

## Comparative X-ray study of three nickel(II)–thiocyanate compounds

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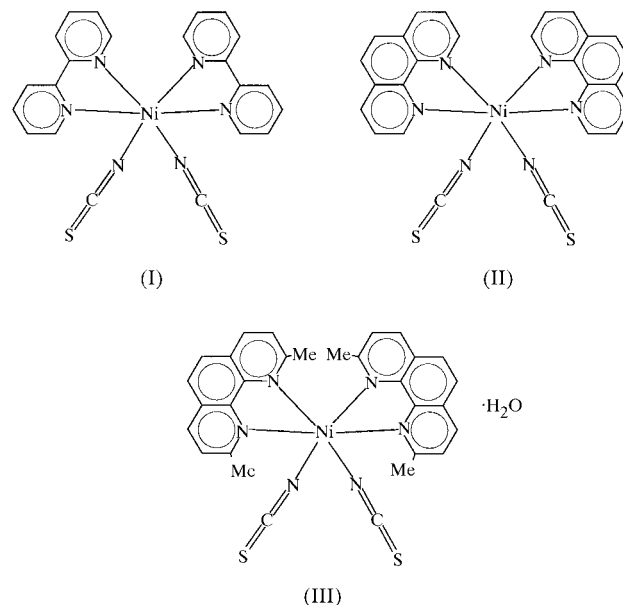
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Three *cis* nickel–dithiocyanate (SCN) complexes with different *N,N'*-bidentate bases have been prepared and their crystal structures determined: bis(2,2′-bipyridine-*N,N'*)bis(ato-*N*)nickel(II), [Ni(SCN)<sub>2</sub>(C<sub>10</sub>H<sub>8</sub>N<sub>2</sub>)<sub>2</sub>], bis(1,10-phenanthroline-*N,N'*)bis(thiocyanato-*N*)nickel(II), [Ni(SCN)<sub>2</sub>(C<sub>12</sub>H<sub>8</sub>N<sub>2</sub>)<sub>2</sub>], and bis(2,9-dimethyl-1,10-phenanthroline-*N,N'*)bis(thiocyanato-*N*)nickel(II) monohydrate, [Ni(SCN)<sub>2</sub>(C<sub>12</sub>H<sub>8</sub>N<sub>2</sub>)<sub>2</sub>]·H<sub>2</sub>O. Distortions due to ligand size are discussed.

### Comment

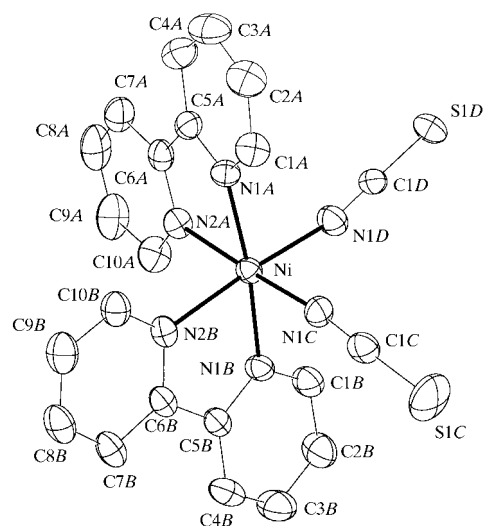
In the last few years, we have developed a sustained interest in the study of complexes with sulfur-containing ambidentate anionic ligands (those with a capability to coordinate through more than one non-equivalent site), like thiosulfate, sulfite, *etc.*, and have focused mainly on the effects introduced in the structure by the change of similar but differently sized *N,N'*-bidentate organic ligands (Freire *et al.*, 1999; Freire, Baggio, Suescun & Baggio, 2000; Freire, Baggio, Mombru & Baggio, 2000; Freire, Baggio, Mariezcurrena & Baggio, 2000). Nickel(II) is quite an interesting probe for this purpose because when coordinating to non-interacting monodentate ligands (Søtøfte *et al.*, 1976; Leban *et al.*, 1987; Vicente *et al.*, 1996; Kruger & McKee, 1996), or when the steric hindrances eventually arising among them can be satisfactorily solved by a spatial rearrangement of the latter (Perec *et al.*, 1999; Povse *et al.*, 1998), the resulting chromophore is absolutely regular. Thus, we tried to examine the distortions which would arise from this ideal octahedral configuration when these non-interacting conditions were set aside in a somehow ‘continuous’ way, for example, by the inclusion of bidentate ligands of increasing size and, concomitantly, disturbing effect. We have thus prepared a series of three Ni<sup>2+</sup> complexes containing both small monodentate ligands (SCN: thiocyanate) as well as larger *N,N'*-bidentate bases (bpy: 2,2′-bipyridine; phen: 1,10-phenanthroline; dmph: 2,9-dimethyl-1,10-phenanthroline),

namely Ni(bpy)<sub>2</sub>(SCN)<sub>2</sub>, (I), Ni(phen)<sub>2</sub>(SCN)<sub>2</sub>, (II), and Ni(dmph)<sub>2</sub>(SCN)<sub>2</sub>·H<sub>2</sub>O, (III), in order to compare the differences appearing in their coordination geometry.

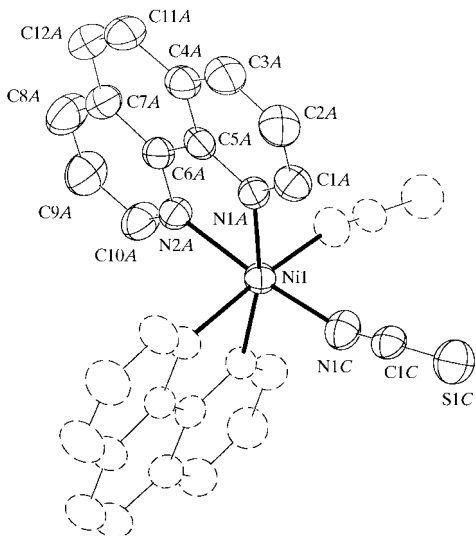


There are, in the literature, examples of structures which are similar to those presented herein: structure (II) has already been reported, though with a larger *R* factor (Travnicek *et al.*, 1998), and it is isostructural with the analogous Cu, Fe and Mn compounds (Parker *et al.*, 1996; Gallois *et al.*, 1990; Holleman *et al.*, 1994, respectively); structure (III) is isostructural with Fe(SCN)<sub>2</sub>(dmph)<sub>2</sub> (Figg *et al.*, 1992); structure (I) displays a similar coordination, though with a different crystal structure, to Ru(SCN)<sub>2</sub>(bipy)<sub>2</sub> (Herber *et al.*, 1989). However, this is the first complete series with a unique cation to be presented, thus making it suitable for comparison purposes.

The three compounds are monomeric. Compounds (I) and (III) have one independent monomer per asymmetric unit.

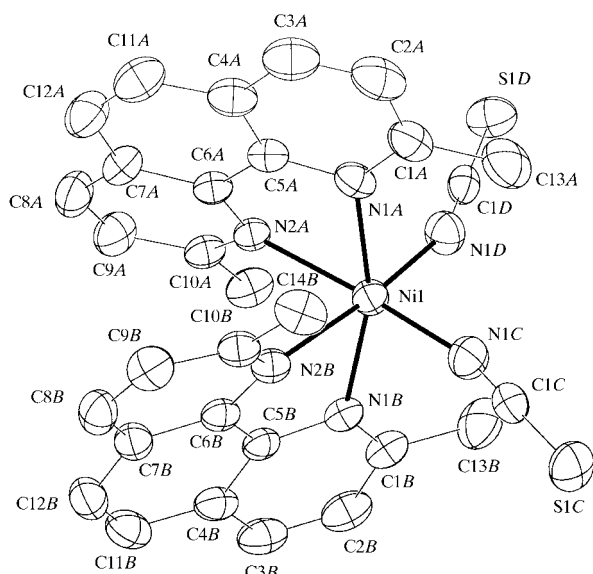


**Figure 1**  
Molecular drawing of the monomer in (I) showing the atomic labeling. Displacement ellipsoids are drawn at the 50% probability level.



**Figure 2**  
Molecular drawing of the monomer in (II) showing the atomic labeling. Note that only half of the unit is independent, the rest being generated by symmetry. Displacement ellipsoids are drawn at the 50% probability level.

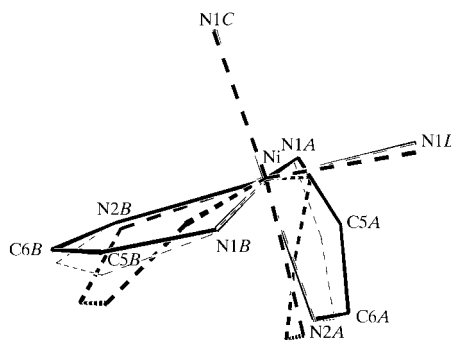
That in (II), instead, is positioned on a twofold axis through the cation thus rendering only half of it independent. In all three structures, the  $\text{Ni}^{2+}$  cation appears surrounded by very similar hexacoordinated octahedral environments (Figs. 1–3) achieved through two SCN molecules binding through N, as expected, and completed by two dinitrogenated bidentate ligands almost at right angles to each other (however, see discussion below). Tables 1, 2 and 3 allow an easy comparison of the coordination bond distances and angles in all three structures. Inspection of the values therein shows both the similarity among the nickel environments, as well as the fact that the main departures from regularity are due to the



**Figure 3**  
Molecular drawing of the monomer in (III) showing the atomic labeling. Displacement ellipsoids are drawn at the 50% probability level.

bidentate character of the bases, with their small bite angle of *ca*  $80^\circ$  promoting the major distortions.

For bipy and phen, the analogies go even further, extending to the way in which the ligands attach to the core, with the planar ligands being almost parallel to the coordination plane defined by their bites, with deviations of  $8.5 (1)$  and  $4.7 (1)^\circ$  for bipy, and  $7.6 (1)^\circ$  for phen, and the linear thiocyanates being almost parallel to the  $\text{Ni}-\text{N}_{\text{SCN}}$  coordination direction and nearly normal to each other [ $93.6 (2)^\circ$  for bipy and  $96.6 (1)^\circ$  for phen]. The situation is quite different for dmph, where to overcome the important steric hindrance introduced by the bulky methyl groups, the planar ligands depart sensibly from the plane of coordination [ $36.7 (2)$  and  $36.2 (2)^\circ$ , respectively], and SCN groups being  $131.9 (2)^\circ$  apart. This situation can be seen clearly in Fig. 4, where a comparative sketch of the three coordination cores is presented. Steric effects are such that the two dmph molecules in (III) are forced to move towards each other in order to make room for the protruding methyl groups, and this leads to a surprisingly small dihedral angle of  $29.7 (2)^\circ$  between the dmph groups, compared with an angle of  $95.6 (2)^\circ$  for bipy and  $90.8 (1)^\circ$  for phen.



**Figure 4**  
Schematic superposition of the cores of the three coordination polyhedra, showing different degrees of deformation. The hard full line represents bipy, thin broken lines phen and hard broken lines dmph.

The whole situation jeopardizes the ability of the free  $sp^2$  orbitals of the dmph N atoms to fully overlap with those of the cation; the result is a clear weakening of the  $\text{Ni}-\text{N}_{\text{dmph}}$  bond, as well as a shortening of the  $\text{Ni}-\text{N}_{\text{SCN}}$  bond length in order to allow for valence-bond conservation.

The stresses arising from coordination are also revealed in the deformation of the dmph ligand, which deviates from planarity. As a measure of this deformation, we compare the dihedral angles between lateral loops in all three cases:  $4.1 (2)/5.3 (2)^\circ$  for bipy (mainly the result of the unhindered rotation around the C5–C6 bond),  $2.2 (1)^\circ$  for phen and  $13.7 (2)/12.2 (2)^\circ$  for dmph. Fig. 4 shows that the dmph distortion also has an important component of twisting around the C5A–C6A bond, as evidenced by the N1–C5–C6–N2 torsion angles presented in Table 3.

As expected, there are no unusually short intermolecular contacts in the structures, packing interactions being mainly van der Waals in nature.

## Experimental

Single crystals of the three title compounds were obtained by diffusion in an undisturbed liquid set-up containing a lower mixture of aqueous solutions of nickel nitrate and potassium thiocyanate and an upper methanolic solution of the corresponding base. In all cases, the reactants were present in a 1:1:1 molar ratio. All the specimens appeared at the interface, those corresponding to (I) and (II) as small thin violet plates, while those for (III) consisting of slightly thicker tablets of a turquoise color. Bulk material in the form of crystalline powder and used for the different analyses performed was easily obtained by direct mixing of the above-mentioned solutions. All starting materials were purchased from Aldrich and used without further purification. Elemental analyses (C, H, N) were performed on a Carlo Erba EA 1108 instrument. Nickel was determined on a Shimadzu AA6501 spectrophotometer.

### Compound (I)

#### Crystal data

[Ni(SCN)<sub>2</sub>(C<sub>10</sub>H<sub>8</sub>N<sub>2</sub>)<sub>2</sub>]  
 $M_r = 487.24$   
 Monoclinic,  $P2_1/c$   
 $a = 16.647$  (3) Å  
 $b = 16.385$  (3) Å  
 $c = 8.0530$  (16) Å  
 $\beta = 98.08$  (3)°  
 $V = 2174.7$  (8) Å<sup>3</sup>  
 $Z = 4$

$D_x = 1.488$  Mg m<sup>-3</sup>  
 Mo  $K\alpha$  radiation  
 Cell parameters from 30 reflections  
 $\theta = 7.5$ – $15^\circ$   
 $\mu = 1.11$  mm<sup>-1</sup>  
 $T = 293$  (2) K  
 Plate, violet  
 $0.24 \times 0.18 \times 0.08$  mm

#### Data collection

Rigaku AFC-7S diffractometer  
 $\omega/2\theta$  scans  
 Absorption correction:  $\psi$  scan  
 (Molecular Structure Corporation, 1988)  
 $T_{\min} = 0.84$ ,  $T_{\max} = 0.92$   
 6054 measured reflections  
 5008 independent reflections  
 3122 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.061$   
 $\theta_{\text{max}} = 27.5^\circ$   
 $h = -21 \rightarrow 21$   
 $k = -21 \rightarrow 0$   
 $l = -1 \rightarrow 10$   
 3 standard reflections  
 every 150 reflections  
 intensity decay: <3%

#### Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.063$   
 $wR(F^2) = 0.205$   
 $S = 1.04$   
 5008 reflections  
 280 parameters

H-atom parameters constrained  
 $w = 1/[\sigma^2(F_o^2) + (0.134P)^2]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\text{max}} < 0.01$   
 $\Delta\rho_{\text{max}} = 1.12$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -1.15$  e Å<sup>-3</sup>

**Table 1**

Selected geometric parameters (Å, °) for (I).

Ni–N1D	2.040 (4)	Ni–N2B	2.104 (3)
Ni–N1A	2.057 (3)	S1C–C1C	1.631 (5)
Ni–N1C	2.065 (4)	C1C–N1C	1.138 (6)
Ni–N1B	2.076 (3)	S1D–C1D	1.622 (4)
Ni–N2A	2.096 (3)	C1D–N1D	1.166 (5)
N1D–Ni–N1A	88.29 (14)	N1B–Ni–N2A	95.87 (14)
N1D–Ni–N1C	91.45 (14)	N1D–Ni–N2B	173.92 (14)
N1A–Ni–N1C	98.04 (14)	N1A–Ni–N2B	97.60 (14)
N1D–Ni–N1B	95.53 (14)	N1C–Ni–N2B	89.26 (13)
N1A–Ni–N1B	172.83 (13)	N1B–Ni–N2B	78.46 (13)
N1C–Ni–N1B	87.96 (15)	N2A–Ni–N2B	90.94 (13)
N1D–Ni–N2A	88.75 (14)	N1C–C1C–S1C	178.0 (4)
N1A–Ni–N2A	78.11 (14)	N1D–C1D–S1D	179.0 (4)
N1C–Ni–N2A	176.13 (14)		
N1A–C5A–C6A–N2A	–1.9 (5)	N1B–C5B–C6B–N2B	–3.8 (5)

### Compound (II)

#### Crystal data

[Ni(SCN)<sub>2</sub>(C<sub>12</sub>H<sub>8</sub>N<sub>2</sub>)<sub>2</sub>]  
 $M_r = 535.28$   
 Orthorhombic,  $Pbcn$   
 $a = 13.018$  (3) Å  
 $b = 10.116$  (2) Å  
 $c = 17.536$  (4) Å  
 $V = 2309.3$  (8) Å<sup>3</sup>  
 $Z = 4$   
 $D_x = 1.540$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation  
 Cell parameters from 30 reflections  
 $\theta = 7.5$ – $15^\circ$   
 $\mu = 1.05$  mm<sup>-1</sup>  
 $T = 293$  (2) K  
 Plate, violet  
 $0.20 \times 0.20 \times 0.14$  mm

#### Data collection

Rigaku AFC-7S diffractometer  
 $\omega/2\theta$  scans  
 Absorption correction:  $\psi$  scan  
 (Molecular Structure Corporation, 1988)  
 $T_{\min} = 0.77$ ,  $T_{\max} = 0.85$   
 3409 measured reflections  
 2663 independent reflections  
 1410 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.032$   
 $\theta_{\text{max}} = 27.5^\circ$   
 $h = -1 \rightarrow 16$   
 $k = -1 \rightarrow 13$   
 $l = -22 \rightarrow 1$   
 3 standard reflections  
 every 150 reflections  
 intensity decay: <3%

#### Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.029$   
 $wR(F^2) = 0.091$   
 $S = 0.97$   
 2663 reflections  
 159 parameters

H-atom parameters constrained  
 $w = 1/[\sigma^2(F_o^2) + (0.0534P)^2]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\text{max}} < 0.01$   
 $\Delta\rho_{\text{max}} = 0.28$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.25$  e Å<sup>-3</sup>

**Table 2**

Selected geometric parameters (Å, °) for (II).

Ni–N1C	2.038 (2)	Ni–N2A	2.1056 (19)
Ni–N1C <sup>i</sup>	2.038 (2)	Ni–N2A <sup>i</sup>	2.1056 (19)
Ni–N1A	2.0990 (19)	S1C–C1C	1.618 (3)
Ni–N1A <sup>i</sup>	2.0990 (19)	C1C–N1C	1.153 (3)
N1C–Ni–N1C <sup>i</sup>	92.51 (13)	N1A <sup>i</sup> –Ni–N2A	92.23 (7)
N1C–Ni–N1A	92.45 (8)	N1C–Ni–N2A <sup>i</sup>	88.68 (9)
N1C <sup>i</sup> –Ni–N1A	96.27 (8)	N1C <sup>i</sup> –Ni–N2A <sup>i</sup>	171.35 (8)
N1C–Ni–N1A <sup>i</sup>	96.27 (8)	N1A–Ni–N2A <sup>i</sup>	92.23 (7)
N1C <sup>i</sup> –Ni–N1A <sup>i</sup>	92.45 (8)	N1A <sup>i</sup> –Ni–N2A <sup>i</sup>	78.90 (7)
N1A–Ni–N1A <sup>i</sup>	167.37 (10)	N2A–Ni–N2A <sup>i</sup>	91.42 (11)
N1C–Ni–N2A	171.35 (8)	N1C–C1C–S1C	178.8 (2)
N1C <sup>i</sup> –Ni–N2A	88.68 (9)	C1C–N1C–Ni	162.0 (2)
N1A–Ni–N2A	78.90 (7)		
N1A–C5A–C6A–N2A	1.6 (5)		

Symmetry code: (i)  $1 - x, y, \frac{3}{2} - z$ .

### Compound (III)

#### Crystal data

[Ni(SCN)<sub>2</sub>(C<sub>14</sub>H<sub>12</sub>N<sub>2</sub>)<sub>2</sub>].H<sub>2</sub>O  
 $M_r = 609.40$   
 Triclinic,  $P\bar{1}$   
 $a = 9.689$  (1) Å  
 $b = 10.757$  (1) Å  
 $c = 14.754$  (1) Å  
 $\alpha = 77.71$  (1)°  
 $\beta = 79.00$  (1)°  
 $\gamma = 70.07$  (1)°  
 $V = 1400.8$  (2) Å<sup>3</sup>

$Z = 2$   
 $D_x = 1.445$  Mg m<sup>-3</sup>  
 Mo  $K\alpha$  radiation  
 Cell parameters from 10 054 reflections  
 $\theta = 3.5$ – $27.5^\circ$   
 $\mu = 0.88$  mm<sup>-1</sup>  
 $T = 293$  (2) K  
 Plate, turquoise  
 $0.23 \times 0.22 \times 0.16$  mm

## Data collection

Bruker SMART 6000 diffractometer  
 $\omega$  scans  
 10079 measured reflections  
 6316 independent reflections  
 3612 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.031$   
 $\theta_{\text{max}} = 27.5^\circ$   
 $h = -12 \rightarrow 12$   
 $k = -13 \rightarrow 13$   
 $l = -19 \rightarrow 18$

## Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.040$   
 $wR(F^2) = 0.081$   
 $S = 0.97$   
 6316 reflections  
 366 parameters

H-atom parameters constrained  
 $w = 1/[\sigma^2(F_o^2) + (0.040P)^2]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\text{max}} < 0.01$   
 $\Delta\rho_{\text{max}} = 0.40 \text{ e } \text{\AA}^{-3}$   
 $\Delta\rho_{\text{min}} = -0.27 \text{ e } \text{\AA}^{-3}$

**Table 3**

Selected geometric parameters ( $\text{\AA}$ ,  $^\circ$ ) for (III).

Ni—N1C	2.011 (2)	Ni—N2A	2.2054 (19)
Ni—N1D	2.022 (2)	S1C—C1C	1.629 (3)
Ni—N1A	2.138 (2)	C1C—N1C	1.158 (3)
Ni—N1B	2.1419 (19)	S1D—C1D	1.620 (3)
Ni—N2B	2.1886 (19)	C1D—N1D	1.157 (3)
N1C—Ni—N1D	97.81 (9)	N1B—Ni—N2B	76.67 (7)
N1C—Ni—N1A	100.37 (9)	N1C—Ni—N2A	172.14 (8)
N1D—Ni—N1A	91.63 (8)	N1D—Ni—N2A	89.55 (8)
N1C—Ni—N1B	90.97 (8)	N1A—Ni—N2A	76.56 (8)
N1D—Ni—N1B	99.07 (8)	N1B—Ni—N2A	90.58 (7)
N1A—Ni—N1B	163.24 (8)	N2B—Ni—N2A	81.85 (7)
N1C—Ni—N2B	91.01 (8)	N1C—C1C—S1C	179.0 (2)
N1D—Ni—N2B	170.31 (8)	N1D—C1D—S1D	178.9 (2)
N1A—Ni—N2B	90.71 (7)		
N1A—C5A—C6A—N2A	−5.6 (3)	N1B—C5B—C6B—N2B	−5.6 (3)

H atoms unambiguously defined by the stereochemistry (C—H's) were placed at calculated positions and allowed to ride on their host C atoms both for the coordinates as well as for the displacement parameters; methyl H atoms were further allowed to rotate around the C—C bond. In compound (III), the hydration water molecule site appeared disordered and slightly depleted (overall s.o.f. ca 0.88); the corresponding H atoms could not be found and were accordingly disregarded. The molecule in compound (II) is positioned on a twofold symmetry axis, with only half of the monomer being independent. Crystals of (I) did not diffract adequately, and accordingly the data set gathered was of poor quality. This was evidenced in the rather large  $R_{\text{int}}$  (0.061) and  $R$  (0.063) factors attained, as well as in the residual peaks in the final difference Fourier map. The latter were scattered around the cation position, at about 0.90  $\text{\AA}$  from its center.

For compounds (I) and (II), data collection, cell refinement and data reduction: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988), for compound (III), data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*. For all compounds, program(s) used to solve

structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL/PC* (Sheldrick, 1991); software used to prepare material for publication: *PARST* (Nardelli, 1983) and *CSD* (Allen & Kennard, 1993).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: BK1604). Services for accessing these data are described at the back of the journal.

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