PHC 462: OFFICIAL METHODS IN DRUG ANALYSIS (2 + 2)

Prereq.: PHC 462

Course Description

The course deals mainly with quantitative official methods of drug analysis compiled in B.P., U.S.P. and N.F. These are gravimetry, colorimetry, ultraviolet spectrophotometry, fluorometry, atomic absorption spectrophotometry, ion-selective potentiometry, oxygen flask combustion technique and Kjeldahl's method. In addition derivatization as applied in different analytical methods will be covered.

Theoretical		No. of Lectures
I.	**Gravimetry:**	3
	a. Inorganic precipitation -Solubility, saturation, supersaturation. -Nucleation and quality of precipitate. -Growth of precipitates, aging, digestion and Ostwald repening. -Optimum conditions for analytical precipitation. -Types of contamination of precipitates. -Improvement of the physical characters of the precipitates. -The weighing form.	
	b. Organic precipitation -Classes of organic precipitates. -Specificity and selectivity. -Cationic precipitants. -Anionic precipitants. -Chelate precipitants e.g. Dioximes, hydroximes, oxine, neocupferrous. -Optimum conditions for precipitation.	3
II.	**UV-Spectrophotometry and Colorimetry**	
	-Formation of coloured derivatives based on selected functional groups.	2
	-Coloured products by complexation of selected organic colorimetric reagents with metal ions.	1
	-Charge transfer complexes.	1
	-Derivatization of UV-absorbing products.	1

Theoretical — No. of Lectures

III. **Atomic Absorption Spectrophotometry** — 4
-Calibration procedures and non-linearity in calibration graphs.
-Precision and accuracy.
-Back-ground correction.
-Choice of the analytical line.
-Interferences: Non-specific, ionization, chemical and
] physical, matrix.
-Thermal atomizers (CRA-Flameless methods) and principles of operation.
-Atomizers for mercury and the hydride-forming elements e.g. Bi, As, Sn, Se, Sb. Principles of operation.
-Preconcentration techniques.

IV. **Fluorometry** — 4
-Fluorescence and chemical structure.
-Chemical events occurring in the excited state.
-Fluorescence spectra and correction.
-Quantum yields.
-Inner filter effect and role of solvents.
-Use of fluorophores in extrinsic fluorescence.
-Analysis of multi-component fluorescence mixtures.

V. **Nuclear Magnetic Resonance (NMR)**

-The principles of quantitation using NMR. — 2
-Selection of internal standard.
-Limitations of NMR for quantitative purposes.

VI. **Ion-Selective Potentiometry** — 3
-Types and structures of ISE(s)
-Selectivity ratio.
-Nernistian behaviour.
-Use and limitation.

VII. **Miscellaneous** — 2
i) Oxygen flask combustion
Principles of application in the analysis of organic halgens and sulphur compounds.

ii) Kjeldahl's method
Principles of application in the analysis of nitrogen containing compounds.

Examinations. 2

Total 28

PHC 462: Practical

Lab. No.

Gravimetric

-Assay of piperazine phosphate tablets (B.P.)
1 -Thiamine hydrochloride tablets (B.P.)
-Sodium lauryl sulphate (U.S.P.)
-Proguanil hydrochloride.
2 -Determination of Ni^{++} by dimethylglyoxime.
-Determination of Al^{+++} by 8-hydroxyquinoline (oxime).

Colorimetry and UV-spectrophotometry

-Determination of tranexamic acid (anti- inflammatry) with dimethylaminobenzaldehyde.
3 -Determination of nomifensine maleate by diazotization and coupling.
-Determination of Pb^{++} and Cu^{++} using the dithizone colorimetric reagent.
-Determination of thioglycollic acid using Fe^{+++}.
4 -Determination of piperazine by charge transfer using benzoquinone.

Fluorometry

-Recording of corrected spectra of indomethacin at alkaline pH.
5 -Demonstration of inner filter effect.
-Demonstration of the effect of quenching agent e.g. KI on fluorescence intensity.
-Demonstration of the effect temperature and pH on fluorescence intensity of phenobarbitone.
6 -Spectrofluorimetric determination of a mixture of alpha- and β-naphthol.

Atomic absorption spectrophotometry

7 -Determination of Zn by using calibration curve and standard addition methods.
-Determination of Hg using vapour sweep technique.
8 -Determination of As, Sb and Se by hydride generation technique.
-Determination of ultra-trace level of Cd using preconcenration technique.
-Determination of Ca and Mg using CRA.

Lab No.	
	NMR
	-Determination of Aspirin, phenacetin and caffeine (A.P.C.) tablets.
	-Determination of tolmetin sodium capsules.
9	-Determination of mexityl capsules.
	Ion-selective-potentiometry
	-Determination of dissolved oxygen in a water sample.
10	-Determination of nitrate in low levels.
	-Determination of low levels of Ca^{2+}.
	Oxygen flask combustion
	-Determination of di-iodohydroxyquinoline.
11	-Determination of dichlorophen.
	-Determination of elemental sulphur in sulphur ointment.
	Kjeldahl method
	-Determination of ammonium chloride tablets (N.F.)
12	-Determination of neostigmine methylsulphate (U.S.P.)
13, 14	**-Practical Exams.**

14 Total