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D.H. Templeton, A. Zalkin, H. Ruben, and L.K. Templeton

April 1985

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Redetermination and Absolute Configuration of Sodium Uranyl Acetate

By David H. Templeton, Allan Zalkin, Helena Ruben
and Lieselotte K. Templeton

Materials and Molecular Research Division,
Lawrence Berkeley Laboratory and
Department of Chemistry, University of California,
Berkeley, California 94720, USA

Abstract. Sodium tris(acetato)dioxouranate(1-), NaUO₂(C₂H₃O₂)₃, Mᵣ =
470.15, cubic, P2₁ 3, a = 10.689(2) Å, V = 1221.3 Å³, Z = 4, Dₘ = 2.562, Dₓ =
2.557(2) g cm⁻³, Mo Kα, λ(α₁) = 0.70930 Å, μ = 126.5 cm⁻¹, F(000) = 848, T =
294 K, R = 0.021 for 1195 unique reflections. A determination of the
anomalous scattering term f'' for uranium and Mo Kα radiation gives 9.7(2)
e/atom. Each uranyl ion [U-O(avg.) = 1.758(3) Å] lies on a 3-fold axis and
is surrounded equatorially by six oxygen atoms of three acetate groups [U-O
(avg.) = 2.464(2) Å]. The absolute configuration determined by the anomalous
X-ray scattering is correlated with the sign of the optical activity.

Introduction. The structure of sodium uranyl acetate was studied by
Fankuchen (1935) and was determined more completely by Zachariasen &
Plettinger (1959). We studied it again to obtain more accurate parameters
for use in the analysis of experiments with synchrotron radiation (Templeton
& Templeton, 1982). The purpose of these experiments was to measure
anomalous scattering terms and the effect of polarization on them.
Incidental to this work we determined that the absolute configuration of the structure, if described in a right-handed coordinate system with the atomic parameters reported by Zachariasen & Plettinger, is that of a crystal which rotates the plane of polarization of visible light to the left.

**Experimental.** Crystals were made by dissolving reagent-grade uranyl acetate and sodium acetate in water, followed by slow evaporation. Crystal with 18 faces of forms \{111\}, \{11\} and \{110\}, 0.11 x 0.11 x 0.23 mm. \(D_m\) is taken from Fankuchen (1935). Picker FACS-I diffractometer, graphite monochromator, \(\theta-2\theta\) scan; cell dimension from 12 reflections \(42^\circ < 2\theta < 48^\circ\); analytical absorption correction, range 2.70 to 3.64; max. \((\sin\theta)/\lambda = 0.705\) \(\AA^{-1}\), \(h\) 0 to 15, \(k\) -15 to 15, \(l\) -15 to 0; three standard reflections, \(\sigma = 1.3, 1.0, 1.4\%\), no correction for decay; 3922 data, 1207 unique (including 12 observed less than background), \(R_{int}(I) = 0.030\); structure from Zachariasen & Plettinger (1959) refined on \(F\), 62 parameters including \(f''\) of \(U\), anisotropic thermal parameters for all atoms except \(H\), \(H\) atoms found in \(\Delta F\) map and refined with isotropic thermal parameters and subject to restraints on \(H-H\) and \(C-H\) distances, \(R = 0.021\) for 1195 reflections, \(wR = 0.015\), \(S = 1.01\), \(w = [\sigma(F)]^{-2}\), derived from \(\sigma^2(F^2) = [(\sigma^2(F^2), counting statistics only, + (0.014 F^2)^2]\); max \(\Delta/\sigma = 0.10\); max empirical isotropic correction for extinction 10% of \(F\); max. and min. of \(\Delta F\) synthesis 0.6 and -0.6 \(e\) \(\AA^{-3}\); atomic \(f\) including dispersion for neutral \(U\), \(Na\), \(O\), \(C\) and spherical bonded \(H\) from International Tables for X-ray Crystallography (1974); local unpublished programs and ORTEP (Johnson, 1965). Optical activity (positive rotation of 2 or 3°) was easy to observe in a well-formed crystal of thickness 1.5 mm with a polarizing microscope illuminated with white light. This crystal was too large for reliable observation of the signs of Bijvoet differences. A small fragment was broken from a corner.
Its diffraction intensities for several Bijvoet pairs, selected to be sensitive to configuration, were consistently reversed from those observed and calculated for the crystal used in the structure determination. Atomic parameters are listed in Table 1.*

*Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Lending Division as Supplementary Publication No. (7 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Discussion. This work confirms, with about a 10-fold improvement of precision, the structure which was determined in projection by Zachariasen & Plettinger (1959). Each uranyl is complexed with six oxygen atoms from three acetate ions (Fig. 1 and Table 2), giving the uranium atom the hexagonal-bipyramidal coordination which is commonplace for uranyl salts. The thermal motion of the methyl carbon atom, C(2), is quite anisotropic, with principal rms amplitudes of 0.18, 0.21 and 0.38 Å. The largest amplitude is nearly perpendicular to the plane of the acetate ion. The other acetate atoms exhibit excess motion in the same direction, but to a lesser extent. This motion explains the short C(1)-C(2) bond length; after correction according to the riding model, it is quite normal (Table 2).

Zachariasen & Plettinger (1959) used the uranyl bond distance in this compound (1.71(4) Å) to revise upward to 1.70 Å the end point of a curve of uranium-oxygen bond lengths vs bond strength. The present results (ave. = 1.758(3) Å) are more in line with other recent values for "bond strength -
2.00" structures, and some further revision of the curve is suggested. A few examples are 1.75(2) Å in bis(tetrahydrofuran)dioxouranium(IV) nitrate (Reynolds, Zalkin & Templeton, 1977), 1.76(1) Å in pentakis(urea)dioxouranium(VI) nitrate (Zalkin, Ruben & Templeton, 1979), and 1.746(4) Å in rubidium uranyl nitrate (to be reported elsewhere). All these values are listed without any correction for thermal motion.

The chiral nature of the molecular environment in this crystal is evident in Fig. 1, which depicts the absolute configuration which produces negative optical rotation. To associate the present atomic coordinates with another specimen, two steps are required: correct designation of axes a, b, and c, and determination of the enantiomer. There are two distinct ways in which right-handed axes can be assigned to a specimen. They can be distinguished by the relative intensities of reflection pairs hko, h0k (e.g., I(720)/I(702) = 52 for our setting) which are independent of the enantiomer. This ratio is inverted for the other setting. Then the handedness can be determined by Bijvoet pairs such as I(712)/I(7,1,-2) = 1.58, I(721)/I(7,2,-1) = 1.26 (for coordinates of Table 1), or by observation of optical rotation.

This data set permits a good determination of f" for uranium at Mo Kα; the result 9.7(2) e/atom is in agreement with 9.654 calculated by Cromer & Liberman (1970) for Mo Kα.

This work was supported by the National Science Foundation under grant No. CHE-8217443 and by the Director, Office of Energy Research, Office of Basic Energy Sciences, Chemical Sciences Division of the U.S. Department of Energy under Contract No. DE-AC03-76SF00098.
References


   Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)

   Laboratory, Tennessee.


Table 1. Fractional coordinates and equivalent isotropic thermal parameters (with e.s.d.'s in parentheses)

\[
B_{eq} = \frac{1}{3} \sum I_i I_j B_{ij} a_i^* a_j^* a_i a_j
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Table 2. Bond distances (Å) and angles (°)

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*Corrected for thermal motion according to the riding model.
Fig. 1. View of the structure down [111]. O(1) is below the central U atom, while O(2) and Na are above it. The central molecule has three more neighbors above it, generated by unit translations of the three depicted below it; e.g., the upper-left molecule shifted by c. Symmetry code: i, 1 - x, -1/2 + x, 1/2 - x; ii, -1/2 + x, 1/2 - x, 1 - x.
Fig. 1
Supplementary Material for:

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Supplementary Table 1. Fractional coordinates and isotropic thermal parameters for hydrogen atoms in sodium uranyl acetate

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Note: These atoms were refined subject to restraints on distances,
C(1)-H = 2.06(7) Å,
C(2)-H = 0.95(3) Å,
H-H = 1.55(5) Å.

Supplementary Table 2. Anisotropic thermal parameters for sodium uranyl acetate

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<th>B₂₂</th>
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Supplementary Table 3. Observed structure factors, standard deviations, and deltas for sodium uranyl acetate (next pages)
OBSERVED STRUCTURE FACTORS, STANDARD DEVIATIONS, AND DIFFERENCES (ALL X 4.0)

SODIUM URANYL ACETATE  MOKA

F(0,0,0) = 3227 (on this scale, including effect of anomalous scattering)

FOB AND FCA ARE THE OBSERVED AND CALCULATED STRUCTURE FACTORS.

SG = ESTIMATED STANDARD DEVIATION OF FOB.  DEL = |FOB| - |FCA|.

* INDICATES ZERO WEIGHTED DATA (observed less than background).

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