larger than the N-C bond in free \(\text{NMe}_3\), 1.434(2) \(\text{Å}\).

The \(\angle \text{Al}-\text{N}-\text{C}\) angle in \(\text{Cl}_2\text{AlNMe}_3\) is larger than the corresponding angles in \(\text{H}_2\text{AlNMe}_3\) and \(\text{Me}_2\text{AlNMe}_3\), 109.0(3)° and 109.3(4)°, respectively, although the difference is of marginal statistical significance. Such a difference might easily be explained as the result of increased steric interaction between donor and acceptor due to the shortening of the Al-N bond.

The Al-Cl bond distance is in excellent agreement with the mean Al-Cl bond distance found in the crystal, 2.123 \(\text{Å}\). Just as the N-C bond distances in the complexes \(\text{X}_2\text{AlNMe}_3\) are longer than in free \(\text{NMe}_3\) and appear to increase in length with increasing acceptor strength of \(\text{X}_2\text{Al}\), so the Al-Cl bond distances in complexes of the type \(\text{Cl}_2\text{Al}-\text{L}\) are longer than the Al-Cl bond in free \(\text{AlCl}_3\) and appear to increase with increasing donor strength of the base \(\text{L}\). The Al-Cl bond in \(\text{Cl}_2\text{AlNMe}_3\) is significantly longer than the mean Al-Cl bond distance in the complex of \(\text{AlCl}_3\) with propionyl chloride, 2.093(3) \(\text{Å}\), which in turn is significantly longer than the Al-Cl bond in free \(\text{AlCl}_3\), 2.06 ± 0.01 \(\text{Å}\).

The shrinkage of the Cl---C(trans) distance was found to be 0.12(6) \(\text{Å}\).

Acknowledgements. We are grateful to professor I. R. Beattie for a sample of \(\text{Cl}_2\text{AlNMe}_3\) and to the Norwegian Research Council for Science and Technology for financial support.


Preparations of \(\text{trans-Bis(2-picolylamine)}\) Complexes of Chromium(III)

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In a recent paper, two series of octahedral \(\text{cis-bis(2-picolylamine)}\) complexes of chromium were described (2-picolylamine = 2-aminomethylpyridine).

The ligands in the \(\text{cis-}\)positions were \(\text{Cl}^-, \text{Br}^-, \) and \(\text{H}_2\text{O}\). This paper reports the preparation of a series of the corresponding \(\text{trans}\)-complexes.

Only one type of geometrical isomer occurs, probably a \(\text{trans,trans,trans}\) isomer (Fig. 1: a). It is likely that the other imaginable isomer, the \(\text{trans,cis,cis}\) (Fig. 1: b), does not exist because of sterical hindrance.

![Diagram](image)

*Fig. 1. The geometrical isomers of the \(\text{trans-[Cr(C_6H_5N_2)xCl]}^{2+}\) ion. P symbolizes the pyridine nitrogens, M, the methyamine nitrogens. a. The \(\text{trans,trans,trans}\) isomer. b. The \(\text{trans,cis,cis}\) isomer.*

The preparations are based upon the reaction between the so-called \(\text{α-cis-[Cr(C_6H_5N_2)_3Cl]}^{2+}\) and moist silver oxide. The resulting solution apparently contains both \(\text{trans-}\) and \(\text{α-cis-[Cr(C_6H_5N_2)_3OH-H}_2\text{O]}^{2+}\). By means of conc. nitric acid \(\text{trans-[Cr(C_6H_5N_2)_3(H}_2\text{O)] (NO}_3\) is isolated from the reaction mixture and used as an initial material for \(\text{trans-[Cr(C_6H_5N_2)_3Cl]}^{2+}\) and \(\text{trans-[Cr(C_6H_5N_2)_3Br]}^{2+}\).

The assignment of geometric configuration is based entirely upon the colours of
Table 1. Spectral data of trans-[Cr en2X2]n+ and trans-[Cr(C8H8N2)2Cl2X2]n+.

<table>
<thead>
<tr>
<th>X–X</th>
<th>Amine</th>
<th>( \lambda_{\text{max}}(1) )</th>
<th>( \varepsilon(1) )</th>
<th>( \lambda_{\text{max}}(2) )</th>
<th>( \varepsilon(2) )</th>
<th>( \lambda_{\text{max}}(3) )</th>
<th>( \varepsilon(3) )</th>
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<tr>
<td>H2O</td>
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<td>29.3</td>
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<td>2</td>
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<tr>
<td>Cl</td>
<td>C8H8N2</td>
<td>~510</td>
<td>~18</td>
<td>443.5</td>
<td>28.1</td>
<td>~360</td>
<td>~34</td>
<td>3</td>
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<tr>
<td>Cl</td>
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<td>24.5</td>
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<td>396</td>
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<tr>
<td>Br</td>
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<tr>
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<td>43.3</td>
<td>~465</td>
<td>~23</td>
<td>405</td>
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</table>

The compounds and a comparison of their visible absorption spectra with those of known bis (ethylendiamine) analogous (Table 1).\(^3\,\!^4\,\!^5\,\!^6\)

Experimental. \( \alpha \)-cis-[Cr(C8H8N2)2Cl4]Cl.H2O was prepared as described before.\(^1\) All other reagents were reagent grade and used without further purification.

Trans-Diaqua Bis (2-picolyamine) Chromium (III) nitrate, [Cr(C8H8N2)2(H2O)2](NO3)2, 1.40 g \( \alpha \)-cis-[Cr(C8H8N2)2Cl4]Cl.H2O (3.57 mmol) was added to moist silver nitrate (18 mmol). After 5 min the violet solution was filtered and acidified with conc. nitric acid. During stirring and ice-cooling 250 ml ethanol (99 %) was added slowly to precipitate yellow-orange crystals of presumed trans-[Cr(C8H8N2)2(H2O)2](NO3)2. They were filtered and washed with ethanol and acetone, 0.78 g. The recrystallization was performed by dissolving the crystals in 5–6 ml boiling water, filtering and cooling on ice after the addition of 1 ml conc. nitric acid. Yield: 0.65 g (36 %) of filtered needles. (Found: Cr 10.24; C 28.3; N 19.4; H 4.40. Calc. for [Cr(C8H8N2)2(H2O)2](NO3)2.H2O: Cr 10.23; C 28.4; N 19.3; H 4.36).

Trans-Dichlorobis (2-picolyamine) Chromium (III) chloride, [Cr(C8H8N2)2Cl4]Cl. 1.40 g trans-[Cr(C8H8N2)2(H2O)2](NO3)2.H2O (2.75 mmol) was heated on a water-bath (100°) with 6–7 ml conc. hydrochloric acid. A violet solution soon formed and grey-green, shining crystals separated. After cooling on ice, they were filtered and washed with conc. hydrochloric acid and acetone. 0.65 g. The compound was recrystallized from 30 ml boiling 6 M hydrochloric acid. Washing as above. Yield: 0.54 g (47 %). (Found: Cr 12.69; C 34.6; N 13.5; Cl 25.5. Calc. for [Cr(C8H8N2)2Cl4]Cl. 2.25H2O: Cr 12.52; C 34.7; N 13.5; Cl 25.6).

The hydrogen analyses for this and the following compound failed to come out reproducibly. Trans-Dibromobis (2-picolyamine) chromium (III) bromide, [Cr(C8H8N2)2Br4]Br. 1.00 g trans-[Cr(C8H8N2)2(H2O)2](NO3)2.H2O (1.97 mmol) was heated on a water-bath (100°) with 10 ml hydrobromic acid (48 %). A grass-green precipitate, probably trans-[Cr(C8H8N2)2Br4] Br.Br2 soon formed. Approx. 300 mg ascorbic acid was added and the heating continued, until the tribromide had been transformed to the more soluble, darkgreen bromide. After cooling on ice the compound was filtered and washed with ice-water and acetone. Yield 0.54 g (52 %). Recrystallization was not necessary. (Found: Cr 9.82; C 27.5; N 10.6; Br 45.7. Calc. for [Cr(C8H8N2)2Br4]Br.H2O: Cr 9.88; C 27.4; N 10.7; Br 45.6).

Electronic absorption spectra were recorded on a Cary Model 14 spectrophotometer. Data for the maxima are given in Table 1 as \( \lambda_{\text{max}} \) and \( \varepsilon \). The wavelength \( \lambda \) in \( \text{m} \) is the molar extinction coefficient \( \varepsilon \) in \( \text{mol}^{-1} \text{cm}^{-1} \). Medium 0.1 M hydrochloric acid.


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