indicated in Fig. 2. As expected, the hydrogen atoms do not lie directly on the N · · · S line. The bond lengths and angles involved in the hydrogen bonding are listed in Table 10.

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The Crystal Structure of Tetrahydroxy-*p*-benzoquinone

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The crystal structure of tetrahydroxy-*p*-benzoquinone dihydrate has been determined from three-dimensional Weissenberg data and refined by anisotropic differential synthesis and least-squares calculations. The crystals are monoclinic, space group $P2_1/c$, with $a=5{\cdot}226\pm0{\cdot}002$, $b=5{\cdot}118\pm0{\cdot}002$, $c=15{\cdot}502\pm0{\cdot}008$ Å and $\beta=103{\cdot}89\pm0{\cdot}02°$. There are two centrosymmetric quinone molecules with attendant water molecules per unit cell.

The molecules, which possess the quinoid structure with the $C=O$ distance 1·229 Å and the $C=C$ distance 1·342 Å, are remarkably planar, and, through pairs of hydrogen bonds, form chains. The water molecules also are hydrogen bonded to form chains along one set of screw axes. Further hydrogen bonding ties the quinone and water chains together. The anisotropic thermal parameters of the atoms have been interpreted successfully in terms of rigid-body vibrations of the quinone molecule. The quinoid structure and self-complexing charge transfer interactions appear to present a reasonable explanation of the glistening black color of the crystals.

Introduction

Tetrahydroxy-*p*-benzoquinone (THQ), $C_6(OH)_4O_2$, is one of a series of labile oxidation products of inositol which have interested organic and structural chemists for many years. Until recently the standard method for their preparation was to treat inositol with fuming nitric acid, a reaction difficult to control (Gelormini & Artz, 1930; Hoglan & Bartow, 1940; Preisler & Berger, 1942). The reaction leads to a mixture of products difficult to separate, and the individual products in some instances are so reactive toward oxygen that the dissolved oxygen in solvents makes recrystallization difficult or impossible. THQ has also been prepared in 10% yield by the self-condensation of glyoxal in the presence of sodium sulfite, air, and a base (Homolka, 1921; Fatiadi, Isbell & Sager, 1963). The proposed molecular structures and unusual properties of these compounds make structural studies of them highly important. This communication presents the results of a detailed investigation of the molecular and crystal structure of THQ.

The glistening black crystals of THQ are actually the dihydrate, a fact apparently not previously recognized. Elementary microanalysis first disclosed the probable presence of water of hydration. The infrared spectrum of THQ in a Nujol mull had been reported by Fatiadi, Isbell & Sager (1963). Spectra prepared in fluorocarbon mulls* clearly presented the following: (1) OH bands at 3540 and 3370 cm^{-1}, presumably from the H_2O molecules; (2) from 3100–2500 cm^{-1}, absorption from OH strongly hydrogen bonded; (3) a strong band at 1625 cm^{-1} with side peak at 1650 cm^{-1} inter-

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* The author is indebted to Dr Foil A. Miller for the preparation and interpretation of these spectra.

preted as C=O stretching and H_2O scissors motion, respectively; the C=C absorption, if present, is covered up by this band; (4) OH bending at 1320, 1235, and 575 cm^{-1}. There is no evidence for the hydration of the >C=O groups to secondary alcohol groups, $>C(OH)_2$, as appears to be the case in triquinoyl (Person & Williams, 1957). The X-ray analysis completely confirms the strong hydrogen bonding and the presence of H_2O molecules.

Experimental

THQ dihydrate, $C_6(OH)_4O_2.2H_2O$, has a molecular weight of 208·12. Single-crystal photographs calibrated with reflections from a standard quartz crystal (Smith & Alexander, 1963), $a=4·2555\pm0·0001_3$ Å, $c/a=1·10000\pm0·00005$, lead to a monoclinic cell with the following constants and e.s.d.'s (Cu $K\alpha=1·5418$ Å):

$$a=5·226\pm0·002,\ b=5·118\pm0·002,\ c=15·502\pm0·008\ \text{Å}$$

$$\beta=103·89\pm0·02°$$

$$Z=2,\ D_x=1·717\ \text{g.cm}^{-3},\ \text{space group},\ P2_1/c\ .$$

The X-ray density reported above is entirely reasonable, since the density of ordinary benzoquinone is 1·320 $g.cm^{-3}$, and that of chloranil, tetrachloro-*p*-benzoquinone, is 1·953 $g.cm^{-3}$ (Trotter, 1960; Chu, Jeffrey & Sakurai, 1962). The quinone molecules possess a center of symmetry.

The structure has been determined from visually estimated, Cu $K\alpha$ Weissenberg intensity data. The 703 observed independent reflection intensities were corrected as usual, and put on an approximate absolute intensity basis with a Wilson plot. No corrections were made for absorption or extinction. Crystals used for the intensity measurements were approximately equidimensional with their largest dimension ≤ 0·4 mm. For THQ $\mu=14·9$ cm^{-1} for Cu $K\alpha$ radiation. It was estimated, therefore, that absorption corrections would usually be much less than 5%. An additional 138 reflections, too weak to measure, comprise the remainder of the 841 reflections estimated to be available in the Cu $K\alpha$ sphere. The corrected reflection intensities were reduced to F's, and, finally, changed to E's, normalized structure factors.

THQ seemed an ideal case for phase determination by the symbolic addition procedure (Karle & Karle, 1963; Karle, Britts & Gum, 1964) based chiefly on the Σ_2 relation of Sayre (1952) and Hauptman & Karle (1953). The three strong reflections, 112, $\bar{2}14$, and $\bar{4}29$, were picked with positive signs to fix the origin. Just for the experience the author calculated 41 signs manually, obtaining two from the Σ_1 relation and the rest from Σ_2. All 41 of these signs later proved to be correct. The entire set of E-value data was then fed into an IBM 1620 computer program (Beurskens, 1963; Kim, 1964) which yielded several hundred more signs. Usually these signs would have been used directly to do a three-dimensional Fourier synthesis, but it was elected, because of the short a and b axes, to try first the success of projections along these two axes.

From a total of 84 $0kl$ reflections there were 33 with E's > 0·8. With the signs determined as described, these 33 reflections gave the excellent E-projection along [100] shown in Fig. 1. From a total of 91 $h0l$ reflections, 36 with E's > 0·8 gave a similarly satisfactory E-projection along [010]. Structure factors calculated (Shiono, 1962) with the C and O parameters from these projections immediately gave an R value of 0·213. The atomic scattering factors used were those of Berghuis, Haanappel, Potters, Loopstra, MacGillavry & Veenendaal (1955) for carbon and oxygen. Refinement of the parameters proceeded smoothly as summarized in Table 1 in which R values are based on observed reflections only. The differential syntheses were computed with Shiono's (1963) program for the IBM 7070 computer. In the least-squares calculations Cruickshank's (1961) weighting scheme was used with Shiono's (1963) IBM 7090 modification of the Busing, Martin & Levy (1962) full-matrix program. Two- and three-dimensional Fourier, and difference Fourier, syntheses were computed with an IBM 7070 program prepared by Chu & McMullan (1962).

The hydrogen atoms were located by a three-dimensional difference Fourier synthesis. Four H atoms were to be located. As is usually the situation, more peaks appeared in the difference Fourier than the number of H atoms sought. Of the eight strongest peaks observed, four were located in positions that were chemically and geometrically logical for bonding to oxygen atoms and for the formation of hydrogen bonds. When the meas-

Table 1. *Summary of the refinement of* THQ

Stage in structure determination	R Value
1. Original C and O parameters from the E-value projections. (Set No. 1)	0·213
2. Parameter set No. 2. Manual changes attempted	0·230
3. First differential synthesis. (Isotropic – Set No. 3)	0·145
4. Second differential synthesis. (Isotropic – Set No. 4)	0·135
5. Third differential synthesis. (Anisotropic – Set No. 6)	0·116
6. Fourth differential synthesis. (Anisotropic – Set No. 7)	0·102
7. Two cycles full-matrix least-squares refinement. (C and 0 only. Anisotropic – Set No. 9)	0·086
8. Parameter set No. 9 for C and O (anisotropic) plus original H parameters	0·079

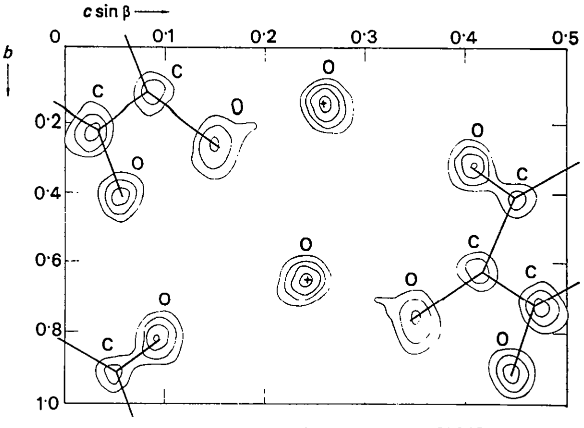

Fig. 1. E-projection of THQ along [100].

Table 2. *Observed and calculated structure factors based on final least-squares parameters*

h	k	ℓ	10 F_o	10 F_c
0	0	2	382	375
0	0	4	20	13
0	0	6	485	-435
0	0	8	163	-135
0	0	10	290	-263
0	0	12	100	107
0	0	14	38	-34
0	0	16	34	-36
0	0	18	21	-24
0	1	1	169	155
0	1	2	221	-216
0	1	3	46	54
0	1	4	20	32
0	1	5	164	-149
0	1	6	64	-59
0	1	7	40	-32
0	1	8	379	-335
0	1	9	25	29
0	1	10	44	40
0	1	11	108	93
0	1	12	52	49
0	1	15	22	-19
0	1	16	59	-60
0	1	17	43	-45
0	1	18	14	16
0	2	0	435	-381
0	2	1	44	-40
0	2	2	106	100
0	2	3	418	391
0	2	4	92	88
0	2	5	8	1
0	2	6	73	-67
0	2	7	144	134
0	2	8	129	-124
0	2	9	62	-61
0	2	10	16	18
0	2	12	67	-72
0	2	13	103	-103
0	2	14	29	29
0	2	15	29	-35
0	2	16	78	77
0	2	17	56	-58
0	2	18	37	40
0	3	1	168	-157
0	3	2	27	33
0	3	3	55	-52
0	3	4	128	-136
0	3	5	108	113
0	3	7	62	67
0	3	8	159	164
0	3	10	64	71
0	3	11	96	-95
0	3	13	24	24
0	3	14	17	23
0	3	15	20	23
0	3	16	17	-21
0	3	17	40	43
0	4	0	100	-90
0	4	1	23	23
0	4	2	35	-36
0	4	4	32	-33
0	4	5	181	166
0	4	6	208	190
0	4	7	98	99
0	4	8	50	48
0	4	9	67	67
0	4	11	30	-29
0	4	12	40	-34
0	4	13	36	-34
0	4	14	19	-18
0	4	15	60	-57
0	5	1	29	32
0	5	2	43	-49
0	5	3	29	35
0	5	4	19	13
0	5	8	29	-29
0	5	9	15	7
0	5	10	28	-23
0	6	0	43	-41
0	6	1	27	27
0	6	2	56	-50
0	6	5	19	-16
0	6	6	36	-32
0	6	7	52	-44
1	0	0	180	-195
1	0	2	303	-322
-1	0	2	66	-70
1	0	4	195	197
-1	0	4	246	239
1	0	6	28	-42
-1	0	6	679	-643
1	0	8	193	184
1	0	10	173	155
-1	0	10	16	-17
1	0	12	40	-36
-1	0	12	83	77
1	0	14	55	-58
-1	0	14	47	-49
1	0	16	34	32
-1	0	16	88	87
1	0	18	49	51
-1	0	18	34	-38
1	1	0	315	256
1	1	1	518	-442
-1	1	1	114	112
1	1	2	767	928
-1	1	2	86	-76
1	1	3	420	-300
-1	1	3	140	143
1	1	4	104	-77
-1	1	4	105	100
1	1	5	349	-287
-1	1	5	394	361
1	1	6	109	-104
1	1	7	105	86
-1	1	7	163	150
1	1	8	149	-135
-1	1	8	198	186
1	1	9	65	60
-1	1	9	60	53
1	1	10	13	15
-1	1	10	79	-76
1	1	11	116	106
-1	1	11	95	-72
1	1	12	83	-80
-1	1	12	60	61
1	1	13	16	15
-1	1	13	62	-59
1	1	14	83	80
-1	1	14	163	160
1	1	15	44	44
-1	1	15	69	-70
-1	1	16	67	66
-1	1	17	34	-32
-1	1	18	37	-38
1	2	0	122	113
1	2	1	64	62
-1	2	1	66	-64
1	2	2	110	112
-1	2	2	109	-114
1	2	3	228	212
-1	2	3	320	327
1	2	4	52	-62
-1	2	4	76	-75
1	2	5	42	37
-1	2	5	39	39
1	2	6	89	93
-1	2	6	313	315
-1	2	7	13	-16
1	2	8	69	-67
-1	2	8	61	60
1	2	9	67	-68
-1	2	9	240	-255
1	2	10	142	-141
-1	2	10	16	-8
-1	2	11	16	19
1	2	12	18	-13
1	2	13	20	20
-1	2	13	30	32
1	2	14	17	13
-1	2	14	22	22
-1	2	15	113	124
1	2	16	18	-22
-1	2	16	44	-48
-1	2	17	17	18
1	3	0	99	100
1	3	1	136	123
-1	3	1	103	-111
1	3	2	23	-26
-1	3	2	198	-201
1	3	3	58	52
-1	3	3	59	55
-1	3	4	28	36
1	3	5	119	110
-1	3	5	29	-35
1	3	6	62	-61
1	3	7	165	-167
-1	3	7	76	85
1	3	8	215	-222
-1	3	8	19	-22
1	3	9	47	-50
-1	3	9	20	20
1	3	10	98	-101
-1	3	10	67	77
1	3	11	52	-49
-1	3	11	31	30
-1	3	12	69	66
1	3	13	44	44
-1	3	13	33	-31
1	3	14	38	36
-1	3	14	31	-29
1	3	16	59	58
-1	3	16	18	-18
1	4	0	84	-82
1	4	1	94	-83
-1	4	1	117	112
1	4	2	45	47
-1	4	2	45	40
1	4	3	70	-60
-1	4	3	49	-48
1	4	4	28	23
-1	4	4	47	-47
-1	4	5	56	-59
1	4	6	36	-36
-1	4	6	66	-71
1	4	7	14	14
-1	4	7	45	-41
1	4	8	19	-15
-1	4	8	52	-55
1	4	9	13	15
-1	4	9	49	49
1	4	10	57	60
-1	4	10	43	46
1	4	11	24	-25
-1	4	11	35	-34
1	4	12	23	28
-1	4	12	41	-41
1	4	13	20	-23
1	4	14	20	21
1	5	0	53	54
1	5	1	74	71
-1	5	1	38	39
-1	5	2	17	-23
1	5	3	41	40
-1	5	3	74	-74
-1	5	4	102	-109
1	5	5	23	18
-1	5	5	119	-120
1	5	6	23	-22
-1	5	6	18	10
1	5	7	50	44
-1	5	7	99	-107
1	5	8	62	59
-1	5	8	63	70
1	5	9	14	15
-1	5	9	42	-48
1	5	10	28	-25
-1	5	10	64	64
1	5	11	13	-12
-1	5	11	26	24
1	6	0	64	52
1	6	1	28	24
-1	6	1	54	-47
1	6	2	25	-22
-1	6	2	35	21
1	6	3	20	-17
-1	6	3	40	-38
1	6	4	38	-33
-1	6	4	26	23
-1	6	5	18	14
2	0	0	102	-111
2	0	2	117	-125
-2	0	2	230	-241
2	0	4	257	-254
-2	0	4	188	-189
2	0	6	211	201
-2	0	6	28	11
2	0	8	90	97
2	0	10	19	20
-2	0	10	74	84
2	0	12	49	-51
-2	0	12	356	359
-2	0	14	141	147
2	0	16	61	-61
-2	0	16	22	-19
-2	0	18	90	-89
2	1	0	37	35
2	1	1	38	-31
-2	1	1	255	248
2	1	2	108	-79
-2	1	2	314	-316
2	1	3	37	32
2	1	4	247	-232
-2	1	4	470	495
2	1	5	112	-101
-2	1	5	19	31
2	1	6	62	58
-2	1	6	156	151
2	1	7	20	-15
-2	1	7	215	-220
2	1	8	57	-61
-2	1	8	107	116
2	1	9	48	-46
2	1	10	132	120
-2	1	10	79	-78
2	1	11	30	32
-2	1	11	17	-12
2	1	12	74	74
-2	1	12	17	-13
-2	1	13	97	92
-2	1	14	162	-157
-2	1	15	17	18
2	1	16	20	-23
-2	1	16	16	21
-2	1	17	15	20
-2	1	19	33	-31
2	2	0	121	-132
2	2	1	99	-91
-2	2	1	13	4
2	2	2	69	78
-2	2	2	90	-81
2	2	3	327	-301
-2	2	3	125	-142
2	2	4	220	225
-2	2	4	125	-125
2	2	5	189	-184
-2	2	5	67	-70
2	2	6	18	14
-2	2	6	156	-164
2	2	7	58	-60
2	2	8	53	51
-2	2	8	56	67
2	2	9	174	178
-2	2	9	50	-54
2	2	10	81	83
-2	2	10	93	97
2	2	11	105	114
-2	2	11	49	-49
2	2	12	34	36
-2	2	12	67	-73
2	2	13	65	70
-2	2	13	17	-26
2	2	14	31	-31
-2	2	14	32	-32
2	2	15	26	-23
-2	2	15	61	61
-2	2	16	15	10
-2	2	17	25	25
2	3	1	129	144
-2	3	1	130	-131
2	3	2	130	133
-2	3	2	13	-18
2	3	3	26	19
-2	3	3	35	-35
-2	3	4	80	-89
2	3	5	82	81
-2	3	5	74	-72
2	3	6	31	-30
-2	3	6	31	-28
2	3	7	20	-12
-2	3	7	123	137
2	3	8	18	17
-2	3	8	80	89
2	3	9	32	31
-2	3	9	36	-46
-2	3	10	34	-35
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-2	3	11	13	-4
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-2	3	12	34	-31
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2	4	7	25	24
-2	4	7	127	-135
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2	4	11	12	13
-2	4	11	49	43
-2	4	12	31	-30
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2	5	1	87	-90
-2	5	1	28	-27
2	5	2	64	-66
-2	5	2	73	70
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-2	5	4	22	19
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-2	5	7	21	22
-2	5	8	59	-65
-2	5	9	28	27
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-2	6	2	87	71
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-2	6	3	32	30
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-2	6	5	28	24
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3	0	0	276	-309
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-3	0	2	303	338
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-3	0	4	231	248
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-3	0	6	217	219
-3	0	8	45	-53
3	0	10	44	48
-3	0	10	79	-85
3	0	12	86	-84
-3	0	12	246	-245
3	0	14	33	-41
-3	0	14	19	-21
-3	0	16	47	42
3	1	0	120	-105
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-3	1	1	91	83
3	1	2	81	74
-3	1	2	13	11
3	1	3	109	96
-3	1	3	36	39
-3	1	4	36	-24
3	1	5	79	69
-3	1	5	67	64
3	1	6	18	-7
-3	1	6	15	-7
3	1	7	39	35
-3	1	7	136	-141
3	1	8	51	50
-3	1	8	118	110
3	1	9	40	-38
-3	1	9	131	-130
3	1	0	28	-25
-3	1	10	213	-223
3	1	11	79	-82
-3	1	11	144	-135
3	1	12	74	-76
-3	1	12	108	-103
3	1	13	50	-53
-3	1	13	31	31
3	1	14	44	-44
-3	1	14	30	-34
-3	1	15	41	43
-3	1	16	38	40
-3	1	17	85	81
-3	1	19	16	17
3	2	0	93	87
3	2	1	96	85
3	2	2	49	-48
-3	2	2	122	-127
3	2	3	105	-101
-3	2	3	112	-109
3	2	4	107	-104
-3	2	4	57	-61
3	2	5	39	39
-3	2	5	84	79
3	2	6	64	-61
-3	2	6	20	27
3	2	7	29	-32
-3	2	7	38	-45
3	2	8	20	20
-3	2	8	14	21
3	2	9	10	10
-3	2	9	48	47
3	2	10	14	-16
-3	2	10	13	-5
3	2	11	49	-52
-3	2	11	32	-40
3	2	12	31	30
-3	2	12	88	84
3	2	14	21	23
-3	2	15	15	-13
-3	2	16	28	-25
-3	2	17	23	18
-3	2	18	43	38
3	3	0	55	-53
3	3	1	47	49
3	3	2	14	16
-3	3	2	136	139
3	3	3	11	5
-3	3	3	53	-55
3	3	4	123	126
-3	3	4	230	245
3	3	5	30	-27
-3	3	5	155	-164
3	3	6	135	143
-3	3	6	65	64
3	3	7	44	-49
3	3	8	31	35
-3	3	8	52	-52
-3	3	9	53	59
3	3	10	13	13
-3	3	10	25	-27
3	3	11	49	46
-3	3	11	97	108
3	3	12	13	8
-3	3	12	13	-12
-3	3	13	25	-20
-3	3	14	47	-51
3	4	0	66	67
-3	4	1	13	13
3	4	2	50	58
-3	4	2	13	16
-3	4	3	52	57
3	4	3	94	100
3	4	4	72	83
3	4	6	49	-52
-3	4	6	95	-99
-3	4	7	45	42
3	4	8	30	-29
-3	4	8	25	26
3	4	9	10	-12
3	4	10	5	-6
-3	4	10	25	32
3	5	0	14	-17
3	5	1	13	-11
-3	5	1	36	-32
-3	5	2	42	39
3	5	3	10	-12
-3	5	4	43	-38
-3	5	5	57	52
3	5	6	10	-10
3	5	7	7	19
-3	5	7	47	47
-3	5	8	17	-16
-3	5	9	15	16
4	0	0	58	-58
4	0	2	40	40
-4	0	2	28	-28
4	0	4	111	-106
-4	0	4	22	-25
4	0	6	23	-22
-4	0	6	227	229
4	0	8	42	-47
-4	0	8	72	-82
-4	0	10	41	-37
4	0	12	56	49
-4	0	12	92	-89
-4	0	14	22	-25
-4	0	16	76	-69
-4	0	18	71	63
4	1	0	103	96
-4	1	1	85	-75
4	1	2	101	-96
-4	1	2	108	-112
4	1	3	17	10
-4	1	3	56	-51
4	1	4	18	16
-4	1	4	61	-51
4	1	5	36	44
-4	1	5	48	-46
4	1	6	42	44
-4	1	6	17	7
4	1	7	17	-18
-4	1	7	46	38
4	1	8	52	50
-4	1	8	47	50
-4	1	9	60	52
-4	1	10	145	145
-4	1	11	31	30
4	1	12	22	22
-4	1	12	30	31
-4	1	14	62	-62
-4	1	15	15	-14
-4	1	16	36	33
-4	1	17	23	-20
-4	1	18	52	46

Table 2 (*cont.*)

h	k	l	10 F_o	10 F_c
4	2	0	52	55
4	2	1	93	101
-4	2	1	102	-102
4	2	2	38	-35
-4	2	2	146	147
4	2	3	17	-16
-4	2	3	176	-198
4	2	4	17	-16
-4	2	4	103	107
-4	2	5	49	51
4	2	6	61	-66
-4	2	6	37	-38
4	2	7	45	-44
-4	2	7	105	107
4	2	8	47	-40
-4	2	8	55	60
-4	2	9	177	204
-4	2	10	29	27
4	2	11	20	-18
-4	2	11	17	18
-4	2	12	20	-23
-4	2	14	34	-33
-4	2	15	78	-81
-4	2	16	12	-9
4	3	0	25	-22
4	3	1	12	-12
-4	3	1	57	56
4	3	2	24	-19
-4	3	2	12	-10
4	3	3	24	-21
-4	3	3	34	32
4	3	4	70	-71
-4	3	4	72	-70
4	3	5	68	-68
-4	3	5	48	46
4	3	6	67	-75
4	3	7	22	22
-4	3	7	19	-18
-4	3	8	19	15
-4	3	10	52	-51
-4	3	11	22	23
-4	3	12	21	-17
-4	3	14	19	17
-4	3	15	19	-15
5	0	0	43	-50
5	0	2	23	-25
5	0	4	21	22
-5	0	6	19	25
5	0	8	15	-21
-5	0	8	61	57
-5	0	10	38	35
-5	0	12	70	-72
-5	0	14	59	62
-5	0	16	70	66
5	1	0	118	-105
5	1	1	58	-53
-5	1	1	111	-110
5	1	2	84	-75
-5	1	2	102	94
5	1	3	33	-38
-5	1	3	17	-19
5	1	4	16	12
-5	1	4	108	-106
5	1	5	28	27
5	1	6	19	-19
-5	1	6	17	-23
5	1	7	26	28
-5	1	7	98	86
5	1	8	24	26
-5	1	8	33	-32
-5	1	9	35	32
-5	1	10	16	17
-5	1	11	41	38
-5	1	12	15	20
-5	1	14	65	48
-5	1	15	10	13
-5	1	16	59	-54
5	2	0	16	22
5	2	1	49	-50
-5	2	1	83	85
5	2	2	27	26
-5	2	2	29	-25
-5	2	3	47	42
-5	2	4	12	-11
5	2	5	36	38
-5	2	5	16	-16
5	2	6	45	43
-5	2	6	21	-26
5	2	7	42	40
-5	2	7	61	-63
-5	2	8	81	-89
-5	2	10	43	-49
-5	2	11	11	-9
-5	2	12	45	39
-5	2	13	12	13
-5	2	15	25	-26
5	3	0	28	25
5	3	1	16	-11
-5	3	1	68	68
5	3	2	45	-42
-5	3	2	37	-42
5	3	4	22	-23
-5	3	4	46	-46
5	3	5	14	-10
-5	3	5	10	-5
-5	3	6	74	-80
-5	3	7	56	-59
-5	3	8	67	-72
-5	3	10	18	21
-5	3	11	18	-15
-5	3	12	40	38
6	0	0	98	99
6	0	2	116	105
-6	0	2	44	-44
6	0	4	72	69
-6	0	4	55	-54
-6	0	6	64	-62
-6	0	8	19	-22
-6	0	12	27	27
6	1	0	47	42
6	1	1	46	43
-6	1	1	18	16
6	1	3	18	17
-6	1	5	28	-24
-6	1	6	28	-25
-6	1	7	22	18
-6	1	8	44	-42
-6	1	10	50	42
-6	1	12	29	-25
6	2	0	38	-36
6	2	1	33	-29
-6	2	1	15	17
6	2	2	16	-20
-6	2	3	28	33
-6	2	4	28	-24
-6	2	6	26	-24
-6	2	9	37	-32

Table 3. *Fractional atomic coordinates, anisotropic thermal parameters,* and their estimated standard deviations, σ*

The σ's, in parentheses, have been multiplied by 10^4

Atom	*x*	*y*	*z*
C(1)	0·6587 (6)	0·2205 (7)	0·0310 (2)
C(2)	0·4690 (6)	0·1243 (7)	0·0809 (2)
C(3)	0·3204 (6)	−0·0858 (7)	0·0512 (2)
O(1)	0·7978 (5)	0·4118 (5)	0·0572 (2)
O(2)	0·4669 (5)	0·2661 (5)	0·1536 (2)
O(3)	0·1428 (5)	−0·1762 (5)	0·0939 (2)
O(4)	0·8523 (5)	0·6547 (6)	0·2418 (2)
H(2)	0·3100	0·2250	0·1700
H(3)	0·0700	0·6800	0·0750
H(4)	−0·0700	−0·1900	0·2400
H(4′)	0·7500	0·5400	0·1850

Atom	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
C(1)	0·0233 (12)	0·0231 (12)	0·0030 (1)	−0·0043 (10)	0·0024 (3)	−0·0003 (3)
C(2)	0·0252 (12)	0·0254 (13)	0·0027 (1)	−0·0034 (11)	0·0030 (3)	−0·0004 (3)
C(3)	0·0250 (12)	0·0278 (14)	0·0028 (1)	−0·0026 (11)	0·0028 (3)	−0·0001 (3)
O(1)	0·0318 (10)	0·0282 (11)	0·0036 (1)	−0·0109 (8)	0·0038 (3)	−0·0016 (3)
O(2)	0·0408 (12)	0·0367 (12)	0·0037 (1)	−0·0135 (11)	0·0059 (3)	−0·0041 (3)
O(3)	0·0333 (11)	0·0319 (12)	0·0038 (1)	−0·0113 (9)	0·0057 (3)	−0·0017 (3)
O(4)	0·0387 (12)	0·0403 (13)	0·0033 (1)	−0·0053 (10)	0·0045 (3)	−0·0005 (3)

* In the expression: $\exp\{-(h^2\beta_{11}+\ldots+2kl\beta_{23})\}$.

ured parameters from plots of these peaks were included as H atoms in the structure they lowered the *R* value to 0·079. Hydrogen parameters were not further refined, and the temperature factor used for them was that of the oxygen atom to which the H atom is attached. Throughout this paper O atoms bear the number of the C atom to which they are attached, and H atoms the number of the O atom to which attached. The remaining four peaks of the eight strongest were found to be in completely illogical positions chemically, and usually at distances too large to be considered as bonded to any atom. The still weaker peaks were not given any consideration.

The observed and calculated structure factors are listed in Table 2. The fractional atomic coordinates, the atomic anisotropic thermal parameters from the anisotropic least-squares refinement, and the estimated standard deviations of each are presented in Table 3.

The crystal structure

The crystal structure with its interesting system of hydrogen bonds is best depicted by projections along the *x* and *y* axes (Figs. 2 and 3). The water molecules are hydrogen bonded to form chains along one of the two sets of screw axes at $z=0·25$ and 0·75. The quinone molecules, through pairs of hydrogen bonds, form chains extending diagonally through the cell in the *ab* planes at $c=0$ and 0·5 (Figs. 2 and 3). These bonds are between a hydroxyl group of one molecule and a quinoid oxygen of its neighbor, and the bond distance, 2·744 Å, is almost the same as that in ice, 2·76 Å (Fuller, 1959). The O–H · · · O angle of these bonds is 156°, as compared with 180° in ice. Each quinone molecule of the chain also is linked on each side by hydrogen bonds to two of the parallel water chains (Fig. 3). One of these bonds, 2·654 Å and O–H · · · O angle of 148°,

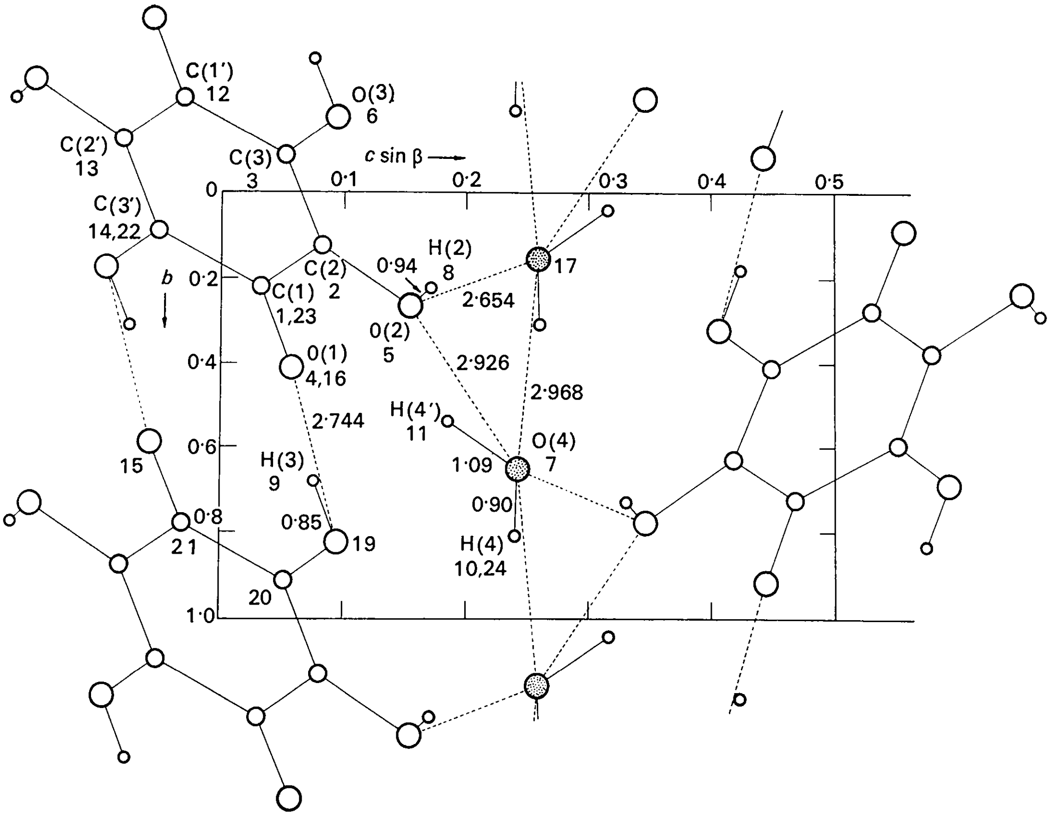

Fig. 2. Projection of the THQ structure along [100]. Oxygen atoms of the water molecules are shown stippled. Hydrogen bonds are indicated by dashed lines.

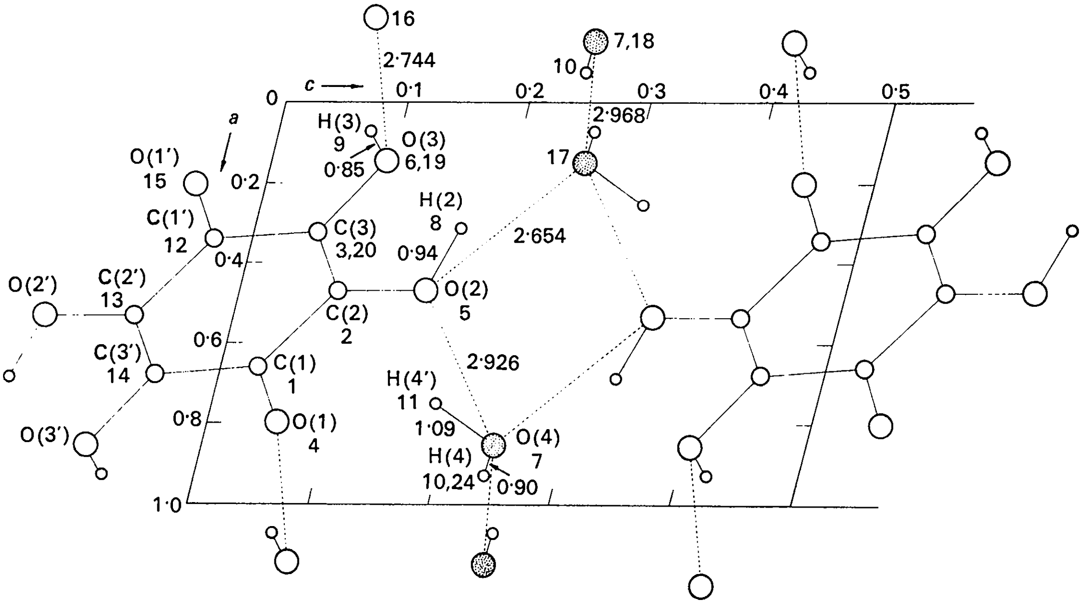

Fig. 3. Projection of the THQ structure along [010]. Oxygen atoms of the water molecules are shown stippled. Hydrogen bonds are indicated by dashed lines.

is very strong; the other one, 2·926 Å and O–H · · · O angle of 140°, is rather weak. The hydrogen bond distances on the projections all have σ's of 0·004 Å. The H_2O molecules in the water chains are bonded together with still weaker hydrogen bonds, distance 2·968 Å and O–H · · · O angle 170°. The H–O–H angle of the water molecules is about 126°. All angles involving the H atom positions may have an uncertainty of ± 5°. The bonded O–H distances vary from 0·85 to 1·09 Å, the average being 0·95 Å. This is exactly the average found by Rabinowitz & Kraut (1964) for 12 O–H distances in myo-inositol.

Geometry of the quinone molecule

Fig. 4 presents a diagram of the centrosymmetrical quinone molecule with bond distances and angles. The estimated standard deviations, σ for C to C and C to O bonds vary from 0·004 to 0·005 Å, and for the angles involving only C and O atoms $\sigma=0{\cdot}3°$. The standard deviations of bond lengths and bond angles were calculated by the method of Cruickshank & Robertson (1953), using an IBM 1620 program (Chu, 1963). These values do not include the standard deviations of the lattice constants, which on the average are two or three times better than those of the atomic parameters. If only bond differences greater than 3σ are considered significant, all chemically equivalent bonds in the molecule must be considered to have equal bond distances. The deviations of certain of the angles from 120°, however, must be considered significant.

The quinoid structure is prominent in the quinone molecule, the C=O distance being 1·229 ± 0·005 Å, and the C=C distance 1·342 ± 0·005 Å. It is interesting to compare these and other distances and angles in the molecule with the corresponding ones in chloranil (Chu, Jeffrey & Sakurai, 1962) and *p*-benzoquinone (Trotter, 1960) summarized in Table 4. The difference in the C=O distances in THQ and chloranil is undoubtedly real. The observed C=O distance is almost exactly the average observed in aldehydes, ketones, amides ($O=\underset{|}{C}-N\langle$), carboxylic acids and esters (shorter bond): 1·23 ± 0·01 Å. The C=C is likewise close to the average value 1·337 ± 0·006 Å reported for simple olefinic structures, and to 1·34 ± 0·01 Å for C=C–C=C found in cyclooctatetraene and several conjugated systems (Sutton, 1958).

Table 4. *Comparison of* THQ *mean bond distances and angles with those in chloranil and p-benzoquinone*

Bond or angle	THQ	Chloranil	*p*-Benzoquinone
C(1)–O(1)	1·229 Å	1·195 Å	1·222 Å
C(1)–C(2), C(1)–C(3)	1·478	1·477	1·477
C(2)–C(3)	1·342	1·342	1·322
C(3)–O(3), C(2)–O(2)	1·344	—	—
C(2)–C(1)–C(3)	119·57°	117·27°	117·80°
O(1)–C(1)–C(3)	119·80	121·37	121·10
C(1)–C(3)–O(3)	117·85	116·25	—
C(1)–C(3)–C(2)	120·93	121·37	121·10
C(2)–C(3)–O(3)	121·19	122·38	—
C(1)–C(2)–O(2)	113·59	116·25	—
C(1)–C(2)–C(3)	119·49	121·37	121·10
C(3)–C(2)–O(2)	126·91	122·38	—

The least-squares best plane through the C and O atoms was calculated by the method of Schomaker, Waser, Marsh & Bergman (1959) using an IBM 1620 program prepared by Chu (1963). The equation for this plane is

$$0{\cdot}5912x-0{\cdot}5978y+0{\cdot}3836z=1{\cdot}5448$$

in which x, y, and z are the atomic coordinates *in Å* units referred to the crystallographic axes, and 1·5448 is the distance from the plane to the origin in Å. Perpendicular distances of the atoms from the least-squares plane are as follows:

C(1)	−0·0000 Å	O(2)	−0·0030 Å
C(2)	0·0050	O(3)	−0·0061
C(3)	0·0121	H(2)	−0·2645
O(1)	0·0003	H(3)	0·0966

It is seen that, except for the H atoms, the entire quinone molecule is remarkably planar. The two molecules in the cell are very close to parallel to $1\bar{1}2$ and 112, respectively. These two reflections have equal intensities, and their intensity was the largest observed.

Analysis of the thermal motion

An analysis of the thermal vibration ellipsoids of the atoms and of the rigid-body translations and librations of the molecules has been made with the use of an IBM 7090 program of Gantzel, Coulter & Trueblood adapted to the Michigan System by Shiono (1964). The β_{ij}'s (Table 3) were used to calculate the observed U_{ij}'s of the atoms relative to an orthogonal set of axes paral-

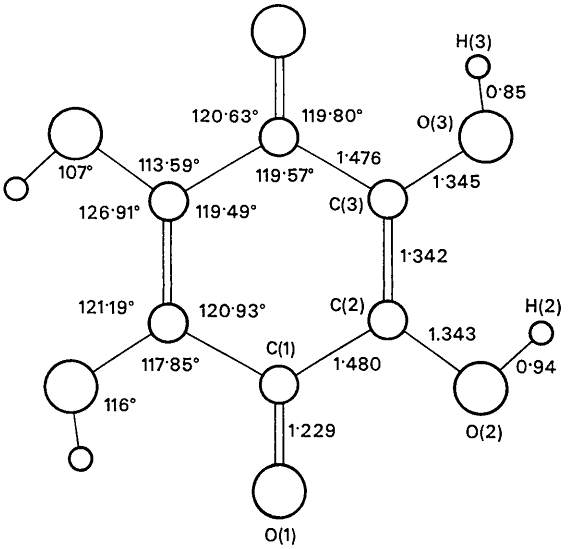

Fig. 4. Diagram of the centrosymmetrical quinone molecule. Bond distances are in Å. The molecule is remarkably planar, except for the hydrogen atoms.

Table 5. *Observed and calculated* U_{ij}

(In 10^{-2} Å^2)

Atom	U_{11}		U_{22}		U_{33}		U_{12}		U_{13}		U_{23}	
	obs.	calc.	obs.	calc.	obs.	calc.	obs.	calc.	obs.	calc.	obs.	calc.
C(1)	2·98	3·00	3·08	3·15	3·38	3·24	−0·55	−0·49	0·13	0·14	−0·11	−0·11
C(2)	3·12	3·23	3·38	3·42	3·13	3·23	−0·43	−0·55	0·41	0·38	−0·17	−0·28
C(3)	3·12	2·99	3·70	3·30	3·22	3·23	−0·34	−0·50	0·34	0·32	−0·03	−0·06
O(1)	3·95	3·87	3·76	3·86	4·15	4·17	−1·34	−1·20	0·49	0·50	−0·61	−0·73
O(2)	4·78	4·78	4·88	4·85	4·19	4·23	−1·44	−1·44	1·34	1·40	−1·62	−1·44
O(3)	3·79	3·86	4·24	4·46	4·33	4·30	−1·39	−1·31	1·22	1·19	−0·65	−0·57

Table 6. *Values of* τ_{ij} (10^{-2} Å^2) *and* ω_{ij} (deg^2) *and their standard deviations*

$$\tau=\begin{pmatrix} 2{\cdot}66 & -0{\cdot}21 & -0{\cdot}01 \\ & 2{\cdot}87 & 0{\cdot}13 \\ & & 2{\cdot}86 \end{pmatrix}, \quad \omega=\begin{pmatrix} 13{\cdot}06 & 6{\cdot}84 & -2{\cdot}78 \\ & 12{\cdot}99 & 2{\cdot}54 \\ & & 8{\cdot}88 \end{pmatrix}$$

$$\sigma\tau=\begin{pmatrix} 0{\cdot}08 & 0{\cdot}07 & 0{\cdot}07 \\ & 0{\cdot}08 & 0{\cdot}07 \\ & & 0{\cdot}08 \end{pmatrix}, \quad \sigma\omega=\begin{pmatrix} 0{\cdot}79 & 0{\cdot}56 & 0{\cdot}58 \\ & 0{\cdot}77 & 0{\cdot}54 \\ & & 0{\cdot}84 \end{pmatrix}$$

lel to *a*, *b*, and *c** (Table 5) by procedures of Cruickshank (1956*a*) and Rollett & Davies (1955). At the same time the principal axes of the atomic ellipsoids and their direction cosines were determined (not presented here). A comprehensive description of the thermal motion is best given by a treatment of the molecule as a rigid body in anisotropic vibration. The motion of the molecule can then be expressed in terms of two symmetric tensors, τ giving the anisotropic translational vibrations of the center of mass and ω the anisotropic angular oscillations (librations) about the center of mass (Cruickshank, 1956*b*). The τ and ω tensors for THQ are shown in Table 6 together with their estimated standard deviations, σ, relative to the orthogonal set of axes. The mean-square amplitudes of translation, root-mean-square amplitudes of angular oscillation, and direction cosines of the principal axes of the τ and ω tensors are listed in Table 7.

For interpretation in terms of the THQ molecule, orthogonal molecular axes (Fig. 5) were defined as follows: Origin at the center of the molecule: Axis M_1, through the C(1)–O(1) bonds; Axis M_3, from center through the midpoint of the C(2)–C(3) bond; Axis M_2 perpendicular to axes 1 and 3 at the origin. The direction cosines of these axes relative to the orthogonal set of axes are:

Axis	$\cos \alpha_i$	$\cos \beta_i$	$\cos \gamma_i$
M_1	0·5101	0·7965	0·3246
M_2	0·5942	−0·5992	0·5365
M_3	0·6240	−0·0774	−0·7776

The r.m.s. amplitudes of the principal axes of τ, 0·18 Å, 0·17 Å, and 0·16 Å are so nearly the same that the translational oscillations of the molecule may be considered essentially isotropic. The angles between the axes of the ω tensor and the molecular axes were calculated to be: angle between ω_1 and M_1 = 23·6°; angle between ω_3 and M_2 = 2·8°; angle between ω_2 and M_3 = 24·0°. The chief librations of the quinone molecules are about ω_1 and ω_2 whose r.m.s. angular oscillations of 4·5° and 3·4°, respectively, cause the molecule to wobble torsionally about these two axes in its plane (Fig. 5). The torsional motion about the perpendicular to the molecular plane, axis ω_3, is much smaller, 1·9°. These motions appear to be in keeping with the expected ease of oscillation about the molecule's axes of minimum, intermediate, and maximum moments of inertia, if it were a free molecule. In the crystal the molecule is bonded in all directions to other molecules by hydrogen bonds which act to damp these oscillations. The observed torsional oscillations appear to be oriented relative to the directions of the H bonds so as to distort these bonds a minimum.

Given the τ and ω tensors, the **U** tensors for each atom may be calculated, thus providing the calculated U_{ij}'s of Table 5. The r.m.s. difference between the observed and calculated U_{ij}'s is 0·0011 Å^2, a value which indicates that the interpretation in terms of rigid-body vibrations is completely justified. This value corresponds to an e.s.d., σ, for the observed U_{ij}'s of $0{\cdot}0011 \times (36/24)^{\frac{1}{2}} = 0{\cdot}0013$ Å^2, since 12 parameters have been determined from 36 U_{ij}'s. It should be remarked that the e.s.d.'s of the U's and related quantities will be affected by errors (not taken into account) due to absorption and extinction. Cruickshank (1956*c*) has shown that the errors in bond lengths due to rotational

Table 7. *Principal axes of the* τ *and* ω *tensors relative to the set of orthogonal axes*

Mean-square amplitude of translation	Direction cosines		
0·0306 Å^2	0·4013	−0·7646	−0·5044
0·0280 Å^2	0·4200	−0·3357	0·8431
0·0252 Å^2	0·8140	0·5502	−0·1864
Root-mean-square angular oscillation			
4·46°	0·7125	0·7015	−0·0182
3·40°	0·3933	−0·4206	−0·8176
1·88°	0·5811	−0·5753	0·5756

oscillations of molecules may be calculated. These corrections in a molecule such as THQ would be expected to increase the bond lengths of Table 4 and Fig. 4 by not more than one standard deviation, 0·005 Å.

Discussion of the structure

One of the most unusual properties of THQ dihydrate is the glistening black color of its crystals. This seems largely a property of the crystals, since THQ in water solution is light red. The lustrous color even suggests the possibility of some metallic character, such as, perhaps, slight conductivity of electricity. However, a crude experiment which readily demonstrated the conductivity of graphite and of solid iodine did not reveal the slightest indication for conductivity in THQ. The quinoid structure, which is prominent in the THQ molecule, is a known chromophoric group, and it might readily explain the light red color of the aqueous solution but hardly the black color of the crystals.

Strong absorption in the visible region of the spectrum is characteristic of charge transfer complexes, and charge transfer interactions between quinones and aromatic hydrocarbons are known (Chowdhury 1961). Mulliken (1953*a*, *b*) regards such complexes as π-coordination componds in which an electron pair from a π-molecular orbital of the hydrocarbon is partially donated to the antibonding molecular orbital of the other component. The quinones are known as electron acceptors, and chloranil is a strong electron acceptor (Hausser & Mulliken, 1960). The stabilization is often greatest when the molecules of the complex are not directly superposed. Complexes involving only one molecular species, *i.e.* self-complexes, can occur as in chloranil (Chu, Jeffrey & Sakurai, 1962). Unusually short non-bonded C to O distances of 2·85 Å permit each carbonyl oxygen to approach an sp^2 carbon atom approximately along the direction of the polar axis of the π-bonded orbital, with the result that each molecule partakes of four such interactions which link the molecules into two-dimensional nets parallel to the *ab* plane. The color of chloranil is yellow.

Certain short C · · · O distances in THQ suggest that self-complexing through charge transfer may be a source of the color in THQ crystals. Some have considered the C · · · O van der Waals distance to be 3·10 Å, based on the half-thickness of the aromatic molecule as 1·70 Å and an O atom radius of 1·40 Å (Pauling 1960). However, a C · · · O distance as small as 3·10 Å has been observed in only a few unusual compounds, and in these cases one or more of the C · · · O distances may be even shorter than 3·10 Å. See Chu, Jeffrey & Sakurai (1962), also Davies & Blum (1955), and Bolton (1963*a*, *b*; 1964). The normal C · · · O distance, on the other hand, appears to be of the order of 3·30–3·40 Å. For instance, in *p*-benzoquinone the shortest C · · · O distances are 3·34, 3·39, and 3·45 Å (Trotter 1960). In barbituric acid dihydrate the C · · · O interlayer distances are 3·33 and 3·35 Å (Jeffrey, Ghose & Warwicker, 1961). Phenylcyclobutenedione has C · · · O distances of 3·29, 3·32, and 3·37 Å which Wong, Marsh & Schomaker (1964) considered 'noteworthily' short. Likewise, in the anthracene–*s*-trinitrobenzene complex the smallest C · · · O distance is 3·26 Å (Brown, Wallwork & Wilson, 1964). Examples such as these suggest that any C · · · O distance shorter than about 3·30 Å is abnormally short.

In THQ each quinone molecule has four C to O distances, two each of C(1) · · · O(1′) and O(1) · · · C(1′) of 3·09 Å, and four C to O distances, two each of C(3) · · · O(3′) and O(3) · · · C(3′), of 3·18 Å with neighboring molecules (Fig. 5, Table 8). (Unprimed atoms are on one molecule; primed atoms are on neighboring molecules). These short C · · · O distances are directed approximately along the axis of the π-bonded orbital of the sp^2 C(1) and C(3) carbon atoms. Thus, each molecule participates in eight such short C · · · O interactions linking the quinone chains into sheets in the *ab* plane.

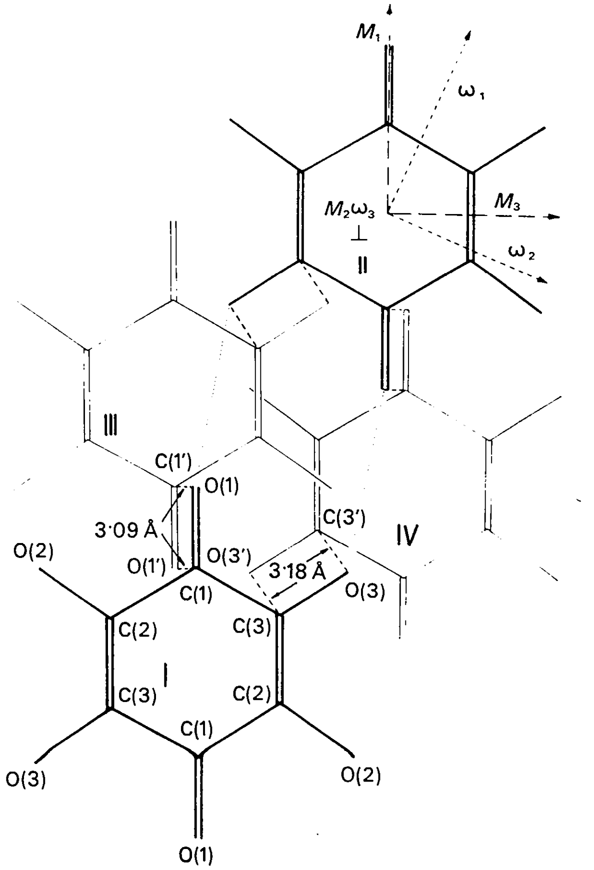

Fig. 5. Projection of the quinone-molecule chains perpendicular to their plane. Molecules I and II, through the hydrogen bonds shown (dotted lines), are part of a chain in the plane of the paper. Molecule III is in a chain above the plane of the paper, and molecule IV in a chain below the plane of the paper. The molecular axes, M_1, M_2, M_3, and the principal axes of the ω libration tensor, ω_1, ω_2, ω_3, have been indicated on molecule II. Abnormally short van der Waals C · · · O distances are shown in projection.

Table 8. *Intermolecular distances shorter than* 3·4 Å

Atoms	Distance	Identifying numbers*
C(1) · · · C(1)	3·331 Å	1–21
C(1) · · · O(1)	3·088	1–15
C(3) · · · O(3)	3·179	22–6
C(1) · · · O(3)	3·208	23–6
C(2) · · · O(1)	3·276	2–15
O(2) · · · O(4)	2·654	5–17 H.B.†
O(1) · · · O(3)	2·744	16–19 H.B.
O(2) · · · O(4)	2·926	5–7 H.B.
O(4) · · · O(4)	2·968	17–18 H.B.
O(3) · · · O(4)	3·055	6–17
O(1) · · · O(4)	3·070	4–7
O(3) · · · O(4)	3·159	6–18
O(1) · · · O(1)	3·198	15–16
O(1) · · · O(1)	3·316	4–15
O(2) · · · O(3)	3·335	5–19

* These numbers refer to numbers on the figures used throughout the study for identification and in computing.

† H.B. identifies these distances as hydrogen bonds.

Sakurai (1965) has observed similar abnormally short C to O distances, 3·2 Å, between the parallel hydroquinone and quinone molecules in the newly found triclinic form of quinhydrone. The structure of monoclinic quinhydrone (Matsuda, Osaki & Nitta, 1958) also reveals these same shortened C to O distances. Sakurai proposes (as did Matsuda, Osaki & Nitta) that the strong color of quinhydrone is explained by a charge transfer complex in which hydroquinone is the donor molecule and quinone is the acceptor molecule, the shortened C to O distances being strong evidence for such complexing. These short C · · · O distances in THQ, although still somewhat longer than the 2·85-Å value reported for chloranil, likewise strongly suggest charge transfer self-complexing similar to the charge transfer complexes observed between quinones and a variety of other donor-type molecules.

Additional intermolecular distances shorter than 3·4 Å are listed in Table 8. Possibly the C(1) · · · C(1) distance of 3·331 Å, the C(1) · · · O(3) distance of 3·208 Å, and the C(2) · · · O(1) distance of 3·276 Å might also contribute to charge transfer forces but their directions are farther from the axis of the π-bonded orbital on the carbon atoms than in the case of those still shorter distances already discussed.

Intermolecular distances involving van der Waals contacts between hydrogen atoms and other atoms have not been listed in Table 8. Those H · · · O distances which constitute the electrostatic half of hydrogen bonds vary from 1·81 Å to 2·08 Å and are not to be considered as normal van der Waals contacts. Of 22 other non-bonded hydrogen atom contacts shorter than 3·4 Å only three are abnormally short. The O(1) · · · H(4′) distance (4–11 on Fig. 3) is only 2·15 Å. This distance just misses being an electrostatic half of a hydrogen bond, for the O(1) · · · O(4) distance of 3·070 Å loses out to the O(2) · · · O(4) distance of 2·926 Å as the hydrogen bond to the water molecule. The intrachain distance between H(4) on 17 and H(4′) on 7 is only 2·15 Å, and the distance between H(2) and H(4) on 17 is only 2·13 Å. It seems that these shorter distances arise as the best equilibrium distances in each case compatible with the network of hydrogen bonds that is formed.

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Crystal Structures of D,L-Homocysteine Thiolactone Hydrochloride: Two Polymorphic Forms and a Hybrid*

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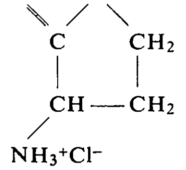
The structures of three different crystalline forms of D,L-homocysteine thiolactone hydrochloride have been determined by X-ray diffraction techniques. Two of the crystal forms are polymorphs: one crystallizes in space group *Pbca* and the other crystallizes in space group $Pbc2_1$. The third form is a hybrid in which both of these polymorphs coexist. The *Pbca* and $Pbc2_1$ domains of the hybrid crystal scatter X-rays independently but have their corresponding unit-cell axes oriented in the same directions.

The structures of the polymorphs were arrived at by conventional heavy-atom Fourier and vector-coincidence methods, and each structure was refined by two different least-squares criteria, one based on reflection amplitudes and the other based on reflection intensities. To verify the structure of the hybrid form, a linear combination of reflection intensities from the *Pbca* and $Pbc2_1$ crystals was scaled by least squares to the reflection intensities of the hybrid crystal; the agreement obtained between the reflection intensities corresponds to a conventional R index of 9 %.

The crystal structures of the two polymorphs are very similar. The five-membered thiolactone ring is in the envelope conformation, with four atoms lying in a plane from which the β carbon atom is displaced by approximately 0·7 Å. In both structures the molecules are packed in layers perpendicular to the unit-cell c axis, and there is a network of hydrogen bonds between the ammonium group and chloride ions. Apparent small differences in bond-lengths and angles between molecules in the two structures are not significant; differences of about the same magnitude were produced by the two alternate methods of refinement for each individual polymorph.

Introduction

The structures of three different crystalline forms of D,L-homocysteine thiolactone hydrochloride (HTL)



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* Most of the material in this communication has been abstracted from a Ph.D. thesis submitted by S.T.F. to the University of Washington in 1964.