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Single-Strand Polymer in catena-Poly{[*trans*-bis(6,8-dimethyl[1,2,4]triazolo[3,4-*b*]pyridazine-*N*²)cadmium]- μ -(thiocyanato-*N*:*S*)- μ -(thiocyanato-*S*:*N*)}¹

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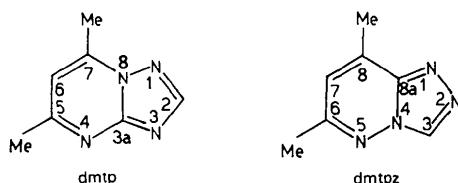
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Abstract. $[\text{Cd}(\text{C}_8\text{H}_8\text{N}_4)_2(\text{NCS})_2]_n$, $M_r = 524.90$ (monomeric unit), monoclinic, $P2_1/c$, $a = 19.560$ (5), $b = 9.562$ (4), $c = 11.521$ (4) Å, $\beta = 94.25$ (1)°, $V = 2149$ (1) Å³, $Z = 4$, D_m (by flotation) = 1.62 (1), $D_x = 1.62$ Mg m⁻³, $\lambda(\text{Cu } K\alpha) = 1.54178$ Å, $\mu = 10.361$ mm⁻¹, $F(000) = 1048$, room temperature, $R = 0.041$ for 2224 independent observed reflections. The Cd^{II} cation is octahedrally coordinated by two monodentate N-bonded organic ligands (*trans*) with Cd-N bonds of 2.396 (8) and 2.357 (8) Å, two N-bonded thiocyanate groups (*cis*) with Cd-N distances of 2.311 (8) and 2.347 (9) Å and two S-bonded thiocyanate groups (*cis*) with Cd-S bonds of 2.783 (3) and 2.680 (3) Å. The double thiocyanate bridge links the Cd^{II} cations to form slightly zigzag polymeric chains running along c .

Introduction. Organic bases differing from purine derivatives in the position and/or number of ring nitrogen atoms (azapurines) are known to form complexes with transition metals (Dillen, Lenstra, Haasnoot & Reedijk, 1983; Biagini Cingi, Manotti Lanfredi, Tiripicchio, Haasnoot & Reedijk, 1983). Therefore, they may be used as model compounds for the study of the interactions of these metals with nucleic bases and their derivatives. The non-hydrazinic triazole-ring nitrogen atom [N(3)] of 5,7-dimethyl[1,2,4]triazolo[1,5-*a*]pyrimidine (dmtp) (see scheme) has been shown to be preferred for coordination from several structural determinations of its metal complexes

(Biagini Cingi, Manotti Lanfredi, Tiripicchio, Haasnoot & Reedijk, 1984, 1985; Biagini Cingi, Manotti Lanfredi, Tiripicchio, Reedijk & Haasnoot, 1986). As was pointed out earlier (Biagini Cingi, Manotti Lanfredi, Tiripicchio, Haasnoot & Reedijk, 1984) this preference may be due to the presence of adjacent bulky substituents in the ring.

We have now investigated the behaviour as a ligand of 6,8-dimethyl[1,2,4]triazolo[3,4-*b*]pyridazine (hereafter abbreviated as dmtpz). In this ligand only hydrazinic nitrogen atoms are available for coordination, two in the triazole ring, one in the pyridazine ring. Steric arguments would predict coordination at N(2) of dmtpz.



IUPAC numbering schemes of dmtp and dmtpz ligands.

Crystals of the thiocyanate complex $\text{Cd}(\text{dmtpz})_2(\text{NCS})_2$ were obtained from aqueous solutions. Their X-ray structure was solved in order to compare it with that of the cadmium thiocyanate complex of the isomeric ligand dmtp, $\text{Cd}(\text{dmtp})_2(\text{NCS})_2(\text{H}_2\text{O})_2$ (Dillen, Lenstra, Haasnoot & Reedijk, 1983).

Experimental. The ligand dmtpz was prepared from 4-amino-1,2,4-triazole and 2,4-pentanedione (Kabe, Bhooshan, Novinson, Hillyard, Garner & Robins, 1983), the 4-amino-1,2,4-triazole from formic acid and hydrazine (Herbst & Garrison, 1953). The title compound was obtained by adding dmtpz (4 mmol) dissolved in 1:1 aqueous ethanol (10 ml) to a solution of Cd(NO₃)₂ (2 mmol) and NH₄SCN (4 mmol) in water (20 ml).

A prismatic crystal approximately 0.15 × 0.18 × 0.25 mm, Siemens AED diffractometer, Ni-filtered Cu K α radiation, 21 reflections ($25.4 \leq \theta \leq 34.4^\circ$) for accurate unit-cell parameters; 4051 independent reflections with $3 \leq \theta \leq 65^\circ$, $-22 \leq h \leq 22$, $0 \leq k \leq 11$, $0 \leq l \leq 13$, 2224 reflections with $I \geq 2\sigma(I)$ considered observed. The standard reflection (511) showed no significant decrease in intensity; Lp correction; absorption ignored; Patterson and Fourier methods; full-matrix least-squares refinement, anisotropic for all non-hydrogen atoms (SHELX76, Sheldrick, 1976). The H atoms were located from a ΔF synthesis; the methyl H atoms at C(111) were found disordered and distributed in two staggered positions, with an occupancy factor of 0.5. H atoms were not refined, but were included in the last structure factor calculations with isotropic thermal parameters; $\sum w|\Delta F|^2$ minimized; final $R = 0.041$, $wR = 0.049$. Unit weights chosen at every stage of the refinement. $(\Delta/\sigma)_{\text{max}} = 0.8$. Max. height in final difference map ≤ 10.6 e Å⁻³. Atomic scattering factors and anomalous-scattering corrections from *International Tables for X-ray Crystallography* (1974). Calculations performed on the Cyber 76 computer of the Consorzio per la Gestione del Centro Elettronico Interuniversitario dell'Italia Nord-Orientale, Casalecchio (Bologna), with financial support from the University of Parma.*

Discussion. Final atomic parameters are listed in Table 1. Bond distances and angles are given in Table 2. Fig. 1 gives a picture of the chain-like structure of the compound together with the atomic numbering as used in the tables.

The Cd^{II} cations are coordinated by two triazole N atoms (*trans*) from monodentate dmtpz ligands; two N atoms (*cis*) and two S atoms (*cis*) from four N,S-bridging thiocyanate groups complete the octahedral environment of the metal ions, which, through the double thiocyanate bridge, give rise to slightly zigzag polymeric chains.

The structure of the present compound is very different from that found for Cd(dmtp)₂(NCS)₂(OH₂)₂

Table 1. Fractional atomic coordinates ($\times 10^4$) with equivalent isotropic thermal parameters for the non-hydrogen atoms, with e.s.d.'s in parentheses

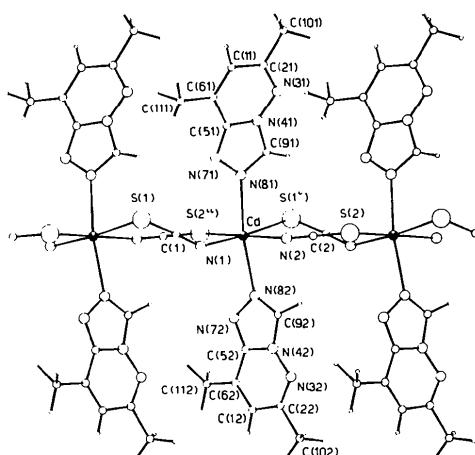
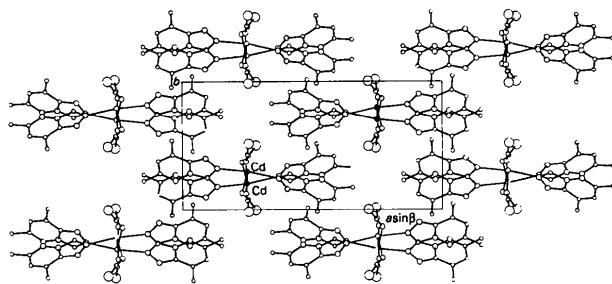
	x	y	z	B_{eq} (Å ²)
Cd	7486 (1)	7036 (1)	930 (1)	2.44 (2)
S(1)	7156 (1)	10029 (3)	4281 (2)	3.07 (6)
S(2)	7435 (2)	10157 (3)	-2630 (2)	3.83 (7)
N(1)	7687 (4)	8691 (9)	2377 (7)	3.25 (22)
N(2)	7540 (4)	8759 (10)	-511 (8)	3.59 (25)
N(31)	4639 (4)	7672 (9)	-572 (8)	3.74 (24)
N(41)	5232 (3)	7800 (9)	160 (7)	2.92 (20)
N(71)	5963 (4)	8389 (8)	1646 (7)	2.91 (22)
N(81)	6288 (4)	7583 (8)	845 (7)	3.23 (24)
N(32)	10262 (4)	7710 (10)	-291 (8)	3.98 (28)
N(42)	9708 (3)	7176 (9)	247 (7)	2.91 (19)
N(72)	9101 (4)	5980 (9)	1457 (8)	3.42 (24)
N(82)	8668 (4)	6795 (9)	704 (7)	3.31 (25)
C(1)	7447 (4)	9216 (11)	3172 (8)	2.50 (24)
C(2)	7492 (4)	9311 (11)	-1395 (9)	2.54 (25)
C(11)	4168 (5)	9175 (12)	894 (10)	3.76 (30)
C(21)	4124 (5)	8358 (10)	-174 (10)	3.49 (28)
C(51)	5324 (5)	8521 (10)	1204 (9)	2.64 (24)
C(61)	4755 (5)	9269 (10)	1578 (9)	3.19 (28)
C(91)	5850 (4)	7235 (11)	-57 (9)	3.38 (27)
C(101)	3450 (5)	8275 (12)	-938 (11)	5.05 (32)
C(111)	4824 (6)	10146 (12)	2677 (10)	4.56 (38)
C(12)	10923 (5)	6186 (11)	1084 (10)	3.46 (28)
C(22)	10845 (4)	7178 (12)	139 (10)	3.87 (29)
C(52)	9725 (5)	6226 (10)	1157 (9)	2.93 (25)
C(62)	10375 (5)	5684 (11)	1582 (9)	3.22 (29)
C(92)	9041 (4)	7529 (11)	-17 (9)	3.45 (28)
C(102)	11484 (5)	7741 (15)	-425 (12)	6.53 (43)
C(112)	10401 (6)	4587 (13)	2541 (10)	4.51 (34)

(Dillen, Lenstra, Haasnoot & Reedijk, 1983), where discrete octahedral complexes (with N-bonded thiocyanate ligands and monodentate dmtp molecules involved in intramolecular hydrogen bonds with coordinated water molecules to form pseudo-chelate rings) are present. Considering the ligand behaviour of the thiocyanate groups and the skeleton of the organic ligand, the structure of the title compound can better be compared with that of the cadmium thiocyanate 1,2,4-triazole complex, Cd(C₂H₃N₃)₂(NCS)₂, (Haasnoot, de Keyzer & Verschoor, 1983). This last compound shows a ...Cd(NCS)₂Cd... linear chain, but with the thiocyanate donor N and S atoms *trans*-coordinated to the metal atoms. The geometries of the eight-membered Cd(NCS)₂Cd rings in both compounds agree rather well, even if these units are asymmetric in the present and centrosymmetric in the 1,2,4-triazole complex. The S-C and C-N bond lengths (in the ring) of the title compound are normal for thiocyanate groups, while the Cd-N(thiocyanate) distances [2.311 (8) and 2.347 (9) Å], comparable to those found in the 1,2,4-triazole complex, are rather long, when compared with those found in cadmium(II) thiocyanate (Cannas, Carta, Cristini & Marongiu, 1976). This lengthening can be related, as pointed out for the triazole complex, to the Cd-N-C(thiocyanate) angles which differ sensibly from 180°, suggesting a partial hybridization of the N atoms; in the centrosymmetric eight-membered

* Anisotropic thermal parameters, a list of structure factors and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43075 (17 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. Relevant bond distances (\AA) and angles ($^\circ$)

(i) In the coordination sphere of the cadmium atom			
Cd—N(1)	2.311 (8)	Cd—N(82)	2.357 (8)
Cd—N(2)	2.347 (9)	Cd—S(1) [†]	2.783 (3)
Cd—N(81)	2.396 (8)	Cd—S(2) [†]	2.680 (3)
N(1)—Cd—N(2)	90.9 (3)	N(81)—Cd—S(1)	87.1 (2)
N(1)—Cd—S(2) [†]	95.8 (2)	N(81)—Cd—S(2) [†]	96.6 (2)
N(2)—Cd—S(1) [†]	92.0 (2)	N(81)—Cd—N(82)	168.9 (3)
S(1) [†] —Cd—S(2) [†]	81.4 (1)	N(82)—Cd—N(1)	91.8 (3)
N(1)—Cd—S(1) [†]	175.5 (2)	N(82)—Cd—N(2)	83.9 (3)
N(2)—Cd—S(2) [†]	173.5 (2)	N(82)—Cd—S(1) [†]	92.0 (2)
N(81)—Cd—N(1)	89.6 (3)	N(82)—Cd—S(2) [†]	94.2 (2)
N(81)—Cd—N(2)	85.0 (3)		
(ii) In the thiocyanate ligands			
N(1)—C(1)	1.173 (13)	N(2)—C(2)	1.145 (14)
C(1)—S(1)	1.634 (10)	C(2)—S(2)	1.633 (11)
N(1)—C(1)—S(1)	176.0 (8)	N(2)—C(2)—S(2)	177.7 (10)
Cd—N(1)—C(1)	143.1 (7)	Cd—N(2)—C(2)	161.4 (8)
Cd—S(1)—C(1)	96.9 (4)	Cd—S(2) [†] —C(2) [†]	98.5 (4)
(iii) In the organic ligands			
C(11)—C(21)	1.455 (16)	C(12)—C(22)	1.443 (16)
C(21)—N(31)	1.313 (13)	C(22)—N(32)	1.312 (12)
N(31)—N(41)	1.388 (10)	N(32)—N(42)	1.385 (11)
N(41)—C(51)	1.386 (13)	N(42)—C(52)	1.386 (13)
C(51)—C(61)	1.417 (14)	C(52)—C(62)	1.426 (14)
C(11)—C(61)	1.347 (14)	C(12)—C(62)	1.341 (15)
N(41)—C(91)	1.364 (11)	N(42)—C(92)	1.360 (10)
N(71)—N(81)	1.392 (11)	N(72)—N(82)	1.403 (11)
C(91)—N(81)	1.339 (12)	C(92)—N(82)	1.343 (13)
C(51)—N(71)	1.320 (12)	C(52)—N(72)	1.314 (13)
C(21)—C(101)	1.532 (14)	C(22)—C(102)	1.547 (15)
C(61)—C(111)	1.516 (15)	C(62)—C(112)	1.522 (16)
C(21)—C(11)—C(61)	121.7 (10)	C(22)—C(12)—C(62)	121.0 (9)
C(11)—C(21)—N(31)	124.4 (9)	C(12)—C(22)—N(32)	125.4 (8)
C(21)—N(31)—N(41)	111.8 (8)	C(22)—N(32)—N(42)	112.0 (8)
N(31)—N(41)—C(51)	128.3 (7)	N(32)—N(42)—C(52)	127.2 (7)
N(41)—C(51)—C(61)	117.4 (8)	N(42)—C(52)—C(62)	118.0 (8)
C(51)—C(61)—C(11)	116.3 (9)	C(52)—C(62)—C(12)	116.3 (9)
C(51)—N(41)—C(91)	107.2 (7)	C(52)—N(42)—C(92)	107.5 (8)
N(41)—C(91)—N(81)	106.5 (7)	N(42)—C(92)—N(82)	106.7 (8)
C(91)—N(81)—N(71)	110.8 (7)	C(92)—N(82)—N(72)	110.0 (7)
C(51)—N(71)—N(81)	105.2 (8)	C(52)—N(72)—N(82)	105.5 (8)
N(41)—C(51)—N(71)	110.2 (8)	N(42)—C(52)—N(72)	110.2 (8)
N(31)—C(21)—C(101)	115.9 (9)	N(32)—C(22)—C(102)	114.4 (9)
C(11)—C(21)—C(101)	120.5 (9)	C(12)—C(22)—C(102)	120.1 (9)
C(11)—C(61)—C(111)	123.0 (9)	C(12)—C(62)—C(112)	124.9 (9)
C(51)—C(61)—C(111)	120.6 (9)	C(52)—C(62)—C(112)	118.7 (9)
Cd—N(81)—N(71)	126.0 (6)	Cd—N(82)—N(72)	122.6 (6)
Cd—N(81)—C(91)	123.0 (6)	Cd—N(82)—C(92)	126.8 (6)

Symmetry codes: (i) $x, \frac{3}{2} - y, -\frac{1}{2} + z$; (ii) $x, \frac{3}{2} - y, \frac{1}{2} + z$.Fig. 1. Polymeric chains of $[\text{Cd}(\text{dmtpz})_2(\text{NCS})_2]$ complexes with the atomic numbering scheme.Fig. 2. Projection of the structure along \mathbf{c} .

system they are 143° , in the present asymmetric eight-membered ring they are $143.1 (7)$ and $161.4 (8)^\circ$ [the N atom of the larger angle shows some intramolecular interaction with the H atom of the triazole C—H group from one of the two coordinated dmtpz molecules, $\text{H}(92)\cdots\text{N}(2)=2.62 \text{ \AA}$]. The values of the Cd—N(organic ligand) bonds are in the range normally found for cadmium complexes involving aza derivatives of similar ligands (Purnell, Estes & Hodgson, 1976).

The analysis of the planarity data of the dmtpz molecules shows that both C(11)—C(91) and C(12)—C(92) nine-frameworks are planar [the maximum deviation from the mean plane through them being $0.029 (10) \text{ \AA}$ for the C(91) atom in the former and $0.015 (10) \text{ \AA}$ for the C(62) atom in the latter]. The packing of the chain-like complexes is shown in Fig. 2. The inversion-related (about $\frac{1}{2}, 0, 0$ and $\frac{1}{2}, \frac{1}{2}, \frac{1}{2}$) polymeric chains show a slipped stack of half of the dmtpz molecules, the separation between the involved nine-frameworks being $3.345 (12) \text{ \AA}$. Van der Waals contacts complete the packing of the chains.

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Structure de l'Octaiodotétraargentate de Bis(diazonia-6,9 dispiro[5.2.5.3]heptadécane)

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(Reçu le 20 octobre 1985, accepté le 20 juin 1986)

Abstract. $2\text{C}_{15}\text{H}_{30}\text{N}_2^{2+} \cdot \text{Ag}_4\text{I}_8^{4-}$, $M_r = 1923.5$, orthorhombic, $Pbca$, $a = 15.316(4)$, $b = 15.083(5)$, $c = 20.777(10)$ Å, $V = 4799.7(5.1)$ Å³, $Z = 4$, $D_m = 2.6$, $D_x = 2.66$ g cm⁻³, Mo $\text{K}\alpha$, $\lambda = 0.71069$ Å, $\mu = 67.1$ cm⁻¹, $F(000) = 7040$, room temperature, $R = 0.055$ for 1705 diffractometer data. The structure consists of an ionic network of dipositive tricyclic dispiro bisquaternary ammonium ions interspersed between di- μ_3 -iodo-tetra- μ_2 -iodo-diiodotetraargentate(I) anions. The organic moiety is composed of a central homopiperazinium ring in a twist-chair conformation and two lateral piperidinium rings with a chair form. The mineral $\text{Ag}_4\text{I}_8^{4-}$ counterion lies on a crystallographic centre of symmetry with silver atoms in a pseudotetrahedral environment of both terminal and bridging iodine atoms. Important deviations from ideal geometry are connected with AgI_4 tetrahedra sharing two and three edges.

Introduction. Diverses associations de l'iode d'argent avec des iodures d'ammonium substitué ont donné naissance à des conducteurs organominéraux de type électrolyte solide (Owens, Christie & Tiedeman, 1971; Thackeray & Coetzer, 1978; Rosenberg, 1983). Dans le cadre d'une étude générale des composés obtenus avec des cations tricycliques, nous avons rapporté les structures du diiodure de diazonia-6,9 dispiro[5.2.5.2]-hexadécane (Estienne, Pierrot, Baldy, Rosenberg & Robert, 1984) et du diiodure de diazonia-6,9 dispiro[5.2.5.3]heptadécane (Estienne, Daviodovics, Pierrot, Baldy, Rosenberg & Robert, 1986) respectivement désignés par DIHEXA et DIHEPTA.

Une série de composés au caractère de conducteur ionique par l'ion Ag^+ a été préparée par réactions solide-solide de l'iode d'argent sur les deux diiodures DIHEXA (Estienne & Rosenberg, 1986a) et DIHEPTA (Estienne & Rosenberg, 1986b). Présente-

ment il ne nous a pas été possible d'isoler directement à partir des milieux réactionnels des échantillons cristallins, toutefois des essais de recristallisation des dérivés de DIHEPTA dans le diméthylformamide se sont avérés concluants (Estienne, 1986). Une stoechiométrie 1:2 entre DIHEPTA et AgI , révélée par rayons X, se trouve à l'origine d'un sel double faisant intervenir un nouvel anion iodoargentate. Nous reportons ici la structure du composé AB_2 correspondant que nous désignerons dans la suite par BDH-TAI (Bisdihépta-tétraiodoargentate).

Partie expérimentale. Densité mesurée par flottation. Cristal incolore $0.15 \times 0.10 \times 0.10$ mm. Diffractomètre CAD-4 Enraf–Nonius. Paramètres de la maille cristalline déterminés à partir d'un affinement par moindres carrés des angles 2θ , compris entre 11,9 et 20,7° de 25 réflexions de forte intensité. $(\sin\theta/\lambda)_{\max} = 0.53$ Å⁻¹; $h = 0, 16$; $k = -15, 15$; $l = 0, 18$. Réflexions standards: $\bar{5}, 0, \bar{1}0, 2, \bar{1}\bar{1}, \bar{2}, \bar{1}, \bar{8}, 10$; chute d'intensité à l'issue de 84 heures d'exposition de 1,4%. Nombre de réflexions mesurées: 5776. Espace moyen de 2904 réflexions ($R_{\text{int}} = 0.057$) dont 1705 sont introduites dans l'affinement final selon le critère $I > 2\sigma(I)$. Structure résolue par méthode directe avec localisation des positions des quatre atomes d'iode et des deux atomes d'argent à l'aide du programme MULTAN (Main *et al.*, 1980). Fonction $\sum w\Delta F^2$ minimisée dans les affinements par moindres carrés selon une pondération unitaire. Affinement anisotrope des atomes de l'anion $\text{Ag}_4\text{I}_8^{4-}$, isotrope pour les atomes de carbone et d'azote du cation $\text{C}_{15}\text{H}_{30}\text{N}_2^{2+}$ sujet à un désordre conformationnel (123 variables). $R = 0.055$; $wR = 0.072$; $S = 14$; (Δ/σ) de valeur nulle dans un dernier cycle d'affinement. Maximum de 1,3 e Å⁻³ dans la dernière Fourier différence, avec présence des pics de plus forte intensité dans l'entourage des atomes d'iode. Les facteurs de diffusion