Crystal Data for $K_2[MoO(O_2)C_2O_4]$ and $K_2[WO(O_2)C_2O_4]$

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Preparation, earlier literature and spectroscopic studies on these compounds were already described. R. Stomberg announced recently the crystal structure of $K_2[MoO(O_2)C_2O_4]$. 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 $K_2[MoO(O_2)C_2O_4]$ or colourless and transparent crystals of $K_2[WO(O_2)C_2O_4]$ 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 and tungsten trioxide. Peroxides were determined by the iodide method and oxalates from the difference after titration (together with peroxides) with cerium(III) sulphate; carbon was also confirmed by standard micro-chemical method.

<table>
<thead>
<tr>
<th>Compound</th>
<th>(1) % Mo (2) % W</th>
<th>% K</th>
<th>% $O_2^-$</th>
<th>% $O_2^-$</th>
<th>% C</th>
</tr>
</thead>
<tbody>
<tr>
<td>(1) $K_2[MoO(O_2)C_2O_4]$</td>
<td>Found 28.05 22.96 18.04 26.15 7.13</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Calc. 28.039 22.856 18.704 25.725 7.021</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>(2) $K_2[WO(O_2)C_2O_4]$</td>
<td>Found 43.04 18.50 14.56 20.37 5.82</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Calc. 42.749 18.184 14.881 20.466 5.586</td>
<td></td>
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</tr>
</tbody>
</table>

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

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TABLE II
Crystal Data

<table>
<thead>
<tr>
<th></th>
<th>a (Å)</th>
<th>b (Å)</th>
<th>c (Å)</th>
<th>β</th>
<th>V (Å³)</th>
<th>Space group</th>
<th>D_m (g·cm⁻³)</th>
<th>D_x (g·cm⁻³)</th>
</tr>
</thead>
<tbody>
<tr>
<td>K₂[MoO(O₂)₂C₂O₄]</td>
<td>13.72 ± 0.01</td>
<td>8.846 ± 0.008</td>
<td>6.893 ± 0.007</td>
<td>92° 44' ± 05'</td>
<td>835.63</td>
<td>4</td>
<td>P2₁/n</td>
<td>2.67</td>
</tr>
<tr>
<td>K₂[WO(O₂)₂C₂O₄]</td>
<td>13.66 ± 0.01</td>
<td>8.894 ± 0.008</td>
<td>6.937 ± 0.007</td>
<td>93° 05' ± 05'</td>
<td>841.38</td>
<td>4</td>
<td>P2₁/n</td>
<td>3.37</td>
</tr>
</tbody>
</table>

Absences of reflexions occur for h0l: h + l = 2n + 1 and 0k0: k = 2n + 1. These uniquely determine the space group as P2₁/n.

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

REFERENCES
4. Ibid., p. 688.

IZVOD
Kristalografski podaci za K₂[MoO(O₂)₂C₂O₄] i K₂[WO(O₂)₂C₂O₄]
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Opisana je preparacija monokristala K₂[MoO(O₂)₂C₂O₄] i K₂[WO(O₂)₂C₂O₄]. 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 (P2₁/n) i imaju veoma slične dimenzije elementarnih čelija, međusobno su izomorfnii ili imaju sličan strukturni raspored.

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ZAGREB

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