

## Structural and IR-spectroscopic characterization of magnesium acesulfamate

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Zeitschrift für Naturforschung 71b, 51-55 (2016).

Magnesium acesulfamate,  $\text{Mg}(\text{C}_4\text{H}_4\text{NO}_4\text{S})_2 \cdot 6\text{H}_2\text{O}$ , was prepared by reaction of acesulfamic acid and magnesium carbonate in aqueous solution, and characterized by elemental analysis. Its crystal structure was determined by single crystal X-ray diffraction methods. The substance crystallizes in the triclinic space group  $P(-1)$  with one molecule per unit cell. The FTIR spectrum of the compound was also recorded and is briefly discussed. Some comparisons with other simple acesulfamate and saccharinate salts are also made.

## Vibrational spectra of barium oxalate hemihydrate

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Spectroscopy Letters 49, 238-240 (2016).

The infrared and Raman spectra of barium oxalate hemihydrate,  $\text{BaC}_2\text{O}_4 \cdot 0.5\text{H}_2\text{O}$ , were recorded and discussed on the basis of their structural peculiarities and in comparison with the spectra of the previously investigated calcium and strontium oxalates.

### **Crystal and molecular structure and spectroscopic behavior of isotopic synthetic analogs of the oxalate minerals stepanovite and zhemchuzhnikovite**

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Physics and Chemistry of Minerals 43, 287-300 (2016).

The crystal structure of synthetic stepanovite,  $\text{Na}[\text{Mg}(\text{H}_2\text{O})_6][\text{Fe}(\text{C}_2\text{O}_4)_3] \cdot 3\text{H}_2\text{O}$ , and zhemchuzhnikovite,  $\text{Na}[\text{Mg}(\text{H}_2\text{O})_6][\text{Al}_{0.55}\text{Fe}_{1-0.45}(\text{C}_2\text{O}_4)_3] \cdot 3\text{H}_2\text{O}$ , have been determined by single-crystal X-ray diffraction methods. The compounds are isotopic to each other and to the previously reported  $\text{Na}[\text{Mg}(\text{H}_2\text{O})_6][\text{M}(\text{C}_2\text{O}_4)_3] \cdot 3\text{H}_2\text{O}$  (M: Cr, Al). They crystallize in the trigonal  $P3c1$  space group with  $Z = 6$  molecules per unit cell and (hexagonal axes)  $a = 17.0483(4)$ ,  $c = 12.4218(4)$  Å for the iron compound, and  $a = 16.8852(5)$ ,  $c = 12.5368(5)$  Å for the Al/Fe solid solution. Comparison of our crystallographic results with previous X-ray diffraction and chemical data of type stepanovite and zhemchuzhnikovite minerals provides compelling evidence that these natural materials possess the same crystal and molecular structure as their synthetic counterparts. It is shown that the originally reported unit cell for stepanovite represents a pronounced sub-cell, and that the correct unit cell and space group are based on weak superstructure reflections. The infrared and Raman spectra of both synthetic analogues were also recorded and are briefly discussed.

## Mean amplitudes of vibration and thermodynamic functions of XeOF<sub>2</sub>

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Physical Chemistry, An Indian Journal, 11, 45-47 (2016).

The mean amplitudes of vibration of XeOF<sub>2</sub>, a new example of a Xe(IV) compound, were calculated from recently reported spectroscopic and structural data in the temperature range between 0 and 500 K. The results are compared with those of related species and the bond peculiarities of the molecule are also briefly discussed. Its thermodynamic functions were also determined, using the rigid rotator/harmonic oscillator approximation.

## Syntheses, crystal structures, NMR spectroscopy, and vibrational spectroscopy of Sr(PO<sub>3</sub>F)·H<sub>2</sub>O and Sr(PO<sub>3</sub>F)

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European Journal of Inorganic Chemistry 2016, 1121-1128.

Single crystals of  $\text{Sr}(\text{PO}_3\text{F})\cdot\text{H}_2\text{O}$  ( $P2_1/c$  and  $Z = 4$ ) were grown from an aqueous solution by a metathesis reaction. The structure comprises  $[\text{SrO}_8]$  polyhedra and  $\text{PO}_3\text{F}$  tetrahedra that form a layered arrangement parallel to (100). The topotactic dehydration of this phase proceeds between 80 and 140 °C to afford  $\text{Sr}(\text{PO}_3\text{F})$ . The monazite-type crystal structure this phase was elucidated from the X-ray powder data simulated by annealing ( $P2_1/c$  and  $Z = 4$ ). During dehydration, the structure of  $\text{Sr}(\text{PO}_3\text{F})\cdot\text{H}_2\text{O}$  collapses along (100) from a layered arrangement into a framework structure. The magic angle spinning (MAS) NMR and vibrational spectroscopy data of both phases are discussed.

### **Natural iron oxalates and their analogous synthetic counterparts: A review**

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Chemie der Erde - Geochemistry 76, 449-460 (2016).

Four iron-containing natural oxalates, usually classified as organic minerals, are known (humboldtine, minguzzite, stepanovite and zhemchuzhnikovite). Their occurrences and general properties are presented and briefly discussed. The subsequent part of the review is devoted to the discussion of synthetic oxalato-complexes, analogous to these natural species, including usual synthetic procedures and a thorough analysis of their crystallographic and structural peculiarities. The thermal, spectroscopic and magnetic properties of these complexes are also analyzed in detail and comparisons with other closely related complexes are also included in the discussions.

## Crystal structure and spectroscopic behavior of three new *tris-oxalato*ferrate(III) salts

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Journal of Coordination Chemistry 69, 3715-3725 (2016).

A new general synthetic procedure for the preparation of  $\text{Na}_3[\text{Fe}(\text{C}_2\text{O}_4)_3]\cdot 5\text{H}_2\text{O}$  (**1**),  $\text{Rb}_3[\text{Fe}(\text{C}_2\text{O}_4)_3]\cdot 3\text{H}_2\text{O}$  (**2**), and  $\text{Cs}_3[\text{Fe}(\text{C}_2\text{O}_4)_3]\cdot 2\text{H}_2\text{O}$  (**3**), was developed. The crystal structures of these salts have been determined by single crystal X-ray diffraction methods. Salt (**1**) crystallizes in the monoclinic space group  $C2/c$  with  $Z = 8$  molecules per unit cell, salt (**2**) in  $P2_1/c$  with  $Z = 4$  and salt (**3**) in  $P2_1/n$  with  $Z = 4$ . The three new salts and the  $\text{K}_3[\text{Fe}(\text{C}_2\text{O}_4)_3]\cdot 3\text{H}_2\text{O}$ , prepared for comparative purposes, were further characterized by infrared and  $^{57}\text{Fe}$ -Mössbauer spectroscopy. These spectra are discussed in comparison with those of other related oxalato complexes.