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# Synthesis, Crystal Structure and Charge Distribution of $InGaZnO_4$. X-ray Diffraction Study of 20kb Single Crystal and 50kb Twin by Reticular Merohedry

Synthesis of $InGaZnO_4$ at 20Kb and 50Kb produced a single crystal and a twinned crystal respectively. The X-ray diffraction crystal structure refinement ($R1$ = 0.015 and 0.019 respectively) and the Charge Distribution analysis are reported. Space group is $R\bar{3}m$; cell parameters in hexagonal axes are $a$ = 3.2990(2)Å, $c$ = 26.1013(25)Å (20kb single crystal) and $a$ = 3.3051(2)Å, $c$ = 26.1029(19)Å (50kb twinned crystal). The cell volume is 246.01(3)Å$^3$ and 246.94(3)Å$^3$ respectively. The In is in regular octahedral coordination, whereas Ga/Zn are in trigonal bipyramid co-ordination. Charge Distribution on cations (2.94 and 2.95 respectively *vs*. 3.0 for In, and 2.53 *vs*. 2.5 for Ga/Zn) shows that the structure is well refined. Charge Distribution on oxygens (–1.96 and –2.04 for O1 and O2, *vs*. –2.0) excludes the presence of valence unbalance effects. A possible structural role of the trigonal bipyramidal coordination is discussed.

Keywords: charge distribution, structure refinement, twinning, single crystal, X-ray diffraction

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## 1. Introduction

Oxides of the transition elements have wide applications as electromagnetic components. Complex oxides of $In^{3+}$ are transparent conductors with high electrical conductivity at room temperature. $In_2O_3$ is used for making transparent conductive electrodes and ZnO is used for varistors (KIMIZUKA *et al*., 1995; BROWN *et al*., 1999). Compounds of the cations with the $d^{10}s^0$ electronic configuration, when synthesised at high pressure take a structure that cannot be obtained in an atmospheric pressure synthesis, and this structure is retained after lowering the pressure. The monoclinic β-$InGaO_3$, in which Ga has fourfold coordination, undergoes a high-pressure reconstructive transformation towards hexagonal $InGaO_3$-II, in which Ga is five-coordinated. The $InGaO_3$-II phase did not revert to β-$InGaO_3$ even after heating at 1020°C: this unusual thermal stability is thought to be related to the reconstructive transformation (SHANNON and PREWITT, 1968). Complex oxides of the $d^{10}s^0$ cations show a high conductivity at room temperature (*e.g.* $In_2Ge_2O_7$: SHANNON and SLEIGHT, 1968). Mixed oxides of the transition elements synthesised at high pressure are thus expected to play an important role in the development of transparent electrodes.

The research on pyroxene minerals containing $In^{3+}$, $Ga^{3+}$ and $Zn^{2+}$ has revealed that the basic features of these cations, such as the effective ionic radii and the electronegativity, change with the strength of the chemical bonds, temperature and pressure (OHASHI, 1989; OHASHI *et al*., 1983; 1990; 1995; 1996). This change is likely related to the cations themselves, and it is thus likely to occur in other compounds as well. The crystal synthesised at high pressure and then quenched at room pressure may preserve the original structure, although undergoing electronic changes that can explain the unusual electrical conductivity shown by these compounds. The relation between the electronic status (unquenchable) and the structure (quenchable) may be the key to explain the interesting features shown by these compounds. In this perspective, the research on In-Ga-Zn compounds has started.

In comparison to other cations having similar ionic radii (*e.g.* $Mg^{2+}$ and $Al^{3+}$), $Zn^{2+}$ and $Ga^{3+}$ take a lower coordination, often in the shape of trigonal bipyramids (SHANNON and PREWITT, 1968; GOPAL and CALVO, 1973; GELLER *et al.*, 1975; NORD and STEFANIDIS, 1982; VELIKODNYI *et al.*, 1989; ISOBE *et al.*, 1994; TRINSCHEK and JANSEN , 1996; FENG *et al.*, 1997). In order to elucidate the relation between this kind of coordination and the structural and electronic properties, it is necessary to further investigate oxides containing trigonal bipyramids.

The synthesis of $InGaZnO_4$, in which In is octahedrally coordinated and Zn/Ga are in trigonal bipyramids, has been successfully performed at 20kb and 50kb, but in the second case a twinned crystal has been obtained. The structures of both crystals, determined by single crystal X-ray diffraction (XRD), is here reported, together with the Charge Distribution (CD) analysis (HOPPE *et al.*, 1989; NESPOLO *et al.*, 1999).

## 2. Experimental

Mixtures of $In_2O_3$, $Ga_2O_3$ and ZnO (proportions 1:1:2) were crystallised by solid state reaction in platinum capsules.

A piston cylinder type high-pressure apparatus was used for 20Kb synthesis (1200ºC, 22hours), and a belt type apparatus for 50Kb synthesis (1200ºC, 20hours). Synthesis at 50kb gave two types of crystals. One had a disc-like morphology and apparent hexagonal symmetry: it turned out to be a twin by reticular merohedry (FRIEDEL, 1904; 1926), with twin law $[001]_\pi$. The second crystal had an elongated morphology and rhombohedral symmetry, with cell parameters practically identical to the 20kb one: the peaks were however too broad for data collection.

Details of the crystal data and of the refinement are given in Table 1. Unit cell dimensions were determined from $2\vartheta$ values of 18 (20kb) and 17 (50kb) reflections respectively, in the range $27.5° \leq \vartheta \leq 32.5°$. The intensities were measured at room temperature with an Enraf-Nonius CAD-4 diffractometer, using graphite-monocromatised MoKα ($\lambda$ = 0.71073Å) radiation (Table 1). Intensity collection was performed in the range $2.34° \leq \vartheta \leq 39.71°$ (20kb) and $2.34° \leq \vartheta \leq 34.87°$ (50kb) using variable-rate $\omega$-$2\vartheta$ scans with scan range 1.05º+0.525ºtan$\vartheta$. The LP corrections were performed through the XCAD4 program (HARMS, 1996), and the analytical absorption correction with the PLATON package (SPECK, 1990). The structure refinement has been performed against $F^2$ with SHELX-97 (SHELDRICK, 1997). Initial positional parameters and isotropic displacement factors were taken from $YbFe_2O_4$ (KATO *et al.*, 1975). Results are shown in Tables 2-4. For the 50kb sample, reflections were initially collected in a hexagonal cell and have been re-indexed in the rhombohedral cell. Reflections satisfying the rhombohedral reflection condition in both settings [$-h_I+k_I+l = 0 \pmod 3$; $-h_{II}+k_{II}+l = 0 \pmod 3$; $h_{II} = -h_I$ and $k_{II} = -k_I$] were assigned to both crystals. Reflections for which the rhombohedral reflection condition was satisfied in only one of the two settings were assigned to the corresponding individual. Reflections violating the rhombohedral reflection condition in both settings were rejected: these included 15 weak reflections with $I > 2\sigma(I)$ ($I_{max}$ = 26.98), whereas all the other rejected reflections had $I < 2\sigma(I)$ (see also HERBST-IRMER and SCHELDRICK, 1997).

## 3. Results

NAKAMURA *et al*. (1991) synthesised a wide range of phases in the system $In_2O_3 – Ga_2ZnO_4 –$ ZnO at atmospheric pressure, and reported the cell constants determined by powder X-ray diffraction data. The cell parameters obtained in the present research are compared with those of the three closest phases synthesised by NAKAMURA *et al*. (1991) in Table 5. With respect to the phase with the same composition, we obtained slightly longer *a* and *c* parameters. These differences are perhaps related to the different pressures at which synthesis was operated.

Observed and computed structure factors are in Table 6. Results of the structure refinement are shown in Tables 2-4. To check the possibility of isomorphic substitution In – Ga in the octahedral site, the occupancy of that site has been varied, but the results indicated no presence of Ga. Therefore, structure refinement has been completed with occupancy 1.0 of In in the octahedral site. The structure refinement of the twinned crystal was performed without merging equivalent reflections: these were merged only before computing the Fourier difference (SHELDRICK, 1997). Therefore, three values of R1 are given for the 50kb (twinned) crystal, instead than the two values for the 20kb (untwinned) crystal. Comparison should be done between homogeneous R1 values, namely those after merging equivalent reflections. The two corresponding values for the single crystal (R1 = 0.015 for 206 reflections and 13 parameters) and for the twinned one (R1 = 0.019 for 173 reflections and 14 parameters) show that the two structures were refined with approximately the same degree of accuracy. The volume ratio for the two individuals in the twin was 0.540(3).

The displacement parameters (both isotropic and anisotropic) for O1 are larger than for the other atoms and in some respects unusual. Compounds isostructural with $YbFe_2O_4$ have been reported since the early times of their investigation to have some anomalies in the displacement parameters of one or more atoms. For $YbFe_2O_4$ it was the case of Yb and O1 (KATO *et al*., 1975). In case of $LuFe_2O_4$ and $LuFeCoO_4$ the $U_{33}$ of Lu were abnormally large (ISOBE *et al*., 1990). In the homologous series $LuFeO_3(ZnO)_m$ (ISOBE *et al*., 1994) only the member corresponding to $m = 1$ showed large displacement parameters, whereas members with $m > 1$ have all the displacement parameters in the normal range. For $LuFeZnO_4$ the adoption of the split atom model for Lu improved the *R* factor and reduced the corresponding displacement factor (CAVA *et* al., 1998; NESPOLO *et al*., 2000). In case of $InGaZnO_4$ investigated in the present research, moving the In atom off from the centre of symmetry ($z \neq 0$) either hindered the refinement (large *z*) or had no effect, resulting again in the $z = 0$ solution. Differently from $LuFeZnO_4$, $InGaZnO_4$ is thus well described in the central atom model.

In order to check whether the anomaly in the displacement parameters of O(1) may be related to some uncertainties of the structure refinement, a Charge Distribution (CD) analysis has been performed. The CD method (HOPPE *et al*., 1989) is a tool alternative to the classical Bond Valence (BV) approach (BROWN, 1981), from which it differs mainly in the usage of experimental bond distances in each coordination polyhedron, instead of empirical parameters, to compute the "bond strength" (PAULING, 1929). The ratio $q/Q$ of the computed "charge" (valence) to the formal oxidation number of the anions is a measure of the valence balance; this ratio is distributed among the cations and the resulting $q/Q$ ratio of the cations is a measure of the correctness of the structure. A $q/Q$ ratio close to 1 is expected for the cations when the structure is correctly solved; on the other hand, the $q/Q$ ratio for the anions significantly differs from 1 when an over/under bonding effect is present (Nespolo *et al*., 1999). Results of the CD calculation for the crystals refined in the present research are given in Table 7. The $q/Q$ ratios for the cations, as well as the corresponding standard deviations, show that the structure has been correctly determined. The $q/Q$ ratios for the anions show that the structure has a good valence balance. The CD calculation confirms that, differently from the case of $LuFeZnO_4$ (CAVA *et* al., 1998; NESPOLO *et al*., 2000), the structure is well described in the central atom model.

## 4. Discussion

The two crystals of $InGaZnO_4$ synthesised at different pressures have practically the same structure. This may indicate either that the high-pressure structure was essentially identical, or that both crystals assumed an identical low-pressure structure. Whatever the answer is, here we emphasise the identical shape of the trigonal bipyramids in the two crystals.

Mixed oxides of the transition elements often have $M^{2+}/M^{3+}$ cations in trigonal bipyramidal coordination. This coordination was first reported for Fe (ADELSKÖLD, 1938). Since then, several other examples have been reported, involving Al (BERTAUT and MARESCHAL, 1963; BERTAUT *et al*., 1965; GÉRARDIN *et al*., 1980), Cu (FLÜGE-KAHLER, 1963; GEBERT and KIHLBORG, 1969; POULSEN and CALVO, 1969; SHANNON and CALVO, 1973; GÉRARDIN *et al*., 1980); Mn (YAKEL *et* al., 1963; GIAQUINTA and ZUR LOYE, 1992; GREEDAN *et al*., 1995), Ga (SHANNON and PREWITT, 1968; GELLER *et al*., 1975), Zn (GOPAL and CALVO, 1973; NORD and STEFANIDIS, 1982; VELIKODNYI *et al*., 1989; ISOBE *et al*., 1994; TRINSCHEK and JANSEN, 1996; FENG *et al*., 1997), Fe (KATO *et al*., 1975; 1976; MALAMAN *et al*., 1976; GIAQUINTA *et al*., 1994; GÉRARDIN *et al*., 1980; OHKAWA *et al*., 1997). Among the structural types, magnetoplumbite (s.g. $P6_3/mmc$) (ADELSKÖLD, 1938), $YAlO_3$ (s.g. $P6_3/mmc$) (BERTAUT and MARESCHAL, 1963), $LuMnO_3$ (s.g. $P63cm$, based on a supercell of $YAlO_3$) (YAKEL *et al*., 1963), hexagonal ferrites (s.g. $P6_3/mmc$) (TOWNES *et al*., 1967), $YbFe_2O_4$ (s.g. $R\bar{3}m$) (KATO *et al*., 1975), perovskite derivatives (*e.g.* YOSHIASA *et al*., 1986; OHKAWA *et al*., 1997) are well represented. Other relevant examples can be found in the phosphates (MODARESSI *et al*., 1983; JAKEMAN and CHEETAM, 1988; BATAILLE *et* al., 1998; Boudin and Lii, 1998).

The compound object of the present study is isotypic with $YbFe_2O_4$, and is built by stacking of oxygens along *c* with a closest packing topology (KATO *et al*., 1975). In (substituting $Yb^{3+}$) is located between two oxygen planes, in octahedral coordination, whereas $Zn^{2+}/Ga^{3+}$ (substituting $Fe^{2+}/Fe^{3+}$) are almost on the same plane as the oxygens, in trigonal bipyramidal coordination. The isomorphic substitution of In for Yb is known since the early stages of the research on these materials (SCHMIDT-DUMONT and KASPER, 1965). Mixed oxides of the transition elements with this kind of structure can be broadly classified into two types. Type-I has the cation in the trigonal bipyramid closer to the basal plane and the bipyramid has a more regular shape (the three basal M-O bonds are shorter than the two apical ones). Type-II has the cation in the trigonal bipyramid significantly outside the basal plane and the bipyramid is more distorted (one of the apical bonds is shorter and the other is longer that the three basal ones). $InGaZnO_4$, the object of the present research, belongs to type-I, which includes also $InFe^{3+}{}_{0.5}Fe^{2+}{}_{1.25}Si_{0.25}O_4$ (GÉRARDIN *et al*., 1980) and one of the two examples of $Yb_2Fe_3O_7$ reported to date (MALAMAN *et al*., 1976). Type-II is more extensively represented and includes $YbFe_2O_4$ (KATO *et al*., 1975), $Yb_{0.5}Eu_{0.5}Fe_2O_4$ (MALAMAN *et al*., 1975), $InCuAlO_4$ (GÉRARDIN *et al*., 1980), $LuFe_2O_4$ (ISOBE *et al*., 1990), $LuCoFeO_4$ (ISOBE *et al*., 1990), $LuFeZnO_4$ (ISOBE *et al*., 1994; CAVA *et al*., 1998; NESPOLO *et al*., 2000) and the other example of $Yb_2Fe_3O_7$ (KATO *et al*., 1976). In Table 8 the results of the CD analysis are given for these compounds. The two representatives of type-I are well balanced from a Charge Distribution point of view, as in the case of $InGaZnO_4$. On the other hand, for all the structures belonging to type-II the $q/Q$ ratio for the cations significantly differs from 1. In the case of $LuFeZnO_4$ recent refinements show that the Lu atom is displaced from the centre of symmetry (split atom model) (CAVA *et al*., 1998; NESPOLO *et al*., 2000). Moreover, for higher members of the homologous series based on the same structure, the existence of a cation ordering scheme in which the trivalent cation avoids the site next to the octahedron has been proposed too (NESPOLO *et al.*, 2000).

The reasons for the existence of two different types of trigonal bipyramids are not clear. The two examples of $Yb_2Fe_3O_7$ have the same chemistry and were synthesised at similar values of temperature and pressure, but with different synthesis methods, which may thus have influenced the shape of the trigonal bipyramid. On the other hand, both crystals of $InGaZnO_4$ reported here belong to type-I, notwithstanding the different pressure and apparatus used for the synthesis. In this case, the chemical composition may have played a dominant role. Further researches are needed to clarify the factors leading to the appearance of either type-I or type-II trigonal bipyramids. Moreover, the effects of the distortions typical of type-II bipyramids on the cation position and distribution have not been completely worked out yet.

Table 1: Experimental data for $InGaZnO_4$

*Crystal data*

$InGaZnO_4$

$M_r$ = 313.91

Space group: $R\bar{3}m$

Mo $K\alpha$ radiation ($\lambda$ = 0.71073Å)

T = 293(2) K

| | 20kb (single crystal) | 50kb (twin) |
|---|---|---|
| Number of reflections for cell parameters | 18 (27.5° ≤ ϑ ≤ 32.5°) | 17 (27.5° ≤ ϑ ≤ 32.5°) |
| a | 3.2990(2) Å | 3.3051(2) Å |
| c | 26.101(3) Å | 26.1029(19) Å |
| V | 246.01(3) Å$^3$ | 246.94(3) Å$^3$ |
| Z | 3 | 3 |
| $D_x$ | 6.357 Mg/m$^3$ | 6.333 Mg/m$^3$ |
| μ | 22.218 mm$^{-1}$ | 22.135 mm$^{-1}$ |
| Range for data collection | 2.34° ≤ ϑ ≤ 39.71° | 2.34° ≤ ϑ ≤ 34.87° |
| Crystal shape | Prism | Prism |
| Dimensions | 70×70×30 μm | 80×70×20 μm |
| Colour | Colourless, transparent | Colourless, transparent |

*Data collection*

Enraf-Nonius CAD-4 diffractometer

ω/2ϑ scan

Absorption correction: Analytical

| | 20kb (single crystal) | 50kb (twin) |
|---|---|---|
| $T_{min}$ | 0.2309 (0, 0, -3) | 0.2604 (0, 0, -2) |
| $T_{max}$ | 0.5236 (1, 1, 0) | 0.6384 (0, 1, -1) |
| $T_{mean}$ | 0.4413 | 0.5351 |
| Reflections collected | 470 | 590 |
| Reflections unique | 236 | 590 (179) |
| Observed reflections [I>2σ(I)] | 206 | 525 (179) |
| Standard reflections | 3 | 3 |
| Intensity monitoring interval | 4 hours | 4 hours |
| Intensity variation | -0.8% | 0.6% |

| | | |
|---|---|---|
| $R_{int}$ (on $F^2$) | 0.0164 | ------ |
| Completeness | 100.0 % (ϑ = 39.71º) | 100.0 % (ϑ = 34.87) |
| Limiting indices | 0 ≤ h ≤ 5, | -4 ≤ h ≤ 4, |
| | 0 ≤ k ≤ 5, | -4 ≤ k ≤ 4, |
| | -46 ≤ l ≤ 46 | -42 ≤ l ≤ 42 |

*Refinement*
Refinement method: Full-matrix least-squares on $F^2$

| | 20kb (single crystal) | 50kb (twin) |
|---|---|---|
| Reflections | 206 | 590 |
| Parameters | 13 | 14 |
| GooF (S) (on $F^2$) | 1.284 | 1.200 |
| R1 [I>2σ(I)] | 0.015 | 0.0320 |
| wR2 [I>2σ(I)] | 0.042 | 0.108 |
| R1 (all data) | 0.019 | 0.037 |
| wR2 (all data) | 0.043 | 0.112 |
| R1 (after merging equivalent reflections) | ---- | 0.019 |
| Extinction coefficient | 0.0060(6) | 0.059(5) |
| $(\Delta/\sigma)_{max}$ | 0.000 | 0.000 |
| $\Delta\rho_{max}$ | 0.698 e Å$^{-3}$ | 1.837 e Å$^{-3}$ |
| $\Delta\rho_{min}$ | -1.891 e Å$^{-3}$ | -1.474 e Å$^{-3}$ |

Table 2: Atomic coordinates and equivalent isotropic displacement parameters (Å$^2$) for $InGaZnO_4$. *U*(eq) is defined as one third of the trace of the orthogonalized $U_{ij}$ tensor.

| | 20kb (single crystal) | | | 50kb (twin) | | |
|---|---|---|---|---|---|---|
| | x = y | z | U(eq) | x = y | z | U(eq) |
| In | 0 | 0 | 0.008(1) | 0 | 0 | 0.008(1) |
| Ga/Zn | 0 | 0.2171(1) | 0.007(1) | 0 | 0.2169(1) | 0.007(1) |
| O(1) | 0 | 0.1282(1) | 0.014(1) | 0 | 0.1282(1) | 0.015(1) |
| O(2) | 0 | 0.2928(1) | 0.008(1) | 0 | 0.2928(1) | 0.009(1) |

Table 3: Anisotropic displacement parameters (Å$^2$) for $InGaZnO_4$. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11} + ... + 2hka^*b^*U_{12}]$

| 20kb | U11 | U22 | U33 | U23 | U13 | U12 |
|---|---|---|---|---|---|---|
| In | 0.006(1) | 0.006(1) | 0.013(1) | 0 | 0 | 0.003(1) |
| Ga/Zn | 0.006(1) | 0.006(1) | 0.010(1) | 0 | 0 | 0.003(1) |
| O(1) | 0.013(1) | 0.013(1) | 0.015(1) | 0 | 0 | 0.007(1) |
| O(2) | 0.008(1) | 0.008(1) | 0.009(1) | 0 | 0 | 0.004(1) |
| 50kb | U11 | U22 | U33 | U23 | U13 | U12 |
| In | 0.005(1) | 0.005(1) | 0.013(1) | 0 | 0 | 0.003(1) |
| Ga/Zn | 0.006(1) | 0.006(1) | 0.010(1) | 0 | 0 | 0.003(1) |
| O(1) | 0.013(1) | 0.013(1) | 0.020(2) | 0 | 0 | 0.006(1) |
| O(2) | 0.007(1) | 0.007(1) | 0.011(1) | 0 | 0 | 0.004(1) |

Table 4: Bond lengths (Å) and angles (º) for $InGaZnO_4$.

| | ***20kb (single crystal)*** | ***50kb (twin)*** |
|---|---|---|
| In-O(2) | 2.1784(15) (×4) | 2.181(2) (×4) |
| | 2.1785(15) (×2) | 2.182(2) (×2) |
| M1-O(1) | 1.9302(6) (×3) | 1.9329(9) (×3) |
| M1-O(2) | 1.977(3) | 1.982(4) |
| M1-O(1) | 2.319(4) | 2.315(6) |
| O(2)-In-O(2) | 98.43(9) (×6) | 98.49(13) (×6) |
| O(2)-In-O(2) | 81.57(9) (×6) | 81.51(13) (×6) |
| O(1)-M1-O(1) | 117.43(6) (×3) | 117.51(9) (×3) |
| O(1)-M1-O(2) | 99.32(11) (×3) | 99.17(16) (×3) |
| O(1)-M1-O(1) | 80.68(11) (×3) | 80.83(16) (×3) |

Table 5: Cell parameters as a function of composition

| In:Ga:Zn | *a* | *c* |
|---|---|---|
| †0.92:1.08:1 | 3.286(1) | 26.01(1) |
| †1:1:1 | 3.296(1) | 26.02(1) |
| ‡[a]1:1:1 | 3.2990(2) | 26.101(3) |
| ‡[b]1:1:1 | 3.3051(2) | 26.1029(19) |
| †1.2:0.8:1 | 3.316(1) | 26.26(1) |

†Nakamura *et al*. (1991); ‡Present study ([a]single crystal; [b]twin)

Table 6: Observed and computed structure factors $InGaZnO_4$ synthesised at 20kb (single crystal) and at 50kb (twin).

20 kb (single crystal)

| *h* | *k* | *l* | 10Fo | 10Fc | $10\sigma$ | *h* | *k* | *l* | 10Fo | 10Fc | $10\sigma$ | *h* | *k* | *l* | 10Fo | 10Fc | $10\sigma$ |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| -1 | 2 | 0 | 2581 | 2633 | 4 | -2 | 3 | 7 | 63 | 68 | 3 | -1 | 3 | 14 | 1423 | 1435 | 15 |
| 0 | 3 | 0 | 1613 | 1624 | 4 | -4 | 4 | 7 | 91 | 79 | 6 | -3 | 4 | 14 | 1063 | 1070 | 8 |
| -2 | 4 | 0 | 1331 | 1375 | 6 | -1 | 4 | 7 | 73 | 74 | 4 | 0 | 4 | 14 | 947 | 951 | 13 |
| -1 | 5 | 0 | 950 | 951 | 8 | -3 | 5 | 7 | 76 | 82 | 14 | -2 | 5 | 14 | 859 | 856 | 4 |
| -1 | 1 | 1 | 1641 | 1655 | 7 | 0 | 5 | 7 | 94 | 80 | 7 | 0 | 0 | 15 | 918 | 908 | 3 |
| 0 | 2 | 1 | 1159 | 1181 | 4 | 0 | 1 | 8 | 1064 | 1078 | 11 | -1 | 2 | 15 | 691 | 706 | 2 |
| -2 | 3 | 1 | 934 | 940 | 3 | -2 | 2 | 8 | 783 | 793 | 2 | -3 | 3 | 15 | 505 | 507 | 6 |
| -4 | 4 | 1 | 602 | 607 | 6 | -1 | 3 | 8 | 638 | 643 | 6 | 0 | 3 | 15 | 506 | 507 | 9 |
| -1 | 4 | 1 | 675 | 685 | 2 | -3 | 4 | 8 | 484 | 482 | 1 | -2 | 4 | 15 | 448 | 449 | 1 |
| -3 | 5 | 1 | 539 | 546 | 2 | 0 | 4 | 8 | 433 | 432 | 4 | -4 | 5 | 15 | 341 | 336 | 3 |
| 0 | 5 | 1 | 459 | 452 | 5 | -5 | 5 | 8 | 346 | 328 | 12 | -1 | 5 | 15 | 341 | 336 | 4 |
| 0 | 1 | 2 | 579 | 518 | 6 | -2 | 5 | 8 | 401 | 392 | 5 | -1 | 1 | 16 | 173 | 151 | 3 |
| -2 | 2 | 2 | 342 | 325 | 1 | 0 | 0 | 9 | 2681 | 2601 | 9 | 0 | 2 | 16 | 131 | 120 | 3 |
| -1 | 3 | 2 | 234 | 224 | 2 | -1 | 2 | 9 | 1891 | 1908 | 17 | -2 | 3 | 16 | 95 | 89 | 2 |
| -3 | 4 | 2 | 115 | 118 | 2 | -3 | 3 | 9 | 1271 | 1273 | 11 | -4 | 4 | 16 | 32 | 41 | 32 |
| 0 | 4 | 2 | 83 | 88 | 4 | 0 | 3 | 9 | 1267 | 1273 | 25 | -1 | 4 | 16 | 34 | 50 | 19 |
| -5 | 5 | 2 | 32 | 45 | 31 | -2 | 4 | 9 | 1095 | 1095 | 3 | -3 | 5 | 16 | 38 | 37 | 20 |
| -2 | 5 | 2 | 63 | 67 | 26 | -4 | 5 | 9 | 776 | 773 | 3 | 0 | 1 | 17 | 681 | 666 | 9 |
| 0 | 0 | 3 | 351 | 351 | 3 | -1 | 5 | 9 | 776 | 773 | 9 | -2 | 2 | 17 | 522 | 522 | 3 |
| -1 | 2 | 3 | 225 | 240 | 1 | -1 | 1 | 10 | 1810 | 1829 | 20 | -1 | 3 | 17 | 441 | 443 | 4 |
| -3 | 3 | 3 | 185 | 188 | 1 | 0 | 2 | 10 | 1370 | 1378 | 11 | -3 | 4 | 17 | 360 | 355 | 2 |
| 0 | 3 | 3 | 187 | 188 | 1 | -2 | 3 | 10 | 1108 | 1120 | 3 | 0 | 4 | 17 | 333 | 325 | 6 |
| -2 | 4 | 3 | 177 | 179 | 2 | -4 | 4 | 10 | 741 | 738 | 11 | -2 | 5 | 17 | 308 | 299 | 2 |
| -4 | 5 | 3 | 160 | 161 | 3 | -1 | 4 | 10 | 816 | 830 | 8 | 0 | 0 | 18 | 1716 | 1649 | 19 |
| -1 | 5 | 3 | 174 | 161 | 5 | -3 | 5 | 10 | 662 | 665 | 2 | -1 | 2 | 18 | 1329 | 1329 | 4 |

| *h* | *k* | *l* | 10Fo | 10Fc | 10σ | *h* | *k* | *l* | 10Fo | 10Fc | 10σ | *h* | *k* | *l* | 10Fo | 10Fc | 10σ |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| -1 | 1 | 4 | 1966 | 1994 | 9 | 0 | 5 | 10 | 563 | 554 | 8 | -3 | 3 | 18 | 963 | 962 | 4 |
| 0 | 2 | 4 | 1494 | 1515 | 17 | 0 | 1 | 11 | 185 | 153 | 3 | 0 | 3 | 18 | 956 | 962 | 18 |
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| 0 | 5 | 4 | 602 | 598 | 5 | -5 | 5 | 11 | 57 | 50 | 28 | 0 | 2 | 19 | 960 | 958 | 3 |
| 0 | 1 | 5 | 1822 | 1875 | 36 | -2 | 5 | 11 | 58 | 45 | 58 | -2 | 3 | 19 | 818 | 826 | 6 |
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| -1 | 3 | 5 | 1236 | 1237 | 4 | -1 | 2 | 12 | 354 | 349 | 3 | -1 | 4 | 19 | 644 | 647 | 12 |
| -3 | 4 | 5 | 905 | 914 | 4 | -3 | 3 | 12 | 161 | 161 | 2 | -3 | 5 | 19 | 533 | 532 | 2 |
| 0 | 4 | 5 | 799 | 808 | 10 | 0 | 3 | 12 | 166 | 161 | 2 | 0 | 1 | 20 | 162 | 169 | 1 |
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| -2 | 5 | 5 | 720 | 725 | 2 | -4 | 5 | 12 | 75 | 50 | 5 | -1 | 3 | 20 | 154 | 156 | 2 |
| 0 | 0 | 6 | 863 | 830 | 2 | -1 | 5 | 12 | 35 | 50 | 23 | -3 | 4 | 20 | 151 | 148 | 5 |
| -1 | 2 | 6 | 546 | 566 | 1 | -1 | 1 | 13 | 1425 | 1418 | 16 | 0 | 4 | 20 | 150 | 143 | 5 |
| -3 | 3 | 6 | 381 | 388 | 4 | 0 | 2 | 13 | 1100 | 1122 | 15 | -2 | 5 | 20 | 144 | 137 | 3 |
| 0 | 3 | 6 | 384 | 388 | 4 | -2 | 3 | 13 | 926 | 934 | 5 | 0 | 0 | 21 | 146 | 130 | 2 |
| -2 | 4 | 6 | 341 | 346 | 1 | -4 | 4 | 13 | 633 | 635 | 6 | -1 | 2 | 21 | 99 | 93 | 4 |
| -4 | 5 | 6 | 271 | 267 | 5 | -1 | 4 | 13 | 707 | 709 | 7 | -3 | 3 | 21 | 15 | 45 | 14 |
| -1 | 5 | 6 | 269 | 267 | 2 | -3 | 5 | 13 | 577 | 575 | 2 | 0 | 3 | 21 | 17 | 45 | 16 |
| -1 | 1 | 7 | 230 | 238 | 1 | 0 | 1 | 14 | 2259 | 2248 | 4 | -2 | 4 | 21 | 12 | 36 | 11 |
| 0 | 2 | 7 | 75 | 93 | 2 | -2 | 2 | 14 | 1737 | 1744 | 5 | -4 | 5 | 21 | 39 | 36 | 15 |

| *h* | *k* | *l* | 10Fo | 10Fc | 10σ | *h* | *k* | *l* | 10Fo | 10Fc | 10s |
|---|---|---|---|---|---|---|---|---|---|---|---|
| -1 | 5 | 21 | 58 | 36 | 9 | -2 | 4 | 30 | 34 | 36 | 33 |
| -1 | 1 | 22 | 735 | 716 | 5 | -1 | 1 | 31 | 521 | 523 | 13 |
| 0 | 2 | 22 | 610 | 608 | 6 | 0 | 2 | 31 | 465 | 463 | 8 |
| -2 | 3 | 22 | 522 | 531 | 1 | -2 | 3 | 31 | 421 | 417 | 2 |
| -4 | 4 | 22 | 395 | 387 | 4 | -1 | 4 | 31 | 352 | 346 | 2 |
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| -3 | 5 | 22 | 369 | 354 | 3 | -2 | 2 | 32 | 819 | 822 | 24 |
| 0 | 1 | 23 | 1579 | 1537 | 14 | -1 | 3 | 32 | 737 | 733 | 2 |
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| -2 | 5 | 23 | 714 | 706 | 6 | 0 | 3 | 33 | 495 | 480 | 4 |
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| -3 | 3 | 24 | 675 | 676 | 2 | 0 | 2 | 34 | 83 | 75 | 5 |
| 0 | 3 | 24 | 678 | 676 | 13 | -2 | 3 | 34 | 85 | 76 | 5 |
| -2 | 4 | 24 | 610 | 606 | 5 | 0 | 1 | 35 | 58 | 72 | 24 |
| -1 | 1 | 25 | 127 | 121 | 3 | -2 | 2 | 35 | 32 | 53 | 23 |
| 0 | 2 | 25 | 92 | 87 | 5 | -1 | 3 | 35 | 0 | 41 | 1 |
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| -4 | 4 | 25 | 0 | 32 | 1 | -1 | 2 | 36 | 396 | 399 | 7 |
| -1 | 4 | 25 | 26 | 37 | 26 | -3 | 3 | 36 | 331 | 333 | 16 |
| -3 | 5 | 25 | 38 | 29 | 16 | 0 | 3 | 36 | 339 | 333 | 8 |
| 0 | 1 | 26 | 0 | 26 | 1 | -1 | 1 | 37 | 781 | 798 | 18 |

| *h* | *k* | *l* | 10Fo | 10Fc | 10σ | *h* | *k* | *l* | 10Fo | 10Fc | 10s |
|---|---|---|---|---|---|---|---|---|---|---|---|
| -2 | 2 | 26 | 70 | 16 | 37 | 0 | 2 | 37 | 697 | 716 | 2 |
| -1 | 3 | 26 | 0 | 25 | 1 | -2 | 3 | 37 | 638 | 649 | 10 |
| -3 | 4 | 26 | 70 | 39 | 22 | 0 | 1 | 38 | 411 | 413 | 4 |
| 0 | 4 | 26 | 17 | 41 | 16 | -2 | 2 | 38 | 383 | 375 | 2 |
| 0 | 0 | 27 | 1034 | 1022 | 3 | -1 | 3 | 38 | 351 | 342 | 5 |
| -1 | 2 | 27 | 891 | 875 | 4 | 0 | 0 | 39 | 84 | 87 | 12 |
| -3 | 3 | 27 | 687 | 685 | 2 | -1 | 2 | 39 | 58 | 73 | 7 |
| 0 | 3 | 27 | 696 | 685 | 7 | -1 | 1 | 40 | 161 | 168 | 3 |
| -2 | 4 | 27 | 617 | 619 | 4 | 0 | 2 | 40 | 162 | 155 | 8 |
| -1 | 1 | 28 | 1036 | 1040 | 26 | -2 | 3 | 40 | 147 | 144 | 13 |
| 0 | 2 | 28 | 909 | 902 | 6 | 0 | 1 | 41 | 618 | 636 | 2 |
| -2 | 3 | 28 | 801 | 797 | 4 | -2 | 2 | 41 | 566 | 578 | 11 |

| | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| -4 | 4 | 28 | 598 | 591 | 9 | 0 | 0 | 42 | 489 | 494 | 4 |
| -1 | 4 | 28 | 660 | 647 | 8 | -1 | 2 | 42 | 448 | 450 | 7 |
| 0 | 1 | 29 | 201 | 198 | 2 | -1 | 1 | 43 | 43 | 13 | 15 |
| -2 | 2 | 29 | 187 | 185 | 2 | 0 | 2 | 43 | 0 | 16 | 1 |
| -1 | 3 | 29 | 176 | 174 | 3 | 0 | 1 | 44 | 38 | 39 | 37 |
| -3 | 4 | 29 | 163 | 153 | 9 | 0 | 0 | 45 | 309 | 311 | 3 |
| 0 | 4 | 29 | 163 | 143 | 4 | -1 | 1 | 46 | 466 | 489 | 8 |
| 0 | 0 | 30 | 84 | 83 | 3 | | | | | | |
| -1 | 2 | 30 | 63 | 61 | 5 | | | | | | |
| -3 | 3 | 30 | 33 | 40 | 32 | | | | | | |
| 0 | 3 | 30 | 14 | 40 | 13 | | | | | | |

50 kb (twin)

| *h* | *k* | *l* | 10Fo | 10Fc | 10σ | *h* | *k* | *l* | 10Fo | 10Fc | 10σ | *h* | *k* | *l* | 10Fo | 10Fc | 10σ |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 0 | 0-42 | 495 | 491 | 2 | 0 | 1-22 | 438 | 467 | 2 | 2 | 1-11 | 0 | 23 | 1 | 0 | -4 | -2 |
| 0 | -1-41 | 498 | 441 | 2 | 0 | -2-22 | 424 | 431 | 2 | -3 | -1-11 | 32 | 27 | 31 | 0 | 4 | -1 |
| 1 | 0-41 | 324 | 407 | 3 | 1 | 2-22 | 355 | 348 | 2 | 4 | 0-11 | 27 | 33 | 26 | -1 | -3 | -1 |
| -1 | 0-40 | 0 | 106 | 1 | -1 | -3-22 | 293 | 304 | 3 | -2 | 0-11 | 57 | 52 | 3 | 2 | 3 | -1 |
| 0 | 1-40 | 79 | 98 | 6 | 0 | 3-21 | 26 | 43 | 25 | 1 | 0-11 | 116 | 95 | 1 | -3 | -2 | -1 |
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| 0 | -2-37 | 574 | 512 | 3 | 1 | 0-20 | 117 | 128 | 2 | -2 | -1-10 | 801 | 830 | 4 | 3 | 1 | -1 |
| 0 | 1-37 | 444 | 526 | 2 | -2 | 0-20 | 124 | 131 | 2 | 0 | 1-10 | 1246 | 1247 | 4 | -4 | 0 | -1 |
| 2 | 0-37 | 403 | 472 | 3 | 4 | 0-20 | 111 | 108 | 4 | 0 | -2-10 | 1006 | 1020 | 5 | 2 | 0 | -1 |
| -1 | 0-37 | 646 | 570 | 3 | -3 | -1-20 | 125 | 121 | 3 | 1 | 2-10 | 758 | 766 | 3 | -1 | 0 | -1 |
| 0 | 0-36 | 411 | 423 | 2 | 2 | 1-20 | 123 | 117 | 3 | -3 | -2-10 | 495 | 496 | 2 | 3 | 0 | 0 |
| 1 | 1-36 | 381 | 381 | 3 | 0 | -1-20 | 139 | 139 | 2 | 2 | 3-10 | 468 | 458 | 2 | 4 | 1 | 0 |
| 0 | 2-35 | 0 | 36 | 1 | 0 | 2-20 | 116 | 121 | 2 | -1 | -3-10 | 606 | 617 | 2 | 1 | 1 | 0 |
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| 0 | -2-34 | 82 | 63 | 6 | 0 | 1-19 | 740 | 778 | 3 | -2 | 0 | -8 | 559 | 577 | 2 | -1 | -2 |
| 1 | 2-34 | 62 | 59 | 9 | -2 | -1-19 | 588 | 612 | 2 | 4 | 0 | -8 | 298 | 294 | 2 | 0 | 2 |
| 1 | 1-33 | 621 | 593 | 3 | 3 | 1-19 | 445 | 442 | 2 | -3 | -1 | -8 | 345 | 355 | 2 | 0 | -1 |
| *h* | *k* | *l* | 10Fo | 10Fc | 10σ | *h* | *k* | *l* | 10Fo | 10Fc | 10σ | *h* | *k* | *l* | 10Fo | 10Fc | 10σ |
| 0 | 0-33 | 669 | 670 | 3 | -4 | 0-19 | 422 | 433 | 2 | 2 | 1 | -8 | 429 | 433 | 2 | 2 | 1 |
| 1 | 0-32 | 511 | 614 | 2 | 2 | 0-19 | 639 | 656 | 3 | 0 | -1 | -8 | 775 | 782 | 3 | -3 | -1 |
| -2 | 0-32 | 659 | 585 | 3 | -1 | 0-19 | 919 | 844 | 4 | 0 | 2 | -8 | 536 | 533 | 2 | 4 | 0 |
| 2 | 1-32 | 467 | 482 | 3 | 0 | 0-18 | 1683 | 1628 | 7 | -1 | -2 | -8 | 461 | 470 | 2 | -2 | 0 |
| 0 | -1-32 | 745 | 666 | 3 | 3 | 0-18 | 942 | 952 | 4 | 3 | 2 | -8 | 279 | 268 | 2 | 1 | 0 |
| 0 | 2-32 | 528 | 540 | 2 | 1 | 1-18 | 1336 | 1313 | 6 | -2 | -3 | -8 | 292 | 290 | 2 | -1 | 0 |
| -1 | -2-32 | 566 | 522 | 3 | 2 | 2-18 | 836 | 839 | 4 | 1 | 3 | -8 | 335 | 327 | 2 | 2 | 0 |
| 1 | 2-31 | 269 | 265 | 3 | 0 | 3-18 | 965 | 952 | 4 | 0 | -4 | -8 | 306 | 319 | 2 | -4 | 0 |
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| 0 | 1-31 | 285 | 328 | 2 | 1 | 3-17 | 242 | 238 | 2 | -1 | -3 | -7 | 63 | 61 | 4 | -2 | -1 |
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| 0 | 0-30 | 79 | 86 | 4 | 2 | 1-17 | 294 | 294 | 2 | 0 | -2 | -7 | 66 | 72 | 2 | -3 | -2 |
| 3 | 0-30 | 38 | 38 | 37 | -3 | -1-17 | 254 | 258 | 2 | 0 | 1 | -7 | 158 | 161 | 1 | 2 | 3 |
| 1 | 1-30 | 60 | 62 | 7 | 4 | 0-17 | 230 | 220 | 3 | -2 | -1 | -7 | 48 | 55 | 4 | -1 | -3 |
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| | | | | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| -2 | 0-29 | 165 | 148 | 3 | 3 | 1-16 | 0 | 30 | 1 | 3 | 0 | -6 | 401 | 399 | 1 | 4 | 1 |
| 1 | 0-29 | 117 | 147 | 3 | -2 | -1-16 | 96 | 61 | 3 | 1 | 1 | -6 | 561 | 576 | 2 | 3 | 0 |
| -1 | 0-28 | 850 | 761 | 4 | 0 | 1-16 | 108 | 101 | 2 | 2 | 2 | -6 | 358 | 357 | 2 | 0 | 0 |
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| -2 | -1-28 | 601 | 583 | 3 | 1 | 2-16 | 69 | 56 | 3 | 0 | -4 | -5 | 580 | 595 | 2 | -2 | 0 |
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| 0 | -2-28 | 700 | 660 | 3 | 0 | 4-16 | 37 | 25 | 12 | -2 | -3 | -5 | 527 | 534 | 2 | -3 | -1 |
| *h* | *k* | *l* | 10Fo | 10Fc | 10σ | *h* | *k* | *l* | 10Fo | 10Fc | 10σ | *h* | *k* | *l* | 10Fo | 10Fc | 10σ |
| 1 | 2-28 | 535 | 538 | 2 | 0 | 3-15 | 511 | 518 | 2 | 3 | 2 | -5 | 497 | 492 | 2 | 2 | 1 |
| 0 | 3-27 | 691 | 669 | 3 | 2 | 2-15 | 458 | 460 | 2 | -1 | -2 | -5 | 911 | 911 | 4 | 0 | -1 |
| 2 | 2-27 | 615 | 605 | 3 | 1 | 1-15 | 704 | 717 | 3 | 0 | 2 | -5 | 1017 | 1016 | 4 | 0 | 2 |
| 1 | 1-27 | 871 | 852 | 4 | 3 | 0-15 | 516 | 518 | 2 | 0 | -1 | -5 | 1359 | 1380 | 4 | -1 | -2 |
| 3 | 0-27 | 692 | 669 | 3 | 0 | 0-15 | 930 | 920 | 4 | 2 | 1 | -5 | 829 | 840 | 4 | 3 | 2 |
| 0 | 0-27 | 989 | 994 | 4 | 1 | 0-14 | 1354 | 1520 | 6 | -3 | -1 | -5 | 660 | 673 | 3 | -2 | -3 |
| 1 | 0-26 | 0 | 23 | 1 | -2 | 0-14 | 1216 | 1280 | 6 | 4 | 0 | -5 | 541 | 549 | 2 | 1 | 3 |
| -2 | 0-26 | 0 | 12 | 1 | 4 | 0-14 | 646 | 646 | 3 | -2 | 0 | -5 | 1085 | 1101 | 5 | 0 | -4 |
| -3 | -1-26 | 36 | 27 | 36 | -3 | -1-14 | 765 | 787 | 3 | 1 | 0 | -5 | 1211 | 1274 | 4 | 0 | 4 |
| 2 | 1-26 | 16 | 15 | 16 | 2 | 1-14 | 956 | 972 | 4 | -1 | 0 | -4 | 1415 | 1463 | 4 | -1 | -3 |
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| 0 | 2-26 | 0 | 11 | 1 | 0 | 2-14 | 1203 | 1181 | 5 | -4 | 0 | -4 | 581 | 591 | 2 | -3 | -2 |
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| 0 | -2-25 | 0 | 56 | 1 | -1 | -3-13 | 499 | 518 | 2 | 1 | 2 | -4 | 829 | 836 | 4 | 3 | 1 |
| 0 | 1-25 | 76 | 75 | 4 | 2 | 3-13 | 400 | 389 | 2 | -3 | -2 | -4 | 527 | 532 | 2 | -4 | 0 |
| -2 | -1-25 | 33 | 38 | 13 | -3 | -2-13 | 414 | 422 | 2 | 2 | 3 | -4 | 490 | 491 | 2 | 2 | 0 |
| 3 | 1-25 | 0 | 21 | 1 | 1 | 2-13 | 630 | 628 | 3 | -1 | -3 | -4 | 653 | 667 | 3 | -1 | 0 |
| 2 | 0-25 | 38 | 52 | 38 | 0 | -2-13 | 785 | 815 | 3 | 0 | 4 | -4 | 547 | 545 | 2 | 0 | 0 |
| -1 | 0-25 | 96 | 81 | 3 | 0 | 1-13 | 941 | 949 | 4 | 1 | 4 | -3 | 178 | 172 | 3 | 3 | 0 |
| 0 | 0-24 | 1093 | 1078 | 5 | -2 | -1-13 | 662 | 680 | 3 | 0 | 3 | -3 | 186 | 194 | 2 | 1 | 1 |
| 3 | 0-24 | 688 | 680 | 3 | 3 | 1-13 | 472 | 478 | 2 | 2 | 2 | -3 | 184 | 187 | 2 | 2 | 2 |
| 1 | 1-24 | 906 | 893 | 4 | -4 | 0-13 | 446 | 465 | 2 | 1 | 1 | -3 | 224 | 241 | 1 | 0 | 3 |
| 2 | 2-24 | 624 | 610 | 3 | 2 | 0-13 | 730 | 752 | 3 | 4 | 1 | -3 | 184 | 172 | 3 | 0 | -4 |
| 0 | 3-24 | 683 | 680 | 3 | -1 | 0-13 | 1022 | 1029 | 5 | 3 | 0 | -3 | 189 | 194 | 2 | 1 | 3 |
| *h* | *k* | *l* | 10Fo | 10Fc | 10σ | *h* | *k* | *l* | 10Fo | 10Fc | 10σ | *h* | *k* | *l* | 10Fo | 10Fc | 10σ |
| 1 | 3-23 | 594 | 577 | 3 | 0 | 0-12 | 666 | 606 | 3 | 0 | 0 | -3 | 349 | 347 | 1 | -2 | -3 |
| -1 | -2-23 | 782 | 798 | 3 | 3 | 0-12 | 154 | 160 | 2 | 1 | 0 | -2 | 378 | 350 | 1 | 3 | 2 |
| 0 | 2-23 | 851 | 856 | 4 | 1 | 1-12 | 348 | 351 | 1 | -2 | 0 | -2 | 235 | 235 | 1 | -1 | -2 |
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| 2 | 1-23 | 733 | 736 | 3 | 0 | 3-12 | 159 | 160 | 2 | -3 | -1 | -2 | 81 | 81 | 3 | 0 | -1 |
| -3 | -1-23 | 617 | 625 | 3 | 0 | -4-11 | 48 | 36 | 7 | 2 | 1 | -2 | 154 | 148 | 1 | 2 | 1 |
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| 2 | 0-22 | 380 | 397 | 2 | -1 | -2-11 | 45 | 25 | 4 | 3 | 2 | -2 | 38 | 39 | 38 | 1 | 0 |
| 3 | 1-22 | 285 | 280 | 3 | 0 | 2-11 | 73 | 48 | 2 | -2 | -3 | -2 | 31 | 43 | 30 | -1 | 0 |
| -2 | -1-22 | 371 | 377 | 2 | 0 | -1-11 | 115 | 102 | 1 | 1 | 3 | -2 | 72 | 74 | 4 | 2 | 0 |
| 1 | 1 | 18 | 1344 | 1313 | 6 | 1 | 1 | 21 | 96 | 94 | 3 | -1 | -2 | 25 | 38 | 38 | 37 |
| 2 | 2 | 18 | 852 | 839 | 4 | 3 | 0 | 21 | 34 | 43 | 11 | 0 | 2 | 25 | 52 | 52 | 6 |
| 0 | 3 | 18 | 964 | 952 | 4 | 0 | 0 | 21 | 145 | 135 | 2 | 0 | -1 | 25 | 82 | 81 | 3 |
| 0 | -4 | 19 | 441 | 433 | 2 | 1 | 0 | 22 | 516 | 467 | 2 | 2 | 1 | 25 | 0 | 35 | 1 |
| 1 | 3 | 19 | 441 | 442 | 2 | -2 | 0 | 22 | 428 | 431 | 2 | -3 | -1 | 25 | 31 | 22 | 22 |
| -1 | -2 | 19 | 615 | 612 | 3 | -3 | -1 | 22 | 305 | 304 | 2 | -2 | 0 | 25 | 58 | 56 | 5 |
| 0 | 2 | 19 | 657 | 656 | 3 | 2 | 1 | 22 | 343 | 348 | 2 | 1 | 0 | 25 | 122 | 75 | 2 |
| 0 | -1 | 19 | 816 | 844 | 4 | 0 | -1 | 22 | 481 | 506 | 2 | -1 | 0 | 26 | 0 | 25 | 1 |
| 2 | 1 | 19 | 554 | 565 | 2 | 0 | 2 | 22 | 406 | 397 | 2 | 2 | 0 | 26 | 15 | 11 | 15 |
| -3 | -1 | 19 | 487 | 479 | 2 | -1 | -2 | 22 | 379 | 377 | 2 | 3 | 1 | 26 | 39 | 25 | 13 |
| 4 | 0 | 19 | 395 | 399 | 2 | 1 | 3 | 22 | 282 | 280 | 2 | -2 | -1 | 26 | 0 | 16 | 1 |
| -2 | 0 | 19 | 720 | 711 | 3 | -1 | -3 | 23 | 630 | 625 | 3 | 0 | 1 | 26 | 0 | 23 | 1 |
| 1 | 0 | 19 | 818 | 778 | 4 | 1 | 2 | 23 | 738 | 736 | 3 | 0 | -2 | 26 | 52 | 12 | 6 |
| -1 | 0 | 20 | 127 | 139 | 2 | 0 | -2 | 23 | 922 | 927 | 4 | 1 | 2 | 26 | 35 | 15 | 35 |
| 2 | 0 | 20 | 117 | 121 | 2 | 0 | 1 | 23 | 1123 | 1026 | 5 | -1 | -3 | 26 | 0 | 27 | 1 |

| | | | | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| -4 | 0 | 20 | 117 | 117 | 4 | -2 | -1 | 23 | 808 | 798 | 3 | 0 | 3 | 27 | 695 | 669 | 3 |

| *h* | *k* | *l* | 10Fo | 10Fc | 10σ | *h* | *k* | *l* | 10Fo | 10Fc | 10σ | *h* | *k* | *l* | 10Fo | 10Fc | 10σ |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 3 | 1 | 20 | 117 | 111 | 4 | 3 | 1 | 23 | 574 | 577 | 2 | 2 | 2 | 27 | 616 | 605 | 2 |
| -2 | -1 | 20 | 126 | 127 | 2 | 2 | 0 | 23 | 856 | 856 | 4 | 1 | 1 | 27 | 879 | 852 | 4 |
| 0 | 1 | 20 | 139 | 128 | 2 | -1 | 0 | 23 | 1019 | 1112 | 5 | 3 | 0 | 27 | 685 | 669 | 3 |
| 0 | -2 | 20 | 126 | 131 | 2 | 0 | 0 | 24 | 1097 | 1078 | 5 | 0 | 0 | 27 | 995 | 994 | 5 |
| 1 | 2 | 20 | 113 | 117 | 3 | 3 | 0 | 24 | 691 | 680 | 3 | 1 | 0 | 28 | 794 | 703 | 3 |
| -1 | -3 | 20 | 122 | 121 | 4 | 1 | 1 | 24 | 922 | 893 | 4 | -2 | 0 | 28 | 651 | 660 | 3 |
| 0 | 4 | 20 | 115 | 108 | 4 | 2 | 2 | 24 | 629 | 610 | 3 | 2 | 1 | 28 | 555 | 538 | 2 |
| 0 | 3 | 21 | 42 | 43 | 9 | 0 | 3 | 24 | 696 | 680 | 3 | 0 | -1 | 28 | 668 | 761 | 3 |
| 2 | 2 | 21 | 40 | 35 | 10 | 1 | 3 | 25 | 30 | 21 | 26 | 0 | 2 | 28 | 661 | 609 | 3 |

| *h* | *k* | *l* | 10Fo | 10Fc | 10σ | *h* | *k* | *l* | 10Fo | 10Fc | 10s |
|---|---|---|---|---|---|---|---|---|---|---|---|
| 14 | 58 | 14 | -4 | 0 | 8 | 322 | 319 | 2 | | | |
| 419 | 416 | 2 | 3 | 1 | 8 | 328 | 327 | 2 | | | |
| 499 | 508 | 2 | -2 | -1 | 8 | 474 | 470 | 2 | | | |
| 379 | 375 | 2 | 0 | 1 | 8 | 718 | 722 | 3 | | | |
| 405 | 407 | 2 | 0 | -2 | 8 | 578 | 577 | 2 | | | |
| 635 | 641 | 3 | 1 | 2 | 8 | 430 | 433 | 2 | | | |
| 862 | 871 | 4 | -3 | -2 | 8 | 305 | 290 | 2 | | | |
| 1140 | 1124 | 3 | 2 | 3 | 8 | 262 | 268 | 2 | | | |
| 682 | 695 | 3 | -1 | -3 | 8 | 354 | 355 | 2 | | | |
| 465 | 469 | 2 | 0 | 4 | 8 | 294 | 294 | 2 | | | |
| 447 | 451 | 2 | 0 | 3 | 9 | 1295 | 1271 | 6 | | | |
| 786 | 804 | 4 | 2 | 2 | 9 | 1122 | 1093 | 5 | | | |
| 1218 | 1219 | 3 | 1 | 1 | 9 | 1999 | 1902 | 5 | | | |
| 1629 | 1630 | 7 | 3 | 0 | 9 | 1294 | 1271 | 6 | | | |
| 960 | 959 | 4 | 0 | 0 | 9 | 2557 | 2591 | 7 | | | |
| 2633 | 2638 | 6 | 1 | 0 | 10 | 1232 | 1247 | 4 | | | |
| 1412 | 1382 | 6 | -2 | 0 | 10 | 1025 | 1020 | 5 | | | |
| 0 | 1649 | 1630 | 7 | 4 | 0 | 10 | 498 | 507 | 2 | | |
| 0 | 973 | 959 | 4 | -3 | -1 | 10 | 615 | 617 | 3 | | |
| 1 | 446 | 451 | 2 | 2 | 1 | 10 | 749 | 766 | 3 | | |
| 1 | 462 | 469 | 2 | 0 | -1 | 10 | 1345 | 1351 | 5 | | |
| 1 | 408 | 407 | 2 | 0 | 2 | 10 | 923 | 941 | 4 | | |
| 1 | 377 | 375 | 2 | -1 | -2 | 10 | 844 | 830 | 4 | | |
| 1 | 691 | 695 | 3 | 3 | 2 | 10 | 463 | 458 | 2 | | |
| 1 | 800 | 804 | 4 | -2 | -3 | 10 | 494 | 496 | 2 | | |
| 1 | 1263 | 1219 | 3 | 1 | 3 | 10 | 558 | 569 | 2 | | |

| *h* | *k* | *l* | 10Fo | 10Fc | 10σ | *h* | *k* | *l* | 10Fo | 10Fc | 10s |
|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | 633 | 641 | 3 | 0 | -4 | 10 | 547 | 550 | 2 | | |
| 1 | 504 | 508 | 2 | 0 | 4 | 11 | 44 | 33 | 8 | | |
| 1 | 414 | 416 | 2 | -1 | -3 | 11 | 34 | 27 | 34 | | |
| 1 | 867 | 871 | 4 | 2 | 3 | 11 | 31 | 38 | 17 | | |
| 1 | 1136 | 1124 | 3 | -3 | -2 | 11 | 49 | 42 | 8 | | |
| 2 | 417 | 379 | 2 | 1 | 2 | 11 | 0 | 23 | 1 | | |
| 2 | 223 | 217 | 1 | 0 | -2 | 11 | 53 | 52 | 3 | | |
| 2 | 48 | 58 | 7 | 0 | 1 | 11 | 115 | 95 | 1 | | |
| 2 | 71 | 74 | 4 | -2 | -1 | 11 | 51 | 25 | 4 | | |
| 2 | 164 | 160 | 1 | 3 | 1 | 11 | 0 | 25 | 1 | | |
| 2 | 383 | 350 | 1 | -4 | 0 | 11 | 52 | 36 | 7 | | |
| 2 | 241 | 235 | 1 | 2 | 0 | 11 | 60 | 48 | 3 | | |
| 2 | 148 | 148 | 1 | -1 | 0 | 11 | 119 | 102 | 1 | | |
| 2 | 0 | 43 | 1 | 0 | 0 | 12 | 663 | 606 | 3 | | |
| 2 | 0 | 39 | 1 | 3 | 0 | 12 | 161 | 160 | 2 | | |
| 2 | 80 | 81 | 4 | 1 | 1 | 12 | 345 | 351 | 1 | | |
| 2 | 55 | 54 | 6 | 2 | 2 | 12 | 111 | 114 | 3 | | |
| 3 | 180 | 172 | 3 | 0 | 3 | 12 | 161 | 160 | 2 | | |
| 3 | 195 | 194 | 2 | 0 | -4 | 13 | 463 | 465 | 2 | | |
| 3 | 180 | 187 | 2 | 1 | 3 | 13 | 471 | 478 | 2 | | |
| 3 | 227 | 241 | 1 | -2 | -3 | 13 | 425 | 422 | 2 | | |
| 3 | 175 | 172 | 3 | 3 | 2 | 13 | 379 | 389 | 2 | | |
| 3 | 186 | 194 | 2 | -1 | -2 | 13 | 677 | 680 | 3 | | |
| 3 | 349 | 347 | 1 | 0 | 2 | 13 | 744 | 752 | 3 | | |

| | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| 4 | 1380 | 1350 | 4 | 0 | -1 | 13 | 1031 | 1029 | 4 | | |
| 4 | 1122 | 1113 | 5 | 2 | 1 | 13 | 620 | 628 | 3 | | |
| 4 | 544 | 545 | 2 | -3 | -1 | 13 | 520 | 518 | 2 | | |
| 4 | 657 | 667 | 3 | 4 | 0 | 13 | 421 | 429 | 2 | | |
| *h* | *k* | *l* | 10Fo | 10Fc | 10σ | *h* | *k* | *l* | 10Fo | 10Fc | 10s |
| 4 | 830 | 836 | 4 | -2 | 0 | 13 | 826 | 815 | 4 | | |
| 4 | 1488 | 1463 | 4 | 1 | 0 | 13 | 950 | 949 | 4 | | |
| 4 | 1021 | 1027 | 4 | -1 | 0 | 14 | 1610 | 1647 | 6 | | |
| 4 | 899 | 906 | 4 | 2 | 0 | 14 | 1165 | 1181 | 5 | | |
| 4 | 489 | 491 | 2 | -4 | 0 | 14 | 712 | 700 | 3 | | |
| 4 | 546 | 532 | 2 | 3 | 1 | 14 | 715 | 726 | 3 | | |
| 4 | 607 | 615 | 3 | -2 | -1 | 14 | 1062 | 1054 | 5 | | |
| 4 | 585 | 591 | 2 | 0 | 1 | 14 | 1506 | 1520 | 6 | | |
| 5 | 540 | 549 | 2 | 0 | -2 | 14 | 1287 | 1280 | 6 | | |
| 5 | 662 | 673 | 3 | 1 | 2 | 14 | 960 | 972 | 4 | | |
| 5 | 488 | 492 | 2 | -1 | -3 | 14 | 784 | 787 | 3 | | |
| 5 | 538 | 534 | 2 | 0 | 4 | 14 | 637 | 646 | 3 | | |
| 5 | 831 | 840 | 4 | 0 | 3 | 15 | 519 | 518 | 2 | | |
| 5 | 1122 | 1101 | 5 | 2 | 2 | 15 | 466 | 460 | 2 | | |
| 5 | 1271 | 1274 | 4 | 1 | 1 | 15 | 721 | 717 | 3 | | |
| 5 | 926 | 911 | 4 | 3 | 0 | 15 | 527 | 518 | 2 | | |
| 5 | 617 | 621 | 3 | 0 | 0 | 15 | 925 | 920 | 4 | | |
| 5 | 590 | 595 | 3 | 1 | 0 | 16 | 115 | 101 | 2 | | |
| 5 | 1033 | 1016 | 5 | -2 | 0 | 16 | 92 | 85 | 2 | | |
| 5 | 1423 | 1380 | 4 | 4 | 0 | 16 | 0 | 25 | 1 | | |
| 6 | 874 | 840 | 4 | -3 | -1 | 16 | 0 | 32 | 1 | | |
| 6 | 397 | 399 | 1 | 2 | 1 | 16 | 62 | 56 | 4 | | |
| 6 | 570 | 576 | 2 | 0 | -1 | 16 | 114 | 110 | 2 | | |
| 6 | 353 | 357 | 2 | 0 | 2 | 16 | 86 | 78 | 2 | | |
| 6 | 393 | 399 | 1 | -1 | -2 | 16 | 62 | 61 | 3 | | |
| 7 | 62 | 66 | 5 | 1 | 3 | 16 | 35 | 30 | 11 | | |
| 7 | 56 | 57 | 5 | 0 | -4 | 16 | 0 | 27 | 1 | | |
| *h* | *k* | *l* | 10Fo | 10Fc | 10σ | *h* | *k* | *l* | 10Fo | 10Fc | 10s |
| 7 | 82 | 69 | 5 | 0 | 4 | 17 | 223 | 220 | 3 | | |
| 7 | 74 | 64 | 5 | -1 | -3 | 17 | 260 | 258 | 2 | | |
| 7 | 11 | 55 | 11 | 1 | 2 | 17 | 288 | 294 | 2 | | |
| 7 | 52 | 66 | 3 | 0 | -2 | 17 | 368 | 373 | 1 | | |
| 7 | 170 | 174 | 1 | 0 | 1 | 17 | 451 | 435 | 2 | | |
| 7 | 61 | 51 | 3 | -2 | -1 | 17 | 317 | 318 | 2 | | |
| 7 | 76 | 61 | 4 | 3 | 1 | 17 | 236 | 238 | 2 | | |
| 7 | 62 | 61 | 5 | -4 | 0 | 17 | 246 | 238 | 2 | | |
| 7 | 67 | 72 | 2 | 2 | 0 | 17 | 338 | 344 | 1 | | |
| 7 | 158 | 161 | 1 | -1 | 0 | 17 | 458 | 472 | 2 | | |
| 8 | 792 | 782 | 3 | 0 | 0 | 18 | 1673 | 1628 | 7 | | |
| 8 | 532 | 533 | 2 | 3 | 0 | 18 | 974 | 952 | 4 | | |
| -1 | -2 | 28 | 587 | 583 | 3 | 1 | 0 | 34 | 75 | 55 | 5 |
| 1 | 2 | 29 | 135 | 128 | 4 | -2 | 0 | 34 | 52 | 63 | 9 |
| 0 | -2 | 29 | 155 | 148 | 3 | 2 | 1 | 34 | 61 | 59 | 9 |
| 0 | 1 | 29 | 169 | 147 | 2 | 0 | -1 | 34 | 60 | 60 | 6 |
| -2 | -1 | 29 | 146 | 139 | 3 | 0 | 2 | 34 | 83 | 58 | 6 |
| 2 | 0 | 29 | 150 | 137 | 3 | -1 | -2 | 34 | 68 | 64 | 8 |
| -1 | 0 | 29 | 133 | 159 | 3 | 0 | -2 | 35 | 40 | 39 | 13 |
| 0 | 0 | 30 | 79 | 86 | 3 | 0 | 1 | 35 | 54 | 49 | 7 |
| 3 | 0 | 30 | 38 | 38 | 15 | 2 | 0 | 35 | 25 | 36 | 24 |
| 1 | 1 | 30 | 23 | 62 | 23 | -1 | 0 | 35 | 0 | 53 | 1 |
| 0 | 3 | 30 | 51 | 38 | 51 | 0 | 0 | 36 | 413 | 423 | 2 |
| -1 | -2 | 31 | 282 | 287 | 3 | 1 | 1 | 36 | 380 | 381 | 3 |
| 0 | 2 | 31 | 332 | 292 | 2 | 0 | 2 | 37 | 543 | 472 | 3 |
| 0 | -1 | 31 | 302 | 356 | 2 | 0 | -1 | 37 | 475 | 570 | 2 |
| 2 | 1 | 31 | 284 | 265 | 3 | -2 | 0 | 37 | 440 | 512 | 3 |
| -2 | 0 | 31 | 297 | 317 | 3 | 1 | 0 | 37 | 607 | 526 | 3 |
| *h* | *k* | *l* | 10Fo | 10Fc | 10σ | *h* | *k* | *l* | 10Fo | 10Fc | 10s |
| 1 | 0 | 31 | 387 | 328 | 2 | -1 | 0 | 38 | 255 | 301 | 3 |

| | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| -1 | 0 | 32 | 553 | 666 | 2 | 0 | 1 | 38 | 335 | 277 | 2 |
| 2 | 0 | 32 | 605 | 540 | 2 | 1 | 1 | 39 | 62 | 69 | 8 |
| -2 | -1 | 32 | 519 | 522 | 3 | 0 | 0 | 39 | 77 | 84 | 5 |
| 0 | 1 | 32 | 708 | 614 | 3 | 1 | 0 | 40 | 110 | 98 | 4 |
| 0 | -2 | 32 | 549 | 585 | 2 | 0 | -1 | 40 | 90 | 106 | 5 |
| 1 | 2 | 32 | 522 | 482 | 3 | 0 | 1 | 41 | 477 | 407 | 2 |
| 1 | 1 | 33 | 611 | 593 | 3 | -1 | 0 | 41 | 367 | 441 | 2 |
| 0 | 0 | 33 | 672 | 670 | 3 | 0 | 0 | 42 | 495 | 491 | 2 |

Table 7: Charge distribution for $InGaZnO_4$. σ measures the deviation of *Q* with respect to *q*.

| | | 20kb (single crystal) | | 50kb (twin) | |
|---|---|---|---|---|---|
| Atom | q | Q | q/Q | Q | q/Q |
| In | 3.00 | 2.94 | 1.02 | 2.95 | 1.02 |
| Ga/Zn | 2.50 | 2.53 | 0.99 | 2.53 | 0.99 |
| | | σ = 0.066 | | σ = 0.058 | |
| O1 | -2.00 | -1.96 | 1.02 | $-1.96_5$ | 1.02 |
| O2 | -2.00 | -2.04 | 0.98 | $-2.03_5$ | 0.98 |
| | | σ = 0.057 | | σ = 0.050 | |

Table 8: Charge distribution of cations for some structures related to $YbFe_2O_4$.

| Compound | Cation | *q* | *Q* | *q*/*Q* |
|---|---|---|---|---|
| $LuFeZnO_4$[a] | Lu | 3.00 | 2.83 | 1.06 |
| | Fe/Zn | 2.50 | 2.58 | 0.97 |
| $LuFe_2O_4$[b] | Lu | 3.00 | 2.84 | 1.06 |
| | $Fe^{II}/Fe^{III}$ | 2.50 | 2.58 | 0.97 |
| $LuFeCoO_4$[b] | Lu | 3.00 | 2.86 | 1.05 |
| | Fe/Co | 2.50 | 2.57 | 0.97 |
| $InCuAlO_4$[c] | In | 3.00 | 2.85 | 1.05 |
| | Al/Cu | 2.50 | 2.57 | 0.97 |
| *$InFe^{3+}_{0.5}Fe^{2+}_{1.25}Si_{0.25}O_4$[c] | In | 3.00 | 2.96 | 1.01 |
| | Fe/Si | 2.50 | 2.52 | 0.99 |
| $YbFe_2O_4$[d] | Yb | 3.00 | 2.82 | 1.06 |
| | $Fe^{II}/Fe^{III}$ | 2.50 | 2.59 | 0.96 |
| $Yb_{0.5}Eu_{0.5}Fe_2O_4$[e] | Yb/Eu | 3.00 | 2.79 | 1.08 |
| | $Fe^{II}/Fe^{III}$ | 2.50 | 2.61 | 0.96 |
| $Yb_2Fe_3O_7$[f] | Yb | 3.00 | 2.76 | 1.09 |
| | Fe | 3.00 | 3.23 | 0.93 |
| | $Fe^{II}/Fe^{III}$ | 2.50 | 2.63 | 0.95 |
| *$Yb_2Fe_3O_7$[g] | Yb | 3.00 | 2.93 | 1.02 |
| | Fe | 3.00 | 3.13 | 0.96 |
| | $Fe^{II}/Fe^{III}$ | 2.50 | 2.51 | 1.00 |

*Compounds with and without star (*) belong to type-I (regular bipyramids: the two apical M-O distances longer than the three basal ones) and type-II (distorted bipyramids: one of the apical M-O distances shorter and the other longer than the three basal ones) respectively. [a]ISOBE *et al*. (1994); [b]ISOBE *et al*. (1990); [c]GÉRARDIN *et al*. (1980); [d]KATO *et al*. (1975); [e]MALAMAN *et al*. (1975); [f]KATO *et al*. (1976); [g]MALAMAN *et al*. (1976).

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### References

ADELSKÖLD, V.: Arkiv for kemi, mineralogi och geologi, Ser. A 12, No. 29 (1938) 1
BATAILLE, T., BÉNARD-ROCHERULLÉ, P., LOUËR, D.: J. Solid St. Chem. **140** (1998) 62
BERTAUT, E.D., MARESCHAL, J.: Comptes Rendu Acad. Sci. Paris **257** (1963) 867
BERTAUT, E.D., BUISSON, G., DURIF, A., MARESCHAL, J., MONTMORY, M.C., QUEZEL-AMBRUNAZ, S.: Bull. Soc. franç. Chem. (1965) 1132
BOUDIN, S., LII, K.-H.: Acta Crystallogr. **C54** (1998) 5
BROWN, I. D.: In *Structure and Bonding in Crystals*, vol. II, M. O'Keeffe and A. Navrotsky, (Ed), chapter 14, Academic Press Inc., New York (1981)
BROWN, F., FLORES, M.J.R., KIMIZUKA, N., MICHIUE, Y., ONODA, M., MOHRI, T., NAKAMURA, M., ISHIZAWA, N.: J. Solid St. Chem. **144** (1999) 91
CAVA, R.J., RAMIREZ, A.P., HUANG, Q., KRAJEWSKI, J.J.: J. Solid St. Chem., **140** (1998) 337.
FENG, P., BU, X., STUCKY, G.D.: Acta Crystallogr., **C53** (1997) 997
FLÜGEL-KAHLER, E.: Acta Crystallogr. **16**, 1009
FRIEDEL, G.: *Étude sur les groupements cristallins*. Extrait du Bullettin de la Société de l'Industrie minérale, Quatrième série, Tomes III e IV. Saint-Étienne: Societè de l'Imprimerie Thèolier J. Thomas et C. (1904)
FRIEDEL, G.: *Leçons de Cristallographie*. Nancy, Paris, Strasbourg: Berger-Levrault (1926)
GEBERT, E.; KIHLBORG, L.: Acta Chem. Scand. **23** (1969) 221
GELLER, S., JEFFRIES, J.B., CURLANDER, P.J.: Acta Crystallogr. **B31** (1975) 2770
GÉRARDIN, R., ALEBOUYEH, A., JEANNOT, F., COURTOIS, A., MALAMAN, B., EVRARD, O.: Mat. Res. Bull. **15** (1980) 647
GIAQUINTA, D.M., DAVIS, W.M., ZUR LOYE, H.-C.: Acta Crystallogr., **C50** (1994) 5
GIAQUINTA, D.M., ZUR LOYE, H.-C., J. Am. Chem. Soc., **114** (1992) 10952
GOPAL, R., CALVO, C.: Can. J. Chem. **51** (1973) 1004
GREEDAN, J.E., BIERINGER, M., BRITTEN, J.F., GIAQUINTA, D.M., ZUR LOYE, H.-C.: J. Solid St. Chem. **116** (1995) 118
HARMS, K.: XCAD4, Program for the LP-correction of CAD4 Diffractometer Data. University of Marburg, Germany (1996)
HERBST-IRMER, R., SCHELDRICK, G.M.: Acta Crystallogr., **B54** (1998) 443
HOPPE, R., VOIGT, S., GLAUM, H., KISSEL, J., MÜLLER, H. P., BERNET, K.: J. Less-Comm. Met. **156** (1989) 105
ISOBE, M., KIMIZUKA, N., IIDA, J., TAKEKAWA, S.: Acta Crystallogr. **C46** (1990) 1917
ISOBE, M., KIMIZUKA, N., NAKAMURA, M., MOHRI, T.: Acta Crystallogr. **C50** (1994) 332
JAKEMAN, J.B., CHEETHAM, A.K.: J. Am. Chem. Soc. **110** (1998) 1140
KATO, K., KAWADA, I., KIMIZUKA, N., KATSURA, T.: Z. Kristallogr. **141** (1975) 314
KATO, K., KAWADA, I., KIMIZUKA, N., SHINDO, I., KATSURA, T.: Z. Kristallogr. **143** (1976) 278
KIMIZUKA. N., ISOBE, M., NAKAMURA, M.: J. Solid St. Chem. **116** (1995) 170
MALAMAN, B., EVRARD. O., TANNIÈERES, N., AUBRY, J., COURTOIS, A., PROTAS, J.: Acta Crystallogr. **B31** (1975) 1310
MALAMAN, B., EVRARD. O., TANNIÈERES, N., COURTOIS, A., PROTAS, J.: Acta Crystallogr., **B32** (1976) 749
MODARESSI, A., COURTOIS, A., GERARDIN, R., MALAMAN, B., GLEITZER, C.: J. Solid St. Chem. **47** (1983) 245
NAKAMURA, M., KIMIZUKA, N., MOHRI, T.: J. Solid St. Chem. **93** (1991) 295
NESPOLO, M., FERRARIS, G., OHASHI, H.: Acta Crystallogr. **B55** (1999) 902
NESPOLO, M., NAKAMURA, M., OHASHI, H.: J. Solid. State Chem. **149** (2000) (2) in print
NORD, A.G., STEFANIDIS, T.: Polyhedron **1** (1982) 349
OHASHI, H.: J. Jpn. Assoc. Min. Petr. Econ. Geol. **84** (1989) 159
OHASHI, H., FUJITA, T., OSAWA, T.: J. Jpn. Assoc. Min. Petr. Econ. Geol. **78** (1983) 159
OHASHI, H., OSAWA, T., Sato, A., TSUKIMURA, K.: J. Jpn. Assoc. Min. Petr. Econ. Geol. **91** (1996) 21

OHASHI, H., OSAWA ,T,., SATO, A.: Acta Crystallogr. **C51** (1995) 2476
OHASHI, H., OSAWA, T., SATO, A.: Acta Crystallogr. **B46** (1990) 742
OHKAWA, M., FUJITA, S., TAKENO, S., NAKATSUKA, A., YOSHIASA, A., UCHIDA, M., OHSUMI, K.: Z. Kristallogr., **212** (1997) 848
PAULING, L.: J. Am. Chem. Soc. **51** (1929) 1010
POULSEN, S.J., CALVO. C.: Can. J. Chem. **46** (1968) 917
SCHMITZ-DUMONT, O., KASPER, H.: Z. anorg. allg. Chem. **341** (1965) 252
SHANNON, R.D., CALVO, C.: Acta Crystallogr. **B29** (1973) 1338
SHANNON, R.D., PREWITT, C.T.: J. inorg. nucl. Chem. **30** (1968) 1389
SHANNON, R.D., SLEIGHT, A.W.: Inorg. Chem. **7** (1968) 1649
SHELDRICK, G.M.: *SHELX97, Program for the Solution and Refinement of Crystal Structrues* (1997) University of Göttingen, Germany
SPECK, A.L.: Acta Crystallogr. **A46** (1990) C34
TOWNES, W.D., FANG. J.H., PERROTTA, A.J.: Z. Kristallogr. **125** (1967) 437
TRINSCHEK, D., JANSEN, M.: Z. anorg. all. Chem. **622** (1996) 245
VELIKODNYI, YU.A., MYRASHOVA, E.V., TRUNOV, V.K.: Sov. Phys. Crystallogr. **34** (1989) 361
YAKEL, H.L., KOEHLER, W.C., BERTAUT, E.F., FORRAT, E.F.: Acta Crystallogr. **16** (1963) 957
YOSHIASA, A., UENO, K., KANAMARU, F., HORIUCHI, H.: Mat. Res. Bull. **21** (1986) 175

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