Potassium

Method 8049 Tetraphenylborate Method
Powder Pillows (0.1 to 7.0 mg/L)

Scope and Application: For water, wastewater, and seawater

Test Preparation

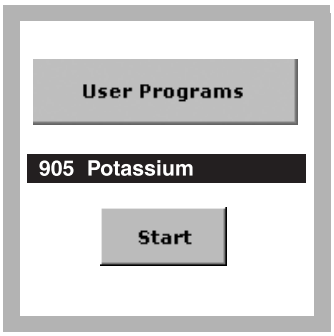
Before starting the test:

Program # 905 has a calibration curve for potassium; however, due to potential variation between lots of Potassium 3 Reagent, perform a new calibration for each lot of reagent to obtain best accuracy. Prepare and store the calibration as directed under Calibration on page 4.
Filter highly colored or turbid samples before analysis.
The final samples are highly acidic. Neutralize to pH 6–9 and flush to drain for disposal. Refer to a current MSDS for pollution prevention and waste management information.
After the test, clean the cells with soap and a brush.

Collect the following items:	**Quantity**
Potassium Reagent 1 Powder Pillow	1
Potassium Reagent 2 Powder Pillow	1
Potassium Reagent 3 Powder Pillow	1
Potassium Standard Solution, 100-mg/L	varies
Clippers	1
Cylinder, mixing, 25-mL	1
Flask, volumetric, 100-mL Class A	8
Pipet, TenSette®, 1–10 mL, plus tips	varies
Sample Cells, 1-inch square, 10-mL	2
Water, deionized	varies

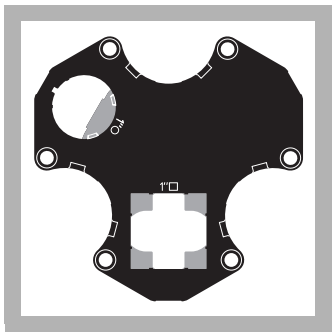
Note: *Reorder information for consumables and replacement items is on page 6.*

Powder Pillows — Method 8049



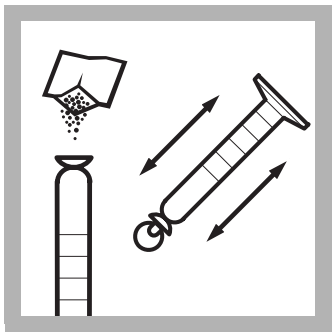
1. Select the test.

When performing this procedure for the first time the instrument must be programmed. See User Programming on page 4.

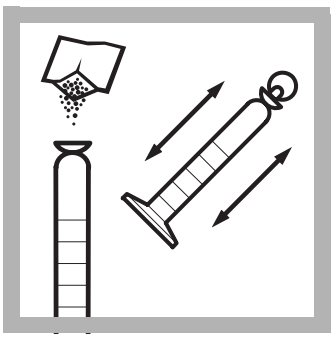


2. Insert the Multi-cell Adapter with the 1-inch square cell holder facing the user.

3. Fill a graduated mixing cylinder with 25 mL of sample.



4. Add the contents of one Potassium 1 Reagent Pillow. Add the contents of one Potassium 2 Reagent Pillow. Stopper and invert several times to mix.

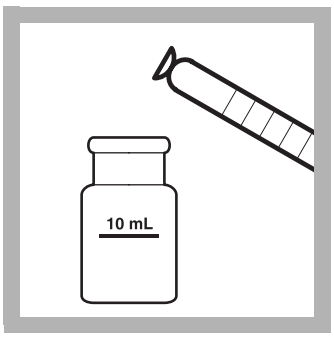


5. Add the contents of one Potassium 3 Reagent Pillow after the solution clears. Stopper and shake the solution for 30 seconds.

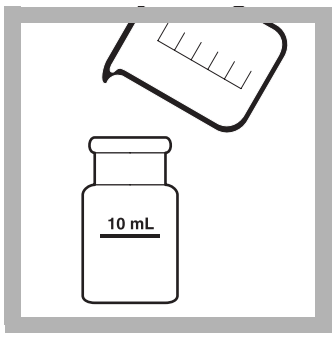
A white turbidity will form if potassium is present.

6. Press **TIMER>OK**.

A three-minute reaction period will begin.

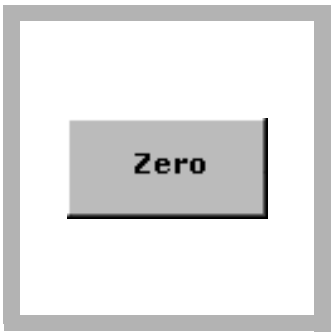


7. Prepared Sample: Pour at least 10-mL of the solution from the cylinder into a square sample cell.



8. Blank Preparation: When the timer expires, fill the second square sample cell with 10 mL of sample.

9. Wipe the blank and insert it into the cell holder with the fill line facing the user.



10. Press **ZERO**.

The display will show:

0.0 mg/L K

11. Within seven minutes after the timer expires, wipe the prepared sample and insert it into the cell holder with the fill line facing the user.

Results are in mg/L K.

Interferences

The substances listed below have been tested and will not interfere at or below the levels stated. If these substances are present at higher levels, conduct interference studies at the higher levels to determine if the substance interferes.

Table 1 Interfering Substances and Levels

Interfering Substance	Interference Levels and Treatments
Ammonium Nitrogen	15 mg/L as N
Calcium	7000 mg/L as $CaCO_3$
Chloride	15,000 mg/L
Magnesium	6000 mg/L as $CaCO_3$

Sample Collection, Preservation, and Storage

Collect samples in acid-washed plastic bottles. Adjust the pH to 2 or less with Nitric Acid (about 2 mL per liter)*. Preserved samples may be stored at least six months at room temperature. Before analysis, adjust the pH to 4–5 with 5.0 N Sodium Hydroxide*. Do not measure pH in the sample container with a pH electrode, as this will introduce potassium from the filling solution. Use pH Paper* or pour off sample and test pH in a separate beaker. Correct the test result for volume additions.

Accuracy Check

Standard Additions Method (Sample Spike)

Note: *This procedure is applicable only to Stored Program 905, and not to User Programs.*

1. After reading test results, leave the sample cell (unspiked sample) in the instrument.

2. Press **OPTIONS>MORE**. Press **STANDARD ADDITIONS**. A summary of the standard additions procedure will appear.

* See Optional Reagents and Apparatus on page 6.

3. Press **OK** to accept the default values for standard concentration, sample volume, and spike volumes. Press **EDIT** to change these values. After values are accepted, the unspiked sample reading will appear in the top row. See the user manual for more information.
4. Snap the neck off a Potassium Voluette® Ampule Standard, 250-mg/L K.
5. Prepare three sample spikes. Fill three mixing cylinders* with 25 mL of sample. Use the TenSette® Pipet to add 0.1 mL, 0.2 mL, and 0.3 mL of standard, respectively, and mix thoroughly.
6. Analyze each sample spike as described in the procedure above, starting with the 0.1 mL sample spike. Accept each standard additions reading by pressing **READ**. Each addition should reflect approximately 100% recovery.
7. After completing the sequence, press **GRAPH** to view the best-fit line through the standard additions data points, accounting for matrix interferences. Press **IDEAL LINE** to view the relationship between the sample spikes and the "Ideal Line" of 100% recovery.

Calibration

Standard Preparation

An approximate calibration curve is preprogrammed within Program 905. For improved accuracy, a new calibration should be performed with each new lot of reagents. Prepare calibration standards containing 1, 2, 3, 4, 5, 6, 7, and 8 mg/L potassium as follows:

1. Into eight different 100-mL Class A volumetric flasks, pipet 1.0, 2.0, 3.0, 4.0, 5.0, 6.0, 7.0, and 8.0 mL of the 100-mg/L Potassium Standard Solution using class A glassware or TenSette Pipet.
2. Dilute to the mark with deionized water. Mix thoroughly.
3. Use deionized water for the 0-mg/L potassium standard.

User Programming

1. Press **USER PROGRAMS** on the main menu.
2. To perform the initial potassium calibration, press **PROGRAM OPTIONS** and **NEW**. Key in any available program number (950-999). Press **OK**.
3. Use the alphanumeric keys to enter a name for the potassium test into the "Program Name?" field. Press **NEXT**.
4. Set up the rest of the parameters as follows, pressing **NEXT** to move to the next screen:
   - Program Type: Single wavelength
   - Units: mg/L
   - Wavelength λ (nm): 650
   - Concentration Resolution: 0.1
   - Chemical Form: K
   - Calibration: Read Standards
5. Press **NEXT>EXIT**.

* See Optional Reagents and Apparatus on page 6.

6. To enter the remainder of the test parameters, press each line to highlight it, press **EDIT**, then enter the value specified below. Press **OK** to accept the value, and press **OK** again to return to the list. Set up the following parameters as:
   - Upper Limit: On, 8.0
   - Lower Limit: On, -0.2
   - Timer 1: Timer 3:00
   - Press Calibration: C=a + bA >Edit>OK
7. Enter the concentrations for the calibration, starting with 0.0, in the left column. (Press **+** and enter each value, then press **OK**.)
8. When all standard concentrations have been entered, press the **UP** arrow several times to move to the 0.0 line.
9. Insert the cell containing the blank (deionized water) and press **ZERO**.
10. Perform the potassium test on each standard and insert the first prepared standard into the cell holder. Press the **DOWN** arrow, if necessary, to highlight the line corresponding to this standard concentration. Press **READ**. Repeat for each standard concentration.
11. Press **GRAPH**. If the graph is acceptable, press **DONE>EXIT**. It may be possible to obtain a better fit to the data by pressing **NEXT CURVE**. The curve which results in the highest r2 value is generally the best fit. After selection of the best curve, press **DONE>EXIT**.
12. Press **YES** in response to the “Store Program?” prompt to save the calibration.

Method Performance

Precision
Standard: 5.0 mg/L K

Program	95% Confidence Limits of Distribution
905	4.7–5.3 mg/L K

Sensitivity

Portion of Curve	ΔAbs	ΔConcentration
Entire range	0.010	0.1 mg/L K

Summary of Method

Potassium in the sample reacts with sodium tetraphenylborate to form potassium tetraphenylborate, an insoluble white solid. The amount of turbidity produced is proportional to the potassium concentration. Test results are measured at 650.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
Potassium Reagent Set:	—	—	24591-00
Potassium Reagent 1 Powder Pillow	1	25/pkg	14321-98
Potassium Reagent 2 Powder Pillow	1	25/pkg	14322-98
Potassium Reagent 3 Powder Pillow	1	100/pkg	14323-99
Potassium Standard Solution, 100-mg/L	varies	500 mL	23517-49
Water, deionized	varies	4 L	272-56

Required Apparatus

Description	Quantity/Test	Unit	Cat. No.
Clippers	1	each	968-00
Cylinder, mixing, 25-mL	1	each	1896-40
Flask, volumetric, 100-mL Class A	8	each	14574-42
Pipet, TenSette®, 1–10 mL	1	each	19700-10
Pipet Tips, for TenSette Pipet 19700-10	varies	50/pkg	21997-96
Sample Cells, 1-inch square, 10 mL, matched pair	2	2/pkg	24954-02

Recommended Standards

Description	Unit	Cat. No.
Potassium Standard Solution, 10-mL Voluette® Ampule, 250 mg/L	16/pkg	14790-10

Optional Reagents and Apparatus

Description	Unit	Cat. No.
Nitric Acid, 1:1	500 mL	2540-49
pH Paper, 1.0–11.0	—	391-33
Sodium Hydroxide, 5.0 N	50 mL	2450-26

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