Potassium

DOC316.53.01127

Tetraphenylborate Method

Method 8049

0.1 to 7.0 mg/L K

Powder Pillows

Scope and application: For water, wastewater and seawater.

Test preparation

Instrument-specific information

Table 1 shows all of the instruments that have the program for this test. The table also shows sample cell and orientation requirements for specific instruments.

To use the table, select an instrument, then read across to find the applicable information for this test.

Table 1 Instrument-specific information

Instrument	Sample cell orientation	Sample cell
DR 6000 DR 3800 DR 2800 DR 2700 DR 1900	The fill line is to the right.	2495402 
DR 5000 DR 3900	The fill line is toward the user.	

Before starting

For turbidimetric methods, install the instrument cap or cover on all instruments before ZERO or READ is pushed.
Clean sample cells with soap, water and a brush soon after each test to prevent a build-up of film on the sample cells.
Filter samples that are turbid with filter paper and a funnel.
The test results can vary with different lots of reagent. For best results, calibrate the instrument with each new lot of reagent. Refer to Calibration on page 4.
Review the Safety Data Sheets (MSDS/SDS) for the chemicals that are used. Use the recommended personal protective equipment.
Dispose of reacted solutions according to local, state and federal regulations. Refer to the Safety Data Sheets for disposal information for unused reagents. Refer to the environmental, health and safety staff for your facility and/or local regulatory agencies for further disposal information.

Items to collect

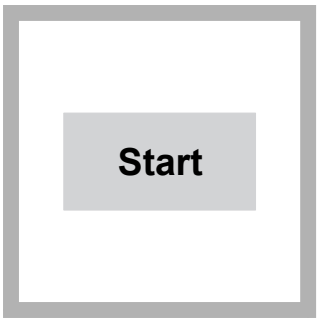
Description	Quantity
Potassium 1 Reagent Powder Pillow	1
Potassium 2 Reagent Solution Pillow	1
Potassium 3 Reagent Powder Pillow	1
Mixing cylinder, graduated, 25-mL, glass stopper	1
Clippers (shears), to open plastic pillows, stainless steel	1
Sample cells (For information about sample cells, adapters or light shields, refer to Instrument-specific information on page 1.)	2

Refer to Consumables and replacement items on page 6 for order information.

Sample collection and storage

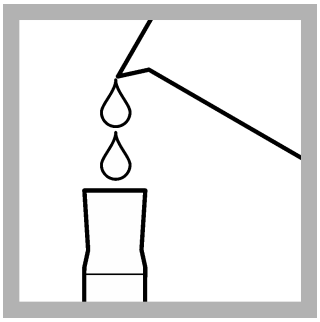
- Collect samples in clean glass or plastic bottles that have been cleaned with 6 N (1:1) hydrochloric acid and rinsed with deionized water.
- To preserve samples for later analysis, adjust the sample pH to less than 2 with concentrated nitric acid (approximately 2 mL per liter). No acid addition is necessary if the sample is tested immediately.
- Keep the preserved samples at room temperature for a maximum of 6 months.
- Before analysis, adjust the pH to 4–5 with 5 N sodium hydroxide solution.
- A pH probe can contaminate the sample. If a pH probe is used, pour a portion of sample into a separate beaker for pH measurement or use pH paper.
- Correct the test result for the dilution caused by the volume additions.

Test procedure

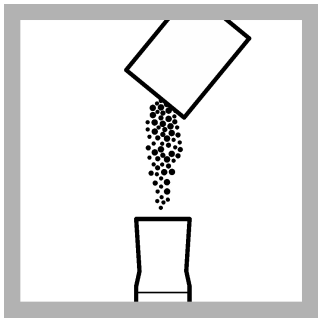


1. Start program **905 Potassium**. For information about sample cells, adapters or light shields, refer to Instrument-specific information on page 1.

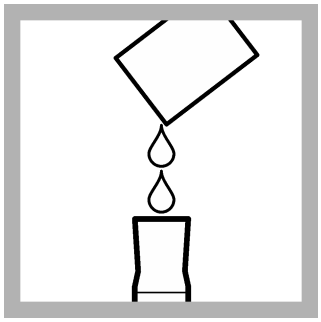
Note: *Although the program name can be different between instruments, the program number does not change.*



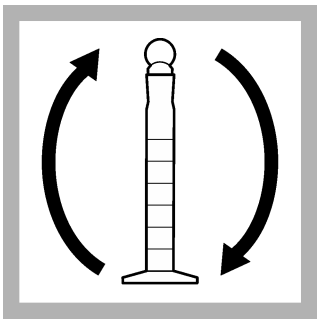
2. Prepare the sample: Fill a mixing cylinder to the 25-mL line with sample.



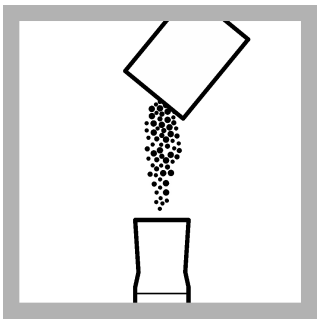
3. Add the contents of one Potassium 1 Reagent Pillow.



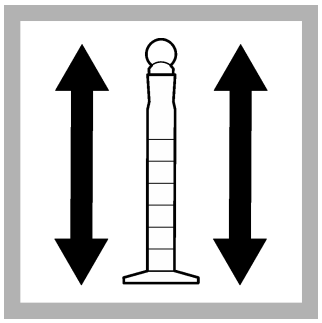
4. Add the contents of one Potassium 2 Reagent Pillow.



5. Put the stopper on the mixing cylinder. Invert the mixing cylinder several times to mix.
Let the solution become clear.

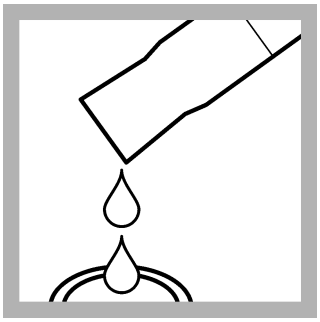


6. Add the contents of one Potassium 3 Reagent Pillow.

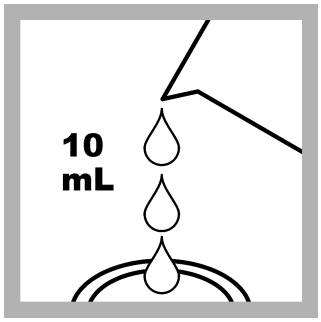


7. Put the stopper on the mixing cylinder. Shake the cylinder for 30 seconds.
A white turbidity forms if potassium is in the sample.

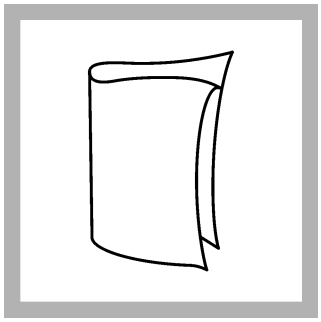
8. Start the instrument timer. A 3-minute reaction time starts.



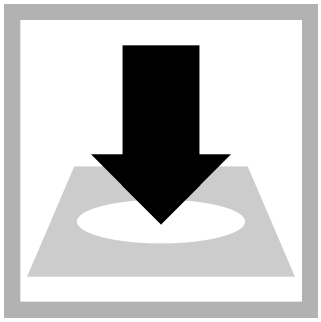
9. Pour 10 mL of the solution from the mixing cylinder into the sample cell.



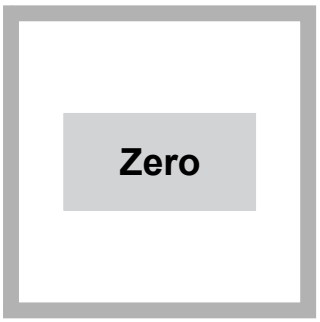
10. Prepare the blank: Fill a sample cell with 10 mL of fresh sample.



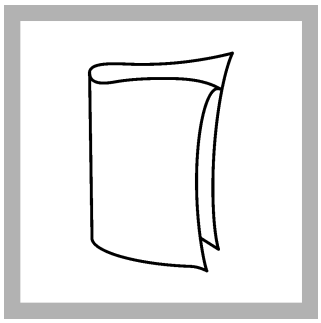
11. When the timer expires, clean the blank sample cell.



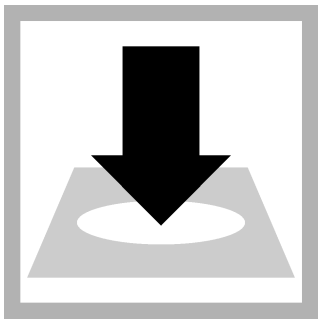
12. Insert the blank into the cell holder.



13. Push **ZERO**. The display shows 0.0 mg/L K.

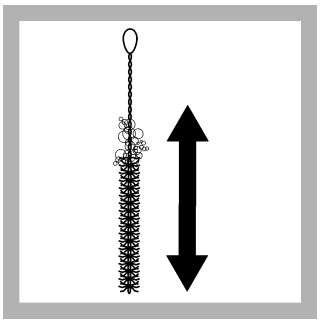


14. Clean the prepared sample cell.



15. Within 7 minutes after the timer expires, insert the prepared sample into the cell holder.

16. Push **READ**. Results show in mg/L K.



17. Immediately clean the graduated cylinder and sample cells with soapy water and a brush. Rinse with deionized water.

Interferences

Table 2 shows the substances that were tested in non-diluted samples 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 2 Interfering substances

Interfering substance	Interference level
NH_4^+–N	15 mg/L
Ca^{2+} as $CaCO_3$	7000 mg/L
Cl^-	15,000 mg/L
Mg^{2+} as $CaCO_3$	6000 mg/L

Accuracy check

Standard additions method (sample spike)

Use the standard additions method (for applicable instruments) to validate the test procedure, reagents and instrument and to find if there is an interference in the sample.

Note: *This procedure is not applicable to user programs.*

Items to collect:

- 250-mg/L Potassium Standard Solution
- 25-mL mixing cylinders, graduated (3)
- Pipet, TenSette®, 0.1–1.0 mL and tips
- Ampule breaker

1. Use the test procedure to measure the concentration of the sample, then keep the (unspiked) sample in the instrument.
2. Go to the Standard Additions option in the instrument menu.
3. Select the values for standard concentration, sample volume and spike volumes.
4. Open the standard solution.
5. Prepare three spiked samples: use the TenSette pipet to add 0.1 mL, 0.2 mL and 0.3 mL of the standard solution, respectively, to three 25-mL portions of fresh sample. Mix well.
6. Use the test procedure to measure the concentration of each of the spiked samples. Start with the smallest sample spike. Measure each of the spiked samples in the instrument.
7. Select **Graph** to compare the expected results to the actual results.

   Note: *If the actual results are significantly different from the expected results, make sure that the sample volumes and sample spikes are measured accurately. The sample volumes and sample spikes that are used should agree with the selections in the standard additions menu. If the results are not within acceptable limits, the sample may contain an interference.*

Calibration

This method requires a user-prepared calibration curve. Enter the calibration curve into the instrument as a user program. Make a new calibration curve when a new lot of reagents is used.

Prepare the standard solutions

Prepare the standard solutions for calibration as follows.

Items to collect:

- 100-mg/L Potassium Standard Solution
- 100-mL volumetric flasks (8), Class A
- 1–10 mL TenSette pipet and tips
- Deionized water

1. Prepare eight calibration standard solutions (1, 2, 3, 4, 5, 6, 7 and 8 mg/L potassium) as follows:
   a. Use a pipet to add 1, 2, 3, 4, 5, 6, 7 and 8 mL of the 100-mg/L standard solution into eight different 100-mL volumetric flasks.
   b. Dilute each flask to the mark with deionized water. Mix well.
2. Use deionized water for the 0 mg/L potassium standard.
3. Go to user programs and enter the calibration information. Refer to Enter the calibration as a user program on page 4.

Enter the calibration as a user program

After the calibration standards are prepared, make a user program to store the calibration information in the instrument. Select the user program to measure the concentration of

samples. The steps that follow are general instructions for all instruments. Refer to the user manual for the instrument that is used for the correct menu options.

1. Go to **User Programs**.
2. For the initial calibration, make a new user program. Set up the basic information for the new program:

Option	Description
Program number	Enter an available number for the user program.
Program name	Enter a name for the user program, (e.g., the name of the parameter).
Program type	Select single wavelength (for applicable spectrophotometers).

3. Enter the settings for the user program:

Option	Description
Units	mg/L
Wavelength	650 nm
Concentration resolution	0.1
Chemical form	K
Upper limit	8.0
Lower limit	–0.2
Timer 1	3:00
Calibration	Read standards

4. In the Read Standards menu, enter the concentration of the prepared standard solutions.
5. Use the test procedure to prepare the standard solutions for measurement.
6. Insert the blank solution into the cell holder and push **ZERO**.
7. Insert the first prepared standard solution into the cell holder. Make sure that the standard solution concentration is selected on the display and push **READ**.
8. Continue to measure the remaining standard solutions.
9. When all of the standard solutions have been measured, compare the graph options. Select the best curve.
10. Save (store) the user program. When the user program is selected in the test procedure, the calibration curve is used to measure the sample concentration.

Method performance

The method performance data that follows was derived from laboratory tests that were measured on a spectrophotometer during ideal test conditions. Users can get different results under different test conditions.

Program	Standard	Precision (95% confidence interval)	Sensitivity Concentration change per 0.010 Abs change
905	5.0 mg/L K	4.7–5.3 mg/L K	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. The measurement wavelength is 650 nm.

Consumables and replacement items

Required reagents

Description	Quantity/test	Unit	Item no.
Potassium Reagent Set, includes:	—	—	2459100
Potassium Reagent 1 Powder Pillow	1	25/pkg	1432198
Potassium Reagent 2 Solution Pillow	1	25/pkg	1432298
Potassium Reagent 3 Powder Pillow	1	100/pkg	1432399

Required apparatus

Description	Quantity/test	Unit	Item no.
Mixing cylinder, graduated, 25-mL, glass stopper	1	each	189640
Clippers (shears), to open plastic pillows, stainless steel	1	each	2369400

Recommended standards

Description	Unit	Item no.
Potassium Standard Solution, 10-mL Voluette® Ampule, 250 mg/L	16/pkg	1479010
Potassium Standard Solution, 100-mg/L	500 mL	2351749

Optional reagents and apparatus

Description	Unit	Item no.
Ampule Breaker, 10-mL Voluette® Ampules	each	2196800
Brush, test tube	each	69000
Flask, volumetric, Class A, 100-mL	each	1457442
Hydrochloric Acid Solution, 6.0 N (1:1)	500 mL	88449
Nitric Acid, concentrated	500 mL	15249
Paper, pH, 0–14 pH range	100/pkg	2601300
Pipet, TenSette®, 0.1–1.0 mL	each	1970001
Pipet tips for TenSette® Pipet, 0.1–1.0 mL	50/pkg	2185696
Pipet, TenSette®, 1.0–10.0 mL	each	1970010
Pipet tips for TenSette® Pipet, 1.0–10.0 mL	50/pkg	2199796
Sodium Hydroxide Solution, 5 N	50 mL	245026
Water, deionized	4 L	27256

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