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The Crystal Structure of γ-Hydroquinone

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γ-Hydroquinone is monoclinic, $P2_1/c$, $a=8\cdot07$, $b=5\cdot20$, $c=13\cdot20$ Å, $\beta=107°$. The unit cell contains 4 molecules, two and two not dependent on space-group symmetry. The centres of the benzene rings are situated at the crystallographic centres of symmetry $(0,0,0)$, $(0,\frac{1}{2},\frac{1}{2})$ and $(\frac{1}{2},0,\frac{1}{2})$, $(\frac{1}{2},\frac{1}{2},0)$. The hydrogen bonds form helices around the screw axes. The O–O distances in the hydrogen bonds are 2·84 Å.

Introduction

The present investigation of γ-hydroquinone, the monoclinic modification, has been undertaken in order to study the arrangement of the hydrogen bonding, to determine the deviation of the C–C–OH angle from 120° (Hirshfeld, 1964), and to look for possible distortions of the benzene rings due to the influence of the hydroxyl groups (Maartmann-Moe, 1965).

Kitaigorodskii (1945) has considered the structure of the γ modification from the point of view of packing, but it does not appear from his paper that a complete X-ray analysis has been attempted.

Experimental

Caspari (1927) obtained the monoclinic modification of hydroquinone by sublimation, and determined the cell dimensions and space group. Likewise Kitaigorodskii (1945) produced his crystal samples by sublimation.

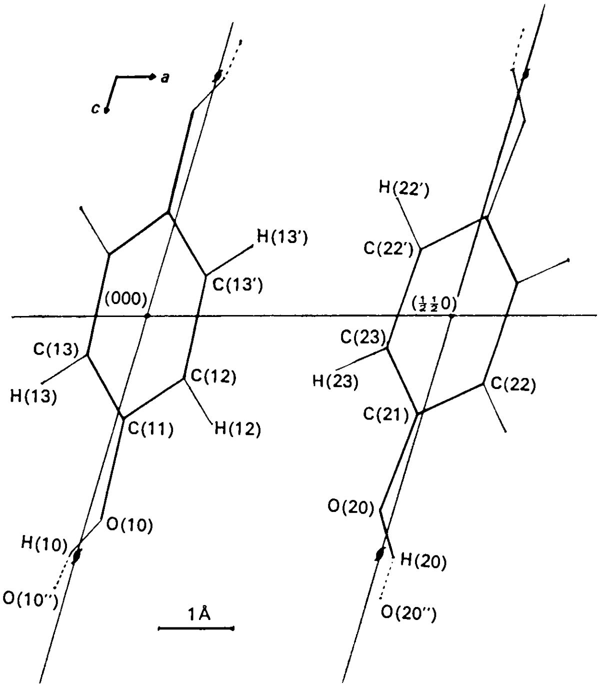

Fig. 1. Atomic positions projected along [010]. O(10'') and O(20'') indicate the oxygen atom positions generated by the screw axes. The planes of the two molecules are inclined to each other by 111·8°.

The author found that the γ modification sometimes crystallizes from an evaporating ether solution, and all measurements in this investigation have been done on such samples. The condition under which the γ modification is formed from ether solution seems to be rapid evaporation at room temperature.

Suitable crystals were rare since the desired modification tends to crystallize as extremely thin plates. The sample used for the recording of the intensities was cut from an exceptionally thick crystal.

Since the crystals have a pronounced cleavage, the author has succeeded in cutting and mounting crystals with only one orientation. The crystal data and intensities are therefore obtained by oscillation and Weissenberg photographs attainable with this orientation only. With the notation used below, the cleavage is parallel to the plane (100), and the crystals were mounted with the *b* axis parallel to the axis of oscillation.

The crystals used for the recording of the intensities had a cross-section of about 0·1 × 0·1 mm². *h0l*, *h1l*, and *h2l* were recorded at room temperature on integrated Weissenberg films with the use of the multiple film technique. All recordings were done with Cu $K\alpha$ radiation. The intensities were measured with a photometer and converted to $|F_{\text{obs}}|^2$ by applying the usual correction for equi-inclination recordings; absorption and extinction effects were neglected. The intensity material includes 594 reflexions; of these 186 were too weak to be measured.

Crystal data

Monoclinic $P2_1/c$ (*b* axis unique)

	a	*b*	*c*	*β*
Caspari (1927)	8·11	5·20	13·24 Å	107°
Kitaigorodskii (1947)	8·06	5·15	13·30	107
Present	8·07	5·20	13·20	107

Density
Experimental (Caspari) 1·325 g.cm⁻³
Calculated 1·380 g.cm⁻³
Molecules per unit cell 4

The cell dimensions (present) are estimated to be accurate to within 0·5%.

Structure analysis

Reflexions having indices $h+l$ odd are consistently much weaker than those with $h+l$ even; this is most apparent for the reflexions of type $h0l$, and Kitaigorodskii (1945) concludes from his consideration of pseudo-symmetry and possible packing arrangements that the axis of the molecules – the O–O line – is parallel to the c axis and that the centres of the benzene rings are situated at the crystallographic centres of symmetry $(0,0,0)$, $(0,\frac{1}{2},\frac{1}{2})$ and $(\frac{1}{2},\frac{1}{2},0)$, $(\frac{1}{2},0,\frac{1}{2})$.

A Patterson synthesis of the (010) projection was consistent with Kitaigorodskii's conclusion but no more information could be extracted from it. Assuming the centres of the molecules to be on the crystallographic centres of symmetry, as suggested by Kitaigorodskii, and the angle between the b axis and the normals to the planes of the benzene rings to be about

Table 1. *Experimental and calculated structure amplitudes*

h	k	l	F_o	F_c
0	0	2	23.72	23.57
0	0	4	12.07	10.75
0	0	6	35.60	-36.99
0	0	8	20.33	-20.15
0	0	10	14.86	14.39
0	0	12	14.78	-16.01
0	0	14	2.05	1.96
0	0	16	7.14	-6.44
1	0	-16	<1.38	.79
1	0	-14	<2.06	.68
1	0	-12	<2.12	-1.68
1	0	-10	3.06	-3.52
1	0	-8	<1.83	.69
1	0	-6	<1.49	-.50
1	0	-4	<1.17	-1.00
1	0	-2	5.28	4.96
1	0	0	< .71	.43
1	0	2	7.47	-7.42
1	0	4	6.14	6.18
1	0	6	11.42	-11.65
1	0	8	4.04	3.99
1	0	10	8.27	-8.97
1	0	12	3.76	-3.88
1	0	14	<1.79	.44
2	0	-16	2.38	-3.64
2	0	-14	<2.21	2.35
2	0	-12	23.61	-26.35
2	0	-10	8.77	7.56
2	0	-8	30.51	-31.34
2	0	-6	39.13	-38.84
2	0	-4	10.55	10.11
2	0	-2	1.23	-1.85
2	0	0		117.23
2	0	2	21.96	-22.13
2	0	4	2.67	2.85
2	0	6	10.16	-9.38
2	0	8	7.54	6.92
2	0	10	23.31	24.18
2	0	12	<2.14	-1.31
2	0	14	<1.53	.40
3	0	-16	<1.49	.34
3	0	-14	<2.12	-.82
3	0	-12	<2.33	1.50
3	0	-10	3.72	4.18
3	0	-8	3.43	-2.85
3	0	-6	11.01	10.96
3	0	-4	8.82	-8.88
3	0	-2	11.90	11.85
3	0	0	<1.32	.65
3	0	2	7.25	-7.04
3	0	4	11.71	11.66
3	0	6	6.61	-7.20
3	0	8	7.93	7.82
3	0	10	6.43	-6.84
3	0	12	3.10	-3.20
3	0	14	<1.00	-.50
4	0	-16	2.09	-3.05
4	0	-14	<2.06	2.41
4	0	-12	17.93	-19.36
4	0	-10	15.31	15.55
4	0	-8	7.75	-7.43
4	0	-6	12.00	-11.82
4	0	-4	2.18	-2.14
4	0	-2	30.72	-30.87
4	0	0	16.46	16.47
4	0	2	30.70	-30.79
4	0	4	8.53	-7.73
4	0	6	<2.21	-.05
4	0	8	14.60	14.92
4	0	10	18.78	20.10
4	0	12	1.64	2.53
5	0	-14	<1.99	-2.12
5	0	-12	3.93	4.09
5	0	-10	6.94	7.28
5	0	-8	7.07	-7.02
5	0	-6	7.06	7.36
5	0	-4	11.18	-11.60
5	0	-2	8.97	8.94
5	0	0	<2.02	-.80
5	0	2	5.74	-5.40
5	0	4	8.04	8.15
5	0	6	<2.33	-1.01
5	0	8	8.27	8.70
5	0	10	2.09	-1.87
6	0	-14	<1.81	1.49
6	0	-12	3.46	-3.05
6	0	-10	23.33	25.52
6	0	-8	12.53	12.50
6	0	-6	2.01	-2.18
6	0	-4	10.46	-10.19
6	0	-2	21.22	-20.55
6	0	0	2.52	-1.84
6	0	2	15.30	-14.99
6	0	4	6.90	-6.97
6	0	6	<2.21	.19
6	0	8	6.79	7.19
7	0	-14	<1.51	-1.20
7	0	-12	4.26	4.62
7	0	-10	4.16	4.49
7	0	-8	8.66	-8.90
7	0	-6	<2.33	1.42
7	0	-4	6.81	-7.30
7	0	-2	5.66	5.37
7	0	0	<2.33	-1.46
7	0	2	4.70	-4.53
7	0	4	2.55	2.69
7	0	6	<1.87	.36
7	0	8	5.80	5.55
8	0	-12	3.70	4.14
8	0	-10	17.50	19.69
8	0	-8	10.86	11.38
8	0	-6	<2.23	-1.52
8	0	-4	6.02	-5.44
8	0	-2	4.93	-4.79
8	0	0	3.09	2.74
8	0	2	<2.04	-1.06
8	0	4	2.13	-2.42
9	0	-10	<1.54	1.54
9	0	-8	5.35	-6.36
9	0	-6	<1.94	-.02
9	0	-4	3.20	-2.11
9	0	-2	3.49	3.51
9	0	0	<1.74	-1.14
9	0	2	3.28	-3.86
10	0	-8	3.58	2.51
10	0	-6	4.27	-2.80
10	0	-4	3.58	-1.38
10	0	-2	4.44	3.53
0	1	2	9.23	-9.52
0	1	3	1.67	1.29
0	1	4	4.12	-4.12
0	1	5	1.49	-1.06
0	1	6	14.26	14.01
0	1	7	<1.98	-.34
0	1	8	15.78	16.23
0	1	9	2.68	2.40
0	1	10	15.46	15.87
0	1	11	6.72	-6.56
0	1	12	9.37	9.77
0	1	13	2.54	2.26
0	1	14	<2.16	.22
0	1	15	2.41	-2.00
1	1	-15	3.22	.83
1	1	-14	<2.27	.15
1	1	-13	5.31	-7.90
1	1	-12	2.72	2.41
1	1	-11	4.31	-4.59
1	1	-10	3.96	4.40
1	1	-9	16.33	19.78
1	1	-8	4.23	3.10
1	1	-7	36.88	-39.28
1	1	-6	4.83	4.68
1	1	-5	15.76	15.40
1	1	-4	2.30	-2.27
1	1	-3	29.14	-32.88
1	1	-2	1.58	-1.08
1	1	-1	36.14	64.66
1	1	0	1.68	1.31
1	1	1	38.04	57.61
1	1	2	1.91	1.54
1	1	3	29.60	-33.22
1	1	4	4.99	5.01
1	1	5	7.97	7.42
1	1	6	<1.90	-.48
1	1	7	27.60	-27.46
1	1	8	<2.28	.54
1	1	9	22.39	22.43
1	1	10	<2.57	-.96
1	1	11	2.59	2.34
1	1	12	<2.49	-1.04
1	1	13	4.53	-4.58
1	1	14	<1.95	-.68
1	1	15	3.53	-1.48
2	1	-15	3.24	-.68
2	1	-14	<2.33	-.13
2	1	-13	<2.52	.21
2	1	-12	<1.64	-1.88
2	1	-11	<2.56	.19
2	1	-10	<2.45	.04
2	1	-9	<2.28	-.43
2	1	-8	8.80	8.13
2	1	-7	<1.94	.79
2	1	-6	8.79	8.39
2	1	-5	2.09	1.70
2	1	-4	2.96	-3.06
2	1	-3	4.47	-3.92
2	1	-2	33.63	-39.54
2	1	-1	13.15	13.76
2	1	0	42.70	-59.30
2	1	1	14.46	-15.72
2	1	2	32.31	-35.49
2	1	3	6.50	6.14
2	1	4	9.65	-8.67
2	1	5	3.11	-3.01
2	1	6	13.04	13.04
2	1	7	<2.27	.33
2	1	8	15.66	15.76
2	1	9	4.15	3.98
2	1	10	13.00	12.89
2	1	11	8.20	-8.09
2	1	12	6.17	5.72
2	1	13	2.59	2.40
2	1	14	<1.60	-.24
3	1	-15	<1.57	1.39
3	1	-14	<2.33	.09
3	1	-13	9.63	-9.75
3	1	-12	2.46	2.51
3	1	-11	4.60	-4.44
3	1	-10	4.11	4.40
3	1	-9	20.01	20.38
3	1	-8	<2.18	1.20
3	1	-7	31.30	-32.15
3	1	-6	<1.88	.39
3	1	-5	8.18	7.77
3	1	-4	4.82	-4.57
3	1	-3	27.25	-29.27
3	1	-2	2.93	-2.91
3	1	-1	29.25	33.39
3	1	0	<1.56	.80
3	1	1	20.23	22.56
3	1	2	<1.75	.01
3	1	3	24.92	-25.25
3	1	4	4.21	4.07
3	1	5	<2.17	.29
3	1	6	2.65	2.45
3	1	7	9.61	-9.86
3	1	8	3.67	3.73
3	1	9	18.74	18.47
3	1	10	3.11	2.85
3	1	11	5.04	5.21
3	1	12	<2.07	1.36
3	1	13	2.93	-2.50
4	1	-15	5.26	.45
4	1	-14	<2.27	.91
4	1	-13	2.35	-1.95
4	1	-12	8.09	7.83
4	1	-11	6.76	6.79
4	1	-10	15.06	15.73
4	1	-9	4.35	-4.09
4	1	-8	21.99	21.51
4	1	-7	<2.18	.99
4	1	-6	11.81	11.17
4	1	-5	1.67	1.87
4	1	-4	8.16	-7.98
4	1	-3	4.93	-4.67
4	1	-2	24.28	-24.43
4	1	-1	15.14	15.35
4	1	0	25.30	-25.40
4	1	1	13.73	-14.32
4	1	2	12.81	-11.88
4	1	3	6.05	6.09
4	1	4	4.20	-4.15
4	1	5	<2.40	-1.86
4	1	6	3.18	2.69
4	1	7	2.16	2.10
4	1	8	2.00	1.60
4	1	9	3.96	3.89
4	1	10	<2.31	-.54
4	1	11	5.91	-5.76
4	1	12	1.77	-1.56
5	1	-15	<1.82	-.45
5	1	-14	<2.16	.82
5	1	-13	6.98	-6.74
5	1	-12	<2.53	1.16
5	1	-11	2.83	2.82
5	1	-10	<2.58	1.06
5	1	-9	19.67	20.78
5	1	-8	2.68	-2.56
5	1	-7	14.62	-13.89
5	1	-6	2.50	-2.35
5	1	-5	<2.21	.81
5	1	-4	2.57	-2.43
5	1	-3	18.49	-18.61
5	1	-2	<2.12	-1.33
5	1	-1	6.96	6.78
5	1	0	<2.20	-.30
5	1	1	3.67	2.83
5	1	2	3.94	-2.25
5	1	3	13.69	-13.18
5	1	4	<2.52	-.19
5	1	5	<2.16	-1.50
5	1	6	<2.59	.25
5	1	7	<2.54	-.56
5	1	8	<2.42	2.03
5	1	9	6.01	9.95
5	1	10	2.33	2.49
5	1	11	2.56	3.22
6	1	-15	<1.57	1.63
6	1	-14	<1.95	.55
6	1	-13	2.09	-2.43
6	1	-12	7.96	7.58
6	1	-11	7.91	8.19
6	1	-10	11.11	11.24
6	1	-9	5.70	-5.87
6	1	-8	10.73	10.31
6	1	-7	<2.53	-.67
6	1	-6	2.61	2.62
6	1	-5	<2.45	0.00
6	1	-4	2.02	-1.94
6	1	-3	3.15	-3.03
6	1	-2	<2.42	-.66
6	1	-1	9.53	9.38
6	1	0	<1.57	1.91
6	1	1	9.23	-8.92
6	1	2	4.80	4.67
6	1	3	3.06	2.84
6	1	4	1.82	1.45
6	1	5	<2.54	-.44
6	1	6	2.54	-2.51
6	1	7	3.04	2.78
6	1	8	5.90	-6.06
6	1	9	2.61	2.90
7	1	-14	<1.67	.90
7	1	-13	3.23	-3.08
7	1	-12	<2.17	-.72
7	1	-11	6.36	6.17
7	1	-10	<2.46	-1.27
7	1	-9	15.52	15.61
7	1	-8	2.62	-2.15
7	1	-7	2.59	-2.75
7	1	-6	<2.59	-.17
7	1	-5	2.83	-2.51
7	1	-4	<2.59	.81
7	1	-3	8.79	-8.60
7	1	-2	<2.59	.40
7	1	-1	<2.46	1.90
7	1	0	<2.59	-.24
7	1	1	<2.30	2.04
7	1	2	<2.28	-2.25
7	1	3	4.99	-4.63
7	1	4	<2.12	-2.04
7	1	5	2.55	-2.54
7	1	6	2.01	-1.71
7	1	7	<1.71	-.31
7	1	8	<1.27	-.56
8	1	-13	<1.46	-1.45
8	1	-12	<1.78	-.23
8	1	-11	6.03	6.26
8	1	-10	2.38	-2.09
8	1	-9	4.72	-4.88
8	1	-8	2.61	-2.87
8	1	-7	2.18	-1.92
8	1	-6	2.21	-1.80
8	1	-5	<2.50	-.49
8	1	-4	3.07	2.50
8	1	-3	<2.62	-.27
8	1	-2	7.59	7.32
8	1	-1	4.99	4.81
8	1	0	8.74	8.65
8	1	1	5.34	-5.21
8	1	2	6.63	6.66
8	1	3	<2.06	-.32
8	1	4	<1.56	1.53
8	1	5	<1.58	-.38
9	1	-11	3.49	4.39
9	1	-10	<1.65	-.86
9	1	-9	7.26	7.85
9	1	-8	<1.95	.15
9	1	-7	<2.04	-1.19
9	1	-6	<1.87	1.75
9	1	-5	3.69	-3.76
9	1	-4	<1.89	2.15
9	1	-3	2.30	-2.19
9	1	-2	<1.29	1.30
9	1	-1	3.51	3.57
9	1	0	<1.87	.32
9	1	1	3.69	3.75
9	1	2	<1.51	-1.05
9	1	3	<1.19	-.85
0	2	1	1.77	-.79
0	2	2	40.82	-55.91
0	2	3	2.54	1.11
0	2	4	8.40	-8.94
0	2	5	1.21	1.15
0	2	6	5.29	-5.03
0	2	7	3.42	-2.92
0	2	8	9.10	8.23
0	2	9	<1.93	-.27
0	2	10	28.37	29.23
0	2	11	3.68	-3.36
0	2	12	8.00	-8.17
0	2	13	<1.79	-.15
0	2	14	<1.54	1.82
0	2	15	<1.13	-.88
1	2	-15	<1.57	.74
1	2	-14	<1.65	.73
1	2	-13	2.51	-2.65
1	2	-12	<2.00	.41
1	2	-11	9.30	-9.16
1	2	-10	<2.00	1.18
1	2	-9	4.19	-4.06
1	2	-8	2.59	2.47
1	2	-7	1.92	-.77
1	2	-6	<1.46	.36
1	2	-5	<1.31	1.30
1	2	-4	1.77	-1.52
1	2	-3	1.77	-.57
1	2	-2	1.92	1.42
1	2	-1	13.74	-14.41
1	2	0	.89	-.41
1	2	1	9.97	-11.38
1	2	2	5.97	-6.14
1	2	3	14.04	-14.46
1	2	4	5.87	6.14
1	2	5	1.91	-1.79
1	2	6	3.41	-3.37
1	2	7	11.06	10.86
1	2	8	9.73	10.23
1	2	9	11.79	11.76
1	2	10	<2.04	-.22
1	2	11	10.26	10.46
1	2	12	<1.88	1.00
1	2	13	1.74	1.95
1	2	14	<1.33	1.50
2	2	-15	<1.39	-.28
2	2	-14	2.28	2.52
2	2	-13	<1.90	0.00
2	2	-12	5.49	-4.90
2	2	-11	5.27	4.63
2	2	-10	36.30	38.75
2	2	-9	<1.90	1.61
2	2	-8	13.08	13.09
2	2	-7	2.74	2.19
2	2	-6	3.12	-2.66
2	2	-5	<1.37	-.64
2	2	-4	8.21	-6.66
2	2	-3	2.72	-1.69
2	2	-2	32.45	-37.48
2	2	-1	2.66	1.89
2	2	0	32.30	38.45
2	2	1	<1.13	.53
2	2	2	23.70	-25.23
2	2	3	1.97	1.19
2	2	4	5.22	-4.10
2	2	5	2.45	2.15
2	2	6	12.62	-11.85
2	2	7	<1.89	-.56
2	2	8	3.51	-4.20
2	2	9	2.96	2.47
2	2	10	9.29	9.34
2	2	11	<1.91	-.26
2	2	12	10.64	-9.98
2	2	13	<1.42	.49
3	2	-15	<1.40	-1.83
3	2	-14	<1.70	.06
3	2	-13	1.79	1.22
3	2	-12	<2.00	-.03
3	2	-11	3.76	3.40
3	2	-10	<2.00	.81
3	2	-9	11.62	11.72
3	2	-8	7.48	-7.29
3	2	-7	9.49	9.69
3	2	-6	3.71	4.07
3	2	-5	2.37	-1.25
3	2	-4	6.35	-6.95
3	2	-3	11.93	-11.40
3	2	-2	8.64	8.65
3	2	-1	24.93	-26.05
3	2	0	1.39	-1.72
3	2	1	18.04	-19.07
3	2	2	10.29	-10.21
3	2	3	9.57	-9.58
3	2	4	5.03	4.90
3	2	5	2.97	2.47
3	2	6	6.13	-6.05
3	2	7	8.46	7.85
3	2	8	8.39	9.05
3	2	9	3.92	4.02
3	2	10	2.57	-2.19
3	2	11	2.43	2.56
3	2	12	<1.45	.24
3	2	13	<1.03	-.43
4	2	-15	<1.35	.56
4	2	-14	<1.65	1.96
4	2	-13	<1.85	.19
4	2	-12	6.79	-6.33
4	2	-11	3.60	2.76
4	2	-10	26.03	26.51
4	2	-9	<1.54	-.54
4	2	-8	2.09	1.12
4	2	-7	<1.82	-1.16
4	2	-6	9.50	-9.45
4	2	-5	1.42	-1.11
4	2	-4	3.80	-3.46
4	2	-3	3.73	-2.74
4	2	-2	2.85	-2.60
4	2	-1	1.72	.91
4	2	0	36.63	41.32
4	2	1	<1.65	.19
4	2	2	1.64	1.90
4	2	3	<1.61	.39
4	2	4	<1.90	-1.41
4	2	5	1.65	1.70
4	2	6	10.75	-11.25
4	2	7	<2.04	.56
4	2	8	6.65	-7.01
4	2	9	2.80	2.78
4	2	10	<1.69	.61
4	2	11	<1.40	1.21
5	2	-15	2.50	-2.66
5	2	-14	1.54	-1.11
5	2	-13	4.29	4.15
5	2	-12	<1.91	-.16
5	2	-11	8.11	7.98
5	2	-10	<2.04	.65
5	2	-9	12.55	12.43
5	2	-8	5.06	-4.71
5	2	-7	10.40	9.65
5	2	-6	6.44	6.64
5	2	-5	<1.85	.45
5	2	-4	4.23	-4.34
5	2	-3	5.27	-5.58
5	2	-2	9.67	10.35
5	2	-1	10.92	-11.22
5	2	0	<1.04	-1.32
5	2	1	4.26	-4.01
5	2	2	6.30	-6.27
5	2	3	<1.98	.80
5	2	4	<1.57	1.69
5	2	5	<2.04	1.02
5	2	6	4.77	-5.07
5	2	7	<1.93	-.80
5	2	8	4.64	4.54
5	2	9	4.20	-3.77
5	2	10	3.57	-3.53
6	2	-14	<1.33	-.76
6	2	-13	<1.54	-.11
6	2	-12	9.89	-9.79
6	2	-11	<1.91	.29
6	2	-10	7.51	7.42
6	2	-9	1.61	-1.49
6	2	-8	7.41	-7.71
6	2	-7	<2.03	-1.97
6	2	-6	10.09	-10.97
6	2	-5	<1.99	-.41
6	2	-4	<1.98	-.93
6	2	-3	1.54	-1.23
6	2	-2	8.27	8.32
6	2	-1	<1.99	.45
6	2	0	23.90	25.62
6	2	1	<2.03	-1.04
6	2	2	6.08	5.97
6	2	3	<2.03	-1.69
6	2	4	2.45	-2.45
6	2	5	<1.93	-.45
6	2	6	5.95	-6.12
6	2	7	<1.64	-.29
6	2	8	1.55	-1.74
6	2	9	<1.05	1.26
7	2	-13	1.57	2.21
7	2	-12	<1.55	.56
7	2	-11	1.95	2.32
7	2	-10	<1.41	1.44
7	2	-9	2.10	2.33
7	2	-8	6.21	-6.47
7	2	-7	2.38	1.07
7	2	-6	5.47	5.46
7	2	-5	<2.03	-1.12
7	2	-4	<2.04	-1.15
7	2	-3	2.50	1.66
7	2	-2	7.20	7.65
7	2	-1	4.01	3.86
7	2	0	<2.01	-.39
7	2	1	6.09	6.32
7	2	2	4.75	-4.51
7	2	3	4.15	3.83
7	2	4	<1.75	.88
7	2	5	<1.60	-.84
7	2	6	<1.39	-1.75
8	2	-12	6.81	-7.22
8	2	-11	<1.39	-.74
8	2	-10	<1.55	.43
8	2	-9	<1.67	-.77
8	2	-8	9.07	-5.06
8	2	-7	<1.81	-.40
8	2	-6	6.84	-7.20
8	2	-5	1.68	1.44
8	2	-4	2.61	-2.10
8	2	-3	<1.89	.41
8	2	-2	4.02	3.82
8	2	-1	<1.83	.71
8	2	0	8.22	8.61
8	2	1	<1.70	-.97
8	2	2	<1.59	.88
8	2	3	1.44	-1.76
8	2	4	2.94	-3.35
9	2	-9	3.94	-4.04
9	2	-8	3.50	-3.80
9	2	-7	2.85	-3.15
9	2	-6	1.61	1.46
9	2	-5	<1.51	-.95
9	2	-4	<1.51	-.47
9	2	-3	[illegible]	[illegible]
9	2	-2	3.69	4.74
9	2	-1	4.39	5.09
9	2	0	<1.26	-.07
9	2	1	3.35	4.44

The reflexion 200 was observed to be extremely strong; its experimental value is not included in the table as relative scaling was uncertain owing to lack of medium strength reflexions.

45°, the corresponding parameters led to qualitative agreement between observed and calculated structure amplitudes for the reflexions of type $h0l$, h even. With a full-matrix least-squares program, written for the IBM 1620 II computer by the author, it was possible to refine these rough structure parameters of the projection. It is worth noting that, starting out from the same parameter values, an ordinary block diagonal program led to false minima.

The data from the three separate layers were normalized by combining the parameters found from refinements based on each layer separately, using the full-matrix program.

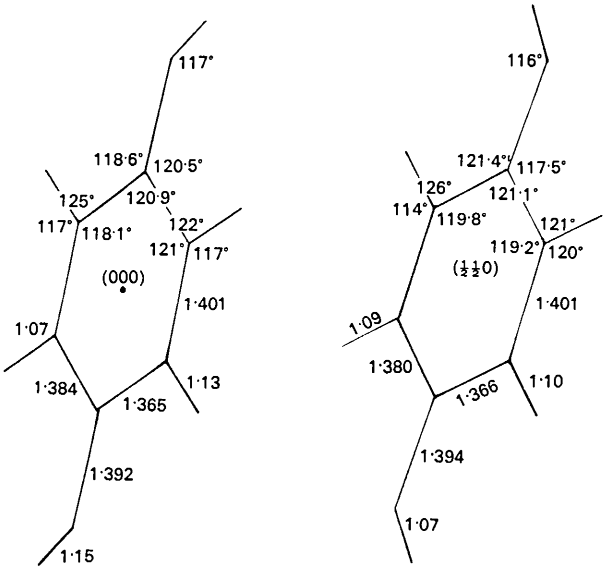

Fig. 2. Bond lengths and angles. For intermolecular distances see Tables 3 and 4.

The least-squares program used for the proper refinement of the structure parameters is 9 × 9, alternatively 4 × 4, block diagonal. In this program, written for the IBM 1620 II computer by the author, the weight assigned to the individual reflexions is not calculated as a function of F_{obs} but is fixed in the form of an array:

i	1	2	3	4	5	6	7	8	9
F_i	1·80	5·00	10·00	15·00	20·00	25·00	30·00	35·00	35·00
w_i	0·25	0·75	1·00	0·75	0·50	0·30	0·20	0·10	0·00

When $F_{i-1} < F_{calc} \le F_i$, then the weight becomes w_i. When $F_{calc} \le F_1$, then $F_{obs} - F_{calc}$ is put equal to 0. Thus the experimental structure amplitudes which are large and inaccurate do not influence the refinement, and experimental structure amplitudes derived from reflexions too weak to be measured are included with their maximum value and not half this value which is usual. This may be done because the difference between F_{obs} and F_{calc} is put equal to zero when F_{calc} is less than F_1 in the above array.

The agreement residual at the end of the last least-squares cycle was 8·5%. F_{obs} and F_{calc} are given in Table 1. The final coordinates and thermal parameters together with the standard deviations are given in Table 2. The positions of the atoms are shown in Fig. 1.

Analytical constants for atomic scattering factors used for calculation of structure amplitudes were those given by Moore (1963).

Discussion

The interatomic distances and angles (Table 3, Figs. 1 and 2) are calculated from the coordinates (Table 2) obtained from the least-squares refinement without corrections for rigid body vibrations. Such corrections cannot be done in the present case (Cruickshank, 1956)

Table 2. *Atomic coordinates and temperature parameters*

The temperature parameters refer to the expressions $\exp(-B\sin^2\theta/\lambda^2)$ and $\exp[-(h^2B_{11}+k^2B_{22}+l^2B_{33}+2hkB_{12}+2klB_{23}+2lhB_{31})]$

	x	y	z	B	B_{11}	B_{22}	B_{33}	$2B_{12}$	$2B_{23}$	$2B_{31}$
O(10)	0·02621	−0·00089	0·21399		0·02577	0·01890	0·00288	−0·00062	−0·00071	0·00875
C(11)	0·01187	−0·00242	0·10627		0·01197	0·00994	0·00315	0·00332	−0·00177	0·00555
C(12)	0·09050	0·18908	0·06660		0·01221	0·00683	0·00352	0·00613	0·00640	0·00329
C(13)	−0·07882	−0·19593	0·04131		0·01335	0·01922	0·00361	0·00041	−0·00156	0·00577
H(10)	−0·00778	−0·18929	0·24971	5·50						
H(12)	0·15983	0·36014	0·11316	1·43						
H(13)	−0·13667	−0·35295	0·07134	1·98						
O(20)	0·47791	0·51784	0·20531		0·02281	0·03190	0·00269	−0·00392	−0·00181	0·00867
C(21)	0·49136	0·50529	0·10250		0·01271	0·01014	0·00334	−0·00215	−0·00186	0·00566
C(22)	0·58358	0·31476	0·07242		0·01358	0·01074	0·00337	−0·00084	0·00362	0·00320
C(23)	0·40756	0·69258	0·03200		0·01278	0·01395	0·00352	0·00417	−0·00192	0·00539
H(20)	0·52478	0·35479	0·25520	3·82						
H(22)	0·64711	0·15265	0·12154	1·71						
H(23)	0·33825	0·84784	0·05738	2·15						

Estimated standard deviations:

	(X)	(Y)	(Z)
O	0·003	0·003	0·002 Å
C	0·003	0·005	0·003
H	0·04	0·05	0·04

Table 3. *Bond lengths and angles*

The positions of the atoms are shown in Fig. 1

O(10)–C(11)	1·392 Å	O(20)–C(21)	1·394 Å
C(11)–C(12)	1·365	C(21)–C(22)	1·366
C(11)–C(13)	1·384	C(21)–C(23)	1·380
C(12)–C(13′)	1·401	C(22)–C(23′)	1·401
H(10)–O(10)	1·15	H(20)–O(20)	1·07
H(12)–C(12)	1·13	H(22)–C(22)	1·10
H(13)–C(13)	1·07	H(23)–C(23)	1·09
C(11) –O(10)–H(10)	117°	C(21) –O(20)–H(20)	116°
O(10) –C(11)–C(13)	120·5	O(20) –C(21)–C(23)	117·5
C(13) –C(11)–C(12)	120·9	C(23) –C(21)–C(22)	121·1
C(12) –C(11)–O(10)	118·6	C(22) –C(21)–O(20)	121·4
C(11) –C(12)–C(13′)	118·1	C(21)– C(22)–C(23′)	119·8
C(13′)–C(12)–H(12)	117	C(23′)–C(22)–H(22)	114
H(12)–C(12)–C(11)	125	H(22)–C(22)–C(21)	126
C(11) –C(13)–H(13)	122	C(21) –C(23)–H(23)	121
H(13)–C(13)–C(12′)	117	H(23)–C(23)–C(22′)	120
C(12′)–C(13)–C(11)	121·0	C(22′)–C(23)–C(21)	119·2
O(10)–O(10″)	2·84 Å	O(20)–O(20″)	2·83 Å
H(10)–O(10″)	1·71	H(20)–O(20″)	1·83
O(10)–H(10)–O(10″)	166°	O(20)–H(20)–O(20″)	154°
H(10)–O(10)–O(10″)	8	H(20)–O(20)–O(20″)	16

without making certain assumptions which would here be dubious, considering the uncertainty in the thermal parameters. This uncertainty is primarily due to the scaling of the three layers on which the structure determination is based. The differences in the C–C bond lengths are probably not real, at least not to the extent shown, as rigid body vibrations certainly influence the results. However, similar differences have recently been observed by Sakurai (1965). Theoretical calculations by Nishimito & Forster (1966) are of relevant interest.

In many phenols it is observed that the angle C–C–OH is somewhat larger on the hydrogen side (Hirshfeld, 1964); it appears to be so in the present case too. In phloroglucinol (Maartmann-Moe, 1965) it is observed that the oxygen atoms are out of the plane of the benzene ring to the side of the hydroxyl hydrogen atoms. In hydroquinone a similar situation is not significantly indicated. The mean planes through the molecules (disregarding the hydrogen atoms) are

$$(0,0,0) \quad -6{\cdot}58447x+2{\cdot}84976y+0{\cdot}82447z=0$$

$$(\tfrac{1}{2},\tfrac{1}{2},0) \quad 6{\cdot}26627x+2{\cdot}96244y+0{\cdot}40093z=4{\cdot}614$$

(x,y,z in fractional coordinates). The oxygen and carbon atom coordinates are all less than 0·006 Å out of the plane. The hydrogen atoms bonded to the benzene ring are less than 0·07 Å out of the plane.

The instability and pronounced cleavage of the γ modification is not surprising as there is no hydrogen bonding and seemingly no other forces but weak van der Waals forces keeping the sheets of hydrogen bonded molecules together. The shortest intermolecular distances are given in Table 4. A tendency to form twins should be expected in a case like this, but no indications of this have been observed during the investigation.

Table 4. *Shortest intermolecular distances*

H(12) –H(23)		3·11 Å
H(12) –C(23)		3·06
H(23) –C(12)	(x, $1+y$, z)	2·70
H(23) –C(13′)	(x, $1+y$, z)	2·79
H(13′)–C(23)		2·83
H(13′)–C(22′)		2·85
C(12)—C(23)		3·78
C(12)—C(23)	(x, $y-1$, z)	3·76
C(13′)–C(23)		3·63
C(13′)–C(23)	(x, $y-1$, z)	3·65
C(13′)–C(22′)		3·84
C(13′)–C(22′)	(x, $y-1$, z)	3·91
C(12)—C(13)	(x, $1+y$, z)	3·53
C(22)—C(23)	(x, $y-1$, z)	3·51

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