case, to the spin density measurements by polarized neutron diffraction experiments (Deeth, Figgis, Forsyth, Kucharski & Reynolds, 1989; Fender, Figgis & Forsyth, 1986*b*; Fender, Figgis, Forsyth, Reynolds & Stevens, 1986). The ferrous Tutton salt structure has been determined at room temperature by X-ray film methods (Montgomery, Chastain, Natt, Witkowska & Lingafelter, 1967).

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β-Hydroquinone Xenon Clathrate

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Abstract. $3C_6H_4(OH)_2.xXe$ ($x = 0{\cdot}866$), $M_r = 444{\cdot}03$, rhombohedral, $R\bar{3}$ (obverse setting), $a = 16{\cdot}610$ (3), $c = 5{\cdot}524$ (1) Å, $V = 1319{\cdot}8$ (4) Å^3, $Z = 3$, $D_m = 1{\cdot}67$ (1), $D_x = 1{\cdot}68$ g cm^{-3} (for $x = 0{\cdot}866$), graphite-monochromatized Mo $K\alpha$ radiation, $\lambda = 0{\cdot}71069$ Å, $\mu = 17{\cdot}88$ cm^{-1}, $F(000) = 660{\cdot}79$, $T = 298$ K, $R = 0{\cdot}0242$, $wR = 0{\cdot}0253$ for 594 unique reflections and 54 parameters. The encaged xenon atom occupies a near-spherical cavity (*ca* 4·8 Å diameter), formed by six interlocking C_6H_4 moieties and two hydrogen-bonded $[OH]_6$ hexagonal rings above and below the plane of the guest atom.

Experimental. The complex was isolated from an aqueous solution of hydroquinone with a modification of the procedure described by Powell (1950). The salient change is an increase in the initial pressure of xenon from 14 to 21 atm (1 atm = $1{\cdot}01 \times 10^5$ Pa). The density of the crystals was determined by flotation in $CCl_4/CHBr_3$ solution. Crystal chosen was ground to a sphere 0·3 mm in diameter with a Nonius crystal grinder and was sealed in a Lindemann capillary. Unit-cell parameters determined from the least-squares refinement of positional angles for 15 strong independent reflections in the range $21{\cdot}8° < 2\theta < 31{\cdot}2°$ on a Nicolet $P2_1$ diffractometer at 298 K with graphite-monochromated Mo $K\alpha$ radiation ($\lambda = 0{\cdot}71069$ Å). Rhombohedral, space group $R\bar{3}$ (No. 148) from systematic absences: $-h+k+l \neq 3n$. Intensities of 4974 reflections with $2\theta \leq 55°$, $-15 \leq h \leq 12$, $0 \leq k \leq 15$, $-7 \leq l \leq 7$, were measured with a θ–2θ scan technique. Range of scan rates used was 5·86 to 29·30° min^{-1} in 2θ. The total background time to scan time is 1:1. The intensities of two standard reflections ($\bar{3}\bar{8}\bar{1}$: 1·59%, and $\bar{1}\bar{1}31$; 1·48%) were monitored every 48 reflections and showed no sign of crystal decomposition or instrument instability. The data were averaged to give 594 independent reflections; $R_{\rm int} = 0{\cdot}0157$. Reflections with $3\sigma I \geq I \geq -3\sigma I$ were treated by the method of French & Wilson (1978). Corrections were made for Lorentz–polarization effects but not for absorption. The coordinates of the xenon atom were found from a three-dimensional Patterson synthesis, and a series of full-matrix least-squares

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Table 1. *Positional parameters* ($\times 10^4$) *and* U_{eq} ($\times 10^4$) *for β-hydroquinone xenon clathrate, with e.s.d.'s in parentheses*

$U_{eq} = \frac{1}{3}(U_{11} + U_{22} + U_{33} + 2\cos\gamma U_{12})$.

	Wyckoff position	x	y	z	U_{eq}(Å^2)
Xe*	3(*b*)	0	0	5000	475
O	18(*f*)	986 (1)	−893 (1)	63 (2)	364
C(1)	18(*f*)	1343 (1)	−1259 (1)	1716 (3)	288
C(2)	18(*f*)	1109 (1)	−2178 (1)	1418 (3)	316
C(3)	18(*f*)	1907 (1)	−743 (1)	3621 (3)	319
H(1)	18(*f*)	1216 (22)	−253 (28)	302 (55)	870†
H(2)	18(*f*)	711 (15)	−2543 (14)	66 (38)	455†
H(3)	18(*f*)	2074 (12)	−91 (14)	3855 (35)	361†

* Positional coordinates of Xe fixed.
† Isotropic temperature factor.

Table 2. *Bond lengths* (Å) *and bond angles* (°) *for β-hydroquinone xenon clathrate with e.s.d.'s in parentheses*

(*a*) β-Hydroquinone cage

C(1)—C(2)	1·384 (3)	C(2)—C(1)—C(3)	120·2 (2)
C(1)—C(3)	1·384 (2)	C(2)—C(1)—O	117·4 (1)
C(1)—O	1·384 (3)	C(3)—C(1)—O	122·4 (2)
C(2)—C(3)^{i}	1·384 (3)	C(1)—C(2)—H(2)	120 (2)
C(3)—C(2)^{i}	1·384 (3)	C(1)—C(3)—H(3)	121 (1)
C(2)—H(2)	0·98 (2)	C(1)—O—H(1)	113 (2)
C(3)—H(3)	0·98 (2)	C(2)^{i}—C(3)—C(1)	119·6 (2)
O—H(1)	0·94 (4)	C(2)^{i}—C(3)—H(3)	121 (1)
		C(3)^{i}—C(2)—C(1)	120·1 (2)
		C(3)^{i}—C(2)—H(2)	120 (2)

(*b*) Hydrogen-bonding network and guest–host interactions

O···O^{ii}	2·705 (2)	O^{ii}···O···O^{iii}	119·9 (1)
O···O^{iii}	2·705 (2)	O···H(1)^{ii}	1·80 (3)
Xe···O	3·840 (1)	Xe···O^{ii}	3·893 (1)
Xe···H(1)	3·44 (3)	Xe···H(1)^{ii}	3·70 (4)
Xe···C(1)	4·160 (2)	Xe···C(3)	4·005 (2)

Symmetry operations on atom at x, y, z: (i) $\frac{1}{3}-x, \frac{2}{3}-y, \frac{2}{3}-z$; (ii) $y, -x+y, -z$; (iii) $x-y, x, -z$.

refinements followed by a three-dimensional electron-density synthesis revealed all the remaining atoms. Anisotropic full-matrix least-squares refinement minimized $\sum w(|F_o| - |F_c|)^2$, $w = [\sigma^2(F) + 0{\cdot}00025F^2]^{-1}$. Scale, positional and anisotropic temperature factors for the non-hydrogen atoms, hydrogen-atom positional and isotropic temperature factors and xenon site occupancy were refined, 54 parameters. Final $R = 0{\cdot}0242$, $wR = 0{\cdot}0253$ and $S = 1{\cdot}3714$. An extinction correction given by $F^* = F(1-0{\cdot}17 \times 10^{-5}F^2/\sin\theta)$ was applied. Refinement was terminated when $(\Delta/\sigma)_{max} = 0{\cdot}003$. Final difference map revealed electron density max. 0·16 e Å^{-3}, 0·59 Å from Xe, min. −0·17 e Å^{-3}. Atomic scattering factors were taken from Cromer & Mann (1968) and corrections for anomalous dispersion for Xe from Cromer & Liberman (1970). Calculations employed *XTAL* (Stewart & Hall, 1983), *SHELX*76 (Sheldrick, 1976), *MOLGEOM* (Stephens, 1973) and *SNOOPI* (Davies, 1983) program systems run on a VAX 8650 mainframe computer. Atomic positional parameters and U_{eq} for all atoms are given in Table 1, bond distances and angles are given in Table 2.* Fig. 1 shows a view of the structure along [001].

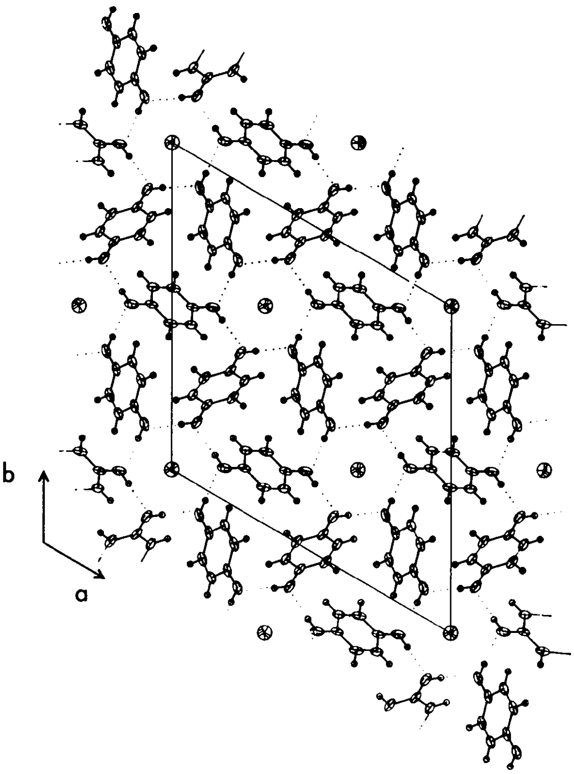

Fig. 1. View of the unit-cell packing showing the hydrogen-bonding network. Projection of the structure is along [001].

Related literature. The β-hydroquinone xenon clathrate is isostructural with the β-hydroquinone hydrogen sulfide clathrate (Mak, Tse, Tse, Lee & Chong, 1976; Ho & Mak, 1982), with the guest molecule showing no interaction with the host lattice. Related structures are that of the methanol clathrate (Mak, 1982), the hydrogen chloride clathrate (Boeyens & Pretorius, 1977) and the α form of quinol (Wallwork & Powell, 1980).

* Lists of structure factors, anisotropic temperature factors and least-squares planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51675 (6 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of Tetraammonium Hexahydrogenhexamolybdocuprate(II) Tetrahydrate

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Abstract. $(NH_4)_4[H_6CuMo_6O_{24}].4H_2O$, $M_r = 1173·4$, monoclinic, $P2_1/c$, $a = 11·421$ (1), $b = 11·126$ (2), $c = 11·919$ (2) Å, $\beta = 107·22$ (1)°, $V = 1446·6$ (4) Å^3, $Z = 2$, $D_m = 2·73$, $D_x = 2·69$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 3·32$ mm^{-1}, $F(000) = 1126$, $T = 298$ K, $R = 0·060$ for 1734 independent reflections. The discrete polyanion $[H_6CuMo_6O_{24}]^{4-}$ is isostructural with $[H_6CrMo_6O_{24}]^{3-}$ [Perloff (1970). *Inorg. Chem.* **9**, 2228–2239]. Mo—O distances range from 1·70 to 2·25 Å, whereas Cu—O distances range from 2·02 to 2·12 Å, exhibiting no typical tetragonal distortion from the Jahn–Teller effect.

Experimental. Preparation: Rosenheim (1916); faintly blue rhombic crystals from a mixed aqueous solution of $CuSO_4.5H_2O$ and $(NH_4)_6Mo_7O_{24}.4H_2O$ in the ratio 1:6, adjusted pH 4–5; D_m by flotation in dibromoethane and diiodomethane; crystal 0·10 × 0·10 × 0·02 mm. AFC-5R diffractometer; cell parameters from 22 indices in θ range 12·0–14·8°; data collected by θ–2θ scan mode up to 55° in 2θ; $-14 \le h \le 14$, $0 \le k \le 14$ and $0 \le l \le 15$; 3516 reflections measured; three standard reflections, no decay; empirical absorption correction

Table 1. *Positional and equivalent isotropic thermal parameters*

Fractional coordinates are multiplied by 10^4 for the Cu and Mo atoms, by 10^3 for the remaining atoms.

$$B_{eq} = \tfrac{8}{3}\pi^2\textstyle\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}(Å^2)
Mo(1)	1515 (1)	2413 (1)	1333 (1)	1·9 (1)
Mo(2)	3042 (1)	0058 (1)	0755 (1)	2·1 (1)
Mo(3)	1514 (1)	−2361 (1)	−0523 (1)	1·8 (1)
Cu	0	0	0	1·6 (1)
O(1)	141 (1)	116 (1)	−015 (1)	1·9 (4)
O(2)	143 (1)	−104 (1)	089 (1)	1·9 (4)
O(3)	−002 (1)	−112 (1)	−134 (1)	1·8 (4)
O(4)	262 (1)	108 (1)	192 (1)	2·1 (4)
O(5)	256 (1)	−100 (1)	−058 (1)	1·8 (4)
O(6)	002 (1)	−306 (1)	−034 (1)	1·8 (4)
O(7)	153 (1)	296 (1)	267 (1)	3·6 (6)
O(8)	244 (1)	339 (1)	090 (1)	3·0 (5)
O(9)	397 (1)	095 (1)	022 (1)	3·4 (6)
O(10)	405 (1)	−080 (1)	178 (1)	4·1 (6)
O(11)	246 (1)	−328 (1)	049 (1)	2·9 (1)
O(12)	154 (1)	−297 (1)	−183 (1)	2·7 (5)
O(13)	013 (1)	044 (1)	355 (1)	4·9 (7)
O(14)*	518 (2)	173 (2)	543 (3)	4·0 (10)
O(15)*	500 (2)	196 (2)	293 (3)	4·6 (11)
N(1)	311 (1)	474 (1)	−088 (1)	3·4 (7)
N(2)	340 (1)	−472 (1)	260 (1)	2·8 (6)

* Occupancies of these atoms are 0·5.

Table 2. *Selected interatomic distances* (Å)

Mo(1)—O(1)	2·23 (1)	Mo(2)—O(1)	2·22 (1)	Mo(3)—O(2)	2·25 (1)
Mo(1)—O(3)	2·24 (1)	Mo(2)—O(2)	2·25 (1)	Mo(3)—O(3)	2·22 (1)
Mo(1)—O(4)	1·94 (1)	Mo(2)—O(4)	1·96 (1)	Mo(3)—O(5)	1·95 (1)
Mo(1)—O(6)	1·94 (1)	Mo(2)—O(5)	1·92 (2)	Mo(3)—O(6)	1·94 (1)
Mo(1)—O(7)	1·70 (1)	Mo(2)—O(9)	1·70 (1)	Mo(3)—O(11)	1·70 (1)
Mo(1)—O(8)	1·70 (1)	Mo(2)—O(10)	1·70 (1)	Mo(3)—O(12)	1·70 (1)
Cu—O(1)	2·12 (1)	Cu—O(2)	2·03 (1)	Cu—O(3)	2·02 (1)

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