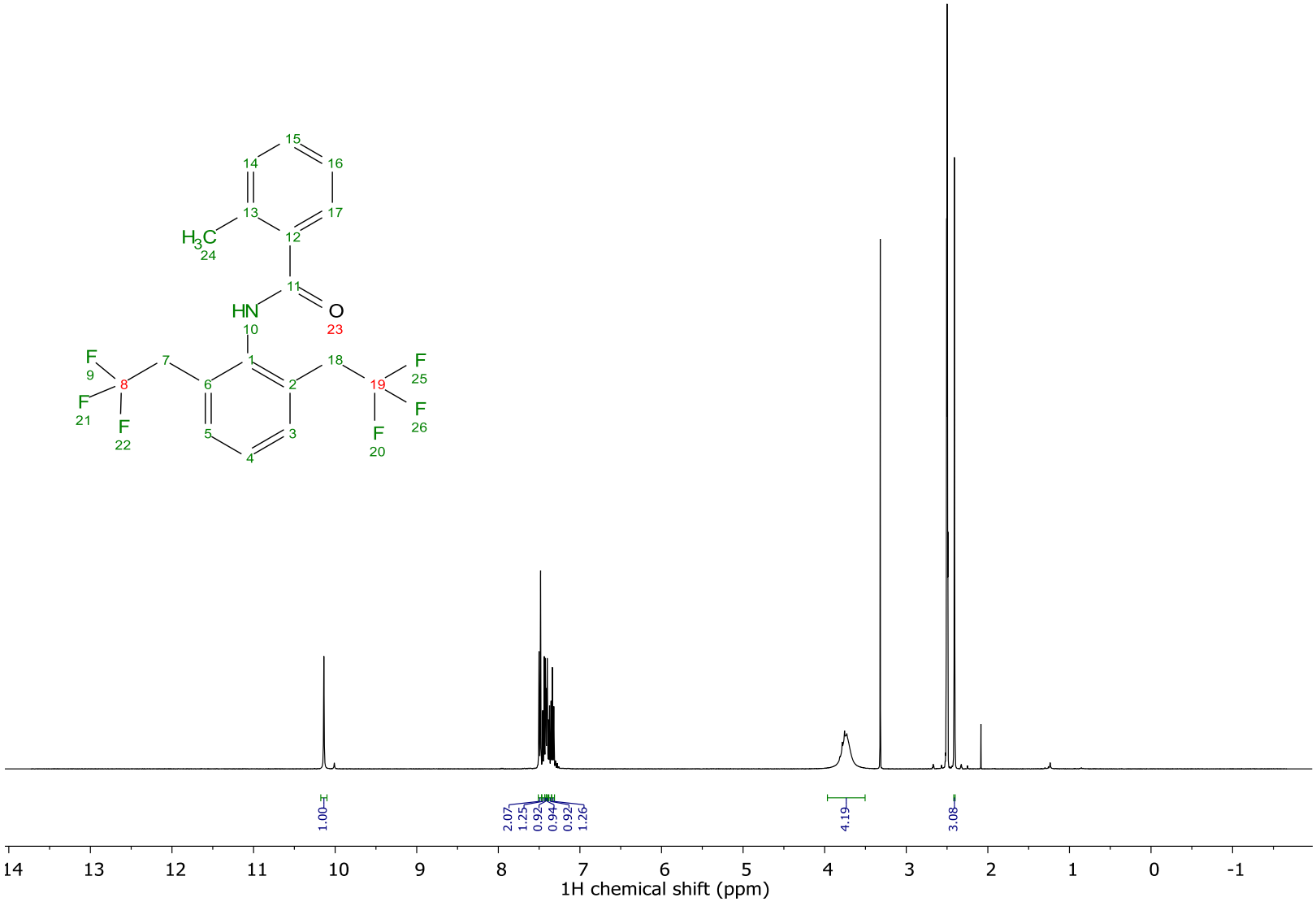
^{13}H}C-NMR (100 MHz) Spectrum of 10m in D6-DMSO, 25 °C



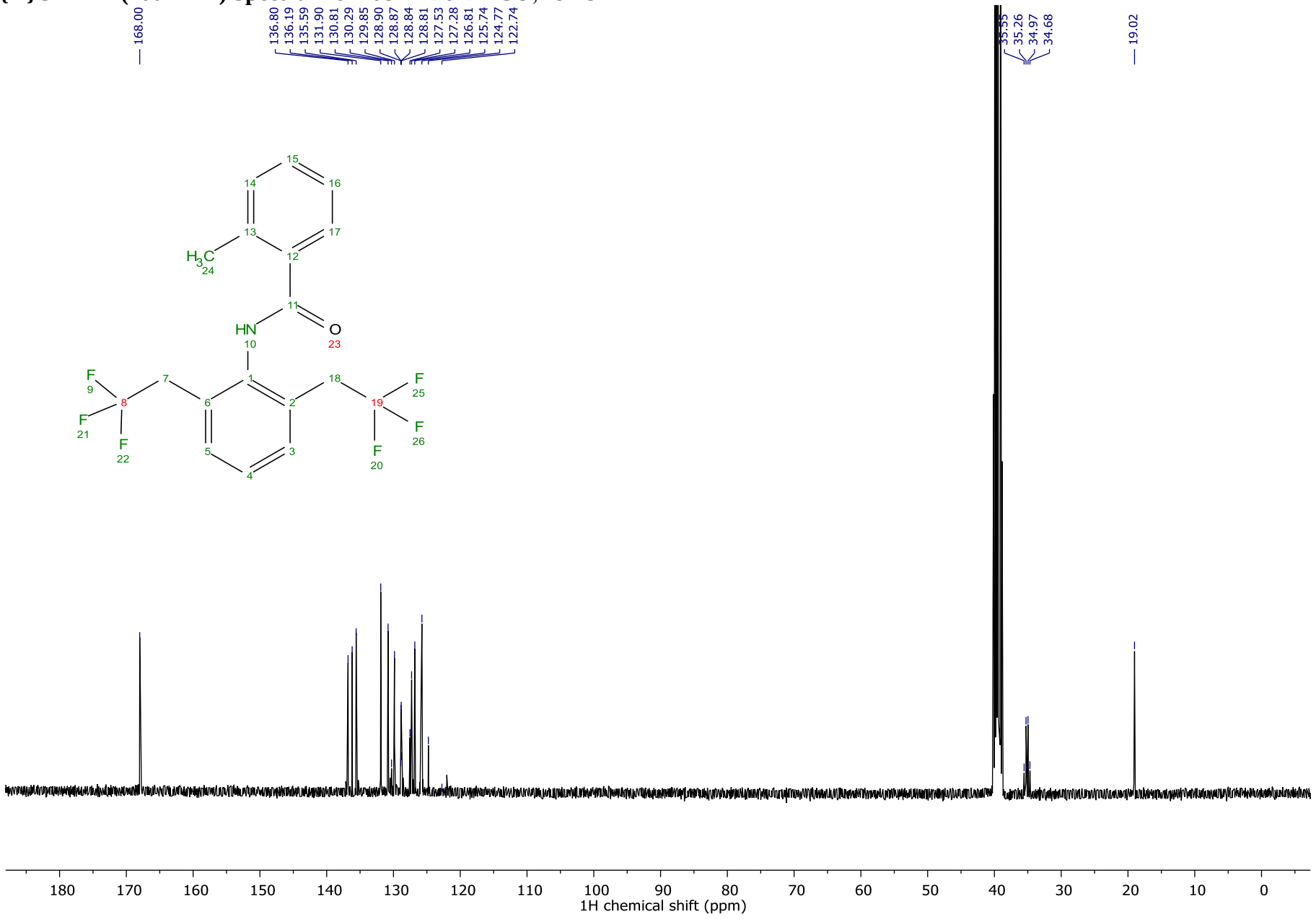
^{19}F-NMR (376 MHz) Spectrum of 10m in D6-DMSO, 25 °C



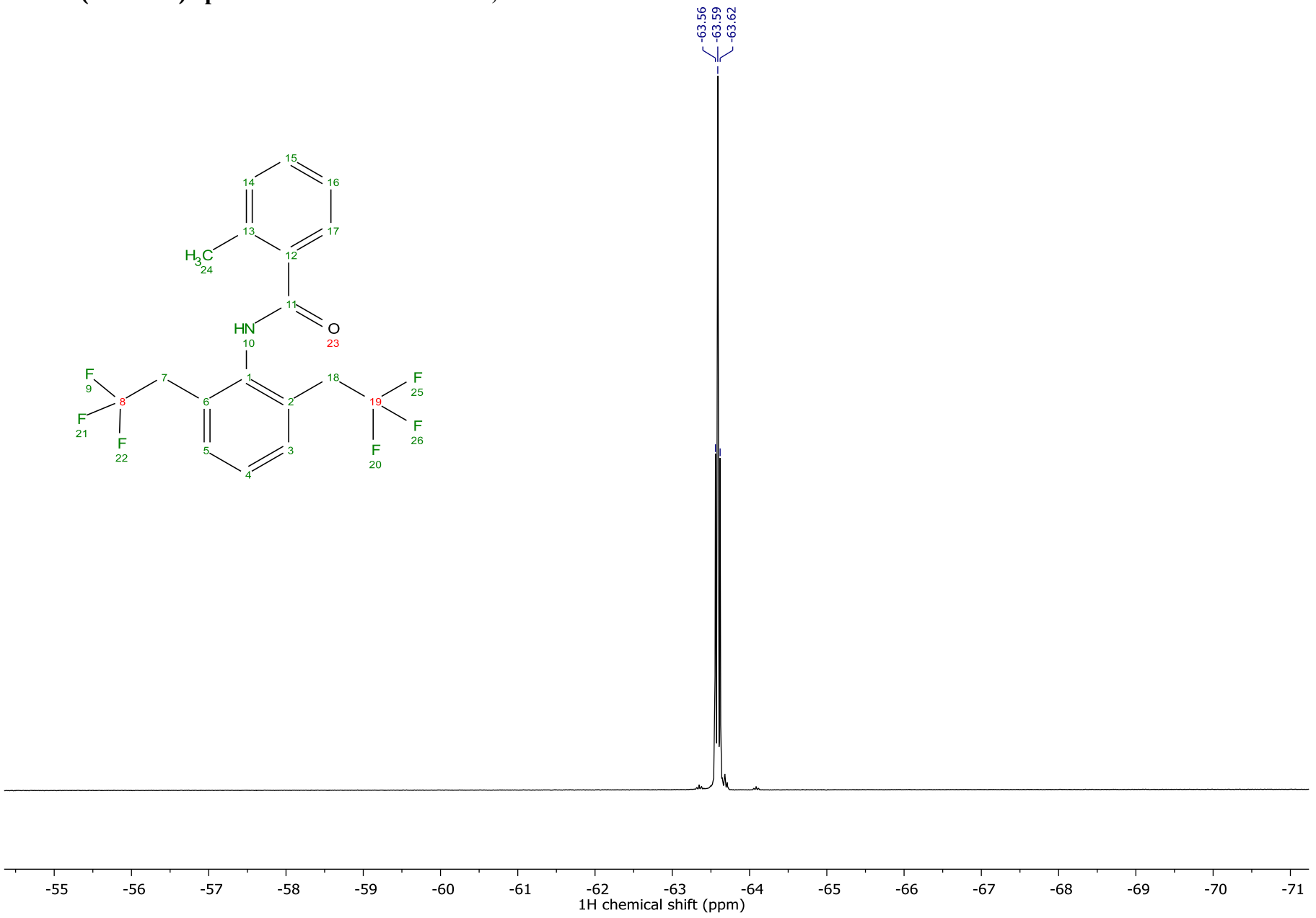
[1]H-NMR (400 MHz) Spectrum of 10b in D6-DMSO, 25 °C



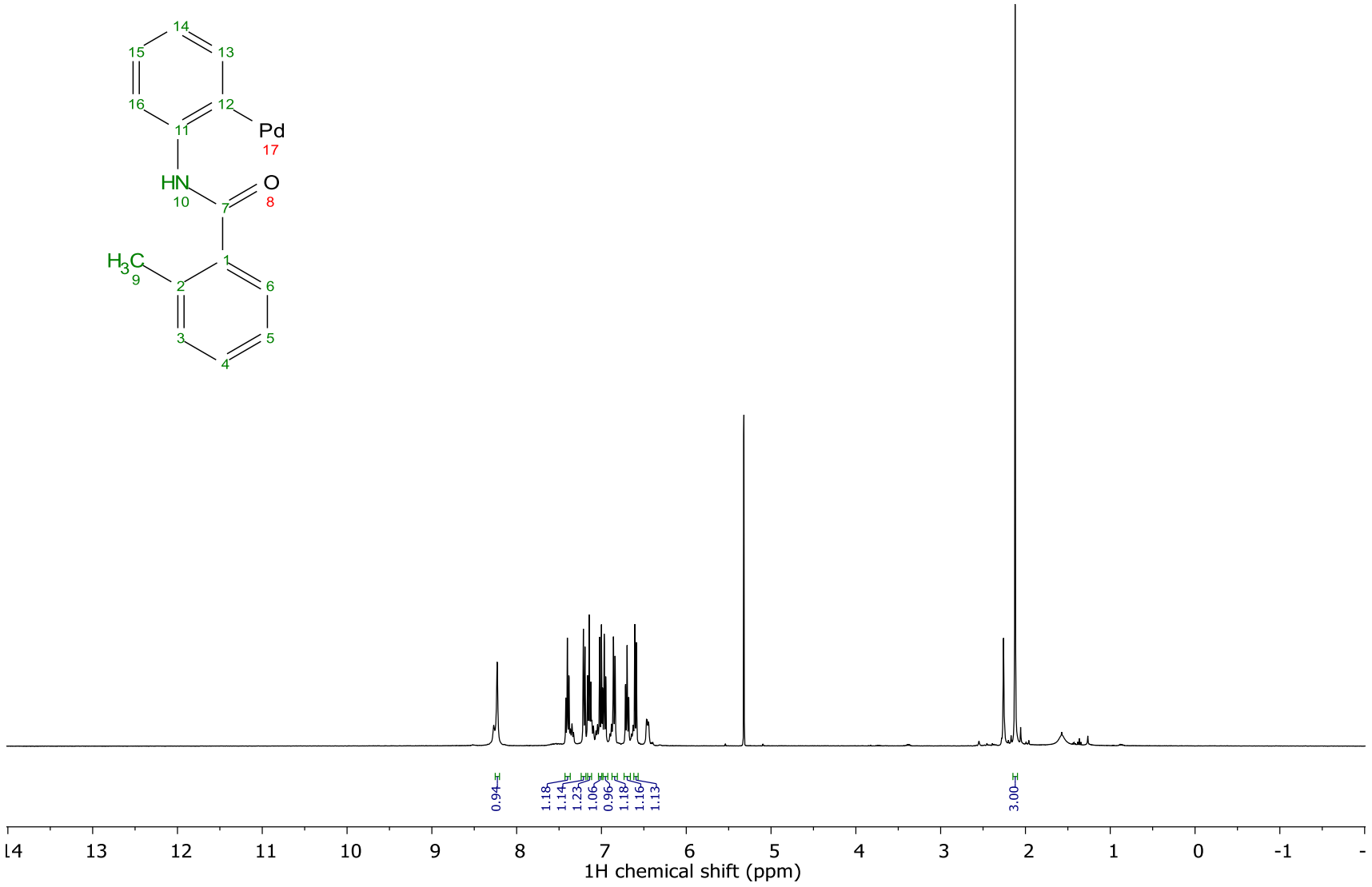
^{13}H}C-NMR (100 MHz) Spectrum of 10b in D6-DMSO, 25 °C



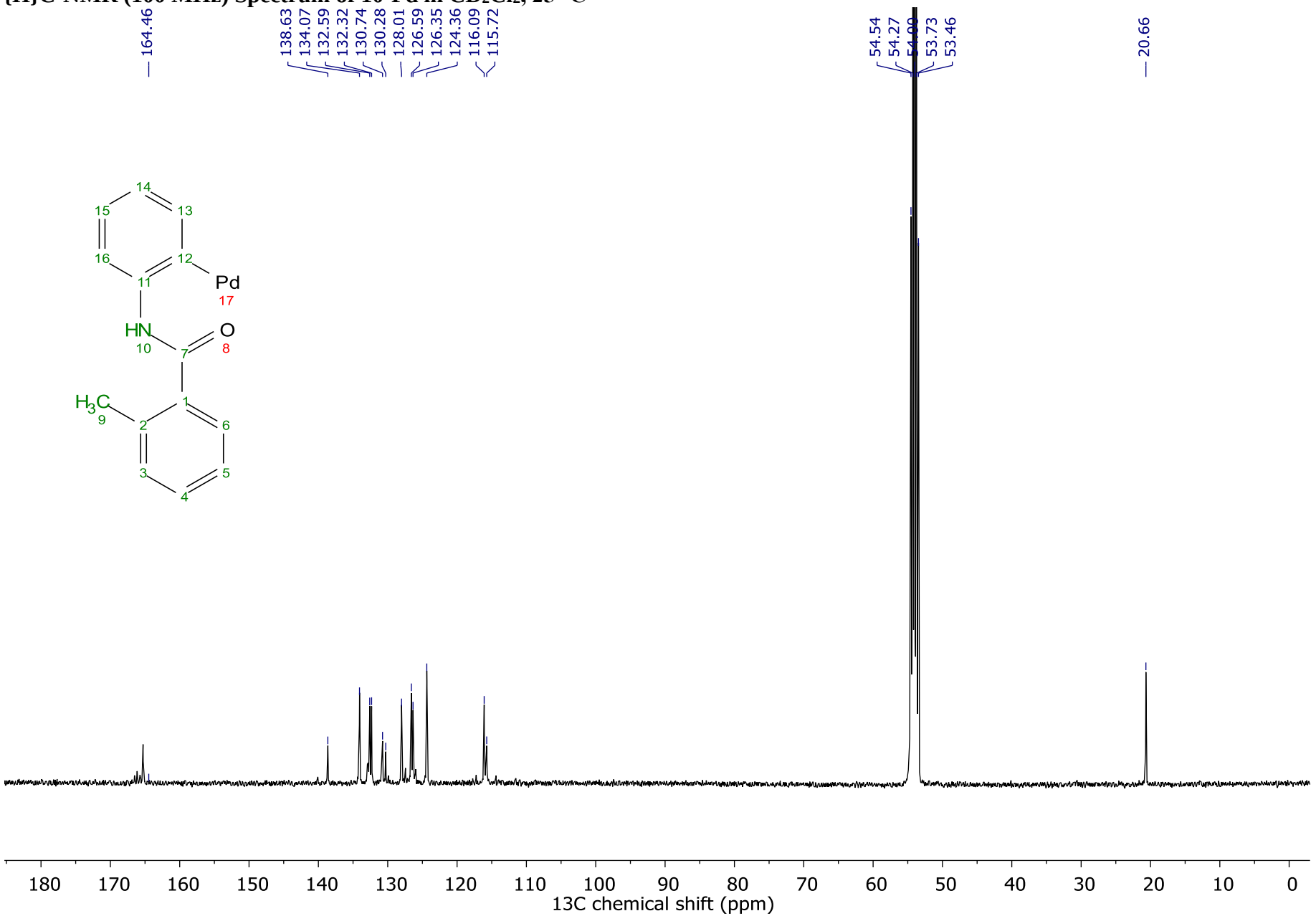
^{19}F-NMR (376 MHz) Spectrum of 10b in D6-DMSO, 25 °C



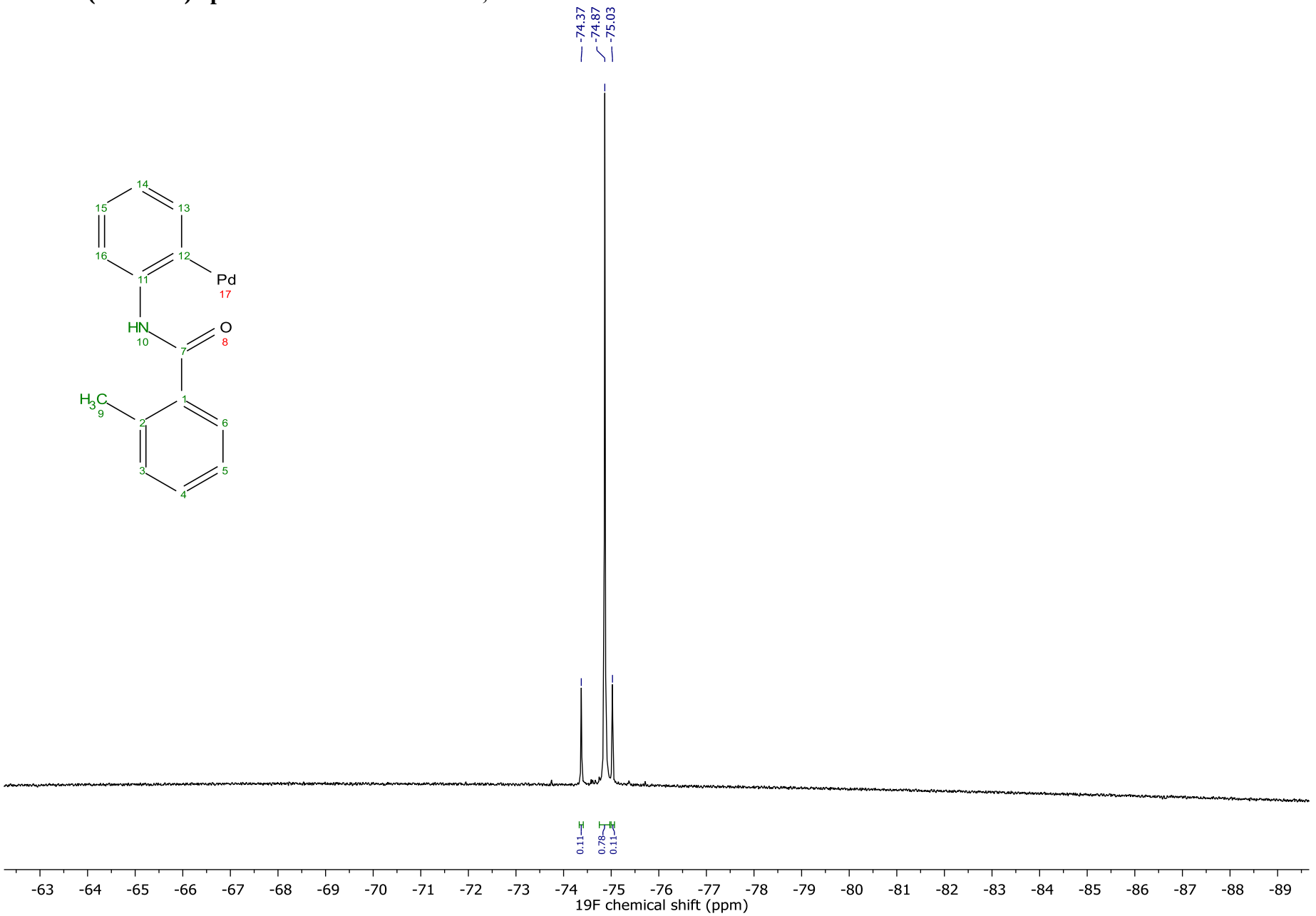
^{1}H-NMR (400 MHz) Spectrum of 10-Pd in CD_2Cl_2, 25 °C



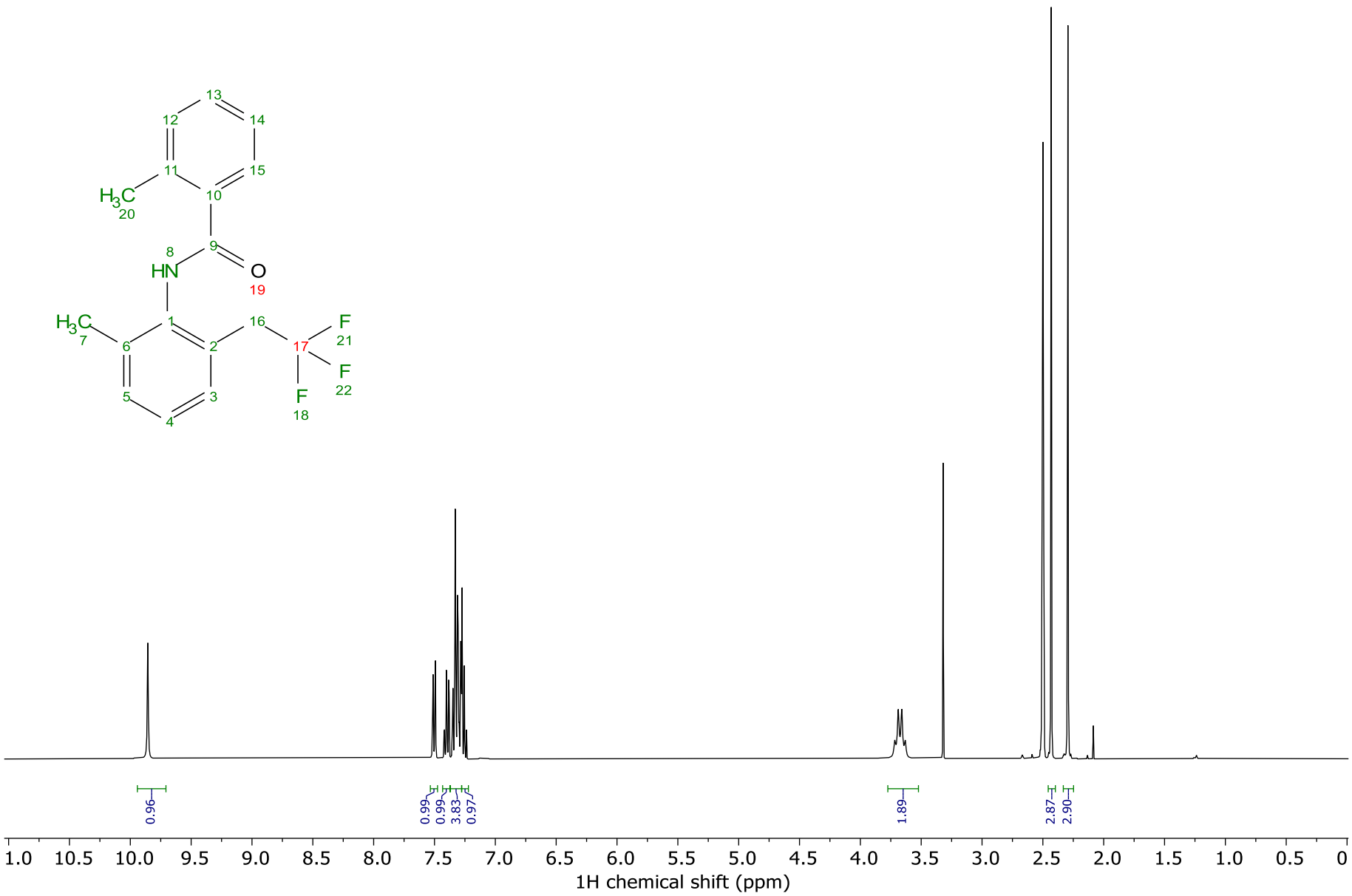
^{13}\{H\}C-NMR (100 MHz) Spectrum of 10-Pd in CD_2Cl_2, 25 °C



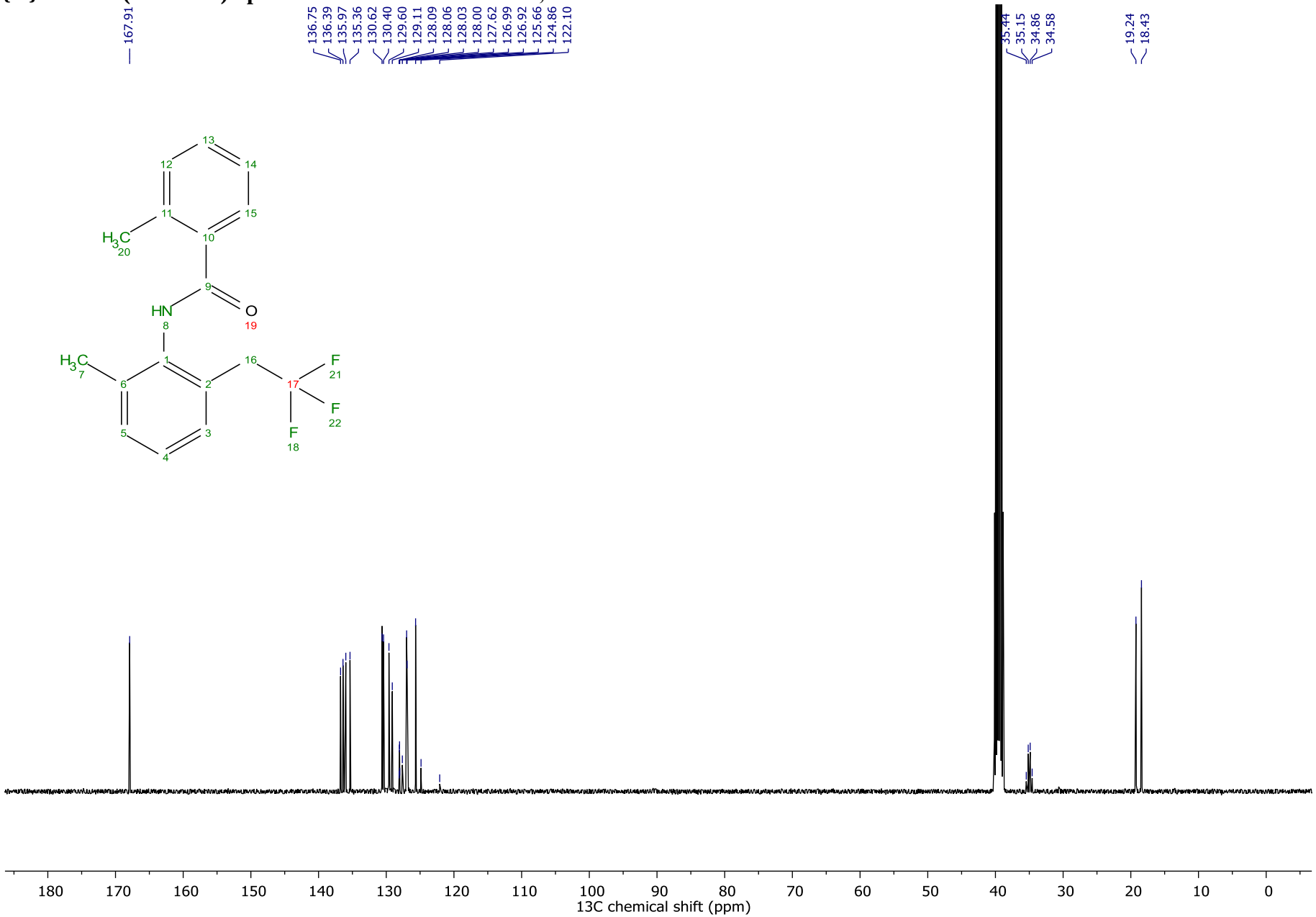
^{19}F-NMR (376 MHz) Spectrum of 10-Pd in CD_2Cl_2, 25 °C



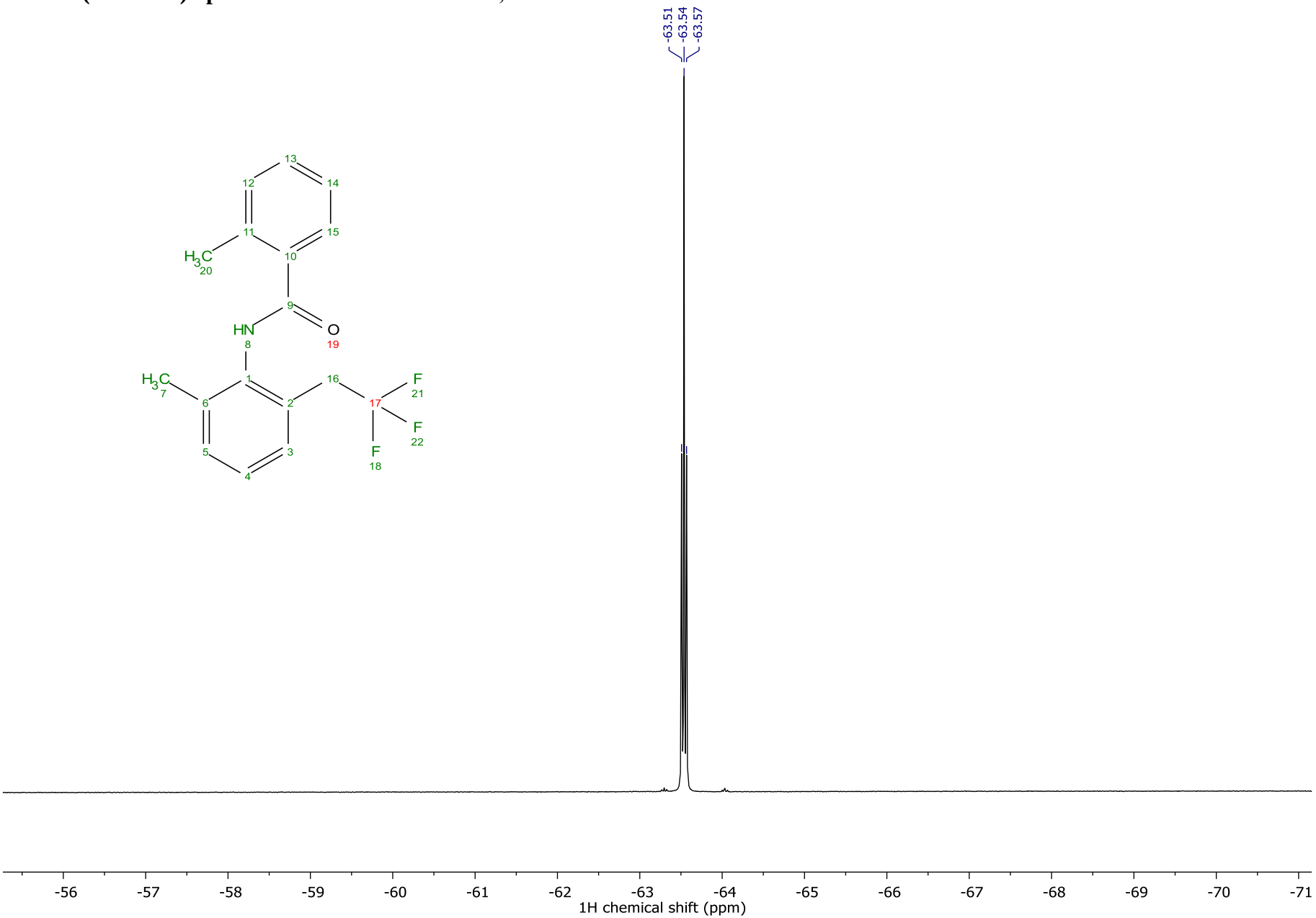
[1]H-NMR (400 MHz) Spectrum of 11m in D6-DMSO, 25 °C



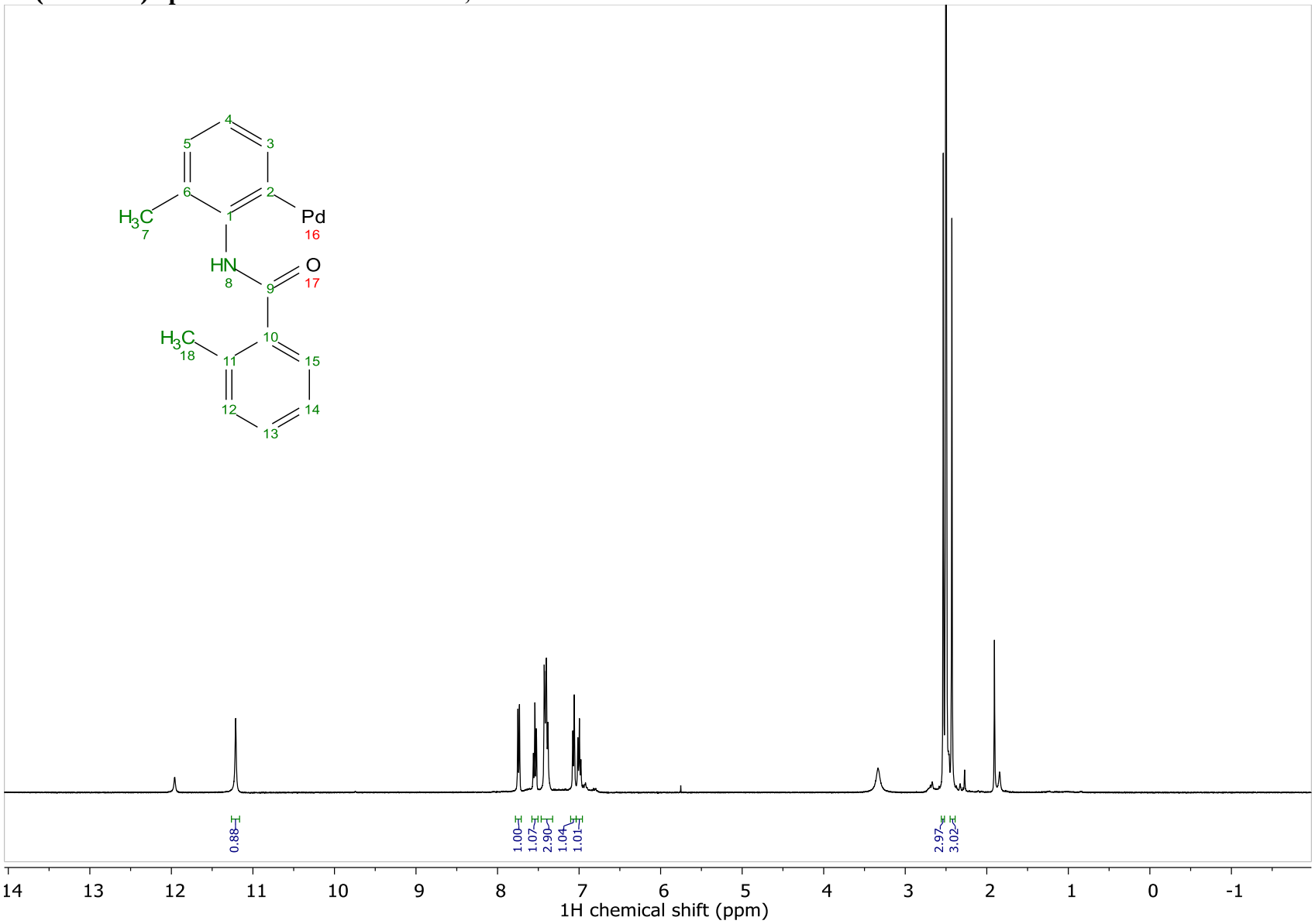
^{13}\{H\}C-NMR (100 MHz) Spectrum of 11m in D6-DMSO, 25 °C



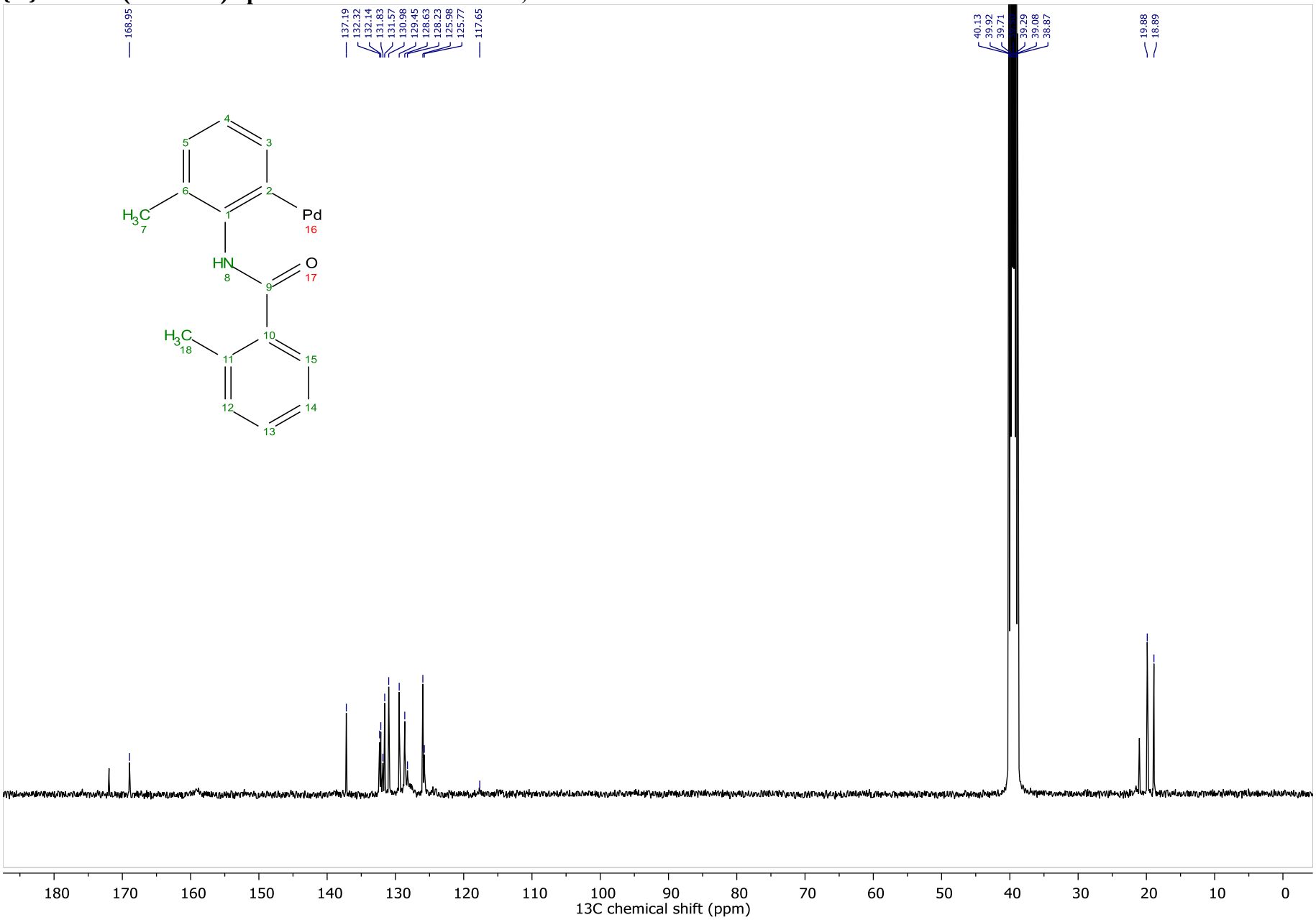
^{19}F-NMR (376 MHz) Spectrum of 11m in D6-DMSO, 25 °C



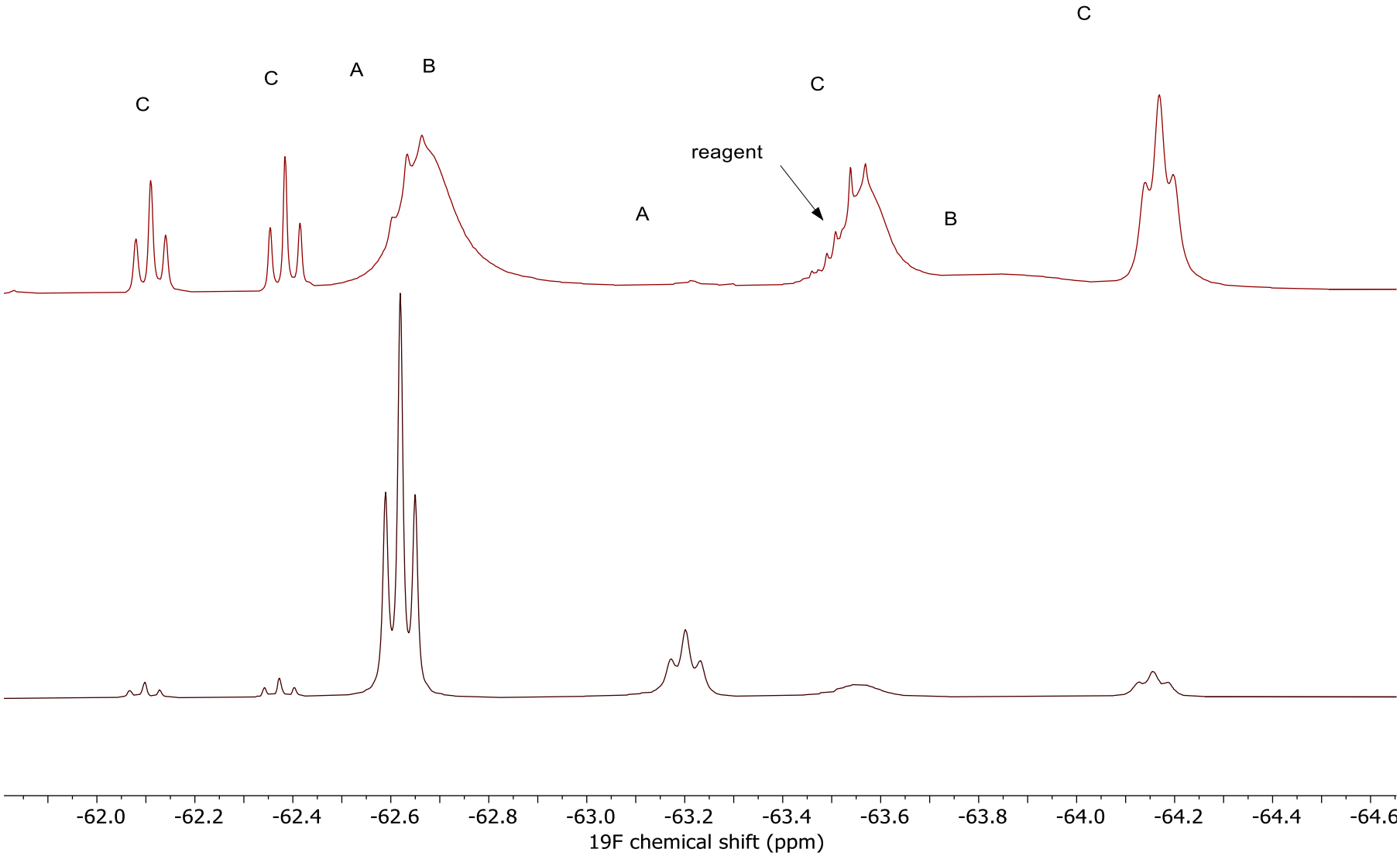
^{1}H-NMR (400 MHz) Spectrum of 11-Pd in CD_2Cl_2, 25 °C



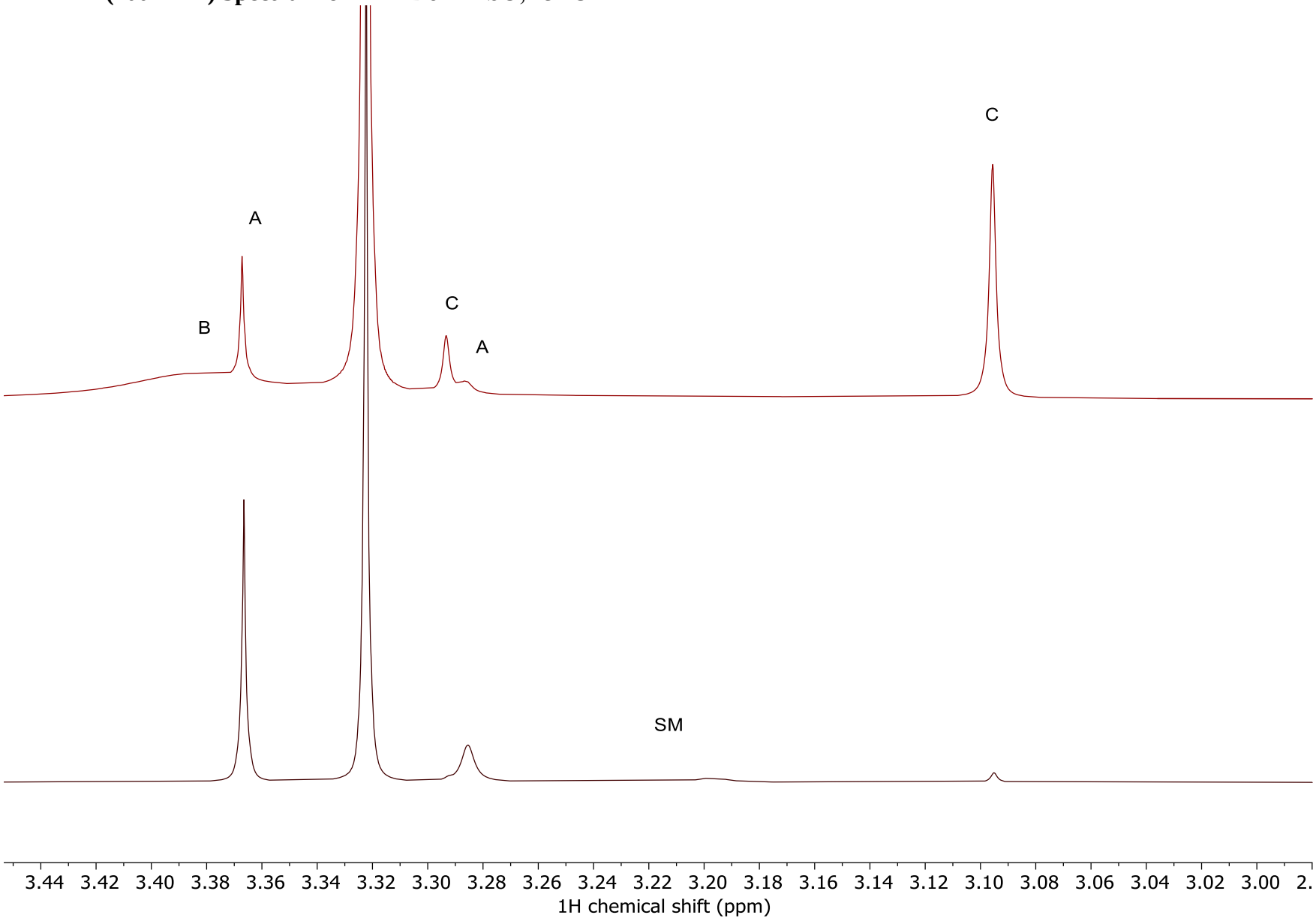
^{13}\{H\}C-NMR (100 MHz) Spectrum of 11-Pd in CD_2Cl_2, 25 °C



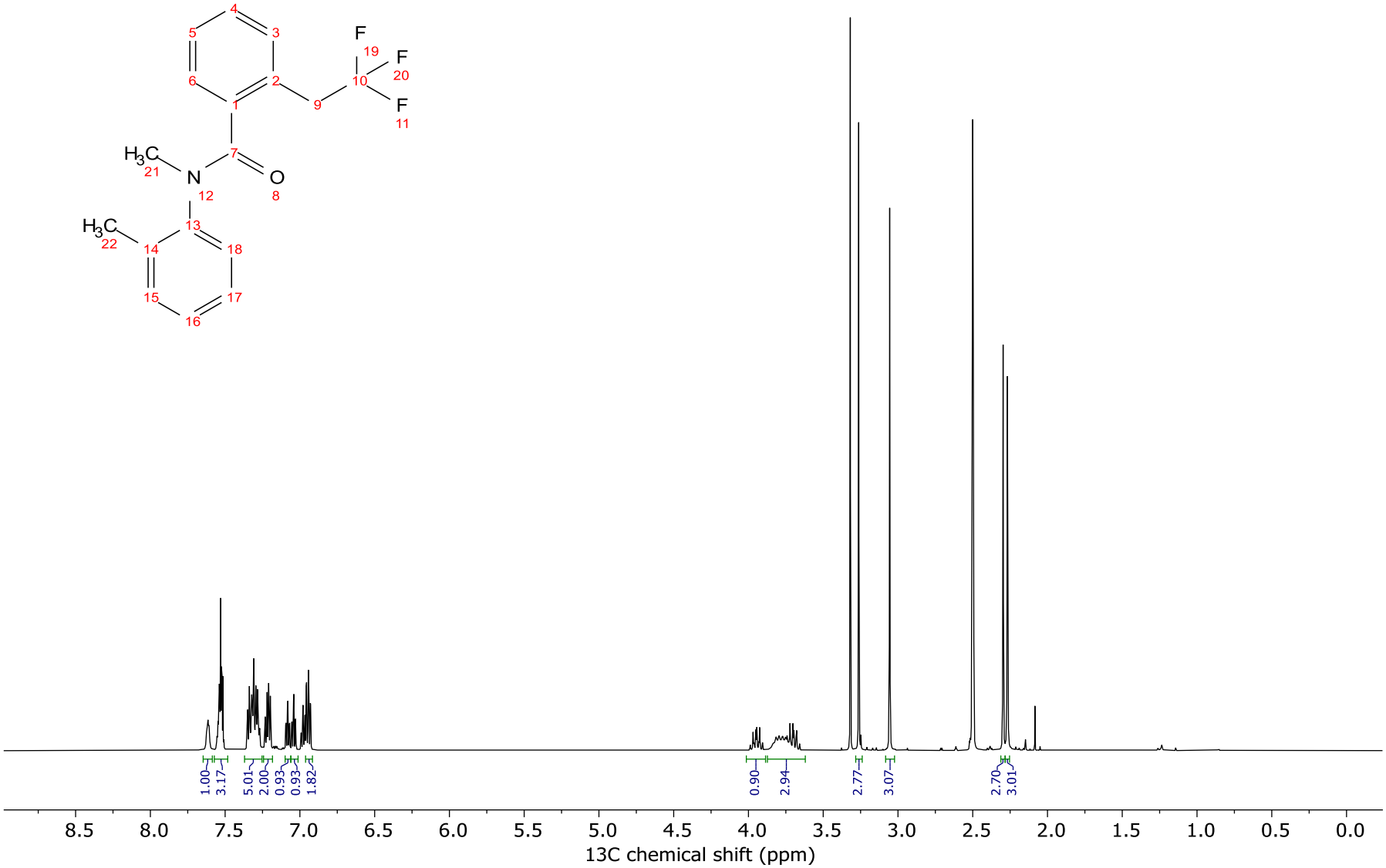
^{1}H-NMR (400 MHz) Spectrum of 12 in D6-DMSO, 25 °C



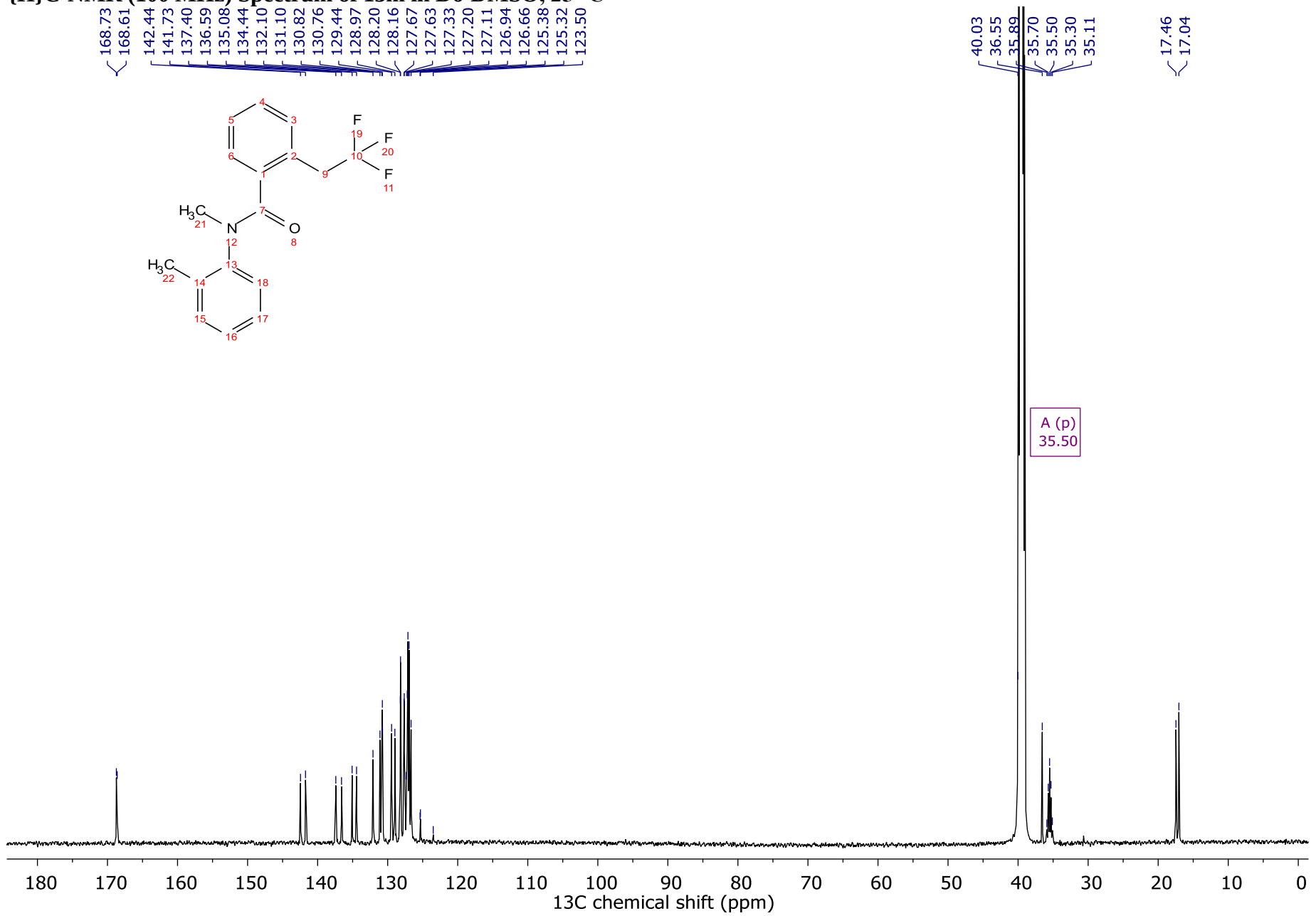
^{19}F-NMR (400 MHz) Spectrum of 12 in D6-DMSO, 25 °C



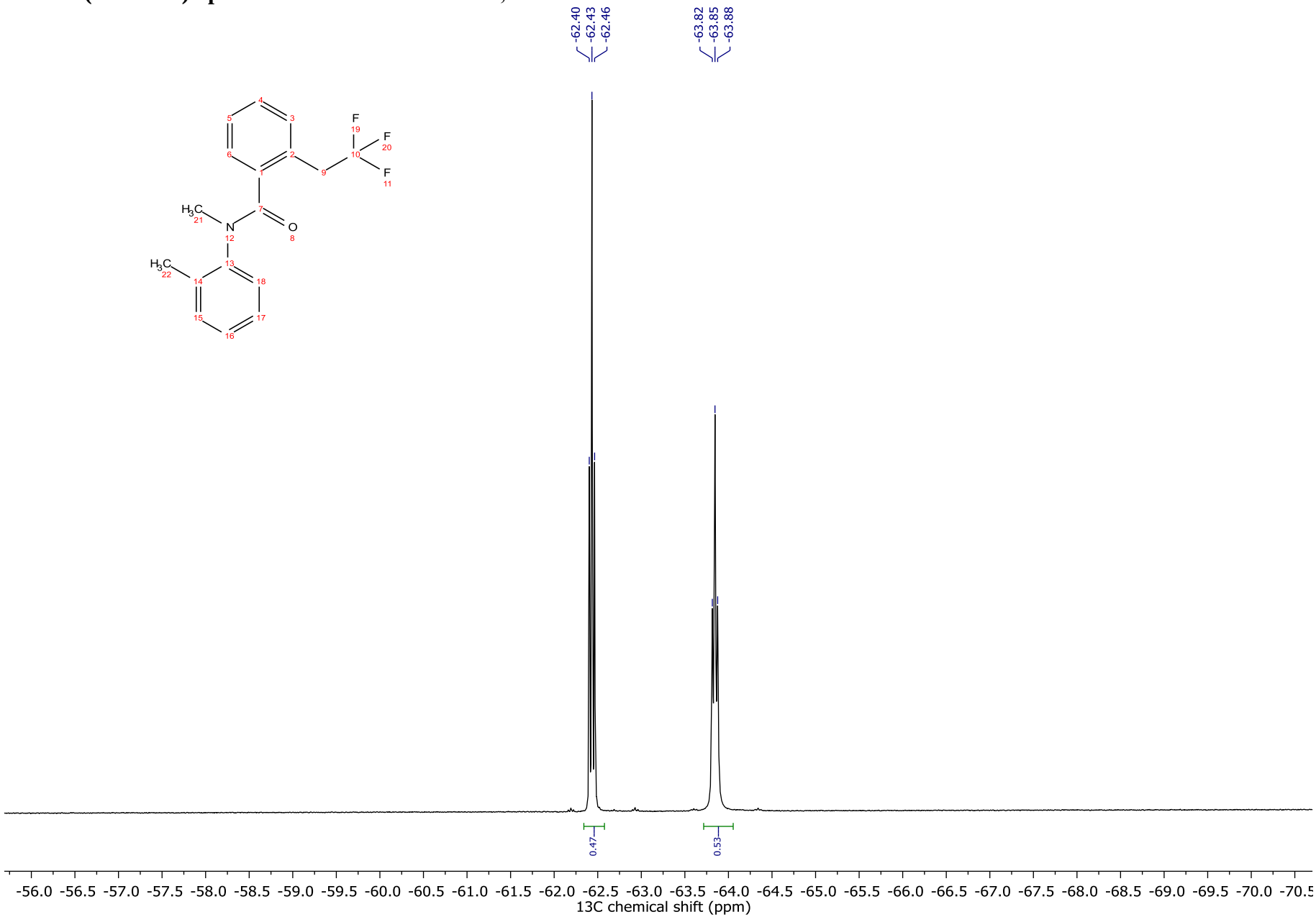
[1]H-NMR (376 MHz) Spectrum of 13m in D6-DMSO, 25 °C



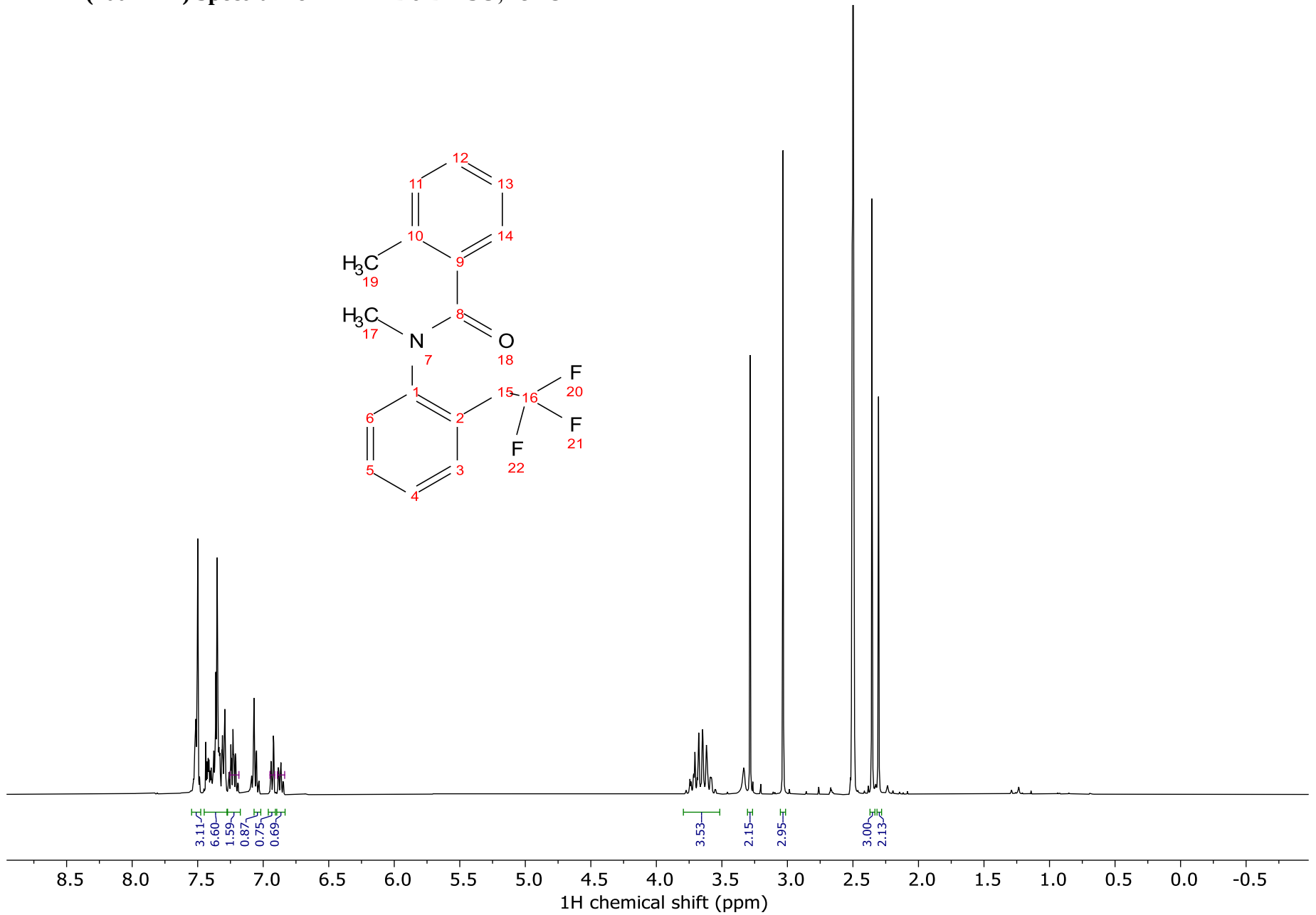
^{13}\{H\}C-NMR (100 MHz) Spectrum of 13m in D6-DMSO, 25 °C



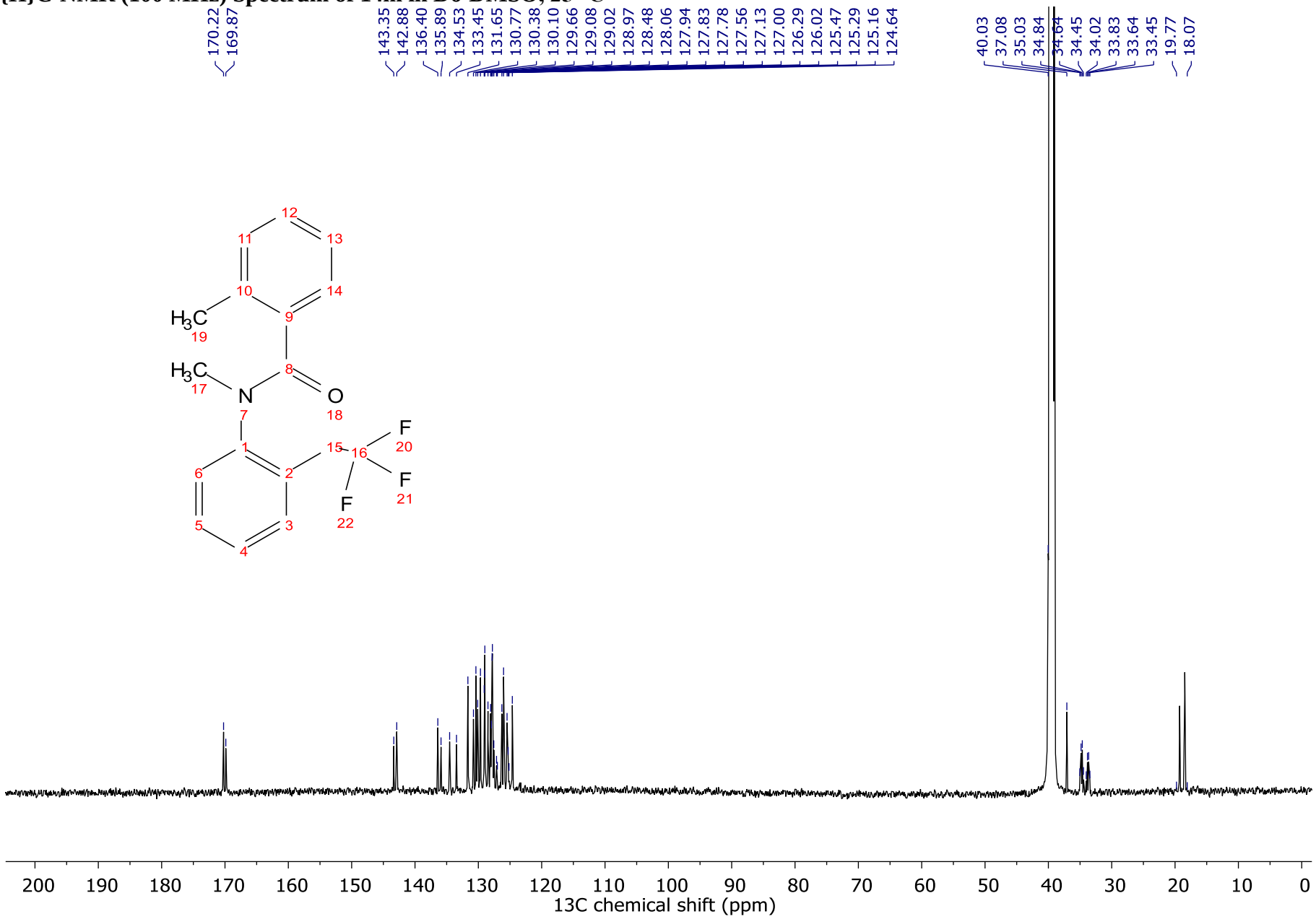
^{19}F-NMR (376 MHz) Spectrum of 13m in D6-DMSO, 25 °C



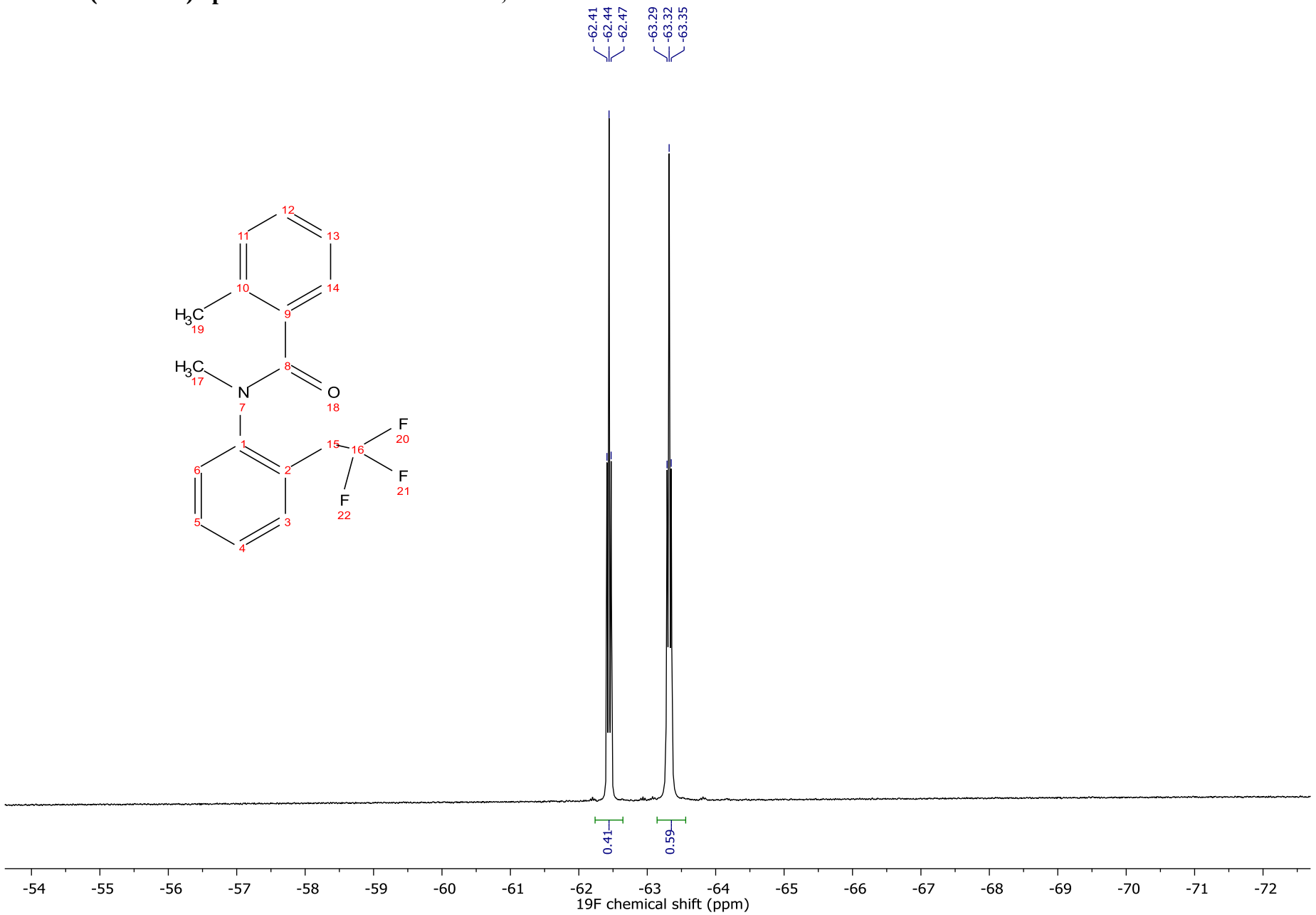
[1]H-NMR (400 MHz) Spectrum of 14m in D6-DMSO, 25 °C



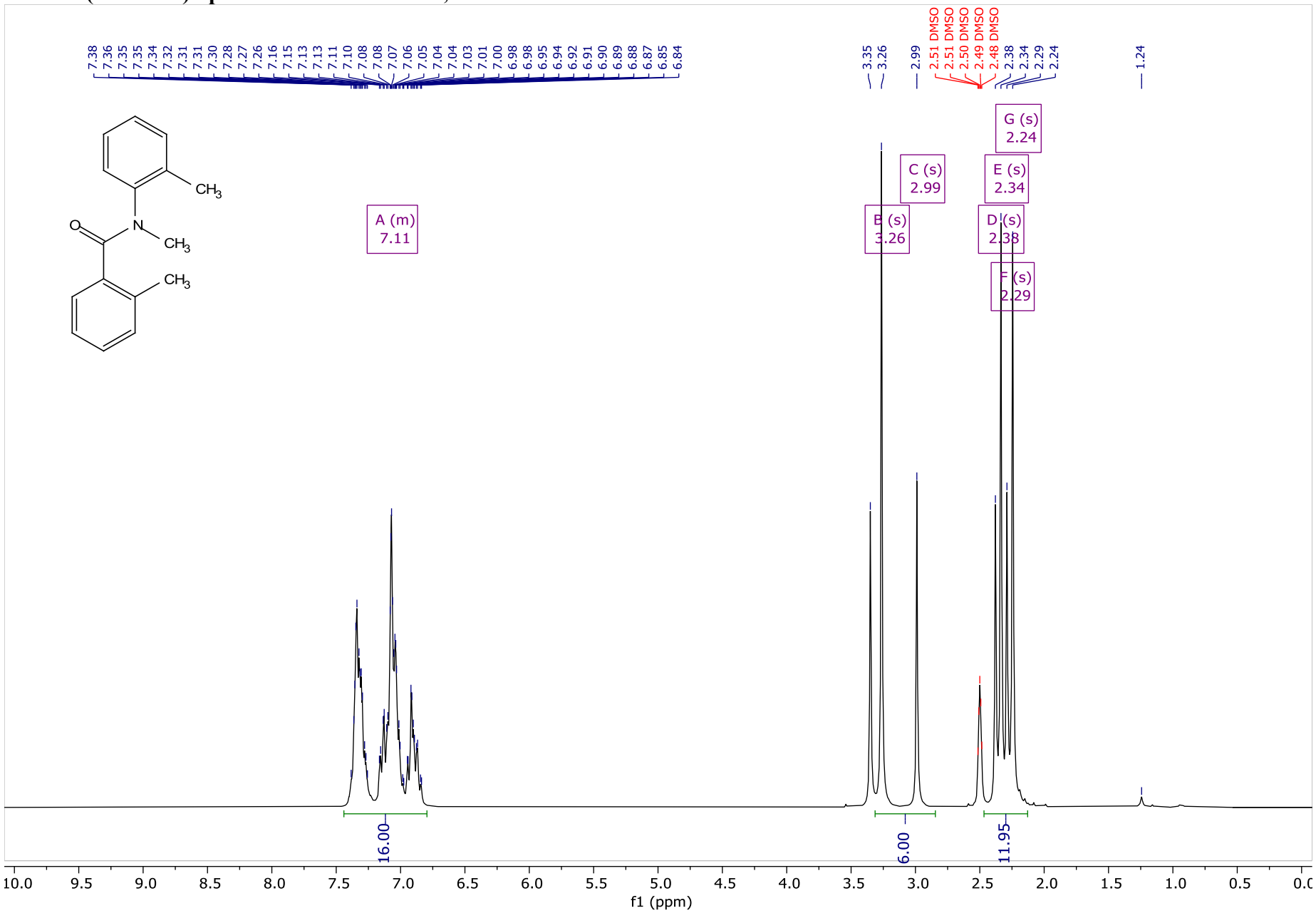
^{13}C{H}-NMR (100 MHz) Spectrum of 14m in D6-DMSO, 25 °C



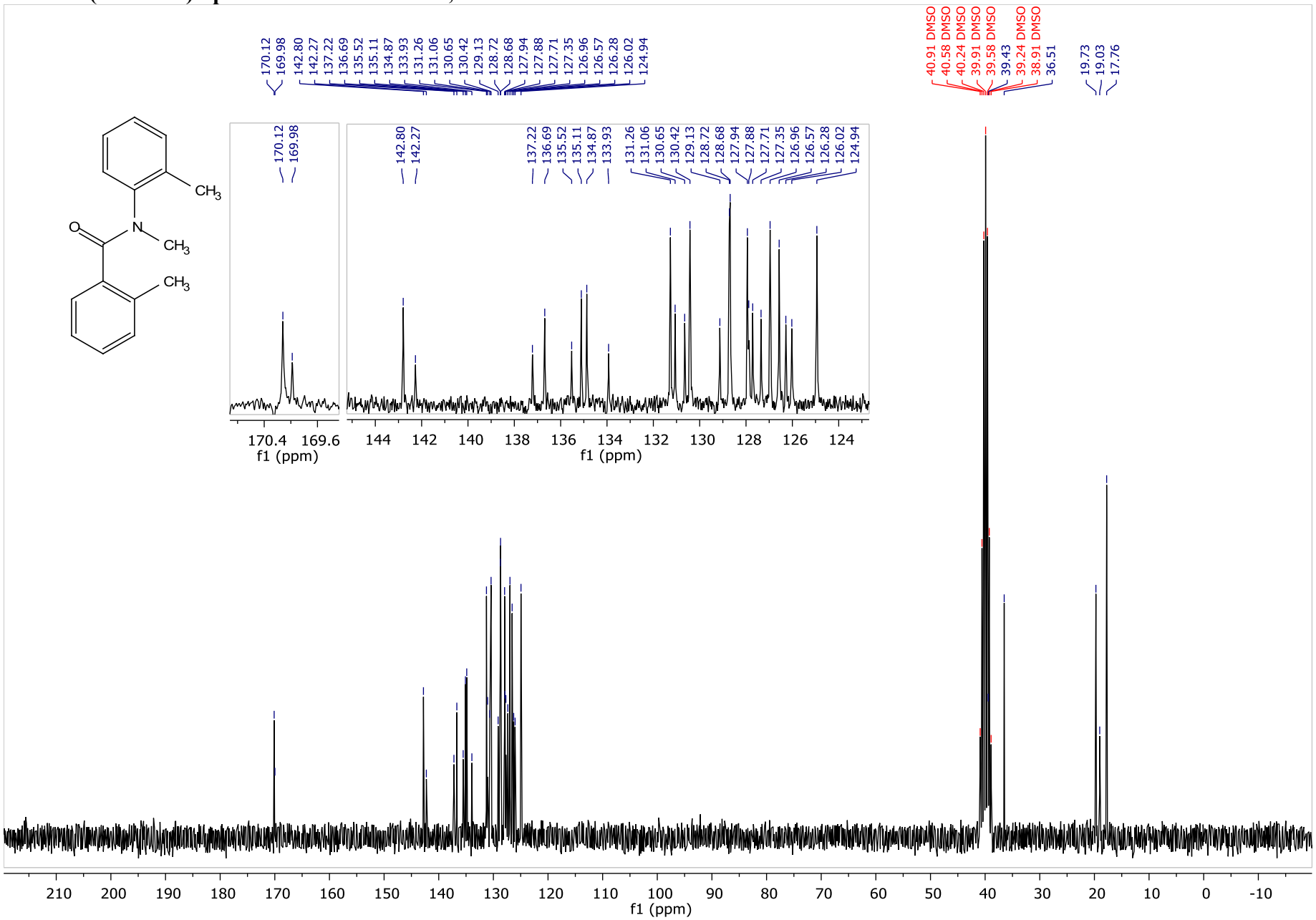
^{19}F-NMR (376 MHz) Spectrum of 14m in D6-DMSO, 25 °C



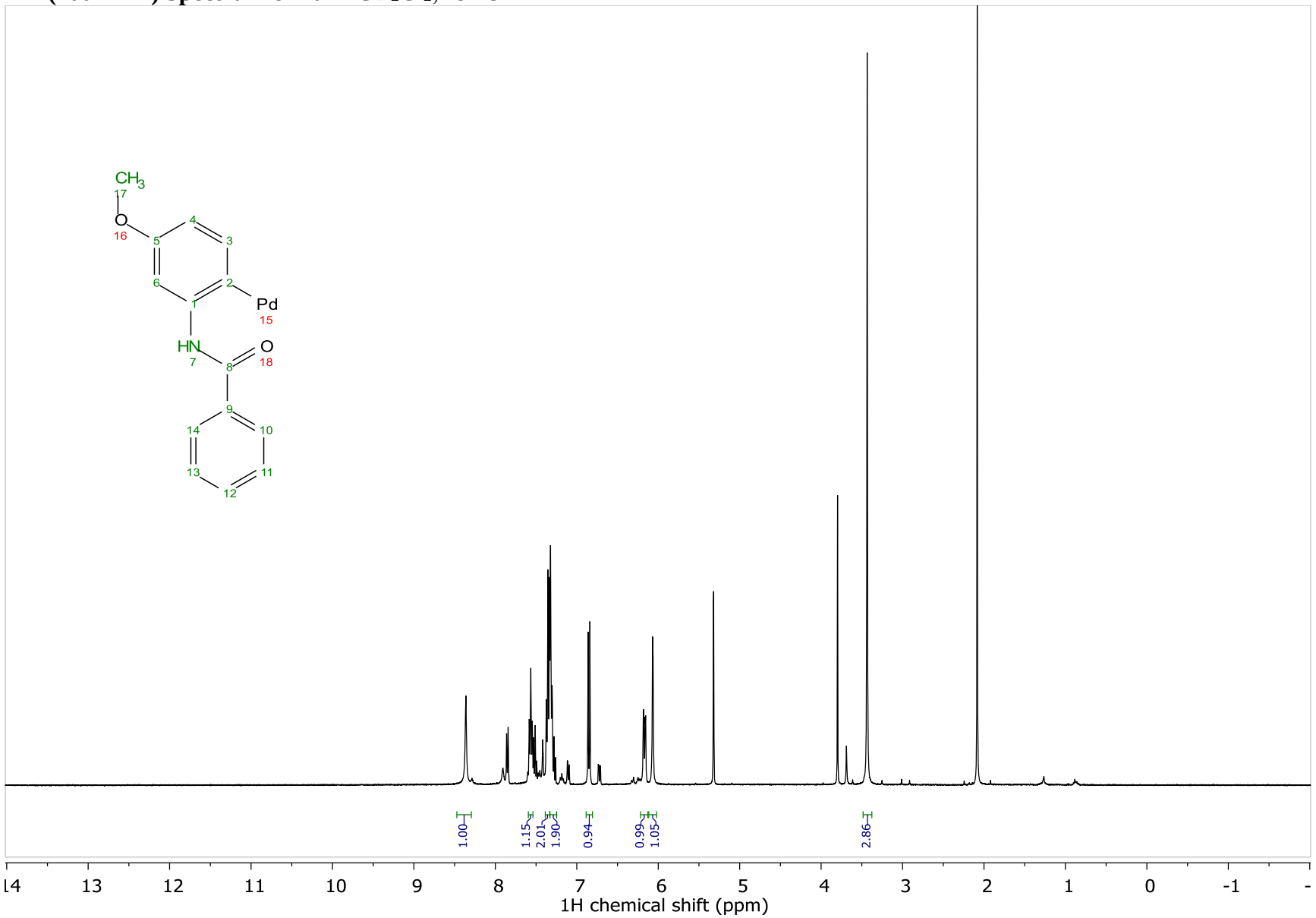
^{1}H-NMR (250 MHz) Spectrum of 31 in DMSO, 25 °C



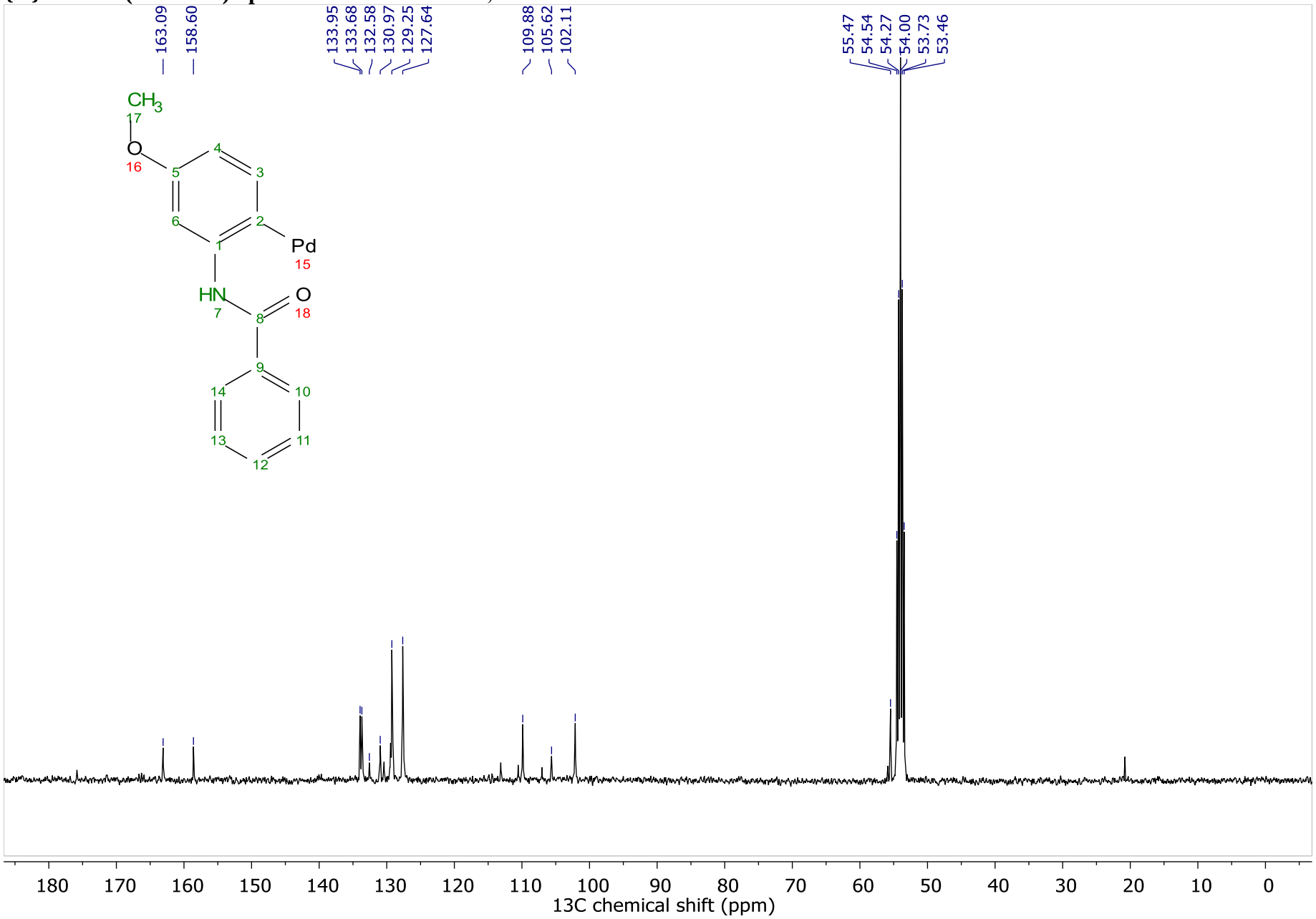
^{13}C-NMR (250 MHz) Spectrum of 31 in DMSO, 25 °C



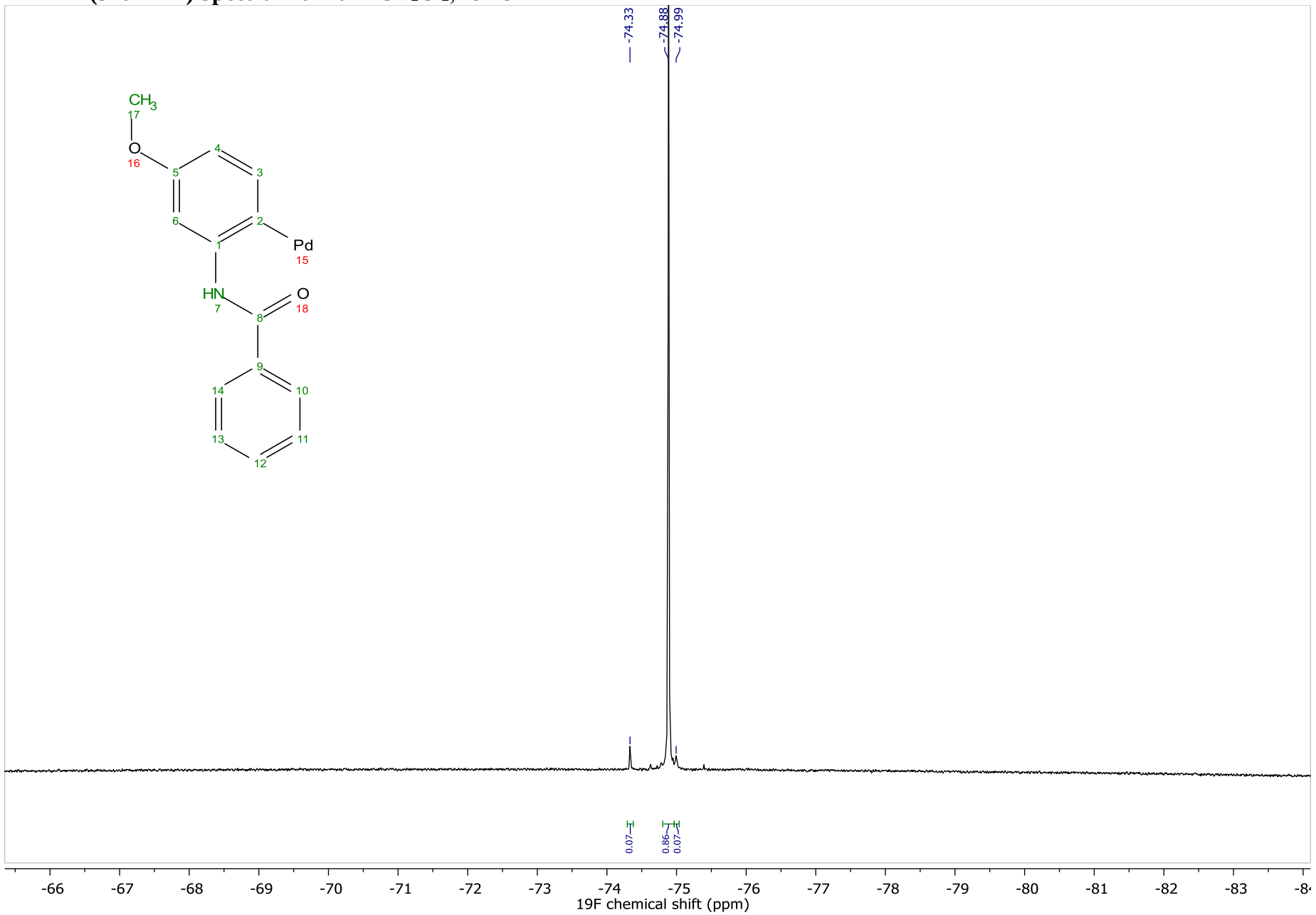
^{1}H-NMR (400 MHz) Spectrum of 40 in CD_2Cl_2, 25 °C



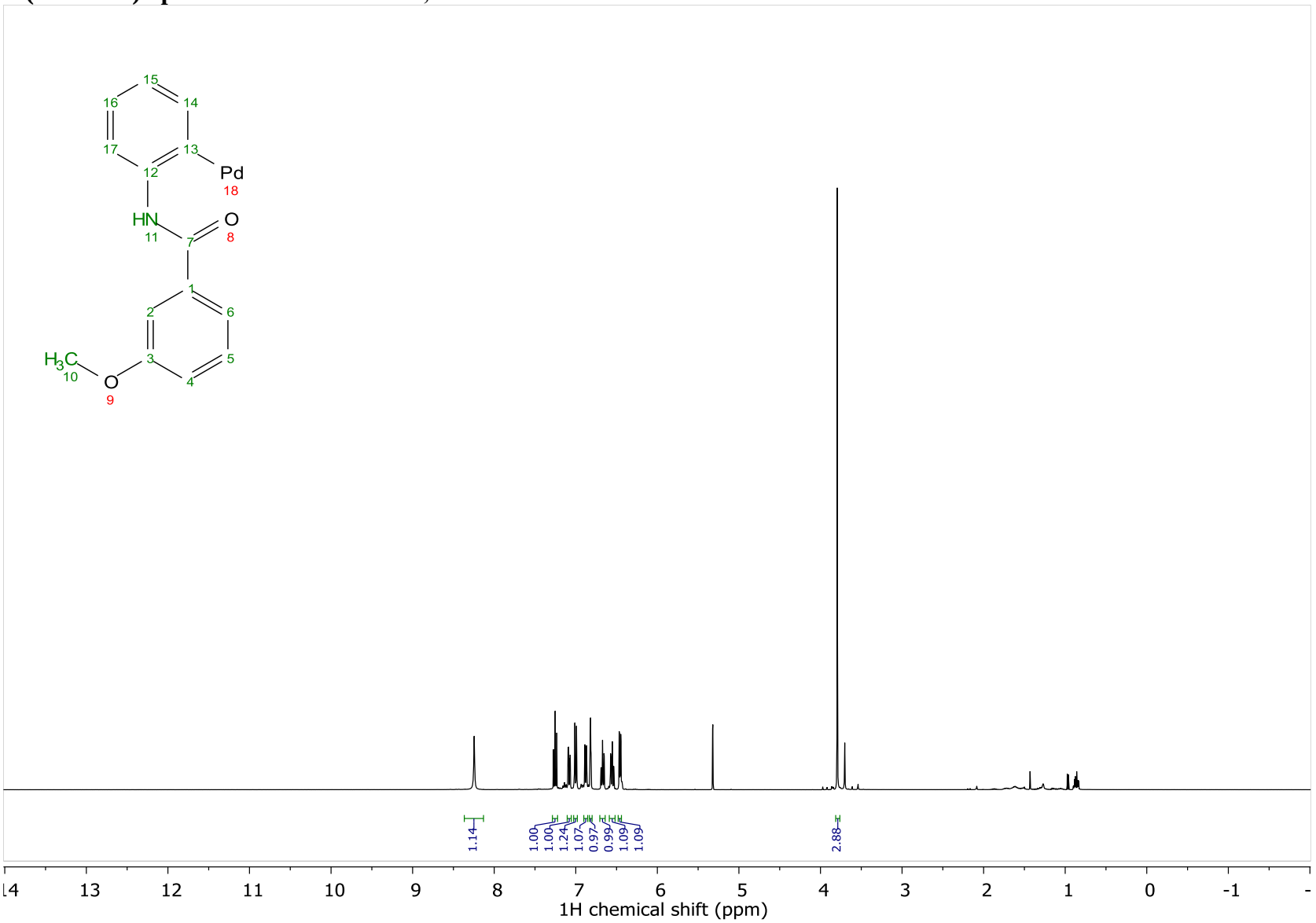
^{13}\{H\}C-NMR (100 MHz) Spectrum of 40 in CD_2Cl_2, 25 °C



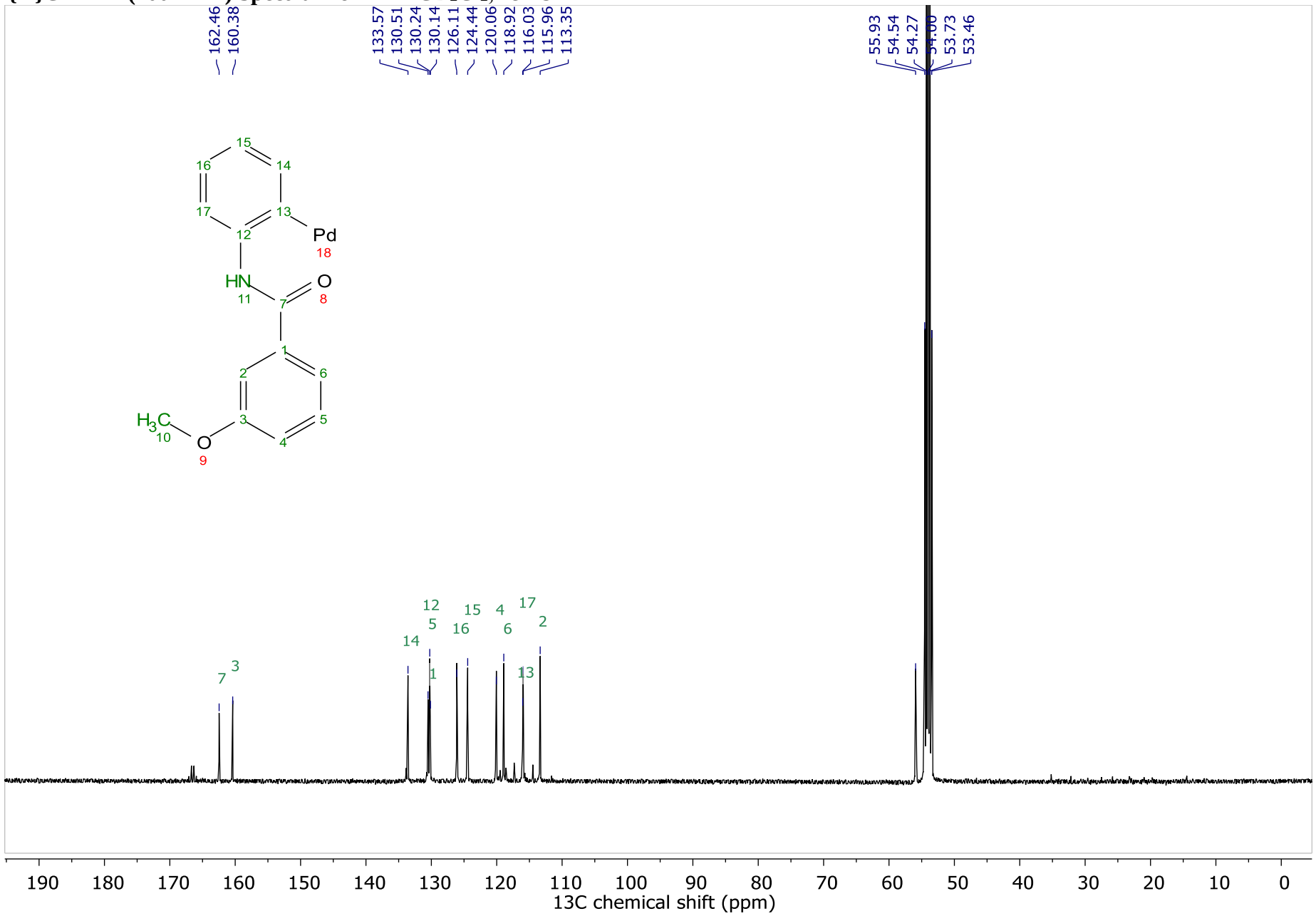
^{19}F-NMR (376 MHz) Spectrum of 40 in CD_2Cl_2, 25 °C



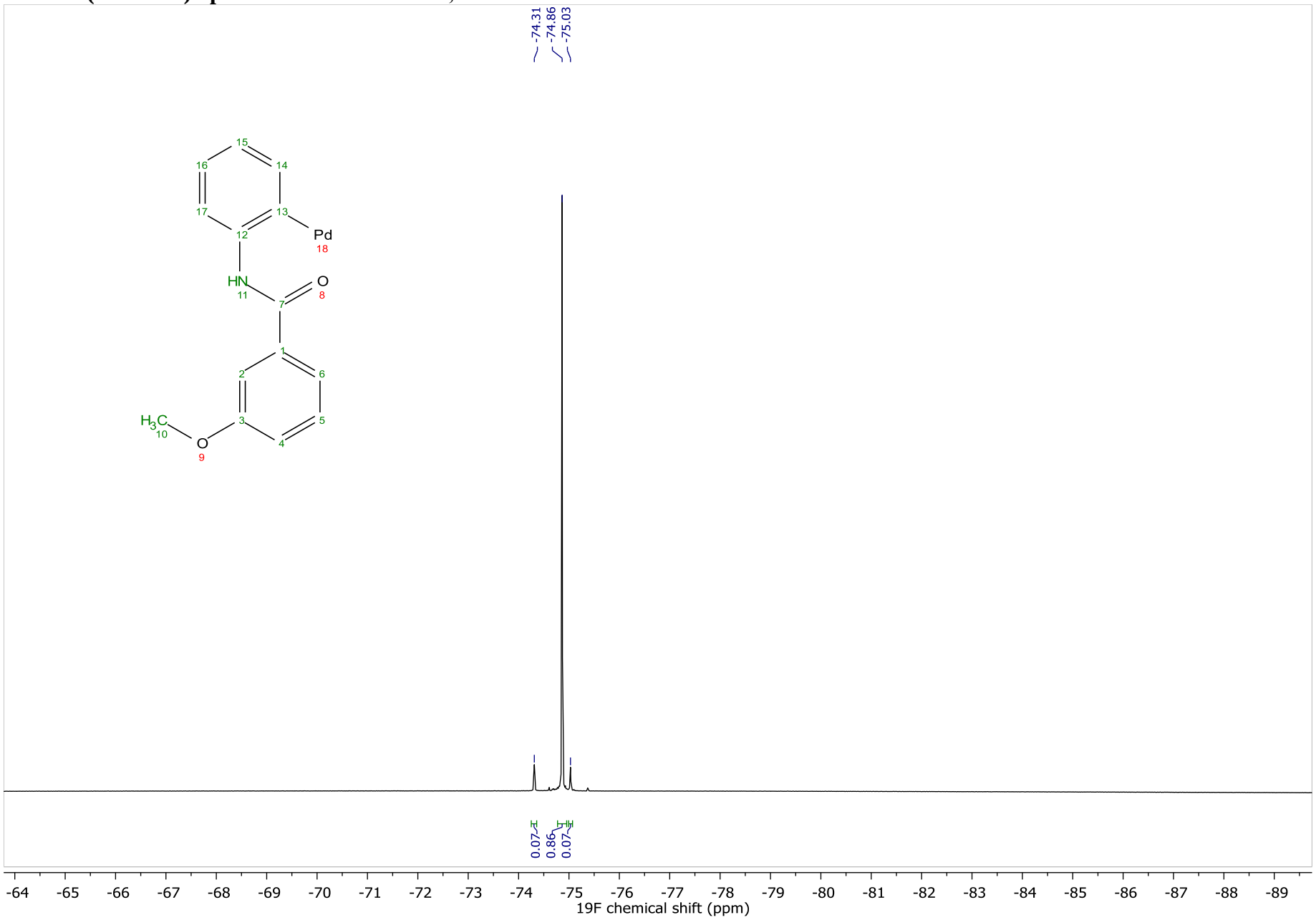
^{1}H-NMR (400 MHz) Spectrum of 41 in CD_2Cl_2, 25 °C



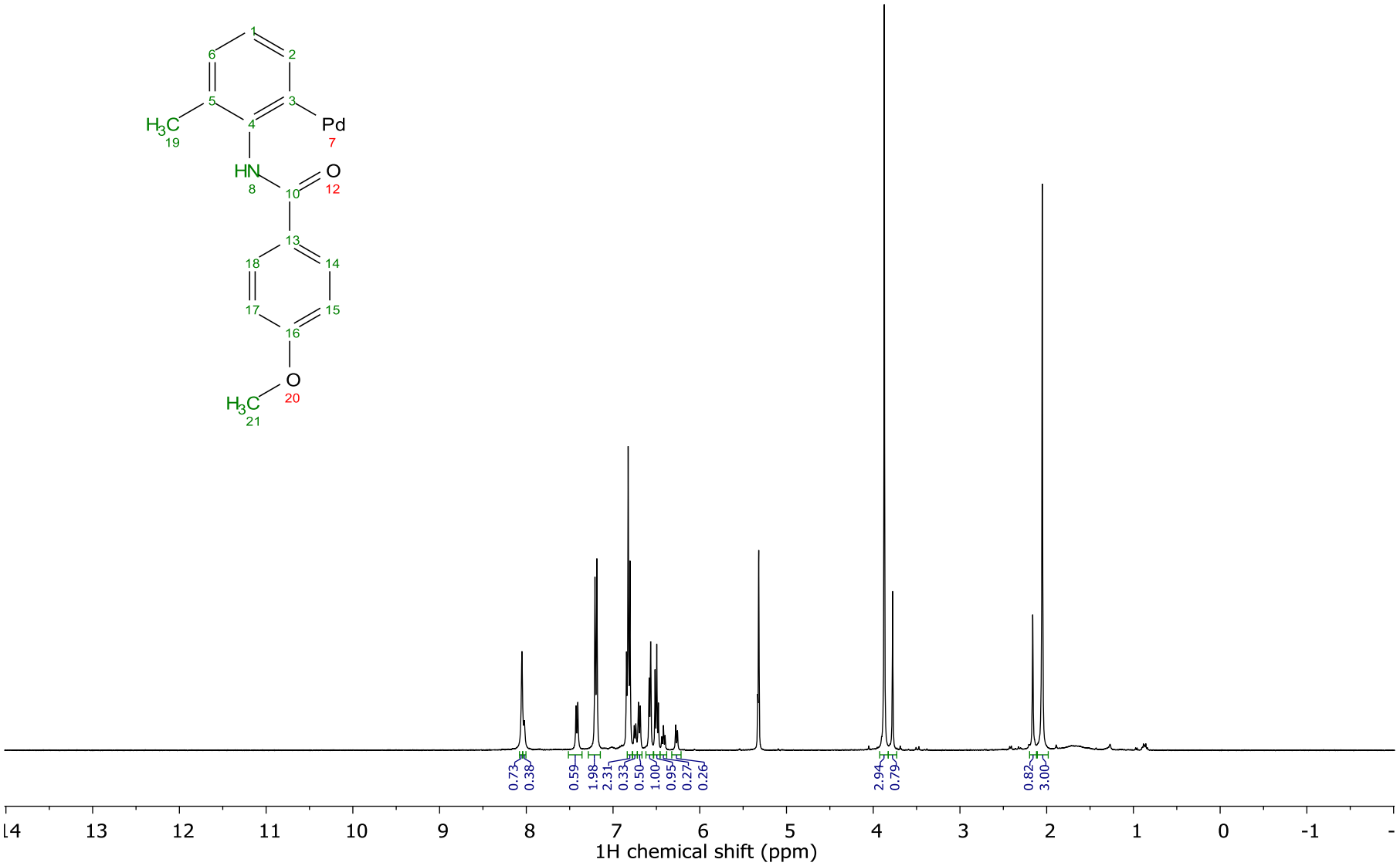
^{13}\{H\}C-NMR (100 MHz) Spectrum of 41 in CD_2Cl_2, 25 °C



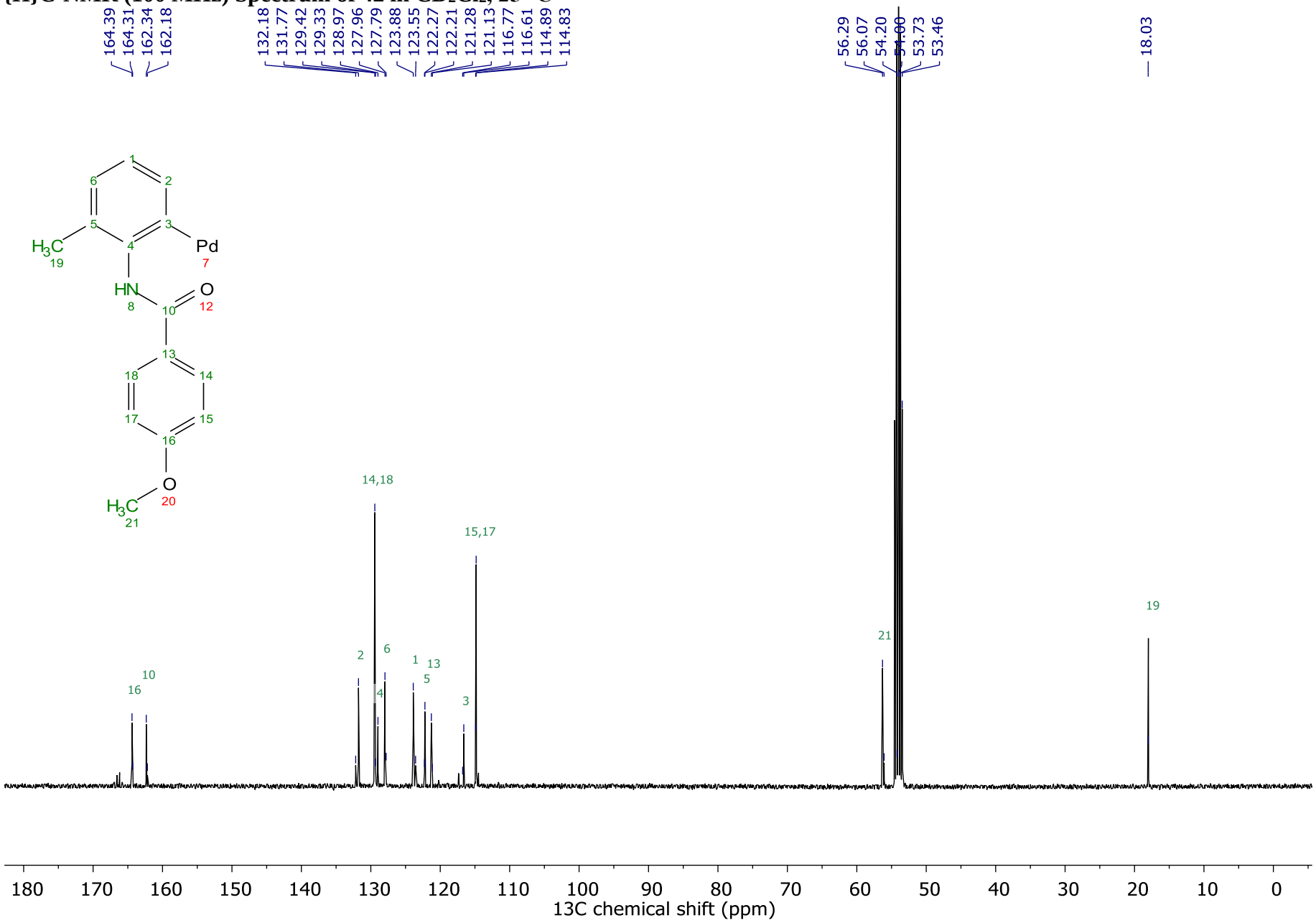
^{19}F-NMR (376 MHz) Spectrum of 41 in CD_2Cl_2, 25 °C



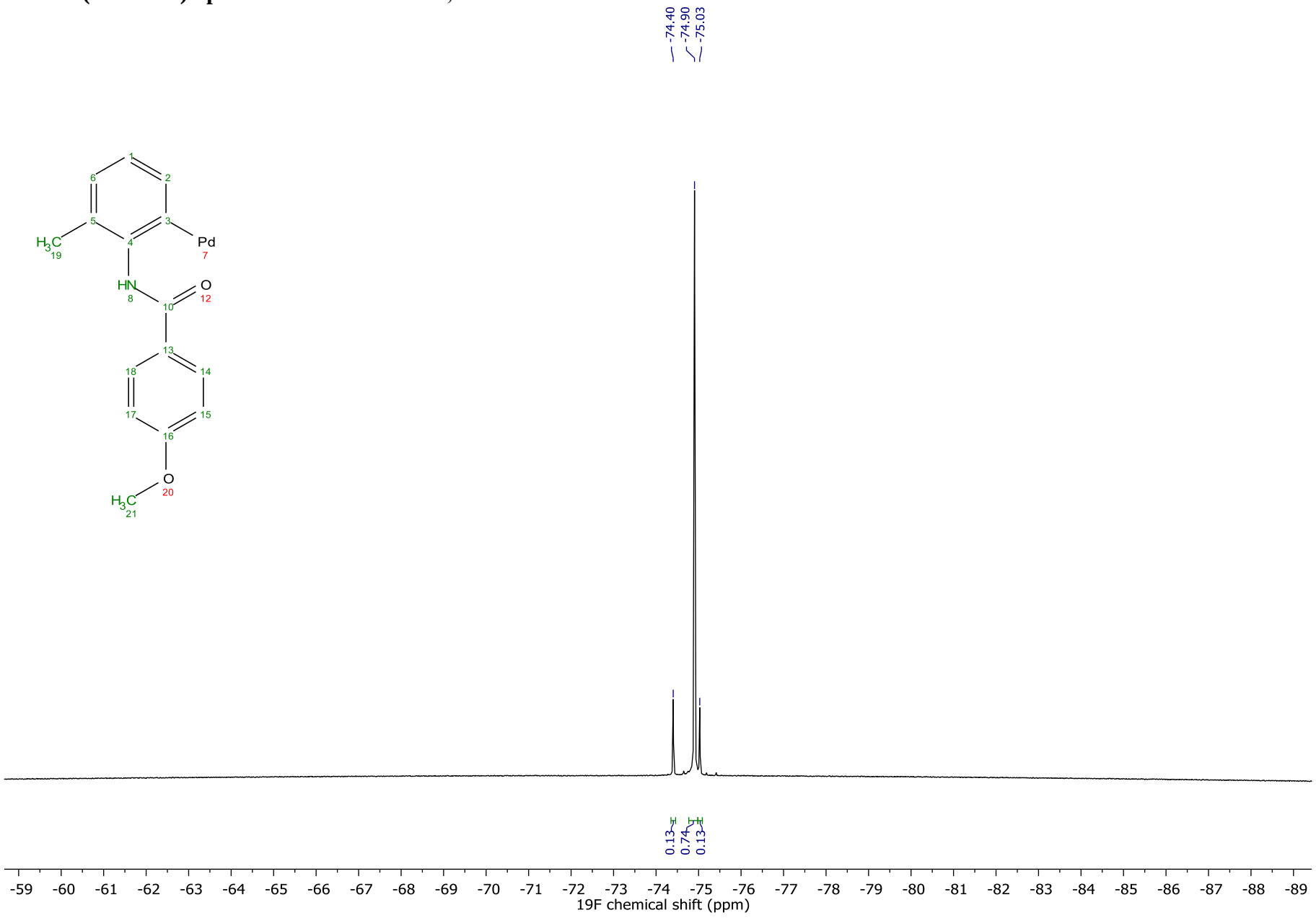
^{1}H-NMR (400 MHz) Spectrum of 42 in CD_2Cl_2, 25 °C



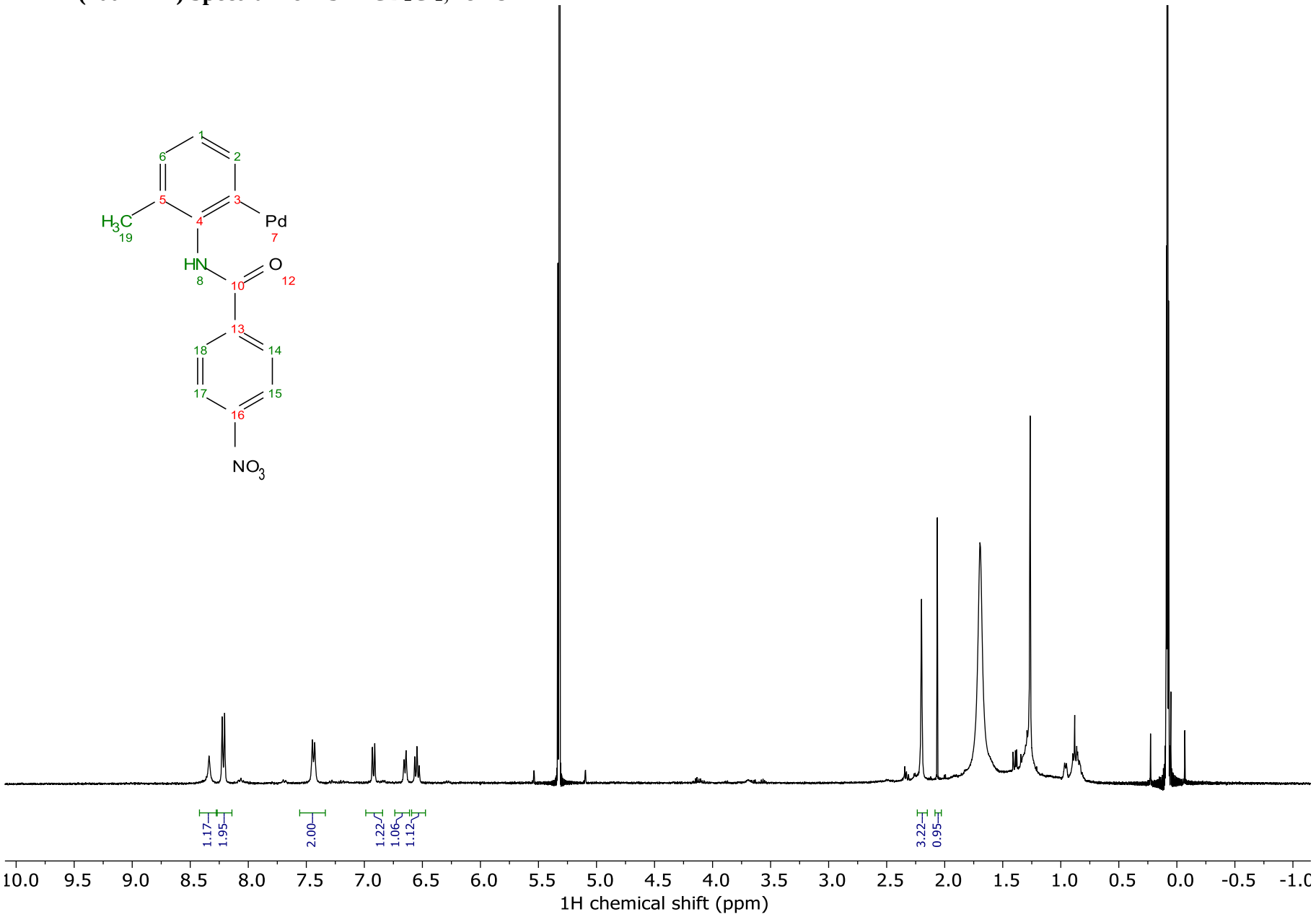
^{13}\{H\}C-NMR (100 MHz) Spectrum of 42 in CD_2Cl_2, 25 °C



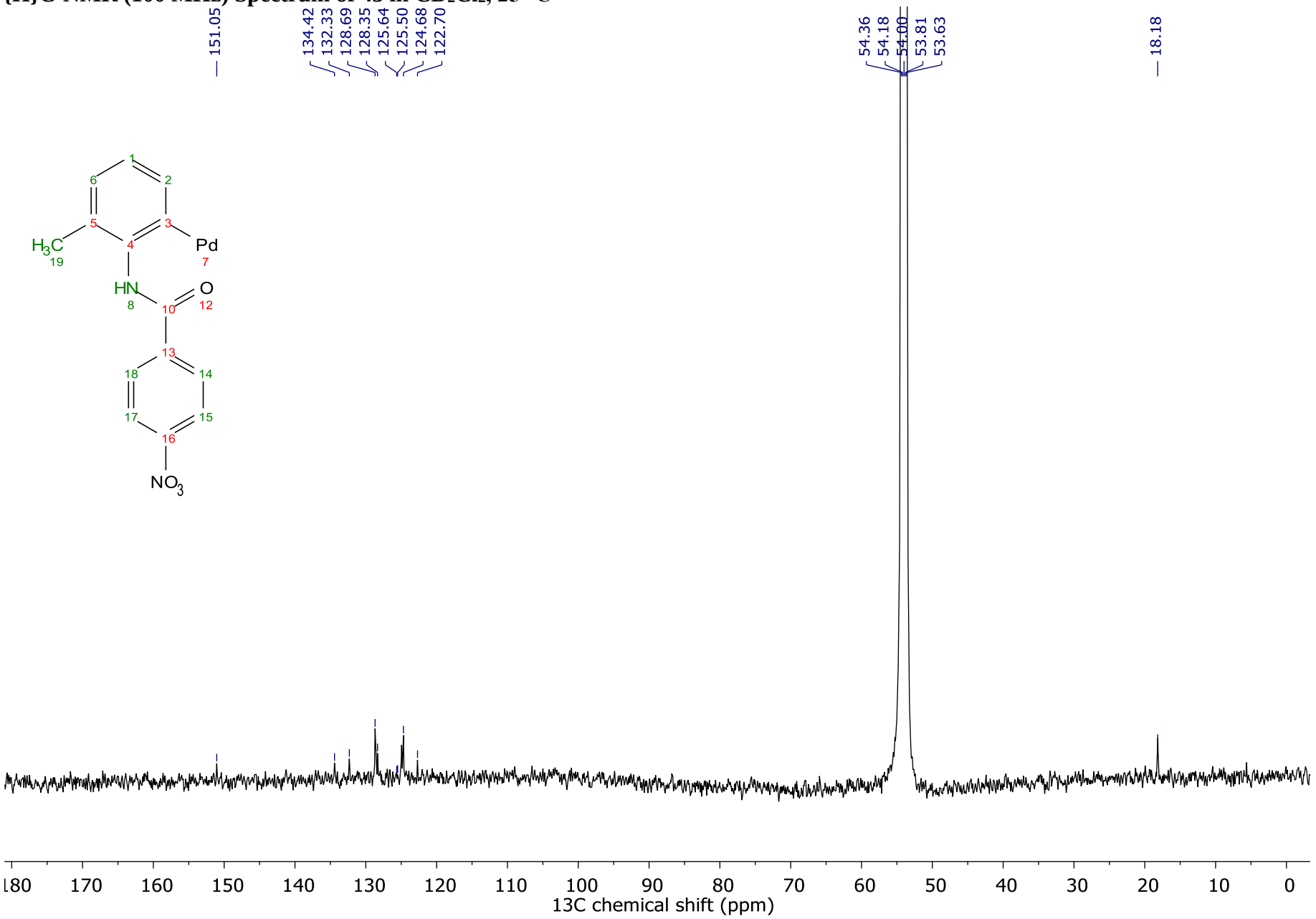
^{19}F-NMR (376 MHz) Spectrum of 42 in CD_2Cl_2, 25 °C



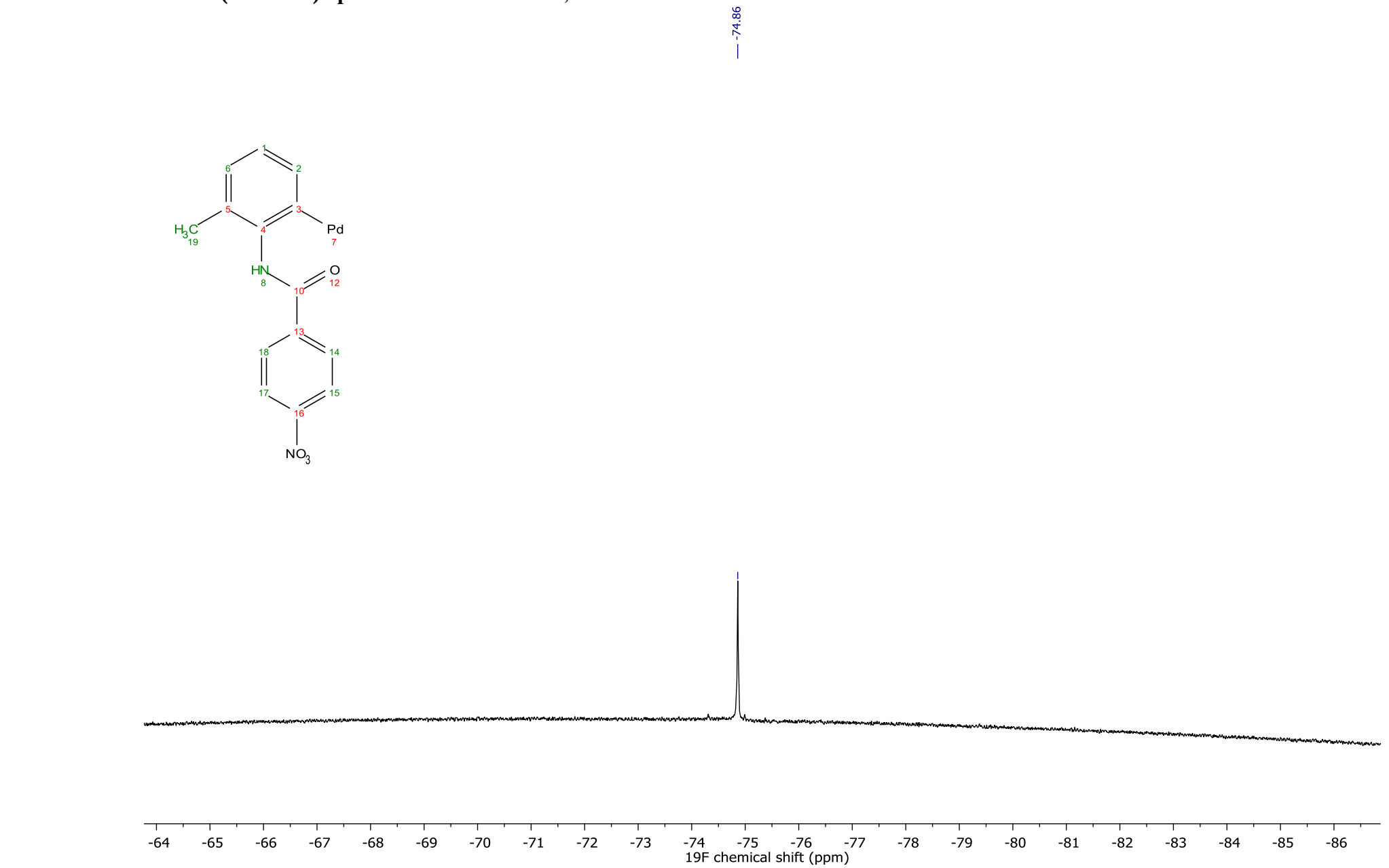
^{1}H-NMR (400 MHz) Spectrum of 43 in CD_2Cl_2, 25 °C



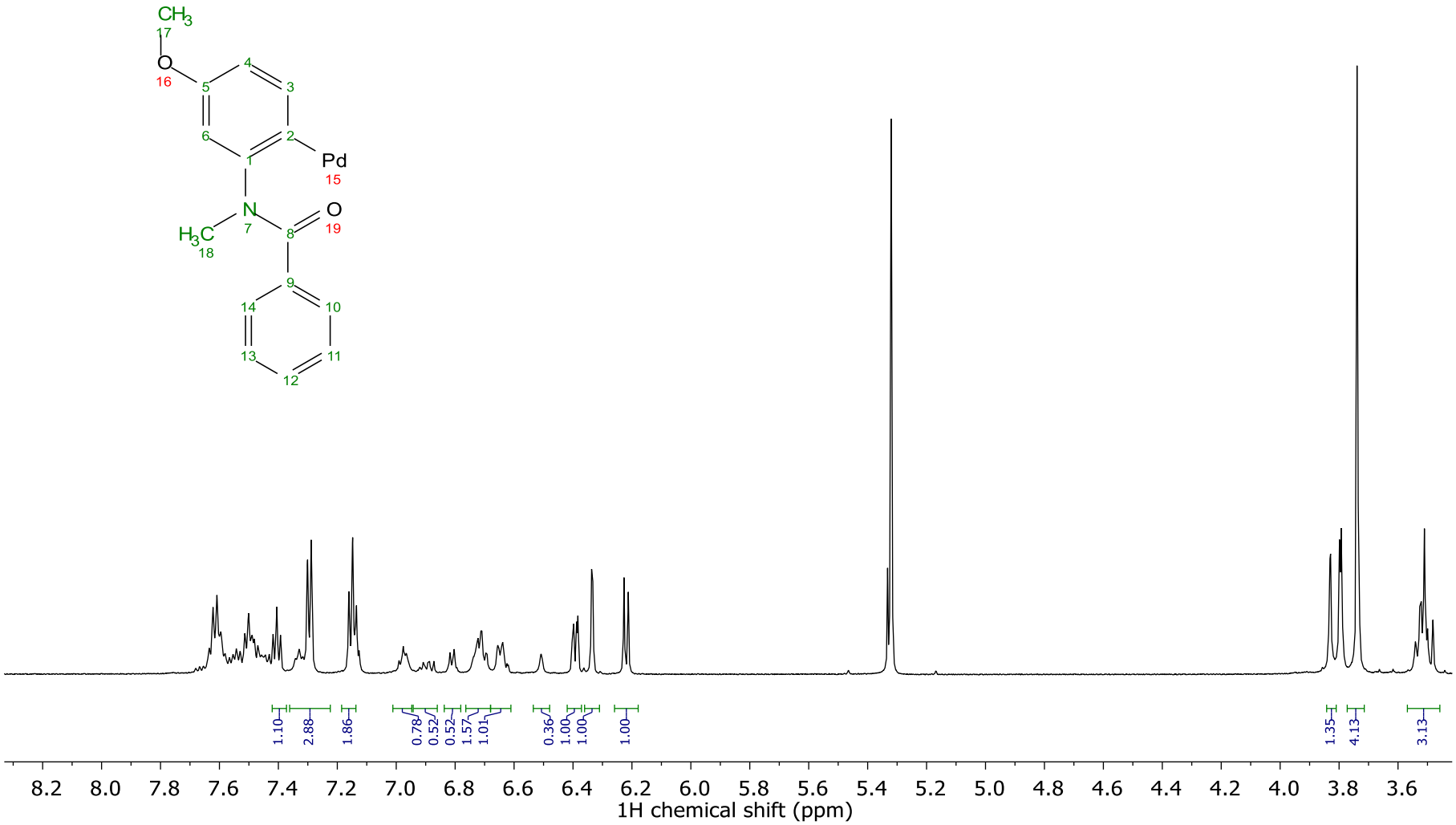
^{13}\{H\}C-NMR (100 MHz) Spectrum of 43 in CD_2Cl_2, 25 °C



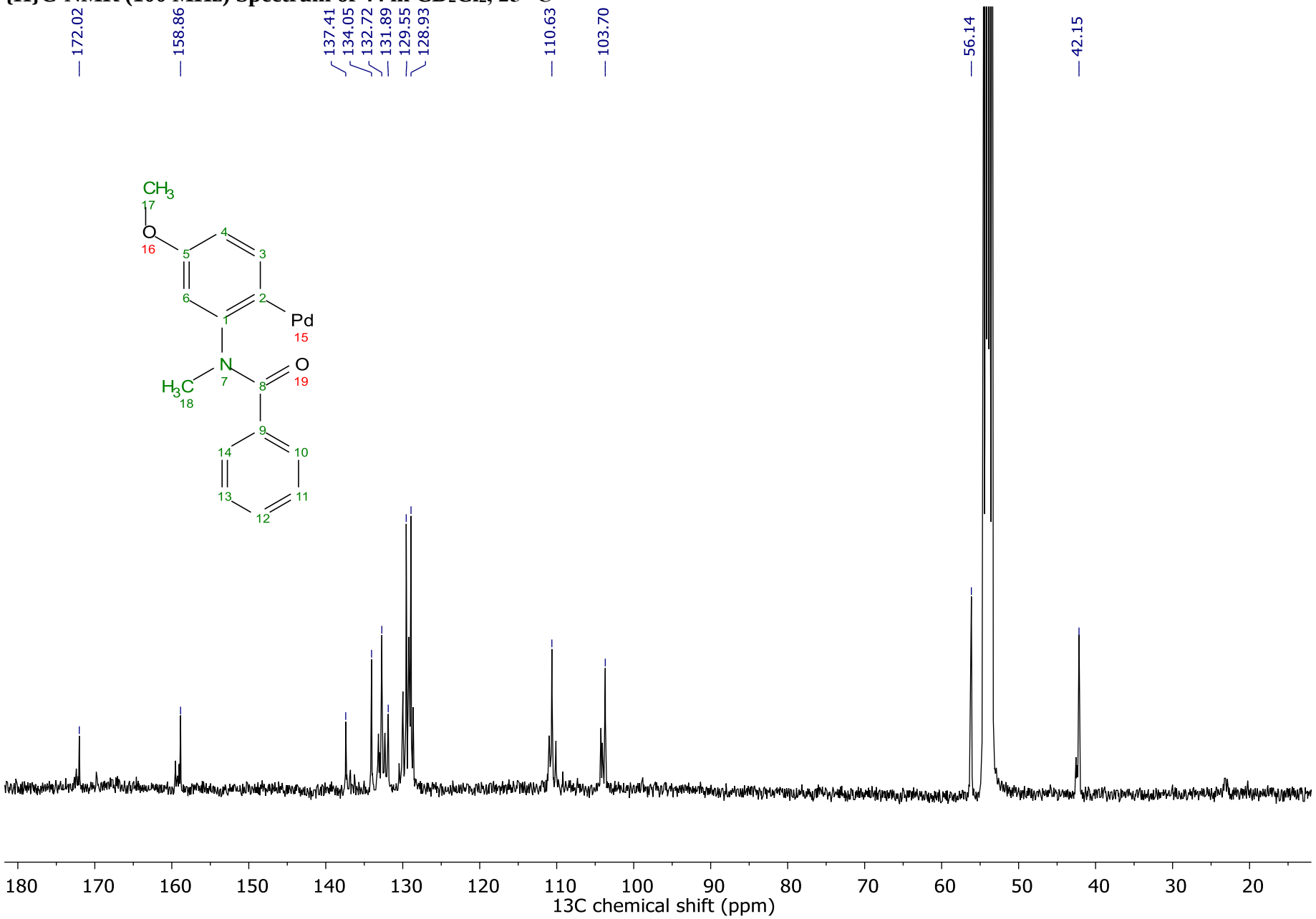
^{19}F-NMR (376 MHz) Spectrum of 43 in CD_2Cl_2, 25 °C



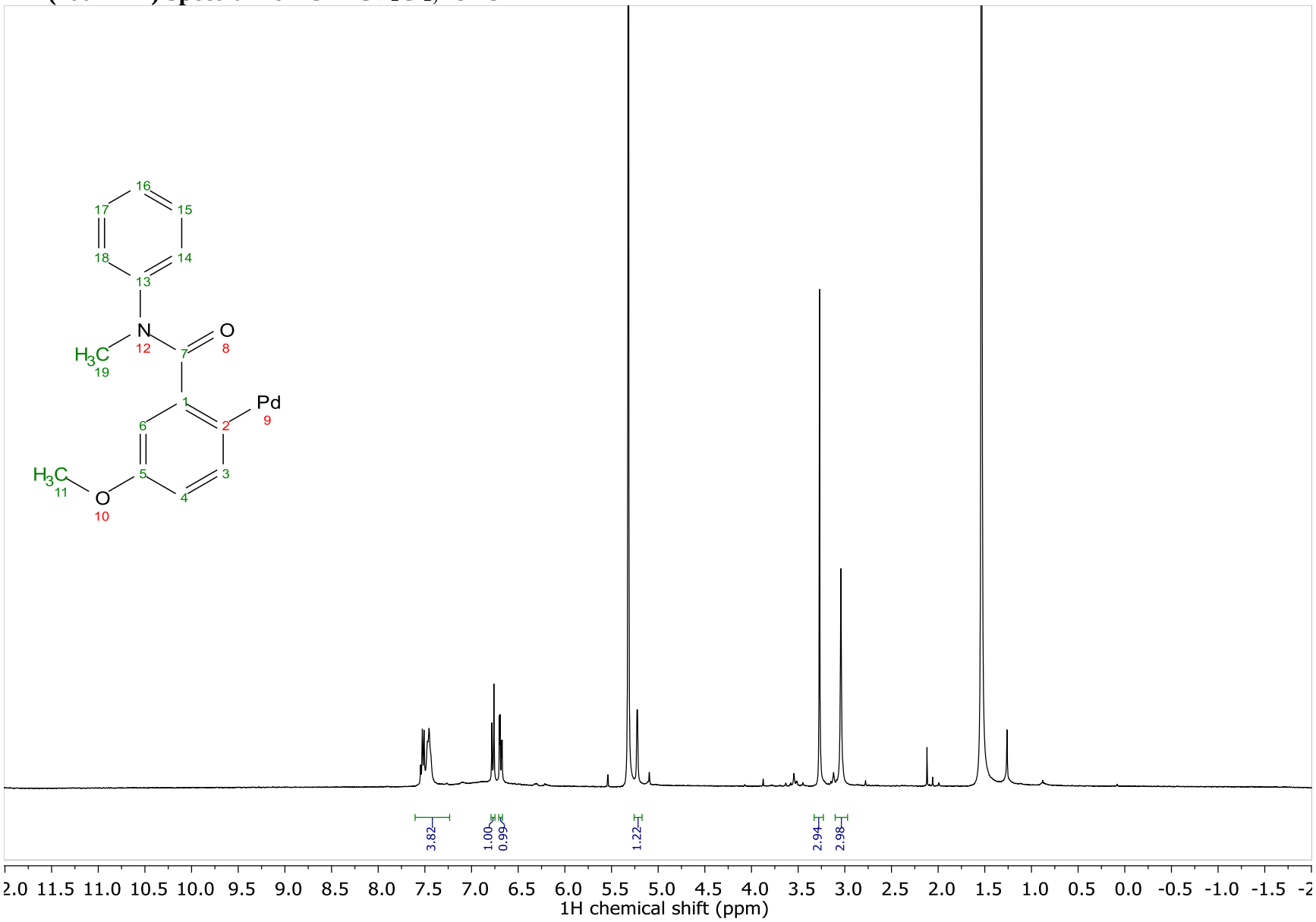
^{1}H-NMR (400 MHz) Spectrum of 44 in CD_2Cl_2, 25 °C



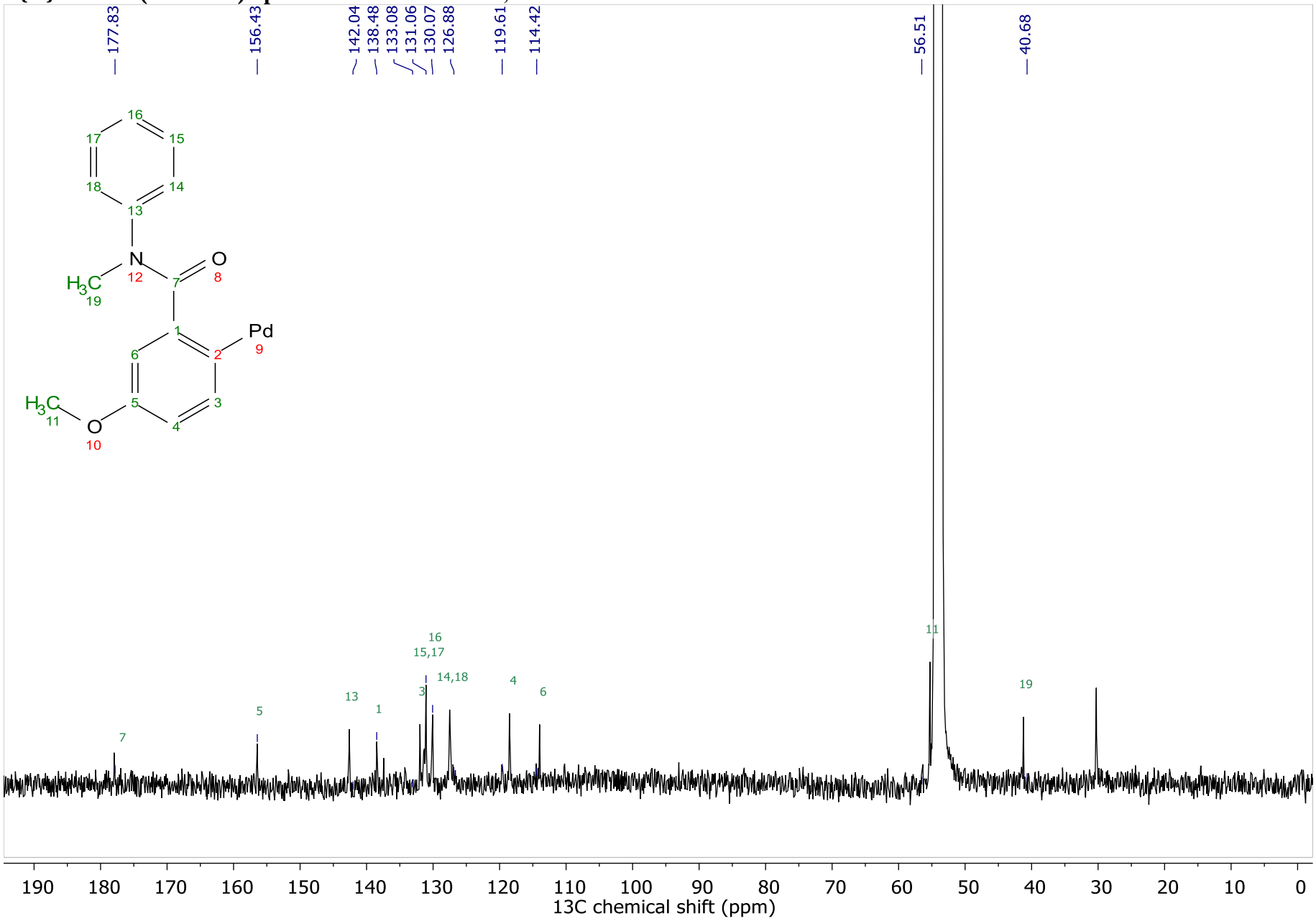
^{13}\{H\}C-NMR (100 MHz) Spectrum of 44 in CD_2Cl_2, 25 °C



^{1}H-NMR (400 MHz) Spectrum of 45 in CD_2Cl_2, 25 °C



^{13}\{H\}C-NMR (100 MHz) Spectrum of 45 in CD_2Cl_2, 25 °C



^{19}F-NMR (376 MHz) Spectrum of 45 in CD_2Cl_2, 25 °C