Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters* (Å^2) *for non*-H *atoms with e.s.d.'s in parentheses*

$U_{eq} = (U_{11} + U_{22} + U_{33})/3.$

	x	y	z	U_{eq}
Cu	0.2216 (1)	0.1536 (1)	0.0546 (1)	0.0377 (4)
O1	0.3352 (8)	−0.0342 (5)	−0.3642 (5)	0.0765 (38)
O2	0.3234 (7)	−0.2747 (5)	−0.1578 (5)	0.0644 (30)
N1	0.2547 (8)	0.1251 (6)	−0.1362 (6)	0.0592 (35)
N2	0.1966 (7)	−0.0660 (5)	−0.0032 (6)	0.0477 (28)
N3	0.2396 (6)	0.3822 (5)	0.1376 (5)	0.0408 (26)
N4	0.2246 (6)	0.2195 (5)	0.2674 (4)	0.0364 (24)
C1	0.2941 (9)	0.0443 (6)	−0.2483 (7)	0.0485 (34)
C2	0.2627 (8)	−0.1664 (5)	−0.0786 (5)	0.0375 (29)
C31	0.2536 (10)	0.4577 (7)	0.0616 (8)	0.0608 (42)
C32	0.2694 (12)	0.6172 (9)	0.1303 (11)	0.0746 (66)
C33	0.2675 (10)	0.6991 (8)	0.2764 (10)	0.0687 (64)
C34	0.2506 (9)	0.6233 (6)	0.3556 (8)	0.0488 (41)
C35	0.2372 (7)	0.4638 (6)	0.2827 (6)	0.0374 (31)
C41	0.2217 (9)	0.1274 (7)	0.3260 (7)	0.0498 (34)
C42	0.2212 (10)	0.1816 (9)	0.4735 (8)	0.0641 (46)
C43	0.2253 (10)	0.3341 (9)	0.5643 (7)	0.0571 (47)
C44	0.2290 (9)	0.4313 (7)	0.5064 (6)	0.0472 (35)
C45	0.2283 (7)	0.3712 (6)	0.3577 (5)	0.0322 (26)

Table 2. *Selected interatomic distances* (Å) *and bond angles* (°) *with e.s.d.'s in parentheses*

Cu—N1	1.907 (5)	C33—C34	1.356 (10)
Cu—N2	1.942 (5)	C34—C35	1.377 (7)
Cu—N3	1.991 (4)	C35—N3	1.335 (6)
Cu—N4	1.983 (4)	C35—C45	1.463 (8)
N1—C1	1.155 (7)	N4—C41	1.321 (7)
C1—O1	1.183 (6)	C41—C42	1.363 (8)
N2—C2	1.151 (6)	C42—C43	1.350 (10)
C2—O2	1.191 (6)	C43—C44	1.362 (10)
N3—C31	1.323 (8)	C44—C45	1.366 (7)
C31—C32	1.375 (9)	C45—N4	1.344 (6)
C32—C33	1.343 (11)	Cu—Cu^{i}	3.375 (6)

N1—Cu—N4	170.1 (2)	N2—Cu—N1	94.5 (4)
N2—Cu—N3	172.9 (2)	N2—C2—O2	177.3 (6)
N3—Cu—N4	79.9 (2)	N1—C1—O1	176.7 (7)
N3—Cu—N1	92.5 (2)	Cu—N2—C2	138.9 (5)
N2—Cu—N4	93.1 (2)	Cu—N1—C1	145.2 (5)

Symmetry code: (i) $-x, -y, -z$.

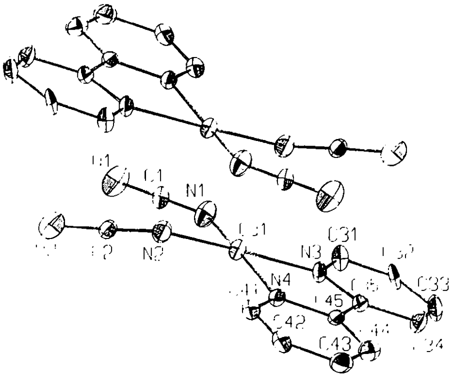

Fig. 1. Schematic representation of the structure of $[Cu_2(NCO)_4(bpy)_2]$ indicating the atom-numbering scheme.

The presence of Cu—Cu bonds in $[Cu_2(NCO)_4(phen)_2]$ and $[Cu_2(NCO)_4(bpy)_2]$ may explain why each Cu^{II} ion is associated with only one phen or bpy ligand. In the analogous NCS$^-$ and NCSe$^-$ complexes the Cu^{II} ions remain pentacoordinate but adopt trigonal bipyramidal coordinations involving two heterocyclic ligands and Cu—Cu bonding interactions do not occur (Sedov, Kabešová, Dunaj-Jurčo, Gažo & Garaj, 1983; Sedov Kožíšek, Kabešová, Dunaj-Jurčo, Gažo & Garaj, 1983).

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Bis[bis(triphenylphosphine)iminium] Dodecacarbonylhexanickelate

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Abstract. $[Ni_6(CO)_{12}][C_{36}H_{30}NP_2]_2$, $M_r = 1765.5$, triclinic, $P\bar{1}$, $a = 13.299\ (4)$, $b = 13.343\ (4)$, $c = 13.051\ (5)$ Å, $\alpha = 106.24\ (3)$, $\beta = 119.04\ (2)$, $\gamma = 81.41\ (3)$°, $V = 1943\ (1)$ Å^3, $Z = 1$, $D_x = 1.508$ g cm^{-3}, λ(Mo $K\alpha$) = 0.71069 Å, $\mu = 15.7$ cm^{-1}, $F(000) = 902$, $T = 296$ K. Final conventional $R = 0.033$ for 5134 observed reflections and 487 variables. The title compound has already been reported with Me_4N as the counter-cation. This sample employs bis(triphenylphosphine)iminium

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0108-2701/93/061121-03$06.00

Table 1. *Atomic coordinates and equivalent isotropic displacement parameters* ($Å^2 \times 10^2$) *with e.s.d.'s in parentheses*

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

	x	y	z	B_{eq}
Ni(1)	0.00875 (4)	0.13790 (3)	0.01932 (4)	3.47 (2)
Ni(2)	0.11318 (4)	0.02898 (3)	0.15715 (4)	3.65 (2)
Ni(3)	−0.09143 (4)	0.04660 (3)	0.07475 (4)	3.63 (2)
O(1)	0.0097 (3)	0.2814 (2)	−0.1094 (3)	7.4 (2)
O(2)	0.3248 (3)	−0.0628 (3)	0.3128 (3)	9.9 (2)
O(3)	−0.3090 (3)	0.0144 (3)	0.0584 (3)	8.1 (2)
O(12)	0.2491 (2)	0.1886 (2)	0.1736 (3)	6.0 (1)
O(13)	−0.2181 (2)	0.2239 (2)	−0.0198 (3)	5.8 (1)
O(23)	0.0204 (3)	−0.0202 (3)	0.2971 (3)	8.2 (2)
C(1)	0.0088 (3)	0.2261 (3)	−0.0574 (4)	4.5 (2)
C(2)	0.2414 (4)	−0.0245 (3)	0.2528 (4)	5.6 (2)
C(3)	−0.2224 (4)	0.0252 (3)	0.0661 (4)	4.9 (2)
C(12)	0.1669 (3)	0.1403 (3)	0.1350 (3)	4.0 (1)
C(13)	−0.1401 (3)	0.1658 (3)	0.0088 (3)	4.0 (1)
C(23)	0.0158 (4)	0.0047 (3)	0.2159 (4)	5.1 (2)

Table 2. *Selected bond lengths* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

Ni(1)—Ni(2)	2.390 (1)	Ni(2)—C(12)	1.887 (5)
Ni(1)—Ni(2*A*)	2.734 (1)	Ni(2)—C(23)	1.897 (6)
Ni(1)—Ni(3)	2.387 (1)	Ni(2*A*)—Ni(3)	2.791 (1)
Ni(1)—Ni(3*A*)	2.755 (1)	Ni(3)—C(3)	1.752 (6)
Ni(1)—C(1)	1.745 (5)	Ni(3)—C(13)	1.899 (4)
Ni(1)—C(12)	1.901 (3)	Ni(3)—C(23)	1.878 (4)
Ni(1)—C(13)	1.903 (5)	O(1)—C(1)	1.138 (7)
Ni(1*A*)—Ni(2)	2.734 (1)	O(2)—C(2)	1.147 (5)
Ni(1*A*)—Ni(3)	2.755 (1)	O(3)—C(3)	1.134 (7)
Ni(2)—Ni(3)	2.393 (1)	O(12)—C(12)	1.166 (5)
Ni(2)—Ni(3*A*)	2.791 (1)	O(13)—C(13)	1.167 (5)
Ni(2)—C(2)	1.745 (4)	O(23)—C(23)	1.168 (7)

Ni(2)—Ni(1)—Ni(2*A*)	91.01 (3)	Ni(1)—C(12)—Ni(2)	78.2 (2)
Ni(2)—Ni(1)—Ni(3)	60.12 (3)	Ni(1)—C(12)—O(12)	140.0 (3)
Ni(2)—Ni(1)—Ni(3*A*)	65.27 (3)	Ni(2)—C(12)—O(12)	141.6 (3)
Ni(2)—Ni(1)—C(1)	149.4 (1)	Ni(1)—C(13)—Ni(3)	77.8 (1)
Ni(2)—Ni(1)—C(12)	50.6 (1)	Ni(1)—C(13)—O(13)	141.9 (4)
Ni(2)—Ni(1)—C(13)	108.6 (1)	Ni(3)—C(13)—O(13)	140.3 (4)
Ni(2*A*)—Ni(1)—Ni(3)	65.65 (3)	Ni(2)—C(23)—Ni(3)	78.7 (2)
Ni(2*A*)—Ni(1)—Ni(3*A*)	51.69 (2)	Ni(2)—C(23)—O(23)	140.6 (3)
Ni(2*A*)—Ni(1)—C(1)	105.2 (1)	Ni(3)—C(23)—O(23)	140.7 (4)
Ni(2*A*)—Ni(1)—C(12)	127.5 (1)	Ni(3*A*)—Ni(2)—C(23)	128.1 (1)
Ni(2*A*)—Ni(1)—C(13)	77.8 (1)	C(2)—Ni(2)—C(12)	101.8 (2)
Ni(3)—Ni(1)—Ni(3*A*)	91.43 (3)	C(2)—Ni(2)—C(23)	99.5 (2)
Ni(3)—Ni(1)—C(1)	150.3 (1)	C(12)—Ni(2)—C(23)	140.0 (2)
Ni(3)—Ni(1)—C(12)	107.9 (2)	C(13)—Ni(3)—C(23)	141.3 (2)
Ni(3)—Ni(1)—C(13)	51.0 (1)	Ni(1)—C(1)—O(1)	178.1 (4)
Ni(3*A*)—Ni(1)—C(1)	104.8 (1)	Ni(2)—C(2)—O(2)	177.7 (4)
Ni(3*A*)—Ni(1)—C(12)	77.8 (1)	Ni(3)—C(3)—O(3)	177.3 (4)
Ni(3*A*)—Ni(1)—C(13)	127.7 (1)	Ni(1)—Ni(2)—Ni(1*A*)	88.99 (3)
C(1)—Ni(1)—C(12)	99.8 (2)	Ni(1)—Ni(2)—Ni(3)	59.88 (3)
C(1)—Ni(1)—C(13)	100.2 (2)	Ni(1)—Ni(2)—Ni(3*A*)	63.69 (3)
C(12)—Ni(1)—C(13)	141.2 (2)	Ni(1)—Ni(2)—C(2)	151.6 (2)
Ni(1*A*)—Ni(2)—C(23)	79.0 (1)	Ni(1)—Ni(2)—C(12)	51.1 (1)
Ni(3)—Ni(2)—Ni(3*A*)	90.42 (3)	Ni(1)—Ni(2)—C(23)	107.4 (1)
Ni(3)—Ni(2)—C(2)	148.5 (2)	Ni(1*A*)—Ni(2)—Ni(3)	64.59 (3)
Ni(3)—Ni(2)—C(12)	108.1 (1)	Ni(1*A*)—Ni(2)—Ni(3*A*)	51.17 (2)
Ni(3)—Ni(2)—C(23)	50.3 (1)	Ni(1*A*)—Ni(2)—C(2)	105.1 (1)
Ni(3*A*)—Ni(2)—C(2)	106.1 (2)	Ni(1*A*)—Ni(2)—C(12)	126.2 (1)
Ni(3*A*)—Ni(2)—C(12)	77.0 (1)	Ni(1)—Ni(3)—Ni(1*A*)	88.57 (3)
Ni(2)—Ni(3)—Ni(2*A*)	89.58 (4)	Ni(1)—Ni(3)—Ni(2)	60.00 (3)
Ni(2)—Ni(3)—C(3)	152.3 (1)	Ni(1)—Ni(3)—Ni(2*A*)	63.18 (3)
Ni(2)—Ni(3)—C(13)	108.6 (1)	Ni(1)—Ni(3)—C(3)	147.3 (1)
Ni(2)—Ni(3)—C(23)	51.0 (2)	Ni(1)—Ni(3)—C(13)	51.2 (1)
Ni(2*A*)—Ni(3)—C(3)	107.1 (1)	Ni(1)—Ni(3)—C(23)	108.1 (2)
Ni(2*A*)—Ni(3)—C(13)	76.4 (1)	Ni(1*A*)—Ni(3)—Ni(2)	63.72 (3)
Ni(2*A*)—Ni(3)—C(23)	127.8 (1)	Ni(1*A*)—Ni(3)—C(3)	109.8 (1)
C(3)—Ni(3)—C(13)	96.9 (2)	Ni(1*A*)—Ni(3)—C(13)	125.6 (1)
C(3)—Ni(3)—C(23)	102.0 (2)	Ni(1*A*)—Ni(3)—C(23)	78.8 (1)

(PPN) as the counter-cation. The metal anion shows an octahedral arrangement for the Ni atoms with one terminal and two bridging carbonyl fragments per metal atom.

Experimental. The compound was obtained from the reduction of $Ni(CO)_4$ with a potassium–bismuth alloy in ethylenediamine. The material was recrystallized from CH_3CN at 253 K. Dark-red diamond-shaped prism, 0.50 × 0.10 × 0.20 mm, mounted on glass fiber. D_m not determined. Rigaku AFC5*S* single-crystal diffractometer, Mo $K\alpha$ radiation, graphite-crystal monochromator. Unit-cell dimensions determined from angular settings of 25 reflections with 2θ between 22.4 and 31.6°. The International Union of Crystallography standard cell (13.051, 13.299, 13.343 Å, 81.41, 73.76, 60.96°) is given by applying the transformation matrix (001, $\bar{1}00$, $0\bar{1}0$) to the unit cell listed above. The space group was determined to be triclinic from the lack of symmetry and systematic absences. $P\bar{1}$ (No. 2) was chosen and shown to be correct by subsequent structure solution and refinement. 8909 reflections were measured, $0 < h < 17$, $-17 < k < 17$, $-17 < l < 17$, $4.0 < 2\theta < 55.0$, of which 8175 were unique (R_{int} =

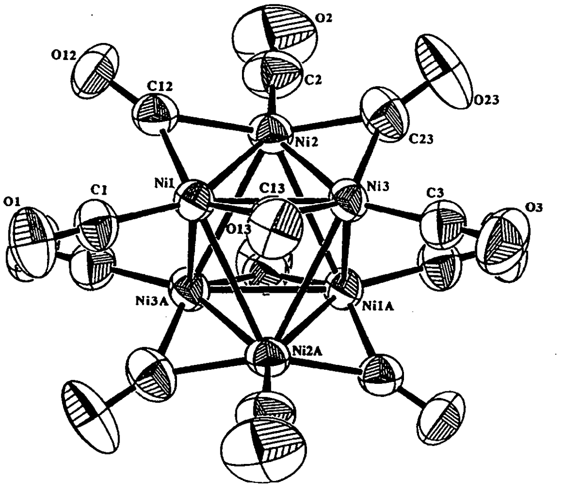

Fig. 1. *ORTEP* (Johnson, 1976) representation showing the cluster geometry and the atomic numbering scheme.

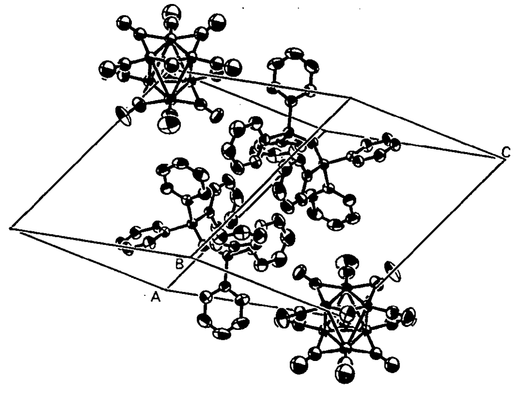

Fig. 2. Packing diagram of the title compound.

1.9%) and 5134 reflections were classified as observed with $I > 3\sigma(I)$. ω–2θ scan technique was employed with an 8.0° $\min^{-1}$ scan rate. Weak reflections [$I < 10\sigma(I)$] were rescanned (maximum two rescans) and the counts accumulated to assure good counting statistics. Stationary background counts were recorded on each side of the reflection. Crystal and diffractometer stability was checked by monitoring three standard reflections every 150 reflections. Only random deviations (±4.3%) were observed over the course of data collection. No decay correction was applied. Empirical absorption correction, based on ψ scans, was applied with correction factors ranging from 0.84 to 1.00. The data were also corrected for Lorentz and polarization effects. The structure was solved by direct methods and Fourier synthesis, both of which are part of the *TEXSAN* program package (Molecular Structure Corporation, 1989) which showed an octahedral cluster sitting about a crystallographic center of inversion. H atoms were included in calculated positions.

During the final stages of refinement the positional and anisotropic thermal parameters of all non-H atoms were refined.* The final conventional agreement factors were $R = 0.033$, $wR = 0.036$ and $S = 1.22$ for the 5134 observed reflections and 487 variables. The function minimized was $\sum w(F_o - F_c)^2$ where $w = 1/\sigma^2(F_o)$ with $\sigma(F_o)$ from counting statistics. The maximum shift/e.s.d. in the last full-matrix least-squares cycle was less than 0.0004. The final difference Fourier map showed no peaks higher than 0.30 or deeper than -0.20 e Å^{-3}. All programs used in the structure solution and refinement are contained in the *TEXSAN* package (Molecular Structure Corporation, 1989). The plot was made using *ORTEP* (Johnson, 1976). The atomic scattering factors included with the software package are from *International Tables for X-ray Crystallography* (1974, Vol. IV). The final positional and displacement parameters for the metal anion are provided in Table 1. Table 2 contains selected bond geometry. Fig. 1 illustrates the metal coordination and the numbering scheme. Fig. 2 shows the unit-cell packing.

Related literature. A report of the tetramethylammonium salt has already appeared (Calabrese *et al.*, 1974).

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters, full geometry and an *ORTEP* plot of the counter-anion have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55923 (72 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HH1042]

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Structure of 17β-Acetoxy-18-methyl-1-thia-*A*-norestra-3,9(10),11-trien-2-one

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Abstract. 18-Methyl-2-oxo-1-thia-*A*-norestra-3,9(10),-11-trien-17β-yl acetate, $C_{19}H_{22}O_3S$, $M_r = 330.44$, triclinic, $P\bar{1}$, $a = 9.1267$ (12), $b = 11.955$ (1), $c = 8.783$ (1) Å, $\alpha = 93.08$ (1), $\beta = 117.70$ (1), $\gamma = 85.49$ (1)°, $V = 845.7$ (1) Å^3, $Z = 2$, $D_x = 1.298$ g cm^{-3}, λ(Mo $K\alpha$) = 0.7107 Å, $\mu = 1.94$ cm^{-1}, $F(000) = 352$, $T = 295$ K, $R = 0.054$, $wR = 0.047$ for 2920 observed reflections having $I \geq \sigma(I)$ (of 3632

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