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# Investigation of the Reactions of Hexanitrocobaltate(III) with Amino Acids. XIII. The Structure and Absolute Configuration of $(-)_{589}$-*ab*-(Histidinato-*N*,*N′*)-*cdf*-(histidinato-*O*,*N*,*N′*)-*e*-nitrocobalt(III) Monohydrate

BY RAJNA HERAK AND B. PRELESNIK

*Laboratory of Solid State Physics and Radiation Chemistry, 'Boris Kidrič' Institute,* 11001 *Belgrade, PO Box* 522, *Yugoslavia*

B. KAMBERI

*Faculty of Sciences, University of Priština,* 83000 *Priština, Yugoslavia*

AND M. B. ĆELAP

*Faculty of Sciences, University of Belgrade,* 11001 *Belgrade, PO Box* 550, *Yugoslavia*

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## Abstract

$C_{12}H_{16}CoN_7O_6.H_2O$, $[CoNO_2(\text{L-his})_2].H_2O$ (L-hisH = L-histidine), $M_r = 431{\cdot}25$, crystallizes in the monoclinic space group $C2$ with $a = 25{\cdot}49$ (2), $b = 8{\cdot}318$ (3), $c = 8{\cdot}408$ (3) Å, $\beta = 100{\cdot}5$ (1)°, $U = 1753$ Å$^3$, $Z = 4$, $D_m = 1{\cdot}63$, $D_c = 1{\cdot}635$ Mg m$^{-3}$, $\mu$(Mo $K\alpha$) = 1·08 mm$^{-1}$. The structure was solved from diffractometric data by the heavy-atom method and refined by full-matrix least-squares techniques to a final $R = 0{\cdot}065$ for 1228 observed reflexions. The crystal structure consists of complex molecules and water molecules joined by hydrogen bonds of the types O—H···O and N—H···O. One of the histidinato ligands is tridentate and coordinated to Co by carboxylato O and amino and imidazole N atoms. The second histidinato ligand is bidentate with imidazole and amino N atoms coordinating to Co. The absolute configuration of the complex molecule is $\Lambda$.

## Introduction

Two of us (BK and MBĆ) have synthesized, by the action of L-histidine on sodium hexanitrocobaltate(III) in a molar ratio 2:1, two geometrical isomers of the bis(L-histidinato)nitrocobalt(III) complex, $[CoNO_2(\text{L-his})_2]$ (Kamberi, Ćelap & Janjić, 1980). One is orange and the other a reddish colour.

The importance of interactions between histidine and transition-metal ions has been recognized for some time and the crystal structures of histidine and its metal complexes have been extensively studied. These studies have shown that histidines can use each of the three potential coordination sites, imidazole nitrogen $N_I$, amino nitrogen $N_\alpha$ and carboxylato oxygen $O^-$, for bonding to metal ions. In the isomers obtained, one histidine residue acts as a bidentate and the other as a tridentate ligand.

Theoretically, 18 geometrical isomers can appear, *i.e.* three groups of six isomers; in each of the six in one group histidine as a bidentate ligand is coordinated either through $N_I$ and $N_\alpha$, $N_I$ and $O^-$ or $N_\alpha$ and $O^-$ respectively. The second notable feature of the structure of histidine compounds is that the histidine moiety occurs in two conformations, 'open' and 'closed'; the magnitude of the torsion angle about the $C^\alpha$—$C^\beta$ bond differentiates between the two conformers. The total number of geometrical isomer–conformers is therefore much larger.

The X-ray crystal-structure analysis reported here was undertaken to determine the structural isomerism of the orange isomer of the $[CoNO_2(\text{L-his})_2]$ complex (Kamberi *et al.*, 1980).

## Experimental

The orange crystals of $(-)$-$[CoNO_2(\text{L-his})_2].H_2O$ have a thick-tabular shape. The X-ray data were obtained from a crystal of dimensions 0·08 × 0·10 × 0·15 mm. The unit-cell dimensions were initially determined from rotation and Weissenberg photographs and later adjusted by least-squares refinement of a series of diffractometer-measured $\theta$ angles. The extinction of $hkl$ reflexions when $h + k = 2n + 1$ is consistent with space groups $C2/m$, $Cm$ and $C2$. In accordance with the established optical activity of the compound, the space

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group $C2$ was chosen and confirmed by successful refinement. The intensity data were collected on a Syntex $P\bar{1}$ four-circle diffractometer, using graphite-monochromatized Mo $K\alpha$ radiation and an $\omega$-scan procedure. All independent reflexions in the sphere $\theta \leq 25°$ were measured; 1228 observations satisfying the criterion $I > 2\sigma(I)$ were used in the subsequent analysis. The intensity of a standard reflexion measured every 30 reflexions remained essentially constant throughout data collection. The values of $I$ and $\sigma(I)$ were corrected for Lorentz and polarization effects, but not for absorption.

The structure was solved by routine application of the heavy-atom method, and the atomic parameters refined by full-matrix least-squares methods. The function minimized was $\sum (|F_o| - |F_c|)^2/\sigma(F_o)^2$. All the atoms were assumed to be uncharged. Values for the atomic scattering factors and anomalous terms for Co were taken from *International Tables for X-ray Crystallography* (1974).

Refinement of positional and thermal parameters (anisotropic for Co and O, isotropic for C and N atoms) gave $R = 0{\cdot}071$. At this stage, the coordinates $(x,y,z)$ of all atoms were replaced by $(\bar{x},\bar{y},\bar{z})$, to obtain the expected arrangement which corresponds to L-histidine. Two cycles of least-squares refinement reduced $R$ to 0·070. The positions of most of the H atoms belonging to the complex molecule were located on the difference Fourier map. The H atoms (except those from the water molecule) were included in the structure-factor calculations at calculated, fixed positions, with temperature factors fixed at $B = 3{\cdot}5$ Å$^2$. Refinement terminated at $R = 0{\cdot}065$, $R_w = 0{\cdot}070$. The maximum shift/error in the final cycle was 0·2, the final $[\sum w(F_o - F_c)^2/(m - n)]^{1/2} = 1{\cdot}25$ ($m$ = number of observations, $n$ = number of parameters).

The final atomic parameters derived from the last cycle of least-squares refinement are given in Table 1, along with standard deviations estimated from the inverse matrix.*

The main computer programs used on the CDC-3600 computer were Zalkin's (1965) *FORDAP* Fourier program, J. A. Ibers and R. J. Doedens's *NUCLS* least-squares program, and *GEOM* (molecular geometry and standard deviations, written by K. W. Muir and P. Mallinson).

## Results and discussion

The structure consists of discrete molecules of $[CoNO_2(L\text{-his})_2]$ and molecules of water, linked together by a three-dimensional arrangement of the O—H···O and N—H···O hydrogen bonds. The projection of the structure down the $b$ axis with the numbering scheme of the atoms is given in Fig. 1.

Table 1. *Final atomic coordinates* (×10$^4$) *and isotropic thermal parameters* (Å$^2$) *with e.s.d.'s in parentheses*

| | $x$ | $y$ | $z$ | $B_{eq}/B_{iso}$ |
|---|---|---|---|---|
| Co | 1365·1 (6) | 7500 | 2148·7 (2) | 1·92 (5)* |
| O($W$) | 1858 (4) | 2777 (16) | 2060 (12) | 5·4 (4)* |
| O(1) | 2093 (3) | 8008 (10) | 2137 (9) | 2·3 (2)* |
| O(2) | 2749 (3) | 7588 (18) | 770 (9) | 4·2 (3)* |
| O(1′) | −410 (4) | 6085 (13) | 1381 (12) | 4·0 (3)* |
| O(2′) | −235 (5) | 3535 (16) | 2195 (17) | 7·7 (5)* |
| O(3) | 884 (5) | 9492 (18) | 4125 (14) | 5·9 (4)* |
| O(4) | 1697 (4) | 9937 (15) | 4288 (12) | 5·4 (4)* |
| N(1) | 1463 (4) | 5887 (13) | 568 (11) | 2·2 (2) |
| N(2) | 1157 (4) | 9029 (13) | 375 (12) | 2·5 (2) |
| N(3) | 806 (4) | 11077 (14) | −1056 (13) | 3·4 (2) |
| N(4) | 1306 (5) | 9177 (15) | 3733 (14) | 3·2 (2) |
| N(1′) | 612 (4) | 6955 (12) | 1978 (11) | 2·3 (2) |
| N(2′) | 1570 (5) | 6032 (14) | 3908 (14) | 2·6 (2) |
| N(3′) | 2084 (4) | 4650 (14) | 5756 (12) | 2·9 (2) |
| C(1) | 2281 (4) | 7418 (22) | 940 (13) | 2·6 (2) |
| C(2) | 1873 (5) | 6510 (17) | −296 (16) | 3·0 (3) |
| C(3) | 1642 (4) | 7657 (21) | −1666 (13) | 2·8 (2) |
| C(4) | 1314 (5) | 9009 (16) | −1100 (14) | 2·9 (3) |
| C(5) | 855 (5) | 10311 (16) | 383 (15) | 2·6 (2) |
| C(6) | 1092 (5) | 10268 (19) | −1969 (16) | 3·4 (3) |
| C(1′) | −117 (5) | 4985 (20) | 1965 (17) | 3·3 (3) |
| C(2′) | 481 (5) | 5302 (15) | 2461 (14) | 2·2 (2) |
| C(3′) | 660 (6) | 5060 (19) | 4249 (17) | 3·5 (3) |
| C(4′) | 1254 (5) | 5071 (16) | 4702 (15) | 2·5 (2) |
| C(5′) | 2088 (5) | 5745 (16) | 4614 (15) | 2·5 (2) |
| C(6′) | 1583 (5) | 4202 (17) | 5845 (16) | 2·9 (3) |
| H(N1)1 | 1597 | 4806 | 1165 | 3·5 |
| H(N1)2 | 1097 | 5715 | −249 | 3·5 |
| H(C2) | 2066 | 5527 | −823 | 3·5 |
| H(C3)1 | 1960 | 8185 | −2195 | 3·5 |
| H(C3)2 | 1395 | 7006 | −2637 | 3·5 |
| H(C5) | 664 | 10671 | 1408 | 3·5 |
| H(N3) | 587 | 12151 | −1379 | 3·5 |
| H(C6) | 1143 | 560 | 6800 | 3·5 |
| H(N1′)1 | 426 | 7786 | 2663 | 3·5 |
| H(N1′)2 | 402 | 7195 | 804 | 3·5 |
| H(C2′) | 703 | 4486 | 1879 | 3·5 |
| H(C3′)1 | 487 | 6022 | 4892 | 3·5 |
| H(C3′)2 | 496 | 3922 | 4591 | 3·5 |
| H(C5′) | 2445 | 6325 | 4297 | 3·5 |
| H(N3′) | 2432 | 4188 | 6497 | 3·5 |
| H(C6′) | 1464 | 3425 | 6690 | 3·5 |

* To these atoms were assigned anisotropic temperature factors. The equivalent isotropic thermal parameter is taken as $B = \frac{4}{3}(\sum_i \sum_j \beta_{ij}\,\mathbf{a}_i . \mathbf{a}_j)$.

The coordination around Co is distorted octahedral. One of the histidinato ligands is coordinated to Co through carboxylato O, amino N and imidazole N atoms. The second one is bidentate, with imidazole N and amino N as donor atoms. The two imidazole rings are in *trans* positions while the amino groups are *cis* to each other. The nitro group is therefore *trans* to the

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* Lists of structure factors, anisotropic thermal parameters and least-squares planes (Table 3) have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 36145 (10 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

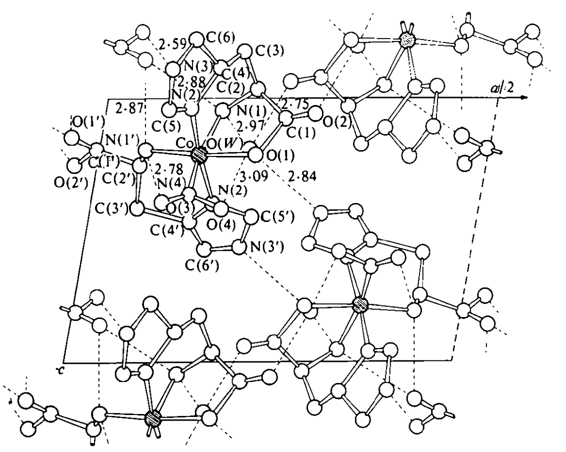

Fig. 1. View of the crystal structure down **b**. A unique set of the atoms is labelled. H atoms are omitted for clarity. Hydrogen bonds are shown by broken lines.

Table 2. *Molecular geometry of* (−)-[CoNO$_2$(L-his)$_2$].H$_2$O

(*a*) Bond lengths (Å)

| | | | |
|---|---|---|---|
| Co—O(1) | 1·902 (8) | Co—N(1′) | 1·952 (11) |
| Co—N(1) | 1·936 (10) | Co—N(2′) | 1·916 (12) |
| Co—N(2) | 1·958 (10) | N(4)—O(3) | 1·210 (18) |
| Co—N(4) | 1·954 (12) | N(4)—O(4) | 1·200 (16) |
| C(1)—O(1) | 1·288 (14) | C(1′)—O(1′) | 1·226 (18) |
| C(1)—O(2) | 1·235 (13) | C(1′)—O(2′) | 1·266 (21) |
| C(1)—C(2) | 1·529 (18) | C(1′)—C(2′) | 1·528 (18) |
| C(2)—N(1) | 1·472 (16) | C(2′)—N(1′) | 1·489 (16) |
| C(2)—C(3) | 1·529 (18) | C(2′)—C(3′) | 1·503 (18) |
| C(3)—C(4) | 1·528 (19) | C(3′)—C(4′) | 1·492 (29) |
| C(4)—N(2) | 1·371 (15) | C(4′)—N(2′) | 1·389 (18) |
| N(2)—C(5) | 1·316 (17) | N(2′)—C(5′) | 1·366 (18) |
| C(5)—N(3) | 1·353 (17) | C(5′)—N(3′) | 1·325 (17) |
| N(3)—C(6) | 1·333 (17) | N(3′)—C(6′) | 1·345 (16) |
| C(6)—C(4) | 1·342 (20) | C(6′)—C(4′) | 1·362 (18) |

(*b*) Bond angles (°)

| | | | |
|---|---|---|---|
| O(1)—Co—N(1) | 84·4 (4) | O(1)—Co—N(1′) | 175·5 (4) |
| O(1)—Co—N(2) | 88·9 (4) | O(1)—Co—N(2′) | 91·0 (4) |
| O(1)—Co—N(4) | 92·4 (4) | N(1)—Co—N(1′) | 92·1 (5) |
| N(1)—Co—N(2) | 88·8 (4) | N(1)—Co—N(2′) | 92·1 (5) |
| N(1)—Co—N(4) | 176·7 (5) | N(2)—Co—N(1′) | 88·2 (5) |
| N(2)—Co—N(4) | 90·7 (5) | N(2)—Co—N(2′) | 179·1 (5) |
| N(4)—Co—N(1′) | 91·1 (5) | N(1′)—Co—N(2′) | 92·0 (5) |
| N(4)—Co—N(2′) | 88·4 (5) | Co—N(4)—O(3) | 121·6 (10) |
| O(3)—N(4)—O(4) | 119·8 (12) | Co—N(4)—O(4) | 118·6 (10) |
| O(1)—C(1)—O(2) | 123·5 (12) | O(1′)—C(1′)—O(2′) | 128·9 (13) |
| O(1)—C(1)—C(2) | 114·5 (9) | O(1′)—C(1′)—C(2′) | 119·0 (13) |
| O(2)—C(1)—C(2) | 122·0 (11) | O(2′)—C(1′)—C(2′) | 112·0 (12) |
| C(1)—C(2)—N(1) | 107·1 (10) | C(1′)—C(2′)—N(1′) | 110·3 (10) |
| C(1)—C(2)—C(3) | 108·9 (12) | C(1′)—C(2′)—C(3′) | 110·8 (11) |
| Co—N(1)—C(2) | 106·7 (9) | Co—N(1′)—C(2′) | 117·4 (8) |
| N(1)—C(2)—C(3) | 112·4 (10) | N(1′)—C(2′)—C(3′) | 110·8 (10) |
| C(2)—C(3)—C(4) | 112·5 (10) | C(2′)—C(3′)—C(4′) | 111·3 (12) |
| C(3)—C(4)—N(2) | 123·9 (11) | C(3′)—C(4′)—N(2′) | 122·1 (12) |
| C(3)—C(4)—C(6) | 127·2 (11) | C(3′)—C(4′)—C(6′) | 130·0 (12) |
| C(4)—N(2)—Co | 126·7 (9) | C(4′)—N(2′)—Co | 129·6 (10) |
| C(4)—N(2)—C(5) | 106·4 (11) | C(4′)—N(2′)—C(5) | 106·8 (11) |
| C(5)—N(2)—Co | 126·9 (9) | C(5′)—N(2′)—Co | 123·5 (10) |
| N(2)—C(5)—N(3) | 109·6 (11) | N(2′)—C(5′)—N(3′) | 107·5 (11) |
| C(5)—N(3)—C(6) | 107·8 (11) | C(5′)—N(3′)—C(6′) | 111·3 (11) |
| N(3)—C(6)—C(4) | 107·5 (11) | N(3′)—C(6′)—C(4′) | 106·4 (12) |
| C(6)—C(4)—N(2) | 108·6 (11) | C(6′)—C(4′)—N(2′) | 108·0 (12) |
| Co—O(1)—C(1) | 114·9 (7) | | |

amino N from the tridentate histidine ligand. The absolute configuration about the Co atom is $\Lambda$.

The tridentate coordination by L-his is similar to that observed in all the Co$^{III}$ complexes so far investigated: [Co(L-his)(D-his)] (Thorup, 1977), [Co(L-his)(D-pen)] (De Meester & Hodgson, 1977), [Co-(L-hydroxybenzylhis)(L-ala)] (Voss, Angelici & Jacobson, 1978), as well as in a number of other metal complexes with histidine (Harding & Long, 1968; Fraser & Harding, 1967; Candlin & Harding, 1970; Delbaere & Prout, 1971; Spivack & Dori, 1975; Freeman, Guss, Healy & Martin, 1969; Sakurai, Iwasaki, Katano & Nakahashi, 1978). In the crystal structure of [Cd(L-his)$_2$] (Fuess & Bartunik, 1976), the Co–O bond length is 0·2 Å longer than the Co—N bond lengths. Metal—oxygen distances are even longer in complexes [Zn(D-his)(L-his)] (Harding & Cole, 1963), [Zn(D-his)$_2$] (Kretsinger, Cotton & Bryan, 1963) and [Cu(L-his)(L-asn)] (Ono, Shimanouchi, Sasada, Sakurai, Yamauchi & Nakahara, 1979); the principal coordination sites of histidine in these compounds are imidazole and amino N atoms, with a close approach of carboxylato O atoms. In the structures of (−)-[CoNO$_2$(L-his)$_2$].H$_2$O and [Cu(L-his)(D-his)(H$_2$O$_2$)].4H$_2$O (Camerman, Fawcett, Kruck, Sarkar & Camerman, 1978), the carboxylato group is out of the coordination sphere of the metal atom, demonstrating imidazole N–amino N bidentate chelation. In all of the investigated complexes, the histidine moiety adopts a closed form, only the bidentate histidine in [CoNO$_2$(L-his)$_2$] is in an extended conformation.

Table 2 presents interatomic distances and angles in the molecule. The Co—O and Co—N bond distances and angles are in agreement with values previously observed in the Co$^{III}$ amino acidato complexes. The largest deviation from octahedral geometry occurs for the O(1)—Co—N(1) angle of the five-membered chelate ring, usually explained as a result of constraints imposed upon chelation of an amino acid to a metal atom.

The bond distances and angles in the histidinato ligands are comparable with the corresponding values in the structures of other Co$^{III}$ histidine compounds (Thorup, 1977; De Meester & Hodgson, 1977; Voss *et al.*, 1978). Differences which exist between the two histidine moieties are less than 3$\sigma$ and do not seem to be significant. The five-membered chelate ring is in an asymmetric envelope conformation, imposed by the attachment of the imidazole side chain to the Co atom. The imidazole rings are planar and in *trans* positions, the configuration which is considered to be favoured

(Camerman *et al.*, 1978). The Co atom deviates only 0·02 and 0·04 Å from the planes of the two imidazole rings (Table 3).*

The geometry of the coordinated nitro group is similar to that found in related complexes (Watson, Johnson, Ćelap & Kamberi, 1972; Herak & Prelesnik, 1976; Herak, Prelesnik, Manojlović-Muir & Muir, 1974; Vasić, Herak & Djurić, 1976).

Extensive hydrogen bonding exists in the crystal. All the O atoms are engaged in intra- and intermolecular hydrogen bonds of the type O—H···O and N—H···O (Fig. 1). The most significant interaction exists between the N(3) atom from the imidazole ring of the tridentate histidine ligand and the O(2′) atom of the uncoordinated carboxylato group of the bidentate histidine in another adjacent complex molecule. Short N(imidazole)—O(carboxyl) distances are apparent in the structures of orthorhombic and monoclinic L-histidine (Madden, McGandy & Seeman, 1972; Madden, McGandy, Seeman, Harding & Hoy, 1972) and L-*N*-acetylhistidine (Kistenmacher, Hunt & Marsh, 1972). The N(3)—H(7)···O(2′) hydrogen bond of 2·59 Å is, to the best of our knowledge, one of the shortest hydrogen bonds of this type.

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* See deposition footnote.

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# Structure of Sodium Methanesulfonate*

By Chin Hsuan Wei and B. E. Hingerty

*Biology Division, Oak Ridge National Laboratory, Oak Ridge, Tennessee* 37830, *USA*

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## Abstract

Sodium methanesulfonate, $NaCH_3SO_3$, crystallizes in the orthorhombic system, space group *Pbma*, with $a = 17{\cdot}0713\ (6)$, $b = 22{\cdot}0217\ (6)$, $c = 5{\cdot}6058\ (3)$ Å, $Z = 20$, $V = 2107{\cdot}4$ Å$^3$, $D_c = 1{\cdot}861$, $D_o = 1{\cdot}88$ Mg m$^{-3}$ (flotation in bromobenzene/$CHBr_3$), $M_r = 118{\cdot}09$, $F(000) = 5200$, $\mu$(Cu $K\alpha$) = 6·55 mm$^{-1}$, transmission = 0·294–0·822 at 297 K. The structure was solved by the heavy-atom technique and refined by the full-matrix least-squares method to $R(F) = 0{\cdot}027$ based on 1953

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