SUPPLEMENTARY MATERIAL

Synthesis of ¹⁵N labelled 3,5-dimethylpyridine

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Supplementary Table 1: Comparison of ¹⁵N chemical shifts of pyridine derivatives measured in CDCl₃ at 298 K and referenced to external CH₃NO₂.

pyridine	2-picoline	3-picoline	4-picoline	2,3-lutidine	2,4-lutidine	2,6-lutidine	3,5-lutidine	2,4,6-collidine	References
							-69.7 ppm		This work
							-69.8 ppm ^a		(1)
-70.1 ppm ^b	-71.6 ppm ^b	-75.8 ppm ^b	-84.1 ppm ^b			-72.2 ppm ^b			(2)
				-71.4 ppm ^c	-80.0 ppm ^c	-72.0 ppm ^c	-71.0 ppm ^c	-80.1 ppm ^c	(3)
-67.7 ppm ^d	-68.8 ppm ^d	-68.4 ppm ^d	-76.7 ppm ^d						(4)

^{a 15}N measurements at natural abundance in different solvents, among them CDCl₃; data were measured at 302 K and referenced to external CH₃NO₂

^b Dodalik et al. reported ¹⁵N data in CDCl₃ as well as DMSO-d₆ at 300K, at natural abundance, referenced to external liquid NH₃; for this table we calculated the chemical

shifts referenced to external CH₃NO₂ by subtracting 381.7 ppm according to Marek and Lycka 2002 (5) and Pazderski et al. 2009 (6).

^c data measured with CDCl₃ at 303 K, at natural abundance, referenced to external CH₃NO₂

^d data measured at with CDCl₃ at 298 K, at natural abundance, referenced to external CH₃NO₂

References:

- 1. Holzer W & von Philipsborn W (1989) Inter-Molecular and Intra-Molecular Hydrogen-Bonding Effects on Geminal N-15, H-1 Spin Coupling and N-15 Chemical-Shifts in Pyridine-Derivatives. *Magn Reson Chem* 27(6):511-514.
- 2. Dokalik A, Kalchhauser H, Mikenda W, & Schweng G (1999) NMR spectra of nitrogen-containing compounds. Correlations between experimental and GIAO calculated data. *Magn Reson Chem* 37(12):895-902.
- 3. Pazderski L, Pawlak T, Sitkowski J, Kozerski L, & Szlyk E (2010) Structural correlations for H-1, C-13 and N-15 NMR coordination shifts in Au(III), Pd(II) and Pt(II) chloride complexes with lutidines and collidine. *Magn Reson Chem* 48(6):417-426.
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- 5. Marek R & Lycka A (2002) N-15 NMR spectroscopy in structural analysis. *Curr Org Chem* 6(1):35-66.
- 6. Pazderski L, *et al.* (2009) Experimental and quantum-chemical studies of H-1, C-13 and N-15 NMR coordination shifts in Au(III), Pd(II) and Pt(II) chloride complexes with picolines. *Magn Reson Chem* 47(3):228-238.







¹⁵N-labeled 3,5-Dimethylpyridine in CDCl₃





Current NAME	Data MAH	Pai RIO	rameters 13C orig	
EXPNO			1	
PROCNO			\perp	
F2 - Pro	cess:	ing	paramete	ers
SI			16384	
SF		67	.9249666	MHz
WDW			no	
SSB	0			
LB			2.00	Ηz
GB	0			
PC			4.00	

¹³C (ppm)

 $^{\rm 15}\rm{N}\mathchar`labeled$ 3,5-Dimethylpyridine in \rm{CDCl}_3

¹⁵N



Current NAME	ma lut 15N 2018
EXPNO	1
PROCNO	1
F2 - Pr	ocessing parameters
SI	32768
SF	50.6976064 MHz
WDW	no
SSB	0
LB	0 Hz
GB	0
PC	1.00

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-60	-80	-100	-120	-140	-160	-180	-200	-220	-240	 Referenced to CH ₃ NO ₂
320	300	280	260	240	220	200	180	160	140	— Referenced to NH ₃ (liquid)
				15	N (ppm)					