

# Practical Laboratory Skills

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The *Practical Laboratory Skills* series of e-Learning modules has been carefully and intelligently designed for use as a training and teaching tool in industrial, academic, environmental, government, and medical laboratories.

Each program contains about 30 minutes of video, comprising between 3 and 8 close-up demonstrations of the correct and safe method of carrying out a particular laboratory procedure or experiment.

Audio narration, text display and a technical glossary enhance the learning experience. Assessment is accomplished through automatically graded multiple-choice quizzes and a set of open-ended review questions for each module.



## Practical Laboratory Skills modules feature:

- High quality video
- Menus giving easy access to all sections
- Full screen option
- Spoken commentary
- Multiple-choice quizzes
- Open-ended questions for class discussion
- On-screen text which can be printed out
- Compatible with Microsoft Windows and Apple Mac

## Basic Laboratory Techniques

**Assembling Apparatus:** Laboratory glassware; greasing joints and using PTFE sleeves; condensers with fixed and detachable side-arms; supporting glassware with clamps, bosses and stands; distillation and refluxing; safety procedures for overnight working. 7:30 Minutes

**Using Stirrers:** Types of stirrers: magnetic, mechanical, paddle; attaching a stirrer rod to motor; sealing of glands for controlled atmospheres, setting up a stirrer with motor, flask and oil cup seal. 6:57 Minutes

**Heating Samples:** The Bunsen and micro burner, steam baths, stirrer hotplates, oil baths, and heating mantles; safety aspects. 10:17 Minutes

**Using a Rotary Evaporator:** Initial set up, mounting the sample flask, evaporation of solvent from sample, the use of a water bath, obtaining the product; shutdown procedure, disposal of unwanted solvent. 4:19 Minutes

### Program PLS-101

## Distillation Techniques

**Distillation at Atmospheric Pressure:** Distillation below 200°C, safe disposal of uncondensed vapors; water-cooled condenser; using a thermometer; oil bath; distillation and collection of distillate; shutdown and dismantling. 7:14 Minutes

**Distillation at Reduced Pressure:** Two-necked Claisen head, water-cooled condenser, capillary bleed, use of oxygen-free nitrogen-filled

balloon, multi-flask receiver-adaptor, water pump, solid carbon dioxide/acetone trap, oil bath for heating; distillation and collection of fractions. Appendix on testing capillary bleed. 7:58 Minutes

**Fractional Distillation:** Design of fractionating columns; demonstration of procedure from initial set up of apparatus to obtaining the fractions, column cleaning, shutting down. 5:11 Minutes

**Steam Distillation:** Use of a two-necked flask, splash head, dropping funnel and double-surface condenser; problems arising from low melting point solids; steam generator system; shutdown procedure. 4:34 Minutes

**Semi-Microdistillation:** Examination of pear-shaped flask for star cracks; use of the micro-burner, glass wool; distillation under reduced pressure; shutdown procedure. 1:49 Minutes

### Program PLS-102



## Purification Techniques

**Gravity Filtration:** Funnel and filter paper selection; cold filtration procedure; hot filtration on a steam bath; simple quantitative filtration.

Appendix on folding small, medium and large sized fluted filter papers. 5:15 Minutes

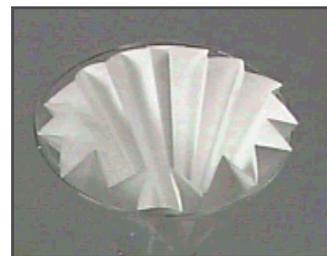
**Suction Filtration:** The Buchner funnel and flask, use of aspirators and vacuum systems, avoidance of solid precipitating out in flask by pre-cooling; drying the solid; Hirsch funnel, Buchner tube; shutdown procedure. 7:02 Minutes

**Recrystallization:** Heating the solvent, adding the compound, hot filtration using a fluted filter paper and short stemmed glass funnel, obtaining, washing and drying the crystals. Appendices on: solubility testing, promoting recrystallization, handling oils and treating discolored samples with activated charcoal. 11:30 Minutes

**Sublimation:** Sublimation at atmospheric pressure; reduced pressure technique for less volatile substances; subliming larger quantities. 3:42 Minutes

**Refluxing:** Setting up apparatus, charging the flask, choosing heat source, controlling temperature, safety hints; shutdown procedure. 3:09 Minutes

### Program PLS-103



## Minimum System Requirements:

- Windows PC/Apple Macintosh
- 64MB RAM
- Adobe Macromedia Flash Player
- Web browser (Internet Explorer/Firefox/Chrome/Safari)
- CD drive for CD version
- 1 Mbps internet connection speed
- Sound card, speakers/headphones

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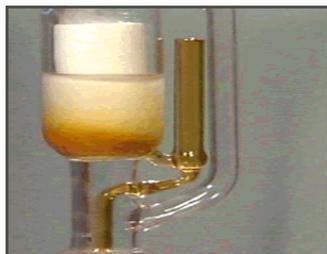
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## Extraction Techniques

**Solvent Extraction:** Use of separating funnel, extractions with organic layer lighter and heavier than water; drying. Appendix on emulsions. 8:44 Minutes

**Soxhlet Extraction:** Apparatus set-up, siphon discharge action, loading sample, filling the flask, heating; shut down procedure. 5:05 Minutes

**Continuous Flow Extraction:** Choosing the solvent; extractions using solvent less or more dense than sample solvent; post-extraction. 7:12 Minutes.

**Drying Samples:** Removal of water from thermally stable solids by suction or oven heating and from thermally unstable compounds with a desiccator; use of desiccator cabinets, choosing desiccants, safe use of a vacuum desiccator; removal of water from liquids using drying agents, removal of drying agent; drying gases using molecular sieve and a drying tower, safe use of anhydrous calcium chloride. 8:35 Minutes

**Program PLS-104**



## IR, UV/Vis & NMR Spectroscopy

**Infrared Spectroscopy of Solids:** Sampling techniques, including: storage and handling of potassium bromide, using a mortar and pestle, disc pressing, transferring sample disc to sample holder; Nujol mull technique including: alkali halide plates, estimating quantities, mull preparation and application, cleaning and care of plates. 9:42 Minutes

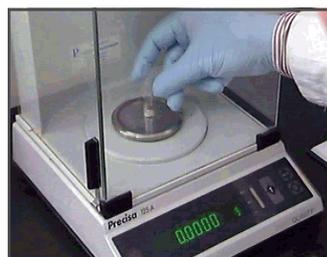
**Infrared Spectroscopy of Liquids:** Handling alkali halide plates, adding liquid between plates, mounting plates in plate holder; obtaining infrared spectra of dilute solutions including: the choice of spectroscopic grade solvent, filling cells with solution, mounting cells in spectrophotometer, cleaning and drying cells. 4:26 Minutes

**Infrared Spectroscopy of Gases:** Filling a gas cell by the purging method using carbon monoxide; loading gas cell into spectrometer, obtaining a rotation-vibration spectrum, and flushing gas cell with nitrogen gas to clean it. Appendix on use of vacuum line to fill cells. 5:16 Minutes

**Ultraviolet and Visible Spectroscopy:** UV/Visible absorption spectra; selecting a solvent, making up samples, cuvette care, adding sample and reference to cuvettes, locating cuvettes in a double beam spectrometer; tips if bands are too strong or too weak. Appendix on spectral interpretation. 4:54 Minutes

**Proton NMR Spectroscopy:** Materials and methods, deuterated solvents, NMR tubes; selecting a solvent, using non-deuterated solvent for solubility testing, cleaning tube, making up a sample, *in situ* filtration of sample solution, inserting tube into spinner, types of instrument; obtaining a spectrum; NMR spectra for other nuclei. 5:38 Minutes

**Program PLS-105**



## Microscale Laboratory Techniques

**Assembling Apparatus:** Microscale apparatus: reaction containers, magnetic stirring, setting up a reflux, use of Teflon tape to join glassware, assembling; protection against moisture including: anhydrous calcium chloride tubes, silicon rubber septum caps, hypodermic needles. Appendix on using apparatus with screw-capped couplings. 4:17 Minutes

**Weighing and Material Transfer:** Use of four-decimal-place balance, procedures for transferring and

weighing solids and liquids; calibrated pipettes; manual variable-volume dispensing pipette; graduated pipette; syringes; handling air and moisture-sensitive compounds including: inserting a needle, using a balloon filled with inert gas, and inserting syringe into a stock liquid container. 6:45 Minutes

**Heating Samples:** Sand bath, hotplate, measuring temperature, exploitation of temperature gradient to effect gentle or vigorous heating. 1:27 Minutes

**Gravity Filtration:** Pasteur pipette using cotton wool, shortening pipette tip, filtering, using extra solvent to rinse pipette so as to ensure maximum recovery of sample. 1:25 Minutes

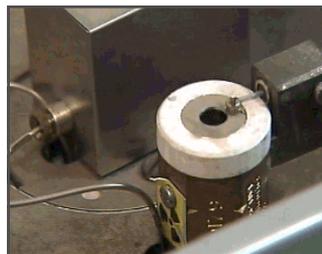
**Suction Filtration:** Assembling a Hirsch funnel, choosing a vacuum source, adding suspended solids to funnel, filtering and washing solid with filtrate, drying sample under vacuum. 2:42 Minutes

**Extraction and Partition:** Pasteur pipette; mixing solvent layers; removing bottom or top layers; extracting immiscible organic solid from aqueous solution; removing inorganic impurities; drying extracts with anhydrous magnesium sulfate; removing drying agent; isolating product by boiling off solvent. 6:56 Minutes

**Distillation at Atmospheric Pressure:** Hickman still, heating on a sand bath, transferring liquid to flask, making secure joints, condensate collection, shut down. 2:54 Minutes

**Distillation of Higher Boiling Liquids:** Assembling apparatus, choosing source of vacuum, insulating flask, collecting distillate. 2:00 Minutes

**Program PLS-106**



## Chromatographic Techniques

**Thin Layer Chromatography (TLC):** Chromatography plate, choice of eluting solvent; marking the plate, using a capillary tube to spot the plate, using a filter paper to saturate atmosphere, the separation, removal of plate, drying; detecting spots using the naked eye, UV light,

staining; interpreting results; monitoring reactions; calculating R<sub>f</sub> values. 6:42 Minutes

**Column Chromatography:** Use of TLC to pre-select solvent system; selection and preparation of columns, adding solvent/silica gel slurry, recycling solvent, elimination of air bubbles, using compressed air to force fresh solvent through adding sample; collection of eluent, analysis of fractions by TLC, combination of fractions and concentration. 6:36 Minutes

**Ion Exchange Chromatography:** Preparation of resin; preparation of column; addition of resin; draining off excess water; regeneration, addition of sample, eluting and testing/titrating eluent. 8:15 Minutes

**Gas Chromatography:** Packed and capillary columns, ovens, mobile phase/carrier gas, setting injector-port and oven temperatures, dissolving sample, sample injection, gaseous samples; detection, retention times; identification of unknown compounds. 7:43 Minutes

**Program PLS-107**



## Volumetric Techniques

**Using Balances:** Weighing in the laboratory: general purpose balance, analytical balance, containment vessels, location of balances, leveling a balance; weighing solids and liquid reagents, use of tongs, tweezers and gloves, quantitative transfer, weighing by difference, absolute weights method, gravimetric analysis and ashing. 9:10 Minutes

**Using a Pipette:** Volumetric and measuring pipettes, pi-pump, rubber bulbs, safety precautions; rinsing and filling, calibration, aliquot transfer, valve leaks. 9:16 Minutes

**Using a Burette:** Capacity, stopcocks, leaks, rinsing; filling; removal of air, burette technique. 4:32 Minutes

**Making up Solutions:** Choosing a volumetric flask; storing standard stock solutions; example based on copper salt: drying and cooling, weighing, dissolution, mixing, diluting stock solutions. 7:04 Minutes

**Program PLS-108**

# live action ... shot in authentic lab settings



## Inorganic Analysis Methods

**Gravimetric Analysis:** Drying sintered glass crucibles, quantitative transfers of liquid or solid samples to reaction vessels, addition of reagents, testing for complete precipitation, digestion, transferring, filtering, drying and weighing. 11:02 Minutes

**Electrogravimetric Analysis for Metals:** Weighing electrode and salt, dissolution, acidification, setting up stirrer, mounting and connecting electrodes, applying voltage to electrogravimetric cell, testing for complete deposition, washing, drying and weighing electrode, procedure for mixed metal solutions. 8:30 Minutes

**Kjeldahl Determination of Nitrogen:** Weighing salt to four decimal places, transfer to digestion flask, adding acid and indicator to receiving flask, adding alkali to digestion flask, heating, titration, Kjeldahl digestion using heat and concentrated acid to break down compounds. 10:01 Minutes

**Program PLS-109**



## Measuring & Controlling Temperatures

**Measuring and Controlling Temperature:** Mercury, Beckman and pentane thermometers, accuracy and limitations, immersion depths, use in Quickfit apparatus, repairing a broken bead; electrical temperature measurement: thermocouple and meter, bimetallic junctions, thermistor, non-contact thermometers; controlling temperatures, constant-temperature baths. 7:54 Minutes

**Determination of Melting Temperature:** Purity, identity; grinding samples, transferring to a melting temperature tube, using an aluminum block apparatus; mixed melting temperature determination using two different substances. Appendices on sealing, Hot Stage Microscope. 8:12 Minutes

**Determination of Boiling Temperature:** Making a fine capillary tube, breaking and sealing, loading into melting temperature apparatus, recording boiling temperature. 2:42 Minutes

**Determining a Liquid/Liquid Phase Diagram:** Using phenol/water system: preparing known composition mixtures, placing tubes, heating water jacket, recording temperature; plotting a phase diagram. 3:44 Minutes

**Determining a Solid/Solid Phase Diagram:** Using acetamide/p-nitrophenol system: preparing known composition mixtures, heating each tube until melting the solids, cooling, recording temperature; plotting a phase diagram. 2:46 Minutes

**Program PLS-110**



## Microscale Chromatography

**Thin Layer Chromatography:** Choosing plate and eluting solvent; marking plate, spotting, saturating atmosphere with solvent, separation, removal, drying; detecting spots by the naked eye, UV light, staining; interpreting results; monitoring reactions, measuring Rf values. 6:42 Minutes

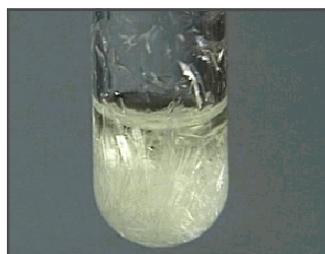
**Column Chromatography:** Comparison of microscale with conventional chromatography; preparing column and reagents including: shortening pipette, inserting plug, clamping and filling, adding solvent, removing air bubbles and cracks; sample loading; eluting fractions; identification of the separated components via TLC; recovery of products. 9:56 Minutes

**Gas Chromatography:** Columns, oven, mobile phase/carrier gas; setting injector-port and oven temperatures, dissolving sample,

injecting onto column, detection of eluents; retention times; identification of unknown compounds. 4:32 Minutes

**Preparation of a Grignard Reagent:** Assembling apparatus and preparation: sealing condenser, fitting it with hypodermic needle and drying tube; adding reactants, refluxing, injection; purification of products: reaction with acid, separation and extraction of aqueous and ether layers, drying, filtering, evaporation. 8:17 Minutes

**Program PLS-111**



## Microscale Purification & Characterization

**Recrystallization:** Solvent test using heated samples, filtration, reducing solvent volume, cooling and crystallization, filtration of product using a Hirsch funnel, washing and drying crystals. 8:46 Minutes

**Recrystallization Using a Craig Tube:** Dissolving sample, filtration, collecting filtrate and washings in a Craig tube, reducing solvent volume, capping, cooling, centrifugation, recovery, washing and drying crystals; dealing with very finely divided crystals. 5:12 Minutes

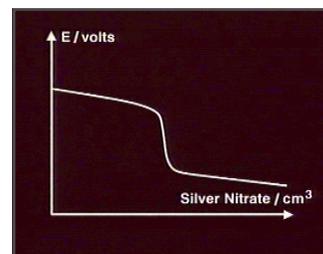
**Determination of Boiling Temperature:** Heating a melting temperature tube to form a very fine capillary, breaking and sealing, placing capillary tube and liquid, loading assembly, recording boiling temperature. 2:42 Minutes

**Determination of Melting Temperature:** Grinding a sample, transfer to melting point tube, using an aluminum block melting point apparatus; mixed melting temperature determination. Appendix on using a Hot Stage Microscope. 5:44 Minutes

**Infrared Spectroscopy of Solids:** Sampling techniques; potassium bromide disc; storage and handling, using pestle and mortar to mix, disc pressing, mounting in the infrared spectrometer, cleaning die by dissolving, oven-drying; Nujol mulls: alkali halide plates, estimating quantities, preparation, application, cleaning. 5:00 Minutes

**Infrared Spectroscopy of Liquids:** Handling alkali halide plates, adding liquid between plates, mounting in a plate holder; obtaining a spectrum; reducing thickness of liquid film; cleaning and care. 1:58 Minutes

**Program PLS-112**



## Volumetric Analysis Methods

**Doing a Titration:** Preparing burette; titration flask; pipetting reagents; preliminary and accurate titration, half-drop technique; using an automatic titrator. 5:09 Minutes

**Some Common End Points:** Using indicators in titrations; endpoint demonstrations: methyl red, methyl orange, screened methyl orange, phenolphthalein, bromothymol blue, bromocresol green, eriochrome black T, murexide, fluorescein, potassium chromate, ammonium iron (III) sulfate, starch, self-indicating immiscible solvents, sodium diphenylamine sulfonate, ferroin, self-indicating potassium permanganate. 6:19 Minutes

**Potentiometric Titrations:** pH titrations: comparison of indicator and instrumental methods, pH meter, calibration, accurate titration, examples, multiple endpoints; redox and silver halide titrations; preparing systems, taking readings, caring for electrodes, plotting results. 17:32 Minutes

**Program PLS-113**



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## Thermochemical Measurements

### Using a Bomb Calorimeter:

Thermochemical measurements; calibration; preparation; firing the bomb; plotting results; correction factors; sampling techniques for solid and liquid sample  
13:15 Minutes

### Using a Dewar Calorimeter:

Description, preparing for a run; obtaining readings; interpreting data; dealing with corrections.  
5:20 Minutes

**Determination of Enthalpy of Neutralisation:** Use of Dewar calorimetry for measuring enthalpies; preparation; taking calibration readings; determining corrections for dilution effects; obtaining results for samples; interpreting data.  
9:20 Minutes

### Program PLS-114



## Solution Kinetics Techniques

**Oxidation of Iodide By Hydrogen Peroxide:** Background to iodine clock reaction; defining experimental conditions; making up solutions; carrying out experiment; plotting results; determining the importance of other variables.  
5:51 Minutes

**Oxidation of Iodide By Potassium Peroxydisulfate:** Background to kinetic determinations; preparing equipment; making up reagents; carrying out kinetic runs; plotting results; obtaining activation energies; determining importance of other variables.  
5:40 Minutes

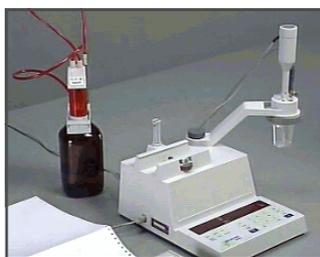
**Iodination of Ketones:** Background to halogenation reactions; importance of controlling temperature; preparing solutions; setting up UV/visible spectrometer; carrying out experiment; plotting results; obtaining kinetic parameters.  
8:45 Minutes

**Measuring Solution Partition Coefficients:** Partitioning material between solvents with differing polarities; determining saturation concentrations for iodine in water

and toluene; using titration and spectrophotometric methods.  
5:56 Minutes

**Colorimetric Analysis:** Preparing solutions; calibration procedure; measuring unknown samples; plotting results; caring for solution cells.  
3:37 Minutes

### Program PLS-115



## Electrochemical Techniques

**Using Galvanic Cells:** Components of galvanic cells; different types of electrodes; caring for electrodes; calibration of digital voltmeters; using salt bridges.  
4:09 Minutes

**Determination of Standard Electrode Potentials:** Preparing sample and reference cell; using a salt bridge; using digital voltmeters; drift; measurements; caring for electrodes; plotting results.  
4:46 Minutes

**Electrochemical Method for Determining Solubility Products:** Preparing sample and reference solutions; setting up measuring system; taking readings; hints for obtaining good data; solubility products.  
2:34 Minutes

**Using Electrochemical Cells to Determine Thermodynamic Parameters:** Electrode potentials; assembling button cell, electrochemical circuitry; taking readings; plotting results.  
2:06 Minutes

**Using Conductivity Cells:** Components of cells; setting up equipment; measuring cell constant and conductivities of sample solutions; caring for dipping electrodes.  
3:19 Minutes

**Conductometric Titrations:** Preparing sample solutions; testing; setting up burette; taking measurements; caring for electrode; plotting results.  
6:05 Minutes

**Using an Automatic Titrator:** Background to multiple sampling techniques; setting up automatic titrator; titration; plotting results; manipulating data, derivative plots; shut-down procedure.  
3:57 Minutes

### Program PLS-116



## Gas Phase Measurements

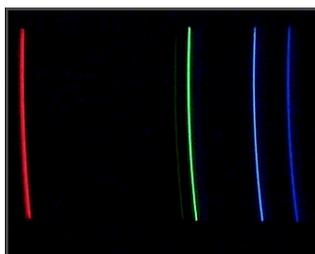
**Determination of the Molecular Weight of Gases:** Introduction and safety aspects; description of typical vacuum line; starting up vacuum line; pumping out; checking for leaks; connecting sample trap; attaching molecular weight bulb; weighing empty and filled bulbs; recovering bulb sample; shut-down procedure.  
12:12 Minutes

**Measuring the  $NO_2/N_2O_4$  Equilibrium:** Equilibrium processes; description of vacuum line; evacuation; finding null point of spiral gauge; taking measurements; shut-down procedure; plotting data.  
10:00 Minutes

**Measuring The Saturated Vapor Pressure of a Liquid at Various Temperatures:** Isotenoscope functions; evacuating system; setting up; taking measurements; suck-back; collecting data; shut-down procedure; plotting data.  
6:15 Minutes

**Using a Fortin Barometer:** Description; making adjustments; taking readings; correcting for temperature effects.  
1:28 Minutes

### Program PLS-117



## Polarimetry, Refractometry & Radiochemistry

**Using a Polarimeter:** Setting up instrument; assembling and filling sample tubes; recording null position; obtaining angular reading for sample solution; measuring time-dependent phenomena.  
8:25 Minutes

**Determination of the Refractive Index:** Abbé refractometer; set-up; calibration; measuring refractive index of a liquid sample; dark liquids; shut-down procedure; correlating refractive indices with structure and other physical data; other types of

refractometers; measuring refractive indices for solids.  
8:20 Minutes

**Measuring Rates of Radioactive Decay:** Radioactive processes; description of equipment to measure decay rates; measuring sample; interpreting data, obtaining half-life, shut-down procedure.  
6:43 Minutes

**Gas Phase Electronic Spectra:** Description of simple and constant deviation spectrometers; calibration; measuring emission and absorption spectra.  
6:10 Minutes

### Program PLS-118



## Flame Photometry, AA & TGA Measurements

**Using a Flame Photometer:** Start-up procedure; selecting filter; instrument setup; calibration; sample measurement; shut-down; partially dissolved samples; handling data.  
10:15 Minutes

**Using an Atomic Absorption Spectrometer:** Start-up procedure; lamp selection; choosing analysis wavelength; calibrating instrument; measuring sample; handling data; shut-down procedure.  
7:12 Minutes

**Thermogravimetric Analysis:** Thermal processes; instrument description; preparing crucibles; start-up procedure; doing a trial run; characteristic features of TGA traces; inspecting residues; doing an accurate run; using derivative plots; shut-down procedure.  
11:01 Minutes

### Program PLS-119



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