SYNTHESIS AND CHARACTERISATION OF SOME ORGANOANTIMONY COMPOUNDS

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Dedicated to

those who like me

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CERTIFICATE

This is to certify that the thesis "SYNTHESIS AND CHARACTERIZATION OF SOME ORGANOANTIMONY COMPOUNDS" being submitted by Mr.Pankaj Sharma to the Indian Institute of Technology, Delhi for the award of the degree of Doctor of Philosphy in Chemistry, is a record of a bonafide research work carried out by him. Mr.Sharma has worked under my guidance and supervision and has fulfilled the requirements for the submission of this thesis which, to my knowledge, has reached the requisite standard.

The results contained in this thesis have not been submitted, in part or in full, to any other university or institute for the award of any degree or diploma.

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ABSTRACT

The thesis contains synthesis and characterisation of monohalodiorganoantimony(V) and triorganoantimony(V) schiff base complexes. All these triorganoantimony(V) and diorganoantimony(V) schiff base complexes were characterised by elemental analysis, vapour pressure osmometry, IR, far IR, ^{1}H and ^{13}C NMR spectroscopy. The compounds have been formulated as R_2SbXL (where R = Me, Ph or Tolyl and LH_2 = $SaltmH_2$ or $SalpdH_2$) and R_3SbL (where R = Me or Ph and LH_2 = $SaltmH_2$, $HacenH_2$, $HactmH_2$, $GbapH_2$ or $AcacenH_2$). Spectroscopic studies indicate the presence of heapta-coordinated antimony in these schiff base complexes, having pentagonal bipyramidal structure.

Present work reports the systhesis and characterisation of a few novel noncyclic catenatristibines containing two Sb-Sb bonds. In these tristibines two antimony atoms have different oxidation states and coordination number than the third antimony atom. The general formula of these compounds are $R_3'Sb(SbR_2)_2$ (where R' = Me, Ph or Tolyl, R = Ph or Tolyl). These compounds are moisture and oxygen sensitive. A trigonal bipyramidal structure has been proposed for these tristibines on the basis of IR, ^{1}H NMR, ^{13}C and ^{121}Sb NMR spectroscopy. On the basis of ^{1}H NMR and ^{13}C NMR data it is clear that in these compounds the two $-SbR_2$ groups are in equatorial positions which may be due to the bulkiness of $-SbR_2$ group whereas one of three R' groups occupies an

equatorial position and the other two occupy the axial positions. Thermogravimetric analysis, in nitrogen atmosphere shows that these tristibines decompose to antimony.

During the present work tertiary stibines containing tetrachloro-4-pyridyl groups namely, tris(tetrachloro-4-pyridyl)stibine, phenylbis(tetrachloro-4-pyridyl)stibine or tolylbis(tetrachloro-4-pyridyl)stibine were synthesised. These stibines were then brominated to get their dibromides. These stibines and their dibromo derivatives were characterised by elemental analysis, vapor pressure osmometry, IR, far IR, ^{1}H and ^{13}C NMR spectroscopy. Thermogravimetric analysis in nitrogen atmosphere shows that these compounds decompose to antimony which corresponds to the amount of antimony present in these compounds. In static air Sb_2O_3 is the end product of decomposition.

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