Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry.


# Supporting Information

Synthesis of 6,8-diaminopurines *via* Acid-induced Cascade Cyclization of 5-Aminoimidazole Precursors and Preliminary Anticancer Evaluation

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## 1 – Single-Crystal X-Ray Diffraction Studies

A single crystal of compound **3c.TFA** was manually harvested from the crystallization vial and immersed in highly viscous FOMBLIN Y perfluoropolyether vacuum oil (LVAC 140/13, Sigma-Aldrich) to avoid degradation caused by the evaporation of the solvent.[1] The crystal was mounted on a MiTeGen MicroLoop with the help of a Stemi 2000 stereomicroscope equipped with Carl Zeiss lenses.

Crystal data was collected at 180(2) K on a Bruker D8 QUEST equipped with Mo Kα sealed tube (λ = 0.71073 Å), a multilayer TRIUMPH X-ray mirror, a PHOTON III CMOS detector, and an Oxford Instruments Cryostrem 700+ Series low temperature device controlled by the APEX3 software package [2]and equipped with an Oxford Cryosystems Series 700 cryostream monitored remotely using the software interface Cryopad.[3] Diffraction images were processed using the software package SAINT+,[4] and data were corrected for absorption by the multiscan semi-empirical method implemented in SADABS 2016/2. [5]

The structure was solved using the algorithm implemented in SHELXT-2014/5,[6] which allowed the immediate location of almost all the heaviest atoms composing their molecular unit. The remaining missing and misplaced non-hydrogen atoms were located from difference Fourier maps calculated from successive full-matrix least-squares refinement cycles on $F^2$ using the latest SHELXL from the 2018/3 release.[6] All structural refinements were performed using the graphical interface ShelXle.[7]

Hydrogen atoms bound to carbon were placed at their idealized positions using *HFIX* instructions in SHELXL: *43* (for aromatic carbon atoms), *23* (for the $–CH_2–$ groups) and *137* (for the $–CH_3$ groups). These hydrogen atoms were included in subsequent refinement cycles with isotropic thermal displacements parameters ($U_{iso}$) fixed at 1.2 or 1.5×$U_{eq}$ (solely for the $–CH_3$ groups) of the parent carbon atoms. Hydrogen atoms bound to nitrogen were directly located from difference Fourier maps and were included in the final structural model with the N–H distances restrained to 0.85(1) Å. Besides the charge-balancing $TFA^-$ anions the crystal structure also contains two solvent molecules, namely a DMSO and a water molecule.

The last difference Fourier map synthesis showed the highest peak (1.224 $eÅ^{-3}$) and the deepest hole (-0.703 $eÅ^{-3}$) located at 0.72 and 0.59 Å from F3' and F5, respectively. All structural drawings have been created using the software package Crystal Impact Diamond.[8]

Crystallographic data (including structure factors) for the two crystals structures have been deposited with the Cambridge Crystallographic Data Centre. Copies of the data can be obtained free of charge on application to CCDC, 12 Union Road, Cambridge CB2 2EZ, U.K. FAX: (+44) 1223 336033. E-mail: deposit@ccdc.cam.ac.uk.

*Crystal data for* **3c.TFA**: $C_{44}H_{46}F_6N_{12}O_6S$, $M$ = 984.99, triclinic, space group $P$-1, $Z$ = 2, $a$ = 12.7714(17) Å , $b$ = 13.5757(17) Å, $c$ = 14.5009(19) Å, $a$ = 100.594(4)º, $b$ = 108.280(4)º, $g$ = 94.426(4)º , $V$ = 2322.0(5) $Å^3$, $\mu$(Mo-Kα) = 0.156 $mm^{-1}$, colourless needle with crystal size of 0.30×0.10×0.02 $mm^3$. Of a total of 59622

reflections collected, 8466 were independent ($R_{int}$ = 0.0585). Final $R1$ = 0.0857 [$I > 2\sigma(I)$] and $wR2$ = 0.2585 (all data). Data completeness to theta = 25.24°, 99.4%. CCDC2287378.

## 2 - NMR spectra

**2e**

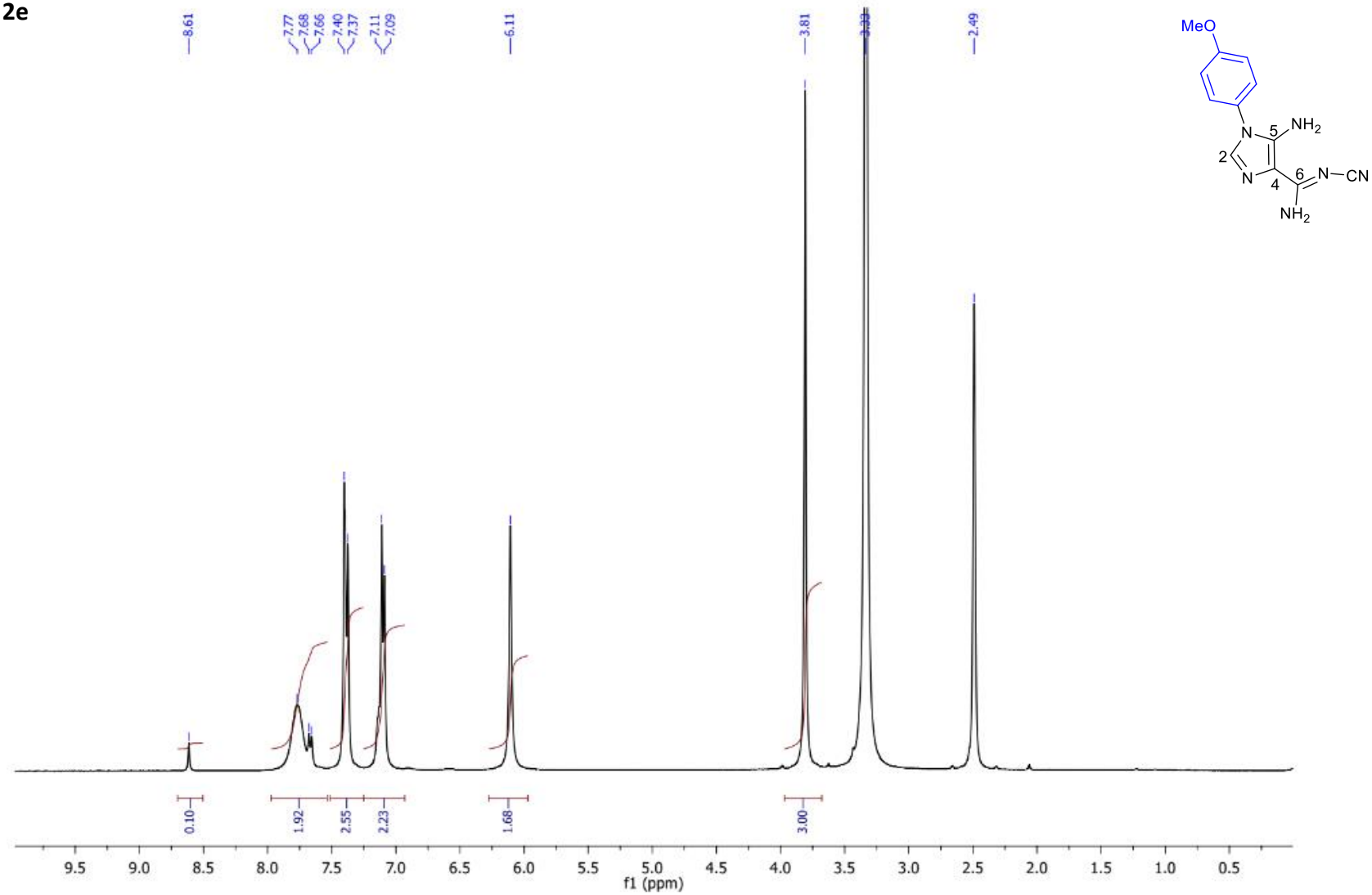



**Figure S2. 1:** $^{1}$H NMR spectrum (400 MHz, DMSO-$d_6$) of *(Z)*-5-amino-*N*'-cyano-1-(4-methoxyphenyl)-1*H*-imidazole-4-carboxamidine (**2e**).

**2f**

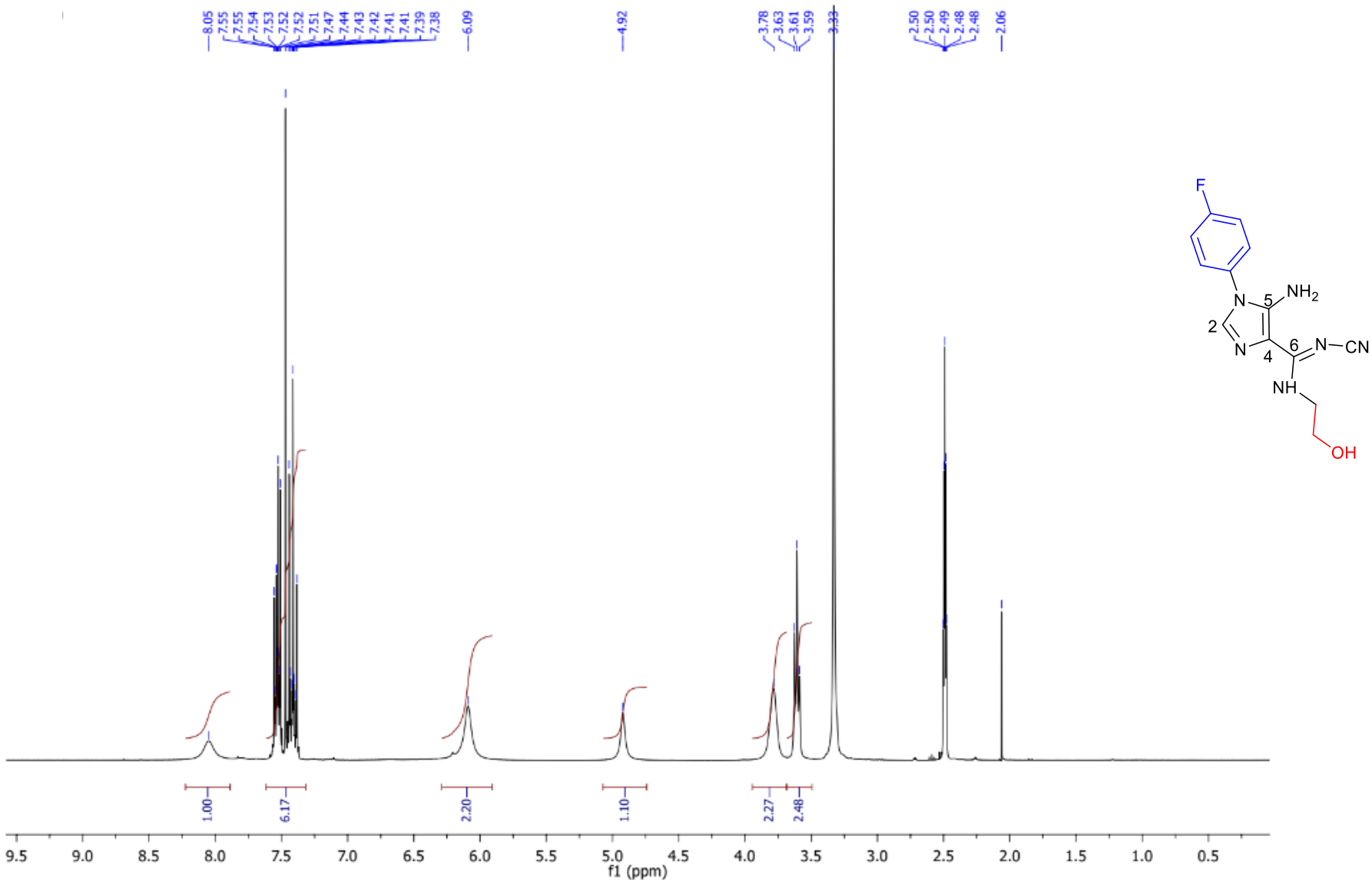



**Figure S2. 2:** $^{1}$H NMR spectrum (400 MHz, DMSO-$d_6$) of *(Z)*-5-amino-*N*'-cyano-1-(4-fluorophenyl)-*N*-(2-hydroxyethyl)-1*H*-imidazole-4-carboxamidine (**2f**).

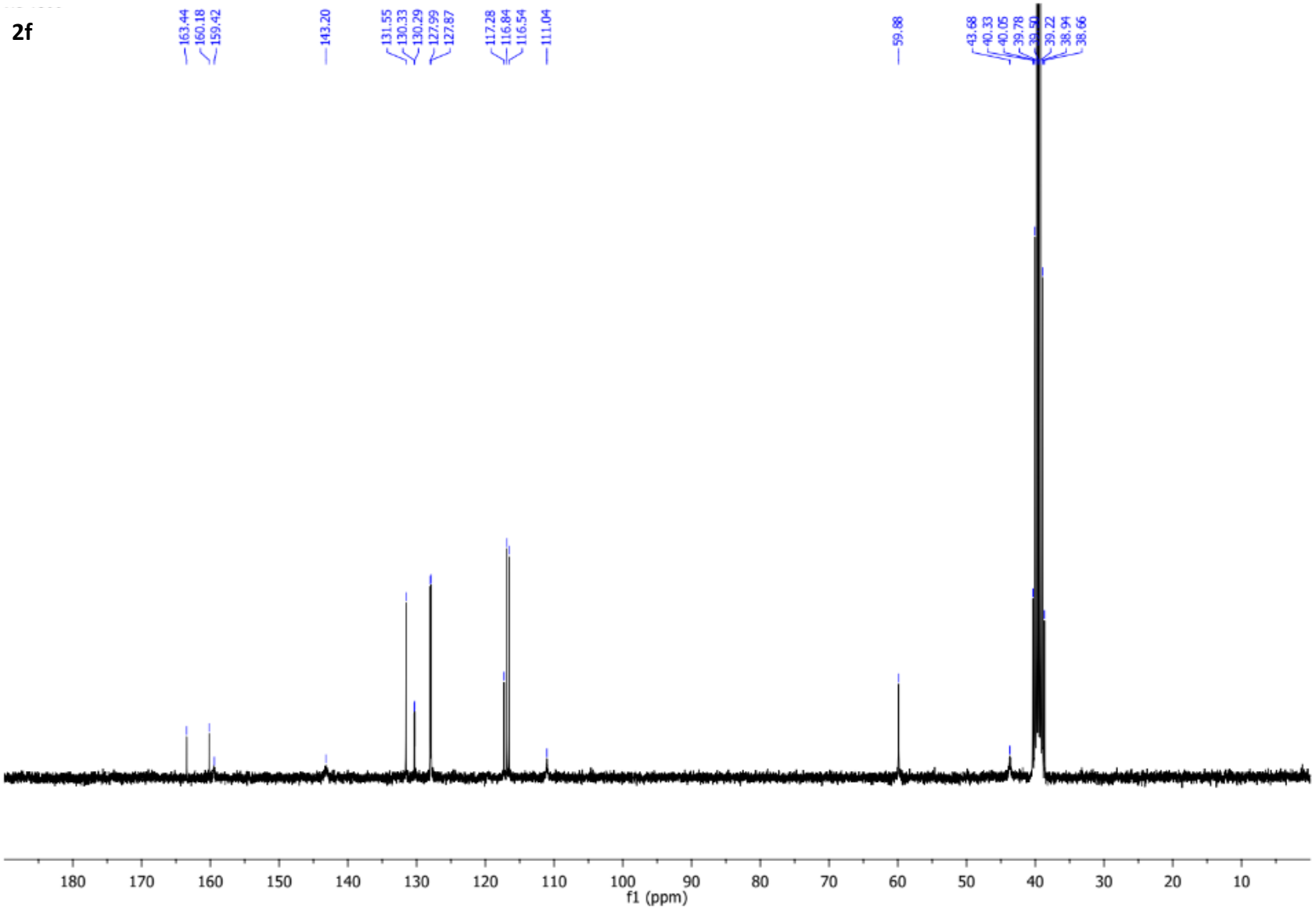



**Figure S2.3:** $^{13}$C NMR spectrum (100 MHz, DMSO-$d_6$) of *(Z)*-5-amino-*N*'-cyano-1-(4-fluorophenyl)-*N*-(2-hydroxyethyl)-1*H*-imidazole-4-carboxamidine (**2f**).

**2g**

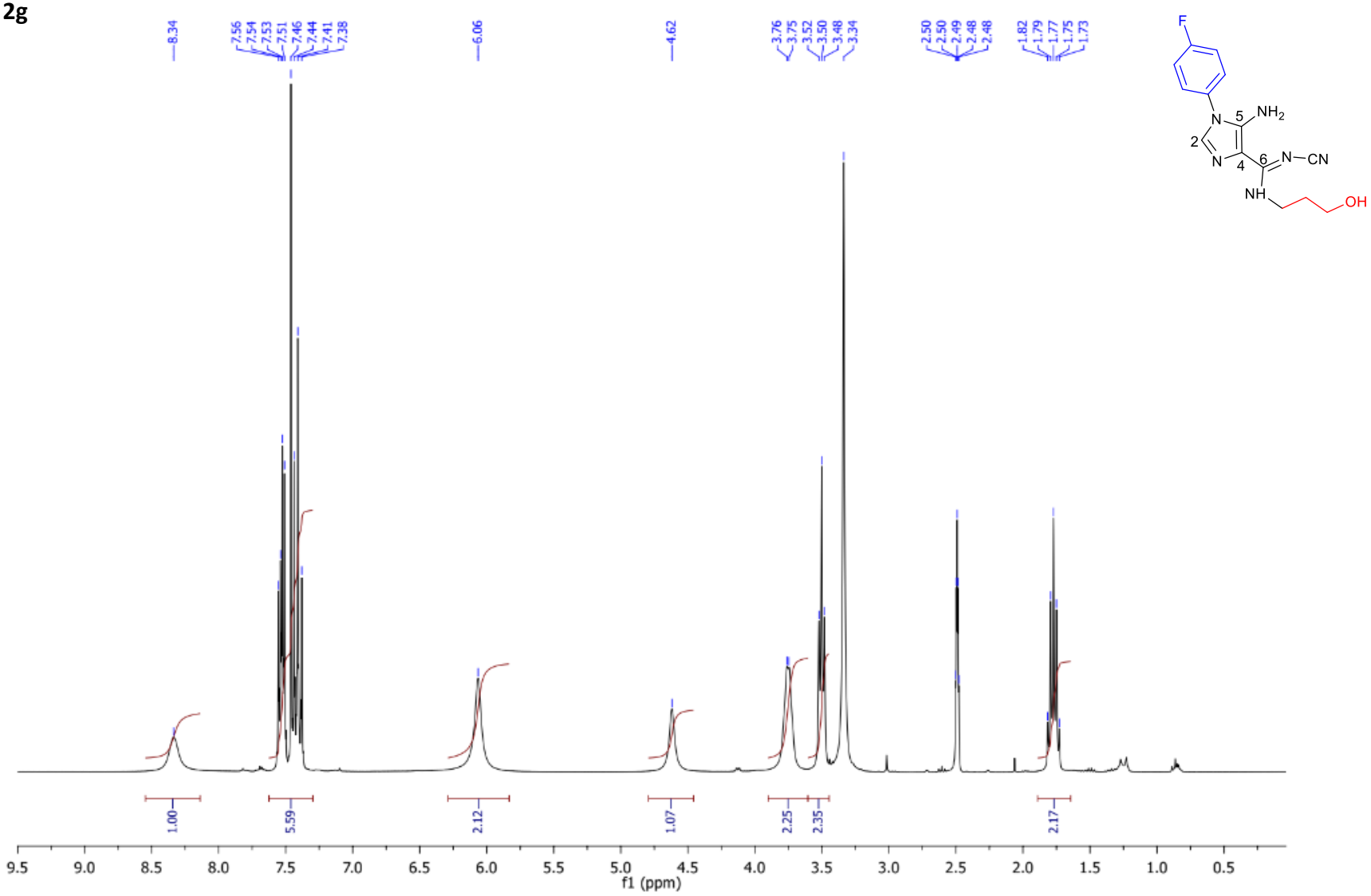



**Figure S2.4:** $^{1}$H NMR spectrum (400 MHz, DMSO-$d_6$) of 5-amino-*N*'-cyano-1-(4-fluorophenyl)-*N*-(3-hydroxypropyl)-1*H*-imidazole-4-carboxamidine (**2g**).

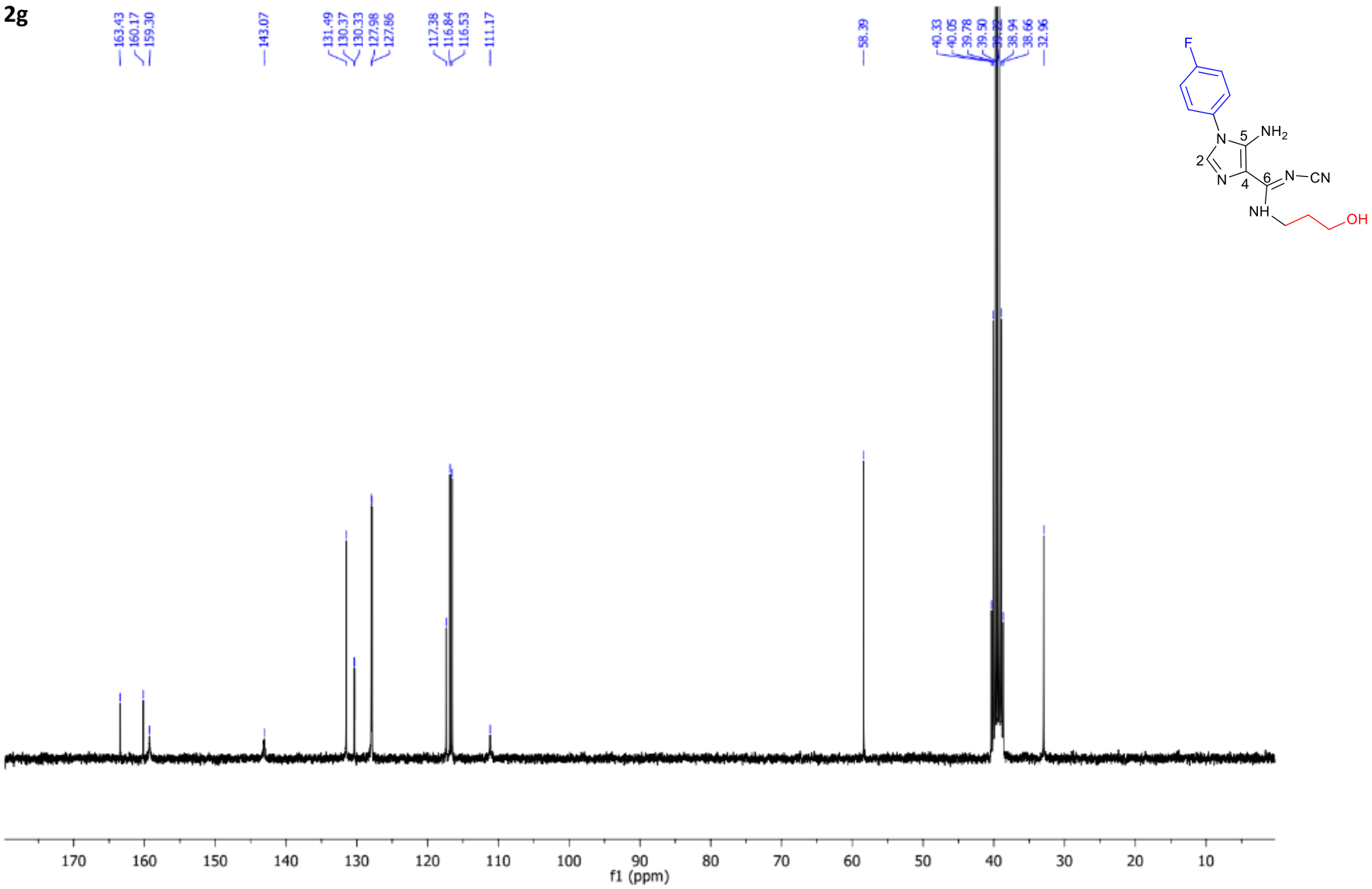



**Figure S2.5:** $^{13}$C NMR spectrum (100 MHz, DMSO-$d_6$) of 5-amino-N'-cyano-1-(4-fluorophenyl)-N-(3-hydroxypropyl)-1H-imidazole-4-carboxamidine (**2g**).

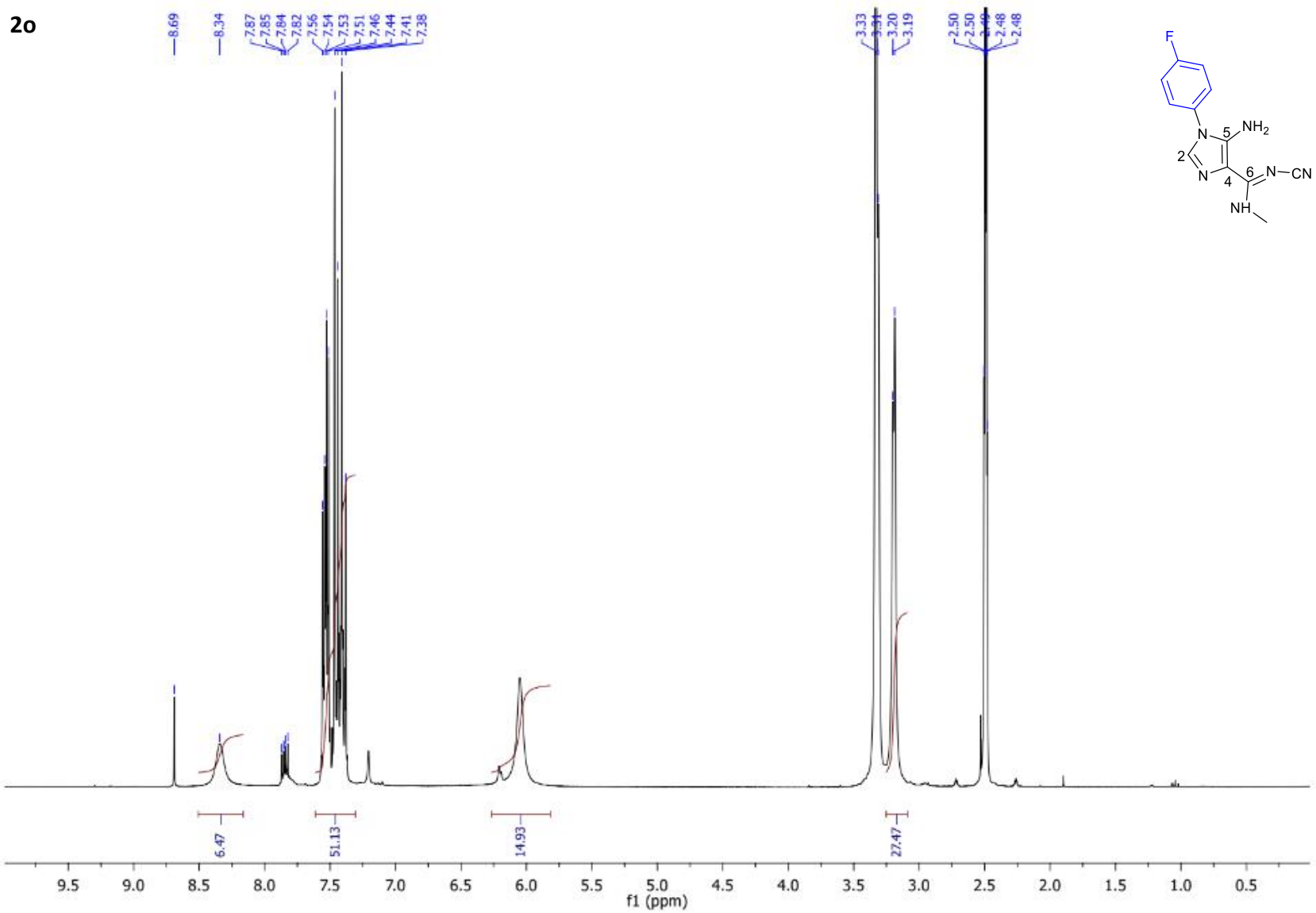



**Figure S2.6:** $^{1}$H NMR spectrum (400 MHz, DMSO-$d_6$) of 5-amino-*N*'-cyano-1-(4-fluorophenyl)-*N*-methyl-1H-imidazole-4-carboxamidine (**2o**).

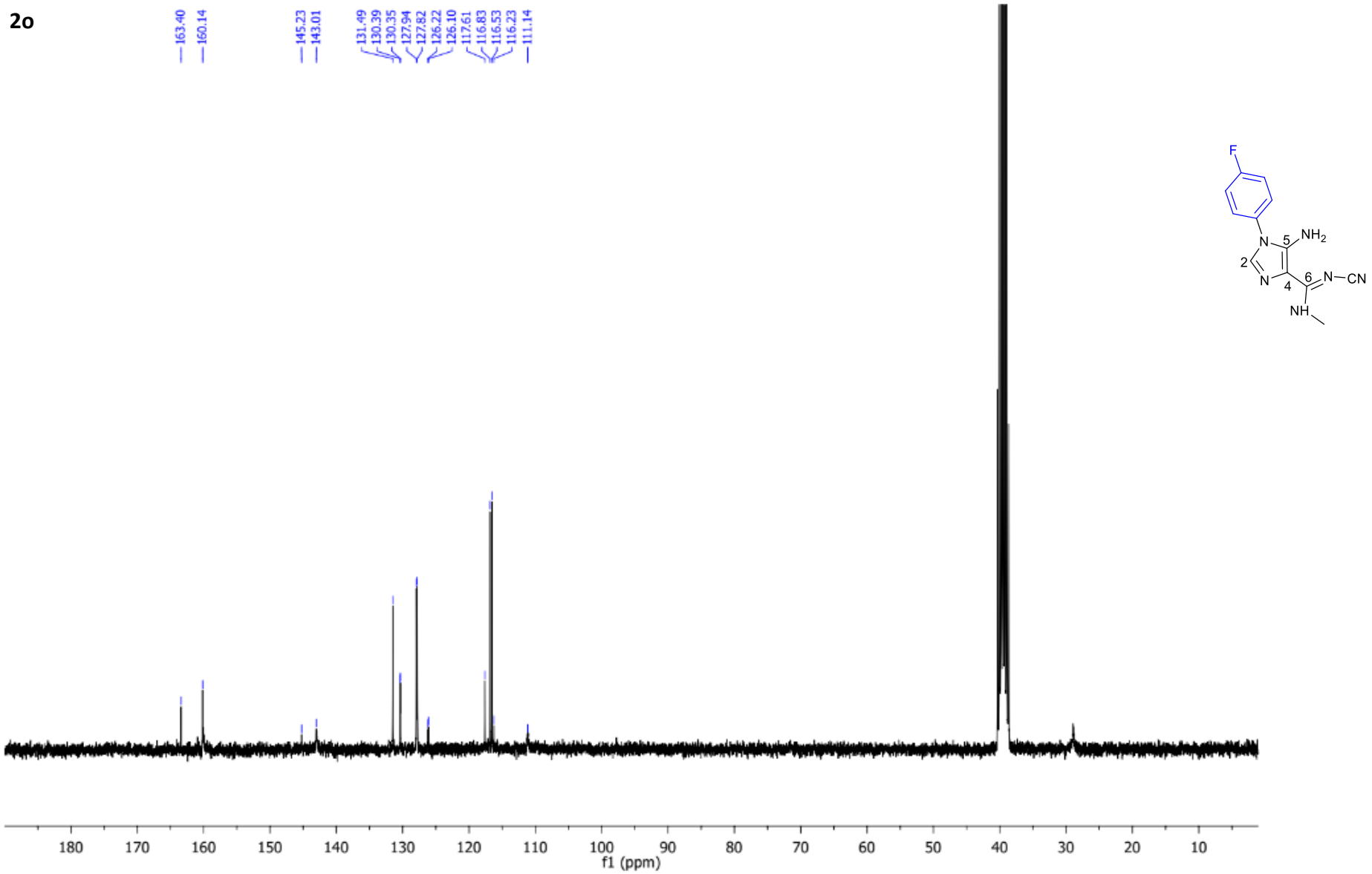



**Figure S2.7:** $^{13}$C NMR spectrum (100 MHz, DMSO-$d_6$) of 5-amino-*N*'-cyano-1-(4-fluorophenyl)-*N*-methyl-1*H*-imidazole-4-carboxamidine (**2o**).

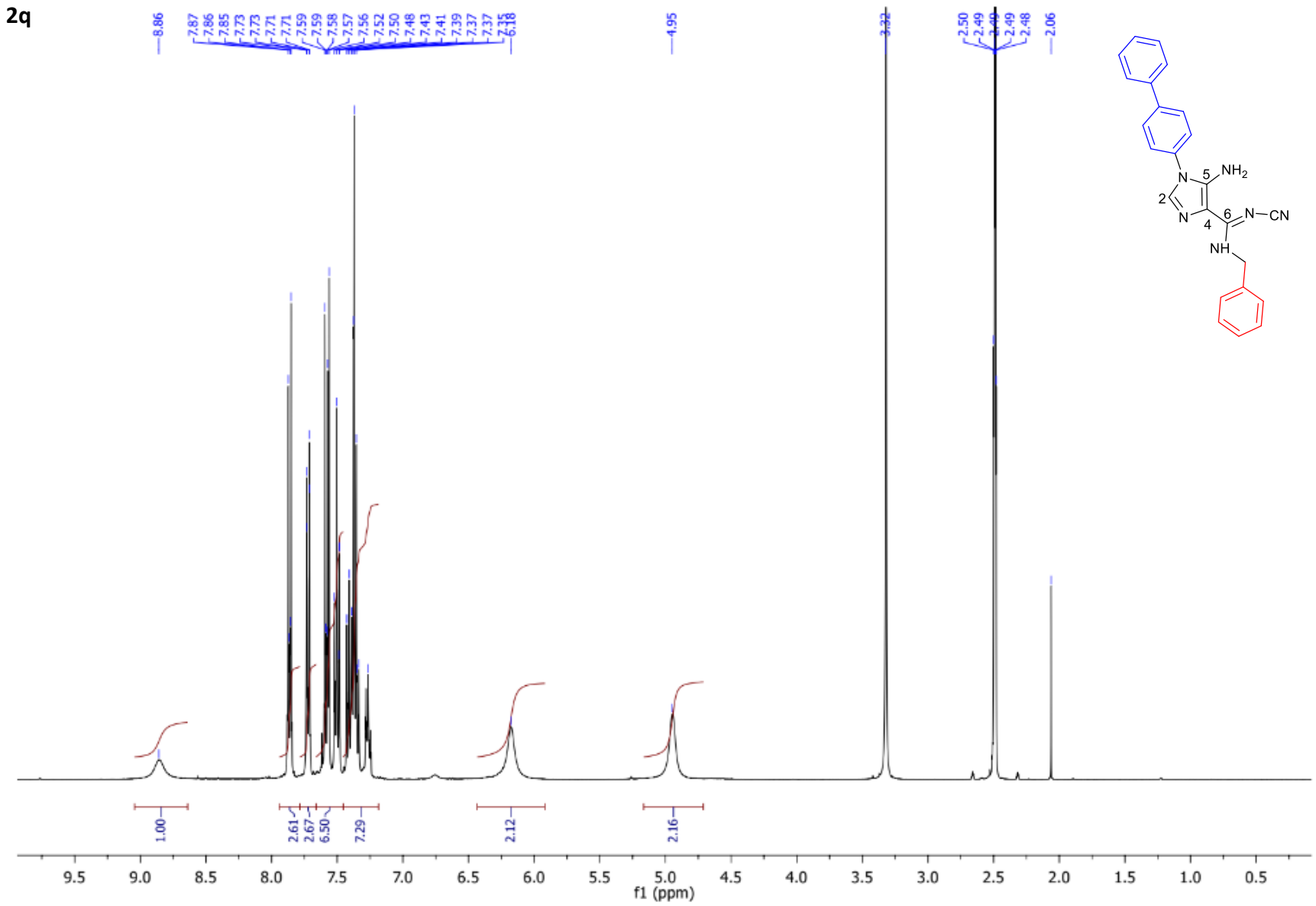



**Figure S2.8:** $^{1}$H NMR spectrum (400 MHz, DMSO-$d_6$) of 1-([1,1'-biphenyl]-4-yl)-5-amino-*N*-benzyl-*N*'-cyano-1*H*-imidazole-4-carboximidamide (**2q**).

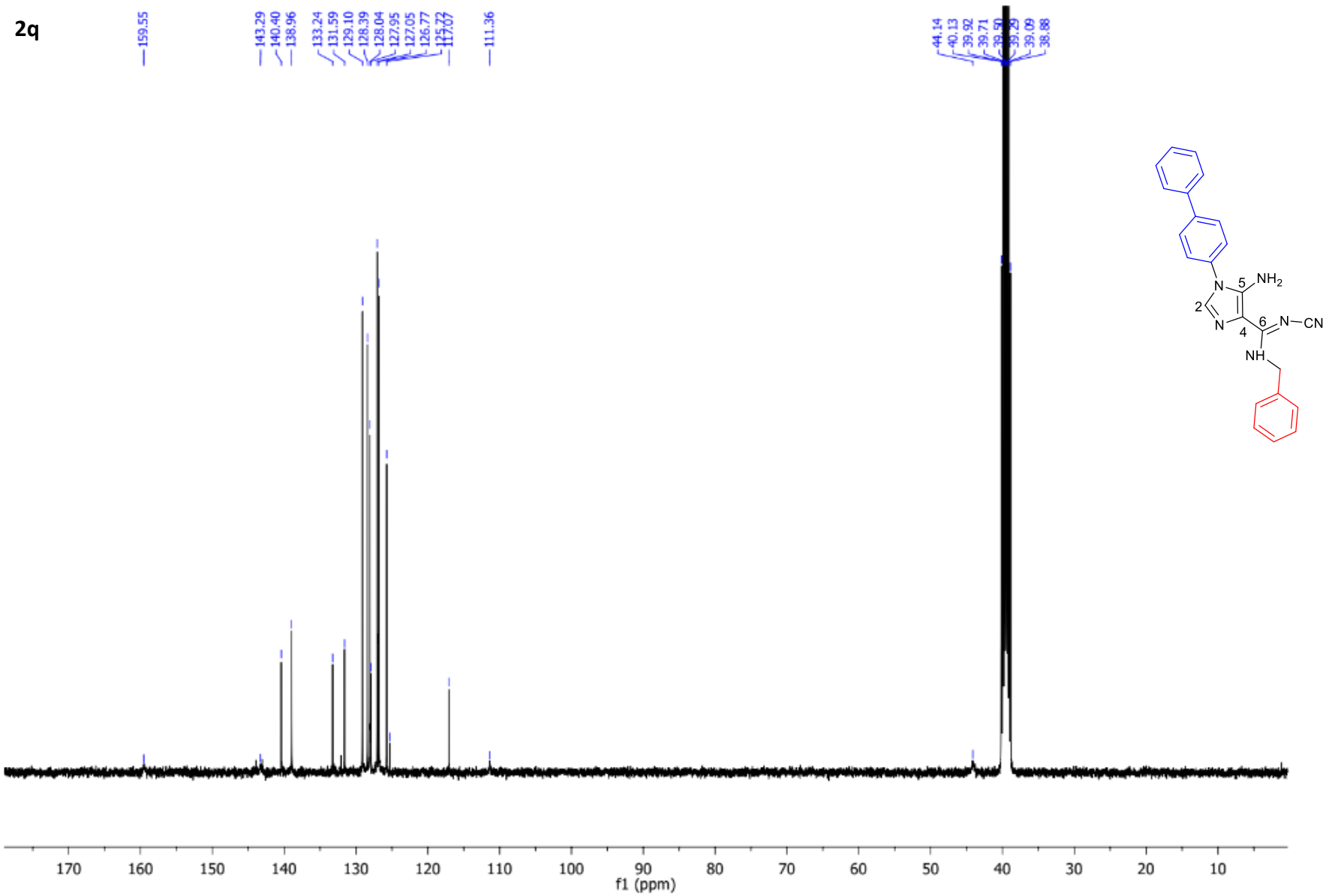



**Figure S2.9:** $^{13}$C NMR spectrum (100 MHz, DMSO-$d_6$) of 1-([1,1'-biphenyl]-4-yl)-5-amino-*N*-benzyl *N*'-cyano-1*H*-imidazole-4-carboximidamide (**2q**).

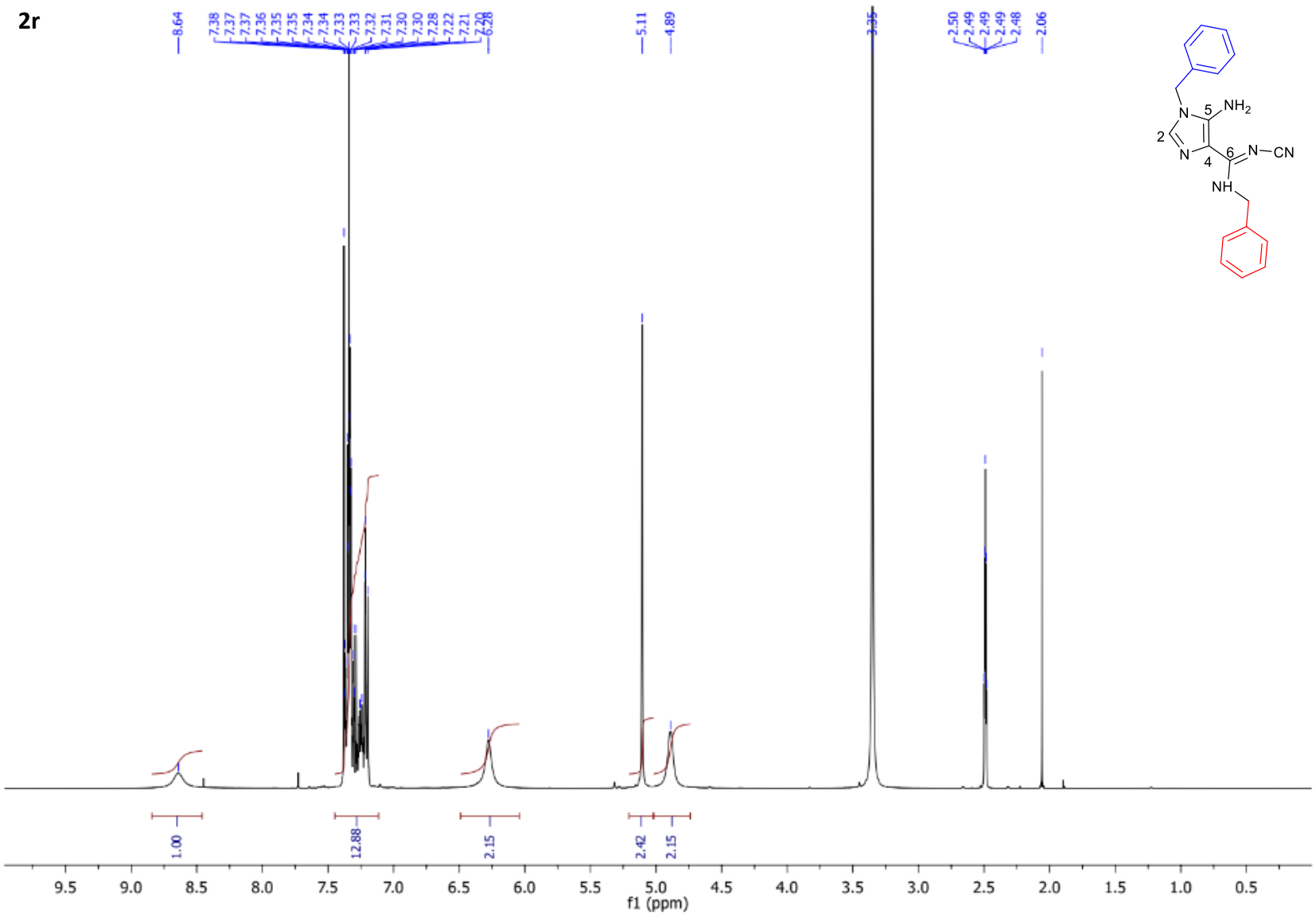



**Figure S2.10:** $^{1}$H NMR spectrum (400 MHz, DMSO-$d_6$) of 5-amino-*N*,1-dibenzyl-*N*'-cyano-1*H*-imidazole-4-carboxamidine (**2r**).

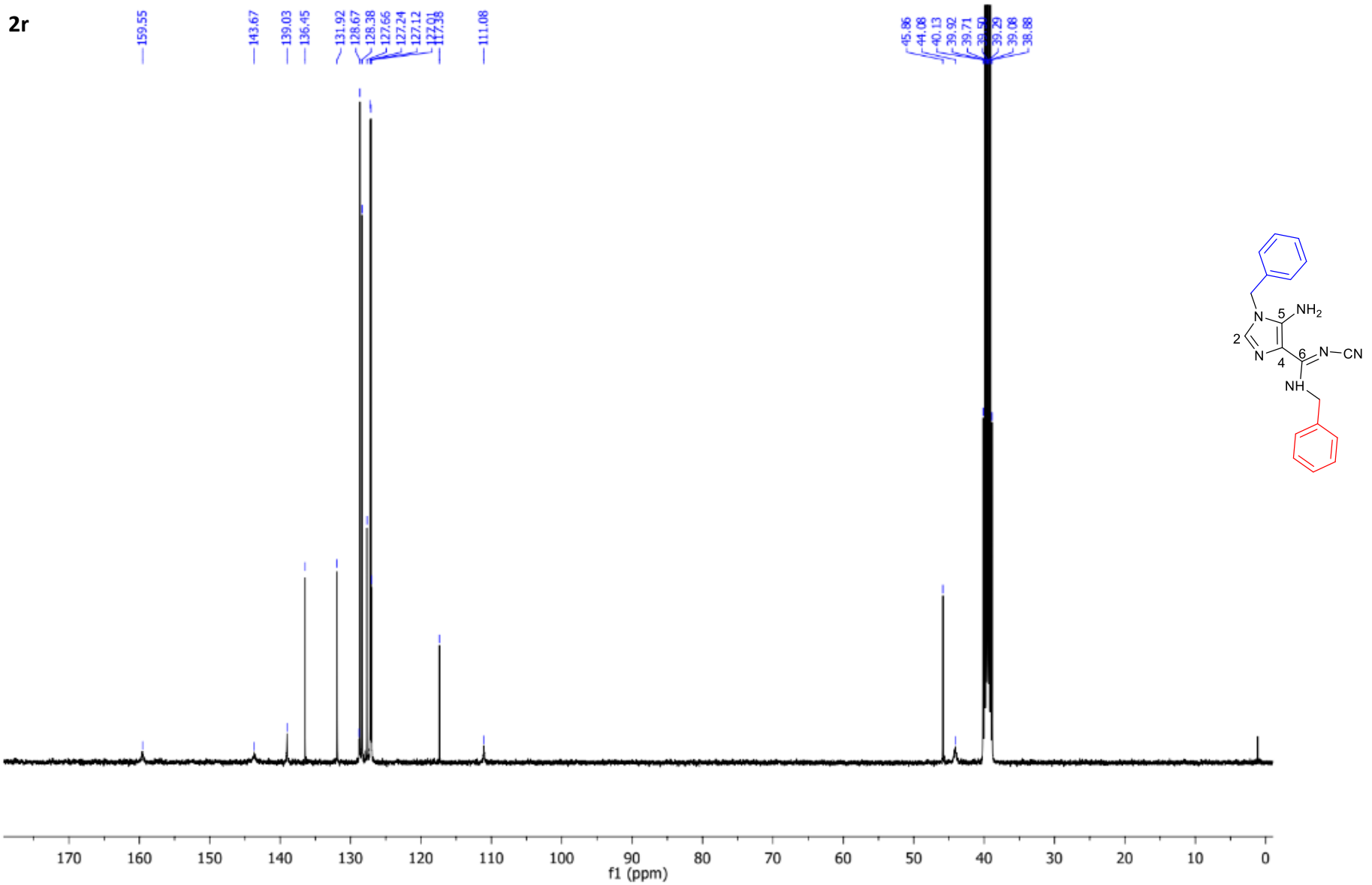



**Figure S2.11:** $^{13}$C NMR spectrum (100 MHz, DMSO-$d_6$) of 5-amino-*N*,1-dibenzyl-N'-cyano-1*H*-imidazole-4-carboxamidine (**2r**).

**2s**

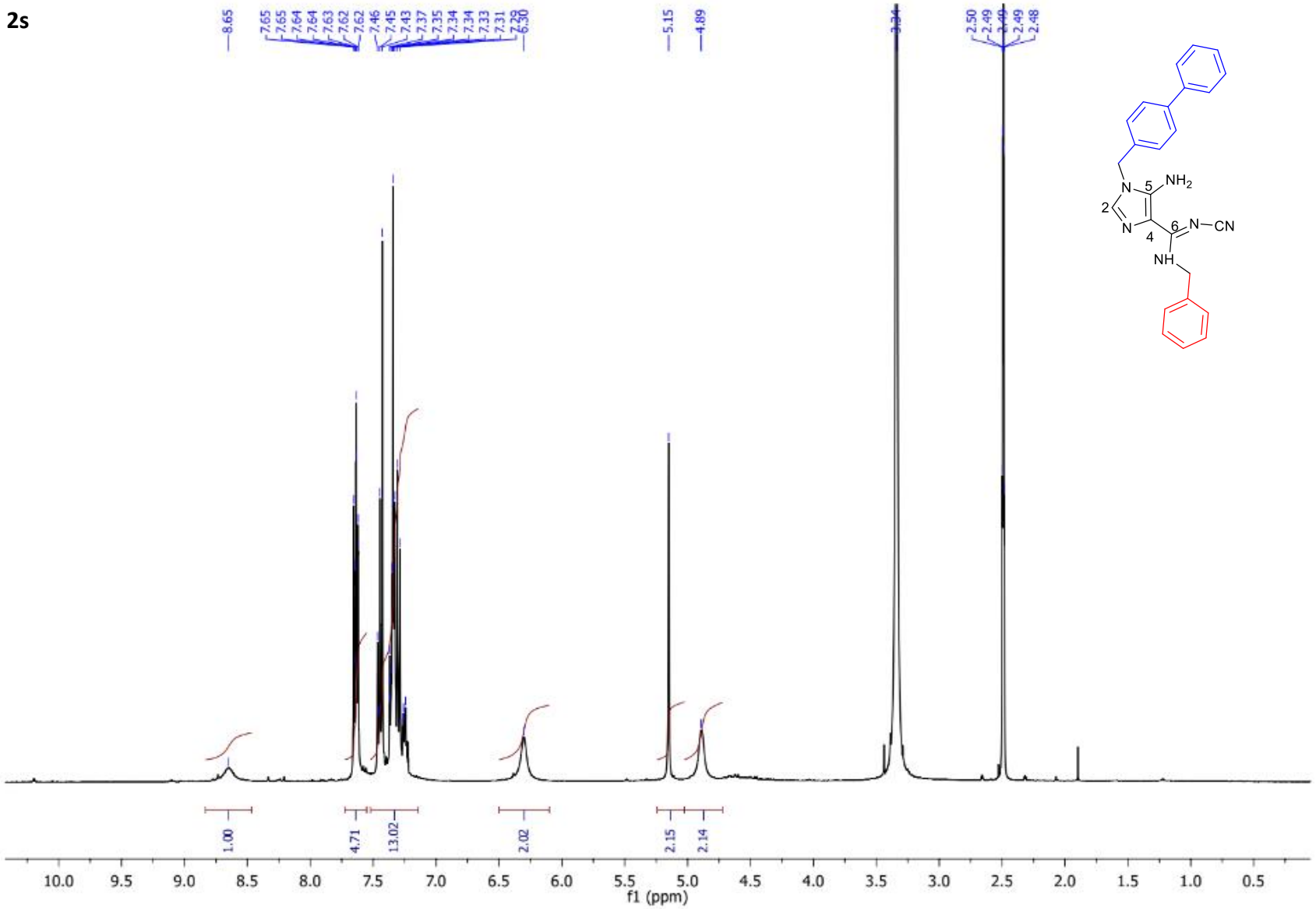



**Figure S2.12:** $^{1}$H NMR spectrum (400 MHz, DMSO-$d_6$) of 1-([1,1'-biphenyl]-4-ylmethyl)-5-amino-*N*-benzyl-*N*'-cyano-1*H*-imidazole-4-carboxamidine (**2s**).

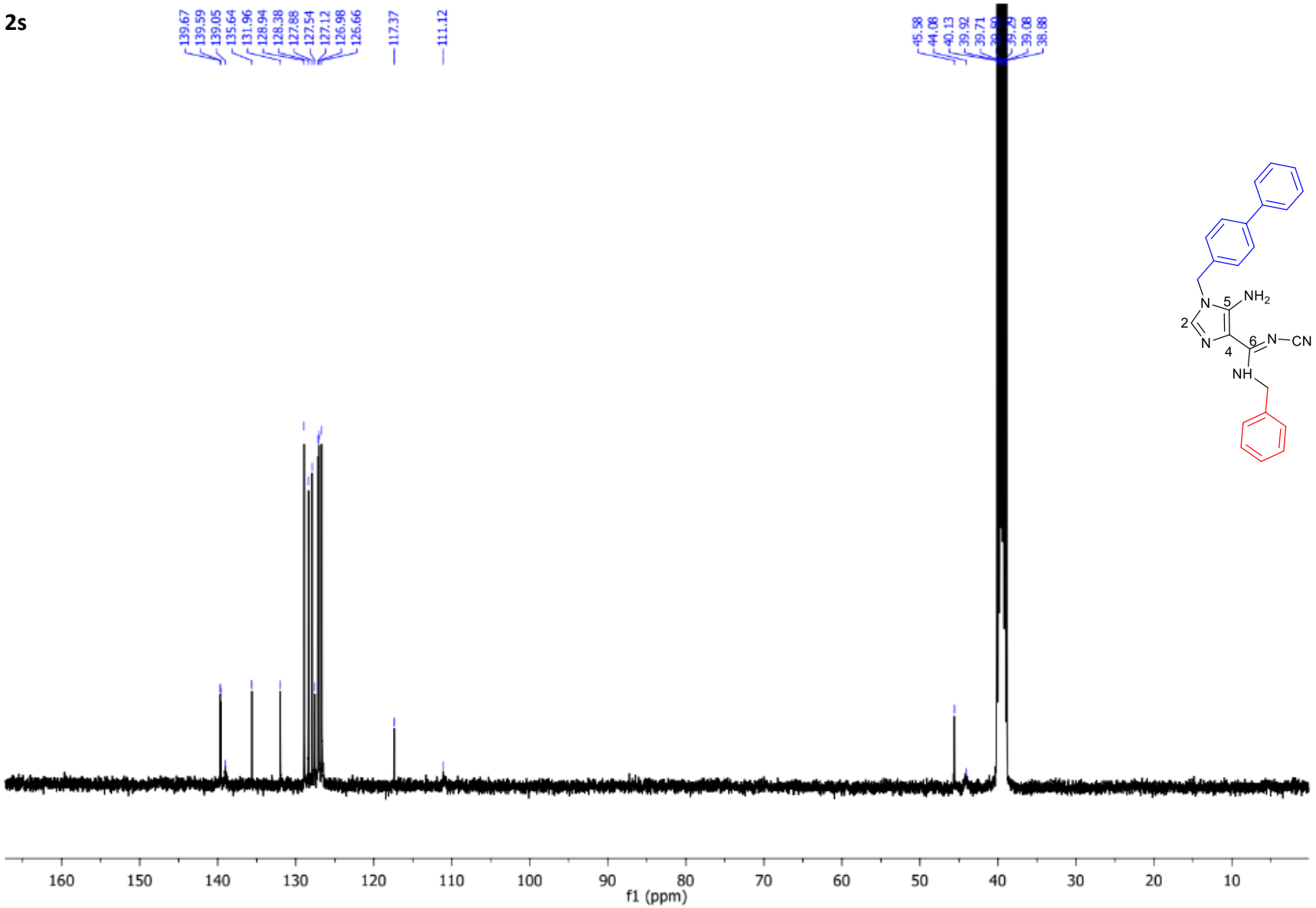



**Figure S2.13:** $^{13}$C NMR spectrum (100 MHz, DMSO-$d_6$) of 1-([1,1'-biphenyl]-4-ylmethyl)-5-amino-*N*-benzyl-*N*'-cyano-1*H*-imidazole-4-carboxamidine (**2s**).

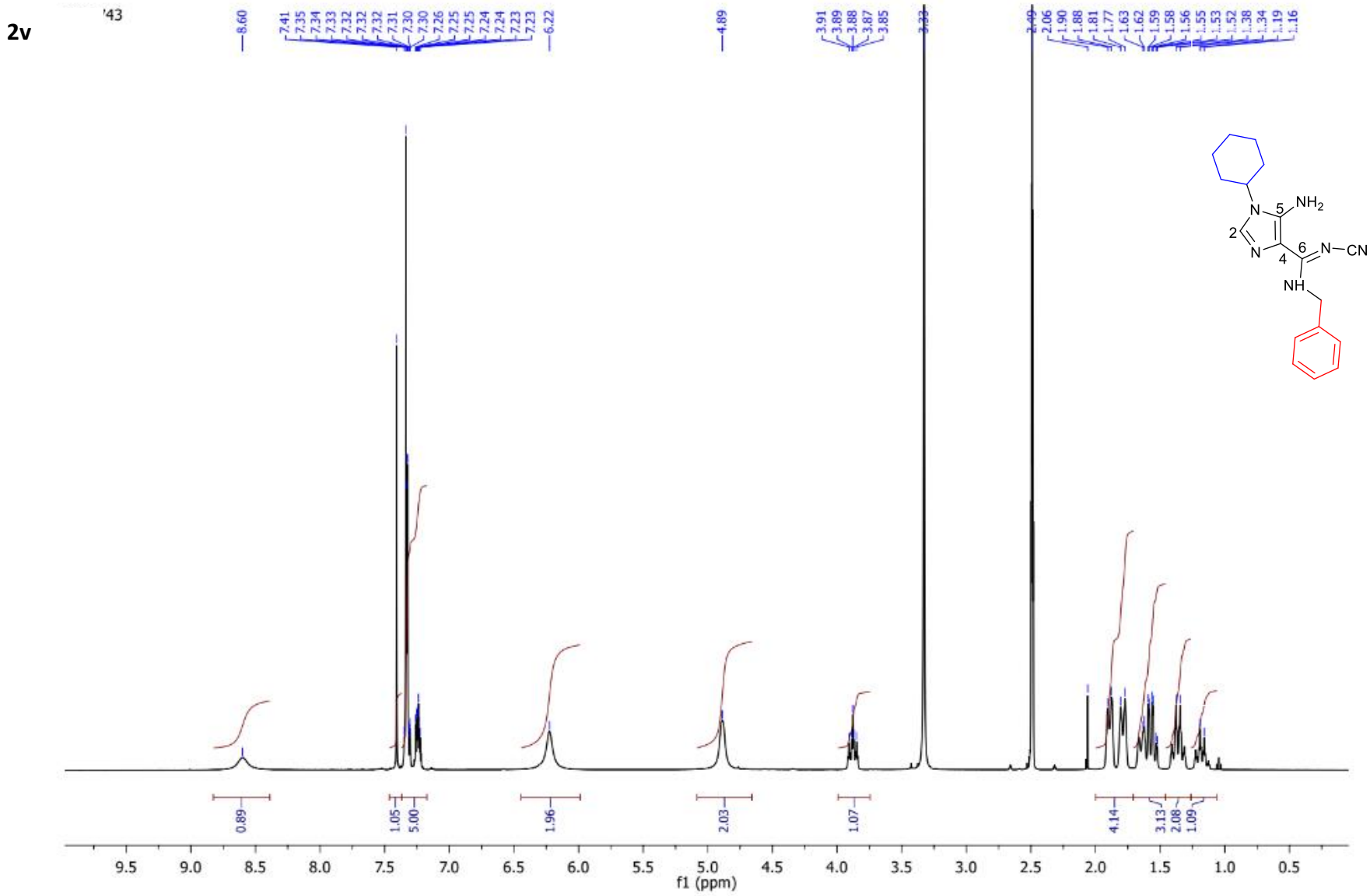



**Figure S2.14:** $^{1}$H NMR spectrum (400 MHz, DMSO-$d_6$) of 5-amino-*N*-benzyl-*N*'-cyano-1-cyclohexyl-1*H*-imidazole-4-carboximidamide (**2v**).

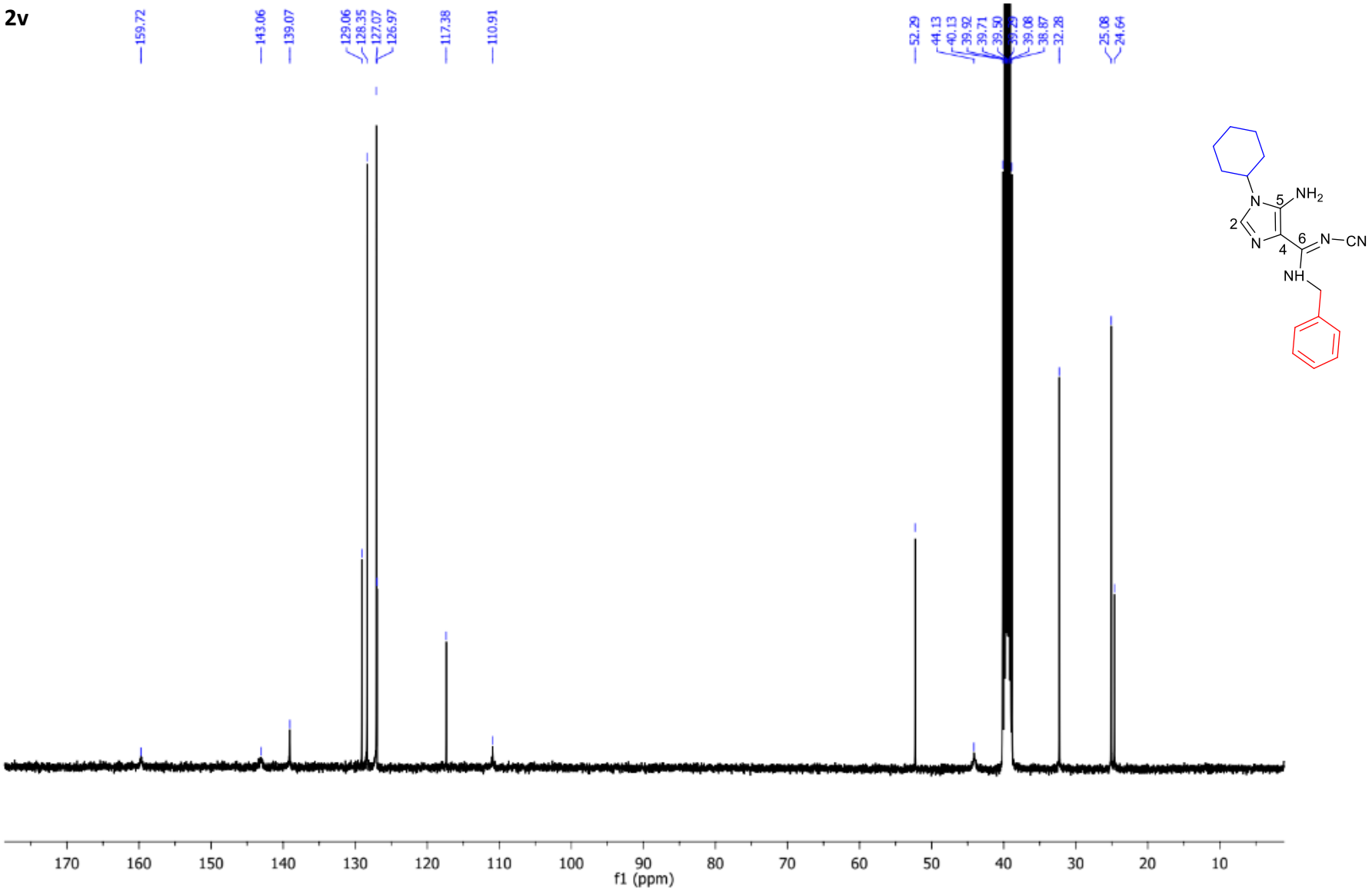



**Figure S2.15:** $^{13}$C NMR spectrum (100 MHz, DMSO-$d_6$) of 5-amino-*N*-benzyl-*N*'-cyano-1-cyclohexyl-1*H*-imidazole-4-carboximidamide (**2v**).

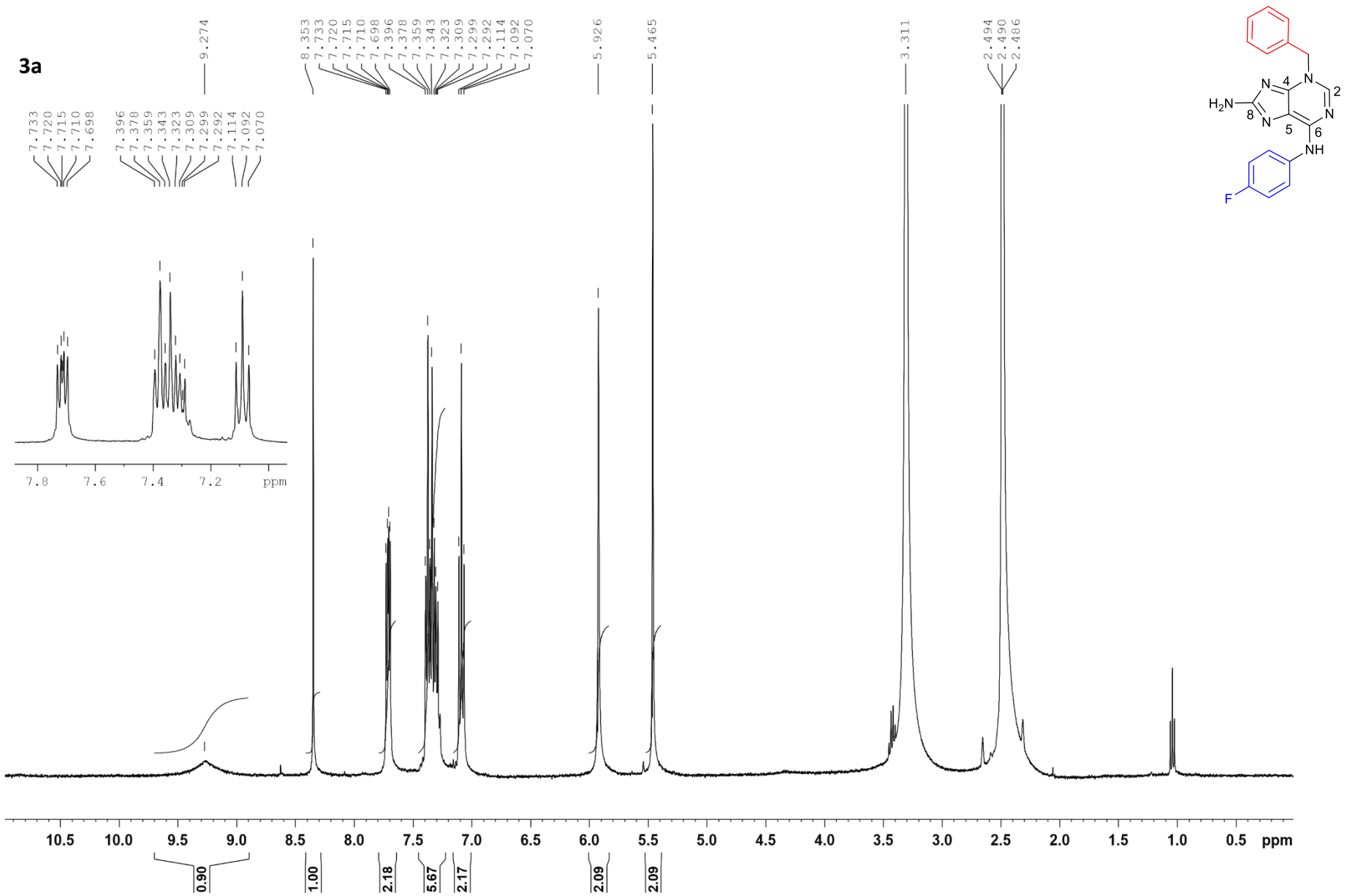



**Figure S2.16:** $^{1}$H NMR spectrum (400 MHz, DMSO-$d_6$) of 3-benzyl-$N^6$-(4-fluorophenyl)-3*H*-purine-6,8-diamine (**3a**).

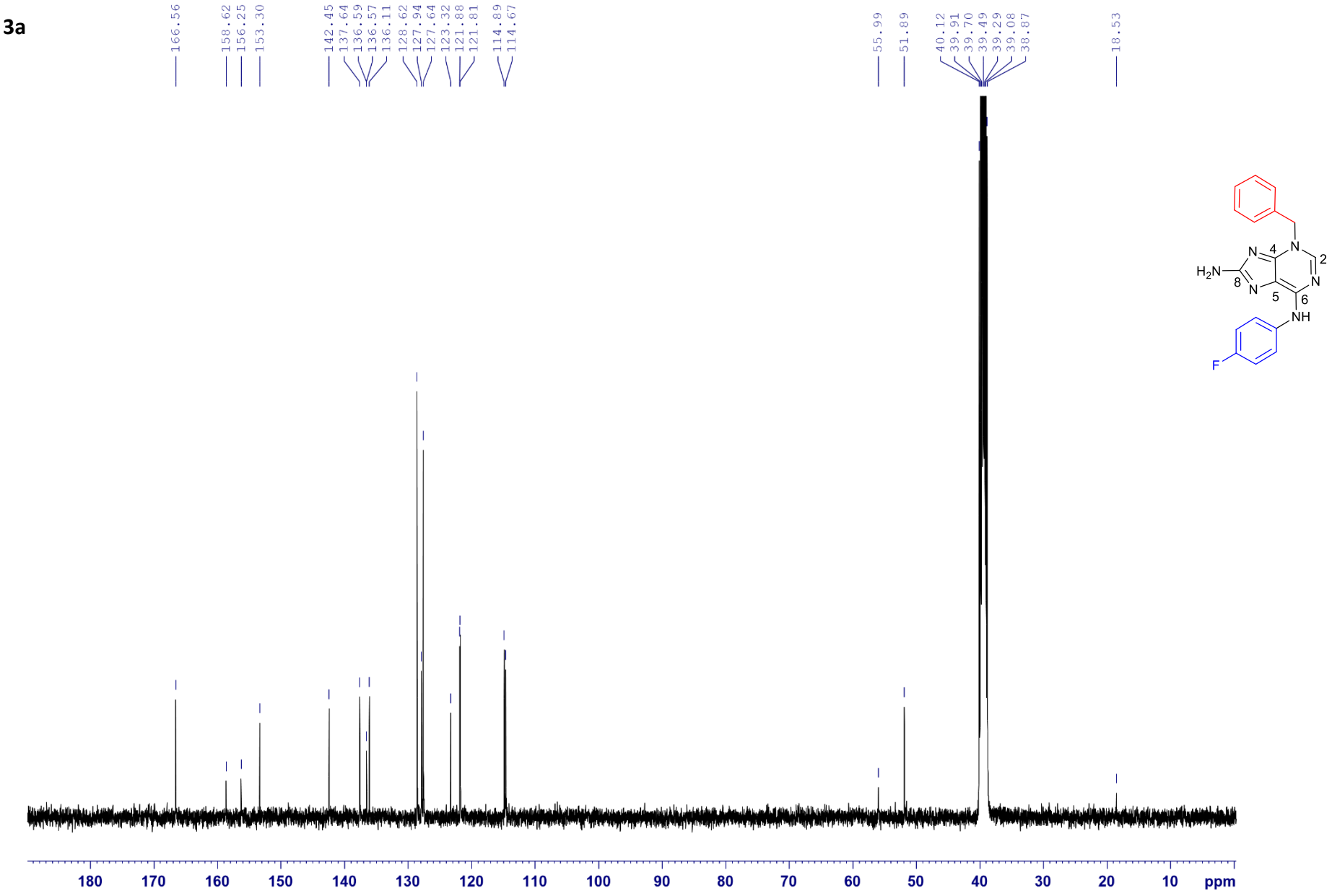



**Figure S2.17:** $^{13}$C NMR spectrum (100 MHz, DMSO-$d_6$) of 3-benzyl-$N^6$-(4-fluorophenyl)-3*H*-purine-6,8-diamine (**3a**).

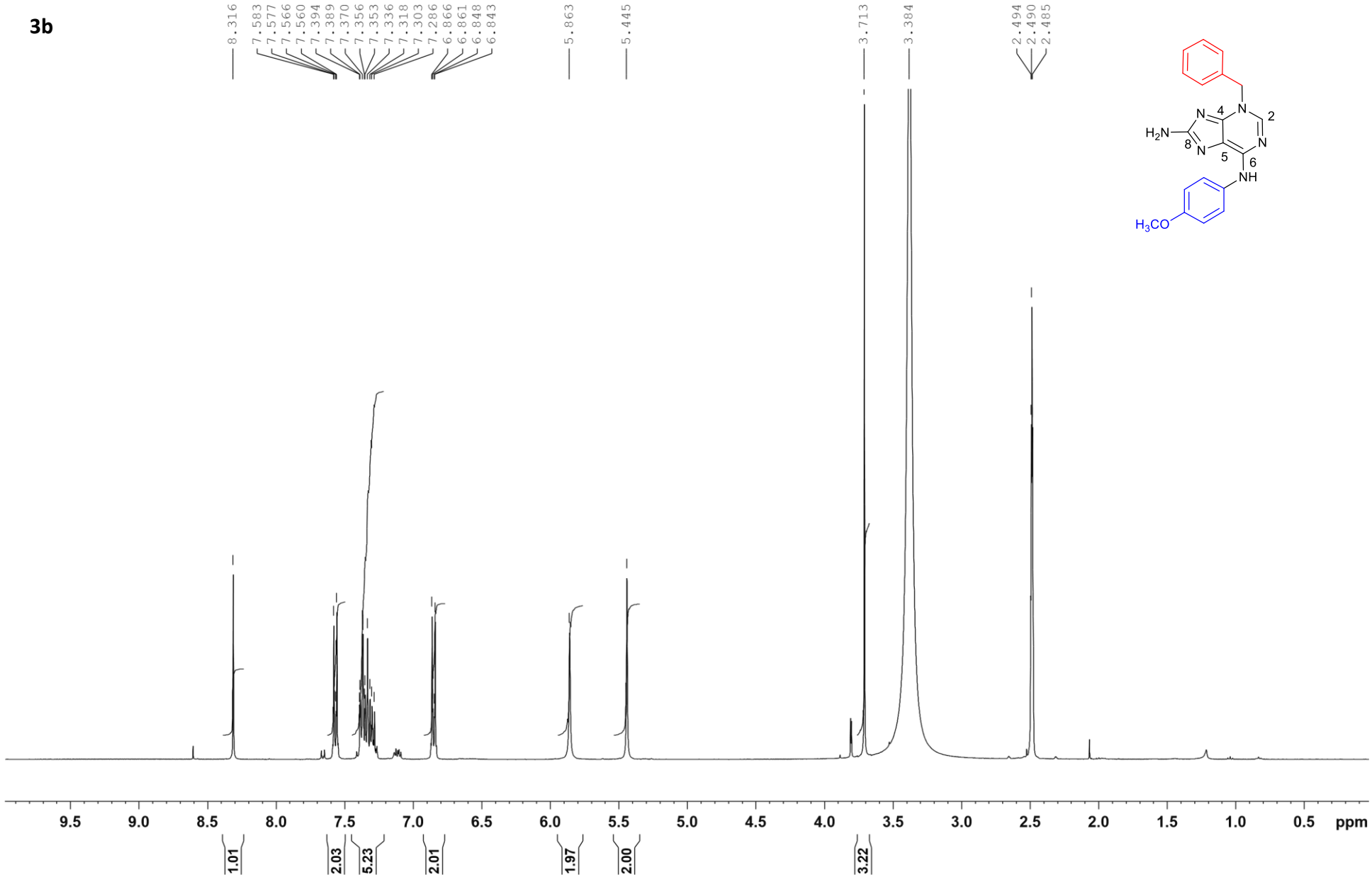



**Figure S2.18:** $^{1}$H NMR spectrum (400 MHz, DMSO-$d_6$) of 3-benzyl-$N^6$-(4-methoxyphenyl)-3*H*-purine-6,8-diamine (**3b**)

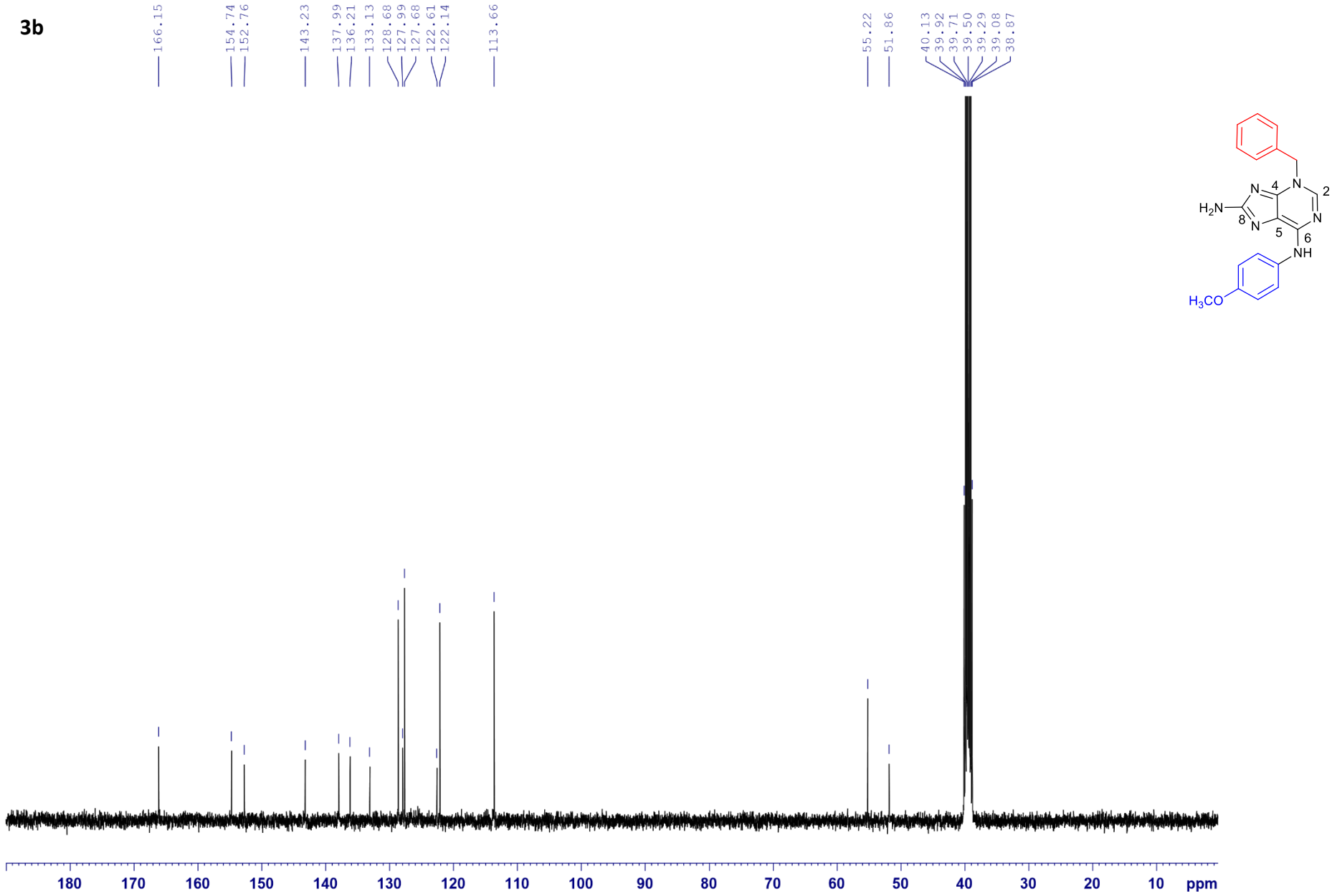



**Figure S2.19:** $^{13}$C NMR spectrum (100 MHz, DMSO-$d_6$) of 3-benzyl-$N^6$-(4-methoxyphenyl)-3*H*-purine-6,8-diamine (**3b**).

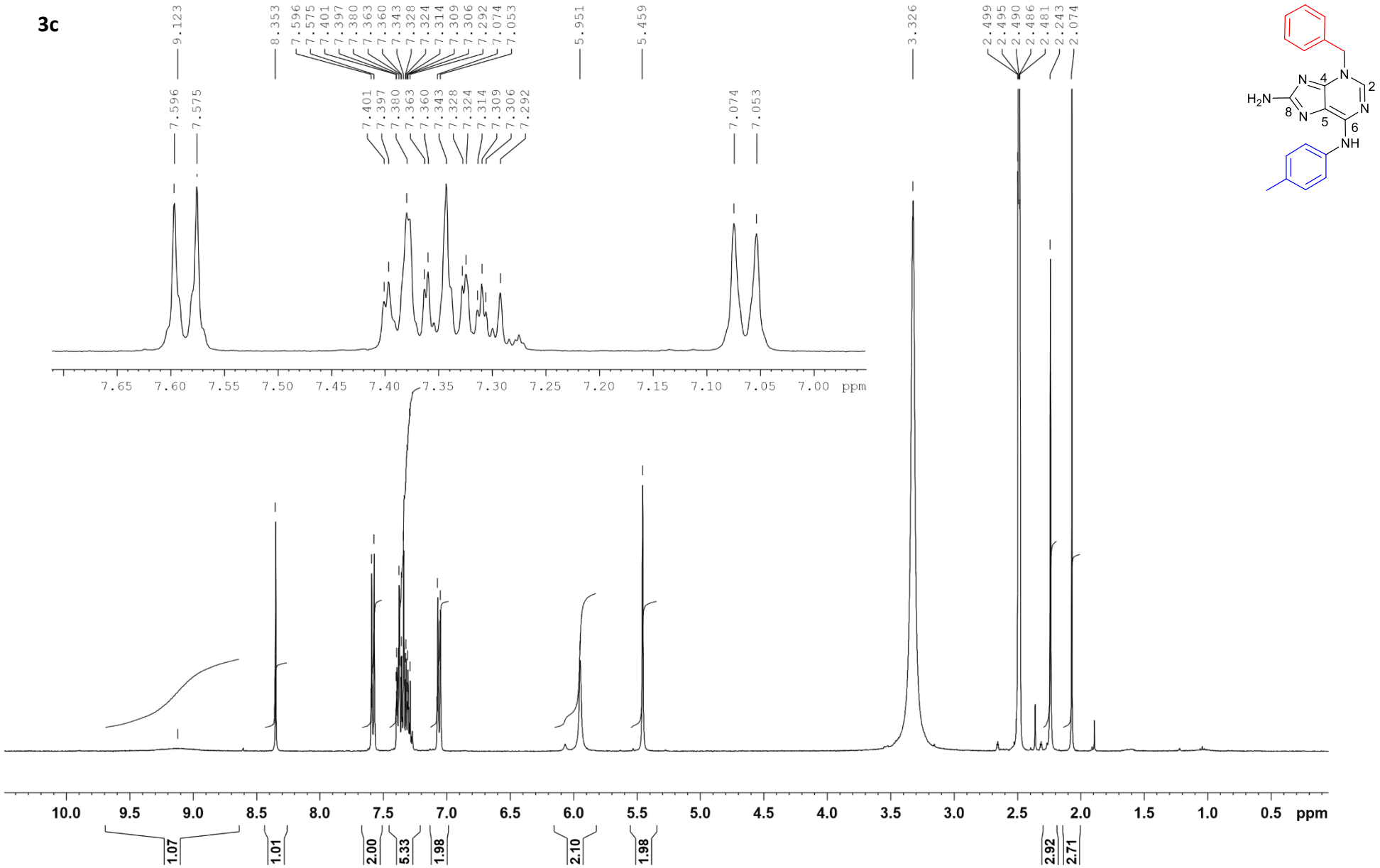



**Figure S2.20:** $^{1}$H NMR spectrum (400 MHz, DMSO-$d_6$) of 3-benzyl-$N^6$-(*p*-tolyl)-3*H*-purine-6,8-diamine (**3c**).

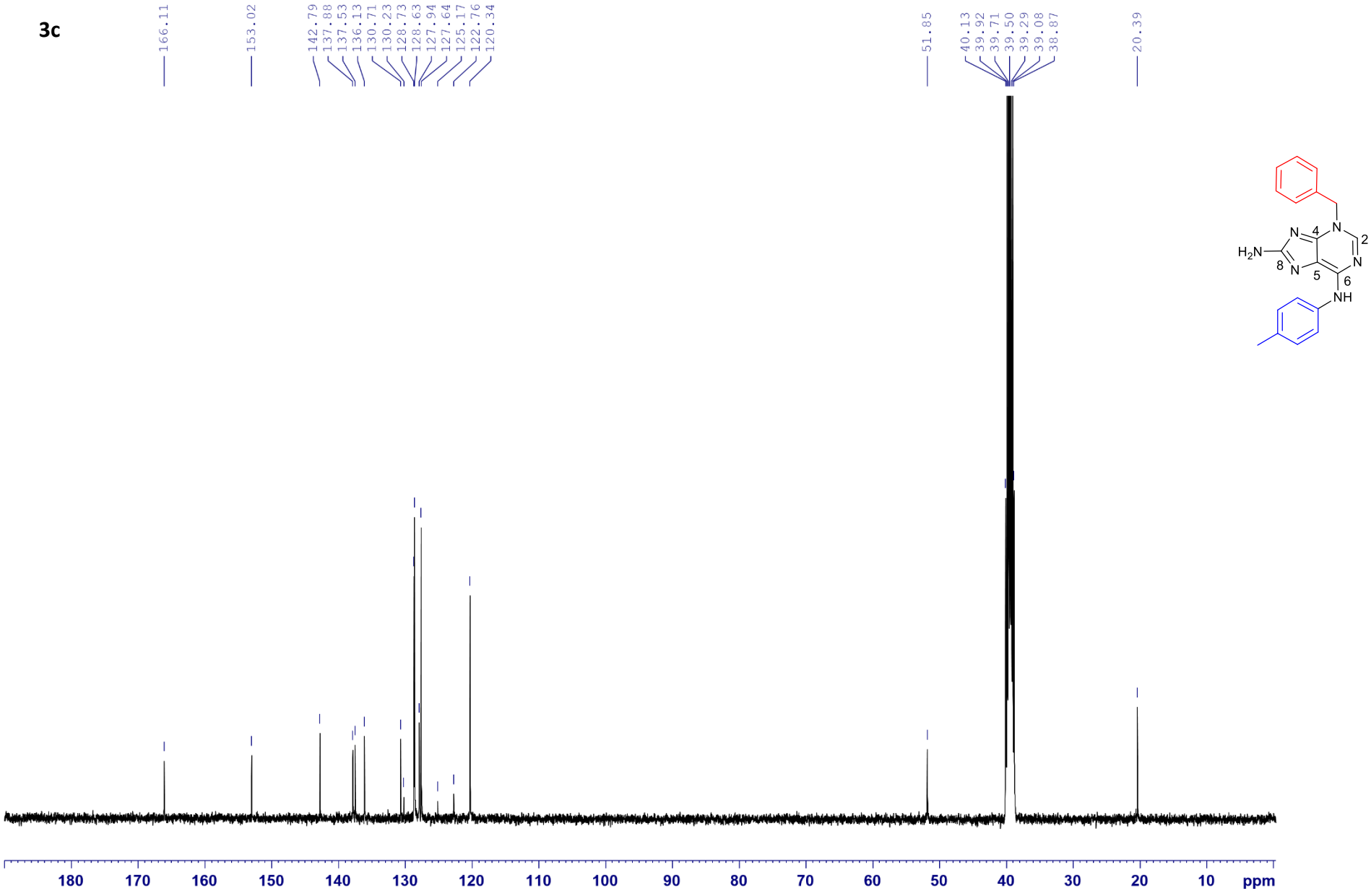



**Figure S2.21:** $^{13}$C NMR spectrum (100 MHz, DMSO-$d_6$) of 3-benzyl-$N^6$-(*p*-tolyl)-3*H*-purine-6,8-diamine (**3c**).

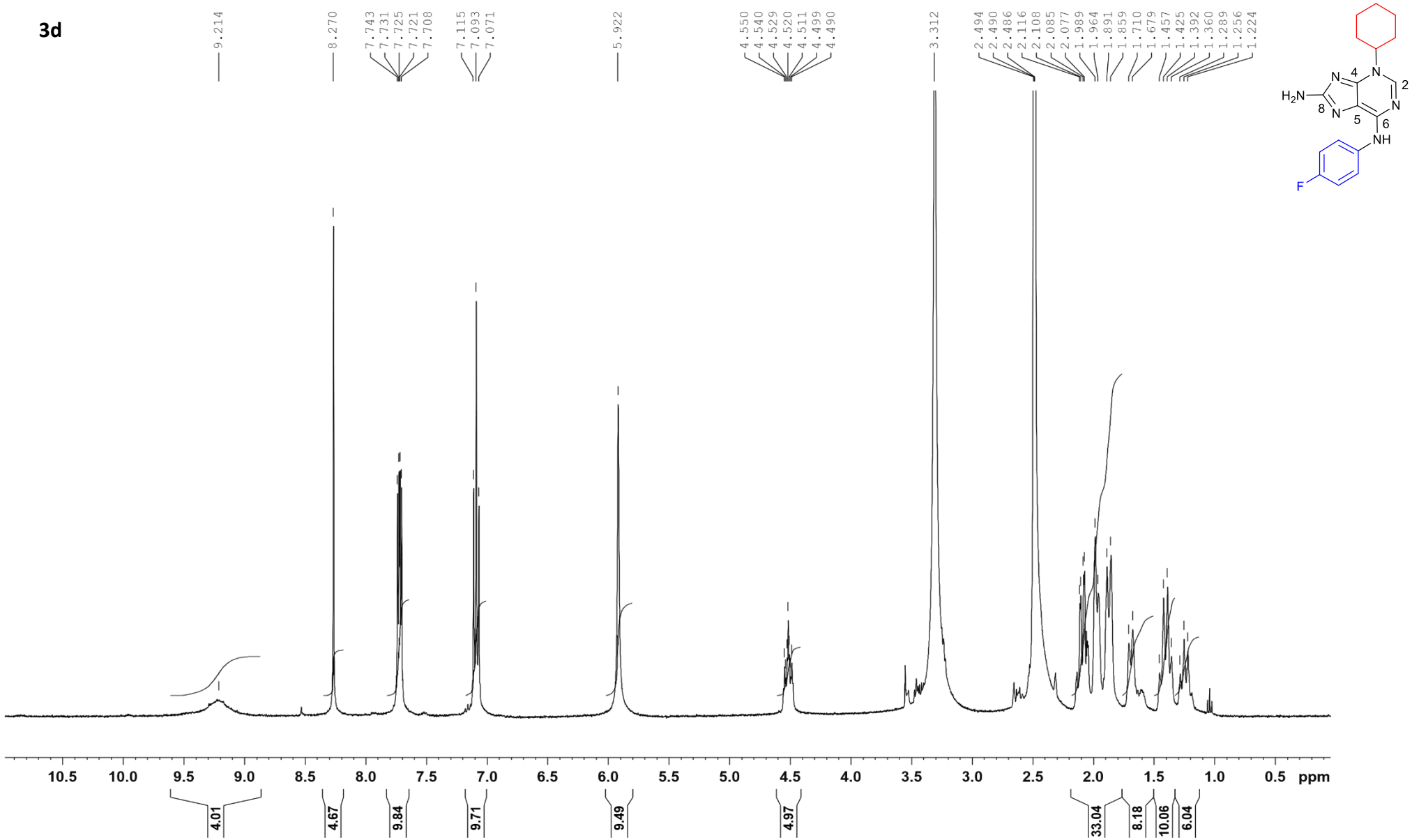



**Figure S2.22:** $^{1}$H NMR spectrum (400 MHz, DMSO-$d_6$) of 3-cyclohexyl-$N^6$-(4-fluorophenyl)-3*H*-purine-6,8-diamine (**3d**).

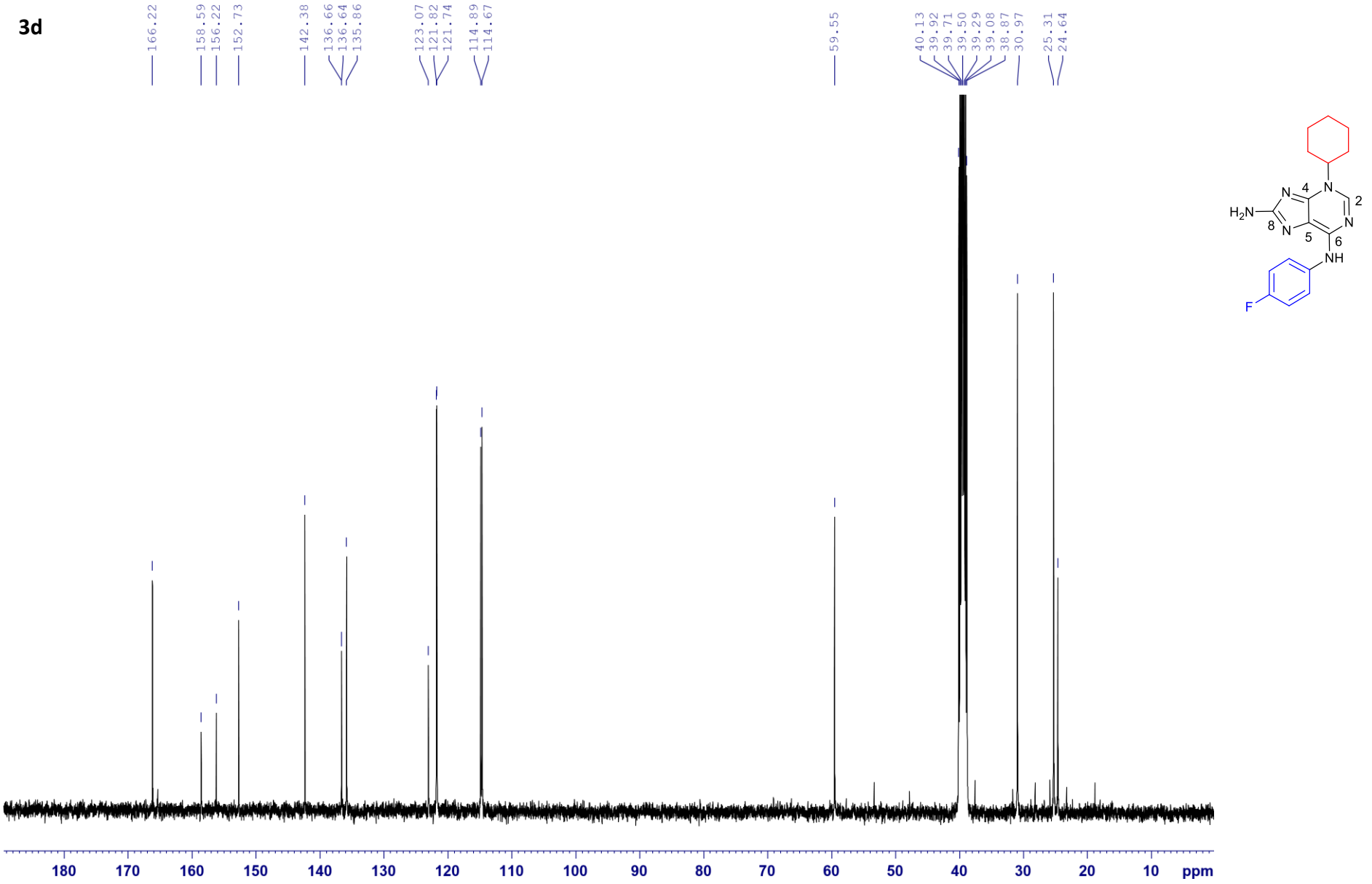



**Figure S2.23:** $^{13}$C NMR spectrum (100 MHz, DMSO-$d_6$) of of 3-cyclohexyl-$N^6$-(4-fluorophenyl)-3*H*-purine-6,8-diamine (**3d**).

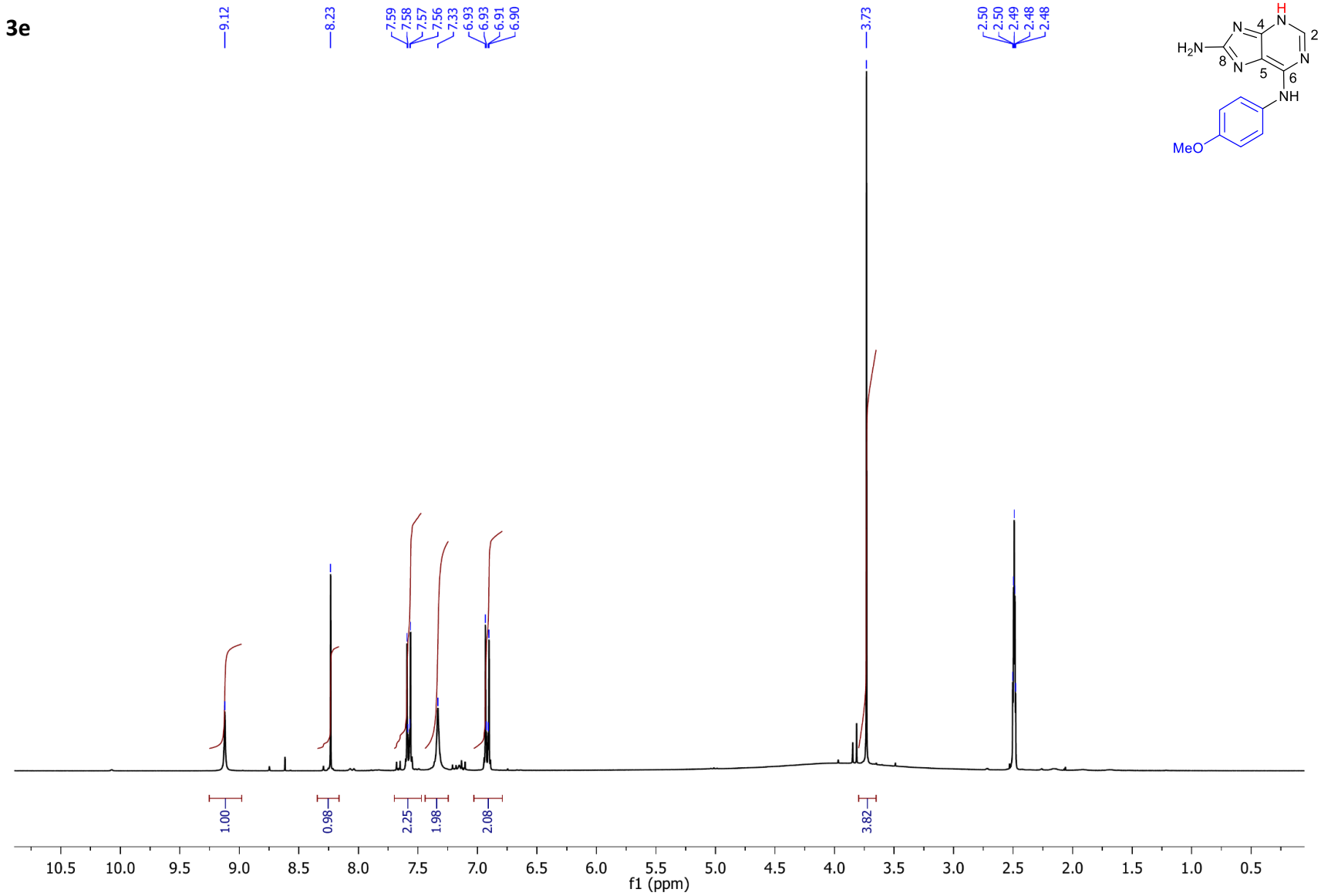



**Figure S2.24:** $^{1}$H NMR spectrum (400 MHz, DMSO-$d_6$) of $N^6$-(4-methoxyphenyl)-9*H*-purine-6,8-diamine sulfate (**3e**).

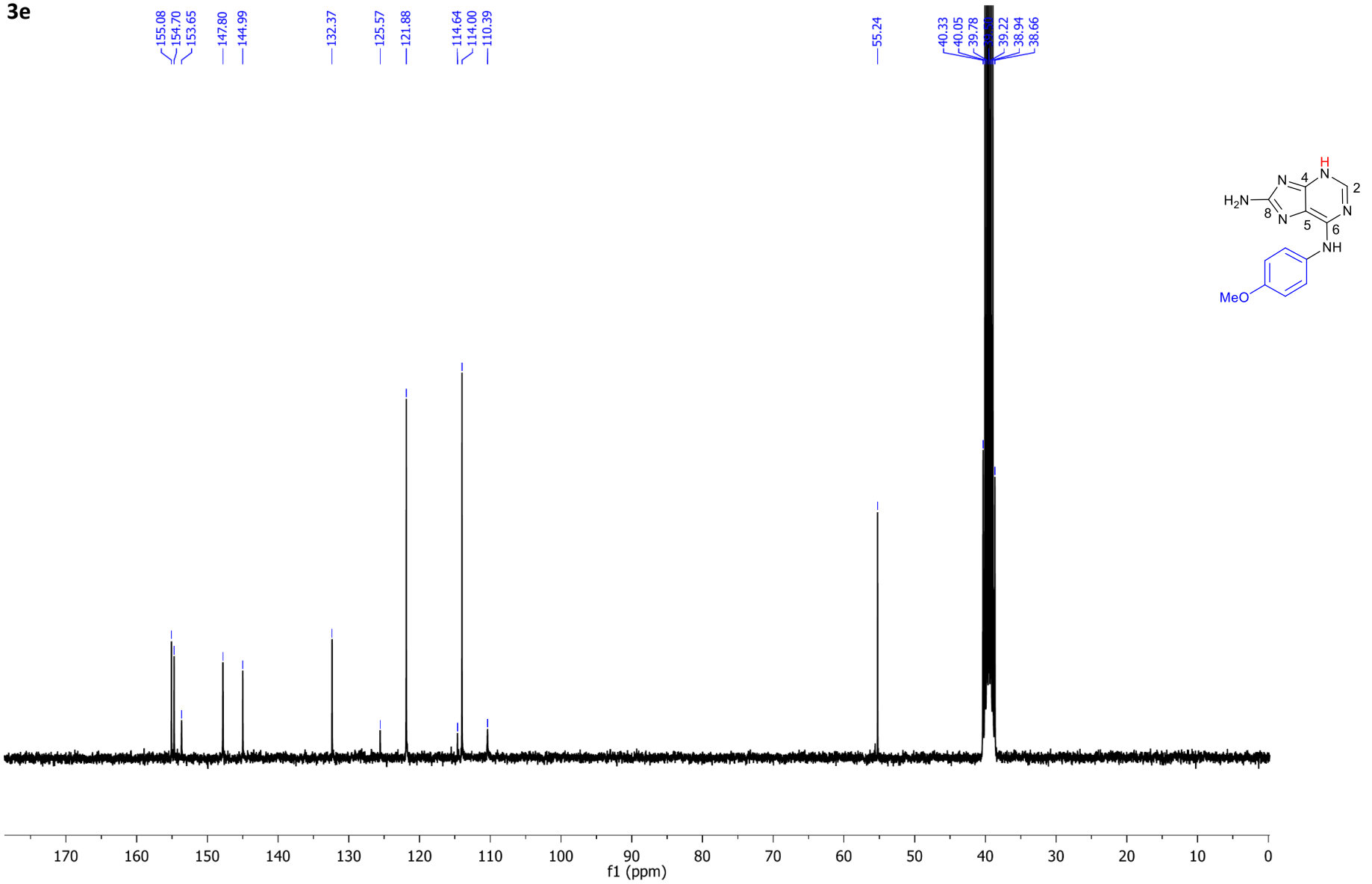



**Figure S2.25:** $^{13}$C NMR spectrum (100 MHz, DMSO-$d_6$) of $N^6$-(4-methoxyphenyl)-9*H*-purine-6,8-diamine sulfate (**3e**).

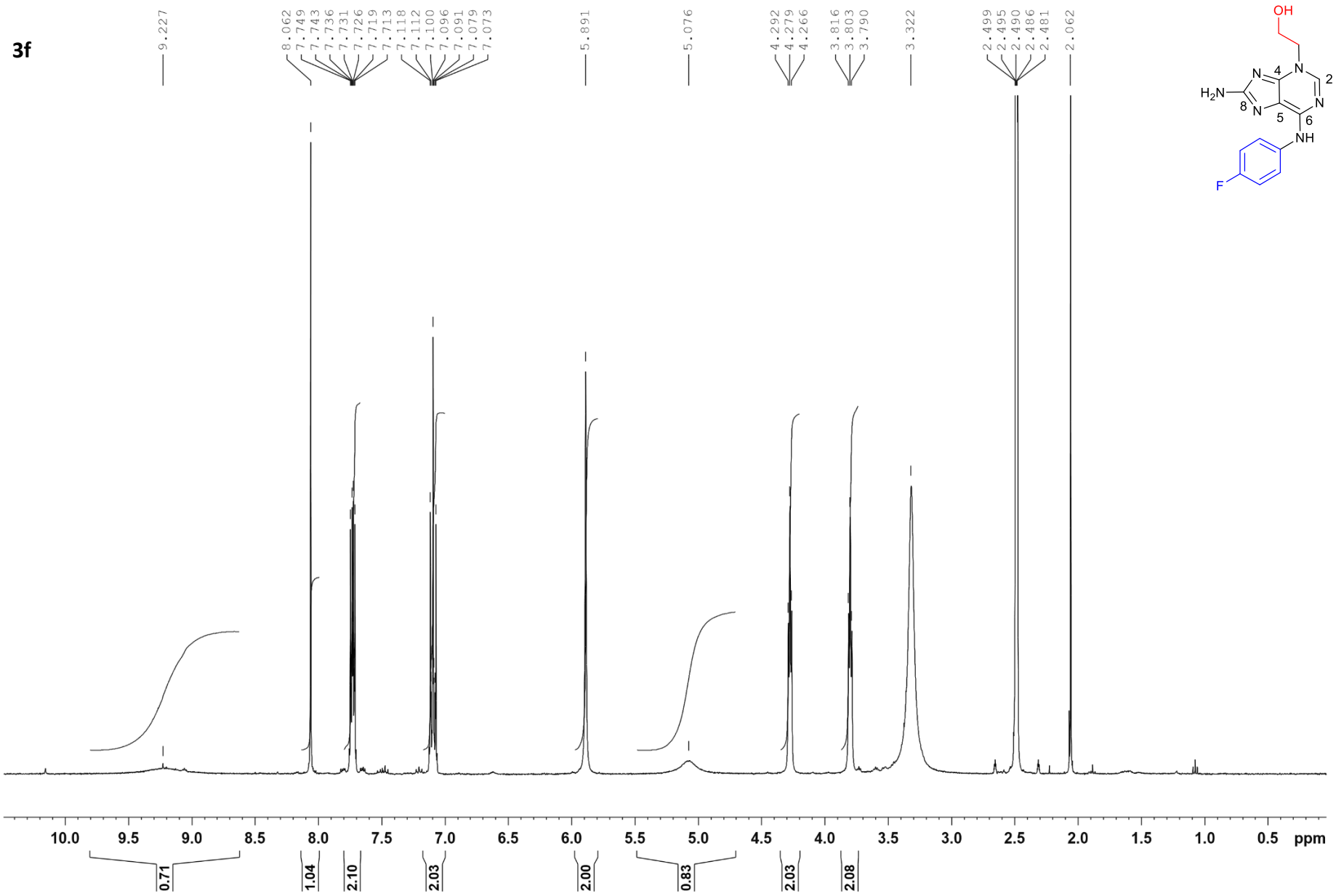



**Figure S2.26:** $^{1}$H NMR spectrum (400 MHz, DMSO-$d_6$) of 2-(8-amino-6-((4-fluorophenyl)amino)-3H-purin-3-yl)ethanol (**3f**).

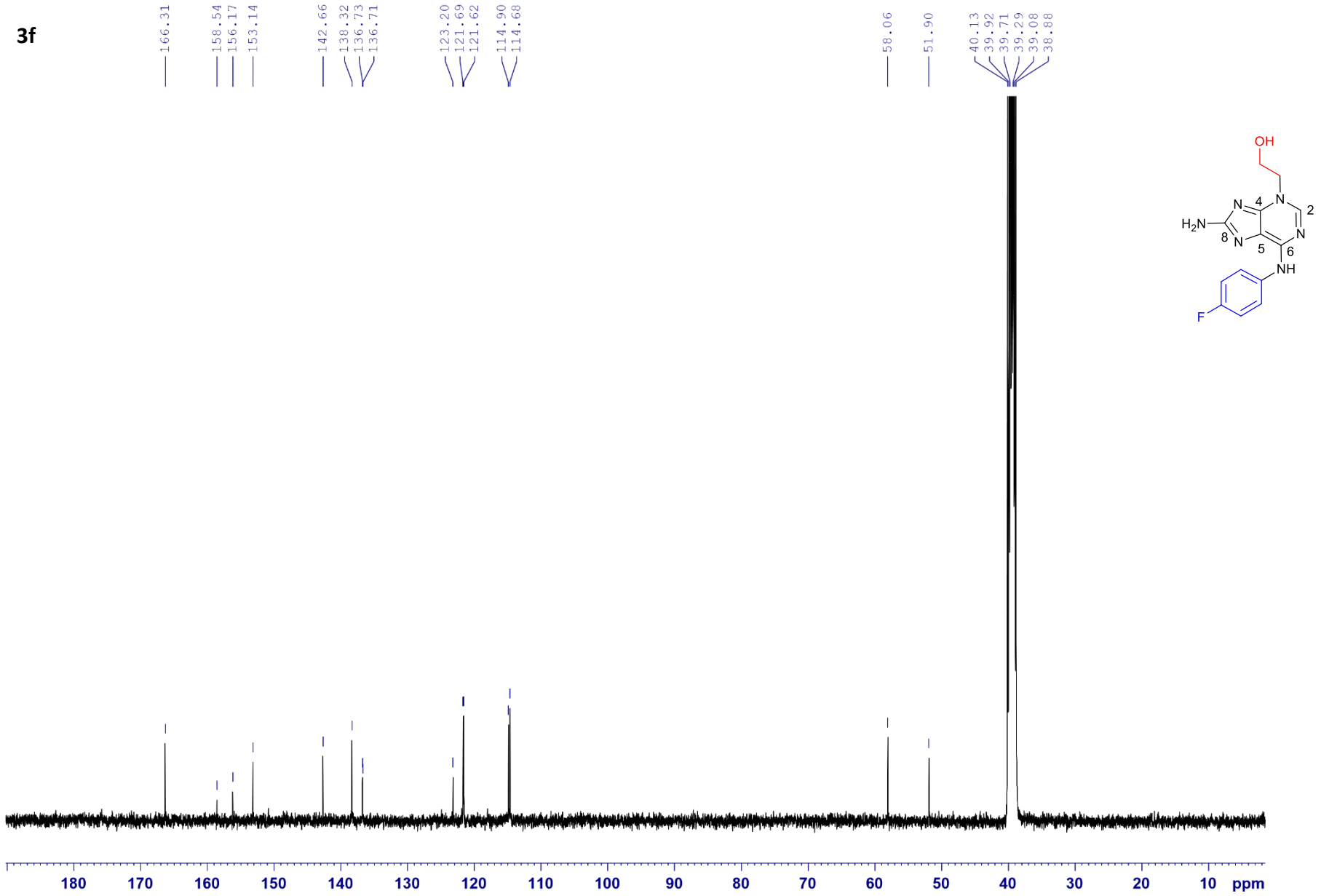



**Figure S2.27:** $^{13}$C NMR spectrum (100 MHz, DMSO-$d_6$) of 2-(8-amino-6-((4-fluorophenyl)amino)-3*H*-purin-3-yl)ethanol (**3f**).

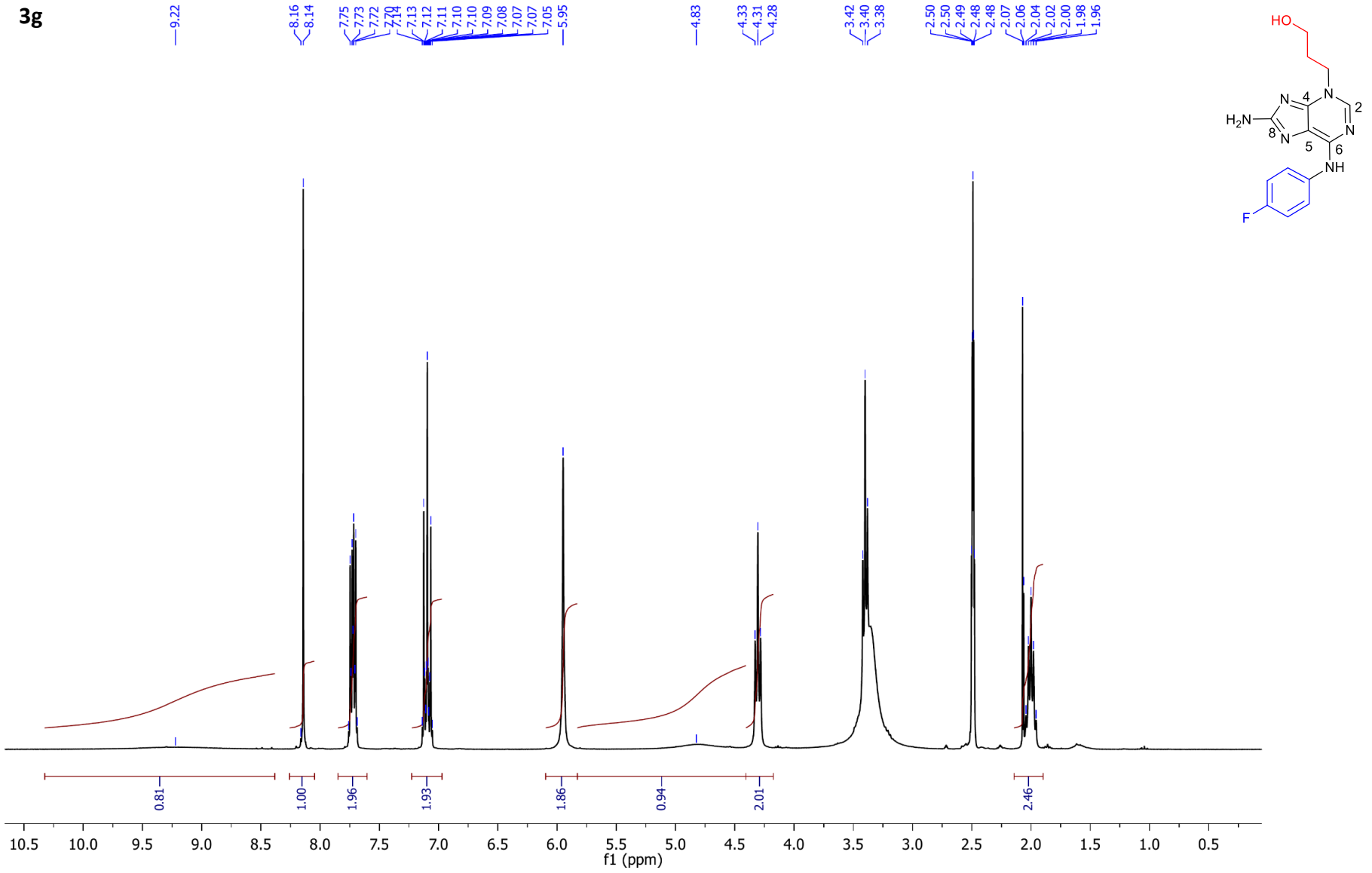



**Figure S2.28:** $^{1}$H NMR spectrum (400 MHz, DMSO-$d_6$) of 3-(8-amino-6-((4-fluorophenyl)amino)-3*H*-purin-3-yl)propan-1-ol (**3g**).

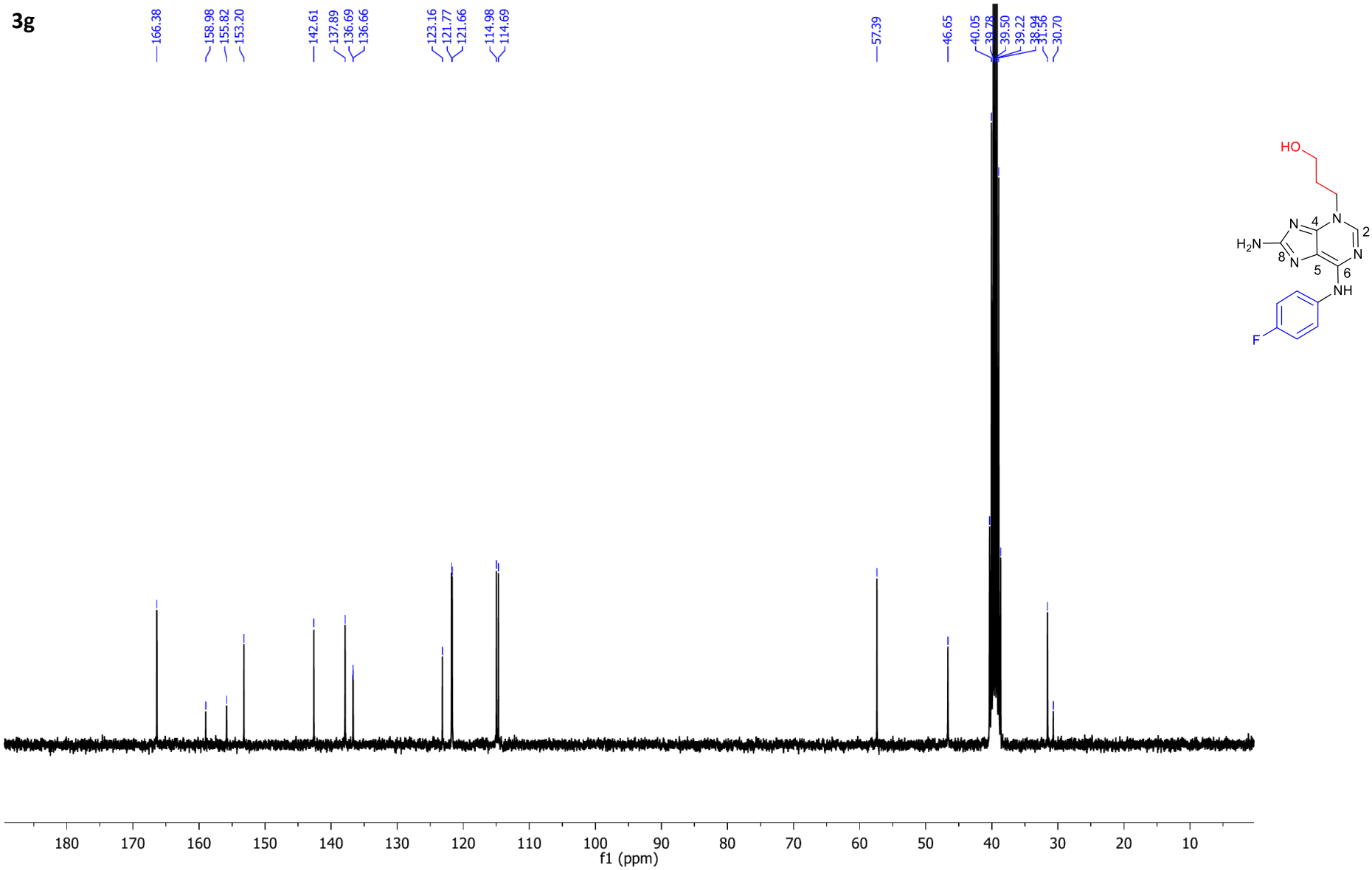



**Figure S2.29:** $^{13}$C NMR spectrum (100 MHz, DMSO-$d_6$) of 3-(8-amino-6-((4-fluorophenyl)amino)-3*H*-purin-3-yl)propan-1-ol (**3g**).

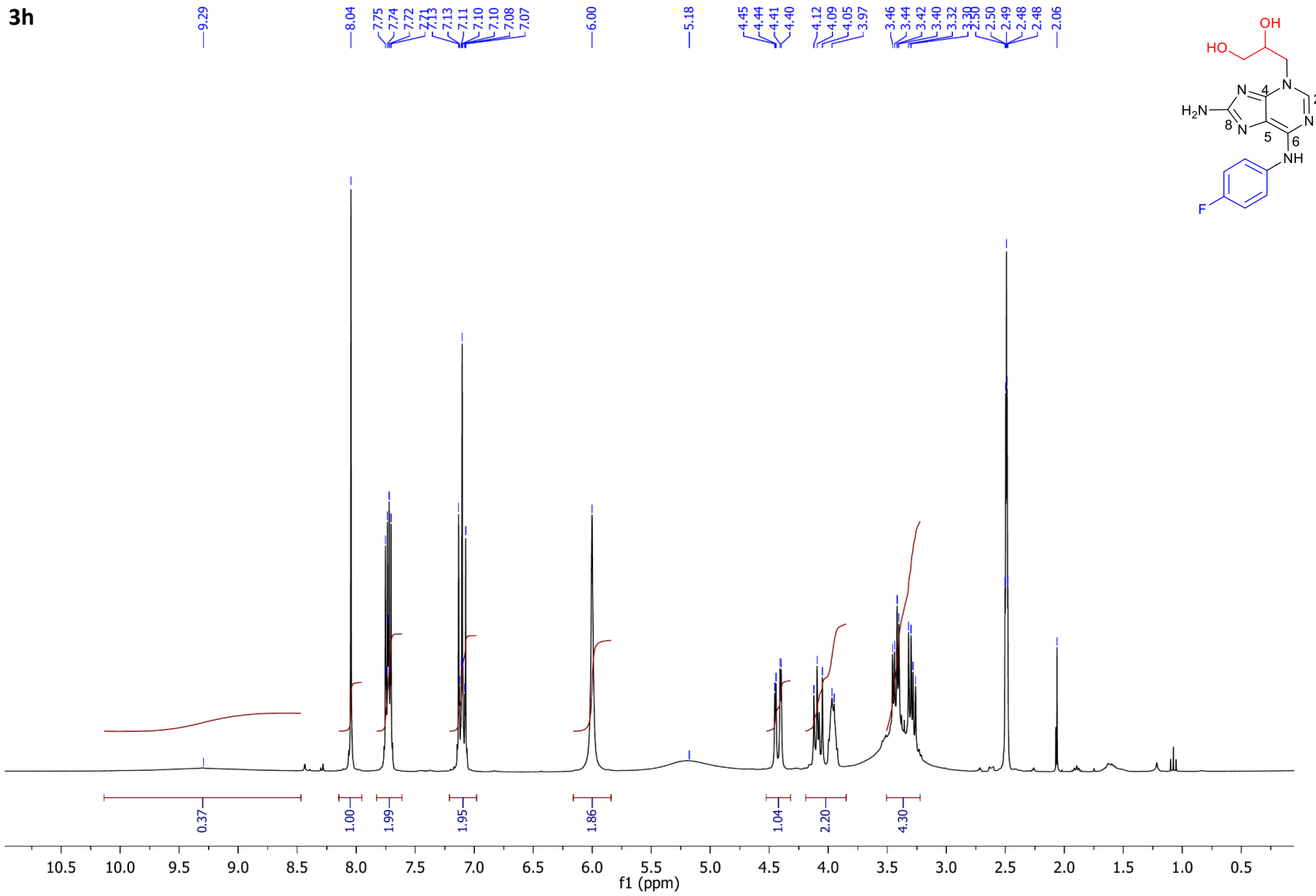



**Figure S2.30:** $^{1}$H NMR spectrum (400 MHz, DMSO-$d_6$) of 4-(8-amino-6-((4-fluorophenyl)amino)-3*H*-purin-3-yl)butane-1,3-diol (**3h**).

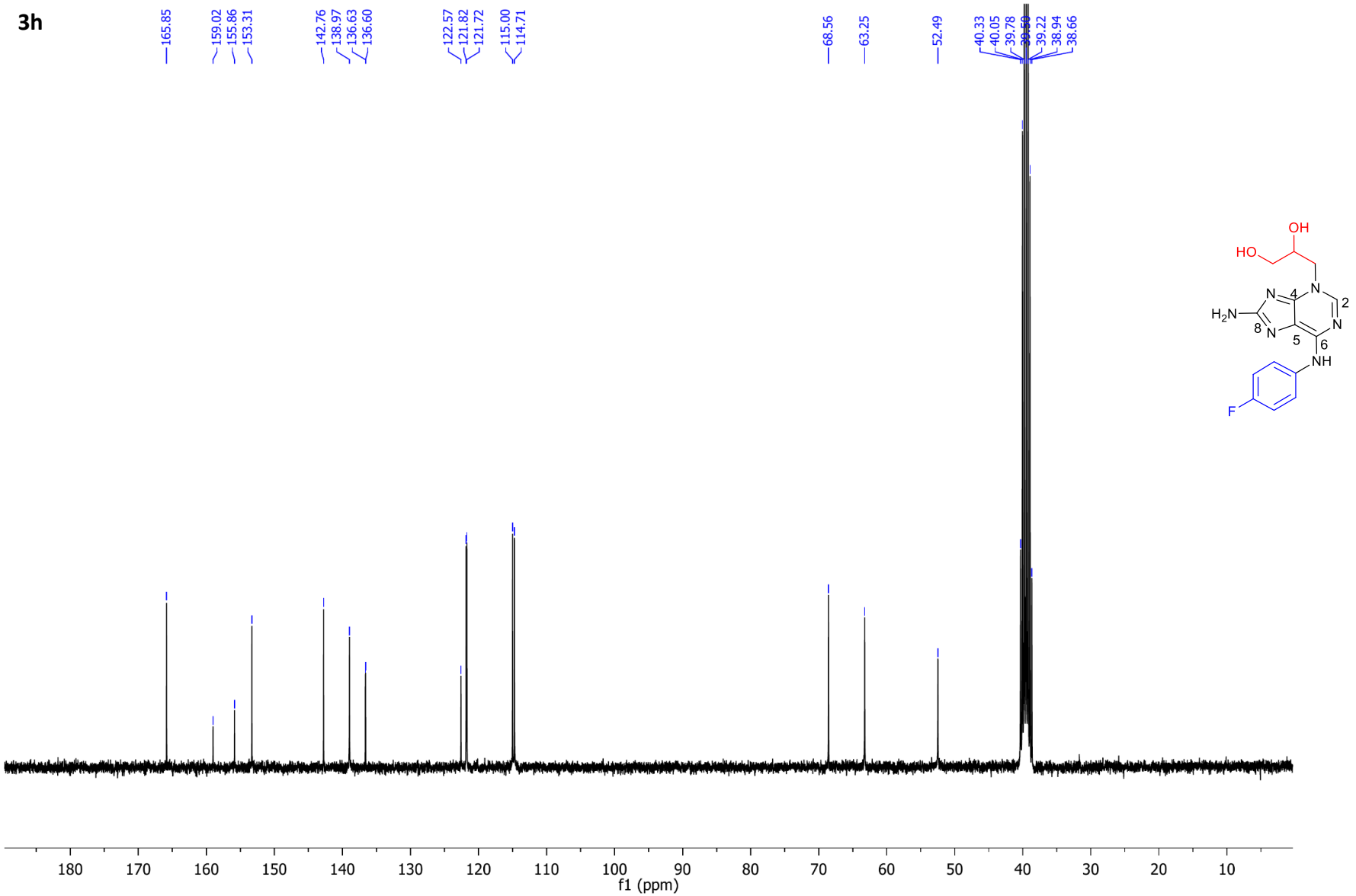



**Figure S2.31:** $^{13}$C NMR spectrum (100 MHz, DMSO-$d_6$) of 4-(8-amino-6-((4-fluorophenyl)amino)-3*H*-purin-3-yl)butane-1,3-diol (**3h**).

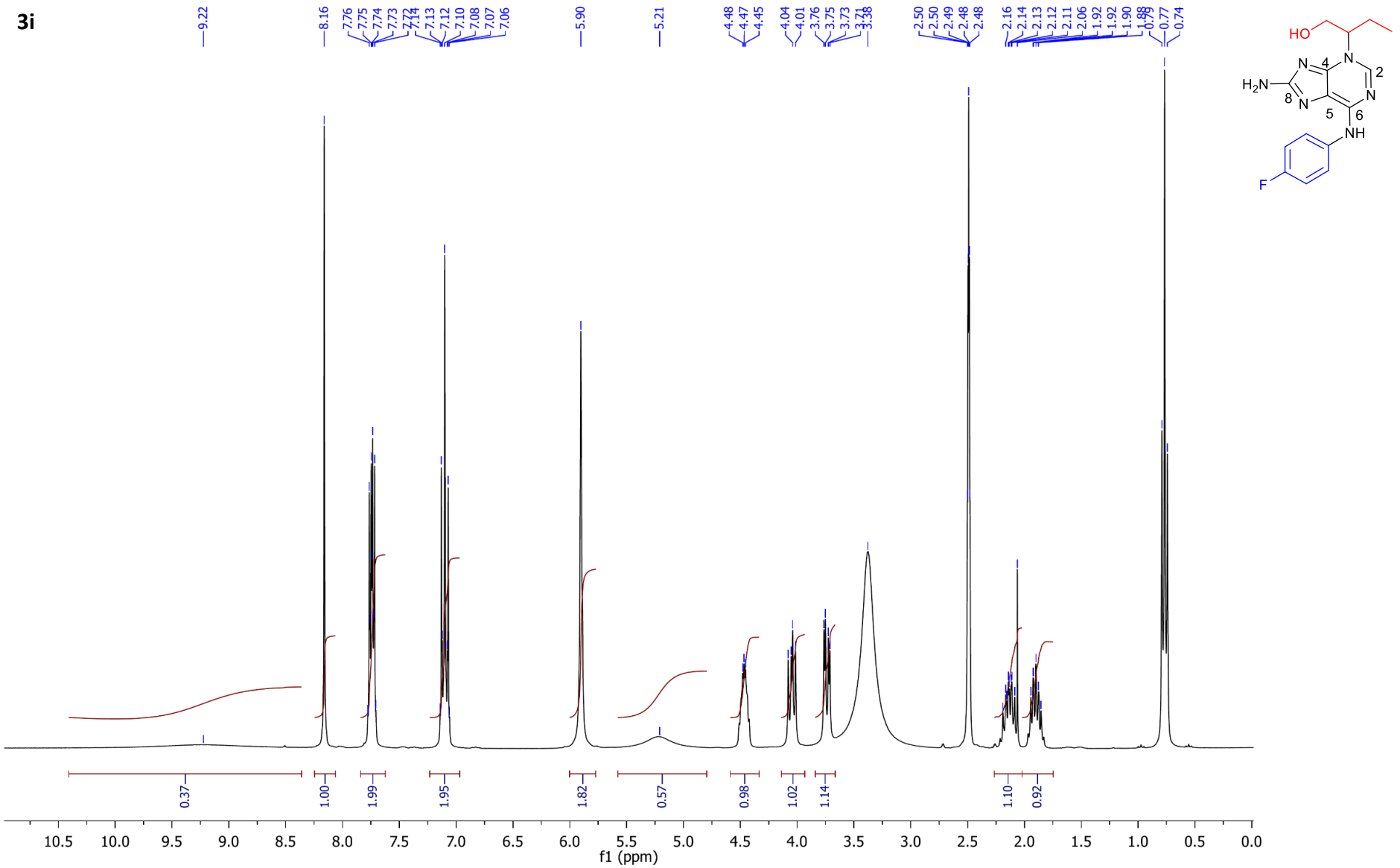



**Figure S2.32:** $^{1}$H NMR spectrum (400 MHz, DMSO-$d_6$) of 2-(8-amino-6-(4'-fluorophenylamino)-3*H*-purin-3-yl)butan-1-ol (**3i**).

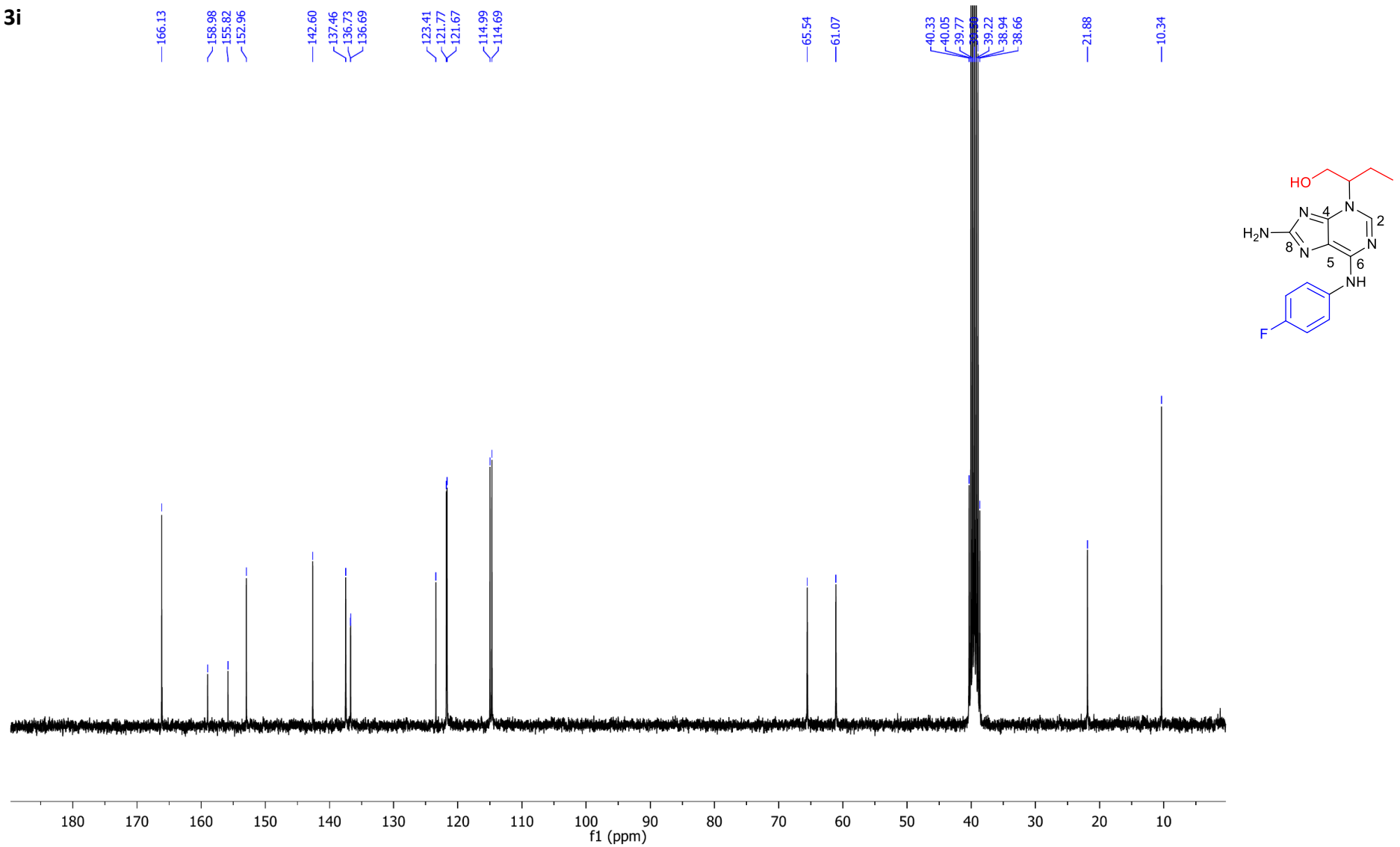



**Figure S2.33:** $^{13}$C NMR spectrum (100 MHz, DMSO-$d_6$) of 2-(8-amino-6-(4'-fluorophenylamino)-3*H*-purin-3-yl)butan-1-ol (**3i**).

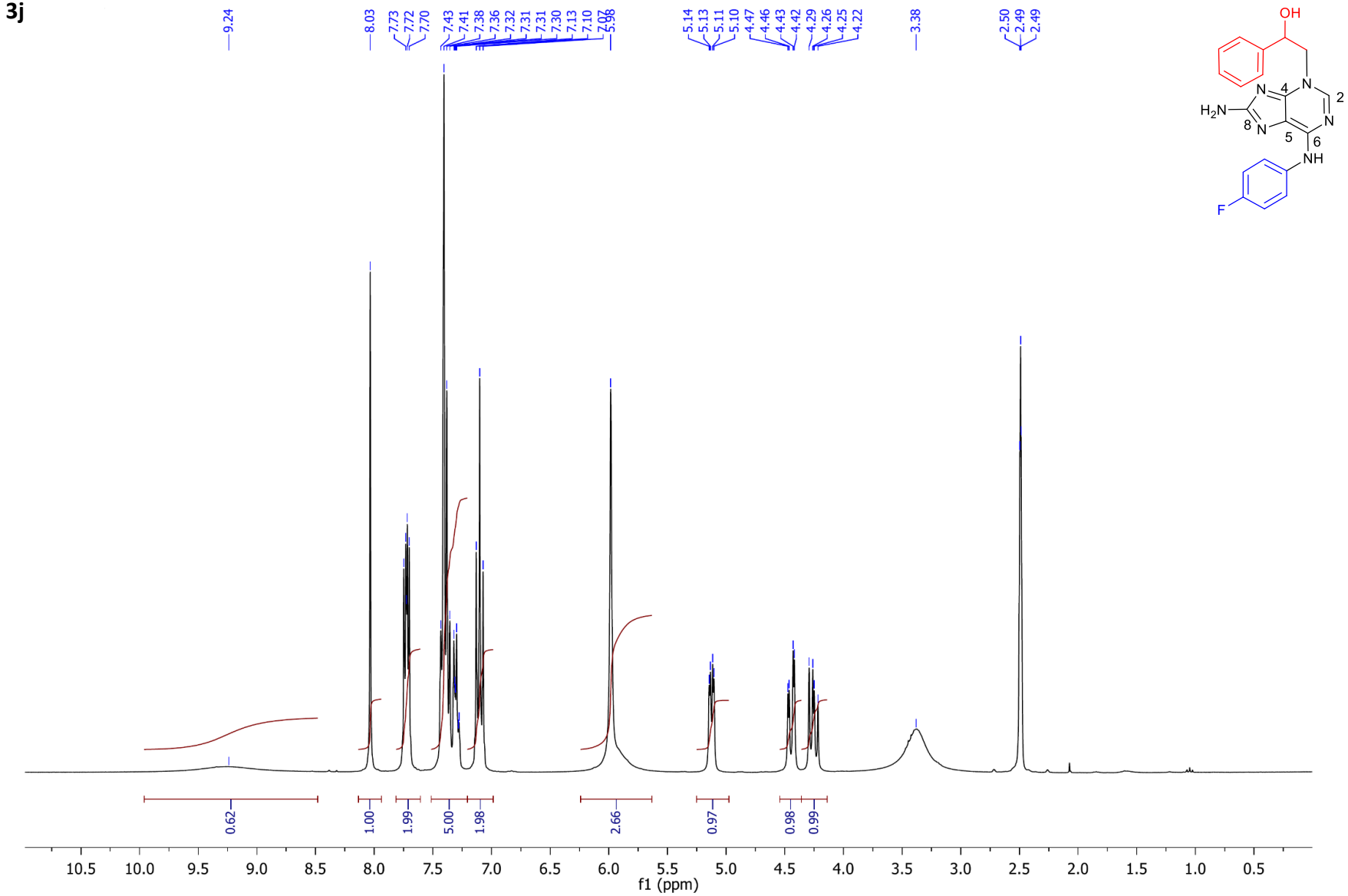



**Figure S2.34:** $^{1}$H NMR spectrum (400 MHz, DMSO-$d_6$) of 2-(8-amino-6-((4-fluorophenyl)amino)-3*H*-purin-3-yl)-1-phenylethanol (**3j**).

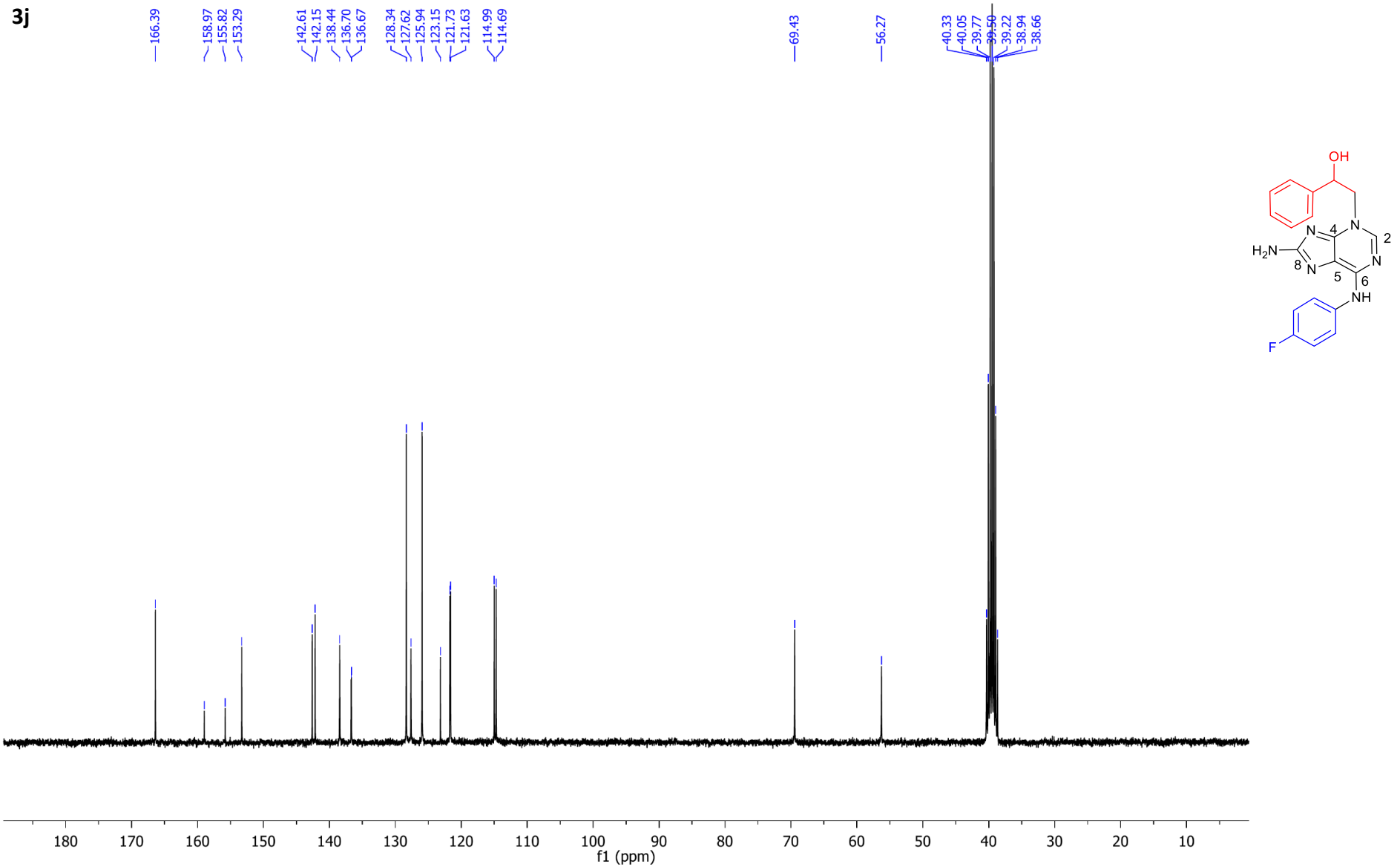



**Figure S2.35:** $^{13}$C NMR spectrum (100 MHz, DMSO-$d_6$) of 2-(8-amino-6-((4-fluorophenyl)amino)-3*H*-purin-3-yl)-1-phenylethanol (**3j**).

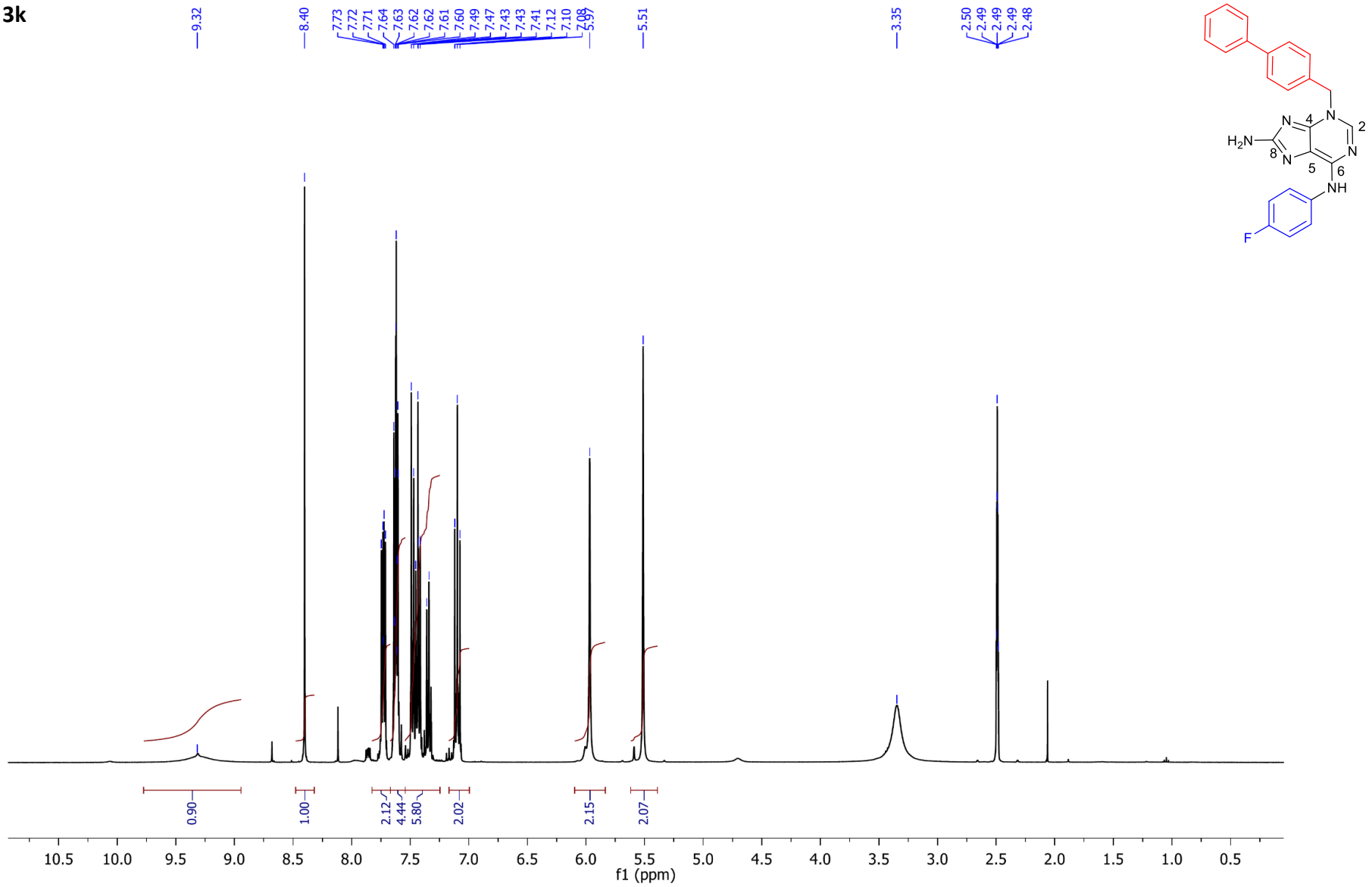



**Figure S2.36:** $^{1}$H NMR spectrum (400 MHz, DMSO-$d_6$) of 3-([1,1'-biphenyl]-4-ylmethyl)-$N^6$-(4-fluorophenyl)-3*H*-purine-6,8-diamine (**3k**).

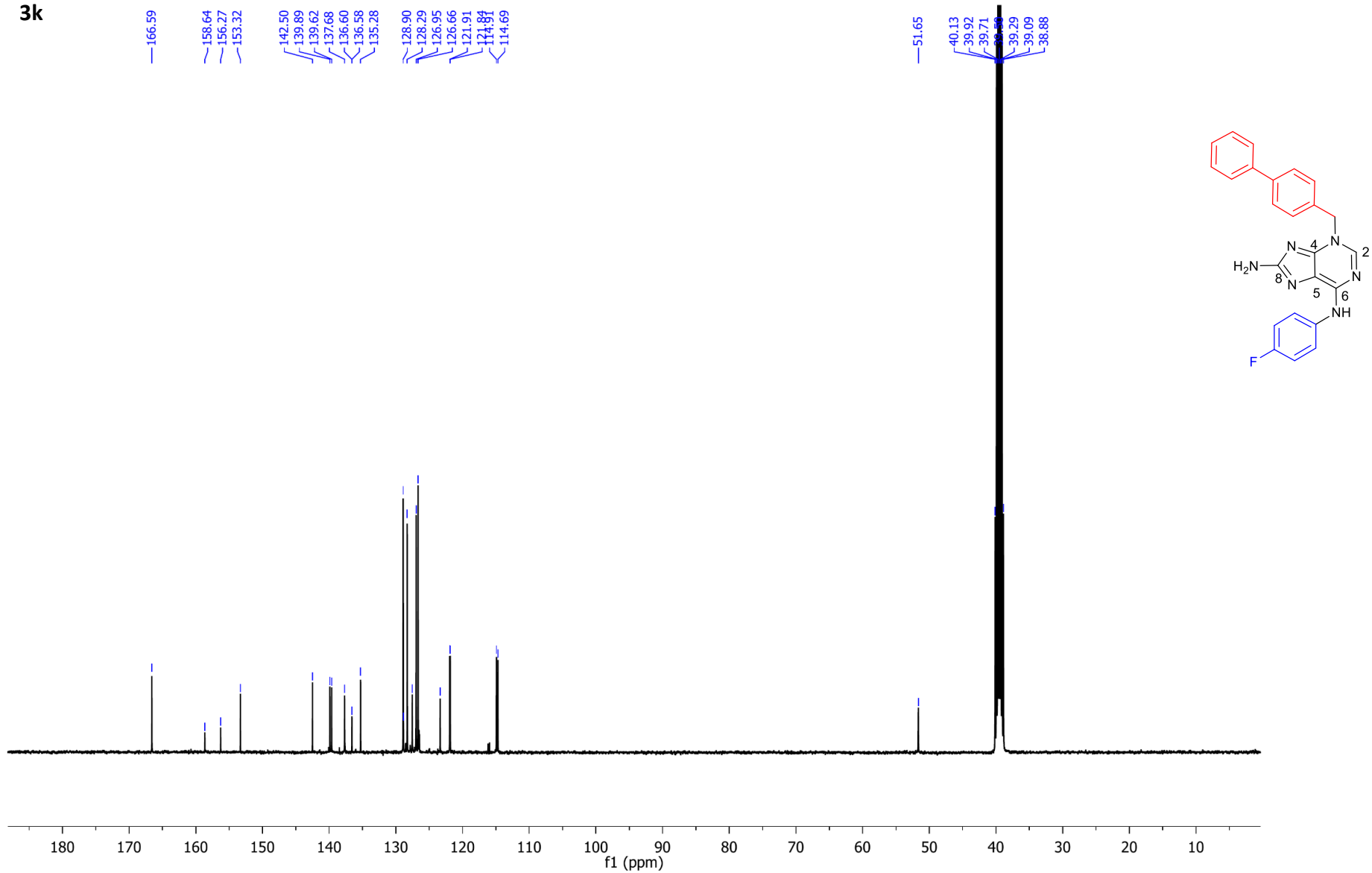



**Figure S2.37:** $^{13}$C NMR spectrum (100 MHz, DMSO-$d_6$) of 3-([1,1'-biphenyl]-4-ylmethyl)-$N^6$-(4-fluorophenyl)-3*H*-purine-6,8-diamine (**3k**).

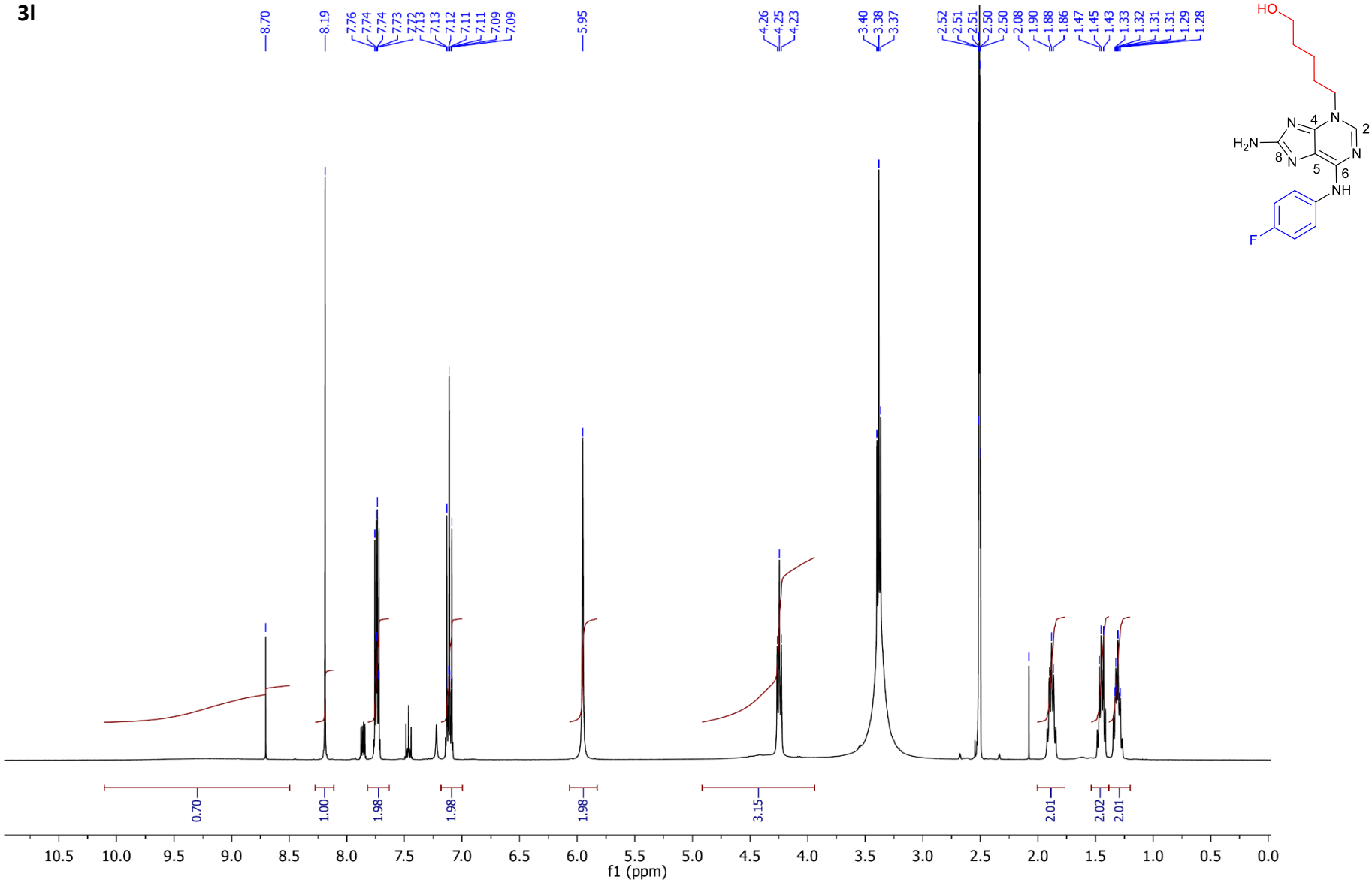



**Figure S2.38:** $^{1}$H NMR spectrum (400 MHz, DMSO-$d_6$) of 5-(8-amino-6-((4-fluorophenyl)amino)-3*H*-purin-3-yl)pentan-1-ol (**3l**).

**3l**

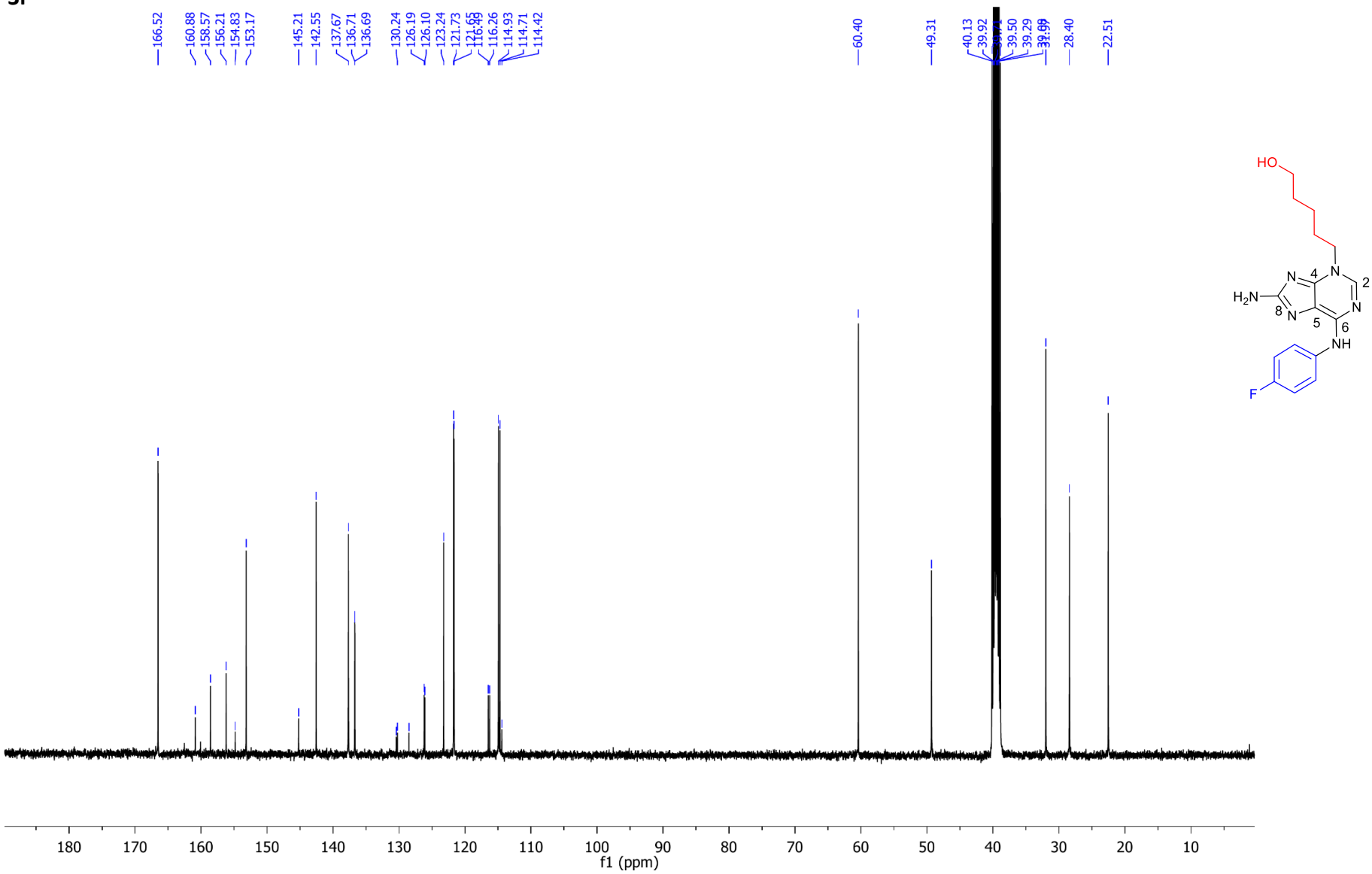



**Figure S2.39:** $^{13}$C NMR spectrum (100 MHz, DMSO-$d_6$) of 5-(8-amino-6-((4-fluorophenyl)amino)-3*H*-purin-3-yl)pentan-1-ol (**3l**).

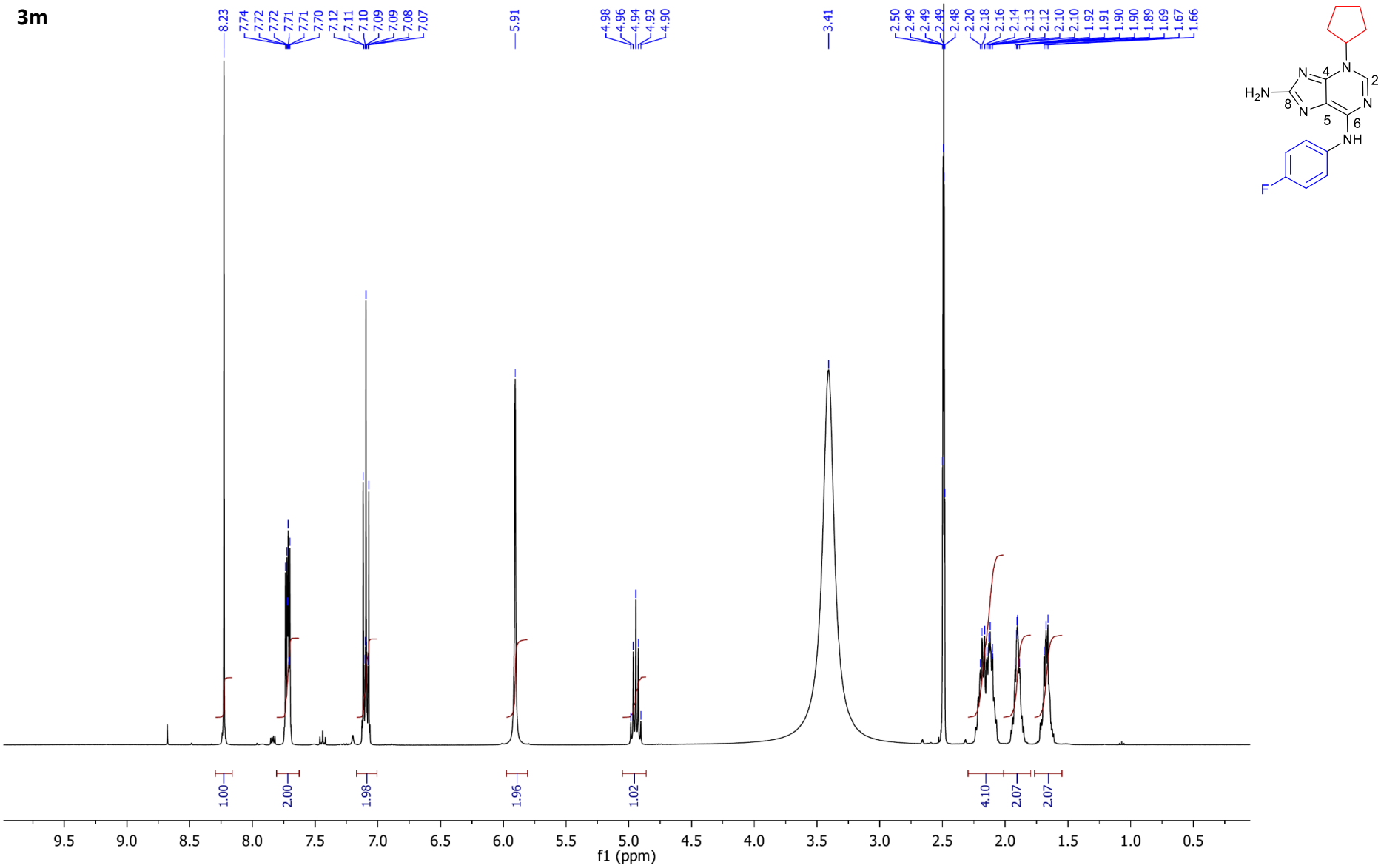



**Figure S2.40:** $^{1}$H NMR spectrum (400 MHz, DMSO-$d_6$) of 3-cyclopentyl-$N^6$-(4-fluorophenyl)-3*H*-purine-6,8-diamine (**3m**).

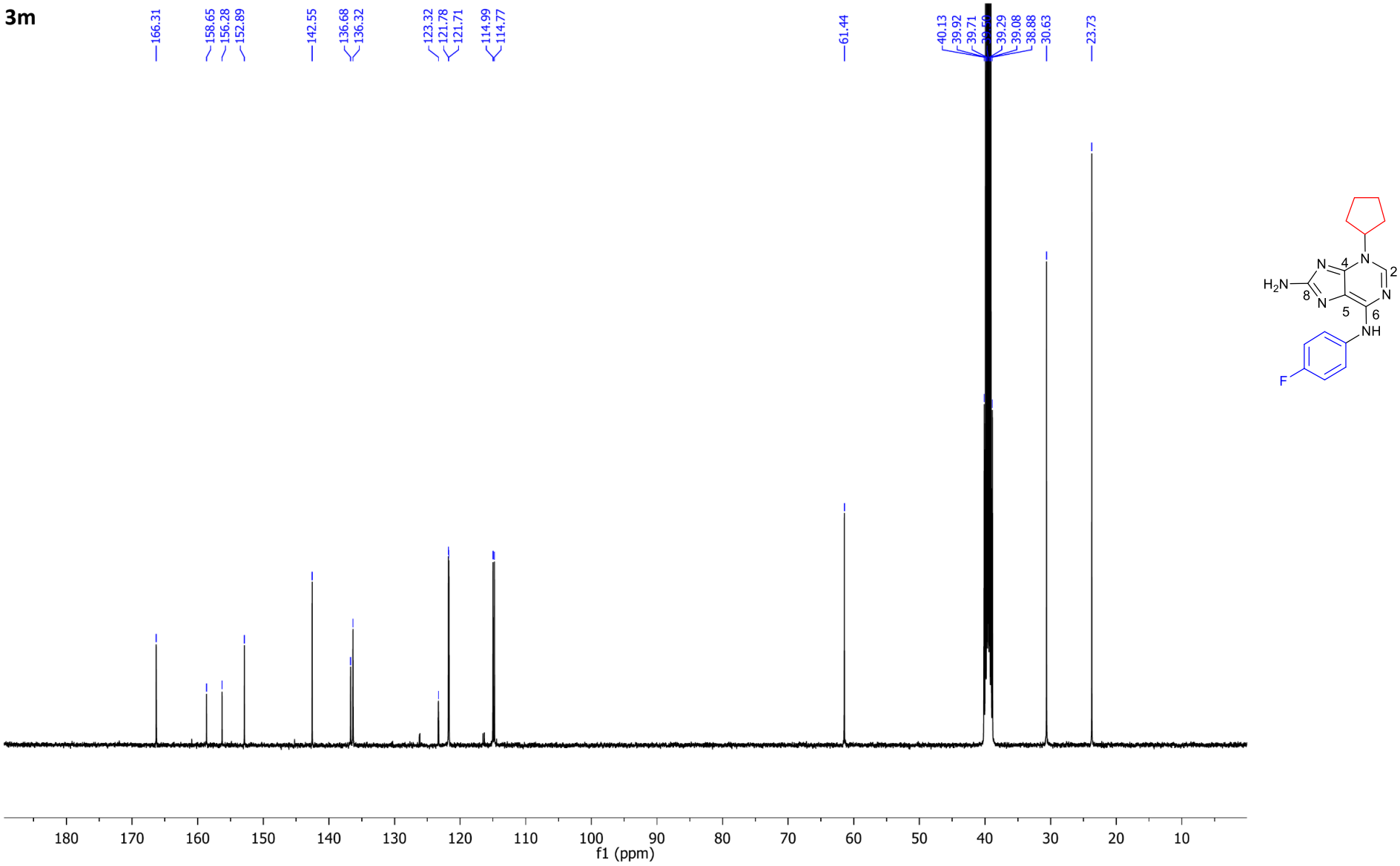



**Figure S2.41:** $^{13}$C NMR spectrum (100 MHz, DMSO-$d_6$) of 3-cyclopentyl-$N^6$-(4-fluorophenyl)-3*H*-purine-6,8-diamine (**3m**).

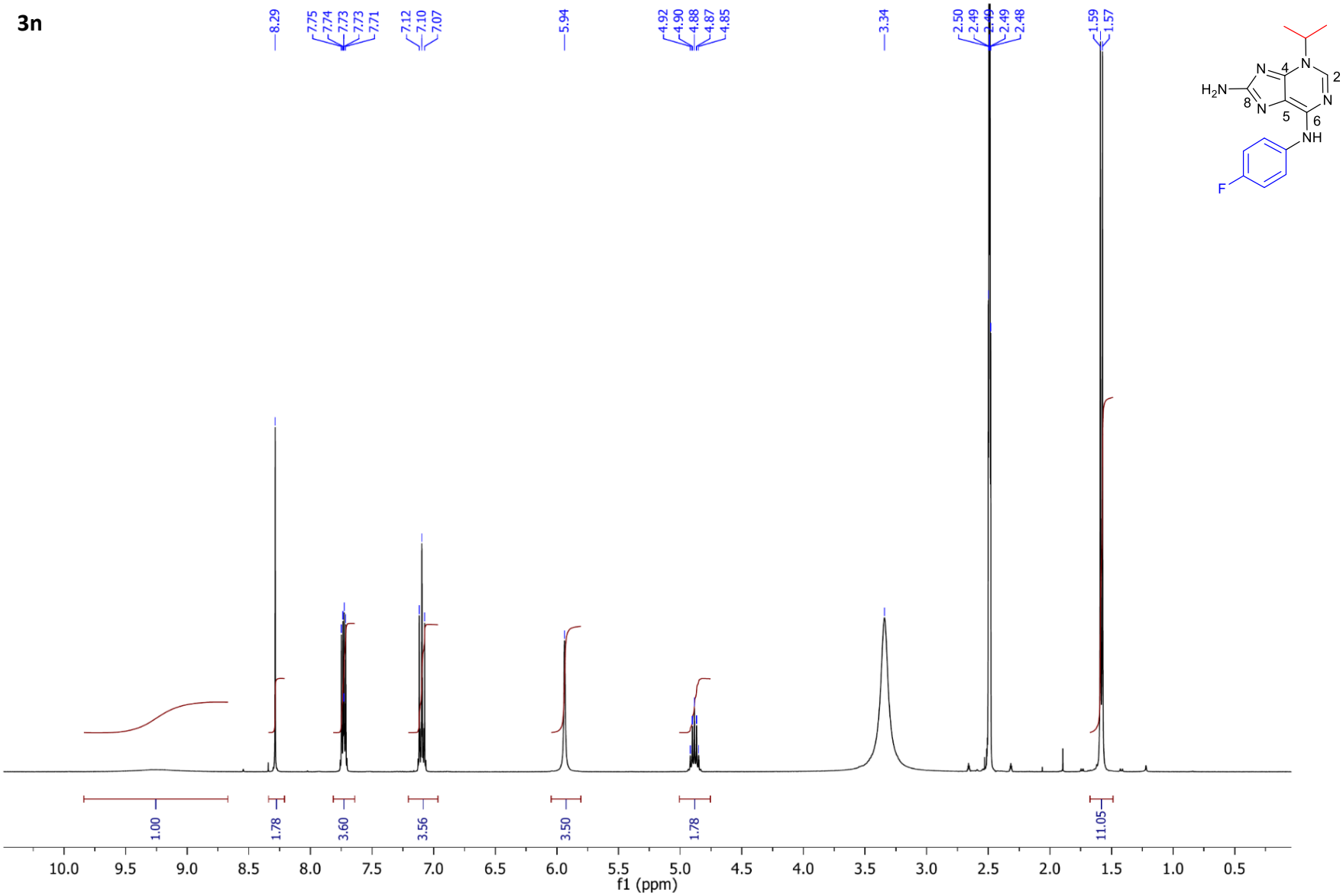



**Figure S2.42:** $^{1}$H NMR spectrum (400 MHz, DMSO-$d_6$) of $N^6$-(4-fluorophenyl)-3-isopropyl-3*H*-purine-6,8-diamine (**3n**).

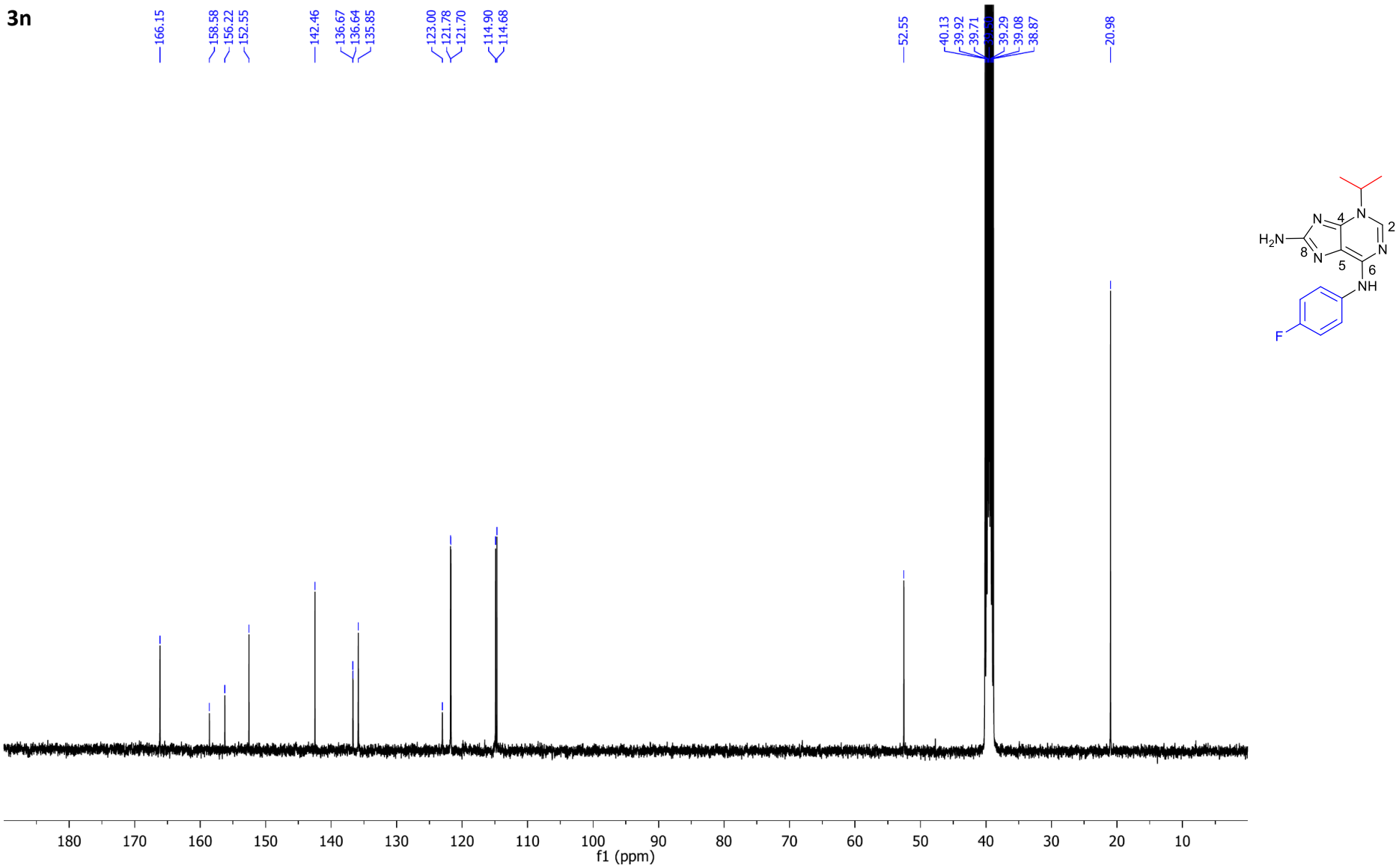



**Figure S2.43:** $^{13}$C NMR spectrum (100 MHz, DMSO-$d_6$) of $N^6$-(4-fluorophenyl)-3-isopropyl-3*H*-purine-6,8-diamine (**3n**).

**3o.TFA**

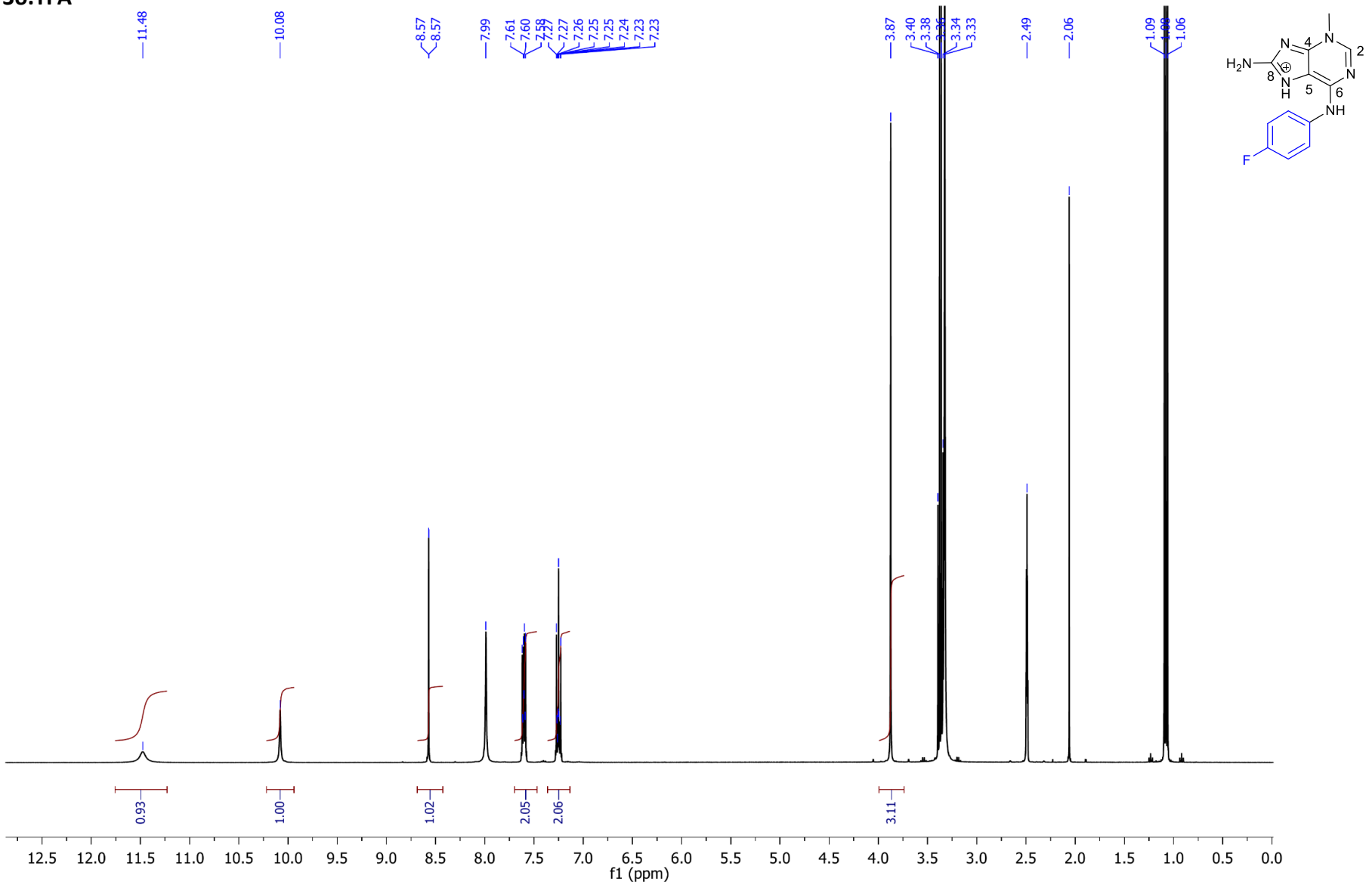



**Figure S2.44:** $^{1}$H NMR spectrum (400 MHz, DMSO-$d_6$) of $N^6$-(4-fluorophenyl)-3-methyl-3*H*-purine-6,8-diamine 2,2,2-trifluoroacetate (**3o.TFA**).

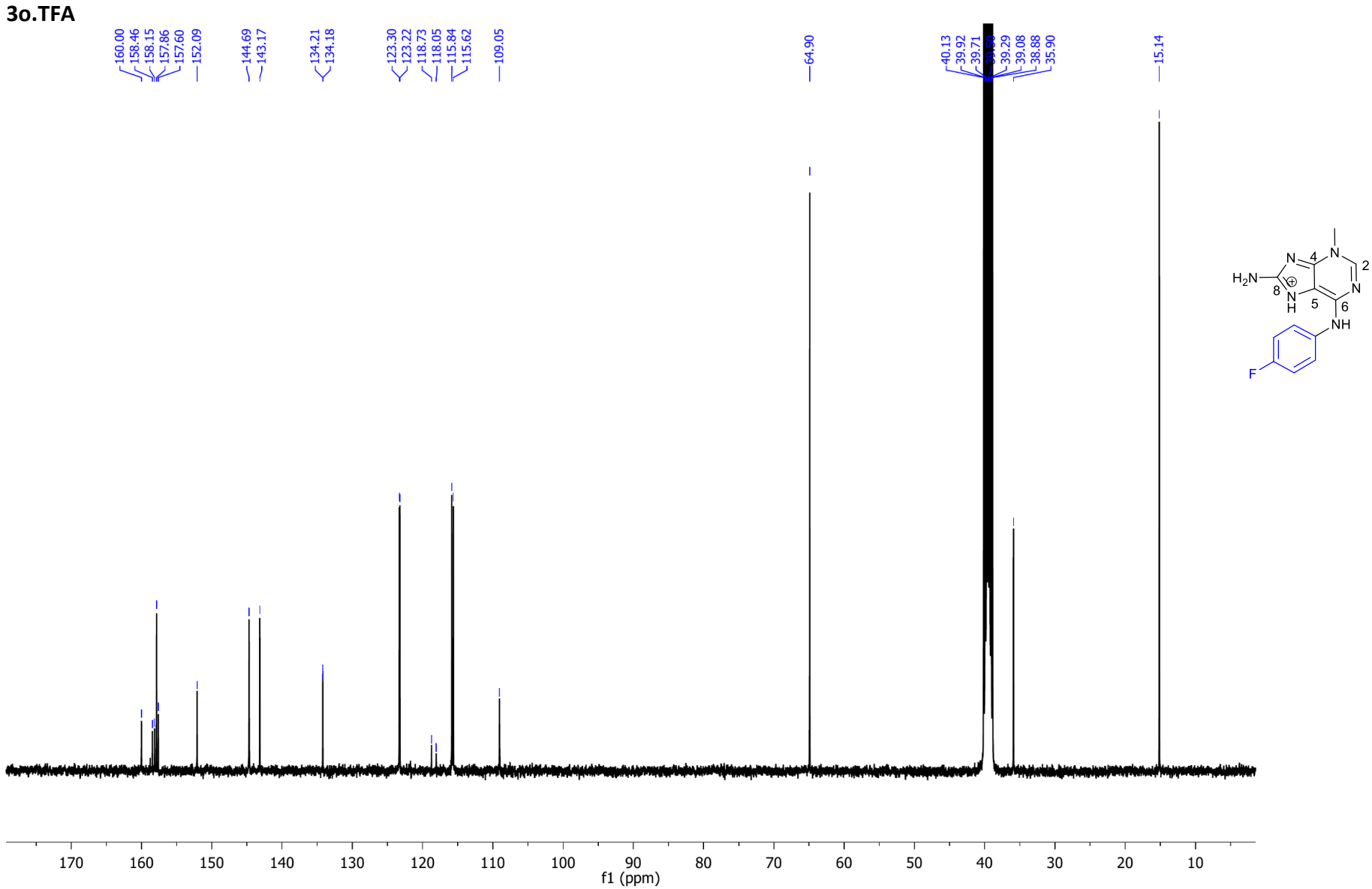



**Figure S2.45:** $^{13}$C NMR spectrum (100 MHz, DMSO-$d_6$) of $N^6$-(4-fluorophenyl)-3-methyl-3*H*-purine-6,8-diamine 2,2,2-trifluoroacetate (**3o.TFA**).

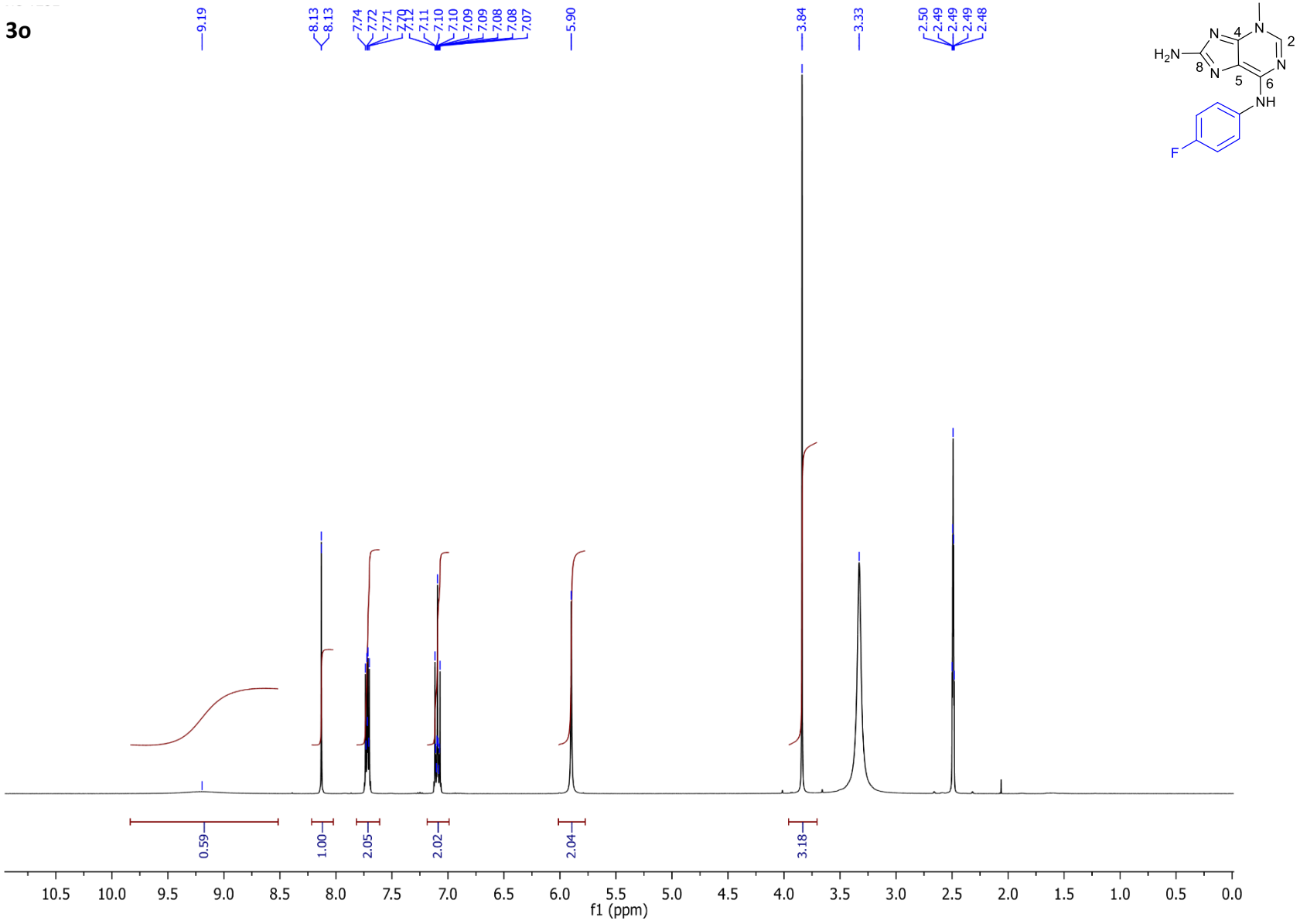



**Figure S2.46:** $^{1}$H NMR spectrum (400 MHz, DMSO-$d_6$) of $N^6$-(4-fluorophenyl)-3-methyl-3*H*-purine-6,8-diamine (**3o**).

**3o**

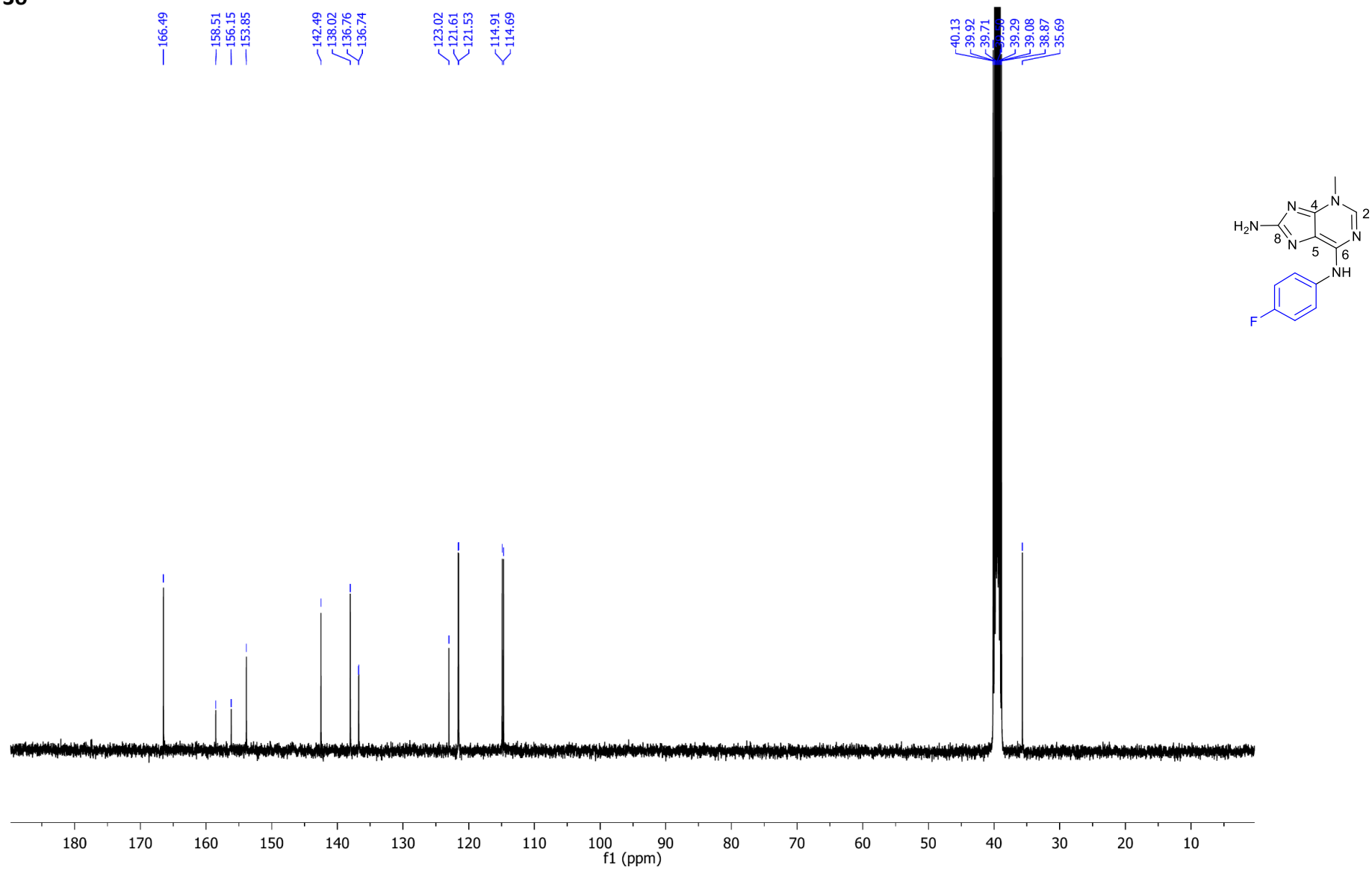



**Figure S2.47:** $^{13}$C NMR spectrum (100 MHz, DMSO-$d_6$) of $N^6$-(4-fluorophenyl)-3-methyl-3*H*-purine-6,8-diamine (**3o**).

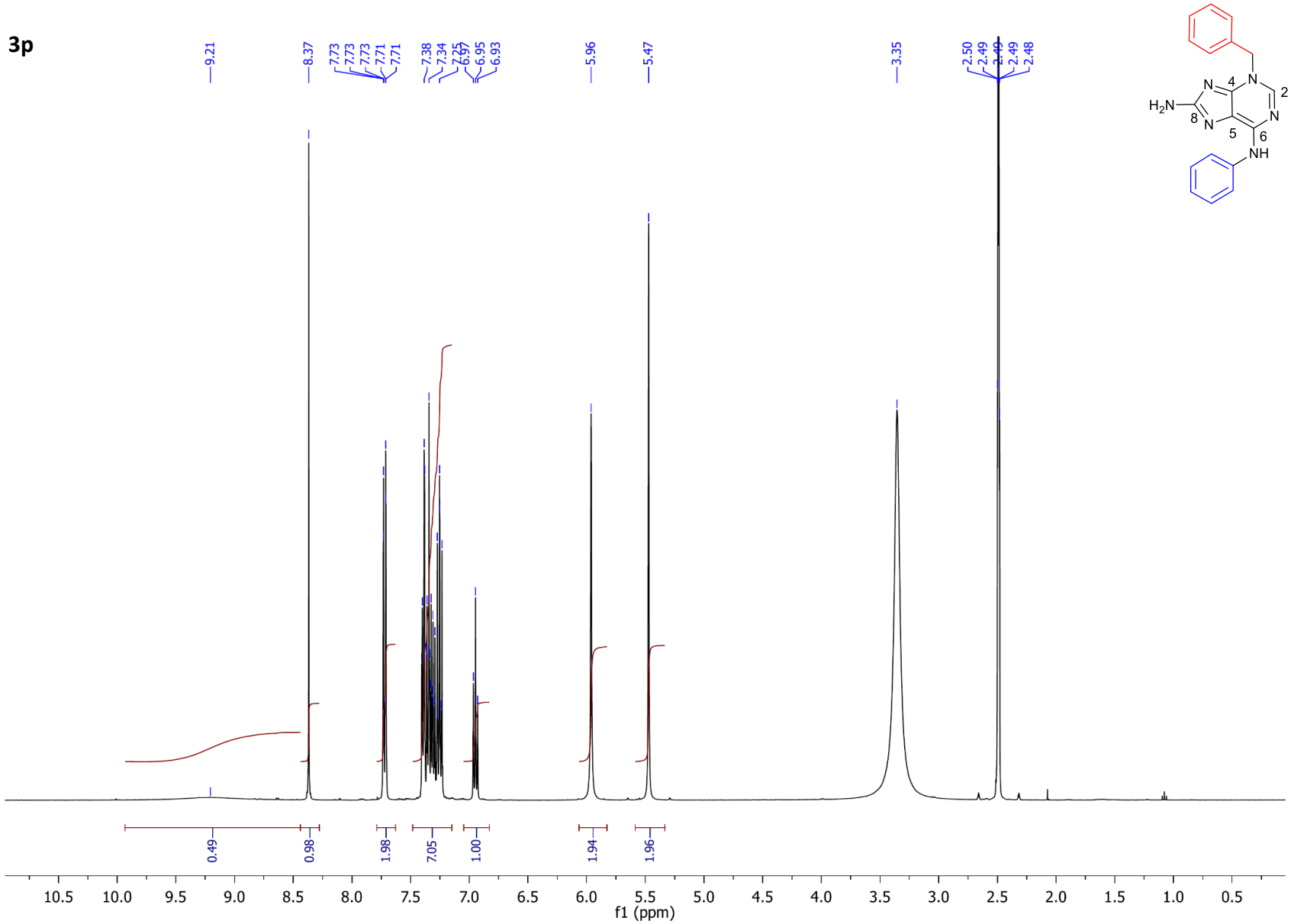



**Figure S2.48:** $^{1}$H NMR spectrum (400 MHz, DMSO-$d_6$) of 3-benzyl-$N^6$-phenyl-3*H*-purine-6,8-diamine (**3p**).

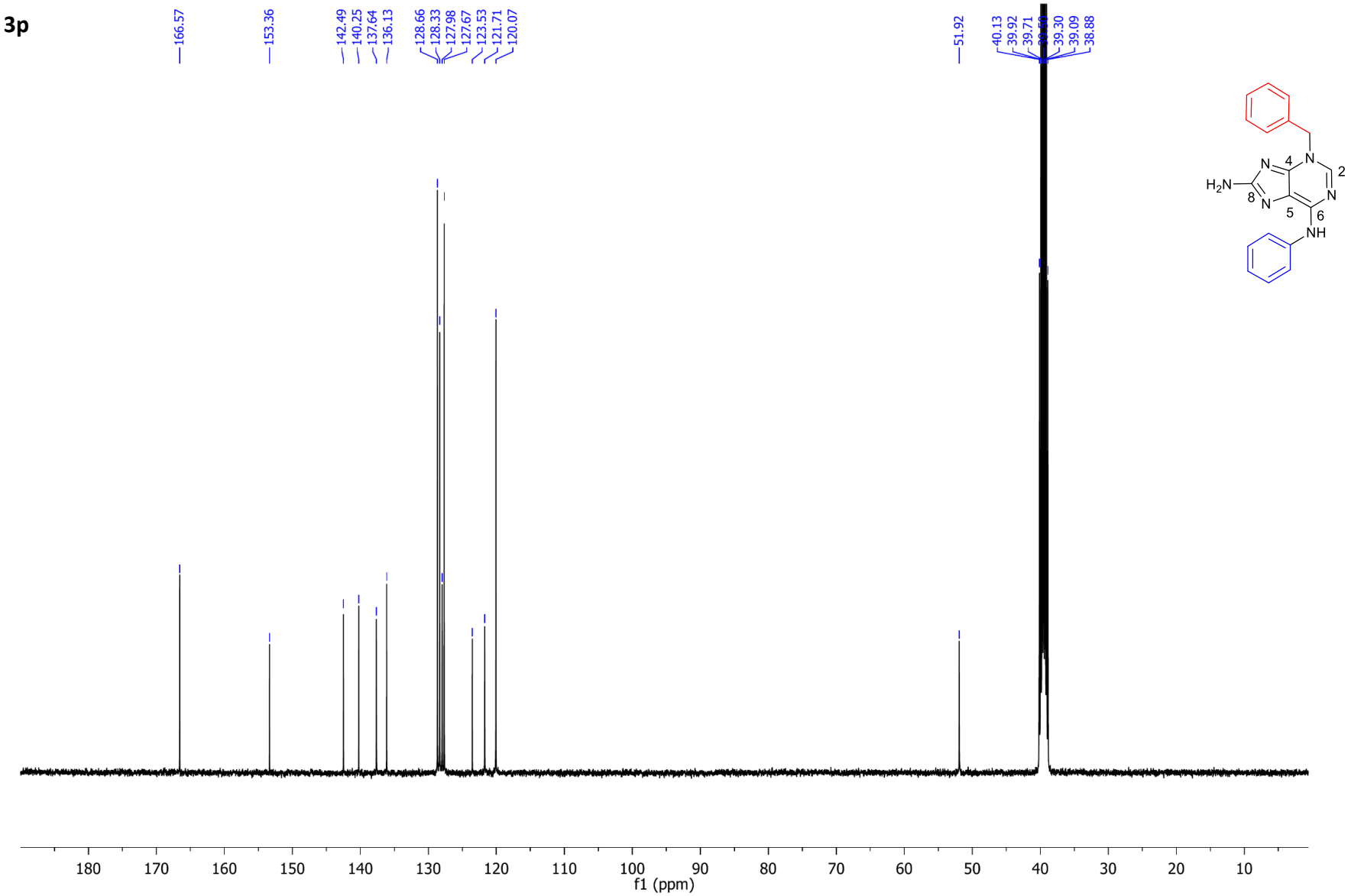



**Figure S2.49:** $^{13}$C NMR spectrum (100 MHz, DMSO-$d_6$) of 3-benzyl-$N^6$-phenyl-3*H*-purine-6,8-diamine (**3p**).

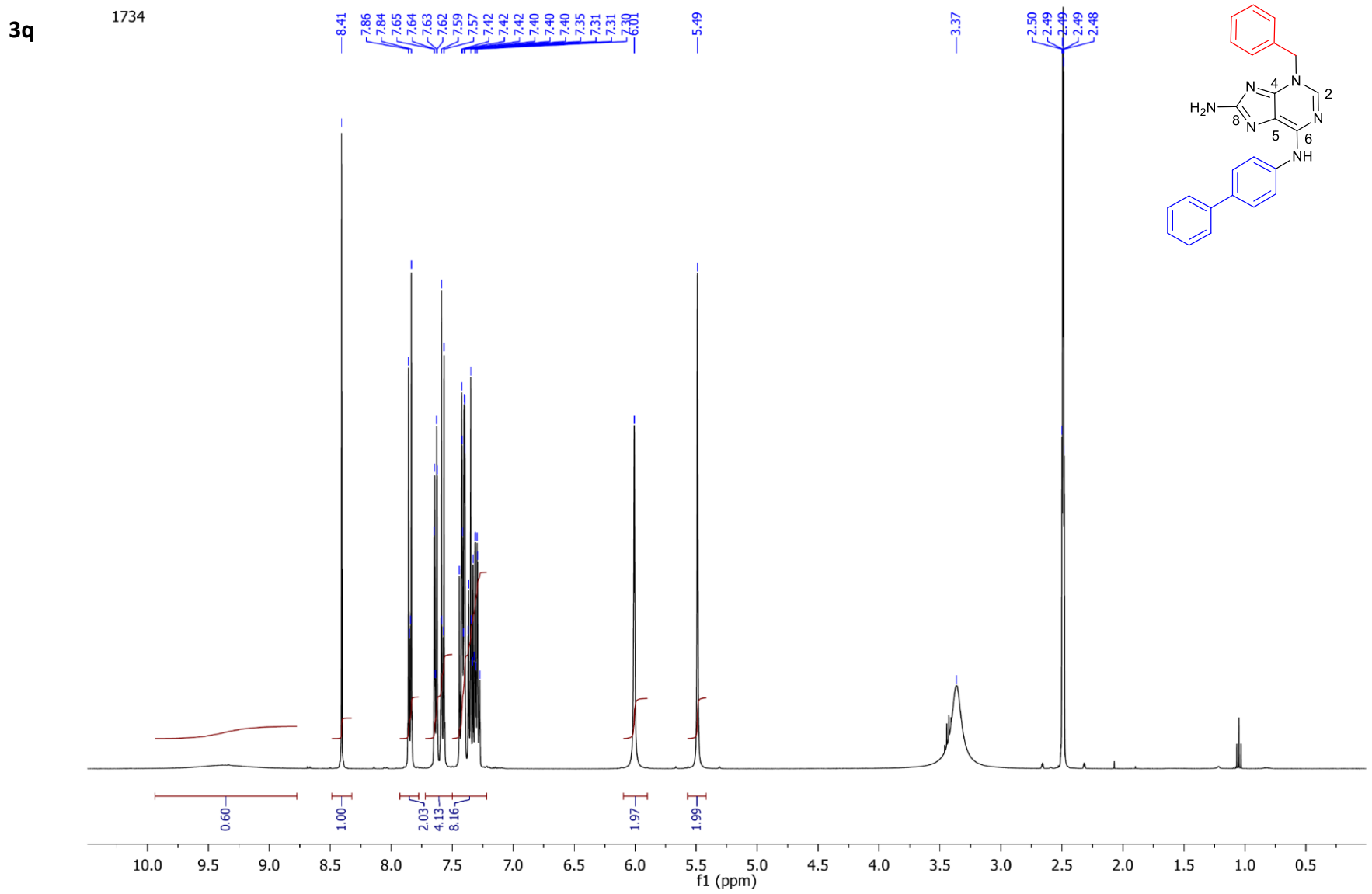



**Figure S2.50:** $^{1}$H NMR spectrum (400 MHz, DMSO-$d_6$) of $N^6$-([1,1'-biphenyl]-4-yl)-3-benzyl-3*H*-purine-6,8-diamine (**3q**).

**3q**

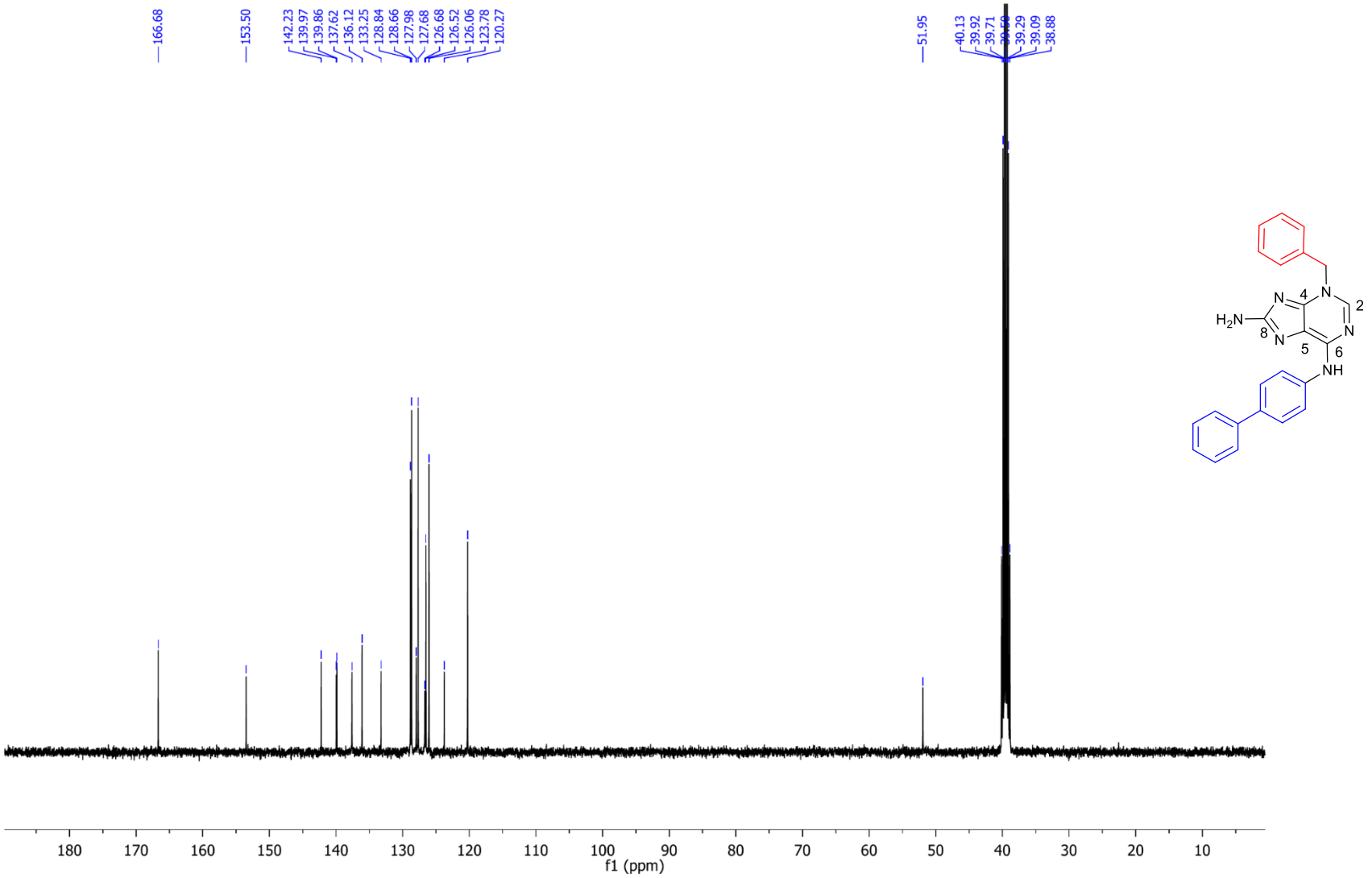



**Figure S2.51:** $^{13}$C NMR spectrum (100 MHz, DMSO-$d_6$) of $N^6$-([1,1'-biphenyl]-4-yl)-3-benzyl-3*H*-purine-6,8-diamine (**3q**).

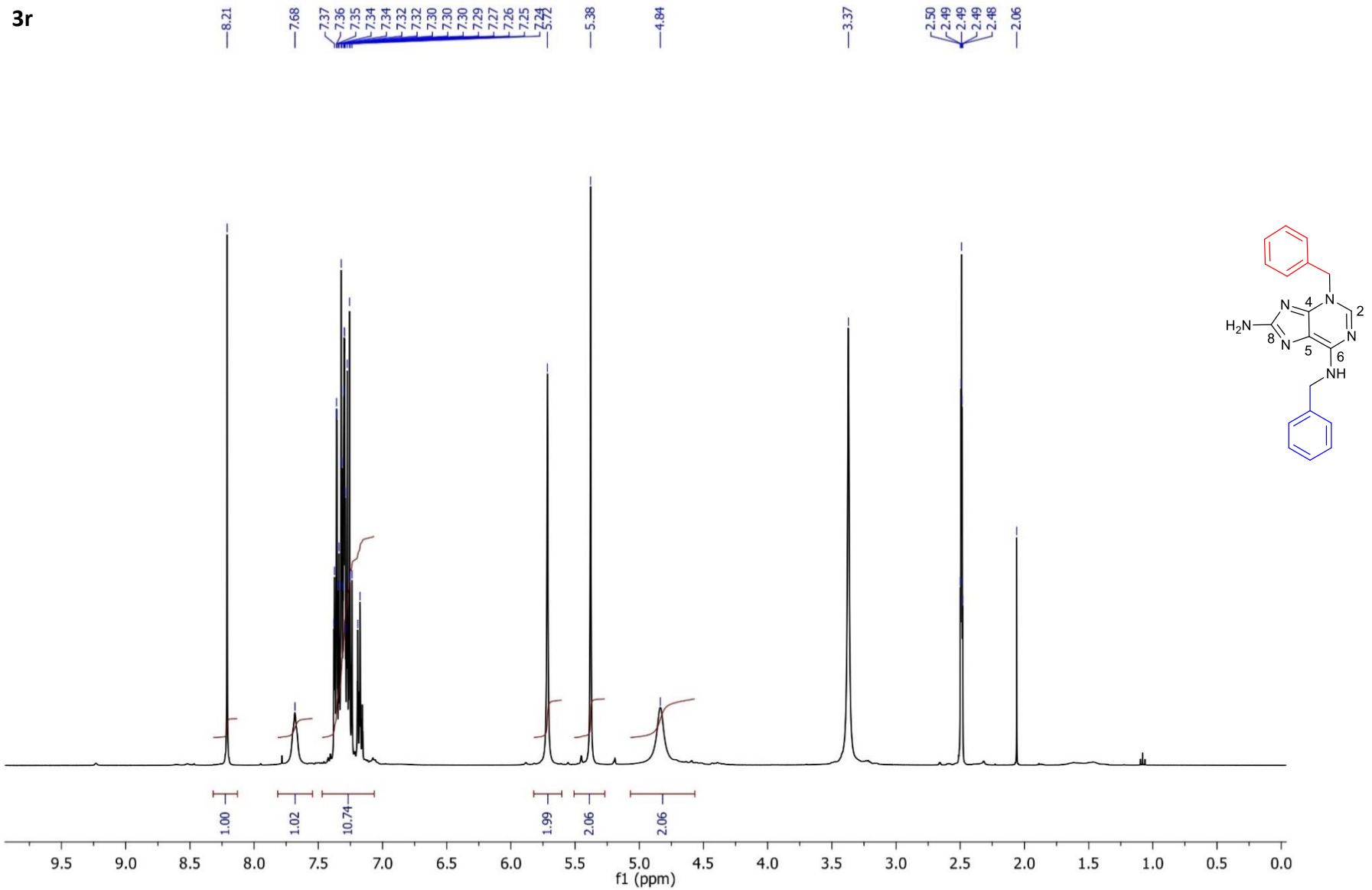



**Figure S2.52:** $^{1}$H NMR spectrum (400 MHz, DMSO-$d_6$) of $N^6$,3-dibenzyl-3*H*-purine-6,8-diamine (**3r**).

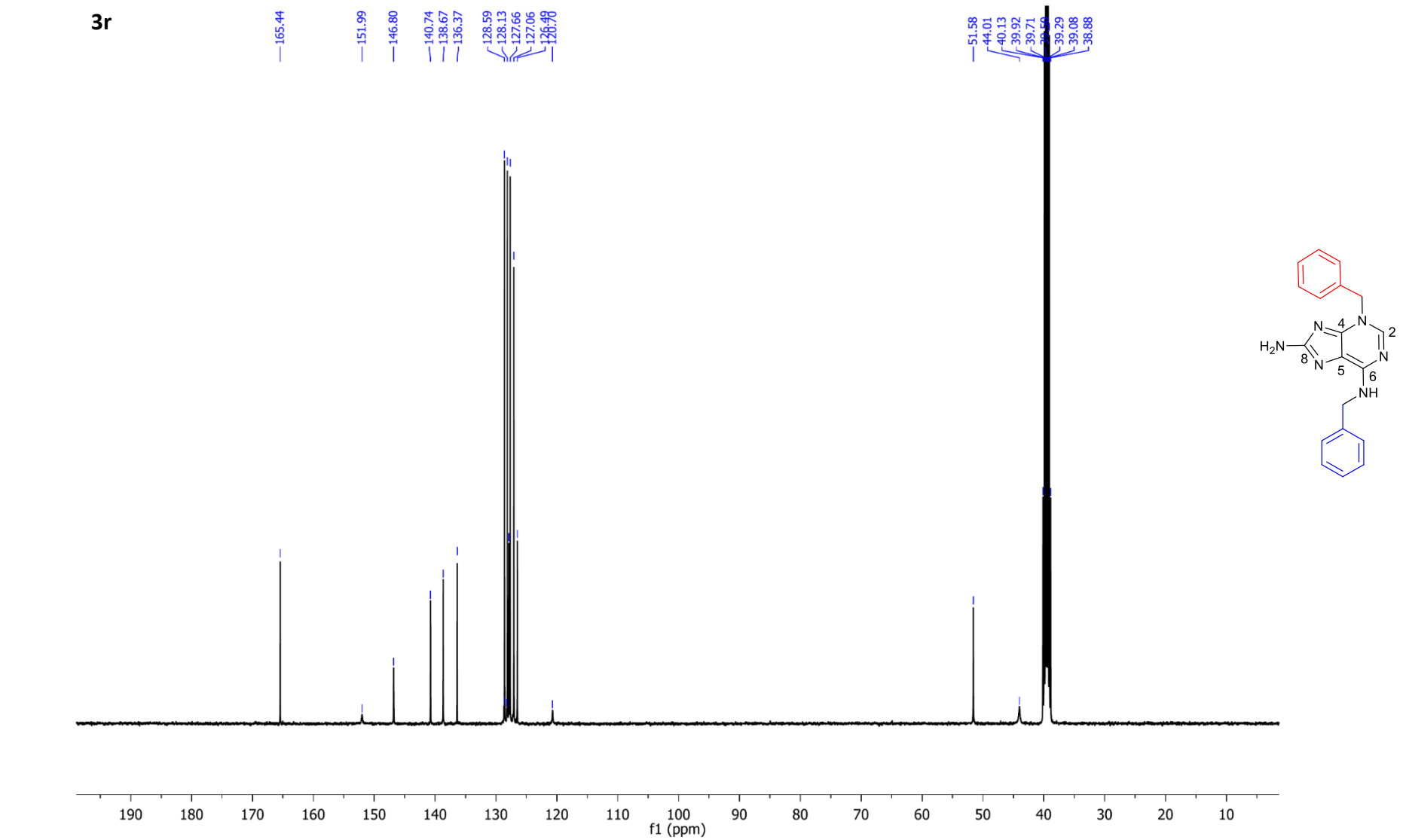



**Figure S2.53:** $^{13}$C NMR spectrum (100 MHz, DMSO-$d_6$) of $N^6$,3-dibenzyl-3*H*-purine-6,8-diamine (**3r**).

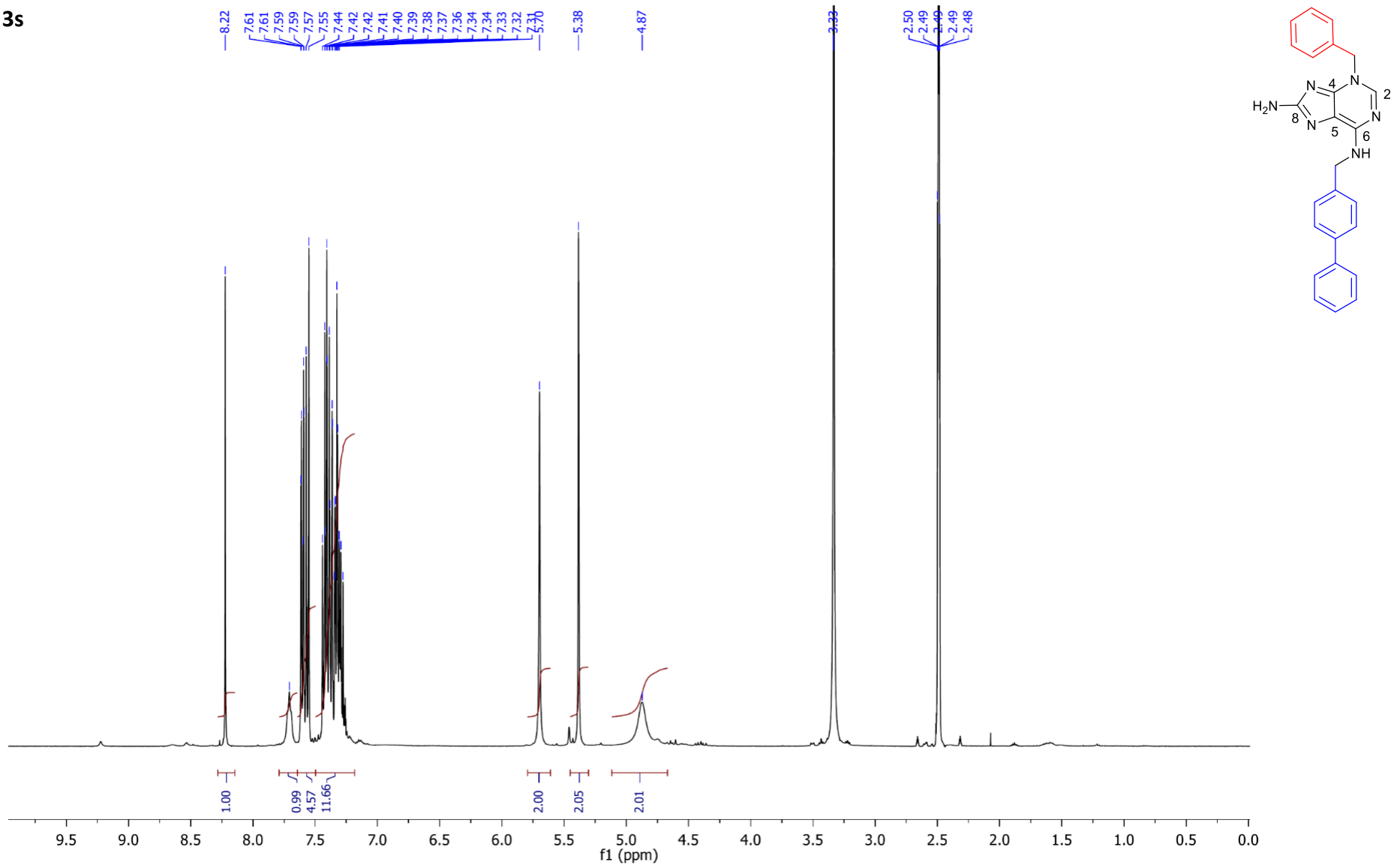



**Figure S2.54:** $^{1}$H NMR spectrum (400 MHz, DMSO-$d_6$) of $N^6$-([1,1'-biphenyl]-4-ylmethyl)-3-benzyl-3*H*-purine-6,8-diamine (**3s**)

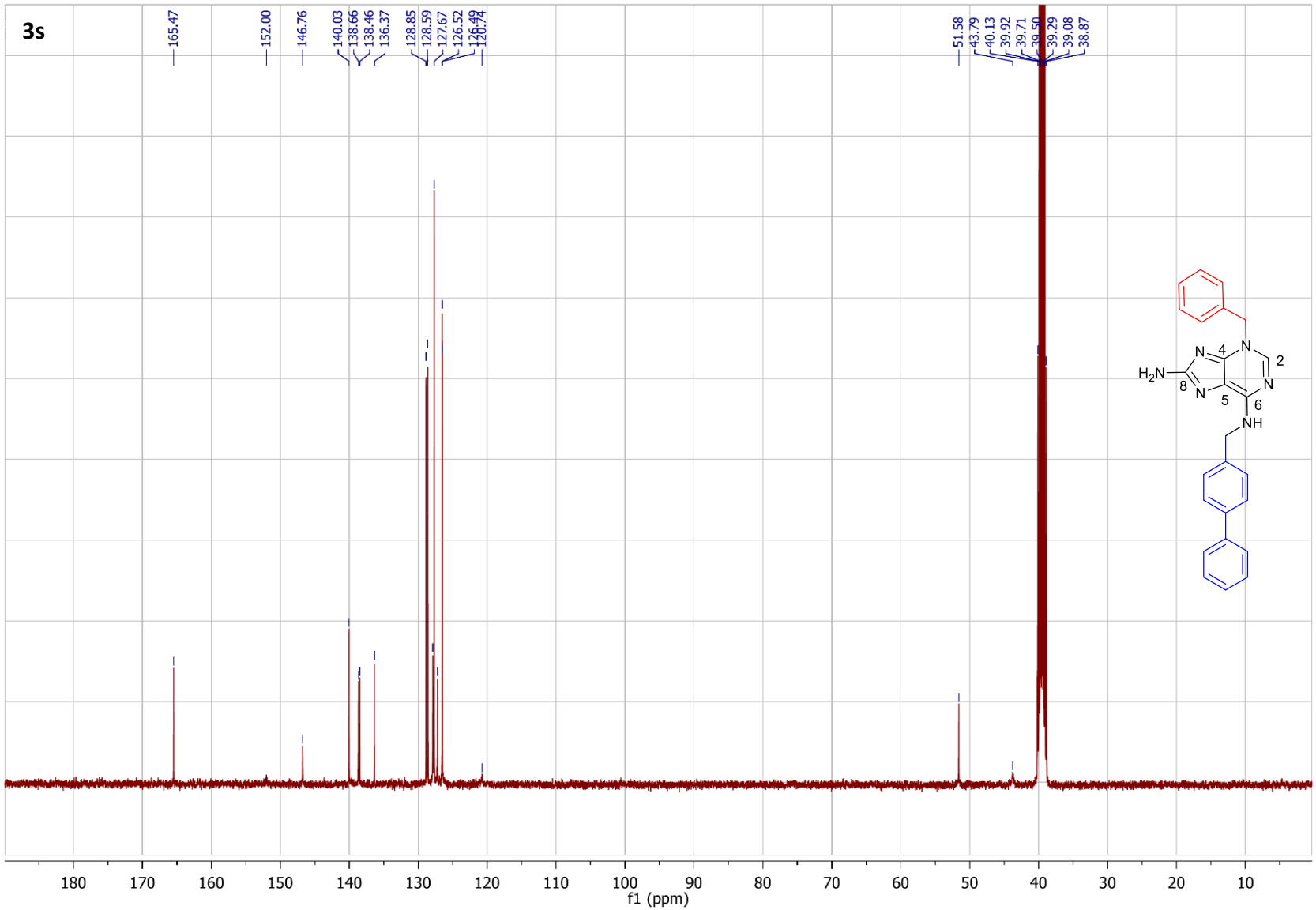



**Figure S2.55:** $^{13}$C NMR spectrum (100 MHz, DMSO-$d_6$) of $N^6$-([1,1'-biphenyl]-4-ylmethyl)-3-benzyl-3*H*-purine-6,8-diamine (**3s**).

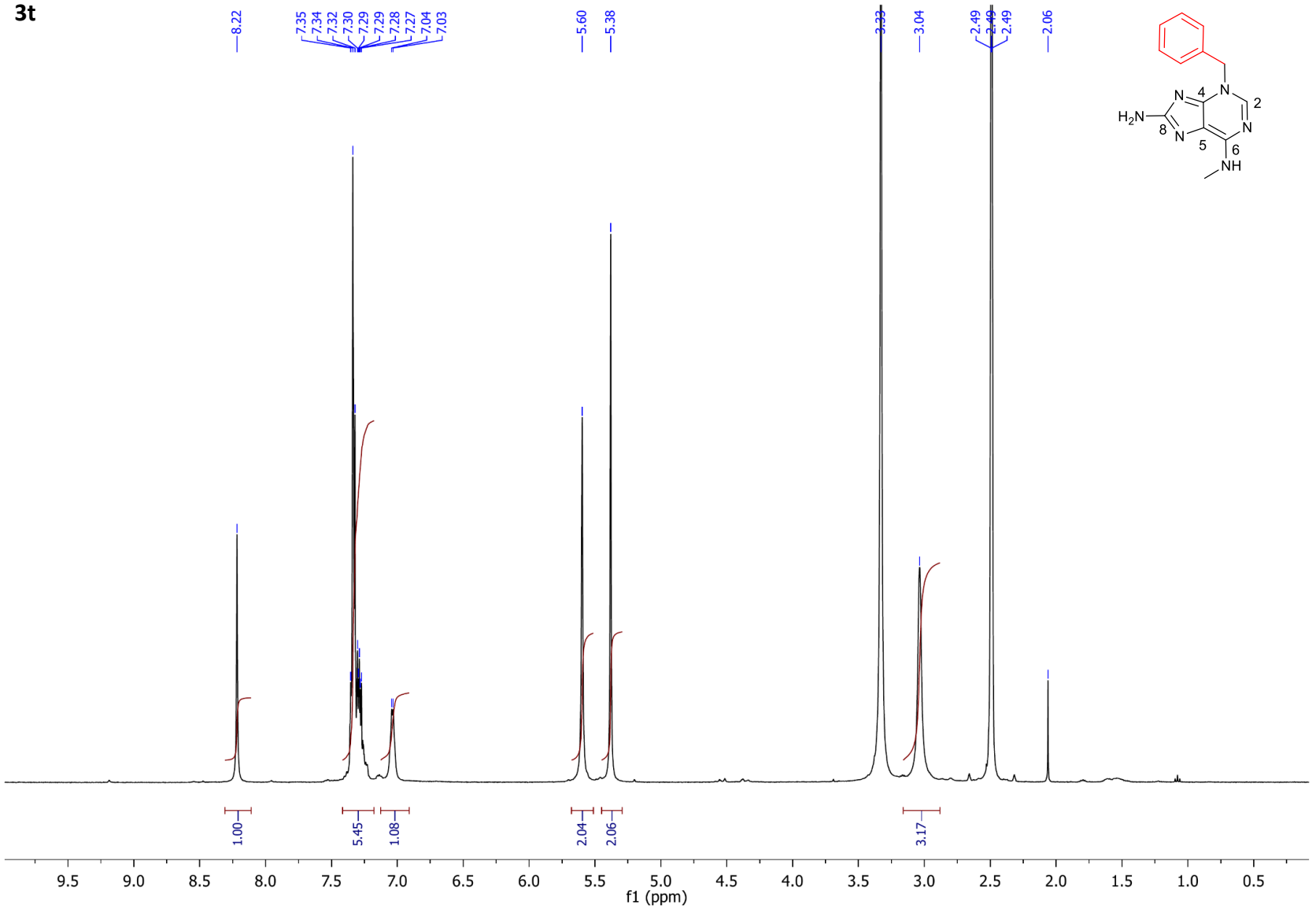



**Figure S2.56:** $^{1}$H NMR spectrum (400 MHz, DMSO-$d_6$) of 3-benzyl-$N^6$-methyl-3*H*-purine-6,8-diamine (**3t**).

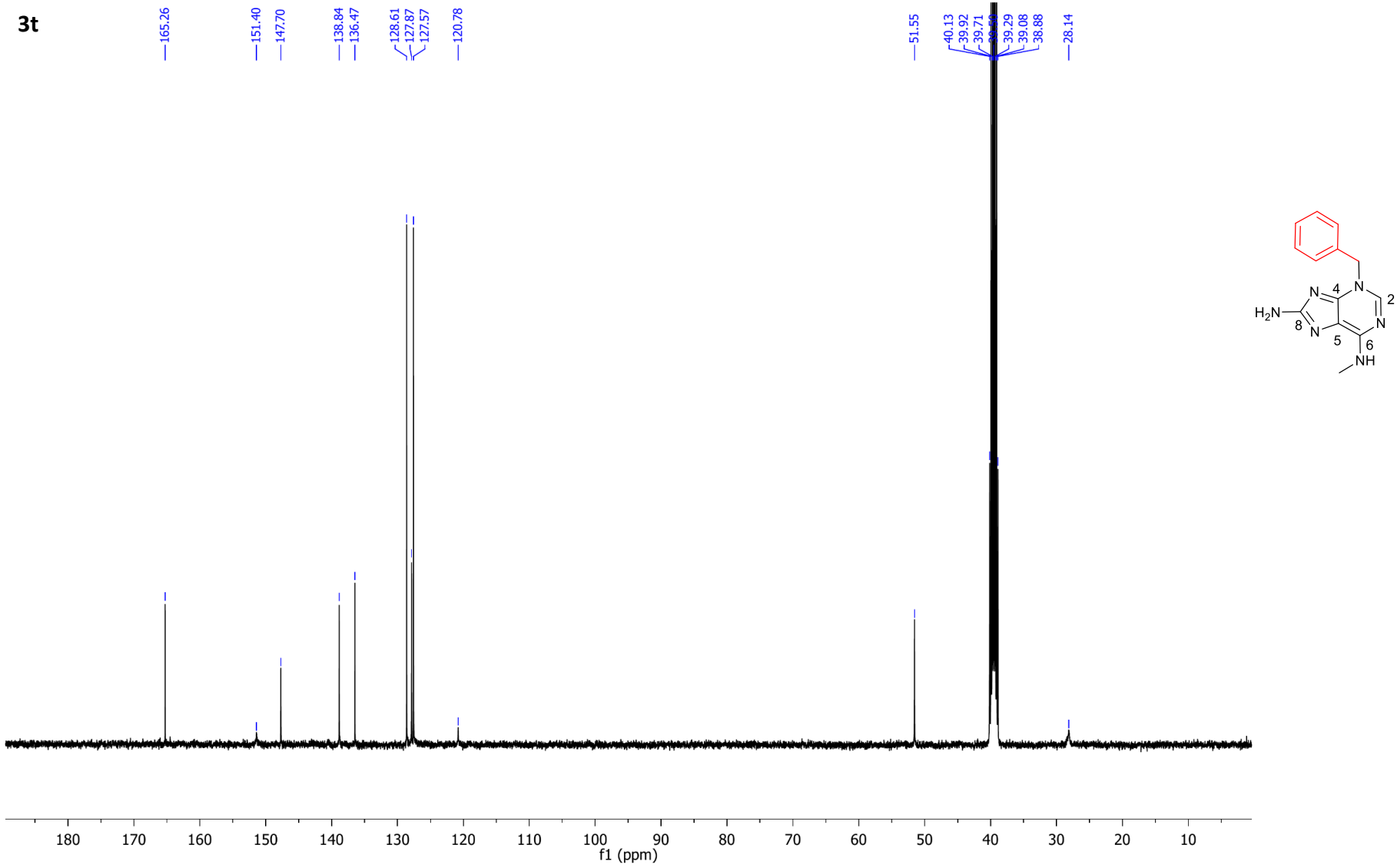



**Figure S2.57:** $^{13}$C NMR spectrum (100 MHz, DMSO-$d_6$) of 3-benzyl-$N^6$-methyl-3*H*-purine-6,8-diamine (**3t**).

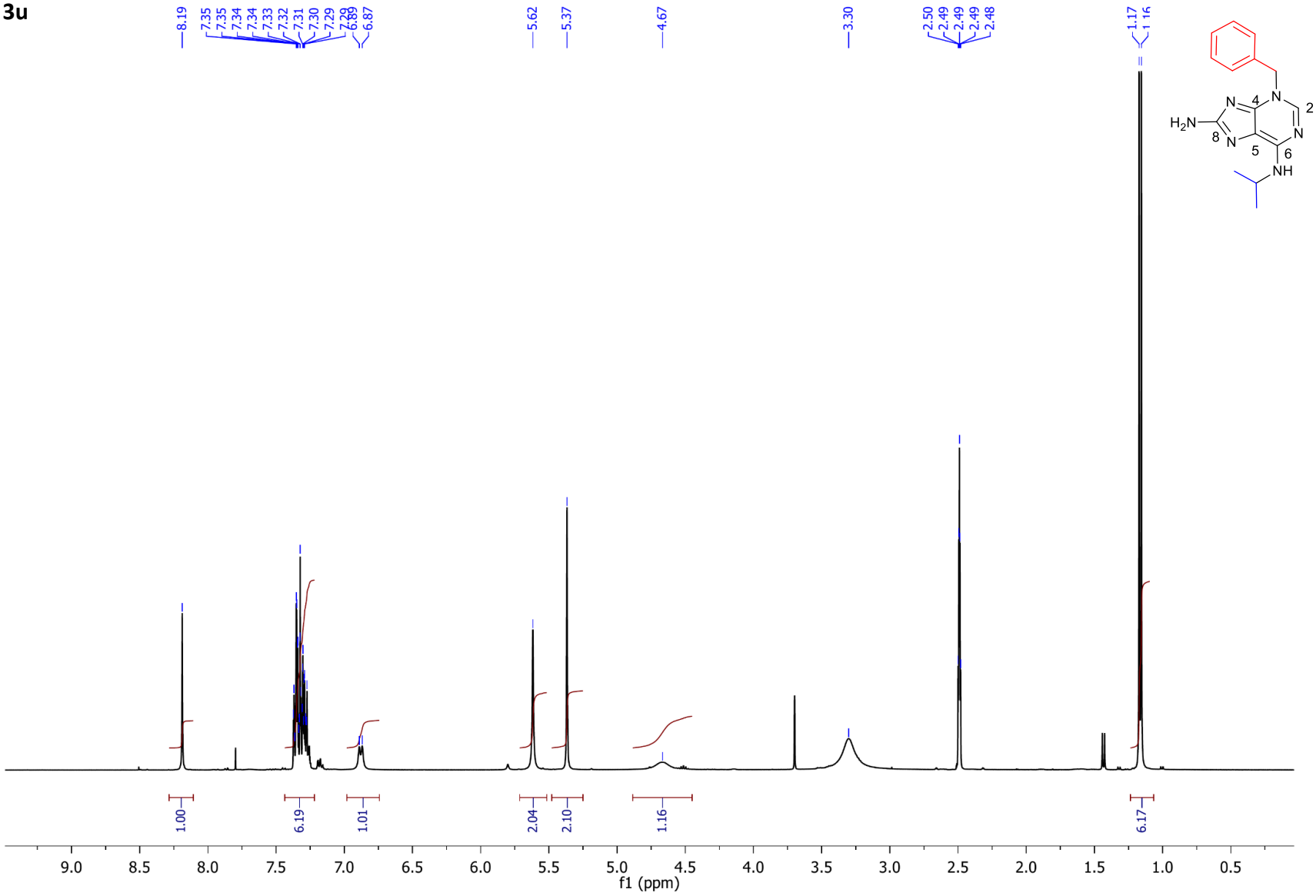



**Figure S2.58:** $^{1}$H NMR spectrum (400 MHz, DMSO-$d_6$) of 3-benzyl- $N^6$-isopropyl-3*H*-purine-6,8-diamine (**3u**).

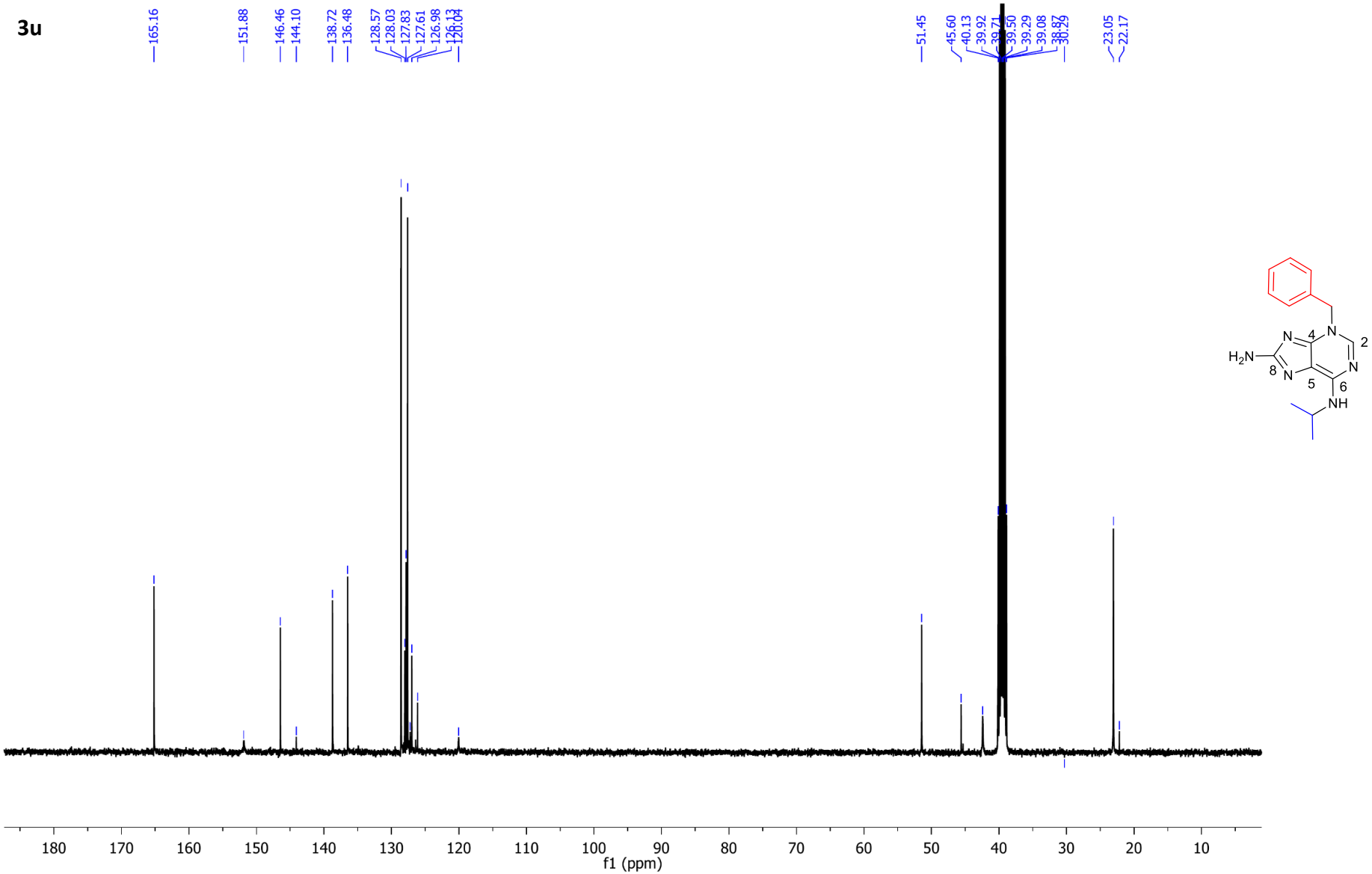



**Figure S2.59:** $^{13}$C NMR spectrum (100 MHz, DMSO-$d_6$) of 3-benzyl- $N^6$-isopropyl-3*H*-purine-6,8-diamine (**3u**).

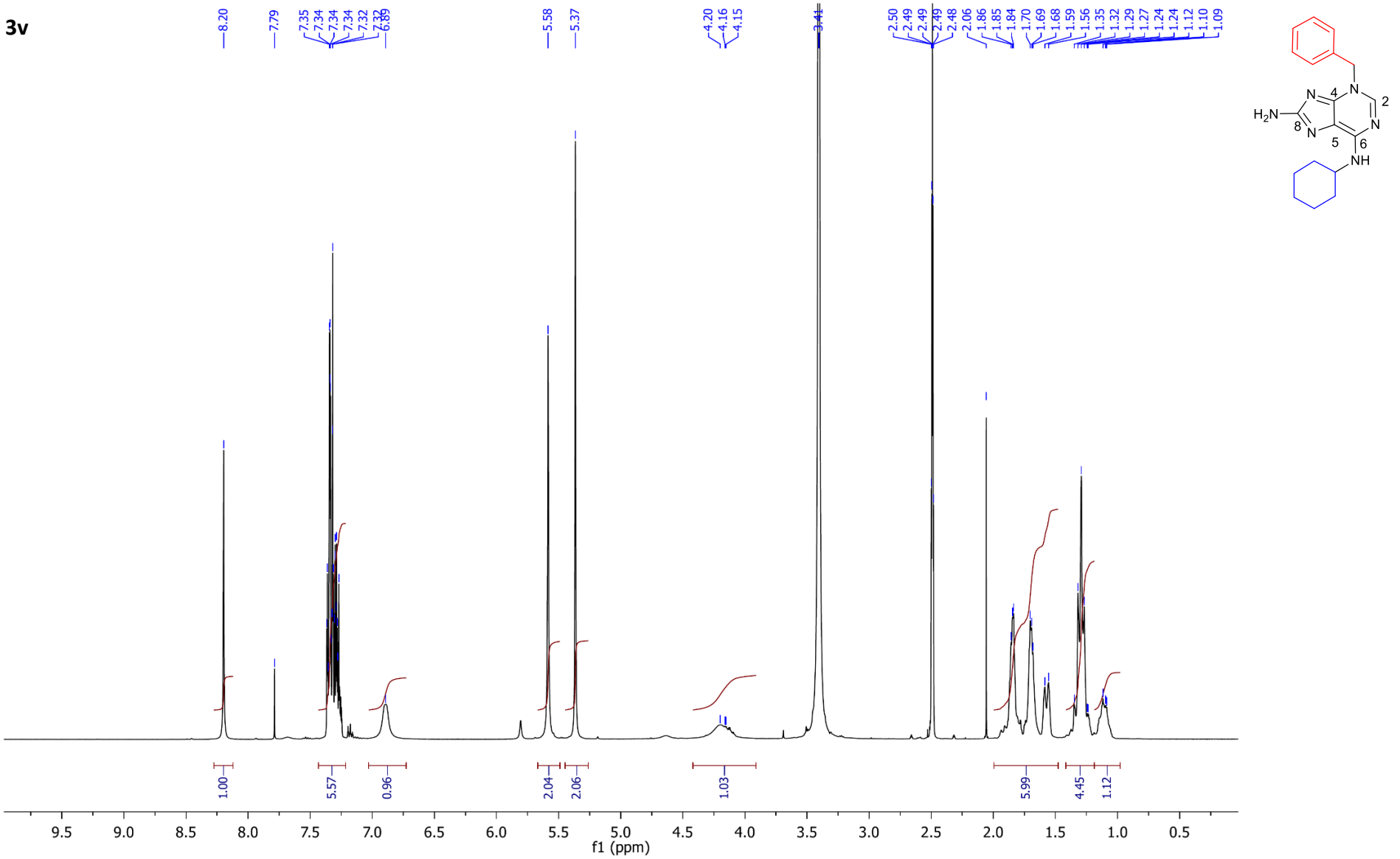



**Figure S2.60:** $^{1}$H NMR spectrum (400 MHz, DMSO-$d_6$) of $N^6$-benzyl-3-cyclohexyl-3*H*-purine-6,8-diamine (**3v**).

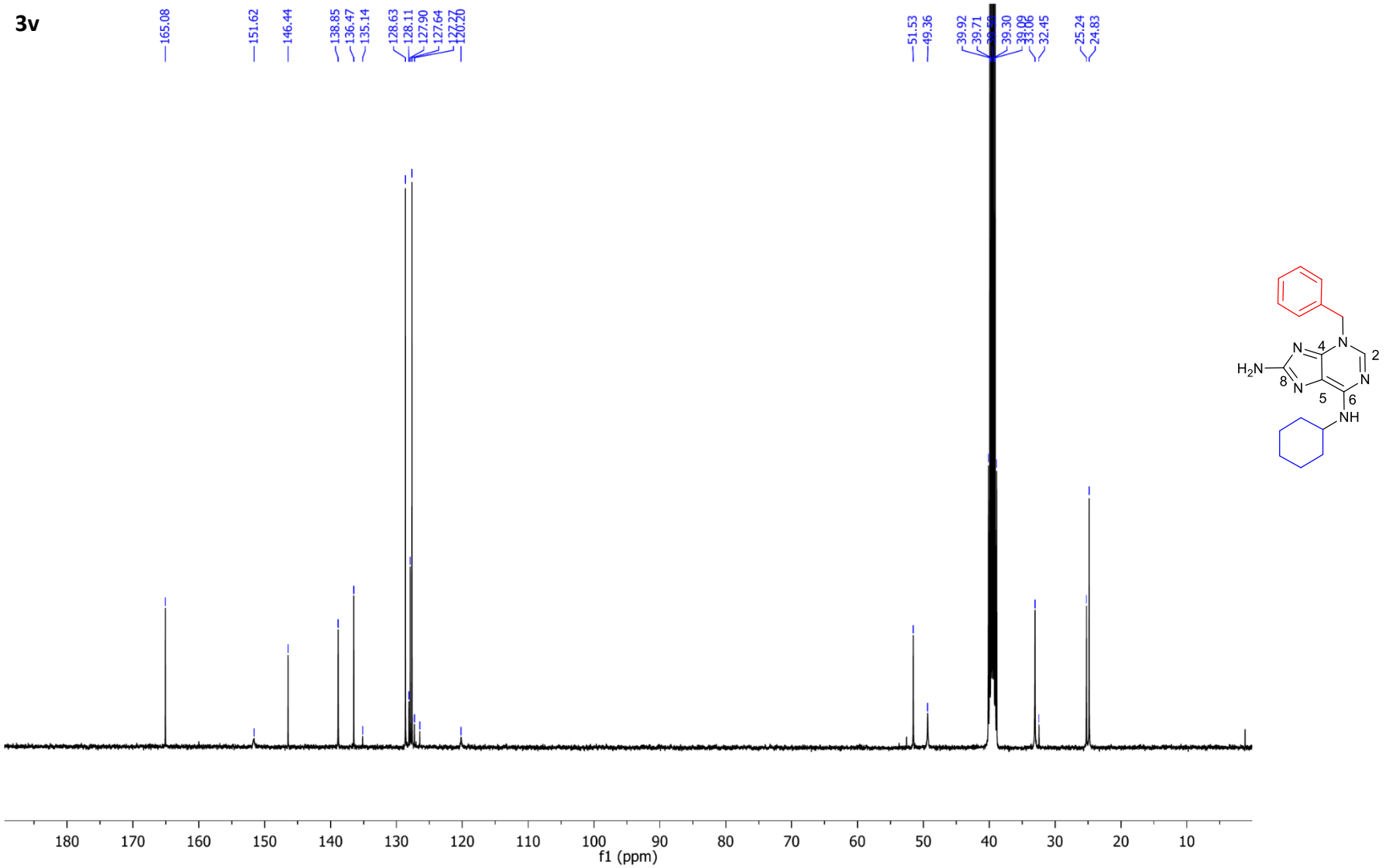
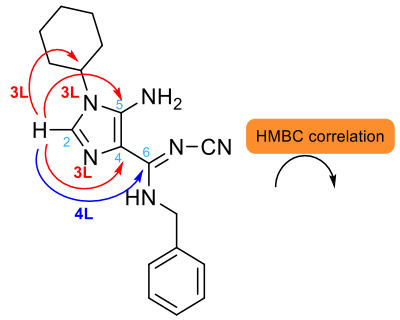
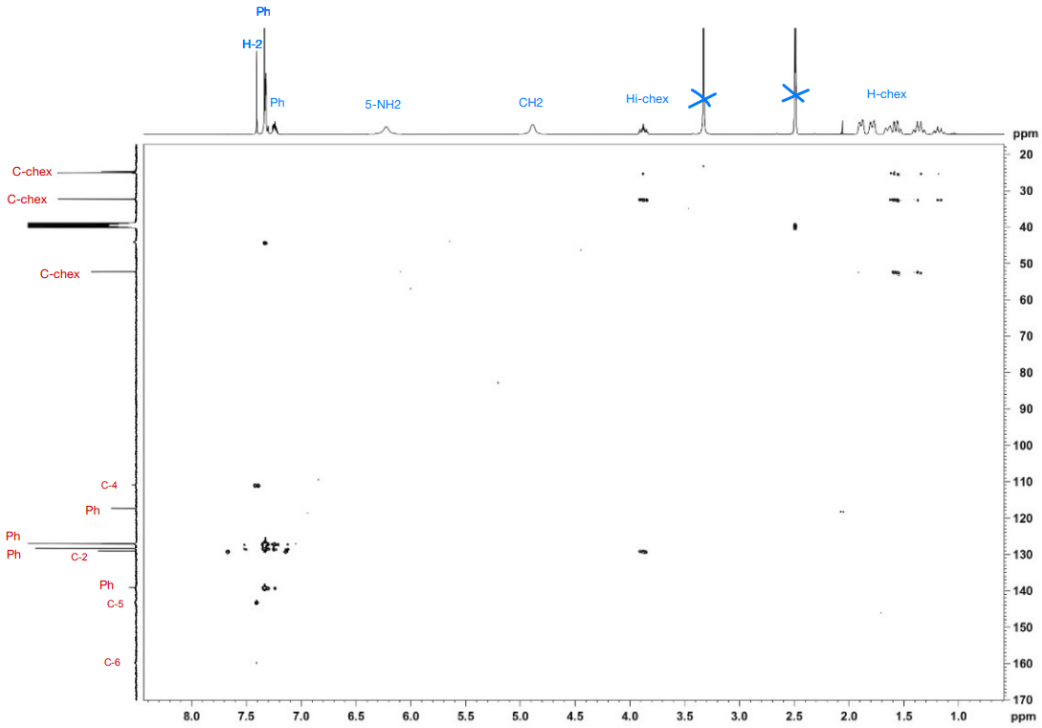



**Figure S2.61:** $^{13}$C NMR spectrum (100 MHz, DMSO-$d_6$) of $N^6$-benzyl-3-cyclohexyl-3*H*-purine-6,8-diamine (**3v**).

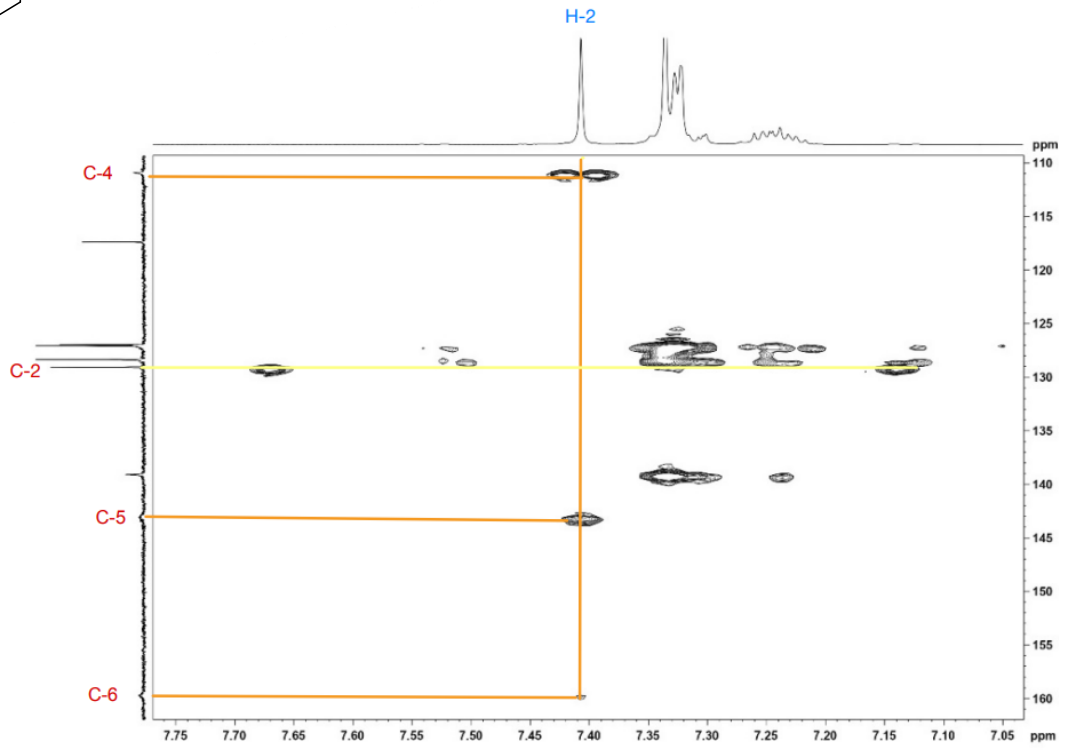
## 3 - HMBC spectra

3.1 - *5-amino-N-benzyl-N'-cyano-1-cyclohexyl-1H-imidazole-4-carboximidamide* (**2v**)



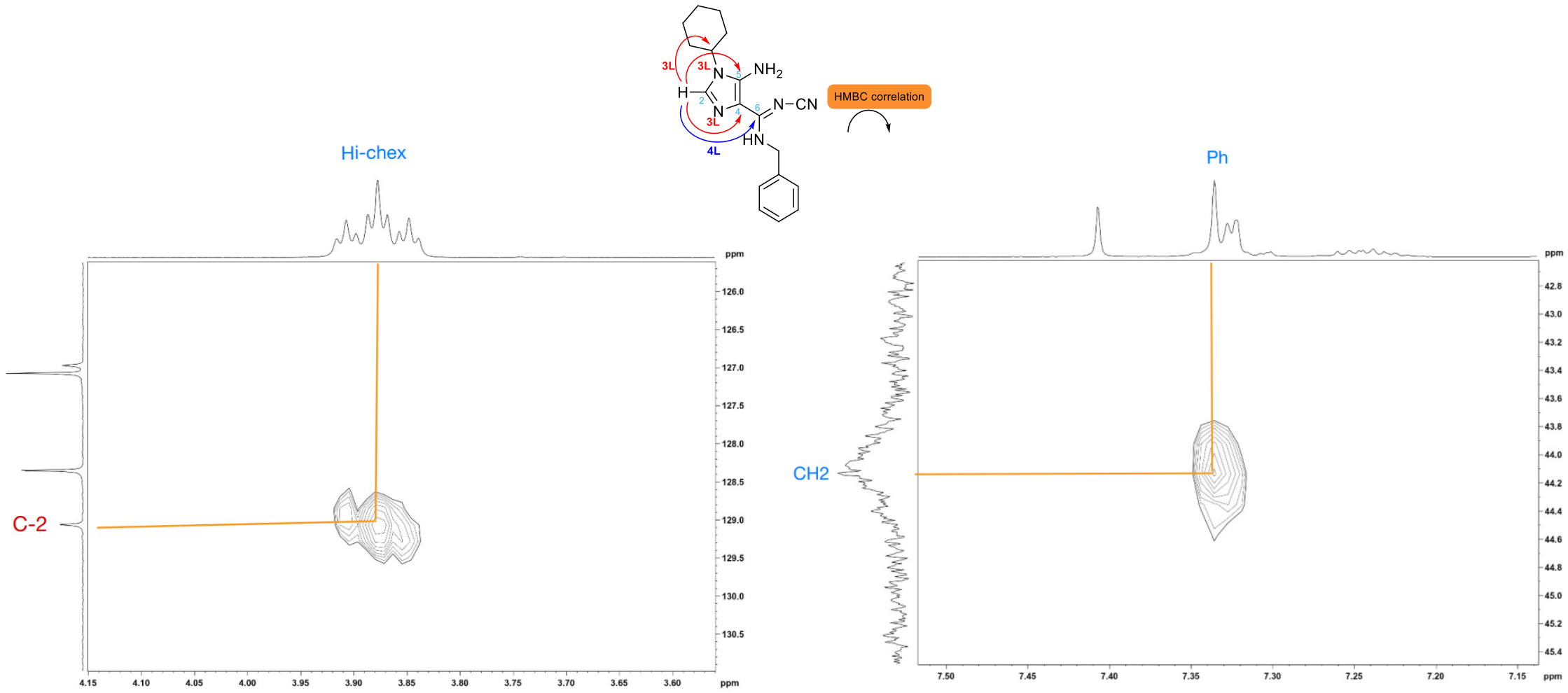

HMBC spectra of compound **2v**




HMBC spectra of compound **2v** (expansion)

## 3 - HMBC spectra

3.1 - *5-amino-N-benzyl-N'-cyano-1-cyclohexyl-1H-imidazole-4-carboximidamide* (**2v**) (continuation)

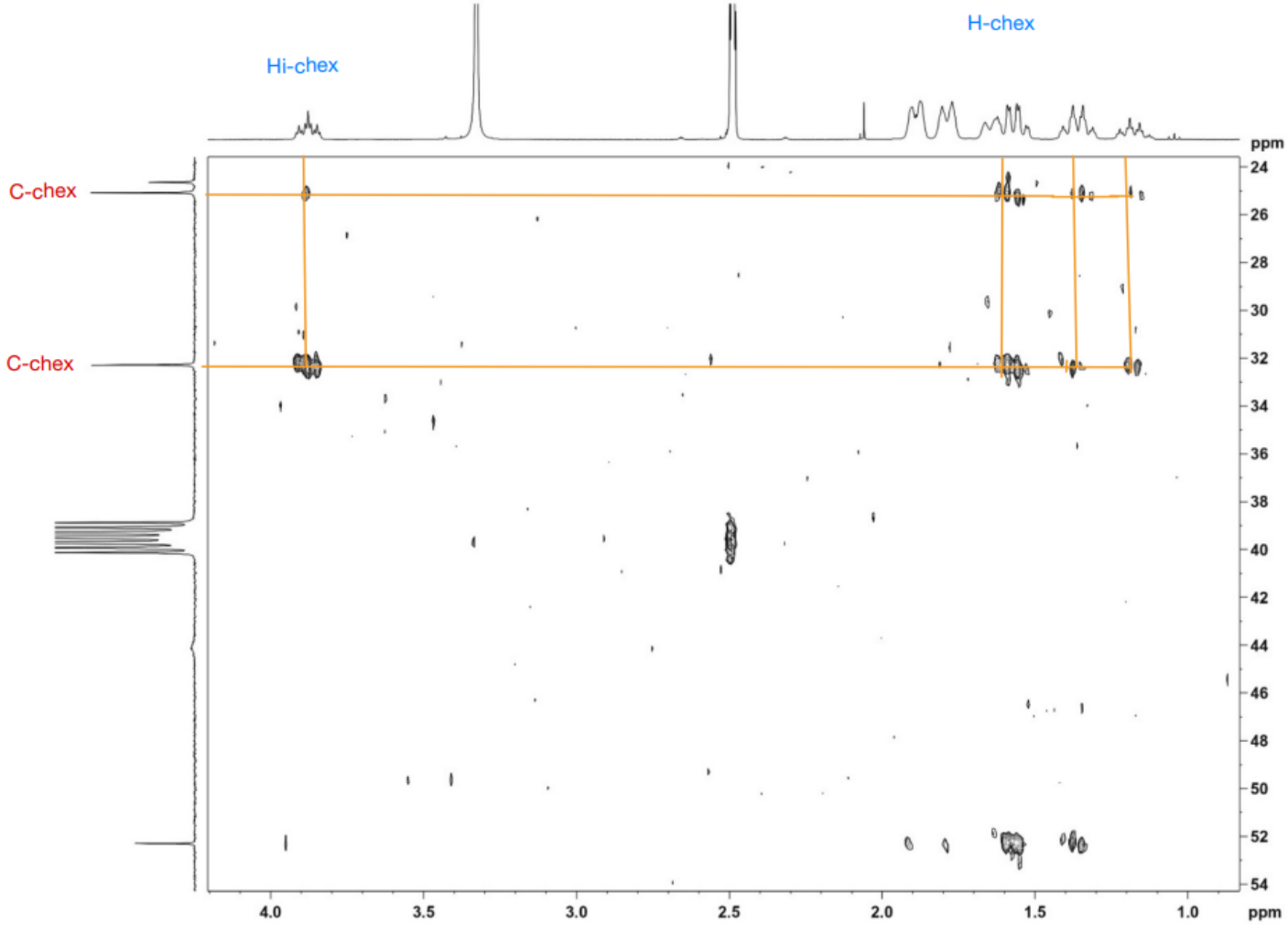


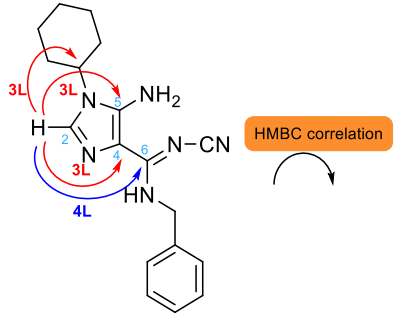

HMBC spectra of compound **2v** (expansion)

HMBC spectra of compound **2v** (expansion)

## 3 - HMBC spectra

3.1 - *5-amino-N-benzyl-N'-cyano-1-cyclohexyl-1H-imidazole-4-carboximidamide* (**2v**) (continuation)

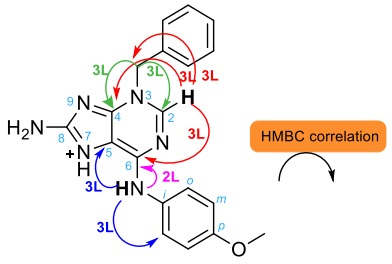


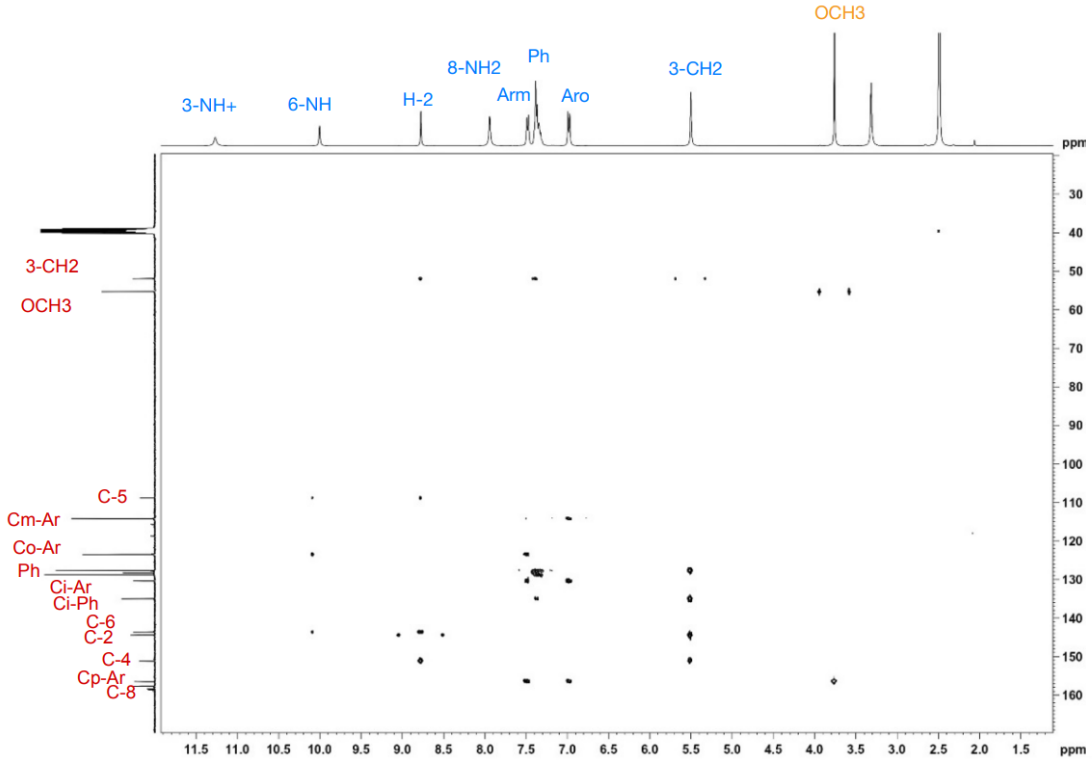




HMBC spectra of compound **2v** (expansion)

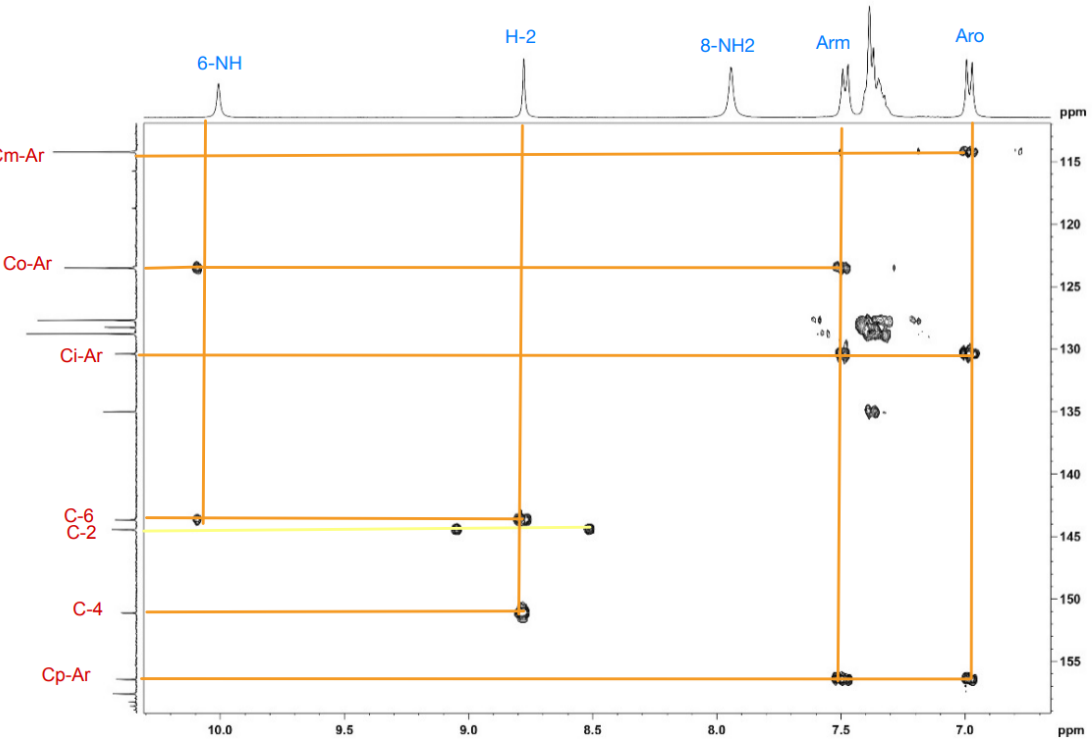
## 3 - HMBC spectra

3.2 - *3-benzyl-N$^6$-(4-methoxyphenyl)-3H-purine-6,8-diamine* (**3b**)



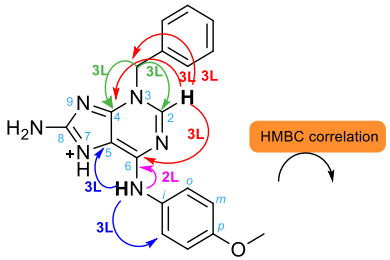

HMBC spectra of compound **3b**

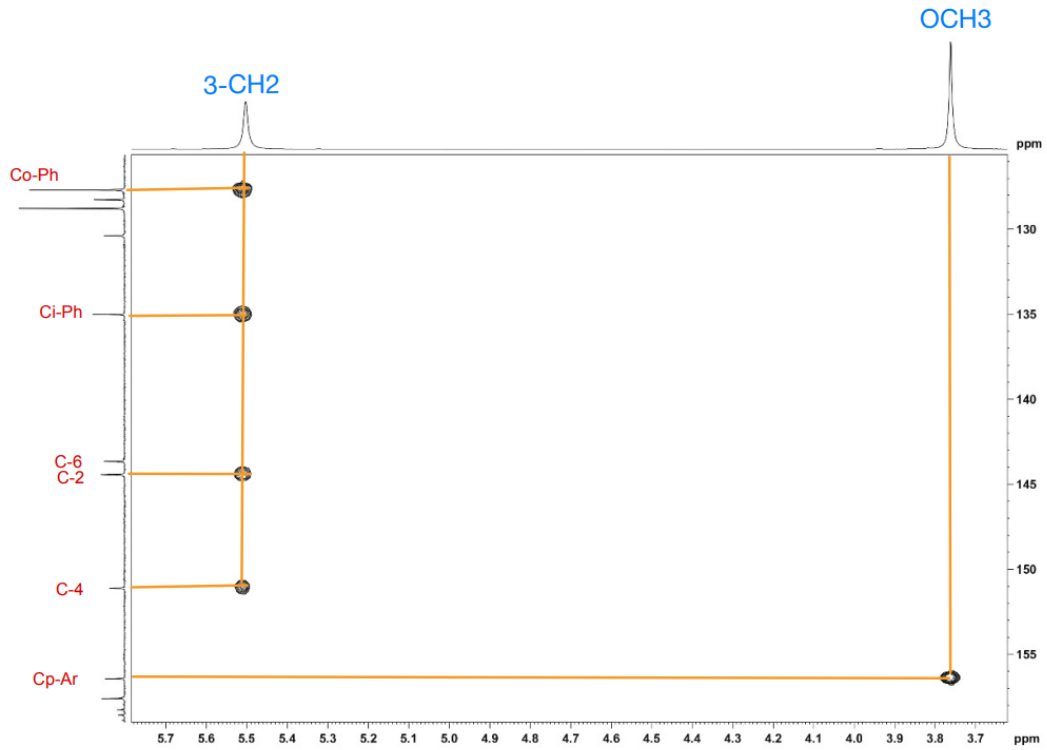



HMBC spectra of compound **3b** (expansion)

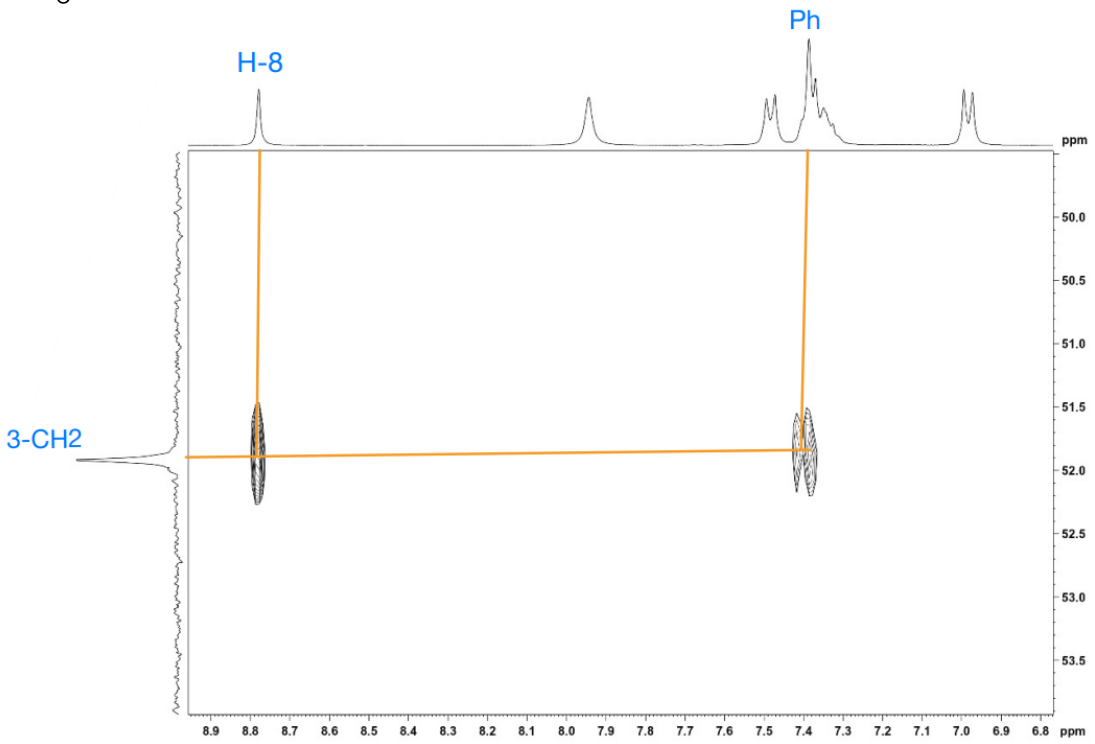
## 3 - HMBC spectra

3.2 - *3-benzyl-N$^6$-(4-methoxyphenyl)-3H-purine-6,8-diamine* (**3b**) (continuation)



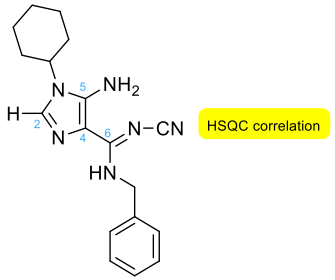

HMBC spectra of compound **3b** (expansion)

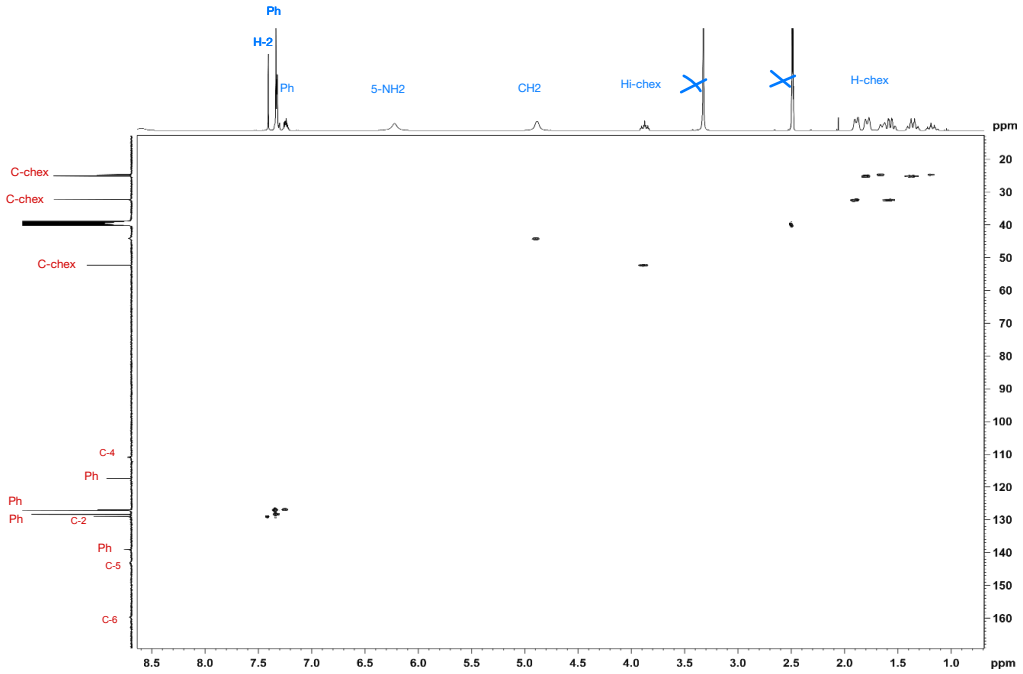



HMBC spectra of compound **3b** (expansion)

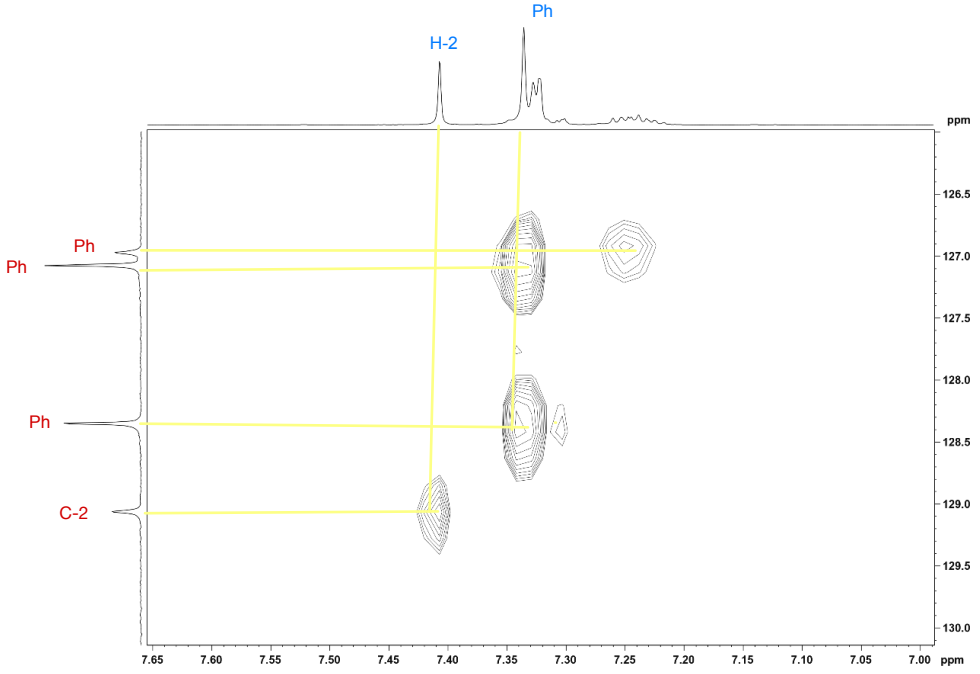
## 4 - HSQC spectra

4.1 - *5-amino-N-benzyl-N'-cyano-1-cyclohexyl-1H-imidazole-4-carboximidamide* (**2v**)



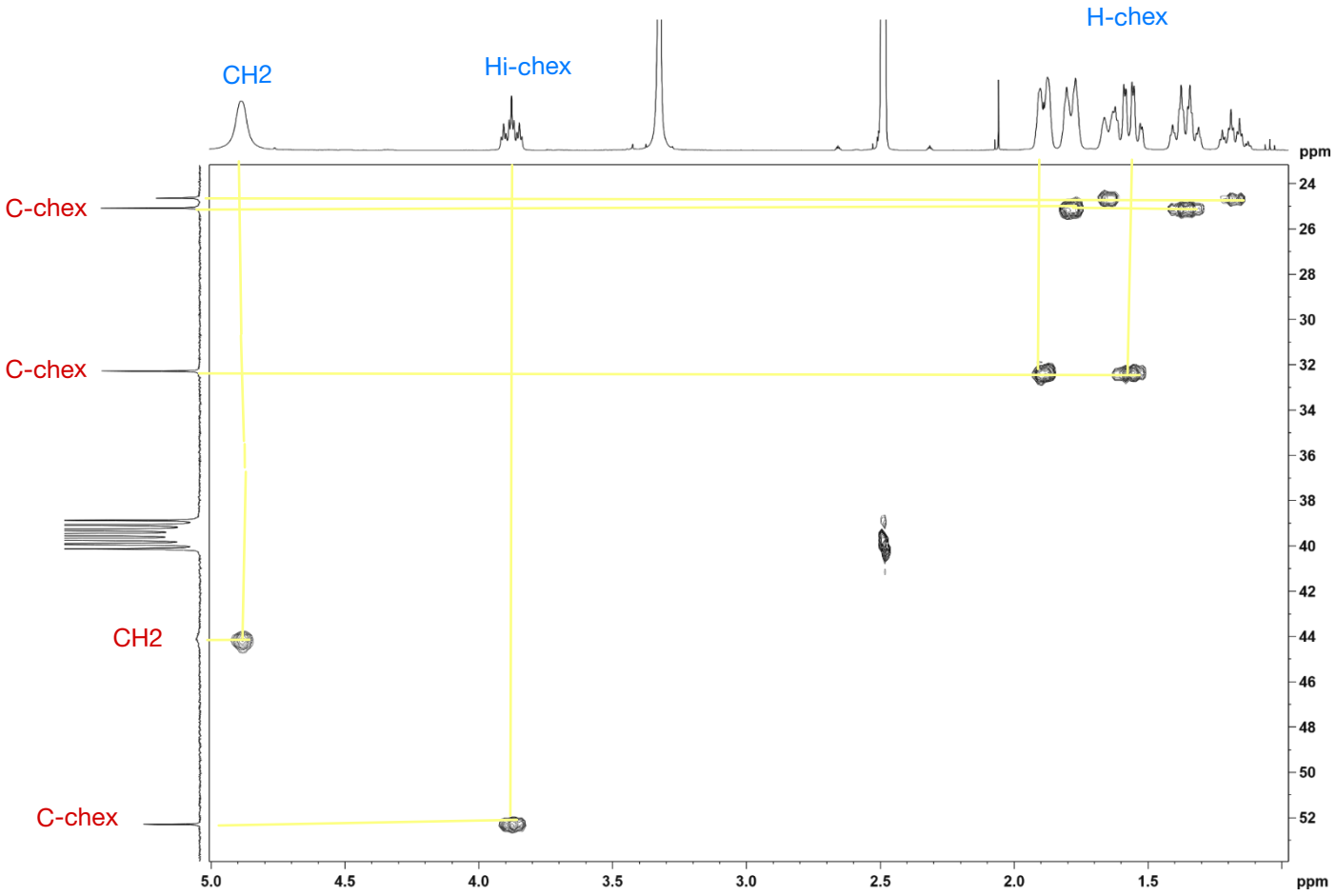

HSQC spectra of compound **2v**

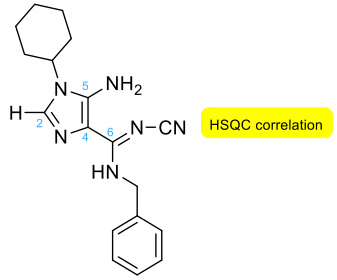



HSQC spectra of compound **2v** (expansion)

## 4 - HSQC spectra

4.1 - *5-amino-N-benzyl-N'-cyano-1-cyclohexyl-1H-imidazole-4-carboximidamide* (**2v**) (continuation)

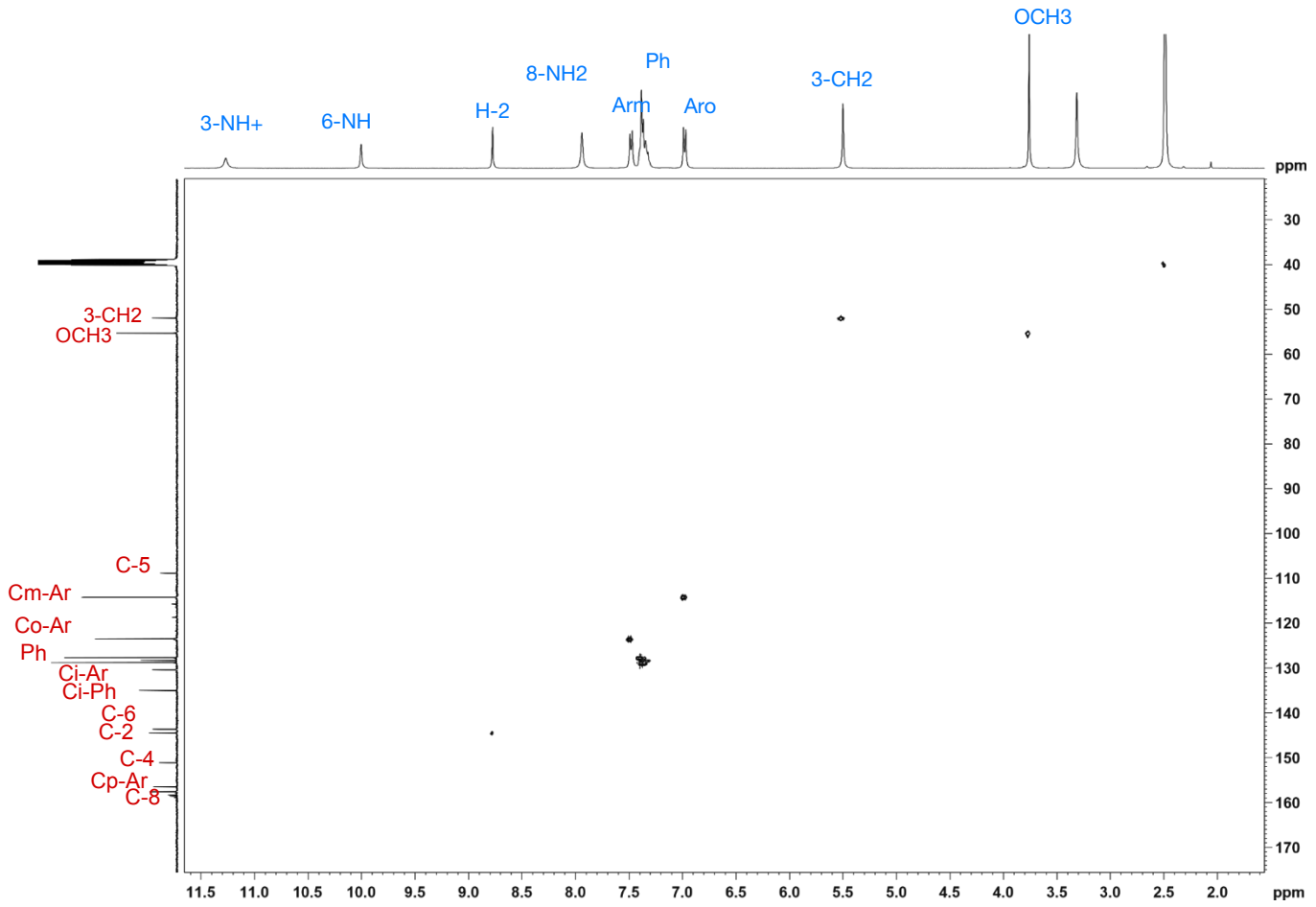


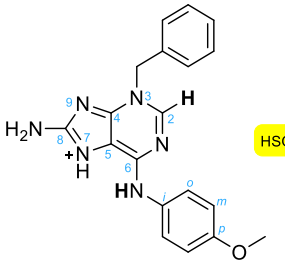




HSQC spectra compound **2v** (expansion)

## 4 - HSQC spectra

4.2 - *3-benzyl-N$^6$-(4-methoxyphenyl)-3H-purine-6,8-diamine* (**3b**)



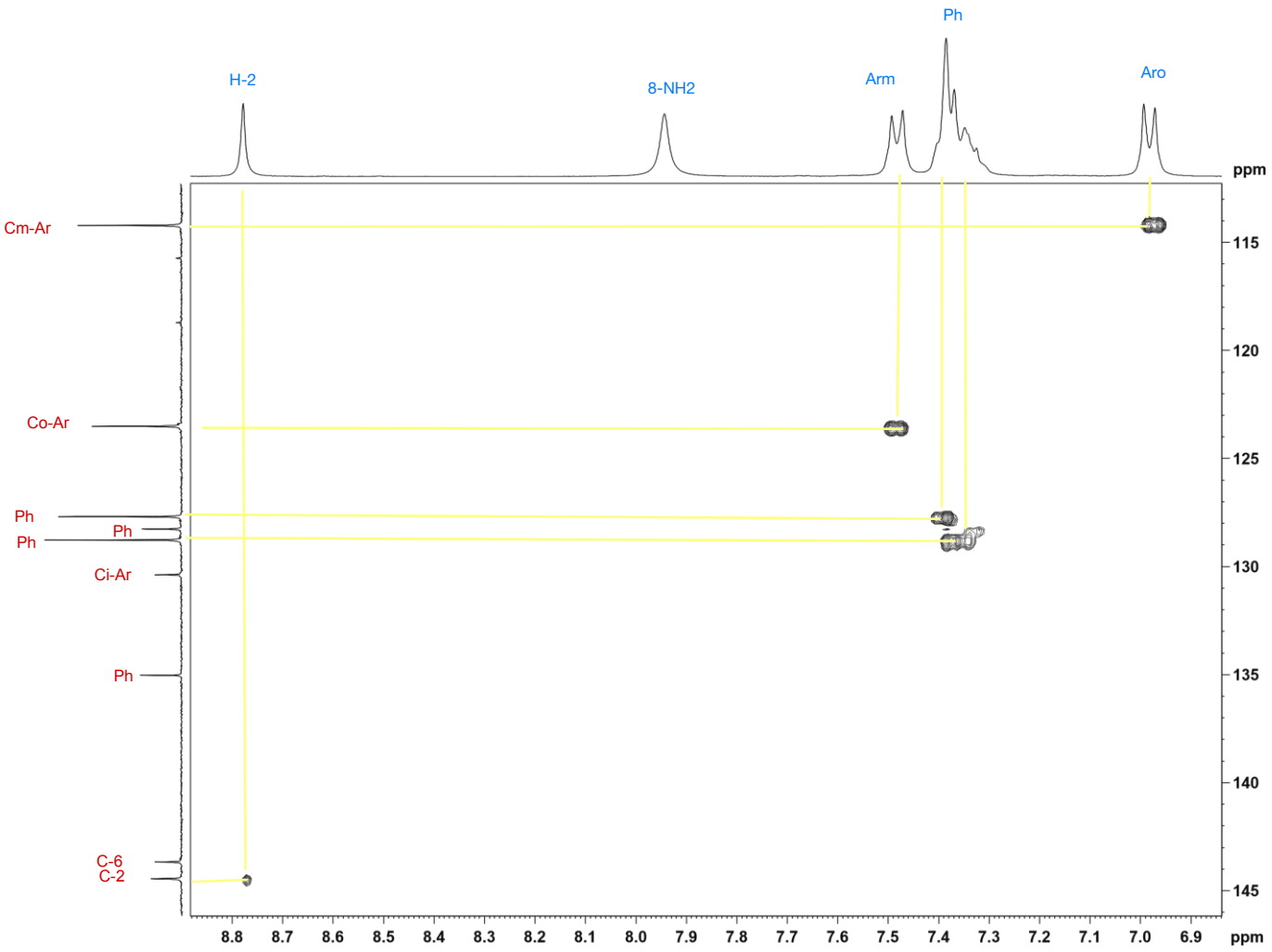



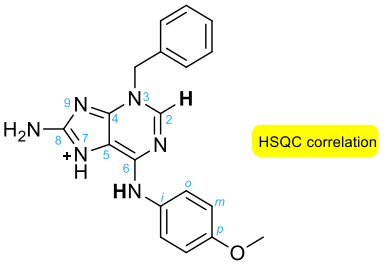
HSQC correlation

HSQC spectra of compound **3b**

## 4 - HSQC spectra

4.2 - *3-benzyl-N$^6$-(4-methoxyphenyl)-3H-purine-6,8-diamine* (**3b**) (continuation)




HSQC spectra of compound **3b** (expansion)

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