

A bis-salicylaldoximato-copper(II) receptor for selective sulfate uptake

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Received (in XXX, XXX) Xth XXXXXXXXXX 200X, Accepted Xth XXXXXXXXXX 200X

First published on the web Xth XXXXXXXXXX 200X

5 DOI: 10.1039/b000000x

Further experimental details:

Experimental section

Synthesis of **L**: N,N'-Dimethyl-N,N'-hexamethylenedi(3-hydroxyiminomethyl-2-hydroxy-5-tert-
10 butylbenzylamine) (**L**) was prepared as reported previously.¹

Synthesis of complexes [SO₄⊂Cu₂L₂]Cl₂·2.5H₂O and [SO₄⊂Cu₂L₂](NO₃)₂·H₂O·3EtOH·MeCN: A suspension of **L** (100 mg, 0.18 mmol) in 15 mL methanol was heated to 40°C and 4 equivalents of either NaCl or NaNO₃, dissolved in 4 mL water was added. CuSO₄ (29 mg, 0.18 mmol) dissolved in 3 mL water was added and an
15 immediate colour change to green was observed. The reaction mixture was cooled down to room temperature and stirred overnight. The volume of the reaction mixture was reduce *in vacuo*. The crude reaction product was filtered, washed with diethyl ether (2 x 5 mL) and dried.

Synthesis of [SO₄⊂Cu₂L₂](H₂PO₄)₂: A suspension of **L** (105 mg, 0.19 mmol) in 15 mL methanol was heated to
20 40°C and K₂HPO₄ (66 mg, 0.38 mmol), followed by CuSO₄ (31 mg, 0.19 mmol), each dissolved in 3 mL water were added. After 3 drops of ortho-phosphoric acid were added to the reaction mixture changed colour to dark green. The reaction mixture was refluxed (30 mins.), cooled to RT and stirred overnight. The dark green solution was reduced in volume resulting in the formation of a dark green precipitate. This was collected by filtration and washed with diethyl ether (2 x 5 mL). The crude complex was crystallised by vapour diffusion of
25 diethyl ether into an ethanol/acetonitrile mixture (1:1) and dried.

Synthesis of [SO₄⊂Cu₂L₂](NO₃)₂·2H₂O: A suspension of **L** (0.28 mmol, 154 mg) in 15 mL methanol was heated to 40°C, 2 equivalents of K₂HPO₄ (0.56 mmol, 97 mg) and NaNO₃ (0.56 mmol, 47 mg) and one equivalent of
30 CuSO₄ (0.28 mmol, 45 mg), each dissolved in 3 mL water, were added. The reaction mixture was stirred at 40°C (30 mins.), cooled to RT and stirred overnight. The solution was reduced in volume resulting in the formation of a bright green precipitate. This was collected by filtration and washed with diethyl ether (2 x 5 mL). The crude complex was crystallised by vapour diffusion of diethyl ether into an ethanol / acetonitrile mixture (1:1) and dried. Yield: 86%, High-Res. ESI-MS m/z 665.2922 [Cu₂L₂SO₄]²⁺, anal calc. for C₆₄H₁₀₀Cu₂N₁₀O₁₈S·2H₂O: C 51.50, H 7.02, N 9.38, S 2.15%, found: C 51.27, H 7.27, N 9.54, S 1.99%.

Synthesis of [NO₃⊂Cu₂L₂](NO₃)₃·2H₂O: A suspension of **L** (95 mg, 0.17 mmol) in 15 mL methanol was heated to 40°C and treated with Cu(NO₃)₂·3H₂O (42 mg, 0.17 mmol), dissolved in 3 mL water. The reaction mixture was cooled to RT and stirred overnight. The volume was reduced and the crude product was filtered, washed with diethyl ether (2 x 5 mL) and dried *in vacuo*. Yield 92%, MS(ESI) m/z 649 [Cu₂L₂NO₃-H]²⁺; anal calc. for
40 C₆₄H₁₀₀Cu₂N₁₂O₂₀·2H₂O: C 50.55, H 6.89, N 11.05%, found: C 50.59, H 6.79, N 10.89%.

Liquid-Liquid Extraction Protocol

Bulong: The complex Cu₂(L-2H)₂ (0.05 M) in CHCl₃ (1 mL) was stirred intensely (24h) at RT with Na₂SO₄ (1.0
45 M) and NaCl (2.25 M) in H₂O (1 mL) under constant ionic strength and pH (HEPES/HCl pH 5.0).

100 times excess: Cu₂(L-2H)₂ (0.005 M) in CHCl₃ (1 mL) was stirred intensely (24h) at RT with the sodium salt of the desired anion (0.5 M) in H₂O (1 mL) combinations of Na₂SO₄+NaNO₃, Na₂SO₄+NaCl and Na₂SO₄+KH₂PO₄ –1:100 excess, respectively), buffered with HEPES/HCl to pH of 3.5

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The organic phases were then spun to separate phases. The CHCl_3 phase was then diluted with MeOH in a 1:1 ratio and transferred to the ESMS.

Crystal structure descriptions:

$[\text{SO}_4\text{Cu}_2\text{L}_2](\text{NO}_3)_2$: the SO_4^{2-} anion is disorder over two positions with an occupancy of 0.8 : 0.2. In $[\text{SO}_4\text{Cu}_2\text{L}_2]\text{Cl}_2$ one of the two Cl^- anions are disordered over two positions with an occupancy of 0.9 : 0.1. Only the major species are considered in the following discussion.

The receptor is formed by two Cu(II) centres, coordinated by two head-to-tail hydrogen-bonded salicylaldoxime units of two ligand molecules. Interligand hydrogen bonding between the oximic hydrogen atoms and phenolate oxygen atoms ($\text{O}\cdots\text{O}$ distances of 2.606(4) to 2.696(4) Å) support the well know trans coordination of Cu(II) by two salicylaldoxime units.²⁻⁶ The bis-salicylaldoximato units bind the Cu(II) centres in distorted square pyramidal Cu(II)-coordination spheres ($\tau^7 = 0.30$ for Cu1 and 0.32 for Cu2 in $[\text{SO}_4\text{Cu}_2\text{L}_2]\text{Cl}_2$ and $\tau^7 = 0.37$ for Cu1 and 0.22 for Cu2 in $[\text{SO}_4\text{Cu}_2\text{L}_2](\text{NO}_3)_2$, respectively) arising from Cu-O- SO_3 interactions toward the encapsulated SO_4^{2-} anion (Cu-O distances of 2.257(3) to 2.294(3) Å) (Table S1 and S3). The Cu-Cu distances are 6.6004(15) and 6.5368(18) Å in $[\text{SO}_4\text{Cu}_2\text{L}_2]\text{Cl}_2$ and $[\text{SO}_4\text{Cu}_2\text{L}_2](\text{NO}_3)_2$, respectively.

In both structures the ligand L is in its zwitterionic form with phenol groups deprotonated and tertiary amines in the 'straps' protonated. Intramolecular H-bonds are formed between these subunits with N-H \cdots O distances of 2.40 to 2.55 Å (N \cdots O distances: 2.975(4) to 3.083(4) Å; N-H \cdots O angles: 118 to 122 ° (see Table S2 and S4). The encapsulated SO_4^{2-} anion is bound by multiple H-bonds of the protonated tertiary amines (N-H \cdots O distances: 1.88 to 2.47 Å; N \cdots O distances: 2.707(5) to 3.229(5) Å, N-H \cdots O angles: 141 to 162 °) and β -methylene substituents on the 'straps' (C-H \cdots O distance 2.33 to 2.49 Å; C \cdots O distance 3.100(5) to 3.422(5) Å; C-H \cdots O angle 136 to 169°) (Fig. S1 and S2 and Table S2 and S4)

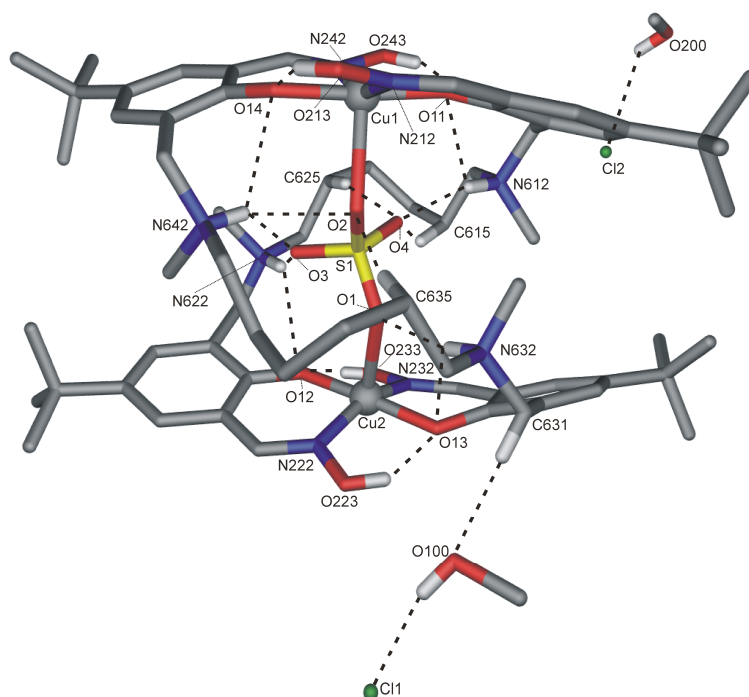


Fig. S1. Structure of $[\text{SO}_4\text{Cu}_2\text{L}_2]\text{Cl}_2 \cdot 2\text{MeOH}$ showing selected intramolecular interactions (dotted lines) with atom labels. Major species only are shown (Cl3 deleted), non H-bonded hydrogen atoms have also been omitted for clarity.

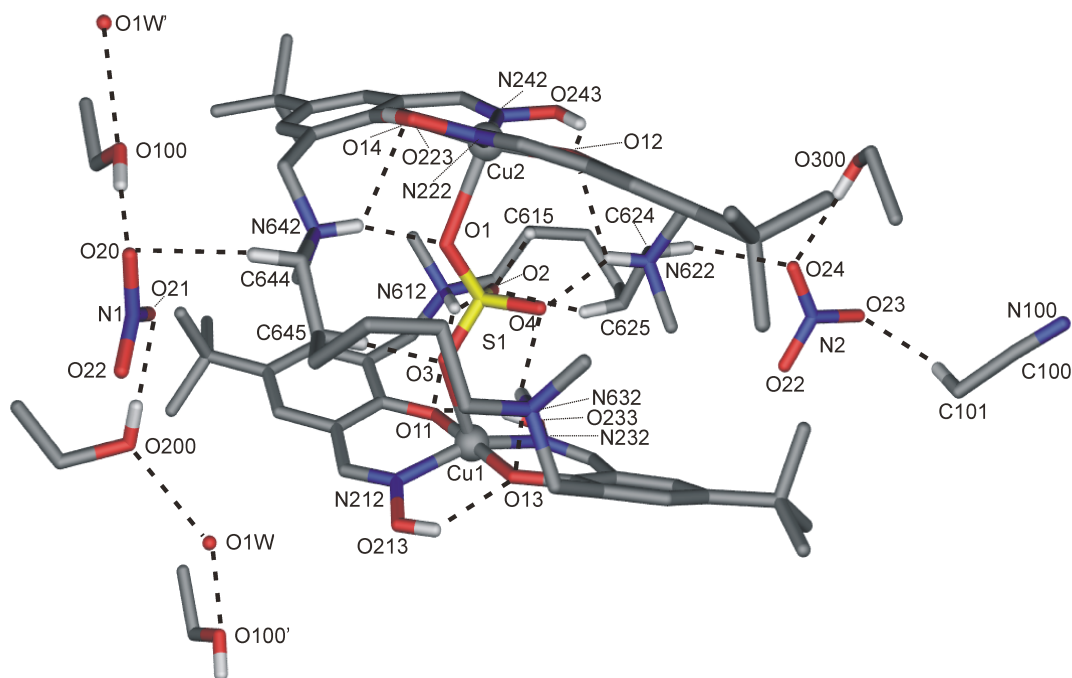


Fig. S2. Structure of $[\text{SO}_4\text{Cu}_2\text{L}_2](\text{NO}_3)_2 \cdot \text{H}_2\text{O} \cdot 3\text{EtOH} \cdot \text{MeCN}$ showing selected intramolecular interactions (dotted lines) with atom labels. Major species only shown, non H-bonded hydrogen atoms have been omitted for clarity

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From the above figures it can be seen that the spherical chloride (Fig. S1) and the trigonal planar nitrate anions (Fig. S2), are placed outside the $[\text{SO}_4\text{Cu}_2\text{L}_2]^{2+}$ cationic complex. Both types of anions are involved in numerous H-bonds towards co-crystallised solvent molecules and connected to the complex cation by weak CH-O interactions (Fig. S1 and S2). A summary of the weak interactions in $[\text{SO}_4\text{Cu}_2\text{L}_2]\text{Cl}_2 \cdot 2\text{EtOH}$ and

10 $[\text{SO}_4\text{Cu}_2\text{L}_2](\text{NO}_3)_2 \cdot \text{H}_2\text{O} \cdot 3\text{EtOH} \cdot \text{MeCN}$ is listed in Table S2 and S4.

Additional information for $[\text{SO}_4\text{Cu}_2\text{L}_2]\text{Cl}_2 \cdot 2\text{EtOH}$

Table S1. Selected bond lengths and angles in $[\text{SO}_4\text{Cu}_2\text{L}_2]\text{Cl}_2 \cdot 2\text{EtOH}$

atoms	Bond lengths [Å]	atoms	Angles [°]	atoms	Angles [°]
Cu1-O2	2.294(3)	O2-Cu1-O11	98.69(12)	O11-Cu1-N212	91.27(13)
Cu1-O11	1.897(3)	O2-Cu1-O14	85.24(12)	O11-Cu1-N242	87.94(13)
Cu1-O14	1.899(3)	O2-Cu1-N212	83.44(12)	O14-Cu1-N212	89.02(13)
Cu1-N212	1.966(3)	O2-Cu1-N242	118.21(12)	O14-Cu1-N242	90.34(13)
Cu1-N242	1.969(3)	O11-Cu1-O14	176.07(12)	N212-Cu1-N242	158.21(13)
Cu2-O1	2.277(3)	O1-Cu2-O12	101.66(12)	O12-Cu2-N222	91.87(14)
Cu2-O12	1.910(3)	O1-Cu2-O13	81.92(11)	O12-Cu2-N232	86.90(13)
Cu2-O13	1.917(3)	O1-Cu2-N222	89.93(12)	O13-Cu2-N222	88.69(14)
Cu2-N222	1.979(3)	O1-Cu2-N232	112.50(12)	O13-Cu2-N232	91.21(13)
Cu2-N232	1.981(3)	O12-Cu2-O13	176.38(12)	N222-Cu2-N232	157.33(13)

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Table S2. H-bond interactions in [SO₄Cu₂L₂]Cl₂·2EtOH

D-H	A	D-H [Å]	H-A [Å]	D-A [Å]	D-H-A[°]
O213-H213	O14	0.82	2.01	2.650(4)	134
O223-H223	O13	0.82	2.06	2.696(4)	134
O233-H233	O12	0.82	1.96	2.610(4)	135
O243-H243	O11	0.82	1.96	2.606(4)	135
N612-H612	O11	0.91	2.55	3.083(4)	118
N622-H622	O12	0.91	2.50	3.031(4)	118
N632-H632	O13	0.91	2.40	2.975(4)	121
N642-H642	O14	0.91	2.50	3.040(4)	119
N612-H612	O4	0.91	1.88	2.707(5)	150
N622-H622	O3	0.91	1.92	2.754(4)	151
N632-H632	O1	0.91	2.02	2.898(4)	162
N642-H642	O2	0.91	2.40	3.229(5)	152
N642-H642	O3	0.91	2.47	3.186(4)	136
C615-H61I	O4	0.97	2.33	3.100(5)	136
C625-H62H	O4	0.97	2.45	3.389(5)	163
C635-H63H	O2	0.97	2.46	3.422(5)	169
O100-H100	Cl1 ⁱ	0.82	2.36	3.113(4)	154
O200-H200	Cl2	0.82	2.53	3.145(5)	133
C631-H63B	O100	0.97	2.37	3.303(6)	161
C422-H42C	Cg1 ⁱⁱ	0.96	2.62	3.549 (6)	163
C444-H44G	Cg2 ⁱⁱⁱ	0.96	2.70	3.581(5)	153

Symmetry codes: i = 1-x,-y,-z; ii = 1-x,-1/2+y,1/2-z; iii = 1-x,1/2+y,1/2-z

CH- π -stacking in $[\text{SO}_4\text{Cu}_2\text{L}_2]\text{Cl}_2\cdot 2\text{EtOH}$:

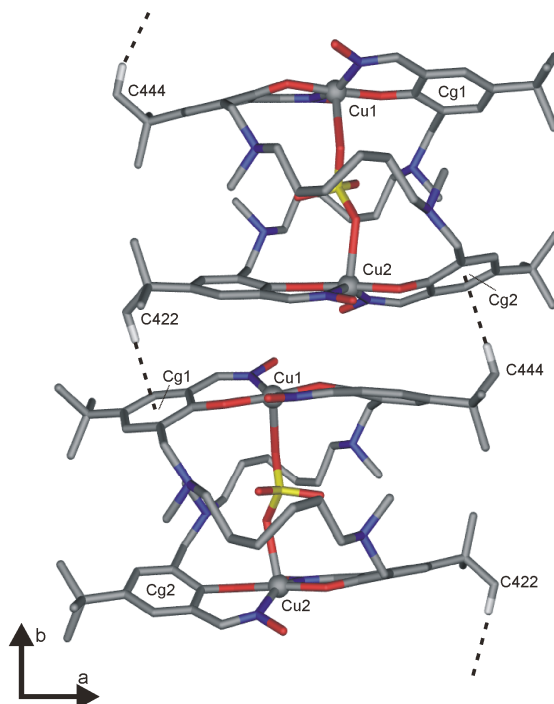


Fig. S3. CH- π -stacking in $[\text{SO}_4\text{Cu}_2\text{L}_2]\text{Cl}_2\cdot 2\text{EtOH}$, selected atom labels, Cl^- anions, EtOH and non CH- π interaction H-atoms removed for clarity. Viewed along the c-axis.

Additional information for $[\text{SO}_4\text{Cu}_2\text{L}_2](\text{NO}_3)_2\cdot \text{H}_2\text{O}\cdot 3\text{EtOH}\cdot \text{MeCN}$

Table S3. Selected bond lengths and angles in $[\text{SO}_4\text{Cu}_2\text{L}_2](\text{NO}_3)_2\cdot \text{H}_2\text{O}\cdot 3\text{EtOH}\cdot \text{MeCN}$

atoms	bond lengs [Å]	atoms	angles [°]	atoms	angles [°]
Cu1-O3	2.266(4)	O3-Cu1-O11	96.09(17)	O11-Cu1-N212	90.68(16)
Cu1-O11	1.898(3)	O3-Cu1-O13	85.91(17)	O11-Cu1-N232	87.67(16)
Cu1-O13	1.907(3)	O3-Cu1-N212	82.82(17)	O13-Cu1-N212	89.68(16)
Cu1-N212	1.979(4)	O3-Cu1-N232	121.61(18)	O13-Cu1-N232	91.18(16)
Cu1-N232	1.975(4)	O11-Cu1-O13	177.99(14)	N212-Cu1-N232	155.55(18)
Cu2-O1	2.257(3)	O1-Cu2-O12	103.64(13)	O12-Cu2-N222	91.83(15)
Cu2-O12	1.922(3)	O1-Cu2-O14	83.15(13)	O12-Cu2-N242	88.58(15)
Cu2-O14	1.919(3)	O1-Cu2-N222	92.32(15)	O14-Cu2-N222	87.66(15)
Cu2-N222	1.971(4)	O1-Cu2-N242	106.81(15)	O14-Cu2-N242	89.61(15)
Cu2-N242	1.985(4)	O12-Cu2-O14	173.20(14)	N222-Cu2-N242	160.21(18)

Table S4: H-bond interactions in $[\text{SO}_4\text{Cu}_2\text{L}_2](\text{NO}_3)_2 \cdot \text{H}_2\text{O} \cdot 3\text{EtOH} \cdot \text{MeCN}$

D-H	A	D-H [Å]	H-A [Å]	D-A [Å]	D-H-A[°]
O213-H213	O13	0.82	2.07	2.695(5)	133
O223-H223	O14	0.82	1.96	2.612(5)	136
O233-H233	O11	0.82	2.01	2.642(5)	134
O243-H243	O12	0.82	2.00	2.649(5)	136
N612-H612	O11	0.91	2.48	3.048(5)	121
N622-H622	O12	0.91	2.45	3.015(5)	120
N632-H632	O13	0.91	2.44	2.992(5)	119
N642-H642	O14	0.91	2.44	3.019(5)	122
N612-H612	O2	0.91	1.96	2.745(7)	143
N622-H622	O4	0.91	2.10	2.873(6)	142
N632-H632	O4	0.91	2.30	3.058(6)	141
N642-H642	O1	0.91	1.97	2.844(5)	160
C615-H61H	O2	0.97	2.32	3.137(8)	141
C625-H62I	O2	0.97	2.47	3.384(8)	156
C645-H64H	O3	0.97	2.46	3.394(6)	162
O100-H10A	O20	0.82	1.90	2.704(8)	166
O200-H20A	O21	0.82	2.00	2.819(11)	177
O300-H30A	O24	0.82	1.92	2.735(8)	179
C611-H61B	O23	0.97	2.44	3.377(7)	164
C624-H62F	O24 ⁱ	0.97	2.49	3.341(7)	145
C644-H64F	O20 ⁱⁱ	0.97	2.48	3.439(7)	172
C101-H10C	O23 ⁱⁱⁱ	0.96	2.45	3.347(12)	156
C634-H63F	O1W ⁱⁱⁱ	0.97	2.45	3.310(8)	148
O100	O1W			2.721(11)	
O200	O1W ^{iv}			2.799(9)	

Symmetry codes: i = 1-x, 1-y, -z; ii = -x, 1-y, 1-z; iii = 1+x, y, z; iv = -1+x, y, z.

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Additional information for $[\text{SO}_4\text{Cu}_2\text{L}_2](\text{HPO}_4)_2$

The Cu(II) coordination sphere, Cu...N, Cu...O bond lengths and angles and other key parameters such as the Cu...Cu (6.5599(13) Å) and Cu...O distances to the encapsulated oxo-anion (Cu1...O1 2.260(4) Å and Cu2...O2 2.263(4) Å) are in agreement to the observed values in $[\text{SO}_4\text{Cu}_2\text{L}_2]\text{Cl}_2 \cdot 2\text{EtOH}$ and $[\text{SO}_4\text{Cu}_2\text{L}_2](\text{NO}_3)_2 \cdot \text{H}_2\text{O} \cdot 3\text{EtOH} \cdot \text{MeCN}$.

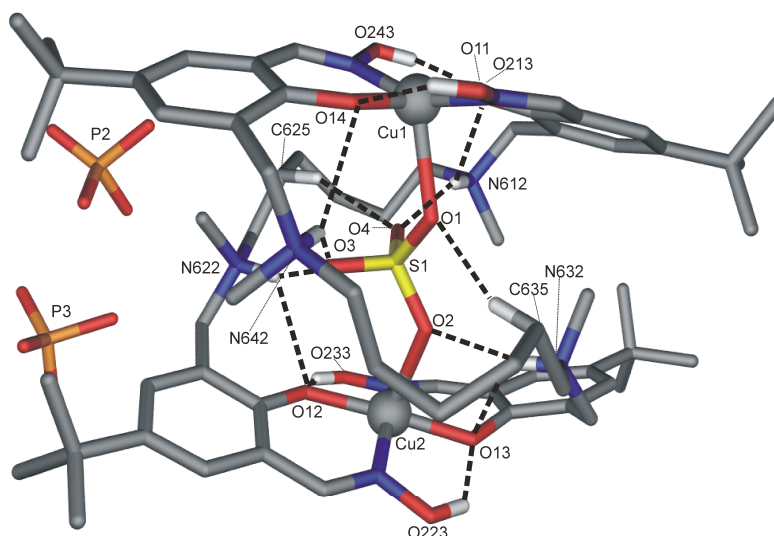


Fig. S4. Structure of $[\text{SO}_4\text{Cu}_2\text{L}_2](\text{H}_2\text{PO}_4)_2$ showing selected intramolecular interactions (dotted lines) with atom labels. Non H-bonded hydrogen atoms have been omitted for clarity.

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Additional information for $[\text{NO}_3\text{Cu}_2\text{L}_2](\text{NO}_3)_3 \cdot 2\text{H}_2\text{O}$

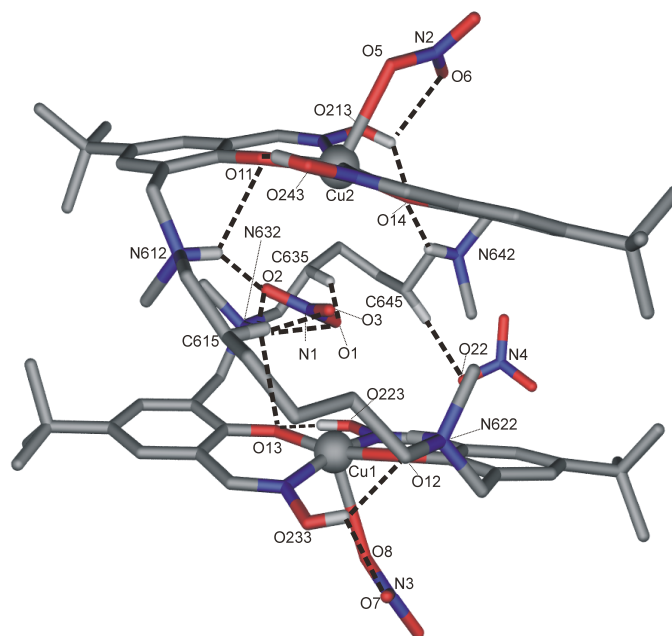


Fig. S5. Structure of $[\text{NO}_3\text{Cu}_2\text{L}_2](\text{NO}_3)_3 \cdot 2\text{H}_2\text{O}$ showing intramolecular interactions (dotted lines), selected atom labels, major species only, non H-bonded H atoms and solvent molecules omitted for clarity of structure.

In $[\text{NO}_3\text{Cu}_2\text{L}_2](\text{NO}_3)_3 \cdot 2\text{H}_2\text{O}$ one nitrate anion (N1) is encapsulated into the metallo-macrocycle formed by two Cu(II) centres, coordinated by two head-to-tail hydrogen-bonded salicylaldehyde units of two ligand molecules. Interligand hydrogen bonding between the oximic hydrogen atoms and phenolate oxygen atoms ($\text{O}\cdots\text{O}$ distances of 2.598(5) to 2.976(5) Å) support the well known trans coordination of Cu(II) by two salicylaldehyde units.²⁻⁶ The bis-salicylaldoximate units bind the Cu(II) centres in distorted square pyramidal Cu(II)-coordination spheres ($\tau^7 = 0.06$ for Cu1 and 0 for Cu2) arising from Cu-O-NO₂ interactions toward two nitrate anions, namely N2 and N3, which are placed outside the metallo-macrocycle (Cu-O-NO₂ distances of 2.556(4) and

2.664(6) Å (Table S5). The Cu-Cu distance is 6.6069(16) Å. The fourth nitrate anion and the aliphatic strap of one ligand is disorder over 2 positions with occupancy of 0.6 : 0.4. Only the major species is considered. Various intramolecular H-bonds support the formation of the 2:2 metallo-macrocyclic and bound to the encapsulated nitrate anion. A summary of the H-bonding in $[\text{NO}_3\text{C}\text{Cu}_2\text{L}_2](\text{NO}_3)_3$ is given in Table S6 and Fig. S5.

Table S5. Selected bond lengths and angles in $[\text{NO}_3\text{C}\text{Cu}_2\text{L}_2](\text{NO}_3)_3 \cdot 2\text{H}_2\text{O}$

atoms	bond lengths [Å]	atoms	angles [°]	atoms	angles [°]
Cu1-O5 ⁱ	2.556(4)	O5-Cu1-O11	110.08(15)	O11-Cu1-N212	90.42(17)
Cu1-O11	1.901(4)	O5-Cu1-O14	84.81(15)	O11-Cu1-N242	87.20(16)
Cu1-O14	1.901(4)	O5-Cu1-N212	86.52(15)	O14-Cu1-N212	94.54(17)
Cu1-N212	1.972(4)	O5-Cu1-N242	83.33(15)	O14-Cu1-N242	90.78(16)
Cu1-N242	1.962(4)	O11-Cu1-O14	164.60(16)	N212-Cu1-N242	168.07(16)
Cu2-O8	2.664(6)	O8-Cu2-O12	80.63(18)	O12-Cu2-N222	90.78(19)
Cu2-O12	1.909(4)	O8-Cu2-O13	111.90(18)	O12-Cu2-N232	93.09(17)
Cu2-O13	1.902(4)	O8-Cu2-N222	80.95(17)	O13-Cu2-N222	88.07(19)
Cu2-N222	1.956(5)	O8-Cu2-N232	87.09(17)	O13-Cu2-N232	90.96(18)
Cu2-N232	1.955(4)	O12-Cu2-O13	167.04(17)	N222-Cu2-N232	166.66(17)

Symmetry codes: $i = 1-x, 1/2+y, 1/2-z$.

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Table S6. H-bond interactions in $[\text{NO}_3\text{C}\text{Cu}_2\text{L}_2](\text{NO}_3)_3 \cdot 2\text{H}_2\text{O}$

D-H	A	D-H [Å]	H-A [Å]	D-A [Å]	D-H-A [°]
O213-H213	O14	0.82	2.39	2.976(5)	129
O223-H223	O13	0.82	2.00	2.635(5)	134
O233-H233	O12	0.82	2.28	2.885(5)	130
O243-H243	O11	0.82	1.96	2.598(5)	134
O213-H213	O6 ⁱ	0.82	2.29	2.684(6)	110
O233-H233	O7	0.82	2.27	2.641(6)	108
N612-H612	O11	0.91	2.42	3.005(5)	123
N622-H622	O12	0.91	1.81	2.605(7)	144
N632-H632	O13	0.91	2.55	3.091(7)	119
N642-H642	O14	0.91	1.92	2.669(7)	138
N612-H612	O2	0.91	2.21	2.990(11)	143
N632-H632	O1	0.91	2.35	3.195(8)	154
N632-H632	O2	0.91	2.50	3.226(11)	137
C615-H61H	O3	0.97	2.44	3.39(1)	165
C635-H63H	O1	0.97	2.42	3.27(3)	147
C645-H64I	O22	0.97	2.33	3.27(3)	164

Symmetry codes: $i = 1-x, 1/2+y, 1/2-z$.

Additional information for the UV-Vis titrations

The Jobs-plot for the acids H_3PO_4 , H_2SO_4 , HNO_3 , HI and HBr (Fig. S6) indicate the formation of a 1:1 anion to the $[\text{Cu}_2\text{L}_2]^{4+}$ host assembly, most likely an anion encapsulated species. In the case of HCl (Fig. S7) more complex species are formed, and supports evidence to the presence of another binding mode, where one ligand is displaced by chloride in related salicylaldoximato $\text{Cu}(\text{II})$ -complex. (R. S. Forgan, P. A. Tasker and P. G. Plieger, Unpublished Work, Massey University, Palmerston North, New Zealand, 2009.) In the ESMS obtained from the solutions used in the spectrophotometric titrations with HCl , no high m/z peak was detected with masses indicating that the metallomacrocyclic unit remained intact. In contrast, solutions containing the other anions all contained peaks corresponding to the metallomacrocycle containing an encapsulated anion, $[\text{X}^n-\text{Cu}_2\text{L}_2]^{(4-n)+}$.

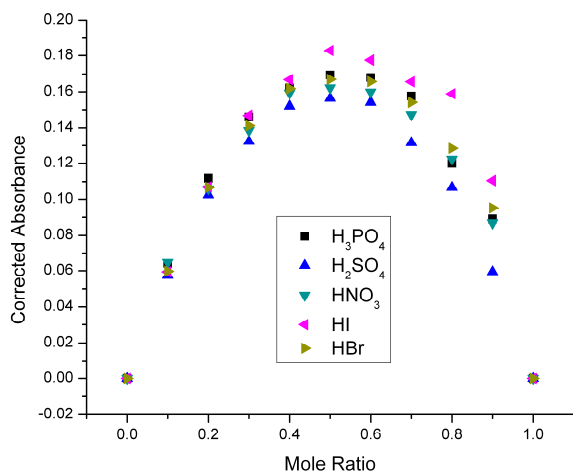


Fig. S6: Jobs plot of $[\text{Cu}_2\text{L}_2]^{4+}$ and H_3PO_4 , H_2SO_4 , HNO_3 , HI and HBr in isopropanol / 1,2-dichloroethane, observed wave lengths 355 nm.

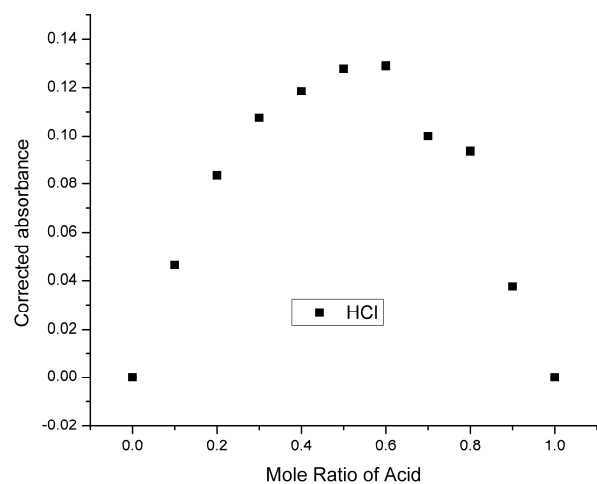
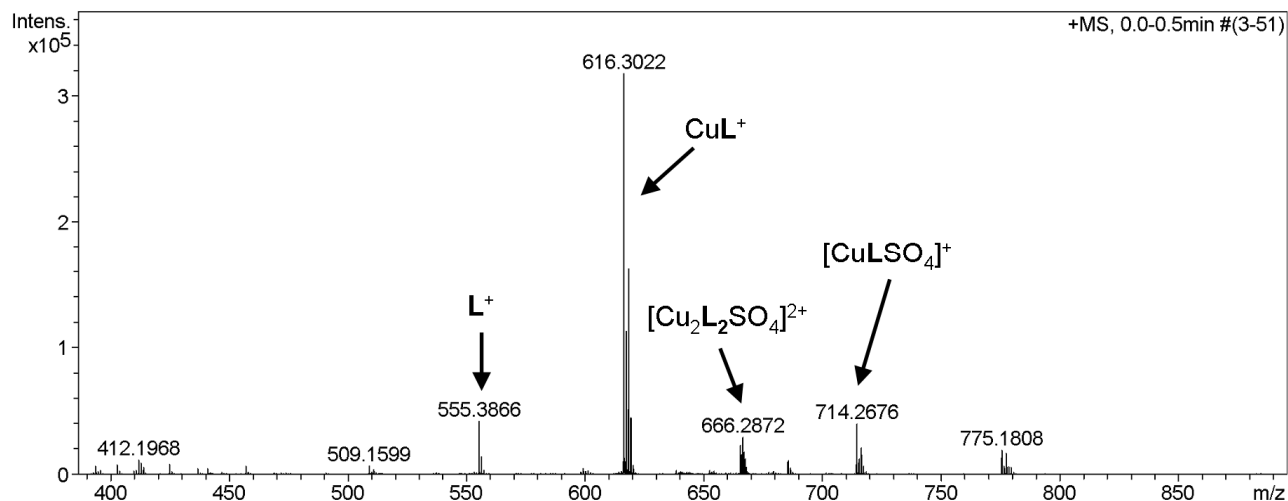


Fig. S7: Jobs plot of $[\text{Cu}_2\text{L}_2]^{4+}$ and HCl in isopropanol / 1,2-dichloroethane, observed wave lengths 355 nm.

High Resolution Mass Spectrum for $[\text{SO}_4\text{Cu}_2\text{L}_2](\text{HPO}_4)_2$



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