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# The Crystal and Molecular Structure of Racemic Potassium Di-μ-tartrato-diantimonate(III) Trihydrate (Racemic 'Tartar Emetic')

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Three-dimensional X-ray analysis shows that racemic potassium antimonyl tartrate is the racemic mixture of potassium di-μ-(+)-tartratodiantimonate(III) and di-μ-(−)-tartratodiantimonate(III) with three water molecules of crystallization. There are four formula units of $K_2[Sb_2(C_4H_2O_6)_2].3H_2O$ in the orthorhombic unit cell of dimensions $a=8{\cdot}79$, $b=16{\cdot}32$, $c=12{\cdot}19$ Å and space group $Pca2_1$. Two antimony atoms are bridged by two tartrate groups which act as double bidentate ligands through the deprotonated carboxyl group and α-hydroxyl oxygen atoms with mean Sb–O distances of 2·20 and 2·01 Å respectively. The chelate rings are nearly planar and the oxygen atoms about the antimony atom occupy four corners of a slightly distorted square pyramid, whose apex belongs to the unshared electron pair. Two of the three molecules of water per formula unit form a three-dimensional hydrogen-bonded network with tartrato-antimonate ions, while the third water molecule forms separate hydrogen bonds with the anion.

## Introduction

The crystal structures of four compounds considered to be 'antimonyl-tartrates' have been reported: ammonium antimonyl-(±)-tartrate monohydrate (Kiosse, Golovastikov & Belov, 1964); potassium antimonyl-(±)-tartrate hemihydrate (Grdenić & Kamenar, 1965), tris-*o*-phenanthroline-iron(II) antimony(+)-tartrate (Templeton, Zalkin & Ueki, 1966); ammonium antimonyl-(+)-tartrate hemihydrate (Kiosse, Golovastikov, Ablov & Belov, 1967). Essentially the same dimeric tartratoantimonate(III) ion $[Sb_2(C_4H_2O_6)_2]^{2-}$ was found in the crystal structures of ammonium compounds and the complex iron compound, but in the potassium compound a monomeric ion was reported. The latter result was obtained on the basis of only one projection and the structure was not completed at the time. The three-dimensional structure analysis, the results of which we now report, has shown that the dimeric tartratoantimonate ion occurs also in the crystal structure of racemic potassium antimonyl tartrate.

## Experimental

Racemic potassium antimonyl tartrate, known as hemihydrate, with the formula $KSbOC_4H_4O_6.\frac{1}{2}H_2O$ was prepared by adding antimony(III) oxide to a warm solution of racemic tartaric acid and potassium hydroxide in water. The crystals obtained were recrystallized from an aqueous solution.

Cell dimensions, determined from oscillation and Weissenberg photographs, were as follows:

$$a = 8{\cdot}79 \pm 0{\cdot}02 \text{ Å}$$
$$b = 16{\cdot}32 \pm 0{\cdot}02$$
$$c = 12{\cdot}19 \pm 0{\cdot}02$$

The systematic absences of $0kl$ for $l$ odd and $h0l$ for $h$ odd indicated $Pca2_1$ ($C_{2v}^5$) or $Pbcm$ ($D_{2h}^{11}$) as the possible space groups. The final choice of $Pca2_1$ was ratified in the course of the Patterson synthesis interpretation and by piezoelectric measurements. The cell contains 8 formula units $KSbOC_4H_4O_6.\frac{1}{2}H_2O$; measured density 2·510 g.cm$^{-3}$, calculated density 2·508 g.cm$^{-3}$. Three-dimensional intensity data were obtained up to the 10th layer around [001] and up to the 7th layer around [100] on an integrating Weissenberg camera using the multiple-film technique and Cu $K\alpha$ radiation. For the photographs taken around [001] the crystal was spherically ground to 0·43 mm in diameter and for the data taken around [100] a sphere of 0·50 mm in diameter was used. The intensities were measured by means of a densitometer and corrected for Lorentz and polarization factors, and for absorption ($\mu = 315$ cm$^{-1}$), and were placed on a common scale by the method of Rollett & Sparks (1960).

Structure factors, cycles of least-squares analysis, and Fourier syntheses were calculated by the use of J. S. Rollett's general crystal structure analysis program on the Oxford University English Electric Leo-Marconi KDF. 9 computer.

## Structure determination

A three-dimensional Patterson function sharpened to 'point atoms at rest' computed from 1873 independent reflexions gave the positions of the antimony and potassium atoms. The light atom positions were found by means of several successive Fourier syntheses. The structure was then refined by several cycles of least-squares using the data uncorrected for absorption. At this stage of refinement the reliability index was $R=0{\cdot}129$. After absorption correction the refinement pro-

Table 1. *Observed and calculated structure factors* ($\times 100$) *with calculated phases*

| h | k | l | $F_o$ | $F_c$ | $\alpha_{hkl}$ |
|---|---|---|---|---|---|
| 0 | 0 | 2 | 6215 | 7098 | 93 |
| | | 4 | 7125 | 7064 | -3 |
| | | 6 | 18862 | 22455 | -25 |
| | | 8 | 22640 | 26478 | 5 |
| | | 10 | 8126 | 9312 | 116 |
| | | 12 | 5885 | 7270 | -17 |
| | | 14 | 6258 | 7510 | -1 |
| 0 | 1 | 2 | 4839 | 9247 | -25 |
| | | 4 | 13065 | 18523 | -27 |
| | | 6 | 8985 | 10119 | 88 |
| | | 8 | 5483 | 7134 | -2 |
| | | 10 | 7535 | 8993 | -35 |
| | | 12 | 4632 | 4456 | 17 |
| | | 14 | 3980 | 4497 | 70 |
| 0 | 2 | 2 | 13955 | 16021 | -138 |
| | | 4 | 10794 | 10746 | 69 |
| | | 6 | 10727 | 11759 | 129 |
| | | 8 | 10539 | 11554 | -139 |
| | | 12 | 2815 | 2550 | -25 |
| | | 14 | 4773 | 5894 | 169 |
| 0 | 3 | 2 | 23549 | 29404 | 138 |
| | | 4 | 17387 | 18103 | -132 |
| | | 6 | 17988 | 20533 | -136 |
| | | 8 | 5436 | 4948 | 92 |
| | | 10 | 16120 | 18409 | 158 |
| | | 12 | 12038 | 14516 | -140 |
| | | 14 | 3435 | 2998 | -107 |
| 0 | 4 | 0 | 5409 | 6086 | 180 |
| | | 2 | 3154 | 2965 | 169 |
| | | 4 | 8583 | 7990 | -55 |
| | | 6 | 10985 | 12295 | 164 |
| | | 8 | 9403 | 9800 | -147 |
| | | 10 | 5791 | 5621 | 164 |
| | | 12 | 3615 | 3452 | 161 |
| | | 14 | 4718 | 5205 | 171 |
| 0 | 5 | 2 | 11297 | 11752 | -74 |
| | | 4 | 19158 | 21486 | 30 |
| | | 6 | 10222 | 10844 | 90 |
| | | 8 | 9058 | 9928 | -97 |
| | | 10 | 8680 | 9058 | -8 |
| | | 12 | 8607 | 9454 | 32 |
| | | 14 | 2855 | 3443 | 129 |
| 0 | 6 | 0 | 21758 | 19158 | 0 |
| | | 2 | 18659 | 20073 | 57 |
| | | 4 | 7566 | 8025 | -44 |
| | | 6 | 13980 | 15097 | -34 |
| | | 8 | 19650 | 21911 | 14 |
| | | 10 | 6367 | 5847 | 52 |
| | | 12 | 5954 | 6924 | -64 |
| | | 14 | 7884 | 10325 | 0 |
| 0 | 7 | 0 | 30836 | 32487 | 0 |
| | | 2 | 7561 | 7252 | -59 |
| | | 4 | 12425 | 12572 | 19 |
| | | 6 | 9390 | 9796 | 4 |
| | | 8 | 9661 | 8917 | -26 |
| | | 10 | 4466 | 4702 | 78 |
| | | 12 | 6064 | 7267 | 0 |
| | | 14 | 3443 | 1715 | 1 |
| 0 | 8 | 0 | 18287 | 16806 | 180 |
| | | 2 | 13153 | 12818 | -104 |
| | | 4 | 15224 | 15798 | -16 |
| | | 6 | 15245 | 16041 | 136 |
| | | 8 | 9708 | 9015 | -172 |
| | | 10 | 8323 | 7665 | -71 |
| | | 12 | 4275 | 5189 | 108 |
| | | 14 | 4969 | 5027 | 145 |
| 0 | 9 | 0 | 5505 | 4199 | 0 |
| | | 2 | 17176 | 17393 | 153 |
| | | 4 | 18695 | 19952 | 180 |
| | | 6 | 8285 | 7818 | -129 |
| | | 8 | 3558 | 2002 | 155 |
| | | 10 | 12259 | 12157 | 158 |
| | | 12 | 7788 | 8712 | -140 |
| 0 | 10 | 0 | 6248 | 5110 | 180 |
| | | 2 | 14179 | 14123 | 150 |
| | | 4 | 8022 | 8422 | -149 |
| | | 6 | 12143 | 12657 | -157 |
| | | 8 | 2994 | 2190 | 59 |
| | | 10 | 6856 | 6970 | -171 |
| | | 12 | 5112 | 6187 | -155 |
| 0 | 11 | 0 | 5806 | 5588 | 180 |
| | | 2 | 12658 | 11696 | -50 |
| | | 4 | 10681 | 9809 | 12 |
| | | 6 | 9335 | 8811 | 91 |
| | | 8 | 6990 | 7093 | -145 |
| | | 10 | 8454 | 8515 | -27 |
| | | 12 | 6126 | 6335 | 49 |
| 0 | 12 | 0 | 20786 | 19897 | 0 |
| | | 2 | 10985 | 10883 | 28 |
| | | 4 | 4328 | 4141 | -45 |
| | | 6 | 13939 | 13685 | -4 |
| | | 8 | 9567 | 8591 | 11 |
| | | 10 | 5004 | 5355 | 60 |
| | | 12 | 4130 | 4195 | -20 |
| 0 | 13 | 0 | 12035 | 11108 | 0 |
| | | 2 | 7855 | 6975 | 49 |
| | | 4 | 7684 | 6450 | -22 |
| | | 6 | 7466 | 8020 | -29 |
| | | 8 | 9504 | 10598 | 21 |
| | | 10 | 1865 | 1805 | 52 |
| 0 | 14 | 0 | 10548 | 10376 | 180 |
| | | 2 | 11202 | 10878 | -99 |
| | | 4 | 9510 | 9036 | 49 |
| | | 6 | 6707 | 6498 | 125 |
| | | 8 | 8454 | 8791 | -134 |
| | | 10 | 3418 | 3252 | -38 |
| 0 | 15 | 0 | 9532 | 9407 | 180 |
| | | 2 | 7353 | 6515 | 145 |
| | | 4 | 8756 | 8689 | -163 |
| | | 6 | 8627 | 8529 | -165 |
| | | 8 | 4706 | 4167 | 161 |
| 0 | 15 | 10 | 4785 | 4728 | 177 |
| 0 | 16 | 0 | 4563 | 4231 | 0 |
| | | 2 | 7048 | 6811 | 146 |
| | | 4 | 10562 | 11027 | -180 |
| | | 6 | 5701 | 5197 | -105 |
| | | 8 | 2191 | 2472 | 165 |
| 0 | 17 | 0 | 8420 | 8170 | 180 |
| | | 2 | 5129 | 4843 | -76 |
| | | 4 | 7954 | 8155 | 1 |
| | | 6 | 5741 | 6007 | 127 |
| | | 8 | 2191 | 2519 | -154 |
| 0 | 18 | 0 | 4675 | 5632 | 0 |
| | | 2 | 5875 | 5344 | 5 |
| | | 4 | 5073 | 4484 | 15 |
| | | 6 | 7068 | 6177 | -6 |
| 0 | 19 | 0 | 8700 | 10024 | 0 |
| | | 2 | 6284 | 6860 | 41 |
| | | 4 | 1985 | 2075 | -124 |
| 0 | 20 | 0 | 3297 | 2562 | 180 |
| | | 2 | 5395 | 4782 | -67 |
| 1 | 1 | 0 | 16705 | 20044 | 180 |
| | | 1 | 13945 | 14414 | 5 |
| | | 2 | 7427 | 7508 | -151 |
| | | 3 | 24632 | 30972 | 75 |
| | | 4 | 6121 | 6073 | 157 |
| | | 5 | 15009 | 17519 | 155 |
| | | 6 | 7924 | 8627 | 178 |
| | | 7 | 6303 | 7205 | -167 |
| | | 8 | 9166 | 11107 | -154 |
| | | 9 | 8592 | 9809 | 35 |
| | | 10 | 5985 | 6585 | -144 |
| | | 11 | 9880 | 9835 | 101 |
| | | 12 | 2751 | 2947 | 173 |
| | | 13 | 5973 | 6095 | 170 |
| | | 14 | 6026 | 6805 | 175 |
| 1 | 2 | 0 | 7570 | 7567 | -180 |
| | | 1 | 14629 | 17596 | 108 |
| | | 2 | 15889 | 20125 | 144 |
| | | 3 | 4499 | 2056 | -84 |
| | | 4 | 11140 | 11682 | -154 |
| | | 5 | 5129 | 5262 | 49 |
| | | 6 | 8842 | 9521 | -148 |
| | | 7 | 15478 | 17911 | 80 |
| | | 8 | 7512 | 7557 | 125 |
| | | 9 | 8148 | 9056 | 147 |
| | | 10 | 8461 | 8726 | -176 |
| | | 11 | 1799 | 2089 | -85 |
| | | 12 | 6814 | 6602 | -166 |
| | | 13 | 5701 | 5989 | 50 |
| | | 14 | 1381 | 1302 | -155 |
| | | 15 | 6848 | 7697 | 101 |
| 1 | 3 | 1 | 8257 | 7527 | 174 |
| | | 2 | 5685 | 5391 | 118 |
| | | 3 | 3443 | 2965 | 178 |
| | | 4 | 12416 | 12105 | -174 |
| | | 5 | 8204 | 7412 | -57 |
| | | 6 | 6074 | 5596 | -20 |
| | | 7 | 4165 | 3603 | 87 |
| | | 8 | 5583 | 5693 | -165 |
| | | 9 | 2935 | 2167 | 99 |
| | | 10 | 5172 | 2591 | 107 |
| | | 15 | 2590 | 2799 | 95 |
| 1 | 4 | 1 | 23552 | 24547 | -52 |
| | | 2 | 17156 | 16654 | -11 |
| | | 3 | 4060 | 3521 | -5 |
| | | 4 | 19402 | 21152 | -4 |
| | | 5 | 10746 | 10825 | -170 |
| | | 6 | 7651 | 6954 | 28 |
| | | 7 | 15549 | 16891 | -87 |
| | | 8 | 7915 | 7557 | 16 |
| | | 9 | 12350 | 12613 | -41 |
| | | 10 | 8657 | 8400 | -19 |
| | | 11 | 5468 | 5642 | 108 |
| | | 12 | 5650 | 5065 | 27 |
| | | 13 | 5629 | 6135 | -129 |
| | | 14 | 2601 | 2571 | 45 |
| | | 15 | 6928 | 8258 | -73 |
| 1 | 5 | 0 | 15654 | 14427 | 0 |
| | | 1 | 9622 | 8559 | -168 |
| | | 2 | 11240 | 10648 | 3 |
| | | 3 | 14996 | 15241 | -139 |
| | | 4 | 9526 | 8512 | 63 |
| | | 5 | 15176 | 15323 | -36 |
| | | 6 | 12527 | 13339 | -24 |
| | | 7 | 3042 | 2855 | -107 |
| | | 8 | 9871 | 8859 | 1 |
| | | 9 | 8578 | 7536 | -138 |
| | | 10 | 5485 | 5587 | 31 |
| | | 11 | 8271 | 7847 | -89 |
| | | 12 | 5044 | 4930 | -15 |
| | | 13 | 4815 | 5195 | -44 |
| | | 14 | 3892 | 4207 | -20 |
| 1 | 6 | 1 | 2393 | 1496 | 12 |
| | | 2 | 6164 | 5292 | 18 |
| | | 3 | 10985 | 10399 | -115 |
| | | 4 | 3541 | 3028 | -151 |
| | | 5 | 8623 | 8182 | -12 |
| | | 6 | 2110 | 1833 | 19 |
| | | 7 | 4805 | 4152 | 95 |
| | | 8 | 5436 | 5259 | 47 |
| | | 9 | 5420 | 4452 | -141 |
| | | 11 | 4067 | 3831 | -92 |
| | | 13 | 3892 | 4484 | 3 |
| | | 14 | 1540 | 1791 | -7 |
| 1 | 7 | 0 | 8994 | 7125 | -180 |
| | | 1 | 17090 | 15775 | -14 |
| | | 2 | 14637 | 13700 | -127 |
| | | 3 | 16465 | 16000 | 70 |
| | | 4 | 7076 | 6198 | 178 |
| | | 5 | 13849 | 12491 | 136 |
| | | 6 | 10337 | 9664 | 155 |
| 1 | 7 | 7 | 7294 | 6325 | -151 |
| | | 8 | 10678 | 10271 | -160 |
| | | 9 | 10352 | 10094 | 24 |
| | | 10 | 5811 | 4941 | -110 |
| | | 11 | 9610 | 9601 | 93 |
| | | 12 | 4502 | 4752 | 133 |
| | | 13 | 5726 | 5855 | 165 |
| | | 14 | 4342 | 4596 | 179 |
| 1 | 8 | 0 | 14476 | 13077 | 180 |
| | | 1 | 13626 | 11990 | 98 |
| | | 2 | 4749 | 3759 | 177 |
| | | 3 | 6759 | 6217 | 134 |
| | | 4 | 15172 | 14700 | -179 |
| | | 5 | 5726 | 5039 | 26 |
| | | 6 | 5634 | 4561 | -164 |
| | | 7 | 11886 | 11213 | 102 |
| | | 8 | 6982 | 6707 | 178 |
| | | 9 | 6555 | 5756 | 108 |
| | | 10 | 5323 | 4222 | -161 |
| | | 11 | 2040 | 1956 | 102 |
| | | 12 | 6581 | 7202 | -170 |
| | | 13 | 2794 | 2796 | 78 |
| | | 14 | 3032 | 2775 | -157 |
| 1 | 9 | 0 | 8580 | 7160 | -180 |
| | | 1 | 13010 | 11857 | 122 |
| | | 2 | 4922 | 4112 | 88 |
| | | 3 | 7570 | 6852 | -136 |
| | | 4 | 4401 | 3750 | 169 |
| | | 5 | 7072 | 6047 | -25 |
| | | 6 | 3915 | 3149 | -118 |
| | | 7 | 9880 | 8972 | 80 |
| | | 8 | 4934 | 4135 | 63 |
| | | 9 | 6294 | 5169 | -180 |
| | | 10 | 2906 | 2090 | 158 |
| | | 11 | 2476 | 2302 | -99 |
| | | 12 | 3181 | 3233 | -156 |
| | | 13 | 2689 | 2778 | 9 |
| 1 | 10 | 0 | 5095 | 3728 | 0 |
| | | 1 | 16539 | 15052 | -58 |
| | | 2 | 10741 | 9758 | -40 |
| | | 3 | 5900 | 4856 | 65 |
| | | 4 | 10684 | 9238 | 25 |
| | | 5 | 7072 | 5647 | -143 |
| | | 6 | 5146 | 4586 | 7 |
| | | 7 | 12799 | 12098 | -108 |
| | | 8 | 2845 | 1911 | -91 |
| | | 9 | 9705 | 9458 | -26 |
| | | 10 | 6303 | 5840 | -10 |
| | | 11 | 3631 | 3614 | 100 |
| | | 12 | 5634 | 6191 | 35 |
| | | 13 | 5701 | 5860 | -154 |
| 1 | 11 | 0 | 7146 | 6019 | 0 |
| | | 1 | 10901 | 9495 | -68 |
| | | 2 | 13520 | 11745 | 9 |
| | | 3 | 11396 | 10456 | -94 |
| | | 4 | 8165 | 7174 | -26 |
| | | 5 | 7516 | 6417 | -74 |
| | | 6 | 9242 | 8491 | 1 |
| | | 7 | 3585 | 2779 | -79 |
| | | 8 | 5670 | 4715 | 23 |
| | | 9 | 8444 | 7616 | -85 |
| | | 10 | 5583 | 5517 | -2 |
| | | 11 | 3323 | 2847 | -98 |
| | | 12 | 2815 | 3130 | 28 |
| | | 13 | 2731 | 3926 | -46 |
| 1 | 12 | 0 | 7206 | 5922 | 0 |
| | | 1 | 7254 | 6319 | 162 |
| | | 2 | 4002 | 3388 | 79 |
| | | 3 | 8232 | 7447 | -151 |
| | | 4 | 6353 | 5720 | 150 |
| | | 5 | 9265 | 8608 | -30 |
| | | 6 | 8627 | 7857 | -23 |
| | | 7 | 5372 | 4568 | 83 |
| | | 8 | 3288 | 2296 | 16 |
| | | 9 | 6555 | 5986 | -166 |
| | | 10 | 2055 | 1799 | 88 |
| | | 11 | 4839 | 5198 | -84 |
| | | 12 | 798 | 1402 | -35 |
| 1 | 13 | 0 | 14733 | 12742 | 180 |
| | | 1 | 10630 | 9240 | -6 |
| | | 2 | 2522 | 1550 | -144 |
| | | 3 | 11572 | 11090 | 89 |
| | | 4 | 2137 | 1551 | 160 |
| | | 5 | 8723 | 7807 | 125 |
| | | 6 | 5118 | 4049 | 172 |
| | | 7 | 4024 | 3486 | -127 |
| | | 8 | 6577 | 5748 | 167 |
| | | 9 | 5483 | 5278 | 16 |
| | | 10 | 3476 | 3408 | -108 |
| | | 11 | 5978 | 7260 | 109 |
| 1 | 14 | 0 | 6376 | 5738 | 180 |
| | | 1 | 5546 | 4912 | 66 |
| | | 2 | 6861 | 5670 | -171 |
| | | 3 | 8582 | 7814 | 110 |
| | | 4 | 7811 | 6465 | -169 |
| | | 5 | 4613 | 3741 | 76 |
| | | 6 | 5605 | 5362 | -161 |
| | | 7 | 4322 | 3432 | 116 |
| | | 8 | 7019 | 6660 | -171 |
| | | 9 | 4607 | 4699 | 71 |
| | | 10 | 4946 | 4940 | -173 |
| | | 11 | 4348 | 4598 | 106 |
| 1 | 15 | 1 | 7266 | 6987 | 124 |
| | | 2 | 4067 | 3617 | 116 |
| | | 3 | 7555 | 6938 | -153 |
| | | 4 | 6585 | 6063 | -166 |
| | | 5 | 7773 | 7343 | -18 |
| | | 6 | 3323 | 2566 | -90 |
| | | 7 | 7505 | 7355 | 89 |
| | | 8 | 3534 | 3056 | 111 |
| | | 9 | 4172 | 4080 | 155 |
| 1 | 16 | 0 | 2906 | 1435 | -180 |
| | | 1 | 8264 | 7299 | -46 |
| | | 2 | 6694 | 5190 | -24 |
| | | 3 | 2886 | 2477 | 8 |
| | | 4 | 4665 | 4001 | 29 |
| | | 5 | 4434 | 5888 | -162 |
| | | 6 | 4460 | 3706 | 43 |
| | | 7 | 4975 | 4783 | -114 |
| | | 8 | 2026 | 1952 | -4 |
| | | 9 | 4779 | 5205 | -41 |
| 1 | 17 | 0 | 5655 | 4454 | 0 |
| | | 1 | 6475 | 6109 | -66 |
| | | 2 | 7341 | 7518 | -23 |
| | | 3 | 2082 | 1820 | -54 |
| | | 4 | 5515 | 5012 | 25 |
| | | 5 | 4060 | 3441 | -104 |
| | | 6 | 4827 | 4581 | 7 |
| | | 7 | 4869 | 4765 | -111 |
| | | 8 | 4052 | 3709 | -17 |
| 1 | 18 | 0 | 3297 | 3539 | 0 |
| | | 1 | 5190 | 5275 | 145 |
| | | 2 | 4095 | 3821 | 22 |
| | | 3 | 7856 | 8121 | -115 |
| | | 4 | 2177 | 1617 | -125 |
| | | 5 | 5816 | 6156 | -34 |
| | | 6 | 3639 | 3625 | -38 |
| | | 7 | 3623 | 3991 | 69 |
| 1 | 19 | 0 | 2984 | 2518 | 180 |
| | | 1 | 1359 | 1270 | -10 |
| | | 2 | 3418 | 3619 | -154 |
| | | 3 | 4234 | 4425 | 87 |
| | | 4 | 3694 | 2957 | 159 |
| | | 5 | 2545 | 2078 | 154 |
| 1 | 20 | 0 | 8048 | 7428 | 180 |
| | | 1 | 4151 | 3515 | 25 |
| | | 2 | 3070 | 3074 | -179 |
| | | 3 | 4575 | 5903 | 93 |
| 2 | 0 | 1 | 8914 | 7144 | 57 |
| | | 2 | 14678 | 16974 | 93 |
| | | 3 | 26173 | 30739 | 87 |
| | | 4 | 9421 | 9904 | 165 |
| | | 5 | 15017 | 20029 | 105 |
| | | 6 | 5811 | 5862 | -89 |
| | | 7 | 7341 | 9200 | 127 |
| | | 8 | 10504 | 11017 | 55 |
| 2 | 8 | 0 | 6607 | 4805 | 180 |
| | | 1 | 6572 | 5368 | -160 |
| | | 2 | 14751 | 12315 | -74 |
| | | 3 | 14392 | 13221 | -128 |
| | | 4 | 12525 | 11507 | 39 |
| | | 5 | 15554 | 14619 | -43 |
| | | 6 | 8087 | 6591 | 81 |
| | | 7 | 4388 | 3789 | 93 |
| | | 8 | 6112 | 5568 | -122 |
| | | 9 | 4675 | 3927 | -151 |
| | | 10 | 3951 | 2962 | -43 |
| | | 11 | 5880 | 5528 | -86 |
| | | 12 | 4220 | 4151 | 38 |
| | | 13 | 4615 | 4876 | -16 |
| | | 14 | 2886 | 3267 | 165 |
| 2 | 9 | 0 | 8246 | 6197 | 0 |
| | | 1 | 18820 | 16765 | -61 |
| | | 2 | 9470 | 7415 | 75 |
| | | 3 | 7435 | 6142 | -70 |
| | | 4 | 5361 | 4226 | -75 |
| | | 5 | 8875 | 7876 | -151 |
| | | 6 | 7250 | 6470 | -79 |
| | | 7 | 12840 | 11851 | -91 |
| | | 8 | 6603 | 6186 | 40 |
| | | 9 | 11116 | 10546 | -66 |
| | | 10 | 3025 | 2208 | 120 |
| | | 11 | 2906 | 2270 | -106 |
| | | 12 | 2794 | 2385 | -131 |
| | | 13 | 4460 | 5755 | -103 |
| 2 | 10 | 0 | 5339 | 4182 | 0 |
| | | 1 | 10205 | 8511 | -80 |
| | | 2 | 5895 | 4483 | 86 |
| | | 3 | 5983 | 4782 | -86 |
| | | 4 | 2855 | 1496 | -152 |
| | | 5 | 7206 | 6380 | -86 |
| | | 6 | 4557 | 3815 | -61 |
| | | 7 | 8720 | 7471 | -84 |
| | | 8 | 2204 | 1056 | -96 |
| | | 9 | 7975 | 7568 | -67 |
| | | 10 | 3810 | 2853 | 68 |
| | | 11 | 3061 | 2290 | -87 |
| | | 12 | 1295 | 1568 | 1 |
| | | 13 | 4045 | 4457 | -99 |
| 2 | 11 | 0 | 11204 | 10027 | -180 |
| | | 1 | 11444 | 9969 | 125 |
| | | 2 | 6457 | 4843 | -62 |
| | | 3 | 8852 | 7245 | -143 |
| | | 4 | 10019 | 8155 | 16 |
| | | 5 | 8570 | 7624 | 7 |
| | | 6 | 6599 | 5990 | 127 |
| | | 7 | 9853 | 8501 | 85 |
| | | 8 | 6349 | 5723 | -157 |
| | | 9 | 5801 | 5329 | 173 |
| | | 10 | 4328 | 4356 | -36 |
| | | 11 | 2579 | 1809 | -104 |
| 2 | 12 | 0 | 2612 | 422 | 0 |
| | | 1 | 8101 | 6775 | 53 |
| | | 2 | 3973 | 2960 | 90 |
| | | 3 | 13120 | 11650 | 97 |
| | | 4 | 4268 | 3445 | 178 |
| | | 5 | 11121 | 9649 | 92 |
| | | 6 | 3443 | 2882 | -69 |
| | | 7 | 6407 | 5642 | 134 |
| | | 8 | 3409 | 2804 | 54 |
| | | 9 | 7588 | 6852 | 68 |
| | | 10 | 3409 | 3058 | 150 |
| 2 | 4 | 13 | 4248 | 4755 | -65 |
| 2 | 5 | 0 | 8423 | 7047 | -180 |
| | | 1 | 11409 | 11680 | 82 |
| | | 2 | 11825 | 10504 | -39 |
| | | 3 | 11447 | 11423 | 172 |
| | | 4 | 4505 | 4464 | -55 |
| | | 5 | 12243 | 11675 | 4 |
| | | 6 | 6638 | 5962 | 122 |
| | | 7 | 12174 | 12607 | 107 |
| | | 8 | 7392 | 7122 | -141 |
| | | 9 | 5152 | 4037 | 151 |
| | | 10 | 6178 | 5732 | -47 |
| | | 11 | 3155 | 2588 | 170 |
| | | 12 | 3163 | 2710 | 70 |
| | | 13 | 5350 | 5419 | 21 |
| | | 14 | 3501 | 4191 | 144 |
| 2 | 6 | 0 | 21039 | 20290 | 0 |
| | | 1 | 15174 | 13229 | 54 |
| | | 2 | 11924 | 9808 | 145 |
| | | 3 | 21890 | 23189 | 85 |
| | | 4 | 13054 | 15211 | -165 |
| | | 5 | 14434 | 13604 | 112 |
| | | 6 | 7506 | 6597 | -76 |
| | | 7 | 6178 | 5297 | 153 |
| | | 8 | 4024 | 2981 | 16 |
| | | 9 | 11101 | 10668 | 52 |
| | | 10 | 6642 | 6099 | 152 |
| | | 11 | 10890 | 11955 | 94 |
| | | 12 | 3583 | 2889 | -128 |
| | | 13 | 5796 | 6478 | 152 |
| | | 14 | 1954 | 2349 | -48 |
| 2 | 7 | 0 | 3647 | 2236 | 180 |
| | | 1 | 11525 | 10409 | 62 |
| | | 2 | 4440 | 3388 | 178 |
| | | 3 | 11261 | 10665 | 95 |
| | | 4 | 9564 | 8527 | 90 |
| | | 5 | 3591 | 2118 | 150 |
| | | 6 | 8994 | 8867 | 111 |
| | | 7 | 5056 | 4134 | 58 |
| | | 8 | 5197 | 4246 | 80 |
| | | 9 | 2429 | 2436 | -114 |
| | | 10 | 5268 | 5238 | 102 |
| | | 11 | 2124 | 2248 | -170 |
| | | 12 | 3375 | 3031 | 105 |
| 2 | 8 | 0 | 6607 | 4805 | 180 |
| | | 1 | 6572 | 5368 | -160 |
| | | 2 | 14751 | 12315 | -74 |
| | | 3 | 14392 | 13221 | -128 |
| | | 4 | 12525 | 11507 | 39 |
| | | 5 | 15554 | 14619 | -43 |
| | | 6 | 8087 | 6591 | 81 |
| | | 7 | 4388 | 3789 | 93 |
| | | 8 | 6112 | 5568 | -122 |
| | | 9 | 4675 | 3927 | -151 |
| | | 10 | 3951 | 2962 | -43 |
| | | 11 | 5880 | 5528 | -86 |
| | | 12 | 4220 | 4151 | 38 |
| | | 13 | 4615 | 4876 | -16 |
| | | 14 | 2886 | 3267 | 165 |
| 2 | 9 | 0 | 8246 | 6197 | 0 |
| | | 1 | 18820 | 16765 | -61 |
| | | 2 | 9470 | 7415 | 75 |
| | | 3 | 7435 | 6142 | -70 |
| | | 4 | 5361 | 4226 | -75 |
| | | 5 | 8875 | 7876 | -151 |
| | | 6 | 7250 | 6470 | -79 |
| | | 7 | 12840 | 11851 | -91 |
| | | 8 | 6603 | 6186 | 40 |
| | | 9 | 11116 | 10546 | -66 |
| | | 10 | 3025 | 2208 | 120 |
| | | 11 | 2906 | 2270 | -106 |
| | | 12 | 2794 | 2385 | -131 |
| | | 13 | 4460 | 5755 | -103 |
| 2 | 10 | 0 | 5339 | 4182 | 0 |
| | | 1 | 10205 | 8511 | -80 |
| | | 2 | 5895 | 4483 | 86 |
| | | 3 | 5983 | 4782 | -86 |
| | | 4 | 2855 | 1496 | -152 |
| | | 5 | 7206 | 6380 | -86 |
| | | 6 | 4557 | 3815 | -61 |
| | | 7 | 8720 | 7471 | -84 |
| | | 8 | 2204 | 1056 | -96 |
| | | 9 | 7975 | 7568 | -67 |
| | | 10 | 3810 | 2853 | 68 |
| | | 11 | 3061 | 2290 | -87 |
| | | 12 | 1295 | 1568 | 1 |
| | | 13 | 4045 | 4457 | -99 |
| 2 | 11 | 0 | 11204 | 10027 | -180 |
| | | 1 | 11444 | 9969 | 125 |
| | | 2 | 6457 | 4843 | -62 |
| | | 3 | 8852 | 7245 | -143 |
| | | 4 | 10019 | 8155 | 16 |
| | | 5 | 8570 | 7624 | 7 |
| | | 6 | 6599 | 5990 | 127 |
| | | 7 | 9853 | 8501 | 85 |
| | | 8 | 6349 | 5723 | -157 |
| | | 9 | 5801 | 5329 | 173 |
| | | 10 | 4328 | 4356 | -36 |
| | | 11 | 2579 | 1809 | -104 |
| 2 | 12 | 0 | 2612 | 422 | 0 |
| | | 1 | 8101 | 6775 | 53 |
| | | 2 | 3973 | 2960 | 90 |
| | | 3 | 13120 | 11650 | 97 |
| | | 4 | 4268 | 3445 | 178 |
| | | 5 | 11121 | 9649 | 92 |
| | | 6 | 3443 | 2882 | -69 |
| | | 7 | 6407 | 5642 | 134 |
| | | 8 | 3409 | 2804 | 54 |
| | | 9 | 7588 | 6852 | 68 |
| | | 10 | 3409 | 3058 | 150 |

Table 1 (*cont.*)

| h k l | $F_o$ | $F_c$ | $\alpha_{hkl}$ |
|---|---|---|---|
| 2 12 11 | 7194 | 7889 | 97 |
| 12 | 3349 | 3415 | -157 |
| 2 13 0 | 5112 | 5719 | 0 |
| 1 | 6856 | 4898 | 79 |
| 2 | 3575 | 2199 | 158 |
| 3 | 11155 | 10864 | 72 |
| 4 | 6875 | 6064 | -166 |
| 5 | 8559 | 7249 | 125 |
| 6 | 2612 | 1924 | -33 |
| 7 | 4492 | 3288 | 109 |
| 8 | 2294 | 1215 | 38 |
| 9 | 5598 | 5152 | 70 |
| 10 | 3988 | 3866 | 145 |
| 11 | 5665 | 5788 | 85 |
| 2 14 0 | 5518 | 4569 | 180 |
| 1 | 5484 | 5072 | 129 |
| 2 | 6844 | 6246 | -83 |
| 3 | 7566 | 6100 | -99 |
| 4 | 5983 | 4775 | -12 |
| 5 | 5766 | 4472 | -47 |
| 6 | 6603 | 5661 | 117 |
| 7 | 4845 | 4657 | 70 |
| 8 | 3493 | 3561 | -148 |
| 9 | 3510 | 3896 | -172 |
| 10 | 4505 | 5355 | -54 |
| 11 | 3951 | 5491 | -85 |
| 2 15 0 | 7943 | 6654 | 0 |
| 1 | 11233 | 9342 | -70 |
| 3 | 5619 | 5039 | -97 |
| 4 | 2751 | 2273 | 133 |
| 5 | 7870 | 6970 | -89 |
| 6 | 4158 | 3198 | -25 |
| 7 | 8580 | 8071 | -94 |
| 8 | 2965 | 2524 | -3 |
| 9 | 4421 | 4686 | -50 |
| 2 16 0 | 4556 | 3600 | 0 |
| 1 | 9664 | 9194 | -71 |
| 2 | 6257 | 5486 | 82 |
| 3 | 4613 | 3612 | -43 |
| 4 | 1985 | 1426 | -133 |
| 5 | 5691 | 5480 | -147 |
| 6 | 2945 | 2586 | -82 |
| 7 | 7493 | 7735 | -82 |
| 8 | 5263 | 5779 | 33 |
| 9 | 4963 | 5056 | -67 |
| 2 17 0 | 4017 | 3704 | -180 |
| 1 | 6475 | 5780 | 144 |
| 2 | 4899 | 4647 | -88 |
| 3 | 3663 | 3443 | -123 |
| 4 | 3937 | 3748 | 25 |
| 5 | 4186 | 3848 | -11 |
| 6 | 2623 | 2485 | 119 |
| 7 | 3181 | 3578 | 67 |
| 8 | 4499 | 4540 | -157 |
| 2 18 0 | 2405 | 2164 | -180 |
| 1 | 6434 | 6459 | 91 |
| 2 | 1102 | 778 | 127 |
| 3 | 4887 | 4828 | 78 |
| 4 | 2464 | 2158 | -99 |
| 5 | 5441 | 5867 | 105 |
| 2 19 0 | 2217 | 1934 | 0 |
| 1 | 2804 | 2796 | 49 |
| 2 | 3208 | 3793 | 115 |
| 3 | 5781 | 7492 | 74 |
| 4 | 3332 | 3285 | -175 |
| 5 | 6248 | 6005 | 119 |
| 2 20 0 | 3199 | 2651 | -180 |
| 1 | 3900 | 3424 | 160 |
| 3 1 0 | 14599 | 16222 | -180 |
| 1 | 12826 | 13568 | -67 |
| 2 | 10864 | 10527 | -119 |
| 3 | 14269 | 14789 | -58 |
| 4 | 7003 | 6704 | -10 |
| 5 | 9903 | 9525 | -148 |
| 6 | 15318 | 16703 | 142 |
| 7 | 9592 | 8876 | -100 |
| 8 | 12315 | 12262 | -167 |
| 9 | 9732 | 9497 | -77 |
| 10 | 7125 | 6750 | -70 |
| 11 | 4095 | 3683 | -73 |
| 12 | 5781 | 6128 | 92 |
| 13 | 4322 | 4653 | -106 |
| 14 | 5690 | 6871 | 159 |
| 3 2 0 | 9160 | 7954 | 0 |
| 1 | 11932 | 12113 | -143 |
| 2 | 10309 | 9845 | 121 |
| 3 | 13841 | 14115 | -83 |
| 4 | 18579 | 20100 | -172 |
| 5 | 14667 | 15257 | -74 |
| 6 | 8753 | 7769 | -73 |
| 7 | 4694 | 3846 | -128 |
| 8 | 5409 | 2595 | 80 |
| 9 | 7329 | 6840 | -103 |
| 10 | 8583 | 8634 | 155 |
| 11 | 8001 | 8226 | -72 |
| 12 | 7258 | 7394 | -143 |
| 13 | 5078 | 5200 | -74 |
| 14 | 2096 | 2060 | -75 |
| 3 3 0 | 10273 | 8655 | -180 |
| 1 | 2825 | 2346 | 50 |
| 2 | 4632 | 4101 | 68 |
| 3 | 13291 | 12426 | -64 |
| 4 | 6126 | 5562 | -159 |
| 5 | 2825 | 2187 | -166 |
| 6 | 4234 | 3611 | -110 |
| 7 | 4466 | 5031 | 19 |
| 8 | 4731 | 5066 | 35 |
| 9 | 3126 | 2987 | -114 |
| 10 | 2429 | 2072 | -105 |
| 11 | 2405 | 2078 | -38 |
| 12 | 4095 | 4044 | -141 |
| 13 | 1295 | 1144 | 29 |
| 14 | 1666 | 1918 | -6 |
| 3 4 0 | 4531 | 3773 | -180 |
| 1 | 11577 | 10323 | 96 |
| 2 | 18211 | 19081 | -71 |
| 3 | 16655 | 17965 | 98 |
| 4 | 17612 | 18458 | 51 |
| 5 | 7744 | 6362 | 90 |
| 6 | 10885 | 10874 | 76 |
| 7 | 4981 | 4894 | 156 |
| 3 4 8 | 7158 | 6956 | -127 |
| 9 | 11129 | 11147 | 69 |
| 10 | 8643 | 8690 | -16 |
| 11 | 6852 | 6594 | 95 |
| 12 | 9391 | 9743 | 36 |
| 13 | 4234 | 5078 | 143 |
| 14 | 2344 | 2872 | 157 |
| 3 5 0 | 14089 | 12556 | 0 |
| 1 | 13358 | 12412 | 95 |
| 2 | 13910 | 13485 | 44 |
| 3 | 5726 | 4943 | 96 |
| 4 | 6928 | 6124 | -87 |
| 5 | 12493 | 11670 | 103 |
| 6 | 9387 | 9349 | -40 |
| 7 | 10460 | 10510 | 82 |
| 8 | 8663 | 8009 | 39 |
| 9 | 7226 | 6998 | 127 |
| 10 | 3671 | 3432 | 64 |
| 11 | 4839 | 4805 | 112 |
| 12 | 5241 | 5929 | 77 |
| 13 | 3615 | 5155 | -13 |
| 3 6 0 | 11046 | 9461 | 0 |
| 1 | 8566 | 7172 | -177 |
| 2 | 5781 | 4806 | 60 |
| 3 | 4328 | 4078 | 104 |
| 4 | 6563 | 6614 | 168 |
| 5 | 4441 | 3509 | 51 |
| 6 | 9797 | 9704 | -41 |
| 8 | 4088 | 2860 | 35 |
| 9 | 5425 | 5574 | 143 |
| 10 | 3271 | 2495 | 126 |
| 11 | 2452 | 2019 | -33 |
| 12 | 2344 | 2056 | -130 |
| 14 | 2556 | 3001 | -39 |
| 3 7 0 | 23967 | 23614 | -180 |
| 1 | 16514 | 16228 | -70 |
| 2 | 11966 | 10916 | -110 |
| 3 | 5634 | 4751 | -50 |
| 4 | 7419 | 6345 | 59 |
| 5 | 5246 | 3887 | -106 |
| 6 | 11492 | 11219 | 136 |
| 7 | 12016 | 11675 | -81 |
| 8 | 10068 | 9592 | -152 |
| 9 | 8420 | 7930 | -61 |
| 10 | 7341 | 7266 | -72 |
| 11 | 2994 | 2431 | -100 |
| 12 | 3959 | 4475 | 72 |
| 13 | 2699 | 3414 | -76 |
| 14 | 3501 | 5683 | 164 |
| 3 8 0 | 5377 | 4552 | -180 |
| 1 | 9958 | 8312 | -97 |
| 2 | 8433 | 7031 | 125 |
| 3 | 12365 | 11954 | -86 |
| 4 | 10482 | 9723 | -164 |
| 5 | 8437 | 7990 | -99 |
| 6 | 5836 | 5014 | -123 |
| 7 | 8522 | 7815 | -68 |
| 8 | 5478 | 4602 | 129 |
| 9 | 6546 | 5830 | -93 |
| 10 | 5510 | 4690 | 176 |
| 11 | 6475 | 6972 | -90 |
| 12 | 3863 | 4413 | -169 |
| 13 | 2804 | 3453 | -72 |
| 3 9 0 | 8822 | 7373 | 0 |
| 1 | 6398 | 5045 | 167 |
| 2 | 7451 | 6742 | 75 |
| 3 | 5462 | 4776 | -102 |
| 4 | 9900 | 8845 | -157 |
| 5 | 6261 | 5268 | -106 |
| 6 | 10002 | 8953 | -55 |
| 8 | 5772 | 3558 | 103 |
| 9 | 2945 | 1840 | 140 |
| 10 | 5061 | 4521 | 135 |
| 11 | 3702 | 3477 | -86 |
| 12 | 3542 | 3961 | -152 |
| 13 | 1360 | 1210 | -138 |
| 3 10 0 | 14200 | 12113 | -180 |
| 1 | 7109 | 5080 | 72 |
| 2 | 13335 | 10952 | -45 |
| 3 | 9794 | 9286 | 44 |
| 4 | 12846 | 11719 | 12 |
| 5 | 12228 | 11108 | 143 |
| 6 | 7993 | 7028 | 97 |
| 7 | 5393 | 4798 | 56 |
| 8 | 4151 | 3440 | -131 |
| 9 | 3779 | 3027 | 72 |
| 10 | 9004 | 9189 | -35 |
| 11 | 5146 | 5629 | 94 |
| 12 | 4499 | 5609 | 46 |
| 3 11 0 | 7380 | 5856 | 0 |
| 1 | 10145 | 8571 | 120 |
| 2 | 5685 | 4577 | -31 |
| 3 | 11793 | 10680 | 86 |
| 4 | 6206 | 5421 | 90 |
| 5 | 8243 | 7271 | 67 |
| 6 | 7822 | 7004 | -6 |
| 7 | 5141 | 4366 | 102 |
| 8 | 5224 | 4720 | 2 |
| 9 | 8218 | 8181 | 102 |
| 10 | 2742 | 3078 | 21 |
| 11 | 3315 | 3437 | 64 |
| 12 | 3070 | 3049 | -1 |
| 3 12 0 | 8365 | 7385 | 0 |
| 1 | 4749 | 3740 | 109 |
| 2 | 9191 | 7923 | 57 |
| 3 | 3172 | 2312 | -101 |
| 4 | 9688 | 8495 | -154 |
| 5 | 5191 | 4221 | 62 |
| 6 | 8119 | 7540 | -54 |
| 7 | 4575 | 3720 | 86 |
| 8 | 6759 | 6074 | 44 |
| 9 | 3409 | 3291 | 177 |
| 10 | 2710 | 2448 | 131 |
| 3 13 0 | 8730 | 7136 | -180 |
| 1 | 7431 | 6667 | -117 |
| 2 | 9270 | 7786 | -90 |
| 3 | 4479 | 3416 | -47 |
| 4 | 3280 | 2679 | 40 |
| 5 | 5118 | 4585 | -126 |
| 6 | 5711 | 5417 | 119 |
| 7 | 7665 | 6724 | -99 |
| 3 13 8 | 8700 | 8759 | -161 |
| 9 | 2522 | 1851 | -78 |
| 10 | 3639 | 3780 | -66 |
| 11 | 2319 | 2358 | -47 |
| 3 14 0 | 9394 | 8465 | -180 |
| 1 | 7085 | 5895 | -76 |
| 2 | 4492 | 4027 | 180 |
| 3 | 10312 | 9524 | -87 |
| 4 | 4220 | 3148 | 171 |
| 5 | 6502 | 5477 | -96 |
| 6 | 4335 | 3749 | 177 |
| 7 | 6577 | 6065 | -78 |
| 8 | 3787 | 3526 | 159 |
| 9 | 5050 | 4765 | -74 |
| 10 | 2763 | 2654 | -115 |
| 3 15 0 | 2499 | 1467 | 0 |
| 1 | 3476 | 2910 | -177 |
| 2 | 6844 | 6494 | 93 |
| 3 | 5572 | 4898 | -83 |
| 4 | 8894 | 8580 | -171 |
| 5 | 3802 | 3589 | -74 |
| 6 | 6102 | 6182 | -69 |
| 7 | 1540 | 2238 | 2 |
| 8 | 4342 | 4549 | 48 |
| 9 | 2151 | 1816 | -136 |
| 3 16 0 | 2835 | 2292 | -180 |
| 1 | 3013 | 1989 | 80 |
| 2 | 7672 | 7053 | -47 |
| 3 | 5741 | 5513 | 72 |
| 4 | 5736 | 5247 | 18 |
| 5 | 5010 | 4865 | 123 |
| 6 | 2916 | 2328 | 66 |
| 7 | 1521 | 980 | 97 |
| 8 | 3315 | 3205 | -142 |
| 3 17 1 | 4575 | 4459 | 75 |
| 2 | 5274 | 4538 | -16 |
| 3 | 5711 | 5781 | 83 |
| 4 | 5027 | 5109 | 6 |
| 5 | 7019 | 7113 | 111 |
| 6 | 2499 | 2554 | 41 |
| 7 | 4144 | 3607 | 94 |
| 3 18 0 | 5721 | 6199 | 0 |
| 1 | 5339 | 5097 | 154 |
| 2 | 4441 | 4772 | 86 |
| 3 | 2417 | 2390 | 99 |
| 4 | 4607 | 5263 | 179 |
| 5 | 1831 | 1939 | 20 |
| 6 | 6564 | 6855 | -36 |
| 3 19 0 | 6694 | 5693 | -180 |
| 1 | 3567 | 3095 | -74 |
| 2 | 2876 | 2370 | -90 |
| 3 | 3023 | 2677 | -75 |
| 4 0 0 | 18001 | 17437 | -180 |
| 1 | 14218 | 16000 | -63 |
| 2 | 5552 | 4558 | -154 |
| 3 | 13427 | 13136 | 49 |
| 4 | 15128 | 15509 | -173 |
| 5 | 10332 | 9622 | -153 |
| 6 | 10451 | 9955 | -163 |
| 7 | 16005 | 16641 | -100 |
| 8 | 13156 | 13295 | -180 |
| 9 | 10826 | 11034 | 9 |
| 10 | 4718 | 4664 | -141 |
| 11 | 4755 | 4260 | 61 |
| 12 | 7721 | 8710 | 167 |
| 13 | 6969 | 8809 | -150 |
| 14 | 3341 | 4224 | -156 |
| 4 1 0 | 6002 | 6404 | -180 |
| 1 | 5639 | 5555 | 52 |
| 2 | 3332 | 2180 | -156 |
| 3 | 9791 | 8994 | -102 |
| 4 | 9555 | 8672 | 159 |
| 5 | 4575 | 3344 | -57 |
| 6 | 5531 | 4453 | -162 |
| 7 | 5866 | 5568 | 74 |
| 8 | 4657 | 4120 | 162 |
| 9 | 4613 | 3923 | -125 |
| 10 | 3217 | 2955 | -139 |
| 11 | 3980 | 3596 | -116 |
| 12 | 4441 | 4208 | 164 |
| 13 | 3840 | 4487 | -9 |
| 14 | 1893 | 2417 | -152 |
| 4 2 0 | 2601 | 798 | 180 |
| 1 | 11898 | 12576 | 140 |
| 2 | 7222 | 7014 | -30 |
| 3 | 5701 | 5231 | 176 |
| 4 | 4928 | 3301 | 61 |
| 5 | 9052 | 7915 | -18 |
| 6 | 7924 | 8041 | -7 |
| 7 | 8427 | 7703 | 91 |
| 8 | 6206 | 5644 | 18 |
| 9 | 8101 | 7556 | 151 |
| 10 | 3392 | 3420 | -41 |
| 11 | 4109 | 3853 | -70 |
| 12 | 3023 | 2514 | 5 |
| 13 | 2896 | 2575 | 20 |
| 14 | 2191 | 2878 | -19 |
| 4 3 0 | 9967 | 9577 | 0 |
| 1 | 11970 | 12307 | -19 |
| 2 | 14192 | 20266 | -5 |
| 3 | 15311 | 15500 | 66 |
| 4 | 10301 | 9654 | -2 |
| 5 | 15341 | 16081 | 136 |
| 6 | 16129 | 16358 | -2 |
| 7 | 6848 | 5878 | -103 |
| 8 | 5934 | 4962 | 23 |
| 9 | 8358 | 6820 | 9 |
| 10 | 8461 | 8362 | -2 |
| 11 | 9319 | 9572 | 97 |
| 12 | 5552 | 5674 | 17 |
| 13 | 3631 | 5198 | 167 |
| 14 | 4348 | 4852 | 2 |
| 4 4 0 | 6924 | 5454 | 0 |
| 1 | 5252 | 4343 | 107 |
| 2 | 14490 | 13755 | 15 |
| 3 | 3460 | 3004 | -173 |
| 4 | 5318 | 4778 | -19 |
| 5 | 6899 | 6065 | 24 |
| 6 | 10754 | 10271 | -7 |
| 7 | 4681 | 4376 | 85 |
| 8 | 2522 | 1928 | 95 |
| 4 4 10 | 6559 | 6448 | 15 |
| 12 | 4002 | 4227 | 22 |
| 13 | 2429 | 2848 | 41 |
| 14 | 2306 | 2598 | -12 |
| 4 5 0 | 11890 | 10568 | 180 |
| 1 | 12663 | 12850 | 151 |
| 2 | 4887 | 4170 | 117 |
| 3 | 11973 | 11749 | -132 |
| 4 | 7814 | 7505 | 167 |
| 5 | 13515 | 12816 | -19 |
| 6 | 4388 | 3409 | -134 |
| 7 | 6621 | 5759 | 75 |
| 8 | 4779 | 4183 | 166 |
| 9 | 6823 | 6325 | 177 |
| 10 | 2191 | 977 | -148 |
| 11 | 6797 | 6934 | -77 |
| 12 | 2096 | 1980 | 174 |
| 13 | 2935 | 3083 | 14 |
| 14 | 1878 | 2105 | -135 |
| 4 6 0 | 15809 | 14403 | -180 |
| 1 | 14376 | 13586 | -43 |
| 2 | 8770 | 7293 | -164 |
| 3 | 5578 | 5164 | 41 |
| 4 | 12372 | 12280 | 168 |
| 5 | 11077 | 10549 | 166 |
| 6 | 9298 | 8892 | -164 |
| 7 | 9248 | 8568 | -99 |
| 8 | 7419 | 6936 | -167 |
| 9 | 8344 | 8271 | -42 |
| 10 | 6642 | 6723 | -153 |
| 11 | 4613 | 4738 | 109 |
| 12 | 5721 | 6431 | -178 |
| 13 | 3591 | 4261 | -148 |
| 4 7 0 | 9809 | 9018 | -180 |
| 1 | 8108 | 7137 | -140 |
| 2 | 6140 | 5196 | -133 |
| 3 | 3280 | 2337 | 52 |
| 4 | 9561 | 8874 | -169 |
| 5 | 4845 | 4617 | -71 |
| 6 | 3810 | 4116 | -165 |
| 7 | 3907 | 3279 | -111 |
| 8 | 6694 | 5889 | 176 |
| 10 | 4550 | 5155 | -148 |
| 11 | 2645 | 2143 | -30 |
| 12 | 4550 | 5420 | 173 |
| 13 | 1863 | 2323 | -130 |
| 4 8 0 | 8444 | 6982 | 0 |
| 1 | 10185 | 9526 | 102 |
| 2 | 4969 | 3484 | 0 |
| 3 | 11847 | 11054 | -111 |
| 4 | 3476 | 2813 | 4 |
| 5 | 8848 | 7813 | 3 |
| 6 | 5436 | 4749 | 17 |
| 7 | 10028 | 9941 | 72 |
| 8 | 5290 | 4280 | 60 |
| 9 | 6307 | 6138 | -175 |
| 10 | 2994 | 2464 | -8 |
| 11 | 4414 | 4864 | -93 |
| 12 | 2269 | 2334 | 56 |
| 13 | 4905 | 6189 | 19 |
| 4 9 0 | 8930 | 7800 | 0 |
| 1 | 6877 | 5103 | -20 |
| 2 | 10616 | 9067 | -5 |
| 3 | 13895 | 13228 | 75 |
| 4 | 9295 | 7934 | 10 |
| 5 | 6616 | 5952 | 151 |
| 6 | 10278 | 9560 | 3 |
| 7 | 5146 | 4738 | -132 |
| 8 | 6389 | 6343 | 8 |
| 9 | 7423 | 7018 | 34 |
| 10 | 6394 | 6404 | -4 |
| 11 | 5489 | 6065 | 94 |
| 12 | 4254 | 5504 | 8 |
| 4 10 0 | 6107 | 4512 | 0 |
| 1 | 5806 | 4754 | 22 |
| 2 | 12477 | 10708 | 1 |
| 3 | 5541 | 4492 | 62 |
| 4 | 5567 | 4917 | 21 |
| 5 | 8347 | 7612 | 152 |
| 6 | 8471 | 7255 | -16 |
| 8 | 4650 | 3825 | -11 |
| 9 | 1815 | 1899 | 63 |
| 10 | 5414 | 5337 | 0 |
| 11 | 3154 | 3510 | 104 |
| 12 | 2476 | 2949 | 2 |
| 4 11 0 | 13147 | 12253 | 180 |
| 1 | 8647 | 7745 | 153 |
| 2 | 1649 | 1464 | 157 |
| 3 | 10332 | 9673 | -124 |
| 4 | 3607 | 2217 | 175 |
| 5 | 10714 | 9605 | -28 |
| 6 | 3253 | 3046 | -144 |
| 7 | 6899 | 6208 | 80 |
| 8 | 2925 | 2346 | -176 |
| 9 | 5665 | 5248 | 171 |
| 10 | 2191 | 1627 | 133 |
| 11 | 4531 | 5756 | -85 |
| 4 12 0 | 11507 | 9893 | 180 |
| 1 | 7489 | 6517 | -71 |
| 2 | 7608 | 6791 | 176 |
| 3 | 5033 | 4512 | 26 |
| 4 | 7458 | 6397 | -171 |
| 5 | 4151 | 3494 | -153 |
| 6 | 7290 | 6976 | 174 |
| 7 | 6389 | 5790 | -91 |
| 8 | 7044 | 6520 | 175 |
| 9 | 4275 | 4421 | -30 |
| 10 | 3639 | 4097 | -180 |
| 4 13 0 | 8505 | 7377 | -180 |
| 1 | 7873 | 6869 | -45 |
| 2 | 5414 | 4665 | -164 |
| 3 | 2319 | 1765 | 25 |
| 4 | 6894 | 5934 | -177 |
| 5 | 4940 | 4104 | 157 |
| 6 | 4220 | 3522 | 171 |
| 7 | 5726 | 5055 | -97 |
| 8 | 4382 | 4019 | -171 |
| 9 | 3401 | 3482 | -39 |
| 10 | 2945 | 3782 | -153 |
| 4 14 0 | 3126 | 1555 | 0 |
| 1 | 9516 | 8872 | 149 |
| 4 14 2 | 2721 | 1615 | -35 |
| 3 | 6982 | 6557 | -147 |
| 4 | 1595 | 556 | -164 |
| 5 | 8789 | 8535 | -19 |
| 6 | 2678 | 1912 | 1 |
| 7 | 6173 | 5812 | 94 |
| 8 | 2522 | 2296 | -8 |
| 9 | 4619 | 5515 | 164 |
| 4 15 0 | 9363 | 8891 | 0 |
| 1 | 3567 | 2429 | 41 |
| 2 | 6439 | 5449 | 8 |
| 3 | 3315 | 2857 | 52 |
| 4 | 6677 | 6030 | 4 |
| 5 | 5361 | 4957 | 131 |
| 6 | 5197 | 4576 | 2 |
| 7 | 2177 | 1889 | 37 |
| 8 | 4067 | 4807 | 10 |
| 4 16 0 | 6634 | 6259 | 0 |
| 1 | 2794 | 2574 | -39 |
| 2 | 4718 | 3651 | 17 |
| 3 | 6603 | 6332 | 71 |
| 4 | 5895 | 6087 | 2 |
| 5 | 3575 | 3592 | 135 |
| 6 | 4081 | 3793 | -5 |
| 7 | 3108 | 3312 | -108 |
| 8 | 2405 | 2590 | 9 |
| 4 17 0 | 1831 | 1710 | 180 |
| 1 | 5285 | 5329 | 133 |
| 2 | 1595 | 981 | 100 |
| 3 | 5875 | 6109 | -112 |
| 5 | 3323 | 3682 | -22 |
| 6 | 1649 | 559 | -80 |
| 4 18 0 | 4839 | 5298 | -180 |
| 1 | 2393 | 2047 | -69 |
| 2 | 4992 | 5459 | -165 |
| 4 | 4151 | 4136 | 178 |
| 4 19 0 | 3262 | 3143 | 180 |
| 1 | 5141 | 5512 | -53 |
| 5 1 1 | 5174 | 5500 | -161 |
| 2 | 15403 | 15557 | -38 |
| 3 | 8126 | 7179 | -94 |
| 4 | 21705 | 21999 | 20 |
| 5 | 6625 | 5269 | -72 |
| 6 | 10117 | 9298 | 67 |
| 7 | 3725 | 2511 | 69 |
| 8 | 6421 | 5432 | -45 |
| 9 | 5510 | 4738 | -139 |
| 10 | 10404 | 9991 | -13 |
| 11 | 5382 | 4842 | -84 |
| 12 | 7859 | 8890 | 26 |
| 13 | 2012 | 2387 | -22 |
| 5 2 0 | 13138 | 14756 | 0 |
| 1 | 8316 | 8463 | -46 |
| 2 | 13759 | 14409 | 41 |
| 3 | 5446 | 4528 | -29 |
| 4 | 2204 | 1079 | -105 |
| 5 | 6537 | 6206 | 167 |
| 6 | 13043 | 12559 | -28 |
| 7 | 6252 | 5354 | -75 |
| 8 | 11622 | 10931 | 14 |
| 9 | 6229 | 5320 | -62 |
| 10 | 6998 | 6900 | 52 |
| 12 | 3476 | 3592 | -51 |
| 13 | 1250 | 1583 | -132 |
| 5 3 0 | 6060 | 5339 | 0 |
| 1 | 4663 | 4195 | -133 |
| 2 | 1799 | 1280 | 141 |
| 3 | 3937 | 3255 | 79 |
| 4 | 4797 | 4110 | -75 |
| 5 | 2955 | 2095 | -71 |
| 6 | 2742 | 1953 | -72 |
| 7 | 3623 | 3080 | -74 |
| 9 | 2876 | 2136 | 18 |
| 10 | 2151 | 1972 | 123 |
| 5 4 0 | 17134 | 17196 | 180 |
| 1 | 9982 | 8772 | 129 |
| 2 | 13903 | 14376 | -128 |
| 3 | 2974 | 1972 | -134 |
| 4 | 4052 | 2778 | 47 |
| 5 | 3725 | 2445 | 103 |
| 6 | 14424 | 14487 | 151 |
| 7 | 8260 | 7415 | 54 |
| 8 | 12280 | 12386 | -151 |
| 9 | 6699 | 5786 | 156 |
| 10 | 6555 | 6396 | -108 |
| 12 | 3550 | 3828 | 113 |
| 13 | 2012 | 3282 | 57 |
| 5 5 0 | 4899 | 3832 | 180 |
| 1 | 1799 | 370 | 99 |
| 2 | 12661 | 12211 | 153 |
| 3 | 9254 | 8787 | 92 |
| 4 | 12871 | 13026 | 177 |
| 5 | 4688 | 3228 | 103 |
| 6 | 5571 | 5092 | -156 |
| 7 | 3358 | 2670 | -126 |
| 8 | 3915 | 3271 | 104 |
| 9 | 5915 | 5817 | 79 |
| 10 | 8871 | 9314 | 166 |
| 11 | 2204 | 2134 | 69 |
| 12 | 6168 | 7549 | -150 |
| 13 | 2204 | 3000 | 152 |
| 5 6 0 | 4421 | 3599 | 0 |
| 1 | 4024 | 3191 | -10 |
| 2 | 8236 | 7071 | 117 |
| 3 | 3367 | 2981 | 36 |
| 4 | 7814 | 7234 | -165 |
| 5 | 4505 | 3756 | 116 |
| 6 | 4088 | 3966 | -94 |
| 7 | 2191 | 1686 | -131 |
| 8 | 2429 | 1764 | 55 |
| 9 | 2230 | 1378 | -23 |
| 10 | 4038 | 3406 | 129 |
| 12 | 2556 | 2992 | -130 |
| 5 7 0 | 2476 | 1928 | 0 |
| 1 | 7547 | 6505 | 179 |
| 2 | 12174 | 11638 | -44 |
| 3 | 9929 | 9057 | -84 |
| 4 | 17186 | 17529 | -2 |
| 5 | 5665 | 4686 | -39 |
| 6 | 6074 | 5390 | 76 |
| 7 | 2230 | 940 | -3 |

Table 1 (*cont.*)

| h k l | $F_o$ | $F_c$ | $\alpha_{hkl}$ |
|---|---|---|---|
| 5 7 8 | 5219 | 4781 | -107 |
| 9 | 4706 | 4464 | -170 |
| 10 | 8647 | 8910 | -21 |
| 11 | 5038 | 5548 | -60 |
| 12 | 6376 | 8372 | 32 |
| 5 8 0 | 17005 | 15883 | 0 |
| 1 | 3032 | 2289 | -92 |
| 2 | 7427 | 6365 | 37 |
| 3 | 6515 | 5860 | -58 |
| 4 | 9495 | 8628 | -4 |
| 5 | 5366 | 4479 | -142 |
| 6 | 8340 | 7544 | -13 |
| 7 | 3686 | 2983 | -84 |
| 8 | 10329 | 10643 | 10 |
| 9 | 3288 | 2366 | -65 |
| 10 | 3208 | 3215 | 44 |
| 12 | 3702 | 5007 | -17 |
| 5 9 0 | 8285 | 6311 | 0 |
| 1 | 3051 | 2183 | 3 |
| 2 | 7470 | 6245 | 80 |
| 3 | 4414 | 3537 | -9 |
| 4 | 6021 | 5076 | 173 |
| 5 | 6416 | 5909 | -178 |
| 6 | 6376 | 5815 | -17 |
| 7 | 2243 | 1629 | -52 |
| 8 | 7373 | 7129 | 28 |
| 9 | 2522 | 2389 | -15 |
| 10 | 3847 | 3577 | 108 |
| 12 | 2945 | 3314 | -108 |
| 5 10 0 | 12907 | 12292 | -180 |
| 1 | 8532 | 6896 | 140 |
| 2 | 11434 | 9798 | -120 |
| 3 | 3409 | 2529 | 0 |
| 4 | 10432 | 10152 | 141 |
| 7 | 4322 | 3327 | 68 |
| 8 | 10323 | 10078 | -154 |
| 9 | 5061 | 5021 | 128 |
| 10 | 3710 | 3404 | -108 |
| 11 | 1997 | 2391 | -64 |
| 5 11 0 | 4388 | 3389 | -180 |
| 1 | 3297 | 2112 | 153 |
| 2 | 10205 | 9428 | 172 |
| 3 | 5446 | 4450 | 52 |
| 4 | 7921 | 6937 | -152 |
| 5 | 6358 | 5175 | 122 |
| 6 | 9039 | 9131 | -163 |
| 7 | 2925 | 2401 | 37 |
| 8 | 5831 | 5812 | -161 |
| 9 | 2012 | 1800 | 146 |
| 10 | 4928 | 5849 | -175 |
| 11 | 2331 | 2261 | 106 |
| 5 12 0 | 4123 | 3170 | 0 |
| 1 | 4427 | 3750 | -112 |
| 2 | 8803 | 7789 | 119 |
| 3 | 7095 | 6355 | 75 |
| 4 | 6317 | 5767 | -171 |
| 5 | 3710 | 2720 | 115 |
| 6 | 4454 | 3916 | -90 |
| 7 | 4712 | 4292 | -94 |
| 8 | 4123 | 3975 | 43 |
| 9 | 2110 | 2078 | 71 |
| 10 | 5095 | 4945 | 139 |
| 5 13 0 | 2522 | 1202 | -180 |
| 1 | 2556 | 1676 | 162 |
| 2 | 7431 | 7045 | -51 |
| 3 | 5880 | 5107 | -108 |
| 4 | 11284 | 10803 | 21 |
| 5 | 2545 | 1175 | -22 |
| 6 | 6021 | 5690 | 84 |
| 7 | 2955 | 1999 | 7 |
| 8 | 3172 | 3227 | -54 |
| 9 | 2533 | 2962 | -150 |
| 5 14 0 | 8399 | 7255 | 0 |
| 1 | 4130 | 3623 | -145 |
| 2 | 7141 | 6848 | -19 |
| 3 | 3929 | 3365 | -69 |
| 4 | 8576 | 7849 | -3 |
| 5 | 3003 | 3038 | -106 |
| 6 | 4969 | 4785 | 14 |
| 7 | 3208 | 2695 | -57 |
| 8 | 3764 | 4486 | -7 |
| 5 15 0 | 9996 | 9568 | 0 |
| 1 | 3476 | 3282 | -27 |
| 2 | 5323 | 5177 | 91 |
| 3 | 2487 | 1992 | -14 |
| 4 | 3877 | 3764 | -125 |
| 5 | 3542 | 3431 | 165 |
| 6 | 4731 | 5357 | -50 |
| 7 | 2784 | 3396 | -83 |
| 8 | 4582 | 5184 | 19 |
| 5 16 0 | 7089 | 6877 | -180 |
| 1 | 4038 | 3196 | 117 |
| 2 | 4408 | 4276 | -146 |
| 3 | 1649 | 1084 | -41 |
| 4 | 1613 | 1429 | 79 |
| 5 | 1893 | 564 | 97 |
| 6 | 5425 | 5333 | 143 |
| 5 17 0 | 5691 | 6453 | 180 |
| 1 | 3409 | 3489 | 139 |
| 2 | 5191 | 5363 | -165 |
| 3 | 2405 | 2174 | 50 |
| 4 | 2634 | 2562 | 162 |
| 5 | 1909 | 1096 | 122 |
| 5 18 0 | 4663 | 3964 | 0 |
| 1 | 1595 | 1504 | -17 |
| 2 | 5339 | 5742 | 126 |
| 6 0 0 | 2855 | 2080 | 0 |
| 1 | 12228 | 14299 | -59 |
| 2 | 9103 | 9236 | -58 |
| 3 | 11154 | 11063 | -47 |
| 4 | 12781 | 12589 | 33 |
| 5 | 11798 | 11128 | -153 |
| 6 | 7873 | 6390 | 87 |
| 7 | 13513 | 13007 | -61 |
| 8 | 4441 | 4053 | -46 |
| 9 | 10551 | 10541 | -53 |
| 10 | 7755 | 7913 | -38 |
| 11 | 2012 | 2018 | -109 |
| 12 | 5841 | 7073 | 40 |
| 15 | 3023 | 3671 | -109 |
| 6 1 0 | 5655 | 6248 | 0 |
| 6 1 1 | 7910 | 8938 | -160 |
| 2 | 4479 | 4447 | 41 |
| 3 | 5101 | 4170 | -86 |
| 4 | 3559 | 2931 | -126 |
| 5 | 5929 | 5153 | -65 |
| 6 | 5915 | 5057 | -28 |
| 7 | 3145 | 3683 | -68 |
| 8 | 5557 | 5089 | 33 |
| 9 | 3607 | 3129 | -178 |
| 10 | 1847 | 189 | 46 |
| 11 | 6002 | 6333 | -72 |
| 13 | 900 | 1192 | -93 |
| 6 2 0 | 4934 | 5414 | 0 |
| 1 | 9842 | 10127 | 150 |
| 2 | 6466 | 5710 | 117 |
| 3 | 2825 | 1377 | -101 |
| 4 | 8707 | 7916 | -152 |
| 5 | 6840 | 6061 | 89 |
| 6 | 5141 | 4718 | -75 |
| 7 | 7210 | 6372 | 69 |
| 8 | 2974 | 2195 | 117 |
| 9 | 6801 | 6467 | 159 |
| 10 | 4761 | 4339 | 143 |
| 12 | 2645 | 3046 | -140 |
| 13 | 2742 | 4220 | 59 |
| 6 3 0 | 16364 | 17997 | -180 |
| 1 | 5696 | 5068 | 83 |
| 2 | 8044 | 7893 | -115 |
| 3 | 16034 | 17451 | 70 |
| 4 | 4652 | 3441 | 39 |
| 5 | 13032 | 12926 | 115 |
| 6 | 9217 | 7869 | 137 |
| 7 | 3070 | 2192 | 157 |
| 8 | 10264 | 10069 | -165 |
| 9 | 7924 | 7795 | 69 |
| 10 | 2896 | 3110 | -71 |
| 11 | 7520 | 7882 | 87 |
| 12 | 2204 | 2182 | 89 |
| 13 | 3485 | 5609 | 136 |
| 6 4 0 | 6275 | 5910 | -180 |
| 1 | 7097 | 7170 | 103 |
| 2 | 2393 | 1971 | 116 |
| 3 | 4200 | 3612 | 57 |
| 4 | 4857 | 4080 | 158 |
| 5 | 5084 | 4308 | 86 |
| 6 | 2499 | 2196 | -120 |
| 7 | 4688 | 4067 | 73 |
| 9 | 5609 | 6057 | 98 |
| 10 | 2925 | 2446 | 154 |
| 11 | 1968 | 1718 | 9 |
| 12 | 2083 | 2184 | -136 |
| 6 5 0 | 11517 | 11069 | 0 |
| 1 | 5027 | 4583 | 152 |
| 2 | 6899 | 5942 | 74 |
| 3 | 10927 | 10119 | -89 |
| 4 | 3583 | 2695 | -156 |
| 5 | 5880 | 5016 | -90 |
| 6 | 6599 | 6140 | -70 |
| 7 | 4712 | 3791 | 90 |
| 8 | 6519 | 5681 | 12 |
| 9 | 4248 | 3811 | -162 |
| 10 | 3163 | 3385 | 120 |
| 11 | 4466 | 5278 | -85 |
| 12 | 1700 | 1786 | -114 |
| 6 6 0 | 4981 | 4137 | 180 |
| 1 | 11495 | 10983 | -86 |
| 2 | 10708 | 10237 | -42 |
| 3 | 5821 | 4654 | -12 |
| 4 | 8878 | 8366 | 7 |
| 5 | 8509 | 8067 | -135 |
| 6 | 6720 | 6284 | 73 |
| 7 | 12383 | 12260 | -95 |
| 8 | 3583 | 2910 | -127 |
| 9 | 4582 | 4241 | -43 |
| 10 | 7044 | 7919 | -21 |
| 11 | 2623 | 2748 | -68 |
| 12 | 3435 | 5165 | 55 |
| 6 7 0 | 3199 | 2620 | 0 |
| 1 | 6182 | 5100 | -60 |
| 2 | 3639 | 2805 | -19 |
| 3 | 8859 | 8274 | -73 |
| 4 | 3455 | 3060 | 38 |
| 5 | 4479 | 3713 | -145 |
| 6 | 3885 | 5161 | 31 |
| 7 | 6425 | 5577 | -50 |
| 8 | 3460 | 2893 | -3 |
| 9 | 5141 | 4791 | -89 |
| 10 | 2040 | 2167 | -7 |
| 11 | 1939 | 2781 | -93 |
| 6 8 0 | 5963 | 4530 | 0 |
| 1 | 11381 | 9750 | 153 |
| 2 | 6425 | 5488 | 121 |
| 4 | 5655 | 5183 | -157 |
| 5 | 5619 | 5217 | -2 |
| 6 | 6553 | 5682 | 176 |
| 7 | 6495 | 5790 | 83 |
| 8 | 5920 | 5746 | 45 |
| 9 | 5567 | 5344 | 146 |
| 10 | 4362 | 5070 | 166 |
| 11 | 2393 | 2989 | -56 |
| 6 9 0 | 8537 | 6618 | -180 |
| 1 | 7349 | 5874 | 65 |
| 2 | 6339 | 5394 | -121 |
| 3 | 8825 | 8537 | 74 |
| 4 | 2306 | 1270 | 122 |
| 5 | 11222 | 11172 | 116 |
| 6 | 5680 | 5071 | 151 |
| 7 | 3567 | 2496 | 74 |
| 8 | 6784 | 7247 | -160 |
| 9 | 4031 | 4015 | 71 |
| 10 | 1851 | 2133 | -106 |
| 11 | 5494 | 6712 | 97 |
| 6 10 0 | 5015 | 3891 | -180 |
| 1 | 5665 | 4921 | 119 |
| 2 | 4081 | 3692 | -174 |
| 3 | 9375 | 8381 | 90 |
| 4 | 2417 | 1394 | -154 |
| 5 | 6407 | 5551 | 97 |
| 6 | 3885 | 3675 | 159 |
| 7 | 3352 | 2404 | 135 |
| 8 | 4031 | 3944 | -177 |
| 6 10 9 | 3725 | 4602 | 87 |
| 6 11 0 | 7443 | 6396 | 0 |
| 1 | 5716 | 4619 | 163 |
| 2 | 7466 | 6648 | -82 |
| 3 | 6831 | 6336 | -109 |
| 4 | 6660 | 5490 | -168 |
| 5 | 5665 | 4828 | -43 |
| 6 | 6475 | 6328 | -46 |
| 7 | 3358 | 3187 | 51 |
| 8 | 4335 | 4426 | 32 |
| 9 | 2876 | 3297 | -169 |
| 10 | 3460 | 2974 | 116 |
| 6 12 0 | 2055 | 517 | 180 |
| 1 | 9119 | 8382 | -64 |
| 2 | 6707 | 5787 | -30 |
| 3 | 6126 | 5283 | -67 |
| 4 | 6936 | 5930 | 9 |
| 5 | 5920 | 4876 | -123 |
| 6 | 3135 | 3102 | 50 |
| 7 | 6518 | 6958 | -78 |
| 8 | 2178 | 2109 | -48 |
| 9 | 4302 | 4766 | -63 |
| 6 13 0 | 2269 | 1222 | -180 |
| 1 | 7105 | 6227 | -80 |
| 2 | 4524 | 4531 | -49 |
| 3 | 3341 | 2252 | -23 |
| 4 | 5355 | 5214 | 3 |
| 5 | 4109 | 3612 | -113 |
| 6 | 2896 | 2577 | 73 |
| 7 | 4899 | 5608 | -97 |
| 6 14 0 | 5856 | 5394 | 0 |
| 1 | 5279 | 5033 | 113 |
| 2 | 5257 | 4961 | 114 |
| 3 | 4767 | 4633 | -121 |
| 4 | 5004 | 4956 | -163 |
| 5 | 2612 | 2453 | 19 |
| 6 | 3199 | 3408 | -72 |
| 7 | 3678 | 4318 | 79 |
| 6 15 0 | 4466 | 4280 | -180 |
| 1 | 4193 | 3866 | 100 |
| 2 | 3226 | 2821 | -153 |
| 3 | 6050 | 6502 | 86 |
| 4 | 1423 | 974 | 147 |
| 5 | 3015 | 3543 | 95 |
| 6 | 3208 | 5260 | 161 |
| 6 16 0 | 4833 | 5379 | -180 |
| 1 | 4254 | 4278 | 42 |
| 2 | 3181 | 3609 | -101 |
| 3 | 4518 | 5143 | 65 |
| 4 | 2380 | 2482 | 98 |
| 5 | 4791 | 4863 | 134 |
| 6 17 0 | 4017 | 3942 | 0 |
| 1 | 2634 | 2593 | 147 |
| 2 | 3748 | 3468 | 105 |
| 7 1 0 | 1381 | 2348 | 0 |
| 1 | 12797 | 16122 | 108 |
| 2 | 4045 | 3574 | -36 |
| 3 | 2319 | 1639 | 166 |
| 4 | 3226 | 2690 | 36 |
| 5 | 8165 | 7070 | 43 |
| 6 | 3944 | 3177 | 55 |
| 7 | 13483 | 13277 | 83 |
| 8 | 3061 | 3024 | -137 |
| 9 | 7600 | 7715 | 158 |
| 10 | 2645 | 2471 | 26 |
| 11 | 1153 | 999 | -69 |
| 12 | 2178 | 3306 | 47 |
| 7 2 0 | 3995 | 4079 | 0 |
| 1 | 5851 | 6594 | 28 |
| 2 | 10388 | 9837 | 91 |
| 3 | 14249 | 14779 | 71 |
| 4 | 3599 | 2859 | -39 |
| 5 | 10393 | 9936 | 131 |
| 6 | 2925 | 2585 | -82 |
| 7 | 5061 | 4413 | 149 |
| 8 | 3929 | 3679 | 24 |
| 9 | 7921 | 8018 | 49 |
| 11 | 6102 | 7748 | 93 |
| 7 3 0 | 1649 | 719 | 0 |
| 1 | 5372 | 4963 | 8 |
| 2 | 1700 | 1100 | 156 |
| 4 | 5468 | 2871 | 142 |
| 5 | 4619 | 4159 | 170 |
| 6 | 3510 | 3224 | -25 |
| 8 | 2164 | 1813 | -88 |
| 9 | 1631 | 1866 | 11 |
| 10 | 1799 | 1664 | 116 |
| 11 | 2256 | 2548 | 118 |
| 12 | 1423 | 2380 | -54 |
| 7 4 0 | 7072 | 7035 | -180 |
| 1 | 9326 | 10019 | -164 |
| 2 | 1831 | 1419 | -160 |
| 3 | 12035 | 12510 | -105 |
| 4 | 3980 | 3869 | -46 |
| 5 | 13941 | 13799 | -50 |
| 6 | 5112 | 4542 | 137 |
| 7 | 3181 | 2546 | -7 |
| 8 | 2825 | 2286 | 152 |
| 9 | 6911 | 7208 | -146 |
| 10 | 1863 | 2182 | -88 |
| 11 | 6515 | 8843 | -74 |
| 7 5 0 | 7242 | 7110 | 0 |
| 1 | 10810 | 11208 | -66 |
| 2 | 3772 | 3109 | 167 |
| 3 | 3631 | 3751 | -72 |
| 4 | 3089 | 2165 | -167 |
| 5 | 4916 | 4448 | -119 |
| 6 | 1878 | 638 | -121 |
| 7 | 10553 | 10635 | -88 |
| 8 | 2040 | 626 | 161 |
| 9 | 7373 | 8330 | -62 |
| 10 | 2381 | 2740 | 157 |
| 7 6 0 | 2656 | 1154 | 0 |
| 1 | 8066 | 7622 | -50 |
| 2 | 2984 | 2225 | 117 |
| 3 | 3559 | 2777 | 104 |
| 4 | 2656 | 1940 | -66 |
| 5 | 2069 | 745 | 96 |
| 6 | 2645 | 2512 | -168 |
| 7 | 6206 | 5900 | -122 |
| 8 | 2688 | 2706 | 3 |
| 7 6 9 | 3631 | 3624 | -8 |
| 10 | 1482 | 1115 | -168 |
| 7 7 0 | 2487 | 1691 | 180 |
| 1 | 11812 | 12271 | 115 |
| 2 | 3409 | 3212 | -99 |
| 3 | 4492 | 3960 | -170 |
| 4 | 5562 | 5009 | 39 |
| 5 | 5685 | 5300 | 31 |
| 6 | 3297 | 2484 | 135 |
| 7 | 11992 | 12752 | 85 |
| 8 | 2567 | 2626 | -65 |
| 9 | 6303 | 7259 | 147 |
| 10 | 2393 | 2514 | -6 |
| 7 8 0 | 2994 | 2697 | 0 |
| 1 | 7400 | 6479 | 68 |
| 2 | 1878 | 1848 | -25 |
| 3 | 9625 | 9325 | 84 |
| 4 | 2269 | 467 | 59 |
| 5 | 7755 | 7622 | 112 |
| 6 | 2499 | 1821 | 10 |
| 7 | 5157 | 4438 | 96 |
| 8 | 1249 | 1240 | -44 |
| 9 | 3980 | 4672 | 69 |
| 10 | 2026 | 2467 | 68 |
| 7 9 0 | 6178 | 4964 | 0 |
| 1 | 4518 | 4052 | -24 |
| 2 | 1831 | 1310 | 120 |
| 3 | 7396 | 6430 | 82 |
| 4 | 3748 | 3447 | -54 |
| 5 | 3550 | 3335 | 125 |
| 6 | 1800 | 684 | 158 |
| 7 | 3748 | 3748 | -120 |
| 8 | 2380 | 2076 | 14 |
| 9 | 3825 | 4545 | 13 |
| 10 | 1751 | 2396 | 149 |
| 7 10 1 | 4644 | 3939 | -177 |
| 2 | 4375 | 3916 | -144 |
| 3 | 11371 | 11475 | -110 |
| 4 | 2678 | 2197 | 51 |
| 5 | 8733 | 8917 | -51 |
| 6 | 2256 | 2130 | 125 |
| 7 | 2815 | 2566 | 55 |
| 8 | 2344 | 2346 | -127 |
| 9 | 5431 | 5806 | -130 |
| 10 | 340 | 725 | -107 |
| 7 11 0 | 5372 | 4592 | -180 |
| 1 | 8399 | 8081 | -86 |
| 2 | 3061 | 3312 | 118 |
| 3 | 5129 | 4998 | -79 |
| 4 | 2164 | 2031 | -110 |
| 5 | 6541 | 6466 | -89 |
| 6 | 2763 | 2651 | 177 |
| 7 | 4731 | 5612 | -98 |
| 7 12 0 | 6182 | 5340 | 0 |
| 1 | 7888 | 7601 | -38 |
| 2 | 3172 | 2503 | 132 |
| 3 | 3208 | 3038 | 35 |
| 4 | 1954 | 996 | -151 |
| 5 | 4632 | 4929 | 177 |
| 6 | 1997 | 1753 | -99 |
| 7 | 3802 | 4839 | -103 |
| 7 13 1 | 7052 | 7371 | 116 |
| 2 | 1613 | 1066 | -48 |
| 3 | 3559 | 3554 | 164 |
| 4 | 3855 | 3966 | -16 |
| 5 | 5425 | 6200 | 12 |
| 6 | 1683 | 1814 | 115 |
| 7 | 5552 | 6443 | 96 |
| 7 14 0 | 2164 | 1885 | 0 |
| 1 | 5801 | 6576 | 89 |
| 2 | 1800 | 1133 | -157 |
| 3 | 4447 | 4797 | 108 |
| 4 | 2380 | 2037 | 26 |
| 5 | 4130 | 4885 | 86 |
| 7 15 0 | 2405 | 2340 | 0 |
| 1 | 3959 | 4483 | -22 |
| 2 | 2256 | 1717 | 147 |
| 3 | 5836 | 6021 | 76 |
| 7 16 1 | 1683 | 1854 | -148 |
| 8 0 0 | 1954 | 1578 | 0 |
| 1 | 5811 | 5610 | 111 |
| 2 | 10077 | 11230 | -45 |
| 3 | 3288 | 3223 | 94 |
| 4 | 13010 | 13984 | -1 |
| 5 | 4328 | 3991 | 86 |
| 6 | 7337 | 7937 | 81 |
| 7 | 4130 | 4529 | 93 |
| 8 | 4524 | 4813 | -87 |
| 9 | 3832 | 4453 | 108 |
| 10 | 5452 | 7748 | -26 |
| 8 1 0 | 11064 | 11356 | 0 |
| 1 | 4206 | 5907 | 101 |
| 2 | 3694 | 2714 | 117 |
| 3 | 2511 | 2215 | 55 |
| 4 | 2731 | 1274 | 35 |
| 5 | 3435 | 3091 | 129 |
| 6 | 4382 | 3826 | -33 |
| 7 | 3217 | 2702 | 59 |
| 8 | 4815 | 5494 | -3 |
| 10 | 2191 | 2842 | 103 |
| 8 2 1 | 2645 | 1867 | -58 |
| 2 | 8485 | 8588 | 127 |
| 4 | 4281 | 3686 | -158 |
| 6 | 4447 | 4141 | -129 |
| 7 | 2151 | 2235 | -118 |
| 8 | 3877 | 4339 | 47 |
| 9 | 1878 | 1565 | -64 |
| 10 | 3418 | 5277 | 144 |
| 8 3 0 | 12898 | 14043 | -180 |
| 1 | 4986 | 4893 | -93 |
| 2 | 8753 | 9003 | -126 |
| 3 | 5811 | 5696 | -92 |
| 4 | 3892 | 2971 | 11 |
| 5 | 5027 | 4245 | -75 |
| 6 | 10071 | 10911 | 150 |
| 7 | 3392 | 3590 | -92 |
| 8 | 8623 | 10533 | -152 |
| 9 | 2306 | 3039 | -93 |
| 10 | 2040 | 2587 | -115 |
| 8 4 1 | 4910 | 4636 | -116 |
| 2 | 5690 | 5304 | 165 |
| 8 4 3 | 3418 | 2982 | -67 |
| 4 | 4441 | 4368 | -141 |
| 5 | 3280 | 2561 | -101 |
| 6 | 3443 | 4144 | -164 |
| 7 | 3032 | 3310 | -71 |
| 9 | 1360 | 1579 | -95 |
| 10 | 2204 | 3456 | 170 |
| 8 5 0 | 9912 | 9757 | 0 |
| 1 | 2191 | 1652 | 83 |
| 2 | 7218 | 7349 | 87 |
| 3 | 3341 | 3538 | 97 |
| 4 | 5101 | 4692 | -146 |
| 5 | 3051 | 2417 | 95 |
| 6 | 4518 | 4691 | -33 |
| 8 | 6294 | 7417 | 22 |
| 9 | 1649 | 1851 | 81 |
| 10 | 2294 | 3380 | 115 |
| 8 6 0 | 3358 | 2800 | 0 |
| 1 | 5934 | 6174 | 91 |
| 2 | 7109 | 7708 | -58 |
| 3 | 4486 | 4560 | 102 |
| 4 | 10767 | 11334 | 19 |
| 5 | 4328 | 4419 | 103 |
| 6 | 5650 | 6450 | 68 |
| 7 | 4492 | 4459 | 72 |
| 8 | 2965 | 4003 | -59 |
| 9 | 2440 | 3395 | 110 |
| 10 | 4144 | 6199 | -20 |
| 8 7 0 | 6050 | 4801 | 0 |
| 1 | 2499 | 2450 | 85 |
| 2 | 3032 | 3050 | -13 |
| 3 | 3702 | 3133 | 81 |
| 4 | 5634 | 5680 | -13 |
| 5 | 3460 | 2920 | 98 |
| 6 | 2763 | 2129 | 24 |
| 7 | 2344 | 2733 | 106 |
| 8 | 2731 | 3159 | 15 |
| 9 | 1595 | 2102 | 99 |
| 8 8 0 | 7174 | 7152 | 0 |
| 1 | 2634 | 1729 | -75 |
| 2 | 6961 | 6731 | 133 |
| 3 | 2825 | 3060 | -55 |
| 4 | 7478 | 7524 | -163 |
| 6 | 4315 | 4465 | -77 |
| 7 | 1613 | 1486 | -70 |
| 8 | 2151 | 2351 | 22 |
| 9 | 1317 | 1481 | -58 |
| 8 9 0 | 11116 | 11467 | -180 |
| 1 | 4009 | 4005 | -116 |
| 2 | 5696 | 5861 | -143 |
| 3 | 3944 | 3738 | -76 |
| 4 | 2915 | 2542 | 26 |
| 5 | 4165 | 4072 | -81 |
| 6 | 7166 | 8599 | 158 |
| 7 | 1102 | 2616 | -98 |
| 8 | 5323 | 6060 | -162 |
| 8 10 0 | 6763 | 6807 | -180 |
| 1 | 2380 | 1623 | -94 |
| 2 | 3079 | 3028 | -156 |
| 3 | 2230 | 2809 | -109 |
| 4 | 2096 | 1857 | 119 |
| 5 | 2612 | 2922 | -79 |
| 6 | 3655 | 4211 | 171 |
| 7 | 1649 | 1972 | -87 |
| 8 11 0 | 7770 | 7470 | 0 |
| 2 | 6220 | 6613 | 90 |
| 3 | 1905 | 1644 | 76 |
| 4 | 3631 | 4078 | -141 |
| 5 | 991 | 1069 | 97 |
| 6 | 3951 | 4882 | -65 |
| 8 12 0 | 2356 | 2558 | 0 |
| 1 | 3509 | 3159 | 85 |
| 2 | 4434 | 5067 | -40 |
| 3 | 2164 | 2505 | 99 |
| 4 | 5050 | 6569 | 3 |
| 5 | 2773 | 3329 | 79 |
| 6 | 3199 | 3922 | 44 |
| 7 | 2667 | 2855 | 83 |
| 8 13 1 | 4002 | 4822 | -43 |
| 3 | 1734 | 1701 | 94 |
| 4 | 4833 | 4940 | 15 |
| 8 14 0 | 4186 | 3528 | 0 |
| 1 | 797 | 587 | -88 |
| 2 | 4869 | 5113 | 106 |
| 9 1 0 | 3443 | 3473 | -180 |
| 1 | 5027 | 5111 | 155 |
| 2 | 5191 | 5572 | 144 |
| 3 | 3900 | 4459 | -121 |
| 4 | 7695 | 8623 | -176 |
| 5 | 4815 | 4917 | -31 |
| 6 | 4335 | 4874 | -141 |
| 7 | 4165 | 4957 | 84 |
| 8 | 1863 | 2465 | 151 |
| 9 | 2645 | 4519 | 169 |
| 9 2 0 | 6865 | 6995 | -180 |
| 1 | 4712 | 4764 | -30 |
| 2 | 6856 | 6858 | -146 |
| 3 | 4095 | 4451 | 58 |
| 4 | 4731 | 5317 | 164 |
| 5 | 4268 | 3994 | 163 |
| 6 | 5350 | 6170 | 155 |
| 7 | 2405 | 2967 | -109 |
| 8 | 4607 | 6665 | -155 |
| 9 | 1831 | 3846 | -7 |
| 9 3 0 | 1983 | 2409 | -180 |
| 2 | 2083 | 1436 | -143 |
| 3 | 2440 | 2297 | 51 |
| 4 | 1734 | 1748 | -75 |
| 6 | 2405 | 2714 | 162 |
| 9 | 1799 | 1317 | -13 |
| 9 4 0 | 8454 | 8854 | 0 |
| 1 | 5061 | 5299 | 127 |
| 2 | 5841 | 6070 | 16 |
| 3 | 7250 | 8380 | -130 |
| 4 | 1683 | 1502 | 17 |
| 5 | 5895 | 6210 | -19 |
| 6 | 6707 | 8516 | -14 |
| 7 | 4193 | 4825 | 81 |
| 8 | 3615 | 5048 | 14 |
| 9 5 1 | 4031 | 4301 | -52 |
| 2 | 6907 | 7842 | -2 |

Table 1 (*cont.*)

| h | k | l | $F_o$ | $F_c$ | $\alpha_{hkl}$ |
|---|---|---|---|---|---|
| 9 | 5 | 5 | 3392 | 3279 | 75 |
| | | 4 | 7502 | 8584 | 3 |
| | | 6 | 3332 | 3860 | 25 |
| | | 7 | 1997 | 2861 | -106 |
| | | 8 | 2522 | 4339 | 9 |
| 9 | 6 | 1 | 3297 | 3193 | -32 |
| | | 2 | 1997 | 2107 | -94 |
| | | 3 | 2835 | 2906 | 60 |
| | | 4 | 2751 | 2962 | 43 |
| | | 5 | 2935 | 2636 | 157 |
| | | 7 | 1851 | 2106 | -82 |
| 9 | 7 | 0 | 2487 | 2168 | -180 |
| | | 1 | 3756 | 4063 | 119 |
| | | 2 | 4981 | 5550 | 145 |
| | | 3 | 4524 | 5147 | -135 |
| | | 4 | 7669 | 9213 | -166 |
| | | 5 | 5078 | 5694 | -20 |
| | | 6 | 3032 | 3463 | -132 |
| | | 7 | 4052 | 4498 | 99 |
| 9 | 8 | 0 | 7321 | 7946 | 180 |
| | | 1 | 3208 | 3091 | -30 |
| | | 2 | 4179 | 4841 | -175 |
| 9 | 8 | 3 | 2306 | 2400 | 84 |
| | | 4 | 3599 | 4452 | 178 |
| | | 5 | 1272 | 1268 | -165 |
| | | 6 | 3061 | 4199 | 179 |
| 9 | 9 | 0 | 3710 | 3864 | -180 |
| | | 1 | 4137 | 4300 | -30 |
| | | 2 | 1939 | 2124 | -120 |
| | | 3 | 2804 | 3169 | 74 |
| | | 4 | 2151 | 2502 | 48 |
| | | 5 | 2623 | 2616 | -179 |
| | | 6 | 1381 | 1297 | 152 |
| 9 | 10 | 0 | 4179 | 4264 | 0 |
| | | 1 | 4095 | 4721 | 144 |
| | | 2 | 4857 | 5947 | 33 |
| | | 3 | 2865 | 4049 | -122 |
| | | 4 | 1666 | 2397 | -54 |
| | | 5 | 4512 | 4623 | -26 |
| 9 | 11 | 0 | 4755 | 6569 | 0 |
| | | 1 | 1767 | 1490 | 7 |
| | | 2 | 5382 | 5413 | -19 |
| | | 3 | 760 | 959 | -143 |
| | | 4 | 2623 | 3176 | 21 |
| 9 | 12 | 0 | 1939 | 1542 | -180 |
| | | 1 | 4749 | 4966 | -56 |
| 10 | 0 | 0 | 5875 | 6837 | -180 |
| | | 1 | 5645 | 7444 | 115 |
| | | 2 | 6699 | 8188 | -139 |
| | | 3 | 3787 | 4831 | 165 |
| | | 4 | 3749 | 4690 | 127 |
| | | 5 | 4342 | 5883 | 9 |
| | | 6 | 3190 | 4782 | 144 |
| | | 7 | 4351 | 6661 | 88 |
| 10 | 1 | 1 | 2855 | 3061 | 7 |
| | | 2 | 2916 | 3121 | 134 |
| | | 3 | 3271 | 3920 | 91 |
| | | 4 | 3042 | 3610 | -149 |
| | | 5 | 2151 | 2332 | 115 |
| | | 6 | 1924 | 2875 | -167 |
| | | 7 | 798 | 1399 | 151 |
| 10 | 2 | 0 | 6664 | 7697 | 0 |
| | | 1 | 3599 | 4268 | -49 |
| | | 2 | 1799 | 1314 | 32 |
| | | 3 | 1924 | 2024 | 36 |
| | | 5 | 2306 | 3065 | -164 |
| 10 | 2 | 6 | 2440 | 3257 | -29 |
| | | 7 | 1443 | 3251 | -111 |
| 10 | 3 | 1 | 1649 | 1427 | -135 |
| | | 2 | 4473 | 5197 | -27 |
| | | 3 | 5776 | 7809 | -111 |
| | | 4 | 6150 | 8284 | 5 |
| | | 5 | 5152 | 6797 | -45 |
| | | 6 | 2256 | 3380 | 64 |
| 10 | 4 | 1 | 3885 | 4046 | -54 |
| | | 2 | 2906 | 3399 | 44 |
| | | 3 | 1613 | 1897 | -141 |
| | | 4 | 2083 | 1963 | 0 |
| | | 5 | 2151 | 2462 | -60 |
| | | 6 | 1295 | 2111 | -4 |
| 10 | 5 | 0 | 2601 | 2308 | 0 |
| | | 1 | 2381 | 2133 | -17 |
| | | 2 | 3032 | 3727 | 135 |
| | | 3 | 3315 | 4348 | 83 |
| | | 4 | 2994 | 3520 | -172 |
| | | 5 | 2306 | 3129 | 136 |
| | | 6 | 1878 | 3328 | -90 |
| 10 | 6 | 0 | 5010 | 5679 | -180 |
| | | 1 | 5382 | 7274 | 108 |
| | | 2 | 2906 | 3911 | -149 |
| | | 3 | 2634 | 2814 | -179 |
| | | 5 | 3623 | 4705 | 27 |
| 10 | 7 | 1 | 2110 | 2611 | 96 |
| | | 2 | 3795 | 4372 | -154 |
| | | 3 | 1863 | 2558 | 98 |
| | | 4 | 2906 | 3688 | 158 |
| 10 | 8 | 1 | 5409 | 6288 | -48 |
| | | 2 | 3079 | 3103 | 85 |
| | | 3 | 2452 | 2892 | 46 |
| 10 | 9 | 0 | 2667 | 2626 | 0 |
| | | 1 | 1649 | 1799 | -134 |
| 10 | 10 | 0 | 1783 | 1551 | 0 |
| 11 | 1 | 0 | 4499 | 5215 | 0 |
| | | 1 | 3271 | 3964 | -100 |
| | | 2 | 4165 | 5394 | 87 |
| 11 | 2 | 0 | 3733 | 4416 | -180 |
| | | 1 | 3817 | 5217 | -81 |
| | | 2 | 2876 | 3152 | -45 |

Table 2. *Atomic and thermal parameters and their standard deviations* ($\times 10^4$, *in parentheses*)

| | *x/a* | *y/b* | *z/c* | $U_{150}$ |
|---|---|---|---|---|
| Sb(1) | 0·3615 (2) | 0·1627 (1) | 0·5019 (1) | 0·0186 (5) |
| Sb(2) | 0·4355 (2) | 0·3199 (1) | 0·1377 (2) | 0·0188 (5) |
| K(1) | 0·3803 (11) | 0·0175 (5) | 0·2367 (8) | 0·0363 (20) |
| K(2) | 0·3024 (10) | 0·5652 (5) | 0·3331 (8) | 0·0329 (18) |
| O(1) | 0·1998 (28) | 0·1040 (15) | 0·3827 (23) | 0·0264 (56) |
| O(2) | 0·0124 (37) | 0·1286 (19) | 0·2694 (24) | 0·0415 (73) |
| O(3) | 0·2601 (28) | 0·2514 (13) | 0·1906 (19) | 0·0205 (48) |
| O(4) | 0·2477 (28) | 0·2614 (12) | 0·4364 (18) | 0·0156 (43) |
| O(5) | 0·5364 (29) | 0·2543 (15) | 0·5284 (21) | 0·0252 (55) |
| O(6) | 0·4961 (27) | 0·1452 (14) | 0·3662 (20) | 0·0201 (48) |
| O(7) | 0·7625 (35) | 0·3019 (16) | 0·4621 (23) | 0·0332 (61) |
| O(8) | 0·6740 (31) | 0·0995 (16) | 0·1601 (23) | 0·0329 (62) |
| O(9) | 0·5167 (27) | 0·1946 (14) | 0.0994 (20) | 0·0219 (50) |
| O(10) | 0·5838 (30) | 0·3056 (16) | 0·2510 (23) | 0·0280 (57) |
| O(11) | 0·3667 (42) | 0·4400 (21) | 0·5202 (33) | 0·0561 (92) |
| O(12) | 0·3303 (27) | 0·4064 (15) | 0·2478 (21) | 0·0219 (51) |
| O(13) | 0·1094 (34) | 0·4281 (18) | 0·3304 (26) | 0·0373 (69) |
| O(14) | 0·6728 (35) | 0·4344 (19) | 0·5993 (26) | 0·0395 (73) |
| O(15) | 0·4679 (52) | −0·0083 (30) | 0·4927 (43) | 0·1019 (124) |
| C(1) | 0·1083 (40) | 0·1543 (21) | 0·3307 (25) | 0·0123 (57) |
| C(2) | 0·1294 (35) | 0·2473 (19) | 0·3618 (30) | 0·0216 (69) |
| C(3) | 0·1548 (35) | 0·2929 (21) | 0·2544 (28) | 0·0156 (63) |
| C(4) | 0·1946 (40) | 0·3797 (19) | 0·2833 (27) | 0·0139 (59) |
| C(5) | 0·6472 (36) | 0·2506 (20) | 0·4586 (30) | 0·0217 (71) |
| C(6) | 0·6376 (37) | 0·1880 (19) | 0·3673 (28) | 0·0157 (61) |
| C(7) | 0·6644 (43) | 0·2270 (20) | 0·2570 (29) | 0·0186 (67) |
| C(8) | 0·6169 (34) | 0·1697 (22) | 0·1689 (31) | 0·0267 (77) |

cess was continued using the full-matrix least-squares method assuming independent isotropic thermal parameters for each atom. The weighting scheme used was

$$\sqrt{w} = \sqrt{\left(\frac{1}{1+\left(\frac{|F_o|-b}{a}\right)^2}\right)},$$

where $a=b=40$ on the same scale as Table 1. The final reliability index for the observed reflexions was $R=0{\cdot}11$. Table 1 lists the observed structure amplitudes and calculated structure factors based on the final atomic coordinates given in Table 2. Atomic scattering factors used were those in *International Tables for X-ray Crystallography* (1962).

## Description and discussion of the structure

It follows from the interatomic distances and angles given in Tables 3 and 4 as well as from Figs. 1 and 2 that the crystal structure of 'tartar emetic' is built up of dimeric tartratoantimonate(III) ions, $[Sb_2(C_4H_2O_6)]^{2-}$. It confirms the previously reported structure of this ion in ammonium antimonyl-(±)-tartrate monohydrate (Kiosse, Golovastikov & Belov, 1964), tris-*o*-phenanthroline-iron(II) antimony(+)-tartrate (Templeton, Zalkin & Ueki, 1966) and ammonium antimonyl-(+)-tartrate hemihydrate (Kiosse, Golovastikov, Ablov & Belov, 1967). Both antimony atoms in the $[Sb_2(C_4H_2\text{-}O_6)_2]^{2-}$ ion are bound to four oxygen atoms in a slightly deformed square pyramid whose apex is

occupied by the unshared electron pair. Two oxygen atoms are from two carboxyl groups and the other two oxygen atoms from the α-hydroxyl groups at mean distances of 2·20 and 2·01 Å respectively. These values of the Sb–O bond lengths are close to those found in $(NH_4)_2[Sb_2(D\text{-tart})_2].3H_2O$ (2·18 and 2·02 Å respectively) as well as in $(NH_4)_2[Sb_2(D,L\text{-tart})_2].4H_2O$ (2·15 and 2·04 Å respectively). The antimony atom thus belongs to two nearly planar five-membered rings with an average O–Sb–O angle of 80°.

The anion $[Sb_2(C_4H_2O_6)]^{2-}$ is asymmetric in the crystal structure, since the space group $Pca2_1$ does not require any point symmetry, but, if idealized, the anion would have point symmetry $D_2$. Racemic 'tartar emetic'

Table 3. *Some interatomic distances with standard deviations (in parentheses)*

The positions are denoted as follows:

| | | | |
|---|---|---|---|
| No superscript | $x$ | $y$ | $z$ |
| (i) | $\frac{1}{2}+x$, | $-y$ | $z$ |
| (ii) | $-x$, | $-y$, | $\frac{1}{2}+z$ |

(*a*) Within the tartratoantimonate(III) ion

| | |
|---|---|
| Sb(1)–O(1) | 2·25 (0·03) Å |
| Sb(1)–O(4) | 2·06 (0·03) |
| Sb(1)–O(5) | 2·17 (0·03) |
| Sb(1)–O(6) | 2·05 (0·02) |
| Sb(2)–O(3) | 2·01 (0·03) |
| Sb(2)–O(9) | 2·22 (0·03) |
| Sb(2)–O(10) | 1·91 (0·03) |
| Sb(2)–O(12) | 2·16 (0·03) |
| O(1)—C(1) | 1·31 (0·04) |
| O(2)—C(1) | 1·20 (0·05) |
| O(4)—C(2) | 1·40 (0·04) |
| O(5)—C(5) | 1·29 (0·04) |
| O(6)—C(6) | 1·43 (0·04) |
| O(7)—C(5) | 1·31 (0·05) |
| O(3)—C(3) | 1·39 (0·04) |
| O(9)—C(8) | 1·29 (0·04) |
| O(8)—C(8) | 1·26 (0·04) |
| O(10)–C(7) | 1·47 (0·05) |
| O(12)–C(4) | 1·34 (0·04) |
| O(13)–C(4) | 1·23 (0·04) |
| C(1)—C(2) | 1·57 (0·05) |
| C(2)—C(3) | 1·52 (0·05) |
| C(3)—C(4) | 1·50 (0·05) |
| C(5)—C(6) | 1·51 (0·05) |
| C(6)—C(7) | 1·51 (0·05) |
| C(7)—C(8) | 1·48 (0·05) |

(*b*) Coordination about K(1)

| | |
|---|---|
| K(1)···O(1) | 2·77 (0·04) Å |
| K(1)···O($2^{ii}$) | 2·68 (0·04) |
| K(1)···O(6) | 2·81 (0·04) |
| K(1)···O($8^{i}$) | 2·79 (0·04) |
| K(1)···O(8) | 3·06 (0·04) |
| K(1)···O($15^{ii}$) | 3·26 (0·06) |

(*c*) Coordination about K(2)

| | |
|---|---|
| K(2)···O($7^{i}$) | 2·70 (0·04) Å |
| K(2)···O($10^{i}$) | 3·02 (0·04) |
| K(2)···O(11) | 3·11 (0·05) |
| K(2)···O(12) | 2·80 (0·04) |
| K(2)···O(13) | 2·81 (0·04) |
| K(2)···O($13^{i}$) | 2·70 (0·04) |
| K(2)···O($14^{ii}$) | 2·86 (0·04) |

(*d*) Hydrogen-bonded contact distances

| | |
|---|---|
| O(11)···O(14) | 2·86 Å |
| O(11)···O(4) | 3·26 |
| O(14)···O($11^{i}$) | 2·84 |
| O(14)···O(7) | 2·85 |
| O(15)···O(6) | 2·95 |
| O(15)···O($8^{ii}$) | 2·82 |

Table 4. *Some interatomic angles with standard deviations (in parentheses)*

(*a*) At the antimony atoms

| | | | |
|---|---|---|---|
| O(1)–Sb(1)–O(4) | 77·0 (1·0)° | O(3)—Sb(2)–O(9) | 78·5 (1·0) |
| O(1)–Sb(1)–O(6) | 77·5 (1·0) | O(3)—Sb(2)–O(12) | 80·6 (1·0) |
| O(4)–Sb(1)–O(5) | 82·0 (1·0) | O(9)—Sb(2)–O(10) | 79·7 (1·0) |
| O(5)–Sb(1)–O(6) | 78·9 (1·0) | O(10)–Sb(2)–O(12) | 85·6 (1·0) |

(*b*) Within the tartrate groups

| | | | |
|---|---|---|---|
| Sb(1)–O(1)—C(1) | 115 (2·3)° | Sb(1)–O(5)—C(5) | 113 (2·2)° |
| Sb(1)–O(4)—C(2) | 119 (2·1) | Sb(1)–O(6)—C(6) | 115 (2·0) |
| O(1)—C(1)—C(2) | 114 (3·0) | O(5)—C(5)—C(6) | 118 (3·1) |
| C(1)—C(2)—O(4) | 113 (2·9) | C(5)—C(6)—O(6) | 112 (2·8) |
| O(1)—C(1)—O(2) | 120 (3·4) | O(5)—C(5)—O(7) | 121 (3·3) |
| O(2)—C(1)—C(2) | 124 (3·3) | O(7)—C(5)—C(6) | 119 (3·2) |
| C(1)—C(2)—C(3) | 106 (2·8) | C(5)—C(6)—C(7) | 111 (2·9) |
| O(4)—C(2)—C(3) | 111 (2·9) | O(6)—C(6)—C(7) | 109 (2·8) |
| C(2)—C(3)—O(3) | 110 (2·8) | C(6)—C(7)—O(10) | 109 (2·9) |
| C(2)—C(3)—C(4) | 107 (2·8) | C(6)—C(7)—C(8) | 109 (3·1) |
| O(3)—C(3)—C(4) | 116 (2·9) | O(10)–C(7)—C(8) | 112 (3·0) |
| C(3)—C(4)—O(12) | 115 (3·0) | C(7)—C(8)—O(9) | 118 (3·3) |
| C(3)—C(4)—O(13) | 125 (3·3) | C(7)—C(8)—O(8) | 121 (3·4) |
| O(12)–C(4)—O(13) | 118 (3·3) | O(9)—C(8)—O(8) | 120 (3·4) |
| Sb(2)–O(3)—C(3) | 114 (2·0) | Sb(2)–O(10)–C(7) | 118 (1·5) |
| Sb(2)–O(12)–C(4) | 111 (2·1) | Sb(2)–O(9)—C(8) | 111 (2·2) |

is therefore a racemic mixture of dimeric (+)-tartratoantimonate(III) and (−)-tartratoantimonate(III), and not a (±)-tartratoantimonate whose dimeric anion would be formed of both (+) and (−)-tartaric acid. The (+), (−) combination has been observed so far only in the case of the divanadyl(IV) tartrate ion (Tapscott, Belford & Paul, 1968) but the divanadyl (IV) tartrate ion occurs in the optically active (+) form (Forrest & Prout, 1967). A *meso*-tartrate of this kind is sterically impossible (Reihlen & Hezel, 1931). There is no antimonyl group in the structure of the so called

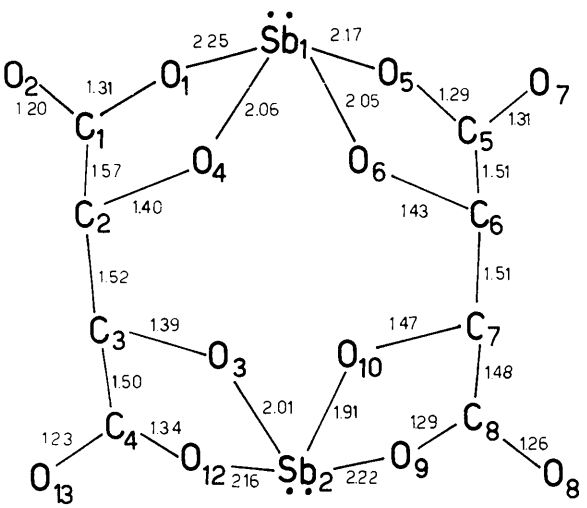


Fig. 1. The $[Sb_2(C_4H_2O_6)_2]^{2-}$ ion with atom designations and bond lengths.

antimonyl-tartrates; the oxygen atoms required by the chemical analysis belong to the water of crystallization. The proper formula of 'tartar emetic' is thus $K_2[Sb_2(C_4H_2O_6)_2].3H_2O$ and not $KSbOC_4H_4O_6.\frac{1}{2}H_2O$ or $K[Sb(C_4H_2O_6)H_2O].\frac{1}{2}H_2O$ (*Gmelin's Handbuch,* 1953).

The three crystallographically independent water molecules are differently bound in the crystal structure. While $H_2O(11)$ and $H_2O(14)$ build up the mutually hydrogen-bonded zigzag chains between $[Sb_2(C_4H_2O_6)_2]^{2-}$ ions along the *a* axis, the third water molecule $[H_2O(15)]$ has no contact with other water molecules. At the same time $H_2O(11)$ and $H_2O(14)$ are more closely bound to the potassium ions than $H_2O(15)$. The potassium–oxygen (11) and potassium–oxygen(14) distances are 3·11 and 2·86 Å respectively, while the potassium–oxygen(15) distance is 3·26 Å. These results explain the observations that potassium antimonyl tartrate on heating loses one third of its water at 100 °C, while the remaining two thirds is driven off only at 200 to 220 °C (*Gmelin's Handbuch,* 1953). This is probably also the reason for the relatively high temperature factor found for the oxygen atom which belongs to the water molecule $H_2O(15)$. The hydrogen bond lengths are given in Table 3.

The coordination of each potassium ion is different both as to the number of and the distances from the surrounding oxygen atoms. There are six oxygen atoms about K(1) at distances of 2·68 to 3·26 Å and sevenfold coordination is completed by the unshared electron pair from the antimony atom Sb(1). There are seven oxygen atoms about K(2) at distances of 2·70 to 3·11 Å and eightfold coordination is similarly completed

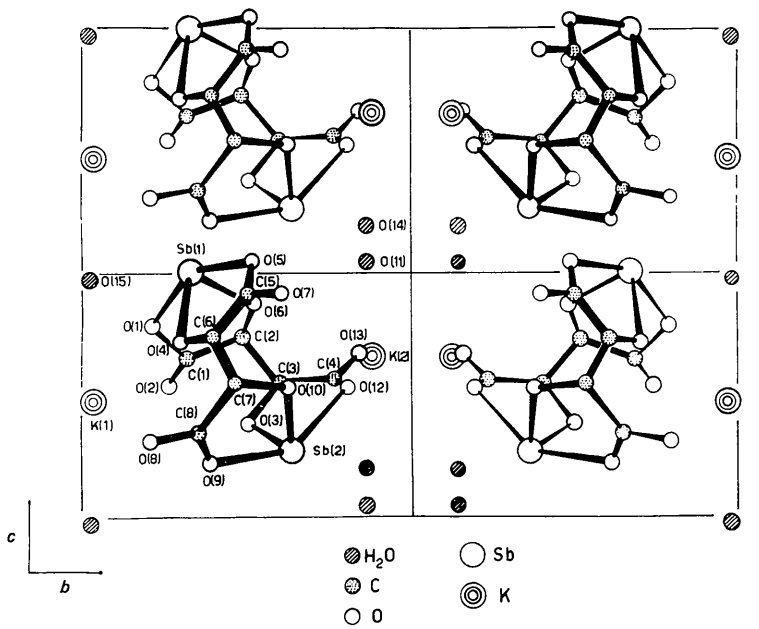


Fig. 2. The crystal structure of racemic potassium di-$\mu$-tartratodiantimonate(III) trihydrate projected along the *a* axis.

by the unshared electron pair from Sb(2). The next nearest oxygen atom to K(1) is O(15) at 3·46 Å, and symmetrically related O(14) to K(2) at 3·44 Å. The coordination polyhedra about the potassium ions are similar to those found in other potassium salts where the potassium-oxygen distances are also in the range 2·62 to 3·20 Å (Palenik, 1967).

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# X-ray Structure Determination of (+)-Dibromodehydrotetrahydrorugulosin, a Heavy Atom Derivative of (+)-Rugulosin

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The crystal structure of (+)-dibromodehydrotetrahydrorugulosin water and methanol solvate, $C_{30}H_{22}O_{10}Br_2.H_2O.2CH_3OH$, has been determined in order to elucidate the molecular structure and absolute configuration of rugulosin, $C_{30}H_{22}O_{10}$, a fungal pigment isolated from *Penicillium rugulosum* Thom. The crystals are monoclinic with space group $P2_1$ and the unit-cell dimensions are $a=9{\cdot}78$, $b=17{\cdot}04$, $c=9{\cdot}45$ Å and $\beta=98{\cdot}0°$. Two formula units are contained in the cell. The crystal structure was solved by the heavy-atom method and refined by the block-matrix least-squares method including anisotropic thermal parameters. The final $R$ value for 1482 non-zero observed structure factors was 0·109. The absolute configuration was determined by the use of the anomalous dispersion of bromine atoms for Cu $K\alpha$ radiation.

The molecule consists of two tricyclic rings of partially hydrogenated anthraquinone cross-linked at the $A$ and $A'$ rings by means of four C–C covalent bonds. On the basis of the results obtained by the present structure determination, the structures and stereochemistry of (+)-rugulosin, (−)-luteoskyrin and (−)-rubroskyrin have been established, including their absolute configurations.

## Introduction

(+)-Rugulosin, $C_{30}H_{22}O_{10}$, is a fungal pigment isolated from *Penicillium rugulosum* Thom and some other fungi (Shibata, Tanaka, Chihara & Mitsuhashi, 1952; Breen, Dacre, Raistrick & Smith, 1955; Shibata & Udagawa, 1963). The chemical structure of rugulosin has since been extensively investigated and it has been shown that it is a representative of a new group of colouring matters having a dimeric structure of partially hydrogenated anthraquinones (Shibata, Murakami, Kitagawa & Kishi, 1956; Shibata, Murakami & Takido, 1956; Briggs & LeQuesne, 1965; Shibata, Ogihara, Kobayashi, Seo & Kitagawa, 1968; Sankawa, Seo, Kobayashi, Ogihara & Shibata, 1968).

Among the related compounds obtained from *Penicillium islandicum* Sopp, (−)-luteoskyrin, $C_{30}H_{22}O_{12}$ and rubroskyrin, $C_{30}H_{22}O_{12}$, were shown to have very close structures to rugulosin. Luteoskyrin was particularly noted as a toxic principle causing liver damage and sometimes liver cancer in experimental animals. To date, several structures have been put forward for these substances mainly on the basis of chemical and spectroscopic data, but they are not conclusive. An X-ray structure analysis of a heavy-atom derivative of rugulosin was therefore undertaken. A preliminary report of the present study has already been published (Kobayashi, Iitaka, Sankawa, Ogihara & Shibata, 1968).

## Experimental

Various attempts at bromination of the skeleton of rugulosin failed to produce a suitable derivative for X-ray analysis. It was finally found that only the hydro-