Synthesis of picolinohydrazides and their evaluation as ligands in the zinc-catalyzed hydrosilylation of ketones

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Supplementary data

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1) General methods

Reagents were obtained from commercial suppliers and used without further purification. TLC analyses were performed using precoated Merck TLC Silica Gel 60 F₂₅₄ plates. Purifications by column chromatography on silica gel were performed using Merck Silica Gel 60 (70–230 mesh). Petroleum ether (PE) used for purifications was the low boiling point fraction (40–60 °C). The ¹H and ¹³C NMR spectra were recorded on a Bruker Avance 300 instrument using TMS and CDCl₃ as internal standard, respectively. Chemical shifts (δ) are reported in parts per million (ppm). The following abbreviations are used for multiplicities: s, singlet; d, doublet; t, triplet; dd, doublet of doublets; td, triplet of doublets; dt, doublet of triplets; ddd, doublet of doublets of doublets; m, multiplet. Coupling constants (*J*) are reported in Hertz (Hz). HRMS analyses were performed by the "Centre Regional de Mesures Physiques de l'Ouest" (CRMPO, Rennes), and were obtained using a Bruker MaXis 4G or a Bruker Micro-TOF Q II for ESI. IR spectra were recorded on a PerkinElmer FT-IR Spectrometer UATR Spectrum Two. Melting points were obtained on a hot bench.

2) Synthesis and characterization of picolinohydrazide ligands L1-L9

General procedure for the synthesis of picolinohydrazides L1 and L2 with TBTU: To a solution of 1 (1 equiv.) in THF and DMF were added the hydrazine hydrochloride (1 equiv.), K_2CO_3 (4 equiv.), and TBTU (1 equiv.). The reaction was stirred overnight at room temperature. After addition of water, the mixture was extracted with EtOAc (x3), the combined organic phases were washed with brine (x3), dried over anhydrous MgSO₄, and evaporated to dryness. The crude product was then purified by flash chromatography on silica gel.

Methyl N-benzyl-N-(picolinamido)glycinate (L1): Following the general procedure with 1 (356 mg, 2.9 mmol), 2.HCl (666 mg, 2.9 mmol), K₂CO₃ (1.6 g, 11.6 mmol), TBTU (931 mg, 2.9

mmol), THF (15 mL), and DMF (15 mL). Elution with PE / EtOAc (1 / 1). White solid (751 mg, 87% yield); m.p. 72 °C. ¹H NMR (300 MHz, CDCl₃, ppm): δ = 3.74 (s, 3 H), 3.84 (s, 2 H), 4.30 (s, 2 H), 7.20–7.32 (m, 3 H), 7.34–7.39 (m, 1 H), 7.43–7.45 (m, 2 H), 7.74–7.80 (m, 1 H), 8.11–8.14 (m, 1 H), 8.49–8.51 (m, 1 H), 9.79 (br. s, 1 H). ¹³C{¹H} NMR (75 MHz, CDCl₃, ppm): δ = 51.7, 55.1, 60.2, 122.3, 126.2, 127.6, 128.4, 129.1, 136.5, 137.1, 148.2, 149.6, 162.3, 171.1. IR (solid, cm⁻¹): 3315, 3051, 2952, 2920, 2851, 1731, 1683, 1496, 1207, 997, 972, 743. IR (CHCl₃, cm⁻¹): 3324, 3065, 3010, 2954, 2854, 1736, 1685, 1509. HRMS (ESI): *m/z* calcd. for C₁₆H₁₇N₃O₃Na [M + Na]⁺ 322.1167; found 322.1171.

N',N'-Dibenzylpicolinohydrazide (L2): Following the general procedure with 1 (300 mg, 2.43 mmol), **3.HCl** (606 mg, 2.43 mmol), K₂CO₃ (1.34 g, 9.72 mmol), TBTU (782 mg, 2.43 mmol), THF (15 mL), and DMF (15 mL). Elution with PE / EtOAc (2 / 1). White solid (654 mg, 85% yield); m.p. 140 °C. ¹H NMR (300 MHz, CDCl₃, ppm): δ = 4.20 (s, 4 H), 7.26–7.44 (m, 11 H), 7.79 (td, *J* = 7.7, 1.7 Hz, 1 H), 8.13 (dt, *J* = 7.7, 1.1 Hz, 1 H), 8.40–8.42 (m, 1 H), 8.82 (br. s, 1 H). ¹³C{¹H} NMR (75 MHz, CDCl₃, ppm): δ = 59.9, 122.2, 126.3, 127.5, 128.4, 129.2, 137.0, 137.3, 148.0, 149.7, 162.6. IR (solid, cm⁻¹): 3290, 3028, 2930, 2820, 1664, 1517, 748. IR (CHCl₃, cm⁻¹): 3309, 3031, 3011, 2929, 1682, 1519, 1495. HRMS (ESI): *m/z* calcd. for C₂₀H₁₉N₃ONa [M + Na]⁺ 340.1425; found 340.1424.

General procedure for the synthesis of picolinohydrazides L3-L6 with CDI: To a suspension of 1 (1.2 equiv.) in CH_2Cl_2 (stabilized with EtOH) was added CDI (1.2 equiv.), followed by the hydrazine (1 equiv.). The reaction mixture was stirred overnight at room temperature, washed with water (x3), dried over anhydrous MgSO₄, and evaporated to dryness.

N-(**Piperidin-1-yl**)**picolinamide (L3):** Following the general procedure with 1 (2 g, 16.2 mmol), 4 (1.35 g, 13.5 mmol), CDI (2.63 g, 16.2 mmol), and CH₂Cl₂ (15 mL). Purification by trituration of the crude product in PE / Et₂O (3 / 1). Off white solid (2.07 g, 75% yield); m.p. 99 °C. ¹H NMR (300 MHz,

CDCl₃, ppm): $\delta = 1.35$ (m, 2 H), 1.64 (m, 4 H), 2.76 (t, J = 6.0 Hz, 4 H), 7.32 (ddd, J = 7.8, 4.8, 1.0 Hz, 1 H), 7.72 (td, J = 7.8, 1.7 Hz, 1 H), 8.09 (dt, J = 7.8, 1.0 Hz, 1 H), 8.41 (ddd, J = 4.8, 1.7, 0.9 Hz, 1 H), 8.62 (br. s, 1 H). ¹³C{¹H} NMR (75 MHz, CDCl₃, ppm): $\delta = 23.1$, 25.1, 56.9, 122.3, 126.1, 137.1, 147.7, 149.8, 160.8. IR (solid, cm⁻¹): 3214, 3048, 2943, 2923, 2853, 2825, 1665, 1519, 671. IR (CHCl₃, cm⁻¹): 3299, 3062, 2989, 2945, 2860, 1685, 1526. HRMS (ESI): *m/z* calcd. for C₁₁H₁₅N₃ONa [M + Na]⁺ 228.1112; found 228.1113.

N'-Phenylpicolinohydrazide (L4): Following the general procedure with 1 (1.37 g, 11.1 mmol), **5** (1 g, 9.2 mmol), CDI (1.8 g, 11.1 mmol), and CH₂Cl₂ (10 mL). Purification by trituration of the crude solid in EtOH and filtration. Yellow solid (1.14 g, 58% yield); m.p. 192 °C. ¹H NMR (300 MHz, CDCl₃, ppm): δ = 6.29 (d, *J* = 6.0 Hz, 1 H), 6.89–6.95 (m, 3 H), 7.22–7.27 (m, 2 H), 7.49 (ddd, *J* = 9.0, 6.5, 1.5 Hz, 1 H), 7.88 (td, *J* = 6.5, 1.5 Hz, 1 H), 8.18 (dt, *J* = 9.0, 1.0 Hz, 1 H), 8.61 (m, 1 H), 9.57 (d, *J* = 6.0 Hz, 1 H). IR (solid, cm⁻¹): 3218, 3058, 1656, 1493, 757, 742, 674. HRMS (ESI): *m/z* calcd. for C₁₂H₁₁N₃ONa [M + Na]⁺ 236.0799; found 236.080.

N'-(Pyridin-2-yl)picolinohydrazide (L5): Following the general procedure with 1 (500 mg, 4.1 mmol), **6** (370 mg, 3.4 mmol), CDI (665 mg, 4.1 mmol), and CH₂Cl₂ (5 mL). Purification by recrystallization of the crude product in EtOH. Yellow solid (502 mg, 69% yield); m.p. 147 °C. ¹H NMR (300 MHz, CDCl₃, ppm): δ = 6.73–6.78 (m, 2 H), 7.43–7.51 (m, 2 H), 7.84 (td, *J* = 7.8, 1.7 Hz, 1 H), 8.15–8.19 (m, 2 H), 8.60 (ddd, *J* = 4.8, 1.7, 0.9 Hz, 1 H), 10.10 (br. s, 1 H). IR (solid, cm⁻¹): 3192, 3059, 2976, 2950, 1663, 1594, 782, 743. HRMS (ESI): *m/z* calcd. for C₁₁H₁₀N₄ONa [M + Na]⁺ 237.0752; found 237.0751.

N',N'-Dibenzyl-*N*-methylpicolinohydrazide (L6): Following the general procedure with 1 (726 mg, 5.9 mmol), 7 (1.1 g, 4.9 mmol), CDI (957 mg, 5.9 mmol), and CH_2Cl_2 (10 mL). Purification by trituration of the crude solid in PE and filtration. White solid (824 mg, 52% yield); m.p. 95 °C. ¹H

NMR (300 MHz, CDCl₃, ppm): δ = 3.28 (s, 3 H), 3.74 (d, *J* = 12.7 Hz, 2 H), 4.05 (d, *J* = 12.7 Hz, 2 H), 6.31 (dt, *J* = 7.7, 1.0 Hz, 1 H), 6.93–6.96 (m, 4 H), 7.21–7.31 (m, 7 H), 7.54 (td, *J* = 7.7, 1.7 Hz, 1 H), 8.56 (ddd, *J* = 4.8, 1.7, 0.9 Hz, 1 H). ¹³C{¹H} NMR (75 MHz, CDCl₃, ppm): δ = 24.8, 56.9, 121.1, 123.3, 127.7, 128.4, 129.4, 136.0, 136.4, 147.8, 156.0, 171.4. IR (solid, cm⁻¹): 3061, 3028, 2843, 1644, 749, 699. HRMS (ESI): *m/z* calcd. for C₂₁H₂₁N₃ONa [M + Na]⁺ 354.1582; found 354.1583.

General procedure for the synthesis of picolinohydrazides L7 and L8 by hydrazinolysis: The hydrazine (5 equiv.) was added to a solution of 8 (1 equiv.) in EtOH. The reaction mixture was stirred for 4 days at room temperature, and evaporated to dryness.

Picolinohydrazide (L7): Following the general procedure with **8** (3 g, 20 mmol), **9** (5 g, 0.1 mol), and EtOH (20 mL). Purification by recrystallisation of the crude product in toluene. Colourless needles (1.55 g, 57% yield); m.p. 100 °C. ¹H NMR (300 MHz, CDCl₃, ppm): δ = 3.98 (br. s, 2 H), 7.43 (ddd, *J* = 7.8, 4.8, 1.1 Hz, 1 H), 7.85 (td, *J* = 7.8, 1.7 Hz, 1 H), 8.16 (dt, *J* = 7.8, 1.1 Hz, 1 H), 8.55 (ddd, *J* = 4.8, 1.7, 0.9 Hz, 1 H). ¹³C{¹H} NMR (75 MHz, CDCl₃, ppm): δ = 122.3, 126.5, 137.4, 148.5, 149.1, 164.8. IR (solid, cm⁻¹): 3303, 3210, 1672, 1644, 1593, 1522, 973, 620.

N-Methylpicolinohydrazide (L8): Following the general procedure with 8 (2 g, 13.2 mmol), 10 (3.1 g, 66 mmol), and EtOH (13 mL). A catalytic amount of NaCN was also added in order to achieve completion of the reaction. Purification by trituration of the crude yellow oil in PE and filtration. White solid (1.70 g, 85% yield); m.p. 53 °C. ¹H NMR (300 MHz, CDCl₃, ppm): δ = 2.72 (s, 3 H), 6.70 (br. s, 2 H), 7.39 (ddd, *J* = 7.7, 4.8, 1.2 Hz, 1 H), 7.80 (td, *J* = 7.8, 1.7 Hz, 1 H), 8.12 (dt, *J* = 7.7, 1.2 Hz, 1 H), 8.51 (ddd, *J* = 4.8, 1.7, 0.9 Hz, 1 H). ¹³C{¹H} NMR (75 MHz, CDCl₃, ppm): δ = 39.3, 122.2, 126.3, 137.3, 148.3, 149.2, 163.3. IR (solid, cm⁻¹): 3382, 3285, 3254, 2955, 1661, 1588, 1485, 1457, 1428, 996, 815, 738, 689. HRMS (ESI): *m/z* calcd. for C₇H₉N₃ONa [M + Na]⁺ 174.0643; found 174.0642.

Synthesis of *N*'-Benzylpicolinohydrazide (L9): A mixture of 11 (1 g, 4.4 mmol) and NaBH₃CN (0.39 g, 6.2 mmol) was placed in EtOH (10 mL), followed by addition of an aqueous 1 N HCl solution to adjust the pH of the reaction medium to 3–5. The reaction was stirred at room temperature overnight and then quenched by addition of an aqueous NaHCO₃ solution until pH = 10. After evaporation of the organic solvent under vacuum, the residue was extracted with CH₂Cl₂ (x3), and the organic layers were dried over anhydrous MgSO₄, and evaporated to dryness. The crude compound was triturated in PE and the solid was collected by filtration. White powder (757 mg, 76% yield); m.p. 66 °C. ¹H NMR (300 MHz, CDCl₃, ppm): δ = 4.11 (d, *J* = 3.9 Hz, 2 H), 5.09 (m, 1 H), 7.26–7.43 (m, 6 H), 7.84 (td, *J* = 7.8, 1.8 Hz, 1 H), 8.16 (dt, *J* = 7.8, 1.2 Hz, 1 H), 8.50 (ddd, *J* = 4.8, 1.5, 0.9 Hz, 1 H), 9.26 (d, *J* = 4.8 Hz, 1 H). ¹³C {¹H} NMR (75 MHz, CDCl₃, ppm): δ = 56.2, 122.3, 126.5, 127.7, 128.6, 129.0, 137.4, 137.5, 148.4, 149.2, 163.6. IR (solid, cm⁻¹): 3278, 3255, 3065, 3027, 2858, 1642, 1454, 815, 748, 698, 658. HRMS (ESI): *m/z* calcd. for C₁₃H₁₃N₃ONa [M + Na]⁺ 250.0956; found 250.0954. X-ray-quality crystals were grown by slow diffusion of pentane into a CH₂Cl₂ solution of L9.



1,1-Dibenzyl-2-methylhydrazine (7)^[1]

Compound $A^{[2]}$ (3 g, 12.7 mmol) and benzaldehyde (2 g, 19 mmol) were dissolved in EtOH (30 mL). NaBH₃CN (1.2 g, 19 mmol) was added and the pH was adjusted to 5 using an aqueous 2 N HCl solution. After 4 h of stirring, water (30 mL) was poured into the solution, and then solid Na₂CO₃ was added fractionally until pH stabilizes at 8. EtOH was eliminated under reduced pressure and the resulting murky solution was extracted with CH₂Cl₂ (2 x 30 mL). After drying and evaporation, the organic phase gave a crude product containing a mixture of benzyl alcohol, **A** (*ca* 1.5 g), and **B** (*ca* 2 g). This crude mixture was placed in EtOH (30 mL) before adding 1.5 equivalent of NaBH₃CN (0.8 g) and glyoxylic acid hydrate (1.2 g) relative to the residual amount of compound **A**. The solution was stirred for 12 h, then acidified to pH = 2 using an aqueous 2 N HCl solution. After removal of EtOH, the aqueous phase was extracted with CH₂Cl₂ (2 x 50 mL). The organic layers were dried on Na₂SO₄ and evaporated to dryness. This residue was dissolved in Et₂O (50 mL), and washed with an aqueous 1 N NaHCO₃ solution (2 x 30 mL) in order to remove undesired compound **C**. The organic phase was dried on Na₂SO₄, filtered, and evaporated to afford hydrazine **B**. Colorless oil (2 g, 48%). ¹H NMR (300 MHz, CDCl₃, ppm): $\delta = 1.49$ (s, 9 H), 2.71 (s, 3 H), 3.90 (d, J = 18 Hz, 2 H), 4.34 (d, J = 18 Hz, 2 H).

7.22–7.48 (m, 10 H). Compound **B** (2 g, 6.13 mmol) was next dissolved in MeOH (30 mL) and cooled in an ice bath before dropwise addition of SOCl₂ (1.4 g, 11.8 mmol). After 6 h of stirring, evaporation under reduced pressure gave a white residue which was triturated in Et₂O and filtrated to release **7.HCl**. White powder (1.55 g, 46% overall yield). ¹H NMR (300 MHz, D₂O, ppm): δ = 2.77 (s, 3 H), 4.06 (s, 4 H), 7.29–7.44 (m, 10 H). ¹³C{¹H} NMR (75 MHz, D₂O, ppm): δ = 30.1, 56.2, 128.8, 129.0, 129.3 (one overlapped peak). *Prior to use*, **7.HCl** was taken up in an aqueous 1 N NaHCO₃ solution and stirred for 1 h. Extraction with CH₂Cl₂ (20 mL), drying over Na₂SO₄ and evaporation gave the free hydrazine 7, which was used without further purification.

N'-Benzylidenepicolinohydrazide (11)

A mixture of L7 (1 g, 7.3 mmol) and benzaldehyde (0.93 g, 8.8 mmol) in Et₂O (5 mL) and EtOH (5 mL) was stirred at room temperature overnight. The solid formed was collected by filtration. White solid (1.48 g, 90% yield); m.p. 194 °C. ¹H NMR (300 MHz, CDCl₃, ppm): δ = 7.38–7.40 (m, 3 H), 7.46 (ddd, *J* = 7.5, 4.8, 1.2 Hz, 1 H), 7.79–7.82 (m, 2 H), 7.87 (td, *J* = 7.8, 1.8 Hz, 1 H), 8.29 (dt, *J* = 7.8, 1.2 Hz, 1 H), 8.30 (s, 1 H), 8.55 (ddd, *J* = 4.8, 1.8, 0.9 Hz, 1 H), 11.01 (s, 1 H). ¹³C{¹H} NMR (75 MHz, CDCl₃, ppm): δ = 123.0, 126.8, 127.9, 128.7, 130.6, 133.7, 137.7, 148.1, 148.9, 149.1, 160.1. IR (solid, cm⁻¹): 3209, 3057, 1661, 1588, 1570, 1515, 1486, 1361, 1137, 766, 666. HRMS (ESI): *m/z* calcd. for C₁₃H₁₁N₃ONa [M + Na]⁺ 248.0799; found 248.0797.

Methyl N-benzamido-N-benzylglycinate (15)

To a solution of benzoic acid (265 mg, 2.17 mmol) in THF (15 mL) and DMF (15 mL) were added **2.HCl** (500 mg, 2.17 mmol), K_2CO_3 (1.2 g, 8.7 mmol), and TBTU (697 mg, 2.17 mmol). The reaction was stirred overnight at room temperature. After addition of water, the mixture was extracted with EtOAc (x3), the combined organic phases were washed with brine (x3), dried over anhydrous MgSO₄, and evaporated to dryness. The crude product was then purified by flash chromatography on silica gel eluting with PE / EtOAc (2 / 1). White solid (420 mg, 65% yield, unoptimized); m.p. 70 °C. ¹H

NMR (300 MHz, CDCl₃, ppm): δ = 3.73 (s, 3 H), 3.89 (s, 2 H), 4.28 (s, 2 H), 7.27–7.61 (m, 8 H), 7.63 (d, *J* = 6.0 Hz, 2 H), 8.20 (br. s, 1 H). ¹³C{¹H} NMR (75 MHz, CDCl₃, ppm): δ = 51.9, 55.1, 60.2, 127.1, 127.9, 128.6, 128.7, 129.3, 131.8, 133.6, 136.5, 166.3, 171.7. IR (solid, cm⁻¹): 3243, 3063, 3030, 2952, 2850, 1746, 1644, 1546, 1282, 1074, 689. HRMS (ESI): *m/z* calcd. for C₁₇H₁₈N₂O₃Na [M + Na]⁺ 321.1215; found 321.1211.

4) General procedure for the zinc-catalyzed hydrosilylation of ketones

In a flame-dried round-bottom flask, to a solution of the ligand (0.05 mmol) in dry toluene (2 mL) under an atmosphere of argon was added ZnEt₂ (50 μ L, 1 M solution in *n*-hexane, 0.05 mmol). After 10 min, the ketone (1 mmol) and (EtO)₂MeSiH (194 μ L, 1.2 mmol) were injected and the reaction was stirred overnight at room temperature. After removal of the solvent under reduced pressure, MeOH (4 mL) and an aqueous 1 N NaOH solution (4 mL) were added and the resulting solution was stirred for 12 h. The reaction medium was quenched with an aqueous 1 N HCl solution (10 mL) and brine (10 mL), and extracted with Et₂O (3 x 10 mL). The combined organic layers were washed with an aqueous 1 N HCl solution (10 mL) and brine (10 mL). After drying over anhydrous MgSO₄, filtration and evaporation to dryness, the residue was submitted to ¹H NMR analysis to determine the conversion. The crude product was then purified by silica gel column chromatography using PE / Et₂O mixtures. ¹H and ¹³C NMR data for all products were in agreement with reported values.

5) ¹H and ¹³C NMR spectra of the new compounds









¹³C NMR spectrum of L6 (75 MHz, CDCl₃)





¹³C NMR spectrum of 11 (75 MHz, CDCl₃)



¹³C NMR spectrum of L9 (75 MHz, CDCl₃)

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6) X-ray diffraction study of L9

Data collection was carried out with a D8 Venture Bruker AXS diffractometer at 150 K. The structure was solved by direct methods using the *SIR97* program,^[3] and then refined with full-matrix least-square methods based on F^2 (*SHELXL-2014*).^[4] All non-hydrogen atoms were refined with anisotropic atomic displacement parameters. Except nitrogen linked hydrogen atoms that were introduced in the structural model through Fourier difference maps analysis, H atoms were finally included in their calculated positions. Details of the data collection, cell dimensions, and structure refinements are given below.

formula	$C_{13}H_{13}N_{3}O$
fw	227.26
crystal system	monoclinic
space group	$P 2_l/n$
a, Å	9.6822(4)
b, Å	8.6332(3)
<i>c</i> , Å	14.1256(6)
α, deg	90
β, deg	102.254(2)
γ, deg	90
$V, \text{\AA}^3$	1153.83(8)
Ζ	4
$d_{\text{calcd}}, \text{g/cm}^3$	1.308
θ range, deg	2.783-27.512
μ , mm ⁻¹	0.086
no. of obsd data, $I > 2\sigma(I)$	2262
data / restraints / parameters	2622 / 0 / 160
R1 (all data) ^a	0.0448
wR2 (all data) ^b	0.0984
$(\Delta \rho)_{\rm min}, e.{\rm \AA}^{-3}$	-0.202

Selected crystallographic data and collection parameters for L9



7) ¹H NMR of the crude product in the reduction of acetophenone (12a) with L1



8) References

- [1] See also: Spinks, A.; Young, E. H. P. Proc. Inter. Pharmacol. Meeting 1963, 7, 303–311.
- [2] Le Grel, P.; Asprogenidi, A.; Huez, P.; Le Grel, B.; Salaün, A.; Roisnel, T.; Potel, M.; Rasti, E.;Hocquet, A. *Chirality* 2013, *25*, 341–349.
- [3] Altomare, A.; Burla, M. C.; Camalli, M.; Cascarano, G.; Giacovazzo, C.; Guagliardi, A.;Moliterni, A. G. G.; Polidori, G.; Spagna, R. J. Appl. Cryst. 1999, 32, 115–119.
- [4] Sheldrick, G. M. Acta Cryst. C71 2015, 3-8.