Dahl, 1970), $[Mo_2(\eta\text{-}C_5H_4CHPh_2)_2(CO)_6]$, 3·227 Å (Drews & Behrens, 1985), $[Mo_2(\eta\text{-}C_5H_4CH_2CH_2CH_2OH)_2(CO)_6]$, 3·213 Å (Coolbaugh, Coots, Santarsiero & Grubbs, 1985), $[Mo_2(\eta\text{-}C_5H_5)_2(CH_3NC)(CO)_5]$, 3·230 Å (Adams, Brice & Cotton, 1973), and $[Mo_2(\eta\text{-}C_5H_5)_2\{C(C_6H_4CH_3)_2\}\{N_2C(C_6H_4CH_3)_2\}(CO)_3]$, 3·052 Å (Messerle & Curtis, 1982). The bond length in $[Mo_2(\eta\text{-}C_5Me_5)_2(CO)_6]$ is 3·281 (1) Å and is the longest unsupported Mo–Mo single bond so far observed. Additional features of interest are the *trans* disposition of the C_5Me_5 ligands and the slight bending at carbon in the four carbonyls *cis* to the Mo–Mo bond. Both features are observed in the parent compound, $[Mo_2(\eta\text{-}C_5H_5)_2(CO)_6]$ (Adams, Collins & Cotton, 1974).

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Di-μ-carbonyl-nonacarbonyl-μ_4-(α–α′-η-diphenylacetylene)-(μ_4-diphenylnitrene)-tetraruthenium(4 *Ru–Ru*)

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Abstract. $[Ru_4(C_{14}H_{10})(C_6H_5N)(CO)_{11}]$, $M_r = 981{\cdot}71$, monoclinic, $P2_1/c$, $a = 16{\cdot}315$ (3), $b = 20{\cdot}037$ (4), $c = 19{\cdot}749$ (4) Å, $\beta = 95{\cdot}75$ (1)°, $V = 6424$ (2) Å^3, $Z = 8$, 2 independent molecules/asymmetric unit, $D_x = 2{\cdot}030$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 18{\cdot}70$ cm^{-1}, $F(000) = 3680$, $T = 296$ K, $R_F = 2{\cdot}99\%$ for 7583 observed reflections and 776 parameters. The structure is the phenyl analog of a previously reported (μ_4-NH)-(diphenylacetylene)tetraruthenium cluster. The two independent molecules are chemically indistinguishable. The structure is a pentagonal bipyramid with an equatorial plane containing two Ru atoms, the acetylene residue and the nitrene N atom. This plane is capped by two $Ru(CO)_2$ units with two edge-bridging carbonyl groups.

Experimental. Orange crystals from hexane (0·41 × 0·41 × 0·41 mm); Nicolet *R*3*m* diffractometer with graphite monochromator; ω scans; lattice parameters from least-squares fit of 25 reflections ($30 \le 2\theta \le 35°$); no absorption correction ($\mu = 18{\cdot}7$ cm^{-1}, uniform crystal shape, $T_{max}/T_{min} = 1{\cdot}11$); $2\theta_{max} = 48°$ ($h = \pm 19$, $k = +23$, $l = +23$); standard reflections 12,0,2, $\bar{6}$,15,1, $\bar{5}$,4,14. 10 933 reflections collected, 10 090

Table 1. *Atomic coordinates* (×10^4) *and equivalent isotropic thermal parameters* (Å^2×10^3)

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

	x	y	z	U_{eq}
Ru(1)	2285·1 (3)	5232·5 (2)	4721·1 (2)	33·4 (1)
Ru(2)	3160·3 (3)	6192·3 (2)	4060·3 (2)	32·1 (1)
Ru(3)	1223·9 (3)	6261·2 (2)	4804·5 (2)	30·6 (1)
Ru(4)	2310·9 (3)	7214·8 (2)	4555·0 (2)	29·9 (1)
Ru(1′)	3256·6 (2)	4628·6 (2)	8647·6 (2)	28·7 (1)
Ru(2′)	4034·6 (3)	3731·5 (2)	9517·2 (2)	29·6 (1)
Ru(3′)	3095·1 (3)	2684·7 (2)	9092·7 (2)	28·4 (1)
Ru(4′)	2045·3 (2)	3629·1 (2)	8542·7 (2)	28·1 (1)

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Table 1 (*cont.*)

	x	y	z	U_{eq}
C(1)	2435 (4)	4874 (3)	5645 (3)	49 (2)
C(2)	3231 (4)	4784 (3)	4464 (3)	48 (2)
C(3)	1579 (4)	4497 (3)	4436 (3)	43 (2)
C(4)	3480 (4)	6032 (3)	3185 (3)	48 (2)
C(5)	4274 (4)	5998 (3)	4342 (3)	49 (2)
C(6)	3362 (3)	7200 (3)	4023 (3)	39 (2)
C(7)	937 (4)	5886 (3)	5619 (3)	47 (2)
C(8)	105 (4)	6174 (3)	4491 (3)	48 (2)
C(9)	1115 (4)	7232 (3)	5038 (3)	39 (2)
C(10)	1981 (3)	7989 (3)	4058 (3)	42 (2)
C(11)	2850 (4)	7765 (3)	5264 (3)	46 (2)
C(12)	1765 (3)	6483 (3)	3797 (2)	28 (2)
C(13)	659 (2)	7195 (2)	3189 (1)	44 (2)
C(14)	307	7530	2612	51 (2)
C(15)	670	7490	2004	49 (2)
C(16)	1385	7114	1971	49 (2)
C(17)	1737	6779	2548	40 (2)
C(18)	1374	6820	3157	32 (2)
C(19)	1796 (3)	5773 (3)	3832 (3)	32 (2)
C(20)	594 (2)	5388 (2)	3033 (2)	48 (2)
C(21)	257	4977	2506	63 (3)
C(22)	760	4532	2195	71 (3)
C(23)	1600	4498	2411	63 (3)
C(24)	1937	4909	2938	48 (2)
C(25)	1433	5354	3248	33 (2)
C(26)	2595 (2)	6581 (2)	6250 (2)	49 (2)
C(27)	2991	6615	6909	63 (3)
C(28)	3788	6364	7047	67 (3)
C(29)	4189	6079	6526	73 (3)
C(30)	3793	6045	5867	58 (2)
C(31)	2996	6296	5729	35 (2)
C(1')	2417 (4)	4997 (3)	8038 (3)	46 (2)
C(2')	4115 (4)	4858 (3)	8056 (3)	43 (2)
C(3')	3415 (3)	5459 (3)	9117 (3)	37 (2)
C(4')	4443 (3)	3968 (3)	10419 (3)	44 (2)
C(5')	5034 (3)	4053 (3)	9237 (3)	44 (2)
C(6')	4368 (4)	2780 (3)	9655 (3)	41 (2)
C(7')	1619 (3)	3678 (3)	7620 (3)	39 (2)
C(8')	954 (3)	3774 (3)	8707 (3)	41 (2)
C(9')	1927 (3)	2623 (3)	8549 (3)	35 (2)
C(10')	2812 (4)	2037 (3)	9718 (3)	39 (2)
C(11')	3452 (4)	1983 (3)	8527 (3)	40 (2)
C(12')	2666 (3)	3549 (3)	9656 (3)	29 (2)
C(13')	1445 (2)	3312 (2)	10308 (2)	57 (2)
C(14')	1106	3113	10897	75 (3)
C(15')	1620	2926	11473	64 (3)
C(16')	2473	2939	11461	61 (3)
C(17')	2812	3139	10872	47 (2)
C(18')	2298	3325	10296	32 (2)
C(19')	2682 (3)	4230 (2)	9489 (3)	29 (2)
C(20')	1575 (2)	5059 (2)	9670 (2)	53 (2)
C(21')	1205	5520	10073	71 (3)
C(22')	1553	5653	10733	74 (3)
C(23')	2273	5326	10990	62 (3)
C(24')	2643	4865	10588	47 (2)
C(25')	2294	4732	9927	36 (2)
C(26')	4551 (2)	3023 (2)	7967 (1)	43 (2)
C(27')	4925	2847	7388	52 (2)
C(28')	4571	3038	6745	53 (2)
C(29')	3841	3406	6681	51 (2)
C(30')	3467	3583	7260	41 (2)
C(31')	3821	3392	7903	29 (2)
O(1)	2496 (3)	4668 (3)	6185 (2)	77 (2)
O(2)	3752 (3)	4454 (2)	4332 (3)	69 (2)
O(3)	1143 (3)	4077 (2)	4263 (2)	61 (2)
O(4)	3685 (3)	5915 (3)	2670 (2)	72 (2)
O(5)	4947 (3)	5871 (3)	4461 (3)	69 (2)
O(6)	3788 (3)	7585 (2)	3808 (2)	60 (2)
O(7)	761 (3)	5663 (3)	6118 (2)	76 (2)
O(8)	−584 (3)	6120 (3)	4315 (2)	76 (2)
O(9)	723 (3)	7648 (2)	5261 (2)	56 (2)
O(10)	1796 (3)	8459 (2)	3762 (3)	69 (2)
O(11)	3196 (3)	8068 (2)	5675 (2)	75 (2)
O(1')	1934 (3)	5281 (2)	7683 (3)	69 (2)
O(2')	4631 (3)	4976 (2)	7742 (2)	66 (2)
O(3')	3501 (3)	5950 (2)	9398 (2)	57 (2)
O(4')	4700 (3)	4111 (3)	10949 (2)	73 (2)
O(5')	5634 (3)	4246 (3)	9063 (3)	70 (2)
O(6')	4868 (3)	2391 (2)	9854 (2)	60 (2)
O(7')	1315 (3)	3687 (2)	7080 (2)	62 (2)
O(8')	273 (3)	3860 (3)	8784 (3)	70 (2)
O(9')	1470 (3)	2199 (2)	8363 (3)	64 (2)
O(10')	2645 (3)	1644 (2)	10094 (2)	58 (2)
O(11')	3618 (3)	1573 (2)	8177 (2)	69 (2)
N	2569 (3)	6240 (2)	5039 (2)	33 (1)
N'	3407 (2)	3569 (2)	8498 (2)	27 (1)

Table 2. *Selected bond lengths* (Å)

Ru(1)–Ru(2)	2·795 (1)	Ru(1)–Ru(3)	2·707 (1)
Ru(1)–C(1)	1·953 (6)	Ru(1)–C(2)	1·899 (6)
Ru(1)–C(3)	1·920 (6)	Ru(1)–C(19)	2·148 (5)
Ru(1)–N	2·152 (4)	Ru(2)–Ru(4)	2·710 (1)
Ru(2)–C(4)	1·882 (6)	Ru(2)–C(5)	1·887 (6)
Ru(2)–C(6)	2·049 (5)	Ru(2)–C(12)	2·357 (5)
Ru(2)–C(19)	2·379 (5)	Ru(2)–N	2·247 (4)
Ru(3)–Ru(4)	2·686 (1)	Ru(3)–C(7)	1·876 (6)
Ru(3)–C(8)	1·876 (6)	Ru(3)–C(9)	2·011 (6)
Ru(3)–C(12)	2·299 (5)	Ru(3)–C(19)	2·425 (5)
Ru(3)–N	2·197 (4)	Ru(4)–C(6)	2·099 (6)
Ru(4)–C(9)	2·257 (6)	Ru(4)–C(10)	1·884 (6)
Ru(4)–C(11)	1·924 (6)	Ru(4)–C(12)	2·218 (5)
Ru(4)–N	2·196 (4)	Ru(1')–Ru(2')	2·712 (1)
Ru(1')–Ru(4')	2·807 (1)	Ru(1')–C(1')	1·880 (6)
Ru(1')–C(2')	1·966 (6)	Ru(1')–C(3')	1·910 (5)
Ru(1')–C(19')	2·142 (5)	Ru(1')–N'	2·161 (4)
Ru(2')–Ru(3')	2·682 (1)	Ru(2')–C(4')	1·898 (6)
Ru(2')–C(5')	1·886 (6)	Ru(2')–C(6')	1·994 (6)
Ru(2')–C(12')	2·305 (5)	Ru(2')–C(19')	2·418 (5)
Ru(2')–N'	2·189 (4)	Ru(3')–Ru(4')	2·706 (1)
Ru(3')–C(6')	2·263 (6)	Ru(3')–C(9')	2·096 (5)
Ru(3')–C(10')	1·879 (6)	Ru(3')–C(11')	1·923 (6)
Ru(3')–C(12')	2·210 (5)	Ru(3')–N'	2·212 (4)
Ru(4')–C(7')	1·886 (6)	Ru(4')–C(8')	1·864 (6)
Ru(4')–C(9')	2·025 (5)	Ru(4')–C(12')	2·332 (5)
Ru(4')–C(19')	2·374 (5)	Ru(4')–N'	2·235 (4)

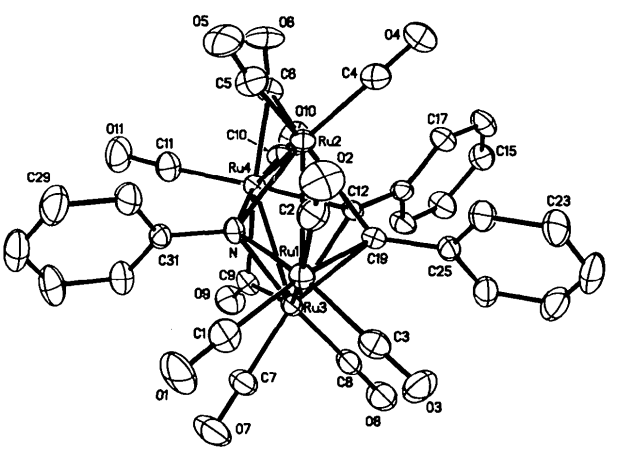

Fig. 1. Molecular structure and labeling scheme for $[Ru_4(NPh)(PhC_2Ph)(CO)_{11}]$. One of two chemically identical but crystallographically independent molecules is shown.

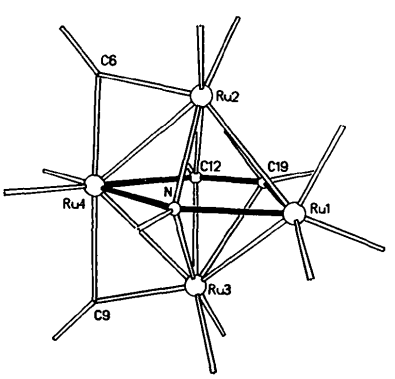

Fig. 2. Cluster framework for $[Ru_4(NPh)(PhC_2Ph)(CO)_{11}]$ emphasizing the equatorial plane of the pentagonal bipyramidal structure.

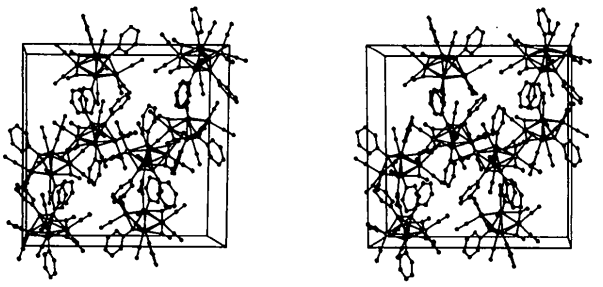

Fig. 3. Unit-cell packing diagram as viewed down the *a* axis.

unique ($R_{int} = 2{\cdot}31\%$), 7583 observed with $F_o > 6\sigma(F_o)$, 2507 unobserved reflections. Direct-methods (*SOLV*) structure solution; least-squares refinement on 776 parameters; all non-H atoms anisotropic, H atoms idealized and updated (C—H = 0·96 Å, $U = 1{\cdot}2\ U$ of attached C); phenyl rings rigid planar hexagons (C—C = 1·395 Å). $R_F = 2{\cdot}99\%$, $wR_F = 4{\cdot}13\%$, $S = 0{\cdot}955$, $w^{-1} = \sigma^2(F_o) + gF_o^2$, $g = 0{\cdot}001$; $(\Delta/\sigma)_{max} = 0{\cdot}733$; $\Delta\rho_{max} = 0{\cdot}47$, $\Delta\rho_{min} = -0{\cdot}45$ e Å^{-3}; atomic scattering factors from *International Tables for X-ray Crystallography* (1974); *SHELXTL* computer program (Sheldrick, 1984).

Atomic and equivalent isotropic thermal parameters are given in Table 1 and selected bond lengths in Table 2. Fig. 1 shows the labeled molecular structure of the compound and Fig. 2 the tetraruthenium cluster framework with bridging CO groups. A unit-cell packing diagram is shown in Fig. 3.*

Related literature. The title compound is the phenyl analog of a structure previously reported by Blohm & Gladfelter (1986).

* Lists of structure factors, anisotropic thermal parameters and full lists of bond lengths and angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44518 (70 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Bis(*N*,*N*-dimethylformamide)[*o*-(*N*-methyliminomethyl)phenyl]palladium(II) Tetrafluoroborate

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Abstract. $[PdC_6H_4(CH_2NCH_3)(C_3H_7NO)_2]^+.BF_4^-$, $M_r = 458{\cdot}5$, triclinic, $P\bar{1}$, $a = 8{\cdot}098$ (2), $b = 9{\cdot}934$ (3), $c = 12{\cdot}860$ (2) Å, $\alpha = 67{\cdot}46$ (2), $\beta = 85{\cdot}41$ (2), $\gamma = 80{\cdot}22$ (2)°, $V = 941{\cdot}5$ (4) Å^3, $Z = 2$, $D_x = 1{\cdot}617$ g cm^{-3}, $\mu = 10{\cdot}08$ cm^{-1} (Mo), λ(Mo $K\alpha$) = 0·71073 Å, $F(000) = 462$, $T = 296$ K, $R_F = 5{\cdot}01\%$ for 3141 observed reflections and 227 parameters. The Pd^{II} coordination environment is nearly square planar with the Pd atom 0·08 Å above the plane. Pd—N 2·018 (5), Pd—C 1·949 (5), Pd—O 2·077 (4) and 2·178 (4) Å. The molecules associate as invertamer pairs with Pd···Pd = 3·488 (1) Å.

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Experimental. Yellow crystals of (1) from chloroform and diethyl ether, 0·3 × 0·3 × 0·4 mm, Nicolet *R*3*m* diffractometer with graphite monochromator, Wyckoff scans, lattice parameters from least-squares fit of 21 reflections ($23 \le 2\theta \le 33°$), absorption correction unnecessary ($\mu = 10{\cdot}08$ cm^{-1}, uniform crystal shape), $2\theta_{max} = 52°$ ($h = \pm 10$, $k = \pm 13$, $l = +16$), standard reflections 500, 250 and 129, variation ≤ 2%. 3783 reflections collected, 3554 unique, $R_{int} = 2{\cdot}67\%$, 3141 observed with $F_o \ge 3\sigma(F_o)$, 369 unobserved reflections, sharpened Patterson structure solution, least-squares refinement on 227 parameters, all non-hydrogen atoms anisotropic, H atoms idealized and updated [C—H = 0·96 Å, $U = 1{\cdot}2\ U$ of attached C], BF_4^- geometry