

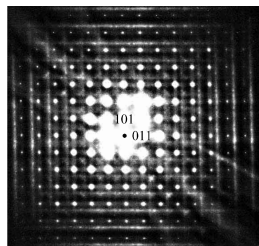
## CONTENTS

## Regular Articles

**An electron diffraction and bond valence sum investigation of oxygen/fluorine ordering in  $\text{Nb}_n\text{O}_{2n-1}\text{F}_{n+2}$ ,  $n = 3$** 

Frank J. Brink, Ray L. Withers, Stéphane Cordier and Marcel Poulain

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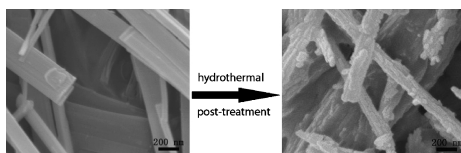


Shows typical  $[1\bar{1}1]$  zone axis EDP for  $\text{Nb}_3\text{O}_5\text{F}_5$ . Note the strong essentially continuous, transverse polarized diffuse streaking along the  $[h0l]^*$  and  $[0kl]^*$  directions of reciprocal space. Such streaking is shown to be consistent with ordering of O/F along the **a** and **b** crystal directions.

**Effects of hydrothermal post-treatment on microstructures and morphology of titanate nanoribbons**

Huogen Yu, Jiaguo Yu, Bei Cheng and Minghua Zhou

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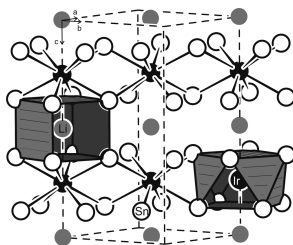


Hydrothermal post-treatment not only promoted the phase transformation from titanate to anatase  $\text{TiO}_2$ , but also was beneficial to the removal of  $\text{Na}^+$  ions remained in the titanate nanoribbons.

**Neutron diffraction and electrochemical studies on  $\text{LiIrSn}_4$** 

Puravankara Sreeraj, Hans-Dieter Wiemhöfer, Rolf-Dieter Hoffmann, Rolf Skowronek, Armin Kirfel and Rainer Pöttgen

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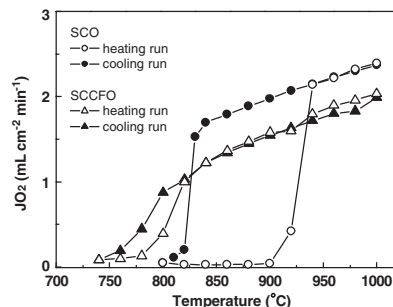
Crystal structure of tetragonal  $\text{LiIrSn}_4$ , space group  $I4/mcm$ . Lithium, iridium, and tin atoms are drawn as grey, filled, and open circles, respectively. The two-dimensional  $[\text{IrSn}_4]$  network, the square-prismatic lithium and the square antiprismatic iridium coordination are emphasised.

## Regular Articles—Continued

**Relationship between transport properties and phase transformations in mixed-conducting oxides**

Z.Q. Deng, W.S. Yang, W. Liu and C.S. Chen

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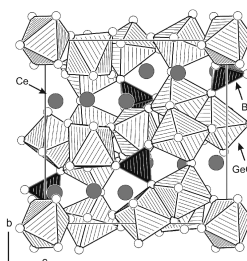


Temperature dependence of oxygen permeation rates through  $\text{Sr}_{0.9}\text{Ca}_{0.1}\text{Co}_{0.89}\text{Fe}_{0.11}\text{O}_{3-\delta}$  (SCCFO) and  $\text{SrCoO}_{3-\delta}$  (SCO) membranes with a thickness of 1.5 mm.

**Multianvil high-pressure/high-temperature synthesis, crystal structure, and thermal behaviour of the rare-earth borogermanate  $\text{Ce}_6(\text{BO}_4)_2\text{Ge}_9\text{O}_{22}$** 

Gunter Heymann and Hubert Huppertz

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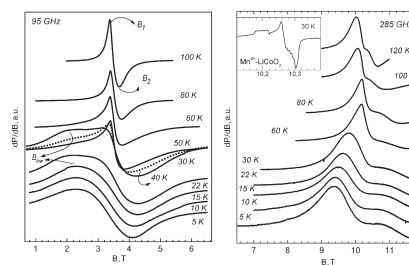


Synthesis of the new borogermanate  $\text{Ce}_6(\text{BO}_4)_2\text{Ge}_9\text{O}_{22}$  via multianvil high-pressure techniques exhibiting  $\text{BO}_4$ -tetrahedra,  $\text{GeO}_6$ -octahedra, and oxygen atoms  $\text{O}^{[3]}$  coordinated by one boron and two germanium atoms.

 **$\text{Mn}^{4+}$  environment in layered  $\text{Li}[\text{Mg}_{0.5-x}\text{Ni}_x\text{Mn}_{0.5}]\text{O}_2$  oxides monitored by EPR spectroscopy**

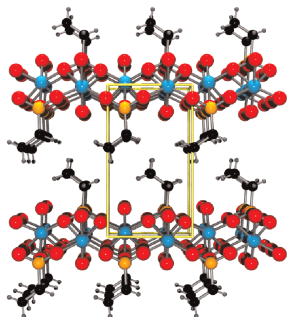
R. Stoyanova, E. Zhecheva and S. Vassilev

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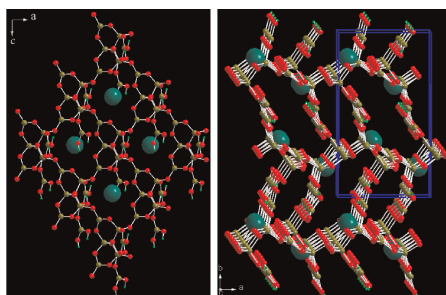
High-frequency EPR spectra of  $\text{LiNi}_{0.5}\text{Mn}_{0.5}\text{O}_2$ .

**Layered hybrid organic–inorganic Co(II) alkylphosphonates. Synthesis, crystal structure and magnetism of the first two members of the series:  $\text{Co}[(\text{CH}_3\text{PO}_3)(\text{H}_2\text{O})]$  and  $\text{Co}[(\text{C}_2\text{H}_5\text{PO}_3)(\text{H}_2\text{O})]$**   
 Elvira M. Bauer, Carlo Bellitto, Marcello Colapietro, Said A. Ibrahim, Mohamed R. Mahmoud, Gustavo Portalone and Guido Righini  
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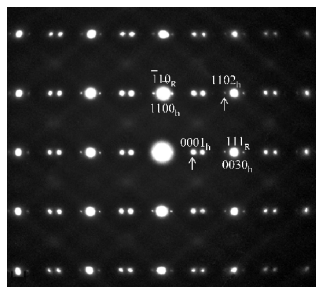
Ball and stick representation of the layered hybrid structure of  $\text{Co}[(\text{C}_2\text{H}_5\text{PO}_3)(\text{H}_2\text{O})]$ .

**Synthesis and characterization of a new layered lead borate**  
 Guo-Ming Wang, Yan-Qiong Sun and Guo-Yu Yang  
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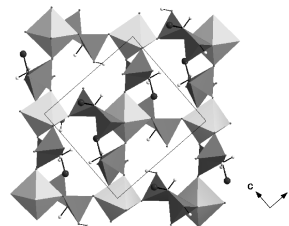
A new layered lead borate of  $\text{Pb}[\text{B}_8\text{O}_{11}(\text{OH})_4]$  has been synthesized under hydrothermal conditions. It consists of layers of 9-membered boron rings enclosing  $\text{Pb}^{2+}$  cations. Adjacent borate layers are interconnected via ionic Pb–O bonds and hydrogen bonding to form a 3D supramolecular network.

**Old friends in a new light: “SnSb” revisited**  
 Lasse Norén, Ray L. Withers, Siegbert Schmid, Frank J. Brink and Valeska Ting  
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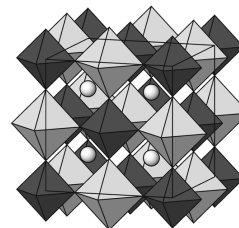
A  $[11\bar{2}]_{\text{R}} \equiv [1\bar{1}0]_{\text{h}}$  zone axis EDP typical of incommensurately modulated “SnSb”. Three integer indexation with the subscript “R” is with respect to the rhombohedral setting unit cell while the  $(3+1)$  integer  $(hklm)$  indexation with the subscript “h” is with respect to the hexagonal setting unit cell.

**Metal phosphonates based on aminomethylenediphosphonate: Syntheses and characterization of  $\text{Na}_4\text{Zn}\{\text{NH}_3\text{CH}(\text{PO}_3)_2\}_2 \cdot 4\text{H}_2\text{O}$ ,  $\text{Ni}\{\text{NH}_3\text{CH}(\text{PO}_3\text{H})_2\}_2 \cdot x\text{H}_2\text{O}$  and  $\text{NaNi}_2\{\text{NH}_3\text{CH}(\text{PO}_3)(\text{PO}_3\text{H}_{0.5})\}_2(\text{H}_2\text{O})_2 \cdot 2\text{H}_2\text{O}$**   
 Song-Song Bao, Tian-Wei Wang, Yi-Zhi Li and Li-Min Zheng  
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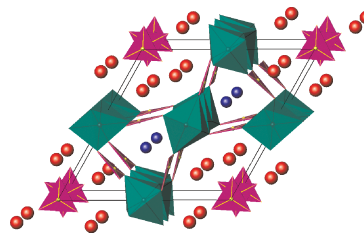
Based on aminomethylenediphosphonate, a zinc compound  $\text{Na}_4\text{Zn}\{\text{NH}_3\text{CH}(\text{PO}_3)_2\}_2 \cdot 4\text{H}_2\text{O}$  (**1**) with 3D open-framework structure and two nickel compounds  $\text{Ni}\{\text{NH}_3\text{CH}(\text{PO}_3\text{H})_2\}_2 \cdot x\text{H}_2\text{O}$  (**2**) and  $\text{NaNi}_2\{\text{NH}_3\text{CH}(\text{PO}_3)(\text{PO}_3\text{H}_{0.5})\}_2(\text{H}_2\text{O})_2 \cdot 2\text{H}_2\text{O}$  (**3**) with square-grid layer and 3D open-framework structures, respectively, are reported. The magnetic studies of compounds **2** and **3** are also investigated.

**Phase transitions in  $\text{K}_3\text{AlF}_6$**   
 Artem M. Abakumov, Marta D. Rossell, Anastasiya M. Alekseeva, Sergey Yu. Vassiliev, Svetlana N. Mudrezova, Gustaaf Van Tendeloo and Evgeny V. Antipov  
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The monoclinic  $\alpha$ - $\text{K}_3\text{AlF}_6$  phase is stable below 132 °C ( $a = 18.8588(2)\text{\AA}$ ,  $b = 34.0278(2)\text{\AA}$ ,  $c = 18.9231(1)\text{\AA}$ ,  $\beta = 90.453(1)^\circ$ , SG  $I2/a$  or  $Ia$ ). The  $\beta$  phase exists in very narrow temperature interval between 132 and 153 °C. The orthorhombic  $\gamma$  polymorph is stable between 153 °C and 306 °C ( $a = 36.1229(6)\text{\AA}$ ,  $b = 17.1114(3)\text{\AA}$ ,  $c = 12.0502(3)\text{\AA}$ , S.G.  $Fddd$ ). Above 306 °C the cubic  $\delta$  polymorph forms with  $a = 8.5786(4)\text{\AA}$  and SG  $Fm\bar{3}m$ .

**Structure of two new borates  $\text{YCa}_3(\text{AlO})_3(\text{BO}_3)_4$  and  $\text{YCa}_3(\text{GaO})_3(\text{BO}_3)_4$**   
 Yi Yu, Q.S. Wu and R.K. Li  
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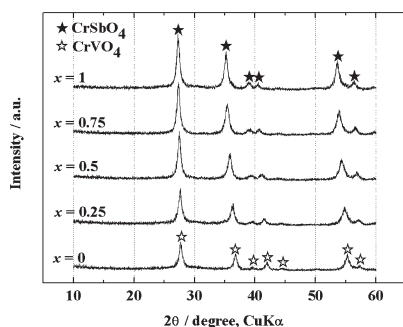


$\text{YCa}_3(\text{MO})_3(\text{BO}_3)_4$  structure is constructed by  $\text{MO}_6$  edge-shared chains interconnected by  $\text{BO}_3$  groups with Y and Ca atoms occupying the trigonal and apatite-like tunnels.

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### Mechanochemical synthesis of rutile-type $\text{CrMO}_4$ ( $M=\text{V}, \text{Sb}$ ) and their solid solutions

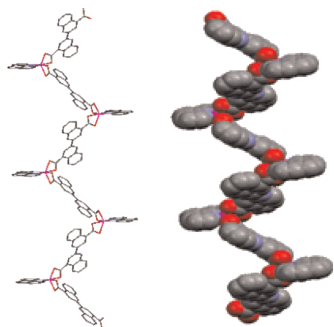
Takatoshi Tojo, Qiwu Zhang and Fumio Saito  
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The solid solutions,  $\text{CrV}_{1-x}\text{Sb}_x\text{O}_4$ , has been mechanochemically synthesized from the mixture of  $\text{Cr}_2\text{O}_3 \cdot \text{H}_2\text{O}$ ,  $\text{V}_2\text{O}_5$  and  $\text{Sb}_2\text{O}_5$ .

### Design and synthesis of four coordination polymers generated from 2,2'-biquinoline-4,4'-dicarboxylate and aromatic bidentate ligands

Junwei Ye, Ping Zhang, Kaiqi Ye, Hongyu Zhang, Shimei Jiang, Ling Ye, Guangdi Yang and Yue Wang  
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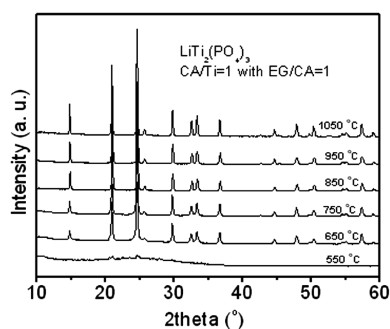


One-dimensional coordination polymer chains.

### Synthesis of nanostructured $\text{LiTi}_2(\text{PO}_4)_3$ powder by a Pechini-type polymerizable complex method

C.R. Mariappan, C. Galven, M.-P. Crosnier-Lopez, F. Le Berre and O. Bohnke

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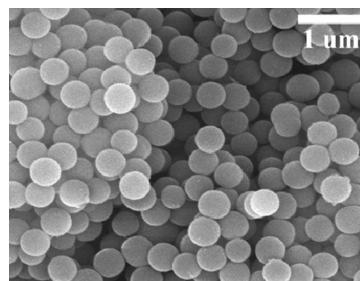


XRD patterns of the powder precursor with  $\text{CA}/\text{Ti}=1$  and  $\text{EG}/\text{CA}=1$ , after heat-treatment at different temperatures for 2 h.

### Preparation of uniform rhodamine B-doped $\text{SiO}_2/\text{TiO}_2$ composite microspheres

Fuyong Yang, Ying Chu, Lei Huo, Yang Yang, Yang Liu and Jinglin Liu

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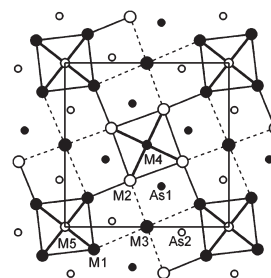


SEM image of RB-doped  $\text{SiO}_2/\text{TiO}_2$  particles calcined at 800 °C.

### Two new titanium molybdenum arsenides: $\text{Ti}_2\text{MoAs}_2$ and $\text{Ti}_3\text{MoAs}_3$ , ternary substitution variants of $\text{V}_3\text{As}_2$ and $\beta\text{-V}_4\text{As}_3$

Abdeljalil Assoud, Shahab Derakhshan, Katja M. Kleinke and Holger Kleinke

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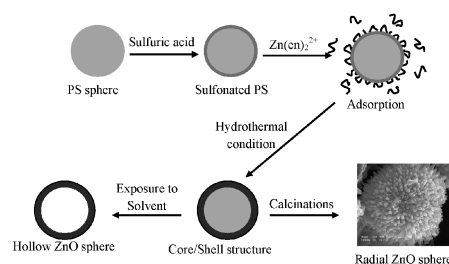


The two title compounds (shown here:  $\text{Ti}_2\text{MoAs}_2$ ) simulate vanadium arsenides by Ti/Mo mixtures on the original V sites ( $M1-M5$ ).

### Polystyrene-ZnO core-shell microspheres and hollow ZnO structures synthesized with the sulfonated polystyrene templates

Yang Yang, Ying Chu, Yanping Zhang, Fuyong Yang and Jinglin Liu

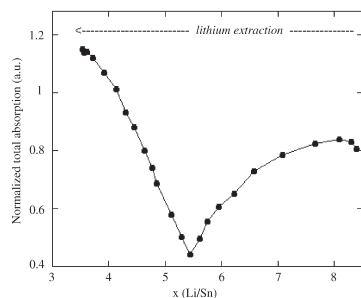
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Schematic illustration for the fabrication of PS-ZnO core-shell microspheres and hollow ZnO structures.

**The chemical changes occurring upon cycling of a  $\text{SnO}_2$  negative electrode for lithium ion cell: In situ Mössbauer investigation**

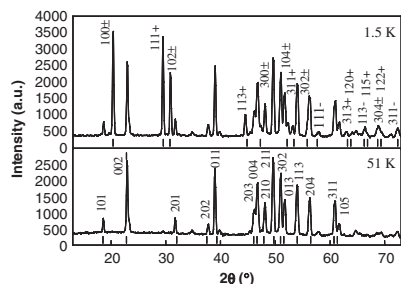
I. Sandu, T. Brousse, D.M. Schleich and M. Danot  
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The first oxidation step of a  $(\text{Li}_{4.4}\text{Sn} + 2\text{Li}_2\text{O})$  electrode could have been expected to consist in progressive lithium extraction from the  $\text{Li}_{4.4}\text{Sn}$  alloy ( $x=8.4$ ) for  $\beta\text{-Sn}$  ( $x=4$ ) to be formed. The variation of the  $^{119}\text{Sn}$  Mössbauer absorption area vs the overall lithium content shows that it is not the case and that a new mechanism takes place when the alloy reaches the composition  $\text{Li}_{1.4}\text{Sn}$  ( $x=5.4$ ).

**Magnetic study of two isotopic manganese chloro-sulfides:  $\text{MnSbS}_2\text{Cl}$  and the new compound  $\text{MnBiS}_2\text{Cl}$**

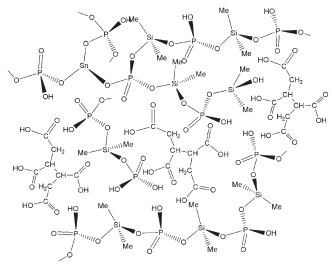
Charlotte Doussier, Gilles André, Philippe Léone, Etienne Janod and Yves Moëlo  
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The crystal structure of a new Bi chloro-sulfide was resolved. The two isotopic compounds,  $\text{MnSbS}_2\text{Cl}$  and  $\text{MnBiS}_2\text{Cl}$ , were studied for their magnetic properties; magnetic susceptibility and specific heat measurements are presented. Neutron diffraction revealed the incommensurate modulation of the magnetic structure of the Sb compound.

**Polycarboxylic acids as network modifiers for water durability improvement of inorganic-organic hybrid tin-silico-phosphate low-melting glasses**

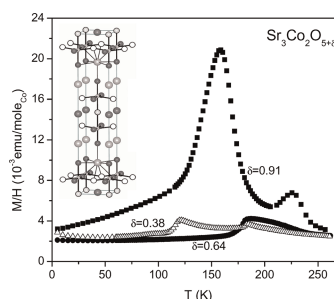
Bouزيد Mena, Megumi Mizuno, Masahide Takahashi, Yomei Tokuda and Toshinobu Yoko  
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Representation of the possible structure formed with the enhancement of the polycondensation and the chain-like structure formation after addition of acid (butane tetracarboxylic acid is shown as an example in the figure).

**Structure and magnetic properties of the orthorhombic  $n=2$  Ruddlesden-Popper phases  $\text{Sr}_3\text{Co}_2\text{O}_{5+\delta}$  ( $\delta=0.91, 0.64$  and  $0.38$ )**

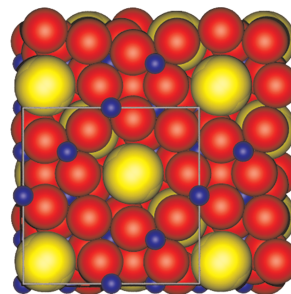
L. Viciu, H.W. Zandbergen, Q. Xu, Q. Huang, M. Lee and R.J. Cava  
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Temperature dependence of the magnetic susceptibility for reduced Ruddlesden-Popper phases,  $\text{Sr}_3\text{Co}_2\text{O}_{5+\delta}$  with  $\delta = 0.91, 0.64$  and  $0.38$ . The average crystal structure of these phases is shown in inset.

**Neutron diffraction and MAS NMR of Cesium Tungstate defect pyrochlores**

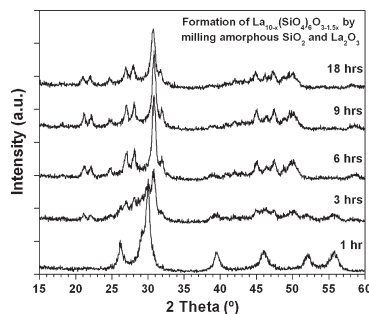
K.R. Whittle, G.R. Lumpkin and S.E. Ashbrook  
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Unit cell representation of  $\text{CsTi}_{0.5}\text{W}_{1.5}\text{O}_6$ , projection of the  $[110]$  plane. Yellow spheres are  $\text{Cs}^+$ , red spheres are  $\text{O}^{2-}$  and blue spheres are the B-site.

**Structural, microstructural and vibrational characterization of apatite-type lanthanum silicates prepared by mechanical milling**

E. Rodríguez-Reyna, A.F. Fuentes, M. Maczka, J. Hanuza, K. Boulahya and U. Amador  
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Crystalline apatite-type lanthanum silicates obtained at room temperature by mechanical milling stoichiometric mixtures of elemental oxides in a planetary ball mill.

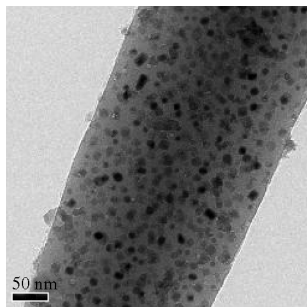
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### Microstructure and luminescence of transparent glass ceramic containing $\text{Er}^{3+}:\text{BaF}_2$ nano-crystals

Daqin Chen, Yuansheng Wang, Yunlong Yu, En Ma and Lihua Zhou

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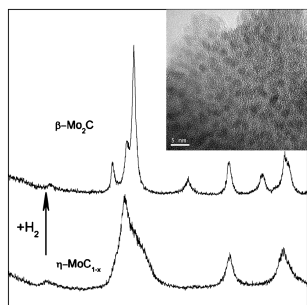


Transparent glass ceramics containing  $\text{BaF}_2$  nano-crystals doped with  $\text{Er}^{3+}$  were prepared by sol-gel route. The upconversion emissions emerged when the sample was heat-treated at  $800^\circ\text{C}$ .

### Synthesis and characterization of molybdenum carbides using propane as carbon source

Xiao-Hui Wang, Hong-Ling Hao, Ming-Hui Zhang, Wei Li and Ke-Yi Tao

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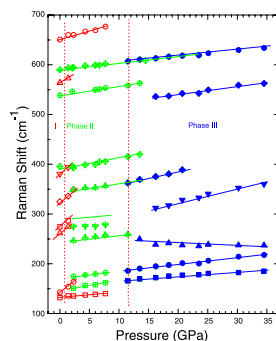


A post-treatment by hydrogen causes the phase transformation of molybdenum carbides in both the conventional TPRE method and a rapid heating method. The HRTEM image shows fairly uniform particles of  $\beta\text{-Mo}_2\text{C}$  display an excellent dispersion on amorphous  $\text{SiO}_2$  support.

### Structural behavior of $\text{Sr}_2\text{Bi}_2\text{O}_5$ at high pressures

F.X. Zhang, B. Manoun, S.K. Saxena and C.S. Zha

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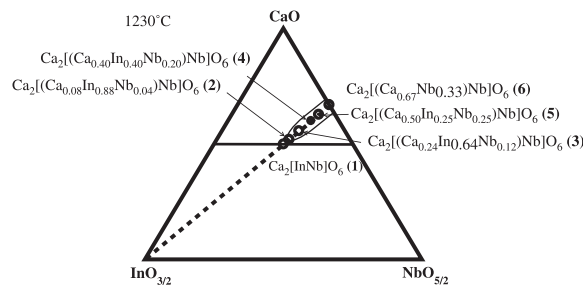


Pressure dependence of the frequency of the observed Raman modes for orthorhombic  $\text{Bi}_2\text{Sr}_2\text{O}_5$  clearly indicates the phase transition.

### A structure and phase analysis investigation of the “1:1” ordered $A_2\text{InNbO}_6$ perovskites ( $A = \text{Ca}^{2+}, \text{Sr}^{2+}, \text{Ba}^{2+}$ )

V. Ting, Y. Liu, R.L. Withers, L. Norén, M. James and J.D. Fitz Gerald

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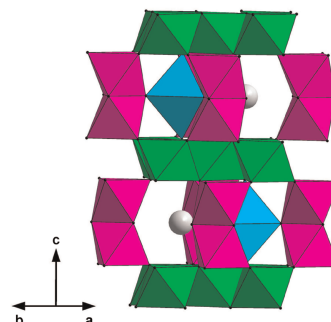


Ternary phase diagram showing the compositions investigated along the quasi-binary join between the  $\text{Ca}_2[\text{InNb}]\text{O}_6$  1:1 material and the  $\text{Ca}_2[(\text{Ca}_{0.67}\text{Nb}_{0.33})\text{Nb}]\text{O}_6$  perovskite structure.

### Synthesis, structure and physical properties of Ru ferrites: $\text{BaMRu}_5\text{O}_{11}$ ( $M = \text{Li}$ and $\text{Cu}$ ) and $\text{BaM}'_2\text{Ru}_4\text{O}_{11}$ ( $M' = \text{Mn}, \text{Fe}$ and $\text{Co}$ )

M.L. Foo, Q. Huang, J.W. Lynn, Wei-Li Lee, Tomasz Klimczuk, I.S. Hagemann, N.P. Ong and R.J. Cava

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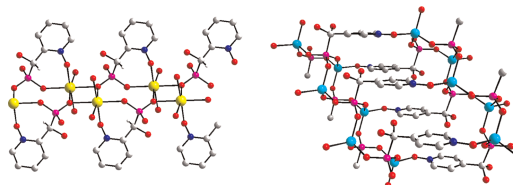


The representative crystal structure of  $R$ -type ferrite  $\text{BaMRu}_5\text{O}_{11}$  ( $M = \text{Li}$  and  $\text{Cu}$ ) and  $\text{BaM}'_2\text{Ru}_4\text{O}_{11}$  ( $M' = \text{Mn}, \text{Fe}$  and  $\text{Co}$ ) with emphasis on the transition metal-oxygen polyhedra. Edge sharing octahedra are shaded green; face sharing octahedra are shaded magenta; isolated trigonal bipyramids are shaded cyan; barium atoms are shaded grey.

### Metal phosphonates containing pyridyl N-oxide groups: Syntheses of $\text{Cd}\{(2\text{-C}_5\text{H}_4\text{NO})\text{CH}(\text{OH})\text{PO}_3\}(\text{H}_2\text{O})_2$ and $\text{Zn}\{(4\text{-C}_5\text{H}_4\text{NO})\text{CH}(\text{OH})\text{PO}_3\}$ with chain and layer structures

Deng-Ke Cao, Yi-Zhi Li and Li-Min Zheng

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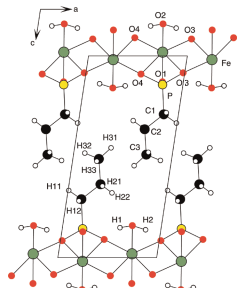


This paper reports the syntheses and characterization of two polymeric phosphonate compounds incorporating pyridyl N-oxide groups namely  $\text{Cd}\{(2\text{-C}_5\text{H}_4\text{NO})\text{CH}(\text{OH})\text{PO}_3\}(\text{H}_2\text{O})_2$  (1) with a chain structure and  $\text{Zn}\{(4\text{-C}_5\text{H}_4\text{NO})\text{CH}(\text{OH})\text{PO}_3\}$  (2) with a layer structure.

**Synthesis, structural determination and magnetic properties of layered hybrid organic–inorganic, iron (II) propylphosphonate,  $\text{Fe}[(\text{CH}_3(\text{CH}_2)_2\text{PO}_3)(\text{H}_2\text{O})]$ , and iron (II) octadecylphosphonate,  $\text{Fe}[(\text{CH}_3(\text{CH}_2)_{17}\text{PO}_3)(\text{H}_2\text{O})]$**

Carlo Bellitto, Elvira M. Bauer, Philippe Léone, Alain Meerschaut, Catherine Guillot-Deudon and Guido Righini

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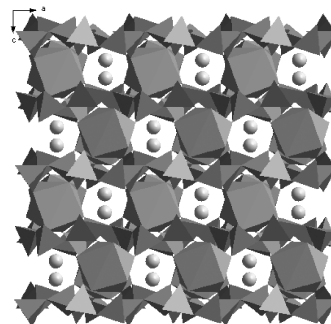


Unit-cell packing of  $\text{Fe}[(\text{CH}_3(\text{CH}_2)_2\text{PO}_3)(\text{H}_2\text{O})]$  viewed along the *b*-axis.

**Structure, energy band, and optical properties of  $\text{NaLa}(\text{PO}_3)_4$  crystal**

J. Zhu, W.-D. Cheng, D.-S. Wu, H. Zhang, Y.-J. Gong and H.-N. Tong

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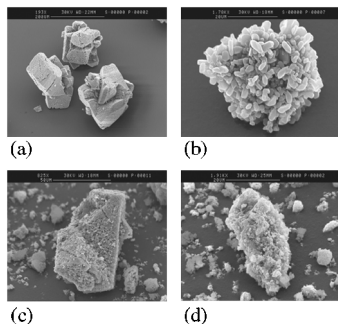


$\text{LaO}_8$  polyhedra and  $[(\text{PO}_3)_4]^{4-}$  chains share oxygen atoms to form a three-dimensional framework, delimiting intersecting tunnels in which the sodium ions are located.

**Nanosized  $\text{LiMn}_2\text{O}_4$  from mechanically activated solid-state synthesis**

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SEM micrographs of (a)  $\text{MnO}$ , (b)  $\text{Li}_2\text{CO}_3$ , (c)  $\text{Li}_2\text{CO}_3/\text{MnO}$  manually grinded stoichiometric mixture and (d) the same mixture after 5 h mechanical grinding.

**NOTICE**

The Keyword Index for Volume 179 will appear in the December 2006 issue as part of a cumulative index for the year 2006.