

## Synthesis, Characterization and Crystal Structure of *trans*-Aqua-hydroxobis(2,2-dimethyl-1,3-propanediamine)chromium(III) Diperchlorate

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Chromium is the seventh most abundant element on earth.<sup>1</sup> It occurs in various oxidation states ranging from Cr(II) to Cr(VI) with trivalent and hexavalent states being the most stable and common in terrestrial environments. The coordination chemistry of chromium(III) complexes is relevant to third generation solar photoconversion scheme.<sup>2</sup> The geometrical isomerism and conformation in transition metal complexes with mixed ligands are very important in the medical application, and likely to be a major factor in determining the antiviral activity and its side-effect.<sup>3,4</sup> 2,2-Dimethyl-1,3-propanediamine (Me<sub>2</sub>tn, Fig. 1) is a bidentate ligand that can coordinate to the central metal ion by forming six-membered chelate ring with a chair conformation. The *trans*-[Cr(Me<sub>2</sub>tn)<sub>2</sub>(OH)(H<sub>2</sub>O)]<sup>2+</sup> can adopt either the *trans* or *cis* geometric isomer. In addition, two different kinds of conformation with respect to the chelate rings of Me<sub>2</sub>tn occur in the *trans* isomer, as shown in the bottom of Figure 1.

The carbon atoms of the two chelate rings of the two conformers may be on the same side (*syn* conformer) or on the opposite side (*anti* conformer) of the coordination plane. In the case of *trans-anti/syn*-[Cr(Me<sub>2</sub>tn)<sub>2</sub>Cl<sub>2</sub>]Cl, *trans-anti/syn*-[Cr(Me<sub>2</sub>tn)<sub>2</sub>Br<sub>2</sub>]<sub>2</sub>Br<sub>2</sub>·HClO<sub>4</sub>·H<sub>2</sub>O and *trans-anti/syn*-[Cr(Me<sub>2</sub>tn)<sub>2</sub>Br<sub>2</sub>]Br, independent *syn* and *anti* conformational isomers were found within the same crystals.<sup>5-7</sup>

However, structural analyses of *trans-anti*-[Cr(Me<sub>2</sub>tn)<sub>2</sub>(NCS)<sub>2</sub>]<sub>2</sub>NCS·1/2H<sub>2</sub>O, *trans-anti*-[Cr(Me<sub>2</sub>tn)<sub>2</sub>Cl<sub>2</sub>]ClO<sub>4</sub>, and

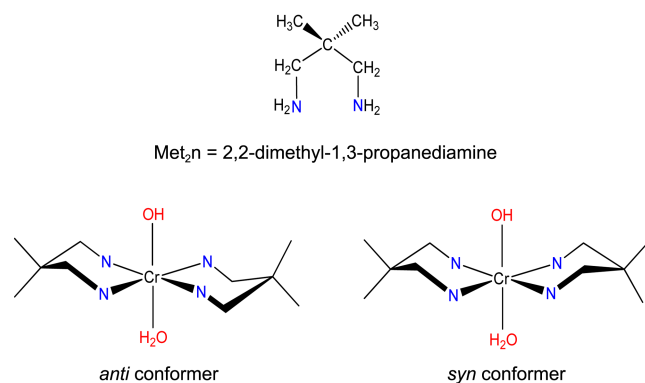
*trans-anti*-[Cr(Me<sub>2</sub>tn)<sub>2</sub>Cl<sub>2</sub>]<sub>2</sub>ZnCl<sub>4</sub> indicated that two chelate rings of Me<sub>2</sub>tn ligands were only in the *anti* chair-chair conformation.<sup>6-9</sup> The different conformations of the two six-membered chelate rings of Me<sub>2</sub>tn ligands may be dependent on the crystallographic disorder, packing forces, hydrated molecules and counter anions in the crystals. The factors which determine the stability of these geometric conformations are subtle and complicated.<sup>5-9</sup> X-ray crystallography is generally used to establish the presence of either conformation but the *syn* or *anti* conformer of the six-membered chelate rings cannot be readily discriminated by infrared and visible absorption spectroscopy. The elucidation of the factors that stabilize the either the *syn*- or *anti*-conformation in these complex cations continues to be of interest.

Here we report the synthesis, crystal structure and spectroscopic properties of the *trans-anti*-[Cr(Me<sub>2</sub>tn)<sub>2</sub>(OH)(H<sub>2</sub>O)]-(ClO<sub>4</sub>)<sub>2</sub> with the purpose of elucidating the influence of O-donor ligands occupying the axial positions and counter anions, on the geometry and the conformation referring to the position of the carbon atoms of the Me<sub>2</sub>tn chelate rings with respect to the equatorial coordination plane.

### Experimental

**Caution!** Although we experienced no difficulty with the perchlorate salt described in this study, the compound is potentially explosive, and should be handled with very great care.

**Materials and Synthesis.** The free ligand 2,2-dimethyl-1,3-propanediamine was obtained from Aldrich Chemical Co. and used as supplied. All chemicals were reagent grade materials and used without further purification. The starting material *trans*-[Cr(Me<sub>2</sub>tn)<sub>2</sub>Cl<sub>2</sub>]Cl was prepared as described previously.<sup>10</sup> A suspension of *trans*-[Cr(Me<sub>2</sub>tn)<sub>2</sub>Cl<sub>2</sub>]Cl (0.5 g, 1.38 mmol) in water (5 mL) containing pyridine (3 mL) was heated at 100 °C for 15 minutes. Then sodium perchlorate (1.0 g, 8.17 mmol) was added, and the solution was allowed to stand at room temperature for one day to give the pink crystals suitable for X-ray structural analysis. Yield 62%. *Anal.* Found: C, 24.40; H, 6.31; N, 11.35%. *Calc.* for Cr(C<sub>10</sub>H<sub>32</sub>N<sub>4</sub>O<sub>2</sub>)(ClO<sub>4</sub>)<sub>2</sub>: C, 24.45; H, 6.57; N, 11.40%. Visible spectral data for an aqueous solution, λ<sub>max</sub> in nm (ε in



**Figure 1.** Structure of ligand Me<sub>2</sub>tn and two possible conformational isomers of *trans*-[Cr(Me<sub>2</sub>tn)<sub>2</sub>(OH)(H<sub>2</sub>O)]<sup>2+</sup>.

$M^{-1} \text{ cm}^{-1}$ ): 403 (68.5), 502 (41.2). IR spectrum (KBr,  $\text{cm}^{-1}$ ): 3423 m and 3371 s ( $\nu$  OH), 3224 vs and 33170 s ( $\nu$  NH), 2966 vs and 2878 s ( $\nu$  CH), 1730 m, 1604 vs and 1563 vs ( $\delta$  NH<sub>2</sub>), 1479 vs ( $\delta$  CH<sub>2</sub>), 1374 m, 1223 m, 1152 vs, 1118 s ( $\nu$  CN), 1081 vs ( $\nu_{\text{as}}$  ClO), 1038 s, 988 s and 975s, 889 s and 841 m ( $\rho$  NH<sub>2</sub>), 810 m and 775 s ( $\rho$  CH<sub>2</sub>), 682 s, 636 s and 627 vs ( $\delta$  OClO), 541 m, 460 m and 438 m ( $\nu$  Cr-N).

**Physical Measurements.** The room-temperature visible absorption spectrum was recorded on a HP 8453 diode array spectrophotometer. The mid-infrared spectrum was obtained using a JASCO 460 plus series FT-IR spectrometer in a KBr pellet. Analyses for C, H and N were performed on a Carlo Erba 1108 Elemental Vario EL analyzer.

**Crystal Structure Analysis.** A plate pink crystal of title complex having approximate dimensions  $0.21 \times 0.21 \times 0.05 \text{ mm}^3$  was coated with paratone-*N* oil because the crystal loses its crystallinity on exposure to air. The diffraction data was measured with synchrotron radiation ( $\lambda = 0.62998 \text{ \AA}$ ) on a 2D SMC ADSC Quantum-210 detector with a silicon (111) double-crystal monochromator at the Pohang Accelerator Laboratory, Korea and a nitrogen cold stream (100 K). The ADSC Quantum-210 ADX program<sup>11</sup> was used for collection of data, and HKL3000sm (Ver. 703r)<sup>12</sup> was used for cell refinement, reduction, and absorption correction. The structure was solved by direct methods with SHELXTL-XS (Ver. 2013/4) program and refined by full-matrix least-squares calculations with the SHELXTL-XL (Ver. 2013/4) program.<sup>13</sup> Molecular graphics were produced using DIAMOND-3.<sup>14</sup> All non-hydrogen atoms were refined with anisotropic displacement coefficients. Hydrogen atoms were assigned isotropic displacement coefficients,  $U(\text{H}) = 1.2U(\text{C}, \text{N})$  or

**Table 1.** Crystallographic data for *trans*-[Cr(Me<sub>2</sub>tn)<sub>2</sub>(OH)(H<sub>2</sub>O)](ClO<sub>4</sub>)<sub>2</sub>

Chemical formula	C <sub>10</sub> H <sub>32</sub> CrN <sub>4</sub> O <sub>2</sub> ·2(ClO <sub>4</sub> )
$M_r$	491.29
Crystal system, space group	Monoclinic, $P2_1/n$
Temperature (K)	100 (2)
$a, b, c$ ( $\text{\AA}$ )	5.980 (2), 8.312 (2), 20.554 (4)
$\beta$ ( $^\circ$ )	92.50 (3)
$V$ ( $\text{\AA}^3$ )	1020.7 (4)
$Z$	2
Radiation type	Synchrotron, $\lambda = 0.62998 \text{ \AA}$
$\mu$ ( $\text{mm}^{-1}$ )	0.63
Crystal size (mm)	$0.21 \times 0.21 \times 0.05$
Diffraction	ADSC Q210 CCD area detector
	diffraction
$T_{\text{min}}, T_{\text{max}}$	0.880, 0.969
$R_{\text{int}}$	0.030
$R[F^2 > 2\sigma(F^2)], wR(F^2), S$	0.032, 0.084, 1.08
No. of reflections	2699
No. of parameters	133
No. of restraints	2
$\Delta\rho_{\text{max}}, \Delta\rho_{\text{min}}$ ( $\text{e \AA}^{-3}$ )	0.47, -0.90

$1.5U$  (C-methyl), and their coordinates were allowed to ride on their respective carbons except coordinated one of the water molecule hydrogen. The least-squares refinement of the structural model was performed under geometry restraints and displacement parameter restraints such as, DFIX, EXYZ and EADP for coordinated hydroxyl group and water molecule. The crystallographic experimental data and refinement parameters are summarized in Table 1.

## Results and Discussion

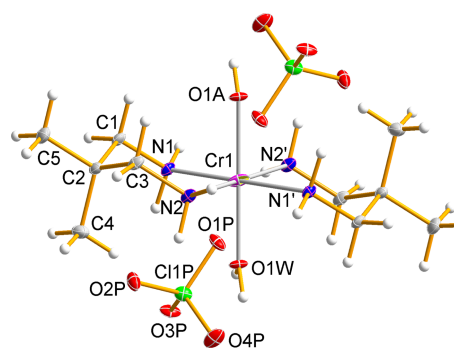
**Crystallography.** To determine the molecular structure unambiguously, single-crystal X-ray structure determination was carried out which revealed that the title complex salt consisted of a  $[\text{Cr}(\text{Me}_2\text{tn})_2(\text{OH})(\text{H}_2\text{O})]^{2+}$  cation and two perchlorate anions. The structure analysis showed the monoclinic space group of  $P2_1/n$  with  $Z=2$ . Principal bond lengths and angles are listed in Table 2. An ellipsoid plot of the complex molecule together with atomic numbering is illustrated in Figure 2. Hydrogen atoms are shown as circles of arbitrary radii.

In molecule the chromium(III) center is roughly coplanar with the four N atoms and adopts an octahedral geometry, where the four N atoms of two bidentate Me<sub>2</sub>tn ligands occupy the equatorial sites. The two O atoms of hydroxyl group and water molecule coordinate to the Cr metal center in the *trans* arrangement. The two chelate rings of Me<sub>2</sub>tn ligands in complex are only in stable *anti* chair-chair conformation. The conformational arrangement is very similar

**Table 2.** Selected bond distances ( $\text{\AA}$ ) and angles ( $^\circ$ ) for *trans*-[Cr(Me<sub>2</sub>tn)<sub>2</sub>(OH)(H<sub>2</sub>O)](ClO<sub>4</sub>)<sub>2</sub>

Cr1—N1	2.088 (9)	Cr1—N2	2.084 (2)
Cr1—O1A	1.971 (1)	Cr1—O1W <sup>i</sup>	1.971 (1)
N1—C1	1.492 (2)	N2—C3	1.493 (2)
C1—C2	1.528 (2)	C2—C3	1.527 (2)
C2—C4	1.532 (2)	C2—C5	1.535 (2)
O1A—Cr1—N2	89.12 (4)	O1A—Cr1—N1	90.73 (5)
O1A—Cr1—N2 <sup>i</sup>	90.88 (4)	O1W <sup>i</sup> —Cr1—N1	89.28 (5)
N2 <sup>i</sup> —Cr1—N1	89.66 (4)	N2—Cr1—N1	90.34 (4)
C1—N1—Cr1	119.05 (8)	C3—N2—Cr1	119.05 (8)

Symmetry code: (*i*)  $-x, -y+1, -z$ .

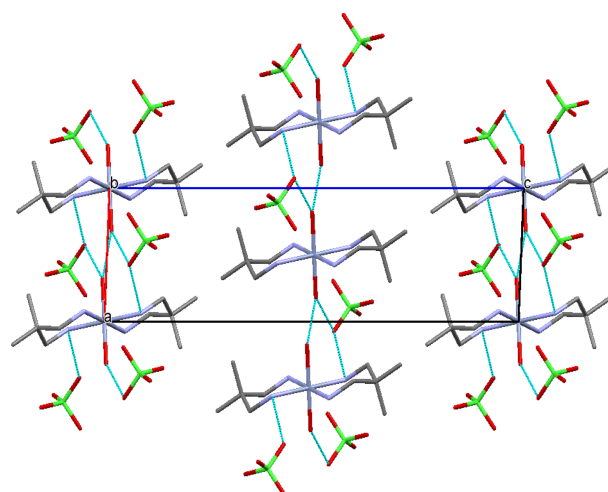


**Figure 2.** Molecular structure of *trans*-[Cr(Me<sub>2</sub>tn)<sub>2</sub>(OH)(H<sub>2</sub>O)](ClO<sub>4</sub>)<sub>2</sub>.

to *anti* conformation which was found in *trans-anti*-[Cr(Me<sub>2</sub>tn)<sub>2</sub>(NCS)<sub>2</sub>]<sub>2</sub>NCS·1/2H<sub>2</sub>O, *trans-anti*-[Cr(Me<sub>2</sub>tn)<sub>2</sub>Cl<sub>2</sub>]-ClO<sub>4</sub> and *trans-anti*-[Cr(Me<sub>2</sub>tn)<sub>2</sub>Cl<sub>2</sub>]<sub>2</sub>ZnCl<sub>4</sub>.<sup>6-9</sup> The *anti* conformational arrangement is quite different from *syn* conformer observed in *trans-anti/syn*-[Cr(Me<sub>2</sub>tn)<sub>2</sub>Cl<sub>2</sub>]Cl, and *trans-anti/syn*-[Cr(Me<sub>2</sub>tn)<sub>2</sub>Br<sub>2</sub>]<sub>2</sub>Br<sub>2</sub>·HClO<sub>4</sub>·H<sub>2</sub>O containing the two conformers.<sup>5,6</sup>

The difference observed in the conformations of the two chelate rings seems to be responsible for the differences in the crystallographic disorder, packing forces, and hydrogen-bonding networks among the solvent molecules, complex cations and anions in the complexes. The Cr–N bond distances for nitrogen atoms of the Me<sub>2</sub>tn are in the range of 2.084 (2) to 2.088 (2) Å and these lengths are in agreement with those observed in *trans-anti*-[Cr(Me<sub>2</sub>tn)<sub>2</sub>(NCS)<sub>2</sub>]-NCS·1/2H<sub>2</sub>O, *trans-anti*-[Cr(Me<sub>2</sub>tn)<sub>2</sub>Cl<sub>2</sub>]ClO<sub>4</sub>, *trans-anti*-[Cr(Me<sub>2</sub>tn)<sub>2</sub>Cl<sub>2</sub>]<sub>2</sub>ZnCl<sub>4</sub>, *trans-anti*-[Cr(tn)<sub>2</sub>Cl<sub>2</sub>]ClO<sub>4</sub>, *trans-anti*-[Cr(tn)<sub>2</sub>Br<sub>2</sub>]ClO<sub>4</sub>, *trans*-[Cr(en)<sub>2</sub>Br<sub>2</sub>]ClO<sub>4</sub>, *trans*-[Cr(15aneN<sub>4</sub>)F<sub>2</sub>]ClO<sub>4</sub>, *trans*-[Cr(2,2,3-tet)F<sub>2</sub>]ClO<sub>4</sub>, *cis*-β-[Cr(2,2,3-tet)Cl<sub>2</sub>]ClO<sub>4</sub> and *cis*-β-[Cr(2,2,3-tet)(N<sub>3</sub>)<sub>2</sub>]Br.<sup>6-9,15-21</sup> The oxygen atoms in *trans* position coordinated chromium(III) both carry one well defined H atom, the displacement parameter of which refined to an acceptably value. Each oxygen atom is also associated with a weaker electron density peak at the correct position to define a second hydrogen atom. The displacement parameters associated with these minor peaks refine to acceptable values if fixed site occupancy of 0.5 is assumed. This situation can be modelled in terms of disorder across the two oxygen sites of the water and hydroxyl ligands. One hydroxyl group plus two perchlorate anions thus balance the +3 charge on chromium, as required. The postulation of disorder at the axial sites accounts for the nearly equal Cr–O bond lengths. Because of X-ray diffraction is such a weak analytical technique with regard to hydrogen placement, the ultimate interpretation of the structure remains unclear. A similar interpretation of a hydroxo/aquo ambiguity is found in *trans*-[Cr(5,12-*meso*-5,7,7,12,14,14-hexamethyl-1,4,8,11-tetraazacyclotetradecane)(OH)(H<sub>2</sub>O)](ClO<sub>4</sub>)<sub>2</sub>·H<sub>2</sub>O.<sup>22</sup> The mean Cr1–O1A bond is 1.971 (2) Å in length and the O1A–Cr1–O1A<sup>i</sup> bond angle is constrained to 180°. The Cr–O bond length is slightly shorter than the distance for the Cr–OH<sub>2</sub> and longer than that for Cr–OH as expected. The bond length is also comparable to the values of Cr–O bond lengths found in *trans*-[Cr(cyclam)(OH)(H<sub>2</sub>O)]Br<sub>2</sub>·H<sub>2</sub>O, *trans*-[Cr(cyclam)(OH)(H<sub>2</sub>O)](SF<sub>3</sub>SO<sub>3</sub>)<sub>2</sub>, *trans*-[Cr(cyclam)(OH)<sub>2</sub>]ClO<sub>4</sub>, *trans*-[Cr(cyclam)(*nic-O*)<sub>2</sub>]ClO<sub>4</sub>, *cis*-[Cr(cyclam)(ONO)<sub>2</sub>]NO<sub>2</sub>, [Cr(cyclam)ox]ClO<sub>4</sub>, [Cr(acac)(edda)] and [Cr(cyclam)(acac)](ClO<sub>4</sub>)<sub>2</sub>·1/2H<sub>2</sub>O.<sup>23-29</sup>

The six-membered ring is in stable chair conformation with the N1–Cr1–N2 angle of 90.34 (4)°. The average Cr–N–C, N–C–C and C–C–C bond angles in the six-membered chair rings are 119.05 (8), 114.08 (2) and 109.43 (2)°, respectively. The presence of two substituted methyl groups on the carbon of the Me<sub>2</sub>tn molecule does not disturb the essential features of the chelate ring. The mean C–N and C–C distances in Me<sub>2</sub>tn ligand are typical, and comparable to those observed in *trans*-[Cr(Me<sub>2</sub>tn)X<sub>2</sub>]<sup>+</sup> (X = Cl, Br, NCS,



**Figure 3.** Hydrogen-bonded thick sheet structure, viewed along the *b* axis. Hydrogen bonding is denoted by blue dashed lines (color figure online).

N<sub>3</sub>) complexes.<sup>5-9</sup> The uncoordinated ClO<sub>4</sub><sup>−</sup> anions remain outside the coordination sphere. The perchlorate anion shows a distorted tetrahedral arrangement of oxygen atoms around the central chloride atom with Cl–O distances in the range 1.427 (2)–1.447 (2) Å and the O–Cl–O angles ranging from 107.61 (9) to 110.96 (8)°.

In the title complex, the crystal lattice is stabilized by hydrogen bonding interactions between the NH groups of the Me<sub>2</sub>tn ligand, hydrogens of OH or H<sub>2</sub>O ligands, and the oxygens of the ClO<sub>4</sub><sup>−</sup> anion. Thus, the [Cr(Me<sub>2</sub>tn)<sub>2</sub>(OH)(H<sub>2</sub>O)]<sup>2+</sup> cations and ClO<sub>4</sub><sup>−</sup> anions are linked together by a network of hydrogen bonds (Fig. 3). The differences found in the conformations of the two six-membered chelate rings may be attributed to the differences in the hydrogen bonding networks, compositional disorder, and crystal packing forces between the chromium(III) complex cation and counter anion present in the lattice.

## Conclusions

The X-ray crystallographic analysis of newly prepared complex indicates that the chromium atom is in a distorted octahedral environment, coordinated by two bidentate, 2,2-dimethyl-1,3-propanediamine ligands and two O atoms of hydroxy group and water molecule in *trans* position. The average Cr–N(Me<sub>2</sub>tn) and Cr–O bonds are 2.086 (2) and 1.907 (9) Å, respectively. The carbon atoms of the two Me<sub>2</sub>tn chelate rings in the complex adopt a stable *anti* conformation on the coordination plane not including *syn* conformer. The crystal-packing and hydrogen-bonding forces as well as the counter anion dimensions are probably responsible for the observed different conformation.

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**Supplementary Material.** Crystallographic data, tables of atomic coordinates and thermal parameters, and full lists of bond lengths and angles have been deposited with the Cambridge Crystallographic Data Centre, CCDC No. 977110. Copies of this information may be obtained free of charge from The Director, CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (fax: +44-1223-336-033; e-mail: deposit@ccdc.cam.ac.uk or www: <http://www.ccdc.cam.ac.uk>).

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