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Note

Crystal Data for $K_2[MoO(O_2)_2C_2O_4]$ and $K_2[WO(O_2)_2C_2O_4]$

M. Šljukić, N. Vuletić**, B. Matković, and B. Kojić-Prodić*

Institute »Ruđer Bošković«, Zagreb, Croatia, Yugoslavia

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Preparation, earlier literature and spectroscopic studies on these compounds were already described[1]. R. Stomberg announced recently[2] the crystal structure of $K_2[MoO(O_2)_2C_2O_4]$. We started independently on structural investigations of both compounds and now report their crystal data.

Preparation of the Crystals

The crystals were obtained from the reaction mixture of molybdates or tungstates (2.3 g K_2MoO_4 or 3.7 g K_2WO_4 in 30 ml water), oxalic acid (1.3 g in 20 ml water) and hydrogen peroxide (8—10 ml 30% H_2O_2) by slow evaporation of this solution at the temperature of 5—8° C. Yellow crystals of $K_2[MoO(O_2)_2C_2O_4]$ or colourless and transparent crystals of $K_2[WO(O_2)_2C_2O_4]$ were separated from the mother liquor, washed with 96% ethanol and dried 24 hours over silica gel. The composition of obtained crystals was proved by analyses (Table I). Compound was dissolved in hydrochloric acid and potassium was determined by the flame photometric method; molybdenum and tungsten as lead molybdate[3] and tungsten trioxide[4]. Peroxides were determined by the iodide method[5,6] and oxalates from the difference after titration (together with peroxides) with cerium(III) sulphate; carbon was also confirmed by standard micro-chemical method.

TABLE I

Analyses

Compound		(1) % Mo (2) % W	% K	% O_2^{2-}	% $C_2O_4^{2-}$	% C
(1) $K_2[MoO(O_2)_2C_2O_4]$	Found	28.05	22.96	18.04	26.15	7.13
	Calc.	28.039	22.856	18.704	25.725	7.021
(2) $K_2[WO(O_2)_2C_2O_4]$	Found	43.04	18.50	14.56	20.37	5.82
	Calc.	42.749	18.184	14.881	20.466	5.586

Crystal Data

Crystal data, as determined from the analysis of rotation and Weissenberg photographs, are listed in Table II. Ni-filtered Cu radiation was used. The densities were determined pycnometrically using decalin as liquid. Systematic

Permanent address:
Institute of Physics* and Institute for Agriculture, Laboratory for Agropedology**, Sarajevo, Bosnia and Herzegovina, Yugoslavia

TABLE II

Crystal Data

	a (Å)	b (Å)	c (Å)	β	V (Å³)	Z	Space group	D_m (g · cm⁻³)	D_x (g · cm⁻³)
$K_2[MoO(O_2)_2C_2O_4]$	13.72 ± 0.01	8.846 ± 0.008	6.893 ± 0.007	92° 44′ ± 05′	835.63	4	$P2_1/n$	2.67	2.72
$K_2[WO(O_2)_2C_2O_4]$	13.66 ± 0.01	8.894 ± 0.008	6.937 ± 0.007	93° 05′ ± 05′	841.38	4	$P2_1/n$	3.37	3.396

absences of reflexions occur for $h0l: h + l = 2n + 1$ and $0k0: k = 2n + 1$. These uniquely determine the space group as $P2_1/n$.

Similar values of lattice constants and the same space group extinctions for both compounds suggest either isomorphism or close relationship of their structures.

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IZVOD

Kristalografski podaci za $K_2[MoO(O_2)_2C_2O_4]$ i $K_2[WO(O_2)_2C_2O_4]$

M. Šljukić, N. Vuletić, B. Matković i B. Kojić-Prodić

Opisana je preparacija monokristala $K_2[MoO(O_2)_2C_2O_4]$ i $K_2[WO(O_2)_2C_2O_4]$. Sastav dobivenih kristala potvrđen je analizom (tabela I). Kristalografski podaci za oba spoja izneseni su u tabeli II. Budući da spadaju u istu prostornu grupu $(P2_1/n)$ i imaju veoma slične dimenzije elementarnih ćelija, međusobno su izomorfni ili imaju sličan strukturni raspored.

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