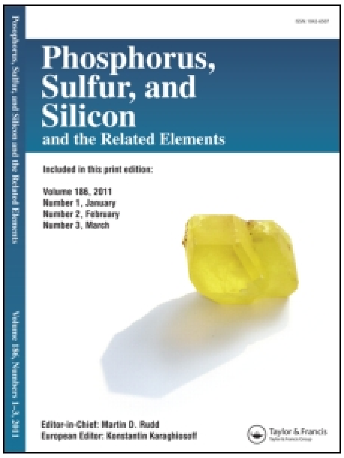

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Synthesis and Characterization of Organoarsenic(III) Derivatives with 2-Aminocyclopentene 1-Carbodithioc Acid and with Their Nitrogen/Sulfur Alkyl Derivatives

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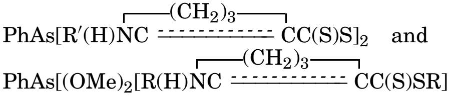
Phenylarsenic(III) Dimethoxide reacts with N-/S- alkyl of 2-aminocyclopentene 1-carbodithioic acid and its nitrogen-/sulfur-alkyl derivatives in 1:2 and 1:1 molar ratio yields some new complexes and addition complexes of the type:



$$\mathrm{PhAs[R'(H)N}\overset{\ulcorner\!-(\mathrm{CH_2})_3-\!\urcorner}{\mathrm{C\,{=}{=}{=}{=}\,C}}\mathrm{C(S)S]_2} \quad \text{and}$$

$$\mathrm{PhAs[(OMe)_2[R(H)N}\overset{\ulcorner\!-(\mathrm{CH_2})_3-\!\urcorner}{\mathrm{C\,{=}{=}{=}{=}\,C}}\mathrm{C(S)SR]}$$

where $R' = -H, -CH_3, -C_2H_5, -C_4H_9$ *and* $R = -CH_3$ *and* $CH_2N(C_2H_5)$. *Reactants were mixed in 1:1 and 1:2 molar ratios and the resulting mixture was refluxed for 5 to 6 h to ensure completion of the reaction. Sodium chloride precipitated was filtered off. Yellow to dark brown colored solids were isolated; these complexes were found to be soluble in common organic solvents (e.g.,* $CHCl_3$*, etc.) These complexes and additional complexes have been characterized by physicochemical and spectroscopic studies.*

Keywords 2-Aminocyclopentene1-carbodithioic acid; phenylarsenic(III) dimethoxide

INTRODUCTION

A number of metal complexes of 2-aminocyclopentene 1-carbodithioc acid and its nitrogen/sulfur alkyl derivatives have been reported in the literature during the last few decades and interesting chemical bonding patterns have been observed with various metals.[1–3] These ligands bind with the metals either in monodentate,[1] or in bidentate fashion;[2] sometimes they show ambidentate behavior, as well. In some cases, these bind with the metal through sulfur-sulfur coordination and sulfur-nitrogen coordination.[4] A survey of the literature reveals

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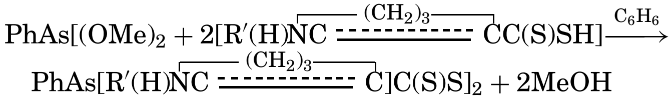
that only stray references[5–8] are available in the literature on the synthesis and characterization of organoarsenic (III) derivatives. In view of the above, we were interested in synthesizing and characterizing some organo-arsenic(III) derivatives with the abovementioned ligands. Recently, arsenic-sulfur (trialkyltri) thioarsenites[5,6] and complexes of N-aryl2-mercaptoacetamides with cyclopentadienyl titanium[7] (IV) have also been reported, which reflects interest in arsenic-sulfur ligand chemistry of metals.

RESULTS AND DISCUSSION

Phenylarsenic(III) derivatives of 2-aminocyclopentene 1-carbodithioic acid and its N-alkyl derivatives have been synthesized by reacting $PhAs(OMe)_2$ (prepared in situ of reacting $PhAsCl_2$with NaOMe) with 2-Aminocyclopentene1- carbodithioc acid, and its N-/S-alkyl derivatives in anhydrous benzene solution under refluxing for 6 h with vigorous stirring (Eq. 1):

$$2NaOMe + PhAsCl_2 \rightarrow PhAs\left(OMe\right)_2 + 2NaCl\downarrow$$

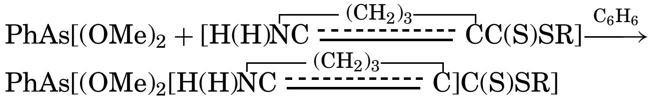


$$PhAs[(OMe)_2 + 2[R'(H)\overbrace{NC \;{=}\; C}^{(CH_2)_3}C(S)SH] \xrightarrow{C_6H_6} PhAs[R'(H)\overbrace{NC \;{=}\; C}^{(CH_2)_3}]C(S)S]_2 + 2MeOH \qquad (1)$$

where $R' = –H,–CH_3,–C_2H_5$ and C_4H_9.

Where as the addition complexes of phenylarsenic (III) dimethoxide with S-alkyl esters of 2-Aminocyclopentene – 1-carbodithioc acid have been synthesized by the reaction of $PhAs(OMe)_2$ with the corresponding ligands in 1:1 molar ratio in refluxing anhydrous benzene solution for ~8 h (Eq. 2):

$$PhAsCl_2 + 2NaOMe \rightarrow PhAs\left(OMe\right)_2 + 2NaCl\downarrow$$



$$PhAs[(OMe)_2 + [H(H)\overbrace{NC \;{=}\; C}^{(CH_2)_3}C(S)SR] \xrightarrow{C_6H_6} PhAs[(OMe)_2[H(H)\overbrace{NC \;{=}\; C}^{(CH_2)_3}]C(S)SR] \qquad (2)$$

where $R = –CH_3, –CH_2NC_2H_5$.

The substituted products and the addition complexes of phenylarsenic(III) of 2-aminocyclopentene-1-carbodithioicacid and its nitrogen-alkyl derivatives and phenyl-arsenic(III) dimethoxide with S-alkyl esters of 2-aminocyclopentene1-carbodithioicacid have been synthesized and isolated after filtering off sodium chloride thus formed during the course of the reaction (Table 1). After removing the benzene a yellow to dark red solids are obtained, which have been purified by dissolving in chloroform and petroleum ether.

It is interesting to mention here that it appears that in these reactions the sulfur being less electronegative (2.44) element in comparison to N(3.0), on one hand and on the other hand in N-alkyl derivatives the donor (+I) capacity of nitrogen is substantially enhanced by the presence of alkyl group on nitrogen atom. This decreases the ionocity of N-H bond thus increase the chances of coordination of nitrogen to metal atom. It also appears that thiols are more acidic than alcohols. The free S-H group seem to be acidic in nature also due to conjugation between carbon atom of the ring and carbon-sulfur double bond (—C—C=C=S) that facilate substitution reaction between phenyl arsenic dimethoxide. While in S-alkyl derivatives there are no chances of substitution due to absence of S—H protons, the formation of 1:2 addition complexes is completely ruled out because the presence of loan pair of electron on arsenic atom repeals back or do not accept another ligand molecule.

IR Spectra

The IR spectra of these complexes have been recorded in the range of 4000–400 and 4000–200 cm^{-1}.

A broad band appearing in the range 3350–3360 cm^{-1} and 1610–1605 cm^{-1} in the spectra of the compounds and the addition compounds may be assigned to NH stretching and NH deformation mode.[9] No significant shift has been observed in NH/NH_2 mode in complexes when compared to its position in parent ligand. This suggests the non-participation of amino groups of ligands in complexation.

A broad absorption bands observed at 2340–2500 cm^{-1} in the spectra of 2-amino cyclopentene 1-carbodithioicacid, and its nitrogen alkyl derivatives, assigned to SH group,[9] are found to be absent in the corresponding compounds. This indicates the deprotonation of SH group on complexation. This has been further supported by the appearance of new absorption band in the range 325–356 cm^{-1}, which has been assigned to As-S.

A strong absorption band has been observed in the region 920–970 cm^{-1} in the organoarsenic(III) derivatives of 2-aminocyclopentene-1-carbodithioicacid and its N-alkyl derivatives which may be assigned to asym CSS mode.[10,11] Appearance of single band for asymmetric CSS in the region indicates the involvement of both the sulfur atoms in the bonding a band due to sym CSS stretching vibration has been observed in the region at 605–610 $cm^{-1.}$

The appearance of two absorption bands for asym CSS in the range of 940–960 cm^{-1}in the phenylarsenic(III) compounds of S-alkyl derivatives of 2-aminocyclopentene -1-carbodithioicacid in the range of 940–960 cm^{-1}indicates the participation of only one sulfur of C(S)SR group in bonding .this has been further supported by the appearance of a new absorption band at 350 cm^{-1} assigned to As-S bond.

The other absorption bands have been observed at their expected position mentioned in Table II.

PMR Spectra

PMR spectra of the newly synthesized derivatives have been recorded in the $CDC1_3$ solution and the chemical shift values have been tabulated in Table III. The PMR spectra of the ligands 2-aminocyclopentene-1-carbodithioic acid and its N-alkyl derivatives exhibits a sharp singlet in the range of δ.13–4.86 ppm, which may be ascribed to SH proton. The absence of the signal for the SH proton in the spectra corresponding organoarsenic (III) complexes supports the deprotonation of SH group.

Broad signals observed in the range of δ 11.0–11.2 and δ 5.89–6.22 ppm may be assigned to NH/NH_2 protons of amino group in organoarsenic(III) complexes, if 2-aminocyclopentene – 1-carbodithioicacid and its N-/S- alkyl derivatives (in ligands in the range 12.2 and6.22 ppm.) for-NH/NH protons. Appearance of both the signals even in the spectra of complexes indicates that the amino group is not deprotonated on complexation.

A slight shift has been observed in the position of NH/NH_2 mode in complexes in comparison to its position in ligands. This is due to breakage of hydrogen bond on complexation.

A singlet observed in the range of δ.23–3.49 ppm may be ascribed for methoxy protons in phenylarsenic(III) dimethoxide compounds of S-alkyl derivatives of 2-aminocyclopentene 1- carbodithioicacid, which favors the formation of addition compounds in phenylarsenic(III) dimethoxide compounds of S- alkyl derivatives of 2- aminocyclopentene-1-carbodithioicacid.

^{13}C NMR

^{13}C NMR spectra of corresponding ligands and complexes were recorded in $CDCl_3$ solution and the observed chemical shifts values are discussed in the following sections.

The ^{13}C NMR spectrum of phenylarsenic(III) complexes of 2-aminocyclopentene – 1-carbodithioic acid exhibits a signal at 205.10 ppm, which may assigned to C(6)carbon; whereas in the spectrum of the ligand, this signal was observed at 197.3 ppm.

TABLE I **Synthetic and Analytical Data of Phenylarsenic(III) of 2-Aminocyclopentene-1-carbodithioic Acid and Its N-/S-Alkyl Derivatives**

	$PhAs[R(H)N\overset{\ulcorner (CH_2)_3 \urcorner}{C\,\overline{\text{- - - - - -}}\,C}C(S)S]_2$	Reactant q (mM)			Molecular formula	Elemental analysis found (calcd.) %		
S. No.	R′	Ligands	Na	$PhAsCl_2$	(% yield*)	S	N	As
1	H	1.10 (6.95)	0.16 (6.95)	0.77 (3.47)	$C_{18}H_{21}S_4N_2As$ (80)	27.90 (27.37)	5.13 (5.97)	15.90 (15.90)
2	CH_3	0.48 (2.60)	0.06 (2.60)	0.46 (1.30)	$C_{20}H_{25}S_4N_2As$ (85)	25.96 (25.82)	5.34 (5.64)	15.04 (15.08)
3	C_2H_5	0.74 (3.91)	0.09 (3.91)	0.44 (1.95)	$C_{22}H_{29}S_4N_2As$ (90)	24.68 (24.44)	5.23 (5.23)	14.27 (14.28)
4	C_4H_9	1.03 (4.78)	0.11 (4.78)	0.53 (2.39)	$C_{26}H_{37}S_4N_2As$ (85)	22.03 (0.28)	4.80 (4.84)	12.80 (12.90)
5	α CH_3	1.12 (6.46)	0.14 (6.08)	0.72 (3.23)	$C_{15}H_{22}S_2O_2N_2As$ (85)	17.11 (17.51)	3.60 (3.75)	19.30 (19.44)
6	$\alpha\beta\gamma$ $-CH_3N(CH_2CH_3)_2$	0.65 (1.30)	0.06 (2.60)	0.31 (1.30)	$C_{18}H_{28}S_2O_2N_2As$ (85)	15.11 (15.42)	6.52 (6.74)	17.91 (18.07)

*After recrystallization.

TABLE II IR Spectral Data of Phenylarsenic(III) of 2-Aminocyclopentene-1-carbodithioic Acid and Its N-/S-Alkyl Derivatives (Cm^{-1})

S. No.	Ligand(a) and corresponding complexes(b) R′\R	νNH	νSH	νNH_2+C=C	νCH_2+C=C	νC=N+C=S	νC=S+C=N	ν asym CSS structure	ν sym CSS structure	As-S	As-C
1	H (a)	3350	2350	1615	1475	1319	1289	878	614	—	—
	H (b)	3340	—	1605	1490	1360	1270	920	610	325	440
2	CH_3 (a)	3340	2340	1615	1495	1346	1284	890	610	—	—
	CH_3 (b)	3350	—	1605	1480	1360	1270	930	605	320	445
3	C_2H_5 (a)	3355	2550	1615	1460	1315	1290	870	615	—	—
	C_2H_5 (b)	3350	—	1610	1490	1365	1280	950	610	335	454
4	C_4H_9 (a)	3350	2500	1615	1470	1320	1290	890	605	—	—
	C_4H_9 (b)	3350	—	1610	1490	1395	1280	970	610	356	454
5	CH_3 (a)	3360	—	1595	1460	1290	1150	905	605	—	—
	CH_3 (b)	3360	—	1590	1480	1270	1150	940	615	350	450
6	$CH_3N(CH_2CH_3)_2$(a)	3350	—	1595	1490	1280	1150	940	615	—	—
	$CH_2N(CH_2CH_3)_2$(b)	3350	—	1590	1480	1270	1160	960	615	350	454

TABLE III ^{1}**H NMR Spectral Data of Phenylarsenic(III) of 2-Aminocyclopentene-1-carbodithioic Acid and Its N-/S-Alkyl derivatives (cm^{-1}) and Their Ligands (δpm)**

S.N.	Ligand (a) and corresponding complexes (b) $R'\backslash R$	$CH_{2(4)}$	$CH_{2(3,5)}$	NH calcd.	$R'\backslash R$	SH	As-C_6H_5	NH (Free) $(OMe)_2$
1	(a) H	1.67–2.04 (q)	2.58–2.87 (m)	11.21 (bs)	6.22 (bs)	4.86	—	—
	(b) H	1.71 (m)	1.37 (q)	11.10 (bs)	6.20	—	6.37–7.89 (m)	—
2	(a) CH_3	1.66–2.02 (q)	2.53–2.81 (m)	12.2 (bs)	3.09-3.10 (m)	3.08– 3.11 (m)	-	—
	(b) CH_3	1.55–2.10 (q)	2.10–2.80 (m)	11.9 (bs)	1.45 (t.	—	6.14–779 (m)	—
3	(a) $CH_{2(7)}CH_{3(8)}$	1.75–2.09 (q)	2.54–3.10 (m)	12.65 (bs)	3.25–3.56 (m) $CH_{2(7)}$, 1.30 (t) $CH_{3(8)}$	3.25–3.56	—	—
	(b) $CH_{2(7)}CH_{3(8)}$	1.64 (q)	2.34–2.75 (m)	11.20 (bs)	2.82–3.07 (t) $CH_{2(7)}$1.80 (t)CH_3	—	7.29–7.92 (m)	—
4	(a) $CH_{2(7)}CH_{2(8)}$ $CH_{2(9)}CH_{3(10)}$	1.90–2.28 (q)	2.63–3.17 (m)	12.55 (bs)	3.17–3.61 (q) $CH_{2(7)}$ 1.26–1.90 (m) $CH_{2(8,9)}$ 0.95 (t) $CH_{2(10)}$	1.99–2.13 (m)	—	—
	(b) $CH_{2(7)}CH_{2(8)}$ $CH_{2(9)}CH_{3(10)}$	1.22–1.70 (q)	2.50–2.90 (m)	11.70 (bs)	3.1–3.4 (4) $CH_{2(7)}$ 1.22-1.77 (m) , CH_2 0.29–0.90 (t) $CH_{3(9)}$	—	6.98–7.79 (m)	—
5	(a) α CH_3	1.23–1.60 (m)	2.60–2.94 (m)	11.20 (bs)	2.60 (s)	—	—	6.20
	(b) α CH_3	1.76–2.01 (q)	2.43–2.96 (m)	11.09 (bs)	2.40–2.96 (m)	—	6.72–7.76 (m)	5.89 [3.49]
6	(a) CH_2N $(CH_2CH_3)_2$	1.10–2.09 (m)	2.30–2.70 (m)	11.45 (bs)	5.21 (s) $CH_2(\alpha)$, 2.73 (m) $CH_2(\beta)$, 1.50 (t)$CH_2(\gamma)$	—	—	6.18 [-]
	(b) $\alpha\beta\gamma$ CH_3N $(CH_2CH_3)_2$	1.76–2.01 (q)	2.47–2.87 (m)	11.11 (bs)	4.32 (s) CH_2N (α), 2.47–2.84 (m) $CH_2(\beta)$, 1.28 (t)$CH_2(\gamma)$	—	7.37–7.89	6.22 [3.23 (s)]

A downward shift of 8 ppm in its position may be due to deshielding of C6 carbon atom because of metallation of SH group and coordination between arsenic and sulfur atom of C(S)S group. The signal for C(2) carbon in same complex observed at 187.1 ppm and at170.1 ppm for ligand. This signal show a downfield shift ∼17 ppm in its position as compared to the ligand which may be due to decrease in electron density between C(2) and C(6) as both the sulfur atom attached to C(6) carbon coordinate to the metal atom there by decreasing the electron density.

In the ^{13}C NMR spectra of addition compounds of phenylarsenic(III) dimethoxide compounds of sulfur alkyl derivatives of 2-aminocarbodithioic acid, the signal for C(6) carbon atom observed at δ79.3–179.5 (in ligand at δ 201.1 ppm)ppm respectively. An upfield shift has been observed in the position of C(6) carbon atom in comparison to its position in the spectra of free ligands.

This favors the coordination between free sulfur atom of dithiocarboxylate group of ligand moiety and arsenic atom. The methoxy carbon signals have been observed in the range of δ 43.4–49.7 ppm in both the compounds; this favors the formation of addition complex of phenylarsenic(III) with sulfur alkyl derivatives of 2- aminocarbodithioic acid and its N- alkyl derivatives from substituted compounds. Other signals appear at their expected position shown in Table IV.

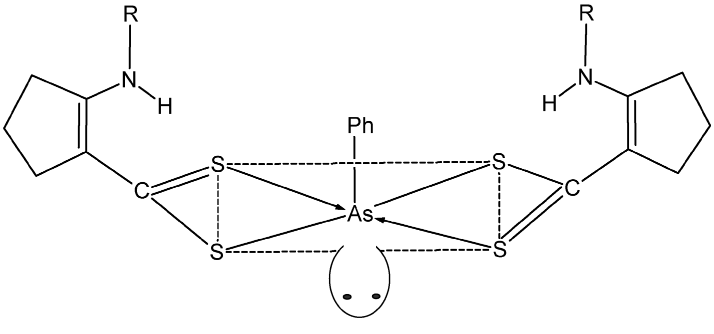
The corrected chemical shift values[12] and Hammet Taft constant $R_0^{13,14}$ have been found to be in the region of δ 7.8–8.8 ppm and δ 0.35–0.37 ppm, respectively. These negative values indicate an electron release from arsenic atom towards phenyl ring through d_π-p_π conjugation and poor donor capability of arsenic atom in these derivatives.

On the basis of strong absorption band for asym C(S)S and absence of SH signal in the IR spectra and the position of the NH_2 in phenyl or R-NH proton in ^{1}H NMR in phenylarsenic(III) complexes of

TABLE IV ^{13}C NMR Spectral Data of 2-Aminocyclopentene-1-carbodithioic Acid and Its N-/S-Alkyl Derivatives and Their Corresponding Phenyl Arsenic (III) Derivatives

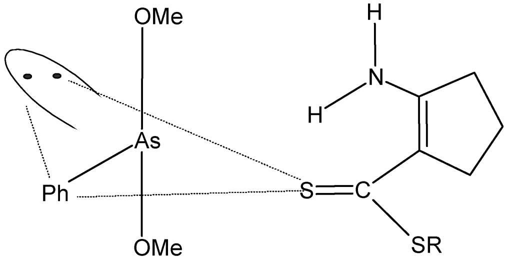
S. N.	Ligand(a) and corresponding complexes(b) R′	$C_{(1)}$	$C_{(2)}$	$C_{(3)}$	$C_{(4)}$	$C_{(5)}$	$C_{(6)}$	R′
1	(a) H	118.8	170.1	36.7	20.0	34.3	197.3	—
	(b) H	118.7	187.1	36.8	19.3	34.1	205.10	—
2	(a) CH_3	118.9	176.5	34.2	20.9	33.2	159.0	—
	(b) CH_3	—	—	—	—	—	—	31.4

2-aminocyclopentene 1- carbodithioic acid and its nitrogen alkyl derivatives , it may be concluded that the NHR group of ligand does not take part in the bonding and the chemical bonding takes place between arsenic and CS_2 group of the ligand. The ligand behaves as bidentate moiety. In view of bidentate nature of ligand and presence of lone pair of electron following structure may be tentatively proposed for these derivatives



Structure of PhAs[R'(H)N$\overbrace{\mathrm{C{=}C}}^{(CH_2)_3}$C(S)S]$_2$

Whereas the appearance of two strong bands at 940–970 cm^{-1} for νasym C(S)S in the IR spectra of phenylarsenic dimethoxide derivatives of S-alkyl derivatives of 2-aminocyclopentene – 1- carbodithioicacid indicates the monodentate behavior of ligand moiety in addition compound and non involvement of amino group in bonding and favors the coordination between the sulfur atom of dithiocarboxylate group of ligand moiety and arsenic atom and on the basis of IR ,NMR (^{1}H & ^{13}C) spectral evidences following probable structure may be assigned.



Structure of PhAs(OMe)$_2$[R(H)N$\overbrace{\mathrm{C{=}C}}^{(CH_2)_3}$C(S)SR]

EXPERIMENTAL

All reaction was carried in anhydrous conditions. The chemicals used were reagent grade. Solvents used were dried by standard methods. The ligands 2-aminocyclopentene-1-carbodithioicacid and its N-/S-alkylderivatives and phenylarsenic (III) derivative have been prepared by standard methods reported in literature.[14,15] These products have been purified by dissolving in choloroform and then precipitating by adding access of petroleum ether. Separated product were decanted off and washed repeatedly with petroleum ether and the last traces of volatile fraction were removed under reduced pressure. These complexes are found to be soluble in common organic solvents, eg-$CHCl_3CCl_4$ and CH_2Cl_2elements arsenic, sulfur and nitrogen were analyzed through literature methods.[16,17] IR absorption spectra were recorded as a nujol mull on CSI pallets in the region 4000–400cm^{-1}, and 4000–200cm^{-1}. The ^{1}H and ^{13}C NMR spectra were recorded in $CDCl_3$ and DMSO solution, respectively, on BRUCKER-270 MHz.Brucker DPX-300 MHz and JEOL FX 90Q MHz using TMS as in internal reference. All the derivatives have been prepared by the same procedure. The preparation method of a representative complexes and addition complexes is described below and the results for the rest are summarized in Table I.

Synthesis of Organic Derivatives of Organoarsenic(III) Aminocyclopentene(I)-Carbodithioicacid and Its N–Alkylderivatives

These derivatives have been prepared by the reaction of $PhAsCl_2$, with corresponding ligands in 1:2 molar ratio in presence of sodium methoxide in benzene solution under constant stirring and heating for 8 h.

Since all the N-alkylderivatives are prepared by similar methods for the sake of brevity and convenience the detailed synthesis of only one representative compounds is being discussed and the rest are summarized in Table IV.

Synthesis of Phenyl Arsenic (III)-Derivatives of Bis[2-Aminocyclopentene-1-carbodithiato-S,S]

To a benzene solution (15 ml) of sodium methoxide [prepared by treating sodium (0.16 gm, 6.95 mM) with small amount (15 ml) of methanol], a benzene solution of phenyl arsenic(III) dichloride (0.81gm, 3.63 mM) was added, and the solution was stirred for 2 h. Then 15 ml of benzene solution of ligand 2-aminocyclopentene – 1- carbodithioicacid (1.16 g, 7.28 mM) was added to it. The reaction mixture was stirred for 4 h to ensure the completion of reaction. After the completion of reaction, NaCl formed was filtered off, and the excess of the solvent was removed from the filtrate under reduced pressure. The yellow colored solid compound thus obtained was recrystallized from chloroform/n-hexane mixture (91% yield). The compound was analyzed for the As, N and S found As = 15.40, N = 5.13 and S = 27.90 %, calculated for $C_{18}H_{21}S_4N_2As$, (As = 15.90, N = 5.97 and S = 27.37 %).

Synthesis of Addition Compounds of Phenylarsenic(III) Methoxide with S-Alkyl Esters of 2-Aminocyclopentene-1-carbodithioicacid

These compounds have been synthesized by the reaction of $PhAsCl_2$ with corresponding ligands in 1:1 molar ratio in presence of a base NaOMe. Since both the compounds have been prepared by a similar method, the synthesis of only one representative compounds is being summarized in Table I.

Synthesis of [S-Methyl-2-aminocyclopentene – 1-carbodithioato-S] Phenyl Arsenic (III) Dimethoxide

Add (0.72 g, 3.23 mM) of sodium in small amount of methanol (20 ml). Then add 20 ml of benzene solution of the ligand S-methyl – 2-aminocyclopentene – 1-carbodithioicacid (1.12 g, 6.46 Mm) was added to it and the reaction mixture was refluxed for 8 h to ensure the completion of reaction. The precipitated sodium chloride was filtered off. The excess of solvent was removed from the filtrate under reduced pressure. The brown solid compound was recrystallized from chloroform/pet. Ether (40–60 OC) mixture (yield 93%) analysis found, As = 19.30, S = 17.11, and N = 3.60%; calculated for $C_{15}H_{22}S_2O_2NAs$ (As = 19.44, S = 17.51 and N = 3.75 %).

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