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#### Key indicators

Single-crystal X-ray study

$T = 273\text{ K}$

Mean  $\sigma(\text{C}-\text{C}) = 0.007\text{ \AA}$

$R$  factor = 0.054

$wR$  factor = 0.125

Data-to-parameter ratio = 16.1

For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

## Tetraethylammonium dichloro[*N,N'*-(4,5-dichloro-1,2-phenylene)bis(pyridine-2-carboxamido)]-chromate(III)

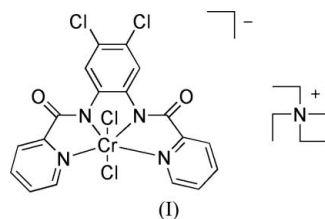
In the title complex,  $(\text{C}_8\text{H}_{20}\text{N})[\text{Cr}(\text{C}_{18}\text{H}_{10}\text{Cl}_2\text{N}_4\text{O}_2)\text{Cl}_2]$ , the coordination geometry of the  $\text{Cr}^{\text{III}}$  atom is distorted octahedral, with the  $\text{Cr}-\text{N}_{\text{amide}}$  distances [1.971 (4) and 1.973 (3) Å] significantly shorter than the  $\text{Cr}-\text{N}_{\text{pyridyl}}$  distances [2.077 (4)–2.093 (3) Å] and a  $\text{Cl}-\text{Cr}-\text{Cl}$  angle of  $169.24 (5)^\circ$ .

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#### Comment

A number of transition metal complexes are known to activate molecular oxygen and transfer it to a substrate (Sono *et al.*, 1996). Such oxygen-carrier metal complexes are of interest as catalysts for the oxidation of organic compounds, and as models for oxygenase enzymes (Que & Ho, 1996). The compound 1,2-bis(2-pyridinecarboxamido)benzene ( $\text{H}_2\text{bpb}$ ) (Lin *et al.*, 2001) and its various derivatives have been extensively employed as tetradentate diamide ligands for various transition metal ions. The crystal structure of the related compound triethylammonium [1,2-bis(2-pyridinecarboxamido)-4,5-dimethylbenzene]dichloroferrate(III) (Lee *et al.*, 2002) has previously been determined. In order to study the catalytic oxidation reactions of hydrocarbons, we have synthesized the title complex, (I), by the reaction of chromium(III) chloride with  $\text{H}_2\text{bpCl}_2$ , and its crystal structure is reported here.



The molecular structure of (I) is shown in Fig. 1 and selected bond lengths and angles are given in Table 1. The four N atoms of the  $\text{bpCl}_2^{2-}$  ligand form the equatorial plane of the distorted octahedral coordination geometry of the  $\text{Cr}^{\text{III}}$  atom. The  $\text{Cr}-\text{N}_{\text{amide}}$  distances are significantly shorter than the  $\text{Cr}-\text{N}_{\text{pyridyl}}$  distances and the  $\text{Cl}-\text{Co}-\text{Cl}$  angle deviates significantly from linearity. In the anion, the pyridine rings are essentially in the plane of the four coordinating N atoms (N1/N2/N3/N4), as demonstrated by the torsion angles of  $-0.9 (4)$  and  $1.4 (4)^\circ$  for  $\text{C1}-\text{N1}-\text{Cr1}-\text{N4}$  and  $\text{C18}-\text{N4}-\text{Cr1}-\text{N1}$ , respectively.

#### Experimental

The ligand  $\text{H}_2\text{bpCl}_2$  was prepared according to a literature procedure (Lee *et al.*, 2002). Equimolar (0.5 mmol) quantities of chromium(III)

chloride hexahydrate and  $\text{H}_2\text{bpCl}_2$  were dissolved in dimethylformamide (10 ml), and triethylamine (1 mmol) was added to the reaction mixture. Tetraethylammonium chloride hydrate (140 mg, 1 mmol) was then added to the mixture and refluxed for 6 h. After cooling, the volume was then reduced to 2 ml. Upon addition of diethyl ether, a brown precipitate was produced and collected by filtration. Red-brown crystals suitable for X-ray diffraction were obtained by slow evaporation of an acetonitrile solution of (I) at room temperature.

## Crystal data

$(\text{C}_8\text{H}_{20}\text{N})[\text{Cr}(\text{C}_9\text{H}_5\text{Cl}_2\text{N}_2\text{O})_2\text{Cl}_2]$   
 $M_r = 638.35$   
 Monoclinic,  $P2_1/c$   
 $a = 8.061$  (5) Å  
 $b = 22.874$  (14) Å  
 $c = 15.653$  (9) Å  
 $\beta = 97.350$  (12)°  
 $V = 2863$  (3) Å<sup>3</sup>

$Z = 4$   
 $D_x = 1.481$  Mg m<sup>-3</sup>  
 Mo  $K\alpha$  radiation  
 $\mu = 0.81$  mm<sup>-1</sup>  
 $T = 273$  (2) K  
 Plate, red-brown  
 $0.10 \times 0.08 \times 0.02$  mm

## Data collection

Bruker SMART CCD  
 diffractometer  
 $\varphi$  and  $\omega$  scans  
 Absorption correction: none  
 15736 measured reflections

5586 independent reflections  
 2638 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.130$   
 $\theta_{\text{max}} = 26.0^\circ$

## Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.054$   
 $wR(F^2) = 0.125$   
 $S = 0.82$   
 5586 reflections  
 347 parameters

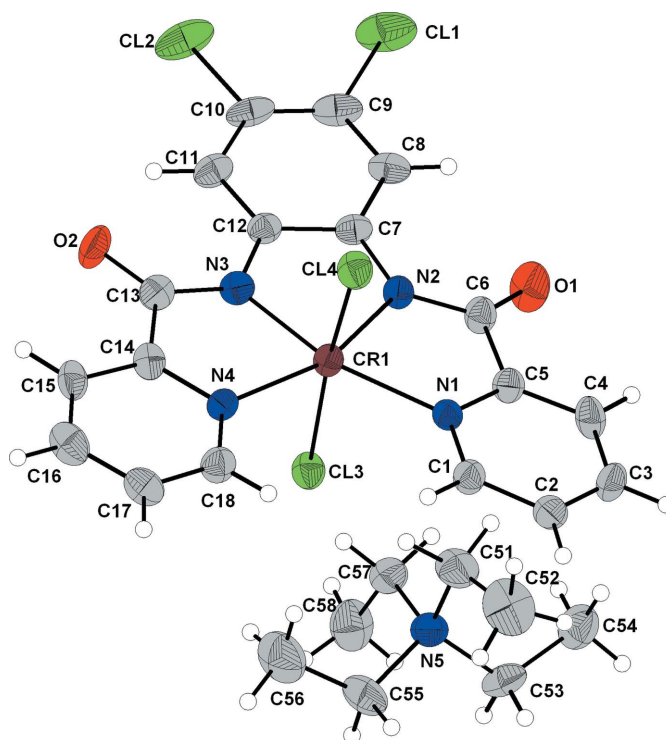
H-atom parameters constrained  
 $w = 1/[\sigma^2(F_o^2) + (0.0456P)^2]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\text{max}} = 0.001$   
 $\Delta\rho_{\text{max}} = 0.59$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.42$  e Å<sup>-3</sup>

**Table 1**

Selected geometric parameters (Å, °).

Cr1–N3	1.971 (4)	Cr1–N4	2.093 (3)
Cr1–N2	1.973 (3)	Cr1–Cl3	2.3318 (18)
Cr1–N1	2.077 (4)	Cr1–Cl4	2.3372 (17)
N3–Cr1–N2	81.50 (15)	N1–Cr1–Cl3	85.37 (11)
N3–Cr1–N1	161.01 (15)	N4–Cr1–Cl3	89.35 (11)
N2–Cr1–N1	79.57 (15)	N3–Cr1–Cl4	94.72 (12)
N3–Cr1–N4	79.39 (15)	N2–Cr1–Cl4	93.85 (12)
N2–Cr1–N4	160.85 (15)	N1–Cr1–Cl4	88.06 (11)
N1–Cr1–N4	119.57 (15)	N4–Cr1–Cl4	86.49 (11)
N3–Cr1–Cl3	94.25 (12)	Cl3–Cr1–Cl4	169.24 (5)
N2–Cr1–Cl3	93.33 (12)		

H atoms were placed in calculated positions, with C–H = 0.93 (pyridine and benzene), 0.97 (methylene) and 0.96 Å (methyl). They were included in the refinement in the riding-model approximation, with  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$  or  $1.5U_{\text{eq}}(\text{methyl C})$ . Only 47% of the data collected to  $26.0^\circ$  in  $\theta$  were considered observed. The high number of weak data used in the refinement has given a larger than normal  $R_{\text{int}}$  value (0.13) and may affect the precision of the structure.



**Figure 1**

The asymmetric unit of (I), showing ellipsoids drawn at the 50% probability level.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINTE* (Bruker, 1997); data reduction: *SAINTE*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1998); software used to prepare material for publication: *SHELXTL*.

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## References

- Bruker (1997). *SMART* and *SAINTE*. Bruker AXS Inc., Madison, Wisconsin, USA.
- Bruker (1998). *SHELXTL*. Version 5.10. Bruker AXS Inc., Madison, Wisconsin, USA.
- Lee, S. J., Lee, J. Y., Kim, C., Nam, W. & Kim, Y. (2002). *Acta Cryst.* **E58**, m191–m193.
- Lin, J., Zhang, J.-Y., Xu, Y., Ke, X.-K. & Guo, Z.-J. (2001). *Acta Cryst.* **C57**, 192–194.
- Que, L. Jr & Ho, R. Y. N. (1996). *Chem. Rev.* **96**, 2607–2624.
- Sheldrick, G. M. (1997). *SHELXL97* and *SHELXS97*. University of Göttingen, Germany.
- Sono, M., Roach, M. P., Coulter, E. D. & Dawson, J. H. (1996). *Chem. Rev.* **96**, 2841–2888.