

## MBTH Method<sup>1</sup>

**Method 8110**
**3 to 500 µg/L CH<sub>2</sub>O**
**Powder Pillows**
**Scope and application:** For water.

<sup>1</sup> Adapted from Matthews, T.G. and Howell, T.C., *Journal of the Air Pollution Control Association*, 31 (11) 1181-1184 (1981).



### 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
DR6000 DR3800 DR2800 DR2700 DR1900	The fill line is to the right.	2495402 
DR5000 DR3900	The fill line is toward the user.	

## Before starting

Analyze the samples immediately. The samples cannot be preserved for later analysis.

Time and temperature are very important to this test. The sample should be  $25 \pm 1$  °C ( $77 \pm 2$  °F) and the times specified in the procedure steps must be followed precisely. A temperature-controlled water bath is recommended for best accuracy.

Prepare formaldehyde-free water as follows: Distill water from alkaline permanganate (4 g sodium hydroxide, 2 g potassium permanganate per 500 mL of water). Discard the first 50–100 mL of distillate.

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.

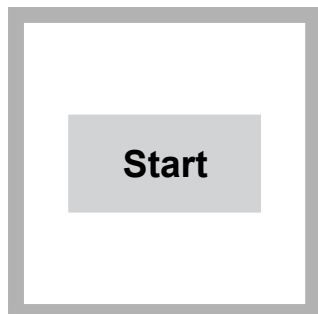
## Items to collect

Description	Quantity
Formaldehyde Reagent Set	1
Mixing cylinder, graduated, 50 mL, with stopper	2
Pipet, serological, 5 mL	1
Pipet filler, safety bulb	1
Sample cells (For information about sample cells, adapters or light shields, refer to <a href="#">Instrument-specific information</a> on page 1.)	2

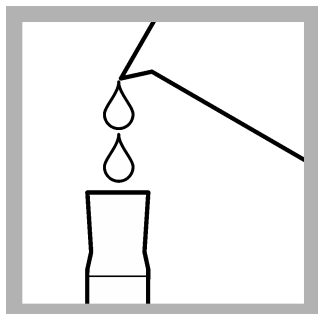
## Sample collection

- Analyze the samples immediately. The samples cannot be preserved for later analysis.
- Collect samples in clean glass or plastic bottles with tight-fitting caps. Completely fill the bottle and immediately tighten the cap.
- Prevent agitation of the sample and exposure to air.

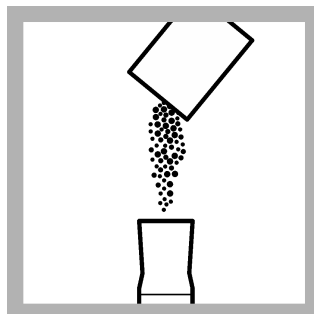
## Test procedure



1. Start program **200 Formaldehyde**. For information about sample cells, adapters or light shields, refer to [Instrument-specific information](#) on page 1.



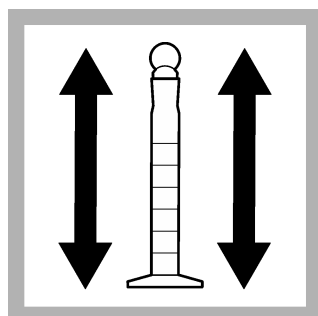
2. **Prepare the blank:** Pour 25 mL of formaldehyde-free water into a 50-mL mixing cylinder.



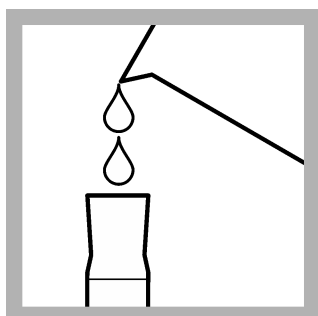
3. Add the contents of one MBTH powder pillow to the blank. Put the stopper on the mixing cylinder.



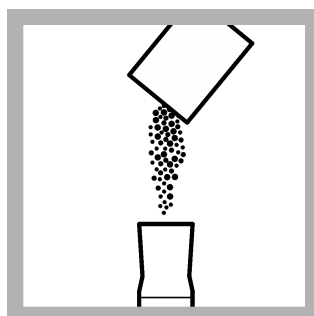
4. Start the instrument timer. A 17-minute reaction time starts. Immediately proceed with the next step after the timer starts.



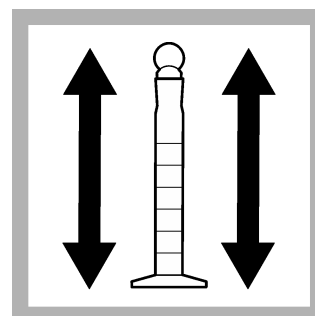
5. Immediately after the reaction period starts, shake the blank sample vigorously for 20 seconds. **Do not wait for the timer to expire.**



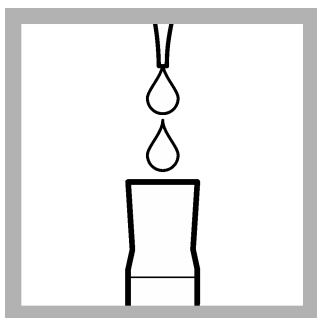
6. **Prepare the sample:** Fill a mixing cylinder to the 25-mL line with sample.



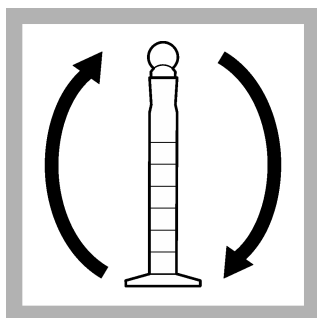
7. When the timer shows "**15:00**", add the contents of one MBTH powder pillow to the prepared sample.



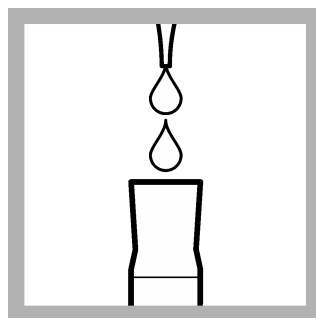
8. Put the stopper on the mixing cylinder. Shake vigorously for 20 seconds.



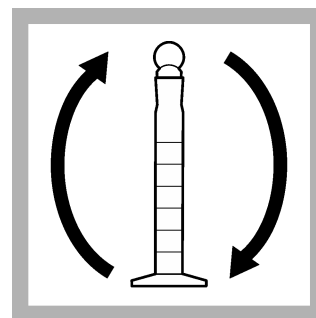
**9.** When the timer shows "12:00", use a pipet to add 2.5 mL of Developing Solution for Low Range Formaldehyde to the blank.



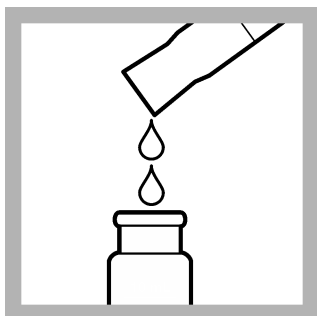
**10.** Put the stopper on the mixing cylinder. Invert the mixing cylinder several times to mix.



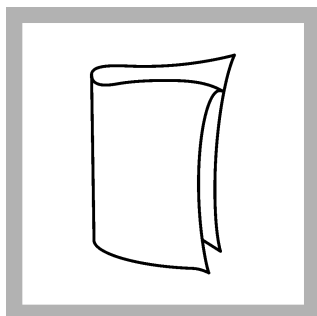
**11.** When the timer shows "10:00", use a pipet to add 2.5 mL of Developing Solution for Low Range Formaldehyde to the prepared sample.



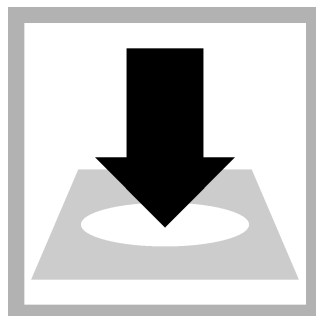
**12.** Put the stopper on the mixing cylinder. Invert the mixing cylinder several times to mix.



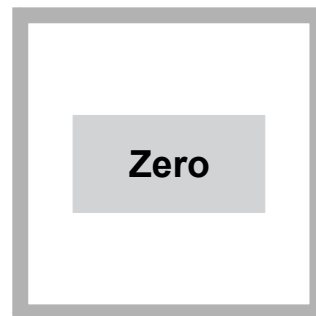
**13.** Just before the timer shows "2:00", pour 10 mL of the blank solution into a sample cell. Pour the solution slowly to prevent bubbles on the cell walls. If bubbles form, swirl to remove the bubbles.



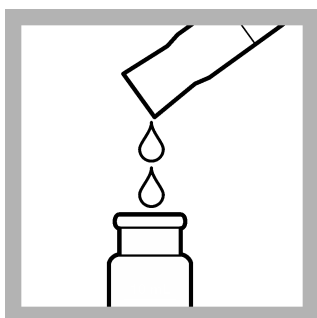
**14.** Immediately clean the blank sample cell.



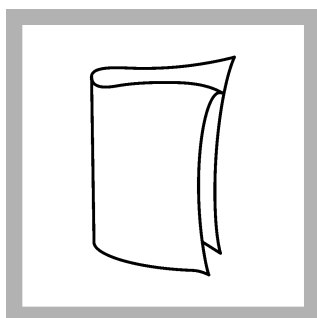
**15.** Insert the blank into the cell holder.



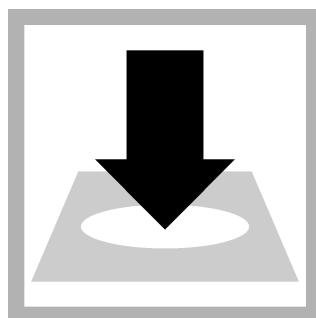
**16.** When the timer shows "2:00" push **ZERO**. The display shows 0 µg/L CH<sub>2</sub>O.



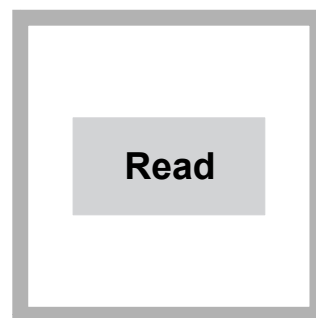
**17.** Fill a second sample cell with 10 mL of the prepared sample.



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



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



**20.** When the timer expires, push **READ**. Results show in µg/L CH<sub>2</sub>O.

## Interferences

Interfering substance	Interference level
Acetate	More than 1000 mg/L
Aldehydes (other)	Positive interference at all levels
Ammonium (as N)	More than 10 mg/L
Aniline	More than 10 mg/L

Interfering substance	Interference level
Bicarbonate	More than 1000 mg/L
Calcium	More than 3500 mg/L
Carbonate	More than 500 mg/L
Chloride	More than 5000 mg/L
Copper	More than 1.6 mg/L
Cyclohexylamine	More than 250 mg/L
Ethanolamine	More than 33 mg/L
Ethylenediamine	More than 1.5 mg/L
Glucose	More than 1000 mg/L
Glycine	More than 1000 mg/L
Iron (Fe <sup>3+</sup> )	More than 12 mg/L
Lead	More than 100 mg/L
Manganese	More than 500 mg/L
Mercury	More than 70 mg/L
Morpholine	More than 0.36 mg/L
Nitrate	More than 1000 mg/L
Nitrite	More than 8 mg/L
Phenol	More than 1050 mg/L
Phosphate	More than 200 mg/L
Silica	More than 40 mg/L
Sulfate	More than 10,000 mg/L
Urea	More than 1000 mg/L
Zinc	More than 1000 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.

Items to collect:

- 4000-mg/L Formaldehyde Standard Solution
  - 50-mL mixing cylinder, graduated (3x)
  - Flask, volumetric, Class A, 100-mL glass
  - Pipet, TenSette®, 0.1–1.0 mL and tips
  - Formaldehyde-free water
1. Prepare a 8000-µg/L (8-mg/L) formaldehyde standard solution as follows:
    - a. Use a pipet to add 0.2 mL of a 4000-mg/L formaldehyde standard solution into a 100-mL volumetric flask.
    - b. Dilute to the mark with formaldehyde-free water. Mix well. Prepare this solution daily.
  2. Use the test procedure to measure the concentration of the sample, then keep the (unspiked) sample in the instrument.
  3. Go to the Standard Additions option in the instrument menu.

4. Select the values for standard concentration, sample volume and spike volumes.
5. Prepare three spiked samples: use the TenSette pipet to add 0.1 mL, 0.2 mL and 0.3 mL of the prepared 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.*

### Standard solution method

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

Items to collect:

- 8000- $\mu\text{g/L}$  (8-mg/L) Formaldehyde Standard Solution (refer to [Standard additions method \(sample spike\)](#) on page 4 for preparation)
- 50-mL mixing cylinder, graduated
- Pipet, TenSette<sup>®</sup>, 0.1–1.0 mL and tips
- Formaldehyde-free water

1. Prepare a 320- $\mu\text{g/L}$  formaldehyde standard solution as follows:
  - a. Use a pipet to add 1.00 mL of an 8000- $\mu\text{g/L}$  formaldehyde standard solution into the mixing cylinder.
  - b. Dilute to the 25-mL mark with formaldehyde-free 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 calibration 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 small 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 Concentration change per 0.010 Abs change
200	320 $\mu\text{g/L}$ $\text{CH}_2\text{O}$	312–328 $\mu\text{g/L}$ $\text{CH}_2\text{O}$	3 $\mu\text{g/L}$ $\text{CH}_2\text{O}$

### Summary of Method

Formaldehyde reacts with MBTH (3-methyl-2-benzothiazoline hydrazone) and a developing solution to form a blue color. The color intensity is proportional to the formaldehyde concentration. The measurement wavelength is 630 nm.

## Consumables and replacement items

### Required reagents

Description	Quantity/test	Unit	Item no.
Formaldehyde Reagent Set (100 tests), includes:	—	each	2257700
Developing Solution for LR Formaldehyde	5 mL	500 mL	2257249
MBTH Powder Pillows	2	100/pkg	2257169

### Required apparatus

Description	Quantity/test	Unit	Item no.
Mixing cylinder, graduated, 50 mL, with glass stopper	1	each	189641
Pipet, serological, 5 mL	1	each	53237
Pipet filler, safety bulb	1	each	1465100

### Recommended standards

Description	Unit	Item no.
Formaldehyde Standard Solution, 10-mL Voluette® Ampule, 4000-mg/L	16/pkg	2257310

### Optional reagents and apparatus

Description	Unit	Item no.
Ampule Breaker, 10-mL Voluette® Ampules	each	2196800
Flask, volumetric, Class A, 100 mL, glass	each	1457442
Pipet, TenSette®, 0.1–1.0 mL	each	1970001
Pipet tips for TenSette® Pipet, 0.1–1.0 mL	50/pkg	2185696
Potassium Permanganate	454 g	16801H
Sodium Hydroxide, ACS	500 g	18734
Water, deionized	4 L	27256



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