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VOLUME II



# Standard X-ray Diffraction Powder Patterns

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# Standard X-ray Diffraction Powder Patterns

Howard E. Swanson and Ruth K. Fuyat


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# Contents

# STANDARD X-RAY DIFFRACTION POWDER PATTERNS
## Vol. II—Data for 30 Inorganic Substances

**Howard E. Swanson** and **Ruth K. Fuyat**[1]

Twenty-eight standard X-ray diffraction powder patterns are presented in revision of one hundred corresponding patterns in the ASTM card file, a system for the identification of unknown crystalline materials, based on the three strongest reflections of each distinct phase. Two patterns not represented in the file are also included. A comparison is made between all powder data available for each of the substances reported. The patterns were made with a high-angle geiger-counter spectrometer, using samples of exceptionally high purity. The *d*-spacings were assigned Miller indices determined in part from calculated patterns of theoretical spacings and from space group considerations. The lattice constants and density were calculated and, whenever possible, the refractive indices were measured.

This report includes X-ray data for the following thirty substances: C (diamond), Si, Ga, Zr, α-Sn, Re, ZnS (wurtzite), ZnS (sphalerite), PbS (galena), $\alpha$-$Al_2O_3$ (corundum), $Cu_2O$ (cuprite), ZnO (zincite), CdO, $Tl_2O_3$, PbO (litharge), PbO (massicot), $UO_2$, $MgAl_2O_4$, (spinel), $ZnAl_2O_4$, NaCl (halite), CsCl, $PbCl_2$ (cotunnite), $PbBr_2$, $NH_4Br$, $CaCO_3$ (calcite), $BaCO_3$ (witherite), $PbCO_3$ (cerussite), $Na_2SO_4$ (thenardite), $SrSO_4$ (celestite), and $Zn_4(OH)_2Si_2O_7 \cdot H_2O$ (hemimorphite).

## 1. Introduction

The National Bureau of Standards program[2] for the revision and evaluation of published X-ray data for the American Society for Testing Materials card file presents in this paper a second volume[3] of standard powder diffraction patterns for five elements and twenty three inorganic compounds. These patterns are recommended to replace one hundred cards now in the file. One element, alpha-tin, and one compound, uranium dioxide, not represented in the file, have been added.

The experimental procedure and general plan of these reports is discussed in the first two papers of this series by Swanson and Tatge [1, 2].[4] However, the essential steps followed in preparing these reports and significant changes in procedure are outlined below.

**ASTM cards.** Each section of this paper, devoted to the discussion of X-ray data for one substance, contains a table listing old and new file card numbers, the ASTM index lines, the radiation used and the literature reference for each card. The old card numbers of these tables refer to the original ASTM file (1939) and the first supplement (1944). The new card numbers are from the second edition and include the second supplement (1950).

**Additional published patterns.** Literature references and radiation data for patterns that have not been published as ASTM cards were listed and the patterns were included in the tables of *d*-spacings and intensities.

**NBS pattern.** The samples used to make the NBS patterns usually were special prepa-

[1] Fellow at the National Bureau of Standards sponsored by the Joint Committee on Chemical Analysis by X-ray Diffraction Methods.

[2] This project is sponsored by the Joint Committee on Chemical Analysis by X-ray Diffraction Methods, composed of members from the American Society for Testing Materials, the American Crystallographic Association, and the British Institute of Physics. Financial support is being given by the National Bureau of Standards and the Flight Research Laboratory, Wright Air Development Center, Wright-Patterson Air Force Base.

[3] The first volume of this series is NBS Circular 539, volume I, Standard X-ray diffraction powder patterns: Data for 53 inorganic substances, by Howard E. Swanson and Eleanor Tatge.

[4] Figures in brackets indicate the literature references at the end of each section of this paper.

rations of exceptionally high purity available only in small quantities.

The purity of each sample with the exception of diamond was determined by a spectrographic or chemical analysis. A phase purity check was made on the nonopaque materials during the refractive index determination. Another excellent check on the phase purity was provided by the X-ray pattern itself, since it was indexed by comparison with theoretical *d*-values. Most samples were heated to remove either gases or moisture, or to sharpen the peaks in their diffraction patterns, depending upon the need of each material.

The equipment and procedures were essentially the same as those previously described by Swanson and Tatge, with the exception that the high-angle X-ray spectrometer equipment now used is the North American Philips Company's latest model, which has replaced the modified machine previously described. This change made possible the use of an inclined instead of a vertical specimen mount, which simplified sample preparation for intensity measurements. In place of the old-model cell with a plastic window, an open-faced sample mounting was used to obtain the desired random orientation. A flat piece of glass was held temporarily over the face of the cell while the sample was drifted in from the top. The holder was then placed horizontally, face up, and the piece of glass was removed, exposing the surface of the powder for subsequent exposure to the X-ray beam. It was possible in most cases to tilt this type of open mounting about 80 degrees without spilling the powder.

The grain sizes of the samples were less than 25 microns to insure more reproducible intensity measurements. The intensity values of each pattern were measured as peak height above background and were expressed as percentages of the strongest line. All of the NBS patterns were made with copper radiation, $\lambda = 1.5405$, and were corrected by an internal standard of tungsten, whose lattice constant at 25°C is 3.1648 A, as determined by Jette and Foote [3].

**Interplanar spacings and intensity measurements.** The interplanar spacing data presented in the tables were converted to angstrom units as internationally defined in 1946 [4], from Bragg angle data, from *d*-spacings in kX units or supposed kX units, using the factor 1.00202, or from *d*-spacings with specifically stated wavelengths other than kX. In each case the type of conversion made was stated. The radiation values given in the tables of *d*-spacings and intensities are the wavelengths in angstroms, whereas the values listed under the first section of the reports, ASTM cards, are the original values taken from the literature.

The tables of patterns contain the data from the original literature, except when there is no source other than an ASTM card.

Intensities, when not numerically evaluated, were given the following abbreviations: strong, s; medium, m; weak, w; very, v; diffuse, D; and doublet, db. An abbreviation B, broad, is found in some ASTM card intensities. No attempt was made to follow in the tables of patterns, the intensities as found on ASTM cards when the original source was available. Those values were obtained by a set of conversions explained in the introduction to the card file.

The indexing of the NBS patterns was accomplished by comparison of the experimental data with theoretical values of all possible Miller indices of a particular cell calculated with the aid of IBM punched-card machines. The unit cells used for these calculations were obtained either through a review of the literature or with an estimated cell based on partial indexing of the NBS pattern. The indexing as it appears in the tables includes all of the probable indices for any given *d*-spacing allowed by the space group of that structure, and although an attempt was made to reconcile these values with published single crystal work when it was available, errors inherent in this method of

indexing undoubtedly are present. A maximum of 40 lines for the NBS pattern were generally considered sufficient for any identification problem, and indexing of a cell large enough to have many more lines would become increasingly indefinite beyond that number.

The intensity of the three strongest lines is particularly important because the ASTM card-file system of identification depends upon comparing the three strongest lines of the unknown material with the file cards, which are arranged according to their first, second, and third strongest lines. Thus a table of the three strongest lines of each pattern is listed for comparison with the NBS values.

**Lattice constants.** The NBS lattice constants of cubic materials were calculated for all $d$-spacings, and the average of the last five lines was assumed to be the best value because of greater accuracy of measurement in the large-angle part of the pattern. The unit-cell values for each noncubic substance were determined from all of the $d$-spacings for its pattern for which there was only one possible index by means of a least-squares calculation made on an IBM Card Program Calculator.

The conversion of published unit-cell data to angstroms follows the same pattern as that used for the $d$-spacings. The unit-cell dimensions were converted to 26°C for comparison with the NBS values if the temperature of measurement and the thermal expansion were known. Unless otherwise indicated, the coefficient of linear thermal expansion as used is defined as the change in length per unit length per degree centigrade in the room-temperature range. Thermal-expansion data have been given whenever the data were readily available, even though no temperature conversions were made in the unit-cell table. The limits of error generally published with unit-cell data have not been included in the table as the number of determinations, their accuracy, and variation were such that a statistical evaluation would be invalid.

The densities calculated from the NBS lattice constants were expressed in grams per cubic centimeter, and the refractive-index measurements were made in white light by grain immersion methods, using oils standardized in sodium light.

## References


[1] H. E. Swanson and E. Tatge, Standard X-ray diffraction patterns, NBS J. Research **46**, 318 (1951) RP 2202.

[2] Howard E. Swanson and Eleanor Tatge, Standard X-ray diffraction powder patterns: Data for 53 inorganic substances, NBS Circular 539, volume I (1953).

[3] E. R. Jette and F. Foote, Precision determination of lattice constants, J. Chem. Phys. **3**, 605-16 (1935).

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# 2. X-ray Data

## 2.1. Elements

### Carbon (diamond) C (cubic)

**ASTM cards**

| Card number | | New index lines | Radiation | Source |
|---|---|---|---|---|
| Old | New | | | |
| 3346 | 3529 | 2.05 | Copper--- | Hull [1] 1917. |
| | 1-1248 | 1.26 | | |
| | 1-1249 | 1.07 | | |
| 2878 | 3528 | 2.06 | Copper--- | General Electric Co., Wembley, England. |
| | 2-1246 | 1.26 | | |
| | 2-1248 | 1.07 | | |

**Additional published patterns**

| Source | Radiation | Wavelength |
|---|---|---|
| Brill, Grimm, Hermann, and Peters [2] 1939---------- | Molybdenum | ---------- |

**NBS pattern.** The sample used for the NBS pattern was an industrial abrasive powder of particle size less than 2 microns. The diamond dust, almost pure white and free of any foreign matter, was not spectrographed because solid-solution reactions are not known to occur in diamond.

**Diamond, C**

| *hkl* | 1917 Hull Mo, 0.70926 A | | | 1939 Brill, Grimm, Hermann, and Peters Mo, 0.70926 A | | | ---- General Electric Co. Wembley, England Cu, 1.5405 A, 18°C | | | 1953 Swanson and Fuyat Cu, 1.5405 A, 27°C | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | *d* | *I* | *a* | *d* | *I*[a] | *a* | *d* | *I* | *a* | *d* | *I* | *a* |
| | *A* | | *A* | *A* | | *A* | *A* | | *A* | *A* | | *A* |
| 111 | 2.04 | 100 | 3.537 | 2.0575 | 100.0 | 3.564 | 2.064 | 100 | 3.575 | 2.060 | 100 | 3.5672 |
| 220 | 1.26 | 50 | 3.550 | 1.2604 | 45.8 | 3.565 | 1.263 | 80 | 3.571 | 1.261 | 27 | 3.5667 |
| 311 | 1.068 | 40 | 3.541 | 1.0752 | 16.9 | 3.566 | 1.076 | 60 | 3.569 | 1.0754 | 16 | 3.5667 |
| 222 | --- | --- | --- | 1.0298 | 0.7 | 3.567 | --- | --- | --- | --- | --- | --- |
| 400 | 0.882 | 10 | 3.526 | 0.8915 | 21.6 | 3.566 | 0.892 | 50 | 3.567 | 0.8916 | 7 | 3.5665 |
| 331 | .810 | 25 | 3.530 | .8179 | 9.8 | 3.565 | .818 | 80? | 3.568 | .8182 | 15 | 3.5666 |
| 422 | .718 | 40 | 3.518 | .7282 | 11.4 | 3.567 | --- | --- | --- | --- | --- | --- |
| 511 | .677 | 20 | 3.519 | .6863 | 5.9 | 3.566 | --- | --- | --- | --- | --- | --- |
| 440 | .623 | 10 | 3.522 | .6306 | 9.0 | 3.567 | --- | --- | --- | --- | --- | --- |
| 531 | .595 | 20 | 3.518 | --- | --- | --- | --- | --- | --- | --- | --- | --- |
| 533 | .556 | 15 | 3.645 | 0.5440 | 3.1 | 3.567 | --- | --- | --- | --- | --- | --- |
| 622 | .536 | 6 | 3.555 | .5379 | 0.07 | 3.568 | --- | --- | --- | --- | --- | --- |
| 444 | .505 | 3 | 3.499 | .5149 | 4.7 | 3.567 | --- | --- | --- | --- | --- | --- |
| 711 | .494 | 8 | 3.528 | .4995 | 2.5 | 3.567 | --- | --- | --- | --- | --- | --- |
| 642 | .471 | 20 | 3.526 | --- | --- | --- | --- | --- | --- | --- | --- | --- |
| 731 | .460 | 15 | 3.535 | 0.4682 | 2.0 | 3.566 | --- | --- | --- | --- | --- | --- |
| 800 | .440 | 0.5 | 3.522 | .4459 | 4.1 | 3.567 | --- | --- | --- | --- | --- | --- |
| 733 | .430 | .3 | 3.522 | .4357 | 2.0 | 3.566 | --- | --- | --- | --- | --- | --- |
| 822 | .415 | 12 | 3.524 | .4204 | 3.9 | 3.567 | --- | --- | --- | --- | --- | --- |
| 751 | .407 | 8 | 3.528 | .4118 | 1.9 | 3.566 | --- | --- | --- | --- | --- | --- |
| 840 | .395 | 5 | 3.537 | --- | --- | --- | --- | --- | --- | --- | --- | --- |
| 911 | .388 | 8 | 3.530 | 0.3914 | 2.3 | 3.566 | --- | --- | --- | --- | --- | --- |
| 664 | .377 | 5 | 3.532 | .3803 | 5.0 | 3.567 | --- | --- | --- | --- | --- | --- |
| 931 | .371 | 5 | 3.535 | --- | --- | --- | --- | --- | --- | --- | --- | --- |
| 844 | .362 | 7 | 3.542 | --- | --- | --- | --- | --- | --- | --- | --- | --- |
| 933 | .357 | 20 | 3.548 | --- | --- | --- | --- | --- | --- | --- | --- | --- |
| Average unit cell for last five lines | | | 3.537 | --- | --- | 3.566 | --- | --- | 3.570 | --- | --- | 3.5667 |

[a] Powder intensities calculated from single-crystal measurements.

**Interplanar spacings and intensity measurements.** The Hull $d$-spacings, based on molybdenum radiation of wavelength 0.712, were converted to angstroms for these tables. The Brill, Grimm, Hermann, and Peters, and the General Electric $d$-spacings were converted from kX to angstrom units.

The intensity measurements listed for the Brill pattern were made from single-crystal determinations and were mathematically converted to their powder-pattern equivalents. They are reproduced in the table in that form.

The three strongest lines for each of the patterns are as follows:

| Pattern | 1 | 2 | 3 |
|---|---|---|---|
| Hull | 111 | 220 | 311 |
| Brill, Grimm, Hermann, and Peters | 111 | 220 | 400 |
| General Electric Co. | 111 | 220 | 331 |
| Swanson and Fuyat | 111 | 220 | 311 |

The Brill, Grimm, Hermann, and Peters pattern lists a line, 222, forbidden by the postulated structure. According to Renninger [3], it is caused by "311 reflection back over $\bar{1}11$."

**Lattice constants.** The structure was determined by Bragg and Bragg [4] in 1913. The modified face-centered cubic lattice has space group $O_h^7$-Fd3m, diamond-structure type, and 8(C) per unit cell. Four other forms of carbon are known, all of them being graphites.

A group of unit-cell measurements, which supposedly were expressed in kλ units, have been converted to angstroms from the temperatures indicated in parentheses and 26°C for comparison with the NBS values, using the coefficient of expansion $1.38 \times 10^{-6}$, as determined by Straumanis and Aka [5] in 1951.

*Lattice constant in angstroms*

| | | |
|---|---|---|
| 1926 | Ehrenberg [6] | 3.5669 at 26°C (18°C) |
| 1932 | Yuching Tu [7] | 3.56689 at 26°C (18°C)<br>3.56679 at 26°C (18°C) |
| 1937 | Trzebiatowski [8] | 3.56669 |
| 1944 | Riley [9] | 3.56684 |
| 1944 | Lonsdale [10] | 3.56693 at 26°C (18°C) |
| 1951 | Straumanis and Aka [5] | 3.56682 at 26°C (20°C) |
| 1953 | Swanson and Fuyat | 3.5667 at 26°C |

The density of diamond calculated from the NBS lattice constant is 3.515 at 26°C. The refractive index of diamond is too high to be determined by liquid grain immersion methods.

**References**


[1] A. W. Hull, A new method of X-ray crystal analysis, Phys. Rev. **10**, 661 (1917).

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[3] M. Renninger, Magnified weak and apparently forbidden X-ray reflections by "detour excitation," Naturwissenschaften **25**, 43 (1937).

[4] W. H. Bragg and W. L. Bragg, The structure of the diamond, Proc. Roy. Soc. (London) **89A**, 277 (1913).

[5] M. E. Straumanis and E. Z. Aka, Precision determination of lattice parameters coefficient of thermal expansion, and atomic weight of carbon in diamond, J. Am. Chem. Soc. **73**, 5643-6 (1951).

[6] W. Ehrenberg, Die Grösse des Diamantgitters, Z. Krist. **63**, 320 (1926).

[7] Yuching Tu, A precision comparison of calculated and observed grating constants of crystals, Phys. Rev. **40**, 662 (1932).

[8] W. Trzebiatowski, Precise determination of the space-lattice constants of diamond and graphite, Roczniki Chem. **17**, 73-82 (1937).

[9] D. P. Riley, Lattice constant of diamond and the c-c single bond, Nature **153**, 587 (1944).

[10] Kathleen Lonsdale, Divergent-beam X-ray photography, Nature **153**, 22 (1944).


## Silicon, Si (cubic)

**ASTM cards**

| Card number | | New index lines | Radiation | Source |
|---|---|---|---|---|
| Old | New | | | |
| 1905 | 1922 | 3.13 | Molybdenum 0.712. | Hull [1] 1917. |
| | 1-0798 | 1.93 | | |
| | 1-0787 | 1.64 | | |
| ------- | 1935 | 3.11 | Copper, unresolved Kα-doublet. | Lehmann [2] 1924. |
| | 3-0556 | 1.90 | | |
| | 3-0549 | 1.63 | | |
| ------- | 1847 | 3.17 | Molybdenum, unresolved Kα-doublet. | Lehmann [2] 1924. |
| | 3-0524 | 1.92 | | |
| | 3-0517 | 1.63 | | |
| 1914 | 1927 | 3.12 | Molybdenum | Hanawalt, Rinn, and Frevel [3] 1938. |
| | 1-0799 | 1.91 | | |
| | 1-0791 | 1.63 | | |
| ------- | 1932 | 3.13 | Iron | Osawa and Okamoto [4] 1939. |
| | 3-0553 | 1.91 | | |
| | 3-0534 | 1.10 | | |

**ASTM cards—Con.**

| Card number | | New index lines | Radiation | Source |
|---|---|---|---|---|
| Old | New | | | |
| ------- | 1933 | 3.12 | Copper------- | Baumann [5] 1941. |
| | 3-0554 | 1.91 | | |
| | 3-0544 | 1.10 | | |
| ------- | 1924 | 3.14 | Copper------- | United Steel Companies, Sheffield, England. |
| | 3-0553 | 1.92 | | |
| | 3-0529 | 1.64 | | |
| II-1223 | 1925 | 3.13 | Molybdenum--- | United Steel Companies, Sheffield, England. |
| | 2-0575 | 1.92 | | |
| | 2-0561 | 1.63 | | |

**Additional published patterns**

| Source | Radiation | Wavelength |
|---|---|---|
| Debye and Scherrer [6] 1916---------- | Copper------- | 1.549 |

**NBS pattern.** The sample used for the NBS pattern was prepared at Johnson, Matthey and Co., Ltd. Their spectrographic analysis indicated 0.001 percent or less of copper, silver, zinc, tin, iron, and magnesium.

**Interplanar spacings and intensity measurements.** The Lehmann and Debye-Scherrer *d*-spacings, expressed in angstroms, were calculated from their Bragg angle data. The Hull *d*-spacings were converted from molybdenum radiation with $\lambda$ of 0.712 to angstroms, and all of the *d*-spacings from the remaining patterns were converted from kX to angstrom units. Lehmann's pattern contains a line at 3.81 A with medium intensity, which he indexed as 110. This reflection is not allowed by the diamond-type structure. The 1.56 A line, indexed 222, is a very weak reflection, which, according to Renninger [7], may occur in the diamond-structure type due to "detour excitation."

Of the two United Steel ASTM cards in the file only the pattern made with copper radiation is presented in the table. The old card indicating molybdenum radiation may be made identical with the pattern using copper radiation by conversion from kX units to angstroms. This, and especially the fact that the intensities are identical, leads one to suspect that all of the patterns might have been made with copper radiation, the one marked copper simply containing three additional lines.

The three strongest lines for each of the patterns are as follows:

| Pattern | 1 | 2 | 3 |
|---|---|---|---|
| Hull---------- | 111 | 220 | 311 |
| Lehmann---------- | 111 | 220 | 311 |
| Lehmann---------- | 111 | 220 | 311 |
| Hanawalt, Rinn, and Frevel---- | 111 | 220 | 311 |
| Osawa and Okamoto---------- | 111 | 220 | 422 |
| Baumann---------- | 111 | 220 | 422 |
| United Steel Companies-------- | 111 | 220 | 311 |
| Debye and Scherrer---------- | 111 | 220 | 311 |
| Swanson and Fuyat---------- | 111 | 220 | 311 |

**Lattice constants.** The structure was determined by Debye and Scherrer [6] in 1916. The space group is $O_h^7$-Fd3m, diamond-structure type with 8(Si) per unit cell. There is also an unstable hexagonal "high" form.

A group of unit-cell measurements have been converted from kX to angstrom units and from the temperatures indicated in parentheses to 26°C for comparison with the NBS values. The linear coefficient of expansion is $4.15 \times 10^{-6}$, according to Straumanis and Aka [15].

*Lattice constant in angstroms*

| | | |
|---|---|---|
| 1922 | Gerlach [8]---------- | 5.426 |
| 1923 | Küstner and Remy [9]------ | 5.431 |
| 1928 | van Arkel [10]---------- | 5.429 |
| 1933 | Jette and Gebert [11]----- | 5.4279 |
| 1935 | Neuburger [12]---------- | 5.4283 at 26°C (20°C) |
| 1935 | Jette and Foote [13]------ | 5.43077 at 26°C (25°C) |
| 1935 | Straumanis and Ievinš [14] | 5.4298 |
| 1952 | Straumanis and Aka [15]--- | 5.43097 at 26°C (25°C) |
| 1953 | Swanson and Fuyat---------- | 5.4301 at 26°C |

The density of silicon calculated from the NBS lattice constant is 2.328 at 26°C.

| hkl | 1917 Hull Mo, 0.709 A | | | 1924 Lehmann Mo, 0.709 A | | | 1924 Lehmann Cu, 1.5405 A | | | 1938 Hanawalt, Rinn, and Frevel Mo, 0.709 A | | | 1939 Osawa and Okamoto Fe, 1.93597 A | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | d | I | a | d | I | a | d | I | a | d | I | a | d | I | a |
| | A | | A | A | | A | A | | A | A | | A | A | | A |
| --- | --- | --- | --- | --- | --- | --- | 3.81 | m | --- | --- | --- | --- | --- | --- | --- |
| 111 | 3.12 | 100 | 5.40 | 3.17 | vvs | 5.49 | 3.11 | vvs | 5.39 | 3.13 | 100 | 5.42 | 3.14 | 100 | 5.44 |
| 220 | 1.92 | 80 | 5.43 | 1.93 | vvs | 5.46 | 1.90 | vvs | 5.37 | 1.91 | 100 | 5.40 | 1.91 | 100 | 5.40 |
| 311 | 1.63 | 75 | 5.41 | 1.64 | vvs | 5.44 | 1.62 | vvs | 5.37 | 1.63 | 63 | 5.41 | 1.63 | 80 | 5.41 |
| 222 | --- | --- | --- | --- | --- | --- | 1.56 | vvw | 5.40 | --- | --- | --- | --- | --- | --- |
| 400 | 1.35 | 25 | 5.40 | 1.35 | m | 5.40 | 1.35 | s | 5.40 | 1.357 | 18 | 5.428 | 1.35 | 60 | 5.40 |
| 331 | 1.24 | 45 | 5.40 | 1.24 | vs | 5.40 | 1.24 | vs | 5.41 | 1.245 | 25 | 5.427 | 1.24 | 70 | 5.41 |
| 422 | 1.11 | 50 | 5.44 | 1.108 | vs | 5.428 | 1.103 | vs | 5.404 | 1.106 | 40 | 5.418 | 1.10 | 100 | 5.39 |
| 511 | 1.04 | 40 | 5.40 | 1.044 | s | 5.425 | 1.040 | s | 5.404 | --- | --- | --- | [a]1.04 | 80 | 5.40 |
| 440 | 0.96 | 20 | 5.43 | --- | --- | --- | 0.957 | s | 5.414 | 0.960 | 6 | 5.431 | --- | --- | --- |
| 531 | .92 | 30 | 5.44 | 0.917 | ms | 5.425 | .914 | vvs | 5.407 | .918 | 13 | 5.431 | --- | --- | --- |
| 620 | .86 | 25 | 5.44 | --- | --- | --- | .856 | vs | 5.414 | .859 | 8 | 5.433 | --- | --- | --- |
| 533 | .83 | 10 | 5.44 | --- | --- | --- | .827 | s | 5.423 | .828 | 3 | 5.430 | --- | --- | --- |
| --- | --- | --- | --- | --- | --- | --- | .804 | ms | --- | --- | --- | --- | --- | --- | --- |
| 444 | 0.79 | 5 | 5.47 | --- | --- | --- | .782 | ms | 5.418 | 0.784 | 1 | 5.432 | --- | --- | --- |
| 711 | .76 | 10 | 5.43 | 0.759 | m | 5.420 | --- | --- | --- | .762 | 4 | 5.442 | --- | --- | --- |
| 642 | .73 | 20 | 5.46 | .724 | m | 5.418 | --- | --- | --- | .725 | 6 | 5.425 | --- | --- | --- |
| 731 | .71 | 15 | 5.45 | .706 | m | 5.423 | --- | --- | --- | .706 | 4 | 5.423 | --- | --- | --- |
| 822 | .64 | 5 | 5.43 | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- |
| 751 | .63 | 5 | 5.46 | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- |
| Average value of last 5 lines | | | 5.45 | --- | --- | 5.422 | --- | --- | 5.415 | --- | --- | 5.430 | --- | --- | 5.40 |

| hkl | 1941 Baumann | | | ---- United Steel Companies Cu, 1.5405 A | | | 1916 Debye and Scherrer Cu, 1.5405 A | | | 1953 Swanson and Fuyat Cu, 1.5405 A, 26°C | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | d | I | a | d | I | a | d | I | a | d | I | a |
| | A | | A | A | | A | A | | A | A | | A |
| --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- |
| 111 | 3.13 | s | 5.42 | 3.14 | 100 | 5.44 | 3.10 | s | 5.37 | 3.138 | 100 | 5.435 |
| 220 | 1.91 | s | 5.40 | 1.920 | 80 | 5.43 | 1.90 | s | 5.37 | 1.920 | 60 | 5.431 |
| 311 | 1.63 | ms | 5.41 | 1.637 | 70 | 5.429 | 1.64 | m-s | 5.44 | 1.638 | 35 | 5.433 |
| 222 | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- |
| 400 | 1.357 | w | 5.428 | 1.357 | 40 | 5.428 | 1.37 | m | 5.48 | 1.357 | 8 | 5.428 |
| 331 | 1.245 | m | 5.427 | 1.246 | 50 | 5.431 | 1.24 | m | 5.41 | 1.246 | 13 | 5.431 |
| 422 | 1.106 | s | 5.418 | 1.108 | 60 | 5.428 | 1.11 | m-s | 5.44 | 1.1083 | 17 | 5.4295 |
| 511 | 1.046 | m | 5.435 | 1.0448 | 50 | 5.4289 | 1.05 | m | 5.46 | 1.0450 | 9 | 5.4300 |
| 440 | 0.960 | m | 5.431 | 0.9597 | 50 | 5.4289 | 0.96 | w-m | 5.43 | 0.9599 | 5 | 5.4300 |
| 531 | .918 | s | 5.431 | .9178 | 70 | 5.4298 | .92 | m-s | 5.44 | .9178 | 11 | 5.4298 |
| 620 | .859 | s | 5.433 | [b].859 | 70 | 5.433 | .86 | m-s | 5.44 | .8586 | 9 | 5.4303 |
| 533 | .828 | m | 5.430 | [b].828 | 60 | 5.430 | .83 | m | 5.44 | .8281 | 5 | 5.4302 |
| --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- |
| 444 | --- | --- | --- | [b]0.784 | 60 | 5.432 | 0.78 | m | 5.40 | --- | --- | --- |
| 711 | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- |
| 642 | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- |
| 731 | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- |
| 822 | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- |
| 751 | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- |
| Average value of last 5 lines | | | 5.432 | --- | --- | 5.431 | --- | --- | 5.43 | --- | --- | 5.4301 |

[a] $K\alpha_2$ line, hkl 1.04, intensity 60 found on ASTM card not included in this table.

[b] These lines found only on new card, number 1924, 3-0552, 3-0529.

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## Gallium, Ga (orthorhombic)

### ASTM cards

| Card number | | New index lines | Radiation | Source |
|---|---|---|---|---|
| Old | New | | | |
| II-1061 | 1675 | 3.27 | Copper--- | Jaeger, Terpstra, and Westenbrink [1] 1928. |
| | 2-0488 | 1.99 | | |
| | 2-0480 | 1.95 | | |
| ------- | 2351 | 2.93 | Copper--- | Laves [2] 1933. |
| | 3-0676 | 1.99 | | |
| | 3-0666 | 1.95 | | |
| ------- | 2235 | 2.95 | Iron----- | Bradley [3] 1935. |
| | 3-0639 | 2.00 | | |
| | 3-0647 | 1.96 | | |

The ASTM cards of the Jaeger, Terpstra and Westenbrink pattern erroneously state that molybdenum radiation was used.

**Additional published patterns.** None.

**NBS pattern.** The gallium sample used for the NBS pattern was contributed by the Eagle-Picher Co., whose spectrographic analysis indicated impurities totaling 0.012 percent. The sample was purified according to the methods described by Hoffman [4] and Hoffman and Scribner [5].

Gallium, which melts at about 30°C, is quite plastic at room temperature, and difficulty was experienced in obtaining relatively finely divided material. When comminution was performed, using "dry ice" as the coolent, a powder passing the No. 200 U. S. Standard Sieve was obtained. Because this 200-mesh material did not permit reproducible intensity values and because an appreciably smaller particle size would have been very difficult to attain, it was deemed necessary that eight intensity patterns be prepared. Therefore, the reported intensities for the NBS gallium pattern are the averages of eight patterns.

**Interplanar spacings and intensity measurements.** *D*-spacings, in angstroms, for all three ASTM card patterns were calculated from the authors Bragg angle data. The three strongest lines for each pattern are as follows:

| Pattern | 1 | 2 | 3 |
|---|---|---|---|
| Jaeger, Terpstra, and Westenbrink---------- | 111 | 113 | 022, 202 |
| Laves---------- | 111 | 113 | 022, 202 |
| Bradley---------- | 111 | 113 | 211 |
| Swanson and Fuyat---------- | 111 | 113 | 020, 200 |

The Jaeger, Terpstra, and Westenbrink ASTM card value for the 111 line was incorrectly recorded, reading 3.27 rather than 3.20 as reported. The *d*-spacings 3.20 and 2.52 of this pattern are still in poor agreement with the other patterns, which give 2.95 and 2.96. The indexing of their intensity pattern corresponds generally with that of the *d*-spacings, but there are some discrepancies in the occurrence of pairs of *hkl*'s common to

| hkl | 1928 Jaeger, Terpstra, and Westenbrink Cu, 1.5405 A | | 1933 Laves Cu, 1.5405 A | | 1935 Bradley Fe, 1.93597 A | | 1953 Swanson and Fuyat Cu, 1.5405 A, 25°C | |
|---|---|---|---|---|---|---|---|---|
| | d | I | d | I | d | I | d | I |
| | A | | A | | A | | A | |
| 002 | 3.82 | 75 | 3.808 | m | 3.827 | 13 | 3.831 | 28 |
| 111 | 3.20 | 100 | 2.928 | s | 2.939 | 100 | 2.953 | 100 |
| 102 | ------- | --- | ------ | ---- | ------ | --- | 2.925 | 50 |
| 020 200 | 2.52 | 75 | 2.255 | s- | 2.253 | 33 | 2.262 | 60 |
| 113 | 1.996 | 88 | 1.992 | s | 1.992 | 70 | 1.996 | 85 |
| 211 | ------- | --- | ------ | ---- | 1.952 | 51 | 1.957 | 56 |
| 022 202 | 1.947 | 88 | 1.949 | s | ------ | --- | 1.947 | 17 |
| 004 | ------- | --- | 1 912 | m-w | 1.913 | 9 | 1.916 | 16 |
| 122 | 1.789 | 25 | 1.785 | m | 1.787 | 10 | 1.789 | 21 |
| 104 | ------- | --- | 1.759 | w | 1.763 | 4 | 1.763 | 6 |
| 220 | 1.600 | 25 | 1.594 | w-m | 1.598 | 7 | 1.599 | 11 |
| 213 | ------- | --- | ------ | ---- | ------ | --- | 1.586 | 3 |
| 222 | ------- | --- | ------ | ---- | ------ | --- | 1.476 | 3 |
| 024 204 | 1.458 | 25 | 1.459 | ms | 1.459 | 12 | 1.461 | 14 |
| 131 | ------- | --- | ------ | ---- | ------ | --- | 1.406 | 9 |
| 311 302 | 1.401 | 50 | 1.402 | s | 1.402 | 16 | 1.404 | 8 |
| 124 | ------- | --- | 1.391 | vw | 1.389 | 3 | 1.391 | 4 |
| 006 | ------- | --- | 1.276 | w+ | 1.276 | 6 | 1.2766 | 4 |
| 133 313 | ----- | --- | 1.243 | m | 1.246 | 15 | 1.2475 | 20 |
| 231 | ------- | --- | 1.234 | w-m | 1.236 | 12 | 1.2379 | 14 |
| 224 | ------- | --- | ------ | ---- | 1.227 | 4 | 1.2276 | 5 |
| 215 | ------- | --- | 1.220 | m+ | 1.220 | 16 | 1.2216 | 17 |
| 322 | ------- | --- | ------ | ---- | 1.192 | 18 | 1.1928 | 15 |
| 304 | ------- | --- | 1.186 | m-s | 1.184 | 6 | 1.1853 | 4 |
| 040 400 | ----- | --- | 1.127 | w+ | 1.129 | 6 | 1.1302 | 5 |
| 026 206 | ----- | --- | 1.109 | w-m | 1.112 | 13 | 1.1119 | 8 |
| 411 | ------- | --- | 1.085 | m | 1.087 | 13 | 1.0866 | 3 |
| 142 | ------- | --- | ------- | ---- | 1.054 | 6 | 1.0540 | 1 |
| 324 | ------- | --- | 1.046 | w+ | 1.049 | 13 | 1.0496 | 3 |
| 117 | ------- | --- | 1.033 | w-m | 1.035 | 19 | 1.0355 | 4 |
| 240 420 | ----- | --- | 1.009 | vw | 1.011 | 9 | 1.0111 | 2 |
| 226 | ------- | --- | 0.9954 | vw | 0.9976 | 10 | 0.9976 | 2 |
| 242 422 | ----- | --- | ------ | ---- | ------ | --- | .9775 | 1 |
| 044 404 | ----- | --- | ------ | ---- | ------ | --- | .9735 | 5 |
| 235 | ------- | --- | 0.9687 | w-m | ------ | --- | .9706 | 7 |
| 217 | ------- | --- | .9612 | vw | ------ | --- | .9626 | 3 |



| hkl | 1928 Jaeger, Terpstra, and Westenbrink Cu, 1.5405 A | | 1933 Laves Cu, 1.5405 A | | 1935 Bradley Fe, 1.93597 A | | 1953 Swanson and Fuyat Cu, 1.5405 A, 25°C | |
|---|---|---|---|---|---|---|---|---|
| | d | I | d | I | d | I | d | I |
| | A | | A | | A | | A | |
| 144 | ------- | --- | 0.9491 | vw | ------ | --- | 0.9515 | 1 |
| 108 | ------- | --- | .9347 | vvw | ------ | --- | .9369 | 1 |
| 431 | ------- | --- | ------ | ---- | ------ | --- | .8986 | 4 |
| 326 | ------- | --- | 0.8963 | w? | ------ | --- | .8948 | 2 |
| 415 | ------- | --- | .8907 | w-m? | ------ | --- | .8923 | 4 |
| 028 208 | ----- | --- | ------ | ---- | ------ | --- | .8817 | 7 |
| 342 | ------- | --- | 0.8779 | m? | ------ | --- | .8802 | 7 |
| 137 317 | ----- | --- | ------ | ---- | ------ | --- | .8690 | 4 |
| 128 | ------- | --- | 0.8642 | w-m | ------ | --- | .8654 | 3 |
| 153 | ------- | --- | ------ | ---- | ------- | --- | .8383 | 8 |
| 513 | ------- | --- | ------ | ---- | ------ | --- | .8376 | 4 |
| 237 | ------- | --- | ------ | ---- | ------ | --- | .8247 | 5 |
| 344 | ------- | --- | ------ | --- | ------- | --- | .8179 | 6 |
| 308 | ------- | --- | ------ | ---- | ------ | --- | .8084 | 3 |

one $d$-spacing; probably due to the radiation used, different pairs of lines were resolved with each type of target. The structure is so nearly tetragonal that only two pairs of lines with indices 131, 311 and 153, 513 can be resolved in the NBS pattern. By calculating intensity values for each member of the unresolved pairs, it was definitely proven that in each case both contributed to the observed value.

**Lattice constants.** The structure was determined by Laves [2] in 1932. The space group is $D_{2h}^{18}$-Abma (Cmca) with 8 (Ga) per unit cell. Black phosphorus and solid bromine and iodine have the same structure.

Several unit-cell measurements have been converted to angstroms for comparison with the NBS values.

*Lattice constants in angstroms*

| | | a | b | c |
|---|---|---|---|---|
| 1933 | Laves [2] | 4.515 | 4.515 | 7.657 |
| 1935 | Bradley [3] | 4.5258 | 4.5198 | 7.6602 |
| 1953 | Swanson and Fuyat | 4.524 | 4.523 | 7.661 at 25°C |

The density of gallium calculated from the NBS lattice constant is 5.907 at 25°C.

## References


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## Alpha-zirconium, Zr (hexagonal)

### ASTM cards

| Card number | | New index lines | Radiation | Source |
|---|---|---|---|---|
| Old | New | | | |
| II-1716 | 2556 | 2.81 | Zinc, | Noethling and |
| | 2-0819 | 1.84 | 1.43510. | Tolksdorf [1] 1925. |
| | 2-0821 | 2.60 | | |
| 2941 | 3123 | 2.44 | Molybdenum | Hanawalt, Rinn, and |
| | 1-1150 | 2.78 | | Frevel [2] 1938. |
| | 1-1147 | 2.56 | | |

**Additional published patterns.** None.

**NBS pattern.** The zirconium sample used for the NBS pattern was furnished by the Johnson-Matthey & Co., Ltd. The following impurities were found by spectrographic analysis at the Bureau: 0.01 to 0.1 percent of hafnium, 0.001 to 0.01 percent each of titanium and iron, and 0.0001 to 0.001 percent each of magnesium, copper, silicon, aluminum, silver, and calcium.

**Interplanar spacings and intensity measurements.** The Noethling and Tolksdorf *d*-spacings expressed in angstroms were calculated from their Bragg angle data and their wavelength value for zinc radiation. The Hanawalt, Rinn, and Frevel *d*-spacings were converted from kX to angstrom units.

The three strongest lines for each pattern are as follows:

| Pattern | 1 | 2 | 3 |
|---|---|---|---|
| Noethling and Tolksdorf | 100 | 102 | 002 |
| Hanawalt, Rinn, and Frevel | 101 | 100 | 002 |
| Swanson and Fuyat | 101 | 100 | 002 |

The first line of the Noethling and Tolksdorf ASTM card pattern is not found in their published data.

**Lattice constants.** The structure was determined by Hull [3] in 1921. The hexagonal close packed lattice has space group $D^4_{6h}$-C6/mmc with 2(Zr) per unit cell. It is generally accepted that zirconium becomes body centered cubic at 850°C.

### Alpha-zirconium, Zr

| *hkl* | 1925 Noethling and Tolksdorf Mo, 0.709 A | | 1938 Hanawalt, Rinn, and Frevel Mo, 0.709 A | | 1953 Swanson and Fuyat Cu, 1.5405 A, 25°C | |
|---|---|---|---|---|---|---|
| | *d* | *I* | *d* | *I* | *d* | *I* |
| | A | | A | | A | |
| 100 | [a] 2.817 | [a] 100 | 2.79 | 31 | 2.798 | 33 |
| 002 | 2.602 | s | 2.57 | 20 | 2.573 | 32 |
| 101 | 2.376 | w | 2.44 | 100 | 2.459 | 100 |
| 102 | 1.844 | vs | 1.88 | 18 | 1.894 | 17 |
| 110 | 1.609 | s | 1.61 | 18 | 1.616 | 17 |
| 103 | 1.527 | s | 1.463 | 18 | 1.463 | 18 |
| 200 | -------- | ------ | ------- | ------ | 1.399 | 3 |
| 112 | 1.365 | w | 1.363 | 15 | 1.368 | 18 |
| 201 | -------- | ------ | 1.348 | 10 | 1.350 | 12 |
| 004 | 1.293 | w | 1.285 | 5 | 1.287 | 4 |
| 202 | -------- | ------ | 1.222 | 3B | 1.2296 | 4 |
| 104 | -------- | ------ | 1.182 | 3B | 1.1689 | 3 |
| 203 | -------- | ------ | 1.084 | 5 | 1.0842 | 4 |
| 210 | 1.058 | w | ------- | ------ | 1.0588 | 2 |
| 211 | -------- | ------ | 1.040 | 8 | 1.0360 | 6 |
| 114 | -------- | ------ | 1.005 | 3 | 1.0063 | 3 |
| 212 | -------- | ------ | 0.979 | 3 | 0.9783 | 2 |
| 105 | -------- | ------ | ------- | ------ | .9660 | 4 |
| 204 | -------- | ------ | ------- | ------ | .9474 | 2 |
| 300 | -------- | ------ | ------- | ------ | .9327 | 3 |
| 213 | -------- | ------ | 0.900 | 3 | .9003 | 5 |
| 302 | -------- | ------ | .879 | 3 | .8771 | 3 |
| 006 | -------- | ------ | ------- | ------ | .8577 | 1 |
| 205 | -------- | ------ | ------- | ------ | .8292 | 2 |
| 106 | -------- | ------ | ------- | ------ | .8201 | 2 |
| 214 | 0.816 | ms | ------- | ------ | ------ | ----- |
| 220 | -------- | ------ | ------- | ------ | ------ | ----- |
| ------- | -------- | ------ | ------- | ------ | ------ | ----- |
| ------- | 0.777 | s | ------- | ------ | ------ | ----- |

[a] This line is not found in the original data.

Several unit cell determinations have been converted to angstroms for comparison with the NBS values.

*Lattice constants in angstroms*

| | | a | c |
|---|---|---|---|
| 1921 | Hull [3] | 3.24 | 5.15 |
| 1927 | Van Arkel [4] | 3.230 | 5.133 |
| 1930 | Hägg [5] | 3.236 | 5.151 |
| 1934 | Shinoda [6] | 3.24 | 5.15 |
| 1952 | Fast [7] | 3.225 | 5.134 |
| 1953 | Swanson and Fuyat | 3.232 | 5.147 at 25°C |

The linear coefficient of expansion from 15 to 100°C is $2.5 \times 10^{-6}$ parallel to the $a$ axis and $14.3 \times 10^{-6}$ perpendicular to it, according to Shinoda [6].

The density of α-zirconium calculated from the NBS lattice constant is 6.505 at 25°C.

**References**


[1] W. Noethling and S. Tolksdorf, Die Kristallstruktur des Hafniums, Z. Krist. **62**, 258 (1925).

[2] J. D. Hanawalt, H. W. Rinn and L. K. Frevel, Chemical analysis by X-ray diffraction, Ind. and Eng. Chem., Anal. Ed. **10**, 457-512 (1938).

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[7] J. D. Fast, The Allotropic transformation of hafnium and a tentative equilibrium diagram of the system zirconium-hafnium. J. Appl. Phys. **23**. 350-51 (1952).


## Alpha-tin, α-Sn (cubic)

**ASTM cards.** None.

**Additional published patterns**

| Source | Radiation | Wavelength |
|---|---|---|
| Bijl and Kolkmeijer [1] 1919 | Copper | 1.541 |

**NBS pattern.** The alpha-tin was prepared by James Becker of the Solid State Section at the NBS from beta-tin (NBS Melting Point Standard Sample 42D) by seeding with a small quantity of alpha-tin. Spectrographic analysis at the Bureau showed 0.0006 percent of copper, 0.0056 percent of iron, and less than 0.005 percent of lead. The sample was refrigerated until shortly before use to insure a minimum of beta lines. [5]

**Interplanar spacings and intensity measurements.** The Bijl and Kolkmeijer $d$-spacings, expressed in angstroms, were calculated from their Bragg angle data.

The three strongest lines for the NBS pattern are as follows:

| Pattern | 1 | 2 | 3 |
|---|---|---|---|
| Swanson and Fuyat | 111 | 220 | 311 |

The intensities of the Bijl and Kolkmeijer pattern are not comparable due to specimen absorption.

**Alpha-tin, α-Sn**

| hkl | 1919 Bijl and Kolkmeijer Cu, 1.5405 A | | | 1953 Swanson and Fuyat Cu, 1.5405 A, 25°C | | |
|---|---|---|---|---|---|---|
| | d | I | a | d | I | a |
| | A | | A | A | | A |
| 111 | 2.94 | w | 5.092 | 3.751 | 100 | 6.497 |
| 220 | 2.25 | m | 6.364 | 2.294 | 83 | 6.488 |
| 311 | 1.95 | m | 6.467 | 1.956 | 53 | 6.487 |
| 400 | 1.608 | w | 6.432 | 1.622 | 12 | 6.488 |
| 331 | 1.473 | m | 6.421 | 1.489 | 20 | 6.490 |
| 422 | 1.314 | s | 6.437 | 1.325 | 21 | 6.491 |
| 511 | 1.225 | m | 6.365 | 1.249 | 11 | 6.490 |
| 440 | 1.134 | w | 6.415 | 1.1470 | 6 | 6.488 |
| 531 | 1.093 | m | 6.466 | 1.0968 | 10 | 6.499 |
| 620 | 1.024 | s | 6.476 | 1.0260 | 9 | 6.489 |
| 533 | 0.985 | w | 6.459 | 0.9895 | 4 | 6.489 |
| 444 | .931 | w | 6.450 | .9365 | 3 | 6.488 |
| 711 | .905 | m | 6.463 | .9087 | 7 | 6.489 |
| 642 | .865 | vs | 6.473 | .8671 | 13 | 6.489 |
| 731 | .843 | vs | 6.475 | .8450 | 12 | 6.491 |
| 800 | .813 | w | 6.504 | ------- | --- | ------- |
| 733 | .791 | m | 6.427 | ------- | --- | ------- |
| Average value of a for last five lines | | | 6.468 | ------- | --- | 6.489 |

[5] The conversion to beta-tin takes place at 13.2°C according to Cohen and Lieshout [2], but the initial rate of transformation at room temperature is sufficiently slow that it does not interfere with analysis as no beta-tin lines were visible.

**Lattice constants.** Bijl and Kolkmeijer [1] determined the structure in 1918. The lattice is modified face-centered cubic, $O_h^7$-Fd3m, diamond structure, with 8(Sn) per unit cell. White or beta-tin, stable at room temperature, is tetragonal.

Two unit-cell determinations have been converted to angstroms for comparison with the NBS values.

*Lattice constant in angstroms*

| | | $a$ |
|---|---|---|
| 1919 | Bijl and Kolkmeijer [1] | 6.46 at 18°C |
| 1950 | Brownlee [3] | 6.4912 |
| 1953 | Swanson and Fuyat | 6.489 at 25°C[a] |

[a] See footnote 5, p. 12.

The density of alpha-tin calculated from the NBS lattice constants is 5.770 at 25°C.

**References**


[1] A. J. Bijl and N. H. Kolkmeijer, Investigation by means of X-rays of the crystal structure of white and gray tin III, Proc. Acad. Sci., Amsterdam, **21**, 501-4 (1919).

[2] E. Cohen and A. K. W. A. van Lieshout, Die Geschwindigkeit polymorpher Umwandlungen, Physikalisch-Chemische Studien am Zinn, Z. Phys. Chem. **173A**, 1-34 (1938).

[3] L. D. Brownlee, Lattice constant of gray tin, Nature **166**, No. 4220, 482 (1950).


## Rhenium, Re (hexagonal)

**ASTM cards**

| Card number | | New index lines | Radiation | Source |
|---|---|---|---|---|
| Old | New | | | |
| II-4088 | 3943 | 1.18 | Copper---- | Agte, Alterthum, |
| | 2-1440 | 2.12 | | Becker, Heyne, and |
| | 2-1437 | 1.39 | | Moers [1] 1931. |
| 3283 | 3387 | 2.10 | Molybdenum | Hanawalt, Rinn, and |
| | 1-1226 | 2.38 | | Frevel [2] 1938. |
| | 1-1231 | 2.22 | | |

**Additional published patterns**

| Source | Radiation | Wavelength |
|---|---|---|
| Goldschmidt [3] 1929 | Copper----- | 1.539 |

**NBS pattern.** The rhenium sample used for the NBS pattern was prepared by Dr. A. D. Melaven at the University of Tennessee in Knoxville. Spectrographic analysis at the Bureau showed 0.01 to 0.1 percent each of silver, aluminum, copper, and iron and 0.001 to 0.01 percent each of calcium, magnesium, nickel, and silicon.

## Rhenium, Re

| *hkl* | 1931 Agte, Alterthum, Becker, Heyne, and Moers Mo, 0.709 A | | 1938 Hanawalt, Rinn, and Frevel Mo, 0.709 A | | 1929 Goldschmidt Cu, 1.5405A | | 1953 Swanson and Fuyat Cu, 1.5405, A, 26°C | |
|---|---|---|---|---|---|---|---|---|
| | *d* | *I* | *d* | *I* | *d* | *I* | *d* | *I* |
| | *A* | | *A* | | *A* | | *A* | |
| 100 | 2.430 | vw | 2.38 | 33 | 2.433 | 30 | 2.388 | 32 |
| 002 | 2.246 | w | 2.22 | 20 | 2.238 | 40-50 | 2.226 | 34 |
| 101 | 2.132 | m | 2.10 | 100 | 2.122 | 100 | 2.105 | 100 |
| 102 | 1.641 | vw | 1.62 | 10 | 1.633 | 40 | 1.629 | 11 |
| 110 | 1.397 | m | 1.382 | 17 | 1.392 | 70 | 1.380 | 22 |
| 103 | 1.271 | m | 1.263 | 13 | 1.267 | 80 | 1.262 | 16 |
| 200 | ----- | ----- | ----- | ---- | 1.200 | 10 | 1.1948 | 3 |
| 112 | 1.187 | ms | 1.172 | 17 | 1.162 | 80 | 1.1730 | 20 |
| 201 | 1.164 | m | 1.153 | 17 | 1.160 | 70 | 1.1540 | 15 |
| 004 | ----- | ----- | ----- | ---- | 1.120 | 20 | 1.1142 | 2 |
| 202 | 1.062 | vm | ----- | ---- | 1.046 | 30 | 1.0530 | 3 |
| 104 | 1.016 | w | ----- | ---- | 1.013 | 20 | 1.0099 | 2 |
| 203 | 0.938 | s | 0.930 | 3 | 0.929 | 60 | 0.9311 | 7 |
| 210 | .906 | w | ----- | ---- | .905 | 30 | .9033 | 3 |
| 211 | .888 | vs | 0.886 | 7 | .886 | 80 | .8854 | 15 |
| 114 | .871 | s | .870 | 3 | .868 | 70 | .8671 | 8 |
| 212 | ----- | ----- | ----- | ---- | ----- | ----- | .8373 | 5 |
| 105 | 0.836 | vs | ----- | ---- | 0.835 | 60 | .8354 | 8 |
| 204 | .819 | ms | ----- | ---- | ----- | ----- | .8151 | 2 |
| 300 | .800 | s-vs | ----- | ---- | ----- | ----- | .7968 | 5 |

**Interplanar spacings and intensity measurements.** The Agte, Alterthum, Becker, Heyne, and Moers and the Goldschmidt $d$-spacings were calculated from Bragg angle data, and the Hanawalt, Rinn, and Frevel $d$-spacings were converted from kX to angstrom units.

The three strongest lines for each of the patterns are as follows:

| Pattern | 1 | 2 | 3 |
|---|---|---|---|
| Agte, Alterthum, etc. | 211 | 105 | 300 |
| Hanawalt, Rinn, and Frevel | 101 | 100 | 002 |
| Goldschmidt | 101 | 103 | 112 |
| Swanson and Fuyat | 101 | 002 | 100 |

**Lattice constants.** The structure was determined by Goldschmidt [3] in 1929. The

space group is $D^4_{6h}$-C6/mmc, hexagonal close packed, with 2(Re) per unit cell.

A group of unit-cell determinations were converted from kX to angstrom units for comparison with the NBS values.

*Lattice constants in angstroms*

| | | *a* | *c* |
|---|---|---|---|
| 1929 | Goldschmidt [3] | 2.758 | 4.457 |
| 1931 | Agte, Alterthum, Becker, Heyne, and Moers [1] | 2.771 | 4.479 |
| 1931 | Moeller [4] | 2.761 | 4.459 |
| 1932 | Stenzel and Weerts [5] | 2.7608 | 4.4582 |
| 1953 | Swanson and Fuyat | 2.760 | 4.458 at 26°C |

The density of rhenium calculated from the NBS lattice constant is 21.034 at 26°C. The linear coefficient of expansion is $12.45 \times 10^{-6}$ parallel to the *c*-axis and $4.67 \times 10^{-6}$ perpendicular to it, according to Agte, Alterthum, Becker, Heyne, and Moers [1].

**References**


[1] C. Agte, H. Alterthum, K. Becker, G. Heyne, K. Moers, Physikalische und chemische Eigenschaften des Rheniums, Z. anorg. allg. Chem. **196**, 129-59 (1931).

[2] J. D. Hanawalt, H. W. Rinn and L. K. Frevel, Chemical analysis by X-ray diffraction, Ind. and Eng. Chem., Anal. Ed. **10**, 457-512 (1938).

[3] V. M. Goldschmidt, Kristallstruktur, Gitterkonstanten und Dichte des Rheniums, Z. Phys. Chem, **B2**, 244-52 (1929).

[4] K. Moeller, Zu den Gitterkonstanten des Rheniums, Naturwiss. **19**, 575 (1931).

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## 2.2. Sulfides

### Alpha-zinc sulfide (wurtzite), α-ZnS (hexagonal)

**ASTM cards**

| Card number | | New index lines | Radiation | Source |
|---|---|---|---|---|
| Old | New | | | |
| ------- | 3680 | 1.90 | Iron------ | Aminoff [1] 1923. |
| | 3-1091 | 1.62 | | |
| | 3-1093 | 1.04 | | |
| II-3104 | 3671 | 1.91 | Cu, 1.539 | Ulrich and Zachariasen [2] 1925. |
| | 2-1303 | 1.76 | | |
| | 2-1310 | 3.30 | Molybdenum | Fuller [3] 1929. |
| 3503 | 3672 | 1.90 | Molybdenum | Fuller [3] 1929. |
| | 1-1280 | 1.76 | | |
| | 1-1280 | 3.29 | | |

**ASTM cards—Con.**

| Card number | | New index lines | Radiation | Source |
|---|---|---|---|---|
| Old | New | | | |
| 1671 | 1636 | 3.29 | Molybdenum | Hanawalt, Rinn, and Frevel [4] 1938. |
| | 1-0694 | 2.91 | | |
| | 1-0677 | 1.76 | | |

The old and new ASTM cards for the Ulrich and Zachariasen pattern erroneously state that molybdenum radiation was used.

**Additional published patterns.** None.

**NBS pattern.** The alpha-zinc sulfide sample used for the NBS pattern was prepared at 1,200°C by solid-state reaction techniques at the Radio Corporation of America Laboratories. Spectrographic analysis at the Bureau showed 0.01 to 0.1 percent of copper, 0.001 to 0.01 percent each of boron, iron, magnesium, and silicon, and less than 0.001 percent each of aluminum and calcium.

**Interplanar spacings and intensity measurements.** The Aminoff and the Ulrich and Zachariasen *d*-spacings expressed in angstroms, were calculated from their Bragg angle data. The Fuller and the Hanawalt, Rinn, and Frevel *d*-spacings were converted from kX to angstrom units.

The three strongest lines for each pattern are as follows:

| Pattern | 1 | 2 | 3 |
|---|---|---|---|
| Aminoff | 110 | 112 | 302 |
| Ulrich and Zachariasen | 110 | 112 | 002 |
| Fuller | 110 | 103 | 100 |
| Hanawalt, Rinn, and Frevel | 100 | 101 | 103 |
| Swanson and Fuyat | 100 | 002 | 101 |

**Lattice constants.** The structure was determined by W. L. Bragg [5] in 1920. The space group is $C^4_{6v}$-C6mc, zinc oxide or wurtzite-type structure, with 2(ZnS) per unit cell. It is generally accepted that alpha-zinc sulfide is stable above 1,020°C; beta-zinc sulfide, the cubic form, is stable below 1,020°C.

A number of unit-cell measurements have been converted to angstroms for comparison with the NBS values.

| hkl | 1923 Aminoff Fe, 1.93597 A | | 1925 Ulrich and Zachariasen Mo, 0.709 A | | 1929 Fuller Mo, 0.709 A | | 1938 Hanawalt, Rinn, and Frevel Mo, 0.709 A | | 1953 Swanson and Fuyat Cu, 1.5405 A, 26°C | |
|---|---|---|---|---|---|---|---|---|---|---|
| | d | I | d | I | d | I | d | I | d | I |
| | A | | A | | A | | A | | A | |
| 100 | 3.28 | 50 | 3.31 | 40 | 3.30 | 80 | 3.30 | 100 | 3.309 | 100 |
| 002 | 3.12 | 75 | 3.13 | 80 | 3.12 | 24 | 3.12 | 47 | 3.128 | 86 |
| 101 | 2.92 | 25 | 2.94 | 40 | 2.92 | 76 | 2.92 | 100 | 2.925 | 84 |
| 102 | ---------- | ---------- | 2.29 | 30 | 2.27 | 20 | 2.27 | 47 | 2.273 | 29 |
| 110 | 1.90 | 100 | 1.92 | 100 | 1.908 | 100 | 1.90 | 83 | 1.911 | 74 |
| 103 | 1.79 | 25 | 1.77 | 60 | 1.763 | 100 | 1.76 | 100 | 1.764 | 52 |
| 200 | ---------- | ---------- | --------- | --------- | 1.654 | 1 | --------- | --------- | 1.654 | 10 |
| 112 | 1.63 | 100 | 1.638 | 90 | 1.631 | 32 | 1.62 | 67 | 1.630 | 45 |
| 201 | ---------- | ---------- | 1.611 | 10 | 1.596 | 8 | 1.59 | 33 | 1.599 | 12 |
| 004 | ---------- | ---------- | --------- | --------- | --------- | --------- | --------- | --------- | 1.564 | 2 |
| 202 | ---------- | ---------- | 1.477 | 10-20 | 1.461 | 4 | 1.462 | 27 | 1.462 | 5 |
| 104 | ---------- | ---------- | --------- | --------- | --------- | --------- | --------- | --------- | 1.414 | 1 |
| 203 | 1.30 | 12.5-25 | 1.302 | 40 | 1.295 | 20 | 1.295 | 40 | 1.296 | 14 |
| 210 | 1.25 | 25 | 1.250 | 50 | 1.252 | 4 | 1.252 | 13 | 1.251 | 6 |
| 211 | ---------- | ---------- | 1.233 | 20 | 1.226 | 4 | 1.226 | 27 | 1.226 | 3 |
| 114 | 1.21 | 25 | --------- | --------- | --------- | --------- | --------- | --------- | 1.210 | 10 |
| 105 | ---------- | ---------- | 1.177 | 40 | 1.168 | 12 | 1.170 | 33 | 1.1703 | 4 |
| 212 | 1.16 | 25 | 1.161 | 20 | --------- | --------- | --------- | --------- | 1.1611 | 8 |
| 204 | ---------- | ---------- | --------- | --------- | --------- | --------- | --------- | --------- | 1.1364 | <1 |
| 300 | 1.10 | 75 | 1.109 | 80 | 1.101 | 8 | 1.101 | 20 | 1.1029 | 13 |
| 213 | 1.07 | 25 | 1.078 | 70 | 1.070 | 24 | 1.072 | 47 | 1.0724 | 6 |
| 302 | 1.04 | 100 | 1.046 | 80 | 1.040 | 4 | 1.041 | 13 | 1.0401 | 5 |
| 205 | 1.01 | 25 | 1.002 | 40 | 0.997 | 4 | 0.999 | 7 | 0.9979 | 6 |
| 214 | ---------- | ---------- | --------- | --------- | --------- | --------- | --------- | --------- | .9766 | <1 |
| 220 | ---------- | ---------- | --------- | --------- | 0.957 | 1 | 0.957 | 7 | .9551 | 6 |
| 310 | ---------- | ---------- | --------- | --------- | --------- | --------- | --------- | --------- | .9175 | 5 |
| 116 | ---------- | ---------- | --------- | --------- | 0.914 | 8 | 0.916 | 20 | .9151 | 7 |
| 311 | ---------- | ---------- | --------- | --------- | --------- | --------- | --------- | --------- | .9080 | 2 |
| 215 | ---------- | ---------- | --------- | --------- | --------- | --------- | 0.885 | 20 | .8845 | 8 |
| 313 | ---------- | ---------- | --------- | --------- | --------- | --------- | --------- | --------- | .8398 | 9 |

*Lattice constants in angstroms*

| | | a | c |
|---|---|---|---|
| 1923 | Aminoff [1] | 3.81 | 6.24 |
| 1925 | Ulrich and Zachariasen [2] | 3.844 | 6.290 |
| 1929 | Fuller [3] | 3.819 | 6.247 |
| 1953 | Swanson and Fuyat | 3.820 | 6.260 at 26°C |

The density of alpha-zinc sulfide calculated from the NBS lattice constant is 4.089 at 26°C. The linear coefficient of expansion is 6.14 or $6.18 \times 10^{-6}$ (2 samples), according to Adenstadt [6]. The refractive index is too high to be measured by ordinary liquid grain immersion methods.

## References


[1] G. Aminoff, Untersuchungen über die Kristallstrukturen von Wurtzit und Rotnickelkies, Z. Krist. **58**, 203-219 (1923).

[2] F. Ulrich and W. Zachariasen, Über die Kristallstruktur des α- und β-CdS, sowie des Wurtzits, Z. Krist. **62**, 260-73 (1925).

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[5] W. L. Bragg, The crystalline structure of zinc oxide, Phil. Mag. **39**, 647 (1920).

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## Beta-zinc sulfide (sphalerite), β-ZnS (cubic)

### ASTM cards

| Card number | | New index lines | Radiation | Source |
|---|---|---|---|---|
| Old | New | | | |
| ------- | 2022 | 3.07 | Iron------ | Aminoff [1] 1923. |
| | 3-0588 | 1.90 | | |
| | 3-0579 | 1.63 | | |
| ------- | 1846 | 3.16 | Copper---- | Lehmann [2] 1924. |
| | 3-0523 | 1.93 | | |
| | 3-0524 | 1.64 | | |
| II-1232 | 1926 | 3.12 | Molybdenum | Blake [3] 1934. |
| | 2-0576 | 1.92 | | |
| | 2-0564 | 1.56 | | |
| 1913 | 1930 | 3.12 | Molybdenum | Hanawalt, Rinn, and Frevel [4] 1938. |
| | 1-0800 | 1.91 | | |
| | 1-0792 | 1.63 | | |
| ------- | 2021 | 3.09 | Copper---- | British Museum. |
| | 3-0587 | 1.91 | | |
| | 3-0570 | 1.63 | | |
| II-1233 | 1928 | 3.12 | Copper---- | Harcourt [5] 1942. |
| | 2-0577 | 1.91 | | |
| | 2-0565 | 1.63 | | |

### Additional published patterns

| Source | Radiation | Wavelength |
|---|---|---|
| Gerlach [6] 1922---------- | Copper----- | ---------- |

**NBS pattern.** The beta-zinc sulfide sample used for the NBS pattern was prepared at 940°C by the Radio Corporation of America Laboratories. Spectrographic analysis at the Bureau showed 0.01 to 0.1 percent of copper, 0.001 to 0.01 percent of boron, iron, magnesium, and silicon, and less than 0.001 percent of aluminum and calcium.

**Interplanar spacings and intensity measurements.** The *d*-spacings, expressed in angstroms for the Aminoff, Lehmann, Blake, and Gerlach patterns, were calculated from their Bragg angle data, while the Hanawalt, Rinn, and Frevel and the Harcourt *d*-spacings from the literature and the British Museum *d*-spacings from the ASTM card file were converted from kX to angstrom units. The 420 line, 1.15 A, of intensity 30 found on Aminoff's ASTM card pattern does not appear in his published data. The unit cell calculated from that *d*-spacing is in such poor agreement with the others that it undoubtedly does not belong to the pattern, although a 420 line appears around 1.20 A in the NBS pattern and a number of the others. Gerlach published two very similar patterns, of which only one appears in the table.

The three strongest lines for each of the patterns are as follows:

| Pattern | 1 | 2 | 3 |
|---|---|---|---|
| All patterns---------- | 111 | 220 | 311 |

**Lattice constants.** The structure was determined by W. H. and W. L. Bragg [7,8] in 1913. The space group is $T_d^2$-F$\bar{4}$3m, sphalerite-type structure, with 4(ZnS) per unit cell. The rare hexagonal form, wurtzite, is stable above 1,020°C, and sphalerite is stable at room temperature.

A group of unit-cell measurements supposedly expressed in kX units have been converted to angstroms for comparison with the NBS value.

*Lattice constant in angstroms*

| | | |
|---|---|---|
| 1922 | Gerlach [6]---------- | 5.405 |
| 1924 | Lehmann [2]---------- | 5.448 |
| 1927 | de Jong [9]---------- | 5.406 |
| 1934 | Braekken [10]---------- | 5.414 |
| 1936 | Moltzau and Kolthoff [11]----- | 5.41 |
| 1953 | Swanson and Fuyat---------- | 5.4060 at 26°C |

The density of beta-zinc sulfide calculated from the NBS lattice constant is 4.096 at 26°C. The refractive index is too high to be measured by the usual liquid grain immersion methods.

| hkl | 1923 Aminoff Fe, 1.93597 A | | | 1924 Lehmann Cu, 1.5405 A | | | 1934 Blake Mo, 0.709 A | | | 1938 Hanawalt, Rinn, and Frevel Mo, 0.709 A | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | d | I | a | d | I | a | d | I | a | d | I | a |
| | A | | A | A | | A | A | | A | A | | A |
| 111 | 3.078 | 100 | 5.331 | 3.16 | 100 | 5.47 | 3.123 | 100 | 5.409 | 3.13 | 100 | 5.42 |
| 200 | 2.694 | 25 | 5.388 | 2.74 | D | 5.48 | | | | 2.70 | 5 | 5.40 |
| 220 | 1.898 | 100 | 5.368 | 1.93 | 100 | 5.46 | 1.917 | 52.8 | 5.422 | 1.91 | 75 | 5.40 |
| 311 | 1.629 | 100 | 5.403 | 1.64 | 100 | 5.44 | 1.636 | 48.4 | 5.426 | 1.63 | 50 | 5.41 |
| 222 | 1.562 | 25 | 5.411 | 1.58 | 20 | 5.47 | | | | | | |
| 400 | 1.353 | 50 | 5.412 | 1.36 | 40 | 5.44 | 1.356 | 5.3 | 5.424 | 1.356 | 5 | 5.424 |
| 331 | 1.240 | 75 | [b]5.405 | 1.25 | 90 | 5.45 | 1.244 | 16.3 | 5.422 | 1.245 | 18 | 5.427 |
| 420 | [a]1.15 | 30 | [b]5.14 | 1.21 | 90 | 5.41 | | | | 1.213 | 3 | 5.425 |
| 422 | 1.104 | 100 | 5.408 | 1.11 | 100 | 5.44 | 1.107 | 18.5 | 5.423 | 1.106 | 15 | 5.418 |
| 511 | 1.042 | 100 | 5.414 | 1.04 | 90 | 5.40 | 1.044 | 8.8 | 5.425 | 1.046 | 5 | 5.435 |
| 440 | | | | 0.961 | 80 | 5.442 | 0.959 | 5.3 | 5.425 | 0.959 | 3 | 5.425 |
| 531 | | | | .916 | 90 | 5.419 | .917 | 4.4 | 5.425 | .915 | 4 | 5.413 |
| 620 | | | | .856 | 70 | 5.420 | .858 | 3.2 | 5.426 | | | |
| 533 | | | | .824 | 60 | 5.410 | | | | | | |
| 622 | | | | .801 | 60 | 5.313 | | | | | | |
| Average value of last five lines | | | 5.410 | | | 5.421 | | | 5.425 | | | 5.423 |

| hkl | ---- British Museum Cu, 1.5405 A | | | 1942 Harcourt Mo, 0.709 A | | | 1922 Gerlach Cu, 1.5405 A | | | 1953 Swanson and Fuyat Cu, 1.5405 A, 26°C | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | d | I | a | d | I | a | d | I | a | d | I | a |
| | A | | A | A | | A | A | | A | A | | A |
| | | | | 3.96 | 8 | | | | | | | |
| 111 | 3.10 | 100 | 5.37 | 3.13 | 100 | 5.42 | 3.097 | s | 5.364 | 3.123 | 100 | 5.409 |
| 200 | 2.71 | 40 | 5.42 | 2.71 | 33 | 5.42 | 2.670 | w | 5.340 | 2.705 | 10 | 5.410 |
| 220 | 1.91 | 100 | 5.40 | 1.91 | 83 | 5.40 | 1.894 | st | 5.357 | 1.912 | 51 | 5.408 |
| 311 | 1.63 | 80 | 5.41 | 1.63 | 67 | 5.41 | 1.620 | s | 5.373 | 1.633 | 30 | 5.416 |
| 222 | 1.56 | 40 | 5.40 | 1.558 | 8 | 5.397 | 1.575 | w | 5.456 | 1.561 | 2 | 5.407 |
| 400 | 1.35 | 60 | 5.40 | 1.353 | 33 | 5.412 | 1.350 | ms | 5.400 | 1.351 | 6 | 5.404 |
| 331 | 1.30 | 70 | | 1.244 | 50 | 5.422 | 1.233 | s | 5.375 | 1.240 | 9 | 5.405 |
| 420 | 1.21 | 40 | 5.41 | 1.212 | 8 | 5.420 | | | | 1.209 | 2 | 5.407 |
| 422 | 1.11 | 80 | 5.44 | 1.107 | 50 | 5.423 | 1.099 | s | 5.384 | 1.1034 | 9 | 5.4055 |
| 511 | 1.04 | 70 | 5.40 | 1.043 | 33 | 5.420 | 1.038 | s | 5.394 | 1.0403 | 5 | 5.4055 |
| 440 | | | | 0.968 | 17 | [b]5.476 | 0.953 | ms | 5.391 | 0.9557 | 3 | 5.4063 |
| 531 | | | | .916 | 33 | 5.419 | .911 | s | 5.390 | .9138 | 5 | 5.4061 |
| 620 | | | | .857 | 17 | 5.420 | .854 | s | 5.401 | .8548 | 3 | 5.4062 |
| 533 | | | | | | | .824 | s | 5.403 | .8244 | 2 | 5.4060 |
| 622 | | | | | | | .811 | ms | 5.380 | | | |
| Average value of last five lines | | | 5.41 | | | 5.420 | | | 5.393 | | | 5.4060 |

[a] This line appears on ASTM card but is not found in original text.

[b] This figure not used to obtain average.

## References

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[4] J. D. Hanawalt, H. W. Rinn, and L. K. Frevel, Chemical analysis by X-ray diffraction, Ind. and Eng. Chem., Anal. Ed. **10**, 457-512 (1938).

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[11] R. Moltzau and J. M. Kolthoff, Mixed crystal formation of ZnS post-precipitated with HgS: The aging of HgS and of ZnS, J. Phys. Chem. **40**, 637 (1936).


## Lead sulfide (galena), PbS, (cubic)

### ASTM cards

| Card number | | New index lines | Radiation | Source |
|---|---|---|---|---|
| Old | New | | | |
| ------- | 2110 | 3.00 | Copper---- | Lehmann [1] 1924. |
| | 3-0607 | 2.18 | | |
| | 3-0614 | 1.81 | | |
| 2162 | 2171 | 2.97 | Molybdenum | Hanawalt, Rinn, and Frevel [2] 1938. |
| | 1-0875 | 3.43 | | |
| | 1-0880 | 2.09 | | |
| II-1452 | 2232 | 2.96 | Copper---- | Harcourt [3] 1942. |
| | 2-0695 | 2.08 | | |
| | 2-0699 | 1.79 | | |
| ------- | 2345 | 2.93 | Copper---- | British Museum. |
| | 3-0674 | 2.07 | | |
| | 3-0665 | 1.78 | | |
| II-3992 | 3910 | 1.32 | Molybdenum | United Steel Companies, England. |
| | 2-1432 | 1.00 | | |
| | 2-1431 | 2.96 | | |

Both the old and new ASTM cards of Harcourt's pattern erroneously state that molybdenum radiation was used.

**Additional published patterns.** None.

**NBS pattern.** The sample of lead sulfide used for the NBS pattern was contributed by the National Lead Co. of New York. Spectrographic analysis by the Bureau showed less than 0.01 percent of copper, iron, silver, aluminum, magnesium, silicon, tin, and calcium.

**Interplanar spacings and intensity measurements.** The interplanar spacings of all patterns were changed from kX to angstrom units except those of Lehmann, which were calculated in angstroms from the Bragg angle data given in his paper. The 220 spacing of Lehmann's pattern listed at 2.18 A on the ASTM card should be 2.13 A and the 720 line, not permitted by the face-centered cubic lattice of galena, is included because it appeared in Lehmann's published pattern.

The three strongest lines for each of the patterns are as follows:

| Pattern | 1 | 2 | 3 |
|---|---|---|---|
| Lehmann---------- | 200 | 220 | 311 |
| Hanawalt, Rinn, and Frevel--- | 200 | 111 | 220 |
| Harcourt (both patterns)------ | 200 | 220 | 311 |
| British Museum---------- | 200 | 220 | 311 |
| United Steel---------- | 420 | 531 | 200 |
| Swanson and Fuyat---------- | 200 | 111 | 220 |

The intensities of the United Steel pattern are poor due to the effects of focusing and X-ray absorption.

**Lattice constants.** The structure was determined by Davey [4] in 1921. The lattice is face-centered cubic, $O_h^5$-Fm3m, sodium-chloride type with 4(PbS) per unit cell. The unit-cell measurements of Zeipel [5] and Wasserstein [6], which supposedly were expressed in kX units, have been converted to angstroms, but Lehmann's value was not, as the wavelength of the radiation used was unknown. The Wasserstein constant was accurately determined, using an exceptionally pure sample. Although Lehmann's unit-cell

| hkl | 1924 Lehmann Cu, 1.5405 A | | | 1938 Hanawalt, Rinn, and Frevel Mo, 0.7093 A | | | 1942 Harcourt Mo, 0.7093 A | | | ---- British Museum Cu, 1.5405 A | | | ---- United Steel Mo, 0.7093 A | | | 1953 Swanson and Fuyat Cu, 1.5405 A | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | d | I | a | d | I | a | d | I | a | d | I | a | d | I | a | d | I | a |
| | A | | A | A | | A | A | | A | A | | A | A | | A | A | | A |
| 111 | 3.49 | w | 6.045 | 3.44 | 80 | 5.958 | 3.43 | 50 | 5.941 | 3.39 | 60 | 5.872 | 3.43 | 70 | 5.941 | 3.429 | 84 | 5.939 |
| 200 | 3.00 | vvs | 6.000 | 2.98 | 100 | 5.960 | 2.97 | 100 | 5.940 | 2.94 | 100 | 5.880 | 2.966 | 90 | 5.932 | 2.969 | 100 | 5.938 |
| 220 | 2.13 | vs | 6.166 | 2.09 | 60 | 5.911 | 2.08 | 83 | 5.883 | 2.07 | 80 | 5.855 | 2.097 | 90 | 5.931 | 2.099 | 57 | 5.937 |
| 311 | 1.81 | vs | 6.003 | 1.79 | 32 | 5.937 | 1.789 | 83 | 5.933 | 1.78 | 80 | 5.904 | 1.789 | 90 | 5.933 | 1.790 | 35 | 5.937 |
| 222 | 1.73 | vw | 5.993 | 1.71 | 16 | 5.924 | 1.713 | 50 | 5.934 | 1.71 | 60 | 5.924 | 1.712 | 70 | 5.931 | 1.714 | 16 | 5.937 |
| 400 | 1.49 | ms | 5.960 | 1.487 | 8 | 5.948 | 1.483 | 50 | 5.932 | 1.48 | 60 | 5.920 | 1.483 | 70 | 5.932 | 1.484 | 10 | 5.936 |
| 331 | 1.37 | w | 5.972 | 1.364 | 8 | 5.946 | 1.363 | 33 | 5.941 | 1.37 | 60 | 5.972 | 1.361 | 70 | 5.932 | 1.362 | 10 | 5.937 |
| 420 | 1.33 | vs | 5.948 | 1.330 | 16 | 5.948 | 1.326 | 83 | 5.930 | 1.33 | 80 | 5.948 | 1.327 | 100 | 5.935 | 1.327 | 17 | 5.935 |
| 422 | 1.21 | vs | 5.928 | 1.214 | 8 | 5.947 | 1.222 | 67 | 5.987 | 1.21 | 70 | 5.928 | 1.2107 | 70 | 5.9312 | 1.212 | 10 | 5.938 |
| 511 | 1.14 | vs | 5.924 | 1.146 | 8 | 5.955 | 1.142 | 50 | 5.934 | 1.14 | 70 | 5.924 | 1.1415 | 70 | 5.9314 | 1.1424 | 6 | 5.9361 |
| 440 | 1.06 | w | 5.996 | 1.052 | 4 | 5.951 | 1.051 | 17 | 5.945 | 1.05 | 50 | 5.940 | 1.0485 | 60 | 5.9312 | 1.0489 | 3 | 5.9335 |
| 531 | 1.01 | s | 5.975 | 1.006 | 4 | 5.952 | 1.006 | 33 | 5.952 | 1.01 | 60 | 5.975 | 1.0026 | 100 | 5.9315 | 1.0034 | 5 | 5.9362 |
| 600 | 0.990 | vs | 5.946 | ----- | --- | ----- | 0.991 | 50 | 5.946 | ---- | --- | ----- | 0.9886 | 110? | 5.9316 | 0.9893 | 6 | 5.9358 |
| 620 | .940 | vs | 5.951 | ----- | --- | ----- | .940 | 50 | 5.945 | ---- | --- | ----- | .9379 | 110? | 5.9318 | .9386 | 4 | 5.9362 |
| 533 | ----- | --- | ----- | ----- | --- | ----- | .907 | 8 | 5.948 | ---- | --- | ----- | .9045 | 70 | 5.9312 | .9050 | 2 | 5.9345 |
| 622 | 0.896 | s | 5.943 | ----- | --- | ----- | .897 | 50 | 5.950 | ---- | --- | ----- | ------ | ---- | ------ | .8952 | 4 | 5.9381 |
| 444 | ----- | --- | ----- | ----- | --- | ----- | .858 | 8 | 5.944 | ---- | --- | ----- | ------ | ---- | ------ | .8568 | 1 | 5.9361 |
| 711 | 0.831 | m | 5.935 | ----- | --- | ----- | .833 | 50 | 5.949 | ---- | --- | ----- | ------ | ---- | ------ | .8312 | 3 | 5.9360 |
| 640 | ----- | --- | ----- | ----- | --- | ----- | .825 | 50 | 5.949 | ---- | --- | ----- | ------ | ---- | ------ | .8232 | 3 | 5.9362 |
| 720 | 0.820 | m | ----- | ----- | --- | ----- | ----- | --- | ----- | ---- | --- | ----- | ------ | ---- | ------ | ------ | --- | ------ |
| 642 | .792 | m | 5.927 | ----- | --- | ----- | ----- | --- | ----- | ---- | --- | ----- | ------ | ---- | ------ | ------ | --- | ------ |
| Average value of last five lines | | | 5.940 | ----- | --- | 5.951 | ----- | --- | 5.948 | ---- | --- | 5.943 | ------ | ---- | 5.9315 | ------ | --- | 5.9362 |

determination is high, the cell, based on an average of the last five lines of his pattern shown in the table, is in good agreement with the most precise determinations.

*Lattice constant in angstroms*

| | | |
|---|---|---|
| 1924 | Lehmann [1] | 5.966 |
| 1935 | Zeipel [5] | 5.935 at 18°C |
| 1951 | Wasserstein [6] | 5.936 |
| 1953 | Swanson and Fuyat | 5.9362 at 26°C |

The density of lead sulfide calculated from the NBS lattice constant is 7.596 at 26°C.

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## 2.3. Oxides

**Alpha-aluminum oxide (corundum), $\alpha$-$Al_2O_3$ (hexagonal)**

**ASTM cards**

| Card number | | New index lines | Radiation | Source |
|---|---|---|---|---|
| Old | New | | | |
| II-2831 | 3512 | 2.08 | Mo, 0.710 | Harrington [1] 1927. |
| | 2-1236 | 1.60 | Molybdenum | Hansen and Brown-Miller [2] 1928. |
| | 2-1227 | 2.55 | Iron | Passerini [3] 1930. |
| II-3590 | 3798 | 1.61 | No data | Kerr [4] 1932. |
| | 2-1371 | 2.08 | Iron | Kovalev [5] 1937. |
| | 2-1373 | 1.37 | | |
| 3314 | 3515 | 2.08 | Molybdenum | Hanawalt, Rinn, and Frevel [6] 1938. |
| | 1-1245 | 1.59 | | |
| | 1-1243 | 2.55 | | |
| 3725 | 3797 | 1.60 | Molybdenum | Aluminum Research Laboratory. |
| | 1-1296 | 2.08 | | |
| | 1-1296 | 2.55 | | |
| ------- | 3514 | 2.06 | Copper | British Museum. |
| | 3-1033 | 1.59 | | |
| | 3-1033 | 3.43 | | |

The old and new ASTM cards of the Kerr and Kovalev composite patterns erroneously state that molybdenum radiation was used when no data is given for the Kerr pattern, and the Kovalev pattern was made with iron radiation.

**Additional published patterns**

| Source | Radiation | Wavelength |
|---|---|---|
| Zachariasen [7] 1928 | Copper | 1.539 |

**NBS pattern.** The alpha-aluminum oxide used for the NBS pattern was a specially purified sample prepared by the Mallinckrodt Chemical Works. Spectrographic analysis at the Bureau showed 0.01 to 0.1 percent each of potassium, sodium, and silicon, 0.001 to 0.01 percent each of calcium, copper, iron, magnesium, and lead, and less than 0.001 percent each of boron, chromium, lithium, manganese, and nickel.

**Interplanar spacings and intensity measurements.** The *d*-spacings expressed in angstroms for the Passerini, Harrington, and Zachariasen patterns were calculated from their Bragg angle data. *D*-spacings for all of the remaining patterns were converted from kX to angstrom units. The Kerr and Boldyrev patterns were combined on one ASTM card, and those of Hansen and Brownmiller, Harrington, and Passerini were combined on another. A number of the patterns contain lines, not found in the NBS pattern, that are theoretically impossible for corundum.

The three strongest lines for each pattern are as follows:

| Pattern | 1 | 2 | 3 |
|---|---|---|---|
| Kerr | 116 | 113 | 300 |
| Boldyrev | 116 | 113 | 300 |
| Hansen and Brownmiller | 014 | 113 | 116 |
| Harrington | 116 | 113 | 300 |
| Passerini | 116 | 300 | 0·1·10 |
| Hanawalt, Rinn, and Frevel | 113 | 116 | 014 |
| Aluminum Research Laboratory | 116 | 113 | 014 |
| British Museum | 113 | 116 | 102 |
| Zachariasen | 116 | 113 | 300 |
| Swanson and Fuyat | 113 | 014 | 116 |

**Lattice constants.** The structure was determined by Bragg [8] in 1922. The space group is $D_{3d}^6$-R3c, corundum structure, with $6(Al_2O_3)$ per unit cell. Six other polymorphic forms for which no crystal structures have been established are known.

A group of unit-cell measurements have been converted from kX to angstrom units for comparison with the NBS values. Harrington's rhombohedral cell has been referred to hexagonal axes, and Passerini's cell has been doubled in the *c*-direction to conform with the accepted cell size.

*Lattice constants in angstroms*

| | | *a* | *c* |
|---|---|---|---|
| 1920 | Davey and Hoffman [9] | 4.87 | 13.26 |
| 1922 | Bragg [8] | 4.76 | 13.00 |
| 1927 | Harrington [1] | 4.770 | 13.020 |
| 1928 | Zachariasen [7] | 4.77 | 13.03 |
| 1930 | Passerini [3] | 4.750 | 12.982 |
| 1953 | Swanson and Fuyat | 4.758 | 12.991 at 26°C |

The density of alpha-aluminum oxide calculated from the NBS lattice constants is 3.987 at 26°C. The linear coefficient of thermal expansion is $6.58 \times 10^{-6}$ parallel to the *c*-axis and $5.43 \times 10^{-6}$ perpendicular to it, according to Sharma [10]. The alpha-alumina used for the NBS pattern was too finely divided for an accurate determination of the refractive indices.

## Alpha-aluminum oxide (corundum), $\alpha$-$Al_2O_3$

| *hkl* | 1932 Kerr Mo, 0.709 A | | 1937 Boldyrev Fe, 1.93597 A | | 1928 Hansen and Brownmiller Mo, 0.709 A | | 1927 Harrington Mo, 0.709 A | | 1930 Passerini Fe, 1.93597 A | |
|---|---|---|---|---|---|---|---|---|---|---|
| | *d* | *I* | *d* | *I* | *d* | *I* | *d* | *I* | *d* | *I* |
| | *A* | | *A* | | *A* | | *A* | | *A* | |
| ---------- | 3.96 | 30 | ---------- | ------- | ---------- | ------- | ---------- | ------- | ---------- | ------ |
| 102 | 3.53 | 60 | 3.442 | 30 | 3.49 | s | 3.49 | 50 | 3.388 | ms |
| 014 | 2.57 | 70 | 2.548 | 60 | 2.56 | vs | 2.56 | 70 | 2.498 | s |
| 110 | 2.39 | 50 | 2.379 | 40 | 2.38 | s | 2.39 | 50 | 2.335 | mw |
| ---------- | ---------- | ------- | 2.299 | 40 | ---------- | ------- | ---------- | ------- | ---------- | ------ |
| 006 | ---------- | ------- | ---------- | ------- | ---------- | ------- | ---------- | ------- | ---------- | ------ |
| 113 | 2.08 | 90 | 2.085 | 90 | 2.08 | vs | 2.09 | 80 | 2.055 | s |
| ---------- | ---------- | ------- | ---------- | ------- | ---------- | ------- | ---------- | ------- | 1.890 | mw |
| 204 | 1.754 | 70 | 1.742 | 50 | 1.744 | s | 1.744 | 70 | 1.718 | ms |
| ---------- | ---------- | ------- | ---------- | ------- | ---------- | ------- | ---------- | ------- | ---------- | ------ |
| 116 | 1.613 | 100 | 1.602 | 100 | 1.603 | vs | 1.601 | 100 | 1.587 | vs |
| 121 | 1.558 | 10 | ---------- | ------- | 1.543 | w | ---------- | ------- | 1.538 | mw |
| 108 | 1.523 | 40 | 1.516 | 50 | 1.513 | m | 1.507 | 20 | 1.500 | ms |
| 124 | 1.408 | 70 | 1.404 | 60 | 1.407 | s | 1.401 | 60 | 1.396 | s |
| 300 | 1.373 | 80 | 1.377 | 70 | 1.375 | s | 1.370 | 80 | 1.364 | vs |
| 028 | ---------- | ------- | ---------- | ------- | ---------- | ------- | ---------- | ------- | 1.265 | w |
| 0·1·10 | 1.237 | 60 | 1.241 | 40 | 1.242 | s | 1.231 | 80 | 1.230 | vs |
| 220 | 1.187 | 20 | 1.192 | 20 | 1.194 | m | 1.188 | 10 | 1.183 | ms |
| 306 | ---------- | ------- | ---------- | ------- | ---------- | ------- | ---------- | ------- | ---------- | ------ |
| 223 | 1.152 | 30 | 1.148 | 30 | 1.149 | m | 1.150 | 10 | 1.141 | ms |
| 311 | 1.127 | 20 | 1.127 | 20 | 1.126 | m | 1.119 | 10 | 1.120 | ms |
| 132 | | | | | | | | | | |
| 2·0·10 | 1.107 | 20 | ---------- | ------- | 1.101 | m | 1.098 | 20 | 1.096 | s |
| 0·0·12 | 1.082 | 20 | ---------- | ------- | 1.079 | m | 1.075 | 20 | 1.081 | s |
| 314 | ---------- | ------- | ---------- | ------- | ---------- | ------- | ---------- | ------- | ---------- | ------ |
| 226 | 1.047 | 40 | ---------- | ------- | 1.043 | s | 1.040 | 50 | 1.040 | vs |
| 402 | 1.022 | 10 | ---------- | ------- | ---------- | ------- | ---------- | ------- | ---------- | ------ |
| 1·2·10 | 1.000 | 30 | ---------- | ------- | 1.000 | m | 0.9918 | 40 | ---------- | ------ |
| 044 | ---------- | ------- | ---------- | ------- | ---------- | ------- | ---------- | ------- | ---------- | ------ |
| 138 | 0.939 | 10 | ---------- | ------- | 0.935 | w | 0.9298 | 10 | ---------- | ------ |
| 229 | ---------- | ------- | ---------- | ------- | ---------- | ------- | ---------- | ------- | ---------- | ------ |
| 234 | ---------- | ------- | ---------- | ------- | 0.910 | m | ---------- | ------- | ---------- | ------ |
| 1·0·14 | 0.903 | 40 | ---------- | ------- | .901 | w | 0.9024 | 30 | ---------- | ------ |
| 410 | ---------- | ------- | ---------- | ------- | ---------- | ------- | ---------- | ------- | ---------- | ------ |
| 413 | 0.882 | 10 | ---------- | ------- | 0.881 | w | ---------- | ------- | ---------- | ------ |
| 408 | ---------- | ------- | ---------- | ------- | ---------- | ------- | ---------- | ------- | ---------- | ------ |
| 3·1·10 | 0.860 | 20 | ---------- | ------- | 0.858 | m | 0.8519 | 20 | ---------- | ------ |
| 3·0·12 | ---------- | ------- | ---------- | ------- | .846 | m | .8407 | 10 | ---------- | ------ |
| 416 | 0.831 | 40 | ---------- | ------- | .832 | s | .8250 | 20 | ---------- | ------ |
| 1·1·15 | ---------- | ------- | ---------- | ------- | ---------- | ------- | .8038 | 10 | ---------- | ------ |
| 0·4·10 | 0.796 | 30 | ---------- | ------- | ---------- | ------- | .7934 | 20 | ---------- | ------ |

*(Continued)*

## Alpha-aluminum oxide (corundum), $\alpha$-$Al_2O_3$—Con.

| *hkl* | 1938 Hanawalt, Rinn, and Frevel Mo, 0.709 A | | ---- Aluminum Research Lab. Mo, 0.709 A | | ---- British Museum Cu, 1.5405 A | | 1928 Zachariasen Cu, 1.5405 A | | 1953 Swanson and Fuyat Cu, 1.5405 A, 26°C | |
|---|---|---|---|---|---|---|---|---|---|---|
| | *d* | *I* | *d* | *I* | *d* | *I* | *d* | *I* | *d* | *I* |
| | A | | A | | A | | A | | A | |
| 102 | 3.48 | 50 | 3.49 | 41 | 3.44 | 80 | 3.48 | 30 | 3.479 | 74 |
| 014 | 2.56 | 75 | 2.56 | 50 | 2.56 | 80 | 2.55 | 50 | 2.552 | 92 |
| 110 | 2.37 | 30 | 2.38 | 31 | 2.35 | 70 | 2.38 | 20 | 2.379 | 42 |
| 006 | ---- | ---- | 2.14 | 6 | ---- | ---- | ---- | ---- | 2.165 | < 1 |
| 113 | 2.08 | 100 | 2.08 | 84 | 2.06 | 100 | 2.09 | 70 | 2.085 | 100 |
| 204 | 1.74 | 50 | 1.74 | 41 | 1.73 | 80 | 1.74 | 30 | 1.740 | 43 |
| | ---- | ---- | 1.64 | 3 | ---- | ---- | ---- | ---- | ---- | ---- |
| 116 | 1.59 | 100 | 1.60 | 100 | 1.59 | 100 | 1.61 | 100 | 1.601 | 81 |
| 121 | 1.54 | 5 | 1.54 | 6 | 1.55 | 40 | ---- | ---- | 1.546 | 3 |
| 108 | 1.50 | 5 | 1.51 | 19 | 1.50 | 60 | 1.52 | 10-20 | 1.510 | 7 |
| 124 | 1.405 | 40 | 1.40 | 41 | 1.40 | 80 | 1.41 | 50 | 1.404 | 32 |
| 300 | 1.373 | 50 | 1.37 | 50 | 1.36 | 80 | 1.37 | 70 | 1.374 | 48 |
| 028 | ---- | ---- | ---- | ---- | ---- | ---- | 1.28 | 10 | 1.276 | 2 |
| 0·1·10 | 1.235 | 20 | 1.23 | 31 | 1.23 | 70 | 1.24 | 40 | 1.239 | 16 |
| 220 | 1.188 | 10 | 1.19 | 19 | 1.19 | 40 | 1.193 | 20 | 1.1898 | 6 |
| 306 | ---- | ---- | ---- | ---- | ---- | ---- | 1.158 | 10-20 | 1.1601 | < 1 |
| 223 | 1.146 | 5 | 1.15 | 9 | 1.14 | 40 | 1.148 | 10-20 | 1.1470 | 4 |
| 311 | 1.124 | 5 | 1.12 | 13 | 1.12 | 40 | 1.129 | 20 | 1.1382 | 1 |
| 132 | | | | | | | | | 1.1255 | 5 |
| 2·0·10 | 1.097 | 5 | 1.10 | 19 | 1.09 | 50 | 1.104 | 20-30 | 1.0988 | 6 |
| 0·0·12 | 1.082 | 5 | 1.08 | 19 | 1.07 | 60 | 1.082 | 20-30 | 1.0831 | 3 |
| 314 | ---- | ---- | ---- | ---- | ---- | ---- | ---- | ---- | 1.0781 | 7 |
| 226 | 1.042 | 8 | 1.04 | 25 | 1.04 | 70 | 1.045 | 40 | 1.0426 | 13 |
| 402 | 1.017 | 5 | 1.02 | 3 | 1.01 | 20 | 1.020 | 10-20 | 1.0175 | 1 |
| 1·2·10 | 0.997 | 5 | 0.99 | 19 | ---- | ---- | 1.000 | 30-40 | 0.9976 | 11 |
| 044 | ---- | ---- | ---- | ---- | ---- | ---- | 0.986 | 10-20 | .9819 | 2 |
| 138 | ---- | ---- | ---- | ---- | ---- | ---- | .936 | 20 | .9345 | 3 |
| 229 | ---- | ---- | ---- | ---- | ---- | ---- | ---- | ---- | .9178 | 2 |
| 234 | ---- | ---- | ---- | ---- | ---- | ---- | 0.9089 | 50 | .9076 | 12 |
| 1·0·14 | ---- | ---- | ---- | ---- | ---- | ---- | ---- | ---- | .9052 | 3 |
| 410 | ---- | ---- | ---- | ---- | ---- | ---- | 0.9007 | 30 | .8991 | 6 |
| 413 | ---- | ---- | ---- | ---- | ---- | ---- | ---- | ---- | .8804 | 4 |
| 408 | ---- | ---- | ---- | ---- | ---- | ---- | ---- | ---- | .8698 | 2 |
| 3·1·10 | ---- | ---- | ---- | ---- | ---- | ---- | ---- | ---- | .8580 | 12 |
| 3·0·12 | ---- | ---- | ---- | ---- | ---- | ---- | ---- | ---- | .8502 | 4 |
| 416 | ---- | ---- | ---- | ---- | ---- | ---- | ---- | ---- | .8303 | 22 |
| 1·1·15 | ---- | ---- | ---- | ---- | ---- | ---- | ---- | ---- | .8137 | 4 |
| 0·4·10 | ---- | ---- | ---- | ---- | ---- | ---- | ---- | ---- | .8075 | 11 |

## References


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## Cuprous oxide (cuprite), $Cu_2O$ (cubic)

### ASTM cards

| Card number | | New index lines | Radiation | Source |
|---|---|---|---|---|
| Old | New | | | |
| ------- | 3101 | 2.46 | Copper, | Niggli [1] 1922. |
| | 3-0892 | 1.51 | 1.541. | |
| | 3-0892 | 1.28 | | |
| II-2360 | 3099 | 2.46 | Iron------ | Neuburger [2] 1931. |
| | 2-1069 | 1.51 | Unknown--- | Waldo [3] 1935. |
| | 2-1067 | 2.13 | Copper---- | Harcourt [4] 1942. |

### ASTM cards—Con.

| Card number | | New index lines | Radiation | Source |
|---|---|---|---|---|
| Old | New | | | |
| 2928 | 3100 | 2.45 | Molybdenum | Hanawalt, Rinn, and Frevel [5] 1938. |
| | 1-1139 | 1.51 | | |
| | 1-1142 | 2.12 | | |
| ------- | 3145 | 2.44 | Copper---- | British Museum. |
| | 3-0908 | 1.50 | | |
| | 3-0898 | 2.12 | | |

The Neuburger, Waldo, and Harcourt patterns have been combined on one ASTM card, No. 2-1067.

**Additional published patterns.** None.

**NBS pattern.** The cuprous oxide sample used for the NBS pattern was prepared by Francis Heckman of the National Bureau of Standards by sintering cuprous chloride and sodium carbonate at approximately 800 °C. The sample was then leached with water and dried. Spectrographic analysis at the Bureau showed 0.1 to 1.0 percent each of calcium and silicon, 0.01 to 0.1 percent each of aluminum and magnesium, 0.001 to 0.01 percent each of silver, boron, barium, iron, and titanium, and less than 0.001 percent each of manganese, lead, and tin.

**Interplanar spacings and intensity measurements.** The Waldo, Hanawalt, Rinn, and Frevel, and the British Museum *d*-spacings were converted from kX to angstrom units, while the patterns of Niggli and Neuburger, expressed in angstroms were calculated directly from their Bragg angle data.

The three strongest lines for each of the patterns are as follows:

| Pattern | 1 | 2 | 3 |
|---|---|---|---|
| Niggli---------- | 111 | 220 | 311 |
| Neuburger---------- | 111 | 220 | 311 |
| Waldo---------- | 111 | 200 | 220 |
| Hanawalt, Rinn, and Frevel--- | 111 | 220 | 200 |
| British Museum---------- | 111 | 220 | 200 |
| Harcourt---------- | 111 | 200 | 110 |
| Swanson and Fuyat---------- | 111 | 200 | 220 |

Niggli's pattern contains five lines not consistent with the structure.

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## Cuprous oxide, $Cu_2O$

| *hkl* | 1922 Niggli Cu, 1.5405 A | | | 1931 Neuburger Fe, 1.93597 A | | | 1935 Waldo ---------- | | | 1938 Hanawalt, Rinn, and Frevel Mo, 0.709 A | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | *d* | *I* | *a* | *d* | *I* | *a* | *d* | *I* | *a* | *d* | *I* | *a* |
| | *A* | | *A* | *A* | | *A* | *A* | | *A* | *A* | | *A* |
| 110 | 3.168 | vw | 4.480 | 3.008 | w | 4.254 | 3.04 | vw | 4.299 | 3.01 | 3 | 4.257 |
| 111 | 2.457 | vvs | 4.256 | 2.454 | s | 4.250 | 2.46 | s | 4.261 | 2.45 | 100 | 4.244 |
| 200 | 2.130 | s | 4.260 | 2.123 | vw | 4.246 | 2.13 | m | 4.260 | 2.12 | 31 | 4.240 |
| 211 | -------- | ------- | ------- | -------- | ------ | ------- | 1.744 | vvw | 4.272 | --------- | ------- | ------- |
| 220 | 1.506 | vs | 4.261 | 1.507 | s | 4.262 | 1.509 | m | 4.268 | 1.51 | 44 | 4.271 |
| ---------- | 1.422 | vvw | ------- | -------- | ------ | ------- | ------- | ------- | ------- | --------- | ------- | ------- |
| ---------- | 1.350 | s | ------- | -------- | ------ | ------- | ------- | ------- | ------- | --------- | ------- | ------- |
| 311 | 1.281 | s-vs | 4.249 | 1.284 | s | 4.259 | 1.285 | m | 4.261 | 1.286 | 31 | 4.265 |
| 222 | 1.229 | mw | 4.257 | 1.230 | vw | 4.261 | 1.232 | w | 4.268 | 1.231 | 5 | 4.264 |
| ---------- | 1.1624 | s | ------- | -------- | ------ | ------- | ------- | ------- | ------- | --------- | ------- | ------- |
| 400 | 1.0635 | w | 4.254 | 1.0654 | s | 4.262 | 1.067 | vw | 4.268 | 1.067 | 3 | 4.268 |
| ---------- | 1.0027 | s | ------- | -------- | ------ | ------- | ------- | ------- | ------- | --------- | ------- | ------- |
| 331 | 0.9773 | ms | 4.260 | -------- | ------ | ------- | 0.980 | w | 4.272 | 0.979 | 5 | 4.267 |
| 420 | .9533 | mw | 4.263 | -------- | ------ | ------- | .954 | w | 4.266 | .955 | 3 | 4.271 |
| 422 | .8703 | ms | 4.264 | -------- | ------ | ------- | .872 | w | 4.272 | .871 | 3 | 4.267 |
| ---------- | .8358 | s | ------- | -------- | ------ | ------- | ------- | ------- | ------- | --------- | ------- | ------- |
| 511 | .8219 | ms | 4.271 | -------- | ------ | ------- | 0.824 | w | 4.282 | 0.821 | 3 | 4.266 |
| Average value of last five lines---------- | | | 4.262 | -------- | ------ | 4.258 | ------- | ------- | 4.272 | --------- | ------- | 4.269 |

| *hkl* | ---- British Museum Cu, 1.5405 A | | | 1942 Harcourt Cu, 1.5405 A | | | 1953 Swanson and Fuyat Cu, 1.5405 A, 26°C | | |
|---|---|---|---|---|---|---|---|---|---|
| | *d* | *I* | *a* | *d* | *I* | *a* | *d* | *I* | *a* |
| | *A* | | *A* | *A* | | *A* | *A* | | *A* |
| 110 | 3.01 | 40 | 4.257 | 3.00 | 10 | 4.24 | 3.020 | 9 | 4.271 |
| 111 | 2.44 | 100 | 4.226 | 2.46 | 100 | 4.26 | 2.465 | 100 | 4.270 |
| 200 | 2.12 | 60 | 4.240 | 2.13 | 20 | 4.26 | 2.135 | 37 | 4.270 |
| 211 | 1.74 | 20 | 4.262 | ---------- | --------- | ---------- | 1.743 | 1 | 4.269 |
| 220 | 1.50 | 80 | 4.243 | 1.51 | 10 | 4.27 | 1.510 | 27 | 4.271 |
| ---------- | ---------- | --------- | ---------- | ---------- | --------- | ---------- | ---------- | --------- | ---------- |
| ---------- | ---------- | --------- | ---------- | ---------- | --------- | ---------- | ---------- | --------- | ---------- |
| 311 | 1.29 | 60 | 4.278 | 1.288 | 6 | 4.27 | 1.287 | 17 | 4.268 |
| 222 | 1.23 | 40 | 4.261 | ---------- | --------- | ---------- | 1.233 | 4 | 4.271 |
| ---------- | ---------- | --------- | ---------- | ---------- | --------- | ---------- | ---------- | --------- | ---------- |
| 400 | 1.06 | 20 | 4.240 | ---------- | --------- | ---------- | 1.0674 | 2 | 4.2696 |
| ---------- | ---------- | --------- | ---------- | ---------- | --------- | ---------- | ---------- | --------- | ---------- |
| 331 | ---------- | --------- | ---------- | ---------- | --------- | ---------- | 0.9795 | 4 | 4.2695 |
| 420 | ---------- | --------- | ---------- | ---------- | --------- | ---------- | .9548 | 3 | 4.2700 |
| 422 | ---------- | --------- | ---------- | ---------- | --------- | ---------- | .8715 | 3 | 4.2695 |
| ---------- | ---------- | --------- | ---------- | ---------- | --------- | ---------- | ---------- | --------- | ---------- |
| 511 | ---------- | --------- | ---------- | ---------- | --------- | ---------- | 0.8216 | 3 | 4.2692 |
| Average value of last five lines---------- | | | 4.257 | ---------- | --------- | 4.26 | ---------- | --------- | 4.2696 |

**Lattice constants.** The structure was determined by Bragg and Bragg [6] in 1915. The space group of the simple cubic lattice is $O_h^4$-Pn3m, cuprous-oxide type with $2(Cu_2O)$ per unit cell.

A group of unit-cell determinations supposedly published in kX units have been converted to angstroms for comparison with the NBS value.

*Lattice constant in angstroms*

| | | |
|---|---|---|
| 1931 | Neuburger [2] | 4.261 |
| 1932 | Wrigge and Meisel [7] | 4.263 |
| 1953 | Swanson and Fuyat | 4.2696 at 26°C |

The density of cuprous oxide calculated from the NBS lattice constant is 6.100 at 26°C. The refractive index is too high to be determined by ordinary liquid grain immersion methods.

**References**


[1] P. Niggli, Die Kristallstruktur einiger Oxyde I, Z. Krist. **57**, 253-99 (1922).

[2] M. C. Neuburger, Präzisionsmessung der Gitterkonstants von Cuprooxyd $Cu_2O$, Z. Physik. **67**, 845-50 (1931); Über die Gitterkonstante von Cuprooxyd $Cu_2O$, Z. Krist. **77**, 169-70 (1931).

[3] A. W. Waldo, Identification of the copper ore minerals by means of X-ray powder diffraction patterns, Am. Min. **20**, 590 (1935).

[4] G. A. Harcourt, Tables for the identification of ore minerals by X-ray powder patterns, Am. Min. **27**, 63-113 (1942).

[5] J. D. Hanawalt, H. W. Rinn, and L. K. Frevel, Chemical analysis by X-ray diffraction, Ind. Eng. Chem., Anal. Ed. **10**, 457-512 (1938).

[6] W. H. Bragg and W. L. Bragg, X-rays and crystal structure, Fifth ed., London (1925).

[7] F. W. Wrigge and K. Meisel, Die Dichte von Kupferoxydul, Z. anorg. allg. Chem. **203**, 312-20 (1931-32).


## Zinc Oxide (zincite), ZnO (hexagonal)

**ASTM cards**

| Card number | | New index lines | Radiation | Source |
|---|---|---|---|---|
| Old | New | | | |
| ---- | 2624 | 2.78 | Iron | Aminoff [1] 1922. |
| | 3-0760 | 2.44 | | |
| | 3-0752 | 1.89 | | |

**ASTM cards—Con.**

| Card number | | New index lines | Radiation | Source |
|---|---|---|---|---|
| Old | New | | | |
| ---- | 3095 | 2.46 | Copper | Weber [2] 1922. |
| | 3-0891 | 1.63 | | |
| | 3-0891 | 1.38 | | |
| 2911 | 3079 | 2.46 | Molybdenum | Hanawalt, Rinn, and Frevel [3] 1938. |
| | 1-1134 | 2.81 | | |
| | 1-1136 | 2.61 | | |
| ---- | 3107 | 2.48 | Cobalt, 1,7902 | United Steel Companies, England. |
| | 3-0895 | 0.91 | | |
| | 3-0888 | 2.81 | | |

**Additional published patterns.** None.

**NBS pattern.** The zinc oxide sample used for the NBS pattern was prepared by the New Jersey Zinc Co. Spectrographic analysis at the NBS showed less than 0.001 percent each of magnesium, silicon, and calcium.

**Interplanar spacings and intensity measurements.** The *d*-spacings of the Hanawalt, Rinn, and Frevel, Aminoff, and Weber patterns were converted from kX to angstrom units. The United Steel spacings were apparently given in angstroms. The Aminoff pattern contains a spacing of 1.23 A inconsistent with the theoretical pattern.

The three strongest lines for each of the patterns are as follows:

| Pattern | 1 | 2 | 3 |
|---|---|---|---|
| Aminoff | 100 | 101 | 102 |
| Weber | 101 | 110 | 112 |
| Hanawalt, Rinn, and Frevel | 101 | 100 | 002 |
| United Steel | 101 | 213 | 100 |
| Swanson and Fuyat | 101 | 100 | 002 |

**Lattice constants.** The structure was determined by W. L. Bragg [4] in 1920. The hexagonal lattice has space group $C_{6v}^4$-C6mc, zinc oxide, or wurtzite structure type, and 2(ZnO) per unit cell.

All of the unit-cell determinations except the Heller, McGannon, and Weber values, determined in angstroms, have been converted from kX to angstrom units for comparison with the NBS cell.

| hkl | 1922 Aminoff Fe, 1.93597 A | | 1922 Weber Cu, 1.5405 A | | 1938 Hanawalt, Rinn, and Frevel Mo, 0.7093 A | | ---- United Steel Co, 1.7902 A | | 1953 Swanson and Fuyat Cu, 1.5405 A, 26°C | |
|---|---|---|---|---|---|---|---|---|---|---|
| | d | I | d | I | d | I | d | I | d | I |
| | A | | A | | A | | A | | A | |
| 100 | 2.79 | 100 | 2.81 | 90 | 2.82 | 50 | 2.82 | 80 | 2.816 | 71 |
| 002 | 2.59 | 80 | 2.61 | 60 | 2.62 | 30 | 2.61 | 70 | 2.602 | 56 |
| 101 | 2.44 | 100 | 2.46 | 100 | 2.46 | 100 | 2.49 | 100 | 2.476 | 100 |
| 102 | 1.89 | 100 | 1.90 | 80 | 1.91 | 16 | 1.91 | 70 | 1.911 | 29 |
| 110 | 1.60 | 100 | 1.63 | 100 | 1.61 | 30 | 1.62 | 80 | 1.626 | 40 |
| 103 | 1.47 | 100 | 1.48 | 90 | 1.477 | 30 | 1.48 | 80 | 1.477 | 35 |
| 200 | 1.40 | 40 | 1.42 | 40 | 1.405 | 2 | 1.41 | 50 | 1.407 | 6 |
| 112 | 1.37 | 100 | 1.38 | 100 | 1.381 | 20 | 1.38 | 80 | 1.379 | 28 |
| 201 | ---------- | ------- | 1.36 | 70 | 1.358 | 8 | 1.36 | 70 | 1.359 | 14 |
| 004 | 1.29 | 60 | 1.31 | 30 | 1.305 | 2 | 1.30 | 40 | 1.301 | 3 |
| 202 | 1.24 | 40 | 1.24 | 40 | 1.238 | 2 | 1.24 | 50 | 1.225 | 5 |
| ---------- | 1.23 | 60 | ---------- | ------- | ---------- | ------- | ---------- | ------- | ---------- | ------- |
| 104 | ---------- | ------- | 1.18 | 20 | 1.181 | 2 | 1.18 | 40 | 1.1812 | 3 |
| 203 | 1.09 | 100 | 1.10 | 80 | 1.095 | 4 | 1.09 | 70 | 1.0929 | 10 |
| 210 | ---------- | ------- | 1.07 | 50 | 1.065 | 2 | 1.06 | 55 | 1.0639 | 4 |
| 211 | ---------- | ------- | 1.04 | 70 | 1.044 | 4 | 1.04 | 70 | 1.0422 | 10 |
| 114 | ---------- | ------- | 1.02 | 50 | 1.018 | 2 | 1.02 | 60 | 1.0158 | 5 |
| 212 | ---------- | ------- | 0.990 | 50 | ---------- | ------- | 0.987 | 60 | 0.9848 | 4 |
| 105 | ---------- | ------- | .980 | 80 | 0.979 | 2 | .979 | 70 | .9764 | 7 |
| 204 | ---------- | ------- | .960 | 20 | .957 | 2 | .957 | 40 | .9555 | 1 |
| 300 | ---------- | ------- | .943 | 60 | .939 | 2 | .940 | 70 | .9382 | 4 |
| 213 | ---------- | ------- | .911 | 90 | .908 | 2 | .909 | 100 | .9069 | 12 |
| 302 | ---------- | ------- | .887 | 70 | ---------- | ------- | ---------- | ------- | .8826 | 6 |
| 006 | ---------- | ------- | .873 | 20 | ---------- | ------- | ---------- | ------- | .8675 | 1 |
| 205 | ---------- | ------- | .842 | 70 | ---------- | ------- | ---------- | ------- | .8369 | 6 |
| 106 | ---------- | ------- | ---------- | ------- | ---------- | ------- | ---------- | ------- | .8290 | 2 |
| 214 | ---------- | ------- | ---------- | ------- | ---------- | ------- | ---------- | ------- | .8237 | 2 |
| 220 | ---------- | ------- | 0.817 | 70 | ---------- | ------- | ---------- | ------- | .8125 | 5 |

*Lattice constants in angstroms*

| | | a | c |
|---|---|---|---|
| 1929 | Natta and Passerini [5] | 3.25 | 5.19 |
| 1929 | Fuller [6]---------- | 3.242 | 5.220 |
| 1935 | Bunn [7]---------- | 3.2492 | 5.2053 at 18°C |
| 1950 | Heller, McGannon, and Weber [8]---------- | 3.2495 | 5.2069 |
| 1953 | Swanson and Fuyat------ | 3.249 | 5.205 at 26°C |

The density of zinc oxide calculated from the NBS lattice constants is 5.680 at 26°C. The sample was too finely divided for determination of the refractive indices.

## References


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[3] J. D. Hanawalt, H. W. Rinn, and L. K. Frevel, Chemical analysis by X-ray diffraction, Ind. Eng. Chem., Anal. Ed. **10**, 457-512 (1938).

[4] W. L. Bragg, The crystalline structure of zinc oxide, Phil. Mag. **39**, 647-51 (1920).

[5] G. Natta and L. Passerini, Soluzioni solidi, isomorfismo e simmorfismo tra gli ossidi dei metalli bivalenti; I, Sistemi: CaO-CdO, CaO-MnO, CaO-CoO, CaO-NiO, CaO-MnO, Gazz. chim. ital. **59**, 129-43 (1929).

[6] M. L. Fuller, A method of determining the axial ratio of a crystal from X-ray diffraction data; the axial ratio and lattice constants of zinc oxide, Science **70**, 196-8 (1929).

[7] C. W. Bunn, The lattice-dimensions of zinc oxide, Proc. Phys. Soc. (London) **47**, 835-42 (1935).

[8] R. B. Heller, J. McGannon, and A. H. Weber, Precision determination of the lattice constants of zinc oxide, J. App. Phys. **21**, 1283-5 (1950).

## Cadmium oxide, CdO (cubic)

### ASTM cards

| Card number | | New index lines | Radiation | Source |
|---|---|---|---|---|
| Old | New | | | |
| 2598 | 2711 | 2.70 | Molybdenum-- | Hanawalt, Rinn, and Frevel [1] 1938. |
| | 1-1044 | 2.34 | | |
| | 1-1049 | 1.65 | | |
| 11-2489 | 3230 | 2.34 | Molybdenum, 0.707831. | Ksanda [2] 1931. |
| | 2-1111 | 1.66 | | |
| | 2-1102 | 2.71 | | |

### Additional published patterns

| Source | Radiation | Wavelength |
|---|---|---|
| Fuller [3] 1929---------- | ---------- | ---------- |
| Natta and Passerini [4] 1929 | Copper----- | ---------- |

**NBS pattern.** The sample of cadmium oxide used for the NBS pattern was prepared by Johnson, Matthey and Co., Ltd., London. Their spectrographic analysis showed very faint traces of calcium, magnesium, copper, silver, sodium, and silicon.

**Interplanar spacings and intensity measurements.** Fuller's pattern, made in the course of a unit-cell determination, does not include intensity values. Ksanda's pattern was made to be used as an internal standard. The high accuracy of his *d*-spacings is the result of averaging values obtained from three samples of exceptionally high purity prepared three different ways, and of special refinements in procedure. *D*-spacings expressed in angstroms for the Ksanda and the Natta and Passerini patterns were calculated from their Bragg angle data. The Fuller and the Hanawalt, Rinn, and Frevel *d*-spacings were converted from kX to angstrom units.

The three strongest lines for each of the patterns are as follows:

| Pattern | 1 | 2 | 3 |
|---|---|---|---|
| Natta and Passerini---------- | 220 | 311 | 331 |
| Ksanda---------- | 200 | 220 | 111 |
| Hanawalt, Rinn, and Frevel---- | 111 | 200 | 220 |
| Swanson and Fuyat---------- | 111 | 200 | 220 |

**Lattice constants.** The structure was determined by Davey [5] in 1920. The lattice is face-centered cubic with space group $O_h^5$-Fm3m, sodium-chloride-type structure and 4 (CdO) per unit cell.

### Cadmium oxide, CdO

| *hkl* | 1929 Fuller ---------- | | | 1929 Natta and Passerini Cu, 1.5405 A | | | 1931 Ksanda Mo, 0.7093 A | | | 1938 Hanawalt, Rinn, and Frevel Mo, 0.7093 A | | | 1953 Swanson and Fuyat Cu, 1.5405 A, 27°C | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | *d* | *I* | *a* | *d* | *I* | *a* | *d* | *I* | *a* | *d* | *I* | *a* | *d* | *I* | *a* |
| | *A* | | *A* | *A* | | *A* | *A* | | *A* | *A* | | *A* | *A* | | *A* |
| 111 | ------- | ---- | ------ | 2.655 | ms | 4.60 | 2.7126 | 80 | 4.6984 | 2.71 | 100 | 4.69 | 2.712 | 100 | 4.697 |
| 200 | ------- | ---- | ------ | 2.315 | ms | 4.63 | 2.3492 | 100 | 4.6984 | 2.34 | 100 | 4.68 | 2.349 | 88 | 4.698 |
| 220 | 1.660 | ---- | 4.695 | 1.657 | s | 4.69 | 1.6611 | 90 | 4.6983 | 1.65 | 100 | 4.67 | 1.661 | 43 | 4.698 |
| 311 | ------- | ---- | ------ | 1.418 | s | 4.70 | 1.4166 | 70 | 4.6983 | 1.415 | 75 | 4.676 | 1.416 | 28 | 4.696 |
| 222 | 1.354 | ---- | 4.690 | 1.358 | ms | 4.71 | 1.3562 | 60 | 4.6980 | 1.355 | 30 | 4.677 | 1.355 | 13 | 4.694 |
| 400 | 1.174 | ---- | 4.696 | 1.174 | mw | 4.70 | 1.1746 | 40 | 4.6984 | 1.173 | 15 | 4.680 | 1.1742 | 5 | 4.6968 |
| 331 | 1.075 | ---- | 4.686 | 1.082 | s | 4.72 | 1.0779 | 30 | 4.6985 | 1.077 | 30 | 4.708 | 1.0772 | 9 | 4.6954 |
| 420 | 1.048 | ---- | 4.687 | 1.058 | s | 4.73 | 1.0506 | 50 | 4.6984 | 1.049 | 40 | 4.676 | 1.0499 | 13 | 4.6953 |
| 422 | ------- | ---- | ------ | 0.966 | s | 4.73 | 0.9590 | 20 | 4.6981 | 0.959 | 25 | 4.703 | 0.9584 | 11 | 4.6952 |
| 511 | 0.903 | ---- | 4.692 | .910 | ms | 4.72 | .9041 | 10 | 4.6978 | .904 | 18 | 4.677 | .9036 | 9 | 4.6952 |
| 440 | .830 | ---- | 4.695 | .837 | m | 4.74 | -------- | --- | ------ | .832 | 2 | 4.695 | .8300 | 5 | 4.6952 |
| 531 | ------- | ---- | ------ | ------- | ---- | ------ | -------- | --- | ------ | .795 | 15 | 4.674 | -------- | --- | ------ |
| 600 | ------- | ---- | ------ | ------- | ---- | ------ | -------- | --- | ------ | .784 | 10 | 4.680 | -------- | --- | ------ |
| 620 | ------- | ---- | ------ | ------- | ---- | ------ | -------- | --- | ------ | ------- | --- | -------- | -------- | --- | ------ |
| Average value of the last five lines--- | | | [a]4.692 | ------- | ----- | 4.73 | -------- | --- | 4.6982 | ------- | --- | 4.6857 | -------- | --- | 4.6953 |

[a] All values used.

Several unit-cell measurements supposedly published in kX units have been converted to angstroms for comparison with the NBS value.

*Lattice constant in angstroms*

| | | |
|---|---|---|
| 1929 | Fuller [3] | 4.690 |
| 1929 | Natta and Passerini [4] | 4.735 |
| 1931 | Ksanda [2] | 4.6991 |
| 1940 | Felipe [6] | 4.6943 |
| 1953 | Swanson and Fuyat | 4.6953 at 27°C |

The density of cadmium oxide calculated from the NBS lattice constant is 8.238 at 27°C. The refractive index is too high to be determined by the usual liquid grain immersion methods.

**References**


[1] J. D. Hanawalt, H. W. Rinn, and L. K. Frevel, Chemical analysis by X-ray diffraction, Ind. and Eng. Chem., Anal. Ed. **10**, 457-512 (1938).

[2] C. J. Ksanda, Comparison standards for the powder spectrum method; NiO and CdO, Amer. Jour. Sci. **22**, 131-138 (1931).

[3] M. L. Fuller, Precision measurements of X-ray reflections from crystal powders, Phil. Mag. **8**, 585-586 (1929).

[4] G. Natta and L. Passerini, Soluzioni solidi, isomorfismo e simmorfismo tra gli ossidi dei metalli bivalenti; 1. sistemi: CaO-CdO, CaO-MnO, CaO-CoO, CaO-NiO, CaO-MgO: Gazz. chim. ital. **59**, 129-43 (1929).

[5] W. P. Davey and E. O. Hoffman, Crystal analysis of metallic oxides, Phys. Rev. **15**, 333 (1920).

[6] J. C. Felipe, Medida de precisión de las constantes reticulares de los cristales, utilizando el método de análisis de Debye-Scherrer, Rev. Acad. Cienc. Exact; Fís. y Nat., Madrid, **34**, 180-95 (1940).


## Thallium oxide, $Tl_2O_3$ (cubic)

**ASTM cards**

| Card number | | New index lines | Radiation | Source |
|---|---|---|---|---|
| Old | New | | | |
| II-3631 | 3812 | 1.56 | Copper | British Museum. |
| | 2-1379 | 2.94 | | |
| | 2-1379 | 1.82 | | |
| 2071 | 2131 | 3.02 | Molybdenum | Hanawalt, Rinn, and Frevel [1] 1938. |
| | 1-0865 | 1.86 | | |
| | 1-0849 | 1.58 | | |

**Additional published patterns.** None.

**NBS pattern.** The sample of thallium oxide used for the NBS pattern was prepared by Johnson, Matthey, and Co., Ltd. Their spectrographic analysis showed the following impurities: 0.001 to 0.01 percent each of calcium, magnesium, sodium, and lithium, and less than 0.001 percent each of lead, aluminum, silicon, and copper. Before the sample was used it was annealed at about 370°C in a sealed tube for 2 hours to prevent volatilization of noxious fumes.

**Interplanar spacings and intensity measurements.** The British Museum and Hanawalt, Rinn, and Frevel $d$-spacings were converted from kX to angstrom units. The $d$-spacings of the British Museum pattern are generally lower than the others, but the differences are so irregular that no single factor would bring them into agreement with the NBS values.

The three strongest lines for each of the patterns are as follows:

| Pattern | 1 | 2 | 3 |
|---|---|---|---|
| British Museum | 622 | 222 | 440 |
| Hanawalt, Rinn, and Frevel | 222 | 440 | 622 |
| Swanson and Fuyat | 222 | 400 | 440 |

**Lattice constants.** The structure was determined by Zachariasen [2] in 1926. The body-centered cubic lattice has space group $T^5$-$I2_13$, $Mn_2O_3$ structure type, with 16 ($Tl_2O_3$) per unit cell.

The Zachariasen unit-cell determination has been converted from kX to angstrom units for comparison with the NBS values.

*Lattice constant in angstroms*

| | | |
|---|---|---|
| 1926 | Zachariasen [2] | 10.59 |
| 1953 | Swanson and Fuyat | 10.543 at 26°C |

The density of thallium oxide calculated from the NBS lattice constant is 10.916 at 26°C.

(See table, p. 29).

**References**


[1] J. D. Hanawalt, H. W. Rinn, and L. K. Frevel, Chemical analysis by X-ray diffraction, Ind. and Eng. Chem., Anal. Ed. **10**, 457-512 (1938).

[2] W. H. Zachariasen, The crystal structure of the modification C of the sesquioxides of the rare earth metals and of indium and thallium, Norsk. geol. Tidsskr. **9**, 310-316 (1926).

## Thallium oxide, $Tl_2O_3$

| *hkl* | ---- British Museum Cu, 1.5405 A | | | 1938 Hanawalt, Rinn, and Frevel Mo, 0.709 A | | | 1953 Swanson and Fuyat Cu, 1.5405 A, 26°C | | |
|---|---|---|---|---|---|---|---|---|---|
| | *d* | *I* | *a* | *d* | *I* | *a* | *d* | *I* | *a* |
| | *A* | | *A* | *A* | | *A* | *A* | | *A* |
| 211 | ---------- | --------- | ---------- | ---------- | --------- | ---------- | 4.304 | 11 | 10.540 |
| 222 | 2.95 | 80 | 10.22 | 3.03 | 100 | 10.50 | 3.042 | 100 | 10.537 |
| 321 | ---------- | --------- | ---------- | ---------- | --------- | ---------- | 2.816 | 3 | 10.537 |
| 400 | 2.56 | 60 | 10.24 | 2.63 | 50 | 10.52 | 2.635 | 42 | 10.540 |
| 411 | 2.43 | 20 | 10.31 | ---------- | --------- | ---------- | 2.484 | 6 | 10.539 |
| 420 | ---------- | --------- | ---------- | ---------- | --------- | ---------- | 2.357 | 2 | 10.541 |
| 332 | 2.21 | 20 | 10.36 | ---------- | --------- | ---------- | 2.248 | 4 | 10.544 |
| 422 | ---------- | --------- | ---------- | ---------- | --------- | ---------- | 2.149 | 1 | 10.530 |
| 510 | 2.02 | 60 | 10.30 | 2.06 | 10 | 10.50 | 2.068 | 8 | 10.545 |
| 521 | ---------- | --------- | ---------- | ---------- | --------- | ---------- | 1.924 | 3 | 10.538 |
| 440 | 1.82 | 80 | 10.30 | 1.86 | 60 | 10.52 | 1.863 | 33 | 10.539 |
| 530 | ---------- | --------- | ---------- | ---------- | --------- | ---------- | 1.808 | 2 | 10.542 |
| 600 | 1.73 | 40 | 10.38 | ---------- | --------- | ---------- | 1.758 | 1 | 10.548 |
| 611 | 1.68 | 40 | 10.36 | ---------- | --------- | ---------- | 1.710 | 5 | 10.541 |
| 620 | ---------- | --------- | ---------- | ---------- | --------- | ---------- | 1.668 | 2 | 10.549 |
| 541 | 1.60 | 20 | 10.37 | ---------- | --------- | ---------- | 1.628 | 4 | 10.550 |
| 622 | 1.565 | 100 | 10.38 | 1.58 | 60 | 10.48 | 1.589 | 27 | 10.540 |
| 631 | 1.533 | 40 | 10.40 | 1.54 | 10 | 10.44 | 1.554 | 6 | 10.540 |
| 444 | 1.503 | 40 | 10.41 | 1.51 | 10 | 10.46 | 1.522 | 6 | 10.545 |
| 710 | 1.479 | 20 | 10.46 | ---------- | --------- | ---------- | 1.491 | 3 | 10.543 |
| 640 | 1.442 | 20 | 10.40 | ---------- | --------- | ---------- | 1.462 | 1 | 10.543 |
| 721 | 1.417 | 40 | 10.41 | ---------- | --------- | ---------- | 1.434 | 3 | 10.538 |
| 642 | 1.387 | 20 | 10.38 | ---------- | --------- | ---------- | 1.409 | 2 | 10.544 |
| 732 | 1.325 | 40 | 10.43 | ---------- | --------- | ---------- | 1.339 | 3 | 10.543 |
| 800 | 1.305 | 40 | 10.44 | ---------- | --------- | ---------- | 1.318 | 3 | 10.544 |
| 811 | 1.281 | 40 | 10.41 | ---------- | --------- | ---------- | 1.298 | 4 | 10.545 |
| 820 | 1.262 | 20 | 10.41 | ---------- | --------- | ---------- | 1.279 | 2 | 10.547 |
| 653 | 1.246 | 40 | 10.42 | ---------- | --------- | ---------- | 1.2597 | 2 | 10.539 |
| 822 | 1.218 | 40 | 10.34 | ---------- | --------- | ---------- | 1.2428 | 1 | 10.546 |
| 831 | 1.199 | 70 | 10.31 | ---------- | --------- | ---------- | 1.2261 | 3 | 10.547 |
| 662 | ---------- | --------- | ---------- | 1.202 | 20 | 10.48 | 1.2094 | 6 | 10.543 |
| 840 | 1.168 | 70 | 10.45 | 1.172 | 10 | 10.48 | 1.1789 | 4 | 10.544 |
| 910 | 1.142 | 20 | 10.34 | ---------- | --------- | ---------- | 1.1646 | 1 | 10.546 |
| 921 | 1.131 | 20 | 10.49 | ---------- | --------- | ---------- | 1.1371 | 2 | 10.545 |
| 930 | 1.112 | 40 | 10.55 | ---------- | --------- | ---------- | 1.1110 | 1 | 10.540 |
| 932 | 1.080 | 40 | 10.47 | ---------- | --------- | ---------- | 1.0874 | 1 | 10.543 |
| 844 | 1.070 | 60 | 10.48 | ---------- | --------- | ---------- | 1.0764 | 2 | 10.546 |
| 941 | 1.058 | 40 | 10.47 | ---------- | --------- | ---------- | 1.0649 | 1 | 10.542 |
| 10·0·0 | 1.050 | 20 | 10.50 | ---------- | --------- | ---------- | ---------- | --------- | ---------- |
| 10·2·0 | 1.027 | 60 | 10.47 | ---------- | --------- | ---------- | ---------- | --------- | ---------- |
| Average value of last five lines-- | | | 10.48 | ---------- | --------- | 10.47 | ---------- | --------- | 10.543 |

## Lead oxide (litharge), PbO (red) (tetragonal)

### ASTM cards

| Card number | | New index lines | Radiation | Source |
|---|---|---|---|---|
| Old | New | | | |
| ---- | 1937 | 3.10 | Copper------ | McMurdie and Bunting [1] 1939. |
| | 3-0557 | 1.86 | | |
| | 3-0561 | 1.67 | | |
| ---- | 2031 | 3.08 | Copper------ | Anderson and Hochgesang, Lehigh University, 1939. |
| | 3-0590 | 1.67 | | |
| | 3-0573 | 1.86 | | |
| 1921 | 1891 | 3.11 | Molybdenum-- | New Jersey Zinc Co. |
| | 1-0788 | 2.80 | | |
| | 1-0796 | 1.86 | | |
| 1922 | 1892 | 3.11 | Molybdenum-- | Hanawalt, Rinn, and Frevel. |
| | 1-0789 | 2.80 | | |
| | 1-0797 | 1.68 | | |
| The following card labeled lead monoxide, litharge, is not litharge, but the yellow orthorhombic lead monoxide, massicot. That card will be discussed with those of the yellow form of lead oxide. | | | | |
| 1999 | 1981 | 3.06 | Molybdenum-- | Hanawalt, Rinn, and Frevel [1] 1938. |
| | 1-0819 | 2.93 | | |
| | 1-0824 | 2.72 | | |

**Additional published patterns.** None.

**NBS pattern.** The sample of red lead monoxide used for the NBS pattern was contributed by the National Lead Company of New York. Spectrographic analysis at the NBS showed less than 0.01 percent each of bismuth, copper, iron, and silicon and less than 0.001 percent each of calcium and magnesium.

**Interplanar spacings and intensity measurements.** *D*-spacings for the four ASTM card patterns were converted from kX to angstrom units. Only the McMurdie and Bunting pattern is published.

The three strongest lines for each of the patterns are as follows:

| Pattern | 1 | 2 | 3 |
|---|---|---|---|
| McMurdie and Bunting---------- | 101 | 112 | 211 |
| Anderson and Hochgesang------- | 101 | 211 | 112 |
| New Jersey Zinc Co.----------- | 101 | 110 | 112 |
| Hanawalt, Rinn, and Frevel---- | 101 | 110 | 211 |
| Swanson and Fuyat------------ | 101 | 110 | 112 |

**Lattice constants.** According to Byström [2], the space group of the red lead monoxide is $D^7_{4h}$-P4/nmm with 2(PbO) per unit cell. Two unit-cell determinations have been converted from kX to angstrom units for comparison with the NBS values.

*Lattice constants in angstroms*

| | | *a* | *b* |
|---|---|---|---|
| 1941 | Moore and Pauling [3]---- | 3.955 | 4.998 |
| 1945 | Byström [2]------------- | 3.9720 | 5.018 |
| 1953 | Swanson and Fuyat-------- | 3.9759 | 5.023 at 27°C |

The density of the red lead oxide calculated from the NBS lattice constants is 9.355 at 27°C. The refractive index was too high to be determined by the usual liquid grain immersion methods.

(See table, p. 31)

### References

[1] H. F. McMurdie and E. N. Bunting, X-ray studies of compounds in the system PbO-$SiO_2$, J. Res. Nat. Bur. Standards **23**, 543 (1939).

[2] A. Byström, The decomposition products of lead peroxide and oxidation products of lead oxide, Arkiv. Kemi. Miner. Geol. **A20**, no. 11, pp. 31 (1945).

[3] W. J. Moore, Jr. and L. Pauling, The crystal structures of the tetragonal monoxides of lead, tin, palladium, and platinum, J. Am. Chem. Soc. **63**, 1392 (1941).

## Lead oxide, PbO (red)

| *hkl* | 1939 McMurdie and Bunting Cu, 1.54050 A | | 1939 Anderson and Hochgesang Cu, 1.54050 A | | ---- New Jersey Zinc Mo, 0.709 A | | ---- Hanawalt, Rinn, and Frevel Mo, 0.709 A | | 1953 Swanson and Fuyat Cu, 1.54050 A, 27°C | |
|---|---|---|---|---|---|---|---|---|---|---|
| | *d* | *I* | *d* | *I* | *d* | *I* | *d* | *I* | *d* | *I* |
| | A | | A | | A | | A | | A | |
| 001 | 5.08 | 20 | | | | | | | 5.018 | 5 |
| | | | 3.17 | 10 | | | | | | |
| 101 | 3.11 | 100 | 3.09 | 100 | 3.12 | 100 | 3.12 | 100 | 3.115 | 100 |
| 110 | 2.80 | 60 | 2.79 | 50 | 2.81 | 43 | 2.81 | 40 | 2.809 | 62 |
| 002 | 2.50 | 60 | 2.50 | 30 | 2.52 | 3 | 2.52 | 7 | 2.510 | 18 |
| 102 | 2.11 | 20 | 2.11 | 3 | | | | | 2.124 | 1 |
| | 2.06 | 20 | | | | | | | | |
| 200 | 1.97 | 60 | 1.97 | 40 | 1.980 | 9 | 1.99 | 20 | 1.988 | 8 |
| 112 | 1.86 | 80 | 1.86 | 70 | 1.867 | 30 | 1.87 | 30 | 1.872 | 37 |
| 211 | 1.67 | 80 | 1.67 | 90 | 1.671 | 30 | 1.68 | 40 | 1.675 | 24 |
| 202 | | | 1.55 | 30 | 1.548 | 15 | 1.55 | 13 | 1.558 | 6 |
| 103 | 1.53 | 80 | 1.54 | 40 | | | | | 1.542 | 11 |
| 113 | 1.43 | 20 | | | | | 1.446 | 1 | 1.438 | 2 |
| 220 | 1.40 | 40 | 1.40 | 30 | 1.403 | 9 | 1.408 | 5 | 1.405 | 5 |
| | 1.34 | 20 | | | | | | | | |
| 301<br>203 | 1.28 | 20 | 1.27 | 30 | | | 1.283 | 5 | 1.282 | 2 |
| 310 | 1.25 | 40 | 1.25 | 30 | 1.253 | 3 | 1.258 | 5 | 1.256 | 3 |
| 222 | 1.22 | 80 | 1.22 | 30 | 1.219 | 11 | 1.222 | 9 | 1.226 | 4 |
| 311<br>213 | | | 1.21 | 60 | | | | | 1.219 | 5 |
| 104 | 1.19 | 40 | 1.19 | 3 | | | | | 1.1977 | <1 |
| 114 | 1.15 | 60 | 1.14 | 30 | 1.145 | 3 | 1.148 | 1 | 1.1462 | 2 |
| 312 | 1.12 | 40 | 1.12 | 50 | 1.120 | 3 | 1.126 | 5 | 1.1232 | 2 |
| 321<br>223 | 1.08 | 40 | 1.07 | 60 | 1.074 | 6 | 1.080 | 5 | 1.0768 | 3 |
| 204 | 1.06 | 40 | 1.06 | 20 | | | | | 1.0610 | 2 |
| 303 | 1.04 | 20 | 1.04 | 20 | | | 1.042 | 1 | 1.0386 | <1 |
| 214 | 1.02 | 20 | 1.02 | 5 | | | | | 1.0254 | <1 |
| 005 | | | 0.994 | 3 | | | 0.997 | 1 | | |
| 401<br>105 | 0.972 | 60 | .974 | 10 | | | .975 | 1 | 0.9738 | 1 |
| 411<br>115 | .948 | 40 | .946 | 10 | | | .950 | 1 | .9462 | 1 |
| 330<br>224 | .937 | 60 | .936 | 10 | | | | | .9365 | 3 |
| 331<br>323 | | | .921 | 20 | | | 0.923 | 1 | .9200 | 3 |
| | | | .889 | 40 | | | | | | |
| | | | .879 | 30 | | | | | | |
| | | | .875 | 40 | | | | | | |
| | | | .837 | 50 | | | | | | |
| | | | .817 | 10 | | | | | | |
| | | | .802 | 20 | | | | | | |
| | | | .802 | 10 | | | | | | |
| | | | .785 | 40 | | | | | | |

**Lead oxide (massicot), PbO (yellow) (orthorhombic)**

**ASTM cards**

| Card number | | New index lines | Radiation | Source |
|---|---|---|---|---|
| Old | New | | | |
| 1999 | 1981 | 3.06 | Molybdenum-- | Hanawalt, Rinn, and Frevel [1] 1938. |
| | 1-0819 | 2.93 | | |
| | 1-0824 | 2.72 | | |
| ---- | 2093 | 3.01 | Copper------ | McMurdie and Bunting [2] 1939. |
| | 3-0603 | 2.72 | | |
| | 3-0610 | 2.35 | | |
| ---- | 2142 | 3.03 | Copper------ | Anderson and Hochgesang, Lehigh University, 1939. |
| | 3-0616 | 1.71 | | |
| | 3-0599 | 1.63 | | |
| ---- | 2143 | ----- | ---------- | A continuation of the preceding card. |
| | 3-0617 | | | |
| | 3-0600 | | | |

The 1-0824 card (Hanawalt, Rinn, and Frevel) is labeled litharge and although it contains tetragonal crystallographic data completely unrelated to the X-ray pattern appearing on the card, it is that of the orthorhombic yellow lead oxide, massicot.

**Additional published patterns.** None.

**NBS pattern.** The sample of yellow lead monoxide used for the NBS pattern was contributed by the National Lead Company of New York. Spectrographic analysis at the NBS showed less than 0.01 percent of both bismuth and iron and less than 0.001 percent each of aluminum, silver, copper, magnesium, silicon, and calcium.

**Interplanar spacings and intensity measurements.** The yellow lead oxide pattern is complicated by a reaction of the oxide on exposure to air which results in a gray-green surface coating giving a number of foreign lines. Clear yellow lead oxide does not give these lines; they have been omitted from the pattern. The following six lines are due to the green phase formed after a flat sample of yellow PbO was exposed to air for several days:

| $d$-spacing | Relative intensity | $d$-spacing | Relative intensity |
|---|---|---|---|
| 4.27 | 3 | 2.623 | 6 |
| 4.010 | 2 | 2.440 | <1 |
| 3.357 | 7 | 1.701 | <1 |

The remaining lines have given consistent results with samples from several different sources, and they agree closely with the other patterns.

$D$-spacings for the three ASTM card patterns were converted from kX to angstrom units. Anderson and Hochgesang listed a $d$-spacing at 3.61 A with an intensity of 5, which falls close to the 110 line of the theoretical pattern which is not permitted by the space group.

The three strongest lines for each of the patterns are as follows:

| Pattern | 1 | 2 | 3 |
|---|---|---|---|
| Hanawalt, Rinn, and Frevel------ | 111 | 002 | 200 |
| McMurdie and Bunting---------- | 111 | 200 | 020 |
| Anderson and Hochgesang--------- | 111 | 113 | 311 |
| Swanson and Fuyat--------------- | 111 | 002 | 200 |

**Lattice constants.** The structure of the yellow lead oxide has not been established definitely. Byström [4] found the space group to be $C_{2v}^5$-Pca, whereas the NBS pattern seems to fit $C_{2v}^8$-Pba better. As the difference between these two space groups is only a difference in choice of axes and as no one has succeeded in growing crystals large enough for single-crystal work, either of these and perhaps other space groups will fit the powder data. There are 4($\beta$-PbO) per unit cell.

Several recent unit-cell determinations presumably in kX units have been converted to angstroms for comparison with the NBS values.

*Lattice constants in angstroms*

| | | $a$ | $b$ | $c$ |
|---|---|---|---|---|
| 1932 | Darbyshire [3]--- | 5.470 | 4.733 | 5.871 |
| 1941 | Petersen [5]----- | 5.485 | 4.755 | 5.899 |
| | | 5.508 | 4.754 | 5.895 |
| 1943 | Byström [4]------ | 5.49 | 4.75 | 5.89 |
| 1945 | Byström [6]------ | 5.487 | 4.753 | 5.888 |
| 1953 | Swanson and Fuyat | 5.489 | 4.755 | 5.891 at 27°C |

The density of the yellow lead oxide calculated from the NBS lattice constants is 9.642 at 27°C. The refractive indices are too high to be measured by the usual liquid grain immersion methods.

## Lead oxide, PbO (yellow)

| hkl | 1938 Hanawalt, Rinn, and Frevel Mo, 0.709 A | | 1939 McMurdie and Bunting Cu, 1.54050 A | | 1939 Anderson and Hochgesang Cu, 1.5405 A | | 1953 Swanson and Fuyat Cu, 1.5405 A, 27°C | |
|---|---|---|---|---|---|---|---|---|
| | d | I | d | I | d | I | d | I |
| | A | | A | | A | | A | |
| 001 | ------- | --- | 5.90 | 20 | ------ | --- | 5.893 | 6 |
| --- | ------- | --- | ------ | --- | 3.61 | 5 | ------- | --- |
| 111 | 3.07 | 100 | 3.02 | 100 | 3.04 | 100 | 3.067 | 100 |
| 002 | 2.94 | 25 | 2.94 | 60 | 2.92 | 70 | 2.946 | 31 |
| 200 | 2.73 | 25 | 2.73 | 80 | 2.73 | 60 | 2.744 | 28 |
| 201 | ------- | --- | ------ | --- | 2.47 | 5 | 2.493 | <1 |
| 020 | 2.36 | 20 | 2.35 | 80 | 2.36 | 50 | 2.377 | 20 |
| 112 | ------- | --- | 2.26 | 20 | 2.27 | 3 | 2.278 | <1 |
| 211 | ------- | --- | 2.19 | 20 | ------ | --- | 2.203 | <1 |
| 202 | 1.99 | 20 | 1.98 | 80 | 1.99 | 70 | 2.008 | 12 |
| 003 | ------- | --- | 1.95 | 40 | 1.95 | 5 | 1.963 | 2 |
| 212, 022 | 1.84 | 20 | 1.83 | 80 | 1.84 | 20 | 1.850 | 14 |
| 220 | 1.79 | 15 | 1.78 | 80 | 1.79 | 50 | 1.797 | 14 |
| 113 | 1.71 | 25 | 1.71 | 80 | 1.71 | 90 | 1.724 | 15 |
| 311 | 1.63 | 23 | 1.60 | 80 | 1.63 | 80 | 1.640 | 13 |
| 203 | ------- | --- | 1.58 | 40 | 1.59 | 3 | 1.596 | <1 |
| 222 | 1.52 | 18 | 1.52 | 80 | 1.53 | 60 | 1.534 | 9 |
| 213 | ------- | --- | 1.51 | 40 | 1.51 | 5 | 1.514 | 2 |
| 131, 004 | 1.470 | 20 | 1.46 | 80 | 1.47 | 80 | 1.474 | 11 |
| 321 | ------- | --- | 1.40 | 40 | 1.40 | 30 | 1.408 | <1 |
| 400 | ------- | --- | 1.37 | 60 | 1.38 | 30 | 1.372 | 1 |
| 114 | ------- | --- | 1.35 | 60 | 1.36 | 30 | 1.363 | <1 |
| 223 | ------- | --- | 1.32 | 40 | 1.33 | 5 | 1.325 | 1 |
| 204 | 1.290 | 8 | 1.29 | 60 | ------ | --- | 1.297 | 2 |
| 313 | ------- | --- | 1.28 | 60 | 1.28 | --- | 1.289 | 3 |
| 024 | ------- | --- | ------ | --- | 1.25 | 40 | 1.252 | 2 |
| 402 | 1.248 | 5 | 1.24 | 60 | 1.24 | 40 | 1.244 | 2 |
| 133 | 1.202 | 5 | 1.20 | 60 | 1.20 | 50 | 1.203 | 4 |
| 040, 420 | ------ | --- | 1.18 | 60 | 1.19 | 30 | 1.188 | 3 |
| 331 | 1.172 | 5 | 1.17 | 60 | 1.17 | 50 | 1.174 | 4 |
| 224 | 1.137 | 3 | 1.13 | 60 | 1.14 | 40 | 1.139 | 2 |
| 115 | 1.117 | 3 | 1.11 | 60 | 1.12 | 70 | 1.120 | 2 |
| 042, 422 | 1.102 | 3 | ------ | --- | 1.10 | 50 | 1.102 | 4 |
| 240 | ------- | --- | 1.09 | 80 | 1.09 | 10 | 1.091 | 2 |
| --- | ------- | --- | 1.09 | 60 | ------ | --- | ------- | --- |
| --- | 1.052 | 3 | ------ | --- | [a] | --- | ------- | --- |
| --- | 1.022 | 5 | ------ | --- | ------ | --- | ------- | --- |
| --- | 0.925 | 3 | ------ | --- | ------ | --- | ------- | --- |

[a] Additional twenty-one lines were not indexed.

### References


[1] J. D. Hanawalt, H. W. Rinn, and L. K. Frevel, Chemical analysis by X-ray diffraction, Ind. and Eng. Chem., Anal. Ed. **10**, 457-512 (1938).

[2] H. F. McMurdie and E. N. Bunting, X-ray studies of compounds in the system PbO-$SiO_2$, J. Res., Nat. Bur. Standards **23**, 543-7 (1939).

[3] J. A. Darbyshire, An X-ray examination of the oxides of lead, J. Chem. Soc., *1932*, 211-19 (1932).

[4] A. Byström, Röntgenographic studies of rhombic PbO, Arkiv. Kemi. Miner. Geol. **17B** No. 8, 1-6 (1943).

[5] M. Petersen, Studies of the preparation and allotropic transformation of lead monoxide, J. Am. Chem. Soc. **63**, 2617-20 (1941).

[6] A. Byström, The decomposition products of lead peroxide and oxidation products of lead oxide, Arkiv. Kemi. Miner. Geol. **A20**, No. 11, 31 pp. (1945).


## Uranium dioxide (uraninite), $UO_2$ (cubic)

**ASTM cards.** None.

### Additional published patterns

| Source | Radiation | Wavelength |
|---|---|---|
| Goldschmidt and Thomassen [1] 1923 | Iron | 1.934 |
| van Arkel [2] 1924 | Copper | --- |
| Schoep and Billiet [3] 1935 | Copper | --- |

**NBS pattern.** The uranium dioxide sample used for the NBS pattern was furnished by the Atomic Energy Commission. Chemical analysis at the NBS showed 80.0 percent of uranium, which is equal to the compound $UO_{2.03}$, based on the uranium analysis and oxygen by difference. Subtracting the 0.02 percent impurity in the sample from the oxygen remainder would give an even more nearly ideal value. The oxygen ratio is important as a solid solution exists in the cubic range between $UO_{2.0}$ and about $UO_{2.2}$, the unit cell size decreasing as the oxygen content increases.

Spectrographic analysis at the NBS showed 0.001 to 0.006 percent each of aluminum, calcium, copper, iron, sodium, and silicon, and 0.0001 to 0.001 percent each of beryllium, chromium, magnesium, manganese, nickel, lead, and tin.

**Interplanar spacings and intensity measurements.** *D*-spacings expressed in angstroms for the three patterns from the literature were calculated from their Bragg angle data.

The three strongest lines for the NBS pattern are as follows:

| Pattern | 1 | 2 | 3 |
|---|---|---|---|
| Swanson and Fuyat---------- | 111 | 220 | 200 |

The intensity values for both the Goldschmidt and Thomassen and the Schoep and Billiet patterns are given in such general terms that they are not comparable to present day values and both show focusing and X-ray absorption. No intensity values were published with the van Arkel pattern.

**Lattice constants.** The structure was determined by Cuy [4] in 1927. The face-centered cubic lattice has space group $O_h^5$-Fm3m, fluorite-structure type and $4(UO_2)$ per unit cell.

A group of unit cell determinations supposedly expressed in kX units have been converted to angstroms for comparison with the NBS value.

*Lattice constant in angstroms*

| | | |
|---|---|---|
| 1923 | Goldschmidt and Thomassen [1] | 5.48 |
| 1935 | Schoep and Billiet [3]-------- | 5.48 |
| 1948 | Grunvold and Haraldsen [5]---- | 5.468 |
| 1948 | Rundle, Baenziger, Wilson, and McDonald [6]---------- | 5.4691 |
| 1948 | Zachariasen [7]---------- | 5.4678 |
| 1949 | Alberman and Anderson [8]----- | 5.468 |
| 1951 | Katz [9]---------- | 5.4696 |
| 1953 | Swanson and Fuyat---------- | 5.4682 at 26°C |

The density of uranium dioxide calculated from the NBS lattice constant is 10.968 at 26°C. The compound is generally opaque, prohibiting refractive index measurements.

**Uranium dioxide, $UO_2$**

| *hkl* | 1923 Goldschmidt and Thomassen Fe, 1.934 A | | | 1924 van Arkel Cu, 1.5405 A | | 1935 Schoep and Billiet Cu, 1.5405 A | | | 1953 Swanson and Fuyat Cu, 1.5405 A, 26°C | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | *d* | *I* | *a* | *d* | *a* | *d* | *I* | *a* | *d* | *I* | *a* |
| | *A* | | *A* | *A* | *A* | *A* | | *A* | *A* | | *A* |
| 111 | 3.153 | s | 5.461 | 3.168 | 5.487 | 3.168 | s | 5.487 | 3.157 | 100 | 5.468 |
| 200 | 2.742 | m | 5.484 | 2.739 | 5.478 | 2.739 | ms | 5.478 | 2.735 | 48 | 5.470 |
| 220 | 1.932 | s | 5.465 | 1.926 | 5.448 | 1.938 | vs | 5.481 | 1.934 | 49 | 5.470 |
| 311 | 1.646 | s | 5.459 | 1.653 | 5.482 | 1.657 | vs | 5.496 | 1.649 | 47 | 5.469 |
| 222 | 1.579 | m | 5.470 | 1.582 | 5.480 | 1.575 | ms | 5.456 | 1.579 | 13 | 5.469 |
| 400 | 1.369 | m | 5.476 | 1.372 | 5.488 | 1.364 | ms | 5.456 | 1.368 | 9 | 5.472 |
| 331 | 1.257 | s | 5.479 | 1.256 | 5.475 | 1.255 | s | 5.469 | 1.255 | 18 | 5.469 |
| 420 | 1.225 | s | 5.478 | 1.224 | 5.474 | 1.221 | s | 5.460 | 1.223 | 15 | 5.469 |
| 422 | 1.1177 | vs | 5.4756 | 1.1186 | 5.4800 | 1.1186 | s | 5.4800 | 1.1163 | 13 | 5.4687 |
| 511 | 1.0545 | vvs | 5.4793 | 1.0532 | 5.4726 | 1.0549 | s | 5.4814 | 1.0523 | 15 | 5.4679 |
| 440 | ---------- | --------- | -------- | 0.9704 | 5.4894 | 0.9692 | s | 5.4826 | 0.9666 | 6 | 5.4679 |
| 531 | ---------- | --------- | -------- | .9272 | 5.4854 | .9280 | vs | 5.4901 | .9243 | 15 | 5.4682 |
| 600 | ---------- | --------- | -------- | .9141 | 5.4846 | .9135 | s | 5.4810 | .9114 | 8 | 5.4684 |
| 620 | ---------- | --------- | -------- | .8666 | 5.4809 | .8660 | s | 5.4771 | .8646 | 9 | 5.4682 |
| 533 | ---------- | --------- | -------- | .8363 | 5.4840 | -------- | ------ | -------- | .8339 | 7 | 5.4682 |
| 622 | ---------- | --------- | -------- | .8258 | 5.4777 | -------- | ------ | -------- | .8243 | 7 | 5.4678 |
| 444 | ---------- | --------- | -------- | .7903 | 5.4754 | -------- | ------ | -------- | -------- | ------- | -------- |
| Average value of last five lines | | | [a]5.4775 | ---------- | 5.4805 | -------- | ------ | 5.4824 | -------- | ------- | 5.4682 |

[a] Average value of last two lines.

## References


[1] V. M. Goldschmidt and L. Thomassen, Die Krystallstruktur Natürlicher und synthetischer Oxyde von Uran, Thorium, und Cerium, Skrifter Norske Videnskaps-Akad. Oslo I. Mat.-Nat. Klasse 1923, No. 2, 48 p. (1923).

[2] A. E. van Arkel, Kristalbouw en physische eigenschappen, Physica **4**, 286-301 (1924).

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[5] F. Grunvold and H. Haraldsen, Oxidation of uranium dioxide ($UO_2$), Nature **162**, 69-70 (1948).

[6] R. E. Rundle, N. C. Baenziger, A. S. Wilson, and R. A. McDonald, The structures of the carbides, nitrides and oxides of uranium, J. Am. Chem. Soc. **70**, 99-105 (1948).

[7] W. H. Zachariasen, Value presented at a meeting of the American Society for X-ray and Electron Diffraction held at New Haven, Conn., April 1948.

[8] K. B. Alberman and J. S. Anderson, The oxides of uranium, J. Chem. Soc. (London) Part V, S303 (1949).

[9] J. J. Katz and E. Rabinowitch, The chemistry of uranium, Part I, National Nuclear Energy Series. McGraw-Hill, 1951 from R. E. Rundle and N. C. Baenziger, Report CC-1980, November 23, 1944, and N. C. Baenziger, Report CC-1781, August 10, 1944.


## Magnesium aluminate (spinel), $MgAl_2O_4$ (cubic)

### ASTM cards

| Card number | | New index lines | Radiation | Source |
|---|---|---|---|---|
| Old | New | | | |
| ------- | 3871 | 1.43 | ---------- | Posnjak [1] 1928. |
| | 3-1161 | 2.44 | | |
| | 3-1160 | 2.01 | | |
| 2957 | 3154 | 2.43 | Molybdenum | Posnjak [1] 1928. |
| | 1-1157 | 1.42 | | |
| | 1-1154 | 2.01 | | |
| II-2417 | 3155 | 2.42 | Molybdenum | Posnjak [1] 1928. |
| | 2-1089 | 1.42 | No data-- | Hansen and Brown-miller [2] 1928. |
| | 2-1084 | 2.00 | No data-- | Passerini [3] 1930. |
| ------- | 3134 | 2.44 | Copper---- | Taylor [4] 1930. |
| | 3-0906 | 2.02 | | |
| | 3-0897 | 1.56 | | |

### ASTM cards—Con.

| Card number | | New index lines | Radiation | Source |
|---|---|---|---|---|
| Old | New | | | |
| ------- | 3873 | 1.43 | Copper, 1.539. | Hauptmann and Novak [5] 1932. |
| | 3-1163 | 2.43 | | |
| | 3-1162 | 2.02 | | |
| ------- | 3148 | 2.43 | Copper---- | Kordes [6] 1935. |
| | 3-0909 | 1.43 | | |
| | 3-0901 | 2.02 | | |
| 2974 | 3157 | 2.41 | Molybdenum | Hanawalt, Rinn, and Frevel [7] 1938. |
| | 1-1159 | 1.41 | | |
| | 1-1157 | 2.00 | | |
| II-2431 | 3151 | 2.41 | Copper---- | British Museum. |
| | 2-1087 | 1.42 | | |
| | 2-1086 | 4.62 | | |

The first Posnjak pattern No. 3-1160 does not exist in the reference given for it on the ASTM card and attempts to trace the data have proved futile. The *d*-spacings from the ASTM card for that pattern have been converted from kX to angstrom units. No radiation data was given.

The British Museum card for spinel, No. 2-1086, states that molybdenum radiation was used. Consideration of the intensities obtained from that pattern and other British Museum patterns would seem to indicate that copper was more likely.

### Additional published patterns

| Source | Radiation | Wavelength |
|---|---|---|
| Clark, Howe, and Badger [8] 1934---------- | Molybdenum | ---------- |
| Andrews [9] 1951---------- | Cobalt---- | 1.7889 |

**NBS pattern.** The spinel sample used for the NBS pattern was prepared by S. M. Lang of the NBS by a solid-state reaction at 1,920°C for 1 hour of a stoichiometric mixture of the component oxides.

Spectrographic analysis at the Bureau showed less than 0.1 percent each of silicon and calcium, less than 0.01 percent each of beryllium, iron, and sodium, and less than 0.001 percent each of copper and titanium.

## Magnesium aluminate (spinel), $MgAl_2O_4$

| *hkl* | 1928 Posnjak Mo, 0.709 A | | | 1928 Posnjak Mo, 0.709 A | | | 1928 Hansen and Brownmiller Mo, 0.709 A | | | 1930 Passerini Mo, 0.709 A | | | 1930 Taylor Cu, 1.5405 A | | | 1932 Hauptmann and Novak Cu, 1.5405 A | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | *d* | *I* | *a* | *d* | *I* | *a* | *d* | *I* | *a* | *d* | *I* | *a* | *d* | *I* | *a* | *d* | *I* | *a* |
| | A | | A | A | | A | A | | A | A | | A | A | | A | A | | A |
| 111 | 4.61 | 60 | 7.985 | 4.67 | 50 | 8.089 | ----- | --- | ----- | ------ | -- | ----- | ------ | ---- | ----- | ----- | --- | ----- |
| ------ | ---- | --- | ----- | ----- | --- | ----- | ----- | --- | ----- | ------ | -- | ----- | ------ | ---- | ----- | ----- | --- | ----- |
| ------ | ---- | --- | ----- | ----- | --- | ----- | ----- | --- | ----- | ------ | -- | ----- | ------ | ---- | ----- | ----- | --- | ----- |
| ------ | ---- | --- | ----- | ----- | --- | ----- | ----- | --- | ----- | ------ | -- | ----- | ------ | ---- | ----- | ----- | --- | ----- |
| 220 | 2.85 | 60 | 8.061 | 2.87 | 60 | 8.118 | 2.85 | s | 8.061 | 2.816 | w | 7.965 | 2.85 | m | 8.061 | ----- | --- | ----- |
| ------ | ---- | --- | ----- | ----- | --- | ----- | ----- | --- | ----- | ------ | -- | ----- | [a]2.55 | vw | | ----- | --- | ----- |
| 311 | 2.43 | 100 | 8.050 | 2.44 | 90 | 8.093 | 2.43 | vs | 8.050 | 2.409 | ms | 7.990 | 2.43 | vs | 8.059 | 2.43 | 80 | 8.059 |
| 222 | 2.32 | 5 | 8.037 | 2.33 | 5 | 8.071 | ----- | --- | ----- | ------ | -- | ----- | ------ | ---- | ----- | ----- | --- | ----- |
| ------ | ---- | --- | ----- | ----- | --- | ----- | ----- | --- | ----- | ------ | -- | ----- | 2.24 | w | ----- | ----- | --- | ----- |
| 400 | 2.01 | 80 | 8.040 | 2.01 | 90 | 8.040 | 2.02 | vs | 8.080 | 2.000 | s | 8.000 | 2.02 | vs | 8.080 | 2.02 | 80 | 8.080 |
| ------ | ---- | --- | ----- | ----- | --- | ----- | ----- | --- | ----- | ------ | -- | ----- | ------ | ---- | ----- | ----- | --- | ----- |
| ------ | ---- | --- | ----- | ----- | --- | ----- | ----- | --- | ----- | ------ | -- | ----- | [a]1.76 | w | ----- | ----- | --- | ----- |
| ------ | ---- | --- | ----- | ----- | --- | ----- | ----- | --- | ----- | ------ | -- | ----- | [a]1.72 | m | 8.068 | ----- | --- | ----- |
| 422 | 1.64 | 20 | 8.034 | 1.65 | 40 | 8.083 | 1.647 | m | 8.069 | 1.635 | w | 8.010 | 1.65 | m | 8.083 | 1.65 | 50 | 8.083 |
| ------ | ---- | --- | ----- | ----- | --- | ----- | ----- | --- | ----- | ------ | -- | ----- | 1.58 | m | ----- | ----- | --- | ----- |
| 511 | 1.55 | 80 | 8.054 | 1.55 | 80 | 8.054 | 1.554 | vs | 8.075 | 1.543 | ms | 8.018 | 1.55 | vs | 8.054 | 1.55 | 80 | 8.054 |
| ------ | ---- | --- | ----- | ----- | --- | ----- | ----- | --- | ----- | ------ | -- | ----- | ------ | ---- | ----- | ----- | --- | ----- |
| 440 | 1.42 | 90 | 8.033 | 1.43 | 100 | 8.089 | 1.426 | vs | 8.067 | 1.420 | vs | 8.033 | 1.43 | vs | 8.089 | 1.43 | 100 | 8.089 |
| ------ | ---- | --- | ----- | ----- | --- | ----- | ----- | --- | ----- | [a]1.412 | -- | ----- | ------ | ---- | ----- | ----- | --- | ----- |
| 531 | 1.36 | 10 | 8.045 | 1.36 | 30 | 8.045 | 1.365 | w | 8.075 | 1.362 | mw | 8.058 | 1.36 | m | 8.046 | ----- | --- | ----- |
| ------ | ---- | --- | ----- | ----- | --- | ----- | ----- | --- | ----- | ------ | -- | ----- | 1.29 | w | ----- | ----- | --- | ----- |
| ------ | ---- | --- | ----- | ----- | --- | ----- | ----- | --- | ----- | ------ | -- | ----- | ------ | ---- | ----- | ----- | --- | ----- |
| 620 | 1.27 | 10 | 8.032 | 1.28 | 40 | 8.095 | 1.277 | w | 8.076 | 1.275 | w | 8.064 | 1.28 | w | 8.095 | 1.28 | 50 | 8.095 |
| ------ | ---- | --- | ----- | ----- | --- | ----- | ----- | --- | ----- | ------ | -- | ----- | ------ | ---- | ----- | ----- | --- | ----- |
| 533 | ---- | --- | ----- | 1.23 | 50 | 8.066 | 1.233 | m | 8.085 | 1.226 | w | 8.039 | 1.23 | s | 8.066 | ----- | --- | ----- |
| 622 | 1.22 | 30 | 8.093 | 1.22 | 50 | 8.093 | ----- | --- | ----- | 1.213 | w | 8.046 | 1.22 | vw | 8.093 | ----- | --- | ----- |
| 444 | 1.16 | 20 | 8.037 | 1.16 | 50 | 8.037 | 1.168 | m | 8.092 | 1.162 | m | 8.051 | 1.16 | s | 8.037 | 1.17 | 50 | 8.106 |
| 711 | 1.12 | 10 | 7.998 | 1.13 | 20 | 8.070 | 1.133 | w | 8.091 | ------ | -- | ----- | 1.13 | w | 8.070 | ----- | --- | ----- |
| ------ | ---- | --- | ----- | ----- | --- | ----- | ----- | --- | ----- | 1.128 | w | ----- | ------ | ---- | ----- | ----- | --- | ----- |
| 642 | 1.08 | 20 | 8.082 | 1.08 | 50 | 8.082 | 1.081 | m | 8.089 | 1.077 | m | 8.060 | 1.08 | s | 8.082 | 1.08 | 50 | 8.082 |
| 731 | 1.05 | 40 | 8.065 | 1.05 | 70 | 8.065 | 1.052 | s | 8.081 | 1.050 | s | 8.065 | 1.05 | vs | 8.065 | 1.05 | 80 | 8.065 |
| ------ | ---- | --- | ----- | ----- | --- | ----- | ----- | --- | ----- | ------ | -- | ----- | [a]1.03 | w | ----- | ----- | --- | ----- |
| 800 | 1.00 | 30 | 8.000 | 1.01 | 50 | 8.080 | 1.010 | w | 8.080 | 1.008 | s | 8.064 | 1.01 | s | 8.080 | ----- | --- | ----- |
| ------ | ---- | --- | ----- | ----- | --- | ----- | ----- | --- | ----- | ------ | -- | ----- | [a]1.00 | w | ----- | ----- | --- | ----- |
| 822 | 0.95 | 10 | 8.061 | 0.951 | 30 | 8.070 | 0.951 | w | 8.070 | ------ | -- | ----- | 0.952 | m | 8.078 | 0.954 | 50 | 8.095 |
| 751 | .93 | 30 | 8.054 | .933 | 50 | 8.080 | .932 | m | 8.071 | ------ | -- | ----- | .933 | s(s) | 8.080 | ----- | --- | ----- |
| ------ | ---- | --- | ----- | ----- | --- | ----- | ----- | --- | ----- | ------ | -- | ----- | ------ | ---- | ----- | ----- | --- | ----- |
| ------ | ---- | --- | ----- | ----- | --- | ----- | ----- | --- | ----- | ------ | -- | ----- | [a]0.914 | m | ----- | ----- | --- | ----- |
| 840 | 0.90 | 20 | 8.050 | 0.903 | 40 | 8.077 | 0.903 | m | 8.077 | ------ | -- | ----- | .902 | s | 8.068 | 0.905 | 50 | 8.095 |
| 911 | ---- | --- | ----- | ----- | --- | ----- | ----- | --- | ----- | ------ | -- | ----- | ------ | ---- | ----- | ----- | --- | ----- |
| 664 | ---- | --- | ----- | ----- | --- | ----- | ----- | --- | ----- | ------ | -- | ----- | ------ | ---- | ----- | ----- | --- | ----- |
| 931 | 0.84 | 20 | 8.013 | 0.847 | 40 | 8.080 | 0.846 | m | 8.070 | ------ | -- | ----- | ------ | ---- | ----- | ----- | --- | ----- |
| 844 | .82 | 40 | 8.034 | .825 | 60 | 8.083 | .824 | s | 8.074 | ------ | -- | ----- | ------ | ---- | ----- | ----- | --- | ----- |
| 933 | ---- | --- | ----- | ----- | --- | ----- | ----- | --- | ----- | ------ | -- | ----- | ------ | ---- | ----- | ----- | --- | ----- |
| 10·2·0 | 0.79 | 10 | 8.056 | 0.793 | 10 | 8.087 | ----- | --- | ----- | ------ | -- | ----- | ------ | ---- | ----- | ----- | --- | ----- |
| 951 | .78 | 20 | 8.068 | .783 | 40 | 8.099 | 0.782 | m | 8.089 | ------ | -- | ----- | ------ | ---- | ----- | ----- | --- | ----- |
| 880 | ---- | --- | ----- | .716 | 40 | 8.101 | .714 | w | 8.078 | ------ | -- | ----- | ------ | ---- | ----- | ----- | --- | ----- |
| 11·5·3 | ---- | --- | ----- | .650 | 40 | 8.092 | .648 | w | 8.068 | ------ | -- | ----- | ------ | ---- | ----- | ----- | --- | ----- |
| 12·4·0 | ---- | --- | ----- | .639 | 40 | 8.083 | .637 | w | 8.057 | ------ | -- | ----- | ------ | ---- | ----- | ----- | --- | ----- |
| 10·6·6 | ---- | --- | ----- | .618 | 40 | 8.105 | .616 | w | 8.079 | ------ | -- | ----- | ------ | ---- | ----- | ----- | --- | ----- |
| Average value of last five lines | | | 8.044 | ----- | --- | 8.096 | ----- | --- | 8.074 | ------ | -- | 8.056 | ------ | ---- | 8.074 | ----- | --- | 8.089 |

[a] This line is not permitted by the space group of spinel.

*(Continued)*

| hkl | 1935 Kordes [a] Cu, 1.5405 A | | | 1938 Hanawalt, Rinn, and Frevel Mo, 0.709 A | | | ---- British Museum Mo, 0.709 A | | | 1934 Clark, Howe, and Badger Mo, 0.709 A | | | 1951 Andrews Co, 1.7889 A | | | 1953 Swanson and Fuyat Cu, 1.5405 A, 26°C | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | d | I | a | d | I | a | d | I | a | d | I | a | d | I | a | d | I | a |
| | A | | A | A | | A | A | | A | A | | A | A | | A | A | | A |
| 111 | 4.68 | 50 | 8.106 | 4.64 | 13 | 8.037 | 4.63 | 80 | 8.019 | ----- | -- | ----- | 4.67 | w | 8.0887 | 4.67 | 4 | 8.089 |
| ------ | ------ | -- | ----- | ----- | -- | ----- | 4.30 | 20 | ----- | ----- | -- | ----- | ------ | -- | ------ | ------ | --- | ------ |
| ------ | [a]3.35 | 12 | ----- | ----- | -- | ----- | ------ | -- | ----- | ----- | -- | ----- | ------ | -- | ------ | ------ | --- | ------ |
| ------ | ------ | -- | ----- | ----- | -- | ----- | [a]2.98 | 60 | ----- | ----- | -- | ----- | ------ | -- | ------ | ------ | --- | ------ |
| 220 | 2.86 | 50 | 8.089 | 2.84 | 30 | 8.033 | 2.87 | 60 | 8.118 | ----- | -- | ----- | 2.858 | mw | 8.0836 | 2.858 | 40 | 8.084 |
| ------ | ------ | -- | ----- | ----- | -- | ----- | ------ | -- | ----- | ----- | -- | ----- | ------ | -- | ------ | ------ | --- | ------ |
| 311 | 2.43 | 100 | 8.059 | 2.41 | 100 | 7.993 | 2.41 | 100 | 7.993 | ----- | -- | ----- | 2.437 | s | 8.0826 | 2.436 | 100 | 8.079 |
| 222 | 2.34 | 12 | 8.106 | 2.31 | 2 | 8.002 | 2.32 | 20 | 8.037 | ----- | -- | ----- | 2.333 | vw | 8.0817 | 2.333 | 3 | 8.082 |
| ------ | [a]2.25 | 10 | ----- | ----- | -- | ----- | ------ | -- | ----- | ----- | -- | ----- | ------ | -- | ------ | ------ | --- | ------ |
| 400 | 2.02 | 80 | 8.080 | 2.00 | 63 | 8.000 | 2.01 | 80 | 8.040 | 2.019 | 50 | 8.08 | 2.021 | ms | 8.0840 | 2.021 | 58 | 8.084 |
| ------ | 1.82 | 2 | ----- | ----- | -- | ----- | 1.815 | 20 | ----- | ----- | -- | ----- | ------ | -- | ------ | ------ | --- | ------ |
| ------ | ------ | -- | ----- | ----- | -- | ----- | ------ | -- | ----- | ----- | -- | ----- | ------ | -- | ------ | ------ | --- | ------ |
| ------ | [a]1.73 | 10 | ----- | ----- | -- | ----- | ------ | -- | ----- | ----- | -- | ----- | ------ | -- | ------ | ------ | --- | ------ |
| 422 | 1.65 | 30 | 8.083 | 1.63 | 8 | 7.985 | 1.641 | 60 | 8.039 | 1.652 | 20 | 8.09 | 1.649 | w | 8.0784 | 1.649 | 10 | 8.078 |
| ------ | [a]1.59 | 6 | ----- | ----- | -- | ----- | ------ | -- | ----- | ----- | -- | ----- | ------ | -- | ------ | ------ | --- | ------ |
| 511 | 1.55 | 80 | 8.054 | 1.54 | 40 | 8.002 | 1.545 | 80 | 8.028 | 1.555 | 70 | 8.08 | 1.555 | ms | 8.0800 | 1.555 | 45 | 8.080 |
| ------ | [a]1.47 | <1 | ----- | ----- | -- | ----- | ------ | -- | ----- | ----- | -- | ----- | ------ | -- | ------ | ------ | --- | ------ |
| 440 | 1.43 | 100 | 8.089 | 1.420 | 75 | 8.033 | 1.423 | 100 | 8.050 | 1.432 | 100 | 8.10 | 1.4288 | s | 8.0825 | 1.429 | 58 | 8.084 |
| ------ | ------ | -- | ----- | ----- | -- | ----- | ------ | -- | ----- | ----- | -- | ------ | ------ | -- | ------ | ------ | --- | ------ |
| 531 | 1.37 | 16 | 8.105 | 1.361 | 1 | 8.052 | ------ | -- | ----- | 1.363 | 10 | 8.07 | 1.3662 | w | 8.0825 | 1.366 | 3 | 8.081 |
| ------ | 1.35 | 5 | ----- | ----- | -- | ----- | ------ | -- | ----- | ----- | -- | ----- | ------ | -- | ------ | ------ | --- | ------ |
| ------ | ------ | -- | ----- | ----- | -- | ----- | [a]1.291 | 20 | ----- | ----- | -- | ----- | ------ | -- | ------ | ------ | --- | ------ |
| 620 | 1.28 | 17 | 8.095 | 1.271 | 2 | 8.039 | 1.277 | 40 | 8.076 | 1.276 | 10 | 8.07 | 1.2779 | w | 8.0821 | 1.278 | 2 | 8.083 |
| ------ | ------ | -- | ----- | ----- | -- | ----- | [a]1.250 | 20 | ----- | ----- | -- | ----- | ------ | -- | ------ | ------ | --- | ------ |
| 533 | 1.23 | 30 | 8.066 | 1.225 | 6 | 8.033 | 1.229 | 60 | 8.059 | 1.232 | 30 | 8.09 | 1.2326 | mw | 8.0827 | 1.232 | 9 | 8.079 |
| 622 | 1.22 | 12 | 8.093 | ----- | -- | ----- | 1.192 | 20 | ----- | ----- | -- | ----- | 1.2185 | w | 8.0826 | 1.218 | 1 | 8.079 |
| 444 | 1.17 | 25 | 8.106 | 1.160 | 3 | 8.037 | 1.162 | 60 | 8.051 | 1.164 | 20 | 8.07 | 1.1666 | mw | 8.0824 | 1.1662 | 7 | 8.0797 |
| 711 | 1.13 | 16 | 8.070 | ----- | -- | ----- | 1.137 | 40 | 8.120 | 1.132 | 10 | 8.09 | 1.1318 | w | 8.0827 | 1.1312 | 2 | 8.0784 |
| ------ | 1.12 | 2 | ----- | ----- | -- | ----- | ------ | -- | ----- | ----- | -- | ----- | ------ | -- | ------ | ------ | --- | ------ |
| 642 | 1.08 | 22 | 8.082 | 1.069 | 2 | 8.000 | 1.077 | 60 | 8.060 | 1.080 | 10 | 8.09 | 1.0801 | mw | 8.0827 | 1.0796 | 4 | 8.0790 |
| 731 | 1.05 | 40 | 8.065 | ----- | -- | ----- | 1.049 | 70 | 8.056 | 1.051 | 30 | 8.08 | 1.0522 | ms | 8.0821 | 1.0518 | 12 | 8.0790 |
| ------ | [a]1.04 | 5 | ----- | 1.042 | 8 | ----- | [a]1.032 | 20 | ----- | ----- | -- | ----- | ------ | -- | ------ | ------ | --- | ------ |
| 800 | 1.01 | 30 | 8.080 | 1.000 | 3 | 8.000 | 1.008 | 60 | 8.064 | 1.011 | 10 | 8.09 | 1.0102 | m | 8.0816 | 1.0100 | 5 | 8.0800 |
| ------ | 0.987 | 4 | ----- | ----- | -- | ----- | ------ | -- | ----- | ----- | -- | ----- | ------ | -- | ------ | ------ | --- | ------ |
| 822 | .950 | 18 | 8.061 | 0.943 | 1 | 8.002 | ------ | -- | ----- | 0.953 | 10 | 8.09 | 0.9525 | mw | 8.0822 | 0.9522 | 3 | 8.0797 |
| 751 | .933 | 40 | 8.080 | .925 | 2 | ----- | ------ | -- | ----- | .933 | 20 | 8.080 | .9333 | s | 8.0826 | .9330 | 10 | 8.0800 |
| ------ | [a].927 | 10 | ----- | ----- | -- | ----- | ------ | -- | ----- | ----- | -- | ----- | .9271 | w | 8.0823 | ------ | --- | ------ |
| ------ | [a].916 | 6 | ----- | ----- | -- | ----- | ------ | -- | ----- | ----- | -- | ----- | ------ | -- | ------ | ------ | --- | ------ |
| 840 | .903 | 30 | 8.077 | 0.895 | 1 | ----- | ------ | -- | ----- | 0.904 | 10 | 8.086 | 0.9036 | s | 8.0820 | 0.9034 | 6 | 8.0803 |
| 911 | .887 | 15 | 8.081 | ----- | -- | 8.154 | ------ | -- | ----- | ----- | -- | ----- | ------ | -- | ------ | .8869 | <1 | 8.0800 |
| 664 | .861 | 12 | 8.077 | ----- | -- | ----- | ------ | -- | ----- | ----- | -- | ----- | ------ | -- | ------ | .8613 | <1 | 8.0800 |
| 931 | .847 | 40 | 8.080 | 0.840 | 2 | 8.013 | ------ | -- | ----- | 0.848 | 10 | 8.089 | ------ | -- | ------ | .8469 | 10 | 8.0789 |
| 844 | .824 | 70 | 8.074 | ----- | -- | ----- | ------ | -- | ----- | .824 | 10 | 8.074 | ------ | -- | ------ | .8247 | 20 | 8.0804 |
| 933 | .812 | 14 | 8.079 | 0.819 | 4 | 8.149 | ------ | -- | ----- | ----- | -- | ----- | ------ | -- | ------ | ------ | --- | ------ |
| 10·2·0 | .792 | 25 | 8.077 | ----- | -- | ----- | ------ | -- | ----- | ----- | -- | ----- | ------ | -- | ------ | ------ | --- | ------ |
| 951 | ------ | -- | ----- | 0.775 | 2 | 8.017 | ------ | -- | ----- | 0.782 | 10 | 8.089 | ------ | -- | ------ | ------ | --- | ------ |
| 880 | ------ | -- | ----- | ----- | -- | ----- | ------ | -- | ----- | ----- | -- | ----- | ------ | -- | ------ | ------ | --- | ------ |
| 11·5·3 | ------ | -- | ----- | ----- | -- | ----- | ------ | -- | ----- | ----- | -- | ----- | ------ | -- | ------ | ------ | --- | ------ |
| 12·4·0 | ------ | -- | ----- | ----- | -- | ----- | ------ | -- | ----- | ----- | -- | ----- | ------ | -- | ------ | ------ | --- | ------ |
| 10·6·6 | ------ | -- | ----- | ----- | -- | ----- | ------ | -- | ----- | ----- | -- | ----- | ------ | -- | ------ | ------ | --- | ------ |
| Average value of last five lines | | | 8.078 | ----- | -- | 8.067 | ------ | -- | 8.070 | ----- | -- | 8.084 | ------ | -- | ------ | ------ | --- | 8.0800 |

[a] This line is not permitted by the space group of spinel.

**Interplanar spacing and intensity measurements.** All of the $d$-spacings were converted from kX to angstrom units except those of Taylor and Kordes, which were calculated from the Bragg angle data in their published articles. The first Posnjak pattern and those of Hansen and Brownmiller and Passerini were combined on one ASTM card. Four of the patterns contain lines not allowed by the space group of spinel.

The three strongest lines for each of the patterns are as follows:

| Pattern | 1 | 2 | 3 |
|---|---|---|---|
| Posnjak | 311 | 440 | 400 |
| Posnjak | 440 | 311 | 400 |
| Hansen and Brownmiller | 311 | 400 | 511 |
| Passerini | 440 | 400 | 731 |
| Taylor | 311 | 400 | 511 |
| Hauptmann and Novak | 440 | 311 | 400 |
| Kordes | 311 | 440 | 400 |
| Hanawalt, Rinn, and Frevel | 311 | 440 | 400 |
| British Museum | 311 | 440 | 111 |
| Clark, Howe, and Badger | 440 | 511 | 400 |
| Andrews | 311 | 440 | 751 |
| Swanson and Fuyat | 311 | 400 | 440 |

**Lattice constants.** The structure was determined by both W. H. Bragg [10] and S. Nishikawa [11] in 1915. The face-centered cubic lattice has space group $O_h^7$-Fd3m, spinel-structure type and $8(MgAl_2O_4)$ per unit cell.

Several unit-cell measurements supposedly published in kX units have been converted to angstroms for comparison with the NBS value.

*Lattice constant in angstroms*

| | | |
|---|---|---|
| 1928 | Posnjak [1] | 8.04 |
| 1930 | Passerini [3] | 8.066 |
| 1930 | Taylor [4] | 8.086 |
| 1931 | Hauptmann and Novak [5] | 8.075 |
| 1934 | Clark, Howe, and Badger [8] | 8.080 |
| 1935 | Kordes [6] | 8.08681 |
| 1951 | Anderson [9] | 8.082 |
| 1953 | Swanson and Fuyat | 8.0800 at 26°C |

The density of magnesium aluminate calculated from the NBS lattice constant is 3.581 at 26°C. The index of refraction of the NBS sample is 1.712.

**References**


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**Zinc aluminate, gahnite, $ZnAl_2O_4$ (cubic)**
**ASTM cards**

| Card number | | New index lines | Radiation | Source |
|---|---|---|---|---|
| Old | New | | | |
| II-3900 | 3877 | 1.42 | Iron | Passerini [1] 1930. |
| | 2-1413 | 2.42 | | |
| | 2-1415 | 1.55 | | |
| ------- | 3872 | 1.43 | Copper, | Hauptmann and Novak |
| | 3-1162 | 2.44 | 1.539. | [2] 1932. |
| | 3-1161 | 1.56 | | |
| 2939 | 3119 | 2.44 | Molybdenum | Hanawalt, Rinn, and |
| | 1-1146 | 2.85 | | Frevel [3] 1938. |
| | 1-1146 | 1.43 | | |

The ASTM card for the Passerini pattern lists molybdenum radiation when iron was actually used.

**Additional published patterns**

| Source | Radiation | Wavelength |
|---|---|---|
| Clark, Ally, and Badger [4] | Molybdenum | 0.712 |
| Andrews [5] 1951 | Cobalt | 1.7889 |

**NBS pattern.** The zinc aluminate sample used for the NBS pattern was prepared at the Radio Corporation of America Laboratories by heating the oxides at 1,600°C for 10 minutes. Spectrographic analysis at the NBS showed 0.1 to 1.0 percent of silicon, 0.01 to 0.1 percent each of arsenic, boron, iron, magnesium, and nickel, 0.001 to 0.01 percent each of calcium, copper, germanium, indium, platinum, and titanium, and less than 0.001 percent each of manganese, lead, and tin.

**Interplanar spacings and intensity measurements.** The Passerini and the Hauptmann and Novak *d*-spacings expressed in angstroms were calculated from their Bragg angle data, while the Hanawalt, Rinn, and Frevel, the Clark, Ally, and Badger, and the Andrews *d*-spacings supposedly expressed in kX units were converted to angstroms. The *d*-values for the first five lines of Passerini's pub-

**Zinc aluminate, $ZnAl_2O_4$**

| *hkl* | 1930 Passerini Fe, 1.93597 A | | | 1931 Hauptmann and Novak Cu, 1.5405 A | | | | | | 1938 Hanawalt, Rinn, and Frevel Mo, 0.709 A | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | Without Ag standard | | | With Ag standard | | | | | |
| | *d* | *I* | *a* | *d* | *I* | *a* | *d* | *I* | *a* | *d* | *I* | *a* |
| | A | | A | A | | A | A | | A | A | | A |
| 111 | ------- | ----- | ------- | ------- | ----- | ------- | ------- | ----- | ------- | ------- | ----- | ------- |
| 220 | 2.765 | ms | 7.832 | 2.864 | 80 | 8.101 | 2.875 | 89 | 8.132 | 2.86 | 53 | 8.089 |
| 311 | 2.378 | s | 7.900 | 2.440 | 90 | 8.093 | 2.464 | 100 | 8,175 | 2.44 | 100 | 8.093 |
| 222 | ------- | ----- | ------- | ------- | ----- | ------- | ------- | ----- | ------- | ------- | ----- | ------- |
| 400 | 1.984 | w | 7.944 | 2.030 | 10 | 8.120 | ------- | ----- | ------- | 2.02 | 7 | 8.080 |
| ------- | ------- | ----- | ------- | ------- | ----- | ------- | ------- | ----- | ------- | 1.91 | 7 | ------- |
| 331 | ------- | ----- | ------- | 1.857 | 30 | 8.094 | ------- | ----- | ------- | 1.85 | 7B | 8.064 |
| 422 | 1.627 | ms | 7.976 | 1.654 | 70 | 8.103 | 1.659 | 56 | 8.127 | 1.65 | 13 | 8.083 |
| 511 | 1.539 | s | 7.997 | 1.559 | 90 | 8.101 | 1.560 | 100 | 8.106 | 1.55 | 33 | 8.054 |
| ------- | ------- | ----- | ------- | ------- | ----- | ------- | ------- | ----- | ------- | 1.483 | 7 | ------- |
| 440 | 1.417 | vs | 8.021 | 1.430 | 100 | 8.089 | 1.430 | 100 | 8.089 | 1.433 | 40 | 8.106 |
| 531 | 1.358 | w | 8.040 | 1.370 | 0-10 | 8.105 | ------- | ----- | ------- | ------- | ----- | ------- |
| 620 | 1.270 | ms | 8.045 | 1.283 | 40 | 8.114 | ------- | ----- | ------- | ------- | ----- | ------- |
| 533 | 1.227 | m | 8.046 | 1.237 | 60 | 8.112 | ------- | ----- | ------- | 1.234 | 7 | 8.092 |
| 622 | 1.213 | mw | 8.046 | ------- | ----- | ------- | ------- | ----- | ------- | ------- | ----- | ------- |
| 444 | 1.159 | w | 8.037 | 1.1705 | 20 | 8.1095 | ------- | ----- | ------- | ------- | ----- | ------- |
| 711 | 1.127 | mw | 8.063 | ------- | ----- | ------- | ------- | ----- | ------- | ------- | ----- | ------- |
| 642 | 1.077 | s | 8.060 | 1.0836 | 80 | 8.1089 | 1.0851 | 56 | 8.1201 | ------- | ----- | ------- |
| 731 | 1.050 | s | 8.065 | 1.0558 | 90 | 8.1098 | 1.0532 | 78 | 8.0898 | ------- | ----- | ------- |
| 800 | 1.009 | ms | 8.072 | 1.0137 | 50 | 8.1096 | 1.0120 | 56 | 8.0960 | ------- | ----- | ------- |
| 733 | ------- | ----- | ------- | ------- | ----- | ------- | ------- | ----- | ------- | ------- | ----- | ------- |
| 822 | ------- | ----- | ------- | 0.9545 | 70 | 8.0992 | 0.9562 | 44 | 8.1136 | ------- | ----- | ------- |
| 751 | ------- | ----- | ------- | .9358 | 90 | 8.1043 | ------- | ----- | ------- | ------- | ----- | ------- |
| 662 | ------- | ----- | ------- | ------- | ----- | ------- | ------- | ----- | ------- | ------- | ----- | ------- |
| 840 | ------- | ----- | ------- | ------- | ----- | ------- | ------- | ----- | ------- | ------- | ----- | ------- |
| 911 | ------- | ----- | ------- | ------- | ----- | ------- | ------- | ----- | ------- | ------- | ----- | ------- |
| 664 | ------- | ----- | ------- | ------- | ----- | ------- | ------- | ----- | ------- | ------- | ----- | ------- |
| 931 | ------- | ----- | ------- | 0.8488 | 90 | 8.0970 | ------- | ----- | ------- | ------- | ----- | ------- |
| 844 | ------- | ----- | ------- | .8251 | 100 | 8.0843 | ------- | ----- | ------- | ------- | ----- | ------- |
| 933 | ------- | ----- | ------- | ------- | ----- | ------- | ------- | ----- | ------- | ------- | ----- | ------- |
| 10·2·0 | ------- | ----- | ------- | ------- | ----- | ------- | ------- | ----- | ------- | ------- | ----- | ------- |
| 9·5·1 | ------- | ----- | ------- | ------- | ----- | ------- | ------- | ----- | ------- | ------- | ----- | ------- |
| Average of last five lines | | | 8.059 | ------- | ----- | 8.0989 | ------- | ----- | [a]8.1049 | ------- | ------ | 8.080 |

[a] Last four lines only.

*(Continued)*

| *hkl* | 1931 Clark, Ally, and Badger Mo, 0.709 A | | | 1951 Andrews Co, 1.7889 A | | | 1953 Swanson and Fuyat Cu, 1.5405 A, 26°C | | |
|---|---|---|---|---|---|---|---|---|---|
| | *d* | *I* | *a* | *d* | *I* | *a* | *d* | *I* | *a* |
| | *A* | | *A* | *A* | | *A* | *A* | | *A* |
| 111 | | | | 4.67 | vvw | 8.0887 | 4.67 | 3 | 8.089 |
| 220 | 2.84 | 80 | 8.033 | 2.858 | ms | 8.0836 | 2.861 | 84 | 8.092 |
| 311 | 2.42 | 100 | 8.026 | 2.437 | s | 8.0826 | 2.438 | 100 | 8.086 |
| 222 | | vvw | | 2.334 | vvw | 8.0852 | 2.335 | 1 | 8.089 |
| 400 | 2.007 | 30 | 8.028 | 2.021 | w | 8.0840 | 2.021 | 8 | 8.084 |
| | | | | | | | | | |
| 331 | | | | 1.855 | mw | 8.0858 | 1.855 | 10 | 8.086 |
| 422 | 1.643 | 40 | 8.049 | 1.650 | m | 8.0833 | 1.650 | 24 | 8.083 |
| 511 | 1.544 | 90 | 8.023 | 1.555 | s | 8.0800 | 1.556 | 40 | 8.085 |
| | | | | | | | | | |
| 440 | 1.420 | 90 | 8.032 | 1.4290 | vs | 8.0836 | 1.429 | 43 | 8.084 |
| 531 | 1.355 | 5 | 8.016 | 1.3664 | vvw | 8.0837 | 1.367 | 1 | 8.087 |
| 620 | 1.269 | 20 | 8.026 | 1.2781 | m | 8.0834 | 1.278 | 6 | 8.083 |
| 533 | 1.225 | 30 | 8.033 | 1.2327 | m | 8.0834 | 1.233 | 9 | 8.085 |
| 622 | | | | 1.2186 | vvw | 8.0833 | 1.219 | 1 | 8.086 |
| 444 | 1.161 | 5 | 8.044 | 1.1668 | vvw | 8.0838 | 1.1670 | <1 | 8.0852 |
| 711 | | vvw | | 1.1319 | vw | 8.0834 | 1.1322 | 1 | 8.0856 |
| 642 | 1.074 | 30 | 8.037 | 1.0802 | m | 8.0835 | 1.0803 | 9 | 8.0842 |
| 731 | 1.046 | 40 | 8.034 | 1.0523 | ms | 8.0829 | 1.0525 | 12 | 8.0844 |
| 800 | 1.005 | 10 | 8.040 | 1.0104 | m | 8.0832 | 1.0104 | 4 | 8.0832 |
| 733 | | | | 0.9875 | w | 8.0830 | 0.9875 | 1 | 8.0830 |
| 822 | 0.947 | 20 | 8.036 | .9526 | m | 8.0831 | .9527 | 5 | 8.0839 |
| 751 | | | | .9334 | ms | 8.0835 | .9334 | 9 | 8.0835 |
| 662 | 0.927 | 20 | [b] 8.081 | .9272 | vvw | 8.0831 | .9273 | 1 | 8.0840 |
| 840 | | | | | | | .9039 | <1 | 8.0847 |
| 911 | | | | | | | .8874 | <1 | 8.0846 |
| 664 | | | | | | | .8619 | 2 | 8.0853 |
| 931 | 0.842 | 20 | 8.032 | | | | .8475 | 7 | 8.0846 |
| 844 | .821 | 20 | 8.044 | | | | .8251 | 13 | 8.0843 |
| 933 | | | | | | | .8126 | 1 | 8.0853 |
| 10·2·0 | 0.788 | 20 | 8.036 | | | | | | |
| 9·5·1 | .777 | 20 | 8.037 | | | | | | |
| Average of last five lines | | | 8.037 | | | | | | 8.0848 |

[b] Not used for average.

lished pattern were in poor agreement with the rest of his pattern as noted by the corresponding unit-cell values. It is apparent that those lines were omitted from the ASTM cards, and calculated values based on his unit cell were substituted.

Hauptmann and Novak listed two patterns, the first contains more lines (no internal standard) than the second (a silver standard). The Hanawalt, Rinn, and Frevel pattern contains a line at 1.91 A of intensity 7, not indexed, and presumably not belonging to the pattern.

The three strongest lines for each pattern are as follows:

| Pattern | 1 | 2 | 3 |
|---|---|---|---|
| Passerini | 440 | 311 | 511 |
| Hauptmann and Novak, without standard | 440 | 844 | 311 |
| Hauptmann and Novak, with standard | 311 | 511 | 440 |
| Hanawalt, Rinn, and Frevel | 311 | 220 | 440 |
| Clark, Ally, and Badger | 311 | 511 | 440 |
| Andrews | 440 | 311 | 511 |
| Swanson and Fuyat | 311 | 220 | 440 |

**Lattice constants.** The structure was determined by W. H. Bragg [6] in 1915. The face-centered cubic lattice has space group $O_h^7$-Fd3m, spinel-structure type and 8($ZnAl_2O_4$) per unit cell.

Several unit-cell measurements supposedly published in kX units have been converted to angstroms for comparison with the NBS value.

*Lattice constant in angstroms*

| | | |
|---|---|---|
| 1930 | Passerini [1] | 8.066 |
| 1931 | Hauptmann and Novak [2] | 8.109 |
| 1931 | Clark, Ally, and Badger [4] | 8.078 |
| 1932 | Krause and Thiel [7] | 8.109 |
| 1949 | Kordes and Becker [8] | 8.083 |
| 1951 | Andrews [5] | 8.083 |
| 1953 | Swanson and Fuyat | 8.0848 at 26°C |

The density of zinc aluminate calculated from the NBS lattice constant is 4.607 at 26°C. The refractive index of the NBS sample is 1.775.

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# 2.4. Halides

## Sodium chloride (halite), NaCl (cubic)

**ASTM cards**

| Card number | | New index lines | Radiation | Source |
|---|---|---|---|---|
| Old | New | | | |
| 2412 | 2551 | 2.81 | Molybdenum-- | Davey [1] 1923. |
| | 1-0997 | 1.99 | | |
| | 1-0994 | 1.26 | | |
| II-1712 | 2550 | 2.81 | Molybdenum, 0.707831. | Ksanda [2] 1931. |
| | 2-0815 | 1.99 | | |
| | 2-0818 | 1.26 | | |
| 2424 | 2548 | 2.81 | Molybdenum-- | Hanawalt, Rinn, and Frevel [3] 1938. |
| | 1-0996 | 1.99 | | |
| | 1-0993 | 1.63 | | |

**Additional published patterns**

| Source | Radiation | Wavelength |
|---|---|---|
| Olshausen [4] 1925 | Copper | 1.53923 |
| Waldo [5] 1935 | | |
| Brill, Grimm, Herman, Peters [6] 1939 | Molybdenum | |
| Wasastjerna [7] 1944 | Copper | |
| Sidhu [8] 1948 | Copper | 1.539 |

**NBS pattern.** The ACS standard sample of sodium chloride used for the NBS pattern was recrystallized twice from hydrochloric acid.

**Interplanar spacings and intensity measurements.** The *d*-spacings for the Davey and Olshausen patterns were not converted to angstroms as no wave length data was stated and no assumptions could be made. All of the other spacings were assumed to be in kX units and were converted to angstroms. The Ksanda and Wasastjerna patterns, both made with exceptional care for special studies, compare closely with the NBS pattern. The pattern of Brill, Grimm, Hermann, and Peters was made in the course of a Fourier analysis with intensities corrected to their powder equivalents. The 731 line of that pattern with *d*-spacing 0.741 A appears to be in error as the unit cell derived from that value is greatly different from the others.

The three strongest lines for each of the patterns are as follows:

| Pattern | 1 | 2 | 3 |
|---|---|---|---|
| Davey | 200 | 220 | 420 |
| Ksanda | 200 | 220 | 420 |
| Hanawalt, Rinn, and Frevel | 200 | 220 | 222 |
| Olshausen | 200 | 220 | 222 |
| Waldo | 200 | 220 | 222 |
| Brill, Grimm, Hermann, and Peters | 200 | 220 | 222 |
| Sidhu | 200 | 220 | 222 |
| Swanson and Fuyat | 200 | 220 | 222 |

**Lattice constants.** The structure was determined by Bragg and Bragg [10] in 1913. The face-centered cubic lattice has space group $O_h^5$-Fm3m, sodium-chloride-structure type, and 4(NaCl) per unit cell.

The following unit-cell values were converted to angstroms, and the two values of Straumanis and Ieviņš were temperature corrected to 26°C for comparison with the NBS values. The linear coefficient of expansion is $4.04 \times 10^{-5}$, according to van Bergen [15].

*Lattice constant in angstroms*

| | | |
|---|---|---|
| 1925 | Olshausen [4] | [a]5.638 |
| 1927 | Barth and Lunde [11] | [a]5.639<br>5.637 |
| 1931 | Ksanda [2] | 5.639 |
| 1932 | Bradley and Jay [12] | 5.637 |
| 1936 | Straumanis and Ieviņš [13] | [a]5.64031 at 26°C<br>5.64056 at 26°C |
| 1937 | Moeller [14] | 5.6397 at 25°C |
| 1941 | Bergen [15] | 5.64006 |
| 1948 | Sidhu [8] | 5.639 |
| 1948 | Mehmel [16] | 5.64 |
| 1953 | Swanson and Fuyat | 5.6402 at 26°C |

[a]The natural rocksalt values as opposed to the other values determined on synthetically prepared sodium chloride.

The density of sodium chloride calculated from the NBS lattice constant is 2.164 at 26°C. The refractive index of the NBS sample is 1.542.

**Sodium chloride, NaCl**

| *hkl* | 1923 Davey Mo, 0.709 A | | | 1931 Ksanda Mo, 0.709 A | | | 1938 Hanawalt, Rinn, and Frevel Mo, 0.709 A | | | 1925 Olshausen Cu, 1.53923 A | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | *d* | *I* | *a* | *d* | *I* | *a* | *d* | *I* | *a* | *d* | *I* | *a* |
| | A | | A | A | | A | A | | A | A | | A |
| 111 | 3.249 | 3 | 5.627 | 3.2558 | 40 | 5.6392 | 3.26 | 5 | 5.646 | 3.331 | w | 5.769 |
| 200 | 2.814 | 100 | 5.628 | 2.8197 | 100 | 5.6394 | 2.82 | 100 | 5.640 | 2.873 | s | 5.746 |
| 220 | 1.990 | 90 | 5.629 | 1.9938 | 90 | 5.6393 | 1.99 | 83 | 5.629 | 1.989 | s | 5.626 |
| 311 | 1.697 | 1 | 5.628 | 1.7003 | 10 | 5.6393 | 1.70 | 2 | 5.638 | 1.690 | w | 5.605 |
| 222 | 1.625 | 15 | 5.629 | 1.6279 | 70 | 5.6392 | 1.63 | 33 | 5.646 | 1.623 | m | 5.622 |
| 400 | 1.407 | 4 | 5.628 | 1.4098 | 50 | 5.6392 | 1.413 | 13 | 5.652 | 1.404 | m | 5.616 |
| 331 | 1.291 | 1 | 5.627 | 1.2937 | 10 | 5.6291 | 1.296 | 1 | 5.649 | 1.290 | w | 5.622 |
| 420 | 1.259 | 20 | 5.630 | 1.2609 | 80 | 5.6389 | 1.263 | 33 | 5.648 | 1.260 | m | 5.639 |
| 422 | 1.149 | 10 | 5.629 | 1.1512 | 60 | 5.6397 | 1.152 | 20 | 5.644 | 1.150 | m | 5.634 |
| 511 | 1.083 | 1 | 5.627 | 1.0852 | 5 | 5.6389 | 1.082 | 1 | 5.622 | 1.084 | w | 5.633 |
| 440 | 0.995 | 2 | 5.629 | 0.9968 | 20 | 5.6388 | 0.999 | 30 | 5.651 | 0.9965 | m | 5.6371 |
| 531 | .951 | 1 | 5.626 | .9532 | 5 | 5.6392 | --- | --- | --- | .9521 | w | 5.6327 |
| 600 | .938 | 3 | 5.628 | .9399 | 30 | 5.6394 | 0.943 | 5 | 5.658 | .9401 | m | 5.6406 |
| 620 | .890 | 2 | 5.629 | .8916 | 20 | 5.6390 | .894 | 3 | 5.654 | .8942 | m | 5.6554 |
| 533 | .858 | 1 | 5.626 | .8599 | 10 | 5.6387 | --- | --- | --- | .8635 | vw | 5.6623 |
| 622 | .848 | 1 | 5.625 | .8501 | 10 | 5.6389 | 0.854 | 3 | 5.665 | .8539 | m | 5.6641 |
| 444 | .812 | 0.2 | 5.626 | .8139 | 5 | 5.6389 | .815 | 1 | 5.646 | --- | --- | --- |
| 711 | .788 | --- | 5.627 | --- | --- | --- | --- | --- | --- | --- | --- | --- |
| 640 | .781 | 0.5 | 5.632 | 0.7820 | 5 | 5.6390 | 0.784 | 1 | 5.654 | --- | --- | --- |
| 642 | .752 | 1 | 5.627 | .7535 | 10 | 5.6387 | .757 | 1 | 5.665 | --- | --- | --- |
| 731 | .733 | --- | 5.630 | --- | --- | --- | --- | --- | --- | --- | --- | --- |
| 800 | .703 | --- | 5.624 | --- | --- | --- | --- | --- | --- | --- | --- | --- |
| 733 | .688 | --- | 5.632 | --- | --- | --- | --- | --- | --- | --- | --- | --- |
| 820 | .682 | --- | 5.624 | --- | --- | --- | --- | --- | --- | --- | --- | --- |
| Average value of last five lines | | | 5.627 | --- | --- | 5.6388 | --- | --- | 5.657 | --- | --- | 5.6510 |

*(Continued)*

| hkl | 1935 Waldo | | | 1939 Brill, Grimm, Hermann, and Peters Mo, 0.709 A | | | 1944 Wasastjerna Cu, 1.5405 A | | 1948 Sidhu Cu, 1.539 A | | | 1953 Swanson and Fuyat Cu, 1.5405 A, 26°C | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | d | I | a | d | I | a | d | a | d | I | a | d | I | a |
| | A | | A | A | | A | A | A | A | | A | A | | A |
| 111 | 3.26 | vw | 5.646 | 3.253 | 6 | 5.634 | 3.255 | 5.638 | 3.25 | w | 5.64 | 3.258 | 13 | 5.643 |
| 200 | 2.82 | s | 5.640 | 2.822 | 100 | 5.644 | 2.820 | 5.638 | 2.81 | vs | 5.63 | 2.821 | 100 | 5.642 |
| 220 | 1.994 | s | 5.640 | 1.996 | 44 | 5.646 | -------- | -------- | 1.99 | s | 5.64 | 1.994 | 55 | 5.640 |
| 311 | 1.699 | vvw | 5.635 | 1.701 | <1 | 5.642 | 1.700 | 5.638 | 1.70 | vw | 5.65 | 1.701 | 2 | 5.642 |
| 222 | 1.628 | m | 5.640 | 1.629 | 28 | 5.643 | 1.628 | 5.640 | 1.63 | m | 5.65 | 1.628 | 15 | 5.640 |
| 400 | 1.410 | w | 5.640 | 1.411 | 18 | 5.644 | 1.410 | 5.638 | 1.41 | w | 5.63 | 1.410 | 6 | 5.640 |
| 331 | 1.294 | vvw | 5.640 | 1.295 | <1 | 5.645 | 1.294 | 5.640 | 1.29 | vw | 5.63 | 1.294 | 1 | 5.640 |
| 420 | 1.261 | m | 5.639 | ------- | --- | ------ | 1.2610 | 5.6398 | 1.27 | m | 5.64 | 1.261 | 11 | 5.639 |
| 422 | 1.151 | m | 5.639 | 1.191 | 9 | 5.835 | 1.1512 | 5.6398 | 1.15 | m | 5.64 | 1.1515 | 7 | 5.6412 |
| 511 | ------- | --- | ------- | 1.086 | <1 | 5.643 | 1.0854 | 5.6398 | 1.08 | vw | 5.62 | 1.0855 | 1 | 5.6404 |
| 440 | 0.997 | w | 5.640 | 0.997 | 5 | 5.640 | 0.9968 | 5.6390 | 0.996 | w | 5.629 | 0.9969 | 2 | 5.6393 |
| 531 | ------- | --- | ------- | ------- | --- | ------ | .9532 | 5.6393 | .952 | vw | 5.631 | .9533 | 1 | 5.6398 |
| 600 | 0.940 | w | 5.640 | 0.940 | 4 | 5.640 | .9400 | 5.6399 | .940 | m | 5.639 | .9401 | 3 | 5.6406 |
| 620 | .892 | w | 5.642 | ------- | --- | ------ | .8916 | 5.6392 | .892 | m | 5.640 | .8917 | 4 | 5.6396 |
| 533 | ------- | --- | ------- | 0.860 | <1 | 5.639 | .8599 | 5.6391 | .859 | vw | 5.631 | .8601 | 1 | 5.6401 |
| 622 | 0.850 | w | 5.638 | .851 | 3 | 5.645 | .8502 | 5.6395 | ------- | -- | ------- | .8503 | 3 | 5.6403 |
| 444 | .814 | vvw | 5.640 | .814 | 2 | 5.640 | .8139 | 5.6395 | ------- | -- | ------- | .8141 | 2 | 5.6403 |
| 711 | ------- | --- | ------- | .790 | <1 | 5.642 | -------- | -------- | ------- | -- | ------- | -------- | --- | -------- |
| 640 | 0.782 | vw | 5.639 | ------- | ---- | ------ | -------- | -------- | ------- | -- | ------- | -------- | --- | -------- |
| 642 | .754 | vw | 5.642 | ------- | --- | ------ | -------- | -------- | ------- | -- | ------- | -------- | --- | -------- |
| 731 | ------- | --- | ------- | 0.741 | <1 | [a]5.692 | -------- | -------- | ------- | -- | ------- | -------- | --- | -------- |
| 800 | ------- | --- | ------- | .705 | 1 | 5.640 | -------- | -------- | ------- | -- | ------- | -------- | --- | -------- |
| 733 | ------- | --- | ------- | .689 | <1 | 5.640 | -------- | -------- | ------- | -- | ------- | -------- | --- | -------- |
| 820 | ------- | --- | ------- | [b].684 | <1 | 5.640 | -------- | -------- | ------- | -- | ------- | -------- | --- | -------- |
| Average value of last five lines | | | 5.640 | ------- | --- | 5.640 | -------- | 5.6394 | ------- | -- | 5.634 | -------- | --- | 5.6402 |

[a] Not used to obtain average. [b] Pattern cut off here, contains 29 additional lines.

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## Cesium chloride, CsCl (cubic)

### ASTM cards

| Card number | | New index lines | Radiation | Source |
|---|---|---|---|---|
| Old | New | | | |
| 2287 | 2376 | 2.90 | Molybdenum | Davey [1] 1923. |
| | 1-0936 | 1.68 | | |
| | 1-0936 | 1.10 | | |
| II-4153 | 3957 | 1.10 | Iron | Natta and Passerini [2] 1931. |
| | 2-1443 | 1.68 | | |
| | 2-1445 | 1.30 | | |
| 2288 | 2373 | 2.90 | Molybdenum | Hanawalt, Rinn, and Frevel [3] 1938. |
| | 1-0935 | 1.68 | | |
| | 1-0935 | 4.11 | | |

### Additional published patterns

| Source | Radiation | Wavelength |
|---|---|---|
| Broch, Oftedal, and Pabst [4] 1929 | Copper | 1.539 |

**NBS pattern.** The cesium-chloride sample used for the NBS pattern was prepared at the NBS by R. B. Johannesen. Cesium dichloroiodide was recrystallized three times from dilute hydrochloric acid and heated at 500°C liberating chlorine iodide. Spectrographic analysis at the NBS showed 0.001 to 0.01 percent each of calcium, potassium, and sodium, and less than 0.001 percent each of aluminum, barium, copper, iron, magnesium, and silicon.

**Interplanar spacings and intensity measurements.** The Davey and Hanawalt, Rinn, and Frevel *d*-spacings were converted from kX to angstrom units while the Broch, Oftedal and Pabst *d*-spacings expressed in angstroms were calculated from their Bragg angle data.

The Natta and Passerini ASTM card is assumed to be a calculated pattern based on their unit cell value. Conversion of Bragg

## Cesium chloride, CsCl

| *hkl* | 1923 Davey Mo, 0.709 A | | | 1931 Natta and Passerini Mo, 0.709 A | | | 1938 Hanawalt, Rinn, and Frevel Mo, 0.709 A, 25°C | | | 1929 Broch, Oftedal, and Pabst Cu, 1.5405 A | | | 1953 Swanson and Fuyat Cu, 1.5405 A, 25°C | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | *d* | *I* | *a* | *d* | *I* | *a* | *d* | *I* | *a* | *d* | *I* | *a* | *d* | *I* | *a* |
| | A | | A | A | | A | A | | A | A | | A | A | | A |
| 100 | 4.2 | 15 | 4.20 | 4.12 | 40 | 4.12 | 4.12 | 38 | 4.12 | ------- | -- | ------- | 4.12 | 45 | 4.12 |
| 110 | 2.91 | 100 | 4.115 | 2.92 | 70 | 4.130 | 2.91 | 100 | 4.115 | 2.927 | s | 4.139 | 2.917 | 100 | 4.125 |
| 111 | 2.38 | 10 | 4.122 | 2.38 | 40 | 4.122 | 2.37 | 8 | 4.105 | 2.383 | w | 4.127 | 2.380 | 13 | 4.122 |
| 200 | 2.05 | 15 | 4.100 | 2.06 | 40 | 4.120 | 2.05 | 18 | 4.100 | 2.061 | m | 4.122 | 2.062 | 17 | 4.124 |
| 210 | 1.844 | 15 | 4.123 | 1.844 | 40 | 4.123 | 1.84 | 15 | 4.114 | 1.841 | m | 4.117 | 1.844 | 14 | 4.123 |
| 211 | 1.684 | 30 | 4.125 | 1.683 | 80 | 4.122 | 1.68 | 44 | 4.115 | 1.680 | vs | 4.115 | 1.683 | 25 | 4.122 |
| 220 | 1.457 | 15 | 4.121 | 1.457 | 50 | 4.121 | 1.457 | 8 | 4.121 | 1.453 | m | 4.110 | 1.457 | 6 | 4.121 |
| 300 | 1.375 | 10 | 4.125 | 1.373 | 50 | 4.119 | 1.377 | 5 | 4.131 | 1.370 | m | 4.110 | 1.374 | 5 | 4.122 |
| 310 | 1.306 | 15 | 4.130 | 1.302 | 80 | 4.117 | 1.303 | 10 | 4.120 | 1.301 | s | 4.114 | 1.304 | 8 | 4.124 |
| 311 | 1.241 | 5 | 4.116 | 1.242 | 40 | 4.119 | 1.242 | 3 | 4.119 | 1.241 | m- | 4.116 | 1.243 | 2 | 4.122 |
| 222 | ------- | 8 | ------- | 1.190 | 40 | 4.122 | 1.190 | 3 | 4.122 | 1.189 | m- | 4.119 | 1.1900 | 2 | 4.1223 |
| 320 | ------- | 5 | ------- | 1.142 | 40 | 4.118 | 1.142 | 3 | 4.118 | 1.141 | w | 4.114 | 1.1434 | 1 | 4.1226 |
| 321 | 1.104 | 20 | 4.131 | 1.102 | 100 | 4.123 | 1.102 | 10 | 4.123 | 1.101 | vs | 4.120 | 1.1019 | 6 | 4.1229 |
| 400 | ------- | 5 | ------- | 1.029 | 20 | 4.116 | ------- | --- | ------- | ------- | -- | ------- | 1.0309 | <1 | 4.1236 |
| 410 | 1.001 | 5 | 4.127 | ------- | --- | ------- | ------- | --- | ------- | 1.000 | m | 4.123 | 0.9997 | <1 | 4.1219 |
| 411 | 0.974 | 8 | 4.132 | ------- | --- | ------- | ------- | --- | ------- | 0.971 | s | 4.120 | .9719 | 2 | 4.1234 |
| 331 | ------- | --- | ------- | ------- | --- | ------- | ------- | --- | ------- | ------- | -- | ------- | .9459 | <1 | 4.1230 |
| 420 | 0.923 | 5 | 4.128 | ------- | --- | ------- | ------- | --- | ------- | 0.922 | m | 4.123 | .9219 | 1 | 4.1229 |
| 421 | .899 | 5 | 4.120 | ------- | --- | ------- | ------- | --- | ------- | ------- | -- | ------- | .8997 | <1 | 4.1230 |
| 332 | .879 | 5 | 4.123 | ------- | --- | ------- | ------- | --- | ------- | ------- | -- | ------- | .8791 | 1 | 4.1233 |
| 422 | ------- | 3 | ------- | ------- | --- | ------- | ------- | --- | ------- | ------- | -- | ------- | .8415 | <1 | 4.1225 |
| ------ | ------- | 5 | ------- | ------- | --- | ------- | ------- | --- | ------- | ------- | -- | ------- | ------- | --- | ------ |
| 510 | 0.808 | 5 | 4.120 | ------- | --- | ------- | ------- | --- | ------- | ------- | -- | ------- | 0.8086 | 3 | 4.1231 |
| Average value of the last five lines---- | | | 4.125 | ------- | --- | 4.120 | ------- | --- | 4.120 | ------- | -- | 4.120 | -------- | --- | 4.1230 |

angle data from their published article, whose reference is listed on that card, does not correspond with their ASTM card or with any other data. Therefore, the data presented in the table is the ASTM card pattern converted from kX to angstrom units.

The three strongest lines for each of the patterns are as follows:

| Pattern | 1 | 2 | 3 |
|---|---|---|---|
| Davey | 110 | 211 | 321 |
| Natta and Passerini | 321 | 211 | 310 |
| Hanawalt, Rinn, and Frevel | 110 | 211 | 100 |
| Broch, Oftedal, and Pabst | 211 | 321 | 110 |
| Swanson and Fuyat | 110 | 100 | 211 |

**Lattice constants.** The structure was determined by Davey and Wick [5] in 1921. The simple cubic cell has space group $O_h^1$-Pm3m, cesium-chloride-structure type and 1(CsCl) per unit cell.

A group of unit-cell determinations have been converted from kX to angstrom units for comparison with the NBS values.

*Lattice constant in angstroms*

| | | |
|---|---|---|
| 1921 | Davey and Wick [5] | 4.13 |
| 1923 | Davey [1] | 4.126 |
| 1924 | Havighurst, Mack, and Blake [6] | 4.118 |
| 1929 | Broch, Oftedal, and Pabst [4] | 4.121 |
| 1931 | Natta and Passerini [2] | 4.12 |
| 1935 | Wagner and Lippert [7] | 4.118 at 25°C |
| 1948 | Mehmel [8] | 4.12 |
| 1953 | Swanson and Fuyat | 4.123 at 25°C |

The density of cesium chloride calculated from the NBS lattice constant is 3.988 at 25°C. The refractive index of the NBS sample is 1.639.

**References**


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## Lead chloride (cotunnite), $PbCl_2$ (orthorhombic)

**ASTM cards**

| Card number | | New index lines | Radiation | Source |
|---|---|---|---|---|
| Old | New | | | |
| II-833 | 1282 | 3.57 | Iron, 1.932 | Bräkken and Harang [1] 1928. |
| | 2-0378 | 2.76 | | |
| | 2-0368 | 2.50 | Cobalt | Döll and Klemm [2] 1939. |
| 1352 | 1259 | 3.57 | Molybdenum | Hanawalt, Rinn, and Frevel [3] 1938. |
| | 1-0540 | 3.87 | | |
| | 1-0536 | 2.77 | | |

The old and new ASTM cards of the Bräkken and Harang and of the Döll and Klemm composite pattern erroneously state that molybdenum radiation was used.

**Additional published patterns.** None.

**NBS pattern.** The sample of lead chloride used for the NBS pattern was furnished by the National Lead Co. Spectrographic analysis at the NBS showed less than 0.01 percent each of bismuth, and iron, and traces of silver, aluminum, calcium, copper, magnesium, and silicon.

**Interplanar spacings and intensity measurements.** Both the Bräkken and Harang and the Döll and Klemm *d*-spacings expressed in angstroms were calculated from their published Bragg angle data. The Hanawalt, Rinn, and Frevel *d*-spacings were converted from kX to angstrom units.

The three strongest lines for each of the patterns are as follows:

| Pattern | 1 | 2 | 3 |
|---|---|---|---|
| Bräkken and Harang | 111 | 012 | 121 |
| Döll and Klemm | 111 | 121 | 103 |
| Hanawalt, Rinn, and Frevel | 111 | 012 | 121 |
| Swanson and Fuyat | 111 | 012 | 121 |

**Lattice constants.** The structure was determined by Bräkken and Harang [1] in 1928. The space group is $D_{2h}^{16}$-Pmnb (Puma) with lead-chloride-structure type and 4($PbCl_2$) per unit cell. The Bräkken and Harang lattice constants were converted from iron radiation of 1.932 to 1.93597, the other three were converted from kX to angstrom units, and the Straumanis and Sauka values were temperature corrected to 26°C for comparison with the NBS values. The linear coefficient of expansion between 15 and 35°C is $33.5 \times 10^{-6}$ parallel to the $a$-axis, $38.8 \times 10^{-6}$ parallel to the $b$-axis, and $17.0 \times 10^{-6}$ parallel to the $c$-axis, according to Straumanis and Sauka [4].

*Lattice constants in angstroms*

| | | $a$ | $b$ | $c$ |
|---|---|---|---|---|
| 1928 | Bräkken and Harang [1] | 4.505 | 7.683 | 9.172 |
| 1932 | Bräkken [5] | 4.543 | 7.623 | 9.048 |
| 1939 | Döll and Klemm [2] | 4.529 | 7.620 | 9.045 |
| 1942 | Straumanis and Sauka [4] | 4.53410 | 7.62005 | 9.04499 at 26°C |
| 1953 | Swanson and Fuyat | 4.535 | 7.62 | 9.05 at 26°C |

**Lead chloride, $PbCl_2$**

| $hkl$ | 1928 Bräkken and Harang, Fe, 1.932 A | | 1939 Döll and Klemm, Co, 1.78890 A | | 1938 Hanawalt, Rinn, and Frevel, Mo, 0.7093 A | | 1953 Swanson and Fuyat, Cu, 1.5405 A, 26°C | |
|---|---|---|---|---|---|---|---|---|
| | $d$ | $I$ | $d$ | $I$ | $d$ | $I$ | $d$ | $I$ |
| | A | | A | | A | | A | |
| 002 | 4.50 | w | ------- | --- | ------- | --- | 4.521 | 18 |
| 101 | 4.04 | w | ------- | --- | 4.05 | 20 | 4.055 | 34 |
| 012 | 3.88 | s | 3.87 | m | 3.88 | 88 | 3.890 | 73 |
| 020 | 3.80 | m | ------- | --- | ------- | --- | 3.811 | 43 |
| 111 | 3.58 | vs | 3.56 | s | 3.58 | 100 | 3.581 | 100 |
| 112 | ------ | --- | ------- | --- | ------- | --- | 2.953 | 7 |
| 022, 120 | 2.91 | m | 2.90 | vw | 2.91 | 20 | 2.916 | 23 |
| 121 | 2.76 | s | 2.76 | s | 2.78 | 75 | 2.778 | 56 |
| 103 | 2.50 | s | 2.50 | s | 2.51 | 50 | 2.513 | 48 |
| 023 | 2.37 | w | 2.37 | vw | ------- | --- | 2.365 | 4 |
| 200, 004 | 2.26 | s | 2.26 | m | 2.26 | 30 | 2.268 | 23 |
| | | | | | | | 2.262 | 24 |
| 032 | 2.21 | m | 2.20 | m | 2.21 | 15 | 2.216 | 28 |
| 014 | ------ | -- | ------- | --- | ------- | --- | 2.165 | 10 |
| 131 | 2.15 | s | 2.15 | s | 2.15 | 50 | 2.150 | 32 |
| 123 | 2.09 | s | 2.09 | s | 2.08 | 50 | 2.097 | 38 |
| 202 | ------ | -- | ------- | --- | ------- | --- | 2.026 | 4 |
| 212, 114 | 1.986 | vw | ------- | --- | ------- | --- | 1.960 | 18 |
| 024, 220 | ----- | -- | ------- | --- | 1.94 | 45 | 1.945 | 13 |
| 040 | ------ | -- | ------- | --- | ------- | --- | 1.906 | 5 |
| 124, 222 | ----- | -- | ------- | --- | ------- | --- | 1.790 | 4 |
| 141 | ------ | -- | ------- | --- | ------- | --- | 1.725 | 2 |
| 034 | ------ | -- | ------- | --- | ------- | --- | 1.690 | 4 |
| 115 | ------ | -- | ------- | --- | 1.63 | 20 | 1.641 | 11 |
| 204 | ------ | -- | ------- | --- | ------- | --- | 1.601 | 7 |
| 232, 134 | ----- | -- | ------- | --- | 1.58 | 20 | 1.585 | 15 |
| 214 | ------ | -- | ------- | --- | ------- | --- | 1.567 | 3 |
| 143 | ------ | -- | ------- | --- | ------- | --- | 1.518 | 5 |
| 006 | ------ | -- | ------- | --- | 1.50 | 5 | 1.508 | 4 |
| 016 | ------ | -- | ------- | --- | ------- | --- | 1.479 | 3 |
| 224 | ------ | -- | 1.473 | vw | ------- | --- | 1.476 | 4 |
| 311 | ------ | -- | ------- | --- | 1.463 | 10 | 1.463 | 4 |
| 044, 240 | ----- | -- | 1.458 | w | ------- | --- | 1.458 | 7 |
| 052 | ------ | -- | ------- | --- | ------- | --- | 1.444 | 3 |
| 151 | ------ | -- | 1.424 | m | 1.428 | 5 | 1.427 | 7 |
| 320, 026, 135 | ----- | -- | 1.400 | s | 1.396 | 10 | 1.402 | 11 |
| 144, 215, 242 | ----- | -- | 1.386 | vw | ------- | --- | 1.388 | 4 |
| 234 | ------ | -- | 1.350 | m | 1.353 | 5 | 1.355 | 4 |
| 303 | ------ | -- | ------- | --- | ------- | --- | 1.351 | 6 |
| 331 | ------ | -- | ------- | --- | ------- | --- | 1.286 | 3 |
| 323 | ------ | -- | ------- | --- | 1.272 | 5 | 1.274 | 4 |
| 060 | ------ | -- | ------- | --- | ------- | --- | 1.271 | 2 |
| 054 | ------ | -- | ------- | --- | ------- | --- | 1.264 | 2 |
| 206 | ------ | -- | ------- | --- | ------- | --- | 1.255 | 3 |
| --- | ------ | -- | ------- | --- | ------- | --- | 1.243 | 2 |
| --- | ------ | -- | ------- | --- | 1.215 | 5 | ------ | --- |

The density of lead chloride calculated from the NBS lattice constant is 5.88 at 26°C. The sample was too finely divided to determine the refractive index.

### References


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## Lead bromide, $PbBr_2$ (orthorhombic)

### ASTM cards

| Card number | | New index lines | Radiation | Source |
|---|---|---|---|---|
| Old | New | | | |
| II-1556 | 2325 | 2.90 | Copper---- | Bräkken and Harang [1] 1928. |
| | 2-0735 | 2.62 | | |
| | 2-0754 | 3.73 | Molybdenum | Döll and Klemm [2] 1939. |
| 1542 | 1412 | 3.40 | Molybdenum | Hanawalt, Rinn and Frevel [3] 1938. |
| | 1-0610 | 4.18 | | |
| | 1-0623 | 2.39 | | |
| ------- | 2303 | 2.91 | Copper----- | S-V |
| | 3-0660 | 3.06 | | |
| | 3-0672 | 2.62 | | |
| ------- | 2304 | ----- | ---------- | A continuation of the preceding card. |
| | 3-0661 | | | |
| | 3-0673 | | | |

The Bräkken and Harang and the Döll and Klemm patterns were combined on one ATSM card. The S-V cards were apparently a personal communication and no explanation of the abbreviation can be found.

**Additional published patterns.** None.

**NBS patterns.** The lead bromide sample used for the NBS pattern was furnished by the National Lead Company. Spectrographic analysis at the NBS showed 0.001 to 0.01 percent of iron, 0.0001 to 0.001 percent each of silver, aluminum, copper, magnesium, and silicon, and less than 0.0001 percent of calcium.

**Interplanar spacings and intensity measurements.** The Bräkken and Harang and the Döll and Klemm *d*-spacings expressed in angstroms were calculated from their Bragg angle data while the Hanawalt, Rinn, and Frevel and the S-V *d*-spacings were converted from kX to angstrom units.

The three strongest lines for each of the patterns are as follows:

| Pattern | 1 | 2 | 3 |
|---|---|---|---|
| Bräkken and Harang---------- | 111 | 121 | 103 |
| Döll and Klemm---------- | 121 | 103 | 200 |
| Hanawalt, Rinn, and Frevel---- | 101 | 004 | 013 |
| S-V---------- | 121 | 120 | 103 |
| Swanson and Fuyat---------- | 121 | 111 | 103 |

The strongest line of the Hanawalt, Rinn, and Frevel pattern at 3.41 A is not possible according to the theoretical pattern for $PbBr_2$. This line could be due to $PbI_2$, which is usually used to prepare $PbBr_2$.

**Lattice constants.** The structure was determined by Bräkken and Harang [1] in 1928. The space group is $D_{2h}^{16}$-Pmnb (Puma) with lead-chloride-structure type, and 4($PbBr_2$) per unit cell.

Several unit-cell determinations have been converted to angstroms for comparison with the NBS values.

*Lattice constants in angstroms*

| | | *a* | *b* | *c* |
|---|---|---|---|---|
| 1928 | Bräkken and Harang [1]---------- | 4.716 | 8.005 | 9.494 |
| 1932 | Nieuwenkamp and Bijvoet [4]------- | 4.725 | 8.04 | 9.504 |
| 1939 | Döll and Klemm [2] | 4.727 | 8.054 | 9.537 |
| 1953 | Swanson and Fuyat--- | 4.732 | 8.060 | 9.553 at 26°C |

The density of lead bromide calculated from the NBS lattice constants is 6.690 at 26°C. The refractive indices of lead bromide are too high and the sample was too fine for an index determination by the usual liquid grain immersion methods.

## Lead bromide, $PbBr_2$

| hkl | 1928 Brakken and Harang Cr, 2.2896 A | | 1939 Döll and Klemm Co, 1.7889 A | | 1938 Hanawalt, Rinn, and Frevel Mo, 0.709 A | | ---- S-V Cu, 1.5405 A | | 1953 Swanson and Fuyat Cu, 1.5405 A, 26°C | |
|---|---|---|---|---|---|---|---|---|---|---|
| | d | I | d | I | d | I | d | I | d | I |
| | A | | A | | A | | A | | A | |
| 011 | ---------- | -------- | ---------- | -------- | 6.00 | 33 | ---------- | ------ | 6.16 | 1 |
| 002 | 4.75 | w | ---------- | -------- | ---------- | --------- | 4.73 | 30 | 4.771 | 29 |
| 101 | 4.22 | w | ---------- | -------- | 4.19 | 83 | 4.22 | 30 | 4.243 | 26 |
| 012 | 4.09 | m | 4.09 | m | ---------- | --------- | ---------- | ------ | 4.107 | 47 |
| 020 | 4.00 | w | ---------- | -------- | 3.93 | 50 | 4.05 | 40B | 4.031 | 35 |
| 111 | 3.73 | s | 3.74 | m | 3.73 | 33 | 3.75 | 80 | 3.754 | 79 |
| ---------- | ---------- | -------- | ---------- | -------- | 3.41 | 100 | ---------- | ------ | ---------- | ------- |
| 112 | ---------- | -------- | ---------- | -------- | ---------- | --------- | ---------- | ------ | 3.100 | 40 |
| 022 | 3.08 | m | 3.08 | m | ---------- | --------- | ---------- | ------ | 3.078 | 35 |
| 120 | 3.06 | m | ---------- | -------- | ---------- | --------- | 3.07 | 90 | 3.070 | 54 |
| 013 | 2.94 | vw | ---------- | -------- | 2.95 | 67 | ---------- | ------ | 2.960 | 11 |
| 121 | 2.91 | s | 2.91 | s | ---------- | --------- | 2.92 | 100 | 2.921 | 100 |
| 103 | 2.63 | s | 2.63 | s | 2.63 | 67 | 2.63 | 90 | 2.640 | 73 |
| 031 | 2.59 | vw | 2.58 | vw | ---------- | --------- | 2.59 | 20 | 2.586 | 5 |
| 122 | 2.57 | vw | ---------- | -------- | ---------- | --------- | ---------- | ------ | 2.582 | 5 |
| 113 | ---------- | -------- | ---------- | -------- | ---------- | --------- | 2.51 | 30 | 2.507 | 5 |
| 023 | 2.48 | m | 2.49 | w | ---------- | --------- | ---------- | ------ | 2.496 | 10 |
| 004 | ---------- | -------- | ---------- | -------- | 2.39 | 83 | ---------- | ------ | 2.385 | 17 |
| 200 | 2.37 | vw | 2.36 | s | ---------- | --------- | ---------- | ------ | 2.366 | 45 |
| 032 | 2.35 | w | 2.34 | m | ---------- | --------- | 2.35 | 60B | 2.341 | 35 |
| 014 | 2.28 | w | ---------- | -------- | 2.29 | 17 | ---------- | ------ | 2.288 | 8 |
| 131 | ---------- | -------- | 2.26 | s | ---------- | --------- | 2.26 | 60 | 2.270 | 35 |
| 211<br>123 | ---------- | -------- | 2.20 | s | 2.21 | 17 | 2.20 | 70 | 2.209 | 44 |
| 202 | ---------- | -------- | ---------- | -------- | 2.12 | 33 | 2.11 | 10B | 2.121 | 6 |
| 132 | ---------- | -------- | ---------- | -------- | ---------- | --------- | ---------- | ------ | 2.099 | 3 |
| 024<br>033<br>212 | ---------- | -------- | ---------- | -------- | 2.05 | 33 | 2.04 | 40 | 2.052 | 14 |
| 040 | ---------- | -------- | ---------- | -------- | ---------- | --------- | 2.01 | 10 | 2.016 | 7 |
| 041 | ---------- | -------- | ---------- | -------- | 1.94 | 33 | 1.97 | 5 | 1.972 | 4 |
| 124<br>133 | ---------- | -------- | ---------- | -------- | ---------- | --------- | 1.88 | 30 | 1.883 | 4 |
| 222 | ---------- | -------- | ---------- | -------- | ---------- | --------- | ---------- | ------ | 1.876 | 9 |
| 140 | ---------- | -------- | ---------- | -------- | 1.84 | 33 | 1.84 | 20 | 1.852 | 4 |
| 034 | ---------- | -------- | ---------- | -------- | ---------- | --------- | ---------- | ------ | 1.784 | 4 |
| 105 | ---------- | -------- | ---------- | -------- | 1.76 | 33 | 1.77 | 30B | 1.770 | 10 |
| 115<br>142 | ---------- | -------- | ---------- | -------- | ---------- | --------- | 1.73 | 40 | 1.728 | 9 |
| 223 | ---------- | -------- | ---------- | -------- | ---------- | --------- | ---------- | ------ | 1.718 | 6 |
| 043 | ---------- | -------- | ---------- | -------- | ---------- | --------- | ---------- | ------ | 1.700 | 2 |
| 204 | ---------- | -------- | ---------- | -------- | 1.69 | 33 | 1.68 | 10 | 1.680 | 6 |
| 232 | ---------- | -------- | ---------- | -------- | ---------- | --------- | 1.66 | 50 | 1.665 | 17 |
| 214 | ---------- | -------- | ---------- | -------- | ---------- | --------- | 1.64 | 10 | 1.645 | 3 |
| 143 | ---------- | -------- | ---------- | -------- | 1.62 | 33 | 1.59 | 20B | 1.602 | 2 |
| 016 | ---------- | -------- | ---------- | -------- | 1.58 | 33 | ---------- | ------ | 1.565 | 4 |

## Lead bromide, $PbBr_2$—Con.

| hkl | 1928 Bräkken and Harang Cr, 2.2896 A | | 1939 Döll and Klemm Co, 1.7889 A | | 1938 Hanawalt, Rinn, and Frevel Mo, 0.709 A | | ---- S-V Cu, 1.5405 A | | 1953 Swanson and Fuyat Cu, 1.5405 A, 26°C | |
|---|---|---|---|---|---|---|---|---|---|---|
| | d | I | d | I | d | I | d | I | d | I |
| | A | | A | | A | | A | | A | |
| 224 | | | | | | | | | | |
| 233 | ---------- | ---------- | ---------- | ---------- | 1.56 | 17 | 1.55 | 10 | 1.550 | 4 |
| 044 | ---------- | ---------- | ---------- | ---------- | ---------- | ---------- | ---------- | ------ | 1.539 | 3 |
| 240 | ---------- | ---------- | ---------- | ---------- | ---------- | ---------- | ---------- | ------ | 1.535 | 5 |
| 052 | | | | | | | | | | |
| 311 | ---------- | ---------- | ---------- | ---------- | ---------- | ---------- | 1.53 | 20 | 1.528 | 5 |
| 151 | ---------- | ---------- | ---------- | ---------- | 1.51 | 17 | 1.51 | 30 | 1.507 | 12 |
| 026 | | | | | | | | | | |
| 135 | ---------- | ---------- | ---------- | ---------- | ---------- | ---------- | 1.48 | 60 | 1.478 | 15 |
| 321 | ---------- | ---------- | ---------- | ---------- | ---------- | ---------- | 1.45 | 20 | 1.452 | 5 |
| 234 | ---------- | ---------- | ---------- | ---------- | ---------- | ---------- | 1.43 | 10 | 1.425 | 3 |
| 303 | | | | | | | | | | |
| 126 | ---------- | ---------- | ---------- | ---------- | ---------- | ---------- | 1.41 | 20 | 1.413 | 6 |
| 331 | ---------- | ---------- | ---------- | ---------- | ---------- | ---------- | 1.35 | 20 | 1.347 | 3 |
| 323 | ---------- | ---------- | ---------- | ---------- | ---------- | ---------- | 1.34 | 40 | 1.334 | 5 |
| 206 | | | | | | | | | | |
| 251 | ---------- | ---------- | ---------- | ---------- | ---------- | ---------- | 1.32 | 20 | 1.319 | 3 |
| 107 | | | | | | | | | | |
| 332 | ---------- | ---------- | ---------- | ---------- | ---------- | ---------- | 1.31 | 20 | 1.309 | 2 |
| 062 | | | | | | | | | | |
| 117 | ---------- | ---------- | ---------- | ---------- | ---------- | ---------- | 1.29 | 30B | 1.294 | 4 |
| 160 | | | | | | | | | | |
| 226 | ---------- | ---------- | ---------- | ---------- | ---------- | ---------- | 1.26 | 30 | 1.254 | 5 |
| 146 | ---------- | ---------- | ---------- | ---------- | ---------- | ---------- | 1.21 | 5 | ---------- | ------- |
| 342 | ---------- | ---------- | ---------- | ---------- | ---------- | ---------- | 1.19 | 10 | ---------- | ------- |
| ---------- | ---------- | ---------- | ---------- | ---------- | ---------- | ---------- | [a] 1.18 | 20 | ---------- | ------- |

[a] 28 lines between 1.16 and 0.795 have been omitted.

### References


[1] H. Bräkken and L. Harang, Zur Kristallstruktur einiger rhombischer Verbindungen $MX_2$; I., Z. Krist. **68**, 123-38 (1928).

[2] W. Döll and W. Klemm, Messungen an zwei-und vierwertigen Verbindungen der selten Erden; VII, Über die Struktur einiger Dihalogenide, Z. anorg. allg. Chem. **241**, 239-58 (1939).

[3] J. D. Hanawalt, H. W. Rinn and L. K. Frevel, Chemical analysis by X-ray diffraction, Ind. and Eng. Chem., Anal. Ed. **10**, 457-512 (1938).

[4] W. Nieuwenkamp and J. M. Bijvoet, Die Kristallstruktur von Bleibromide, $PbBr_2$, Z. Krist. **84**, 49-61 (1932).


## Ammonium bromide ($NH_4Br$) (cubic)

### ASTM cards

| Card number | | New index lines | Radiation | Source |
|---|---|---|---|---|
| Old | New | | | |
| 2389 | 2557 | 2.83 | Molybdenum, 0.712. | Bartlett and Langmuir [1] 1921. |
| | 1-0998 | 1.79 | | |
| | 1-0986 | 1.64 | | |
| 2331 | 2396 | 2.86 | Molybdenum-- | Havighurst, Mack, and Blake [2] 1924. |
| | 1-0946 | 4.05 | | |
| | 1-0958 | 1.65 | | |
| 2372 | 2474 | 2.85 | Molybdenum, 0.70783. | Hanawalt, Rinn, and Frevel [3] 1938. |
| | 1-0973 | 1.65 | | |
| | 1-0974 | 1.81 | | |

**Additional published patterns.** None.

**NBS pattern.** The ammonium bromide sample used for the NBS pattern was prepared by R. B. Johannesen of the NBS from distilled constant-boiling hydrobromic acid and distilled ammonium hydroxide. Chemical analysis at the Bureau showed the following impurities:

| | |
|---|---|
| Iron | 0.0001% |
| Bromate | Passes test (limit about 0.001%). |
| Chloride | 0.003% |
| Nonvolatile matter | 0.002% |

**Interplanar spacings and intensity measurements.** The Havighurst, Mack, and Blake *d*-spacings expressed in angstroms were calculated from their Bragg angle data. The Bartlett and Langmuir *d*-values were converted from molybdenum radiation, $\lambda = 0.712$, to angstroms, and the Hanawalt, Rinn, and Frevel *d*-spacings were converted from kX to angstrom units.

The three strongest lines for each pattern are as follows:

| Pattern | 1 | 2 | 3 |
|---|---|---|---|
| Bartlett and Langmuir | 110 | 210 | 211 |
| Havighurst, Mack, and Blake | 110 | 100 | 211 |
| Hanawalt, Rinn, and Frevel | 110 | 321 | 211 |
| Swanson and Fuyat | 110 | 100 | 211 |

**Lattice constants.** The structure was determined by Havighurst, Mack, and Blake [2] in 1924. The simple cubic cell has space group $O_h^1$-Pm3m, cesium-chloride-structure type and $1(NH_4Br)$ per unit cell. It is generally accepted that this material is stable at room temperature. The face-centered NaCl type exists above 250°C, and a tetragonal phase, below -73°C.

**Ammonium bromide, $NH_4Br$**

| *hkl* | 1921 Bartlett and Langmuir Mo, 0.712 A | | | 1924 Havighurst, Mack, and Blake Mo, 0.709 A | | | 1938 Hanawalt, Rinn, and Frevel Mo, 0.709 A | | | 1953 Swanson and Fuyat Cu, 1.5405 A, 26°C | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | *d* | *I* | *a* | *d* | *I* | *a* | *d* | *I* | *a* | *d* | *I* | *a* |
| | A | | A | A | | A | A | | A | A | | A |
| 100 | --- | --- | --- | 4.045 | 40 | 4.045 | 4.08 | 17 | 4.08 | 4.06 | 68 | 4.06 |
| 110 | 2.821 | 100 | 3.989 | 2.861 | 100 | 3.982 | 2.86 | 100 | 4.045 | 2.871 | 100 | 4.061 |
| 111 | 2.294 | 20 | 3.973 | 2.331 | 20 | 4.037 | 2.33 | 10 | 4.037 | 2.345 | 18 | 4.062 |
| 200 | 1.983 | 20 | 3.966 | 2.020 | 20 | 4.040 | 2.02 | 10 | 4.040 | 2.031 | 17 | 4.062 |
| 210 | 1.778 | 40 | 3.977 | 1.808 | 35 | 4.043 | 1.81 | 23 | 4.047 | 1.816 | 21 | 4.061 |
| 211 | 1.633 | 40 | 4.000 | 1.652 | 40 | 4.047 | 1.65 | 42 | 4.042 | 1.658 | 25 | 4.061 |
| 220 | 1.408 | 20 | 3.982 | 1.432 | 15 | 4.050 | 1.432 | 10 | 4.050 | 1.436 | 6 | 4.062 |
| 300 | 1.322 | 20 | 3.966 | 1.346 | 10 | 4.038 | 1.353 | 13 | 4.059 | 1.354 | 8 | 4.062 |
| 310 | 1.256 | 20 | 3.972 | 1.278 | 10 | 4.041 | 1.283 | 10 | 4.057 | 1.284 | 7 | 4.060 |
| 311 | 1.186 | 15 | 3.934 | 1.216 | 8 | 4.033 | 1.222 | 3 | 4.053 | 1.2242 | 4 | 4.0602 |
| 222 | 1.160 | 10 | 4.018 | 1.167 | 5 | 4.043 | 1.171 | 3 | 4.056 | 1.1722 | 2 | 4.0606 |
| 320 | 1.101 | 10 | 3.970 | 1.119 | 5 | 4.035 | --- | --- | --- | 1.1258 | 3 | 4.0591 |
| 321 | 1.055 | 15 | 3.947 | 1.082 | 20 | 4.048 | 1.083 | 67 | 4.052 | 1.0851 | 4 | 4.0601 |
| 400 | --- | --- | --- | --- | --- | --- | --- | --- | --- | 1.0151 | <1 | 4.0604 |
| 410 | --- | --- | --- | 0.981 | 2 | 4.045 | --- | --- | --- | 0.9846 | 2 | 4.0596 |
| 411 | --- | --- | --- | .953 | 3 | 4.043 | 0.957 | 3 | 4.060 | .9568 | 1 | 4.0594 |
| 331 | --- | --- | --- | --- | --- | --- | --- | --- | --- | .9314 | 1 | 4.0599 |
| 420 | --- | --- | --- | --- | --- | --- | --- | --- | --- | .9077 | 1 | 4.0594 |
| 421 | --- | --- | --- | --- | --- | --- | --- | --- | --- | .8859 | 2 | 4.0597 |
| 332 | --- | --- | --- | --- | --- | --- | --- | --- | --- | .8655 | 1 | 4.0596 |
| 422 | --- | --- | --- | --- | --- | --- | --- | --- | --- | .8286 | 1 | 4.0593 |
| 500 | --- | --- | --- | --- | --- | --- | --- | --- | --- | .8118 | 1 | 4.0590 |
| 521 | --- | --- | --- | 0.738 | 2 | 4.042 | --- | --- | --- | --- | --- | --- |
| Average value of the last five lines | | | 3.968 | --- | --- | 4.043 | --- | --- | 4.056 | --- | --- | 4.0594 |

Several reported unit-cell determinations have been converted to angstroms for comparison with the NBS value.

*Lattice constant in angstroms*

| | | $a$ |
|---|---|---|
| 1921 | Bartlett and Langmuir [1]------- | 3.996 at 26°C |
| 1921 | Vegard [4]---------- | 4.078 |
| 1924 | Havighurst, Mack, and Blake [2]-- | 4.055 |
| 1926 | Erdal [5]---------- | 4.066 |
| 1953 | Swanson and Fuyat---------- | 4.0594 at 26°C |

The density of ammonium bromide calculated from the NBS lattice constant is 2.431 at 26°C. The refractive index of the NBS sample is 1.710.

**References**


[1] G. Bartlett and I. Langmuir, Crystal structures of the ammonium halides above and below the transition temperatures, J. Am. Chem. Soc. **43**, 84-91 (1921).

[2] R. J. Havighurst, E. Mack, Jr., and F. C. Blake, Precision crystal measurements on some alkali and ammonium halides, J. Am. Chem. Soc. **46**, 2368-74 (1924).

[3] J. D. Hanawalt, H. W. Rinn, and L. K. Frevel, Chemical analysis by X-ray diffraction, Ind. Eng. Chem., Anal. Ed. **10**, 457-512 (1938).

[4] L. Vegard, The constitution of mixed crystals and the space occupied by atoms, Vid. Selsk, Skrifter I. Mat. Nat. Kl. 1921.

[5] A. Erdal, Contributions to the analysis of mixed crystals and alloys, Z. Krist. **65**, 69-82 (1926).


## 2.5. Carbonates

**Calcium carbonate (calcite), $CaCO_3$ (hexagonal)**

**ASTM cards**

| Card number | | New index lines | Radiation | Source |
|---|---|---|---|---|
| Old | New | | | |
| ------- | 2048 | 3.04 | Copper, | Olshausen [1] 1925. |
| | 3-0594 | 4.29 | 1.53923. | |
| | 3-0593 | 1.91 | | |
| II-1338 | 2123 | 3.03 | Molybdenum, | Harrington [2] 1927. |
| | 2-0650 | 1.92 | 0.710. | |
| | 2-0623 | 1.87 | | |
| ------- | 2018 | 3.09 | ---------- | Krieger [3] 1930. |
| | 3-0585 | 1.94 | | |
| | 3-0569 | 1.88 | | |
| II-1349 | 2122 | 3.02 | Copper------ | Nagelschmidt [4] |
| | 2-0649 | 1.92 | | 1934, Boldyrev [5] |
| | 2-0629 | 1.87 | | 1938. |

**ASTM cards—Con.**

| Card number | | New index lines | Radiation | Source |
|---|---|---|---|---|
| Old | New | | | |
| 2035 | 2121 | 3.04 | Molybdenum-- | Hanawalt, Rinn, and |
| | 1-0863 | 1.92 | | Frevel [6] 1938. |
| | 1-0837 | 2.28 | | |
| ------- | 2128 | 3.03 | Iron-------- | Allis-Chalmers Mfg. |
| | 3-0613 | 1.88 | | Co. |
| | 3-0596 | 3.34 | | |
| ------- | 2124 | 3.01 | Copper------ | British Museum. |
| | 3-0611 | 1.91 | | |
| | 3-0612 | 1.86 | | |
| The following ASTM card, labeled calcite, appears to be aragonite. | | | | |
| ------- | 2357 | 2.92 | Copper------ | Norton, 1937. |
| | 3-0680 | 1.86 | | |
| | 3-0670 | 1.85 | | |

The ASTM pattern, old card No. II-1349 and new card No. 2-0629, is a combination of Nagelschmidt's pattern and that of Boldyrev. The latter is a combination of the same Nagelschmidt pattern and the Krieger pattern, which is listed independently on card No. 3-0569.

**Additional published patterns**

| Source | Radiation | Wavelength |
|---|---|---|
| Mizgier [7] 1929---------- | Copper---- | ---------- |

**NBS pattern.** The calcium carbonate used for the NBS pattern is a specially purified sample contributed by the Mallinckrodt Chemical Works. Their spectrographic analysis shows 0.01 to 0.1 percent of strontium, 0.001 to 0.01 percent of barium, 0.0001 to 0.001 percent each of aluminum, boron, cesium, copper, potassium, magnesium, sodium, silicon, and tin, and less than 0.0001 percent each of silver, chromium, iron, lithium, and manganese.

**Interplanar spacings and intensity measurements.** The Olshausen and Mizgier $d$-spacings expressed in angstroms were calculated from their published Bragg angle data. $D$-spacings for all of the other patterns were converted from kX to angstrom units.

The three strongest lines for each of the patterns are as follows:

| Pattern | 1 | 2 | 3 |
|---|---|---|---|
| Olshausen | 104 | 204, 108 | 102 |
| Harrington | 104 | 204, 108 | 116 |
| Krieger | 104 | 204, 108 | 116 |
| Nagelschmidt | 104 | 204, 108 | 116 |
| Hanawalt, Rinn, and Frevel | 104 | 204, 108 | 113 |
| Allis Chalmers | 104 | 116 | 113 |
| British Museum | 104 | 204, 108 | 116 |
| Swanson and Fuyat | 104 | 113 | 202 |

The $d$-spacing at 4.29 A of Olshausen's pattern is a possible vaterite line and the $d$-spacing at 3.35 A of the Allis-Chalmers and British Museum patterns are possibly due to aragonite. These have not been included among the three strongest lines as they do not fit the theoretical indexing for calcite. The Krieger ASTM card $d$-spacing at 3.09 A should be at 3.08 A according to his published data.

**Lattice constants.** The structure was determined by W. L. Bragg [8] in 1914. The space group is $D_{3d}^6$-$R\bar{3}c$ with sodium-nitrate-structure type and $6(CaCO_3)$ per unit cell. Other polymorphic forms of calcium carbonate are aragonite (orthorhombic) and vaterite (hexagonal).

The unit cell values expressed by Bergen in terms of the hexagonal unit cell were compatible with the established values when the $a$ direction was divided by two and the $c$ direction multiplied by two. The other values, given in terms of the rhombohedral cell, were converted to hexagonal axes, and all were converted from kX to angstrom units for comparison with the NBS values.

**Calcium carbonate (calcite), $CaCO_3$**

| $hkl$ | 1925 Olshausen Cu, 1.5405 A | | 1927 Harrington Mo, 0.7093 A | | 1930 Krieger --- | | 1934 Nagelschmidt Mo, 0.7093 A | | 1938 Hanawalt, Rinn, and Frevel Mo, 0.7093 A | | ---- Allis-Chalmers Fe, 1.93597 A | | ---- British Museum Cu, 1.5405 A | | 1929 Mizgier Cu, 1.5405 A | | 1953 Swanson and Fuyat Cu, 1.5405 A, 26°C | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | $d$ | $I$ | $d$ | $I$ | $d$ | $I$ | $d$ | $I$ | $d$ | $I$ | $d$ | $I$ | $d$ | $I$ | $d$ | $I$ | $d$ | $I$ |
| | A | | A | | A | | A | | A | | A | | A | | A | | A | |
| ------ | [a]4.29 | s | ----- | ---- | ----- | -- | ----- | --- | ----- | ---- | ------- | --- | ------ | --- | ------- | -- | ------ | --- |
| 102 | 3.86 | m | ----- | ---- | ----- | -- | 3.85 | m | 3.87 | 8 | 3.84 | 30 | 3.85 | 60 | 3.67 | -- | 3.86 | 12 |
| ------ | ------ | --- | ----- | ---- | ----- | -- | ----- | --- | ----- | ---- | [b]3.35 | 60 | [b]3.35 | 70 | ------- | -- | ------ | --- |
| ------ | ------ | --- | ----- | ---- | ----- | -- | ----- | --- | ----- | ---- | ------- | --- | ------ | --- | [b]3.29 | -- | ------ | --- |
| 014 | 3.03 | vs | 3.03 | 100 | 3.08 | 100 | 3.03 | vs | 3.05 | 100 | 3.04 | 100 | 3.02 | 100 | 2.98 | -- | 3.035 | 100 |
| 006 | ------ | --- | ----- | ---- | ----- | -- | ----- | --- | ----- | ---- | 2.86 | 10 | ------ | --- | ------- | -- | 2.845 | 3 |
| 110 | 2.49 | m | 2.49 | 40 | 2.53 | 30 | 2.50 | s | 2.50 | 20 | 2.50 | 50 | 2.51 | 60 | 2.44 | -- | 2.495 | 14 |
| 113 | 2.30 | m | 2.28 | 60 | 2.31 | 50 | 2.28 | s | 2.28 | 24 | 2.29 | 60 | 2.28 | 70 | 2.25 | -- | 2.285 | 18 |
| 022 | 2.10 | m | 2.10 | 60 | 2.11 | 50 | 2.09 | s | 2.09 | 20 | 2.10 | 50 | 2.07 | 70 | 2.07 | -- | 2.095 | 18 |
| 204 | 1.90 | sd | 1.918 | 80 | 1.94 | 80 | 1.918 | vs | 1.92 | 32 | 1.93 | 10 | 1.91 | 80 | ------ | -- | 1.927 | 5 |
| 108 | | | | | | | | | | | 1.92 | 60 | | | ------ | -- | 1.913 | 17 |
| 116 | ------ | --- | 1.868 | 70 | 1.88 | 70 | 1.870 | vs | 1.87 | 24 | 1.88 | 70 | 1.86 | 80 | 1.88 | -- | 1.875 | 17 |
| ------ | ------ | --- | ----- | ---- | ----- | -- | ----- | --- | ----- | ---- | ------- | --- | [b]1.77 | 40 | [b]1.77 | -- | ------ | --- |
| ------ | ------ | --- | ----- | ---- | ----- | -- | ----- | --- | ----- | ---- | ------- | --- | 1.68 | 20 | 1.67 | -- | ------ | --- |
| 121 | ------ | --- | ----- | ---- | 1.62 | 30 | ----- | w | ----- | ---- | 1.63 | 10 | 1.63 | 40 | ------- | -- | 1.626 | 4 |
| 212 | 1.61 | m | 1.600 | 50 | ----- | -- | 1.598 | m | 1.60 | 16 | 1.61 | 30 | 1.59 | 60 | 1.60 | -- | 1.604 | 8 |
| 0·1·10 | ------ | --- | ----- | ---- | ----- | -- | ----- | --- | ----- | ---- | 1.59 | 10 | ------ | --- | ------- | -- | 1.587 | 2 |

[a] A possible vaterite line.
[b] A possible aragonite line.

## Calcium carbonate (calcite), $CaCO_3$—Con.

| *hkl* | 1925 Olshausen Cu, 1.5405 A | | 1927 Harrington Mo, 0.7093 A | | 1930 Krieger --------- | | 1934 Nagel-schmidt Mo, 0.7093 A | | 1938 Hanawalt, Rinn, and Frevel Mo, 0.7093 A | | ---- Allis-Chalmers Fe, 1.93597 A | | ---- British Museum Cu, 1.5405 A | | 1929 Mizgier Cu, 1.5405 A | | 1953 Swanson and Fuyat Cu, 1.5405 A, 26°C | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | *d* | *I* | *d* | *I* | *d* | *I* | *d* | *I* | *d* | *I* | *d* | *I* | *d* | *I* | *d* | *I* | *d* | *I* |
| | *A* | | *A* | | *A* | | *A* | | *A* | | *A* | | *A* | | *A* | | *A* | |
| 124 | ------ | --- | 1.518 | 50 | 1.53 | 40 | 1.518 | s | ----- | ---- | 1.53 | 10 | ------ | --- | ------- | -- | 1.525 | 5 |
| 028 | 1.52 | m | ----- | ---- | ----- | -- | ----- | --- | 1.51 | 12 | 1.51 | 10 | 1.51 | 60 | 1.51 | -- | 1.518 | 4 |
| 119 | 1.47 | vw | ----- | ---- | ----- | -- | ----- | w | 1.478 | 5 | 1.48 | 10 | 1.47 | 20 | ------- | -- | 1.510 | 3 |
| 215 | ------ | --- | ----- | ---- | ----- | -- | ----- | --- | ----- | ---- | ------- | --- | ------ | --- | ------- | -- | 1.473 | 2 |
| 300 | ------ | --- | 1.436 | 40 | 1.45 | 30 | 1.440 | m | 1.442 | 8 | 1.44 | 20 | 1.43 | 40 | ------- | -- | 1.440 | 5 |
| 0·0·12 | 1.43 | m | ----- | ---- | ----- | -- | ----- | w | 1.428 | 5 | 1.43 | 10 | 1.41 | 20 | 1.43 | -- | 1.422 | 3 |
| ------ | ------ | --- | ----- | ---- | ----- | -- | ----- | --- | ----- | ---- | [b] 1.36 | 10 | [b] 1.36 | 20 | ------- | -- | ------ | --- |
| 217 | 1.35 | vw | 1.354 | 10 | 1.35 | 10 | ----- | vw | 1.353 | 3 | ------- | --- | ------ | --- | ------- | -- | 1.356 | 1 |
| 2·0·10 | ------ | --- | ----- | ---- | ----- | -- | ----- | w | ----- | ---- | 1.34 | 10 | 1.34 | 20 | 1.34 | -- | 1.339 | 2 |
| 218 | 1.29 | w | 1.294 | 10 | 1.31 | 20 | 1.296 | m | 1.298 | 5 | 1.30 | 10 | 1.30 | 40 | 1.29 | -- | 1.297 | 2 |
| 306 | ------ | --- | ----- | ---- | ----- | -- | ----- | --- | ----- | ---- | 1.26 | 10 | ------ | --- | ------- | -- | 1.284 | 1 |
| 220 | ------ | --- | ----- | ---- | 1.25 | 20 | ----- | --- | 1.246 | 3 | 1.25 | 10 | 1.24 | 40 | ------- | -- | 1.247 | 1 |
| 1·1·12 | 1.24 | w | 1.234 | 20 | ----- | -- | ----- | --- | ----- | ---- | 1.24 | 10 | 1.23 | 40 | 1.24 | -- | 1.235 | 2 |
| 1·2·10 | 1.18 | m | 1.177 | 20 | 1.19 | 20 | 1.178 | m | 1.181 | 3 | 1.18 | 10 | 1.18 | 40 | 1.179 | -- | 1.1795 | 3 |
| 314 | 1.15 | m | 1.148 | 30 | 1.16 | 30 | ----- | m | 1.152 | 5 | 1.16 | 30 | 1.15 | 60 | 1.151 | -- | 1.1538 | 3 |
| 226 | ------ | --- | ----- | ---- | ----- | -- | 1.141 | w | ----- | ---- | 1.14 | 20 | 1.14 | 20 | 1.124 | -- | 1.1425 | 1 |
| 2·1·11 | 1.12 | vw | ----- | ---- | ----- | -- | ----- | --- | ----- | ---- | ------- | --- | ------ | --- | ------- | -- | 1.1244 | <1 |
| 0·2·14 | 1.06 | vw | ----- | ---- | ----- | -- | ----- | --- | ----- | ---- | ------- | --- | ------ | --- | 1.065 | -- | 1.0613 | 1 |
| 044 | 1.05 | m | ----- | ---- | 1.05 | 50 | ----- | m | 1.047 | 6 | 1.05 | 40 | ------ | --- | ------- | -- | 1.0473 | 3 |
| 138 | 1.04 | vw | 1.043 | 30 | ----- | -- | ----- | --- | ----- | ---- | 1.04 | 10 | 1.04 | 70 | 1.044 | -- | 1.0447 | 4 |
| 0·1·16 | ----- | --- | ----- | ---- | ----- | -- | ----- | --- | ----- | ---- | ------- | --- | ------ | --- | ------- | -- | 1.0352 | 2 |
| 1·1·15 | | | | | | | | | | | | | | | | | | |
| 1·2·13 | ------ | --- | ----- | ---- | 1.02 | 30 | ----- | --- | ----- | ---- | ------- | --- | ------ | --- | 1.017 | -- | 1.0234 | <1 |
| 3·0·12 | 1.01 | w | 1.013 | 20 | ----- | -- | ----- | m | 1.013 | 3 | 1.01 | 10 | 1.01 | 60 | ------- | -- | 1.0118 | 2 |
| 231 | ------ | --- | ----- | ---- | 0.990 | 20 | ----- | --- | ----- | ---- | ------- | --- | ------ | --- | 0.988 | -- | 0.9895 | <1 |
| 322 | 0.985 | wd | 0.983 | 20 | ----- | -- | ----- | --- | ----- | ---- | ------- | --- | ------ | --- | ------- | -- | .9846 | 1 |
| 1·0·17 | ------ | --- | ----- | ---- | ----- | -- | ----- | --- | ----- | ---- | ------- | --- | ------ | --- | ------- | -- | 0.9782 | 1 |
| 2·1·14 | ------ | --- | ----- | ---- | 0.972 | 20 | ----- | --- | ----- | ---- | ------- | --- | ------ | --- | ------- | -- | .9767 | 3 |
| 234 | 0.965 | w | 0.964 | 10 | ----- | -- | 0.965 | m | ----- | ---- | ------- | --- | ------ | --- | 0.968 | -- | .9655 | 2 |
| 408 | ------ | --- | ----- | ---- | ----- | -- | ----- | --- | ----- | ---- | ------- | --- | ------ | --- | ------- | -- | .9636 | 4 |
| 2·0·16 | ------ | --- | ----- | ---- | ----- | -- | ----- | --- | ----- | ---- | ------- | --- | ------ | --- | ------- | -- | .9562 | <1 |
| 410 | 0.942 | m | 0.942 | 10 | 0.947 | 10 | ----- | --- | ----- | ---- | ------- | --- | ------ | --- | 0.946 | -- | .9429 | 2 |
| 2·2·12 | ------ | --- | ----- | ---- | ----- | -- | ----- | --- | ----- | ---- | ------- | --- | ------ | --- | ------- | -- | .9376 | 2 |
| ------ | 0.924 | vw | ----- | ---- | ----- | -- | ----- | --- | ----- | ---- | ------- | --- | ------ | --- | ------- | -- | ------ | --- |
| ------ | .899 | m | ----- | ---- | 0.897 | 5 | ----- | --- | ----- | ---- | ------- | --- | ------ | --- | ------- | -- | ------ | --- |
| ------ | .888 | m | ----- | ---- | ----- | -- | ----- | --- | ----- | ---- | ------- | --- | ------ | --- | ------- | -- | ------ | --- |
| ------ | .876 | w | ----- | ---- | ----- | -- | ----- | --- | ----- | ---- | ------- | --- | ------ | --- | ------- | -- | ------ | --- |
| ------ | .862 | m | ----- | ---- | ----- | -- | ----- | --- | ----- | ---- | ------- | --- | ------ | --- | ------- | -- | ------ | --- |
| ------ | .852 | w | ----- | ---- | 0.856 | 5 | ----- | --- | ----- | ---- | ------- | --- | ------ | --- | ------- | -- | ------ | --- |
| ------ | .842 | m | ----- | ---- | ----- | -- | ----- | --- | ----- | ---- | ------- | --- | ------ | --- | ------- | -- | ------ | --- |
| ------ | ------ | --- | ----- | ---- | 0.838 | 3 | ----- | --- | ----- | ---- | ------- | --- | ------ | --- | ------- | -- | ------ | --- |
| ------ | ------ | --- | ----- | ---- | .801 | 3 | ----- | --- | ----- | ---- | ------- | --- | ------ | --- | ------- | -- | ------ | --- |
| ------ | ------ | --- | ----- | ---- | .792 | 3 | ----- | --- | ----- | ---- | ------- | --- | ------ | --- | ------- | -- | ------ | --- |

[b] A possible aragonite line.

*Lattice constants in angstroms*

| | | *a* | *c* |
|---|---|---|---|
| 1925 | Olshausen [1] | 4.989 | 17.078 |
| 1927 | Harrington [2] | 4.993 | 17.061 |
| 1941 | Bergen [9] | 5.00011 | 17.09498 |
| 1942 | Huber and Wagener [10] | 4.982 | 17.061 |
| 1947 | Vegard [11] | 4.968 | 16.975 |
| 1953 | Swanson and Fuyat | 4.989 | 17.062 at 26°C |

The density of calcium carbonate calculated from the NBS lattice constant is 2.711 at 26°C. The refractive indices of the sample are $\epsilon = 1.487$ and $\omega = 1.659$ with negative optical sign. The linear coefficient of expansion from 18 to 100°C is $2.10 \times 10^{-5}$ parallel to the *c* axis and $-0.380 \times 10^{-5}$ perpendicular to it, according to Weigle and Saihi [12].

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## Barium carbonate (witherite), $BaCO_3$ (orthorhombic)

### ASTM cards

| Card number | | New index lines | Radiation | Source |
|---|---|---|---|---|
| Old | New | | | |
| 1230 | 1145 | 3.72 | Molybdenum | Hanawalt, Rinn, and Frevel [1] 1938. |
| | 1-0500 | 2.63 | | |
| | 1-0506 | 2.14 | | |
| The following British Museum pattern labeled "$BaCO_3$, Witherite, Alston Moor" must be mislabeled, as it is in no way similar to the Hanawalt and NBS patterns. | | | | |
| II-825 | 1292 | 3.58 | Copper | British Museum. |
| | 2-0381 | 2.56 | | |
| | 2-0364 | 1.91 | | |

The optical properties of the NBS sample and typical witherite from the Alston Moor locality are identical with the generally accepted values. Attempts to identify the pattern have proved futile.

### Additional published patterns

| Source | Radiation | Wavelength |
|---|---|---|
| Zachariasen [2] 1928 | Copper | 1.539 |
| Lander [3] 1949 | Copper | |

**NBS pattern.** The barium carbonate used for the NBS pattern was a specially purified sample prepared by the Mallinckrodt Chemical Works. Spectrographic analysis at the Bureau showed 0.001 to 0.01 percent each of aluminum, calcium, sodium, and strontium, and less than 0.001 percent each of copper, iron, magnesium, and lead.

**Interplanar spacings and intensity measurements.** The Hanawalt, Rinn, and Frevel *d*-spacings were converted from kX to angstrom units, and those from the Zachariasen pattern were calculated from his Bragg angle data. The Lander *d*-spacings were published in angstroms. The three strongest lines for each of the patterns are as follows:

| Pattern | 1 | 2 | 3 |
|---|---|---|---|
| Hanawalt | 111, 021 | 200, 112 | 221 |
| Zachariasen | 111, 021 | 130 | 221 |
| Lander | 111 | 221 | 113 |
| Swanson and Fuyat | 111 | 021 | 221 |

**Lattice constants.** The structure was determined by Wilson [4] in 1928. The primitive orthorhombic lattice is pseudo-hexagonal with space group $D_{2h}^{16}$-Pmcn (Pnma), potassium-nitrate-structure type, and 4($BaCO_3$) per unit cell. It is generally accepted that barium carbonate inverts to a rhombohedral form above 800°C and to a cubic form above 980°C.

Several unit-cell measurements have been converted to angstroms for comparison with the NBS values.

*Lattice constants in angstroms*

| | | *a* | *b* | *c* |
|---|---|---|---|---|
| 1928 | Zachariasen [2] | 5.30 | 8.90 | 6.42 |
| 1928 | Wilson [4] | 5.263 | 8.846 | 6.557 |
| 1935 | Colby and LaCoste [5] | 5.29 | 8.84 | 6.40 |
| 1942 | Huber and Wagener [6] | 5.27 | 8.82 | 6.44 |
| 1949 | Lander [3] | 5.30 | 8.88 | 6.42 |
| 1953 | Swanson and Fuyat | 5.314 | 8.904 | 6.430 at 26°C |

The density of barium carbonate calculated from the NBS lattice constants is 4.308 at 26°C. The NBS sample is optically negative with refractive indices of $\alpha = 1.530$, $\beta = 1.679$, and $\gamma = 1.680$.

**Barium carbonate (witherite), $BaCO_3$**

| *hkl* | 1938 Hanawalt, Rinn, and Frevel, Mo, 0.7093 A | | 1928 Zachariasen, Cu, 1.5405 A | | 1949 Lander, Cu, 1.5405 A 20°C | | 1953 Swanson and Fuyat, Cu, 1.5405 A 25°C | |
|---|---|---|---|---|---|---|---|---|
| | *d* | *I* | *d* | *I* | *d* | *I* | *d* | *I* |
| | *A* | | *A* | | *A* | | *A* | |
| 110 | 4.57 | 7 | ------ | ---- | 4.54 | 5 | 4.56 | 9 |
| 020 | ------ | ---- | ------ | ---- | 4.45 | 3 | 4.45 | 4 |
| 111 | 3.73 | 100 | 3.70 | s(db) | 3.70 | 100 | 3.72 | 100 |
| 021 | | | | | 3.65 | 30 | 3.68 | 53 |
| 002 | 3.26 | 8 | ------ | ---- | 3.22 | 10 | 3.215 | 15 |
| 012 | 3.06 | 2 | 3.01 | vw | 3.02 | 5 | 3.025 | 4 |
| 102 | ------ | ---- | ------ | ---- | ------ | --- | 2.749 | 3 |
| 200 | 2.64 | 40 | 2.64 | m-s | 2.66 | 10 | 2.656 | 11 |
| 112 | | | | | 2.62 | 20 | 2.628 | 24 |
| 130 | ------ | ---- | 2.59 | s | 2.59 | 20 | 2.590 | 23 |
| 220 | 2.27 | 3 | 2.27 | w | 2.28 | 10 | 2.281 | 6 |
| 040 | ------ | ---- | 2.23 | vw | 2.22 | 5 | 2.226 | 2 |
| 221 | 2.14 | 20 | 2.144 | s | 2.13 | 40 | 2.150 | 28 |
| 041 | ------ | ---- | 2.102 | w | 2.101 | 20 | 2.104 | 12 |
| 202 | ------ | ---- | 2.043 | w | 2.052 | 20 | 2.048 | 10 |

**Barium carbonate (witherite), $BaCO_3$—Con.**

| *hkl* | 1938 Hanawalt, Rinn, and Frevel, Mo, 0.7093 A | | 1928 Zachariasen, Cu, 1.5405 A | | 1949 Lander, Cu, 1.5405 A 20°C | | 1953 Swanson and Fuyat, Cu, 1.5405 A 25°C | |
|---|---|---|---|---|---|---|---|---|
| | *d* | *I* | *d* | *I* | *d* | *I* | *d* | *I* |
| | *A* | | *A* | | *A* | | *A* | |
| 132 | 2.03 | 20 | 2.013 | m | 2.020 | 30 | 2.019 | 21 |
| 113 | 1.94 | 20 | 1.932 | m | 1.937 | 40 | 1.940 | 15 |
| 222 | 1.85 | 1 | 1.854 | w | 1.858 | 5 | 1.859 | 3 |
| 042 | ------ | ---- | ------ | ---- | 1.827 | 3 | 1.830 | 2 |
| 310 | ------ | ---- | ------ | ---- | 1.731 | 3 | 1.737 | 2 |
| 033 | | | | | | | | |
| 240 | ------ | ---- | ------ | ---- | 1.705 | 3 | 1.706 | 1 |
| 311 | ------ | ---- | 1.669 | m | 1.672 | 15 | 1.677 | 5 |
| 133 | 1.65 | 6 | 1.647 | m | 1.645 | 15 | 1.649 | 4 |
| 241 | | | | | | | | |
| 151 | ------ | ---- | 1.627 | w-m | 1.626 | 20 | 1.633 | 4 |
| 223 | 1.56 | 2 | 1.553 | w-m | 1.606 | 3 | 1.563 | 3 |
| 043 | ------ | ---- | ------ | ---- | 1.558 | 5 | 1.543 | < 1 |
| 330 | 1.52 | 5 | 1.516 | s(db) | 1.517 | 5 | 1.521 | 4 |
| 242 | ------ | ---- | | | | | 1.508 | 2 |
| 060 | ------ | ---- | ------ | ---- | ------ | --- | 1.484 | 1 |
| 143 | | | | | | | | |
| 332 | 1.378 | 10 | ------ | ---- | ------ | --- | 1.375 | 6 |
| 204 | | | | | | | | |
| 134 | ------ | ---- | 1.366 | s(db) | ------ | --- | 1.366 | 4 |
| 313 | 1.343 | 4 | 1.346 | s | ------ | --- | 1.348 | 4 |
| 062 | | | | | | | | |
| 243 | ------ | ---- | ------ | ---- | ------ | --- | 1.335 | 3 |
| 400 | ------ | ---- | 1.324 | s(db) | ------ | --- | 1.328 | 4 |
| 153 | | | | | | | | |
| 260 | 1.303 | 1 | 1.293 | s | ------ | --- | 1.295 | 3 |
| 234 | 1.242 | 4 | ------ | ---- | ------ | --- | 1.248 | 1 |
| 421 | | | | | | | | |
| 071 | | | | | | | | |
| 025 | ------ | ---- | ------ | ---- | ------ | --- | 1.233 | 2 |
| 351 | | | | | | | | |
| 253 | ------ | ---- | ------ | ---- | ------ | --- | 1.215 | < 1 |
| 412 | | | | | | | | |
| 171 | | | | | | | | |
| 125 | ------ | ---- | ------ | ---- | ------ | --- | 1.202 | < 1 |
| 262 | | | | | | | | |
| 244 | 1.175 | 1 | ------ | ---- | ------ | --- | 1.1703 | 1 |
| 432 | 1.122 | 2B | ------ | ---- | ------ | --- | 1.1335 | 2 |
| 081 | ------ | ---- | ------ | ----- | ------ | --- | 1.0951 | 2 |
| 423 | | | | | | | | |
| --- | 1.072 | 1B | ------ | ---- | ------ | --- | ------ | --- |
| --- | ------ | ---- | ------ | ---- | ------ | --- | ------ | --- |
| --- | 1.024 | 1B | ------ | ---- | ------ | --- | ------ | --- |
| --- | 0.994 | 1 | ------ | ---- | ------ | --- | ------ | --- |

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## Lead carbonate (cerussite), $PbCO_3$ (orthorhombic)

### ASTM cards

| Card number | | New index lines | Radiation | Source |
|---|---|---|---|---|
| Old | New | | | |
| 1361 | 1308 | 3.56 | Molybdenum | Hanawalt, Rinn, and Frevel [1] 1938. |
| | 1-0561 | 2.47 | | |
| | 1-0541 | 2.07 | | |
| ---- | 1303 | 3.53 | Copper---- | Anderson and Hochgesang, Lehigh University 1939. |
| | 3-0358 | 2.48 | | |
| | 3-0351 | 2.06 | | |
| ---- | 1304 | ----- | ---------- | A continuation of the preceding card. |
| | 3-0359 | | | |
| | 3-0352 | | | |
| ---- | 1326 | 3.51 | Copper---- | British Museum. |
| | 3-0362 | 1.83 | | |
| | 3-0357 | 1.18 | | |
| ---- | 1327 | ----- | ---------- | A continuation of the preceding card. |
| | 3-0363 | | | |
| | 3-0358 | | | |

### Additional published patterns

| Source | Radiation | Wavelength |
|---|---|---|
| Zachariasen [2] 1928---------- | Iron-------- | 1.934 |

**NBS pattern.** The lead carbonate sample used for the NBS pattern was prepared by the National Lead Co. Spectrographic analysis at the Bureau showed 0.001 to 0.01 percent each of bismuth, iron, magnesium, and silicon, and less than 0.001 percent each of aluminum, copper, silver, and calcium.

**Interplanar spacings and intensity measurements.** *D*-spacings from all of the ASTM card patterns were converted from kX to angstrom units, the Hanawalt, Rinn, and Frevel pattern from the literature and the other two directly from the ASTM cards. The Zachariasen *d*-spacings expressed in angstroms were calculated from his published Bragg angle data.

The three strongest lines for each of the patterns are as follows:

| Pattern | 1 | 2 | 3 |
|---|---|---|---|
| Hanawalt, Rinn, and Frevel----- | 111 | 130 | 221 |
| Anderson and Hochgesang-------- | 111 | 130 | 221 |
| Zachariasen---------- | 111 | 130 | 132 |
| Swanson and Fuyat---------- | 111 | 021 | 130 |

The strongest line of the British Museum pattern is also 111, but the pattern suffers from focusing and absorption so that the other intensity values are meaningless.

**Lattice constants.** The structure was determined by Zachariasen [2] in 1928. The space group is $D_{2h}^{16}$-Pmcn (Pnma) with potassium-nitrate-structure type and $4(PbCO_3)$ per unit cell.

Several unit-cell measurements have been converted to angstroms for comparison with the NBS values.

*Lattice constants in angstroms*

| | | *a* | *b* | *c* |
|---|---|---|---|---|
| 1928 | Zachariasen [2]----- | 5.15 | 8.46 | 6.11 |
| 1932 | LaCoste [3]--------- | 5.18 | 8.49 | 6.14 |
| 1933 | Colby and LaCoste [4] | 5.176 | 8.485 | 6.158 |
| 1939 | Lindsay and Hoyt [5] | 5.1830 | 8.4971 | 6.1426 |
| 1941 | Wyart [6]---------- | 5.18 | 8.48 | 6.14 |
| 1953 | Swanson and Fuyat---- | 5.195 | 8.436 | 6.152 at 26°C |

The density of lead carbonate calculated from the NBS lattice constants is 6.582 at 26°C. The sample reacts with the high index liquids available thus prohibiting a refractive-index measurement.

## Lead carbonate (cerussite), $PbCO_3$

| *hkl* | 1938 Hanawalt, Rinn, and Frevel Mo, 0.7093 A | | 1939 Anderson and Hochgesang Cu, 1.5405 A | | ---- British Museum Cu, 1.5405 A | | 1928 Zachariasen Fe, 1.93597 A | | 1953 Swanson and Fuyat Cu, 1.5405 A, 26°C | |
|---|---|---|---|---|---|---|---|---|---|---|
| | *d* | *I* | *d* | *I* | *d* | *I* | *d* | *I* | *d* | *I* |
| | *A* | | *A* | | *A* | | *A* | | *A* | |
| 110 | 4.41 | 6 | ---------- | ------- | ---------- | -------- | 4.40 | vw | 4.427 | 17 |
| 020 | 4.24 | 4 | ---------- | ------- | ---------- | -------- | 4.23 | vvw | 4.255 | 7 |
| ---------- | ---------- | ------- | 3.91 | 50 | ---------- | -------- | ---------- | ------- | ---------- | ------- |
| ---------- | ---------- | ------- | ---------- | ------- | 3.79 | 25 | ---------- | ------- | ---------- | ------- |
| 111 | 3.57 | 100 | 3.54 | 100 | 3.52 | 100 | 3.58 | s | 3.593 | 100 |
| 021 | 3.48 | 12 | ---------- | ------- | 3.22 | 50 | 3.48 | w | 3.498 | 43 |
| 002 | 3.06 | 16 | 3.06 | 40 | 3.01 | 50 | 3.06 | w | 3.074 | 24 |
| 012 | ---------- | ------- | ---------- | ------- | ---------- | -------- | ---------- | ------- | 2.893 | 2 |
| ---------- | ---------- | ------- | ---------- | ------- | 2.70 | 25 | ---------- | ------- | ---------- | ------- |
| 102 | ---------- | ------- | ---------- | ------- | ---------- | -------- | ---------- | ------- | 2.644 | 2 |
| 200 | 2.58 | 8 | 2.56 | 20 | 2.59 | 50 | 2.57 | w | 2.589 | 11 |
| 112 | ---------- | ------- | ---------- | ------- | ---------- | -------- | 2.50 | m | 2.522 | 20 |
| 130 | 2.48 | 32 | 2.49 | 80 | 2.44 | 75 | 2.47 | s | 2.487 | 32 |
| ---------- | ---------- | ------- | ---------- | ------- | 2.27 | 25 | ---------- | ------- | ---------- | ------- |
| 220 | 2.19 | 4 | 2.19 | 20 | 2.18 | 50 | 2.193 | w-m | 2.213 | 7 |
| 040 | ---------- | ------- | ---------- | ------- | ---------- | -------- | 2.122 | w | 2.129 | 2 |
| 221 | 2.07 | 28 | 2.06 | 80 | 2.09 | 50 | 2.069 | m | 2.081 | 27 |
| ---------- | ---------- | ------- | ---------- | ------- | 2.04 | 75 | ---------- | ------- | ---------- | ------- |
| 041 | 1.99 | 6 | 1.99 | 30 | 1.98 | 50 | 1.997 | w | 2.009 | 11 |
| 202 | 1.96 | 6 | 1.96 | 30 | 1.96 | 50 | 1.967 | w | 1.981 | 9 |
| 132 | 1.91 | 20 | 1.92 | 80 | 1.90 | 75 | 1.917 | s | 1.933 | 19 |
| 113 | ---------- | ------- | ---------- | ------- | ---------- | -------- | 1.843 | s(db) | 1.859 | 21 |
| 023 | 1.83 | 24 | 1.84 | 80 | 1.83 | 85 | | | 1.847 | 8 |
| 222 | 1.79 | 4 | ---------- | ------- | 1.78 | 25 | 1.788 | w | 1.796 | 4 |
| 042 | 1.74 | 4 | ---------- | ------- | ---------- | -------- | 1.737 | w | 1.750 | 2 |
| 310 | ---------- | ------- | 1.68 | 3 | 1.68 | 50 | 1.681 | w | 1.693 | 1 |
| 240 | | | | | | | | | | |
| 051 | 1.62 | 6 | 1.62 | 60 | 1.62 | 50 | 1.621 | m-s | 1.642 | 2 |
| 311 | | | | | | | | | 1.632 | 6 |
| 150 | | | | | | | | | 1.615 | 2 |
| 241 | 1.58 | 6 | 1.58 | 40 | 1.58 | 50 | 1.579 | m | 1.588 | 6 |
| 151 | 1.55 | 4 | 1.55 | 40 | 1.55 | 50 | 1.553 | m | 1.563 | 5 |
| 004 | ---------- | ------- | 1.53 | 5 | 1.52 | 50 | ---------- | ------- | 1.536 | 5 |
| 302 | 1.50 | 4 | 1.49 | 30 | 1.49 | 50 | 1.493 | m | 1.503 | 4 |
| 223 | | | | | | | | | | |
| 104 | | | | | | | | | | |
| 330 | 1.472 | 4 | 1.47 | 40 | 1.46 | 60 | ---------- | ------- | 1.475 | 5 |
| 043 | | | | | | | | | | |
| 411 | ---------- | ------- | 1.44 | 30 | 1.44 | 60 | ---------- | ------- | 1.449 | 3 |
| 242 | | | | | | | | | | |
| 152 | ---------- | ------- | ---------- | ------- | ---------- | -------- | ---------- | ------- | 1.430 | 2 |
| 060 | | | | | | | | | | |
| 143 | ---------- | ------- | ---------- | ------- | 1.41 | 25 | ---------- | ------- | 1.417 | 1 |
| 322 | | | | | | | | | | |
| 332 | ---------- | ------- | ---------- | ------- | ---------- | -------- | ---------- | ------- | 1.330 | 5 |
| 204 | ---------- | ------- | 1.32 | 40 | 1.32 | 50 | ---------- | ------- | 1.321 | 3 |

## Lead carbonate (cerussite), $PbCO_3$—Con.

| *hkl* | 1938 Hanawalt, Rinn, and Frevel Mo, 0.7093 A | | 1939 Anderson and Hochgesang Cu, 1.5405 A | | ---- British Museum Cu, 1.5405 A | | 1928 Zachariasen Fe, 1.93597 A | | 1953 Swanson and Fuyat Cu, 1.5405 A 26°C | |
|---|---|---|---|---|---|---|---|---|---|---|
| | *d* | *I* | *d* | *I* | *d* | *I* | *d* | *I* | *d* | *I* |
| | *A* | | *A* | | *A* | | *A* | | *A* | |
| 313<br>214<br>134 | ---------- | ------- | 1.30 | 60 | 1.30 | 75 | ---------- | ------- | 1.306 | 5 |
| 400 | ---------- | ------- | ---------- | ------- | ---------- | -------- | ---------- | ------- | 1.296 | 2 |
| 243 | ---------- | ------- | 1.28 | 30 | 1.28 | 50 | ---------- | ------- | 1.282 | 3 |
| 153 | ---------- | ------- | 1.26 | 20 | 1.26 | 25 | ---------- | ------- | 1.269 | 3 |
| 206 | ---------- | ------- | 1.24 | 30 | 1.24 | 50 | ---------- | ------- | 1.243 | 2 |
| 350<br>421 | ---------- | -------- | 1.21 | 30 | 1.21 | 75 | ---------- | ------- | 1.214 | 1 |
| ---------- | ---------- | ------- | 1.18 | 40 | 1.18 | 85 | ---------- | ------- | ---------- | ------- |
| ---------- | ---------- | ------- | 1.15 | 30 | 1.15 | 60D | ---------- | ------- | ---------- | ------- |
| ---------- | ---------- | ------- | ---------- | ------- | 1.12 | 25D | ---------- | ------- | ---------- | ------- |
| ---------- | ---------- | ------- | 1.09 | 30 | 1.08 | 50 | ---------- | ------- | ---------- | ------- |
| ---------- | ---------- | ------- | 1.07 | 20 | 1.07 | 50 | ---------- | ------- | ---------- | ------- |
| ---------- | ---------- | ------- | 1.06 | 40 | 1.06 | 75 | ---------- | ------- | ---------- | ------- |
| ---------- | ---------- | ------- | 1.04 | 30 | 1.04 | 75 | ---------- | ------- | ---------- | ------- |
| ---------- | ---------- | ------- | 1.02 | 30 | ---------- | -------- | ---------- | ------- | ---------- | ------- |
| ---------- | ---------- | ------- | 1.01 | 10 | ---------- | -------- | ---------- | ------- | ---------- | ------- |
| ---------- | ---------- | ------- | 0.995 | 10 | ---------- | -------- | ---------- | ------- | ---------- | ------- |
| ---------- | ---------- | ------- | .973 | 20 | ---------- | -------- | ---------- | ------- | ---------- | ------- |
| ---------- | ---------- | ------- | .945 | 10 | ---------- | -------- | ---------- | ------- | ---------- | ------- |
| ---------- | ---------- | ------- | .929 | 30 | ---------- | -------- | ---------- | ------- | ---------- | ------- |
| ---------- | ---------- | ------- | .888 | 20 | ---------- | -------- | ---------- | ------- | ---------- | ------- |
| ---------- | ---------- | ------- | .876 | 20 | ---------- | -------- | ---------- | ------- | ---------- | ------- |
| ---------- | ---------- | ------- | .864 | 20 | ---------- | -------- | ---------- | ------- | ---------- | ------- |
| ---------- | ---------- | ------- | .852 | 20 | ---------- | -------- | ---------- | ------- | ---------- | ------- |
| ---------- | ---------- | ------- | .841 | 20 | ---------- | -------- | ---------- | ------- | ---------- | ------- |
| ---------- | ---------- | ------- | .831 | 10 | ---------- | -------- | ---------- | ------- | ---------- | ------- |
| ---------- | ---------- | ------- | .823 | 30 | ---------- | -------- | ---------- | ------- | ---------- | ------- |
| ---------- | ---------- | ------- | .815 | 40 | ---------- | -------- | ---------- | ------- | ---------- | ------- |
| ---------- | ---------- | ------- | .803 | 30 | ---------- | -------- | ---------- | ------- | ---------- | ------- |
| ---------- | ---------- | ------- | .796 | 20 | ---------- | -------- | ---------- | ------- | ---------- | ------- |

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## 2.6. Sulfates

### Sodium sulfate (thenardite), $Na_2SO_4$ (orthorhombic)

**ASTM cards**

| Card number | | New index lines | Radiation | Source |
|---|---|---|---|---|
| Old | New | | | |
| II-1756 | 2586 | 2.78 | Molybdenum, 0.712 | Colby [1] 1931. |
| | 2-0832 | 4.63 | | |
| | 2-0838 | 2.64 | | |
| 2476 | 2651 | 2.78 | Molybdenum-- | Hanawalt, Rinn, and Frevel [2] 1938. |
| | 1-1025 | 1.87 | | |
| | 1-1009 | 4.66 | | |
| II-1678 | 2486 | 2.82 | Copper------ | British Museum. |
| | 2-0793 | 4.62 | | |
| | 2-0805 | 3.10 | | |

**Additional published patterns**

| Source | Radiation | Wavelength |
|---|---|---|
| Kracek and Ksanda [3] 1930--- | ---------- | ---------- |

**NBS pattern.** The sodium sulfate used for the NBS pattern was a specially purified sample contributed by the Mallinckrodt Chemical Works. Their spectrographic analysis shows 0.001 to 0.01 percent of aluminum, 0.0001 to 0.001 percent each of calcium and magnesium, and less than 0.0001 percent each of silver, copper, iron, and silicon.

**Interplanar spacings and intensity measurements.** The British Museum $d$-spacings taken from the ASTM cards were converted from kX to angstrom units. The other $d$-values taken from their source articles were converted from kX to angstrom units. For the British Museum pattern, the $d$-spacing at 3.46 A is not theoretically possible and spacings at 2.07 and 1.18 A are not allowed by the space group.

The three strongest lines for each of the patterns are as follows:

| Pattern | 1 | 2 | 3 |
|---|---|---|---|
| Colby---------- | 113 | 111 | 220 |
| Hanawalt, Rinn, and Frevel----- | 113 | 153 | 111 |
| British Museum---------- | 113 | 111 | 040 |
| Kracek and Ksanda---------- | 113 | 111 | 153 |
| Swanson and Fuyat---------- | 113 | 111 | 131 |

**Lattice constants.** The structure was determined by Gossner and Mussgnug [4] in 1929. The space group is $D_{2h}^{24}$-Fddd with sodium-sulfate-structure type and $8(Na_2SO_4)$ per unit cell. This phase, thenardite, is also called $Na_2SO_4(V)$. Between 160 and 180°C it inverts to monoclinic $Na_2SO_4(IV)$, at 185°C to orthorhombic $Na_2SO_4(III)$, and to hexagonal $Na_2SO_4(I)$ at 241°C; on cooling, it may invert to $Na_2SO_4(II)$ at 236°C.

A group of unit-cell determinations have been converted to angstroms for comparison with the NBS values.

*Lattice constants in angstroms*

| | | $a$ | $b$ | $c$ |
|---|---|---|---|---|
| 1929 | Gossner and Mussgnug [4]---------- | 5.90 | 12.33 | 9.81 |
| 1931 | Colby [1]---------- | 5.824 | 12.32 | 9.84 |
| 1932 | Zachariasen and Ziegler [5]-------- | 5.86 | 12.31 | 9.77 |
| 1953 | Swanson and Fuyat---- | 5.863 | 12.304 | 9.821 at 25°C |

The density of sodium sulfate calculated from the NBS lattice constants is 2.663 at 25°C. The NBS sample is optically positive with large 2V and refractive indices of $\alpha = 1.470$, $\beta = 1.475$ and $\gamma = 1.481$.

(See table, p. 60)

**References**


[1] M. Y. Colby, The crystal structure of anhydrous sodium sulfate, Z. Krist. **77**, 49 (1931).

[2] J. D. Hanawalt, H. W. Rinn and L. K. Frevel, Chemical analysis by X-ray diffraction, Ind. Eng. Chem., Anal. Ed. **10**, 457-512 (1938).

[3] F. C. Kracek and C. J. Ksanda, The polymorphism of sodium sulfate: IV. X-ray analysis, J. Phys. Chem. **34**, 1741-4 (1930).

[4] B. Gossner and F. Mussgnug, Über strukturelle Beziehungen bei Alkalisulfaten, Z. Krist. **69**, 446 (1929).

[5] W. H. Zachariasen and G. E. Ziegler, The crystal structure of anhydrous sodium sulfate, Z. Krist. **81**, 92 (1932).

## Sodium sulfate (thenardite) $Na_2SO_4$

| *hkl* | 1931 Colby Mo, 0.7093 A | | 1938 Hanawalt, Rinn, and Frevel Mo, 0.7093 A | | ---- British Museum Cu, 1.5405 A | | 1930 Kracek and Ksanda ---------- | | 1953 Swanson and Fuyat Cu, 1.5405 A, 25°C | |
|---|---|---|---|---|---|---|---|---|---|---|
| | *d* | *I* | *d* | *I* | *d* | *I* | *d* | *I* | *d* | *I* |
| | *A* | | *A* | | *A* | | *A* | | *A* | |
| 111 | 4.64 | 80 | 4.67 | 40 | 4.63 | 80 | 4.68 | 80 | 4.66 | 73 |
| 022 | 3.83 | 20 | 3.85 | 13 | 3.87 | 40 | 3.85 | 40 | 3.84 | 18 |
| ---------- | ---------- | ------- | ---------- | ------- | 3.46 | 40 | ---------- | ------- | ---------- | ------- |
| 131 | 3.18 | 40 | 3.19 | 33 | ---------- | -------- | 3.181 | 70 | 3.178 | 51 |
| 040 | 3.08 | 27 | 3.09 | 27 | 3.10 | 80B | 3.077 | 60 | 3.075 | 47 |
| 113 | 2.79 | 100 | 2.79 | 100 | 2.82 | 100 | 2.778 | 100 | 2.783 | 100 |
| 220 | 2.65 | 80 | 2.65 | 40 | 2.64 | 60 | 2.640 | 70 | 2.646 | 48 |
| 222 | 2.33 | 60 | 2.33 | 27 | 2.31 | 60 | 2.327 | 60 | 2.329 | 21 |
| 151 | 2.20 | 10 | 2.22 | 3 | 2.21 | 40 | 2.197 | 20 | 2.211 | 5 |
| ---------- | ---------- | ------- | ---------- | ------- | 2.07 | 60 | ---------- | ------- | ---------- | ------- |
| 044 | 1.92 | 5 | ---------- | ------- | ---------- | -------- | ---------- | ------- | 1.919 | 4 |
| 311 | } ---------- | -------- | ---------- | ------- | ---------- | -------- | ---------- | ------- | 1.891 | 4 |
| 062 | } | | | | | | | | | |
| 153 | 1.86 | 80 | 1.87 | 53 | 1.86 | 80 | 1.864 | 80 | 1.864 | 31 |
| 115 | ---------- | ------- | ---------- | ------- | ---------- | -------- | ---------- | ------- | 1.841 | 6 |
| 224 | 1.80 | 10 | 1.79 | 5 | 1.79 | 20 | 1.797 | 30 | 1.798 | 4 |
| 260 | 1.67 | 40 | 1.67 | 17 | ---------- | -------- | 1.679 | 50 | 1.680 | 12 |
| 313 | ---------- | ------- | ---------- | ------- | 1.66 | 60 | ---------- | ------- | 1.662 | 8 |
| 244 | 1.60 | 10 | 1.60 | 5 | 1.60 | 40 | 1.604 | 20 | 1.605 | 5 |
| 262 | ---------- | ------- | ---------- | ------- | ---------- | -------- | ---------- | ------- | 1.589 | 3 |
| 333 | 1.55 | 30 | 1.55 | 20 | ---------- | -------- | 1.550 | 40 | 1.553 | 10 |
| 080 | ---------- | ------- | ---------- | ------- | 1.54 | 60 | ---------- | ------- | 1.537 | <1 |
| 351 | ---------- | ------- | ---------- | ------- | ---------- | -------- | ---------- | ------- | 1.512 | 2 |
| 173 | 1.50 | 20 | 1.50 | 8 | 1.50 | 40 | 1.499 | 20 | 1.497 | 5 |
| 400 | 1.46 | 5 | ---------- | ------- | ---------- | -------- | ---------- | ------- | 1.465 | <1 |
| 206 | 1.43 | 20 | 1.43 | 11 | 1.43 | 60 | 1.427 | 30 | 1.429 | 5 |
| 264 | } 1.38 | 10 | 1.39 | 5 | 1.38 | 50 | 1.382 | 20 | 1.386 | 3 |
| 353 | } | | | | | | | | | |
| 440 | 1.32 | 10 | 1.32 | 4 | 1.32 | 50 | 1.321 | 20 | 1.324 | 3 |
| 084 | ---------- | ------- | 1.30 | 16 | ---------- | -------- | ---------- | ------- | 1.304 | 3 |
| 246 | 1.29 | 20 | ---------- | ------- | 1.29 | 60 | 1.296 | 40 | 1.297 | 6 |
| 066 | ---------- | ------- | 1.28 | 8 | 1.28 | 60 | 1.277 | 30 | 1.279 | 5 |
| 404 | 1.25 | 5 | ---------- | ------- | 1.25 | 40 | 1.258 | 10 | 1.258 | 1 |
| 424 | } 1.23 | 10 | ---------- | ------- | 1.23 | 20 | 1.234 | 20 | 1.233 | 1 |
| 193 | } | | | | | | | | | |
| 373 | 1.21 | 10 | 1.21 | 3 | 1.21 | 40 | 1.213 | 20 | 1.214 | 1 |
| 0·10·2 | ---------- | ------- | ---------- | ------- | 1.19 | 20 | 1.189 | 10 | 1.1922 | <1 |
| ---------- | ---------- | ------- | ---------- | ------- | 1.18 | 20 | ---------- | ------- | ---------- | ------- |
| 444 | 1.16 | 5 | 1.16 | 1 | 1.16 | 20 | 1.160 | 20 | 1.1654 | <1 |
| 2·10·0 | ---------- | ------- | 1.13 | 5 | 1.14 | 60 | 1.134 | 30 | 1.1345 | 3 |
| ---------- | 1.10 | 10 | 1.10 | 1 | 1.10 | 40 | 1.098 | 10 | ---------- | ------- |
| ---------- | 1.07 | 10 | 1.08 | 5 | 1.08 | 60 | 1.073 | 30 | ---------- | ------- |
| ---------- | ---------- | ------- | ---------- | ------- | ---------- | -------- | 1.066 | 30 | ---------- | ------- |
| ---------- | 1.06 | 10 | 1.06 | 10 | 1.06 | 60 | 1.060 | 20 | ---------- | ------- |
| ---------- | 1.04 | 10 | 1.04 | 4 | 1.04 | 40 | 1.042 | 20 | ---------- | ------- |
| ---------- | 1.03 | 5 | ---------- | ------- | 1.03 | 20 | ---------- | ------- | ---------- | ------- |
| ---------- | 1.01 | 5 | ---------- | ------- | ---------- | -------- | ---------- | ------- | ---------- | ------- |
| ---------- | 0.98 | 5 | 0.98 | 3 | ---------- | -------- | 0.982 | 20 | ---------- | ------- |
| ---------- | .96 | 10 | .97 | 4 | ---------- | -------- | .964 | 30 | ---------- | ------- |
| ---------- | ---------- | ------- | .93 | 1 | ---------- | -------- | .931 | 20 | ---------- | ------- |

## Strontium sulfate (celestite), $SrSO_4$ (orthorhombic)

**ASTM cards**

| Card number | | New index lines | Radiation | Source |
|---|---|---|---|---|
| Old | New | | | |
| 2171 | 2200 | 2.97 | Molybdenum-- | Hanawalt, Rinn, and Frevel [1] 1938. |
| | 1-0890 | 2.70 | | |
| | 1-0885 | 2.03 | | |
| ---- | 1566 | 3.32 | Copper------ | British Museum. |
| | 3-0438 | 2.98 | | |
| | 3-0437 | 2.05 | | |

**Additional published patterns.** None.

**NBS pattern.** The strontium sulfate used for the NBS pattern was a specially purified sample prepared by the Mallinckrodt Chemical Works. Their spectrographic analysis showed the following impurities: 0.01 to 0.1 percent of barium, 0.001 to 0.01 percent of sodium, 0.0001 to 0.001 percent of aluminum, and less than 0.0001 percent each of calcium, cesium, copper, iron, potassium, lithium, magnesium, and silicon.

**Interplanar spacings and intensity measurements.** The Hanawalt, Rinn, and Frevel and British Museum *d*-spacings were converted from kX to angstrom units. The three strongest lines for each pattern are as follows:

| Pattern | 1 | 2 | 3 |
|---|---|---|---|
| Hanawalt, Rinn, and Frevel---- | 211 | 112 | 113 |
| British Museum---------- | 210 | 211 | 113 |
| Swanson and Fuyat---------- | 211 | 210 | 112 |

The line at *d*-spacing 4.69 A for the British Museum pattern is not allowed by the space group. The 1.88 line of the Hanawalt, Rinn, and Frevel pattern could be indexed but does not occur in the NBS pattern.

**Lattice constants.** The structure was determined by James and Wood [2] in 1925. The space group is $D_{2h}^{16}$-Pnma with barium-sulfate-structure type and $4(SrSO_4)$ per unit cell.

Several unit-cell determinations have been converted from kX to angstrom units for comparison with the NBS values.

*Lattice constants in angstroms*

| | | *a* | *b* | *c* |
|---|---|---|---|---|
| 1925 | James and Wood [2]--- | 8.36 | 5.36 | 6.84 |
| 1926 | Basche and Mark [3]-- | 8.31 | 5.3 | 6.8 |
| 1953 | Swanson and Fuyat----- | 8.359 | 5.352 | 6.866 at 26°C |

The density of strontium sulfate calculated from the NBS lattice constant is 3.971 at 26°C. The NBS sample was too finely divided for a refractive-index determination.

**Strontium sulfate (celestite), $SrSO_4$**

| *hkl* | 1938 Hanawalt, Rinn, and Frevel Mo, 0.709 A | | ---- British Museum Cu, 1.5405 A | | 1953 Swanson and Fuyat Cu, 1.5405 A, 26°C | |
|---|---|---|---|---|---|---|
| | *d* | *I* | *d* | *I* | *d* | *I* |
| | *A* | | *A* | | *A* | |
| --- | --------- | ----- | 4.69 | 25 | --------- | ------- |
| 011 | 4.23 | 14 | 4.21 | 50 | 4.23 | 11 |
| 111 | --------- | ----- | 3.73 | 50 | 3.77 | 35 |
| 201 | --------- | ----- | ------- | ----- | 3.57 | 2 |
| 002 | 3.43 | 14 | ------- | ----- | 3.433 | 30 |
| 210 | 3.30 | 29 | 3.33 | 100 | 3.295 | 98 |
| 102 | 3.17 | 21 | 3.18 | 75 | 3.177 | 59 |
| 211 | 2.98 | 100 | 2.99 | 100 | 2.972 | 100 |
| 112 | 2.71 | 72 | 2.77 | 75 | 2.731 | 63 |
| 020 | --------- | ----- | 2.69 | 75 | 2.674 | 49 |
| 301 | --------- | ----- | ------- | ----- | 2.582 | 6 |
| 121 | --------- | ----- | 2.38 | 50 | 2.388 | 7 |
| 212 | 2.37 | 14 | ------- | ----- | 2.377 | 17 |
| 220 | 2.26 | 14 | 2.27 | 60 | 2.253 | 18 |
| 103 | --------- | ----- | 2.23 | 50 | 2.208 | 5 |
| 302 | --------- | ----- | ------- | ----- | 2.164 | 7 |
| 221 | 2.12 | 14 | 2.14 | 50 | 2.141 | 25 |
| 122 | --------- | ----- | ------- | ----- | 2.045 | 55 |
| 113 | 2.03 | 43 | 2.05 | 100 | 2.041 | 57 |
| 203 | --------- | ----- | ------- | ----- | 2.006 | 40 |
| 401 | 2.00 | 21 | 2.00 | 100 | 1.999 | 48 |
| 410 | 1.94 | 14 | 1.96 | 25 | 1.947 | 15 |
| --- | 1.88 | 14 | ------- | ----- | --------- | ------- |
| 321 | 1.84 | 14 | 1.86 | 25 | 1.857 | 7 |
| 303 | 1.76 | 14 | 1.77 | 50 | 1.769 | 17 |
| 031 | --------- | ----- | ------- | ----- | 1.728 | 2 |
| 004 | --------- | ----- | 1.72 | 25 | 1.715 | 3 |
| 412 / 131 | --------- | ----- | ------- | ----- | 1.691 | 3 |
| 313 | 1.67 | 14 | 1.68 | 50 | 1.679 | 9 |
| 230 | --------- | ----- | 1.64 | 25 | 1.640 | 5 |
| 501 | --------- | ----- | ------- | ----- | 1.625 | 2 |

**Strontium sulfate (celestite), $SrSO_4$—Con.**

| *hkl* | 1938 Hanawalt, Rinn, and Frevel Mo, 0.709 A | | ---- British Museum Cu, 1.5405 A | | 1953 Swanson and Fuyat Cu, 1.5405 A, 26°C | |
|---|---|---|---|---|---|---|
| | *d* | *I* | *d* | *I* | *d* | *I* |
| | *A* | | *A* | | *A* | |
| 223 | | | | | | |
| 114 | -------- | ----- | ------- | ----- | 1.604 | 7 |
| 421 | | | | | 1.601 | 15 |
| 231 | 1.59 | 14 | 1.60 | 75 | 1.596 | 10 |
| 132 | | | | | | |
| 511 | 1.55 | 14 | 1.56 | 25 | 1.555 | 11 |
| 214 | --------- | ----- | ------- | ----- | 1.521 | 1 |
| 323 | 1.468 | 14 | 1.46 | 60 | 1.475 | 16 |
| 512 | --------- | ----- | | | 1.447 | 6 |
| 024 | --------- | ----- | 1.45 | 25 | 1.444 | 5 |
| 124 | 1.418 | 14 | 1.43 | 25 | 1.424 | 6 |
| 314 | --------- | ----- | ------- | ----- | 1.410 | 3 |
| 521 | --------- | ----- | 1.39 | 50 | 1.388 | 9 |
| 332 | --------- | ----- | ------- | ----- | 1.376 | 5 |
| 430 | | | | | | |
| 105 | -------- | ----- | 1.36 | 50 | 1.356 | 4 |
| 610 | --------- | ----- | ------- | ----- | 1.348 | 7 |
| 040 | | | | | | |
| 423 | -------- | ----- | ------- | ----- | 1.338 | 5 |
| 233 | --------- | ----- | ------- | ----- | 1.334 | 4 |
| 015 | --------- | ----- | 1.33 | 50 | 1.330 | 3 |
| 611 | --------- | ----- | ------- | ----- | 1.323 | 9 |
| 513 | --------- | ----- | ------- | ----- | 1.309 | 6 |
| 215 | --------- | ----- | 1.27 | 25 | 1.268 | 4 |
| --- | --------- | ----- | 1.24 | 25 | --------- | ------- |
| --- | --------- | ----- | 1.21 | 50 | --------- | ------- |
| --- | --------- | ----- | 1.18 | 25 | --------- | ------- |
| --- | --------- | ----- | 1.15 | 50 | --------- | ------- |
| --- | --------- | ----- | 1.13 | 25 | --------- | ------- |
| --- | --------- | ----- | 1.12 | 50 | --------- | ------- |
| --- | --------- | ----- | 1.08 | 50 | --------- | ------- |
| --- | --------- | ----- | 1.07 | 25 | --------- | ------- |
| --- | --------- | ----- | 1.06 | 50 | --------- | ------- |
| --- | --------- | ----- | 1.04 | 50 | --------- | ------- |

## References


[1] J. D. Hanawalt, H. W. Rinn, and L. K. Frevel, Chemical analysis by X-ray diffraction, Ind. Eng. Chem., Anal. Ed. **10**, 457-512 (1938).

[2] R. W. James and W. A. Wood, The crystal structure of barytes, celestine, and anglesite, Proc. Roy. Soc. (London) **109A**, 598-620 (1925).

[3] W. Basche and H. Mark, Über die Struktur von Verbindungen des Typus $MeXO_4$, Z. Krist. **64**, 1 (1926).


## 2.7. Silicates

### Zinc pyrosilicate hydrate (hemimorphite), $Zn_4(OH)_2Si_2O_7 \cdot H_2O$ (orthorhombic)

**ASTM cards**

| Card number Old | Card number New | New index lines | Radiation | Source |
|---|---|---|---|---|
| II-1224 | 1860 | 3.12 | Copper------ | British Museum. |
| | 2-0554 | 6.7 | | |
| | 2-0562 | 3.29 | | |
| ------- | 1968 | 3.08 | Molybdenum-- | New Jersey Zinc Co. |
| | 3-0568 | 3.26 | | |
| | 3-0571 | 2.54 | | |

Hemimorphite is not an orthosilicate as stated on the new ASTM cards but rather a pyrosilicate with the formula given above as proved by Ito and West [1] and others.

**Additional published patterns.** None.

**NBS patterns.** The hemimorphite sample used for the NBS pattern is a mineral specimen from Sterling Hill, New Jersey, U. S. National Museum Sample No. 101991.

Spectrographic analysis at the Bureau shows 0.01 to 0.1 percent of phosphorus, 0.001 to 0.01 percent each of aluminum, arsenic, boron, beryllium, and magnesium, and 0.0001 to 0.001 percent each of calcium, copper, and iron.

**Interplanar spacings and intensity measurements.** *D*-spacings from both ASTM card patterns were converted from kX to angstrom units. The three strongest lines for each of the patterns are as follows:

| Pattern | 1 | 2 | 3 |
|---|---|---|---|
| British Museum---------- | 211 | 110 | 220, 130 |
| New Jersey Zinc--------- | 211 | 220, 130 | 002 |
| Swanson and Fuyat------- | 211 | 110 | 130 |

The British Museum *d*-spacing at 3.68 A is not allowed by the space group of hemimorphite.

**Lattice constants.** The structure was determined by Gossner and Mussgnug [1] in 1929. The body-centered orthorhombic lattice has space group $C_{2v}^{20}$-Imm, hemimorphite-type structure, and $2(Zn_4(OH)_2Si_2O_7 \cdot H_2O)$ per unit cell.

Two unit-cell determinations have been converted from kX to angstrom units for comparison with the NBS values.

*Lattice constants in angstroms*

| | | *a* | *b* | *c* |
|---|---|---|---|---|
| 1929 | Gossner and Mussgnug [1] | 8.43 | 10.75 | 5.15 |
| 1932 | Ito and West [2] | 8.40 | 10.72 | 5.12 |
| 1953 | Swanson and Fuyat | 8.370 | 10.719 | 5.120 at 25°C |

The density of hemimorphite calculated from the NBS lattice constant is 3.366 at 25°C. The NBS sample is optically positive with 2V of roughly 40° and refractive indices $\alpha = 1.611$, $\beta = 1.619$, and $\gamma = 1.630$.

**Zinc pyrosilicate hydrate (hemimorphite) $Zn_4(OH)_2Si_2O_7 \cdot H_2O$**

| *hkl* | ---- British Museum Cu, 1.5405 A | | ---- New Jersey Zinc Mo, 0.709 A | | 1953 Swanson and Fuyat Cu, 1.5405 A, 25°C | |
|---|---|---|---|---|---|---|
| | *d* | *I* | *d* | *I* | *d* | *I* |
| | *A* | | *A* | | *A* | |
| 110 | 6.7 | 80 | --- | --- | 6.60 | 86 |
| 020 | 5.4 | 70 | --- | --- | 5.36 | 55 |
| 011 | 4.6 | 70 | 4.64 | 4 | 4.62 | 41 |
| 200 | 4.24 | 60 | 4.19 | 4 | 4.18 | 38 |
| ---- | 3.68 | 40 | --- | --- | --- | --- |
| 220, 130 | 3.30 | 80 | 3.27 | 72 | 3.296; 3.288 | 73; 75 |
| 211 | 3.13 | 100 | 3.09 | 100 | 3.104 | 100 |
| 031 | 2.95 | 40 | 2.92 | 16 | 2.929 | 40 |
| 310 | 2.70 | 20 | --- | --- | 2.698 | 10 |
| 040 | --- | --- | 2.65 | 2 | 2.679 | 7 |
| 002 | 2.57 | 70 | 2.55 | 48 | 2.559 | 51 |
| 301, 231 | 2.43 | 80d | 2.42; 2.38 | 4; 40 | 2.450; 2.400 | 32; 54 |
| 022 | 2.32 | 20 | --- | --- | 2.309 | 3 |
| 141 | --- | --- | --- | --- | 2.284 | 2 |
| 321 | 2.21 | 70 | --- | --- | 2.229 | 11 |
| 330 | --- | --- | --- | --- | 2.198 | 19 |
| 202 | --- | --- | 2.18 | 20 | 2.183 | 16 |
| 400 | 2.10 | 20 | --- | --- | 2.092 | 10 |
| 150 | --- | --- | 2.06 | 2 | 2.077 | 1 |
| 222 | 2.03 | 40 | 2.01 | 12 | 2.020 | 13 |
| 051 | 1.99 | 20 | --- | --- | 1.977 | 2 |
| 312 | 1.86 | 40 | 1.85 | 4 | 1.857 | 4 |
| 042 | --- | --- | --- | --- | 1.851 | 7 |
| 341 | 1.81 | 70 | --- | --- | 1.808 | 17 |
| 060, 251 | --- | --- | 1.79 | 20 | 1.786 | 16 |
| 431 | 1.71 | 50 | --- | --- | 1.702 | 6 |
| 350 | --- | --- | --- | --- | 1.699 | 4 |

**Zinc pyrosilicate hydrate (hemimorphite) $Zn_4(OH)_2Si_2O_7 \cdot H_2O$**

| *hkl* | ---- British Museum Cu, 1.5405 A | | ---- New Jersey Zinc Mo, 0.709 A | | 1953 Swanson and Fuyat Cu, 1.5405 A, 25°C | |
|---|---|---|---|---|---|---|
| | *d* | *I* | *d* | *I* | *d* | *I* |
| | *A* | | *A* | | *A* | |
| 242 | --- | --- | 1.69 | 2 | 1.693 | 4 |
| 013 | --- | --- | --- | --- | 1.686 | 3 |
| 332 | 1.67 | 50 | --- | --- | 1.668 | 10 |
| 161 | --- | --- | 1.65 | 8 | 1.654 | 6 |
| 440 | --- | --- | --- | --- | 1.650 | 8 |
| 260 | --- | --- | --- | --- | 1.644 | 4 |
| 402 | 1.61 | 40d | --- | --- | 1.620 | 3 |
| 123, 501 | --- | --- | --- | --- | 1.590 | 3 |
| 213 | 1.569 | 40 | 1.56 | 4 | 1.563 | 8 |
| 033 | 1.535 | 40 | --- | --- | 1.540 | 6 |
| 521 | 1.527 | 70 | --- | --- | 1.526 | 4 |
| 530 | --- | --- | 1.51 | 12 | 1.516 | 14 |
| 071, 062 | 1.475 | 60 | --- | --- | 1.465 | 9 |
| 303 | 1.454 | 80 | 1.46 | 8 | 1.456 | 10 |
| 233, 361 | --- | --- | 1.43 | 32 | 1.445 | 16 |
| 143, 352 | 1.417 | 20 | --- | --- | 1.417 | 1 |
| 323 | --- | --- | --- | --- | 1.405 | 4 |
| 600 | 1.392 | 70 | --- | --- | 1.395 | 1 |
| 512 | --- | --- | --- | --- | 1.389 | 5 |
| 442, 271, 262 | --- | --- | 1.38 | 20 | 1.384 | 12 |
| 541 | 1.371 | 20 | --- | --- | 1.369 | 2 |
| 460 | --- | --- | --- | --- | 1.358 | 2 |
| 053, 611 | --- | --- | --- | --- | 1.335 | 1 |
| 532 | 1.311 | 60 | 1.30 | 16 | 1.304 | 11 |
| 181, 004 | 1.287 | 40 | --- | --- | 1.279 | 5 |
| ---- | 1.261 | 20 | 1.27 | 12 | --- | --- |
| ---- | --- | --- | [a] 1.22 | 2 | --- | --- |

[a] Seven additional lines have been omitted.

## References


[1] B. Gossner and F. Mussgnug, Vergleichende röntgenographische Untersuchung von Silikaten, Z. Krist. **70**, 171-84 (1929).

[2] T. Ito and J. West, The structure of hemimorphite ($H_2Zn_2SiO_5$), Z. Krist. **83**, 1-8 (1932).

# 3. Cumulative Index to Volumes I and II





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