

+ 0.000835 F_o^2], giving acceptable agreement analyses; maximum and minimum $\Delta\rho = +0.64$ and $-0.21 \text{ e } \text{\AA}^{-3}$. Maximum shift/e.s.d. = 0.023 (*SHELX76*, Sheldrick 1976). The computations were made on a VAX 11/750 computer. Atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974).

Discussion. The refined atomic coordinates and isotropic temperature factors are presented in Table 1.* Bond distances and bond angles are given in Table 2. A *SNOOPI* (Davies, 1983) drawing of the molecular structure and the numbering of atoms are shown in Fig. 1. A number of structure determinations have been made on the two chair forms (CA and CE) of 1,3,2-dioxathiane 2-oxides and the results have been summarized by Geise, Lenstra, Petit, van Alsenoy & van Nuffel (1984). Interestingly, only one twist-boat conformation of a sulfite containing *tert*-butyl groups has been studied by X-ray analysis, namely, *cis*-4,*trans*-6-di-*tert*-butyl-5-chloro-1,3,2-dioxathiane 2-oxide (2) (Carbonnelle, Jeannin & Robert, 1978). The present X-ray structure determination of the title compound (1) was undertaken to enable comparison of geometrical differences arising from the substitution at C(5) changing from a chloro to dimethyl

groups. A selection of geometrical parameters is shown in Table 3.

Bond lengths and bond angles are quite similar for compounds (1) and (2), and most parameters lie within the range of values found for the CA/CE forms. The C—C bond length is apparently longer in twist-boat forms. One notable difference is the shortening of the S=O bond length in (1) compared with (2) and the CA/CE forms. The reason for this anomaly is not clear and with only two compounds to compare, further structural studies of related twist forms are required before any definite conclusions can be drawn.

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters, and bond lengths and angles involving H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53009 (13 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Ammonium Bismuth Chromate Dichromate Monohydrate

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Abstract. $\text{NH}_4\text{Bi}(\text{CrO}_4)(\text{Cr}_2\text{O}_7)\cdot\text{H}_2\text{O}$, $M_r = 577.01$, triclinic, $P\bar{1}$, $a = 6.916$ (2), $b = 7.249$ (3), $c = 10.798$ (3) \AA , $\alpha = 80.28$ (3), $\beta = 90.52$ (2), $\gamma =$

86.49 (3) $^\circ$, $V = 532.5$ (6) \AA^3 , $Z = 2$, $D_m = 3.58$ (1), $D_x = 3.598$ (4) Mg m^{-3} , $\text{Mo K}\alpha$, $\lambda = 0.71073$ \AA , $\mu = 19.938$ mm^{-1} , $F(000) = 528$, room temperature, $R =$

Table 1. Final atomic coordinates and equivalent isotropic temperature factors for NH₄Bi(CrO₄)(Cr₂O₇).H₂O

$$B_{\text{eq}} = (4/3) \sum_i \sum_j \beta_{ij} a_i \cdot a_j$$

	x	y	z	B _{eq} (Å ²)
N	0.870 (2)	0.015 (2)	0.313 (1)	2.3 (3)
Bi	0.34412 (7)	0.20675 (8)	0.07529 (6)	1.057 (9)
Cr(1)	0.8369 (3)	0.2462 (3)	0.9581 (2)	0.78 (4)
Cr(2)	0.3523 (3)	0.4710 (3)	0.7435 (2)	1.13 (4)
Cr(3)	0.5892 (3)	0.1930 (3)	0.6098 (2)	1.34 (4)
O(1)	0.919 (1)	0.235 (2)	0.822 (1)	2.2 (2)
O(2)	0.023 (1)	0.203 (1)	0.065 (1)	1.7 (2)
O(3)	0.676 (1)	0.077 (1)	0.000 (1)	1.4 (2)
O(4)	0.721 (2)	0.452 (1)	0.954 (1)	2.2 (2)
O(5)	0.618 (1)	0.332 (1)	0.215 (1)	2.0 (2)
O(6)	0.833 (2)	0.515 (2)	0.348 (1)	2.6 (3)
O(7)	0.676 (1)	0.703 (1)	0.136 (1)	1.6 (2)
O(8)	0.438 (1)	0.587 (1)	0.336 (1)	1.6 (2)
O(9)	0.565 (2)	0.034 (2)	0.734 (1)	2.2 (2)
O(10)	0.795 (2)	0.177 (2)	0.545 (1)	2.7 (3)
O(11)	0.587 (2)	0.814 (2)	0.489 (1)	2.6 (3)
O(w)	0.813 (1)	0.711 (2)	0.712 (1)	2.1 (2)

0.052 for 1942 observed reflections. The compound is isotypic with KBi(CrO₄)(Cr₂O₇).H₂O.

Experimental. The preparation of single crystals of NH₄Bi(CrO₄)(Cr₂O₇).H₂O has been reported in our previous publications (Cudennec, Lecerf, Riou & Gerault, 1981). Crystals were obtained from a mixture composed of 1 mol of NH₄Bi(CrO₄)(Cr₂O₇).H₂O and 4 mol of CrO₃ in a large quantity of water. After reaction, the system containing BiOHCrO₄ was slowly evaporated at room temperature over several days without stirring. Orange parallelepipedal crystals of NH₄Bi(CrO₄)(Cr₂O₇).H₂O appeared in the solution. A suitable crystal of 0.20 × 0.15 × 0.09 mm was mounted on an Enraf-Nonius CAD-4 diffractometer. Density was measured by immersion in bromobenzene at room temperature. The unit-cell dimensions were determined and refined on the basis of 25 carefully selected reflections in the angular range 5 < 2θ < 30°. Data were collected in the ω-2θ scan mode with ω-scan width (2.00 + 0.50 tan θ)° and slit amplitude (1.00 + 0.35 tan θ) mm. 2700 reflections were measured. The hkl range was 0 ≤ h ≤ 9, -9 ≤ k ≤ 9, -14 ≤ l ≤ 14 (2θ < 56°). A periodic check of three standard reflections showed no significant intensity variations. The internal consistency index, R_{int}, was 0.024. Reflections were merged to a unique set of 2563. 1942 reflections were considered as observed with I > σ(I). Data were corrected for Lorentz and polarization effects and absorption corrections were applied according to the procedure of Walker & Stuart (1983). The structural determination was carried out on a PDP 11/60 computer using the SDP

package (Frenz, 1978). Scattering factors were from Cromer & Waber (1965). The structure was determined on the basis of isomorphism with KBi(CrO₄)(Cr₂O₇).H₂O. Refinement was started with atomic coordinates reported by Riou, Gerault & Cudennec (1982). Full-matrix least-squares refinement was based on F and the function minimized was ∑w(|F_o| - |F_c|)², where w(F) = 1/σ²(F). The weighting scheme had a non-Poisson contribution with p = 0.050. Corrections were made for anomalous-dispersion and secondary-extinction effects (g = 4.0 × 10⁻⁷). During the last refinement cycle, the r.m.s. shift/e.s.d. was 0.07. The final difference Fourier map showed no peaks higher than 1.5 e Å⁻³. In these conditions, the final R was 0.052 and wR = 0.064. Final atomic coordinates, except H atoms, and equivalent isotropic temperature factors are given in Table 1.* The comparison between these atomic coordinates and those of the KBi(CrO₄)(Cr₂O₇).H₂O structure confirms their isomorphism.

Related literature. Little is known about chromate dichromate compounds. Such compounds containing Fe³⁺ or Al³⁺ ions were prepared some years ago, and their structures solved (Gerault & Bonnin, 1976; Cudennec, 1977).

The structure of a ternary compound containing bismuth has been published recently: Bi₂(CrO₄)₂(Cr₂O₇) (Gerault, Riou, Cudennec & Bonnin, 1987). The crystal structure of KBi(CrO₄)(Cr₂O₇).H₂O was reported by Riou *et al.* (1982).

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52978 (14 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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