

Symmetrization reaction of mercurated ferrocenylienes. X-ray crystal structure of $[\text{Hg}\{(\eta^5\text{-C}_5\text{H}_5)\text{Fe}(\eta^5\text{-C}_5\text{H}_3\text{CPh}=\text{NC}_6\text{H}_4\text{-4-Br})\}_2]$

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Abstract

The symmetrization reaction of mercurated anils of benzoylferrocene **1** was easily achieved by refluxing **1** and PPh_3 in CHCl_3 solution, and afforded the symmetrized products $[\text{Hg}\{(\eta^5\text{-C}_5\text{H}_5)\text{Fe}(\eta^5\text{-C}_5\text{H}_3\text{CPh}=\text{NAr})\}_2]$ (Ar = a variety of substituted phenyls and β -naphthyl) **2**. During the reaction the organometallic complex salts $[\text{RHgPPh}_3]\text{Cl}$ formed; this can disproportionate to give the symmetrized products. The molecular structure of $[\text{Hg}\{(\eta^5\text{-C}_5\text{H}_5)\text{Fe}(\eta^5\text{-C}_5\text{H}_3\text{CPh}=\text{NC}_6\text{H}_4\text{-4-Br})\}_2]$ has been determined by single-crystal X-ray analysis. It crystallizes in the triclinic space group $P\bar{1}$, with $a = 13.001(4)$, $b = 13.029(2)$, $c = 12.236(2)$ Å, $\alpha = 103.00(1)$, $\beta = 104.10(2)$, $\gamma = 78.86(2)^\circ$ and $Z = 2$. Refinement led to $R = 0.027$ ($R_w = 0.037$) using 3271 unique reflections with $I > 3\sigma(I)$. The distance between N(2) and Hg is 2.981(6) Å, shorter than the sum of van der Waals radii of N and Hg (3.05–3.15 Å), indicating an intramolecular coordination of N(2) and Hg. The ^1H NMR spectra of $[\text{Hg}\{(\eta^5\text{-C}_5\text{H}_5)\text{Fe}(\eta^5\text{-C}_5\text{H}_3\text{CPh}=\text{NAr})\}_2]$ (Ar = $\text{C}_6\text{H}_4\text{-2-Cl}$ and β -naphthyl) show that both compounds exist as rotamers in solution.

Keywords: Symmetrization; Mercury; Iron; Ferrocene; Crystal structure

1. Introduction

As with other organomercury compounds, symmetrized organomercury compounds can be widely used in organic synthesis, such as in the synthesis of other organometallic compounds through transmetallation reactions, in particular when other organomercury compounds do not react in some transmetallation reactions [1]. For symmetrized ferrocene-containing organomercury compounds, their oxidized derivatives may also be considered to be organometallic mixed-valence complexes [2,3] which may be used as high temperature and novel superconducting materials. Here we report a facile method for preparing the symmetrized organomercury compounds containing a ferrocene group and the crystal structure of the complex $[\text{Hg}\{(\eta^5\text{-C}_5\text{H}_5)\text{Fe}(\eta^5\text{-C}_5\text{H}_3\text{CPh}=\text{NC}_6\text{H}_4\text{-4-Br})\}_2]$ **2e**. The crystal structure of these kinds of Hg-bridged complex was seldom found in the literature [3].

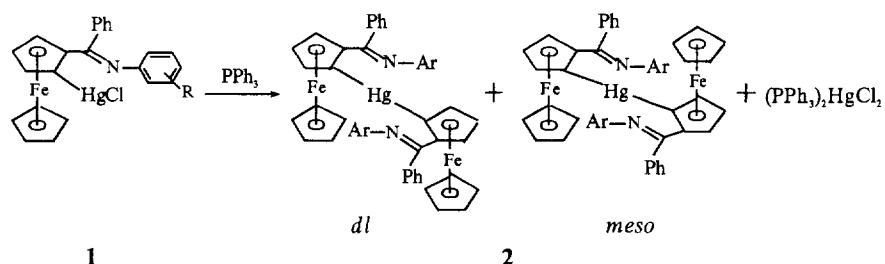
2. Results and discussion

2.1. Symmetrization reaction

2.1. Symmetrization reaction

It has been reported that organomercury halides symmetrized in the presence of NaI , $\text{Na}_2\text{S}_2\text{O}_3$, $\text{Na}_2\text{Sn}_2\text{O}_3$, or PPh_3 [4–6]. The reaction between organolithium compounds and HgX_2 also affords the symmetrized products [6]. Coates and Lauder reported that the reaction between alkylmercury halides and tertiary alkyl phosphines gave salts $[\text{RHgPR}'_3]\text{X}$ [5], which slowly disproportionated in solution and changed into diorganomercury compounds. In our system, we found that PPh_3 is an efficient reagent to symmetrize the organomercury compounds **1**.

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Ar = *p*-CH₃O-C₆H₄ (**a**), *p*-CH₃-C₆H₄ (**b**), C₆H₅ (**c**), *p*-Cl-C₆H₄ (**d**), *p*-Br-C₆H₄ (**e**), *p*-I-C₆H₄ (**f**), *m*-CH₃-C₆H₄ (**g**), *m*-Cl-C₆H₄ (**h**), *m*-Br-C₆H₄ (**i**), *o*-Cl-C₆H₄ (**j**), β -naphthyl (**k**).

On refluxing mercurated complexes **1** with PPh₃ in CHCl₃ solvent for 0.5 h, a TLC examination (elution with methylene chloride) showed that three bands appeared. The first band ($R_f \sim 0.87$) contained the symmetrized products. The second band ($R_f \sim 0.17$) contained the complex salts [RHgPPh₃]Cl, which are unstable and can disproportionate to afford the symmetrized products during the work-up of chromatography separation, or standing in solution. So the pure complex salts were not isolated. The third band contained the complex (PPh₃)₂HgCl₂ ($R_f < 0.17$). If the second band was collected and subjected to a second chromatography separation, some symmetrized products and (PPh₃)₂HgCl₂ were obtained again. The new compounds **2** are air-stable crystals. They are highly soluble

in chloroform, dichloromethane, acetone, etc., but insoluble in petroleum ether and *n*-hexane. The yields, analytical data and melting points or decomposition temperatures of the compounds are shown in Table 1. The ¹H NMR data are summarized in Table 2. The rate of the disproportionation is influenced by the substituents on the *N*-phenyl ring in the order electron-donating substituent > electron-withdrawing substituent.

The infrared spectra of **2** show an intense, sharp band in the range of 1578–1593 cm⁻¹ assigned to the stretching of the C=N bond, which shifted to lower energy by 12–34 cm⁻¹ compared with the unmercurated imines [7], indicating an intramolecular N → Hg coordination in the molecules [8].

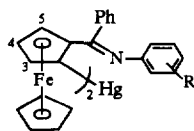
The symmetrized products displayed similar ¹H NMR spectra pattern for all the protons as starting materials **1**. The proton signals of *dl* and *meso* isomers were not separated. This may be due to the similar magnetic environments between the protons of the *dl* and *meso*

Table 1
Yields, analytical data, melting points and IR data for compounds **2**

Compound (substituent)	M.p. (°C)	Empirical formula	Yield ^a	Elemental analysis ^b			IR data (cm ⁻¹)			
				C	H	N				
2a (<i>p</i> -CH ₃ O)	> 224 ^c	C ₄₈ H ₄₀ Fe ₂ HgN ₂ O ₂	70	58.11 (58.29)	4.05 (4.08)	2.94 (2.83)	3052	1590	1495	1423
2b (<i>p</i> -CH ₃)	283–284 ^c	C ₄₄ H ₄₈ Fe ₂ HgN ₂	71	59.88 (60.24)	4.08 (4.21)	2.90 (2.93)	3070	1590	1500	1425
2c (<i>p</i> -H)	258–260 ^c	C ₄₆ H ₃₆ Fe ₂ HgN ₂	72	59.02 (59.47)	3.81 (3.91)	3.02 (3.02)	3050	1588	1420	1100
2d (<i>p</i> -Cl)	313–314 ^c	C ₄₆ H ₃₄ Cl ₂ Fe ₂ HgN ₂	48	54.85 (55.37)	3.51 (3.43)	2.65 (2.81)	3050	1590	1420	1100
2e (<i>p</i> -Br)	213–214 ^c	C ₄₆ H ₃₄ Br ₂ Fe ₂ HgN ₂	40	50.46 (50.84)	3.00 (3.15)	2.31 (2.58)	3065	1593	1428	1105
2f (<i>p</i> -I)	> 260 ^c	C ₄₆ H ₃₄ Fe ₂ HgI ₂ N ₂	37	46.55 (46.79)	2.62 (2.90)	2.02 (2.37)	3048	1590	1430	1100
2g (<i>m</i> -CH ₃)	268–270	C ₄₈ H ₄₀ Fe ₂ HgN ₂	71	59.97 (60.24)	4.47 (4.21)	2.55 (2.93)	3050	1585	1422	1100
2h (<i>m</i> -Cl)	274–276 ^c	C ₄₈ H ₃₄ Cl ₂ Fe ₂ HgN ₂	50	55.44 (55.37)	3.44 (3.43)	2.30 (2.81)	3046	1580	1430	1105
2i (<i>m</i> -Br)	275–276 ^c	C ₅₅ H ₃₄ Br ₂ Fe ₂ HgN ₂	65	51.11 (50.84)	3.17 (3.15)	2.69 (2.58)	3050	1578	1422	1100
2j (<i>o</i> -Cl)	258–262 ^c	C ₄₆ H ₃₄ Cl ₂ Fe ₂ HgN ₂	52	55.18 (55.37)	3.47 (3.43)	2.87 (2.81)	3050	1591	1420	1103
2k (β -naphthyl)	> 270 ^c	C ₅₄ H ₄₀ Fe ₂ HgN ₂	43	62.50 (63.02)	3.91 (3.92)	2.55 (2.72)	3022	1591	1418	1102

^a Based on the first chromatography separation; ^b Calculated values in parentheses; ^c Melts with decomposition.

Table 2
¹H NMR data (δ ppm) of compounds **2**^{a,b}



Compound	C ₅ H ₅	5	4	3	N-Ph		CH ₃	C-Ph
2a	4.08s	4.13d (1.5)	4.49t (2.4)	4.38t (2.4)	6.74d (8.9)	6.70d (8.9)	3.72s	7.30m
2b	4.07s	4.26m	4.46t (2.4)	4.36dd (0.8, 1.9)	6.67d (8.2)	6.93d (8.2)	2.22s	7.26m
2c	4.06s	4.28dd (1, 2.4)	4.47t (2.4)	4.35dd (1.1, 2.1)	6.79dd (1.1, 8.2)	7.14t (8.1)	6.90m	7.26m
2d	4.09s	4.29m	4.51t (2.4)	4.33m	6.69d (8.1)	7.10d (8.1)		7.28m
2e	4.09s	4.28m	4.51t (2.4)	4.33m	6.62d (8.1)	7.24d (8.1)		7.28m
2f	4.08s	4.28dd (0.8, 2.4)	4.51t (2.4)	4.33dd (1.0, 2.1)	6.51d (8.5)	7.40d (8.5)		7.28m
2g	4.08s	4.28m	4.46t (2.4)	4.33t (1.1)	6.54d (7.9)	7.00t (7.9)	6.71d (7.6)	6.63s 2.22s
2h	4.11s	4.32m	4.52t (2.3)	4.36dd (1.0, 2.0)	6.56m	7.02t (7.9)	6.88m 6.86m	7.28m
2i	4.12s	4.32m	4.52t (2.4)	4.36m	6.58m	6.96t (8.1)	7.03m	7.27m
2j	4.13s 4.07s	4.32m 4.37m	4.48m 4.48m	4.48m				
2k	4.05s 4.08s	4.33m 4.28m	4.46t 4.43t	4.36m 4.29m				

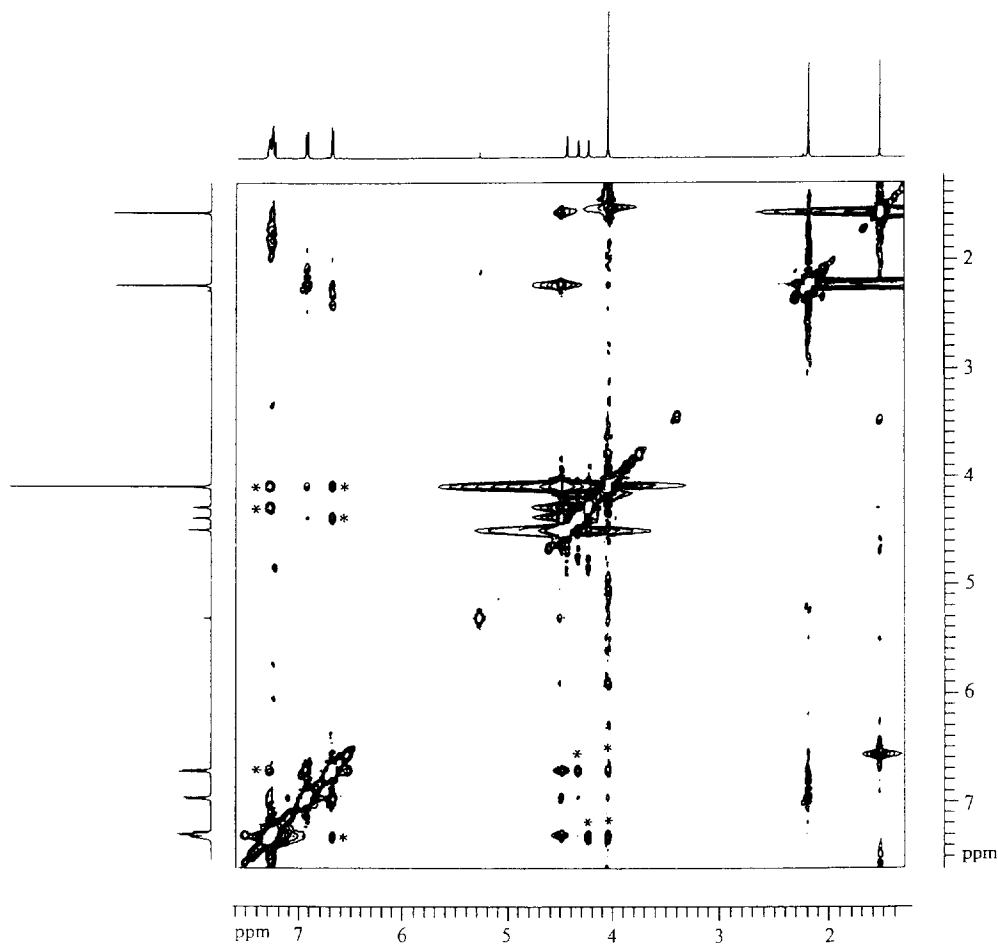
^a CDCl₃, as the solvent, TMS as the internal standard. ^b Coupling constants in parentheses and all *J* values in hertz.

forms. For ferrocene resonances, protons 3 should appear at higher field owing to the shielding effect of the mercury group and *N*-phenyl ring (vide infra); protons 5 should also appear at higher field because they can be shielded by the *C*-phenyl ring. 2D-NOESY spectrum of **2b** (Fig. 1) was measured to assign unambiguously the signals of protons 3 and 5. It was confirmed that the signals at δ 4.26 ppm correspond to the protons 5, due to the NOE cross-peak between the protons 5 and *C*-phenyl ring, and the signals at δ 4.36 ppm represent the resonance of protons 3 because of the appearance of a NOE cross-peak between the protons 3 and *N*-phenyl ring. Additionally, the protons of the Cp ring showed NOE with the protons of the *C*-phenyl and *N*-phenyl rings, suggesting the more-or-less perpendicular nature of the unsubstituted Cp ring with the *C*-phenyl and *N*-phenyl rings. It is noteworthy that in the cases of **2j** and **2k**, ¹H NMR spectra indicated that two isomers were present in CDCl₃ solution (Fig. 2). This isomerism may not be caused by the *dl* and meso isomers because in other cases the proton signals of the *dl* and meso forms were not separated. It is likely that the free rotation of the *N*-phenyl ring about the C–N bond is blocked by the steric hindrance between the ortho-substituent or β-naphthyl group and the C–Hg–C bond,

resulting in two isomers. Similar isomerism was also found in cyclopalladated ferrocenylamines [9].

2.2. Structure elucidation of **2e** by X-ray crystallography

The conclusive evidence for the structure of **2** has been obtained from X-ray structure analysis. A perspective view of the molecular structure of **2e** (*dl* form) is shown in Fig. 3. Selected bond distances and angles are listed in Table 3, and dihedral angles in Table 4. The final atomic parameters are presented in Table 5. Two ferrocenyl moieties are linked by a mercury bridge and at the same side of the mercury bridge. The lone electron pairs of two N atoms are directed toward the Hg atom. The distance Hg–N(2) (2.981(6) Å; 2.870 Å in **1c**), is shorter than the sum of van der Waals radii of N and Hg (3.05–3.15 Å) [10,11], and corresponds to the intramolecular coordination, whereas the Hg–N(1) distance 3.040(6) Å shows a weak interaction between Hg and N(1). The distances Hg–C(6) (2.069 Å) and Hg–C(24) (2.062 Å) are typical of organic derivatives of mercury [10]. The bond angle C(6)–Hg–C(24) is 174.7°, narrower than the ideal value of 180° in organic derivatives of mercury.

Fig. 1. 2D NOESY spectra of **2b**: * indicates NOE peaks.Table 3
Selected bond distances (Å) and bond angles (deg) for **2e**

Fe(1)–C(1)	2.061(8)	Fe(2)–C(26)	2.032(8)	N(2)–C(34)	1.297(9)
Fe(1)–C(2)	2.045(8)	Fe(2)–C(27)	2.032(8)	N(2)–C(41)	1.400(10)
Fe(1)–C(3)	2.033(9)	Fe(2)–C(28)	2.040(7)	C(6)–C(10)	1.43(1)
Fe(1)–C(4)	2.029(8)	Fe(2)–C(29)	2.045(8)	C(10)–C(11)	1.45(1)
Fe(1)–C(5)	2.048(9)	Fe(2)–C(30)	2.052(8)	C(11)–C(12)	1.52(1)
Fe(1)–C(6)	2.056(7)	Fe(2)–C(31)	2.024(9)	C(24)–C(28)	1.43(1)
Fe(1)–C(7)	2.045(7)	Fe(2)–C(32)	2.022(9)	C(28)–C(34)	1.47(1)
Fe(1)–C(8)	2.042(7)	Fe(2)–C(33)	2.022(9)	C(34)–C(35)	1.485(10)
Fe(1)–C(9)	2.020(7)	Hg–C(6)	2.069(7)	Br(1)–C(20)	1.899(9)
Fe(1)–C(10)	2.036(7)	Hg–C(24)	2.062(7)	Br(2)–C(43)	1.898(8)
Fe(2)–C(24)	2.076(7)	N(1)–C(11)	1.300(9)	Hg–N(1)	3.040(6)
Fe(2)–C(25)	2.039(7)	N(1)–C(18)	1.416(10)	Hg–N(2)	2.981(6)
C(6)–Hg–C(24)	174.7(3)	Hg–C(24)–C(28)	123.7(5)		
C(11)–N(1)–C(18)	120.5(6)	C(24)–C(28)–C(34)	125.5(6)		
C(34)–N(2)–C(41)	122.0(6)	C(27)–C(28)–C(34)	124.4(7)		
Hg–C(6)–C(7)	127.5(6)	N(2)–C(34)–C(28)	119.5(6)		
Hg–C(6)–C(10)	124.8(5)	N(2)–C(34)–C(35)	121.3(7)		
C(6)–C(10)–C(11)	126.3(6)	C(28)–C(34)–C(35)	119.1(6)		
C(9)–C(10)–C(11)	126.3(7)	C(34)–C(35)–C(36)	122.4(7)		
N(1)–C(11)–C(10)	118.8(6)	C(34)–C(35)–C(40)	119.9(7)		
N(1)–C(11)–C(12)	122.6(7)	N(2)–C(41)–C(42)	120.8(8)		
C(10)–C(11)–C(12)	118.6(7)	N(2)–C(41)–C(46)	120.2(8)		
C(11)–C(12)–C(13)	120.4(8)	N(1)–Hg–N(2)	157.1(2)		
N(1)–C(18)–C(19)	118.1(7)	N(2)–Hg–C(24)	68.6(3)		
N(1)–C(18)–C(23)	122.2(7)	Br(1)–C(20)–C(19)	118.6(7)		
Hg–C(24)–C(25)	129.8(5)	Br(1)–C(20)–C(21)	118.8(7)		

Table 4
Dihedral angles for compound **2e**

Defining plane	1 ^a	2	3	4	5	6	7	8	9	10
N(1) C(10)	1 ^a									
C(11) C(18)										
N(2) C(28)	2	126.59								
C(34) C(41)										
N(2) C(24) Hg	3	136.15	10.10							
C(28) C(34)										
C(1)–C(5)	4	14.57	135.08	143.22						
C(6)–C(10)	5	13.21	138.06	146.86	5.52					
C(12)–C(17)	6	103.75	78.33	74.09	93.28	98.41				
C(18)–C(23)	7	70.01	61.26	71.23	83.35	83.22	113.57			
C(24)–C(28)	8	130.54	4.10	6.01	138.57	141.78	76.74	65.27		
C(29)–C(33)	9	128.67	4.75	7.53	135.87	139.37	73.84	65.16	3.63	
C(35)–C(40)	10	88.24	94.27	91.05	76.60	81.51	17.63	116.32	93.15	90.01
C(41)–C(46)	11	58.82	72.87	82.87	72.54	72.03	117.58	11.68	76.89	76.68
										116.41

^a: Plane numbers.

only 13.21° and 4.10° respectively, indicating the better conjugation between the substituted Cp ring and the C=N bond. All these features accord with the NMR results. The chelate cycle Hg–N(2)–C(24)–C(28)–C(34) is nearly planar, with a mean deviation of 0.0617 Å. The average Fe(1)–C(1–5) and Fe(1)–C(6–10) distances are 2.0432 Å and 2.0398 Å; Fe(2)–C(24–28) and Fe(2)–C(29–33) distances are 2.0438 Å and 2.033 Å, and the average C–C distances in the ferrocenyl moieties are C (1–5) 1.402 Å, C (6–10) 1.418 Å, C(24–28) 1.422 Å, C(29–33) 1.4 Å.

3. Experimental section

Melting points were determined on a WC-1 microscopic apparatus and are uncorrected. ¹H NMR spectra were recorded using a Bruker ARX 500 spectrometer, in chloroform, and all *J* values are in hertz. IR spectra were recorded on a Shimadzu IR 435 spectrophotometer. Elemental analyses were determined with a Carlo Erba 1106 Elemental Analyzer. 2D NOESY spectra were recorded in CDCl₃ at room temperature with a mixing time of 300 ms. Chromatographic work was carried out using a silica gel packed dry column under reduced pressure.

3.1. General procedure for the symmetrization reaction

A mixture of cyclomercurated complexes **1** (1 mmol) [7] and a molar equivalent PPh₃ (0.26 g, 1 mmol) in CHCl₃ solution was refluxed for 0.5 h. After standing in a fumehood for one night, the solution were concentrated in vacuo and subjected rapidly to a short silica gel column. The first band was collected to afford the symmetrized products **2**. This can be recrystallized from dichloromethane–petroleum ether (60–90 °C). The second band contained the complex salts [RHgPPh₃]Cl;

this was cut off and eluted with acetone. After removal of acetone and carrying out a second chromatography separation, some symmetrized products can be obtained again. The third band contained complex (PPh₃)₂HgCl₂, m.p. 265–267 °C, consistent with the value in the literature [13].

3.2. X-ray crystal structure determination for **2e**

3.2.1. Crystal data

C₄₆H₃₄Br₂Fe₂HgN₂, *M_r* = 1086.88, triclinic, *P* $\bar{1}$ (No. 2), *a* = 13.001(4), *b* = 13.029(2), *c* = 12.236(2) Å, α = 103.00(1), β = 104.10(2), γ = 78.86(2)°, *V* = 1937.6(8) Å³, *Z* = 2, *D_c* = 1.86 g cm⁻³, *F*(000) = 1052.00, λ = 0.71609 Å, μ (Mo K α) = 68.05 cm⁻¹.

3.2.2. Data collection

A red plate crystal of **2e** with approximate dimensions 0.20 × 0.20 × 0.45 mm³ was mounted on a Rigaku AFC7R diffractometer. Unit cell parameters were determined from the angular setting of 22 reflections with 2 θ angles in the range 23.77–26.60°, and refined by a least squares method. Intensities were collected with graphite monochromated Mo K α radiation, using the ω –2 θ scan technique. A total of 3822 reflections were measured, 3271 reflections were considered as observed with *I* > 3 σ (*I*). Three reflections were measured after every 200 reflections as orientation and intensity controls and no significant intensity decay was observed. Data were corrected for Lorentz and polarization effects and also for absorption by an empirical method using the program DIFABS [14].

3.2.3. Structure solution and refinement

The structure was solved by direct methods and expanded using Fourier techniques, and refined by full-matrix least squares methods. All calculations were

Table 5
Final positional parameters for compound **2e**

Atom	x	y	z	B_{eq}
Hg(0)	0.90673(2)	0.61107(2)	0.43907(2)	2.35(1)
Br(1)	0.32489(9)	0.5623(1)	0.2622(1)	7.04(3)
Br(2)	1.36380(9)	0.92333(8)	0.61680(10)	5.87(3)
Fe(1)	0.96642(8)	0.73316(8)	0.23004(9)	2.67(3)
Fe(2)	0.82323(9)	0.78378(8)	0.68313(9)	2.84(3)
N(1)	0.6986(5)	0.6571(5)	0.2680(5)	3.0(2)
N(2)	1.1038(5)	0.6591(5)	0.6141(5)	3.6(2)
C(1)	0.9925(9)	0.8517(7)	0.3726(8)	4.9(3)
C(2)	1.0860(7)	0.8139(7)	0.3322(8)	4.4(2)
C(3)	1.0662(8)	0.8307(7)	0.2182(9)	4.7(3)
C(4)	0.9579(10)	0.8782(7)	0.1907(9)	5.6(3)
C(5)	0.9127(8)	0.8898(7)	0.2878(10)	4.8(3)
C(6)	0.9296(6)	0.6043(6)	0.2760(6)	2.5(2)
C(7)	1.0274(6)	0.5774(6)	0.2380(6)	3.0(2)
C(8)	1.0075(7)	0.5941(6)	0.1235(7)	3.2(2)
C(9)	0.9002(7)	0.6331(6)	0.0902(6)	3.2(2)
C(10)	0.8490(6)	0.6391(6)	0.1843(6)	2.5(2)
C(11)	0.7360(6)	0.6732(6)	0.1850(6)	2.5(2)
C(12)	0.6647(6)	0.7218(7)	0.0850(6)	3.4(2)
C(13)	0.5960(9)	0.6639(9)	0.0023(9)	5.9(3)
C(14)	0.530(1)	0.707(1)	-0.0860(9)	7.8(4)
C(15)	0.5317(10)	0.807(1)	-0.0936(9)	7.0(4)
C(16)	0.599(1)	0.8682(10)	-0.013(1)	7.2(4)
C(17)	0.6664(9)	0.8222(9)	0.0760(8)	6.0(3)
C(18)	0.5913(6)	0.6974(7)	0.2773(6)	2.9(2)
C(19)	0.5211(7)	0.6247(7)	0.2637(7)	3.5(2)
C(20)	0.4199(7)	0.6619(7)	0.2815(7)	3.7(2)
C(21)	0.3853(7)	0.7673(9)	0.3151(7)	4.2(2)
C(22)	0.4557(8)	0.8392(7)	0.3294(8)	4.7(3)
C(23)	0.5580(7)	0.8028(7)	0.3099(7)	3.9(2)
C(24)	0.8814(6)	0.6330(6)	0.6033(6)	2.5(2)
C(25)	0.7858(6)	0.6329(6)	0.6412(6)	3.1(2)
C(26)	0.8027(7)	0.6698(6)	0.7620(7)	3.5(2)
C(27)	0.9094(7)	0.6942(6)	0.8000(6)	3.5(2)
C(28)	0.9562(6)	0.6718(6)	0.7039(6)	2.4(2)
C(29)	0.8589(8)	0.9187(7)	0.6507(9)	4.8(3)
C(30)	0.7783(8)	0.8783(7)	0.5611(8)	4.2(2)
C(31)	0.6926(8)	0.8749(7)	0.6078(9)	5.0(3)
C(32)	0.7174(9)	0.9132(8)	0.7278(10)	5.6(3)
C(33)	0.8203(10)	0.9403(7)	0.7526(8)	5.4(3)
C(34)	1.0664(6)	0.6872(5)	0.7072(6)	2.2(2)
C(35)	1.1343(6)	0.7292(6)	0.8188(6)	2.7(2)
C(36)	1.1338(8)	0.8344(8)	0.8598(7)	4.9(3)
C(37)	1.204(1)	0.8735(9)	0.9611(9)	6.4(3)
C(38)	1.2691(9)	0.803(1)	1.0211(8)	6.2(3)
C(39)	1.2703(10)	0.700(1)	0.9845(9)	7.2(3)
C(40)	1.2019(8)	0.6602(8)	0.8827(8)	5.3(3)
C(41)	1.2068(6)	0.6743(7)	0.6115(6)	3.0(2)
C(42)	1.2292(6)	0.7743(7)	0.6160(6)	3.3(2)
C(43)	1.3313(7)	0.7853(6)	0.6070(6)	3.3(2)
C(44)	1.4111(7)	0.7001(7)	0.5955(7)	3.6(2)
C(45)	1.3855(8)	0.6007(7)	0.5919(7)	4.3(3)
C(46)	1.2874(7)	0.5870(7)	0.5991(7)	3.9(2)

performed using the TEXSAN program package [15]. Some non-hydrogen atoms were refined anisotropically, while the rest were refined isotropically. Hydrogen

atoms were included but not refined. The final R factor was 0.027 ($R_w = 0.037$). The maximum and minimum peaks on the final difference Fourier map corresponded to $1.72 e^- \text{Å}^{-3}$ and $-0.74 e^- \text{Å}^{-3}$ respectively.

3.2.4. Supplementary material

Full tables of bond lengths and angles, hydrogen coordinates, thermal parameters and structure factors are available from the authors.

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