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# The Crystal Structure of Tetrapotassium Dihydrogendecaoxodiiodate(VII) Octahydrate

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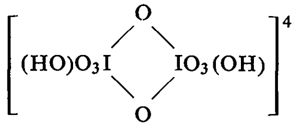
The crystal structure of tetrapotassium dihydrogendecaoxodiiodate (VII) octahydrate, $K_4H_2I_2O_{10}.8H_2O$, has been determined and refined.

The binuclear anion



can be recognized in the structure. It is formed by two octahedral groups with an edge in common, *i.e.* with two oxygen atoms shared between two iodine atoms. Distances within the binuclear anion are $I\text{–}O_{av}{}^* = 1{\cdot}996 \pm 0{\cdot}021$ Å for shared oxygen atoms, including that of the probable hydroxyl group, and $I\text{–}O_{av} = 1{\cdot}807 \pm 0{\cdot}011$ Å for unshared oxygen atoms.

The anions are interposed between $K^+$ cations and water molecules. The water molecules form a shell around the anion to which they are bound by hydrogen bonds. Each water molecule is bound to two different anions, and a three-dimensional network of hydrogen bonds branches all over the crystal.

The potassium ions are surrounded each by eight water molecules or oxygen atoms but their environments are not equivalent.

## Introduction

The molecular and crystal structures of tetraoxoiodates(VII) (*i.e.* metaperiodates) (Hazlewood, 1938; Jaeger & Beintema, 1933) and of some orthoperiodates (Helmholz, 1937; Ferrari, Curti & Cavalca, 1944) are already known. The tetrahedral group $[IO_4]^-$ is present in the former compounds, the octahedral group $[IO_6]^{5-}$ in the latter. A distorted octahedral group has been found in orthoperiodic acid, $(HO)_5IO$ (Feikema, 1961, 1963). The existence of other oxoiodic(VII) acids or oxoiodates(VII) has been demonstrated or postulated by several authors (Rammelsberg, 1868; Blomstrand, 1886; Kimmins, 1889; Rae, 1931; Partington & Bahl, 1934; Pačesová & Hauptmann, 1963). In particular Siebert (1960*a*, *b*), from an examination of the infrared spectra of several oxoiodates(VII) infers the existence of the so-called mesoperiodic anion $[IO_5]^{3-}$, which should be present, for instance, in the salt $K_2HIO_5.4H_2O$. The formula of this salt can also be written $K_4I_2O_9.9H_2O$ (Hill, 1928) or $K_4H_2I_2O_{10}.8H_2O$. The crystal structure of this compound has now been investigated and the last formulation has been proved to be correct.

## Experimental

Crystals of the salt $K_4H_2I_2O_{10}.8H_2O$ can be obtained, either by evaporating or by cooling, from solutions of potassium periodate in concentrated aqueous potassium hydroxide. Its crystallographic properties were studied by Groth (1908), who assigned them to the triclinic system, pinacoidal class.

A crystal elongated parallel to [001], according to the orientation of Groth, has been used to determine the unit-cell constants by the roentgenographic method. The crystal has been rotated around the elongation axis, which is parallel to **c**. The linear constants have been measured on the photographs and the angular constants have been calculated from values given by Groth (1908) after transformation by the matrix 101/010/001. The resulting unit-cell contants are:

$a = 7{\cdot}06 \pm 0{\cdot}01$ Å    $\alpha = 97°\ 47'$
$b = 10{\cdot}57 \pm 0{\cdot}01$    $\beta = 121°\ 30'$
$c = 7{\cdot}56 \pm 0{\cdot}01$    $\gamma = 81°\ 55'$

One stoichiometric unit $K_4H_2I_2O_{10}.8H_2O$ is contained in the unit cell. ($\varrho_{calc} = 2{\cdot}500$ g.cm$^{-3}$, $\varrho_{obs} = 2{\cdot}509$ g.cm$^{-3}$). The cell reported above can be transformed to the conventional cell (*International Tables for X-ray Crystallography*, 1959) by the matrix $101/010/\bar{1}00$.

The space group $P\bar{1}$ was assumed, in accordance with the morphological properties.

For the structural determinations, a crystal rotating around [001] was reduced to a cylindrical shape by fining it by a paper strip wetted by alkali solution. The intensities of the integrated reflexions *hk*0, *hk*1, *hk*2, *hk*3, *hk*4, *hk*5 have been recorded on multiple films in a Weissenberg camera and then measured by a microphotometer. Cu $K\alpha$ radiation was used.

Lorentz and polarization factors were calculated. The transmission factors were obtained by interpolation from data given by Bond (1959) for cylindrical specimens ($\mu R = 1{\cdot}9$).

The atomic form factors were calculated by the Forsyth & Wells (1959) function, using the improved constants by Moore (1963). All the corrections, structure factors, three-dimensional Fourier functions, differential syntheses and anisotropic temperature factor refinement were calculated with an Olivetti Elea 6001/S computer.

## Determination of structure

The first step of the determination of the structure was the interpretation of the Patterson projection $P(uv)$. Vectors I–I, K(1)–I and K(2)–I could be identified and satisfactory agreement between observed and calculated structure factors $F(hk0)$ was obtained. The agreement was improved by calculating a few electron density projections $\varrho_0(xy)$. The $z$ coordinates of I, K(1) and K(2) were found approximately by generalized Patterson functions ${}_{C}P_1(uv)$ and ${}_{S}P_1(uv)$. The remaining atoms were identified in the three-dimensional $\varrho_0(xyz)$ (Fig. 1).

The refinement started at $R = 0{\cdot}164$. The coordinates were refined by the differential synthesis method (Booth, 1946); for the refinement of the anisotropic thermal parameters the method described by Nardelli, Fava & Giraldi (1963) has been followed. The two refinements were applied alternately. The final atomic positions with their e.s.d.'s are reported in Table 1. (Final $R_{hkl} = 0{\cdot}099$, observed reflexions only). The anisotropic thermal parameters are quoted in Table 2. The

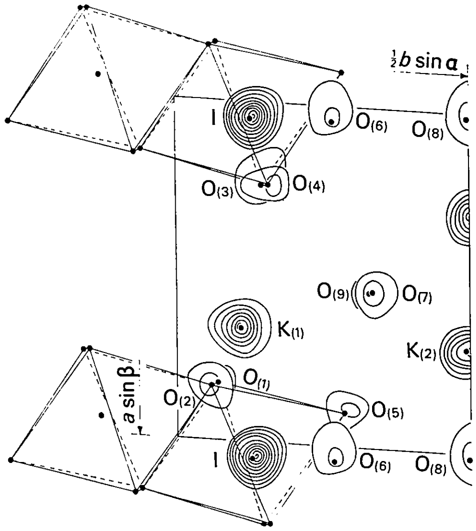


Fig. 1. $\varrho(xyz)$, viewed down [001]. Composite map, arbitrary intervals. Octahedra forming binuclear anions are also drawn.

Table 1. *Final atomic coordinates*

| Atom | $x/a$ | $\sigma(x/a)$ | $y/b$ | $\sigma(y/b)$ | $z/c$ | $\sigma(z/c)$ |
|---|---|---|---|---|---|---|
| I | 0·0503 | 0·0001 | 0·1261 | 0·0001 | 0·4512 | 0·0002 |
| K(1) | 0·6699 | 0·0006 | 0·1117 | 0·0004 | 0·7654 | 0·0007 |
| K(2) | 0·6957 | 0·0006 | 0·4931 | 0·0004 | 0·8454 | 0·0007 |
| O(1) | 0·8300 | 0·0023 | 0·0721 | 0·0014 | 0·1601 | 0·0022 |
| O(2) | 0·8424 | 0·0014 | 0·0611 | 0·0006 | 0·5147 | 0·0018 |
| O(3) | 0·2436 | 0·0022 | 0·1445 | 0·0011 | 0·3722 | 0·0022 |
| O(4) | 0·2373 | 0·0017 | 0·1558 | 0·0010 | 0·7238 | 0·0023 |
| O(5) | 0·8976 | 0·0021 | 0·2833 | 0·0008 | 0·4025 | 0·0025 |
| O(6) | 0·0433 | 0·0023 | 0·2637 | 0·0014 | 0·9733 | 0·0029 |
| O(7) | 0·5404 | 0·0021 | 0·3341 | 0·0011 | 0·9953 | 0·0027 |
| O(8) | 0·0120 | 0·0023 | 0·4967 | 0·0011 | 0·7319 | 0·0028 |
| O(9) | 0·5414 | 0·0022 | 0·3302 | 0·0012 | 0·4844 | 0·0033 |

Table 2. *Anisotropic thermal parameters** (Å$^2$)

| | $B_{11}$ | $B_{22}$ | $B_{33}$ | $B_{12}$ | $B_{13}$ | $B_{23}$ |
|---|---|---|---|---|---|---|
| I | 1·302 | 1·006 | 1·418 | −0·112 | 0·488 | 0·137 |
| K(1) | 1·848 | 2·195 | 1·827 | 0·092 | 0·552 | 0·195 |
| K(2) | 2·457 | 1·940 | 2·714 | −0·138 | 1·024 | 0·195 |
| O(1) | 3·092 | 3·236 | 2·035 | −0·923 | 0·210 | 0·840 |
| O(2) | 1·496 | 1·387 | 2·343 | 0·010 | 0·236 | 0·434 |
| O(3) | 2·191 | 1·400 | 2·480 | −0·759 | 0·703 | −0·429 |
| O(4) | 2·228 | 1·727 | 3·255 | 0·120 | 0·946 | 0·730 |
| O(5) | 3·254 | 1·231 | 3·277 | −0·931 | 1·484 | 0·186 |
| O(6) | 2·355 | 2·505 | 3·469 | 0·146 | 0·867 | 0·148 |
| O(7) | 2·825 | 1·798 | 3·697 | −0·269 | 1·326 | 0·521 |
| O(8) | 4·977 | 2·582 | 4·869 | −0·410 | 2·428 | −0·219 |
| O(9) | 2·093 | 2·718 | 3·442 | 0·587 | 0·543 | 0·898 |

* In the last cycle, for each atomic species I, K and O, the average shift, $|\Delta B_{ij}|$, and the maximum shift, $|\Delta B_{ij}|_{\max}$, were:

for I: $|\Delta B_{ij}| = 0{\cdot}019$ and $|\Delta B_{ij}|_{\max} = 0{\cdot}062$
for K: $|\Delta B_{ij}| = 0{\cdot}020$ and $|\Delta B_{ij}|_{\max} = 0{\cdot}060$
for O: $|\Delta B_{ij}| = 0{\cdot}031$ and $|\Delta B_{ij}|_{\max} = 0{\cdot}179$

The averages have been made over all the parameter shifts of all the atoms of the same species.

Table 3. *Observed and calculated structure factors*

| h | k | l | 10$F_o$ | 10$F_c$ |
|---|---|---|---|---|
| 1 | 0 | 0 | 819 | 962 |
| 2 | 0 | 0 | 471 | 434 |
| 3 | 0 | 0 | 631 | 609 |
| 4 | 0 | 0 | 283 | 274 |
| 5 | 0 | 0 | 194 | 162- |
| 6 | 0 | 0 | — | 5 |
| 7 | 0 | 0 | 181 | 169- |
| 0 | 1 | 0 | 635 | 758 |
| 1 | 1 | 0 | 587 | 711 |
| 1- | 1 | 0 | 684 | 787 |
| 2 | 1 | 0 | 440 | 376- |
| 2- | 1 | 0 | 603 | 649 |
| 3 | 1 | 0 | — | 81- |
| 3- | 1 | 0 | 314 | 305 |
| 4 | 1 | 0 | 465 | 446- |
| 4- | 1 | 0 | 421 | 417 |
| 5 | 1 | 0 | 489 | 405- |
| 5- | 1 | 0 | 605 | 570 |
| 6 | 1 | 0 | 487 | 439- |
| 6- | 1 | 0 | 351 | 337 |
| 7 | 1 | 0 | 332 | 302- |
| 7- | 1 | 0 | 108 | 96- |
| 0 | 2 | 0 | 269 | 196 |
| 1 | 2 | 0 | 224 | 236- |
| 1- | 2 | 0 | 279 | 198 |
| 2 | 2 | 0 | 744 | 874- |
| 2- | 2 | 0 | 193 | 177 |
| 3 | 2 | 0 | 363 | 344- |
| 3- | 2 | 0 | 720 | 720 |
| 4 | 2 | 0 | 436 | 450- |
| 4- | 2 | 0 | 437 | 359 |
| 5 | 2 | 0 | 873 | 831- |
| 5- | 2 | 0 | 731 | 685 |
| 6 | 2 | 0 | 264 | 230- |
| 6- | 2 | 0 | 506 | 474 |
| 7 | 2 | 0 | — | 93- |
| 7- | 2 | 0 | 307 | 230 |
| 0 | 3 | 0 | 921 | 1034- |
| 1 | 3 | 0 | 517 | 528- |
| 1- | 3 | 0 | 783 | 800- |
| 2 | 3 | 0 | 327 | 330- |
| 2- | 3 | 0 | 460 | 435 |
| 3 | 3 | 0 | 1101 | 1110- |
| 3- | 3 | 0 | 423 | 347- |
| 4 | 3 | 0 | 506 | 559- |
| 4- | 3 | 0 | — | 58 |
| 5 | 3 | 0 | 440 | 405- |
| 5- | 3 | 0 | 574 | 527 |
| 6 | 3 | 0 | 237 | 209- |
| 6- | 3 | 0 | 316 | 291 |
| 7 | 3 | 0 | — | 78 |
| 7- | 3 | 0 | 156 | 182 |
| 0 | 4 | 0 | 775 | 839- |
| 1 | 4 | 0 | — | 10- |
| 1- | 4 | 0 | 823 | 813- |
| 2 | 4 | 0 | 298 | 298- |
| 2- | 4 | 0 | 537 | 508- |
| 3 | 4 | 0 | 231 | 184- |
| 3- | 4 | 0 | 362 | 312- |
| 4 | 4 | 0 | 321 | 279- |
| 4- | 4 | 0 | — | 30- |
| 5 | 4 | 0 | — | 42- |
| 5- | 4 | 0 | — | 38- |
| 6 | 4 | 0 | — | 80 |
| 6- | 4 | 0 | 131 | 128 |
| 7 | 4 | 0 | 315 | 299 |
| 7- | 4 | 0 | 173 | 200 |
| 0 | 5 | 0 | 1220 | 1322- |
| 1 | 5 | 0 | — | 36- |
| 1- | 5 | 0 | 903 | 819- |
| 2 | 5 | 0 | 251 | 243 |
| 2- | 5 | 0 | 765 | 759- |
| 3 | 5 | 0 | 310 | 304- |
| 3- | 5 | 0 | 664 | 623- |
| 4 | 5 | 0 | 220 | 226 |
| 4- | 5 | 0 | 399 | 386- |
| 5 | 5 | 0 | 455 | 460 |
| 5- | 5 | 0 | 149 | 127- |
| 6 | 5 | 0 | 232 | 226 |
| 6- | 5 | 0 | 265 | 294- |
| 7 | 5 | 0 | 201 | 214 |
| 0 | 6 | 0 | 271 | 256 |
| 1 | 6 | 0 | — | 34- |
| 1- | 6 | 0 | 337 | 309- |
| 2 | 6 | 0 | 556 | 549 |
| 2- | 6 | 0 | 566 | 548- |
| 3 | 6 | 0 | 489 | 493 |
| 3- | 6 | 0 | 340 | 350- |
| 4 | 6 | 0 | 525 | 527 |
| 4- | 6 | 0 | — | 117- |
| 5 | 6 | 0 | 442 | 463 |
| 5- | 6 | 0 | 520 | 526- |
| 6 | 6 | 0 | 351 | 336 |
| 6- | 6 | 0 | 364 | 402- |
| 7 | 6 | 0 | 217 | 224 |
| 0 | 7 | 0 | 347 | 339 |
| 1 | 7 | 0 | 587 | 565 |
| 1- | 7 | 0 | 412 | 432 |
| 2 | 7 | 0 | 658 | 663 |
| 2- | 7 | 0 | — | 32 |
| 3 | 7 | 0 | 588 | 591 |
| 3- | 7 | 0 | 169 | 155- |
| 4 | 7 | 0 | 318 | 331 |
| 4- | 7 | 0 | — | 76- |
| 5 | 7 | 0 | 372 | 394 |
| 5- | 7 | 0 | 301 | 302- |
| 6 | 7 | 0 | — | 10- |
| 6- | 7 | 0 | 278 | 314- |
| 0 | 8 | 0 | 746 | 775 |
| 1 | 8 | 0 | 327 | 296 |
| 1- | 8 | 0 | 657 | 718 |
| 2 | 8 | 0 | 461 | 446 |
| 2- | 8 | 0 | 232 | 191 |
| 3 | 8 | 0 | 646 | 667 |
| 3- | 8 | 0 | 470 | 510 |
| 4 | 8 | 0 | 165 | 151 |
| 4- | 8 | 0 | 191 | 205 |
| 5 | 8 | 0 | 164 | 160- |
| 5- | 8 | 0 | — | 40- |
| 6 | 8 | 0 | — | 74- |
| 0 | 9 | 0 | 268 | 213 |
| 1 | 9 | 0 | 163 | 75- |
| 1- | 9 | 0 | 279 | 289 |
| 2 | 9 | 0 | — | 40 |
| 2- | 9 | 0 | 543 | 609 |
| 3 | 9 | 0 | 127 | 86- |
| 3- | 9 | 0 | 416 | 425 |
| 4 | 9 | 0 | 253 | 193- |
| 4- | 9 | 0 | 286 | 258 |
| 5 | 9 | 0 | 278 | 300- |
| 5- | 9 | 0 | 199 | 253 |
| 6 | 9 | 0 | 217 | 281- |
| 0 | 10 | 0 | — | 39 |
| 1 | 10 | 0 | 248 | 237- |
| 1- | 10 | 0 | — | 86 |
| 2 | 10 | 0 | 413 | 430- |
| 2- | 10 | 0 | 273 | 269 |
| 3 | 10 | 0 | 129 | 145- |
| 3- | 10 | 0 | 422 | 423 |
| 4 | 10 | 0 | 366 | 406- |
| 4- | 10 | 0 | 236 | 256 |
| 5 | 10 | 0 | 349 | 397- |
| 0 | 11 | 0 | 420 | 429- |
| 1 | 11 | 0 | 245 | 210- |
| 1- | 11 | 0 | 267 | 273- |
| 2 | 11 | 0 | 394 | 410- |
| 2- | 11 | 0 | — | 50 |
| 3 | 11 | 0 | 407 | 460- |
| 3- | 11 | 0 | — | 64 |
| 4 | 11 | 0 | 206 | 218- |
| 4- | 11 | 0 | — | 54- |
| 5 | 11 | 0 | — | 183- |
| 0 | 12 | 0 | 253 | 281- |
| 1 | 12 | 0 | 332 | 339- |
| 1- | 12 | 0 | 364 | 407- |
| 2 | 12 | 0 | 283 | 312- |
| 2- | 12 | 0 | 237 | 239- |
| 3 | 12 | 0 | 188 | 186- |
| 3- | 12 | 0 | — | 173- |
| 4 | 12 | 0 | — | 27 |
| 0 | 13 | 0 | 285 | 323- |
| 1 | 13 | 0 | — | 56- |
| 1- | 13 | 0 | 277 | 267- |
| 2 | 13 | 0 | 137 | 124- |
| 0 | 0 | 1 | 920 | 917- |
| 1 | 0 | 1 | 1336 | 1317- |
| 1- | 0 | 1 | 494 | 566- |
| 2 | 0 | 1 | 271 | 255- |
| 2- | 0 | 1 | 899 | 1074- |
| 3 | 0 | 1 | 274 | 202- |
| 3- | 0 | 1 | 166 | 135- |
| 4 | 0 | 1 | 646 | 534- |
| 4- | 0 | 1 | 356 | 372 |
| 5 | 0 | 1 | 186 | 71- |
| 5- | 0 | 1 | — | 17- |
| 6 | 0 | 1 | 154 | 117 |
| 6- | 0 | 1 | 233 | 217 |
| 7 | 0 | 1 | — | 11 |
| 7- | 0 | 1 | 419 | 406 |
| 8- | 0 | 1 | — | 189 |
| 0 | 1 | 1 | 850 | 993- |
| 0 | 1- | 1 | 941 | 877- |
| 1 | 1 | 1 | 648 | 636- |
| 1- | 1 | 1 | 944 | 1183- |
| 1 | 1- | 1 | 378 | 377- |
| 1- | 1- | 1 | — | 81- |
| 2 | 1 | 1 | 403 | 353- |
| 2- | 1 | 1 | 601 | 694- |
| 2 | 1- | 1 | 258 | 229- |
| 2- | 1- | 1 | 241 | 198 |
| 3 | 1 | 1 | 237 | 210- |
| 3- | 1 | 1 | 197 | 187- |
| 3 | 1- | 1 | 900 | 795- |
| 3- | 1- | 1 | — | 29 |
| 4 | 1 | 1 | 115 | 6- |
| 4- | 1 | 1 | 345 | 376- |
| 4 | 1- | 1 | 671 | 560- |
| 4- | 1- | 1 | 356 | 403 |
| 5 | 1 | 1 | 293 | 261 |
| 5- | 1 | 1 | 241 | 252- |
| 5 | 1- | 1 | 340 | 277- |
| 5- | 1- | 1 | 491 | 525 |
| 6 | 1 | 1 | 320 | 263 |
| 6- | 1 | 1 | — | 33 |
| 6 | 1- | 1 | 461 | 367- |
| 6- | 1- | 1 | 377 | 404 |
| 7 | 1 | 1 | 200 | 193 |
| 7- | 1 | 1 | — | 23 |
| 7 | 1- | 1 | 120 | 135- |
| 7- | 1- | 1 | 344 | 331 |
| 8- | 1 | 1 | — | 77 |
| 8- | 1- | 1 | — | 229 |
| 0 | 2 | 1 | 170 | 106 |
| 0 | 2- | 1 | 213 | 152 |
| 1 | 2 | 1 | 461 | 415- |
| 1- | 2 | 1 | 589 | 660- |
| 1 | 2- | 1 | 464 | 358- |
| 1- | 2- | 1 | 857 | 1005 |
| 2 | 2 | 1 | 110 | 139 |
| 2- | 2 | 1 | 874 | 1146- |
| 2 | 2- | 1 | 189 | 159- |
| 2- | 2- | 1 | 398 | 446 |
| 3 | 2 | 1 | 683 | 651 |
| 3- | 2 | 1 | 401 | 455- |
| 3 | 2- | 1 | 532 | 429- |
| 3- | 2- | 1 | 408 | 432 |
| 4 | 2 | 1 | 373 | 268 |
| 4- | 2 | 1 | 507 | 540- |
| 4 | 2- | 1 | 675 | 546- |
| 4- | 2- | 1 | 787 | 919 |
| 5 | 2 | 1 | 395 | 337 |
| 5- | 2 | 1 | 677 | 742- |
| 5 | 2- | 1 | 582 | 463- |
| 5- | 2- | 1 | 477 | 520 |
| 6 | 2 | 1 | 537 | 503 |
| 6- | 2 | 1 | 361 | 367- |
| 6 | 2- | 1 | 466 | 380- |
| 6- | 2- | 1 | 190 | 204 |
| 7 | 2 | 1 | 218 | 217 |
| 7- | 2 | 1 | 138 | 179- |
| 7 | 2- | 1 | — | 250- |
| 7- | 2- | 1 | 272 | 279 |
| 8- | 2 | 1 | — | 109- |
| 8- | 2- | 1 | — | 71 |
| 0 | 3 | 1 | 403 | 509 |
| 0 | 3- | 1 | 382 | 441 |
| 1 | 3 | 1 | 826 | 919 |
| 1- | 3 | 1 | 457 | 374- |
| 1 | 3- | 1 | 1133 | 944 |
| 1- | 3- | 1 | 707 | 831 |
| 2 | 3 | 1 | 475 | 500 |
| 2- | 3 | 1 | — | 58 |
| 2 | 3- | 1 | 336 | 253 |
| 2- | 3- | 1 | 997 | 1239 |
| 3 | 3 | 1 | 782 | 759 |
| 3- | 3 | 1 | 166 | 136- |
| 3 | 3- | 1 | 301 | 206- |
| 3- | 3- | 1 | 418 | 465 |
| 4 | 3 | 1 | 824 | 766 |
| 4- | 3 | 1 | 639 | 697- |
| 4 | 3- | 1 | 126 | 108 |
| 4- | 3- | 1 | 455 | 492 |
| 5 | 3 | 1 | 392 | 360 |
| 5- | 3 | 1 | 366 | 428- |
| 5 | 3- | 1 | 174 | 155- |
| 5- | 3- | 1 | 533 | 596 |
| 6 | 3 | 1 | 241 | 210 |
| 6- | 3 | 1 | 312 | 307- |
| 6 | 3- | 1 | 597 | 429- |
| 6- | 3- | 1 | — | 53 |
| 7 | 3 | 1 | — | 135 |
| 7- | 3 | 1 | 429 | 464- |
| 7- | 3- | 1 | 149 | 147- |
| 8- | 3- | 1 | — | 76- |
| 0 | 4 | 1 | 675 | 844 |
| 0 | 4- | 1 | 939 | 1080 |
| 1 | 4 | 1 | 455 | 570 |
| 1- | 4 | 1 | 514 | 682 |
| 1 | 4- | 1 | 896 | 998 |
| 1- | 4- | 1 | 544 | 585 |
| 2 | 4 | 1 | 393 | 464 |
| 2- | 4 | 1 | 469 | 509 |
| 2 | 4- | 1 | 742 | 593 |
| 2- | 4- | 1 | 206 | 205 |
| 3 | 4 | 1 | 708 | 729 |
| 3- | 4 | 1 | 372 | 350 |
| 3 | 4- | 1 | 782 | 609 |
| 3- | 4- | 1 | 84 | 68 |
| 4 | 4 | 1 | 371 | 391 |
| 4- | 4 | 1 | — | 35 |
| 4 | 4- | 1 | 549 | 433 |
| 4- | 4- | 1 | 87 | 21 |
| 5 | 4 | 1 | — | 27 |
| 5- | 4 | 1 | 140 | 108- |
| 5 | 4- | 1 | — | 62 |
| 5- | 4- | 1 | 136 | 129- |
| 6 | 4 | 1 | — | 52 |
| 6- | 4 | 1 | 265 | 260- |
| 6 | 4- | 1 | — | 5- |
| 6- | 4- | 1 | 313 | 321- |
| 7 | 4 | 1 | — | 81- |
| 7- | 4 | 1 | 228 | 266- |
| 7- | 4- | 1 | 273 | 273- |
| 8- | 4- | 1 | — | 238- |
| 0 | 5 | 1 | 358 | 461 |
| 0 | 5- | 1 | 400 | 348 |
| 1 | 5 | 1 | 589 | 686 |
| 1- | 5 | 1 | 485 | 615 |
| 1 | 5- | 1 | 893 | 941 |
| 1- | 5- | 1 | 319 | 257- |
| 2 | 5 | 1 | — | 10 |
| 2- | 5 | 1 | 1032 | 1223 |
| 2 | 5- | 1 | 418 | 405 |
| 2- | 5- | 1 | 102 | 116 |
| 3 | 5 | 1 | 184 | 167- |
| 3- | 5 | 1 | 638 | 594 |
| 3 | 5- | 1 | 388 | 353 |
| 3- | 5- | 1 | 373 | 363- |
| 4 | 5 | 1 | — | 87 |
| 4- | 5 | 1 | 209 | 189 |
| 4 | 5- | 1 | 654 | 590 |
| 4- | 5- | 1 | 658 | 740- |
| 5 | 5 | 1 | 271 | 270- |
| 5- | 5 | 1 | 412 | 357 |
| 5 | 5- | 1 | 350 | 319 |
| 5- | 5- | 1 | 366 | 366- |
| 6 | 5 | 1 | 415 | 377- |
| 6- | 5 | 1 | 142 | 100 |
| 6 | 5- | 1 | 204 | 190 |
| 6- | 5- | 1 | 347 | 369- |
| 7- | 5 | 1 | 79 | 110- |
| 7- | 5- | 1 | 406 | 451- |
| 0 | 6 | 1 | — | 34 |
| 0 | 6- | 1 | — | 24- |
| 1 | 6 | 1 | 203 | 146- |
| 1- | 6 | 1 | 447 | 523 |
| 1 | 6- | 1 | 181 | 175- |
| 1- | 6- | 1 | 619 | 593- |
| 2 | 6 | 1 | 158 | 156- |
| 2- | 6 | 1 | 425 | 478 |
| 2 | 6- | 1 | 85 | 52- |
| 2- | 6- | 1 | 642 | 656- |
| 3 | 6 | 1 | 303 | 346- |
| 3- | 6 | 1 | 242 | 266 |
| 3 | 6- | 1 | 417 | 399 |
| 3- | 6- | 1 | 350 | 351- |
| 4 | 6 | 1 | 395 | 405- |
| 4- | 6 | 1 | 381 | 482 |
| 4 | 6- | 1 | 306 | 247 |
| 4- | 6- | 1 | 549 | 565- |
| 5 | 6 | 1 | 382 | 387- |
| 5- | 6 | 1 | 327 | 363 |
| 5 | 6- | 1 | 262 | 218 |
| 5- | 6- | 1 | 539 | 542- |
| 6 | 6 | 1 | 370 | 331- |
| 6- | 6 | 1 | 158 | 168 |
| 6 | 6- | 1 | — | 429 |
| 6- | 6- | 1 | 239 | 222- |
| 7- | 6 | 1 | — | 236 |
| 7- | 6- | 1 | 139 | 148- |
| 0 | 7 | 1 | 460 | 511- |
| 0 | 7- | 1 | 659 | 620- |
| 1 | 7 | 1 | 184 | 254- |
| 1- | 7 | 1 | 151 | 101- |
| 1 | 7- | 1 | 437 | 388- |
| 1- | 7- | 1 | 786 | 730- |
| 2 | 7 | 1 | 364 | 441- |
| 2- | 7 | 1 | 169 | 215 |
| 2 | 7- | 1 | 270 | 234- |
| 2- | 7- | 1 | 489 | 467- |
| 3 | 7 | 1 | 546 | 632- |
| 3- | 7 | 1 | 115 | 101 |
| 3 | 7- | 1 | — | 72- |
| 3- | 7- | 1 | 413 | 383- |
| 4 | 7 | 1 | 288 | 288- |
| 4- | 7 | 1 | 193 | 223 |
| 4 | 7- | 1 | — | 10- |
| 4- | 7- | 1 | 495 | 489- |
| 5 | 7 | 1 | 140 | 192- |
| 5- | 7 | 1 | 328 | 383 |
| 5 | 7- | 1 | 197 | 146 |
| 5- | 7- | 1 | 113 | 106- |
| 6 | 7 | 1 | 294 | 281- |
| 6- | 7 | 1 | 254 | 292 |
| 6- | 7- | 1 | 132 | 128 |
| 7- | 7- | 1 | — | 34 |
| 0 | 8 | 1 | 461 | 528- |
| 0 | 8- | 1 | 352 | 286- |
| 1 | 8 | 1 | 626 | 747- |
| 1- | 8 | 1 | 186 | 157- |
| 1 | 8- | 1 | 782 | 739- |
| 1- | 8- | 1 | 313 | 279- |
| 2 | 8 | 1 | 312 | 365- |
| 2- | 8 | 1 | 315 | 415- |
| 2 | 8- | 1 | 435 | 386- |
| 2- | 8- | 1 | 578 | 536- |
| 3 | 8 | 1 | 298 | 401- |
| 3- | 8 | 1 | 160 | 247- |
| 3 | 8- | 1 | 164 | 132- |
| 3- | 8- | 1 | 140 | 130- |
| 4 | 8 | 1 | 297 | 341- |
| 4- | 8 | 1 | 147 | 162 |
| 4 | 8- | 1 | 396 | 344- |
| 4- | 8- | 1 | — | 37 |
| 5 | 8 | 1 | — | 8 |
| 5- | 8 | 1 | — | 41 |
| 5 | 8- | 1 | 157 | 158- |
| 5- | 8- | 1 | — | 34- |
| 6 | 8 | 1 | — | 87 |
| 6- | 8 | 1 | — | 93 |
| 6- | 8- | 1 | 290 | 268 |
| 7- | 8- | 1 | — | 314 |
| 0 | 9 | 1 | 334 | 348- |
| 0 | 9- | 1 | 325 | 272- |
| 1 | 9 | 1 | 219 | 282- |
| 1- | 9 | 1 | 345 | 432- |
| 1 | 9- | 1 | 487 | 414- |
| 1- | 9- | 1 | 87 | 95 |
| 2 | 9 | 1 | 171 | 196- |
| 2- | 9 | 1 | 391 | 458- |
| 2 | 9- | 1 | 390 | 336- |
| 2- | 9- | 1 | 166 | 146 |
| 3 | 9 | 1 | 121 | 168- |
| 3- | 9 | 1 | 362 | 379- |
| 3 | 9- | 1 | 558 | 470- |
| 3- | 9- | 1 | 155 | 142 |
| 4 | 9 | 1 | — | 32 |
| 4- | 9 | 1 | 254 | 291- |
| 4 | 9- | 1 | 456 | 397- |
| 4- | 9- | 1 | 315 | 311 |
| 5 | 9 | 1 | 192 | 213 |
| 5- | 9 | 1 | 191 | 193- |
| 5- | 9- | 1 | 405 | 350 |
| 6- | 9- | 1 | 329 | 300 |
| 0 | 10 | 1 | — | 9- |
| 0 | 10- | 1 | 213 | 201 |
| 1 | 10 | 1 | 183 | 101- |
| 1- | 10 | 1 | 126 | 155- |
| 1 | 10- | 1 | 192 | 142- |
| 1- | 10- | 1 | 512 | 431 |
| 2 | 10 | 1 | 178 | 212 |
| 2- | 10 | 1 | 497 | 564- |
| 2 | 10- | 1 | 176 | 132- |
| 2- | 10- | 1 | 282 | 219 |
| 3 | 10 | 1 | 337 | 357 |
| 3- | 10 | 1 | 350 | 402- |
| 3 | 10- | 1 | 229 | 182- |
| 3- | 10- | 1 | 486 | 435 |
| 4 | 10 | 1 | 168 | 172 |
| 4- | 10 | 1 | 280 | 295- |
| 4 | 10- | 1 | 293 | 284- |
| 4- | 10- | 1 | 654 | 576 |
| 5 | 10 | 1 | — | 317 |
| 5- | 10 | 1 | — | 356- |
| 5- | 10- | 1 | 314 | 282 |
| 6- | 10- | 1 | — | 182 |
| 0 | 11 | 1 | 260 | 306 |
| 0 | 11- | 1 | 311 | 273 |
| 1 | 11 | 1 | 335 | 365 |
| 1- | 11 | 1 | — | 22- |
| 1 | 11- | 1 | 480 | 420 |
| 1- | 11- | 1 | 524 | 441 |
| 2 | 11 | 1 | 243 | 282 |
| 2- | 11 | 1 | — | 36- |
| 2 | 11- | 1 | 298 | 255 |
| 2- | 11- | 1 | 571 | 482 |
| 3 | 11 | 1 | 365 | 370 |
| 3- | 11 | 1 | — | 40- |
| 3 | 11- | 1 | — | 72- |
| 3- | 11- | 1 | 280 | 233 |
| 4 | 11 | 1 | — | 362 |
| 4- | 11 | 1 | — | 267- |
| 4- | 11- | 1 | 312 | 262 |
| 5- | 11- | 1 | — | 186 |
| 0 | 12 | 1 | 417 | 444 |
| 0 | 12- | 1 | 445 | 391 |
| 1 | 12 | 1 | 309 | 306 |
| 1- | 12 | 1 | 220 | 310 |
| 1 | 12- | 1 | 463 | 412 |
| 1- | 12- | 1 | 344 | 291 |
| 2 | 12 | 1 | 290 | 286 |
| 2- | 12 | 1 | 179 | 186 |
| 2 | 12- | 1 | 344 | 311 |
| 2- | 12- | 1 | 201 | 197 |
| 3 | 12 | 1 | — | 283 |
| 3- | 12 | 1 | — | 146 |
| 3- | 12- | 1 | 119 | 83 |
| 4- | 12- | 1 | — | 24- |
| 0 | 13 | 1 | — | 285 |
| 0 | 13- | 1 | — | 11- |
| 1 | 13 | 1 | — | 338 |
| 1- | 13 | 1 | — | 186 |
| 1 | 13- | 1 | — | 287 |
| 1- | 13- | 1 | — | 26- |
| 2- | 13- | 1 | 104 | 75 |
| 3- | 13- | 1 | — | 172- |
| 0 | 0 | 2 | 463 | 555 |
| 1 | 0 | 2 | 540 | 642 |
| 1- | 0 | 2 | 734 | 720 |
| 2 | 0 | 2 | 1110 | 1214 |
| 2- | 0 | 2 | 598 | 546 |
| 3 | 0 | 2 | 266 | 249 |
| 3- | 0 | 2 | 477 | 455- |
| 4 | 0 | 2 | 198 | 244 |
| 4- | 0 | 2 | — | 51 |
| 5 | 0 | 2 | 276 | 306 |
| 5- | 0 | 2 | 295 | 315- |
| 6 | 0 | 2 | 166 | 100 |
| 6- | 0 | 2 | 537 | 593- |
| 7- | 0 | 2 | 230 | 272- |
| 8- | 0 | 2 | 211 | 291- |
| 0 | 1 | 2 | 777 | 946 |
| 0 | 1- | 2 | 226 | 222- |
| 1 | 1 | 2 | 1060 | 1281 |
| 1- | 1 | 2 | 723 | 863 |
| 1 | 1- | 2 | 789 | 906 |
| 1- | 1- | 2 | 233 | 277- |
| 2 | 1 | 2 | 574 | 572 |
| 2- | 1 | 2 | 673 | 793 |
| 2 | 1- | 2 | 451 | 454 |
| 2- | 1- | 2 | 215 | 244- |
| 3 | 1 | 2 | 142 | 186 |
| 3- | 1 | 2 | 641 | 686 |
| 3 | 1- | 2 | 419 | 369 |
| 3- | 1- | 2 | 482 | 451- |
| 4 | 1 | 2 | — | 27- |
| 4- | 1 | 2 | 509 | 518 |
| 4 | 1- | 2 | 701 | 698 |
| 4- | 1- | 2 | 887 | 942- |
| 5 | 1 | 2 | — | 6- |
| 5- | 1 | 2 | — | 35- |
| 5 | 1- | 2 | 344 | 370 |
| 5- | 1- | 2 | 260 | 305- |
| 6 | 1 | 2 | 301 | 282- |
| 6- | 1 | 2 | — | 43 |
| 6 | 1- | 2 | 228 | 269 |
| 6- | 1- | 2 | 379 | 420- |
| 7- | 1 | 2 | 168 | 94- |
| 7- | 1- | 2 | 303 | 349- |
| 8- | 1 | 2 | 160 | 211- |
| 8- | 1- | 2 | 239 | 239- |
| 0 | 2 | 2 | 130 | 39 |
| 0 | 2- | 2 | 714 | 838- |
| 1 | 2 | 2 | 199 | 120- |
| 1- | 2 | 2 | 694 | 844 |
| 1 | 2- | 2 | 158 | 186- |
| 1- | 2- | 2 | 543 | 671- |
| 2 | 2 | 2 | 237 | 214 |
| 2- | 2 | 2 | 235 | 136- |
| 2 | 2- | 2 | 139 | 147- |
| 2- | 2- | 2 | 829 | 921- |
| 3 | 2 | 2 | 270 | 230- |
| 3- | 2 | 2 | 630 | 613 |
| 3 | 2- | 2 | 228 | 175 |
| 3- | 2- | 2 | 1388 | 1442- |
| 4 | 2 | 2 | 490 | 504- |
| 4- | 2 | 2 | 839 | 840 |
| 4 | 2- | 2 | 179 | 158 |
| 4- | 2- | 2 | 668 | 687- |
| 5 | 2 | 2 | 223 | 260- |
| 5- | 2 | 2 | 444 | 449 |
| 5 | 2- | 2 | 338 | 305 |
| 5- | 2- | 2 | 299 | 307- |
| 6 | 2 | 2 | 283 | 273- |
| 6- | 2 | 2 | — | 96 |
| 6 | 2- | 2 | 280 | 287 |
| 6- | 2- | 2 | 296 | 262- |
| 7- | 2 | 2 | 218 | 255 |
| 7- | 2- | 2 | 122 | 158- |
| 8- | 2 | 2 | — | 1 |
| 8- | 2- | 2 | 92 | 132 |
| 0 | 3 | 2 | 435 | 383 |
| 0 | 3- | 2 | 114 | 9- |
| 1 | 3 | 2 | 529 | 572- |
| 1- | 3 | 2 | 252 | 207- |
| 1 | 3- | 2 | 281 | 374- |
| 1- | 3- | 2 | 935 | 1075- |
| 2 | 3 | 2 | 428 | 462- |
| 2- | 3 | 2 | 194 | 202 |
| 2 | 3- | 2 | 671 | 709- |
| 2- | 3- | 2 | 200 | 161- |
| 3 | 3 | 2 | 398 | 387- |
| 3- | 3 | 2 | 673 | 713 |
| 3 | 3- | 2 | 336 | 370- |
| 3- | 3- | 2 | 367 | 434- |
| 4 | 3 | 2 | 382 | 364- |
| 4- | 3 | 2 | 663 | 652 |
| 4 | 3- | 2 | 219 | 137 |
| 4- | 3- | 2 | 507 | 521- |
| 5 | 3 | 2 | 407 | 423- |
| 5- | 3 | 2 | 369 | 375 |
| 5 | 3- | 2 | — | 80- |
| 5- | 3- | 2 | — | 52 |
| 6 | 3 | 2 | 248 | 257- |
| 6- | 3 | 2 | 474 | 508 |
| 6 | 3- | 2 | 148 | 123 |
| 6- | 3- | 2 | 229 | 247 |
| 7- | 3 | 2 | 239 | 286 |
| 7- | 3- | 2 | — | 68 |
| 8- | 3 | 2 | — | 70 |
| 8- | 3- | 2 | 184 | 220 |
| 0 | 4 | 2 | 478 | 521- |
| 0 | 4- | 2 | 404 | 420- |
| 1 | 4 | 2 | 801 | 883- |
| 1- | 4 | 2 | 180 | 164- |
| 1 | 4- | 2 | 737 | 825- |
| 1- | 4- | 2 | 213 | 173- |
| 2 | 4 | 2 | 571 | 597- |
| 2- | 4 | 2 | 366 | 358- |
| 2 | 4- | 2 | 783 | 826- |
| 2- | 4- | 2 | 571 | 563- |
| 3 | 4 | 2 | 197 | 227- |
| 3- | 4 | 2 | 195 | 112- |
| 3 | 4- | 2 | 452 | 437- |
| 3- | 4- | 2 | 180 | 126 |
| 4 | 4 | 2 | 556 | 573- |
| 4- | 4 | 2 | 318 | 274 |
| 4 | 4- | 2 | 435 | 384- |
| 4- | 4- | 2 | — | 110 |
| 5 | 4 | 2 | 260 | 242- |
| 5- | 4 | 2 | 226 | 206 |
| 5 | 4- | 2 | 270 | 242- |
| 5- | 4- | 2 | 314 | 374 |
| 6 | 4 | 2 | — | 55- |
| 6- | 4 | 2 | 309 | 335 |
| 6 | 4- | 2 | — | 60 |
| 6- | 4- | 2 | 267 | 269 |
| 7- | 4 | 2 | 165 | 212 |
| 7- | 4- | 2 | 303 | 328 |
| 8- | 4- | 2 | 235 | 303 |
| 0 | 5 | 2 | 478 | 543- |
| 0 | 5- | 2 | 138 | 123 |
| 1 | 5 | 2 | 404 | 424- |
| 1- | 5 | 2 | 827 | 826- |
| 1 | 5- | 2 | 185 | 152- |
| 1- | 5- | 2 | 130 | 138- |
| 2 | 5 | 2 | 525 | 536- |
| 2- | 5 | 2 | 611 | 581- |
| 2 | 5- | 2 | 671 | 702- |
| 2- | 5- | 2 | 301 | 248 |
| 3 | 5 | 2 | — | 19 |
| 3- | 5 | 2 | — | 64- |
| 3 | 5- | 2 | 291 | 300- |
| 3- | 5- | 2 | 695 | 699 |
| 4 | 5 | 2 | — | 71 |
| 4- | 5 | 2 | 600 | 522- |
| 4 | 5- | 2 | 406 | 372- |
| 4- | 5- | 2 | 389 | 407 |
| 5 | 5 | 2 | 142 | 120- |
| 5- | 5 | 2 | — | 4- |
| 5 | 5- | 2 | 302 | 279- |
| 5- | 5- | 2 | 348 | 347 |
| 6 | 5 | 2 | 166 | 165 |
| 6- | 5 | 2 | 211 | 229 |
| 6- | 5- | 2 | 625 | 651 |
| 7- | 5 | 2 | 122 | 81 |
| 7- | 5- | 2 | 247 | 269 |
| 8- | 5- | 2 | 150 | 190 |
| 0 | 6 | 2 | 658 | 577- |
| 0 | 6- | 2 | 582 | 561 |
| 1 | 6 | 2 | 371 | 386- |
| 1- | 6 | 2 | 797 | 758- |
| 1 | 6- | 2 | 201 | 257- |
| 1- | 6- | 2 | 625 | 636 |
| 2 | 6 | 2 | — | 96 |
| 2- | 6 | 2 | 431 | 390- |
| 2 | 6- | 2 | 189 | 139 |
| 2- | 6- | 2 | 488 | 472 |
| 3 | 6 | 2 | 188 | 154 |
| 3- | 6 | 2 | 705 | 678- |
| 3 | 6- | 2 | 133 | 134- |
| 3- | 6- | 2 | 508 | 501 |
| 4 | 6 | 2 | 192 | 265 |
| 4- | 6 | 2 | 584 | 545- |
| 4 | 6- | 2 | 451 | 392- |
| 4- | 6- | 2 | 619 | 621 |
| 5 | 6 | 2 | 255 | 260 |
| 5- | 6 | 2 | 265 | 268- |
| 5 | 6- | 2 | 238 | 235- |
| 5- | 6- | 2 | 217 | 151 |
| 6 | 6 | 2 | — | 329 |
| 6- | 6 | 2 | 213 | 240- |
| 6- | 6- | 2 | 170 | 186 |
| 7- | 6 | 2 | 112 | 148- |
| 7- | 6- | 2 | 215 | 231 |
| 8- | 6- | 2 | — | 11- |
| 0 | 7 | 2 | 210 | 121 |
| 0 | 7- | 2 | 539 | 583 |
| 1 | 7 | 2 | 493 | 579 |
| 1- | 7 | 2 | 270 | 256- |
| 1 | 7- | 2 | 488 | 522 |
| 1- | 7- | 2 | 891 | 864 |
| 2 | 7 | 2 | 193 | 199 |
| 2- | 7 | 2 | — | 47 |
| 2 | 7- | 2 | 367 | 385 |
| 2- | 7- | 2 | 762 | 684 |
| 3 | 7 | 2 | 433 | 420 |
| 3- | 7 | 2 | 166 | 176- |
| 3 | 7- | 2 | 166 | 177 |
| 3- | 7- | 2 | 746 | 719 |
| 4 | 7 | 2 | 559 | 521 |
| 4- | 7 | 2 | 543 | 488- |
| 4 | 7- | 2 | 173 | 99 |
| 4- | 7- | 2 | 176 | 134 |
| 5 | 7 | 2 | 280 | 319 |
| 5- | 7 | 2 | 422 | 427- |
| 5 | 7- | 2 | — | 4- |
| 5- | 7- | 2 | — | 10- |
| 6- | 7 | 2 | 262 | 277- |
| 6- | 7- | 2 | — | 50- |
| 7- | 7- | 2 | 137 | 109- |
| 0 | 8 | 2 | 259 | 258 |
| 0 | 8- | 2 | 183 | 172 |
| 1 | 8 | 2 | 630 | 610 |
| 1- | 8 | 2 | 557 | 470 |
| 1 | 8- | 2 | 249 | 257 |
| 1- | 8- | 2 | 639 | 566 |
| 2 | 8 | 2 | 606 | 542 |
| 2- | 8 | 2 | 255 | 218 |

Table 3 (*cont.*)

| h | k | l | $10F_o$ | $10F_c$ |
|---|---|---|---|---|
| 2 | 8- | 2 | 584 | 566 |
| 2- | 8- | 2 | — | 98 |
| 3 | 8 | 2 | 283 | 245 |
| 3- | 8 | 2 | — | 51- |
| 3 | 8- | 2 | 491 | 479 |
| 3- | 8- | 2 | 236 | 176- |
| 4 | 8 | 2 | 331 | 292 |
| 4- | 8 | 2 | — | 88- |
| 4 | 8- | 2 | 234 | 196 |
| 4- | 8- | 2 | — | 24 |
| 5 | 8 | 2 | 187 | 214 |
| 5- | 8 | 2 | — | 95- |
| 5- | 8- | 2 | 388 | 364- |
| 6- | 8 | 2 | 331 | 402- |
| 6- | 8- | 2 | 416 | 403- |
| 7- | 8- | 2 | 272 | 250- |
| 0 | 9 | 2 | 587 | 521 |
| 0 | 9- | 2 | — | 40- |
| 1 | 9 | 2 | 456 | 403 |
| 1- | 9 | 2 | 434 | 375 |
| 1 | 9- | 2 | 273 | 219 |
| 1- | 9- | 2 | 226 | 224- |
| 2 | 9 | 2 | 288 | 244 |
| 2- | 9 | 2 | 524 | 438 |
| 2 | 9- | 2 | 370 | 367 |
| 2- | 9- | 2 | — | 75- |
| 3 | 9 | 2 | — | 70 |
| 3- | 9 | 2 | 358 | 312 |
| 3 | 9- | 2 | 281 | 239 |
| 3- | 9- | 2 | 511 | 465- |
| 4 | 9 | 2 | 199 | 176 |
| 4- | 9 | 2 | 275 | 277 |
| 4 | 9- | 2 | 445 | 458 |
| 4- | 9- | 2 | 515 | 447- |
| 5- | 9 | 2 | — | 78 |
| 5- | 9- | 2 | 401 | 356- |
| 6- | 9- | 2 | 360 | 334- |
| 7- | 9- | 2 | 227 | 254- |
| 0 | 10 | 2 | — | 61 |
| 0 | 10- | 2 | 464 | 421- |
| 1 | 10 | 2 | — | 63- |
| 1- | 10 | 2 | 550 | 488 |
| 1 | 10- | 2 | 172 | 182- |
| 1- | 10- | 2 | 257 | 264- |
| 2 | 10 | 2 | — | 9- |
| 2- | 10 | 2 | 365 | 318 |
| 2 | 10- | 2 | — | 6 |
| 2- | 10- | 2 | 519 | 478- |
| 3 | 10 | 2 | 156 | 175- |
| 3- | 10 | 2 | 217 | 193 |
| 3 | 10- | 2 | — | 56 |
| 3- | 10- | 2 | 561 | 497- |
| 4 | 10 | 2 | 270 | 271- |
| 4- | 10 | 2 | 473 | 461 |
| 4- | 10- | 2 | 381 | 344- |
| 5- | 10 | 2 | 192 | 245 |
| 5- | 10- | 2 | 233 | 223- |
| 6- | 10- | 2 | 346 | 332- |
| 0 | 11 | 2 | — | 3- |
| 0 | 11- | 2 | 507 | 456- |
| 1 | 11 | 2 | 253 | 229- |
| 1- | 11 | 2 | 198 | 152 |
| 1 | 11- | 2 | 192 | 152- |
| 1- | 11- | 2 | 585 | 524- |
| 2 | 11 | 2 | 308 | 278- |
| 2- | 11 | 2 | — | 75 |
| 2 | 11- | 2 | 381 | 346- |
| 2- | 11- | 2 | 158 | 168- |
| 3 | 11 | 2 | 185 | 240- |
| 3- | 11 | 2 | 305 | 291 |
| 3 | 11- | 2 | — | 159- |
| 3- | 11- | 2 | 209 | 161- |
| 4- | 11 | 2 | 194 | 235 |
| 4- | 11- | 2 | 198 | 149- |
| 5- | 11- | 2 | — | 49 |
| 6- | 11- | 2 | — | 93 |
| 0 | 12 | 2 | 292 | 293- |
| 0 | 12- | 2 | 288 | 256- |
| 1 | 12 | 2 | 481 | 440- |
| 1- | 12 | 2 | 142 | 149- |
| 1 | 12- | 2 | 408 | 367- |
| 1- | 12- | 2 | 343 | 331- |
| 2 | 12 | 2 | — | 186- |
| 2- | 12 | 2 | 117 | 124- |
| 2 | 12- | 2 | — | 275- |
| 2- | 12- | 2 | 132 | 137- |
| 3- | 12 | 2 | — | 84- |
| 3- | 12- | 2 | — | 1 |
| 4- | 12- | 2 | 170 | 179 |
| 5- | 12- | 2 | — | 211 |
| 0 | 13- | 2 | — | 2- |
| 1- | 13- | 2 | — | 37- |
| 2- | 13- | 2 | 171 | 161 |
| 3- | 13- | 2 | 315 | 338 |
| 0 | 0 | 3 | 587 | 689- |
| 1 | 0 | 3 | 388 | 399- |
| 1- | 0 | 3 | 125 | 176- |
| 2 | 0 | 3 | 470 | 477- |
| 2- | 0 | 3 | 353 | 398 |
| 3 | 0 | 3 | 456 | 431- |
| 3- | 0 | 3 | — | 56- |
| 4 | 0 | 3 | 251 | 231- |
| 4- | 0 | 3 | 422 | 346 |
| 5 | 0 | 3 | 320 | 285- |
| 5- | 0 | 3 | 811 | 739 |
| 6- | 0 | 3 | 343 | 301 |
| 7- | 0 | 3 | 217 | 217 |
| 8- | 0 | 3 | 431 | 438 |
| 0 | 1 | 3 | 858 | 1004- |
| 0 | 1- | 3 | 389 | 428 |
| 1 | 1 | 3 | 701 | 814- |
| 1- | 1 | 3 | 592 | 714- |
| 1 | 1- | 3 | 81 | 48- |
| 1- | 1- | 3 | 250 | 192- |
| 2 | 1 | 3 | 657 | 658- |
| 2- | 1 | 3 | 283 | 291- |
| 2 | 1- | 3 | 562 | 605- |
| 2- | 1- | 3 | 240 | 339 |
| 3 | 1 | 3 | 254 | 235- |
| 3- | 1 | 3 | 278 | 245- |
| 3 | 1- | 3 | 210 | 202- |
| 3- | 1- | 3 | 854 | 967 |
| 4 | 1 | 3 | — | 0 |
| 4- | 1 | 3 | 124 | 119- |
| 4 | 1- | 3 | 232 | 208- |
| 4- | 1- | 3 | 566 | 545 |
| 5 | 1 | 3 | 138 | 143- |
| 5- | 1 | 3 | 198 | 149 |
| 5 | 1- | 3 | 506 | 504- |
| 5- | 1- | 3 | 479 | 423 |
| 6- | 1 | 3 | 186 | 191 |
| 6- | 1- | 3 | 383 | 358 |
| 7- | 1 | 3 | 253 | 260 |
| 7- | 1- | 3 | 113 | 138 |
| 8- | 1 | 3 | 282 | 273 |
| 8- | 1- | 3 | 138 | 129 |
| 0 | 2 | 3 | 1049 | 1229- |
| 0 | 2- | 3 | 459 | 463 |
| 1 | 2 | 3 | 508 | 527- |
| 1- | 2 | 3 | 325 | 348- |
| 1 | 2- | 3 | 427 | 420 |
| 1- | 2- | 3 | 351 | 355 |
| 2 | 2 | 3 | — | 39 |
| 2- | 2 | 3 | 149 | 113- |
| 2 | 2- | 3 | 125 | 137 |
| 2- | 2- | 3 | 649 | 750 |
| 3 | 2 | 3 | 201 | 178- |
| 3- | 2 | 3 | 803 | 887- |
| 3 | 2- | 3 | 86 | 27- |
| 3- | 2- | 3 | 861 | 1024 |
| 4 | 2 | 3 | 153 | 134 |
| 4- | 2 | 3 | 549 | 520- |
| 4 | 2- | 3 | 166 | 175- |
| 4- | 2- | 3 | 654 | 719 |
| 5 | 2 | 3 | 336 | 323 |
| 5- | 2 | 3 | 245 | 187- |
| 5 | 2- | 3 | 209 | 227- |
| 5- | 2- | 3 | 513 | 469 |
| 6- | 2 | 3 | 354 | 288- |
| 6- | 2- | 3 | 152 | 114 |
| 7- | 2 | 3 | 88 | 13 |
| 7- | 2- | 3 | 132 | 103- |
| 8- | 2 | 3 | 166 | 174 |
| 8- | 2- | 3 | — | 33- |
| 0 | 3 | 3 | 190 | 137- |
| 0 | 3- | 3 | 907 | 970 |
| 1 | 3 | 3 | — | 20 |
| 1- | 3 | 3 | 207 | 204- |
| 1 | 3- | 3 | 657 | 728 |
| 1- | 3- | 3 | 104 | 86 |
| 2 | 3 | 3 | 375 | 342 |
| 2- | 3 | 3 | 447 | 501- |
| 2 | 3- | 3 | 490 | 483 |
| 2- | 3- | 3 | 145 | 157 |
| 3 | 3 | 3 | 380 | 370 |
| 3- | 3 | 3 | 536 | 564- |
| 3 | 3- | 3 | 528 | 531 |
| 3- | 3- | 3 | 743 | 885 |
| 4 | 3 | 3 | 410 | 362 |
| 4- | 3 | 3 | 711 | 632- |
| 4 | 3- | 3 | 138 | 111 |
| 4- | 3- | 3 | 209 | 187 |
| 5 | 3 | 3 | 418 | 400 |
| 5- | 3 | 3 | 849 | 851- |
| 5 | 3- | 3 | — | 48- |
| 5- | 3- | 3 | 345 | 301- |
| 6- | 3 | 3 | 463 | 442- |
| 6- | 3- | 3 | — | 21- |
| 7- | 3 | 3 | 91 | 115- |
| 7- | 3- | 3 | 245 | 236- |
| 8- | 3 | 3 | 161 | 182- |
| 8- | 3- | 3 | 388 | 401- |
| 0 | 4 | 3 | 278 | 277 |
| 0 | 4- | 3 | 374 | 336 |
| 1 | 4 | 3 | 534 | 513 |
| 1- | 4 | 3 | 517 | 504 |
| 1 | 4- | 3 | 549 | 546 |
| 1- | 4- | 3 | 335 | 339 |
| 2 | 4 | 3 | 737 | 727 |
| 2- | 4 | 3 | 134 | 92 |
| 2 | 4- | 3 | 816 | 923 |
| 2- | 4- | 3 | 91 | 87- |
| 3 | 4 | 3 | 372 | 365 |
| 3- | 4 | 3 | 200 | 183- |
| 3 | 4- | 3 | 519 | 507 |
| 3- | 4- | 3 | 270 | 270- |
| 4 | 4 | 3 | 378 | 322 |
| 4- | 4 | 3 | 304 | 215- |
| 4 | 4- | 3 | 176 | 157 |
| 4- | 4- | 3 | 338 | 332- |
| 5 | 4 | 3 | 475 | 462 |
| 5- | 4 | 3 | 464 | 420- |
| 5 | 4- | 3 | 269 | 314 |
| 5- | 4- | 3 | 472 | 467- |
| 6- | 4 | 3 | 420 | 430- |
| 6- | 4- | 3 | 434 | 432- |
| 7- | 4 | 3 | 218 | 233- |
| 7- | 4- | 3 | 372 | 364- |
| 8- | 4 | 3 | — | 231- |
| 8- | 4- | 3 | 350 | 347- |
| 0 | 5 | 3 | 843 | 932 |
| 0 | 5- | 3 | 107 | 65- |
| 1 | 5 | 3 | 316 | 309 |
| 1- | 5 | 3 | 537 | 595 |
| 1 | 5- | 3 | 76 | 44- |
| 1- | 5- | 3 | 321 | 288- |
| 2 | 5 | 3 | 252 | 213 |
| 2- | 5 | 3 | 185 | 164 |
| 2 | 5- | 3 | 313 | 293 |
| 2- | 5- | 3 | 662 | 731- |
| 3 | 5 | 3 | 352 | 316 |
| 3- | 5 | 3 | 540 | 526 |
| 3 | 5- | 3 | 364 | 352 |
| 3- | 5- | 3 | 486 | 564- |
| 4 | 5 | 3 | 170 | 117 |
| 4- | 5 | 3 | 255 | 227 |
| 4 | 5- | 3 | 207 | 226 |
| 4- | 5- | 3 | 514 | 569- |
| 5 | 5 | 3 | — | 51 |
| 5- | 5 | 3 | 305 | 261- |
| 5 | 5- | 3 | 231 | 260 |
| 5- | 5- | 3 | 607 | 651- |
| 6- | 5 | 3 | 88 | 87- |
| 6- | 5- | 3 | 313 | 295- |
| 7- | 5 | 3 | 95 | 99- |
| 7- | 5- | 3 | 237 | 227- |
| 8- | 5- | 3 | 303 | 301- |
| 0 | 6 | 3 | 630 | 604 |
| 0 | 6- | 3 | 714 | 718- |
| 1 | 6 | 3 | 217 | 212 |
| 1- | 6 | 3 | 561 | 651 |
| 1 | 6- | 3 | 429 | 449- |
| 1- | 6- | 3 | 328 | 286- |
| 2 | 6 | 3 | — | 5 |
| 2- | 6 | 3 | 512 | 527 |
| 2 | 6- | 3 | 106 | 33 |
| 2- | 6- | 3 | 643 | 682- |
| 3 | 6 | 3 | 120 | 87- |
| 3- | 6 | 3 | 440 | 427 |
| 3 | 6- | 3 | 95 | 110- |
| 3- | 6- | 3 | 874 | 968- |
| 4 | 6 | 3 | 181 | 183- |
| 4- | 6 | 3 | 330 | 345 |
| 4 | 6- | 3 | — | 10- |
| 4- | 6- | 3 | 221 | 207- |
| 5 | 6 | 3 | — | 148- |
| 5- | 6 | 3 | 261 | 274 |
| 5 | 6- | 3 | — | 245 |
| 5- | 6- | 3 | — | 1- |
| 6- | 6 | 3 | 153 | 171 |
| 6- | 6- | 3 | 179 | 142- |
| 7- | 6 | 3 | — | 33- |
| 7- | 6- | 3 | — | 41 |
| 8- | 6- | 3 | 102 | 113 |
| 0 | 7 | 3 | 410 | 351 |
| 0 | 7- | 3 | 423 | 374- |
| 1 | 7 | 3 | 150 | 147- |
| 1- | 7 | 3 | 188 | 159 |
| 1 | 7- | 3 | 367 | 373- |
| 1- | 7- | 3 | 506 | 445- |
| 2 | 7 | 3 | 416 | 413- |
| 2- | 7 | 3 | 148 | 115 |
| 2 | 7- | 3 | 439 | 454- |
| 2- | 7- | 3 | 550 | 557- |
| 3 | 7 | 3 | 127 | 126- |
| 3- | 7 | 3 | 468 | 424 |
| 3 | 7- | 3 | 308 | 332- |
| 3- | 7- | 3 | 391 | 411- |
| 4 | 7 | 3 | 327 | 283- |
| 4- | 7 | 3 | 279 | 272 |
| 4 | 7- | 3 | 72 | 78- |
| 4- | 7- | 3 | — | 42- |
| 5- | 7 | 3 | 316 | 299 |
| 5- | 7- | 3 | 177 | 163 |
| 6- | 7 | 3 | 370 | 392 |
| 6- | 7- | 3 | 230 | 208 |
| 7- | 7 | 3 | — | 183 |
| 7- | 7- | 3 | 288 | 288 |
| 0 | 8 | 3 | 335 | 331- |
| 0 | 8- | 3 | 310 | 283- |
| 1 | 8 | 3 | 297 | 277- |
| 1- | 8 | 3 | 204 | 185- |
| 1 | 8- | 3 | 214 | 210- |
| 1- | 8- | 3 | — | 19 |
| 2 | 8 | 3 | 414 | 387- |
| 2- | 8 | 3 | 196 | 147 |
| 2 | 8- | 3 | 358 | 362- |
| 2- | 8- | 3 | — | 89 |
| 3 | 8 | 3 | 389 | 384- |
| 3- | 8 | 3 | 81 | 35 |
| 3 | 8- | 3 | 470 | 523- |
| 3- | 8- | 3 | — | 91- |
| 4 | 8 | 3 | 255 | 263- |
| 4- | 8 | 3 | 99 | 89 |
| 4 | 8- | 3 | 219 | 258- |
| 4- | 8- | 3 | 320 | 322 |
| 5- | 8 | 3 | 430 | 450 |
| 5- | 8- | 3 | 464 | 476 |
| 6- | 8 | 3 | 328 | 350 |
| 6- | 8- | 3 | 221 | 212 |
| 7- | 8- | 3 | 321 | 292 |
| 0 | 9 | 3 | 379 | 383- |
| 0 | 9- | 3 | 170 | 172 |
| 1 | 9 | 3 | 351 | 353- |
| 1- | 9 | 3 | 464 | 483- |
| 1 | 9- | 3 | 92 | 78 |
| 1- | 9- | 3 | — | 26 |
| 2 | 9 | 3 | 438 | 403- |
| 2- | 9 | 3 | 313 | 273- |
| 2 | 9- | 3 | 393 | 374- |
| 2- | 9- | 3 | 405 | 400 |
| 3 | 9 | 3 | 242 | 232- |
| 3- | 9 | 3 | 184 | 180- |
| 3 | 9- | 3 | 293 | 308- |
| 3- | 9- | 3 | 522 | 529 |
| 4- | 9 | 3 | 114 | 126- |
| 4- | 9- | 3 | 317 | 335 |
| 5- | 9 | 3 | — | 37 |
| 5- | 9- | 3 | 363 | 353 |
| 6- | 9- | 3 | 364 | 345 |
| 7- | 9- | 3 | 203 | 203 |
| 0 | 10 | 3 | 433 | 433- |
| 0 | 10- | 3 | 295 | 292 |
| 1 | 10 | 3 | — | 7 |
| 1- | 10 | 3 | 403 | 383- |
| 1 | 10- | 3 | 277 | 280 |
| 1- | 10- | 3 | 431 | 393 |
| 2 | 10 | 3 | — | 19 |
| 2- | 10 | 3 | 223 | 262- |
| 2 | 10- | 3 | 86 | 109 |
| 2- | 10- | 3 | 570 | 537 |
| 3 | 10 | 3 | 99 | 105- |
| 3- | 10 | 3 | 470 | 456- |
| 3 | 10- | 3 | — | 27- |
| 3- | 10- | 3 | 386 | 391 |
| 4- | 10 | 3 | 322 | 331- |
| 4- | 10- | 3 | 253 | 229 |
| 5- | 10 | 3 | — | 87- |
| 5- | 10- | 3 | 277 | 268 |
| 6- | 10- | 3 | 139 | 121 |
| 0 | 11 | 3 | — | 20- |
| 0 | 11- | 3 | 478 | 503 |
| 1 | 11 | 3 | 199 | 136 |
| 1- | 11 | 3 | 164 | 175- |
| 1 | 11- | 3 | 357 | 381 |
| 1- | 11- | 3 | 315 | 274 |
| 2 | 11 | 3 | 174 | 220 |
| 2- | 11 | 3 | 327 | 318- |
| 2 | 11- | 3 | 169 | 208 |
| 2- | 11- | 3 | 369 | 330 |
| 3- | 11 | 3 | 296 | 291- |
| 3- | 11- | 3 | 297 | 280 |
| 4- | 11 | 3 | — | 219- |
| 4- | 11- | 3 | 191 | 154- |
| 5- | 11- | 3 | 215 | 196- |
| 6- | 11- | 3 | — | 26- |
| 0 | 12 | 3 | 106 | 141 |
| 0 | 12- | 3 | 180 | 186 |
| 1- | 12 | 3 | — | 159 |
| 1 | 12- | 3 | 107 | 124 |
| 1- | 12- | 3 | 265 | 214 |
| 2- | 12 | 3 | — | 59 |
| 2- | 12- | 3 | 87 | 32 |
| 3- | 12- | 3 | 177 | 151- |
| 4- | 12- | 3 | 260 | 244- |
| 5- | 12- | 3 | 195 | 287- |
| 1- | 13- | 3 | 133 | 169- |
| 2- | 13- | 3 | 238 | 308- |
| 3- | 13- | 3 | 176 | 221- |
| 0 | 0 | 4 | 417 | 415 |
| 1 | 0 | 4 | 412 | 411 |
| 1- | 0 | 4 | 233 | 228- |
| 2 | 0 | 4 | 363 | 421 |
| 2- | 0 | 4 | 189 | 184 |
| 3 | 0 | 4 | 421 | 392 |
| 3- | 0 | 4 | 240 | 210- |
| 4 | 0 | 4 | 450 | 436 |
| 4- | 0 | 4 | 684 | 660- |
| 5 | 0 | 4 | 210 | 269 |
| 5- | 0 | 4 | 503 | 423- |
| 6- | 0 | 4 | 342 | 320- |
| 7- | 0 | 4 | 491 | 475- |
| 8- | 0 | 4 | 288 | 289- |
| 9- | 0 | 4 | — | 81- |
| 0 | 1 | 4 | 689 | 805 |
| 0 | 1- | 4 | 209 | 195- |
| 1 | 1 | 4 | 570 | 581 |
| 1- | 1 | 4 | 445 | 451 |
| 1 | 1- | 4 | 307 | 320- |
| 1- | 1- | 4 | 399 | 476- |
| 2 | 1 | 4 | 275 | 255 |
| 2- | 1 | 4 | 286 | 320 |
| 2 | 1- | 4 | 117 | 25 |
| 2- | 1- | 4 | — | 1204- |
| 3 | 1 | 4 | 472 | 478 |
| 3- | 1 | 4 | 191 | 204 |
| 3 | 1- | 4 | 407 | 403 |
| 3- | 1- | 4 | 396 | 450- |
| 4 | 1 | 4 | 198 | 187 |
| 4- | 1 | 4 | — | 66- |
| 4 | 1- | 4 | 176 | 170 |
| 4- | 1- | 4 | 759 | 747- |
| 5 | 1 | 4 | — | 32 |
| 5- | 1 | 4 | 432 | 336- |
| 5 | 1- | 4 | 171 | 206 |
| 5- | 1- | 4 | 753 | 693- |
| 6- | 1 | 4 | 279 | 219- |
| 6- | 1- | 4 | 320 | 283- |
| 7- | 1 | 4 | 413 | 366- |
| 7- | 1- | 4 | — | 59- |
| 8- | 1 | 4 | 267 | 272- |
| 8- | 1- | 4 | 162 | 135- |
| 9- | 1- | 4 | — | 45 |
| 0 | 2 | 4 | 431 | 434 |
| 0 | 2- | 4 | 554 | 636- |
| 1 | 2 | 4 | 737 | 782 |
| 1- | 2 | 4 | 507 | 535 |
| 1 | 2- | 4 | 461 | 445- |
| 1- | 2- | 4 | 533 | 526- |
| 2 | 2 | 4 | 146 | 166 |
| 2- | 2 | 4 | 770 | 786 |
| 2 | 2- | 4 | 247 | 234- |
| 2- | 2- | 4 | 382 | 522- |
| 3 | 2 | 4 | — | 15- |
| 3- | 2 | 4 | 411 | 485 |
| 3 | 2- | 4 | 144 | 103- |
| 3- | 2- | 4 | 473 | 566- |
| 4 | 2 | 4 | 138 | 110 |
| 4- | 2 | 4 | — | 12 |
| 4 | 2- | 4 | 143 | 115- |
| 4- | 2- | 4 | 397 | 375- |
| 5 | 2 | 4 | — | 135- |
| 5- | 2 | 4 | 514 | 468 |
| 5 | 2- | 4 | — | 164 |
| 5- | 2- | 4 | 329 | 273- |
| 6- | 2 | 4 | 141 | 53 |
| 6- | 2- | 4 | 181 | 144 |
| 7- | 2 | 4 | 194 | 181- |
| 7- | 2- | 4 | — | 95 |
| 8- | 2 | 4 | 79 | 83- |
| 8- | 2- | 4 | 215 | 212 |
| 0 | 3 | 4 | 221 | 236 |
| 0 | 3- | 4 | 349 | 355- |
| 1 | 3 | 4 | 122 | 48- |
| 1- | 3 | 4 | 316 | 270 |
| 1 | 3- | 4 | 906 | 924- |
| 1- | 3- | 4 | 267 | 260- |
| 2 | 3 | 4 | — | 90- |
| 2- | 3 | 4 | 436 | 446 |
| 2 | 3- | 4 | 390 | 371- |
| 2- | 3- | 4 | 355 | 417- |
| 3 | 3 | 4 | 251 | 247- |
| 3- | 3 | 4 | 284 | 317 |
| 3 | 3- | 4 | 344 | 343- |
| 3- | 3- | 4 | 99 | 77- |
| 4 | 3 | 4 | 193 | 184- |
| 4- | 3 | 4 | 731 | 607 |
| 4 | 3- | 4 | 315 | 326- |
| 4- | 3- | 4 | 408 | 392 |
| 5- | 3 | 4 | 517 | 477 |
| 5- | 3- | 4 | — | 29 |
| 6- | 3 | 4 | 210 | 197 |
| 6- | 3- | 4 | 263 | 246 |
| 7- | 3 | 4 | 223 | 214 |
| 7- | 3- | 4 | 529 | 492 |
| 8- | 3 | 4 | 55 | 56 |
| 8- | 3- | 4 | 227 | 216 |
| 0 | 4 | 4 | 356 | 341- |
| 0 | 4- | 4 | 468 | 459- |
| 1 | 4 | 4 | 100 | 76- |
| 1- | 4 | 4 | 75 | 67 |
| 1 | 4- | 4 | 209 | 209- |
| 1- | 4- | 4 | 306 | 245 |
| 2 | 4 | 4 | 365 | 338- |
| 2- | 4 | 4 | 471 | 446 |
| 2 | 4- | 4 | 230 | 233- |
| 2- | 4- | 4 | 281 | 236 |
| 3 | 4 | 4 | 476 | 502- |
| 3- | 4 | 4 | 170 | 162 |
| 3 | 4- | 4 | 410 | 480- |
| 3- | 4- | 4 | 307 | 316 |
| 4 | 4 | 4 | 292 | 280- |
| 4- | 4 | 4 | 450 | 462 |
| 4 | 4- | 4 | 257 | 273- |
| 4- | 4- | 4 | 450 | 482 |
| 5- | 4 | 4 | 568 | 544 |
| 5- | 4- | 4 | 615 | 685 |
| 6- | 4 | 4 | 365 | 370 |
| 6- | 4- | 4 | 339 | 329 |
| 7- | 4 | 4 | 218 | 225 |
| 7- | 4- | 4 | 400 | 371 |
| 8- | 4 | 4 | 200 | 262 |
| 8- | 4- | 4 | 283 | 274 |
| 0 | 5 | 4 | 490 | 522- |
| 0 | 5- | 4 | 129 | 180 |
| 1 | 5 | 4 | 523 | 562- |
| 1- | 5 | 4 | 170 | 138- |
| 1 | 5- | 4 | — | 75 |
| 1- | 5- | 4 | 434 | 422 |
| 2 | 5 | 4 | 336 | 324- |
| 2- | 5 | 4 | 568 | 523- |
| 2 | 5- | 4 | 167 | 118- |
| 2- | 5- | 4 | 333 | 334 |
| 3 | 5 | 4 | 378 | 329- |
| 3- | 5 | 4 | 180 | 157- |
| 3 | 5- | 4 | 167 | 164- |
| 3- | 5- | 4 | 346 | 360 |
| 4 | 5 | 4 | 269 | 305- |
| 4- | 5 | 4 | 206 | 209 |
| 4 | 5- | 4 | 202 | 233- |
| 4- | 5- | 4 | 580 | 686 |
| 5- | 5 | 4 | 143 | 154 |
| 5- | 5- | 4 | 256 | 284 |
| 6- | 5 | 4 | 127 | 117 |
| 6- | 5- | 4 | 222 | 207 |
| 7- | 5 | 4 | 366 | 392 |
| 7- | 5- | 4 | 230 | 229 |
| 8- | 5- | 4 | 93 | 102 |
| 0 | 6 | 4 | 485 | 498- |
| 0 | 6- | 4 | 415 | 431 |
| 1 | 6 | 4 | 423 | 369- |
| 1- | 6 | 4 | 532 | 569- |
| 1 | 6- | 4 | 524 | 547 |
| 1- | 6- | 4 | 510 | 476 |
| 2 | 6 | 4 | — | 59- |
| 2- | 6 | 4 | 495 | 484- |
| 2 | 6- | 4 | 301 | 298 |
| 2- | 6- | 4 | 909 | 940 |
| 3 | 6 | 4 | 169 | 148- |
| 3- | 6 | 4 | 447 | 470- |
| 3 | 6- | 4 | 122 | 106- |
| 3- | 6- | 4 | 322 | 338 |
| 4 | 6 | 4 | — | 21 |
| 4- | 6 | 4 | 284 | 231- |
| 4 | 6- | 4 | — | 95 |
| 4- | 6- | 4 | 303 | 274 |
| 5- | 6 | 4 | 125 | 119- |
| 5- | 6- | 4 | 309 | 307 |
| 6- | 6 | 4 | — | 1- |
| 6- | 6- | 4 | 99 | 98- |
| 7- | 6 | 4 | 87 | 94 |
| 7- | 6- | 4 | 182 | 176- |
| 8- | 6- | 4 | 81 | 86- |
| 0 | 7 | 4 | 140 | 151- |
| 0 | 7- | 4 | 403 | 428 |
| 1 | 7 | 4 | 225 | 221- |
| 1- | 7 | 4 | 264 | 254- |
| 1 | 7- | 4 | 460 | 497 |
| 1- | 7- | 4 | 329 | 308 |
| 2 | 7 | 4 | 123 | 121 |
| 2- | 7 | 4 | 535 | 606- |
| 2 | 7- | 4 | 266 | 270 |
| 2- | 7- | 4 | 233 | 197 |
| 3 | 7 | 4 | 255 | 303 |
| 3- | 7 | 4 | 362 | 401- |
| 3 | 7- | 4 | 289 | 328 |
| 3- | 7- | 4 | 304 | 278 |
| 4- | 7 | 4 | 285 | 234- |
| 4- | 7- | 4 | 146 | 132- |
| 5- | 7 | 4 | 488 | 491- |
| 5- | 7- | 4 | 198 | 175- |
| 6- | 7 | 4 | 196 | 239- |
| 6- | 7- | 4 | 335 | 329- |
| 7- | 7 | 4 | 97 | 115- |
| 7- | 7- | 4 | 303 | 298- |
| 8- | 7- | 4 | 282 | 302- |
| 0 | 8 | 4 | 216 | 204 |
| 0 | 8- | 4 | — | 45 |
| 1 | 8 | 4 | 345 | 331 |
| 1- | 8 | 4 | — | 29 |
| 1 | 8- | 4 | 373 | 394 |
| 1- | 8- | 4 | 152 | 144- |
| 2 | 8 | 4 | 266 | 295 |
| 2- | 8 | 4 | — | 56- |
| 2 | 8- | 4 | 278 | 264 |
| 2- | 8- | 4 | — | 9 |
| 3 | 8 | 4 | 264 | 325 |
| 3- | 8 | 4 | — | 16- |
| 3 | 8- | 4 | 289 | 342 |
| 3- | 8- | 4 | 364 | 337- |
| 4- | 8 | 4 | 434 | 371- |
| 4- | 8- | 4 | 514 | 512- |
| 5- | 8 | 4 | 303 | 302- |
| 5- | 8- | 4 | 305 | 308- |
| 6- | 8 | 4 | 194 | 235- |
| 6- | 8- | 4 | 272 | 270- |
| 7- | 8- | 4 | 420 | 428- |
| 0 | 9 | 4 | 432 | 455 |
| 0 | 9- | 4 | — | 22- |
| 1 | 9 | 4 | 232 | 252 |
| 1- | 9 | 4 | 217 | 222 |
| 1 | 9- | 4 | 197 | 213- |
| 1- | 9- | 4 | 490 | 448- |
| 2 | 9 | 4 | 273 | 304 |
| 2- | 9 | 4 | 131 | 136 |
| 2 | 9- | 4 | — | 14- |
| 2- | 9- | 4 | 575 | 502- |
| 3- | 9 | 4 | 167 | 148 |
| 3- | 9- | 4 | 463 | 409- |
| 4- | 9 | 4 | — | 11- |
| 4- | 9- | 4 | 497 | 486- |
| 5- | 9 | 4 | 164 | 169- |
| 5- | 9- | 4 | 491 | 465- |
| 6- | 9- | 4 | 189 | 181- |
| 7- | 9- | 4 | 143 | 117- |
| 0 | 10 | 4 | 224 | 275 |
| 0 | 10- | 4 | 341 | 383- |
| 1 | 10 | 4 | 323 | 329 |
| 1- | 10 | 4 | 154 | 189 |
| 1 | 10- | 4 | 278 | 322- |
| 1- | 10- | 4 | 300 | 312- |
| 2 | 10 | 4 | — | 56 |
| 2- | 10 | 4 | 467 | 433 |
| 2 | 10- | 4 | 156 | 198- |
| 2- | 10- | 4 | 437 | 366- |
| 3- | 10 | 4 | 343 | 329 |
| 3- | 10- | 4 | 286 | 225- |
| 4- | 10 | 4 | 107 | 106 |
| 4- | 10- | 4 | 280 | 254- |
| 5- | 10- | 4 | — | 6- |
| 6- | 10- | 4 | — | 29 |
| 0 | 11 | 4 | — | 90 |
| 0 | 11- | 4 | 204 | 221- |
| 1- | 11 | 4 | 232 | 223 |
| 1 | 11- | 4 | 334 | 417- |
| 1- | 11- | 4 | 220 | 225- |
| 2- | 11 | 4 | 250 | 254 |
| 2- | 11- | 4 | 317 | 318- |
| 3- | 11 | 4 | 237 | 297 |
| 3- | 11- | 4 | — | 23 |
| 4- | 11- | 4 | 241 | 205 |
| 5- | 11- | 4 | — | 59 |
| 6- | 11- | 4 | 263 | 271 |
| 0 | 12- | 4 | 86 | 96- |
| 1- | 12- | 4 | — | 31 |
| 2- | 12- | 4 | 110 | 123 |
| 3- | 12- | 4 | 105 | 123 |
| 4- | 12- | 4 | 292 | 314 |
| 5- | 12- | 4 | 258 | 301 |
| 2- | 13- | 4 | — | 193 |
| 3- | 13- | 4 | — | 345 |
| 0 | 0 | 5 | 123 | 90 |
| 1 | 0 | 5 | 149 | 163- |
| 1- | 0 | 5 | — | 100 |
| 2 | 0 | 5 | 307 | 279- |
| 2- | 0 | 5 | 284 | 307 |
| 3 | 0 | 5 | 282 | 277- |
| 3- | 0 | 5 | 530 | 645 |
| 4 | 0 | 5 | 233 | 283- |
| 4- | 0 | 5 | 454 | 406 |
| 5- | 0 | 5 | 204 | 193 |
| 6- | 0 | 5 | 510 | 451 |
| 7- | 0 | 5 | 327 | 307 |
| 8- | 0 | 5 | 97 | 105 |
| 9- | 0 | 5 | — | 123 |
| 0 | 1 | 5 | 213 | 209- |
| 0 | 1- | 5 | 247 | 215 |
| 1 | 1 | 5 | 616 | 588- |
| 1- | 1 | 5 | 128 | 102- |
| 1 | 1- | 5 | 104 | 54- |
| 1- | 1- | 5 | 677 | 752 |
| 2 | 1 | 5 | 459 | 413- |
| 2- | 1 | 5 | 282 | 307- |
| 2 | 1- | 5 | 220 | 212 |
| 2- | 1- | 5 | 462 | 546 |
| 3 | 1 | 5 | 267 | 242- |
| 3- | 1 | 5 | 226 | 189 |
| 3 | 1- | 5 | — | 55- |
| 3- | 1- | 5 | 525 | 598 |
| 4 | 1 | 5 | 244 | 314- |
| 4- | 1 | 5 | 522 | 399 |
| 4 | 1- | 5 | 226 | 240- |
| 4- | 1- | 5 | 712 | 717 |
| 5- | 1 | 5 | 273 | 244 |
| 5- | 1- | 5 | 211 | 200 |
| 6- | 1 | 5 | 402 | 365 |
| 6- | 1- | 5 | 162 | 64 |
| 7- | 1 | 5 | 480 | 439 |
| 7- | 1- | 5 | 244 | 211 |
| 8- | 1 | 5 | 307 | 324 |
| 8- | 1- | 5 | 95 | 67- |
| 9- | 1- | 5 | 163 | 174- |
| 0 | 2 | 5 | 369 | 343- |
| 0 | 2- | 5 | 220 | 226 |
| 1 | 2 | 5 | 514 | 487- |
| 1- | 2 | 5 | 724 | 731- |
| 1 | 2- | 5 | 421 | 419 |
| 1- | 2- | 5 | 425 | 491 |
| 2 | 2 | 5 | 595 | 573- |
| 2- | 2 | 5 | 473 | 502- |
| 2 | 2- | 5 | 391 | 383 |
| 2- | 2- | 5 | 578 | 616 |
| 3 | 2 | 5 | 171 | 166- |
| 3- | 2 | 5 | 150 | 160- |
| 3 | 2- | 5 | 132 | 138 |
| 3- | 2- | 5 | 311 | 359 |
| 4 | 2 | 5 | — | 3 |
| 4- | 2 | 5 | 450 | 347- |
| 4 | 2- | 5 | 123 | 126 |

Table 3 (*cont.*)

| h | k | l | $10F_o$ | $10F_c$ | h | k | l | $10F_o$ | $10F_c$ | h | k | l | $10F_o$ | $10F_c$ | h | k | l | $10F_o$ | $10F_c$ | h | k | l | $10F_o$ | $10F_c$ | h | k | l | $10F_o$ | $10F_c$ | h | k | l | $10F_o$ | $10F_c$ |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 4- | 2- | 5 | 115 | 142 | 5- | 3 | 5 | 322 | 272- | 6- | 4- | 5 | 465 | 495- | 0 | 6 | 5 | 358 | 318 | 1- | 7- | 5 | 235 | 217- | 4- | 8- | 5 | 286 | 300 | 1 | 10- | 5 | 245 | 320 |
| 5- | 2 | 5 | — | 31- | 5- | 3- | 5 | 223 | 245- | 7- | 4 | 5 | 311 | 358- | 0 | 6- | 5 | 417 | 425- | 2 | 7 | 5 | 141 | 144 | 5- | 8 | 5 | 226 | 249 | 1- | 10- | 5 | 379 | 392 |
| 5- | 2- | 5 | 123 | 21 | 6- | 3 | 5 | 229 | 216- | 7- | 4- | 5 | 450 | 464- | 1 | 6 | 5 | 471 | 459 | 2- | 7 | 5 | 331 | 315 | 5- | 8- | 5 | 317 | 343 | 2- | 10 | 5 | 238 | 319- |
| 6- | 2 | 5 | 310 | 247 | 6- | 3- | 5 | 535 | 529- | 8- | 4 | 5 | 165 | 164- | 1- | 6 | 5 | 315 | 300 | 2 | 7- | 5 | 257 | 295- | 6- | 8 | 5 | 264 | 274 | 2- | 10- | 5 | 250 | 263 |
| 6- | 2- | 5 | — | 85- | 7- | 3 | 5 | 160 | 139- | 8- | 4- | 5 | 179 | 169- | 1 | 6- | 5 | 195 | 204- | 2- | 7- | 5 | 201 | 184- | 6- | 8- | 5 | 394 | 452 | 3- | 10 | 5 | 178 | 129- |
| 7- | 2 | 5 | 130 | 101 | 7- | 3- | 5 | 301 | 306- | | | | | | 1- | 6- | 5 | 855 | 819- | 3- | 7 | 5 | 277 | 265 | 7- | 8- | 5 | 170 | 170 | 3- | 10- | 5 | 153 | 124 |
| 7- | 2- | 5 | 195 | 191- | 8- | 3 | 5 | 73 | 87 | | | | | | 2 | 6 | 5 | 241 | 221 | 3- | 7- | 5 | 301 | 191 | | | | | | 4- | 10 | 5 | — | 169- |
| 8- | 2 | 5 | 175 | 192 | 8- | 3- | 5 | 218 | 238- | 0 | 5 | 5 | 240 | 209 | 2- | 6 | 5 | 483 | 452 | 4- | 7 | 5 | 526 | 465 | | | | | | 4- | 10- | 5 | — | 45 |
| 8- | 2- | 5 | 280 | 283- | 9- | 3- | 5 | 280 | 282- | 0 | 5- | 5 | 402 | 391- | 2 | 6- | 5 | 342 | 388- | 4- | 7- | 5 | 277 | 262 | 0 | 9 | 5 | 217 | 222- | 5- | 10- | 5 | — | 15 |
| 9- | 2- | 5 | 222 | 240- | | | | | | 1 | 5 | 5 | 438 | 414 | 2- | 6- | 5 | 437 | 398- | 5- | 7 | 5 | 247 | 233 | 0 | 9- | 5 | 266 | 311 | 6- | 10- | 5 | — | 49- |
| | | | | | | | | | | 1- | 5 | 5 | 362 | 326 | 3 | 6 | 5 | — | 13 | 5- | 7- | 5 | 226 | 251 | 1 | 9 | 5 | 365 | 399- | | | | | |
| | | | | | 0 | 4 | 5 | 149 | 64 | 1 | 5- | 5 | 206 | 192- | 3- | 6 | 5 | 240 | 256 | 6- | 7 | 5 | — | 24 | 1- | 9 | 5 | — | 66- | | | | | |
| 0 | 3 | 5 | 216 | 217- | 0 | 4- | 5 | 119 | 91- | 1- | 5- | 5 | 591 | 549- | 3 | 6- | 5 | 141 | 182- | 6- | 7- | 5 | 314 | 348 | 1 | 9- | 5 | — | 23 | 0 | 11- | 5 | 212 | 256 |
| 0 | 3- | 5 | 199 | 213 | 1 | 4 | 5 | 359 | 343 | 2 | 5 | 5 | 471 | 442 | 3- | 6- | 5 | 156 | 171- | 7- | 7- | 5 | 314 | 356 | 1- | 9- | 5 | 447 | 451 | 1- | 11- | 5 | 310 | 319 |
| 1 | 3 | 5 | 259 | 250- | 1- | 4 | 5 | 260 | 235- | 2- | 5 | 5 | 257 | 245 | 4- | 6 | 5 | 124 | 101 | 8- | 7- | 5 | 221 | 261 | 2- | 9 | 5 | 168 | 172- | 2- | 11- | 5 | — | 9- |
| 1- | 3 | 5 | 344 | 326- | 1 | 4- | 5 | 300 | 317 | 2 | 5- | 5 | — | 86- | 4- | 6- | 5 | 222 | 262- | | | | | | 2 | 9- | 5 | — | 104 | 3- | 11- | 5 | 232 | 164- |
| 1 | 3- | 5 | 350 | 376 | 1- | 4- | 5 | 371 | 303- | 2- | 5- | 5 | 601 | 589- | 5- | 6 | 5 | — | -8 | | | | | | 2- | 9- | 5 | 267 | 224 | 4- | 11- | 5 | — | 62- |
| 1- | 3- | 5 | 525 | 507 | 2 | 4 | 5 | 126 | 134 | 3 | 5 | 5 | 229 | 236 | 5- | 6- | 5 | 132 | 87 | 0 | 8 | 5 | — | 37 | 3- | 9 | 5 | — | 11- | 5- | 11- | 5 | 270 | 259- |
| 2 | 3 | 5 | 143 | 124- | 2- | 4 | 5 | 140 | 109- | 3- | 5 | 5 | 190 | 167- | 6- | 6 | 5 | — | 70- | 0 | 8- | 5 | 104 | 66 | 3- | 9- | 5 | 393 | 350 | 6- | 11- | 5 | 323 | 337- |
| 2- | 3 | 5 | 461 | 464- | 2 | 4- | 5 | 159 | 200 | 3 | 5- | 5 | — | 80 | 6- | 6- | 5 | 225 | 248 | 1 | 8 | 5 | — | 103- | 4- | 9 | 5 | 267 | 258 | | | | | |
| 2 | 3- | 5 | 520 | 567 | 2- | 4- | 5 | 143 | 120- | 3- | 5- | 5 | 469 | 526- | 7- | 6 | 5 | 102 | 125- | 1- | 8 | 5 | — | 56 | 4- | 9- | 5 | 511 | 473 | | | | | |
| 2- | 3- | 5 | 219 | 202 | 3 | 4 | 5 | 264 | 264 | 4- | 5 | 5 | 160 | 134- | 7- | 6- | 5 | 94 | 107 | 1 | 8- | 5 | — | 65- | 5- | 9 | 5 | 183 | 197 | 1- | 12- | 5 | — | 118- |
| 3 | 3 | 5 | 126 | 129 | 3- | 4 | 5 | 399 | 410- | 4- | 5- | 5 | 215 | 290- | 8- | 6- | 5 | 213 | 254 | 1- | 8- | 5 | — | 35 | 5- | 9- | 5 | 230 | 227 | 2- | 12- | 5 | 94 | 119- |
| 3- | 3 | 5 | 563 | 626- | 3 | 4- | 5 | 211 | 224 | 5- | 5 | 5 | 124 | 104- | | | | | | 2 | 8 | 5 | 139 | 183- | 6- | 9- | 5 | 197 | 193 | 3- | 12- | 5 | 282 | 322- |
| 3 | 3- | 5 | 346 | 374 | 3- | 4- | 5 | 410 | 486- | 5- | 5- | 5 | 185 | 192- | | | | | | 2- | 8 | 5 | — | 65 | 7- | 9- | 5 | 138 | 131 | 4- | 12- | 5 | 298 | 332- |
| 3- | 3- | 5 | 245 | 287- | 4- | 4 | 5 | 757 | 730- | 6- | 5 | 5 | 315 | 334- | 0 | 7 | 5 | 203 | 200 | 2 | 8- | 5 | 169 | 194- | | | | | | 5- | 12- | 5 | — | 242- |
| 4 | 3 | 5 | — | 140 | 4- | 4- | 5 | 575 | 644- | 6- | 5- | 5 | 278 | 285- | 0 | 7- | 5 | 113 | 134- | 2- | 8- | 5 | 311 | 248 | | | | | | | | | | |
| 4- | 3 | 5 | 465 | 498- | 5- | 4 | 5 | 388 | 354- | 7- | 5 | 5 | 194 | 231- | 1 | 7 | 5 | 177 | 158 | 3- | 8 | 5 | 252 | 285 | 0 | 10 | 5 | — | 230- | | | | | |
| 4 | 3- | 5 | 169 | 247 | 5- | 4- | 5 | 322 | 364- | 7- | 5- | 5 | 126 | 101- | 1- | 7 | 5 | 499 | 490 | 3- | 8- | 5 | 524 | 469 | 0 | 10- | 5 | 323 | 355 | | | | | |
| 4- | 3- | 5 | — | 39- | 6- | 4 | 5 | 243 | 264- | 8- | 5- | 5 | 108 | 122 | 1 | 7- | 5 | 303 | 334- | 4- | 8 | 5 | 392 | 347 | 1- | 10 | 5 | 310 | 341- | | | | | |

Table 4. *Main interatomic distances and angles**

### Anion

I–O(2)$_{\text{I}}$ = 1·992 ± 0·012 Å
I–O(2)$_{\text{II}'}$ = 2·017 ± 0·007
I–O(1)$_{\text{I}}$ = 1·980 ± 0·016
I–O(3) = 1·793 ± 0·016
I–O(4) = 1·810 ± 0·015
I–O(5)$_{\text{I}}$ = 1·818 ± 0·010

O(2)$_{\text{I}}$–I–O(4) = 88·2 ± 0·6°
O(4)–I–O(3) = 97·1 ± 0·7
O(3)–I–O(1)$_{\text{I}}$ = 89·6 ± 0·7
O(1)$_{\text{I}}$–I–O(2)$_{\text{I}}$ = 83·9 ± 0·6
O(5)$_{\text{I}}$–I–O(4) = 97·6 ± 0·6
O(5)$_{\text{I}}$–I–O(3) = 100·0 ± 0·7
O(5)$_{\text{I}}$–I–O(1)$_{\text{I}}$ = 87·0 ± 0·7
O(5)$_{\text{I}}$–I–O(2)$_{\text{I}}$ = 92·0 ± 0·6
O(2)$_{\text{II}'}$–I–O(4) = 88·0 ± 0·5
O(2)$_{\text{II}'}$–I–O(3) = 89·6 ± 0·5
O(2)$_{\text{II}'}$–I–O(1)$_{\text{I}}$ = 86·2 ± 0·6
O(2)$_{\text{II}'}$–I–O(2)$_{\text{I}}$ = 77·6 ± 0·4

### Hydrogen bonds O · · · $H_2O$

O(5)$_{\text{I}}$–O(8) = 2·972 ± 0·021 Å
O(5)′–O(8) = 2·930 ± 0·020
O(4)–O(7) = 2·792 ± 0·018
O(5)$_{\text{III}}$–O(7) = 2·842 ± 0·024
O(3)–O(9) = 2·800 ± 0·020
O(5)–O(9) = 2·854 ± 0·026
O(4)–O(6) = 2·872 ± 0·026
O(3)$_{\text{III}}$–O(6) = 2·958 ± 0·026

O(5)$_{\text{I}}$–O(8)–O(5)′ = 108·9 ± 0·8°
O(4)–O(7)–O(5)$_{\text{III}}$ = 123·3 ± 0·6
O(3)–O(9)–O(5) = 121·1 ± 0·7
O(4)–O(6)–O(3)$_{\text{III}}$ = 113·0 ± 0·6

### Coordination around K(1)

K(1)–O(2) = 2·705 ± 0·014 Å
K(1)–O(3) = 2·941 ± 0·017
K(1)–O(3)$_{\text{II}'}$ = 2·868 ± 0·013
K(1)–O(9) = 3·065 ± 0·019

K(1)–O(6)$_{\text{IV}}$ = 2·860 ± 0·016 Å
K(1)–O(4) = 2·892 ± 0·014
K(1)–O(7) = 3·055 ± 0·016
K(1)–O(1)$_{\text{III}}$ = 2·653 ± 0·017

### Coordination around K(2)

K(2)–O(8)$_{\text{IV}}$ = 2·786 ± 0·020 Å
K(2)–O(9) = 2·783 ± 0·020
K(2)–O(7) = 2·766 ± 0·017
K(2)–O(9)′ = 2·920 ± 0·020

K(2)–O(8)$_{\text{III}'}$ = 2·749 ± 0·020 Å
K(2)–O(6)$_{\text{IV}}$ = 3·053 ± 0·016
K(2)–O(6)$_{\text{III}'}$ = 3·101 ± 0·015
K(2)–O(7)$_{\text{III}'}$ = 2·866 ± 0·017

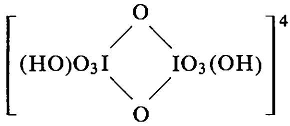
* Asymmetric units (in fractional coordinates).

| No label | $x$ | $y$ | $z$ |
|---|---|---|---|
| ′ | $1-x$ | $1-y$ | $1-z$ |
| I | $x-1$ | $y$ | $z$ |
| II′ | $1-x$ | $-y$ | $1-z$ |
| III | $x$ | $y$ | $1+z$ |
| III′ | $1-x$ | $1-y$ | $2-z$ |
| IV | $1+x$ | $y$ | $z$ |
| V′ | $-x$ | $-y$ | $1-z$ |

observed and calculated structure factors are compared in Table 3. The main interatomic distances and angles have been calculated (Table 4). The e.s.d.'s in the coordinates were obtained by Cruickshank's (1949) method, the e.s.d.'s of the distances by Ahmed & Cruickshank (1953) method and the e.s.d.'s of the angles by Darlow (1960) method.

## Discussion of the structure

The crystal structure of the compound (Fig. 2) is formed by anions



$$\left[(HO)O_3I\begin{matrix}O\\ \\O\end{matrix}IO_3(OH)\right]^{4-}$$

or written more simply $[H_2I_2O_{10}]^{4-}$, interposed between $K^+$ cations and $H_2O$ molecules. These water molecules, corresponding to O(6), O(7), O(8) and O(9), form a shell around the anion, to which they are bound by hydrogen bonds. Each water molecule forms two such hydrogen bonds with two anions, so giving rise to a three-dimensional network all over the structure. The hydrogen bond distances are in the range 2·79–2·97 Å. The angles O–$H_2O$–O between two hydrogen bonds formed by the same water molecule have values 109°, 113°, 121° and 123°.

The anion $[H_2I_2O_{10}]^{4-}$ has the configuration shown in Fig. 3. It can be regarded formally as obtained by condensation of two pseudo-octahedral groups $[H_3IO_6]^{2-}$ and elimination of $2H_2O$, although the actual chemical process might be different; after condensation, the two octahedra have an edge in common. The two atoms O(2) and O(2′), lying on the common edge, form bridges between two I atoms. The location of the hydrogen atoms is only probable but it is supported by the following considerations. The bonds between iodine and the six neighbouring oxygen atoms are not all equivalent; three bonds, namely I–O(1), I–O(2) and I–O(2′) have an average value $I\text{–}O^*_{av} = 1{\cdot}996$ Å (maximum deviation ±0·021 Å) and the remaining three, namely I–O(3), I–O(4) and I–O(5), have an average value $I\text{–}O_{av} = 1{\cdot}807$ Å (maximum deviation ±0·011 Å). One possible explanation of this difference is that I–O(2) and I–O(2′) are longer because O(2) and O(2′) are shared between two iodine atoms, while I–O(1) is longer because O(1) is actually part of a hydroxyl group, *i.e.* O(1) is shared between I and H.

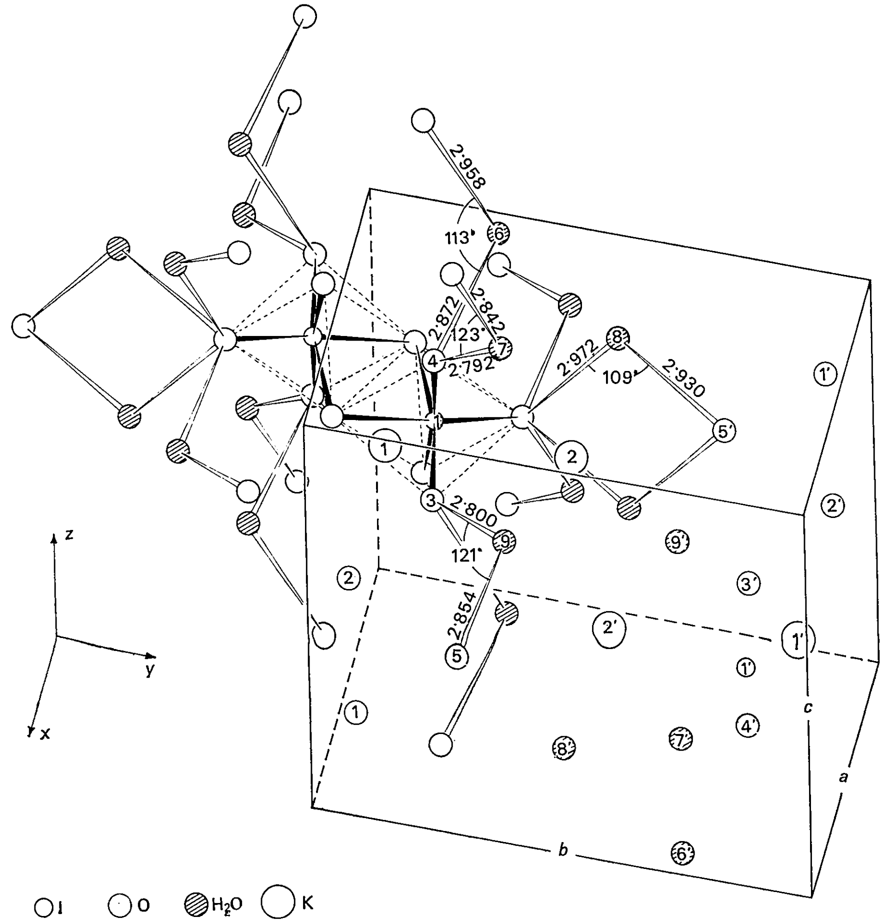


Fig. 2. Crystal structure of $K_4H_2I_2O_{10}$ . $8H_2O$. Numbered atoms belong to the basic unit cell. Two octahedra forming an anion $[H_2I_2O_{10}]^{4-}$ are dotted, heavy lines indicating I–O bonds. Hydrogen bond distances and angles O–$H_2O$–O are quoted.

The distance I–O(1), however, is longer than the corresponding distances in orthoperiodic acid, $(HO)_5IO$, where $I\text{–}OH_{av} = 1{\cdot}89 \pm 0{\cdot}02$ Å (Feikema, 1963) and in $HI_3O_8$ where I–OH = 1·90 Å (Feikema & Vos, 1963). The lengths of the three bonds I–O with unshared O [I–O(3) = 1·79 Å, I–O(4) = 1·81 Å and I–O(5) = 1·82 Å] are practically equal to the distance I–O = 1·79 Å found in the tetrahedral group $[IO_4]^-$ (Hazlewood, 1938) and the distance I–O = 1·78 Å found in orthoperiodic acid (Feikema, 1963). The same distances are shorter than the distances I–O = 1·93 Å found in the single octahedral group $[IO_6]^{5-}$ (Helmholz, 1937). Comparison with the distances between iodine and oxygen in $HI_3O_8$ (Feikema & Vos, 1963) shows surprising similarities in bond distances, despite the different valence of iodine. In the structure of $HI_3O_8$, which is an addition compound of $I_2O_5$ and $HIO_3$, the distances between iodine and oxygen are $I\text{–}O_{av} = 1{\cdot}97$ Å for shared oxygen in the molecule $I_2O_5$ and $I\text{–}O_{av} = 1{\cdot}78$ Å and $I\text{–}O_{av} = 1{\cdot}84$ Å for unshared oxygen in $I_2O_5$ and $HIO_3$, respectively.

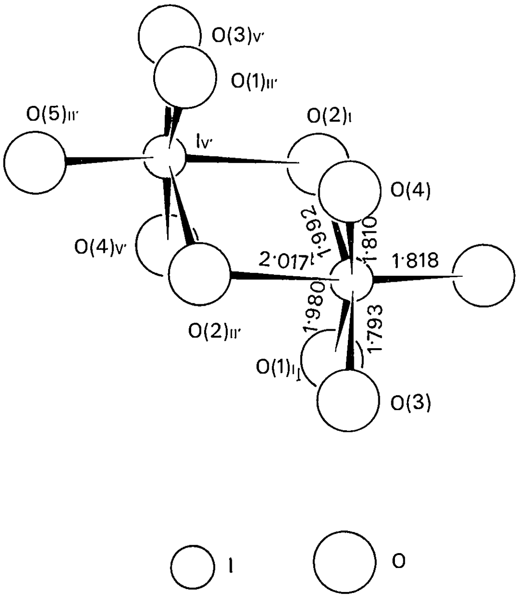


Fig. 3. The anion $[H_2I_2O_{10}]^{4-}$. The hydrogen atoms are probably bound to $O(1)_I$ and $O(1)_{II'}$ (for labelling of asymmetric units see Table 4).

The distances found are in agreement with a diminution of the double bond character of the bond I–O, when passing from bonds with unshared oxygen to bonds with shared oxygen atoms. The double bond character prevails in bonds I–O of the tetrahedral group $[IO_4]^-$ and in the bond I–O of the acid $(HO)_5IO$, but it is lowered in the bonds I–OH of the acid. The excess of double bond character causes the formation of polynuclear anions; in these the excess is eliminated at those atoms which bridge two groups and therefore the single bond character prevails in those bonds I–O where the oxygen atom is shared. The difference between bonds with shared and bonds with unshared atoms is normal, *e.g.* distances P–O in $Na_4P_2O_7 . 10H_2O$, which are P–O = 1·47 Å, unshared and P–O = 1·63 Å, shared (MacArthur & Beevers, 1957). The two iodine atoms I and $I_{V'}$ and the oxygen atoms O(3), $O(5)_I$, $O(2)_I$, $O(2)_{II'}$, $O(5)_{II'}$ and $O(3)_{V'}$, all belonging to the same anion, are not exactly in the same plane. The plane of best fit (Schomaker, Waser, Marsh & Bergman, 1959) is represented by

$$1{\cdot}36644x + 2{\cdot}23739y + 5{\cdot}53956z - 2{\cdot}66978 = 0.$$

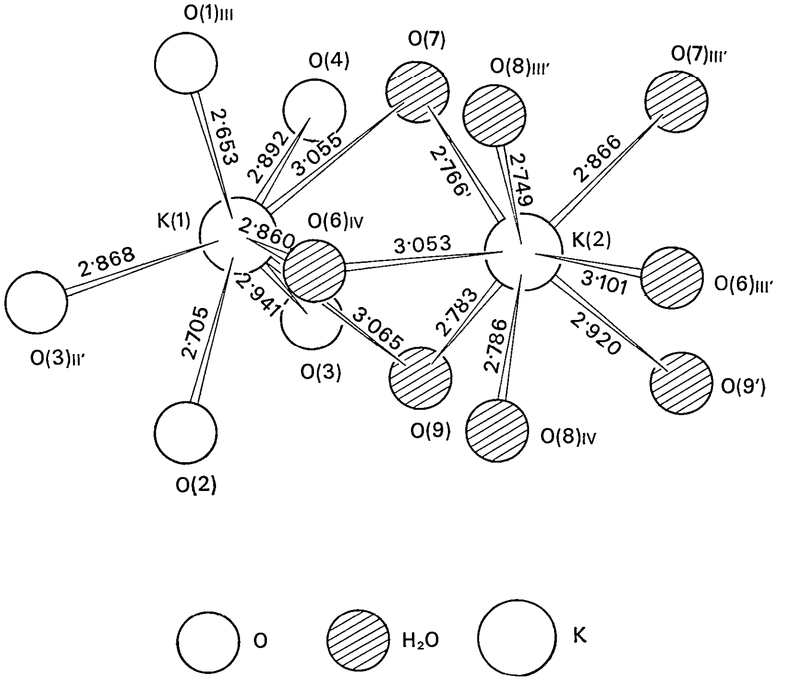


Fig. 4. Potassium ions K(1) and K(2) and surrounding atoms (for labelling of asymmetric units see Table 4).

The deviations are +0·0903 Å for I, −0·0002 Å for $O(2)_I$, −0·0262 Å for O(3) and −0·0267 Å for $O(5)_I$ and deviations with opposite signs for centrosymmetrically related atoms of the anion. The iodine atom is significantly out of the plane and the distortion is in agreement with the fact that the bonds with respect to the plane are not equal, one bond I–OH being faced by one bond I–O.

Only the unshared oxygen atoms form hydrogen bonds with the surrounding water molecules.

The two K atoms are surrounded each by eight oxygen atoms or water molecules (Fig. 4). The coordination polyhedra differ in both form and nature for the two atoms. K(2) is surrounded by eight water molecules forming a distorted square antiprism, while K(1) is surrounded by five anionic oxygen atoms and by three water molecules. These molecules are common with the antiprism around K(2) and form a screen between the two positive ions. The distances K–O fall in the range 2·65–3·10 Å in accordance with values quoted by *International Tables for X-ray Crystallography* (1962).

The results of the structure determination show that the anion of the salt studied is correctly formulated $[H_2I_2O_{10}]^{4-}$ and the salt should be called tetrapotassium dihydrogendecaoxodiiodate(VII) octahydrate (*Nomenclature of Inorganic Chemistry*, 1959). All the so-called mesoperiodates which Siebert (1960*a*, *b*) was able to identify on the basis of the characteristic infrared spectra must, whether hydrated or not, be regarded as salts of the same acid. There is a possible indication from some chromatograms prepared in this laboratory that anions derived from decaoxodiiodic (VII) acid can also exist in solution.

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## References

AHMED, F. R. & CRUICKSHANK, D. W. J. (1953). *Acta Cryst.* **6**, 385.

BLOMSTRAND, C. W. (1886). *J. Prakt. Chem.* [2], **34**, 455.

BOND, W. L. (1959). *Acta Cryst.* **12**, 375.

BOOTH, A. D. (1946). *Trans. Faraday Soc.* **42**, 444.

DARLOW, S. F. (1960). *Acta Cryst.* **13**, 683.

FEIKEMA, Y. D. (1961). *Acta Cryst.* **14**, 315.

FEIKEMA, Y. D. (1963). Thesis, Groningen.

FEIKEMA, Y. D. & VOS, A. (1963). *Acta Cryst.* **16**, A38.

FERRARI, A., CURTI, R. & CAVALCA, L. (1944). *Gazz. chim. ital.* **74**, 43.

FORSYTH, J. B. & WELLS, M. (1959). *Acta Cryst.* **12**, 412.

GROTH, P. (1908). *Chemische Kristallographie,* Vol. II, p. 182. Leipzig: Engelmann.

HAZLEWOOD, E. A. (1938). *Z. Kristallogr.* A, **98**, 439.

HELMHOLZ, L. (1937). *J. Amer. Chem. Soc.* **59**, 2036.

HILL, A. E. (1928). *J. Amer. Chem. Soc.* **50**, 2678.

*International Tables for X-ray Crystallography* (1959). Vol. II, p. 106. Birmingham: Kynoch Press.

*International Tables for X-ray Crystallography* (1962). Vol. III, p. 258. Birmingham: Kynoch Press.

JAEGER, F. M. & BEINTEMA, J. (1933). *Proc. Acad. Amsterdam,* **36**, 523.

KIMMINS, C. W. (1889). *Chem. News,* **59**, 57.

MACARTHUR, D. M. & BEEVERS, C. A. (1957). *Acta Cryst.* **10**, 428.

MOORE, F. H. (1963). *Acta Cryst.* **16**, 1169.

NARDELLI, M., FAVA, G. & GIRALDI, G. (1963). *Acta Cryst.* **16**, 343.

*Nomenclature of Inorganic Chemistry* (1959), p. 46. London: Butterworths.

PAČESOVÁ, L. & HAUPTMANN, Z. (1963). *Z. anorg. Chem.* **325**, 325.

PARTINGTON, J. R. & BAHL, R. K. (1934). *J. Chem. Soc.* p. 1088.

RAE, N. (1931). *J. Chem. Soc.* p. 876.

RAMMELSBERG, C. F. (1868). *Ann. Phys. Lpz.* **134**, 371.

SCHOMAKER, V., WASER, J., MARSH, R. E. & BERGMAN, G. *Acta Cryst.* **12**, 600.

SIEBERT, H. (1960*a*). *Z. anorg. Chem.* **303**, 162.

SIEBERT, H. (1960*b*). *Z. anorg. Chem.* **304**, 266.