2008 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.

2007 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 seven pipets that contain solutions of $AgNO_3$, $BaCl_2$, $Cu(NO_3)_2$, $CuSO_4$, $Pb(NO_3)_2$, KI, and $Na_2S_2O_3$, though not necessarily in this order. Using the materials provided, devise and carry out an experiment to correctly determine the contents of each pipet.

Lab Problem 2

Given a sample of an unknown metal carbonate, M_xCO_3, and 3.0M hydrochloric acid, HCl(aq), a balloon, and some laboratory equipment, devise and carry out an experiment by combining these two substances to determine the volume of the gas produced *and* the unknown metal. The possible metals are Ba, Ca, Li, or Na.

Room Temp. = 25°C, Standard Pressure = 1 atm

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.

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. Based on your observations, write the relevant equations that led to your conclusions.

4. Conclusions

Pipet	Contents	Justification
#1		
#2		
#3		
#4		
#5		
#6		
#7		

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. Calculations and Conclusions.

4. Conclusions: The volume of gas produced: [ ] The unknown metal: [ ]

5. Sources of Error in this experiment (please number):

PERIODIC TABLE OF THE ELEMENTS

1 **H** 1.008																	2 **He** 4.003
3 **Li** 6.941	4 **Be** 9.012											5 **B** 10.81	6 **C** 12.01	7 **N** 14.01	8 **O** 16.00	9 **F** 19.00	10 **Ne** 20.18
11 **Na** 22.99	12 **Mg** 24.31											13 **Al** 26.98	14 **Si** 28.09	15 **P** 30.97	16 **S** 32.07	17 **Cl** 35.45	18 **Ar** 39.95
19 **K** 39.10	20 **Ca** 40.08	21 **Sc** 44.96	22 **Ti** 47.88	23 **V** 50.94	24 **Cr** 52.00	25 **Mn** 54.94	26 **Fe** 55.85	27 **Co** 58.93	28 **Ni** 58.69	29 **Cu** 63.55	30 **Zn** 65.39	31 **Ga** 69.72	32 **Ge** 72.61	33 **As** 74.92	34 **Se** 78.96	35 **Br** 79.90	36 **Kr** 83.80
37 **Rb** 85.47	38 **Sr** 87.62	39 **Y** 88.91	40 **Zr** 91.22	41 **Nb** 92.91	42 **Mo** 95.94	43 **Tc** (98)	44 **Ru** 101.1	45 **Rh** 102.9	46 **Pd** 106.4	47 **Ag** 107.9	48 **Cd** 112.4	49 **In** 114.8	50 **Sn** 118.7	51 **Sb** 121.8	52 **Te** 127.6	53 **I** 126.9	54 **Xe** 131.3
55 **Cs** 132.9	56 **Ba** 137.3	57 **La** 138.9	72 **Hf** 178.5	73 **Ta** 181.0	74 **W** 183.8	75 **Re** 186.2	76 **Os** 190.2	77 **Ir** 192.2	78 **Pt** 195.1	79 **Au** 197.0	80 **Hg** 200.6	81 **Tl** 204.4	82 **Pb** 207.2	83 **Bi** 209.0	84 **Po** (209)	85 **At** (210)	86 **Rn** (222)
87 **Fr** (223)	88 **Ra** 226.0	89 **Ac** 227.0	104 **Rf** (261)	105 **Db** (262)	106 **Sg** (263)	107 **Bh** (262)	108 **Hs** (265)	109 **Mt** (266)	110 **Ds** (269)	111 **Rg** (272)	112 **Uub** (277)		114 **Uuq** (2??)		116 **Uuh** (2??)		118 **Uuo** (2??)

58 **Ce** 140.1	59 **Pr** 140.9	60 **Nd** 144.2	61 **Pm** (145)	62 **Sm** 150.4	63 **Eu** 152.0	64 **Gd** 157.3	65 **Tb** 158.9	66 **Dy** 162.5	67 **Ho** 164.9	68 **Er** 167.3	69 **Tm** 168.9	70 **Yb** 173.0	71 **Lu** 175.0
90 **Th** 232.0	91 **Pa** 231.0	92 **U** 238.0	93 **Np** 237.0	94 **Pu** (244)	95 **Am** (243)	96 **Cm** (247)	97 **Bk** (247)	98 **Cf** (251)	99 **Es** (252)	100 **Fm** (257)	101 **Md** (258)	102 **No** (259)	103 **Lr** (260)

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NATIONAL EXAM—PART III

Prepared by the American Chemical Society Olympiad Laboratory Practical Task Force

Examiner's Instructions

Directions to the Examiner:

Thank you for administering the 2008 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. Students are to work alone. 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. Students can use all materials for both lab problems, but each experiment is designed to work best with equipment and materials provided specifically for each lab 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, 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. There is a periodic table and constants on the last 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 UPS return envelope 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 United Parcel Service - UPS (1-800-742-5877) for it to be picked up (or it can be dropped in a UPS collection box). The cost of shipping will be billed to the Exams Institute. You can write down the tracking number on the label to allow you to track your shipment.

Wednesday, April 23, 2008, 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 21, 2008** 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 List: 2008 USNCO Lab Practical Equipment and Chemicals

Lab Problem #1: Materials and Equipment

Each student should have available the following equipment and materials:

- Clear acetate sheet
- One grease pencil, used to write in the acetate sheet
- Seven Microtip or thin-stem Beral-style pipets (approx. 2.5-mL volume) to contain unknown solutions
- One 150-mL or 250-mL beaker to hold the filled pipets
- 3-4 toothpicks for stirring
- Access to distilled water

Lab Problem #1: Chemicals

Each student will need:

- Solution of $AgNO_3$ (mw=170), $BaCl_2$ (mw=208), $Cu(NO_3)_2$ (mw=188), $CuSO_4$ (mw=160), Pb $(NO_3)_2$ (mw=331), KI (mw=166), and $Na_2S_2O_3$ (mw=158).

Notes to Coordinators:

DO NOT IDENTIFY FOR THE STUDENTS WHICH SOLUTION IS IN EACH PIPET!

- All of the solutions should be 0.10M. The solutions should be filled to the maximum in each pipet and stored upside-down (bulbs down) in the 150- or 250-mL beakers on the day of the exam. Students DO NOT have access to additional quantities of any of these solutions.
- The pipets should be labeled using an indelible marker, i.e., a Sharpie®, writing the letter in a clear, legible capital letter on the bulb portion of each pipet.
- IMPORTANT: The key for this experiment is as follows:
  $Na_2S_2O_3$ = 1
  $CuSO_4$ = 2
  KI = 3
  $AgNO_3$ = 4
  $Cu(NO_3)_2$ = 5
  $Pb(NO_3)_2$ = 6
  $BaCl_2$ = 7

Lab Problem #2: Materials and Equipment

Each student will need:

- One 50-mL beaker (to contain the 3.0 M HCl) labeled '3.0M HCl'
- One 10-mL graduated cylinder
- One balloon (Included)
- One scissors
- One metric ruler with mm precision
- One length of string approximately 30 cm (12") in length
- 2-3 sheets of waxed weighing paper
- One metal scoopula
- Access to a 0.01 or better electronic balance
- Access to distilled water

Lab Problem #2: Chemicals

- Approximately 1.5 g sample $CaCO_3$ in an unlabeled, capped 10- or 20-mL capacity vial
- 20 – 25-mL of 3.0 M HCl

Quick Check to be sure this experiment works for your examinees:

Notes to Coordinators:

- You can pour the HCl into the 50- mL beaker the day of the exam.
- The $CaCO_3$ should be powdered, not granular or in rock form. Be sure that the $CaCO_3$ vial is not labeled and is capped. For your reference, below is $CaCO_3$ "Fisher Science Education Catalog" product number:
    - S719221 Calcium Carbonate: Reagent Grade - Powder; 100g Calcium Carbonate, White, Application: CO_2 generation, $CaCO_3$ ($7.10)
    - S71922 Calcium Carbonate: Reagent Grade - Powder; 500g Calcium Carbonate, White, Application: CO_2 generation, $CaCO_3$ ($8.70)
- Please give each balloon a few stretches prior to placing at the student lab bench to ensure that the balloon will adequately inflate.

USNCO 2008 Part III Answers

Lab Problem #1

This lab problem involves knowledge of precipitation and solubility. The focus of this problem in qualitatively determining each of the seven unknown solutions is to apply knowledge and understanding of predicted reactions from observations made combining the solutions with one another using the provided acetate sheet as a spot plate.

Procedure

Students should have constructed some kind of data table that examines combinations of solutions with one another. The various color changes and formed precipitates providing evidence which students must use to form conclusions about each unknown solution.

Qualitative Evidence:
The two copper solutions are blue.
To distinguish between both blue solutions of copper sulfate and copper nitrate, the copper (II) sulfate will precipitate with barium but not with copper (II) nitrate; both copper (II) solutions will precipitate with the KI.
Copper (II) ions will form a redish-brown precipitate with iodide.
Silver nitrate forms white solid silver chloride and darkish silver sulfate.
Barium forms insoluble whitish barium sulfate and barium iodide.
Nitrates are soluble and will thus not form precipitates in combination with any of the other cations.
Lead reacts with iodide to form yellow lead iodide, with chloride to form white lead chloride, and with sulfate to form whitish lead sulfate.
Thiosulfate will react with silver to form a darkish precipitate that then dissolves on further addition of thiosulfate.

Answers: **A** = $Na_2S_2O_3$, **B** = $CuSO_4$, **C** = KI, **D** = $AgNO_3$, **E** = $Cu(NO_3)_2$, **F** = $Pb(NO_3)_2$, **G** =$BaCl_2$.

Lab Problem #2

This lab problem involves identification of an unknown metal carbonate (here, $CaCO_3$) by quantitatively reacting a measured mass of the solid sample to 3.0M HCl, capturing the CO_2 gas evolved, and determining the volume of the gas collected in the balloon provided to then calculate the molar mass of the metal carbonate and conclude which metal carbonate was present from the choices given. One experiment might have been to weigh the gas volume produced or measure the gas volume by water displacement (though materials provided were insufficient to approximate the volume produced by water displacement). Another avenue to determine the unknown metal, though not implied with the materials and chemicals provided for this lab problem, could have been to react the solid white calcium carbonate with several of the identified solutions from lab problem #1, but there is no quantitative evidence here, and the lab problem specifies not only to identify the unknown metal, *M*, but to also determine the volume of the gas produced.
Students needed to figure out a method of gas collection without the volume of $HCl_{(aq)}$ occupying the balloon. One possibility was to insert a measured mass of the solid carbonate into the balloon, then carefully pull the lip of the balloon over the top of the provided graduated cylinder (filled with the HCl. By shaking the solid carbonate into the cylinder with the balloon still over the top of the cylinder, the

two chemical reactants can combine to produce CO_2 gas to fill the balloon. One method for measuring the volume could have been to wait for the completed reaction, then carefully twist shut the balloon at its lip, tie off the balloon to make a more spherical shape, then use the string and ruler to measure the balloon's circumference to then calculate its approximate spherical volume using the formula for circumference and volume of a sphere. Students might have also measured the circumference, emptied the balloon of the products, then refilled it with water to the approximate volume when filled with CO_2, and finally measure the volume of water using the graduated cylinder. The distinguish between the possibility of Na_2CO_3 (molar mass = 106) vs. $CaCO_3$ (molar mass = 100), students might have taken a small solid sample remaining and test its relative solubility in water. Though not explicitly encouraged, students might also have tested the unknown carbonate with the known Cu (II) solutions from Experiment #1 to observe precipitates form.
Points of error abound, including assuming standard pressure (1 atm), room temperature (25 ^oC), a perfect sphere, CO_2 collected as a 'wet' gas, completed reaction, and some air and uncaptured CO_2 gas remaining in the graduated cylinder.

Sample Student Data:

General reaction: M_xCO_3 + HCl → MCl_y + $H_2O_{(l)}$ + $CO_{2(g)}$, where the mol ratio of M_xCO_3 : CO_2 is 1 : 1.

Mass of M_xCO_3 used = 1.00 g

Circumference of balloon = 23.9 cm

Radius of balloon, using C = 2πr = 3.8 cm

Calculation of volume from a sphere, $V = 4/3\pi r^3$ = 240 cm^3

Use ideal gas law, PV = nRT, to find moles of CO_2 = (1)(0.240) = n(.0821)(298), n = 0.00981

Molar mass of M_xCO_3: 0.00981 mol = 1.00g/mol. mass = 102 or close to 100, mol. mass if *M* = Ca, therefore unknown compound is $CaCO_3$.

USNCO 2008 Part III Grading Results

Lab Problem #1

Excellent Students Results:
Student provided clear explanation of procedure that included an organized and methodical plan to react solutions with each other and obtain qualitative data from which to make conclusions.
Student showed an organized data table with all possible trial combinations and complete descriptions of each reaction.
Student showed at least four relevant equations that follow a logical conclusion based on student observations. Written equations were all balanced and indicated both precipitates and aqueous ions.
Student clearly based their conclusions on chemical knowledge about solubility and color of possible precipitates. Student used deduction and analysis to infer the identity of the unknowns using the qualitative data they obtained in this experiment.

Average Student Results:
Student conducted most of the trial combinations and made use of chemical knowledge to form conclusions about the unknowns, with some incorrect and missing information.
Not all solutions correctly identified.
Written equations correctly identified precipitates but were either not completely balanced or did not correctly show aqueous ions. Only several of the relevant equations were included or the minimum were included but not al balanced correctly.
Some qualitative information was given to demonstrate an understanding of precipitate formation and solubility

Below Average Student Results:
Student was unable to apply adequate chemical knowledge from solution combinations.
Written equations were incomplete.
Little or no qualitative information was used to make predictions about the identity of the unknown solutions.

Lab Problem #2

Excellent Students Results:
Student proposed a clear, detailed procedure for determining the unknown metal by gas collection and measured gas volume.
Data was organized and calculations were clear and followed a logical plan based on the student's experiment.
A detailed and complete listing of points of error was given following the experiment.
Multiple trials were performed.
Credibly creative alternative methods to determine the unknown carbonate were used, including testing solubility and precipitation with known solutions from Exp. #1

Average Student Results:
Student made adequate measurements and had a general understanding of connection between gas collection, volume measurement, and identifying the unknown metal by determining the carbonate's molar mass.
Student incorrectly identified the unknown metal or miscalculated the volume of gas due to one or more sources of error.

Only 1-2 points of error were given following the experiment.

Below Average Student Results:

Student was not able to connect the volume of gas collected to the calculation of the unknown carbonate's molar mass.
An experiment was performed to collect the gas but both reactants were placed in the balloon together.
One or no points of error were given.