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The Crystal Structure of 4-Acetyl-2′-fluorobiphenyl

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The crystal structure of 4-acetyl-2′-fluorobiphenyl, $C_{14}H_{11}OF$, has been determined from three-dimensional X-ray diffraction data by a Patterson interpretation method. The crystals are monoclinic, space group $P2_1/c$, with unit-cell dimensions $a=13·687$, $b=5·971$, $c=14·766$ Å, $\beta=116°10'$. The structure was refined with use of anisotropic thermal parameters to an R value of 0·094 for the 1280 observed structure factors. The angle between the two phenyl rings was found to be 50·5°. All the intermolecular contacts correspond to normal van der Waals interactions.

Introduction

Recent studies of the liquid-crystalline properties of biphenyl derivatives (Branch, Byron, Gray, Ibbotson & Worrall, 1964) have created interest in the interplanar angles for both sterically affected and non-sterically affected biphenyl nuclei in the liquid crystalline states. Residual crystal forces must operate upon the molecules in a liquid crystal, and interplanar angles derived from studies involving solutions or the vapour phases of biphenyl derivatives are probably not relevant to the liquid-crystalline states. Thus the molecule of biphenyl is planar in the crystal (Dhar, 1932; Trotter, 1961; Hargreaves & Rizvi, 1962), but has an interplanar angle of about 45° in solution (Brande & Forbes, 1955) or in the vapour phase (Bastiansen, 1949). To help to assess more exactly the marked changes in liquid-crystalline transition temperatures which occur on introducing a simple substituent into the 2 or 2′ position of a suitable biphenyl derivative, it seemed of value to establish interplanar angles and details of molecular structure for the crystalline states of some related, but less complex, systems. Since the simple 2-halogenobiphenyls are liquids, the higher melting 4-acetyl-2′-halogenobiphenyls and 2′-halogenobiphenyl-4-carboxylic acids were selected for study (Gray, Sutherland & Young, 1965). This paper describes an X-ray crystallographic study of 4-acetyl-2′-fluorobiphenyl, the molecular formula for which is shown in Fig. 1, along with the numbering system used.

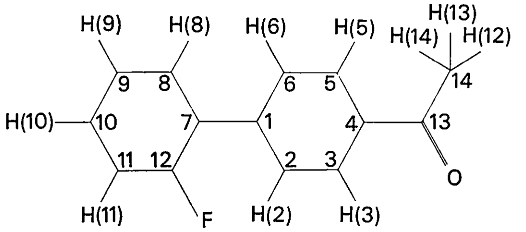

Fig. 1. The molecular formula of 4-acetyl-2′-fluorobiphenyl and the numbering adopted throughout the paper.

The structure was also of interest as it gave an opportunity of applying the methods developed by Tollin & Cochran (1964) to a structure containing more than one planar group in the asymmetric unit.

Experimental

The crystals of 4-acetyl-2′-fluorobiphenyl, produced by slow evaporation from ethanol, were well formed bladed needles, elongated along **b**, the main face of the blade being the (101) plane. On examination under the polarizing microscope many of the larger crystals were found to be twinned. The space group and unit-cell dimensions were obtained from Weissenberg and precession photographs with Cu $K\alpha$ radiation. The systematic absences, $0k0$ with $k=2n+1$ and $h0l$ with $l=2n+1$, uniquely determined the space group as $P2_1/c$. The unit-cell dimensions were

$$a=13·687\pm0·005,\quad b=5·971\pm0·003,\quad c=14·766\pm0·005\ \text{Å};$$
$$\beta=116°10'\pm5'$$
$$(\lambda\ \text{Cu}\ K\alpha_1=1·54050\ \text{Å};\ \lambda\ \text{Cu}\ K\alpha_2=1·54434\ \text{Å})\,.$$

The density measured at 17°C by the method of flotation with aqueous cadmium n-dodecatungstaborate was $1·31\pm0·01$ g.cm^{-3}. The calculated value, assuming four molecules per unit cell, was 1·313 g.cm^{-3}.

In an attempt to minimize absorption effects the crystals were cut into needles of approximately square cross section. The same single crystal was used to collect the $h0l$ to $h5l$ layers by the multiple-film equi-inclination Weissenberg method with Cu $K\alpha$ radiation and the $hk0$, $0kl$, hkh, $h,k,2h$, $hk\bar{h}$ and $h,k,2\bar{h}$ spectra on a precession camera using timed exposures with Cu $K\alpha$ and Mo $K\alpha$ radiation. The intensity of every spot on all films was estimated visually by comparison with a calibrated scale and corrected for Lorentz and polarization factors. Corrections for spot shape were applied to the upper level Weissenberg data: the Phillips (1954) correction factor to the extended reflexions and an empirical correction to the contracted reflexions obtained by a comparison of the intensity of the contracted reflexion with that of the corrected extended reflexion, for those reflexions common to both sides of the film. The intensities from the precession films, scaled to the *h*0*l* data, were used to increase the number of such comparisons at low values of sin θ. After the data were brought on to the same arbitrary scale, using the common reflexions and timed exposures, and before averaging, the agreement between different observations of the same structure factor was of the order of 10%.

Structure determination

The function $I(\theta,\varphi)$ (Tollin & Cochran, 1964) was computed from the 50 largest sharpened Patterson coefficients, $|F_s(\mathbf{h})|^2$, and $R=3\cdot4$ Å and plotted on a Sanson–Flamsteed projection (Fig. 2). The projection contained only one large peak which had a little fine structure suggesting that there might be two very close peaks which overlap. The $I(\theta,\varphi)$ map was recomputed in the region of this peak with the use of all the $|F_s|^2$ data. This showed a single peak from which the fine structure had disappeared. The angular coordinates of the peak were $\theta=27\cdot5°$ and $\varphi=100°$ where θ and φ are spherical polar angles referred to the orthogonal triad $\mathbf{a}^*,\mathbf{b}^*,\mathbf{c}$.

This single peak can be interpreted as indicating that either the whole molecule is essentially planar, or that the angle between the two planar parts of the molecule is such that the peak due to one part overlaps the peak due to the second part in a symmetry related molecule. This would occur if the normals to the two planar parts were related by the equations $\theta_1=\theta_2$ and $\varphi_1=-\varphi_2$. These two possibilities were denoted as the parallel and anti-parallel configurations.

The section through the sharpened Patterson function, computed in the orientation given by these θ and φ values, is shown in Fig. 3. The line X is the intersection of the molecular plane with the (010) plane.

Theoretical vector sets, constructed for both the parallel and anti-parallel configurations of the biphenyl moiety and the atom C(13), were superimposed on the Patterson section and rotated until the best fit was obtained. It was impossible to distinguish between the two configurations; both gave the best fit when oriented so that the long axis of the molecule lay along the line L in Fig. 3. The orientation of the acetyl group and the position of the fluorine atom could not be identified at this stage.

To distinguish between the parallel and antiparallel configurations and determine the position of the molecule in the unit cell, the method of Q-functions (Tollin, 1966) was used. The function $Q(Y_0)$ which determines the position of the molecule relative to the c-glide was

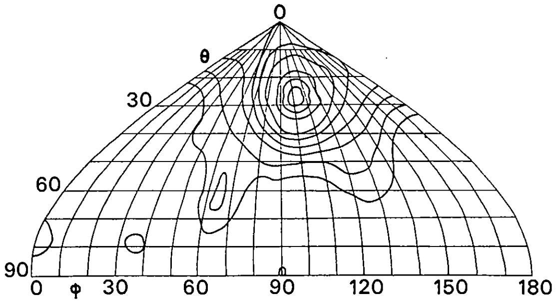

Fig. 2. Plot of the $I(\theta,\varphi)$ function.

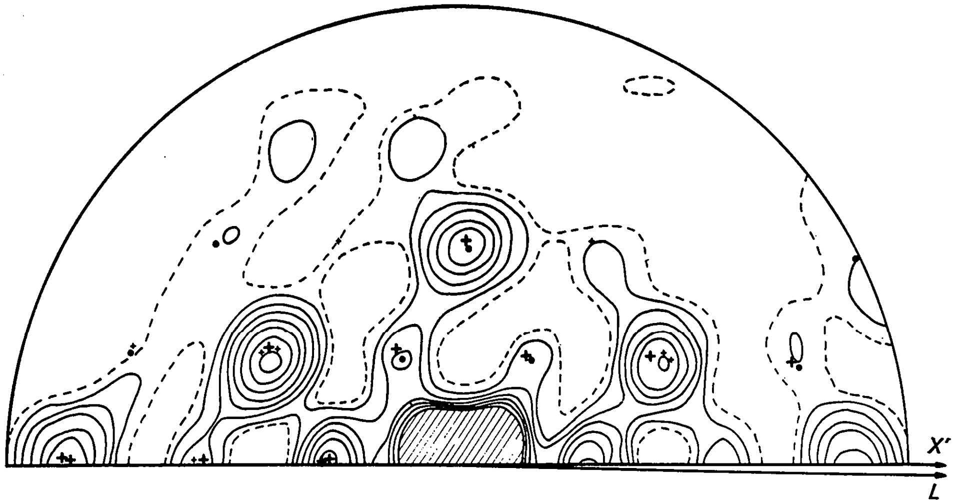

Fig. 3. Section through the sharpened Patterson function, with contours at equal arbitrary intervals. Superimposed is the theoretical vector set, the relative weights of the vectors are shown by black dots for weight 1, small + for weight 2 and large + for weight 4. The radius of the section is 5 Å.

calculated for both configurations (Fig. 4). In Fig. 4(*a*), corresponding to the parallel configuration, all three peaks are approximately the same height, whereas in Fig. 4(*b*) corresponding to the antiparallel configuration there is one peak which is larger than the other two. The function $Q(X_0, Z_0)$ was calculated for both configurations to determine the position of the molecule relative to the 2_1 axis. The map for the antiparallel configuration is shown in Fig. 5. The centre of symmetry in this map, and the centre in the $Q(Y_0)$ function for the parallel configuration, arise from the symmetrical shape of the part of the molecule used. Within the asymmetric unit of the map in Fig. 5 there is one peak which is very large compared with the remainder whereas the function $Q(X_0, Z_0)$ for the parallel configuration showed no outstandingly large peak. It was concluded that the antiparallel configuration was the correct one, and that the position of the molecule in the unit cell was defined by the largest peaks in Fig. 4(*b*) and Fig. 5.

There are two possible positions for the acetyl group, assuming it to be coplanar with the phenyl ring to which it is attached. On the basis of structure factor calculations on the (*h*0*l*) zone, one of the possibilities was eliminated. A similar technique was employed to position the fluorine atom. The structure was then refined in projection to $R(h0l) = 0{\cdot}14$, where $R = \Sigma\, ||F_o| - |F_c|| / \Sigma\, |F_o|$ initially by Fourier refinement and then by a least-squares process using an overall isotropic temperature factor. The least-squares refinement program used was that due to J. C. Schoone (Rogers, 1961) for the Statec ZEBRA computer.

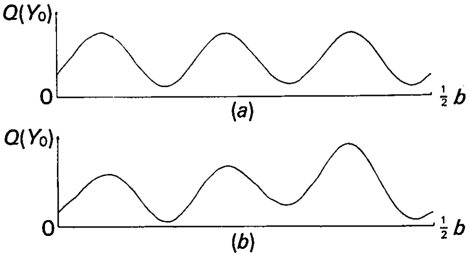

Fig. 4. The function $Q(Y_0)$, (*a*) for the parallel configuration, (*b*) for the anti-parallel configuration.

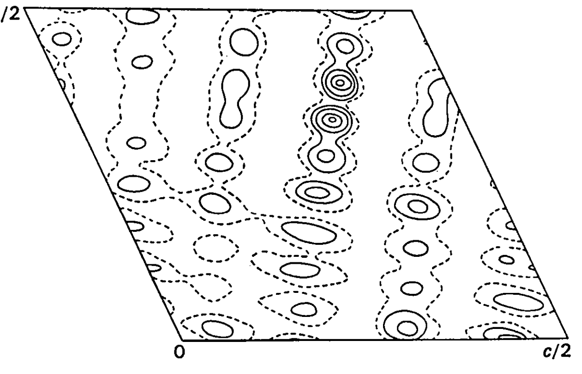

Fig. 5. The function $Q(X_0, Z_0)$ for anti-parallel configuration.

In order to confirm the proposed structure, a refinement based on the refined *x* and *z* coordinates and the *y* coordinates from $Q(Y_0)$ was carried out on the 460 largest general structure factors. The structure was quickly refined to $R = 0{\cdot}15$.

Refinement

Refinement of the structure was continued with all the observed data, by least-squares using an overall isotropic temperature factor and proceeded from $R = 0{\cdot}35$ to 0·19 in 5 cycles. A three-dimensional electron density map was computed with the program of Daly, Stephens & Wheatley (1963) for the Elliot 803B computer, to check that the peak heights and the background were reasonable. Introducing individual isotropic temperature factors a further four cycles reduced *R* to 0·17. Coordinates for eight of the eleven hydrogen atoms were computed on the assumption of their planarity with the phenyl ring to which they were attached and a C–H bond length of 1·05 Å. After two further cycles *R* fell to 0·15. Further least-squares refinement was carried out with the SFLS program of G. A. Mair for the IBM 1620 II computer. The program uses the block-diagonal approximation and the coefficients were weighted by

$$\omega = 1/\{1 + (KF_o - b)^2/a^2\}\,,$$

where *a* and *b* are constants selected to discriminate against weak reflexions and against very strong reflexions. Anisotropic temperature factors of the form

$$\exp\,[-(B_{11}h^2 + B_{22}k^2 + B_{33}l^2 + B_{12}hk + B_{23}kl + B_{13}hl)]$$

were incorporated into the refinement process which proceeded to $R = 0{\cdot}11$. A three-dimensional difference Fourier synthesis showed regions of density up to 0·4 e.Å^{-3} around the methyl carbon atom. These regions were in the form of long elongated distributions curving round the carbon atom, suggesting that the methyl group may undergo hindered rotation about the C–C bond. Hydrogen atom positions were allocated to three of the highest regions, lying between 0·9 and 1·2 Å from the carbon atom and chosen so that the C–C–H bond angles were of the order of 110°. Further refinement proceeded to $R = 0{\cdot}094$ for the observed data. In the latter stages of refinement the reflexions 002 and 204 were excluded as it was suspected that these suffered from extinction. The temperature parameters of the methyl hydrogen atoms increased to about 8 Å^2, much larger than the *B* values for the hydrogen atoms connected to the phenyl rings and consistent with the smearing effect noted in the difference synthesis. Little significance can be attributed to the positions of the methyl hydrogen atoms. The final structure factors are given in Table 1. Table 2 gives the final heavy-atom coordinates and anisotropic thermal parameters. The hydrogen atom parameters appear in Table 3.

Table 1. *The observed and calculated structure factors*

H	K	L	100FO	100FC
0	0	2	3000	3477
0	0	4	393	-344
0	0	6	1651	1663
0	0	8	378	289
0	0	10	175	188
0	0	12	301	329
0	0	14	•	-190
0	0	16	547	606
1	0	0	421	-435
1	0	2	1567	-1768
1	0	4	1257	1169
1	0	6	675	-632
1	0	8	259	214
1	0	10	203	167
1	0	12	•	-20
1	0	14	450	389
1	0	16	414	495
1	0	-2	•	-228
1	0	-4	1672	1728
1	0	-6	386	-375
1	0	-8	182	90
1	0	-10	218	-192
1	0	-12	358	-334
1	0	-14	•	-70
2	0	0	1988	2105
2	0	2	133	140
2	0	4	554	474
2	0	6	463	-495
2	0	8	513	-487
2	0	10	182	213
2	0	12	905	-836
2	0	14	878	-893
2	0	-2	1419	-1430
2	0	-4	8792	-10030
2	0	-6	407	-344
2	0	-8	337	288
2	0	-10	646	-611
2	0	-12	•	187
2	0	-14	295	225
3	0	0	2585	-2749
3	0	2	2459	2681
3	0	4	990	-851
3	0	6	1067	-1021
3	0	8	513	531
3	0	10	•	-15
3	0	12	822	-814
3	0	14	189	156
3	0	-2	1601	1730
3	0	-4	801	-728
3	0	-6	779	-734
3	0	-8	948	932
3	0	-10	323	-311
3	0	-12	997	-1082
3	0	-14	•	-179
3	0	-16	•	63
4	0	0	2002	-2104
4	0	2	435	-416
4	0	4	1096	-1006
4	0	6	456	-511
4	0	8	344	334
4	0	10	667	546
4	0	12	•	-53
4	0	14	218	-227
4	0	-2	1531	-1699
4	0	-4	1173	-1198
4	0	-6	990	-1055
4	0	-8	934	869
4	0	-10	295	-293
4	0	-12	182	156
4	0	-14	393	362
4	0	-16	•	-61
5	0	0	3407	3514
5	0	2	3485	3706
5	0	4	•	66
5	0	6	225	-198
5	0	8	892	810
5	0	10	•	-14
5	0	12	•	2
5	0	-2	1419	-1358
5	0	-4	2269	2339
5	0	-6	675	741
5	0	-8	1299	1364
5	0	-10	547	549
5	0	-12	161	91
5	0	-14	477	447
5	0	-16	675	648
6	0	0	1616	1497
6	0	2	477	-422
6	0	4	•	114
6	0	6	252	230
6	0	8	288	223
6	0	10	•	-56
6	0	12	182	194
6	0	-2	913	-893
6	0	-4	161	56
6	0	-6	745	-708
6	0	-8	962	-972
6	0	-10	323	347
6	0	-12	182	110
6	0	-14	•	-52
6	0	-16	•	-111
7	0	0	456	-417
7	0	2	225	199
7	0	4	•	-169
7	0	6	358	-322
7	0	8	407	-342
7	0	10	•	-86
7	0	-2	2494	-2629
7	0	-4	2031	-2124
7	0	-6	808	794
7	0	-8	•	-31
7	0	-10	•	-97
7	0	-12	295	-269
7	0	-14	•	88
8	0	0	•	-81
8	0	2	•	-82
8	0	4	•	153
8	0	6	779	746
8	0	8	•	213
8	0	10	182	147
8	0	-2	675	731
8	0	-4	667	-598
8	0	-6	815	837
8	0	-8	456	435
8	0	-10	1292	1201
8	0	-12	547	623
8	0	-14	•	-205
9	0	0	1060	-1052
9	0	2	•	-31
9	0	4	182	235
9	0	6	•	89
9	0	8	•	-255
9	0	10	211	201
9	0	-2	315	-309
9	0	-4	520	-541
9	0	-6	•	-24
9	0	-8	295	228
9	0	-10	603	-658
9	0	-12	456	-502
9	0	-14	•	-185
10	0	0	•	-113
10	0	2	653	-711
10	0	4	646	-640
10	0	6	•	-171
10	0	8	175	192
10	0	-2	•	-134
10	0	-4	632	695
10	0	-6	506	545
10	0	-8	513	502
10	0	-10	182	108
10	0	-12	•	27
10	0	-14	660	-674
11	0	0	•	67
11	0	2	•	-232
11	0	4	315	264
11	0	6	•	-115
11	0	-2	•	185
11	0	-4	267	227
11	0	-6	583	-584
11	0	-8	407	361
11	0	-10	•	162
11	0	-12	•	-117
11	0	-14	•	92
12	0	0	554	607
12	0	2	•	-49
12	0	4	189	-222
12	0	-2	•	184
12	0	-4	•	112
12	0	-6	407	416
12	0	-8	443	-445
12	0	-10	499	-485
12	0	-12	•	-105
12	0	-14	•	-48
13	0	0	•	-38
13	0	2	182	143
13	0	4	•	-48
13	0	-2	569	548
13	0	-4	•	-85
13	0	-6	•	8
13	0	-8	941	877
13	0	-10	997	895
13	0	-12	•	-197
14	0	0	•	82
14	0	2	•	67
14	0	-2	•	130
14	0	-4	443	-366
14	0	-6	•	-98
14	0	-8	386	-418
14	0	-10	443	-468
14	0	-12	•	-26
15	0	0	•	-81
15	0	2	•	-58
15	0	-2	•	-130
15	0	-4	•	16
15	0	-6	365	-333
15	0	-8	281	233
15	0	-10	•	37
15	0	-12	709	-733
15	0	-14	499	-525
0	1	1	3470	4065
0	1	2	1345	-1406
0	1	3	2740	-2878
0	1	4	2344	2385
0	1	5	1012	-987
0	1	6	127	-97
0	1	7	141	-132
0	1	8	778	-762
0	1	9	290	-295
0	1	10	•	82
0	1	11	•	-191
0	1	12	481	477
0	1	13	•	-180
0	1	14	•	-180
0	1	15	•	37
0	1	16	•	-22
1	1	0	567	558
1	1	1	481	-467
1	1	2	1083	1126
1	1	3	1444	1428
1	1	4	1579	1612
1	1	5	906	-847
1	1	6	297	-274
1	1	7	630	623
1	1	8	651	697
1	1	9	687	693
1	1	10	304	327
1	1	11	•	38
1	1	12	240	194
1	1	13	•	-17
1	1	14	318	347
1	1	15	311	352
1	1	16	120	154
1	1	-1	1098	1128
1	1	-2	3548	3939
1	1	-3	304	347
1	1	-4	4235	4604
1	1	-5	969	980
1	1	-6	969	997
1	1	-7	488	387
1	1	-8	637	625
1	1	-9	1203	1232
1	1	-10	197	-253
1	1	-11	•	20
1	1	-12	•	95
1	1	-13	•	-115
1	1	-14	•	87
1	1	-15	•	-24
1	1	-16	•	-48
1	1	-17	•	-23
2	1	0	1423	-1442
2	1	1	•	47
2	1	2	•	-42
2	1	3	•	-74
2	1	4	326	231
2	1	5	•	104
2	1	6	1076	-1049
2	1	7	453	388
2	1	8	778	813
2	1	9	•	221
2	1	10	559	513
2	1	11	•	178
2	1	12	240	205
2	1	13	630	-683
2	1	14	•	83
2	1	15	177	-227
2	1	-1	569	944
2	1	-2	283	254
2	1	-3	5284	-5802
2	1	-4	•	-46
2	1	-5	2104	2084
2	1	-6	545	-539
2	1	-7	842	856
2	1	-8	1317	1279
2	1	-9	361	-334
2	1	-10	184	167
2	1	-11	318	-357
2	1	-12	318	-286
2	1	-13	•	22
2	1	-14	•	37
2	1	-15	•	-74
2	1	-16	•	-72
2	1	-17	•	15
3	1	0	261	-140
3	1	1	233	173
3	1	2	1282	1299
3	1	3	1339	-1231
3	1	4	1019	-871
3	1	5	1693	-1723
3	1	6	226	180
3	1	7	375	-342
3	1	8	•	-73
3	1	9	•	72
3	1	10	785	-816
3	1	11	552	-516
3	1	12	•	14
3	1	13	276	-297
3	1	14	163	232
3	1	-1	1196	-1211
3	1	-2	1841	1831
3	1	-3	•	-45
3	1	-4	4568	4574
3	1	-5	545	-474
3	1	-6	969	-961
3	1	-7	424	393
3	1	-8	1905	-2107
3	1	-9	•	-16
3	1	-10	•	-20
3	1	-11	368	-349
3	1	-12	212	-130
3	1	-13	567	-505
3	1	-14	•	45
3	1	-15	•	92
3	1	-16	•	1
3	1	-17	•	118
4	1	0	446	426
4	1	1	835	768
4	1	2	481	324
4	1	3	•	55
4	1	4	1423	-1360
4	1	5	573	532
4	1	6	729	-686
4	1	7	•	32
4	1	8	637	-628
4	1	9	878	923
4	1	10	•	76
4	1	11	545	465
4	1	12	197	223
4	1	13	•	62
4	1	-1	969	963
4	1	-2	921	-841
4	1	-3	2047	2007
4	1	-4	1203	-1151
4	1	-5	1026	1009
4	1	-6	410	-420
4	1	-7	2833	2974
4	1	-8	998	-1008
4	1	-9	785	818
4	1	-10	446	-411
4	1	-11	•	-11
4	1	-12	594	-562
4	1	-13	311	313
4	1	-14	269	-244
4	1	-15	219	144
4	1	-16	•	-118
4	1	-17	•	-80
5	1	0	630	601
5	1	1	1409	1250
5	1	2	538	-537
5	1	3	•	71
5	1	4	354	-328
5	1	5	665	-683
5	1	6	744	734
5	1	7	•	32
5	1	8	524	-501
5	1	9	481	478
5	1	10	680	-720
5	1	11	•	-81
5	1	12	197	-237
5	1	-1	687	-646
5	1	-2	552	-494
5	1	-3	962	-923
5	1	-4	318	-266
5	1	-5	744	-709
5	1	-6	1523	-1429
5	1	-7	382	-347
5	1	-8	2032	-2114
5	1	-9	156	-199
5	1	-10	226	237
5	1	-11	580	-643
5	1	-12	552	523
5	1	-13	212	-224
5	1	-14	•	-6
5	1	-15	226	259
5	1	-16	226	198
5	1	-17	•	155
6	1	0	1317	317
6	1	1	120	150
6	1	2	212	161
6	1	3	•	84
6	1	4	1126	1212
6	1	5	594	-646
6	1	6	204	197
6	1	7	510	-443
6	1	8	333	-359
6	1	9	240	-204
6	1	10	•	64
6	1	11	•	-64
6	1	-1	495	406
6	1	-2	1643	1570
6	1	-3	545	522
6	1	-4	226	-202
6	1	-5	127	-153
6	1	-6	431	392
6	1	-7	1161	-1127
6	1	-8	672	621
6	1	-9	•	-78
6	1	-10	177	185
6	1	-11	517	-520
6	1	-12	531	579
6	1	-13	•	-133
6	1	-14	•	-1
6	1	-15	•	17
6	1	-15	•	-59
6	1	-17	•	-52
7	1	0	333	-283
7	1	1	531	488
7	1	2	403	-361
7	1	3	446	492
7	1	4	517	540
7	1	5	403	-380
7	1	6	538	498
7	1	7	•	84
7	1	8	304	319
7	1	9	•	-207
7	1	10	•	-153
7	1	-1	431	-305
7	1	-2	290	-314
7	1	-3	1140	-1104
7	1	-4	382	-412
7	1	-5	651	636
7	1	-6	885	-902
7	1	-7	333	319
7	1	-8	368	-321
7	1	-9	233	198
7	1	-10	283	257
7	1	-11	197	-152
7	1	-12	410	369
7	1	-13	•	-61
7	1	-14	•	-7
7	1	-15	•	47
7	1	-16	•	145
7	1	-17	•	-51
8	1	0	254	-220
8	1	1	•	10
8	1	2	290	-263
8	1	3	•	-114
8	1	4	311	267
8	1	5	•	-13
8	1	6	•	-27
8	1	7	276	278
8	1	8	•	-7
8	1	9	•	-63
8	1	-1	431	-443
8	1	-2	1211	1238
8	1	-3	694	-696
8	1	-4	156	142
8	1	-5	410	-405
8	1	-6	715	-623
8	1	-7	680	-646
8	1	-8	460	465
8	1	-9	•	55
8	1	-10	446	433
8	1	-11	680	646
8	1	-12	233	262
8	1	-13	•	-108
8	1	-14	•	-56
8	1	-15	•	145
8	1	-16	•	-234
8	1	-17	•	190
8	1	-18	170	172
9	1	0	304	-289
9	1	1	368	464
9	1	2	269	287
9	1	3	•	80
9	1	4	•	-4
9	1	5	457	499
9	1	6	333	283
9	1	7	•	-118
9	1	8	•	199
9	1	-1	453	412
9	1	-2	403	-372
9	1	-3	658	663
9	1	-4	226	184
9	1	-5	771	753
9	1	-6	•	-19
9	1	-7	1062	1064
9	1	-8	219	-203
9	1	-9	•	39
9	1	-10	431	396
9	1	-11	•	133
9	1	-12	•	113
9	1	-13	•	79
9	1	-14	•	79
9	1	-15	•	93
9	1	-16	•	49
9	1	-17	149	99
10	1	0	•	82
10	1	1	•	21
10	1	2	247	-252
10	1	3	545	-533
10	1	4	226	-247
10	1	5	•	6
10	1	6	•	65
10	1	7	•	49
10	1	-1	•	-90
10	1	-2	368	344
10	1	-3	170	-213
10	1	-4	744	-718
10	1	-5	•	54
10	1	-6	1105	-1106
10	1	-7	269	236
10	1	-8	361	-347
10	1	-9	197	-236
10	1	-10	•	80
10	1	-11	•	39
10	1	-12	•	-137
10	1	-13	340	-324
10	1	-14	191	-207
10	1	-15	333	-375
10	1	-16	177	-200
11	1	0	283	-156
11	1	1	538	-561
11	1	2	•	-192
11	1	3	•	-13
11	1	4	•	-53
11	1	5	•	35
11	1	6	•	97
11	1	-1	•	129
11	1	-2	382	-410
11	1	-3	•	-165
11	1	-4	•	124
11	1	-5	892	-961
11	1	-6	354	378
11	1	-7	559	-554
11	1	-8	212	177
11	1	-9	•	-126
11	1	-10	261	282
11	1	-11	389	-389
11	1	-12	197	139
11	1	-13	•	44
11	1	-14	•	-87
11	1	-15	•	27
12	1	0	•	177
12	1	1	•	147
12	1	2	•	-122
12	1	3	•	81
12	1	4	•	45
12	1	5	•	-175
12	1	-1	567	538
12	1	-2	•	63
12	1	-3	•	134
12	1	-4	615	-669
12	1	-5	333	383
12	1	-6	665	-694
12	1	-7	•	180
12	1	-8	354	290
12	1	-9	•	-1
12	1	-10	318	267
12	1	-11	•	-112
12	1	-12	•	81
12	1	-13	•	93
12	1	-14	•	-26
12	1	-15	•	-21
12	1	-16	•	-25
12	1	-17	127	175
13	1	0	•	-155
13	1	1	•	-104
13	1	2	•	-168
13	1	3	197	174
13	1	4	•	-28
13	1	-1	•	26
13	1	-2	•	38
13	1	-3	•	174
13	1	-4	340	410
13	1	-5	474	-477
13	1	-6	340	352
13	1	-7	•	-27
13	1	-8	283	272
13	1	-9	722	842
13	1	-10	283	253
13	1	-11	191	197
13	1	-12	•	-41
13	1	-13	•	-88
13	1	-14	•	-160
14	1	0	•	-35
14	1	1	•	152
14	1	2	•	43
14	1	3	•	-21
14	1	-1	•	-17
14	1	-2	•	73
14	1	-3	•	-134
14	1	-4	•	34
14	1	-5	•	-152
14	1	-6	•	138
14	1	-7	•	17
14	1	-8	417	400
14	1	-9	•	-146
14	1	-10	403	404
14	1	-11	•	95
14	1	-12	•	88
14	1	-13	•	85
14	1	-14	•	41
15	1	0	•	100
15	1	1	120	134
15	1	-1	•	-180
15	1	-2	•	76
15	1	-3	•	75
15	1	-4	•	117
15	1	-5	•	-40
15	1	-6	•	49
15	1	-7	•	-15
15	1	-8	215	-204
15	1	-9	226	239
15	1	-10	•	-81
15	1	-11	•	-140
15	1	-12	240	283
15	1	-13	424	546
15	1	-14	222	300
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0	2	1	535	533
0	2	2	693	-703
0	2	3	844	907
0	2	4	514	435
0	2	5	1881	2053
0	2	6	206	184
0	2	7	191	181
0	2	8	480	520
0	2	9	206	231
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0	2	13	281	-355
0	2	14	178	38
0	2	15	151	59
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1	2	2	494	408
1	2	3	144	-136
1	2	4	1641	-1685
1	2	5	103	-185
1	2	6	1002	-979
1	2	7	391	-413
1	2	8	673	663
1	2	9	185	-163
1	2	10	•	27
1	2	11	308	-335
1	2	12	191	-148
1	2	13	•	-120
1	2	14	•	-107
1	2	15	•	26
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1	2	-2	954	-928
1	2	-3	206	182
1	2	-4	253	250
1	2	-5	789	755
1	2	-6	761	-762
1	2	-7	•	-18
1	2	-8	301	220
1	2	-9	•	-138
1	2	-10	288	267
1	2	-11	185	83
1	2	-12	460	-506
1	2	-13	213	203
1	2	-14	288	-247
1	2	-15	158	-40
1	2	-16	•	-19
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2	2	1	974	-960
2	2	2	754	-738
2	2	3	748	712
2	2	4	713	-659
2	2	5	616	801
2	2	6	494	494
2	2	7	789	854
2	2	8	•	107
2	2	9	974	991
2	2	10	•	120
2	2	11	391	373
2	2	12	•	65
2	2	13	•	4
2	2	-1	1236	1274
2	2	-2	616	-820
2	2	-3	144	116
2	2	-4	391	-265
2	2	-5	514	526
2	2	-6	1084	1097
2	2	-7	1366	1390
2	2	-8	432	447
2	2	-9	1050	1149
2	2	-10	•	102
2	2	-11	•	70
2	2	-12	•	-128
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2	2	-14	•	56
2	2	-15	226	-224
2	2	-16	•	-82
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3	2	1	2231	-2421
3	2	2	871	867
3	2	3	164	-220
3	2	4	611	-644
3	2	5	411	-395
3	2	6	391	436
3	2	7	178	151
3	2	8	274	251
3	2	9	611	-636
3	2	10	198	158
3	2	11	226	-303
3	2	12	•	-94
3	2	13	•	160
3	2	-1	1407	-1446
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3	2	-3	473	440
3	2	-4	439	452
3	2	-5	253	-238
3	2	-6	1002	1025
3	2	-7	2292	-2450
3	2	-8	268	-266
3	2	-9	974	-1009
3	2	-10	370	419
3	2	-11	274	-267
3	2	-12	185	191
3	2	-13	•	-110
3	2	-14	•	26
3	2	-15	•	-117
3	2	-16	206	250
3	2	-17	151	-176
4	2	0	96	-80
4	2	1	411	-322
4	2	2	789	-788
4	2	3	123	-102
4	2	4	•	53
4	2	5	789	-823
4	2	6	425	-429
4	2	7	624	-730
4	2	8	191	-162
4	2	9	316	-307
4	2	10	501	493
4	2	11	•	163
4	2	12	•	177
4	2	13	•	33
4	2	14	93	164
4	2	-1	473	403
4	2	-2	1345	1382
4	2	-3	947	977
4	2	-4	645	584
4	2	-5	•	-29
4	2	-6	1407	1446
4	2	-7	432	530
4	2	-8	1221	1302
4	2	-9	316	332
4	2	-10	521	-543
4	2	-11	•	-37
4	2	-12	446	-456
4	2	-13	•	-33
4	2	-14	•	-68
4	2	-15	•	59
4	2	-16	•	-21
5	2	0	494	472
5	2	1	844	885
5	2	2	521	-479
5	2	3	240	209
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5	2	5	699	1008
5	2	6	158	147
5	2	7	926	953
5	2	8	425	460
5	2	9	•	-200
5	2	10	•	125
5	2	11	•	-226
5	2	12	•	46
5	2	13	231	262
5	2	-1	•	107
5	2	-2	•	-61
5	2	-3	2086	2252
5	2	-4	611	-610
5	2	-5	596	545
5	2	-6	288	-191
5	2	-7	1043	-1141
5	2	-8	144	-153
5	2	-9	356	379
5	2	-10	144	-163
5	2	-11	1104	1283
5	2	-12	425	370
5	2	-13	•	32
5	2	-14	•	-65
5	2	-15	•	11
5	2	-16	•	-58
6	2	0	549	-508
6	2	1	425	-392
6	2	2	446	432
6	2	3	253	-190
6	2	4	151	-65
6	2	5	563	571
6	2	6	1166	-1217
6	2	7	281	-306
6	2	8	425	-438
6	2	9	185	-209
6	2	10	213	-229
6	2	-1	954	-952
6	2	-2	618	611
6	2	-3	541	-510
6	2	-4	556	524
6	2	-5	761	-783
6	2	-6	1126	-1161
6	2	-7	624	-645
6	2	-8	350	-342
6	2	-9	233	-243
6	2	-10	947	-1064
6	2	-11	274	260
6	2	-12	1064	-1154
6	2	-13	198	44
6	2	-14	•	-90
6	2	-15	227	-209
6	2	-16	164	92
7	2	0	350	-347
7	2	1	•	-35
7	2	2	611	-647
7	2	3	631	-625
7	2	4	803	-905
7	2	5	377	387
7	2	6	206	-142
7	2	7	549	532
7	2	8	•	-29
7	2	9	•	-100
7	2	10	•	-63
7	2	11	145	-146
7	2	-1	316	317
7	2	-2	377	-389
7	2	-3	130	70
7	2	-4	191	-168
7	2	-5	748	823
7	2	-6	•	-24
7	2	-7	308	-250
7	2	-8	308	-303
7	2	-9	350	359
7	2	-10	•	-2
7	2	-11	659	659
7	2	-12	185	50
7	2	-13	240	294
7	2	-14	213	-109
7	2	-15	356	-414
7	2	-16	307	-206
7	2	-17	158	143
8	2	0	459	448
8	2	1	1283	-1496
8	2	2	1132	1365
8	2	3	583	-572
8	2	4	404	393
8	2	5	301	298
8	2	6	•	124
8	2	7	•	49
8	2	8	•	-10
8	2	9	191	-131
8	2	-1	635	636
8	2	-2	•	5
8	2	-3	•	92
8	2	-4	398	414
8	2	-5	418	-398
8	2	-6	178	-172
8	2	-7	329	-320
8	2	-8	233	-182
8	2	-9	171	-139
8	2	-10	459	530
8	2	-11	206	-53
8	2	-12	•	107
8	2	-13	•	-129
8	2	-14	432	458
8	2	-15	624	-644
8	2	-16	521	598
8	2	-17	•	-162
8	2	-18	158	203
9	2	0	796	903
9	2	1	418	-429
9	2	2	206	-170
9	2	3	301	-335
9	2	4	•	-24
9	2	5	206	-144
9	2	6	206	245
9	2	7	•	37
9	2	8	•	-131
9	2	9	128	-81
9	2	-1	308	284
9	2	-2	809	914
9	2	-3	•	-89
9	2	-4	356	365
9	2	-5	•	-3
9	2	-6	713	696
9	2	-7	•	55
9	2	-8	•	-6
9	2	-9	•	-159
9	2	-10	219	-176
9	2	-11	198	78
9	2	-12	185	177
9	2	-13	•	38
9	2	-14	•	28
9	2	-15	•	36
9	2	-16	•	-28
9	2	-17	233	270
10	2	0	•	-39
10	2	1	185	114
10	2	2	•	112
10	2	3	•	-77
10	2	4	•	106
10	2	5	•	71
10	2	6	•	56
10	2	7	164	95
10	2	8	120	142
10	2	-1	1421	1500
10	2	-2	541	-517
10	2	-3	1297	1562
10	2	-4	350	-324
10	2	-5	549	-593
10	2	-6	171	-177
10	2	-7	158	167
10	2	-8	•	-33
10	2	-9	576	573
10	2	-10	•	70
10	2	-11	233	209
10	2	-12	•	79
10	2	-13	•	12
10	2	-14	•	-98
10	2	-15	•	-76
10	2	-16	•	104
11	2	0	•	80
11	2	1	•	24
11	2	2	240	-278
11	2	3	•	11
11	2	4	178	189
11	2	5	•	66
11	2	6	151	-172
11	2	-1	501	-535
11	2	-2	439	354
11	2	-3	•	44
11	2	-4	541	-476
11	2	-5	473	-451
11	2	-6	721	-714
11	2	-7	439	-452
11	2	-8	301	-278
11	2	-9	•	-137
11	2	-10	404	-481
11	2	-11	•	-18
11	2	-12	•	-65
11	2	-13	185	-98
11	2	-14	•	112
12	2	0	191	-143
12	2	1	206	192
12	2	2	•	7
12	2	3	•	-77
12	2	4	•	-151
12	2	5	172	102
12	2	-1	•	104
12	2	-2	268	-276
12	2	-3	•	-105
12	2	-4	219	-234
12	2	-5	782	-829
12	2	-6	281	-263
12	2	-7	541	-499
12	2	-8	•	-40
12	2	-9	473	430
12	2	-10	•	97
12	2	-11	•	-19
12	2	-12	•	-62
12	2	-13	•	-154
12	2	-14	•	-101
13	2	0	•	-164
13	2	1	•	-22
13	2	2	193	183
13	2	3	•	-155
13	2	4	176	121
13	2	-1	•	-7
13	2	-2	•	116
13	2	-3	•	42
13	2	-4	•	-17
13	2	-5	398	400
13	2	-6	226	-198

Table 1 (*cont.*)

H	K	L	100FO	100FC
13	2	-7	213	-177
13	2	-8	191	132
13	2	-9	226	201
13	2	-10	336	323
13	2	-11	185	164
13	2	-12	•	143
13	2	-13	253	-254
13	2	-14	242	262
13	2	-15	151	-162
14	2	0	237	179
14	2	1	•	-80
14	2	2	109	83
14	2	-1	•	23
14	2	-2	•	-75
14	2	-3	273	-259
14	2	-4	•	-146
14	2	-5	•	42
14	2	-6	•	131
14	2	-7	•	112
14	2	-8	198	173
14	2	-9	178	213
14	2	-10	261	276
14	2	-11	246	171
14	2	-12	•	126
14	2	-13	•	36
14	2	-14	•	-11
14	2	-15	178	56
15	2	0	281	-60
15	2	-1	350	345
15	2	-2	226	-101
15	2	-3	•	51
15	2	-4	•	99
15	2	-5	•	9
15	2	-6	•	-29
15	2	-7	•	10
15	2	-8	•	-61
15	2	-9	158	-73
15	2	-10	•	31
15	2	-11	151	110
15	2	-12	•	-24
15	2	-13	123	-141
16	2	0	•	58
16	2	-1	•	6
16	2	-2	•	-14
16	2	-3	•	53
16	2	-4	192	-187
16	2	-5	134	-117
16	2	-6	•	73
16	2	-7	•	19
16	2	-8	229	313
16	2	-9	166	-227
16	2	-10	144	57
16	2	-11	130	-66
0	3	1	•	-17
0	3	2	149	137
0	3	3	884	863
0	3	4	428	328
0	3	5	1081	1133
0	3	6	510	495
0	3	7	211	204
0	3	8	•	3
0	3	9	176	-176
0	3	10	299	-278
0	3	11	•	-16
0	3	12	516	-502
0	3	13	305	290
1	3	0	666	606
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1	5	-7	•	-77
1	5	-8	277	350
1	5	-9	•	68
1	5	-10	•	-50
1	5	-11	•	-29
1	5	-12	•	10
1	5	-13	•	-73
1	5	-14	•	59
1	5	-15	•	-25
2	5	0	180	141
2	5	1	153	122
2	5	2	319	282
2	5	3	22	-288
2	5	4	•	100
2	5	5	256	250
2	5	6	311	307
2	5	7	•	101
2	5	8	242	244
2	5	9	233	271
2	5	-1	429	421
2	5	-2	146	-149
2	5	-3	735	743
2	5	-4	735	730
2	5	-5	311	319
2	5	-6	402	472
2	5	-7	277	-285
2	5	-8	•	-99
2	5	-9	•	-94
2	5	-10	208	189
2	5	-11	333	333
2	5	-12	•	156
2	5	-13	•	252
2	5	-14	•	-65
2	5	-15	•	34
3	5	0	229	194
3	5	1	311	326
3	5	2	166	74
3	5	3	187	248
3	5	4	•	108
3	5	5	298	250
3	5	6	•	-190
3	5	7	222	-104
3	5	8	242	-223
3	5	9	235	-301
3	5	10	•	15
3	5	-1	180	149
3	5	-2	•	-107
3	5	-3	763	748
3	5	-4	923	-965
3	5	-5	1096	1194
3	5	-6	631	-670
3	5	-7	166	-126
3	5	-8	264	-303
3	5	-9	235	-235
3	5	-10	305	-368
3	5	-11	222	115
3	5	-12	•	-200
3	5	-13	•	-45
3	5	-14	•	47
3	5	-15	•	-10
4	5	0	215	-158
4	5	1	576	-638
4	5	2	333	-362
4	5	3	333	-349
4	5	4	367	-415
4	5	5	•	308
4	5	6	•	-169
4	5	7	•	47
4	5	8	513	-462
4	5	9	208	-195
4	5	10	291	-359
4	5	-1	124	71
4	5	-2	•	118
4	5	-3	555	-627
4	5	-4	208	220
4	5	-5	832	-924
4	5	-6	•	40
4	5	-7	596	-643
4	5	-8	374	-474
4	5	-9	•	-171
4	5	-10	222	-111
4	5	-11	•	158
4	5	-12	•	-64
4	5	-13	•	46
4	5	-14	•	-200
5	5	0	229	-238
5	5	1	•	-192
5	5	2	•	9
5	5	3	•	145
5	5	4	333	253
5	5	5	347	358
5	5	6	187	-137
5	5	7	•	-115
5	5	8	•	-59
5	5	9	333	-363
5	5	-1	222	265
5	5	-2	•	-12
5	5	-3	291	334
5	5	-4	311	-283
5	5	-5	478	499
5	5	-6	•	54
5	5	-7	•	84
5	5	-8	200	321
5	5	-9	•	-289
5	5	-10	•	-75
5	5	-11	•	43
6	5	0	291	296
6	5	1	429	-523
6	5	2	235	251
6	5	3	208	-149
6	5	4	•	171
6	5	5	•	75
6	5	-1	153	-63
6	5	-2	235	232
6	5	-3	311	335
6	5	-4	•	40
6	5	-5	187	-168
6	5	-6	319	-326
6	5	-7	•	130
6	5	-8	•	-61
6	5	-9	•	256
6	5	-10	•	136
6	5	-11	•	147
6	5	-12	•	95
6	5	-13	•	33
6	5	-14	•	60
7	5	0	•	-8
7	5	1	•	-15
7	5	2	187	112
7	5	3	•	-79
7	5	4	242	242
7	5	5	•	2
7	5	6	187	-137
7	5	-1	•	76
7	5	-2	333	353
7	5	-3	208	245
7	5	-4	347	410
7	5	-5	•	-33
7	5	-6	166	-73
7	5	-7	•	-33
7	5	-8	180	-65
7	5	-9	222	-229
7	5	-10	208	-174
7	5	-11	•	-179
7	5	-12	•	-26
7	5	-13	•	2
7	5	-14	111	85
8	5	0	•	1
8	5	1	•	54
8	5	2	291	206
8	5	3	208	-103
8	5	4	•	-103
8	5	5	200	-231
8	5	-1	•	131
8	5	-2	374	-404
8	5	-3	624	589
8	5	-4	•	-199
8	5	-5	367	302
8	5	-6	200	-175
8	5	-7	200	-219
8	5	-8	•	-22
8	5	-9	•	-37
8	5	-10	•	215
9	5	0	927	-952
9	5	1	230	207
9	5	2	•	30
9	5	3	•	123
9	5	-1	•	96
9	5	-2	174	101
9	5	-3	425	-441
9	5	-4	•	-183
9	5	-5	458	-419
9	5	-6	•	-276
9	5	-7	277	-283
9	5	-8	•	-153
9	5	-9	•	-209
9	5	-10	•	232
10	5	0	•	16
10	5	1	•	137
10	5	2	•	159
10	5	-1	•	66
10	5	-2	•	-208
10	5	-3	•	64
10	5	-4	•	79
10	5	-5	•	146
10	5	-6	208	260
10	5	-7	•	-229
10	5	-8	•	-20
11	5	0	232	-221
11	5	1	159	87
11	5	2	281	-378
11	5	-1	•	-108
11	5	-2	•	-132
11	5	-3	284	-257
11	5	-4	418	400
11	5	-5	•	-98
11	5	-6	•	-143
11	5	-7	245	208
11	5	-8	•	195
11	5	-9	•	256
12	5	0	•	-94
12	5	-1	•	-184
12	5	-2	•	-112
12	5	-3	166	-41
12	5	-4	•	55
12	5	-5	•	43
12	5	-6	•	-87
12	5	-7	•	-149
13	5	-4	102	98
0	6	0	704	-748
0	6	1	509	590
0	6	2	582	-709
0	6	3	509	454
0	6	4	509	-406
0	6	5	•	42
0	6	6	•	15
0	6	7	•	147
1	6	0	663	-776
2	6	0	341	327
2	6	-4	509	517
3	6	0	274	-160
4	6	0	•	-39
5	6	0	402	-414
6	6	0	281	-226
7	6	0	274	237

* Unobserved reflexions.

Table 2. *Final fractional coordinates and anisotropic temperature factors for the non-hydrogen atoms*

The anisotropic temperature factors have been multiplied by 10^5.

	x	y	z	B_{11}	B_{22}	B_{33}	B_{23}	B_{31}	B_{12}
F	0·3757	0·8924	0·4910	782	1815	787	1043	603	−461
O	−0·1636	0·7933	0·3252	791	4514	1251	464	658	1501
C(1)	0·2287	0·5682	0·4937	536	1234	465	194	452	−26
C(2)	0·1686	0·3891	0·4340	562	1244	643	−83	327	313
C(3)	0·0568	0·4046	0·3771	679	2041	511	−196	215	−235
C(4)	0·0018	0·5981	0·3809	627	2091	461	463	466	381
C(5)	0·0609	0·7719	0·4398	698	1875	727	205	755	546
C(6)	0·1704	0·7592	0·4959	727	1669	531	−641	528	−519
C(7)	0·3478	0·5501	0·5550	580	1077	438	−59	476	−220
C(8)	0·3953	0·3651	0·6171	474	1780	489	201	257	−328
C(9)	0·5069	0·3463	0·6701	740	2084	549	342	523	230
C(10)	0·5745	0·5131	0·6648	503	2994	610	−8	466	−136
C(11)	0·5288	0·6970	0·6042	716	2261	612	−383	628	−796
C(12)	0·4190	0·7108	0·5525	631	1537	449	152	403	73
C(13)	−0·1197	0·6197	0·3216	687	3811	504	478	424	173
C(14)	−0·1857	0·4274	0·2603	597	5594	793	322	214	−121

Discussion

The arrangement of the molecules in the unit cell viewed along the *b* axis is shown in Fig. 6. The intramolecular bond lengths and bond angles along with their standard deviations are given in Table 4.

Table 3. *The hydrogen atom fractional coordinates*

	x	y	z
H(2)	0·209	0·253	0·429
H(3)	0·011	0·277	0·335
H(5)	0·026	0·894	0·439
H(6)	0·212	0·878	0·528
H(8)	0·342	0·229	0·618
H(9)	0·530	0·246	0·709
H(10)	0·642	0·508	0·692
H(11)	0·573	0·826	0·595
H(12)	−0·269	0·446	0·242
H(13)	−0·180	0·417	0·195
H(14)	−0·171	0·282	0·291

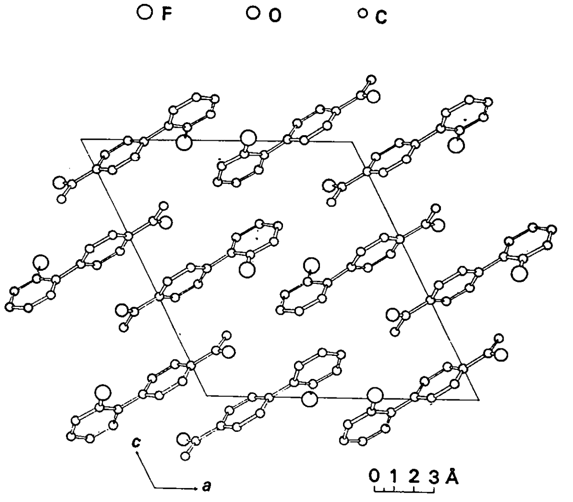

Fig. 6. The arrangement of the molecules viewed along the *b* axis.

Table 4. *The bond lengths and bond angles with their estimated standard deviations*

	Length	e.s.d.		Length	e.s.d.
F—C(12)	1·369 Å	0·008 Å	C(5)—C(6)	1·358 Å	0·012 Å
O—C(13)	1·211	0·009	C(7)—C(8)	1·399	0·009
C(1)-C(2)	1·399	0·009	C(7)—C(12)	1·380	0·008
C(1)-C(6)	1·400	0·008	C(8)—C(9)	1·380	0·011
C(1)-C(7)	1·479	0·010	C(9)—C(10)	1·385	0·009
C(2)-C(3)	1·386	0·012	C(10)-C(11)	1·380	0·010
C(3)-C(4)	1·394	0·009	C(11)-C(12)	1·355	0·011
C(4)-C(5)	1·366	0·010	C(13)-C(14)	1·495	0·013
C(4)-C(13)	1·505	0·012			

	Angle	e.s.d.		Angle	e.s.d.
C(2)-C(1)-C(6)	117·0°	0·7°	C(8)—C(7)—C(12)	116·0°	0·7°
C(2)-C(1)-C(7)	120·8	0·5	C(7)—C(8)—C(9)	120·9	0·5
C(6)-C(1)-C(7)	122·1	0·6	C(8)—C(9)—C(10)	120·7	0·6
C(1)-C(2)-C(3)	121·1	0·5	C(9)—C(10)-C(11)	119·1	0·7
C(2)-C(3)-C(4)	120·2	0·7	C(10)-C(11)-C(12)	119·1	0·6
C(3)-C(4)-C(5)	118·5	0·7	F—C(12)-C(7)	117·7	0·7
C(3)-C(4)-C(13)	121·7	0·7	F—C(12)-C(11)	118·0	0·5
C(5)-C(4)-C(13)	119·8	0·6	C(7)—C(12)-C(11)	124·3	0·6
C(4)-C(5)-C(6)	122·0	0·6	O—C(13)-C(4)	119·3	0·7
C(1)-C(6)-C(5)	121·2	0·6	O—C(13)-C(14)	120·4	0·8
C(1)-C(7)-C(8)	121·8	0·5	C(4)—C(13)-C(14)	120·3	0·6
C(1)-C(7)-C(12)	122·2	0·6			

The equations for the mean plane of the ring systems, C(1)–C(6): C(7)–C(12): C(7)–C(12) and F and the acetyl radical calculated by the method of Schomaker, Waser, Marsh & Bergman (1959), are

$$\begin{aligned} -0\cdot5182X-0\cdot3746Y+0\cdot7689Z&=3\cdot8148\\ -0\cdot4239X+0\cdot4724Y+0\cdot7728Z&=6\cdot7559\\ -0\cdot4249X+0\cdot4784Y+0\cdot7685Z&=6\cdot7350\\ -0\cdot5049X-0\cdot3404Y+0\cdot7934Z&=4\cdot0079 \end{aligned}$$

where X,Y,Z are the atomic coordinates in Å referred to the orthogonal axial triad $\mathbf{a},\mathbf{b},\mathbf{c}^*$. The deviations of the atoms from the mean planes of the phenyl rings are given in Table 5. The angle between the acetyl radical and the phenyl ring C(1)–C(6) is 2·5° and the angle between the two phenyl rings is 50·5°. The equivalent angle calculated from the $I(\theta,\varphi)$ map was 55°. The normal to the molecular plane as given by the $I(\theta,\varphi)$ function and the normals to the phenyl rings are thus in agreement to within 2·4°.

Table 5. *The deviations of the atoms from the mean planes of the phenyl rings*

C(1)	−0·009 Å	C(7)	−0·005 Å
C(2)	+0·008	C(8)	+0·002
C(3)	−0·005	C(9)	−0·002
C(4)	+0·004	C(10)	+0·003
C(5)	−0·005	C(11)	−0·006
C(6)	+0·008	C(12)	+0·006

The distance of 1·369 Å for the carbon–fluorine bond, obtained in this analysis, is considerably larger than the value published in *International Tables for X-ray Crystallography* (1962) of 1·30 ± 0·01 Å. In *o*-fluorobenzoic acid (Krausse & Dunken, 1966) the C–F distance was determined as 1·368 ± 0·019 Å in good agreement with the value found in the present analysis. The bond length distribution found in the fluoro-substituted phenyl ring is remarkably similar to that in *o*-fluorobenzoic acid, as can be seen in Table 6, which also shows the averaged equivalent bond lengths in biphenyl.

Table 6. *Comparison of equivalent bond distances in the fluoro-substituted phenyl ring in* 4-*acetyl*-2′-*fluorobiphenyl* (YTS) *and o-fluorobenzoic acid* (KD) *with those in the phenyl ring of biphenyl* (T, HR)*

Bond	(YTS)	(KD)	(T)	(HR)
C(7)—C(12)	1·380	1·380	1·372	1·383
C(12)–C(11)	1·355	1·365	1·416	1·421
C(11)–C(10)	1·380	1·395	1·376	1·361
C(10)–C(9)	1·385	1·395	1·376	1·361
C(9)—C(8)	1·380	1·375	1·416	1·421
C(8)—C(7)	1·399	1·395	1·372	1·383
Mean	1·382	1·384		

* YTS this investigation
KD Krausse & Dunken (1966)
T Trotter (1961)
HR Hargreaves & Rizvi (1962)

The C(1)–C(7) bond length of 1·479 ± 0·010 Å is significantly shorter than the quoted value of 1·54 Å. However in biphenyl this measured 1·506 ± 0·017 Å, which is possibly significantly larger than in this determination. This is perhaps due to the fact that biphenyl is a planar molecule and the steric repulsion between H(2) and H(12) and between H(6) and H(8) causes a strain between C(1) and C(7) which lengthens the bond. In the present structure determination, the molecule is non-planar and the steric repulsion between H(6) and H(8) no longer exists because of the twist about C(1)–C(7). The F–H(2) distance of 2·53 Å is just smaller than the value of 2·55 Å obtained by considering the van der Waals radii. The value obtained for C(1)–C(7) is in good agreement with the theoretical calculations of Dewar & Schmeising (1959) and Cruickshank & Sparks (1960), who suggest that the (sp^2) single-bond length between trigonally linked carbon atoms should be taken as 1·477 Å. This would indicate that there was no conjugation taking place between the rings in this structure.

All the intermolecular distances correspond to normal van der Waals interactions. All contacts less than 3·6 Å are given in Table 7.

Table 7. *Intermolecular distances between atoms less than* 3·6 Å *apart* (*excluding hydrogen atoms*)

i	j	Equipoint indication			d_{ij}
F	C(8)	$x,$	$1+y,$	z	3·326 Å
C(10)	O	$1+x,$	$1\cdot5-y,$	$0\cdot5+z$	3·508
C(11)	F	$1-x,$	$2-y,$	$1-z$	3·364
C(12)	C(10)	$1-x,$	$1-y,$	$1-z$	3·513
C(14)	F	$-x,$	$-0\cdot5+y,$	$0\cdot5-z$	3·473

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