

s13.m35.p16 **Variable-Temperature Powder Diffraction Studies of the n-Alkylammonium Iodides.** G.J. Kruger<sup>a</sup>, M. Rademeyer<sup>a</sup> and D.G. Billing<sup>b</sup>, <sup>a</sup>Rand Afrikaans University, Box 524, Auckland Park 2006, South Africa, <sup>b</sup>University of the Witwatersrand, Private Bag 3, Wits 2050, South Africa. E-mail: gjk@na.rau.ac.za

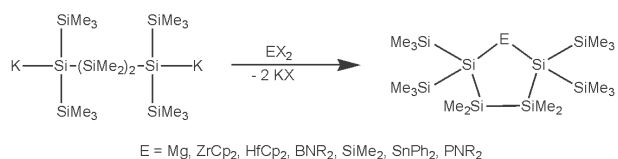
**Keywords:** Alkylammonium halides; Polymorphism; Powder diffraction

Long-chain n-alkylammonium halides exhibit polymorphism at room temperature and a complex sequence of solid-solid phase transitions at higher temperatures. The polymorphs of the n-alkylammonium iodides, with alkyl chain lengths between 11 and 18 carbon atoms, were studied by powder and single crystal X-ray diffraction and by thermal analysis techniques. All members of this series exhibit polymorphism. There exist five isostructural series, namely the *i*, *m*, *ε*, *b* and *γ* forms. The first three are similar to known structures observed in the analogous chlorides and bromides, but the last two are previously unknown forms. The crystal forms can be classified according to their long spacings as determined by powder diffraction. The *i* form has the smallest long spacing (because of the chain interdigitation), the *ε* form the biggest (not interdigitated) and the *m* form somewhere in between (interdigitated, not tilted). The crystal structure of the *i* form of n-octadecylammonium iodide, C<sub>18</sub>H<sub>37</sub>NH<sub>3</sub><sup>+</sup>I<sup>-</sup>, was determined by single crystal X-ray diffraction. The molecular chains are in the all-*trans* conformation, interdigitated and tilted relative to the ionic layer. Hydrogen bonding interactions are present between the ammonium groups and iodide anions in the ionic layer. The transition sequences between these forms and the high temperature phases could be determined by thermal analysis (DSC) and a phase sequence diagram constructed. Variable-temperature powder diffraction studies confirmed the transition sequences and proved that the *e* form consistently crystallizes from the melt.

s13.m35.p17 **Synthesis and Structural Aspects of Cyclosilanes.** Christoph Marschner and Judith Baumgartner, Institut für Anorganische Chemie, Technische Universität Graz, Austria. E-mail: marschner@anorg.tu-graz.ac.at

**Keywords:** Cyclosilanes; Heterocycles; Crystal structure

Our recent development of the chemistry of oligosilyldianions has brought about the opportunity to use these compounds for the synthesis of homo- and heterocyclic cyclopentasilanes.[1], [2] By reaction with some difunctional electrophiles a number of five-membered rings with heteroatoms ranging from magnesium to phosphorous could be obtained. Most of the solid state structures of these compounds could be elucidated by means of single crystal X-ray diffraction analysis.



In addition also some cyclohexasilanes could be obtained by the same type of chemistry.[3] A comparison of structural parameters of the studied five- and six-membered cyclosilanes will be given.

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