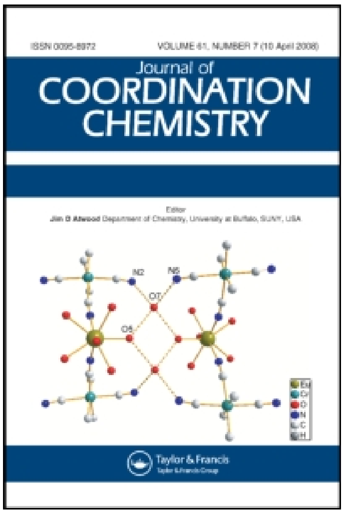

Journal of Coordination Chemistry
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713455674

Synthesis, Spectra and Crystal Structure of Tetrakis(Triphenylarsine Oxide)Iron(III)-μ-Oxo-Tribromoiron(III) Tetrabromoferrate(III)-Acetonitrile

Iveta Ondrejkovičová[a]; Viktor Vrábel[b]
[a] Department of Inorganic Chemistry, Slovak Technical University, Bratislava, Slovakia [b] Department of Analytical Chemistry, Faculty of Chemical Technology, Slovak Technical University, Bratislava, Slovakia

Online publication date: 15 September 2010

To cite this Article Ondrejkovičová, Iveta and Vrábel, Viktor(2002) 'Synthesis, Spectra and Crystal Structure of Tetrakis(Triphenylarsine Oxide)Iron(III)-μ-Oxo-Tribromoiron(III) Tetrabromoferrate(III)-Acetonitrile', Journal of Coordination Chemistry, 55: 3, 335 — 343

To link to this Article: DOI: 10.1080/00958970211888

URL: http://dx.doi.org/10.1080/00958970211888

J. Coord. Chem., 2002, Vol. 55(3), pp. 335 – 343

SYNTHESIS, SPECTRA AND CRYSTAL STRUCTURE OF TETRAKIS(TRIPHENYLARSINE OXIDE)IRON(III)-μ-OXO-TRIBROMOIRON(III) TETRABROMOFERRATE(III)-ACETONITRILE

IVETA ONDREJKOVIČOVÁ[a,]* and VIKTOR VRÁBEL[b]

[a]*Department of Inorganic Chemistry,* [b]*Department of Analytical Chemistry, Faculty of Chemical Technology, Slovak Technical University, Radlinského 9, 812 37 Bratislava, Slovakia*

(Received 24 October 2000)

The oxidation of triphenylarsine with dioxygen in reaction systems containing some iron compound and Br^- anions in acetonitrile leads to the formation of a novel unsymmetrical oxo-bridged diiron(III) complex $[(OAsPh_3)_4Fe(\mu\text{-}O)FeBr_3]^+[FeBr_4]^- \cdot CH_3CN$, where $OAsPh_3$ is triphenylarsine oxide. The title complex is also formed by direct reaction of iron(III) bromide and $OAsPh_3$ with dioxygen in acetonitrile solution. The crystal structure of the complex was solved by X-ray diffraction techniques. The cation contains two unsymmetrical species with an Fe—O—Fe bond angle of 159.2(2)°; one iron atom is pentacoordinated by four $OAsPh_3$ ligands and a μ-oxo ligand which connects the tetracoordinated Fe atom with the $FeBr_3O$ chromophore. Structural parameters and IR spectra of similar complexes are compared and discussed.

Keywords: Iron(III) complex; Unsymmetrical oxo-bridged complex; Triphenylarsine oxide; Oxidation by dioxygen; Crystal structure

INTRODUCTION

The (μ-oxo)diiron(III) unit has been the subject of great interest for several years. The studied complex is one of the very rare examples of a μ-oxo

*Corresponding author.

ISSN: 0095-8972 © 2002 Taylor & Francis Ltd DOI: 10.1080/00958970290005385

bridged diiron(III) system where each iron atom is located in a substantially different coordination sphere [1]. The title complex belongs to the family of unsymmetrical oxo-bridged Fe(III) complexes containing cations of the composition $[Fe_2O(OAsPh_3)_4X_3]^+$, where X = Cl [2, 3] or Br, with penta- and tetracoordinated iron atoms.

Complexes containing of $[Fe_2O(OAsPh_3)_4X_3]^+$ cations were prepared in similar ways, mainly by autocatalytic oxidation of $AsPh_3$ to $OAsPh_3$ by dioxygen in the presence of some iron(III) salts and X^- anions (X = Cl [2, 3] or Br). In this paper, we discuss and compare the structural parameters and IR spectra of these complexes.

EXPERIMENTAL

Analyses and Measurements

All chemicals used were of analytical grade. Iron(III) was determined chelatometrically with Chelaton 3 using sulphosalicylic acid. Carbon, hydrogen and nitrogen were determined by microanalytical methods (Carlo Erba Instruments EA 1108). Analytical data for solid samples are given in Table I. The yields of products are related to the initial metal content in reacting mixtures. Infrared spectra of the powdered samples were recorded in nujol mulls on KBr plates over the $4000-200\,cm^{-1}$ range using an FTIR-Magna 750 spectrophotometer (Nicolet) at room temperature with $4\,cm^{-1}$ resolution. Electronic spectra of acetonitrile solutions were recorded on a Specord 200 spectrophotometer (Analytik Jena). The measurements of dioxygen uptake were performed at constant temperature and constant atmospheric pressure of dioxygen in a gas burette apparatus using a procedure described previously [4].

TABLE I Analytical data[a] for $[Fe_2O(OAsPh_3)_4Br_3][FeBr_4]\cdot CH_3CN$ prepared by four methods

		Elemental analysis (%): *found*				
Method	*Starting compounds*	*Fe*	*C*	*H*	*N*	*Yield* (%)
1	Fe, HBr, $AsPh_3$, O_2	8.00	43.20	3.13	0.55	75
2	$FeBr_3$, $AsPh_3$, O_2	8.05	42.65	2.97	0.56	85
3	$Fe_2(SO_4)_3\cdot 7H_2O$, KBr, $AsPh_3$, O_2	8.13	42.44	3.00	0.52	55
4	$[FeBr_2(OAsPh_3)_4][FeBr_4]$, O_2	8.11	42.68	2.96	0.50	85
Calculated composition		8.08	42.88	3.06	0.68	

[a] Microanalysis results obtained with maximum deviations: Fe, ± 0.4; C, ± 0.4; H, ± 0.5; N, ± 0.4.

Preparation of $[Fe_2O(OAsPh_3)_4Br_3][FeBr_4] \cdot CH_3CN$

Method 1

Acetonitrile (ca. 30 cm^3) and 0.43 cm^3 of a 46% solution (by weight) of aqueous HBr (3.5 mmol) were added into a vessel containing excess iron powder (0.09 g, 1.6 mmol) and $AsPh_3$ (0.61 g, 2.0 mmol). The reaction mixture was stirred for 5 days at 60°C under an O_2 atmosphere. A small amount of insoluble solid was filtered off. When the resulting red-brown solution was allowed to stand at room temperature, microcrystals of the desired complex were obtained. The completion of the oxidation of $AsPh_3$ to $OAsPh_3$ was ascertained by comparing the electronic absorption spectrum of the reaction solution with that of pure $OAsPh_3$, which in acetonitrile exhibits four absorption bands with maxima at 252, 259, 262 and 270 nm.

Method 2

A solution of $AsPh_3$ (2.0 g, 6.5 mmol) in acetonitrile (ca. 30 cm^3) was added to $FeBr_3$ (1.5 g, 5.1 mmol) and the reaction mixture was stirred at 80°C under an O_2 atmosphere for two days. Then the mixture was filtered and well-formed orange needles suitable for X-ray analysis were obtained by leaving the filtrate to standing for several days at room temperature.

When the reaction mixture is stirred at lower temperatures and for shorter times, a mixture of two complexes is formed, *i.e.*, the title complex and $[FeBr_2(OAsPh_3)_4][FeBr_4]$ [5]. A similar mixture was also obtained when ethanol was used instead of acetonitrile.

Method 3

A mixture of excess $Fe_2(SO_4)_3 \cdot 7H_2O$ (0.53 g, 1.0 mmol), KBr (0.42 g, 3.5 mmol) and $AsPh_3$ (0.62 g, 2.0 mmol) in 40 cm^3 of acetonitrile was stirred and dioxygen was supplied continuously until all $AsPh_3$ was oxidized. The reaction took place at 70°C for about 5 days. After the oxidation, the K_2SO_4 as by-product and other insoluble solids were filtered out. The filtrate was cooled to room temperature and set aside for crystallization.

Method 4

A solution of $[FeBr_2(OAsPh_3)_4][FeBr_4]$ (0.40 g, 0.21 mmol) in 30 cm^3 of acetonitrile was stirred for 1 day at 60–70°C under an O_2 atmosphere. The resulting solution was filtered and set aside for crystallization.

TABLE II Crystal data and structure refinement details for $[(OAsPh_3)_4FeOFeBr_3][FeBr_4] \cdot CH_3CN$

Empirical formula	$C_{74}H_{63}As_4Br_7Fe_3NO_5$
Formula weight	2072.8
Temperature	293(2) K
Wavelength	0.71069 Å
Crystal system	Monoclinic
Space group	$P2_1/c$
Unit cell dimensions	$a = 13.858(8)$ Å
	$b = 21.818(10)$ Å
	$c = 25.677(18)$ Å
	$\beta = 90.64(5)°$
Volume	7763(8) Å^3
Z	4
ρ_c	1.774 mg/m^3
ρ_m	1.752 mg/m^3
Absorption coefficient	5.896 mm^{-1}
$F(000)$	4036
Crystal size	0.10 × 0.15 × 0.15 mm
Reflections collected	4976
Refinement method	Full-matrix least-squares on F^2
Final R indices $[I > 2\sigma(I)]$	$R1 = 0.056$, $wR2 = 0.090$
R indices (all data)	$R1 = 0.178$, $wR2 = 0.107$
Largest diff. peak and hole	0.565 and -0.357 eA^{-3}

TABLE III Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($A^2 \times 10^3$) for the title complex. U(eq) is defined as one third of the trace of the orthogonalized Uij tensor

	x/a	y/b	z/c	$U(eq)$
Fe(1)	7398(3)	8520(2)	1480(1)	43(1)
Fe(2)	7443(3)	10071(2)	1822(2)	69(1)
Fe(3)	7492(3)	4419(2)	717(2)	72(2)
Br(1)	8202(2)	10027(2)	2641(1)	101(1)
Br(2)	5921(3)	10449(2)	1939(2)	146(2)
Br(3)	8335(3)	10744(2)	1307(1)	151(2)
Br(4)	7112(3)	3391(1)	744(1)	90(1)
Br(5)	6471(3)	4871(2)	120(1)	104(1)
Br(6)	7187(3)	4807(2)	1542(1)	153(2)
Br(7)	9043(3)	4572(2)	513(2)	189(2)
As(1)	9710(2)	8097(2)	1313(1)	49(1)
As(2)	5139(2)	8024(2)	1538(1)	50(1)
As(3)	7744(2)	8043(1)	2719(1)	50(1)
As(4)	7112(2)	8227(1)	190(1)	51(1)
C(111)	9774(26)	7354(17)	1590(15)	77(16)
C(112)	10258(18)	7143(18)	2121(12)	66(12)
C(113)	10268(27)	6496(24)	2233(11)	106(17)
C(114)	9783(37)	6098(16)	1882(20)	116(18)
C(115)	9382(21)	6285(23)	1430(16)	91(16)
C(116)	9393(36)	6894(23)	1339(18)	143(25)
C(121)	10476(24)	8010(15)	713(11)	77(11)
C(122)	10303(19)	8485(12)	305(11)	53(9)
C(123)	10881(23)	8421(15)	−152(12)	69(12)
C(124)	11622(25)	8065(18)	−178(11)	62(11)
C(125)	11788(21)	7595(16)	213(14)	83(13)

TABLE III (Continued)

	x/a	y/b	z/c	$U(eq)$
C(126)	11185(25)	7560(14)	657(13)	83(13)
C(131)	10375(26)	8679(17)	1686(10)	49(10)
C(132)	9957(24)	9267(22)	1766(12)	86(13)
C(133)	10470(35)	9743(15)	2040(14)	97(14)
C(134)	11478(37)	9597(24)	2171(15)	137(23)
C(135)	11947(33)	9099(33)	2053(16)	207(32)
C(136)	11290(36)	8603(22)	1865(16)	148(22)
C(211)	5190(25)	7259(14)	1146(14)	50(10)
C(212)	5636(20)	6802(21)	1389(14)	68(11)
C(213)	5675(35)	6244(23)	1148(19)	144(21)
C(214)	5141(25)	6142(15)	712(20)	77(14)
C(215)	4723(19)	6675(24)	428(12)	65(11)
C(216)	4711(21)	7238(16)	642(13)	54(10)
C(221)	4490(23)	8646(16)	1174(10)	53(10)
C(222)	4997(22)	9231(17)	1174(10)	62(10)
C(223)	4523(23)	9729(17)	936(11)	77(12)
C(224)	3576(37)	9713(17)	730(20)	185(25)
C(225)	3168(29)	9174(19)	711(13)	136(21)
C(226)	3522(25)	8582(15)	960(11)	80(12)
C(231)	4233(21)	7801(20)	2107(14)	75(13)
C(232)	3829(26)	7246(16)	2144(14)	80(12)
C(233)	3132(35)	7174(24)	2528(17)	162(29)
C(234)	2989(43)	7498(31)	2903(22)	170(26)
C(235)	3445(31)	8029(26)	2896(19)	144(22)
C(236)	4139(22)	8223(13)	2504(19)	76(12)
C(311)	8819(18)	7946(17)	3047(10)	64(11)
C(312)	9526(24)	8424(20)	3100(9)	121(19)
C(313)	10315(32)	8413(15)	3370(14)	86(16)
C(314)	10690(27)	7752(37)	3508(16)	221(37)
C(315)	10013(34)	7430(19)	3640(11)	128(23)
C(316)	9095(29)	7382(14)	3323(13)	81(14)
C(321)	6933(18)	8532(14)	3130(15)	89(17)
C(322)	6426(27)	8927(16)	2879(14)	99(16)
C(323)	5800(27)	9344(14)	3128(15)	110(16)
C(324)	5787(22)	9266(19)	3678(15)	135(19)
C(325)	6490(32)	8982(18)	3955(11)	103(15)
C(326)	7040(34)	8541(18)	3656(14)	158(24)
C(331)	7282(29)	7235(19)	2643(15)	123(27)
C(332)	7509(34)	6670(26)	2379(12)	98(20)
C(333)	7128(41)	6154(16)	2414(15)	157(30)
C(334)	6461(23)	6094(27)	2772(21)	196(37)
C(335)	5938(30)	6655(28)	2961(15)	157(23)
C(336)	6456(35)	7173(13)	2916(16)	112(21)
C(411)	7811(19)	8882(14)	− 86(12)	57(10)
C(412)	8194(20)	9381(17)	186(10)	52(9)
C(413)	8754(25)	9862(14)	− 38(16)	88(13)
C(414)	8897(22)	9805(18)	− 563(15)	82(14)
C(415)	8624(22)	9300(15)	− 873(13)	72(12)
C(416)	8072(24)	8827(14)	− 644(11)	80(11)
C(421)	7797(36)	7513(20)	151(13)	122(22)
C(422)	7354(25)	6938(22)	334(11)	86(14)
C(423)	7825(25)	6387(15)	274(11)	55(10)
C(424)	8691(29)	6376(19)	− 5(11)	84(15)
C(425)	8990(28)	6912(21)	− 253(10)	91(16)

TABLE III (Continued)

	x/a	y/b	z/c	$U(eq)$
C(426)	8584(33)	7460(16)	– 182(13)	111(19)
C(431)	5982(24)	8199(21)	– 244(10)	70(12)
C(432)	5251(36)	8716(20)	– 230(14)	107(16)
C(433)	4446(27)	8638(19)	– 503(14)	108(18)
C(434)	4224(35)	8179(28)	– 826(18)	136(20)
C(435)	4919(34)	7720(22)	– 892(17)	111(18)
C(436)	5818(27)	7656(15)	– 577(12)	60(10)
O(1)	8589(10)	8309(6)	1166(5)	35(5)
O(2)	6196(10)	8239(7)	1753(6)	53(5)
O(3)	8054(11)	8264(7)	2115(5)	57(5)
O(4)	6726(10)	8346(6)	807(5)	41(5)
O(5)	7373(10)	9345(7)	1538(5)	41(5)
N(1)	2656	6600	1317	1008(116)
C(1)	2050	5600	1023	662(96)
C(2)	2312	6155	1170	375(43)

TABLE IV Selected bond lengths (Å) and angles (°) for $[(OAsPh_3)_4FeOFeBr_3][FeBr_4] \cdot CH_3CN$

Fe(1)—Fe(2)	3.496(4)	Fe(2)—Br(1)	2.342(5)
Fe(1)—O(5)	1.808(5)	Fe(3)—Br(7)	2.243(5)
Fe(1)—O(2)	1.915(5)	Fe(3)—Br(5)	2.297(5)
Fe(1)—O(1)	1.902(5)	Fe(3)—Br(4)	2.305(5)
Fe(1)—O(3)	1.938(5)	Fe(3)—Br(6)	2.324(5)
Fe(1)—O(4)	1.993(5)	As(1)—O(1)	1.659(6)
Fe(2)—O(5)	1.745(5)	As(2)—O(2)	1.628(6)
Fe(2)—Br(2)	2.289(5)	As(3)—O(3)	1.685(6)
Fe(2)—Br(3)	2.340(5)	As(4)—O(4)	1.698(6)
O(5)—Fe(1)—O(2)	105.8(6)	Br(2)—Fe(2)—Br(1)	107.6(2)
O(5)—Fe(1)—O(1)	107.1(6)	Br(3)—Fe(2)—Br(1)	107.4(2)
O(2)—Fe(1)—O(1)	147.1(6)	Br(7)—Fe(3)—Br(5)	111.4(2)
O(5)—Fe(1)—O(3)	103.1(7)	Br(7)—Fe(3)—Br(4)	111.8(2)
O(2)—Fe(1)—O(3)	90.1(6)	Br(5)—Fe(3)—Br(4)	107.3(2)
O(1)—Fe(1)—O(3)	83.5(6)	Br(7)—Fe(3)—Br(6)	110.0(3)
O(5)—Fe(1)—O(4)	104.6(6)	Br(5)—Fe(3)—Br(6)	109.6(2)
O(2)—Fe(1)—O(4)	81.7(6)	Br(4)—Fe(3)—Br(6)	106.5(2)
O(1)—Fe(1)—O(4)	89.2(5)	As(1)—O(1)—Fe(1)	141.7(8)
O(3)—Fe(1)—O(4)	152.3(6)	As(2)—O(2)—Fe(1)	138.7(9)
O(5)—Fe(2)—Br(2)	109.6(5)	As(3)—O(3)—Fe(1)	137.3(9)
O(5)—Fe(2)—Br(3)	111.1(5)	As(4)—O(4)—Fe(1)	133.6(8)
Br(2)—Fe(2)—Br(3)	109.9(2)	Fe(2)—O(5)—Fe(1)	159.6(8)
O(5)—Fe(2)—Br(1)	111.1(5)	N(1)—C(2)—C(1)	170.7(4)

X-ray Structure Determination

Data collection and cell refinement was carried out using Syntex $P2_1$ software. Intensity data were corrected to Lorentz and polarization factors using $XP2_1$ [6]. The structure was solved by the heavy atom method with SHELX86 [7] and subsequent Fourier synthesis using the same program.

Anisotropic thermal parameters were refined for all non-hydrogen atoms. Geometrical analysis was performed using SHELXL93 [8].

Structures were drawn using ORTEP [9]. Basic crystallographic data are summarized in Table II. Atomic coordinates and equivalent isotropic displacement parameters are listed in Table III. Selected interatomic distances and bond angles are given in Table IV.

Hydrogen atom coordinates, thermal parameters and list of observed and calculated structure factors are available on request from the corresponding author.

RESULTS AND DISCUSSION

The reaction of anhydrous $FeBr_3$ with $AsPh_3$ and dioxygen in acetonitrile at moderate temperatures proceeded to form a new (μ-oxo)diiron(III) complex $[(OAsPh_3)_4FeOFeBr_3][FeBr_4] \cdot CH_3CN$. This complex was also prepared by another methods (Tab. I) as was its isostructural analogous chloro-complex [3]. During autocatalytic oxidation of $AsPh_3$ to $OAsPh_3$ the reaction systems (methods 1–3) consumed O_2, as was demonstrated by measurements of dioxygen uptake. If excess $AsPh_3$ was used, free $OAsPh_3$ was generated besides the title complex. However, the oxidation of $AsPh_3$ was much slower than the oxidation of PPh_3 in similar systems [10]. This probably is caused by the lower reactivity of $AsPh_3$ and weaker energy of the As—O bond in comparison with PPh_3 [11].

The molecular structure of the title complex is shown in Figure 1. The crystal structure consists of the dimeric cation $[Fe_2O(OAsPh_3)_4Br_3]^+$, the $[FeBr_4]^-$ anion and an acetonitrile solvate molecule. The coordination polyhedron around the Fe(1) atom is a distorted tetragonal pyramid. Fe(1)—O interatomic distances varying from 1.808(5) to 1.993(5) Å and the bond angles from 81.7(6) to 107.1(6)°. As can be seen from Figure 1, the central Fe(1) atom is coordinated by four $OAsPh_3$ ligands which form a plane. The apical position is occupied by the bridged O(5) atom of the $OFeBr_3$ moiety. The Fe(1) atom is 0.511(4) Å from the O(1)—O(5) mean plane, on the same side as the μ-oxo ligand. The Fe(2) atom is tetrahedrally coordinated by three bromine atoms and the oxo-bridged ligand. The Fe(2)—O(5) bond of 1.745(5) Å is slightly shorter than the Fe(1)—O(5) bond of 1.808(5) Å, as expected. Both bond lengths, as well as the Fe—O—Fe angle, fall in the range of values observed for (μ-oxo)diiron analogues [1–3]. Bond lengths for Fe(1)—$OAsPh_3$ in the title complex (average 1.937(5) Å) are shorter than those observed in the

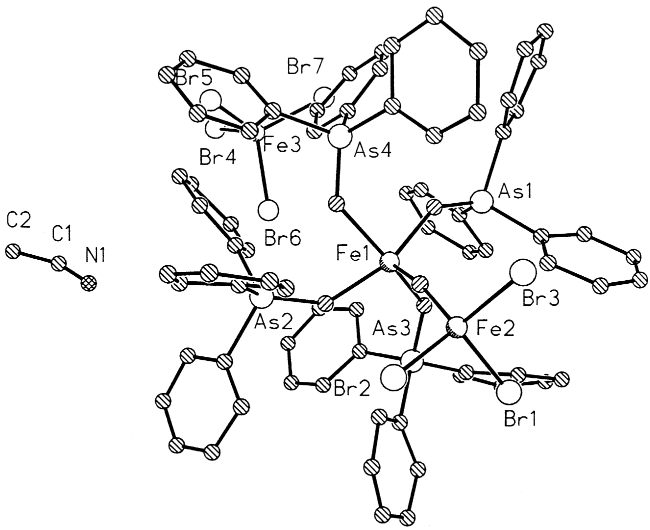

FIGURE 1 The molecular structure of $[(OAsPh_3)_4Fe(\mu\text{-}O)FeBr_3][FeBr_4] \cdot CH_3CN$.

$[Fe_2O(OAsPh_3)_4Cl_3]^+$ cations [2, 3] (average 1.981(5) Å). The mean Fe(2)—Br bond distance in the title complex (2.324(5) Å) is slightly shorter than that found in the $[(n5)FeOFeBr_3]^+$ cation (2.348(3) Å) [12]. The $[Fe(3)Br_4]^-$ anion shows significant distortion from regular tetrahedral coordination for which the Fe(3)—Br distances vary from 2.243(6) to 2.324(6) Å and the bond angles from 106.5(2) to 111.8(2)°. Similar distortions of tetrabromoferrate anion were observed for $[FeBr_2(OPPh_3)_4][FeBr_4]$ [13] and other species [14].

The dominant feature in the infrared spectrum of the title complex is the presence of three strong bands at about 850 cm^{-1}. Two absorption bands at 862 and 874 cm^{-1} are assigned to the stretching vibration ν(As—O). These bands are shifted to lower energies compared with free $OAsPh_3$ (880 cm^{-1}), as the result of its coordination [3]. Another strong band at 839 cm^{-1} is attributed to the asymmetric Fe—O—Fe stretching vibration (ν_{as}). The symmetric Fe—O—Fe stretch (ν_s) is located at 409 cm^{-1}. Bands assigned to the Fe—O—Fe stretching vibrations (ν_{as}, ν_s) are within the range expected for these monobridged structures [15]. The Fe—O—Fe stretching vibrations of $[Fe_2O(OAsPh_3)_4X_3]^+$, where X = Cl [2, 3] or Br, correlate with bond angles; the Fe—O—Fe angles can be predicted to within 10° from a knowledge of ν_{as} and ν_s. IR spectra of the title complex show a characteristic strong band at 292 cm^{-1} (ν_3) for $[FeBr_4]^-$. Another band at 214 cm^{-1} is assigned as ν(Fe—Br) for the $[Fe_2O(OAsPh_3)_4Br_3]^+$ cation [16].

In conclusion, we have synthesized and characterized a new unsymmetrical (μ-oxo)diiron(III) complex with $OAsPh_3$ and Br ligands. The Fe—O bonds, as well as the Fe—O—Fe angle for the $[Fe_2O(OAsPh_3)_4Br_3]^+$ cation and for its chloro-analogues fall in the range of values observed for oxo-bridged diiron(III) complexes and correlate with their Fe—O—Fe vibrations [1, 15].

Acknowledgements

This work was financially supported by grant 1/6106/99 of the Slovak Grant Agency for Science.

References

[1] M. Melník, V. Vančová, I. Ondrejkovičová and C. E. Holloway, *Rev. Inorg. Chem.* **18**, 1 (1998).
[2] I. Ondrejkovičová, T. Lis, J. Mrozinski, V. Vančová and M. Melník, *Polyhedron* **17**, 3181 (1998).
[3] I. Ondrejkovičová, T. Lis, J. Mrozinski, V. Vančová and M. Melník, *Inorg. Chim. Acta* **277**, 127 (1998).
[4] V. Vančová, I. Ondrejkovičová and G. Ondrejovič, *Chem. Zvesti* **38**, 363 (1984).
[5] I. Ondrejkovičová, D. Mikloš and V. Vrábel, In: *Coordination Chemistry at the Turn of the Century*, **4**, G. Ondrejovič and A. Sirota, Eds. (Slovak Technical University Press, Bratislava, Slovakia, 1999), 115.
[6] F. Pavelčík, *Program $XP2_1$* (Comenius University, Pharmaceutical Faculty Bratislava, Slovakia, 1993).
[7] G. M. Sheldrick, SHELXS-86, *Crystallographic Computing*, Vol. 3 (Oxford University Press, Oxford, 1985), p. 175.
[8] G. M. Sheldrick, SHELXL93, *Program for Refinement of Crystal Structures* (University of Göttingen, Germany, 1994).
[9] C. K. Johnson, ORTEP, *Report ORNL-3794* (Oak Ridge National Laboratory, TN, 1965).
[10] I. Ondrejkovičová, V. Vančová and G. Ondrejovič, *Stud. Surf. Sci. Catal.* **66**, 649 (1991).
[11] C. Srinivasan and K. Pitchumani, *Can. J. Chem.* **63**, 2285 (1985).
[12] P. Gómez-Romero, E. H. Witten, W. M. Reiff, G. Backes, J. Sanders-Loehr and G. B. Jameson, *J. Am. Chem. Soc.* **111**, 9039 (1989).
[13] E. Ďurčanská, T. Glowiak, E. Gyepes, I. Ondrejkovičová and G. Ondrejovič, *Acta Facultatis Rerum Natur. Univ. Comenianae Chimia* **39**, 3 (1991).
[14] M. Melník, I. Ondrejkovičová, V. Vančová and C. E. Holloway, *Rev. Inorg. Chem.* **17**, 55 (1997).
[15] D. M. Kurtz Jr., *Chem. Rev.* **90**, 585 (1990).
[16] S. A. Cotton and J. F. Gibson, *J. Chem. Soc. A* p. 859 (1971).