**PS07.00.17** SYNTHESIS AND STRUCTURE OF Ag-TMTMS COMPLEX: (Ag-TETRAMETHYLTHIURAMMONOSUL-FIDE) By M. Kawaminami<sup>\*</sup>, C. S. Mendoza and S. Kamata, Dept. of Phys. College of Liberal Arts<sup>\*</sup> and Dept of Applied Chemistry and Chemical Engineering, Kagoshima University, Kagoshima 890, JAPAN.

The silver selectivity of TMTMS using solvent extraction is studied. A 1g AgClO<sub>4</sub> was dissolved in a 50-ml acetone solution (90% acetone + 10% water) and mixed with a 2.5g TMTMS dissolved in a 50-ml acetone. After long standing, yellowish crystals suitable for X-ray analysis were formed, separated and washed with acetone. The data collection was made by using a CAD4 diffractometer, and the structure was solved by direct methods and refined in full-matrix least-squares. All calculations were performed on a VAX computer using MOLEN.

AgClO<sub>4</sub>(C<sub>6</sub>H<sub>24</sub>S<sub>3</sub>N<sub>2</sub>)<sub>2</sub>, M<sub>r</sub>=624.06, monoclinic,  $P2_1/n$ , a=13.9249(6)Å, b=9.9342(4)Å, c=18.0702(15)Å, ß=108.37(1)°, V=2372.3(2)Å<sup>3</sup>, Z=4, Dcal=1.75g/cm<sup>3</sup>,  $\lambda$ (Moka)=0.71073Å,  $\mu$ =14.9cm<sup>-1</sup>, w/2 $\theta$  scan, 2 $\theta$  max=60.9°, F(000)=1264, T=21°C, R= .043, wR= .057 for 4028 reflections.

Ag atom bonded with 4 S atoms of two molecules of the ligand forming a bidentate molecule, in which a center of symmetry are connected to each other making a ring of Agl-S-C-S-C-S-Ag2.



PS07.00.18 SYNTHESIS AND STRUCTURE OF ISOCYA-NIDE COMPLEXES OF CHROMIUM IN DIFFERENT OXI-DATION STATES. Gabriele Kociok-Köhn, Dirk Wössner, Alexander C. Filippou<sup>\*</sup>. Fachinstitut für Anorganische Chemie, Humboldt Universität, D-10115 Berlin, Germany

Group VI transition-metal isocyanide complexes have been recently shown to undergo a variety of carbon-carbon bond forming reactions (A. C. Filippou, W. Grünleitner, Z. Naturforschung, 46b (1991) 216). The chromium derivatives display a considerably different redox chemistry than the analogous molybdenum and tungsten compounds. In this context we have synthesized various isocyanide complexes of chromium in the oxidation state 0, I, II and III, in order to study the effect of the oxidation state on the nature of the metal-ligand bonding. We were able to obtain crystal structures of the complexes [CrCl<sub>3</sub>(CNtBu)<sub>3</sub>]·(CH<sub>2</sub>Cl<sub>2</sub>)<sub>3</sub> 1, [CrI<sub>2</sub>(CNiPr)<sub>4</sub>][I<sub>3</sub>] 2 and [Cr(CNtBu)<sub>6</sub>] [PF<sub>6</sub>] 3. All three structures feature six coordinated chromium. In complex 1 the three isocyanide ligands adopt a meridional arrangement around the Cr(III) centre (Cr-C: 2.087-2.104(3) Å). The unit-cell contains an extraordinary amount of CH2Cl2, which interacts via hydrogen bonds with the chloro ligands of the complex. In complex 2 the two I-ligands are trans-oriented giving rise to a tetragonally distorted octahedron with the four isocyanide ligands in the square plane (Cr-I: 2.649(10), Cr-C: 2.064(9) Å). The six t-butyl isocyanide ligands in complex 3 are bound to a Cr(I) centre, forming a slightly distorted octahedron (Cr-C: 1.977(5) and 1.981(4) Å).

**PS07.00.19** THE NEW THERMOSTABILITY CRITERION OF INDUSTRIAL PHLOGOPITE CRYSTALS. G.I. Kosmacheva. V.M Kalikhman, G.A. Kuznetsova, M.S. Metsik. Irkutsk State University, Irkutsk, Russia.

Crystal structure thermostability of industrial phlogopite has been investigated. The structural thermostability was estimated as a temperature of nonreversible decrease of basal interplanar distances  $d_{0012}$  at the heating during 30 min.14 various genetic type specimens of different chemical composition from Aldan and Carel-Colsky mica provinces have been studied.

The interplanar distances varied from 1,018 nm to 1,028 nm, the thermostability ranged between 1170-870 K with admixture changes of F 0,4-5,5%, Fe -2-9%, Ti -0,3- 0,8%. The most thermostable crystals (T=1170 K) contained iron less than 5%. The thermostability decreased to 870 K with the increase of the iron content, especially Fe<sup>3+</sup> even if the F-content was 4-5%.

Structure phlogopite thermostability has been experimentally determined to depend on iron concentration in the octahedral layer but not depend on fluorine content, which substituted the hydroxyl in mica lattice.

The octahedral substitution of Mg to  $Fe^{3+}$  resulted the formation of localized quasimuscovite defective domains with lower thermostability. The formation of these domains promoted the crystal dehydroxylating process and decreased the thermostability at the heating with the iron conctntration over 5%.

PS07.00.20 AMINO(THIO)- AND DIAMINOCARBENE COMPLEXES OF GOLD(I) AND (III). G.J.Kruger, H.G.Raubenheimer, P.J.Olivier and M.Desmet, Department of Chemistry and Biochemistry, Rand Afrikaans University, Johannesburg-Auckland Park 2006, South Africa

Gold(I) compounds tend to form aurate complexes upon reaction with 2-lithio-thiazolyl and 2-lithio-imidazolyl reagents. These complexes form mono- and bis(carbene) complexes of gold after electrophilic attack with H+ or CH<sub>3</sub>+. The following compounds were prepared and their crystal structures determined:

1. *bis[bis*(4-methylthiazolinylidene)gold(I)] tetrachloro zincate dichloro- methane-[Au(CNHCCH<sub>3</sub>=CHS)<sub>2</sub>]<sub>2</sub>ZnCl<sub>4</sub>.CH<sub>2</sub>Cl<sub>2</sub>

2. bis(3 - methylthiazolinylidene)gold(I)trifluoromethanesulphonate- [Au (CNCH<sub>3</sub>CH=CHS)<sub>2</sub>]CF<sub>3</sub>SO<sub>3</sub>

3.(1,3-dimethylimidazolinylidene) (methylimidazolinylidene) gold(I) trifluoromethanesulphonate-[Au(CNCH<sub>3</sub>CH=CHNCH<sub>3</sub>)-(CNHCH=CHNCH<sub>3</sub>)]CF<sub>3</sub>SO<sub>3</sub>

4. *bis*(1,3-dimethylimidazolinylidene)gold(III) dichloride trifluoromethane-sulphonate-[Au(CNCH<sub>3</sub>CH<sub>3</sub>CH<sub>2</sub>CHNCH<sub>3</sub>)<sub>2</sub>Cl<sub>2</sub>]CF<sub>3</sub>SO<sub>3</sub>

5. Chloro(3-methylbenzothiazolinylidene)gold(I) - AuCl(CNCH<sub>3</sub>C<sub>6</sub>H<sub>4</sub>S-*o*)

6. bis(3-methylbenzothiazolinylidene)gold(I)trifluoromethanesulphonate - [Au(CNCH<sub>3</sub>C<sub>6</sub>H<sub>4</sub>S-o)<sub>2</sub>]CF<sub>3</sub>SO<sub>3</sub>

In all of the gold(I) complexes the gold is linearly coordinated to the ligands, and the gold(III) complex is square planar, with Au-C distances of 1.92 to 2.13 Å. Short intermolecular Au...Au contacts of 3.19 to 3.51 Å occur in complexes 1, 2 and 5.