originate through the *a* glide: C(29)–Cl, 3.579; C(34)–C(6), 3.608; C(17)–C(9), 3.618; C(35)–C(6), 3.691.

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

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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 $a = 13.76 \pm 0.04$ Å, $b = 10.33 \pm 0.03$ Å, $c = 20.35 \pm 0.06$ Å, and $\beta = 95.0 \pm 0.2°$, contains four formula units; the calculated X-ray density is 1.30 g/cm³. 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 pentacoordinated to one chlorine and four nitrogen atoms in a trigonal-bipyramidal configuration.

Introduction

Although the coordination number 5 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 $[M^{II}Me_6trenX]Y$ or $[M^{II}trenX]Y$. 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 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, or Zn, and for which X = Y = Cl, Br, I, NO_3, or ClO_4.[3,4] Much physical evidence, including conductivity, spectral, and magnetic measurements, indicated that these complexes are five-coordinated. In addition, crystal structure determinations of [Cu(tren)(NCS)]SCN,[5] $[Co(Me_6tren)Br]Br$, and $[Cu(Me_6tren)Br]Br$[6] reveal that the metal atoms are indeed five-coordinated and situated nearly at centers of slightly distorted trigonal bipyramids. A recent report[7] on the structure of Zn(tren)(NCS)(SCN) also shows trigonal-bipyramidal symmetry.

More recently, a series of complexes has been prepared, [Zn(tren)X]Y, for which X = Cl, Br, or I and Y = X, ZnX_3, or $B(C_6H_5)_4$.[8] We report here 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 Kα_1, λ 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° takeoff 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}. No absorption correction could be made because the crystal was lost and its dimensions are unknown. The Lorentz and polarization corrections were applied to the data. No extinction correction was found necessary.

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 weighting factor. Atomic scattering factors[9,10] for neutral zinc, chlorine, boron, carbon, nitrogen, oxygen, and hydrogen were used. Both the real and imaginary

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

(3) M. Ciampolini and N. Nardi, *ibid.*, **5**, 41 (1966).

(4) M. Ciampolini and N. Nardi, *ibid.*, **5**, 1150 (1966).

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

(6) M. DiVaira and P. L. Orioli, *Inorg. Chem.*, **6**, 955 (1967); M. DiVaira and P. L. Orioli, *Acta Crystallogr., Sect. B*, **24**, 595 (1968).

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

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

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

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

TABLE I

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR TRIS(2-AMINOETHYL)AMINOCHLOROZINC(II) TETRAPHENYLBORATE[a]

```
FCA(0,0,0) =  4768

 H FOB FCA
K,L=  0,  0
  1 371 356
  2 629 638
  3 168 157
  4 272 275
  5 307 304
  6 209 216
  7 200 198
  8  54  57
  9 235 241
 10 296 296
 11   0  10*
 12  69  32
 13  30  16*
K,L=  0,  2
-13  66  43
-12 161 160
-11 311 320
-10 123 117
 -9  62  70
 -8 278 277
 -7 224 232
 -6  76  74
 -5 124 120
 -4 186 184
 -3 102  99
 -2 193 190
 -1 395 384
  0 299 264
  1  99 104
  2 901 917
  3  64  63
  4  39  42
  5 170 172
  6 135 128
  7 204 211
  8 219 219
  9 333 336
 10 127 113
 11 126 123
 12 184 182
 13  30   5*
K,L=  0,  4
-13  30  27*
-12  89  76
-11 189 195
-10 287 276
 -9 497 517
 -8 114 114
 -7  21  11*
 -6 207 211
 -5  48  45
 -4 465 461
 -3 607 609
 -2 744 734
 -1 343 344
  0 840 787
 110631071
  2 288 282
  3 388 385
  4 141 142
  5  92  93
  6 419 421
  7 185 186
  8 146 150
  9  49  37
 10 215 208
 11 220 215
 12  29  24*
K,L=  0,  6
-12 121 121
-11 154 155
-10 257 254
 -9 117 115
 -8  65  73
 -7 125 127
 -6  46  21
 -5 142 139
 -4 163 165
 -3  38  42
 -3  38  42
 -2 168 170
 -1 264 267
  0 878 840
  1 775 766
  2  23  21
  3 318 321
  4  33  26
  5 245 247
  6 270 274
  7  64  69
  8 134 134
  9 237 234
 10 253 247
 11  40  21
 12 136 129
K,L=  0,  8
-12 109  99
-11 214 216
-10  27  15*
 -9 191 189
 -8 113 120
 -7 262 262
 -6  65  67
 -5 320 322
 -4 144 146
 -3 304 311
 -2 575 574
 -1 478 502
  0 148 169
  1 476 468
  2 207 214
  3 181 180
  4 191 185
  5 124 123
  6 147 150
  7 345 342
  8 337 331
  9  99 102
 10  56  55
 11 113 112
K,L=  0, 10
-11 125 119
-10   0  16*
 -9 193 197
 -8  62  67
 -7  24  36*
 -6  61  47
 -5 137 135
 -4 344 356
 -3 344 350
 -2 177 181
 -1 116 111
  0 466 459
  1 668 666
  2 183 183
  3 239 233
  4   0  14*
  5 134 135
  6 235 232
  7 174 168
  8 172 165
  9  83  61
 10 176 167
K,L=  0, 12
-11 188 192
-10 216 219
 -9  91 100
 -8 121 114
 -7 105 108
 -6  95  95
 -5 170 165
 -4 177 175
 -3  51  35
 -2 518 522
 -1 217 220
  0  31  46*
  1 161 138
  2 210 215
  3  23  22*
  4  54  49
  5 213 220
  6 166 159
  7 190 185
  8 205 200
  9 184 175
K,L=  0, 14
-10  52  61
 -9  76  84
 -8  93  97
 -7 130 138
 -6 129 131
 -5 106  99
 -4  35   6
 -3 105 110
 -2 332 333
 -1 270 272
  0 169 167
  1 180 172
  2 134 136
  3  88  83
  4 160 159
  5 114 108
  6  39  16
  7 211 209
  8 114 107
K,L=  0, 16
 -8 102 111
 -7  41  18
 -6  28  27*
 -5  47  35
 -4 283 296
 -3  53  23
 -2 115 119
 -1 182 179
  0 118 116
  1 135 140
  2  54  57
  3 139 132
  4   0  18*
  5  95  94
  6 233 227
K,L=  0, 18
 -6  30  28*
 -5  83  88
 -4  50  49
 -3  57  47
 -2 272 273
 -1 221 224
  0  40  50
  1 103  97
  2  58  56
  3  59  64
  4 119 110
K,L=  1,  0
  1  20  17
  2 355 349
  3 271 259
  4 117 113
  5 168 167
  6  85  89
  7 106 108
  8   0  14*
  9  58  64
 10  25   6*
 11  38  23
 12  40  58
 13   0   2*
K,L=  1,  1
-13  30  31*
-12  56  50
-11 131 126
-10 173 170
 -9  92  88
 -8 104 113
 -7 248 249
 -6 105 113
 -5  25  19
 -4 212 210
 -3 242 233
 -2  21   8
 -1 687 660
  0 496 478
  1 161 169
  2 478 490
  3 554 558
  4  78  79
  5  93  94
  6 257 262
  7  88  89
  8 191 185
  9 359 366
 10 223 220
 11  97  89
 12  69  57
 13  42  48
K,L=  1,  2
-13   0   9*
-12   0  30*
-11 103 102
-10  56  57
 -9 101 104
 -8 149 156
 -7  83  86
 -6 190 195
 -5 125 118
 -4 276 274
 -3 521 510
 -2 571 552
 -1 194 192
  0 647 622
  1  86  89
  2 302 296
  3 191 193
  4  43  45
  5 249 299
  6  62  62
  7 156 151
  8  39  48
  9   0  16*
 10  44  43
 11  27  21*
 12 107 102
K,L=  1,  3
-13  59  44
-12 132 123
-11 156 154
-10  62  50
 -9 307 313
 -8 401 411
 -7 143 150
 -6 121 121
 -5 337 340
 -4 313 310
 -3 556 546
 -2 913 894
 -1  97  91
  0  46  43
  1  25  29
  2 284 282
  3 206 204
  4 189 192
  5 141 144
  6  89  86
  7 117 124
  8 239 250
  9 127 130
 10   0  28*
 11 177 167
 12 149 164
K,L=  1,  4
-13  52  43
-12   0  16*
-11  38  33
-10  25   7*
 -9  24  18*
 -8  55  55
 -7  56  54
 -6  20   6*
 -5  75  72
 -4 265 262
 -3  96  91
 -2  95  90
 -1 151 149
  0 131 115
  1 194 187
  2 134 134
  3 110 109
  4  82  81
  5  19  18*
  6  58  68
  7  87  84
  8  84  88
  9   0  19*
 10  37  21
 11   0  13*
 12  71  78
K,L=  1,  5
-12  29  11*
-11  86  77
-10 131 132
 -9 206 205
 -8   0   2*
 -7 114 116
 -6 211 215
 -5   0   2*
 -4 262 260
 -3 110 107
 -2 201 194
 -1  33  23
  0 839 810
  1 448 444
  2  22  13
  3  70  74
  4 256 258
  5 247 252
  6 207 206
  7 221 228
  8  41  40
  9 179 176
 10 230 235
 11  39  30
 12  29  31*
K,L=  1,  6
-12 155 168
-11  61  63
-10 119 115
 -9  74  73
 -8  99 108
 -7 164 171
 -6  77  73
 -5 334 336
 -4 309 302
 -3 152 154
 -2 206 193
 -1 303 290
  0 289 271
  1 103  84
  2  79  68
  3 349 359
  4  87  93
  5 149 156
  6  83  75
  7 141 138
  8   0  26*
  9  51  40
 10 111 118
 11  49  47
 12  30  37*
K,L=  1,  7
-12 164 164
-11 189 194
-10  98  98
 -9  35  35
 -8 182 191
 -7 110 109
 -6  90  90
 -5  98  99
 -4 333 327
 -3 246 237
 -2 583 561
 -1  95  85
  0 284 273
  1 237 235
  2 484 496
  3 265 278
  4  28  10
  5  81  85
  6 271 273
  7 146 149
  8 198 202
  9 240 233
 10 194 189
 11  50  34
K,L=  1,  8
-12   0  15*
-11  40  33
-10  27  18*
 -9  25  22*
 -8 116 120
 -7   0  10*
 -6   0   6*
 -5 148 141
 -4  40  31
 -3 300 294
 -2  89  71
 -1  60  47
  0  41  20
  1 190 184
  2 188 190
  3 206 208
  4   0   8*
  5 109 117
  6  97  97
  7  54  58
  8  84  78
  9  38  21
 10  28  30*
 11  29  13*
K,L=  1,  9
-12  79  70
-11  49  55
-10  94  95
 -9 124 125
 -8 135 148
 -7   0  11*
 -6 119 121
 -5 242 242
 -4  80  86
 -3 306 296
 -2 461 471
 -1 208 196
  0 393 388
  1 197 201
  2 150 152
  3 152 150
  4 277 281
  5  32  36
  6 171 174
  7 180 190
  8  64  64
  9 115 117
 10  94  91
 11 107  96
K,L=  1, 10
-11  58  68
-10   0  12*
 -9  65  62
 -8  25  23*
 -7  42  44
 -6  33  27
 -5 124 126
 -4 272 266
 -3 103 114
 -2  65  37
 -1  29  27
  0 165 158
  1 202 204
  2  36  31
  3  87  90
  4 146 148
  5  62  63
  6 204 203
  7  62  64
  8 199 200
  9  48  52
 10  41  34
K,L=  1, 11
-11 144 139
-10 116 122
 -9 173 180
 -8 110 109
 -7  83  84
 -6   0  27*
 -5  69  73
 -4 352 349
 -3 167 168
 -2 369 368
 -1 282 275
  0 211 204
  1 254 255
  2  96 101
  3  93  99
  4   0   8*
  5  84  93
  6 263 261
  7 170 180
  8  95  93
  9 110  99
 10 129 127
K,L=  1, 12
-11   0   2*
-10  64  56
 -9  39  17
 -8   0   6*
 -7 185 188
 -6  82  80
 -5  64  65
 -4  47  44
 -3  97  89
 -2  96  71
 -1   0   5*
  0   0  27*
  1  64  31
  2  80  88
  3 136 140
  4  42  52
  5 104  96
  6 163 163
  7  76  72
  8 101 110
  9  51  60
K,L=  1, 13
-10   0   4*
 -9 150 153
 -8 109  97
 -7 149 147
 -6 156 157
 -5 176 179
 -4 116 125
 -3 122 119
 -2 381 379
 -1 190 186
  0 156 155
  1 138 141
  2   0  15*
  3  65  66
  4 131 136
  5  37  33
  6  27  19*
  7  83  72
  8 175 175
  9   0  15*
K,L=  1, 14
 -9  50  65
 -8  84  87
 -7   0   8*
 -6  65  60
 -5  51  55
 -4 136 127
 -3  74  75
 -2 101  83
 -1  49  41
  0  42  31
  1 140 149
  2 109 117
  3  57  63
  4  45  37
  5  93  93
  6 114 114
  7  95  90
  8  30  40*
K,L=  1, 15
 -9 107 114
 -8  29  28*
 -7 149 151
 -6  86  77
 -5  53  60
 -4 128 124
 -3 244 242
 -2  26   9*
 -1  51  39
  0 163 153
  1 168 165
  2  37  30
  3 103 105
  4  66  68
  5  28  13*
  6 134 139
  7 184 184
K,L=  1, 16
 -8   0  12*
 -7  58  63
 -6  56  54
 -5   0  29*
 -4 206 218
 -3 128 114
 -2 157 144
 -1  46  29
  0  79  89
  1  75  68
  2  47  52
  3  27  15*
  4  49  52
  5  41  34
  6  51  58
K,L=  1, 17
 -7  66  63
 -6  29  13*
 -5 124 120
 -4  63  52
 -3 152 149
 -2 184 170
 -1 248 246
  0  27  10*
  1  48  41
  2  97 110
  3  28  13*
  4  50  36
  5  98  87
K,L=  1, 18
 -6  30  17*
 -5  51  48
 -4   0  16*
 -3 115 117
 -2  40   5
 -1  80  62
  0  49  44
  1   0  20*
  2  82  92
  3 106 114
K,L=  1, 19
 -4   0  19*
 -3  98  91
 -2 150 141
 -1 106  85
  0  42  52
  1 157 153
K,L=  2,  0
  0 237 235
  1 121 119
  2 302 293
  3 381 371
  4 282 274
  5 308 302
  6 345 348
  7 144 149
  8   0  30*
  9 103 105
 10 145 134
 11  27  30*
 12   0   8*
K,L=  2,  1
-12 121 128
-11  77  79
-10  26  30*
 -9 111 109
 -8  32  29
 -7  70  70
 -6 152 154
 -5 397 397
 -4 140 138
 -3 213 217
 -2  81  81
 -1 721 711
  0 150 140
  1 470 470
  2 468 459
  3 266 268
  4 256 264
  5 288 295
  6  75  75
  7  21  19*
  8  86  92
  9   0  14*
 10  63  65
 11  67  64
 12   0  15*
K,L=  2,  2
-12  81  73
-11 115 114
-10  99 103
 -9   0   5*
 -8 318 312
 -7 143 146
 -6 196 197
 -5 270 277
 -4 334 336
 -3  93  91
 -2 125 124
 -1  67  65
  0 144 138
  1  89  87
  2 498 504
  3  27  16
  4  17  20*
  5 182 189
  6 257 263
  7  37  33
  8 139 143
  9 141 142
 10   0   4*
 11 106 105
 12   0  89*
K,L=  2,  3
-12 131 131
-11  38  28
-10 141 127
 -9  64  60
 -8 119 120
 -7 251 250
 -6 112 119
 -5 220 217
 -4 227 234
 -3  45  44
 -2 312 307
 -1  51  42
  0 238 245
  1 313 312
  2 275 279
  3  33  36
  4 209 216
  5 304 296
  6 204 203
  7 110 114
  8 190 194
  9  35  33
 10 114 116
 11  62  62
 12 133 130
K,L=  2,  4
-12   0  11*
-11   0   6*
-10  26   7*
 -9 109 113
 -8 146 141
 -7  69  60
 -6 374 370
 -5 231 237
 -4 373 370
 -3  97 102
 -2  56  57
 -1 167 167
  0 483 480
  1 352 357
  2  97  97
  3 260 255
  4 544 556
  5  90  85
  6  76  76
  7  50  49
  8 231 227
  9  25   9*
 10  88  84
 11 108 108
 12  78  82
K,L=  2,  5
-12   0   7*
-11 140 143
-10  45  41
 -9 105 105
 -8 121 120
 -7  88  84
 -6 248 254
 -5 295 297
 -4 324 313
 -3  71  71
 -2 208 200
 -1  16  11*
  0  22  24
  1 191 190
  2 223 219
  3  89  87
  4  76  80
  5 260 264
  6 349 352
  7  32  37
  8  76  76
  9  25  25*
 10  38  28
 11  85  83
 12  98 112
K,L=  2,  6
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-11 146 155
-10  59  59
 -9  94  93
 -8 119 116
 -7 273 266
 -6  21  11*
 -5 112 110
 -4 292 297
 -3 314 301
 -2  87  86
 -1  76  71
  0 158 154
  1 187 183
  2 175 176
  3 328 335
  4 120 118
  5  21  18*
  6 310 318
  7  84  88
  8  74  71
  9  69  65
 10 102  97
 11  91  85
K,L=  2,  7
-12  41  33
-11  48  41
-10  38  34
 -9  84  73
 -8 149 147
 -7 152 156
 -6 252 252
 -5   0  14*
 -4 236 224
 -3  82  77
 -2 286 262
 -1  57  56
  0  62  56
  1  73  76
  2 256 261
  3 175 175
  4 209 221
  5 207 212
  6 116 118
  7  76  70
  8  94  89
  9   0  15*
 10 104  92
 11   0   6*
K,L=  2,  8
-12   0  11*
-11  28  29*
-10   0   6*
 -9 103 106
 -8 177 186
 -7 137 136
 -6 112 111
 -5 154 149
 -4 116 111
 -3 260 256
 -2 216 217
 -1  98  94
  0 148 157
  1 175 180
  2 344 349
  3 149 149
  4 209 216
  5 300 309
  6  47  49
  7  78  78
  8  77  76
  9  85  80
 10  28  13*
 11  42  49
K,L=  2,  9
-11  64  65
-10  39  30
 -9  64  54
 -8 112 118
 -7 127 129
 -6  92  89
 -5 117 120
 -4 329 335
 -3 225 226
 -2  73  67
 -1  28  15
  0 225 221
  1 229 242
  2 383 388
  3 111 118
  4  44  40
  5 148 141
  6 130 135
  7 147 138
  8  26   6*
  9  62  58
 10   0  13*
K,L=  2, 10
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 -8  26  18*
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 -6 164 163
 -5 116 120
 -4 117 110
 -3 222 224
 -2 114 103
 -1 207 211
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  1 147 154
  2 140 153
  3 149 159
  4 300 314
  5   0   9*
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  7 144 153
  8 101 109
  9  80  69
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K,L=  2, 11
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 -8  53  47
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 -6 264 261
 -5 281 292
 -4 126 126
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 -2  73  72
 -1  38  24
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  1 115 114
  2  23   7*
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  4 131 135
  5 135 132
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  7   0   9*
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K,L=  2, 12
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 -7 173 178
 -6   0   3*
 -5 183 194
 -4 160 166
 -3   0   6*
 -2 272 268
 -1 119 118
  0   0   8*
  1  80  79
  2 147 156
  3 102 110
  4   0   4*
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K,L=  2, 13
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 -7 197 197
 -6 104 113
 -5  25  20*
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 -3 157 153
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 -1  89  93
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  1  42  56
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  3 213 208
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  5  53  54
  6 133 130
  7  63  68
  8   0  17*
K,L=  2, 14
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 -3 273 267
 -2 130 128
 -1  25  23*
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  2  72  66
  3  58  48
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  5  72  62
  6  49  45
  7  77  76
  8  73  70
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 -6  55  57
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  5  74  62
  6  71  62
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K,L=  2, 16
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  4   0   9*
  5   0   7*
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K,L=  2, 18
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 -3   0   6*
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K,L=  3,  0
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 -5  20   6*
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 -3 398 395
 -2 443 435
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  1 242 244
  2 453 455
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K,L=  3,  2
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  2 169 165
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  2 100 102
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  6 182 185
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K,L=  3,  5
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 -2 139 138
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  2  18  15*
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 -7 175 173
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 -1 130 132
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  1 186 186
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  5 477 483
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 -5 324 318
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  2 170 169
  3  21  28*
  4  21  19*
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  6 176 181
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 -1 175 175
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  1 128 127
  2 158 163
  3 275 282
  4 210 215
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  6 194 196
  7 140 134
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 -6 129 130
 -5 197 201
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 -2  82  88
 -1 136 138
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  2 300 307
  3 166 164
  4 204 202
  5 217 228
  6 111 112
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K,L=  3, 11
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  1  56  63
  2 219 221
  3 131 124
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  5  98 103
  6 134 141
  7  86  73
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  9  29  34*
K,L=  3, 12
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 -6 238 237
 -5 159 158
 -4  49  51
 -3 154 156
 -2 130 133
 -1  95  91
  0 136 136
  1  93 108
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K,L=  3, 13
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  0  60  46
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  2 161 166
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  4 126 134
  5 126 137
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  8  30  33*
K,L=  3, 14
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 -7 168 179
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 -5 113 101
 -4 146 148
 -3  78  73
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  2 179 182
  3 143 146
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  5  96 102
  6  64  65
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K,L=  3, 15
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 -6  84  73
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  2  27  13*
  3  39  32
  4  74  72
  5  64  69
  6 102 107
K,L=  3, 16
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 -5 114 115
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 -2  27  40*
 -1   0   5*
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  1 138 143
  2 142 142
  3  28   7*
  4 139 131
  5 129 120
K,L=  3, 17
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 -4  82  91
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 -2   0   8*
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  1  29  24*
  2 124 123
  3 131 136
  4  52  42
K,L=  3, 18
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  1  30  43*
  2  73  70
K,L=  4,  0
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  3 135 139
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  5 113 116
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 10   0   7*
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 12   0  25*
K,L=  4,  1
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 -7 119 120
 -6 286 297
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 -3 387 380
 -2 397 405
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  2 242 247
  3  47  52
  4 531 538
  5 332 337
  6  31  22
  7 170 173
  8  74  62
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 11  49  47
 12   0   5*
K,L=  4,  2
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-10  47  41
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 -8  49  56
 -7   0   5*
 -6 201 198
 -5 113 118
 -4  28  36
 -3 332 339
 -2 185 184
 -1 187 187
  0 157 161
  1 127 124
  2  79  81
  3 247 247
  4  73  73
  5  21  14*
  6 232 223
  7 106 113
  8  25  15*
  9  37  42
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 11  29  27*
K,L=  4,  3
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-11   0  13*
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 -9 127 123
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 -6 291 289
 -5  21  14*
 -4 291 291
 -3 402 402
 -2 138 134
 -1 192 197
  0 197 201
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  2 275 279
  3 227 237
  4 115 104
  5  31  45
  6 274 275
  7 179 182
  8  43  27
  9 115 112
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K,L=  4,  4
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 -6  75  83
 -5 154 153
 -4  29  26
 -3  63  62
 -2 136 138
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  2  28  27
  3  81  80
  4 124 126
  5  31  28
  6 124 124
  7  54  54
  8  51  44
  9  27  17*
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 11   0   5*
K,L=  4,  5
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 -8 208 209
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 -6 102  99
 -5 305 310
 -4 390 400
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 -2 260 264
 -1 181 186
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  2 217 223
  3 174 176
  4 118 116
  5 245 245
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  9  85  78
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K,L=  4,  6
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 -6   0  16*
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 -4 235 234
 -3 183 183
 -2 148 150
 -1 100 102
  0 164 168
  1 150 150
  2 101 110
  3 153 155
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  5  32  38
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  8  52  49
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K,L=  4,  7
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 -7  60  63
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 -5 220 232
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 -3 121 125
 -2 186 194
 -1  78  73
  0 119 128
  1 219 227
  2  48  42
  3 237 246
  4 261 270
  5 105 103
  6 162 165
  7 199 197
  8  75  73
  9 104 102
 10 186 185
K,L=  4,  8
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K,L=  4,  9
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 -7 168 169
 -6  49  40
 -5  86  91
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 -3 189 186
 -2 165 165
 -1 144 137
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  3 317 324
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  6 206 203
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K,L=  4, 10
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 -7 120 118
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K,L=  4, 11
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  1 215 215
  2 129 127
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  6  38  31
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  8 123 122
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  5  27  17*
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K,L=  4, 13
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 -8   0   4*
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 -6 169 163
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K,L=  4, 15
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  2 139 138
  3 116 118
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K,L=  4, 16
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K,L=  4, 17
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  1 128 129
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K,L=  5,  0
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  3 153 151
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  5 108 109
  6  24  18*
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  9 135 140
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 -4 145 148
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 -2 151 151
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  2 169 172
  3 160 155
  4 147 144
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K,L=  5,  3
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  3 184 183
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  9  27   2*
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K,L=  5,  4
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 -7 139 140
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 -2 184 193
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  2 212 216
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  5 133 136
  6 131 134
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  9 179 176
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  8  27  13*
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K,L=  5,  6
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 -8 130 121
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 -4  56  62
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 -2 235 229
 -1  65  71
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  1 224 227
  2  86  93
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  5 137 134
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  7 114 119
  8 159 153
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  3  23  35*
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K,L=  5,  8
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 -1 130 126
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  1 222 227
  2 157 153
  3 140 140
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  4  85  92
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  7 146 146
  8  40  34
  9 168 155
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 -6  90  97
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 -1  92  97
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  1 104 106
  2  24  22*
  3   0  14*
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  6   0  22*
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  8  86  82
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 -6  26  19*
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 -2 206 208
 -1 211 213
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  1 172 175
  2 165 162
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  4 140 135
  5 113 113
  6   0   7*
  7  40  27
  8 101  93
K,L=  5, 11
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 -8  29  32*
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 -3  44  36
 -2  71  68
 -1  50  45
  0  79  79
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K,L=  5, 12
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 -2 183 177
 -1  78  88
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  2  26  33*
  3  89 104
  4   0  14*
  5  89  83
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  7 122 122
K,L=  5, 13
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 -6   0   9*
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  1 107 107
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  4   0  10*
  5   0   9*
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 -4 122 117
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  2  48  51
  3  69  67
  4   0   4*
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 -4 104 105
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 -2  28  31*
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  1  70  68
  2  58  51
  3   0  14*
  4   0  24*
K,L=  5, 16
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 -2 157 159
 -1 101 102
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  1  88  94
  2  89  94
K,L=  6,  0
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  1  54  56
  2  32  34
  3 105 110
  4 115 119
  5  34  47
  6  90  89
  7  37  25
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  9  53  55
 10  41  46
K,L=  6,  1
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 -7  37  28
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 -5 116 115
 -4  41  39
 -3 157 152
 -2 280 269
 -1 123 121
  0 293 290
  1 384 393
  2 185 189
  3 119 111
  4   0  12*
  5  81  84
  6 118 114
  7 114 101
  8   0  19*
  9  49  52
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K,L=  6,  2
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 -9 109 111
 -8 141 138
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 -6   0   1*
 -5  64  63
 -4  41  37
 -3  23  15*
 -2 207 217
 -1 130 132
  0   0  27*
  1 116 109
  2  23  23*
  3  33  38
  4  82  82
  5 127 133
  6 179 177
  7  95  99
  8 119 125
  9  40  35
 10  59  59
K,L=  6,  3
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 -9 123 112
 -8  27  11*
 -7  78  86
 -6 129 129
 -5   0  22*
 -4 164 163
 -3  52  54
 -2  75  73
 -1 316 318
  0 309 311
  1 189 203
  2  65  67
  3 146 148
  4  41  53
  5  89  84
  6 234 233
  7 109 105
  8  95  88
  9 164 157
 10 107  97
K,L=  6,  4
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 -7   0   9*
 -6  25  10*
 -5  99  98
 -4  24  40*
 -3 110 114
 -2  69  73
 -1  56  50
  0 242 247
  1  23  26*
  2   0  14*
  3  24  38*
  4  34  35
  5   0   8*
  6  52  40
  7  71  65
  8 100 101
  9  29   4*
K,L=  6,  5
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 -9 103 107
 -8 228 227
 -7  70  71
 -6 118 117
 -5  43  40
 -4  42  43
 -3 196 191
 -2 240 239
 -1 212 211
  0  57  55
  1 125 124
  2 173 177
  3 102  97
  4  49  42
  5  57  50
  6  26  17*
  7  98  93
  8 208 199
  9 152 150
K,L=  6,  6
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 -9  29  17*
 -8 144 153
 -7  27   7*
 -6   0  23*
 -5  91  92
 -4 156 154
 -3  24  30*
 -2 111 113
 -1 105 108
  0 127 130
  1  24  24*
  2 134 132
  3   0  15*
  4  83  81
  5  26  12*
  6 146 148
  7  39  30
  8  28  21*
  9  98  87
K,L=  6,  7
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 -8  74  68
 -7   0   8*
 -6  46  36
 -5  96 104
 -4 106 110
 -3 256 250
 -2 128 136
 -1  24  26*
  0 224 213
  1 339 342
  2 130 125
  3  71  75
  4  63  62
  5  37  24
  6 183 175
  7 214 203
  8   0   7*
K,L=  6,  8
 -9  66  55
 -8  28  28*
 -7   0  18*
 -6  60  54
 -5  26   7*
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 -2 214 216
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  0  35  36
  1  61  59
  2 112 114
  3  25  22*
  4  52  53
  5   0   9*
  6  67  70
  7   0   7*
  8  97  95
K,L=  6,  9
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 -8  58  58
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 -6  27  37*
 -5  88  88
 -4 154 152
 -3  36  34
 -2 124 118
 -1 266 269
  0 208 201
  1  67  73
  2 149 152
  3  94  92
  4  60  63
  5  55  44
  6  97  98
  7  82  79
  8 123 110
K,L=  6, 10
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 -7  75  69
 -6   0  12*
 -5  47  45
 -4  46  45
 -3  83  87
 -2  26  27*
 -1  26   7*
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  1  73  75
  2  37  39
  3  27  52*
  4  39  42
  5  28  24*
  6  76  72
  7 106  98
K,L=  6, 11
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 -6  40  45
 -5 121 128
 -4  38  31
 -3 197 194
 -2 291 299
 -1 150 153
  0  37   9
  1 107  98
  2 159 159
  3  72  80
  4  92  92
  5  40  30
  6  72  64
K,L=  6, 12
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 -6  76  74
 -5  57  59
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 -3  39  29
 -2 102  99
 -1 118 121
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  1  27   5*
  2  87  83
  3  49  44
  4  49  51
  5  88  74
  6  60  60
K,L=  6, 13
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 -5  50  35
 -4  70  73
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 -2  40  31
 -1 104 100
  0 158 157
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  2  49  48
  3   0  22*
  4  65  63
  5  52  47
K,L=  6, 14
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 -4   0  25*
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  1  76  89
  2  29  27*
  3   0   8*
K,L=  6, 15
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 -1 118 119
  0  83  82
  1   0   7*
  2  60  71
K,L=  7,  0
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  3 177 187
  4  36  24
  5  26  26*
  6  46  43
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  8 148 144
  9  59  57
K,L=  7,  1
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 -8  70  74
 -7  39  34
 -6 125 132
 -5  52  48
 -4 258 249
 -3 136 139
 -2  49  58
 -1 108 101
  0 230 227
  1 111 111
  2 132 130
  3  25  18*
  4 174 175
  5 180 181
  6 272 270
  7  48  41
  8  57  62
  9  30  24*
K,L=  7,  2
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 -8  75  66
 -7  28  22*
 -6 149 145
 -5 119 116
 -4 174 176
 -3 261 257
 -2  42  51
 -1 191 195
  0 245 249
  1 175 184
  2 101 100
  3  35  27
  4 111 109
  5  26   4*
  6 126 120
  7  39  36
  8  29  13*
  9  30  22*
K,L=  7,  3
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 -8  81  83
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 -4  51  40
 -3  50  45
 -2 139 137
 -1  69  74
  0 144 137
  1 207 211
  2  25   9*
  3  56  61
  4 103 104
  5  37  44
  6   0  21*
  7  63  56
  8 112 108
K,L=  7,  4
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 -8  50  26
 -7 133 142
 -6  27  17*
 -5  64  65
 -4  36  14
 -3  56  50
 -2 114 116
 -1 126 128
  0 144 137
  1  61  60
  2  25  17*
  3  84  92
  4  58  72
  5   0   8*
  6  47  53
  7  28   8*
  8 162 165
K,L=  7,  5
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 -8  96  94
 -7   0   9*
 -6  27  18*
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 -4 193 191
 -3 209 203
 -2  50  52
 -1  35  15
  0 201 193
  1  75  72
  2  91  95
  3 118 113
  4 186 179
  5  74  76
  6 202 200
  7 103  99
  8   0   4*
K,L=  7,  6
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 -6  28  32*
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 -2 155 159
 -1  95  96
  0  36  23
  1 140 145
  2  96 102
  3  45  42
  4  65  65
  5   0  12*
  6  93  96
  7 100  97
  8  84  68
K,L=  7,  7
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 -3 139 135
 -2  45  33
 -1 191 195
  0  58  43
  1 122 125
  2 172 170
  3  38  40
  4  38  46
  5  96  89
  6 145 146
  7  59  56
K,L=  7,  8
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 -7   0  12*
 -6   0   9*
 -5   0   1*
 -4  27   8*
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 -2  26   1*
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  0 141 140
  1 169 167
  2  75  78
  3  38  39
  4  87  88
  5  85  79
  6 164 159
  7  52  41
K,L=  7,  9
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 -6  95  87
 -5 166 163
 -4  55  58
 -3 100 182
 -2  85  88
 -1 100  99
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  1 227 230
  2  27  30*
  3  83  80
  4 161 159
  5  50  51
  6  59  58
K,L=  7, 10
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 -6   0  14*
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 -4  40  36
 -3  78  72
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 -1 125 132
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  1  48  43
  2  28  13*
  3   0   6*
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  5  83  74
K,L=  7, 11
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 -5  29  15*
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 -3  69  65
 -2  28  29*
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  4  41  37
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K,L=  7, 12
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 -3 104 105
 -2  76  78
 -1  57  45
  0  70  45
  1 119 117
  2  87  84
  3  29  29*
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K,L=  7, 13
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  1  29  34*
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K,L=  8,  0
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  6 106 112
  7  41  28
K,L=  8,  1
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 -6  28  13*
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 -4   0   3*
 -3 113 112
 -2  46  38
 -1   0   8*
  0  52  10
  1  69  61
  2  26   7*
  3   0  16*
  4  27  12*
  5  48  37
  6  64  58
  7   0  10*
K,L=  8,  2
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 -6  40  38
 -5 198 186
 -4 133 132
 -3  54  42
 -2 102 107
 -1  79  85
  0  69  68
  1  64  70
  2 150 155
  3   0  13*
  4  86  82
  5 128 129
  6 143 139
  7   0   1*
K,L=  8,  3
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 -6  79   7*
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 -4  61  51
 -3  27  23*
 -2  27  28*
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  1  53  53
  2   0   6*
  3  54  51
  4   0  19*
  5 132 133
  6  41  43
  7  30   3*
K,L=  8,  4
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 -6 188 186
 -5 148 146
 -4  39  43
 -3 184 186
 -2  80  78
 -1  38  42
  0  59  43
  1  60  63
  2  93  87
  3 109 108
  4 208 204
  5  75  58
  6  29  14*
  7  60  51
K,L=  8,  5
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 -6  50  37
 -5  63  58
 -4  55  51
 -3  61  70
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 -1  27  26*
  0  38  27*
  1  27  11*
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  3  67  72
  4  49  44
  5   0  17*
  6  59  49
K,L=  8,  6
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 -6  29   5*
 -5  49  45
 -4 153 150
 -3  73  66
 -2  77  81
 -1  72  74
  0  82  76
  1  27  36*
  2 110 107
  3  62  58
  4 106 111
  5  41  28
  6 228 230
K,L=  8,  7
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 -5  41  27*
 -4  28  30*
 -3  48  42
 -2 104 109
 -1  39  25
  0  73  79
  1   0  10*
  2   0   3*
  3  40  37
  4  41  37
  5  78  76
K,L=  8,  8
 -6 166 166
 -5 134 136
 -4  86  85
 -3  70  68
 -2 129 136
 -1  49  56
  0  28  10*
  1 105 101
  2  90  88
  3 100  99
  4 134 139
  5  60  62
K,L=  8,  9
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 -4  41  32
 -3  29  25*
 -2  57  58
 -1  28  25*
  0  75  64
  1   0   7*
  2   0  17*
  3  65  65
  4  73  60
K,L=  8, 10
 -4  89  86
 -3 152 151
 -2   0   5*
 -1   0  29*
  0 133 139
  1  29  41*
  2  88  84
  3 136 133
K,L=  8, 11
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 -2  30  17*
 -1  51  56
  0  59  64
  1  42  47
K,L=  9,  0
  1  28  25*
  2  28  22*
  3  99 108
  4  29  19*
  5  42  34
K,L=  9,  1
 -5  51  50
 -4 217 218
 -3 107 106
 -2  75  72
 -1  49  57
  0  28  13*
  1   0  16*
  2 113 110
  3 131 131
  4  50  54
  5 126 128
K,L=  9,  2
 -5  30  21*
 -4  71  73
 -3  29  27*
 -2  70  73
 -1   0   5*
  0  28  38*
  1  28  20*
  2  70  61
  3 135 125
  4  29  30*
  5  66  63
K,L=  9,  3
 -5 173 163
 -4  97 103
 -3  41  33
 -2 148 143
 -1  28  13*
  0  80  76
  1  40  42
  2  57  58
  3  29  22*
  4 117 125
  5 140 134
K,L=  9,  4
 -5  73  77
 -4  29  21*
 -3  71  72
 -2  41  42
 -1  40  42
  0  70  57
  1   0   4*
  2   0  10*
  3  71  71
  4   0   9*
K,L=  9,  5
 -4 118 118
 -3 117 113
 -2  29  25*
 -1   0   5*
  0  81  84
  1  29  33*
  2  92  87
  3 110 106
  4 115 118
K,L=  9,  6
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 -3  29  24*
 -2   0  13*
 -1   0  16*
  0  77  79
  1   0  11*
  2  42  43
  3  84  81
K,L=  9,  7
 -3  89  89
 -2  93  99
 -1  42  38
  0  29  15*
  1  29  30*
  2 149 149
K,L=  9,  8
 -2  30  16*
 -1  30  12*
  0  52  43
  1  42  41
```

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[a] Zero-weighted data are indicated with asterisks.

parts of the anomalous dispersion for zinc and chlorine[11] 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. A value of $p = 0.04$ was found necessary to reduce the weights of the intense reflections so that their weighted residuals were comparable to those of the less intense data. A value of $q = 12$, which represents 1.2 counts/sec uncertainty in the backgrounds, was used to bring the weighted residuals of the very weak reflections into line with the others.

Cell dimensions were obtained from a set of Weissenberg photographs. The monoclinic unit cell contains four formula units and has cell dimensions $a = 13.76 \pm 0.04$ Å, $b = 10.33 \pm 0.03$ Å, $c = 20.35 \pm 0.06$ Å, and $\beta = 95.0 \pm 0.2°$. The errors quoted are subjective estimates. The absent reflections are consistent with space group $P2_1/c$. The density calculated from the X-ray data is 1.304 g/cm³. The crystals were observed to float readily in carbon tetrachloride ($d = 1.58$ g/cm³).

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 nonhydrogen 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 $\exp(-\beta_{11}h^2 - \beta_{22}k^2 - \beta_{33}l^2 - 2\beta_{12}hk - 2\beta_{13}hl - 2\beta_{23}\cdot kl)$. In reporting the thermal parameters below we have converted β_{ij} to B_{ij} which is in units of square ångströms; 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. The largest nonhydrogen peak had a peak height of 0.33 e⁻/Å³ and was the 36th in size on the difference map.

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 two cycles of refinement, the scheme was changed and the parameters of all 37 heavy atoms were refined in one pass; 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 nonzero 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 II

POSITIONAL PARAMETERS OF THE NONHYDROGEN ATOMS[a]

ATOM	X	Y	Z
ZN	.15353(4)	.05548(6)	.09866(3)
CL	.0476(1)	-.1178(1)	.08697(7)
N1	.2522(3)	.2384(4)	.1079(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)

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

TABLE III

ANISOTROPIC THERMAL PARAMETERS[a]

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)	4.60(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)

[a] Units of these parameters are square ångströms as described in the text.

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 threefold symmetry of the coordinated tren molecule neatly coiled about the zinc atom (Figure 1). The zinc atom lies 0.38 Å out of the plane of the three nitrogen atoms (N2, N3, and N4) toward the chlorine

[11] D. T. Cromer, *Acta Crystallogr.*, **18**, 17 (1965).

TABLE IV
POSITIONAL AND THERMAL PARAMETERS OF THE HYDROGEN ATOMS[a]

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)

[a] Units of *B* are square ångströms.

atom. The Cl–Zn–Ni 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, 2.223–2.358 Å, reported in the $[Co(NH_3)_6][ZnCl_4]Cl$ structure.[12]

Some of the dimensions of the trenZn 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 probably due to packing conditions. Similar deviations may be observed in the structures of complexes of tren with Cu[5] and Co.[6]

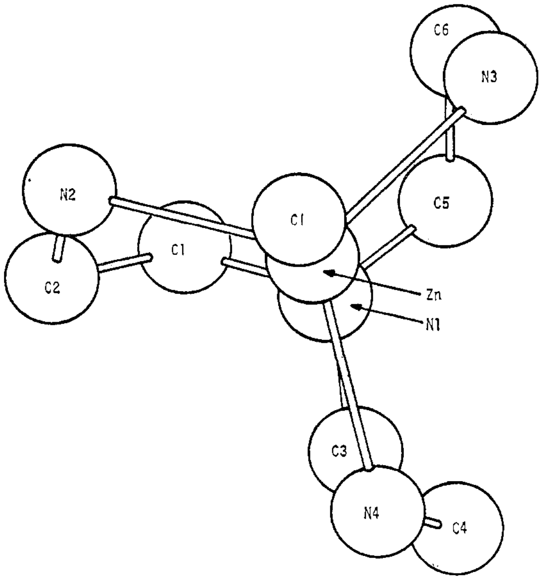

Figure 1.—The $Zn(C_2H_4NH_2)_3NCl$ cation drawn to indicate the nearly threefold nature of the cluster.

TABLE V
INTERATOMIC DISTANCES (Å) AND ANGLES (DEG) IN THE $Zn(C_2H_4NH_2)_3NCl^+$ ION

Distances			
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)
Angles			
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 $(C_6H_5)_4B^-$

Distances		Distances	
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)		
C21–C26	1.382 (7)	Angles	
C22–C23	1.381 (8)	C11–B–C21	102.9 (5)
C23–C24	1.358 (9)	C11–B–C31	111.3 (5)
C24–C25	1.369 (9)	C11–B–C41	111.5 (5)
C25–C26	1.370 (8)	C21–B–C31	115.1 (5)
C31–C32	1.386 (7)	C21–B–C41	112.1 (5)
C31–C36	1.399 (7)	C31–B–C41	104.2 (4)

TABLE VII
HYDROGEN INTERATOMIC DISTANCES (Å)[a]

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

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

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 Figure 2.

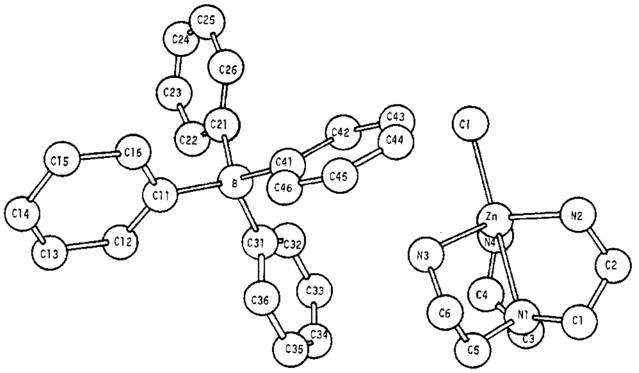

Figure 2.—Structure of the anion and its relation to one of its cation neighbors.

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 pseudocubic pseudocell 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. The closest approach of the two ions is a distance of 2.56 Å between a carbon atom (C43) of the anion and a hydrogen atom (H1N3) of the cation.

Table VII shows 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 0.1 Å from the generally accepted values for the internuclear distances.

CONTRIBUTION FROM THE LABORATORIO DI TEORIA E STRUTTURA ELETTRONICA E COMPORTAMENTO
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The Crystal and Molecular Structure of the Adduct of Bis(diphenyldithiophosphinato)nickel(II) with Pyridine

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The crystal structure of the adduct of bis(diphenyldithiophosphinato)nickel(II) with pyridine, $Ni[S_2P(C_6H_5)_2]_2 \cdot 2py$, has been determined by three-dimensional single-crystal X-ray analysis. Fourier methods were applied and the positional and isotropic thermal parameters of the atoms were refined by least-squares methods on three-dimensional photographic data. The compound forms monoclinic crystals, space group $P2_1/c$, with two molecules in the unit cell, whose dimensions, determined using zinc oxide as the standard, are $a = 12.38 \pm 0.02$ Å, $b = 8.98 \pm 0.01$ Å, $c = 15.97 \pm 0.02$ Å, and $\beta = 106.95 \pm 0.08°$. The measured density (by flotation) is 1.394 ± 0.006 g/cm^3 while that calculated for two formula units is 1.399 ± 0.008 g/cm^3. The nickel atom lies at a center of symmetry on the plane formed by the four sulfur atoms and completes its coordination sphere with the nitrogen atoms of two pyridine molecules. The average Ni–S distance is 2.50 (2) Å and the Ni–N bond length is 2.08 (1) Å. The final R factor is 0.104 for 1220 independent nonzero reflections. The correlation between Ni–S distances and the variations in the chelate rings are given and discussed for the tetracoordinated pyridine-free compound and the present octahedral pyridine adduct. The optical spectra are also given and discussed.

Introduction

Syntheses and spectral investigations of nickel complexes with molecules containing two sulfur atoms which may act as chelating ligands are at present being carried out in this laboratory. The crystal structure of the planar bis(diphenyldithiophosphinato)nickel(II) compound, $Ni[S_2P(C_6H_5)_2]_2$, hereafter $Ni(dtpi)_2$, has already been determined,[1] and it was thought interesting to carry out a single-crystal three-dimensional X-ray structure determination of the adduct of the above compound with two molecules of pyridine.

The purpose of this work was mainly to evaluate, with sufficient accuracy, the actual Ni–S bond distances and to compare them with the corresponding ones of the tetracoordinated chelate. Moreover, it would be interesting to observe the possible changes in the structure of the metal chelate caused by the completion of the coordination sphere of the metal atom with solvent molecules such as pyridine.

Experimental Section

Preparation of the Compound.—$Ni(dtpi)_2$ was prepared as described by Kuchen, *et al.*[2] Subsequently, green, prismatic crystals of bis(diphenyldithiophosphinato)nickel(II)-2-pyridine, $Ni[S_2P(C_6H_5)_2]_2 \cdot 2C_5H_5N$ (hereafter called $Ni(dtpi)_2 \cdot 2py$) were obtained by addition of pyridine to a solution of $Ni(dtpi)_2$ in toluene. The crystals, when exposed to air, slowly reverted to the original violet pyridine-free compound with loss of pyridine. In order to prevent decomposition, the crystals were enclosed in

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[1] P. Porta, A. Sgamellotti, and N. Vinciguerra, *Inorg. Chem.*, **7**, 2625 (1968).

[2] W. Kuchen, J. Metten, and A. Jadat, *Chem. Ber.*, **97**, 2306 (1964).