
A discussion is made of crystal structures of tetratetramethylammonium $\text{Si}_4\text{O}_{15}\cdot\text{H}_2\text{O}$ and tetratetramethylammonium $\text{Si}_4\text{O}_{15}\cdot\text{H}_2\text{O}$ silicates as well as of tetratetramethylammonium aluminiumsilicate $\text{Si}_4\text{Al}_2\text{O}_{12}\text{(OH)}_8\cdot\text{H}_2\text{O}$ determined by X-ray diffraction at -100°C. In the structure of TMA silicate the silicon-oxygen radical in the form of double four-membered rings $\text{Si}_4\text{O}_{15}$ is built up of silicon-oxygen tetrahedra 15 surrounded by six TMA groups which directly adjoin the anion. The nitrogen atoms of the tetratetramethylammonium radical are situated between the layers, being octahedrally coordinated by Cl ions. The second reason is the polarization effect: the extra polarization of the bromine made $\text{Rb}_{11}\text{InBr}_3$ does not form infinite chains of $\text{Rb}_{11}\text{Br}_3$. The second reason is the polarization effect: the extra polarization of the bromine made $\text{Rb}_{11}\text{InBr}_3$ does not form infinite chains of $\text{Rb}_{11}\text{Br}_3$.

08.2-22 THE ACCOUNT OF THE STRUCTURAL DIFFERENCE BETWEEN $\text{Na}_2\text{C}_6\text{H}_5\text{O}_7$ AND $\text{RbMnBr}_3$ by E.M. Ali

Faculty of Science, King Saud University, Riyadh, Saudi Arabia.

The crystal structure of $\text{RbMnCl}_3$ was determined by the author and others (Acta Cryst. (1977) B33, 256) to be hexagonal $P6_3/mmc$, $a=9.589(2)$, $c=11.193(3)$, $Z=10$. The compound has been identified definitely as $\text{Rb}^+\text{In}^3_2\text{Cl}_6\text{H}_2\text{O}$. Two of the six protons of decavanadic acid are captured by two HMT molecules, forming the protonated cation (HMT+). The remaining two are kept by acid group itself to form the protonated species (H$_3$V$_{10}$O$_{28}$)$^3+$. Both the interesting protonated cation and anion have been confirmed by several evidences, such as the significant lengthening effect of the N-O bonds during the attachment of proton to N atom of HMT. Due to the good qualities of the crystals, it is thought to be possible to utilize this kind of adduct to separate vanadium from other elements.


By using $\text{NH}_4\text{VO}_3$, NaOH and HMT (hexamethylenetetramine) in aqueous solution with pH=6, we have got the title adduct which crystallizes in $P\overline{1}$ with cell constants $a=9.589(2)$, $b=10.486(2)$, $c=11.193(3)$, $\alpha=103.27(2)^\circ$, $\beta=96.20(2)^\circ$, $\gamma=103.16(2)^\circ$ and $D_2.29$ g.cm$^{-3}$. Intensities of 2931 independent reflections were collected with MoK$_\alpha$ radiation. The coordinates of all V atoms were found from successive Fourier and difference syntheses. The block-digonal least-square refinement for all atomic parameters gave a final discrepancy factor $R=0.0273$. The compound has been identified definitely as $\text{(Na}^+\text{)}_2(\text{H}_3\text{V}_{10}\text{O}_{28})^3+\cdot(\text{C}_6\text{H}_5\text{O}_7)^{2-}\cdot\text{H}_2\text{O}$. Two of the six protons of decavanadic acid are captured by two HMT molecules, forming the protonated cation (HMT+), two are replaced by Na$^+$ and the remaining two are kept by acid group itself to form the protonated species (H$_3$V$_{10}$O$_{28}$)$^3+$. Both the interesting protonated cation and anion have been confirmed by several evidences, such as the significant lengthening effect of the N-O bonds during the attachment of proton to N atom of HMT. Due to the good qualities of the crystals, it is thought to be possible to utilize this kind of adduct to separate vanadium from other elements.

08.2-24 BLEITHALOGENIDE UNgewöhnlicher Zusammensetzung. Von K.-F. Tebbe. Institut für Anorganische Chemie der Universität zu Köln, Greinstrasse 6, D-5000 Köln 41, Bundesrepublik Deutschland.

In der älteren Literatur beschriebene Halogen-Überschuß-Verbindungen des Blei werden röntgenkristallographisch und strukturell untersucht. Das variationsfähige Iodid von Wells [2, anorg. Chem. 346 (1893)] $\text{K}_3\text{PbI}_4\cdot4\text{H}_2\text{O}$ (P4/mmm, a = 13,183, c = 4.539 $\AA$, $Z = 1$) enthält parallel den Oktadekenteraten $[\text{PbI}_4]_{1/2}^2$-längs (0 0 1) durch Kaliumionen. Wassermoleküle und Triiodidionen teilweise fehlgeordnet gefüllte Kanäle. Die Schweratom-Teilstruktur des Iodids von Johnson [J. chem. Soc. 33, 183 (1878)] $\text{PbI}_3\cdot3\text{H}_2\text{O}$ (P21/n, a = 10,689 $\AA$, b = 106.31 $\AA$, c = 29,256 $\AA$, $Z = 4$) läßt einen schichtartigen Aufbau aus Bleiatomen erkennen, der durch Triiodidionen und Iod-Iod- Ketten $\cdot(\cdot\cdot\cdot\cdot\cdot)$ gestützt wird. Das Salz von Gröger (MH. Chem. 151 (1892)) $\text{Pb}_2\text{H}_3\text{O}_7$ (P21/n, a = 9.539 $\AA$, b = 12,293, c = 10.689 $\AA$, b = 106.31 $\AA$, Z = 4) enthält neben Polyido- Gruppen- und -Verbänden vermutlich Polyhydroxybekenaten. Mit der abschließenden Strukturanalyse dieser und der Darstellung, Untersuchung und strukturellen Charakterisierung weiterer Substanzen sind wir beschäftigt. In diesen Verbindungen gibt es bisher keine Hinweise auf eine Valenzmischung in der Blei-Teilstruktur. Der Halogenüberschuß wird stets in Polyhalogenid-Ionen gebunden. Diese Arbeit wurde von der Deutschen Forschungs- gemeinschaft durch Sachmittel unterstützt.