lated positions (C—H 0.96 Å). 154 parameters were refined. Weights $w = 1.0/[\sigma^2(F) + (0.005F^2)]$ led to a featureless analysis of variance in terms of $\sin\theta$ and F_o. The refinement converged to $S = 0.92$, $R = 0.048$, $wR = 0.071$, $(\Delta/\sigma)_{\max} = 0.033$ (no extinction correction). The correctness of the space-group choice was checked by using *MISSYM* (Le Page, 1987). The largest peaks in final $\Delta\rho$ map were ± 0.1 (1) e Å^{-3}. Atomic scattering factors for neutral atoms and real and imaginary dispersion terms were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). The programs used were *PARST* (Nardelli, 1983), *SHELX76* (Sheldrick, 1976), *SHELXTL-Plus* (Sheldrick, 1987), *PCK83* (Williams, 1984), *PLATON* (Spek, 1982). The molecule and the numbering scheme are shown in Fig. 1 and a stereoscopic view of the unit-cell contents is in Fig. 2. Positional parameters and equivalent values of the anisotropic displacement parameters for the non-H atoms are given in Table 1.* Bond lengths, bond angles, torsion angles, least-squares planes, a dihedral angle and possible hydrogen bonds are given in Table 2.

Related literature. For further details concerning different methods of preparation, see de Diesbach & Bulliard (1924) and Seidel & Bezner (1932).

One of us (TsK) thanks the Alexander von Humboldt Stiftung for a research followship.

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54782 (10 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AL0513]

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Structure of Xanthoherquein Tetraacetate*

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Abstract. $C_{23}H_{20}O_{11}.0.25H_2O$, $M_r = 476.91$, monoclinic, $C2/c$, $a = 24.146$ (1), $b = 8.919$ (2), $c = 22.044$ (2) Å, $\beta = 107.69$ (1)°, $V = 4523$ (1) Å^3, $Z = 8$, D_m (flotation in aqueous KI) = 1.391, $D_x = 1.400$ g cm^{-3}, Cu $K\alpha$, $\lambda = 1.54178$ Å, $\mu = 9.4$ cm^{-1}, $F(000) = 1988$, $T = 294$ K, $R = 0.060$ for 2810 reflections. The molecule contains an approximately planar perinaphthenone ring skeleton, with a delocalized π system and a symmetrical intramolecular O···H···O hydrogen bond [O···O = 2.483 (3), O···H = 1.29, 1.23 Å, O···H···O = 159°]. Small electron-density peaks on twofold (C_2) axes are interpreted as two partially occupied sites for water molecules, which are weakly hydrogen bonded to the carbonyl oxygen of an acetate group [O···O = 2.92, 2.95 (1) Å].

Experimental. Yellow prisms (Neill & Raistrick, 1956), 0.10 × 0.40 × 0.10 mm, Rigaku AFC-6S diffractometer, monochromatized Cu $K\alpha$ radiation, lattice parameters from 25 reflections with $2\theta = 55$–95°. Intensities for $2\theta \le 155°$, *hkl*: −30 to 29, 0 to 11, 0 to 24, ω–2θ scan, ω-scan width $(1.10 + 0.20\tan\theta)°$ at 32° min^{-1}, up to nine scans, stationary background counts (peak:background time = 2:1), three standard reflections showed no significant variation, Lp and absorption corrections (ψ scan, relative transmission factors 0.63–1.0). 4556 unique reflections, 2810 with $I \ge 3\sigma(I)$. Structure by direct methods, refined by full-matrix least-squares procedures, H1 from a difference map, other H atoms in calculated posi-

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* 2,3,6,7-Acetoxy-9-hydroxy-8-methoxy-4-methylperinaphthen-1-one; 2,3,6,7-acetoxy-9-hydroxy-8-methoxy-4-methylphenalen-1-one.

Table 1. *Positional (fractional) and equivalent isotropic thermal parameters* (Å²)

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

	x	y	z	B_{eq}
O1	0.76964 (10)	−0.10410 (24)	0.52383 (12)	3.9 (1)
O2	0.85481 (9)	0.0791 (3)	0.50849 (10)	3.42 (8)
O3	0.86381 (9)	0.3616 (3)	0.55240 (10)	3.54 (9)
O4	0.89651 (12)	−0.0945 (3)	0.58263 (13)	5.2 (1)
O5	0.93962 (13)	0.2578 (4)	0.62473 (17)	7.7 (2)
O6	0.66999 (10)	0.48339 (25)	0.69361 (10)	3.56 (9)
O7	0.61893 (9)	0.2158 (3)	0.67715 (10)	3.64 (9)
O8	0.60650 (10)	−0.0546 (3)	0.62173 (12)	4.1 (1)
O9	0.68463 (10)	−0.1498 (3)	0.56240 (12)	4.0 (1)
O10	0.72034 (13)	0.3776 (3)	0.78573 (12)	5.0 (1)
O11	0.54972 (11)	0.2586 (3)	0.58474 (13)	5.3 (1)
O12†	0	−0.382 (3)	$\frac{1}{4}$	10.2 (7)
O13	0	−0.125 (4)	$\frac{1}{4}$	13 (1)
C1	0.77460 (13)	0.0271 (3)	0.54948 (14)	3.0 (1)
C2	0.81990 (13)	0.1248 (3)	0.54427 (14)	3.0 (1)
C3	0.82410 (12)	0.2666 (3)	0.56734 (14)	2.8 (1)
C3a	0.78705 (12)	0.3260 (3)	0.60174 (14)	2.8 (1)
C3b	0.74185 (11)	0.2283 (3)	0.60801 (13)	2.6 (1)
C4	0.79187 (14)	0.4720 (3)	0.62804 (16)	3.4 (1)
C5	0.75229 (16)	0.5157 (4)	0.65864 (16)	3.7 (1)
C6	0.70907 (14)	0.4231 (3)	0.66454 (14)	3.1 (1)
C6a	0.70216 (12)	0.2772 (3)	0.64014 (13)	2.8 (1)
C7	0.65739 (12)	0.1747 (3)	0.64396 (14)	3.0 (1)
C8	0.65179 (12)	0.0352 (4)	0.61923 (15)	3.2 (1)
C9	0.69097 (13)	−0.0167 (3)	0.58668 (15)	3.0 (1)
C9a	0.73623 (12)	0.0812 (3)	0.58200 (13)	2.7 (1)
C10	0.89099 (14)	−0.0417 (4)	0.53139 (17)	3.7 (1)
C11	0.91903 (17)	−0.0898 (5)	0.48322 (20)	4.7 (2)
C12	0.92190 (15)	0.3411 (5)	0.58150 (19)	4.5 (2)
C13	0.95569 (18)	0.4380 (6)	0.55101 (22)	5.9 (2)
C14	0.83849 (19)	0.5850 (4)	0.62664 (21)	5.1 (2)
C15	0.68054 (16)	0.4520 (4)	0.75708 (15)	3.6 (1)
C16	0.63638 (19)	0.5265 (5)	0.78116 (18)	5.0 (2)
C17	0.56311 (14)	0.2444 (4)	0.64091 (18)	3.9 (1)
C18	0.52408 (19)	0.2540 (7)	0.68167 (24)	6.8 (2)
C19	0.62134 (21)	−0.1610 (5)	0.6720 (3)	6.3 (2)
H1	0.7271	−0.1531	0.5425	4.8

† O12 and O13 occupancies are 0.25 (one of each/cell).

tions based on difference map peaks. Two small peaks on a C_2 axis were interpreted as partially occupied water sites [the crystals used were about 35 years old, and although it is possible that the water content changed over that period, this seems unlikely since the present unit-cell parameters are close to those determined originally by film methods (Trotter, 1957)]. *TEXSAN* (Molecular Structure Corporation, 1985), refinement on F, with $w = 1/\sigma^2(F)$ (including a p factor, adjusted to 0.08 to give uniform average values of $\sum w\Delta F^2$), atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV). Final $R = 0.060$, $wR = 0.083$ for 2810 reflections with $I > 3\sigma(I)$, 311 parameters, $S = 1.6$, maximum $\Delta/\sigma = 0.01$, $\Delta\rho = -0.33$ to $+0.33$ e Å^{-3} (the largest peaks suggest possible H disorder in the methyl groups C11, C18 and C19). Positional parameters are in Table 1, bond lengths and angles in Table 2, and a view of the molecule in Fig. 1.*

Table 2. *Bond distances* (Å) *and angles* (°)

O1—C1	1.289 (4)	C2—C3	1.355 (4)
O2—C2	1.380 (3)	C3—C3a	1.438 (4)
O2—C10	1.382 (4)	C3a—C3b	1.436 (4)
O3—C3	1.392 (3)	C3a—C4	1.416 (4)
O3—C12	1.366 (4)	C3b—C6a	1.422 (4)
O4—C10	1.194 (4)	C3b—C9a	1.422 (4)
O5—C12	1.181 (5)	C4—C5	1.383 (5)
O6—C6	1.399 (4)	C4—C14	1.518 (5)
O6—C15	1.373 (4)	C5—C6	1.368 (5)
O7—C7	1.395 (3)	C6—C6a	1.398 (4)
O7—C17	1.366 (4)	C6a—C7	1.438 (4)
O8—C8	1.369 (3)	C7—C8	1.349 (4)
O8—C19	1.421 (5)	C8—C9	1.427 (4)
O9—C9	1.292 (4)	C9—C9a	1.426 (4)
O10—C15	1.181 (4)	C10—C11	1.486 (5)
O11—C17	1.188 (4)	C12—C13	1.483 (5)
C1—C2	1.430 (4)	C15—C16	1.485 (5)
C1—C9a	1.418 (4)	C17—C18	1.489 (5)

Hydrogen bonds (*cf. e.g.* Hamilton & Ibers, 1968)

O1...O9	2.483 (3)	O1—H1—O9	159
O1—H1	1.29	C1—O1—H1	98
O9—H1	1.23	C9—O9—H1	99

C2—O2—C10	116.7 (2)	C3b—C6a—C6	117.2 (3)
C3—O3—C12	119.4 (3)	C3b—C6a—C7	117.8 (3)
C6—O6—C15	116.5 (2)	C6—C6a—C7	125.0 (3)
C7—O7—C17	116.0 (2)	O7—C7—C6a	119.7 (3)
C8—O8—C19	114.0 (3)	O7—C7—C8	117.0 (3)
O1—C1—C2	119.3 (3)	C6a—C7—C8	123.2 (3)
O1—C1—C9a	122.9 (3)	O8—C8—C7	120.4 (3)
C2—C1—C9a	117.8 (3)	O8—C8—C9	119.4 (3)
O2—C2—C1	118.7 (3)	C7—C8—C9	120.1 (3)
O2—C2—C3	120.1 (3)	O9—C9—C8	119.5 (3)
C1—C2—C3	120.7 (3)	O9—C9—C9a	122.0 (3)
O3—C3—C2	117.2 (3)	C8—C9—C9a	118.5 (3)
O3—C3—C3a	119.0 (3)	C1—C9a—C3b	121.4 (2)
C2—C3—C3a	123.6 (3)	C1—C9a—C9	117.5 (3)
C3—C3a—C3b	116.1 (3)	C3b—C9a—C9	121.1 (3)
C3—C3a—C4	124.6 (3)	O2—C10—O4	122.0 (3)
C3b—C3a—C4	119.3 (3)	O2—C10—C11	109.8 (3)
C3a—C3b—C6a	120.6 (3)	O4—C10—C11	128.2 (3)
C3a—C3b—C9a	120.2 (2)	O3—C12—O5	122.1 (3)
C6a—C3b—C9a	119.2 (2)	O3—C12—C13	109.7 (3)
C3a—C4—C5	118.4 (3)	O5—C12—C13	128.2 (4)
C3a—C4—C14	124.9 (3)	O6—C15—O10	121.9 (3)
C5—C4—C14	116.7 (3)	O6—C15—C16	110.0 (3)
C4—C5—C6	122.4 (3)	O10—C15—C16	128.1 (3)
O6—C6—C5	116.8 (3)	O7—C17—O11	122.5 (3)
O6—C6—C6a	121.1 (3)	O7—C17—C18	110.5 (3)
C5—C6—C6a	122.0 (3)	O11—C17—C18	126.9 (4)

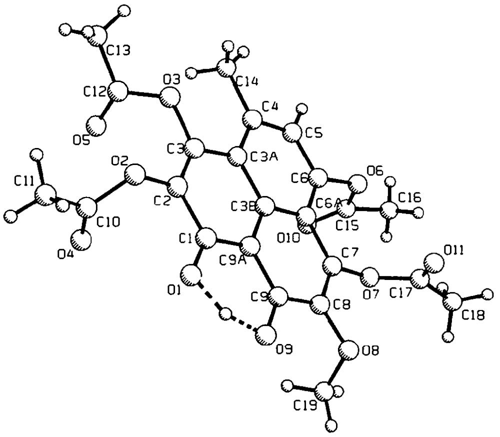

Fig. 1. View of the molecule.

Related literature. Previous efforts to determine the structure of the title compound (I) from two-dimensional film data were unsuccessful (Trotter,

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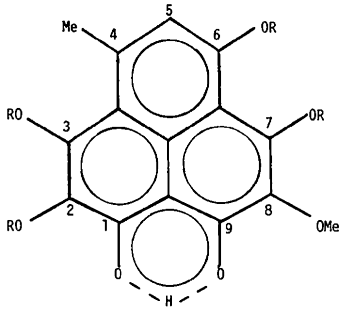
* Lists of anisotropic thermal parameters, hydrogen positions, intramolecular distances and angles involving H atoms, torsion angles, intermolecular distances and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54740 (30 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

1957). The general structure of this type of molecule has been established by other studies (*e.g.* Barton, de Mayo, Morrison & Raistrick, 1959; Paul & Sim, 1965; Svensson, Abrahams, Bernstein & Haddon, 1979).



(I) $R = COCH_3$

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Structure of $[Ni(OH_2)_2(NH_2CH_2CH_2CH_2NH_2)_2][picrate]_2$

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Abstract. Diaquabis(1,3-diaminopropane)nickel(II) picrate, $C_6H_{24}N_4NiO_2^{2+}.2C_6H_2N_3O_7^-$, $M_r = 699.2$, monoclinic, $I2/a$ (non-standard setting of $C2/c$), $a = 16.2880\,(6)$, $b = 7.5741\,(3)$, $c = 22.6836\,(8)$ Å, $\beta = 96.606\,(4)$°, $V = 2779.8$ Å^3, $Z = 4$, $D_m = 1.69\,(1)$, $D_x = 1.670$ Mg m^{-3}, $\lambda(\text{Cu } K\alpha) = 1.54184$ Å, $\mu = 1.82$ mm^{-1}, $F(000) = 1448$, $T = 295$ K, $R = 0.0699$ for 2300 unique observed reflections. The structure contains discrete $[Ni(OH_2)_2(NH_2CH_2CH_2CH_2NH_2)_2]^{2+}$ cations and picrate anions, which are hydrogen bonded to the aqua ligands [O···O 2.670 (6) Å]. The Ni atom lies on a crystallographic centre of inversion; octahedral coordination around it is effected by two water and two bidentate 1,3-diaminopropane molecules to give a *trans*-N_4O_2 donor set. The Ni—O [2.126 (2) Å] and Ni—N [2.109 (2) and 2.101 (2) Å] distances are within normal ranges, and *cis* coordination angles range from 88.9 (1) to 91.2 (1)°. The six-membered chelate rings have a chair conformation.

Experimental. The compound was prepared by reaction of 0.51 g (1 mmol) nickel picrate (Black & McLean, 1971) with 0.15 g (2 mmol) of freshly distilled 1,3-diaminopropane in 25 ml acetonitrile. An orange solution was obtained, which upon slow evaporation at room temperature afforded well formed orange crystals. The density was measured by flotation. A crystal of size 0.5 × 0.5 × 0.5 mm, on a glass fibre, was examined on a Stoe–Siemens diffractometer. Unit-cell parameters were refined from 2θ values of 32 reflections (45–50°) measured at $\pm\omega$. Data collection employed ω/θ scans and on-line profile fitting (Clegg, 1981), to a maximum 2θ of 130°; index ranges were h $-19\rightarrow19$, k $0\rightarrow8$, l $0\rightarrow26$, together with a partial set of equivalent reflections. No significant variation was observed in the intensities of three standard reflections. Semi-empirical absorption corrections were applied, with transmission factors in the range 0.261–0.392. The 3557 measured reflections yielded 2335 unique data, 2300 with $F > 4\sigma_c(F)$ (σ_c from counting statistics only); $R_{\text{int}} = 0.042$.

The structure was solved by direct methods, and blocked-cascade least-squares refinement on F, with weighting $w^{-1} = \sigma^2(F) = \sigma_c^2(F) + 54 - 229G + 857G^2$

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