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Syntheses, Characterization and Antimicrobial Activity of Triphenyl Antimony (V) Derivatives of Heterocyclic Dithiocarbamate

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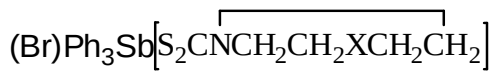
Abstract: ***The reactions of Ph_3SbBr_2 with the sodium salt of heterocyclic dithiocarbamates in 1:1 and 1:2 stoichiometries yield the derivatives***



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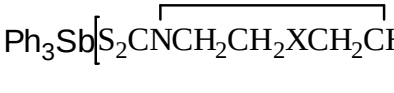


, respectively [Where X = >CH_2 (Pipdtc) (1,6), >CH–CH_3 (4-MePipdtc) (2,7), >O (Morphdtc) (3,8), >N-CH_3 (N-mePzdtc) (4,9) and >NH (Pzdtc) (5,10)]. All the newly synthesized compounds have been characterized by elemental analyses, molecular weight measurements and spectral (IR, ^1H & ^{13}C NMR and ESI mass) studies. Sodium salt of heterocyclic dithiocarbamates and their corresponding triphenyl antimony(V) bromo- and bis(dithiocarbamate) derivatives have also been screened for their antimicrobial activity against bacteria S. aureus (gram +ve), E. coli, Z. mobilies and P. aeruginosa (gram –ve) and fungi (P. chrysosprium, A. solani, P. chrysogenum and F. oxisporum).

Keywords: triphenyl antimony(V) derivatives, heterocyclic dithiocarbamates, antimicrobial activity, spectral studies, anisobidante manner

1. Introduction

The interest in the chemistry of dithiocarbamate has been renewed due to their strong complexing properties[1-7], significant antimicrobial properties[8-11] and antitumor activities which are associated with cytostatic activity similar to that of *cis*-platin as anticancer drug[12]. These metal derivaties are also employed in common pesticides and as vulcanizing or analytical agent from several decades.[13]

As we know dithiocarbamates are reduced form of thiuram disulfides[14] which exhibit rich coordination chemistry with large number of transition metals[15,16] and some main group metals due to their strong chelation property[17-19]. Complexation of dithiocarbamates by metals reduces their aqueous solubility and stabilizes the compounds as it is necessary for the application of the compounds in various fields. Most of the dithiocarbamate derivatives are sparingly soluble in water but are soluble in non polar solvents, they seems to be lipophilic enough to pass through cell membrane[13]. Heterocyclic dithiocarbamate derivatives of organo-antimony(III) have been reported from our laboratory[6,20]. However work on heterocyclic dithiocarbamate derivatives of organo-antimony(V) have not been reported so far. Due to the higher positive charge on central antimony atom these compounds are expected to exhibit more biological activity then the corresponding organo-antimony(III) compounds[21].

In view of the above, we report synthesis and characterization of triphenyl antimony(V) derivatives of bromo- and bis-heterocyclic dithiocartbamates. A comparison of antimicrobial activity of sodium salt of heterocyclic dithiocarbamates and their corresponding triphenyl antimony (V) bromo- and bis-(heterocyclic dithiocarbamate) derivatives have been made.

2. Experimental

2.1 Materials and method

All preparations and subsequent manipulations were carried out under moisture free environment. All glass wares were carefully dried prior to use. Benzene and n-hexane were dried by standard methods[24].Triphenylantimony dibromide[25] (Ph_3SbBr_2) and sodium salt of heterocyclic dithiocarbamates[26] have been synthesized by the literature methods. Antimony and sulfur were estimated by iodometric and gravimetric methods[27], respectively. IR spectra of these compounds (1-10) have been recorded as Nujol mull using KBr pellets in the range 4000-400 cm^{-1} on FT-IR spectrophotometer model 8400s Shimadzu. The NMR (^1H and ^{13}C NMR) spectra have been recorded on JEOL-FTAL spectrometer at 300.15 and 75.47 MHz, respectively in $CDCl_3$/DMSO-D_6 solutions, using TMS as an internal reference.

2.2 Preparation of triphenyl antimony(V) bromo-(Piperidinedithiocarbamate) derivative

A freshly prepared sodium salt of pipdtc [0.61g (3.32 M mole)] and Ph_3SbBr_2 [1.72g (3.35 M mole)] in benzene (~50 ml) was stirred for ~6 hours at room temperature (~25°C). The NaBr thus formed was filtered off and the excess of solvent was removed from the filtrate under reduced pressure to obtain the title compound as a creamish colour solid in quantitative yield.

The product was recrystallized from benzene and n-hexane mixture.

The product on analysis was found to have Sb % (20.52), S % (10.80) calculated for $C_{24}H_{25}SbS_2NBr$ Sb % (20.90), S% (11.08). Rests **of the compounds have** been synthesized by similar method and their synthetic and analytical data are summarized in Table III.

2.3. Preparation of tri phenyl antimony (V) bis-(Piperidinedithiocarbamate) derivative:

Ph_3SbBr_2 [1.52 g (2.96 M mole)]was mixed with freshly prepared sodium salts of pipdtc [1.08 g (5.89 M mole)], dissolved in benzene (~50ml). The reaction mixture was stirred for ~6 hours at room temperature (~25°C). The NaBr thus formed was filtered off then the excess solvent was removed under reduced pressure to obtain the title compound as a white coloured solid in quantitative yield. The product was recrystallized from benzene and n-hexane mixture.

The product on analysis found to have Sb% (18.07), S%(19.03) calculated for $C_{30}H_{35}SbN_2S_4$ Sb %(18.90), S% (19.95). Rest of the compounds have also been synthesized by similar method and their synthetic and analytical data are summarized in Table III.

(iii) Test Organisms
All the microbial strains (bacteria and fungi) of human pathogens, used in the antimicrobial bioassay were procured from Institute of Microbial Technology (IMTECH), Chandigarh.

Bacteria: Pure cultures of all experimental bacteria viz. Gram-negative bacteria such as Escherichia coli (MTCC 1652), Zymomonas mobilis (MTCC 88) pseudomonas aeruginosa (MTCC 4676); and the Gram-positive bacteria such as Staphylococcus aureus (MTCC 3160) were maintained on nutrient agar (Hi-media) in The Institute of Applied Sciences and Biotechnology (Chemind Biosolutions Laboratory), Jaipur. Each bacterial culture was further maintained on the same medium after every 48 hours of transferring and stored at 4°C before use in experiments.

Fungi: Pure cultures of all experimental fungi viz. Phanerochaete chrysosprium (MTCC 787), Alternaria solani (MTCC 2101), Penicillium chrysogenum (MTCC 161) and F. oxisporum(MTCC 6659) were maintained on Potato Dextrose Agar (PDA) (Hi-media) and the cultures stored at 4°C and sub-cultured once in a month in the Institute of Applied Sciences and Biotechnology (Chemind Biosolutions Laboratory), Jaipur.

(iv) Media preparation and its sterilization
For agar well diffusion method (Bauer et al., 1996), antimicrobial susceptibility was tested on solid (Agar-agar) media in petri plates. All the media prepared was then sterilized by autoclaving the media at (121°C) for 20 min.

(v) Preparation of plates
Prepared agar was allowed to be sterilized and then again allowed to cool till 50 °C in a water-bath. Pouring of about 20 ml agar into pre-labeled sterile Petri dishes was made. They were then permitted to set at room temperature and were dried as no drops of moisture remain on the surface of the agar.

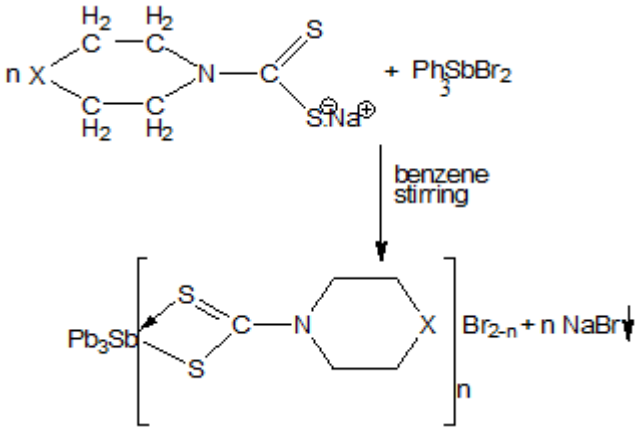
(vi) Agar well diffusion
Antibacterial and Antifungal activities of the plant extract were tested using well diffusion method. Nutrient agar (NA) and Potato Dextrose Agar (PDA) plates were swabbed (sterile cotton swabs) with 8 hour old - broth culture of respective bacteria and fungi. Wells were made on the agar surface with 6mm in diameter and about 2 cm a part punctured in the culture medium using sterile cork borers. The plates were then turned up side down and the wells were labeled with a marker. Stock solution of each plant extract was prepared at a concentration of 1 mg/ml in methanol. Each of the wells was filled with 100 µl of the compound using sterile syringe. Only one well was filled with 100 µl of DMSO which served as a negative or a positive control, respectively. The plates were incubated at 37 ºC for 24 hours for bacterial and 25 ºC for 48 hours for fungal activity. The plates were observed for the zone clearance around the wells.

The zone of inhibition was calculated by measuring the diameter of the inhibition zone around the well (in mm) including the well diameter. The readings were taken in three different fixed directions in all three replicates and the average values were tabulated.

3. Results and Discussion

3.1 Synthesis

The reactions of Ph_3SbBr_2 with the sodium salt of hetrocyclic dithiocarbamates in 1:1 and 1:2 molar ratio yield corresponding triphenylantimony(V) bromo- and bis-(heterocyclic dithiocarbamate) derivatives.



[Where X = >CH_2 (Pipdtc), CH_3-CH< (4-MePipdtc), >N-CH_3 (N-MePzdtc), >NH (Pzdtc) and >O(Morphdtc)] [n = 1or 2]

Bromo triphenylantimony (V)-(heterocyclic dithiocarbamate) derivatives are colored solids whereas triphenyl antimony (V) bis-dithiocarbamates derivatives are white crystalline solids. All these derivatives (**1-10**) are soluble in common organic solvents and are purified by benzene and n-hexane mixture.

3.2 IR spectra

IR spectra of triphenylantimony(V) bromo- and bis(dithiocarbamate) derivatives **(1-10)** exhibit strong band in the region 1440-1450 cm^{-1} due to ν(C=N) stretching vibrations and their comparison to the corresponding sodium salt of dithiocarbamates show small shifting towards higher frequencies (~15-25 cm^{-1}). Splitted bands in the region ~1125-995 cm^{-1} due to ν(C=S) stretching vibrations have been observed for these triphenylantimony(V) bromo and bis(heterocyclic dithiocarbamate) derivatives as well as their corresponding sodium salts of heterocyclic dithiocarbamate.

To determine the coordination pattern of these triphenylantimony (V) dithiocarbamate derivatives the value of $\Delta\nu[\nu(CS_2)_{assy} - \nu(CS_2)_{symm}]$ may be used. The $\nu(CS_2)_{assy}$ and $\nu(CS_2)_{symm}$ stretching bands appeared at 1110-1125 cm^{-1} and 995-1010 cm^{-1}, respectively. The $\Delta\nu$ values $[\nu(CS_2)_{assy} - \nu(CS_2)_{symm}]$ observed in the range 105-125 cm^{-1} are smaller than the observed $\Delta\nu$ values for bidentatelly coordinated derivatives and are larger than the corresponding free sodium dithiocarbamates values, as reported earlier in the literature[2]. In view of the above mentioned data it can be concluded that the dithiocarbamate ligand moieties are coordinated to antimony atom in an anisobidentate manner. A sharp absorption band at 450±20 cm^{-1} may be assigned to ν(Sb-S) stretching, which also indicates the bonding of central antimony with sulphur atom .A band due to ν(Sb-C) has also been observed at 470-455 cm^{-1} in all these derivatives.

3.3 ^{1}H NMR Spectra

The ^{1}H NMR spectra of these triphenyl antimony(V) bromo- and bis(heterocyclic dithiocarbamate) derivatives **(1-10)** have been recorded in $CDCl_3$ and DMSO-d_6 and their data are summarized in Table I.

The ^{1}H signals due to the, $–CH_2–$, $–CH(CH_3)–$, $N(CH_3)–$, $CH_2–O–CH_2$, have been observed at δ 1.57 and 1.76, 2.34 and 3.09, 3.10 and 2.60, 2.78 and 2.76, respectively and do not show any appreciable shift in their positions as compared to their corresponding sodium salt of dithiocarbamate ligands . The aromatic ring attached to antimony atom has also appeared in the region δ 7.57-8.50 as a multiplet.

3.4 ^{13}C NMR Spectra

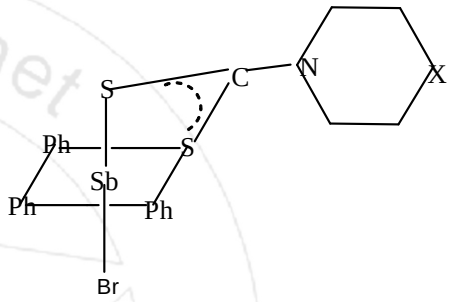
The ^{13}C NMR of these derivatives are interpreted and data are summarized in Table II.

A comparative study of ^{13}C NMR data of triphenylantimony bromo and bis-heterocyclic dithiocarbamate derivatives (1-10) with the corresponding sodium salts of ligands indicates the remarkable up field shift of ~15 - 20 ppm in the position of CS_2 carbon signal, due to strong chelation of dithiocarbamate moiety. Remaining carbon signals of triphenyl antimony(V) bromo- and bis(heterocyclic dithiocarbamate) derivatives have been appeared at their expected positions.

The **corrected chemical shift value**,δ' [22] and **Hammett-Taft constant** σR°[23] for phenyl carbons attached to antimony atom (V) have been calculated by the relation $\delta' =$ Cp–Cm (where δ Cp and δ Cm are the chemical shift values of para and meta carbons of phenyl ring , attached to antimony atom) and by the equation $\delta = 23.06\,\sigma R°$, respectively. The δ' and σR° values are found to be negative for compound (1-5) and (6-10) in range of δ (-) 3.66 to 1.18 and δ (-) 0.07 to (-) 0.24 and δ (-) 2.25 to (–) 3.34 and δ (-) 0.11 to (-) 0.27 respectively. These negative values are indicative of the release of electron from metal towards phenyl ring through dπ - pπ conjugation in these derivatives and poor donor capability of antimony atom.

3.5 Proposed Structure

All the above mentioned spectroscopic evidences show that the dithiocarbamate moieties are coordinated to antimony atom in an anisobidante manner therefore , the following structures in which antimony atom aquires Octahedral geometry for compounds **1-5** and Pentagonal bipyramidal geometry for compounds **6-10** have been proposed.



(Where X = >CH_2, >CH–CH_3, >N–CH_3, and >O)
Figure 1: Proposed structure for compounds (1-5)

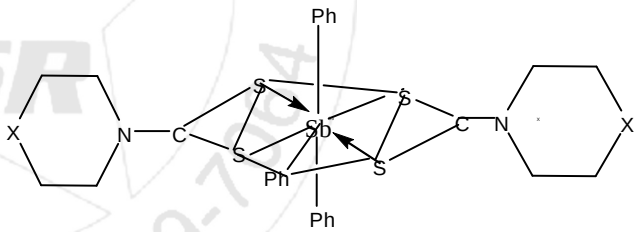


Figure 2: Proposed structure for compounds (6 -10)

4. Biologycal Activity

Bromo-triphenylantimony(V) heterocyclic dithiocarbamate and triphenylantimony(V) bis(heterocyclic dithiocarbamate) derivatives were screened against *E. coli, Z .mobilies, S. aureus, P.aeruginosa* (bacteria) and *A. solani, P. chrysogenum, P. chrysosprium, F. oxisporum* (fungi) to examine their inhibition zone towards the tested microorganisms. The results indicate that the bromo-phenyl antimony(V) dithiocarbamates were more inhibitory than triphenyl antimony(V) bis(dithiocarbamate)derivatives as the tested organism (Table-) shows that the inhibition zone were higher in the bromo-triphenylantimony(V) derivatives **(1-5)** as compared to triphenylantimony(V) bis(dithiocarbamates) derivatives. It shows higher activity due to presence of bromine atom[27]. The enhanced activity of metal derivatives may be ascribed to the increased liphophilic nature of these derivatives arising due to the chelation. The observed toxicity with bacteria and fungi can be explained on the basis of the tweedy's chelation theory

and overtone's concept[25]. The results indicate that the order of the activity of these derivatives and free ligands is 1-5 > 6-10 > L_1-L_5.The data are summarized in table VI.

5. Acknowledgement

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Table I: 1H NMR spectral data of Triphenyl antimony (V) bromo and bis (hetrocyclic dithiocarbamates)in ppm

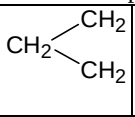
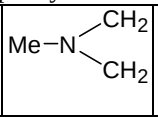
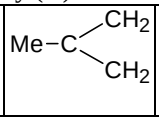
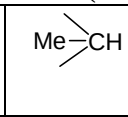
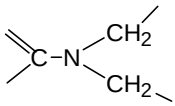
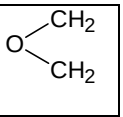
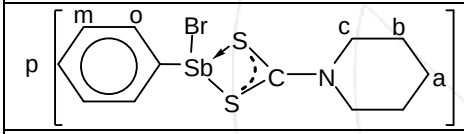
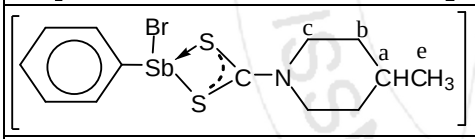
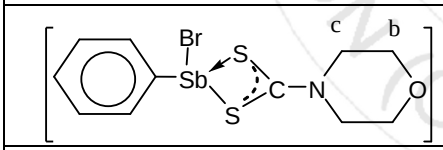
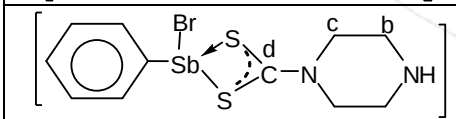
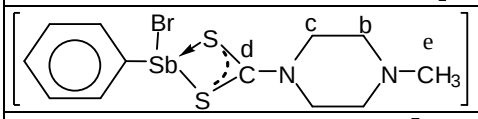
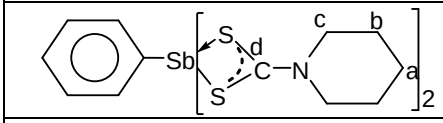
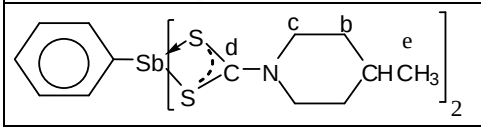
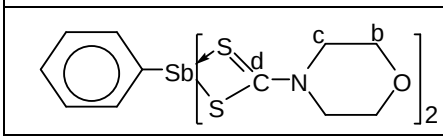
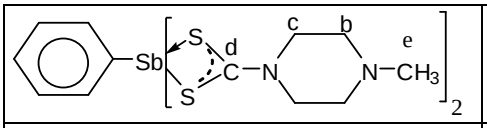
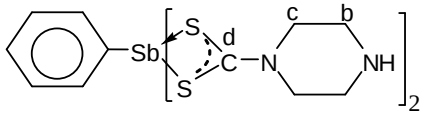
Compound	–CH$_3$						–Sb-Ph	
[Ph$_3$Sb(Pipdtc)Br]	-	1.57-1.76 (m)	-	-	-	4.24 (s)	7.59-8.28 (m) (5H)	-
[Ph$_3$Sb(4-Me-pipdtc Br]	0.94-0.96 (m) -	-	-	2.34-2.94 t(4H)	1.23-1.27 (m) (1H)	4.04-4.77 (d) (4H)	7.57-8.27 m (5H)	
[Ph$_3$Sb(Morphdtc)Br]	-	-	-			3.70	7.26 - 8.24(m)	2.78,2.47
[Ph$_3$Sb(N-Me pzdtc)Br]	1.25 (s)	-	3.10-3.48	-	-	3.48- 3.87(m)	7.59-8.28 (m)	-
[Ph$_3$Sb(Pzdtc)Br]	-	-	-	-		3.85-4.30	7.75 -8.18 (m)	
[Ph$_3$Sb(Pipdtc)$_2$]		1.76-2.56 (m)	-	-	-	4.23 (s)	7.90-8.18(m)	-
[Ph$_3$Sb(4-Me pipdtc)$_2$]	0.95-0.97		-	3.09-3.46	1.24-1.33	4.83-4.88	7.75-8.10 (m)	-
[Ph$_3$Sb(Morphdtc)$_2$]	-	-	-	-	-	3.68-372	7.26-8.20(m)	2.74, 2.76, 2.78 (t)
[Ph$_3$Sb(N- Me-pzdtc)$_2$	1.25-2.12	-	2.60 -2.70	-	-	4.12 (s)	7.72-8.07 (m)	-
[Ph$_3$Sb[(Pzdtc)$_2$]	-	-	-	-	-	-	7.59 - 8.28 (m)	-

Table II: ^{13}C NMR spectral data of new Triphenyl antimony(V)bromo and bis(heterodithiocarbamates) (1-10)

Complex	Ca	Cb	Cc	Cd	Ce	Sb-C$_6$H$_5$*			Ci	□ ′	□ R^0
						C(p)	C(m)	C(o)			
	22.36	26.83	52.78	192.65		128.28	129.46	131.33	132.43	-1.18	-0.07
	22.45	44.58	52.77	192.60	55.80	128.28	130.03	131.42	133.70	-1.75	-0.24
		51.64	53.36	192.86		128.10	131.76	134.46	148.67	-3.66	-0.15
		52.53	50.95	193.86		128.59	131.21	134.15	141.01	-3.06	-0.13
		51.17	39.15	193.85	51.37	128.90	131.79	135.17	142.42	-2.89	-0.12
	18.40	54.33	58.34	193.58		128.56	131.21	134.00	138.35	-2.90	-0.14
	21.33	31.18	52.07	192.74	51.20	128.31	131.65	136.17	141.67	-3.34	-0.14
		76.13	66.12	193.83		128.54	130.94	135.03	138.36	-2.25	-0.25

		50.78	45.57	193.59	54.49	128.33	130.79	133.49	138.37	-2.46	-0.27
		52.75	51.99	193.83		128.39	130.83	133.89	138.36	-2.55	-0.11

*Metal phenyl value are given in the order C(i), C(o), C(m) and C(p), respectively.

Table 3: Synthetic and analytical data of Triphenylantimony(V) bromo and bis(heterocyclic dithiocarbamate) derivatives

S.No	Complex	Reactants (gm) / m mole		Product (% yield)	NaBr (gm) found (calc)	M.P.° (±1°C)	Mol. wt. Found (Calc.)	Analyses % Found (Calc)	
		Na salt of hetero cyclic dithiocarbamate	Ph_3SbBr2					S	Sb
1.	$Ph_3(Br)Sb(Pipdtc)$	(0.61) 3.32	(1.72) 3.35	80	0.34 (0.40)	153	594.26 (594.29)	10.80 (11.08)	20.52 (20.90)
2.	$Ph_3(Br)Sb(4\text{-}MePipdtc)$	(0.64) 3.24	(1.68) 3.27	78	0.33 (0.39)	158	607.28 608.25	10.55 (11.05)	20.04 20.75
3.	$Ph_3(Br)Sb(Morphdtc)$	(0.62) 3.34	(1.72) 3.35	78	0.34 (0.37)	157	595.20 (596.11)	10.77 (11.24)	20.45 (21.05)
4.	$Ph_3(Br)Sb(N\text{-}mePzdtc)$	(0.65) 3.37	(1.68) 3.27	80	0.33 (0.40)	162	608.25 (608.96)	10.54. (10.98)	20.01 (20.65)
5.	$Ph_3(Br)Sb(Pzdtc)$	(0.62) 3.34	(1.72) 3.35	75	0.34 (0.36)	140	594.21 (595.08)	10.79 (11.20)	20.48 (20.90)
6.	$Ph_3Sb(Pipdtc)_2$	(1.08) 5.89	(1.52) 2.96	83	0.61 (0.69)	178	673.53 (674.10)	19.03 (19.95)	18.07 (18.90)
7.	$Pb_3Sb(4\text{-}MePipdtc)_2$	(1.12) 5.67	(1.46) 2.84	80	0.58 (0.63)	143	701.53 (702.21)	18.28 18.70	17.35 (18.02)
8.	$Ph_3Sb(Morphdtc)_2$	(1.09) 5.88	(1.51) 2.94	80	0.58 (0.63)	108	667.57 (668.23)	19.20 (20.05)	18.23 (18.79)
9.	$Ph_3Sb(N\text{-}mePzdtc)_2$	(1.12) 5.64	(1.45) 2.82	78	0.58 (0.65)	120	703.65 (676.21)	18.22 (19.08)	17.30 (17.91)
10.	$Ph_3Sb(Pzdtc)_2$	(1.09) 5.91	(1.51) 2.94	76	(0.60) (0.67)	135	675.59 (676.21)	18.98 (19.74)	18.02 (18.85)