

of cuprous oxide and TFAA until water is added. The addition of 4 equiv of water brings about immediate reaction, presumably through the formation of TFA and its dissolution of the Cu(I) ion. Uptake of 1 equiv of carbon monoxide per Cu(I) is rapid and stops sharply. Separation of the crystals of the carbonyl complex from the cooled solution by filtration in a glove bag is necessary, as the indicative blue-green color of Cu(II) is almost impossible to avoid.

Analysis of the crystals, their infrared spectrum, and

the pumping experiment all support the postulated stoichiometry of  $\text{Cu}^+:\text{CO}:\text{TFA}^-:\text{TFA}$  as 1:1:1:1. That the carbon monoxide is still present in the solvent-free crystals prepared by brief pumping is shown by its appearance at  $2155\text{ cm}^{-1}$ ; the frequency is unshifted from that characteristic of the initially prepared complex.

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## Correspondence

### On the Existence of the $\beta$ -*cis* Configuration of Chelated Ethylenediamine-N,N'-diacetate

Sir:

The use of ethylenediaminediacetate (edda) as a coordinating ligand has been the subject of several recent papers.<sup>1-3</sup> These papers concern the preparation, resolution, and characterization of complexes in which edda acts as a tetradentate ligand and the other two positions in the coordination sphere are occupied by various bidentate groups.

Legg and Cooke<sup>1</sup> distinguished the coordination of edda in the so-called *trans* configuration from the so-called *cis* configuration by means of nuclear magnetic resonance spectroscopy. We feel that complexes with edda in these configurations should be called  $\alpha$ -*cis* and  $\beta$ -*cis*, respectively, in accord with the nomenclature used for triethylenetetramine complexes.<sup>4</sup> This enables complexes where edda assumes a planar disposition about the central metal atom (allowing two unidentate ligands to occupy positions *trans* to one another) to be described as *trans*. Such a compound has been prepared.<sup>5</sup>

Small amounts of the  $\beta$ -*cis* isomer of some edda complexes have been isolated by ion-exchange chromatography.<sup>1,2</sup> However, in the systems so far reported<sup>1-3</sup> the  $\alpha$ -*cis* isomer appears to have been formed almost exclusively. This together with an analysis of nmr data and a consideration of molecular models led Legg and Cooke<sup>1</sup> to conclude that edda favored an  $\alpha$ -*cis* configuration due to increased "steric interaction between the ligands" in the  $\beta$ -*cis* isomers. Specifically there appears to be a greater interaction between the amine protons of ethylenediamine and the protons of edda in the  $\beta$ -*cis* isomer. Our present work has shown that the  $\alpha$ -*cis* configuration of edda is not preferred in all complexes and in particular in the case of the  $\text{Co}(\text{edda})\text{CO}_3^-$  isomers and their oxalate (ox) and malonate (mal) analogs.

It is noteworthy that in the  $\beta$ -*cis* isomers of these complexes the steric interactions postulated for the corresponding diamine analogs are absent. Van Saun and Douglas only found the  $\alpha$ -*cis* isomers for  $\text{Co}(\text{edda})\text{CO}_3^-$  and related complexes.<sup>3</sup> However, they isolated less than 30% of the total possible yield, and it is conceivable that the  $\beta$ -*cis* isomers still remained in solution.

We have prepared a series of edda complexes and are studying the kinetics of their aquation and isomerization. The  $\alpha$ -*cis* and  $\beta$ -*cis* isomers of  $\text{Co}(\text{edda})\text{CO}_3^-$ ,  $\text{Co}(\text{edda})(\text{ox})^-$ ,  $\text{Co}(\text{edda})(\text{mal})^-$ ,  $\text{Co}(\text{edda})(\text{OH}_2)_2^+$ , and  $\text{Co}(\text{edda})\text{Cl}_2^-$  have been prepared. A previous nmr study showed that in the  $\alpha$ -*cis* isomers the acetate ring protons exhibited only one AB quartet but in the  $\beta$ -*cis* isomers the spectrum was much more complex.<sup>1</sup> Our complexes confirm this conclusion. Further support for our assignment of configuration has come from the retention of configuration when the carbonate isomers are aquated with dilute perchloric acid and regenerated with bicarbonate. These reactions occur with retention as had previously been found in the reactions of related complexes.<sup>6</sup>

The  $\alpha$ -*cis* and  $\beta$ -*cis* isomers of  $\text{Co}(\text{edda})(\text{OH}_2)_2^+$  were separable by cation-exchange chromatography and in aqueous solution the  $\beta$ -*cis* form isomerizes to the more stable  $\alpha$ -*cis*. However, in contrast to the findings of Van Saun and Douglas<sup>3</sup> and unlike any of the systems so far investigated<sup>1-3</sup>, the  $\beta$ -*cis* isomer of  $\text{Co}(\text{edda})\text{CO}_3^-$  is more stable than the  $\alpha$ -*cis* isomer in aqueous solution. The  $\beta$ -*cis* isomer is thermodynamically favored over the  $\alpha$ -*cis* by about 600 cal/mol.

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SCHOOL OF CHEMISTRY  
THE UNIVERSITY OF WESTERN AUSTRALIA  
NEDLANDS, WESTERN AUSTRALIA 6009

P. J. GARNETT  
D. W. WATTS

DEPARTMENT OF CHEMISTRY  
WASHINGTON STATE UNIVERSITY  
PULLMAN, WASHINGTON 99163

J. IVAN LEGG<sup>7</sup>

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