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A Hydrazine Polyuranate — Preparation and X-Ray Diffraction Data

M. Komac, D. Kolar

Nuclear Institute »Jožef Stefan«, Ljubljana, Slovenia and

M. Sikirica

Institute for Inorganic and Analytical Chemistry, the University, Zagreb, Croatia, Yugoslavia

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The formation of a yellow precipitate upon addition of $50^{0/0}$ hydrazine water solution to a diluted solution of uranyl nitrate in water was observed by R. Salvadori back in 1912¹. Author reported that the precipitate obtained from slightly alkaline solutions contains uranium in 6 + valence state and gave the formula of precipitate as $(N_2H_5)_2O \cdot 5 UO_3 \cdot 8 H_2O$.

Our preliminary studies of reactions in the system $H_2O-N_2H_4-UO_2(NO_3)_2$ and some characteristics of the products were reported recently². In further work, the influence of preparation conditions on the chemical composition and X-ray structure of products was investigated.

Hydrazine polyuranate was quantitavely precipitated by the addition of 50% solution of hydrazine hydrate in water to dilute solution of uranyl nitrate (3 g $UO_2(NO_3)_2 \cdot 6 H_2O$ in 100 ml H_2O) at room temperature. The precipitate was filtered off, washed with methanol and ether and dried over P_2O_5 . Chemical analysis showed the composition close to $N_2H_4 \cdot 4 UO_3 \cdot 6 H_2O$. (Anal., found: U, 73.5; N, 2.10; H_2O , 9.25%; calc'd.: U, 74.1; N, 2.24; H_2O , 8.4%). The analytical procedure was the same as already described².

The above composition was the same regardless of the degree of precipitation and was not influenced by the rate of addition of hydrazine solution.

X-ray diffraction photographs were taken by means of a recording Philips diffractometer using Ni-filtered Cu K radiation. The obtained lattice parameters: a = 14.016, b = 16.688 and $c = 14.848 \pm 0.005$ Å were calculated using the CuK α wavelenght up to $2 \Theta = 40^{\circ}$. The higher angle reflections were calculated with the CuK α_1 wavelenght. Assuming that the unit cell contains eight molecules of $4 \text{ UO}_3 \cdot 6 \text{ H}_2\text{O} \cdot \text{N}_2\text{H}_4$, the X-ray density amounts to 4.91 g/cm^3 as compared with the pycnometrically determined 4.88 g/cm^3 .

Table I contains the comparison of the observed and calculated $\sin^2 \Theta$ values and their relative intensities for ten strongest reflections. The complete data may be obtained from the authors.

Since the obtained X-ray powder diffraction diagram of this compound is very much like the one β -UO₃ · 2 H₂O described by P. C. Debets and B. O. Loopstra³, we conclude that the sublattices of the heavy atoms (U) must be identical.

TABLE 1	ĉ
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Powder Diffraction Data for Ten Strongest Reflections

$\sin^2 \Theta imes 10^5$ (obs)	$sin^2 \Theta imes 10^5$ (calc'd.)	hkl	I (obs)
1075	1076	002	100
4309	4306	004	25
4611	4608 4617	$\frac{104}{240}$	81
4826	4821 4832	$\frac{114}{400}$	38
5682	5684 5693	$\begin{array}{c} 420\\ 242 \end{array}$	88
5902	5908	402	47
8923	8923	244	24
9143	9138	404	11
14288	$\begin{array}{c}14281\\14305\end{array}$	$\begin{array}{c} 640 \\ 246 \end{array}$	20
15368	$15357 \\ 15372$	642 426	17
	. 1000 B 1 B 1 B 1		

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IZVOD

Hidrazin poliuranat — priređivanje i rendgenska analiza

M. Komac, D. Kolar i M. Sikirica

Opisana je sinteza hidrazin poliuranata; kemijski sastav, određen analizom, odgovara formuli 4 UO3 · 6 H2O · N2H4. Spoj je rompski s dimenzijama elementarne ćelije: a = 14.016, b = 16.688, c = 14.848 (\pm 0.005) Å, Z = 8. Rendgenska analiza je ustanovila da su položaji U-atoma identični u $4 \text{ UO}_3 \cdot 6 \text{ H}_2\text{O} \cdot \text{N}_2\text{H}_4$ i u β -UO₃ · 2 H₂O.

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A Simple Device for Measuring the Direct Piezoelectric Effect by the Static Method

M. Topić

Institute »Ruđer Bošković«, Zagreb, Croatia, Yugoslavia

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The static method for measuring piezoelectric properties is the oldest method used for the determination of piezoelectric strain constants. We quote only a few among numerous papers in which the static method has been described¹⁻³. Recently, this method has been replaced by more reliable dynamic and quasistatic measurements. However, for the measurement of the properties of polarized ceramics with high resistance it is possible to apply successfully the static method too. This method offers several advantages, such as simplicity and promptness in measuring. Owing to these advantages the static method is sometimes used for routine measurements⁴.

A simple device which has been constructed for the determination of piezoelectric strain constants by measuring the direct effect under the static conditions is described in this note.

Fig. 1 represents the device. The specimen to be measured C is placed into a brass cell of double walls A. The thermostating of the cell is carried out by

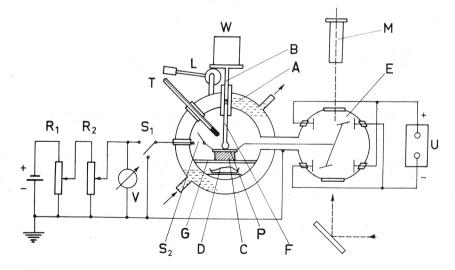


Fig. 1. Diagram of the device for measuring the direct piezoeffect under static conditions. (B) battery, (R_1) (R_2) potentiometers 5 k Ω , (V) compensating voltmeter, (S_1) switch, (S_2) switch pneumatically operated from the outside, (D) dessicant, (C) specimen, (B) and (F) force transmission axies, (W) weight, (A) cell for thermostating the specimen, (P) brass plate, (G) base for placing the specimen, (L) device for lifting and lowering the weight, (T) thermometer, (E) Lindemann electrometer, (M) microscope, (U) supply unit.

circulation of thermostated water. A thermometer T is used for the control of temperature. In order to keep the atmosphere dry, there is a small vessel D with a dessicant at the bottom of the cell. P_2O_5 is usually used at lower temperature, whereas a molecular sieve is used at higher temperature. The source of force is a weight W. The axle for force transmission consists of two parts - the upper part B and the lower one F. A ball is placed between the two parts. During measurements the weight W is lifted and lowered together with the part B, whereas the part F is left lying on the specimen. In that way the capacitance of the system remains unchanged, and undesirable displacements of the specimen are avoided. The end of the stick F is insulated by a teflon ball. In order to lift and lower the weight equally, a special device L is used, consisting of an eccentric wheel and a handle.

A Cambridge Lindemann electrometer E is used for measuring the potential difference. The electrometer is mounted directly onto the cell A. The whole capacitance of the cell (without specimen) and electrometer is 11 pF. This capacitance is valid when the distance between the plate P (2.9 cm in diameter) and the base G amounts to 0.18 cm. The sensitivity of the electrometer is 0.1 V. A microscope M is used for reading the needle deflection of the electrometer. U represents a stabilized supply unit for supplying the plates inside the electrometer. The device is gauged by means of the battery B, resistances R_1 and R_2 , compensating voltmeter V and switches S_1 and S_2 . The switch S_2 is a pneumatically operated switch »Cambridge 42391«. It is operated from the outside by pressing a rubber bulb connected to the switch by a rubber tube. The switch is constructed in such a way that there is practically no change in its capacitance during switching.

The described device can be used in the temperature region from 15° C to 80° C. A specimen of about 4.5 cm in diameter and 3 cm in thickness can be placed in this cell, and a force of up to 5 kp can be applied. The instrument is built and used for measuring the piezoelectric strain constant d_{aa} of polarized specimens prepared from a mixture of $Pb(Zr, TI)O_3$ ceramic and bakelite⁵. The cell for holding the specimen is also suitable for other electrical measurements.

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IZVOD

Uređaj za mjerenje direktnog piezoefekta statičkom metodom

M. Topić

Opisuje se jednostavan uređaj za mjerenje direktnoga piezoefekta u statičkim uvjetima. Uređaj je adaptiran za mjerenje pod termostatskim uvjetima na temperaturi od $+15^{\circ}$ do $+80^{\circ}$ C uz upotrebu sile do 5 kp. Za mjerenje razlike potencijala koristi se Lindemannov elektrometar osjetljivosti 0.01 V. Ukupni kapacitet uređaja bez uzorka za mjerenje, iznosi oko 11 pF.

INSTITUT »RUĐER BOŠKOVIĆ« ZAGREB

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