The influence of the ligand chelate effect on iron-amine-catalysed Kumada cross-coupling

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SUPPORTING INFORMATION

Crystallographic Experimental

X-ray diffraction experiments on single crystal of **9** were carried out at 100K on a Bruker Proteum Microstar diffractometer using Cu-K_α radiation (λ = 1.54178 Å) and on **8** at 100K on a Bruker APEX II diffractometer using Mo-K_α radiation (λ = 0.71073 Å). Data collections were performed using a CCD area detector from a single crystal mounted on a glass fibre. Intensities were integrated[S1] from several series of exposures measuring 0.5° in ω or φ. Absorption corrections were based on equivalent reflections using SADABS.[S2] The structures were solved using SHELXS and refined against all Fo2 data with hydrogen atoms riding in calculated positions using SHELXL.[S3] The structures have been deposited at the Cambridge Crystallographic Data Centre, CCDC numbers for **8** and **9**, CCDC 1478297 and CCDC 1478298 respectively.

Crystallographic Data

Identification code	**8**	**9**·3CH_3CN
Empirical formula	$C_{12}H_{30}Cl_2FeN_4$	$C_{20}H_{41}Cl_2FeN_7$
Formula weight	357.15	506.35
Temperature/K	100	100
Crystal system	monoclinic	triclinic
Space group	C2/c	P-1
a/Å	14.884(3)	8.4114(17)
b/Å	7.9168(16)	13.145(3)
c/Å	14.972(3)	13.243(3)
α/°	90	65.49(3)
β/°	104.91(3)	81.50(3)
γ/°	90	81.42(3)
Volume/Å^3	1704.7(6)	1311.5(6)
Z	4	2
ρ_{calc}g/cm^3	1.392	1.282
μ/mm^{-1}	1.192	0.799
F(000)	760.0	540.0
Crystal size/mm^3	0.24 × 0.2 × 0.15	0.6 × 0.4 × 0.4
Radiation	MoKα (λ = 0.71073)	CuKα (λ = 1.54178)
2Θ range for data collection/°	5.632 to 54.964	3.396 to 51.728
Index ranges	-19 ≤ h ≤ 19 -10 ≤ k ≤ 10 -18 ≤ l ≤ 19	-6 ≤ h ≤ 9 -15 ≤ k ≤ 15 -15 ≤ l ≤ 15
Reflections collected	11668	8897
Independent reflections	1951 [R_{int} = 0.0560, R_{sigma} = 0.0429]	4071 [R_{int} = 0.0353, R_{sigma} = 0.0508]
Data/restraints/parameters	1951/0/90	4071/0/278
Goodness-of-fit on F^2	0.891	1.206
Final R indexes [I>=2σ (I)]	R_1 = 0.0383, wR_2 = 0.1083	R_1 = 0.0512, wR_2 = 0.1239
Final R indexes [all data]	R_1 = 0.0588, wR_2 = 0.1245	R_1 = 0.0515, wR_2 = 0.1240
Largest diff. peak/hole / e Å^{-3}	0.47/-0.58	0.61/-0.49

Figure S1. Structure of complex **8**, thermal ellipsoids set at 50% probability, hydrogen atoms omitted for clarity.

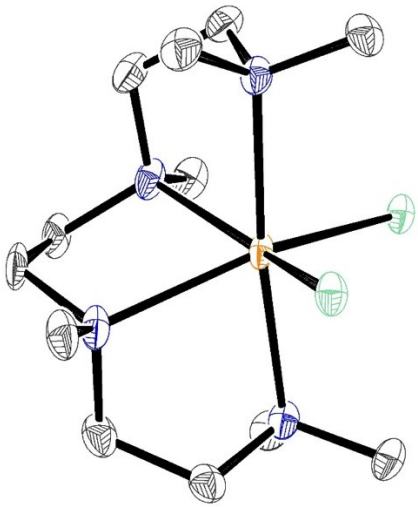
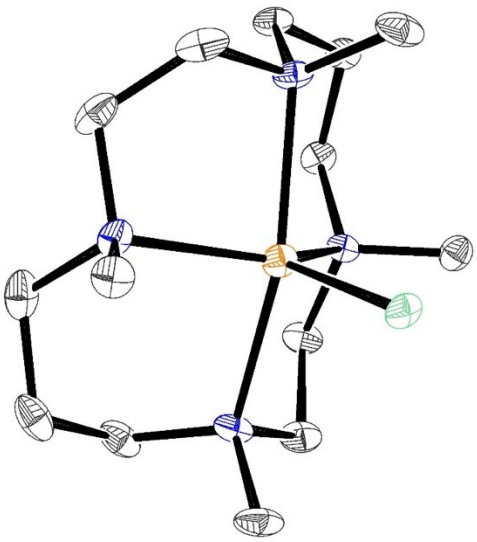


Figure S2. Structure of complex **9**, thermal ellipsoids set at 50% probability, hydrogen atoms and three molecules of MeCN omitted for clarity.



References

S1. Bruker-AXS SAINT V7.68A, Madison, Wisconsin.

S2. G. M. Sheldrick, SADABS V2008/1, University of Göttingen, Germany.

S3. G. M. Sheldrick, Acta Cryst. 2008, **A64**, 112.