Orion Silica Reagent Instruction Sheets

to be used with the 2030 Silica Analyzer

There are three silica reagents used with the 2030 Silica Analyzer. This instruction sheet gives a list of required chemicals, equipment and detailed directions on how to prepare the reagents. Please read all instructions before beginning.

Each reagent should be made in a one month supply batch size. 5 liters of solution is enough for 1 month supply for operating the 2030 silica analyzer. The silica reagents can be made up by volume using a volumetric flask or by weight.

It is recommended to make these reagents by weight, since it is difficult to find volumetric glassware large enough to make 5 Liters. This is the most accurate method of making the reagents. In order to make the silica reagents by weight, you will need a 7 - 10 kg balance and a 6-Liter Erlenmeyer flask. DO NOT MAKE THE REAGENTS IN THE SUPPLIED RECTANGULAR BOTTLES since the chemicals will not mix properly.

The reagents should be made in a clean and dried 6-Liter or larger Erlenmeyer flask and then transferred into the supplied reagent bottles. Make sure the chemicals are all thoroughly mixed before transferring.

1.0 Equipment and Chemicals:

- 16 MΩ (or better) deionized water
- glassware for building reagents (6 L Erlenmeyer flask or two 2 L volumetric flasks)
- large stir bar and a large stir plate
- 2 kg capacity balance with 0.01g (or better) precision for weighing individual chemicals
- 7 - 10 kg capacity balance with 0.1 g (or better) precision for weighing total solution
- large funnel

Chemicals required for Silica Reagent # 1

Redistilled Sulfuric Acid	CAS # 7664-93-9
Molybdic Acid, 85% MoO_3, ACS Reagent Grade	CAS # 7782-91-4
Sodium Bisulfate Monohydrate	CAS # 10034-88-5

Chemicals required for Silica Reagent # 2

L-Tartaric Acid, ACS Reagent Grade	CAS # 87-69-4
Tween 20	CAS # 9005-64-5

Chemicals required for Silica Reagent # 3

4-Methylamino phenol sulfate, ACS Reagent Grade	CAS # 55-55-0
Potassium Metabisulfite	CAS # 16731-55-8

NOTE: Many of the chemicals used in these reagents have different isomers. It is VERY important to obtain the chemical with the correct CAS number listed above. After you receive the chemicals, double check to make sure the correct CAS number is listed on the bottle BEFORE using the chemicals to prepare the reagents.

Read all Material Safety Data Sheets before handling chemicals. Wear appropriate clothing, safety glasses, gloves, etc.

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2.0 Reagent # 1 Preparation by Weight (5 Liters)

2.1 **TOTAL WEIGHT**: 5 L of Rgt. # 1 x 1.2846 kg/liter (s.g. of Rgt. # 1) = **6.423** kg of Rgt. # 1

2.2 Obtain a clean and dry 6 Liter Erlenmeyer flask and label it.

2.3 Place the labeled flask, stirbar and any cover/top used for the flask onto a top loading balance with a 7 -10 kg capacity. Tare the balance. REMEMBER TO TARE OUT ALL ITEMS THAT WILL NOT BE IN THE FINAL TOTAL SOLUTION WEIGHT!

2.4 When the balance reads 0.0 kg, add approximately **3.0 Liters** of deionized water to the flask.

2.5 **REDISTILLED SULFURIC ACID:** *Weigh out in a well-ventilated area (fume hood)!*

2.5.1 On an analytical balance (with 0.01 g precision or better), carefully weigh out **671.50 grams** of Redistilled Sulfuric Acid.

2.5.2 Carefully and slowly add it to the flask.

2.5.3 Remove the flask from the top loading balance, cover, and place onto a stir plate and turn on the stirrer. (Record the tare weight in case the balance gets accidentally retared. _____ kg.)

2.5.4 Cover the top of the flask while the reagent is stirring.

2.5.5 Allow the solution to stir for at least 30 minutes.

2.6 **MOLYBDIC ACID:**

2.6.1 On the same analytical balance carefully weigh out **410.00 grams** of Molybdic Acid.

2.6.2 Carefully and quantitatively add it to the flask.

2.7 **SODIUM BISULFATE MONOHYDRATE:**

2.7.1 On the same analytical balance, carefully weigh out **1380.80 grams** of Sodium bisulfate monohydrate.

2.7.2 Carefully and quantitatively add it to the flask. Cover and stir for 1 hour. Remove from the stir plate and place back onto the top loading balance.

2.8 Carefully and slowly, add deionized water to the flask until the total solution weight **(6.423 kg)** is reached. Once you have reached this weight, cover and stir the solution for a minimum of 2 hours. If the chemicals have not dissolved after 2 hours of stirring, allow the solution to stir longer, overnight if necessary. Do not fill bottle until ALL the particulates have dissolved. This reagent may have a bluish tint.

2.9 When transferring the reagent, make sure there is proper ventilation. Wear protective clothing, safety glasses, gloves and face mask. Using a large funnel, transfer the reagent into the bottle labeled Reagent # 1 and screw the cap on tightly until you are ready to connect the reagents to the 2030 Silica Analyzer.

2.10 Mark the date prepared on the bottle and a disposal date of 12 months from the date of preparation.

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3.0 Reagent # 1 Preparation by Volume

3.1 If you choose to make the reagents using volumetric glassware, use the following guidelines. Use **two** 2-liter volumetric flasks for a total of 4 liters.

3.2 Thoroughly rinse out the volumetric flasks with deionized water. Label the flasks as 1A and 1B. Fill each of the flasks approximately 50 % with deionized water. Add a stir bar to each of the flasks to stir the reagent throughout the entire production process.

3.3 **Add 146 ml/2L of redistilled Sulfuric Acid** to each flask. Make sure you are working in a properly ventilated area (fume hood) when adding the acid.

3.4 Cover and stir the solutions for at least 15 minutes before proceeding onto the next step.

3.5 On an analytical balance, carefully weigh out **164.00 g/2L of Molybdic Acid.** Carefully and slowly add it to the first flask. Repeat for the second flask.

3.6 On an analytical balance, carefully weigh out **552.32 g/2L of Sodium bisulfate monohydrate**. Carefully and slowly add it to the first flask. Repeat for the second flask.

3.7 Carefully and slowly add deionized water to each of the flasks until 95% full. Cover both flasks and stir each solution for two hours. If the chemicals have not dissolved after 2 hours of stirring, allow the solution to stir longer, overnight if necessary.

3.8 Make sure that ALL the particulates have dissolved. Remove the stir bars using a magnetic extractor. Fill the volumetric flasks to the line with deionized water. Cover and invert about 5 times to make sure the solutions are mixed.

3.9 When transferring the reagent, make sure there is proper ventilation. Wear protective clothing, safety glasses, gloves and face mask. Transfer the reagents from both flasks into the bottle labeled Reagent # 1 using a large funnel. Screw the cap on tightly until you are ready to connect the reagents to the 2030 Silica Analyzer.

3.10 Mark the date prepared on the bottle and a disposal date of 12 months from the date of preparation.

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4.0 Reagent # 2 Preparation by Weight (5 Liters)

4.1 **TOTAL WEIGHT**: 5 L of Rgt. # 2 x 1.1519 kg/liter (s.g. of Rgt. # 2) = **5.760 kg** of Rgt. # 2

4.2 Obtain a clean and dry 6 Liter Erlenmeyer flask and label it.

4.3 Place the labeled flask, stirbar and any cover/top used for the flask onto a top loading balance with a 7 -10 kg capacity. Tare the balance. REMEMBER TO TARE OUT ALL ITEMS THAT WILL NOT BE IN THE FINAL TOTAL SOLUTION WEIGHT!

4.4 When the balance reads 0.0 kg, add approximately **3.0 Liters** of deionized water to the flask.

4.5 **L-TARTARIC ACID:** *Weigh out in a well-ventilated area (fume hood)!*

4.5.1 On an analytical balance (with 0.01 g precision or better), carefully weigh out **1400.13 grams** of L-Tartaric Acid.

4.5.2 Carefully and slowly add it to the flask.

4.5.3 Remove the flask from the top loading balance, cover and place onto a stir plate and turn on the stirrer. (Record the tare weight in case the balance gets accidentally retared. _____kg.)

4.5.4 Cover the top of the flask while the reagent is stirring.

4.5.5 Allow the solution to stir for at least 30 minutes.

4.6 **TWEEN 20:**

4.6.1 Carefully add 5 drops of Tween 20 to the flask using a plastic disposable pipette.

4.6.2 Cover the top of the flask while the reagent is stirring.

4.6.3 Allow the solution to stir for at least 30 minutes.

4.7 Carefully and slowly, add deionized water to the flask until the total solution weight **(5.760 kg)** is reached. Once you have reached this weight, cover and stir the solution for a minimum of 2 hours. If the chemicals have not dissolved after 2 hours of stirring, allow the solution to stir longer, overnight if necessary. Do not fill bottle until ALL the particulates have dissolved.

4.8 When transferring the reagent, make sure there is proper ventilation. Wear protective clothing, safety glasses, gloves and face mask. Using a large funnel, transfer the reagent into the bottle marked Reagent # 2. Screw the cap on tightly until you are ready to connect the reagents to the 2030 Silica Analyzer.

4.9 Mark the date prepared on the bottle and a disposal date of 12 months from the date of preparation.

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5.0 Reagent # 2 Preparation by Volume

5.1 If you choose to make the reagents using volumetric glassware, use the following guidelines. Use **two** 2-liter volumetric flasks for a total of 4 liters.

5.2 Thoroughly rinse out the volumetric flasks with deionized water. Label the flasks as 2A and 2B. Fill each of the flasks approximately 50 % with deionized water. Add a stir bar to each of the flasks to stir the reagent throughout the entire production process.

5.3 On an analytical balance, carefully weigh out **560.00 g/2L of L-Tartaric Acid**. Carefully and slowly add it to the first flask. Repeat for the second flask.

5.4 Carefully add **2 drops/2L of Tween 20,** using a plastic disposable pipette, to the first flask. Repeat for the second flask.

5.5 Carefully and slowly add deionized water to each of the flasks until 95% full. Cover and stir for 2 hours. If the chemicals have not dissolved after 2 hours of stirring, allow the solution to stir longer, overnight if necessary.

5.6 Make sure that ALL the particulates have dissolved. Remove the stir bar using a magnetic extractor. Fill the volumetric flasks to the line with deionized water. Cover and invert about 5 times to make sure the solutions are mixed.

5.7 When transferring the reagent, make sure there is proper ventilation. Wear protective clothing, safety glasses, gloves and face mask. Transfer the reagents from both flasks into the bottle labeled Reagent # 2 using a large funnel. Screw the cap on tightly until you are ready to connect the reagents to the 2030 Silica Analyzer.

5.8 Mark the date prepared on the bottle and a disposal date of 12 months from the date of preparation.

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6.0 Reagent # 3 Preparation by Weight (5 Liters)

6.1 **TOTAL WEIGHT:** 5 L of Rgt. # 3 X 1.06 kg/liter (s.g. of Rgt. # 3) = **5.30 kg of Rgt. # 3**

6.2 Obtain a clean and dry 6 Liter Erlenmeyer flask and label it.

6.3 Place the labeled flask, stirbar and any cover/top used for the flask onto a top loading balance with a 7 - 10 kg capacity. Tare the balance. REMEMBER TO TARE OUT ALL ITEMS THAT WILL NOT BE IN THE FINAL TOTAL SOLUTION WEIGHT.

6.4 When the balance reads 0.0 kg, add approximately **3.0 Liters** of deionized water to the flask.

CAUTION: THE VAPORS FROM THE CHEMICALS USED IN THIS SOLUTION CAN BE HARMFUL IF INHALED. USE EXTREME CAUTION WHEN WEIGHING AND ADDING CHEMICALS TO THE FLASK. WEAR A PROTECTIVE MOUTH MASK, GLOVES AND EYE GOGGLES. WORK IN A WELL VENTILATED AREA!

6.5 **4-METHYLAMINO PHENOL SULFATE:** *Weigh out in a well-ventilated area (fume hood)!*

6.5.1 On an analytical balance (with 0.01 g precision or better), carefully weigh out **100.01 grams** of 4-methylaminophenol sulfate.

6.5.2 Carefully and quantitatively add it to the flask.

6.5.3 Remove the flask from the top loading balance, cover and place onto a stir plate and turn on the stirrer. (Record the tare weight in case the balance gets accidentally retared. _____kg.)

6.5.4 Cover the top of the flask while the reagent is stirring.

6.5.5 Allow the solution to stir for at least 30 minutes.

6.6 **POTASSIUM METABISULFITE:**

6.6.1 On the same analytical balance, carefully weigh out **400.00 grams** of potassium metabisulfite.

6.6.2 Carefully and quantitatively add it to the flask and stir for 1 hour. Remove from the stir plate and place back into the top loading balance.

6.7 Carefully and slowly, add deionized water to the flask until the total solution weight **(5.30 kg)** is reached. Once you have reached this weight, cover and stir the solution for a minimum of 2 hours. If the chemicals have not dissolved after 2 hours of stirring, allow the solution to stir longer, overnight if necessary. Do not fill until ALL the particulates have dissolved.

6.8 When transferring the reagent, make sure there is proper ventilation. Wear protective clothing, safety glasses, gloves and face mask. Using a large funnel, transfer the reagent into the bottle marked Reagent # 3. Screw the cap on tightly until you are ready to connect the reagents to the 2030 Silica Analyzer.

6.9 Mark the date prepared on the bottle and a disposal date of 12 months from the date of preparation.

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7.0 Reagent # 3 Preparation by Volume

7.1 If you choose to make the reagents using volumetric glassware, use the following guidelines. Use **two** 2 liter volumetric flasks for a total of 4 liters.

7.2 Thoroughly rinse out the volumetric flasks with deionized water. Label the flasks as 3A and 3B. Fill each of the flasks approximately 50 % with deionized water. Add a stir bar to each of the flasks to stir the reagent throughout the entire production process.

CAUTION: THE VAPORS FROM THE CHEMICALS USED IN THIS SOLUTION CAN BE HARMFUL IF INHALED. USE EXTREME CAUTION WHEN WEIGHING AND ADDING CHEMICALS TO THE FLASK. WEAR A PROTECTIVE MOUTH MASK, GLOVES AND EYE GOGGLES. WORK IN A WELL VENTILATED AREA!

7.3 On an analytical balance, carefully weigh out **40.0 g/2L of 4-methylamino phenol sulfate.** Carefully and slowly add it to the first flask. Repeat for the second flask.

7.4 On a separate balance, carefully weigh out **160.0 g/2L of Potassium Metabisulfite**. Carefully and slowly add it to the first flask. Repeat for the second flask.

7.5 Carefully and slowly add deionized water to each of the flasks until 95% full. Cover and stir each of the solutions for two hours. If the chemicals have not dissolved after 2 hours of stirring, allow the solutions to stir longer, overnight if necessary.

7.6 Make sure that ALL of the particulates have dissolved. Remove the stir bar using a magnetic extractor. Fill the volumetric flasks to the line with deionized water. Cover and invert about 5 times to make sure the solutions are mixed.

7.7 When transferring the reagent, make sure there is proper ventilation. Wear protective clothing, safety glasses, gloves and face mask. Transfer the reagents from both flasks into the bottle labeled Reagent #3 using a large funnel. Screw the cap on tightly until you are ready to connect the reagents to the 2030 Silica Analyzer.

7.8 Mark the date prepared on the bottle and a disposal date of 12 months from the date of preparation.

Environmental Instruments
Water Analysis

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