

# Kristallstruktur, Infrarot- und Ramanspektren sowie thermische Zersetzung von Magnesiumtetrahydrogendimesoperiodat, $\text{MgH}_4\text{I}_2\text{O}_{10} \cdot 6\text{H}_2\text{O}$

Crystal Structure, Infrared and Raman Spectra and Thermal Decomposition of Magnesium Tetrahydrogen Dimesoperiodate,  $\text{MgH}_4\text{I}_2\text{O}_{10} \cdot 6\text{H}_2\text{O}$

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*Herrn Prof. Dr. Ch. Balarew zum 65. Geburtstag gewidmet*

Z. Naturforsch. **54 b**, 999–1008 (1999); eingegangen am 16. April 1999

Magnesium Periodate Hydrate, Zinc Periodate Hydrate, Dimesoperiodate Ions,  
Crystal Structure, Raman Data

Crystal structure, DRIFT, infrared and Raman spectra, and the results of thermal analyses of the hitherto wrongly as  $\text{Mg}(\text{H}_4\text{IO}_6)_2 \cdot 4\text{H}_2\text{O}$  and  $\text{Mg}(\text{IO}_4) \cdot 8\text{H}_2\text{O}$  described dimesoperiodate  $\text{MgH}_4\text{I}_2\text{O}_{10} \cdot 6\text{H}_2\text{O}$  and of the isostructural zinc compound are presented. The compounds crystallize in the monoclinic space group  $\text{P2}_1$  ( $Z = 2$ ) with  $a = 1071.0(2)$ ,  $b = 547.0(1)$ ,  $c = 1194.9(2)$  pm, and  $\beta = 112.58(3)^\circ$  and  $a = 1073.3(3)$ ,  $b = 545.3(2)$ ,  $c = 1188.3(5)$  pm, and  $\beta = 112.52(3)^\circ$ , respectively. The structure, which was refined from X-ray single crystal data of the magnesium compound ( $R1 = 2.72\%$ , 3824 independent reflections), is built up from isolated distorted  $M(\text{H}_2\text{O})_6^{2+}$  octahedra and dimesoperiodate ions  $\text{H}_4\text{I}_2\text{O}_{10}^{2-}$  connected by a network of hydrogen bonds formed by the  $\text{H}_4\text{I}_2\text{O}_{10}^{2-}$  ions and six crystallographically different hydrate  $\text{H}_2\text{O}$  molecules. The strength of the hydrogen bonds ranges from unusually weak bonds corresponding to uncoupled (isotopically dilute samples) OD stretching modes of  $> 2600 \text{ cm}^{-1}$  and very strong ones ( $\nu_{\text{OD}}: < 2200 \text{ cm}^{-1}$ ). The IO stretching modes of the *trans*-configured  $\text{H}_4\text{I}_2\text{O}_{10}^{2-}$  ions are assigned to terminal I-O groups ( $816 \text{ cm}^{-1}$ ), I-OH groups ( $746$  and  $762 \text{ cm}^{-1}$ ) and bridging I-O groups ( $618$  and  $647 \text{ cm}^{-1}$ ). On heating,  $\text{MgH}_4\text{I}_2\text{O}_{10} \cdot 6\text{H}_2\text{O}$  undergoes dehydration in the range of 373 - 485 K (Differential Scanning Calorimetry) to two different polymorphs of magnesium metaperiodate ( $\text{H}_4\text{I}_2\text{O}_{10}^{2-} \rightarrow 2\text{IO}_4^- + 2\text{H}_2\text{O}$ ). Anhydrous  $\text{Mg}(\text{IO}_4)_2$  is instable. Above 423 K (high-temperature Raman data), it decomposes to magnesium iodates.