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Biological Activities of some Sterically Congested Organoantimony (v)-Carboxylates, -(Halo) Carboxylates and -Pseudohalides

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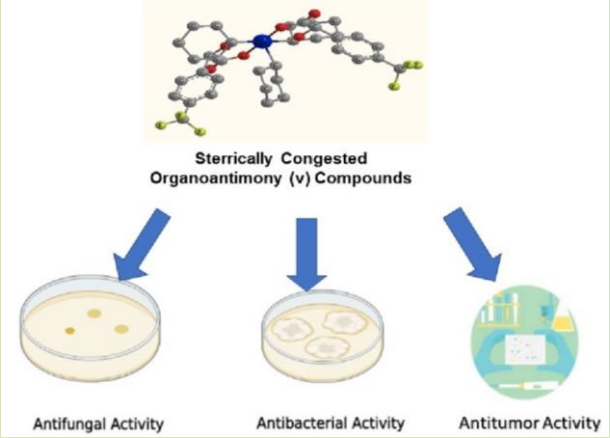
ABSTRACT: Since the initial discovery of applications of Antimony complexes in the clinical treatment of many kinds of disease, efficiency of Antimony complexes in inhibiting the expansion of various types of Antifungals, Antibacterial and Antitumor disease. A series of Sterically Congested Organoantimony(v)-Carboxylates, -(Halo) Carboxylates and –Pueudohalides [(α-$C_6H_{11})_3Sb(NCS)_2$, (cyclo-$C_6H_{11})_3Sb(NCS)_2$, (α-$C_6H_{11})_3Sb(N_3)_2$, (cyclo-$C_6H_{11})_3Sb(N_3)_2$, (cyclo-$C_6H_{11})_3Sb$(2-Pyrazine Carbox.)$_2$] complexes were evaluated for their activity against Antifungal, Antibacterial and Antitumor disease. One of the main obstacles in my work is that complex should sometimes not be oriented correctly. In this research work the derivatives shown promising results against Antifungal, Antibacterial and Antitumor disease and the derivatives oriented correctly with the metal which shown optimum biological activity.

Graphical Representation:



Keywords: Organoantimony (v), Antifungal, Antibacterial, Antitumor.

A voluminous amount of work has been reported on the various synthetic, reactivity, structural and biocidal activity aspects of organoantimony derivatives, where antimony is present either in +3 or +5 oxidation state Doak and Freedman (1970). The detailed indepth investigation by various group of workers on organoantimony compound having different organic group(s) bound to antimony reveals that geometry and bonding around antimony is significantly affected on altering the composition and nature of organic groups surrounding antimony (Lipshultz *et al*., 2021).

However, despite a considerable and varied interest involved during the past few decades, the investigations more or less are confined to aryl (phenyl, p-tolyl, pentafluorophenyl, p-fluorophenyl and p-chlorophenyl etc.) and alkyl (methyl, ethyl and butyl) derivatives (Doak and Freedman1970). Only a very little amount of research has been done on those with sterically bigger moieties (naphthyl, cyclohexyl, and mesityl group) linked to antimony (Challenger & Pritchard 1924; Hartmann *et al.*, 1961; Issleib *et al.*, 1964; Sharma and Rastogi 1983). Contrastingly, the literature Avasthi *et al.* (1982); Blunden *et al.* (1987); Vornefeld *et al.* (1992) is well-documented with respect to equivalent derivatives of Group 14 elements (M=Ge, Sn, Pb)]. Additionally, sterically hindered cyclohexyltin compounds are useful biocides and are employed as insecticides, bacteriocide, fungicides, and miticides (Lai Ziyang *et al.*, 2022). They have been tested for their antileukemia activity, and in-vitro the effects of cyclohexyltin peptide derivatives have been studied on breast cancer cells by Vornefeld *et al.* (1992).

With the exception of early studies on the synthesis of tri(cyclohexyl)antimony(V) dicarboxylates Avasthi *et al.* (1982); Chirca Ionut *et al.* (2018), R_3SbX_2 (R=α-$C_{10}H_7$, cyclo-C_6H_{11}; X= Cl, Br), and triorganoantimony(V) dihalides (Agnihotri *et al.*, 2002); Rieche *et al.* (1964). Agnihotri *et al.* (2002); Rathore *et al.* (1964) reported that interhalogen and halo-pseudohalogens add oxidatively to tri(α-naphthy) antimony (III) to afford penta-coordinate products, and hydrolysis of R_3SbCl_2 (R=α-$C_{10}H_7$, Cyclo-C_6H_{11}) afforded $(R_3SbCl)_2O$, type compounds. Tri(-naphthyl) antimony (III) has also been reported to insert into the N-chloro bonds of N-chlorosuccinimide and N-chlorophthalimide. In addition, the same researchers produced penta-coordinate tri(α-naphthyl) antimony(V) -haloimides, -halo amines, -halo carboxylates, and -dioximates with the general formulas $(\alpha\text{-}C_{10}H_7)_3SbLX$ and $(\alpha\text{-}C_{10}H_7)_3SbL_2$(where L=amide, amine, carboxylate, and oxime; X= These were made by reacting the appropriate metal salts of the organic ligands with tri(α-naphthyl) antimony(V) dihalide, and a possible trigonal-bipyramidal structure has been proposed for these compounds.

Naphthyl and cyclohexyl derivatives though little studied are much more thermally and hydrolytically stable and thus affect the biological activity (Varadwaj *et al.*, 2022). The synthetic methods for such bulkier groups containing compounds are in fact much more tedious and need greater degree of attention. In most of the cases the yields of the products are poor which is probably one of the major discouraging factors for pursuing research investigation in this little exploited area. Nevertheless, compounds of such bulky moieties being stable, solid and sharp melting with reduced toxicity, arouse interest to study more about their synthetic pattern, reactivity, geometry, mode of bonding of other anions attached to the metal atom and on top of that sterric congestion offered by the presence of bulky groups.

One of the principal objectives of this investigation was to explore the role of sterically larger groups, viz., cyclohexyl and to a limited extent α-naphthyl groups bound to antimony, on the synthesis, biological activity and reactions of antimony compounds, vis-á-vis well studied aryl and alkyl antimony derivatives (Sharutin *et al.*, 2020). Though a complete study Would have involved corresponding mesityl derivatives as well, providing more insight and depth in to the chemical behavior of such compounds as well as comparison, the same could not be done due to very poor yield. It is more so in case of cyclohexyl derivatives which could never be isolated as tricyclohexylantimony (III) (Hartmann *et al.*, 1961), and the pyrophoric nature of mesityl magnesium bromide inhibited us to carry on further investigation due to limited laboratory facilities, *viz.*, absence of argon atmosphere and other constraints.

RESULTS AND DISCUSSION

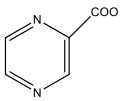
Triorganoantimony(V) dihalides were reacted in 1:1 and 1:2 molar ratios with pseudohalides and the silver salt of the appropriate carboxylic acid to produce a series of triorganoantimony(V) -dicarboxylates and triorganoantimony(V) -pseudohalides, -halo carboxylates. All reactions were conducted in a dark, oxygen-free environment using anhydrous organic solvent at room temperature (in case of reactions with silver salt of acids). The equation (Eq. 1-3) below can be used to represent these reactions:

$$R_3SbCl_2 \xrightarrow{\text{THF, Benzene or Acetone}} R_3SbL_2 + 2MCl \quad (1)$$

Where, R= Cyclo-C_6H_{11}, α-C_6H_{11} (Naphthyl)
ML = KSCN (M= K, L=SCN) OR NaN_3 (M = Na, L= N_3)

$$Cy_3SbCl_2 + 2AgL \xrightarrow{\text{THF or Benzene}} Cy_3SbL_2 + 2AgCl \quad (2)$$

Where, L= p-$CF_3C_6H_4$, p-$OCH_3C_6H_4$, RCH(OH)COO (R=C_6H_5, and o-OHC_6H_4COO)

and  Cy = cyclo-C_6H_{11}

$$Cy_3SbCl_2 + AgL \xrightarrow{\text{THF}} Cy_3Sb(Cl)L + AgCl \quad (3)$$

Where, Cy = cyclo-C_6H_{11}; L = o-OHC_6H_4COO, RCH(OH)COO (R=C_6H5, p-CFC_6H_4 and p- $OCH_3C_6H_4$).

In the instance of pyrazine carboxylic acid, the 1:2 and 1:1 molar ratio reaction went smoothly and produced different products for all the α- and β-hydroxy carboxylic acid moieties (Eq.1-3). With triphenylantimony(V) dichloride, mandelic acid and its derivatives create cyclometalated, but this was not the case here. These findings suggest that the cyclization is prevented by sterically hindered cyclohexyl groups. The recently synthesised chemicals are quite stable to air and moisture and do not disintegrate in storage for several days. With the exception of the losses sustained throughout the workup process, the product yields were almost quantitative. In the majority of organic solvents, the complexes are easily soluble. With the exception of a-naphthyl derivatives, which are off-white and have acute melting temperatures, they are all white

crystalline solids. The complexes have been identified using IR, NMR, and elemental analysis methods. It has been determined that they are non-electrolytes and monomeric in nature based on molecular weight data from freezing benzene and conductivity tests of 10 M solutions in acetonitrile at room temperature.

Infrared Spectra

All of the compounds (1–13) had infrared spectra that were recorded on an FT IR spectrophotometer in the 4000-400 cm^{-1} range (Shimadzu 8201 PC). The published values of similar tri(α-naphthyl) antimony are closely correlated with the IR absorptions for the -naphthyl group linked to antimony Silverstein *et al.* (1981). The cyclohexyl group's related absorption bands are comparable to those of analogous organotin compounds (Vornefeld *et al.* 1992). The newly synthesised compounds' diagnostic IR absorption bands have been found and are given in Table 3.

The α-naphthyl and cyclohexyl groups in all the compounds' infrared spectra exhibit essentially comparable absorption. The Sb-C stretching frequency associated with the y-mode was found to be a medium to weak band between 426 and 405 cm^{-1} (Doak *et al.*, 1965; Li *et al.*, 1998; Li *et al.*, 2001). The halo carboxylates' spectra do not exhibit the antimony-halogen stretching frequency in the 4000-400 cm^{-1} range.

The position and distance (Δv= νasym- νsym) between the asymmetric and symmetric OCO stretching modes in an IR spectrum can be used to determine the carboxylate coordination mode (Singhal *et al.*, 1987; Gibbons and Sowerby 1998; Li *et al.*, 2002). These compounds' IR spectra show distinctive carboxylate bands in the range of νasym (OCO) between 1668 and 1634 cm^{-1} and Nsym OCO) between 1383 and 1300 cm^{-1}. Because there is no contact between the carbonyl oxygen atoms of the carboxylate groups and the antimony atom, which would indicate the presence of monodentate "ester type", both compounds are classified as belonging to the same class based on their (OCO) values (338 - 274cm^{-1}) (Li *et al.*, 2001).

The absorption bands resulting from the -NCS and -N groups are substantially similar to those previously described for other covalent organometallic pseudohalides (Agnihotri *et al.*, 2002; Thayer *et al.*, 1967; Norbury *et al.*, 1968; Goel and Ridley 1974; Bhattacharya *et al.*, 1982; Srivastava and Bhattacharya (1966). For compounds (11, 13), the symmetric stretching frequency was found at (1323, 1272 cm^{-1}) as a strong to medium intensity band, whereas the asymmetric vibration was detected at (2069, 2074 cm^{-1}) as a very strong band. It is possible to attribute a medium to weak intensity band at (665, 677cm^{-1}) to the bending mode δ(N3). These bands are located at a location that is very similar to that of Group-14 and Group-15 organometal Azides that have been covalently bound Goel and Ridley (1974); Bhattacharya *et al.* (1982); Goel (1969). In the case of compounds (10, 12), a very strong and wide band caused by asymmetric stretching, (NCS), at (2014, 2025cm^{-1}), respectively, points to an iso bonding, or a nitrogen bond. The emergence of the weak to medium intensity C-S symmetric stretching at (835, 793 cm^{-1}) and a weak band due to (N-C-S) bending are two additional significant characteristics in favour of nitrogen linkage (463, 485cm^{-1}). The iso structure (N=C=S) is favoured by these vibrational modes Agnihotri *et al.* (2002); Thayer *et al.* (1967); Norbury *et al.* (1968); Srivastava and Bhattacharya (1966).

^{1}H NMR SPECTRA

On a Brucker DRX-300 (300 MHz FT NMR) spectrometer, ^{1}H NMR spectra of the representative compounds were captured. Tetramethyl silane (TMS) was used as a reference, and chemical shifts were reported as δ(ppm) from that. The integration of the spectra is fairly consistent with the values that are predicted for the complex compounds. Table provides a list of recently synthesised substances' proton magnetic resonance spectra Table (4).

The chemical shifts of the cyclohexyl ring's protons are multiplets and range from δ (1.13 to 3.85) ppm. Low to high long-range coupling is the cause of the multiplet appearance of protons of the cyclohexyl ring signals. When four sigma bonds between interacting protons take on a W-arrangement, as in the case of the 1,3-diequatorial protons in a stiff cyclohexyl system, this sort of coupling is seen (Fig. 1). In other words, this is caused by the stiff cyclohexyl system's protons being coupled in diaxial, axial-equatorial, and diequatorial directions.

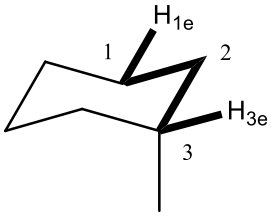


Fig. 1.

Each axial and equatorial proton of the cyclohexyl ring emits a distinct -value of signals. The signal range for the proton (H1) attached to the ipso-carbon of the cyclohexyl ring is δ (3.63–3.86) ppm. The equatorial proton at position 3 and the axial proton at position 2 both encountered the same electronic environment and were determined to be magnetically equivalent, emitting signals at the same spot in the range of δ (1.70-1.78) ppm. The indications for cyclohexyl protons were thus discovered to be in strong agreement with those previously reported Li *et al.* (1998); Vornefeld *et al.* (1992). The proton signal for α-hydrogen is present in the range of δ (4.96-4.13 ppm) in the α-hydroxy acids containing α-hydrogen (3,5,7,8). The methoxy proton was detected as a singlet in Compounds (7,8) at δ (3.78, 3.8) ppm, respectively. The facts are summarised in the Table. The multiplet of the phenyl ring protons occurs in the region of δ (6.58-7.93) ppm (4). At 89.04(s), 8.79(d), and 8.87(d) ppm, respectively, the three magnetically non-equivalent protons (H2, H3, and H4) of 2-pyrazine carboxylate (9) were detected. The peaks of the ligand are equivalent to those of free carboxylic acids and earlier reported organometallic compounds

that contain analogous molecules Barucki *et al.* (2001); Barucki *et al*. (2000).

^{13}C NMR SPECTRRA

On a 300 MHz FT NMR (Bruker DRX-300) spectrometer running at 75 MHz with $CDCl_3$as the solvent and reference (δ 76.0 ppm), ^{13}C NMR spectra of typical compounds were captured. Chemical shifts were reported as δ(ppm). Table compares, identifies, and lists the peaks (5).

At various -values in the range δ (25.03-56.73) ppm, the chemical shift of four magnetically non-equivalent carbons of the cyclohexyl ring was detected. In every example, the ipso-carbon (C_1) was completely exposed, and the signal falls within the range of δ (55.10-56.73) ppm. When compared to free acid moieties, the carbon centres of carboxylate groups in all complexes shift to a lower field due to deshielding, which suggests that the carboxylate group participated in antimony coordination Holecek *et al*. (1986). Chemical shift values that fall within a certain range are recognised for the position of the carbon in ligands. The chemical shift of the ligands in all of the sterically crowded organoantimony carboxylates differs significantly from the chemical shift of the carbon in free acid for the carbon centres C_1 and C_2, but other carbons are essentially unaffected. When compared to that of triphenylantimony(V) carboxylates, the influence of the sterically hindered cyclohexyl group on the chemical shift of the carbon centres of the ligand acid is notable Barucki *et al.* (2001); Barucki *et al*. (2000). The existence of all tricyclohexyl group carbon peaks and the ligand acid, on the one hand, and the alteration in the chemical shift of various ligand acid carbon centres relative to those of free acid, on the other, confirm the establishment of the Sb-O-C(O) bond (Verma, 2020).

Consequently, it can be deduced from spectral data combined with molecular weight and conductance data that the newly synthesised compounds have a monomeric, covalent constitution with trigonal-bipyramidal geometry surrounding antimony (Fig. 2). The three cyclohexyl or α-naphthyl groups are located in the equatorial positions, while the ligands or chlorine are located in the apical or axial places (Jain *et al*., 1980).

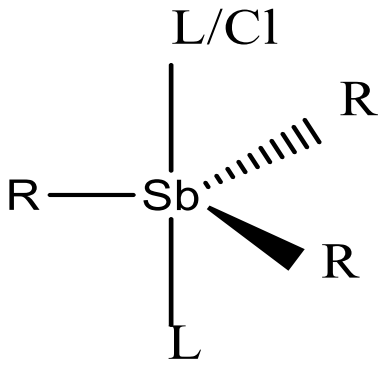
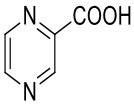


Fig. 2

R= cyclo-C_6H_{11} or α-$C_{10}H_7$ (Naphthyl);

L= o-OHC_6H_5COO, R' CH(OH)COO, NCS or



N_3

and R' = C_6H_5, p-$CF_3C_6H_4$ or p-$OCH_3C_6H_4$

EXPERIMENTAL

The mode of preparation of $RSbCl_2$ (R= cyclo-C_6H_{11} or α-$C_{10}H_7$). Tricyclohexylantimony Dichloride: A Grignard solution was prepared from (49.17g. 0.3 mol) of cyclohexyl bromide and Mg turning (7.2g, 0.3mol) in dry THF (150mL) solvent in presence of nitrogen atmosphere with exclusion of light. A solution of $SbCl_3$ (22.8g, 0.1 mol) in dry benzene (100mL) was added dropwise, the reaction mixture was stirred vigrously after complete addition of $SbCl_3$, then refluxed for 2h. This solution containing tricyclohexylantimony was bubbled with freshly generated chlorine gas for the reaction mixture was hydrolysed by addition of 10% HCI in cold water. The organic layer was separated and dried over anhydrous Na_2SO_4. Removal of solvent followed by crystallization from absolute ethyl alcohol afforded needle shaped crystals of tricyclohexylantimony(V) dichloride, (cyclo-$C_6H_{11})_3SbCl_2$

M.P 202°C Lit: 203°C [6,7] Yield: 30.9 g (70%).

Tri(α-naphthyl) antimony(V) Dichloride naphthyl was prepared as a white crystalline solid by passing freshly generated chlorine gas to a stirred solution of tri(α-naphthyl) antimony (8.0 g. 0.015mol) in carbon tetrachloride diluted with light petroleum ether (40-60℃) Compound was repeatedly extracted with hot chloroform.

M.P. 260°C Lit.: 260°C [5] Yield: 6.39 g (70%)

Without additional purification, potassium thiocyanate and sodium azide were employed as-is. The carboxylicacids were employed as silver salts, which were created by reacting the sodium salt of the appropriate acid with silver nitrate. Tetrahydrofuran (THF) was created by distilling sodium-benzophenone under a nitrogen environment. To keep out oxygen and moisture, special measures were required. To prevent degradation, the silver salt reactions were carried out in a dark environment.

Typical examples of R_3SbX_2. (R=Cyclohexyl or α-naphthyl, X-OCOR, N_3 or SCN) and tricyclohexylantimony(V) (chloro) carboxylates are described below in detail, and the condition for the other reaction are summarised in Table (1). Analytical data are given in Tables (2-5).

1:2 Molar Ratio Reaction of Tricyclohexylantimony(V) Dichloride with Silver Salt of Salicylic Acid (1)

The silver salt of salicylic acid (0.490g, 2.0mmol) was added to a heterogeneous solution of tricyclohexylantimony(V)dichloride (0.441g, 1.0mmol) and THF (20mL), and the mixture was agitated at room temperature for 24 hours. AgCl was filtered out as a result of this formation. Tricyclohexylantimony(V) disalicylate was obtained by concentrating the filtrate in vacuo (2–3 mL), adding n–hexane (5 mL), and obtaining a white crystalline substance (1)

M.P.: 146°C Yield: 0.440g (68.3%6)

Similarly, 1:1 molar ratio reaction of tricyclohexylantimony(V) dichloride (0.441g, 1.0mmol) with silver salt of salicylic acid (0.245g 1.0mmol) in THF (15mL) afforded white crystalline solid characterised as tricyclohexylantimony(V) (chloro) (salicylate) (2)

M.P:108°C Yield: 0.374g (69.0%)

1:2 Molar Ratio Reaction of Tricyclohexylantimony(V) Dichloride. with Silver Salt of (RS)-Mandelic Acid (3)

A mixture of tricyclohexylantimony(V) dichloride (0.441 g, 1.0 mmol) and the silver salt of (RS)-mandelic acid (0.5180, 2.0 mmol) in THF (20 mL) was agitated at room temperature for 24 hours in a dry nitrogen environment. The heterogeneous solution containing precipitate of AgCl was filtered. The filtrate on concentration in vacuo followed by addition of n-hexane afforded white crystalline solid. The chemical, which was identified as (RS)-tricyclohexylantimony(V) dimandelate, crystallised from a combination of THF and petroleum ether at a temperature between 60 and 80 degrees Celsius (3).

M.P.: 112°C Yield:0.418g (62.2%)

In the same manner, 1:1 molar ratio reaction of tricyclohexylantimony(V) dichloride (0.441g, 1.0mmol) with silver salt of (RS)-mandelic acid (0.259g, 1.0mmol) in THF (20mL) afforded the white crystalline compound characterised as (RS)-tricyclohexylantimony(V)(chloro) (mandelate) (4).

M.P.: 88°C Yield 0.353g (64.5 %)

1:2 Molar Ratio Reaction of Tricyclohexylantimony(V) Dichloride with Silver Salt of 2-Pyrazine Carboxylic Acid (9)

Tricyclohexylantimony(V) dichloride (0.441g, 1.0mmol) and the silver salt of 2-pyrazine carboxylic acid (0.462g, 2.0 mmol) were mixed uniformly and agitated in dry benzene (20mL) for 24 hours at room temperature without the presence of oxygen or moisture. AgCl that resulted was filtered out. The entire solvent was evaporated under vacuum to create white crystal. A solid compound called tricyclohexylantimony(V) di (2-pyrazine carboxylate was recrystallized from benzene as petroleum ether (60-80°C) in the ratio 1:3. (9).

M.P.: 102° C Yield: 0.371g (60.2%)

Reaction of Tricyclohexylantimony(V) Dichloride with KSCN (10)

The mixture of excess KSCN (0.388 g, 4.0 mmol), (cyclo-C_6H_{11})3$SbCl_2$, and THF (25 mL) was agitated at room temperature for 12 hours. To confirm that the reaction was complete, the mixture was refluxed for a further two hours. The resulting KCI was filtered off. An 100% ethyl alcohol and acetone combination were recrystallized to produce a white crystalline solid after the solvent had been completely removed from the filtrate under vacuum (1:1). The substance's chemical formula was (cyclo-$C_6H_{11})_3Sb$ $\{NCS)_2$(10).

M.P.: 148° C Yield:0.386g (79.4%)

Reaction of Tri(α-naphthyl) antimony(V) Dichloride with Sodium Azide (13)

A mixture of tri (α-naphthyl) antimony(V) dichloride (0.573g, 1.0mmol) and excess sodium azide (0.130g, 2.0mmol) in dry benzene (20mL) was refluxed with constant stirring for 10 hours in an environment free of oxygen and moisture. The resulting NaCl was filtered out. Filtrate was entirely solvent-free in vacuo, producing an off-white crystalline solid. The chemical, $(\alpha\text{-}C_6H_{11})_3Sb(N_3)_2$ was recrystallized from a combination of benzene and n-hexane (1:4). (13).

M.P.: 198°C Yield: 0.421g (71.8 %)

BIOLOGICAL ACTIVITY

The antimicrobial and antitumor activity of organoantimony compounds based on hydrocarbon partially fluorosubstituted hydrocarbon ligands or fully substituted pentafluorophenyl derivatives has been extensively studied by Italian, Romanian, Chinese and Lucknow workers Li *et al.* (1998); Singhal *et al.* (1987); Li *et al.* (2002); Alonzo *et al.* (1984); Silvestru *et al.* (1990); Silvestru *et al.* (1995); Kant *et al.* (2008); Keppler *et al.* (1994); Socaciu *et al.* (1994); Hu *et al.* (1995); Tiekink (2002). Apparently, these studies are confined to aryl and pentafluorophenyl group derivatives of central metal atom. Seldom report has appeared with bulky group like cyclohexyl, α-naphthyl, mesityl or tertiary butyl groups. Cyclohexyl and naphthyl derivatives have been found to display remarkable biological activity in case of organotin compounds (8-10). Our preliminary study on the naphthyl derivatives of antimony was found to be quite promising and the compound trinaphthylantimony diisothiocynogen was found to exhibit high antitermite activity. Since, activity is mainly governed by the nature of organic group bound to central metal atom, oxidation state of the central metal and variation in nature of the ligand; we considered it worthwhile to extend the biological activity reported in case of organotin compounds, to the newly synthesised organoantimony compounds by incorporating varied ligands like pseudohalides, carboxylates and halo carboxylates. The compounds were designed in such a fashion to give (chloro) carboxylate derivatives involving monodentate ligands i.e., involving the presence of chloro-group onto the antimony atom (in case of organotin compounds chloro-substituent are known to enhance biological activity). Biologically active pyrazine carboxylic acid was also involved in synthesising newer derivatives. Although tricyclohexyl

and tri(α-naphthyl) pseudohalides have been reported earlier by our research group their biological activity was not examined, therefore these compounds are also included in this study. The biological activity experimentation details Arsenic, antimony, and bismuth are group 15 elements that are mostly utilised in the treatment of acute ulcers, leishmaniasis, and syphilis in therapeutic settings. These metals with organic compounds may have benefits for medicinal therapy. For instance, the coordination of an organic molecule to a metal centre may change the body's usual metabolic pathway and result in slow-release mechanisms for organic molecule delivery (Duffin *et al.*, 2020). The majority of the compounds had their antifungal, antibacterial, and antitumor activities evaluated. Below is a brief summary of biological activity experimental details.

Antifungal Activity. At a concentration of 10 g/mL of the test substance, all of the sterically congested organoantimony(V) -pseudohalides, -halo carboxylates, and -dicarboxylates were examined for antifungal activity against the two strains of Aspergillus flavus and *Aspergillus niger*. The colony diameter of the control and test samples was measured in order to compute the percentage inhibition. A potato dextose agar plate diffusion method Rathore *et al.* (2008) was used to assess the compounds' antifungal activity against *Aspergillus niger* and *Aspergillus flavus* at 10 g/mL of test compound. A petri dish containing 20–25 mL of molten potato–dextrose agar medium was filled with the 1.0 mL of each drug. Petri dishes containing the fungal isolates were cultured separately and stored at 27 °C for 72 hours as the medium solidified. All of the percentage inhibition values were recorded. Various organometallic compounds % inhibition was estimated using the mathematical formula shown below Balouiri Mounyr *et al.* (2016).

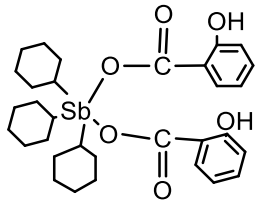
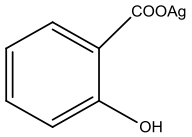
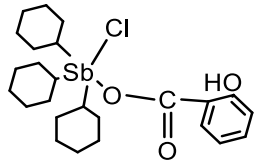
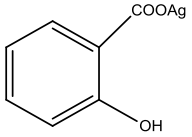
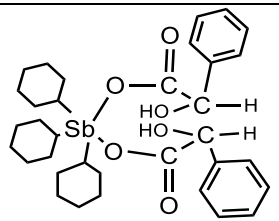
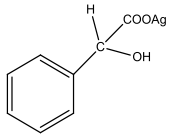
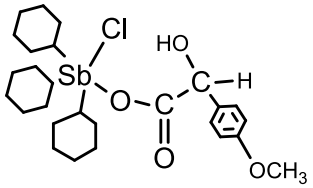
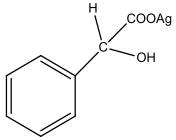
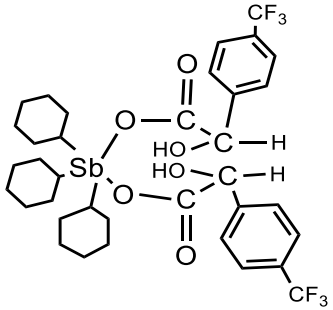
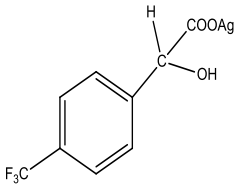
Percentage inhibition= C–T /C × 100
C=Diameter of fungus in control.
T= Diameter of fungus in test compounds.
the results obtained are summarised in Table 6.
Most of the carboxylates were found moderately active against both the strains. In general, it was observed that (chloro) carboxylates show more activity in comparison to dicarboxylates. Among the carboxylates, the maximum activity was shown by compound (2) against A. flavus followed by compound (6) and as per expectations both the compounds are (halo) carboxylates. Surprisingly, mandelate and its derivatives (3,7,8) were found inactive against *A. niger*. The pseudohalide derivatives of sterically hindered organoantimony(V) show very high activity against both the strains.

It was that observed comparison of activity of compounds (10,11,12) on isothiocyanogen substituted compounds show higher activity in comparison to azide substituted compounds.

Antibacterial Activity. The data for antibacterial activity of all sterically hindered organoantimony(V) -pseudohalides and -carboxylates against human pathogenic strains *viz.*, *Staphylococcus aureus* and *Klebsiela pneumoniae* are listed in Table (7).
By using the disc-diffusion method, the antibacterial activity of organometallic compounds was evaluated Varma Rajendra *et al.* (1973). The filter paper (Whatman No. 1) sterile disc was placed on the nutritional agar plate at 37°C for 72 hours after being impregnated with the test chemicals (10 g/mL in ethanol). After 72 hours, the inhibition zone surrounding the dried impregnated discs was measured. Highly active (diameter = 10-15 mm) and marginally active (diameter = 5–10 mm) were the two categories assigned to the activity. Diameters less than 5 mm were regarded to be inactive.
Most of the compounds show moderate to high activity against both the strains. The very high activity was observed in case of pseudohalide derivatives (9-11) against *S. aureus* and K. pneumoniae. Interestingly, the activity of same extent was observed for (chloro) salicylate derivative (2) against S. aureus and this may be attributed to the chlorine atom bonded to antimony. Whereas, dicarboxylates (1,3,5,7,9) were found moderately active against S. aureus. The poorest activity was shown by mandelic acid derivative (3), which is p0orly active against *S. aureus* and was found inactive against K. pneumoniae. It was surprising to note that p-methoxy mandelic acid derivatives (7,8) show no activity against K. pneumoniae. In general, mandelate and its derivatives(3-8) are poorly active against K. pneumoniae and show moderate activity against S. aureus.

Antitumor Activity. The *in vitro* antitumor activity of some representative sterically congested organoantimony compounds was carried out against human breast adenocarcinoma cancer cell line (MCF-7). The data obtained are listed in Table 8.
The most of the compounds were found either inactive or poorly active against MCF-7 cell line. Though the antitumor activity of sterically hindered compounds with tin have shown potential of being used as antitumor agent as has been reported by Vornefeld *et al.* (1992), the results in case of antimony derivatives are disappointing. Surprisingly, tri(a-naphthyl) antimony (1), tricyclohexylantimony(V) dichloride (3) and 2-pyrazine carboxylate (9) were found inactive. The rest of the compounds are poorly active against human breast adenocarcinoma cell line.

Table 1: Preparation and properties of sterically congested organoantimony(v)-carboxylates and –pseudohalides.

Comp. No.	Complex	Organometallic halide (g)	Ligand(g)	Molar Ratio/Solvent (ml)	M.P(°C)	Yield(g) (%)	colour	Recrystallisation solvent
1.		$(Cyclo\text{-}C_6H_{11})_3SbCl_2$ (0.441)	 (0.490)	1:2 THF (20)	146	0.440(68.3)	White	THF/n-Hexane
2.		$(Cyclo\text{-}C_6H_{11})_3SbCl_2$ (0.441)	 (0.245)	1:1 THF (15)	108	0.374(6.9)	White	THF/ n-Hexane
3.		$(Cyclo\text{-}C_6H_{11})_3SbCl_2$ (0.441)	 (0.518)	1:2 THF (20)	112	0.418(62.2)	White	THF/ Petr.ether(60-80°C)
4.		$(Cyclo\text{-}C_6H_{11})_3SbCl_2$ (0.441)	 (0.259)	1:1 THF (20)	88	0.352(64.5)	White	THF/ Petr.ether(60-80°C)
5.		$(Cyclo\text{-}C_6H_{11})_3SbCl_2$ (0.441)	 (0.654)	1:2 THF (20)	150	0.481(59.5)	White	THF/ n-Hexane

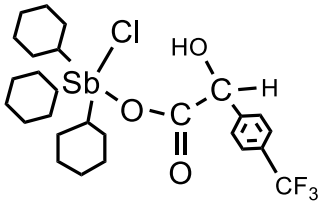
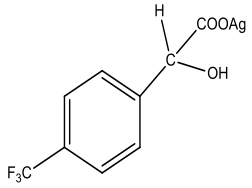
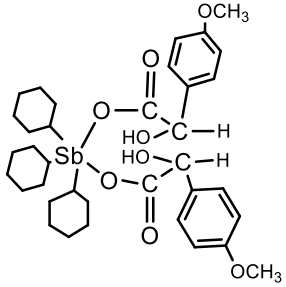
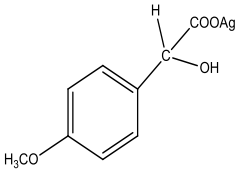
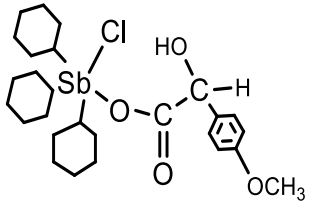
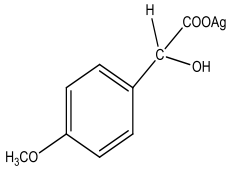
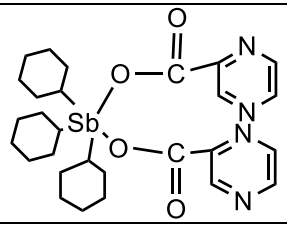
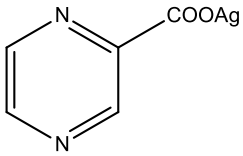
6.		$(Cyclo\text{-}C_6H_{11})_3SbCl_2$ (0.441)	 (0.327)	1:1 THF (20)	126	0.382(61.2)	White	THF/ n-Hexane
7.		$(Cyclo\text{-}C_6H_{11})_3SbCl_2$ (0.441)	 (0.578)	1:2 THF (20)	134	0.420(57.4)	White	THF/ n-Hexane
8.		$(Cyclo\text{-}C_6H_{11})_3SbCl_2$ (0.441)	 (0.289)	1:2 THF (20)	112	0.340(58.0)	White	THF/ n-Hexane
9.		$(Cyclo\text{-}C_6H_{11})_3SbCl_2$ (0.441)	 (0.462)	1:2 Benzene (20)	102	0.371(60.2)	White	THF/ Petr.ether(60-80 °C)
10.	$(Cyclo\text{-}C_6H_{11})_3Sb\ (SCN)_2$	$(Cyclo\text{-}C_6H_{11})_3SbCl_2$ (0.883)	KSCN (0.388)	1:2 THF (25)	148	0.386(79.4)	White	Acetone/Ethanol
11.	$(Cyclo\text{-}C_6H_{11})_3Sb(N_3)_2$	$(Cyclo\text{-}C_6H_{11})_3SbCl_2$ (0.441)	NaN_3 (0.2excess)	1:2 Benzene (20)	130	0.334(73.4)	White	Acetone/Benzene
12.	$(\alpha\text{-}C_{10}H_7)_3Sb\ (SCN)_2$	$(\alpha\text{-}C_6H_{11})_3SbCl_2$ (0.573)	KSCN (0.194)	1:2Benzene (20) /Acetone (20)	232	0.459(74.2)	Off White	Acetone/n-Hexane
13.	$(\alpha\text{-}C_{10}H_7)_3Sb(N_3)_2$	$(\alpha\text{-}C_6H_{11})_3SbCl_2$ (0.573)	NaN_3 (0.2excess)	1:2 Benzene (20)	224	0.421(71.8)	Off White	Benzene/n-Hexane

Table 2: Elemental Analysis of sterric Congested Organoantimony (V)-Carboxylates and –Pseudohalides.

Comp. (No.)	Empirical Formula	Molecular Weight	Found (Calcd.) %		
			C	H	N
1.	$C_{32}H_{43}O_6Sb$	645.10	59.44 (59.58)	6.69 (6.66)	-
2.	$C_{25}H_{38}O_2ClSb$	543.47	55.21 (55.25)	7.06 (6.99)	-
3.	$C_{34}H_{47}O_6Sb$	673.09	60.66 (59.93)	6.98 (7.08)	-
4.	$C_{26}H_{40}O_3ClSb$	557.46	56.01 (56.83)	7.17 (7.18)	-
5.	$C_{36}H_{45}O_6F_6Sb$	809.14	53.40 (53.43)	5.61 (5.56)	-
6.	$C_{27}H_{39}O_3F_3ClSb$	625.50	51.88 (51.84)	6.19 (6.23)	-
7.	$C_{36}H_{51}O_8Sb$	733.14	59.07(58.97)	7.02 (6.95)	-
8.	$C_{27}H_{42}O_4ClSb$	587.50	55.10 (55.19)	7.18 (7.14)	-
9.	$C_{28}H_{39}O_4N_4Sb$	617.05	54.53 (54.50)	6.30 (6.32)	9.12 (9.07)
10.	$C_{20}H_{33}N_2S_2Sb$	487.09	49.20 (49.31)	6.83 (6.77)	5.78 (5.74)
11.	$C_{18}H_{33}N_6Sb$	454.94	47.50 (47.52)	7.21 (7.25)	18.50 (18.46)
12.	$C_{32}H_{21}N_2S_2Sb$	619.22	62.12 (62.07)	3.43 (3.39)	4.50 (4.52)
13.	$C_{30}H_{21}N_6Sb$	587.08	61.41 (61.37)	3.62 (3.57)	14.24 (14.30)

Table 3: Characteristic Infrared Absorption Frequencies of the Compounds in cm^{-1}.

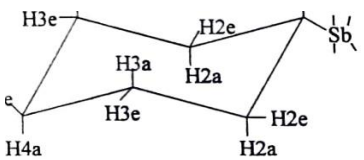
Comp. (No.)	ν (Sb-C) (y-mode)	ν (OCO)			N-N-N			N-C-S			ν (O-H)
		ν_{asym}	ν_{sym}	Δν	$\nu_{asym}(N_3)$	$\nu_{sym}(N_3)$	$\delta(N_3)$	ν(C-N)	ν(C-S)	δ(N-C-S)	
1.	405 (m)	1633 (vs)	1359 (vs)	274	-	-	-	-	-	-	3233 (w)
2.	407 (m)	1633 (vs)	1351 (vs)	282	-	-	-	-	-	-	3234 (w)
3.	405(w)	1638 (vs)	1300 (s)	338	-	-	-	-	-	-	3456 (m)
4.	423 (w)	1638 (vs)	1310 (m)	328	-	-	-	-	-	-	3458 (s)
5.	426 (w)	1643 (s)	(o)	-	-	-	-	-	-	-	3454 (s)
6.	427 (w)	1638 (vs)	1311 (vs)	327	-	-	-	-	-	-	3460 (s)
7.	425 (w)	1642 (vs)	1320 (vs)	322	-	-	-	-	-	-	3457 (s)
8.	422 (w)	1639 (vs)	1316 (m)	323	-	-	-	-	-	-	3464 (s)
9.	424 (s)	1667 (vs)	1384 (s)	283	-	-	-	-	-	-	-
10.	413 (w)	-	-	-	-	-	-	2014 (vs)	835 (w)	463 (w)	-
11.	426 (w)	-	-	-	2069 (vs)	1323 (s)	665 (m)	-	-	-	
12.	411 (m)	-	-	-	-	-	-	2025 (vs)	793 (m)	485 (w)	-
13.	423 (m)	-	-	-	2074 (vs)	1272 (m)	667 (w)	-	-	-	-

W= weak; m=medium; s=strong; vs=very strong; o=overlapped

Table 4: ^{1}H NMR Data of Sterically congested Organoantimony(v) Carboxylates in δ (ppm).

Compd. No.	Cyclohexyl						Ligand				
	H1	H2e	H2a/H3e	H3a	H4e	H4a	Hα	OCH3	H2	H3	H4
2.	3.80-3.86 (m)	2.91-2.97 (m)	1.73-1.78 (m)	1.52-1.56 (m)	1.58-1.62 (m)	1.19-1.20 (m)	-	-	6.67-7.93 (m)		
3.	3.75-3.81 (m)	2.80-2.88 (m)	1.71-1.75 (m)	1.52-1.55 (m)	1.57-1.61 (m)	1.17-1.24 (m)	5.10 (s)	-	7.17-7.29 (m)	6.58-6.69 (m)	
5.	3.77-3.80 (m)	2.83-2.89 (m)	1.71-1.77 (m)	1.55-1.59(m)	1.60-1.65 (m)	1.17-1.22 (m)	5.13 (s)	-	6.73-7.40 (m)		-
7.	3.78-3.69 (m)	2.83-2.91 (m)	1.73-1.77 (m)	1.54-1.59 (m)	1.62-1.65 (m)	1.17-1.18 (m)	4.96 (s)	3.78 (s)	7.26-7.34 (m)	6.81-6.88 (m)	-
8.	3.63-3.69 (m)	2.71-2.82 (m)	1.70-1.76 (m)	1.55-1.62 (m)	1.64-1.68 (m)	1.13-1.20 (m)	5.02 (s)	3.81 (s)	7.28-7.41 (m)	6.80-7.01 (m)	-
9.	3.82-3.86 (m)	2.90-2.96 (m)	1.74-1.78 (m)	1.52-1.58 (m)	1.61-1.64 (m)	1.15-1.20 (m)	-	-	9.04 (s)	8.79 (d)	8.87 (d)

Where, m=multiplet; d= doublet; s=singlet;



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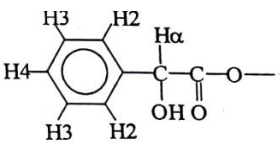

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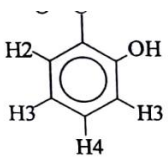


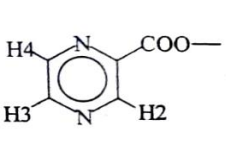
and


Table 5: ^{13}C NMR Data of Sterically Organoantimony(V) Carboxylates in δ (ppm).

Compd. No.	Cyclohexyl				Ligand						
	C_1	C_2	C_3	C_4	>C=O	CF_3/OCH_3	Cα	C1'	C2'	C3'	C4'
1.	56.72	28.08	26.14	25.57	176.03	-	-	133.50	133.59 163.90 (OH)	103.40 104.47	133.21
3.	55.43	30.01	26.14	26.00	176.88	-	70.75	158.92	131.57	112.38	125.02
5.	55.09	29.61	28.34	26.02	176.91	127.56	70.11	159.04	125.13		126.37
7.	55.31	29.63	28.23	25.90	177.12	50.56	73.35	159.29	132.75	113.51	127.49
8.	55.24	29.45	28.20	25.87	177.34	50.54	73.86	259.31	132.77	113.60	127.52
9.	55.58	28.05	25.03	25.55	167.14	-	-	162.46	144.79	146.71	142.88

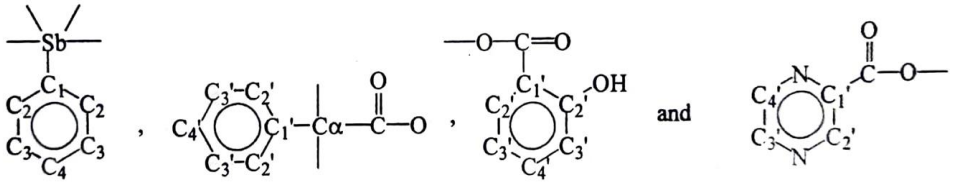


Table 6: Antifungal Activity of Sterically Congested Organoantimony(V) compounds.

Sr. No.	Compound (N0.)	Aspergillus flavus		Aspergilus niger	
		Colony Diameter (nm)	Inhibition (%)	Colony Diameter (nm)	Inhibition (%)
1.	(Cyclo-$C_6H_{11})_3Sb$ (Sal.)$_2$ (1)	1.4	30	1.4	30
2.	(Cyclo-$C_6H_{11})_3Sb$ (Cl)(Sal.) (2)	1.0	50	1.7	35
3.	(Cyclo-$C_6H_{11})_3Sb$ (Mand.)$_2$ (3)	1.7	15	No Active	-
4.	(Cyclo-$C_6H_{11})_3Sb$ (p-CF_3 Mand.)$_2$ (5)	1.3	35	1.3	35
5.	(Cyclo-$C_6H_{11})_3Sb$ (Cl) (p-CF_3 Mand.) (6)	1.2	40	1.3	35
6.	(Cyclo-$C_6H_{11})_3Sb$ (p-MeO Mand.)$_2$ (7)	1.3	35	No Active	-
7.	(Cyclo-$C_6H_{11})_3Sb$ (Cl) (p-CF_3 Mand.) (8)	1.3	35	No Active	-
8.	(Cyclo-$C_6H_{11})_3Sb$ (2-Pyrazine Carbox.)$_2$ (9)	1.4	30	1.4	30
9.	(Cyclo-$C_6H_{11})_3Sb(N_3)_2$ (11)	1.0	50	1.0	50
10.	(Cyclo-$C_6H_{11})_3Sb$ (NCS)$_2$ (10)	0.7	65	0.6	70
11.	(α-$C_6H_{11})_3Sb$ (NCS)$_2$ (12)	0.6	70	0.6	70
12.	(α-$C_6H_{11})_3Sb(N_3)_2$ (13)	0.7	65	0.7	65
13.	**Control**	**2.0**	**-**	**2.0**	**-**

Conc. =10 µg/ml of test compound, control =10 µg/ml of streptomycin,
Incubation period = 72 h, Method = disc diffusion method.

Table 7: Antibacterial Activity of Sterically Congested Organoantimony(v) Compounds.

Sr. No.	Compound (No.)	Staphylococcus aureus	Klebsiella pneumoniae
1.	$(Cyclo\text{-}C_6H_{11})_3Sb\ (Sal.)_2$ (1)	++	++
2.	$(Cyclo\text{-}C_6H_{11})_3Sb\ (Cl)(Sal.)$ (2)	+++	++
3.	$(Cyclo\text{-}C_6H_{11})_3Sb\ (Mand.)_2$ (3)	+	–
4.	$(Cyclo\text{-}C_6H_{11})_3Sb\ (p\text{-}CF_3\ Mand.)_2$ (5)	++	+
5.	$(Cyclo\text{-}C_6H_{11})_3Sb\ (Cl)\ (p\text{-}CF_3\ Mand.)$ (6)	++	+
6.	$(Cyclo\text{-}C_6H_{11})_3Sb\ (p\text{-}MeO\ Mand.)_2$ (7)	++	–
7.	$(Cyclo\text{-}C_6H_{11})_3Sb\ (Cl)\ (p\text{-}MeO\ Mand.)$ (8)	++	–
8.	$(Cyclo\text{-}C_6H_{11})_3Sb\ (2\text{-}Pyrazine\ Carbox.)_2$ (9)	++	++
9.	$(Cyclo\text{-}C_6H_{11})_3Sb(N_3)_2$ (11)	+++	+++
10.	$(Cyclo\text{-}C_6H_{11})_3Sb\ (NCS)_2$ (10)	+++	+++
11.	$(\alpha\text{-}C_6H_{11})_3Sb\ (NCS)_2$ (12)	+++	+++
12.	$(\alpha\text{-}C_6H_{11})_3Sb(N_3)_2$ (13)	+++	+++

Conc. =10 µg/ml of test compound, control = 10 µg/ml of ampicillin,
Incubation period = 72 h, (+) =diameter up to 5mm, (++) =diameter 5-10mm,
(+++) = diameter 10-15 mm.

Table 8: *In vitro* Antitumor Activity of Sterically Congested Organoantimony (III) and -(V) Compounds against MCF-7 Cell Line.

Sr. No.	Compound (No.)	Cell Count × 10^4	Activity
1.	$(\alpha\text{-}C_6H_{11})_3Sb$	10.73 ± 1.13	–
2.	$(\alpha\text{-}C_6H_{11})_3SbCl_2$	10.02± 1.21	+
3.	$(\alpha\text{-}C_6H_{11})_3Sb\ (NCS)_2$	10.23 ± 1.02	–
4.	$(\alpha\text{-}C_6H_{11})_3Sb\ (NCS)_2$ (12)	10.06 ± 1.04	+
5.	$(Cyclo\text{-}C_6H_{11})_3Sb\ (NCS)_2$ (10)	10.12 ± 1.06	+
6.	$(Cyclo\text{-}C_6H_{11})_3Sb(N_3)_2$ (11)	10.12 ± 1.06	+
7.	$(\alpha\text{-}C_6H_{11})_3Sb(N_3)_2$ (13)	10.04 ± 1.08	+
8.	$(Cyclo\text{-}C_6H_{11})_3Sb\ (Mand.)_2$ (3)	10.18 ± 0.96	+
9.	$(Cyclo\text{-}C_6H_{11})_3Sb\ (p\text{-}CF_3\ Mand.)_2$ (5)	10.12 ± 1.13	+
10.	$(Cyclo\text{-}C_6H_{11})_3Sb\ (2\text{-}Pyrazine\ Carbox.)_2$ (9)	11.27 ± 2.13	–

Positive Control – 40.28 ± 3.29; Negative Control – 10.22 ± 1.05

CONCLUSIONS

In conclusion, organoantimony(v) compounds are highly versatile and attractive reagents for the advancement of synthetic organic chemistry. Organoantimony (v) compounds are easily synthesised from low-toxicity antimony salt. These compounds are mostly trivalent and pentavalent, with antimony in the +3 and +5 oxidation states. Organoantimony compounds have a wide range of applications in organic chemistry, as well as organometallic, medicinal and polymer chemistry. Organoantimony compounds have received less attention than other lighter pnictogens. Experimental studies on the antifungal, antibacterial, and antitumor activities of organoantimony (v) compounds revealed that they are better biocides than hydrocarbon-based ligands, which may be due to the presence of Florine atoms, which causes electronical and chemical changes in the molecule, it is reasonable to believe that the future of organoantimony chemistry should lead to interesting and valuable discovery.

FUTURE SCOPE

To produce organo metallic based derivatives which showed an effective role in the biological activity. The obtained results are believed to have fulfilled the objective and scope of the study. As a result, there are still numerous chances to find new compounds that are strong biological activity.

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