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We have used a combination of advanced transmission electron microscopy techniques to study the physical and chemical properties of intracellular ferrimagnetic magnetite (Fe<sub>3</sub>O<sub>4</sub>) crystals inside magnetotactic bacteria collected from lakes and streams.

The orientations and morphologies of the crystals in a double magnetosome chain were identified using electron diffraction, high-resolution electron microscopy and high-angle annular dark field electron tomography. The chain is analogous to beads on a string, in which biological control appears to be stricter in setting the [111] magnetocrystalline easy axis of the crystals to be parallel to the chain axis than in constraining their orientation about this direction. We have used off-axis electron holography to record magnetic induction maps from the same particles. The magnetic signal is dominated by inter-particle interactions and by the shapes of the individual crystals.

We have also studied the diversity of magnetosomes in bacteria collected from Lake Balaton. In stained thin sections of cocci, magnetite crystals appear to be anchored to the inner cell membrane. They are enveloped by stained material, apparently representing the magnetosome membrane. The cells do not contain detectable iron outside the magnetite magnetosomes.

**Keywords:** biomineral, magnetism, advanced electron microscopy

#### P.10.05.19

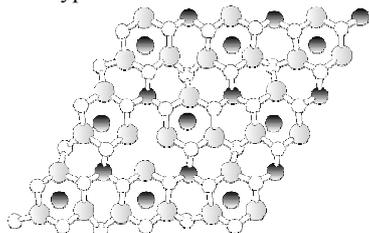
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##### Going Inside Fettelite, a Hg-Sulfosalt Mineral

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Fettelite's name honours Mr. M Fettel who found it. The empirical formula from microprobe analysis (based on 24 Ag atoms) is Ag<sub>24</sub>HgCu<sub>0.04</sub>Fe<sub>0.03</sub>Tl<sub>0.02</sub>Pb<sub>0.01</sub>Sb<sub>0.07</sub>As<sub>5.01</sub>S<sub>20.07</sub>, ideally Ag<sub>24</sub>HgAs<sub>5</sub>S<sub>20</sub>, and the crystal system trigonal [1].

Despite Fettelite sensitivity to X-Ray exposition, we have tried to elucidate its structure. As in Laffittite [2], metal and sulphur atoms form hexagonal rings linked by sharing their edges giving rise to parallel sheets when view along the c axis (see figure). The sheets join themselves composing a three dimensional network. Coordination around the metal ion is typical for sulfosalt structures.



[1] Wang N., Paniagua A., *N. Jb. Miner. Mh.*, 1996, H. 7, 313-320. [2] Nakai I., Appleman D. E., *American Mineralogist*, 1983, 68, 235-244.

**Keywords:** sulfosalts, mineral crystal structures, mineralogy and crystallography using X-ray diffraction

#### P.10.05.20

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##### Crystal Structure of Non-Metamict Minerals of Chevkinite Group

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Crystal chemistry and structure of chevkinites occurring in a high-temperature pneumatolithic vein from Tangir Valley (Pakistan) and in some silica-saturated/oversaturated syenite clasts from São Miguel Island (Azores) were investigated.

Electron microprobe analyses suite the formula A<sub>4</sub>BC<sub>4</sub>O<sub>8</sub>(Si<sub>2</sub>O<sub>7</sub>)<sub>2</sub>, where A = REE, Ca, Th; B = Fe, Mn, Mg; C = Ti, Fe, Nb, Ce and La

are the predominant elements in A sites: Ce<sub>2</sub>O<sub>3</sub> range from 22% to 24% (wt.) and La<sub>2</sub>O<sub>3</sub> from 10% to 14%. The major components in octahedral sites (B and C) are Fe and Ti: FeO range from 11% to 12% (wt.) and TiO<sub>2</sub> from 15% to 17%. The crystal structure was refined in the space group *C2/m* to conventional R(I/σ(I)>4) of ≅ 2%. Site occupancies were calculated on the basis of electron microprobe results, refinement electrons and bond valence analysis. An Fe<sup>3+</sup>/ΣFe ratio of 0.58 for Pakistan chevkinite was determined from Mössbauer spectroscopy. A preliminary spectral analysis points to the presence of two different types of Fe<sup>2+</sup>, both in octahedral sites. TEM investigations revealed the high crystallinity of both chevkinites. At the SAED scale the Pakistan chevkinite shows absence of structural defects, whereas the Azores one shows streaked spots along [001] and [-111] directions. HRTEM study is in progress in order to explain the presence of defects in some directions and to evidence structural regularity in others.

**Keywords:** silicates of rare elements, crystal chemistry and structure, TEM

#### P.10.06.1

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##### Structure of New Rare-Earth Borates Ln[B<sub>6</sub>O<sub>9</sub>(OH)<sub>3</sub>] and its Relation to Boracites

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The crystal structures of rare-earth borates Ln[B<sub>6</sub>O<sub>9</sub>(OH)<sub>3</sub>], Ln=Sm-Lu, synthesized under hydrothermal conditions, are solved in the space group *R3c* (Ln=Ho, *a* 8.385(9), *c* 20,71(4), *R* = 29%, and Ln=Gd, *a* 8.410(4), *c* 20,72(1), *R* = 4.8%). New borates belong to hexaborate group. The polar anionic framework consists of fundamental building blocks FBB [3T+3Δ]: six-membered rings of regularly alternating (BO<sub>4</sub>)-tetrahedra and (BO<sub>3</sub>)-triangles. Atoms of Ln and H are located in wide channels along threefold axis. Ln-borates have the closest structural relation to synthetic boracite Li<sub>4</sub>B<sub>7</sub>O<sub>12</sub>Cl with the same FBB [1].

Structural relationship between all boracite modifications can be revealed, if to pay attention to clusters of four hexaborate blocks of two types [6T] or [3T+3Δ]. In the cubic boracites M<sub>3</sub>B<sub>7</sub>O<sub>13</sub>Cl (M = Mg, Fe) [2] and many synthetic analogues four blocks [6T] are linked via vertices. The trigonal and orthorhombic distortion [3] is caused by increase one of B-O bond and corresponding decrease of B coordination from tetrahedron to triangle. In the cubic Li<sub>4</sub>B<sub>7</sub>O<sub>12</sub>Cl such clusters consist of four blocks [3T+3Δ]. In the trigonal Ln[B<sub>6</sub>O<sub>9</sub>(OH)<sub>3</sub>], in comparison with Li<sub>4</sub>B<sub>7</sub>O<sub>12</sub>Cl, there is one B-atom less, what leads to destruction of such clusters with keeping single six-membered rings.

[1] Jeitschko W., Bither T.A., Bierstedt P.E., *Acta Cryst.*, 1977, B33, 2767. [2] Sueno S., Clark J.R., Papike J.J., Konnert J.A., *Am. Mineral.*, 1973, 58, 691. [3] Dowty E., Clark J.R., *Z. Kristallogr.*, 1973, 138, 64.

**Keywords:** rare-earth borates, boracites, structural topology

#### P.10.06.2

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##### Zeolites Gismondine and Li-ABW under Pressure: Synchrotron XRPD and MD Simulations

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The response to compression of the zeolites gismondine and of LiABW were explored by synchrotron X-ray powder diffraction and ab-initio Molecular Dynamics simulations. Different pressure-transmitting media were used: silicon oil and propane-isopropane as non-penetrating media, and a water-methanol mixture as penetrating one. The applied pressure spans from room pressure to about 10 GPa. The data collection was performed at SNBL1 beamline at ESRF (Grenoble) using a wavelength of 0.7 Å and a MAR 345 (pixel size

100 mm) IP detector; the 2 $\theta$  accessed range is 0 – 36°. The behaviour of the unit cell parameters as a function of pressure and the bulk modulus values were derived by processing the powder patterns. The ab-initio MD simulations, performed on the basis of the experimental cell parameters, provided the atomistic structural interpretation of the response of gismondine to compression.

Gismondine cell parameters regularly decrease and the P-induced modifications are reversible upon decompression. The bulk modulus calculated on the basis of the experiment performed with silicon oil is  $K_0 = 63.8(2)$  GPa. The bulk modulus derived for the three data sets collected on LiABW using different P-transmitting media are  $K_0 = 68(2)$ , 64.8(8) and 60(5) GPa for silicon oil, alcohol-water and pentane-isopentane, respectively.

**Keywords:** zeolites, high-pressure diffraction, molecular dynamics simulations

#### P.10.06.3

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##### The Crystal Structure of Chabasite-K

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The chabasite group contains four minerals: chabasite-Ca, chabasite-Na, chabasite-K, and chabasite-Sr. The crystal structure of the K representative in this group has not been investigated before.

The good isometric crystals of chabasite-K were found in late hydrothermalites of Khibiny alkaline massif, Kola Peninsula, Russia. The average result of electron-microprobe analyses is: Na<sub>2</sub>O 2.62, K<sub>2</sub>O 7.22, CaO 4.61, SrO 1.44, BaO 0.04, Al<sub>2</sub>O<sub>3</sub> 18.75, SiO<sub>2</sub> 46.41, H<sub>2</sub>O 19.35, total 100.44 wt.%. The crystal structure of chabasite-K (K<sub>1.33</sub>Na<sub>1.02</sub>Ca<sub>0.84</sub>)[Al<sub>4</sub>Si<sub>8</sub>O<sub>24</sub>]12.17H<sub>2</sub>O, hexagonal, space group *R* 3c, *a*=13.831(3), *c*=15.023(5)Å, *V*=2489.3(1) Å<sup>3</sup>, *Z*=3, has been determined using a pseudomerohedrally twinned crystal [Enraf Nonius CAD4, MoK $\alpha$ , graphite monochromator, twin ratio 0.535(1) : 0.465(1), *w*R<sub>2</sub>=0.1177 for all 6265 unique reflection, *R*=0.047 for 4745 observed reflection with *I* > 1.96 $\sigma$ (*I*)]. Because of a high mobility of cations within the big framework cavities the experimental data were received at low temperature *T*=193 K.

It has been established that K-bearing chabasite contains five independent sites of cations within the large cavities of Si,Al tetrahedral framework. Eight sites for water molecules were found in the structure. We compared the atoms' distribution in the framework cavities with similar data for chabasites of different composition and origin. Basing on the crystal chemical, mineralogical and geochemical data for zeolites of alkaline formation we assume that the number of cations in the chabasite-K from low-temperature hydrothermalites could be the result of a natural ion-exchange process.

**Keywords:** zeolite, chabasite-K, crystal structure

#### P.10.06.4

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##### Crystallochemistry of Natural Zeolites from "Tuful de Dej" (Romania)

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In Romania, natural zeolites occur in different geological environments (magmatic, crystalline and sedimentary), but the largest spreading is known in the sedimentary formations of the Transylvanian Basin, hosted by the Badenian volcanic tuffs ("Tuful de Dej").

The X-Ray investigations in two different zones of the Transylvanian Basin (the Dej-Bistrita Nasaud and Cluj Napoca Zones) have emphasized (i) a large variation of the zeolitic species, (ii) common and different species (iii) some differences between those two zones: a relative mineralogical homogeneity in the Dej-Bistrita Nasaud (*ferrierite*, *mordenite*, *epistilbite*, *thomsonite*, *natrolite*, *scolecite*, *faujasite*) and more variations in the Cluj Napoca One (*thomsonite*, *faujasite*, *scolecite*, *mesolite*, *heulandite*, *mordenite*, *epistilbite*, *gonnardite*, *ferrierite*, *clinoptilolite*, *chabazite*, *natrolite*).

Chemically, they include Al, Si, Na and Ca in their crystalline

network but Fe<sup>3+</sup> and K are presented too, emphasizing some possible substitutions between Al - Fe<sup>3+</sup> and Na - K.

Chemical homogeneity emphasized by zeolites from the Dej Bistrita-Nasaud Zone and some obvious differences remarked in the Cluj Napoca Zone have determined the similar zeolitic minerals and zeolitic mineral assemblages in the Dej-Bistrita Nasaud Zone and more differences in the Cluj Napoca Zone.

**Keywords:** zeolites, chemistry, X-ray

#### P.10.06.5

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##### Open-framework Germanates by Organic Templating

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Templated germanates have recently drawn great interests due to their remarkably abundant crystal chemistry. The structure diversity of germanates is associated with the ability of germanium to form oxygen polyhedra with four, five and six coordinations.

Recently, a number of new zeolite topologies was discovered in germanates and silicogermanates (ASV, IWR, IWW, UOZ). The major impact of incorporating germanium into zeolite structures is that the T-O-T angles can be much smaller for Ge (~130°) than for Si (~145°). The smaller T-O-T angles are needed for framework structures with 3- and 4-rings, and thus very open frameworks with low framework densities.

We present several germanates with novel zeolite topologies, including a pure germanate FOS-5 (BEC) and a borogermanate SU-16<sup>[1]</sup> (SOS). The building units are double 4-rings (D4R) in FOS-5 and two edge-sharing 3-rings (B<sub>2</sub>Ge) in SU-16.

We also summarize several novel germanates and silicogermanates with extra-large rings, formed by one or two types of clusters. Two of them are SU-12<sup>[2]</sup> and SU-14. Both SU-12 and SU-14 are built from the same (Ge, Si)<sub>7</sub> clusters and contain 24-ring channels. Due to the different connections of the (Ge, Si)<sub>7</sub> clusters, the 3D structures of SU-12 and SU-14 are different, and as well as their 3D intersecting channels.

[1] Li Y.F., Zou X.D., *Angew. Chem. Int. Ed.*, 2005, **44**, early view. [2] Tang L.Q., Dadachov M.S., Zou X.D., *Chem. Mater.*, 2005, in press.

**Keywords:** zeolites, open-frameworks, germanates

#### P.10.06.6

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##### Synthesis and Characterization of Highly Dispersed Tungsten Oxide into SBA Molecular Sieve

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Incorporation of transition metals such as Ti, V, Cr into the framework of the molecular sieves is a general method for introducing catalytic sites into mesoporous materials. Numerous attempts have been made to prepared transition metal-substituted mesoporous materials for making them into effective catalysts which are available of treating large molecules. Tungsten-containing mesoporous materials have attracted much attention in past years. Several groups have tried to synthesized mesoporous tungsten oxide materials or incorporated tungsten into mesoporous materials. However, the oligomer species formed as framework-skeleton synthesized are still a problem for all transition-metal doped mesoporous materials. Herein, the new tungsten SBA molecular sieve using highly dispersed method was showed the attractive performance in direct catalytic conversion. The *p6mm* hexagonal structure of WSBA molecular sieve was showed on powder diffraction patterns. TEM and BET measurement show WSBA with narrowed distribution pore size and rigid structure after the reaction. The high dispersion of tungsten oxide into molecular skeleton was confirmed by UV and Raman spectrum.

**Keywords:** tungsten, high dispersion, mesoporous materials