

Volume 180, Number 3, March 2007

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Regular Articles

Synthesis, crystal and band structures, and properties of a new supramolecular complex (Hg₂As)₂(CdI₄) Jian-Ping Zou, Dong-Sheng Wu, Shu-Ping Huang, Jing Zhu, Guo-Cong Guo and Jin-Shun Huang *Page 805*



A new quaternary supramolecular complex (Hg₂As)₂(CdI₄) (1) has been prepared by the solid-state reaction, and structurally characterized by single crystal X-ray diffraction analysis. The structure of 1 is characterized by a 3-D tridymite-like cationic framework with the channels being occupied by discrete CdI_4^{2-} tetrahedral guest-anions.

Structural modulation in K₂V₃O₈

B.C. Chakoumakos, R. Custelcean, T. Kamiyama, K. Oikawa, B.C. Sales and M.D. Lumsden *Page 812*



The 115 K phase transition in the fresnoite-type $K_2V_3O_8$ is elucidated by neutron powder and single-crystal X-ray diffraction. Below 115 K, weak superlattice reflections are clearly evident in X-ray diffraction patterns recorded by a CCD detector, and these extra reflections can be indexed with the wave vector $\pm 1/3 \langle 110 \rangle^* + 1/2c^*$.

Regular Articles—Continued

Tight binding prediction of the $\alpha\text{-}Gd_2S_3$ magnetic structure

Lindsay E. Roy and Timothy Hughbanks *Page 818*



The spin-dependent EHTB method correctly predicts the magnetic structure of α -Gd₂S₃ determined from neutron diffraction experiments.

Phase transitions in A-site substituted perovskite compounds: The $(Ca_{1-2x}Na_xLa_x)TiO_3$ ($0 \le x \le 0.5$) solid solution

Yuan Li, Shan Qin and Friedrich Seifert *Page 824*



X-ray diffraction data and calculated pattern for $Na_{1/2}La_{1/2}TiO_3$, the end-member of the $Ca_{1-2x}Na_xLa_xTiO_3$ (x = 0.5). The experimental data are shown as crosses, the calculated fits and difference curves as solid lines. Tick marks (upper row for internal standard silicon) indicate the calculated positions of peaks.

Crystal structure and magnetic properties of the solid-solution phase $Ca_3Co_{2-\nu}Sc_\nu O_6$

Charles H. Hervoches, Vivian Miksch Fredenborg, Arne Kjekshus, Helmer Fjellvåg and Bjørn C. Hauback *Page 834*



The quasi-one-dimensional Ca₃Co₂O₆ phase forms a substitutional solid-solution system with Sc, in which the Sc atoms enter the Co2 sublattice exclusively. The homogeneity range of Ca₃Co_{2-v}Sc_vO₆ extends up to $v \approx 0.55$. The magnetic moment decreases rapidly with increasing amount of the non-magnetic Sc solute in the lattice.

Synthesis and characterization of mixed-morphology CePO₄ nanoparticles

L. Karpowich, S. Wilcke, Rong Yu, G. Harley, J.A. Reimer and L.C. De Jonghe *Page 840*



High resolution electron microscopy image of amorphous intergranular phase (a) in polycrystalline monazite CePO₄.

Enthalpies of formation of rare earth orthovanadates, *RE*VO₄ M. Dorogova, A. Navrotsky and L.A. Boatner *Page 847*



Comparison of enthalpies of formation from oxides at 298 K for $REVO_4$ [this work] and $REPO_4$ compounds [S.V. Ushakov, K.B. Helean, A. Navrotsky, L.A. Boatner, J. Mater. Res. 16(9) (2001) 2623] vs. RE³⁺ ionic radius. Filled symbols indicate scheelite structure, open symbols zircon structure.

 $Zr_{1-x}Ln_xW_2O_{8-x/2}$ (*Ln* = Eu, Er, Yb): Solid solutions of negative thermal expansion-synthesis, characterization and limited solid solubility

Hai-Hua Li, Jing-Sa Han, Hui Ma, Ling Huang and Xin-Hua Zhao *Page 852*



The graphic displays the lattice parameters of saturated solid solutions and solubility of $Zr_{1-x}Ln_xW_2O_{8-y}$ as a function of the radius of substituted Ln^{3+} ions. A more sensitive method is developed to measure the low solubility of the solid solutions. This correlation demonstrates the increase in limited solid solubility follows the lanthanide contraction trend in a way that is opposite to that observed for lattice parameters.

Structured diffuse scattering and polar nano-regions in the $Ba(Ti_{1-x}Sn_x)O_3$ relaxor ferroelectric system

Yun Liu, Ray L. Withers, Xiaoyong Wei and John D. Fitz Gerald

Page 858



The characteristic $\mathbf{G} \pm \{001\}^*$ sheets of diffuse intensity characteristic of the relaxor ferroelectric Ba $(\mathrm{Ti}_{1-x}\mathrm{Sn}_x)\mathrm{O}_3$ (BTS), $0.1 \le x \le 0.5$, system along with the inherently polar, transverse optical modes of distortion responsible.

Three-dimensional framework of uranium-centered polyhedra with non-intersecting channels in the uranyl oxy-vanadates $A_2(UO_2)_3(VO_4)_2O$ (A = Li, Na) S. Obbade, L. Duvieubourg, C. Dion and F. Abraham *Page 866*



A view of the 3-D structure of Li₂(UO₂)₃(VO₄)₂O.

Continued

Stabilization of Co²⁺ in layered double hydroxides (LDHs) by microwave-assisted ageing

M. Herrero, P. Benito, F.M. Labajos and V. Rives *Page 873*



The use of microwave-hydrothermal treatment, controlling both temperature and ageing time, permits to synthesize well-crystallized nanomaterials with controlled surface properties. An enhancement in the crystallinity degree and an increase in the particle size are observed when the irradiation time is prolonged.

TEM investigation of the microporous compound VSB-1: Building units and crystal growth mechanisms

Marie Colmont and Osamu Terasaki
Page 885



TEM investigation of the microporous compound VSB-1 evidenced defects and information about building units and crystal growth. As usually observed for microporous materials, the crystal growth via a layer by layer mechanism.

Effect of sorbed molecules on the resistivity of alkali metal-graphite intercalation compounds

Noboru Akuzawa, Yoji Kunihashi, Yuki Sato, Ken-ichi Tsuchiya and Rika Matsumoto *Page 894*



The resistivity of MC_{24} increased with increase of the sorbed amount of H₂. The magnitude of the increase was in the order $KC_{24} > RbC_{24} > CsC_{24}$. This resistivity increase was considered to be due to the expansion along *c*-direction which reduces the chargetransfer interaction between the carbon layers and potassium ions.

Preparation of mesoporous cadmium sulfide nanoparticles with moderate pore size

Zhaohui Han, Huaiyong Zhu, Jeffrey Shi, Gordon Parkinson and G.Q. Lu





The preparation involves a hydrothermal process and a subsequent acid treatment process, the majority of the particles have a moderate pore size, which complements and fills the gap between existing porous materials usually have a pore size either less than 10 nm or well above 100 nm.

 $ZnCl_2 + NaOH + NaHS \cdot xH_2O \xrightarrow[Hydrothermal process]{CdCl_2}$

 $Zn_{5}(OH)_{5}Cl_{2} \cdot H_{2}O/Cd_{7.23}Zn_{2.7}S_{10} + ZnO/Cd_{7.23}Zn_{2.7}S_{10} \xrightarrow{Dilute HCl} Porous CdS.$

Growth of new ternary intermetallic phases from Ca/Zn eutectic flux

Milorad Stojanovic and Susan E. Latturner *Page 907*



The calcium/zinc eutectic is a useful synthesis medium for the growth of new intermetallic phases. Addition of group 10 transition metals to this flux produces ternary phases $CaNi_2Zn_3$, $Ca_{21}Ni_2Zn_{36}$, $CaPd_{0.85}Zn_{1.15}$, and $Ca_6Pt_3Zn_5$. The nickel-centered zinc icosahedron surrounded by a pentagonal dodecahedron of calcium atoms is found in $Ca_{21}Ni_2Zn_{36}$.

Defect structure of $Sb_{2-x}Fe_xTe_3$ single crystals

J. Horák, P. Lošťák, Č. Drašar, J. Navrátil and C. Uher *Page 915*



The concentration of holes P and the concentration of defects c in $Sb_{2-x}Fe_xTe_3$ single crystals on Fe-content.

Li₂Si₃O₇: Crystal structure and Raman spectroscopy Hannes Krüger, Volker Kahlenberg and Reinhard Kaindl

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Structure of $Li_2Si_3O_7$: $[Si_6O_{14}]$ silicate layers formed by two independent *zweier* single chains. Lithium atoms represented by 95% probability displacement ellipsoids.

Synthesis and characterization of new strontium 4-carboxyphenylphosphonates

Vítězslav Zima, Jan Svoboda, Ludvík Beneš, Klára Melánová, Miroslava Trchová and Jiří Dybal *Page 929*



The structure of β -Sr(HOOCC₆H₄PO₃H)₂ viewed in the direction of the the *bc* plane.

Comparative high-pressure study and chemical bonding analysis of Rh₃Bi₁₄ and isostructural Rh₃Bi₁₂Br₂ Q.F. Gu, G. Krauss, Yu. Grin and W. Steurer *Page 940*



 Rh_3Bi_{14} isostructural with $Rh_3Bi_{12}Br_2$ consists of a 3D framework of edge-sharing cubes and square antiprisms. High-pressure X-ray powder diffraction measurements of Rh_3Bi_{14} and $Rh_3Bi_{12}Br_2$ indicate a high stability of both compounds in the investigated range from ambient pressure to ca 30 GPa at room temperature.

Synthesis and characterizations of hierarchical biomorphic titania oxide by a bio-inspired bottom-up assembly solution technique

Qun Dong, Huilan Su, Wei Cao, Di Zhang, Qixin Guo and Yijian Lai Page 949



Hierarchical mesoporous TiO₂ is assembled by nanoparticles from the nanoscale to the macroscale through a bio-inspired sol-gel approach with eggshell membrane used as the biotemplate. As-prepared hierarchical titania shows porous characters within the pore size range of $2 \text{ nm}-4 \mu \text{m}$.

Crystal chemistry of $M[PO_2(OH)_2]_2 \cdot 2H_2O$ compounds (M = Mg, Mn, Fe, Co, Ni, Zn, Cd): Structural investigation of the Ni, Zn and Cd salts

Violeta Koleva and Herta Effenberger *Page 956*



IR spectroscopy showed the internal PO₄ vibrations to be insensitive to the size and the electronic configuration of the M^{2+} ions and reflect the more ionic character of the Mg–O bonds. The observed "*ABC* trio" characteristic for acidic salts is discussed. Single-crystal X-ray investigations were performed at 120 and 295 K. One of the four hydrogen bonds is worth mentioning because of the short O_h...O bond distance of 2.57–2.61 Å at room temperature (2.56–2.57 Å at 120 K).

Experimental and theoretical contributions to the determination of optical properties of synthetic paramelaconite

J.F. Pierson, E. Duverger and O. Banakh

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Comparison between experimental (lines) and calculated (points) refractive index and extinction coefficient values. For experimental measurements, the film was deposited on silicon substrate.

Ab initio structural, electronic and optical properties of orthorhombic CaGeO₃

J.M. Henriques, E.W.S. Caetano, V.N. Freire, J.A.P. da Costa and E.L. Albuquerque *Page 974*



Orthorhombic CaGeO₃ was studied using density-functional theory (DFT) considering both the local density and generalized gradient approximations, LDA and GGA, respectively. The electronic band structure, density of states, effective masses, dielectric function and optical absorption were obtained after geometry optimization of the unit cell.

Crystal structures and spectroscopic properties of a new zinc phosphite cluster and an unexpected chainlike zinc phosphate obtained by hydrothermal reactions

Zhi-En Lin, Wei Fan, Jinlou Gu and Tatsuya Okubo Page 981



A new molecular zinc phosphite and a new chainlike zinc phosphate have been synthesized under hydrothermal conditions. The two compounds exhibit intensive photoluminescence originated from the intraligand $\pi - \pi^*$ transitions.

A simple method for systematically controlling ZnO crystal size and growth orientation

Rong Zhang and Lei L. Kerr Page 988



Illustration of ZnO crystals morphology evolution from long-andslim hexagonal rods to fat-and-short hexagonal pyramids, and then to twinning hexagonal dots as a function of Cd^{2+}/Zn^{2+} molar ratio, R_m .

Crystal structures of Na_{1/2}*Ln*_{1/2}**TiO**₃ (*Ln*: La, Eu, Tb) Rajeev Ranjan, Anatoliy Senyshyn, Hans Boysen, Carsten Baehtz and Friedrich Frey *Page 995*



Rietveld plot of $Na_{1/2} Tb_{1/2} TiO_3$ after refinement with space group Pbnm.

Cation ordering in the fluorite-like transparent conductors $In_{4+x}Sn_{3-2x}Sb_xO_{12}$ and In_6TeO_{12}

J. Choisnet, L. Bizo, M. Allix, M. Rosseinsky and B. Raveau

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Hexagonal cell of the fluorite-like oxygen-deficient M_7O_{12} structure: symmetrical octahedra and distorted sevenfold-coordinated polyhedra are shown.

Synthesis and structural study of a new NASICON-type solid solution: $Li_{1-x}La_{x/3}Zr_2(PO_4)_3$

M. Barré, M.P. Crosnier-Lopez, F. Le Berre, E. Suard and J.L. Fourquet

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Schematic drawing of the space group evolution at 800 °C in the solid solution $\text{Li}_{1-x}\text{La}_{x/3}\text{Zr}_2(\text{PO}_4)_3$ ($0 \le x \le 1$).

Single-crystal synthesis, structure analysis, and physical properties of the calcium ferrite-type $Na_xTi_2O_4$ with 0.558 < x < 1

Yasuhiko Takahashi, Kunimitsu Kataoka, Ken-ichi Ohshima, Norihito Kijima, Junji Awaka, Kenji Kawaguchi and Junji Akimoto

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Magnetic susceptibility of three $Na_xTi_2O_4$ samples below 300 K. The broad maximum around 40 K observed in as-grown $NaTi_2O_4$ is suppressed by an Na deficiency and vanishes in $Na_{0.717}Ti_2O_4$.

Large aspect ratio titanate nanowire prepared by monodispersed titania submicron sphere via simple wet-chemical reactions

Baoxiang Wang, Yong Shi and Dongfeng Xue *Page 1028*



Monodispersed raw titania powders are beneficial to get the long and uniform $K_2 Ti_6 O_{13}$ nanowires with a large aspect ratio. A formation mechanism of nanowires is proposed based on the dissolving, growth, thickening and splitting of $K_2 Ti_6 O_{13}$ nano-intermediates.

Magnetic diphase nanostructure of $ZnFe_2O_4/\gamma$ -Fe₂O₃ Xiangxi Bo, Guangshe Li, Xiaoqing Qiu, Yanfeng Xue and Liping Li

Page 1038



Spherical diphase nanostructure of $ZnFe_2O_4/\gamma$ -Fe_2O_3 was obtained by varying the initial molar ratio of Fe/Zn.

Crystal structure, phase relations and electrochemical properties of monoclinic Li₂MnSiO₄

V.V. Politaev, A.A. Petrenko, V.B. Nalbandyan, B.S. Medvedev and E.S. Shvetsova *Page 1045*



Arrangement of Mn (large white balls) and Si (small grey balls) atoms in Li_2MnSiO_4 structure. Bold lines connect atoms linked via common oxygen (not shown).

Crystal structure and magnetic properties of two new cobalt selenite halides: $Co_5(SeO_3)_4X_2$ (X = Cl, Br) Richard Becker, Mladen Prester, Helmuth Berger, Ping Hui Lin, Mats Johnsson, Djuro Drobac and Ivica Zivkovic

Page 1051



Two new iso-structural cobalt selenite halides $Co_5(SeO_3)_4Cl_2$ and $Co_5(SeO_3)_4Br_2$ have been synthesized which are iso-structural to $Ni_5(SeO_3)_4Br_2$. Magnetic susceptibility measurements on oriented single-crystalline samples show anisotropic response in a broad temperature range, revealing significant single-ion anisotropy effects.

Structure and basic magnetic properties of the honeycomb lattice compounds $Na_2Co_2TeO_6$ and $Na_3Co_2SbO_6$

L. Viciu, Q. Huang, E. Morosan, H.W. Zandbergen, N.I. Greenbaum, T. McQueen and R.J. Cava *Page 1060*



The layer of edge-shared MO_6 octahedra, showing the honeycomb Co array.

Structural properties of a family of hydrogen-bonded co-crystals formed between gemfibrozil and hydroxy derivatives of *t*-butylamine, determined directly from powder X-ray diffraction data

Eugene Y. Cheung, Sarah E. David, Kenneth D.M. Harris, Barbara R. Conway and Peter Timmins *Page 1068*



Structural properties of a family of co-crystals containing gemfibrozil and hydroxy derivatives of *t*-butylamine are discussed and rationalized.

Synthesis and single-crystal X-ray diffraction studies of new framework substituted type II clathrates,

 $Cs_8Na_{16}Ag_xGe_{136-x}$ (x < 7)

M. Beekman, W. Wong-Ng, J.A. Kaduk, A. Shapiro and G.S. Nolas

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The synthesis of framework-substituted type II germanium clathrates, $Cs_8Na_{16}Ag_xGe_{136-x}$, is reported for the first time. Single crystal X-ray diffraction studies indicate that Ag substitutes for Ge on the clathrate framework, with preferential substitution occurring on the 96g site. These results offer new direction in the synthesis of novel clathrate compounds, materials of interest for potential thermoelectric applications.

Structures and phase diagram for the system CaTiO₃-La_{2/3}TiO₃

Zhaoming Zhang, Gregory R. Lumpkin, Christopher J. Howard, Kevin S. Knight, Karl R. Whittle and Keiichi Osaka

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Phase diagram for $Ca_{(1-x)}La_{3x/2}TiO_3$ based on transition temperatures obtained from *in situ* neutron and synchrotron X-ray powder diffraction studies: depending on whether or not we have in-phase and/or out-of-phase octahedral tilting, whether we have long-range cation/vacancy ordering (the degree of ordering is indicated by shading), six distinct structures occur. The incommensurately modulated NiGe_{1-x}P_x, ~ $0.3 \le x \le \sim 0.7$, solid solution: The 'missing link' between the *NiP* and *MnP* structure types A.-K. Larsson, F.J. García-García and R.L. Withers *Page 1093*



The NiP (top) and NiGe (bottom) crystal structures. The structural relationship is emphasised by indicating the xsin[Ge/P] modulation wave in the 4-d space group setting $Aman(00\gamma)s00$; $\gamma = 1$ for NiGe, $\gamma = 1/2$ for NiP and $0.78 < \gamma < 0.70$ for NiGe_{1-x} P_x with 0.4 > x > 0.7.

Electronic structure, galvanomagnetic and magnetic properties of the bismuth subhalides Bi_4I_4 and Bi_4Br_4

T.G. Filatova, P.V. Gurin, L. Kloo, V.A. Kulbachinskii, A.N. Kuznetsov, V.G. Kytin, M. Lindsjo and B.A. Popovkin *Page 1103*



Quasi one-dimensional compounds Bi_4Br_4 and Bi_4I_4 have been investigated theoretically (electronic structure calculations) and experimentally (galvanomagnetic and magnetic measurements). Both compounds are found to be diamagnetic, room-temperature semiconductors with n-type conductivity and a possibility of significant directional anisotropy. Magnetoresistivity data indicate some weak interactions between isolated bismuth fragments in Bi_4I_4 .

$AgGaS_2$ -type photocatalysts for hydrogen production under visible light: Effects of post-synthetic H_2S treatment

Jum Suk Jang, Sun Hee Choi, Namsoo Shin, Chungjong Yu and Jae Sung Lee

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Post-treatment of $AgGaS_2$ under H_2S flow at 1123 K resulted in the well-defined chalcopyrite structure, which was revealed mainly by Rietveld analysis of XRD and detailed EXAFS study. The diameters of $AgGaS_2$ particles treated at 873–1123 K under H_2S gas flow are ca. 2–3 nm, almost the same as that of H_2S -untreated material. In untreated sample, $AgGaS_2$ particles were surrounded by the broken powders having sharp edges. But there were almost no broken particles and the surface became smoother for $AgGaS_2$ treated with H_2S gas at 1123 K. The rates of hydrogen evolution over H_2S treated $AgGaS_2$ at higher temperatures (1073 and 1123 K) were higher than those of photocatalysts untreated at lower temperatures (873, 973 K). There is almost no correlation between BET surface areas and hydrogen evolution rates.

Sulfur radicals embedded in various cages of ultramarine analogs prepared from zeolites

Stanisław Kowalak, Aldona Jankowska, Sebastian Zeidler and Andrzej B. Więckowski





Structures of SOD, LTA, cancrinite and erionite.

Intercalation behavior of *n*-alkylamines into an *A*-site defective layered perovskite $H_2W_2O_7$ Bingshan Wang, Xiaowen Dong, Qingyi Pan,

Zhixuan Cheng and Yanze Yang

Page 1125



H₂W₂O₇ can accommodate *n*-alkylamines (C_nH_{2n+1}NH₂: n=3, 4, 7, 8, 12, 16) to form intercalation compounds via an acid–base mechanism. After intercalation, the [001] reflections of H₂W₂O₇ shift to lower angles. The basal space of the intercalation products with *n*-alkylamines increases with an increase of the number of carbon atoms in *n*-alkyl chain. The relationship between the interlayer distance, *d* and the number of carbon atoms in the *n*-alkyl chain, *n*, is demonstrated in figure. A linear relationship is clearly observed, as expressed with d=0.241n+0.67.

Characterization and acidic properties of Al-SBA-15 materials prepared by post-synthesis alumination of a low-cost ordered mesoporous silica

M. Gómez-Cazalilla, J.M. Mérida-Robles, A. Gurbani, E. Rodríguez-Castellón and A. Jiménez-López *Page 1130*



Al KLL spectra of Al-SBA-15 materials with different Si/Al ratios.

A new molecular precursor route for the synthesis of Bi–Y, Y–Nb and Bi-doped Y–Nb oxides at moderate temperatures D.A. Bayot, A.M. Dupont and Michel M. Devillers *Page 1141*



Bi–Y, Nb–Y and Bi-doped Nb–Y oxides were prepared by a molecular precursors method from pre-isolated water-soluble edtabased complexes. The cubic $Bi_{1-x}Y_xO_{1.5}$ and Y_3NbO_7 oxides were obtained in a pure form at the moderate temperature of 650 °C. A distorted tetragonal YNbO₄ phase was also stabilized at room temperature by calcining the precursor at 800 °C, and the pure corresponding monoclinic oxide has been obtained near 1100 °C.

Hydrothermal synthesis and crystal structure of the $Ni_2(C_4H_4N_2)(V_4O_{12})(H_2O)_2$ and $Ni_3(C_4H_4N_2)_3(V_8O_{23})$ inorganic–organic hybrid compounds. Thermal, spectroscopic and magnetic studies of the hydrated phase Edurne S. Larrea, José L. Mesa, José L. Pizarro, María I. Arriortua and Teófilo Rojo *Page 1149*



Crystal structure of a sheet of $Ni_2(C_4H_4N_2)(V_4O_{12})(H_2O)_2$.

Rapid Communications

Thermoelectric properties of Ag-doped n-type $(Bi_2Te_3)_{0.9}$ - $(Bi_{2-x}Ag_xSe_3)_{0.1}$ (x = 0-0.4) alloys prepared by spark plasma sintering

J.L. Cui, W.J. Xiu, L.D. Mao, P.Z. Ying, L. Jiang and X. Qian *Page 1158*



The temperature dependence of dimensionless thermoelectric figure of merit *ZT* for different $(Bi_2Te_3)_{0.9}$ - $(Bi_{2-x}Ag_xSe_3)_{0.1}$ (x=0-0.4) alloys prepared by spark plasma sintering.

Erratum

Erratum to "CONTENTS" [J. Solid State Chem. 179(12) (2006)]

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