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The Crystal and Molecular Structure of Bis(thiocarbohydrazide-N,S)cadmium Dichloride

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The crystals of bis(thiocarbohydrazide–N,S)cadmium dichloride, $Cd(S{=}C(NH{-}NH_2)_2)_2Cl_2$, belong to monoclinic space group $P2_1/c$. The structure of the compound, determined from three-dimensional data, consists of trans*–octahedral complexes where the ligand molecule forms pentatomic chelate rings with N,S as donor atoms. The chlorine atoms lie along the normal to the plane containing metal and organic molecules. The distances Cd–N = 2.34(3) and Cd–S = 2.59(1) Å are slightly shorter than those found in non-chelate cadmium complexes; Cd–Cl = 2.73(1) Å is very close to the sum of ionic radii. The configuration of the ligand, with one $-NH_2$ group turned towards S=C, repeats that of crystallized thiocarbohydrazide. Some differences, however, in distances, planarity, and angles between complexed or free ligand can be detected. The complexes are held together in layers parallel to (102) by weak hydrogen bonds NH . . . Cl.*

Introduction

Thiocarbohydrazide, $S{=}C(NH{-}NH_2)_2$, forms complexes which can be assigned structures with pentatomic chelate rings with S,N as donor atoms. Such structures have been assumed to interpret IR spectra[1] and equilibria in solution.[2] In order to confirm these assumptions, the crystal structure of bis(thiocarbohydrazide–N,S)cadmium dichloride has now been determined.

Experimental Section

Preparation. Crystals of the compound, $Cd(SC(NH{-}NH_2)_2)_2Cl_2$ have been obtained by evaporation of aqueous solutions of thiocarbohydrazide and cadmium chloride in stoichiometric ratio. Small, colorless crystals were obtained.

Crystal Data. Compound: bis(thiocarbohydrazide–N,S)cadmium dichloride, $Cd(SC(NH{-}NH_2)_2)_2Cl_2$, F.W. 395.62.

Crystal class: Monoclinic prismatic.

Unit cell (radiation CuKα, $\bar{\lambda}=1.5418$ Å, from rotation and Weissenberg photographs around [100] and [010]):

$a=8.64(1)$, $b=5.78(1)$, $c=13.78(1)$ Å;

$\beta=119.5(3)°$, $V=599.3$ Å^3, $Z=2$;

$D_x=2.19$, $D_m=2.17$ g.cm^{-3};

$\mu(CuK\alpha)=224.7$ cm^{-1};

Space group $P2_1/c$ (C_{2h} (5), No. 14).

Intensity Data. Intensities of reflections $0kl, \ldots, 7kl$, *and* $h0l, \ldots, h5l$ were recorded on integrating Weissenberg camera and then measured by a microdensitometer (934 independent reflections out of 1369 possible).

Calculations. Usual corrections were applied. Absorption corrections as for cylindrical specimens were introduced ($\mu R_{[100]}=1.01$, $\mu R_{[010]}=0.67$). Atomic form factors according to Cromer and Mann[3] were used.

Table I. Fractional atomic coordinates (with e.s.d's ×10⁴).

	x	*y*	*z*
Cd	1.0000	.0000	.0000
S	.6777(8)	—.1555(5)	—.0947(5)
Cl	.9028(8)	.2939(6)	—.1755(5)
N(1)	.6763(27)	.2649(17)	—.0094(18)
N(2)	.8578(34)	.2493(22)	.0655(19)
N(3)	.4141(24)	.1321(16)	—.1499(17)
N(4)	.3324(34)	.3398(26)	—.1450(26)
C	.5812(23)	.1021(24)	—.0864(17)

Table II. Anisotropic thermal parameters[a] (Å²).

	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
Cd	2.825	3.130	3.248	0.131	1.197	0.042
S	2.951	2.898	3.361	—0.004	1.365	—0.139
Cl	3.067	3.076	3.153	—0.023	1.074	0.345
N(1)	2.561	3.378	3.249	0.109	0.657	—0.143
N(2)	3.663	3.046	3.767	—0.398	1.594	—0.788
N(3)	3.604	2.758	3.919	—0.100	1.550	0.283
N(4)	2.445	3.951	4.252	—0.026	0.977	0.128
C	2.800	3.166	2.987	—0.476	1.022	0.126

[a] In the last cycle for all the atoms the average shift, $|\Delta B_{ij}|_{av}$, and the maximum shift, $|\Delta B_{ij}|_{max}$, were:

$|\Delta B_{ij}|_{av} = 0.049$ $\quad$ $|\Delta B_{ij}|_{max} = 0.173$

(1) G. R. Burns, *Inorg. Chem.* 7, 277 (1968).
(2) A. Braibanti, F. Dallavalle and E. Leporati, *Inorg. Chim. Acta*, 3, 459 (1969).

Table III. Observed and calculated structure factors

= after F_o values indicates unobserved reflections.

h	k	l	$10F_o$	$10F_c$	h	k	l	$10F_o$	$10F_c$	h	k	l	$10F_o$	$10F_c$	h	k	l	$10F_o$	$10F_c$	h	k	l	$10F_o$	$10F_c$	h	k	l	$10F_o$	$10F_c$
0	2	0	272	-239	1	1	1	390	403	1	0	8	289	329	2	1	-2	656	-711	2	3	-9	185	158	3	5	-3	220	201	3	0	12	83	112
0	4	0	365	337	1	1	-1	668	655	1	0	-8	80-	-53	2	2	2	850	876	2	4	9	163	-123	3	6	3	111	99	3	0	-12	644	682
0	6	0	275	287	1	2	1	458	-468	1	1	8	176	-190	2	2	-2	801	760	2	4	-9	159	-172	3	6	-3	80-	60	3	1	12	56-	3
0	1	1	1056	1170	1	2	-1	155	-101	1	1	-8	187	-148	2	3	2	122	101	2	5	9	122	124	3	7	-3	77	108	3	1	-12	271	290
0	2	1	325	-351	1	3	1	759	831	1	2	8	456	519	2	3	-2	102	63	2	5	-9	222	244	3	0	4	530	566	3	2	12	36-	143
0	3	1	371	440	1	3	-1	657	720	1	2	-8	525	619	2	4	2	259	242	2	6	-9	62	39	3	0	-4	136	160	3	2	-12	146	131
0	4	1	301	332	1	4	1	271	273	1	3	8	111-	-16	2	4	-2	459	433	2	0	10	197	220	3	1	4	144	-119	3	3	-12	100-	-36
0	5	2	193	168	1	4	-1	97	98	1	3	-8	127	104	2	5	2	163	-124	2	0	-10	161	172	3	1	-4	144	-126	3	4	-12	172	153
0	6	1	100-	-116	1	5	1	361	336	1	4	8	108-	-2	2	5	-2	100-	30	2	1	10	131	-107	3	2	4	210	168	3	5	-12	51-	-55
0	7	1	701	744	1	5	-1	272	214	1	4	-8	299	359	2	6	2	185	174	2	1	-10	159	115	3	2	-4	904	917	3	1	-13	195	190
0	0	2	987	1082	1	6	1	99-	-85	1	5	8	77-	68	2	6	-2	191	183	2	2	10	207	157	3	3	4	101	-88	3	2	-13	119-	83
0	1	2	265	341	1	6	-1	101-	62	1	5	-8	121	-72	2	7	2	33-	14	2	2	-10	427	475	3	3	-4	265	233	3	3	-13	285	259
0	2	2	537	630	1	7	1	63-	53	1	6	8	175	189	2	7	-2	40-	-7	2	3	10	52-	36	3	4	4	510	528	3	4	-13	76-	-37
0	3	2	115	-13	1	7	-1	133	204	1	6	-8	68-	67	2	1	3	797	819	2	3	-10	92-	-17	3	4	-4	424	378	3	5	-13	28-	24
0	4	2	365	409	1	0	2	163	105	1	1	9	385	403	2	1	-3	436	460	2	4	10	251	224	3	5	4	129	111	3	0	-14	220	211
0	5	2	111	67	1	0	-2	848	895	1	1	-9	301	337	2	2	3	355	356	2	4	-10	166	171	3	5	-4	205	-183	3	1	-14	101-	-37
0	6	2	211	176	1	1	2	169	170	1	2	9	131-	-29	2	2	-3	203	-190	2	5	10	89	-57	3	6	4	97	70	3	2	-14	161	147
0	7	2	63-	-19	1	1	-2	44-	-35	1	2	-9	124-	-65	2	3	3	240	215	2	6	-10	52	79	3	6	-4	77-	51	3	3	-14	80-	80
0	1	3	755	796	1	2	2	904	959	1	3	9	109-	126	2	3	-3	697	683	2	1	11	173	144	3	7	-4	41-	30	3	4	-14	126	144
0	2	3	207	-208	1	2	-2	715	753	1	3	-9	783	304	2	4	3	293	-263	2	1	-11	244	235	3	1	5	172	205	3	1	-15	153	116
0	3	3	360	386	1	3	2	232	-232	1	4	9	101-	-13	2	4	-3	141	-101	2	2	11	48-	19	3	1	-5	594	657	3	2	-15	94-	66
0	4	3	204	165	1	3	-2	169	117	1	4	-9	91-	8	2	5	3	284	285	2	2	-11	114-	9	3	2	5	208	-161	3	3	-15	158	174
0	5	3	443	449	1	4	2	612	434	1	5	9	166	178	2	5	-3	371	377	2	3	11	284	239	3	2	-5	264	-244	3	0	-16	57-	-29
0	6	3	97-	35	1	4	-2	311	305	1	5	-9	235	272	2	6	3	100	95	2	3	-11	319	317	3	3	5	566	568	3	1	-16	70-	-31
0	7	3	60-	45	1	5	2	95-	67	1	6	-9	55-	-2	2	6	-3	186	179	2	4	11	60-	-19	3	3	-5	673	668	3	2	-16	184	210
0	0	4	34-	62	1	5	-2	124	103	1	0	10	321	329	3	7	3	20-	135	2	4	-11	82-	22	3	4	5	147	106	3	1	-17	79	99
0	1	4	786	-840	1	6	2	102	107	1	0	-10	181	137	2	7	-3	60	34	2	5	-11	104	142	3	4	-5	91	84	4	0	0	92	-68
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0	5	4	100-	72	1	1	3	513	623	1	2	-10	309	388	2	1	-4	638	-692	2	1	-12	100	71	3	6	-5	76-	-27	4	4	0	331	271
0	6	4	92-	68	1	1	-3	1234	1337	1	3	10	107-	59	2	2	4	134	125	2	2	12	219	176	3	7	-5	36-	27	4	5	0	168	152
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0	2	5	94-	90	1	3	3	493	537	1	4	-10	249	240	2	3	-4	67-	-18	2	3	-12	82	-58	3	1	6	150	125	4	1	-1	617	647
0	3	5	745	849	1	3	-3	98-	-10	1	5	10	51-	55	2	4	4	250	238	2	4	-12	296	277	3	1	-6	80-	-59	4	2	1	133	-104
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0	6	5	87-	86	1	5	3	321	349	1	1	-11	215	226	2	5	-4	195	174	2	1	-13	151	138	3	3	6	168	-155	4	3	-1	331	301
0	7	5	73-	122	1	5	-3	612	418	1	2	11	119-	-60	2	6	4	210	239	2	2	13	61-	31	3	3	-6	219	-174	4	4	1	61-	-27
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0	1	6	70-	57	1	6	-3	97-	11	1	3	11	291	304	2	7	-4	37-	-37	2	3	-13	304	288	3	4	-6	451	442	4	5	1	328	322
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0	6	6	82-	121	1	1	4	84	86	1	0	12	87-	15	2	3	5	361	364	2	3	-14	65-	15	3	1	7	306	298	4	0	-2	1032	1112
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0	5	7	133	155	1	3	-4	211	-216	1	2	-12	82-	60	2	5	-5	386	393	2	0	-16	77	85	3	3	-7	646	674	4	3	2	94-	-10
0	6	7	100	-119	1	4	4	363	393	1	3	12	85-	-72	2	6	5	47-	-14	2	1	-16	55-	11	3	4	7	80-	-63	4	3	-2	94	-52
0	0	8	269	282	1	4	-4	97-	-61	1	3	-12	97-	-21	2	6	-5	95	-82	2	2	-16	122	146	3	4	-7	193	146	4	4	2	331	302
0	1	8	385	428	1	5	4	222	-222	1	4	12	147	173	2	7	-5	52-	107	3	0	0	1102	1136	3	5	7	181	183	4	4	-2	365	282
0	2	8	425	512	1	5	-4	86-	12	1	4	-12	181	182	2	0	6	539	591	3	1	0	682	628	3	5	-7	131	98	4	5	2	99	68
0	3	8	108-	-65	1	6	4	126	140	1	5	-12	58	73	2	0	-6	783	822	3	2	0	552	671	3	6	-7	68-	63	4	5	-2	77-	46
0	4	8	159	157	1	6	-4	252	314	1	1	13	112	108	2	1	6	52-	39	3	3	0	235	-175	3	0	8	351	367	4	6	2	105	98
0	5	8	171	-123	1	7	4	60-	29	1	1	-13	183	213	2	1	-6	155	123	3	4	0	169	156	3	0	-8	797	882	4	6	-2	277	262
0	6	8	124	151	1	7	-4	57-	-2	1	2	13	86-	-16	2	2	6	296	315	3	5	0	70	36	3	1	8	100-	-21	4	1	3	289	287
0	1	9	410	469	1	1	5	567	591	1	2	-13	115-	22	2	2	-6	383	436	3	6	0	268	295	3	1	-8	506	-568	4	1	-3	695	711
0	2	9	131-	128	1	1	-5	797	854	1	3	13	112	143	2	3	6	92-	36	3	7	0	38-	16	3	2	8	233	219	4	2	3	37-	21
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0	4	9	118	-122	1	2	-5	98-	33	1	4	-13	65-	27	2	4	6	211	221	3	1	-1	657	654	3	3	8	133	97	4	3	3	904	904
0	5	9	244	251	1	3	5	409	469	1	0	14	52-	28	2	4	-6	287	255	3	2	1	272	-204	3	3	-8	82-	68	4	3	-3	316	288
0	6	9	40-	14	1	3	-5	350	360	1	0	-14	168	199	2	5	6	63-	49	3	2	-1	178	191	3	4	8	163	156	4	4	3	107	-89
0	0	10	399	443	1	4	5	139	-139	1	1	14	58-	12	2	5	-6	66-	-14	3	3	1	92-	70	3	4	-8	360	403	4	4	-3	60-	-47
0	1	10	109-	-68	1	4	-5	137	83	1	1	-14	92-	-38	2	6	6	151	160	3	3	-1	617	565	3	5	8	105	-99	4	5	3	67-	69
0	2	10	222	225	1	5	5	97	104	1	2	14	34-	198	2	6	-6	235	255	3	4	1	179	130	3	5	-8	89	94	4	5	-3	233	184
0	3	10	109-	29	1	5	-5	186	191	1	2	-14	124	148	2	7	-6	23-	5	3	4	-1	62-	7	3	6	-8	109	150	4	6	3	52-	38
0	4	10	183	151	1	6	5	82-	-41	1	3	-14	67-	33	2	1	7	357	367	3	5	1	394	385	3	1	9	276	270	4	6	-3	95	71
0	5	10	67-	-21	1	6	-5	92-	-136	1	1	-15	186	197	2	1	-7	681	719	3	5	-1	173	144	3	1	-9	367	443	4	7	-3	13-	221
0	1	11	287	323	1	7	-5	140	164	1	2	-15	72-	56	2	2	7	198	-176	3	6	1	73-	2	3	2	9	129	135	4	0	4	486	536
0	2	11	129-	-21	1	0	6	696	727	1	0	-16	96	141	2	2	-7	163	118	3	6	-1	131	-106	3	2	-9	107	79	4	0	-4	824	885
0	3	11	173	154	1	0	-6	873	928	1	1	-16	36-	151	2	3	7	225	212	3	7	1	26-	122	3	3	9	227	184	4	1	4	373	417
0	4	11	89-	-31	1	1	6	271	313	2	0	0	555	596	2	3	-7	250	256	3	7	-1	184	206	3	3	-9	217	201	4	1	-4	126	-129
0	5	11	68-	56	1	1	-6	204	247	2	1	0	923	972	2	4	7	178	165	3	0	2	569	651	3	4	9	122	-92	4	2	4	287	290
0	0	12	451	470	1	2	6	306	362	2	2	0	838	787	2	4	-7	51-	-4	3	0	-2	1074	1157	3	4	-9	94	-89	4	2	-4	458	429
0	1	12	92-	14	1	2	-6	323	389	2	3	0	188	-168	2	5	7	268	272	3	1	2	195	-221	3	5	-9	230	238	4	3	4	133	-116
0	2	12	119-	68	1	3	6	104-	-95	2	4	0	443	428	2	5	-7	137	116	3	1	-2	58-	6	3	6	-9	54-	87	4	3	-4	146	169
0	3	12	131	-110	1	3	-6	166	-125	2	5	0	210	-152	2	6	7	28-	-21	3	2	2	553	517	3	0	10	431	424	4	4	4	223	230
0	4	12	94	122	1	4	6	225	235	2	6	0	163	133	2	6	-7	54-	-10	3	2	-2	371	350	3	0	-10	355	378	4	4	-4	225	190
0	1	13	227	201	1	4	-6	321	328	2	7	0	55	63	2	0	8	131	-106	3	3	2	90-	64	3	1	10	119	94	4	5	4	63-	-69
0	2	13	154	-118	1	5	6	94-	-63	2	1	1	935	956	2	0	-8	874	902	3	3	-2	267	213	3	1	-10	72-	-30	4	5	-4	101	-83
0	3	13	111	116	1	5	-6	100-	-55	2	1	-1	510	500	2	1	8	286	-267	3	4	2	247	224	3	2	10	100-	62	4	6	4	30-	101
0	4	13	40-	112	1	6	6	171	194	2	2	1	303	260	2	1	-8	171	-149	3	4	-2	500	450	3	2	-10	299	360	4	6	-4	276	253
0	0	14	118	91	1	6	-6	144	191	2	2	-1	140	-118	2	2	8	476	462	3	5	2	69	76	3	3	10	68-	19	4	1	5	436	429
0	1	14	79-	10	1	7	-6	30-	47	2	3	1	303	273	2	2	-8	269	336	3	5	-2	210	-169	3	3	-10	106-	64	4	1	-5	555	606
0	2	14	173	174	1	1	7	547	631	2	3	-1	966	953	2	3	8	179	127	3	6	2	184	208	3	4	10	38-	126	4	2	5	115	72
0	3	14	64-	-18	1	1	-7	336	373	2	4	1	80-	-68	2	3	-8	239	270	3	6	-2	203	184	3	4	-10	153	150	4	2	-5	283	274
0	1	15	54-	92	1	2	7	121-	87	2	4	-1	70-	-36	2	4	8	316	271	3	7	-2	64-	17	3	3	-10	72-	-1	4	3	5	172	160
0	2	15	28-	12	1	2	-7	213	230	2	5	1	410	377	2	4	-8	158	108	3	1	3	650	687	3	4	10	107	142	4	3	-5	282	256
1	0	0	626	673	1	3	7	108-	75	2	5	-1	126	78	2	5	8	56-	-26	3	1	-3	678	716	3	1	11	183	196	4	4	5	67-	51
1	1	0	111	112	1	3	-7	546	606	2	6	1	140	-136	2	5	-8	156	-159	3	2	3	92	-67	3	1	-11	600	655	4	6	-5	136	-96
1	2	0	418	457	1	4	7	118	97	2	6	-1	80-	2	2	6	8	181	220	3	2	-3	623	625	3	2	11	79-	-15	4	5	5	147	149
1	3	0	383	-379	1	4	-7	227	-224	2	7	1	47	65	2	1	9	208	150	3	3	3	414	403	3	2	-11	126-	90	4	5	-5	402	372
1	4	0	660	723	1	5	7	212	262	2	7	-1	140	150	2	1	-9	451	529	3	3	-3	604	551	3	3	11	67	73	4	6	-5	60-	-68
1	5	0	335	299	1	5	-7	154	132	2	0	2	329	354	2	2	9	121	79	3	4	3	104-	-26	3	3	-11	104-	19	4	0	6	205	198
1	6	0	153	130	1	6	7	60-	-70	2	0	-2	530	570	2	2	-9	195	221	3	4	-3	393	-349	3	4	-11	99-	63	4	0	-6	377	435
1	7	0	65-	-67	1	6	-7	77-	38	2	1	2	62-	83	2	3	9	402	364	3	5	3	190	167	3	5	-11	181	200	4	1	6	296	277

Table III. (cont.)

h	k	l	$10F_o$	$10F_c$	h	k	l	$10F_o$	$10F_c$	h	k	l	$10F_o$	$10F_c$	h	k	l	$10F_o$	$10F_c$	h	k	l	$10F_o$	$10F_c$	h	k	l	$10F_o$	$10F_c$
4	1	-6	693	549	5	4	1	243	-199	5	0	10	16-	83	6	3	-5	486	412	7	4	2	206	228	8	3	-1	244	229	9	2	-3	121-	13
4	2	6	422	381	5	4	-1	143	133	5	0	-10	86-	129	6	4	5	51-	15	7	4	-2	158	113	8	4	-1	77-	-21	9	3	-3	156	153
4	2	-6	673	515	5	5	1	203	220	5	1	-10	57-	5	6	4	-5	112-	-73	7	5	-2	65-	78	8	0	2	218	231	9	4	-3	31-	1
4	3	6	70-	-25	5	5	-1	208	197	5	2	-10	395	408	6	5	-5	156	153	7	1	3	149	113	8	0	-2	89	66	9	0	-4	82-	69
4	3	-6	110	-82	5	6	1	44-	19	5	3	-10	136	132	6	6	-5	80	93	7	1	-3	351	349	8	1	2	87-	105	9	1	-4	102-	75
4	4	6	129	86	5	6	-1	77	-57	5	4	-10	181	192	6	0	6	84-	48	7	2	3	51-	-38	8	1	-2	116-	39	9	2	-4	127-	180
4	4	-6	409	350	5	0	2	409	429	5	5	-10	102	-78	6	0	-6	176	146	7	2	-3	163	-162	8	2	2	101-	100	9	3	-4	80-	-31
4	5	6	45-	-43	5	0	-2	457	521	5	6	-10	26-	107	6	1	6	115	-78	7	3	3	311	292	8	2	-2	386	362	9	4	-4	208	214
4	5	-6	107	-69	5	1	2	76	-60	5	1	-11	239	263	6	1	-6	153	-161	7	3	-3	195	180	8	3	2	56-	-20	9	1	-5	107-	85
4	6	-6	95	93	5	1	-2	80	-76	5	2	-11	207	177	6	2	6	215	262	7	4	3	50	74	8	3	-2	95-	-29	9	2	-5	131-	-67
4	1	7	370	344	5	2	2	361	324	5	3	-11	257	261	6	2	-6	508	513	7	4	-3	87-	88	8	4	-2	137	134	9	3	-5	321	311
4	1	-7	372	413	5	2	-2	617	579	5	4	-11	99	-77	6	3	6	76	45	7	5	-3	95	129	8	1	3	169	183	9	4	-5	65-	36
4	2	7	151	142	5	3	2	237	227	5	5	-11	190	186	6	3	-6	105-	-44	7	0	4	55-	16	8	1	3	211	236	9	0	-6	181	202
4	2	-7	191	185	5	3	-2	101	-89	5	0	-12	92-	-36	6	4	-6	262	240	7	0	-4	586	607	8	2	3	79-	31	9	1	-6	107-	-169
4	3	7	84-	28	5	4	2	204	190	5	1	-12	124	86	6	5	-6	99	80	7	1	4	183	156	8	2	-3	146-	-112	9	2	-6	131-	153
4	3	-7	560	534	5	4	-2	637	389	5	2	-12	399	433	6	6	-6	60	55	7	1	-4	169	185	8	3	-3	225	221	9	3	-6	86-	97
4	4	7	77	-73	5	5	2	169	-145	5	3	-12	80-	-47	6	1	7	115	97	7	2	4	262	257	8	4	-3	90-	116	9	4	-6	146	167
4	4	-7	68-	23	5	5	-2	72-	36	5	4	-12	140	131	6	1	-7	333	356	7	2	-4	101	85	8	5	-3	16-	101	9	1	-7	219	210
4	5	7	20-	202	5	6	2	30-	157	5	5	-12	68-	17	6	2	7	97	87	7	3	4	52-	-20	8	0	6	92	137	9	2	-7	136-	-9
4	5	-7	126	109	5	6	-2	86	79	5	1	-13	200	218	6	2	-7	255	-267	7	3	-4	60	-50	8	0	-6	386	426	9	3	-7	143	136
4	6	-7	151	-136	5	1	3	329	322	5	2	-13	179	-166	6	3	-7	314	292	7	4	-4	212	215	8	1	6	51-	-15	9	4	-7	68-	-7
4	0	8	346	340	5	1	-3	294	290	5	3	-13	227	200	6	4	-7	165	148	7	5	-4	51-	-38	8	1	-6	119-	-66	9	0	-8	230	247
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4	1	8	150	-128	5	2	-3	195	-168	5	5	-13	28-	66	6	6	-7	51-	-23	7	1	-5	233	245	8	3	-6	102-	-59	9	2	-8	131-	207
4	1	-8	166	181	5	3	3	291	281	5	0	-14	271	252	6	0	8	158	219	7	2	5	52	68	8	4	-6	97-	98	9	3	-8	86-	16
4	2	8	148	117	5	3	-3	742	660	5	1	-14	133	-93	6	0	-8	508	561	7	2	-5	268	257	8	5	-6	35-	68	9	4	-8	68-	62
4	2	-8	249	262	5	4	3	92	-71	5	2	-14	187	137	6	1	8	65-	78	7	3	-5	357	314	8	1	-5	333	348	9	1	-9	277	282
4	3	8	61-	39	5	4	-3	90-	18	5	3	-14	83-	-30	6	1	-8	101-	-11	7	4	-5	161	-170	8	2	-5	151-	-66	9	2	-9	131-	-60
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4	4	8	126	124	5	5	-3	105	87	5	1	-15	163	154	6	3	-8	148	-127	7	0	6	101	120	8	4	-5	97-	-28	9	4	-9	65-	46
4	4	-8	457	425	5	6	-3	67	58	5	2	-15	101	-86	6	4	-8	211	179	7	0	-6	119	104	8	5	-5	37-	61	9	0	-10	360	378
4	5	-8	80-	-17	5	0	4	402	436	5	3	-15	143	132	6	5	-8	126	88	7	1	6	47-	-17	8	0	-6	287	301	9	1	-10	106-	23
4	4	-8	34-	22	5	0	-4	532	576	5	0	-16	155	160	6	6	-8	162	136	7	1	-6	118	98	8	1	-6	168	-190	9	2	-10	127-	34
4	1	9	134	150	5	1	4	121	113	5	1	-16	115	-127	6	1	-9	355	381	7	2	-6	399	405	8	2	-6	261	273	9	3	-10	80-	-29
4	1	-9	319	359	5	1	-4	589	-606	5	2	-16	163	136	6	2	-9	62-	-66	7	3	-6	82	60	8	3	-6	107-	61	9	4	-10	96	100
4	2	9	87	-69	5	2	4	211	206	5	3	-16	45-	29	6	3	-9	203	206	7	4	-6	168	175	8	4	-6	97-	36	9	1	-11	101-	178
4	2	-9	62-	-51	5	2	-4	392	381	5	1	-17	112	162	6	4	-9	80-	2	7	5	-6	90	-60	8	5	-6	60-	-10	9	2	-11	121-	37
4	3	9	190	183	5	3	4	77	-65	5	2	-17	68-	7	6	5	-9	76	63	7	1	-7	299	295	8	1	-7	323	366	9	3	-11	116	120
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4	4	-9	88	-67	5	4	4	139	117	6	1	0	104	-101	6	1	-10	191	-191	7	3	-7	256	220	8	3	-7	107-	17	9	1	-12	96-	-9
4	5	-9	116	80	5	4	-4	323	298	6	2	0	287	276	6	2	-10	168	171	7	4	-7	61-	-23	8	4	-7	97-	-53	9	2	-12	112-	106
4	6	-9	126	84	5	5	4	47-	10	6	3	0	131	96	6	3	-10	87	79	7	5	-7	173	203	8	5	-7	199	202	9	3	-12	66-	60
4	0	10	233	221	5	5	-4	100	77	6	4	0	195	162	6	4	-10	126	102	7	0	-8	77-	69	8	0	-8	119	129	9	1	-13	87-	108
4	0	-10	213	209	5	6	-4	155	149	6	5	0	86	-68	6	5	-10	65-	-35	7	1	-8	183	184	8	1	-8	119-	165	9	2	-13	101-	76
4	1	10	51-	7	5	1	5	341	302	6	1	1	341	347	6	1	-11	313	349	7	2	-8	350	375	8	2	-8	286	298	9	3	-13	96	117
4	1	-10	195	-184	5	1	-5	607	674	6	1	-1	393	389	6	2	-11	79	90	7	3	-8	115	-98	8	3	-8	102-	-23	9	0	-14	61-	13
4	2	10	101	85	5	2	5	206	-162	6	2	1	76-	-37	6	3	-11	107-	63	7	4	-8	247	228	8	4	-8	97-	96	8	1	-14	79-	60
4	2	-10	373	427	5	2	-5	108-	-25	6	2	-1	203	183	6	4	-11	136	-108	7	5	-8	50-	31	8	5	-8	37-	-75	9	2	-14	86-	157
4	3	10	15-	-71	5	3	5	116	90	6	3	1	163	175	6	5	-11	168	183	7	1	-9	217	201	8	1	-9	116-	166	9	1	-15	62-	66
4	3	-10	115	-83	5	3	-5	68	52	6	3	-1	166	171	6	0	-12	96-	103	7	2	-9	171	-160	8	2	-9	146-	56	9	2	-15	56-	-22
4	4	-10	191	192	5	4	5	109	93	6	4	1	87	-79	6	1	-12	97	69	7	3	-9	367	354	8	3	-9	249	235	9	0	-16	36-	68
4	5	-10	126	109	5	4	-5	65	-57	6	4	-1	108-	-36	6	2	-12	276	287	7	4	-9	127	118	8	6	-9	92-	5	10	0	-2	101	131
4	6	-10	60	77	5	5	5	181	208	6	5	1	156	166	6	3	-12	70-	35	7	5	-9	65	69	8	5	-9	79	110	10	1	-2	56-	-37
4	1	11	63-	115	5	5	-5	346	321	6	5	-1	255	266	6	4	-12	131	111	7	0	-10	331	538	8	0	-10	218	233	10	1	-3	187	200
4	1	-11	309	313	5	6	-5	60-	69	6	6	-1	65	-82	6	5	-12	75	-72	7	1	-10	161	-149	8	1	-10	116-	67	10	2	-3	67-	-9
4	2	-11	259	-256	5	0	6	131	126	6	0	2	538	608	6	1	-13	180	152	7	2	-10	187	166	8	2	-10	146-	133	10	0	-4	170	215
4	3	-11	195	172	5	0	-6	405	420	6	0	-2	471	514	6	2	-13	61-	42	7	3	-10	92-	15	8	3	-10	100-	37	10	1	-4	76-	146
4	4	-11	124	100	5	1	6	190	-160	6	1	2	239	-221	6	3	-13	204	187	7	4	-10	195	204	8	4	-10	101	195	10	2	-4	79-	116
4	5	-11	212	208	5	1	-6	86	65	6	1	-2	368	355	6	4	-13	51-	19	7	5	-10	65	63	8	1	-11	112	117	10	1	-5	80-	128
4	0	12	259	288	5	2	6	308	310	6	2	2	133-	70	6	0	-14	175	171	7	1	-11	195	233	8	2	-11	140-	95	10	2	-5	90-	39
4	1	-12	107-	66	5	2	-6	522	519	6	2	-2	259	226	6	1	-14	105-	93	7	2	-11	87	-62	8	3	-11	255	260	10	3	-5	36-	161
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4	4	-12	155	133	5	4	6	94	65	6	4	2	198	195	6	4	-14	165	189	7	5	-11	63	91	8	1	-12	109-	-125	10	2	-6	97-	76
4	5	-12	126	112	5	4	-6	122	106	6	4	-2	373	313	6	1	-15	91	77	7	0	-12	235	257	8	2	-12	228	236	10	3	-6	46-	9
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4	2	-13	80	-59	5	6	-6	101	206	6	5	-2	95	-97	6	3	-15	235	235	7	2	-12	185	216	8	4	-12	160	160	10	2	-7	97-	68
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4	4	-14	140	103	5	4	-7	161	115	6	4	3	87-	102	7	1	0	229	-239	7	1	-14	92	97	8	3	-14	63-	-52	10	3	-9	99	128
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4	1	-16	57-	63	5	1	8	159	-129	6	0	4	175	184	7	1	1	204	197	7	2	-15	89-	19	8	1	-16	63-	4	10	1	-11	79-	125
4	2	-16	210	169	5	1	-8	275	315	6	0	-4	526	505	7	1	-1	659	644	7	3	-15	77	102	8	2	-16	62-	40	10	2	-11	86-	-30
4	3	-16	60-	37	5	2	8	151	121	6	1	4	84-	-9	7	2	1	100-	10	7	0	-16	155	162	9	0	0	324	335	10	0	-12	227	270
4	1	-17	133	109	5	2	-8	185	189	6	1	-4	92-	-41	7	2	-1	173	-154	7	1	-16	67-	21	9	1	0	76-	-12	10	1	-12	72-	-44
5	0	0	55-	18	5	3	8	51-	15	6	2	4	266	255	7	3	1	316	307	7	2	-16	99-	49	9	2	0	86-	46	10	2	-12	75-	65
5	1	0	70	76	5	3	-8	72-	-58	6	2	-4	272	262	7	3	-1	60-	0	7	1	-17	52	61	9	1	1	62-	153	10	1	-13	118	163
5	2	0	785	722	5	4	-8	223	224	6	3	4	96	-85	7	4	1	89-	-27	8	0	0	83-	72	9	1	-1	176	182	10	2	-13	56-	36
5	3	0	146	106	5	5	-8	70-	-59	6	3	-4	114	98	7	4	-1	57	46	8	1	0	106-	44	9	2	1	51-	-36	10	0	-14	83	130
5	4	0	232	187	5	6	-8	163	197	6	4	4	163	151	7	5	-1	165	203	8	2	0	127-	217	9	2	-1	101-	125	10	1	-14	36-	25
5	5	0	199	-133	5	1	9	118	97	6	4	-4	436	397	7	0	2	325	291	8	3	0	86-	69	9	3	-1	56-	97	11	0	-6	25-	216
5	6	0	69	76	5	1	-9	222	249	6	5	-4	72-	-56	7	0	-2	380	396	8	4	0	183	196	9	0	2	31-	223	11	1	-7	33-	162
5	1	1	515	510	5	2	9	58-	-12	6	6	-4	57-	27	7	1	2	52-	21	8	1	1	99-	162	9	0	-2	75-	90	11	0	-8	110	120
5	1	-1	626	607	5	2	-9	218	220	6	1	5	208	203	7	1	-2	111	135	8	1	-1	109-	134	9	1	-2	96-	22	11	1	-8	61-	70
5	2	1	355	337	5	3	-9	432	419	6	1	-5	363	338	7	2	2	163	131	8	2	1	116-	78	9	2	-2	228	235	11	1	-9	61-	118
5	2	-1	255	-209	5	4	-9	153	-123	6	2	5	112-	-76	7	2	-2	282	267	8	2	-1	136-	19	9	3	-2	68-	7	11	0	-10	36-	68
5	3	1	288	192	5	5	-9	136	110	6	2	-5	121-	-24	7	3	2	32-	-16	8	3	1	208	212	9	1	-3	101-	156	11	1	-10	36-	71
5	3	-1	517	455	5	6	-9	63-	6	6	3	5	176	144	7	3	-2	279	-195															

Table IV. Atomic peak heights (e.Å^{-3}), curvatures (e.Å^{-5}), and e.s.d.'s.

		ρ	A_{hh}	A_{kk}	A_{ll}	A_{hk}	A_{hl}	A_{kl}
Cd	obs.	109.1	1028	1084	1017	12	460	—24
	calc.	110.5	1030	1087	1032	9	470	—25
S	obs.	31.1	304	319	280	—3	134	—15
	calc.	31.3	305	317	286	—2	137	—15
Cl	obs.	31.2	278	297	283	—1	121	11
	calc.	31.6	282	298	286	0	125	7
N(1)	obs.	10.7	79	98	79	—2	32	2
	calc.	11.0	78	100	83	—4	36	4
N(2)	obs.	9.7	71	78	86	—7	37	4
	calc.	9.8	73	79	88	—7	38	6
N(3)	obs.	10.6	99	84	85	—3	42	—7
	calc.	10.7	100	83	85	—4	43	—6
N(4)	obs.	8.2	70	61	57	0	29	—1
	calc.	8.2	69	62	59	1	30	—1
C	obs.	9.9	113	71	85	—10	45	4
	calc.	10.1	115	73	85	—7	48	0
e.s.d.'s		3	4	5	6	3	3	3

Table V. Main interatomic distances and angles (with e.s.d.'s).

Coordination around metal			
Cd-S	2.59(1) Å	S-Cd-N(2)	76.7(6)°
Cd-Cl	2.73(1)	S-Cd-Cl	89.1(2)
Cd-N(2)	2.34(3)	Cl-Cd-N(2)	88.1(5)
Thiocarbohydrazide molecule			
S-C	1.74(2)	Cd-S-C	95.8(7)
C-N(1)	1.35(2)	S-C-N(1)	121.5(17)
C-N(3)	1.28(3)	S-C-N(3)	119.2(13)
N(1)-N(2)	1.39(4)	C-N(1)-N(2)	124.3(17)
N(3)-N(4)	1.41(3)	C-N(3)-N(4)	120.4(18)
		N(1)-N(2)-Cd	111.5(14)
		N(1)-C-N(3)	119.2(17)
Hydrogen bonds			
N(2)-Cl^{i}	3.23(2)	Cd-N(2)-Cl^{i}	110.3(11)
N(3)-Cl^{ii}	3.26(2)	N(1)-N(2)-Cl^{i}	121.4(9)
N(4)-N(1^{iii})	3.15(3)	C-N(3)-Cl^{ii}	132.8(9)
		N(4)-N(3)-Cl^{ii}	106.7(16)
		N(3)-N(4)-N(1^{iii})	146.0(20)
		N(4)-N(1^{iii})-N(2^{iii})	86.5(13)
		N(4)-N(1^{iii})-Cl^{iii}	145.9(19)

Asymmetric units

i $-x+2, -y+1, -z$ ii $-x+1, y-\frac{1}{2}, -z-\frac{1}{2}$ iii $-x+1, -y+1, -z$

The structure was solved by Patterson and Fourier methods and refined by differential syntheses.

Anisotropic temperature factors were introduced. (Final $R=9.7\%$). Hydrogen atoms were not identified in the difference Fourier map. The results of the structure determination are summarized in Tables I→V.

Discussion

The structure (Figure 1) consists of octahedral complexes with chelate molecules lying in the same plane and the chlorine atoms in the apical positions.

The chelating atoms (Figure 2) are N, of $-NH_2$, and S, thus confirming the assumptions on which were based the interpretations of IR spectra[1] and of equilibria in solution.[2] Atoms N, S are in *trans* positions and all of them lie by symmetry conditions in the same plane as the metal atom. The chlorine atoms lie very close to the normal to that plane. The distance Cd–Cl = 2.73(1) Å is comparable with the sum of ionic radii (2.78 Å) and shows that the bond should be prevalently ionic. The organic ligand forms pentatomic chelate rings as expected; it is therefore in the same configuration, with one $-NH_2$ group turned toward S=C, as in the neutral molecule in crystals.[4] This configuration is different from that found in thiocarbohydrazide dichloride[5] where both $-NH_2$ groups are turned toward S=C.

(3) D. T. Cromer and J. B. Mann, *Acta Cryst.*, A24, 321 (1968).
(4) A. Braibanti, A. Tiripicchio and M. Tiripicchio Camellini, *Acta Cryst.*, B25, 2286 (1969).
(5) A. Braibanti, M. A. Pellinghelli, A. Tiripicchio and M. Tiripicchio Camellini, *Inorg. Chim. Acta* (in the press).

The distance Cd–N = 2.34(3) Å is only slightly shorter than that found in chelates of hydrazinecarboxylic acid (Cd–N = 2.38(3) Å,[6] 2.40(2) Å[7] and is equal to Cd–O in octahedral complexes (Cd–O = 2.34(3)Å[8]). The distance Cd–S = 2.59(1) Å is significantly shorter than that found in complexes of thiourea (Cd–S = 2.638(4), 2.647(4) Å[9]); the shortening is probably due to chelation.

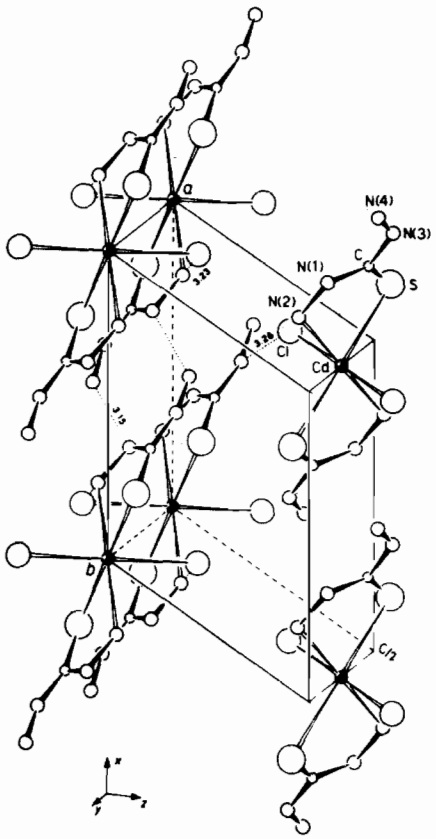

Figure 1. Clinographic projection of the structure of bis-(thiocarbohydrazide-N,S)cadmium dichloride.

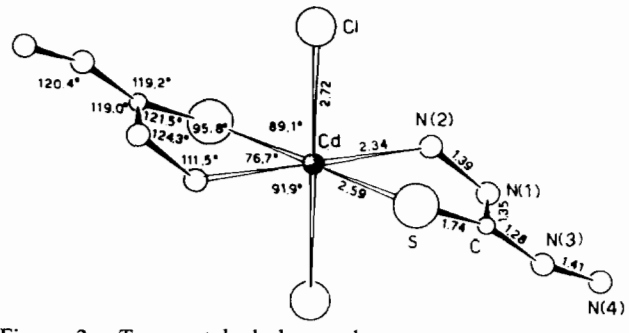

Figure 2. *Trans*-octahedral complex.

(6) A. Braibanti, A. Tiripicchio, A. M. Manotti Lanfredi and F. Bigoli, *Zeit. Kristallogr.*, *126*, 307 (1968).
(7) A. Braibanti, A. M. Manotti Lanfredi, A. Tiripicchio and F. Bigoli, *Acta Cryst.*, B25, 100 (1969).

In the chelate ligand some differences with the isolated molecule can be detected. The distance C–S = 1.74(2) Å is comparable with that in thiocarbohydrazide (C–S = 1.724(10) Å[4]), in complexed thiourea (C–S = 1.76(1) Å,[9] 1.73(1) Å[10] and in free thiourea (C–S = 1.71(1) Å[11]). Also the distances N(1)–N(2) = 1.39(4) Å and N(3)–N(4) = 1.41(3) Å are not different from one another and equal to that found in the free ligand (1.405(8) Å[4]). In any case they are shorter than those found in free (N–N = 1.46 Å[12]) and complexed hydrazine (N–N = 1.47(2)[13]). The distances C–N(1) = 1.35(2) and C–N(3) = 1.28(3) Å as compared with single bond C–N = 1.47 Å indicate that they possess some double bond character, although in different amounts. They can be compared with values found in thiocarbohydrazide (C–N = 1.327(7) Å[4]), in thiourea (C–N = 1.33(1) Å[11]), and in thiosemicarbazide (C–N = 1.323(2) Å[14]). The angle S–C–N(1) = 121.5(17)° is smaller than the corresponding angle in the free molecule.

Table VI. Planarity of thiocarbohydrazide molecule. Equation of plane $5.2772x + 2.4606y - 12.1516z - 4.3449 = 0$

	Δ	σ	
Cd	0.9323 Å	0.0000 Å	
*S	−0.0004	0.0075	
*C	0.0233	0.0247	
*N(1)	−0.0099	0.0264	
*N(3)	−0.0130	0.0245	
*N(2)	−0.0006	0.0297	
*N(4)	0.0073	0.0369	$\Sigma(\Delta/\sigma)^2 = 1.3542$

* Atoms lying in the plane.

The whole ligand molecule lies in one plane (Table VI). This aspect is again different from that found in the free ligand where both terminal $-NH_2$ groups are out of the plane of the thioureide group, N–CS–N.

The complexes are held together in the crystal by hydrogen bonds N(3)–H . . . Cl = 3.26(2) Å and N(2)–H . . . Cl = 3.23(2) Å, thus forming layers parallel to (102). Weak contacts N(1) . . . N(4) = 3.15(3) Å join the layers to one another.

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