Supporting Information for:

Dimerisation of β-diketiminato Calcium Complexes through Dihapto-Acetylide Ligation

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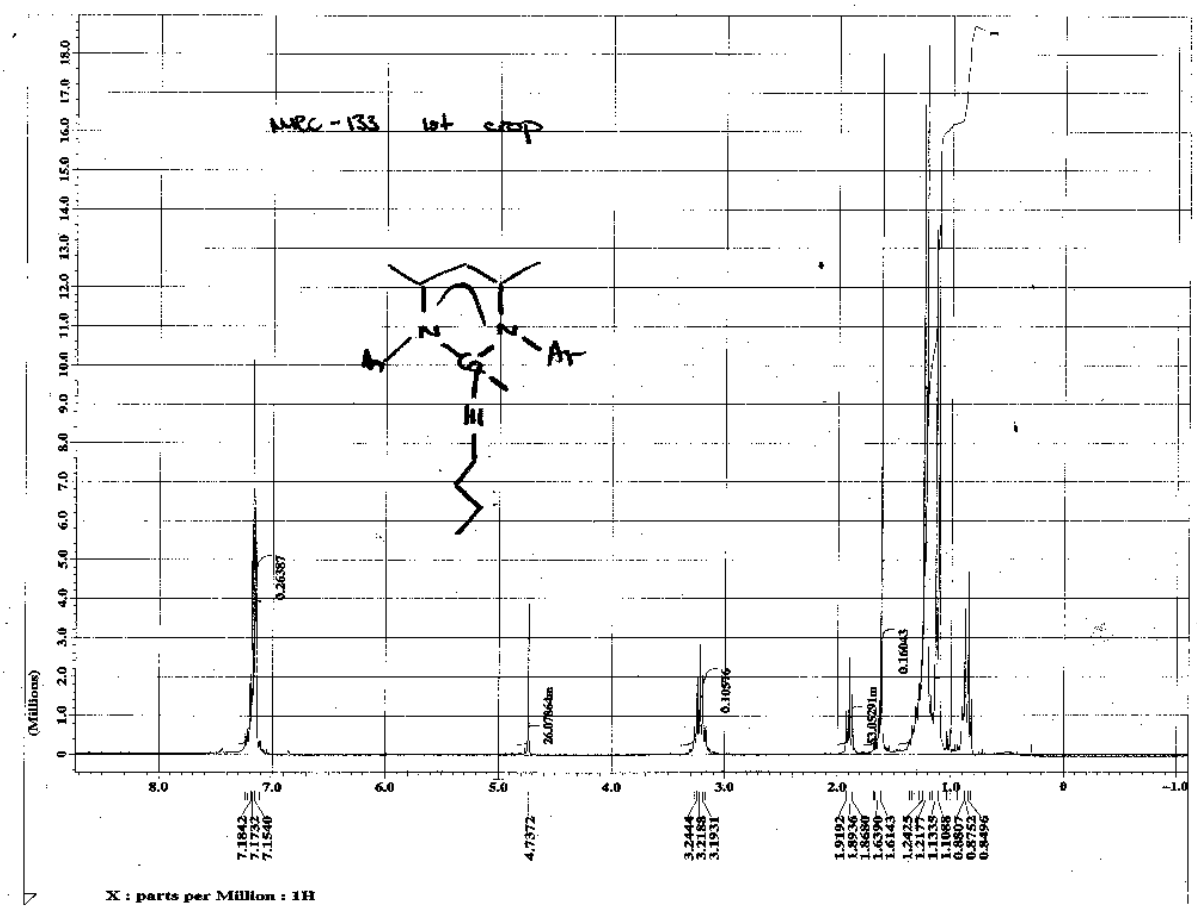
^{1}H NMR spectra of **2a-c**

Details of crystal data and structure refinement for compound **2b**

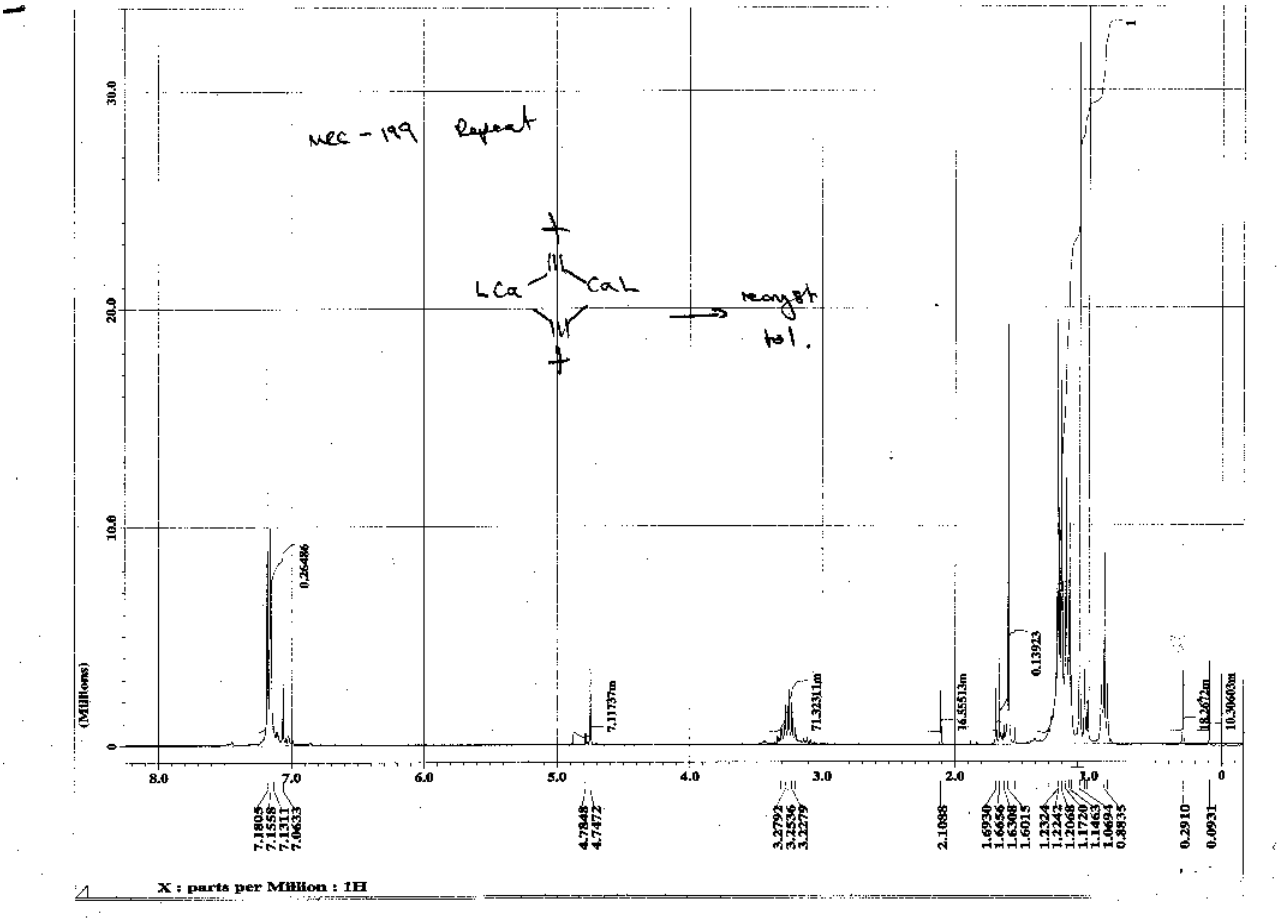
Details of crystal data and structure refinement for compound **2c**

Details of crystal data and structure refinement for compound **2d**

^{1}H NMR spectrum of 2a



^{1}H NMR spectrum of 2b



^{1}H NMR specrum of 2c

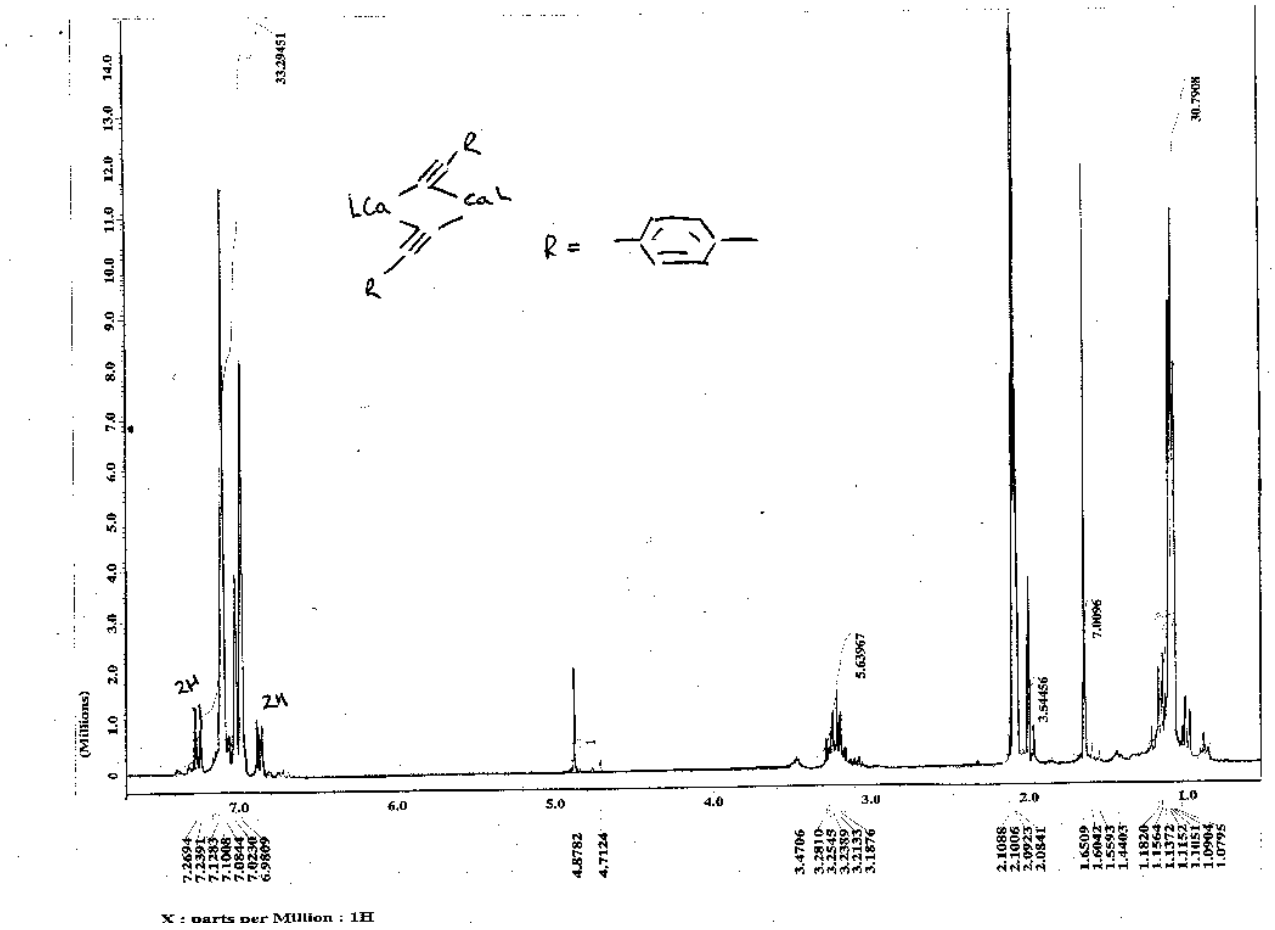


Table 1. Crystal data and structure refinement for [{Ca(ArNCMeCHCMeNAr)(CCtBu)}2].. 2(toluene)

Identification code	Compound 2a	
Empirical formula	C70 H100 Ca2 N4 . 2(C7 H8)	
Formula weight	1261.97	
Temperature	173(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	$P\bar{1}$ (No.2)	
Unit cell dimensions	a = 11.9217(2) Å	α= 89.254(1)°.
	b = 13.2905(2) Å	β= 72.703(1)°.
	c = 13.4317(2) Å	γ = 71.976(1)°.
Volume	1924.91(5) $Å^3$	
Z	1	
Density (calculated)	1.09 Mg/m^3	
Absorption coefficient	0.19 mm^{-1}	
F(000)	688	
Crystal size	0.40 x 0.40 x 0.35 mm^3	
Theta range for data collection	3.73 to 27.49°.	
Index ranges	-15<=h<=15, -17<=k<=17, -17<=l<=17	
Reflections collected	30434	
Independent reflections	8687 [R(int) = 0.039]	
Reflections with I>2sigma(I)	7795	
Completeness to theta = 27.49°	98.1 %	
Tmax. and Tmin.	0.953 and 0.900	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	8687 / 0 / 408	
Goodness-of-fit on F^2	1.032	
Final R indices [I>2sigma(I)]	R1 = 0.045, wR2 = 0.113	
R indices (all data)	R1 = 0.051, wR2 = 0.117	
Largest diff. peak and hole	0.52 and -0.26 $e.Å^{-3}$	

Data collection KappaCCD , Program package WinGX , Abs correction MULTISCAN

Refinement using SHELXL-97 , Drawing using ORTEP-3 for Windows (20% ellipsoids)

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for apr1104. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Ca	6482(1)	4245(1)	4160(1)	24(1)
N(1)	8020(1)	2593(1)	3941(1)	23(1)
N(2)	7795(1)	4487(1)	2572(1)	23(1)
C(1)	9201(1)	2456(1)	3434(1)	23(1)
C(2)	9638(1)	3173(1)	2761(1)	25(1)
C(3)	9017(1)	4038(1)	2297(1)	23(1)
C(4)	10207(1)	1467(1)	3536(1)	32(1)
C(5)	9846(1)	4436(1)	1400(1)	35(1)
C(6)	7696(1)	1737(1)	4496(1)	25(1)
C(7)	7762(1)	1593(1)	5520(1)	29(1)
C(8)	7428(2)	752(1)	6023(1)	37(1)
C(9)	7020(2)	88(1)	5547(1)	42(1)
C(10)	6917(2)	257(1)	4559(1)	40(1)
C(11)	7245(1)	1078(1)	4016(1)	32(1)
C(12)	8127(2)	2336(1)	6116(1)	37(1)
C(13)	6992(2)	2984(1)	7009(1)	47(1)
C(14)	9189(2)	1743(2)	6546(2)	55(1)
C(15)	7153(2)	1232(1)	2918(1)	41(1)
C(16)	8338(2)	563(3)	2114(2)	83(1)
C(17)	6043(3)	996(2)	2760(2)	76(1)
C(18)	7271(1)	5218(1)	1908(1)	26(1)
C(19)	6885(1)	6320(1)	2167(1)	32(1)
C(20)	6318(2)	6998(1)	1527(1)	44(1)
C(21)	6107(2)	6615(1)	679(2)	47(1)
C(22)	6482(2)	5530(1)	433(1)	41(1)
C(23)	7074(1)	4815(1)	1031(1)	31(1)
C(24)	7083(1)	6796(1)	3099(1)	36(1)
C(25)	5889(2)	7565(2)	3821(2)	58(1)
C(26)	8057(2)	7361(2)	2746(2)	64(1)
C(27)	7452(2)	3631(1)	736(1)	35(1)
C(28)	8217(2)	3324(2)	-416(2)	60(1)

C(29)	6307(2)	3272(2)	1003(2)	56(1)
C(30)	4297(1)	4468(1)	4246(1)	32(1)
C(31)	3449(1)	4307(1)	4019(1)	31(1)
C(32)	2475(1)	4059(1)	3687(1)	38(1)
C(33)	2995(2)	3721(3)	2512(2)	80(1)
C(34)	1333(2)	5041(2)	3860(2)	63(1)
C(35)	2076(3)	3205(2)	4322(2)	80(1)
C(36)	11919(2)	908(2)	530(1)	59(1)
C(37)	12185(3)	-176(2)	652(2)	73(1)
C(38)	13169(3)	-699(2)	990(2)	83(1)
C(39)	13924(3)	-166(2)	1240(2)	90(1)
C(40)	13635(2)	891(2)	1130(2)	82(1)
C(41)	12672(2)	1420(2)	781(2)	58(1)
C(42)	10860(3)	1486(2)	171(2)	84(1)

Table 3. Bond lengths [Å] and angles [°] for apr1104.

Ca-N(2)	2.3265(11)
Ca-N(1)	2.3391(10)
Ca-C(30)	2.4960(15)
Ca-C(30)’	2.5099(14)
Ca-C(31)’	3.1668(14)
N(1)-C(1)	1.3246(16)
N(1)-C(6)	1.4418(16)
N(2)-C(3)	1.3283(16)
N(2)-C(18)	1.4368(15)
C(1)-C(2)	1.4140(18)
C(1)-C(4)	1.5171(17)
C(2)-C(3)	1.4104(17)
C(3)-C(5)	1.5185(18)
C(6)-C(7)	1.4073(19)
C(6)-C(11)	1.4086(19)
C(7)-C(8)	1.3997(19)
C(7)-C(12)	1.517(2)
C(8)-C(9)	1.374(2)
C(9)-C(10)	1.378(2)
C(10)-C(11)	1.396(2)
C(11)-C(15)	1.518(2)
C(12)-C(13)	1.534(2)
C(12)-C(14)	1.536(2)
C(15)-C(16)	1.514(3)
C(15)-C(17)	1.523(3)
C(18)-C(19)	1.4074(19)
C(18)-C(23)	1.4099(19)
C(19)-C(20)	1.399(2)
C(19)-C(24)	1.520(2)
C(20)-C(21)	1.373(3)
C(21)-C(22)	1.383(3)
C(22)-C(23)	1.395(2)
C(23)-C(27)	1.520(2)
C(24)-C(25)	1.521(2)

C(24)-C(26)	1.531(2)
C(27)-C(29)	1.525(2)
C(27)-C(28)	1.529(2)
C(30)-C(31)	1.216(2)
C(31)-C(32)	1.485(2)
C(32)-C(35)	1.519(3)
C(32)-C(34)	1.527(3)
C(32)-C(33)	1.530(3)
C(36)-C(41)	1.394(3)
C(36)-C(37)	1.395(3)
C(36)-C(42)	1.472(3)
C(37)-C(38)	1.367(4)
C(38)-C(39)	1.416(4)
C(39)-C(40)	1.357(4)
C(40)-C(41)	1.358(3)

N(2)-Ca-N(1)	82.89(4)
N(2)-Ca-C(30)	117.10(4)
N(1)-Ca-C(30)	123.66(4)
N(2)-Ca-C(30)’	123.06(4)
N(1)-Ca-C(30)’	124.30(4)
C(30)-Ca-C(30)’	89.73(5)
N(2)-Ca-C(31)’	109.71(4)
N(1)-Ca-C(31)’	109.81(4)
C(30)-Ca-C(31)’	110.63(4)
C(1)-N(1)-C(6)	118.75(10)
C(1)-N(1)-Ca	122.16(8)
C(6)-N(1)-Ca	118.52(8)
C(3)-N(2)-C(18)	118.88(10)
C(3)-N(2)-Ca	122.35(8)
C(18)-N(2)-Ca	118.56(8)
N(1)-C(1)-C(2)	124.84(11)
N(1)-C(1)-C(4)	120.54(11)
C(2)-C(1)-C(4)	114.62(11)
C(3)-C(2)-C(1)	131.54(12)
N(2)-C(3)-C(2)	124.76(11)

N(2)-C(3)-C(5)	119.83(11)
C(2)-C(3)-C(5)	115.39(11)
C(7)-C(6)-C(11)	120.27(12)
C(7)-C(6)-N(1)	121.05(12)
C(11)-C(6)-N(1)	118.59(12)
C(8)-C(7)-C(6)	118.49(13)
C(8)-C(7)-C(12)	118.63(13)
C(6)-C(7)-C(12)	122.84(12)
C(9)-C(8)-C(7)	121.50(14)
C(8)-C(9)-C(10)	119.65(14)
C(9)-C(10)-C(11)	121.38(14)
C(10)-C(11)-C(6)	118.64(14)
C(10)-C(11)-C(15)	120.38(13)
C(6)-C(11)-C(15)	120.96(12)
C(7)-C(12)-C(13)	109.63(13)
C(7)-C(12)-C(14)	112.51(14)
C(13)-C(12)-C(14)	110.36(14)
C(16)-C(15)-C(11)	110.49(16)
C(16)-C(15)-C(17)	110.09(18)
C(11)-C(15)-C(17)	113.33(15)
C(19)-C(18)-C(23)	120.42(12)
C(19)-C(18)-N(2)	120.40(12)
C(23)-C(18)-N(2)	119.05(12)
C(20)-C(19)-C(18)	118.26(14)
C(20)-C(19)-C(24)	119.20(13)
C(18)-C(19)-C(24)	122.53(12)
C(21)-C(20)-C(19)	121.81(15)
C(20)-C(21)-C(22)	119.52(14)
C(21)-C(22)-C(23)	121.26(15)
C(22)-C(23)-C(18)	118.70(14)
C(22)-C(23)-C(27)	119.15(13)
C(18)-C(23)-C(27)	122.12(12)
C(19)-C(24)-C(25)	112.46(14)
C(19)-C(24)-C(26)	111.28(14)
C(25)-C(24)-C(26)	109.27(15)
C(23)-C(27)-C(29)	110.06(14)

C(23)-C(27)-C(28)	112.37(14)
C(29)-C(27)-C(28)	110.97(16)
C(31)-C(30)-Ca	157.99(12)
C(31)-C(30)-Ca’	111.61(11)
Ca-C(30)-Ca’	90.27(5)
C(30)-C(31)-C(32)	176.23(15)
C(30)-C(31)-Ca’	47.46(8)
C(32)-C(31)-Ca’	136.29(10)
C(31)-C(32)-C(35)	110.07(14)
C(31)-C(32)-C(34)	110.85(14)
C(35)-C(32)-C(34)	107.99(17)
C(31)-C(32)-C(33)	108.03(14)
C(35)-C(32)-C(33)	112.38(19)
C(34)-C(32)-C(33)	107.51(18)
C(41)-C(36)-C(37)	117.7(2)
C(41)-C(36)-C(42)	121.8(2)
C(37)-C(36)-C(42)	120.5(3)
C(38)-C(37)-C(36)	119.7(3)
C(37)-C(38)-C(39)	121.8(2)
C(40)-C(39)-C(38)	117.3(3)
C(39)-C(40)-C(41)	121.6(3)
C(40)-C(41)-C(36)	121.8(2)

Symmetry transformations used to generate equivalent atoms:

‘ -x+1,-y+1,-z+1

Least-squares planes (x,y,z in crystal coordinates) and deviations from them
(* indicates atom used to define plane)

0.6265 (0.0042) x + 10.4083 (0.0034) y - 8.2141 (0.0043) z = 1.4104 (0.0057)

*	-0.0035	(0.0002)	Ca
*	0.0211	(0.0012)	C30
*	-0.0129	(0.0008)	C31
*	0.0035	(0.0002)	Ca’
*	-0.0211	(0.0012)	C30’
*	0.0129	(0.0008)	C31’

Rms deviation of fitted atoms = 0.0144

Table 1. Crystal data and structure refinement for [{Ca(ArNCMeCHCMeNAr)(CC-p-tol)}2].

Identification code	Compound 2c	
Empirical formula	C76 H96 Ca2 N4	
Formula weight	1145.73	
Temperature	173(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	$P2_1/n$ (No. 14)	
Unit cell dimensions	a = 14.9183(3) Å	α= 90°.
	b = 13.1452(2) Å	β= 103.916(1)°.
	c = 18.3729(4) Å	γ = 90°.
Volume	3497.25(12) $Å^3$	
Z	2	
Density (calculated)	1.09 Mg/m^3	
Absorption coefficient	0.205 mm^{-1}	
F(000)	1240	
Crystal size	0.3 x 0.2 x 0.2 mm^3	
Theta range for data collection	3.72 to 25.06°.	
Index ranges	-17<=h<=17, -15<=k<=15, -21<=l<=21	
Reflections collected	43924	
Independent reflections	6160 [R(int) = 0.045]	
Reflections with I>2sigma(I)	5147	
Completeness to theta = 25.06°	99.3 %	
Tmax. and Tmin.	0.962 and 0.857	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	6160 / 0 / 373	
Goodness-of-fit on F^2	1.060	
Final R indices [I>2sigma(I)]	R1 = 0.041, wR2 = 0.101	
R indices (all data)	R1 = 0.053, wR2 = 0.109	
Largest diff. peak and hole	0.21 and -0.22 $e.Å^{-3}$	

Data collection KappaCCD , Program package WinGX , Abs correction MULTISCAN

Refinement using SHELXL-97 , Drawing using ORTEP-3 for Windows

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for sep1804. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Ca	6124(1)	5139(1)	4822(1)	32(1)
N(1)	7330(1)	4359(1)	4443(1)	29(1)
N(2)	7069(1)	6533(1)	4815(1)	30(1)
C(1)	7975(1)	4780(1)	4156(1)	33(1)
C(2)	8105(1)	5836(1)	4116(1)	35(1)
C(3)	7742(1)	6638(1)	4463(1)	33(1)
C(4)	8654(2)	4123(2)	3870(1)	50(1)
C(5)	8181(1)	7672(2)	4426(1)	48(1)
C(6)	7315(1)	3280(1)	4565(1)	32(1)
C(7)	7813(1)	2879(1)	5256(1)	37(1)
C(8)	7742(2)	1839(2)	5385(1)	48(1)
C(9)	7203(2)	1214(2)	4857(1)	57(1)
C(10)	6706(2)	1617(2)	4190(1)	54(1)
C(11)	6738(1)	2653(1)	4032(1)	41(1)
C(12)	8410(1)	3547(2)	5854(1)	45(1)
C(13)	8098(2)	3499(2)	6586(1)	60(1)
C(14)	9432(2)	3264(2)	5996(1)	72(1)
C(15)	6151(1)	3068(2)	3304(1)	49(1)
C(16)	6535(2)	2802(3)	2631(1)	86(1)
C(17)	5141(2)	2719(2)	3160(1)	63(1)
C(18)	6869(1)	7368(1)	5255(1)	31(1)
C(19)	7310(1)	7403(1)	6023(1)	37(1)
C(20)	7143(2)	8233(2)	6444(1)	45(1)
C(21)	6535(2)	8989(2)	6124(1)	47(1)
C(22)	6057(1)	8910(2)	5392(1)	47(1)
C(23)	6202(1)	8102(1)	4937(1)	38(1)
C(24)	7939(2)	6545(2)	6397(1)	49(1)
C(25)	7773(2)	6261(2)	7155(1)	65(1)
C(26)	8949(2)	6774(3)	6466(2)	90(1)
C(27)	5625(2)	8026(2)	4140(1)	50(1)
C(28)	5772(2)	8931(2)	3653(1)	76(1)

C(29)	4599(2)	7939(2)	4121(2)	73(1)
C(30)	5524(1)	4839(2)	5958(1)	39(1)
C(31)	5309(1)	4687(1)	6550(1)	37(1)
C(32)	5085(1)	4514(1)	7265(1)	37(1)
C(33)	4214(1)	4148(2)	7295(1)	45(1)
C(34)	4008(2)	3977(2)	7979(1)	50(1)
C(35)	4649(2)	4159(2)	8650(1)	53(1)
C(36)	5508(2)	4534(2)	8615(1)	57(1)
C(37)	5724(1)	4711(2)	7935(1)	47(1)
C(38)	4419(2)	3933(2)	9390(1)	83(1)

Table 3. Bond lengths [Å] and angles [°] for sep1804.

Ca-N(2)	2.3131(14)
Ca-N(1)	2.3190(14)
Ca-C(30)	2.492(2)
Ca-C(30)’	2.530(2)
Ca-C(31)’	2.895(2)
N(1)-C(1)	1.324(2)
N(1)-C(6)	1.438(2)
N(2)-C(3)	1.327(2)
N(2)-C(18)	1.436(2)
C(1)-C(2)	1.406(3)
C(1)-C(4)	1.518(2)
C(2)-C(3)	1.406(2)
C(3)-C(5)	1.517(2)
C(6)-C(11)	1.405(3)
C(6)-C(7)	1.409(3)
C(7)-C(8)	1.396(3)
C(7)-C(12)	1.517(3)
C(8)-C(9)	1.373(3)
C(9)-C(10)	1.377(3)
C(10)-C(11)	1.396(3)
C(11)-C(15)	1.512(3)
C(12)-C(13)	1.527(3)
C(12)-C(14)	1.530(3)
C(15)-C(16)	1.524(3)
C(15)-C(17)	1.535(3)
C(18)-C(19)	1.407(2)
C(18)-C(23)	1.409(2)
C(19)-C(20)	1.394(3)
C(19)-C(24)	1.521(3)
C(20)-C(21)	1.379(3)
C(21)-C(22)	1.367(3)
C(22)-C(23)	1.399(3)
C(23)-C(27)	1.514(3)
C(24)-C(26)	1.512(4)

C(24)-C(25)	1.519(3)
C(27)-C(29)	1.526(3)
C(27)-C(28)	1.535(3)
C(30)-C(31)	1.221(2)
C(31)-C(32)	1.449(2)
C(32)-C(37)	1.388(3)
C(32)-C(33)	1.400(3)
C(33)-C(34)	1.383(3)
C(34)-C(35)	1.387(3)
C(35)-C(36)	1.388(3)
C(35)-C(38)	1.510(3)
C(36)-C(37)	1.383(3)

N(2)-Ca-N(1)	80.24(5)
N(2)-Ca-C(30)	118.16(6)
N(1)-Ca-C(30)	130.00(6)
N(2)-Ca-C(30)’	119.88(6)
N(1)-Ca-C(30)’	123.54(5)
C(30)-Ca-C(30)’	88.91(6)
N(2)-Ca-C(31)’	104.82(5)
N(1)-Ca-C(31)’	103.70(5)
C(30)-Ca-C(31)’	113.77(6)
C(1)-N(1)-C(6)	120.92(14)
C(1)-N(1)-Ca	128.73(11)
C(6)-N(1)-Ca	110.36(10)
C(3)-N(2)-C(18)	118.58(14)
C(3)-N(2)-Ca	128.28(11)
C(18)-N(2)-Ca	113.14(10)
N(1)-C(1)-C(2)	123.82(15)
N(1)-C(1)-C(4)	120.62(16)
C(2)-C(1)-C(4)	115.53(15)
C(3)-C(2)-C(1)	129.99(16)
N(2)-C(3)-C(2)	124.21(15)
N(2)-C(3)-C(5)	120.08(15)
C(2)-C(3)-C(5)	115.71(15)
C(11)-C(6)-C(7)	120.70(16)

C(11)-C(6)-N(1)	119.97(16)
C(7)-C(6)-N(1)	119.01(15)
C(8)-C(7)-C(6)	118.30(18)
C(8)-C(7)-C(12)	119.93(17)
C(6)-C(7)-C(12)	121.77(16)
C(9)-C(8)-C(7)	121.4(2)
C(8)-C(9)-C(10)	119.7(2)
C(9)-C(10)-C(11)	121.6(2)
C(10)-C(11)-C(6)	118.19(19)
C(10)-C(11)-C(15)	119.64(18)
C(6)-C(11)-C(15)	122.18(17)
C(7)-C(12)-C(13)	111.35(17)
C(7)-C(12)-C(14)	111.73(18)
C(13)-C(12)-C(14)	110.04(18)
C(11)-C(15)-C(16)	112.61(19)
C(11)-C(15)-C(17)	112.36(18)
C(16)-C(15)-C(17)	109.63(19)
C(19)-C(18)-C(23)	120.39(16)
C(19)-C(18)-N(2)	118.62(15)
C(23)-C(18)-N(2)	120.87(15)
C(20)-C(19)-C(18)	118.65(17)
C(20)-C(19)-C(24)	120.15(17)
C(18)-C(19)-C(24)	121.18(16)
C(21)-C(20)-C(19)	121.01(18)
C(22)-C(21)-C(20)	119.89(18)
C(21)-C(22)-C(23)	121.76(18)
C(22)-C(23)-C(18)	117.93(17)
C(22)-C(23)-C(27)	119.65(17)
C(18)-C(23)-C(27)	122.40(16)
C(26)-C(24)-C(25)	110.53(19)
C(26)-C(24)-C(19)	112.63(18)
C(25)-C(24)-C(19)	112.23(18)
C(23)-C(27)-C(29)	111.11(19)
C(23)-C(27)-C(28)	112.65(18)
C(29)-C(27)-C(28)	109.12(19)
C(31)-C(30)-Ca	174.37(15)

C(31)-C(30)-Ca’	94.53(13)
Ca-C(30)-Ca’	91.09(6)
C(30)-C(31)-C(32)	178.06(19)
C(30)-C(31)-Ca’	60.61(11)
C(32)-C(31)-Ca’	121.28(12)
C(37)-C(32)-C(33)	118.36(17)
C(37)-C(32)-C(31)	121.14(16)
C(33)-C(32)-C(31)	120.50(17)
C(34)-C(33)-C(32)	120.16(19)
C(33)-C(34)-C(35)	121.59(19)
C(34)-C(35)-C(36)	117.88(18)
C(34)-C(35)-C(38)	120.6(2)
C(36)-C(35)-C(38)	121.5(2)
C(37)-C(36)-C(35)	121.2(2)
C(36)-C(37)-C(32)	120.76(18)

Symmetry transformations used to generate equivalent atoms: ‘ -x+1,-y+1,-z+1

Least-squares planes (x,y,z in crystal coordinates) and deviations from them
(* indicates atom used to define plane)

9.2161 (0.0415) x - 0.7082 (0.0183) y + 11.2609 (0.0501) z = 11.6910 (0.0207)

```
*    0.0000 (0.0000)  C1
*    0.0000 (0.0000)  C2
*    0.0000 (0.0000)  C3
```

Rms deviation of fitted atoms = 0.0000

7.0917 (0.0154) x - 1.4079 (0.0079) y + 13.4726 (0.0140) z = 10.5752 (0.0084)

Angle to previous plane (with approximate esd) = 10.10 (0.39)

```
*   -0.0055 (0.0007)  N1
*    0.0055 (0.0007)  N2
*    0.0064 (0.0009)  C1
*   -0.0064 (0.0009)  C3
    -0.1042 (0.0026)  C2
    -0.4591 (0.0028)  Ca
```

Rms deviation of fitted atoms = 0.0060

3.5348 (0.0084) x - 2.3174 (0.0082) y + 15.9912 (0.0053) z = 8.6851 (0.0060)

Angle to previous plane (with approximate esd) = 15.03 (0.11)

```
*    0.0000 (0.0000)  Ca
*    0.0000 (0.0000)  N1
*    0.0000 (0.0000)  N2
```

Rms deviation of fitted atoms = 0.0000

- 1.1627 (0.0041) x + 12.9784 (0.0018) y + 2.8121 (0.0182) z = 7.3139 (0.0097)

Angle to previous plane (with approximate esd) = 86.54 (0.07)

```
*    0.0000 (0.0000)  Ca
*    0.0000 (0.0000)  C30
*    0.0000 (0.0000)  Ca_$1
*    0.0000 (0.0000)  C30_$1
    -0.0070 (0.0035)  C31
    -0.0040 (0.0046)  C32
```

Rms deviation of fitted atoms = 0.0000

- 5.2247 (0.0128) x + 12.3126 (0.0042) y + 1.5143 (0.0163) z = 4.0074 (0.0142)

Angle to previous plane (with approximate esd) = 17.86 (0.04)

```
*   -0.0066 (0.0014)  C32
*    0.0027 (0.0015)  C33
*    0.0037 (0.0016)  C34
*   -0.0062 (0.0016)  C35
*    0.0023 (0.0017)  C36
*    0.0041 (0.0016)  C37
```

Rms deviation of fitted atoms = 0.0045

Table 1. Crystal data and structure refinement for [{(ArNCMeCHCMeNAr)Ca(CCPh)}2].

Identification code	Compound 2d	
Empirical formula	C74 H92 Ca2 N4	
Formula weight	1117.68	
Temperature	173(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	$P2_1/n$ (No.14)	
Unit cell dimensions	a = 15.0572(3) Å	α= 90°.
	b = 13.0076(3) Å	β= 100.482(1)°.
	c = 17.5209(4) Å	γ = 90°.
Volume	3374.34(13) $Å^3$	
Z	2	
Density (calculated)	1.10 Mg/m^3	
Absorption coefficient	0.21 mm^{-1}	
F(000)	1208	
Crystal size	0.4 x 0.4 x 0.3 mm^3	
Theta range for data collection	3.88 to 25.03°.	
Index ranges	-17<=h<=17, -15<=k<=14, -20<=l<=20	
Reflections collected	23366	
Independent reflections	5859 [R(int) = 0.033]	
Reflections with I>2sigma(I)	5121	
Completeness to theta = 25.03°	98.4 %	
Tmax. and Tmin.	0.965 and 0.887	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	5859 / 0 / 363	
Goodness-of-fit on F^2	1.030	
Final R indices [I>2sigma(I)]	R1 = 0.040, wR2 = 0.098	
R indices (all data)	R1 = 0.048, wR2 = 0.103	
Largest diff. peak and hole	0.26 and -0.21 e.$Å^{-3}$	

Data collection KappaCCD , Program package WinGX , Abs correction MULTISCAN

Refinement using SHELXL-97 , Drawing using ORTEP-3 for Windows(20% ellipsoids)

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for may1704. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Ca	1117(1)	109(1)	-202(1)	29(1)
N(1)	2036(1)	1531(1)	-211(1)	27(1)
N(2)	2325(1)	-660(1)	-632(1)	27(1)
C(1)	2721(1)	1650(1)	-583(1)	30(1)
C(2)	3091(1)	853(1)	-972(1)	33(1)
C(3)	2963(1)	-212(1)	-948(1)	30(1)
C(4)	3170(1)	2695(1)	-593(1)	43(1)
C(5)	3639(1)	-857(1)	-1285(1)	47(1)
C(6)	1834(1)	2372(1)	260(1)	28(1)
C(7)	1170(1)	3097(1)	-40(1)	33(1)
C(8)	1042(1)	3934(1)	424(1)	42(1)
C(9)	1537(1)	4049(1)	1162(1)	43(1)
C(10)	2144(1)	3296(1)	1469(1)	40(1)
C(11)	2295(1)	2438(1)	1034(1)	33(1)
C(12)	593(1)	2991(1)	-844(1)	39(1)
C(13)	-415(1)	2976(2)	-804(1)	56(1)
C(14)	769(2)	3848(2)	-1393(1)	73(1)
C(15)	2925(1)	1588(2)	1398(1)	46(1)
C(16)	3910(2)	1879(3)	1517(2)	100(1)
C(17)	2683(2)	1222(2)	2157(1)	67(1)
C(18)	2334(1)	-1753(1)	-525(1)	30(1)
C(19)	1726(1)	-2370(1)	-1035(1)	36(1)
C(20)	1729(1)	-3427(1)	-903(1)	50(1)
C(21)	2289(1)	-3864(2)	-282(1)	55(1)
C(22)	2856(1)	-3247(2)	234(1)	50(1)
C(23)	2893(1)	-2186(1)	124(1)	38(1)
C(24)	1088(1)	-1909(1)	-1719(1)	42(1)
C(25)	128(2)	-2350(2)	-1827(1)	66(1)
C(26)	1456(2)	-1984(2)	-2466(1)	76(1)
C(27)	3500(1)	-1528(2)	716(1)	48(1)
C(28)	3162(2)	-1510(2)	1482(1)	65(1)

C(29)	4482(1)	-1892(3)	846(2)	84(1)
C(30)	466(1)	-221(1)	993(1)	35(1)
C(31)	243(1)	-362(1)	1623(1)	34(1)
C(32)	2(1)	-504(1)	2379(1)	37(1)
C(33)	616(1)	-255(2)	3050(1)	53(1)
C(34)	383(2)	-383(2)	3773(1)	69(1)
C(35)	-459(2)	-751(2)	3837(1)	71(1)
C(36)	-1073(2)	-997(2)	3178(1)	60(1)
C(37)	-848(1)	-876(2)	2453(1)	46(1)

Table 3. Bond lengths [Å] and angles [°] for may1704.

Ca-N(1)	2.3113(13)
Ca-N(2)	2.3182(12)
Ca-C(30)	2.5056(16)
Ca-C(30)’	2.5350(16)
Ca-C(31)’	2.9403(16)
N(1)-C(1)	1.3265(19)
N(1)-C(6)	1.4351(19)
N(2)-C(3)	1.3274(19)
N(2)-C(18)	1.434(2)
C(1)-C(2)	1.409(2)
C(1)-C(4)	1.519(2)
C(2)-C(3)	1.399(2)
C(3)-C(5)	1.519(2)
C(6)-C(7)	1.405(2)
C(6)-C(11)	1.409(2)
C(7)-C(8)	1.394(2)
C(7)-C(12)	1.521(2)
C(8)-C(9)	1.377(2)
C(9)-C(10)	1.381(2)
C(10)-C(11)	1.393(2)
C(11)-C(15)	1.520(2)
C(12)-C(14)	1.527(3)
C(12)-C(13)	1.533(3)
C(15)-C(16)	1.509(3)
C(15)-C(17)	1.517(3)
C(18)-C(23)	1.403(2)
C(18)-C(19)	1.408(2)
C(19)-C(20)	1.393(3)
C(19)-C(24)	1.517(2)
C(20)-C(21)	1.373(3)
C(21)-C(22)	1.382(3)
C(22)-C(23)	1.396(3)
C(23)-C(27)	1.516(3)
C(24)-C(26)	1.515(3)

C(24)-C(25)	1.534(3)
C(27)-C(28)	1.520(3)
C(27)-C(29)	1.530(3)
C(30)-C(31)	1.224(2)
C(31)-C(32)	1.448(2)
C(32)-C(33)	1.395(3)
C(32)-C(37)	1.397(2)
C(33)-C(34)	1.385(3)
C(34)-C(35)	1.379(3)
C(35)-C(36)	1.378(3)
C(36)-C(37)	1.382(3)

N(1)-Ca-N(2)	80.63(4)
N(1)-Ca-C(30)	117.93(5)
N(2)-Ca-C(30)	130.34(5)
N(1)-Ca-C(30)’	117.08(5)
N(2)-Ca-C(30)’	124.20(5)
C(30)-Ca-C(30)’	89.70(5)
N(1)-Ca-C(31)’	103.34(4)
N(2)-Ca-C(31)’	103.86(4)
C(30)-Ca-C(31)’	114.11(5)
C(30)’-Ca-C(31)’	24.41(5)
C(1)-N(1)-C(6)	117.33(12)
C(1)-N(1)-Ca	128.08(10)
C(6)-N(1)-Ca	114.58(9)
C(3)-N(2)-C(18)	119.98(12)
C(3)-N(2)-Ca	128.01(10)
C(18)-N(2)-Ca	112.01(9)
N(1)-C(1)-C(2)	124.21(14)
N(1)-C(1)-C(4)	119.78(14)
C(2)-C(1)-C(4)	116.00(13)
C(3)-C(2)-C(1)	130.18(14)
N(2)-C(3)-C(2)	124.12(14)
N(2)-C(3)-C(5)	120.38(14)
C(2)-C(3)-C(5)	115.45(13)
C(7)-C(6)-C(11)	120.75(14)

C(7)-C(6)-N(1)	120.49(13)
C(11)-C(6)-N(1)	118.73(13)
C(7)-C(6)-Ca	109.85(10)
C(11)-C(6)-Ca	112.65(10)
N(1)-C(6)-Ca	41.25(6)
C(8)-C(7)-C(6)	118.06(14)
C(8)-C(7)-C(12)	119.67(15)
C(6)-C(7)-C(12)	122.27(14)
C(9)-C(8)-C(7)	121.62(16)
C(8)-C(9)-C(10)	119.66(16)
C(9)-C(10)-C(11)	121.20(15)
C(10)-C(11)-C(6)	118.32(15)
C(10)-C(11)-C(15)	120.32(15)
C(6)-C(11)-C(15)	121.35(14)
C(7)-C(12)-C(14)	112.47(15)
C(7)-C(12)-C(13)	111.23(15)
C(14)-C(12)-C(13)	109.02(17)
C(16)-C(15)-C(17)	110.20(18)
C(16)-C(15)-C(11)	113.48(18)
C(17)-C(15)-C(11)	111.37(16)
C(23)-C(18)-C(19)	120.68(15)
C(23)-C(18)-N(2)	119.58(14)
C(19)-C(18)-N(2)	119.51(14)
C(20)-C(19)-C(18)	118.40(17)
C(20)-C(19)-C(24)	120.20(16)
C(18)-C(19)-C(24)	121.40(15)
C(21)-C(20)-C(19)	121.49(19)
C(20)-C(21)-C(22)	119.67(18)
C(21)-C(22)-C(23)	121.32(18)
C(22)-C(23)-C(18)	118.35(17)
C(22)-C(23)-C(27)	119.86(16)
C(18)-C(23)-C(27)	121.75(15)
C(26)-C(24)-C(19)	112.22(16)
C(26)-C(24)-C(25)	110.73(18)
C(19)-C(24)-C(25)	113.37(16)
C(23)-C(27)-C(28)	110.87(16)

C(23)-C(27)-C(29)	111.91(19)
C(28)-C(27)-C(29)	110.11(18)
C(31)-C(30)-Ca	172.85(13)
C(31)-C(30)-Ca’	96.68(11)
Ca-C(30)-Ca’	90.30(5)
C(30)-C(31)-C(32)	178.04(18)
C(30)-C(31)-Ca’	58.91(10)
C(32)-C(31)-Ca’	122.52(11)
C(33)-C(32)-C(37)	118.71(16)
C(33)-C(32)-C(31)	120.27(16)
C(37)-C(32)-C(31)	121.02(15)
C(34)-C(33)-C(32)	120.22(19)
C(35)-C(34)-C(33)	120.4(2)
C(36)-C(35)-C(34)	119.91(19)
C(35)-C(36)-C(37)	120.30(19)
C(36)-C(37)-C(32)	120.44(18)

Symmetry transformations used to generate equivalent atoms: ‘ -x,-y,-z

Least-squares planes (x,y,z in crystal coordinates) and deviations from them
(* indicates atom used to define plane)

9.6911 (0.0362) x - 0.9107 (0.0167) y + 11.0790 (0.0411) z = 1.8407 (0.0150)

```
*    0.0000 (0.0000)  C1
*    0.0000 (0.0000)  C2
*    0.0000 (0.0000)  C3
```

Rms deviation of fitted atoms = 0.0000

6.7235 (0.0146) x - 1.7946 (0.0058) y + 13.8079 (0.0118) z = 0.7924 (0.0041)

Angle to previous plane (with approximate esd) = 13.87 (0.31)

```
*   -0.0643 (0.0013)  C1
*    0.0368 (0.0008)  C4
*    0.0112 (0.0002)  N1
*    0.0163 (0.0003)  N2
    -0.2094 (0.0023)  C2
    -0.3394 (0.0023)  Ca
```

Rms deviation of fitted atoms = 0.0384

3.9478 (0.0074) x - 2.4562 (0.0072) y + 15.4687 (0.0043) z = 0.1021 (0.0011)

Angle to previous plane (with approximate esd) = 11.54 (0.10)

```
*    0.0000 (0.0000)  Ca
*    0.0000 (0.0000)  N1
*    0.0000 (0.0000)  N2
```

Rms deviation of fitted atoms = 0.0000

0.5953 (0.0056) x - 12.8360 (0.0007) y - 2.8312 (0.0053) z = 0.0000 (0.0000)

Angle to previous plane (with approximate esd) = 87.24 (0.04)

```
*   -0.0165 (0.0006)  Ca
*    0.0298 (0.0014)  C30
*    0.0192 (0.0014)  C31
*   -0.0270 (0.0010)  C32
*    0.0165 (0.0006)  Ca_$1
*   -0.0298 (0.0014)  C30_$1
*   -0.0192 (0.0014)  C31_$1
*    0.0270 (0.0010)  C32_$1
```

Rms deviation of fitted atoms = 0.0237

5.3041 (0.0135) x - 12.1614 (0.0045) y - 0.3930 (0.0165) z = 0.5191 (0.0050)

Angle to previous plane (with approximate esd) = 21.58 (0.04)

```
*    0.0011 (0.0014)  C32
*   -0.0013 (0.0016)  C33
*    0.0008 (0.0018)  C34
*   -0.0001 (0.0018)  C35
*   -0.0001 (0.0016)  C36
*   -0.0004 (0.0014)  C37

Rms deviation of fitted atoms =   0.0008
```