Synthesis and Characterization of Some Mixed (Isopropyl Xanthato) (β-Ketoaminato) Phenylarsenic (III) And (Isopropyl Xanthato) (Thiosemi Carbazonato) Phenylarsenic (III) Derivatives

Rita Gupta[1] and Yashpal Singh[2*]

[1]*Government. P.G. College Dausa Rajasthan, India*
[2]*Department of Chemistry, University of Rajasthan, Jaipur-302055, India*

ABSTRACT

Mixed ligand compounds of phenylarsenic (III) having the general formula Ph As [CH_3CO CH CHC(CH_3) N(R)][S_2 $COPr^i$], where R = CH_3 (1), C_2H_5(2), n-C_3H_7 (3), i-C_3H_7 (4) and n-C_4H_9 (5), Ph As [SC (NH_2) NNC (R) R] [S_2 $COPr^i$] (where R = H; R^1 = C_6H_5 (6), C_6H_4 CHCH(7), $C_6H_4OCH_3$ (8), R = CH_3; R^1 = C_6H_5,(9), $C_6H_4CH_3$,(10), (C6H_4OCH_3) (11), have been synthesised by the reactions of K[S_2 $COPr^i$] with Cl Ph As [CH_3CO CH CHC(CH_3) N(R)] and Cl Ph As [SC (NH_2) NNC (R) R^1] respectively, in 1:1 molar ratio in chloroform solution. All these compounds have been characterised by elemental analysis and molecular weight measurements, and probable structures of all these compounds 1-11 have been proposed on the basis of spectral evidences.

INTRODUCTION

During the last two to three decades, a large number of organic derivatives of arsenic (III), have been reported /1-4/. Mixed ligand derivatives of arsenic (III) and antimony (III) have also been reported /5-7/ due to their intresting structural features. Although mixed chloro-phenylarsenic (III) derivaties of the ligands, like, heterocyclic dithiocarbamates /8/ 2-mercapto acetamides/9/, thiosemicarbazones /10/ and β-ketoamine/11/ have been reported, no mixed ligand compounds of organoarsenic (III) have been reported so far.

In this paper we report the synthesis and characterization of mixed phenyl (isopropyl xanthato) arsenic(III) compounds.

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[*] E-mail – yp_singh07@yahoo.co.in

EXPERIMENTAL

All the reactions were carried out under anhydrous conditions. The chemicals used were of reagent grade. Phenyl arsenic (III) dichloride/12/ , thiosemicarbazones/13/ , β-ketoamines /14/, and isopropyl xanthates /15/ were prepared by reported methods. Arsenic, sulphur and nitrogen were estimated by iodometric, Messenger and Kjeldal method respectively/16/. molecular weights were determined cryoscopically in dry chloroform solution. IR spectra have been recorded as nujol mulls using CsI cells on FT IR spectrophotometer model 8400 S Shimadzu in the range 4000-200 cm, ^{1}H, and ^{13} C NMR spectra of the samples have been recorded in $CDCl_3$, on JEOL FT AL 300 MHz spectrometer.

Mixed chloro(β-ketoaminato) (isopropylxanthato) phenylarsenic(III)/12/ and chloro (thiosemicarbazonato) (isopropylxanthato) phenylarsenic(III)/11/ have been prepared by literature methods.All the compounds of each series of mixed ligand compounds, 1-5 and 6-11, have been prepared using the similar method. Therefore the synthetic procedure of one representative compound of each series is being discussed in detail and the results of the rest of the compounds are summarized in **Table 1** and **2** respectively:

(i) Synthesis of PhAs $[OC(CH_3)\ CH\ C\ (CH_3)\ N\ CH_3]\ [S_2COC_3H_2]$

To a suspension of potassium isopropyl xanthate (0.43 g, 2.40 millimole) in dry chloroform solution, a solution of calculated amount of Ph As Cl $[OC(CH_3)\ CH\ C(CH_3)\ N\ CH_3]$ [0.71 g, 2.37 mmole] was added, the reaction contents were continuously stirred for ~ 3-4 hours and the resultant potassium chloride was filtered off. After removing the excess solvent under reduced pressure, a dark solid compound was obtained, in quantitative yield, which was purified from chloroform n-hexane mixture. The synthetic and analytical data of all these compounds are given in **Table 1**.

(ii) Synthesis of Ph As $[SC\ (NH_2)\ NNC\ (H)\ C_6H_5]\ [S_2COC_3H_2)$

A chloroform solution of chlorophenylarsenic(III) (1–benzal–3– thiosemicarbazide) (0.76 g, 2.07 m mole) was added to a solution of potassium isopropyl xanthate (0.36 g, 2.01m mole). The reaction mixture was stirred for ~3 hours at room temperature and KCl thus formed was filtered off. The excess of the solvent was removed under reduced pressure to yield a viscous liquid which was purified from chloroform and n-hexane mixture. The synthetic and analytical data of all the compounds are given in **Table 2**.

RESULTS AND DISCUSSION

Reactions of PhClAs $[OC\ (CH_3CH(C\ H_3)\ NR]$ and $K[S_2\ COPr^i]$ in 1:1 molar ratio in chloroform solution yield the compounds of the type Ph As$[OC\ (CH_3)\ CHC\ (CH_3)\ NR]\ [S_2\ COPr^i]$

Ph As $[OC(CH_3CHC(CH_3)NR]Cl+[KS_2\ COPr^i]\rightarrow PhAs[OC\ (CH_3)\ CH\ C(CH_3)\ NR]\ [S_2\ COPr^i] + KCl$

where R = CH_3(1), C_2H_5(2), n-C_3H_7 (3), i- C_3H_7(4), n – C_4H_9(5)

TABLE 1

SYNTHETIC AND ANALYTICAL DATA OF MIXED ISO-PROPYL XANTHATO (THIOSEMICARBAZONATO) PHENYL ARSENIC (III) COMPOUNDS

S. No.	Reactants gm (mM)		Product % yield	% Found (Calcd.)			Mol. Wt. found (CalCd.)
	$PhAsCl[SC(NH_2):NN:C(R)R^1)$	$KS_2COC_3H_7$		AS	N	S	
1	$R=H, R^1 = C_6H_5$ 0.76 (2.07)	0.36 (2 06)	$PhAs[SC(NH_2):NN:C(H)C_6H_5][S_2COC_3H_7]$ 93	15.92 (16.12)	9.00 (9.03)	20 00 (20 64)	400 (465)
2	$R=H, R^1=C_6H_5CHCH$ 0.47 (1.20)	0.21 (1.20)	$PhAs[SC(NH_2):NN:C(H)C_6H_5CHCH][S_2COC_3H_7]$ 92	15.00 (15.27)	8.00 (8.54)	19.42 (19.54)	480 (491)
3	$R=H, R^1=C_5H_4OCH_3$ 0 26 (0.659)	0.11 (0.632)	$PhAs[SC(NH_2):NN:C(H)CH_3OC_6H_4][S_2COC_3H_7]$ 85	14.91 (15.15)	8.00 (8.48)	19.00 (19.39)	450 (495)
4	$R=CH_3, R^1=C_6H_5$ 1.00 (2.63)	0 46 (2 64)	$PhAs[SC(NH_2):NN:C(CH_3)C_6H_5][S_2COC_3H_7]$ 81	15.13 (15.65)	8.32 (8.76)	19 98 (20.04)	480 (479)
5	$R=CH_3, R^1=C_6H_4CH_3$ 0.39 (0.991)	0.17 (0.977)	$PhAs[SC(NH_2):NN:C(CH_3)C_6H_4CH_3][S_2COC_3H_7]$ 93	14 98 (15.21)	8.32 (8.51)	19.00 (19.47)	470 (493)
6	$R=CH_3, R^1=C_6H_4OCH_3$ 0.41 (1.00)	0.18 (1.03)	$PhAs[SC(NH_2):NN:C(CH_3)CH_3OC_6H_4][S_2COC_3H_7]$ 90	14.60 (14.73)	8 01 (8 25)	18.65 (18.86)	500 (509)

TABLE 2

SYNTHETIC AND ANALYTICAL DATA OF MIXED ISO-PROPYL XANTHATO (β-KETOAMINATO) PHENYL ARSENIC (III) COMPOUNDS

S. No.	Reactants gm (mM)		Product %yield	% Found (Calcd.)			Mol. Wt. found (CalCd.)
	PhAsCl [OC(CH$_3$)CH C(CH$_3$)NR]	KS$_2$COC$_3$H$_7$		AS	N	S	
7	R = CH$_3$ 0.74 (2.47)	0.43 (2.47)	PhAs[OC(CH$_3$)CH C(CH$_3$)NCH$_3$] [S$_2$COC$_3$H$_7$] 80	18.64 (18.79)	3.48 (3.50)	16.00 (16.04)	380 (399)
8	R = C$_2$H$_5$ 0.51 (1.62)	0.28 (1.60)	PhAs[OC(CH$_3$)CH C(CH$_3$)NC$_2$H$_5$] [S$_2$COC$_3$H$_7$] 89	18.00 (18.15)	3.29 (3.38)	15.38 (15.49)	400 (413)
9	R = C$_3$H$_7^n$ 0.82 (2.50)	0.44 (2.52)	PhAs[OC(CH$_3$)CH C(CH$_3$)NC$_3$H$_7$^{n}] [S$_2$COC$_3$H$_7$] 90	17.43 (17.56)	3.00 (3.27)	14.88 (14.98)	441 (427)
10	R = C$_3$H$_7^i$ 0.73 (2.22)	0.39 (2.24)	PhAs[OC(CH$_3$)CH C(CH$_3$)NC$_3$H$_7$^{i}] [S$_2$COC$_3$H$_7$] 93	17.44 (17.56)	3.12 (3.27)	14.88 (14.98)	410 (427)
11	R = C$_4$H$_9^n$ 0.66 (1.93)	0.34 (1.95)	PhAs[OC(CH$_3$)CH C(CH$_3$)NC$_4$H$_9$^{n}] [S$_2$COC$_3$H$_7$] 89	16.92 (17.00)	2.99 (3.17)	14.00 (14.51)	420 (441)

All these compounds are coloured solids or viscous liquids, soluble in common organic solvents. Molecular weight measurement data of these compounds indicate their monomeric nature.Similarly the reactions of PhAs [SC (NH_2) NN C(R) (R^1) Cl with $K[S_2\,COPr^i]$ in 1:1 molar ratio chloroform solution yield corresponding compounds 6-11,

Ph As $[SC(NH_2)NNC(R)R^1]Cl + K[S_2\,COPr^i] \rightarrow$ Ph As $[SC(NH_2)NNC(R)R^1]\,[S_2\,COPr^i] + KCl$

where

R = H, $R^1 = C_6H_5$(6), C_6H_5CH (7), $C_6H_4OCH_3$ (8)R = CH_3, $R^1 = C_6H_5$ (9), $C_6H_4CH_3$ (10), $C_6H_4OCH_3$ (11)

All these derivatives are coloured solids or viscous liquids soluble in common organic solvents. Molecular weight determinations in freezing benzene solution shows their monomeric nature.

SPECTRAL STUDIES

IR Spectra

The medium intensity bands observed in the spectra of β-ketoamines at 2870-2890 cm^{-1}, due to υN- H vibrations, were found to be absent from the spectra of corresponding arsenic compounds (1-5). This is due to the deprotonation of NH group and formation of As – N bond, which is supported by the appearance of a new band at 413-405 cm^{-1} due to υ As – N/17/ mode. Lowering in the position of the band for υ C = N and υC = O also shows the involvement of both groups in bonding and formation of As-N as well as As -O bond during the complexation, which is supported by the appearance of a new band at 560 - 576 cm^{-1} due to υ As-O vibrations/18/.

The medium intensity bands observed at 2900-2800 cm^{-1} in the spectra of thiosemicarbazones, due to υN-H vibrations, was found to be absent in the spectra of compounds (6-11). This indicates the deprotonation of NH group on complexation and involvement of nitrogen in bonding. This was further supported by the appearance of a new band in the region 413-405 cm^{-1} which may be assigned to υAs – N/17/ mode. In these derivatives the absence of any shift in the position of υ asym NH_2 and υ sym NH_2 clearly indicates that NH_2 group is not involved in the bonding. Two medium intensity bands observed in the region 1610-1600 and 1090-1080 cm^{-1}, due to υC = N and υC = S stretching modes in ligands, are shifted by ~40 cm^{-1} and appear in the regions 1586-1520 and 1044-1011cm^{-1} respectively in the spectra of compounds 6 –11.

The I.R. spectra of all these compounds, 1-11, show the presence of single band for υ CSS in the region 1030-1010 cm^{-1} which shows that both sulphur atoms are bonded to arsenic atom symmetrically, leading to bidentate behaviour of CS_2 group. A weak band at 350 cm^{-1} has been observed due to υAs-S/18/. A bond due to υAs-C/20/ has been observed at 470 cm^{-1} in the spectra of all these compounds.

^{1}H NMR Spectra

^{1}H NMR Spectra of 1-5 compounds (**Table 3**) shows the absence of a broad signal observed at 10.28-16.68 ppm due to the NH proton (observed in the spectra of β-ketoamines ligands), indicating the deprotonation of NH proton.

TABLE 3

^{1}H NMR DATA OF MIXED ISO-PROPYL XANTHATO (β KETOAMINATO) PHENYL ARSENIC (III) COMPOUNDS

S. No.	Compound	CH	CH_2	CH_3	C_6H_5
1.	a^1 a b^1 c^1 b d^1 PhAs [OC(CH_3) CH C(CH_3) N CH_3] [S_2 COCH $(CH_3)_2$]	a 4.96s b, 5.40-5.72m	--	a^1 1.94s b^1 1.87s c^1 3.12s d^1 1.29d	7.33 – 7.95 m
2.	a^1 a b^1 c^1 b d^1 PhAs [OC(CH_3) CH C(CH_3) N CH_2CH_3] [S_2 COCH $(CH_3)_2$]	a = 5.00s b = 5.45-5.81m	3.30q	a^1 2.91s b^1 1.92s c^1 1.20s d^1 1.38d	7.33 – 8.00 m
3.	a^1 a b^1 a'' b'' c^1 b d^1 PhAs [OC(CH_3) CH C(CH_3) N CH_2 CH_2CH_3] [S_2 COCH $(CH_3)_2$]	a = 5.00s b = 5.45-5.81m	a'' = 3.21t b'' = 1.43– 1.74m	a^1 2.01s b^1 1.92s c^1 0.98t d^1 1.38d	7.37 – 7.95 m
4.	a^1 a b^1 c'' c^1 b d^1 PhAs [OC(CH_3) CH C(CH_3) N CH $(CH_3)_2$] [S_2 COCH $(CH_3)_2$]	a = 4.91s b = 5.40-5.94m c = 3.44-4.06m	-	a^1 1.96s b^1 1.92s c^1 1.20d d^1 1.38d	*7.28 – 7.95 m*
5.	a^1 a b^1 a'' b'' c'' c' b d^1 PhAs [OC(CH_3) CH C(CH_3) NCH_2 CH_2 CH_2 CH_3][$S_2COCH(CH_3)_2$]	a = 4.98s b = 5.45-5.81m	a'' = 3.21t b'' c'' = 1.43–1.69m	a^1 2.01s b^1 1.92s c^1 0.89t d^1 1.34d	*7.33 – 7.95 m*

The methyl (CH_3CO & CH_3CN) and methine (- CH =) protons have been observed in the range δ 0.89 – 3.12 ppm and 4.96 – 5.81 ppm respectively and exhibit a downfield shift of δ 0.23 – 0.54 ppm as compared to their positions in thecorresponding β-ketoamine ligands. This shift shows the delocalisation of electrons in the six-membered quasi-aromatic chelate ring formed during the complexation.The aromatic protons attached to arsenic appear in the range δ7.28 – 8.00 ppm as a multiplet.

The ^{1}H NMR spectra of 6-11 compounds (**Table 4**) exhibit the disappearance of signal observed at δ1.84-1.55 ppm due to NH/SH groups. Absence of any shift in the position of NH_2 signal indicates that this group does not take part in the bonding. Aryl proton of the ligand moiety (thiosemicarbazone) appears as a complex multiplet in the region δ8.18 – 6.88 ppm.

In the ^{1}H spectra of all the compounds, 1-11, the phenyl protons attached to arsenic atom have been observed in the range 6.88 - 8.18 ppm. The isopropyl protons of xanthate moiety appear as doublet in the region δ1.43–1.29 ppm, due to methyl protons, and as septate /multiplet/ in δ5.94 – 5.40 ppm, due to methine proton.

^{13}C NMR Spectra

The ^{13}C NMR spectra of compounds 1-5 (**Table 5**) exhibit one singlet for C=0 and C=N each in the range of 193.8-194.8 and δ161.7 – 165.0 ppm. Signals for >C=O and >C=N, as well as alkyl and methine carbons , show downfield shift in their positions as compared to their positions in the ligand, which is due to the involvement of >C=O and >C=N groups in bonding and formation of a quasiaromatic ring.

In the ^{13}C NMR spectra of compounds 6-11, (**Table** 6) signals for C=S and C=N have been observed in the range 183.5-177.5 and 150.1-144.8 ppm respectively. The signals of methyl carbons are observed in the range of δ55.0 – 13.0 ppm. These signals show a downfield shift in their position as compared to their position in the corresponding ligands. CS_2 carbon of xanthate group has been observed around δ273.4 ppm, whereas CH_3 and methine carbons of isopropyl group have been observed at δ21.2 and 79.4 ppm, confirming the presence of isopropyl xanthate in the complex.

In the spectra of all these compounds four singlets for C_6H_5-As carbon have been observed in the range of 143.0-127.0 ppm. The corrected chemical shift values δ', defined /20/ as δ'= δCp-Cm (where δ'– δCp and Cm are the chemical shift values of para- and meta-carbon atoms of the phenyl group attached to arsenic atom) and Hammet Taft constant σR^0 /21/ calculated by the relation (σR^0 =δ /22.06). have been found to be in the region δ-3.0 -2.4 and (-)0.13 - 0.10 ppm. These negative values indicate an electron release from As atom towards the phenyl ring through d π- pπ conjugation and poor donor capability of arsenic atom in these derivatives.

In view of the monomeric nature of all these compounds (1-11), the mono functional bidentate nature of all three ligands, viz. β-ketoamines, thiosemicarbozones and isopropyl xanthate, following structures, may proposed in which arsenic has pseudooctahedral geometry for these derivatives (**Figure 1a** and **b**):

TABLE 4

^{1}H NMR DATA OF MIXED ISO-PROPYL XANTHATO (THIOSEMICARBAZONATO) PHENYL ARSENIC (III) COMPOUNDS

S. No.	Compound	CH	CH_3	C_6H_5	NH_2
6.	a b $PhAs[SC(NH_2):NN=C(H)C_6H_5][S_2COCH(CH_3)_2]$	a, 8.04 s b, 5.49 – 5.86 m	1.38 d	7.33 – 7.95 m	6.66 b
7	a b c d $PhAs[SC(NH_2) NN=C(H)CHCHC_6H_5] [S_2COCH(CH_3)_2]$	a 7.01 d b mixed in phenyl c, 7.30 d d, 5.54 – 5.85 m	1.43 d	7.33 – 8.09 m	6.79 b
8	a a' b b' $PhAs[SC(NH_2):NN=C(H)C_6H_4OCH_3][S_2COCH(CH_3)_2]$	a, 8.67 s b, 5.49 – 5.90 m	a^1 3.79 s b^1 1.38 d	6.92 – 8.00 m	6.52 b
9	a' b' $PhAs[SC(NH_2):NN=C(CH_3)C_6H_5][S_2COCH(CH_3)_2]$	5.45 – 5.81 m	a', 2.19 s b', 1.38 d	7.33 – 8.00 m	6.92 b
10	a' b' c' $PhAs[SC(NH_2):NN=C(CH_3)C_6H_4CH_3][S_2COCH(CH_3)_2]$	5.45 – 5.85 m	a', 2.59 s b', 2.36 s c', 1.43 d	7.24 – 8.04 m	6.52 b
11	a' b' c' $PhAs[SC(NH_2):NN=C(CH_3)C_6H_4OCH_3][S_2COCH(CH_3)_2]$	5.41 – 5.96 m	a', 3.38 s b', 2.27 s c', 1.43 d	6.88 – 8.18 m	6.66 b

TABLE 5

^{1}H NMR DATA OF MIXED ISO-PROPYL XANTHATO (β KETOAMINATO) PHENYL ARSENIC (III) COMPOUNDS

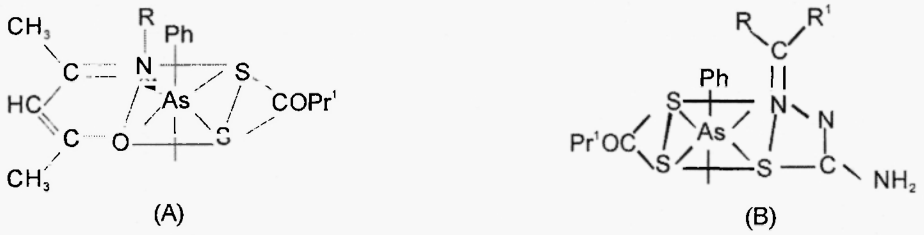
S. No	Compound	CH	CH$_2$	CH$_3$	CS$_2$	CO	CN	C$_6$H$_3$*	□ '	□ R
1.	a^1 a b^1 c^1 b d^1 PhAs [OC(CH$_3$) CH C(CH$_3$) N CH$_3$] [S$_2$ COCH CH$_3$)$_2$]	a 94.8 b.79.2	--	a" 27.5 b" 18.7 c 28.1 d^1 21.5	213.0	194.8	165.0	138.0, 129.9 134.9, 128.2	-5.0	-0.226
2.	a^1 a b^1 c^1 b d^1 PhAs [OC(CH$_3$) CH C(CH$_3$) N CH$_2$CH$_3$] [S$_2$ COCH (CH$_3$)$_2$]	a.95.0 b.79.2	37.3	a^1 27.3 b^1 18.6 c^1 14.1 d^1 21.4	213.0	194.7	164.3	138.1, 129.8 135.1, 128.2	-5.3	-0.24
3.	a^1 a b^1 c" b" c^1 b d^1 PhAs [OC(CH$_3$) CH C(CH$_3$) N CH$_2$ CH CH$_3$] [S$_2$ COCH (CH$_3$)$_2$]	a 95.2 b.79.2	a"44.3 b'22.9	a^1 28.2 b^1 20.9 c^1 11.3 d^1 21.5	212.4	193.8	163.4	138.6, 129.1, 133.6, 128.2	-4.5	-0.203
4.	a^1 a b^1 c" c^1 b d^1 PhAs [OC(CH$_3$) CH C(CH$_3$) N CH (CH$_3$)$_2$] [S$_2$ COCH (CH$_3$)$_2$]	a.95.4 b.79.3 c.44.3	-	a^1 28.3 b^1 18.3 c^1 23.8 d^1 21.8	213.1	193.8	161.7	138.2, 129.0 132.6, 128.2	-3.6	-0.163
5.	a^1 a b^1 a' b" c" c^1 b d^1 PhAs [OC(CH$_3$) CH C(CH$_3$) N CH$_2$ CH$_2$ CH$_2$ CH$_3$] [S$_2$ COCH(CH$_3$)$_2$]	a.94.7 b.79.3	a'42.4 b'18.3 c"19.6	a^1 28.3 b^1 18.3 c^1 13.3 d^1 20.9	213.2	194.0	162.7	138.3, 134.6 130.0, 128.2	-4.6	-0.208

* = Value are given in the order of ipso, meta, para and ortho respectively

TABLE 6

^{13}C N.M.R. DATA OF MIXED ISO-PROPYL XANTHATO (THIOSEMICARBAZONATO) PHENYL ARSENIC (III) COMPOUNDS

S. No.	Compound	CH	CH_3	CS_2	CO	CN	C_6H_5*	□ '	□ R
6.	a b $PhAs[SC(NH_2):NN{=}C(H)C_6H_5][S_2COCH(CH_3)_2]$	79.4	21.2	a' 178.1 b' 213.8		148.3	133.1, 127.6 129.7. 126.5	-2.1	-0.09
7.	a b c d $PhAs[SC(NH_2):NN{=}C(H)CHCHC_6H_5]\ [S_2COCH(CH_3)_2]$	79.4	21.2	a' 178.1 b' 213.6		a 148.2 b 144.3 c 127.0	133.1, 127.8 130.5, 126.5	-2.7	-0.11
8.	a a' b b' $PhAs[SC(NH_2):NN{=}C(H)C_6H_4OCH_3][S_2COCH(CH_3)_2]$	79.5	a 55.2 b 21.2	a' 172.7 b' 213.6		150.3	131.5, 128.6 130.6, 127.7	-2.0	-0.09
9.	a' b' $PhAs[SC(NH_2):NN{=}C(CH_3)C_6H_5][S_2COCH(CH_3)_2]$	79.3	a 13.5 b 21.0	a' 180.8 b' 213.6		157.0	133.1, 127.2 129.8, 126.1	-2.6	-0.11
10.	a' b' c' $PhAs[SC(NH_2):NN{=}C(CH_3)C_6H_4CH_3][S_2COCH(CH_3)_2]$	79.4	a 20.8 b 13.1 c 21.0	a' 178.3 b' 213.8		150.1	133.1, 127.0 129.6, 126.3	-2.6	-0.11
11.	a' b' c' $PhAs[SC(NH_2):NN{=}C(CH_3)C_6H_4OCH_3][S_2COCH(CH_3)_2]$	79.5	a 55.1 b 13.4 c 21.1	a' 173.6 b' 213.6		150.6	133.1, 127.6 129.8, 126.5	-2.1	-0.09



(A) (B)

Fig. 1: (A) Structure of PhAs $[CH_3\ CO\ CH\ C\ (CH_3)\ NHR]\ [S_2C(OPr^1)]$
(B) Structure of Ph $As[SC(NH_2)NNC(R)R^1]\ [S_2C(OPr^i)]$

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