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The Crystal Structure of 4-Acetyl-3′-bromobiphenyl

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The crystal structure of 4-acetyl-3′-bromobiphenyl, $C_{14}H_{11}OBr$, has been determined from three-dimensional X-ray diffraction data. The crystals are monoclinic, space group $P2_1/c$, with unit-cell dimensions $a=3{\cdot}91$, $b=9{\cdot}06$, $c=32{\cdot}76$ Å, $\beta=98{\cdot}1°$. The structure was refined by block-diagonal least-squares with anisotropic thermal parameters to a residual of 9% for the 936 observed structure factors. The angle between the two phenyl rings and the angle between the acetyl group and the phenyl ring to which it is attached were found to be 38·6° and 2·9° respectively. 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 & Worral, 1964) have created an interest in the interplanar angles of biphenyl nuclei in the liquid crystalline states. Whereas the molecule of biphenyl is planar in the solid phase (Dhar, 1932; Trotter, 1961; Hargreaves & Rizvi, 1962), several simple derivatives with 2 and 2′ halogen substitutions have interplanar angles of around 48° (Young, Tollin & Sutherland, 1968; Sutherland & Hoy, 1968, 1969; Sutherland, 1969). Angles of this order are expected from a simple treatment of overcrowding (Branch *et al.*, 1964) and it was of interest to study a derivative with a 3′ substituted halogen. Here details of the crystal and molecular structure of 4-acetyl-3′-bromobiphenyl are given, the fifth in a series of related compounds being investigated.

Experimental

4-Acetyl-3′-bromobiphenyl was crystallized from methanol. The lattice constants have been refined from provisional photographic estimates made with a Hilger and Watts linear diffractometer.

Crystal data

$C_{14}H_{11}OBr$, $M=275{\cdot}16$. Monoclinic, $a=3{\cdot}91\pm01$, $b=9{\cdot}06\pm01$, $c=32{\cdot}76\pm03$ Å, $\beta=98{\cdot}1\pm0{\cdot}1°$, $U=1149$ Å^3, $D_m=1{\cdot}59$ g.cm^{-3}, $D_c=1{\cdot}59$ g.cm^{-3}. $Z=4$, $F(000)=552$, Cu $K\alpha$ ($\lambda=1{\cdot}5418$ Å). *M*o $K\alpha$ ($\lambda=0{\cdot}7107$ Å).
Absent spectra: $0k0$ when k odd and $h0l$ when l odd.
Space group $P2_1/c$ (No. 14).

The crystals were colourless needles, the needle axis corresponding to the *a* axis of the unit cell. A suitable crystal of cross section 0·03 × 0·1 cm perpendicular to the needle axis was selected for intensity data collection about the *a* axis. Data for the $0kl$, $1kl$, $2kl$, $3kl$ levels of reciprocal space were collected by the multiple-film equi-inclination Weissenberg technique with Cu $K\alpha$ radiation. The $h0l$ intensity data were collected from a crystal cut to 0·15 × 0·03 × 0·02 cm. Packs of six films of Ilford Industrial B were used throughout.

Intensities of the spots were measured on a Joyce–Loebl Flying Spot Densitometer; no correction for absorption was applied as variations of less than 5% were predicted from the range of μR values (0·08–0·15). The data were brought to the same arbitrary scale and finally on to an approximately absolute scale by applying the method of Wilson (1942) to the $0kl$ level.

Determination and refinement of the structure

The $0kl$ projection of the unit cell was solved from the sharpened Patterson projection and refined by a combination of electron density distribution and block-diagonal least-squares methods to a residual of $R=0{\cdot}15$ where $R=\Sigma\,||F_o|-|F_c||/\Sigma\,|F_o|$. The calculations were carried out on an Elliott 803B computer with the use of the programs of Daly, Stephens & Wheatley (1963).

Apart from the Br–Br vectors, the $h0l$ projection of the Patterson function was poorly defined, although it appeared that the molecule was orientated close to the (104) plane. From this, provisional x coordinates were calculated.

A structure factor calculation with full three-dimensional data gave a residual of $R=0{\cdot}34$. This structure was refined by two cycles of three-dimensional electron density distributions and structure factor calculations to a residual of $R=0{\cdot}20$. An overall isotropic thermal parameter of 0·04 Å² was used, which had been calculated by the method of Wilson (1942). Individual iso-

Table 1. *Observed and calculated structure factors*

A dot denotes an unobserved reflexion.

H	K	L	100FO	100FC
0	0	2		-8969
0	0	4	3460	4514
0	0	6	6979	-7996
0	0	8	3483	3770
0	0	10	4084	-3860
0	0	12	4369	-3824
0	0	14	6709	8338
0	0	16	•	-698
0	0	18	6154	6092
0	0	20	3565	-3935
0	0	22	4894	5082
0	0	24	4400	-4104
0	0	26	4761	4445
0	0	28	2778	-2908
0	0	30	2178	1762
0	0	32	•	-1015
0	0	34	•	-189
0	0	36	•	738
0	0	38	•	146
0	0	40	•	85
0	1	1	4123	3395
0	1	2	993	-776
0	1	3	2364	-1584
0	1	4	5847	-4461
0	1	5	5481	-3930
0	1	6	8071	-9487
0	1	7	1323	-1620
0	1	8	7163	7885
0	1	9	4032	3779
0	1	10	6562	-9638
0	1	11	2061	-2373
0	1	12	7641	8167
0	1	13	2427	1969
0	1	14	7297	-7519
0	1	15	935	-581
0	1	16	9406	9083
0	1	17	3060	2560
0	1	18	6494	-5976
0	1	19	4116	-3693
0	1	20	•	1043
0	1	21	•	562
0	1	22	2589	-1939
0	1	23	•	-119
0	1	24	•	-439
0	1	25	•	-13
0	1	26	•	1229
0	1	27	•	910
0	1	28	•	-1746
0	1	29	•	-673
0	1	30	•	540
0	1	31	•	-22
0	1	32	3033	-2882
0	1	33	•	-624
0	1	34	1969	2193
0	1	35	•	840
0	1	36	2046	-1877
0	1	37	•	-225
0	1	38	1191	1426
0	1	39	•	389
0	1	40	•	-897
0	1	41	•	40
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0	2	3	7191	7195
0	2	4	3415	-10711
0	2	5	1615	-2235
0	2	6	3610	3743
0	2	7	5263	4776
0	2	8	6254	-7772
0	2	9	4004	-3347
0	2	10	2624	-1657
0	2	11	845	301
0	2	12	880	-400
0	2	13	2421	2028
0	2	14	2688	-2417
0	2	15	2794	-2714
0	2	16	4039	3532
0	2	17	2258	2519
0	2	18	4270	-4189
0	2	19	3426	-3126
0	2	20	3912	3520
0	2	21	2279	2197
0	2	22	6016	-5106
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0	2	25	1351	1924
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0	2	29	•	1746
0	2	30	•	-1301
0	2	31	•	-2368
0	2	32	•	1057
0	2	33	•	17
0	2	34	•	826
0	2	35	•	585
0	2	36	•	355
0	2	37	•	452
0	2	38	•	545
0	2	39	•	-360
0	2	40	•	-368
0	2	41	•	-102
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0	3	3	6150	5941
0	3	4	3572	-3394
0	3	5	3672	-3714
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0	3	8	6640	-6445
0	3	9	3630	-4094
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0	3	11	1984	2456
0	3	12	1365	-1247
0	3	13	6768	-6365
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0	3	22	•	-808
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0	3	25	792	-507
0	3	26	697	968
0	3	27	3070	-2273
0	3	28	2224	3002
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0	3	30	•	-730
0	4	0	4461	4911
0	4	1	6353	6987
0	4	2	•	-1206
0	4	3	2794	-3916
0	4	4	6106	5087
0	4	5	8577	8374
0	4	6	•	-626
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0	4	8	2498	2106
0	4	9	•	1036
0	4	10	•	-219
0	4	11	•	-145
0	4	12	•	-693
0	4	13	•	-1377
0	4	14	•	714
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0	4	16	2224	-1245
0	4	17	2279	-2451
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0	4	23	2575	1938
0	4	24	•	-1422
0	4	25	2977	-2594
0	4	26	•	1077
0	4	27	•	1135
0	5	1	•	596
0	5	2	•	-480
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0	5	4	•	-351
0	5	5	4349	4160
0	5	6	1527	-1664
0	5	7	6101	-5943
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0	5	13	5009	4732
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0	5	27	•	1573
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0	6	4	•	683
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0	6	7	4531	3774
0	6	8	•	-516
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0	6	10	•	-787
0	6	11	•	42
0	6	12	•	19
0	6	13	2293	1108
0	6	14	•	-867
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0	6	20	•	-173
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0	6	26	•	512
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0	7	2	•	15
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0	7	5	•	-198
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1	0	34	1141	-1349
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1	1	27	•	81
1	1	28	•	-687
1	1	29	•	-162
1	1	30	807	-1082
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1	2	-15	3424	-3261
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1	2	-10	5562	5339
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1	2	-8	665	-976
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1	2	-3	883	795
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1	2	9	2432	2500
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1	2	11	3315	-3191
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1	2	18	•	-487
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1	2	24	1985	-2144
1	2	25	•	-900
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1	2	28	1342	-1271
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1	2	31	•	502
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1	3	-31	•	1265
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1	3	-29	1906	-1804
1	3	-28	2617	2486
1	3	-27	2029	1771
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1	3	-25	•	-655
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1	3	-23	2752	2308
1	3	-22	1254	-1268
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1	3	-16	•	243
1	3	-15	1091	-328
1	3	-14	1645	1779
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1	3	-3	5125	-7514
1	3	-2	5976	5914
1	3	-1	2743	3147
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1	3	26	•	1125
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1	3	29	•	767
1	4	-27	•	1445
1	4	-26	•	-360
1	4	-25	1508	-1788
1	4	-24	•	753
1	4	-23	2432	2365
1	4	-22	1036	-1179
1	4	-21	5289	-5372
1	4	-20	1941	2215
1	4	-19	3948	3860
1	4	-18	2410	-2487
1	4	-17	3413	-3654
1	4	-16	•	673
1	4	-15	4711	4884
1	4	-14	3424	-3415
1	4	-13	3359	-3169
1	4	-12	1810	1643
1	4	-11	2313	2582
1	4	-10	•	-774
1	4	-9	1033	-1981
1	4	-8	828	678
1	4	-7	•	601
1	4	-6	•	-128
1	4	-5	3196	-2726
1	4	-4	1069	-1106
1	4	-3	3544	-3498

Table 1 (*cont.*)

H	K	L	100FO	100FC
2	4	8	2443	2232
2	4	9	5011	5037
2	4	10	•	-770
2	4	11	3002	-3044
2	4	12	2740	2653
2	4	13	2785	2819
2	4	14	1484	-1082
2	4	15	5445	-5578
2	4	16	1210	967
2	4	17	3310	2992
2	4	18	•	-937
2	4	19	•	-901
2	4	20	•	-813
2	4	21	•	-589
2	4	22	•	-118
2	4	23	•	1045
2	4	24	•	156
2	4	25	•	1418
2	4	26	•	1355
2	4	27	•	1084
2	4	28	•	-489
2	4	29	1929	-1940
2	4	30	•	697
2	4	31	•	-83
2	5	-31	•	1039
2	5	-30	•	605
2	5	-29	•	-908
2	5	-28	•	57
2	5	-27	1941	1892
2	5	-26	•	-849
2	5	-25	2443	-2394
2	5	-24	•	440
2	5	-23	3880	3938
2	5	-22	•	-1330
2	5	-21	2306	-2558
2	5	-20	•	278
2	5	-19	1861	1839
2	5	-18	924	906
2	5	-17	3744	-3572
2	5	-16	1632	2015
2	5	-15	•	338
2	5	-14	•	1079
2	5	-13	879	950
2	5	-12	1438	-1385
2	5	-11	•	-370
2	5	-10	845	881
2	5	-9	2682	2501
2	5	-8	•	-182
2	5	-7	2329	-2423
2	5	-6	1530	-1362
2	5	-5	3299	3404
2	5	-4	•	266
2	5	-3	4086	-3851
2	5	-2	2100	1750
2	5	-1	3733	3579
2	5	0	•	-239
2	5	1	4360	-4572
2	5	2	1746	1545
2	5	3	3653	3559
2	5	4	1027	-647
2	5	5	2465	-2620
2	5	6	856	506
2	5	7	1552	1622
2	5	8	1381	818
2	5	9	1392	-1189
2	5	10	•	125
2	5	11	•	-136
2	5	12	•	539
2	5	13	•	554
2	5	14	1541	-2051
2	5	15	•	-887
2	5	16	•	1063
2	5	17	•	795
2	5	18	•	-766
2	6	-25	•	498
2	6	-24	•	67
2	6	-23	•	-209
2	6	-22	1564	1978
2	6	-21	•	-1159
2	6	-20	•	432
2	6	-19	•	126
2	6	-18	936	-929
2	6	-17	1324	-1207
2	6	-16	1678	-1726
2	6	-15	2134	2314
2	6	-14	1039	1102
2	6	-13	2659	-2535
2	6	-12	•	-75
2	6	-11	3333	3437
2	6	-10	1210	-1308
2	6	-9	3847	-3975
2	6	-8	•	952
2	6	-7	4509	4477
2	6	-6	1895	1638
2	6	-5	2602	-2627
2	6	-4	1609	-1669
2	6	-3	•	-347
2	6	-2	•	522
2	6	-1	•	-703
2	6	0	1941	-1677
2	6	1	•	-558
2	6	2	1096	1104
2	6	3	•	906
2	6	4	•	-224
2	6	5	1872	-1902
2	6	6	•	-978
2	6	7	2911	2937
2	6	8	•	906
2	6	9	2934	-2925
2	6	10	•	-309
2	6	11	2465	2529
2	6	12	•	581
2	6	13	2283	-2287
2	6	14	1392	-1326
2	6	15	2774	2432
2	6	16	•	134
2	6	17	2168	-2042
2	6	18	•	188
2	6	19	981	1213
2	6	20	•	-403
2	6	21	•	-375
2	7	-25	•	1965
2	7	-24	•	181
2	7	-23	1598	-1625
2	7	-22	•	-893
2	7	-21	1404	1369
2	7	-20	•	381
2	7	-19	1757	-2006
2	7	-18	•	-840
2	7	-17	•	712
2	7	-16	•	157
2	7	-15	•	-115
2	7	-14	•	112
2	7	-13	•	283
2	7	-12	•	624
2	7	-11	1404	1515
2	7	-10	•	11
2	7	-9	•	-1385
2	7	-8	•	-1020
2	7	-7	•	1584
2	7	-6	•	1158
2	7	-5	936	-1324
2	7	-4	1975	-2164
2	7	-3	•	175
2	7	-2	•	-507
2	7	-1	3642	-3802
2	7	0	•	-641
2	7	1	3458	3722
2	7	2	1975	1673
2	7	3	1142	-540
2	7	4	•	490
2	7	5	•	981
2	7	6	•	773
2	7	7	•	-993
2	7	8	1541	-1203
2	7	9	1666	1926
2	7	10	•	155
2	7	11	•	272
2	7	12	1701	-1989
2	7	13	902	-1265
2	7	14	•	585
2	7	15	•	657
2	8	-16	•	952
2	8	-15	•	-905
2	8	-14	970	-980
2	8	-13	1244	1318
2	8	-12	•	906
2	8	-11	•	-1072
2	8	-10	1609	-1746
2	8	-9	1552	1679
2	8	-8	1426	1433
2	8	-7	1603	-1275
2	8	-6	1358	-924
2	8	-5	•	154
2	8	-4	1290	1416
2	8	-3	•	-1333
2	8	-2	•	-1132
2	8	-1	•	353
2	8	0	•	992
2	8	1	•	1130
2	8	2	•	877
2	8	3	•	213
2	8	4	•	-896
2	8	5	1895	1054
2	8	6	•	-116
2	8	7	1244	-1234
2	8	8	1438	-1830
2	8	9	1210	1199
2	8	10	2237	2092
2	8	11	2123	-2439
2	8	12	•	-602
2	8	13	•	402
2	8	14	•	446
2	9	-9	•	38
2	9	-8	•	1396
2	9	-7	•	-1268
2	9	-6	•	-1274
2	9	-5	994	1888
2	9	-4	•	465
2	9	-3	1621	-1626
2	9	-2	1621	-1256
2	9	-1	•	816
2	9	0	•	954
2	9	1	•	150
2	9	2	•	-764
2	9	3	•	409
2	9	4	•	756
3	0	-36	•	131
3	0	-34	1743	2168
3	0	-32	1804	-2447
3	0	-30	2871	2923
3	0	-28	1948	-2196
3	0	-26	2113	1570
3	0	-24	2768	-2222
3	0	-22	1292	1838
3	0	-20	•	373
3	0	-18	•	-1003
3	0	-16	1345	1366
3	0	-14	1289	-1405
3	0	-12	2756	2836
3	0	-10	2855	-2888
3	0	-8	3265	3193
3	0	-6	5906	-5708
3	0	-4	5332	5289
3	0	-2	3732	-3592
3	0	0	1046	1067
3	0	2	•	69
3	0	4	3551	3552
3	0	6	1604	1332
3	0	8	3145	-3240
3	0	10	•	-719
3	0	12	2297	-2103
3	0	14	1906	1838
3	0	16	2027	-1887
3	0	18	2543	2424
3	0	20	•	-753
3	0	22	•	1675
3	0	24	1066	-1222
3	0	26	•	80
3	0	28	•	-108
3	0	30	•	93
3	1	-27	•	-7
3	1	-26	•	1849
3	1	-25	•	192
3	1	-24	2019	-1632
3	1	-23	•	-794
3	1	-22	•	169
3	1	-21	•	-168
3	1	-20	2793	-2591
3	1	-19	•	-354
3	1	-18	3447	3569
3	1	-17	•	661
3	1	-16	3522	-3499
3	1	-15	933	-1649
3	1	-14	3297	2966
3	1	-13	1872	1984
3	1	-12	3173	-3018
3	1	-11	•	-176
3	1	-10	•	982
3	1	-9	1619	1576
3	1	-8	1117	-974
3	1	-7	•	-727
3	1	-6	•	597
3	1	-5	1569	-1794
3	1	-4	628	601
3	1	-3	•	578
3	1	-2	922	-787
3	1	-1	1166	-1173
3	1	0	2184	2205
3	1	1	•	-335
3	1	2	3538	-3431
3	1	3	•	36
3	1	4	3025	3130
3	1	5	3442	3551
3	1	6	5211	-5309
3	1	7	•	-211
3	1	8	2086	2605
3	1	9	1282	-1465
3	1	10	3167	-2734
3	1	11	•	-994
3	1	12	2636	2721
3	1	13	1319	1439
3	1	14	•	-898
3	1	15	•	-1355
3	1	16	•	115
3	1	17	•	234
3	1	18	•	977
3	1	19	•	633
3	1	20	•	-368
3	1	21	•	73
3	1	22	•	299
3	1	23	•	144
3	1	24	975	-1292
3	1	25	•	-706
3	1	26	•	899
3	2	-31	•	767
3	2	-30	•	-1255
3	2	-29	•	-1043
3	2	-28	•	1528
3	2	-27	2537	2007
3	2	-26	2706	-2399
3	2	-25	1739	-1289
3	2	-24	•	439
3	2	-23	•	-124
3	2	-22	2677	-2503
3	2	-21	1466	-1574
3	2	-20	•	752
3	2	-19	•	610
3	2	-18	1059	1447
3	2	-17	1940	2069
3	2	-16	•	-124
3	2	-15	•	-609
3	2	-14	•	674
3	2	-13	2274	2003
3	2	-12	2769	-2282
3	2	-11	2605	-2462
3	2	-10	4947	4855
3	2	-9	•	350
3	2	-8	4933	-4828
3	2	-7	1078	-1337
3	2	-6	2521	2529
3	2	-5	1506	1360
3	2	-4	2803	-3066
3	2	-3	2346	-2149
3	2	-2	2479	2356
3	2	-1	2711	2682
3	2	0	1751	-1537
3	2	1	•	-373
3	2	2	•	-41
3	2	3	•	757
3	2	4	•	525
3	2	5	•	-1252
3	2	6	•	-64
3	2	7	•	536
3	2	8	•	1205
3	2	9	•	925
3	2	10	•	-918
3	2	11	1186	-1766
3	2	12	•	858
3	2	13	•	1300
3	2	14	•	-816
3	2	15	•	-846
3	2	16	1372	1321
3	2	17	•	453
3	2	18	2357	-2003
3	2	19	•	-370
3	2	20	1621	1972
3	2	21	•	402
3	2	22	•	-950
3	3	-22	•	-1267
3	3	-21	•	-940
3	3	-20	2616	2423
3	3	-19	2654	2548
3	3	-18	•	-386
3	3	-17	1530	-1171
3	3	-16	1996	1716
3	3	-15	908	867
3	3	-14	1545	-1520
3	3	-13	1751	-1545
3	3	-12	•	282
3	3	-11	2615	2697
3	3	-10	1166	-742
3	3	-9	3137	-3407
3	3	-8	•	590
3	3	-7	1360	989
3	3	-6	1003	-1185
3	3	-5	•	359
3	3	-4	627	1205
3	3	-3	•	-702
3	3	-2	1988	1941
3	3	-1	2102	1930
3	3	0	2223	-1887
3	3	1	1660	-1460
3	3	2	•	1554
3	3	3	2613	2425
3	3	4	2229	-2240
3	3	5	2850	-3050
3	3	6	2993	2947
3	3	7	2248	2394
3	3	8	1819	-1678
3	3	9	1603	-1209
3	3	10	2099	2345
3	3	11	1735	1575
3	3	12	1357	-1685
3	3	13	2088	-2470
3	3	14	•	-369
3	3	15	•	679
3	4	-29	•	901
3	4	-28	•	-447
3	4	-27	1707	-1752
3	4	-26	•	759
3	4	-25	2152	2102
3	4	-24	•	-1038
3	4	-23	2523	-2532
3	4	-22	•	620
3	4	-21	•	876
3	4	-20	•	-480
3	4	-19	•	546
3	4	-18	•	-144
3	4	-17	•	329
3	4	-16	•	888
3	4	-15	•	73
3	4	-14	•	-158
3	4	-13	3334	-3386
3	4	-12	1416	1391
3	4	-11	4497	4688
3	4	-10	1092	-1904
3	4	-9	3447	-3401
3	4	-8	•	1048
3	4	-7	887	1326
3	4	-6	1439	-1590
3	4	-5	1432	-1120
3	4	-4	1676	1142
3	4	-3	3575	3177
3	4	-2	•	-563
3	4	-1	2276	-2191
3	4	0	725	833
3	4	1	•	336
3	4	2	•	86
3	4	3	•	-1177
3	4	4	•	-153
3	4	5	•	481
3	4	6	•	510
3	4	7	•	528
3	4	8	•	-561
3	4	9	•	-594
3	4	10	•	973
3	4	11	•	445
3	4	12	•	-1257
3	4	13	1676	-2046
3	4	14	•	334
3	4	15	•	1336
3	4	16	•	-1129
3	5	-21	•	531
3	5	-20	•	508
3	5	-19	2673	-2575
3	5	-18	•	559
3	5	-17	•	1334
3	5	-16	•	-451
3	5	-15	•	19
3	5	-14	•	-370
3	5	-13	1855	2328
3	5	-12	•	-1601
3	5	-11	2360	-2320
3	5	-10	•	198
3	5	-9	2143	2010
3	5	-8	•	690
3	5	-7	1347	-1503
3	5	-6	•	-842
3	5	-5	•	-481
3	5	-4	1341	1234
3	5	-3	•	472
3	5	-2	•	-395
3	5	-1	2391	-1681
3	5	0	1100	527
3	5	1	2467	2174
3	5	2	•	-994
3	5	3	2001	-2072
3	5	4	•	-7
3	5	5	2953	2552
3	5	6	•	163
3	5	7	2169	-2258
3	5	8	•	-366
3	5	9	2305	2101
3	5	10	•	219
3	5	11	•	-1595
3	5	12	•	-828
3	6	-16	•	-298
3	6	-15	•	-993
3	6	-14	•	1675
3	6	-13	1653	1650
3	6	-12	•	504
3	6	-11	2291	-2348
3	6	-10	•	-1160
3	6	-9	1675	1551
3	6	-8	•	1192
3	6	-7	1119	-1536
3	6	-6	•	-464
3	6	-5	1372	1287
3	6	-4	•	-660
3	6	-3	2626	-2567
3	6	-2	•	-759
3	6	-1	2017	2207
3	6	0	•	619
3	6	1	•	13
3	6	2	•	895
3	6	3	•	707
3	6	4	•	-280
3	6	5	•	-219
3	6	6	•	-407
3	6	7	•	-215
3	6	8	•	696
3	6	9	•	827
3	6	10	•	-843
4	0	0	2174	-2263
4	0	2	2502	2499
4	0	8	2769	-3673
4	0	10	1209	1636
4	0	-2	1497	1414
4	0	-8	1620	-1955
4	0	-18	1661	-2093

tropic thermal parameters were then included and the structure refined by block-diagonal least-squares to a residual of $R=0{\cdot}12$.

At this stage refinement was continued on the University of Leeds KDF9 computer with the use of the program of Cruickshank & Smith (1966). Individual anisotropic thermal parameters were defined as

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

(Cruickshank, Pilling, Bujosa, Lovell & Truter, 1961); individual scale factors for the batches of data were included in the refinement and a weighting scheme,

$$\sqrt{W}=1/\left\{10|F_{\min}|+|F_o|+\frac{2|F_o|^2}{|F_{\max}|}+\frac{5|F_o|^3}{|F_{\max}|^2}\right\}^{1/2}$$

was employed. At a residual of $R=0{\cdot}11$ a three-dimensional electron density difference synthesis was calculated, which showed regions of density where the eight hydrogen atoms attached to the phenyl rings would be expected. Another region of density appeared around the methyl carbon atom; this had no distinct peaks, indicating that the methyl group may undergo hindered rotation about the C–C bond. The methyl hydrogen atoms were allocated to the highest regions such that

Table 1 (*cont.*)

H	K	L	100FO	100FC
1	4	-2	632	483
1	4	-1	6325	5917
1	4	0	4166	-4166
1	4	1	5464	-5708
1	4	2	1930	1912
1	4	3	6684	6612
1	4	4	3446	-3828
1	4	5	6837	-7072
1	4	6	906	306
1	4	7	1000	2070
1	4	8	4263	-4340
1	4	9	3196	-3371
1	4	10	•	643
1	4	11	6540	6547
1	4	12	1462	-1294
1	4	13	1495	-1419
1	4	14	•	421
1	4	15	•	132
1	4	16	•	656
1	4	17	•	-259
1	4	18	•	194
1	4	19	1614	1316
1	4	20	•	-46
1	4	21	2501	-2669
1	4	22	2442	-2334
1	4	23	1777	-2151
1	4	24	•	361
1	4	25	2624	2942
1	4	26	1167	-1130
1	4	27	1243	-1102
1	4	28	•	705
1	4	29	2530	2393
1	4	30	•	-740
1	4	31	1756	-1760
1	4	32	•	774
1	4	33	•	1577
1	4	34	•	-361
1	5	-29	•	1159
1	5	-28	•	62
1	5	-27	1665	-1668
1	5	-26	•	168
1	5	-25	1352	1420
1	5	-24	•	761
1	5	-23	2192	-2064
1	5	-22	•	-377
1	5	-21	3500	3438
1	5	-20	3163	-3314
1	5	-19	•	953
1	5	-18	•	-450
1	5	-17	•	-302
1	5	-16	•	-5
1	5	-15	•	-751
1	5	-14	•	-5
1	5	-13	3253	-2925
1	5	-12	•	-135
1	5	-11	4595	4562
1	5	-10	1312	726
1	5	-9	6303	-6163
1	5	-8	2192	1921
1	5	-7	6194	6321
1	5	-6	3740	-3436
1	5	-5	4711	-4410
1	5	-4	•	-213
1	5	-3	6445	6443
1	5	-2	1690	-1935
1	5	-1	4602	-4577
1	5	0	1112	-662
1	5	1	3272	3369
1	5	2	552	364
1	5	3	3141	-2666
1	5	4	•	-1196
1	5	5	2662	2250
1	5	6	•	11
1	5	7	•	472
1	5	8	•	-135
1	5	9	1906	-1733
1	5	10	1756	1520
1	5	11	4231	3942
1	5	12	•	-15
1	5	13	3511	-3666
1	5	14	•	354
1	5	15	4220	4195
1	5	16	1211	-1159
1	5	17	4700	-4634
1	5	18	•	-30
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1	5	20	•	-653
1	5	21	3130	-3517
1	5	22	•	-614
1	5	23	•	343
1	5	24	•	37
1	5	25	•	736
1	5	26	•	1023
1	5	27	1930	1041
1	5	28	1352	1162
1	5	29	•	-75
1	5	30	•	-784
1	6	-27	•	-653
1	6	-26	•	-1528
1	6	-25	2640	2557
1	6	-24	•	-1214
1	6	-23	1833	-1855
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1	6	-21	3097	3187
1	6	-20	1352	1470
1	6	-19	3239	-3729
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1	6	-17	3435	3372
1	6	-16	•	369
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1	6	-14	•	125
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1	6	-12	•	956
1	6	-11	1333	-1905
1	6	-10	1315	-1362
1	6	-9	•	406
1	6	-8	•	496
1	6	-7	862	808
1	6	-6	1342	1359
1	6	-5	•	429
1	6	-4	•	-102
1	6	-3	1625	1757
1	6	-2	840	1020
1	6	-1	1625	-1397
1	6	0	•	-331
1	6	1	3871	3559
1	6	2	•	923
1	6	3	4602	-4615
1	6	4	•	915
1	6	5	4340	4517
1	6	6	•	-582
1	6	7	3446	-3598
1	6	8	•	-336
1	6	9	2748	2982
1	6	10	1365	1831
1	6	11	3163	-3164
1	6	12	•	-464
1	6	13	1799	1959
1	6	14	•	910
1	6	15	•	-1072
1	6	16	•	-266
1	6	17	•	443
1	6	18	1429	1256
1	6	19	•	135
1	6	20	•	-344
1	6	21	•	-677
1	6	22	•	-182
1	6	23	916	1365
1	6	24	•	125
1	6	25	1145	-1134
1	6	26	•	692
1	6	27	1505	1706
1	6	28	•	213
1	6	29	•	-1637
1	6	30	•	-169
1	7	-13	•	994
1	7	-12	1254	1175
1	7	-11	2061	-1594
1	7	-10	•	-559
1	7	-9	2530	2351
1	7	-8	1319	1415
1	7	-7	2977	-2872
1	7	-6	•	475
1	7	-5	6360	6546
1	7	-4	3195	2507
1	7	-3	3504	-4136
1	7	-2	4002	-4104
1	7	-1	•	10
1	7	0	•	725
1	7	1	2308	-2370
1	7	2	•	-1565
1	7	3	•	1055
1	7	4	•	735
1	7	5	•	-1125
1	7	6	•	1040
1	7	7	2345	-2256
1	7	8	340	[illegible]
1	7	9	1635	1646
1	7	10	•	1337
1	7	11	1369	-1335
1	7	12	1068	-1421
1	7	13	3097	2867
1	7	14	•	33
1	7	15	2033	-2446
1	7	16	•	-726
1	7	17	2202	1621
1	7	18	•	146
1	7	19	1658	-1561
1	7	20	•	-661
1	7	21	•	1002
1	7	22	•	1175
1	7	23	1170	-1695
1	7	24	•	-720
1	7	25	•	469
1	8	-22	•	909
1	8	-21	•	-1350
1	8	-20	1143	-1501
1	8	-19	1635	1256
1	8	-18	1132	1220
1	8	-17	1273	-1737
1	8	-16	1515	-1441
1	8	-15	1306	1344
1	8	-14	1365	1770
1	8	-13	1047	-1760
1	8	-12	•	-523
1	8	-11	•	1055
1	8	-10	•	195
1	8	-9	•	65
1	8	-8	•	-446
1	8	-7	•	756
1	8	-6	1265	-1452
1	8	-5	•	774
1	8	-4	•	1160
1	8	-3	•	-2070
1	8	-2	1723	-1575
1	8	-1	•	-124
1	8	0	•	-890
1	8	1	2792	-2456
1	8	2	•	1056
1	8	3	1352	1333
1	8	4	2760	2461
1	8	5	•	-1329
1	8	6	2062	-2075
1	8	7	2761	2750
1	8	8	•	241
1	8	9	•	-224
1	8	10	•	-1645
1	8	11	•	1333
1	8	12	2149	1742
1	8	13	1232	-1624
1	8	14	2116	-2726
1	8	15	•	24
1	8	16	•	814
1	9	-7	•	550
1	9	-6	1963	2166
1	9	-5	•	-201
1	9	-4	2454	-2576
1	9	-3	•	587
1	9	-2	2857	2516
1	9	-1	•	-1407
1	9	0	1712	-1517
1	9	1	1298	1525
1	9	2	•	664
1	9	3	•	-84
1	9	4	•	-712
1	9	5	•	168
1	9	6	•	-238
1	9	7	•	622
1	9	8	1102	1023
1	9	9	•	-755
2	0	-40	•	689
2	0	-38	1641	-1728
2	0	-36	•	957
2	0	-34	1907	-1454
2	0	-32	•	535
2	0	-30	•	-364
2	0	-28	2276	2320
2	0	-26	2379	-2295
2	0	-24	•	-53
2	0	-22	•	541
2	0	-20	1595	-1729
2	0	-18	2642	2502
2	0	-16	4610	-4624
2	0	-14	5492	5694
2	0	-12	5594	-5654
2	0	-10	3912	3505
2	0	-8	5590	-5732
2	0	-6	6259	5625
2	0	-4	•	-5138
2	0	-2	•	2067
2	0	0	3516	-3555
2	0	2	5570	-5346
2	0	4	5650	5703
2	0	6	1394	643
2	0	8	10144	10255
2	0	10	6766	-6812
2	0	12	4460	4420
2	0	14	3690	-3790
2	0	16	1363	1750
2	0	18	3292	-3560
2	0	20	2288	2098
2	0	22	•	267
2	0	24	•	583
2	0	26	•	638
2	0	28	1252	-1435
2	0	30	1676	1607
2	0	32	•	-430
2	0	34	•	1501
2	0	36	•	-1200
2	1	-40	•	-231
2	1	-39	•	75
2	1	-38	•	441
2	1	-37	•	-107
2	1	-36	•	-655
2	1	-35	•	-443
2	1	-34	•	613
2	1	-33	•	250
2	1	-32	•	1454
2	1	-31	•	236
2	1	-30	2545	-3430
2	1	-29	•	-325
2	1	-28	3202	2992
2	1	-27	1521	1706
2	1	-26	1321	-1622
2	1	-25	•	-1225
2	1	-24	4418	4712
2	1	-23	573	1445
2	1	-22	3961	-4056
2	1	-21	1564	-1366
2	1	-20	4165	4153
2	1	-19	1637	1675
2	1	-18	2397	-2757
2	1	-17	•	-395
2	1	-16	2606	2541
2	1	-15	1678	-1332
2	1	-14	•	430
2	1	-13	753	-634
2	1	-12	2306	1459
2	1	-11	1637	-1460
2	1	-10	1632	1573
2	1	-9	3173	2915
2	1	-8	5141	-5043
2	1	-7	1073	1026
2	1	-6	6655	6505
2	1	-5	1153	-723
2	1	-4	5102	-5222
2	1	-3	2225	1323
2	1	-2	5824	6450
2	1	-1	2751	2231
2	1	0	4563	-4775
2	1	1	7134	-6566
2	1	2	7784	6150
2	1	3	719	-536
2	1	4	993	-1446
2	1	5	1062	659
2	1	6	5126	3290
2	1	7	3892	3752
2	1	8	3310	-3323
2	1	9	2203	-2022
2	1	10	1689	1413
2	1	11	697	237
2	1	12	1064	-339
2	1	13	1062	536
2	1	14	1598	-1203
2	1	15	1450	-1204
2	1	16	1610	1477
2	1	17	•	31
2	1	18	2397	-2236
2	1	19	•	-441
2	1	20	3452	3632
2	1	21	924	1040
2	1	22	2420	-2239
2	1	23	•	-161
2	1	24	2237	2257
2	1	25	•	149
2	1	26	1575	-1491
2	1	27	•	-890
2	1	28	1221	1057
2	1	29	•	746
2	1	30	890	-589
2	1	31	•	-245
2	1	32	•	1045
2	1	33	•	210
2	2	-29	•	-756
2	2	-28	•	-553
2	2	-27	•	475
2	2	-26	2717	2646
2	2	-25	1724	1988
2	2	-24	•	739
2	2	-23	•	-80
2	2	-22	2443	-2527
2	2	-21	1666	-1780
2	2	-20	•	-243
2	2	-19	993	-536
2	2	-18	1673	-1849
2	2	-17	•	-1333
2	2	-16	4166	4112
2	2	-15	2318	2271
2	2	-14	6609	-6291
2	2	-13	2318	-2189
2	2	-12	4257	4297
2	2	-11	5102	4768
2	2	-10	1106	-1472
2	2	-9	4929	-4763
2	2	-8	3972	4105
2	2	-7	5342	5346
2	2	-6	5593	-5371
2	2	-5	4053	-3977
2	2	-4	4668	4343
2	2	-3	993	-627
2	2	-2	3766	-3331
2	2	-1	731	543
2	2	0	742	721
2	2	1	753	-539
2	2	2	•	15
2	2	3	•	337
2	2	4	3642	-3768
2	2	5	1649	-1984
2	2	6	4066	4211
2	2	7	2888	2333
2	2	8	5536	-5345
2	2	9	2306	-2400
2	2	10	4714	4710
2	2	11	2191	2252
2	2	12	4743	-4956
2	2	13	2313	-1888
2	2	14	3995	3792
2	2	15	2556	2363
2	2	16	3162	-3275
2	2	17	2729	-2535
2	2	18	1175	719
2	2	19	1575	1775
2	2	20	•	50
2	2	21	•	-516
2	2	22	•	591
2	2	23	•	-202
2	3	-31	•	-379
2	3	-30	•	640
2	3	-29	•	1555
2	3	-28	1301	-1292
2	3	-27	1061	-1268
2	3	-26	1404	1442
2	3	-25	2345	3093
2	3	-24	2620	-2770
2	3	-23	4200	-4244
2	3	-22	•	647
2	3	-21	2397	2247
2	3	-20	4053	-4144
2	3	-19	2737	-2714
2	3	-18	2500	2670
2	3	-17	2112	2032
2	3	-16	•	-440
2	3	-15	•	-365
2	3	-14	1552	1131
2	3	-13	•	360
2	3	-12	536	-956
2	3	-11	2100	2021
2	3	-10	•	-471
2	3	-9	1308	-2400
2	3	-8	2625	1663
2	3	-7	2477	2346
2	3	-6	7607	-7603
2	3	-5	3744	-3576
2	3	-4	4169	4053
2	3	-3	1626	2052
2	3	-2	2065	-2574
2	3	-1	4520	-4687
2	3	0	2038	5052
2	3	1	5910	5632
2	3	2	5333	-3255
2	3	3	3218	-3377
2	3	4	759	1043
2	3	5	1675	2066
2	3	6	2581	-2591
2	3	7	2523	-2317
2	3	8	[illegible]	569
2	3	9	2659	2605
2	3	10	1065	606
2	3	11	•	716
2	3	12	•	-527
2	3	13	•	-438
2	3	14	•	-458
2	3	15	1062	1004
2	3	16	•	-229
2	3	17	1100	-542
2	3	18	1326	1659
2	3	19	•	-453
2	3	20	2133	-2042
2	3	21	1572	-1762
2	3	22	1561	1505
2	3	23	1735	1673
2	3	24	1015	-1131
2	3	25	1552	-1379
2	3	26	1073	1075
2	3	27	1746	2090
2	3	28	1073	-1417
2	3	29	•	-336
2	3	30	•	-306
2	4	-22	•	334
2	4	-21	•	-20
2	4	-20	•	-644
2	4	-19	•	14
2	4	-18	•	125
2	4	-17	3367	3358
2	4	-16	1712	-1650
2	4	-15	3950	-3778
2	4	-14	2134	2037
2	4	-13	3070	3093
2	4	-12	879	-905
2	4	-11	3219	-3342
2	4	-10	2316	2544
2	4	-9	4121	4394
2	4	-8	2054	-1934
2	4	-7	5387	-5593
2	4	-6	611	1008
2	4	-5	3573	3451
2	4	-4	1567	-1229
2	4	-3	•	-795
2	4	-2	•	165
2	4	-1	2458	2263
2	4	0	•	254
2	4	1	•	-36
2	4	2	810	-620
2	4	3	1473	-1502
2	4	4	2271	1775
2	4	5	3214	2090
2	4	6	1678	-1699
2	4	7	3047	-2887

they were 0·97 Å from the carbon atoms and made tetrahedral bonds with the C–C bond. The positions of the phenyl hydrogen atoms were calculated at a distance of 1·08 Å from the carbon atoms, assuming them to be in the plane of the phenyl ring.

The eight phenyl hydrogen atoms were allocated isotropic thermal parameters of 0·04 Å^2 and the methyl hydrogen atoms values of 0·06 Å^2; they were included in the structure factor calculations but not refined. The final residual for the 936 observed structure factors was R=0·09. A final electron density distribution was calculated, which had no unusual features.

Final structure factors are given in Table 1; those reflexions which were not observed in the region of reciprocal space examined are marked with a dot. Table 2 gives the heavy atom coordinates and Table 3 their thermal parameters. As a result of the method of data collection, the resolution and the precision of the atomic parameters is somewhat unsymmetrical, being worse in the **a** direction. The hydrogen atom parameters are quoted in Table 4. The numbering of the atoms in the molecule and the arrangement of the molecules in the unit cell viewed along the a axis is shown in Fig. 1. All intermolecular contacts of less than 3·7 Å are shown in Table 5; the bond lengths and angles together with some of the intramolecular non bonded distances are in Table 6. The equations of the ring systems C(1)–C(6), C(7)–C(12), and the group C(10), C(13), C(14) and O calculated with respect to the orthogonal triad a, b, c', where c' is perpendicular to the a and b crystal axes are given in Table 7. Deviations of the atoms from the planes are also given. The angle between the phenyl rings is 38·6° and the angle between the acetyl group and the phenyl ring to which it is attached is 2·9°.

Table 2(a). *Coordinates of the non-hydrogen atoms*

	X/a	Y/b	Z/c
Br	0·3757 (6)	−0·2952 (3)	0·2711 (1)
C(1)	0·0904 (39)	−0·1896 (22)	0·1494 (5)
C(2)	0·2217 (34)	−0·1816 (16)	0·1909 (5)
C(3)	0·1823 (36)	−0·3031 (21)	0·2153 (5)
C(4)	0·0122 (42)	−0·4331 (21)	0·2017 (6)
C(5)	−0·1303 (40)	−0·4330 (22)	0·1594 (5)
C(6)	−0·0853 (41)	−0·3176 (24)	0·1342 (5)
C(7)	0·1324 (35)	−0·0659 (19)	0·1215 (5)
C(8)	0·2091 (42)	−0·0896 (22)	0·0818 (5)
C(9)	0·2675 (38)	0·0292 (20)	0·0562 (5)
C(10)	0·2623 (37)	0·1683 (19)	0·0712 (5)
C(11)	0·1857 (38)	0·1968 (22)	0·1100 (5)
C(12)	0·1203 (39)	0·0783 (20)	0 1355 (5)
C(13)	0 3428 (46)	0·2921 (26)	0·0438 (5)
C(14)	0·3513 (58)	0·4321 (24)	0·0589 (6)
O	0·4145 (53)	0 2646 (22)	0 0056 (6)

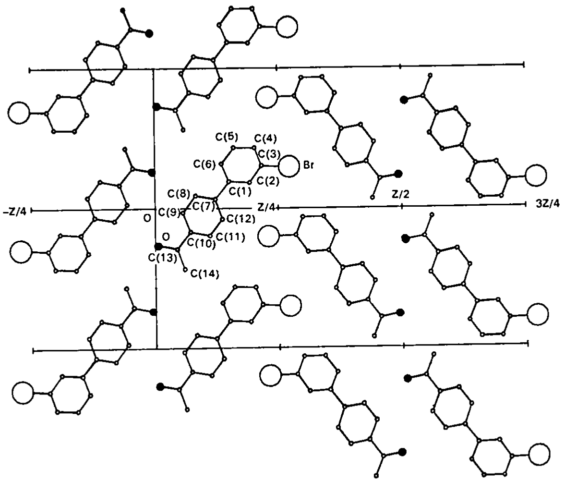

Fig. 1. The arrangement of the molecules in the unit cell viewed along the a axis.

Table 2(b). *Orthogonal coordinates of the non-hydrogen atoms*

	$X'=X+Z\cos\beta$	$Y'=Y$	$Z'=Z\sin\beta$
	X'	Y'	Z'
Br	0·218	−2·675	8·792
C(1)	−0·336	−1·718	4·846
C(2)	−0·015	−1·645	6·193
C(3)	−0·281	−2·746	6·983
C(4)	−0·883	−3·924	6·542
C(5)	−1·245	−3·923	5·171
C(6)	−0·953	−2·878	4·352
C(7)	−0·043	−0·597	3·939
C(8)	0·440	−0·812	2·654
C(9)	0·787	0·265	1·820
C(10)	0·697	1·524	2·308
C(11)	0·218	1·771	3·567
C(12)	−0·155	0·709	4·396
C(13)	1·138	2·647	1·422
C(14)	1·102	3·915	1·910
O	1·595	2·398	0·183

Appendix I gives a table of rotations about the biphenyl bond with the notation of Chawdhury, Hargreaves & Sullivan (1968); this includes other compounds previously investigated.

Discussion

In comparison with earlier investigations on 2′ substituted biphenyls (4-acetyl-2′-fluorobiphenyl, Young, Tollin & Sutherland, 1968; 4-acetyl-2′-chlorobiphenyl, Sutherland & Hoy, 1968; 2-chlorobiphenyl-4-carboxylic acid, Sutherland & Hoy, 1969; 2′-chlorobiphenyl-4-carboxylic acid, Sutherland, 1969) the main feature of

Table 3. *Thermal parameters in Å² for the heavy atoms*

	U_{11}	U_{22}	U_{33}	$2U_{12}$	$2U_{23}$	$2U_{13}$
Br	0·062 (1)	0·061 (1)	0·058 (1)	0·021 (3)	0·025 (2)	0·020 (3)
C(1)	0·023 (9)	0·042 (13)	0·049 (11)	−0·035 (21)	0·034 (14)	−0·018 (18)
C(2)	0·014 (8)	0·009 (9)	0·035 (9)	0·007 (15)	0·012 (12)	0·010 (13)
C(3)	0·018 (8)	0·036 (11)	0·040 (10)	−0·002 (19)	0·035 (13)	0·029 (16)
C(4)	0·030 (10)	0·026 (12)	0·069 (13)	0·016 (21)	0·049 (18)	0·001 (16)
C(5)	0·030 (10)	0·038 (12)	0·047 (11)	−0·044 (20)	0·015 (15)	0·002 (17)
C(6)	0·032 (10)	0·057 (15)	0·035 (10)	0·007 (22)	0·035 (15)	−0·008 (19)
C(7)	0·014 (8)	0·031 (11)	0·026 (9)	−0·009 (17)	0·015 (12)	−0·009 (14)
C(8)	0·036 (10)	0·040 (12)	0·036 (10)	−0·024 (19)	0·011 (15)	0·002 (17)
C(9)	0·026 (9)	0·034 (9)	0·030 (9)	−0·008 (17)	0·020 (13)	0·001 (16)
C(10)	0·027 (9)	0·029 (11)	0·024 (8)	−0·008 (16)	0·003 (13)	−0·016 (15)
C(11)	0·028 (9)	0·039 (12)	0·029 (9)	0·011 (20)	0·022 (13)	−0·010 (17)
C(12)	0·032 (9)	0·033 (12)	0·027 (9)	−0·015 (18)	0·002 (13)	0·016 (16)
C(13)	0·048 (11)	0·054 (14)	0·037 (10)	0·026 (23)	−0·015 (16)	0·024 (22)
C(14)	0·104 (17)	0·033 (13)	0·039 (12)	−0·002 (22)	0·075 (22)	−0·029 (24)
O	0·152 (18)	0·075 (15)	0·066 (12)	0·013 (22)	0·054 (21)	−0·013 (25)

Table 4. *Coordinates of the hydrogen atoms*

H(14), H(15) and H(16) are attached to C(14); the numbering of the remainder corresponds to that of the carbon atom to which they are attached.

	X/a	Y/b	Z/c
H(2)	0·355	−0·081	0·204
H(4)	−0·011	−0·534	0·223
H(5)	−0·275	−0·525	0·147
H(6)	−0·185	−0·325	0·102
H(8)	0·223	−0·199	0·071
H(9)	0·317	0·010	0·024
H(11)	0·176	0·308	0·121
H(12)	0·060	0·099	0·166
H(14)	0·512	0·444	0·090
H(15)	0·099	0·476	0·063
H(16)	0·461	0·512	0·039

Table 5. *Intermolecular distance between heavy atoms less than* 3·6 Å *apart*

i	j	Equipoint indication			d_{ij}
Br	C(12)	$1-x$	$\frac{1}{2}+y$	$\frac{1}{2}-z$	3·587 Å
C(1)	C(6)	$1+x$	y	z	3·528
C(3)	C(4)	$1+x$	y	z	3 533
C(2)	C(5)	$1+x$	y	z	3·664
C(3)	C(5)	$1+x$	y	z	3·661
C(6)	C(8)	$1+x$	y	z	3·691
O	C(13)	$1+x$	y	z	3·672
O	C(9)	$1-x$	$-y$	$-z$	3·664
C(8)	C(7)	$1+x$	y	z	3·675
C(10)	C(11)	$1+x$	y	z	3·669
C(14)	O	$1-x$	$1-y$	$-z$	3·659

Table 7. *The equations of the mean planes of the ring systems* (1), C(1)–C(6); (11), C(7)–C(12) *and* (111), C(10), C(13), C(14) *and* O.

(1) $-0{\cdot}8971X'+0{\cdot}3872Y'+0{\cdot}2125Z'=0{\cdot}6749$
(11) $0{\cdot}9338X'-0{\cdot}0490Y'+0{\cdot}3543Z'=1{\cdot}3827$
(111) $0{\cdot}9305X'-0{\cdot}0986Y'+0{\cdot}3529Z'=1{\cdot}3097$

Deviations from the mean planes in Å

	(1)	(11)	(111)
Br	−0·038		
C(1)	−0·009	0·105	0·257
C(2)	0·017		
C(3)	−0·002		
C(4)	−0·012	0·303	0·563
C(5)	0·022		
C(6)	−0·010		
C(7)	−0·031	0·003	0·099
C(8)		0·008	
C(9)		−0·016	
C(10)	−0·220	0·011	0·003
C(11)		−0·002	
C(12)		−0·005	
C(13)	−0·369	0·054	−0·010
C(14)		0·131	0·004
O		0·054	0·003

the molecule is the regularity around the biphenyl bond. No bond lengths or angles differ by more than one standard deviation from the quoted values (*International Tables for X-ray Crystallography*, 1962; Cruickshank & Sparks, 1960). Steric distortions appear in the region of the bromine atom, the angles of 125·2° (1·5) and 114·5° (1·6) for C(2)–C(3)–C(4) and C(3)–C(4)–C(5) respectively being significant and the angle of the 116·8°(1·3) for C(4)–C(3)–Br being possibly significant. The bromine atom is displaced by 0·0378 Å from the plane of C(1)–C(6) representing an angle of 1·8° at the bond length of 1·878(17) Å. This Br–C(3) bond length is in good agreement with the value of 1·885 Å quoted for σ-bromobenzoic acid (Ferguson & Sim, 1962), and is possibly significantly larger than the quoted value of 1·85(01) Å (*International Tables for X-ray Crystallography*, 1962). Unlike the displacements

Table 6. *Bond lengths and bond angles with e.s.d.'s*

	Length	e.s.d.		Length	e.s.d.
Br —C(3)	1·878 Å	0·017 Å	C(10)–C(11)	1·369 Å	0·023 Å
C(1)–C(2)	1·387	0·023	C(11)–C(12)	1·398	0·024
C(1)–C(6)	1·403	0·025	C(12)–C(7)	1·388	0·022
C(2)–C(3)	1·381	0·022	C(13)–C(14)	1·359	0·029
C(3)–C(4)	1·395	0·025	C(13)–O	1·344	0·028
C(4)–C(5)	1·418	0·026	C(14)–O	2·351	0·029
C(5)–C(6)	1·360	0·026	O——C(9)	2·807	0·025
C(7)–C(8)	1·390	0·023	O——C(10)	2·467	0·025
C(8)–C(9)	1·406	0·024	C(14)–C(10)	2·457	0·026
C(9)–C(10)	1·354	0·023	C(14)–C(11)	2·849	0·027
C(1)–C(7)	1·471	0·023	C(10)–C(13)	1·496	0·025
	Angle	e.s.d.		Angle	e.s.d.
Br —C(3)—C(2)	118·0°	1·2°	C(1)—C(7)—C(8)	121·4°	1·5°
Br —C(3)—C(4)	116·8	1·3	C(1)—C(7)—C(12)	119·9	1·4
C(1)–C(2)—C(3)	118·0	1·4	C(7)—C(8)—C(9)	121·1	1·6
C(2)–C(3)—C(4)	125·2	1·5	C(8)—C(9)—C(10)	118·8	1·5
C(3)–C(4)—C(5)	114·5	1·6	C(9)—C(10)–C(11)	121·5	1·5
C(4)–C(5)—C(6)	121·9	1·7	C(10)–C(11)–C(12)	120·1	1·5
C(5)–C(6)—C(1)	121·2	1·7	C(11)–C(12)–C(7)	119·9	1·5
C(6)–C(1)—C(2)	119·1	1·6	C(12)–C(7)—C(8)	118·5	1·5
C(6)–C(1)—C(7)	120·0	1·6	C(13)–C(10)–C(9)	117·7	1·5
C(2)–C(1)—C(7)	120·8	1·5	C(13)–C(10)–C(11)	120·8	1·5
O—–C(13)–C(14)	120·9	1·9	C(10)–C(13)–O	120·5	1·7
			C(10)–C(13)–C(14)	118·6	1·7

Table 8. *A comparison of the non-planarity of several derivatives of biphenyl*

	φ_1	φ_2	φ_3	φ_4	φ_5
4-Acetyl-3′-bromobiphenyl	38·6°	1·2°	4·1°	2·9°	2·1°
4-Acetyl-2′-chlorobiphenyl	49·2	0·5	2·4	7·9	3·1
4-Acetyl-2′-fluorobiphenyl	50·5	0·7	2·3	2·5	0·5
2′-Chlorobiphenyl-4-carboxylic acid	48·9	0·4	2·9	5·6	7·1
2-Chlorobiphenyl-4-carboxylic acid	46·1	1·7	5·7	7·9	3·2

of the halogen in 2 and 2′ substituted biphenyls, which have been attributed to overcrowding, this displacement is probably caused by crystal forces – the approach of Br–C(12), 3·587 Å, being one of the shorter intermolecular contacts.

The ring systems C(1)–C(6) and C(7)–C(12) are planar within the standard deviations of the atoms, the only possible significantly short bond being C(9)–C(10), 1·354 (23) Å.

The acetyl group shows some indications of disorder *i.e.* the C(13)–O and C(13)–C(14) bonds of 1·344 (28) and 1·359 (29) Å compare with 1·23 and 1·50(1) Å, the quoted values for C O and C–C bonds. Also the standard deviations and thermal parameters of C(14) and O are somewhat higher than those for the remaining non-bromine atoms. A similar effect was present in the crystal structure of 4-acetyl-2′-chlorobiphenyl (Sutherland & Hoy, 1968) but not in 4-acetyl-2′-fluorobiphenyl (Young, Tollin & Sutherland, 1968). The angle between the acetyl group and the phenyl ring to which it is attached is 2·9° compared with 7·9° and 2·5° for 4-acetyl-2′-chlorobiphenyl and 4-acetyl-2′-fluorobiphenyl respectively, which could be caused by relayed steric effects or crystal forces. The acetyl groups form non-bonded dimers across centres of symmetry in the cell, C(14)···O being 3·659 Å. This was also a feature of the structure of 4-acetyl-2′-chlorobiphenyl.

The angle of 38·6° between the phenyl rings can be compared with the values of 21° for 4,4′-diamino-3,3′-dichlorobiphenyl (Chawdhury, Hargreaves & Rizvi, 1968) indicating that crystal forces probably determine the inter-ring angle to a greater extent than steric effects when there are no 2′ substituents.

APPENDIX I

The relative positions of the rings may be described in terms of three angles φ_1,φ_2, φ_3. Starting with the *trans* planar configuration the rings are moved to their observed positions by the following operations: (I) rotation of one ring with respect to the other by φ_1 about C(1)–C(7); (II) rotation of ring C(1)–C(6) by φ_2 about an axis which is in the plane of that ring and passes through C(1) perpendicular to C(1)–C(7); (III) rotation of ring C(7)–C(12) by angle φ_3 about an axis which is in the plane of that ring and passes through C(7) perpendicular to C(1)–C(7). When φ_2 and φ_3 are small φ_1 is within a few minutes of the true interplanar angle. Similarly φ_4 and φ_5 can be used to describe the angle between the plane of the acid/acetyl group and the ring to which it is attached.

Table 8 shows values for previously determined structures together with those for 4-acetyl-3′-bromobiphenyl.

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