# 2003 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.

# 2003 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

Sani-Flush®, a commercial toilet bowl cleaner, contains sodium bisulfate (sodium hydrogen sulfate) and sodium carbonate as its active ingredients. Other ingredients include sodium chloride, sodium lauryl sulfate, talc and some fragrance. Given the information from the manufacturer that the sodium bisulfate is substantially in excess compared to the sodium carbonate, carry out an experiment to determine the percent by weight of the sodium carbonate in a sample of the product.

## Lab Problem 2

You have been provided with eight vials, each of which is labeled with a number from 1 to 8. Each vial contains one of the following chemicals:

$Na_3PO_4$, $NH_4Cl$, $ZnCl_2$, $KNO_3$, $Mg(OH)_2$, $Pb(NO_3)_2$, $CaCO_3$, $Na_2SO_3$.

You are allowed to use distilled water, test tubes or well plates, and only ***TWO*** additional reagents from the following choices:

6 M $H_2SO_4$, 6 M HCl, 6 M $AgNO_3$, phenolphthalein indicator solution

You must designate your choice of reagents prior to the start of your testing.

# 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.** Show your calculations.

# 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:** |

When you wish to request the optional reagents, return to the Examiner with this sheet.

| | |
|---|---|
| **I request these additional reagents:** | **Examiner's Initials:** |

**2.** Record your data and other observations.

**3.** Identify the substance in each numbered vial.

| Vial # | Contains |
|---|---|
| **1.** | |
| **2.** | |
| **3.** | |
| **4.** | |
| **5.** | |
| **6.** | |
| **7.** | |
| **8.** | |

**4.** Explain clearly how you arrived at the identity of each of the vial's contents.

# 2003 U. S. NATIONAL CHEMISTRY OLYMPIAD

## 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 2003 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.*

*One of this year's problems requires the use of a plastic syringe with a Luer-lock® tip cap.*

Show a syringe and Luer-lock® tip cap.

*This problem also requires you to use a balance, which is located _____.*

*Another of this year's problems uses small-scale chemistry equipment. Small-scale chemistry techniques help to minimize the amount of materials you use, thereby increasing safety and minimizing waste. Specialized equipment for small-scale chemistry that you will use today include Beral-type pipets and reaction plates.*

Show a 5-mL Beral-type pipet, and show a 24-well reaction plate or small test tubes.

*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** to this address:

ACS DivCHED Exams Institute
Department of Chemistry
University of Wisconsin – Milwaukee

US Postal Service:

P.O. Box 413
Milwaukee, WI 53201

FedEx or UPS:

3210 N Cramer Street
Milwaukee, WI 53211

***Tuesday, April 29, 2003***, is the ***absolute*** deadline for ***receipt*** of the exam materials at the Examinations Institute. Materials received after this deadline CANNOT be graded.

**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:

- 60-mL plastic syringe with Luer-lock® tip cap
- Small plastic cap to hold sample inside syringe
- Electronic balance, 0.01 grams. One balance can serve 3-4 students. Please do **not** substitute a milligram balance. Student processing skills, not precision, are being evaluate here.
- Scoopula or spatula
- 400-mL or 600-mL beaker
- Weighing boats or weighing paper
- Capped vial or small beaker with film covering, at least 50 mL capacity
- Easy access to sinks
- Supply of paper towels
- 1 pair safety goggles
- 1 lab coat or apron (optional)

### Lab Problem #1: Chemicals

Each student will need:

Approximately 5 grams solid sample of Sani-Flush® **Note:** Solid Sani-Flush® is sold in the cleaning supply section of most supermarkets and convenience stores.
Bottle of distilled water, at least 100 mL

Examiner access to room temperature and barometric pressure

### Lab Problem #1: Notes

1. Note that the examiner will need to initial each student's experimental plan to be sure that safety is considered.
2. You will need to provide students with the room Celsius temperature and barometric pressure (in mm Hg or atm). These data can be recorded on the board and also announced at the start of the experiment.
3. The small plastic cap can be from a soda bottle and should be able to fit with room inside the syringe without interfering with plunger operation.
4. The Sani-Flush should be in solid crystalline form.
5. Be sure that the syringe caps fit tightly over the tip of the syringe.
6. Check that the plastic bottle cap fit easily and completely inside the syringe and does the plunger slide easily when pushed.
7. It is your responsibility to ensure that students wear their 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:

- Eight vials, with caps, 10-25 mL capacity each
- 24-well plate or several spot plates or approximately fifteen 13 x 100mm test tubes
- tube rack (if test tubes are used)
- A disposal container for chemical waste (designated for heavy metal waste)
- 3-4 Beral-style pipets, ungraduated 5-mL capacity
- 400-mL beaker to hold vials
- paper towels
- easy access to sink
- one pair safety goggles
- one pair lab coat or apron (optional)

**Note:** The 24-well plates and Beral-style pipets can be purchased through Educational Innovations or Micro Mole Scientific among other vendors.

**Lab Problem #2: Chemicals**

3-4 grams each of $Na_3PO_4$, $NH_4Cl$, $ZnCl_2$, $KNO_3$, $Mg(OH)_2$, $Pb(NO_3)_2$, $CaCO_3$, and $Na_2SO_3$ in vials labeled 1 – 8.
Since this is an identification experiment, ***do not*** identify the contents of each vial! The order should be as follows:

| Vial 1 | Vial 2 | Vial 3 | Vial 4 | Vial 5 | Vial 6 | Vial 7 | Vial 8 |
|---|---|---|---|---|---|---|---|
| $NH_4Cl$ | $CaCO_3$ | $Pb(NO_3)_2$ | $Mg(OH)_2$ | $KNO_3$ | $Na_2SO_3$ | $Na_3PO_4$ | $ZnCl_2$ |

Bottle of distilled water, at least 100 mL

Additionally, the examiner will prepare 250 mL of 6 M $H_2SO_4$, 250 mL of 6 M HCl, 250 mL of 1 M $AgNO_3$, 250 mL of 0.1% phenolphthalein. It is suggested that these reagents be placed in 400 mL beakers. Fill the number of 5 mL Beral-type pipets with each reagent as the number of students present. Student may select only two of these four reagents to use in their experiment. When they request the two solutions, be sure to indicate these on their answer sheets. They may *not* change their choices once they've begun using these two solutions, nor may they use more than one pipet of each of the two solutions selected.

**Lab Problem #2: Notes**

1. Note that the examiner will need to initial each student's experimental plan to be sure that safety is considered.
2. Make sure that the vials are NOT labeled with their contents.
3. Do not let students know which reagents might be better choices to select. Remind them that they may only select one pipet of each of two of these additional reagents, without refills.
4. The hydrated form of several of these salts ($Na_3PO_4$, $ZnCl_2$) should be used, *not* the anhydrous form.
5. The phenolphthalein solution is a 0.1% solution dissolved in ethanol. Most pre-prepared solutions are the standard 0.1% solution.

# 2003 U.S. NATIONAL CHEMISTRY OLYMPIAD

## KEY FOR NATIONAL EXAM – PART III

**Problem 1.** Sani-Flush®, a commercial toilet bowl cleaner, contains sodium bisulfate and sodium carbonate as its active ingredients and other ingredients such as sodium chloride, sodium lauryl sulfate, talc and some fragrance. Given the information from the manufacturer that the sodium bisulfate is substantially in excess compared to the sodium carbonate, carry out an experiment to determine the percent by weight of the sodium carbonate in a sample of the product.

Experimental Plan:

$2HSO_4^- + CO_3^{2-} \rightarrow CO_2 + H_2O + 2SO_4^{2-}$

A good plan consisted of weighing a sample of Sani-Flush® and determining the volume of $CO_2$ produced when the sample is added to water.

For example, a good plan might include these steps,

1. Weigh approximately 1 g of Sani-Flush®.
2. Add to syringe.
3. Draw water into the syringe and cover with cap.
4. Determine the volume of gas ($CO_2$) produced.
5. Use ideal gas equation to calculate moles of $CO_2$
6. Convert moles of $CO_2$ to moles of sodium carbonate and then mass of $Na_2CO_3$
7. Divide mass of $Na_2CO_3$ by sample mass to find percentage.
8. Repeat with second sample.

An average plan was either missing one of these components or had a procedure based solely on the difference in mass. Such a procedure is subject to greater error due to the smaller change in mass relative to the change in volume.

A weak plan had minimal detail about how the experiment would be conducted.

Data and other Observations:

An example of good work on observations and data recording would be,

1.45 g sample placed in syringe.
Set plunger at 5.0 mL.
Drew in 14.0 mL of H2O.
Gas was evolved and the solution was blue.
Plunger reached 47.8 mL after the reaction was complete.

| | mass Sani-Flush® | initial plunger level | $H_2O$ volume added | final plunger level |
|---|---|---|---|---|
| Trial 1 | 2.05 g | 5.0 mL | 14.0 mL | 47.8 mL |
| Trial 2 | 1.60 g | 5.0 mL | 15.0 mL | 39.0 mL |

Temperature is 23 $^oC$ and pressure is 755 mmHg

Sample Calculations:

Sample 1: 47.8 – 14 = 33.8 mL
removing water volume: 33.8 mL – 5.0 mL = 28.8 mL gas evolved.

mole of gas: PV = nRT therefore $n = \frac{PV}{RT} = \frac{\left(\frac{755 \text{ mmHg}}{760 \text{ mmHg} \cdot \text{atm}^{-1}}\right)(0.0288 \text{ L})}{(0.0821 \text{L} \cdot \text{atm} \cdot \text{mol}^{-1} \cdot \text{K}^{-1})(296 \text{ K})}$
$= 0.00118 \text{ mol } CO_2$

convert to mass: 0.00118 mol $CO_2$ = 0.00118 mol $Na_2CO_3 \times \frac{106 \text{ g}}{1 \text{ mol}} = 0.125 \text{ g } Na_2CO_3$

calculate percentage:

$$\% = \frac{0.125 \text{ g}}{2.05 \text{ g}} \times 100 = 6.1\%$$

**Problem 2:** You have been provided with eight vials, each of which is labeled with a number from 1 to 8. Each vial contains one of the following chemicals:

$Na_3PO_4$, $NH_4Cl$, $ZnCl_2$, $KNO_3$, $Mg(OH)_2$, $Pb(NO_3)_2$, $CaCO_3$, $Na_2SO_3$.

You are allowed to use distilled water, test tubes or well plates, and only ***TWO*** additional reagents from the following choices:

6 M $H_2SO_4$, 6 M HCl, 6 M $AgNO_3$, phenolphthalein indicator solution

You must designate your choice of reagents prior to the start of your testing.

Experimental Plan
A good plan involved stating that samples of each of the eight unknowns would be placed in wells of the spot plates, distilled water would be added and observations would be made regarding the solubility of the salts. Then, each of th SELECTED reagents would be added and specific identifying tests would be detailed. It should be noted that several combinations of reagents could have been used to identify the unknowns.

For example, a good test might include these tests.

1. Place each sample into each of three wells in the spot plate. Add distilled water to each and record solubility r
2. Two of the samples will not dissolve. Add HCl to these. One sample should dissolve and the other should fiz former is magnesium hydroxide, the latter is calcium carbonate.
3. Add HCl to the other solutions. Lead (II) nitrate will form a precipitate. Sodium sulfite will release sulfur dio which can be identified by odor.
4. Add silver nitrate to the other four solutions. The sample which does not react is potassium nitrate. The yello precipitate is produced by sodium phosphate and the two white solids are zinc chloride and ammonium chlorid
5. Prepare additional solutions of the two chlorides and the identified sodium phosphate. Add the latter to the chlorides. The zinc chloride will form a precipitate while the ammonium chloride will not react.

An average plan did not detail the expected results of specific tests.

A weak plan did not include solution formation or specific results of tests.

Observations and results:

An example of good work on observations and results would be:

| Reagent | Sample 1 | Sample 2 | Sample 3 | Sample 4 | Sample 5 | Sample 6 | Sample 7 | Sample 8 |
|---|---|---|---|---|---|---|---|---|
| | | | | | | | | |
| water | soluble | insoluble | soluble | insoluble | soluble | soluble | soluble | soluble |
| HCl | No Rx | dissolves | White ppt | fizz | No Rx | Sharp odor | No Rx | No Rx |
| AgNO3 | White ppt | | | | No Rx | | Yellow Ppt | White ppt |
| Na3PO4 | No Rx | | | | | | | White ppt |