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The structure of *p*-bromobenzoic anhydride.

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Crystals of *p*-bromobenzoic anhydride, obtained by hydrolysis of the acid chloride and crystallized from ethanol, are colourless plates with (100) developed and smaller (010) and (001) faces. Crystal data were determined from various rotation, Weissenberg, and precession films (λ(Cu $K\alpha$) = 1·5418 Å, λ(Mo $K\alpha$) = 0·7107 Å):

$(Br.C_6H_4.CO)_2O$; M.W. 384·0; m.p. 220 °C.
Monoclinic, $a = 28{\cdot}04$, $b = 5{\cdot}91$, $c = 3{\cdot}97$ Å; $\beta = 94{\cdot}8°$.
$U = 655{\cdot}6$ Å^3. D_m (flotation in aqueous $AgNO_3$) = 1·9, $Z = 2$, $D_x = 1{\cdot}94$ g.cm^{-3}.
Absorption coefficients for X-rays: μ(Cu $K\alpha$) = 87 cm^{-1}, μ(Mo $K\alpha$) = 65 cm^{-1}.
$F(000) = 372$.

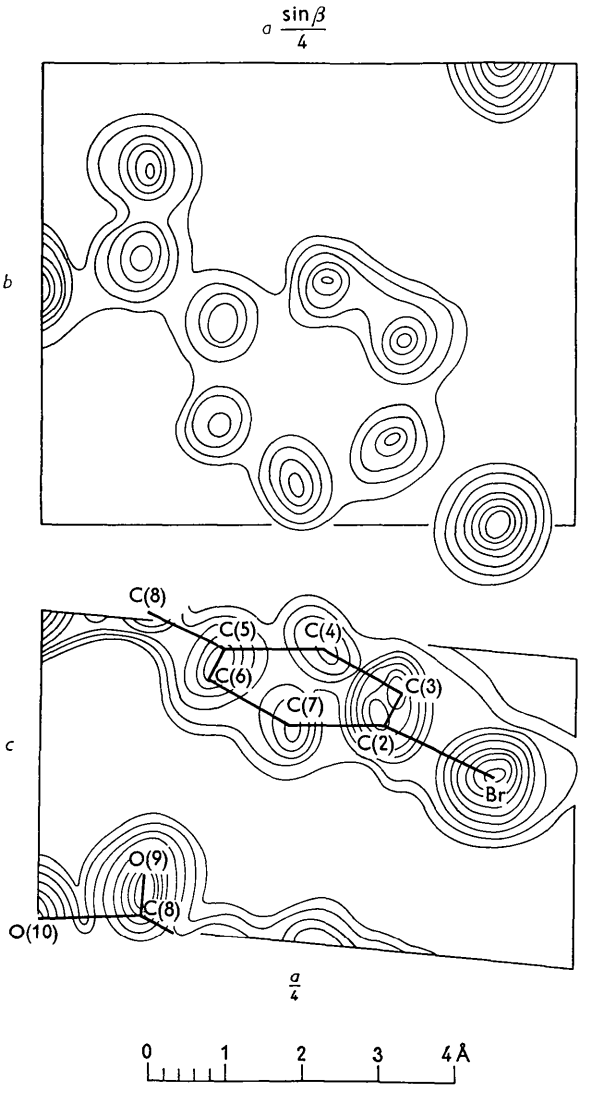

Fig. 1. Electron-density projections along *c* and *b*.

Absent spectra: *hkl* when $h+k$ is odd. Space group is *C*2, *Cm* or *C*2/*m*. *C*2 was confirmed by the structure analysis.

The intensities of the *h*0*l* and *hk*0 reflexions were measured visually from Weissenberg films of crystals having cross-sections 0·3 × 0·05 mm perpendicular to the rotation axes. No corrections for absorption were applied, so that there are some inaccuracies in the measured structure amplitudes. The structure was determined from Patterson, electron-density (Fig. 1), and difference projections along *b* and *c*, all of which indicated space group *C*2. The final positional parameters are listed in Table 1, together with displacements of the atoms from the plane through all the carbon and bromine atoms, which has equation:

$$0{\cdot}098X' - 0{\cdot}348Y + 0{\cdot}932Z' - 2{\cdot}712 = 0 \qquad (1)$$

(X', Y, Z' are coordinates in Å referred to orthogonal axes *a*, *b*, and *c**), and displacements from the plane through C(5), C(8), O(9), O(10), which has equation:

$$0{\cdot}024X' - 0{\cdot}464Y + 0{\cdot}885Z' - 2{\cdot}056 = 0\,. \qquad (2)$$

Table 1. *Positional parameters (fractional) and deviations* (Å) *from planes* (1) *and* (2)

Atom	x	y	z	Δ_1	Δ_2
Br(1)	0·209	0·000	0·585	0	—
C(2)	0·162	0·173	0·715	+0·01	—
C(3)	0·167	0·401	0·843	−0·01	—
C(4)	0·128	0·540	0·949	0	—
C(5)	0·084	0·458	0·934	+0·01	−0·01
C(6)	0·079	0·229	0·817	−0·02	—
C(7)	0·166	0·089	0·700	+0·02	—
C(8)	0·048	0·591	1·036	+0·01	+0·03
O(9)	0·049	0·784	1·197	—	−0·01
O(10)	0	0·522	1	—	−0·01

The final R indices are 0·25 for 134 *h*0*l* reflexions, and 0·23 for 93 *hk*0 reflexions; a large part of these residuals is a result of absorption and extinction errors, F_o for the intense reflexions being usually considerably smaller than F_c, and F_o for the high-angle reflexions being larger than F_c (Table 2). Since our interest was in the general shape of the molecule, and not in detailed values of the bond distances and angles, no corrections were made for these effects and no three-dimensional work was carried out.

The molecule has symmetry C_2, the twofold molecular axis coinciding with the diad axis of the space group. The angle between the planes of the carboxyl groups is 55°, so that the groups are each twisted about 28°, in opposite directions, from a planar arrangement; this twisting increases the O · · · O intramolecular separation of about 2·4 Å which would pertain in a planar molecule to 3·04 Å. In addition the six-membered rings are twisted 8° out of the planes of their respective

Table 2. *Measured and calculated structure amplitudes*

H	K	L	F OBS	F CALC
2	0	0	49.9	55.3
4	0	0	10.4	13.0
6	0	0	0.0	5.5
8	0	0	51.9	68.9
10	0	0	38.7	54.8
12	0	0	63.1	71.3
14	0	0	53.7	59.8
16	0	0	32.1	26.0
18	0	0	24.4	20.5
20	0	0	19.7	14.8
22	0	0	24.7	20.6
24	0	0	43.6	44.7
26	0	0	13.0	11.7
28	0	0	14.1	1.5
30	0	0	4.9	4.1
32	0	0	9.1	5.6
34	0	0	3.5	3.0
-34	0	1	12.2	4.6
-32	0	1	10.2	4.1
-30	0	1	4.2	3.9
-28	0	1	5.5	4.9
-26	0	1	23.1	6.8
-24	0	1	1.8	0.0
-22	0	1	50.2	53.2
-20	0	1	40.1	37.3
-18	0	1	11.1	10.3
-16	0	1	14.4	4.3
-14	0	1	51.7	49.2
-12	0	1	52.9	60.8
-10	0	1	65.5	68.9
- 8	0	1	61.6	80.4
- 6	0	1	5.5	13.3
- 4	0	1	15.8	22.6
- 2	0	1	35.5	42.4
0	0	1	13.7	18.8
2	0	1	154.6	200.0
4	0	1	116.3	119.3
6	0	1	25.2	6.6
8	0	1	18.5	20.1
10	0	1	39.6	56.2
12	0	1	54.6	65.6
14	0	1	17.9	26.1
16	0	1	56.3	53.1
18	0	1	20.8	13.3
20	0	1	21.8	10.6
22	0	1	23.0	8.0
24	0	1	23.4	14.0
26	0	1	30.6	19.3
28	0	1	25.6	12.7
30	0	1	10.8	4.0
32	0	1	1.8	0.9
34	0	1	5.1	3.5
-32	0	2	17.7	4.8
-30	0	2	21.8	7.0
-28	0	2	2.8	1.8
-26	0	2	5.5	4.7
-24	0	2	18.2	10.3
-22	0	2	8.0	1.5
-20	0	2	25.6	31.7
-18	0	2	28.0	39.3
-16	0	2	8.9	11.6
-14	0	2	8.9	2.7
-12	0	2	25.6	28.4
-10	0	2	74.0	70.4
- 8	0	2	25.7	36.3
- 6	0	2	60.3	63.0
- 4	0	2	12.5	16.9
- 2	0	2	7.1	1.0
0	0	2	23.9	24.9
2	0	2	36.6	39.3
4	0	2	54.9	68.6
6	0	2	56.7	60.3
8	0	2	27.1	31.4
10	0	2	8.5	5.4
12	0	2	22.2	20.1
14	0	2	39.8	47.2
16	0	2	6.3	14.3
18	0	2	18.5	21.9
20	0	2	10.8	13.9
22	0	2	4.8	2.6
24	0	2	10.7	5.4
26	0	2	9.8	7.7
28	0	2	4.4	6.5
30	0	2	7.3	4.4
-30	0	3	13.5	4.1
-28	0	3	18.5	5.0
-26	0	3	4.5	2.9
-24	0	3	2.7	3.1
-22	0	3	9.6	8.2
-20	0	3	10.0	6.5
-18	0	3	15.3	17.8
-16	0	3	17.5	17.1
-14	0	3	7.8	12.6
-12	0	3	5.7	3.2
-10	0	3	9.9	10.2
- 8	0	3	32.6	41.9
- 6	0	3	29.2	28.5
- 4	0	3	28.9	23.2
- 2	0	3	17.0	17.7
0	0	3	5.4	4.0
2	0	3	15.2	17.8
4	0	3	26.1	24.2
6	0	3	22.4	31.6
8	0	3	18.8	15.8
10	0	3	18.8	17.7
12	0	3	1.8	6.2
14	0	3	4.5	8.4
16	0	3	9.8	18.7
18	0	3	8.6	9.1
20	0	3	11.4	6.9
22	0	3	11.6	6.0
24	0	3	4.4	3.3
26	0	3	6.3	3.6
-24	0	4	3.6	1.0
-22	0	4	0.0	0.8
-20	0	4	5.4	4.6
-18	0	4	6.3	5.6
-16	0	4	7.7	11.1
-14	0	4	7.6	5.7
-12	0	4	4.8	3.2
-10	0	4	0.0	4.2
- 8	0	4	5.1	4.2
- 6	0	4	17.6	16.5
- 4	0	4	17.7	14.1
- 2	0	4	9.9	10.0
0	0	4	7.8	5.9
2	0	4	7.8	7.5
4	0	4	7.5	6.8
6	0	4	13.5	12.6
8	0	4	13.0	11.9
10	0	4	12.0	5.7
12	0	4	2.1	4.0
14	0	4	0.0	2.2
16	0	4	0.0	2.0
18	0	4	3.9	5.5
- 6	0	5	1.5	0.9
- 4	0	5	4.1	5.8
- 2	0	5	4.1	3.5
0	0	5	2.2	4.1
1	1	0	19.8	12.3
3	1	0	88.0	107.5
5	1	0	80.3	103.3
7	1	0	90.2	105.5
9	1	0	77.1	71.1
11	1	0	56.0	48.6
13	1	0	31.1	38.5
15	1	0	31.6	32.1
17	1	0	40.6	47.1
19	1	0	44.2	43.7
21	1	0	41.7	34.2
23	1	0	7.9	4.6
25	1	0	5.2	6.5
27	1	0	9.4	5.6
29	1	0	15.9	10.4
31	1	0	18.0	9.9
33	1	0	12.0	5.7
0	2	0	96.8	109.2
2	2	0	84.6	83.4
4	2	0	30.1	38.8
6	2	0	10.3	7.5
8	2	0	6.0	18.0
10	2	0	62.9	79.1
12	2	0	64.0	61.7
14	2	0	56.7	50.0
16	2	0	20.3	17.5
18	2	0	4.7	5.2
20	2	0	9.2	9.5
22	2	0	17.0	17.0
24	2	0	30.7	22.4
26	2	0	17.8	14.5
28	2	0	12.5	6.4
30	2	0	6.2	1.3
32	2	0	3.0	0.9
34	2	0	8.6	5.4
1	3	0	19.8	12.4
3	3	0	37.4	45.2
5	3	0	63.1	64.2
7	3	0	65.5	59.7
9	3	0	38.2	40.3
11	3	0	7.9	16.2
13	3	0	11.8	16.3
15	3	0	12.7	22.5
17	3	0	14.8	30.1
19	3	0	16.1	24.3
21	3	0	15.5	15.9
23	3	0	9.4	1.7
25	3	0	2.8	3.2
27	3	0	12.0	5.1
29	3	0	13.3	8.9
31	3	0	12.2	6.1
0	4	0	45.9	48.6
2	4	0	24.3	27.7
4	4	0	25.1	25.4
6	4	0	5.4	9.6
8	4	0	15.7	19.1
10	4	0	16.7	32.0
12	4	0	50.5	33.8
14	4	0	28.3	31.4
16	4	0	4.3	13.7
18	4	0	3.0	6.5
20	4	0	6.0	6.9
22	4	0	9.0	6.5
24	4	0	15.2	11.2
26	4	0	11.2	5.7
28	4	0	6.4	4.0
30	4	0	2.8	1.5
1	5	0	9.4	11.5
3	5	0	16.7	17.9
5	5	0	28.1	27.0
7	5	0	28.8	27.3
9	5	0	15.2	2.0
11	5	0	5.2	9.9
13	5	0	14.4	3.2
15	5	0	5.2	4.6
17	5	0	13.7	12.7
19	5	0	13.3	13.2
21	5	0	13.3	6.1
23	5	0	3.9	4.0
25	5	0	9.0	1.5
0	6	0	8.8	9.0
2	6	0	16.5	13.8
4	6	0	4.1	9.0
6	6	0	4.9	5.0
8	6	0	4.1	4.8
10	6	0	13.3	9.4
12	6	0	10.1	7.4
14	6	0	10.5	9.3
16	6	0	3.2	5.0
18	6	0	2.1	0.4
20	6	0	6.7	3.7
1	7	0	0.0	0.7
3	7	0	0.0	5.5
5	7	0	10.3	7.1
7	7	0	8.2	5.2

carboxyl groups, these displacements being in the opposite sense to the carboxyl-group twists. The bond distances, valency angles and intermolecular separations have not been accurately determined, but all apppear to be normal.

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