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Molecular Complexes Exhibiting Polarization Bonding. VIII. The Crystal Structures of the 1:1 Complexes formed by *p*-Chlorophenol and *p*-Bromophenol with *p*-Benzoquinone.

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The isomorphous structures of the 1:1 complexes formed by *p*-chlorophenol and *p*-bromophenol with *p*-benzoquinone were determined by Patterson and electron-density projections of the bromo-complex. The structure of the chloro-complex was then refined by anisotropic structure-factor least-squares methods. The crystals are triclinic, space group $P\bar{1}$, with two molecules of the complex in each unit cell. The phenol and quinone molecules are stacked alternately, plane-to-plane, in infinite columns and the columns are linked in pairs by hydrogen bonds between the phenol and quinone molecules. The structural relationships between the 1:1 and 2:1 complexes are discussed.

Introduction

Although phenol forms only a 2:1 complex with *p*-benzoquinone (Harding & Wallwork, 1953), *p*-halogenophenols form both 2:1 and 1:1 complexes. The isomorphous structures of the 2:1 complexes of *p*-chlorophenol (PCP) and *p*-bromophenol (PBP) with *p*-benzoquinone (Q) have already been described in part VII (Shipley & Wallwork, 1967) and the present paper describes the isomorphous structures of the corresponding 1:1 complexes (QmonoPCP and QmonoPBP). The reason for the existence of both 2:1 and 1:1 complexes is discussed in terms of the roles of charge-transfer interaction and hydrogen bonding.

Experimental

Orange needle-shaped crystals of the QmonoPCP and QmonoPBP complexes were deposited when warm concentrated solutions of the appropriate components in light petroleum were mixed in a 1:1 molecular ratio and allowed to cool. In plane-polarized light the crystals exhibited pleochroism, the colour being deeper when the electric vector was parallel to the needle (*b*) axis, suggesting that the molecules are probably stacked in columns parallel to this axis. Crystals were sealed into thin-walled glass capillary tubes for X-ray photography and Weissenberg photographs with the crystals oscillating about each of the three crystallographic axes showed that the crystals belong to the triclinic system and permitted the determination of the unit-cell dimensions for each of the complexes. Exposures for intensity estimation were made with Cu $K\alpha$ radiation by the multiple-film, equi-inclination Weissenberg technique. The $0kl$ and $h0l$ reflexions were recorded in the case of the QmonoPBP complex, and $0kl$ to $3kl$ and $h0l$ to $h4l$ reflexions in the case of the QmonoPCP complex. The intensities for the QmonoPBP structure were measured by a photometer method (Wallwork & Standley, 1954) and those for QmonoPCP were estimated visually by comparison with a series of timed exposures of a selected reflexion. The intensities were converted into F_o^2 values in the usual way and no corrections were made for absorption or extinction effects. For the QmonoPCP complex the number of independent F_o^2 values was 1127.

Crystal data

(*a*) QmonoPBP, $C_6H_4O_2.p\text{-}BrC_6H_4OH$, $M=281{\cdot}1$
$a=6{\cdot}84$, $b=7{\cdot}86$, $c=12{\cdot}49$, all $\pm 0{\cdot}03$ Å
$\alpha=105{\cdot}8°$, $\beta=104{\cdot}4°$, $\gamma=58{\cdot}7°$, all $\pm 0{\cdot}5°$
$U=548$ Å^3; $F(000)=280$
$D_m=1{\cdot}71$ g.cm^{-3}, $Z=2$, $D_c=1{\cdot}70$ g.cm^{-3}.
Space group $P\bar{1}$ (indicated by pyroelectric and statistical tests); Cu $K\alpha$ ($\lambda=1{\cdot}542$ Å).

(*b*) QmonoPCP, $C_6H_4O_2.p\text{-}ClC_6H_4OH$, $M=236{\cdot}6$
$a=6{\cdot}80$, $b=7{\cdot}90$, $c=12{\cdot}25$, all $\pm 0{\cdot}03$ Å
$\alpha=106{\cdot}9°$, $\beta=106{\cdot}5°$, $\gamma=58{\cdot}0°$, all $\pm 0{\cdot}5°$
$U=527$ Å^3; $F(000)=244$
$D_m=1{\cdot}48$ g.cm^{-3}, $Z=2$, $D_c=1{\cdot}49$ g.cm^{-3}.
Space group $P\bar{1}$ (indicated by statistical tests); Cu $K\alpha$ ($\lambda=1{\cdot}542$ Å).

Structure determination and refinement

The $h0l$ Patterson projection for the QmonoPBP structure revealed the positions of the bromine atoms, and a Fourier electron density projection based on signs calculated for the contributions of the bromine atoms only to the structure factors showed the general arrangement of the whole structure, with phenol and quinone molecules almost exactly overlapping in this projection. Successive structure-factor calculations, with a constant overall temperature factor $B=3{\cdot}5$ Å^2,

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Table 1. *Fractional atomic coordinates for QmonoPBP*

	x/a	*y/b*	*z/c*
Br	0·868	0·319	0·080
O(1)	0·365	0·312	0·410
O(2)	0·811	0·870	0·123
O(3)	0·376	0·831	0·406
C(1)	0·714	0·338	0·185
C(2)	0·472	0·379	0·156
C(3)	0·360	0·372	0·229
C(4)	0·447	0·317	0·339
C(5)	0·716	0·271	0·367
C(6)	0·828	0·279	0·295
C(7)	0·714	0·786	0·185
C(8)	0·472	0·905	0·156
C(9)	0·360	0·900	0·229
C(10)	0·477	0·840	0·338
C(11)	0·716	0·797	0·367
C(12)	0·828	0·804	0·295

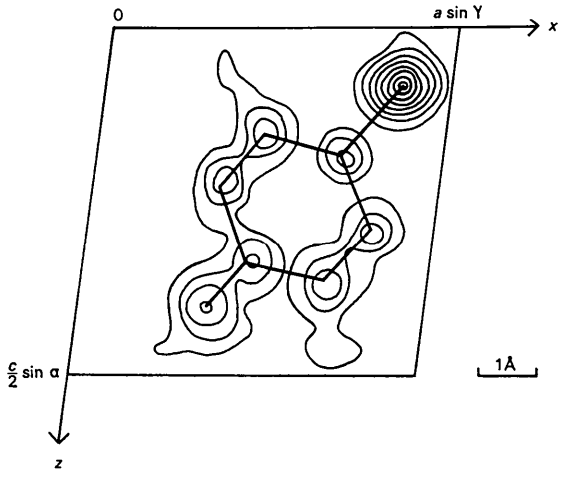

Fig. 1. QmonoPBP, *h0l* electron density projection. Contours at equal arbitrary intervals. The position of a *p*-bromophenol molecule is indicated and a *p*-benzoquinone overlaps it almost exactly in this projection.

Table 2. *Observed structure amplitudes and final calculated structure factors for QmonoPBP* ($\frac{1}{2}$ *absolute scale*)

hkℓ	$\lvert F_o\rvert$	F_c	hkℓ	$\lvert F_o\rvert$	F_c	hkℓ	$\lvert F_o\rvert$	F_c	hkℓ	$\lvert F_o\rvert$	F_c	hkℓ	$\lvert F_o\rvert$	F_c	hkℓ	$\lvert F_o\rvert$	F_c
h0ℓ projection																	
0014	3.4	+3.9	202	15.9	+16.6	40$\bar{2}$	10.2	+6.2	008	13.4	-11.2	02$\bar{2}$	17.6	-20.4	04$\bar{1}$	18.5	+21.1
0013	6.1	+5.6	201	26.2	+29.8	40$\bar{3}$	13.4	+13.8	007	18.5	-16.5	02$\bar{3}$	27.6	-44.5	04$\bar{5}$	6.5	+7.7
0012	6.9	+7.7	200	9.2	-11.6	40$\bar{5}$	14.9	+15.3	006	16.5	-18.4	02$\bar{4}$	10.5	+8.1	04$\bar{7}$	8.8	-4.4
0011	8.2	+4.2	20$\bar{1}$	28.6	-33.7	40$\bar{7}$	4.7	+2.8	005	13.7	-9.9	02$\bar{5}$	10.5	+6.8	04$\bar{8}$	11.2	-10.0
0010	7.0	+4.2	20$\bar{2}$	14.9	-12.3	40$\bar{10}$	6.8	-2.3	004	28.8	-33.1	02$\bar{6}$	9.3	+8.3	04$\bar{9}$	14.8	-19.4
009	3.3	-3.6	20$\bar{3}$	24.2	-25.5	40$\bar{12}$	4.1	-6.1	003	4.4	+1.7	02$\bar{7}$	17.3	+19.1	04$\bar{10}$	13.0	-9.9
008	10.1	+8.7	20$\bar{4}$	18.0	-16.2	40$\bar{13}$	4.7	-5.7	002	14.1	+12.4	02$\bar{8}$	17.4	+17.7	04$\bar{11}$	7.5	-4.4
007	15.8	-13.5	20$\bar{5}$	16.5	-17.8	509	4.3	-3.9	0115	1.7	-8.2	02$\bar{9}$	17.2	+16.4	04$\bar{12}$	6.0	-4.7
006	14.8	-15.9	20$\bar{6}$	9.7	+7.2	508	3.3	-4.8	0114	5.2	-8.6	02$\bar{10}$	13.4	+2.0	04$\bar{14}$	6.2	+3.5
005	11.7	-8.1	20$\bar{7}$	17.6	+14.2	507	3.3	-4.4	0113	7.6	-6.6	02$\bar{12}$	8.9	-2.9	0510	4.9	-7.1
004	26.9	-32.3	20$\bar{8}$	9.5	+5.5	504	3.3	+5.9	0112	9.1	-2.5	02$\bar{13}$	10.5	-8.2	057	5.9	+7.4
003	2.6	+1.7	20$\bar{9}$	18.8	+15.5	503	7.5	+6.5	0111	6.7	+4.3	02$\bar{14}$	11.0	-9.4	056	8.8	+13.0
002	14.0	+12.6	20$\bar{12}$	6.6	+10.9	502	10.4	+9.3	0110	11.0	+11.0	02$\bar{15}$	5.3	-8.5	055	8.8	+12.8
001	12.2	+17.9	20$\bar{14}$	4.8	-3.1	501	9.0	+9.4	019	15.2	+16.9	0312	8.1	+8.5	054	9.7	+10.2
1014	4.7	-4.5	3012	4.9	+3.6	500	5.9	+6.7	018	18.4	+19.0	0311	8.7	+5.5	053	9.4	+5.3
1013	3.4	-1.6	3011	4.4	+5.1	50$\bar{2}$	8.7	-4.8	017	15.0	+13.7	039	6.3	-5.1	051	9.0	-7.7
1012	4.5	-2.9	3010	4.1	+3.6	50$\bar{3}$	8.8	-7.0	016	10.7	+3.9	038	11.1	-13.4	050	12.8	-15.4
1011	4.3	-5.5	309	4.2	+3.9	50$\bar{4}$	15.6	-15.7	015	14.5	-9.5	037	13.5	-17.9	05$\bar{1}$	13.7	-16.4
1010	5.2	+6.9	305	23.3	-29.8	50$\bar{5}$	6.6	-2.9	014	16.4	-20.8	036	16.6	-16.9	05$\bar{2}$	14.6	-19.2
109	10.4	+9.1	304	20.6	-20.8	50$\bar{6}$	8.6	+4.9	013	22.4	-24.2	035	9.3	-10.2	053	7.1	-6.4
108	14.9	+13.8	303	6.9	-3.2	50$\bar{7}$	3.8	+8.1	012	20.6	-25.6	034	5.8	+1.4	056	9.7	+9.9
107	8.4	+7.3	302	9.9	-7.2	50$\bar{8}$	5.0	+2.8	011	16.3	-22.3	033	10.7	+8.1	057	11.6	+10.8
106	18.3	+17.1	301	26.2	+28.6	50$\bar{11}$	5.6	-7.5	010	9.4	-8.4	032	18.5	+13.9	058	13.3	+12.2
105	23.1	+25.8	300	19.9	+16.3	50$\bar{12}$	3.5	+3.7	01$\bar{1}$	5.8	+1.5	031	18.7	+27.0	059	8.8	+8.1
104	22.4	-27.3	30$\bar{1}$	18.2	+18.0	605	3.5	-6.2	01$\bar{2}$	10.2	+11.3	030	16.9	+20.9	05$\bar{12}$	8.7	-6.9
103	20.8	-21.6	30$\bar{2}$	18.2	+18.0	604	1.7	-2.9	01$\bar{3}$	18.2	+22.9	03$\bar{1}$	10.8	+13.1	05$\bar{13}$	6.9	-8.8
102	28.5	-40.8	30$\bar{3}$	8.8	+10.0	603	2.9	-5.2	01$\bar{4}$	14.6	+22.4	03$\bar{2}$	8.3	+7.5	05$\bar{14}$	4.4	-8.4
101	29.9	-50.2	30$\bar{4}$	6.4	+6.6	602	3.2	-3.9	01$\bar{5}$	22.7	+22.1	033	11.3	-10.6	067	3.5	-8.5
100	13.6	+12.4	30$\bar{5}$	18.4	-14.3	601	3.7	-3.1	01$\bar{6}$	17.2	+13.9	03$\bar{4}$	9.8	-12.6	063	7.1	+9.2
10$\bar{2}$	6.7	-6.7	30$\bar{6}$	12.6	-5.8	600	5.2	+5.4	018	8.5	-5.3	03$\bar{5}$	14.1	-15.1	062	9.3	+6.9
10$\bar{3}$	17.1	+15.3	30$\bar{7}$	5.7	-5.8	60$\bar{1}$	8.4	+6.3	01$\bar{9}$	17.5	-13.5	036	19.6	-16.7	061	10.2	+11.7
10$\bar{4}$	19.9	+21.8	30$\bar{8}$	13.1	-13.1	60$\bar{2}$	3.5	+2.1	01$\bar{10}$	17.6	-15.6	037	16.4	-13.0	060	13.7	+14.3
10$\bar{5}$	22.1	+22.3	30$\bar{9}$	4.8	-6.6	60$\bar{3}$	6.8	+7.3	01$\bar{11}$	16.8	-14.7	038	10.7	-9.2	06$\bar{1}$	7.9	+3.6
10$\bar{6}$	12.0	+13.5	30$\bar{12}$	5.0	+3.0	60$\bar{5}$	4.6	-2.5	01$\bar{12}$	11.4	-8.4	03$\bar{10}$	9.6	+5.1	06$\bar{2}$	9.8	+7.0
10$\bar{7}$	7.1	+9.8	30$\bar{13}$	5.7	+4.1	60$\bar{7}$	3.5	-3.4	01$\bar{14}$	5.1	+3.6	03$\bar{11}$	14.1	+11.3	06$\bar{3}$	8.9	-5.2
10$\bar{8}$	5.2	-2.9	30$\bar{14}$	4.5	+5.0	608	5.3	-3.6	01$\bar{15}$	6.1	+6.9	03$\bar{12}$	15.0	+14.7	06$\bar{4}$	18.6	-15.2
10$\bar{9}$	10.6	-6.3	4010	4.5	-5.0	609	4.3	-4.4	0212	8.8	-9.0	03$\bar{13}$	11.2	+9.9	06$\bar{5}$	10.7	-11.7
10$\bar{10}$	16.6	-17.5	408	3.0	+4.1	60$\bar{10}$	4.1	-5.0	0211	11.0	-12.7	03$\bar{14}$	7.6	+5.1	06$\bar{6}$	11.0	-13.2
10$\bar{11}$	8.4	-7.4	407	6.7	+8.6	701	3.5	-4.2	0210	12.8	-12.2	03$\bar{15}$	4.2	-0.4	067	8.7	-8.2
10$\bar{13}$	3.8	-5.1	406	15.8	+20.0	70$\bar{3}$	2.4	-2.3	029	12.1	-7.3	049	9.7	+11.9	068	7.2	-4.1
2011	6.3	-4.1	405	4.6	+0.9	70$\bar{4}$	2.5	-2.0	027	7.5	+4.6	048	9.2	+8.5	069	8.9	+6.4
2010	9.6	-7.3	404	5.1	-3.1	0kℓ projection			026	13.6	+14.4	047	10.7	+5.6	06$\bar{10}$	9.0	+11.0
209	10.5	-10.1	403	3.2	-1.9	0014	7.5	+7.3	025	7.4	+10.0	044	14.5	-12.3	06$\bar{11}$	6.5	+7.3
208	8.4	-6.0	402	11.6	-11.0	0013	10.5	+10.3	024	20.8	+22.7	043	8.4	-15.6	06$\bar{12}$	6.5	+8.1
207	7.1	-12.9	401	10.2	-8.7	0012	11.2	+12.0	023	9.6	+13.3	041	29.3	-27.1	073	6.0	-6.1
206	3.1	+3.6	400	13.0	-13.7	0011	12.5	+9.0	022	18.6	+13.5	040	15.5	-11.3	072	8.1	-6.3
205	11.9	+12.5	40$\bar{1}$	11.4	-8.8	0010	11.9	+5.6	021	37.4	+45.3	04$\bar{1}$	12.5	+13.9	07$\bar{1}$	8.5	+5.2
204	16.5	+15.6	40$\bar{2}$	6.4	+6.0	009	7.9	-4.3	020	35.9	-48.1	04$\bar{2}$	9.8	+6.3	07$\bar{2}$	6.4	+13.5
203	23.2	+25.4	40$\bar{3}$	2.8	+0.5				02$\bar{1}$	33.7	-53.8	04$\bar{3}$	25.7	+32.2			

and Fourier syntheses reduced the agreement index, $R=\Sigma\,||F_o|-|F_c||/\Sigma\,|F_o|$, to 0·21 and gave the electron density map shown in Fig. 1. The *h*0*l* projection of the isomorphous QmonoPCP structure was solved and refined similarly to the point where R was 0·25.

Attempts were made to derive the 0*kl* projection for QmonoPBP in the same way, but the Patterson map could not be interpreted unambiguously even when compared with the corresponding projection of the QmonoPCP structure. A solution was obtained eventually after calculation of a three-dimensional Patterson function for the QmonoPCP structure, and the 0*kl* projection for the QmonoPBP complex was refined to an R value of 0·24 with the use of temperature factors $B=1{\cdot}5$ Å^2 for bromine and 3·5 Å^2 for all other atoms. Table 1 gives the final, approximate, atomic coordinates for QmonoPBP ($\sigma\simeq 0{\cdot}005$), and the final calculated structure factors are compared with the observed structure amplitudes in Table 2.

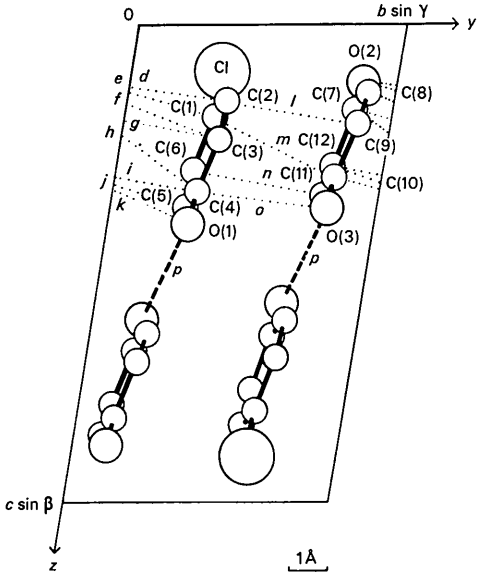

Fig. 2. The QmonoPCP structure projected along the *a* axis showing principal intermolecular contacts:

(*d*)	C(2)...O(2)	3·37 Å	(*e*)	C(1)...O(2)	3·24 Å
(*f*)	C(3)...C(8)	3·31	(*g*)	C(3)...C(7)	3·38
(*h*)	C(4)...C(7)	3·36	(*i*)	C(4)...C(12)	3·40
(*j*)	C(5)...C(12)	3·31	(*k*)	O(1)...C(10)	3·36
(*l*)	C(9)...C(2)	3·45	(*m*)	C(10)..C(1)	3·36
(*n*)	C(11)..C(6)	3·42	(*o*)	O(3)...C(4)	3·44
(*p*)	O(1)...O(3)	2·70	(H-bond).		

The approximate coordinates for the QmonoPCP structure, derived from the *h*0*l* projection and the three-dimensional Patterson function, were refined by the block-diagonal structure-factor least-squares method with the program written by Dr J. S. Rollett (Pepinsky, Robertson & Speakman, 1961) for the Oxford MERCURY computer and a modification of the same program written by Dr R. D. Diamand for the ATLAS computer. Atomic positions and anisotropic temperature factors for all atoms other than hydrogen were refined, together with an overall scale factor (a total of 145 parameters), 1127 independent observations being used. In the early stages of the refinement the F_o values were all given unit weights, but later the terms larger than a selected value F^* were downweighted according to the scheme $\sqrt{w}=F^*/F_o$ with F^* equal to 600 (on the scale of Table 4). The refinement converged with $R=0{\cdot}18$, probably owing to the use of rather restricted visual data of only moderate quality. In view of this, it is felt that little significance can be attached to the anisotropy of the atomic vibrations and only spherically averaged values are quoted in Table 3. The final atomic positions and their standard deviations obtained by inversion of the normal equations matrix are also given in Table 3. The observed structure factors are compared with the final calculated values in Table 4.

Results and discussion

In both complexes the phenol and quinone molecules are stacked alternately along the *b* axis but with their plane normals tilted approximately 30° away from this axis. This tilt brings the C–O or the C=O groups of

Table 3. *Final fractional atomic coordinates and their standard deviations and spherically averaged thermal parameters for QmonoPCP*

	x/a	$\sigma(x/a)$	y/b	$\sigma(y/b)$	z/c	$\sigma(z/c)$	B_{av}
Cl	0·8515	0·0006	0·3359	0·0006	0·0918	0·0002	3·0
O(1)	0·3808	0·0019	0·3023	0·0017	0·4310	0·0009	4·0
O(2)	0·8141	0·0021	0·8693	0·0018	0·1183	0·0010	4·9
O(3)	0·3711	0·0018	0·8133	0·0016	0·3816	0·0008	3·5
C(1)	0·7096	0·0023	0·3349	0·0019	0·1924	0·0009	2·5
C(2)	0·4826	0·0024	0·3779	0·0020	0·1601	0·0012	3·2
C(3)	0·3712	0·0025	0·3691	0·0021	0·2353	0·0011	3·1
C(4)	0·4952	0·0025	0·3196	0·0020	0·3441	0·0011	3·0
C(5)	0·7195	0·0027	0·2832	0·0021	0·3752	0·0011	3·1
C(6)	0·8328	0·0027	0·2940	0·0022	0·3013	0·0012	3·3
C(7)	0·7123	0·0026	0·8598	0·0021	0·1768	0·0012	3·4
C(8)	0·4703	0·0025	0·8977	0·0020	0·1395	0·0012	3·3
C(9)	0·3628	0·0025	0·8736	0·0020	0·2055	0·0011	2·9
C(10)	0·4783	0·0026	0·8239	0·0022	0·3206	0·0011	3·0
C(11)	0·7148	0·0025	0·7869	0·0023	0·3572	0·0013	3·6
C(12)	0·8328	0·0029	0·8066	0·0023	0·2949	0·0014	3·9

successive molecules approximately over the centres of the rings of the adjacent molecules as shown in Fig. 3, indicating a specific interaction involving these groups (Prout & Wallwork, 1966). Such a staggered arrangement of molecules in an infinite stack inevitably brings the C-halogen bond in each phenol molecule into a position adjacent to a quinone ring on one side, and this causes the average molecular separation to be larger in this direction (3·35 Å in QmonoPCP, compared with 3·27 Å in the opposite direction) as well as

Table 4. *Comparison of observed and final calculated structure factors on* 50 *times the absolute scale for QmonoPCP*

The asterisked line gives h, k. Other lines gives values in the order l, F_o, F_c,

*	0	0										
	1	412	392	2	-1847	-2425	4	-2925	-3128	5	-446	-202
	6	-426	-188	7	-803	-530	8	-179	-17	9	233	189
	10	906	770	11	446	402	12	192	158	13	240	185
*	0	1										
	1	-666	-442	2	-1003	-767	3	-1270	-1006	4	-536	-359
	5	-1003	-741	6	797	510	7	1188	770	8	845	574
	9	646	520	10	309	98	12	-563	-581	13	-398	-440
	14	-165	-206	-1	350	256	-2	227	122	-3	838	778
	-5	1174	1048	-6	886	713	-7	-165	-136	-8	-398	-238
	-9	-879	-736	-10	-391	-334	-11	-536	-507	-12	-261	-164
	-13	316	351	-14	302	354	-15	282	462			
*	0	2										
	0	-3083	-3573	1	3873	5636	2	1799	1625	4	1291	1054
	5	-906	-772	6	268	169	8	-494	-346	9	-584	-448
	10	-639	-510	11	-206	-65	13	192	154	-1	-3022	-3808
	-2	-817	-696	-3	-2074	-2700	-4	1373	1342	-5	1174	1025
	-6	227	219	-7	803	819	-8	666	601	-9	762	608
	-10	-330	-344	-11	-391	-503	-12	-206	-186	-13	-412	-396
	-14	-254	-242									
*	0	3										
	0	666	617	1	838	800	2	749	673	3	206	162
	4	-391	-337	5	-467	-401	6	-810	-828	7	-474	-366
	10	282	307	11	501	467	12	309	376	-1	179	130
	-2	192	148	-3	-687	-655	-4	158	123	-5	-199	-120
	-6	-831	-714	-7	-536	-339	-8	-405	-340	-9	412	353
	-10	206	159	-11	220	225	-12	597	550	-13	233	208
	-14	124	109									
*	0	4										
	0	-1415	-1339	1	-2445	-2668	2	1257	1059	3	543	387
	4	-302	-235	5	403	339	6	-220	-144	7	309	208
	8	426	404	9	282	243	11	-192	-230	-1	687	606
	-2	-336	-259	-3	1861	1964	-4	1051	933	-5	-419	-173
	-6	309	250	-7	-206	-133	-8	-350	-306	-9	-902	-883
	-10	-515	-443	-11	247	140	-13	220	216	-14	247	341
*	0	5										
	0	-453	-444	1	-206	-166	2	302	205	3	522	486
	4	433	450	5	220	223	7	-350	-163	8	-254	-223
	9	-275	-196	-1	-398	-381	-2	-570	-541	-5	-206	-150
	-6	220	226	-7	220	288	-8	433	502			
*	0	6										
	0	865	763	2	-1140	-1001	3	302	236	4	288	233
	5	-192	-161	7	-275	-277	-2	996	856	-4	-865	-714
	-6	-302	-248	-7	-364	-276	-8	-398	-321	-9	192	227
	-10	474	498	-12	179	206						
*	0	7										
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	-8	-323	-318	-9	-247	-256						
*	0	8										
	1	240	251	-1	151	145	-2	-302	-318	-3	-364	-396
*	1	0										
	0	-1930	-2549	1	2307	3371	2	1943	2596	3	1202	914
	4	2026	2021	5	-2335	-2199	6	-1862	-1488	8	-858	-641
	9	-323	-348	11	759	649	12	494	505	13	288	197
	-1	-680	-643	-2	-571	-307	-3	-1003	-912	-4	-961	-741
	-5	-1353	-980	-8	611	530	-9	707	714	-10	1058	983
	-11	405	181	-12	-652	-538						
*	1	1										
	0	584	393	1	-213	-192	2	-611	-621	3	-618	-558
	4	-1415	-1116	5	192	150	6	-439	-335	7	-385	-297
	8	659	600	9	584	515	10	673	660	14	-213	-340
	-1	343	477	-2	652	757	-3	700	940	-4	824	1048
	-5	240	181	-7	-872	-853	-8	-988	-853	-9	-913	-857
	-10	-172	-146	-11	838	841						
*	1	-1										
	0	-707	-847	1	-604	-627	4	989	1167	5	439	370
	7	838	741	10	-728	-757	11	-460	-451	13	-199	-214
	14	117	195	-1	-742	-939	-2	-597	-708	-3	-261	-355
	-4	158	134	-5	632	630	-6	666	735	-7	1257	926
	-10	-858	-781	-11	-659	-578	-12	268	227	-13	-316	-331
*	1	2										
	0	-617	-1086	1	-3049	-5180	2	488	413	3	1037	1208
	4	872	659	5	2802	2691	6	-302	-206	7	-755	-490
	8	350	297	9	-282	-222	10	-480	-487	11	-577	-612
	-1	398	344	-2	-419	-564	-3	639	570	-4	261	238
	-5	1119	1078	-6	749	744	-7	646	591	-8	391	339
	-10	-563	-721	-11	-288	-883						
*	1	-2										
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	4	-2184	-2544	5	-817	-756	6	927	754	8	-735	469
	9	652	589	10	858	753	12	-330	-292	14	-371	-474
	-2	652	735	-3	378	396	-4	316	356	-6	-323	-374
	-7	-494	-551	-8	-831	-693	-9	-433	-376	-11	783	698
	-12	349	497									
*	2	3										
	1	577	529	2	687	600	3	529	498	4	364	371
	5	-316	-339	7	-558	-470	8	-728	-647	9	-213	-207
	11	254	371	-3	-124	-95	-4	-810	-789	-5	-494	-527
	-6	-652	-661	-7	-192	-206	-8	398	264	-9	783	672

Table 4 (*cont.*)

-10	1112	1033	-13	-165	-160	-14	-460	-435	-15	96	47
• 1	-3										
0	456	465	2	419	391	3	666	674	4	-220	-134
5	-556	-456	7	-522	-505	8	-412	-393	9	-501	-484
10	185	120	11	522	508	12	110	174	13	261	348
14	-110	-75	-1	192	195	-2	-220	-211	-3	-385	-432
-4	-412	-523	-5	-378	-466	-6	-302	-377	-8	330	427
• 1	4										
0	1413	1512	1	1003	894	2	-2616	-2775	3	-810	-793
4	-378	-255	5	-522	-473	6	975	1089	7	220	114
8	-261	-185	9	282	378	10	254	339	-1	336	362
-2	679	1059	-3	233	207	-4	446	369	-5	-179	-108
-6	-522	-460	-7	-563	-452	-8	-687	-557	-9	-639	-526
-10	-247	-139	-11	908	343	-12	350	331	-13	439	449
-14	357	268	-15	76	31						
• 1	-4										
0	-376	-362	1	-529	-498	3	-790	-689	4	577	485
5	1181	993	6	247	219	7	330	264	10	-536	-491
11	-556	-489	13	-185	-177	-1	-275	-159	-2	-523	-324
-3	199	120	-5	275	311	-6	330	408	-7	330	436
• 1	5										
0	-268	-284	1	-494	-519	2	-282	-236	4	213	220
5	343	414	6	213	194	7	295	405	9	-199	-264
10	-124	-225	-1	-124	-125	-3	-192	-180	-4	137	107
-5	240	189	-6	350	376	-7	419	459	-8	213	284
-10	-481	-541	-11	-357	-431						
• 1	-5										
3	-433	-423	4	-371	-402	8	144	168	9	426	458
10	364	400	-1	261	278	-2	302	365	-3	220	253
• 1	6										
0	-302	-230	1	982	1010	2	1147	1354	3	-714	-716
4	-364	-301	6	-438	-439	7	295	307	9	-172	-316
-2	-536	-475	-3	-446	-354	-4	-201	-271	-5	-371	-388
-9	364	385	-10	343	300						
• 1	-6										
1	144	187	8	295	318	4	295	252	5	-199	-176
6	-137	-180	7	-124	-172	8	-117	-167	9	-261	-276
-2	-144	-162	-3	-192	-239						
• 1	7										
0	261	214	1	144	143	2	-206	-163	3	-240	-240
4	-179	-266	-1	213	186	-7	-199	-194	-8	-192	-167
-9	-261	-318									
• 1	-7										
4	158	145									
• 1	8										
1	-436	-368	2	158	157	3	577	763	-1	-309	-312
-3	227	153	-5	179	188						
• 2	0										
0	-1078	-996	1	2033	2189	2	536	504	3	1305	1189
4	790	659	5	398	219	7	-597	-443	8	-467	-345
9	-536	-514	10	-419	-419	11	-275	-206	12	233	134
13	213	278	-1	-2438	-2760	-2	-378	-303	-3	-1346	-1175
-4	-591	-395	-5	-961	-522	-6	1318	987	-7	1779	1599
-8	385	295	-9	886	705	-10	-879	-704	-11	-975	-874
-12	254	362									
• 2	1										
0	1051	524	1	529	382	2	714	455	3	-426	-249
4	-240	-155	5	-1181	-660	6	-1181	-869	9	288	241
10	446	367	11	522	533	12	201	324	-1	913	675
-2	1140	949	-3	515	430	-4	233	207	-5	-975	-735
-6	-1628	-1286	-7	-282	-269	-8	-700	-613	-9	501	465
-10	254	247	-11	-439	-359	-12	577	875			
• 2	-1										
0	-824	-653	1	-530	-369	2	-577	-605	4	-275	-239
6	797	728	7	563	444	8	591	445	9	316	265
11	-309	-259	12	-371	-499	13	-233	-355	-1	-439	-466
-2	-185	-126	-3	474	435	-4	597	610	-5	941	1058
-6	398	446	-7	-309	-243	-9	-783	-666	-11	-288	-236
-12	-714	-670	-13	213	239	-14	117	310			
• 2	2										
0	-1552	-2150	1	-1062	-1873	3	-906	-709	4	247	75
5	227	100	6	1112	812	7	886	700	8	268	173
9	343	270	11	-227	-288	12	-419	-444	13	-137	-294
-1	692	963	-3	707	1079	-4	625	943	-5	1573	1556
-6	1112	1093	-7	-845	-846	-8	-364	-355	-9	-1435	-1640
-10	-398	-822									
• 2	-2										
0	1895	2522	2	385	236	3	-453	-454	4	-1064	-1069
5	-385	-289	6	-439	-336	7	-282	-217	10	261	335
11	378	420	12	206	263	-1	735	768	-2	-687	-714
-3	323	227	-4	-385	-381	-5	-522	-405	-6	-1339	-1144
-7	-302	-278	-8	700	494	-10	776	713	-12	-254	-435
• 2	3										
0	-185	-175	1	-151	-130	2	268	297	3	755	728
4	577	530	5	790	678	7	-543	-452	8	-282	-162
9	-385	-385	10	-316	-316	11	-213	-221	12	165	202
-1	-144	-161	-2	-522	-697	-3	-597	-705	-4	-687	-745
-5	-501	-553	-6	735	771	-7	453	400	-8	961	1007
-9	172	217	-10	-680	-614	-11	769	665	-13	-435	-447
-14	-213	-264	-15	-728	-631						
• 2	-3										
0	179	160	1	247	199	2	501	491	3	323	290
5	433	433	8	-268	-252	9	-385	-397	10	-350	-340
11	-179	-213	-1	-282	-260	-2	-556	-506	-3	-563	-536
-4	-309	-376	-5	-137	-214	-6	330	373	-7	268	377
• 2	4										
0	1133	1365	4	-529	-498	5	-584	-508	6	-989	-788
8	385	272	10	234	291	11	247	351	-1	481	518
-2	460	613	-3	179	207	-5	-453	-491	-6	-1058	-1333
-7	-501	-493	-8	-886	-961	-9	261	370	-10	1373	1470
-13	261	241	-14	-391	-181						

Table 4 (*cont.*)

0	-803	-739	1	-481	-472	2	-646	-605	3	-749	-650
4	536	485	5	453	360	7	316	298	-1	824	785
-2	549	526	-3	-192	-145	-4	288	296	-5	350	329
-6	240	278	-7	-412	-461						
• 2	5										
1	-240	-246	2	-329	-574	3	-364	-376	6	515	529
7	261	208	9	151	204	-1	-282	-244	-3	316	293
-4	398	379	-5	749	854	-6	247	222	-8	-536	-603
-9	-707	-880	10	398	398						
• 2	-5										
0	268	254	3	-268	-358	-1	336	381	-2	268	243
• 2	6										
2	268	116	3	356	481	4	268	355	5	192	243
7	-536	-533	-1	-371	-267	-2	-364	-341	-3	-364	-377
-4	-371	-310	-5	-330	-243	-6	735	838	-9	872	894
-10	-385	-373	11	-254	-217						
• 2	-6										
2	570	41	-1	-426	-553	-3	206	173			
• 2	7										
1	268	218	2	192	223	6	-192	-189	7	110	68
-2	282	220	-4	343	93	-5	-391	-310	-6	-439	-358
-8	-508	-234									
• 2	8										
3	-286	-291	4	247	119	-3	254	106	-5	336	180
-7	474	320									
• 3	0										
0	-1071	-954	1	-2108	-2326	2	536	405	3	-282	-248
4	1051	1204	5	1861	2150	6	-591	-438	7	-522	-324
9	-419	-405	11	-213	-218	12	-192	-232	-1	446	204
-2	-810	-621	-3	330	66	-4	-151	-36	-5	1902	1647
-6	735	612	-7	-625	-393	-8	501	313	-9	-522	-421
-10	-268	-210	12	-172	-324						
• 3	1										
0	721	662	1	721	760	2	261	229	3	543	415
4	-412	-265	5	-604	-459	6	350	231	7	-742	-611
8	-610	-497	9	-288	-112	11	268	368	12	268	326
-1	577	472	-3	-279	-201	-4	-1099	-1011	-5	-728	-555
-6	-405	-389	-7	-679	-664	-8	426	308	-9	172	197
-10	714	748	11	501	805						
• 3	-1										
1	-550	-548	2	-398	-404	3	-707	-743	4	-385	-429
5	536	536	8	371	375	10	391	438	11	199	117
-1	426	326	-2	728	567	-3	762	624	-4	783	651
-7	-350	-281	-8	-838	-715	-9	-419	-407	-10	-494	-414
-12	391	394									
• 3	2										
0	1373	1704	1	254	220	2	-2088	-2478	3	-666	-475
4	-1089	-1417	5	-343	-233	6	1593	1484	9	357	398
11	179	106	-1	286	208	-2	687	1125	-3	453	678
-4	1346	1857	-5	-906	-675	-6	-2012	-1808	-8	-687	-928
• 3	-2										
0	-611	-588	1	1058	1060	2	323	351	3	968	1046
4	955	864	5	-1449	-1479	6	-735	-714	8	-254	-356
9	165	116	-1	-385	-310	-2	227	174	-3	-330	-327
-4	-446	-371	-5	-577	-646	-6	446	252	-7	261	218
• 3	3										
0	-439	-460	1	-282	-289	4	529	527	5	330	295
6	247	221	7	687	641	9	-302	-354	10	-227	-300
11	-282	-354	-1	-549	-610	-2	-433	-532	-3	-536	-628
-4	350	382	-5	625	582	-7	927	869	-8	227	185
-9	426	394	10	378	296	-11	-1003	-806	-12	-604	-535
-13	-426	-316	15	330	257						
• 3	-3										
0	-453	-479	3	371	342	4	790	831	8	-179	-150
-1	-419	-452	-2	-288	-285	-5	220	292	-6	220	298
-7	304	403									
• 3	4										
0	-261	-178	1	1483	1710	2	1236	1399	3	-659	-623
5	-1037	-837	6	-934	-738	7	522	358	9	-227	-222
-1	446	313	-2	-350	-228	-3	103	143	-4	-1497	-1824
-5	-1394	-1694	-6	652	640	-7	-151	-142	-8	1003	948
-9	1016	955	10	-266	-295	-11	206	239	-12	288	246
• 3	-4										
0	474	469	2	268	266	4	-1243	-1259	6	460	377
8	433	411	-3	268	311	-4	172	224	-6	-199	-234
• 3	5										
2	-288	-266	3	-323	-290	4	-446	-446	5	-185	-150
8	446	562	9	185	230	-1	295	253	-2	378	343
-3	687	709	-4	412	379	-5	-398	-362	-7	-467	-472
-10	-634	-813									
• 3	-5										
2	316	368	3	309	329	4	-295	-318	5	-240	-289
• 3	6										
0	-350	-359	1	-927	-844	2	446	369	3	879	765
5	357	419	7	-254	-213	8	172	178	-1	-376	-333
-2	-233	-161	-3	-953	-831	-4	-165	-108	-5	1367	1230
-6	433	321	-7	824	516	-8	316	254	-9	-886	-879
• 3	7										
0	261	225	1	185	189	2	185	210	3	172	194
7	-206	-314	-1	185	144	-3	-364	-302	-4	-584	-568
-6	-185	-288	-7	254	214	-8	316	333	-9	-309	-340
-11	227	374									
• 3	8										
0	172	165	2	-536	-529	4	172	139	-2	-172	-117
-3	398	327	-4	714	614	-7	-343	-380	-8	-749	-790
• 3	9										
-1	-137	-148	-2	-199	-215	-3	-137	-145	-4	199	181
-5	157	164	-6	275	313	-8	-233	-330			
• 4	0										
0	-460	-438	1	-247	-144	2	-652	-541	3	-179	-98
4	-742	-602	5	-268	-209	6	1250	1284	7	460	489
9	158	149	10	-446	-461	11	-191	-250	-1	-357	-285

Table 4 (*cont.*)

-3	247	241	-4	371	576	-5	790	617	-6	783	615
-7	-865	-373	-8	-389	-382	-9	-323	-351	-10	-316	-402
* 4	1										
1	506	299	2	879	548	3	543	375	4	948	595
5	515	369	6	-989	-678	7	-302	-257	9	-350	-327
11	-165	-194	-1	-151	-148	-2	-563	-447	-3	-961	-772
-4	-694	-574	-5	-625	-501	-6	227	231	-7	172	435
-8	494	491	-9	501	662						
* 4	-1										
-2	694	427	-3	508	317	-5	-419	-318	-6	-838	-628
-7	-419	-302									
* 4	2										
0	316	572	1	391	207	2	639	523	3	-130	-358
5	-852	-1054	6	-975	-1286	7	494	567	8	275	348
9	172	267	10	286	509	-1	886	693	-2	570	369
-3	666	466	-4	-639	-468	-5	-474	-373	-6	-996	-832
-7	-549	-596									
* 4	3										
0	-405	-404	1	-419	-491	2	-405	-476	4	-124	-138
5	378	443	-1	-453	-423	-2	-309	-269	-3	460	408
-4	468	403	-5	831	785	-6	824	665	-9	-742	-719
-10	-385	-812	-11	-391	-346	-12	-577	-585	-14	282	293
-15	453	516									
* 4	-3										
0	-185	-204	1	-227	-261	3	-179	-205	4	-213	-218
5	330	330	6	302	378	7	165	256	8	185	236
9	-89	-196	-4	179	273	-5	240	371			
* 4	4										
1	288	114	2	323	246	3	906	887	4	282	324
-1	-591	-527	-2	-419	-399	-3	-1147	-1183	-4	-302	-234
-5	233	132	-7	659	589	-8	549	518	-9	700	605
-11	-364	-219	-13	-323	-309	-14	-350	-393			
* 4	-4										
2	-158	-190	5	-336	-318	6	-233	-204	-6	-309	-520
* 5	0										
1	-330	-255	2	-488	-379	3	-268	-325	4	-323	-307
7	151	195	9	206	275	-2	790	809	-3	467	406
-4	1071	923	-5	-192	-169	-6	-1511	-1329			
* 5	1										
0	-350	-306	2	254	162	3	309	295	4	254	304
6	288	408	9	-213	-267	-1	-488	-467	-2	-488	-519
-3	-385	-324	-5	426	386	-7	474	516	-8	350	458
* 5	2										
0	364	485	1	549	434	2	316	283	6	-185	-271
7	-261	-374	-1	755	680	-4	-1291	-1133	-5	-1566	-1670
-6	563	732									
* 5	3										
0	-172	-139	1	-336	-324	2	-371	-452	3	-295	-361
-1	117	142	-2	549	497	-3	666	630	-4	652	540
-5	-124	-161	-6	179	171	-8	-536	-493	-9	-185	-178
-10	-755	-808	-11	-254	-255	-12	405	364	-13	316	364
-14	433	490									
* 5	-3										
2	-137	-185	3	-151	-220	-2	144	197	-3	96	257
* 5	4										
0	-266	-228	1	-282	-187	2	288	176	3	288	250
-1	-721	-609	-2	-515	-319	-3	-941	-845	-4	-666	-535
-5	1703	1651	-6	783	604	-7	350	253	-8	584	470
-9	-755	-649	-10	-286	-175	-12	-330	-377	-13	-233	-262
* 6	0										
8	597	592	1	-172	-144	2	-165	-104	5	-439	-423
6	-357	-307	-1	625	619	-2	-254	-126	-3	309	194
-4	-501	-395	-5	-364	-332	-6	350	398			
* 6	1										
0	-309	-389	1	-385	-317	2	-240	-205	5	240	375
6	172	260	-3	254	162	-4	309	225	-5	350	268
* 6	2										
0	206	216	1	515	736	2	199	215	3	192	263
5	295	267	6	-213	-253	7	-343	-458	-1	-714	-822
-2	-206	-203	-3	-838	-841	-4	-206	-245	-5	453	867
* 6	3										
0	268	281	3	-227	-301	4	-213	-326	-1	385	397
-2	467	410	-3	233	199	-5	268	171	-6	-385	-365
-7	-604	-502	-8	-508	-492	-9	-439	-427	-10	295	219
-11	385	346	-12	453	455	-13	357	364			
* 6	4										
0	-898	-1017	1	-467	-481	-2	-508	-369	-3	776	584
-4	1579	1408	-5	-591	-374	-7	302	242	-8	-419	-289
-10	-268	-273	-11	-309	-356						
* 7	0										
0	110	163	1	247	234	-2	117	110	-4	-110	-158
-5	-103	-274									
* 7	1										
2	-206	-308	3	-206	-165	-2	199	179	-3	199	266
* 7	2										
0	-302	-311	1	-206	-197	2	220	242	3	137	189
-2	-124	-303									
* 7	3										
0	240	276	1	261	367	-1	213	244	-4	-228	-215
-5	-357	-365	-6	-343	-320	-7	-330	-323	-9	199	230
-10	330	402	-11	378	356						
* 7	4										
0	385	475	1	-323	-321	2	-488	-561	-2	282	283
-3	350	269	-5	350	312	-8	-261	-234	-9	-330	-331
-10	-220	-260									
* 8	4										
0	206	238	-1	240	207	-2	262	245	-3	426	408
-4	-364	-200	-5	-357	-229	-6	-144	-19			

causing a dihedral angle of about 3° between the two molecular planes. In this way the closest C···Cl distance is 3·61 Å, to C(7), whereas the C···O interaction distances are of the order of 3·3 Å, as shown in Fig. 2. There are no close approaches between molecules in adjacent stacks apart from the OH···O=C hydrogen bond of length 2·70 Å between the phenol molecule in one stack and a quinone molecule approximately coplanar with it in an adjacent stack. Because of the 1:1 stoichiometry of the complex only one of the two C=O groups in each quinone molecule is hydrogen-bonded in this way and the centrosymmetric repetition of this hydrogen bond causes the stacks of molecules to be linked in pairs in the z direction. The pairs of stacks are then held together in the x and z directions by van der Waals forces.

The dimensions of the two component molecules in QmonoPCP are shown in Fig. 4. The standard deviations of the bond distances are all approximately 0·02 Å, apart from that of the C–Cl bond, which is 0·013 Å. The bond angles have standard deviations of about 1·5°. The dimensions appear normal within the rather wide limits of accuracy, and, as in the case of the 2:1 complex of *p*-chlorophenol, the irregularity of bond angles at the point of attachment of the OH group to the ring of the phenol molecule can be explained in terms of repulsion between the OH group and the C(5)–H group adjacent to it in the ring. In both the 2:1 and 1:1 complexes there is a tendency for the pair of ring C–C bonds parallel to the C–Cl and C–OH bonds to be larger than the other four bonds. The difference is not statistically significant in either structure, but its appearance in both structures suggests that it is probably real. The lengths of these two types of bonds, averaged between the two structures, are 1·41 and 1·38 Å. The corresponding bonds in the quinone molecules (which are formally C=C and C–C respectively) have lengths 1·34 and 1·46 Å when averaged between the two structures. The other bond lengths, similarly averaged, are C–Cl 1·76, C–OH 1·38, C=O 1·21 Å, and these all compare reasonably well with expected dimensions (*cf.* 1·322, 1·477, 1·222 Å found for the lengths of C=C, C–C and C=O in *p*-benzoquinone; Trotter, 1960).

It is interesting to compare the crystal structures of the 1:1 phenol–quinone complexes with those of the 2:1 complexes already reported (Shipley & Wallwork, 1967). The 2:1 stoichiometry permits each carbonyl oxygen atom to be hydrogen bonded to a phenolic OH group, but this is achieved at the expense of restricting the intermolecular charge-transfer interaction to isolated groups of three molecules (one quinone sandwiched between two phenol molecules). The combined effect of both types of interaction is to form bands of molecules one unit cell wide (the limit of the charge-transfer interaction), which extend indefinitely in the hydrogen-bonded direction. The 1:1 complexes, on the other hand, have infinite stacks of charge-transfer-linked molecules with the stacks hydrogen-bonded into pairs so that, again, infinite bands of molecules are formed, which are one unit cell wide (the limit of the hydrogen-bond interaction). The fact that the 2:1 and 1:1 complexes are formed under similar conditions, the product depending on the composition of the solution, and appear to be about equally stable, indicates that the charge-transfer forces and the hydrogen bonding are roughly similar in energy. No 1:1 complex has been reported between unsubstituted phenol and *p*-benzoquinone, so presumably the increased van der Waals forces due to the halogen atoms are necessary to stabilize the structure of the 1:1 complex. In quin-

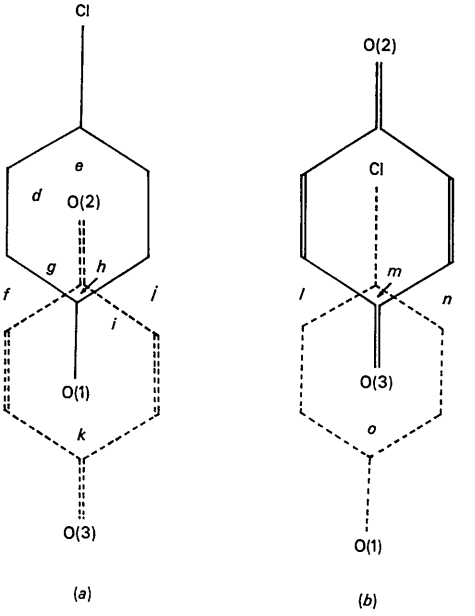

Fig. 3. Projections of (*a*) *p*-chlorophenol on to the *p*-benzoquinone molecule below (smaller y coordinate) and (*b*) *p*-benzoquinone on to the *p*-chlorophenol below, showing the mode of overlap in relation to the close approaches designated as in Fig. 2.

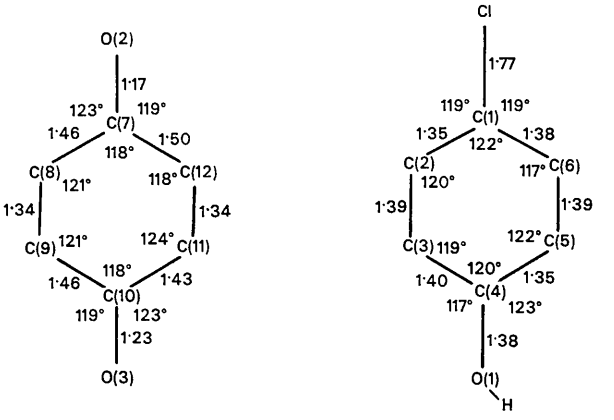

Fig. 4. Molecular dimensions (Å) of *p*-benzoquinone and *p*-chlorophenol in QmonoPCP.

hydrone, both charge-transfer interaction and hydrogen bonding are optimized in the 1:1 structure, so it is not surprising that there is no 2:1 complex in this case.

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Short Communications

Contributions intended for publication under this heading should be expressly so marked; they should not exceed about 1000 *words; they should be forwarded in the usual way to the appropriate Co-editor; they will be published as speedily as possible. Publication will be quicker if the contributions are without illustrations.*

Acta Cryst. (1967). **22**, 601

A comparison of analytical and numerical methods for the evaluation of the absorption correction in crystal structure analysis.*

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A comparison is made between analytical evaluation of the absorption correction using Howells polyhedra and numerical calculation with the Gaussian method. It is shown that convergence can be reached with the numerical method, provided a sufficiently fine grid is chosen. When absorption is large (*i.e.* > 70–80 %) the analytical method is to be preferred as it produces the required accuracy in a much smaller amount of computer time.

An analytical method of evaluating the absorption correction in crystal structure analysis has been described recently (de Meulenaer & Tompa, 1965). The only completely general method available previously was introduced by Busing & Levy (1957) and is based on a numerical evaluation of $\int \exp(-\mu L)dV$ (see also Coppens, Leiserowitz & Rabinovich, 1965). The accuracy of this method depends evidently on the formula and the number of points used. Busing & Levy claim specifically a precision of 0·2 % for transmissions between 50 and 62 % using Gauss's formula and an $8\times8\times8$ grid and it is intuitively clear and easy to verify that for a constant grid the accuracy decreases with increasing absorption coefficient μ. Clearly, any desired accuracy can be obtained by using finer grids, but it is the purpose of this note to point out that great care must be taken to verify that the number of points used is sufficient for the desired accuracy, and that the computation effort required may become prohibitive.

We have recomputed, using the analytical method, the transmission for some of the reflexions of a needle-shaped crystal of $YFeO_3$ ($\mu = 878{\cdot}8$ cm^{-1} for Cu $K\alpha$ radiation) investigated recently (Coppens & Eibschütz, 1965) and find values differing from those obtained by the Busing & Levy method using a $12\times12\times32$ grid. We have, therefore, repeated the computation on a finer grid and have obtained values which converge towards those of the analytical method, as shown in Table 1.

Table 1. *Transmission of some reflexions of a crystal of* $YFeO_3$ (*Coppens & Eibschütz*, 1965) *computed by Gaussian integration on the grids indicated and by the analytical method of de Meulenaer & Tompa* (1965)

hkl	$12\times12\times32$	$24\times24\times32$	$32\times32\times32$	Analytical
025	0·0463	0·0378	0·0387	0·0390
$0\bar{2}5$	0·0463	0·0378	0·0387	0·0390
125	0·0375	0·0335	0·0339	0·0340
$1\bar{2}5$	0·0377	0·0332	0·0337	0·0339
225	0·0613	0·0583		0·0587
$2\bar{2}5$	0·0541	0·0509		0·0514
325	0·0695	0·0681		0·0684

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