

Modern Characterization of Some Novel Complexes By Pyrazine as Ligand

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ABSTRACT

Pyrazine is less basic than pyridine, pyridazine and pyrimidine. Derivatives such as Phenazine are well known for their antitumor, antibiotic and diuretic activities. Tetramethyl pyrazine, also known as ligustrazine, is reported to scavenge superoxide anion and decrease nitric oxide production in human polymorphonuclear leukocytes and is a component of some herbs in traditional Chinese medicine. Alkylpyrazines are chemical compounds based on pyrazine with different substitution patterns. Some alkylpyrazines are naturally occurring highly aromatic substances which often have a very low odour threshold and contribute to the taste and aroma of various foods including coffee and wines. Alkylpyrazines are also formed during the cooking of some foods via Maillard reactions. 2,3-Dimethylpyrazine is a component of the aroma of roasted sesame seeds.

Dimethylpyrazine is used as flavour additive and odorant in foods such as cereals and products such as cigarettes. It occurs naturally in asparagus, black or green tea, crisp bread, malt, raw shrimp, soya, Swiss cheeses, and wheat bread. Methoxy pyrazines are a class of chemical compounds that produce odours. The odours tend to be undesirable as in the case of certain wines, or as in the case of the Asian lady beetle which produces isopropyl methoxy pyrazine. They have also been identified as additive in cigarettes.

Keywords :- pyridazine and pyrimidine, black or green tea, crisp bread, malt, raw shrimp, soya, Swiss cheese

I. INTRODUCTION

Like the related pyrazinamide, pyrazine-2-thio carboxamide has Mycobacteriostatic and mycobacteriocidal activities in vitro suggesting that it might be an effective antituberculosis drug. The

emergence of drugresistant pathogenic strains in recent years, e.g ., streptococcus pneumoniae, streptococcus aureus, Entrococcus faecium, Pseudomonas aeruginosa, stenotrophomonas maltophilia, Salmonella typhi, etc. have been of major concern. Among other infectious diseases,

tuberculosis, caused by *Mycobacterium tuberculosis*, seems to be the most invasive, and multidrug resistance (MDR) phenomenon makes it the world's number one killer especially for immune suppressed AIDS patients. Because of this, there is a great need for antibacterial and antitubercular drugs with improved properties such as enhanced activities against MDR strains and reduced toxicity. Pyrazinamide is one of the most effective antitubercular drugs. Various pyrazine derivatives and pyrazinamide analogs also exhibit high antibacterial activity, e.g. Pyrazinoic acid esters, pyrazine thiocarboxamide and N-hydroxymethylpyrazine thiocarboxamide and ring substituted pyrazinyl chalcones. 4-Mono and 4, 4-disubstituted 1-pyrazinoyl thiosemicarbazides are reported to have high tuberculostatic activity. Decorating thieno-[3,4-b] pyrazine with bulky tetraphenyl ethylene groups generated efficient solid-state fluorophores. Organic lightemitting diodes were fabricated using these derivatives as non-doped emitting layers. Saturated reelectroluminescence have been obtained with best performance for thieno [3,4-b] pyrazine derivatives reported so far. Pyrazine substituted 1,3,4-thiadiazole derivatives have been reported to be anticonvulsant. Pyrrolo [2,3-b] pyrazine derivatives act as submicromolar affinity activators of wild type cystic fibrosistrans membrane conductance regulator chloride channels.

The transition metal complexes have great diversity in their applications. They have anticancer, anti-inflammatory, anti-diabetic, antibacterial and anti-fungal activities. The development of transition metal complexes as drug is not an easy task, a considerable effort is required to get a compound of choice. In spite of all these limitations, coordination compounds are the most widely used chemotherapeutics. The number of platinum complexes having antitumor activity is rapidly increasing because of attempts to find complexes with greater therapeutic potency and lesser toxicity than existing drugs. Consequently attention

has shifted to other platinum group metals, like palladium, ruthenium, osmium, iridium and rhodium. Special attention has been focussed on ruthenium compounds because they exhibit cytotoxicity against cancer cells and no cross resistance with cis-platin. Ruthenium complexes demonstrate similar ligand exchange kinetics to those of Pt (II) antitumor drugs already used in clinical treatment while displaying only low toxicity. This is partially due to the ability of the ruthenium complexes to mimic the binding of iron to the molecules of biological significance exploiting the mechanisms the organisms have evolved for iron transport.

It is known that pyrazine ring is a part of polycyclic derivative and plays important industrial and biological roles. The pharmacological activities of pyrazine derivatives vary and include substances with multidimensional actions. A low toxicity of these groups of compounds allow us to use them as a pharmacophore in designing new compounds to be used as drugs.

The discovery of natural pyrazine derivatives that showed the pharmacological effect, initiated the search for novel and more effective synthetic compounds exhibiting biological activities. There are a number of substances having antitubercutotic, antibacterial, antifungal and cytotoxic effects in the group of synthetic pyrazine derivatives. Furthermore, the compounds belonging to this group display antioxidant antiproliferative and antitumor activities.

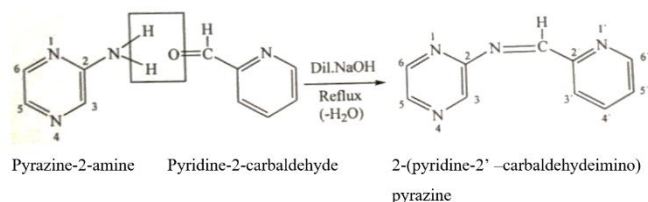
The ability of different pyrazinecarboxylic acids (2-pyrazine carboxylic acid, pyrazine-2,3-dicarboxylic acid etc.) to act as chelating and bridging ligands for different metal ions is well known and has been extensively studied during last few decades. TG, DTG, DTA, IR and electronic spectra have been applied to investigate thermal and spectral properties of these complexes. The chemical composition of the complexes, the solid intermediates and resultant

products of thermolysis have been identified by means of elemental analysis and complexometric titration. Schemes of destruction of these complexes are suggested. Heating of the compounds first resulted in the release of water molecules. The final products of thermal decomposition was CuO in all cases. IR data suggested a unidentate coordination of carboxylates to Cu(II) in all the complexes.

II. EXPERIMENT

Preparation of the Ligand

The Schiff base ligand 2-(pyridine-2'-carbaldehydeimino) pyrazine was prepared by the condensation of pyrazin-2-amine and pyridine-2-carbaldehyde in the presence of dil. NaOH.



Procedure:

0.01 mol of pyrazin-2-amine was dissolved in 20 ml of hot ethanol and the solution was filtered. 0.01 mol of pyridine-2-carbaldehyde was also dissolved in 20 ml of hot ethanol and the solution was filtered. The two hot solutions were mixed in 250 ml round-bottom flask and 10 ml of 10% NaOH solution was added to it. The content in the flask was refluxed for about 4 hour on a water-bath using water condenser. The solution was cooled to room temperature and then left overnight when light yellow crystals of 2-(pyridine-2'-carbaldehydeimino) pyrazine were separated. The precipitate was filtered under suction, washed with cold water and then with ethanol. It was then dried in an electric oven at about 80°C. The m.p. of the ligand was recorded to be 112°C.

III. PREPARATION OF COORDINATION COMPOUNDS

Preparation of complexes of Pd (II) with 2-(pyridine-2'-carbaldehydeimino) pyrazine:

Preparation of complexes of Pd(II) with the ligand 2-(pyridine-2'-carbaldehydeimino) pyrazine (L) was carried out in the presence of various bases like water, ammonia, pyridine and different picolines by a general procedure described here under: 0.001 mol of the ligand 2-(pyridine-2'-carbaldehydeimino) pyrazine (L) dissolved in the minimum volume of ethanol was added to ethanolic solution of 0.01mol of palladium (II) chloride in presence of H₂O/NH₃/C₆H₅N/C₆H₇N with regular shaking and stirring. The resulting solution was then refluxed for an hour on a water bath using water condenser. The colour of the solution changed gradually and a coloured precipitate was separated out on allowing the solution to stand for more than two days at room temperature. The product was filtered, washed with a small amount of ethanol and then dried over KOH in a desiccator. The melting points of coordination compounds were then recorded. The coordination compounds of Pd(II) ions with the ligand 2(pyridine-2'-carbaldehydeimino) pyrazine were prepared separately, keeping the metal ligand ratio as 1:1 in each case in presence of bases like water, ammonia, pyridine and different picolines.

Preparation of complexes of Pt(II) with the ligand 2-(pyridine-2'-carbaldehydeimino) pyrazine (L)

The preparation of coordination compounds Pt(II) with the ligand 2(pyridine-2'-carbaldehydeimino) pyrazine(L) was carried out in presence of various bases like water, ammonia, pyridine and different picolines by a general procedure described hereunder: 0.001 mole of platinum (II) chloride dissolved in ethanolic solution was mixed with ethanolic solution of 0.001 mole of 2-(pyridine-2'-carbaldehydeimino) pyrazine(L) with regular shaking and stirring. The resulting solution was then refluxed for an hour on a water bath using water condenser. The colour of the solution changed gradually and coloured crystals were separated out on allowing the solution to stand for two days. The product was filtered, washed with a small amount of ethanol and then dried over KOH in a desiccator. The melting points of coordination

compounds were then recorded. The coordination compounds of Pt(II) ions with the ligand 2(pyridine-2'-carbaldehydeimino) pyrazine(L) were prepared separately in the presence of bases like water, ammonia, pyridine and different picolines, keeping the metal ligand molar ratio as 1 : 1 in each case.

Carbon, hydrogen and nitrogen contents in ligand and corresponding complexes were quantitatively estimated in micro analytical section of Indian Institute of Technology, Patna. The estimation of metals in complexes was carried out by standard methods (133). The complexes were first decomposed with a view to bring the metals in their proper ionic form in solution and then they were quantitatively analysed.

Electrical conductivity of solutions of complexes were measured by conductivity meter bridge manufactured by Wiss-Techen Werch Stathen type LBR at room temperature in dimethyl formamide. The cell constant was determined at (room temperature, 30°C) using N/10 KCl solution. Pure DMF and conductivity water were used as solvent.

The centre of the maximum field between the pole pieces were determined by a topographical survey with the tube containing a column of paramagnetic substance like ferrous ammonium sulphate. The tube containing the specimen for measurement was always suspended between the poles in such a way that its lower end coincides with the centre. The bottom of the suspension tube was, therefore, always in the region of maximum field even under oscillation during weighing. The field was found to be practically negligible at 11.6 cm above this point. The specimen tube was, therefore always filled upto height of about 11.6 cm.

Filling up of the specimen tube requires some skill. Generally the substance was well powdered in a agate mortar. A small amount of the substance then introduced into the tube and rammed with a properly fitting glass rod. In this way the required length was filled in uniformly.

Determination of Maximum Field Strength:

The field was determined by using a number of standard substances like copper sulphate, ferrous ammonium sulphate. Accurate values of their mass susceptibilities are known.

RESULTS AND DISCUSSION

The analytical, conductometric, magnetic and spectroscopic investigations of the coordination compounds of Pd(II) and Pt(II) ions with the ligand 2-(pyridine-2'-carbaldehydeimino) pyrazine (L) in the presence of bases like water, ammonia, pyridine and picolines. The results of these investigations are discussed herein and suitable conclusions have been drawn regarding the structure of coordination compounds and the nature of bonding of the ligand (s) to the metal ions in these coordination compounds.

IV. DETERMINATION OF STRUCTURE OF COORDINATION COMPOUNDS

From microanalytical data and determination of molar masses by cryoscopic method, the molecular formula of coordination compounds were determined. The microanalytical data and observed molar masses of coordination compounds are in good agreement with the proposed molecular formula of respective coordination compounds. The prepared coordination compounds are insoluble in common organic solvents viz ., carbon tetrachloride, chloroform, benzene, toluene, ether, methanol, ethanol, dioxane, THF (tetrahydrofuran), acetone and pyridine but they are soluble in DMSO (dimethyl sulphoxide) and DMF (dimethyl formamide). The molar conductances of prepared coordination compounds were measured in 10⁻³ M DMF solution at room temperature. The molar conductance data of coordination compounds.

The band at 1680 cm⁻¹ in the spectrum of ligand due to $\nu_{C=N}$ (azomethine) mode of vibration is redshifted by 10-15 cm⁻¹ in all the complexes indicating the coordination of the ligand to Pd(II) and Pt(II) through nitrogen atom of the azomethine group. There is a broad band in the infrared spectra of aqua complexes of Pd(II) and Pt(II) in the range 3420-3650 cm⁻¹,

which is absent in the spectrum of the ligand (L), is assigned to voH mode of vibrations due to coordinated water molecule. This is further supported by the appearance of a new band at 830 cm⁻¹ which is characteristic of the wagging mode of vibration of the coordinated water molecule. In ammine complexes, two new infrared spectral bands appear at 3380-3420 cm⁻¹ and 3440-3460 cm⁻¹ which are reasonably assigned to VNH (sym.) and VNH(antisym.) modes of vibrations respectively.

V. CONCLUSION

The importance and various applications of pyrazine and its derivatives in food and pharmaceutical industries. The review of the previous works related to the present work. The review of the previous works is sufficient enough to justify the selection of the work for study. The experimental works and physico-chemical methodologies applied for the research work. The various experimental data obtained have been interpreted and valuable conclusions have been drawn.

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