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The Crystal Structure of 2′-Chlorobiphenyl-4-carboxylic Acid

By H. H. Sutherland

Physics Department, The University, Hull, England

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The crystal structure of 2′-chlorobiphenyl-4-carboxylic acid ($C_{13}H_8O_2Cl$) has been determined from three-dimensional X-ray diffraction data. The crystals are monoclinic, space group $P2_1/c$, with unit-cell dimensions $a=3{\cdot}94$, $b=35{\cdot}81$, $c=7{\cdot}61$ Å, $\beta=101°6'$. The structure, which consists of centrosymmetrical hydrogen-bonded dimers, was refined by block-diagonal least-squares refinement to a residual of 8% for the 1028 observed structure factors. The angle between the two phenyl rings and the angle between the carboxyl group and the phenyl ring to which it is attached were found to be 48·9° and 5·6° respectively.

Introduction

In recent investigations (Branch, Byron, Gray, Ibbotson & Worral, 1964) on the properties of materials exhibiting liquid crystalline properties it became obvious that accurate information regarding the structure in the solid phase of related materials was required. This paper describes the structure of the fourth of a series of related compounds being investigated by the author.

Experimental

2′-Chlorobiphenyl-4-carboxylic acid was prepared by oxidizing 4-acetyl-2′-chlorobiphenyl with sodium hypobromite in 50% aqueous dioxan (Byron, Gray & Wilson, 1966). Slow evaporation from ethanol produced transparent, needle shaped crystals. The observed density at 17°C was measured by the method of flotation using aqueous cadmium n-dodecatungstaborate.

Crystal data

$C_{13}H_8O_2Cl$, $M=232{\cdot}7$, monoclinic, $a=3{\cdot}94\pm0{\cdot}01$, $b=35{\cdot}81\pm0{\cdot}02$, $c=7{\cdot}61\pm0{\cdot}01$ Å, $\beta=101°6'\pm5'$, $U=1053{\cdot}4$ Å^3, $D_m=1{\cdot}47$ g.cm^{-3}, $D_c=1{\cdot}46$ g.cm^{-3}, $Z=4$, $F(000)=476$, Cu $K\alpha$ ($\lambda=1{\cdot}5418$ Å), Mo $K\alpha$ ($\lambda=0{\cdot}7107$ Å). Absent spectra, $0k0$ when k odd and $h0l$ when l odd. Space group $P2_1/c$ (No. 14).

In order to minimize absorption effects a needle crystal of approximately square cross section was chosen for intensity data collection about the a axis. Data for the $0kl$, $1kl$, $2kl$, $3kl$ and $hk0$ zones were collected by the multiple film equi-inclination Weissenberg technique, using Cu $K\alpha$ radiation. The $hk0$ zone intensity data were collected using a cube shape crystal cut from one of the needles.

Initially the intensities of the spots were estimated visually – only those on the extended side of the films on the upper levels being measured – corrected for Lorentz and polarization factors and the Phillips (1954) correction factor applied. The data were then brought to the same arbitrary scale and finally on to an approximate absolute scale by the method of Wilson (1942) applied to the $0kl$ zone. No correction for absorption was applied to the data. Subsequently all the intensities were remeasured on a Joyce Loebl Flying Spot Densitometer as a check on the instrument. These data did not require the Phillips (1954) correction factor.

Determination and refinement of the structure

A trial structure was deduced from the $0kl$ and $hk0$ sharpened Patterson projections. A structure-factor calculation involving the full three-dimensional data gave a residual $R=0{\cdot}40$ where $R=\Sigma\,||F_o|-|F_c||/\Sigma\,|F_o|$. The structure was refined by two cycles of three-dimensional electron density distribution and structure-factor calculations to a residual $R=0{\cdot}23$.

Two cycles of block-diagonal least squares applied to positional parameters and overall scale factor on an Elliot 803B computer using the block-diagonal least-squares program of Daly, Stephens & Wheatley (1963) reduced the residual to $R=0{\cdot}18$. The inclusion of individual isotropic thermal parameters in the refinement resulted in a residual of $R=0{\cdot}15$.

At this stage the three-dimensional data collected by means of the Joyce Loebl Flying Spot Densitometer became available and it was decided to use this in the subsequent refinement. As a starting point the coordinates corresponding to the previous residual $R=0{\cdot}18$ were chosen. Three cycles of least-squares refinement on scale, coordinate and overall thermal parameters gave a residual of $R=0{\cdot}13$. On including individual isotropic thermal parameters in the refinement a residual of $R=0{\cdot}11$ was obtained. After three cycles incorporating anisotropic thermal parameters defined as

$$\exp\,[-2\pi^2(U_{11}h^2a^{*2}+U_{22}k^2b^{*2}+U_{33}l^2c^{*2}+2U_{23}klb^*c^*+2U_{31}lhc^*a^*+2U_{12}hka^*b^*)]\,;$$

(Cruickshank, Pilling, Bujosa, Lovell & Truter, 1961), a three-dimensional difference electron density distribution was calculated; this gave a clear indication of the approximate positions of all the hydrogen atoms.

Table 1. *The observed and calculated structure factors*

Unobserved reflexions are denoted by an asterisk

H	K	L	100FO	100FC
0	2	0	•	684
0	4	0	1886	-2133
0	6	0	3811	4343
0	8	0	5239	-6009
0	10	0	4904	5181
0	12	0	6946	-7635
0	14	0	2677	2822
0	16	0	1605	1619
0	18	0	1987	-2167
0	20	0	5259	5154
0	22	0	1004	-915
0	24	0	3066	3177
0	26	0	1571	1436
0	28	0	1079	-1192
0	30	0	1823	1709
0	32	0	•	-62
0	34	0	2554	2411
0	36	0	•	338
0	38	0	1646	-1694
0	40	0	•	241
0	42	0	•	-701
0	44	0	•	62
0	1	1	2398	2343
0	2	1	2137	-1958
0	3	1	4131	-4164
0	4	1	7718	8039
0	5	1	3025	-3001
0	6	1	1932	2179
0	7	1	5839	-5887
0	8	1	1468	1355
0	9	1	2452	2847
0	10	1	628	685
0	11	1	2753	3327
0	12	1	1899	2137
0	13	1	2787	-3045
0	14	1	273	513
0	15	1	6017	6017
0	16	1	2076	-2242
0	17	1	1223	1227
0	18	1	•	60
0	19	1	•	131
0	20	1	1284	1196
0	21	1	•	36
0	22	1	560	628
0	23	1	3422	-3476
0	24	1	•	-22
0	25	1	1256	1219
0	26	1	1079	1122
0	27	1	2049	-1908
0	28	1	1188	-1129
0	29	1	2069	-1962
0	30	1	1332	-968
0	31	1	•	29
0	32	1	2042	1936
0	33	1	778	567
0	34	1	1332	1054
0	35	1	2376	2176
0	36	1	1448	1244
0	37	1	1100	-936
0	38	1	•	408
0	39	1	•	529
0	40	1	•	-18
0	41	1	•	570
0	42	1	1154	548
0	43	1	465	-822
0	44	1	•	-96
0	45	1	•	414
0	0	2	4480	3983
0	1	2	1277	1234
0	2	2	5300	-5356
0	3	2	1660	-1613
0	4	2	320	151
0	5	2	1318	-1375
0	6	2	677	589
0	7	2	2186	-2104
0	8	2	6824	-6552
0	9	2	•	142
0	10	2	465	454
0	11	2	600	562
0	12	2	7943	-8356
0	13	2	4432	-4685
0	14	2	2104	-2106
0	15	2	•	158
0	16	2	1318	1167
0	17	2	1161	1361
0	18	2	1147	-1340
0	19	2	936	767
0	20	2	1714	1780
0	21	2	1830	1786
0	22	2	3716	-3941
0	23	2	793	532
0	24	2	2254	2372
0	25	2	1127	1165
0	26	2	839	798
0	27	2	1482	-1432
0	28	2	3477	-3281
0	29	2	1571	-1597
0	30	2	1728	1819
0	31	2	•	353
0	32	2	2970	-2778
0	33	2	•	39
0	34	2	•	-82
0	35	2	•	-62
0	36	2	683	724
0	37	2	•	-266
0	38	2	1413	-1175
0	39	2	•	-573
0	40	2	1216	1145
0	41	2	•	-156
0	42	2	1038	-971
0	43	2	•	183
0	44	2	•	416
0	45	2	•	-11
0	1	3	1004	-1010
0	2	3	833	-573
0	3	3	•	143
0	4	3	3681	-3473
0	5	3	4740	4712
0	6	3	1557	1608
0	7	3	1517	-1341
0	8	3	1974	1656
0	9	3	4426	4218
0	10	3	600	-336
0	11	3	969	-1151
0	12	3	1284	-1168
0	13	3	1899	-1927
0	14	3	1421	-1512
0	15	3	3668	3804
0	16	3	643	-882
0	17	3	1735	-1883
0	18	3	621	-585
0	19	3	1421	1344
0	20	3	•	-234
0	21	3	•	-9
0	22	3	•	-110
0	23	3	1352	-1347
0	24	3	1167	-1230
0	25	3	1195	1018
0	26	3	•	-511
0	27	3	1434	-1356
0	28	3	•	400
0	29	3	1448	1341
0	30	3	•	-293
0	31	3	•	195
0	32	3	•	-12
0	33	3	799	-876
0	34	3	•	-18
0	35	3	1448	1398
0	36	3	•	-457
0	37	3	•	-93
0	38	3	854	-774
0	39	3	499	471
0	40	3	•	-403
0	41	3	•	239
0	42	3	•	485
0	43	3	•	-658
0	44	3	•	-301
0	0	4	5798	5669
0	1	4	977	662
0	2	4	922	-817
0	3	4	1468	-1358
0	4	4	4255	4278
0	5	4	2834	-2253
0	6	4	2992	2986
0	7	4	1906	1821
0	8	4	4357	-4014
0	9	4	•	325
0	10	4	2533	2410
0	11	4	1223	1072
0	12	4	1249	-1238
0	13	4	458	649
0	14	4	•	397
0	15	4	•	116
0	16	4	•	59
0	17	4	2179	2214
0	18	4	•	-40
0	19	4	1455	-1415
0	20	4	4262	4546
0	21	4	•	-438
0	22	4	854	-918
0	23	4	•	220
0	24	4	888	866
0	25	4	1058	-887
0	26	4	•	442
0	27	4	827	939
0	28	4	1167	-935
0	29	4	•	223
0	30	4	2321	2212
0	31	4	•	421
0	32	4	888	-829
0	33	4	•	56
0	34	4	•	366
0	35	4	•	52
0	36	4	•	85
0	37	4	•	88
0	38	4	915	-820
0	39	4	•	-27
0	40	4	•	1190
0	41	4	•	924
0	42	4	•	-467
0	1	5	499	-409
0	2	5	1236	1263
0	3	5	2609	-2636
0	4	5	•	-528
0	5	5	839	671
0	6	5	1216	1071
0	7	5	2213	-2008
0	8	5	949	-644
0	9	5	1427	-1357
0	10	5	•	-533
0	11	5	547	605
0	12	5	1185	1219
0	13	5	•	-26
0	14	5	•	621
0	15	5	2875	2779
0	16	5	847	969
0	17	5	977	-1161
0	18	5	•	-700
0	19	5	•	267
0	20	5	1032	-1135
0	21	5	2391	2374
0	22	5	•	89
0	23	5	2383	-2456
0	24	5	•	567
0	25	5	1782	-1919
0	26	5	1271	1192
0	27	5	1325	-1430
0	28	5	•	383
0	29	5	•	268
0	30	5	•	162
0	31	5	•	409
0	32	5	922	580
0	33	5	•	-491
0	34	5	•	-218
0	35	5	793	752
0	36	5	•	-108
0	37	5	•	-369
0	38	5	•	196
0	39	5	•	612
0	0	6	2903	3153
0	1	6	•	-24
0	2	6	1632	-1419
0	3	6	1011	-675
0	4	6	2076	1937
0	5	6	1551	1405
0	6	6	•	413
0	7	6	1995	-1402
0	8	6	4835	-4976
0	9	6	3907	-3577
0	10	6	•	-101
0	11	6	•	365
0	12	6	1912	-1965
0	13	6	•	184
0	14	6	•	47
0	15	6	839	694
0	16	6	•	-394
0	17	6	•	74
0	18	6	1011	-1039
0	19	6	•	-397
0	20	6	1618	1704
0	21	6	861	748
0	22	6	1271	-1393
0	23	6	•	-450
0	24	6	•	70
0	25	6	•	600
0	26	6	•	-363
0	27	6	•	151
0	28	6	•	-521
0	29	6	1154	-1252
0	30	6	1141	1263
0	31	6	•	-237
0	32	6	•	-677
0	33	6	•	-428
0	34	6	•	114
0	35	6	•	-348
0	36	6	•	150
0	1	7	•	-176
0	2	7	•	1030
0	3	7	•	1042
0	4	7	1393	1178
0	5	7	1762	1510
0	6	7	•	752
0	7	7	1728	-1787
0	8	7	894	1092
0	9	7	•	486
0	10	7	•	-85
0	11	7	•	759
0	12	7	•	-596
0	13	7	•	149
0	14	7	•	-477
0	15	7	1537	1657
0	16	7	•	111
0	17	7	•	-226
0	18	7	•	-504
0	19	7	•	276
0	20	7	•	-754
0	21	7	•	14
0	22	7	•	206
0	0	8	1475	1423
0	1	8	•	354
0	2	8	•	-243
0	3	8	1058	-1034
0	4	8	•	-163
0	5	8	•	-307
0	6	8	•	246
0	7	8	•	560
0	8	8	•	-293
0	9	8	•	-536
0	10	8	•	702
0	14	8	•	224
0	12	8	•	0
0	13	8	•	-804
0	14	8	778	980
0	15	8	•	299
0	16	8	•	9
0	17	8	•	844
0	18	8	772	-956
0	19	8	•	48
0	20	8	•	1103
0	21	8	1078	1456
0	22	8	•	203
1	0	0	5672	5988
1	1	0	372	-167
1	2	0	1713	-1721
1	3	0	3035	2900
1	4	0	5440	-5885
1	5	0	8542	10669
1	6	0	6238	-7478
1	7	0	6468	7552
1	8	0	2850	-3189
1	9	0	2821	-3141
1	10	0	2605	2768
1	11	0	1280	1568
1	12	0	2258	-2166
1	13	0	658	-655
1	14	0	1145	-1395
1	15	0	2241	-2146
1	16	0	801	591
1	17	0	•	-171
1	18	0	1734	-1513
1	19	0	2871	-3060
1	20	0	445	-433
1	21	0	1482	1444
1	22	0	818	-803
1	23	0	1351	-1141
1	24	0	854	-854
1	25	0	•	-105
1	26	0	•	-183
1	27	0	1556	1437
1	28	0	1427	-1215
1	29	0	2403	-2206
1	30	0	1794	-1800
1	31	0	1867	1810
1	32	0	864	838
1	33	0	•	16
1	34	0	1552	1359
1	35	0	•	111
1	36	0	492	-574
1	37	0	1626	1401
1	38	0	•	-510
1	39	0	•	-193
1	40	0	•	94
1	41	0	1607	1336
1	42	0	•	64
1	43	0	1278	-938
1	44	0	•	-98
1	45	0	•	-563
1	1	1	2254	2250
1	2	1	3002	2714
1	3	1	4317	-4508
1	4	1	3195	-3309
1	5	1	2966	-2859
1	6	1	3088	2882
1	7	1	1178	-1017
1	8	1	5956	-6079
1	9	1	1165	883
1	10	1	2295	-2258
1	11	1	2098	2221
1	12	1	709	-748
1	13	1	1801	-1762
1	14	1	4158	-4428
1	15	1	386	502
1	16	1	3790	4378
1	17	1	917	1023
1	18	1	2142	-1947
1	19	1	486	-408
1	20	1	1433	1355
1	21	1	1268	-1108
1	22	1	2960	2986
1	23	1	•	-131
1	24	1	751	-550
1	25	1	2215	2173
1	26	1	1929	1939
1	27	1	1927	-2053
1	28	1	4579	-4679
1	29	1	1947	-1690
1	30	1	1477	-1409
1	31	1	•	-148
1	32	1	745	772
1	33	1	957	-851
1	34	1	2496	-2354
1	35	1	737	759
1	36	1	1053	1061
1	37	1	•	-517
1	38	1	591	-788
1	39	1	•	124
1	40	1	•	11
1	41	1	•	285
1	42	1	•	421
1	43	1	•	-3
1	44	1	•	-852
1	45	1	•	238
1	1	-1	11644	12707
1	2	-1	4503	4518
1	3	-1	6214	-6767
1	4	-1	8154	-9107
1	5	-1	3947	4386
1	6	-1	7400	8148
1	7	-1	•	-48
1	8	-1	3861	-4214
1	9	-1	569	-629
1	10	-1	2632	-2629
1	11	-1	2006	-2092
1	12	-1	599	728
1	13	-1	1890	-1903
1	14	-1	2649	-2611
1	15	-1	3112	3305
1	16	-1	5781	5980
1	17	-1	505	-503
1	18	-1	•	-538
1	19	-1	1729	1477
1	20	-1	1738	1741
1	21	-1	425	495
1	22	-1	2632	2500
1	23	-1	882	-467
1	24	-1	1228	-1149
1	25	-1	1466	1464
1	26	-1	2201	2023
1	27	-1	•	-75
1	28	-1	•	-718
1	29	-1	844	692
1	30	-1	670	649
1	31	-1	•	283
1	32	-1	•	-169
1	33	-1	345	656
1	34	-1	2022	-1963
1	35	-1	1516	1317
1	36	-1	1173	832
1	37	-1	928	-513
1	38	-1	•	365
1	39	-1	•	-474
1	40	-1	1403	1208
1	41	-1	•	-234
1	42	-1	840	575
1	43	-1	•	-88
1	44	-1	740	-646
1	45	-1	•	-106
1	0	2	1067	975
1	1	2	3586	3528
1	2	2	3356	3480
1	3	2	2625	-2377
1	4	2	2860	2656
1	5	2	2913	2920
1	6	2	1497	1165
1	7	2	4575	4311
1	8	2	4518	4507
1	9	2	1341	-1323
1	10	2	1466	1230
1	11	2	2048	2163
1	12	2	3002	-2986
1	13	2	3422	-3358
1	14	2	1397	1164
1	15	2	•	-348
1	16	2	990	915
1	17	2	930	-959
1	18	2	•	-214
1	19	2	2591	-2574
1	20	2	•	-174
1	21	2	2617	2710
1	22	2	•	238
1	23	2	•	-352
1	24	2	2579	2471
1	25	2	651	598
1	26	2	1198	1159
1	27	2	598	474
1	28	2	678	657
1	29	2	1348	-1279
1	30	2	•	428
1	31	2	1115	1123
1	32	2	•	-476
1	33	2	•	-49
1	34	2	786	592
1	35	2	•	351
1	36	2	•	362
1	37	2	•	42
1	38	2	•	115
1	39	2	1395	-1259
1	40	2	•	115
1	41	2	928	734
1	42	2	•	-255
1	43	2	•	19
1	44	2	•	-159
1	0	-2	5284	-5583
1	1	-2	5893	6042
1	2	-2	2417	2348
1	3	-2	•	-339
1	4	-2	3112	3211
1	5	-2	467	-548
1	6	-2	940	824
1	7	-2	3397	3500
1	8	-2	1295	1368
1	9	-2	2724	-2700
1	10	-2	1983	2137
1	11	-2	2020	2044
1	12	-2	•	327
1	13	-2	2212	-2340
1	14	-2	1029	383
1	15	-2	•	-108
1	16	-2	4578	4507
1	17	-2	1129	1239
1	18	-2	1840	1876
1	19	-2	4403	-4582
1	20	-2	852	1078
1	21	-2	934	896
1	22	-2	1357	-1363
1	23	-2	•	262
1	24	-2	748	-890
1	25	-2	•	-388
1	26	-2	1228	1322
1	27	-2	1145	1250
1	28	-2	940	-718
1	29	-2	•	405
1	30	-2	•	262
1	31	-2	1015	679
1	32	-2	•	-378
1	33	-2	1934	-1810
1	34	-2	•	493
1	35	-2	•	-612
1	36	-2	751	635
1	37	-2	•	229
1	38	-2	•	-341
1	39	-2	572	-568
1	40	-2	973	798
1	41	-2	•	453
1	42	2	791	-410
1	43	-2	691	-680
1	44	-2	•	32
1	1	3	1248	-921
1	2	3	2178	1858
1	3	3	1447	-911
1	4	3	6114	-5843
1	5	3	2447	2218
1	6	3	3187	3104
1	7	3	870	-492
1	8	3	1403	1132
1	9	3	•	407
1	10	3	1079	1015
1	11	3	837	-396
1	12	3	2020	1941
1	13	3	•	283
1	14	3	3142	-3030
1	15	3	•	154
1	16	3	3247	3300
1	17	3	•	4

Table 1 (*cont.*)

H	K	L	100FO	100FC
1	18	3	•	-96
1	19	3	1943	1888
1	20	3	•	196
1	21	3	1255	-1300
1	22	3	2403	2206
1	23	3	•	-212
1	24	3	1976	-1881
1	25	3	1116	972
1	26	3	1209	1257
1	27	3	693	-723
1	28	3	•	-673
1	29	3	•	222
1	30	3	•	146
1	31	3	•	-186
1	32	3	877	960
1	33	3	•	15
1	34	3	871	-742
1	35	3	•	373
1	36	3	1166	1202
1	37	3	•	-78
1	38	3	824	-730
1	39	3	762	391
1	40	3	969	812
1	41	3	648	473
1	42	3	•	1393
1	1	-3	2871	-2879
1	2	-3	2536	2478
1	3	-3	2264	-2400
1	4	-3	2668	-2623
1	5	-3	2854	2944
1	6	-3	2294	2236
1	7	-3	3016	-2901
1	8	-3	2712	-2886
1	9	-3	392	-401
1	10	-3	2178	-2132
1	11	-3	1662	-1699
1	12	-3	3019	3100
1	13	-3	3291	-3201
1	14	-3	1324	-1209
1	15	-3	•	407
1	16	-3	841	843
1	17	-3	1165	-1292
1	18	-3	2066	-2047
1	19	-3	1831	1732
1	20	-3	•	141
1	21	-3	953	-986
1	22	-3	1152	1257
1	23	-3	960	-1009
1	24	-3	2649	-2611
1	25	-3	1453	1541
1	26	-3	1729	1711
1	27	-3	2267	-2078
1	28	-3	1194	-1170
1	29	-3	•	880
1	30	-3	1383	-1210
1	31	-3	•	-16
1	32	-3	•	36
1	33	-3	2264	-2212
1	34	-3	1590	-1556
1	35	-3	•	462
1	36	-3	1480	1407
1	37	-3	•	-236
1	38	-3	•	-626
1	39	-3	•	107
1	40	-3	•	73
1	41	-3	•	196
1	42	-3	•	588
1	43	-3	•	-60
1	0	4	1043	-927
1	1	4	2599	2426
1	2	4	756	-471
1	3	4	1082	-983
1	4	4	1712	-1579
1	5	4	670	-587
1	6	4	1215	-889
1	7	4	2797	2822
1	8	4	1139	-899
1	9	4	844	-612
1	10	4	2168	-2415
1	11	4	1818	1549
1	12	4	837	-869
1	13	4	2003	-1816
1	14	4	•	147
1	15	4	1735	-1647
1	16	4	•	-25
1	17	4	967	1142
1	18	4	850	-937
1	19	4	1904	-1885
1	20	4	957	758
1	21	4	•	510
1	22	4	•	608
1	23	4	•	-397
1	24	4	1993	-2113
1	25	4	•	-196
1	26	4	969	-1245
1	27	4	1181	1173
1	28	4	•	17
1	29	4	624	-704
1	30	4	•	-116
1	31	4	916	818
1	32	4	•	-103
1	33	4	•	-488
1	34	4	•	-16
1	35	4	765	-409
1	36	4	878	-632
1	37	4	•	-56
1	38	4	•	-203
1	39	4	•	-628
1	40	4	•	166
1	0	-4	1881	-1747
1	1	-4	2957	2847
1	2	-4	2397	-2292
1	3	-4	1913	1520
1	4	-4	1291	1079
1	5	-4	3138	2849
1	6	-4	756	-586
1	7	-4	691	642
1	8	-4	498	-512
1	9	-4	•	-200
1	10	-4	1328	1181
1	11	-4	3761	3824
1	12	-4	1741	-1792
1	13	-4	2916	-3058
1	14	-4	•	-26
1	15	-4	947	-1084
1	16	-4	1560	-1619
1	17	-4	•	196
1	18	-4	1861	-1921
1	19	-4	889	-1005
1	20	-4	993	1102
1	21	-4	1867	1983
1	22	-4	•	-228
1	23	-4	864	-849
1	24	-4	923	-790
1	25	-4	1178	1185
1	26	-4	870	-772
1	27	-4	•	379
1	28	-4	•	130
1	29	-4	1205	-1194
1	30	-4	969	966
1	31	-4	1539	1415
1	32	-4	•	-391
1	33	-4	•	-288
1	34	-4	•	-207
1	35	-4	•	577
1	36	-4	•	257
1	37	-4	940	887
1	38	-4	•	-236
1	39	-4	•	-435
1	40	-4	•	251
1	41	-4	•	301
1	42	-4	•	-34
1	1	5	817	606
1	2	5	1215	1012
1	3	5	•	-90
1	4	5	2079	-1916
1	5	5	1833	1530
1	6	5	2491	2549
1	7	5	605	-732
1	8	5	3592	-3997
1	9	5	2531	-1809
1	10	5	1414	-1095
1	11	5	923	847
1	12	5	1355	1280
1	13	5	492	397
1	14	5	2212	-2169
1	15	5	•	-265
1	16	5	1541	1537
1	17	5	•	30
1	18	5	651	-787
1	19	5	1010	857
1	20	5	883	1211
1	21	5	•	601
1	22	5	•	446
1	23	5	•	-719
1	24	5	963	-1074
1	25	5	1189	-1205
1	26	5	1753	1861
1	27	5	•	-497
1	28	5	1009	-1393
1	29	5	698	720
1	30	5	1023	-994
1	31	5	620	680
1	32	5	•	-378
1	33	5	•	304
1	34	5	919	-839
1	35	5	•	-265
1	2	-5	1327	950
1	3	-5	•	-408
1	4	-5	2014	-2194
1	5	-5	3513	3444
1	6	-5	1687	1469
1	7	-5	1228	-1022
1	8	-5	1780	-1607
1	9	-5	1334	-1125
1	10	-5	•	340
1	11	-5	2304	2300
1	12	-5	•	-336
1	13	-5	•	-433
1	14	-5	3102	-3214
1	15	-5	618	508
1	16	-5	803	722
1	17	-5	1023	-994
1	18	-5	916	910
1	19	-5	1248	1506
1	20	-5	3513	3719
1	21	-5	2418	2362
1	22	-5	1361	1483
1	23	-5	1281	-802
1	24	-5	1149	-1058
1	25	-5	1209	1003
1	26	-5	770	1497
1	27	-5	•	-405
1	28	-5	1149	-1260
1	29	-5	•	359
1	30	-5	•	-451
1	31	-5	•	710
1	32	-5	•	84
1	33	-5	584	-649
1	34	-5	817	-903
1	35	-5	551	470
1	36	-5	1011	920
1	37	-5	749	-672
1	38	-5	•	-266
1	39	-5	•	340
1	0	6	670	-569
1	1	6	2059	1943
1	2	6	1308	1055
1	3	6	2284	2055
1	4	6	2810	2861
1	5	6	1673	1643
1	6	6	•	5
1	7	6	990	651
1	8	6	631	-756
1	9	6	•	-438
1	10	6	•	393
1	11	6	797	755
1	12	6	•	-105
1	13	6	737	-869
1	14	6	•	250
1	15	6	•	70
1	16	6	•	-5
1	17	6	•	297
1	18	6	•	701
1	19	6	1156	-1208
1	20	6	•	326
1	21	6	•	558
1	22	6	•	444
1	23	6	•	-219
1	24	6	•	291
1	25	6	•	586
1	26	6	•	-311
1	27	6	•	475
1	28	6	•	920
1	29	6	1094	-1116
1	30	6	•	389
1	31	6	624	787
1	32	6	•	291
1	33	6	•	-176
1	0	-6	•	1289
1	1	-6	•	1211
1	2	-6	•	-1076
1	3	-6	•	218
1	4	-6	•	-191
1	5	-6	•	393
1	6	-6	666	656
1	7	-6	1631	1737
1	8	-6	1294	-1155
1	9	-6	•	588
1	10	-6	727	611
1	11	-6	1069	1180
1	12	-6	•	630
1	13	-6	2231	-2321
1	14	-6	2424	2326
1	15	-6	2351	-2263
1	16	-6	1661	1742
1	17	-6	•	544
1	18	-6	1301	-1186
1	19	-6	•	-478
1	20	-6	1301	1146
1	21	-6	1162	923
1	22	-6	•	209
1	23	-6	•	-867
1	24	-6	1441	-1451
1	25	-6	1083	-1091
1	26	-6	•	-362
1	1	7	1249	-1355
1	2	7	1016	1136
1	3	7	•	-247
1	4	7	•	-454
1	5	7	•	-266
1	6	7	•	970
1	7	7	1010	504
1	8	7	•	-910
1	9	7	•	-505
1	10	7	644	-366
1	11	7	•	-29
1	12	7	•	47
1	13	7	•	1273
1	14	7	1029	-935
1	15	7	890	211
1	16	7	•	1532
1	17	7	1249	-203
1	18	7	•	-452
1	19	7	•	-260
1	20	7	•	613
1	21	7	•	11
1	22	7	1024	1213
1	23	7	•	229
1	24	7	848	-894
1	25	7	•	-337
1	26	7	682	760
1	27	7	•	-454
1	28	7	•	-425
2	0	0	1581	-1419
2	1	0	1224	1109
2	2	0	815	839
2	3	0	344	-404
2	4	0	3310	-3029
2	5	0	1860	1529
2	6	0	728	-546
2	7	0	1399	1446
2	8	0	3534	3584
2	9	0	3631	-3680
2	10	0	1642	1768
2	11	0	1052	-1013
2	12	0	4926	5451
2	13	0	1659	-1938
2	14	0	955	-851
2	15	0	850	815
2	16	0	481	432
2	17	0	1484	1566
2	18	0	2175	2118
2	19	0	1537	-1666
2	20	0	2270	-2241
2	21	0	1773	1742
2	22	0	1467	1485
2	23	0	•	-63
2	24	0	•	-11
2	25	0	880	853
2	26	0	•	-224
2	27	0	•	-259
2	28	0	•	-228
2	29	0	2648	-2626
2	30	0	1705	-1536
2	31	0	1498	1565
2	32	0	1115	1160
2	33	0	1241	-1252
2	34	0	715	-486
2	35	0	1217	-972
2	36	0	289	-219
2	37	0	818	832
2	38	0	509	563
2	39	0	•	1
2	40	0	709	-503
2	41	0	943	784
2	42	0	•	956
2	1	1	1092	1155
2	2	1	938	-590
2	3	1	4009	4172
2	4	1	990	839
2	5	1	3495	-3602
2	6	1	2837	2630
2	7	1	3026	3129
2	8	1	2026	-1820
2	9	1	2000	1833
2	10	1	1112	-905
2	11	1	612	-578
2	12	1	660	-599
2	13	1	•	52
2	14	1	560	710
2	15	1	2087	-2234
2	16	1	1646	1689
2	17	1	626	415
2	18	1	2093	-2156
2	19	1	1918	-1880
2	20	1	805	782
2	21	1	413	218
2	22	1	1767	1863
2	23	1	3322	3215
2	24	1	1122	1211
2	25	1	914	839
2	26	1	1325	1148
2	27	1	1732	1729
2	28	1	1024	-969
2	29	1	•	157
2	30	1	•	277
2	31	1	•	-192
2	32	1	•	420
2	33	1	•	532
2	34	1	•	-210
2	35	1	687	-679
2	1	-1	•	-549
2	2	-1	653	-674
2	3	-1	426	491
2	4	-1	1953	1838
2	5	-1	1814	-2138
2	6	-1	5245	5786
2	7	-1	508	-419
2	8	-1	337	-324
2	9	-1	907	-1007
2	10	-1	1979	-1952
2	11	-1	2118	-2174
2	12	-1	•	107
2	13	-1	857	-740
2	14	-1	1956	-1850
2	15	-1	2147	-2344
2	16	-1	•	-27
2	17	-1	496	564
2	18	-1	748	-819
2	19	-1	918	-871
2	20	-1	726	-715
2	21	-1	887	-843
2	22	-1	•	166
2	23	-1	694	528
2	24	-1	1464	-1376
2	25	-1	1803	-1800
2	26	-1	•	222
2	27	-1	1992	2102
2	28	-1	•	-172
2	29	-1	743	-588
2	30	-1	•	-264
2	31	-1	1271	-1266
2	32	-1	•	56
2	33	-1	1555	1529
2	34	-1	844	-798
2	35	-1	1589	-1479
2	36	-1	•	120
2	0	2	4553	-4686
2	1	2	2343	2084
2	2	2	632	-532
2	3	2	1892	-1753
2	4	2	1614	-1497
2	5	2	495	277
2	6	2	1345	-1217
2	7	2	2723	2737
2	8	2	2424	2269
2	9	2	427	-331
2	10	2	1662	-1526
2	11	2	1406	1469
2	12	2	1786	1540
2	13	2	804	-647
2	14	2	274	-249
2	15	2	1115	948
2	16	2	1736	-1771
2	17	2	283	-273
2	18	2	1471	1479
2	19	2	2208	-2364
2	20	2	3066	-3121
2	21	2	1049	971
2	22	2	598	-617
2	23	2	•	-302
2	24	2	1141	877
2	25	2	•	211
2	26	2	•	-246
2	27	2	•	-344
2	28	2	•	347
2	29	2	770	-766
2	30	2	1430	-1467
2	31	2	1032	1109
2	32	2	578	460
2	33	2	•	13
2	34	2	•	-182
2	0	-2	2268	-2138
2	1	-2	5788	5942
2	2	-2	344	-70
2	3	-2	2955	-2663
2	4	-2	2736	-2710
2	5	-2	1594	-1521
2	6	-2	1826	1889
2	7	-2	2697	2612
2	8	-2	2168	2037
2	9	-2	3426	-3605
2	10	-2	4381	-4560
2	11	-2	1536	1823
2	12	-2	650	796
2	13	-2	1581	-1619
2	14	-2	•	-72
2	15	-2	1323	1216
2	16	-2	•	-55
2	17	-2	1571	1490
2	18	-2	1321	1186
2	19	-2	1477	-1417
2	20	-2	1847	-1793
2	21	-2	2444	2306
2	22	-2	•	-347
2	23	-2	•	-274
2	24	-2	1726	-1710
2	25	-2	•	460
2	26	-2	•	-4
2	27	-2	•	590
2	28	-2	1757	1667
2	29	-2	•	-245
2	30	-2	•	-320
2	31	-2	•	660
2	32	-2	•	380
2	33	-2	1189	-1108
2	34	-2	•	-52
2	35	-2	•	35
2	1	3	825	-805
2	2	3	667	-581
2	3	3	914	806
2	4	3	2060	-1988
2	5	3	2759	-2642
2	6	3	413	-159
2	7	3	1952	1904
2	8	3	1304	1193
2	9	3	722	-627
2	10	3	735	-238
2	11	3	2290	-2443
2	12	3	523	-474
2	13	3	1429	1350
2	14	3	1720	-1561
2	15	3	639	-642
2	16	3	•	-12
2	17	3	•	552
2	18	3	578	214
2	19	3	761	-826
2	20	3	763	-856
2	21	3	•	-165
2	22	3	825	820
2	23	3	783	932
2	24	3	•	-228
2	25	3	1424	-1405
2	26	3	•	-444
2	27	3	873	910
2	28	3	•	-230
2	29	3	•	-472
2	30	3	•	-136
2	31	3	1066	-987
2	32	3	•	30
2	33	3	•	435
2	34	3	427	-623
2	35	3	818	-1015
2	36	3	644	-571
2	37	3	•	-77
2	38	3	•	-742
2	1	-3	2406	-2165
2	2	-3	2592	2368
2	3	-3	5638	5635
2	4	-3	604	-676
2	5	-3	922	-801
2	6	-3	907	-743
2	7	-3	2620	2497
2	8	-3	•	-127
2	9	-3	769	684
2	10	-3	762	676
2	11	-3	•	-58
2	12	-3	1235	1258
2	13	-3	1289	1213
2	14	-3	1527	-1600
2	15	-3	1425	-1565
2	16	-3	900	968
2	17	-3	2481	2429
2	18	-3	722	-719
2	19	-3	743	677
2	20	-3	1106	-1071
2	21	-3	•	-91
2	22	-3	2184	2064
2	23	-3	728	636
2	24	-3	•	-160
2	25	-3	•	-180
2	26	-3	984	948
2	27	-3	1562	1667

Table 1 (*cont.*)

H	K	L	100FO	100FC
2	28	-3	1032	816
2	29	-3	•	240
2	30	-3	•	60
2	0	4	1014	-808
2	1	4	825	699
2	2	4	1662	1641
2	3	4	1655	-1571
2	4	4	867	-619
2	5	4	756	557
2	6	4	1107	1007
2	7	4	1373	1373
2	8	4	1375	1316
2	9	4	1615	-1565
2	10	4	1555	-1516
2	11	4	1232	1277
2	12	4	2756	2906
2	13	4	985	-925
2	14	4	591	-363
2	15	4	1115	-1108
2	16	4	•	26
2	17	4	•	386
2	18	4	1038	1213
2	19	4	502	583
2	20	4	965	-853
2	21	4	950	1192
2	22	4	1004	1217
2	23	4	838	-865
2	24	4	984	-1018
2	25	4	385	501
2	26	4	•	-256
2	27	4	•	464
2	28	4	936	812
2	29	4	•	-536
2	30	4	•	-121
2	31	4	•	165
2	32	4	915	1415
2	33	4	•	-212
2	34	4	•	-69
2	35	4	•	203
2	36	4	•	-375
2	0	-4	3189	-2945
2	1	-4	2269	2179
2	2	-4	810	-672
2	3	-4	1004	-1036
2	4	-4	•	-181
2	5	-4	584	-507
2	6	-4	186	-213
2	7	-4	365	309
2	8	-4	2184	2242
2	9	-4	1818	-1742
2	10	-4	862	949
2	11	-4	2610	2767
2	12	-4	1774	1805
2	13	-4	715	-606
2	14	-4	•	66
2	15	-4	1130	-1196
2	16	-4	1454	-1439
2	17	-4	584	-325
2	18	-4	1230	1120
2	19	-4	701	-470
2	20	-4	776	-803
2	21	-4	1790	2000
2	22	-4	•	400
2	23	-4	2286	-2318
2	24	-4	523	517
2	25	-4	•	-167
2	26	-4	1193	-1163
2	27	-4	888	875
2	28	-4	•	390
2	29	-4	2189	-2088
2	30	-4	•	-64
2	1	5	825	-511
2	2	5	1196	1213
2	3	5	2173	1911
2	4	5	907	979
2	5	5	•	70
2	6	5	•	361
2	7	5	•	-229
2	8	5	•	-305
2	9	5	•	-78
2	10	5	701	870
2	11	5	•	76
2	12	5	•	-345
2	13	5	•	-19
2	14	5	632	-665
2	15	5	1107	-1338
2	16	5	•	312
2	17	5	•	624
2	18	5	•	236
2	19	5	•	53
2	20	5	•	585
2	21	5	•	73
2	22	5	•	-129
2	23	5	915	1029
2	24	5	•	445
2	25	5	873	-991
2	26	5	•	465
2	27	5	•	761
2	28	5	557	-502
2	29	5	•	442
2	1	-5	•	-636
2	2	-5	1128	1018
2	3	-5	632	651
2	4	-5	•	117
2	5	-5	1736	-1766
2	6	-5	1304	1336
2	7	-5	770	672
2	8	-5	791	-672
2	9	-5	447	-391
2	10	-5	2574	2685
2	11	-5	598	-536
2	12	-5	•	-293
2	13	-5	•	-401
2	14	-5	3856	-4030
2	15	-5	2687	-2813
2	16	-5	•	-57
2	17	-5	1291	-1315
2	18	-5	•	-15
2	19	-5	1696	-1765
2	20	-5	1080	1129
2	21	-5	•	552
2	22	-5	•	-131
2	23	-5	1045	1070
2	24	-5	942	-955
2	25	-5	728	-777
2	26	-5	956	910
2	27	-5	1417	1267
2	28	-5	694	-657
2	29	-5	•	-77
2	0	6	2565	-2905
2	1	6	1306	818
2	2	6	•	-529
2	3	6	1306	1395
2	4	6	•	-675
2	5	6	•	420
2	6	6	•	116
2	7	6	•	-431
2	8	6	942	943
2	9	6	•	-141
2	10	6	1168	-1270
2	11	6	•	45
2	12	6	1299	1173
2	13	6	•	-487
2	14	6	•	-127
2	15	6	•	34
2	16	6	•	-516
2	17	6	•	-106
2	18	6	•	443
2	19	6	•	-479
2	20	6	936	-1129
2	9	-6	1342	-1428
2	10	-6	•	-391
2	11	-6	873	893
2	12	-6	1753	2057
2	13	-6	1415	-1481
2	14	-6	•	-128
2	15	-6	1085	-1159
2	16	-6	•	95
2	17	-6	1368	1504
2	18	-6	•	-108
2	19	-6	1318	1358
2	20	-6	•	-645
2	21	-6	2262	2482
2	22	-6	•	445
2	23	-6	•	-721
2	24	-6	•	-394
2	25	-6	•	91
2	26	-6	•	645
2	27	-6	•	995
2	28	-6	•	48
2	29	-6	•	-1245
2	30	-6	•	-908
2	31	-6	•	205
2	32	-6	•	255
2	33	-6	•	-682
2	34	-6	•	-81
2	1	7	1224	-1104
2	2	7	•	331
2	3	7	1299	1472
3	0	0	1967	-1845
3	1	0	2891	-2963
3	2	0	1404	1416
3	3	0	•	140
3	4	0	801	-810
3	5	0	1073	818
3	6	0	862	-842
3	7	0	1506	-1453
3	8	0	1764	1615
3	9	0	•	65
3	10	0	•	-252
3	11	0	2199	-2183
3	12	0	692	748
3	13	0	896	847
3	14	0	1825	-1782
3	15	0	1778	1817
3	16	0	868	-800
3	17	0	•	-160
3	18	0	665	-686
3	19	0	671	572
3	20	0	1126	-1139
3	21	0	•	-231
3	22	0	2009	1865
3	23	0	1031	1069
3	24	0	•	83
3	25	0	549	526
3	26	0	•	257
3	27	0	570	-466
3	28	0	779	824
3	29	0	665	604
3	30	0	•	-192
3	31	0	•	-129
3	32	0	•	344
3	33	0	•	-31
3	34	0	563	-561
3	35	0	•	-296
3	36	0	•	-224
3	37	0	•	-253
3	1	1	1628	-1428
3	2	1	577	-334
3	3	1	1187	1078
3	4	1	998	820
3	5	1	2124	-2136
3	6	1	•	360
3	7	1	998	845
3	8	1	746	657
3	9	1	•	-79
3	10	1	•	-170
3	11	1	•	272
3	12	1	787	-683
3	13	1	1207	1088
3	14	1	2625	2397
3	15	1	2735	-2716
3	16	1	•	-286
3	17	1	•	-114
3	18	1	2036	-2184
3	19	1	984	-953
3	20	1	1364	-1126
3	21	1	1472	-1308
3	22	1	848	-1009
3	23	1	848	992
3	24	1	760	754
3	25	1	•	-211
3	26	1	726	-622
3	27	1	•	404
3	28	1	•	-326
3	29	1	•	-453
3	30	1	468	556
3	31	1	•	-64
3	32	1	•	80
3	33	1	•	163
3	34	1	•	438
3	35	1	587	-730
3	36	1	•	-404
3	1	-1	•	-1118
3	2	-1	773	-641
3	3	-1	420	485
3	4	-1	1187	1162
3	5	-1	489	-316
3	6	-1	203	153
3	7	-1	2212	2326
3	8	-1	1344	1307
3	9	-1	408	303
3	10	-1	916	-861
3	11	-1	1391	1347
3	12	-1	1100	-884
3	13	-1	1723	1829
3	14	-1	760	670
3	15	-1	1540	-1523
3	16	-1	•	-364
3	17	-1	2158	2138
3	18	-1	1187	1083
3	19	-1	•	-141
3	20	-1	•	-201
3	21	-1	•	-522
3	22	-1	•	-415
3	23	-1	801	830
3	24	-1	604	592
3	25	-1	1242	-1091
3	26	-1	•	-87
3	27	-1	1513	1531
3	28	-1	•	-35
3	29	-1	•	-411
3	30	-1	•	321
3	31	-1	•	-130
3	32	-1	596	576
3	33	-1	618	705
3	34	-1	•	-363
3	35	-1	929	-1123
3	36	-1	•	-402
3	37	-1	676	849
3	0	2	1207	-1088
3	1	2	1723	-1665
3	2	2	679	443
3	3	2	190	-263
3	4	2	679	-405
3	5	2	990	-986
3	6	2	685	-410
3	7	2	990	-890
3	8	2	1662	1661
3	9	2	1133	1075
3	10	2	990	-750
3	11	2	1445	-1576
3	12	2	644	498
3	13	2	1051	1117
3	14	2	1533	1612
3	15	2	977	952
3	16	2	•	-76
3	17	2	•	-280
3	18	2	•	197
3	19	2	•	-270
3	20	2	596	-668
3	21	2	1046	-1119
3	22	2	549	544
3	23	2	1242	1526
3	24	2	•	174
3	25	2	•	-252
3	26	2	•	-279
3	27	2	882	-1028
3	28	2	557	536
3	29	2	557	597
3	30	2	780	-894
3	31	2	•	-426
3	32	2	•	-141
3	33	2	•	284
3	34	2	•	-167
3	0	-2	1608	-1420
3	1	-2	1289	-1161
3	2	-2	733	630
3	3	-2	•	-55
3	4	-2	536	383
3	5	-2	1207	949
3	6	-2	706	568
3	7	-2	•	-409
3	8	-2	1011	1037
3	9	-2	•	-117
3	10	-2	1818	-1703
3	11	-2	455	-308
3	12	-2	•	-121
3	13	-2	591	586
3	14	-2	•	-140
3	15	-2	584	-549
3	16	-2	•	-93
3	17	-2	387	-429
3	18	-2	1201	1129
3	19	-2	1051	987
3	20	-2	848	-831
3	21	-2	1018	-1072
3	22	-2	•	33
3	23	-2	•	29
3	24	-2	1147	-1100
3	25	-2	665	-663
3	26	-2	•	239
3	27	-2	692	-673
3	28	-2	1838	1788
3	29	-2	685	768
3	30	-2	1207	-1220
3	31	-2	1282	-1324
3	32	-2	•	231
3	33	-2	•	455
3	34	-2	•	1
3	35	-2	•	246
3	36	-2	745	-605
3	37	-2	•	-579
3	1	3	•	353
3	2	3	1880	-1576
3	3	3	936	967
3	4	3	•	-54
3	5	3	963	-860
3	6	3	•	211
3	7	3	1833	1961
3	8	3	1038	974
3	9	3	1051	-1100
3	10	3	767	-673
3	11	3	•	-148
3	12	3	•	292
3	13	3	1493	1508
3	14	3	•	511
3	15	3	882	-924
3	16	3	1344	-1202
3	17	3	746	929
3	18	3	753	895
3	19	3	624	-761
3	20	3	•	350
3	21	3	•	41
3	22	3	•	-40
3	23	3	•	572
3	24	3	596	577
3	25	3	509	-300
3	26	3	•	-199
3	27	3	•	1061
3	28	3	•	545
3	29	3	591	-162
3	30	3	340	267
3	31	3	•	503
3	1	-3	1031	-1043
3	2	-3	1723	1799
3	3	-3	753	659
3	4	-3	1180	-1109
3	5	-3	2931	-2792
3	6	-3	1547	-1305
3	7	-3	3073	2928
3	8	-3	868	749
3	9	-3	1567	-1462
3	10	-3	400	438
3	11	-3	1859	-1684
3	12	-3	•	277
3	13	-3	1309	1212
3	14	-3	•	267
3	15	-3	1900	-1947
3	16	-3	611	-661
3	17	-3	929	889
3	18	-3	197	-307
3	19	-3	998	-896
3	20	-3	•	-316
3	21	-3	1025	-908
3	22	-3	•	-357
3	23	-3	•	-279
3	24	-3	•	179
3	25	-3	1398	-1243
3	26	-3	•	-233
3	27	-3	1222	1160
3	28	-3	•	399
3	29	-3	•	203
3	30	-3	•	100
3	31	-3	•	71
3	32	-3	•	-240
3	33	-3	•	230
3	34	-3	509	551
3	35	-3	•	-180
3	36	-3	•	-11
3	0	4	902	-919
3	1	4	875	-775
3	2	4	•	221
3	3	4	•	33
3	4	4	•	146
3	5	4	•	213
3	6	4	•	-501
3	7	4	•	-295
3	8	4	•	-335
3	9	4	998	1053
3	10	4	•	149
3	11	4	•	-199
3	12	4	•	247
3	13	4	•	-397
3	14	4	753	-857
3	15	4	•	-249
3	16	4	•	-45
3	17	4	•	-129
3	18	4	•	-121
3	19	4	1078	1190
3	20	4	584	-623
3	21	4	340	-648
3	22	4	•	398
3	0	-4	2265	-2420
3	1	-4	1289	-1295
3	2	-4	1513	1499
3	3	-4	848	760
3	4	-4	•	194
3	5	-4	2097	-1795
3	6	-4	1710	-1689
3	7	-4	2565	-2350
3	8	-4	1656	1603
3	9	-4	2171	1930
3	10	-4	•	142
3	11	-4	•	302
3	12	-4	1147	938
3	13	-4	549	596
3	14	-4	801	-829
3	15	-4	482	-409
3	16	-4	889	-876
3	17	-4	•	310
3	18	-4	1452	1411
3	19	-4	780	786
3	20	-4	868	-864
3	21	-4	•	-221
3	22	-4	•	605
3	23	-4	909	812
3	24	-4	658	613
3	25	-4	•	-333
3	26	-4	•	-78
3	27	-4	•	468
3	28	-4	•	450
3	29	-4	•	697
3	30	-4	679	-605
3	31	-4	549	-506
3	32	-4	•	473
3	33	-4	•	171
3	34	-4	•	-207
3	1	5	767	-677
3	2	5	•	120
3	3	5	•	-398
3	4	5	1004	1108
3	5	5	882	-442
3	6	5	713	-998
3	7	5	286	694
3	8	5	•	533
3	9	5	•	-33
3	10	5	•	344
3	11	5	•	120
3	12	5	•	-697
3	13	5	•	345
3	14	5	•	270
3	15	5	638	-774
3	16	5	618	-795
3	1	-5	•	-444
3	2	-5	•	-234
3	3	-5	•	482
3	4	-5	•	-148
3	5	-5	1567	-1489
3	6	-5	•	11
3	7	-5	1500	1565
3	8	-5	509	443
3	9	-5	1656	1571
3	10	-5	1833	1581
3	11	-5	1228	1098
3	12	-5	679	601
3	13	-5	1445	1420
3	14	-5	211	-322
3	15	-5	1317	-1309
3	16	-5	1147	-1136
3	17	-5	644	557
3	18	-5	•	65
3	19	-5	•	-640
3	20	-5	1174	1239
3	21	-5	509	-657
3	22	-5	1588	-1515
3	23	-5	1594	1650
3	24	-5	1073	-1093
3	25	-5	•	-395
3	26	-5	591	616
3	0	6	1153	-1351
3	1	6	•	-525
3	2	6	•	187
3	3	6	•	740
3	4	6	•	490
3	5	6	•	-394
3	6	6	•	311
3	7	6	909	-1358
3	8	6	•	727
3	0	-6	•	-649
3	1	-6	•	-693
3	2	-6	•	912
3	3	-6	•	390
3	4	-6	•	-767
3	5	-6	•	84
3	6	-6	•	127
3	7	-6	1100	-1197
3	8	-6	882	997
3	9	-6	746	679
3	10	-6	1126	-1100
3	11	-6	•	227
3	12	-6	•	527
3	13	-6	902	-967
3	14	-6	1174	-1137
3	15	-6	773	-801
3	16	-6	408	-538
3	17	-6	•	440
3	18	-6	733	-651
3	19	-6	1004	1000
3	20	-6	1323	-1454
3	21	-6	462	-570
3	22	-6	1031	1208
3	23	-6	•	-209

At this stage the refinement was transferred to the University of Leeds KDF9 using the program of Cruickshank & Smith (1966). During the subsequent block-diagonal least-squares refinement of positional, thermal and scale factors, the hydrogen atoms were fixed at distances of 1·08 Å for those attached to the phenyl rings and at 0·97 Å for that bonded to O(1). The weighting scheme

$$\sqrt{w}=1\Big/\left\{2|F_{\min}|+|F_o|+\frac{2|F_o|^2}{|F_{\max}|}+\frac{5|F_o|^3}{|F_{\max}|^2}\right\}^{\frac{1}{2}},$$

was employed, it being found necessary to include the F_o^3 term in the expression to discriminate against the strongest reflexions. The refinement was concluded with a residual for the 1028 observed reflexions of R=0·08. A final three-dimensional electron density distribution and a difference electron density distribution were calculated.

The final structure factors are given in Table 1; those reflexions which were not observed in the region of reciprocal space examined, either from camera geometry or from being too weak to measure, are marked with an asterisk. Table 2 gives the heavy atom coordinates and Table 3 their thermal parameters. The hydrogen atom parameters are quoted in Table 4.

Table 2(*a*). *The final coordinates with their e.s.d.'s* (*in parenthesis*)

	X/a	Y/b	Z/c
Cl	−0·1980 (5)	0·2002 (1)	−0·0105 (3)
O(1)	−1·4337 (16)	0·0046 (2)	−0·2697 (8)
O(2)	−0·3709 (17)	0·0407 (2)	−0·4782 (8)
C(1)	0·1600 (19)	0·1476 (2)	0·2153 (10)
C(2)	0·0661 (18)	0·1846 (2)	0·1887 (10)
C(3)	0·1616 (20)	0·2122 (2)	0·3184 (12)
C(4)	0·3714 (21)	0·2021 (2)	0·4842 (12)
C(5)	0·4606 (20)	0·1651 (2)	0·5137 (11)
C(6)	0·3552 (19)	0·1382 (2)	0·3817 (10)
C(7)	0·0579 (18)	0·1173 (2)	0·0795 (10)
C(8)	−0·0729 (19)	0·0841 (2)	0·1305 (10)
C(9)	−0·1858 (20)	0·0563 (2)	0·0004 (11)
C(10)	−0·1494 (19)	0·0616 (2)	−0·1771 (10)
C(11)	0·0061 (19)	0·0944 (2)	−0·2218 (10)
C(12)	0·1106 (19)	0·1219 (2)	−0·0975 (11)
C(13)	−0·2957 (20)	0·0346 (2)	−0·3180 (11)

Table 2(*b*). *Orthogonal coordinates for the heavy atoms with respect to the axes*

$X'=X+Z\cos\beta$; $Y'=Y$ and $Z'=Z\sin\beta$

	X'	Y'	Z'
Cl	−0·7646	7·1677	−0·0783
O(1)	−1·3137	0·1644	−2·0140
O(2)	−0·3669	1·4585	−3·5709
C(1)	0·3150	5·2870	1·6077
C(2)	−0·0159	6·6116	1·4087
C(3)	0·1702	7·5985	2·3773
C(4)	0·7541	7·2386	3·6159
C(5)	1·0624	5·9119	3·8362
C(6)	0·8401	4·9489	2·8502
C(7)	0·1115	4·1987	0·5933
C(8)	−0·4786	3·0131	0·9743
C(9)	−0·7324	2·0143	0·0026
C(10)	−0·3291	2·2066	−1·3223
C(11)	0·3490	3·3815	−1·6565
C(12)	0·5784	4·3649	−0·7280
C(13)	−0·6991	1·2390	−2·3748

The arrangements of the molecules in the unit cell viewed along the *a* axis is shown in Fig. 1. All intermolecular contacts of less than 3·6 Å are given in Table 5; the bond lengths and angles together with some of the intramolecular non-bonded distances are in Table 6. The equations of the mean planes of the ring systems C(1)–C(6); C(1)–C(6) and chlorine; C(7)–C(12) and the group C(10), C(13), O(1) and O(2), calculated with respect to the orthogonal triad *a*,*b*,*c*′, where *c*′ is perpendicular to the *a* and *b* crystal axes, are given in Table 7. The deviations of the atoms from these planes are also quoted. The angle between the carboxylic group and the phenyl ring C(7)–C(12) is 5·6° and the angle between the two phenyl rings 48·9°.

Discussion

The angle of 48·9° between the two phenyl rings is in good agreement with the values of 49·2° and 46·1° for 4-acetyl-2′-chlorobiphenyl (Sutherland & Hoy, 1967*a*),

Table 3. *Thermal parameters* (Å²) *for the heavy atoms*

	U_{11}	U_{22}	U_{33}	$2U_{23}$	$2U_{31}$	$2U_{12}$
Cl	0·0482	0·0374	0·0461	0·0139	0·0187	0·0069
O(1)	0·0701	0·0472	0·0357	−0·0148	0·0154	−0·0462
O(2)	0·0837	0·0509	0·0313	−0·0051	0·0306	−0·0378
C(1)	0·0362	0·0419	0·0288	−0·0246	0·0337	−0·0212
C(2)	0·0295	0·0385	0·0319	−0·0059	0·0167	−0·0085
C(3)	0·0382	0·0446	0·0463	−0·0287	0·0267	−0·0247
C(4)	0·0419	0·0496	0·0467	−0·0277	0·0259	−0·0265
C(5)	0·0363	0·0616	0·0373	−0·0063	0·0335	−0·0184
C(6)	0·0391	0·0501	0·0221	−0·0225	0·0125	−0·0117
C(7)	0·0256	0·0331	0·0366	−0·0036	0·0139	−0·0019
C(8)	0·0456	0·0372	0·0185	−0·0032	0·0254	−0·0109
C(9)	0·0412	0·0395	0·0381	−0·0029	0·0263	−0·0103
C(10)	0·0433	0·0375	0·0200	0·0003	0·0196	0·0016
C(11)	0·0378	0·0364	0·0233	0·0163	0·0211	0·0045
C(12)	0·0367	0·0318	0·0327	−0·0065	0·0254	−0·0155
C(13)	0·0430	0·0337	0·0371	−0·0019	0·0060	−0·0150

Table 4. *Coordinates of the hydrogen atoms*

H(1) is attached to O(1) and the numbers of the remaining hydrogens correspond to that of the carbon atom to which they are attached.

	X/a	*Y/b*	*Z/c*
H(1)	−0·556	−0·014	−0·375
H(3)	0·075	0·242	0·292
H(4)	0·468	0·223	0·586
H(5)	0·606	0·157	0·639
H(6)	0·415	0·125	0·414
H(8)	−0·094	0·080	0·265
H(9)	−0·307	0·030	0·041
H(11)	0·035	0·098	−0·359
H(12)	0·230	0·148	−0·135

Table 5. *Intermolecular distances between atoms less than* 3·6 Å *apart* (*excluding hydrogen atoms*)

i	*j*	equi-point indication	d_{ij}
O(1)	C(13)	$1+x, y, z$	3·513 Å
O(1)	C(9)	$1-x, \bar{y}, \bar{z}$	3·518
C(2)	Cl	$1+x, y, z$	3·564
C(4)	C(3)	$1+x, y, z$	3·595
C(7)	C(8)	$1+x, y, z$	3·574
C(9)	C(11)	$1+x, y, z$	3·577
C(10)	C(11)	$1+x, y, z$	3·483
O(1)	O(1)	$1-x, \bar{y}, 1-z$	3·459
O(1)	C(13)	$1-x, \bar{y}, 1-z$	3·415
O(1)	O(2)	$1-x, \bar{y}, 1-z$	2·610
O(2)	O(2)	$1-x, \bar{y}, 1-z$	3·413
O(2)	C(13)	$1-x, \bar{y}, 1-z$	3·403
O(2)	C(8)	$x, y, 1+z$	3·576

and 2-chlorobiphenyl-4-carboxylic acid (Sutherland & Hoy, 1967*b*) respectively. The displacement of the chlorine atom from the phenyl ring C(1)–C(6) is −0·056 Å. This corresponds to the C(2)–Cl bond bend-

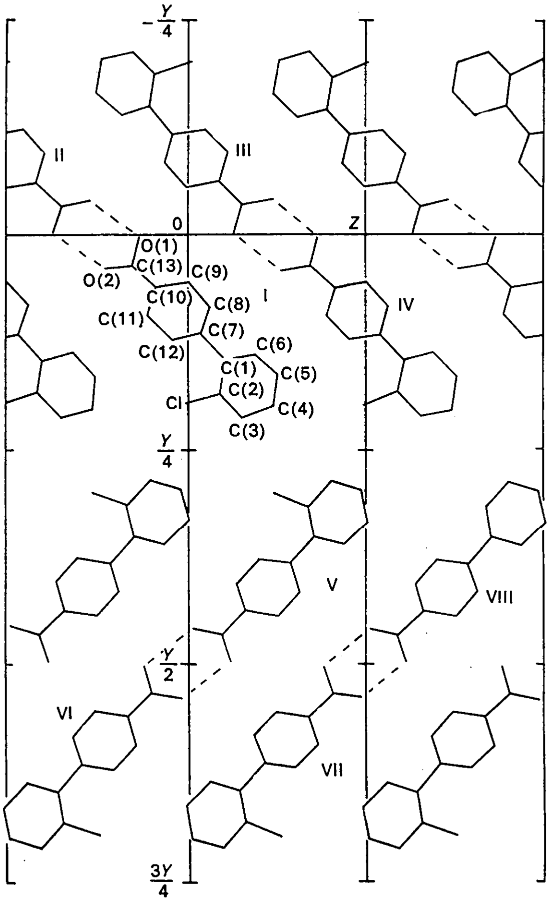

Fig. 1. The arrangement of the molecules in the unit cell viewed along the *a* axis. The symmetry code is as follows: I. x, y, z; II. $\bar{x}-1, \bar{y}, \bar{z}-1$; III. $\bar{x}, \bar{y}, \bar{z}$; IV. $x+1, y, z+1$; V. $x, \frac{1}{2}-y, \frac{1}{2}+z$; VI. $\bar{x}-1, \frac{1}{2}+y, -\frac{1}{2}-z$; VII. $\bar{x}, \frac{1}{2}+y, \frac{1}{2}-z$; VIII. $x+1, \frac{1}{2}-y, \frac{3}{2}+z$.

Table 6. *Bond lengths and bond angles with their estimated standard deviations* (*e.s.d.'s*)

	Length	e.s.d.
Cl—C(2)	1·755 Å	0·008 Å
Cl—C(7)	3·168	0·008
Cl—C(12)	3·177	0·008
O(1)–C(9)	2·798	0·010
O(1)–C(10)	2·370	0·010
O(1)–C(13)	1·289	0·010
O(2)–C(10)	2·370	0·010
O(2)–C(11)	2·806	0·010
O(2)–C(13)	1·261	0·010
C(1)–C(2)	1·380	0·010
C(1)–C(6)	1·391	0·011
C(1)–C(7)	1·502	0·010

	Length	e.s.d.
C(2)—C(3)	1·395 Å	0·011 Å
C(3)—C(4)	1·416	0·012
C(4)—C(5)	1·380	0·012
C(5)—C(6)	1·396	0·011
C(7)—C(8)	1·378	0·010
C(7)—C(12)	1·411	0·010
C(8)—C(9)	1·416	0·011
C(9)—C(10)	1·398	0·011
C(10)–C(11)	1·397	0·011
C(10)–C(13)	1·477	0·011
C(11)–C(12)	1·372	0·011

	Angle	e.s.d.
Cl—C(2)—C(1)	121·9°	0·6°
Cl—C(2)—C(3)	114·9	0·6
O(1)–C(13)–O(2)	112·4	0·7
O(1)–C(13)–C(10)	117·8	0·7
C(2)–C(1)—C(7)	124·5	0·7
C(1)–C(2)—C(3)	123·2	0·7
C(6)–C(1)—C(7)	118·6	0·7
C(1)–C(6)—C(5)	121·6	0·7
C(1)–C(7)—C(8)	119·7	0·7
C(1)–C(7)—C(12)	120·2	0·7

	Angle	e.s.d.
C(2)—C(3)—C(4)	118·9°	0·8°
C(3)—C(4)—C(5)	118·4	0·8
C(4)—C(5)—C(6)	121·0	0·8
C(8)—C(7)—C(12)	120·1	0·7
C(8)—C(9)—C(10)	120·1	0·7
C(9)—C(10)-C(11)	118·8	0·7
C(9)—C(10)-C(13)	120·9	0·7
C(11)-C(10)-C(13)	120·1	0·7
C(10)-C(11)-C(12)	121·5	0·7

ing out of the plane of the benzene ring through an angle of 1·8°. A similar displacement was reported in *o*-chlorobenzoic acid by Ferguson & Sim (1961). The C(2)–Cl bond length of 1·755 ± 0·008 is significantly longer than the value of 1·70 ± 0·01 quoted in Volume III of *International Tables for X-ray Crystallography*. Values for the C–Cl bond length of: 1·753 Å in 2-chloro-5-nitrobenzoic acid (Ferguson & Sim, 1962); 1·737 Å in *o*-chlorobenzoic acid (Ferguson & Sim, 1961); 1·738 Å in 4-acetyl-2′-chlorobiphenyl (Sutherland & Hoy, 1967*a*), do not differ significantly from that obtained in the present determination.

The C(1)–C(7) bond length of 1·502 ± 0·010 is similar to that of 1·506 ± 0·017 Å for biphenyl (Hargreaves & Rizvi, 1962) and is not significantly different from the biphenyl bond lengths of 1·487 ± 0·010 Å in 2-chlorobiphenyl-4-carboxylic acid, 1·490 ± 0·010 Å in 4-acetyl-2′-chlorobiphenyl and 1·479 ± 0·010 Å in 4 acetyl-2′ fluorbiphenyl.

The exocyclic C(10)–C(13) bond in the structure under investigation was determined as 1·477 ± 0·011 Å. This compares favourably with the value of 1·482 ± 0·010 Å for 2-chlorobiphenyl-4-carboxylic acid.

Neither the C(1)–C(7) bond nor the C(10)–C(13) bond differ significantly from the theoretical value of 1·477 quoted by Dewar & Schmeising (1959) and Cruickshank & Sparks (1960) for the (sp^2) single bond length between trigonally linked carbon atoms.

Biphenyl in the solid phase is planar. The introduction of the chlorine atom in the 2′ position causes a strain which results in the twisting of the two phenyl rings about the phenyl bond and the displacement of the chlorine out of the plane of the phenyl ring C(1)–C(6). As seen from Table 7, the carboxyl group is displaced from the plane of the ring C(7)–C(12) and also rotated about the C(10)–C(13) bond. It may be that there is a relayed steric effect similar to that observed by Ferguson & Sim (1962) in 2-chloro-5-nitrobenzoic acid. This effect is almost certainly present in 2-chlorobiphenyl-4-carboxylic acid where a rotation of 7·9° was observed.

The C(13)–O(1) bond of 1·289 ± 0·070 Å and C(13)–O(2) bond of 1·261 ± 0·010 Å are respectively shorter and longer than had been expected. The corresponding bonds in 2-chlorobiphenyl-4-carboxylic acid are 1·315 ± 0·010 Å and 1·205 ± 0·010 Å. In benzoic acid Sim, Robertson & Goodwin (1955) quote 1·29 Å and 1·24 Å for the corresponding bond lengths. The O–H···O bond of 2·61 Å, which is the shortest intermolecular distance, is similar to the values quoted for benzoic acid of 2·64 Å, *o*-chlorobenzoic acid of 2·63 Å and for 2-chlorobiphenyl-4-carboxylic acid of 2·62 Å.

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References

Branch, S. G., Byron, D. J., Gray, G. W., Ibbotson, A. & Worrall, B. M. (1964). *J. Chem. Soc.* p. 3279.

Byron, D. J., Gray, G. W. & Wilson, R. C. (1966). *J. Chem. Soc.* p. 840.

Cruickshank, D. W. J. & Smith, J. G. F. (1966). IUC, *World List of Crystallographic Computer Programs.* 2nd ed. Program 3512.

Cruickshank, D. W. J., Pilling, D. E., Bujosa, A., Lovell, F. M. & Truter, M. R. (1961). *Computing Methods and the Phase Problem in* X-*ray Crystal Analysis.* Oxford: Pergamon Press.

Table 7. *Deviations of the atoms in Å from the planes*

(i) $0{\cdot}9119X' + 0{\cdot}1706Y - 0{\cdot}3733Z' = 0{\cdot}5635$ C(1)–C(6) and Cl
(ii) $-0{\cdot}8808X' + 0{\cdot}4164Y - 0{\cdot}2255Z' = 1{\cdot}4848$ C(7)–C(12)
(iii) $0{\cdot}9175X' + 0{\cdot}1665Y - 0{\cdot}3612Z' = 0{\cdot}5764$ C(1)–C(6)
(iv) $-0{\cdot}8724X' + 0{\cdot}4676Y - 0{\cdot}1422Z' = 1{\cdot}5121$ C(10, C(13), O(1) and O(2)

	(i)	(ii)	(iii)	(iv)
Cl	−0·009 Å		−0·056 Å	
O(1)		0·194 Å		−0·003 Å
O(2)		0·251		−0·002
C(1)	0·026	0·077	0·012	0·457
C(2)	0·024		0·001	
C(3)	0·001		−0·014	
C(4)	0·009	0·049	0·015	0·700
C(5)	−0·018		−0·003	
C(6)	−0·017		−0·011	
C(7)	0·033	0·031	−0·081	0·270
C(8)		−0·028		0·176
C(9)		−0·002		0·068
C(10)	−0·007	0·002	−0·033	−0·005
C(11)		−0·011		0·000
C(12)		−0·013		0·128
C(13)	−0·103	0·182	−0·154	0·015

CRUICKSHANK, D. W. J. & SPARKS, R. A. (1960). *Proc. Roy. Soc.* A**258**, 270.
DALY, J. J., STEPHENS, F. S. & WHEATLEY, P. J. (1963). M.R.S.A. Final Report No. 52.
DEWAR, M. J. S. & SCHMEISING, H. N. (1959). *Tetrahedron* **5**, 166.
FERGUSON, G. & SIM, G. A. (1961). *Acta Cryst.* **14**, 1262.
FERGUSON, G. & SIM, G. A. (1962). *J. Chem. Soc.* p. 1768.
HARGREAVES, R. & RIZVI, S. H. (1962). *Acta Cryst.* **15**, 365.
International Tables for X-ray Crystallography (1962). Vol. III. Birmingham: Kynoch Press.
PHILLIPS, D. C. (1954). *Acta Cryst.* **7**, 746.
SIM, G. A., ROBERTSON, J. M. & GOODWIN, T. H. (1955). *Acta Cryst.* **8**, 157.
SUTHERLAND, H. H. & HOY, T. G. (1967*a*). To be published.
SUTHERLAND, H. H. & HOY, T. G. (1967*b*). To be published.
WILSON, A. J. C. (1942). *Nature, Lond.* **150**, 151.
YOUNG, D. W., TOLLIN, P. & SUTHERLAND, H. H. (1967). *Acta Cryst.* B**24**, 161.

Acta Cryst. (1969). B**25**, 178

The Crystal Structure of β-Protactinium Pentabromide

BY D. BROWN*, T. J. PETCHER† AND A. J. SMITH
Chemistry Department, The University, Sheffield S3 7HF, England

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Protactinium pentabromide crystallizes in two distinct modifications. A three-dimensional structure analysis of the β form (final $R=9{\cdot}05\%$) shows it to be essentially isostructural with uranium pentachloride. The molecular Pa_2Br_{10} units are bis-octahedral with symmetric bromine bridging (in contrast to U_2Cl_{10}), and are formed by the protactinium atoms occupying one fifth of the octahedral holes of a close-packed bromine lattice. The monoclinic unit cell of β-$PaBr_5$ has $a=8{\cdot}385$, $b=11{\cdot}205$, $c=8{\cdot}950$ Å and $\beta=91{\cdot}1$ °. The structural relationship of β-$PaBr_5$ with other pentahalides and with other protactinium compounds is discussed.

Introduction

Protactinium pentabromide, $PaBr_5$, has previously been reported (Maddock, 1961) on the basis of X-ray powder diffraction data, to possess orthorhombic symmetry with the lattice parameters $a=7{\cdot}25$, $b=12{\cdot}12$ $c=9{\cdot}13$ Å. Recent work at A.E.R.E., Harwell (Brown 1966), again powder studies, indicated that $PaBr_5$ was possibly dimorphic but the powder results were not satisfactorily interpreted. The present single-crystal investigation was undertaken in an attempt to clarify this situation and in order to provide structural data on protactinium pentabromide to permit a comparison with the information then available for niobium pentachloride and pentabromide (Zalkin & Sands, 1958) and protactinium pentachloride (Dodge, Smith, Johnson & Elson, 1967).

Experimental

Protactinium pentabromide was prepared as described previously (Brown & Jones, 1966), and purified by vacuum sublimation at 400°. The compound is highly sensitive to atmospheric moisture, and single crystals were obtained by slow sublimation of small quantities (*ca.* 10 μg) of the product sealed in evacuated thin-walled silica capillaries. Investigation of the resulting crystals confirmed the existence of two forms of this compound. Sublimation at approximately 400–410° resulted in the formation of β-$PaBr_5$ whereas at approximately 390–400° a low temperature modification, α-$PaBr_5$, was obtained. Capillaries invariably contained entirely one modification, not an equilibrium mixture, and complete transformation, *e.g.* $\alpha \rightarrow \beta$, could be achieved by varying the sublimation conditions. Although we have been able to obtain some preliminary results for α-$PaBr_5$ this modification is more difficult to crystallize than β-$PaBr_5$ and crystals suitable for a full structure determination have not yet been prepared. Preliminary investigations indicate that α-$PaBr_5$ possesses monoclinic symmetry with $a=12{\cdot}69$, $b=12{\cdot}82$, $c=9{\cdot}92$ Å; $\beta=108$°, the space group being $P2_1/c$.

The results of oscillation, Weissenberg and precession studies have shown that β-$PaBr_5$ possesses monoclinic symmetry, space group $P2_1/n$ with reflexions systematically absent for $h0l$ with $h+l=2n+1$ and $0k0$ with $k=2n+1$. The unit-cell parameters are $a=8{\cdot}385$, $b=11{\cdot}205$, $c=8{\cdot}950$ Å; $\beta=91{\cdot}1$° (all with an accuracy of $\pm 0{\cdot}3\%$); the calculated density for four $PaBr_5$ molecules per unit cell is 4·98 g.cm^{-3}. The non-standard setting of the space group, $P2_1/n$, rather than the standard $P2_1/c$, was chosen to bring out the

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* Present address: Chemistry Division, Building 220, A.E.R.E., Harwell, Didcot, Berks., England.

† Present address: Chemistry Department, University College, London, England.