NORTH MAHARASHTRA UNIVERSITY,
JALGAON
SCHOOL OF CHEMICAL SCIENCES
(Academic Flexibility Since-2009)

SYLLABUS

for

MASTER OF SCIENCE in CHEMISTRY
With
Specialization in
ANALYTICAL CHEMISTRY
M.Sc. II (Semester III and IV)
(Choice Based Credit System, 60:40 Pattern)

w. e. f. June 2016
North Maharashtra University, Jalgaon

School of Chemical Sciences
Department of Analytical Chemistry

M.Sc. II Analytical Chemistry

(Syllabus Structure for Semester-III)
(With effect from June 2016)

AN–301: Concept in Analytical Chemistry. [100]
AN–302: Analytical Techniques in Chemical Analysis. [100]
AN–303: Modern Spectroscopic Techniques. [100]
AN–304: Modern Separation Science. [100]
AN–004: Laboratory Course in Analytical Chemistry –I. [100]

(Syllabus Structure for Semester-IV)
(With effect from June 2016)

AN–401: Advanced Analytical Chemistry. [100]
AN–402: Applied Analytical Chemistry. [100]
AN–403: Pharmaceutical and Cosmetic Analysis [100]
AN–005: Laboratory Course in Analytical Chemistry –II. [100]
AN–006: Project* [100]

* The projects will be initiated in the beginning of Semester III and the examination will be conducted at the end of Semester IV.

* Educational Tour: Organizing Educational Tour aiming at giving practical exposure to second year students is expected (at their own cost).
Unit 1. A) Analytical Science – a Perspective: 

B) Reference Materials (RM)s: 
Analytical standards, primary and secondary standards, high purity substances, reference materials, use of RMs in statistical control schemes and in intercomparisons, role of certified reference materials (CRMs), production and requirements, obtaining reference value and certified value.

Unit 2. Sampling and Calibration: 
Important terms involved in sampling and microanalysis, Methods of sampling, Construction of calibration curves, comparison with single standard, matrix matching, bracketing of std, standard addition and internal standard methods, calibration of Glassware, buoyancy errors, numerical.

Unit 3. Methods for Elemental Analysis: 
Organic samples: Dry and wet ashing methods, Advantages and disadvantages over conventional methods, Special methods, Dissolution of organic samples, Hazards and Safety in laboratory. Inorganic samples: Acids as solvents, fluxes

Unit 4. Optimization of Experimental Design: 
Accuracy, precision, classification of errors, minimization of errors, significant figures and computation, mean deviation and standard deviation, Detection, reduction and compensation of errors, propagation of intermediate error, confidence level, confidence limit when sigma is known and when sigma is unknown, statistical treatment of random error, properties of Gaussian distribution, Test of significance, F test, Q test (Student T test), Construction and interpretation of graphs, fitting the least squares lines, Chemometrics.

Unit 5. Automated Methods of Analysis: 

References:
3. Gas Chromatography, Open Book Learning Series
5. Encyclopedia of Analytical Chemistry.

***
AN–302: Analytical Techniques in Chemical Analysis       [60 Lectures ]

Unit 1. Chemical Analysis and Quality Control:          [12 Lectures ]

A) Industrial Analysis: Quality characteristics of chemical analysis, errors occurring at the start, during or by the end of analysis, interpretation and presentation of results, Shewhart Chart, CUSUM chart and EWMA chart; Batch and process evaluation, QA schemes, experimental designs for optimization studies and ruggedness testing, system management.

B) Clinical Analysis: Introduction, analytical responsibilities, Managerial responsibilities, practical approaches to QA, characterization of a method, Results and preparation of reports. Internal QC, Accuracy and external quality assessment, near-patient testing and QC.

C) Water Industry: Water quality field sampling QA/QC program, QA/QC documentation, QA project plan, designing a water quality monitoring plan, Site selection, sampling frequency and sample size, cost considerations, training of field personnel, field trip preparations, Water quality sampling, toxic chemicals in bottom sampling and biota, bacterial sample collection, sequential triplicate sampling, sample handling, preservation, storage and transport, chain of custody, field safety, field audit program, laboratory QC procedures inter- and intra-laboratory QC, detection limits, reporting of analytical results, data handling and data management.

Unit 2. A) Radiochemical Methods of Analysis:             [12 Lectures]

Neutron activation analysis: Principle, Definition of various terms, various steps involved. Absolute & comparative method, PLNAA pulse N
Isotope Dilution analysis: Principle, Direct, reverse double derivative
IDA. Radiometric titration: Principle, types and instrumentation.
Radio immunoarray: principle and applications.

B) Thermal Methods of Analysis:
Thermogravimetry[TG], Differential thermal analysis [DTA], Differential Scanning calorimetry [DSC], Thermomechanical analysis [TMA] Instrumentation and application, Thermometric titrations.

Unit 3. Polarography, Voltammetry and Ion Selective Electrodes (ISE):       [12 Lectures]

Polarography: Principles, Factors affecting polarographic wave, pulse polarography, and differential pulse polarograph, Voltammetry: Voltammetric principles, Hydrodynamic voltammetry, Stripping voltammetry, Cyclic voltammetry, criteria of reversibility of electrochemical reactions, quasi-reversible and irreversible processes, qualitative and quantitative analysis by these techniques. Types and applications of ISE.

Unit 4. Coulometry:                                           [12 Lectures]

Unit 5. Electrogravimetry: [12 Lectures]

Important terms used in electrogravimetric methods, Overpotential, Electrogravimetric methods, Instrumentation, Electrolysis using a mercury Cathode, Spontaneous Electrolysis, Electrography.

References:

***
Unit 1. Infrared and Raman Spectrometry: [12 Lectures]

Brief introduction to Spectroscopy, Theory of Infrared Absorption Spectrometry, Instrumentation, Sample handling, FT technique, group frequencies, Vibrational coupling, NIR spectroscopy, New applications, Scattering phenomena, Raman spectroscopy, Qualitative and Quantitative analysis.

Unit 2. A) Nuclear Magnetic Resonance Spectroscopy (NMR): [12 Lectures]

Theory of NMR- Basic theory, Quantum and classical description of NMR, Relaxation Processes in NMR, Environmental effects on NMR Spectra-[Chemical shift and factors affecting the same, Spin-spin splitting (1H-1H, 13C-1H, 2D-13C systems), temperature (cyclohexane, DMF and ferrocene)], Rules governing the interpretation of first order spectra, Effect of chemical exchange (D2O Exchange), Low resolution NMR spectra of ethanol, NMR spectra of absolute alcohol, in formic acid and in CCl4 on high resolution instrument. Instrumentation of NMR Spectrometers, Applications of proton NMR, 13C NMR, Simplification of complex spectra, 13C NMR satellite peaks, Spinning sidebands, FTNMR, Pulse technique, double resonance, Off resonance, DEPT, INEPT, Nuclear Overhauser Effect (NOE), 19F and 31P NMR, 2D NMR, Solid State NMR.

B) Electron Spin Resonance Spectroscopy (ESR): [12 Lectures]

NMR and ESR, Theory of ESR, Experimental technique, ESR:- Principle, Instrumentation, ESR spectra, densities and factors affecting g values, Spin labeling ESR spectroscopy, FTESR, Applications, numerical.

Unit 3. Atomic Absorption Spectrophotometry (AAS): [12 Lectures]

Principle-spectral line width, doppler and pressure broadening, Instrument-sources-line and continuous, electrodeless discharge lamps, Hollow cathode lamps, temperature gradients, cells, flames, furnaces, detectors, interferences and modifications in instrumentations, applications, problems discussions.

Unit 4. Molecular Luminescence Spectroscopy: [12 Lectures]

Theory of fluorescence and phosphorescence, variable that affects fluorescence and phosphorescence, Instrumentation for measuring fluorescence, phosphorescence, application of fluorescence and phosphorescence.

Unit 5. Photoelectron Spectroscopy: [12 Lectures]


Reference:

2. Analytical Chemistry, Kellneretal, Wiley VCH.
3. Instrumental Techniques for Analytical Chemistry settle – PTR PH.
5. Analytical Chemistry–Christain G.D, Wiley WSE.

***
AN–304: Modern Separation Science

Unit 1. A) Exclusion (Gel) Chromatography:
Instrumentation, sources of errors, GPC calibration, Column packing, Theory of size of exclusion chromatography, Application of size exclusion chromatography (GPC).

B) Supercritical Fluid Chromatography:
Properties of Supercritical Fluid (SFC) – Instrumentation and operating variables, Comparison with other types of chromatography (HPLC and GLC), Applications.

Unit 2. Capillary Electrophoresis and Electrochromatography:
Overview of Electrophoresis, Capillary Electrophoresis, Applications of Capillary Electrophoresis and Capillary electrochromatography.

Unit 3. Gas Chromatography:

Unit 4. High Performance Liquid Chromatography:
Introduction, GC and HPLC, Instrumentation, Refractive index detector, luminescence detector, ultraviolet detector and electrochemical detector, Quantitative analysis and data display, Derivatisation technique in HPLC, Chiral columns, C8 and C18 columns, Applications.

Unit 5. Solvent Extraction Separation:
Principles of solvent extraction, formation of metal complexes, distribution of extractable species, quantitative treatment of extractable equilibria, Methods of extraction, techniques in extraction, Extraction chromatography, theoretical aspects of extraction chromatography, correlation between solvent extraction and extraction chromatography, techniques in extraction chromatography, chromatographic inert support, stationary phases, use of extraction chromatography for separation of fission products.

References:
2. Analytical Chemistry, Kellneretal, Wiley VCH
3. Analytical Chemistry-Christain G.D, Wiley WSE.

***
Analytical Chemistry Practical–I: (Any 15 experiments)

1. Consumer products (e.g. Inorganic Pigment [e.g. chromium from Zinc chrome]; Pharmaceutical product [magnesium from tablet of “Milk of magnesia” / calcium from calcium-supplementary tablet / aluminium from alum]
2. Qualitative and quantitative determination of metals (Zn, Cd, and Mn) alone and in mixture by classical and differential pulse polarography.
3. Thermogravimetry [Determination of percentage of MgCO$_3$ in Dolomite]
4. Cyclic voltametry [Study of cyclic voltammogram of K$_3$[Fe(CN)$_6$]
5. Determination of moisture content in food sample using Karl-Fischer Titrator.
6. Determination of Phosphate in Detergents by Spectrophotometry.
7. Determination of phosphoric acid in cola beverages by pH titration.
8. Photometric Titrations: (a) Cu Vs EDTA (b) Fe Vs EDTA using salicylic acid.
9. Determination of sap value and iodine, sap, acid value of an oil.
10. Determination of chloride and sulfate with an adsorption indicator.
11. Determination of total salts by cation exchange.
14. Chromatographic separation of sugars, amino acids by paper, T.L.C. and Ion exchange methods (both qualitative and quantitative method) separation organic compounds by column chromatography.
15. Estimation of milk powder for Ca, Fe and P content.
16. Determination of chemical oxygen demand (COD) of polluted water samples.
17. Determination of percentage purity of commercial washing soda by potentiometric titration.
18. Determination of calcium in egg shell by flame photometric method.
19. Determination of potassium in soil by flame photometric method (Lithium internal standard method).
20. Estimation of vitamin B$_2$ in drug sample by fluorometry.
22. Determination of organic amines by potentiometric titration in glacial acetic acid.
23. Determination of pKa value of an indicator.
24. Determination of sulfate nephelometrically.
25. Analysis of copper by extractive photometry using diethyl dithiocarbamate.

Each experiment includes standardization of the reagents, calibration of the instrument with known reagents and analysis of an unknown.

References
1. A. I. Vogel, A Textbook of Quantitative Inorganic analysis, 2$^{nd}$ Ed., ELBS Ed.
AN–401: Advanced Analytical Chemistry

Unit 1. Fundamentals of X–ray Diffraction:

Unit 2. Mossbauer Spectroscopy:
Introduction to Mossbauer effect, recoilless emission & absorption of X–rays, Instrumentation, Spectral parameters of Mossbauer spectra such as Isomer shift, Quadruple splitting and Hyperfine interactions, application of Mossbauer effect to the investigations of compounds of iron and tin.

Unit 3. Atomic Emission Spectrometry (AES):
Inductively coupled plasma–ICP / AES and Flame emission spectroscopy (FES). Sources electrical discharge, dc/ac arcs, spark laser microprobe, qualitative and quantitative analysis, problems discussion.

Unit 4. A) Laser Based Techniques:
Atomic fluorescent spectrometry (AFS), resonant ionisation spectroscopy (RIS), Laser enhanced ionization (LEI). Principle–types of transition tunable laser, Classification of medium pumping and controlling mechanisms, Instrumentation detailing of various gaseous, liquid and solid sources, cell, Monochromators, Detectors.

B) Hyphenated Techniques:
GC–MS, LC–MS, Total ion current (TIC) chromatogram, MS–MS (Tandem) Spectrometry, ICP–MS.

Unit 5. Imaging Techniques including MRI:
Magnetic resonance imaging (MRI)–Principle, Instrumentation, Magnetic resonance angiography, $^1$H-NMR of relevant diamagnetic and paramagnetic compounds, Contrast agents and Clinical applications.

References:
2. Diffraction Method, Wormald, Oxford University, Press, 1973
Unit 1. Metallurgy: [12 Lectures]
Ores and minerals, dressing of ore, Methods of metal dressing (hand picking, magnetic separation, centrifuge, froth flotation etc.), Pollution due to metallurgical process (Metal dressing, calcinations, smelting), Alloying: Definition, Purposeful development of alloy, Carat of Gold (precious material) and its method of analysis, Techniques of purification: Zone refining, Analysis of high purity materials like silicon, Vacuum fusion and extraction techniques.

Unit 2. Food Chemistry: [12 Lectures]
Definition and importance, Water in food, Water activity and shelf life of food, Carbohydrates – chemical reactions, Functional properties of Sugars and Polysaccharides in foods, Lipids: Classification and use of lipids in foods, Physical and Chemical properties, Effects of processing on functional properties and nutritive value, Protein and amino acids – Physical and Chemical properties, Distribution, amount and functions of proteins in foods, functional properties, Effect of processing-loss of vitamins and minerals due to processing, Pigments in food, Food Flavors, Browning reaction in foods, Enzymes in foods and food industry, Bio-deterioration of foods, Food contaminants (Adulterants), Additives and toxicants.

Unit 3. Analysis of Agrochemicals: [12 Lectures]
Sampling, Extraction, Clean Up, Analysis of pesticides, Application of chemical as well as Bioassay methods for analysis of pesticide residue, Analysis of DDT, Malathion, BHC from dust residue.

Unit 4. Analysis of Soil: [12 Lectures]
Method of soil analysis, soil fertility its determination, determination of inorganic constituents of plant materials, Chemical analysis as measure of soil fertility, analysis of Fertilizers.

Unit 5. Forensic Analysis: [12 Lectures]
Overview, Destructive and Nondestructive techniques, Data interpretation.
Blood Analysis: Blood preservation and ageing effects, Analysis of blood components and exogenic substances, blood stain analysis.
Determination of alcohol in body fluids: Legal background, Sampling and sample preservation, analysis–GC, IR, enzymatic and other methods.
Fingerprint analysis: Latent fingerprints; Optical, Physical, Physico–chemical & Chemical detection methods; Fingerprints in blood, Fingerprint detection sequences.
Hair analysis: Structure and composition of hair, Morphological examination, Chemical analysis of hair components and components remaining on or in hair.
Systematic Drug Identification: Classification and categories of compounds involved, Analytical strategy–EMIT, FPIA, TLC, LC, GC–MS, etc., Requirements for identification,
Possibilities & limitations of selected techniques, Isotope detection method with numerical, New drug groups.

References:


***
Unit 1. Pharmaceutical Industry: a review
Definition and classification of drugs and medicines, introduction to pharmaceutical formulations, classification of dosage forms, Sources of impurities in pharmaceutical chemicals and raw materials.

Unit 2. Official Methods of Standardization and Quality Control:
Standardization of finished products and their characteristics, Official methods of Quality control, Dissolution Tests and Kinetics.

Unit 3. Assay and Tests for Drugs:
Analysis of compounds based on functional groups, instrumental methods for analysis of drugs, assays involving chromatographic separations, proximate assays, assays of enzyme containing substances, biological and microbiological assays and tests. Limit tests, solubility tests, disintegration tests, stability studies, impurity profile of drugs, bioequivalence and bioavailability studies.

Unit 4. Introduction to Cosmetics:

Unit 5. Analysis of Cosmetics:
Deodorants and Antiperspirants: Al, Zn, Zr, Boric acid, chlorides, sulphates, hexachlorophene, methanamine, phenolsulphonates and urea

Face powder: Fats, fatty acids, boric acid, Ca, Mg, BaSO4, Ti, Fe, oxides of Tl, Fe and Al (total).

Hair tonic: 2,5-diaminotoluene, potassium bromates, sodium perborate, pyrogallol, resorcinol, salicylic acid, dithioglycollic acid (in permanent wavers)

Creams and lotions: types of emulsions, chloroform soluble material, glycerol,pH emulsion, ash analysis, non volatile matter by IR spectroscopy.

Lipsticks: General analysis, determination of nonvolatile matter, ash analysis determination of lakes and fillers, trichloroethylene – acetone soluble contents.

References:
G) Harry’s Cosmetology, Longman scientific co.
H) Formulation and Function of cosmetics, Sa Jellineck.
I) Cosmetic Technology, Saggarin.
K) Encyclopaedia of industrial chemical analysis, Snell et al Inter science.
L) Govt of India publications of food drug cosmetic act and rules.
Analytical Chemistry Practical–I: (Any 15 experiments)

5. Pharmaceutical Analysis-Dissolution test, Disintegration test, Weight Variation test, Test for uniformity of content.
6. Estimation of following functional groups
   Phenolic/amino group, ester group, amide group
7. Fertilizer analysis for N, P, K.
8. Analysis of vitamin–A in food products.
11. Chemical analysis of chill/turmeric powder.
12. Potentiometric Titrations: (a) FAS Vs K2Cr2O7 (b) FAS Vs KMnO4.
13. Determination of strength of acetic acid in commercial vinegar sample conductometrically.
14. Molecular weight of polymer from Viscosity measurements
   (Experiments in physical chemistry–J. M. Wilson and others page no. 202)
15. Differential potentiometric titration
   (Experiments in physical chemistry–J. M. Wilson and others page no. 272)
16. TGA for a mixture of CuSO4 and NaCl, find out the percentage of each constituent in the Mixture.
17. Estimation of Zn and Cd from their mixture by polarographic technique
18. Quantitative analysis of mixture by gas Chromatography [e. g. Chloroform and carbon Tetrachloride / methanol and ethanol]
19. Table work for IR-spectra, NMR, UV–Visible spectra.
21. Estimation of lactose in milk iodometrically and Isolation of casein from given milk sample.
22. Determination of exchange capacity of a cation or anion exchange resin.
23. Separation of dichromate and permanganate ion using an alumina column.
24. Determination of number of theoretical plates of a C18 HPLC column.
25. Separation and estimation of a mixture of acetophenone, benzene and toluene by using C18 column with acetonitrile: water (60:40) mobile phase
26. Simultaneous determination of copper and bismuth in a mixture using EDTA spectrophotometrically.
27. Determination of composition of the complex by Job's continuous variation method and stability constant study.
28. Determination of HCl and H2SO4 by conductometric titration with NaOH and BaCl2.
29. Spectrophotometric determination of pH of a buffer mixture.

Each experiment includes standardization of the reagents, calibration of the instrument with known reagents and analysis of an unknown.

References
* The projects will be initiated in the beginning of Semester III and the examination will be conducted at the end of Semester IV.

***