## Title

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# The Crystal Structure of Tris-(2-aminoethyl)aminochlorozinc(II) Tetraphenylborate[1]

By Rodney J. Sime, Richard P. Dodge, Allan Zalkin, and David H. Templeton

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(1) Work done under the auspices of the U. S. Atomic Energy Commission.

The crystal structure of tris-(2-aminoethyl)aminochlorozinc(II) tetraphenylborate has been determined from an X-ray study of a single crystal specimen. The monoclinic cell, space group $P2_1/c$, with $\underline{a}$ = 13.76±0.04, $\underline{b}$ = 10.33±0.03, $\underline{c}$ = 20.35±0.06 Å, and $\underline{\beta}$ = 95.0±0.2°, contains four formula units; the calculated X-ray density is 1.30 g/cc. The structure was refined to a conventional R factor of 0.041 for 2193 structure factors. The structure consists of a $Zn(C_2H_4NH_2)_3NCl$ cation and a $(C_6H_5)_4B$ anion. The cation has approximate $C_3$ symmetry. Within the cation the Zn atom is penta-coordinated to one chlorine and four nitrogen atoms in a trigonal bipyramidal configuration.

## Introduction

Although the coordination number five is generally regarded as an unusual one for first-row transition metals, a steadily increasing number of five-coordinated complexes are being described in the literature. In particular, the quadridentate ligands, tris(2-dimethylaminoethyl)amine and tris(2-aminoethyl)amine appear to form a variety of five-coordinated complexes with the first-row transition metal ions from manganese(II) to zinc(II). These ligands are more conveniently designated $Me_6$tren and tren, respectively. In general, these may be formulated as $[M^{II}(Me_6tren)X]Y$ or $[M^{II}(tren)X]Y$. The relative stability of five coordination among these metals is favored in the order (Co, Cu, Zn) > (Fe, Ni) > Mn.[2] Because of the increased bulkiness

(2) M. Ciampolini and P. Paoletti, Inorg. Chem., 6, 1261 (1967).

of $Me_6$tren, it forms more stable five-coordinated complexes than tren. $Me_6$tren complexes have been described, for which M = Mn, Fe, Co, Ni, Cu and Zn, and for which X = Y = Cl, Br, I, $NO_3$, and $ClO_4$.[3,4] Much physical evidence,

(3) M. Ciampolini and N. Nardi, Inorg. Chem., 5, 41 (1966).

(4) M. Ciampolini and N. Nardi, Inorg. Chem., 5, 1150 (1966).

including conductivity, spectral and magnetic measurements, indicated that these complexes are five-coordinated. In addition, crystal structure determinations of $[Cu(tren)(NCS)]SCN$[5] and $[Co(Me_6tren)Br]Br$[6] reveal that the copper

(5) P. C. Jain and E. C. Lingafelter, J. Am. Chem. Soc., 89, 724 (1967).

(6) M. DiVaira and P. L. Orioli, Inorg. Chem., 6, 955 (1967).

and cobalt ions are indeed five-coordinated, and situated nearly at the center of a slightly distorted trigonal bipyramid. A recent report[7] on the structure

(7) G. D. Andreetti, P. C. Jain, and E. C. Lingafelter, J. Am. Chem. Soc., 91, 4112 (1969).

of Zn(tren)(NCS)(SCN) also shows trigonal bipyramidal symmetry.

More recently, a series of complexes have been prepared, [Zn(tren)X]Y, for which X = Cl, Br and I, and Y = X, $ZnX_3$, and $B(C_6H_5)_4$.[8] We report here

(8) L. V. Interrante, Inorg. Chem., 7, 943 (1968).

the crystal structure of the five-coordinated complex $[Zn(C_2H_4NH_2)_3NCl]B(C_6H_5)_4$.

## Experimental Section

Dr. L. V. Interrante kindly supplied us with some well-formed, colorless, prisms of $[Zn(tren)Cl]B(C_6H_5)_4$, which were suitable for the structural analysis. The determination of the space group and cell dimensions was made using the precession technique and molybdenum radiation (Mo $K\alpha_1$, $\lambda$ = 0.70926 Å). A General Electric XRD-5 X-ray diffraction apparatus equipped with a molybdenum X-ray tube, a scintillation counter, a pulse height discriminator, and a quarter-circle Eulerian-cradle type of goniostat was used to collect the intensity data. The X-ray tube was operated at 45 kv and 20 ma; a 0.003 in. thick Zr filter was used on the receiving slit. The crystal was oriented such that the c axis was parallel to the $\phi$ axis of the instrument.

A total of 2692 independent intensities were measured, of which 233 were recorded as having zero intensity. A stationary-crystal, stationary-counter technique with a 10-sec. count for every reflection was used. The diffractometer was set at a 4° take-off angle to the tube. The maximum $2\theta$ angle was 40° [$(\sin\theta)/\lambda$ = 0.596]. Background was plotted as a function of $2\theta$ and these values were used for most of the intensities; in the cases where background was seriously affected by streaking, individual backgrounds were measured. The absorption parameter is 9.9 $cm^{-1}$, and gives an estimated $\mu R \sim 1.0$ for the crystal used. Lorentz and polarization corrections were made, but no correction was made for either absorption or extinction.

Fourier, least-squares and distance calculations were performed using our own unpublished programs. The full-matrix least squares program, which is a modification of an early unpublished version of one given us by P. Gantzel, R. Sparks, and K. Trueblood, minimizes the function $\Sigma w(|F_o| - |F_c|)^2/\Sigma wF_o^2$; $F_o$ and $F_c$ are the observed and calculated structure factors respectively, and

w is the weighing factor. Atomic scattering factors[9,10], for neutral zinc,

(9) D. T. Cromer and J. T. Waber, Acta Cryst., 18, 104 (1965).

(10) R. F. Stewart, E. R. Davidson, W. T. Simpson, J. Chem. Phys., 42, 3175 (1965).

chlorine, boron, carbon, nitrogen, oxygen, and hydrogen were used. Both the real and imaginary parts of the anomalous dispersion for zinc and chlorine[11]

(11) D. T. Cromer, Acta Cryst., 18, 17 (1965).

were included in the least squares calculations. For each reflection w was set to $1.0/\sigma^2(F_o)$ with the exception that w = 0 when I(net count) $\leq \sigma(I)$. The standard deviation of the observed structure factor was calculated as $\sigma(F_o) = F_o - (F_o^2 - s\sigma(I)/Lp)^{1/2}$ where s is a scaling factor, $F_o = (sI/Lp)^{1/2}$, and Lp is the Lorentz-polarization correction. $\sigma(I) = (I + p^2I^2 + 2B + q^2)^{1/2}$, where p is a fractional uncertainty in I, B is the background, and q represents an uncertainty in the background; p was set to 0.04 and q to 12.

The monoclinic unit cell contains four formula units and has cell dimensions; a = 13.76±0.04, b = 10.33±0.03, c = 20.35±0.06 Å, and $\beta = 95.0\pm0.2^\circ$. The extinctions observed were consistent with space group $P2_1/c$. The density calculated from the X-ray data is 1.304 g/cc. The crystals were observed to float readily in carbon tetrachloride (d = 1.58 g/cc).

## Determination of Structure

Trial coordinates for the zinc and chlorine atoms were derived from the Patterson function. A three dimensional electron density map revealed the locations of the remaining 35 non-hydrogen atoms. Three cycles of least squares refinements with isotropic temperature factors of the form $\exp(-B\lambda^{-2}\sin^2\theta)$ gave a value of $R = 0.12$, where $R = \Sigma|\Delta F|/\Sigma|F_o|$ and $\Delta F = |F_o| - |F_c|$. Several more cycles of least-squares calculations with anisotropic thermal parameters reduced $R$ to 0.077. The anisotropic temperature factors have the form $\exp(-\beta_{11}h^2-\beta_{22}k^2-\beta_{33}\ell^2-2\beta_{12}hk-2\beta_{13}h\ell-2\beta_{23}k\ell)$. In reporting the thermal parameters below we have converted $\beta_{ij}$ to $B_{ij}$ which is in units of $\text{Å}^2$; the relation between these two quantities is $4\beta_{ij} = a_i^* a_j^* B_{ij}$, where $a_i^*$ is the $i$th reciprocal axis. All the hydrogen positions were located from a difference Fourier and included in the least squares refinements with isotropic temperature factors.

Due to the limitation of the memory size of our computer, ~120,000 words, it was necessary to divide the structure into two parts and refine the two parts alternately. We arbitrarily divided the structure into the cation and anion and refined one group while the other was kept fixed. All atoms with the exception of the hydrogen atoms were treated with anisotropic temperature factors. For the very last 2 cycles of refinement, the scheme was changed and the parameters of all 37 heavy atoms were refined in one pass; and the 40 hydrogen atoms were included but not refined. No positional parameter shifted more than 8% of its standard deviation. The final R value for 2193 non-zero weighted data is 0.041, and 0.059 for all 2692 data. The weighted R value, $(\Sigma w(\Delta F)^2)/\Sigma wF_o^2)^{1/2}$, is 0.040. The standard deviation of an observation of unit weight is 1.02. The observed and calculated structure factors are given

in Table I. The final positional parameters of the heavy atoms are listed in Table II, and the corresponding anisotropic thermal parameters in Table III. The positional and thermal parameters for hydrogen are listed in Table IV.

TABLE I

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR

TRIS-(2-AMINOETHYL)AMINOCHLOROZINC(II) TETRAPHENYLBORATE[a]

[a]Zero weighted data are indicated with asterisks.

UNSERVED AND CALCULATED STRUCTURE FACTORS OF TRIS-(2-AMINOETHYL)AMINOCHLOROZINC(II)TETRAPHENYLBORATE.
FCA10.0,01 = 4788

TABLE II

POSITIONAL PARAMETERS OF THE NON-HYDROGEN ATOMS[a]

[a]Numbers in parentheses in this and subsequent tables are estimated standard deviations of the last digits.

| ATOM | X | Y | Z |
|---|---|---|---|
| ZN | .15353( 4) | .05548( 6) | .09866( 3) |
| CL | .0476( 1) | -.1178( 1) | .08697( 7) |
| N1 | .2522( 3) | .2384( 4) | .1078( 2) |
| N2 | .1915( 3) | .0569( 5) | .1992( 2) |
| N3 | .2661( 3) | -.0002( 4) | .0443( 2) |
| N4 | .0580( 3) | .1967( 5) | .0613( 2) |
| C1 | .2893( 4) | .2481( 6) | .1790( 3) |
| C2 | .2151( 4) | .1902( 7) | .2199( 3) |
| C3 | .1880( 5) | .3479( 6) | .0858( 3) |
| C4 | .1093( 4) | .3057( 6) | .0363( 3) |
| C5 | .3299( 4) | .2155( 6) | .0660( 3) |
| C6 | .3548( 4) | .0743( 6) | .0631( 3) |
| B | .7365( 4) | .2874( 6) | .1185( 3) |
| C11 | .6621( 3) | .3573( 5) | .1667( 2) |
| C12 | .6280( 4) | .2996( 5) | .2221( 3) |
| C13 | .5693( 4) | .3643( 6) | .2623( 3) |
| C14 | .5403( 4) | .4883( 6) | .2492( 3) |
| C15 | .5740( 4) | .5499( 6) | .1962( 3) |
| C16 | .6343( 4) | .4851( 6) | .1564( 3) |
| C21 | .8424( 4) | .3476( 5) | .1446( 3) |
| C22 | .8938( 4) | .3038( 5) | .2031( 3) |
| C23 | .9776( 5) | .3622( 8) | .2312( 3) |
| C24 | 1.0104( 4) | .4718( 8) | .2038( 4) |
| C25 | .9639( 5) | .5192( 6) | .1466( 4) |
| C26 | .8821( 4) | .4576( 6) | .1191( 3) |
| C31 | .7288( 4) | .1292( 5) | .1224( 2) |
| C32 | .8069( 4) | .0441( 6) | .1267( 2) |
| C33 | .7955( 4) | -.0911( 5) | .1245( 3) |
| C34 | .7057( 5) | -.1447( 6) | .1172( 3) |
| C35 | .6251( 4) | -.0659( 7) | .1119( 3) |
| C36 | .6381( 4) | .0673( 6) | .1142( 2) |
| C41 | .7065( 4) | .3208( 5) | .0406( 2) |
| C42 | .7726( 4) | .3092( 5) | -.0076( 3) |
| C43 | .7460( 5) | .3247( 5) | -.0742( 3) |
| C44 | .6522( 6) | .3544( 6) | -.0949( 3) |
| C45 | .5839( 4) | .3684( 7) | -.0512( 3) |
| C46 | .6120( 4) | .3487( 6) | .0155( 3) |

TABLE III

ANISOTROPIC THERMAL PARAMETERS[a]

[a]Units of these parameters are $\AA^2$ as described in the text.

| ATOM | B11 | B22 | B33 | B12 | B13 | B23 |
|---|---|---|---|---|---|---|
| ZN | 3.75( 3) | 3.67( 3) | 3.29( 3) | -.09( 3) | .29( 2) | .05( 3) |
| CL | 4.44( 8) | 4.79( 8) | 4.80( 8) | -.96( 7) | .77( 6) | -.25( 7) |
| N1 | 4.47(26) | 3.94(26) | 3.92(26) | -.61(22) | .76(22) | -.16(21) |
| N2 | 4.43(24) | 6.30(30) | 3.89(24) | -.63(25) | .29(18) | .19(24) |
| N3 | 4.75(25) | 4.32(26) | 3.91(23) | -.62(21) | .73(19) | -.60(19) |
| N4 | 4.49(25) | 5.06(28) | 4.60(25) | -.79(22) | .24(20) | -.54(22) |
| C1 | 5.42(34) | 4.36(34) | 5.08(35) | .16(29) | .10(29) | -.80(28) |
| C2 | 5.52(37) | 7.30(44) | 3.54(31) | 1.04(33) | -.74(27) | -1.67(31) |
| C3 | 6.87(41) | 4.09(36) | 5.90(38) | -.56(33) | .39(31) | .85(30) |
| C4 | 5.90(36) | 4.35(35) | 4.83(33) | .96(30) | -.26(29) | .75(29) |
| C5 | 5.41(38) | 5.34(42) | 5.04(34) | -1.80(31) | .23(29) | .29(29) |
| C6 | 4.59(32) | 5.30(37) | 4.34(30) | .29(30) | 1.01(25) | -.51(28) |
| B | 3.11(34) | 2.55(34) | 3.51(33) | .22(26) | .51(26) | .69(26) |
| C11 | 2.79(26) | 3.19(32) | 3.13(29) | .18(24) | .11(21) | .09(24) |
| C12 | 5.53(35) | 3.92(33) | 4.58(33) | .61(28) | 1.93(28) | .38(29) |
| C13 | 6.57(38) | 4.53(38) | 4.49(34) | .38(33) | 2.29(29) | .67(30) |
| C14 | 4.60(32) | 5.10(38) | 3.62(31) | .86(28) | 1.09(26) | -.74(27) |
| C15 | 5.62(32) | 3.82(31) | 4.26(31) | 1.96(31) | .10(26) | .15(30) |
| C16 | 4.34(30) | 3.65(33) | 3.70(29) | .68(25) | 1.02(24) | -.13(25) |
| C21 | 3.48(28) | 3.06(32) | 2.98(28) | .98(25) | .66(24) | -.19(23) |
| C22 | 4.32(33) | 3.94(32) | 4.18(32) | .12(28) | .92(26) | -.30(27) |
| C23 | 4.61(39) | 7.34(47) | 3.89(33) | .50(35) | .24(28) | -1.33(33) |
| C24 | 3.35(32) | 6.78(48) | 7.03(46) | -.80(34) | .08(32) | -3.30(38) |
| C25 | 4.72(36) | 4.93(39) | 7.16(42) | -.77(32) | .67(32) | -.37(34) |
| C26 | 4.01(31) | 4.57(36) | 4.16(30) | -.56(30) | .16(25) | .10(29) |
| C31 | 2.86(28) | 3.41(29) | 2.86(26) | -.06(27) | .61(21) | -.14(22) |
| C32 | 3.46(29) | 3.38(32) | 4.05(29) | .04(27) | .44(22) | -.02(26) |
| C33 | 4.56(36) | 2.88(36) | 4.36(30) | .95(26) | -.06(25) | .54(23) |
| C34 | 5.91(38) | 3.46(33) | 3.96(30) | .11(35) | .68(28) | .60(25) |
| C35 | 4.45(34) | 4.17(38) | 4.82(32) | -.64(32) | 1.25(24) | -.13(28) |
| C36 | 3.25(31) | 3.68(35) | 5.10(32) | .10(27) | 1.17(23) | -.29(26) |
| C41 | 3.11(28) | 2.68(27) | 3.75(29) | .03(23) | .06(25) | .24(22) |
| C42 | 4.95(32) | 3.31(29) | 2.76(29) | .16(24) | .55(25) | -.14(23) |
| C43 | 6.06(39) | 3.77(32) | 3.16(34) | -.22(29) | .86(27) | -.09(25) |
| C44 | 7.87(46) | 4.89(36) | 2.67(31) | .11(35) | -.69(34) | -.34(27) |
| C45 | 4.87(36) | 8.78(46) | 4.54(38) | .44(34) | -.99(31) | .01(34) |
| C46 | 3.96(34) | 6.36(39) | 4.32(35) | .55(30) | .40(26) | -.66(28) |

TABLE IV

POSITIONAL AND THERMAL PARAMETERS OF THE HYDROGEN ATOMS[a]

[a]Units of B are $Å^2$.

| ATOM | X | Y | Z | B |
|---|---|---|---|---|
| H1 N2 | .1407(45) | .0337(63) | .2152(30) | 7.7(24) |
| H2 N2 | .2434(37) | .0149(51) | .2071(25) | 4.6(19) |
| H1 N3 | .2813(48) | -.0843(68) | .0510(33) | 9.6(27) |
| H2 N3 | .2538(34) | .0063(47) | .0034(24) | 4.1(17) |
| H1 N4 | .0190(30) | .1559(41) | .0354(20) | 1.8(13) |
| H2 N4 | .0268(39) | .2122(56) | .0939(26) | 5.3(20) |
| H1 C1 | .3501(32) | .1959(43) | .1868(20) | 3.5(13) |
| H2 C1 | .3047(32) | .3489(50) | .1895(22) | 4.9(13) |
| H1 C2 | .2371(26) | .1950(36) | .2609(19) | 1.1(10) |
| H2 C2 | .1530(37) | .2342(50) | .2140(23) | 5.0(15) |
| H1 C3 | .1577(42) | .3727(62) | .1212(30) | 7.6(22) |
| H2 C3 | .2303(32) | .4095(47) | .0715(22) | 3.8(14) |
| H1 C4 | .0603(31) | .3807(44) | .0266(21) | 4.0(12) |
| H2 C4 | .1357(37) | .2804(55) | -.0139(27) | 8.1(17) |
| H1 C5 | .3134(34) | .2423(49) | .0226(23) | 4.7(15) |
| H2 C5 | .3855(33) | .2706(45) | .0799(20) | 3.8(13) |
| H1 C6 | .4036(36) | .0611(50) | .0325(24) | 5.4(15) |
| H2 C6 | .3804(30) | .0517(43) | .1049(22) | 3.0(12) |
| H C12 | .6554(36) | .2131(51) | .2282(23) | 4.9(15) |
| H C13 | .5559(33) | .3213(46) | .3022(24) | 4.1(14) |
| H C14 | .4968(33) | .5346(47) | .2768(22) | 4.0(13) |
| H C15 | .5592(32) | .6387(48) | .1871(22) | 3.6(14) |
| H C16 | .6581(32) | .5363(45) | .1200(23) | 3.8(13) |
| H C22 | .8689(26) | .2291(38) | .2179(17) | .3(10) |
| H C23 | 1.0039(39) | .3329(54) | .2753(28) | 6.7(18) |
| H C24 | 1.0634(37) | .5036(51) | .2186(25) | 4.2(17) |
| H C25 | .9840(41) | .5909(59) | .1239(28) | 6.4(20) |
| H C26 | .8521(31) | .4956(42) | .0837(21) | 2.3(13) |
| H C32 | .8745(35) | .0716(47) | .1326(22) | 4.1(14) |
| H C33 | .8489(32) | -.1457(46) | .1311(21) | 3.0(13) |
| H C34 | .6967(34) | -.2314(51) | .1187(22) | 3.5(15) |
| H C35 | .5595(34) | -.0982(43) | .1009(21) | 3.1(13) |
| H C36 | .5857(30) | .1149(41) | .1115(19) | 1.6(12) |
| H C42 | .8443(31) | .2827(40) | .0018(19) | 2.5(11) |
| H C43 | .7985(35) | .3167(48) | -.1048(25) | 5.3(16) |
| H C44 | .6389(33) | .3613(46) | -.1392(25) | 3.9(14) |
| H C45 | .5212(41) | .3741(55) | -.0661(27) | 5.8(18) |
| H C46 | .5665(32) | .3600(44) | .0408(22) | 2.5(13) |

## Description of the Structure

In the cation, the zinc is surrounded by four nitrogen atoms, from the tren molecule, and one chlorine atom. A view of the structure down the Cl-Zn bond, reveals the nearly three-fold symmetry of the coordinated tren molecule neatly coiled about the zinc atom (Fig. 1). The zinc atom lies 0.38 Å out of the plane of the three nitrogen atoms (N2, N3, and N4) towards the chlorine atom. The Cl-Zn-N1 angle is 176.4±0.2° and indicates some of the deviation from true $C_3$ symmetry. It is of interest to note that the N(1)-Zn-N(5) angle reported in Zn(tren)(NCS)(SCN)[7] is 176.8±0.2°. The Zn-Cl distance of 2.308 Å, is well within the range of distances, 2.223 to 2.358 Å, reported in the $[Co(NH_3)_6][ZnCl_4]Cl$ structure.[12]

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(12) D. W. Meek and J. A. Ibers, Inorg. Chem., 9, 465 (1970).

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Some of the dimensions of the tren-Zn cation are shown in Table V. The Cl-Zn-N4 angle is observed to be smaller than the comparable angles for Cl-Zn-N2 and Cl-Zn-N3; this anomaly is probable due to packing conditions. Similar deviations may be observed in the structures of complexes of tren with Cu[5] and Co.[6]

The dimensions of the tetraphenylborate anion are shown in Table VI. Its structure and position relative to one of its cation neighbors is shown in Fig. 2. The molecular packing can be described in terms of a distorted sodium chloride type structure. The positions of the bulky anions are near a cubic-closest-packing based on a pseudo-cubic pseudo-cell with axes $\underset{\sim}{a}$, $\underset{\sim}{b} + \underset{\sim}{c}/2$, $\underset{\sim}{b} - \underset{\sim}{c}/2$. The cations are in octahedral holes in this anion packing, as in the sodium chloride structure, with atoms N1 close to the centers of the holes.

Table VII show the interatomic distances to hydrogen in this structure. The average N-H bond distance is 0.85 Å, and the average C-H distance is 0.95 Å. As is generally the case in X-ray structure determinations involving hydrogen, these distances between the centers of gravity of electron clouds tend to be shorter by about a tenth of an angstrom from the generally accepted values for the internuclear distances.

TABLE V

INTERATOMIC DISTANCES (Å) AND ANGLES (DEG) in the $Zn(C_2H_4NH_2)_3Cl^+$ ion

| Atoms | Dist. | Atoms | Dist. |
|---|---|---|---|
| Zn-Cl | 2.308(5) | N1-C5 | 1.444(8) |
| Zn-N1 | 2.325(7) | N2-C2 | 1.469(8) |
| Zn-N2 | 2.068(7) | N3-C6 | 1.466(7) |
| Zn-N3 | 2.063(7) | N4-C4 | 1.445(7) |
| Zn-N4 | 2.065(6) | C1-C2 | 1.496(8) |
| N1-C1 | 1.498(8) | C3-C4 | 1.478(8) |
| N1-C3 | 1.482(7) | C5-C6 | 1.501(9) |
| | Angle | | Angle |
| Cl-Zn-N1 | 176.4(2) | Zn-N3-C6 | 111.2(4) |
| Cl-Zn-N2 | 102.1(2) | Zn-N4-C4 | 111.4(4) |
| Cl-Zn-N3 | 103.0(3) | N3-C6-C5 | 109.4(5) |
| Cl-Zn-N4 | 97.7(3) | N2-C2-C1 | 111.1(5) |
| N1-Zn-N2 | 79.6(2) | N4-C4-C3 | 110.0(5) |
| N1-Zn-N3 | 79.0(3) | C6-C5-N1 | 111.5(4) |
| N1-Zn-N4 | 78.7(3) | C2-C1-N1 | 108.6(5) |
| N2-Zn-N3 | 113.3(3) | C4-C3-N1 | 111.3(5) |
| N3-Zn-N4 | 118.9(3) | C1-N1-Zn | 106.2(3) |
| N4-Zn-N2 | 117.2(3) | C3-N1-Zn | 105.5(4) |
| Zn-N2-C2 | 108.7(3) | C5-N1-Zn | 106.1(4) |

TABLE VI

INTERATOMIC DISTANCES (Å) AND ANGLES (DEG) IN $(C_6H_5)_4B^-$

| Atoms | Dist. | Atoms | Dist. |
|---|---|---|---|
| B-C11 | 1.646(8) | C32-C33 | 1.406(8) |
| B-C21 | 1.631(9) | C33-C34 | 1.350(8) |
| B-C31 | 1.641(9) | C34-C35 | 1.373(8) |
| B-C41 | 1.641(9) | C35-C36 | 1.389(8) |
| C11-C12 | 1.392(7) | C41-C42 | 1.400(7) |
| C11-C16 | 1.386(8) | C41-C46 | 1.385(8) |
| C12-C13 | 1.373(8) | C42-C43 | 1.380(8) |
| C13-C14 | 1.361(8) | C43-C44 | 1.359(8) |
| C14-C15 | 1.367(8) | C44-C45 | 1.357(8) |
| C15-C16 | 1.383(7) | C45-C46 | 1.392(9) |
| C21-C22 | 1.405(8) | | Angle |
| C21-C26 | 1.382(7) | C11-B-C21 | 102.9(5) |
| C22-C23 | 1.381(8) | C11-B-C31 | 111.3(5) |
| C23-C24 | 1.358(9) | C11-B-C41 | 111.5(5) |
| C24-C25 | 1.369(9) | C21-B-C31 | 115.1(5) |
| C25-C26 | 1.370(8) | C21-B-C41 | 112.1(5) |
| C31-C32 | 1.386(7) | C31-B-C41 | 104.2(4) |
| C31-C36 | 1.399(7) | | |

TABLE VII

HYDROGEN INTERATOMIC DISTANCES[a]

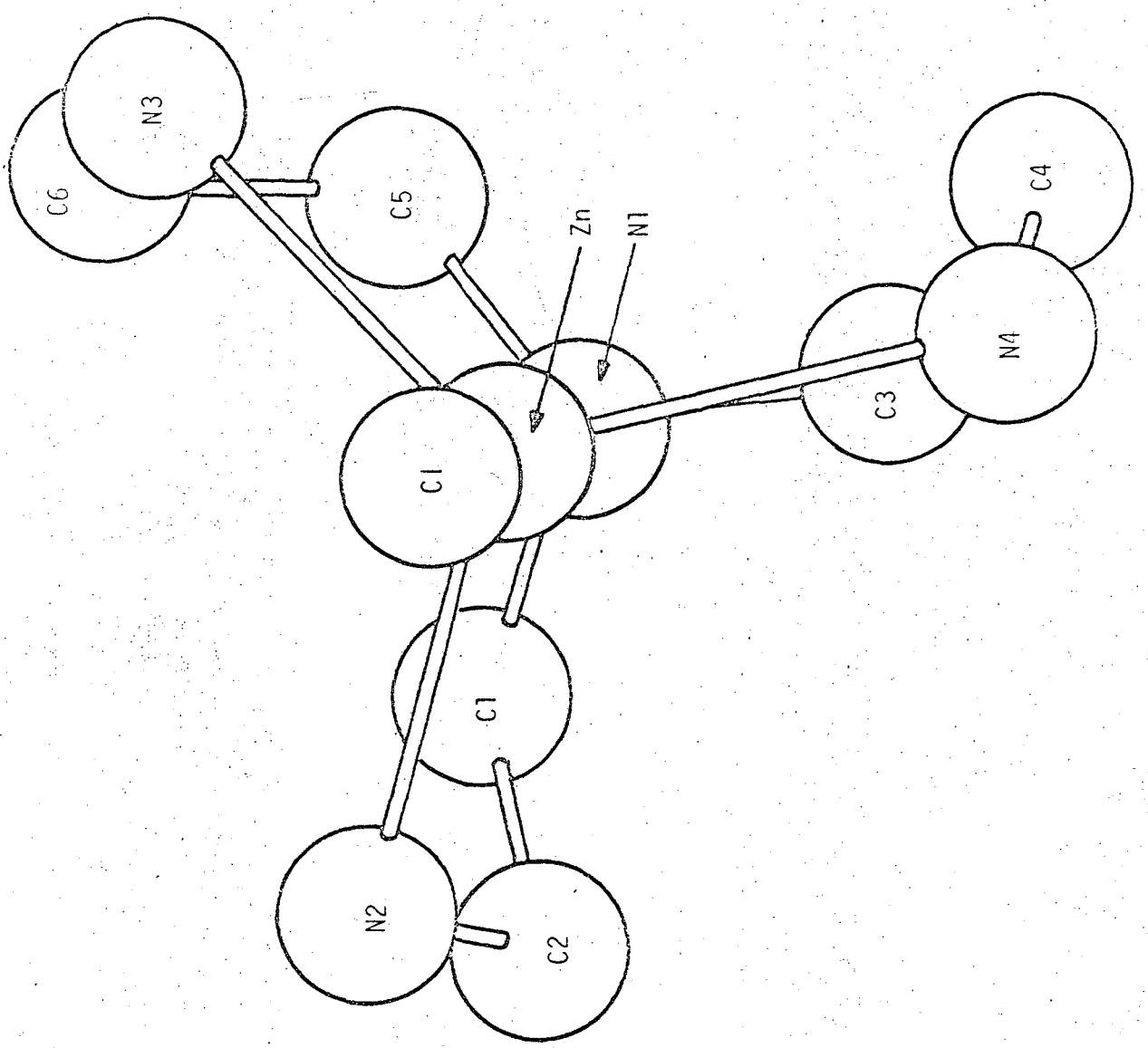
| Atoms | Dist.Å | Atoms | Dist.Å |
|---|---|---|---|
| N2 - H1 | 0.83 | C12 - H | 0.97 |
| - H2 | 0.84 | C13 - H | 0.96 |
| N3 - H1 | 0.90 | C14 - H | 0.98 |
| - H2 | 0.84 | C15 - H | 0.95 |
| N4 - H1 | 0.83 | C16 - H | 0.99 |
| - H2 | 0.84 | C22 - H | 0.91 |
| C1 - H1 | 1.00 | C23 - H | 0.99 |
| - H2 | 1.08 | C24 - H | 0.83 |
| C2 - H1 | 0.86 | C25 - H | 0.93 |
| - H2 | 0.97 | C26 - H | 0.89 |
| C3 - H1 | 0.90 | C32 - H | 0.97 |
| - H2 | 0.93 | C33 - H | 0.93 |
| C4 - H1 | 1.04 | C34 - H | 0.91 |
| - H2 | 1.14 | C35 - H | 0.97 |
| C5 - H1 | 0.93 | C36 - H | 0.87 |
| - H2 | 0.98 | C42 - H | 1.03 |
| C6 - H1 | 0.96 | C43 - H | 1.00 |
| - H2 | 0.92 | C44 - H | 0.91 |
| | | C45 - H | 0.89 |
| | | C46 - H | 0.85 |

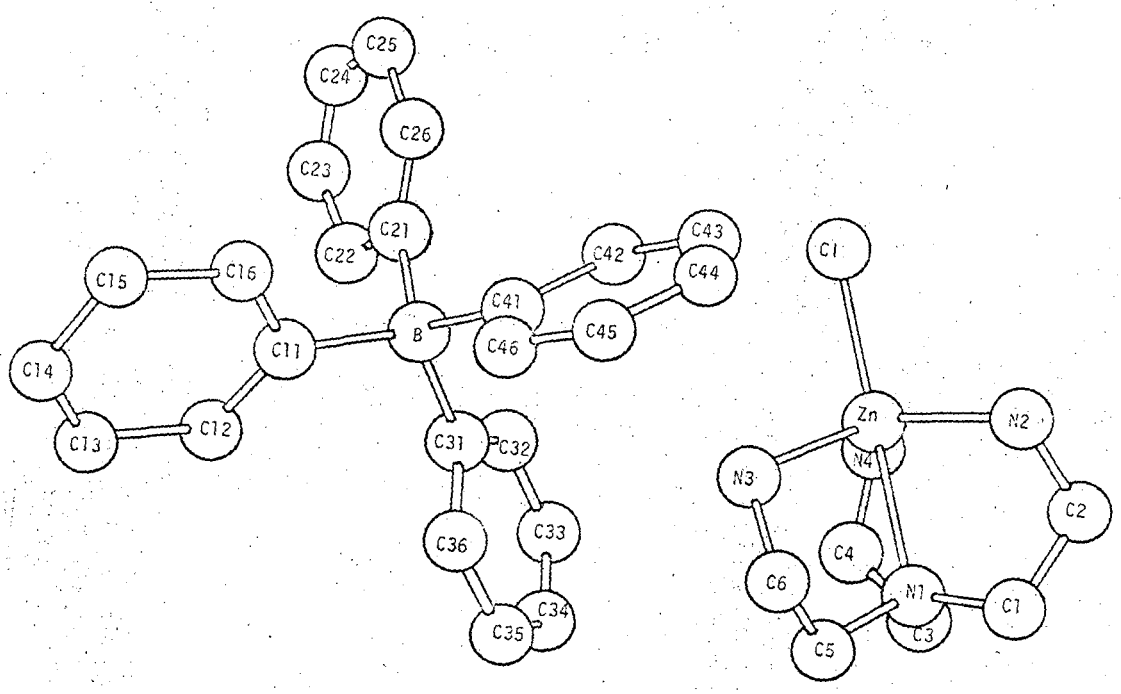
[a]The estimated standard deviations of these distances based on the least squares results are ±0.06 Å.

## Figure Captions

Figure 1: The $Zn(C_2H_4NH_2)_3NC\ell$ cation drawn to indicate the nearly 3-fold nature of the cluster.

Figure 2: Structure of the anion and its relation to one of its cation neighbors.


N3
C6
C5
Zn
N1
C4
N4
C3
C1
C1
N2
C2

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