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The Crystal Structure of Palladium n-Propyl Mercaptide

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The crystal structure of palladium-n-propyl mercaptide has been determined from Patterson and Fourier syntheses using three-dimensional counter data and refined by least squares. The final R index is 0·053 2833 observed reflexions. The space group is $P\bar{1}$ and the cell constants are (at 75 °F) $a = 13{\cdot}766 \pm 0{\cdot}004$ Å, $b = 10{\cdot}564 \pm 0{\cdot}004$ Å, $c = 11{\cdot}499 \pm 0{\cdot}004$ Å, $\alpha = 98{\cdot}36 \pm 0{\cdot}02°$, $\beta = 111{\cdot}40 \pm 0{\cdot}02°$, $\gamma = 102{\cdot}83 \pm 0{\cdot}02°$. The molecule exists as a hexamer $\{Pd(SC_3H_7)_2\}_6$ in the crystal. The palladium atoms form a six-membered puckered ring. Each adjacent pair of metal atoms is joined by a double mercaptide bridge which is folded along the sulphur–sulphur axis. Each palladium is surrounded by four sulphur atoms in an approximately square planar configuration.

Introduction

This work forms part of a programme of study of the structures of various metal mercaptides. Results of the structure analysis of mercury mercaptides by Bradley & Kunchur (1964, 1965) and Kunchur (1964) show that the sulphur of the mercapto group acts as a bridge between two mercury atoms. This gives rise to interesting structural arrangements in these compounds. Mercaptides of palladium were prepared by Mann & Purdie (1935) and Hayter & Humiec (1964). The ethyl and phenyl derivatives were found to be highly insoluble and hence linear polymeric structures for these compounds were proposed. The compounds of palladium with n-propyl and higher mercaptans are, however, soluble in organic solvents and can be readily crystallized. Preliminary work on single crystals of palladium n-propylmercaptide showed that the unit cell is triclinic and contains six formula units of this compound (Mann & Purdie, 1935). This is consistent with the results of the molecular weight determination of this compound in solution, which showed the molecular complexity n to have a value approaching six. On the basis of these data, Hayter & Humiec (1964) proposed two possible structures for palladium n-propyl mercaptide, both of which are based on square planar palladium (II).

In one structure palladium atoms are at the corners of a regular octahedron and the bridging mercaptide groups lie above each edge. In the other possibility, the palladium atoms form a six-membered planar ring, each adjacent pair of metals being joined by a double mercaptide bridge, which is folded along the sulphur–sulphur axis. A detailed structure analysis of this compound was undertaken to discover the correct one of these possibilities, and to define the molecular geometry.

Experimental

The crystals of palladium n-propyl mercaptide, kindly supplied by Dr Hayter, were small, well defined orange-red needles along the b-axis. The unit-cell dimensions and space group of this compound were determined by Mann & Purdie (1935). Their values are: $a = 14{\cdot}3$, $b = 10{\cdot}3$, $c = 11{\cdot}3$ Å, $\alpha = 98°$, $\beta = 113{\cdot}5°$, $\gamma = 101°$. There are six formula units of this compound in the unit cell. The space group could be $P1$ or $P\bar{1}$, but, the piezoelectric test being negative, they assigned the space group $P\bar{1}$ to the compound.

In the present work, the unit-cell dimensions and the space group were redetermined on the G.E. XRD-6 counter diffractometer using Cu $K\alpha$ radiation. The results are as follows: palladium n-propyl mercaptide $\{Pd(S.C_3H_7)_2\}_6$. Molecular weight: 1539·5. Triclinic: $a = 13{\cdot}766 \pm 0{\cdot}004$ Å, $b = 10{\cdot}564 \pm 0{\cdot}004$ Å, $c = 11{\cdot}499 \pm 0{\cdot}004$ Å, $\alpha = 98{\cdot}36 \pm 0{\cdot}02°$, $\beta = 111{\cdot}40 \pm 0{\cdot}02°$, $\gamma = 102{\cdot}83 \pm 0{\cdot}02°$. The dimensions were obtained using high order axial reflexions under the 'fine conditions' of the diffractometer, at 75 °F. The volume of the unit cell is 1470·2 Å^3. The absorption coefficient for X-rays, $\lambda = 1{\cdot}5404$ Å, $\mu = 188{\cdot}0$ cm^{-1}. The density = 1·73 g.cm^{-3}, calculated on the basis of six formula units of the monomeric compound in the unit cell, agrees approximately with the experimental value of 1·75 g.cm^{-3}. Total number of electrons in the unit cell $F(000) = 768$.

The space group $P\bar{1}$ was assumed to be correct on the basis of the work of the previous authors. The crystals employed for the unit-cell measurements and data collection were needles of dimensions 0·06 × 0·06 × 0·05 mm. For the linear absorption coefficient $\mu = 188{\cdot}0$ cm^{-1}, the value for μr is 0·56. φ-absorption corrections were applied. Data were collected on the G.E. XRD-6 diffractometer with scintillation counter using Cu $K\alpha$ radiation and a balanced pair of cobalt and nickel filters. The stationary-crystal stationary-counter technique was used. All 2929 possible independent reflexions within a range $0° < 2\theta > 100°$ were examined. Of these, 96 were too weak to be measured. α-splitting and the usual 1/Lp corrections were applied and the raw intensities were converted into F^2 and F.

Structure determination

A three-dimensional 'unsharpened' Patterson synthesis was computed. In the space group $P\bar{1}$, there are eight centres of symmetry and since there are six palladium

atoms in the unit cell, the job of fixing the palladium positions was rather complicated. The structure was solved by use of Buerger's 'image seeking' method. A palladium–palladium single weight peak was located and a line was drawn from the origin to the center of the peak. This line was used as an image to locate the remaining palladium atoms. No attempt was made to locate the sulphur atoms at this stage.

Three cycles of structure-factor least squares were carried out for the six palladium atoms. This gave an agreement index $R=0{\cdot}31$. A palladium-phased three-dimensional electron density Fourier series was then computed. This yielded the positions of all the twelve sulphur and two carbon atoms. The agreement index R at this stage was 0·21. A second electron density difference Fourier series was computed, using the coefficients obtained by subtracting the contributions of all palladium and sulphur atoms from the F_{obs}. This gave the coordinates of all but two carbon atoms. The agreement index R for all the atoms located up to this stage was 0·16. Another electron density difference Fourier series was computed; this gave the remaining two carbon atoms. The structure, now essentially determined, was refined by a series of least-squares analyses of the three-dimensional data. Four cycles, with isotropic atomic temperature factors, were carried out on the IBM 7040 computer, using the program written by Okaya and modified by Kartha & Harris. The agreement index R for all observed reflexions fell to 0·11. Another four cycles of refinement with individual anisotropic temperature factors and a block-diagonal approximation, reduced the R to 0·053. A $1/f$ weighting with f corresponding to the contribution of sulphur was adopted in the final stages of the least-squares analysis. This weighting is approximately equivalent to differential synthesis weighting. Atomic scattering factors for palladium, sulphur, and carbon were taken from the *International Tables for Crystallography*, Vol. III (1962). The anomalous corrections $\Delta f'$ and $\Delta f''$ for palladium with Cu $K\alpha$ were applied. The atomic shifts in the final cycle were much smaller than the standard deviations of the atomic positions, and so the refinement was considered to be complete. An electron density difference Fourier series, computed using the final coordinates of all the atoms except the hydrogens, was free from details of any significance. No attempt was made to locate the hydrogen atoms; the calculations described above do not take into account the contributions of hydrogen atoms to the structure factors. The final agreement index R was 0·053 for all observed reflexions and 0·058 if all unobserved reflexions were included. The final atomic and thermal parameters as well as the F_{obs} and F_{calc} are those of the final least-squares cycle. The atomic and thermal parameters are listed in Tables 1 and 7 while the F_{obs} and F_{calc} are listed in Table 2.

Discussion and description of the structure

The various bond lengths, bond angles and their estimated standard deviations are shown in Table 3. The standard deviations in the atomic parameters were obtained by inverting the matrix in the final least-squares cycle. The labelling scheme used to describe the palladium n-propyl mercaptide molecule can be seen in Fig. 1. For every atom in the unit cell, there is another atom

Table 1. *Fractional final coordinates and their standard deviations* (Å)

	x/a	y/b	z/c	σ_X	σ_Y	σ_Z
Pd(1)	0·16796	0·01108	0·27509	0·00070	0·00138	0·00089
Pd(2)	0·01487	0·25220	0·88932	0·00072	0·00140	0·00090
Pd(3)	0·18416	0·26365	0·16557	0·00070	0·00138	0·00088
S(1)	0·0583	0·3800	0·0945	0·0026	0·0049	0·0032
S(2)	0·3049	0·1390	0·2302	0·0024	0·0049	0·0032
S(3)	0·1538	0·2218	0·3455	0·0028	0·0051	0·0033
S(4)	0·8245	0·1997	0·8081	0·0026	0·0051	0·0032
S(5)	0·9739	0·1179	0·6877	0·0026	0·0052	0·0032
S(6)	0·2018	0·2844	0·9750	0·0026	0·0054	0·0033
C(1)	0·948	0·327	0·1469	0·013	0·0251	0·0163
C(2)	0·967	0·422	0·270	0·013	0·025	0·015
C(3)	0·882	0·377	0·317	0·020	0·039	0·025
C(4)	0·307	0·038	0·087	0·010	0·023	0·013
C(5)	0·420	0·111	0·079	0·016	0·033	0·019
C(6)	0·517	0·099	0·182	0·016	0·043	0·022
C(7)	0·285	0·308	0·480	0·012	0·023	0·014
C(8)	0·308	0·457	0·509	0·021	0·029	0·023
C(9)	0·581	0·477	0·373	0·022	0·038	0·023
C(10)	0·782	0·269	0·663	0·011	0·023	0·014
C(11)	0·668	0·190	0·562	0·012	0·026	0·015
C(12)	0·582	0·198	0·608	0·016	0·040	0·028
C(13)	0·080	0·034	0·702	0·012	0·022	0·015
C(14)	0·162	0·105	0·663	0·010	0·025	0·013
C(15)	0·245	0·031	0·671	0·013	0·036	0·020
C(16)	0·259	0·467	0·996	0·012	0·028	0·017
C(17)	0·578	0·341	0·937	0·020	0·040	0·036
C(18)	0·624	0·484	0·924	0·019	0·036	0·030

Table 2. *Observed and calculated structure factors*

The unobserved reflections are marked by an asterisk.

H	K	L	FOBS	FCAL	H	K	L	FOBS	FCAL	H	K	L	FOBS	FCAL	H	K	L	FOBS	FCAL	H	K	L	FOBS	FCAL	H	K	L	FOBS	FCAL
0	1	0	1838	1877	0	1	6	932	942	-1	7	-1	864	854	* 1	4	4	81	71	-1	3	-7	919	960	-2	2	1	1365	1181
0	2	0	2243	2417	0	2	6	1756	1682	1	7	-1	1702	1757	-1	4	4	148	149	1	3	-7	919	922	-2	2	-1	2932	2645
0	3	0	1148	1155	0	2	-6	932	932	1	8	-1	1135	1107	-1	4	-4	445	544	1	4	-7	621	662	2	2	1	1486	1436
0	4	0	3446	3442	0	3	-6	905	969	1	8	1	891	879	1	5	-4	959	906	1	4	7	243	273	2	3	-1	3108	3185
0	5	0	324	468	0	4	6	581	506	-1	8	1	1486	1517	-1	5	4	1067	1027	-1	4	7	1013	981	-2	3	1	513	446
0	6	0	824	831	0	4	-6	189	170	-1	8	-1	351	306	-1	5	-4	1310	1295	-1	4	-7	324	359	-2	3	-1	378	251
0	7	0	1081	1128	0	5	-6	689	677	1	9	1	527	552	1	5	4	324	339	-1	5	7	446	432	2	3	1	1324	1341
0	8	0	1783	1796	0	5	6	864	840	-1	9	1	202	125	-1	6	-4	1067	1009	-1	5	-7	1675	1687	2	4	1	1473	1452
* 0	9	0	94	62	0	6	-6	1297	1260	-1	9	-1	405	426	1	6	-4	3081	3065	1	5	7	824	847	-2	4	1	3297	3420
0	0	1	2365	2489	0	6	6	243	168	1	9	-1	446	462	* 1	6	4	94	101	-1	6	-7	608	613	-2	4	-1	1892	1836
0	1	-1	391	345	0	7	-6	932	937	-1	10	-1	810	842	-1	6	4	797	825	1	6	-7	729	753	2	4	-1	1081	1189
0	1	1	1675	1547	0	8	-6	527	527	-1	0	2	891	727	1	7	4	621	600	-1	6	7	243	211	2	5	-1	175	132
0	2	1	1337	1384	0	9	-6	243	199	1	0	2	3013	2867	-1	7	4	243	198	1	7	-7	378	352	-2	5	1	2905	3010
0	2	-1	3121	3126	0	0	7	837	799	1	1	-2	2243	2174	-1	7	-4	1743	1714	-1	7	-7	229	270	-2	5	-1	1486	1416
0	3	1	351	275	0	-1	7	1432	1339	1	1	2	648	706	* 1	7	-4	81	51	-1	8	-7	783	757	2	5	1	297	241
0	3	-1	1283	1186	0	1	7	202	192	-1	1	2	310	394	1	8	-4	932	939	-1	9	-7	959	898	2	6	1	391	417
0	4	1	675	612	0	2	7	635	589	-1	1	-2	1838	1757	-1	8	4	135	120	1	9	-7	135	139	-2	6	1	1243	1224
0	4	-1	1648	1652	0	3	7	2162	2128	1	2	2	1351	1254	-1	8	-4	554	523	1	0	8	297	273	-2	6	-1	405	499
0	5	-1	297	315	0	3	-7	756	730	-1	2	2	1527	1441	-1	9	-4	459	453	-1	0	8	756	743	2	6	-1	716	727
0	5	1	837	848	0	4	7	689	670	-1	2	-2	2405	2172	1	9	-4	459	461	-1	-1	8	864	870	2	7	-1	1175	1322
0	6	-1	1027	1073	0	4	-7	1337	1322	1	2	-2	986	999	-1	10	-4	648	607	1	1	8	1094	999	-2	7	1	1162	1154
0	6	1	1121	1172	0	5	7	675	689	1	3	2	1581	1601	-1	0	5	2243	2195	1	-1	8	189	173	-2	7	-1	527	483
0	7	-1	324	359	0	5	-7	1459	1418	-1	3	2	2054	2012	1	0	5	1540	1590	1	2	8	756	747	2	7	1	783	766
0	7	1	216	109	0	6	-7	135	104	-1	3	-2	1202	1248	1	1	-5	189	220	1	-2	8	256	211	-2	8	-1	337	424
0	8	-1	1337	1415	* 0	7	-7	94	95	1	3	-2	1148	1196	1	1	5	1310	1314	-1	2	8	135	82	2	8	-1	243	264
0	9	1	932	983	0	8	-7	540	498	1	4	-2	202	233	1	-1	5	1162	1297	* 1	2	-8	81	88	-2	8	1	1851	1783
0	10	-1	337	343	0	9	-7	527	516	1	4	2	729	657	-1	1	5	1027	918	-1	3	8	1283	1291	-2	9	1	1432	1448
0	0	2	2716	2645	0	0	8	175	140	-1	4	2	1243	1137	1	2	5	1364	1337	-1	3	-8	756	769	-2	9	-1	824	819
0	1	-2	2878	2867	0	-1	8	500	479	-1	4	-2	527	513	-1	2	5	1838	1735	1	3	-8	243	284	2	9	-1	540	651
0	1	2	3297	3025	0	1	8	635	617	1	5	-2	1756	1753	-1	2	-5	1959	1956	1	4	-8	2027	2035	-2	10	1	148	90
0	2	2	1229	1140	0	3	8	837	835	-1	5	2	202	217	1	2	-5	3013	2901	1	4	8	243	234	-2	10	-1	1040	1045
0	2	-2	621	601	0	3	-8	473	499	-1	5	-2	1500	1423	1	3	5	486	409	-1	4	8	716	770	2	0	-2	1067	1067
0	3	2	1756	1762	0	4	-8	878	887	1	5	2	1202	1211	-1	3	5	946	861	-1	4	-8	297	320	2	0	2	891	906
0	3	-2	824	871	0	5	-8	1202	1253	-1	6	-2	337	372	-1	3	-5	702	741	1	5	-8	1351	1291	2	1	2	2757	2663
0	4	2	1513	1504	0	6	-8	135	137	1	6	-2	1013	1040	1	3	-5	986	946	-1	5	8	283	269	-2	1	2	2838	2637
0	4	-2	216	148	0	7	-8	189	146	1	6	2	621	558	1	4	-5	1540	1475	-1	5	-8	270	322	-2	1	-2	391	339
0	5	-2	2081	1966	0	8	-8	567	605	-1	6	2	797	770	1	4	5	121	89	1	6	-8	621	591	2	1	-2	1310	1212
0	5	2	297	282	0	0	9	135	139	1	7	2	621	596	-1	4	5	716	706	-1	6	-8	432	389	2	2	-2	1027	930
0	6	-2	1229	1211	0	-1	9	189	132	-1	7	2	283	304	-1	4	-5	500	409	-1	7	-8	459	439	-2	2	2	1959	1732
* 0	6	2	81	47	0	1	9	540	506	1	8	-2	486	533	1	5	-5	418	413	-1	8	-8	135	117	-2	2	-2	973	959
0	7	-2	1067	1050	0	2	9	810	876	-1	8	2	459	429	-1	5	5	283	210	1	8	-8	1027	992	2	2	2	391	478
0	7	2	459	493	0	-2	9	229	231	-1	8	-2	756	761	-1	5	-5	932	918	1	0	9	310	311	2	3	-2	2635	2644
0	8	2	1027	993	0	3	9	486	480	-1	9	2	1067	1038	1	5	5	648	685	-1	-1	9	297	259	-2	3	2	1594	1585
0	8	-2	405	357	0	3	-9	756	822	-1	9	-2	770	755	-1	6	-5	1594	1552	1	1	9	243	174	-2	3	-2	3013	2840
0	9	-2	567	559	0	4	-9	797	828	1	9	-2	445	432	1	6	-5	2878	2853	1	-1	9	459	488	2	3	2	1540	1534
0	9	2	364	385	0	5	-9	270	304	-1	10	-2	459	437	1	6	5	135	77	-1	1	9	554	553	2	4	2	1635	1606
0	10	-2	513	558	0	6	-9	432	471	1	10	-2	337	361	-1	6	5	432	420	1	2	9	810	881	-2	4	2	297	325
0	0	3	337	412	0	7	-9	391	433	-1	0	3	1283	1306	* 1	7	5	94	52	1	-2	9	310	260	-2	4	-2	189	215
0	1	-3	445	353	0	0	10	202	241	1	0	3	1081	1025	-1	7	-5	446	439	-1	2	9	540	542	2	4	-2	864	898
0	1	3	2311	2237	0	-1	10	675	755	1	1	-3	175	194	-1	7	-5	959	912	1	2	-9	378	334	2	5	-2	135	132
0	2	3	2986	2822	0	1	10	635	674	1	1	3	3284	3273	1	7	-5	473	489	* -1	3	9	94	126	-2	5	2	1513	1485
0	2	-3	2432	2448	* 0	-2	10	94	138	-1	1	3	351	367	-1	8	-5	864	852	-1	3	-9	135	93	-2	5	-2	1919	1967
0	3	3	743	719	0	3	-10	418	465	-1	1	-3	1121	1105	1	8	-5	1108	1039	1	3	-9	608	644	2	5	2	2797	2792
0	3	-3	1648	1561	0	4	-10	459	573	1	2	3	1297	1403	-1	9	-5	418	420	1	4	-9	445	441	2	6	2	256	249
0	4	3	554	531	0	5	-10	621	678	-1	2	3	1729	1543	1	9	-5	337	341	-1	4	-9	337	372	-2	6	2	1054	1078
0	4	-3	1013	962	1	0	0	4284	4544	-1	2	-3	3040	2948	-1	10	-5	189	231	-1	5	-9	635	685	-2	6	-2	986	984
0	5	-3	675	638	-1	1	0	3027	3383	1	2	-3	202	207	-1	0	6	418	400	1	5	-9	472	527	2	6	-2	405	383
0	5	3	1310	1305	1	1	0	1919	1925	1	3	3	1202	1156	-1	-1	6	513	512	1	6	-9	675	653	2	7	-2	1202	1280
0	6	-3	1527	1516	-1	2	0	1783	1903	-1	3	3	1027	1002	1	1	6	783	731	-1	6	-9	540	604	-2	7	2	797	747
0	6	3	662	696	1	2	0	2256	2097	-1	3	-3	2540	2489	1	-1	6	2040	1953	1	7	-9	337	344	-2	7	-2	1067	1101
0	7	-3	229	174	-1	3	0	810	732	1	3	-3	175	223	-1	1	6	148	123	-1	7	-9	243	276	2	7	2	445	378
0	7	3	135	152	1	3	0	2108	2091	1	4	-3	1635	1727	1	2	6	432	426	-1	0	10	378	423	2	8	-2	486	480
0	8	3	202	201	-1	4	0	3013	3254	1	4	3	486	505	-1	2	6	1729	1666	-1	-1	10	445	459	2	8	2	783	875
0	8	-3	783	778	1	4	0	810	739	-1	4	3	1743	1655	-1	2	-6	148	123	1	-1	10	486	498	-2	8	2	662	666
0	10	-3	432	385	1	5	0	905	869	-1	4	-3	973	934	1	2	-6	621	582	-1	1	10	135	88	2	9	-2	621	681
0	0	4	932	883	-1	5	0	2229	2272	1	5	-3	1473	1401	1	3	6	1770	1786	1	-2	10	364	417	-2	9	2	202	147
0	1	-4	378	353	-1	6	0	1662	1684	-1	5	3	635	550	-1	3	6	1729	1630	* -1	-2	10	94	49	-2	9	-2	581	526
0	1	4	2513	2429	1	6	0	581	574	-1	5	-3	1270	1228	-1	3	-6	1337	1431	1	3	-10	756	773	-2	10	-2	148	146
0	2	4	2689	2591	1	7	0	837	850	1	5	3	1865	1893	1	3	-6	1378	1325	-1	3	-10	959	1076	2	0	3	1297	1206
0	2	-4	2892	2826	-1	8	0	2135	2142	-1	6	-3	2473	2434	1	4	-6	1108	1091	1	4	-10	986	1079	-2	0	3	1621	1655
0	3	4	540	549	1	8	0	391	350	1	6	-3	702	699	* 1	4	6	94	74	-1	4	-10	648	719	2	1	3	1919	1910
0	3	-4	1756	1755	-1	9	0	919	929	1	6	3	216	236	-1	4	6	567	571	-1	5	-10	270	295	2	-1	3	378	384
0	4	4	324	243	1	9	0	675	742	-1	6	3	635	607	-1	4	-6	635	621	1	5	-10	337	335	-2	1	3	1162	969
0	4	-4	1919	1931	-1	10	0	216	165	1	7	3	189	165	1	5	-6	1000	979	1	6	-10	135	75	2	1	-3	2284	2073
0	5	-4	310	286	1	0	-1	5270	5750	* -1	7	3	94	142	-1	5	6	324	326	2	0	0	1121	1033	2	2	-3	1419	1336
0	5	4	986	986	1	0	1	2527	2486	-1	7	-3	2270	2222	-1	5	-6	1094	1070	-2	1	0	932	864	-2	2	3	648	612
0	6	-4	3689	3667	1	1	-1	1013	929	1	7	-3	1148	1152	1	5	6	1054	1059	2	1	0	2878	2903	-2	2	-3	986	984
0	6	4	229	252	1	1	1	3473	3324	1	8	-3	513	504	-1	6	-6	432	399	-2	2	0	932	988	2	2	3	324	437
0	7	-4	2000	1914	-1	1	1	2554	2602	1	8	3	554	570	1	6	-6	243	160	2	2	0	162	179	2	3	-3	567	518
0	7	4	135	191	-1	1	-1	2135	2020	-1	8	3	702	637	1	6	6	581	651	2	3	0	864	807	-2	3	3	2689	2522
* 0	8	4	94	67	1	2	1	729	716	-1	8	-3	283	341	-1	6	6	419	517	-2	3	0	2135	2181	-2	3	-3	2216	2230
0	8	-4	946	936	-1	2	1	1716	1584	* 1	9	-3	94	87	-1	7	6	243	262	-2	4	0	689	661	2	3	3	1527	1536
0	10	-4	1000	999	-1	2	-1	3162	3036	-1	9	-3	135	123	-1	7	-6	675	642	2	4	0	1851	1786	2	4	3	864	808
0	0	5	1797	1652	1	2	-1	4351	4232	-1	10	-3	932	880	1	7	-6	716	768	-2	5	0	1405	1445	-2	4	3	2392	2342
0	-1	5	1473	1472	1	3	1	270	262	1	10	-3	283	272	* -1	8	-6	94	81	2	5	0	1621	1624	-2	4	-3	540	568
0	1	5	94	91	-1	3	-1	932	865	-1	0	4	2324	2194	1	8	-6	716	657	-2	6	0	567	619	2	4	-3	1500	1529
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0	2	-5	229	137	1	4	-1	2608	2630	1	1	-4	621	603	1	9	-6	135	234	-2	7	0	1162	1255	-2	5	3	216	235
0	3	5	2135	2070	1	4	1	1202	1084	1	1	4	1013	1056	1	0	7	500	455	2	7	0	243	271	-2	5	-3	1378	1318
0	3	-5	162	161	-1	4	1	3189	3348	1	-1	4	837	827	-1	0	7	1000	1002	-2	8	0	783	822	2	5	3	1108	1157
0	4	5	391	395	-1	4	-1	324	338	-1	1	4	1689	1564	-1	-1	7	513	517	* 2	8	0	94	225	2	6	3	135	147
0	4	-5	729	721	1	5	-1	337	330	1	2	4	446	389	1	1	7	175	178	-2	9	0	675	690	-2	6	3	162	81
0	5	-5	1270	1253	-1	5	1	1459	1458	-1	2	4	4689	4512	1	-1	7	1716	1691	2	9	0	540	500	-2	6	-3	1081	1100
0	6	-5	391	417	-1	5	-1	378	411	-1	2	-4	121	155	-1	1	7	445	407	-2	10	0	432	452	2	6	-3	1256	1289
0	6	5	189	164	1	5	1	1175	1139	1	2	-4	3716	3662	1	2	7	1283	1296	2	0	-1	3189	3118	2	7	-3	567	583
0	7	-5	175	123	-1	6	-1	1108	1078	1	3	4	1527	1481	-1	2	7	919	873	2	0	1	1283	1253	* -2	7	3	94	66
0	7	5	337	406	1	6	-1	418	417	-1	3	4	851	808	-1	2	-7	973	1034	-2	1	1	3513	3597	-2	7	-3	2027	2015
0	8	-5	959	910	1	6	1	919	959	-1	3	-4	1202	1194	1	2	-7	905	919	-2	1	-1	2108	2244	2	7	3	486	529
0	9	-5	243	222	-1	6	1	1432	1603	1	3	-4	864	716	1	3	7	1689	1717	2	1	-1	1662	1651	* -2	8	-3	94	91
0	0	6	432	411	1	7	1	540	591	1	4	-4	1513	1482	-1	3	7	783	683	2	2	-1	148	276	2	8	-3	270	214

Table 2 (*cont.*)

H	K	L	FOBS	FCAL		H	K	L	FOBS	FCAL		H	K	L	FOBS	FCAL		H	K	L	FOBS	FCAL		H	K	L	FOBS	FCAL
2	8	3	135	157		-2	8	-6	364	363		3	6	0	1297	1216		-3	4	3	1716	1685		-3	3	-6	270	338		4	1	0	229	190
-2	8	3	756	722		2	8	-6	689	649		-3	7	0	905	892		3	5	-3	1040	1035		-3	4	-6	1324	1316		-4	2	0	1594	1591
-2	9	3	594	584		2	9	-6	608	585		3	7	0	324	288		3	5	3	324	405		3	4	-6	216	127		4	2	0	1243	1312
-2	9	-3	135	148		-2	9	-6	1094	1074		-3	8	0	243	247		-3	5	3	243	174		3	4	6	581	653		-4	3	0	919	890
2	9	-3	243	279		-2	0	7	297	259	*	3	8	0	94	63		-3	5	-3	1243	1261		-3	4	6	702	647		4	3	0	1338	1158
-2	10	-3	891	821		2	0	7	662	587		-3	9	0	256	169		-3	6	-3	459	469		3	5	-6	1365	1325		-4	4	0	1081	1119
-2	0	4	2838	2719		2	1	7	621	697	*	-3	10	0	94	680		3	6	-3	162	92		-3	5	6	472	439		4	4	0	1013	989
2	0	4	1000	1498		2	-1	7	783	778		3	0	1	1121	1194		3	6	3	689	691		-3	5	-6	864	867		-4	5	0	2378	2406
2	1	4	94	91		-2	1	7	229	191		3	0	-1	500	433		-3	6	3	783	777		-3	6	-6	1446	1419		4	5	0	229	161
2	-1	4	1581	1644		-2	-1	7	189	123		3	1	1	1716	1890		-3	7	-3	797	775		3	6	-6	919	912		-4	6	0	675	677
-2	1	4	1027	918		2	2	-7	1567	1510		-3	1	1	2500	2387		3	7	-3	689	699		-3	6	6	1108	1038		4	6	0	1351	1287
2	1	-4	1878	1902		2	-2	7	1121	1115		-3	1	-1	1716	1623		3	7	3	459	455		-3	7	6	581	556		-4	7	0	459	507
2	2	-4	756	758		-2	2	7	1229	1218		3	1	-1	1797	1747		-3	7	3	797	706		-3	7	-6	135	81	*	4	7	0	94	76
-2	2	4	2689	2493		2	2	7	270	257		3	2	1	297	340		-3	8	-3	270	232		3	7	-6	500	481		-4	8	0	135	155
-2	2	-4	1581	1570		2	3	-7	1838	1770		-3	2	1	1135	1032		3	8	-3	135	86		-3	8	-6	135	122		4	8	0	662	676
2	2	4	229	225		-2	3	7	770	703		-3	2	-1	418	344		-3	8	3	135	237		3	9	-6	1162	1134		-4	9	0	527	549
2	3	-4	1256	1180		-2	3	-7	797	796		3	2	-1	702	667		3	9	-3	608	607		-3	9	-6	1013	970		-4	10	0	581	601
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2	3	4	716	689		-2	4	7	189	131		-3	3	1	1162	1203		3	0	4	1824	1773		3	1	7	513	413		4	1	-1	1932	1928
2	4	4	797	824		-2	4	-7	527	573		-3	3	-1	2527	2518		-3	0	4	148	157		3	-1	7	783	763		4	-1	1	283	517
-2	4	4	797	797		2	4	-7	567	553		3	4	-1	797	763		3	1	4	202	269		-3	1	7	797	791		-4	1	1	189	220
-2	4	-4	527	480		2	5	-7	419	372		3	4	1	1702	1852		3	-1	4	1270	1178	*	3	1	-7	81	80		4	1	1	1905	1990
2	4	-4	1540	1534		-2	5	7	459	412		-3	4	1	324	335		-3	1	4	1581	1653		3	2	7	432	503		4	2	1	1567	1660
2	5	-4	878	906		-2	5	-7	878	960		3	5	-1	851	898		3	1	-4	202	141		3	-2	7	432	438		-4	2	1	310	384
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2	5	-4	1000	1009		-2	6	7	567	638		-3	5	1	2635	2715		-3	2	4	175	114		3	2	-7	1054	1020		4	2	-1	662	621
2	5	4	554	553		-2	6	-7	500	501		-3	5	-1	1932	1893		-3	2	-4	756	669		3	3	-7	2067	1991		-4	3	-1	3351	3294
2	6	4	337	395		2	7	-7	554	476		-3	6	-1	108	78		3	2	-4	2189	2133		3	3	7	283	243		4	3	-1	1310	1262
-2	6	4	1256	1252	*	-2	7	-7	94	91		3	6	-1	175	168		3	3	-4	2067	2013		-3	3	7	581	547		4	3	1	67	54
-2	6	-4	1013	981		-2	8	-7	243	240		3	6	1	567	605		3	3	4	567	551		-3	3	-7	905	1011		-4	3	1	1500	1603
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2	7	4	418	445		-2	0	8	1270	1234		-3	7	-1	621	639		-3	3	-4	1702	1709		3	4	-7	608	590		4	4	1	3486	3668
2	7	-4	1189	1207		2	0	8	378	291		3	7	-1	391	391		-3	4	-4	567	540		-3	4	7	554	533		-4	4	1	2365	2501
-2	7	4	540	548		2	1	8	946	847		3	7	1	419	422		3	4	-4	1121	1128		3	5	-7	216	216		-4	4	-1	3405	3349
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-2	2	-5	283	247		2	4	-8	1594	1570		3	-1	2	716	640	*	3	8	-4	94	94		-3	-1	8	486	490		-4	8	1	310	336
2	2	5	635	640		-2	5	8	135	126		-3	1	2	1865	1880		-3	8	4	689	692		3	-2	8	878	844		-4	8	-1	675	630
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2	4	5	364	339		2	7	-8	432	359		3	2	-2	1500	1603		-3	10	-4	770	754		-3	3	-8	405	442		4	0	2	1810	1748
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-2	4	-5	148	130		-2	8	-8	567	585		3	3	2	1135	1075		-3	0	5	1243	1121		-3	4	8	824	832		4	1	-2	527	586
2	4	-5	918	926		2	8	-8	635	616		-3	3	2	135	209		3	1	5	229	160		-3	4	-8	243	235		4	-1	2	621	422
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2	9	-5	905	930		2	5	-9	581	579		3	7	-2	1419	1514		3	5	5	310	267		3	-2	9	459	418		4	5	-2	229	210
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2	1	-6	716	661		-2	1	10	540	520		-3	8	2	878	895		3	6	5	1189	1270		3	4	-9	1783	1794		-4	6	-2	256	306
2	2	-6	837	800		-2	-1	10	135	98		-3	9	2	932	958		-3	6	5	1621	1539		3	5	-9	270	278		4	6	-2	891	960
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2	2	6	1635	1536		2	3	-10	824	861		-3	10	-2	608	564		-3	7	-5	540	524		3	7	-9	959	874		4	7	-2	621	676
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-2	3	-6	1013	1017		-2	-1	11	445	449		3	1	3	878	757		3	8	-5	202	148		-3	1	10	135	68		-4	8	-2	554	507
2	3	6	1527	1515		-2	-2	11	243	246		3	-1	3	1202	1300		3	9	-5	810	768		-3	2	10	689	694		-4	9	2	1121	1123
2	4	6	851	818		2	3	-11	283	256		-3	1	3	946	964		-3	9	-5	689	722		3	2	-10	581	553	*	-4	10	-2	94	55
-2	4	6	1202	1116		3	0	0	1486	1438		3	1	-3	1973	1987		3	0	6	1365	1373		3	3	-10	270	290		4	0	3	1324	1325
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2	4	-6	1175	1119		3	1	0	675	825		-3	2	3	148	153		3	-1	6	1054	1026		3	4	-10	1027	1044		4	1	-3	621	648
2	5	-6	500	533		-3	2	0	243	222		-3	2	-3	1189	1159		-3	1	6	1635	1549		3	5	-10	202	189		4	-1	3	1554	1528
-2	5	6	324	277		3	2	0	121	289		3	2	-3	702	783		3	1	-6	2635	2638		3	6	-10	919	932		-4	1	3	1229	1384
-2	5	-6	1756	1727		-3	3	0	1973	1967		3	3	-3	1256	1227		3	2	6	1175	1184		-3	0	11	500	575		4	1	3	364	289
2	5	6	527	535		3	3	0	1554	1481		3	3	3	1297	1267		3	-2	6	3027	2980		-3	-1	11	513	577		4	2	3	337	377
-2	6	-6	932	871		-3	4	0	2473	2531		-3	3	3	2702	2577		-3	2	6	1797	1769		3	2-11		418	394		-4	2	3	1189	1152
2	6	-6	986	972		3	4	0	1581	1578		-3	3	-3	310	285		3	2	-6	2311	2276		3	3-11		500	466		-4	2	-3	1635	1605
-2	7	6	527	524		-3	5	0	1135	1100		-3	4	-3	1189	1215		3	3	-6	175	98		3	4-11		310	342		4	2	-3	2986	2755
-2	7	-6	229	216		3	5	0	986	832		3	4	-3	337	418		3	3	6	270	351		4	0	0	783	783		-4	3	-3	1446	1405
2	7	-6	513	490		-3	6	0	229	219		3	4	3	486	442		-3	3	6	1486	1443		-4	1	0	1175	1202		4	3	-3	3121	3082

Table 2 (*cont.*)

H	K	L	FOBS	FCAL	H	K	L	FOBS	FCAL	H	K	L	FOBS	FCAL	H	K	L	FOBS	FCAL	H	K	L	FOBS	FCAL	H	K	L	FOBS	FCAL
4	3	3	1337	1287	4	2	-6	1675	1646	5	4	0	2784	2633	-5	5	3	2202	2229	-5	7	6	1797	1746	-6	2	1	1986	2049
-4	3	3	837	906	-4	3	-6	216	113	-5	5	0	486	541	5	6	-3	1662	1741	* -5	7	-6	94	69	-6	2	-1	1594	1552
4	4	-3	229	216	4	3	-6	175	245	5	5	0	243	170	5	6	3	540	533	5	7	-6	648	651	6	2	-1	932	954
4	4	3	1270	1287	4	3	6	432	503	-5	6	0	851	907	-5	6	3	1648	1658	-5	8	6	662	650	6	3	1	689	537
-4	4	3	405	423	-4	3	6	2419	2323	5	6	0	1013	1012	-5	6	-3	662	579	-5	8	-6	324	350	-6	3	1	567	651
-4	4	-3	770	775	4	4	-6	1108	1086	-5	7	0	608	598	5	7	-3	608	648	5	8	-6	135	144	-6	3	-1	1135	1079
4	5	-3	513	548	4	4	6	351	471	5	7	0	621	550	-5	7	3	297	241	5	0	7	594	554	5	3	-1	1067	1007
4	5	3	783	868	-4	4	6	175	172	-5	8	0	918	947	-5	7	-3	135	127	-5	0	7	1297	1274	6	4	-1	1175	1156
-4	5	3	2094	2063	-4	4	-6	1135	1226	-5	9	0	378	332	5	8	-3	473	463	-5	1	7	364	286	6	4	1	148	80
-4	5	-3	1770	1757	4	5	-6	986	961	-5	10	0	554	605	-5	8	3	472	428	5	1	-7	2108	2064	-6	4	1	878	983
4	6	3	202	205	-4	5	6	905	851	5	0	-1	729	699	-5	8	-3	594	552	5	1	7	500	450	-6	4	-1	2175	2083
-4	6	3	243	262	4	6	-6	310	281	5	0	1	3432	3720	-5	9	-3	148	175	5	-1	7	486	445	6	5	-1	473	415
-4	6	-3	851	810	-4	6	6	1148	1089	-5	1	1	324	337	-5	9	3	959	933	5	2	-7	108	77	6	5	1	1094	905
4	6	-3	1297	1359	-4	6	-6	783	757	5	1	-1	3013	2873	-5	10	-3	256	247	5	-2	7	418	403	-6	5	1	621	589
-4	7	3	770	745	4	7	-6	378	379	5	1	1	216	595	5	0	4	1148	1107	-5	2	7	648	607	-6	5	-1	1148	1139
-4	7	-3	256	210	-4	7	6	1986	1913	5	-1	1	567	630	5	0	-4	878	987	-5	3	-7	567	613	6	6	1	310	237
4	7	-3	1175	1254	4	8	-6	689	672	5	2	-1	2446	2408	-5	1	4	1094	1198	5	3	-7	310	231	-6	6	-1	878	823
* -4	8	-3	94	56	-4	8	-6	783	741	-5	2	1	1135	1135	5	1	-4	1432	1534	-5	3	7	1459	1390	6	6	-1	513	494
4	8	-3	378	379	4	0	7	270	224	-5	2	-1	2716	2709	5	1	4	1554	1517	5	4	-7	1027	989	-6	6	1	1446	1528
* -4	8	3	94	81	-4	0	7	283	276	5	2	1	946	1166	5	-1	4	337	333	-5	4	7	405	331	6	7	-1	892	850
4	9	-3	432	406	4	1	-7	1459	1452	-5	3	-1	1027	960	5	2	-4	1175	1249	-5	5	7	1000	877	-6	7	1	743	810
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-4	9	-3	297	279	-4	1	7	1310	1315	5	3	1	851	940	-5	2	4	378	331	5	5	-7	743	762	-6	8	-1	1067	1063
-4	10	-3	770	741	4	1	7	243	234	-5	3	1	364	300	5	2	4	1324	1340	-5	6	7	135	162	* -6	8	1	94	105
4	0	4	1338	1352	4	2	7	662	610	5	4	1	905	2394	-5	3	-4	1675	1751	-5	6	-7	324	315	-6	9	1	689	656
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4	1	-4	3027	2970	-4	2	7	324	266	-5	4	-1	4040	3952	5	3	4	364	354	-5	7	7	1216	1268	-6	10	-1	635	588
4	-1	4	513	501	4	2	-7	162	139	-5	5	-1	1513	1461	-5	3	4	1351	1356	-5	7	-7	500	482	-6	10	1	675	700
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4	1	4	810	701	4	3	-7	1081	1118	5	5	1	81	256	-5	4	4	1067	1127	-5	0	8	783	764	6	0	-2	2270	2269
4	2	4	1365	1317	-4	3	7	513	491	-5	5	1	2121	2161	-5	4	-4	310	386	-5	1	8	1094	1107	-6	1	2	243	371
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-4	3	-4	364	392	-4	5	7	959	894	-5	6	-1	297	332	5	5	4	986	1056	-5	2	8	729	692	6	2	-2	851	938
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4	4	-4	851	798	-4	7	7	135	79	5	7	-1	256	205	-5	6	-4	1783	1769	-5	3	8	189	191	6	3	-2	256	249
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4	5	-4	986	997	-4	8	-7	391	428	-5	9	-1	337	263	-5	8	-4	1216	1107	5	5	-8	459	446	-6	4	-2	581	520
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4	6	4	892	937	4	-1	8	608	749	5	0	-2	1554	1484	-5	9	-4	202	168	-5	6	8	581	580	-6	5	-2	1851	1808
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-4	7	4	648	667	* 4	-2	8	94	147	-5	1	2	1121	1033	-5	1	5	1202	1247	5	1	-9	797	772	-6	5	2	459	387
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-4	10	-4	243	146	* 4	5	-8	94	147	5	3	2	919	841	-5	3	-5	1135	1204	5	6	-9	202	163	-6	8	-2	297	334
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4	1	-5	1013	945	4	6	-8	135	131	5	4	2	729	627	-5	3	5	391	398	-5	-1	10	865	902	-6	9	-2	378	384
4	-1	5	554	625	-4	6	8	770	826	-5	4	2	1405	1434	5	4	-5	270	195	-5	1	10	2000	1955	-6	10	-2	594	590
-4	1	5	1838	2001	-4	6	-8	391	442	-5	4	-2	337	210	-5	4	5	162	139	5	2	-10	310	284	6	0	3	500	421
4	1	5	594	629	4	7	-8	527	519	-5	5	-2	1527	1500	-5	4	-5	648	704	-5	2	10	135	140	6	0	-3	1446	1454
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-4	2	5	135	121	-4	-1	9	608	611	-5	5	2	1162	1155	-5	5	5	473	479	* 5	4	-10	94	173	6	1	3	1905	1875
4	2	-5	2338	2206	-4	1	9	1189	1166	5	6	-2	189	128	-5	6	-5	1351	1345	5	5	-10	540	578	6	-1	3	810	776
-4	3	-5	973	1045	4	2	-9	297	281	5	6	2	229	184	5	6	-5	189	139	-5	0	11	527	558	6	2	-3	2338	2339
4	3	-5	1527	1615	-4	2	9	608	598	-5	6	2	1027	1073	-5	6	5	1054	1031	-5	-1	11	810	812	6	2	3	702	741
4	3	5	270	229	-4	3	9	446	453	-5	6	-2	1540	1513	5	7	-5	892	904	-5	1	11	946	988	6	-2	3	567	671
-4	3	5	1364	1366	4	3	-9	918	860	-5	7	2	513	503	-5	7	5	310	337	5	2	-11	202	138	-6	2	3	2013	2100
4	4	-5	472	442	4	4	-9	648	635	* -5	7	-2	81	141	-5	7	-5	959	954	5	3	-11	797	754	6	3	-3	283	263
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-4	4	-5	1351	1384	4	6	-9	243	191	-5	8	-2	405	422	-5	8	-5	1067	985	6	1	0	783	704	-6	3	-3	1202	1235
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-4	5	5	1229	1246	-4	-1	10	500	463	-5	9	2	513	542	-5	0	6	1337	1337	-6	2	0	2676	2503	6	4	3	148	213
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-4	6	5	135	122	4	2	-10	243	223	5	0	3	946	1022	5	1	-6	2000	1969	-6	3	0	1067	987	-6	5	-3	1081	1113
-4	6	-5	1892	1926	-4	2	10	581	556	5	0	-3	608	472	* 5	1	6	94	111	6	3	0	1716	1594	6	5	-3	486	538
4	6	-5	378	316	-4	3	10	446	401	-5	1	3	1702	1714	5	-1	6	959	921	-6	4	0	3297	3348	6	5	3	824	866
-4	7	-5	364	335	4	3	-10	1000	1011	5	1	-3	946	939	5	2	6	648	706	6	4	0	1689	1531	-6	5	3	635	652
4	7	-5	756	835	4	4	-10	891	909	5	1	3	689	667	5	2	-6	783	825	-6	5	0	1729	1723	6	6	-3	770	775
-4	7	5	1000	999	4	5	-10	621	619	5	-1	3	1297	1247	5	-2	6	1027	982	6	5	0	418	302	-6	6	3	1581	1543
-4	8	5	243	192	4	6	-10	283	295	5	2	-3	4284	4231	-5	2	6	189	175	-6	6	0	594	598	-6	6	-3	554	518
-4	8	-5	864	904	* -4	0	11	94	55	-5	2	3	1635	1736	-5	3	-6	621	685	6	6	0	432	372	6	7	-3	351	375
4	8	-5	135	100	-4	-1	11	675	692	-5	2	-3	243	234	5	3	-6	513	534	-6	7	0	189	130	-6	7	3	432	451
4	9	-5	202	125	4	2	-11	445	499	5	2	3	216	220	5	3	6	513	541	6	7	0	1067	1046	-6	7	-3	1040	1053
-4	9	-5	581	550	4	3	-11	202	160	-5	3	-3	148	179	-5	3	6	1648	1655	-6	8	0	1756	1827	6	8	-3	459	481
4	0	6	756	759	4	4	-11	135	171	5	3	-3	1946	1987	5	4	-6	919	922	-6	9	0	797	781	-6	8	3	324	260
-4	0	6	892	894	5	0	0	3527	3446	5	3	3	1162	1058	-5	4	6	851	828	-6	10	0	702	710	-6	8	-3	581	593
4	1	-6	3175	3569	5	1	0	702	698	-5	3	3	94	91	-5	4	-6	770	792	6	0	-1	986	1069	-6	9	3	702	746
4	-1	6	783	712	5	-1	0	81	77	5	4	-3	1932	1872	5	5	-6	432	477	6	0	1	378	390	-6	9	-3	445	427
-4	1	6	1513	1439	-5	2	0	3148	3012	5	4	3	662	592	-5	5	6	743	685	6	1	1	1243	1281	6	0	4	189	208
4	1	6	351	310	5	2	0	1473	1419	-5	4	3	175	204	-5	5	-6	513	604	6	-1	1	1432	1531	6	0	-4	689	722
4	2	6	135	90	-5	3	0	783	752	-5	4	-3	1067	1159	5	6	-6	418	449	6	1	-1	1446	1333	-6	1	4	756	842
4	-2	6	810	727	5	3	0	810	733	-5	5	-3	2081	2076	* -5	6	6	94	78	6	-1	-1	1337	1263	6	1	-4	878	938
-4	2	6	1365	1371	-5	4	0	2527	2556	5	5	-3	337	318	-5	6	-6	351	391	6	2	1	891	734	6	1	4	1567	1461

Table 2 (*cont.*)

H	K	L	FOBS	FCAL	H	K	L	FOBS	FCAL	H	K	L	FOBS	FCAL	H	K	L	FOBS	FCAL	H	K	L	FOBS	FCAL	H	K	L	FOBS	FCAL
6	-1	4	702	700	-6	6	7	310	303	-7	3	-2	1000	1023	7	5	-5	1216	1202	8	1	-1	243	297	-8	5	-4	608	586
6	2	-4	3094	3157	-6	6	-7	202	249	7	3	-2	648	630	-7	6	5	810	889	8	-1	-1	3000	2885	8	5	-4	689	652
6	2	4	540	494	* 6	7	-7	94	135	7	3	2	648	819	-7	6	-5	1081	1204	8	-1	1	1905	1935	-8	6	4	1027	1045
6	-2	4	1189	1124	-6	7	7	1351	1325	-7	3	2	1216	1202	7	6	-5	1297	1359	8	2	-1	973	887	-8	6	-4	932	970
-6	2	4	2878	3020	-6	0	8	662	691	7	4	2	202	216	-7	7	5	351	334	-8	2	1	1121	1163	8	6	-4	351	361
6	3	-4	675	690	6	1	-8	1148	1153	-7	4	2	459	372	-7	7	-5	567	658	8	2	1	513	439	-8	7	-4	216	181
* 6	3	4	94	92	-6	1	8	121	138	-7	4	-2	121	131	-7	8	-5	148	82	8	-2	1	283	353	-8	7	4	864	824
-6	3	4	175	240	* -6	2	8	94	68	7	4	-2	189	113	-7	8	5	540	537	-8	3	1	756	761	-8	8	4	297	209
-6	3	-4	1216	1318	6	2	-8	648	681	-7	5	2	121	126	7	0	-6	973	904	-8	3	-1	175	222	* -8	8	-4	94	60
-6	4	-4	310	273	-6	3	8	446	440	-7	5	-2	1432	1342	7	0	6	297	251	8	3	-1	675	661	8	0	5	675	723
6	4	-4	2135	2132	6	4	-8	1202	1228	7	5	-2	729	733	7	-1	6	851	802	8	3	1	973	830	8	0	-5	297	324
6	4	4	500	406	-6	4	8	527	479	7	5	2	418	583	-7	1	6	1594	1664	8	4	1	446	334	8	-1	5	148	90
-6	4	4	1283	1314	-6	5	8	527	483	-7	6	-2	554	506	7	1	-6	540	515	* 8	4	-1	94	118	-8	1	5	851	929
-6	5	-4	189	185	6	5	-8	716	726	-7	6	2	364	359	7	2	-6	459	421	-8	4	-1	567	536	8	1	-5	2081	2098
6	5	-4	648	693	-6	6	8	243	243	7	6	-2	810	732	7	-2	6	527	560	-8	4	1	1162	1202	8	2	-5	1405	1446
-6	5	4	729	752	6	6	-8	283	274	7	7	-2	297	267	-7	2	6	1310	1296	8	5	-1	297	241	-8	2	5	2676	2905
-6	6	4	1162	1206	6	7	-8	243	135	-7	7	2	1000	990	7	-3	6	702	656	-8	5	1	2013	2019	8	-2	5	621	598
-6	6	-4	1756	1788	-6	0	9	1094	980	-7	7	-2	283	235	7	3	-6	851	859	-8	5	-1	1189	1116	8	3	-5	608	558
6	6	-4	2202	2262	6	1	-9	189	214	-7	8	-2	540	570	-7	3	6	1392	1410	-8	6	1	270	256	8	-3	5	297	311
-6	7	-4	1986	1978	-6	1	9	878	841	-7	8	2	824	812	7	4	-6	892	921	-8	6	-1	405	405	-8	3	5	3162	3252
6	7	-4	594	653	-6	2	9	946	901	-7	9	2	675	660	-7	4	6	635	597	8	6	-1	946	800	8	4	-5	189	193
-6	7	4	243	185	6	2	-9	135	99	-7	9	-2	635	648	-7	4	-6	364	399	-8	7	-1	202	219	-8	4	5	783	773
-6	8	-4	513	492	6	3	-9	851	835	7	0	-3	1405	1518	7	5	-6	135	178	-8	7	1	946	969	-8	4	-5	554	622
6	8	-4	148	170	-6	3	9	621	599	7	0	3	1324	1199	-7	5	6	297	256	-8	8	1	1040	1047	-8	5	5	824	788
* -6	8	4	94	124	6	4	-9	500	491	-7	1	3	919	958	-7	5	-6	837	884	-8	8	-1	986	918	-8	5	-5	391	391
-6	9	4	418	378	-6	4	9	135	107	7	-1	3	364	305	-7	6	6	554	551	-8	9	-1	743	714	8	5	-5	1594	1614
-6	9	-4	608	655	-6	5	9	135	177	7	1	3	2243	2031	7	6	-6	351	333	-8	9	1	2013	1985	8	6	-5	891	865
* 6	0	5	94	77	6	5	-9	310	257	7	1	-3	851	786	7	7	-6	878	912	8	0	-2	378	333	-8	6	-5	256	215
6	0	-5	1635	1617	6	6	-9	540	548	7	2	3	216	158	-7	7	6	689	676	8	0	2	1324	1358	-8	6	5	1162	1216
-6	1	5	378	315	-6	0	10	635	597	7	2	-3	297	312	-7	7	-6	824	821	8	1	2	1702	1823	-8	7	5	891	896
6	1	-5	1216	1245	6	1	-10	608	580	7	-2	3	581	634	7	0	-7	824	804	8	-1	2	337	493	-8	7	-5	797	866
6	-1	5	810	769	-6	1	10	689	715	-7	2	3	310	299	7	1	-7	270	245	8	1	-2	1256	1238	-8	8	5	148	206
6	2	-5	108	135	-6	2	10	310	287	-7	3	3	189	115	-7	1	7	459	523	8	-1	-2	2027	1906	8	0	-6	337	382
6	2	5	729	816	6	2	-10	621	622	7	3	3	378	306	7	2	-7	1135	1118	-8	2	2	108	152	8	1	-6	770	770
6	-2	5	445	443	6	3	-10	283	304	-7	3	-3	1054	1125	-7	2	7	405	451	8	2	2	364	493	-8	1	6	1202	1194
6	3	5	635	655	-6	3	10	486	490	7	3	-3	702	786	7	3	-7	175	193	* 8	-2	2	81	67	-8	2	6	216	140
-6	3	5	1351	1344	6	4	-10	621	637	7	4	3	648	664	* 7	-3	7	94	276	8	2	-2	621	586	8	2	-6	283	304
* -6	3	-5	94	295	6	5	-10	581	561	-7	4	3	1554	1666	-7	3	7	175	114	8	3	2	378	543	8	-2	6	1270	1333
6	3	-5	364	303	-6	0	11	202	167	-7	4	-3	459	433	-7	4	7	973	1013	-8	3	2	1027	1161	* 8	-3	6	94	68
* -6	4	-5	94	361	-6	-1	11	594	602	7	4	-3	675	772	7	4	-7	513	490	-8	3	-2	1310	1270	-8	3	6	175	127
6	4	-5	1243	1309	-6	1	11	1419	1484	-7	5	3	418	398	-7	5	7	202	138	8	3	-2	919	806	8	3	-6	892	921
-6	4	5	689	741	6	2	-11	554	527	-7	5	-3	472	405	7	5	-7	135	138	8	4	2	1000	1105	8	4	-6	486	485
6	5	-5	135	177	6	3	-11	919	920	7	5	-3	391	290	7	6	-7	594	670	* -8	4	2	81	56	-8	4	6	1297	1283
-6	5	5	581	609	7	0	0	1324	1239	-7	6	3	851	877	-7	6	7	743	702	-8	4	-2	824	820	-8	4	-6	689	733
-6	5	-5	694	793	7	1	0	783	755	-7	6	-3	959	912	7	7	-7	243	254	8	4	-2	243	188	-8	5	-6	892	914
6	6	-5	729	745	7	-1	0	2851	2656	7	6	-3	797	863	-7	7	7	378	345	-8	5	2	756	796	8	5	-6	729	723
-6	6	5	486	453	-7	2	0	662	622	-7	7	3	243	259	-7	0	8	1432	1483	-8	5	-2	608	562	-8	5	6	189	215
-6	6	-5	432	491	7	2	0	283	259	-7	7	-3	1811	1702	7	1	-8	405	344	8	5	-2	513	464	8	6	-6	243	300
6	7	-5	513	517	-7	3	0	1851	1812	7	7	-3	810	915	-7	1	8	851	872	8	6	-2	567	528	-8	7	6	297	268
-6	7	5	1108	1069	7	3	0	892	853	-7	8	-3	216	135	-7	2	8	432	397	-8	6	2	135	105	8	0	-7	283	317
-6	7	-5	554	557	-7	4	0	1594	1546	-7	8	3	662	644	7	2	-8	1148	1105	-8	7	-2	1337	1267	8	1	-7	121	216
6	8	-5	635	643	7	5	0	743	718	-7	9	3	148	99	-7	3	8	702	728	-8	7	2	675	776	-8	1	7	986	1047
-6	8	5	702	670	-7	5	0	2000	2028	-7	9	-3	459	512	7	3	-8	689	669	-8	8	-2	270	283	-8	2	7	1067	1145
* -6	8	-5	94	308	-7	6	0	297	270	7	0	-4	729	705	-7	4	8	1216	1269	-8	9	-2	513	449	8	2	-7	824	851
6	0	6	243	189	* 7	6	0	94	104	7	0	4	243	209	7	4	-8	1986	2006	-8	9	2	256	265	8	3	-7	770	728
6	0	-6	189	168	-7	7	0	716	703	-7	1	4	364	224	* -7	5	8	94	68	8	0	3	1419	1356	-8	3	7	1094	1060
-6	1	6	1229	1273	-7	8	0	1405	1420	7	1	-4	2000	2013	7	5	-8	446	480	8	0	-3	540	497	8	4	-7	662	661
6	1	-6	189	130	-7	9	0	1743	1747	7	1	4	324	349	-7	6	8	283	306	8	0	-3	527	497	-8	4	7	243	210
6	1	6	621	617	7	0	1	2000	2105	7	-1	4	878	888	7	6	-8	378	340	8	-1	3	202	234	8	5	-7	608	589
6	-1	6	202	209	7	0	-1	2148	2121	7	2	4	581	625	* -7	0	9	94	187	8	1	3	1081	1003	* -8	5	7	94	76
6	2	-6	1500	1517	7	-1	1	513	492	7	-2	4	243	280	7	1	-9	459	367	8	-1	-3	1162	1159	-8	6	7	743	721
6	2	6	378	374	7	1	1	1567	1378	7	2	-4	2500	2566	-7	1	9	472	485	8	1	-3	1351	1315	8	6	-7	432	447
6	-2	6	621	508	7	-1	-1	3419	3334	-7	2	4	3446	3523	-7	2	9	648	630	8	2	3	729	645	-8	7	7	202	216
-6	2	6	1310	1336	7	1	-1	189	146	7	3	4	378	467	7	2	-9	797	773	-8	2	3	1486	1527	8	0	-8	1067	1115
6	3	-6	743	718	-7	2	-1	256	355	-7	3	4	2392	2405	7	3	-9	594	564	8	2	-3	594	547	-8	1	8	810	800
-6	3	6	202	156	7	2	-1	473	443	7	3	-4	418	378	* -7	3	9	94	87	8	3	-3	297	341	-8	2	8	229	257
-6	3	-6	810	933	7	2	1	540	465	-7	3	-4	391	408	* -7	4	9	94	97	-8	3	-3	608	612	8	2	-8	783	720
6	4	-6	175	145	-7	2	1	554	545	-7	4	4	1324	1343	7	4	-9	783	763	8	3	3	310	335	8	3	-8	973	948
-6	4	6	621	580	-7	3	1	1040	951	-7	4	-4	270	252	7	5	-9	135	105	-8	3	3	1108	1134	* -8	3	8	94	95
-6	4	-6	473	495	-7	3	-1	1243	1117	7	4	-4	1162	1155	-7	5	9	473	506	-8	4	3	1229	1258	-8	4	8	1419	1379
* -6	5	6	94	145	7	3	-1	1486	1445	7	5	-4	1594	1667	7	6	-9	608	605	-8	4	-3	756	693	8	4	-8	1216	1149
-6	5	-6	202	195	7	3	1	432	366	-7	5	-4	432	439	-7	0	10	702	699	8	4	-3	243	303	-8	5	8	594	581
6	5	-6	418	371	-7	4	1	1973	1916	-7	5	4	486	431	7	1	-10	135	81	8	5	-3	837	856	8	5	-8	351	384
-6	6	-6	932	910	-7	4	-1	324	271	7	6	-4	1202	1240	-7	1	10	567	556	-8	5	3	283	307	-8	6	8	351	329
6	6	-6	770	758	7	4	-1	594	615	-7	6	4	1554	1562	-7	2	10	243	236	-8	5	-3	283	249	8	6	-8	351	343
-6	6	6	905	870	7	4	1	702	565	-7	6	-4	148	106	7	2	-10	202	147	8	6	-3	1081	942	-8	0	9	1027	1096
-6	7	6	243	200	7	5	-1	256	194	-7	7	4	810	791	-7	3	10	405	425	-8	6	3	364	376	-8	1	9	567	563
-6	7	-6	675	687	7	5	1	608	547	-7	7	-4	1283	1240	7	3	-10	756	747	-8	6	-3	202	131	8	1	-9	310	294
6	7	-6	1094	1057	-7	5	-1	310	278	7	7	-4	243	214	7	4	-10	824	832	-8	7	-3	1351	1313	8	2	-9	837	853
-6	8	6	351	403	-7	5	1	1635	1677	-7	8	-4	148	143	7	5	-10	594	581	-8	7	3	540	548	-8	2	9	527	526
* -6	8	-6	94	111	7	6	1	378	320	* -7	8	4	94	95	-7	0	11	391	411	-8	8	-3	675	670	-8	3	9	202	179
6	8	-6	540	563	7	6	-1	459	399	-7	9	4	391	434	7	1	-11	135	148	-8	8	3	918	930	8	3	-9	1081	1076
* 6	0	7	94	58	-7	6	-1	959	902	7	0	-5	1351	1336	-7	1	11	675	687	-8	9	3	527	579	-8	4	9	1040	996
* 6	0	7	94	58	-7	6	1	432	435	7	0	5	554	518	7	2	-11	716	724	8	0	4	554	615	8	4	-9	1838	1844
-6	0	7	1756	1841	-7	7	-1	878	798	-7	1	5	405	444	7	3	-11	364	358	8	0	-4	243	219	-8	5	9	202	165
6	-1	7	878	944	-7	7	1	702	687	7	1	-5	1162	1114	8	0	0	1662	1588	-8	1	4	1405	1415	* -8	0	10	94	89
-6	1	7	445	474	-7	8	-1	148	91	7	1	5	864	807	8	-1	0	418	331	8	1	-4	1162	1145	-8	1	10	446	605
6	1	-7	1310	1260	-7	8	1	1189	1207	7	-1	5	527	494	8	1	0	1135	1170	8	-1	4	1135	1131	* 8	1	-10	94	99
6	2	-7	905	888	-7	9	1	716	717	7	2	5	473	428	-8	2	0	337	287	8	1	4	351	384	8	2	-10	675	632
6	-2	7	324	400	-7	9	-1	270	218	7	-2	5	243	219	8	2	0	135	96	8	2	4	351	359	-8	2	10	486	608
-6	2	7	473	489	7	0	-2	878	801	-7	2	5	1824	1929	-8	3	0	1405	1375	8	-2	4	554	559	-8	3	10	243	273
-6	3	7	1202	1163	7	0	2	378	467	7	2	-5	1905	1898	-8	4	0	283	223	-8	2	4	1067	1135	8	3	-10	513	510
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-6	4	7	973	962	7	-1	-2	351	288	-7	3	-5	432	440	-8	5	0	594	596	8	3	-4	1081	1120	9	0	0	1000	1141
-6	4	-7	283	413	7	1	2	973	1038	7	3	-5	297	284	8	5	0	756	746	-8	3	4	2000	2028	9	-1	0	1202	1065
6	4	-7	1202	1213	7	-1	2	1094	1305	-7	4	-5	148	197	-8	7	0	1067	1066	-8	3	-4	148	174	9	1	0	283	242
6	5	-7	581	600	* 7	2	2	94	79	7	4	-5	1094	1078	-8	8	0	216	112	-8	4	4	770	766	9	2	0	513	476
-6	5	7	567	473	7	2	-2	337	367	-7	4	5	1459	1493	-8	9	0	959	914	-8	4	-4	148	116	9	-2	0	986	910
-6	5	-7	1148	1294	-7	2	2	1621	1653	-7	5	-5	486	435	8	0	-1	905	817	8	4	-4	229	210	9	3	0	310	291
6	6	-7	310	301	7	-2	2	364	469	-7	5	5	256	267	8	0	1	1459	1263	-8	5	4	797	792	-9	3	0	1148	1164

Table 2 (*cont.*)

H	K	L	FOBS	FCAL	H	K	L	FOBS	FCAL	H	K	L	FOBS	FCAL	H	K	L	FOBS	FCAL	H	K	L	FOBS	FCAL	H	K	L	FOBS	FCAL
-9	4	0	1121	1049	-9	7	-4	567	520	-10	6	-1	783	956	10	0	-8	527	470	-11	7	5	756	709	-12	3	8	202	224
-9	5	0	675	676	-9	8	4	216	201	-10	7	1	216	141	-10	1	8	310	356	11	0	-6	1013	1010	* 13	-2	-1	94	207
-9	6	0	202	182	9	0	-5	459	446	-10	7	-1	581	534	10	1	-8	675	620	11	1	-6	351	348	-13	3	1	594	683
* -9	7	0	94	148	9	1	-5	1324	1372	-10	8	1	689	676	-10	2	8	324	349	11	-1	-6	283	254	-13	4	1	459	404
-9	8	0	919	938	-9	1	5	1135	1228	-10	8	-1	1027	899	10	2	-8	810	822	-11	2	6	567	617	* 13	-2	-2	94	153
-9	9	0	513	493	9	-2	5	1283	1223	10	0	-2	1000	998	10	3	-8	729	724	11	2	-6	283	261	-13	3	2	648	662
9	0	1	1094	1128	-9	2	5	783	826	10	0	2	810	1023	-10	3	8	378	399	-11	3	6	527	536	-13	4	2	310	348
9	0	-1	1081	1055	9	2	-5	175	145	10	-1	-2	919	867	-10	4	8	148	140	11	4	-6	432	382	* -13	5	2	94	150
9	1	1	1040	1049	-9	3	5	2675	3025	10	1	2	81	116	10	4	-8	770	770	-11	4	6	689	683	13	0	-3	513	478
9	-1	1	756	610	9	3	-5	500	578	10	-1	2	54	247	-10	5	8	324	297	-11	6	6	202	182	13	-1	-3	581	496
9	1	-1	540	493	9	-3	5	378	378	10	-2	2	527	710	-10	1	9	864	892	11	0	-7	1365	1387	-13	3	3	378	299
9	-1	-1	1054	1027	-9	4	5	554	446	10	2	-2	1594	1486	10	1	-9	743	733	11	1	-7	973	995	-13	4	3	216	271
9	-2	1	810	758	-9	4	-5	919	924	-10	2	2	1459	1466	* 10	2	-9	94	125	-11	2	7	148	102	-13	5	3	594	675
9	2	1	1040	935	9	4	-5	648	635	10	3	-2	554	558	10	3	-9	810	794	* 11	2	-7	94	62	13	-1	-4	364	348
-9	2	1	635	652	-9	5	5	824	809	-10	3	2	837	809	-10	3	9	797	795	11	3	-7	432	426	13	-2	-4	1067	1012
9	2	-1	797	726	9	5	-5	891	886	-10	3	-2	148	80	-10	4	9	378	348	-11	3	7	689	718	-13	3	4	1297	1361
-9	3	1	473	491	-9	5	-5	297	321	-10	4	-2	1392	1376	10	0	-10	783	795	-11	4	7	608	613	-13	4	4	540	529
-9	3	-1	837	758	9	6	-5	148	181	10	4	-2	540	485	-10	1	10	1229	1277	11	0	-8	202	202	-13	5	4	675	740
9	3	-1	486	310	-9	6	5	756	806	-10	4	2	432	381	10	1	-10	283	302	-11	1	8	729	758	13	0	-5	337	332
9	3	1	270	214	-9	7	5	1392	1407	-10	5	-2	554	516	-10	2	10	135	123	11	1	-8	202	115	13	-1	-5	500	484
9	4	-1	378	365	-9	8	5	148	164	-10	5	2	1054	1060	11	0	0	986	929	-11	2	8	432	438	-13	2	5	445	447
9	4	1	1283	1102	-9	1	6	1324	1418	-10	6	2	1094	1071	11	1	0	310	247	11	2	-8	594	554	-13	3	5	635	651
-9	4	-1	608	589	9	1	-6	1202	1235	-10	6	-2	878	880	11	-1	0	864	783	11	3	-8	432	459	-13	4	5	716	690
-9	5	1	905	859	-9	2	6	891	928	-10	7	2	337	298	11	2	0	405	378	* 11	3	8	94	128	13	0	-6	527	562
-9	5	-1	837	940	9	2	-6	459	453	* -10	7	-2	94	140	11	-2	0	662	684	11	0	-9	851	828	13	-1	-6	851	853
9	5	-1	567	522	-9	3	6	1946	1992	-10	8	2	310	238	-11	3	0	540	560	-11	1	9	283	326	-13	2	6	810	784
-9	6	-1	540	624	9	3	-6	135	155	10	0	3	405	526	-11	4	0	1459	1360	11	1	-9	608	566	-13	3	6	297	210
-9	6	1	635	573	-9	4	6	135	131	10	0	-3	919	861	-11	5	0	459	389	-11	2	9	243	159	-13	4	6	216	142
-9	7	-1	702	781	9	4	-6	743	818	10	-1	-3	973	978	-11	6	0	851	850	-11	3	9	473	437	13	-1	-7	283	253
-9	7	1	729	730	9	5	-6	1121	1171	10	1	-3	391	257	* -11	7	0	94	136	12	0	0	824	731	-13	2	7	459	464
-9	8	1	148	111	-9	5	6	486	501	10	-1	3	986	1113	11	0	-1	256	135	12	-1	0	1040	1137	-8	7	-5	797	866
* -9	8	-1	94	79	-9	6	6	459	432	10	2	-3	1716	1644	11	0	1	905	946	12	-2	0	216	164					
-9	9	1	1419	1327	9	6	-6	148	101	10	-2	3	635	721	11	-1	1	756	759	-12	3	0	783	772					
-9	9	-1	486	657	-9	7	6	689	734	-10	2	3	783	795	11	1	-1	864	768	-12	4	0	1243	1407					
9	0	-2	283	292	9	0	-7	189	136	10	3	-3	1189	1113	11	-1	-1	364	327	-12	5	0	1756	1742					
9	0	2	1973	2151	-9	1	7	851	905	10	-3	3	351	374	11	1	1	540	410	-12	6	0	270	259					
9	-1	-2	2216	2137	9	1	-7	229	213	-10	3	3	270	275	11	-2	-1	527	472	12	0	-1	148	112					
9	1	-2	486	437	9	2	-7	797	798	10	4	-3	500	444	11	-2	1	648	590	12	-1	-1	743	772					
9	1	2	851	964	-9	2	7	621	651	-10	4	-3	378	388	11	2	-1	865	832	12	1	-1	648	638					
9	-1	2	310	543	-9	3	7	621	641	-10	4	3	310	321	-11	3	-1	432	437	12	-2	1	162	130					
9	-2	2	121	218	9	3	-7	1067	1038	-10	5	3	675	732	-11	3	1	1135	1086	12	-2	-1	216	187					
-9	2	2	1459	1359	-9	4	7	729	612	-10	5	-3	486	478	11	3	-1	310	278	-12	3	1	648	787					
9	2	-2	1486	1425	9	4	-7	648	622	10	5	-3	256	237	-11	4	-1	2013	1995	12	-3	1	756	785					
9	2	2	1000	1093	* 9	5	-7	94	67	-10	6	3	702	706	-11	4	1	608	509	-12	4	-1	270	284					
* 9	3	2	94	54	-9	5	7	608	616	-10	6	-3	459	490	-11	5	1	919	853	* -12	4	1	94	120					
-9	3	2	324	307	-9	6	7	783	810	-10	7	3	594	557	-11	5	-1	1621	1523	-12	5	1	148	79					
-9	3	-2	1013	934	9	0	-8	405	348	-10	8	3	337	379	-11	6	1	513	487	-12	5	-1	973	890					
9	3	-2	973	929	9	1	-8	472	427	10	0	-4	189	138	-11	6	-1	810	755	-12	6	1	527	631					
-9	4	2	689	668	-9	1	8	513	481	10	0	-4	135	138	-11	7	-1	270	283	12	0	-2	770	725					
-9	4	-2	1473	1419	-9	2	8	310	334	10	-1	-4	540	502	-11	7	1	148	121	12	-1	-2	216	211					
9	5	-2	148	228	9	2	-8	716	734	10	1	-4	1000	984	* 11	0	2	94	63	12	1	-2	851	817					
-9	5	2	1351	1393	9	3	-8	94	68	10	2	-4	581	584	11	0	-2	1067	974	12	2	-2	810	710					
-9	6	2	648	606	-9	3	8	486	508	-10	2	4	959	985	11	-1	2	40	186	12	-2	-2	256	218					
-9	6	-2	540	542	-9	4	8	445	470	10	-2	4	567	546	11	1	-2	500	407	-12	3	2	729	721					
-9	7	2	905	852	9	4	-8	405	413	-10	3	4	1243	1255	* 11	-1	-2	94	85	-12	4	2	864	822					
-9	7	-2	986	1040	-9	5	8	378	342	10	-3	4	540	507	11	-2	2	432	487	-12	5	2	729	688					
-9	8	-2	932	999	9	5	-8	716	721	10	3	-4	148	162	11	2	-2	256	242	-12	6	2	310	262					
* -9	8	2	94	131	9	0	-9	1121	1079	-10	4	4	1054	1092	11	-2	-2	621	591	12	0	-3	729	613					
-9	9	2	1256	1223	-9	1	9	1243	1336	10	4	-4	756	780	11	-3	2	527	620	12	1	-3	851	893					
9	0	-3	297	289	9	1	-9	594	572	-10	4	-4	635	670	-11	3	2	202	162	12	-1	-3	446	355					
9	0	3	243	241	-9	2	9	378	390	10	5	-4	554	468	11	3	-2	459	463	12	2	-3	1040	920					
9	-1	3	675	704	9	2	-9	243	147	-10	5	-4	337	273	-11	4	2	418	445	12	-2	-3	1000	991					
9	1	-3	1554	1466	-9	3	9	351	348	-10	5	4	756	731	* -11	5	2	94	49	-12	3	3	662	647					
9	-1	-3	554	620	9	3	-9	1689	1701	-10	6	4	445	474	-11	6	2	837	842	-12	4	3	445	536					
-9	2	3	864	799	9	4	-9	1027	1050	-10	7	4	689	680	-11	6	-2	851	797	-12	5	3	148	199					
9	2	-3	135	128	-9	4	9	1216	1237	-10	8	4	256	263	* -11	7	2	94	106	-12	6	3	1175	1340					
9	-2	3	756	738	-9	0	10	621	623	10	0	-5	527	629	11	0	-3	1040	1083	12	0	-4	851	765					
9	2	3	581	652	-9	1	10	378	382	10	-1	-5	540	577	11	1	-3	946	927	12	-1	-4	202	148					
-9	3	3	1337	1376	9	1	-10	310	322	-10	2	5	919	998	* 11	-1	-3	94	130	12	1	-4	1351	1262					
-9	3	-3	256	251	9	2	-10	486	471	10	2	-5	892	935	-11	2	3	1702	1712	-12	2	4	1446	1427					
9	3	-3	608	574	9	3	-10	756	816	10	3	-5	729	766	11	2	-3	2513	2312	12	2	-4	1662	1774					
-9	4	3	905	968	10	0	0	459	482	-10	3	5	810	832	11	3	-3	432	413	-12	3	4	864	888					
-9	4	-3	472	454	10	-1	0	378	343	10	4	-5	256	220	-11	3	3	756	732	-12	4	4	594	594					
9	4	-3	324	316	10	2	0	1094	983	-10	4	5	446	408	-11	4	3	148	118	* -12	5	4	94	70					
-9	5	-3	148	190	10	-2	0	1175	1056	* 10	5	-5	94	98	-11	5	3	702	679	-12	6	4	1473	1443					
9	5	-3	621	579	-10	3	0	608	575	-10	5	5	608	572	-11	6	3	1567	1492	12	0	-5	756	898					
-9	6	3	243	194	* 10	3	0	94	102	-10	6	5	297	269	-11	7	3	216	178	12	1	-5	500	435					
-9	6	-3	648	700	-10	5	0	1081	981	-10	7	5	621	573	11	0	-4	783	742	12	-1	-5	486	516					
-9	7	3	675	586	-10	6	0	675	654	10	0	-6	837	914	11	0	-4	810	742	-12	2	5	148	118					
-9	7	-3	310	287	-10	7	0	405	430	10	1	-6	1148	1192	11	-1	-4	729	663	12	2	-5	148	177					
-9	8	3	581	547	-10	8	0	270	206	-10	1	6	946	976	11	1	-4	283	180	-12	3	5	256	258					
-9	8	-3	337	332	10	0	1	1702	1766	10	2	-6	310	257	-11	2	4	783	768	-12	4	5	662	594					
9	0	-4	324	289	10	0	-1	756	685	-10	3	6	1716	1826	11	2	-4	1175	1115	-12	5	5	148	104					
9	0	4	1013	850	10	-1	1	932	791	10	3	-6	472	512	-11	3	4	405	384	-12	6	5	216	184					
9	1	-4	175	122	10	1	1	216	190	-10	4	6	689	718	-11	4	4	635	702	12	-1	-6	283	318					
9	-1	4	1000	1027	10	1	-1	378	389	10	4	-6	905	969	11	4	-4	662	619	12	1	-6	527	531					
9	-1	-4	1081	1106	10	-1	-1	459	379	10	5	-6	770	764	* -11	5	4	94	67	-12	2	6	648	583					
-9	2	4	1459	1481	10	2	-1	364	345	-10	5	6	351	286	-11	6	4	337	309	12	2	-6	527	471					
9	2	-4	1013	972	10	-2	-1	243	224	-10	6	6	445	443	-11	7	4	837	798	-12	3	6	256	160					
-9	3	4	418	405	10	-2	1	500	419	-10	7	6	1392	1378	11	0	-5	351	296	-12	4	6	297	249					
9	3	-4	986	969	10	2	1	1040	1113	10	0	-7	364	367	11	-1	-5	243	170	12	0	-7	1419	1487					
-9	4	4	770	757	-10	3	1	1094	1089	10	1	-7	662	733	11	1	-5	1054	1071	12	1	-7	243	296					
-9	4	-4	473	542	-10	3	-1	689	740	-10	1	7	783	770	11	2	-5	837	859	12	-1	-7	702	670					
9	4	-4	378	444	10	3	-1	270	292	10	2	-7	202	193	-11	2	5	729	736	-12	2	7	148	134					
9	5	-4	432	531	-10	4	-1	2284	2217	-10	2	7	243	235	11	3	-5	919	857	12	2	-7	202	251					
-9	5	-4	337	252	10	4	-1	567	506	-10	3	7	351	374	-11	3	5	513	501	-12	3	7	391	420					
-9	5	4	919	993	-10	4	1	1013	1039	* 10	3	-7	94	153	-11	4	5	324	313	-12	4	7	729	732					
-9	6	-4	364	353	-10	5	-1	473	464	10	4	-7	351	333	11	4	-5	459	480	12	0	-8	1000	986					
9	6	-4	851	778	-10	5	1	513	507	-10	5	7	662	627	-11	5	5	391	355	12	1	-8	283	259					
-9	7	4	202	125	-10	6	1	364	291	-10	6	7	567	547	-11	6	5	810	824	-12	2	8	432	446					

related to it by a centre of inversion. Thus, for convenience of identification, Pd(1) for example, is related to Pd(1′) by a centre of symmetry. A diagrammatic representation of the structure projected down the c^* and b^* axes is shown in Figs. 3 and 4 respectively. From these diagrams it becomes obvious that in this crystal structure the compound exists not as a monomer but as a hexamer consisting of six $Pd(S.C_3H_7)_2$ fragments. The basic unit of this large molecule consists of one palladium and four sulphur atoms arranged in an approximately square planar configuration. Six units of this type are linked to each other by the sharing of two sulphur atoms between every two adjacent palladium atoms. The sulphur atoms thus act as a bridge between two palladium atoms. The Pd–S–Pd bridge angles vary between 79·7 and 85·8°. The nature of the sharing of sulphur atoms can be seen in Fig. 2(*a*). In the resulting structure then, there are two six-membered sulphur rings which are S(1), S(3), S(5′), S(6′), S(2′), S(4) and S(1′), S(3′), S(5), S(6), S(2), S(4′). These are packed approximately above each other and one ring is related to the other by a centre of inversion. The average distance between the two rings is 3·09 Å. There is also a ring of six palladium atoms which is

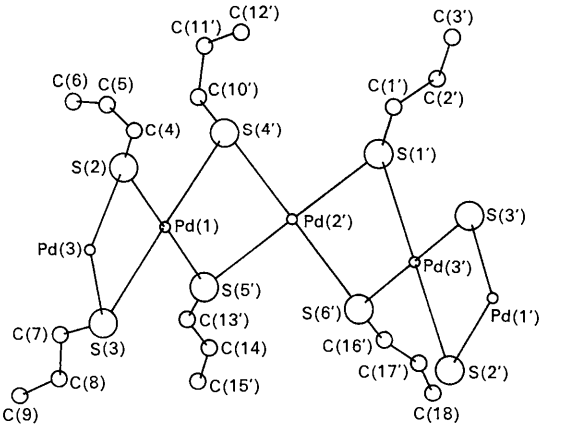

Fig. 1. Schematic diagram of a part of the molecule of palladium n-propyl mercaptide.

Table 3. *Dimensions of the molecule*

	Bond length (Å)	σ (Å)		Angles (°)	σ (°)
Pd(1)–S(2)	2·305	0·004	S(5)—Pd(1)–S(4)	82·0	0·2
Pd(1)–S(3)	2·399	0·004	S(5)—Pd(1)–S(3)	98·2	0·2
Pd(1)–S(4)	2·413	0·004	S(5)—Pd(1)–S(2)	177·8	0·2
Pd(1)–S(5)	2·314	0·005	S(4)—Pd(1)–S(3)	176·6	0·2
			S(4)—Pd(1)–S(2)	97·6	0·2
			S(3)—Pd(1)–S(2)	82·1	0·2
Pd(2)–S(1)	2·450	0·005	S(6)—Pd(2)–S(5)	98·7	0·2
Pd(2)–S(4)	2·358	0·004	S(6)—Pd(2)–S(4)	174·6	0·2
Pd(2)–S(5)	2·454	0·004	S(6)—Pd(2)–S(1)	80·3	0·2
Pd(2)–S(6)	2·331	0·005	S(5)—Pd(2)–S(4)	80·3	0·2
			S(5)—Pd(2)–S(1)	177·9	0·2
			S(4)—Pd(2)–S(1)	100·5	0·2
Pd(3)–S(1)	2·282	0·004	S(6)—Pd(3)–S(3)	174·0	0·2
Pd(3)–S(2)	2·288	0·004	S(6)—Pd(3)–S(2)	94·7	0·2
Pd(3)–S(3)	2·303	0·005	S(6)—Pd(3)–S(1)	84·5	0·2
Pd(3)–S(6)	2·308	0·005	S(3)—Pd(3)–S(2)	84·6	0·2
			S(3)—Pd(3)–S(1)	96·0	0·2
			S(2)—Pd(3)–S(1)	177·4	0·2
S(1)–C(1)	1·82	0·02	C(1)—S(1)—Pd(3)	113·3	0·7
C(1)–C(2)	1·60	0·03	C(1)—S(1)—Pd(2)	110	0·7
C(2)–C(3)	1·46	0·04	C(2)—C(1)—S(1)	110	1·4
			C(3)—C(2)—C(1)	110	2·0
S(2)–C(4)	1·92	0·02	C(4)—S(2)—Pd(1)	104·9	0·6
C(4)–C(5)	1·62	0·03	C(4)—S(2)—Pd(3)	110·5	0·6
C(5)–C(6)	1·46	0·04	C(5)—C(4)—S(2)	104	1·4
			C(4)—C(5)—C(6)	115	2·2
S(3)–C(7)	1·89	0·05	C(7)—S(3)—Pd(3)	105	1·6
C(7)–C(8)	1·52	0·03	C(7)—S(3)—Pd(1)	108	0·6
C(8)–C(9)	1·59	0·04	C(8)—C(7)—S(3)	116	1·7
			C(7)—C(8)—C(9)	113	2·3
S(4)—C(10)	1·81	0·02	C(10)–S(4)—Pd(1)	99	0·6
C(10)–C(11)	1·58	0·03	C(10)–S(4)—Pd(2)	107	0·6
C(11)–C(12)	1·48	0·04	C(11)–C(10)–S(4)	114	1·4
			C(10)–C(11)–C(12)	110	201
S(5)—C(13)	1·84	0·02	C(13)–S(5)—Pd(1)	112	0·6
C(13)–C(14)	1·43	0·03	C(13)–S(5)—Pd(2)	110	0·7
C(14)–C(15)	1·50	0·03	C(14)–C(13)–S(5)	113	1·4
			C(13)–C(14)–C(15)	111	2·0
S(6)—C(16)	1·88	0·02	C(16)–S(6)—Pd(3)	100	0·7
C(16)–C(17)	1·47	0·04	C(16)–S(6)—Pd(2)	105	0·7
C(17)–C(18)	1·54	0·04	C(17)–C(16)–S(6)	115	1·8
			C(16)–C(17)–C(18)	112	2·8

sandwiched between the two sulphur rings in an antiprismatic configuration. Mean planes passing through the palladium and sulphur atoms were calculated to see if these rings were planar. From the results of these calculations it can be seen that (see Table 6) the rings are not planar but puckered. The dimensions of the rings are shown in Figs. 2(*a*) and 2(*b*). The sulphur–sulphur bonds in the sulphur ring vary between 3·38–3·70 Å and the S–S–S angles between 114·1°–124·2°. In the palladium ring the palladium distances vary between 3·014 and 3·235 Å and the angles between 116·3° and 123·7°.

The n-propyl groups to which sulphur is bonded are located outside the framework of palladium and sulphur atoms. Since the carbon–sulphur bond is a single bond, the n-propyl groups can rotate around this bond and orient themselves to sterically suitable positions in the crystal structure. The geometry of the molecule of (Pd n-propyl mercaptide)$_6$ is very interesting. The molecule has a cage structure and has a large hole of 7 Å diameter within it.

The Pd–S distances vary between 2·282 and 2·454 Å. Mean planes were calculated to test the planarity of the PdS_4 units. The deviations of a number of atoms from the mean planes are significant in all the three independent PdS_4 units. Of the six different carbon–sulphur bonds, three are normal, the lengths being about 1·82 Å. The remaining three bonds have somewhat larger value (see Table 3). The agreement between the carbon–carbon distances in the six independent n-propyl groups is not very satisfactory. But considering that the carbon atoms were located in the presence of heavy palladium and sulphur atoms, little significance need be attached to these deviations.

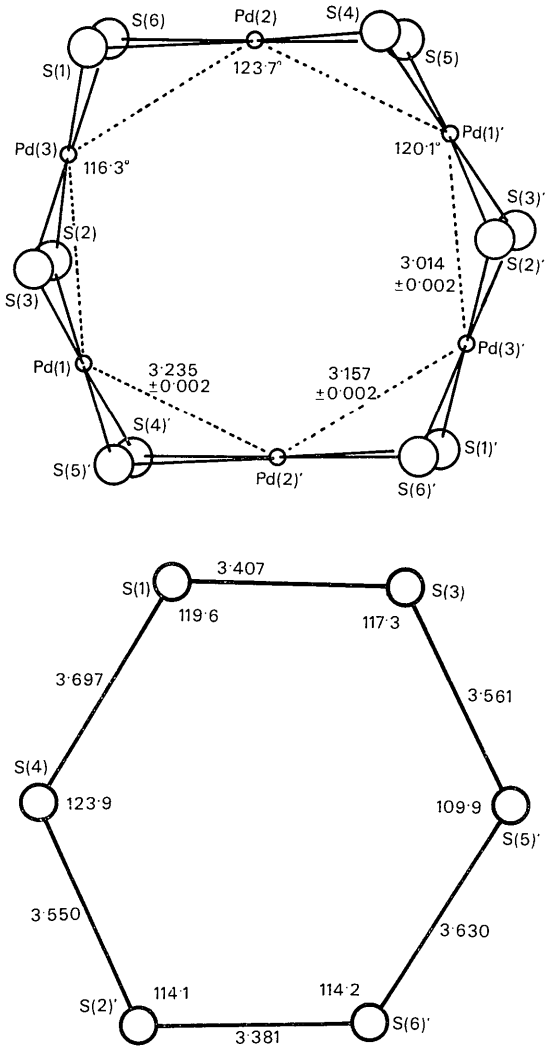

Fig. 2. A diagram showing (*a*) the dimensions of the ring of six palladium atoms and the sharing of sulphur atoms between them. (*b*) The dimensions of the ring of six sulphur atoms.

Table 4. *Intermolecular approaches*

	Lenght (Å)	σ (Å)
C(6′)—C(6)	4·39	0·04
C(12′)–C(18)	4·02	0·04
C(9) —C(18)	5·10	0·04
C(9) —C(9′)	4·39	0·04
C(9) —C(18′)	4·27	0·04
C(6) —C(12)	4·66	0·04
C(6) —C(18′)	4·10	0·04
C(9) —C(15)	5·33	0·04
C(9) —C(12)	4·55	0·04
C(7) —C(15)	3·81	0·04
C(16) –C(18)	4·53	0·04
C(6) —C(9)	4·19	0·04

The manner in which the various groups are packed in the crystal structure can be seen in Figs. 3 and 4. The various intermolecular close approaches are listed in Table 4. The short intermolecular approaches are those between the terminal methyl groups of the n-propyl chains, and their values vary between 3·81 and 5·33 Å. None of their values are smaller than the van der Waals diameter of the methyl group.

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Table 5. *Sulphur bridge angles*

Pd(1)–S(2)–Pd(3)	82·0° ±0·1°
Pd(1)–S(3)–Pd(3)	79·7 ±0·1
Pd(3)–S(1)–Pd(2)	83·6 ±0·1
Pd(3)–S(6)–Pd(2)	85·8 ±0·1
Pd(2)–S(4)–Pd(1′)	85·4 ±0·1
Pd(2)–S(5)–Pd(1′)	85·4 ±0·1

Science Foundation, NIH-A-3942 from the National Institutes of Health and Roswell Park Buildings Research Grant.

Table 6. *Least-squares planes and the out-of-plane distances of the plane determining atoms*

Plane number 1. This plane is defined by 5 atoms. The equation of the plane is $0{\cdot}1524P+0{\cdot}9550Q-0{\cdot}2546R=0{\cdot}242$ Å. The standard deviation of the distances from the plane is 0·613 Å. Out-of-plane distances of the plane-determining atoms are:

Pd(1)	−1·130 Å
S(3)	0·495
S(4′)	0·086
S(5′)	−0·041
S(2)	0·590

Plane number 2. This plane is defined by 5 atoms. The equation of the plane is $0{\cdot}1168P+0{\cdot}9494Q+0{\cdot}2916R=3{\cdot}738$ Å. The standard deviation of the distances from this plane is 0·306 Å. Out-of-plane distances of the plane-determining atoms are:

Pd(2)	−0·384 Å
S(1)	0·067
S(5)	−0·330
S(4)	0·334
S(6)	0·313

Plane number 3. This plane is defined by 5 atoms. The equation of the plane is $0{\cdot}5168P+0{\cdot}8045Q+0{\cdot}2929R=2{\cdot}971$ Å. The standard deviation of the distances from the plane is 0·2197 Å. Out-of-plane distances of the plane-determining atoms are:

Pd(3)	0·141 Å
S(1)	0·096
S(2)	0·095
S(3)	−0·438
S(6)	0·107

Plane number 4. This plane is defined by 6 atoms. The equation of the plane is $-0{\cdot}4961P+0{\cdot}8180Q-0{\cdot}2912R=0{\cdot}000$ Å. The standard deviation of the distances from the plane is 1·160 Å. Out-of-plane distances of the plane-determining atoms are:

Pd(1)	−1·683
Pd(2)	0·242
Pd(3)	0·884
Pd(1′)	1·683
Pd(2′)	−0·242
Pd(3′)	−0·884

Plane number 5. This plane is defined by 6 atoms. The equation of the plane is $0{\cdot}1147P+0{\cdot}9698Q-0{\cdot}2154R=-0{\cdot}771$ Å. The standard deviation of the distances from the plane is 1·342 Å. Out-of-plane distances of the plane-determining atoms are:

S(2)	1·605 Å
S(5)	0·471
S(6)	−0·377
S(1′)	−2·604
S(3′)	−0·129
S(4′)	1·035

Angles between the planes

Plane *A*	Plane *B*	Angle
1	3	140·6°
2	3	155·4
1	4	148·2

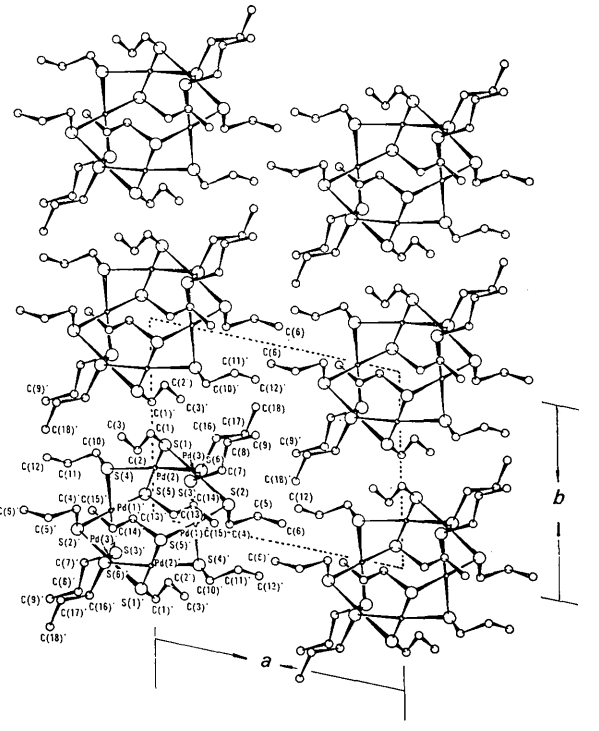

Fig. 3. A diagrammatic representation of the structure projected down the c^* axis.

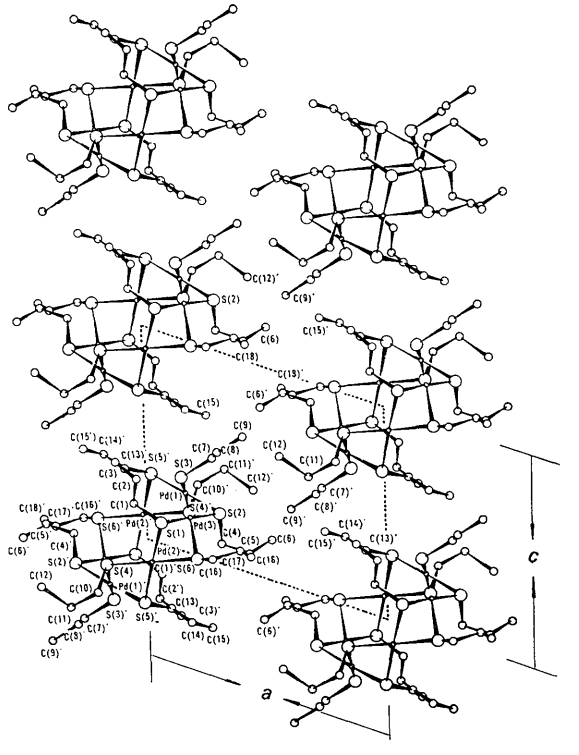

Fig. 4. A diagrammatic representation of the structure projected down the b^* axis.

Table 7. *Anisotropic thermal vibration parameters*

The B_{ij} are coefficients in the temperature factor expression:
$\exp[-(B_{11}h^2+B_{22}k^2+B_{33}l^2+B_{12}hk+B_{13}hl+B_{23}kl)]$
$U_{11}=B_{11}/2\pi^2a^{*2}$; $U_{12}=B_{12}/4\pi^2a^*b^*$; *etc.*

	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Pd(1)	0·0399	0·0489	0·0496	0·000	−0·0015	−0·006
Pd(2)	0·0451	0·0486	0·0503	0·0000	−0·0047	0·0019
Pd(3)	0·0402	0·0489	0·0481	0·0003	−0·0026	−0·0009
S(1)	0·0494	0·0474	0·0520	0·0064	−0·0062	−0·0031
S(2)	0·0348	0·0516	0·0523	0·0005	0·0010	0·0001
S(3)	0·0457	0·0506	0·0563	0·0047	0·0109	0·0026
S(4)	0·0466	0·0551	0·0494	0·0029	−0·0078	−0·0035
S(5)	0·0444	0·0560	0·0473	−0·0020	−0·0003	0·0079
S(6)	0·0459	0·0599	0·0503	−0·0019	0·0000	0·0046
C(1)	0·0502	0·0841	0·0785	0·0046	0·0174	0·0017
C(2)	0·0683	0·0857	0·0651	0·0120	0·0176	−0·0072
C(3)	0·1049	0·1377	0·1272	0·0057	0·0290	0·0041
C(4)	0·0467	0·0890	0·0536	0·0019	−0·0019	−0·0130
C(5)	0·0879	0·1300	0·0895	−0·0247	0·0091	−0·0163
C(6)	0·0638	0·1927	0·1152	0·0340	−0·0090	0·0099
C(7)	0·0776	0·0691	0·0627	−0·0059	0·0150	−0·0005
C(8)	0·2111	0·0819	0·1163	0·0313	−0·0545	−0·0428
C(9)	0·2451	0·1225	0·0999	0·0203	−0·0841	−0·0454
C(10)	0·0747	0·0621	0·0629	−0·0063	−0·0234	0·0154
C(11)	0·0787	0·0839	0·0648	0·0032	−0·0334	−0·0021
C(12)	0·00469	0·1519	0·1819	0·0334	−0·0334	−0·0142
C(13)	0·0516	0·0604	0·0842	0·0172	0·0172	0·0082
C(14)	0·0388	0·0991	0·0570	−0·0072	−0·0015	0·0136
C(15)	0·0777	0·1553	0·1021	0·0184	−0·0138	0·0190
C(16)	0·0709	0·0874	0·0998	−0·0600	−0·0099	0·0113
C(17)	0·0840	0·0939	0·2147	−0·0096	0·0059	0·0131
C(18)	0·1264	0·1119	0·2526	−0·0426	−0·0343	−0·0337

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The Crystal and Molecular Structure of 4,4′-Diamino-3,3′-dichlorobiphenyl

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4,4′-Diamino-3,3′-dichlorobiphenyl, $C_{12}H_{10}N_2Cl_2$, crystallizes with cell dimensions $a=12{\cdot}5$, $b=3{\cdot}85$, $c=23{\cdot}7$ Å, $\beta=108°$, space group $P2_1/c$ and $Z=4$. The structure has been determined from three-dimensional X-ray data and refined by the minimum residual method, with isotropic temperature factors for individual atoms, to give a final R index of 14%. The two phenyl rings are not coplanar, but are twisted around the linkage between them, C(1)–C(1′), so as to be mutually inclined at an angle of 21°; in addition, each phenyl ring is bent through a small angle (approximately 2·2° and 3·4° respectively) away from the line C(1)–C(1′). The length of the bond C(1)–C(1′) is 1·515 ± 0·024 Å.

Introduction and experimental

A preliminary examination of one projection of the structure of 4,4′-diamino-3,3′-dichlorobiphenyl by Toussaint (1948) gave an electron density map with spurious symmetry in addition to the true symmetry of the actual structure. The map suggested that the molecules are probably planar, or very nearly so, with chlorine atoms in the *trans* positions. We have now completed the analysis of the structure, using three-dimensional X-ray data kindly supplied by Dr Toussaint, and find that the phenyl rings are mutually inclined at an angle of 21°.

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