2004 U. S. NATIONAL CHEMISTRY OLYMPIAD

NATIONAL EXAM—PART III

Prepared by the American Chemical Society Olympiad Laboratory Practical Task Force

OLYMPIAD LABORATORY PRACTICAL TASK FORCE

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DIRECTIONS TO THE EXAMINER–PART III

The laboratory practical part of the National Olympiad Examination is designed to test skills related to the laboratory. Because the format of this part of the test is quite different from the first two parts, there is a separate, detailed set of instructions for the examiner. This gives explicit directions for setting up and administering the laboratory practical.

There are two laboratory tasks to be completed during the 90 minutes allotted to this part of the test. Students do not need to stop between tasks, but are responsible for using the time in the best way possible. Each procedure must be approved for safety by the examiner before the student begins that procedure.

Part III	**2 lab problems**	**laboratory practical**	**1 hour, 30 minutes**

Students should be permitted to use non-programmable calculators.

DIRECTIONS TO THE EXAMINEE–PART III

DO NOT TURN THE PAGE UNTIL DIRECTED TO DO SO. WHEN DIRECTED, TURN TO PAGE 2 AND READ THE INTRODUCTION AND SAFETY CONSIDERATIONS CAREFULLY BEFORE YOU PROCEED.

There are two laboratory-related tasks for you to complete during the next 90 minutes. There is no need to stop between tasks or to do them in the given order. Simply proceed at your own pace from one to the other, using your time productively. You are required to have a procedure for each problem approved for safety by an examiner before you carry out any experimentation on that problem. You are permitted to use a non-programmable calculator. At the end of the 90 minutes, all answer sheets should be turned in. Be sure that you have filled in all the required information at the top of each answer sheet. Carefully follow all directions from your examiner for safety procedures and the proper disposal of chemicals at your examining site.

2004 UNITED STATES NATIONAL CHEMISTRY OLYMPIAD
PART III — LABORATORY PRACTICAL

Student Instructions

Introduction

These problems test your ability to design and carry out laboratory experiments and to draw conclusions from your experimental work. You will be graded on your experimental design, on your skills in data collection, and on the accuracy and precision of your results. Clarity of thinking and communication are also components of successful solutions to these problems, so make your written responses as clear and concise as possible.

Safety Considerations

You are required to wear approved eye protection at all times during this laboratory practical. You also must follow all directions given by your examiner for dealing with spills and with disposal of wastes.

Lab Problem 1

You have been given a vial containing either maleic acid, $C_4H_4O_4$, fumaric acid, $C_4H_4O_4$, or tartaric acid, $C_4H_6O_6$. Your lab instructor will identify the acid you have been given.

Design and carry out an experiment with the materials provided to determine the number of ionizable H^+ ions possible for each molecule of the acid given.

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Lab Problem 2

You have been given 4 (four) black pens. Design and carry out an experiment to determine whether the dye used in each pen is a *compound* or a *mixture*. You will need to provide evidence to justify your conclusions.

Answer Sheet for Laboratory Practical **Problem 1**

Student's Name: _____

Student's School:_____ **Date:** _____

Proctor's Name: _____

ACS Section Name :_____**Student's USNCO test #:** _____

1. Give a brief description of your experimental plan.

The acid you have been given is _____

Before beginning your experiment, you must get approval (for safety reasons) from the examiner.	**Examiner's Initials:**

2. Record your data and other observations.

3. Show your calculations.

4. Conclusions.

Bonus: Draw the structure for this acid.

Answer Sheet for Laboratory Practical Problem 2

Student's Name: _____

Student's School: _____ **Date:** _____

Proctor's Name: _____

ACS Section Name : _____ **Student's USNCO test #:** _____

1. Give a brief description of your experimental plan.

Before beginning your experiment, you must get approval (for safety reasons) from the examiner.	**Examiner's Initials:**

2. Record your data and other observations.

3. Conclusions.

Pen	Compound or Mixture?	Evidence
#1		
#2		
#3		
#4		

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Laboratory Practical Task Force

Examiner's Instructions

Directions to the Examiner:

Thank you for administering the 2004 USNCO laboratory practical on behalf of your Local Section. It is essential that you follow the instructions provided, in order to insure consistency of results nationwide. There may be considerable temptation to assist the students after they begin the lab exercise. It is extremely important that you do not lend any assistance or hints whatsoever to the students once they begin work. As in the international competition, the students are not allowed to speak to anyone until the activity is complete.

The equipment needed for each student for both lab exercises should be available at his/her lab station or table when the students enter the room. The equipment should be initially placed so that the materials used for Lab Problem 1 are separate from those used for Lab Problem 2.

After the students have settled, read the following instructions (in italics) to the students.

Hello, my name is _____. Welcome to the lab practical portion of the U.S. National Chemistry Olympiad Examination. In this part of the exam, we will be assessing your lab skills and your ability to reason through a laboratory problem and communicate its results. Do not touch any of the equipment in front of you until you are instructed to do so.

You will be asked to complete two laboratory problems. All the materials and equipment you may want to use to solve each problem has been set out for you and is grouped by the number of the problem. You must limit yourself to this equipment for each problem. You will have ***one hour and thirty minutes*** *to complete the* ***two problems****. You may choose to start with either problem. You are required to have a procedure for each problem approved for safety by an examiner. (Remember that approval does not mean that your procedure will be successful–it is a safety approval.) When you are ready for an examiner to come to your station for each safety approval, please raise your hand.*

Safety is an important consideration during the lab practical. ***You must wear goggles at all times.*** *Wash off any chemicals spilled on your skin or clothing with large amounts of tap water. The appropriate procedures for disposing of solutions at the end of this lab practical are:*

_____

_____

We are about to begin the lab practical. Please do not turn the page until directed to do so, but read the directions on the front page. Are there any questions before we begin?

Distribute **Part III** booklets and again remind students not to turn the page until the instruction is given. **Part III** contains student instructions and answer sheets for both laboratory problems. There is a periodic table on the last page of the booklet. Allow students enough time to read the brief cover directions.

> *Do not turn to page 2 until directed to do so. When you start to work, be sure that you fill out all information at the top of the answer sheets. Are there any additional questions?*

If there are no further questions, the students should be ready to start **Part III**.

> *You may begin.*

After **one hour and thirty minutes**, give the following directions.

> *This is the end of the lab practical. Please stop and bring me your answer sheets. Thank you for your cooperation during this test.*

Collect all the lab materials. Make sure that the student has filled in his or her name and other required information on the answer sheets. At this point, you may want to take five or ten minutes to discuss the lab practical with the students. They can learn about possible observations and interpretations and you can acquire feedback as to what they actually did and how they reacted to the problems. After this discussion, please take a few minutes to complete the Post-Exam Questionnaire; this information will be extremely useful to the Olympiad subcommittee as they prepare next year's exam.

Please remember to return the post-exam Questionnaire, the answer sheets from **Part III**, the Scantron sheets from **Part I**, and the "Blue Books" from **Part II** in the Airborne "Flight-Ready" return envelope (yellow) you were provided to this address:

ACS DivCHED Exams Institute
Department of Chemistry
University of Wisconsin – Milwaukee
3210 N Cramer Street
Milwaukee, WI 53211

The label on the envelope should have this address already, you will need only to include your return address and call Airborne (1-800-247-2676) for it to be picked up (or it can be dropped in an Airborne Express collection box). The cost of shipping will be billed to the Exams Institute. You can keep the top copy of the label to allow you to track your shipment.

Tuesday, April 20, 2004, is the ***absolute*** deadline for ***receipt*** of the exam materials at the Examinations Institute. Materials received after this deadline CANNOT be graded. Be sure to have your envelope picked up no later than April 19, 2004 for it to arrive on time.

THERE WILL BE NO EXCEPTIONS TO THIS DEADLINE DUE TO THE TIGHT SCHEDULE FOR GRADING THIS EXAMINATION.

EXAMINER'S NOTES

Lab Problem #1: Materials and Equipment

Each student should have available the following equipment and materials:

- access to electronic balance, at least 0.01 g or better precision
- 125-mL Erlenmeyer flasks
- 500-mL Erlenmeyer flask
- one 50-mL graduated cylinder
- one 10-mL graduated cylinder
- plastic spatula or plastic spoon
- thin stem marked 5-mL capacity Beral-style pipets
- waxed weighing paper, at least three sheets
- 1 glass stirring rod
- distilled water
- Supply of paper towels
- 1 pair safety goggles
- 1 lab coat or apron (optional)

Lab Problem #1: Chemicals

Each student will need:

one capped vial containing EITHER 3-5 g of maleic acid OR 3-5 g of tartaric acid. Can substitute fumaric acid for maleic acid, the *trans*-isomer, but maleic acid is preferred to fumaric acid, given its greater solubility, though perhaps less available to some.

0.250 M NaOH, approximately 350 mL in a film-covered 500-mL flask

phenolphthalein solution, 1% in ethanol, in medicine dropper *or* marked Beral-style pipet

Quick Check to be sure this lab problem will work for your examinees:

- Be sure that the caped vial of acid is labeled and the cap can be easily removed without spillage. Only the name and the molecular formula should be shown for the labeled acid, e.g. 'maleic acid, $C_4H_4O_4$' or 'tartaric acid, $C_4H_6O_6$'. DO NOT write the possible number of ionizable hydrogen atoms in front of the formula here!
- Do *not* confuse maleic acid with malic acid
- When setting up the lab station, be careful to keep the phenolphthalein (medicine dropper or pipet) away from the NaOH solution in case of possible contamination.

Lab Problem #1: Notes

Note that the examiner will need to initial each student's experimental plan. Please do not comment on the plan other than looking for any potentially unsafe practices.

Safety: It is your responsibility to ensure that all students wear safety goggles during the lab practical. A lab coat or apron for each student is desirable but not mandatory. You will also need to give students explicit directions for handling spills and for disposing of waste materials, following approved safety practices for your examining site. Please check and follow procedures appropriate for your site.

Lab Problem #2: Materials and Equipment

Each student will need:

- Access to tap water
- 4 (four) 50-mL beakers
- 4 thin stem Beral-style pipets
- at least 10 pieces of 11-cm filter paper, medium speed
- scissors
- supply of paper towels
- 1 pair safety goggles
- 1 lab coat or apron (optional)

Lab Problem #2: Chemicals

1 250-mL covered, labeled Erlenmeyer flask with approximately 200 mL distilled water, labeled 'Distilled Water'.
1 250-mL covered Erlenmeyer flask with approximately 200 mL clear ammonia solution (any commercial ammonia cleaning solution without suds is fine, but it *must* be clear), labeled 'Ammonia Solution'
4 black markers provided: Papermate® black felt-tip marker, Gelly Roll® medium black marker, Crayola® black felt marker (not the Posterboard marker), and Sanford® Sharpie marker.

IMPORTANT: During Part III, coordinators are asked to centrally locate all of the provided pens, arranged in groups of #1, #2, #3, and #4. Placing them in clearly marked groups on an available lab bench or desk is fine. Students can have access to the pens by coming to the coordinator, or briefly bringing them back to their lab station and returning them immediately when done for other students to use.

Quick Check to be sure this lab problem will work for your examinees:

1. Be sure that only #1 pens are in the #1 grouping, and so on. It is suggested that you wrap a piece of labeling tape around the pen and make a 'flag' on which the number of the pen is placed. Only the 'Sharpie' markers indicate the type of ink (permanent). It is important that students have no knowledge of the type of ink used here, so be sure when you wrap tape around the pen to indicate the pen number, that you wrap over the 'permanent marker' writing on the pen.

Lab Problem #2: Notes

1. Note that the examiner will need to initial each student's experimental plan. Please do not comment on the plan other than looking for any potentially unsafe practices.

2. Safety: It is your responsibility to ensure that all students wear safety goggles during the lab practical. A lab coat or apron for each student is desirable but not mandatory. You will also need to give students explicit directions for handling spills and for disposing of waste materials, following approved safety practices for your examining site. Please check and follow procedures appropriate for your site.

2004 U.S. NATIONAL CHEMISTRY OLYMPIAD

KEY FOR NATIONAL EXAM – PART III

Problem 1. You have been given a vial containing either maleic acid, $C_4H_4O_4$, fumaric acid, $C_4H_4O_4$, or tartaric acid, $C_4H_6O_6$. Your lab instructor will identify the acid you have been given.

Design and carry out an experiment with the materials provided to determine the number of ionizable H^+ ions possible for each molecule of the acid given.

This is a solid acid titration experiment. Students had to determine the number of moles of NaOH base present given a 0.25M solution, then perform a titration against a weighed sample of the solid maleic, fumaric, or tartaric acid. The endpoint is detected using phenolphthalein as the indicator. All of these solid organic acids have two ionizable protons.

Experimental Plan:

An experiment plan had to describe the steps needed to carry out a successful, microscale titration.

Data and Observations:

The recording of data and observations needed to include information about the mass of the acid used and the volume of the base used. Where the quantity was obtained by difference, both observations used to determine the amount should be shown. Significant figures had to be used appropriately.

Sample Calculations and Conclusions:

Students needed to provide calculations that supported their conclusions.

An example of excellent student work:

Plan:

Dissolve a pre-weighed acid sample in distilled water adding two drops phenolphthalein. Fill the 10.0 ml graduated cylinder with .25M NaOH. Add base gradually until a permanent pink color. Record total volume of base used. Repeat.

Data:

Acid used: Maleic MM=116.0 g/mol

	Trial One	Trial Two
Mass of vial + acid	4.50g	4.00 g
Mass of vial less acid	4.00 g	3.48g
Mass of acid	.50g	.52g
Volume base	34.4 ml	35.0 ml

Calculations:

Moles acid = mass acid/MM	.0043	.0045
Moles base= (liters base x M_{base})	.0086	.0088
Mole base/moles acid	2.0	2.0

Conclusion:

For maleic acid, there are two moles of ionizable protons/mol acid. It is diprotic.

An example of good student work:

Plan:

Dissolve maleic acid in water. Add phenolphthalein and titrate with .25M NaOH. Calculate.

Data:

1.462g maleic in 125 ml solution. Molarity solution = .101
Use 25 ml of solution and titrate. 19.5 ml NaOH used.

Calculations:

Mol Maleic = .0025	(Work not shown)
Mol NaOH = 19.5ml/1000ml/L(.25mol/L) = .004875	(Poor use sig. fig)
Mol NaOH/Mol Maleic = 2	(Only one trial)

Conclusion:

Maleic acid is diprotic.

Problem 2: You have been given 4 (four) black pens. Design and carry out an experiment to determine whether the dye used in each pen is a *compound* or a *mixture*. You will need to provide evidence to justify your conclusions.

This is a paper-chromatography identification experiment. Ammonia and water are provided as the carrying liquids. Students had to create an experiment using pieces of filter paper to observe a possible separation of the black ink dyes from each of the four pens provided. Situating the filter paper on or in provided beakers allowed the solvent front to rise, showing a possible separation.

Experimental Plan

The experimental plan needed to identify a way to use paper chromatography to investigate the inks in the various pens. Some detail about how to carry out the experiment – marking the filter paper above the liquid level, for example, was useful in this component of the exercise.

Observations and results:

Students needed to summarize observations about the chromatography experiment. Students who did an excellent job carried out more than one trial to verify results and included detail about separations. A table of probable observations includes,

Pens	Water	Ammonia	Conclusions
#1 Crayola ®	Separation	Separation	= a mixture
#2 Gel-Pen ®	No separation or movement	No separation	= a compound
#3 Papermate ®	Separation	Separation	= a mixture
#4 Sharpie ®	No separation	No separation	= a compound

An example of excellent student work:

Using Ammonia as solvent:

- Pen 1: Different colors appear: orange, blue, green and purple
- Pen 2: Solid black color only appears.
- Pen 2 (again): Still shows only solid black color.
- Pen 3: Different colors appear, but they are light: green, blue and purple
- Pen 3 (again): Different colors appear again, darker than previous run.
- Pen 4: Mark does not move, only black color appears.
- Pen 4 (again): Mark does not move, only black color.

Using water as solvent – retry #2 and #4.

- Pen 2: Solid black color only appears
- Pen 4: No movement observed.
- Pen 4 (again): No movement observed.

Conclusions:
Pen 1 is a mixture because different colored pigments are observed.
Pen 2 is a compound because only one color is observed even though the ink moved along the filter paper.
Pen 3 is a mixture because different colored pigments are observed. The pigments used are different than those used in Pen 1.

Pen 4 can not be determined in this experiment because no solvent was found that dissolved the ink. Based on the fact that separation of color is not observed, the ink might be a single compound.

An example of good student work:

Pen 1: Mulitple layers form. Fastest moving layer is light blue green. A darker blue-green is next and a purple color is the slowest.
Pen 2: One dark black layer that doesn't seem to move at all. One light black layer that moves slowly. One gray layer that moves faster.
Pen 3: One black layer doesn't move much. One light black layer that moves slowly. One blue-purple layer that doesn't move at all.
Pen 4: Black mark doesn't move at all and remains dark black.

Conclusion:
The pens with multiple layers in the paper chromatography were mixtures – in this case pens 1, 2 and 3. The pen with a single layer in the paper chromatography, pen 4, was a single compound.