

surface, so that the adjacent soil will freeze and thaw in a non-horizontal plane, or non-uniform vertical sorting must exist.

4) The amount of moisture at the freezing plane. In materials deposited by man, the amount of sorting is directly proportional to the moisture content of the soil (7). The actual presence of a layer of water between the ice front and the particle was proposed by Taber (8) but still remains to be demonstrated. This water would have to be continuously replenished as the ice advanced, moving the particle in front of it. If the water layer froze faster than it was replenished, the particles would be trapped by the ice. It is reasonable to assume that the layer of water would be more easily and rapidly replenished in the presence of small, rather than large, particles. This would account for the fact that coarse particles move only when the rate of freezing is low.

The results of these experiments may be important in determining the susceptibility of materials to frost over long periods of time. Evidently, the chances of frost-heaving or formation of ice lenses will increase in proportion to the concentration of fine particles. On the other hand, it may be predicted that the soil adjacent to a retaining wall will have a tendency to become less susceptible to the formation of ice lenses as fine particles move away from it.

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Sodium Perxenate Hexahydrate

Abstract. *Sodium perxenate hexahydrate ($\text{Na}_4\text{XeO}_6 \cdot 6\text{H}_2\text{O}$) has been identified from study of its crystal structure by x-ray diffraction. The perxenate ion (XeO_6^{4-}) has the shape of a regular octahedron with the Xe-O bond distance 1.84 Å.*

Some hydrated sodium salts of xenon in the +8 oxidation state were produced by Malm, Holt, and Bane (1) by hydrolysis of XeF_6 in the presence of sodium hydroxide. Crystal data were reported (2) for three phases produced in this way. By determination of the crystal structure one of these phases is $\text{Na}_4\text{XeO}_6 \cdot 8\text{H}_2\text{O}$, containing perxenate ion (XeO_6^{4-}) in the shape of a regular octahedron (3).

We have produced another salt; this is characterized as $\text{Na}_4\text{XeO}_6 \cdot 6\text{H}_2\text{O}$ by determination of the crystal structure. We find the same shape for the perxenate ion with dimensions close to those reported in the work on the octahydrate (3). This hexahydrate is not one of the phases reported by Siegel and Gebert (2).

The crystals of $\text{Na}_4\text{XeO}_6 \cdot 6\text{H}_2\text{O}$ were obtained from the reaction of pure xenic acid (aqueous XeO_3) (4) and sodium hydroxide. When 0.100 ml of 0.208M XeO_3 (aq) and 0.060 ml of 6M NaOH were mixed the resulting solution was pale yellow. After one day in a refrigerator at 5°C, crystals grew as very thin fragile sheets. The production of crystals from the reaction is much more rapid at elevated temperatures, about 60°C, but at this higher temperature the product is the octahydrate. It was identified by its cell dimensions which we measured as $a = 11.864$, $b = 10.426$, and $c = 10.358$ Å (each ± 0.005 Å), in agreement with the previous work (2, 3).

The hexahydrate is much less stable than the octahydrate. The x-ray study was made of crystals sealed in thin-walled glass capillaries containing some of the mother liquor. Most crystals survived only a few hours in the capillaries, and several preparations were necessary before adequate data were obtained. Photography of one crystal by the Weissenberg technique gave some preliminary information. Intensity data with $\text{MoK}\alpha$ radiation were gathered by direct counting with a scintillation counter for about 1250 reflections (including about 250 absent because of the space group) on one

or more of 10 different crystals. At higher angles many of the reflections with mixed (even and odd) indices were too weak to be observed reliably. The determination of the structure was based on 491 reflections from one or another of 5 different crystals, including 14 reflections which were assigned zero intensity. These crystals were 0.3 mm or less in the largest dimension, with thicknesses too thin to be measured with our microscope. Experiments by a technique described elsewhere (5) showed that absorption reduced some intensities about 20 percent, but was negligible for most of the data.

The crystals are orthorhombic, space group $Pbca$, with $a = 18.44 \pm 0.01$, $b = 10.103 \pm 0.007$, and $c = 5.873 \pm 0.005$ Å. The density is calculated as 2.59 g/ml with four molecules per unit cell. The crystals were observed to sink in ethylene bromide (density 2.17 g/ml). Xenon atoms are on centers of inversion at the origin and face centers. All other atoms are in general eight-fold sets of positions.

All atoms (except hydrogen, which is not detected with these data) were recognized in the Patterson function. The main features of the structure never changed during the refinement process which was circuitous because change of sign of several of the coordinates has little effect on most of the intensities and because strong correlation among some of the parameters results from pseudosymmetry of the structure. Refinement by least squares eventually reduced

$$R = \sum |F_{obs}| - |F_{calc}| / \sum |F_{obs}|$$

to 0.072 with the atomic parameters listed in Table 1. Standard deviations of coordinates correspond to 0.01 Å for sodium and 0.02 Å for oxygen. A three-

Table 1. Atomic coordinates and thermal parameters in $\text{Na}_4\text{XeO}_6 \cdot 6\text{H}_2\text{O}$.

Atom	x	y	z	B, Å ²
Xe	0	0	0	(0.85±0.04)*
Na(1)	0.009	0.164	0.492	1.4±0.2
Na(2)	.252	.155	.539	1.7±0.2
O(1)	.060	.005	.252	1.2±0.2
O(2)	.064	.115	.854	1.8±0.4
O(3)	.951	.136	.127	1.0±0.4
O(W1)	.169	.200	.242	1.6±0.3
O(W2)	.339	.183	.836	1.5±0.3
O(W3)	.192	.518	.242	2.2±0.4

*Isotropic B equivalent to average of anisotropic temperature factor.

dimensional Fourier synthesis of $F_{obs} - F_{calc}$ showed no peaks as high as 2 electrons/Å³.

The perxenate ion is centric by the space-group symmetry. The three independent Xe-O distances are: Xe-O(1), 1.85 ± 0.02 Å; Xe-O(2), 1.86 ± 0.02 Å; Xe-O(3), 1.81 ± 0.02 Å. The deviations from the average value 1.84 Å 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 Å. The bond angles O-Xe-O which would be 90° for regular octahedral shape range from 87.1° to 92.9° with standard deviations of about 1°. We do not consider these deviations from 90° to be significant. These results are in accord with those from the octahydrate (3) in which the angles were found closer to 90° and the average bond distance was reported as 1.875 ± 0.021 Å, 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 Å. Each Na(2) has six oxygen neighbors (water molecules) at an average distance 2.44 Å. 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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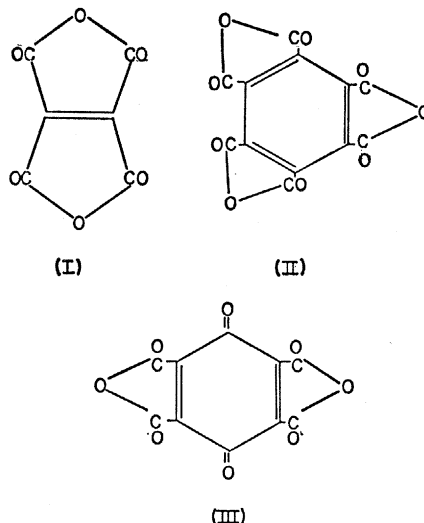
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1,4-Benzoquinone Tetracarboxylic Acid Dianhydride, C₁₀O₈: A Strong Acceptor

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

Small, electronegative substituents attached to planar π -electron systems enhance the acceptor properties of these systems with regard to forming π - π molecular complexes. Larger substituents constrained in a coplanar manner also afford stable complexes with π -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 C₁₀O₈ with methylbenzenes in dichloromethane at 19°C.

Donor	ν_{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 π -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°C without decomposition, although above this temperature it darkens and loses its complexing ability (5).

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