A New Ligand for the Formation of Triangular Building Blocks in Supramolecular Chemistry

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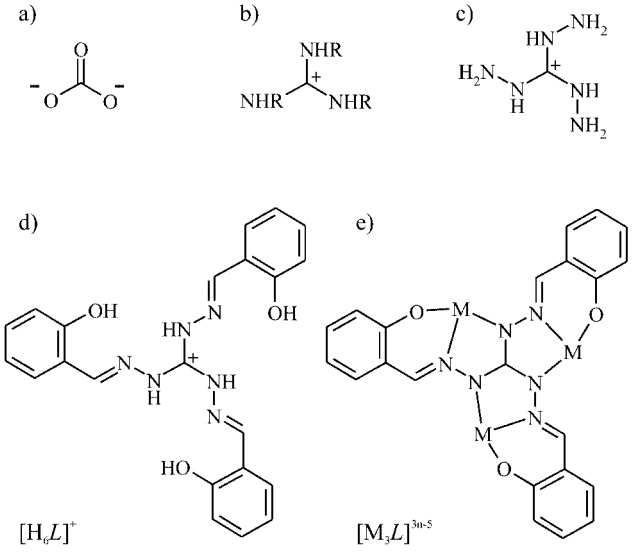
In this article we describe the synthesis, characterisation and metal binding properties of tris(5-bromo-2-hydroxybenzylidene)triaminoguanidinium chloride $[H_6Br_3L]Cl$ (**1**), which can be seen as a triangular building block for supramolecular chemistry. **1** crystallises either in a triclinic (**1a**) or a rhombohedral (**1b**) system depending on the used solvent . The pure ligand adopts a conformation unfavourable for the coordination of metal centres, but in the presence of a base or a basic metal salt, a change in the conformation can be observed. The reaction of **1** with $(NH_4)_6[Mo_7O_{24}]$ in the absence of further base results in the formation of small, red crystal needles with the formula $[Mo(O)_2(OH_2)(H_3Br_3L)]\cdot 2$ DMF (**2**), while the reaction of **1** with $CuCl_2$, sodium 5,5-diethylbarbiturate (NaH*bar*) and Et_3N leads to the formation of dark red-black crystals with the formula $(Et_4N)_2[\{Cu(Hbar)\}_3Br_3L]$ (**3**).

Introduction

There are two different ways of subdividing the rapidly growing field of supramolecular chemistry. One is by classifying the products that can be formed, namely polymers [1] or discrete cages and helical molecules.[2] The other way is based on the molecular building units, especially on the properties of the participating ligands. Up to now, the use of asymmetric or C_2 symmetric ligands has predominated, and despite their potential, far fewer C_3 symmetric ligands or ligands of higher symmetry have been employed for supramolecular coordination chemistry.[2] In particular, the C_3-symmetric ligands can be regarded as triangular building blocks, which should be suitable for the construction of most of the Platonic and Archimedic bodies. The smallest conceivable C_3 symmetric ligand is the carbonate dianion (Scheme 1, a). The guanidinium cation, which is isoelectronic with carbonic acid (Scheme 1, b; R = H), is known to be able to form metal complexes and can be modified chemically.[3]

A possible way of designing more stable products is to put the stabilizing chelate effect to use as in the case of the triaminoguanidinium cation (Scheme 1, c). Due to its character as a reducing agent, the introduction of protection groups is necessary, and if these also contain a donor atom, a tris(chelating) ligand with threefold symmetry will be formed (Scheme 1, d), which can be used to bind three metal centres as shown in Scheme 1 (e). The preferred coordination geometry of the metal centres determines the number and position of additional ligands. With the use of this strategy, different types of complexes, from monomeric species[4] to cage-like compounds with the outer shape of a doughnut,[4] a rectangular box,[5] a tetrahedron[6] and an octahedron,[5] can be prepared as described previously. The structural characterisation of large cage compounds by X-ray crystallography is often difficult due to rapid solvent loss and a typical high degree of disorder of both the counter ions and solvent molecules. The introduction of heavy atoms into the ligand should increase the scattering power of such oligomeric complexes and therefore improve the resolution and quality of the experimental X-ray data. With



Scheme 1

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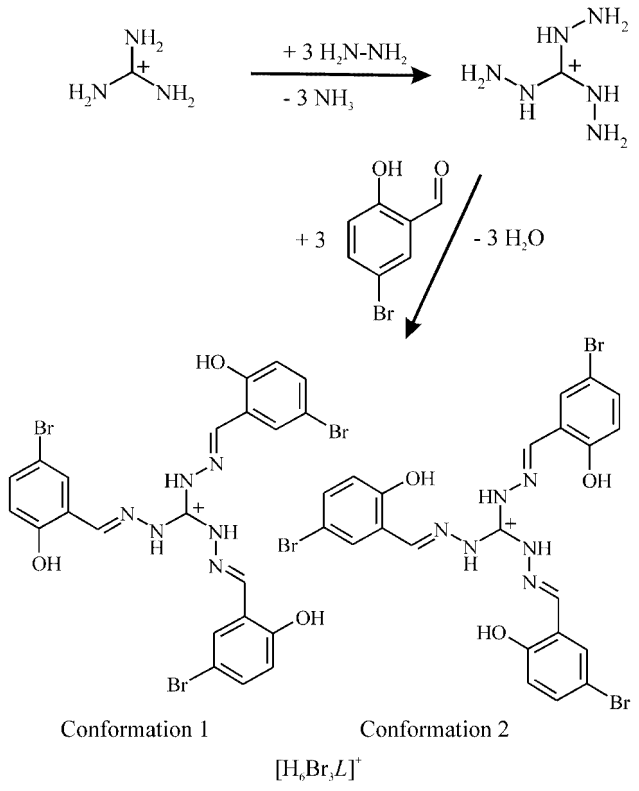
this goal in mind, we present here the synthesis and characterisation of tris(5-bromo-2-hydroxybenzylidene)triaminoguanidinium chloride and report on its metal binding properties.

Results and Discussion

Tris(5-bromo-2-hydroxybenzylidene)triaminoguanidinium chloride $[H_6Br_3L]Cl$ (**1**), can be easily obtained from triaminoguanidinium chloride and 5-bromosalicylaldehyde (Scheme 2).



Scheme 2

Diffusion of HCl into an aqueous solution of **1** leads to triclinic pale yellow crystals of **1a**, whose molecular structure (space group $P\bar{1}$) is shown in Figure 1 (a).[7] In the solid state, **1** adopts conformation 1 (Scheme 2), which is stabilized by close contacts between the hydroxy groups and the Cl^- anions (Table 1). This results in the formation of a two-dimensional hydrogen-bonded network.

Diffusion of HCl into a solution of **1** in DMF leads to rhombohedral pale yellow crystals of **1b** (space group $R\bar{3}$).[8] The conformation of the $[H_6Br_3L]^+$ cation is the same as that observed in **1a** (Figure 1, b). However, hydrogen bonds lead, in this case, to the formation of double layers, which enclose a layer of DMF molecules. The bond length and angles observed in **1a** and **1b** fall in the expected ranges. All carbon and nitrogen atoms show sp^2-hybridisation with the distance between the central carbon and the nitrogen atoms exhibiting an average value of 1.327(7) Å,

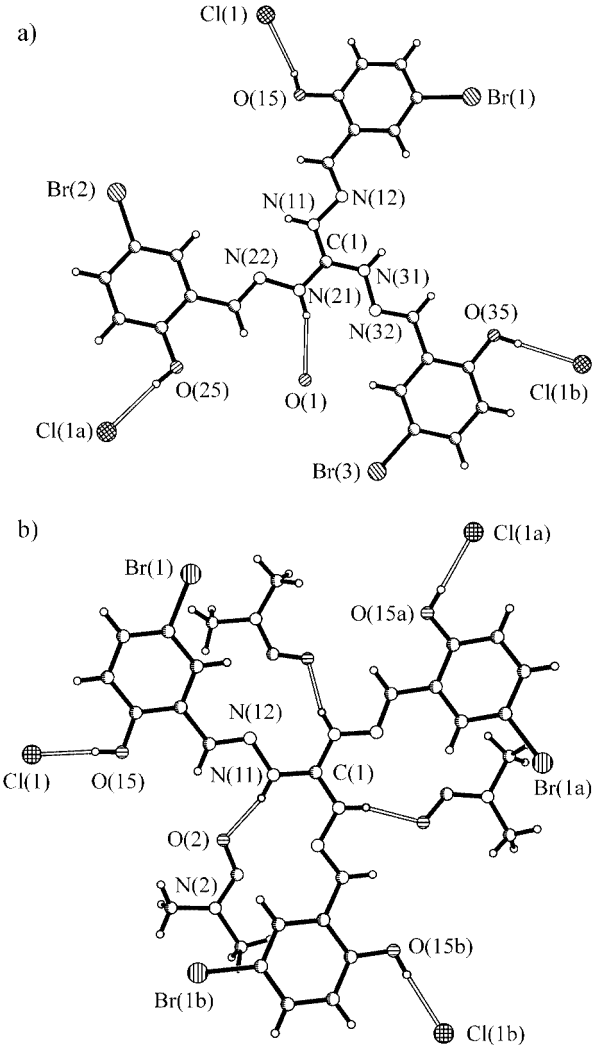


Figure 1. Crystal structures of **1**; a) **1a**; b) **1b**

Table 1. Hydrogen bonds (* = intramolecular)

	Proton	Bond to	Distance	Angle at H
1a	H15	Cl	2.49 Å	126°
	H25	Cl	2.27 Å	163°
	H35	Cl	2.30 Å	168°
	H21	O (H_2O)	2.19 Å	160°
	H11	N	2.31 Å*	101°*
	H21	N	2.33 Å*	101°*
	H31	N	2.32 Å*	102°*
1b	H15	Cl	2.15 Å	173°
	H11	O (DMF)	1.94 Å	165°
	H11	N	2.45 Å*	95°*
2	H25	O (DMF)	1.87 Å	160°
	H35	N	2.13 Å*	142°*
	H31	N	2.13 Å*	106°*
	H3a	O (DMF)	2.35 Å	123°
	H3b	N11	1.89 Å	175°
3	H43	O (*bar*)	2.04 Å	172°
	H53	O (*bar*)	1.98 Å	173°
	H63	N (CH_3CN)	2.24 Å	161°

in accordance with a partial double-bond character. A propeller-like distortion can be observed, and the dihedral angles between the central CN_6 core and the aromatic systems adopt values between 1.5° and 22.3°. The ^1H NMR spectrum of **1** contains only a few sharp signals. This suggests that in solution one conformation must be dominant (Figure 2, a). The very weak interaction between H3 and H4 seen in the H,H-NOESY experiment suggests that in solution only conformation 1 will be of importance. It therefore appeared interesting to determine how readily conformation 1 can be transformed into conformation 2, which is the required conformation for the coordination of metal centres. In high-temperature ^1H NMR experiments (up to 80 °C), a shift of the signals can be observed, but no line-broadening can be seen. This indicates that no change in the conformation takes place within this temperature range.

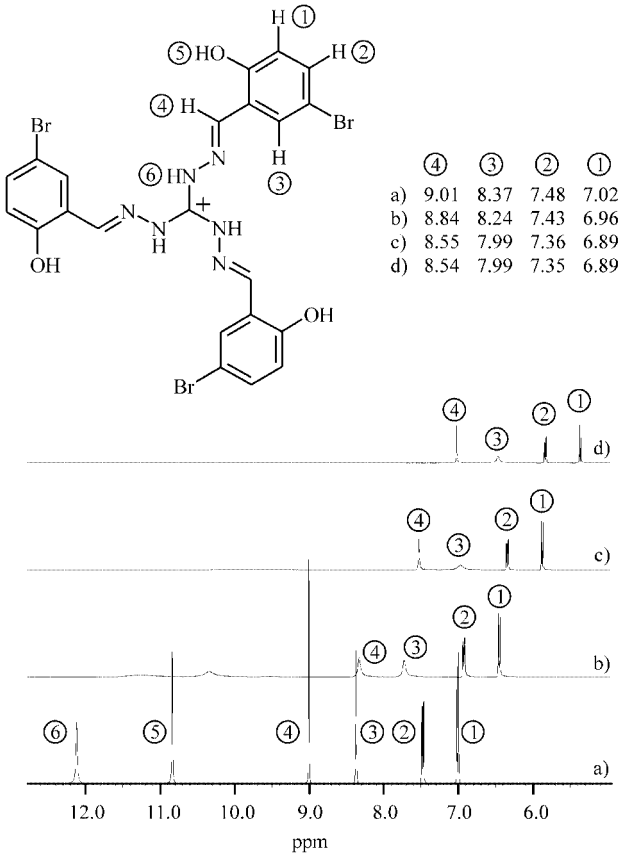


Figure 2. ^1H NMR in $[D_6]$DMSO and chemical shifts in ppm of **1**; a) pure solution; b) + 1 equivalent Et_3N; c) + 3 equivalents Et_3N; d) + 6 equivalents Et_3N

In contrast, the addition of one to six equivalents of base (triethylamine) to a solution of **1** leads to the upfield shift of the signals for H1−H4 and the broadening of the signals for H3 and H4 as depicted in Figure 2 (b−d). This phenomenon can be explained by the transformation of conformation 1 into conformation 2 upon deprotonation, with resulting long-range coupling between the affected protons, whose proximity is confirmed by the observed strengthening of their cross peak in the H,H-NOESY spectrum. In solution a base is able to transform the conformation of **1**, but can a metal ion achieve the same result by metalating its N and O atoms? The reaction of $[H_6Br_3L]Cl$ with neutral $CdCl_2$ in acetone leads, for instance, even at higher temperatures, only to the formation of crystals with the formula $[H_6Br_3L]_2[CdCl_4]$, in which the ligand still adopts conformation 1.[9]

The reaction of **1** with $(NH_4)_6[Mo_7O_{24}]$ as a basic metal salt results in the formation of small, red crystal needles. The X-ray structure shows a complex with the formula $[Mo(O)_2(OH_2)(H_3Br_3L)]\cdot 2DMF$ (**2**), whose asymmetric unit is shown in Figure 3 (a).[10]

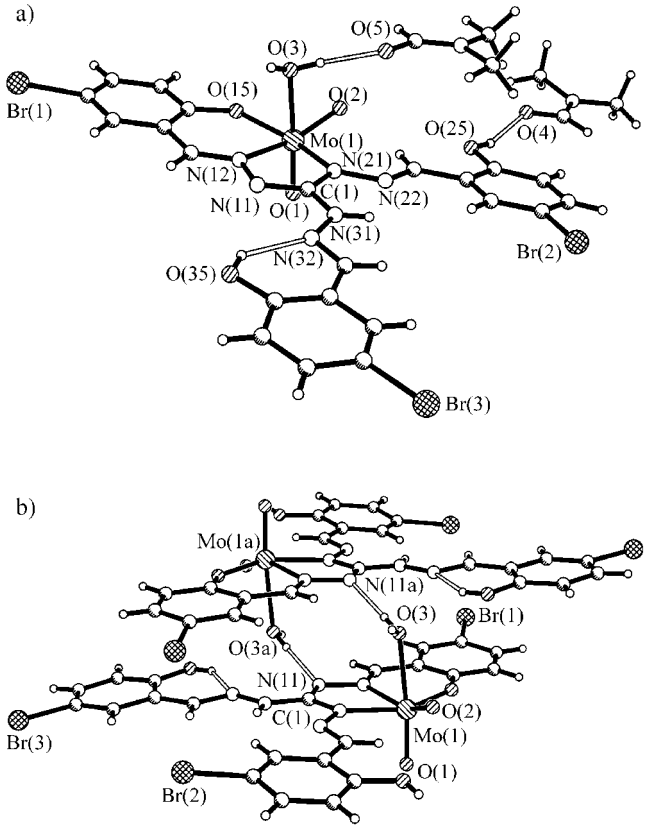


Figure 3. Crystal structure of **2**; a) asymmetric unit; b) hydrogen-bonded dimer

In **2** the ligand is partially deprotonated, and N12, N21 and O15 participate in the Mo(VI) coordination sphere. Two of the three arms of **1** (starting with N11 and N31) are transformed into conformation 2, the third arm (starting with N21) remains in conformation 1 and is stabilized by a strong hydrogen bond with a DMF molecule [*d*(H25···O4) = 1.87 Å, Table 1]. In this complex, $[H_3Br_3L]^{2-}$ coordinates to a Mo(VI) centre with a distorted octahedral geometry. Three of the four equatorial positions are occupied by the ligand, the fourth by a doubly bonded oxygen atom (O2). The two axial coordination sites contain a second oxygen atom (O1) and a water molecule (O3). Due to the relatively rigid geometry of the ligand, the distortion

Table 2. Selected bond lengths and angles (ϕ = average value, Σ = angle sum, ^{c} = coordinating atom, ^{nc} = noncoordinating atom)

	1a (213 K)	**1b** (213 K)	**2** (100 K)	**3** (100 K)
C−N [Å]	1.327(7)	1.336(2)	N11: 1.323(8) 1.360(8)	1.350(7)
N−N [Å]	1.380(5)	1.389(3)	1.395(7)^{c} 1.362(8)^{nc}	1.390(6)
N=C [Å]	1.279(5)	1.278(3)	1.293(8)^{c} 1.287(8)^{nc}	1.294(6)
C−Br [Å]	1.896(6)	1.906(3)	1.905(7)	1.904(6)
Angle at C1	ϕ 120.4(4)° Σ 360.0°	ϕ 120.0(1)° Σ 360.0°	123.6(6)° ^{c} 118.2(6)° ^{nc} Σ 360.0°	ϕ 120.0(5)° Σ 360.0°
Angle at N1	ϕ 117.6(4)°	ϕ 117.4(2)°	113(2)° − 130.0(5)° Σ 359.7°	110.9(8)° − 135(1)° Σ 356.8°
Angle at N2	ϕ 115.3(4)°	ϕ 115.1(2)°	116(3)° − 128.2(4)° Σ 360.0°	116.0(9)° − 126.2(4)° Σ 359.7°
Angle at O	−	−	138.3(4)°	127.9(7)°
Average deviation from CN_6 plane	± 0.03(2) Å	± 0.19(2) Å	± 0.014(5) Å	± 0.04(1) Å
Torsion angle CN_6 vs. phenyl	1.5° − 22.3°	18.8°	3.5° − 7.1°	9.9° − 31.3°

at the metal centre can clearly be seen at the angles along the N21−Mo1−O15 [152.1(2)°] and N12−Mo1−O2 axes [153.8(2)°, Table 3]. The Mo atom is displaced at a distance of 0.32(1) Å from the O1/N21/O3/O15 equatorial plane towards the doubly bonded oxygen atom (O1). The $[H_3Br_3L]^{2-}$ ligand itself shows no further distortion due to the coordination. The observed bond lengths (Table 2) are within the tolerance of 3σ, similar to those in the isolated $[H_6Br_3L]^{+}$ cation. The propeller-like distortion of the ligand (dihedral angles of 3.5° to 7.1°) in **2** is lower or similar to that in **1a** and **1b**. Due to the chelation, the angles observed in the central part of the ligand in **2** are markedly different from those in **1**.

2 is electronically neutral; the ligand nitrogen N11 is deprotonated and stabilized by a strong hydrogen bond from the coordinated water molecule of a neighbouring complex [*d*(N11···H3b) = 1.89 Å, angle at H3b: 175°). This and a symmetry-related bridge results in the formation of dimers as shown in Figure 3 (b).

The reaction of $[H_6Br_3L]Cl$ with $CuCl_2$, sodium 5,5-diethylbarbiturate (NaH*bar*) and Et_3N at room temperature leads to the formation of dark red-black crystals with the formula $(Et_4N)_2[\{Cu(Hbar)\}_3Br_3L]$ (**3**).[11] The X-ray structure shows that the ligand is now fully deprotonated and coordinated to three Cu(II) centres (Figure 4).

The metal atoms exhibit square-planar coordination spheres, in which three positions are occupied by the ligand, the fourth by a partly deprotonated diethylbarbiturate anion ($Hbar^{-}$). The copper centres show only minor distortions from the idealised coordination geometry, with observed *trans* angles of 168(2)° and 171(5)° (Table 3). The metals lie almost in the ideal plane formed by the ligand, with a deviation of only 0.03(1) Å. Furthermore, the distortion of the ligand is limited despite the fact that it is involved in the coordination with three metal atoms. The bond lengths observed in **3** are similar to those in **1a** and **1b**. Only the first nitrogen atom in each arm is twisted slightly out of plane, which can clearly be seen in the sum of the observed angles (Table 2). The propeller like distor-

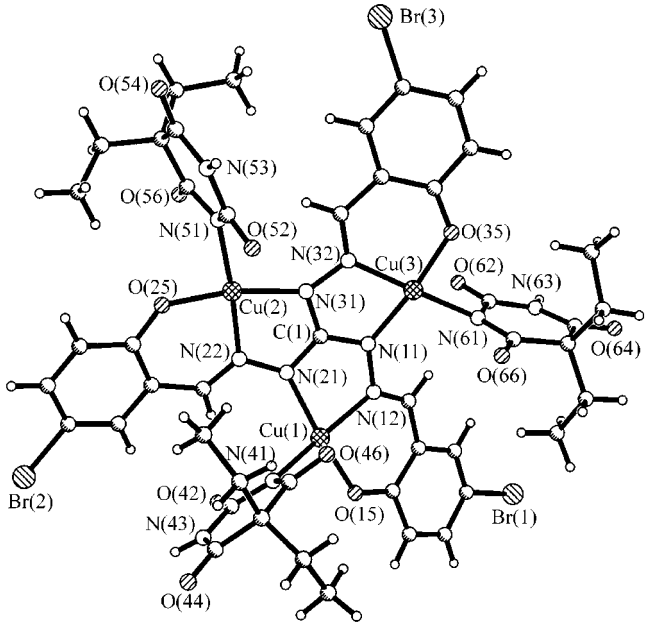


Figure 4. Crystal structure of **3** (countercations omitted for clarity)

Table 3. Bond lengths and angles at the metal centres in **2** and **3**

	2 (100 K)	**3** (100 K)
M−N(1)	2.115(5) Å	1.98(1) Å
M−N(2)	2.235(5) Å	1.959(6) Å
M−O	1.933(4) Å	1.906(5) Å
M−L′(eq)	1.717(4) Å	−
M−L′′(ax)	1.701(5) Å (=O) 2.325(5) Å ($-OH_2$)	−
N(1)−M−N(2)	71.2(2)°	79.4(4)°
N(2)−M−O	82.1(2)°	92.3(4)°
N(1)−M−O	152.1(2)°	168(2)°
N(2)−M−L′(eq)	153.8(2)°	171(5)°
L′′(ax)−M−L′′(ax)	173.1(2)°	−

tion as gauged by its dihedral angle of 31.3° is significantly more pronounced for one of the phenyl groups than in previously reported compounds. The other two angles are 9.9° and 13.2°. This once again emphasises the high degree of flexibility of the $[Br_3L]^{5-}$ ligand. The H*bar*$^-$ ligands in **3** are still protonated at the second nitrogen atom. Two of the three *bar* ligands form hydrogen bonds with the carbonyl oxygen atom of an adjacent H*bar*$^-$ ligand (Figure 5, a), the third shows a close contact to a neighbouring acetonitrile molecule (Table 1). This results in the formation of the infinite chains depicted in Figure 5, b.

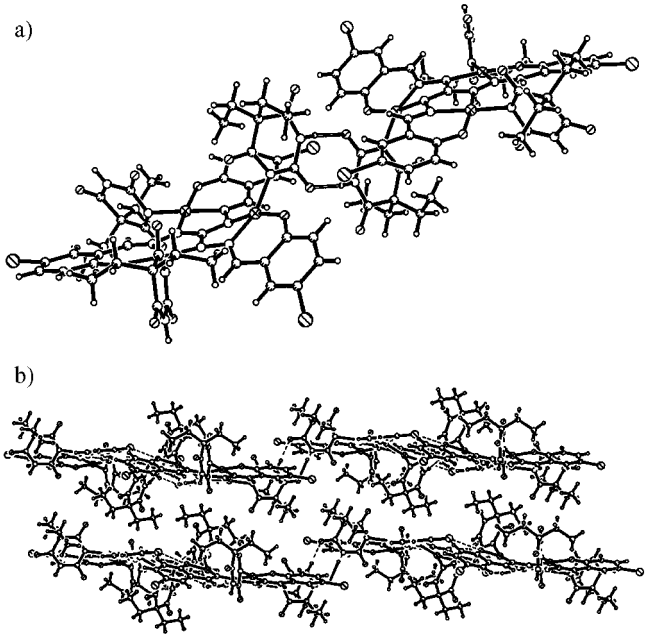


Figure 5. Packing diagram of **3** (countercations omitted for clarity); a) hydrogen-bonded dimer; b) stacking of two chains

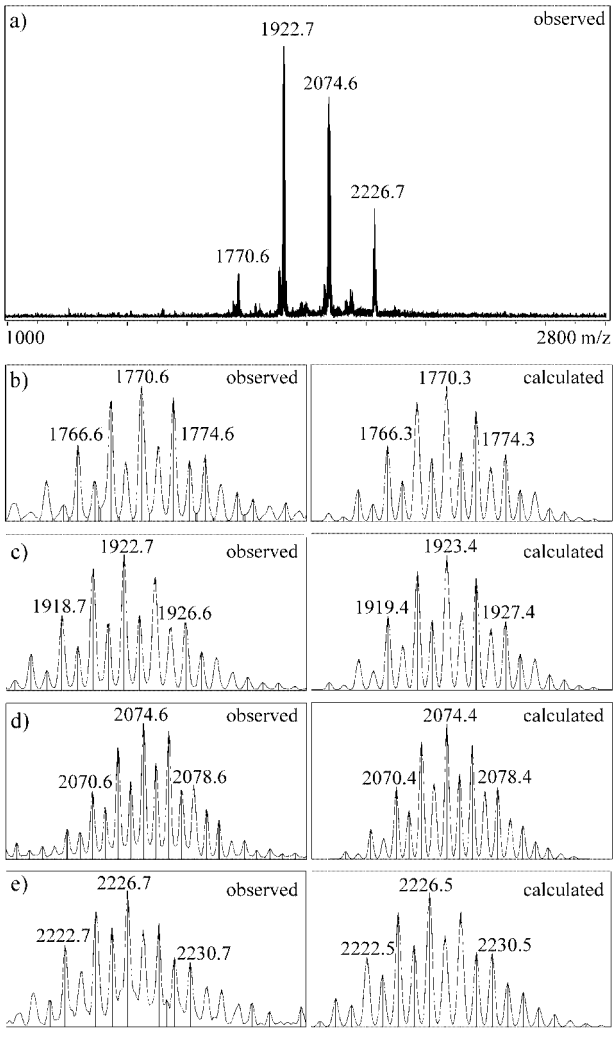


Figure 6. a) ESI mass spectrum for **3** in MeOH; b)−e) observed and calculated isotope pattern for the peaks observed in a).

The structure of **3** demonstrates that it is possible to bind three ligands, with a steric demand as high as that of H*bar*$^-$, to a M_3Br_3L unit. This is an important requirement when employing $[M_3Br_3L]^{3n-5}$ units for the construction of coordination polymers and cages. In fact, in the ESI mass spectrum of **3** in methanol, the only observed molecular ions can be explained by the formation of a dimeric species (Figure 6, a). Two $[Cu_3Br_3L]^+$ units are bridged by three monoanionic coligands: three OCH_3^-, $[C_{47}H_{33}N_{12}O_9Cu_6Br_6]^-$ (Figure 6, b); two CH_3O^-, one H*bar*$^-$, $[C_{54}H_{42}N_{14}O_{11}Cu_6Br_6]^-$ (Figure 6, c); one CH_3O^-, two H*bar*$^-$, $[C_{61}H_{49}N_{16}O_{13}Cu_6Br_6]^-$ (Figure 6, d); three H*bar*$^-$, $[C_{68}H_{57}N_{18}O_{15}Cu_6Br_6]^-$ (Figure 6, e).

In summary, we are able to show that $[H_6Br_3L]^+$ should be a suitable C_3-symmetric ligand for the formation of coordination cages and polymers. The free coordination sites at the metal centres bound by $[Br_3L]^{5-}$ can be occupied by a coligand with high steric demands, without the distortion of the $[M_3Br_3L]^{3n-5}$ unit or the C_3 symmetry. In the solid state, $[H_6Br_3L]^+$ adopts a conformation that is disadvantageous for the coordination of metal centres. Deprotonation by adding a base enables the transformation into the more favourable conformation. In this way, the reaction with metal ions can easily be controlled.

Experimental Section

Triaminoguanidinium chloride was prepared by literature methods.[12] 5-Bromosalicylaldehyde and sodium 5,5-diethylbarbiturate were purchased and used without further purification.

$[C_{22}H_{18}N_6O_3Br_3]Cl$ (1): Triaminoguanidinium chloride (4.681 g, 33.3 mmol) was dissolved in a hot mixture of H_2O (50 mL) and ethanol (100 mL). A solution of 5-bromosalicylaldehyde (20.0392 g, 99.7 mmol) in ethanol (140 mL) was slowly added. The resulting suspension was cooled to room temperature. **1** was crystallised from acetone (400 mL). Yield: 22.2691 g (32.3 mmol, 97%). $C_{22}H_{18}Br_3ClN_6O_3$ (689.58), calcd. values are given in parentheses, based on M + 1 H_2O: C 37.42 (37.34), H 2.57 (2.85), N 11.77 (11.88). ^{1}H NMR (400 MHz, [D$_6$]DMSO, 25 °C): δ = 12.12 (s, 1 H, NH), 10.83 (s, 1 H, OH), 9.01 (s, 1 H, N=CH−), 8.37 [s(d), ^4J = 2.52 Hz, 1 H, C(6)−H], 7.48 [d(d), ^3J = 9.04, ^4J = 2.52 Hz, 1 H, C(4)−H], 7.01 [d, ^3J = 9.04 Hz, 1 H, C(3)−H] ppm. ^{13}C

NMR (100 MHz, [D$_6$]DMSO, 25°C): δ = 156.38 (C2), 148.79 (C$^+$), 145.92 (C=N), 135.70 (C4), 128.70 (C6), 121.55 (C1), 118.50 (C3), 110.95 (C5) ppm. Crystals suitable for X-ray crystallography were grown by slow diffusion of HCl gas into a solution of **1** in water with Et_3N (pH 8−9, resulting in **1a**) or into a solution of **1** in DMF (resulting in **1b**).

[Mo(O)$_2$(OH$_2$)(C$_{22}$H$_{15}$N$_6$O$_3$Br$_3$)]·2(C$_3$H$_7$NO) (2): Solutions of tris(5-bromo-2-hydroxybenzylidene)triamminoguanidinium chloride (69.2 mg, 0.1 mmol) and ammonium heptamolybdate tetrahydrate (52.0 mg, 0.042 mmol) in DMF (1 mL each) were mixed. Water (1 mL) was added. Red crystals of **2** were formed after a couple of days. Yield: 28.0 mg (0.0297 mmol, 30%). $C_{28}H_{31}Br_3MoN_8O_8$ (943.3), calcd. values are given in parentheses, based on M + 1 H_2O: C 35.27 (34.99), H 3.68 (3.46), N 11.53 (11.66). ^{1}H NMR (400 MHz, [D$_6$]acetone, 25°C): δ = 11.67 (s, 1 H, N−H), 10.40 (s, 1 H, O−H), 9.69 (s, 1 H, O−H), 8.72 (s, 1 H, N=C−H), 8.54 (s, 1 H, N=C−H), 8.41 (s, 1 H, N=C−H), 7.84 [s(d), ^4J = 2.48 Hz, 1 H, C(6)−H], 7.52 [d(d), ^3J = 8.52, ^4J = 2.52 Hz, 1 H, C(4)−H], 7.51 [s(d), ^4J = 2.52 Hz, 1 H, C(6)−H], 7.36 [d(d), ^3J = 8.52, ^4J = 2.48 Hz, 1 H, C(4)−H], 7.35 [s(d), ^4J = 2.48 Hz, 1 H, C(6)−H], 7.34 [d(d), ^3J = 9.04, ^4J = 2.48 Hz, 1 H, C(4)−H], 6.90 [d, ^3J = 8.52 Hz, 1 H, C(3)−H], 6.88 [d, ^3J = 9.04 Hz, 1 H, C(3)−H], 6.83 [d, ^3J = 8.52 Hz, 1 H, C(3)−H] ppm.

[(C$_2$H$_5$)$_4$N]$_2$[{Cu(C$_8$H$_{11}$N$_2$O$_3$)}$_3$C$_{22}$H$_{12}$N$_6$O$_3$Br$_3$] (3): Tris(5-bromo-2-hydroxybenzylidene)triaminoguanidinium chloride (34.6 mg, 0.05 mmol), copper(II) chloride (20.24 mg, 0.15 mmol), sodium 5,5-diethylbarbiturate (20.9 mg, 0.10 mmol) and tetraethylammonium chloride (14.5 mg, 0.088 mmol) were dissolved in acetonitrile (2 mL) and triethylamine (1 mL). More triethylamine (1 mL) was slowly diffused into the reaction mixture. After three weeks dark red−black crystals of **2** were formed. Yield: 36.0 mg (0.022 mmol, 65.5%). $C_{62}H_{85}Br_3Cu_3N_{14}O_{12}$ (1648.78), calcd. values are given in parentheses, based on M + 2 CH_3CN + 2 H_2O: C 44.75 (44.86), H 5.10 (5.42), N 12.66 (12.68), Cu 11.2 (10.8).

X-ray Analysis: Intensity data for **1a** and **1b** were collected with an AXS Smart/CCD diffractometer (Mo-K_α radiation) and for **2** and **3** with a Nonius Kappa CCD (Mo-K_α rotating anode) always employing the ω scan method. All data were corrected for Lorentz and polarisation effects. Absorption corrections were performed for **1a** and **1b** by SADABS and for **2** and **3** by the Gauss method. **1a**−**3** were solved by using direct methods (SHELXS-97)[13] and refined by using a full-matrix least-squares refinement procedure (SHELXL-97).[14] The protons were placed at geometrically estimated positions. CCDC-241614 to -241617 contain the supplementary crystallographic data for this paper. These data can be obtained free of charge at www.ccdc.cam.ac.uk/conts/retrieving.html [or from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; Fax: (internat.) +44-1223-336-033; E-mail: deposit@ccdc.cam.ac.uk].

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[7] **1a**: 0.12 × 0.20 × 0.22 mm^3, triclinic, $P\bar{1}$, a = 7.488(2) Å, b = 12.889(3) Å, c = 14.489(3) Å, α = 109.735(5)°, β = 102.471(5)°, γ = 96.972(5)°, V = 1256.1(4) Å^3, $\rho_{calcd.}$ = 1.871 g·cm^{-3}, $2\theta_{max.}$ = 50.16°, λ = 0.71073 Å, T = 213 K, 7057 measured reflections, 4334 independent reflections (R_{int} = 0.0308), 3133 observed reflections ($I > 2\sigma(I)$), μ = 4.966 mm^{-1}, empirical absorption correction, $T_{min.}$ = 0.383, $T_{max.}$ = 0.551, 325 parameters, $R_1(I > 2\sigma(I))$ = 0.0396, wR_2 (all data) = 0.0986, max./min. residual electron density 0.652/−0.737 e·Å^{-3}.

[8] **1b**: 0.27 × 0.51 × 0.51 mm^3, trigonal, $R\bar{3}$, a = 15.576(2) Å, b = 15.576(2) Å, c = 28.452(5) Å, V = 5978(1) Å^3, $\rho_{calcd.}$ = 1.515 g·cm^{-3}, $2\theta_{max.}$ = 58.14°, λ = 0.71073 Å, T = 213 K, 4419 measured reflections, 3107 independent reflections (R_{int} = 0.0772), 2290 observed reflections ($I > 2\sigma(I)$), μ = 3.155 mm^{-1}, empirical absorption correction, $T_{min.}$ = 0.221, $T_{max.}$ = 0.429, 156 parameters, $R_1(I > 2\sigma(I))$ = 0.0486, wR_2(all data) = 0.1440, max./min. residual electron density 0.563/−0.907 e·Å^{-3}.

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[10] **2**: 0.015 × 0.035 × 0.13 mm^3, triclinic, $P\bar{1}$, a = 9.7149(5) Å, b = 13.1846(6) Å, c = 15.0352(7) Å, α = 71.394(2)°, β = 82.217(2)°, γ = 71.008(2)°, V = 1724.7(1) Å^3, $\rho_{calcd.}$ = 1.816 g·cm^{-3}, $2\theta_{max.}$ = 56.82°, λ = 0.71073 Å, T = 100 K, 14140 measured reflections, 8219 independent reflections (R_{int} = 0.0659), 4923 observed reflections ($I > 2\sigma(I)$), μ = 3.915

mm^{-1}, numerical absorption correction, $T_{min.} = 0.630$, $T_{max.} = 0.944$, 444 parameters, $R_1(I > 2\sigma(I)) = 0.0695$, wR_2(all data) = 0.1377, max./min. residual electron density 1.141/−0.728 e·Å^{-3}.

[11] **3**: 0.03 × 0.04 × 0.20 mm^3, triclinic, $P\bar{1}$, $a = 9.683(2)$ Å, $b = 17.576(4)$ Å, $c = 23.238(5)$ Å, $\alpha = 82.83(3)°$, $\beta = 81.41(3)°$, $\gamma = 81.66(3)°$, $V = 3847(1)$ Å^3, $\rho_{calcd.} = 1.530$ g·cm^{-3}, $2\theta_{max.} = 50.50°$, $\lambda = 0.71073$ Å, $T = 100$ K, 44104 measured reflections, 13909 independent reflections ($R_{int} = 0.0978$), 8964 observed reflections ($I > 2\sigma(I)$), $\mu = 2.449$ mm^{-1}, numerical absorption correction, $T_{min.} = 0.748$, $T_{max.} = 0.937$, 1022 parameters, $R_1(I > 2\sigma(I)) = 0.0613$, wR_2(all data) = 0.1166, max./min. residual electron density 0.679/−0.752 eÅ^{-3}.

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