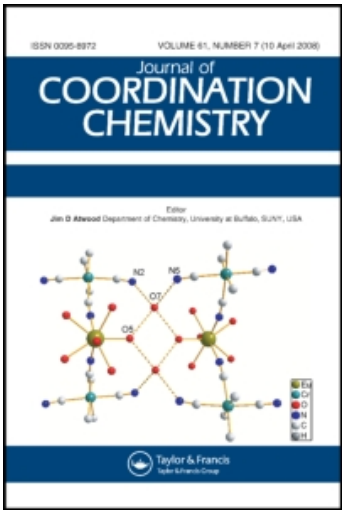

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Coordination Chemistry of Alkali and Alkaline Earth Cations: X-Ray Structural Analysis of Calcium(Picrate)$_2$(2,2'-Bipyridyl)$_2$

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COORDINATION CHEMISTRY OF ALKALI AND ALKALINE EARTH CATIONS: X-RAY STRUCTURAL ANALYSIS OF CALCIUM(PICRATE)$_2$(2,2′-BIPYRIDYL)$_2$

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$Ca(Pic)_2$ (Pic = 2,4,6-trinitrophenolate) and 2,2′-bipyridyl (BIPY) react fast to produce $Ca(Pic)_2(BIPY)_2$ irrespective of whether the reaction medium is protic (alcoholic) or aprotic (acetone). Crystals of the title compound $C_{32}H_{20}N_{10}O_{14}Ca$, $Mr = 808.7$, are orthorhombic, space group *Pbca*, $a = 24.154(5)$, $b = 16.128(5)$, $c = 18.068(6)$ Å, $V = 7038.5$ Å^3, $Z = 8$, $D_x = 1.528$, $D_m = 1.532$ g cm^{-3} (flotation), for MoKα, $= 2.53$ cm^{-1}. X-ray data were collected at room temperature. R refined to 0.68 for 1263 observed reflections. The calcium ion is 8-coordinate, four N atoms of the two BIPY molecules and four O atoms of the two Pic anions constituting a distorted square antiprism. In the coordination polyhedron, the Ca–O distances (2.292 and 2.357 Å) to phenoxide oxygens are shorter than the Ca–O nitro interactions (2.574 and 2.502 Å) and Ca–N (2.492–2.578 Å). The four binding groups are not symmetrically distributed around the cation. The nitro groups are severely rotated out of the plane of the ring. In both BIPY molecules the rings are twisted.

Keywords: 2,4,6-trinitrophenolate, 2,2′-bipyridyl, calcium complex, X-ray structure

INTRODUCTION

2,2′-Bipyridyl (BIPY) is a commonly known ligand for the complexation of transition cations.[1–3] Ligation of the ligand with alkali and alkaline earth cations has been found to be, in general, poor.[4–7] One of the reasons for this weakness is the tendency of the ligand molecule to exist in the *trans*-conformation[8–9] due to coulombic repulsions between the nitrogen atoms and a relatively poor $M^{z+}\cdots$ N(BIPY) bond strength.

Conductometric solution phase studies in acetone[10] revealed that the M^{2+}–BIPY interaction, for Pic as a counterion, is favoured by small cations, but that Ca was an exception and exhibited the lowest value of all the M^{2+} ions (Mg > Sr > Ba > Ca). Since one key to the understanding of the coordination chemistry of s-block cations concerns ligand *versus* anion preference,[11–13] the Ca behaviour led us to study the complex through single-crystal X-ray analysis. This is the only technique which can be used to study bonding features precisely in the absence of solvent effects.

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EXPERIMENTAL

The $Ca(Pic)_2(BIPY)_2$ crystals were grown by slowly evaporating at room temperature (25°C), an equimolar mixture (0.05 M) of $Ca(Pic)_2$ and BIPY in 1:1 acetone–ethanol. Yellow ochre, square crystals (m.p. 215–225°C) were obtained. Anal.; Calc.: C, 56.5; H, 2.9; N, 14.7%. Found: C, 56.6; H, 2.0; N, 14.5%.

Intensity data were collected on an indigenously fabricated computer-controlled four-circle diffractometer using Zr-filtered MoKα radiation, using a ω-2θ step scan. A total of 3889 reflections were collected using a crystal of size 3.50 × 0.25 × 0.75 mm. Of these, 1263 reflections, in the range $7° < 2\theta \leqslant 50°$, with $I > 2.5\sigma$ (I) were considered to be observed. The index range was h 0 → 22, k 0 → 15, and l 0 → 16. Corrections were applied for Lp and absorption effects; maximum and minimum transmission values were 0.939 and 0.834, respectively. Cell parameters were calculated from a least-squares refinement of the setting angles of 25 reflections (θ range, 8–15°). Two check reflections for every 100 data reflections did not vary significantly over the course of the data collection. Following Patterson and Fourier calculations, all hydrogens were located in a difference map. Structural refinement was carried out using least-squares methods based on F values with anisotropic thermal parameters for non-hydrogen atoms and isotropic parameters for hydrogen atoms. Final $R = 0.067$, $R_w = 0.097$ with $w = 0.5641/[\sigma^2(F) + 0.01058(F)^2]$; Δ/σ (max) = 0.63; Δp in final difference map <0.4 eA^{-3}. For C–H distances in the range 0.916(16) to 1.224(14) Å, e.s.d's are found to be large. This may be due to the poor ratio of observations to the number of variables (non-hydrogen 513 variables; hydrogen 80 variables). SHELX-76[14] and PLUTO-78[15] programmes were used for the structure solution and refinement. Atomic scattering factors were taken from SHELX-76.

RESULTS AND DISCUSSIONS

Structural aspects of the $Ca(Pic)_2(BIPY)_2$ molecule are shown in Figure 1, the calcium coordination sphere in Figure 2, and the molecular packing arrangement in Figure 3. Final positional parameters are listed in Table I while bond distances and angles are given in Table II. The calcium ion is 8-coordinate through two phenoxide oxygen (2.292 and 2.351 Å), two nitro oxygens of each Pic (2.574 and 2.502 Å), and four nitrogen atoms of the two BIPY molecules (Ca–N, 2.492 to 2.578 Å). One significant observation is that the two anions are located on one side. The two BIPY molecules fail to cause a symmetrical distribution of donors about the cation.

The Pic O–Ca–O bite angles (66.8(3) and 67.5(3)°) are less than the BIPY bite angles (64.6(4) and 63.2(4)°). The bond lengths and angles in the benzene rings of the two Pic ions agree with those reported for a number of nitrophenolate complexes,[16–18] and show common features such as larger C–C bond lengths and smaller C–C–C angles at phenoxide, and enlarged angles (~126°) at *ortho* C-atoms. The ring substituents show deviations from the least-squares phenyl plane in Pic_1 (N(5),0.12(1); N(10),0.06(1); and N(15),0.04(1) Å) and in Pic_2, these are less (N(21),0.01(1); N(26),0.010(1); and N(3),0.00(1) Å). Apart from bending relative to the plane of the benzene ring, the nitro groups display various degrees of twisting—moderate for *para-* nitro groups and large for *ortho-* nitro groups (Pic_1 : 7.2(7), 39.4(8), 23.6(8)°; Pic_2: 10.7(7), 18.5(8)). The two Pic planes are inclined at 55.9(8)° to each other. In the two BIPY molecules, the intraligand twist angles (based on parameters defined by Cordes *et al.*[19]) were found to be 19.7(9) and 0.2(9)° and are

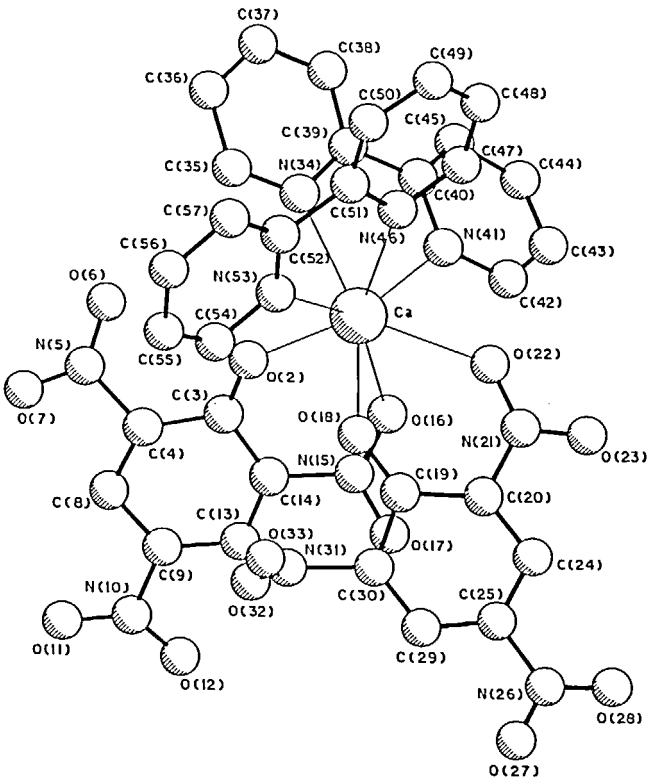

FIGURE 1 Perspective view of the molecule showing atomic labelling scheme.

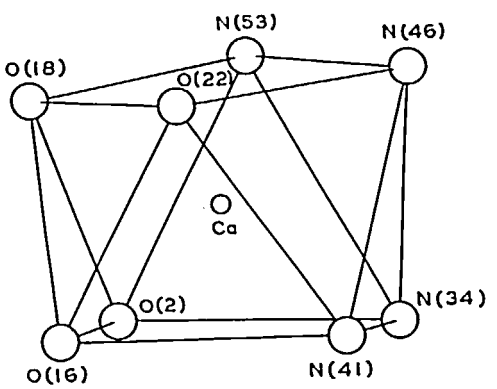

FIGURE 2 Atoms in the Ca coordination polyhedron.

approximately equal to the dihedral angles between the individual pyridine rings (19.4(7) and 5.4(7)°). This shows that both BIPY molecules are twisted and not bowed. The packing of the molecules is shown in Figure 3. No short non-bonding contacts are observed.

TABLE I

Final atomic coordinates ($\times 10^4$) and equivalent temperature factors (Å^2 $\times 10^3$) with e.s.d.'s in parentheses.

	x/a	*y/b*	*z/c*	*U*eq*
Ca(1)	1781(1)	665(2)	2633(2)	42(3)
(tnp1)				
O(2)	1438(4)	51(6)	1585(5)	54(6)
C(3)	1025(7)	−360(9)	1362(9)	51(8)
C(4)	923(6)	−484(8)	580(8)	42(7)
N(5)	1356(5)	−270(9)	14(8)	56(7)
O(6)	1826(5)	−408(8)	209(6)	93(7)
O(7)	1179(5)	15(8)	−567(7)	85(7)
C(8)	440(7)	−874(9)	271(8)	53(7)
C(9)	42(6)	−1161(9)	801(9)	48(7)
N(10)	−467(6)	−1590(9)	521(9)	71(8)
O(11)	−523(5)	−1626(9)	−149(8)	110(7)
O(12)	−769(5)	−1887(7)	977(7)	75(7)
C(13)	85(6)	−1049(9)	1561(9)	52(8)
C(14)	573(6)	−666(11)	1799(8)	56(8)
N(15)	602(5)	−548(9)	2627(8)	66(7)
O(16)	1063(4)	−469(7)	2921(6)	63(6)
O(17)	173(5)	−556(8)	2963(6)	89(7)
(tnp2)				
O(18)	898(3)	1278(7)	2646(5)	53(5)
O(19)	487(6)	1398(10)	3070(9)	51(6)
O(20)	478(6)	1285(9)	3878(9)	47(7)
N(21)	984(6)	965(8)	4224(8)	61(7)
O(22)	1450(4)	1013(7)	3907(6)	65(6)
O(23)	942(6)	686(9)	4857(7)	106(7)
C(24)	40(6)	1432(9)	4344(7)	45(7)
C(25)	−447(6)	1713(8)	4019(9)	52(7)
N(26)	−892(6)	1938(10)	4496(9)	80(9)
O(27)	−1305(5)	2308(8)	4336(7)	96(7)
O(28)	−900(6)	1748(9)	5127(8)	121(8)
C(29)	−487(7)	1876(10)	3301(10)	65(8)
C(30)	−62(8)	1673(10)	2845(10)	70(8)
N(31)	−84(7)	1841(12)	2025(11)	89(8)
O(32)	−193(7)	1229(11)	1668(8)	141(10)
O(33)	−29(8)	2475(11)	1782(9)	179(10)
(bip1)				
N(34)	2661(5)	80(8)	2057(6)	42(6)
C(35)	2829(7)	382(10)	1409(8)	53(7)
C(36)	3401(8)	302(10)	1153(9)	69(8)
C(37)	3761(7)	−169(12)	1627(10)	72(9)
C(38)	3562(7)	−463(11)	2285(9)	70(9)
C(39)	3021(6)	−364(10)	2513(10)	55(8)
C(40)	2800(5)	−732(9)	3178(7)	35(7)
N(41)	2313(5)	−388(7)	3439(7)	54(8)
C(42)	2106(7)	−701(10)	4071(8)	59(9)
C(43)	2359(7)	−1356(11)	4455(8)	65(9)
C(44)	2833(7)	−1699(10)	4145(9)	68(9)
C(45)	3076(7)	−1392(11)	3529(8)	65(9)
(bip2)				

TABLE I (*continued*)

	x/a	*y/b*	*z/c*	*Ueq**
N(46)	2568(5)	1637(7)	2995(6)	47(8)
C(47)	2921(6)	1485(10)	3594(9)	62(7)
C(48)	3364(6)	1977(11)	3759(8)	50(6)
C(49)	3526(6)	2598(12)	3319(9)	60(7)
C(50)	3187(6)	2715(9)	2702(7)	44(6)
C(51)	2724(7)	2277(9)	2530(9)	57(6)
C(52)	2367(6)	2413(10)	1862(8)	42(6)
N(53)	1924(5)	1909(8)	1838(6)	51(7)
C(54)	1558(7)	2072(9)	1258(9)	60(7)
C(55)	1623(7)	2665(11)	789(9)	63(7)
C(56)	2083(7)	3201(10)	806(8)	63(7)
C(57)	2472(7)	3025(9)	1383(8)	54(6)

* $Ueq = (U11 + U22 + U33)/3$.

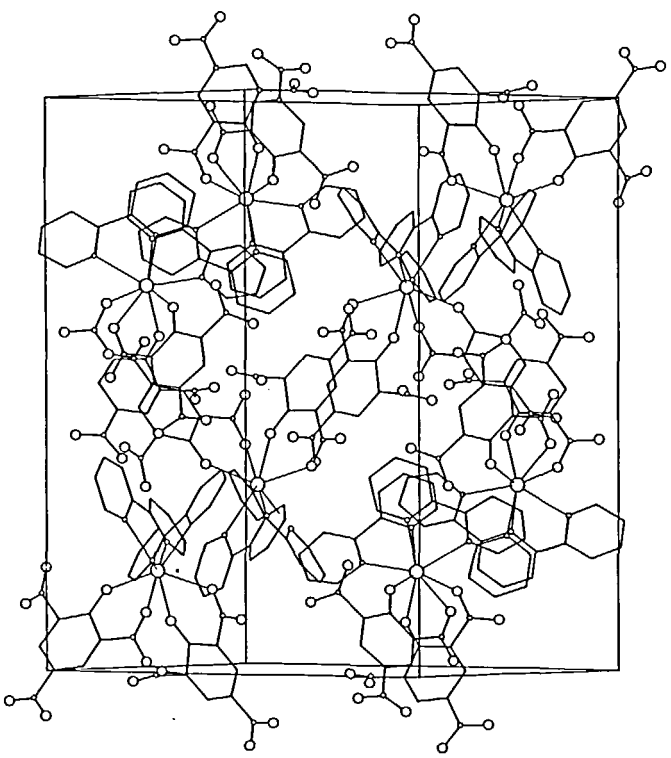

FIGURE 3 Molecular packing in the unit cell.

TABLE II

Bond lengths (Å) and angles (°) with e.s.d.'s in parentheses.

(a) lengths			
Ca(1)–O(2)	2.292(10)	O(2)–O(16)	2.712(12)
Ca(1)–O(18)	2.351(9)	O(16)–N(41)	3.164(13)
Ca(1)–O(16)	2.574(11)	N(41)–N(31)	2.741(13)
Ca(1)–O(22)	2.502(10)	N(34)–O(2)	3.075(14)
Ca(1)–N(34)	2.547(11)	O(18)–O(22)	2.675(14)
Ca(1)–N(41)	2.579(12)	O(22)–N(46)	3.321(14)
Ca(1)–N(46)	2.550(12)	N(46)–N(53)	2.643(13)
Ca(1)–N(53)	2.492(12)	N(53)–O(18)	3.050(13)
(tnp1)		(tnp2)	
O(2)–C(3)	1.265(18)	O(18)–C(19)	1.270(17)
C(3)–C(4)	1.448(21)	C(19)–C(20)	1.472(23)
C(4)–C(8)	1.439(21)	C(20)–C(24)	1.372(20)
C(8)–C(9)	1.433(20)	C(24)–C(25)	1.392(21)
C(9)–C(13)	1.388(21)	C(25)–C(29)	1.327(24)
C(13)–C(14)	1.398(20)	C(29)–C(30)	1.356(25)
C(14)–C(3)	1.435(21)	C(30)–C(19)	1.457(23)
C(4)–N(5)	1.502(19)	C(20)–N(21)	1.467(20)
C(9)–N(10)	1.499(20)	C(25)–N(26)	1.425(22)
C(14)–N(15)	1.511(20)	C(30)–N(31)	1.507(26)
N(5)–O(6)	1.211(17)	N(21)–O(22)	1.266(17)
N(5)–O(7)	1.223(18)	N(21)–O(23)	1.233(18)
N(10)–O(11)	1.218(20)	N(26)–O(27)	1.258(19)
N(10)–O(12)	1.201(20)	N(26)–O(28)	1.181(20)
N(15)–O(16)	1.239(16)	N(31)–O(32)	1.209(25)
N(15)–O(17)	1.202(18)	N(31)–O(33)	1.121(27)
(bip1)		(bip2)	
N(34)–C(35)	1.332(18)	N(46)–C(47)	1.400(19)
C(35)–C(36)	1.463(23)	C(47)–C(48)	1.365(21)
C(36)–C(37)	1.437(24)	C(48)–C(49)	1.337(23)
C(37)–C(38)	1.369(24)	C(49)–C(50)	1.397(21)
C(38)–C(39)	1.378(22)	C(50)–C(51)	1.359(21)
C(39)–N(34)	1.395(19)	C(51)–N(46)	1.383(19)
C(39)–C(40)	1.444(21)	C(51)–C(52)	1.499(21)
C(40)–N(41)	1.382(18)	C(52)–N(53)	1.344(18)
N(41)–C(42)	1.345(19)	N(53)–C(54)	1.395(20)
C(42)–C(43)	1.403(23)	C(54)–C(55)	1.287(22)
C(43)–C(44)	1.390(23)	C(55)–C(56)	1.408(23)
C(44)–C(45)	1.352(22)	C(56)–C(57)	1.433(22)
C(45)–C(40)	1.408(21)	C(57)–C(52)	1.336(20)
(b) angles			
O(2)–Ca(1)–O(16)	67.5(3)	O(2)–O(16)–N(41)	86.2(6)
N(34)–Ca(1)–N(41)	64.6(4)	O(16)–N(41)–N(34)	92.1(8)
O(18)–Ca(1)–O(22)	66.8(3)	N(41)–N(34)–O(2)	87.3(8)
N(46)–Ca(1)–N(53)	63.2(4)	N(34)–O(2)–O(16)	94.5(6)
O(2)–Ca(1)–O(18)	82.1(3)	O(18)–O(22)–N(46)	86.2(6)
O(18)–Ca(1)–O(16)	71.7(3)	O(22)–N(46)–N(53)	87.9(8)
O(16)–Ca(1)–O(22)	76.0(3)	N(46)–N(53)–O(18)	92.6(6)
O(22)–Ca(1)–N(41)	77.7(4)	N(53)–O(18)–O(22)	93.0(6)
N(41)–Ca(1)–N(46)	83.6(4)		
N(46)–Ca(1)–N(34)	73.2(4)		
N(34)–Ca(1)–N(53)	87.0(4)		

TABLE II (*continued*)

N(53)–Ca(1)–O(2)	85.5(4)		
(tnp1)		(tnp2)	
O(2)–C(3)–C(4)	111.1(13)	O(18)–C(19)–C(20)	126.3(13)
C(3)–C(4)–N(5)	121.0(13)	C(19)–C(20)–N(21)	117.0(13)
C(4)–N(5)–O(6)	115.3(12)	C(20)–N(21)–O(22)	121.8(12)
C(4)–N(5)–O(7)	114.3(13)	C(20)–N(21)–O(23)	117.2(13)
O(6)–N(5)–O(7)	130.3(14)	O(22)–N(21)–O(23)	120.9(14)
N(5)–C(4)–C(8)	113.6(12)	N(21)–C(20)–C(24)	116.2(14)
C(3)–C(4)–C(8)	125.2(13)	C(19)–C(20)–C(24)	126.8(13)
C(4)–C(8)–C(9)	115.3(12)	C(20)–C(24)–C(25)	116.6(13)
C(8)–C(9)–N(10)	118.2(13)	C(24)–C(25)–N(26)	117.7(14)
C(9)–N(10)–O(11)	116.6(14)	C(25)–N(26)–O(27)	118.8(15)
C(9)–N(10)–O(12)	116.8(14)	C(25)–N(26)–O(28)	122.1(15)
O(11)–N(10)–O(12)	126.5(15)	O(27)–N(26)–O(28)	118.8(15)
N(10)–C(9)–C(13)	117.1(13)	N(26)–C(25)–C(29)	119.1(14)
C(8)–C(9)–C(13)	124.6(13)	C(24)–C(25)–C(29)	122.6(15)
C(9)–C(13)–C(14)	115.1(13)	C(25)–C(29)–C(30)	119.3(16)
C(13)–C(14)–N(15)	113.6(12)	C(29)–C(30)–N(31)	121.8(15)
C(14)–N(15)–O(16)	118.7(12)	C(30)–N(31)–O(32)	112.6(16)
C(14)–N(15)–O(17)	117.2(12)	C(30)–N(31)–O(33)	123.1(18)
O(16)–N(15)–O(17)	124.0(14)	O(32)–N(31)–O(33)	124.2(20)
N(15)–C(14)–C(3)	117.7(13)	N(31)–C(30)–C(19)	111.1(14)
C(13)–C(14)–C(3)	128.6(14)	C(29)–C(30)–C(19)	126.5(16)
C(14)–C(3)–C(4)	127.3(14)	C(30)–C(19)–C(20)	107.5(13)
C(14)–C(3)–O(2)	127.3(14)	C(30)–C(19)–O(18)	126.2(14)
(bip1)		(bip2)	
N(34)–C(35)–C(36)	122.2(13)	N(46)–C(47)–C(48)	105.8(12)
C(35)–C(36)–C(37)	115.4(14)	C(47)–C(48)–C(49)	122.4(14)
C(36)–C(37)–C(38)	119.3(15)	C(48)–C(49)–C(50)	113.8(14)
C(37)–C(38)–C(39)	123.4(16)	C(49)–C(50)–C(51)	126.6(14)
C(38)–C(39)–N(34)	118.3(14)	C(50)–C(51)–N(46)	118.2(14)
C(39)–N(34)–C(35)	121.2(12)	C(51)–N(46)–C(47)	115.8(12)
C(38)–C(39)–C(40)	123.5(14)	C(50)–C(51)–C(52)	125.6(14)
N(34)–C(39)–C(40)	118.1(12)	N(46)–C(51)–C(52)	116.3(13)
C(39)–C(40)–N(41)	123.6(12)	C(51)–C(52)–N(53)	113.3(12)
C(40)–N(41)–C(42)	117.1(12)	C(52)–N(53)–C(54)	114.5(12)
N(41)–C(42)–C(43)	126.6(14)	N(53)–C(54)–C(55)	123.8(15)
C(42)–C(43)–C(44)	117.4(14)	C(54)–C(55)–C(56)	122.6(15)
C(43)–C(44)–C(45)	122.9(15)	C(55)–C(56)–C(57)	114.3(14)
C(44)–C(45)–C(40)	116.2(14)	C(56)–C(57)–C(52)	119.5(15)
C(45)–C(40)–N(41)	123.6(12)	C(57)–C(52)–N(53)	125.2(13)
C(45)–C(40)–C(39)	120.7(12)	C(57)–C(52)–C(51)	121.3(13)

The symmetry of the coordination polyhedron was examined by applying the Lippard and Russ[20] test. The angle between intersecting trapezoidal best planes was 85.4(9)°. On these grounds the coordination sphere is best described as intermediate between an ideal square antiprism (77.4°) and the ideal dodecahedron (90°). The additional test based on the comparison of average distances of ligand atoms to trapezoidal planes ($dT_1 = 0.275(15)$, $dT_2 = 0.314(14)$ Å) with average distances of the ligand atoms to the best planes through the square faces of the antiprism ($dS_1 =$

0.019(8), $dS_2 = 0.23(9)$ Å) suggests that the best description would be a distorted square antiprism.

Calcium is highly anionphilic towards the Pic anion[21], and the Ca–O bonds are indeed very short (2.292 and 2.351 Å). However, the cation has successfully polarized the N(BIPY) atoms so that Ca–N (2.492–2.599 Å) distances are comparable to Ca–O(NO), 2.562 and 2.574 Å. Hard O-donors are usually preferred to the alkaline earth ions. The BIPY molecules do not appear to have caused much $Ca^{2+} \cdots Pic^-$ bond loosening. Hence "undissociated" $Ca(Pic)_2$ appears to have undergone complexation with BIPY molecules to yield a complex involving an unsymmetrical distribution of ligands as is the case with $Ca(NCS)_2(BIPY)_2.H_2O$,[7] but not with the analogous complex $Ba(NCS)_2$ $(BIPY)_2$.[7] Unsymmetrical ligation of Ca^{2+} is also noted for Pic even under the complexing effect of benzo-15-crown-5.[21] This helps to explain why $Ca(Pic)_2$ in acetone is the least conducting of all the $M(Pic)_2$ salts examined and the conductance sequence Mg > Sr > Ba > Ca as mentioned above.

SUPPLEMENTARY DATA

Lists of observed and calculated structure factors, least-squares plane data and torsion angles, anisotropic thermal parameters and hydrogen atom positional and thermal data are available from NSP upon request.

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