is too small in comparison to the 4 Å required for two methyl-groups in contact.

In Ni-dimethyl-glyoxime, Godycki & Rundle (1953) have found an exceptionally short hydrogen bond of 2·44 Å. Recently Williams, Wohlauer & Rundle (1959) in a least-squares refinement of the structure of the Ni-dimethyl-glyoxime have reduced this bond length to 2·40 Å. In the Ni-methyl-ethyl-glyoxime the hydrogen bond is a few hundredths of an Ångström shorter.

At present we are working on the structure of the Cu-methyl-ethyl-glyoxime to clarify the reasons for non-isomorphism of the two complexes.

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The Crystal Structure of *mono*-Thiourea-Lead(II) Acetate

BY MARIO NARDELLI, GIOVANNA FAVA AND GERMANO BRANCHI

Structural Chemistry Laboratory, Institute of Chemistry, University of Parma, Italy

mono-Thiourea-lead(II) acetate, $Pb[SC(NH_2)_2](C_2H_3O_2)_2$, is monoclinic,

$$a = 4{\cdot}55, \ b = 15{\cdot}81, \ c = 14{\cdot}28 \ \text{Å}, \ \beta = 106{\cdot}4°;$$

the space group is $P2_1/c$, and there are four formula units in the unit cell. The structure was determined by X-ray analysis, the final parameters being refined from $F_o - F_c$ syntheses of $0kl$, $1kl$ and $2kl$ data (856 observed reflections). The structure consists of chains of coordination polyhedra joined by sulphur atoms, each forming two coordinative bonds of 3·10 Å with adjacent Pb atoms. These chains, running along [100], are linked in pairs through weaker Pb–S bonds (3·34 Å). The coordination polyhedra can be described as distorted pentagonal bipyramids with three sulphur and four oxygen atoms at the vertices.

Introduction

Lead(II) acetate forms two different coordination compounds with thiourea $(SC(NH_2)_2 = tu)$: $Pbtu(C_2H_3O_2)_2$ and $Pbtu_6(C_2H_3O_2)_2$. Crystals suitable for X-ray analysis can be grown from aqueous solutions (Nardelli & Chierici, 1959). A short account of the structure of *mono*-thiourea-lead(II) acetate has been given previously (Nardelli & Fava, 1959a), in which the orientation and lengths of the Pb–S bonds were reported only. The present paper gives a full account of this structure, including the positions of the light C, N and O atoms, which were located satisfactorily in spite of the presence of the heavy Pb atom.

Crystal data

$Pbtu(C_2H_3O_2)_2$: colourless monoclinic needles, elongated along [100].

$$a = 4{\cdot}55 \pm 0{\cdot}01, \ b = 15{\cdot}81 \pm 0{\cdot}03, \ c = 14{\cdot}28 \pm 0{\cdot}07 \ \text{Å},$$
$$\beta = 106{\cdot}4 \pm 0{\cdot}1°.$$

The unit cell ($V = 986 \ \text{Å}^3$) contains four formula units (density: observed 2·66, calculated 2·70 g.cm.$^{-3}$). Space group: $P2_1/c$. Absorption coefficient for Cu $K\alpha$ radiation: $\mu = 358$ cm.$^{-1}$.

Experimental

Multiple-film integrated (Wiebenga & Smits, 1950) and non-integrated equi-inclination Weissenberg photographs (Cu $K\alpha$ radiation) were taken of the $0kl$, $1kl$ and $2kl$ layers, using a slender, nearly cylindrical crystal (mean radius: 0·003 cm.). In addition, the $h0l$ reflections were recorded using an irregular fragment cut from one of the larger needles. The intensities were determined photometrically. Absorption in the $0kl$,

$1kl$ and $2kl$ reflections was corrected, at first, as for cylindrical samples. The $h0l$ reflections were not corrected for absorption because of the irregularity of the sample; these reflections were used to give a rough initial x coordinate for Pb and were not taken into account in the final refinement.

The structure factors were derived after making the usual corrections for Lorentz and polarization factors and, in the case of non-equatorial layers, for the spot shape. The $F_o(0kl)$'s were put on an absolute scale by Wilson's (1942) method, and this method gave an overall isotropic temperature factor of $B = 2 \cdot 5$ Å².

In the photographs taken by rotation around [100] 183 $0kl$, 363 $1kl$ and 310 $2kl$ reflections (out of 276, 541 and 484 possible reflections) were observed.

Structure analysis

The initial coordinates for Pb were obtained from $P(V, W)$ and $P(U, W)$ Patterson projections. A first $\varrho_0(Y, Z)$ projection, calculated using the $F_o(0kl)$'s with the signs of the Pb contributions, gave the y and z coordinates of S. The $R(0kl) = \Sigma||F_o| - |F_c||/\Sigma|F_o|$
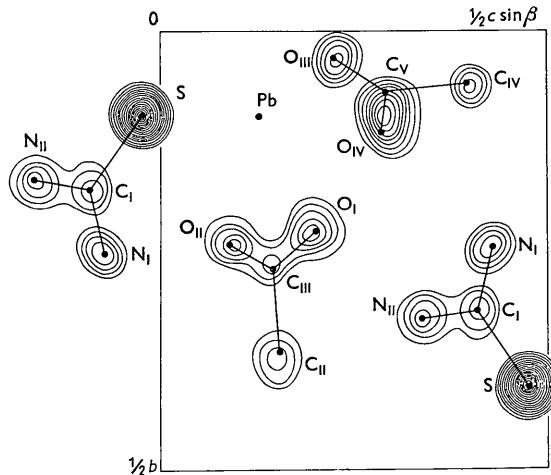
index at this stage, with the contributions of Pb and S only, was 0·195.

The x coordinate of the S atom was deduced from the sine, $S_1(Y, Z)$, and cosine, $C_1(Y, Z)$, components of the $|\varrho_1(Y, Z)|$ generalized projection, calculated with the signs of the Pb contributions of the $F_o(1kl)$'s. The light atoms were located using the $\varrho_0(Y, Z)$ and $|\varrho_1(Y, Z)|$ projections from which the Pb and S peaks had been removed. The y and z coordinates were then refined by several cycles of $(F_o - F_c)$ $0kl$-syntheses; the reliability index was now $R(0kl) = 0 \cdot 123$.
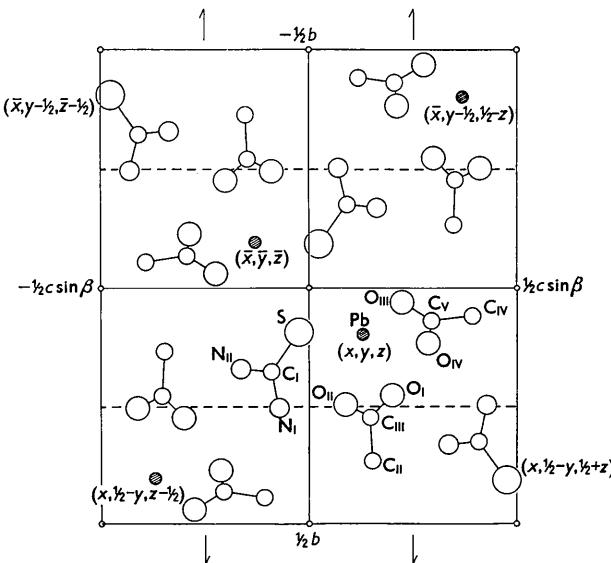
Fig. 2. Diagrammatic projection along [100].

Improved isotropic thermal factors for Pb, S and light atoms were next calculated by a least-squares method (Cochran, 1951). The B values obtained were: 2·6 for Pb, 3·5 for S, and 5·4 Å² for the light atoms.

Further refinement was carried out by examining the three-dimensional Fourier lines $\varrho(X, Y_i, Z_i)$ round each atomic centre; these were calculated from the $\varrho_0(Y, Z)$ and the $S_1(Y, Z)$, $C_1(Y, Z)$, $S_2(Y, Z)$, $C_2(Y, Z)$ data (the subscripts 0, 1, 2 refer to the $0kl$,

Fig. 1. Final $\varrho_0(Y, Z)$ projection; contours at intervals of 1 e.Å⁻² beginning from 4 e.Å⁻². Pb peak removed.

Table 1. Final atomic coordinates

	x	y	z	X'	Y'	Z'
Pb	0·3855	0·0972	0·1294	1·232 Å	1·537 Å	1·773 Å
S	0·7488	0·0930	−0·0239	3·503	1·470	−0·327
N_I	0·6739	0·2533	−0·0713	3·353	4·005	−0·977
N_{II}	0·9241	0·1697	−0·1639	4·866	2·683	−2·245
O_I	0·1632	0·2272	0·2009	−0·067	3·592	2·752
O_{II}	0·4098	0·2460	0·0878	1·511	3·889	1·203
O_{III}	0·0202	0·0311	0·2236	−0·810	0·492	3·063
O_{IV}	−0·2732	0·1158	0·2872	−2·401	1·831	3·934
C_I	0·7915	0·1768	−0·0908	3·967	2·795	−1·244
C_{II}	0·2330	0·3662	0·1531	0·443	5·790	2·097
C_{III}	0·2655	0·2722	0·1467	0·616	4·303	2·010
C_{IV}	0·1970	0·0580	0·3933	−0·690	0·917	5·387
C_V	−0·0223	0·0689	0·2950	−1·290	1·089	4·042

Considering the standard deviations, the fourth decimal figure for the light atoms is not significant; it is reported here because it was used in the calculations.

$1kl$ and $2kl$ reflections used in calculating these functions). The series-termination errors were corrected in each series by two cycles of difference syntheses. At the end of this refinement the reliability indices (observed reflections only) were:

$$R(0kl)=0{\cdot}106,\ R(1kl)=0{\cdot}089,\ R(2kl)=0{\cdot}092\ .$$

A few of the strongest $0kl$ and $1kl$ reflections showed F_c values higher than the observed ones. Correcting these for secondary extinction by the method of Pinnock, Taylor & Lipson (1956), the R values were:

$$R(0kl)=0{\cdot}071,\ R(1kl)=0{\cdot}079\ .$$

A further improvement was achieved by correcting for the discontinous absorption effects, following

Table 2. *Observed and calculated structure factors*

A minus sign for $|F_o|$ means 'less than'. A few reflections out of the angular field registered in the photographs are omitted in $|F_o|$ columns. Reflections corrected for secondary extinction are starred

l	$10\lvert F_o\rvert$	$10F_c$
	0 0 l	
2	436	−382
* 4	2550	−2581
6	427	413
8	1514	1760
10	262−	−257
12	1205	−1171
14	431	380
16	481	517
	0 1 l	
* 1	2301	2106
* 2	1810	−1655
3	1152	−1175
4	567	541
5	938	−944
6	1489	1564
7	1468	1538
8	207−	−150
9	727	727
10	780	−767
11	981	−1007
12	322	309
13	231−	−362
14	508	548
15	430	548
16	163−	−152
17	119−	122
	0 2 l	
0	632	566
* 1	2333	−2234
2	236	−169
3	956	−994
4	315	−264
* 5	1648	1736
6	213	237
7	1304	1419
8	861	854
9	916	−1032
10	232−	−130
11	640	−592
12	529	−492
13	1067	1074
14	232−	67
15	372	339
16	179−	194
17	484	−495
	0 3 l	
1	838	−812
* 2	2176	−2068
3	497	446
4	232	204
5	542	525
* 6	2476	2470
7	499	−479
8	204−	−356
9	216−	−165
10	1061	−1132
11	233−	176
12	597	581
13	228−	156
14	737	869
15	186−	−170
16	153	−209
17	109−	−37
	0 4 l	
* 0	2304	−2096
* 1	1852	−1940
2	137−	−52
3	1003	−1000
4	1469	1549
5	1406	1452
6	768	−694
7	1002	940
8	1407	−1459
9	701	−727
10	236−	341
11	244−	−271
12	911	868
13	627	653
14	222−	−199
15	196−	197
16	374	−409
17	243	−296
	0 5 l	
* 1	1931	−1983
2	443	−438
3	983	1054
4	423	329
5	1381	1319
6	196−	97
7	1840	−1911
8	222−	129
9	719	−718
10	246−	−95
11	1155	1112
12	253−	68
13	444	448
14	225−	48
15	598	−643
16	160−	−20
	0 6 l	
* 0	2969	−2970
1	576	603
2	337	−297
3	781	730
4	1565	1624
5	956	−925
6	303	−262
7	606	−549
8	1313	−1353
9	605	600
10	254−	207
11	258−	195
12	991	932
13	451	−458
14	273	−257
15	193−	−147
16	412	−446
	0 7 l	
1	902	−817
* 2	2199	2228
3	555	503
4	253	−289
5	256	282
6	1901	−2012
7	631	−616
8	235−	227
9	311	−310
10	828	871
11	446	426
12	238−	−325
13	222−	177
14	633	−686
15	167−	−252
16	123−	196
	0 8 l	
0	752	584
1	1566	1593
2	200−	−44
3	1153	1166
4	313	−315
5	1577	−1628
6	224−	−12
7	958	−961
8	239−	179
9	758	847
10	245−	−85
11	370	370
12	230−	−145
13	777	−833
14	207−	95
15	279	−279
16	100−	84
	0 9 l	
1	1028	1014
2	1018	1049
3	780	−786
4	221−	−139
5	648	−636
6	1270	−1250
7	839	848
8	250−	132
9	574	513
10	572	617
11	572	−599
12	306	−289
13	354	−305
14	553	−540
15	350	394
	0 10 l	
0	1747	1698
1	231−	11
2	231−	62
3	232−	248
4	1306	−1308
5	495	−395
6	475	415
7	245−	−191
8	1226	1272
9	244−	200
10	238−	−281
11	224−	8
12	795	−824
13	180−	−153
14	150−	205
15	106−	−65
	0 11 l	
1	1311	1319
2	757	−738
3	892	−943
4	238−	115
5	793	−741
6	539	557
7	1128	1108
8	250−	−92
9	437	467
10	364	−365
11	721	−696
12	204−	129
13	177−	−260
14	225	242
	0 12 l	
0	923	937
1	821	−881
2	232−	215
3	671	−659
4	635	−649
5	963	985
6	246−	125
7	658	617
8	482	498
9	733	−720
10	230−	−107
11	214−	−226
12	298	−308
13	557	555
14	111−	79
	0 13 l	
1	271−	−39
2	1199	−1206
3	266−	106
4	265−	198
5	263−	122
6	1178	1172
7	254−	−82
8	243−	−44
9	231−	−28
10	718	−721
11	191−	69
12	254	254
13	123−	1
	0 14 l	
0	892	−906
1	629	−629
2	252−	−19
3	454	−489
4	657	662
5	725	691
6	248−	−51
7	528	476
8	557	−536
9	428	−430
10	200−	115
11	175−	−166
12	312	340
	0 15 l	
1	845	−859
2	304	−310
3	743	740
4	245−	94
5	407	448
6	275	254
7	661	−673
8	212−	25
9	437	−386
10	173−	−128
11	470	505
	0 16 l	
0	904	−955
1	244−	248
2	243−	−16
3	241−	191
4	624	702
5	229−	−202
6	278	−214
7	207−	−140
8	707	−679
9	169−	183
10	200	164
	0 17 l	
1	449	−413
2	658	658
3	217−	296
4	209−	−102
5	201−	191
6	560	−574
7	335	−335
8	155−	70
9	126−	−200
	0 18 l	
0	213−	−23
1	514	518
2	198−	−55
3	439	440
4	173−	−33
5	567	−583
6	140−	3
7	370	−378
8	84−	−10
	0 19 l	
1	267	255
2	492	523
3	251	−266
4	142	−97
5	182	−191
6	463	−481
	1 0 l	
0	1498	−1639
* $\underline{2}$	3551	−3247
* 2	2314	2431
$\underline{4}$	1988	2128
* 4	1869	1956
$\underline{6}$	804	761
6	1095	−970
$\underline{8}$	1504	−1584
8	1195	−1023
$\underline{10}$	671	−663
10	1567	1529
$\underline{12}$	807	849
12	609	649
$\underline{14}$	205−	162
14	708	−749
$\underline{16}$	−	−646
$\underline{16}$	178−	−206
18	464	543
	1 1 l	
0	959	−883
* $\underline{1}$	2193	−2233
1	206	−80
$\underline{2}$	1640	1854
2	1048	−942
$\underline{3}$	306	−162
* 3	1817	2623
$\underline{4}$	1134	1156
4	1377	1444
$\underline{5}$	1581	1609
5	444	240
$\underline{6}$	1105	−1140
6	980	905
$\underline{7}$	479	−525
7	1667	−1660
$\underline{8}$	517	−449
8	1080	−941
$\underline{9}$	1280	−1270
9	588	459
$\underline{10}$	682	661
10	395	−410
$\underline{11}$	238−	184
11	1292	1268
$\underline{12}$	237−	240
12	658	614
$\underline{13}$	586	589
13	236−	−57
$\underline{14}$	422	−420
14	230−	181
$\underline{15}$	−	−303
15	683	−656
$\underline{16}$	−	−115
$\underline{16}$	444	−420
$\underline{17}$	239	250
18	106−	−119
	1 2 l	
0	294	−274
$\underline{1}$	980	905
1	1815	−1796
$\underline{2}$	207	54
2	226	−52
* $\underline{3}$	2440	2715
3	235	162
$\underline{4}$	793	684
4	711	639
$\underline{5}$	650	−545
* 5	1944	2366
$\underline{6}$	176−	13
6	919	−789
$\underline{7}$	1263	−1298
7	168−	101
$\underline{8}$	679	−636
8	183−	−173
$\underline{9}$	420	418
9	1657	−1713
$\underline{10}$	300	−279
10	763	678
$\underline{11}$	870	980
11	430	422
$\underline{12}$	208−	281
12	282	279
$\underline{13}$	193−	−334
13	920	960
$\underline{14}$	168−	88
14	230−	−250
$\underline{15}$	−	−622
15	422	−371
$\underline{16}$	−	−200
$\underline{16}$	188−	−122
$\underline{17}$	594	−608
18	192	197
	1 3 l	
0	1247	−992
$\underline{1}$	541	475
1	232	−7
* $\underline{2}$	1760	1992
2	1796	−1762
$\underline{3}$	137−	149
3	732	−650
$\underline{4}$	1477	1567
* 4	2167	2481
$\underline{5}$	454	−431
5	152−	−63
$\underline{6}$	1108	−1251
6	1565	1588
$\underline{7}$	194−	101
7	394	386
$\underline{8}$	761	−676
8	1491	−1452
$\underline{9}$	393	381
9	207−	59
$\underline{10}$	1103	1170
10	716	−716
$\underline{11}$	229−	−123
11	522	−442
$\underline{12}$	293	311
12	828	916
$\underline{13}$	208−	−153
13	235−	34
$\underline{14}$	705	−752
14	460	455
$\underline{15}$	153−	81
15	209−	202
$\underline{16}$	−	−191
$\underline{16}$	741	−739
17	147−	−73
	1 4 l	
0	1400	1373
$\underline{1}$	128−	45
1	1028	−991
$\underline{2}$	916	838
2	1591	−1652
$\underline{3}$	1749	1856
3	370	359
$\underline{4}$	1190	−1173
4	804	−882
$\underline{5}$	171−	−124
5	1508	1653
$\underline{6}$	602	−560
6	1129	1153
$\underline{7}$	1013	−999
7	235	−102
$\underline{8}$	1203	1238
8	670	660
$\underline{9}$	394	362
9	986	−988
$\underline{10}$	618	575
10	1189	−1162
$\underline{11}$	614	657
11	231−	213
$\underline{12}$	580	−665
12	722	−640
$\underline{13}$	200−	−290
13	498	577
$\underline{14}$	175−	−127
14	575	570
$\underline{15}$	397	−404
15	211−	−196
$\underline{16}$	−	420
$\underline{16}$	183−	205
17	402	−409
	1 5 l	
0	529	−456
* $\underline{1}$	2018	2220
1	632	483
$\underline{2}$	440	395
2	141−	128
$\underline{3}$	151−	−227
* 3	2505	−2897
$\underline{4}$	394	371
4	169	133
$\underline{5}$	1483	−1521
5	168−	130
$\underline{6}$	233	−144
6	343	321
$\underline{7}$	475	524
7	1532	1604
$\underline{8}$	217−	−94
8	211−	−77
$\underline{9}$	1594	1523
9	743	−575
$\underline{10}$	235−	3
10	240−	−177
$\underline{11}$	333	−296
11	1464	−1390
$\underline{12}$	228−	72
12	257−	54
$\underline{13}$	645	−652
13	257−	184
$\underline{14}$	185−	−66
14	246−	49
$\underline{15}$	296	321
$\underline{15}$	740	705
$\underline{16}$	195−	−58
17	300	−282
	1 6 l	
0	1777	1749
$\underline{1}$	463	−448
1	1424	1353
$\underline{2}$	1439	1423
2	1550	−1482
$\underline{3}$	995	−960
3	374	−375
$\underline{4}$	1316	−1358
4	1483	−1569
$\underline{5}$	186−	11
5	1074	−1103
$\underline{6}$	558	−556
6	783	847
$\underline{7}$	734	701
7	194−	32
$\underline{8}$	1208	1187
8	700	699
$\underline{9}$	222−	−137
9	819	783
$\underline{10}$	516	522
10	1242	−1203
$\underline{11}$	510	−513
11	236−	−101
$\underline{12}$	541	−598
12	452	−490
$\underline{13}$	188−	195
13	510	−498
$\underline{14}$	162−	−150
14	639	627
$\underline{15}$	221	265
$\underline{15}$	198−	153
$\underline{16}$	167−	138
17	225	274
	1 7 l	
0	1210	1058
$\underline{1}$	1175	1121
1	213	−101
$\underline{2}$	1531	−1625
2	1182	1094
$\underline{3}$	175−	−137
3	982	−990
$\underline{4}$	1320	−1312
4	1638	−1652
$\underline{5}$	553	−599
5	181−	−33
$\underline{6}$	1073	1170
6	1068	−1069
$\underline{7}$	208−	227
7	528	564
$\underline{8}$	686	614
8	966	989
$\underline{9}$	537	557
9	221−	−136
$\underline{10}$	908	−946
10	727	679
$\underline{11}$	209−	−48
11	682	−624
$\underline{12}$	195−	−231
12	745	−779
$\underline{13}$	174−	−247
13	223−	60
$\underline{14}$	475	576
14	206−	−296
$\underline{15}$	104−	134
$\underline{15}$	237	250
$\underline{16}$	500	555
17	98−	−105
	1 8 l	
0	606	−537
$\underline{1}$	386	−351
1	1840	1758
$\underline{2}$	479	−438
2	536	484
$\underline{3}$	1914	−2050
3	609	−431
$\underline{4}$	354	343
4	288	266
$\underline{5}$	211−	121
5	1574	−1759
$\underline{6}$	221−	170
6	200−	−101
$\underline{7}$	1254	1213
7	209−	−105
$\underline{8}$	239−	−101
8	216−	−172
$\underline{9}$	375	−375
9	1197	1318
$\underline{10}$	241−	−110
10	224−	140
$\underline{11}$	830	−814
11	224−	−205
$\underline{12}$	215−	135
12	218−	30
$\underline{13}$	302	320
13	711	−826
$\underline{14}$	156−	31
$\underline{14}$	185−	−138
$\underline{15}$	293	288
16	125−	−18
	1 9 l	
0	553	531
$\underline{1}$	1107	−1145
1	194−	−202
$\underline{2}$	1178	−1170
2	831	803
$\underline{3}$	199−	203
3	1231	1207
$\underline{4}$	624	−627
4	1210	−1127
$\underline{5}$	972	967
5	203−	100
$\underline{6}$	644	625
6	755	−750
$\underline{7}$	367	−396
7	939	−958
$\underline{8}$	396	365
8	700	672
$\underline{9}$	792	−739
9	241	273
$\underline{10}$	687	−654
10	498	480
$\underline{11}$	217−	154
11	795	793
$\underline{12}$	197−	−181
12	606	−603
$\underline{13}$	374	387
13	200−	−161
$\underline{14}$	403	424
$\underline{14}$	177−	−207
$\underline{15}$	329	−404
16	421	425
	1 10 l	
0	1221	−1249
$\underline{1}$	346	−283
1	193−	219
$\underline{2}$	908	−884
2	1271	1254
$\underline{3}$	206−	−252
3	197−	−104
$\underline{4}$	1045	1056
4	991	1070
$\underline{5}$	213−	−30
5	407	−389
$\underline{6}$	480	503
6	1038	−1039
$\underline{7}$	341	280
7	219−	156
$\underline{8}$	1005	−1057
8	604	−647

Table 2 (cont.)

The following structure-factor listings are organised as eight column-groups, each with headers l, 10|Fo| and 10Fc. A trailing "–" on an |Fo| value denotes an unobserved (less-than) reflection.

Column group 1

l	10|Fo|	10Fc
	1 1 0 1	
9	208–	–129
9	228–	133
10	523	–506
10	1079	1059
11	178–	–144
11	224–	–92
12	595	506
12	632	573
13	129–	65
13	197–	–94
14	84–	143
14	522	–563
15	141–	50
	1 11 1	
0	401	–365
1	1378	–1409
1	207–	34
2	350	369
2	395	–352
3	223–	58
3	1450	1523
4	483	418
4	394	418
5	1012	1020
5	219–	–96
6	474	–457
6	442	396
7	320	–347
7	881	–941
8	233–	–130
8	430	–431
9	1062	–953
9	420	320
10	364	340
10	228–	–170
11	241	220
11	854	864
12	167–	95
12	205–	271
13	380	430
13	185–	–109
14	157–	129
15	365	–429
	1 12 1	
0	601	–568
1	211–	247
1	1090	–1134
2	523	–521
2	588	551
3	1184	1234
3	222–	175
4	235	338
4	666	625
5	220–	–149
5	1223	1222
6	275	296
6	290	–328
7	842	–831
7	232–	–165
8	484	–466
8	231–	–257
9	202–	221
9	768	–809
10	186–	–205
10	411	405
11	603	590
11	209–	173
12	203	227
12	247	255
13	505	535
14	213	–227
	1 13 1	
0	503	–558
1	240–	50
1	247–	47
2	1121	1121
2	797	–771
3	232–	34
3	243–	–137
4	739	694
4	957	927
5	228	–145
5	240–	40
6	752	–797
6	843	813
7	203–	14
7	234–	168
8	359	–360
8	807	–773
9	171–	43

Column group 2

l	10|Fo|	10Fc
9	218–	–54
10	527	612
10	360	–420
11	125–	–50
11	187–	–1
12	233	213
12	538	585
13	135–	15
	1 14 1	
0	546	568
1	129–	188
1	760	–756
2	544	512
2	586	–543
3	977	940
3	256–	91
4	463	–476
4	683	–562
5	222–	–68
5	964	919
6	393	–282
6	404	429
7	543	–567
7	230–	–21
8	348	400
8	370	334
9	175–	174
9	571	–603
10	152–	204
10	304	–394
11	336	387
11	163–	116
12	239	–228
13	292	389
	1 15 1	
0	219–	–159
0	938	981
1	238–	–34
2	297	271
2	238–	–64
3	215–	–61
3	911	–929
4	210–	172
4	299	235
5	841	–802
5	233–	–39
6	195–	–139
6	228–	181
7	280	275
7	732	754
8	167–	–42
8	209–	–139
9	608	616
9	201	–189
10	117–	140
10	177–	–99
11	580	–626
12	116–	130
	1 16 1	
0	593	603
1	221–	–46
1	301	330
2	570	530
2	638	–602
3	282	–289
3	224–	2
4	623	–598
4	604	–590
5	184–	88
5	227	–249
6	255	–288
6	580	548
7	151–	204
7	198–	17
8	572	588
8	275	319
9	102–	–50
9	197	219
10	634	–559
11	110–	–57
	1 17 1	
0	372	396
1	472	464
1	221–	11
2	520	–530
2	403	419
3	174–	–59
3	498	–459
4	412	–364
4	481	–483

Column group 3

l	10|Fo|	10Fc
5	314	–338
5	179–	–23
6	388	432
6	445	–414
7	222	157
7	309	333
8	155	183
8	352	421
9	190	–142
	1 18 1	
0	190–	21
1	187–	–124
1	656	650
2	182–	27
2	189–	–3
3	826	–704
4	186–	–88
4	162–	26
4	180–	–72
5	146–	71
6	718	–717
6	122–	7
7	159–	–7
7	142	73
	1 19 1	
0	251	273
1	360	–355
1	184–	–9
2	439	–442
2	317	288
3	108–	40
3	406	362
4	292	–311
4	419	–394
5	92–	–36
6	222	–327
	2 0 1	
0	531	–346
2	1740	1705
2	2592	–2793
4	170	–227
4	435	–518
6	1721	–1708
6	1931	1939
8	285	337
8	257	–147
10	819	931
10	1420	–1478
12	352	–345
12	377	291
14	492	–500
14	916	965
16	239	–163
18	628	–477
	2 1 1	
0	1194	1256
1	1119	1117
1	1107	–1228
2	240	–179
2	60–	257
3	459	496
3	1855	–1858
4	1463	–1435
4	1022	–1005
5	1301	–1300
5	1334	1113
6	167–	121
6	344	237
7	644	–588
7	869	850
8	733	770
8	911	942
9	819	889
9	1021	–1037
10	188–	–309
10	169–	–47
11	180–	224
11	712	–689
12	517	–437
12	354	–492
13	496	–511
13	869	859
14	111–	127
14	171–	186
15	158–	364
16	334	411
17	372	–444
	2 2 1	
0	364	260

Column group 4

l	10|Fo|	10Fc
1	1231	1212
1	1738	1811
2	332	365
2	1106	–1075
3	1646	–1766
3	1413	–1284
4	431	–333
4	394	260
5	1023	–938
5	1246	–1277
6	572	–550
6	579	570
7	1038	1057
7	1569	1549
8	194–	135
8	243	–198
9	201–	273
9	1097	1152
10	245	336
10	561	–567
11	753	–859
11	881	–899
12	177–	–105
12	173–	8
13	153–	–243
13	373	–400
14	121–	–150
14	356	431
15	670	767
16	140–	–51
17	115–	305
	2 3 1	
0	2087	2006
1	272	–271
1	314	275
2	646	–535
2	213	235
3	226	–128
3	661	614
4	1518	–1567
4	2108	–1937
5	177	309
5	377	–376
6	443	297
6	613	409
7	194	136
7	562	–508
8	844	952
8	1587	1711
9	177–	–223
9	468	489
10	546	–560
10	155–	96
11	159–	–73
11	269	227
12	670	–768
12	835	–1036
13	120–	159
13	260	–265
14	88–	242
14	330	342
15	138–	–139
16	558	744
17	97–	150
	2 4 1	
0	250	150
1	606	609
1	1093	1137
2	1164	–1244
2	1404	1511
3	763	–851
3	1201	–1043
4	630	688
4	108–	59
5	763	–648
5	607	–687
6	1106	1036
6	1333	–1279
7	602	595
7	1197	1104
8	197–	–263
8	150–	194
9	200–	219
9	728	792
10	710	–711
10	1152	1221
11	527	–605
11	532	–552
12	170–	233
12	186–	–192
13	144–	–171
13	469	–346

Column group 5

l	10|Fo|	10Fc
14	332	384
14	721	–748
15	487	528
16	148–	93
17	120–	202
	2 5 1	
0	121–	27
1	1376	–1417
1	1696	1640
2	137–	–79
2	111–	195
3	824	–713
3	1728	1723
4	374	–245
4	118–	–34
5	1468	1505
5	1276	–1168
6	180–	30
6	135–	–48
7	712	653
7	755	–895
8	193–	87
8	307	241
9	865	–946
9	1074	1139
10	191–	–47
10	177–	–9
11	185	–307
11	597	668
12	159–	–60
12	187–	–89
13	549	594
13	872	–873
14	92–	–3
14	178–	85
15	479	–426
16	143–	32
17	426	501
	2 6 1	
0	313	137
1	694	–646
1	605	–591
2	1483	–1533
2	2041	2047
3	898	929
3	556	516
4	334	388
4	368	413
5	247	229
5	566	526
6	1282	1289
6	1396	–1358
7	455	–461
7	757	–651
8	191–	–268
8	164–	152
9	189–	–153
9	597	–545
10	654	–701
10	904	1003
11	363	408
11	463	496
12	149–	260
12	363	–240
13	125–	130
13	184–	176
14	662	–701
15	324	–342
16	135–	129
	2 7 1	
0	1583	–1500
1	426	–410
1	573	580
2	157–	269
2	141–	–223
3	373	–325
3	567	582
4	1388	1429
4	1436	1471
5	669	697
5	142–	–226
6	180–	–161
6	147–	–172
7	182–	220
7	488	–447
8	808	–842
8	1497	–1526
9	337	–364
9	545	494
10	337	358
10	162–	65

Column group 6

l	10|Fo|	10Fc
11	151–	–111
11	161–	231
12	604	608
12	722	806
13	99–	223
13	397	–342
14	136–	–222
15	119–	–160
16	580	–589
	2 8 1	
0	148–	–92
1	685	736
1	1036	–1019
2	174–	–66
2	175–	89
2	151–	–237
3	1240	1322
3	807	815
4	170–	–43
4	149–	–146
5	788	712
5	1083	1115
6	196	–249
6	154–	132
7	698	–834
7	1200	–1242
8	181–	70
8	159–	85
9	177–	–238
9	943	–987
10	166–	122
10	263	–107
11	694	744
11	874	778
12	125–	–43
12	152–	84
13	218	213
13	397	332
14	126–	53
15	604	–637
16	84–	–41
	2 9 1	
0	1009	–1126
1	666	775
1	764	–748
2	159–	264
2	160–	–72
3	475	488
3	1060	–1052
4	913	960
4	922	941
5	922	–817
5	771	728
6	174–	–221
6	172–	–63
7	587	–402
7	597	584
8	359	–468
8	1157	–1092
9	536	563
9	646	–675
10	149–	283
10	188–	32
11	131–	142
11	411	–387
12	404	444
12	462	539
13	493	489
14	153–	–205
15	272	258
16	381	–419
	2 10 1	
0	166–	–3
1	169–	–178
1	172–	–226
2	1092	1136
2	1365	–1456
3	368	250
3	166–	237
4	515	–527
4	165–	–117
5	182–	175
5	164–	1
6	1013	–1036
6	1155	1183
7	179–	–107
7	309	–221
8	270	304
8	305	–186
9	162–	–70
9	162–	–81
10	605	647

Column group 7

l	10|Fo|	10Fc
10	981	–1053
11	124–	158
11	149–	16
12	90–	–233
12	139–	202
13	125–	53
14	616	685
15	85–	–140
	2 11 1	
0	515	487
1	685	736
1	1036	–1019
2	174–	–66
2	175–	89
3	425	459
3	894	–945
4	177–	–364
4	684	–670
5	775	–869
5	776	721
6	174–	39
6	182–	122
7	523	603
7	330	329
8	494	452
8	504	586
9	760	–747
10	128–	–154
10	179–	–7
11	187	211
11	400	–458
12	394	–337
13	550	563
14	123–	68
15	250	293
	2 12 1	
0	195–	12
1	646	588
1	828	803
2	472	436
2	853	–766
3	858	–867
3	719	–673
4	197–	–173
4	190–	–186
5	598	–454
5	457	–528
6	330	–432
6	530	530
7	594	602
7	633	695
8	165–	90
8	190–	–53
9	148–	199
9	617	545
10	349	275
10	342	–421
11	500	–488
12	155–	86
13	134–	–245
14	328	306
	2 13 1	
0	1129	1119
1	188–	–108
1	206–	42
2	188–	–270
2	206–	168
3	185–	–42
3	206–	64
4	1032	–993
4	1121	–1042
5	177–	3
5	206–	–185
6	170–	153
7	158–	49
8	542	595
8	1036	968
9	123–	–77
9	191–	3
10	250	–263
11	167–	50
12	445	–581
13	122–	–33
	2 14 1	
0	186–	–54

Column group 8

l	10|Fo|	10Fc
1	385	395
1	590	622
2	558	–555
2	727	703
3	555	–603
3	452	–437
4	170	152
4	179–	79
5	473	–378
5	452	–446
6	422	438
6	556	–560
7	422	435
7	561	559
8	104–	–132
8	164–	11
9	79–	106
9	402	426
10	470	470
11	394	–330
	2 15 1	
0	192–	217
1	587	–562
1	624	579
2	182–	–11
2	168–	–11
3	330	–355
3	714	740
4	167–	–213
4	165–	–192
5	583	615
5	537	–547
6	139–	23
6	160–	40
7	330	313
7	482	–469
8	95–	88
8	285	273
9	514	540
10	122–	9
11	333	345
	2 16 1	
0	173–	–38
1	169–	–197
1	159–	–198
2	575	–578
2	815	806
3	157–	224
3	157–	162
4	147–	219
4	156–	74
5	135–	117
5	152–	197
6	584	594
6	683	–628
7	89–	–178
7	139–	–152
8	129–	97
9	116–	–131
10	467	549
	2 17 1	
0	567	–557
1	259	–267
1	383	346
2	141–	99
2	147–	–56
3	133–	–156
3	294	357
4	489	490
4	577	566
5	318	322
5	138–	–245
6	131–	–58
7	121–	–192
	2 18 1	
0	127–	6
1	400	–339
1	479	–477
2	111–	24
2	137–	61
3	425	485
3	482	405
4	129–	22
4	251	360
5	110–	38
6	467	–454
	2 19 1	
2	85–	–56

Bertaut, Blum & Sagnières (1959). The final R values were:

$$R(0kl) = 0{\cdot}047, \quad R(1kl) = 0{\cdot}071, \quad R(2kl) = 0{\cdot}078 .$$

The overall R(hkl) for the 856 observed reflections was 0·068.

Fig. 1 represents the final $\varrho_0(Y, Z)$ projection with the Pb peak removed. A diagrammatic projection is

shown in Fig. 2. Table 1 lists the final atomic coordinates x, y, z and the coordinates X', Y', Z' referred to orthogonal axes:

$$X' = xa + zc \cos \beta, \quad Y' = yb, \quad Z' = zc \sin \beta .$$

A comparison of the final observed and calculated structure factors is given in Table 2.

Accuracy

For the $0kl$, $1kl$ and $2kl$ layers the corresponding two-dimensional standard deviations of the electron density (Cruickshank, 1949) are:

$$\sigma(\varrho_0) = 0.62, \quad \sigma(\varrho_1) = 1.07, \quad \sigma(\varrho_2) = 0.79 \text{ e.Å}^{-2}.$$

The three-dimensional value is: $\sigma(\varrho) = 0.95$ e.Å^{-3}.

The mean values of the standard deviations in the atomic coordinates, calculated by Cruickshank's method, are:

	$\sigma(x)$	$\sigma(y)$	$\sigma(z)$
Pb	0.0015 Å	0.0006 Å	0.0007 Å
S	0.0073	0.0020	0.0024
N	0.033	0.018	0.017
C	0.094	0.055	0.044
O	0.031	0.018	0.019

The $\sigma(x)$ values are much higher than the others because the $(\partial^2\varrho/\partial x^2)$'s are low on account of the elongated shape of the peaks in x direction, the reflections with $h = 3, 4, 5$ being neglected in the three-dimensional Fourier synthesis.

The standard deviations of the distances and angles were calculated from the formulae of Ahmed & Cruickshank (1953).

Discussion

The interatomic distances between Pb and its neighbours are as follows. (When the atomic coordinates are not given, the atom is at x, y, z)

Pb–S	$= 3.09 \pm 0.01$ Å
Pb–S($x - 1, y, z$)	$= 3.10 \pm 0.01$
Pb–S($1 - x, \bar{y}, \bar{z}$)	$= 3.34 \pm 0.01$
Pb–O$_I$	$= 2.62 \pm 0.02$
Pb–O$_{II}$	$= 2.44 \pm 0.02$
Pb–O$_{III}$	$= 2.63 \pm 0.03$
Pb–O$_{IV}(1 + x, y, z)$	$= 2.37 \pm 0.02$
Pb–O$_{III}(1 + x, y, z)$	$= 3.01 \pm 0.03$

Two of the Pb–S distances are practically equal and slightly longer than the corresponding distance found in Pbtu_2Cl$_2$ (mean value: 3.02 ± 0.05 Å; Nardelli & Fava, 1959b). The third Pb–S distance is much longer.

The two stoichiometric carboxylic groups are not equivalent, as one (O$_I$C$_{III}$O$_{II}$) has both its oxygen atoms bonded to the same Pb, while the oxygen atoms of the other (O$_{III}$C$_V$O$_{IV}$) are bonded to two different Pb's. Nevertheless, there are two different kinds of Pb–O distances practically equal for both COO⁻

groups: 2.37, 2.44 and 2.62, 2.63 Å. The 3.01 Å Pb–O distance probably involves a van der Waals contact. Ignoring this last contact, the coordination polyhedron around Pb can be described as a distorted pentagonal bipyramid. If all eight Pb-neighbours are considered, the coordination polyhedron is a dodecahedron with triangular faces similar to that found for [Mo(CN)$_8$]$^{4-}$ by Hoard & Nordsieck (1939). Fig. 3 is a stereographic projection showing the environment of Pb. The bond angles around Pb, as given in this figure, have the following mean standard deviations: 0.1° for S–Pb–S, 0.5° for S–Pb–X, 0.7° for X–Pb–X' ($X, X' =$ light atoms).
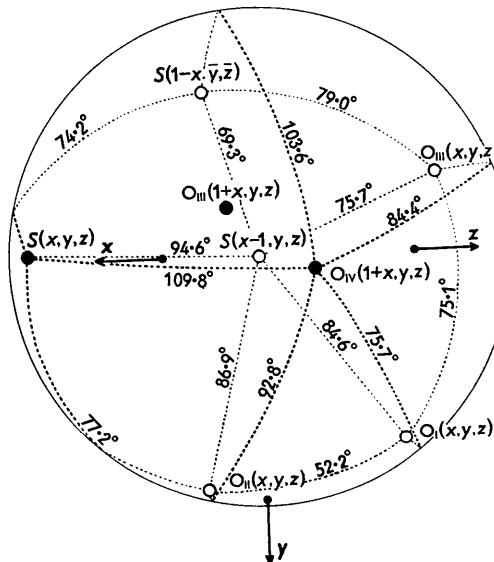

Fig. 3. Stereographic projection of the environment of Pb, viewed down the Pb–S($x - 1, y, z$) bond.

The same coordination number, 7, for Pb has already been found in *bis*-thiourea-lead(II) chloride, but in that case the shape of the polyhedron is different (trigonal prism with a seventh coordination position beyond one face). The pentagonal-bipyramid arrangement of seven bonding pairs of electrons can correspond to the configuration $(d_\varepsilon)^2 (d_\gamma)^4 (s)^2 (p)^6$ (Gillespie & Nyholm, 1957), but the small difference in energy between $6d$ and $5f$ orbitals in Pb makes a contribution from these last orbitals probable. This contribution, involving different ligands and bonds, can explain the distortion.

The coordination polyhedra are linked together as shown in Fig. 4. The Pb and S atoms, with coordinates $n + x, y, z$ (n integral), form a zig-zag chain running in the x direction, each S atom being equidistant from two adjacent Pb atoms (~ 3.10 Å). Two of these chains, related by symmetry centres, are joined by weaker Pb–S $= 3.34$ Å bonds. Because of this, each S atom is bonded coordinatively to three Pb atoms. It should be pointed out that, among thiourea metal complexes of known structure, Pbtu(C$_2$H$_3$O$_2$)$_2$ is the
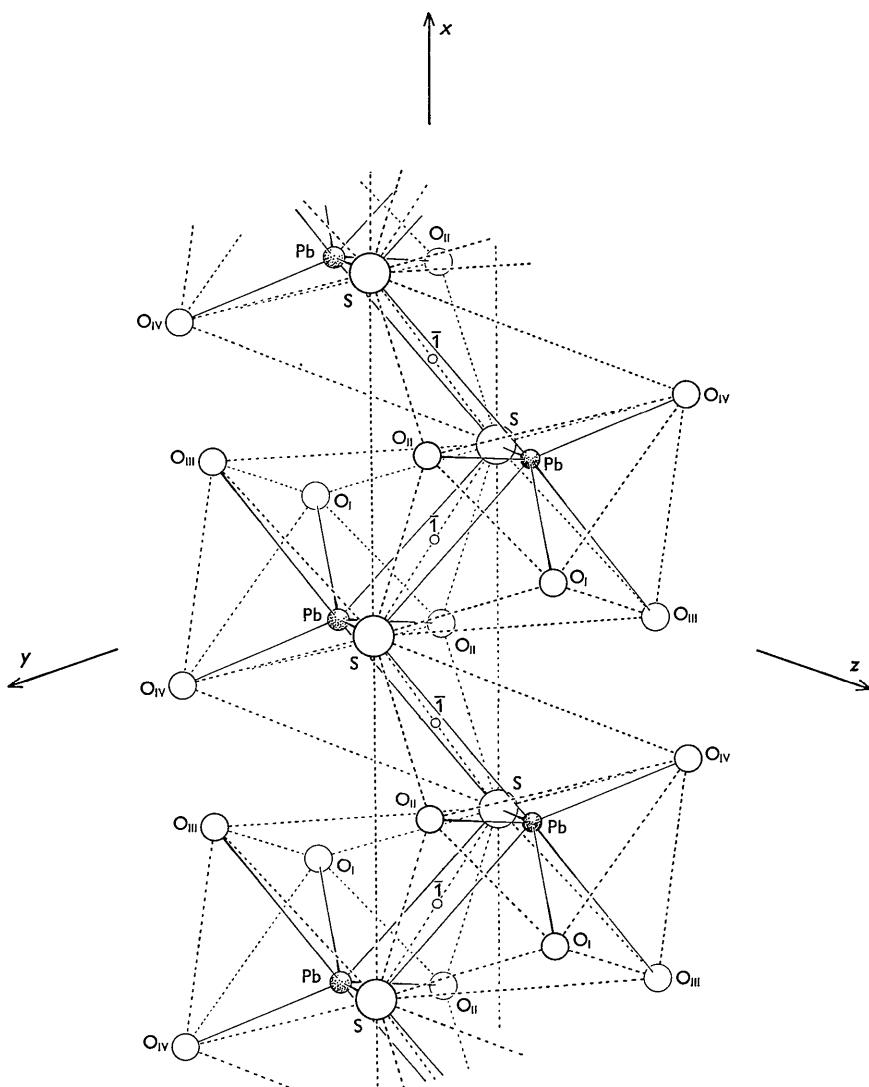
Fig. 4. Clinographic projection of a double chain of coordination polyhedra.

only compound in which sulphur forms three coordinative bonds. Sulphur is mono-coordinated in $Nitu_4Cl_2$ (Cavalca, Nardelli & Braibanti, 1956), $Cdtu_2Cl_2$ (Nardelli, Cavalca & Braibanti, 1957) and $Zntu_2Cl_2$ (Kunchur & Truter, 1958), bi-coordinated in $Nitu_2(NCS)_2$ (Nardelli, Braibanti & Fava, 1957) and $Pbtu_2Cl_2$ (Nardelli & Fava, 1959b), and mono- and bi-coordinated in $Cutu_3Cl$ (Knobler, Okaya & Pepinsky, 1959).

The different natures of the four atoms bonded to each S are probably the cause of the deviations from the tetrahedral bond angles:

Owing to the presence of the very heavy Pb atom, the coordinates of the light atoms are affected by rather high standard deviations. Nevertheless, the bond distances and angles in the organic molecules are consistent with those expected:

in the thiourea molecule

$$S–C_I = 1\cdot68 \pm 0\cdot06 \text{ Å}$$
$$C_I–N_I = 1\cdot38 \pm 0\cdot07$$
$$C_I–N_{II} = 1\cdot35 \pm 0\cdot07$$

$Pb–S–C_I$	$= 123\cdot8 \pm 3\cdot1°$
$C_I–S–Pb(1-x, \bar{y}, \bar{z})$	$= 119\cdot3 \pm 2\cdot1$
$Pb–S–Pb(1+x, y, z)$	$= 94\cdot6 \pm 0\cdot1$
$C_I–S–Pb(1+x, y, z)$	$= 98\cdot6 \pm 2\cdot6$
$Pb–S–Pb(1-x, \bar{y}, \bar{z})$	$= 105\cdot8 \pm 0\cdot1$
$Pb(1-x, \bar{y}, \bar{z})–S–Pb(1+x, y, z)$	$= 110\cdot7 \pm 0\cdot1$

$$S–C_I–N_I = 117\cdot6 \pm 3\cdot3°$$
$$S–C_I–N_{II} = 121\cdot6 \pm 2\cdot8$$
$$N_I–C_I–N_{II} = 120\cdot7 \pm 3\cdot1$$

and in the acetic groups

$$C_{II}-C_{III} = 1\cdot50 \pm 0\cdot08 \text{ Å}$$
$$O_I-C_{III} = 1\cdot23 \pm 0\cdot07$$
$$O_{II}-C_{III} = 1\cdot27 \pm 0\cdot08$$
$$C_{IV}-C_V = 1\cdot48 \pm 0\cdot08$$
$$O_{III}-C_V = 1\cdot24 \pm 0\cdot06$$
$$O_{IV}-C_V = 1\cdot34 \pm 0\cdot09$$

$$C_{II}-C_{III}-O_I = 118\cdot1 \pm 4\cdot5°$$
$$C_{II}-C_{III}-O_{II} = 116\cdot1 \pm 4\cdot5$$
$$O_I-C_{III}-O_{II} = 125\cdot6 \pm 3\cdot5$$
$$C_{IV}-C_V-O_{III} = 120\cdot2 \pm 4\cdot9$$
$$C_{IV}-C_V-O_{IV} = 118\cdot3 \pm 4\cdot6$$
$$O_{III}-C_V-O_{IV} = 121\cdot5 \pm 3\cdot6$$

The planarity of the organic molecules is shown by the sum of the bond angles around the central C atoms. The equations of the least-squares planes for the (x, y, z) molecules are:

$$0\cdot2752X' + 0\cdot0788Y' + 0\cdot2462Z' = 1$$
$$\text{for } SC_IN_IN_{II}$$

$$0\cdot3289X' + 0\cdot0248Y' + 0\cdot3399Z' = 1$$
$$\text{for } C_{II}C_{III}O_IO_{II}$$

$$-0\cdot9530X' - 1\cdot3212Y' + 0\cdot2906Z' = 1$$
$$\text{for } C_{IV}C_VO_{III}O_{IV}$$

The distances of these atoms from the corresponding molecular planes are less than the standard deviations of the coordinates; the mean distance is 0·01 Å.

There are three interatomic N–O distances less than 3 Å:

$$N_{II}-O_I(x+1, \tfrac{1}{2}-y, z-\tfrac{1}{2}) = 2\cdot96 \pm 0\cdot03 \text{ Å}$$
$$N_I-O_{II} = 2\cdot86 \pm 0\cdot04$$
$$N_I-O_{IV}(x+1, \tfrac{1}{2}-y, z-\tfrac{1}{2}) = 2\cdot95 \pm 0\cdot03$$

and these are presumably hydrogen bonds of the kind NH $\cdots$ O. The oxygen atoms are not far from the plane of the thiourea molecule; their distances are: 0·32 (O_I), 0·05 (O_{II}) and 0·25 Å (O_{IV}). While the following two angles are not unfavorable for hydrogen bonding

$$C_I-N_I \cdots O_{II} = 113\cdot5°$$
$$C_I-N_I \cdots O_{IV}(x+1, \tfrac{1}{2}-y, z-\tfrac{1}{2}) = 111\cdot4$$

the angle

$$C_I-N_{II} \cdots O_I (x+1, \tfrac{1}{2}-y, z-\tfrac{1}{2}) = 141\cdot8°$$

is rather high for an sp^2 trigonal configuration of the

bonds on the nitrogen atoms. The other O–N distances are larger than those usually considered in hydrogen bonding:

$$O_{III}-N_{II}(1-x, \bar{y}, \bar{z}) = 3\cdot32 \pm 0\cdot02 \text{ Å}$$
$$O_{IV}-N_{II}(x-1, \tfrac{1}{2}-y, \tfrac{1}{2}+z) = 3\cdot53 \pm 0\cdot02$$
$$O_I-N_I (x, \tfrac{1}{2}-y, \tfrac{1}{2}+z) = 3\cdot44 \pm 0\cdot03$$
$$O_{II}-N_I (x-1, y, z) = 3\cdot48 \pm 0\cdot04$$

There are no other interatomic distances smaller than the van der Waals contacts.

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