2·637 Å, and by the fifth weak coordination bond of the O(3″) atom to the copper ion. Also there is a weak hydrogen bond $W(2)$–O(3″), 2·952 Å, holding the free water molecule $W(2)$ in the crystal. The close intermolecular contacts are listed in Table 5. Many of them make fairly rigid contacts among molecules. Of these some are unusually short; for instance, C(4)–C(4′) is 3·276 Å. Probably corresponding to such rigid intermolecular contacts, smaller values were found for the thermal parameters.

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The Crystal Structure of Monothiourea-cadmium Sulphate Dihydrate

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The crystal structure of $Cd[SC(NH_2)_2]SO_4.2H_2O$ has been determined by a three-dimensional X-ray analysis and refined by differential methods using anisotropic thermal parameters; final $R = 12{\cdot}9\,\%$. Eight formula units are contained in the orthorhombic (*Pbca*) unit cell: $a = 13{\cdot}46_1$, $b = 7{\cdot}78_3$, $c = 15{\cdot}9_7$ Å. Coordination around each cadmium atom is octahedral as it coordinates to two sulphur atoms from two thiourea molecules [Cd–S 2·638(4) and 2·647(4) Å], two oxygen atoms from two sulphate groups [Cd–O 2·27(2) and 2·29(2) Å] and two oxygen atoms from two water molecules [Cd–O 2·31(1) and 2·27(2) Å]. The coordination polyhedra are linked in chains by sulphur and sulphato bridges, with the sulphur atom of thiourea and two oxygen atoms of each sulphate group bonded to two adjacent metal atoms.

Introduction

Cadmium sulphate forms with thiourea (*tu*) three complex compounds, $Cdtu SO_4.2H_2O$, $Cdtu_3SO_4$ and $Cdtu_4SO_4$, which can be obtained from aqueous solutions of the components by varying their molar ratios (Nardelli & Chierici, 1958). An X-ray analysis of the crystal structure of the first compound was undertaken to study the binding of the thiourea molecule and the sulphato group in it. Indeed, not only is the chemical composition alone insufficient to explain the behaviour of the ligands, but also it could lead to the erroneous conclusion that there is tetrahedral coordination around the cadmium atom.

Experimental

Cell constants, refined by a least-squares procedure on powder diffractometer data, are not significantly different from those reported by Nardelli & Chierici (1958).

Crystal data

$Cd[SC(NH_2)_2]SO_4.2H_2O$, $M=320{\cdot}6$, orthorhombic,
$a=13{\cdot}461$ (5), $b=7{\cdot}783$ (3), $c=15{\cdot}967$ (12) Å
$U=1673$ Å^3, $Z=8$, $D_m=2{\cdot}51$, $D_x=2{\cdot}545$ g.cm^{-3}
$\mu=265{\cdot}8$ cm^{-1} (Cu $K\alpha$)
$F(000)=1248$
Space group: *Pbca* (D_{2h}^{15}) (from systematic absences).
(The e.s.d.'s, given in parentheses, are quoted in units in the last place).

A set of intensity data was obtained up to the 11th layer around [100] and up to the 6th layer around [010] on an integrating Weissenberg camera using the multiple film technique and Cu $K\alpha$ radiation. For the photographs taken around [100] the sample used was a fragment of mean radius 0·034 cm and for the data taken around [010] a needle of rectangular cross-section of mean thickness 0·04 cm. The absorption correction was assumed to be that appropriate to a sphere for the first set of data and to a cylinder for the second set. The intensities were measured photometrically and corrected for Lorentz and polarization factors. The shape of the spots of non-equatorial layers was taken into account following Phillips (1956). The absolute scale was determined by Wilson's method after applying the cross-correlation method of Rollett & Sparks (1960) to put all the data on a common scale. The number of the observed independent reflexions was 1561 out of 1911 contained in the limiting sphere for Cu $K\alpha$.

Structure determination and refinement

After an early two-dimensional study on (010), which was used to get a complete description of the projection of the structure, the analysis was continued in three dimensions, starting with a Patterson synthesis and then using the heavy atom technique. The agreement index, $R(hkl)=28{\cdot}2\%$, obtained considering only the contributions of cadmium and sulphur atoms to the structure factors, dropped to 19·5% at the first introduction of the contributions of all the other atoms.

The refinement was carried out with several cycles of Booth's differential synthesis using anisotropic thermal parameters. The final values of the agreement indices were: $R(hkl)=12{\cdot}9\%$ and $R'(hkl)=13{\cdot}9\%$ (R for observed reflexions only, R' considering also the unobserved reflexions; multiplicities not considered). The absolute scale for F_o, obtained by Wilson's method, was kept unchanged during the whole analysis. At the end, rescaling by comparison of ΣF_o and ΣF_c gave a small increase in the R values ($R=13{\cdot}1$, $R'=14{\cdot}1\%$), while a sensible improvement to $R=10{\cdot}6$ and $R'=11{\cdot}6\%$ was achieved by rescaling data from layers about [010]; this was mainly due to the neglect of discontinuous absorption effects which were never considered. Nevertheless, these rescalings were not applied.

At the end of the refinement the atomic parameters were as reported in Table 1, which shows also the e.s.d. (Cruickshank, 1949, 1950) and the ratio $r=$ (e.s.d.)/(shift) for each coordinate. The B values were determined from the second derivatives of electron density; their e.s.d.'s were calculated following Cruickshank (1956). In Table 2 the observed atomic peak shapes are compared with the calculated ones. The structure factors reported in Table 3 are calculated with the use of the final parameters of Table 1 and atomic scattering factors taken from *International Tables for X-ray Crystallography* (1962).

No attempt was made to locate the hydrogen atoms directly; the coordinates of the hydrogen atoms of thiourea were calculated on the assumption of a trigonal bond configuration around each nitrogen atom with a distance N–H of 1·03 Å and a complete planarity of the molecule. The positions of the hydrogen atoms of the H_2O molecules can be reasonably guessed as indicated in the next section. No contributions from hydrogen atoms were introduced in the structure factor calculations.

Discussion

In the following discussion, atoms belonging to different asymmetric units are labelled as follows:

Superscript	Coordinates	Superscript	Coordinates
none	x,y,z	vi	$\frac{1}{2}-x,1-y,\frac{1}{2}+z$
′	$\frac{1}{2}-x,y-\frac{1}{2},z$	vii	$x,\frac{1}{2}-y,\frac{1}{2}+z$
″	$\frac{1}{2}-x,y+\frac{1}{2},z$	viii	$x-\frac{1}{2},\frac{1}{2}-y,\bar{z}$
‴	$\bar{x},y+\frac{1}{2},\frac{1}{2}-z$	ix	$x,\frac{1}{2}-y,z-\frac{1}{2}$
iv	$\bar{x},y-\frac{1}{2},\frac{1}{2}-z$	x	$1-x,1-y,\bar{z}$
v	$x-\frac{1}{2},y,\frac{1}{2}-z$		

The cadmium atom is octahedrally surrounded by two sulphur atoms from two thiourea molecules, two oxygen atoms from two sulphate groups and two water [O(1) and O(2)] molecules (Figs. 1 and 2) with the bond distances and angles quoted in Table 4. The sulphur atom S(2) of thiourea coordinates to two adjacent metal atoms with Cd–S distances practically equal and sensibly shorter than those (2·74 and 2·71 Å) found in bisthiourea-cadmium formate (Nardelli, Fava Gasparri & Boldrini, 1965) in which the ligand coordinates in a similar way; these values are intermediate between the sums of Pauling's covalent (2·52 Å) and ionic (2·87 Å) radii. The four Cd–O distances are not significantly different and agree well with those found in other compounds: *e.g.* 2·28 Å in bisthiourea-cadmium formate (Nardelli, Fava Gasparri & Boldrini, 1965), 2·23 Å in bisacetamide-cadmium chloride (Cavalca, Nardelli & Coghi, 1957), 2·28 Å in bisurea-cadmium chloride (Nardelli, Cavalca & Fava, 1957) and 2·34 Å in bisbiuret-cadmium chloride (Cavalca, Nardelli &

Fava, 1960). Also in this case all these values are intermediate between the sums of Pauling's covalent (2·22 Å) and ionic (2·43 Å) radii. The octahedra are linked in chains by sulphur and sulphato bridges as shown in Fig. 2; the chains run along the y axis.

Bond distances and angles in the SO_4^{2-} group, quoted in Table 5, are in good agreement with those generally observed in other sulphates.

The thiourea molecule is planar, the least-squares plane being: $-0{\cdot}0394x+0{\cdot}9242y+0{\cdot}3799z=4{\cdot}7770$; the largest distance from this plane is 0·01 Å for N(1). Bond distances and angles for the molecule are quoted in Table 5. The S–C distance is significantly larger than that in free thiourea [1·707 (12) Å, Kunchur & Truter (1958*a*)]. Thus, as with bisthiourea-nickel(II) thiocyanate [1·758 (11) Å, Nardelli, Fava Gasparri, Giraldi Battistini & Domiano (1966)] and bisthiourea-zinc chloride [1·78 (2) Å, Kunchur & Truter (1958*b*)], the coordination seems to influence the dimensions of the ligand molecule. The two C–N distances are not signi-

Table 1. *Final atomic fractional coordinates* ($\times 10^4$), *thermal parameters* ($\times 10$ Å^2) *with e.s.d.'s and ratios* (*e.s.d.*)/(*coordinate shift*)

	x (σ)	y (σ)	z (σ)	B_{11}(σ)	B_{22}(σ)	B_{33}(σ)	B_{12}(σ)	B_{13}(σ)	B_{23}(σ)	$\lvert r(x)\rvert$	$\lvert r(y)\rvert$	$\lvert r(z)\rvert$
Cd	1900 (1)	1771 (2)	1687 (1)	28 (0)	38 (1)	26 (0)	0 (1)	0 (0)	−1 (1)	8	5	7
S(1)	4074 (2)	3118 (5)	751 (2)	23 (1)	35 (2)	26 (1)	−1 (2)	0 (2)	−2 (2)	∞	9	6
S(2)	2015 (3)	4611 (5)	2583 (2)	26 (1)	37 (3)	24 (1)	−2 (2)	0 (2)	1 (2)	27	9	11
O(1)	422 (11)	1085 (21)	2347 (8)	33 (4)	68 (14)	53 (7)	−2 (10)	0 (9)	−16 (10)	7	2	13
O(2)	1226 (14)	3394 (22)	655 (10)	48 (5)	41 (9)	46 (6)	0 (8)	−15 (8)	−2 (9)	7	43	24
O(3)	3356 (17)	1743 (23)	951 (11)	39 (6)	39 (8)	61 (13)	−3 (10)	8 (13)	1 (13)	2	7	2
O(4)	4482 (10)	2772 (28)	−77 (14)	34 (5)	61 (12)	35 (5)	−1 (9)	5 (9)	−11 (9)	19	9	14
O(5)	4883 (11)	3180 (26)	1370 (10)	46 (5)	62 (16)	36 (4)	4 (11)	−15 (8)	0 (11)	108	7	97
O(6)	3549 (13)	4727 (31)	746 (11)	39 (5)	53 (11)	24 (4)	−6 (8)	−2 (7)	1 (9)	17	7	26
N(1)	1987 (20)	3318 (42)	4091 (16)	58 (3)	76 (32)	33 (9)	8 (25)	4 (18)	17 (24)	25	10	40
N(2)	3525 (11)	3878 (31)	3576 (13)	44 (7)	59 (20)	36 (8)	0 (14)	−3 (12)	13 (17)	21	18	9
C	2590 (10)	3883 (20)	3510 (7)	36 (7)	37 (12)	26 (6)	2 (11)	−3 (9)	4 (11)	25	25	73

Table 2. *Atomic peak heights* (e.Å^{-3}), *curvatures* (e.Å^{-5}) *and e.s.d.'s*

		ϱ	$-A_{hh}$	$-A_{kk}$	$-A_{ll}$	A_{kl}	A_{hl}	A_{hk}
Cd	obs.	122·6	1268	1135	1262	−9	−2	9
	calc.	123·6	1262	1147	1251	−6	−2	5
S(1)	obs.	36·1	385	335	360	−12	−5	−1
	calc.	36·2	380	342	356	−10	−4	−1
S(2)	obs.	35·9	378	341	392	4	5	−15
	calc.	36·4	374	351	387	4	5	−14
O(1)	obs.	10·2	76	77	69	−16	−18	3
	calc.	9·9	74	79	69	−15	−18	4
O(2)	obs.	10·2	70	75	70	−13	−3	4
	calc.	10·6	81	78	74	−13	−4	3
O(3)	obs.	9·9	56	82	51	−11	−10	11
	calc.	9·9	58	83	53	−11	−10	9
O(4)	obs.	10·6	104	66	64	3	0	−2
	calc.	10·6	103	69	64	4	0	−2
O(5)	obs.	10·3	87	71	88	1	−2	1
	calc.	10·6	87	73	86	1	0	1
O(6)	obs.	12·7	98	67	106	17	0	−20
	calc.	12·0	98	67	105	17	0	−18
N(1)	obs.	7·5	52	57	70	12	0	4
	calc.	7·8	52	59	68	11	0	4
N(2)	obs.	8·7	78	65	70	8	−9	2
	calc.	9·0	78	65	70	6	−9	1
C	obs.	9·0	85	85	100	−3	−13	−2
	calc.	9·5	85	85	99	−3	−12	−2
e.s.d.'s		0·6	6	6	6	3	3	3

Table 3. *Observed and calculated structure factors*

A minus sign with F_o means less' than'.

h	k	l	$10F_o$	$10F_c$
0	2	0	2084	−2593
0	4	0	847	−797
0	6	0	1810	1815
0	8	0	1154	−934
0	10	0	113	62
0	2	1	832	−801
0	4	1	1377	1945
0	6	1	141	48
0	8	1	101	−99
0	10	1	437	352
0	0	2	2494	−1472
0	2	2	923	−831
0	4	2	404	380
0	6	2	254	−320
0	8	2	206	220
0	10	2	81	76
0	2	3	1573	1603
0	4	3	871	−1021
0	6	3	156	−147
0	8	3	111	−136
0	0	4	2866	−1858
0	2	4	1673	1795
0	4	4	776	867
0	6	4	676	−800
0	8	4	398	308
0	2	5	1703	1829
0	4	5	1304	−1504
0	6	5	857	1028
0	8	5	616	580
0	0	6	1069	537
0	2	6	2014	−1999
0	4	6	586	−619
0	6	6	462	535
0	8	6	202	−220
0	2	7	1630	−1817
0	4	7	1144	1260
0	6	7	675	−806
0	8	7	434	−405
0	0	8	1400	−953
0	2	8	1159	1215
0	4	8	437	417
0	6	8	771	−885
0	8	8	411	376
0	2	9	109	183
0	4	9	394	424
0	6	9	72−	−14
0	8	9	158	133
0	0	10	1856	−1426
0	2	10	275	−223
0	4	10	205	243
0	6	10	299	−283
0	8	10	225	166
0	2	11	248	223
0	4	11	619	−813
0	6	11	162	−101
0	8	11	40−	19
0	0	12	3226	2813
0	2	12	979	−870
0	4	12	98−	−81
0	6	12	860	866
0	8	12	679	−511
0	2	13	1336	−1232
0	4	13	1276	1263
0	6	13	120	−106
0	0	14	572	−306
0	2	14	99−	11
0	4	14	77	17
0	6	14	241	−249
0	2	15	255	382
0	4	15	441	−407
0	6	15	47−	−55
0	0	16	72−	−26
0	2	16	393	317
0	4	16	220	217
0	6	16	113	−117
0	2	17	773	835
0	4	17	568	−544
0	0	18	482	374
0	2	18	563	−526
0	4	18	158	−162
0	2	19	696	−593
0	0	20	613	−485
0	2	20	349	366
1	1	1	1841	819
1	2	1	1514	−1315
1	3	1	823	−837
1	4	1	803	768
1	5	1	762	782
1	6	1	81−	18
1	7	1	163	−172
1	8	1	118	−127
1	9	1	91	−74
1	0	2	4721	−3168
1	1	2	106−	93
1	2	2	1991	1856
1	3	2	313	−273
1	4	2	164	141
1	5	2	366	275
1	6	2	1093	−1289
1	7	2	140−	29
1	8	2	922	660
1	9	2	80−	−27
1	1	3	990	−530
1	2	3	2688	3130
1	3	3	943	976
1	4	3	1575	−1885
1	5	3	397	−435
1	6	3	380	435
1	7	3	138−	−87
1	8	3	387	325
1	9	3	302	184
1	0	4	1588	984
1	1	4	1680	1588
1	2	4	1371	−1476
1	3	4	164	−137
1	4	4	359	−412
1	5	4	327	−410
1	6	4	541	689
1	7	4	474	470
1	8	4	530	−369
1	9	4	273	−193
1	1	5	275	245
1	2	5	1014	−1056
1	3	5	440	−462
1	4	5	613	734
1	5	5	55−	4
1	6	5	358	−393
1	7	5	133−	60
1	8	5	347	−227
1	9	5	91	−53
1	0	6	1028	−705
1	1	6	598	−532
1	2	6	281	230
1	3	6	187	123
1	4	6	301	325
1	5	6	174	195
1	6	6	119	−86
1	7	6	130−	−11
1	8	6	99−	5
1	9	6	64−	−4
1	1	7	391	−305
1	2	7	1117	−1170
1	3	7	422	−398
1	4	7	776	808
1	5	7	90	114
1	6	7	131	−115
1	7	7	124−	49
1	8	7	61−	−54
1	9	7	69	−45
1	0	8	1710	−1300
1	1	8	1247	−1025
1	2	8	779	769
1	3	8	223	210
1	4	8	69−	88
1	5	8	201	234
1	6	8	607	−656
1	7	8	388	−390
1	8	8	540	374
1	9	8	186	114
1	1	9	266	−200
1	2	9	1110	1068
1	3	9	579	563
1	4	9	1165	−1267
1	5	9	422	−435
1	6	9	125	116
1	7	9	108−	7
1	8	9	192	119
1	0	10	3214	2605
1	1	10	201	164
1	2	10	925	−816
1	3	10	114−	−56
1	4	10	163	−135
1	5	10	201	−210
1	6	10	864	855
1	7	10	151	142
1	8	10	735	−486
1	1	11	147	146
1	2	11	687	−559
1	3	11	450	−384
1	4	11	969	961
1	5	11	302	259
1	6	11	74−	−43
1	7	11	90−	−37
1	8	11	220	−132
1	0	12	498	−308
1	1	12	242	−236
1	2	12	197	−199
1	3	12	212	178
1	4	12	88	79
1	5	12	51	−53
1	6	12	67−	4
1	7	12	227	−181
1	8	12	40−	−4
1	1	13	495	410
1	2	13	472	−421
1	3	13	398	−324
1	4	13	95−	23
1	5	13	388	331
1	6	13	62−	−24
1	7	13	83	−78
1	0	14	908	−676
1	1	14	336	−301
1	2	14	827	698
1	3	14	99−	76
1	4	14	230	196
1	5	14	169	163
1	6	14	451	−390
1	7	14	48−	−48
1	1	15	218	−178
1	2	15	1153	1071
1	3	15	168	166
1	4	15	825	−780
1	5	15	37	−2
1	6	15	424	399
1	0	16	782	611
1	1	16	620	533
1	2	16	707	−644
1	3	16	127	−128
1	4	16	435	−383
1	5	16	258	−237
1	6	16	332	306
1	1	17	213	171
1	2	17	421	−376
1	3	17	155	−120
1	4	17	279	258
1	5	17	45	53
1	0	18	291	−227
1	1	18	85	25
1	2	18	310	275
1	3	18	55−	18
1	4	18	194	165
1	1	19	168	−140
1	2	19	170	−134
1	3	19	38−	32
1	0	20	796	−621
1	1	20	264	−222
2	0	0	3351	−2882
2	1	0	1255	−922
2	2	0	101−	−60
2	3	0	166	−224
2	4	0	301	335
2	5	0	545	609
2	6	0	776	−877
2	7	0	633	−551
2	8	0	520	398
2	9	0	55−	−19
2	1	1	1217	−903
2	2	1	646	789
2	3	1	1630	1520
2	4	1	1745	−2134
2	5	1	609	−622
2	6	1	248	244
2	7	1	96−	7
2	8	1	149	117
2	9	1	219	161
2	0	2	2937	2188
2	1	2	2431	1927
2	2	2	687	−501
2	3	2	91−	−78
2	4	2	383	328
2	5	2	66−	−14
2	6	2	433	465
2	7	2	711	623
2	8	2	404	−327
2	9	2	133	−106
2	1	3	969	−733
2	2	3	1032	755
2	3	3	938	−803
2	4	3	65−	121
2	5	3	354	−357
2	6	3	325	362
2	7	3	186	178
2	8	3	168	134
2	9	3	52−	12
2	0	4	1874	1213
2	1	4	871	221
2	2	4	1246	−1129
2	3	4	805	−799
2	4	4	523	−550
2	5	4	344	−391
2	6	4	172	196
2	7	4	92−	67
2	8	4	76−	−46
2	9	4	109	−100
2	1	5	1224	1091
2	2	5	1592	−1664
2	3	5	1166	−1248
2	4	5	676	794
2	5	5	372	457
2	6	5	444	−573
2	7	5	91−	119
2	8	5	419	−356
2	9	5	309	−242
2	0	6	1536	−1270
2	1	6	1472	−1623
2	2	6	1150	1278
2	3	6	625	687
2	4	6	690	824
2	5	6	633	821
2	6	6	563	−666
2	7	6	725	−742
2	8	5	395	342
2	9	6	430	337
2	1	7	1067	−971
2	2	7	1237	1454
2	3	7	959	1089
2	4	7	583	−617
2	5	7	600	−732
2	6	7	383	441
2	7	7	84−	−59
2	8	7	349	315
2	9	7	395	280
2	0	8	1168	892
2	1	8	838	724
2	2	8	987	−1023
2	3	8	317	−294
2	4	8	297	−296
2	5	8	555	−577
2	6	8	310	334
2	7	8	79−	14
2	8	8	151	−111
2	9	8	105	−56
2	1	9	483	386
2	2	9	574	−536
2	3	9	119	156
2	4	9	123	−136
2	5	9	469	474
2	6	9	74	−204
2	7	9	74−	−104
2	8	9	52−	−74
2	0	10	1196	869
2	1	10	830	657
2	2	10	110−	32
2	3	10	99−	−68
2	4	10	137	99
2	5	10	67−	−32
2	6	10	140	124
2	7	10	291	311
2	8	10	120	−99
2	1	11	629	531
2	2	11	629	−565
2	3	11	1096	−1089
2	4	11	1054	1108
2	5	11	579	622
2	6	11	144	−152
2	7	11	51−	−55
2	8	11	38−	−41
2	0	12	1866	−1429
2	1	12	769	−656
2	2	12	354	314
2	3	12	135	−105
2	4	12	145	162
2	5	12	299	294
2	6	12	563	−554
2	7	12	344	−319
2	8	12	330	266
2	1	13	120	−103
2	2	13	563	497
2	3	13	801	745
2	4	13	725	−704
2	5	13	321	−306
2	6	13	51−	−16
2	7	13	44−	−55
2	0	14	1242	1016
2	1	14	1011	991
2	2	14	459	−424
2	3	14	84−	−35
2	4	14	91	56
2	5	14	129	−116
2	6	14	253	269
2	7	14	411	364
2	1	15	258	−222
2	2	15	90−	−87
2	3	15	98	−108
2	4	15	150	163
2	5	15	63	−72
2	6	15	152	145
2	0	16	376	299
2	1	16	237	216
2	2	16	422	−380
2	3	16	160	−151
2	4	16	140	−135
2	5	16	90	−109
2	6	16	102	115
2	1	17	110	117
2	2	17	362	−330
2	3	17	166	−203
2	4	17	147	141
2	5	17	70	88
2	0	18	522	−449
2	1	18	636	−587
2	2	18	469	464
2	3	18	130	128
2	4	18	141	133
2	1	19	338	−296
2	2	19	423	461
2	3	19	373	357
2	0	20	134	124
2	1	20	116	131
3	1	1	700	−508
3	2	1	1503	1700
3	3	1	249	225
3	4	1	984	−1157
3	5	1	470	−542
3	6	1	378	405
3	7	1	101−	−16
3	8	1	106	87
3	9	1	186	160
3	0	2	1222	−712
3	1	2	2459	2137
3	2	2	109−	−129
3	3	2	343	−288
3	4	2	88−	−15
3	5	2	612	−922
3	6	2	108	121
3	7	2	620	545
3	8	2	91	−55
3	9	2	294	−204
3	1	3	1089	828
3	2	3	677	−614
3	3	3	1702	−1603
3	4	3	578	574
3	5	3	938	1146
3	6	3	102	−122
3	7	3	99−	23
3	8	3	186	−144
3	0	4	2440	−2163
3	1	4	2669	−2643
3	2	4	1187	1201
3	3	4	454	399
3	4	4	205	209
3	5	4	719	858
3	6	4	658	−761
3	7	4	811	−797
3	8	4	480	418
3	9	4	297	243
3	1	5	637	−568
3	2	5	112−	−82
3	3	5	1175	1227
3	4	5	61−	−9
3	5	5	445	−539
3	6	5	77−	36
3	7	5	95−	−56
3	8	5	73−	57
3	9	5	321	244
3	0	6	530	−345
3	1	6	177	−173
3	2	6	163	−134
3	3	6	269	−269
3	4	6	61−	9
3	5	6	169	−194
3	6	6	85−	−74
3	7	6	91−	35
3	8	6	70−	50
3	9	6	45−	−29
3	1	7	275	263
3	2	7	192	−138
3	3	7	693	798
3	4	7	129	62
3	5	7	147	−197
3	6	7	168	−221
3	7	7	87−	−60
3	8	7	66	−75
3	9	7	188	135
3	0	8	1614	1592
3	1	8	2046	2185
3	2	8	550	−636
3	3	8	102−	0
3	4	8	94−	−44
3	5	8	540	−656
3	6	8	423	472
3	7	8	633	695
3	8	8	305	−299
3	9	8	181	−140
3	1	9	855	769
3	2	9	923	−941
3	3	9	1314	−1577
3	4	9	705	795
3	5	9	693	604
3	6	9	101	−72
3	7	9	77−	13
3	8	9	122	−100
3	0	10	255	−169
3	1	10	1325	−1252
3	2	10	116	−89
3	3	10	146	167
3	4	10	85−	115
3	5	10	498	503
3	6	10	76−	−84
3	7	10	476	−525
3	8	10	45−	20
3	1	11	593	−534
3	2	11	550	492
3	3	11	1051	1042
3	4	11	581	−583
3	5	11	343	−341
3	6	11	114	110
3	7	11	61−	−62
3	8	11	35−	7
3	0	12	1032	830
3	1	12	108	−51
3	2	12	142	−107
3	3	12	142	139
3	4	12	85−	−43
3	5	12	140	143
3	6	12	140	145
3	7	12	151	134
3	1	13	466	−423
3	2	13	641	584
3	3	13	114	103
3	4	13	343	−372
3	5	13	343	−342
3	6	13	280	275
3	7	13	44−	8
3	0	14	151	79
3	1	14	508	466
3	2	14	360	−331
3	3	14	229	−258
3	4	14	98	−67
3	5	14	382	−386
3	6	14	133	146
3	7	14	198	194
3	1	15	659	584
3	2	15	183	−173
3	3	15	799	−803
3	4	15	85	67
3	5	15	467	491
3	6	15	87	−77
3	0	16	637	−563
3	1	16	611	−685
3	2	16	494	470
3	3	16	280	269
3	4	16	144	140
3	5	16	421	401
3	6	16	231	−266
3	1	17	408	−381
3	2	17	131	150
3	3	17	237	234
3	4	17	88	−82
3	5	17	275	−254
3	0	18	198	−191
3	1	18	99	91
3	2	18	70	−72
3	3	18	131	−137
3	4	18	66	−71
3	1	19	84	103
3	2	19	108	−80
3	3	19	226	248
3	0	20	383	324
3	1	20	134	−104
3	2	20	116	131
4	0	0	1274	−381
4	1	0	3702	3578
4	2	0	102−	177
4	3	0	935	−916
4	4	0	115	7
4	5	0	616	−646
4	6	0	299	−273
4	7	0	1359	1328
4	8	0	67	101
4	9	0	344	−256
4	1	1	212	−43
4	2	1	259	−188
4	3	1	1531	−1879
4	4	1	180	190
4	5	1	598	711
4	6	1	67−	37
4	7	1	152	125
4	8	1	64−	32
4	9	1	321	−223
4	0	2	1822	−1553
4	1	2	792	−599
4	2	2	101−	−101
4	3	2	271	−290
4	4	2	85−	72
4	5	2	90	126
4	6	2	236	−245
4	7	2	746	−696
4	8	2	159	134
4	9	2	62	−35
4	1	3	949	731
4	2	3	413	−404
4	3	3	1229	1320
4	4	3	125	46
4	5	3	371	−390
4	6	3	301	−326
4	7	3	77−	32
4	8	3	49	33
4	0	4	1111	573
4	1	4	1058	−847
4	2	4	249	300
4	3	4	421	386
4	4	4	338	249
4	5	4	1967	−1982
4	6	4	640	−603
4	3	5	1562	1751
4	4	5	382	421
4	5	5	830	−973
4	6	5	70−	−7
4	7	5	74−	19
4	8	5	57−	22
4	9	5	444	375
4	0	6	1386	1093
4	1	6	1946	2031
4	2	6	594	−620
4	3	6	942	−641
4	4	6	87−	−21
4	5	6	827	−1010
4	6	6	297	331
4	7	6	432	464
4	8	6	137	−144
4	9	6	275	−235
4	1	7	1558	1741
4	2	7	240	−237
4	3	7	1392	−1670
4	4	7	123	124
4	5	7	807	1054
4	6	7	101	−124
4	7	7	67−	−6
4	8	7	70	−101
4	9	7	441	−365
4	0	8	1287	1116
4	1	8	1132	−1163
4	2	8	103−	−89
4	3	8	259	302
4	4	8	87−	75
4	5	8	733	907
4	6	8	162	189
4	7	8	254	−212
4	8	8	87	−110
4	1	9	423	−397
4	2	9	415	399
4	3	9	540	−601
4	4	9	192	−183
4	5	9	69−	53
4	6	9	238	254
4	7	9	57−	−54
4	8	9	62	67
4	0	10	367	−270
4	1	10	534	−511
4	2	10	155	−85
4	3	10	99	−64
4	4	10	110	−19
4	5	10	81	64
4	6	10	60−	−11
4	7	10	436	−464
4	8	10	33−	36
4	1	11	142	−143
4	2	11	352	319
4	3	11	918	974
4	4	11	354	−361
4	5	11	327	−338
4	6	11	101	93
4	7	11	142	−119
4	8	11	42	34
4	0	12	202	−135
4	1	12	1647	1676
4	2	12	187	163
4	3	12	262	−241
4	4	12	83−	−30
4	5	12	413	−417
4	6	12	125	−112
4	7	12	753	705
4	1	13	241	201
4	2	13	99	69
4	3	13	1126	−1178
4	4	13	105	90
4	5	13	529	544
4	6	13	44−	23
4	7	13	33−	35
4	0	14	493	−407
4	1	14	494	−476
4	2	14	68−	−32
4	3	14	177	−188
4	4	14	77	−52
4	5	14	138	131
4	6	14	112	−125
4	1	15	81	99
4	2	15	81−	−79
4	3	15	487	465
4	4	15	59−	34
4	5	15	159	−138
4	6	15	134	−134
4	0	16	110	−76
4	1	16	141	−137
4	2	16	73−	56
4	3	16	110	114
4	4	16	64	74
4	5	16	199	203
4	1	17	443	−419
4	2	17	101	−99
4	3	17	506	530
4	4	17	88	89
4	5	17	320	−383
4	0	18	99	75

Table 3 (*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	18	544	546	5	4	11	313	−322	6	4	5	173	−254	7	4	1	991	1076	7	2	13	458	−475	8	4	8	226	−360	9	6	5	256	291
4	2	18	76	−77	5	5	11	160	191	6	5	5	786	942	7	5	1	388	−408	7	3	13	298	307	8	5	8	52−	89	9	7	5	48−	42
4	3	18	156	−161	5	6	11	56	56	6	6	5	73−	114	7	6	1	269	−295	7	4	13	340	364	8	6	8	309	369	9	8	5	131	112
4	4	18	24−	14	5	7	11	75	86	6	7	5	62−	−66	7	7	1	73−	42	7	5	13	179	−212	8	7	8	142	174	9	0	6	105	−70
4	1	19	416	380	5	8	11	12−	29	6	8	5	191	173	7	8	1	229	−194	7	6	13	198	−199	8	8	8	162	−184	9	1	6	119	84
4	2	19	166	−160	5	0	12	255	215	6	9	5	282	−272	7	9	1	103	116	7	0	14	608	−561	8	1	9	336	−369	9	2	6	287	−342
5	1	1	812	565	5	1	12	83	−60	6	0	6	1340	1321	7	0	2	1804	−1495	7	1	14	290	263	8	2	9	109	−60	9	3	6	88−	14
5	2	1	593	508	5	2	12	248	−224	6	1	6	744	−747	7	1	2	686	631	7	2	14	341	377	8	3	9	231	284	9	4	6	79−	19
5	3	1	866	−872	5	3	12	209	−158	6	2	6	1085	−1203	7	2	2	768	711	7	3	14	98	−116	8	4	9	53−	−114	9	5	6	56−	23
5	4	1	646	−664	5	4	12	73−	−64	6	3	6	437	537	7	3	2	129	−145	7	4	14	234	237	8	5	9	166	−218	9	6	6	99	116
5	5	1	255	268	5	5	12	72	−65	6	4	6	298	−347	7	4	2	351	365	7	5	14	205	−213	8	6	9	137	−182	9	7	6	45−	33
5	6	1	136	176	5	6	12	42−	42	6	5	6	746	957	7	5	2	219	−234	7	6	14	221	−266	8	7	9	91	98	9	8	6	61	−82
5	7	1	174	158	5	7	12	131	−96	6	6	6	524	651	7	6	2	524	−540	7	1	15	359	356	8	0	10	409	404	9	1	7	102	−83
5	8	1	51−	−14	5	1	13	340	302	6	7	6	284	−299	7	7	2	279	267	7	2	15	529	566	8	1	10	72−	−1	9	2	7	383	441
5	9	1	209	−157	5	2	13	100−	40	6	8	6	390	−423	7	8	2	262	233	7	3	15	137	−159	8	2	10	95	71	9	3	7	191	225
5	0	2	1209	1154	5	3	13	69	−5	6	1	7	967	−1051	7	9	2	86−	−45	7	4	15	352	−399	8	3	10	73	−113	9	4	7	709	−838
5	1	2	1658	−1745	5	4	13	255	−267	6	2	7	714	−838	7	1	3	595	542	7	5	15	225	222	8	4	10	52−	79	9	5	7	179	−204
5	2	2	218	−229	5	5	13	51−	56	6	3	7	725	845	7	2	3	1147	1317	7	0	16	648	777	8	5	10	84	−116	9	6	7	180	216
5	3	2	445	443	5	6	13	63	75	6	4	7	427	548	7	3	3	559	−591	7	1	16	395	−373	8	6	10	192	204	9	7	7	41−	79
5	4	2	79−	−72	5	7	13	66	66	6	5	7	569	−756	7	4	3	1086	−1001	7	2	16	544	−590	8	7	10	98	−96	9	0	8	1268	1321
5	5	2	578	683	5	0	14	372	326	6	6	7	147	−196	7	5	3	378	321	7	3	16	134	162	8	1	11	110	154	9	1	8	245	254
5	6	2	310	379	5	1	14	772	−760	6	7	7	56−	−22	7	6	3	332	376	7	4	16	162	−179	8	2	11	637	−726	9	2	8	173	−290
5	7	2	715	−718	5	2	14	172	−148	6	8	7	170	−174	7	7	3	70−	44	7	1	17	231	−234	8	3	11	393	448	9	3	8	82−	−122
5	8	2	145	−119	5	3	14	310	290	6	0	8	258	−175	7	8	3	273	238	7	2	17	397	−390	8	4	11	612	759	9	4	8	192	−238
5	9	2	337	301	5	4	14	57−	0	6	1	8	654	744	7	9	3	151	−152	7	3	17	92	76	8	5	11	140	−161	9	5	8	118	−150
5	1	3	1409	−1348	5	5	14	433	453	6	2	8	101−	101	7	0	4	2146	2416	7	0	18	156	133	8	6	11	40	−27	9	6	8	351	413
5	2	3	669	−633	5	6	14	95	112	6	3	8	515	−605	7	1	4	1111	−1172	7	1	18	28−	−13	8	7	11	30	44	9	7	8	102	101
5	3	3	1673	2093	5	1	15	696	−676	6	4	8	84−	87	7	2	4	1133	−1353	7	2	18	30−	30	9	0	12	1377	−1458	9	1	9	177	212
5	4	3	490	521	5	2	15	203	−176	6	5	8	347	−453	7	3	4	345	363	8	0	0	2692	−2379	8	1	12	66−	11	9	2	9	700	−811
5	5	3	899	−1175	5	3	15	690	737	6	6	8	91	−121	7	4	4	415	−470	8	1	0	334	251	8	2	12	522	588	9	3	9	134	−198
5	6	3	84	−108	5	4	15	208	201	6	7	8	162	184	7	5	4	497	591	8	2	0	1012	1006	8	3	12	96	125	9	4	9	697	843
5	7	3	61−	−15	5	5	15	572	−574	6	8	8	33−	3	7	6	4	720	830	8	3	0	233	219	8	4	12	81	116	9	5	9	83	125
5	8	3	203	−161	5	6	15	37	−61	6	1	9	213	232	7	7	4	347	−360	8	4	0	124	36	8	5	12	81	98	9	6	9	105	−113
5	9	3	441	369	5	0	16	231	−216	6	2	9	513	550	7	8	4	426	−455	8	5	0	216	206	8	6	12	548	−506	9	7	9	55	47
5	0	4	231	104	5	1	16	550	538	6	3	9	177	199	7	1	5	266	−329	8	6	0	764	−801	8	1	13	76	−65	9	0	10	1290	−1437
5	1	4	1742	1688	5	2	16	241	232	6	4	9	413	−502	7	2	5	315	−335	8	7	0	173	142	8	2	13	477	510	9	1	10	135	−178
5	2	4	543	492	5	3	16	168	−167	6	5	9	59−	47	7	3	5	56−	−8	8	8	0	502	505	8	3	13	184	−202	9	2	10	639	762
5	3	4	163	−179	5	4	16	85	90	6	6	9	188	234	7	4	5	138	154	8	1	1	524	−452	8	4	13	662	−735	9	3	10	144	149
5	4	4	148	105	5	5	16	434	−437	6	7	9	46−	6	7	5	5	134	−172	8	2	1	1443	1503	8	5	13	41	33	9	4	10	133	159
5	5	4	738	−888	5	1	17	434	419	6	8	9	81	53	7	6	5	66−	−13	8	3	1	532	−544	8	6	13	95	104	9	5	10	148	153
5	6	4	140	−153	5	2	17	53−	63	6	0	10	494	−456	7	7	5	66−	−15	8	4	1	1326	−1442	8	0	14	929	901	9	6	10	593	−572
5	7	4	459	467	5	3	17	310	−318	6	1	10	490	490	7	8	5	142	−132	8	5	1	233	237	8	1	14	260	−239	9	7	10	88	−114
5	8	4	174	156	5	4	17	91	−88	6	2	10	194	164	7	0	6	280	253	8	6	1	61−	54	8	2	14	160	−153	9	1	11	112	−110
5	9	4	130	−100	5	0	18	63	69	6	3	10	108	130	7	1	6	197	−171	8	7	1	51−	−28	8	3	14	64	43	9	2	11	404	461
5	1	5	865	795	5	1	18	237	−235	6	4	10	79−	−45	7	2	6	103−	−62	8	8	1	137	111	8	4	14	145	−122	9	3	11	70−	30
5	2	5	98−	81	5	2	18	42−	2	6	5	10	123	149	7	3	6	94−	38	8	0	2	1635	1437	8	5	14	66	33	9	4	11	393	−408
5	3	5	943	−1019	5	3	18	184	168	6	6	10	95	−149	7	4	6	87−	49	8	1	2	187	−196	8	1	15	92	106	9	5	11	66	−89
5	4	5	138	−131	5	1	19	51	−50	6	7	10	220	241	7	5	6	66−	22	8	2	2	95−	5	8	2	15	56−	−2	9	6	11	35−	−49
5	5	5	637	739	5	2	19	27−	26	6	8	10	96	96	7	6	6	63−	42	8	3	2	85−	11	8	3	15	45−	−37	9	0	12	203	194
5	6	5	113	125	6	0	0	2469	1957	6	1	11	82−	37	7	7	6	113	−116	8	4	2	94	−128	8	4	15	188	199	9	1	12	71−	−63
5	7	5	57−	−63	6	1	0	2343	−1996	6	2	11	677	740	7	8	6	42−	−36	8	5	2	114	−66	8	5	15	83	93	9	2	12	156	162
5	8	5	51	48	6	2	0	422	−390	6	3	11	860	−998	7	1	7	84−	15	8	6	2	506	491	8	0	16	179	159	9	3	12	64−	−44
5	9	5	242	−217	6	3	0	523	−500	6	4	11	718	−826	7	2	7	266	268	8	7	2	269	−288	8	1	16	59	−72	9	4	12	61	89
5	0	6	359	224	6	4	0	201	−232	6	5	11	220	262	7	3	7	119	−140	8	8	2	256	−260	8	2	16	242	−243	9	5	12	34−	−17
5	1	6	612	−474	6	5	0	390	362	6	6	11	149	179	7	4	7	98	29	8	1	3	513	473	8	3	16	49	83	9	6	12	95	66
5	2	6	99−	−21	6	6	0	642	660	6	7	11	35−	31	7	5	7	63−	49	8	2	3	319	300	8	4	16	63	−94	9	1	13	59−	−1
5	3	6	233	224	6	7	0	919	−898	6	0	12	662	613	7	6	7	63−	43	8	3	3	397	−396	8	1	17	130	−108	9	2	13	133	118
5	4	6	81−	70	6	8	0	269	−232	6	1	12	1022	−1032	7	7	7	59−	−32	8	4	3	137	197	8	2	17	359	−341	9	3	13	87−	32
5	5	6	198	234	6	9	0	133	119	6	2	12	114	−120	7	8	7	77	−57	8	5	3	297	302	8	3	17	147	−159	9	4	13	55	85
5	6	6	63−	40	6	1	1	321	−188	6	3	12	73−	−71	7	0	8	1497	−1529	8	6	3	210	212	8	0	18	576	−536	9	5	13	59	47
5	7	6	98	−90	6	2	1	1204	−1098	6	4	12	68−	−91	7	1	8	759	812	8	7	3	131	−126	8	1	18	67	98	9	0	14	608	506
5	8	6	40−	−14	6	3	1	1785	1860	6	5	12	237	257	7	2	8	623	751	8	8	3	158	141	9	1	1	74−	−35	9	1	14	180	192
5	9	6	112	91	6	4	1	1153	1206	6	6	12	298	309	7	3	8	158	−194	8	0	4	98	−25	9	2	1	483	480	9	2	14	540	−548
5	1	7	349	331	6	5	1	428	−439	6	7	12	573	−545	7	4	8	135	192	8	1	4	194	−174	9	3	1	90−	−61	9	3	14	49−	−9
5	2	7	422	501	6	6	1	147	−192	6	1	13	113	−112	7	5	8	140	−200	8	2	4	689	−712	9	4	1	363	−375	9	4	14	216	−176
5	3	7	1023	−1161	6	7	1	67−	−85	6	2	13	567	−555	7	6	8	398	−582	8	3	4	85−	−39	9	5	1	62−	27	9	5	14	24−	−18
5	4	7	81−	−110	6	8	1	162	−112	6	3	13	714	734	7	7	8	262	344	8	4	4	245	−258	9	6	1	110	121	9	1	15	83	54
5	5	7	561	703	6	9	1	212	151	6	4	13	545	551	7	8	9	345	335	8	5	4	293	311	9	7	1	55−	−34	9	2	15	687	−649
5	6	7	98	81	6	0	2	1514	−1298	6	5	13	249	−257	7	1	9	145	121	8	6	4	367	388	9	8	1	242	170	9	3	15	244	−249
5	7	7	98	−111	6	1	2	1579	1478	6	6	13	77	−90	7	2	9	766	962	8	7	4	73	98	9	0	2	1799	1572	9	4	15	547	514
5	8	7	52	60	6	2	2	761	722	6	0	14	808	−737	7	3	9	641	−645	8	8	4	188	−186	9	1	2	550	473	9	0	16	344	−316
5	0	8	534	426	6	3	2	219	220	6	1	14	605	568	7	4	9	826	−1143	8	1	5	359	−380	9	2	2	1015	−1050	9	1	16	35−	−34
5	1	8	1215	−1253	6	4	2	84−	52	6	2	14	447	437	7	5	9	242	334	8	2	5	1139	−1274	9	3	2	135	−122	9	2	16	372	341
5	2	8	299	−368	6	5	2	63−	−38	6	3	14	67−	−9	7	6	9	226	269	8	3	5	393	−438	9	4	2	417	−384	9	3	16	99	−86
5	3	8	87−	20	6	6	2	402	−410	6	4	14	54	96	7	7	9	50−	31	8	4	5	676	613	9	5	2	113	−141	9	1	17	24−	−20
5	4	8	113	−164	6	7	2	452	437	6	5	14	87	−83	7	8	9	164	154	8	5	5	76	103	9	6	2	651	610	9	2	17	266	382
5	5	8	366	454	6	8	2	299	275	6	6	14	316	−280	7	0	10	803	838	8	6	5	348	−399	9	7	2	179	172	10	0	0	2102	1667
5	6	8	101	115	6	9	2	32−	25	6	1	15	74	−23	7	1	10	187	−236	8	7	5	48−	−44	9	8	2	452	−385	10	1	0	534	507
5	7	8	434	−519	6	1	3	79−	−88	6	2	15	66	−66	7	2	10	270	−337	8	8	5	271	−248	9	1	3	170	96	10	2	0	764	−745
5	8	8	88	−83	6	2	3	409	−434	6	3	15	327	−307	7	3	10	152	−175	8	0	6	1229	−1331	9	2	3	1054	−1120	10	3	0	109	−93
5	1	9	405	−436	6	3	3	411	−479	6	4	15	48−	24	7	4	10	63−	−76	8	1	6	543	571	9	3	3	269	−238	10	4	0	76−	−19
5	2	9	259	−314	6	4	3	373	414	6	5	15	106	80	7	5	10	53−	−6	8	2	6	1032	1260	9	4	3	1146	1226	10	5	0	271	−254
5	3	9	1064	1401	6	5	3	62−	−46	6	0	16	206	−179	7	6	10	337	392	8	3	6	328	−392	9	5	3	76	73	10	6	0	801	795
5	4	9	237	298	6	6	3	180	−224	6	1	16	262	226	7	7	10	186	−204	8	4	6	216	269	9	6	3	417	−433	10	7	0	231	211
5	5	9	476	−592	6	7	3	66−	−20	6	2	16	226	222	7	1	11	60−	−6	8	5	6	262	−314	9	7	3	55−	38	10	8	0	568	−522
5	6	9	56−	−41	6	8	3	49−	−28	6	3	16	186	−185	7	2	11	604	−639	8	6	6	618	−705	9	8	3	290	−251	10	1	1	347	289
5	7	9	44	−91	6	9	3	106	79	6	4	16	125	36	7	3	11	586	654	8	7	6	42−	45	9	0	4	1479	−1340	10	2	1	313	−412
5	8	9	63	−60	6	0	4	1108	−1000	6	5	16	187	−207	7	4	11	659	741	8	8	6	340	354	9	1	4	315	−330	10	3	1	644	−623
5	0	10	827	−764	6	1	4	864	826	6	1	17	397	404	7	5	11	258	−289	8	1	7	382	416	9	2	4	529	551	10	4	1	798	782
5	1	10	1547	1726	6	2	4	623	661	6	2	17	202	210	7	6	11	122	−132	8	2	7	957	1236	9	3	4	110	−78	10	5	1	406	415
5	2	10	112−	29	6	3	4	620	−711	6	3	17	305	−295	7	7	11	34−	19	8	3	7	70	93	9	4	4	321	340	10	6	1	87	102
5	3	10	326	−301	6	4	4	123	167	6	4	17	92	−85	7	0	12	369	−289	8	4	7	676	−865	9	5	4	113	163	10	7	1	40−	5
5	4	10	129	159	6	5	4	451	−552	6	0	18	501	424	7	1	12	302	273	8	5	7	53−	29	9	6	4	528	−534	10	8	1	22−	−17
5	5	10	408	−459	6	6	4	241	−306	6	1	18	341	−337	7	2	12	84	88	8	6	7	319	414	9	7	4	135	−123	10	0	2	1122	−928
5	6	10	178	−220	6	7	4	130	103	6	2	18	363	−333	7	3	12	79−	32	8	7	7	40−	−10	9	8	4	191	191	10	1	2	696	−634
5	7	10	665	770	6	8	4	151	135	6	3	18	153	154	7	4	12	67−	−35	8	8	7	313	286	9	1	5	240	−222	10	2	2	71−	−48
5	8	10	80	60	6	9	4	158	−172	6	1	19	237	−276	7	5	12	45−	−14	8	0	8	436	448	9	2	5	746	751	10	3	2	177	198
5	1	11	76−	63	6	1	5	1103	1114	7	1	1	497	−397	7	6	12	188	−191	8	1	8	328	359	9	3	5	555	562	10	4	2	280	−258
5	2	11	333	385	6	2	5	551	561	7	2	1	1200	−1209	7	7	12	124	132	8	2	8	642	−737	9	4	5	687	−728	10	5	2	302	308
5	3	11	657	−682	6	3	5	631	−704	7	3	1	1068	1087	7	1	13	286	−263	8	3	8	83−	−70	9	5	5	251	−244	10	6	2	270	−245

Table 3 (*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	$10F_o$	$10F_c$
10	7	2	192	−231	10	5	11	316	−311	11	4	6	73−	38	12	0	2	738	557	12	1	13	206	−204	13	2	11	48−	−65	15	1	1	70	95
10	8	2	147	140	10	6	11	53	−58	11	5	6	51−	3	12	1	2	747	627	12	2	13	42−	−25	13	3	11	520	415	15	2	1	325	301
10	1	3	72−	12	10	0	12	1121	1041	11	6	6	72	−54	12	2	2	152	155	12	3	13	605	526	13	0	12	113	97	15	3	1	56	−79
10	2	3	140	91	10	1	12	334	340	11	7	6	33−	18	12	3	2	81−	−11	12	0	14	373	312	13	1	12	69	62	15	4	1	317	−290
10	3	3	166	−157	10	2	12	390	−365	11	1	7	188	200	12	4	2	67−	53	12	1	14	382	434	13	2	12	37−	4	15	0	2	474	369
10	4	3	833	−813	10	3	12	55−	−47	11	2	7	94−	−76	12	5	2	245	−222	13	1	1	231	−205	13	1	13	199	−157	15	1	2	505	−479
10	5	3	52−	43	10	4	12	101	−102	11	3	7	96	−84	12	6	2	98	106	13	2	1	88−	43	14	0	0	621	−468	15	2	2	223	−217
10	6	3	51−	−30	10	5	12	192	−194	11	4	7	323	341	12	1	3	108	−109	13	3	1	447	419	14	1	0	1144	1150	15	3	2	195	167
10	7	3	39−	2	10	1	13	203	197	11	5	7	46	−73	12	2	3	96−	112	13	4	1	120	−107	14	2	0	529	475	15	4	2	145	−140
10	8	3	88	−87	10	2	13	312	−314	11	6	7	40	−35	12	3	3	80−	77	13	5	1	191	−156	14	3	0	62−	3	15	1	3	264	−258
10	0	4	558	−439	10	3	13	291	−318	11	7	7	57	79	12	4	3	66−	44	13	6	1	67	65	14	4	0	205	195	15	2	3	524	−484
10	1	4	472	−409	10	4	13	642	591	11	0	8	873	−843	12	5	3	162	−156	13	0	2	691	−495	14	5	0	377	−333	15	3	3	450	400
10	2	4	918	935	10	5	13	188	185	11	1	8	877	−824	12	6	3	260	261	13	1	2	960	893	14	1	1	57−	−11	15	4	3	516	454
10	3	4	83−	−44	10	0	14	548	−490	11	2	8	170	207	12	0	4	730	−564	13	2	2	391	337	14	2	1	433	429	15	0	4	905	−721
10	4	4	184	192	10	1	14	348	−311	11	3	8	177	206	12	1	4	421	356	13	3	2	231	−223	14	3	1	582	−588	15	1	4	147	134
10	5	4	127	114	10	2	14	83	−78	11	4	8	66	72	12	2	4	141	−155	13	4	2	59−	34	14	4	1	216	−187	15	2	4	382	352
10	6	4	259	−255	10	3	14	90	107	11	5	8	310	340	12	3	4	79−	−19	13	5	2	458	−392	14	5	1	186	174	15	3	4	44−	−40
10	7	4	105	−106	10	4	14	27−	−15	11	6	8	261	−246	12	4	4	66−	−74	13	6	2	215	−197	14	0	2	284	168	15	4	4	45	70
10	1	5	166	−180	10	1	15	75	−71	11	1	9	463	−453	12	5	4	305	−307	13	1	3	675	563	14	1	2	340	−315	15	1	5	140	149
10	2	5	1169	1257	10	2	15	61	75	11	2	9	273	274	12	6	4	34−	15	13	2	3	88	109	14	2	2	180	−147	15	2	5	163	122
10	3	5	376	361	10	3	15	66	56	11	3	9	664	936	12	1	5	519	504	13	3	3	672	−654	14	3	2	76	−108	15	3	5	203	−205
10	4	5	591	−595	10	0	16	61	7	11	4	9	422	−433	12	2	5	252	−210	13	4	3	114	−132	14	4	2	102	−109	15	4	5	145	−152
10	5	5	215	−220	10	1	16	176	−150	11	5	9	350	−434	12	3	5	470	−493	13	5	3	503	425	14	5	2	122	−59	15	0	6	504	46
10	6	5	340	366	10	2	16	192	170	11	6	9	42	59	12	4	5	94	−119	13	6	3	26−	−9	14	1	3	56−	40	15	1	6	57	−77
10	7	5	34−	−34	11	1	1	444	379	11	0	10	628	540	12	5	5	469	454	13	0	4	236	−59	14	2	3	74−	−125	15	2	6	49−	−15
10	0	6	444	369	11	2	1	91	−93	11	1	10	498	459	12	6	5	98	−97	13	1	4	685	−638	14	3	3	428	417	15	3	6	74	61
10	1	6	1124	1100	11	3	1	662	−656	11	2	10	199	−195	12	0	6	1200	−1136	13	2	4	194	177	14	4	3	45−	−6	15	4	6	22−	−20
10	2	6	451	−511	11	4	1	137	136	11	3	10	67−	37	12	1	6	1065	−1097	13	3	4	145	165	14	5	3	124	−106	15	1	7	74	73
10	3	6	65	−116	11	5	1	417	427	11	4	10	55−	−47	12	2	6	601	559	13	4	4	56−	2	14	0	4	286	253	15	2	7	94	82
10	4	6	260	−319	11	6	1	125	−110	11	5	10	187	−196	12	3	6	323	326	13	5	4	401	348	14	1	4	530	−471	15	3	7	262	−353
10	5	6	341	−441	11	7	1	49−	−17	11	6	10	220	237	12	4	6	61−	12	13	6	4	23−	−13	14	2	4	73−	−59	15	0	8	578	444
10	6	6	307	384	11	0	2	695	−729	11	1	11	174	161	12	5	6	572	544	13	1	5	355	−351	14	3	4	270	262	15	1	8	230	−207
10	7	6	355	351	11	1	2	426	−361	11	2	11	220	−216	12	6	6	190	−167	13	2	5	241	−173	14	4	4	44−	13	15	2	8	304	−255
10	1	7	276	329	11	2	2	454	450	11	3	11	477	−450	12	1	7	558	−542	13	3	5	428	420	14	5	4	312	293	15	3	8	23−	−19
10	2	7	802	−1066	11	3	2	93−	78	11	4	11	285	278	12	2	7	176	198	13	4	5	252	276	14	1	5	552	−492	15	1	9	63	−55
10	3	7	494	−640	11	4	2	202	225	11	5	11	254	223	12	3	7	597	643	13	5	5	344	−299	14	2	5	221	−224	15	2	9	461	−395
10	4	7	451	558	11	5	2	227	220	11	0	12	92	−39	12	4	7	56−	5	13	6	5	77	−69	14	3	5	487	470	15	0	10	362	−290
10	5	7	219	314	11	6	2	246	−238	11	1	12	348	−283	12	5	7	458	−450	13	0	6	146	−81	14	4	5	412	404	16	0	0	548	475
10	6	7	247	−348	11	7	2	264	−254	11	2	12	153	−142	12	6	7	178	162	13	1	6	118	110	14	5	5	472	−471	16	1	0	212	−193
10	7	7	56	45	11	1	3	635	−587	11	3	12	52−	−29	12	0	8	338	−275	13	2	6	80−	1	14	0	6	591	−462	16	2	0	265	−235
10	0	8	773	−645	11	2	3	443	414	11	4	12	147	−135	12	1	8	288	297	13	3	6	62−	18	14	1	6	259	228	16	3	0	33−	−28
10	1	8	147	−184	11	3	3	969	955	11	5	12	145	126	12	2	8	128	−121	13	4	6	49−	−29	14	2	6	72	82	16	1	1	103	122
10	2	8	799	857	11	4	3	493	−443	11	1	13	145	155	12	3	8	120	−86	13	5	6	69	−76	14	3	6	119	−112	16	2	1	441	−423
10	3	8	74−	−28	11	5	3	483	−497	11	2	13	80	−97	12	4	8	153	−157	13	6	6	15−	−10	14	4	6	38−	−1	16	3	1	261	250
10	4	8	249	317	11	6	3	172	178	11	3	13	366	−347	12	5	8	294	−259	13	1	7	226	−192	14	5	6	323	−329	16	0	2	569	−451
10	5	8	36	61	11	7	3	35	−16	11	4	13	49	−27	12	6	8	15	22	13	2	7	216	−221	14	1	7	422	420	16	1	2	35−	62
10	6	8	251	−266	11	0	4	921	699	11	0	14	162	−130	12	1	9	179	178	13	3	7	377	387	14	2	7	191	198	16	2	2	273	214
10	7	8	79	−99	11	1	4	1353	1241	11	1	14	363	−293	12	2	9	74−	−54	13	4	7	155	186	14	3	7	382	−346	16	3	2	108	102
10	1	9	108	−109	11	2	4	384	−368	11	2	14	326	282	12	3	9	134	−123	13	5	7	230	−200	14	4	7	387	−338	16	1	3	265	−264
10	2	9	83−	−36	11	3	4	387	−396	11	3	14	77	74	12	4	9	46−	−29	13	0	8	95	83	14	0	8	413	336	16	2	3	91	−99
10	3	9	206	251	11	4	4	168	−156	11	1	15	201	−183	12	5	9	57	71	13	1	8	473	505	14	1	8	485	−470	16	3	3	91	−88
10	4	9	325	338	11	5	4	544	−527	11	2	15	354	348	12	0	10	294	243	13	2	8	69−	20	14	2	8	160	−139	16	0	4	191	−181
10	5	9	87	−91	11	6	4	290	276	12	0	0	544	−357	12	1	10	241	254	13	3	8	164	−130	14	3	8	298	263	16	1	4	83−	−4
10	6	9	156	139	11	7	4	619	520	12	1	0	1057	−1057	12	2	10	153	140	13	4	8	41−	22	14	4	8	27−	−41	16	2	4	158	130
10	0	10	572	−551	11	1	5	144	166	12	2	0	306	−288	12	3	10	66	76	13	5	8	213	−196	14	1	9	140	−136	16	3	4	63	−65
10	1	10	269	−270	11	2	5	511	−438	12	3	0	83−	−51	12	4	10	41−	54	13	1	9	181	202	14	2	9	48−	61	16	1	5	231	220
10	2	10	79−	62	11	3	5	155	−151	12	4	0	109	−85	12	5	10	90	−62	13	2	9	124	95	14	3	9	167	−152	16	2	5	297	301
10	3	10	66	63	11	4	5	326	315	12	5	0	328	307	12	1	11	116	135	13	3	9	626	−633	14	4	9	17−	25	16	0	6	832	776
10	4	10	169	−173	11	5	5	127	111	12	6	0	36−	22	12	2	11	109	−107	13	4	9	53	51	14	0	10	62−	51	16	1	6	166	−134
10	5	10	96	117	11	6	5	129	−130	12	1	1	242	−238	12	3	11	676	−673	13	0	10	148	221	14	1	10	333	−286	16	2	6	483	−465
10	6	10	209	−187	11	7	5	98	86	12	2	1	183	174	12	4	11	321	274	13	1	10	664	−655	14	2	10	129	−108	16	1	7	216	−211
10	1	11	244	−236	11	0	6	118−	76	12	3	1	1095	1081	12	0	12	106	−231	13	2	10	270	−281	14	3	10	77	−58	17	1	1	80	−62
10	2	11	265	276	11	1	6	67	14	12	4	1	360	−363	12	1	12	652	−603	13	3	10	77	78	14	1	11	59	51	17	0	2	383	−379
10	3	11	483	501	11	2	6	147	170	12	5	1	543	−538	12	2	12	101	−105	13	4	10	119	−91	14	2	11	198	−192	17	1	2	197	−190
10	4	11	438	−444	11	3	6	84−	−34	12	6	1	84	−103	12	3	12	38−	34	13	1	11	57	−57	14	0	12	349	−314	17	0	4	506	530

Table 4. *Distances and angles in the cadmium coordination sphere*

Cd–S(2)	2·638 (4) Å	S(2) Cd S(2′)	101·9 (0·1)°
Cd–S(2′)	2·647 (4)	S(2) Cd O(1)	89·8 (0·4)
Cd–O(1)	2·31 (1)	S(2) Cd O(2)	87·2 (0·4)
Cd–O(2)	2·27 (2)	S(2) Cd O(3)	103·7 (0·5)
Cd–O(3)	2·29 (2)	S(2′) Cd O(1)	94·7 (0·4)
Cd–O(6′)	2·27 (2)	S(2′) Cd O(3)	78·4 (0·5)
		S(2′) Cd O(6′)	93·4 (0·5)
		O(1) Cd O(2)	96·6 (0·6)
		O(1) Cd O(6′)	84·9 (0·6)
		O(2) Cd O(3)	88·6 (0·7)
		O(2) Cd O(6′)	78·6 (0·7)
		O(3) Cd O(6′)	83·1 (0·7)

Table 5. *Bond distances and angles*

In the sulphate group			
S(1)–O(3)	1·48 (2) Å	O(3)S(1)O(4)	108·0 (1·1)°
S(1)–O(4)	1·46 (2)	O(3)S(1)O(5)	111·3 (1·1)
S(1)–O(5)	1·47 (2)	O(3)S(1)O(6)	108·1 (1·1)
S(1)–O(6)	1·44 (2)	O(4)S(1)O(5)	109·7 (0·9)
		O(4)S(1)O(6)	110·0 (1·1)
		O(5)S(1)O(6)	109·8 (1·1)
In thiourea			
S(2) –C	1·76 (1) Å	S(2)CN(1)	115·5 (1·5)°
N(1)–C	1·31 (3)	S(2)CN(2)	120·6 (1·3)
N(2)–C	1·26 (2)	N(1)CN(2)	123·9 (1·8)

ficantly different and the same can be said for the two S–C–N angles.

The angles around S(2):

CdS(2)C	102·2 (0·5)°
Cd″S(2)C	114·6 (0·5)
CdS(2)Cd″	105·7 (0·1) ,

indicate a distorted tetrahedral environment; the fourth direction involves a S(2)···O(1‴) = 3·48 (2) Å contact with the following angles:

CdS(2)O(1‴)	103·8 (0·3)°
Cd″S(2)O(1‴)	109·2 (0·3)
CS(2)O(1‴)	119·5 (0·5) .

The $H_2O(1)$ molecule forms two contacts which can be considered as hydrogen bonds with the oxygen atoms of two SO_4^{2-} groups:

O(1)···O(5′)	2·78 (2) Å
O(1)···O(5^{v})	2·72 (2) ;

the angle O(5′)O(1)O(5^{v}) = 150·8 (0·8)°, indicating a mean angular displacement of about 20° of the O–H line from the O···O direction. These two contacts are almost coplanar with the Cd–O(1) bond. Also the $H_2O(2)$ molecule forms two hydrogen bonds with two oxygen atoms from two SO_4^{2-} tetrahedra:

O(2)···O(3″)	= 2·71 (3) Å
O(2)···O(4^{viii})	= 2·68 (2) ;

the angle O(3″)O(2)O(4^{viii}) = 124·6 (0·9)° and the bonds almost lie within the same plane with the Cd–O(2) bond.

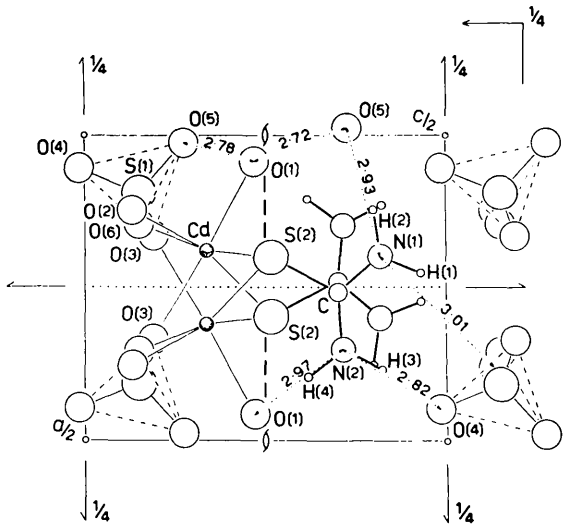

Fig. 1. Diagrammatic projection of the structure on (010).

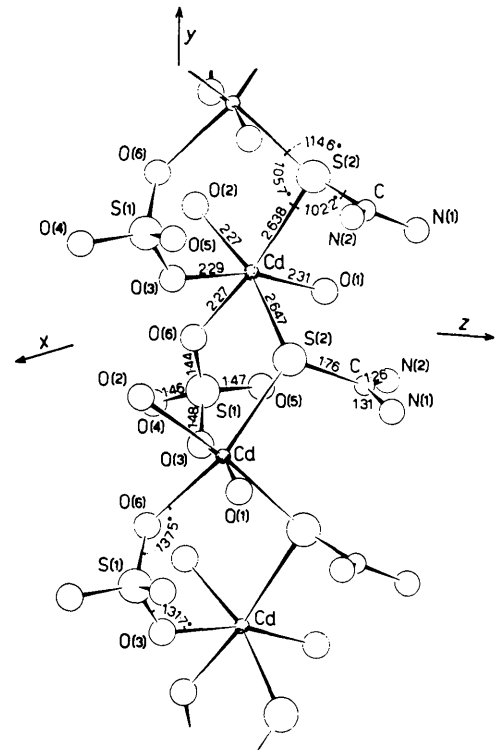

Fig. 2. Clinographic projection of a chain of coordination polyhedra.

Table 6. *Calculated hydrogen fractional coordinates for thiourea*

	x	*y*	*z*
H(1)	0·227	0·288	0·465
H(2)	0·123	0·336	0·400
H(3)	0·385	0·345	0·412
H(4)	0·396	0·433	0·309

For the thiourea molecule it is possible to deduce reasonable coordinates for the H atoms (Table 6) as indicated in the previous section. The contacts, concerning the two $-NH_2$ groups, which can be considered as hydrogen bonds, are:

N(1)···O(2^{vii})	3·01 (3) Å	H(1)N(1)O(2^{vii})	42·1°
N(1)···O(5^{v})	2·93 (3)	H(2)N(1)O(5^{v})	6·9
N(2)···O(4^{vii})	2·82 (3)	H(3)N(2)O(4^{vii})	9·0
N(2)···O(1″)	2·97 (2)	H(4)N(2)O(1′)	16·4

The other packing distances of 3·5 Å or less are:

S(2)···O(3″)	3·13 (2) Å	O(3)···O(6′)	3·02 (3) Å
S(2)···O(1‴)	3·48 (2)	O(3)···N(1^{ix})	3·50 (3)
S(2)···O(5^{v})	3·50 (2)	O(4)···O(6^{x})	3·46 (3)
O(1)···O(6′)	3·09 (2)	N(1)···O(6^{vi})	3·13 (3) .

All the calculations were performed on the Olivetti Elea 6001/S computer of the Centro di Calcolo Elettronico of the University of Parma, using the programs of Nardelli, Musatti, Domiano & Andreetti (1964, 1965). The authors are indebted to the Consiglio Nazionale delle Ricerche for financial support.

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The Crystal Structure of *p*-Aminobenzoic Acid*

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The crystal structure of a monoclinic modification of *p*-aminobenzoic acid has been determined from three-dimensional X-ray diffraction data. The unit-cell dimensions are: $a=18{\cdot}551$, $b=3{\cdot}860$, $c=18{\cdot}642$ Å, $\beta=93{\cdot}56°$; the space group is $P2_1/n$. There are 8 molecules in the unit cell, and hence two in the asymmetric unit. The structure was refined by least-squares methods to an R index, for 1916 observed reflections, of 0·073 and a goodness of fit of 1·29; the resulting standard deviations in the bond distances are 0·006 Å. The dimensions of the two structurally distinct molecules are closely similar, and suggest a small amount of quinoid character. The amino and carboxyl groups are displaced slightly from the planes of the benzene rings, and the nitrogen atoms are non-planar. Pairs of molecules are linked together, to form dimers, through two O–H···O hydrogen bonds arranged about a center of symmetry; an additional N–H···O hydrogen bond is formed by one of the two kinds of molecule. Twinning and disorder are common for these crystals. In the disordered structure, which is based on an orthorhombic unit cell half as large as the monoclinic cell, the hydrogen-bonded dimers apparently remain intact but the arrangement of N–H···O bonds becomes random.

Introduction

Preliminary X-ray diffraction photographs of crystals of *p*-aminobenzoic acid, $NH_2C_6H_4CO_2H$, indicated an interesting combination of twinning and disorder. Although the crystals are monoclinic, the a and c axes are very nearly equal in length, leading to an approximately orthogonal cell bounded by (101), (10$\bar{1}$), and (010). Zero-level Weissenberg photographs about **b** show almost exact *mm* symmetry, a reflection $h0l$ having essentially the same intensity (and spacing) as the corresponding reflection $l0h$. In addition, twinning about (101) or (10$\bar{1}$) is common, as evidenced by increased symmetry of upper-level photographs and by a slight splitting of the high-angle reflections. Finally, extensive streaking along alternate reciprocal lattice rows of upper-level Weissenberg photographs about **b** indicates severe disorder for many crystals; in the limiting case, when these lattice rows become continua, the intensities of the remaining spots show orthorhombic symmetry corresponding to the space group *Pnma* and a unit cell half as large as the monoclinic cell. In view of these interesting observations and because of the importance of *p*-aminobenzoic acid in certain biological processes, we have undertaken the present investigation.

During the course of this investigation, the work of Killean, Tollin, Watson & Young (1965; hereafter, KTWY) was reported. They have apparently observed the same sort of twinning and disorder; and after suitable transformation of axes, the two-dimensional structure they report is in satisfactory agreement with our results.

Experimental

Our crystals of *p*-aminobenzoic acid were obtained by evaporation of aqueous ethanol and methanol solutions. They grow in the form of long, fibrous needles similar to modification I found by KTWY; we have not obtained the other forms reported by them, nor that reported by Prasad, Kapadia & Thakar (1937)

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