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Prof. T. J.SAWANT
D.E.E,B.E.(Elec.), MISTE
Founder-Secretary

Dr V.V. POTNIS
M.Pharm., Ph.D.
Principal

Date: 16/08/2019

To,
The Coordinator,
NAAC, Bengaluru.

Subject: Proof of teaching learning process of the institution nurtures creativity, analytical skills and innovation among students.

Reference: 2.3.5 The teaching learning process of the institution nurtures creativity, analytical skills and innovation among students.

Dear Sir/Madam,

2.3.5 The teaching learning process of the institution nurtures creativity, analytical skills and innovation among students.

Principal
Jayawantrao Sawant
College of Pharmacy & Research
Hadapsar, Pune - 411 028.

JSPM's

Jayawantrao Sawant College of Pharmacy & Research

Hadapsar, Pune-411028.

Department: Pharmaceutical Chemistry

The Catalyst Club

Activity: Development of reaction database

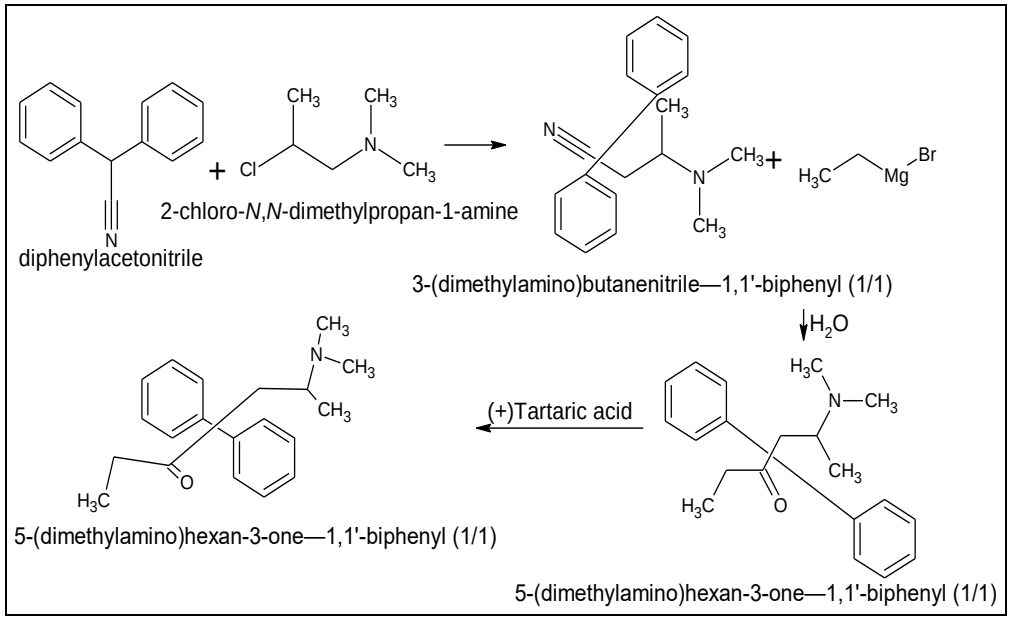
Introduction:

The development of a reaction database with the objective to collect data for different reactions involved in pharmaceutical processes with a search engine to retrieve necessary data in investigations of reaction-separation schemes, such as the role of organic solvents in reaction performance improvement. The focus of this reaction database is to provide a data rich environment with process information available to assist during the early stage synthesis of pharmaceutical products. The database is structured in terms of reaction classification of reaction types; compounds participating in the reaction; use of organic solvents and their function; information for single step and multistep reactions; target products; reaction conditions and reaction data. The application of the database is illustrated through the synthesis of different pharmaceutical intermediates & compounds.

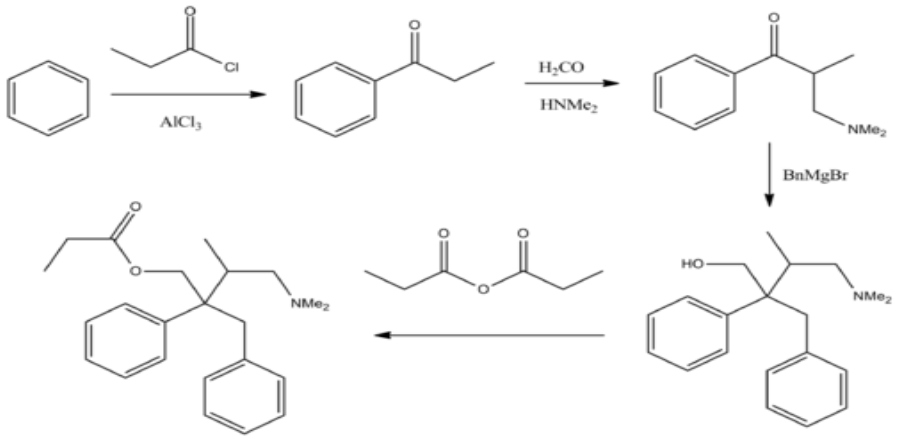
Objectives:

1. To identify reactions that are used to produce different types of pharmaceutical intermediates & products.
2. To facilitate the choice of the reaction conditions.
3. To evaluate the reaction pathway in terms of yield, cost and sustainability metrics.

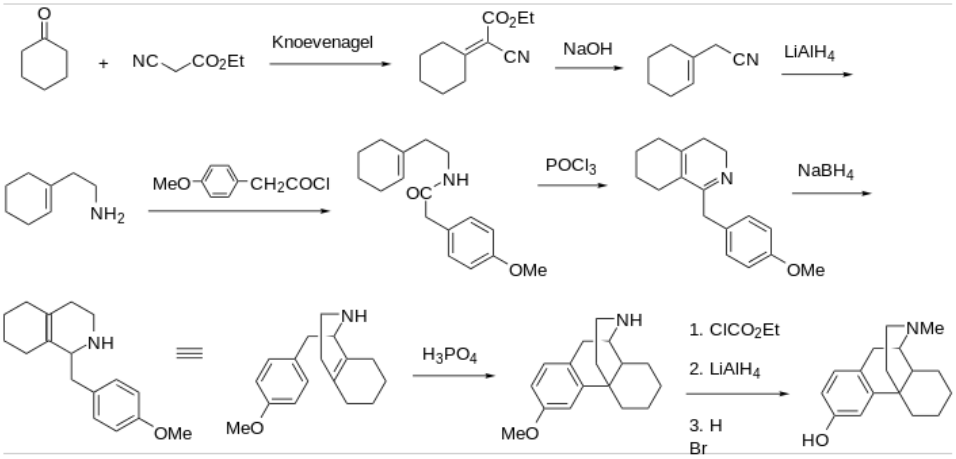
Synthesis of Methadone: (Narcotic Analgesic)



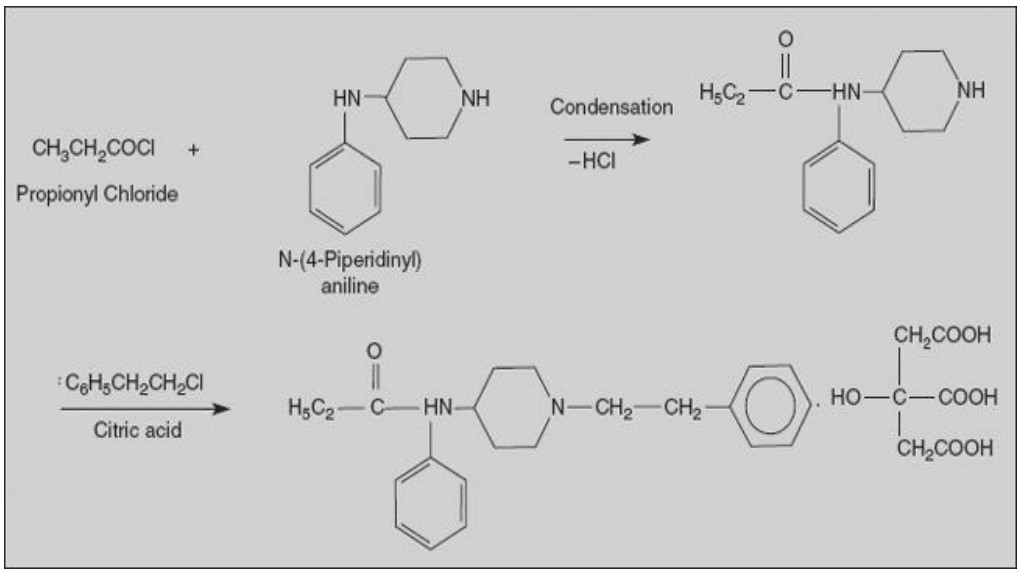
Synthesis of Propoxyphene: (Narcotic Analgesic)



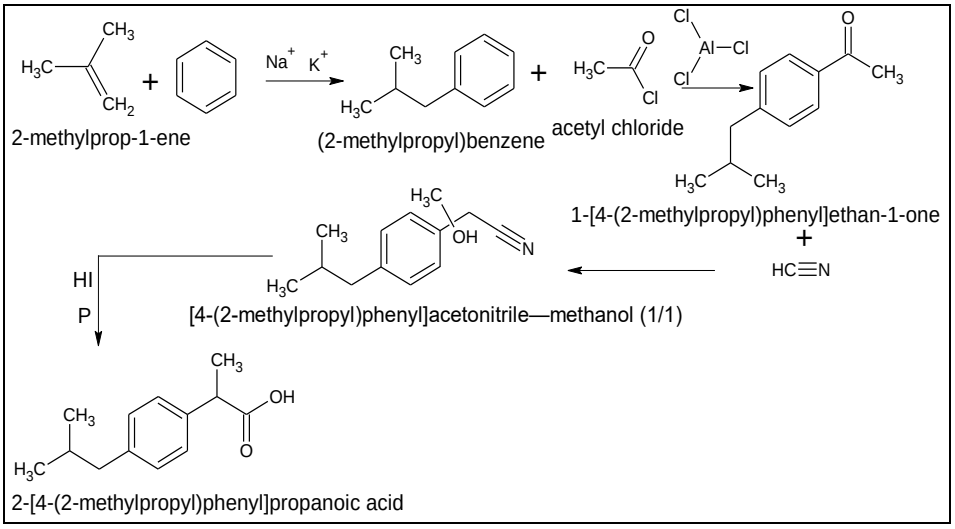
Synthesis of Dextromethorphan: (Narcotic Analgesic)



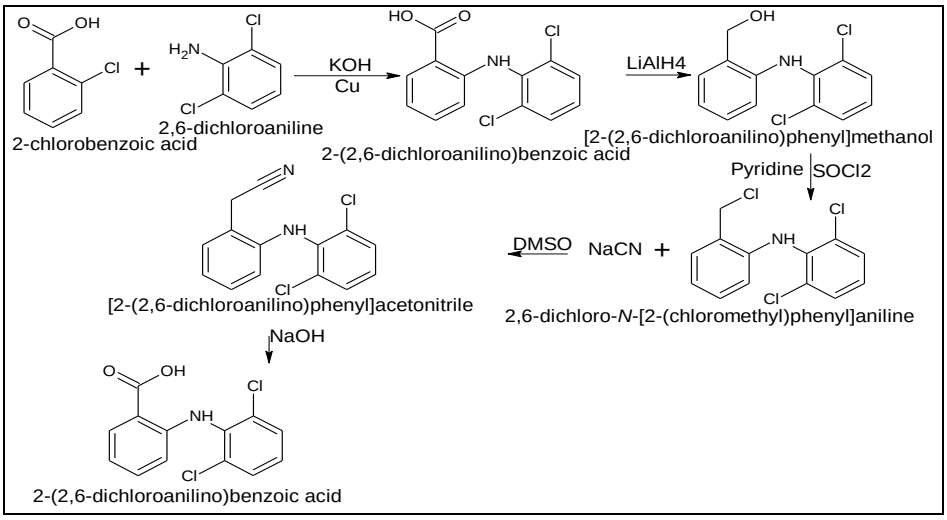
Synthesis of Fentanyl Citrate: (Narcotic Antagonist)



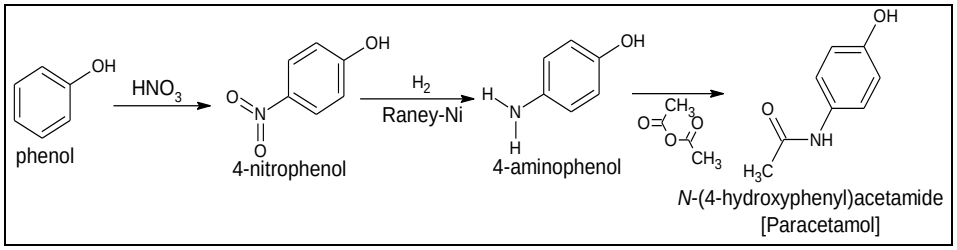
Synthesis of Ibuprofen: (NSAID's)



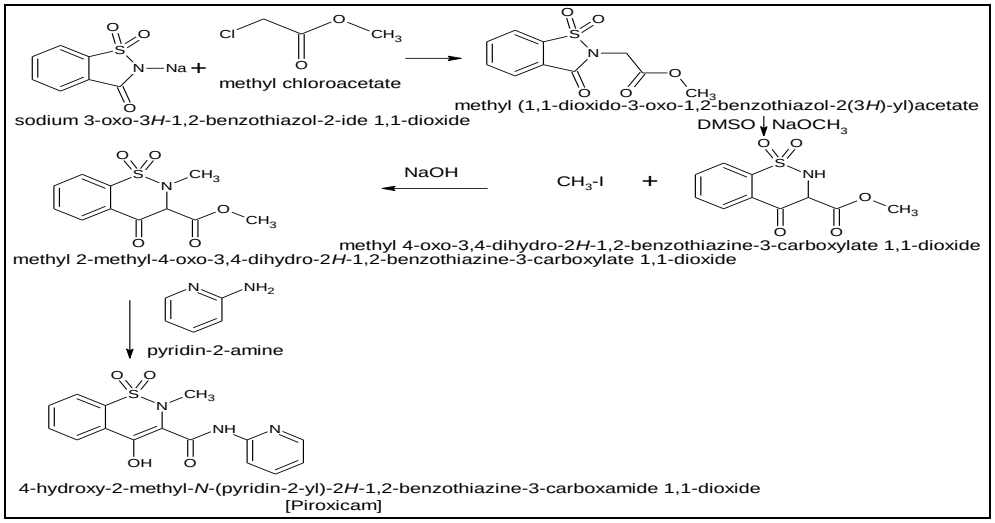
Synthesis of Diclofenac: (NSAID's)



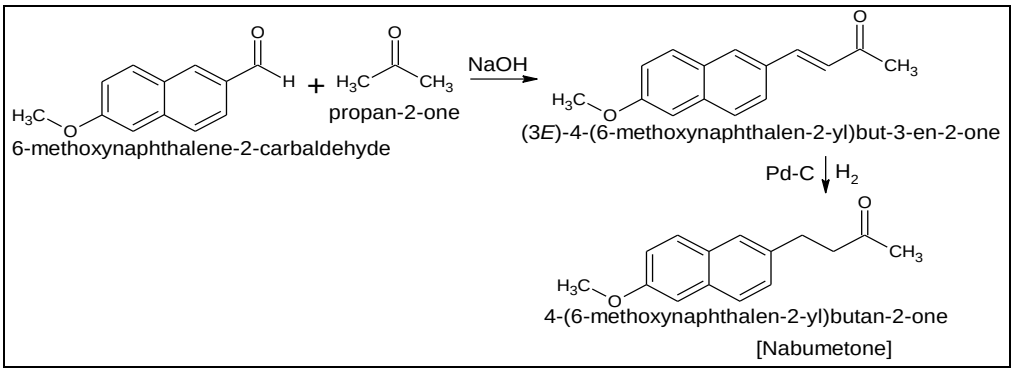
Synthesis of Paracetamol: (Analgesic, Antipyretic)



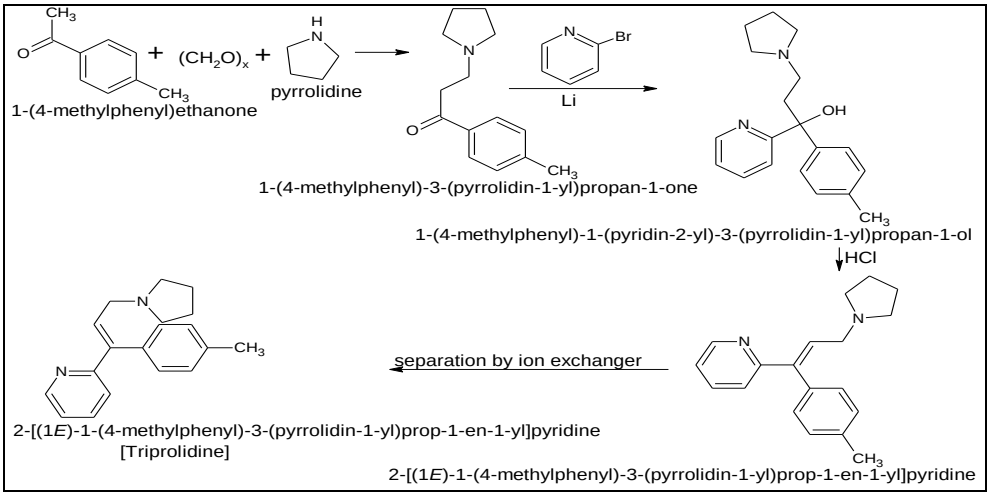
Synthesis of Piroxicam: (Anti-inflammatory)



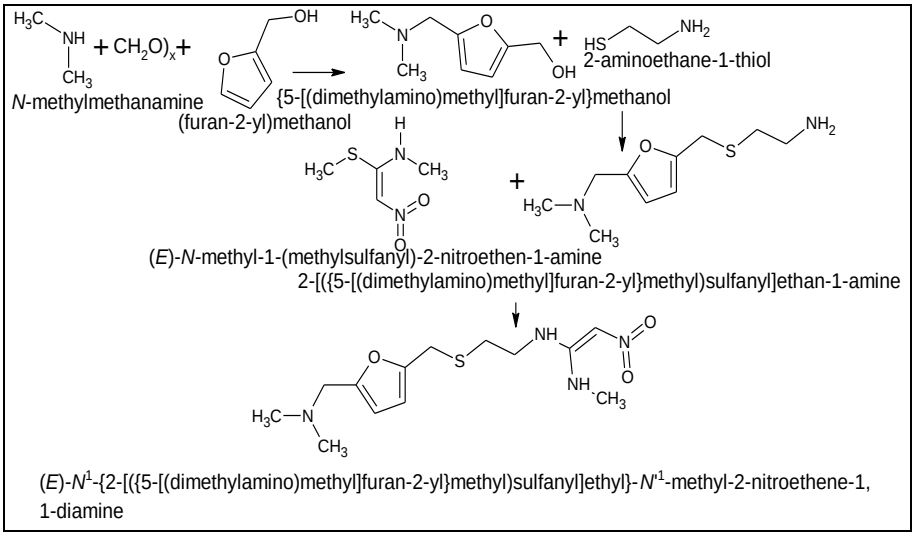
Synthesis of Nabumetone: (Analgesic, Anti-inflammatory)



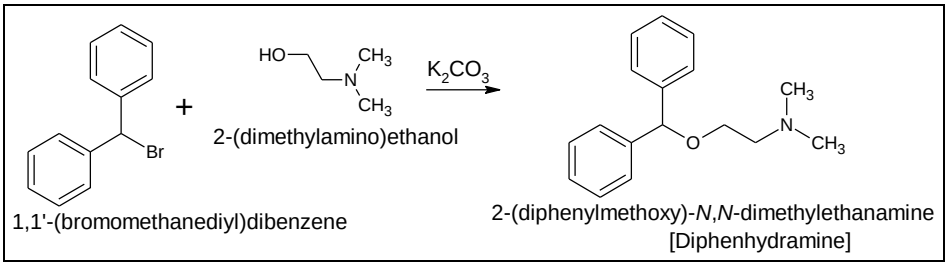
Synthesis of Triprolidine: (Antihistaminic)



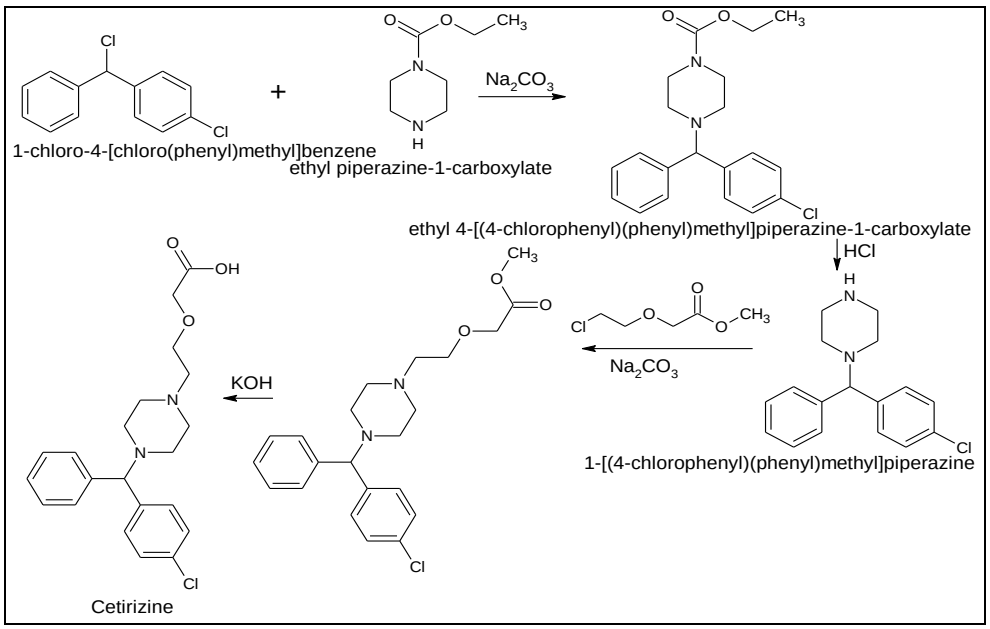
Synthesis of Ranitidine: (Antiulcer)



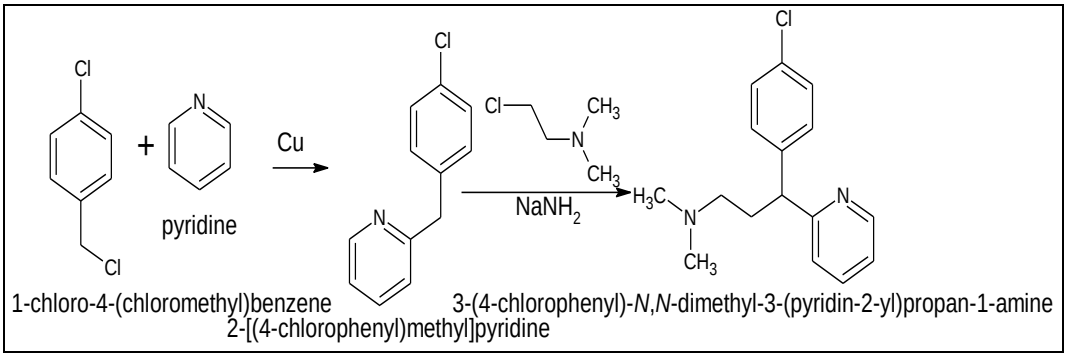
Synthesis of Diphenhydramine: (Antihistaminic)



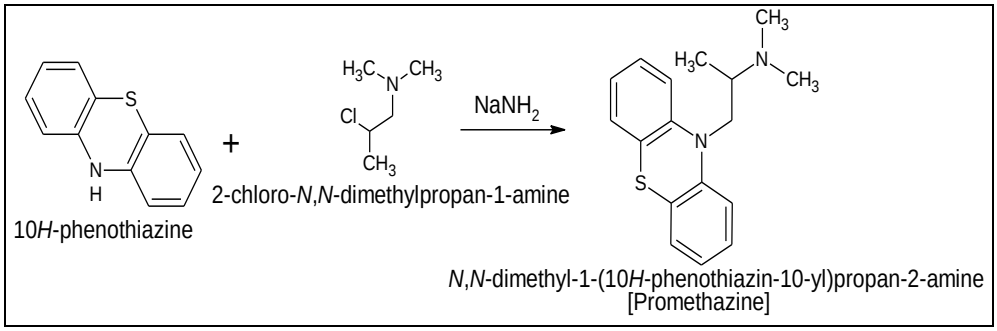
Synthesis of Cetirizine: (Non- sedative Antihistaminic)



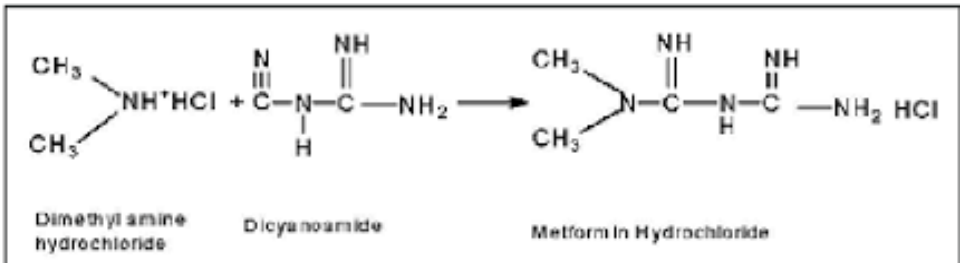
Synthesis of Chlorpheniramine: (Antihistaminic)



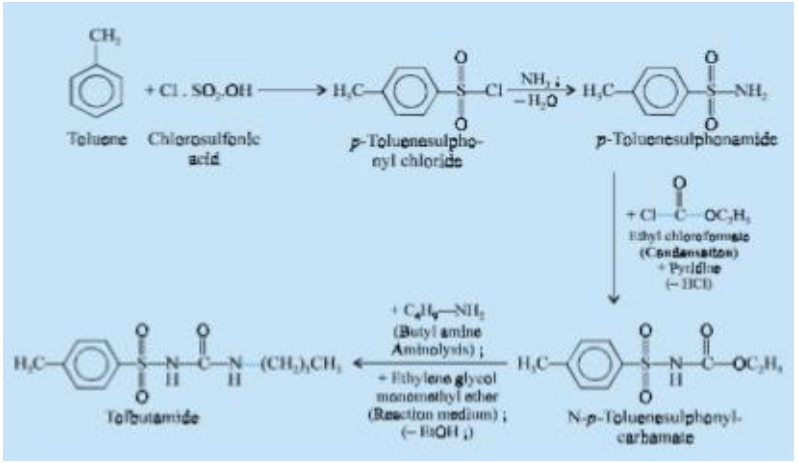
Synthesis of Promethazine: (Antihistaminic, Neuroleptic)



Synthesis of Metformin: (Oral hypoglycaemic agent)



Synthesis of Tolbutamide: (Oral hypoglycaemic agent)



Department: Pharmaceutical Analysis

The Analytica Club

Activity: Introduction to modern analytical instruments and its application in pharmaceutical Industry **.**

Introduction:

The aim of the programme is to produce postgraduates with advanced knowledge and skills of Pharmaceutical Analysis followed with professional ethics to be relevant and competent in the evolving sector of Pharmacy both in national and global perspectives; higher order critical, analytical, problem solving and transferable skills; ability to think rigorously and independently to meet higher level expectations of Pharmaceutical industry, academics, research or take up entrepreneurial route.

The **Intended Learning Outcomes of the Programme are:-**

1. Knowledge and Understanding,
2. Cognitive Skills
3. Practical Skills and
4. Capability/ Transferable Skills.

After undergoing this programme, a student will be able to, discuss the principles involved in modern techniques of Pharmaceutical Analysis, explain the concepts of validation of analytical methods and instruments, identify the drugs and pharmaceuticals based on the functional groups present in the compounds, explain the use of various methods of estimation of different drugs.

Programme Objectives:-

The objectives of the programme are to enable the students to:

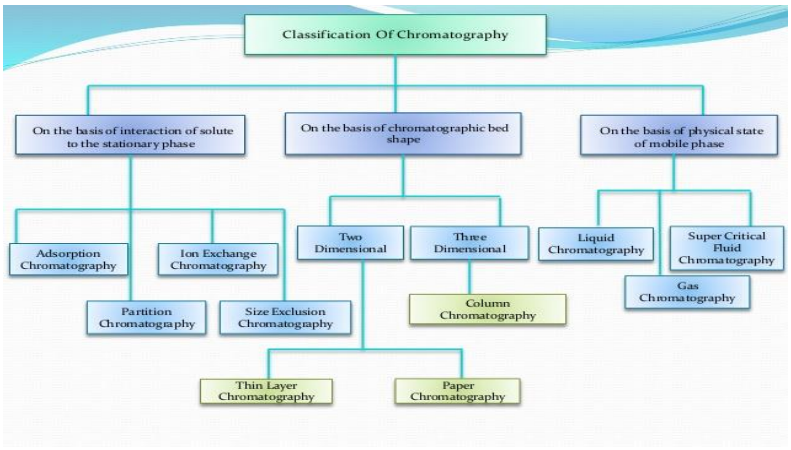
1. Appraise the latest advances in the field of Pharmaceutical Analysis
2. Discuss the principles of different analytical techniques and apply them in professional practice
3. To proficiently use various analytical instruments for analysis of drugs and pharmaceuticals.

ANALYTICAL TECHNIQUES

Chromatographic Techniques

Chromatography is an analytical technique based on the separation of molecules due to differences in their structure and/or composition. In general, chromatography involves moving a sample through the system over a stationary phase. The molecules in the sample will have different affinities and interactions with the stationary support, leading to separation of molecules. Sample components that display stronger interactions with the stationary phase will move more slowly through the column

than components with weaker interactions. Different compounds can be separated from each other as they move through the column.



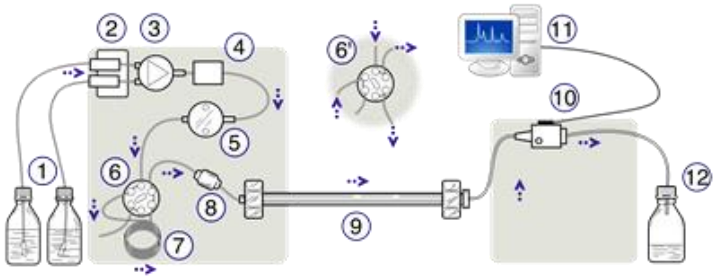
HIGH-PERFORMANCE LIQUID CHROMATOGRAPHY (HPLC)

HPLC: A Powerful Separation Method

A powerful separation method must be able to resolve mixtures with a large number of similar analytes. A chromatogram provides directly both qualitative and quantitative information, each compound in the mixture has its own elution time (the point at which the signal appears on the screen) under a given set of conditions; and both the area and height of each signal are proportional to the amount of the corresponding substance. This example shows that high-performance liquid chromatography (HPLC) is very efficient, i.e. it yields excellent separations in a short time. The stationary phase requires very small particles and hence a high pressure is essential for forcing the mobile phase through the column. As a result, HPLC was sometimes referred to as high pressure liquid chromatography.

HPLC typically utilizes different types of stationary phases, a pump that moves the mobile phase(s) and analyte through the column, and a detector that provides a characteristic retention time for the analyte.

With HPLC, a pump (rather than gravity) provides the higher pressure required to propel the mobile phase and analyte through the densely packed column. The increased density arises from smaller particle sizes. This allows for a better separation on columns of shorter length when compared to ordinary column chromatography.



(1) Solvent reservoirs, (2) Solvent degasser, (3) Gradient valve, (4) Mixing vessel for delivery of the mobile phase, (5) High-pressure pump, (6) Switching valve in "inject position", (6') Switching valve in "load position", (7) Sample injection loop, (8) Pre-column (guard column), (9) Analytical column, (10) Detector (i.e. IR, UV), (11) Data acquisition, (12) Waste or fraction collector.

The choice of solvents, additives and gradient depend on the nature of the column and sample. Often a series of tests are performed on the sample together with a number of trial runs in order to find the HPLC method which gives the best peak separation.

In Reverse Phase HPLC compounds stick to reverse phase HPLC columns in high aqueous mobile phase and are eluted from RP HPLC columns with high organic mobile phase. In RP HPLC compounds are separated based on their hydrophobic character. Peptides can be separated by running a linear gradient of the organic solvent. I often tell my fellow researchers to run the 60/60 gradient when chromatographing an unknown. The 60/60 gradient means that the gradient starts at near 100% aqueous and ramps to 60% organic solvent in 60 minutes. The majority of peptides (10 to 30 amino acid residues in length) will elute by the time the gradient reaches 30% organic.

HPLC Coupling With Various Spectroscopic Techniques

HPLC can be combined with numerous other analytical techniques but the most important coupling principle is the one with spectroscopy. Chromatography is a separation method and spectroscopy is a technique which yields a 'fingerprint' of molecules. Coupling with atomic spectrometry is rarely used although it allows the detection of toxic metals in environmental samples or of metalloproteins. Four other techniques,HPLC-UV, HPLC-FTIR, HPLC-MS and HPLC-NMR are more important because excellent spectra are obtained with them, thus allowing structure elucidation.

HPLC-UV: The Diode Array Detector

Compared to conventional UV detectors, the diode array detector is built with inverse optics. The full light first goes through the detector cell and is subsequently divided spectrally in a polychromatic (which is a grating). The spectral light then reaches the diode array, a chip with a large Number (100-1000) of light-sensitive diodes that are arranged side by side. Each diode only obtains a well defined fraction of the information which is read by the electronics for data processing. This type of detector allows a wealth of information to be obtained from UV spectra. It is possible to obtain and store spectra of individual peaks during a chromatogram. This process does not take more than half a second, including data processing; therefore it is possible to obtain several spectra, even from narrow peaks. For identification the spectra can be compared on-line with a library. In order to improve

correlations, the computer calculates the second derivative which shows more maxima and minima than the original spectrum.

- The knowledge of spectra of the compounds involved then allows detection at selected wavelengths. Interfering peaks can be eliminated due to the proper choice of detection conditions. It is often possible to obtain an accurate quantitation of a compound even if the resolution is poor. The detection wavelength can be altered during a chromatographic run.
- Simultaneous detection at two different wavelengths allows calculation of the absorbance ratio. If this ratio is constant over the whole width of a peak it can be assumed that it is pure (with the exception of the exact coincidence of two peaks with identical shape or of an impurity with identical UV spectrum). If the ratio is not constant this is proof that the peak is not pure.
- Subtraction of two wavelengths allows baseline drift to be reduced during gradient elution as well as noise. The reference wavelength can be chosen in a region where none of the compounds of interest shows absorption.

HPLC-FTIR

The coupling with Fourier-transform infrared spectroscopy allows spectra to be obtained. The interfaces with solvent elimination are more interesting and flexible but technically more demanding; they can even register spectra of trace analytes. A volatile mobile phase is needed; it may be aqueous.

HPLC-MS10

Mass spectrometers for HPLC consist of three different parts: the interface where the eluate enters the MS and the ions are generated, the mass analyzer, and the detector, an electron multiplier which determines the ion beam intensity. Thanks to the different possibilities for ion generation at atmospheric pressure, HPLC-MS is possible with rather low expenditure. The main two ionization techniques are APCI and ESI.

APCI: **Atmospheric pressure chemical ionization**

- Ions are generated by corona discharge (3-6 kV).
- Yields molecule ions (M + H) + (no spectra), negative ionization is also possible.
- Suitable for small, medium and nonpolar molecules; however, they need some proton affinity and volatility.
- Not suitable for thermolabile analytes,
- For aqueous and nonaqueous mobile phases, flow must be at least 1 ml min-1. With nonaqueous eluents no additives are necessary;reactions with the solvent are possible during ionization.
- With aqueous eluents an additive may be necessary for efficient ionization.
- Mass-sensitive signal.

ESI or APESI: **(Atmospheric pressure)**

electro spray ionization (Figure No.4).

- Ions are generated by 'coulomb explosion' (disintegration) of electrically charged droplets.
- Yields ions with single or multiple charge; in the latter case, spectra with many peaks are obtained which must not be mixed up with classical spectra showing molecule fragments.
- Suitable for thermolabile analytes and macromolecules, including biopolymers.
- Suitable for aqueous eluents; flow must be small,therefore useful for micro HPLC. For positive ionization a pH of 5 is suitable, additives are formic and acetic acid, perhaps together with ammonium acetate
- For negative ionization a pH of 9 is suitable; additives are ammonia, triethylamine and diethylamine, perhaps together with ammonium acetate

Concentration-sensitive signal.

Quadrupoles and ion traps are the most frequently used mass analyzers. Both systems are rather low-priced and robust but their resolution is limited to 1Da. The ions can be sent through several mass analysers and fragmentations, setup in series, thus yielding more information about the structure. These techniques are known as MS/MS (two steps) or MSn (n steps).

HPLC-NMR11

Nuclear magnetic resonance can be coupled with HPLC if the solvents are chosen properly (one solvent signal can be suppressed but the others must be avoided by using deuterated solvents) and if the analyte concentration is not too low. Detection limit depends on radiofrequency; therefore a 500-800MHz instrument must be used. If the analyte concentration is high enough, it is possible to runonline HPLC-NMR separations, but usually the individual peaks are stored in loops and spectra are obtained offline with a long enough measuringtime4.

MODEL PRESENTATION BY THIRD YEAR STUDENTS

SPECTROSCOPY MODELS BY STUDENTS

The Herbalist-Cosmo Club

Clubs help students develop their talent. So different activities are planned for the students such as discussion on research paper and plant- photography competition. Formulation preparation is a regular practice, but to create healthy competition a formulation of the week activity is conducted. Details of each club are as under:

Activities under club

I. Research/ Review paper discussion

Purpose:

- Provide insight and new development in the research area

II. Formulation of the week activity and Formulation competition

Purpose:

- Display of formulations prepared by the students.
- Motivating students to observe the marketed formulations, their labels and other labelling details for preparation of better labels of prepared formulations
- Promoting creative and presentation skills of the students.
- Creating healthy competitive atmosphere among the students for better performance

III. Plant-Photo-gallery competition

Purpose:

- To develop interest and is one of the way to take the students towards nature.