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# Refinement of the Crystal Structure of Rubidium Cyanodinitromethanide

HANS J. BJØRNSTAD and BERNT KLEWE*

*Department of Chemistry, University of Oslo, Oslo 3, Norway*

The crystals are orthorhombic, space group $P2_12_12_1$, with cell dimensions $a = 7.56_0$ Å, $b = 14.96_7$ Å, and $c = 5.09_8$ Å. 1129 X-ray reflections recorded on an automatic four-circle diffractometer were used in the full-matrix least squares refinement ($R_w = 4.8\%$, $R = 6.3\%$). The anion is not propeller shaped, the nitro groups being twisted 3° and 6° in opposite directions from a planar conformation.

Several crystal structure determinations and other studies of 1,1-dinitro compounds and dinitrocarbanions have been carried out; *cf.* Ref. 1 and references therein. Various physical and chemical properties of the $RC(NO_2)_2^-$ anions are sensitive to the steric requirements of R as discussed by Kaplan [2] for the substituents $NO_2$ and CN. The increased acidity of cyanodinitromethane relative to trinitromethane must be due to differences in anion stability. The substitution of the linear cyano group for a nitro group permits the cyanodinitromethanide ion to have an essentially planar conformation, whereas the trinitromethanide anion is non-planar.

X-Ray diffraction studies of the rubidium and the potassium salts of cyanodinitromethane were undertaken in this laboratory, using two- and three-dimensional photographic data, respectively. The potassium salt was independently investigated by Dr. James R. Holden, but as we had progressed further with the collection of data he discontinued his work on this compound. After the crystal structures had been determined, a diffractometer became available, and further refinement, using three-dimensional diffractometer data, was carried out. The structures had been determined when papers by Grigor'eva *et al.* on the same subject appeared.[3–5] The accuracy of their determinations was rather low, however, and a meaningful discussion of relevant structural data was scarcely possible. In the present paper our results from the refinement of the crystal structure of rubidium cyanodinitromethanide is reported. The numbering of atoms is similar to that of Grigor'eva *et al.* However, the atomic parameters, *etc.*, are different due to another choice of axes and origin.

* To whom correspondence should be addressed.

## EXPERIMENTAL

Rubidium cyanodinitromethanide was obtained in a metathetic reaction between the corresponding potassium salt and rubidium chloride in aqueous solution. To avoid potassium contamination the salt was repeatedly dissolved and precipitated from water by addition of rubidium chloride. The batch from which the crystal used in the X-ray experiments was taken was analyzed for potassium by means of flame photometry. The resulting potassium content of 0.84 % corresponds to a maximum atomic fraction (K/Rb) of 4.8 % in the crystal (if contaminated).

Rubidium cyanodinitromethanide crystallizes (from water) as slightly yellow plates or diamond shaped prisms in the orthorhombic space group $P2_12_12_1$. The cell dimensions and estimated standard deviations were determined on a manual four-circle diffractometer using Cu$K$ radiation. All diffractometer setting angles were optimized in the least squares refinement.

About 1700 reflections with $2\theta < 75°$ were registered on an automatic four-circle diffractometer using Mo$K\alpha$ radiation (0.002″ Nb filter) and $\omega/2\theta$ scan technique. A crystal of length 0.32 mm mounted with $b^*$ along the $\varphi$ axis of the diffractometer was used for the data collection. The (101) and (10$\bar{1}$) planes were well developed and the cross section was a parallelogram with diagonals of 0.36 and 0.23 mm. The effects of secondary extinction were greatly reduced by repeatedly dipping the crystal in liquid nitrogen. The intensities of several strong reflections were more than doubled after such a treatment. The intensities of the standard reflections showed an increase of about 2.5 % during the first 500 measurements, the reason probably being a reduction in crystal perfection. During the last part of the data collection two of the standard reflections were quite stable whereas the intensity of the third one was reduced by 13 %. 1195 reflections were regarded as observed having intensities greater than twice their estimated standard deviations from counting statistics. A 2 % uncertainty in scaling and diffractometer stability was included in the standard deviations. The data set obtained by using the standard reflections for scaling did not lead to significant differences in the atomic parameters compared with those from the non-scaled data set. The unscaled data set was eventually used.

The data were corrected for absorption. The linear absorption coefficient $\mu$ is 9.03 mm$^{-1}$ and the transmission factor varied between 0.13 and 0.29.

Computer programs used are described in Ref. 6.

The atomic form factors of Cromer and Waber [7] were applied. For the anomalous scattering factor for rubidium the values $\Delta f' = -0.8$ and $\Delta f'' = 3.0$ were obtained by extrapolation from the values calculated by Cromer and Liberman [8] for Cu, Zn, Ge, Br, and Kr.

## CRYSTAL DATA

Rubidium cyanodinitromethanide, $RbC_2N_3O_4$, F.W. 215.5. Slightly yellow plates or diamond shaped prisms, orthorhombic. $a = 7.560(3)$, $b = 14.967(5)$, $c = 5.098(2)$ Å, $V = 576.8$ Å$^3$. $F(000) = 404$; $Z = 4$; $\varrho_{obs} = 2.46$ g cm$^{-3}$, $\varrho_{calc} = 2.481$ g cm$^{-3}$; $\mu = 9.03$ mm$^{-1}$. Space group $P2_12_12_1$.

## STRUCTURE REFINEMENT

The atomic parameters determined earlier from two-dimensional film data by the heavy atom method were used as starting parameters in the full-matrix least squares refinements. The $R_w$ value of 9 % using isotropic temperature factors was lowered to 6 % when anisotropic temperature factor for rubidium was included. Anomalous scattering factor by rubidium was taken into account with the sign of $\Delta f''$ determined by calculations based on 400 reflections. Anisotropic thermal parameters for all atoms were then introduced. By inspection of the strong reflections, the secondary extinction

*Table 1.* Observed and calculated structure factors. (The five columns list values of $h$, $k$, $l$, 10 $F_o$, and 10 $F_c$.)

| h | k | l | Fo | Fc | h | k | l | Fo | Fc | h | k | l | Fo | Fc | h | k | l | Fo | Fc | h | k | l | Fo | Fc |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 0 | 2 | 0 | 319 | 284 | 5 | 14 | 0 | 102 | 98 | 0 | 15 | 1 | 167 | 157 | 5 | 5 | 1 | 316 | 319 | 11 | 5 | 1 | 86 | 81 |
| 0 | 4 | 0 | 1507 | 1623 | 5 | 15 | 0 | 105 | 109 | 0 | 17 | 1 | 138 | 129 | 5 | 6 | 1 | 433 | 451 | 11 | 7 | 1 | 64 | 53 |
| 0 | 8 | 0 | 561 | 524 | 5 | 17 | 0 | 78 | 80 | 0 | 18 | 1 | 162 | 167 | 5 | 7 | 1 | 287 | 296 | 11 | 8 | 1 | 92 | 84 |
| 0 | 10 | 0 | 115 | 101 | 5 | 18 | 0 | 49 | 39 | 0 | 19 | 1 | 58 | 63 | 5 | 8 | 1 | 225 | 232 | 11 | 9 | 1 | 63 | 64 |
| 0 | 12 | 0 | 512 | 483 | 5 | 19 | 0 | 62 | 64 | 0 | 20 | 1 | 63 | 60 | 5 | 9 | 1 | 243 | 252 | 11 | 11 | 1 | 71 | 53 |
| 0 | 14 | 0 | 86 | 33 | 5 | 20 | 0 | 57 | 38 | 0 | 21 | 1 | 68 | 57 | 5 | 10 | 1 | 339 | 347 | 11 | 12 | 1 | 67 | 63 |
| 0 | 16 | 0 | 393 | 388 | 6 | 0 | 0 | 423 | 426 | 1 | 0 | 1 | 541 | 528 | 5 | 11 | 1 | 219 | 228 | 12 | 0 | 1 | 75 | 65 |
| 0 | 20 | 0 | 237 | 224 | 6 | 1 | 0 | 507 | 529 | 1 | 1 | 1 | 677 | 665 | 5 | 12 | 1 | 65 | 66 | 12 | 2 | 1 | 58 | 56 |
| 1 | 1 | 0 | 262 | 276 | 6 | 2 | 0 | 93 | 94 | 1 | 2 | 1 | 821 | 830 | 5 | 13 | 1 | 163 | 157 | 12 | 4 | 1 | 76 | 63 |
| 1 | 2 | 0 | 108 | 113 | 6 | 3 | 0 | 509 | 533 | 1 | 3 | 1 | 305 | 300 | 5 | 14 | 1 | 217 | 220 | 12 | 5 | 1 | 62 | 50 |
| 1 | 3 | 0 | 438 | 481 | 6 | 4 | 0 | 292 | 291 | 1 | 4 | 1 | 981 | 984 | 5 | 15 | 1 | 128 | 131 | 0 | 0 | 2 | 391 | 368 |
| 1 | 4 | 0 | 255 | 276 | 6 | 5 | 0 | 356 | 361 | 1 | 5 | 1 | 1032 | 1035 | 5 | 17 | 1 | 100 | 100 | 0 | 1 | 2 | 1260 | 1231 |
| 1 | 5 | 0 | 594 | 652 | 6 | 7 | 0 | 283 | 289 | 1 | 6 | 1 | 571 | 564 | 5 | 18 | 1 | 117 | 118 | 0 | 2 | 2 | 336 | 294 |
| 1 | 6 | 0 | 526 | 570 | 6 | 8 | 0 | 258 | 262 | 1 | 7 | 1 | 419 | 413 | 6 | 0 | 1 | 290 | 301 | 0 | 3 | 2 | 824 | 747 |
| 1 | 7 | 0 | 561 | 602 | 6 | 9 | 0 | 236 | 238 | 1 | 8 | 1 | 406 | 392 | 6 | 1 | 1 | 328 | 342 | 0 | 4 | 2 | 291 | 265 |
| 1 | 8 | 0 | 134 | 137 | 6 | 10 | 0 | 37 | 32 | 1 | 9 | 1 | 506 | 492 | 6 | 2 | 1 | 96 | 95 | 0 | 5 | 2 | 892 | 805 |
| 1 | 9 | 0 | 518 | 557 | 6 | 11 | 0 | 151 | 155 | 1 | 10 | 1 | 413 | 408 | 6 | 3 | 1 | 158 | 159 | 0 | 6 | 2 | 78 | 71 |
| 1 | 10 | 0 | 327 | 345 | 6 | 12 | 0 | 129 | 125 | 1 | 11 | 1 | 442 | 435 | 6 | 4 | 1 | 248 | 248 | 0 | 7 | 2 | 488 | 446 |
| 1 | 11 | 0 | 248 | 256 | 6 | 13 | 0 | 183 | 185 | 1 | 12 | 1 | 213 | 211 | 6 | 5 | 1 | 317 | 327 | 0 | 8 | 2 | 209 | 201 |
| 1 | 12 | 0 | 82 | 70 | 6 | 15 | 0 | 175 | 178 | 1 | 13 | 1 | 166 | 162 | 6 | 6 | 1 | 105 | 105 | 0 | 9 | 2 | 554 | 500 |
| 1 | 13 | 0 | 268 | 270 | 6 | 16 | 0 | 81 | 76 | 1 | 14 | 1 | 227 | 229 | 6 | 7 | 1 | 55 | 48 | 0 | 11 | 2 | 389 | 367 |
| 1 | 14 | 0 | 221 | 226 | 6 | 17 | 0 | 126 | 112 | 1 | 15 | 1 | 192 | 199 | 6 | 8 | 1 | 194 | 190 | 0 | 12 | 2 | 54 | 37 |
| 1 | 15 | 0 | 114 | 108 | 6 | 19 | 0 | 115 | 114 | 1 | 16 | 1 | 168 | 164 | 6 | 9 | 1 | 156 | 159 | 0 | 13 | 2 | 242 | 230 |
| 1 | 17 | 0 | 76 | 71 | 6 | 20 | 0 | 73 | 61 | 1 | 17 | 1 | 120 | 110 | 6 | 10 | 1 | 167 | 175 | 0 | 14 | 2 | 66 | 47 |
| 1 | 19 | 0 | 110 | 100 | 7 | 1 | 0 | 103 | 96 | 1 | 18 | 1 | 114 | 115 | 6 | 11 | 1 | 157 | 162 | 0 | 15 | 2 | 226 | 221 |
| 1 | 23 | 0 | 67 | 57 | 7 | 2 | 0 | 209 | 206 | 1 | 19 | 1 | 65 | 72 | 6 | 12 | 1 | 170 | 167 | 0 | 17 | 2 | 119 | 117 |
| 2 | 0 | 0 | 65 | 64 | 7 | 3 | 0 | 39 | 34 | 1 | 20 | 1 | 72 | 64 | 6 | 13 | 1 | 78 | 78 | 0 | 19 | 2 | 118 | 106 |
| 2 | 1 | 0 | 513 | 601 | 7 | 6 | 0 | 450 | 442 | 1 | 21 | 1 | 83 | 67 | 6 | 15 | 1 | 128 | 124 | 0 | 21 | 2 | 116 | 103 |
| 2 | 2 | 0 | 132 | 134 | 7 | 7 | 0 | 103 | 115 | 1 | 22 | 1 | 86 | 81 | 6 | 16 | 1 | 84 | 84 | 0 | 23 | 2 | 76 | 56 |
| 2 | 3 | 0 | 786 | 922 | 7 | 9 | 0 | 50 | 42 | 2 | 0 | 1 | 836 | 910 | 6 | 17 | 1 | 67 | 64 | 0 | 24 | 2 | 67 | 33 |
| 2 | 4 | 0 | 330 | 329 | 7 | 10 | 0 | 340 | 328 | 2 | 1 | 1 | 793 | 883 | 6 | 20 | 1 | 57 | 46 | 1 | 0 | 2 | 680 | 619 |
| 2 | 5 | 0 | 613 | 670 | 7 | 11 | 0 | 47 | 34 | 2 | 2 | 1 | 467 | 420 | 7 | 0 | 1 | 306 | 306 | 1 | 1 | 2 | 720 | 640 |
| 2 | 6 | 0 | 391 | 403 | 7 | 14 | 0 | 173 | 170 | 2 | 3 | 1 | 611 | 638 | 7 | 1 | 1 | 299 | 301 | 1 | 2 | 2 | 249 | 226 |
| 2 | 7 | 0 | 617 | 666 | 8 | 0 | 0 | 470 | 470 | 2 | 4 | 1 | 617 | 655 | 7 | 2 | 1 | 180 | 185 | 1 | 3 | 2 | 187 | 179 |
| 2 | 8 | 0 | 66 | 64 | 8 | 1 | 0 | 179 | 175 | 2 | 5 | 1 | 463 | 477 | 7 | 3 | 1 | 284 | 289 | 1 | 4 | 2 | 829 | 751 |
| 2 | 9 | 0 | 568 | 611 | 8 | 2 | 0 | 65 | 54 | 2 | 6 | 1 | 127 | 129 | 7 | 4 | 1 | 356 | 399 | 1 | 5 | 2 | 472 | 420 |
| 2 | 10 | 0 | 100 | 108 | 8 | 3 | 0 | 131 | 128 | 2 | 7 | 1 | 251 | 249 | 7 | 5 | 1 | 273 | 270 | 1 | 6 | 2 | 206 | 180 |
| 2 | 11 | 0 | 351 | 367 | 8 | 4 | 0 | 316 | 301 | 2 | 8 | 1 | 442 | 454 | 7 | 6 | 1 | 113 | 107 | 1 | 7 | 2 | 165 | 155 |
| 2 | 12 | 0 | 79 | 79 | 8 | 5 | 0 | 122 | 116 | 2 | 9 | 1 | 303 | 309 | 7 | 7 | 1 | 205 | 205 | 1 | 8 | 2 | 670 | 601 |
| 2 | 13 | 0 | 318 | 331 | 8 | 7 | 0 | 82 | 72 | 2 | 10 | 1 | 71 | 63 | 7 | 8 | 1 | 238 | 243 | 1 | 9 | 2 | 143 | 127 |
| 2 | 14 | 0 | 63 | 65 | 8 | 8 | 0 | 220 | 215 | 2 | 11 | 1 | 186 | 189 | 7 | 9 | 1 | 175 | 171 | 1 | 10 | 2 | 67 | 61 |
| 2 | 15 | 0 | 273 | 280 | 8 | 9 | 0 | 85 | 84 | 2 | 12 | 1 | 266 | 271 | 7 | 10 | 1 | 94 | 93 | 1 | 11 | 2 | 289 | 195 |
| 2 | 17 | 0 | 173 | 172 | 8 | 11 | 0 | 66 | 68 | 2 | 13 | 1 | 231 | 238 | 7 | 11 | 1 | 141 | 144 | 1 | 12 | 2 | 361 | 327 |
| 2 | 18 | 0 | 65 | 61 | 8 | 12 | 0 | 166 | 173 | 2 | 14 | 1 | 170 | 178 | 7 | 12 | 1 | 170 | 163 | 1 | 13 | 2 | 186 | 168 |
| 2 | 19 | 0 | 177 | 173 | 8 | 13 | 0 | 73 | 78 | 2 | 15 | 1 | 181 | 186 | 7 | 13 | 1 | 106 | 102 | 1 | 15 | 2 | 207 | 192 |
| 2 | 21 | 0 | 72 | 62 | 8 | 15 | 0 | 51 | 50 | 2 | 16 | 1 | 131 | 135 | 7 | 14 | 1 | 54 | 59 | 1 | 16 | 2 | 186 | 177 |
| 3 | 1 | 0 | 431 | 460 | 8 | 16 | 0 | 135 | 127 | 2 | 17 | 1 | 124 | 130 | 7 | 15 | 1 | 78 | 82 | 1 | 17 | 2 | 83 | 86 |
| 3 | 2 | 0 | 839 | 995 | 8 | 17 | 0 | 53 | 29 | 2 | 18 | 1 | 58 | 62 | 7 | 16 | 1 | 93 | 89 | 1 | 19 | 2 | 74 | 72 |
| 3 | 3 | 0 | 331 | 353 | 9 | 1 | 0 | 210 | 192 | 2 | 19 | 1 | 78 | 75 | 7 | 17 | 1 | 71 | 70 | 1 | 20 | 2 | 84 | 89 |
| 3 | 4 | 0 | 323 | 342 | 9 | 2 | 0 | 41 | 23 | 2 | 20 | 1 | 65 | 68 | 7 | 19 | 1 | 56 | 40 | 1 | 22 | 2 | 73 | 10 |
| 3 | 5 | 0 | 387 | 402 | 9 | 3 | 0 | 257 | 243 | 2 | 21 | 1 | 77 | 72 | 7 | 20 | 1 | 66 | 39 | 2 | 0 | 2 | 287 | 269 |
| 3 | 6 | 0 | 510 | 563 | 9 | 5 | 0 | 162 | 157 | 3 | 0 | 1 | 510 | 533 | 8 | 0 | 1 | 173 | 176 | 2 | 1 | 2 | 293 | 276 |
| 3 | 7 | 0 | 289 | 298 | 9 | 7 | 0 | 182 | 175 | 3 | 1 | 1 | 663 | 707 | 8 | 1 | 1 | 248 | 244 | 2 | 2 | 2 | 615 | 611 |
| 3 | 8 | 0 | 249 | 246 | 9 | 8 | 0 | 50 | 33 | 3 | 2 | 1 | 581 | 608 | 8 | 2 | 1 | 217 | 210 | 2 | 3 | 2 | 260 | 241 |
| 3 | 9 | 0 | 75 | 78 | 9 | 9 | 0 | 159 | 149 | 3 | 3 | 1 | 430 | 446 | 8 | 3 | 1 | 156 | 152 | 2 | 4 | 2 | 96 | 88 |
| 3 | 10 | 0 | 539 | 578 | 9 | 10 | 0 | 69 | 56 | 3 | 4 | 1 | 347 | 357 | 8 | 4 | 1 | 184 | 183 | 2 | 5 | 2 | 196 | 189 |
| 3 | 11 | 0 | 150 | 152 | 9 | 11 | 0 | 121 | 111 | 3 | 5 | 1 | 426 | 445 | 8 | 5 | 1 | 213 | 209 | 2 | 6 | 2 | 866 | 862 |
| 3 | 12 | 0 | 97 | 102 | 9 | 13 | 0 | 116 | 111 | 3 | 6 | 1 | 260 | 269 | 8 | 6 | 1 | 122 | 116 | 2 | 7 | 2 | 150 | 140 |
| 3 | 13 | 0 | 70 | 74 | 9 | 15 | 0 | 76 | 66 | 3 | 7 | 1 | 251 | 262 | 8 | 7 | 1 | 104 | 97 | 2 | 8 | 2 | 129 | 130 |
| 3 | 14 | 0 | 271 | 282 | 10 | 0 | 0 | 148 | 143 | 3 | 8 | 1 | 466 | 474 | 8 | 8 | 1 | 153 | 140 | 2 | 9 | 2 | 143 | 137 |
| 3 | 15 | 0 | 65 | 58 | 10 | 1 | 0 | 105 | 98 | 3 | 9 | 1 | 258 | 266 | 8 | 9 | 1 | 108 | 110 | 2 | 10 | 2 | 586 | 579 |
| 3 | 17 | 0 | 84 | 86 | 10 | 2 | 0 | 57 | 24 | 3 | 10 | 1 | 222 | 232 | 8 | 10 | 1 | 111 | 100 | 2 | 11 | 2 | 159 | 150 |
| 3 | 18 | 0 | 170 | 170 | 10 | 3 | 0 | 128 | 120 | 3 | 11 | 1 | 189 | 194 | 8 | 11 | 1 | 94 | 80 | 2 | 12 | 2 | 48 | 40 |
| 3 | 19 | 0 | 52 | 59 | 10 | 4 | 0 | 191 | 166 | 3 | 12 | 1 | 200 | 208 | 8 | 12 | 1 | 86 | 84 | 2 | 14 | 2 | 307 | 307 |
| 3 | 22 | 0 | 77 | 75 | 10 | 5 | 0 | 77 | 74 | 3 | 13 | 1 | 211 | 215 | 8 | 14 | 1 | 65 | 66 | 2 | 16 | 2 | 61 | 48 |
| 4 | 0 | 0 | 581 | 651 | 10 | 7 | 0 | 122 | 112 | 3 | 14 | 1 | 177 | 187 | 8 | 15 | 1 | 62 | 63 | 2 | 17 | 2 | 70 | 62 |
| 4 | 1 | 0 | 201 | 211 | 10 | 8 | 0 | 113 | 104 | 3 | 15 | 1 | 144 | 144 | 8 | 16 | 1 | 65 | 60 | 2 | 18 | 2 | 173 | 169 |
| 4 | 2 | 0 | 36 | 26 | 10 | 9 | 0 | 56 | 60 | 3 | 16 | 1 | 57 | 64 | 9 | 0 | 1 | 55 | 25 | 3 | 0 | 2 | 559 | 562 |
| 4 | 3 | 0 | 30 | 23 | 10 | 11 | 0 | 97 | 81 | 3 | 17 | 1 | 102 | 110 | 9 | 1 | 1 | 83 | 82 | 3 | 1 | 2 | 466 | 478 |
| 4 | 4 | 0 | 743 | 821 | 10 | 13 | 0 | 60 | 47 | 3 | 18 | 1 | 78 | 81 | 9 | 2 | 1 | 215 | 203 | 3 | 2 | 2 | 185 | 178 |
| 4 | 5 | 0 | 243 | 254 | 11 | 1 | 0 | 51 | 38 | 3 | 19 | 1 | 97 | 92 | 9 | 3 | 1 | 101 | 93 | 3 | 3 | 2 | 534 | 539 |
| 4 | 6 | 0 | 293 | 293 | 11 | 2 | 0 | 177 | 165 | 3 | 20 | 1 | 74 | 64 | 9 | 5 | 1 | 138 | 141 | 3 | 4 | 2 | 504 | 500 |
| 4 | 7 | 0 | 108 | 114 | 11 | 6 | 0 | 149 | 138 | 3 | 21 | 1 | 51 | 50 | 9 | 6 | 1 | 197 | 187 | 3 | 5 | 2 | 396 | 406 |
| 4 | 8 | 0 | 537 | 559 | 11 | 8 | 0 | 58 | 21 | 4 | 0 | 1 | 218 | 229 | 9 | 7 | 1 | 137 | 121 | 3 | 6 | 2 | 97 | 95 |
| 4 | 9 | 0 | 157 | 160 | 11 | 10 | 0 | 98 | 95 | 4 | 1 | 1 | 250 | 270 | 9 | 9 | 1 | 125 | 114 | 3 | 7 | 2 | 379 | 391 |
| 4 | 10 | 0 | 58 | 62 | 12 | 0 | 0 | 83 | 70 | 4 | 2 | 1 | 554 | 591 | 9 | 10 | 1 | 149 | 138 | 3 | 8 | 2 | 250 | 248 |
| 4 | 11 | 0 | 193 | 200 | 12 | 1 | 0 | 87 | 72 | 4 | 3 | 1 | 248 | 263 | 9 | 11 | 1 | 105 | 93 | 3 | 9 | 2 | 456 | 453 |
| 4 | 12 | 0 | 399 | 403 | 12 | 3 | 0 | 59 | 58 | 4 | 4 | 1 | 282 | 293 | 9 | 14 | 1 | 93 | 85 | 3 | 10 | 2 | 152 | 154 |
| 4 | 13 | 0 | 131 | 130 | 12 | 4 | 0 | 84 | 78 | 4 | 5 | 1 | 478 | 498 | 9 | 15 | 1 | 73 | 52 | 3 | 11 | 2 | 272 | 279 |
| 4 | 14 | 0 | 48 | 49 | 12 | 5 | 0 | 88 | 73 | 4 | 6 | 1 | 524 | 557 | 10 | 0 | 1 | 80 | 77 | 3 | 12 | 2 | 175 | 182 |
| 4 | 15 | 0 | 105 | 106 | 12 | 7 | 0 | 79 | 64 | 4 | 7 | 1 | 301 | 312 | 10 | 1 | 1 | 110 | 114 | 3 | 13 | 2 | 263 | 256 |
| 4 | 16 | 0 | 219 | 222 | 12 | 8 | 0 | 65 | 55 | 4 | 8 | 1 | 121 | 125 | 10 | 2 | 1 | 128 | 116 | 3 | 15 | 2 | 135 | 142 |
| 4 | 17 | 0 | 70 | 62 | 12 | 9 | 0 | 76 | 56 | 4 | 9 | 1 | 343 | 348 | 10 | 3 | 1 | 112 | 107 | 3 | 16 | 2 | 127 | 119 |
| 4 | 20 | 0 | 93 | 98 | 0 | 1 | 1 | 302 | 267 | 4 | 10 | 1 | 232 | 239 | 10 | 4 | 1 | 89 | 80 | 3 | 17 | 2 | 115 | 112 |
| 5 | 1 | 0 | 504 | 542 | 0 | 2 | 1 | 614 | 551 | 4 | 11 | 1 | 169 | 175 | 10 | 5 | 1 | 112 | 100 | 3 | 18 | 2 | 64 | 25 |
| 5 | 2 | 0 | 304 | 308 | 0 | 3 | 1 | 441 | 392 | 4 | 12 | 1 | 46 | 50 | 10 | 6 | 1 | 99 | 91 | 3 | 19 | 2 | 121 | 111 |
| 5 | 3 | 0 | 382 | 398 | 0 | 4 | 1 | 865 | 787 | 4 | 13 | 1 | 144 | 146 | 10 | 7 | 1 | 107 | 100 | 3 | 23 | 2 | 92 | 79 |
| 5 | 4 | 0 | 144 | 156 | 0 | 5 | 1 | 357 | 333 | 4 | 14 | 1 | 239 | 245 | 10 | 8 | 1 | 98 | 83 | 3 | 23 | 2 | 85 | 56 |
| 5 | 5 | 0 | 412 | 423 | 0 | 6 | 1 | 453 | 413 | 4 | 15 | 1 | 116 | 115 | 10 | 9 | 1 | 84 | 78 | 4 | 0 | 2 | 200 | 205 |
| 5 | 6 | 0 | 130 | 129 | 0 | 7 | 1 | 265 | 256 | 4 | 16 | 1 | 42 | 45 | 10 | 10 | 1 | 67 | 59 | 4 | 1 | 2 | 358 | 368 |
| 5 | 7 | 0 | 261 | 267 | 0 | 8 | 1 | 96 | 95 | 4 | 17 | 1 | 75 | 82 | 10 | 11 | 1 | 69 | 69 | 4 | 2 | 2 | 158 | 157 |
| 5 | 8 | 0 | 36 | 21 | 0 | 9 | 1 | 364 | 345 | 4 | 18 | 1 | 136 | 130 | 10 | 12 | 1 | 67 | 44 | 4 | 3 | 2 | 551 | 568 |
| 5 | 9 | 0 | 265 | 266 | 0 | 10 | 1 | 460 | 433 | 4 | 19 | 1 | 48 | 49 | 10 | 13 | 1 | 73 | 58 | 4 | 4 | 2 | 109 | 113 |
| 5 | 10 | 0 | 248 | 253 | 0 | 11 | 1 | 235 | 232 | 5 | 1 | 1 | 336 | 350 | 11 | 0 | 1 | 123 | 113 | 4 | 5 | 2 | 306 | 311 |
| 5 | 11 | 0 | 154 | 157 | 0 | 12 | 1 | 85 | 83 | 5 | 2 | 1 | 454 | 474 | 11 | 1 | 1 | 98 | 100 | 4 | 6 | 2 | 208 | 216 |
| 5 | 12 | 0 | 116 | 116 | 0 | 13 | 1 | 196 | 183 | 5 | 3 | 1 | 298 | 306 | 11 | 3 | 1 | 84 | 78 | 4 | 7 | 2 | 280 | 293 |
| 5 | 13 | 0 | 140 | 137 | 0 | 14 | 1 | 191 | 195 | 5 | 4 | 1 | 41 | 44 | 11 | 4 | 1 | 86 | 85 | 4 | 8 | 2 | 169 | 173 |

*Table 1. Continued*

| h | k | l | Fo | Fc |
|---|---|---|---|---|
| 4 | 9 | 2 | 356 | 356 |
| 4 | 10 | 2 | 242 | 243 |
| 4 | 11 | 2 | 170 | 175 |
| 4 | 12 | 2 | 96 | 106 |
| 4 | 13 | 2 | 251 | 247 |
| 4 | 14 | 2 | 150 | 151 |
| 4 | 15 | 2 | 134 | 133 |
| 4 | 17 | 2 | 84 | 98 |
| 4 | 18 | 2 | 67 | 46 |
| 4 | 19 | 2 | 88 | 69 |
| 5 | 0 | 2 | 715 | 773 |
| 5 | 1 | 2 | 151 | 156 |
| 5 | 2 | 2 | 96 | 92 |
| 5 | 3 | 2 | 178 | 182 |
| 5 | 4 | 2 | 538 | 563 |
| 5 | 5 | 2 | 90 | 98 |
| 5 | 6 | 2 | 87 | 86 |
| 5 | 7 | 2 | 181 | 182 |
| 5 | 8 | 2 | 279 | 287 |
| 5 | 9 | 2 | 158 | 157 |
| 5 | 10 | 2 | 89 | 92 |
| 5 | 11 | 2 | 164 | 165 |
| 5 | 12 | 2 | 179 | 188 |
| 5 | 13 | 2 | 118 | 111 |
| 5 | 15 | 2 | 54 | 39 |
| 5 | 16 | 2 | 156 | 161 |
| 5 | 17 | 2 | 60 | 54 |
| 5 | 20 | 2 | 91 | 87 |
| 6 | 0 | 2 | 76 | 84 |
| 6 | 1 | 2 | 183 | 191 |
| 6 | 2 | 2 | 464 | 490 |
| 6 | 3 | 2 | 89 | 88 |
| 6 | 4 | 2 | 47 | 16 |
| 6 | 5 | 2 | 266 | 274 |
| 6 | 6 | 2 | 405 | 421 |
| 6 | 7 | 2 | 191 | 195 |
| 6 | 8 | 2 | 40 | 39 |
| 6 | 9 | 2 | 190 | 192 |
| 6 | 10 | 2 | 274 | 275 |
| 6 | 11 | 2 | 134 | 138 |
| 6 | 13 | 2 | 77 | 73 |
| 6 | 14 | 2 | 173 | 171 |
| 6 | 18 | 2 | 94 | 97 |
| 7 | 1 | 2 | 225 | 228 |
| 7 | 2 | 2 | 52 | 61 |
| 7 | 3 | 2 | 198 | 202 |
| 7 | 4 | 2 | 53 | 57 |
| 7 | 5 | 2 | 248 | 254 |
| 7 | 6 | 2 | 71 | 69 |
| 7 | 7 | 2 | 222 | 234 |
| 7 | 8 | 2 | 63 | 69 |
| 7 | 9 | 2 | 174 | 180 |
| 7 | 11 | 2 | 172 | 169 |
| 7 | 13 | 2 | 123 | 120 |
| 7 | 15 | 2 | 116 | 119 |
| 7 | 16 | 2 | 61 | 54 |
| 7 | 17 | 2 | 89 | 59 |
| 7 | 18 | 2 | 74 | 24 |
| 8 | 1 | 2 | 192 | 199 |
| 8 | 2 | 2 | 197 | 202 |
| 8 | 3 | 2 | 150 | 155 |
| 8 | 4 | 2 | 76 | 88 |
| 8 | 5 | 2 | 189 | 185 |
| 8 | 6 | 2 | 110 | 115 |
| 8 | 7 | 2 | 156 | 158 |
| 8 | 8 | 2 | 48 | 57 |
| 8 | 9 | 2 | 147 | 142 |
| 8 | 10 | 2 | 100 | 100 |
| 8 | 11 | 2 | 130 | 130 |
| 8 | 13 | 2 | 87 | 79 |
| 8 | 14 | 2 | 83 | 83 |
| 8 | 15 | 2 | 68 | 66 |
| 8 | 18 | 2 | 60 | 45 |
| 9 | 0 | 2 | 287 | 285 |
| 9 | 1 | 2 | 71 | 61 |
| 9 | 4 | 2 | 255 | 253 |
| 9 | 7 | 2 | 50 | 13 |
| 9 | 8 | 2 | 189 | 185 |
| 9 | 9 | 2 | 67 | 54 |
| 9 | 11 | 2 | 58 | 31 |
| 9 | 12 | 2 | 140 | 141 |
| 9 | 16 | 2 | 92 | 82 |
| 10 | 1 | 2 | 124 | 106 |
| 10 | 2 | 2 | 102 | 102 |
| 10 | 3 | 2 | 101 | 98 |
| 10 | 4 | 2 | 59 | 15 |
| 10 | 5 | 2 | 111 | 112 |
| 10 | 6 | 2 | 114 | 109 |
| 10 | 7 | 2 | 68 | 78 |
| 10 | 9 | 2 | 87 | 65 |
| 10 | 10 | 2 | 94 | 85 |
| 10 | 11 | 2 | 66 | 58 |
| 10 | 12 | 2 | 63 | 28 |
| 11 | 1 | 2 | 104 | 104 |
| 11 | 3 | 2 | 144 | 129 |
| 11 | 5 | 2 | 79 | 75 |
| 11 | 7 | 2 | 92 | 86 |
| 11 | 9 | 2 | 78 | 66 |
| 11 | 11 | 2 | 62 | 45 |
| 12 | 2 | 2 | 93 | 76 |
| 12 | 3 | 2 | 66 | 46 |
| 12 | 6 | 2 | 96 | 86 |
| 0 | 1 | 3 | 395 | 366 |
| 0 | 2 | 3 | 320 | 315 |
| 0 | 3 | 3 | 392 | 369 |
| 0 | 4 | 3 | 51 | 47 |
| 0 | 5 | 3 | 274 | 265 |
| 0 | 6 | 3 | 633 | 614 |
| 0 | 7 | 3 | 170 | 161 |
| 0 | 8 | 3 | 41 | 53 |
| 0 | 9 | 3 | 221 | 221 |
| 0 | 10 | 3 | 416 | 416 |
| 0 | 11 | 3 | 222 | 228 |
| 0 | 12 | 3 | 52 | 35 |
| 0 | 13 | 3 | 60 | 53 |
| 0 | 14 | 3 | 255 | 260 |
| 0 | 15 | 3 | 173 | 175 |
| 0 | 17 | 3 | 68 | 63 |
| 0 | 18 | 3 | 95 | 87 |
| 0 | 19 | 3 | 186 | 96 |
| 0 | 22 | 3 | 65 | 47 |
| 1 | 0 | 3 | 656 | 600 |
| 1 | 1 | 3 | 509 | 460 |
| 1 | 2 | 3 | 310 | 292 |
| 1 | 3 | 3 | 318 | 298 |
| 1 | 4 | 3 | 369 | 342 |
| 1 | 5 | 3 | 600 | 563 |
| 1 | 6 | 3 | 245 | 238 |
| 1 | 7 | 3 | 231 | 216 |
| 1 | 8 | 3 | 266 | 252 |
| 1 | 9 | 3 | 286 | 272 |
| 1 | 10 | 3 | 237 | 229 |
| 1 | 11 | 3 | 221 | 212 |
| 1 | 12 | 3 | 157 | 153 |
| 1 | 13 | 3 | 162 | 169 |
| 1 | 14 | 3 | 134 | 130 |
| 1 | 15 | 3 | 137 | 139 |
| 1 | 16 | 3 | 126 | 113 |
| 1 | 17 | 3 | 99 | 93 |
| 1 | 19 | 3 | 71 | 80 |
| 1 | 20 | 3 | 77 | 77 |
| 2 | 0 | 3 | 642 | 602 |
| 2 | 1 | 3 | 423 | 395 |
| 2 | 2 | 3 | 90 | 89 |
| 2 | 3 | 3 | 524 | 493 |
| 2 | 4 | 3 | 517 | 492 |
| 2 | 5 | 3 | 317 | 311 |
| 2 | 7 | 3 | 380 | 374 |
| 2 | 8 | 3 | 414 | 404 |
| 2 | 9 | 3 | 274 | 267 |
| 2 | 10 | 3 | 155 | 149 |
| 2 | 11 | 3 | 178 | 177 |
| 2 | 12 | 3 | 268 | 268 |
| 2 | 13 | 3 | 180 | 189 |
| 2 | 14 | 3 | 65 | 53 |
| 2 | 15 | 3 | 103 | 98 |
| 2 | 16 | 3 | 125 | 132 |
| 2 | 17 | 3 | 69 | 61 |
| 2 | 18 | 3 | 62 | 41 |
| 2 | 19 | 3 | 73 | 69 |
| 2 | 20 | 3 | 67 | 68 |
| 2 | 23 | 3 | 69 | 48 |
| 3 | 0 | 3 | 416 | 411 |
| 3 | 1 | 3 | 460 | 457 |
| 3 | 2 | 3 | 262 | 266 |
| 3 | 3 | 3 | 279 | 277 |
| 3 | 4 | 3 | 489 | 484 |
| 3 | 5 | 3 | 193 | 189 |
| 3 | 6 | 3 | 140 | 141 |
| 3 | 7 | 3 | 196 | 196 |
| 3 | 8 | 3 | 280 | 279 |
| 3 | 9 | 3 | 115 | 110 |
| 3 | 10 | 3 | 56 | 24 |
| 3 | 11 | 3 | 139 | 132 |
| 3 | 12 | 3 | 210 | 217 |
| 3 | 13 | 3 | 139 | 141 |
| 3 | 14 | 3 | 93 | 99 |
| 3 | 15 | 3 | 82 | 75 |
| 3 | 16 | 3 | 141 | 149 |
| 3 | 17 | 3 | 93 | 94 |
| 3 | 20 | 3 | 67 | 56 |
| 4 | 0 | 3 | 72 | 71 |
| 4 | 1 | 3 | 202 | 192 |
| 4 | 2 | 3 | 405 | 398 |
| 4 | 3 | 3 | 245 | 242 |
| 4 | 4 | 3 | 154 | 148 |
| 4 | 5 | 3 | 393 | 391 |
| 4 | 6 | 3 | 353 | 346 |
| 4 | 7 | 3 | 376 | 369 |
| 4 | 8 | 3 | 149 | 144 |
| 4 | 9 | 3 | 293 | 293 |
| 4 | 10 | 3 | 291 | 292 |
| 4 | 11 | 3 | 210 | 211 |
| 4 | 12 | 3 | 77 | 66 |
| 4 | 13 | 3 | 120 | 123 |
| 4 | 14 | 3 | 129 | 130 |
| 4 | 15 | 3 | 132 | 131 |
| 4 | 17 | 3 | 63 | 32 |
| 4 | 18 | 3 | 87 | 84 |
| 5 | 0 | 3 | 114 | 125 |
| 5 | 1 | 3 | 243 | 241 |
| 5 | 2 | 3 | 300 | 294 |
| 5 | 3 | 3 | 248 | 247 |
| 5 | 4 | 3 | 209 | 212 |
| 5 | 5 | 3 | 206 | 211 |
| 5 | 6 | 3 | 236 | 230 |
| 5 | 7 | 3 | 208 | 208 |
| 5 | 8 | 3 | 94 | 81 |
| 5 | 9 | 3 | 194 | 191 |
| 5 | 10 | 3 | 176 | 172 |
| 5 | 11 | 3 | 116 | 114 |
| 5 | 12 | 3 | 79 | 62 |
| 5 | 13 | 3 | 155 | 159 |
| 5 | 14 | 3 | 109 | 118 |
| 5 | 15 | 3 | 68 | 80 |
| 5 | 17 | 3 | 92 | 87 |
| 5 | 18 | 3 | 86 | 81 |
| 6 | 0 | 3 | 273 | 288 |
| 6 | 1 | 3 | 179 | 180 |
| 6 | 2 | 3 | 106 | 108 |
| 6 | 3 | 3 | 154 | 157 |
| 6 | 4 | 3 | 245 | 247 |
| 6 | 5 | 3 | 192 | 188 |
| 6 | 6 | 3 | 139 | 140 |
| 6 | 7 | 3 | 159 | 159 |
| 6 | 8 | 3 | 151 | 148 |
| 6 | 9 | 3 | 187 | 197 |
| 6 | 10 | 3 | 111 | 96 |
| 6 | 11 | 3 | 176 | 172 |
| 6 | 12 | 3 | 124 | 127 |
| 6 | 13 | 3 | 105 | 108 |
| 6 | 14 | 3 | 81 | 84 |
| 6 | 15 | 3 | 93 | 90 |
| 6 | 16 | 3 | 68 | 76 |
| 7 | 0 | 3 | 361 | 375 |
| 7 | 1 | 3 | 147 | 148 |
| 7 | 2 | 3 | 49 | 38 |
| 7 | 3 | 3 | 164 | 168 |
| 7 | 4 | 3 | 246 | 246 |
| 7 | 5 | 3 | 168 | 173 |
| 7 | 6 | 3 | 47 | 41 |
| 7 | 7 | 3 | 106 | 104 |
| 7 | 8 | 3 | 147 | 152 |
| 7 | 9 | 3 | 125 | 118 |
| 7 | 11 | 3 | 81 | 78 |
| 7 | 12 | 3 | 97 | 105 |
| 7 | 13 | 3 | 65 | 62 |
| 7 | 16 | 3 | 90 | 88 |
| 7 | 19 | 3 | 63 | 83 |
| 8 | 0 | 3 | 151 | 153 |
| 8 | 1 | 3 | 139 | 148 |
| 8 | 2 | 3 | 172 | 169 |
| 8 | 3 | 3 | 114 | 119 |
| 8 | 4 | 3 | 148 | 140 |
| 8 | 5 | 3 | 142 | 137 |
| 8 | 6 | 3 | 186 | 183 |
| 8 | 7 | 3 | 86 | 89 |
| 8 | 8 | 3 | 113 | 106 |
| 8 | 9 | 3 | 108 | 92 |
| 8 | 10 | 3 | 109 | 106 |
| 8 | 11 | 3 | 115 | 98 |
| 8 | 14 | 3 | 66 | 66 |
| 8 | 15 | 3 | 63 | 63 |
| 9 | 1 | 3 | 120 | 119 |
| 9 | 2 | 3 | 106 | 103 |
| 9 | 3 | 3 | 127 | 119 |
| 9 | 5 | 3 | 111 | 97 |
| 9 | 6 | 3 | 89 | 89 |
| 9 | 7 | 3 | 83 | 92 |
| 9 | 9 | 3 | 106 | 98 |
| 9 | 10 | 3 | 59 | 58 |
| 9 | 11 | 3 | 57 | 64 |
| 9 | 13 | 3 | 82 | 71 |
| 9 | 14 | 3 | 68 | 38 |
| 10 | 1 | 3 | 71 | 69 |
| 10 | 2 | 3 | 101 | 93 |
| 10 | 3 | 3 | 87 | 82 |
| 10 | 4 | 3 | 74 | 83 |
| 10 | 5 | 3 | 80 | 64 |
| 10 | 6 | 3 | 65 | 69 |
| 10 | 7 | 3 | 105 | 88 |
| 10 | 8 | 3 | 129 | 109 |
| 10 | 10 | 3 | 71 | 60 |
| 10 | 11 | 3 | 65 | 59 |
| 10 | 12 | 3 | 92 | 62 |
| 10 | 13 | 3 | 87 | 44 |
| 11 | 0 | 3 | 109 | 90 |
| 11 | 1 | 3 | 94 | 72 |
| 11 | 4 | 3 | 107 | 90 |
| 11 | 5 | 3 | 76 | 43 |
| 11 | 6 | 3 | 82 | 46 |
| 11 | 8 | 3 | 72 | 64 |
| 12 | 1 | 3 | 68 | 50 |
| 0 | 0 | 4 | 786 | 726 |
| 0 | 1 | 4 | 39 | 26 |
| 0 | 2 | 4 | 133 | 126 |
| 0 | 3 | 4 | 175 | 173 |
| 0 | 4 | 4 | 625 | 597 |
| 0 | 8 | 4 | 294 | 280 |
| 0 | 9 | 4 | 50 | 56 |
| 0 | 10 | 4 | 67 | 49 |
| 0 | 12 | 4 | 229 | 218 |
| 0 | 16 | 4 | 152 | 149 |
| 0 | 20 | 4 | 74 | 79 |
| 1 | 1 | 4 | 384 | 362 |
| 1 | 2 | 4 | 356 | 349 |
| 1 | 3 | 4 | 311 | 293 |
| 1 | 4 | 4 | 56 | 61 |
| 1 | 5 | 4 | 391 | 373 |
| 1 | 6 | 4 | 262 | 299 |
| 1 | 7 | 4 | 248 | 243 |
| 1 | 9 | 4 | 289 | 283 |
| 1 | 10 | 4 | 273 | 280 |
| 1 | 11 | 4 | 173 | 175 |
| 1 | 12 | 4 | 66 | 54 |
| 1 | 13 | 4 | 143 | 155 |
| 1 | 14 | 4 | 154 | 151 |
| 1 | 15 | 4 | 102 | 103 |
| 1 | 17 | 4 | 90 | 97 |
| 1 | 18 | 4 | 93 | 87 |
| 1 | 19 | 4 | 67 | 58 |
| 2 | 0 | 4 | 121 | 122 |
| 2 | 1 | 4 | 224 | 218 |
| 2 | 2 | 4 | 213 | 200 |
| 2 | 3 | 4 | 258 | 246 |
| 2 | 4 | 4 | 85 | 81 |
| 2 | 5 | 4 | 344 | 341 |
| 2 | 6 | 4 | 98 | 80 |
| 2 | 7 | 4 | 318 | 317 |
| 2 | 8 | 4 | 79 | 84 |
| 2 | 9 | 4 | 294 | 291 |
| 2 | 11 | 4 | 208 | 214 |
| 2 | 12 | 4 | 69 | 66 |
| 2 | 13 | 4 | 121 | 131 |
| 2 | 14 | 4 | 70 | 62 |
| 2 | 15 | 4 | 113 | 119 |
| 2 | 16 | 4 | 62 | 46 |
| 2 | 17 | 4 | 61 | 67 |
| 3 | 0 | 4 | 48 | 21 |
| 3 | 1 | 4 | 277 | 277 |
| 3 | 2 | 4 | 501 | 503 |
| 3 | 3 | 4 | 218 | 214 |
| 3 | 4 | 4 | 82 | 80 |
| 3 | 5 | 4 | 271 | 274 |
| 3 | 6 | 4 | 369 | 377 |
| 3 | 7 | 4 | 144 | 143 |
| 3 | 9 | 4 | 80 | 85 |
| 3 | 10 | 4 | 240 | 240 |
| 3 | 11 | 4 | 102 | 97 |
| 3 | 13 | 4 | 61 | 53 |
| 3 | 14 | 4 | 215 | 223 |
| 3 | 15 | 4 | 72 | 67 |
| 3 | 17 | 4 | 78 | 76 |
| 3 | 18 | 4 | 119 | 121 |
| 4 | 0 | 4 | 314 | 317 |
| 4 | 1 | 4 | 192 | 191 |
| 4 | 3 | 4 | 68 | 73 |
| 4 | 4 | 4 | 275 | 273 |
| 4 | 5 | 4 | 161 | 161 |
| 4 | 7 | 4 | 74 | 82 |
| 4 | 8 | 4 | 322 | 328 |
| 4 | 9 | 4 | 90 | 88 |
| 4 | 10 | 4 | 54 | 43 |
| 4 | 11 | 4 | 136 | 133 |
| 4 | 12 | 4 | 185 | 207 |
| 4 | 13 | 4 | 72 | 63 |
| 4 | 15 | 4 | 81 | 86 |
| 4 | 16 | 4 | 79 | 74 |
| 4 | 19 | 4 | 73 | 41 |
| 5 | 1 | 4 | 349 | 347 |
| 5 | 2 | 4 | 217 | 223 |
| 5 | 3 | 4 | 241 | 238 |
| 5 | 4 | 4 | 73 | 73 |
| 5 | 5 | 4 | 296 | 297 |
| 5 | 6 | 4 | 146 | 147 |
| 5 | 7 | 4 | 133 | 140 |
| 5 | 8 | 4 | 89 | 94 |
| 5 | 9 | 4 | 168 | 176 |
| 5 | 10 | 4 | 89 | 86 |
| 5 | 11 | 4 | 101 | 115 |
| 5 | 13 | 4 | 101 | 106 |
| 5 | 14 | 4 | 97 | 99 |
| 5 | 15 | 4 | 103 | 99 |
| 5 | 17 | 4 | 82 | 77 |
| 6 | 0 | 4 | 158 | 158 |
| 6 | 1 | 4 | 194 | 191 |
| 6 | 2 | 4 | 80 | 72 |
| 6 | 3 | 4 | 167 | 166 |
| 6 | 4 | 4 | 189 | 194 |
| 6 | 5 | 4 | 196 | 201 |
| 6 | 7 | 4 | 130 | 131 |
| 6 | 8 | 4 | 118 | 120 |
| 6 | 9 | 4 | 127 | 138 |
| 6 | 11 | 4 | 100 | 96 |
| 6 | 12 | 4 | 69 | 70 |
| 6 | 15 | 4 | 79 | 73 |
| 6 | 17 | 4 | 61 | 48 |
| 7 | 0 | 4 | 48 | 22 |
| 7 | 2 | 4 | 227 | 229 |
| 7 | 4 | 4 | 65 | 53 |
| 7 | 6 | 4 | 240 | 234 |
| 7 | 9 | 4 | 68 | 60 |
| 7 | 10 | 4 | 210 | 209 |
| 7 | 11 | 4 | 56 | 45 |
| 7 | 12 | 4 | 60 | 32 |
| 7 | 13 | 4 | 60 | 41 |
| 7 | 14 | 4 | 117 | 113 |
| 8 | 0 | 4 | 140 | 138 |
| 8 | 1 | 4 | 90 | 97 |
| 8 | 2 | 4 | 58 | 33 |
| 8 | 3 | 4 | 89 | 76 |
| 8 | 4 | 4 | 146 | 145 |
| 8 | 5 | 4 | 86 | 86 |
| 8 | 7 | 4 | 83 | 64 |
| 8 | 8 | 4 | 106 | 107 |
| 8 | 9 | 4 | 63 | 56 |
| 8 | 12 | 4 | 93 | 78 |
| 9 | 0 | 4 | 58 | 7 |
| 9 | 1 | 4 | 155 | 147 |
| 9 | 2 | 4 | 59 | 49 |
| 9 | 3 | 4 | 147 | 134 |
| 9 | 5 | 4 | 117 | 114 |
| 9 | 7 | 4 | 88 | 93 |
| 9 | 8 | 4 | 70 | 18 |
| 9 | 9 | 4 | 93 | 91 |
| 9 | 11 | 4 | 99 | 69 |
| 9 | 13 | 4 | 77 | 70 |
| 10 | 0 | 4 | 109 | 101 |
| 10 | 3 | 4 | 71 | 33 |
| 10 | 4 | 4 | 110 | 102 |
| 10 | 8 | 4 | 96 | 80 |
| 11 | 2 | 4 | 119 | 113 |
| 0 | 1 | 5 | 242 | 238 |
| 0 | 2 | 5 | 150 | 150 |
| 0 | 3 | 5 | 256 | 260 |
| 0 | 4 | 5 | 76 | 79 |
| 0 | 5 | 5 | 241 | 254 |
| 0 | 6 | 5 | 233 | 244 |
| 0 | 7 | 5 | 242 | 262 |
| 0 | 9 | 5 | 178 | 176 |
| 0 | 10 | 5 | 123 | 140 |
| 0 | 11 | 5 | 116 | 115 |
| 0 | 13 | 5 | 163 | 171 |
| 0 | 14 | 5 | 81 | 67 |
| 0 | 15 | 5 | 95 | 107 |
| 0 | 17 | 5 | 94 | 87 |
| 0 | 19 | 5 | 82 | 63 |
| 1 | 0 | 5 | 276 | 273 |
| 1 | 1 | 5 | 162 | 161 |
| 1 | 2 | 5 | 218 | 216 |
| 1 | 3 | 5 | 93 | 96 |
| 1 | 4 | 5 | 200 | 188 |
| 1 | 5 | 5 | 114 | 108 |
| 1 | 6 | 5 | 185 | 191 |
| 1 | 7 | 5 | 159 | 166 |
| 1 | 8 | 5 | 128 | 116 |
| 1 | 9 | 5 | 133 | 138 |
| 1 | 10 | 5 | 142 | 141 |
| 1 | 11 | 5 | 133 | 148 |
| 1 | 13 | 5 | 61 | 82 |
| 1 | 14 | 5 | 77 | 82 |
| 1 | 15 | 5 | 83 | 78 |
| 1 | 16 | 5 | 88 | 68 |
| 2 | 0 | 5 | 437 | 435 |
| 2 | 1 | 5 | 175 | 165 |
| 2 | 2 | 5 | 178 | 170 |
| 2 | 3 | 5 | 187 | 188 |
| 2 | 4 | 5 | 335 | 339 |
| 2 | 5 | 5 | 137 | 130 |
| 2 | 7 | 5 | 103 | 104 |
| 2 | 8 | 5 | 197 | 199 |
| 2 | 9 | 5 | 166 | 162 |
| 2 | 11 | 5 | 80 | 85 |
| 2 | 12 | 5 | 154 | 161 |
| 2 | 13 | 5 | 105 | 110 |
| 2 | 16 | 5 | 88 | 118 |
| 2 | 20 | 5 | 81 | 64 |
| 3 | 0 | 5 | 136 | 137 |
| 3 | 1 | 5 | 161 | 160 |
| 3 | 2 | 5 | 120 | 113 |
| 3 | 3 | 5 | 209 | 221 |
| 3 | 4 | 5 | 156 | 169 |
| 3 | 5 | 5 | 157 | 168 |
| 3 | 6 | 5 | 158 | 179 |
| 3 | 7 | 5 | 149 | 165 |
| 3 | 8 | 5 | 100 | 105 |
| 3 | 9 | 5 | 108 | 117 |
| 3 | 10 | 5 | 111 | 115 |
| 3 | 11 | 5 | 88 | 107 |
| 3 | 13 | 5 | 66 | 75 |
| 3 | 15 | 5 | 67 | 62 |
| 3 | 19 | 5 | 62 | 43 |
| 4 | 0 | 5 | 132 | 130 |
| 4 | 1 | 5 | 202 | 214 |
| 4 | 2 | 5 | 165 | 157 |
| 4 | 3 | 5 | 226 | 228 |
| 4 | 4 | 5 | 122 | 116 |
| 4 | 5 | 5 | 161 | 153 |
| 4 | 6 | 5 | 177 | 179 |
| 4 | 7 | 5 | 112 | 108 |
| 4 | 8 | 5 | 72 | 59 |
| 4 | 9 | 5 | 146 | 151 |
| 4 | 10 | 5 | 155 | 159 |
| 4 | 11 | 5 | 86 | 100 |
| 4 | 12 | 5 | 72 | 66 |
| 4 | 13 | 5 | 90 | 93 |
| 4 | 14 | 5 | 107 | 84 |
| 4 | 17 | 5 | 70 | 65 |
| 4 | 18 | 5 | 69 | 50 |
| 4 | 19 | 5 | 72 | 56 |
| 5 | 0 | 5 | 65 | 62 |
| 5 | 1 | 5 | 134 | 132 |
| 5 | 2 | 5 | 120 | 136 |
| 5 | 3 | 5 | 168 | 163 |
| 5 | 4 | 5 | 60 | 57 |
| 5 | 5 | 5 | 106 | 98 |
| 5 | 6 | 5 | 153 | 165 |
| 5 | 7 | 5 | 101 | 98 |
| 5 | 9 | 5 | 55 | 52 |
| 5 | 10 | 5 | 148 | 145 |
| 5 | 11 | 5 | 71 | 63 |
| 5 | 13 | 5 | 81 | 42 |
| 5 | 14 | 5 | 69 | 80 |
| 6 | 0 | 5 | 201 | 202 |
| 6 | 1 | 5 | 108 | 112 |

*Table 1. Continued.*

| h | k | l | Fo | Fc | h | k | l | Fo | Fc | h | k | l | Fo | Fc | h | k | l | Fo | Fc | h | k | l | Fo | Fc |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 6 | 3 | 5 | 93 | 87 | 10 | 2 | 5 | 69 | 54 | 3 | 4 | 6 | 108 | 116 | 7 | 5 | 6 | 79 | 99 | 3 | 5 | 7 | 66 | 75 |
| 6 | 4 | 5 | 181 | 183 | 10 | 3 | 5 | 83 | 50 | 3 | 5 | 6 | 144 | 154 | 7 | 7 | 6 | 86 | 64 | 3 | 8 | 7 | 75 | 93 |
| 6 | 5 | 5 | 124 | 118 | 10 | 4 | 5 | 63 | 63 | 3 | 6 | 6 | 69 | 54 | 7 | 9 | 6 | 84 | 91 | 3 | 9 | 7 | 72 | 55 |
| 6 | 6 | 5 | 80 | 78 | 10 | 6 | 5 | 65 | 32 | 3 | 7 | 6 | 148 | 162 | 7 | 11 | 6 | 77 | 77 | 3 | 12 | 7 | 79 | 70 |
| 6 | 7 | 5 | 54 | 61 | 0 | 1 | 6 | 200 | 199 | 3 | 8 | 6 | 114 | 118 | 8 | 1 | 6 | 76 | 63 | 4 | 2 | 7 | 118 | 119 |
| 6 | 8 | 5 | 162 | 163 | 0 | 3 | 6 | 177 | 180 | 3 | 9 | 6 | 110 | 114 | 8 | 5 | 6 | 90 | 85 | 4 | 3 | 7 | 70 | 72 |
| 6 | 9 | 5 | 89 | 81 | 0 | 5 | 6 | 222 | 232 | 3 | 11 | 6 | 84 | 105 | 8 | 6 | 6 | 82 | 54 | 4 | 5 | 7 | 58 | 14 |
| 6 | 10 | 5 | 76 | 72 | 0 | 7 | 6 | 80 | 65 | 3 | 12 | 6 | 58 | 63 | 9 | 0 | 6 | 76 | 82 | 4 | 6 | 7 | 77 | 94 |
| 6 | 12 | 5 | 115 | 112 | 0 | 9 | 6 | 116 | 132 | 3 | 15 | 6 | 61 | 66 | 9 | 2 | 6 | 62 | 24 | 4 | 11 | 7 | 70 | 35 |
| 6 | 13 | 5 | 79 | 62 | 0 | 10 | 6 | 63 | 59 | 4 | 0 | 6 | 62 | 57 | 9 | 4 | 6 | 90 | 77 | 5 | 0 | 7 | 69 | 63 |
| 6 | 14 | 5 | 58 | 40 | 0 | 11 | 6 | 85 | 95 | 4 | 1 | 6 | 174 | 199 | 0 | 1 | 7 | 70 | 81 | 5 | 2 | 7 | 70 | 68 |
| 6 | 15 | 5 | 72 | 36 | 0 | 15 | 6 | 85 | 93 | 4 | 2 | 6 | 77 | 67 | 0 | 2 | 7 | 143 | 152 | 5 | 6 | 7 | 98 | 72 |
| 6 | 16 | 5 | 81 | 77 | 1 | 0 | 6 | 187 | 191 | 4 | 3 | 6 | 107 | 125 | 0 | 5 | 7 | 69 | 49 | 5 | 9 | 7 | 75 | 55 |
| 7 | 0 | 5 | 137 | 146 | 1 | 1 | 6 | 145 | 154 | 4 | 4 | 6 | 59 | 36 | 0 | 6 | 7 | 151 | 144 | 6 | 2 | 7 | 60 | 44 |
| 7 | 1 | 5 | 129 | 122 | 1 | 2 | 6 | 100 | 97 | 4 | 5 | 6 | 99 | 103 | 0 | 10 | 7 | 116 | 127 | 6 | 4 | 7 | 61 | 35 |
| 7 | 3 | 5 | 98 | 95 | 1 | 3 | 6 | 94 | 92 | 4 | 7 | 6 | 82 | 87 | 0 | 14 | 7 | 75 | 90 | 6 | 5 | 7 | 60 | 57 |
| 7 | 4 | 5 | 96 | 98 | 1 | 4 | 6 | 199 | 199 | 4 | 9 | 6 | 71 | 79 | 1 | 0 | 7 | 70 | 66 | 6 | 7 | 7 | 65 | 59 |
| 7 | 5 | 5 | 80 | 63 | 1 | 5 | 6 | 133 | 137 | 4 | 10 | 6 | 57 | 58 | 1 | 1 | 7 | 86 | 102 | 7 | 0 | 7 | 61 | 77 |
| 7 | 7 | 5 | 107 | 96 | 1 | 6 | 6 | 82 | 68 | 4 | 11 | 6 | 59 | 63 | 1 | 3 | 7 | 75 | 71 | 7 | 4 | 7 | 77 | 68 |
| 7 | 8 | 5 | 91 | 75 | 1 | 7 | 6 | 64 | 55 | 4 | 13 | 6 | 66 | 69 | 1 | 4 | 7 | 71 | 73 | 0 | 0 | 8 | 100 | 110 |
| 7 | 9 | 5 | 60 | 55 | 1 | 8 | 6 | 175 | 180 | 5 | 0 | 6 | 172 | 178 | 1 | 5 | 7 | 98 | 93 | 0 | 4 | 8 | 79 | 108 |
| 7 | 12 | 5 | 69 | 65 | 1 | 9 | 6 | 107 | 107 | 5 | 1 | 6 | 56 | 43 | 1 | 7 | 7 | 84 | 85 | 0 | 7 | 8 | 65 | 50 |
| 7 | 15 | 5 | 62 | 26 | 1 | 11 | 6 | 101 | 120 | 5 | 3 | 6 | 97 | 84 | 1 | 9 | 7 | 70 | 87 | 0 | 8 | 8 | 77 | 85 |
| 8 | 1 | 5 | 127 | 134 | 1 | 12 | 6 | 103 | 115 | 5 | 4 | 6 | 175 | 193 | 1 | 10 | 7 | 89 | 90 | 0 | 9 | 8 | 63 | 1 |
| 8 | 2 | 5 | 59 | 56 | 1 | 15 | 6 | 72 | 73 | 5 | 7 | 6 | 99 | 94 | 1 | 14 | 7 | 66 | 47 | 1 | 0 | 8 | 72 | 40 |
| 8 | 3 | 5 | 121 | 121 | 1 | 16 | 6 | 61 | 56 | 5 | 8 | 6 | 137 | 143 | 2 | 0 | 7 | 104 | 99 | 1 | 1 | 8 | 79 | 62 |
| 8 | 4 | 5 | 73 | 62 | 2 | 1 | 6 | 96 | 98 | 5 | 11 | 6 | 61 | 42 | 2 | 1 | 7 | 107 | 112 | 1 | 3 | 8 | 71 | 63 |
| 8 | 5 | 5 | 113 | 118 | 2 | 2 | 6 | 233 | 260 | 5 | 12 | 6 | 81 | 90 | 2 | 3 | 7 | 119 | 113 | 2 | 1 | 8 | 62 | 79 |
| 8 | 6 | 5 | 59 | 64 | 2 | 3 | 6 | 55 | 29 | 5 | 13 | 6 | 68 | 41 | 2 | 5 | 7 | 92 | 100 | 2 | 3 | 8 | 75 | 78 |
| 8 | 7 | 5 | 66 | 91 | 2 | 4 | 6 | 62 | 55 | 6 | 1 | 6 | 59 | 45 | 2 | 7 | 7 | 63 | 84 | 2 | 5 | 8 | 70 | 68 |
| 8 | 9 | 5 | 64 | 50 | 2 | 6 | 6 | 198 | 219 | 6 | 2 | 6 | 136 | 140 | 2 | 8 | 7 | 57 | 29 | 2 | 6 | 8 | 62 | 31 |
| 8 | 11 | 5 | 60 | 35 | 2 | 10 | 6 | 120 | 134 | 6 | 5 | 6 | 80 | 86 | 2 | 9 | 7 | 65 | 62 | 2 | 7 | 8 | 65 | 71 |
| 9 | 2 | 5 | 75 | 78 | 2 | 14 | 6 | 82 | 101 | 6 | 6 | 6 | 108 | 103 | 2 | 14 | 7 | 66 | 10 | 3 | 1 | 8 | 77 | 46 |
| 9 | 6 | 5 | 106 | 94 | 2 | 16 | 6 | 60 | 15 | 6 | 10 | 6 | 60 | 74 | 3 | 0 | 7 | 101 | 119 | 3 | 2 | 8 | 60 | 60 |
| 9 | 9 | 5 | 73 | 35 | 3 | 0 | 6 | 103 | 103 | 7 | 1 | 6 | 103 | 103 | 3 | 1 | 7 | 58 | 65 | 4 | 0 | 8 | 82 | 100 |
| 9 | 10 | 5 | 81 | 79 | 3 | 1 | 6 | 109 | 108 | 7 | 2 | 6 | 71 | 51 | 3 | 3 | 7 | 87 | 96 | 4 | 4 | 8 | 84 | 83 |
| 10 | 0 | 5 | 64 | 83 | 3 | 3 | 6 | 107 | 102 | 7 | 3 | 6 | 77 | 81 | 3 | 4 | 7 | 106 | 114 | 4 | 5 | 8 | 71 | 38 |

*Table 2.* Fractional atomic coordinates ($\times 10^5$) and thermal parameters ($\times 10^5$ for $Rb^+$ and $\times 10^4$ for other atoms) with estimated standard deviations. The temperature factor is given by exp $-(B_{11}h^2+B_{22}k^2+B_{33}l^2+B_{12}hk+B_{13}hl+B_{23}kl)$. For numbering of atoms, see Fig. 1.

| Atom | $x$ | $y$ | $z$ | $B_{11}$ | $B_{22}$ | $B_{33}$ | $B_{12}$ | $B_{13}$ | $B_{23}$ |
|---|---|---|---|---|---|---|---|---|---|
| $Rb^+$ | 36183 | 12463 | 88042 | 1037 | 370 | 2514 | 14 | 218 | 284 |
| | 6 | 4 | 12 | 8 | 3 | 22 | 8 | 31 | 17 |
| O11 | 22896 | 43127 | 16168 | 111 | 42 | 265 | 7 | −85 | 59 |
| | 51 | 28 | 92 | 7 | 2 | 21 | 6 | 21 | 12 |
| O12 | 49647 | 47815 | 21442 | 145 | 37 | 251 | −30 | 21 | 30 |
| | 59 | 28 | 93 | 8 | 2 | 19 | 7 | 22 | 11 |
| O21 | 66032 | 41476 | 62849 | 109 | 49 | 370 | −29 | −55 | 36 |
| | 49 | 32 | 117 | 7 | 2 | 21 | 6 | 27 | 14 |
| O22 | 51122 | 31658 | 84999 | 184 | 39 | 182 | 1 | −85 | 81 |
| | 58 | 28 | 86 | 8 | 2 | 17 | 7 | 24 | 12 |
| N1 | 36978 | 43251 | 28394 | 106 | 24 | 186 | 0 | −33 | − 9 |
| | 56 | 29 | 95 | 8 | 2 | 17 | 7 | 22 | 10 |
| N2 | 51972 | 37109 | 66848 | 107 | 29 | 266 | 5 | −30 | 6 |
| | 58 | 31 | 99 | 7 | 2 | 22 | 8 | 21 | 16 |
| N3 | 9371 | 29811 | 64500 | 153 | 44 | 333 | −48 | 1 | − 2 |
| | 63 | 36 | 142 | 9 | 3 | 26 | 8 | 32 | 16 |
| C0 | 37653 | 37965 | 50799 | 86 | 24 | 196 | − 6 | −20 | −23 |
| | 59 | 38 | 112 | 7 | 2 | 18 | 9 | 22 | 14 |
| C3 | 22036 | 33401 | 58256 | 121 | 29 | 208 | − 8 | −46 | − 8 |
| | 74 | 39 | 128 | 9 | 2 | 28 | 8 | 29 | 14 |

*Table 3.* The root mean square amplitudes of vibration $(\overline{u^2})^{\frac{1}{2}}$ (Å) and $B$-values (Å$^2$) along the principal axes given by the components of a unit vector in fractional coordinates ($\times 10^3$).

| Atom | $(\overline{u^2})^{\frac{1}{2}}$ | $B$ | $e_x$ | $e_y$ | $e_z$ |
|---|---|---|---|---|---|
| | .211 | 3.53 | 12 | 60 | 86 |
| $Rb^+$ | .179 | 2.52 | 85 | −26 | 129 |
| | .169 | 2.25 | 100 | 15 | −120 |
| | .234 | 4.31 | − 17 | 56 | 105 |
| O11 | .196 | 3.03 | 102 | 28 | − 92 |
| | .148 | 1.72 | 82 | −24 | 137 |
| | .226 | 4.04 | − 87 | 49 | 32 |
| O12 | .196 | 3.04 | 82 | 28 | 130 |
| | .166 | 2.18 | 56 | 36 | −143 |
| | .252 | 5.00 | − 39 | 55 | 97 |
| O21 | .211 | 3.52 | 2 | 35 | −167 |
| | .168 | 2.24 | 126 | 16 | 33 |
| | .240 | 4.56 | −102 | 32 | 81 |
| O22 | .224 | 3.96 | 81 | 50 | 50 |
| | .119 | 1.13 | 25 | −30 | 171 |
| | .179 | 2.53 | 119 | 9 | − 81 |
| N1 | .167 | 2.20 | 33 | −63 | 47 |
| | .150 | 1.78 | 47 | 21 | 172 |
| | .191 | 2.89 | − 58 | 7 | 175 |
| N2 | .183 | 2.65 | 55 | 61 | 19 |
| | .169 | 2.26 | 106 | −27 | 86 |
| | .247 | 4.81 | 84 | −51 | 4 |
| N3 | .209 | 3.46 | − 4 | 0 | 196 |
| | .182 | 2.61 | 102 | 42 | 4 |
| | .176 | 2.45 | 2 | −51 | 128 |
| C0 | .162 | 2.08 | −114 | 22 | 78 |
| | .142 | 1.60 | 68 | 38 | 127 |
| | .194 | 2.97 | −117 | 22 | 65 |
| C3 | .182 | 2.62 | 25 | 60 | − 78 |
| | .157 | 1.94 | 57 | 19 | 168 |

*Table 4.* Bond distances and angles of the anion. Distances in parentheses are corrected for libration.

| Bond distances (Å) | | Bond angles (°) | |
|---|---|---|---|
| N1 – O11 | 1.234 (1.238) | C0 – N1 – O11 | 116.0 |
| N1 – O12 | 1.229 (1.233) | C0 – N1 – O12 | 121.6 |
| N2 – O21 | 1.264 (1.268) | O11 – N1 – O12 | 122.4 |
| N2 – O22 | 1.235 (1.239) | C0 – N2 – O21 | 121.5 |
| C0 – N1 | 1.390 (1.396) | C0 – N2 – O22 | 118.1 |
| C0 – N2 | 1.363 (1.370) | O21 – N2 – O22 | 120.4 |
| C0 – C3 | 1.416 (1.422) | N1 – C0 – N2 | 125.2 |
| C3 – N3 | 1.143 (1.146) | N1 – C0 – C3 | 117.7 |
| | | N2 – C0 – C3 | 117.1 |
| | | C0 – C3 – N3 | 179.1 |

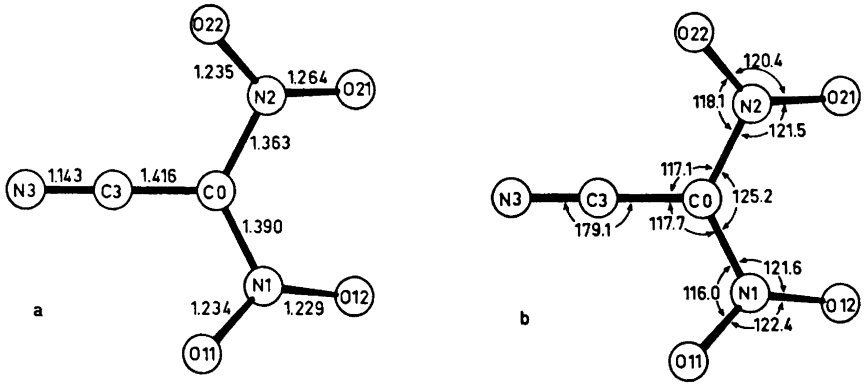


*Fig. 1.* Bond distances (a) and bond angles (b) (uncorrected values) of the anion.

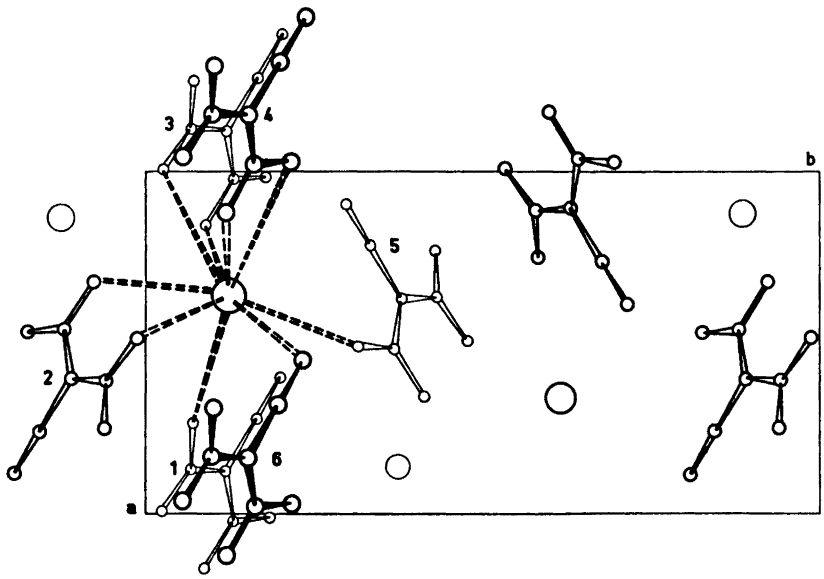


*Fig. 2.* Schematic drawing of the structure viewed along *c*. Equivalent position numbers of Table 5 are indicated.

effects were found to be dependent on the time of registration, and 66 reflections having $F_{obs} > 50$ were excluded from the refinement. The final $R_w$ and $R$ values for 1129 intensities were 4.8 % and 6.3 %. The weight analysis indicates that a higher estimate of the fluctuation in the measurements might be appropriate. No improvement was obtained by omitting more reflections of high intensities or small sin $\theta$ values.

Observed and calculated structure factors are listed in Table 1 and final atomic parameters in Table 2. The eigenvalues of the atomic vibration tensors

are given in Table 3. The r.m.s. discrepancy between "observed" atomic vibration tensor components and those calculated for a rigid-body model is 0.0030 Å$^2$ for the anion. This may indicate a fairly rigid anion although the thermal parameters in particular may be influenced by systematic errors. The corrected as well as the uncorrected bond distances and bond angles are presented in Table 4. The latter values may also be found in Fig. 1. The estimated standard deviations calculated from the correlation matrix of the last least squares refinement cycle are 0.006 Å and 0.007 Å for N–O bonds and other bonds, respectively, and 0.7° or 0.5° or less for angles of 180° and 120°. The coordination of the cation is shown in Fig. 2, and the corresponding distances are given in Table 5.

*Table 5.* Coordination distances of the rubidium ion.

| Atom | Equiv. pos. | No. | (Å) |
|---|---|---|---|
| O11 | $\frac{1}{2}+x$, $\frac{1}{2}-y$,1$-z$ | 1 | 2.91 |
| O12 | 1$-x$,$-\frac{1}{2}+y$,3/2$-z$ | 2 | 3.20 |
| O12 | $-\frac{1}{2}+x$, $\frac{1}{2}-y$, 1$-z$ | 3 | 3.20 |
| O21 | | 2 | 3.15 |
| O21 | | 3 | 3.07 |
| O21 | $-\frac{1}{2}+x$, $\frac{1}{2}-y$, 2$-z$ | 4 | 2.99 |
| O22 | | 4 | 3.11 |
| O22 | $x$, $y$, $z$ | 5 | 3.09 |
| N3 | $\frac{1}{2}+x$, $\frac{1}{2}-y$, 2$-z$ | 6 | 3.20 |

## DISCUSSION

The anion is essentially planar with the nitro groups slightly twisted out of the plane. The twists have opposite directions, however, and the anion is thus not propeller shaped. This is contrary to what is observed for the same anion in the crystal structure of the potassium salt,[5,9] but similar to that observed for the anions of rubidium dinitromethanide[10] and potassium *p*-chlorophenyldinitromethanide.[11]

The central carbon atom (C0) is coplanar with its neighbours, the deviation from the plane through N1, N2, and C3 being 0.019 Å. N3 deviates 0.035 Å in opposite direction to C0 from this plane. O12 and O21 are 0.065 and 0.092 Å above the least squares plane through C0, N1, N2, and C3. O11 and O22 are 0.030 and 0.117 Å below the same plane. The twist angles of O11,N1,O12 and O21,N2,O22 are 2.7 and 5.5°, respectively. The symmetry of the anion is close to $C_s$.

The bond distances and angles of the anion are normal (see Table 6 in Ref. 1) contrary to the unsymmetrical arrangement discussed by Grigor'eva *et al.*[3] Their comments on the N1–C0–N2 angle is also incorrect. The value of this angle (125.2°) is significantly larger than 120° as being predicted by Kemlet *et al.*[12] The C–C bond length (1.416 Å) is not significantly shorter than a normal $sp^2-sp$ C–C single bond (1.43 Å). The O12···O21 contact is 2.62 Å.

The conformation of the anion is determined by crystal forces. The cation is coordinated to eight oxygen atoms and one cyano nitrogen atom of six different anions in an irregular arrangement (Table 5 and Fig. 2). The C3 $-N3\cdots Rb^+$ angle is 120° and the coordination distance of 3.20 Å is quite reasonable.[13] The shortest *intra*-molecular distances are C0···N3 (3.22 Å) and N2···N3 (3.05 Å) between anions related by a screw axis along *a*, C0···O11 (3.04 Å) and N1···O11 (2.90 Å) between anions related by a screw axis along *c*, and N1···O22 (3.00 Å) between translational equivalent anions (along *c*).

Finally, the discrepancy in cell dimensions between the earlier findings[3] and those of the present investigation must be mentioned; the values for the *b* axis being 15.18 and 14.97 Å, respectively. Testing for contamination of potassium in the crystal, if any, by refinement of the occupation factor of rubidium in the least squares refinement must be regarded with caution because of the coupling with the overall scale factor and has therefore not been carried out. A Guinier diagram from another batch of crystals, not analyzed for potassium, gave within limits of errors indentical values to those obtained from diffractometer measurements.

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