therefore suggest that the lengthening of the C(1)–C(7) bond is caused by this rotation, which has decreased the overlap between the two p_z orbitals and therefore decreased the double-bond character of the bond.

The molecule is distorted from planarity by an unusual amount. Omitting the 'outside' atoms of the isobutyl group, C(10), C(11), C(12), the distortion of one half of the molecule from complete planarity may be approximately described by a rotation of the (nearly) planar group C(1), C(7), C(8), N, C(9), Cu through an angle of 25° about the line C(1)–C(9). Equations for various least-squares planes and the distances of the several atoms from these planes are given in Table 4. Comparison of these distances with those found in other compounds shows a number of differences. The metal atom is farther (1·12 Å) out of the plane defined by the benzene ring, the farthest heretofore reported being 0·75 Å in bis-(*N*-t-butylsalicylaldiminato)palladium(II) (Day, Glick & Hoard, 1968). The nitrogen atom is also farther (0·25 Å) out of the plane and is out in the same direction as the metal, whereas in other compounds it is out in the opposite direction from the metal atom (*cf.* Table 6, Jain & Lingafelter, 1967). The dihedral angle between the coordination plane CuN_2O_2 and the mean plane of the *o*-hydroxyacetophenone imine residue is 136·6°, which may be compared with the corresponding angle of 144·4° in bis-(*N*-t-butylsalicylaldiminato)palladium.

The packing of the molecules may be seen in Fig. 2. None of the intermolecular contact distances is unusual.

This investigation was supported by the U.S. National Science Foundation under Grant GP-9201.

References

ALEXANDER, L. E. & SMITH, G. S. (1964). *Acta Cryst.* **17**, 1195.

CROMER, D. & WABER, J. (1965). *Acta Cryst.* **18**, 104.

DAY, V. W., GLICK, M. D. & HOARD, J. L. (1968). *J. Amer. Chem. Soc.* **90**, 4803.

HALL, D., SHEAT, S. V. & WATERS, T. N. (1968). *J. Chem. Soc.* A**1968**, 460.

JAIN, P. C. & LINGAFELTER, E. C. (1967). *Acta Cryst.* **23**, 127.

LINGAFELTER, E. C. & BRAUN, R. L. (1966). *J. Amer. Chem. Soc.* **88**, 2951.

LINGAFELTER, E. C., SIMMONS, G. L., MOROSIN, B., SCHERINGER, C. & FREIBURG, C. (1961). *Acta Cryst.* **14**, 1222.

STEWART, J. M. (1964). *Crystal Structure Calculation System for the IBM* 709, 7090, 7094. Technical Report TR-64-6, Computer Science Center, Univ. of Maryland and Research Computer Laboratory, Univ. of Washington.

STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* **42**, 3175.

WATERS, T. N. & HALL, D. (1959). *J. Chem. Soc.* p. 1200.

WEI, L., STOGSDILL, R. M. & LINGAFELTER, E. C. (1964). *Acta Cryst.* **17**, 1058.

Acta Cryst. (1970). B**26**, 1812

The Crystal Structure of Compounds with (N-P)$_n$ Rings. VII.* Refinement of the Crystal Structure of Hexabromocyclotriphosphazene, $N_3P_3Br_6$

BY H. ZOER AND A. J. WAGNER

Laboratory of Structural Chemistry, The University, Bloemsingel 10, *Groningen, The Netherlands*

(*Received* 4 *August* 1969)

The crystal structure of $N_3P_3Br_6$, as determined by Giglio & Puliti (1967), has been refined from three-dimensional intensity data, collected with Mo radiation on an automatic three-circle diffractometer. Two crystals were used. Data processing and refinement were carried out independently with the data obtained from each crystal. The intensities were corrected for absorption and during the refinement a correction for extinction was applied according to Zachariasen (1967, 1968). Atomic coordinates obtained from the two crystals agree within the standard deviations. The molecules $N_3P_3Br_6$ lie on crystallographic mirror planes of the space group *Pnma*, with unit-cell dimensions $a=14·463$ (0·002), $b=13·410$ (0·003) and $c=6·601$ (0·001) Å. There are four short intermolecular Br···Br distances (3·65–3·85 Å) and a short N···Br distance (3·24 Å). The two independent angles P–N–P of a molecule are significantly different: 119·3 (0·6) and 122·4 (0·5)°. Other chemically equivalent bond lengths and angles are equal within experimental error. Mean values (and individual standard deviations) are P–N 1·576 (0·008), P–Br 2·162 (0·004) Å, N–P–N 118·5 (0·5) and Br–P–Br 102·1 (0·1)°. The $(N–P)_3$ ring is non-planar and slightly chair-shaped. The dihedral angles of the ring bonds are $-6·7$, 6·3 and $-5·5$° and the largest distance from a ring atom to the least-squares plane through the ring is 0·047 Å.

Introduction

The crystal structures of several chloro- and fluorocyclophosphazenes are known with some precision, *viz.* $N_3P_3Cl_6$ (Wilson & Carroll, 1960), $N_4P_4Cl_8$ [two crystal modifications, *K* form (Hazekamp, Migchelsen & Vos, 1962) and *T* form (Wagner & Vos, 1968)], $N_5P_5Cl_{10}$ (Schlueter & Jacobson, 1968), $N_3P_3F_6$ (Dougill, 1963) and $N_4P_4F_8$ (McGeachin & Tromans, 1961).

---

* Part VI: Olthof (1969).

As to the bromo compounds the only crystal structure known is that of the trimer $N_3P_3Br_6$. This structure was determined by Giglio & Puliti (1967) from the intensities of 538 X-ray reflexions, estimated visually on Weissenberg films. The accuracy of the results is not high, *e.g.* the values found for the P–Br bond lengths range from 2·14 to 2·20 Å with an individual standard deviation of 0·015 Å.

We intend to investigate the crystal structures of the homologous bromocyclophosphazenes in order to elucidate the conformations of the phosphazene rings. Refinement of the crystal structure of the trimer $N_3P_3Br_6$, from new experimental data, seemed necessary, if reliable comparisons with the structures of related compounds are to be made.

Since absorption effects of X-rays in crystals of the bromocyclophosphazenes will be very substantial (linear absorption coefficient for Mo radiation more than 200 cm^{-1}), we thought it worth while to use two independent sets of intensities, obtained from two different crystals, for the refinement of the crystal structure of $N_3P_3Br_6$, in order to see whether the determined atomic coordinates would be essentially the same.

The results of the refinement of $N_3P_3Br_6$ are given in the present paper. The crystal structure of the tetramer $N_4P_4Br_8$, which has been determined by us, will be described in a following paper.

Experimental

The substance was prepared from phosphorus(III) bromide, ammonium bromide and bromine according to the procedure of Coxon, Sowerby & Tranter (1965). From the reaction mixture, containing trimeric and tetrameric bromocyclophosphazene and possibly higher homologues, suitable crystals of $N_3P_3Br_6$ were obtained by fractional crystallization from petroleum.

The crystals are orthorhombic. Weissenberg photographs showed the systematic absences $0kl$ for $k+l=2n+1$ and $hk0$ for $h=2n+1$. The space group therefore is *Pnma* (no. 62) or *Pn*2$_1$*a* (no. 33). The unit cell contains four molecules $N_3P_3Br_6$. In the refinement it was assumed that *Pnma* is the correct space group, implying that the molecules have a crystallographic mirror plane.

Accurate unit-cell dimensions were determined from zero-level Weissenberg photographs around the *b* and *c* axes. Reflexions from a NaCl crystal were superposed on the films for calibration. The lengths of the unit-cell edges are:

	Present work		Giglio & Puliti (1967)	
$a=$	14·463, s.d.	0·002 Å	14·43, s.d.	0·02 Å
$b=$	13·410	0·003	13·36	0·02
$c=$	6·601	0·001	6·63	0·01

Intensities were measured at room temperature on a single-crystal three-circle automatic Nonius diffractometer by the θ–2θ scan method. All 1176 independent reflexions up to $\sin\theta/\lambda=0{\cdot}60$ Å^{-1} were measured using Zr filtered Mo radiation. The intensities of all reflexions were measured twice; two different crystals (I and II) of approximate dimensions $0{\cdot}1\times0{\cdot}1\times0{\cdot}2$ mm and $0{\cdot}2\times0{\cdot}2\times0{\cdot}3$ mm respectively were used. Crystal I gave 995 non-zero reflexions and crystal II 1004. The data for each crystal were processed separately and the refinement was carried out with each series of F_o values independently.

The intensities were corrected for Lorentz and polarization effects and for absorption. The latter corrections were calculated with a computer program based on the Busing & Levy (1957) scheme. The linear absorption coefficient for Mo radiation is very high, *viz.* 203 cm^{-1}. The transmission factor $(1/V)\int\exp(-\mu d)dV$ ranged from 0·10 to 0·14 for I and from 0·02 to 0·06 for II. After the absorption correction the F_o values of the two crystals were compared. After scaling the factor $R'=\sum||F_{\rm I}|-|F_{\rm II}||/\sum|F_{\rm I}|$ was 10·6% for the 899 common reflexions. For the stronger reflexions the scaled F_o values of II were generally smaller than those of I. As will be seen later this behaviour can only partly be ascribed to extinction effects.

Refinement

Least-squares refinements were carried out based on the F_o values of each crystal separately. A least-squares program working according to the block-diagonal approximation (Cruickshank, 1961) was used. The atomic coordinates and thermal parameters from the paper by Giglio & Puliti (1967) were taken as a

Table 1. *Final fractional coordinates*

Standard deviations are in parentheses.

	Crystal I			Crystal II		
	x	*y*	*z*	*x*	*y*	*z*
N(1)	0·1430 (10)	0·25	0·5967 (30)	0·1434 (11)	0·25	0·5940 (29)
N(2)	−0·0077 (7)	0·1499 (8)	0·4566 (19)	−0·0081 (8)	0·1507 (9)	0·4528 (24)
P(1)	−0·0605 (3)	0·25	0·4121 (9)	−0·0607 (4)	0·25	0·4117 (9)
P(2)	0·0907 (2)	0·1477 (3)	0·5556 (6)	0·0910 (3)	0·1480 (3)	0·5554 (6)
Br(1)	−0·1895 (2)	0·25	0·5783 (4)	−0·1895 (2)	0·25	0·5789 (4)
Br(2)	−0·1078 (2)	0·25	0·1015 (4)	−0·1078 (2)	0·25	0·1019 (4)
Br(3)	0·0823 (1)	0·0714 (1)	0·8445 (3)	0·0824 (1)	0·0713 (1)	0·8442 (3)
Br(4)	0·1806 (1)	0·0507 (1)	0·3845 (3)	0·1806 (1)	0·0505 (1)	0·3844 (3)

starting point. Atomic scattering factors were taken from Doyle & Turner (1968).

In the initial cycles the scale factor, the atomic coordinates and anisotropic thermal parameters were refined without accounting for anomalous scattering effects and with uniform weights. Later the anomalous scattering by phosphorus and bromine (*International Tables for X-ray Crystallography*, 1962) was incorporated and a weighting scheme of the form $w=\{w_c^{-1}+p|F_o|^2\}^{-1}$ was introduced. The term w_c is the weight from counting statistics and p is a constant, chosen so as to make $(\Delta F)^2$, averaged over groups of reflexions, as far as possible independent on $|F_o|$. The value 4×10^{-4} was given to p for crystal I and 9×10^{-4} for II. Furthermore, the extinction parameter $p_{\rm ex}$ was refined according to Zachariasen's (1967, 1968) method. This parameter occurs in the function M, which is to be minimized, as follows:

$$M=\sum w[|F_o|-K|F_c|\{1+C(\theta)p_{\rm ex}|F_c|^2\}^{-1/4}]^2$$

$$\text{with } C(\theta)=10^{-2}\,(p_2/p_1)\,(1/\sin 2\theta)\,\bar{T}$$

$$\text{and } p_{\rm ex}=2\times10^2\,(e^2/mc^2)^2\,(\lambda^2/V^2)\,r^*$$

(See Zachariasen (1968) for the meaning of the symbols.)

At the end of the refinement the indices $R=\{\sum|\Delta F|^2/\sum|F_o|^2\}^{1/2}$ and $R_w=\{\sum w|\Delta F|^2/\sum w|F_o|^2\}^{1/2}$ were 9·6 and 6·5% for crystal I and 9·4 and 8·0% for II. The theoretical values for R and R_w based on counting statistics are lower, *viz.* 8·5 and 4·2% for I, and 7·6 and 5·2% for II. The differences between F_o and F_c are thus larger than could be expected on the basis of counting statistics alone, indicating that there are additional errors in the F_o values. Presumably these errors are mainly due to the difficulties encountered in calculating the absorption corrections. The refined value of $p_{\rm ex}$ is 0·005 for I and 0·014 for II. With these numbers the extinction corrections $y^{1/2}=\{1+C(\theta)p_{\rm ex}|F_c|^2\}^{-1/4}$ are calculated as about 0·95 and 0·85 for the strong reflexions of I and II respectively. It thus appeared that the extinction effects are rather small in both crystals, though somewhat larger in the bigger one.

The last four cycles of the least-squares refinement were also run without applying extinction corrections. It appeared that this brought about only slight changes in the atomic coordinates, in no case larger than the standard deviations. Furthermore, neglect of the extinction corrections caused only moderate changes in the thermal parameters, the scale factor, the standard deviations and the indices R and R_w. The sense of these changes is as expected: compared with the refinement with extinction corrections the thermal parameters and scale factor decreased, the standard deviations in all parameters increased and R increased (1% for I and 4% for II). The change in R_w was less than 1%.

Results and discussion

The final coordinates with their standard deviations calculated by the least-squares program are given in Table 1. It is seen that corresponding coordinates, determined from crystal I and crystal II, are in most cases equal within the standard deviations. The anisotropic temperature factor parameters and their standard deviations are given in Table 2. Here again corresponding values obtained for the two crystals agree within experimental error, indicating that the two sets of F_o values (after correction for extinction) do not show significant differences in their variation with $\sin\theta/\lambda$. Observed and calculated structure factors are compared in Table 3. In this Table the extinction correction is applied to the observed amplitudes F_o.

Table 2. *Parameters* (Å$^2\times10^{-4}$) *of the anisotropic temperature factors* $\exp[-2\pi^2(h^2a^{*2}U_{11}+\ldots+2hka^*b^*U_{12}+\ldots)]$

Standard deviations are in parentheses.

	U_{11}	U_{22}	U_{33}	$2U_{12}$	$2U_{23}$	$2U_{13}$
			Crystal I			
N(1)	257 (94)	345 (99)	929 (160)	0	0	−526 (217)
N(2)	349 (65)	262 (64)	652 (90)	−84 (110)	107 (132)	−262 (131)
P(1)	222 (27)	386 (31)	439 (34)	0	0	−98 (54)
P(2)	268 (19)	290 (20)	496 (25)	64 (34)	1 (38)	−61 (36)
Br(1)	375 (12)	615 (15)	620 (16)	0	0	233 (26)
Br(2)	555 (15)	752 (17)	441 (14)	0	0	−224 (26)
Br(3)	669 (12)	655 (11)	519 (10)	−6 (19)	274 (19)	9 (19)
Br(4)	509 (9)	514 (9)	702 (12)	197 (17)	−216 (20)	271 (19)
			Crystal II			
N(1)	205 (88)	409 (101)	610 (130)	0	0	−439 (184)
N(2)	326 (70)	337 (72)	952 (121)	−88 (123)	−168 (165)	−234 (156)
P(1)	201 (26)	389 (30)	458 (34)	0	0	−131 (52)
P(2)	274 (19)	278 (19)	462 (23)	48 (35)	18 (38)	−79 (35)
Br(1)	381 (13)	592 (15)	608 (16)	0	0	208 (25)
Br(2)	587 (16)	755 (18)	408 (14)	0	0	−155 (25)
Br(3)	692 (12)	629 (11)	526 (10)	9 (20)	289 (20)	38 (19)
Br(4)	506 (10)	513 (10)	728 (12)	230 (17)	−196 (21)	238 (19)

A projection of the crystal structure along the *c* axis is given in Fig. 1. The $(N–P)_3$ rings of the molecules *A* and *C* lie approximately at height $z=0·5$, and those of the molecules *B* and *D* approximately at height $z=0$. The numbering of the atoms in molecule *A* corresponds to that used in the Tables. Fig. 2 shows a

Table 3. *Observed and calculated structure factors*

Crystal I

H	K	L	FO	FC	AC	BC
2	0	0	234	310	303	-66
4	0	0	2893	2759	-2751	-211
6	0	0	566	632	-631	-25
8	0	0	2564	2514	-2501	-258
10	0	0	2113	2035	2024	207
12	0	0	1307	1283	1277	119
14	0	0	697	625	-615	-108
16	0	0	345	362	357	62
4	1	0	754	747	-744	-70
6	1	0	377	326	325	-22
8	1	0	243	229	228	-16
10	1	0	640	630	626	64
12	1	0	347	357	350	69
14	1	0	788	795	-791	-70
16	1	0	371	250	-249	-26
0	2	0	99	173	-156	74
2	2	0	434	442	-442	4
4	2	0	178	166	166	-1
6	2	0	392	368	368	20
8	2	0	968	962	-955	-115
10	2	0	240	276	-273	-44
12	2	0	857	823	815	112
16	2	0	115	144	-144	-13
2	3	0	5445	5594	-5577	-440
4	3	0	155	128	-121	40
6	3	0	1079	1080	-1077	-81
8	3	0	1458	1462	1456	132
10	3	0	1899	1963	1952	201
12	3	0	1165	1194	-1183	-161
14	3	0	144	127	127	13
0	4	0	1877	1846	1836	190
2	4	0	1512	1486	-1483	-99
4	4	0	728	696	-693	-59
6	4	0	1026	1024	1020	90
8	4	0	761	747	-745	-56
10	4	0	119	111	79	78
12	4	0	494	508	-507	-23
14	4	0	520	503	-496	-84
16	4	0	628	566	563	54
2	5	0	386	406	-396	-89
4	5	0	946	936	936	24
6	5	0	618	642	-638	-75
8	5	0	823	816	-815	-33
10	5	0	794	789	776	143
14	5	0	383	276	-271	-52
16	5	0	152	60	-60	-4
0	6	0	3416	3388	-3370	-350
2	6	0	378	324	323	8
4	6	0	1201	1179	1173	116
6	6	0	514	501	497	64
8	6	0	1273	1315	1309	127
10	6	0	521	551	-536	-129
12	6	0	111	38	-9	-37
14	6	0	330	197	193	42
2	7	0	2180	2221	-2211	-216
4	7	0	1004	1052	1041	145
6	7	0	627	633	-631	-53
8	7	0	201	195	194	7
10	7	0	856	870	866	91
12	7	0	796	813	-804	-121
14	7	0	324	294	292	35
0	8	0	553	338	-324	-96
2	8	0	188	188	187	-9
8	8	0	665	659	652	96
10	8	0	165	19	18	7
12	8	0	529	478	-471	-81
2	9	0	911	883	873	130
4	9	0	509	512	508	63
6	9	0	351	354	354	9
8	9	0	480	471	-461	-94
14	9	0	388	355	-354	-24
0	10	0	2819	2862	-2851	-252
2	10	0	256	289	277	81
4	10	0	652	708	705	71
6	10	0	238	250	-243	-62
8	10	0	934	904	897	115
10	10	0	751	755	-750	-86
12	10	0	431	432	-431	-35
2	11	0	397	407	406	32
4	11	0	422	447	443	60
8	11	0	442	415	-414	-32
10	11	0	383	361	-357	-49
12	11	0	151	17	-3	-17
0	12	0	653	631	629	50
2	12	0	435	481	479	47
4	12	0	322	244	-242	-28
6	12	0	861	682	-676	-88
8	12	0	110	27	25	10
10	12	0	279	204	200	39
2	13	0	1097	1033	1026	124
6	13	0	344	357	354	41
8	13	0	334	269	-268	-24
2	14	0	457	429	427	49
6	14	0	533	521	-516	-67
8	14	0	101	99	99	4
2	15	0	191	256	-256	-11
4	15	0	571	589	-586	-60
1	0	1	1105	1036	-1035	47
2	0	1	1501	1438	1436	71
3	0	1	2005	1907	1898	181
4	0	1	4094	4088	4075	324
5	0	1	3239	3162	-3146	-320
6	0	1	1229	1195	-1189	-115
8	0	1	1634	1633	-1625	-164
9	0	1	1074	1083	1075	132
10	0	1	152	214	208	36
11	0	1	923	953	-950	-78
12	0	1	556	553	-550	-63
13	0	1	455	447	437	92
14	0	1	533	456	453	59
15	0	1	316	216	-214	-29
16	0	1	264	310	307	41
0	1	1	180	256	255	17
1	1	1	185	221	221	-10
3	1	1	902	894	893	51
4	1	1	33	47	-41	24
5	1	1	742	726	-725	-37
6	1	1	392	394	-393	-31
7	1	1	1330	1282	-1279	-84
8	1	1	418	467	-463	-60
9	1	1	270	231	226	50
11	1	1	372	271	270	27
12	1	1	282	288	287	29
13	1	1	483	472	472	30
15	1	1	247	157	157	-4
16	1	1	87	120	-120	3
17	1	1	393	369	-366	-47
1	2	1	618	602	602	-12
2	2	1	602	582	-582	-21
3	2	1	855	874	871	69
4	2	1	997	988	982	104
5	2	1	139	157	-157	1
6	2	1	273	271	270	30
7	2	1	832	847	-843	-76
8	2	1	576	560	-557	-58
9	2	1	267	254	-254	-17
11	2	1	107	85	-84	-10
12	2	1	136	218	-215	-43
13	2	1	406	359	357	33
14	2	1	55	135	134	13
15	2	1	275	443	439	53
16	2	1	348	362	358	58
0	3	1	925	899	897	64
1	3	1	1169	1152	1148	88
2	3	1	2244	2322	2315	185
3	3	1	2076	2048	-2037	-215
4	3	1	1014	1025	-1020	-96
5	3	1	686	677	-669	-101
6	3	1	2749	2746	-2735	-253
7	3	1	2292	2277	2264	242
8	3	1	458	505	500	75
9	3	1	728	722	-721	-53
10	3	1	93	131	130	18
12	3	1	234	224	222	26
14	3	1	417	445	441	62
15	3	1	597	573	-567	-79
16	3	1	276	312	-308	-52
1	4	1	265	264	264	-10
2	4	1	301	311	-309	-31
3	4	1	1043	1012	1009	74
4	4	1	638	653	650	64
5	4	1	1538	1590	-1583	-147
6	4	1	582	597	-593	-68
7	4	1	390	420	417	45
8	4	1	195	25	-15	-20
9	4	1	1199	1220	1216	94
10	4	1	587	619	615	72
12	4	1	121	276	-276	-16
13	4	1	177	243	242	21
15	4	1	469	418	-417	-38
16	4	1	139	95	-94	-12
0	5	1	444	410	-409	-22
1	5	1	250	204	199	44
2	5	1	770	759	754	83
3	5	1	902	963	-963	-32
4	5	1	314	287	287	18
5	5	1	1265	1295	-1292	-95
6	5	1	759	804	-796	-119
7	5	1	486	526	525	26
9	5	1	744	758	757	33
11	5	1	383	353	352	26
12	5	1	82	64	62	16
13	5	1	266	198	194	39
14	5	1	496	470	465	66
15	5	1	289	359	-356	-50
1	6	1	1062	1059	-1056	-79
2	6	1	874	871	-867	-83
3	6	1	827	803	-800	-66
4	6	1	1727	1718	-1709	-178
5	6	1	1988	2015	2004	207
6	6	1	482	552	548	67
7	6	1	214	98	97	13
8	6	1	581	610	604	87
9	6	1	881	912	-909	-68
10	6	1	122	5	5	2
11	6	1	405	332	-332	6
12	6	1	311	366	365	31
13	6	1	434	491	-487	-61
15	6	1	451	422	420	42
0	7	1	511	530	-526	-62
1	7	1	1147	1130	1124	112
2	7	1	854	877	874	82
3	7	1	542	557	-551	-81
4	7	1	147	151	-150	-19
5	7	1	199	202	-196	-48
6	7	1	1058	1041	-1035	-104
7	7	1	1328	1338	1328	159
8	7	1	677	640	634	93
9	7	1	849	848	-843	-86
11	7	1	122	208	-207	-16
12	7	1	137	214	-212	-31
13	7	1	230	144	142	27
14	7	1	221	195	193	29
15	7	1	397	331	-329	-43
1	8	1	550	520	-520	-12
3	8	1	477	479	-477	-49
4	8	1	704	771	-765	-102
5	8	1	384	374	373	25
7	8	1	606	591	589	56
8	8	1	395	415	411	53
9	8	1	208	154	154	12
10	8	1	158	122	120	23
11	8	1	185	29	-29	2
12	8	1	194	172	168	34
13	8	1	388	343	-341	-35
14	8	1	266	154	-153	-21
0	9	1	767	769	-764	-91
1	9	1	439	448	447	33
2	9	1	522	510	-508	-51
3	9	1	1105	1103	1097	108
4	9	1	357	384	379	63
5	9	1	115	56	-55	8
6	9	1	488	495	490	68
7	9	1	820	839	-836	-71
9	9	1	338	277	-277	-14
11	9	1	211	105	-103	-19
12	9	1	188	187	-184	-34
13	9	1	475	422	421	38
14	9	1	288	40	-40	-3
1	10	1	793	826	825	31
2	10	1	31	26	5	25
3	10	1	811	742	-735	-102
4	10	1	1098	1139	-1132	-122
5	10	1	775	787	777	126
6	10	1	464	460	457	60
7	10	1	480	374	-373	-31
8	10	1	416	430	427	50
9	10	1	442	438	-433	-70
10	10	1	260	305	-301	-52
11	10	1	636	652	648	68
12	10	1	124	257	255	35
0	11	1	326	325	-323	-40
1	11	1	31	25	20	15
2	11	1	142	213	-210	-34
4	11	1	180	197	197	12
5	11	1	141	287	286	22
6	11	1	446	396	393	49
7	11	1	416	389	388	25
8	11	1	218	152	149	29
11	11	1	280	196	-194	-25
12	11	1	284	224	-222	-31
1	12	1	227	198	192	50
2	12	1	650	604	600	70
3	12	1	310	261	-259	-31
4	12	1	62	168	167	15
5	12	1	218	177	-174	-30
8	12	1	100	60	-59	-10
10	12	1	117	218	-215	-31
11	12	1	226	202	200	29
1	13	1	385	454	-450	-54
2	13	1	462	453	-450	-56
3	13	1	193	163	156	47
4	13	1	144	136	135	18
5	13	1	149	35	26	24
6	13	1	601	561	556	70
7	13	1	429	470	-464	-75
8	13	1	275	197	-193	-41
9	13	1	434	396	394	43
2	14	1	297	282	279	40
4	14	1	184	84	83	10
5	14	1	290	259	257	27
0	15	1	382	407	403	52
1	15	1	206	97	-92	-29
3	15	1	181	138	137	-10
4	15	1	233	180	-179	-22
0	0	2	1594	1585	1584	44
1	0	2	3764	3756	3738	364
2	0	2	283	256	-245	-74
3	0	2	998	955	943	151
4	0	2	244	36	34	10
5	0	2	955	933	-931	-68
6	0	2	442	415	410	63
7	0	2	856	799	795	74
8	0	2	322	252	-250	-28
9	0	2	1953	1945	-1929	-243
10	0	2	700	680	678	53
11	0	2	231	277	276	17
12	0	2	63	38	-13	-36
13	0	2	890	912	903	124
14	0	2	200	231	-198	-38
15	0	2	571	559	-554	-70
1	1	2	80	72	-71	-10
2	1	2	529	550	545	73
3	1	2	1097	1078	1076	59
4	1	2	207	161	-161	-10
5	1	2	353	355	355	3
6	1	2	430	451	451	21
7	1	2	323	337	-336	-32
8	1	2	66	35	-14	-32
9	1	2	807	826	-822	-75
10	1	2	156	100	-97	-25
11	1	2	150	223	-222	-9
12	1	2	317	140	137	32
13	1	2	537	539	534	77
14	1	2	392	280	-280	-17
15	1	2	197	204	204	8
0	2	2	1714	1672	-1669	-109
1	2	2	918	943	941	58
2	2	2	356	350	-350	-11
3	2	2	686	705	705	27
4	2	2	466	459	458	40
5	2	2	361	376	375	23
6	2	2	468	488	486	42
7	2	2	852	861	856	88
8	2	2	386	354	352	33
9	2	2	645	618	-614	-68
10	2	2	405	372	-370	-44
11	2	2	954	986	-980	-106
12	2	2	124	183	-182	-14
13	2	2	195	134	130	33
14	2	2	345	20	-19	5
1	3	2	2784	2823	2810	277
2	3	2	536	361	-361	-17
3	3	2	2505	2555	-2545	-227
4	3	2	694	677	671	90
6	3	2	350	352	-351	-32
7	3	2	821	845	-835	-130
8	3	2	109	139	139	-1
9	3	2	83	106	-102	-30
10	3	2	161	49	48	-9
11	3	2	1478	1535	1522	198
12	3	2	383	372	-368	-52
13	3	2	441	437	-433	-58
14	3	2	259	301	298	43
0	4	2	498	477	470	82
1	4	2	1387	1423	1417	137
2	4	2	646	611	-610	-33
4	4	2	135	103	-103	-12
5	4	2	864	869	-864	-89
6	4	2	228	265	265	17
8	4	2	505	498	-496	-41
9	4	2	617	649	-646	-56
10	4	2	211	42	8	41
11	4	2	620	619	616	62
12	4	2	319	262	-262	-9
13	4	2	600	667	664	55
14	4	2	157	107	-105	-20
15	4	2	278	279	-272	-62
16	4	2	255	264	263	24
1	5	2	1197	1215	1212	93
2	5	2	1269	1274	1271	91
3	5	2	458	489	-486	-52
4	5	2	698	731	730	40
5	5	2	42	134	-130	30
7	5	2	1104	1101	-1099	-71
8	5	2	795	803	-801	-54
9	5	2	1026	1045	-1039	-113
10	5	2	465	500	-499	-35
11	5	2	710	735	731	82
13	5	2	407	441	438	58
14	5	2	245	144	144	11
15	5	2	240	112	-111	-10
0	6	2	295	204	204	-9
1	6	2	1971	1943	-1933	-204
2	6	2	683	646	644	47
3	6	2	1235	1259	-1252	-133
4	6	2	239	149	-148	-15
6	6	2	453	456	-454	-44
7	6	2	285	323	323	15
8	6	2	218	206	206	9
9	6	2	1375	1401	1392	157
10	6	2	149	97	94	-24
11	6	2	230	360	-358	-31
12	6	2	302	400	399	34
13	6	2	651	681	-678	-64
14	6	2	80	84	82	18
1	7	2	852	815	808	103
2	7	2	569	534	-532	-49
3	7	2	1699	1703	-1694	-176
4	7	2	257	289	285	47
5	7	2	723	763	759	76
6	7	2	329	282	-280	-30
7	7	2	196	157	157	-9
8	7	2	185	212	212	16
9	7	2	101	110	-107	-28
10	7	2	209	199	198	17
11	7	2	752	746	739	103
12	7	2	298	270	-267	-42
13	7	2	461	494	-489	-73
14	7	2	88	191	190	25
0	8	2	1099	1098	1092	93
1	8	2	748	760	-757	-67
2	8	2	299	252	252	12
3	8	2	655	655	-654	-44
4	8	2	367	350	-348	-35
5	8	2	333	349	-348	-28
6	8	2	398	386	-384	-35
7	8	2	457	432	-429	-53
8	8	2	321	306	-305	-32
9	8	2	638	630	626	76
10	8	2	298	257	255	32
11	8	2	620	590	587	68
12	8	2	251	222	221	21
13	8	2	141	117	-114	-28
14	8	2	243	27	26	-4
1	9	2	872	907	-901	-107
2	9	2	284	193	-193	-2
3	9	2	445	456	455	31
4	9	2	257	321	-319	-31
5	9	2	786	828	824	79
6	9	2	114	205	205	9
7	9	2	575	582	578	75
8	9	2	274	210	210	3
9	9	2	441	441	-439	-45
10	9	2	117	178	177	14
11	9	2	413	493	-490	-56
0	10	2	788	783	-781	-43
1	10	2	1060	1050	-1039	-154
2	10	2	77	30	-7	30
3	10	2	377	355	355	2
4	10	2	152	78	78	1
5	10	2	715	702	698	74
6	10	2	142	21	12	-17
7	10	2	640	638	-633	-78
9	10	2	523	475	468	83
10	10	2	304	372	-371	-34
11	10	2	190	232	-231	-24
1	11	2	292	279	-277	-34
2	11	2	193	289	-285	-51
3	11	2	378	281	-280	-18
5	11	2	43	23	13	19
6	11	2	342	211	-211	-14
7	11	2	401	365	362	49
8	11	2	221	22	-13	18
9	11	2	248	284	283	28
10	11	2	60	124	122	26
11	11	2	199	36	-32	-17
0	12	2	195	181	181	9
1	12	2	61	25	-24	8
3	12	2	241	302	296	59
5	12	2	184	200	197	36
6	12	2	61	10	-10	1
7	12	2	476	458	-453	-63
8	12	2	157	8	-8	1
9	12	2	141	147	-146	-23
1	13	2	287	336	-331	-62
2	13	2	165	146	146	8
3	13	2	625	639	633	86
5	13	2	272	340	-338	-37
6	13	2	254	103	102	13
8	13	2	166	111	-111	-6
1	14	2	233	162	-160	-22
3	14	2	175	159	156	30
4	14	2	227	24	22	8
5	14	2	266	289	288	44
6	14	2	130	70	-70	-1
2	15	2	121	204	-204	-13
3	15	2	277	166	164	28
1	0	3	1670	1670	-1665	-129
2	0	3	398	410	409	-26
3	0	3	759	758	-755	-64
4	0	3	3053	3092	3080	265
5	0	3	1978	1928	1918	196
6	0	3	1482	1453	-1444	-162
7	0	3	1035	1065	1058	116
8	0	3	998	992	-988	-92
9	0	3	750	763	-759	-74
10	0	3	609	611	603	102
11	0	3	357	367	-367	-19
12	0	3	522	574	-570	-72
13	0	3	431	408	-406	-46
14	0	3	598	524	521	61
15	0	3	119	99	-96	-25
0	1	3	49	147	-146	-19
2	1	3	632	613	-612	-41
3	1	3	760	753	751	60
4	1	3	152	167	156	59
5	1	3	593	592	589	58
6	1	3	221	219	215	43
7	1	3	402	394	-392	-30
8	1	3	520	515	-510	-72
9	1	3	348	397	-396	-28
10	1	3	246	210	210	2
11	1	3	334	347	-344	-40
12	1	3	216	190	190	9
13	1	3	144	96	96	5
14	1	3	233	55	52	19
15	1	3	107	250	248	36
1	2	3	227	257	-255	-32
2	2	3	1262	1270	-1267	-90
3	2	3	802	796	-791	-85
4	2	3	315	339	-339	-7
5	2	3	909	927	922	99
6	2	3	374	389	386	51
7	2	3	611	584	580	67
12	2	3	175	119	-114	-34
13	2	3	319	371	-366	-57
14	2	3	116	66	-64	-14
0	3	3	311	289	-284	-52
1	3	3	383	393	392	34
2	3	3	1621	1716	1709	155
3	3	3	1707	1717	1708	171
4	3	3	700	724	-720	-72
5	3	3	897	895	890	100
6	3	3	1910	1950	-1942	-182
7	3	3	1156	1189	-1182	-127
8	3	3	965	959	948	145
9	3	3	571	581	-577	-66
10	3	3	185	220	-219	-24
12	3	3	155	18	18	-2
14	3	3	198	275	272	39
15	3	3	428	358	354	52
1	4	3	490	479	479	18
2	4	3	363	335	-334	-29
3	4	3	80	85	85	2
4	4	3	979	994	987	115
5	4	3	166	129	-129	-4
6	4	3	732	739	-732	-100
7	4	3	170	148	-147	-15
8	4	3	76	42	30	-30
9	4	3	69	185	-182	-36
10	4	3	666	666	661	83
11	4	3	321	281	280	23
12	4	3	446	494	-493	-33
13	4	3	268	250	249	30
15	4	3	288	102	-102	-14
0	5	3	1394	1381	-1376	-110
1	5	3	471	481	-479	-39
2	5	3	136	108	-108	8
3	5	3	700	707	701	96
4	5	3	874	880	878	69
5	5	3	872	894	888	108
6	5	3	524	515	515	-3
7	5	3	451	456	-449	-77
8	5	3	299	299	299	-1
10	5	3	168	278	-276	-29
11	5	3	263	237	-234	-34
12	5	3	137	242	-241	-19
13	5	3	229	188	-187	-19
14	5	3	343	330	326	47
1	6	3	666	627	619	96
2	6	3	95	121	-119	-18
4	6	3	1098	1121	-1113	-133
5	6	3	893	895	-888	-113
6	6	3	646	647	640	93
7	6	3	561	544	-540	-68
8	6	3	158	73	63	37
9	6	3	201	136	133	29
10	6	3	355	313	-310	-41
11	6	3	187	55	52	20
12	6	3	442	471	470	35
13	6	3	227	170	167	31
14	6	3	125	50	-34	-37
0	7	3	632	638	-631	-90
1	7	3	264	160	-159	-21
2	7	3	1018	1000	995	100
3	7	3	299	205	204	21
4	7	3	224	255	-253	-29
5	7	3	157	189	187	27
6	7	3	1064	1118	-1112	-113
7	7	3	478	480	-477	-53
8	7	3	768	797	787	126
9	7	3	86	28	27	7
10	7	3	159	218	-217	-26
11	7	3	296	233	231	33
12	7	3	112	179	-177	-27
1	8	3	237	291	288	42
2	8	3	748	729	726	56
3	8	3	541	543	539	68
4	8	3	178	219	219	0
5	8	3	715	751	-745	-98
7	8	3	442	422	-418	-59
8	8	3	182	124	-123	-11
11	8	3	124	67	-67	1
13	8	3	236	298	294	52
0	9	3	293	305	-301	-46
1	9	3	349	334	-330	-49
2	9	3	377	386	-384	-38
3	9	3	435	456	-451	-72
4	9	3	138	160	152	51
6	9	3	90	154	149	39
7	9	3	144	113	110	27
8	9	3	210	132	-130	-25
9	9	3	230	143	138	36
10	9	3	209	174	174	6
1	10	3	151	239	239	10
2	10	3	142	46	9	46
3	10	3	292	327	325	37
4	10	3	1155	1191	-1185	-121
5	10	3	511	487	-483	-58
6	10	3	556	596	590	84
7	10	3	267	182	-181	-22
8	10	3	303	348	347	31
9	10	3	341	387	384	44
10	10	3	262	331	-324	-68
0	11	3	118	37	-37	-2
1	11	3	186	54	-54	-2
3	11	3	585	582	-578	-70
5	11	3	337	344	-340	-46
6	11	3	125	42	-38	-17
7	11	3	257	331	329	39
9	11	3	156	241	239	25
1	12	3	316	365	-362	-49
2	12	3	528	529	526	58
3	12	3	43	194	192	30
4	12	3	210	179	178	12
8	12	3	67	40	-39	-8
9	12	3	269	47	46	11
0	13	3	285	173	168	43
2	13	3	387	415	-413	-49
3	13	3	413	279	-277	-35
5	13	3	234	183	-181	-28
6	13	3	503	471	469	50
7	13	3	395	307	304	42
1	14	3	382	326	-325	-34
2	14	3	157	202	200	23
3	14	3	238	51	-50	-5
0	0	4	2864	2880	-2863	-308
1	0	4	209	189	181	52
2	0	4	275	264	263	27
3	0	4	1504	1514	-1510	-110
4	0	4	1119	1108	1101	119
5	0	4	837	875	-872	-76
7	0	4	649	720	716	71
8	0	4	1086	985	979	110
10	0	4	554	464	-457	-82
11	0	4	713	709	704	85
12	0	4	449	395	-389	-67
13	0	4	31	71	-71	4
14	0	4	403	384	378	67
1	1	4	832	839	-834	-89
2	1	4	210	202	-202	-15
3	1	4	814	808	806	63
4	1	4	210	137	-137	0
5	1	4	537	514	513	26
6	1	4	375	315	312	46
7	1	4	504	519	516	53
8	1	4	160	145	145	10
9	1	4	259	273	-272	-20
10	1	4	264	251	-247	-46
11	1	4	665	643	-639	-67
13	1	4	257	140	138	24
14	1	4	294	169	167	20
0	2	4	480	463	-459	-64
1	2	4	606	600	-594	-82
2	2	4	265	286	284	26
3	2	4	422	422	-415	-75
4	2	4	120	129	-128	-10
5	2	4	62	42	41	-10
6	2	4	319	334	-332	-34
7	2	4	230	245	244	29
8	2	4	657	660	653	92
9	2	4	465	450	445	67
10	2	4	155	59	-59	6
11	2	4	199	206	-206	-15
12	2	4	470	451	-447	-59
13	2	4	237	206	-205	-26
14	2	4	216	12	11	-6
1	3	4	387	413	-413	-26
2	3	4	1989	2005	1992	223
3	3	4	755	758	-754	-82
5	3	4	1043	1064	1059	103
6	3	4	307	265	264	17
7	3	4	393	398	398	20
8	3	4	383	382	-379	-51
9	3	4	253	243	242	22
10	3	4	770	772	-766	-98
12	3	4	522	493	487	78
13	3	4	536	597	-591	-87
14	3	4	201	26	-26	3
0	4	4	992	976	-972	-83
1	4	4	840	907	902	95
2	4	4	147	154	151	33
3	4	4	292	308	307	19
4	4	4	493	419	417	47
6	4	4	294	256	-254	-37
7	4	4	191	109	-108	13
8	4	4	147	39	39	1
9	4	4	665	703	-699	-73
10	4	4	137	164	-162	-27
11	4	4	383	379	377	38
13	4	4	445	433	432	32
14	4	4	131	277	272	50
1	5	4	424	468	-461	-85
2	5	4	524	567	564	65
3	5	4	286	217	215	33
4	5	4	475	468	-466	-46
6	5	4	321	374	369	64
7	5	4	136	48	-7	48
8	5	4	61	45	42	15
9	5	4	253	140	-139	-12
10	5	4	671	657	-651	-94
11	5	4	142	193	-188	-45
12	5	4	268	113	112	11
0	6	4	1722	1727	1717	181
1	6	4	256	321	-319	-32
2	6	4	240	218	218	12
3	6	4	254	315	311	51
4	6	4	644	632	-628	-77
5	6	4	240	329	326	40
6	6	4	383	389	-387	-45
8	6	4	404	394	-390	-50
9	6	4	233	212	212	13
10	6	4	376	406	402	62
11	6	4	492	503	-500	-58
13	6	4	304	234	-234	-6
1	7	4	203	138	134	31
2	7	4	815	822	816	99
3	7	4	577	600	-596	-71
4	7	4	545	490	-485	-73
5	7	4	434	485	483	48
6	7	4	249	142	142	6
8	7	4	250	132	131	16
9	7	4	243	102	102	6
10	7	4	229	302	300	-39
11	7	4	313	297	293	45
12	7	4	327	331	326	56
0	8	4	490	475	469	70
1	8	4	396	420	414	68
3	8	4	283	294	287	64
4	8	4	289	8	8	0
6	8	4	124	125	124	14
7	8	4	42	106	-104	-17
8	8	4	417	477	-471	-74
9	8	4	260	306	-300	-56
10	8	4	112	145	145	10
11	8	4	60	83	83	0
12	8	4	249	298	295	42
1	9	4	181	6	0	6
2	9	4	550	600	-596	-73
3	9	4	331	322	321	24
4	9	4	448	507	-504	-57
8	9	4	411	436	432	57
9	9	4	289	230	-229	-23
10	9	4	165	135	135	11
11	9	4	285	162	-162	-7
0	10	4	879	811	803	109
2	10	4	371	332	-329	-45
3	10	4	560	569	567	38
4	10	4	365	279	-276	-37
5	10	4	290	313	312	27
6	10	4	207	324	320	50
7	10	4	375	310	-308	-33
8	10	4	319	365	-362	-46
9	10	4	283	30	17	24
10	10	4	167	88	83	28
1	11	4	493	467	463	65
2	11	4	115	93	-91	-18
3	11	4	366	363	-361	-37
5	11	4	270	290	-289	-23
6	11	4	30	149	-147	-27
7	11	4	280	284	-281	-38
8	11	4	120	59	57	14
0	12	4	195	188	-186	-21
1	12	4	243	27	-27	4
2	12	4	262	271	-268	-38
4	12	4	177	146	145	19
6	12	4	391	412	407	60
7	12	4	109	35	-35	-4
2	13	4	386	364	-360	-56
3	13	4	73	159	157	23
4	13	4	148	191	188	30
1	0	5	383	364	363	23
2	0	5	210	308	-299	-73
3	0	5	609	660	-655	-81
4	0	5	484	433	-425	-85
5	0	5	910	954	944	132
6	0	5	83	126	-125	-15
7	0	5	426	442	-440	-43
8	0	5	471	438	430	83
9	0	5	773	782	-776	-94
11	0	5	435	411	405	67
13	0	5	225	77	-75	-20
0	1	5	209	149	149	7
1	1	5	218	218	-216	-26
2	1	5	465	499	-496	-57
3	1	5	503	439	-435	-61
5	1	5	154	195	195	9
6	1	5	279	317	311	65
7	1	5	597	554	550	65
8	1	5	31	100	-99	-9
10	1	5	199	199	198	14
11	1	5	195	245	-243	-26
13	1	5	205	190	-190	-17
1	2	5	430	418	412	69
2	2	5	236	196	-196	-10
3	2	5	395	443	-440	-52
4	2	5	1039	1040	-1033	-117
5	2	5	303	288	-285	-44
6	2	5	140	140	138	21
8	2	5	474	444	441	54
9	2	5	147	288	287	26
10	2	5	193	131	131	10
11	2	5	243	296	294	35
12	2	5	169	189	187	28
13	2	5	174	139	-137	-17
0	3	5	648	656	-649	-94
1	3	5	575	553	-550	-64
2	3	5	338	314	-310	-45
3	3	5	372	380	375	62
4	3	5	148	158	155	30
6	3	5	548	559	552	92
7	3	5	902	952	-944	-120
8	3	5	269	142	139	29
9	3	5	499	519	515	65
10	3	5	120	279	-275	-43
11	3	5	168	217	216	23
1	4	5	174	55	55	-8
2	4	5	118	171	-171	-1
3	4	5	260	209	-207	-31
4	4	5	226	211	207	43
5	4	5	669	673	668	88
6	4	5	156	52	-49	-16
7	4	5	129	168	-167	-18
8	4	5	244	178	178	4
9	4	5	525	559	-555	-66
10	4	5	68	64	-62	-13
0	5	5	393	329	-328	-15
1	5	5	560	539	-533	-74
2	5	5	679	709	-704	-83
3	5	5	334	245	-240	-50
4	5	5	417	401	400	23
5	5	5	316	345	345	25
6	5	5	1111	1153	1148	115

Table 3 (*cont.*)

Crystal I

H	K	L	FO	FC	AC	BC	H	K	L	FO	FC	AC	BC	H	K	L	FO	FC	AC	BC	H	K	L	FO	FC	AC	BC	H	K	L	FO	FC	AC	BC	H	K	L	FO	FC	AC	BC
9	5	5	109	180	177	35	0	9	5	638	605	601	74	9	0	6	455	400	398	44	7	3	6	134	104	103	11	0	8	6	793	744	-738	-93	7	1	7	367	330	327	42
10	5	5	280	139	-139	-9	1	9	5	184	119	-118	-17	10	0	6	234	256	-253	-42	9	3	6	106	38	37	10	1	8	6	182	203	200	33	1	2	7	137	39	35	17
11	5	5	240	172	-171	-18	2	9	5	172	112	111	17	1	1	6	85	131	-131	-7	10	3	6	178	48	45	17	2	8	6	239	230	-228	-24	2	2	7	473	511	505	73
12	5	5	286	250	-249	-28	3	9	5	82	196	-194	-33	2	1	6	659	629	-623	-86	0	4	6	684	696	-690	-91	3	8	6	167	146	-145	-16	3	2	7	238	190	187	31
2	6	5	654	693	689	74	4	9	5	305	341	-339	-29	4	1	6	249	168	-167	-17	4	4	6	180	184	181	30	4	8	6	188	309	305	44	4	2	7	162	98	97	11
3	6	5	52	127	125	22	6	9	5	397	386	-384	-36	5	1	6	82	68	-61	-30	5	4	6	297	279	277	32	5	8	6	203	43	-42	-5	5	2	7	357	325	-320	-56
4	6	5	602	542	539	60	8	9	5	103	184	-183	-24	6	1	6	135	88	-88	0	7	4	6	80	43	-42	4	6	8	6	391	336	333	44	7	2	7	96	70	-67	-19
5	6	5	581	566	-561	-77	9	9	5	147	43	-43	-4	7	1	6	328	339	337	31	8	4	6	302	269	267	31	1	9	6	219	104	101	25	0	3	7	221	197	194	32
6	6	5	225	20	-19	6	1	10	5	283	273	-272	-26	8	1	6	334	222	218	39	9	4	6	146	189	-188	-19	3	9	6	166	50	50	3	1	3	7	257	247	-245	-31
7	6	5	52	212	211	26	2	10	5	166	224	-224	-7	10	1	6	184	188	186	26	10	4	6	30	155	-149	-42	4	9	6	154	126	124	23	2	3	7	200	148	-147	-17
8	6	5	502	540	-537	-60	3	10	5	275	325	322	49	11	1	6	195	190	-189	-21	1	5	6	188	40	-37	-15	5	9	6	189	109	-103	-35	3	3	7	366	304	-302	-33
9	6	5	418	400	397	47	4	10	5	258	107	-107	7	0	2	6	1003	960	954	108	2	5	6	747	782	-776	-91	0	10	6	391	377	375	39	4	3	7	120	33	31	11
10	6	5	141	182	-180	-26	5	10	5	309	352	-347	-62	1	2	6	209	261	-259	-37	3	5	6	233	199	195	40	1	10	6	140	273	270	35	5	3	7	216	246	-244	-28
1	7	5	520	457	-453	-58	6	10	5	141	57	57	7	2	2	6	211	288	287	28	4	5	6	544	558	-555	-56	2	10	6	201	87	-83	-25	7	3	7	141	42	42	6
2	7	5	240	120	119	12	7	10	5	238	254	252	29	3	2	6	68	209	208	25	5	5	6	551	588	-585	-59	1	0	7	473	467	465	46	1	4	7	284	122	-121	-14
4	7	5	68	144	-143	-13	0	11	5	100	28	26	11	4	2	6	401	417	-413	-54	8	5	6	361	393	390	50	2	0	7	384	247	245	30	3	4	7	185	103	-101	-18
6	7	5	144	79	-79	1	1	11	5	139	84	83	13	6	2	6	429	463	-460	-55	9	5	6	363	330	327	46	4	0	7	307	182	-180	-31	4	4	7	403	307	-305	-33
7	7	5	698	695	-688	-94	2	11	5	327	315	312	46	7	2	6	425	453	-449	-60	1	6	6	259	219	215	41	5	0	7	263	230	-230	-20	5	4	7	336	325	322	44
9	7	5	407	385	382	53	3	11	5	197	159	156	29	8	2	6	365	213	-212	-19	2	6	6	304	242	-239	-39	6	0	7	290	261	256	47	0	5	7	583	557	550	87
1	8	5	178	241	-236	-49	4	11	5	254	16	1	-16	9	2	6	278	215	212	38	3	6	6	122	165	160	42	7	0	7	490	424	-421	-52	1	5	7	97	154	154	14
2	8	5	177	227	227	17	5	11	5	106	24	-23	6	10	2	6	270	193	190	39	6	6	6	295	273	270	39	0	1	7	535	487	483	59	3	5	7	205	136	-130	-42
3	8	5	234	264	262	34	0	0	6	356	420	-419	-25	1	3	6	559	557	-552	-72	8	6	6	105	25	-25	5	1	1	7	60	42	41	10	4	5	7	150	152	-147	-37
4	8	5	856	839	833	105	1	0	6	782	756	-751	-89	2	3	6	245	155	154	9	9	6	6	251	89	-87	-22	2	1	7	198	124	123	12	1	6	7	148	190	-187	-32
5	8	5	250	157	155	28	2	0	6	367	399	393	69	3	3	6	452	450	446	56	2	7	6	388	423	420	53	3	1	7	346	333	-330	-45	2	6	7	236	105	105	3
8	8	5	400	387	-384	-50	3	0	6	474	579	-577	-57	4	3	6	419	477	-469	-84	3	7	6	249	314	312	41	4	1	7	323	298	-296	-36	0	7	7	77	85	83	18
9	8	5	188	192	-191	-19	6	0	6	323	360	-354	-69	5	3	6	322	239	238	23	6	7	6	134	173	172	17	5	1	7	75	133	-131	-24	1	7	7	336	66	-65	-7
10	8	5	169	119	-119	-9	8	0	6	157	167	167	11	6	3	6	150	179	178	15	8	7	6	99	110	-109	-15	6	1	7	315	307	-305	-31							

projection along the b axis of the molecules lying on the mirror plane $y=0{\cdot}25$. All independent intermolecular distances which are nearly equal to or shorter than the sum of the corresponding van der Waals radii are given in the Figures (Pauling, 1960; $r(\mathrm{N})=1{\cdot}5$, $r(\mathrm{P})=1{\cdot}9$, $r(\mathrm{Br})=1{\cdot}95$ Å).

It is interesting to note that in the crystal structure of $N_3P_3Cl_6$ (Wilson & Carroll, 1960) only the intermolecular distances of the type given in Fig. 2 are shorter than the corresponding van der Waals distances, whereas in the crystal structure of $N_3P_3F_6$ (Dougill, 1963) no short intermolecular distances occur.

The short distance N(1)···Br(1), shown in Fig. 2, is part of the approximately linear array N(1)···Br(1)–P(1). Likewise the short distance Br(1)···Br(2) is part of the linear array Br(1)···Br(2)–P(1,*B*). Such linear arrays have been observed in the crystal structures of polyhalogen compounds and have there been interpreted in terms of three-centre four-electron bonding (Wiebenga &

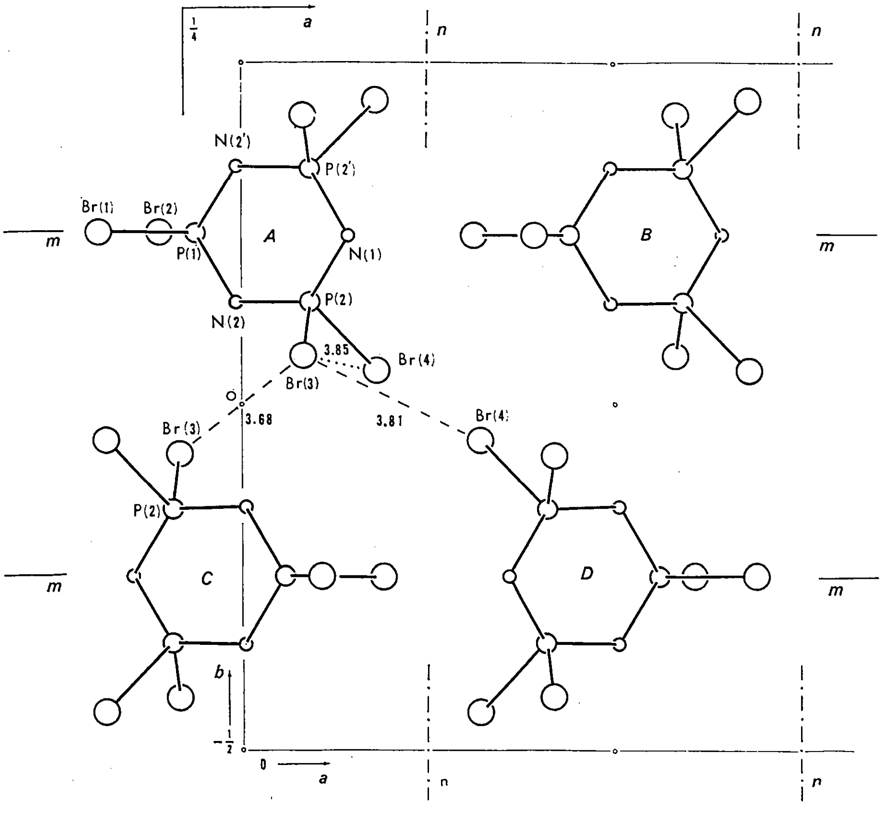

Fig. 1. Projection of the crystal structure along the c axis. The molecules lie on mirror planes at $y=0{\cdot}25$ and $y=0{\cdot}75$. The rings of the molecules *A* and C are approximately at height $z=0{\cdot}5$, and those of *B* and *D* approximately at height $z=0$. The short intermolecular distances are as follows: Br(3,*A*)···Br(3,*C*[$z+1$]), 3·68 Å; Br(3,*A*)···Br(4,*D*[$z+1$]), 3·81 Å; Br(3,*A*)···Br(4,*A*[$z+1$]), 3·85 Å.

Kracht, 1969). A similar type of interaction may be present in the arrays N(1)···Br(1)–P(1) and Br(1)···Br(2)–P(1,*B*) of Fig. 2. It may be noted that these arrays are approximately perpendicular to each other; such a perpendicular orientation has also been observed with the polyhalogens.

As seen in Fig. 1, there is a short intermolecular distance Br(3,*A*)···Br(3,*C*[*z*+1]), which is comparable in length to Br(1)···Br(2), Fig. 2. However, Br(3,*A*)···Br(3,*C*[*z*+1]) is not part of a linear array of the type just considered: the angle P(2,*A*)–Br(3,*A*)–Br(3,*C*[*z*+1]) is 141°. Also, in the crystal structure of $N_4P_4Br_8$ short Br···Br distances (3·66, 3·74 Å) are observed (Zoer & Wagner, to be published), that do not form linear arrays with a phosphorus atom.

The molecule $N_3P_3Br_6$ is shown in Fig. 3 and the bond lengths and valence angles, calculated from the mean atomic coordinates of I and II, are given in Table 4. The standard deviations in this Table were derived from the standard deviations in the mean coordinates $\sigma_m=\frac{1}{2}\{\sigma^2(\mathrm{I})+\sigma^2(\mathrm{II})\}^{1/2}$. For comparison Giglio & Puliti's results have been added to Table 4.

Table 3 (*cont.*)

Crystal II

H	K	L	FO	FC	AC	BC
4	0	0	2774	2762	-2754	-210
6	0	0	516	607	-606	-23
8	0	0	2527	2504	-2490	-257
10	0	0	2030	2007	1996	204
12	0	0	1214	1269	1263	117
14	0	0	585	617	-607	-106
16	0	0	348	375	370	63
2	1	0	64	58	-42	39
4	1	0	726	735	-732	-70
6	1	0	376	345	344	-21
8	1	0	230	232	231	-15
10	1	0	614	605	602	62
12	1	0	315	331	325	66
14	1	0	809	781	-778	-68
16	1	0	287	223	-222	-23
2	2	0	426	442	-442	4
4	2	0	189	159	159	-1
6	2	0	381	378	377	21
8	2	0	981	953	-946	-114
10	2	0	232	269	-266	-43
12	2	0	870	810	802	111
14	2	0	31	35	-31	16
16	2	0	156	145	-144	-12
2	3	0	5649	5589	-5572	-440
4	3	0	155	121	-114	40
6	3	0	1107	1083	-1080	-82
8	3	0	1480	1450	1444	130
10	3	0	1964	1951	1941	199
12	3	0	1192	1183	-1172	-160
14	3	0	147	149	148	15
0	4	0	1886	1879	1869	192
2	4	0	1499	1477	-1474	-99
4	4	0	746	694	-692	-59
6	4	0	1026	1037	1033	91
8	4	0	845	776	-774	-58
10	4	0	23	91	50	75
12	4	0	511	473	-472	-20
14	4	0	386	469	-462	-81
16	4	0	580	568	565	54
2	5	0	417	411	-401	-90
4	5	0	948	920	920	23
6	5	0	661	647	-642	-74
8	5	0	796	826	-825	-34
10	5	0	779	782	769	143
12	5	0	74	106	105	13
14	5	0	294	278	-273	-51
16	5	0	84	50	-50	-1
0	6	0	3434	3379	-3361	-350
2	6	0	348	323	323	8
4	6	0	1215	1159	1153	114
6	6	0	507	507	503	65
8	6	0	1285	1321	1315	127
10	6	0	554	548	-533	-129
14	6	0	211	178	173	41
2	7	0	2234	2256	-2246	-218
4	7	0	1041	1054	1043	146
6	7	0	652	641	-639	-54
8	7	0	169	188	188	5
10	7	0	852	908	903	95
12	7	0	800	796	-787	-119
14	7	0	285	289	287	34
0	8	0	614	369	-356	-97
2	8	0	71	149	149	-12
6	8	0	72	76	-75	12
8	8	0	666	652	645	95
12	8	0	475	473	-467	-79
14	8	0	109	45	43	-11
2	9	0	910	887	877	130
4	9	0	527	521	517	64
6	9	0	337	367	367	11
8	9	0	478	495	-485	-97
10	9	0	160	120	-117	-24
14	9	0	389	371	-370	-26
0	10	0	2754	2878	-2866	-255
2	10	0	303	300	288	81
4	10	0	731	722	719	71
6	10	0	249	238	-230	-62
8	10	0	933	935	928	118
10	10	0	730	752	-747	-86
12	10	0	471	437	-435	-36
2	11	0	357	414	413	32
4	11	0	478	495	491	65
6	11	0	130	100	-98	16
8	11	0	468	450	-448	-36
10	11	0	421	351	-348	-47
0	12	0	665	648	646	52
2	12	0	483	488	485	47
4	12	0	282	241	-239	-28
6	12	0	658	704	-698	-90
10	12	0	106	220	217	41
2	13	0	1031	1045	1038	125
4	13	0	150	183	-175	-54
6	13	0	368	369	367	43
8	13	0	341	274	-273	-25
2	14	0	422	436	433	51
6	14	0	591	561	-557	-72
2	15	0	305	277	-277	-12
4	15	0	556	591	-588	-60
1	0	1	1082	1034	-1033	46
2	0	1	1445	1440	1438	71
3	0	1	1927	1919	1911	180
4	0	1	3978	4095	4082	325
5	0	1	3109	3131	-3114	-318
6	0	1	1196	1189	-1183	-115
7	0	1	98	103	102	-8
8	0	1	1603	1608	-1599	-162
9	0	1	1085	1083	1075	132
10	0	1	105	216	213	36
11	0	1	911	963	-960	-80
12	0	1	516	566	-562	-64
13	0	1	433	435	426	90
14	0	1	487	453	449	59
15	0	1	164	207	-205	-27
16	0	1	293	308	305	40
0	1	1	127	249	249	17
1	1	1	208	227	226	-10
2	1	1	32	18	-9	15
3	1	1	885	894	892	50
5	1	1	735	735	-734	-37
6	1	1	384	384	-383	-30
7	1	1	1300	1274	-1271	-83
8	1	1	449	457	-453	-59
9	1	1	280	240	234	50
10	1	1	222	162	162	7
11	1	1	200	266	264	25
12	1	1	244	271	270	28
13	1	1	461	462	461	29
14	1	1	263	181	179	30
15	1	1	113	159	159	-3
16	1	1	64	124	-124	0
17	1	1	338	365	-362	-45
1	2	1	574	605	605	-12
2	2	1	557	582	-582	-21
3	2	1	846	869	866	69
4	2	1	968	982	976	104
5	2	1	149	167	-167	1
6	2	1	224	267	265	30
7	2	1	799	852	-848	-76
8	2	1	590	561	-558	-58
9	2	1	251	236	-235	-15
11	2	1	165	85	-84	-10
12	2	1	214	211	-207	-40
13	2	1	315	352	351	31
15	2	1	432	443	440	52
16	2	1	379	349	345	56
0	3	1	939	913	911	65
1	3	1	1145	1142	1138	87
2	3	1	2223	2336	2328	186
3	3	1	2024	2044	-2033	-215
4	3	1	1020	1025	-1020	-96
5	3	1	707	667	-660	-100
6	3	1	2713	2733	-2722	-252
7	3	1	2274	2263	2250	241
8	3	1	523	508	502	75
9	3	1	731	739	-737	-54
12	3	1	234	223	222	26
13	3	1	76	66	65	12
14	3	1	455	445	440	62
15	3	1	597	577	-571	-79
16	3	1	343	305	-301	-52
1	4	1	261	249	245	-11
2	4	1	286	319	-318	-32
3	4	1	1040	1012	1009	75
4	4	1	667	678	675	66
5	4	1	1523	1589	-1582	-146
6	4	1	588	589	-585	-67
7	4	1	405	410	408	44
8	4	1	127	33	-25	-21
9	4	1	1214	1223	1219	94
10	4	1	616	605	601	71
12	4	1	295	294	-293	-17
13	4	1	231	227	226	21
15	4	1	385	411	-410	-35
0	5	1	410	390	-390	-20
1	5	1	242	194	189	43
2	5	1	756	764	760	83
3	5	1	943	967	-967	-32
4	5	1	331	290	290	17
5	5	1	1323	1295	-1292	-96
6	5	1	774	800	-791	-119
7	5	1	514	544	543	27
8	5	1	53	93	92	-13
9	5	1	761	768	767	34
10	5	1	79	10	-10	1
11	5	1	326	333	333	24
12	5	1	129	52	50	14
13	5	1	229	197	193	39
14	5	1	431	466	461	65
15	5	1	423	343	-340	-49
16	5	1	172	79	-77	-15
1	6	1	1079	1066	-1063	-79
2	6	1	885	881	-877	-85
3	6	1	843	787	-784	-66
4	6	1	1763	1715	-1706	-178
5	6	1	2015	2025	2014	207
6	6	1	548	558	554	68
7	6	1	110	85	84	12
8	6	1	615	592	586	84
9	6	1	904	904	-902	-66
10	6	1	94	18	18	3
11	6	1	328	325	-325	7
12	6	1	388	381	379	32
13	6	1	460	485	-481	-61
14	6	1	205	174	-169	-43
15	6	1	477	435	433	43
0	7	1	522	539	-535	-63
1	7	1	1143	1145	1139	113
2	7	1	870	887	883	84
3	7	1	543	544	-538	-80
4	7	1	243	161	-160	-18
5	7	1	167	213	-208	-49
6	7	1	1058	1074	-1068	-106
7	7	1	1364	1331	1321	159
8	7	1	619	629	622	92
9	7	1	829	862	-858	-87
10	7	1	129	79	-78	-7
11	7	1	243	215	-215	-16
12	7	1	168	213	-211	-30
13	7	1	307	162	159	30
14	7	1	151	208	206	32
15	7	1	325	337	-334	-43
1	8	1	540	519	-519	-14
2	8	1	168	95	94	-9
3	8	1	495	457	-454	-47
4	8	1	751	778	-771	-102
5	8	1	383	381	380	26
6	8	1	49	54	-53	-10
7	8	1	604	593	590	56
8	8	1	357	411	408	53
10	8	1	74	121	119	23
11	8	1	108	28	-28	1
12	8	1	124	168	164	34
13	8	1	345	335	-333	-34
14	8	1	258	157	-156	-21
0	9	1	773	784	-778	-93
1	9	1	465	452	450	33
2	9	1	563	509	-506	-52
3	9	1	1143	1116	1110	109
4	9	1	395	407	402	64
5	9	1	172	69	-69	6
6	9	1	474	494	490	67
7	9	1	832	836	-833	-71
9	9	1	245	257	-257	-13
12	9	1	277	200	-196	-36
13	9	1	412	432	430	39
14	9	1	299	35	-35	-2
1	10	1	806	817	816	31
2	10	1	170	24	1	24
3	10	1	784	767	-760	-103
4	10	1	1199	1163	-1156	-125
5	10	1	760	774	763	126
6	10	1	469	459	455	60
7	10	1	383	385	-384	-31
8	10	1	446	431	428	50
9	10	1	475	439	-433	-70
10	10	1	251	299	-294	-51
11	10	1	646	661	657	70
12	10	1	290	272	269	37
0	11	1	390	362	-359	-45
1	11	1	100	40	36	18
2	11	1	166	213	-210	-34
4	11	1	180	218	217	15
5	11	1	274	273	272	21
6	11	1	388	401	398	49
7	11	1	417	397	396	26
8	11	1	181	166	163	29
9	11	1	86	89	-84	-28
10	11	1	168	91	-90	-5
11	11	1	160	204	-203	-26
12	11	1	237	233	-230	-33
1	12	1	212	198	192	50
2	12	1	602	615	611	71
3	12	1	252	262	-260	-31
5	12	1	201	178	-175	-32
8	12	1	242	57	-56	-10
11	12	1	198	196	194	29
0	13	1	130	46	43	16
1	13	1	476	457	-454	-54
2	13	1	456	471	-467	-58
3	13	1	129	174	167	48
4	13	1	157	138	136	19
6	13	1	591	558	553	71
7	13	1	430	472	-466	-75
8	13	1	210	202	-198	-41
9	13	1	389	398	396	44
2	14	1	269	309	306	43
3	14	1	225	207	-205	-28
5	14	1	311	271	269	27
6	14	1	90	53	51	13
7	14	1	243	97	-95	-19
0	15	1	405	413	410	52
2	15	1	176	12	11	-5
3	15	1	223	142	142	-11
4	15	1	161	184	-182	-22
5	15	1	271	205	204	16
0	0	2	1677	1553	1552	43
1	0	2	3755	3747	3729	364
2	0	2	251	286	-276	-74
3	0	2	990	942	930	151
4	0	2	161	24	-22	8
5	0	2	949	933	-930	-68
6	0	2	450	416	411	65
7	0	2	829	799	795	75
8	0	2	282	227	-225	-25
9	0	2	1936	1936	-1921	-241
10	0	2	656	656	654	51
11	0	2	275	283	282	18
12	0	2	165	35	-8	-34
13	0	2	899	903	895	123
14	0	2	250	202	-198	-38
15	0	2	542	564	-560	-70
16	0	2	208	99	96	27
1	1	2	137	88	-87	-11
2	1	2	536	528	523	73
3	1	2	1085	1098	1096	60
4	1	2	198	165	-165	-10
5	1	2	371	354	354	3
6	1	2	461	454	453	21
7	1	2	304	330	-328	-30
9	1	2	813	811	-808	-73
11	1	2	228	221	-221	-10
12	1	2	115	123	119	30
13	1	2	484	520	514	74
14	1	2	249	288	-288	-18
15	1	2	49	199	199	7
0	2	2	1817	1676	-1673	-109
1	2	2	921	938	937	57
2	2	2	330	337	-337	-11
3	2	2	719	715	715	27
4	2	2	489	473	472	39
5	2	2	354	364	363	22
6	2	2	494	488	486	41
7	2	2	852	858	853	87
8	2	2	360	353	351	33
9	2	2	612	597	-594	-66
10	2	2	359	358	-355	-43
11	2	2	954	981	-975	-105
12	2	2	273	169	-168	-12
15	2	2	143	164	162	24
16	2	2	131	29	-29	-4
1	3	2	2812	2820	2806	277
2	3	2	423	350	-350	-17
3	3	2	2515	2567	-2556	-227
4	3	2	702	687	681	91
5	3	2	31	21	13	-17
6	3	2	346	327	-326	-29
7	3	2	869	845	-834	-130
8	3	2	235	127	127	-3
10	3	2	223	47	46	-9
11	3	2	1569	1515	1503	196
12	3	2	324	367	-364	-51
13	3	2	445	441	-437	-59
14	3	2	337	292	289	42
15	3	2	166	100	-100	-11
0	4	2	507	480	473	80
1	4	2	1411	1439	1432	139
2	4	2	603	596	-595	-35
3	4	2	38	28	28	0
4	4	2	183	96	-95	-12
5	4	2	868	868	-863	-89
6	4	2	297	289	288	19
7	4	2	150	94	87	35
8	4	2	514	479	-477	-40
9	4	2	670	653	-651	-56
11	4	2	625	608	605	59
12	4	2	329	255	-255	-10
13	4	2	680	674	672	55
14	4	2	108	86	-84	-19
15	4	2	235	271	-264	-60
16	4	2	206	272	271	25
1	5	2	1203	1214	1210	93
2	5	2	1213	1281	1277	92
3	5	2	477	476	-473	-51
4	5	2	732	735	734	40
5	5	2	37	151	-148	28
6	5	2	21	22	-21	4
7	5	2	1106	1096	-1093	-70
8	5	2	800	805	-804	-53
9	5	2	1050	1035	-1029	-112
10	5	2	504	493	-491	-35
11	5	2	712	734	729	82
12	5	2	166	185	184	21
13	5	2	489	424	420	56
14	5	2	213	130	130	8
15	5	2	129	119	-118	-11
0	6	2	269	191	191	-9
1	6	2	2008	1949	-1938	-204
2	6	2	630	641	639	48
3	6	2	1231	1268	-1261	-133
4	6	2	282	157	-156	-14
5	6	2	106	25	25	1
6	6	2	484	487	-485	-45
7	6	2	362	329	329	16
8	6	2	270	190	189	8
9	6	2	1416	1403	1395	158
10	6	2	192	101	98	-23
11	6	2	387	386	-385	-33
12	6	2	461	390	389	34
13	6	2	640	678	-675	-62
14	6	2	150	69	67	18
1	7	2	833	838	831	104
2	7	2	513	526	-524	-48
3	7	2	1700	1700	-1691	-177
4	7	2	260	291	287	48
5	7	2	751	793	789	77
6	7	2	291	269	-267	-28
7	7	2	297	171	171	-10
8	7	2	61	202	201	13
10	7	2	214	198	197	16
11	7	2	774	750	742	103
12	7	2	190	263	-260	-41
13	7	2	485	483	-477	-72
14	7	2	292	186	184	25
0	8	2	1084	1104	1100	94
1	8	2	753	744	-741	-65
2	8	2	224	249	249	13
3	8	2	649	663	-661	-46
4	8	2	339	356	-354	-35
5	8	2	360	356	-355	-29
6	8	2	413	387	-386	-35
7	8	2	359	412	-409	-51
8	8	2	272	305	-303	-33
9	8	2	646	626	622	76
10	8	2	296	252	250	31
11	8	2	556	583	579	66
12	8	2	259	211	210	19
14	8	2	86	32	32	-3
1	9	2	900	926	-920	-107
2	9	2	141	195	-195	-2
3	9	2	471	441	440	31
4	9	2	319	336	-335	-32
5	9	2	805	818	814	79
6	9	2	167	210	209	9
7	9	2	595	588	583	77
8	9	2	86	211	211	3
9	9	2	479	465	-463	-48
10	9	2	138	167	166	13
11	9	2	479	493	-490	-55
13	9	2	86	84	82	17
0	10	2	774	761	-760	-42
1	10	2	1059	1059	-1048	-156
3	10	2	339	356	356	2
4	10	2	195	103	103	2
5	10	2	702	698	694	73
7	10	2	657	652	-647	-79
8	10	2	164	177	176	23
9	10	2	534	483	476	85
10	10	2	378	362	-360	-33
11	10	2	330	229	-228	-23
1	11	2	276	281	-279	-35
2	11	2	256	286	-281	-52
3	11	2	414	306	-305	-21
4	11	2	93	13	12	-3
5	11	2	153	35	27	22
6	11	2	186	213	-213	-14
7	11	2	402	370	367	50
8	11	2	168	22	-14	17
9	11	2	291	265	264	26
10	11	2	142	129	126	26
3	12	2	273	296	290	59
5	12	2	275	200	196	37
7	12	2	483	462	-458	-64
8	12	2	311	19	-19	0
9	12	2	235	151	-149	-24
10	12	2	236	44	44	8
1	13	2	341	334	-328	-63
2	13	2	61	140	140	7
3	13	2	616	647	642	86
4	13	2	118	73	-69	-26
5	13	2	340	328	-326	-37
6	13	2	278	98	97	17
0	14	2	181	151	-145	-42
1	14	2	232	171	-170	-23
2	14	2	149	120	119	7
3	14	2	193	174	171	32
4	14	2	146	16	13	9
5	14	2	288	311	307	47
6	14	2	183	76	-76	-1
1	15	2	113	76	-76	6
2	15	2	160	217	-216	-14
3	15	2	99	157	155	25
1	0	3	1696	1651	-1646	-130
2	0	3	437	441	440	-25
3	0	3	759	726	-723	-64
4	0	3	3037	3063	3052	262
5	0	3	1990	1970	1960	198
6	0	3	1485	1470	-1461	-162
7	0	3	1060	1063	1057	114
8	0	3	1029	986	-982	-92
9	0	3	734	741	-738	-72
10	0	3	630	623	614	103
11	0	3	357	366	-366	-20
12	0	3	585	571	-566	-71
13	0	3	360	409	-407	-47
14	0	3	532	513	510	59
0	1	3	188	117	-115	-19
1	1	3	22	51	-49	-14
2	1	3	642	617	-616	-42
3	1	3	738	743	741	58
4	1	3	147	162	151	60
5	1	3	596	576	574	56
6	1	3	254	231	227	43
7	1	3	388	376	-375	-29
8	1	3	506	500	-495	-72
9	1	3	334	370	-369	-26
10	1	3	103	226	226	5
11	1	3	314	321	-319	-38
12	1	3	214	186	185	9
13	1	3	142	86	86	3
15	1	3	207	230	227	33
1	2	3	261	268	-266	-32
2	2	3	1340	1293	-1290	-90
3	2	3	802	800	-795	-84
4	2	3	378	358	-358	-9
5	2	3	889	895	889	97
6	2	3	411	410	406	53
7	2	3	543	559	555	65
8	2	3	21	74	73	9
12	2	3	101	86	-80	-30
13	2	3	210	354	-350	-55
15	2	3	86	127	-126	-14
0	3	3	251	287	-282	-51
1	3	3	390	396	394	35
2	3	3	1735	1714	1707	153
3	3	3	1621	1734	1725	172
4	3	3	683	713	-710	-73
5	3	3	881	887	881	99
6	3	3	1911	1934	-1926	-180
7	3	3	1161	1186	-1180	-126
8	3	3	958	968	957	145
9	3	3	569	575	-571	-65
10	3	3	167	204	-202	-24
11	3	3	29	27	-27	-4
12	3	3	242	17	16	-3
14	3	3	333	273	270	39
15	3	3	334	359	355	52
1	4	3	519	461	460	18
2	4	3	352	329	-327	-29
3	4	3	150	49	49	-1
4	4	3	910	988	981	115
5	4	3	65	120	-120	-2
6	4	3	724	741	-734	-100
8	4	3	31	49	39	-30
9	4	3	121	184	-181	-35
10	4	3	694	663	658	83
11	4	3	254	264	263	21
12	4	3	461	517	-516	-34
14	4	3	71	140	-139	14
15	4	3	150	102	-102	-13
0	5	3	1484	1380	-1376	-109
1	5	3	520	471	-469	-38
2	5	3	147	142	-142	6
3	5	3	727	700	693	96
4	5	3	767	878	875	70
5	5	3	809	880	874	98
6	5	3	496	527	527	-2
7	5	3	434	440	-433	-76
8	5	3	270	292	292	-7
9	5	3	96	50	-43	-25
10	5	3	301	269	-268	-26
11	5	3	224	243	-241	-34
12	5	3	248	258	-257	-21
13	5	3	121	209	-208	-21
14	5	3	399	316	313	45
1	6	3	715	664	657	98
2	6	3	158	125	-123	-18
4	6	3	774	1103	-1095	-133
5	6	3	714	881	-873	-113
6	6	3	626	670	664	95
7	6	3	484	523	-518	-67
9	6	3	127	130	127	27
10	6	3	267	314	-311	-42
11	6	3	98	74	71	22
12	6	3	473	489	488	35
13	6	3	241	189	186	32
0	7	3	668	654	-647	-91
1	7	3	191	167	-165	-22
2	7	3	1054	990	985	100
3	7	3	251	227	226	23
4	7	3	220	259	-258	-28
5	7	3	199	205	203	28
6	7	3	1105	1136	-1130	-113
7	7	3	457	487	-484	-54
8	7	3	798	784	774	126
9	7	3	174	23	23	7
10	7	3	292	215	-214	-26
11	7	3	303	220	218	30
12	7	3	201	181	-179	-28
13	7	3	212	149	-147	-24
1	8	3	277	306	303	44
2	8	3	789	720	717	55
3	8	3	537	528	524	66
4	8	3	229	228	228	3
5	8	3	468	746	-740	-97
6	8	3	248	152	-150	-25
7	8	3	290	420	-416	-58
8	8	3	268	117	-117	-10
10	8	3	241	108	-108	0
11	8	3	116	79	-79	0
12	8	3	72	52	47	23
13	8	3	289	292	287	51
0	9	3	237	280	-276	-45
1	9	3	337	343	-339	-51
2	9	3	430	378	-376	-39
3	9	3	418	462	-456	-74
4	9	3	120	184	176	53
5	9	3	83	98	-96	-18
6	9	3	168	169	165	39
7	9	3	211	106	103	26
8	9	3	137	127	-124	-26
10	9	3	196	193	193	8
1	10	3	195	241	240	12
2	10	3	260	46	5	46
3	10	3	292	311	309	38
4	10	3	1149	1190	-1183	-121
5	10	3	193	521	-518	-60
6	10	3	581	604	598	84
8	10	3	195	349	348	31
9	10	3	291	375	372	43
10	10	3	365	333	-325	-68
0	11	3	127	66	-66	-4
1	11	3	86	81	-81	-5
2	11	3	250	177	176	14
3	11	3	601	603	-599	-72
4	11	3	34	92	91	-12
5	11	3	360	341	-338	-45
7	11	3	295	320	318	38
8	11	3	216	132	129	28
9	11	3	156	237	236	25
1	12	3	357	363	-360	-50
2	12	3	550	542	538	58
3	12	3	215	213	211	31
4	12	3	200	202	202	14
5	12	3	133	115	114	16
6	12	3	112	70	-68	-16
7	12	3	140	115	113	17
1	13	3	180	36	35	9
2	13	3	397	426	-423	-49
3	13	3	221	295	-293	-38
4	13	3	123	131	130	16
5	13	3	78	183	-181	-28
6	13	3	475	452	449	49
7	13	3	294	309	306	42
1	14	3	402	342	-340	-37
2	14	3	91	214	212	25
0	0	4	3180	2909	-2892	-308
1	0	4	101	171	163	52
2	0	4	239	242	240	27
3	0	4	1472	1517	-1513	-109
4	0	4	1041	1088	1082	119
5	0	4	870	886	-882	-79
6	0	4	142	32	32	2
7	0	4	647	721	718	70
8	0	4	962	960	954	109
9	0	4	211	143	143	-11
10	0	4	492	473	-466	-84
11	0	4	663	697	692	83
12	0	4	390	384	-379	-65
13	0	4	195	70	-70	4
14	0	4	342	362	356	66
1	1	4	879	877	-873	-90
2	1	4	150	229	-228	-18
3	1	4	797	811	809	64
4	1	4	183	146	-146	0
5	1	4	521	500	500	25
6	1	4	306	287	284	44
7	1	4	480	486	483	51
9	1	4	292	281	-280	-20
10	1	4	228	237	-233	-43
11	1	4	617	621	-618	-63
13	1	4	275	142	139	25
0	2	4	473	396	-391	-59
1	2	4	617	601	-595	-83
2	2	4	306	292	291	26
3	2	4	372	406	-399	-74
4	2	4	125	128	-128	-11
6	2	4	348	328	-327	-35
7	2	4	141	227	225	27

Table 3 (*cont.*)

Crystal II

H	K	L	FO	FC	AC	BC
8	2	4	641	637	631	88
9	2	4	420	444	439	66
11	2	4	238	222	-221	-17
12	2	4	423	437	-433	-57
13	2	4	97	184	-183	-23
1	3	4	483	413	-412	-26
2	3	4	2036	1994	1981	223
3	3	4	769	790	-786	-83
5	3	4	1018	1046	1041	102
6	3	4	267	269	269	17
7	3	4	404	428	427	24
8	3	4	367	402	-399	-54
9	3	4	207	227	226	21
10	3	4	750	760	-754	-96
12	3	4	510	495	489	78
13	3	4	584	585	-579	-85
0	4	4	1056	1002	-998	-86
1	4	4	916	898	893	94
2	4	4	255	169	166	33
3	4	4	234	308	307	19
4	4	4	557	442	439	47
6	4	4	235	244	-241	-38
7	4	4	152	113	-113	13
8	4	4	143	56	56	3
9	4	4	637	656	-652	-69
10	4	4	45	162	-160	-27
11	4	4	402	385	383	36
13	4	4	454	426	425	30
14	4	4	244	268	264	48
1	5	4	541	462	-454	-86
2	5	4	557	549	546	51
3	5	4	122	248	245	34
4	5	4	453	456	-454	-45
6	5	4	370	351	346	62
7	5	4	177	48	2	48
9	5	4	127	117	-117	-12
10	5	4	636	619	-613	-90
11	5	4	86	155	-150	-43
0	6	4	1763	1715	1706	182
1	6	4	394	333	-331	-33
2	6	4	77	198	197	12
3	6	4	360	339	334	52
4	6	4	654	658	-653	-77
5	6	4	365	334	331	42
6	6	4	403	419	-416	-47
7	6	4	134	28	17	-22
8	6	4	417	397	-394	-49
9	6	4	197	213	212	12
10	6	4	405	438	433	64
11	6	4	556	509	-505	-58
13	6	4	145	243	-243	-6
2	7	4	666	625	619	100
3	7	4	572	603	-599	-71
4	7	4	565	489	-484	-73
5	7	4	492	494	492	48
6	7	4	217	150	150	7
8	7	4	124	117	116	15
10	7	4	342	316	-314	-40
11	7	4	283	268	265	42
12	7	4	311	325	321	56
0	8	4	397	453	448	66
1	8	4	405	429	424	70
3	8	4	324	302	295	65
4	8	4	96	17	17	0
5	8	4	118	22	-20	10
6	8	4	48	115	115	13
7	8	4	149	94	-92	-16
8	8	4	436	450	-444	-71
9	8	4	106	308	-303	-56
10	8	4	148	155	155	10
11	8	4	237	92	92	1
12	8	4	269	293	290	40
2	9	4	611	613	-609	-74
3	9	4	331	329	328	27
4	9	4	481	520	-516	-59
5	9	4	147	55	51	-20
6	9	4	172	36	34	11
7	9	4	201	17	16	-5
8	9	4	464	455	451	60
9	9	4	266	241	-240	-23
10	9	4	91	129	129	11
11	9	4	165	169	-169	-7
0	10	4	866	825	818	109
1	10	4	156	111	-104	-39
2	10	4	296	318	-315	-45
3	10	4	628	588	586	40
4	10	4	316	262	-259	-36
5	10	4	333	322	320	29
6	10	4	290	330	327	49
7	10	4	385	325	-323	-34
8	10	4	348	363	-359	-47
10	10	4	201	93	88	29
1	11	4	519	500	495	67
2	11	4	117	83	-81	-17
3	11	4	373	360	-358	-37
5	11	4	242	300	-299	-24
7	11	4	322	288	-286	-39
0	12	4	178	224	-223	-24
2	12	4	254	267	-264	-38
3	12	4	21	154	-154	-9
4	12	4	152	140	138	20
5	12	4	99	116	-116	-11
6	12	4	445	409	404	61
1	13	4	84	159	159	9
2	13	4	397	358	-353	-56
4	13	4	180	186	183	35
1	0	5	352	374	373	21
2	0	5	310	299	-290	-73
3	0	5	633	635	-630	-79
4	0	5	496	435	-427	-85
5	0	5	935	964	954	133
6	0	5	147	133	-133	-14
7	0	5	440	432	-430	-44
8	0	5	386	410	402	81
9	0	5	763	788	-782	-95
11	0	5	417	391	386	64
1	1	5	249	225	-223	-26
2	1	5	474	459	-455	-55
3	1	5	458	469	-465	-64
4	1	5	84	93	-93	12
5	1	5	197	207	207	9
6	1	5	301	304	297	62
7	1	5	548	568	564	66
8	1	5	115	95	-95	-10
10	1	5	200	219	218	15
11	1	5	118	218	-216	-23
12	1	5	178	65	-61	-23
13	1	5	179	174	-174	-16
1	2	5	390	413	407	70
2	2	5	72	204	-203	-10
3	2	5	389	419	-416	-50
4	2	5	981	1013	-1007	-114
5	2	5	374	325	-321	-49
6	2	5	20	158	156	22
8	2	5	451	422	419	51
9	2	5	218	278	277	25
10	2	5	184	121	121	8
11	2	5	300	286	284	33
12	2	5	213	194	192	29
0	3	5	708	661	-654	-95
1	3	5	544	526	-522	-62
2	3	5	301	308	-304	-45
3	3	5	366	397	392	63
5	3	5	138	50	-48	15
6	3	5	577	553	545	91
7	3	5	950	959	-951	-121
8	3	5	197	154	151	29
9	3	5	510	525	521	65
10	3	5	251	258	-255	-42
11	3	5	271	222	221	24
1	4	5	155	49	48	-9
2	4	5	184	176	-176	-2
3	4	5	275	225	-223	-31
4	4	5	109	181	177	39
5	4	5	670	674	668	89
7	4	5	183	163	-162	-19
8	4	5	220	199	199	5
9	4	5	513	554	-550	-65
12	4	5	247	264	-263	-23
0	5	5	358	361	-361	-16
1	5	5	492	536	-531	-72
2	5	5	735	725	-721	-82
3	5	5	220	296	-291	-54
4	5	5	393	397	397	25
5	5	5	289	340	339	23
6	5	5	1142	1124	1119	112
7	5	5	63	182	181	20
10	5	5	205	153	-153	-7
11	5	5	252	161	-160	-15
12	5	5	223	257	-256	-27
1	6	5	257	23	-21	10
2	6	5	666	717	713	76
4	6	5	567	552	549	60
5	6	5	609	568	-563	-79
7	6	5	215	219	217	27
8	6	5	520	548	-545	-60
9	6	5	419	412	409	47
10	6	5	203	197	-195	-28
11	6	5	127	41	-36	-20
0	7	5	94	71	71	1
1	7	5	471	446	-442	-57
2	7	5	283	114	113	11
3	7	5	115	204	200	39
4	7	5	201	138	-138	-12
7	7	5	663	684	-678	-93
8	7	5	131	30	-30	1
9	7	5	413	402	398	53
1	8	5	269	258	-253	-50
2	8	5	142	243	242	19
3	8	5	181	231	229	31
4	8	5	823	821	814	103
8	8	5	387	376	-373	-48
0	9	5	649	656	652	77
1	9	5	37	119	-118	-16
2	9	5	220	131	130	17
3	9	5	281	213	-210	-35
4	9	5	369	338	-336	-30
5	9	5	205	175	174	14
6	9	5	413	376	-374	-36
7	9	5	209	243	241	31
8	9	5	237	182	-180	-25
9	9	5	46	55	-55	-5
1	10	5	298	287	-285	-26
2	10	5	215	227	-227	-6
3	10	5	298	316	312	48
4	10	5	270	104	-104	7
5	10	5	341	350	-345	-61
7	10	5	336	252	250	31
0	11	5	171	23	19	13
1	11	5	89	84	83	12
2	11	5	294	297	293	45
3	11	5	234	177	174	31
4	11	5	217	20	-10	-17
2	12	5	218	216	-212	-41
0	0	6	394	452	-452	-27
1	0	6	735	756	-751	-88
2	0	6	381	396	390	69
3	0	6	571	591	-588	-58
5	0	6	222	180	177	30
6	0	6	307	363	-356	-68
8	0	6	184	147	147	9
9	0	6	447	424	421	45
10	0	6	299	240	-237	-40
1	1	6	197	135	-135	-4
2	1	6	624	626	-620	-87
4	1	6	127	158	-158	-15
5	1	6	65	81	-75	-30
6	1	6	136	96	-96	0
7	1	6	240	310	308	28
8	1	6	269	222	219	39
9	1	6	139	159	157	25
10	1	6	161	172	170	24
11	1	6	137	189	-188	-19
0	2	6	1074	1002	996	112
1	2	6	127	234	-232	-33
2	2	6	207	274	272	26
3	2	6	269	225	224	27
4	2	6	406	428	-424	-54
5	2	6	174	53	52	7
6	2	6	431	447	-444	-54
7	2	6	393	448	-444	-59
8	2	6	161	209	-208	-20
9	2	6	94	179	176	34
10	2	6	237	195	191	39
1	3	6	598	568	-563	-72
2	3	6	58	158	158	12
3	3	6	510	444	440	55
4	3	6	476	476	-468	-84
5	3	6	235	257	255	26
6	3	6	181	167	166	12
7	3	6	161	105	104	12
9	3	6	57	6	1	6
10	3	6	160	46	42	17
0	4	6	702	709	-703	-92
3	4	6	75	124	124	-6
5	4	6	259	267	265	31
7	4	6	102	52	-52	4
8	4	6	186	247	246	29
10	4	6	205	142	-136	-41
2	5	6	782	792	-786	-93
3	5	6	139	196	192	38
4	5	6	547	548	-545	-54
5	5	6	551	583	-580	-59
7	5	6	168	29	24	17
8	5	6	443	416	413	51
9	5	6	369	341	338	44
1	6	6	218	213	209	41
2	6	6	205	266	-263	-40
3	6	6	251	165	159	43
4	6	6	108	50	49	5
5	6	6	210	77	-77	-5
6	6	6	312	288	286	41
9	6	6	100	92	-90	-23
1	7	6	208	138	-136	-19
2	7	6	417	419	416	53
3	7	6	320	309	306	41
4	7	6	164	130	-125	-34
5	7	6	60	17	-15	-9
6	7	6	187	161	160	16
8	7	6	153	119	-118	-14
0	8	6	769	759	-753	-96
1	8	6	232	181	179	30
2	8	6	198	213	-211	-23
3	8	6	152	153	-152	-17
4	8	6	349	323	320	45
6	8	6	353	337	334	44
2	9	6	178	43	43	3
4	9	6	134	142	140	26
5	9	6	193	125	-119	-36
0	10	6	368	386	384	39
1	10	6	278	270	268	34
1	0	7	480	470	467	45
2	0	7	202	222	220	27
3	0	7	178	27	-27	-5
4	0	7	243	175	-172	-30
5	0	7	181	238	-237	-20
6	0	7	351	267	262	48
7	0	7	434	420	-417	-51
0	1	7	591	508	504	60
1	1	7	78	32	31	10
3	1	7	290	340	-338	-45
4	1	7	264	298	-295	-39
6	1	7	361	315	-314	-33
7	1	7	336	329	326	42
2	2	7	446	521	516	74
3	2	7	213	189	187	29
5	2	7	371	321	-316	-55
7	2	7	107	50	-47	-15
0	3	7	163	190	187	29
1	3	7	300	246	-244	-32
2	3	7	242	142	-141	-17
3	3	7	352	298	-296	-33
4	3	7	134	45	43	14
5	3	7	228	241	-239	-27
6	3	7	111	136	134	21
1	4	7	200	122	-121	-14
4	4	7	393	312	-311	-34
5	4	7	355	331	328	43
6	4	7	258	185	183	30
0	5	7	518	526	519	86
1	5	7	68	153	152	14
2	5	7	196	42	42	4
3	5	7	215	132	-126	-41
4	5	7	129	164	-159	-39
1	6	7	200	186	-183	-33
4	6	7	152	158	157	13

The P–Br bonds (mean value 2·162 Å) are equal within experimental error, as well as the angles Br–P–Br (mean 102·1°). Within the (N–P)$_3$ ring no significant differences are found between the P–N bonds (mean 1·576 Å), or between the angles N–P–N (mean 118·5°). However, there is a small, but significant, difference (3°) between the two independent angles P–N–P (119·3 and 122·4°). The smaller value of the angle P(2)–N(1)–P(2′), as compared with the angle P(2)–N(2)–P(1), together with the differences in the P–N bonds and the angles N–P–N (though the latter differences are not significant) describe a slight deformation of the six-membered ring of a type consistent with the bonding interaction, discussed above, between the nitrogen atom N(1) and the group P(1)–Br(1) of a second molecule

The (N–P)$_3$ ring is not entirely planar. The dihedral angles assignable to the P–N bonds are: $-6·7°$ for N(1)–P(2), 6·3° for P(2)–N(2) and $-5·5°$ for N(2)–P(1), indicating a slight chair-shaped conformation. The best plane through the six ring atoms, the phosphorus atoms having a weight four times that of the nitrogen atoms, is given by the equation

$$-0·3958\ X+0·9183\ Z=2·836 \quad (X \text{ and } Z \text{ in Å}).$$

The distances from the ring atoms to this plane and their standard deviations are ΔN(1) = $-0·047$ (0·011), ΔP(2) = 0·011 (0·003), ΔN(2) = $-0·035$ (0·008) and ΔP(1) = 0·008 (0·004) Å. The χ^2 test (Cruickshank & Robertson, 1953) on the planarity of the ring yielded $\chi^2 = 9990$ and $P < 0·001$ for $n = 6$.

In the crystal structures of the related compounds $N_3P_3Cl_6$, $N_3P_3Cl_5F$ (Olthof, 1969) and $N_3P_3F_6$ the (N–P)$_3$ ring has been found to be planar within experimental error. The departure from planarity in the case of $N_3P_3Br_6$ is most likely due to intermolecular steric interactions, though the non-planarity is not apparent from the short intermolecular contacts, presented in Fig. 1 and 2.

The authors are indebted to Professor Aafje Vos for valuable discussions and to Mr F. van Bolhuis for assistance in operating the Nonius diffractometer.

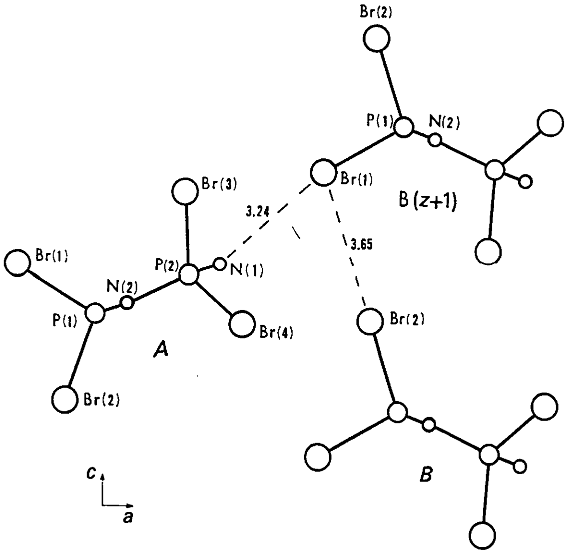

Fig. 2. Projection along the *b* axis of the molecules lying on the mirror plane $y=0·25$ and short intermolecular distances in this plane.

Table 4. *Bond lengths and valence angles with standard deviations*

	Present work		Giglio & Puliti (1967)	
N(1)-P(2)	1·588 Å	0·006 Å	1·55 Å	0·035 Å
N(2)-P(1)	1·565	0·008	1·60	0·033
N(2)-P(2)	1·575	0·008	1·60	0·045
P(1)-Br(1)	2·165	0·004	2·20	0·011
P(1)-Br(2)	2·158	0·004	2·17	0·017
P(2)-Br(3)	2·168	0·003	2·20	0·019
P(2)-Br(4)	2·159	0·003	2·14	0·013
P(2)—N(1)-P(2′)	119·3°	0·6°	126·8°	2·6°
P(2)—N(2)-P(1)	122·4	0·5	118·6	2·4
N(2)-P(1)-N(2′)	117·4	0·6	118·3	1·8
N(1)-P(2)-N(2)	119·0	0·4	115·8	2·0
Br(1)-P(1)-Br(2)	102·1	0·1	101·7	0·4
Br(3)-P(2)-Br(4)	102·0	0·1	103·4	0·4
N(1)-P(2)-Br(3)	106·9	0·5	106·7	2·4
N(1)-P(2)-Br(4)	108·7	0·4	111·9	1·7
N(2)-P(2)-Br(3)	109·3	0·4	114·4	1·8
N(2)-P(2)-Br(4)	109·7	0·4	104·0	1·8
N(2)-P(1)-Br(1)	109·1	0·4	113·4	1·7
N(2)-P(1)-Br(2)	109·0	0·4	103·8	2·1

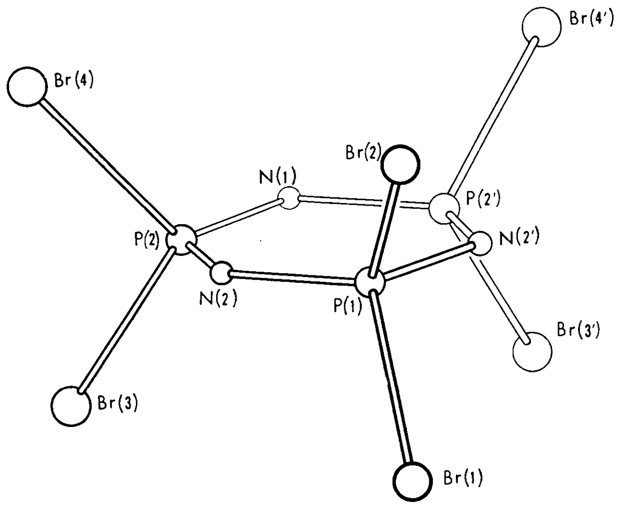

Fig. 3. Molecule $N_3P_3Br_6$.

References

Busing, W. R. & Levy, H. A. (1957). *Acta Cryst.* **10**, 180.

Coxon, G. E., Sowerby, D. B. & Tranter, G. C. (1965). *J. Chem. Soc.* p. 5697.

Cruickshank, D. W. J. & Robertson, A. P. (1953). *Acta Cryst.* **6**, 698.

Cruickshank, D. W. J. (1961). *Computing Methods and the Phase Problem in X-ray Crystal Analysis*. Oxford: Pergamon Press.

Dougill, M. W. (1963). *J. Chem. Soc.* p. 3211.

Doyle, P. A. & Turner, P. S. (1968). *Acta Cryst.* A**24**, 390.

Giglio, E. & Puliti, R. (1967). *Acta Cryst.* **22**, 304.

Hazekamp, R., Migchelsen, T. & Vos, A. (1962). *Acta Cryst.* **15**, 539.

International Tables for X-ray Crystallography (1962). Vol. III. Birmingham: Kynoch Press.

McGeachin, H. McD. & Tromans, F. R. (1961). *J. Chem. Soc.* p. 4777.

Olthof, R. (1969). *Acta Cryst.* B**25**, 2040.

Pauling, L. (1960). *The Nature of the Chemical Bond.* Ithaca: Cornell Univ. Press.

Schlueter, A. W. & Jacobson, R. A. (1968). *J. Chem. Soc.* A, p. 2317.

Wagner, A. J. & Vos, A. (1968). *Acta Cryst.* B**24**, 707.

Wiebenga, E. H. & Kracht, D. (1969). *Inorg. Chem.* **8**, 738.

Wilson, A. & Carroll, D. F. (1960). *J. Chem. Soc.* p. 2548.

Zachariasen, W. H. (1967). *Acta Cryst.* **23**, 558.

Zachariasen, W. H. (1968). *Acta Cryst.* A**24**, 212.