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A comparative study of supramolecular assemblies containing N''-(5,6-dimethyl-1H-benzimidazol-2-yl)guanidine, 2-guanidinobenzimidazole and their Ni(II) complexes

Michael M. Bishop [a,b], Andrew H.W. Lee [b], Leonard F. Lindoy [a,*], Peter Turner [a]

[a] *Centre for Heavy Metals Research, School of Chemistry, University of Sydney, Sydney NSW 2006, Australia*
[b] *Sydney Grammar School, College Street, Darlinghurst NSW 2010, Australia*

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Abstract

Previous studies have shown that 2-guanidinobenzimidazole and the neutral complex [bis(2-guanidinobenzimidazolo)nickel(II)] are capable of forming extended hydrogen bonded arrays in the solid state, either alone or with other transition metal complexes as well as with organic molecules with complementary hydrogen bonding motifs. Additionally, the complex of the free ligand with phthalimide contains a NH–N hydrogen bond so short that it compares with the short hydrogen bonds in some of the 'proton sponges'. In this paper we report on the molecular structures and simple supramolecular assemblies containing the related ligand N''-(5,6-dimethyl-1H-benzimidazol-2-yl)guanidine which has a higher pK_a than the parent (unsubstituted) ligand and which has steric requirements which mean that it cannot be incorporated into the same supramolecular structures. In particular, we examine the structure of the free ligand, of the complex formed by the ligand and phthalimide and of the extended structure of the neutral bis-ligand nickel(II) complex and dmso. The structures are compared with those mentioned above incorporating the unsubstituted ligand, 2-guanidinobenzimidazole.

Keywords: Supramolecular; X-ray structure; Nickel(II); Guanidinobenzimidazole derivatives

1. Introduction

In a series of papers [1–5], we have investigated the assembly of metal complexes incorporating potential hydrogen bonding triplets into crystalline assemblies alone, with organic molecules with complementary hydrogen bonding motifs, or with other transition metal complexes.

One of the ligands of interest was 2-guanidinobenzimidazole, gbH, which itself possesses a donor–acceptor–donor (DAD) hydrogen bonding motif that can also be present when the anionic form of the ligand forms complexes. The neutral Ni(II) bis-ligand complexes themselves have been observed to display different protomeric forms [2,6] (Scheme 1). In addition, the complexes cannot be planar because of the steric requirements of the ligands and were observed in a step-like geometry with the nickel and the four donor nitrogens adopting a square planar arrangement (Scheme 2a) or a coordination geometry in which the four donor atoms are distorted from square planar towards a tetrahedral geometry, (Scheme 2b) [2,5,6]. The electronic nature of the ligands is also of interest because of the capacity it allows for 'tuning' the system. The replacement of a nitrogen in the benzimidazole residue to form a benzoxazole or benzothiazole, is relatively straightforward [7,8] as is the synthesis of derivatives with substituents on the benzene ring [9]. Both of these changes are expected to alter the ligand's pK_a and hence may affect the hydrogen bonding present [10]. Of course, the presence of substituents on the molecule

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* Corresponding author. Tel.: +61-2-9351-4400; fax: +61-2-9351-7067.

E-mail address: lindoy@chem.usyd.edu.au (L.F. Lindoy).

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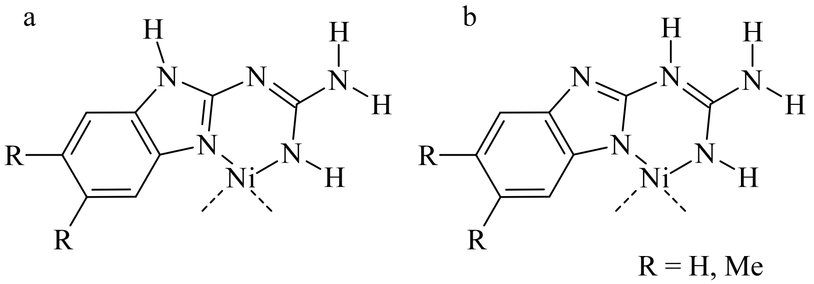


Scheme 1.

introduces new steric requirements which may outweigh (or complement) any induced electronic effects.

These relatively simple guanidinobenzimidazole ligands have the potential to introduce flexibility at the junction between the guanidine and benzimidazole residues, to display different protomeric forms, and to exhibit different coordination geometries. It was anticipated that a comparison of the supramolecular assemblies containing the dimethyl-substituted derivative with those of unsubstituted guanidinobenzimidazole might provide an aid to a better understanding of rational crystal design involving such systems.

In this paper we report the structure of *N″*-(5,6-dimethyl-1*H*-benzimidazol-2-yl)guanidine, Me_2gbH, crystallised from methanol, of its supramolecular assembly with phthalimide, and of the structure of the $[Ni(Me_2gb)_2]$ complex crystallised from dmso. The structures of all three are also compared with the corresponding structures incorporating unsubstituted gbH.

2. Experimental

2.1. Synthesis

N″-(5,6-dimethyl-1*H*-benzimidazol-2-yl)guanidine was prepared from 1,2-diamino-4,5-dimethylbenzene and dicyandiamide by the literature method [9] (Scheme 3), and recrystallisation of the crude product from water gave the monohydrate **1a**. Crystals (**1**) for X-ray analysis were grown from methanol solution; the crystals lost solvent once removed from the growth solution.

The 1:1 complex of Me_2gbH and phthalimide was prepared by dissolving Me_2gbH (0.20 g, 1 mmol) in absolute ethanol (10 ml) and adding to this a solution of phthalimide (0.15 g, 1 mmol) in the same solvent (10 ml). The orange crystals (**2**), which formed, were filtered at the pump and air dried. Yield 0.20 g (58%).

The nickel(II) complex was prepared by dissolving Me_2gbH (1.62 g, 7.8 mmol) in methanol (30 ml) and adding to this an aqueous ammoniacal solution of nickel(II) chloride hexahydrate (0.95 g, 4.0 mmol). The mixture was stirred for 10 min. The dark red product that precipitated was filtered off, washed (methanol 2 × 5 ml; diethyl ether 2 × 5 ml) and air-dried. Yield 1.53 g (41%). The material for microanalysis (**3a**) was dried at room temperature over silica gel.

Crystals for X-ray analysis (**3**) were prepared by crystallisation of this product from dmso. The crystals were left in the growth solution until the structure was determined. The material for microanalysis (**3b**) was removed from the growth solution and dried at room temperature over silica gel.

2.2. Elemental analysis

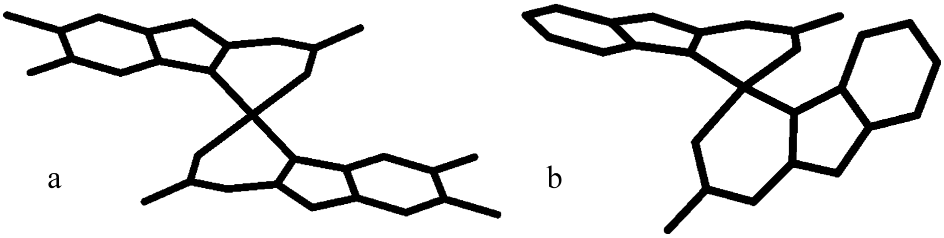
C, H and N analysis was carried out by the Microanalytical Laboratory in the Research School of Chemistry of the Australian National University.

2.2.1. $Me_2gbH\cdot H_2O$ (1a)

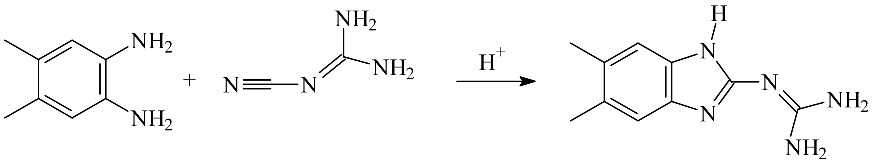
Found: C, 54.09; H, 7.46; N, 31.59%. Calc. for $C_{10}H_{13}N_5\cdot H_2O$ (**1**): C, 54.30; H, 6.79; N, 31.67%.

2.2.2. [Me_2gbH:phthalimide] (2)

Found: C, 61.46; H, 5.42; N, 23.91%. Calc. for $C_{18}H_{18}N_6O_2$ (**2**): C, 61.70; H, 5.18; N, 23.99%.



Scheme 2.



Scheme 3.

2.2.3. [Ni(Me$_2$gb)$_2$]·1.5H$_2$O (**3a**)

Found: C, 49.27; H, 5.30; N, 28.21%. Calc. for $C_{20}H_{24}N_{10}Ni\cdot1.5H_2O$ (**3a**): C, 49.01; H, 5.51; N, 28.59%.

2.2.4. [Ni(Me$_2$gb)$_2$].2C$_2$H$_6$SO·0.5H$_2$O (**3b**)

Found: C, 45.80; H, 5.96; N, 22.01%. Calc. for $C_{20}H_{24}N_{10}Ni$. $2C_2H_6SO\cdot0.5H_2O$ (**3b**): C, 45.87; H, 5.89; N, 22.30%.

2.3. Single crystal X-ray diffraction

Diffraction data were collected using a Bruker SMART 1000 CCD diffractometer employing graphite monochromated Mo Kα radiation generated from a sealed tube. The crystals for **1** and **2** were supported, using Exxon Paratone N, on a short length of fibre glued to a thin piece of copper wire inserted in a copper mounting pin, and cooled to 150(2) K with an Oxford Cryosystems Cryostream. The data for **3** were collected at 293(2) K and the crystal was glued to a thin glass fibre. The data integration and reduction were undertaken with SAINT and XPREP [11], and subsequent computations were carried out with the teXsan [12], WinGX [13] and XTAL [14] graphical user interfaces. A Gaussian absorption correction [11,15] was applied to the data.

The structures were solved by direct methods with SIR-97 [16] and extended and refined with SHELXL-97 [17]. The non-hydrogen atoms were modelled with anisotropic displacement parameters, and in general a riding atom model was used for the hydrogen atoms. Amine and methoxy hydrogen atoms were located and modelled with isotropic displacement parameters. ORTEP [18] depictions of the molecules with 20% displacement ellipsoids are provided in Figs. 1–3. Crystallographic details are summarised in Table 1, hydrogen bond geometry summarised in Table 2, and selected molecular geometry details are listed in Table 3.

3. Results and discussion

3.1. Me$_2$gbH·2MeOH

The Me$_2$gbH crystals contain two methanol solvate molecules for each Me$_2$gbH molecule, and these solvate molecules are readily lost upon removal of the crystals from the growth solution. The Me$_2$gbH molecules form dimers through the formation of $R_2^2(8)$ hydrogen bonded rings [19] centred at (0, 0, 0), involving N(2) and N(3). Dimers related by a unit cell translation along the c axis are linked by hydrogen bond interactions with the methanol solvate molecules (Fig. 4). The methanol molecules are in different environments; one forms three hydrogen bonds and the other forms two, with the acceptors being N(5) and N(3), respectively (Table 2). The hydrogen bonding appears to involve the ‘nitrogen’

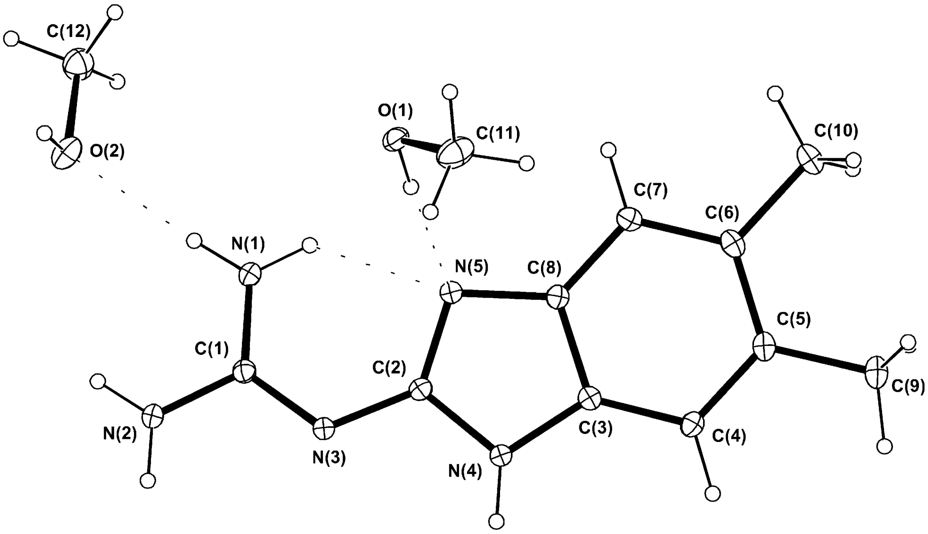


Fig. 1. An ORTEP [18] depiction of Me$_2$gbH·2CH$_3$OH, with displacement ellipsoids at the 20% level and showing the numbering scheme.

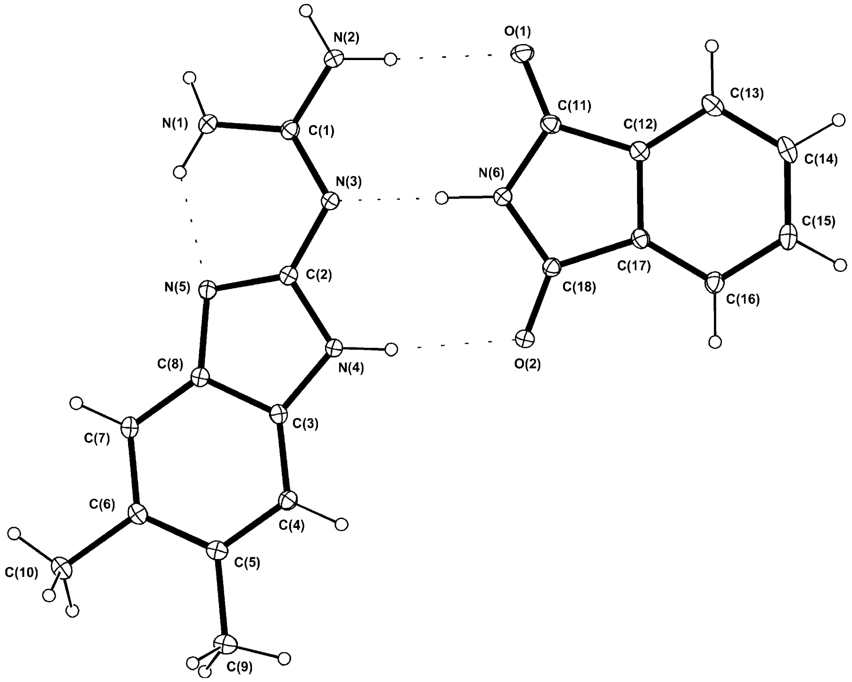


Fig. 2. An ORTEP [18] depiction of the [Me_2gbH:phthalimide] complex, with displacement ellipsoids at the 20% level and showing the numbering scheme.

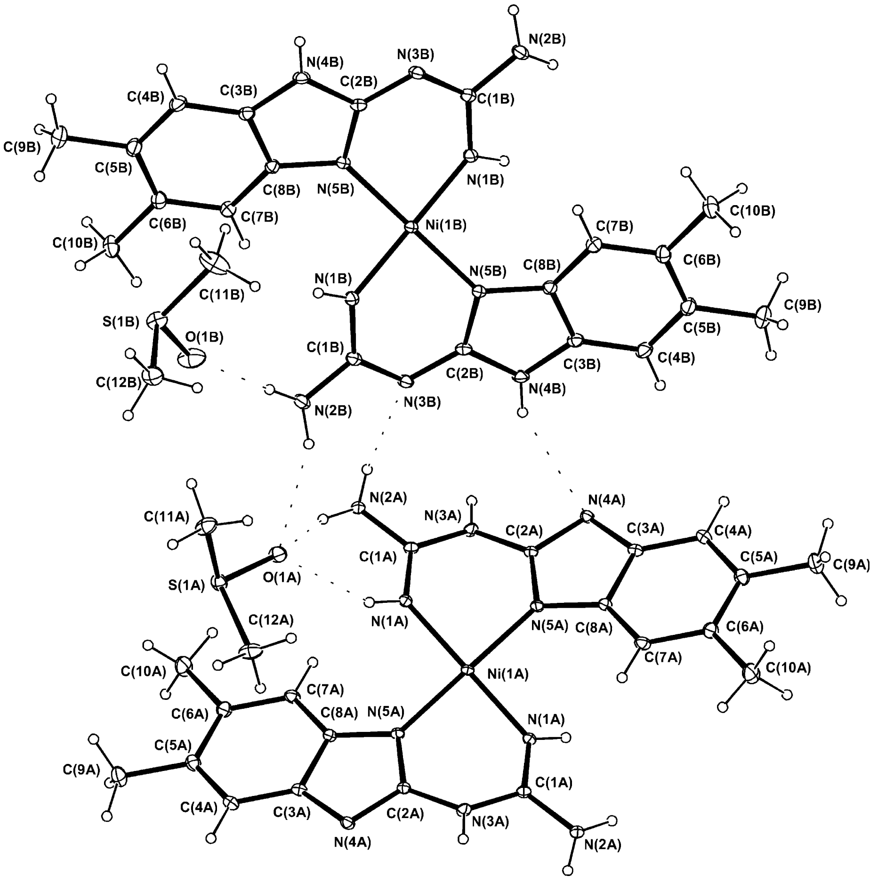


Fig. 3. An ORTEP [18] depiction, of the complex $[Ni(Me_2gb)_2]\cdot 2dmso$, with displacement ellipsoids at the 20% level and showing the numbering scheme.

Table 1
Summary of X-ray crystallographic determinations

	1	**2**	**3**
Formula of the refinement model	$C_{12}H_{21}N_5O_2$	$C_{18}H_{18}N_6O_2$	$C_{24}H_{36}N_{10}NiO_2S_2$
Model molecular weight	267.34	350.38	619.46
Crystal system	triclinic	triclinic	triclinic
Space group	$P\bar{1}$ (#2)	$P\bar{1}$ (#2)	$P\bar{1}$ (#2)
a (Å)	8.543(5)	7.827(5)	12.981(5)
b (Å)	12.572(5)	15.738(5)	13.358(5)
c (Å)	7.228(5)	7.430(5)	9.615(5)
α (°)	85.181(5)	91.541(5)	101.777(5)
β (°)	106.284(5)	113.658(5)	83.795(5)
γ (°)	107.246(5)	101.816(5)	117.593(5)
V (Å^3)	711.7(7)	814.5(8)	1446.3(11)
D_{calc} (g cm^{-3})	1.248	1.429	1.422
Z	2	2	2
Crystal size (mm)	0.456 × 0.258 × 0.160	0.387 × 0.317 × 0.085	0.368 × 0.243 × 0.061
Crystal colour	colourless	orange	red
Crystal habit	blade	blade	plate
Temperature (K)	150(2)	150(2)	293(2)
λ (Mo Kα)	0.71069 Å	0.71069 Å	0.71069 Å
μ (Mo Kα)	0.088 mm^{-1}	0.098 mm^{-1}	0.856 mm^{-1}
T (Gaussian)$_{min,max}$	0.956, 0.988	0.955, 0.991	0.7497, 0.9579
$2\theta_{max}$ (°)	56.56	56.58	56.58
hkl range	−10 11, −16 16, −9 9	−10 9, −20 20, −9 9	−17 14, −17 17, −12 12
N	6966	8083	13 828
N_{ind}	3279 (R_{merge} 0.02390)	3748 (R_{merge} 0.0294)	6554 (R_{merge} 0.0219)
N_{obs} [$I > 2\sigma(I)$]	2889	3135	5615
N_{var}	205	262	395
Residuals: $R_1(F)$, $wR_2(F^2)$ [a]	0.0362, 0.0760	0.0356, 0.0859	0.0298, 0.0752
GoF (all)	1.140	1.296	1.251
Residual extrema (e$^-$ Å^{-3})	−0.210, 0.269	−0.210, 0.282	−0.325, 0.593

$w = 1/[\sigma^2(F_o^2)+(0.01P)^2+0.3P]$ for **1**; $w = 1/[\sigma^2(F_o^2)+(0.03P)^2+0.1P]$ for **2** and $w = 1/[\sigma^2(F_o^2)+(0.03P)^2+0.3P]$ for **3**, where $P = (F_o^2+2F_c^2)/3$.

[a] $R_1 = \Sigma||F_o|-|F_c||/\Sigma|F_o|$ for $F_o > 2\sigma(F_o)$; $wR_2 = (\Sigma w(F_o^2-F_c^2)^2/\Sigma(wF_c^2)^2)^{1/2}$ all reflections.

π electrons (Fig. 4). There is an intramolecular hydrogen bond between N(1) and N(5).

The atoms N(1), C(1), N(2) and N(3) are co-planar (mean deviation from plane 0.002 Å) but the plane is twisted with respect to the imidazole plane by 17.47(08)°; the torsion angle C(1)–N(3)–C(2)–N(5) is 19.03(16)°.

Crystals of 2-guanidinobenzimidazole obtained from ethanol are solvent-free, and again contain dimers by forming centrosymmetric R_2^2 (8) hydrogen bonded rings [20]. The hydrogen bond N–N distances in these rings are slightly shorter than in Me_2gbH; 2.948 Å compared with 3.010(2) Å. In addition, the $(gbH)_2$ dimers are linked by hydrogen bonding between the imidazole nitrogens of adjacent molecules forming a three-dimensional network. Interestingly, some potential hydrogen bond donors remain unused.

3.2. [Me_2gbH:phthalimide]

The complex formed between Me_2gbH and phthalimide has complementary DAD/ADA hydrogen bonding motifs paired to form dimers held together by a triplet of hydrogen bonds. The dimers are further linked into ribbons (Fig. 5) by the formation of hydrogen bonds between O(1) and H(2NB). In contrast to **1**, the guanidino is twisted so that it is almost in the same plane as the benzimidazole residue and the torsion angle C(1)–N(3)–C(2)–N(5) is now only −6.18(17)°, a change that presumably occurs in response to the formation of the hydrogen bond triplet. Triplet interactions are not necessarily coplanar; we have previously observed non-planar systems [3,4] associated with the formation of further hydrogen bonds between the component molecules. In the present case, the formation of the triplet of hydrogen bonds increases the planarity of the Me_2gbH molecule, and this is hence expected to affect its electronic nature.

Both the supramolecular structure of the current complex **2** and the details of the dimers differ markedly from the previously reported complex formed between gbH and phthalimide [4]. The most significant difference is in the nature of the hydrogen bonded triplet. In the gbH complex, the proton from the phthalimide has been transferred to N(3) (current numbering scheme) of gbH and the hydrogen bond N–N distance is particularly

Table 2
Hydrogen bond geometries

Donor	Hydrogen	Acceptor	D–H (Å)	H–A (Å)	D–A (Å)	DHA angle (°)
(a) Me$_2$gbH·2CH$_3$OH						
N(1)	H(1NA)	N(5)	0.878(16)	2.069(16)	2.7463(17)	133.3(13)
N(1)	H(1NB)	O(2)	0.875(16)	2.061(16)	2.9338(19)	174.9(14)
N(2)	H(2NA)	N(3 [a])	0.895(16)	2.122(17)	3.010(2)	171.5(14)
N(2)	H(2NB)	O(1 [b])	0.860(16)	2.208(15)	2.9183(16)	139.9(13)
N(4)	H(4N)	O(1 [c])	0.873(15)	1.997(15)	2.853(2)	166.6(13)
O(1)	H(1O)	N(5)	0.871(18)	1.904(18)	2.7749(19)	176.9(16)
O(2)	H(2O)	N(3 [b])	0.861(19)	2.151(19)	3.0026(18)	169.7(17)
(b) [Me$_2$gbH:phthalimide]						
N(2)	H(2NA)	O(1 [d])	0.911(16)	2.115(17)	3.0239(17)	175.3(13)
N(4)	H(4N)	O(2 [d])	0.920(16)	2.155(16)	3.0602(15)	167.8(13)
N(1)	H(1NA)	N(5)	0.896(17)	1.966(17)	2.6671(17)	134.0(14)
N(6)	H(6N)	N(3 [d])	0.978(17)	1.787(17)	2.7656(16)	179.7(16)
N(2)	H(2NB)	O(2 [e])	0.890(16)	2.081(16)	2.9519(19)	165.7(13)
(c) [Ni(Me$_2$gb)$_2$]·2dmso						
N(2)	H(2NB)	O(1)	0.850(19)	2.191(19)	2.952(2)	148.9(16)
N(1)	H(1N)	O(1)	0.795(19)	2.232(2)	2.961(2)	152.5(18)
N(2)	H(2NA)	N(8)	0.78(2)	2.43(2)	3.110(2)	145.3(19)
N(9)	H(9N)	N(4)	0.80(2)	2.54(2)	3.265(2)	151.1(2)
N(3)	H(9N)	N(4 [f])	0.80(2)	2.05(2)	2.850(2)	172(2)
N(7)	H(7NB)	O(2)	0.86(2)	1.99(2)	2.856(2)	175.9(19)
N(7)	H(7NA)	O(1)	0.86(2)	2.29(2)	3.086(2)	153.8(19)

[a] $-x, 2-y, 2-z$.
[b] $-x, 2-y, 1-z$.
[c] $x, y, 1+z$.
[d] $-x, -y, 1-z$.
[e] 1-x, $-y, 2-z$.
[f] $-x, -y, -z$.

short (2.694 Å). There is no such transfer in **2**, and this is surprising as the dimethyl derivative is more basic than the unsubstituted gbH [10]. In fact, the triplet resembles more that formed between gbH and 4-nitrophthalimide [4]. The supramolecular structure of the present complex is much simpler than that of the gbH analogue; presumably a reflection of the steric requirements of the two methyl groups in the former. In the gbH analogue, the hydrogen bonded dimers are stacked very close together. Adjacent layers are separated by approximately 3.3 Å and the nitrogen atoms that form the central hydrogen bond of the triplet are almost directly above the centre of the π electron system of a phthalimide molecule in an adjacent layer. Both phthalimide oxygens accept a second hydrogen bond leading to a three-dimensional hydrogen bonded network. The stacking is less close in the 4-nitrophthalimide complex, and the orientation of the stacked dimers is also quite different from that in each of the phthalimide complexes, apparently reflecting the steric requirements of the nitro substituent.

The ribbon formation observed in this complex resembles that found when bemegride (3-ethyl-3-methylglutarimide) forms a 2:1 hydrogen bonded complex with bis(dithiobiureto)nickel(II) [21]. Only one of the two imide oxygens accepts two hydrogen bonds and we have

Table 3
Selected bond lengths

	N(1)–C(1)	N(2)–C(1)	N(3)–C(1)	N(3)–C(2)	N(4)–C(2)	N(5)–C(2)
gbH[11]	1.350(4)	1.357(4)	1.321(4)	1.373(3)	1.371(3)	1.331(3)
1	1.3364(17)	1.3407(16)	1.3409(16)	1.3730(15)	1.3687(16)	1.3376(16)
2	1.3415(16)	1.3408(16)	1.3257(16)	1.3704(15)	1.3765(16)	1.3273(15)
3 Ni(1A)	1.299(2)	1.372(2)	1.358(2)	1.376(2)	1.341(2)	1.351(2)
3 Ni(1B)	1.323(2)	1.354(2)	1.333(2)	1.355(2)	1.371(2)	1.358(2)

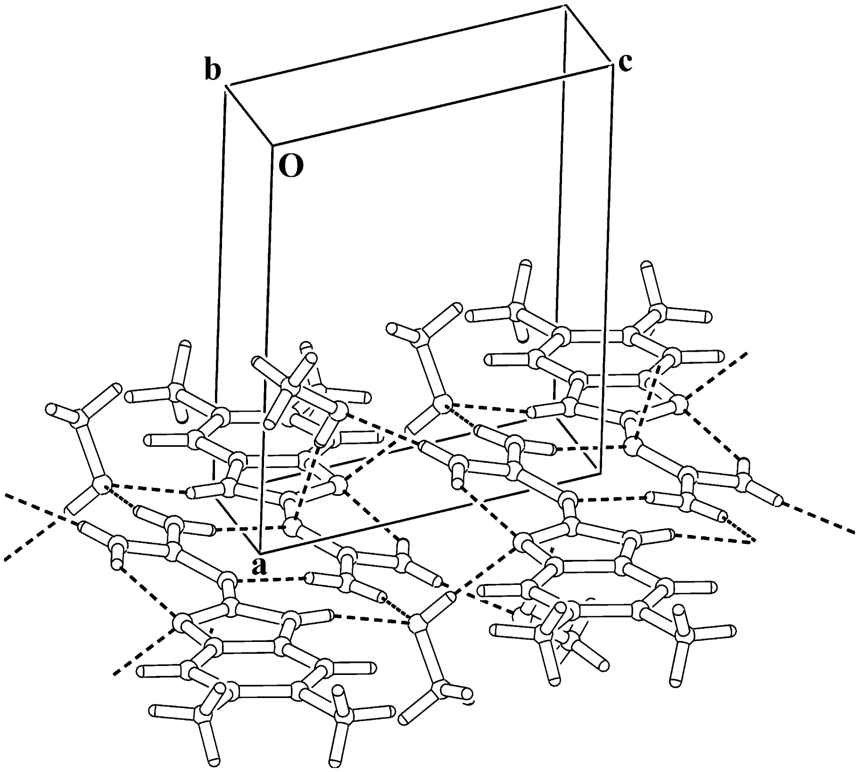


Fig. 4. A Platon [22] view of the supramolecular structure of $Me_2gbH \cdot 2CH_3OH$. Me_2gbH dimers are linked into chains by methanol molecules.

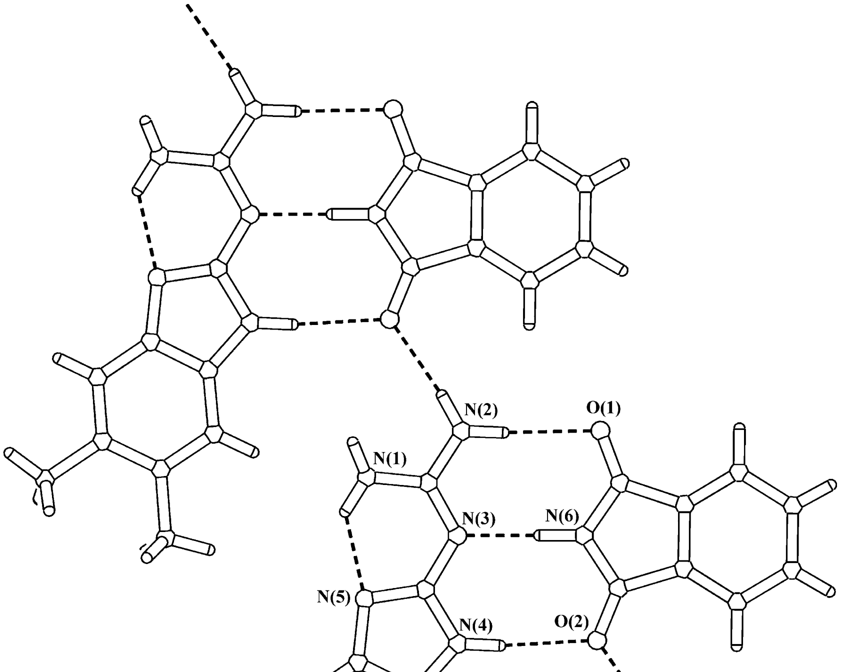


Fig. 5. A Pluton [22] depiction of the structure of [Me_2gbH:phthalimide] showing the hydrogen bonded chain structure.

shown [3] that the less symmetrical 3-iminoisoindolinone can also be accommodated into this supramolecular structure, orienting itself so that the carbonyl occupies the position in which the bridging hydrogen bond is formed and the imino group, which can accept only one hydrogen bond, occupies the other.

3.3. [Ni(Me$_2$gbH)$_2$]·2dmso

The asymmetric unit in the crystal structure of **3** contains two crystallographically independent complex molecules, each residing on an inversion site, and two dimethylsulfoxide solvate molecules.

The crystals of **3** are less dense than those of [Ni(gb)$_2$] and contain two different protomeric forms of the complex [Ni(Me$_2$gb)$_2$]. In one, containing Ni(1), the arrangement of protons on the potential hydrogen bonding triplet is DDA and in the other, Ni(2), DAD. In both forms of the complex, the NiN$_4$ units adopt a square planar geometry, with the ligand inclined with respect to the coordination plane to form a step-like shape about the inversion centre. The effects of the different protonation forms can also be seen in the lengths of the bonds that contain heteroatoms (Table 3).

The complexes containing Ni(1) form chains along the *c* axis, with complex components linked by centrosymmetric $R_2^2(8)$rings, which, as in the unmethylated complex above (but different from the free ligand in 1), involve N(3) and N(4). The $R_2^2(8)$rings are planar in **3**, not puckered. The Ni(2)-containing complexes form cross-links between chains separated by translations along the *b* axis, with $R_2^2(10)$rings involving N(8) and N(9) and N(2) and N(4); the hydrogen bonds are long and far from linear (Table 2). The effect of the cross-linking is to define channels along (1/2, 0, 1/2) that contain the solvate molecules (Fig. 6). The N(7)–N(2) and N(9)–N(3) distances, 3.439(3) and 3.476(2) Å respectively, are too long for the interactions to be considered hydrogen bonds. The terminal amine residues of the ligands of both complexes are involved in hydrogen bond interactions with nearby dmso solvate molecules.

When [Ni(gb)$_2$] is crystallised from a dmso solution, [2] the geometry about the nickel shows a distortion from square planar towards a tetrahedral arrangement (Scheme 2b). The neutral complexes are linked together into tapes by the formation of $R_2^2(8)$ rings, which, unlike the free ligand, [20] involve N(3) and N(4) (current numbering scheme) rather than N(2) and N(3). The rings are not planar, but have an approximate chair configuration, again differing from the planar rings in crystals of the free ligand. Four of these tapes are assembled into centro-symmetric convex-walled channels by interactions with dmso molecules which, by accepting hydrogen bonds from NH groups in neighbouring complexes, link the tapes together. Water molecules within the channels play a similar role. At two of the 'joins' between tapes, the aryl rings of the ligands approach quite closely (two carbons are separated by just 3.73 Å). In the current work there are methyl groups on these carbon atoms.

4. Summary

N″-(5,6-dimethyl-1*H*-benzimidazol-2-yl)guanidine has been synthesised, as has the neutral complex bis(*N*″-(5,6-dimethyl-1*H*-benzimidazol-2-yl)guanidino)nickel-

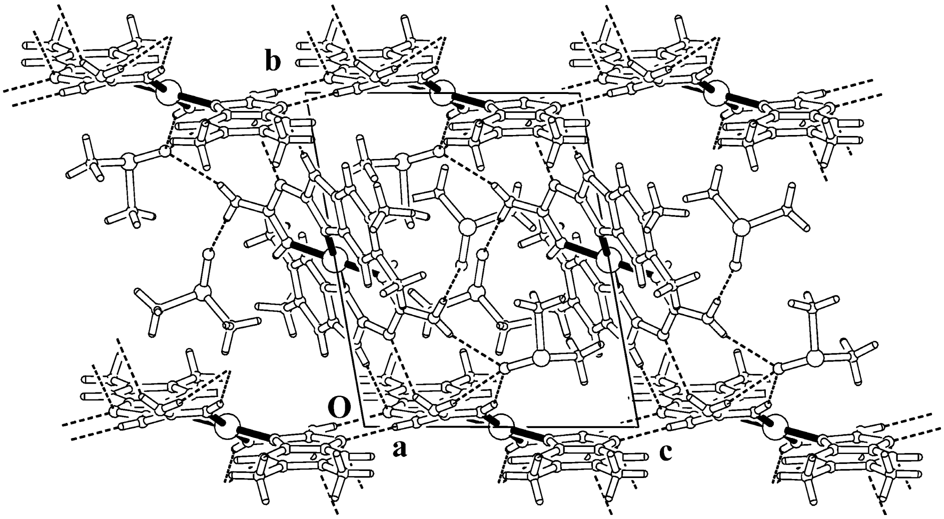


Fig. 6. A Pluton [22] depiction of the [Ni(Me$_2$gb)$_2$]·2dmso unit cell, viewed along the *a* axis, showing the hydrogen bonding interactions. Chains of one protomeric form of the complex run along the *c* axis and chains separated by a *b* translation are linked by hydrogen bond interactions with the second protomeric form of the complex.

(II), and both these have been incorporated into solid state supramolecular arrays. The structures of the arrays, determined by single crystal X-ray diffraction, have been compared with relevant arrays based on the related 2-guanidinobenzimidazole, which were reported previously. Although some of the hydrogen bonding patterns remain the same, there are differences that reflect the steric requirements of the methyl groups in the present compounds.

The proton transfer from phthalimide previously observed in [gbH:phthalimide] does not occur in **2**, [Me_2gbH:phthalimide], even though Me_2gbH is more basic than the unsubstituted gbH. Presumably local environment effects are responsible for this unexpected difference.

Interestingly, crystals of **3**, grown from dmso solution, contain two different protomeric forms of the neutral complex, [Ni(Me_2gb)$_2$]. For this system both the metal coordination geometries and the overall supramolecular structure adopted differ from those found in crystals of the related [Ni(gb)$_2$] array—clearly illustrating the influence of the methyl groups in dictating structure in systems of this type.

5. Supplementary data

Crystallographic data for the structures in this paper have been deposited at the Cambridge Crystallographic Data Centre: deposition numbers 192 769 (**1**), 192 770 (**2**) and 192 771 (**3**). Copies of this information can be obtained free of charge from The Director, CCDC 12 Union Road, Cambridge, CB2 1EZ, UK (fax: +44-1223-336033; e-mail: deposit@ccdc.cam.ac.uk or http://www.ccdc.cam.ac.uk.

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References

[1] M.M. Bishop, L.F. Lindoy, S. Mahadev, P. Turner, J. Chem. Soc., Dalton Trans. (2000) 233.
[2] M.M. Bishop, L.F. Lindoy, B. Skelton, A.H. White, Supramolecular Chem. 13 (2001) 293.
[3] M.M. Bishop, L.F. Lindoy, B. Skelton, A.H. White, J. Chem. Soc., Dalton Trans. (2002) 377.
[4] M.M. Bishop, L.F. Lindoy, R.O. Piltz, O.T. Thorn-Seshold, P. Turner, J. Heterocyclic Chem. 38 (2001) 1377.
[5] M.M. Bishop, L.F. Lindoy, P. Turner, Supramolecular Chem. 14 (2002) 179.
[6] S.E. Castillo-Blum, N. Barba-Behrens, Coord. Chem. Rev. 196 (2000) 3.
[7] G.B.L. Smith, J.H. Kane, C.W. Mason, J. Am. Chem. Soc. 51 (1929) 2522.
[8] G.B.L. Smith, C.W. Mason, R.H. Carroll, J. Am. Chem. Soc. 53 (1931) 4103.
[9] F.E. King, R.M. Acheson, P.C. Spenseley, J. Chem. Soc. (1948) 1366.
[10] C. Acerete, J. Catalán, F. Fabero, M. Sánchez-Cabezudo, R.M. Claramunt, J. Elquero, Heterocycles 26 (1987) 1581.
[11] S. Bruker, SMART, SAINT and XPREP. Area detector control and data integration and reduction software, Bruker Analytical X-ray Instruments Inc, Madison, WI, 1995.
[12] Molecular Structure Corporation (1997–1998). teXsan for Windows: Single Crystal Structure Analysis Software, MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.
[13] L.J. Farrugia, J. Appl. Cryst. 32 (1999) 837.
[14] S.R. Hall, D.J. du Boulay, R. Olthof-Hazekamp (Eds.), Xtal3.6 System University of Western Australia, 1999.
[15] P. Coppens, L. Leiserowitz, D. Rabinovich, Acta Crystallogr. 18 (1965) 1035.
[16] A. Altomare, M.C. Burla, M. Camalli, G.L. Cascarano, C. Giacovazzo, A. Guagliardi, A.G.G. Moliterni, G. Polidori, R.J. Spagna, Appl. Cryst. 32 (1999) 115.
[17] G.M. Sheldrick, SHELX-97 Programs for Crystal Structure Analysis. University of Göttingen. Institüt für Anorganische Chemie der Universität, Tammanstrasse 4, D-3400 Göttingen, Germany, 1998.
[18] C.K. Johnson, ORTEPII. Report ORNL-5138. Oak Ridge National Laboratory, Oak Ridge, TN, 1976.
[19] J. Bernstein, R.E. Davis, L. Shimoni, N.-L. Chang, Angew. Chem., Int. Ed. Engl. 34 (1995) 1555.
[20] P.J. Steel, J. Heterocyclic Chem. 28 (1991) 1817.
[21] A. Houlton, D.M.P. Mingos, D.J. Williams, Transition Metal Chem. 19 (1994) 653.
[22] (a) A.L. Spek, Acta Crystallogr., Sect. A C34 (1990) 46;
(b) A.L. Spek, PLATON, a Multipurpose Crystallographic Tool, Utrecht University, Utrecht, The Netherlands, 1998.