Table 1. *Atomic coordinates* ($\times 10^4$) *and equivalent isotropic displacement coefficients* ($\text{Å}^2 \times 10^3$)

U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
Ni	0	0	5000	82 (1)
O(1)	1693 (10)	1693	5000	121 (5)
O(2)	−1197 (6)	1197	4197 (4)	98 (3)
C(1)	3204 (20)	1474 (25)	5000	250 (18)
C(2)	4038 (22)	2865 (25)	5000	241 (17)
C(3)	−995 (10)	995	3488 (8)	94 (4)
C(4)	0	0	3120 (9)	88 (4)
C(5)	−2042 (14)	2042	3026 (9)	123 (5)
F(1)	−1937 (9)	1937	2294 (5)	187 (5)
F(2)	−3500 (9)	1810 (14)	3183 (5)	247 (5)

Table 2. *Bond lengths* (Å) *and bond angles* (°)

Ni—O(1)	2·041 (12)	Ni—O(2)	2·011 (5)
Ni—O(1*A*)	2·041 (12)	Ni—O(2*A*)	2·011 (5)
Ni—O(2*B*)	2·012 (5)	Ni—O(2*C*)	2·012 (5)
O(1)—C(1)	1·301 (19)	O(1)—C(1*A*)	1·301 (19)
O(2)—C(3)	1·262 (15)	C(1)—C(2)	1·383 (29)
C(2)—C(2*A*)	1·415 (41)	C(3)—C(4)	1·360 (10)
C(3)—C(5)	1·497 (12)	C(4)—C(3*A*)	1·360 (10)
C(5)—F(1)	1·283 (19)	C(5)—F(2)	1·288 (15)
C(5)—F(2*A*)	1·288 (15)		

O(1)—Ni—O(2)	90·0 (2)	O(1)—Ni—O(1*A*)	180·0 (1)
O(2)—Ni—O(1*A*)	90·0 (2)	O(1)—Ni—O(2*A*)	90·0 (2)
O(2)—Ni—O(2*A*)	180·0 (1)	O(1*A*)—Ni—O(2*A*)	90·0 (2)
O(1)—Ni—O(2*B*)	90·0 (2)	O(2)—Ni—O(2*B*)	91·7 (3)
O(1*A*)—Ni—O(2*B*)	90·0 (2)	O(2*A*)—Ni—O(2*B*)	88·3 (3)
O(1)—Ni—O(2*C*)	90·0 (2)	O(2)—Ni—O(2*C*)	88·3 (3)
O(1*A*)—Ni—O(2*C*)	90·0 (2)	O(2*A*)—Ni—O(2*C*)	91·7 (3)
O(2*B*)—Ni—O(2*C*)	180·0 (1)	Ni—O(1)—C(1)	126·7 (10)
Ni—O(1)—C(1*A*)	126·7 (10)	C(1)—O(1)—C(1*A*)	106·5 (20)
Ni—O(2)—C(3)	123·0 (1)	O(1)—C(1)—C(2)	112·7 (17)
C(1)—C(2)—C(2*A*)	104·0 (11)	O(2)—C(3)—C(4)	129·3 (8)
O(2)—C(3)—C(5)	111·4 (6)	C(4)—C(3)—C(5)	119·3 (12)
C(3)—C(4)—C(3*A*)	123·7 (15)	C(3)—C(5)—F(1)	116·9 (6)
C(3)—C(5)—F(2)	111·7 (12)	F(1)—C(5)—F(2)	105·6 (12)
C(3)—C(5)—F(2*A*)	111·6 (12)	F(1)—C(5)—F(2*A*)	105·6 (12)
F(2)—C(5)—F(2*A*)	104·5 (11)		

atoms located on difference Fourier maps. H atoms were placed in idealized positions with fixed isotropic U. All non-H atoms were refined anisotropically using scattering factors, including terms for anomalous dispersion, taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). Refinement based on F using weights of the form $w^{-1} = [\sigma^2(F) + 0{\cdot}0022(F^2)]$. Convergence to conventional R values of $R = 0{\cdot}0645$ and $wR = 0{\cdot}0935$ obtained using 58 variable parameters and 312 observed reflections with $I > 3\sigma(I)$. No reflections had intensities beyond the range for valid coincidence correction. For final cycle, max. shift/σ = 0·000 with min. and max. residual electron densities of −0·30 and 0·28 e Å^{-3}, respectively. A perspective drawing of the structure illustrating the atomic numbering scheme is given in Fig. 1; Fig. 2 is a stereoview packing diagram of the complex. Atomic positional parameters and equivalent isotropic thermal parameters for all non-H atoms are presented in Table 1.* Bond distances and angles are summarized in Table 2.

Related literature. A number of transition metal complexes containing hfa ligands have been characterized crystallographically and the coordination chemistry of β-diketones has been reviewed (Fackler, 1965; Kawaguchi, 1986). The structure reported here is similar to a recently reported Ni(hfa)$_2$ complex containing nitroxyl radical ligands (Porter, Dickman & Doedens, 1988).

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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 52662 (4 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Tetrakis(μ-acetato)bis(2-pyridone)dicopper

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Abstract. $[Cu_2(C_5H_5NO)_2(C_2H_3O_2)_4]$, $M_r = 553{\cdot}4$, monoclinic, $C2/c$, $a = 13{\cdot}569\ (3)$, $b = 8{\cdot}6013\ (13)$, $c = 18{\cdot}564\ (4)$ Å, $\beta = 93{\cdot}541\ (11)°$, $V = 2162$ Å^3, $Z = 4$ (implying that each molecule lies on a twofold special position), $D_x = 1{\cdot}700$ Mg m^{-3}, $\lambda(\text{Mo } K\alpha) = 0{\cdot}71073$ Å, $\mu = 2{\cdot}027$ mm^{-1}, $F(000) = 1128$, $T =$

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Table 1. *Atomic coordinates with e.s.d.'s*

	x	y	z	U_{iso}(Å^2)
Cu	−0·02258 (3)	0·07371 (5)	0·439520 (20)	0·01599 (23)
O(1)	0·12162 (16)	0·0964 (3)	0·43522 (13)	0·0238 (13)
O(2)	−0·15967 (17)	0·0282 (3)	0·46127 (14)	0·0289 (14)
O(3)	−0·02691 (19)	0·2617 (3)	0·49991 (14)	0·0265 (14)
O(4)	−0·01114 (19)	−0·1355 (3)	0·39711 (13)	0·0256 (14)
O(5)	−0·04868 (17)	0·1821 (3)	0·33519 (12)	0·0233 (13)
N(1)	−0·12858 (22)	0·2545 (4)	0·23029 (16)	0·0220 (16)
C(1)	0·18244 (24)	0·0415 (4)	0·48239 (18)	0·0181 (17)
C(2)	0·2908 (3)	0·0605 (5)	0·47185 (22)	0·0255 (20)
C(3)	−0·01007 (24)	0·2559 (4)	0·56644 (19)	0·0191 (17)
C(4)	−0·0186 (3)	0·4055 (5)	0·60909 (24)	0·0311 (22)
C(5)	−0·12672 (25)	0·2352 (4)	0·30293 (18)	0·0193 (17)
C(6)	−0·2143 (3)	0·2778 (5)	0·33603 (22)	0·0268 (21)
C(7)	−0·2936 (3)	0·3372 (5)	0·29681 (22)	0·0302 (22)
C(8)	−0·2912 (3)	0·3566 (5)	0·22175 (22)	0·0307 (21)
C(9)	−0·2078 (3)	0·3128 (5)	0·19018 (21)	0·0271 (20)
H(1)	−0·082 (3)	0·228 (5)	0·2121 (21)	0·023 (11)
H(21)	0·325 (3)	−0·034 (5)	0·4808 (21)	0·029 (11)
H(22)	0·317 (3)	0·137 (5)	0·5021 (22)	0·030 (11)
H(23)	0·301 (4)	0·099 (5)	0·429 (3)	0·052 (15)
H(41)	−0·015 (3)	0·497 (5)	0·5832 (22)	0·028 (11)
H(42)	0·022 (3)	0·398 (5)	0·6559 (24)	0·037 (12)
H(43)	−0·083 (3)	0·414 (5)	0·6237 (23)	0·037 (12)
H(6)	−0·216 (3)	0·258 (4)	0·3810 (22)	0·023 (10)
H(7)	−0·351 (3)	0·365 (4)	0·3163 (20)	0·020 (9)
H(8)	−0·354 (3)	0·399 (4)	0·1916 (21)	0·029 (11)
H(9)	−0·195 (3)	0·327 (4)	0·1420 (21)	0·024 (10)

Table 2. *Bond lengths* (Å) *and angles* (°) *with e.s.d.'s*

Cu—Cu′	2·6183 (8)	O(5)—C(5)	1·269 (4)
Cu—O(1)	1·9728 (24)	N(1)—C(5)	1·357 (5)
Cu—O(2)	1·967 (3)	N(1)—C(9)	1·365 (5)
Cu—O(3)	1·970 (3)	N(1)—H(1)	0·77 (4)
Cu—O(4)	1·9741 (25)	C(1)—C(2)	1·503 (5)
Cu—O(5)	2·1589 (24)	C(3)—C(4)	1·519 (5)
O(2′)—C(1)	1·260 (4)	C(5)—C(6)	1·419 (5)
O(4′)—C(3)	1·261 (4)	C(6)—C(7)	1·362 (6)
O(1)—C(1)	1·257 (4)	C(7)—C(8)	1·406 (6)
O(3)—C(3)	1·243 (4)	C(8)—C(9)	1·358 (6)

Cu′—Cu—O(1)	84·40 (7)	Cu—O(3)—C(3)	121·50 (23)
Cu′—Cu—O(2)	84·36 (8)	Cu—O(5)—C(5)	131·66 (22)
Cu′—Cu—O(3)	85·59 (8)	C(5)—N(1)—C(9)	123·5 (3)
Cu′—Cu—O(4)	83·14 (7)	C(5)—N(1)—H(1)	115·7 (29)
Cu′—Cu—O(5)	174·40 (7)	C(9)—N(1)—H(1)	120·7 (29)
O(1)—Cu—O(2)	168·75 (10)	O(2′)—C(1)—O(1)	124·9 (3)
O(1)—Cu—O(3)	90·37 (10)	O(2′)—C(1)—C(2)	116·8 (3)
O(1)—Cu—O(4)	88·35 (10)	O(1)—C(1)—C(2)	118·2 (3)
O(1)—Cu—O(5)	91·68 (10)	O(4′)—C(3)—O(3)	126·0 (3)
O(2)—Cu—O(3)	89·12 (11)	O(4′)—C(3)—C(4)	116·0 (3)
O(2)—Cu—O(4)	89·95 (11)	O(3)—C(3)—C(4)	118·0 (3)
O(2)—Cu—O(5)	99·51 (10)	O(5)—C(5)—N(1)	118·5 (3)
O(3)—Cu—O(4)	168·73 (11)	O(5)—C(5)—C(6)	125·8 (3)
O(3)—Cu—O(5)	98·46 (10)	N(1)—C(5)—C(6)	115·7 (3)
O(4)—Cu—O(5)	92·78 (10)	C(5)—C(6)—C(7)	121·4 (4)
Cu′—O(2′)—C(1)	123·26 (23)	C(7)—C(8)—C(9)	117·8 (4)
Cu′—O(4′)—C(3)	123·77 (23)	C(6)—C(7)—C(8)	120·5 (4)
Cu—O(1)—C(1)	123·00 (22)	N(1)—C(9)—C(8)	121·0 (4)

Primed atoms are related to their unprimed equivalents by inversion through (0,0,1/2).

173·0 ± 0·1 K, $R = 0·0313$ for 1938 unique observed reflections. The molecule lies across a crystallographic inversion centre with the Cu—Cu vector of 2·6183 (8) Å bridged by four acetate groups and with the pyridone ligands in axial positions. N—H···O hydrogen bonding between pyridone ligands links the molecules into infinite chains running parallel to **c** and pyridyl rings from neighbouring chains stack parallel to **b**.

Experimental. Reaction of potassium pyridonate, copper nitrate and gadolinium perchlorate in methanol gave a purple solid which dissolved in 15:1 methanol:acetic acid to give a green solution from which crystals were obtained by concentration at 276 K. Turquoise rhomboidal tablet, 0·48 × 0·32 × 0·20 mm, Stoe STADI-4 four-circle diffractometer equipped with Oxford Cryosystems low-temperature device (Cosier & Glazer, 1986), graphite-monochromated Mo $K\alpha$ X-radiation, cell parameters from 2θ values of 33 reflections measured at $\pm\omega$ ($30 < 2\theta < 32°$). For data collection, $T = 173$ K, ω–2θ scans with ω-scan width $(1·40 + 0·347\tan\theta)°$, $2\theta_{max} = 52°$, $h\ -16\rightarrow16$, $k\ 0\rightarrow10$, $l\ 0\rightarrow22$, 3 standard reflections indicated no significant crystal movement or decay in intensities, no absorption correction. 2243 unique reflections of which 1938 with $F > 6\sigma(F)$ were used for structure solution [from a Patterson synthesis followed by iterative cycles of least-squares refinement and difference Fourier synthesis] and refinement [using full-matrix least squares on F

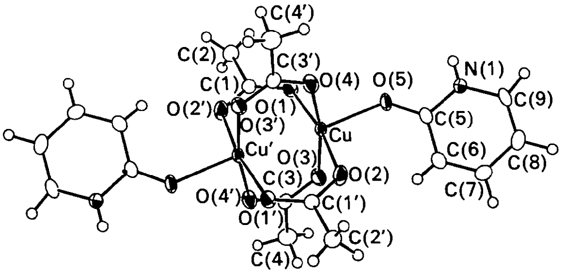

Fig. 1. A general view of the centrosymmetric molecule showing the atom-numbering scheme: primed atoms are related to their unprimed equivalents by inversion through (0,0,1/2). Thermal ellipsoids are drawn at the 30% probability level, except those of the H atoms which have artificial radii of 0·10 Å for clarity.

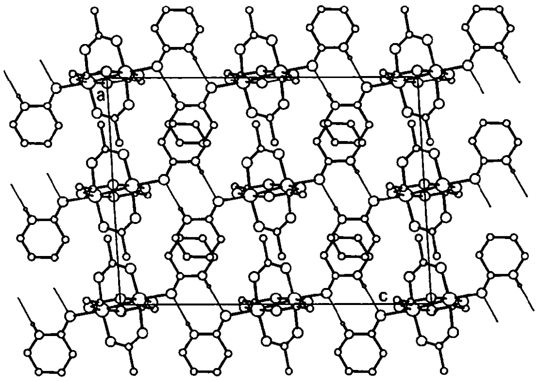

Fig. 2. A view along the b axis showing the molecular packing, with H atoms not involved in hydrogen bonding omitted for clarity.

(*SHELX*76; Sheldrick, 1976)]. Anisotropic thermal parameters for all non-H atoms, H-atom positions freely refined. At final convergence, $R = 0{\cdot}0313$, $wR = 0{\cdot}0405$, $S = 1{\cdot}063$ for 190 parameters, $(\Delta/\sigma)_{\max}$ in final cycle 0·006, max. and min. residues in final ΔF synthesis 0·50, $-0{\cdot}78\ \text{e Å}^{-3}$, respectively. The weighting scheme $w^{-1} = \sigma^2(F) + 0{\cdot}000354F^2$ gave satisfactory agreement analyses. Scattering factors were inlaid (Sheldrick, 1976) except for Cu (Cromer & Mann, 1968). Atomic coordinates and equivalent isotropic thermal parameters are given in Table 1, while selected bond lengths and angles appear in Table 2.* The atom-numbering scheme for the molecule is shown in Fig. 1, which was generated using *ORTEP*II (Mallinson & Muir, 1985). Fig. 2 shows the molecular packing and was produced using *PLUTO* (Motherwell, 1976). Molecular geometry calculations were performed using *CALC* (Gould & Taylor, 1985).

Related literature. A large number of analogous species have been reported (Catterick & Thornton, 1977) with different axial ligands, including 2-phenoxybenzoic acid (Mak, Yip, Kennard & Smith, 1990), H_2O (Brown & Chidambaram, 1973) and methanol, acetic acid, dimethylformamide and 1,4-diazabicyclo[2.2.2]octane (Rao, Sathyanarayana & Munohar, 1983) which have Cu–Cu distances in the range 2·581–2·632 Å.

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Structure du [4-Phénylacétylimino-1-(1,2,4-triazolio)] Acétate de Sodium Dihydrate

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(*Reçu le* 11 *octobre* 1990, *accepté le* 26 *octobre* 1990)

Abstract. *N*-[1-Carboxymethyl-4-(1,2,4-triazolio)]-phenylacetamidate sodium salt dihydrate, $C_{12}H_{11}N_4O_3^-.Na^+.2H_2O$, $M_r = 318{\cdot}3$, monoclinic, $P2_1/n$, $a = 5{\cdot}382\,(1)$, $b = 32{\cdot}005\,(6)$, $c = 8{\cdot}285\,(2)$ Å, $\beta = 100{\cdot}78\,(2)°$, $V = 1401{\cdot}8\,(1)\ \text{Å}^3$, $Z = 4$, $D_x = 1{\cdot}507\ \text{g cm}^{-3}$, $\lambda(\text{Cu}\ K\bar{\alpha}) = 1{\cdot}5418$ Å, $\mu = 11{\cdot}3\ \text{cm}^{-1}$, $F(000) = 664$, $T = 294\,(1)$ K, $R = 0{\cdot}049$ for 1704 independent reflections. The triazolium ring is planar [maximum deviation 0·005 (3) Å]. C(2) and N(4) are significantly out of the plane by 0·141 (3) and 0·137 (2) Å respectively. The coordination about the Na atom involves six O atoms with distances in the range 2·282–2·507 (2) Å. All the H atoms of the water molecules participate in O—O hydrogen bonding [the range of H···*A* distances is 1·86–1·98 (2) Å; the O—H···*A* angle range is 161–170 (2)°]. Presu-

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