

dimensional Fourier synthesis of  $F_{obs} - F_{calc}$  showed no peaks as high as 2 electrons/ $\text{\AA}^3$ .

The perxenate ion is centric by the space-group symmetry. The three independent Xe-O distances are: Xe-O(1),  $1.85 \pm 0.02 \text{ \AA}$ ; Xe-O(2),  $1.86 \pm 0.02 \text{ \AA}$ ; Xe-O(3),  $1.81 \pm 0.02 \text{ \AA}$ . The deviations from the average value  $1.84 \text{ \AA}$  are not significant. Our data concerning thermal motion are incomplete, but we estimate that the correction for thermal motion would increase this distance less than  $0.01 \text{ \AA}$ . The bond angles O-Xe-O which would be  $90^\circ$  for regular octahedral shape range from  $87.1^\circ$  to  $92.9^\circ$  with standard deviations of about  $1^\circ$ . We do not consider these deviations from  $90^\circ$  to be significant. These results are in accord with those from the octahydrate (3) in which the angles were found closer to  $90^\circ$  and the average bond distance was reported as  $1.875 \pm 0.021 \text{ \AA}$ , with somewhat more scatter from the average than in the hexahydrate.

Each Na(1) has six oxygen neighbors belonging to perxenate ions at an average distance  $2.46 \text{ \AA}$ . Each Na(2) has six oxygen neighbors (water molecules) at an average distance  $2.44 \text{ \AA}$ . In each case the six neighbors are at the corners of an irregular octahedron.

The overall structure is quite different from that of the octahydrate. It consists of layers perpendicular to the  $a$  axis. Layers containing the perxenate and half of the sodium alternate with layers containing the rest of the sodium and all of the water. The six independent hydrogen atoms are assigned to hydrogen bonds which all have reasonable distances and angles. Five of these bonds, between water and oxygen of perxenate, hold the layers to each other (6).

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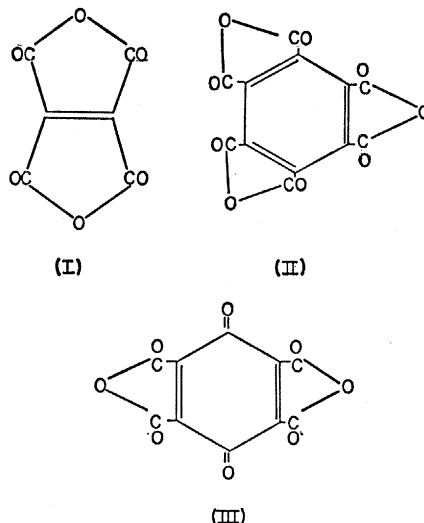
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6. This work was done in part under the auspices of the U. S. Atomic Energy Commission.

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### 1,4-Benzoquinone Tetracarboxylic Acid Dianhydride, $C_{10}O_8$ : A Strong Acceptor

**Abstract.** *This exceptionally reactive compound 1,4-benzoquinone tetracarboxylic acid dianhydride is one of the strongest  $\pi$ -acids yet described.*

Small, electronegative substituents attached to planar  $\pi$ -electron systems enhance the acceptor properties of these systems with regard to forming  $\pi$ - $\pi$  molecular complexes. Larger substituents constrained in a coplanar manner also afford stable complexes with  $\pi$ -donors and the  $o$ -dicarboxylic acid anhydride group is an example of this as seen in the acceptors maleic anhydride (1) and pyromellitic dianhydride (2). Several families of planar carbon oxides may therefore be visualized, and they should exhibit interesting acceptor properties. Indeed, colored complexes of II with anthracene, naphthalene, and hexamethylbenzene have been reported (3).



The quinone III has been prepared by oxidation of the quinol (4) with nitrogen oxides. Because of its unusual solution properties, the resulting pale-brown powder could not be freed from a trace of molecular sieve 4A used in the preparation and it was character-

Table 1. Charge-transfer absorptions of complexes of  $C_{10}O_8$  with methylbenzenes in dichloromethane at  $19^\circ\text{C}$ .

Donor	$\nu_{\max}$ (ev)	Half-life (min)
Benzene	2.98*	$10^3$
Toluene	2.57*	12
<i>o</i> -Xylene	2.53	60
<i>m</i> -Xylene	2.41	6
<i>p</i> -Xylene	2.30	3
Mesitylene	2.34	0.3
Pseudocumene	2.10	0.3
Durene	Blue	0.2
Pentamethylbenzene	Blue-green	0.1
Hexamethylbenzene	Green	0.05

\* Homogeneous.

ized as a deep red (1:1) benzene complex.

Studies of the powder and of the complex show that the oxide is insoluble in most solvents (ether, petroleum ether, carbon tetrachloride, dichloromethane, carbon disulphide). Towards those in which it does dissolve (acetone, ethyl acetate, tetrahydrofuran, ethanol, water) it exhibits remarkable chemical reactivity, for it cannot be isolated from them without decomposition. It dissolves in the methylbenzenes giving colored solutions of the charge-transfer complexes—benzene, orange; toluene, red; *m*-xylene, magenta; mesitylene, violet. The colors are associated with the single, broad bands shown in Table 1 and the oxide is thus one of the strongest  $\pi$ -electron acceptors so far described. Approximate half-lives of the unstable complexes are also shown and even the benzene adduct has to be stored at low temperatures in the absence of light.

The oxide is stable in dry air, although moist air turns it blue within 10 minutes. It can be heated to  $140^\circ\text{C}$  without decomposition, although above this temperature it darkens and loses its complexing ability (5).

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