

The Crystal and Molecular Structure of Tetrakis[μ -propionato]bis(nicotinamide)dicopper(II)

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The crystal and molecular structure of the tetrakis[μ -propionato]bis(nicotinamide)-dicopper(II), $[\text{Cu}(\text{CH}_3\text{CH}_2\text{COO})_2(\text{nia})_2]_2$ (nia = nicotinamide), was determined by direct methods and Fourier techniques. The compound crystallizes in the triclinic space group *P1* with two dimeric formula units in a cell with dimensions $a = 9.895(7)$, $b = 11.204(7)$, $c = 13.509(7)$ Å, $\alpha = 87.36(5)$, $\beta = 89.17(5)$ and $\gamma = 74.25(5)^\circ$. The structure was refined by full-matrix least-squares methods to a weighted *R* factor of 0.059, based on 2830 independent reflections. The compound is dimeric, with square-pyramidal geometry at each copper centre. The two copper atoms are bridged by four carboxylate groups, while the apical ligands are nicotinamides. The Cu–Cu distance is 2.635(2) Å. The Cu atoms are displaced by 0.20 Å from the plane containing four oxygen atoms towards the apical nicotinamides. The structural data are compared with those found in similar copper(II) propionates and with the nicotinamide adduct of copper(II) acetate.

The nature of the copper–copper interaction in binuclear copper(II) carboxylates has been a subject of much discussion since the structure of copper(II) acetate monohydrate, as the first of its type, was reported in 1953.¹ Extensive work on dimeric copper(II) carboxylates employing spectroscopic, magnetic and X-ray crystallographic techniques has been reported.^{2–7} The effects of the carboxylate substituents and of the axial donor ligands on the exchange coupling constant ($-2J$),⁸ and also on the Cu–Cu distances,^{5–7} have attracted great interest.

The present paper describes the crystal and molecular structure of tetrakis(μ -propionato)bis(nicotinamide)dicopper(II). Structural data are now available for seven binuclear copper(II) propionate compounds. These data will be analysed later.

Experimental

Preparation of the complex. Light green $\text{Cu}(\text{CH}_3\text{CH}_2\text{COO})_2\text{nia}$ (nia = nicotinamide) was prepared by treating nicotinamide (1.22 g, 0.01 mol) with copper(II) propionate (2.28 g, 0.01 mol) in hot methanol solution. The solution was left to stand at room temperature. The fine microcrystals that precipitated were filtered off (ca. 70% yield), washed with cold methanol and dried at room temperature. The crude product was recrystallized from methanol with a yield of 60%.

Calc. for $\text{Cu}(\text{CH}_3\text{CH}_2\text{COO})_2\text{nia}$: Cu, 19.15; C, 43.44; H, 4.86; N, 8.44%. Found: Cu, 19.16; C, 43.74; H, 5.04; N, 8.78%.

Data collection. The crystal and refinement data are given in Table 1. The unit-cell parameters and the orientation matrix were determined by a least-squares refinement based on 24 well-centred reflections measured at 20 °C on a Nicolet R3m diffractometer.

Intensity data were collected on the same diffractometer by the ω -scan technique using graphite-crystal monochromated $\text{MoK}\alpha$ radiation ($\lambda = 0.71069$ Å). The intensity of one reference reflection, recorded after every 99 measurements, remained essentially constant throughout data collection. The intensities were corrected for Lorentz and polarization effects and for absorption from empirical ψ -scan data from eight reflections.

Structure determination. The structure was solved by direct methods and Fourier techniques and refined by blocked-cascade full-matrix least-squares methods with anisotropic temperature factors for all non-hydrogen atoms.

The hydrogen atoms bonded to carbon atoms were included at calculated positions with a fixed bond length (C–H = 0.96 Å) and constrained angles, and their thermal parameters were set at 0.080 Å². The hydrogen atoms of the amino groups were located from a difference Fourier map.

The calculations were performed on a Nicolet R3m diffractometer system with SHELXTL software for the minicomputer MicroVAX II. The program uses neutral

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Table 1. Crystal data, data collection and refinement data.

Formula	C ₂₄ H ₃₂ Cu ₂ O ₁₀ N ₄
Formula weight	663.68
Crystal system	triclinic
Space group	<i>P</i> 1
<i>a</i> /Å	9.895(7)
<i>b</i> /Å	11.204(7)
<i>c</i> /Å	13.509(7)
α /°	87.36(5)
β /°	89.17(5)
γ /°	74.25(5)
<i>V</i> /Å ³	1439.8(1.5)
<i>Z</i>	2
<i>D_c</i> /g cm ⁻³	1.53
<i>D_x</i> /g cm ⁻³	1.53
λ /Å (MoK α)	0.71069
Monochromator	graphite
Linear absorption coefficient/cm ⁻¹	15.4
Crystal size/mm	0.20×0.24×0.31
Diffractometer	Nicolet R3m
Data collection method	ω
2 θ range/°	4 to 50
Scan rate/° min ⁻¹	2.5 to 29.3
No. of standard reflections	1
Variation in standard intensities	±1 %
Reflections collected	5026
Reflections used in refinement <i>I</i> > 2.5 σ (<i>I</i>)	2830
No. of variables	373
$R = \sum \Delta / \sum F_o $ ^a	0.062
$R = \sum \Delta^{1/2} / \sum F_o w^{1/2}$ ^b	0.059
Residual electron density/e Å ⁻³	0.70
<i>T</i> /K	293
<i>F</i> (000)/e	684
<i>h</i> range	0–11
<i>k</i> range	–13 to 13
<i>l</i> range	–16 to 16

$$^a \Delta = ||F_o - |F_c||. \quad ^b w^{-1} = \sigma^2(F_o) + 0.0005F_o^2.$$

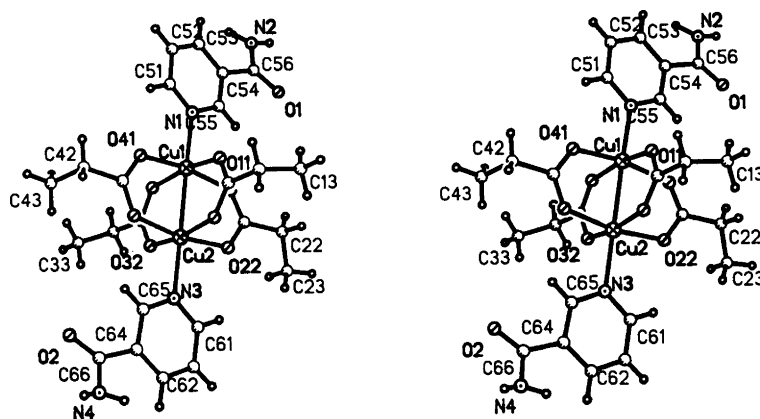
atom scattering factors from Ref. 9 and takes anomalous dispersion into account. The figures were drawn with the SHELXTL program on a Hewlett–Packard-plotter.

The final atomic coordinates and equivalent isotropic thermal parameters with their estimated standard deviations for non-hydrogen atoms are given in Table 2.

Table 2. Atomic coordinates ($\times 10^4$) and equivalent isotropic temperature factors ($\times 10^3$ Å²) for Cu₂(C₃H₅O₂)₄(C₆H₆N₂O)₂.

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> ^a
Cu(1)	4085(1)	5508(1)	2888(1)	29(1)
Cu(2)	4420(1)	7698(1)	2340(1)	31(1)
O(11)	2976(6)	6470(5)	3973(5)	45(2)
O(12)	3283(6)	8317(5)	3538(5)	42(2)
C(11)	2754(9)	7627(8)	4083(6)	34(3)
C(12)	1775(11)	8150(11)	4902(9)	63(5)
C(13)	300(14)	8247(14)	4630(11)	111(8)
O(21)	2406(6)	6041(5)	2054(5)	39(2)
O(22)	2618(6)	7933(6)	1636(5)	49(3)
C(21)	1940(9)	7117(8)	1657(7)	38(3)
C(22)	496(10)	7483(9)	1227(9)	60(4)
C(23)	282(13)	8332(12)	347(10)	96(7)
O(31)	5315(6)	4873(5)	1728(4)	38(2)
O(32)	5464(7)	6767(5)	1232(5)	47(3)
C(31)	5775(9)	5601(8)	1158(7)	36(3)
C(32)	6799(10)	5055(9)	346(8)	47(4)
C(33)	8178(11)	5272(13)	449(11)	99(7)
O(41)	5813(7)	5304(6)	3642(5)	47(3)
O(42)	6111(6)	7157(6)	3151(5)	48(3)
C(41)	6489(9)	6128(9)	3615(7)	39(4)
C(42)	7871(10)	5805(10)	4167(8)	58(4)
C(43)	9081(14)	5502(17)	3540(12)	134(9)
N(1)	3769(7)	3710(6)	3328(5)	33(3)
N(2)	1204(8)	693(7)	2795(8)	51(3)
O(1)	280(7)	2723(6)	2400(6)	68(3)
C(51)	4612(9)	2961(8)	3966(7)	39(3)
C(52)	4478(11)	1796(9)	4271(8)	52(4)
C(53)	3395(9)	1391(8)	3856(7)	42(4)
C(54)	2488(8)	2172(7)	3200(6)	29(3)
C(55)	2698(8)	3316(7)	2947(6)	32(3)
C(56)	1212(9)	1890(8)	2761(7)	41(4)
N(3)	4726(7)	9469(6)	1783(6)	34(3)
O(2)	8475(7)	10308(6)	2333(7)	76(3)
N(4)	7474(9)	12367(8)	2103(9)	58(4)
C(61)	3853(9)	10216(8)	1130(7)	38(3)
C(62)	4059(10)	11311(8)	759(7)	48(4)
C(63)	5212(10)	11658(9)	1069(8)	46(4)
C(64)	6136(8)	10918(8)	1726(7)	35(3)
C(65)	5863(9)	9826(7)	2069(7)	33(3)
C(66)	7461(10)	11168(9)	2084(8)	47(4)

^aEquivalent isotropic *U* defined as one third of the trace of the orthogonalized *U_{ij}* tensor.

Fig. 1. Stereoview of Cu(CH₃CH₂COO)₂nia (nia = nicotinamide).

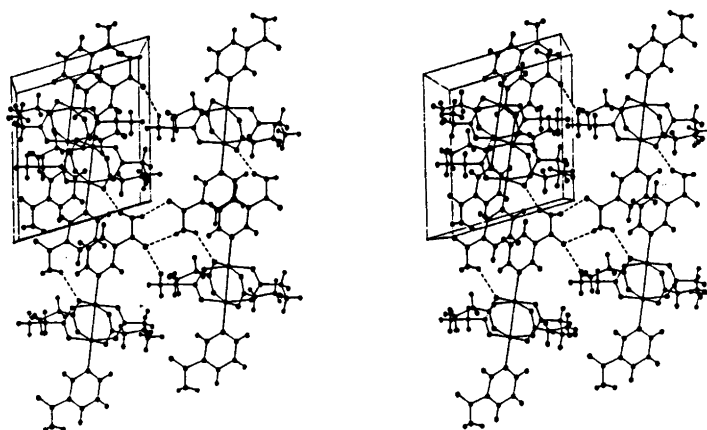


Fig. 2. Stereoview of the crystal packing.

Results and discussion

Description of the structure. A stereoview of the crystal structure of $[\text{Cu}(\text{CH}_3\text{CH}_2\text{COO})_2(\text{nia})]_2$ with labelling of atoms is shown in Fig. 1. The crystal packing is shown in Fig. 2. The compound exists as a dimer consisting of two distorted square-based pyramidal copper(II) polyhedra joined by four carboxylic groups of propionato ions with a nicotinamide molecule coordinated through the nitrogens of the pyridine ring at each apex at a distance of 2.176(8) Å for Cu(1) and 2.186(8) Å for Cu(2). The bridge, structurally similar to that found in copper(II) carboxylates,⁶ is almost planar and contains crystallographically non-equivalent Cu–O bond distances and bond angles (Tables 3 and 4). The displacement of the copper(II) atom from the basal plane towards the apical ligand by 0.20 Å is in agreement with that found in most square-pyramidal copper(II) compounds.⁶

This molecule follows the trends already noted for this type of dimer. The Cu–Cu separation [2.635(2) Å] is similar to those observed in other Cu(II) propionate compounds (Table 5). Correlations between metal–metal separations and bond lengths or angles in the Cu–Cu–O–C–O rings have been discussed.^{6,16} The Cu–Cu–O angle has been found to be most sensitive to a change in the Cu–Cu distance. From the equation derived by Koh and Christoph,¹⁶ the Cu–Cu distance in the present study should correspond to a Cu–Cu–O angle of 84.0°, and the average value of the Cu–Cu–O angles is in fact found to be 84.1°. Our results for other parameters also compare well with those found for

other copper(II) propionate compounds (Table 5). There exists a relationship between the displacement of the copper(II) atom from the basal plane toward the apical ligand, and the increase in the copper–copper distance.⁶ The Cu(II) atom in the present compound is displaced by 0.20 Å, which is in agreement with this trend.

Ideally, the cage should have a fourfold axis along the Cu–Cu direction. Deviation from this symmetry is significant, but not very large. The dihedral angle between adjacent Cu–Cu–O–C–O rings deviates by only about 2° from the ideal 90° value. The steric effect of the –CONH₂ group of the nicotinamide molecule introduces small distortions. For instance, nitrogens N(1) and N(3) of the nicotinamide molecules do not lie exactly along the Cu–Cu axis: the angles Cu(2)–Cu(1)–N(1) and Cu(1)–Cu(2)–N(3) are 178.9(2) and 176.0(2)°, respectively.

The Cu(1)–N(1) and Cu(2)–N(3) bonds to the apical nicotinamide, which are 2.176(8) and 2.186(8) Å, respectively, are somewhat longer than those of 2.168(5) Å found in dimeric $[\text{Cu}(\text{CH}_3\text{COO})_2(\text{nia})]_2 \cdot 4\text{H}_2\text{O}$.¹⁷ In all other respects the geometry of the coordinated nicotinamide in this study does not differ appreciably from that of the Cu(II) acetate adduct.¹⁷ Two crystallographically independent dimeric units, differing mostly in their degree of distortion (Table 5), have been found in the structures of Cu(prop)₂py and Cu(prop)₂4pic.^{11,15} The existence of two species or two isomeric forms with the same coordination number about the central atom, differing mostly in their degree of distortion within the same crystal, is typical of the general class of distortion isomerism.^{18,19}

Table 3. Bond lengths (in Å).

The copper environments		The propionato ligands		The nicotinamide ligands	
Cu(1)–Cu(2)	2.635(2)	O(11)–C(11)	1.270(11)	N(1)–C(51)	1.311(11)
Cu(1)–O(11)	1.993(6)	O(12)–C(11)	1.253(11)	N(1)–C(55)	1.368(12)
Cu(1)–O(21)	1.960(6)	C(11)–C(12)	1.492(14)	N(2)–C(56)	1.342(13)
Cu(1)–O(31)	2.005(6)	C(12)–C(13)	1.485(18)	O(1)–C(56)	1.2090(10)
Cu(1)–O(41)	1.957(7)	O(21)–C(21)	1.264(10)	C(51)–C(52)	1.390(14)
Cu(1)–N(1)	2.176(8)	O(22)–C(21)	1.272(12)	C(52)–C(53)	1.406(16)
Cu(2)–O(12)	1.998(6)	C(21)–C(22)	1.493(13)	C(53)–C(55)	1.372(11)
Cu(2)–O(22)	1.981(7)	C(22)–C(23)	1.469(17)	C(54)–C(55)	1.380(12)
Cu(2)–O(32)	1.978(6)	O(31)–C(31)	1.265(11)	C(54)–C(56)	1.516(13)
Cu(2)–O(42)	1.952(6)	O(32)–C(31)	1.267(11)	N(3)–C(61)	1.336(11)
Cu(2)–N(3)	2.186(8)	C(31)–C(32)	1.515(13)	N(3)–C(65)	1.358(12)
		C(32)–C(33)	1.460(16)	O(2)–C(66)	1.226(10)
		O(41)–C(41)	1.277(13)	N(4)–C(66)	1.349(14)
		O(42)–C(41)	1.253(11)	C(61)–C(62)	1.368(14)
		C(41)–C(42)	1.514(13)	C(62)–C(63)	1.378(15)
		C(42)–C(43)	1.429(17)	C(63)–C(64)	1.362(12)
				C(64)–C(65)	1.382(13)
				C(64)–C(66)	1.503(14)

Table 4. Bond angles (in °).

The copper environments		The propionato ligands		The nicotinamide ligands	
Cu(2)–Cu(1)–O(11)	82.2(2)	Cu(1)–O(11)–C(11)	126.2(6)	Cu(1)–N(1)–C(51)	120.7(7)
Cu(2)–Cu(1)–O(21)	83.8(2)	Cu(2)–O(12)–C(11)	121.9(5)	Cu(1)–N(1)–C(55)	121.6(5)
O(11)–Cu(1)–O(21)	88.2(2)	O(11)–C(11)–O(12)	123.6(8)	C(51)–N(1)–C(55)	117.7(8)
Cu(2)–Cu(1)–O(31)	85.8(2)	O(11)–C(11)–C(12)	115.9(9)	N(1)–C(51)–C(52)	123.6(9)
O(11)–Cu(1)–O(31)	167.9(3)	O(12)–C(11)–C(12)	120.5(8)	C(51)–C(52)–C(53)	118.1(8)
O(21)–Cu(1)–O(31)	92.2(2)	O(11)–C(12)–C(13)	10.8(10)	C(52)–C(53)–C(54)	119.0(9)
Cu(2)–Cu(1)–O(41)	84.7(2)	Cu(1)–O(21)–C(21)	124.9(6)	C(53)–C(54)–C(55)	118.7(8)
O(11)–Cu(1)–O(41)	90.1(3)	Cu(2)–O(22)–C(21)	123.0(5)	C(53)–C(54)–C(56)	124.3(8)
O(21)–Cu(1)–O(41)	168.4(3)	O(21)–C(21)–O(22)	123.3(8)	C(55)–C(54)–C(56)	116.9(7)
O(31)–Cu(1)–O(41)	87.0(3)	O(21)–C(21)–C(22)	119.2(9)	N(1)–C(55)–C(54)	122.9(7)
Cu(2)–Cu(1)–N(1)	178.9(2)	O(22)–C(21)–C(22)	117.5(8)	N(2)–C(56)–O(1)	123.7(9)
O(11)–Cu(1)–N(1)	97.6(3)	C(21)–C(22)–C(23)	116.3(10)	N(2)–C(56)–C(54)	116.3(7)
O(21)–Cu(1)–N(1)	95.2(3)	Cu(1)–O(31)–C(31)	121.0(5)	O(1)–C(56)–C(54)	120.0(9)
O(31)–Cu(1)–N(1)	94.5(3)	Cu(2)–O(32)–C(31)	125.7(6)	Cu(2)–N(3)–C(61)	122.3(7)
O(41)–Cu(1)–N(1)	96.4(3)	O(31)–C(31)–O(32)	124.3(8)	Cu(2)–N(3)–C(65)	120.2(5)
Cu(1)–Cu(2)–O(12)	85.9(2)	O(31)–C(31)–C(32)	118.6(8)	C(61)–N(3)–C(65)	117.4(8)
Cu(1)–Cu(2)–O(22)	84.4(2)	O(32)–C(31)–C(32)	117.1(8)	N(3)–C(61)–C(62)	122.7(9)
O(12)–Cu(2)–O(22)	87.2(3)	C(31)–C(32)–C(33)	113.6(10)	C(61)–C(62)–C(63)	118.8(8)
Cu(1)–Cu(2)–O(32)	82.6(2)	Cu(1)–O(41)–C(41)	122.3(5)	C(62)–C(63)–C(64)	120.5(9)
O(12)–Cu(2)–O(32)	168.5(3)	Cu(2)–O(42)–C(41)	124.1(7)	C(63)–C(64)–C(65)	117.6(9)
O(22)–Cu(2)–O(32)	90.9(3)	O(41)–C(41)–O(42)	125.0(8)	C(63)–C(64)–C(66)	125.4(9)
Cu(1)–Cu(2)–O(42)	83.8(2)	O(41)–C(41)–C(42)	116.7(8)	C(65)–C(64)–C(66)	117.0(7)
O(12)–Cu(2)–O(42)	90.3(3)	O(42)–C(41)–C(42)	118.2(9)	N(3)–C(65)–C(64)	123.1(7)
O(22)–Cu(2)–O(42)	168.1(3)	C(41)–C(42)–C(43)	114.1(10)	N(4)–C(66)–O(2)	122.6(10)
O(32)–Cu(2)–O(42)	89.3(3)			N(4)–C(66)–C(64)	116.9(8)
Cu(1)–Cu(2)–N(3)	176.0(2)			O(2)–C(66)–C(64)	120.5(9)
O(12)–Cu(2)–N(3)	97.8(3)				
O(22)–Cu(2)–N(3)	94.3(3)				
O(32)–Cu(2)–N(3)	93.7(3)				
O(42)–Cu(2)–N(3)	97.6(3)				

Table 5. Structural data for binuclear copper(II) propionate compounds.^a

Compound ^b	Crystal class	Space group	Z	a/Å b/Å c/Å	α/° β/° γ/°	Cu—O (basal) /Å	Cu—L (apical) /Å	Cu—Cu/Å Cu—O—C—O—Cu/Å	O—C—O/° Cu—Cu—L _{ax} /°	Ref.
Cu(prop) ₂ (dox) _{0.5}	m	P2 ₁ /c	4	9.009(2) 8.137(2) 15.121(4)	109.92(5)	1.953(2,5)	O ^c 2.227(3)	2.5634(4) 6.42	125.1(3,1) 178(1)	10
Cu(prop) ₂ py ^d	tr	P $\bar{1}$	4	16.204(9) 8.442(5) 11.618(7)	103.9(1) 121.0(1) 90.4(1)	1.97(1,2) 1.96(1,0)	N 2.13(1) N 2.17(1)	2.619(2) 6.42 2.642(2) 6.42	127(1,1) 175(1) 126(1,1) 173(1)	11
Cu(prop) ₂ nia	tr	P $\bar{1}$	2	9.895(7) 11.204(7) 13.509(7)	87.36(5) 89.17(5) 74.25(5)	1.978(6,27)	N 2.181(8,5)	2.635(2) 6.48	124.1(8,1.8) 177.5(2,1.5)	This work
Cu(prop) ₂ 3pic	m	C2/c	8	20.702(4) 7.306(2) 18.957(4)	107.78(5)	1.970(2,12)	N 2.167(2)	2.6312(4) 6.44	124.2(1,5) 175(1)	12
Cu(prop) ₂ 3,5lut	tr	P $\bar{1}$	1	8.6652(4) 8.7357(6) 10.8223(4)	77.31(8) 67.67(13) 85.69(9)	1.975(3,4)	N 2.168(2)	2.6447(6) 6.44	126.0(3,1) 177.42(6)	13
Cu(prop) ₂ 2pic	tr	P $\bar{1}$	2	8.080(7) 8.290(7) 12.444(10)	111.9(2) 75.7(1) 119.4(1)	1.97(1,1)	N 2.21(2)	2.647(4) 6.44	126.5(1,1.5) 179(1)	14
Cu(prop) ₂ 4pic ^d	or	C _{oom}	8	20.300(3) 14.244(3) 20.479(6)		1.970(9,3) 1.972(9,22)	N 2.143(9) N 2.148(9)	2.655(3) 6.46 2.659(3) 6.44	125(1) 180(1) 124(1) 180(1)	15

^aWhere more than one chemically equivalent distance or angle is present, the mean value is tabulated. The first number in parenthesis is the s.d., and the second is the maximum deviation from the mean value. ^bdox, 1,4 dioxane; py, pyridine; 3pic, 3-picoline; 3,5lut, 3,5-lutidine; 2pic, 2-picoline; 4pic, 4-picoline; nia, nicotinamide. ^cThe chemical identity of the coordinated atom. ^dThere are two crystallographically independent dimeric units.

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