08-Inorganic and Mineralogical Crystallography

(Pb, a = 13.477(2), b = 7.015(1), c = 26.323(2) Å, β = 92.828(8)°, Z = 8, 2292 reflections, 303 variables, R = 0.051, R1 = 0.071) in vacuo loose the boron-coordinated solvent molecule; crystals of the borondane

(C2/c, a = 11.234(2), b = 10.133(1), c = 7.507(1) Å, β = 118.133(8)°, Z = 4, 649 reflections, 64 variables, R = 0.050, R1 = 0.073) are obtained. Vacuum treatment of single crystals with known orientation matrices reveals that reorientation of the molecules occurs and that the solid state reaction is entirely reconstructive.

PS-08.04.13 13C ISOPE EFFECT ON DIAMOND STRUCTURE. By T. Yamamaki1, S. Morimoto1 and S. Sasaki
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Diamond with 13C isotope doped has been paid a large attention for the new material with the enormous extension of thermal conductivity. The influences of the isotope ratio on the lattice parameter and structure of diamond were investigated by X-ray diffraction study using four-circle diffractometer and postion synchrotron radiation (2.5GeV 30bnA) at Photon Factory, Tsukuba together with Raman spectroscopy. A finely monochromated radiation of λ = 0.697148 ± 0.0000012Å was used as an incident beam.

Single-crystals of diamonds composed of various ratios of 12C and 13C isotopes were prepared by high pressure apparatus. The specimens with the size of about 20μm in diameter were used for the diffraction studies. The isotope concentrations in the synthesized diamonds were analyzed by secondary ion mass spectroscopy (SIMS).

The isotope dependence on the lattice constant a of diamond is expressed by a(Å) = 3.56712 × 10⁻³ × 12C / (3.56712 × 10⁻³ × 13C). The variation of the lattice constant with the isotope ratio, 12C/13C is in the same fashion as those of 14N/15N, 30Si/31Si and 74Ge/72Ge. All isotopes having a greater atomic weight in each element are characterized by the smaller lattice constant. The present equation is different from a linear function reported by Holloway et al., (1991).

Influence of the nuclear weight on the valence electron density is examined by the refinement of k-parameter in ψ(kr). After extinction and absorption correction, the least-squares refinements converged within less than R = 0.01.

In order to elucidate the isotope dependence on anharmonic thermal vibration of atoms, refinements of thermal parameters have been made by 3-dimensional Gram-Charlier expansion up to 4th order parameters of the trivariate Gaussian probability density function. Anharmonic potentials along with the directions <100>, <110> and <111> of 12C and 13C diamond have been also evaluated.

Raman peak shift of F2g proved that the lattice vibration is varied with the isotope mass difference between 12C and 13C and with the force constant. The shift is not a linear relation with the reduced mass but is represented by a binary equation. This relation is consistent with the lattice constant variation mentioned above. It has been clarified that the lattice vibration gives an effect to the thermal atomic displacement and the thermal conductivity induced from the phonon dispersion.

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PS-08.04.14 CRYSTAL STRUCTURE OF SOME CATALYTIC PRECURSORS WITH Pt-Ir COMPLEXES. By E. Ganiere
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Multinuclear formulations are currently used to improve the properties of metal catalysis. We grew Pt-Ir complexes by diffusion of Ir(NH3)5Cl2+ and PtCl62+ in teiraethoxyxylene or sodium metaslicate gel. Five precursors have been prepared: (A) Ir(NH3)5Cl2PtCl4, (B) Ir(NH3)5Cl2PtCl6, (C) Ir(NH3)5Cl2IrCl6, (D) Pt(NH3)3IrCl5Cl2 and (E) Ir(NH3)3IrCl5Cl2O. 2 H2O. These precursors are crystallised in the porosity of aluminia and reduced with H2 to get the bimetallic aggregates. (A) crystallises with nearly octahedral b(NH3)5Cl2+ cations and planar PtCl62- anions. The coordination of the cations is 6/6. The structure of (C) is constituted of Cl+ and PtCl62- anions and Ir(NH3)5Cl2+ cations. It can be regarded as ion layers parallel to (001) and successively one PtCl62- layer and two IrCh layers, each of these two layers containing Cl+ and Ir(NH3)5Cl2+. (E) is a layered compound. Each layer, parallel to (001) is octahedrally. These structures present an extended hydrogen bonding network involving the amine donors and the chloride ions.

(111)Ir(NH3)5Cl2PtCl4, Pnma
a = 16.5835(2)Å, b = 8.1113(1)Å, c = 9.4582(5)Å
Z = 4, R = 0.0392.

(111)Ir(NH3)5Cl2PtCl6, C2/m,
\( a = 12.343(2)Å, b = 7.894(2)Å, c = 13.406(2)Å, \beta = 89.44(2)°\)
Z = 2, R = 0.0090.

(E): Pt(NH3)3IrCl5(H2O), 2 H2O, \( \beta = 144(2)°\)
\( a = 15.555(2)Å, b = 23.554(3)Å, c = 16.70(2)Å\)
Z = 16, R = 0.0404.