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Triorganoarsenic(V) compounds with internally functionalized oximes: synthetic and spectroscopic aspects of $[R_3As(Cl)L]$, $[R_3As(OH)L]$ and $[R_3AsL_2]$: crystal and molecular structure of $[Pr^i_3AsOH]^+Cl^-$

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Abstract

Triorganoarsenic(V) oximates of the type $[R_3As\{ON{=}C(Me)Ar\}_2]$ (**1**) ($R = Pr^i$, Bu^i; $Ar = C_5H_4N$-2, C_4H_3O-2) are formed by the reactions of R_3AsCl_2 with the sodium salts of internally functionalized oximes in 1:2 molar ratio in anhydrous benzene. The redistribution products $[R_3As(X)\{ON{=}C(Me)Ar\}]$ (**2**) (X = Cl, Br, OH) are obtained by treatment of **1** with equimolar R_3AsX_2. $[Pr^i_3As(OH)\{ON{=}C(Me)C_5H_4N\text{-}2\}]$ may also be obtained by the reaction of $Pr^i_3As(OH)_2$ with the corresponding oxime in 1:1 molar ratio. All of these complexes are characterized by IR and NMR (^1H and ^{13}C) spectroscopy and elemental analyses. Controlled hydrolysis of a representative monochloro-complex $[Pr^i_3As(Cl)\{ON{=}C(Me)C_4H_3O\text{-}2\}]$ yields crystals of $Pr^i_3As(OH)Cl$ in which single crystal X-ray diffraction indicates that there is a distorted tetrahedral environment around arsenic.

Keywords: Trialkylarsenic(V); Oximes; NMR; X-ray

1. Introduction

There has been a renewal of interest in organoarsenic compounds [1] arising from their wide ranging applications, which include their use as precursors for chemical vapor deposition [2] and as versatile ligands, along with their biocidal/medicinal aspects. The trivalent compounds show considerable structural diversity, which is extended by the presence of a stereochemically active lone pair [1]. By contrast, pentavalent compounds, with the exception of tetraorganoarsonium derivatives, often adopt a trigonal bipyramidal geometry [3–7].

Internally functionalized oximes are known to stabilize diverse stereochemistries [8–10]. There have been several investigations on oximates of arsenic(III) [11–15] but studies with arsenic(V) are scanty [16]. In view of this and in persuance of our work on organometallic compounds of Group 15, we have prepared triorganoarsenic(V) compounds derived from internally functionalized oximes.

2. Results and discussion

The reaction of R_3AsCl_2 with the sodium salt of 2-heteroaryl methylketone oxime in 1:2 molar ratio yields $[R_3As\{ON{=}C(Me)Ar\}_2]$ (**1**) [$R = Pr^i$, Bu^i; $Ar = C_5H_4N$-2, C_4H_3O-2]. Compounds **1** on treatment with one equivalent of R_3AsX_2 give the redistribution products $[R_3As(X)\{ON{=}C(Me)Ar\}]$ (**2**) (X = Cl, Br or OH). Reactions involving halide derivatives take place instantaneously, whereas the corresponding redistribution reaction of $[Pr^i_3As\{ON{=}C(Me)C_5H_4N\text{-}2\}_2]$ with Pr^i_3A-

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s(OH)$_2$ is relatively slow. The hydroxo complex can also be prepared by the reaction of $Pr^i_3As(OH)_2$ with free oxime in a 1:1 molar ratio. All these complexes are orange to brown liquids/pastes that are soluble in common organic solvents (Scheme 1).

The interpretation of the IR spectra of these triorganoarsenic(V) complexes was carried out by comparison with the spectra of free oximes, R_3AsCl_2 (R = Pr^i, Bu^i), $Pr^i_3As(OH)_2$ and other related complexes [17]. A medium to strong intensity band in the region 580–670 cm^{-1} is assigned to νAs–C and a band of variable intensity in the region 427–435 cm^{-1} to νAs–O (for some relevant IR data see Section 4).

The ^{1}H- and $^{13}C\{^1H\}$-NMR spectra (see Section 4) of these complexes show characteristic peaks and peak multiplicities for R-As and ligand protons as well as for the carbon atoms. The alkyl proton and carbon resonances for Pr^i_3As and Bu^i_3As are shielded on substituting halide with oximate in R_3AsX_2. The shielding of these resonances increases in the order: $R_3AsCl_2 < R_3As(Cl)L < R_3AsL_2$. However, the Pr^i_3As proton and carbon resonances are deshielded on substituting the OH group with oximate in $Pr^i_3As(OH)_2$. The deshielding order is: $Pr^i_3As(OH)_2 < Pr^i_3As(OH)L < Pr^i_3AsL_2$. For a given series these resonances are almost unaffected by the nature of the oxime. The ligand proton and carbon resonances show no appreciable shift, except for the methyl signal which, in general, is shielded relative to that of the corresponding free oxime. The NMR data are comparable with those for the analogous organoantimony complexes for which a trigonal bipyramidal configuration has been unambiguously established by X-ray structural analyses [8,10].

2.1. X-ray crystal structure of $[Pr^i_3AsOH]^+Cl^-$

It has been known for a long time that mild hydrolysis of Ph_3AsCl_2 or Ph_3AsBr_2 leads to the formation of $[Ph_3AsOH]^+X^-$, where X = Cl or Br [18]. The structures of these compounds were first reported [19] in 1968 and subsequently several structural reports have appeared of the cation $[Ph_3AsOH]^+$ [20–24], as well as of

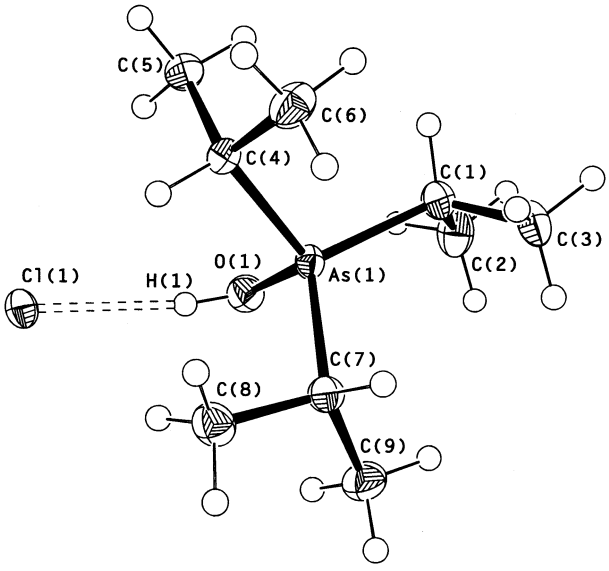
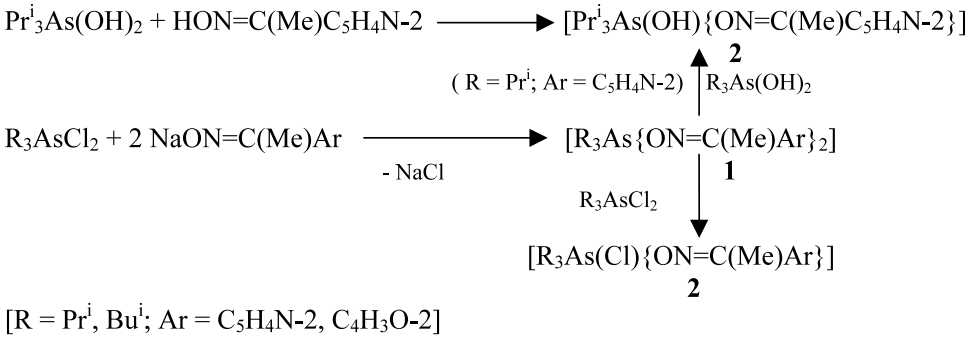


Fig. 1. ORTEP plot of $[(Pr^i)_3AsOH]^+Cl^-$. The atoms are drawn with 50% probability ellipsoids.

$[(Ph_3AsO)_2H]^+$ [25–27], sometimes as a result of their formation as unexpected products.

The ORTEP diagram of $[Pr^i_3AsOH]^+Cl^-$ (Fig. 1) clearly shows the hydrogen bonding between OH and the chloride ion. It also demonstrates, along with the bond lengths and angles in Table 1, that the distorted tetrahedral environment around arsenic that is found for $[Ph_3AsOH]^+$, regardless of the counter ion [19–24], is also found in $[Pr^i_3AsOH]^+Cl^-$. Thus, the O–As–C angles range from 104.72(9) to 108.82(9)° and the C–As–C from 109.3(1) to 115.2(1)°, compared with 102.6(4) to 110.7(4) and 110.0(4) to 112.7(4)°, respectively, for $[Ph_3AsOH]^+Cl^-$ [23]. The average As–C bond length of 1.937(2) Å in $[Pr^i_3AsOH]^+Cl^-$ is longer than those observed, for example, in $[Ph_3AsOH]^+Cl^-$ (1.893(16) ave.) [23] and $[Ph_3AsOH]_2^+Cl^-\cdot ICl_2^-$ (1.901(10) ave. Å) [21] which are typical for phenylarsonium bonds. However, the As–O bond length of 1.726(2) Å in $[Pr^i_3AsOH]^+Cl^-$ is not significantly different from those reported for example for $[Ph_3AsOH]^+Cl^-$ (1.70(1) ave. [19] and 1.723(4) ave.)



Scheme 1.

Table 1
Bond lengths (Å) and angles (°) for $[Pr^i_3AsOH]^+Cl^-$

(*a*) *Bond lengths*			
As(1)–O(1)	1.726(2)	O(1)–H(1)	0.82
H(1)···Cl(1)	2.127	O(1)···Cl(1)	2.941(2)
As(1)–C(1)	1.939(2)	As(1)–C(4)	1.936(2)
As(1)–C(7)	1.935(2)	C(1)–C(3)	1.523(3)
C(1)–C(2)	1.520(3)	C(4)–C(6)	1.530(3)
C(4)–C(5)	1.518(3)	C(7)–C(9)	1.526(3)
C(7)–C(8)	1.527(3)		
(*b*) *Bond angles*			
O(1)–As(1)–C(1)	104.72(9)	O(1)–As(1)–C(4)	108.82(9)
O(1)–As(1)–C(7)	107.95(9)	C(1)–As(1)–C(4)	110.65(10)
C(1)–As(1)–C(7)	115.17(10)	C(4)–As(1)–C(7)	109.25(10)
C(2)–C(1)–C(3)	112.8(2)	C(5)–C(4)–C(6)	113.0(2)
C(2)–C(1)–As(1)	111.92(17)	C(5)–C(4)–As(1)	111.58(16)
C(3)–C(1)–As(1)	110.62(15)	C(6)–C(4)–As(1)	108.72(16)
C(9)–C(7)–C(8)	112.4(2)	C(8)-C(7)-As(l)	108.78(16)
C(9)–C(7)–As(1)	109.30(16)	O(1)–H(1)···Cl(1)	171.7

[23], $[Ph_3AsOH]^+Br^-$ (1.712(12)) [19], $[Ph_3AsOH]^+_2$-$Cl^-\cdot ICl^-_2$ (1.722(8) ave.) [21], $[Ph_3AsOH]^+[SO_3$-(OH)] (1.727(5)) [24], and $[Ph_3AsOH]^+_2[(Nb_6Cl_{12})$-$Cl_6]^-_2$ (1.73(2) Å) [22]. It is, however, significantly longer than a typical As=O bond length of 1.644 Å [28].

The As–O···Cl angle in $[Pr^i_3AsOH]^+Cl^-$ has been found to be 108.85(9)° which is very close to the all-tetrahedral angle. It also gets support from a near linear O–H···Cl angle (171.7°). The O···Cl distance in $[Pr^i_3AsOH]^+Cl^-$ of 2.941(2) Å is longer than in $[Ph_3AsOH]^+Cl^-$ (2.850(5) ave. Å) [23] but similar to the distances found in $[Ph_3AsOH]^+_2Cl^-\cdot ICl^-_2$ (2.946(8) and 2.953(8) Å) where both OH groups are hydrogen-bonded to the chloride ion [21]. Given that an acceptable mean value for the O···Cl distance in an O–H···Cl– hydrogen bond is 3.034(12) Å [29], the hydrogen bonding in $[Pr^i_3AsOH]^+Cl^-$ can be considered to be normal and that in $[Ph_3AsOH]^+Cl^-$ unusually strong, as was suggested earlier [19].

3. Experimental

2-Acetylpyridine and 2-acetylfuran were obtained from Sisco-Chem. Oximes [30], $Pr^i_3AsCl_2$ and $Bu^i_3AsCl_2$ [31] were prepared according to literature methods. All the reactions were carried out in anhydrous solvents unless stated otherwise. IR spectra were recorded as Nujol mulls between CsI plates on a Bomen MB-102 FTIR spectrometer. ^1H- and $^{13}C\{^1H\}$-NMR spectra were recorded on a Bruker DPX-300 spectrometer operating at 300 and 75.47 MHz, respectively, in 5 mm NMR tubes as freshly prepared $CDCl_3$ solutions. Spectra were referenced to internal chloroform ((7.26 for ^1H and 77.0 for ^{13}C).

3.1. Preparation of $[Pr^i As(OH)_2]$

To a stirred benzene solution (25 ml) of $Pr^i_3AsCl_2$ (2.411 g, 8.76 mmol) was added a methanol solution of sodium methoxide [prepared from sodium metal (404 mg, 17.57 mmol) in methanol]. The mixture was stirred with refluxing for 4 h. After cooling to room temperature, 0.3 ml of distilled water was added and stirring continued with heating for 30 min. The solvents were stripped off under vacuum and the residue was extracted with dichloromethane (10 × 2 ml) and filtered. The filtrate was concentrated in vacuo to yield an orange liquid (yield 1.423 g, 68%).

3.2. Preparation of $[Bu^i_3As\{ON{=}C(Me)C_5H_4N\text{-}2\}_2]$

To a benzene solution of $Bu^i_3AsCl_2$ (853 mg, 2.69 mmol), was added a methanol solution of the sodium salt of 2-acetylpyridine ketooxime [prepared from sodium metal (124 mg, 5.39 mmol) dissolved in methanol (10 ml) and 2-acetylpyridine ketooxime (737 mg, 5.41 mmol)] with constant stirring. The reaction mixture was refluxed for 4 h. The solvents were evaporated in vacuo and the compound was extracted with benzene (2 × 15 ml) and filtered through a G-3 sintered funnel. The solvent was removed to give an orange liquid (1.383 g, 99%). Similarly all other $[R_3As\{ON{=}C(Me)Ar\}_2]$ complexes were prepared and the data are summarized in Table 2.

3.3. Preparation of $[Pr^i_3As(Cl)\{ON{=}C(Me)C_4H_3O\text{-}2\}]$

To a stirred benzene solution (15 ml) of $[Pr^i_3As\{ON{=}C(Me)C_4H_3O\text{-}2\}_2]$ (485 mg, 1.07 mmol), was added a solution of $Pr^i_3AsCl_2$ in benzene (295 mg, 1.07 mmol) and the mixture was stirred well for 30 min. The solvent was evaporated in vacuo to give a quantitative yield of $[Pr^i_3As(Cl)\{ON{=}C(Me)C_4H_3O\text{-}2\}]$ (780 mg, 99%). Similarly all other mono(halo)complexes were prepared.

Controlled hydrolysis of a representative mono-chloro-complex $[Pr^i_3As(Cl)\{ON{=}C(Me)C_4H_3O\text{-}2\}]$ was carried out to yield crystals of $Pr^i_3As(OH)Cl$.

$$Pr^i_3As(Cl)\{ON{=}C(Me)C_4H_3O\text{-}2\} + H_2O \rightarrow Pr^i_3As(OH)Cl + HON{=}C(Me)C_4H_3O\text{-}2$$

3.4. Preparation of $[Pr^i_3As(OH)\{ON{=}C(Me)C_5H_4N\text{-}2\}]$

a) To a benzene solution (10 ml) of $Pr^i_3As(OH)_2$ (710 mg, 2.98 mmol) was added a benzene solution of 2-acetylpyridine ketooxime (406 mg, 2.98 mmol) with constant stirring and the mixture was refluxed for 4

Table 2
Synthetic and analytical data of organoarsenic(V) complexes with internally functionalized oximes

Compounds	Percentage yield	Analysis Found (Calc.) (%)				
		As	Cl/Br	C	H	N
$[Pr^i_3As(OH)_2]$	68	31.3(31.5)		45.2(45.4)	9.5(9.7)	
$[Pr^i_3As\{ON=C(Me)C_5H_4N\}_2]$	98	15.4(15.8)		58.0(58.2)	7.2(7.4)	11.5(11.8)
$[Pr^i_3As(Br)\{ON=C(Me)C_5H_4N\}]$	99	17.6(17.9)	18.6(19.1)	45.7(45.8)	6.6(6.7)	6.4(6.7)
$[Pr^i_3As(OH)\{ON=C(Me)C_5H_4N\}]$	93	20.9(21.0)		53.4(53.8)	8.1(8.4)	7.6(7.8)
$[Pr^i_3As\{ON=C(Me)C_4H_3O\}_2]$	99	16.6(16.5)		55.6(55.7)	7.3(7.4)	6.0(6.2)
$[Pr^i_3As(Cl)\{ON=C(Me)C_4H_3O\}]$	99	20.3(20.6)	9.2(9.7)	49.2(49.5)	7.2(7.5)	3.4(3.8)
$[Bu^i_3As\{ON=C(Me)C_5H_4N\}_2]$	99	13.9(14.5)		60.4(60.5)	7.7(8.0)	10.6(10.8)
$[Bu^i_3As(Cl)\{ON=C(Me)C_5H_4N\}]$	99	17.4(18.0)	8.3(8.5)	54.3(54.7)	8.0(8.2)	6.5(6.7)
$[Bu^i_3As\{ON=C(Me)C_4H_3O\}_2]$	98	15.5(15.1)		58.0(58.3)	7.7(7.9)	5.4(5.7)
$[Bu^i_3As(Cl)\{ON=C(Me)C_4H_3O\}]$	99	18.2(18.5)	8.5(8.7)	53.1(53.3)	8.1(8.2)	3.1(3.4)

h and the water liberated was removed azeotropically. Then solvent was removed under vacuum to give a brown liquid (yield 994 mg, 93%).

b) A $CDCl_3$ solution (0.5 ml) of $[Pr^i_3As\{ON=C(Me)C_5H_4N\text{-}2\}_2]$ (21 mg, 0.04 mmol), was added to liquid $[Pr^i_3As(OH)_2]$ (11 mg, 0.04 mmol) in a 5 mm NMR tube. The progress of the reaction was monitored by ^{1}H-NMR spectroscopy.

3.5. X-ray structure determination

A colorless, block crystal of $[Pr^i_3AsOH]^+Cl^-$ was mounted on a glass fiber. An Enraf Nonius Kappa CCD area detector diffractometer, with ϕ and ω scans chosen to give a complete asymmetric unit, was used for data collection. Cell refinement [32] gave cell constants corresponding to an orthorhombic cell whose dimensions are given in Table 3 along with other experimental parameters. An absorption correction was applied [33]. The structure was solved by direct methods [34] and the structure was refined using the WINGX version [35] of SHELX-97 [36]. All of the non-hydrogen atoms were treated anisotropically. Hydrogen atoms were included in idealized positions with isotropic thermal parameters set at 1.2 times that of the carbon atom to which they were attached. The final cycle of full-matrix least-squares refinement was based on 2796 observed reflections (2127 for $F^2 > 4\sigma(F^2)$) and 112 variable parameters and converged (largest parameter shift was 0.001 times its estimated S.D.).

Table 3
Crystal data and structure refinement for $[Pr^i_3AsOH]^+Cl^-$ (**1**)

Empirical formula	$C_9H_{22}OClAs$
Formula weight	256.64
Temperature (K)	120(2)
Wavelength (Å)	0.71073
Crystal system	Orthorhombic
Space group	*Pbca*
a (Å)	12.8649(3)
b (Å)	13.6944(3)
c (Å)	13.9276(3)
Volume (Å^3)	2453.72(9)
Z	8
Density (calculated) (g cm^{-3})	1.389
Absorption coefficient (mm^{-1})	2.950
$F(000)$	1072
Crystal size (mm^3)	$0.25 \times 0.25 \times 0.13$
θ Range for data collection (°)	2.93–27.48
Index ranges	$-16 \le h \le 6$, $-16 \le k \le 17$, $-18 \le l \le 18$
Reflections collected	5164
Independent Reflections	2796 [$R_{int} = 0.0203$]
Max and min transmission	0.7093 and 0.5259
Refinement method	Full-matrix least-squares on F^2
Data/restraints/parameters	2796/0/112
Goodness-of-fit on F^2	1.076
Final R indices [$F^2 > 4\sigma F^2$)]	$R_1 = 0.0308$, $wR_2 = 0.0770$
R indices (all data)	$R_1 = 0.0403$, $wR_2 = 0.0812$
Extinction coefficient	0.0020(3)
Largest difference peak and hole (e Å^{-3})	0.864 and −0.672

4. Supplementary materials

Crystallographic data for the structural analysis as well as IR and NMR tables have been deposited with the Cambridge crystallographic Data Center CCDC no. 192164 for $[Pr^i_3AsOH]^+Cl^-$. Copies of this information may be obtained free of charge from, The Director, CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (Fax: +44-1223-336033; e-mail: deposit@ccdc.cam.ac.uk or www: http://www.ccdc.cam.ac.uk).

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