Supplementary Material (ESI) for Chemical Communications

An improved method for the synthesis of zirconiuim (CCC-N-heterocyclic carbene) pincer complexes and applications in hydroamination

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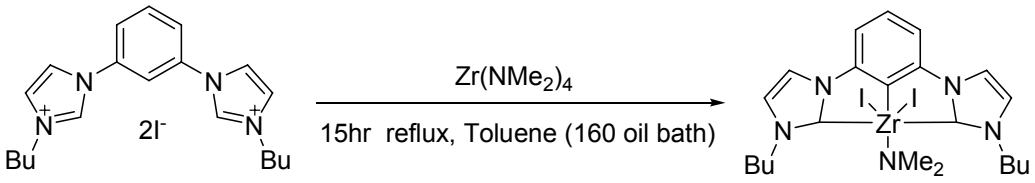
Supporting Information

2-(1,3-*bis*(N-butyl-imidazol-2-ylidene)phenylene)(dimethylamido) bis (iodo) zirconium (IV), 3



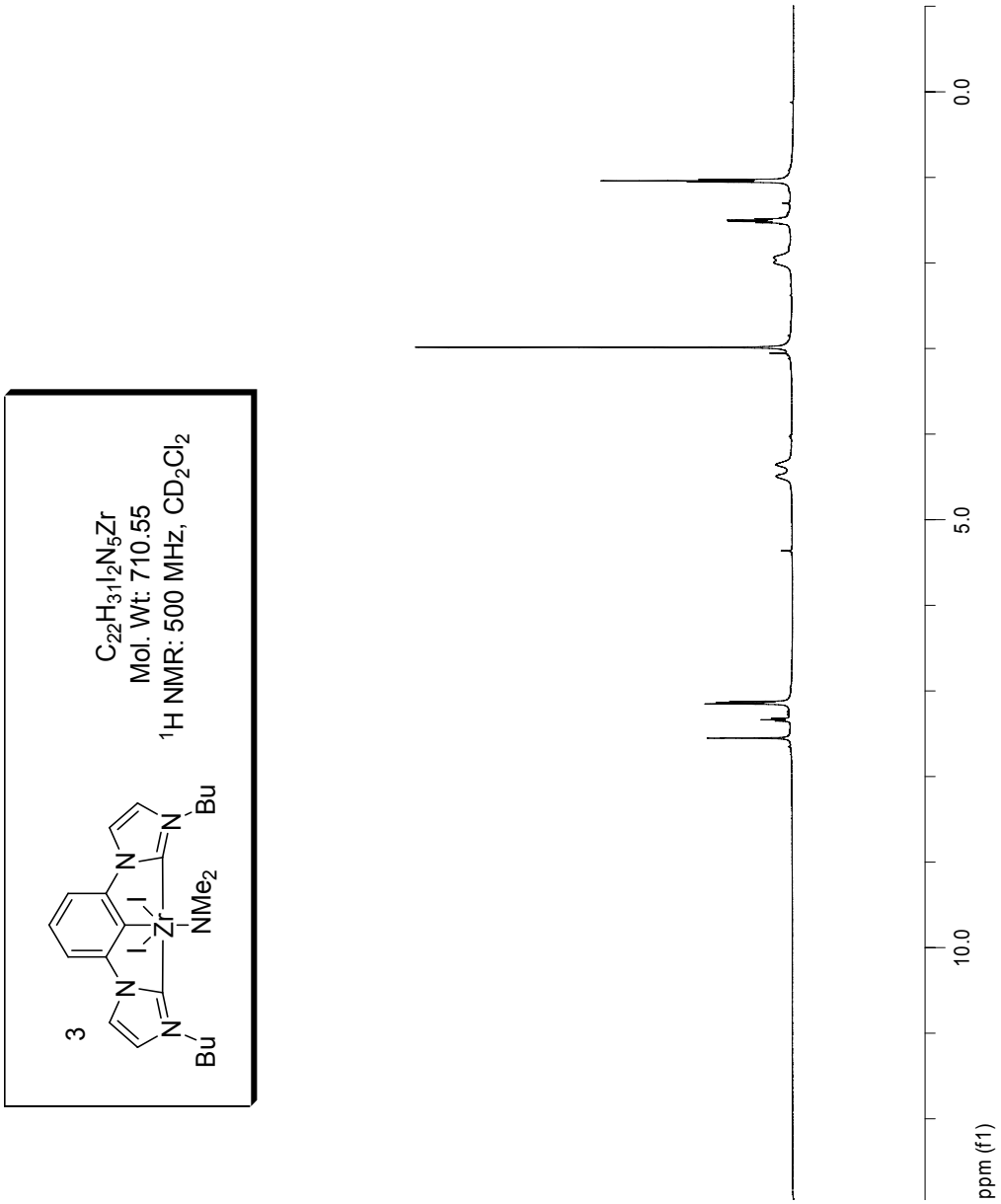
1,3-*Bis*(1-butylimidazol-3-yl) benzene diiodide **1** (2.17 g, 3.75 mmol), $Zr(NMe_2)_4$ (1.10 g, 4.12 mmol) and toluene (150 mL) were combined in sealable reaction tube. The resulting mixture was stirred for 15 h in a 160 ^oC oil bath. The reaction was cooled to room temperature during which time a solid precipitated. It was collected and dried yielding a lemon-color solid **3** (1.77 g, 67%): ^1H NMR (CD_2Cl_2) δ 7.51 (s, 2H), 7.30 (t, J = 8 Hz, 1H), 7.11 (s, 2H), 7.09 (d, J = 8 Hz, 2H), 4.44 (br s, 2H), 4.32 (br s, 2H), 2.95 (s, 6H), 1.95 (br s, 2H), 1.90 (br s, 2H), 1.47 (sextet, J = 7.5 Hz, 4H), 1.00 (t, J = 7.5 Hz, 6H)); $^{13}C\{^1H\}$ (125 MHz, CD_2Cl_2): δ 193.7, 164.8, 146.9, 129.2, 121.8, 115.7, 110.6, 52.2, 42.4, 34.1, 20.3, 14.2. Anal. Calcd For $C_{22}H_{31}I_2N_5Zr$: C, 37.19; H, 4.40; N, 9.86. Found: C, 37.19; H, 4.17; N, 9.79.

Experimental Procedure for hydroamination

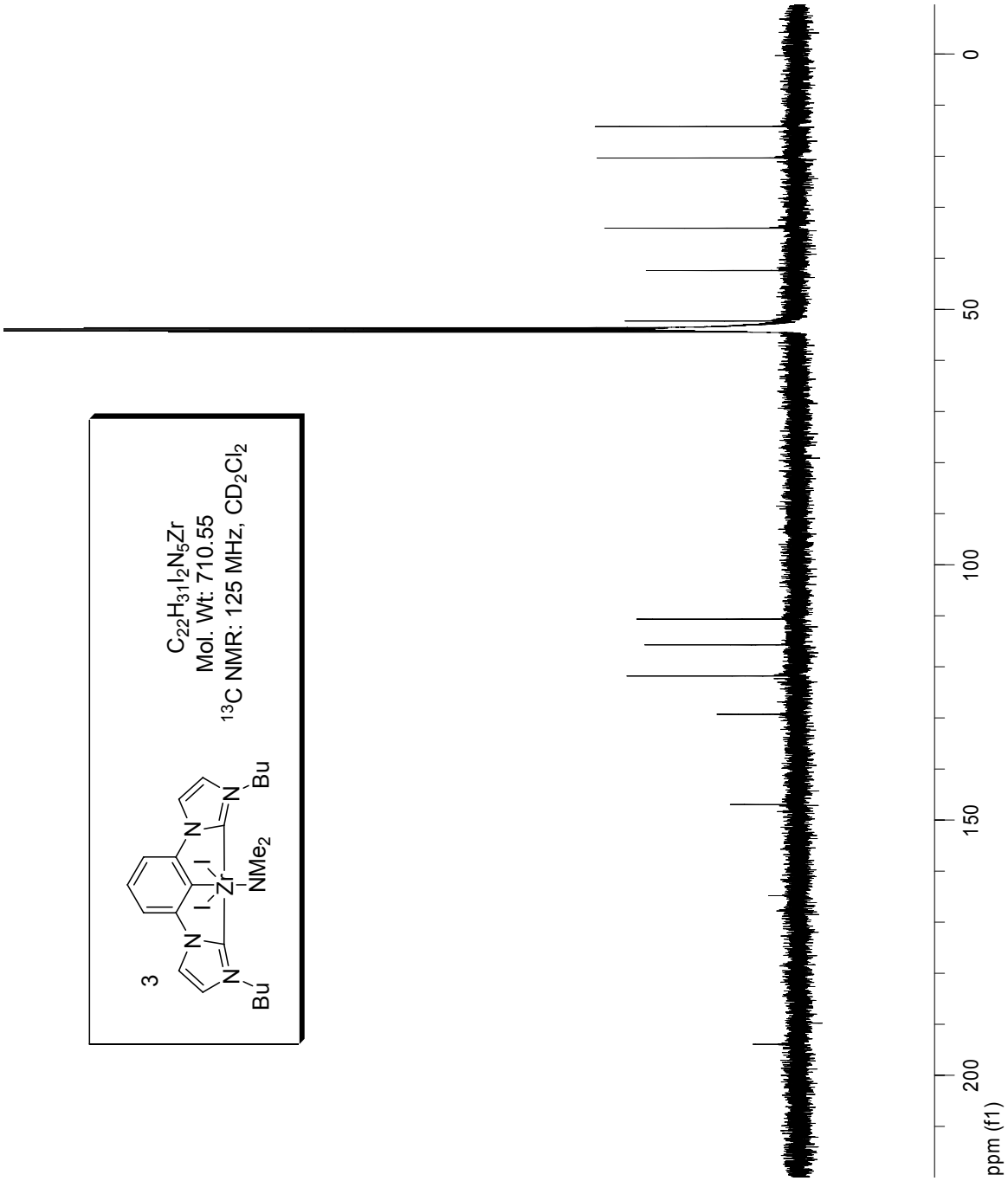
Substrates for hydroamination were prepared according to literature procedures.[1] All

(1) (a) Thompson, R. K.; Bexrud, J.A.; Schafer, L. L. *Organometallics* **2006**, *25*, 4069. (b) Watson, D. A.; Chiu, M.; Bergman, R. G. *Organometallics* **2006**, *25*, 4731. (c) Kim, H. S.; Kim, Y. K.; Shim, J. H.; Kim, M. S.; Han, M. J.;

hydroaminaton reactions were performed using screw-cap or J. Young NMR tube. The substrate, catalyst and deuterated solvent were combined under an argon atmosphere and heated in an oil bath. The samples were removed from heat for NMR analyses.



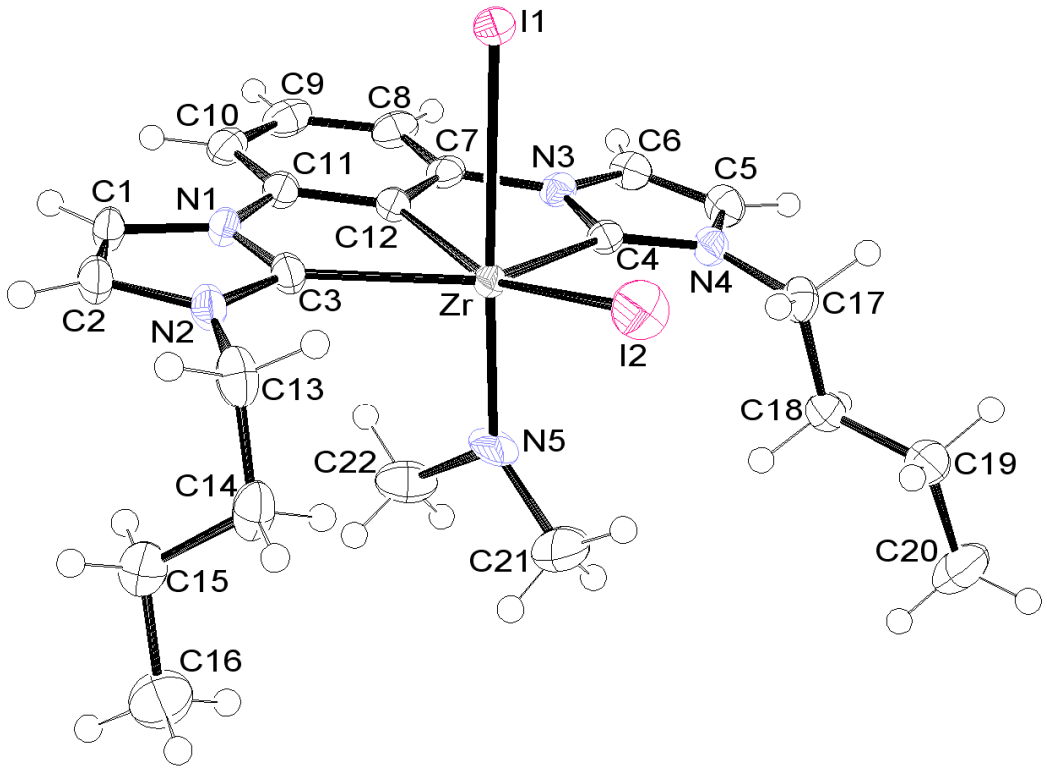
Livinghouse, T.; Lee, P. H. *Adv. Synth. Catal.* **2006**, *348*, 2609. (d) Wood, M. C.; Leitch, D. C.; Yeung, C. S.; Kozak, J. A.; Schafer, L. L. *Angew. Chem. Int. Ed.* **2007**, *46*, 354. (e) Gott, A. L.; Clarke, A. J.; Clarkson, G. J.; Scott, P. *Organometallics* **2007**, *26*, 1729. (f) Stubbert, B. D.; Marks, T. J. *J. Am. Chem. Soc.* **2007**, *129*, 6149.


3
Bu
Bu
NMe2
I
I
Zr
N
N
N
N
C22H31I2N5Zr
Mol. Wt: 710.55
13C NMR: 125 MHz, CD2Cl2
0
50
100
150
200
ppm (f1)

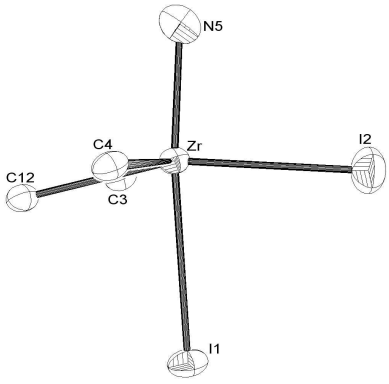
Supplementary Material (ESI) for Chemical Communications

CRYSTAL STRUCTURE REPORT

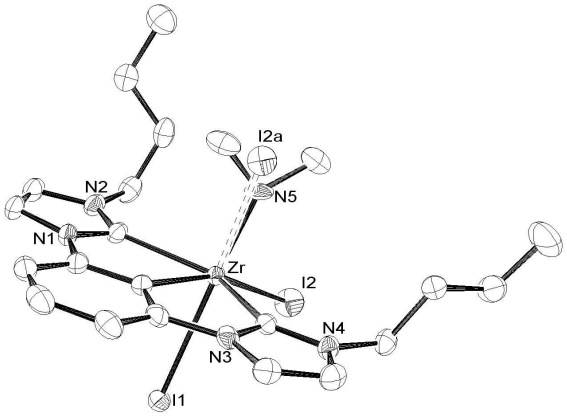
3, $C_{22}H_{33}N_5I_2Zr$



Plot of the coordination sphere of **3.**



Plot of the disorder in the coordination sphere of **3**.



Data collection

A bright yellow air-sensitive prism was carried to the laboratory immersed in a thick protective oil under an argon atmosphere. The specimen was retrieved from the oil by pipette, and a specimen of dimensions 0.27 x 0.38 x 0.52 mm^3 was transferred to an 0.7mm nylon loop within a droplet of oil, and the loop was attached to a stout glass fibre and mounted on a goniometer head. The crystallographic properties and data were collected using MoKα radiation and the charge-coupled area detector (CCD) detector on an Oxford Diffraction Systems Gemini S diffractometer at 153(1)K.[2] A preliminary set of cell constants was calculated from reflections observed on three sets of 5 frames which were oriented approximately in mutually orthogonal directions of reciprocal space. Data collection was carried out using MoKα radiation (graphite monochromator) with 9 runs consisting of 382 frames with a frame time of 20.0 sec, and a crystal-to-CCD distance of 50.000mm. The runs were collected by omega scans of 1.0 degree width, and at detector positions of -30.031 and 28.624 degrees in 2θ. The intensity data were corrected for absorption with an analytical correction.[3] Final cell constants were calculated from 12058 stronger reflections from the actual data collection after integration. See Table 1 for crystal and refinement information.

Structure solution and refinement

The structure was solved using by the Patterson function in SHELXS-86, and refined using SHELXL.[4] The space group P2(1)/n (#14) was determined based on the cell, systematic absences, intensity statistics, and successful solution structure and refinement. The positions of the zirconium and two iodide ions were found from the Patterson map, and the remaining C, N and H atoms were found in successive difference Fourier computations. All non-hydrogen atoms were refined with anisotropic displacement parameters. Hydrogen atoms were placed in ideal positions; all were refined as riding atoms with relative isotropic displacement parameters. There is evidence of a slight disorder between axial and equatorial I / NMe_2. This has been

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(2) CrysalisPro (2007), Version 171.32.5, Oxford Diffraction Ltd., Abingdon, Oxfordshire, OX14 4RX, United Kingdom.

(3) ABSORPTION CORRECTION (ANALYTICAL) - C. Katayama, Acta Crystallogr., Sect A 1986, 42, 19-23.

(4) a) SHELX97 [Includes SHELXS97, SHELXL97, CIFTAB] - Programs for Crystal Structure Analysis (Release 97-2). G. M. Sheldrick, Institüt für Anorganische Chemie der Universität, Tammanstrasse 4, D-3400 Göttingen, Germany, 1998. b) SHELXS86 - G. M. Sheldrick, In "Crystallographic Computing 3", Ed. G. M. Sheldrick, C. Kruger and R. Goddard, Oxford University Press. pp. 175-189, 1985. SHELXS86 - Program for Crystal Structure solution. G. M. Sheldrick, Institüt für Anorganische Chemie der Universität, Tammanstrasse 4, D-3400 Göttingen, Germany, 1986.

modeled with a second axial iodine (with an isotropic vibrational factor); an occupancy factor for the equatorial / axial positions refines to 0.94(3) / 0.06(3). In the vicinity of the equatorial iodide (I2), there are several small difference positive electron density peaks off the Zr-I2 bond axis; it has not proved possible to model these peaks with a low occupancy dimethylamide group due to the dominance of the electron density feature of the higher occupancy iodide (I2). But in contrast, there are no significant positive difference electron density features near I1 and off the Zr-I1 bond axis. Thus the model supports the interpretation that there is a small disorder between equatorial and axial I2/NMe2 groups as described, as opposed to an model which includes presence of a small amount of a tri-iodide complex. An alternative model with the disorder between the axial iodide (I1) and the dimethylamide group produced a significantly inferior refinement. The final full-matrix least-squares refinement converged to R1 = 0.0363 and wR2 = 0.0863 (F^2, $I > 2\sigma(I)$), 277 parameters, 0 restraints, goodness-of-fit (S) 1.045.

Structure description

The structure consists of the discrete molecular complexes of formula $C_{22}H_{33}N_5I_2Zr$. The zirconium is six coordinate with distorted octahedral geometry. The tridentate biscarbene ligand ($C_{19}H_{27}N_4$) binds the zirconium approximately in the equatorial plane, with an iodide completing the coordination in the approximate plane. A second iodide is axial, as is a dimethylamide nitrogen. Zr-C bond lengths are 2.367(3) and 2.362(3)Å at the ends, and 2.310(3)Å at the middle of the ligand; and Zr-I2(eq) is 2.8431(4) while Zr-I1(ax) is 3.0038(4)Å, and Zr-N(ax) is 1.986(3)Å.

Other Information

Data collection and structure solution were conducted at the Mississippi College Diffraction Facility, 413 Hederman Science, Department of Chemistry & Biochemistry, Mississippi College, Clinton, MS, 39058. All calculations were performed using Pentium computers using the current SHELX suite of programs.

Relevant Equations used in this report:

$$R_{int} = \Sigma\, |F_o^2 - < F_o^2 >| \,/\, \Sigma |F_o^2|$$

$$R_1 = \Sigma ||F_o| - |F_c|| \,/\, \Sigma |F_o|$$

$$wR2 = [\Sigma[w(F_o^2 - F_c^2)^2] \,/\, \Sigma\, [w(F_o^2)^2]]^{1/2}$$

where $w = q \,/\, [\sigma^2 (F_o^2) + (a*P)^2 + b*P + d + e*\sin(\theta)]$

$$\text{GooF} = S = [\Sigma[w(F_o^2 - F_c^2)^2] \,/\, (n-p)]^{1/2}$$

Table 1. Crystal data and structure refinement for 3.

Empirical formula	$C_{22}H_{31}I_2N_5Zr$
Formula weight	710.56
Temperature	153(2) K
Wavelength	0.71073 A
Crystal system, space group	Monoclinic, P 1 21/n 1 (#14)
Unit cell dimensions	a = 10.1754(2) A alpha = 90 deg.
	b = 23.4958(4) A beta = 98.495(2) deg.
	c = 10.8780(2) A gamma = 90 deg.
Volume	2572.17(8) A^3
Z, Calculated density	4, 1.835 Mg/m^3
Absorption coefficient	2.846 mm^-1
F(000)	1376
Crystal size	0.5239 x 0.3841 x 0.2732 mm
Theta range for data collection	3.09 to 30.64 deg.
Limiting indices	-14<=h<=14, -33<=k<=33, -14<=l<=15
Reflections collected / unique	20493 / 7719 [R(int) = 0.0199]
Completeness to theta = 30.64	97.0 %
Absorption correction	Analytical
Max. and min. transmission	0.506 and 0.318
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	7719 / 0 / 277
Goodness-of-fit on F^2	1.045
Final R indices [I>2sigma(I)]	R1 = 0.0363, wR2 = 0.0863
R indices (all data)	R1 = 0.0535, wR2 = 0.0941
Largest diff. peak and hole	2.619 and -2.273 e.A^-3

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for 3.

Atom	x	y	z	U(eq)
C(1)	1407(4)	2533(1)	10188(3)	30(1)
C(2)	2737(4)	2496(2)	10506(3)	31(1)
C(3)	2095(3)	1735(1)	9315(3)	23(1)
C(4)	-144(3)	394(1)	7105(3)	21(1)
C(5)	-1737(3)	-167(2)	6071(3)	27(1)
C(6)	-2356(3)	299(2)	6434(3)	26(1)

C(7)	-1462(3)	1166(1)	7703(3)	23(1)
C(8)	-2662(4)	1444(2)	7760(3)	29(1)
C(9)	-2611(4)	1953(2)	8413(3)	32(1)
C(10)	-1416(4)	2178(2)	8988(3)	29(1)
C(11)	-257(3)	1878(1)	8898(3)	24(1)
C(12)	-237(3)	1368(1)	8263(3)	22(1)
C(13)	4503(4)	1784(2)	10169(4)	33(1)
C(14)	4718(4)	1345(2)	11192(4)	34(1)
C(15)	4452(4)	1561(2)	12452(4)	38(1)
C(16)	4663(5)	1117(2)	13472(4)	47(1)
C(17)	603(3)	-531(1)	6312(3)	26(1)
C(18)	565(4)	-1035(2)	7164(3)	28(1)
C(19)	1527(4)	-1490(2)	6877(4)	37(1)
C(20)	1441(8)	-2018(2)	7623(5)	75(2)
C(21)	2803(6)	-146(2)	9942(4)	52(1)
C(22)	1323(4)	529(2)	10583(4)	40(1)
N(1)	1032(3)	2063(1)	9459(2)	24(1)
N(2)	3140(3)	2006(1)	9968(3)	26(1)
N(3)	-1366(3)	637(1)	7072(2)	22(1)
N(4)	-395(3)	-99(1)	6483(2)	23(1)
N(5)	2059(3)	370(1)	9560(3)	32(1)
Zr	1722(1)	894(1)	8115(1)	18(1)
I(1)	1561(1)	1657(1)	5882(1)	28(1)
I(2)	4001(1)	458(1)	7205(1)	39(1)
I(2A)	1862(5)	269(2)	10124(5)	39

U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

Table 3. Bond lengths [A] and angles [deg] for 3.

C(1)-C(2)	1.349(6)	C(3)-Zr	2.367(3)
C(1)-N(1)	1.380(4)	C(4)-N(4)	1.347(4)
C(2)-N(2)	1.382(4)	C(4)-N(3)	1.364(4)
C(3)-N(2)	1.348(4)	C(4)-Zr	2.362(3)
C(3)-N(1)	1.356(4)	C(5)-C(6)	1.351(5)
		C(5)-N(4)	1.381(4)
		C(6)-N(3)	1.386(4)

C(7)-C(12)	1.388(5)	C(8)-C(7)-N(3)	123.4(3)
C(7)-C(8)	1.394(5)	C(9)-C(8)-C(7)	117.3(3)
C(7)-N(3)	1.431(4)	C(10)-C(9)-C(8)	121.5(3)
C(8)-C(9)	1.389(5)	C(9)-C(10)-C(11)	118.1(3)
C(9)-C(10)	1.388(5)	C(12)-C(11)-C(10)	123.3(3)
C(10)-C(11)	1.391(5)	C(12)-C(11)-N(1)	113.4(3)
C(11)-C(12)	1.383(4)	C(10)-C(11)-N(1)	123.3(3)
C(11)-N(1)	1.430(4)	C(11)-C(12)-C(7)	115.9(3)
C(12)-Zr	2.310(3)	C(11)-C(12)-Zr	121.9(2)
C(13)-N(2)	1.467(5)	C(7)-C(12)-Zr	122.2(2)
C(13)-C(14)	1.509(6)	N(2)-C(13)-C(14)	112.5(3)
C(14)-C(15)	1.523(6)	C(13)-C(14)-C(15)	114.3(3)
C(15)-C(16)	1.515(6)	C(16)-C(15)-C(14)	113.9(4)
C(17)-N(4)	1.468(4)	N(4)-C(17)-C(18)	112.6(3)
C(17)-C(18)	1.507(5)	C(17)-C(18)-C(19)	110.6(3)
C(18)-C(19)	1.513(5)	C(20)-C(19)-C(18)	112.5(4)
C(19)-C(20)	1.491(7)	C(3)-N(1)-C(1)	111.3(3)
C(21)-N(5)	1.457(6)	C(3)-N(1)-C(11)	118.2(3)
C(22)-N(5)	1.479(5)	C(1)-N(1)-C(11)	130.4(3)
N(5)-Zr	1.986(3)	C(3)-N(2)-C(2)	110.9(3)
Zr-I(2A)	2.620(5)	C(3)-N(2)-C(13)	124.4(3)
Zr-I(2)	2.8431(4)	C(2)-N(2)-C(13)	124.5(3)
Zr-I(1)	3.0038(4)	C(4)-N(3)-C(6)	111.5(3)
		C(4)-N(3)-C(7)	118.6(3)
C(2)-C(1)-N(1)	106.2(3)	C(6)-N(3)-C(7)	129.9(3)
C(1)-C(2)-N(2)	107.1(3)	C(4)-N(4)-C(5)	111.5(3)
N(2)-C(3)-N(1)	104.5(3)	C(4)-N(4)-C(17)	125.1(3)
N(2)-C(3)-Zr	137.2(2)	C(5)-N(4)-C(17)	123.3(3)
N(1)-C(3)-Zr	118.2(2)	C(21)-N(5)-C(22)	107.2(3)
N(4)-C(4)-N(3)	104.0(3)	C(21)-N(5)-Zr	139.8(3)
N(4)-C(4)-Zr	137.9(2)	C(22)-N(5)-Zr	113.0(3)
N(3)-C(4)-Zr	118.1(2)	N(5)-Zr-C(12)	107.11(12)
C(6)-C(5)-N(4)	107.1(3)	N(5)-Zr-C(4)	95.28(12)
C(5)-C(6)-N(3)	105.9(3)	C(12)-Zr-C(4)	68.28(11)
C(12)-C(7)-C(8)	123.8(3)	N(5)-Zr-C(3)	94.98(12)
C(12)-C(7)-N(3)	112.8(3)	C(12)-Zr-C(3)	68.20(11)

C(4)-Zr-C(3)	136.40(11)	I(2A)-Zr-I(2)	98.15(11)
N(5)-Zr-I(2A)	8.30(14)	N(5)-Zr-I(1)	172.86(10)
C(12)-Zr-I(2A)	98.83(13)	C(12)-Zr-I(1)	79.88(7)
C(4)-Zr-I(2A)	92.82(12)	C(4)-Zr-I(1)	88.67(7)
C(3)-Zr-I(2A)	91.37(13)	C(3)-Zr-I(1)	86.18(8)
N(5)-Zr-I(2)	89.85(10)	I(2A)-Zr-I(1)	177.52(11)
C(12)-Zr-I(2)	162.66(7)	I(2)-Zr-I(1)	83.278(11)
C(4)-Zr-I(2)	107.42(8)		
C(3)-Zr-I(2)	114.86(8)		

Table 4. Anisotropic displacement parameters (A^2 x 10^3) for 3

The anisotropic displacement factor exponent takes the form:

-2 pi^2 [h^2 a*^2 U11 + ... + 2 h k a* b* U12]

Atom	U11	U22	U33	U23	U13	U12
C(1)	50(2)	18(1)	24(2)	-6(1)	7(1)	-2(1)
C(2)	47(2)	21(2)	25(2)	-6(1)	7(2)	-7(2)
C(3)	31(2)	21(1)	19(1)	-3(1)	4(1)	-5(1)
C(4)	27(1)	17(1)	19(1)	3(1)	5(1)	-4(1)
C(5)	28(2)	27(2)	27(2)	-2(1)	3(1)	-9(1)
C(6)	23(1)	28(2)	26(2)	2(1)	2(1)	-6(1)
C(7)	28(2)	19(1)	23(1)	6(1)	7(1)	0(1)
C(8)	28(2)	28(2)	34(2)	9(1)	8(1)	1(1)
C(9)	32(2)	31(2)	37(2)	11(1)	16(1)	8(1)
C(10)	42(2)	23(2)	25(2)	4(1)	16(1)	6(1)
C(11)	33(2)	21(1)	20(1)	1(1)	8(1)	0(1)
C(12)	29(2)	19(1)	18(1)	1(1)	6(1)	1(1)
C(13)	32(2)	34(2)	35(2)	-12(2)	10(1)	-10(2)
C(14)	29(2)	28(2)	44(2)	-12(2)	2(2)	-1(1)
C(15)	45(2)	32(2)	37(2)	-3(2)	6(2)	2(2)
C(16)	49(2)	43(2)	50(2)	8(2)	11(2)	-3(2)
C(17)	28(2)	23(2)	28(2)	-4(1)	8(1)	-5(1)
C(18)	34(2)	27(2)	24(2)	0(1)	6(1)	1(1)
C(19)	39(2)	40(2)	31(2)	-2(2)	2(2)	12(2)
C(20)	127(5)	41(3)	59(3)	15(2)	22(3)	40(3)
C(21)	80(3)	38(2)	39(2)	14(2)	11(2)	12(2)

C(22)	40(2)	47(2)	34(2)	17(2)	10(2)	-2(2)
N(1)	37(2)	16(1)	21(1)	-2(1)	7(1)	0(1)
N(2)	38(2)	21(1)	22(1)	-4(1)	7(1)	-7(1)
N(3)	24(1)	18(1)	23(1)	3(1)	3(1)	-4(1)
N(4)	29(1)	18(1)	23(1)	-2(1)	4(1)	-6(1)
N(5)	48(2)	36(2)	15(1)	8(1)	10(1)	-6(1)
Zr	24(1)	15(1)	17(1)	-1(1)	4(1)	-2(1)
I(1)	43(1)	20(1)	22(1)	3(1)	6(1)	-3(1)
I(2)	27(1)	49(1)	42(1)	-1(1)	10(1)	7(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (A^2 x 10^3) for 3

Atom	x	y	z	U(eq)
H(1A)	843	2824	10420	36
H(2A)	3293	2758	11006	37
H(5A)	-2146	-481	5618	33
H(6A)	-3281	379	6283	31
H(8A)	-3482	1291	7367	35
H(9A)	-3413	2152	8468	38
H(10A)	-1391	2528	9431	35
H(13A)	5129	2104	10382	39
H(13B)	4702	1611	9387	39
H(14A)	5647	1209	11275	41
H(14B)	4130	1016	10949	41
H(15A)	5045	1889	12700	46
H(15B)	3525	1699	12370	46
H(16A)	4479	1285	14253	57
H(16B)	5585	983	13573	57
H(16C)	4062	796	13248	57
H(17A)	1498	-357	6469	31
H(17B)	451	-665	5439	31
H(18A)	-346	-1194	7060	34
H(18B)	801	-911	8038	34
H(19A)	1339	-1586	5982	44

H(19B)	2443	-1338	7047	44
H(20A)	2087	-2297	7412	90
H(20B)	543	-2177	7438	90
H(20C)	1636	-1926	8510	90
H(21A)	3413	-72	10710	62
H(21B)	3311	-263	9287	62
H(21C)	2184	-450	10087	62
H(22A)	768	209	10770	48
H(22B)	758	860	10333	48
H(22C)	1955	625	11324	48

Table 6. Torsion angles [deg] for 3.

N(1)-C(1)-C(2)-N(2)	-0.2(4)	Zr-C(3)-N(1)-C(11)	1.2(4)
N(4)-C(5)-C(6)-N(3)	-0.6(4)	C(2)-C(1)-N(1)-C(3)	0.0(4)
C(12)-C(7)-C(8)-C(9)	0.3(5)	C(2)-C(1)-N(1)-C(11)	178.0(3)
N(3)-C(7)-C(8)-C(9)	179.5(3)	C(12)-C(11)-N(1)-C(3)	0.1(4)
C(7)-C(8)-C(9)-C(10)	0.0(5)	C(10)-C(11)-N(1)-C(3)	179.7(3)
C(8)-C(9)-C(10)-C(11)	-0.2(5)	C(12)-C(11)-N(1)-C(1)	-177.7(3)
C(9)-C(10)-C(11)-C(12)	0.2(5)	C(10)-C(11)-N(1)-C(1)	1.9(5)
C(9)-C(10)-C(11)-N(1)	-179.4(3)	N(1)-C(3)-N(2)-C(2)	-0.2(4)
C(10)-C(11)-C(12)-C(7)	0.1(5)	Zr-C(3)-N(2)-C(2)	-179.3(3)
N(1)-C(11)-C(12)-C(7)	179.7(3)	N(1)-C(3)-N(2)-C(13)	174.8(3)
C(10)-C(11)-C(12)-Zr	179.0(2)	Zr-C(3)-N(2)-C(13)	-4.2(5)
N(1)-C(11)-C(12)-Zr	-1.4(4)	C(1)-C(2)-N(2)-C(3)	0.3(4)
C(8)-C(7)-C(12)-C(11)	-0.3(5)	C(1)-C(2)-N(2)-C(13)	-174.8(3)
N(3)-C(7)-C(12)-C(11)	-179.6(3)	C(14)-C(13)-N(2)-C(3)	-79.9(4)
C(8)-C(7)-C(12)-Zr	-179.2(2)	C(14)-C(13)-N(2)-C(2)	94.5(4)
N(3)-C(7)-C(12)-Zr	1.5(4)	N(4)-C(4)-N(3)-C(6)	-0.2(3)
N(2)-C(13)-C(14)-C(15)	-59.2(4)	Zr-C(4)-N(3)-C(6)	179.0(2)
C(13)-C(14)-C(15)-C(16)	179.7(3)	N(4)-C(4)-N(3)-C(7)	177.6(3)
N(4)-C(17)-C(18)-C(19)	175.1(3)	Zr-C(4)-N(3)-C(7)	-3.2(3)
C(17)-C(18)-C(19)-C(20)	-175.1(4)	C(5)-C(6)-N(3)-C(4)	0.5(4)
N(2)-C(3)-N(1)-C(1)	0.1(4)	C(5)-C(6)-N(3)-C(7)	-177.0(3)
Zr-C(3)-N(1)-C(1)	179.4(2)	C(12)-C(7)-N(3)-C(4)	1.2(4)
N(2)-C(3)-N(1)-C(11)	-178.1(3)	C(8)-C(7)-N(3)-C(4)	-178.1(3)
		C(12)-C(7)-N(3)-C(6)	178.5(3)

C(8)-C(7)-N(3)-C(6)	-0.8(5)	N(3)-C(4)-Zr-C(12)	2.8(2)
N(3)-C(4)-N(4)-C(5)	-0.2(3)	N(4)-C(4)-Zr-C(3)	-174.9(3)
Zr-C(4)-N(4)-C(5)	-179.2(3)	N(3)-C(4)-Zr-C(3)	6.2(3)
N(3)-C(4)-N(4)-C(17)	-176.9(3)	N(4)-C(4)-Zr-I(2A)	-80.0(3)
Zr-C(4)-N(4)-C(17)	4.1(5)	N(3)-C(4)-Zr-I(2A)	101.2(2)
C(6)-C(5)-N(4)-C(4)	0.5(4)	N(4)-C(4)-Zr-I(2)	19.5(3)
C(6)-C(5)-N(4)-C(17)	177.3(3)	N(3)-C(4)-Zr-I(2)	-159.4(2)
C(18)-C(17)-N(4)-C(4)	102.7(4)	N(4)-C(4)-Zr-I(1)	102.0(3)
C(18)-C(17)-N(4)-C(5)	-73.7(4)	N(3)-C(4)-Zr-I(1)	-76.8(2)
C(21)-N(5)-Zr-C(12)	158.5(5)	N(2)-C(3)-Zr-N(5)	71.2(3)
C(22)-N(5)-Zr-C(12)	-19.8(3)	N(1)-C(3)-Zr-N(5)	-107.8(3)
C(21)-N(5)-Zr-C(4)	89.6(5)	N(2)-C(3)-Zr-C(12)	177.6(4)
C(22)-N(5)-Zr-C(4)	-88.7(3)	N(1)-C(3)-Zr-C(12)	-1.3(2)
C(21)-N(5)-Zr-C(3)	-132.8(5)	N(2)-C(3)-Zr-C(4)	174.2(3)
C(22)-N(5)-Zr-C(3)	48.9(3)	N(1)-C(3)-Zr-C(4)	-4.8(3)
C(21)-N(5)-Zr-I(2A)	162.7(13)	N(2)-C(3)-Zr-I(2A)	78.7(3)
C(22)-N(5)-Zr-I(2A)	-15.6(9)	N(1)-C(3)-Zr-I(2A)	-100.3(3)
C(21)-N(5)-Zr-I(2)	-17.9(5)	N(2)-C(3)-Zr-I(2	-21.0(4)
C(22)-N(5)-Zr-I(2)	163.9(3)	N(1)-C(3)-Zr-I(2)	160.1(2)
C(21)-N(5)-Zr-I(1)	-33.7(11)	N(2)-C(3)-Zr-I(1)	-101.8(3)
C(22)-N(5)-Zr-I(1)	148.0(6)	N(1)-C(3)-Zr-I(1)	79.3(2)
C(11)-C(12)-Zr-N(5)	90.0(3)		
C(7)-C(12)-Zr-N(5)	-91.2(3)		
C(11)-C(12)-Zr-C(4)	178.9(3)		
C(7)-C(12)-Zr-C(4)	-2.3(2)		
C(11)-C(12)-Zr-C(3)	1.5(2)		
C(7)-C(12)-Zr-C(3)	-179.7(3)		
C(11)-C(12)-Zr-I(2A)	89.4(3)		
C(7)-C(12)-Zr-I(2A)	-91.8(3)		
C(11)-C(12)-Zr-I(2)	-102.3(3)		
C(7)-C(12)-Zr-I(2)	76.5(4)		
C(11)-C(12)-Zr-I(1)	-88.5(2)		
C(7)-C(12)-Zr-I(1)	90.3(2)		
N(4)-C(4)-Zr-N(5)	-72.0(3)		
N(3)-C(4)-Zr-N(5)	109.1(2)		
N(4)-C(4)-Zr-C(12)	-178.3(4)		