

## Crystal Violet Method<sup>1</sup>

**Method 8028**
**0.002 to 0.275 mg/L as LAS (spectrophotometers)**
**0.020 to 0.300 mg/L as LAS (colorimeters)**
**Scope and application:** For water, wastewater and seawater.

<sup>1</sup> Adapted from Analytical Chemistry, 38, 791 (1966).




### Test preparation

## Instrument-specific information

[Instrument specific information PP](#) shows all of the instruments that have the program for this test. The table also shows sample cell and orientation requirements for reagent addition tests, such as powder pillow or bulk reagent tests.

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<br>DR 3800<br>DR 2800<br>DR 2700<br>DR 1900 | The fill line is to the right.           | 2612602<br>  |
| DR 5000<br>DR 3900                                  | The fill line is toward the user.        |  |
| DR 900  | The orientation mark is toward the user. | 2401906<br> |

## Before starting

Install the instrument cap on the DR 900 cell holder before ZERO or READ is pushed.

This procedure must be done in a well-ventilated area or fume hood.

Acetone can be used to clean benzene from glassware.

To prevent the formation of water droplets in the sample cells, use only dry sample cells and discard the first few mL of benzene. Additionally, it can help to transfer the liquid from the funnel to a sample cell, let it sit for a few seconds and decant to a second cell for the measurement.

Excessive shaking can cause an emulsion to form, which makes the phases separate more slowly. If this occurs, remove most of the water layer, then gently mix the contents of the funnel with a clean Teflon®-coated rod or other inert tool.

Spilled reagent will affect test accuracy and is hazardous to the skin and other materials.

For the best results, measure the reagent blank value for each new lot of reagent. Replace the sample with deionized water in the test procedure to determine the reagent blank value. Subtract the reagent blank value from the sample results automatically with the reagent blank adjust option.

In bright light conditions (e.g., direct sunlight), close the cell compartment, if applicable, with the protective cover during measurements.

Do not use the Pour-Thru Cell or sipper module (for applicable instruments) with this test.

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

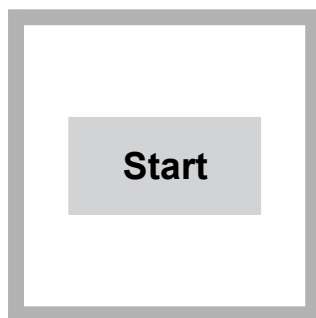
| Description   | Quantity |
|---|----------|
| Benzene, ACS  | 40 mL    |
| Buffer Solution, sulfate-type   | 10 mL    |
| Detergent Reagent Powder Pillows  | 1        |
| Clippers (for powder pillows)   | 1        |
| Cylinder, graduated, 25-mL  | 1        |
| Cylinder, graduated, 50-mL  | 1        |
| Cylinder, graduated, 500-mL   | 1        |
| Funnel, separatory, 500-mL  | 1        |
| Sample cells (For information about sample cells, adapters or light shields, refer to <a href="#">Instrument-specific information</a> on page 1.) | 2        |
| Support Ring, 4-inch  | 1        |
| Support Ring Stand, 5 x 8 inch base   | 1        |

Refer to [Consumables and replacement items](#) on page 6 for order information.

## Sample collection and storage

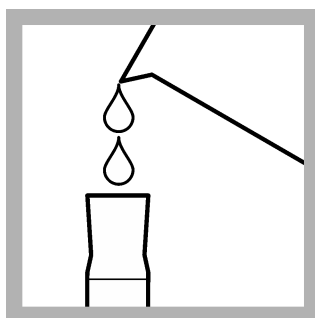
- Collect samples in clean glass or plastic bottles.
- Analyze the samples as soon as possible for best results.
- To preserve samples for later analysis, keep the samples at or below 6 °C (43 °F) for up to 48 hours.
- Let the sample temperature increase to room temperature before analysis.

## Powder pillow procedure

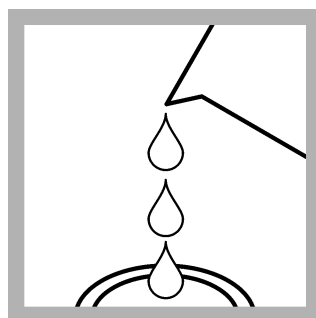


1. Start program **710 Surfactants**. For information about sample cells, adapters or light shields, refer to [Instrument-specific information](#) on page 1.

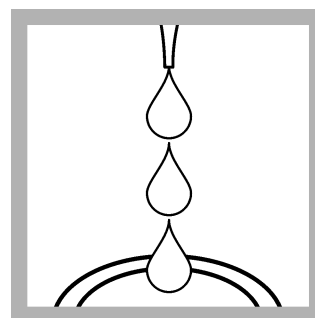
**Note:** Although the program name can be different between instruments, the program number does not change.



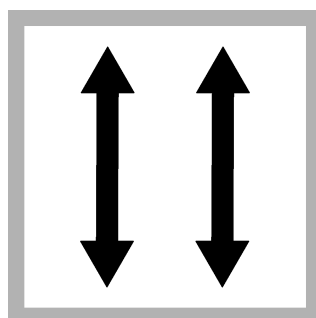
2. Fill a clean 500-mL graduated cylinder to the 300-mL mark with sample.



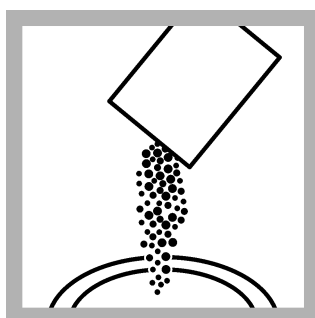
3. Pour the sample from the cylinder to a clean 500-mL separatory funnel.



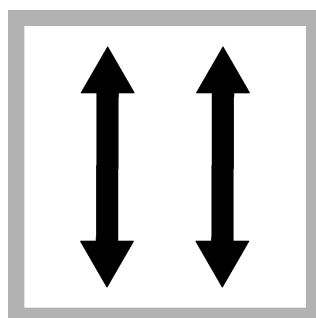
4. Add 10 mL of Sulfate Buffer Solution.



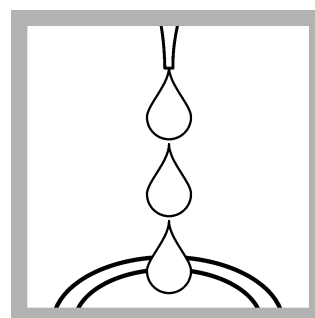
5. Close the funnel. Shake the funnel for 5 seconds



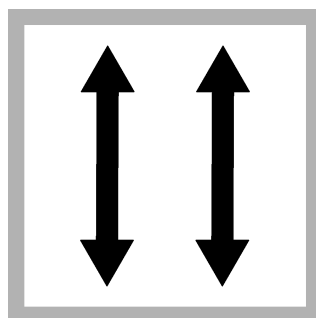
6. Add the contents of one Detergent Reagent Powder Pillow to the funnel.



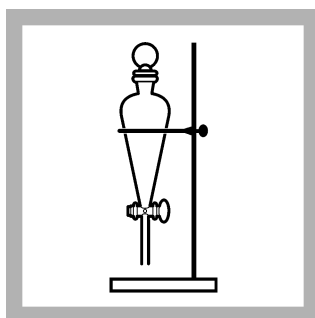
7. Close the funnel and shake until the powder dissolves completely. The powder will dissolve slowly.



8. Add 30 mL of benzene to the funnel.



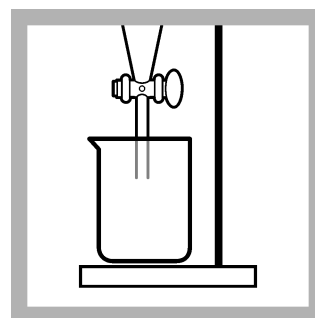
9. Close the funnel and shake gently for one minute.



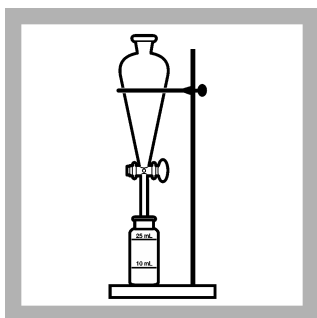
10. Put the funnel in the support stand.



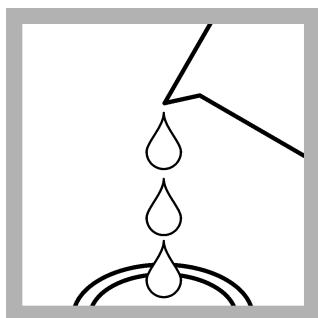
11. Start the instrument timer. A 30-minute reaction time starts.



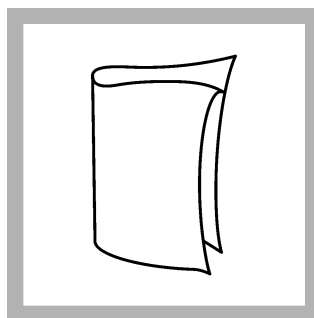
12. When the timer expires, remove the stopper and drain the bottom water layer. Collect this water for safe disposal.



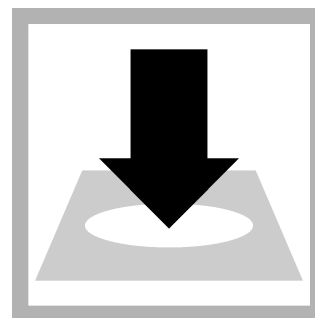
**13. Prepare the sample:** Drain the top benzene layer into a clean sample cell. Close the sample cell. Do not filter the benzene layer before color measurement. Filtration removes the blue color.



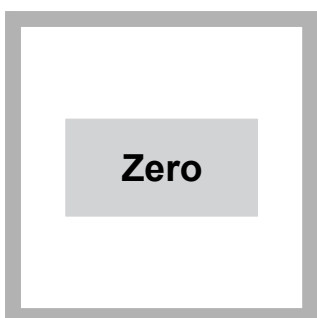
**14. Prepare the blank:** Fill another sample cell to the 10-mL mark with pure benzene. Close the sample cell.



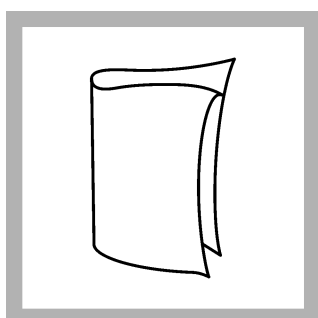
**15.** Clean the blank sample cell.



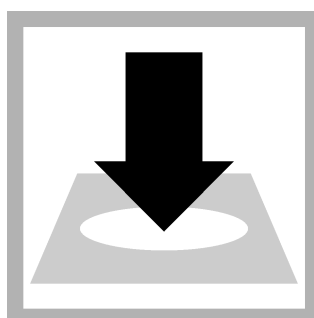
**16.** Insert the blank into the cell holder.



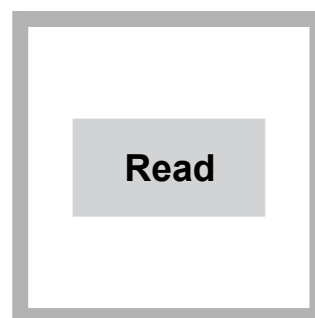
**17.** Push **ZERO**. The display shows 0.000 mg/L LAS.



**18.** Clean the prepared sample cell.



**19.** Insert the prepared sample into the cell holder.



**20.** Push **READ**. Results show in mg/L LAS.

## Interferences

| Interfering substance | Interference level   |
|-----------------------|--|
| Chloride              | High amounts of chloride, such as in brines and seawater, cause low results. |
| Perchlorate ions      | Interfere at all levels  |
| Periodate ions        | Interfere at all levels  |

## 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.

Items to collect:

- Detergent Voluette® Ampule Standard, 60-mg/L LAS
- Ampule breaker
- Pipet, TenSette®, 0.1–1.0 mL and tips

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 300-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.*

### Standard solution method

Use the standard solution method to validate the test procedure, the reagents and the instrument.

Items to collect:

- Detergent Voluette® Ampule Standard, 60 mg/L LAS
- 1-L volumetric flask, Class A
- 3-mL volumetric pipet, Class A and pipet filler safety bulb
- Deionized water

1. Prepare a 0.180 mg/L LAS standard solution as follows:
  - a. Use a pipet to add 3.0 mL of 60 mg/L LAS standard solution into the volumetric flask.
  - b. Dilute to the mark with deionized water. Mix well. Prepare this solution daily.
2. Use the test procedure to measure the concentration of the prepared standard solution.
3. Compare the expected result to the actual result.

*Note: The factory calibration can be adjusted slightly with the standard adjust option so that the instrument shows the expected value of the standard solution. The adjusted calibration is then used for all test results. This adjustment can increase the test accuracy when there are slight variations in the reagents or instruments.*

### 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<br>Concentration change per 0.010 Abs change |
|---------|----------------|-------------------------------------|--|
| 710     | 0.180 mg/L LAS | 0.172–0.188 mg/L LAS                | 0.002 mg/L LAS   |

### Summary of method

Detergents, ABS (alkyl benzene sulfonate) or LAS (linear alkylate sulfonate) are determined by association with crystal violet dye and extraction of the ion-pair complex into benzene. The measurement wavelength is 605 nm for spectrophotometers or 610 nm for colorimeters.

### Pollution prevention and waste management

Reacted samples contain benzene and must be disposed of as a hazardous waste. Dispose of reacted solutions according to local, state and federal regulations.

## Consumables and replacement items

### Required reagents

| Description                     | Quantity/test | Unit     | Item no. |
|---------------------------------|---------------|----------|----------|
| Detergents Reagent Set          | —             | —        | 2446800  |
| Includes:                       |               |          |          |
| Benzene, ACS                    | 40 mL         | 4 liters | 1444017  |
| Buffer Solution, sulfate-type   | 10 mL         | 500 mL   | 45249    |
| Detergent Reagent Powder Pillow | 1 pillow      | 25/pkg   | 100868   |

### Required apparatus

| Description                               | Quantity/test | Unit | Item no. |
|---|---------------|------|----------|
| Clippers for plastic pillows              | 1             | each | 96800    |
| Cylinder, graduated, 25-mL                | 1             | each | 50840    |
| Cylinder, graduated, 50-mL                | 1             | each | 50841    |
| Cylinder, graduated, 500-mL               | 1             | each | 50849    |
| Funnel, separatory, 500-mL                | 1             | each | 52049    |
| Support Ring, 4-inch                      | 1             | each | 58001    |
| Support, Ring Stand, 5-inch x 8-inch base | 1             | each | 56300    |

### Recommended standards

| Description  | Unit   | Item no. |
|--|--------|----------|
| Detergent Standard Solution, 10-mL Voluette <sup>®</sup> Ampule, 60-mg/L LAS | 16/pkg | 1427110  |

### Optional reagents and apparatus

| Description  | Unit   | Item no. |
|--|--------|----------|
| Acetone, ACS   | 500 mL | 1442949  |
| Beaker, 600-mL   | each   | 50052    |
| Flask, volumetric, Class A, 1000-mL glass              | each   | 1457453  |
| Pipet filler, safety bulb                              | each   | 1465100  |
| Pipet, TenSette <sup>®</sup> , 0.1–1.0 mL              | each   | 1970001  |
| Pipet tips for TenSette <sup>®</sup> Pipet, 0.1–1.0 mL | 50/pkg | 2185696  |
| Pipet, volumetric, Class A, 3-mL                       | each   | 1451503  |



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