

400 16-Molecular Structure Determination by Methods other than Diffraction

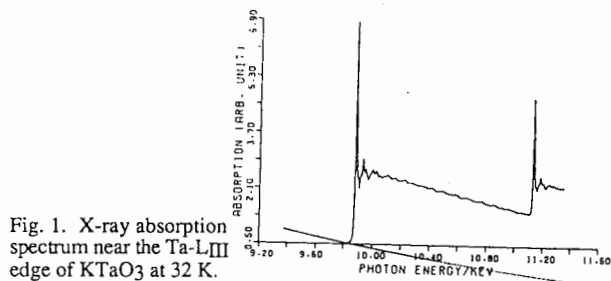


Fig. 1. X-ray absorption spectrum near the Ta-L_{III} edge of KTaO₃ at 32 K.

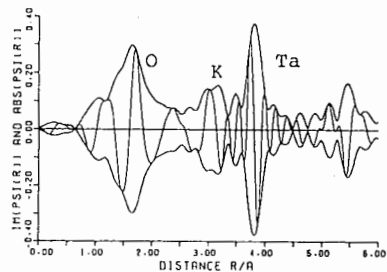


Fig. 2. Radial structure function around Ta atom at 32 K.

Harada, J., Axe, J. D. & Shirane, G. (1970). *Acta Cryst.* A26, 608.
 Maeda, H. (1987). *J. Phys. Soc. Jpn.* 56, 2777.
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PS-16.01.07 RELATIONSHIP BETWEEN CATALYTIC ACTIVITY FOR WATER-GAS SHIFT AND STRUCTURE OF Co-Mo-Mg/Al₂O₃ CATALYST
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The deactivation of two commercial Co-Mo-Mg/Al₂O₃ WGS catalysts has been studied by XRD, ICP, IR, SEM, XPS etc. The results showed that formation sulfates, impurities, regenerative conditions and mechanical strength of catalyst have significant effect on the activity except ratio of steam/gas, H₂S content and operating temperature.

The Co-Mo-Mg/Al₂O₃ catalyst was prepared by incipient wetness impregnation on a γ-Al₂O₃ with a Mg(NO₃)₂ and calcined at 900°C for 2h after drying. Then this same material was impregnated with a Co(NO₃)₂ and (NH₄)₆Mo₇O₂₄ mixed solution.

A typical γ-Al₂O₃ XRD pattern was given by Co-Mo-Mg/γ-Al₂O₃ catalyst A in the oxidic state (P. J. Gajardo, *Less-Common Met.*, 1977, 54, 311). This implied that cobalt and molybdenum as well as magnesium had been well dispersed on the support. Catalyst B showed three XRD patterns consistent with MgO, Al₂O₃ and MgAl₂O₄. The crystal phases MgO and MgAl₂O₄ in the catalysts led to a gradual drop in activity (J. P. R. Visser, *Bull. Chim. Bdg.*, 1984, 93, 813). The spinel MgAl₂O₄ structure formation was observed with increasing the calcination temperature, so it is correlated with the procedure of catalyst preparation.

The specific surface areas of oxide state catalyst (>150m²/g) decrease to about 100m²/g as the catalyst converted to sulfided state. It was about 70m²/g after the catalyst used for a year, and the catalyst mechanical strength decreased to 1/3 after regeneration, comparing with new one.

The Co, Mo and Mg in catalyst were not lost after it used for four years. The ICP results of catalyst A and B are shown in Fig. 1. There was a small part of impurity K⁺ and Na⁺ in catalyst lost. The inorganic impurities in feed gas are mainly Fe, Ca, Pb and K etc. the activity is decreased with its deposition on the surface of the catalyst and it is reconverted after regeneration.

The deactivation is mainly due to the formation of sulfates, increasing of MoS₂ and MgAl₂O₄, the active center Co-Mo-S is destroyed. over-heating in the regeneration, sulfidation and operation process enhance formation of inactive MgO, MgAl₂O₄ and crystal phase MoS₂. The higher ratio of steam/gas and the higher concentration of SO₂ etc enhance also the sulfates formation.

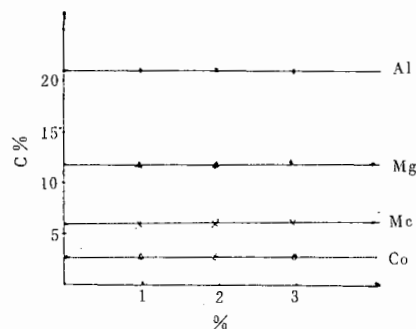


Fig. 1. ICP results of catalyst A and B.

PS-16.01.08 CRYSTAL STRUCTURE AND HALOGEN NQR OF DIAMMONIUMALKANE HALIDES. By Shi-qi Dou*, Helmut Paulus and Alarich Weiss, Institut für Physikalische Chemie, Technische Hochschule Darmstadt, Petersenstr.20, D-6100 Darmstadt, Germany.

The crystal structures of the isomorphous compounds 1,3-diammoniumpropyl- dibromide and -diiodide are reported. Monoclinic, P2₁/n, Z=4. Lattice constants in pm. (H₃N(CH₂)₃NH₃)²⁺ 2Br⁻: a= 1343.8(5), b=457.9(1), c=1347.1(5), β=109.14(1)°; (H₃N(CH₂)₃NH₃)²⁺ 2I⁻: a=1421.3(5), b=478.0(2), c=1419.6(5), β=110.10(1)°. The diammoniumpropyl cations are centrosymmetric and there are two crystallographic independent anions and two independent cations in the unit cell. The positive (H₃N(CH₂)₃NH₃)²⁺ cations and halogen anions are proportionate distributed in the lattice. Weak hydrogen bonds N-H...X-, X=Br, I are observed. The discrepancy in the ⁷⁹Br and ¹²⁷I NQR spectra, respectively, [1] from which isomorphism was denied, is removed by reinvestigation of the spectra. Additionally to the already known ⁹¹Br line at 22.95MHz a second line at 12.07MHz was found at room temperature. Structures and NQR spectra are compared and discussed.

[1]; J. Hartmann, S.-q. Dou, and Al. Weiss, *Z. Naturforsch.* 44a, 41 (1989)

PS-16.01.09 N-TRICHLORO- AND DICHLOROACETYL AMINO ACIDS AND COMPOUNDS OF AMINO ACIDS WITH HALOGENO ACETIC ACIDS. ⁵¹CL NUCLEAR QUADRUPOLE RESONANCE SPECTROSCOPY; CRYSTAL STRUCTURE OF N-TRICHLOROACETYL-GLYCINE, -DL-ALANINE, AND -L-ALANINE. By Shi-qi Dou*, Armin Kehrner, Armin R. Ofial, and Alarich Weiss, Institut für Physikalische Chemie, Technische Hochschule Darmstadt, D-6100 Darmstadt, Germany