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The Crystal and Molecular Structure of Di-μ-(*N*,*N*-diethylnicotinamide-*O*,*N*)-tetraisothiocyanatodizinc

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The title compound, $Zn_2(C_{10}H_{14}N_2O)_2(NCS)_4$, is triclinic, space group $P\bar{1}$, with $a = 10·387$ (6), $b = 12·436$ (6), $c = 7·835$ (5) Å, $\alpha = 108·4$ (1), $\beta - 117·2$ (1), $\gamma - 97·8$ (1)°, $Z = 1$. The structure has been determined from X-ray intensities collected on an automated diffractometer. The structure, solved by Patterson and Fourier methods and refined by least squares, consists of centrosymmetric dimers with tetrahedral coordination around zinc. The two tetrahedra of the dimer are held together by *N*,*N*-diethylnicotinamide molecules acting as bridges through pyridine N and amide O. Packing of the dimers is determined by van der Waals interactions only.

Introduction

Structural studies on metal complexes of *N*,*N*-diethylnicotinamide (DENA) were undertaken in order to define the ligand properties of this respiration stimulant. The structures of the following complexes have been determined so far: Cd(DENA)$(SCN)_2$, (I) [μ-(*N*,*N*-diethylnicotinamide-*O*,*N*)-di-μ-thiocyanato-cadmium; Bigoli, Braibanti, Pellinghelli & Tiripicchio (1972)], $Mn(DENA)_2(NCS)_2$, (II) [di-μ-(*N*,*N*-diethylnicotinamide-*O*,*N*)-diisothiocyanatomanganese; Bigoli, Braibanti, Pellinghelli & Tiripicchio (1973*a*)], $Zn(DENA)_2(NCS)_2(H_2O)_2$, (III) [diaquobis-(*N*,*N*-diethylnicotinamide)diisothiocyanatozinc; Bigoli, Braibanti, Pellinghelli & Tiripicchio (1973*b*)]. In these compounds the complexes are octahedral but differ from one another in the disposition of the donor atoms and in the type and number of bridges. In (I) and (II) the DENA ligand forms one and two bridges respectively through pyridine N and amide O; in (III) the organic ligand is bound to the metal through pyridine N only. Crystals of (III), when left to stand in the presence of the mother liquor, change, with a complete transformation of the metal complex, into crystals of the title compound, $Zn_2(DENA)_2(NCS)_4$, (IV). The coordination of the metal changes from octahedral to tetrahedral.

In the present paper the results of the crystal structure determination of (IV) are given.

Experimental

Preparation

Crystals of $Zn(DENA)_2(NCS)_2(H_2O)_2$ remain unaltered in the mother liquor for many days, then transform rapidly into the stable $Zn_2(DENA)_2(NCS)_4$.

Crystal data

Compound: di-μ-(*N*,*N*-diethylnicotinamide-*O*,*N*)-tetraisothiocyanatodizinc, $Zn_2(C_{10}H_{14}N_2O)_2(NCS)_4$.
F.W. 719·54.

Unit cell: the parameters, determined from rotation and Weissenberg photographs and refined with data obtained on an automated single-crystal diffractometer (Cu $K\alpha$, $\lambda = 1·54178$ Å), are:
$a = 10·387$ (6), $b = 12·436$ (6), $c = 7·835$ (5) Å;
$\alpha = 108·4$ (1), $\beta = 117·2$ (1), $\gamma = 97·8$ (1)°;
$V = 805·7$ Å^3; $Z = 1$;
$D_x = 1·483$, $D_m = 1·485$ g cm^{-3};
μ(Cu $K\alpha$) $= 44·96$ cm^{-1}; $F(000) = 368$.

Space group: $P\bar{1}(C_i^1$, No. 2) from the structure determination.

Intensity data

The intensities were collected on a Siemens diffractometer on line with a Siemens 304/P computer with Ni-filtered Cu $K\alpha$ radiation and the ω–2θ scan technique. A rather irregular prism of approximate dimensions $0{\cdot}14\times0{\cdot}10\times0{\cdot}30$ mm was aligned with its [001] axis along the φ axis of the diffractometer and all reflexions with $2\theta<140°$ were registered. 3061 independent reflexions were measured and 2767 were used in the analysis, taking as unobserved reflexions whose intensities were less than twice their standard deviations. Corrections for Lorentz and polarization factors were made in the usual way but no correction was made for absorption effects. The absolute scale factor and overall isotropic temperature factor were determined by Wilson's (1942) method.

Structure determination and refinement

The structure was solved by Patterson and Fourier methods. The refinement was carried out by block-diagonal least-squares methods, first with isotropic, then with anisotropic thermal parameters. The hydrogen atoms were located from a difference synthesis. Further least-squares cycles were computed including the hydrogen atoms with isotropic thermal parameters. Unit weights were assumed for all the reflexions at the beginning of the refinement, then the scheme $1/w=(A+BF_o)^2$ was used in which $A=0{\cdot}465$ and $B=0{\cdot}0247$ were determined by plotting ΔF against $|F_o|$. The final R was 0·045 (observed reflexions only).

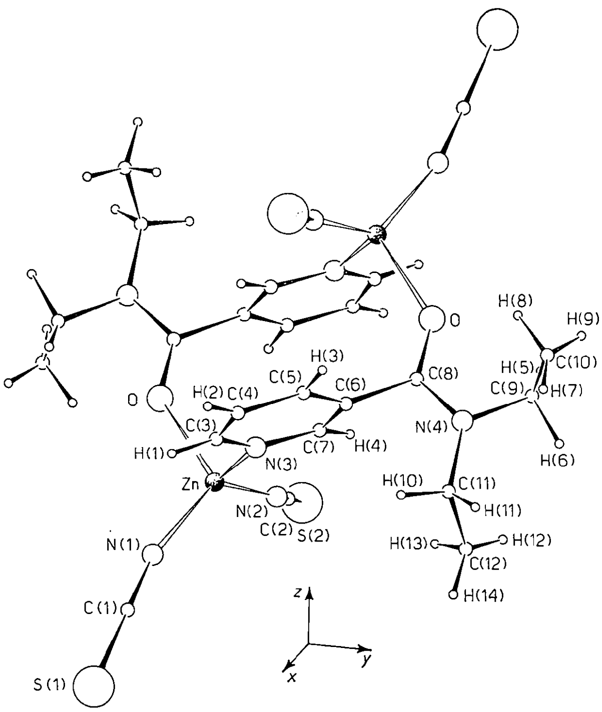

Fig. 1. Clinographic projection of the dimer $Zn_2(DENA)_2(NCS)_4$.

Atomic scattering factors given by Cromer & Mann (1968) were used for non-hydrogen atoms and those of Stewart, Davidson & Simpson (1965) for hydrogen atoms.

All the calculations were performed on the CDC 6600 computer of the Centro di Calcolo Elettronico Interuniversitario dell'Italia Nord-Orientale (Bologna), with the programs written by Immirzi (1967).

Final atomic coordinates and thermal parameters are reported in Tables 1 and 2.*

Discussion

The basic units of the structure are centrosymmetric dimers $Zn_2(DENA)_2(NCS)_4$, with tetrahedral coordination for the metal atoms. The two tetrahedra are held together by two bridging DENA molecules, bound

Table 1. *Final fractional atomic coordinates* ($\times 10^4$) *with e.s.d.'s in parentheses*

	x	y	z
Zn	198 (1)	3707 (1)	1219 (1)
S(1)	2372 (1)	2595 (1)	−2748 (2)
S(2)	−4590 (1)	3013 (1)	−4073 (2)
O	563 (3)	7674 (2)	8287 (4)
N(1)	1367 (4)	3149 (3)	24 (5)
N(2)	−1614 (3)	3962 (3)	−649 (5)
N(3)	1532 (3)	5032 (2)	4168 (4)
N(4)	1918 (3)	8900 (2)	7683 (5)
C(1)	1781 (3)	2919 (3)	−1140 (5)
C(2)	−2868 (4)	3568 (3)	−2075 (5)
C(3)	2853 (4)	4945 (3)	5523 (5)
C(4)	3809 (4)	5786 (3)	7613 (5)
C(5)	3394 (4)	6770 (3)	8357 (5)
C(6)	2029 (3)	6863 (2)	6938 (4)
C(7)	1128 (3)	5992 (2)	4877 (4)
C(8)	1450 (3)	7854 (2)	7689 (4)
C(9)	1311 (4)	9855 (3)	8410 (6)
C(10)	2299 (6)	10573 (4)	10815 (7)
C(11)	3027 (5)	9207 (3)	7085 (7)
C(12)	2256 (6)	9162 (5)	4920 (8)
H(1)	3133 (36)	4311 (29)	4980 (50)
H(2)	4730 (40)	5684 (31)	8523 (54)
H(3)	4042 (40)	7386 (31)	9918 (53)
H(4)	141 (33)	6073 (26)	3910 (45)
H(5)	113 (42)	9453 (33)	7717 (57)
H(6)	1368 (46)	10364 (36)	7580 (63)
H(7)	3403 (51)	11044 (39)	11239 (68)
H(8)	2271 (50)	10019 (40)	11454 (71)
H(9)	1951 (51)	11021 (42)	11264 (70)
H(10)	3647 (49)	8645 (38)	7188 (65)
H(11)	3903 (47)	10089 (37)	8035 (65)
H(12)	1717 (59)	9570 (47)	4896 (78)
H(13)	1654 (54)	8317 (43)	3912 (74)
H(14)	3235 (56)	9415 (44)	4776 (75)

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* A list of observed and calculated structure factors is available from the authors and has also been deposited with the National Lending Library, England, as Supplementary Publication No. SUP 30191 (15 pp., 1 microfiche). Copies may be obtained through the Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

Table 2. *Thermal parameters with e.s.d.'s in parentheses*

The anisotropic thermal parameters are in the form $\exp\,[-0{\cdot}25\,(h^2a^{*2}B_{11}+\ldots 2klb^*c^*B_{23})]$.

	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
Zn	4·620 (17)	3·554 (15)	4·176 (16)	1·631 (13)	2·633 (14)	1·586 (13)
S(1)	5·866 (45)	5·340 (43)	5·204 (42)	1·981 (36)	4·039 (39)	2·363 (36)
S(2)	3·828 (38)	8·438 (67)	5·905 (51)	2·337 (41)	2·352 (37)	3·114 (49)
O	5·031 (101)	3·039 (79)	5·096 (103)	1·765 (74)	3·581 (91)	2·022 (77)
N(1)	5·903 (148)	4·158 (122)	5·829 (151)	2·244 (113)	4·274 (133)	2·234 (114)
N(2)	4·457 (124)	4·643 (128)	4·787 (129)	1·893 (105)	2·248 (109)	2·576 (110)
N(3)	3·539 (94)	2·788 (85)	3·324 (93)	1·279 (74)	2·031 (81)	1·497 (75)
N(4)	4·813 (119)	2·638 (89)	5·228 (127)	1·654 (85)	3·263 (108)	1·947 (90)
C(1)	3·725 (119)	2·917 (107)	4·351 (131)	1·240 (92)	2·218 (108)	1·823 (100)
C(2)	4·669 (139)	4·355 (135)	4·929 (145)	2·417 (115)	3·463 (127)	2·823 (121)
C(3)	3·757 (121)	3·628 (119)	4·250 (131)	1·906 (101)	2·204 (109)	2·008 (106)
C(4)	3·803 (129)	4·579 (141)	4·300 (137)	2·017 (113)	1·879 (114)	2·298 (118)
C(5)	3·781 (123)	3·762 (123)	3·611 (120)	1·310 (101)	1·815 (104)	1·739 (102)
C(6)	3·663 (110)	2·562 (96)	3·557 (111)	1·292 (85)	2·211 (97)	1·562 (87)
C(7)	3·515 (109)	2·758 (98)	3·532 (110)	1·342 (86)	2·124 (96)	1·652 (89)
C(8)	3·845 (117)	2·561 (99)	3·306 (109)	1·316 (89)	1·772 (98)	1·292 (88)
C(9)	6·125 (179)	3·221 (123)	6·693 (193)	2·792 (126)	4·167 (162)	2·637 (130)
C(10)	8·687 (285)	4·994 (190)	6·089 (215)	3·426 (195)	3·966 (212)	1·294 (165)
C(11)	5·931 (184)	4·473 (152)	6·558 (198)	2·016 (137)	4·169 (169)	3·057 (149)
C(12)	8·209 (275)	7·156 (248)	6·373 (227)	3·886 (220)	4·351 (218)	3·470 (201)

Table 2 (*cont.*)

	B
H(1)	3·83 (68)
H(2)	4·38 (74)
H(3)	4·34 (73)
H(4)	2·95 (57)
H(5)	4·93 (80)
H(6)	5·80 (91)
H(7)	6·80 (106)
H(8)	6·91 (107)
H(9)	7·02 (107)
H(10)	6·30 (98)
H(11)	6·09 (94)
H(12)	8·61 (131)
H(13)	7·78 (119)
H(14)	8·29 (128)

through pyridine N and amide O (Fig. 1). Two corners of each tetrahedron are occupied by N atoms of thiocyanate groups and the other two by pyridine N and amide O of two DENA molecules. The tetrahedra are only slightly distorted, as shown by the angles which are in the range 102·7 to 115·4°. Bond distances in the coordination polyhedron are given in Table 3. The Zn–O distance is within the range of values quoted in the literature for tetrahedral zinc, the shortest ones being those involving anionic oxygen: 1·962 (7) Å in bis(dipivaloylmethanido)zinc (Cotton & Wood, 1964), 1·973 (6) and 1·954 (8) Å in bis(thiourea)zinc acetate (Cavalca, Fava, Andreetti & Domiano, 1967), 1·975 (9) Å in tris(thiourea)zinc sulphate (Andreetti, Cavalca & Musatti, 1968), 2·01 (1) Å in dichlorobis-(2,6-lutidine *N*-oxide)zinc (Sager & Watson, 1968), 1·997 (4) and 2·019 (6) Å in dichlorobis(antipyrine)zinc (Biagini Cingi, Guastini, Musatti & Nardelli, 1972). The Zn–N bond distances agree with the tetrahedral values of the literature listed by Baenziger & Schultz (1971). As in the other complexes of DENA, the Zn–N(pyridine) distance is longer than Zn–N(isothiocyanate), which indicates that the bond involving the isothiocyanate is the stronger, probably as a consequence of the electrostatic attracting power of the negative charge of the anionic ligand.

Table 3. *Bond distances* (Å) *and angles* (°)

(1) In the coordination polyhedron.

Zn—N(1)	1·921 (5)	N(1)–Zn—N(2)	115·3 (1)
Zn—N(2)	1·929 (4)	N(1)–Zn—N(3)	112·3 (2)
Zn—N(3)	2·006 (5)	N(1)–Zn—O$^{\rm i}$	103·4 (2)
Zn—O$^{\rm i}$	2·014 (4)	N(2)–Zn—N(3)	115·4 (1)
		N(2)–Zn—O$^{\rm i}$	105·8 (2)
		N(3)–Zn—O$^{\rm i}$	102·7 (1)

(2) In the thiocyanate anions.

N(1)–C(1)	1·151 (6)	N(1)–C(1)–S(1)	179·4 (4)
C(1)–S(1)	1·606 (5)	N(2)–C(2)–S(2)	179·0 (4)
N(2)–C(2)	1·156 (5)	Zn—N(1)–C(1)	159·2 (3)
C(2)–S(2)	1·597 (5)	Zn—N(2)–C(2)	149·2 (4)

Asymmetric units:

i	$-x$	$1-y$	$1-z$
ii	$-x$	$1-y$	$-z$

The most interesting feature of the structure is perhaps the dimeric nature of the complex with bridging ligands.

Another interesting point is the transformation of the compound (III) into the dimeric compound (IV). This corresponds to a change of coordination around zinc from the less stable octahedral to the more stable tetrahedral and a change of behaviour of the ligand from terminal to bridging. In the cadmium complex, which shows the same stoichiometry and the same molecular volume as the zinc compound [809·7 Å^3 for 2 Cd(DENA)$\mathrm{(SCN)_2}$] coordination is octahedral, which is the more stable for cadmium.

The isothiocyanate groups are linear as expected and the C–N and C–S bond distances agree well with those generally found (Bigoli, Braibanti, Pellinghelli & Tiripicchio, 1972).

The DENA molecule does not show notable variations in bond distances and angles (Table 4 and Fig. 2) when compared with those found in the other complexes.

The pyridine ring is perfectly planar (Table 5) and does not show the small distortions from planarity observed in cadmium and manganese complexes. The whole amide group is not perfectly planar; the bonds radiating from C(8) lie in one plane but the bonds from nitrogen N(4) are not exactly in one plane. However, the angle between the plane through C(8) and the average plane through N(4) is only 1·7°. The DENA molecule shows only small changes in the different complexes, whether it is terminal or acting as a bridge. The different packings do not seem to affect the structure of DENA. The DENA molecules in the different complexes are represented in Fig. 3. The greater rotation angle of the pyridine ring around $\mathrm{C(6)_{ring}}$–$\mathrm{C(8)_{amide}}$ is observed for the dimeric complexes (85·4°); this is probably due to the repulsion between the pyridine rings facing each other in the dimer (the shortest inter-ring contacts are N(3)···C(7) = 3·369 Å). The distance between the donor atoms of the organic molecule, pyridine N and amide O, is 4·404 Å in the present compound, the shortest of all the complexes.

Table 4. *Bond distances* (Å) *and angles* (°) *in the organic molecule*

N(3)—C(3)	1·343 (5)
N(3)—C(7)	1·357 (4)
C(3)—C(4)	1·383 (5)
C(4)—C(5)	1·395 (6)
C(5)—C(6)	1·394 (5)
C(6)—C(7)	1·377 (4)
C(6)—C(8)	1·515 (5)
C(8)—O	1·234 (5)
C(8)—N(4)	1·328 (4)
N(4)—C(9)	1·496 (6)
C(9)—C(10)	1·524 (6)
N(4)—C(11)	1·476 (8)
C(11)-C(12)	1·486 (8)
C(3)—H(1)	0·92 (4)
C(4)—H(2)	0·95 (4)
C(5)—H(3)	1·03 (3)
C(7)—H(4)	1·00 (3)
C(9)—H(5)	1·07 (4)
C(9)—H(6)	1·05 (6)
C(10)-H(7)	1·06 (5)
C(10)-H(8)	0·98 (6)
C(10)-H(9)	0·78 (6)
C(11)-H(10)	1·01 (5)
C(11)-H(11)	1·09 (4)
C(12)-H(12)	0·80 (6)
C(12)-H(13)	0·98 (4)
C(12)-H(14)	1·09 (7)
C(7)—N(3)—C(3)	118·5 (3)
N(3)—C(3)—C(4)	122·7 (4)
C(3)—C(4)—C(5)	119·0 (4)
C(4)—C(5)—C(6)	118·0 (3)
C(5)—C(6)—C(7)	120·0 (3)
C(5)—C(6)—C(8)	120·6 (3)
C(7)—C(6)—C(8)	119·2 (3)
C(6)—C(7)—N(3)	121·7 (3)
C(6)—C(8)—O	119·7 (3)
C(6)—C(8)—N(4)	118·6 (3)
O——C(8)—N(4)	121·8 (3)
C(8)—N(4)—C(9)	117·2 (3)
C(8)—N(4)—C(11)	125·2 (3)
C(9)—N(4)—C(11)	117·5 (3)
N(4)—C(9)—C(10)	110·7 (4)
N(4)—C(11)-C(12)	111·9 (5)
N(3)—C(3)—H(1)	116·8 (21)
C(4)—C(3)—H(1)	120·3 (21)
C(3)—C(4)—H(2)	119·9 (23)
C(5)—C(4)—H(2)	121·0 (23)
C(4)—C(5)—H(3)	121·2 (24)
C(6)—C(5)—H(3)	120·7 (24)
C(6)—C(7)—H(4)	118·0 (18)
N(3)—C(7)—H(4)	120·3 (18)
N(4)—C(9)—H(5)	108·3 (23)
N(4)—C(9)—H(6)	101·6 (27)
C(10)-C(9)—H(5)	117·5 (21)
C(10)-C(9)—H(6)	113·2 (23)
H(5)—C(9)—H(6)	104·2 (35)
C(9)—C(10)-H(7)	108·8 (24)
C(9)—C(10)-H(8)	107·9 (28)
C(9)—C(10)-H(9)	113·3 (34)
H(7)—C(10)-H(8)	115·2 (43)
H(7)—C(10)-H(9)	110·0 (50)
H(8)—C(10)-H(9)	101·6 (51)
N(4)—C(11)-H(10)	109·4 (31)
N(4)—C(11)-H(11)	118·9 (27)
C(12)-C(11)-H(10)	110·6 (24)
C(12)-C(11)-H(11)	102·5 (24)
H(10)-C(11)-H(11)	103·0 (40)
C(11)-C(12)-H(12)	106·2 (37)
C(11)-C(12)-H(13)	107·1 (30)
C(11)-C(12)-H(14)	101·2 (26)
H(12)-C(12)-H(13)	111·7 (55)
H(12)-C(12)-H(14)	121·3 (56)
H(13)-C(12)-H(14)	108·0 (46)

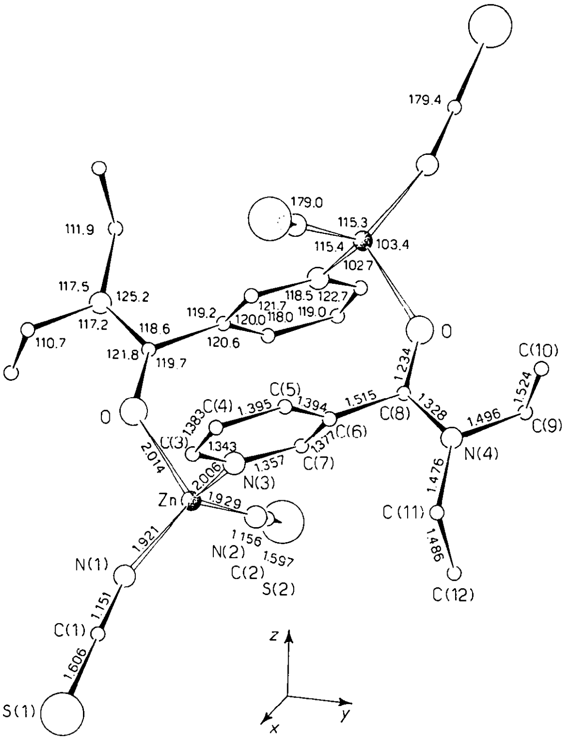


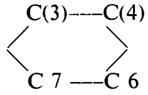
Fig. 2. Bond distances and angles in the complex.

Table 5. *Analysis of planarity of groups of atoms*

The plane of best fit is defined by atoms marked with an asterisk. X, Y, Z are orthogonal coordinates in Å.†

(1) Equation of the plane through C(3), C(4), C(6), C(7) of the pyridine ring $0{\cdot}7501X+0{\cdot}6030Y-0{\cdot}2717Z=2{\cdot}2607$.

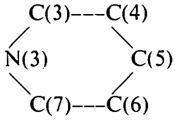
	$\Delta\,.\,10^4$ (Å)	$\sigma\,.\,10^4$ (Å)
C(3)*	9	43
C(4)*	−8	43
C(6)*	5	31
C(7)*	−5	31
N(3)	−56	31
C(5)	−69	43



$\sum_4(\Delta/\sigma)^2=0{\cdot}13$

(2) Equation of the plane through the pyridine ring $+0{\cdot}7504X+0{\cdot}6025Y-0{\cdot}2719Z=2{\cdot}2555$.

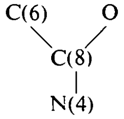
	$\Delta\,.\,10^4$ (Å)	$\sigma\,.\,10^4$ (Å)
N(3)*	−31	31
C(3)*	37	43
C(4)*	17	43
C(5)*	−52	43
C(6)*	17	31
C(7)*	12	31



$\sum_6(\Delta/\sigma)^2=3{\cdot}81$

(3) Equation of the plane through the carbonyl carbon [C(8)] $-0{\cdot}3334X+0{\cdot}1277Y-0{\cdot}9341Z=-3{\cdot}0280$.

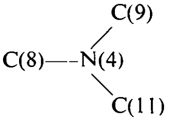
	$\Delta\,.\,10^4$ (Å)	$\sigma\,.\,10^4$ (Å)
C(6)*	0	26
C(8)*	0	26
O*	0	26
N(4)*	0	32



$\sum_4(\Delta/\sigma)^2=0{\cdot}0$

(4) Equation of the plane through the amide nitrogen [N(4)] $-0{\cdot}3047X+0{\cdot}1273Y-0{\cdot}9439Z=-3{\cdot}1205$

	$\Delta\,.\,10^4$ (Å)	$\sigma\,.\,10^4$ (Å)
C(8)*	−21	26
N(4)*	113	32
C(9)*	−36	39
C(11)*	−56	46



$\sum_4(\Delta/\sigma)^2=15{\cdot}46$

† Transformation matrix from fractional to orthogonal coordinates $\begin{pmatrix} a\sin\gamma & 0 & -c\sin\alpha\cos\beta^* \\ a\cos\gamma & b & c\cos\alpha \\ 0 & 0 & c\sin\alpha\sin\beta^* \end{pmatrix}$.

Packing of the dimers is determined by van der Waals interactions only, unlike the other complexes where the polyhedra are held together in three-dimensional networks by bridges of thiocyanate anions and DENA molecules or by hydrogen bonds. The shortest contacts are given in Table 6.

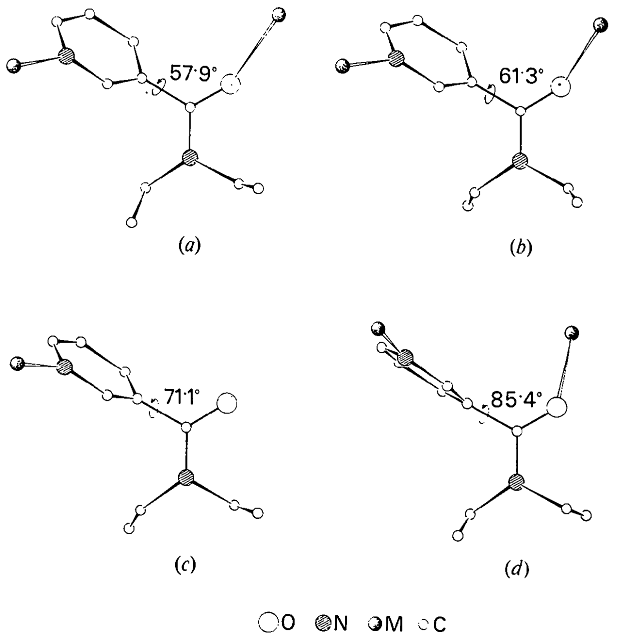


Fig. 3. Projection of the DENA molecule in the plane of the amide group in different complexes, (*a*) Cd(DENA) $(NCS)_2$, (*b*) $Mn(DENA)_2(NCS)_2$, (*c*) $Zn(DENA)_2(NCS)_2(H_2O)_2$, (*d*) $Zn_2(DENA)_2(NCS)_4$.

Table 6. *Shortest intermolecular distances* (Å)

$N(2)\cdots N(1^{ii})$	3·426 (6)
$N(2)\cdots N(2^{ii})$	3·438 (7)
$N(3)\cdots N(2^{ii})$	3·397 (6)
$C(7)\cdots N(2^{ii})$	3·588 (6)
$C(2)\cdots N(3^{ii})$	3·297 (7)
$C(7)\cdots C(2^{ii})$	3·536 (7)
$N(1)\cdots O^{i}$	3·089 (7)
$N(2)\cdots O^{i}$	3·145 (6)
$N(3)\cdots O^{i}$	3·140 (7)
$C(5)\cdots N(2^{i})$	3·329 (7)
$C(6)\cdots N(2^{i})$	3·550 (6)
$C(7)\cdots N(3^{i})$	3·369 (6)
$C(5)\cdots C(2^{i})$	3·336 (7)
$C(7)\cdots C(7^{i})$	3·280 (6)

Asymmetric units:

i	$-x$	$1-y$	$1-z$
ii	$-x$	$1-y$	$-z$

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