SYNTHESIS AND X-RAY STUDY OF TRIFERROCENYLIODIDEPHOSPHONIUM TRIIODIDE

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Summary

A series of iodine derivatives of ferrocenylphosphorus compounds has been obtained. An X-ray investigation of $[Fc_3PI]^+I_3^-$ has been carried out. All ferrocenyl fragments possess close geometric parameters, similar to those of ferrocene and its derivatives. The positive charge of the cation is localized on the phosphorus atom. Symmetrical I_3^- anions of linear configuration form zigzag-shaped chains, as is usual for triiodides. The cation—anion interaction is realized through short I···I contacts.

Introduction

According to the reported data, ferrocene and its derivatives form adducts with iodine containing 3 to 20 iodine atoms per ferrocenyl residue (independent of the amount of iodine used [1]). There are some contradictory reports on the structures of the ferrocene-iodine derivatives [2].

We considered it of interest to obtain iodine derivatives of ferrocenylphosphorus compounds of the type: Fc_nPL_{3-n}, where n = 2, 3; L = C_6H_5; Fc = —$C_5H_4FeC_5H_5$ and to examine their structure.

To obtain the iodine derivatives, to a benzene solution of the corresponding ferrocenylphosphorus compound * an ether solution of iodine was gradually added at 50°C. After cooling to 0°C a crystalline precipitate settled out. It was separated and thoroughly washed off from the starting materials with absolute ether. At equimolar ratio of Fc_3P and iodine a yellow adduct was separated, which contained two iodine atoms, and at the ratio equal to 1:2 beautiful dark-

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* Triferrocenylphosphine and diferrocenylphosphine were synthesized according to Nesmeyanov [3] and Sollott [4].

red crystals of triferrocenyliodidephosphonium triiodide were obtained. Diferrocenylphenylphosphine treated with an equimolar quantity of iodine gave an adduct containing two iodine atoms.

The composition and structure of the iodine derivatives obtained were confirmed by elemental analysis and IR spectroscopy. The characteristic bands of ferrocenylphosphorus compounds [5] are present in the IR spectra of these substances. To determine the structure of $[Fc_3PI]^+I_3^-$, an X-ray crystal study was carried out.

Results and discussion

Crystals of $\{[(\eta^5\text{-}C_5H_5)Fe(\eta^5\text{-}C_5H_4)]_3PI\}^+I_3^-$ are orthorhombic, $a = 12.034(6)$, $b = 15.291(6)$, $c = 17.722(9)$ Å, $V = 3261.1$ Å^3, $M = 1094$, $d_{calc.} = 2.23$ g/cm^3, $Z = 4$ $C_{30}H_{27}Fe_3PI_4$, space group $P2_12_12_1$.

The atomic coordinates, corresponding to the correct absolute structure, and their thermal parameters are listed in Tables 1 and 2, bond length values and the geometry of the cation are given in Fig. 1, Fe—C(Cp) bond lengths in Table 3, and bond angles in Table 4.

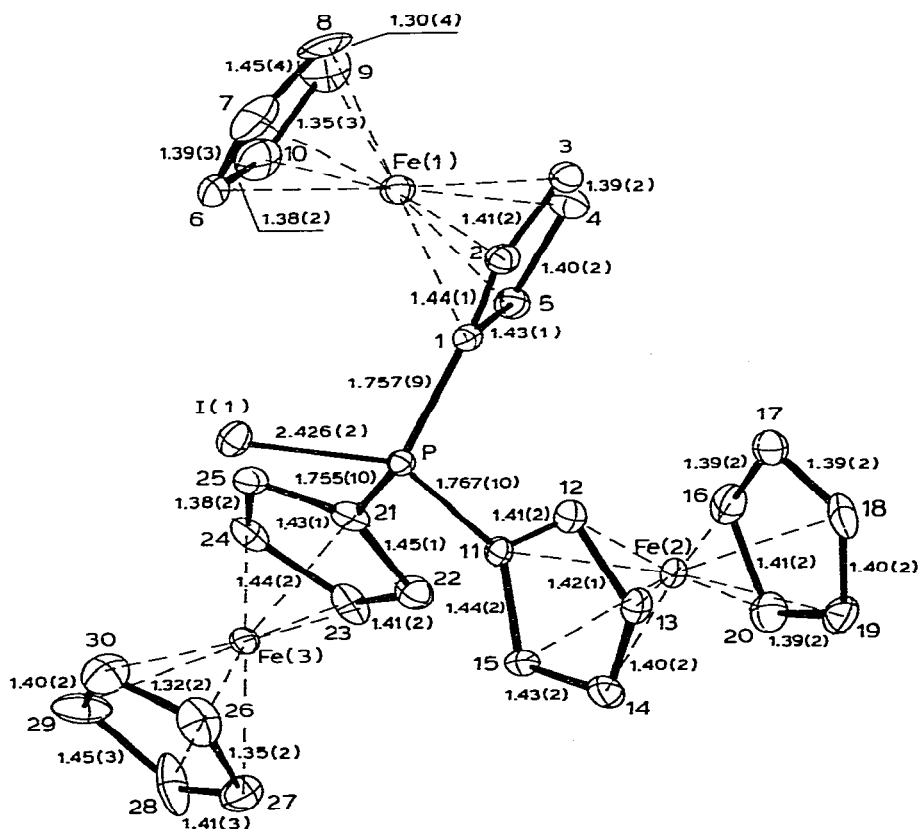The P atom in the triferrocenyliodidephosphonium cation has a slightly dis-

Fig. 1. Geometry of cation with bond lengths.

TABLE 1. FRACTIONAL ATOMIC COORDINATES OF NON-HYDROGEN ATOMS (X 10^4, for I and Fe X 10^5) AND ANISOTROPIC THERMAL PARAMETERS (X 10^4, for I and Fe X 10^5. The anisotropic thermal parameters are in the form $T = \exp[-1/4(B_{11}h^2 a^{*2} + ... + 2B_{23}klb^* c^*)]$.)

Atom	x	y	z	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
I(1)	−26564(5)	3523(4)	57880(4)	379(4)	394(3)	374(3)	54(6)	73(6)	71(5)
I(2)	−91862(6)	−38240(5)	27104(4)	644(5)	371(3)	251(2)	66(6)	−73(6)	−14(5)
I(3)	−77370(8)	−24868(7)	20913(6)	894(7)	633(5)	524(4)	−460(10)	−172(9)	388(8)
I(4)	−104954(7)	−52263(6)	33664(6)	729(6)	534(4)	592(5)	−114(9)	228(8)	278(8)
Fe(1)	−13410(10)	−17412(9)	45103(9)	780(10)	287(6)	244(5)	−240(10)	−60(10)	−29(9)
Fe(2)	12190(10)	14684(9)	46866(8)	418(9)	319(6)	225(5)	−60(10)	60(10)	10(9)
Fe(3)	−110(10)	−9(9)	73923(8)	615(9)	336(6)	174(5)	−40(10)	30(20)	−42(9)
P	−701(2)	145(1)	5506(1)	37(1)	25(1)	18(1)	−1(2)	−1(2)	1(1)
C(1)	−599(7)	−575(6)	4736(5)	41(6)	27(4)	20(3)	−12(8)	−6(7)	2(6)
C(2)	−1178(9)	−532(7)	4029(6)	71(8)	35(5)	24(4)	−20(10)	−20(9)	8(7)
C(3)	−700(10)	−1167(9)	3554(7)	110(10)	52(6)	27(4)	−40(20)	0(10)	−6(9)
C(4)	130(10)	−1599(8)	3946(7)	90(10)	40(5)	32(5)	−10(10)	20(10)	−29(9)
C(5)	213(9)	−1255(7)	4672(7)	58(8)	36(5)	33(4)	0(10)	−4(9)	−14(8)
C(6)	−2470(10)	−2102(9)	5318(9)	110(10)	46(6)	44(6)	−60(20)	40(20)	−10(10)
C(7)	−2980(10)	−2010(10)	4620(10)	70(10)	80(10)	80(20)	−80(20)	−40(20)	60(20)
C(8)	−2400(30)	−2640(20)	4150(10)	360(60)	120(20)	47(9)	−380(70)	−10(40)	−10(20)
C(9)	−1650(20)	−3042(9)	4540(20)	250(30)	25(6)	90(20)	−50(20)	130(40)	−40(20)
C(10)	−1650(10)	−2730(9)	5257(9)	140(20)	50(7)	46(7)	−40(20)	−20(20)	30(10)
C(11)	−186(7)	1189(6)	5260(6)	42(6)	33(4)	24(3)	−2(9)	4(8)	1(7)
C(12)	−439(8)	1610(6)	4572(6)	46(6)	32(4)	25(4)	0(9)	2(8)	6(7)
C(13)	66(9)	2450(6)	4572(7)	69(8)	20(4)	44(5)	9(9)	30(10)	19(8)
C(14)	630(10)	2547(7)	5257(7)	90(10)	29(4)	30(4)	−10(10)	30(10)	−8(8)
C(15)	482(9)	1781(7)	5703(7)	71(8)	36(4)	26(4)	−20(10)	4(9)	−9(7)
C(16)	2252(9)	416(7)	4561(8)	59(8)	33(5)	53(6)	10(10)	30(10)	16(9)
C(17)	1740(10)	634(9)	3884(9)	62(9)	66(8)	42(6)	−20(10)	30(10)	−30(10)
C(18)	2071(9)	1482(9)	3699(7)	65(9)	74(8)	25(4)	20(10)	30(10)	19(9)
C(19)	2754(9)	1794(8)	4281(9)	52(7)	48(6)	55(6)	−30(10)	30(10)	0(10)
C(20)	2861(8)	1141(9)	4821(8)	44(7)	67(7)	40(5)	10(10)	30(10)	20(10)
C(21)	−22(8)	−318(6)	6285(5)	55(6)	30(4)	18(3)	11(9)	−5(8)	−8(6)
C(22)	1106(8)	−146(8)	6532(7)	37(7)	59(6)	30(4)	10(10)	0(8)	−11(9)
C(23)	1370(10)	−731(9)	7118(7)	80(10)	59(7)	20(4)	60(10)	−20(10)	−3(9)
C(24)	410(10)	−1275(8)	7237(7)	140(10)	39(5)	24(4)	30(20)	−20(10)	9(8)
C(25)	−410(10)	−1032(6)	6734(6)	82(9)	28(4)	21(3)	−10(10)	−16(9)	−2(7)
C(26)	−880(10)	1110(10)	7583(8)	110(10)	64(8)	30(5)	60(20)	0(10)	−30(10)
C(27)	140(10)	1207(9)	7896(8)	110(10)	50(6)	28(5)	−40(20)	50(10)	−30(10)
C(28)	270(10)	530(20)	8426(9)	90(10)	90(10)	25(7)	10(30)	−50(20)	−50(20)
C(29)	−770(20)	50(10)	8409(9)	180(20)	49(7)	24(5)	−30(20)	60(20)	0(10)
C(30)	−1450(10)	460(10)	7876(8)	80(10)	71(8)	37(6)	0(20)	40(10)	−20(10)

TABLE 2

POSITIONAL PARAMETERS OF HYDROGEN ATOMS ($\times 10^3$)

Atom	x	y	z	Atom	x	y	z
H(2)	−166(9)	−10(7)	386(6)	H(17)	140(10)	35(8)	370(7)
H(3)	−107(9)	−126(7)	305(6)	H(18)	178(9)	181(8)	332(6)
H(4)	49(9)	−199(8)	375(7)	H(19)	296(9)	239(8)	439(6)
H(5)	64(9)	−137(8)	504(7)	H(20)	328(9)	118(7)	536(7)
H(6)	−248(9)	−184(7)	582(7)	H(22)	164(9)	18(7)	629(7)
H(7)	−350(10)	−171(8)	465(7)	H(23)	202(9)	−78(7)	746(6)
H(8)	−270(10)	−245(9)	392(8)	H(24)	29(9)	−166(8)	756(7)
H(9)	−113(9)	−350(7)	408(6)	H(25)	−109(9)	−127(7)	676(7)
H(10)	−117(9)	−272(7)	583(7)	H(26)	−97(9)	146(8)	719(6)
H(12)	−87(9)	136(7)	419(7)	H(27)	29(9)	166(7)	781(6)
H(13)	−13(9)	290(7)	409(6)	H(28)	70(10)	50(10)	858(8)
H(14)	115(9)	294(7)	541(6)	H(29)	−80(10)	−48(8)	852(7)
H(15)	92(9)	167(8)	612(7)	H(30)	−227(9)	4(7)	785(6)
H(16)	217(8)	−17(7)	485(6)				

torted tetrahedral coordination. The values of the angles CPC and CPI vary from 105.8(3) to 113.3(5)° (average 109(3)°). The distortion is, apparently, due to the presence in the cation of several noncovalent C···C and C···I contacts, shortened in comparison to the normal Van der Waals C···C distance of 3.42 Å [6] and the sum of the Van der Waals radii of C and I atoms of 4.0 Å [7]: C(1)···C(12) 3.36(1), C(5)···C(21) 3.21(2), C(15)···C(22) 3.38(2), I(1)···C(2) 3.84(1), I(1)···C(25) 3.82(1), I(1)···C(6) 3.85(1) and I(1)···C(18) 3.93(1) Å. Similar angle deviations from the ideal value of 109.5° at the P atom are observed, for example, in the benzyltriphenylphosphonium cation [8,9] and, in general, are characteristic of organic phosphonium cations.

The P—I bond length of 2.426(2) Å is substantially shorter than in the molecule of phosphorus triiodide (2.463 Å) [10]. The P—C bond lengths have practically equal values (average 1.763(6) Å) and are clearly lengthened in com-

TABLE 3

Fe—C BOND LENGTHS d (Å) IN THE CATION

Bond	d	Bond	d
Fe(1)—C(1)	2.04(1)	Fe(2)—C(16)	2.05(1)
Fe(1)—C(2)	2.05(1)	Fe(2)—C(17)	2.01(2)
Fe(1)—C(3)	2.06(1)	Fe(2)—C(18)	2.03(1)
Fe(1)—C(4)	2.04(1)	Fe(2)—C(19)	2.04(1)
Fe(1)—C(5)	2.03(1)	Fe(2)—C(20)	2.05(1)
Fe(1)—C(6)	2.05(2)	Fe(3)—C(21)	2.02(1)
Fe(1)—C(7)	2.02(2)	Fe(3)—C(22)	2.04(1)
Fe(1)—C(8)	1.98(4)	Fe(3)—C(23)	2.06(1)
Fe(1)—C(9)	2.02(2)	Fe(3)—C(24)	2.03(1)
Fe(1)—C(10)	2.04(2)	Fe(3)—C(25)	2.02(1)
Fe(2)—C(11)	2.02(1)	Fe(3)—C(26)	2.02(2)
Fe(2)—C(12)	2.02(1)	Fe(3)—C(27)	2.06(1)
Fe(2)—C(13)	2.05(1)	Fe(3)—C(28)	2.03(2)
Fe(2)—C(14)	2.06(1)	Fe(3)—C(29)	2.02(2)
Fe(2)—C(15)	2.06(1)	Fe(3)—C(30)	2.06(2)

TABLE 4

BOND ANGLES ω (degrees)

Angle	ω	Angle	ω
I(3)I(2)I(4)	175.89(4)	C(11)C(12)C(13)	108.6(9)
I(1)PC(1)	108.1(3)	C(12)C(13)C(14)	107.7(10)
I(1)PC(11)	105.8(3)	C(13)C(14)C(15)	109.5(10)
I(1)PC(21)	110.0(3)	C(14)C(15)C(11)	106.5(9)
C(1)PC(11)	110.5(4)	C(17)C(16)C(20)	108.9(11)
C(1)PC(21)	109.0(4)	C(16)C(17)C(18)	107.6(12)
C(11)PC(21)	113.3(5)	C(17)C(18)C(19)	108.0(12)
PC(1)C(2)	127.9(7)	C(18)C(19)C(20)	108.5(12)
PC(1)C(5)	124.3(7)	C(19)C(20)C(16)	107.0(11)
C(2)C(1)C(5)	107.2(9)	PC(21)C(22)	126.9(8)
C(1)C(2)C(3)	107.2(9)	PC(21)C(25)	126.3(8)
C(2)C(3)C(4)	108.7(11)	C(22)C(21)C(25)	106.2(9)
C(3)C(4)C(5)	109.6(11)	C(21)C(22)C(23)	108.5(10)
C(4)C(5)C(1)	107.3(10)	C(22)C(23)C(24)	107.1(11)
C(7)C(6)C(10)	108.7(15)	C(23)C(24)C(25)	108.9(12)
C(6)C(7)C(8)	102.9(19)	C(24)C(25)C(21)	109.2(10)
C(7)C(8)C(9)	110.3(26)	C(27)C(26)C(30)	113.2(14)
C(8)C(9)C(10)	109.4(24)	C(26)C(27)C(28)	107.1(14)
C(9)C(10)C(6)	108.7(17)	C(27)C(28)C(29)	105.5(16)
PC(11)C(12)	123.3(7)	C(28)C(29)C(30)	106.5(16)
PC(11)C(15)	128.9(8)	C(29)C(30)C(26)	107.7(14)
C(12)C(11)C(15)	107.7(9)		

parison to ylide P—C(Cp) bonds, e.g. 1.718 Å in triphenylphosphonium cyclopentadienylide [11]. On the other hand, these bonds are noticeably shorter compared to the P—C(Ph) distances of 1.828 Å in triphenylphosphine [12] and P—C(Cp) 1.802 Å in the diphenylferrocenylphosphine ligand [13]. This shortening reflects, perhaps, some contribution from $p_\pi(C)—d_\pi(P)$ interaction.

The values of the C—C bond lengths in the Cp rings, bound to the P atom vary in the range of 1.38(2)—1.45(1) Å (average 1.42(2) Å), and in unsubstituted Cp rings from 1.30(4)—1.45(4) Å, with a somewhat lower average value (1.39(4) Å), which is caused by more intense thermal vibrations of the carbon atoms of the unsubstituted rings in the periphery of the cation. The Fe—C distances of 1.98(4)—2.06(1) Å (average 2.04(2) Å) are close to the values obtained for ferrocene [14] and its derivatives [15].

All the Cp rings are planar within 0.015 Å. In each ferrocenyl nucleus the rings are not perfectly parallel which is common for crystalline unbridged substituted ferrocene derivatives: the dihedral angles are 7.7, 5.1 and 3.8° for the sandwiches with atoms Fe(1), Fe(2) and Fe(3), respectively.

Noncovalent C···C distances between the Cp rings vary within approximately the same limits (from 3.21(4) to 3.41(2) Å), the substituted C atoms having the "longest" contacts. The P atom displacement from the Cp-ring planes is 0.21, —0.06 and 0.17 Å in the direction away from the Fe(1), Fe(2) and Fe(3) atoms.

A difference is observed between the conformations of the ferrocenyl fragments, which is described by the angles of mutual rotation of the Cp rings, φ. If the ferrocenyl group, which includes Fe(1) atom, has a conformation close to the ideal eclipsed (average $\varphi_1 = 1.7°$), then the sandwiches including the Fe(2) and Fe(3) atoms are in the intermediate conformations ($\varphi_2 = 10.7$ and

$\varphi_3 = 16.0°$), but closer to the eclipsed. These data confirm the conclusion [16], that molecules of the majority of unbridged ferrocene derivatives tend to the eclipsed conformation with $\varphi < 17°$.

The distances between cyclopentadienyl ring centers in all the ferrocenyl fragments are the same and are equal to 3.30 Å, which is in good agreement with the data on ferrocene in the gaseous phase (3.319(15) Å [17]). The Fe atoms are slightly displaced from the lines joining the centers of the Cp rings of each sandwich: angles A—Fe—A' (A and A' are the centroids of the Cp rings) are in the range 175.1—177.4°.

Thus, all the ferrocenyl fragments of the cation have similar geometric characteristics (except for the differences in angle φ, which can be caused by the action of the crystal field) close to the corresponding parameters of ferrocene itself and its unbridged derivatives. In contrast to the complexes with metal atoms of mixed oxidation state, e.g., diferrocenylselenium [18] and ferricenyltris(ferrocenyl)borate [19] cations, in which one of the ferrocenyl moieties is
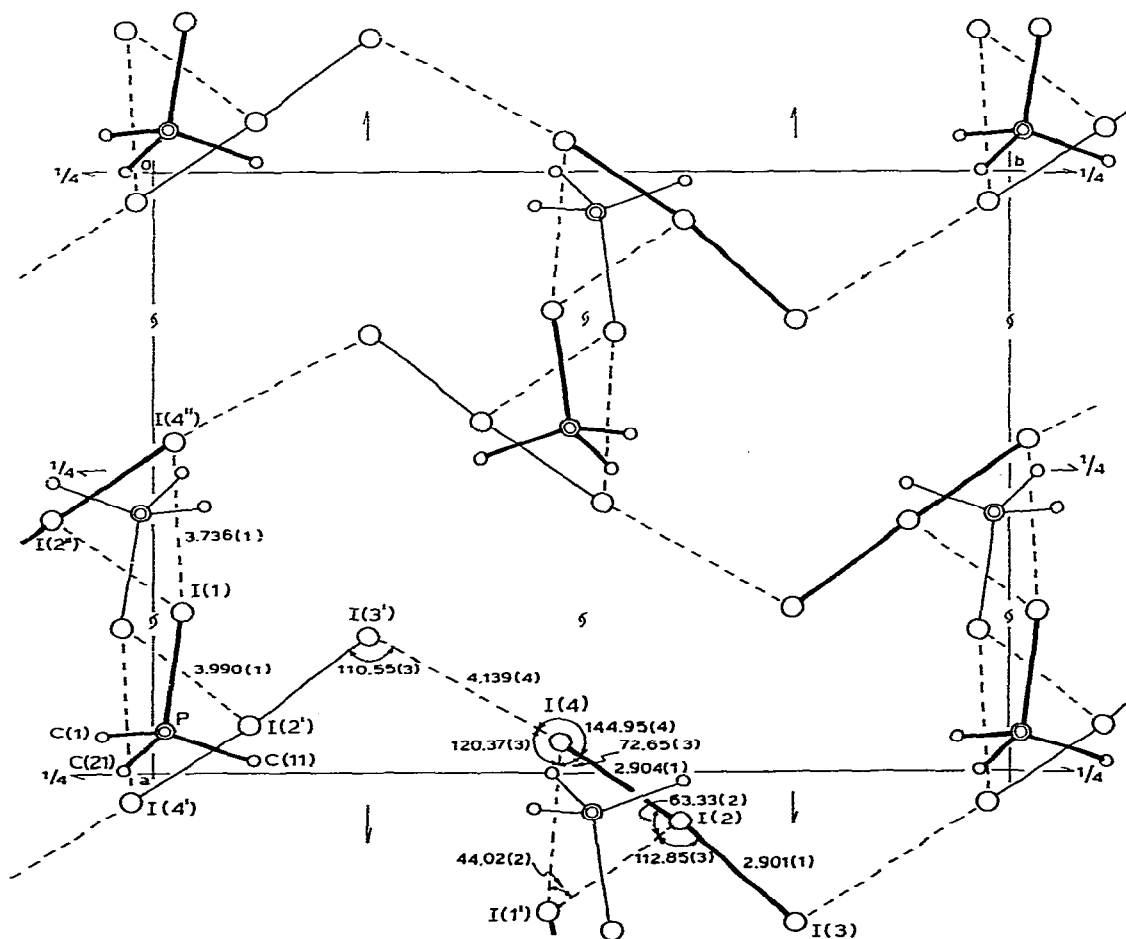

Fig. 2. Projection of the crystal structure down c. Only the neighbouring coordination about the P atoms in the cation is shown.

transformed into a ferricenyl fragment (with increase of the formal oxidation state of the central atom from $Fe^{2+} \to Fe^{3+}$, which leads to a slight increase in the distance between the centers of the Cp rings), in our case, the positive charge is not delocalized over the ferrocenyl moieties, but is mainly concentrated on the P atom.

The I_3^- anion has quite a usual configuration close to linear [20] (angle I(3)I(2)I(4) is equal to 175.89(4)°). The bond lengths I(2)—I(3), 2.901(1) Å, and I(2)—I(4), 2.904(1) Å, are virtually the same, i.e. the I_3^- anion has a symmetrical structure. It is interesting to note that according to the reported data, symmetrical I_3^- anions, as a rule, occupy special positions (with symmetry C_i, C_2, C_s) [20—25], although in the hydridobisbenzamide triiodide structure [26] nearly symmetrical I_3^- anions are present (distances I—I 2.924 and 2.934; 2.935 and 2.939 Å in two independent anions), which are situated, as in our case, in general positions.

The distance D between terminal I atoms is equal to 5.802(1) Å, which contradicts the statement [21] that the value of $D \sim 5.87$ Å is common for all the I_3^- anions. This structure supports the assumptions [27—29], that the value of D decreases with an increase of the cation dimensions, and that at small D values (~ 5.80 Å) the existence of a symmetrical I_3^- anion is energetically more favourable. The fact that the I—I bond lengths in the previously studied symmetrical I_3^- anions vary greatly from the value of 2.920 Å characteristic of an "isolated" (i.e. not forming short contacts with the environment) I_3^- anion (for example, in $[(C_6H_5)_4As]^+I_3^-$ [20]) is explained [20] by the influence of short interanion contacts, corresponding to the secondary I$\cdots$I interactions (see below); the increase of the I—I bond length is caused by contacts with linear configuration I—I—I$\cdots$I, whereas contacts with angular configuration I—I—I

$$\vdots$$

$$I$$

give rise to the shortening of the I—I bond. The latter case is also observed in the structure under study.

The I_3^- anions, connected by weak I(3)$\cdots$I(4) interactions (4.139(1) Å: doubled Van der Waals radius of I atom = 4.3 Å[7]), form infinite zigzag-shaped chains around screw axes of the type $2_1/0; y; 1/4 /$ (Fig. 2). Each chain of I_3^- anions is surrounded by four identical chains, related to the central chain by the $2_1/\frac{1}{4}; 0; z/$ - operation. The cations are positioned on both sides of each anionic chain and participate in the secondary interactions with it (3.990(1) and 3.736(1) Å for I(1)$\cdots$I(2″) and I(1)$\cdots$I(4″), respectively).

Experimental

To a solution of 0.12 g (0.0002 mol) of triferrocenylphosphine in 10 ml of dry benzene a solution of 0.05 g (0.0002 mol) of iodine in 30 ml of absolute diethylether was gradually added at 50°C over a period of 30 minutes. Then the reaction mass was cooled to 0°C, yellow precipitate of triferrocenyliodide-phosphonium iodide was filtered off, thoroughly washed with diethyl ether and dried in vacuo. Weight 0.14 g (81% yield). M.p. 209°C (dec.).

Anal. Found: C, 43.03; H, 3.44; Fe, 18.82; I, 29.88. Calcd. for $C_{30}H_{27}Fe_3PI_2$: C, 42.91; H, 3.24; Fe, 19.93; I, 30.22%.

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Similarly, the interaction of 0.6 g (0.001 mol) of triferrocenylphosphine and 0.51 g (0.002 mol) of iodine yielded a dark red precipitate of triferrocenyl-iodidephosphonium triiodide. Weight 0.84 g (75% yield). M.p. 195°C (dec.).

Anal. Found: C, 32.53; H, 2.57; Fe, 14.88; I, 45.86. Calcd. for $C_{30}H_{27}Fe_3PI_4$; C, 32.90; H, 2.49, Fe, 15.53; I, 46.42%.

Diferrocenylphenyliodidephosphonium iodide was obtained from 0.1 g (0.0002 mol) of diferrocenylphosphine and 0.05 g (0.0002 mol) of iodine under similar conditions. Weight 0.092 g (61% yield). M.p. 175°C (dec.).

Anal. Found: C, 42.44; H, 3.32; I, 33.81. Calcd. for $C_{26}H_{23}Fe_2PI_2$; C, 42.70; H, 3.17; I, 34.72%.

The cell parameters and reflection intensities were measured on a four-circle automatic Syntex P2$_1$ diffractometer at 20°C (λMo-K_α, graphite monochromator, $2\theta \leqslant 57°$, $\theta/2\theta$ scan technique). The structure was solved and refined using 2912 independent reflections.

The structure was solved by direct methods using the MULTAN program set; absorption correction for the real shape of the crystal was carried out according to a published method [30].

A full-matrix least-squares isotropic, and then anisotropic (H atoms were not taken into account) refinement gave $R = 0.035$. The H atoms were placed in the geometrically calculated positions and considered in the refinement with fixed isotropic thermal factors ($B_{iso} = 4$ Å^2). At the final stage of refinement anomalous scattering by the I, Fe and P atoms was taken into account and the final values of the discrepancy factors were obtained: $R = 0.029$ ($R_w = 0.034$) for the initial and $R = 0.027$ ($R_w = 0.032$) for the inverted structure. According to the Hamilton test, the inverted structure with 99.5% probability is absolute.

The modified EXTL * program set was used for all calculations with an Eclipse S/200 computer.

Acknowledgement

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