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

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

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UNIVERSITY OF CALIFORNIA

Lawrence Radiation Laboratory
Berkeley, California 94720

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[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 $Zn(C_2H_4NH_2)_3NC\ell$ 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.

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_6tren 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_6tren, it forms more stable five-coordinated complexes than tren. Me_6tren 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 $[Zn(tren)C\ell]B(C_6H_5)_4$, 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 ϕ 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 2θ angle was 40° $[(\sin\theta)/\lambda = 0.596]$. Background was plotted as a function of 2θ 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 β = 95.0±0.2°. 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 β_{ij} to B_{ij} which is in units of $\mathring{A}^2$; the relation between these two quantities is $4\beta_{ij} = a_i^* a_j^* B_{ij}$, where a_i^* is the ith 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.

OBSERVED AND CALCULATED STRUCTURE FACTORS OF TRIS-(2-AMINOETHYL)AMINOCHLOROZINC[(I)]TETRAPHENYLBORATE.
FCA(0,0,0) = 4768

```
 H FOB FCA      0 169 167      5 247 252   -10  0   4*  -6 112 119   -3 222 224   -3 398 395   -3 320 318   3 131 136   -7  60  63   1  85  86    2  0  35*   3 293 290   -6  0  12*   5  76  76   -4  39  43
K,L,= 0, 0      1 180 172      6 207 206   -9 150 153   -5 220 217   -2  63 463   -5  52  42   -6 237 238   2  76  78    1  0  59*   3 384 383   -5  47  45   6 202 200   -3 184 186
   1 371 356    3 134 136      7 221 228   -8 109 97    -4 227 234   -1 207 211   -1  66  66   -1 175 175  K,L= 3, 18   -4 220 232   3  0  56*   4 120 126   2 185 189   -4  46  45   7 103  99   -2  80  78
   2 629 638    3  88  83      8  41  40   -7 149 147   -3  45  44    0 144 150    0 475 465    0  78  82   -4  58  63   4  94  91   5  55  45   3 119 111   -3  83  87   8  0  4*   -1  38  42
   3 168 157    4 160 159      9 179 176   -6 156 157   -2 312 307    1 147 154    1 242 244    1 128 127   -3  88  78   -3 121 125  K,L= 4, 17   6  0  6*   4  0 12*   -2  26  27*  K,L= 7, 6   0  59  43
   4 272 275    5 114 108     10 230 235   -5 176 179   -1  51  42    2 140 153    2 453 455    2 158 163   -2  29 200   -2 186 184   -4  30 199*   7  46  35   5  81  84   -1  26  7*   -8 124 123   1  60  61
   5 307 304    6 339 316     11  39  30   -4 116 125    0 248 245    3 144 159    3 214 217    3 215 202   -1  91  41   -1  78  73   -3  41  45   8  88  91   6 118 114   0 214 218   -7  57  53   2  93  87
   6 209 214    7 211 209     12  29  31*  -3 122 119    1 313 312    4 300 314    4  37  28    4 210 215    0  83  76    0 119 128   -2 105 108   9  29  17*   7 114 101   1  73  75   -6  28  32*   3 109 108
   7 200 198    8 114 107  K,L= 1, 6   -2 381 379    2 275 279    5  0   9*   5 180 183    5  0   9*   1 219 227   -1  0   3* K,L= 5, 8   8  0  1**   2  37  39   -5  89  90   4 206 204
   8  54  57  K,L= 0, 16   -12 155 168   -1 190 186    3  33  36    6 233 244    6 223 226    6 194 196    2  23  70    2  49  42   0 113 117   -10 183 187   4  44  52   3  27  52*   -4  46  50   5  75  58
   9 235 241    9  102 101   -9 153 154    8 175 175    5 209 206    7 144 153    7 126 128    7 140 134  K,L= 4, 0   5 237 246   1 128 129   -9  56  60   10 138 137   4  39  42   -3 188 187   6  29  14*
  10 296 296   -7  61  18   -10 119 115    1 138 141    5 304 296    8 101 109    8  41  50    8  46  39    0  85  86   4 261 270    2  0  2*   -8  90  87  K,L= 6, 2   5  28  24*   -2 155 159   7  60  51
  11   0  10*   -6  28  27*   -4  74  73    2  0  15*   6 204 203    9  80  69    9  71  68    9  67  64    1  18  49   5 105 103  K,L= 5, 0   -7 105 108   -10  65  60   6  76  72   -1  95  96  K,L= 8, 5
  12  69  32   -5  47  35   -8  99 108    3  65  66    7 110 114   10   0 14*   10  53  39   10  41  31    2 117 116   6 152 165   -1 540 540   -6 178 183   -9 101 111   7 106  98   0  35  68   -2  42  33
  13  30  16*   -4 203 206   -7 180 171    4 131 130   10 190 194  K,L= 2, 11   1  42  65  K,L= 3, 1   3 135 139   -8 199 197   2  65  63   -5  35  36   -8 141 138  K,L= 6, 11   1 140 145   -6  50  37
K,L= 0, 2   -3  53  23   -6  77  73    5  37  33    9  35  33   -11  54  54   12  83  82   -11  51  53   4  60  51   8  75  73   3 153 151   -4 219 212   -7  64  69   -8 140 135   2  96 102   -5  63  58
  -13  66  43   -2 115 119   -5 334 336    6  27  19*   10 114 116   -10  85  71  K,L= 3, 2   -10  40  34   5 113 116   9  47  86   -3 122 126   -6  0  1*   -7  82  82   3  45  42   -4  55  53
  -12 161 160   -1 182 179   -4 309 302    7  83  72   11  62  62   -9  82  80   -12 112 117   -9  38  33   6  0  6*   10 186 185   5 108 109   2  0  22*   -5  64  63   4  65  50   5  61  70
  -11 311 320    0 118 118   -3 153 154   8 175 175    12 133 130   -8 144 147   -11  42  43   7  74  73   0 KL,K= 4, 8   6  26  28*   -1 130 126   -4  41  37   -5 121 128   5  0  12*   -2  27  38*
  -10 123 117    1 135 140   -2 206 193    9  0  15*  K,L= 2, 4   -7 119 121   -10  64  64   -7 159 159   8 141 148   -11  42  43   7 125 124   0 415 424   -3  23  15*   -4  38  31   6  93  96   -1  27  26*
   -9  62  70    2  54  57   -1 303 290  K,L= 1, 14   -12  0  11*   -6 264 261   -9  93 102   -6 158 159   9  45  45   -10  63  67   8 275 275   1 222 227   -2 207 217   -3 197 194   7 100  97   0  38  20
   -8 278 277    3 139 132    0 289 271   -9  50  67   -11  0  6*   -5 281 292   -8  67  63   -5 334 335   10  0  7*   -9  27  74   9 135 140   -1 157 153   -1 291 299   8 86  68   2 178 181*
   -7 224 232    4  0  18*    1 103  84   -8  86  87   -10  26  7*   -4 156 160   -6 166 170   -4  66  79   11  57  68   -8  38  39   10  28  35*   3 140 140   0  0  27*   -1 150 153  K,L= 7, 7   2  47  44
   -6  76  74    5  94  95    2  79  68   -7  0  8*   -9 109 113   -3  63  64   -6 436 442   -3  48  43   12  0  25*   -7  25  27*   11 181 166   5  25  14*   1 116 109   0  37  9   -8  88  91   3  67  72
   -5 124 120    6 233 227    3 344 359   -6  65  60   -8 146 141   -2  73  72   -5 168 167   -2  37  39  K,L= 4, 1   -6  0  13* K,L= 5, 1   4  85  92   2  23  23*   1 107  98   -7  29 209*   4  43  48
   -4 186 184  K,L= 0, 18   4  87  93   -5  51  55   -7  69  60   -1  38  24   -4 266 265   -1  30  32   -12  99  98   -5  97  60   5  41  38   3 168 155   3  0  18*   2 159 155   -6  29  28*   5  53  17*
   -3 102  99   -5  30  28   5 149 156   -4 116 127   -6 104 170    0  63  84    0  63  299    0  63  71   -4  72  72   -10  28 128   7 146 146   4  82  82   3  72  80   -5 141 137   6  59  49
   -2 193 190   -5  83  88   6  83  75   -3  74  75   -5 231 237   1 115 114   -2 212 210   1 200 200   -10  27  34*   -1  0  14*   -9  54  56   8  40  34   5 127 133   4  92  92   -4  38  22  K,L= 8, 6
   -1 395 384   -4  50  49   7 141 138   -2 101  83   -4 373 370   2  23   7*   -1  90  89   2  57  59   -9 169 172   -2  48  67   -8 109 111   9 168 155   6 179 177   5  40  30   -3 139 135   -7  84  68
    0 299 264   -3  57  47   8  0  26*   -1  49  41   -3  97 102   3 170 172    0  31  25   3 155 157   -8 120 121   -1  88  91   -7  55  66  K,L= 5, 2   7  95  99   6  72  64   -2  85  85   -6  29  69
    1  99 104   -2 272 273   9  50  47    0  42  31   -2  74  74   4 131 135    1 154 156   4 350 360   -7 119 120    0  53  44   -6  0 19*   -10  30  5*   8 119 125  K,L= 6, 12   -1 191 195   -5  49  35
   -1 901 917   -1 221 224   10 111 118   1 140 149   -1 167 167   5 139 132   2 169 165   5  67  74   -6 286 297   1  65  72   -4  23  24*   -9  99 101   9  40  35   -7  84  82   0  58  43   -4 153 150
    3  64  63   0  40  50   11  49  47   2 109 117   0 483 480   6  26  25*   3 634 632   6  82  85   -5 397 405   2  94  72   -3  36  21   -7  76  59  K,L= 6, 3   1 122 125   -3  73  66
    4  39  42   1 103  97   12  30  37   3  57  63   1 352 357   7   0  9*   4 465 478   7  26  32*   -4  0  32*   3 111 113   -3  36  21   -6  57  55  K,L= 6, 4   2 172 170   -2  73  67
    5 170 172   2  58  56  K,L= 1, 7   4  45  52   2  97  97   8  95  94   8 111 112   -3 380  6   8 109 109   -2  74  72   -10  21  6*   -5  90  97   -10 216 210   -4  28  29*   3  38  40   -1  72  74
    6 135 126   3  59  64   -12 164 164   5  93  93   3 260 255   9  29  30*   6 249 252   9  74  76   -2 397 405   5  24  39*   -1  75  72   -5  84  87   -9 123 112   -3  39  29   4  38  46   0  82  86
    7 204 211   4 119 110   -11 189 194   6 114 114   4 544 556  K,L= 2, 12   7 188 190   10  88  89   -1  69  71   6  43  31   0 313 306   -4  65  65   -8  27  11*   -2 102  99   5  96  89   1  27  36*
    8 219 219  K,L= 1, 0   -10  98  98   7  95  90   5  90  85   -10  65  70   8 107 113  K,L= 3, 10   0 373 362   7  87  95   1 302 302   -3  0  4*   -7  80  86   1 110 107   6 145 143   2 110 107
    9 335 336   1  20  17   -9  25  16   8  30  40*   -5  76  76   -9  85  74   -1  42  44   1  45  54   -8  71  72   1 134 130   -2  41  36   -6 129 129   0  27  12*   7  59  56   3  62  59
   10 127 113   2 355 349   -8 182 191  K,L= 1, 15   7  50  49   -8 137 134   10 110 105   -10  0  15*   2 242 247   9  28  26*   3  30  36   -1  92  97   -5  0  22*   1  27  5* K,L= 7, 8   4 106 111
   11 126 123   3 271 254   -7 110 109   -9 107 114   8 231 227   -7  41  39   -9  61  59   3  47  52   10  28  9*   4  44  51   0 160 164   -4 164 163   7  87  83   -8  0  18*   5  41  28
   12 184 182   4 117 113   -6  90  90   -8  29  28*   9  25  9*   -6  0  3*   12  42  48   -8 174 178   4 531 538  K,L= 4, 9   5  93  47   1 104 104   -3  52  53   3  49  46   -7  0  12*   -2 228 230
   13  30  54   5 168 167   -5  98  99   -7 148 151   10  88  86   -5 183 184   -5  3,  3   -7  51  50   5 337 330   -10  50  53   0  0  8*   2  94 122*   -2  73  73   4  49  51   -6  0  1*   -6  42  41
  K,L= 0, 4   6  85  89   -4 333 327   -6  86  77   11 108 108   -4 160 166   -12  29  27*   -6 129 130   6  31  22   -9  0 12*   7  76  81   3  0  14*   -1 316 318   5  88  74   -5  0  1*   -6  42  41
   -13  30  27*   7 106 108   -3 246 237   -5  53  60   12  78  82   -3  0  6*   -11  39  31   -5 197 201   7 170 173   -8 155 156   8  45  39   4  36  19   0 307 311   6  60  60   -4  27  8*   -5  41  27
   -12  89  76   8  0  14*   -2 583 561   -4 128 124  K,L= 2, 5   -2 272 266   -10  87  82   -4 316 324   8  79  62   -7 168 169   9  0 10*   5  90  85   1 189 203  K,L= 6, 13   -3 116 113   -7  28  28*
   -11 189 195   9  58  64   -1  95  85   -3 244 242   -12  0  7*   1 119 118   -9  87  84   -3  32  24   9  26  27*   -6  40  40   10  85  71   6  29  21*   7  62  67   3 146 148   -5  50  35   -1 120 119   -2 104 105
   -10 287 276   10 25  6*   0 284 273   -2  26  9*   -11 140 143   0  0  8*   -8 150 151   -2  82  88   10 105  99   -5  86  91   11  29  21*   7  62  67   3 146 148   -5  70  73   0 141 140   -1  39  25
   -9 497 517   11  38  23   1 237 235   -1  51  39   -10  45  41   1  80  79   -7  50  41   -1 136 138   11  49  47   -4 234 233  K,L= 5, 2   8  86  82   4  59  25   -3 132 119   1 169 167   0  73  78
   -8 114 114   12  40  58   2 484 496   0 163 153   -9 105 105   2 147 156   -6 327 317   0  38  42   12  0  5*   -3 189 186   -11  29  5*   9  30  36   -1  92  97   -5  0  8*   2 170 178   0  10*
   -7  21 114   13  0  2*   3 265 278   1 168 161   3 121 120   3 102 110   -4 311 318   1 311 319  K,L= 4, 2   -2 145 140   0  40  31   2 283 283   -10 97 93  K,L= 5, 9   -9 164 164   -9  29  19*   7 104 100   3  38  39   2  0  3*
   -6 207 211  K,L= 1, 4   4  28  10   2  37  30   -7  88  84   4  0  4*   -4  26  18   2 300 307   -10  42  50   -1 144 137   -9 164 164   -9  29  19*   7 104 100   3  38  39   2  0  3*
   -5  48  45   -13  30  31*   5  81  85   3 103 105   -6 248 254   5  44  38   -3 101  97   3 166 164   -11  40  30   0  83  89   -8  26  16*   -8 123 125   8  95  88   0 158 157   4  87  88   3  43  37
   -4 465 461   -12  56  50   6 271 273   4  66  68   -5 295 297   6  37  38   -2 112 111   4 206 202   -10  47  41   1  50  51   -7 150 151   -7  47  45   9 164 157   1  74  78   5  85  79   4  43  38
   -3 607 609   -11 131 126   7 146 149   5  28  13*   -4 324 313   7  5  48   -1 317 322   4 317 322   -9  26  25*   2 265 271   -6 167 165   -6  26  19*   10  67  97   2  49  48   6 162 159   5  78  76
   -2 744 734   -10 173 170   8 198 202   6 134 139   -3  11  11   8  80  83   0 243 242   6 111 112   -8  49  56   3 317 324   -5  64  66   -5 109 114  K,L= 6, 4   3  0  22*   7  52  41  K,L= 8, 8
   -1 343 344   -9  92  88   9 240 233   7 184 184   -2 208 200   9  72  69   1 489 488   7  70  76   -7  0  5*   4  68  69   -4 145 148   -4 113 107   -10  83  67   4  65  63  K,L= 7, 3   -6 166 166
    0 840 787   -8 104 113   10 194 189  K,L= 1, 16   -1  16  11*  K,L= 2, 13   2 100 102   8  83  78   -6 201 198   5 187 190   -3 185 185   -3 131 123   -4  49  26   5  52  47   -7  0  4*   -5 134 130
  110631071   -7 248 249   11  50  34   -8  0  12*   0  22  24   -10  42  25   3 219 223   8  29  14*   -5 113 118   6 208 209   -2 151 151   -2 206 208   -8  27  15  K,L= 6, 14   -6  95  87   -4  95  86
   0 288 282   -6 105 113  K,L= 1, 8   -7  58  45   1 191 190   -9  51  64   4 337 343   10  0  4*   -4  28  36   7  38  37   -1  78  72   -1 211 213   -7  0  9*   -5  73  76   -5 166 163   -3  73  68
   3 388 385   -5  25  19   -12  0  15*   -6  56  54   2 223 219   -8 144 143   5 205 208  K,L= 3, 11   -3 352 339   8 149 144   0 488 494   0  42  47   -6  25  10*   -4  0  25*   -4  55  58   -7 129 136
   4 141 142   -4 212 210   -11  40  33   -5  0  2*   3  89  87   -7 197 197   6  76  79   -10  29  14*   -2 185 184   9  41  49   1 279 271   1 172 175   -5 126 133   -3 180 182   -1  49  38
   5  92  93   -3 247 233   -10  27  18*   -4 206 210   4  79  80   -6 104 113   7 153 156   -9  0  2*   -1 187 187  K,L= 4, 10   7 169 172   2 165 162   -4  24  40*   -2  64  74   -2  85  88   0  29  10*
   6 419 421   -2  28  29   -9  25  22*   5 260 264   -5  52  20*   8  24  23*   -6  76  71   0 157 161   -10  0  13*   7 160 159   3  0  8*   -3 120 121   -1  0  4*   1 105 101   1 145 151
   7 185 186   -1 687 660   -8 116 120   -2 157 144   6 349 352   -4 105 100   9  72  65   -7  97  86   1 127 124   -9  84  91   4 147 144   4 140 135   -2  69  73   0  86  76   0  27  24*   2  90  88
   8 146 150   0 496 478   -7  0  10*   -1  46  29   7  32  37   -3 157 153   10  27  22*   -6  35  23   2  79  81   -8 118 112   5 113 113   -1  56  50   1  76  89   1 227 230   3 100  99
   9  49  47   1 161 169   -6  0  6*   0  79  89   8  76  76   -2  24  45   11  18  22*   -5  24 109   3 247 247   -7 128 116   6 117 129   6  0  2*   2  29  27*   2  37  30*   4 134 139
  10 215 208   2 478 490   -5 148 141   1  75  68   9  25 25*   -1  84  93   12  78  77   -4  63  67   4  73  73   -6 105 102   8  86  80   7  40  27   1  23  26*   3  0  8*   3  83  83   5  64  62
  11 220 215   3 554 558   -4  40  31   2  47  52   10  38  28   0  0  11*  K,L= 3, 4   -3 131 130   5  71  14*   -5 135 136   8 101  93   2  0  14*  K,L= 6, 15   4 161 159  K,L= 8, 9
  12  29  24*   4  78  79   -3 300 294   3  27  15*   11  85  83   1  42  56   -12 112 112   -2  0  7*   9  56  47  K,L= 5, 11   5  50  51   -5  51  54
 K,L= 0, 6   5  93  94   -2  89  71   4  49  52   12  98 112   2 171 178   -11  39  40   -1  73   7   1 106 113   -3 137 139   10 170 170   -9  30  35*   6  36  35   -2  83  86   6  50  58   0  53  52
   -12 121 121   6 257 262   -1  60  47   5  41  34  K,L= 2, 6   3 213 208   -10  65  67   0  72  73   8  25  15*   -7  0  17*   11  66  66   -8  29  32*   5  0  8*   -1 118 119  K,L= 7, 10   -3  29  25*
   -11 154 154   7  88  89   0  41  20   6  51  58   -12  96  98   4  96  94   -9  0  7*   1  56  63   9  37  42   -1  86  84  K,L= 5, 3   -7  62  65   6  52  40   0  8  7*   -7  84  85   -2  57  58
   -10 257 254   8 191 185   1 190 184  K,L= 1, 17   -11 146 155   5  53  54   -8 225 225   2 219 221   10  27  14*   0  0  5*   -11  42  37   -6  0  3*   7  71  65   1  0  7*   -6  0  16*   -1  28  25*
   -9 117 115   9 359 366   2 188 190   -7  66  63   -10  59  59   6 133 130   -7  25 241   4 131 124   11  29  7*   1  73  72   -10  56  50   -5  37  9   9 101 101   2  60  71   -5  81  70   0  75  66
   -8  65  70   10 223 228   3 206 208   -6  28 113*   -9  93  93   7  63  68   -6  40   4 101  99  K,L= 4, 3   2  97 111   -9 108 102   -4  45  31   9  29  4* K,L= 7, 0   -4  40  36   1  0  7*
   -7 125 127   11  97  89   4  0  8*   -5 124 120   -8 119 116   8  0  17*   -5 193 192   5  98 103   -12  0  19*   3 110 112   -8  52  45   -3  44  36  K,L= 6, 5   1  0  10*   -3  78  72   2  0  11*
   -6  46  21   12  69  57   5 109 117   -4  63  52   -7 273 286  K,L= 2, 14   -4 491 499   6 134 141   -11  0  13*   4  82  75   -7  26  42   -2  71  68   -10  78  63   2 169 179   -2 135 135   3  65  65
   -5 142 139   13  42  48   6  97  97   -3 152 140   -6  21  11*   -4  60  60   3 225 219   7  86  74   -10 133 131   5  62  56   -6  47  46   -1  50  45   3 177 187   -1 125 132   4  73  60
   -4 163 165  K,L= 1, 2   7  54  58   -2 184 170   -5 112 118   8  40  45   -2   7  9*   8  0  17*   9 127 123   6  59  50   -5  47  43   0  79  79   -8 228 227   4  36  32  K,L= 8, 10
   -3  38  47   -13  0  9*   8  84  78   -1 248 246   -4 292 297   -7  28  27*   -1  49  32   9  29  34*   -8  92  81   7  95  96   -4  0  12*   1 160 165   7  70  71   5  26  26*   1  48  43   -4  89  86
   -3  38  42   -12  0  30*   9  38  21   0  27  10*   -3 314 301   -6  89  94   0  44  35  K,L= 3, 12   -7 244 242   8  28  15*   -3  48  49   2  26  9*   -6 118 117   6  46  43   2  28  13*   -3 152 151
   -2 168 170   -11 103 102   10  28  30*   1  12  15*   2  87  86   -4 153 152   1 143 147   -10  53  53   -6 291 289   9  0  4*   -2  93  90   3  78  79   -5  81  81   7 141 139   3  49  52
   -1 264 267   -10  56  57   11  29  19*   2  97 110   -7  76  71   -4  57  54   2 349 340   -9  90  87   -5  21  14* K,L= 4, 11   -1  65  57   4  80  72   -4  42  43   8 148 144   4  64  60   -1  0  23*
    0 878 840   -9 101 104  K,L= 1, 9   3  28  13*   0 158 154   -3 273 267   3 301 307   -8  39  36   -4 291 291   -10  0  24*   0 162 156   5  67  66   -3 196 191   9  59  57   5  83  74   0 131 139
    1 775 766   -8 149 156   -12  79  70   4  50  36   1 187 193   -2 130 126   4  36  31   -7 146 143   -3 402 402   -9 137 128   1  61  64   6  28  7*   -2 240 239  K,L= 7, 1   1  0  4*
    2  23  51   -7  83  86   -11  85  85   5  27  18*   2 175 176   -1  25  23*   1 173 183   -6 218 237   -2 138 134   8 173 173   1 180 185   7  58  55   -1 212 215   -6  30  29*   3  83  86
    3 318 321   -6 190 175   -10  94  95  K,L= 1, 18   3 328 335   0 141 141   6 182 185   -5 159 158   -1 192 197   -7  54  44   3 184 183   8  0  15*   0  57  55   -8  70  74   -5  29 15*   3 136 133
    4  33  26   -5 125 118   -9 124 125   -6  30  17*   4 120 118   1  79  83   7  73  80   -4  49  51   0 217 221   -6  86  79   4  59  53  K,L= 5, 12   1 125 124   -7  39  34   -4 103  97  K,L= 8, 11
    5 245 247   -4 276 274   -8 135 148   -5  51  48   5  21  18*   2  72  68   8  49  44   -3 154 156   1  76  72   -5 147 151   5  33  19   -8  0  22*   2 173 177   -6 125 132   -3  69  65   1  42  40
    6 270 274   -3 521 510   -7  0  11*   -4  53  51   6 310 318   3 50  48   9  58  57   -2 130 133   2 275 279   -4  58  40   6  39  49   -7  78  69   1 102 104   -5  52  54   -2  29 29*   -1  91  90
    7  64  69   -2 571 552   -6 119 121   -3 115 117   7  84  88   4 176 182   10  0  26*   -1  95  91   3 227 237   -3 114 113   7  43  45   -6 154 156   4  49  42   -4 258 249   -1  93  90   -1  51  57
    8 134 134   -1 194 192   -5 242 242   -2  40  5   8  74  71   5  72  62   11  75  73   0 136 136   4 115 104   -2 238 235   8  0  4*   -5  54  57   5  57  50   -3 136 139   0 112 102   0  59  68
    9 237 234   0 647 622   -4  80  86   -1  80  62   9  69  65   6  49  45   12 116 109   1  93 108   5  31  45   -1 137 136   9  27  24   -4  92  94   6  26  17*   -2  49  58   1  89 100   1  42  24
   10 255 247   1  86  89   -3 305 296   0  49  46   10 102  97   7  77  74   K,L= 3, 5   2  42  55   6 274 275   1 252 220   10  29  7*   3 201 207   7  40  40   -1 108 101   2 133 130  K,L= 8, 0
   11  40  21   2 302 296   -2 461 471   1  0  20*   11  41  85   8  73  70   -12  83  74   3 231 235   7 179 187   1 215 215   11  30  45*   -2 183 177   8 208 193   0 230 227   3  50  47   1  28  19*
   12 136 129   3 191 141   -1 208 196   2  82  92  K,L= 2, 7   K,L= 2, 15   -11  97 106   4 249 255   8  43  27   2 129 127  K,L= 5, 4   -1  78  88   9 152 150   1 111 111   4  41  37   2  26  4*
 K,L= 0, 8   4  43  45   3 393 388   3 106 114   -12  41  33   -8 116 114   -10  27  41*   5  64  60   9 115 112   3  75  70   -11 221 225   0 210 216  K,L= 6, 6   2 132 130   5  30 13*   3  99 100
   -12 109  99   5 249 239   4 197 201  K,L= 1, 19   -11  46  41   -7  80  82   -9  25  25*   6  67  43   10  68  74   5 199 196   -10 232 227   1 282 280   -10  68  62   3  25 18* K,L= 7, 12   4  29 15*
   -11 214 216   6  62  62   2 150 152   -4  0  19*   -10  38  34   -6  55  57   -8  68  81   7  28  32*   11  0  6*   5 178 177   -9  94 191   2  26  33*   -9  29  17*   4 174 175   -5  0  20*   5  42  40
   -10  27  15*   7 156 151   3 152 150   -3  98  91   -9  84  73   -5 196 192   -7 174 177   8  0  6* K,L= 4, 4   6  38  31   -8 137 143   3  89 104   -8 144 153   5 180 181   -4  51  72  K,L= 9, 1
   -9 191 189   8  39  48   4 277 261   -8 149 147   -4  26  17*   -6 340 347  K,L= 3, 13   -12  30  24*   7 166 169   -7 139 140   4  0  14*   -7  27  7*   6 272 270   -3 104 105   -5  51  60
   -8  73  73   9  0  16*   5 143 120   4 106  85   -3 258 254   -5 223 227   -5  68  -11  31  28   2 271 122   -6  0  23*   7  48  51   -3  78  78   7 147 148   -2 135 134   3  67 218
   -7 262 262   10  44  43   6 171 174   0  42  52   -6 252 252   -2  90  85   -4 124 117   -8  44  46   -10  72  63  K,L= 4, 12   -5 154 151   6 125 120   -5  71  42   8  57  62   -1  57  45   -3 107 106
   -6  65  67   11  27  21*   7 180 190   1 157 153   -5  0 14*   -1  37  21   -3  95  96   -7  95  84   -9  26 15*   -9  0  17*   -4 183 182   7 122 122   -4 156 154   9  30  24*   0  70  45   -2  75  72
   -5 320 322   12 107 107   8  64  64  K,L= 2, 0   -4 236 226   0  73  67   -2 139 138   -6  46  21   -8  99  90   -8  49  35   -3  43  41  K,L= 5, 13   -3  24 30* K,L= 7, 2   1 119 117   -1  49  56
   -4 144 144   13  0  4*   9 115 117   0 237 235   1 111 112   7  70  70   -5 124 117   -7 166 166   -7  27  84   -2 184 191   8  47  47   -2  84  91   2 181 184   -1  93  92   2 218 214   0  28 23*
   -3 309 311   -13  59  44   10  94  91   1 121 119   -2 286 262   2 118 118   0  46  37   -4  67  64   -6  75  83   -6  0  14*   -1 233 230   -7  0  24*   -1 105 108   -8  75  66   3  79  29*   1  0  13*
   -2 575 574   -12 132 123   11 107  46   2 302 293   -1  57  56   3  66  56   1  18  26*   -3  25  10*   -5 154 153   -5  37  22   0 283 277   -6  0  9*   0 127 130   -7  28  22*   4  0  37*   2 113 111
   -1 478 502   -11 156 156  K,L= 1, 13   3 381 371   0  62  56   4  67  58   2  18 15*   -2  96 101   -4  29  26   -4  81  78   1 100 104   -5  83  81   1  74  24*   -6 149 145  K,L= 7, 13   3 131 132
    0 148 169   -10  62  60   -11  58  68   4 282 274   1  73  76   5  74  62   3  33  362   -1  63  82   -3  63  67   -3  71  69   0 212 216   -4  53  53   2  0  5*   -5 119 116   -1  52  58   5  37  29
    1 476 462   -9 307 313   -10  0  12*   5 338 302   2 256 261   0  71  62   4 293 299   0  60  46   -7 136 138   -2 129 118   3  58  61   -3  54  50   3  0  15*   -4 174 176   -3  0  3*   5 126 130
    2 237 214   -8 401 411   -9  65  62   6 345 348   3 175 175   7  30  35*   5  97  96   1 129 133   -1  19  7*   -1  25  25*   4 113 109   -2 110 104   4  83  81   -3 261 257   -2  97  98  K,L= 9, 2
    3 181 180   -7 143 150   -8  25  23*   7 144 149   4 209 221  K,L= 2, 16   6  74  72   2 161 166   0 125 120   0  89  88   5 133 136   -1  88  88   5  26 12*   -2  42  51   -1  29  36*   -5  30  22*
    4 191 185   -6 121 121   -7  42  44   8  0  30*   5 207 212   -10  0  15*   -2  36  2   -1  47  43   3 187 175   6 133 134   0  46  46   1 148 139   2 139 139   -3  30  34   0  74  86
    5 124 123   -5 337 340   -6  33  27   9 103 105   6 118 118   -7 120 119   8  35  30   3 126 134   2  28  27   2  25 11*   7  25  22*   1 107 107   7  34  30   0 245 249   1  29  36*   -3  29  28*
    6 147 150   -4 313 310   -5 124 126   10 145 134   7  76  70   -6  28  20*   9  26  4*   5 126 137   3  81  80   3  44  48   8 119 116   2  47  45   8  28  21*   1 175 184   2 139 145   -2  70  64
    7 345 342   -3 556 546   -4 272 266   11  27  30*   8  94  89   -5  39  30   10  82  82   6  62  57   4 124 126   4  26  29*   9 179 176   1  0  18*   9  98  87   2 101 100  K,L= 8, 0   -1  0  14*
    8 331 331   -2 913 894   -3 103 114   12  0  8*   9  0  15*   -4  39  37   -9  0  31*   11  28  27*   1 174 179   10 129 123   3  0  18* K,L= 6, 6   3  35  27   1 133 130   3  0  14*
    9  99 102   -1  97  91   -2  65  37  K,L= 2, 1   10 104  92   -3  81  76  K,L= 3, 6   6  30  33*   6 124 124   6  34 24* K,L= 5, 5   5  0  9*   -4  96  98   4 111 109   1  83  75   1  28  4*
   10  56  55   0  46  43   -1  29  27   -12 121 128   11  0  6*   -7  27  22*   -12  84  67  K,L= 3, 14   7  54  54   7 103 101   -11  51  50   6  42  56   -8  74  68   5  26  4*   2 109 120   2  70  79
   11 113 112   1  25  29   0 165 158   -11  77  79  K,L= 2, 8   -1 139 131   -11  40  31   -9  0  6*   8  51  44   8  0  12*   -10  49 101  K,L= 5, 14   -7  0  8*   6 126 120   3 218 226   3 135 125
  K,L= 0, 10   2 284 282   1 202 204   -10  26  30*   -12  0  11*   0  85  75   -10  47  39   -9 153 146   9  27  17* K,L= 4, 13   -6  0  7*   -5 165 161   -7  39  45   7  39  45   7  39  45   2 210 210   -4  27  24*
   -11 129 119   2 206 204   -9  26  24*   -11  0  24*   1  10  27   -7 168 170   0  0  8*   -9  0  27*   -8  37  34   -6  29  41*   -5  96 104   8  29  13*   5  55  54   5  66  63
   -10  0  16*   4 189 192   3  87  90   -8  32  29   -10  0  6*   2  67  57   -8 106 108   -6  39  30   11  0  5*   -8  0  4*   -7  36  32   -5 114 107   -4 106 110   9  30  22*   6 106 112  K,L= 9, 3
   -9 193 197   5 141 144   4 146 148   -7  70  70   -9 103 106   3  0  10*   -7 175 173   -4 113 101  K,L= 4, 5   -5  7 143 139   -6  59  65   -4 122 117   -3 256 250  K,L= 7, 3   7  48  28   -5 173 169
   -8  62  67   6  49  86   5  62  63   -6 192 174   -8 177 186   4  0  9*   -6 245 243   -4 146 148   -11  24  30*   -4 169 163   -4  51  50   -3  51  50  K,L= 8, 1   -1  51  50   K,L= 8, 1   -9 107 103
   -7  24  36*   7 117 124   6 204 203   5 397 397   -3  53  30   5  0  7*   -5 330 322   -3  78  73   -10  73  71   -5  27  20*   -4  32  33   -2  62  64   -1  24  26*   -8  81  83   -7  29  11*   -3  41  39
   -6  61  47   8 239 250   7  62  64   -4 140 138   -6 112 111   6  84  82   -4  98 102   -2  63  55   -9 105 101   -4 148 153   -3  31  28   -1 134 137   0 224 213   -7 114 100   -6  28  13*   -2 148 143
   -5 137 145   9 127 130   8 199 200   -3 213 217   -5 154 149  K,L= 2, 17   -3 130 130   -1  51  56   -8 208 209   -3 129 128   -2  71  73   0  78  73   1 339 342   -6 255 255   -5  96  96   -1  28  20*
   -4 344 356   10  0  28*   9  48  52   -2  81  75   -4 206 211   2  0  11*   0  72  77   -7  41  35   -2  64  61   -1  35  -2   -5 148 138   3  0  18*   2 139 139   -4  30  34   0  74  74
   -3 344 350   11 177 167   10  41  34   -1 721 711   -3 216 215   -5 137 133   1 130 137   1  45  38   -6 102  99   -1 187 191   0 139 138   2  48  51   3  71  75   -4  51  40   -3 113 112   1  40  42
   -2 177 181   12 149 164  K,L= 1, 11   0 150 140   -2 216 217   -4  49  26   0 219 220   2 179 182   -5 305 310   0 147 144   1  71  71   3  69  67   4  63  62   -3  50  45   -2  46  38   2  57  56
   -1 116 111  K,L= 1, 4   -11 144 139   1 470 470   -1  84  82   1 186 186   3 143 146   -4 390 400   1  58  52   2  22  29*   4  0  4*   5  37  24   -2 139 137   -1  8*   1  47  42
    0 466 454   -10  52  43   -10 116 122   2 184 159   0  7*   2  79  75   4  0  19*   -3 182 184   2 152 149   5  50  55   5 181 177   -1  69  74   0  52  0   4 117 125
    1 668 666   -12  0  16*   -7 173 180   3 266 268   1 175 180   -1  56  47   7 236 231   5  96 102   -7 260 264   3 161 158   4  56  50  K,L= 5, 15   7 214 203   0 144 137   1  69  61   5 140 134
    2 183 183   -11  38  33   -8 110 109   4 256 264   2 344 349   0  48  51   4 454 458   6  64  65   -1 181 186   4  61  55   5  24  16*   -6  30  31*   8  0  7*   1 173 201   2  26  7*  K,L= 9, 4
    3 239 233   -10  25  7*   -7  83  86   5 288 295   3 149 149   1  56  53   5 477 483   7  0  24*   0  64  59   5  43  36   6  74  79   -5 113 115  K,L= 6, 8   2  25  9*   3  0  16*   -5  0  7*
    4  0  14*   -9  0  27*   -6  75  75   6 209 214   4  0  11*   4 123 119  K,L= 3, 15   1 347 350   6  64  61   7  0  6*   -4  60  55   7  60  55   4  0  14*   -4  27  12*   -4  29 15*
    5 134 135   -8  55  55   -5  69  73   7  21  14*   5 300 309   5 137 141   7 134 138   -8  42  41   2 217 223   7  88  96   8  27  13*   -3  29  29*   -8  28  28*   4 103 104   5  48  37   -3  71  72
    6 235 232   -7  56  54   -4 352 349   8  86  92   6  47  49   4 149 159   8  80  82   -7  86  81   3 174 176  K,L= 4, 14   9  48  36   -2  28  31*   -7  0  18*   5  37  44   6  66  58   -2  41  27
    7 174 168   -6  20  6*   -3 167 188   9  0  14*   7  78  78  K,L= 2, 18   9  27 18*   -6  86  73   4 118 116   -8  51  51   10  98  64   -1  75  77   -6  60  55   6  0  10*   -1  41  42   7  0  10*   -1  47  42
    8 172 165   -2  51  45   -2 369 365   10  63  58   8  77  76   K,L= 3, 16   10  14  14   -5  0  7*   5 245 245   -7  71  64   5  0  15*   -3  78  73   -9  29  15*   -5  0  7*   7  63  56  K,L= 8, 7   0  70  57
    9  83  61   -4 265 262   -1 282 275   11  67  64   9  85  80   -4  0  17*   11  97  97   -4  47  56   6  78  84   -6  49  32   -11  85  89   1  70  68   -4  36  35   8 112 108   -7  97  97   1  0  25*
   10 176 167   -3  96  91   0 211 204   12  0  15*   10  28  13*   -3  0  6*  K,L= 3, 7   -3  71  74   7  0  16*   -5  83  81   -10  57  45   2  58  51   -3  15  16  K,L= 7, 4   -6  40  38   2  0  10*
  K,L= 0, 12   -2  95  90   1 254 255  K,L= 2, 2   11  42  49   -2 132 126   -12  73  69   -2  27  7*   8  89  84   -4 118 118   -9 163 167   3  0  14*   -2 214 216   -9  30  23*   -5 198 196   3  71  71
   11 188 192   -1 151 153   2  96  
```

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 $Å^2$ 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).

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 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 $Zn(C_2H_4NH_2)_3C\ell^+$ 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]

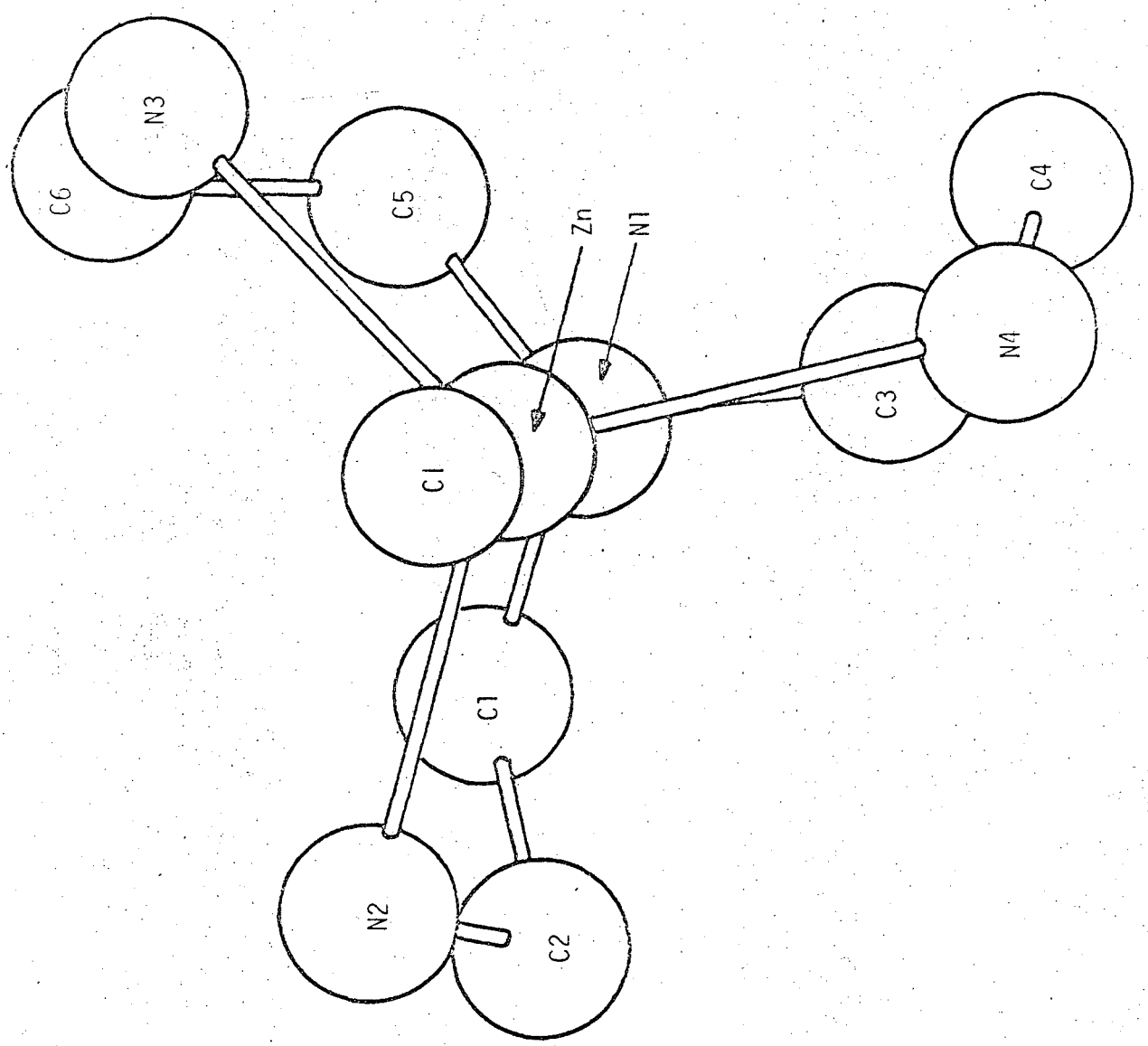
Atoms	Dist. Å	Atoms	Dist. Å
N2 - H1	0.83	C12 - H	0.97
- H2	0.84	C13 - H	0.96
N3 - H1	0.90	C14 - H	0.98
- H2	0.84	C15 - H	0.95
N4 - H1	0.83	C16 - H	0.99
- H2	0.84	C22 - H	0.91
C1 - H1	1.00	C23 - H	0.99
- H2	1.08	C24 - H	0.83
C2 - H1	0.86	C25 - H	0.93
- H2	0.97	C26 - H	0.89
C3 - H1	0.90	C32 - H	0.97
- 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.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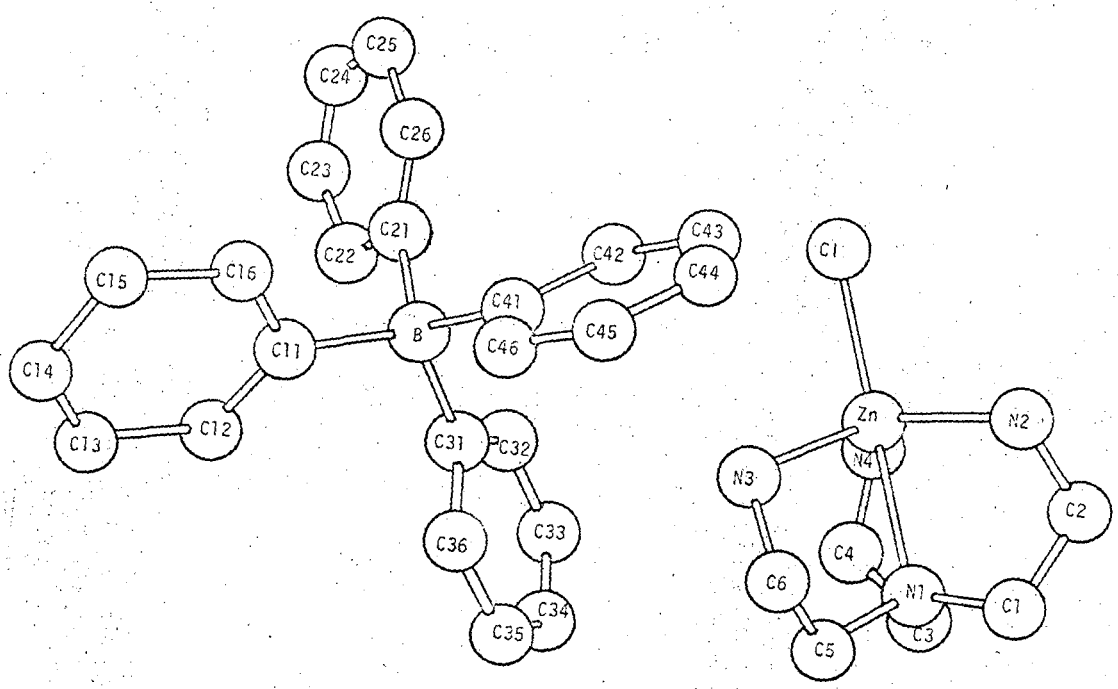
C6
C5
Zn
N1
C4
N4
C3
C1
C1
N2
C2



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LAWRENCE RADIATION LABORATORY
UNIVERSITY OF CALIFORNIA
BERKELEY, CALIFORNIA 94720