VNIVERSITAT DE VALÈNCIA

*Servei de Recursos Humans (PTGAS)*

# PRUEBAS SELECTIVAS DE ACCESO, POR EL TURNO LIBRE, A LA ESCALA TÉCNICA SUPERIOR DE INVESTIGACIÓN (GRUPO A, SUBGRUPO A1).

## Subescala de Difracción de Rayos X

## 3er Ejercicio

## 9 de mayo de 2025



.

.

**Caso práctico 1.**

**Estudio estructural de materiales híbridos polímero-inorgánicos mediante SAXS y WAXD**

Un equipo de investigación está caracterizando nuevos materiales híbridos formados por una matriz polimérica y dominios inorgánicos basados en molibdeno. A temperatura ambiente, estos materiales presentan una **estructura cúbica primitiva simple (cP)** observable por SAXS, con una ordenación de largo alcance (de varios nanómetros) atribuible a la autoorganización del sistema híbrido.

Después de un tratamiento térmico a temperaturas superiores a 600 °C, el polímero se elimina por descomposición y oxidación, siendo el material resultante principalmente **óxido de molibdeno(VI) ($MoO_3$)**, que cristaliza en un sistema ortorrómbico con patrones difractométricos típicos observables mediante WAXD.

Los patrones bidimensionales obtenidos para una muestra representativa, tal como se ha sintetizado y después del tratamiento térmico, se muestran en la figura 1.

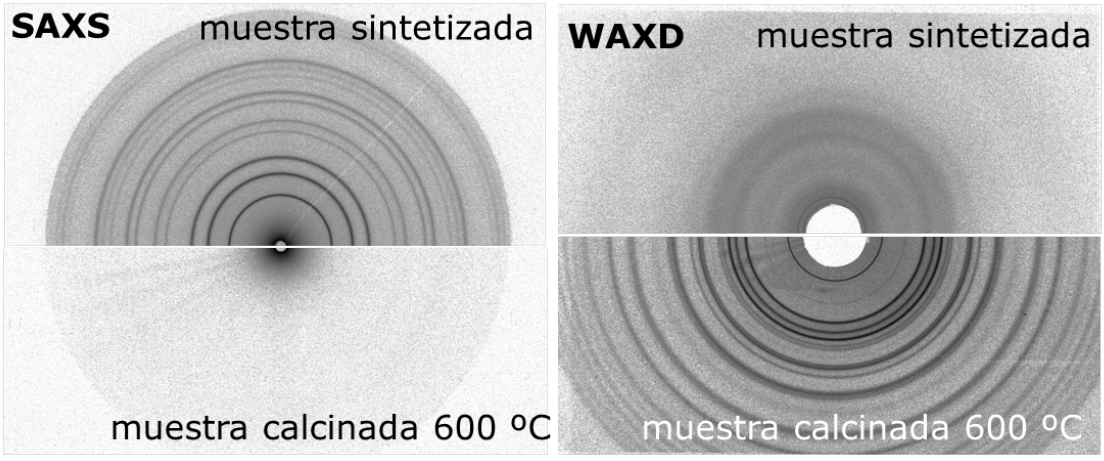


Figura 1. Patrones de SAXS y WAXD para una muestra representativa del estudio.

**Objetivo de la investigación:** Estudiar los cambios estructurales asociados al calentamiento de estos materiales utilizando SAXS y WAXD.



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**PREGUNTAS:**

**1. Preparación de muestras (*2 puntos*):**

a) ¿Cómo prepararíais las muestras en estado inicial (híbrido) y después del tratamiento térmico para las técnicas de SAXS y WAXD?

b) ¿Cómo acondicionaríais las muestras para evitar la sublimación o descomposición parcial del $MoO_3$ durante la medida?

c) ¿Qué formato de muestra (film, polvo compactado, capilar...) preferiríais para cada técnica?

**2. SAXS a temperatura ambiente (*2 puntos*):**

a) ¿Qué tipo de señales difractométricas esperaríais ver para una estructura cúbica primitiva? ¿Se ajustan con los patrones 2D observados?

b) ¿Cómo interpretaríais la distancia entre picos en términos de parámetro de red y periodicidad de los dominios ordenados?

**3. WAXD después del tratamiento (>600 °C) (*2 puntos*):**

a) ¿Cómo confirmaríais si la muestra es puramente ortorrómbica o si coexisten otras fases (como $MoO_2$)?

b) ¿Cómo podríais cuantificar la cristalinidad del $MoO_3$ obtenido?

**4. Análisis complementario (*2 puntos*):**

a) Si se quisiera seguir *in situ* la evolución de la transición estructural durante el calentamiento, ¿qué opciones instrumentales ofrecerían SAXS/WAXD?

b) ¿Cómo podríais controlar o estudiar posibles cambios de oxidación del molibdeno durante el tratamiento (de $Mo^{6+}$ a $Mo^{5+}$ o $Mo^{4+}$)?

**5. Interpretación final (proponedla, según vuestros conocimientos y opinión, no hay una única respuesta correcta) (*2 puntos*):**

a) ¿Qué relación creéis que podría haber entre la desaparición de los picos SAXS y la formación de $MoO_3$ cristalino?

b) ¿Qué implicaciones tiene la volatilidad del $MoO_3$ (punto de sublimación ~700–800°C) para el procesamiento térmico del material?

c) ¿Cómo modificaríais la composición inicial o las condiciones de tratamiento para controlar mejor la morfología y cristalinidad final?



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**Caso práctico 2.**

El difractograma adjunto corresponde a una muestra denominada CCIGME2D, mezcla de portlandita con un carbonato, y fue obtenido con radiación Cu $K\alpha_1$ $K\alpha_2$, efectuándose corrección de Rachinger (eliminación de $K\alpha_2$) y sustracción de fondo. Debajo se incluyen fichas de portlandita, y posibles carbonatos acompañantes. El registro presenta desviación (*offset*) constante de 2θ en todo el registro, y no se observaron picos a valores 2θº menores de 18º.

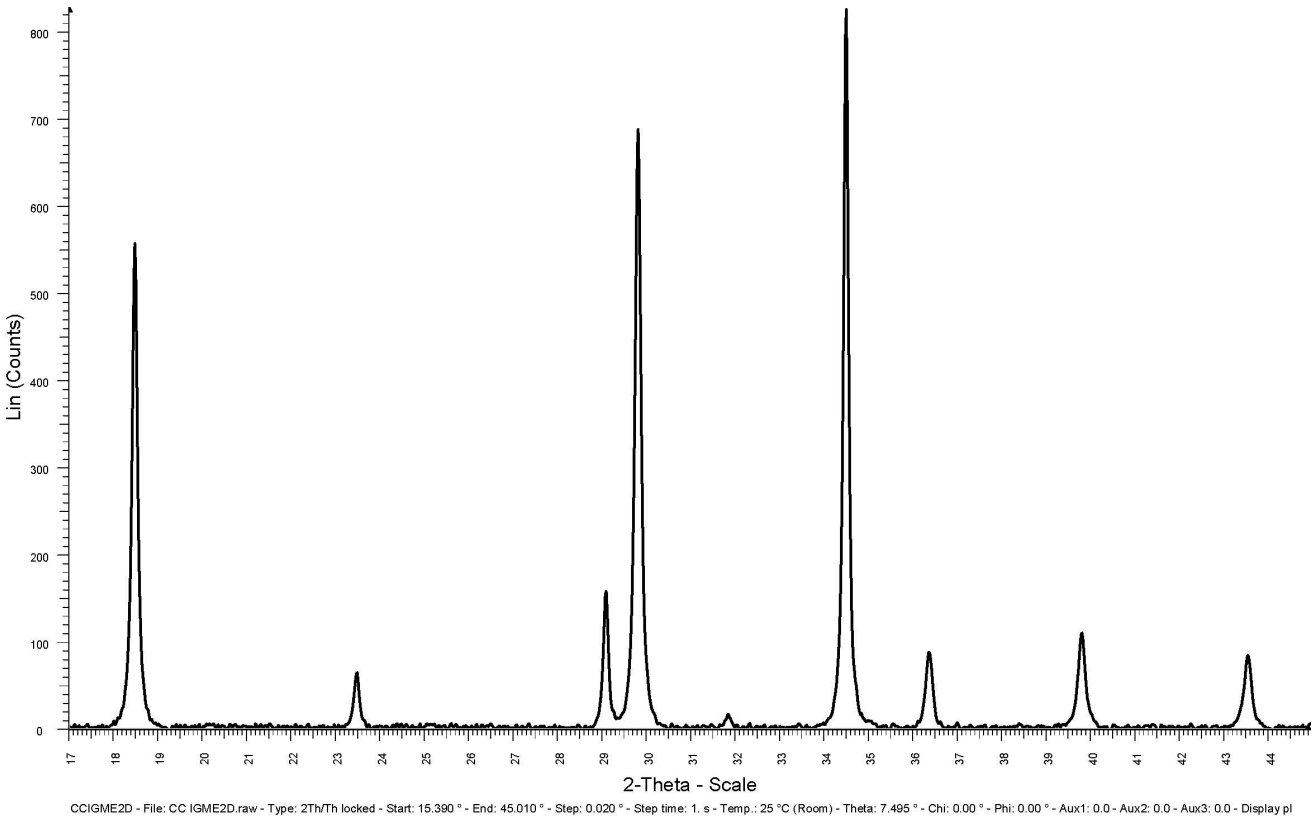
λ Cu $K\alpha_1$ = 1,5406 Å.

Se pide:

1) Identificar el carbonato presente (*7 puntos*).

2) Efectuar una estimación semicuantitativa de los dos constituyentes, utilizando el método de las intensidades de referencia, aplicado a los picos de mayor intensidad, admitiendo que el valor de FWHM es constante para todos los picos (*3 puntos*).

**Difractograma**



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## FICHAS POSIBLES (1 y 2):

CALCITE

_database_code_amcsd 0000098

CELL PARAMETERS: 4.9900 4.9900 17.0615 90.000 90.000 120.000
SPACE GROUP: R-3c
X-RAY WAVELENGTH: 1.541838
Cell Volume: 367.916
Density (g/cm3): 2.710
MAX. ABS. INTENSITY / VOLUME**2: 26.50717622
RIR: 3.185
RIR based on corundum from Acta Crystallographica A38 (1982) 733-739

| 2-THETA | INTENSITY | D-SPACING | H | K | L | Multiplicity |
|---|---|---|---|---|---|---|
| 23.07 | 8.10 | 3.8550 | 0 | 1 | 2 | 6 |
| 29.42 | 100.00 | 3.0357 | 1 | 0 | 4 | 6 |
| 31.46 | 2.43 | 2.8436 | 0 | 0 | 6 | 2 |
| 36.00 | 13.71 | 2.4950 | 1 | 1 | 0 | 6 |
| 39.44 | 20.16 | 2.2848 | 1 | 1 | 3 | 12 |
| 43.19 | 14.26 | 2.0946 | 2 | 0 | 2 | 6 |
| 47.15 | 6.16 | 1.9275 | 0 | 2 | 4 | 6 |
| 47.54 | 19.67 | 1.9125 | 0 | 1 | 8 | 6 |
| 48.54 | 20.02 | 1.8754 | 1 | 1 | 6 | 12 |
| 56.61 | 3.73 | 1.6259 | 2 | 1 | 1 | 12 |
| 57.44 | 9.18 | 1.6042 | 1 | 2 | 2 | 12 |
| 60.72 | 5.28 | 1.5253 | 2 | 1 | 4 | 12 |
| 61.05 | 2.41 | 1.5179 | 2 | 0 | 8 | 6 |
| 61.43 | 3.18 | 1.5094 | 1 | 1 | 9 | 12 |
| 63.10 | 2.29 | 1.4733 | 1 | 2 | 5 | 12 |
| 64.71 | 6.84 | 1.4405 | 3 | 0 | 0 | 6 |
| 65.67 | 3.91 | 1.4218 | 0 | 0 | 12 | 2 |
| 69.24 | 1.51 | 1.3569 | 2 | 1 | 7 | 12 |
| 70.30 | 2.13 | 1.3390 | 0 | 2 | 10 | 6 |
| 72.95 | 2.69 | 1.2967 | 1 | 2 | 8 | 12 |
| 77.23 | 1.93 | 1.2353 | 1 | 1 | 12 | 12 |
| 81.60 | 2.24 | 1.1799 | 2 | 1 | 10 | 12 |
| 83.84 | 1.57 | 1.1539 | 1 | 3 | 4 | 12 |

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Portlandite
_database_code_amcsd 0009875

CELL PARAMETERS: 3.5890 3.5890 4.9110 90.000 90.000 120.000
SPACE GROUP: P-3m1
X-RAY WAVELENGTH: 1.541838
Cell Volume: 54.783
Density (g/cm3): 2.246
MAX. ABS. INTENSITY / VOLUME**2: 25.25582270
RIR: 3.662
RIR based on corundum from Acta Crystallographica A38 (1982) 733-739

| 2-THETA | INTENSITY | D-SPACING | H | K | L | Multiplicity |
|---|---|---|---|---|---|---|
| 18.06 | 61.35 | 4.9110 | 0 | 0 | 1 | 2 |
| 28.72 | 16.95 | 3.1082 | 1 | 0 | 0 | 6 |
| 34.14 | 97.31 | 2.6264 | 1 | 0 | 1 | 6 |
| 34.14 | 2.69 | 2.6264 | 0 | 1 | 1 | 6 |
| 47.17 | 25.54 | 1.9268 | 1 | 0 | 2 | 6 |
| 47.17 | 19.26 | 1.9268 | 0 | 1 | 2 | 6 |
| 50.88 | 29.44 | 1.7945 | 1 | 1 | 0 | 6 |
| 54.44 | 16.27 | 1.6855 | 1 | 1 | 1 | 12 |
| 56.19 | 1.40 | 1.6370 | 0 | 0 | 3 | 2 |
| 59.48 | 2.31 | 1.5541 | 2 | 0 | 0 | 6 |
| 62.71 | 11.29 | 1.4817 | 0 | 2 | 1 | 6 |
| 64.29 | 1.17 | 1.4488 | 1 | 1 | 2 | 12 |
| 64.32 | 1.42 | 1.4484 | 1 | 0 | 3 | 6 |
| 64.32 | 11.11 | 1.4484 | 0 | 1 | 3 | 6 |
| 71.90 | 4.22 | 1.3132 | 2 | 0 | 2 | 6 |
| 71.90 | 5.63 | 1.3132 | 0 | 2 | 2 | 6 |
| 77.79 | 1.83 | 1.2277 | 0 | 0 | 4 | 2 |
| 79.20 | 2.84 | 1.2094 | 1 | 1 | 3 | 12 |
| 84.87 | 2.64 | 1.1425 | 2 | 1 | 1 | 12 |
| 84.93 | 1.57 | 1.1419 | 0 | 1 | 4 | 6 |
| 86.31 | 4.16 | 1.1271 | 2 | 0 | 3 | 6 |

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## FICHAS POSIBLES (3 y 4):

Ankerite
_database_code_amcsd 0001273

CELL PARAMETERS: 4.8240 4.8240 16.1217 90.000 90.000 120.000
SPACE GROUP: R-3
X-RAY WAVELENGTH: 1.541838
Cell Volume: 324.905
Density (g/cm3): 3.091
MAX. ABS. INTENSITY / VOLUME**2: 29.35603608
RIR: 3.093
RIR based on corundum from Acta Crystallographica A38 (1982) 733-739

| 2-THETA | INTENSITY | D-SPACING | H | K | L | Multiplicity |
|---|---|---|---|---|---|---|
| 16.50 | 1.91 | 5.3739 | 0 | 0 | 3 | 2 |
| 23.99 | 13.10 | 3.7092 | 0 | 1 | 2 | 6 |
| 30.83 | 100.00 | 2.9006 | 1 | 0 | 4 | 6 |
| 33.35 | 1.55 | 2.6869 | 0 | 0 | 6 | 2 |
| 37.28 | 14.90 | 2.4120 | 1 | 1 | 0 | 6 |
| 41.02 | 10.32 | 2.2005 | 2 | -1 | 3 | 6 |
| 41.02 | 9.67 | 2.2005 | 1 | 1 | 3 | 6 |
| 44.82 | 16.09 | 2.0221 | 2 | 0 | 2 | 6 |
| 49.12 | 6.67 | 1.8546 | 0 | 2 | 4 | 6 |
| 50.27 | 18.32 | 1.8151 | 0 | 1 | 8 | 6 |
| 50.87 | 9.11 | 1.7949 | 1 | 1 | 6 | 6 |
| 50.87 | 14.70 | 1.7949 | 2 | -1 | 6 | 6 |
| 58.75 | 2.28 | 1.5715 | 2 | 1 | 1 | 6 |
| 59.67 | 6.95 | 1.5496 | -1 | 3 | 2 | 6 |
| 59.67 | 3.57 | 1.5496 | 1 | 2 | 2 | 6 |
| 61.67 | 1.34 | 1.5041 | 1 | 0 | 10 | 6 |
| 63.25 | 4.26 | 1.4702 | 3 | -1 | 4 | 6 |
| 63.25 | 3.30 | 1.4702 | 2 | 1 | 4 | 6 |
| 64.22 | 3.31 | 1.4503 | 2 | 0 | 8 | 6 |
| 64.83 | 1.69 | 1.4381 | 1 | 1 | 9 | 6 |
| 64.83 | 1.00 | 1.4381 | 2 | -1 | 9 | 6 |
| 67.23 | 8.02 | 1.3926 | 3 | 0 | 0 | 6 |
| 70.03 | 3.53 | 1.3435 | 0 | 0 | 12 | 2 |
| 74.32 | 2.94 | 1.2763 | 0 | 2 | 10 | 6 |
| 76.67 | 1.02 | 1.2429 | -1 | 3 | 8 | 6 |
| 76.67 | 2.85 | 1.2429 | 1 | 2 | 8 | 6 |
| 82.12 | 1.90 | 1.1737 | 1 | 1 | 12 | 6 |
| 86.22 | 1.21 | 1.1281 | 2 | 1 | 10 | 6 |
| 86.22 | 1.41 | 1.1281 | 3 | -1 | 10 | 6 |
| 87.62 | 1.37 | 1.1136 | 1 | 3 | 4 | 6 |

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Dolomite
_database_code_amcsd 0000086

CELL PARAMETERS: 4.8150 4.8150 16.1190 90.000 90.000 120.000
SPACE GROUP: R-3
X-RAY WAVELENGTH: 1.541838
Cell Volume: 323.639
Density (g/cm3): 2.838
MAX. ABS. INTENSITY / VOLUME**2: 24.14536753
RIR: 2.770
RIR based on corundum from Acta Crystallographica A38 (1982) 733-739

| 2-THETA | INTENSITY | D-SPACING | H | K | L | Multiplicity |
|---|---|---|---|---|---|---|
| 22.02 | 2.05 | 4.0370 | 1 | 0 | 1 | 6 |
| 24.03 | 2.21 | 3.7036 | 0 | 1 | 2 | 6 |
| 30.86 | 100.00 | 2.8977 | 1 | 0 | 4 | 6 |
| 33.35 | 4.60 | 2.6865 | 0 | 0 | 6 | 2 |
| 35.19 | 5.19 | 2.5505 | 0 | 1 | 5 | 6 |
| 37.35 | 11.44 | 2.4075 | 1 | 1 | 0 | 6 |
| 41.08 | 17.99 | 2.1970 | 1 | 1 | 3 | 6 |
| 41.08 | 5.68 | 2.1970 | 2 | -1 | 3 | 6 |
| 43.78 | 4.13 | 2.0677 | 0 | 2 | 1 | 6 |
| 44.91 | 13.87 | 2.0185 | 2 | 0 | 2 | 6 |
| 49.20 | 4.36 | 1.8518 | 0 | 2 | 4 | 6 |
| 50.29 | 18.07 | 1.8142 | 0 | 1 | 8 | 6 |
| 50.93 | 6.78 | 1.7929 | 1 | 1 | 6 | 6 |
| 50.93 | 12.85 | 1.7929 | 2 | -1 | 6 | 6 |
| 50.99 | 1.24 | 1.7910 | 0 | 0 | 9 | 2 |
| 58.87 | 2.51 | 1.5686 | 3 | -1 | 1 | 6 |
| 58.87 | 1.93 | 1.5686 | 2 | 1 | 1 | 6 |
| 59.79 | 3.00 | 1.5468 | 1 | 2 | 2 | 6 |
| 59.79 | 6.96 | 1.5468 | -1 | 3 | 2 | 6 |
| 63.37 | 3.19 | 1.4678 | 3 | -1 | 4 | 6 |
| 63.37 | 2.44 | 1.4678 | 2 | 1 | 4 | 6 |
| 64.29 | 2.68 | 1.4489 | 2 | 0 | 8 | 6 |
| 64.89 | 2.24 | 1.4370 | 2 | -1 | 9 | 6 |
| 65.98 | 2.73 | 1.4159 | -1 | 3 | 5 | 6 |
| 67.37 | 6.67 | 1.3900 | 3 | 0 | 0 | 6 |
| 70.05 | 3.72 | 1.3432 | 0 | 0 | 12 | 2 |
| 72.70 | 2.17 | 1.3006 | 2 | 1 | 7 | 6 |
| 74.39 | 2.92 | 1.2752 | 0 | 2 | 10 | 6 |
| 76.78 | 2.53 | 1.2414 | 1 | 2 | 8 | 6 |
| 82.17 | 1.87 | 1.1730 | 1 | 1 | 12 | 6 |
| 86.33 | 1.01 | 1.1269 | 2 | 1 | 10 | 6 |
| 86.33 | 1.43 | 1.1269 | 3 | -1 | 10 | 6 |
| 87.81 | 1.52 | 1.1116 | 1 | 3 | 4 | 6 |

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**<u>Caso práctico 3</u>.**

**Identificación de filosilicatos en arcillas.**

Las arcillas son materiales muy estudiados en geología, ciencias del suelo, ciencias de materiales, etc. Presentan como constituyentes principales filosilicatos, cuya identificación en difracción de rayos X de polvo, utilizando únicamente registros de difracción de la muestra de polvo desorientado no permite distinguir los diferentes grupos de filosilicatos al ser los difractogramas de polvo muy parecidos. Para la determinación de los filosilicatos se recurre a difractogramas de muestras orientadas obtenidas a partir de suspensiones acuosas de la fracción fina del material sometidas a diversos tratamientos, al menos tres: secada a temperatura ambiente (N), calentada a 550 ºC durante 2 horas (Q), y tratada con etilenglicol durante 12 horas (E).

**Ejercicio:**

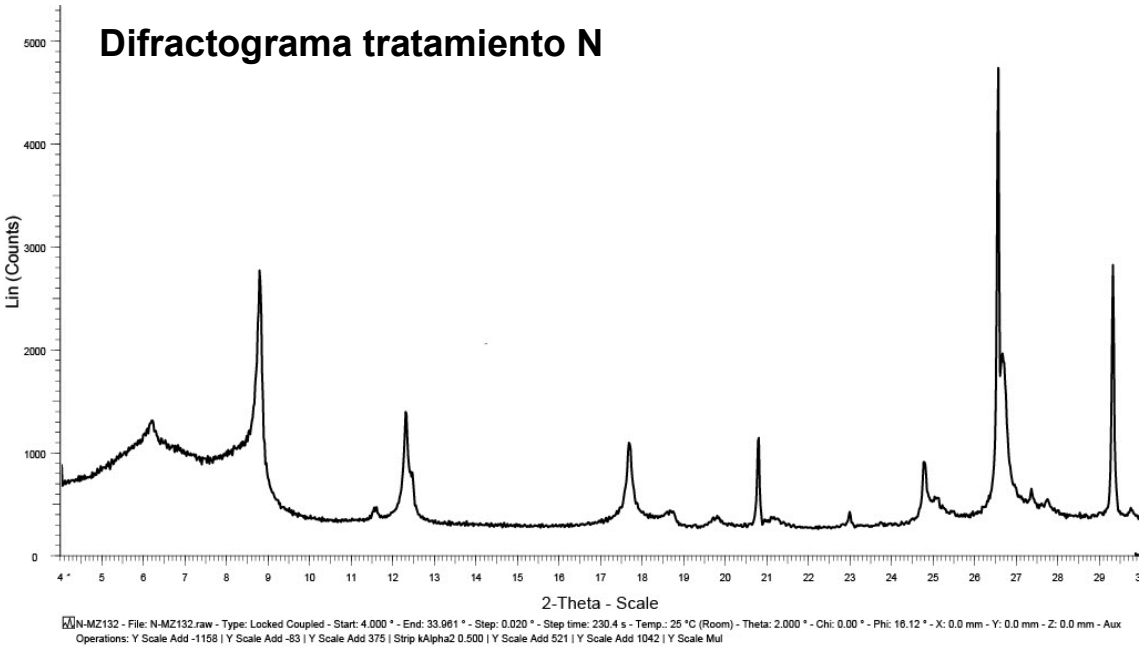
Se presentan difractogramas de la muestras MZ132, con los citados tratamientos N, Q y E. Y se aportan fichas de la base de datos AMSD (American Mineralogical Society).

1) Se trata de medir los espaciados en el intervalo angular 2 theta entre 4 y 22º, e identificar en dicho intervalo los constituyentes entre los minerales cuyas fichas se aportan (*6 puntos*)

2) Se trata de identificar los filosilicatos presentes entre las fichas aportadas de illita, caolinita, clorita, montmorillonita y vermiculita. La muestra contiene además otros constituyentes como cuarzo, yeso, calcita y otros (*3 puntos*).

3) En su caso, proponer algún criterio adicional para una identificación más precisa (*1 punto*).

Los registros corresponden a radiación Cu $K\alpha_1$, tras eliminación del componente $K\alpha_2$. Con λ Cu $K\alpha_1$ = 1,5406 Å.



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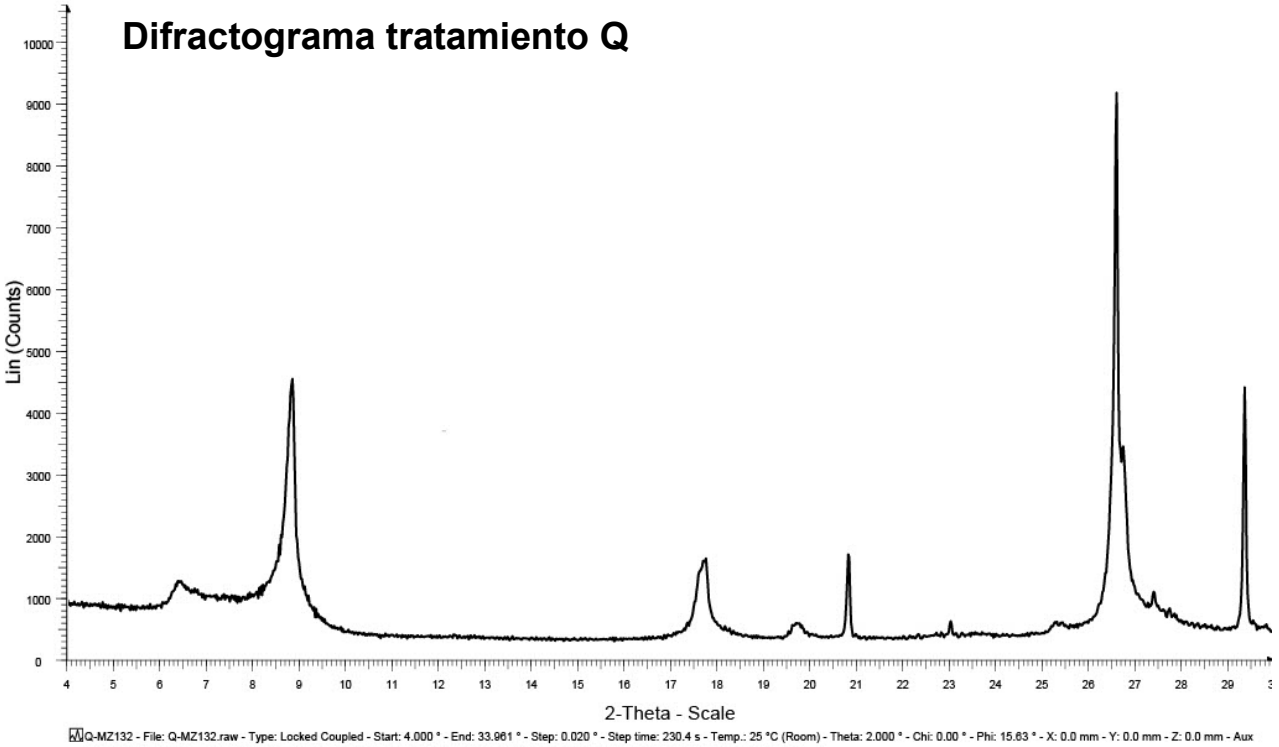
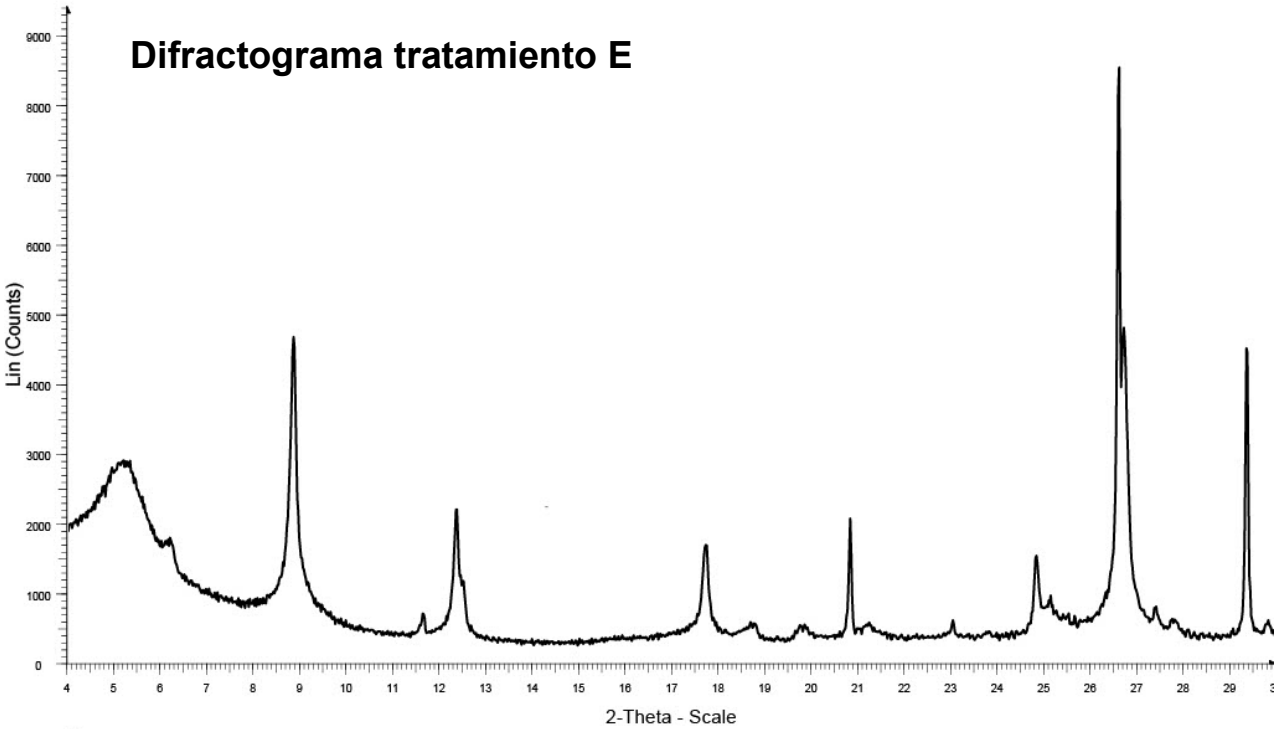
## Difractograma tratamiento Q




Q-MZ132 - File: Q-MZ132.raw - Type: Locked Coupled - Start: 4.000 ° - End: 33.961 ° - Step: 0.020 ° - Step time: 230.4 s - Temp.: 25 °C (Room) - Theta: 2.000 ° - Chi: 0.00 ° - Phi: 15.63 ° - X: 0.0 mm - Y: 0.0 mm - Z: 0.0 mm - Aux
Operations: X Offset -0.120 | X Offset -0.120 | Y Scale Add 1 | Y Scale Add -3125 | Y Scale Add 417 | Y Scale Add -1000 | Y Scale Add 2896

## Difractograma tratamiento E




EMZ132 - File: EMZ132.raw - Type: Locked Coupled - Start: 3.000 ° - End: 33.964 ° - Step: 0.020 ° - Step time: 230.4 s - Temp.: 25 °C (Room) - Theta: 1.500 ° - Chi: 0.00 ° - Phi: 15.63 ° - X: 0.0 mm - Y: 0.0 mm - Z: 0.0 mm - Aux1:
Operations: Y Scale Add 1 | Y Scale Add -7288 | Y Scale Add 1292 | Y Scale Mul 0.833 | X Offset -0.009 | X Offset 0.000 | Y Scale Add 521



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Kaolinite
Gruner W
Zeitschrift fur Kristallographie 83 (1932) 75-88
The crystal structure of kaolinite
_cod_database_code 1011045
_database_code_amcsd 0017947

CELL PARAMETERS: 5.1480 8.9200 14.5350 90.000 100.200 90.000
SPACE GROUP: Cc
X-RAY WAVELENGTH: 1.541838
Cell Volume: 656.901
Density (g/cm3): 2.569
MAX. ABS. INTENSITY / VOLUME**2: 8.502879376
RIR: 1.078
RIR based on corundum from Acta Crystallographica A38 (1982) 733-739

| 2-THETA | INTENSITY | D-SPACING | H | K | L | Multiplicity |
|---|---|---|---|---|---|---|
| 12.37 | 100.00 | 7.1526 | 0 | 0 | 2 | 1 |
| 20.16 | 19.70 | 4.4055 | 1 | 1 | 0 | 2 |
| 20.16 | 28.35 | 4.4055 | -1 | 1 | 1 | 2 |
| 20.86 | 25.36 | 4.2579 | 0 | 2 | 1 | 2 |
| 22.01 | 21.61 | 4.0391 | 1 | 1 | 1 | 2 |
| 22.01 | 28.36 | 4.0391 | -1 | 1 | 2 | 2 |
| 23.51 | 26.35 | 3.7845 | 0 | 2 | 2 | 2 |
| 24.90 | 56.10 | 3.5763 | 0 | 0 | 4 | 1 |
| 25.32 | 6.20 | 3.5171 | 1 | 1 | 2 | 2 |
| 25.32 | 13.95 | 3.5171 | -1 | 1 | 3 | 2 |
| 27.38 | 10.50 | 3.2573 | 0 | 2 | 3 | 2 |
| 29.65 | 7.83 | 3.0129 | 1 | 1 | 3 | 2 |
| 32.08 | 3.55 | 2.7901 | 0 | 2 | 4 | 2 |
| 34.65 | 7.73 | 2.5884 | 1 | 1 | 4 | 2 |
| 34.66 | 4.38 | 2.5884 | -1 | 1 | 5 | 2 |
| 34.99 | 8.86 | 2.5644 | 1 | 3 | 0 | 2 |
| 35.43 | 9.01 | 2.5333 | 2 | 0 | 0 | 1 |
| 35.43 | 12.10 | 2.5333 | -2 | 0 | 2 | 1 |
| 36.13 | 20.53 | 2.4857 | -1 | 3 | 2 | 2 |
| 37.34 | 3.11 | 2.4082 | 0 | 2 | 5 | 2 |
| 37.73 | 10.27 | 2.3842 | 0 | 0 | 6 | 1 |
| 38.34 | 67.42 | 2.3480 | 1 | 3 | 2 | 2 |
| 39.80 | 1.92 | 2.2651 | 2 | 0 | 2 | 1 |
| 39.80 | 29.09 | 2.2651 | -2 | 0 | 4 | 1 |
| 40.14 | 2.22 | 2.2467 | 1 | 1 | 5 | 2 |
| 40.14 | 4.82 | 2.2466 | -1 | 1 | 6 | 2 |
| 40.45 | 1.50 | 2.2300 | 0 | 4 | 0 | 2 |
| 40.97 | 1.06 | 2.2028 | 2 | 2 | 0 | 2 |
| 40.97 | 3.19 | 2.2028 | -2 | 2 | 2 | 2 |
| 41.45 | 1.87 | 2.1785 | -1 | 3 | 4 | 2 |
| 42.47 | 1.16 | 2.1284 | 2 | 2 | 1 | 2 |
| 43.02 | 1.54 | 2.1026 | 0 | 2 | 6 | 2 |
| 44.87 | 1.36 | 2.0200 | 0 | 4 | 3 | 2 |
| 44.88 | 2.92 | 2.0195 | 2 | 2 | 2 | 2 |
| 45.33 | 14.76 | 2.0008 | 1 | 3 | 4 | 2 |
| 45.99 | 1.20 | 1.9735 | -1 | 1 | 7 | 2 |
| 47.52 | 3.54 | 1.9136 | 2 | 0 | 4 | 1 |
| 47.52 | 6.37 | 1.9136 | -2 | 0 | 6 | 1 |



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Illite
Drits V A, Zviagina B B, McCarty D K, Salyn A L
American Mineralogist 95 (2010) 348-361
Factors responsible for crystal-chemical variations in the solid solutions from illite to aluminoceladonite and from glauconite to celadonite
Locality: Silver caldera, San Juan Mountains, Colorado
Sample Name: RM30
_database_code_amcsd 0005015

CELL PARAMETERS: 5.2021 8.9797 10.2260 90.000 101.570 90.000
SPACE GROUP: C2/m
X-RAY WAVELENGTH: 1.541838
Cell Volume: 467.984
Density (g/cm3): 2.820
MAX. ABS. INTENSITY / VOLUME**2: 7.451790421
RIR: 0.860
RIR based on corundum from Acta Crystallographica A38 (1982) 733-739

| 2-THETA | INTENSITY | D-SPACING | H | K | L | Multiplicity |
|---|---|---|---|---|---|---|
| 8.83 | 39.54 | 10.0182 | 0 | 0 | 1 | 2 |
| 17.71 | 17.67 | 5.0091 | 0 | 0 | 2 | 2 |
| 19.77 | 50.68 | 4.4899 | 0 | 2 | 0 | 2 |
| 20.45 | 27.88 | 4.3433 | -1 | 1 | 1 | 4 |
| 21.69 | 21.03 | 4.0972 | 0 | 2 | 1 | 4 |
| 23.32 | 3.23 | 3.8146 | 1 | 1 | 1 | 4 |
| 24.38 | 100.00 | 3.6504 | -1 | 1 | 2 | 4 |
| 26.66 | 30.93 | 3.3434 | 0 | 2 | 2 | 4 |
| 26.69 | 30.40 | 3.3394 | 0 | 0 | 3 | 2 |
| 29.14 | 81.94 | 3.0647 | 1 | 1 | 2 | 4 |
| 30.58 | 20.47 | 2.9234 | -1 | 1 | 3 | 4 |
| 33.44 | 29.99 | 2.6795 | 0 | 2 | 3 | 4 |
| 34.54 | 9.91 | 2.5971 | -2 | 0 | 1 | 2 |
| 34.76 | 22.12 | 2.5810 | 1 | 3 | 0 | 4 |
| 35.01 | 48.96 | 2.5631 | -1 | 3 | 1 | 4 |
| 35.22 | 22.25 | 2.5482 | 2 | 0 | 0 | 2 |
| 35.85 | 5.52 | 2.5046 | 0 | 0 | 4 | 2 |
| 36.20 | 6.38 | 2.4812 | -2 | 0 | 2 | 2 |
| 36.40 | 2.33 | 2.4682 | 1 | 1 | 3 | 4 |
| 36.83 | 11.95 | 2.4402 | 1 | 3 | 1 | 4 |
| 37.55 | 14.50 | 2.3955 | -1 | 3 | 2 | 4 |
| 38.06 | 1.55 | 2.3643 | -1 | 1 | 4 | 4 |
| 38.15 | 11.08 | 2.3591 | 2 | 0 | 1 | 2 |
| 39.97 | 1.64 | 2.2557 | -2 | 0 | 3 | 2 |
| 40.11 | 2.74 | 2.2481 | -2 | 2 | 1 | 4 |
| 40.17 | 5.13 | 2.2449 | 0 | 4 | 0 | 2 |
| 40.71 | 7.21 | 2.2162 | 2 | 2 | 0 | 4 |
| 40.93 | 2.55 | 2.2050 | 1 | 3 | 2 | 4 |
| 41.21 | 6.24 | 2.1906 | 0 | 4 | 1 | 4 |
| 41.59 | 4.82 | 2.1717 | -2 | 2 | 2 | 4 |
| 42.01 | 19.75 | 2.1506 | -1 | 3 | 3 | 4 |
| 42.93 | 9.94 | 2.1068 | 2 | 0 | 2 | 2 |
| 43.33 | 8.38 | 2.0884 | 2 | 2 | 1 | 4 |
| 44.97 | 4.33 | 2.0156 | -2 | 2 | 3 | 4 |
| 45.26 | 17.62 | 2.0036 | 0 | 0 | 5 | 2 |
| 45.40 | 7.37 | 1.9977 | -2 | 0 | 4 | 2 |

Chlorite
Lister J S, Bailey S W
American Mineralogist 52 (1967) 1614-1631
Chlorite polytypism: IV. Regular two-layer structures
refined structure
_database_code_amcsd 0000162

CELL PARAMETERS: 5.3350 9.2400 28.7350 90.000 90.000 90.000
SPACE GROUP: C1
X-RAY WAVELENGTH: 1.541838
Cell Volume: 1416.503
Density (g/cm3): 2.561
MAX. ABS. INTENSITY / VOLUME**2: 7.069927399
RIR: 0.899
RIR based on corundum from Acta Crystallographica A38 (1982) 733-739

| 2-THETA | INTENSITY | D-SPACING | H | K | L | Multiplicity |
|---|---|---|---|---|---|---|
| 6.15 | 100.00 | 14.3675 | 0 | 0 | 2 | 1 |
| 12.32 | 71.11 | 7.1838 | 0 | 0 | 4 | 1 |
| 18.53 | 65.38 | 4.7892 | 0 | 0 | 6 | 1 |
| 19.21 | 8.64 | 4.6202 | 1 | 1 | 0 | 1 |
| 19.21 | 9.94 | 4.6202 | -1 | 1 | 0 | 1 |
| 19.21 | 8.01 | 4.6200 | 0 | 2 | 0 | 1 |
| 19.46 | 5.17 | 4.5616 | 1 | -1 | 1 | 1 |
| 19.46 | 3.75 | 4.5616 | 1 | 1 | 1 | 1 |
| 19.46 | 3.99 | 4.5616 | -1 | -1 | 1 | 1 |
| 19.46 | 3.56 | 4.5616 | -1 | 1 | 1 | 1 |
| 20.19 | 1.16 | 4.3984 | 1 | -1 | 2 | 1 |
| 20.19 | 7.74 | 4.3984 | 1 | 1 | 2 | 1 |
| 20.19 | 1.50 | 4.3984 | -1 | -1 | 2 | 1 |
| 20.19 | 6.24 | 4.3984 | -1 | 1 | 2 | 1 |
| 20.19 | 6.07 | 4.3982 | 0 | -2 | 2 | 1 |
| 20.19 | 16.20 | 4.3982 | 0 | 2 | 2 | 1 |
| 21.35 | 2.82 | 4.1614 | 1 | -1 | 3 | 1 |
| 21.35 | 2.54 | 4.1614 | 1 | 1 | 3 | 1 |
| 21.35 | 2.77 | 4.1614 | -1 | -1 | 3 | 1 |
| 21.35 | 3.22 | 4.1614 | -1 | 1 | 3 | 1 |
| 22.89 | 7.22 | 3.8859 | 1 | -1 | 4 | 1 |
| 22.89 | 2.39 | 3.8859 | 1 | 1 | 4 | 1 |
| 22.89 | 8.16 | 3.8859 | -1 | -1 | 4 | 1 |
| 22.89 | 2.24 | 3.8859 | -1 | 1 | 4 | 1 |
| 22.89 | 10.97 | 3.8858 | 0 | -2 | 4 | 1 |
| 24.72 | 1.65 | 3.6008 | 1 | -1 | 5 | 1 |
| 24.72 | 1.98 | 3.6008 | 1 | 1 | 5 | 1 |
| 24.72 | 2.20 | 3.6008 | -1 | -1 | 5 | 1 |
| 24.72 | 1.80 | 3.6008 | -1 | 1 | 5 | 1 |
| 24.79 | 59.24 | 3.5919 | 0 | 0 | 8 | 1 |
| 26.81 | 3.62 | 3.3251 | 1 | 1 | 6 | 1 |
| 26.81 | 3.62 | 3.3251 | -1 | 1 | 6 | 1 |
| 26.81 | 7.31 | 3.3250 | 0 | 2 | 6 | 1 |
| 29.10 | 1.61 | 3.0687 | 1 | -1 | 7 | 1 |
| 29.10 | 1.72 | 3.0687 | 1 | 1 | 7 | 1 |
| 29.10 | 1.35 | 3.0687 | -1 | 1 | 7 | 1 |
| 31.12 | 17.25 | 2.8735 | 0 | 0 | 10 | 1 |
| 31.55 | 1.93 | 2.8357 | 1 | -1 | 8 | 1 |

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Montmorillonite
Viani A, Gualtieri A, Artioli G
American Mineralogist 87 (2002) 966-975
The nature of disorder in montmorillonite by simulation of X-ray powder patterns
Note: Structural simulation model
_database_code_amcsd 0002868

CELL PARAMETERS: 5.1800 8.9800 15.0000 90.000 90.000 90.000
SPACE GROUP: P1
X-RAY WAVELENGTH: 1.541838
Cell Volume: 697.746
Density (g/cm3): 1.801
MAX. ABS. INTENSITY / VOLUME**2: 103.9485592
RIR: 18.798
RIR based on corundum from Acta Crystallographica A38 (1982) 733-739

| 2-THETA | INTENSITY | D-SPACING | H | K | L | Multiplicity |
|---|---|---|---|---|---|---|
| 5.89 | 100.00 | 15.0000 | 0 | 0 | 1 | 1 |
| 17.74 | 2.93 | 5.0000 | 0 | 0 | 3 | 1 |
| 19.77 | 2.29 | 4.4900 | 0 | 2 | 0 | 1 |
| 20.65 | 1.28 | 4.3014 | 0 | -2 | 1 | 1 |
| 20.65 | 1.28 | 4.3014 | 0 | 2 | 1 | 1 |
| 20.66 | 1.36 | 4.2988 | -1 | -1 | 1 | 1 |
| 20.66 | 1.37 | 4.2988 | -1 | 1 | 1 | 1 |
| 23.10 | 1.71 | 3.8505 | -1 | -1 | 2 | 1 |
| 23.10 | 1.71 | 3.8505 | -1 | 1 | 2 | 1 |
| 29.78 | 1.07 | 3.0000 | 0 | 0 | 5 | 1 |

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For reference, see Downs et al. (1993) American Mineralogist 78, 1104-1107.

Vermiculite
Gruner J W
American Mineralogist 19 (1934) 557-575
The structures of vermiculites and their collapse by dehydration
Locality: Structure results from data of many samples
_database_code_amcsd 0000012

CELL PARAMETERS: 5.3100 9.2000 28.4600 90.000 97.140 90.000
SPACE GROUP: Cc
X-RAY WAVELENGTH: 1.541838
Cell Volume: 1379.546
Density (g/cm3): 1.816
MAX. ABS. INTENSITY / VOLUME**2: 120.7170254
RIR: 21.644
RIR based on corundum from Acta Crystallographica A38 (1982) 733-739

| 2-THETA | INTENSITY | D-SPACING | H | K | L | Multiplicity |
|---|---|---|---|---|---|---|
| 6.26 | 100.00 | 14.1197 | 0 | 0 | 2 | 1 |
| 12.54 | 1.13 | 7.0598 | 0 | 0 | 4 | 1 |
| 19.33 | 1.82 | 4.5922 | -1 | 1 | 1 | 2 |
| 21.50 | 1.30 | 4.1329 | 0 | 2 | 3 | 2 |
| 22.52 | 1.19 | 3.9487 | 1 | 1 | 3 | 2 |
| 25.23 | 1.27 | 3.5299 | 0 | 0 | 8 | 1 |
| 31.69 | 1.10 | 2.8239 | 0 | 0 | 10 | 1 |
| 35.42 | 2.06 | 2.5340 | -1 | 3 | 4 | 2 |
| 35.43 | 1.01 | 2.5336 | 2 | 0 | 2 | 1 |
| 37.90 | 2.06 | 2.3737 | -1 | 3 | 6 | 2 |
| 37.91 | 1.00 | 2.3734 | 2 | 0 | 4 | 1 |

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For reference, see Downs et al. (1993) American Mineralogist 78, 1104-1107.



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Quartz
Glinnemann J, King H E, Schulz H, Hahn T, La Placa S J, Dacol F
Zeitschrift fur Kristallographie 198 (1992) 177-212
Crystal structures of the low-temperature quartz-type phases of SiO2 and GeO2 at elevated pressure
P = 0.0 GPa
_database_code_amcsd 0011007

CELL PARAMETERS: 4.9210 4.9210 5.4163 90.000 90.000 120.000
SPACE GROUP: P3_121
X-RAY WAVELENGTH: 1.541838
Cell Volume: 113.590
Density (g/cm3): 2.635
MAX. ABS. INTENSITY / VOLUME**2: 35.49608771
RIR: 4.387
RIR based on corundum from Acta Crystallographica A38 (1982) 733-739

| 2-THETA | INTENSITY | D-SPACING | H | K | L | Multiplicity |
|---|---|---|---|---|---|---|
| 20.84 | 20.26 | 4.2617 | 1 | 0 | 0 | 6 |
| 26.61 | 30.19 | 3.3492 | 0 | 1 | 1 | 6 |
| 26.61 | 69.81 | 3.3492 | 1 | 0 | 1 | 6 |
| 36.52 | 6.99 | 2.4605 | 1 | 1 | 0 | 3 |
| 39.42 | 6.17 | 2.2857 | 0 | 1 | 2 | 6 |
| 40.26 | 3.28 | 2.2402 | 1 | 1 | 1 | 6 |
| 42.42 | 5.36 | 2.1309 | 2 | 0 | 0 | 6 |
| 45.76 | 2.22 | 1.9829 | 0 | 2 | 1 | 6 |
| 45.76 | 1.05 | 1.9829 | 2 | 0 | 1 | 6 |
| 50.09 | 12.89 | 1.8211 | 1 | 1 | 2 | 6 |
| 54.82 | 3.27 | 1.6746 | 2 | 0 | 2 | 6 |
| 55.26 | 1.66 | 1.6624 | 1 | 0 | 3 | 6 |
| 59.91 | 5.05 | 1.5439 | 2 | 1 | 1 | 6 |
| 59.91 | 4.24 | 1.5439 | 1 | 2 | 1 | 6 |
| 63.96 | 1.81 | 1.4556 | 1 | 1 | 3 | 6 |
| 67.68 | 1.48 | 1.3844 | 1 | 2 | 2 | 6 |
| 67.68 | 4.44 | 1.3844 | 2 | 1 | 2 | 6 |
| 68.06 | 5.58 | 1.3775 | 0 | 2 | 3 | 6 |
| 68.06 | 1.46 | 1.3775 | 2 | 0 | 3 | 6 |
| 68.26 | 4.38 | 1.3741 | 3 | 0 | 1 | 6 |
| 73.36 | 1.83 | 1.2905 | 0 | 1 | 4 | 6 |
| 75.59 | 2.03 | 1.2580 | 0 | 3 | 2 | 6 |
| 79.79 | 2.51 | 1.2019 | 1 | 2 | 3 | 6 |
| 81.06 | 2.63 | 1.1863 | 1 | 1 | 4 | 6 |

Gypsum
Boeyens J C A, Ichharam V V H
Zeitschrift fur Kristallographie 217 (2002) 9-10
Redetermination of the crystal structure of calcium sulphate dihydrate, CaSO4*2H2O
Locality: synthetic
_database_code_amcsd 0011093

CELL PARAMETERS: 6.2840 15.2000 6.5230 90.000 127.410 90.000
SPACE GROUP: C2/c
X-RAY WAVELENGTH: 1.541838
Cell Volume: 494.899
Density (g/cm3): 2.310
MAX. ABS. INTENSITY / VOLUME**2: 12.00207308
RIR: 1.691
RIR based on corundum from Acta Crystallographica A38 (1982) 733-739

| 2-THETA | INTENSITY | D-SPACING | H | K | L | Multiplicity |
|---|---|---|---|---|---|---|
| 11.64 | 77.51 | 7.6000 | 0 | 2 | 0 | 2 |
| 18.71 | 1.41 | 4.7423 | 1 | 1 | 0 | 4 |
| 20.75 | 100.00 | 4.2811 | 0 | 2 | 1 | 4 |
| 23.41 | 10.56 | 3.8000 | 0 | 4 | 0 | 2 |
| 23.43 | 2.59 | 3.7970 | -1 | 3 | 1 | 4 |
| 28.15 | 4.09 | 3.1702 | -1 | 1 | 2 | 4 |
| 29.14 | 71.18 | 3.0642 | 0 | 4 | 1 | 4 |
| 31.13 | 47.99 | 2.8730 | -2 | 2 | 1 | 4 |
| 31.19 | 3.09 | 2.8674 | -2 | 0 | 2 | 2 |
| 32.11 | 9.31 | 2.7875 | 1 | 1 | 1 | 4 |
| 32.80 | 1.09 | 2.7305 | -1 | 3 | 2 | 4 |
| 33.40 | 35.89 | 2.6828 | -2 | 2 | 2 | 4 |
| 34.55 | 3.75 | 2.5964 | 1 | 5 | 0 | 4 |
| 34.62 | 2.12 | 2.5906 | 0 | 0 | 2 | 2 |
| 35.99 | 11.05 | 2.4957 | 2 | 0 | 0 | 2 |
| 36.31 | 1.15 | 2.4744 | 1 | 3 | 1 | 4 |
| 36.65 | 6.87 | 2.4521 | 0 | 2 | 2 | 4 |
| 37.41 | 3.76 | 2.4036 | -2 | 4 | 1 | 4 |
| 40.69 | 13.18 | 2.2174 | -1 | 5 | 2 | 4 |
| 42.22 | 1.79 | 2.1405 | 0 | 4 | 2 | 4 |
| 43.38 | 15.19 | 2.0860 | 2 | 4 | 0 | 4 |
| 43.52 | 4.40 | 2.0796 | -2 | 2 | 3 | 4 |
| 43.65 | 6.20 | 2.0736 | 1 | 5 | 1 | 4 |
| 43.66 | 5.57 | 2.0731 | -3 | 1 | 2 | 4 |
| 44.26 | 4.65 | 2.0465 | -1 | 1 | 3 | 4 |
| 45.56 | 3.03 | 1.9912 | 1 | 7 | 0 | 4 |
| 46.47 | 2.91 | 1.9541 | -3 | 1 | 1 | 4 |
| 47.88 | 1.15 | 1.9000 | 0 | 8 | 0 | 2 |
| 47.92 | 13.69 | 1.8985 | -2 | 6 | 2 | 4 |
| 48.44 | 12.46 | 1.8792 | -2 | 4 | 3 | 4 |
| 48.83 | 2.77 | 1.8652 | 1 | 1 | 2 | 4 |
| 50.38 | 12.68 | 1.8113 | 0 | 6 | 2 | 4 |
| 50.76 | 5.59 | 1.7987 | 2 | 2 | 1 | 4 |
| 51.07 | 1.18 | 1.7885 | -3 | 3 | 3 | 4 |
| 51.21 | 2.03 | 1.7838 | 0 | 8 | 1 | 4 |
| 51.39 | 9.81 | 1.7779 | 2 | 6 | 0 | 4 |
| 53.65 | 1.03 | 1.7083 | -1 | 5 | 3 | 4 |

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