Synthesis and Characterization of Bis(η^2-alkyne)dihalogenomercury(II) Compounds: Crystal Structure of $[NBu_4]_2[\{cis\text{-}Pt(C_6F_5)_2(C{\equiv}CSiMe_3)_2\}HgBr_2]\cdot CH_2Cl_2$†

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The reactions of $[NBu_4]_2[cis\text{-}Pt(C_6F_5)_2(C{\equiv}CR)_2]$ (R = Bu^t or $SiMe_3$) with HgX_2 (X = Cl, Br or I) in a 1:1 molar ratio afforded the simple monomeric bis(η^2-alkyne)mercury(II) compounds $[NBu_4]_2[\{cis\text{-}Pt(C_6F_5)_2(C{\equiv}CR)_2\}HgX_2]$ (R = Bu^t or $SiMe_3$; X = Cl, Br or I) in which both alkyne units are co-ordinated η^2 side-on to the precursor mercury(II) halides. Similar treatment of $[NBu_4]_2[Pt(C{\equiv}CR)_4]\cdot 2H_2O$ (R = Bu^t or $SiMe_3$) with HgX_2 in a 1:2 molar ratio gave the corresponding trinuclear 1:2 adducts. All the complexes have been characterized by analytical and spectroscopic data and, in addition, the molecular structure of $[NBu_4]_2[\{cis\text{-}Pt(C_6F_5)_2(C{\equiv}CSiMe_3)_2\}HgBr_2]\cdot CH_2Cl_2$ has been determined by X-ray diffraction methods. The structure of the anion shows that the $HgBr_2$ unit is attached to the dianionic fragment $[Pt(C_6F_5)_2(C{\equiv}CSiMe_3)_2]^{2-}$ only through η^2 side-on co-ordination of the two (trimethylsilyl)ethynyl ligands. The platinum–mercury distance is 3.627(1.5) Å.

Compounds containing one or more mercury atoms bonded to one low-valent transition-metal centre were among the first heterometallic species to be reported,[1] and since then a large number of such complexes have been described.[2,3] Many of the preparative reactions reported involved the treatment of mercury(II) halides with nucleophilic metal reagents such as low-valent metal complexes or mono- or poly-nuclear carbonylmetalate anions and led to: (*i*) retention of both halide atoms bonded to mercury to produce simple Lewis-base adducts,[2,3d,3g,4] (*ii*) displacement of one halide ion to yield complexes containing the very versatile HgX unit[2,3c,3g,3j,5] or (*iii*) complete displacement of both halide ions with the formation of a cluster in which mercury only forms Hg–M bonds.[2,6] To our knowledge there have been no reports of similar reactions using homoleptic or mixed σ-alkynyl anionic complexes.

We have recently found that the readily accessible homoleptic $[Pt(C{\equiv}CR)_4]^2$ and mixed $cis\text{-}[Pt(C_6F_5)_2(C{\equiv}CR)_2]^{2-}$ σ-alkynyl anionic substrates react with transition-metal Lewis acids to give heteropolynuclear platinum acetylide complexes.[7] The crystal structures of some of the resulting complexes show that the metal centres are connected by the alkynyl ligands which are σ-bonded to the platinum atoms and π bonded side-on to the silver atoms, thus suggesting that the driving force in the formation of these compounds are the π metal M–alkyne bonds.

Following on from our interest in polynuclear complexes in which the metal centres are only connected by bridging alkynyl ligands,[7,8] and with the aim of synthesising mixed platinum–mercury compounds, we decided to study the reactivity of the anionic complexes $[NBu_4]_2[Pt(C{\equiv}CR)_4]$ and $[NBu_4]_2[cis\text{-}Pt(C_6F_5)_2(C{\equiv}CR)_2]$ towards mercury dihalides. Our interest was also stimulated by the fact that, as far as we knew, no complexes containing a π-mercury–alkyne moiety had been reported[9a] and furthermore the reactivity of σ-alkynyl complexes towards HgX_2 had been scarcely explored.[9] In this paper we report the successful synthesis of a variety of di- and tri-nuclear platinum–mercury complexes, $[NBu_4]_2[\{cis\text{-}Pt(C_6F_5)_2(C{\equiv}CR)_2\}HgX_2]$ and $[NBu_4]_2[\{Pt(C{\equiv}CR)_4\}(HgX_2)_2]$ (R = Bu^t or $SiMe_3$; X = Cl, Br or I), which have been obtained upon treating $[NBu_4]_2[cis\text{-}Pt(C_6F_5)_2(C{\equiv}CR)_2]$ or $[NBu_4]_2[Pt(C{\equiv}CR)_4]\cdot 2H_2O$ (R = Bu^t or $SiMe_3$) with HgX_2 (X = Cl, Br or I) in a 1:1 or 1:2 molar ratio respectively. The molecular structure of one of the binuclear complexes has also been determined by X-ray crystallography.

Results

The compounds $[NBu_4]_2[cis\text{-}Pt(C_6F_5)_2(C{\equiv}CR)_2]$ (R = Bu^t or $SiMe_3$) react with 1 equivalent of HgX_2 (X = Cl, Br or I) in acetone, at room temperature, to give the corresponding 1:1 adducts $[NBu_4]_2[\{cis\text{-}Pt(C_6F_5)_2(C{\equiv}CR)_2\}HgX_2]$ **1**–**6** in high yield. Similarly, the treatment of $[NBu_4]_2[Pt(C{\equiv}CR)_4]\cdot 2H_2O$ (R = Bu^t or $SiMe_3$) with 2 equivalents of HgX_2 led to the isolation of the corresponding 1:2 adducts $[NBu_4]_2[Pt(C{\equiv}CR)_4(HgX_2)_2]\cdot 2H_2O$ **7**–**12** albeit in lower yields. Both processes are shown in Scheme 1.

All the complexes were isolated as white solids and characterised by microanalysis, conductivity measurements and by IR and NMR (^1H, ^{19}F) spectroscopy (Table 1). The way in which the HgX_2 unit is attached to the $cis\text{-}[Pt(C_6F_5)_2(C{\equiv}CR)_2]^{2-}$ dianionic fragment in the binuclear complexes **1**–**6** was established from the crystal structure determination of compound **4**. The structure of the anion showing the atom numbering scheme is shown in Fig. 1. Selected bond lengths and angles are collected in Table 2. Examination of Fig. 1 shows that each of the two (trimethylsilyl)ethynyl ligands of the $cis\text{-}[Pt(C_6F_5)_2(C{\equiv}CSiMe_3)_2]^{2-}$ dianionic fragment are η^2 co-ordinated to the mercury atom. The two bromine atoms complete a distorted tetrahedral co-ordination environment for the mercury atom, within which the angles vary from 89.21 [midpoint C(13), C(14)–Hg–midpoint C(18),(19)] to 118.34° [Br(1)–Hg–midpoint C(13), (14)].

The platinum atom is located in a slightly distorted square-planar environment formed by the two *ipso* C atoms of the pentafluorophenyl groups (mutually *cis*) and both C_α atoms of

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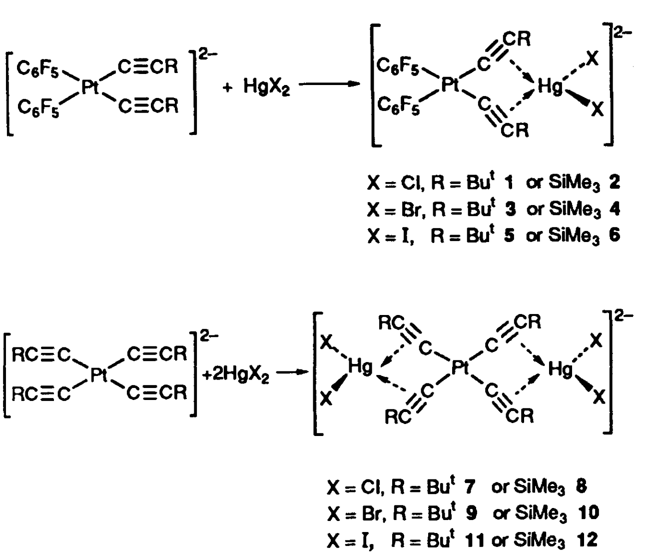
† *Supplementary data available:* see Instructions for Authors, *J. Chem. Soc., Dalton Trans.*, 1994, Issue 1, pp. xxiii–xxviii.

Table 1 Elemental analyses (%), conductivity, IR, ^1H and ^{19}F NMR ($CDCl_3$, δ in ppm, *J* in Hz) of complexes **1–12**

	Analysis[a]								^{19}F		
Complex	C	H	N	Λ_m[b]	$\nu(C\equiv C)$[c]/cm^{-1}	$\nu(C\equiv C)$[d]/cm^{-1}	$\nu(C_6F_5)$/cm^{-1}	$^1H(R)$[e]	F_o[f]	F_p	F_m
1	46.35 (46.45)	6.40 (6.25)	1.90 (1.95)	187.92[g]	2031s	2024w	786s 777s	1.19 (s)	−114.8 (d) [396]	−166.1 (m)	−166.7 (m)
2	43.55 (43.80)	6.65 (6.15)	1.90 (1.90)	181.97[g]	1958vs 1933s	1957m 1943w	787s 778s	0.16 (s)	−115.3 (d) [374]	−166.0 (t)	−167.2 (m)
3	43.70 (43.75)	6.00 (5.90)	1.90 (1.80)	199.35[g]	2034m	2030w	786s 776s	1.18 (s)	−115.7 (d) [409]	−167.1 (t)	−167.7 (m)
4	41.30 (41.35)	5.80 (5.80)	1.80 (1.80)	191.14[g]	1956vs 1933s	1958m 1932w	786s 778s	0.16 (s)	−115.1 (d) [378]	−166.0 (t)	−167.2 (m)
5	41.00 (41.25)	5.50 (5.55)	1.75 (1.70)	210.14[g]	*h*	2037w		1.16 (s)	−115.6 (d) [409]	−167.3 (t)	−167.9 (m)
6	39.50 (39.00)	6.20 (5.45)	1.65 (1.70)	247.70[g]	*h*	1959m 1937w		0.14 (s)	−114.8 (d) [379]	−166.2 (t)	−167.2 (m)
7	43.30 (43.45)	7.50 (7.00)	1.85 (1.80)	243.29[i]	2036vs	2036w		1.27 (s)			
8	38.75 (38.75)	8.00 (6.75)	1.75 (1.75)	220.57[i]	1964vs, br	1965s		0.21 (s)			
9	38.75 (39.00)	6.30 (6.30)	1.65 (1.60)	221.73[i]	2036vs 2025vs	2038m		1.27 (s)			
10	34.90 (34.90)	6.30 (6.10)	1.55 (1.55)	225.09[i]	1964vs, br	1964s		0.22 (s)			
11	35.00 (35.15)	5.80 (5.70)	1.40 (1.45)	220.78[i]	2048vs	2038w		1.25 (s)			
12	31.65 (31.60)	5.65 (5.50)	1.35 (1.40)	204.95[i]	1963vs, br	1967s		0.19 (s)			

[a] Calculated values in parentheses. [b] ohm^{-1} cm^2 mol^{-1}. [c] In Nujol mulls between polyethylene sheets; $\nu(C\equiv C)$ for $[NBu_4]_2[Pt(C_6F_5)_2(C\equiv CR)_2]$ (R = Bu^t 2090, 2084 cm^{-1}; R = $SiMe_3$ 2028, 2009 cm^{-1}) and $[NBu_4][Pt(C\equiv CR)_4]\cdot 2H_2O$ (R = Bu^t 2080 cm^{-1}; R = $SiMe_3$ 2015, 2009 cm^{-1}) derivatives. [d] In CH_2Cl_2 solution. [e] All complexes showed signals of $[NBu_4]^+$ cation at δ *ca.* 0.9 (CH_3), 1.33 (α-CH_2), 1.6 (β-CH_2) and 3.35 (γ-CH_2) in the expected ratio. [f] $^3J(Pt\text{-}F_o)$ in square brackets. [g] In Me_2CO. [h] It was not possible to mix the crude solid with Nujol. [i] In MeCN.



X = Cl, R = Bu^t **1** or SiMe$_3$ **2**
X = Br, R = Bu^t **3** or SiMe$_3$ **4**
X = I, R = Bu^t **5** or SiMe$_3$ **6**

X = Cl, R = Bu^t **7** or SiMe$_3$ **8**
X = Br, R = Bu^t **9** or SiMe$_3$ **10**
X = I, R = Bu^t **11** or SiMe$_3$ **12**

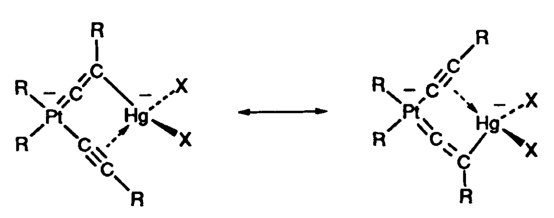
Scheme 1

Table 2 Selected bond lengths (Å) and angles (°) for $[NBu_4]_2[\{$*cis*-$Pt(C_6F_5)_2(C{\equiv}CSiMe_3)_2\}HgBr_2]{\cdot}CH_2Cl_2$ **4**

Hg–C(14)	2.41(2)	Hg–C(19)	2.46(2)
Hg–Br(2)	2.572(3)	Hg–C(18)	2.57(2)
Hg–Br(1)	2.588(3)	Hg–C(13)	2.64(2)
Pt–C(13)	1.95(3)	Pt–C(18)	1.98(3)
Pt–C(1)	2.06(2)	Pt–C(7)	2.10(3)
C(13)–C(14)	1.21(3)	C(14)–Si(1)	1.92(3)
C(18)–C(19)	1.19(3)	C(19)–Si(2)	1.87(3)
C(14)–Hg–Br(2)	103.3(6)	C(19)–Hg–Br(2)	111.2(6)
Br(2)–Hg–C(18)	119.2(6)	Br(2)–Hg–Br(1)	113.01(10)
C(14)–Hg–C(13)	27.3(7)	Br(2)–Hg–C(13)	115.4(5)
C(18)–Hg–C(13)	63.1(8)	Br(1)–Hg–C(13)	122.3(5)
C(13)–Pt–C(18)	88.0(9)	C(13)–Pt–C(1)	93.9(9)
C(13)–Pt–C(7)	178.1(9)	C(18)–Pt–C(7)	90.3(10)
C(14)–C(13)–Pt	168(2)	C(14)–C(13)–Hg	66(2)
Pt–C(13)–Hg	103.4(9)	C(13)–C(14)–Si(1)	156(2)
C(19)–C(18)–Pt	176(2)	C(19)–C(18)–Hg	71(2)
Pt–C(18)–Hg	104.9(10)	C(18)–C(19)–Si(2)	160(2)

the acetylide ligands. The HgBr(1)Br(2) fragment lies almost perpendicular to the co-ordination plane of the platinum atom, the interplanar angle being 84.91°. The long Pt–Hg distance [3.627(1.5) Å], out of the range of values found in dinuclear complexes with Pt–Hg bonds (2.51–2.83 Å),[3b,m] and the position of the mercury atom between the alkynyl bridging system, clearly indicates that both metal centres are connected only through the double alkynyl bridging system. The central PtC(13)C(14)C(18)C(19)Hg core of the resulting metallacycle is almost planar (maximum deviation 0.076 Å for the Hg atom). This contrasts with the non-planar PtC_4M core previously found in similar binuclear systems such as $[\{Pt(Ph_2PCH_2CH_2PPh_2)(C{\equiv}CPh)_2\}Pt(C_6F_5)_2]$[8a] and $[\{Pt(C_6F_5)_2(C{\equiv}CSiMe_3)_2\}Pd(C_3H_5)]^-$.[10] Similar planar M (acetylides)M′ cores have been found in related systems such as $[(C_5H_4SiMe_3)_2Ti(C{\equiv}CSiMe_3)_2ML_n]$ (ML_n = CuCl, $FeCl_2$[11] or $AgNO_2$[12]) and $[\{Ti(C_5HMe_4)_2(C{\equiv}CSiMe_3)_2\}MgCl(thf)]$ (thf = tetrahydrofuran).[13]

The Pt–C$_\alpha$[Pt–C(13) 1.95(3), Pt–C(18) 1.98(3) Å] and C≡C bond lengths [C(13)–C(14) 1.21(3), C(18)–C(19) 1.19(3) Å] are quite short but they lie within the ranges found for other σ-alkynyl platinum complexes and μ-η^2-alkynyl bridging ligands.[7,8] Although the C≡C distances in **4** are not very different to those observed in complexes with terminal acetylide ligands,[9b] the IR spectra of complexes **1**–**6** (see Table 1) show that the ν(C≡C) absorptions are shifted to lower wavelengths



Scheme 2

than those observed for the corresponding starting materials $[NBu_4]_2[$*cis*-$Pt(C_6F_5)_2(C{\equiv}CR)_2]$ (see footnote *c*, Table 1) as expected for carbon–carbon triple bonds co-ordinated side-on to a transition-metal centre.[7,8,9b] The η^2 mercury–alkynide linkages are clearly asymmetric, the Hg–C$_\beta$ distances [Hg–C(14) 2.41(2), Hg–C(19) 2.46(2) Å] being shorter than the corresponding Hg–C$_\alpha$ distances [Hg–C(13) 2.64(2), Hg–C(18) 2.57(2) Å]. Although this structural feature is not usual in side-on co-ordinated alkynyl ligands, it has been observed previously[14] and implies some degree of participation of the formal structures shown in Scheme 2. The distance between the mercury atom and the midpoints of the C≡C triple bonds [Hg–C(13),C(14) 2.4550(12), Hg–C(18),C(19) 2.4552(10) Å] are identical and shorter than those found in $[\{Hg(C_6Me_6)(CF_3CO_2)_2\}_2]$ (Hg–C 2.56 and 2.57 Å).[15]

As a consequence of the η^2 co-ordination of the alkynyl ligands to mercury, these groups are distorted from linearity. The angles Pt–C(13)–C(14) 168(2), Pt–C(18)–C(19) 176(2), C(13)–C(14)–Si(1) 156(2) and C(18)–C(19)–Si(2) 160(2)° are similar to those found in related systems.[8a,10–13] On the other hand, the angles formed by the C≡C triple bonds and the corresponding vectors defined by mercury and the midpoints of the C≡C bonds are 101.41(14) and 95.57(1.57)° respectively, and the C≡C triple bonds are inclined by 53.5 [C(13),C(14)] and 126.18° [C(18),C(19)] to the normal to the HgBr(1)Br(2) plane.

Although we have not been able to grow adequate crystals of complexes $[NBu_4]_2[\{Pt(C{\equiv}CR)_4\}(HgX_2)_2]$ **7**–**12** for X-ray crystallography, it seems sensible to assume that the anions are trinuclear species with the dianionic fragment chelating two neutral HgX_2 units. This formulation is also in good agreement with their IR [ν(C≡C) 1963–2048 cm^{-1}] and ^1H NMR spectra (Table 1). It is noteworthy that these 1:1 (**1**–**6**) or 1:2 (**7**–**12**) adducts are not significantly dissociated in solution. In fact, no noticeable changes in the corresponding ν(C≡C) absorptions were observed in the IR spectra of the complexes in dichloromethane solutions (Table 1), suggesting that the η^2-alkynyl to mercury interactions remain in this solvent.

Reactions with cis-$[Pt(C_6F_5)_2(thf)_2]$.—With the aim of preparing complexes of higher nuclearity we explored the reactivity of some of the complexes towards *cis*-$[Pt(C_6F_5)_2(thf)_2]$, since the latter has been proven to be an excellent synthon for the preparation of polynuclear complexes containing the *cis*-$Pt(C_6F_5)_2$ moiety.[8,16]

However, treatment of the bimetallic **1** and **2** and trimetallic **7** and **8** derivatives with 1 or 2 equivalents of *cis*-$[Pt(C_6F_5)_2(thf)_2]$ in acetone did not render the expected tri- PtHgPt or penta-nuclear PtHgPtHgPt compounds. The reactions gave the binuclear derivative $[NBu_4]_2[Pt_2(\mu\text{-}Cl)_2(C_6F_5)_4]$[17] (see Experimental section) which can be separated after evaporating to dryness and adding EtOH. Unfortunately, from the mother-liquors only intractable oils can be obtained.

Discussion

The formation of these 1:1 complexes (**1**–**6**) and 1:2 (**7**–**12**) adducts is surprising for several reasons.

First, the interaction of mercury with π systems has been reported only rarely.[9,15,18] It is well known that alkynes (and

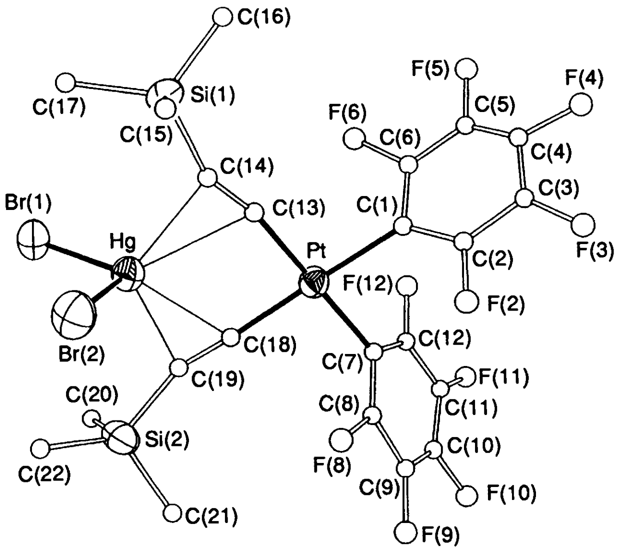


Fig. 1 Structure of the $[\{cis\text{-Pt}(C_6F_5)_2(\mu\text{-C}\equiv\text{CSiMe}_3)_2\}\text{HgBr}_2]^{2-}$ anion of complex **4** showing the atom-labelling scheme

also olefins) react with electrophilic mercury salts yielding adducts in which the mercury moiety and the solvent have added across the carbon–carbon unsaturation.[19] In fact, this reaction, called solvomercuriation, has become an extremely valuable tool for the functionalization of acetylenes and olefins when it is coupled with a convenient method for replacing the mercury moiety by hydrogen or other substituents.[20] Although, intermediates with a bridged mercurinium ion have been postulated,[19,20] evidence for the formation of the π complex in these reactions has not been reported. On the other hand, complexes containing σ-alkynyl ligands connected to mercury are known[9] but none with bridging alkynyl ligands containing π mercury–acetylene linkages. To the best of our knowledge, these complexes are the first examples of bi- and tri-nuclear transition-metal–mercury compounds containing π mercury–alkyne bonds.

Secondly, as mentioned above many metal complexes have been shown to form simple adducts with HgX_2. However in these reactions the metal complex is acting as a metallic base and the mercury dihalide as a Lewis acid. By contrast, in the bi- and tri-nuclear Pt–Hg compounds **1**–**6** and **7**–**12** respectively the dianionic metal substrates are acting only as mono- or bis-chelating ligands towards HgX_2.

In addition, the formation of complexes **1**–**12** is in contrast to the following previously reported facts: (*a*) treatment of the neutral derivatives $[\text{Au}(\text{C}\equiv\text{CPh})(\text{PPh}_3)]$,[21] *cis*-$[\text{Pt}(\text{C}\equiv\text{CR})(\text{C}\equiv\text{CR}')\text{L}(\text{CO})]$ (R,R′ = Me or Ph; R ≠ R′)[22] or *cis*-$[\text{Pt}(\text{C}\equiv\text{CPh})_2\text{L}_2]$ (L = PPh_3 or $PMePh_2$)[23] with $HgCl_2$ produces transfer of the ethynyl groups but not the formation of π alkynyl–mercury complexes and (*b*) although Shaw and co-workers[24–26] have recently synthesized binuclear complexes of the type *trans*-$[\text{M}(\text{C}\equiv\text{CR})_2(\mu\text{-Ph}_2\text{PCH}_2\text{PPh}_2)_2\text{HgCl}_2]$ (M = Ni,[24] Pd[25] or Pt[26]), these derivatives contain the alkynyl groups only acting as terminal ligands and the metal centres connected by bridging $Ph_2PCH_2PPh_2$ ligands.

In conclusion, our results demonstrate the peculiar ability of the anionic substrates $[\text{PtX}_2(\text{C}\equiv\text{CR})_2]^{2-}$ (X = C_6F_5 or C≡CR) to stabilize compounds in which the metal centres are connected by a double alkynyl bridging system. Thus, it has been possible to isolate for the first time compounds, including trinuclear HgPtHg derivatives, containing π mercury–alkyne bonds.

Experimental

The C, H and N analyses were determined with a Carlo Erba microanalyser. Infrared spectra (range 4000–200 cm^{-1}) were recorded on a Perkin–Elmer 883 spectrometer and ^{1}H and ^{19}F NMR on a Varian Unity 300 spectrometer. Chemical shifts are given in ppm relative to external standards ($SiMe_4$ and $CFCl_3$). Conductivities were measured in *ca.* 5 × 10^{-4} mol dm^{-3} acetone solutions using a Philips 9501/01 conductimeter. The starting complexes $[\text{NBu}_4]_2[\text{Pt}(\text{C}\equiv\text{CR})_4]\cdot2\text{H}_2\text{O}$ (R = Bu^t [7a] or SiMe$_3$ [8d]), $[\text{NBu}_4]_2[cis\text{-Pt}(\text{C}_6\text{F}_5)_2(\text{C}\equiv\text{CR})_2]$ (R = Bu^t [7b] or SiMe$_3$ [10]) and *cis*-$[\text{Pt}(\text{C}_6\text{F}_5)_2(\text{thf})_2]$[27] were prepared according to the literature methods.

Preparation of $[\text{NBu}_4]_2[\{cis\text{-Pt}(\text{C}_6\text{F}_5)_2(\text{C}\equiv\text{CR})_2\}\text{HgX}_2]$ **1**–**6**.—The stoichiometric amount of HgX_2 (X = Cl, Br or I) (molar ratio 1:1) was added to a colourless solution of $[\text{NBu}_4]_2[cis\text{-Pt}(\text{C}_6\text{F}_5)_2(\text{C}\equiv\text{CR})_2]$ (R = Bu^t or SiMe$_3$) in acetone (*ca.* 15 cm^3), and the mixture stirred at room temperature for *ca.* 15 min. The resulting pale yellow solution was evaporated to dryness and the residue treated with CH_2Cl_2–hexane (2:3) On cooling in the freezer (−30 °C) overnight, a white microcrystalline solid formed in each case. The respective solids were filtered off, repeatedly washed with hexane and air dried. For complex **5** (R = Bu^t, X = I) the resulting residue was treated with hexane to give a beige solid. Table 3 collects pertinent preparative information.

Preparation of $[\text{NBu}_4]_2[\{\text{Pt}(\text{C}\equiv\text{CR})_4(\text{HgX}_2)_2\}]$ **7**–**12**.—To a colourless solution of $[\text{NBu}_4]_2[\text{Pt}(\text{C}\equiv\text{CR})_4]\cdot2\text{H}_2\text{O}$ in acetone (20 cm^3) was added the stoichiometric amount of HgX_2 in a 1:2 molar ratio, whereupon a white solid formed. The mixture was stirred for *ca.* 10 min (see Table 2), concentrated to *ca.* 10 cm^3 and finally cooled in the freezer. The white solid that precipitated was filtered off, washed with hexane and air dried. Pertinent preparative information is given in Table 2.

Reaction of $[\text{NBu}_4]_2[\{cis\text{-Pt}(\text{C}_6\text{F}_5)_2(\text{C}\equiv\text{CR})_2\}\text{HgCl}_2]$ (R = Bu^t **1** *or* SiMe$_3$ **2**) *with cis*-$[\text{Pt}(\text{C}_6\text{F}_5)_2(\text{thf})_2]$.—To a solution of $[\text{NBu}_4]_2[\{cis\text{-Pt}(\text{C}_6\text{F}_5)_2(\text{C}\equiv\text{CBu}^t)_2\}\text{HgCl}_2]$ **1** (0.100 g, 0.068 mmol) in acetone (10 cm^3) was added *cis*-$[\text{Pt}(\text{C}_6\text{F}_5)_2(\text{thf})_2]$ (0.045 g, 0.068 mmol) and the mixture stirred at room temperature for 15 min. The resulting solution was evaporated to dryness and after addition of EtOH (5 cm^3), a white solid was obtained. It was filtered off and identified by IR spectroscopy as $[\text{NBu}_4]_2[\text{Pt}_2(\mu\text{-Cl})_2(\text{C}_6\text{F}_5)_4]$ (38% yield). From the mother-liquors only an intractable oil could be obtained.

Similar results were obtained with $[\text{NBu}_4]_2[\{cis\text{-Pt}(\text{C}_6\text{F}_5)_2(\text{C}\equiv\text{CSiMe}_3)_2\}\text{HgCl}_2]$ **2** and $[\text{NBu}_4]_2[\text{Pt}_2(\mu\text{-Cl})_2(\text{C}_6\text{F}_5)_4]$ was recovered in 40% yield.

Reaction of $[\text{NBu}_4]_2[\{\text{Pt}(\text{C}\equiv\text{CR})_4\}(\text{HgCl}_2)_2]$ (R = Bu^t **6** *or* SiMe$_3$ **7**) *with cis*-$[\text{Pt}(\text{C}_6\text{F}_5)_2(\text{thf})_2]$.—*cis*-$[\text{Pt}(\text{C}_6\text{F}_5)_2(\text{thf})_2]$ (0.087 g, 0.129 mmol) was added to a white suspension of **6** (0.100 g, 0.065 mmol) in acetone (15 cm^3) and the mixture stirred for 15 min at room temperature. The resulting orange solution was then filtered and evaporated to dryness. On addition of EtOH a white solid, identified as $[\text{NBu}_4]_2[\text{Pt}_2(\mu\text{-Cl})_2(\text{C}_6\text{F}_5)_4]$ separated in 38% yield.

Similar results were obtained using complex **7** but in this case the mixture darkened immediately and was stirred only for 2 min. The compound $[\text{NBu}_4]_2[\text{Pt}_2(\mu\text{-Cl})_2(\text{C}_6\text{F}_5)_4]$ was recovered in 40% yield.

Crystal-structure Determination of $[\text{NBu}_4]_2[\{cis\text{-Pt}(\text{C}_6\text{F}_5)_2(\text{C}\equiv\text{CSiMe}_3)_2\}\text{HgBr}_2]\cdot\text{CH}_2\text{Cl}_2$. **4**.—Suitable crystals of **4** were obtained by slow diffusion of hexane into a CH_2Cl_2 solution at −40 °C.

Crystal data. $C_{55}H_{92}Br_2Cl_2F_{10}HgN_2PtSi_2$, $M = 1653.89$, monoclinic, space group $P2_1/c$, $a = 18.256(3)$, $b = 18.757(4)$, $c = 21.184(4)$ Å, $\beta = 99.05(1)°$, $U = 7164(1)$ Å^3, $Z = 4$, $D_c = 1.53$ g cm^{-3}, $\lambda(\text{Mo-K}\alpha) = 0.710\,73$ Å, $\mu = 53.7$ cm^{-1}, $F(000) = 3264$, $T = 293 \pm 1$ K, crystal dimensions 0.35 × 0.40 × 0.42 mm.

Crystallographic data were collected by Crystalytics (Lincoln, Nebraska) on a four-circle Nicolet (Siemens) autodiffractometer, $3 < 2\theta < 43°$ (ω scan); 6521 independent

Table 3 Some experimental details for the synthesis of the complexes

Complex	Amount (g, mmol)	HgX_2 (g, mmol)	*t*/min	Yield (%)
1 $[NBu_4]_2[\{cis\text{-}Pt(C_6F_5)_2(\mu\text{-}C{\equiv}CBu^t)_2\}HgCl_2]$	0.15, 0.13[a]	0.035, 0.13[b]	30	80
2 $[NBu_4]_2[\{cis\text{-}Pt(C_6F_5)_2(\mu\text{-}C{\equiv}CSiMe_3)_2\}HgCl_2]$	0.20, 0.16[c]	0.045, 0.16[b]	15	90
3 $[NBu_4]_2[\{cis\text{-}Pt(C_6F_5)_2(\mu\text{-}C{\equiv}CBu^t)_2\}HgBr_2]$	0.16, 0.14[a]	0.051, 0.14[d]	15	65
4 $[NBu_4]_2[\{cis\text{-}Pt(C_6F_5)_2(\mu\text{-}C{\equiv}CSiMe_3)_2\}HgBr_2]$	0.17, 0.14[c]	0.051, 0.14[d]	20	84
5 $[NBu_4]_2[\{cis\text{-}Pt(C_6F_5)_2(\mu\text{-}C{\equiv}CBu^t)_2\}HgI_2]$	0.15, 0.13[a]	0.058, 0.13[e]	15	75
6 $[NBu_4]_2[\{cis\text{-}Pt(C_6F_5)_2(\mu\text{-}C{\equiv}CSiMe_3)_2\}HgI_2]$	0.11, 0.09[c]	0.041, 0.09[e]	15	40
7 $[NBu_4]_2[\{Pt(\mu\text{-}C{\equiv}CBu^t)_4\}(HgCl_2)_2]$	0.15, 0.14[f]	0.078, 0.29[b]	10	58
8 $[NBu_4]_2[\{Pt(\mu\text{-}C{\equiv}CSiMe_3)_4\}(HgCl_2)_2]$	0.15, 0.14[g]	0.074, 0.27[b]	5	43
9 $[NBu_4]_2[\{Pt(\mu\text{-}C{\equiv}CBu^t)_4\}(HgBr_2)_2]$	0.20, 0.19[f]	0.14, 0.38[d]	10	30
10 $[NBu_4]_2[\{Pt(\mu\text{-}C{\equiv}CSiMe_3)_4\}(HgBr_2)_2]$	0.20, 0.19[g]	0.13, 0.37[d]	10	45
11 $[NBu_4]_2[\{Pt(\mu\text{-}C{\equiv}CBu^t)_4\}(HgI_2)_2]$	0.15, 0.14[f]	0.13, 0.29[e]	20	70
12 $[NBu_4]_2[\{Pt(\mu\text{-}C{\equiv}CSiMe_3)_4\}(HgI_2)_2]$	0.20, 0.19[g]	0.17, 0.37[e]	10	35

[a] $[NBu_4]_2[cis\text{-}Pt(C_6F_5)_2(C{\equiv}CBu^t)_2]$. [b] X = Cl. [c] $[NBu_4]_2[cis\text{-}Pt(C_6F_5)_2(C{\equiv}CSiMe_3)_2]$. [d] X = Br. [e] X = I. [f] $[NBu_4]_2[Pt(C{\equiv}CBu^t)_4]\cdot 2H_2O$. [g] $[NBu_4]_2[Pt(\mu\text{-}C{\equiv}CSiMe_3)_4]\cdot 2H_2O$.

Table 4 Fractional atomic coordinates (× 10^4) for $[NBu_4]_2[\{cis\text{-}Pt(C_6F_5)_2(C{\equiv}CSiMe_3)_2\}HgBr_2]\cdot CH_2Cl_2$ **4**

Atom	*x*	*y*	*z*	Atom	*x*	*y*	*z*
Hg	1 545(1)	1 368(1)	2 646(1)	C(24)	898(20)	8 105(17)	1 342(17)
Pt	2 715(1)	−126(1)	2 366(1)	C(25)	688(26)	7 506(22)	846(20)
Br(1)	121(2)	1 362(1)	2 602(2)	C(26A)	1 184(52)	6 875(42)	1 101(53)
Br(2)	2 139(2)	2 580(2)	2 984(2)	C(26B)	1 338(43)	7 219(58)	541(51)
C(1)	3 247(16)	−512(13)	1 651(12)	C(27)	896(12)	8 807(13)	2 673(12)
C(2)	3 966(18)	−587(16)	1 650(17)	C(28)	940(19)	8 057(19)	3 058(16)
C(3)	4 269(19)	−881(23)	1 134(25)	C(29)	1 564(19)	8 171(16)	3 616(15)
C(4)	3 802(32)	−1 074(22)	602(20)	C(30)	1 686(24)	7 533(21)	4 018(19)
C(5)	3 121(22)	−1 090(18)	596(21)	C(31)	348(15)	9 534(12)	1 775(11)
C(6)	2 847(19)	−762(17)	1 085(18)	C(32)	−297(16)	9 673(12)	1 239(12)
F(2)	4 434(10)	−328(10)	2 160(9)	C(33)	−135(18)	10 390(14)	934(13)
F(3)	5 026(12)	−893(11)	1 242(10)	C(34)	−759(20)	10 616(17)	377(16)
F(4)	4 180(15)	−1 385(13)	164(12)	C(35)	−534(14)	8 760(15)	2 385(14)
F(5)	2 707(15)	−1 319(14)	57(11)	C(36)	−562(18)	9 381(21)	2 842(15)
F(6)	2 072(10)	−734(9)	966(8)	C(37)	−1 342(22)	9 306(21)	3 040(19)
C(7)	3 232(15)	−938(18)	2 954(16)	C(38)	−1 477(22)	9 893(27)	3 544(20)
C(8)	3 666(19)	−777(15)	3 522(16)	N(2)	5 364(14)	7 095(12)	2 245(14)
C(9)	3 979(23)	−1 309(30)	3 933(16)	C(39)	4 541(16)	7 317(20)	1 897(19)
C(10)	3 850(32)	−1 954(26)	3 776(31)	C(40)	4 173(26)	6 679(23)	1 603(25)
C(11)	3 449(24)	−2 166(25)	3 205(27)	C(41)	3 539(32)	7 002(25)	1 208(30)
C(12)	3 119(20)	−1 646(17)	2 815(20)	C(42)	3 066(30)	6 537(32)	705(29)
F(8)	3 826(10)	−80(11)	3 700(8)	C(43)	5 789(17)	7 799(16)	2 441(14)
F(9)	4 397(12)	−1 078(12)	4 495(11)	C(44)	5 524(24)	8 236(17)	2 914(17)
F(10)	4 178(13)	−2 462(12)	4 224(11)	C(45)	6 016(21)	8 904(18)	3 050(22)
F(11)	3 341(12)	−2 846(10)	3 116(12)	C(46)	5 839(25)	9 410(18)	3 491(20)
F(12)	2 718(10)	−1 868(9)	2 290(11)	C(47)	5 799(19)	6 659(20)	1 828(18)
C(13)	2 208(13)	629(13)	1 836(11)	C(48)	5 879(19)	7 017(21)	1 203(23)
C(14)	1 901(15)	1 166(14)	1 612(13)	C(49)	6 237(24)	6 561(24)	768(19)
Si(1)	1 610(4)	1 882(4)	975(4)	C(50)	6 252(24)	6 929(22)	155(21)
C(15)	2 314(13)	2 602(14)	1 080(12)	C(51)	5 164(23)	6 656(21)	2 832(26)
C(16)	1 561(20)	1 445(13)	171(13)	C(52)	5 841(20)	6 518(14)	3 230(29)
C(17)	681(15)	2 288(12)	1 067(15)	C(53)	5 559(25)	6 106(20)	3 853(26)
C(18)	2 215(13)	217(13)	3 072(13)	C(54)	6 173(30)	5 928(23)	4 480(24)
C(19)	1 902(14)	459(14)	3 475(13)	C(55)	7 198(19)	328(34)	1 487(19)
Si(2)	1 617(5)	598(5)	4 277(4)	Cl(1)	7 998(11)	775(11)	1 822(12)
C(20)	720(16)	103(20)	4 315(16)	Cl(2)	6 533(12)	381(11)	1 990(13)
C(21)	2 367(19)	235(22)	4 862(14)	C(56)	7 127(36)	215(30)	972(48)
C(22)	1 469(20)	1 587(16)	4 428(15)	Cl(3)	7 367(16)	−629(18)	1 270(17)
N(1)	232(14)	8 828(11)	2 119(10)	Cl(4)	6 297(17)	493(18)	1 202(19)
C(23)	197(17)	8 171(13)	1 642(14)				

reflections [3873 with $I > 2\sigma(I)$] were used for all calculations. Cell constants were refined from 2θ values of 30 reflections including Friedel pairs (2θ > 15°). An absorption correction based on ψ scans was applied (transmission factors: 1.000–0.579). Six standard reflections were measured every 300 reflections but showed no decay. The structure was solved by direct methods and refined on $|F_o^2|$ (SHELX 93[28]) to $wR2 = 0.190$ $\{wR2 = [\Sigma w(F_o^2 - F_c^2)^2/\Sigma w(F_o^2)^2]^{\frac{1}{2}}$; weighting scheme $w^{-1} = [\sigma^2(F_o^2) + (0.1006P)^2]$ where $P = [\max(F_o^2, 0) + 2F_c^2]/3\}$ for 677 variables ($R = 0.070$, $R = \Sigma||F_o| - |F_c||/\Sigma|F_o|$), highest shift/error 0.010. All non-hydrogen atoms were refined anisotropically, except for one methyl carbon atom of one of the tetrabutylammonium cations which appeared disordered over two sites with occupancy 0.5. In the final stages of the refinement the presence of disordered solvent was discovered. After several attempts, the best model was found by identifying this solvent as two molecules of dichloromethane with partial occupancies of 0.6 and 0.4, very near one another. In these molecules the C–Cl distances were fixed to 1.74 and the three atoms of the same molecule were refined with a common thermal parameter. A difference map following convergence showed three peaks higher than 1 e Å^{-3} (maximum, minimum difference density $1.56, -0.69$ e Å^{-3}) near the solvent atoms. Final atomic coordinates are given in Table 4.

Additional material available from the Cambridge Crystallographic Data Centre comprises H-atom coordinates, thermal parameters and remaining bond lengths and angles.

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