

THESE NOTES PROVIDE A SUMMARY OF POINTS THAT YOUR TA WILL COVER IN THE PRELAB TALK EACH WEEK. IT WILL BE HELPFUL TO HAVE THESE IN FRONT OF YOU AS THE PRELAB TALK IS PRESENTED.

DAY 1: CHECK IN.

GENERAL LAB RULES:

NEVER POUR ANYTHING EXCEPT WATER INTO THE SINKS – ALL OTHER LIQUIDS AND SOLIDS ARE CONSIDERED HAZARDOUS WASTE.

WASTE DISPOSAL:

DISPOSE OF ALL WASTE IN THE PROPER CONTAINERS. YOUR TA WILL POINT OUT THE PROPER WASTE CONTAINERS.

REGULAR TRASH: NO GLASS OR CHEMICALS – EVER.

GLASS WASTE BOX: GLASS ONLY! NO CHEMICAL WASTE OR REGULAR TRASH.

WASTE HOOD: LIQUID WASTE BOTTLE, SOLID WASTE JAR

SAFETY EQUIPMENT:

EYEWASH STATION: IF A CHEMICAL GETS INTO YOUR EYES, IMMEDIATELY ALERT YOUR TA IN A LOUD VOICE – DON'T BE SHY! HAVE NEARBY STUDENTS HELP IF NECESSARY.

YOUR TA WILL LEAD YOU TO THE EYEWASH STATION, TURN ON THE WATER, AND HELP YOU HOLD YOUR EYES IN THE FOUNTAIN FOR 15 MINUTES. YOU WILL THEN GO TO THE HEALTH CENTER. ALWAYS RINSE YOUR EYES IF YOU GET CHEMICALS IN THEM – THE DAMAGE COULD BE PERMANENT.

SAFETY SHOWER: THE SAFETY SHOWER IS USED ONLY IN EXTREME EMERGENCIES WHEN YOU HAVE SPILLED LARGE AMOUNTS OF CHEMICALS ONTO YOUR BODY.

AGAIN, ALERT YOUR TA, WHO WILL DECIDE WHETHER YOU NEED THE SAFETY SHOWER OR CAN SAFELY RINSE AT A SINK. YOU WILL NEED TO REMOVE CONTAMINATED CLOTHING. DO NOT BE SHY – YOUR HEALTH AND SAFETY IS MORE IMPORTANT THAN TEMPORARY EMBARRASSMENT. ALL STUDENTS SHOULD BE SUPPORTIVE AND RESPECTFUL OF ANYONE IN AN EMERGENCY SITUATION.

Never use the safety shower unless it is absolutely required. It will flood the floor with 55 gallons of water.

CLEANUP:

BEFORE LEAVING THE LAB EACH DAY YOU SHOULD PUT AWAY ALL YOUR EQUIPMENT AND FULLY CLEAN YOUR HOOD AND BENCH WORK AREAS. THE TA WILL CHECK THAT YOU HAVE DONE SO. FAILURE TO CLEAN YOUR AREA WILL RESULT IN LOSS OF LAB TECHNIQUE POINTS ON YOUR LAB REPORT GRADE.

MAKE SURE THAT YOUR TA SIGNS YOUR NOTEBOOK BEFORE YOU LEAVE THE LAB.

CHECK IN:

COMPARE THE EQUIPMENT IN YOUR DRAWER WITH THE APPARATUS LIST PROVIDED.

PICTURES OF THE EQUIPMENT WILL BE ON THE CENTER BENCH AND ARE ALSO POSTED ON THE WEB. THE MICROSCALE EQUIPMENT PICTURES ARE LABELED WITH LETTERS MATCHING THOSE ON THE LIST.

PLACE ANY EXTRA ITEMS INTO THE PLASTIC BIN ON THE SUPPLY BENCH.

REPLACE ANY ITEMS THAT ARE MISSING. YOUR TA WILL POINT OUT THE SPECIFIC LOCATIONS OF REPLACEMENT EQUIPMENT YOU MIGHT NEED.

LOCK YOUR DRAWER AND KEEP THE KEY FOR THE SEMESTER. YOU MAY LEAVE YOUR SAFETY GLASSES IN YOUR DRAWER IF YOU WISH TO DO SO.

BEFORE LEAVING, BE SURE TO GIVE YOUR SIGNED APPARATUS LIST TO YOUR TA.

EXP 1. DISTILLATION

CLEANUP:

AT THE END OF EACH LAB YOU SHOULD COMPLETELY CLEAN YOUR HOOD AND BENCH WORK AREAS. YOUR TA WILL CHECK THEM BEFORE SIGNING YOUR NOTEBOOK.

PUT ALL EQUIPMENT AWAY.

CLEAN YOUR BENCH TOP.

THOROUGHLY CLEAN YOUR HOOD SPACE, INCLUDING ANY SPILLED SAND.

FAILURE TO CLEAN YOUR WORK AREAS WILL RESULT IN POINTS BEING DEDUCTED FROM YOUR LAB TECHNIQUE GRADE.

DISTILLATION:

FIRST: TURN ON YOUR SAND BATH TO SETTING OF 40.

DEMONSTRATION SETUPS FOR SIMPLE AND FRACTIONAL DISTILLATION, AND OF GOOD AND BAD CONNECTORS, ARE ON THE SUPPLY BENCH.

NEVER THROW AWAY A ROUND BOTTOMED FLASK – THEY COST \$9 EACH. IF YOUR FLASK IS CONTAMINATED, PLEASE GIVE IT TO YOUR TA AND S/HE WILL REPLACE IT. WE CAN CLEAN THEM WITH A SPECIAL SOLUTION.

A DEMONSTRATION OF FRACTIONATING COLUMN PACKING IS ON THE SUPPLY BENCH.

SAVE YOUR FRACTIONAL DISTILLATION COLUMN, WITH THE COPPER MESH INSIDE AND THE COPPER WIRE FOR USE NEXT WEEK. MINIMIZE WASTE.

EMPTY YOUR UNKNOWN VIAL INTO THE LIQUID WASTE BOTTLE. RINSE THE VIAL WITH A VERY SMALL AMOUNT OF ACETONE AND LEAVE IT UNCAPPED IN THE WASTE HOOD.

WASTE:

LIQUID WASTE BOTTLE: ALL LIQUID WASTES

UNUSED OR EXCESS COPPER WIRE, COPPER SPONGE AND ALUMINUM FOIL CAN BE PUT INTO THE REGULAR TRASH – THEY ARE NOT HAZARDOUS WASTE.

EXP 2 OR 3 CYCLOHEXENE

FIRST: TURN ON YOUR SAND BATH TO SETTING OF 40.

THE CYCLOHEXANOL BOTTLES ARE AT THE BALANCES. THEY HAVE BEEN HEATED TO MELT THE CYCLOHEXANOL. CAUTION: THE BOTTLES ARE HOT!

PERFORM THE FIRST DISTILLATIONS RAPIDLY (THE REACTION AND CHASER STEPS).

PERFORM THE FINAL DISTILLATION SLOWLY AND CAREFULLY (SEPARATING THE CYCLOHEXENE AND TOLUENE).

YOUR TA WILL DEMONSTRATE THE LIQUID:LIQUID EXTRACTION TECHNIQUE USING A RED DEMONSTRATION SOLUTION AND TOLUENE. BE SURE TO USE THE COLORLESS SODIUM CHLORIDE SOLUTION FOR YOUR OWN EXTRACTION.

IF YOUR PRODUCT IS CLOUDY AFTER THE FINAL DISTILLATION, THERE IS WATER PRESENT AND YOU CANNOT RUN IT ON THE GC. GET ADVICE FROM YOUR TA.

WHEN YOU ARE READY FOR GAS CHROMATOGRAPHY ANALYSIS, SIGN UP ON THE WHITEBOARD AND WAIT FOR YOUR TA TO CALL YOU TO THE GC.

WHILE YOU ARE WAITING FOR THE GC, PERFORM THE ALKENE TESTS AND CLEAN UP YOUR EQUIPMENT AND LAB BENCH.

WASTE: LIQUID WASTE BOTTLE: ALL LIQUID WASTES

 EVAPORATING DISH: USED CALCIUM CHLORIDE PELLETS

EXP 2 OR 3: MELTING POINTS

YOUR TA WILL DEMONSTRATE THE USE OF THE MELTEMP APPARATUS.

NOTE: THE DIAL ON THE MELTEMP CONTROLS THE RATE OF HEATING, NOT THE ABSOLUTE TEMPERATURE.

YOUR TA WILL DEMONSTRATE LOADING OF THE MP CAPILLARIES.

USED CAPILLARIES ARE CONSIDERED HAZARDOUS WASTE. PLEASE DISPOSE OF THEM INTO THE LABELED EVAPORATING DISHES AT YOUR MELTEMP.

THE THREE STANDARD COMPOUNDS AND THE NINE UNKNOWN COMPOUNDS ARE IN CRYSTALLIZATION DISHES ON SUPPLY BENCH. LOAD YOUR CAPILLARIES THERE, THEN GO TO A MELTEMP. USE ANY MELTEMP THAT IS AVAILABLE – THERE ARE NO ASSIGNED SPACES FOR MPS.

MELTING POINTS FOR ALL COMPOUNDS, INCLUDING STANDARDS, ARE LISTED IN THE MELTING POINT TABLE IN YOUR HANDOUT. DO NOT USE THE MP VALUES IN REFERENCE TEXTS.

WHEN YOU ARE FINISHED FOR THE DAY, CLEAN UP YOUR AREA, INCLUDING ANY CHEMICALS SPILLED AROUND YOUR MELTEMP. ONLY THEN SHOULD YOU ASK YOUR TA TO SIGN YOUR NOTEBOOK.

EXP 4: RECRYSTALLIZATION 1

WASTE:

THERE ARE THREE SEPARATE WASTE CONTAINERS IN THE WASTE HOOD. PLEASE USE THE PROPER CONTAINERS FOR WASTE DISPOSAL.

YOUR TA WILL POINT OUT THE WASTE CONTAINERS.

LIQUID WASTE BOTTLE: ALL LIQUIDS AND LIQUID/SOLID MIXTURES.

SOLID WASTE JAR: SOLIDS ONLY. NO LIQUIDS, NO BOILING STICKS.

BOILING STICKS BOTTLE: BOILING STICKS ONLY. NO LIQUIDS.

SOLUBILITY TESTS:

LOAD SOLIDS INTO YOUR REACTION TUBES AT THE SUPPLY BENCH. USE THE SAMPLE TUBES WITH 10 mg OF SOLID AS A GUIDE. USE THAT APPROXIMATE AMOUNT – NO WEIGHING.

YOUR TA WILL DEMONSTRATE THE USE OF A SCOOPULA AND A MICRO-FUNNEL FOR LOADING REACTION TUBES.

CLEAN UP SOLID SPILLS USING THE BRUSHES AND INDEX CARDS AVAILABLE ON THE CENTER BENCH; DISPOSE OF SOLIDS IN THE SOLID WASTE CONTAINER.

TEST ONE SOLID AT A TIME USING THE THREE SOLVENTS. YOU MAY TEST THE SOLIDS IN ANY ORDER.

USE DISTILLED WATER, NOT TAP WATER.

ALWAYS USE BOILING STICKS.

PHTHALIC ACID RECRYSTALLIZATION:

REMINDER: 60 mg IS 0.060 g

YOUR TA WILL DEMONSTRATE SOLVENT REMOVAL USING THE PASTEUR PIPET METHOD.

EXP 5: RECRYSTALLIZATION 2

YOUR TA WILL DEMONSTRATE VACUUM (SUCTION) FILTRATION.

RUN SOLUBILITY TESTS ON YOUR UNKNOWN USING APPROXIMATE 10 mg SAMPLES - NO WEIGHING. DETERMINE WHICH TWO SOLVENTS ARE BEST FOR YOUR UNKNOWN.

THEN WEIGH 60 MG OF YOUR UNKNOWN INTO EACH OF TWO REACTION TUBES AND RECRYSTALLIZE WITH THE TWO SOLVENTS YOU HAVE SELECTED.

DETERMINE THE MELTING POINT OF THE CRUDE UNKNOWN DURING CLASS. THAT WILL SAVE TIME WHEN YOU COME IN LATER TO WEIGH AND MELT YOUR RECRYSTALLIZED UNKNOWN.

MORNING HOURS FOR WEIGHING AND TAKING MELTING POINTS ARE LISTED OUTSIDE THE ENTRANCE TO THE LAB IN ISB 263:

MON, TUES, WED, FRI: 10:10-12; THURS: 9:05-11

YOU MAY NOT COME IN AT ANY OTHER TIME. NOTE THAT MORNING HOURS END PROMPTLY; YOU MAY NOT STAY LATE TO FINISH A MELTING POINT. PLEASE PLAN YOUR TIME ACCORDINGLY.

EXP 6: EXTRACTION

RECALL THE LIQUID:LIQUID EXTRACTION TECHNIQUE WE USED IN THE CYCLOHEXENE LAB. MIX THE TWO LIQUID LAYERS FOR ABOUT THREE MINUTES IN A REACTION TUBE USING A PASTEUR PIPET. ALLOW THE LAYERS TO SEPARATE, AND THEN WITHDRAW THE LOWER LAYER.

KEEP TRACK OF THE COMPOUND IN EACH REACTION TUBE. DO NOT THROW AWAY ANY LIQUID UNTIL YOU ARE CERTAIN IT IS NO LONGER NEEDED.

THE EVAPORATION STEP MUST BE DONE CAREFULLY SO THAT THE AIR STREAM DOES NOT SPATTER YOUR SOLUTION AND CAUSE LOSS OF PRODUCT. CAREFULLY TURN ON THE AIR TO A LOW FLOW.

WHEN NEUTRALIZING THE BICARBONATE (CARBOXYLIC ACID) FRACTION WITH CONC. HYDROCHLORIC ACID, MIX THE CONTENTS COMPLETELY WITH YOUR GLASS ROD BEFORE CHECKING THE pH WITH INDICATOR PAPER.

EXP 7: ALCOHOL, ALDEHYDE AND KETONE UNKNOWNNS

ACETONE IS A KETONE. CONTAMINATION OF GLASSWARE WITH ACETONE WILL AFFECT SOME TESTS. IF YOU WASH GLASSWARE WITH ACETONE, DRY IT COMPLETELY BEFORE USING.

DO NOT CONTAMINATE TEST SOLUTIONS WITH YOUR PIPETS. USE THE CALIBRATED PIPETS PROVIDED WITH THE TEST SOLUTIONS. NEVER USE YOUR OWN, CONTAMINATED PIPET IN THE TEST SOLUTION BOTTLES.

WHEN PREPARING THE 2,4-DNP DERIVATIVE, MIX THE TUBE THOROUGHLY WITH YOUR GLASS ROD TO MAKE SURE THE REACTION IS COMPLETE.

THE SCHIFF'S, IODOFORM, AND CERIC NITRATE TESTS ALL SHOULD BE RUN ON THREE SAMPLES: A CONTROL (POSITIVE), A BLANK (NEGATIVE) AND YOUR UNKNOWN. COMPARE YOUR UNKNOWN RESULT WITH THE CONTROL AND BLANK RESULTS.

THE CERIC NITRATE AND IODOFORM TESTS REQUIRE 1,2-DIMETHOXYETHANE FOR UNKNOWNNS THAT ARE NOT WATER SOLUBLE; IT IS IN THE CHEMICAL SUPPLY HOOD.

WASTE:

DISCARD PIPETS USED FOR YOUR UNKNOWN SAMPLE INTO THE MARKED BIN IN THE WASTE HOOD, NOT IN THE GLASS WASTE BOX. DO NOT DISCARD YOUR PIPET BULBS.

WHEN YOU ARE FINISHED FOR THE DAY, EMPTY YOUR UNKNOWN VIAL INTO THE LIQUID WASTE. RINSE THE VIAL WITH A LITTLE ACETONE AND LEAVE IT OPEN IN THE WASTE HOOD TO DRY.

PLEASE DISPOSE OF WASTE INTO THE PROPER CONTAINERS. DO NOT THROW PASTEUR PIPETS OR BOILING STICKS INTO THE REGULAR TRASH.

EXP 8: TRIMYRISTIN

TURN YOUR SAND BATHS ON TO 30, THEN DOWN TO 20; MAKE SURE INDICATOR LIGHT IS STILL ON.

REMOVE COPPER SPONGE FROM YOUR DISTILLATION COLUMN. THIS IS A REFLUX, NOT A DISTILLATION.

BOIL CAREFULLY TO AVOID BUMPING.

USE BOILING CHIPS.

MAKE A DEPRESSION IN THE SAND.

LOWER THE FLASK SLOWLY TO JUST ABOVE THE SURFACE OF THE SAND.

BEGIN BOILING CAREFULLY.

ADJUST THE HEIGHT OF THE FLASK TO A SLOW BOIL.

YOUR TA WILL DEMONSTRATE PREPARATION OF THE PRESSURE FILTRATION ASSEMBLY.

WHEN FILTERING THE EXTRACT THROUGH THE FILTER ASSEMBLY, BE SURE TO START WITH THE TIP OF YOUR FILTER ASSEMBLY IN YOUR 25 ML FLASK- SOME LIQUID WILL DRIP THROUGH THE FILTER WITHOUT ANY PRESSURE BEING APPLIED. WEIGH THE FLASK BEFORE USING SO THAT YOU CAN EASILY DETERMINE THE AMOUNT OF CRUDE TRIMYRISTIN OBTAINED.

THE FIRST, CRUDE RECRYSTALLIZATION OF TRIMYRISTIN IS DONE IN THE SAME 25 ML FLASK. USE A BOILING STICK.

IF THE ONCE-RECRYSTALLIZED TRIMYRISTIN STILL HAS DARK AREAS, COLLECT AS CLEAN A SAMPLE AS POSSIBLE FOR THE HYDROLYSIS.

IF YOU DO NOT HAVE ENOUGH TRIMYRISTIN FOR THE FULL HYDROLYSIS, USE 30 MG INSTEAD OF 60 MG. REMEMBER TO USE HALF THE QUANTITIES OF SODIUM HYDROXIDE AND 95% ETHANOL.

EXP 9: ESTERIFICATION

YOUR TA WILL DEMONSTRATE THE PROPER USE OF A PIPET AND PIPET PUMP.

AFTER ADDING SULFURIC ACID TO YOUR REACTANTS, SWIRL THE FLASK TO MAKE SURE THE LIQUIDS ARE WELL MIXED. REMEMBER TO ADD BOILING STONES.

IT WILL TAKE MORE THAN 15 MINUTES FROM THE START OF THE SYNTHESIS FOR WATER TO SEPARATE FROM THE ORGANIC LAYER. DO NOT TIP THE APPARATUS UNTIL THERE IS A VISIBLE AMOUNT OF WATER IN THE BOTTOM CORNER OF THE SIDE ARM.

WHEN YOU TIP THE APPARATUS TO REPLACE THE UPPER (ORGANIC) LAYER FROM THE SIDE ARM INTO FLASK, BE CAREFUL NOT TO INCLUDE THE LOWER (WATER) LAYER.

PERFORM THE AQUEOUS, BICARBONATE, AND SODIUM CHLORIDE EXTRACTIONS IN YOUR 15 ML CONICAL CENTRIFUGE TUBE.

IF YOUR FINAL PRODUCT IS CLOUDY THERE IS WATER PRESENT. CONSULT WITH YOUR TA TO REMOVE THE WATER BEFORE PERFORMING INFRARED ANALYSIS.

EXP 10: LUMINOL/CHECKOUT

LUMINOL:

SYNTHESIZE LUMINOL AND PERFORM THE LIGHT REACTION. TURN IN YOUR PRELAB TO YOUR TA – THERE IS NO LAB REPORT.

CHECKOUT:

FOLLOW ALL INSTRUCTIONS ON THE BACK OF LOCKER SHEET.

ALL GLASSWARE MUST BE CLEAN; TRY TO WASH CONTAMINATED GLASSWARE. DO NOT REPLACE IT UNLESS YOU CANNOT CLEAN IT

PLACE EXTRA OR UNCLEANABLE EQUIPMENT INTO THE PLASTIC BIN ON THE CENTER BENCH.

PLACE EQUIPMENT IN THE CHECKLIST ORDER ONTO YOUR BENCH TOP; YOUR TA WILL PUT IT INTO THE DRAWER AS IT IS CHECKED

RETURN YOUR KEY. IF YOU DO NOT HAVE IT, TELL THE TA WHETHER IT IS LOST OR YOU CAN RETURN IT AT A LATER TIME. IF YOU HAVE FORGOTTEN IT, PLEASE RETURN IT TO THE LAB REPORT BOX IN THE ISB263 ENTRYWAY.

ONCE YOUR TA CLOSES YOUR DRAWER YOU HAVE NO FURTHER ACCESS TO IT.

MAKE SURE TO RETURN YOUR LOCKER SHEET TO YOUR TA.