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THE CRYSTAL STRUCTURE OF TRIS-(2-AMEN0ETHYL) AKCNOCHLOROZINC(II) TETRAPHENYLBORATE

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## THE CRYSTAL STRUCTURE OF TRIS-(2-AMINOETHYL) AMINOCHLOROZINC(II) TETRAPHENYLBORATE

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### UNIVERSITY OF CALIFORNIA

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CONTRIBUTION FROM THE LAWRENCE RADIATION LABORATORY AND DEPARTMENT OF CHEMISTRY, UNIVERSITY OF CALIFORNIA, BERKELEY, CALIFORNIA 94720

The Crystal Structure of Tris-(2-aminoethyl)aminochlorozinc(II) Tetraphenylborate<sup>1</sup> By Rodney J. Sime, Richard P. Dodge, Allan Zalkin, and David H. Templeton

(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 <u>a</u> = 13.76±0.04, <u>b</u> = 10.33±0.03, <u>c</u> = 20.35±0.06 Å, and <u>β</u> = 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  $Zn(C_2H_4NH_2)_3NC\ell$  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.

-1-

### Introduction

-2-

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 Mestren and tren, respectively. In general, these may be formulated as  $[M^{II}(Mestren)X]Y$  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.<sup>2</sup> Because of the increased bulkiness

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

of Me<sub>6</sub>tren, it forms more stable five-coordinated complexes than tren. Me<sub>6</sub>tren complexes have been described, for which M = Mn, Fe, Co, Ni, Cu and Zn, and for which X = Y = Cl, Br, I, NO<sub>3</sub>, and ClO<sub>4</sub>.<sup>3,4</sup> Much physical evidence,

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

(4) M. Ciampolini and N. Nardi, <u>Inorg. Chem.</u>, 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<sup>5</sup> and [Co(Meetren)Br]Br<sup>6</sup> 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, <u>Inorg. Chem.</u>, 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

-3-

(7) G. D. Andreetti, P. C. Jain, and E. C. Lingafelter, J. <u>Am. Chem. Soc.</u>, 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 = C1, Br and I, and Y = X, ZnX<sub>3</sub>, and B(C<sub>6</sub>H<sub>5</sub>)<sub>4</sub>.<sup>8</sup> We report here

(8) L. V. Interrante, <u>Inorg. Chem.</u>, 7, 943 (1968).

the crystal structure of the five-coordinated complex[Zn(C2H4NH2)3NC1]B(C6H5)4.

### Experimental Section

Dr. L. V. Interrante kindly supplied us with some well-formed, colorless, prisms of  $[Zn(tren)C\ell]B(C_{6}H_{5})_{\mu}$ , 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 <u>c</u> 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  $4^{\circ}$  take-off angle to the tube. The maximum 20 angle was  $40^{\circ}$  $[(\sin\theta)/\lambda = 0.596]$ . Background was plotted as a function of 20 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<sup>-1</sup>, and gives an estimated  $\mu$ R~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  $\Sigma_{\underline{w}}(|\underline{\mathbf{F}}_{\underline{o}}| - |\underline{\mathbf{F}}_{\underline{c}}|)^2 / \Sigma_{\underline{w}} \underline{\mathbf{F}}_{\underline{o}}^2$ ;  $\underline{\mathbf{F}}_{\underline{o}}$  and  $\underline{\mathbf{F}}_{\underline{c}}$  are the observed and calculated structure factors respectively, and <u>w</u> 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, <u>J. Chem. Phys.</u>, <u>42</u>, 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) D. T. Cromer, <u>Acta Cryst.</u>, 18, 17 (1965).

were included in the least squares calculations. For each reflection <u>w</u> was set to  $1.0/\sigma^2(\underline{F}_0)$  with the exception that w = 0 when  $I(\text{net count}) \stackrel{\leq}{=} \sigma(I)$ . The standard deviation of the observed structure factor was calculated as  $\sigma(\underline{F}_0) = \underline{F}_0 - (\underline{F}_0^2 - s\sigma(I)/Lp)^{1/2}$  where s is a scaling factor,  $\underline{F}_0 = (sI/Lp)^{1/2}$ , 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; <u>a</u> = 13.76±0.04, <u>b</u> = 10.33±0.03, <u>c</u> = 20.35±0.06 Å, and  $\beta$  = 95.0±0.2°. The extinctions observed were consistent with space group P2<sub>1</sub>/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

-6-

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  $\underline{R} = 0.12$ , where  $\underline{R} = \sum |\Delta \underline{F}| / \sum |\underline{F}_0|$  and  $\Delta \underline{F} = |\underline{F}_0| - |\underline{F}_c|$ . Several more cycles of least-squares calculations with anisotropic thermal parameters reduced  $\underline{R}$  to 0.077. The anisotropic temperature factors have the form  $\exp(-\underline{\beta}_{11}\underline{h}^2 - \underline{\beta}_{22}\underline{k}^2 - \underline{\beta}_{33}\underline{k}^2 - 2\underline{\beta}_{12}\underline{h}\underline{k} - 2\underline{\beta}_{13}\underline{h}\underline{k} - 2\underline{\beta}_{23}\underline{k}\underline{k})$ . In reporting the thermal parameters below we have converted  $\underline{\beta}_{\underline{i},\underline{j}}$  to  $\underline{B}_{\underline{i},\underline{j}}$  which is in units of  $\underline{\lambda}^2$ ; the relation between these two quantities is  $4\underline{\beta}_{\underline{i},\underline{j}} = \underline{a}_{\underline{i}}\underline{a}_{\underline{j}}^{\mathbf{R}}\underline{b}_{\underline{i},\underline{j}}$ , where  $\underline{a}_{\underline{i}}^{\underline{x}}$  is the <u>i</u>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,  $(\Sigma_{\underline{W}}(\Delta \underline{r})^2)/\Sigma_{\underline{W}}\underline{r}^2)^{1/2}$ , 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.

-7-

13

### TABLE I

-8-

## OBSERVED AND CALCULATED STRUCTURE FACTORS FOR

TRIS-(2-AMINOETHYL)AMINOCHLOROZINC(II) TETRAPHENYLBORATE

 $\frac{a}{2}$ Zero weighted data are indicated with asterisks.

UNSERVED AND CALCULATED STRUCTURE FACTORS OF TRIS-IZ-ARINOETHVLJAMINOCHLOROZINCITIJTETRAPHENVLBORATE. FCATO.0.01 = 4768	
H FOB FCA 0 189 167 5 247 252 -10 0 4* -6 112 114 -3 222 224 -3 398 395 -3 320 316 3 131 106 -7 60 60 1 85 66 2 0 38* 0 29 290 -6 0 K 1= 0, 0 1 180 175 6 20 726 -9 150 153 -5 220 217 -2 114 103 -3 44 355 -2 64 69 4 52 42 -6 217 238 2 78 78 17 3 35* 134 -33 -5 47 1 371 356 2 13* 136 7 221 228 -8 100 97 -4 227 234 -1 207 211 -1 66 66 -1 179 175 4.4 -3, 18 -4 20 223 3 0 -5 4 123 228 2 185 189 -4 40 2 6 75 6 19 3 68 8 41 40 -1 49 427 234 -1 207 211 -1 66 66 -1 179 175 4.4 -3, 18 -4 20 223 3 0 -5 4 123 228 2 185 189 -4 40 2 6 75 6 11 3 68 8 41 40 -1 49 417 -2 3 70 0 143 154 0 239 254 -1 207 211 -2 156 18 -2 128 127 -2 18 128 -2 14 -2 128 128 -2 148 189 -4 40 -1 24 -2 20 -2 138 -1 40 -1 40 -1 40 -1 40 -1 40 -1 24 -2 -2 0 -2 -2 -2 -2 -2 -2 -2 -2 -2 -2 -2 -2 -2	$\begin{array}{cccccccccccccccccccccccccccccccccccc$
4 272 275 5 114 101 10 230 235 -5 126 179 -1 31 42 2 140 153 2 435 455 2 159 163 -2 29 204 -2 186 194 -4 30 194 7 46 36 5 81 84 -1 26 5 107 104 6 39 16 11 39 30 -4 116 127 0 236 255 3 144 157 3 214 217 327 260 -14 14 -1 78 71 -3 41 45 88 91 6 118 114 0 27 6 209 214 7 711 209 12 29 314 -3 127 119 1 313 312 4 3100 314 4 37 28 4 210 215 0 83 76 0 119 128 -2 105 108 92 11* 7 114 101 1 7 7 700 194 8 114 107 41 - 1 0 -2 381 374 2 275 279 5 0 9 5 140 183 0 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5	7* -8 124 123 1 611 63 218 -7 51 53 2 93 81 75 -6 28 32* 5 109 104 39 -5 89 90 4 208 204
n     54     57     λ <sub>1</sub> k     0, 16     -12     155     160     -11     160     18     187     -10     181     167     -10     181     187     -10     181     187     -10     181     187     -10     181     187     -10     181     131     116     161     -10     181     181     131     116     161 </td <td>52" -4 46 50 5 /5 78 42 -3 188 187 6 24 14" 24" -2 155 159 / 60 51 /2 -1 95 96 Kal* 8, 5 98 0 36 23 -7 42 33</td>	52" -4 46 50 5 /5 78 42 -3 188 187 6 24 14" 24" -2 155 159 / 60 51 /2 -1 95 96 Kal* 8, 5 98 0 36 23 -7 42 33
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-11 311 320 0 118 115 -3 152 155 8 8 175 175 12 33 130 -8 53 47 -11 68 80 -8 45 33 7 76 73 5.5 48 6 24 188 -1 130 126 -4 41 37 -5 121 -10 123 117 1 155 169 -2 206 193 4 0 15× 8.5 3 47 -10 46 64 -71 10 112 81 44 73 7 125 124 0 415 42 -3 7 45 124 -9 52 70 2 54 57 -1 30 240 8.5 1, 14 -12 0 114 -8 254 261 -9 93 162 -6 158 159 9 45 45 -10 65 67 8 275 275 1 227 227 -2 207 217 -3 107 -9 77 77 1 10 127 0 788 71 -9 50 65 -11 0 6 6 -5 261 229 -8 47 65 -5 39 435 10 0 78 -9 27 78 9 135 149 -10 51 67 5 -2 20 72 7 -2 207 217 -3 107	128 5 0 120 -2 27 380 31 6 93 96 -1 27 260 194 7 100 97 0 30 20 299 8 84 68 1 27 110
-7 224 232 4 0 149 1103 84 -8 86 87 -10 26 78 -4 120 120 -7 135 160 -4 66 79 11 57 6M -8 26 19 10 28 3* 3 140 140 0 0 12 78* -1 130 150 -7 0 85 10 -1 0 12 110 120 137 -1 0 130 -1 0 13 140 140 0 1 12 110 120 137 -1 0 130 -1 0 13 140 140 0 1 12 110 140 140 0 137 -5 120 140 140 140 140 140 0 15 -5 140 140 140 0 15 -5 140 140 140 0 15 -5 140 140 140 0 15 -5 140 140 140 0 15 -5 140 140 140 0 15 -5 140 140 140 0 15 -5 140 140 140 0 15 -5 140 140 140 0 15 -5 140 140 140 0 15 -5 140 140 140 140 140 140 140 140 140 140	153 R,L= 7, 7 2 47 44 9 -8 88 41 3 67 72 98 -7 29 294 4 43 48 159 -6 62 63 5 0 174 80 -5 141 137 6 59 49
1 (102 49 -6 30 78* 5 (49 (56 -4 ))5 (27 -5 )7 (37 (2 80 94 - 3 80 74 (2	92 -4 38 22 K+1* 9, 6 30 -3 139 135 -7 84 68 64 -2 45 33 -6 29 5* 12 -1 191 195 -5 49 35
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6 115 128 3 59 66 - 12 166 164 5 91 91 5 200 235 8.1. 7 100 6 748 252 10 68 90 - 4 68 77 5 43 31 - 1 31 306 - 4 65 65 - 4 27 118 - 2 107 70 211 4 119 110 - 11 188 164 6 115 164 5 95 556 - 10 57 10 410 11 5 10 10 10 10 - 1 10 10 10 10 - 1 10 10 10 10 - 1 10 10 10 10 - 1 10 10 10 10 - 1 10 10 10 10 10 10 10 10 10 10 10 10 1	99 5 46 89 1 27 36* 121 6 145 146 2 110 107 122 7 59 56 3 62 59 5* K,L* 7, 8 4 106 111
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-14 30 27* 7 106 108 -3 240 237 -5 33 60 12 78 62 -3 0 6* 11 39 11 -5 17 201 7 170 13 -0 166 108 5 3 77 4 6 6 5 16 5 0 169 20 10 - -12 89 76 8 0 14* -2 585 561 -4 128 12* 15* -2 272 268 -10 87 78 -16 33 4 6 74 27* -164 140 10 5 16 5 74 5 40 16 -11 187 195 5 5 6 4* -1 95 65 -3 24* 242 -12 0 14* -1 11 01 8* 0 150 1-2 22 28 4 9 10 150 29* -5 86 11 12 9 15* 0 25* -2 16 70 7* -5 10 -10 281 271 1 29 10 12 3 0 12* 12* 12* 12* 12* 12* 13* 13* 13* 13* 13* 13* 10* 10* 10* 10* 10* 10* 10* 10* 10* 10	, 13 -3 116 113 -4 28 30* 33* -2 26 1* -1 48 5H 35 -1 120 114 -2 104 105 73 0 141 140 -1 34 25
-s 114 116 12 40 56 2 446 446 0 163 153 -4 005 105 2 (47 156 -6 27 1)7 0 16 42 12 0 59 -1 169 168 -1 12 9 59 9 30 219 5 69 69 -1 122 -7 21 119 13 0 29 3265 276 1 169 165 -6 121 120 1 102 1102 116 168 166 1 311 319 4 (4 4 2 -2 165 165 -1 0 9 7 9 3 (4 -5 1) 0 5 234 233 -7 40 -6 207 71 4 (4 1 , 1 4 28 10 2 37 30 -7 88 64 4 0 49 -4 28 18 2 300 307 -1 2 42 50 -1 144 137 -0 164 164 -9 29 199 7 109 105 -1 109 -5 46 45 -1 3 30 316 5 81 8 5 3105 -6 248 254 5 45 36 -1 101 97 3 (16 164 -1 40 5) 0 58 9 -6 26 163 -1 123 145	119         1169         167         0         73         79           31         2         75         76         1         0         10*           100         3         34         2         0         3*           157         4         87         88         3         40         3*
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0 100 101 - 7 201 240 11 100 1 100 2 2 2 2 - 10 42 25 32 102 2 3 2 2 2 5 - 1 10 42 25 32 0 2 0 2 4 4 - 5 11 11 6 4 206 20 3 - 2 151 151 - 2 206 200 - 8 27 159 74.1 6 2 200 201 - 2 151 2 2 2 2 2 2 1 0 - 2 1 2 1 2 2 - 1 0 5 1 1 3 1 1 0 - 2 6 4 3 3 1 3 4 5 0 2 1 0 - 1 4 - 2 2 0 2 0 0 - 8 2 - 2 1 2 1 2 3 - 7 3 4 - 7 3	+ 14 -6 95 87 -4 86 85 i 76 -5 166 163 -3 70 68 i 254 -4 55 58 -7 124 136 i L13 -3 180 182 -1 49 38
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-12 [12] 12] 577 262 -1 60 47 5 41 34 Kr(t 2, 6 3 21] 206 -10 65 67 0 72 73 6 75 15 -7 0 17 11 66 66 -6 79 32 5 0 8 -11 16 -11 15 15 -7 8 17 1 16 -10 67 11 16 -10 67 11 16 -10 67 11 16 -11 16 -11 16 -11 16 -11 16 -11 16 -11 16 -11 16 -11 16 -11 16 -11 16 -11 16 -11 16 -11 16 -11 16 -11 16 -11 16 -11 16 -10 16 -10 -10 -10 -10 -10 -10 -10 -10 -10 -10	114 x,1+ 7, 10 -3 24 25* 82 -7 H4 85 -2 57 58 7* -6 0 14* -1 2H 25* 71 -5 81 75 0 75 64 0 -5 40 34 1 0 3*
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-3 38 42 -13 0 94 5 84 78 -12 48 246 -4 292 297 -7 78 274 -1 45 40 9 29 344 -6 92 81 7 95 96 -4 0 124 1 160 165 -7 70 71 5 76 -3 38 42 -12 0 320 9 38 21 0 27 105 -8 79 94 0 43 35 K:14 3, 12 -7 24 224 28 18 -28 48 49 2 76 94 -0 118 117 -0 15 37 -12 48 24 -12 0 27 10 71 5 76 -3 14 30 1 -8 79 94 0 43 35 K:14 3, 12 -7 24 224 28 18 -28 48 49 2 76 94 -0 118 117 -0 15 13 -6 20 129 9 -9 0 43 37 K; 17 -5 45 40 7 14 1 -2 8 30 1 -4 71 78 -7 10 71 5 76 -21 10 10 10 2 10 28 30 1 -4 74 18 -11 -2 87 86 -5 157 152 1 143 147 -10 51 53 -6 20 129 9 9 0 -4 3 78 77 -5 43 40 7 14 1 -2 87 86 -3 157 152 1 143 147 -10 51 53 -6 20 129 9 9 0 -4 8 -2 18 -17 -6 75 74 6 0 7 -4 6 -4 6 4 6 8 18 6 -2 18 -17 -10 71 12 -12 12 12 -12 13 12 -1	26* 1 48 43 -4 89 86 43 2-28 13* -3 152 151 -124 3 0 6* -2 0 5* 144 4 64 60 -1 0 23* 44 5 63 72 0 14 130
1 775 766 -6 149 156 -12 70 70 4 50 56 1 147 105 -2 150 126 4 16 31 -7 146 143 -5 402 402 -9 137 128 1 21 164 6 28 79 -2 240 219 K.L.* 7. 7 23 21 -7 83 86 -11 49 5 K.L. 8 7 2175 176 -1 25 25 9 5 173 181 - 6 238 237 -2 138 134 - 8 173 173 2 150 185 7 55 55 -1 212 211 -9 7 3 18 321 -5 190 179 -10 94 95 K.L. 1, 16 3 222 335 0 141 141 6 182 185 -5 159 136 -1 192 197 -7 35 46 3 184 183 8 0 155 -5 159 135 -8 70 4 33 29 -5 127 118 -9 127 125 -6 30 179 4 120 118 1 79 83 7 13 80 -4 9 51 0 197 201 -6 85 79 9 5 K.L. 9 157 125 118 -7 159	, t «,L= 7, t1 1 29 4te 32e -6 30 29e 2 88 86 74 -5 29 t5e 3 136 133 34 -4 103 97 K,L= 8, 11
5 26 247 - 4 276 274 - 78 193 148 - 5 31 49 5 21 199 7 276 68 69 49 - 2 194 159 1 76 7 2 - 4 4 151 5 1 5 19 19 - 6 0 22* 2 17 177 - 5 12 5 6 270 274 - 3 571 150 - 7 0 119 - 4 0 159 6 310 319 3 56 69 0 58 57 - 3 10 130 2 27 22* - 14 131 5 0 4 58 57 - 3 10 130 - 7 0 14 41 6 4 58 57 - 6 10 58 57 - 3 50 150 - 7 0 14 41 6 4 58 57 - 6 10 58 57 - 3 50 150 - 10 128 - 9 - 3 0 - 3 0 128 - 9 - 3 0 12	132         -3         64         65         -1         57           48         -2         28         29*         -2         30         30*           249         -1         93         90         -1         51         57           139         0         112         102         0         59         68           58         1         89         100         1         42         24
10 253 247 1 66 69 -3 306 296 0 49 44 10 102 97 7 77 76 K-1 5, 5 2 42 55 6 274 275 0 244 220 10 29 74 -3 201 207 7 48 93 -1 100 11 40 2 102 296 -2 461 471 1 0 209 11 41 65 8 73 10 -12 83 74 3231 235 71 719 187 L 215 215 11 30 459 -2 169 177 120 194 0 230 12 136 129 3 191 144 -1 201 196 2 82 92 K-1 2, 7 K-1 2, 15 -11 97 106 4 29 255 8 43 27 2 129 127 4, 5, 4 -1 78 88 9 157 150 11 14 0 2 10 2 10 2 10 2 10 1 1 1 1 1 1 1 1 1	101         2         133         136         K+L=         9,         0           227         3         50         47         1         28         19*           111         4         41         37         2         8         4*           130         5         30         13*         3         99         100
-12 109 49 5 29 29 29 119 201 4 24 1, 19 -11 48 41 - 4 70 5 3 - 4 2 5 2 4 5 5 4 5 5 4 5 10 5 4 7 4 7 10 22 27 1 22 20 -13 80 5 3 5 3 5 1 - 2 10 20 20 - 2 10 10 5 1 - 2 10 20 20 - 2 10 10 5 1 - 2 10 20 20 - 2 10 10 10 20 2 - 2 10 10 10 10 - 2 10 20 20 - 2 10 10 10 10 - 2 10 20 20 - 2 10 10 10 10 - 2 10 20 20 - 2 10 10 10 - 2 10 20 20 - 2 10 10 10 - 2 10 20 20 - 2 10 10 10 - 2 10 20 20 - 2 10 10 10 - 2 10 20 20 - 2 10 10 10 - 2 10 20 20 - 2 10 10 10 - 2 10 20 20 - 2 10 10 10 - 2 10 20 20 - 2 10 10 10 - 2 10 20 20 - 2 10 10 10 - 2 10 20 20 - 2 10 10 10 - 2 10 20 20 - 2 10 10 10 - 2 10 20 20 - 2 10 10 10 - 2 10 20 20 20 - 2 10 10 10 - 2 10 20 20 20 - 2 10 10 10 - 2 10 20 20 20 - 2 10 10 10 - 2 10 20 20 20 - 2 10 10 10 - 2 10 20 20 20 - 2 10 10 10 - 2 10 20 20 20 - 2 10 10 10 - 2 10 20 20 20 - 2 10 10 10 - 2 10 20 20 20 - 2 10 10 10 - 2 10 20 20 20 - 2 10 10 10 - 2 10 20 20 20 - 2 10 10 10 - 2 10 20 20 20 - 2 10 10 10 - 2 10 20 20 20 - 2 10 10 10 - 2 10 20 20 20 - 2 10 10 10 - 2 10 20 20 20 - 2 10 10 10 - 2 10 20 20 20 - 2 10 10 10 - 2 10 20 20 20 - 2 10 10 10 - 2 10 20 20 20 - 2 10 10 10 - 2 10 20 20 20 - 2 10 10 10 - 2 10 20 20 20 - 2 10 10 - 2 10 20 20 - 2 10 10 - 2 10 20 20 - 2 10 10 - 2 10 20 20 - 2 10 10 - 2 10 20 20 -	175 -5. 0 204 5 42 40 161 -4 51 72 K,L* 9, 1 270 -3 104 105 -5 51 60 41 -2 76 76 -4 217 218
-7 262 262 10 44 43 6 171 174 0 42 52 -6 252 252 -7 2 90 85 -4 124 117 -8 44 46 -10 72 83 kit 4, 12 -5 154 151 6 125 120 -5 71 47 8 57 -6 65 67 11 77 21 17 7 8 47 46 -10 72 83 kit 4, 12 -5 154 151 6 125 120 -5 71 47 8 57 -6 30 57 -5 12 7 21 21 22 -5 71 57 156 159 7 30 -5 20 227 12 107 107 8 66 66 Kit 7, 0 -4 53 027 7 1 111 113 -6 64 7 158 -6 71 7 166 168 -7 27 6 -7 33 7 16 7 16 16 7 16 7 16 7 16 7 16 7	62 -1 57 45 -3 107 106 24* 0 70 45 -2 75 72 , 2 1 119 117 -1 49 54 96 2 87 84 0 28 23* 44 3 70 84 0 21 23*
-2 575 574 -12 132 123 11 107 46 2 502 29 -1 37 55 3 6 56 5 1 18 250 -3 25 108 -5 144 153 -5 37 22 0 265 277 -6 0 94 0 127 150 -7 28 -1 478 507 -11 55 154 64 4 1 0 3 181 371 0 67 56 4 67 56 2 18 159 -2 196 101 -4 29 26 -4 81 78 1 100 104 -5 88 81 1 7 4 244 -6 14 0 (48 169 -10 67 50 -11 56 156 4 87 274 1 73 76 5 74 62 3 353 362 -1 82 82 -3 88 67 -3 71 89 2 212 216 -4 72 73 2 134 132 -5 119 1 476 66 -9 107 313 -10 0 124 53 8 302 2 255 261 0 71 62 4 293 299 0 0 46 -7 156 138 -2 129 118 3 6 61 -3 54 50 0 15 -5 15 1 476 466 -9 107 313 -10 0 124 53 36 302 2 255 261 0 71 62 4 293 299 0 0 46 -7 156 138 -2 129 118 36 61 -3 54 50 0 155 -5 175	22* 4 0 37* 2 113 111 145 K,L* 7, 13 3 131 132 116 -4 52 58 4 50 37 170 -3 0 3* 5 126 130
2 237 214 - # 401 411	257 -2 97 98 K,L= 9, 2 51 -1 29 34* -5 30 22* 195 0 29 27* -4 71 66 249 1 29 34* -3 29 18* 184 -3 139 145 -2 70 64
7 145 142 - 355 546 - 4 272 266 11 27 309 6 94 89 -5 39 30 10 82 82 6 6 87 97 6 124 125 6 6 26 299 6 176 176 1 0 118 9 38 87 2 101 6 337 331 -2 913 99 - 3 10 119 12 0 89 9 0 154 -4 55 37 11 29 278 7 0 318 9 31 28 57 178 0129 123 4 0 108 142 6 9 9 102 -1 97 91 -2 65 37 14 2, 1 10 104 92 -3 81 76 14 29 278 13 8 126 124 6 39 27 14 10 129 12 4 4 0 108 14 9 10 56 57 0 66 43 -12 92 17 12 12 12 12 13 10 6 92 -3 81 76 14 8 15 15 15 15 15 15 16 0 6 42 56 -8 17 18 15 15 10	100 K.L. 8, 0 -1 0 14* 27 0 133 141 0 28 14* 109 1 83 75 1 78 4* 4* 2 109 120 2 70 79
11 (13) 112 1 25 29 0 (65) 166 -11 77 79 K-( $z$ 2, 6 -1 )39 (3) -11 40 31 -3 0 0 * 6 31 4* 6 0 (4* -10) 46 (10 K-( $z$ 3* 6 -7 0 0 * 6 12 0 K-( $z$ 3* 6 -7 0 0 * 6 12 0 + 0 + 0 + 0 + 0 + 0 + 0 + 0 + 0 + 0	$\begin{array}{cccccccccccccccccccccccccccccccccccc$
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	50 K,L= 8, L -4 47 103 83 -7 24 L1 -3 41 39 100 -6 28 L3 -2 148 L43 255 -5 96 96 -1 28 20 138 -4 0 3 41 83 76
	40 -3 113 112 1 40 42 45 -2 46 58 2 57 56 137 -1 0 8+ 3 24 34+ 74 0 52 10 4 117 125
1 con eco -12 0 1 co -1173 180 3 266 260 1 173 180 -1 50 47 3 238 231 5 46 102 -7 260 264 3 161 158 4 56 50 K, K 5, 15 7 214 203 0 1 40 7 183 181 -11 38 3 -6 110 100 4 258 264 2 345 349 0 48 51 4 555 458 6 6 50 -1 181 158 6 4 56 50 K, K 5, 15 7 214 203 0 1 4 7 2 28 233 -10 25 7 -7 8 3 85 5 268 255 3 149 149 1 55 53 5 47 485 7 0 24* 0 64 55 5 39 43 6 75 79 -5 113 115 K, L 6, 8 2 25 8 0 14* - 2 5, 16* -6 0 7 7* 6 3 7 5 4 209 216 2 0 1 1 6 123 159 4 55 53 5 47 485 7 0 24* 0 64 55 5 39 43 6 75 79 -5 113 115 K, L 6, 8 2 25 8 0 14* - 2 5, 16* -6 10 7* 6 3 7 55 4 209 216 2 0 11* 6 123 159 4 53 55 3 57 50 68 66 7 7 2 159 -10 20 55* -6 18 25 8 0 14* - 2 5, 16* -6 10 7* 6 3 7 55 4 209 216 2 0 11* 6 123 159 4 50 53 7 507 68 6 6 6 7 7 9 159 159 -10 20 55* -6 18 75 7 50 7 55* -6 18 75* 7 50 7 55* -6 18 75* 7 50 7 55* -6 18 75* 7 50 7 55* -6 18 75* 7 50 7 55* -6 18 75* 7 50 7 55* -6 18 75* 7 50 7 55* -6 18 75* 7 50* 7	137 1 69 61 5 140 134 211 2 26 78 4,14 9, 4 9* 3 0 16* -5 73 77 61 4 27 12* -4 74 15* 10* 5 48 37 -5 71 72
6 20 232 -7 56 54 -4 332 345 8 86 92 6 47 44 4 (49 159 8 80 82 -7 86 81 3 174 176 4 (1 - 4, 1 + 9 4 8 16 -2 28 314 -7 0 184 5 37 7 174 165 -6 20 6 -1 167 164 9 0 1 49 7 76 78 4 4 (4 - 5 0 7 7 6 94 7 3 + 18 116 - 16 5 15 1 10 56 64 - 1 7 7 7 6 (6 0 54 6 0 8 1/72 165 -5 75 72 -2 369 368 10 6 3 65 8 77 76 -5 59 52 10 116 114 -5 0 7 5 245 245 -7 71 77 6 (1 - 5 7 7 6 ( 9 83 61 -4 205 262 -1 282 275 1 1 67 64 9 85 80 -4 0 1 79 11 9 97 -4 5 59 52 10 116 1 14 -5 0 7 5 58 54 5 -7 71 77 6 (1 - 5 7 7 6 -5 26 7 4 7 6 5 -5 1 -2 10 2 7 6 -5 1 -2 10 2 7 -1 -2 10 2 7 -1 -2 10 -2	46 6 64 58 -2 41 27 21* 7 0 10* -1 40 47 56 641* 8, 2 6 70 57 108 -7 47 97 1 0 25*
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	234 - 5 198 186 3 71 71 26 -4 133 132 4 () 184 142 -3 54 42 K,L= 9, 5 17 - 2 102 107 -4 118 121
B 121 116 2 116 136 3 86 93 -9 0 59 -9 65 55 2 121 121 -8149154 2 27 139 5,16 4 5 0 37 42 -5 79 52 -3 135 138 2 112 116 -5 64 -7 105 100 3 110 109 6 263 263 -8 318 312 -8 132 116 3 42 14 -7 176 183 3 9 32 -11 29 214 1 0 289 -4 56 62 -2 157 159 3 25 25 4 6 -6 95 95 4 82 81 7 170 180 -7 143 166 -7 127 129 5,16 2 4 7 175 183 3 9 32 -10 28 7 2 28 39 -3 208 200 -1 101 102 4 52 53 -1 5 -5 170 165 5 19.188 8 9 95 39 -159 109 -7 127 129 5,16 -7 128 318 9 -5 328 318 9 49 65 7 7 -2 38 39 -3 208 200 -1 101 102 4 52 53 -1 56	65 -1 79 85 -3 117 113 14 0 69 68 -2 74 37+ 50 1 64 70 -1 0 9 116 7 150 155 0 61 87 124 3 0 134 13 23 70+
	110         4         86         82         2         92         88           60         5         128         129         3         110         110           1/*         6         143         139         4         315         114           92         7         0         1*         K+L+         4         6
1 hol 116 11 0 13* -→ 39 17 0 144 138 0 225 221 3 51 44 1200 204 -4 0 7* -> 181 181 -6 58 50 4 134 137 2 12 14 -8 58 58 4 58 2 10 121 24 14 58 158 -1 1229 24 4 468 157 2 17 16 150 -5 58 94 5 151 17 14 3 105 110 -7 0 15* 0	72 Kil+ 8, 3 -4 30 25* 8 -7 59 56 -3 29 21* 53 -6 29 7* -2 3 11* 8 -5 48 47 -1 0 7*
5 c 1 s c c 1 m	5 -3 27 23* L 0 2* 33* -2 27 28* 2 42 43 94 -1 59 53 3 84 77 9* 0 75 71 F.L* 9* 7
K <sub>1</sub> LE 0, L46 211 215 Ú 0 274 9 (44) L42 9 62 58 12 58 67 10 28 394 5129 1270 6 115 116 118 -6 16 59 59 K <sub>1</sub> LE 6, 1 1 67 173 -6 27 -10 52 61 -5 0 2 4 64 51 10 0 44 10 0 154 K <sub>1</sub> LE 2, 10 -12 58 49 K <sub>1</sub> LE 3, 11 8 7 7 0 80 0 44 121 -8 97 92 -10 77 60 177 60 2 49 152 -5 20 -9 76 64 -4 662 260 2 60 88 11 106 105 K <sub>1</sub> LE 2, 10 -12 58 49 K <sub>1</sub> LE 3, 8 -6 30 44 8 32 69 3 78 81 -7 78 87 -9 166 164 3 49 42 -4 133 -8 93 97 -3 110 107 3 116 169 112 0 89 -11 77 76 -11 28 20 -11 0 154 -5 163 149 97 69 52 69 -5 71 -8 52 17 -9 166 164 3 49 42 -4 133	18+ 1 43 53 -3 89 84 205 2 0 6+ -2 93 95 191 3 54 51 -1 42 46 203 4 0 19+ 0 29 21+ 52 5 132 133 1 20 14+
	15 6 41 43 2 149 149 193 7 30 3* K.L. 9, 8 72 K.L. 8, 4 -2 30 16* 95 -7 118 115 -1 30 25*
-7 137 333 3 70 74 9 51 60 -6 119 120 -5 116 120 -5 20 60 -5 77 50 1 20 240 -9 163 158 -1 40 26 0 39 35 -2 240 299 -8 59 51 3 110 -1 270 272 4 256 258 Krt. 1, 13 -7 251 250 -4 117 110 -4 317 336 -4 57 65 2 129 123 -8 63 50 0 40 23 1 39 47 -1 123 121 -7 15 64 4 186	LLF -0 180 180 0 52 45 179 -5 148 146 1 47 41 X81 6812 6285

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### TABLE II

-10-

## POSITIONAL PARAMETERS OF THE NON-HYDROGEN ATOMS

<sup>a</sup>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	.04761	1)	1178(	1)	.08697	(7)
NI	.2522(	3)	.2384(	4)	.1078(	2)
N2	.1915(	3)	.05691	5)	.1992(	2)
N3	.26611	3)	00021	4)	.0443(	2)
N4	.0580(	3)	.1967(	5)	.06131	2)
C1	.2893(	4)	.2481(	6)	.1790(	3)
C2	.2151(	4)	.19021	7)	.2199(	3)
C3	.1880(	5)	.3479(	6).	.08581	3)
C4	.1093(	4)	.30571	6)	.0363(	3)
C5	.32991	4)	.2155(	6)	.06601	3)
C6	.3548(	4)	.07431	6).	.0631(	3)
В	.7365(	4)	.28741	6)	.1185(	3)
C11	.66211	3)	.35731	5)	.1667(	2.)
C12	.62801	4)	.2996(	5)	.2221(	3)
C13	.56931	4)	.3643(	6)	.26231	3)
C14	.5403(	4)	.4883(	6)	.24921	3)
C15	.5740(	.4)	.5499(	6).	.1962(	3)
C16	.63431	4)	.4851(	6)	.15641	3)
C21	.8424(	4)	.34761	5)	.1446(	3)
C22	.89381	4)	.3038(	5)	.2031(	3)
C23	.9776(	5)	.36221	8)	.2312(	3)
C24	1.0104(	4)	.4718(	8)	.2038(	4)
C25 ·	.9639(	5)	.51921	6)	.1466(	. 4)
C26	.8821(	4)	.45761	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	.70571	5)	14471	6)	.1172(	3)
C35	.6251(	4)	0659(	7)	.1119(	3)
C36	.6381(	4)	.0673(	6)	.1142(	2)
C41	.7065(	4)	.3208(	5)	.04061	2)
C42	.77261	4)	.30921	5)	00761	3)
C43	.7460(	5)	.3247(	5)	0742(	3)
C44	.6522(	6)	.3544(	6)	0949(	3)
C45	.58391	4)	.3684(	7)	0512(	3)
C46	.6120(	4)	.3487(	6)	.0155(	3)

## TABLE III

-12-

## ANISOTROPIC THERMAL PARAMETERS<sup>a</sup>

 $\overset{a}{-}$ Units of these parameters are  $\overset{a}{A}^2$  as described in the text.

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• .			•		· .	· .		
			· ·	· · · ·				• 
		· · ·						
	ATOM	B11	B22	B33	B12	813	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( 1)	
	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)	
	C 1	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)	
	<b>C</b> 6	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)	ti de
	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)	
	<b>C</b> 24	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)	1.5
	C 31	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)	
	C 33	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)	
	C 35	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)	

UCRL-19941

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## TABLE IV

-14-

POSITIONAL AND THERMAL PARAMETERS OF THE HYDROGEN ATOMS A

 $\frac{a}{Units}$  of B are  $\mathbb{A}^2$ .

ATOM X	Y	2	E
H1 N2 .1407(45)	.0337(63)	.2152(30)	1.1(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)
HI 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	.5363(45)	.1200(23)	3.8(13)
'H C22	.2291(38)	.2179(17)	.3(10)
H C23 1.0039(39)		.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)	• 04 08 ( 22 )	2.5(13)

-15-

-16-

## 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  $176.4\pm0.2^{\circ}$  and indicates some of the deviation from true C<sub>3</sub> symmetry. It is of interest to note that the N(1)-Zn-N(5) angle reported in Zn(tren)(NCS)(SCN)<sup>7</sup> is  $176.8\pm0.2^{\circ}$ . 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.<sup>12</sup>

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

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 a, b + c/2,

b - c/2. The cations are in octahedral holes in this anion packing, as in the sodium chloride structure, with atoms Nl 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.

-17-

INTERATOMIC DISTANCES (A) AND ANGLES (DEG) in the  $Zn(C_2H_4NH_2)_3C\ell^+$  ion

			· · · · · · · · · · · · · · · · · · ·
Atoms	Dist.	Atoms	Dist.
Zn-Cł	2.308(5)	N1-C5	1.444(8)
Zn-N1	2.325(7)	N2-C2	1.469(8)
Zn-N2	2.068(7)	N <b>3-</b> C6	1,466(7)
Zn-N3	2.063(7)	N4-C4	1.445(7)
Zn-N4	2.065(6)	Ø1-C2	1.496(8)
N1-C1	1,498(8)	C <b>3</b> -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)
N7-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( <b>8</b> )
N4-Zn-N2	117.2(3)	C3-N1-Zn	105.5(4)
Zn-N2-C2	108.7(3)	C5-N1-Zn	106.1(4)
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#### -19-

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INTERATOMIC DISTANCES	(Å)	AND	ANGLES	(DEG)	IN	(C,H,	),B
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Atoms	Dist.	Atoms	Dist.
B-C11	1.646(8)	C32-C33	1.406(8)
BC21	1.631(9)	C3 <b>3-C3</b> 4	1.350(8)
B-C31	1.641(9)	C34-C35	1.373(8)
BC41	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)
022-023	1.381(8)	C11-B-C31	111.3(5)
C23-C24	1.358(9)	C11-B-C41	111.5(5)
024-025	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)
031-036	1 300(7)		

### TABLE VII

-20-

#### HYDROGEN INTERATOMIC DISTANCES Dist.A Atoms Dist.A Atoms N2 - H1 0.83 C12 - H 0.97 - H2 0.84 C13 - H 0.96 N3 - H1 0.90 C14 - H0.98 - 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 0.89 С26 - Н C3 - H1 0.90 C32 - H0.97 – H2 0.93 C33 - H 0.93 C4 - H1 1.04 С34 - Н 0.91 - H2 1.14 C35 - H 0.97 C5 - H1 0.93 С36 – Н 0.87 - H2 0.98 C42 - H 1.03 C6 - H1 0.96 C43 - H 1.00 - H2 0.92 С44 – Н 0.91 C45 - H 0.89 С46 - Н 0.85

<sup>a</sup>The estimated standard deviations of these distances based on the least squares results are  $\pm 0.06$  Å.

### Figure Captions

-21-

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.





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