

## Iron Reduction Method

**Method 8140**

3 to 450 µg/L DEHA; 5 to 600 µg/L carbohydrazide; 9 to 1000 µg/L hydroquinone; 13 to 1500 µg/L iso-ascorbic acid (ISA); 15 to 1000 µg/L methylethyl ketoxime (MEKO)

**Powder Pillows**

**Scope and application:** For testing residual corrosion inhibitors (oxygen scavengers) in boiler feed water or condensate



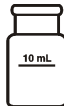
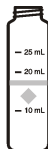
### Test preparation

## 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 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 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.	
DR 900	The orientation mark is toward the user.	2401906 

## Before starting

Samples must be analyzed immediately after collection and cannot be preserved for later analysis.

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

The sample temperature should be  $25 \pm 3$  °C ( $77 \pm 5$  °F).

Clean all glassware with 6.0 N (50%) hydrochloric acid, then rinse thoroughly with deionized water to remove iron contaminants.

To measure the ferrous iron concentration, repeat the test procedure but do not add the DEHA Reagent 2. To automatically subtract the ferrous iron concentration from the test results, use the reagent blank adjust option. Use the ferrous iron concentration as the reagent blank value.

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

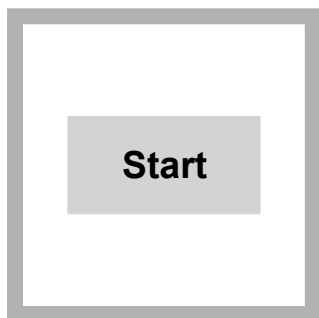
Description	Quantity
Bottle, glass mixing, with 25-mL mark	2
DEHA Reagent 1 Powder Pillows	2
DEHA Reagent 2 Solution	1 mL
Dropper, 0.5 and 1.0 mL marks	1
Hydrochloric acid, 1:1, 6.0 N	varies
Water, deionized	25 mL
Sample cells (For information about sample cells, adapters or light shields, refer to <a href="#">Instrument-specific information</a> on page 1.)	2

Refer to [Consumables and replacement items](#) on page 4 for order information.

## Sample collection

- Samples must be analyzed immediately after collection and cannot be preserved for later analysis.
- Collect samples in clean, dry glass or plastic bottles with tight-fitting caps.
- Rinse the container several times with the sample before collection.
- Prevent agitation of the sample or exposure to sunlight or air.
- Fill the bottle completely and let the sample overflow. Immediately tighten the cap so that there is no air above the sample.

## Powder pillow procedure

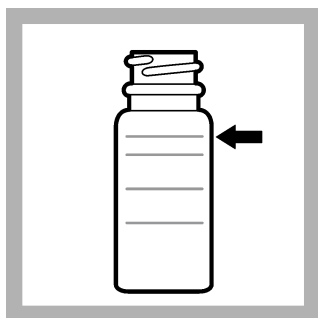


1. Start a program:

- **180 O Scav-Carbohy**
- **181 O Scav-DEHA**
- **182 O Scav-Hydro**
- **183 O Scav-ISA**
- **184 O Scav-MEKO**

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

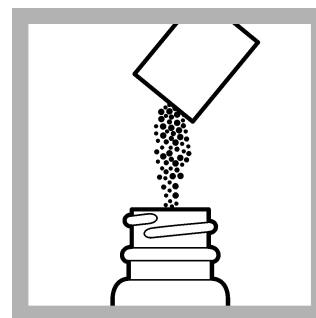
**Note:** Although the program name may vary between instruments, the program number does not change.



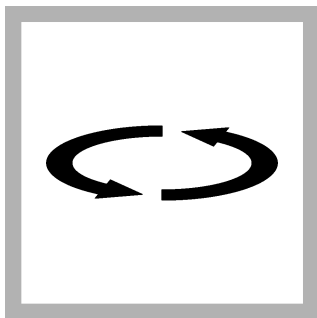
2. **Prepare the blank:** Fill a mixing bottle with 25 mL of deionized water.



3. **Prepare the sample:** Fill a second mixing bottle with 25 mL of sample. To measure oxygen scavengers that react quickly with oxygen at room temperature, close the bottle.



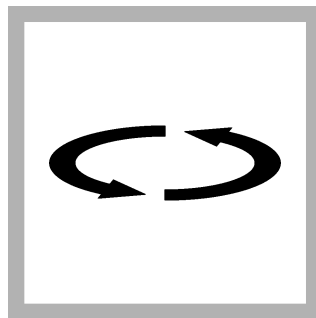
4. Add the contents of one DEHA Reagent 1 Powder Pillow to each mixing bottle.



5. Swirl to mix.



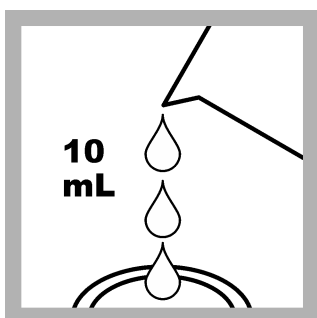
6. Use a pipet to add 0.5 mL of DEHA Reagent 2 Solution to each bottle.



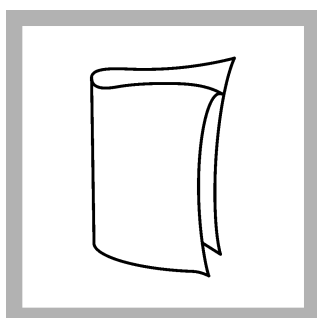
7. Swirl to mix. Put both mixing bottles in a dark location. A purple color shows if an oxygen scavenger is present in the sample.



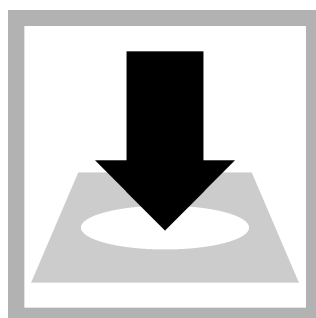
8. Start the instrument timer. A 10-minute (2-minute for hydroquinone) reaction time starts. Keep the mixing bottles in the dark during the reaction period.



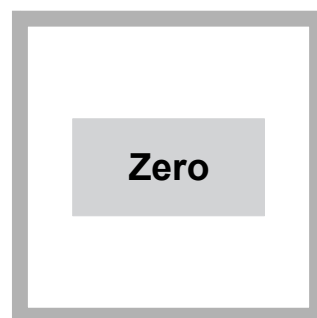
9. Complete the rest of the steps as quickly as possible. When the timer expires, immediately transfer the blank and prepared samples to the sample cells.



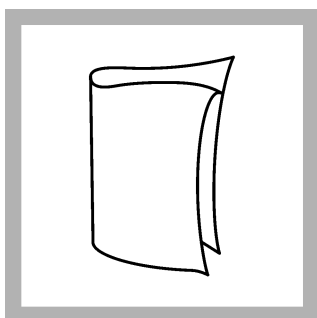
10. Clean the blank sample cell.



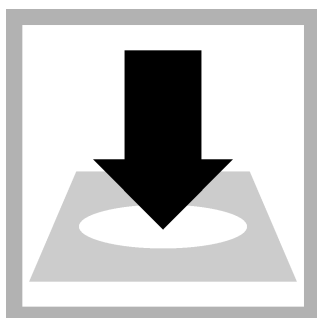
11. Insert the blank into the cell holder.



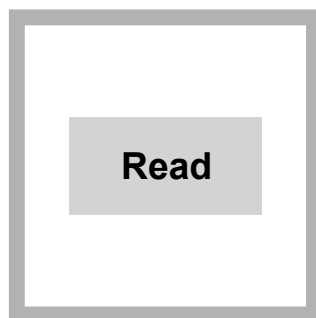
12. Push **ZERO**. The display shows 0 µg/L. For greater accuracy, read the result immediately after the timer expires.



13. Clean the prepared sample cell.



14. Insert the prepared sample into the cell holder.



15. Push **READ**. Results show in µg/L of the selected oxygen scavenger.

## Interferences

Substances which reduce ferric iron will cause a positive interference. Substances which complex iron strongly may also interfere.

Interfering substance	Interference level
Borate (as Na <sub>2</sub> B <sub>4</sub> O <sub>7</sub> )	More than 500 mg/L
Cobalt	More than 0.025 mg/L

Interfering substance	Interference level
Copper	More than 8.0 mg/L
Ferrous Iron	All levels. Measure and subtract (refer to <a href="#">Before starting</a> on page 1)
Hardness (as CaCO <sub>3</sub> )	More than 1000 mg/L
Light	Light may cause a positive interference. Keep sample cells in the dark during color development.
Lignosulfonates	More than 0.05 mg/L
Manganese	More than 0.8 mg/L
Molybdenum	More than 80 mg/L
Nickel	More than 0.8 mg/L
Phosphate	More than 10 mg/L
Phosphonates	More than 10 mg/L
Sulfate	More than 1000 mg/L
Temperature	Sample temperatures below 22 °C or above 28 °C (below 72 °F or above 82 °F) may affect test accuracy.
Zinc	More than 50 mg/L

## 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
180	299 µg/L	295–303 µg/L	4 µg/L
181	226 µg/L	223–229 µg/L	3 µg/L
182	600 µg/L	591–609 µg/L	8 µg/L
183	886 µg/L	873–899 µg/L	12 µg/L
184	976 µg/L	962–990 µg/L	14 µg/L

## Summary of method

Diethylhydroxylamine (DEHA) or other oxygen scavengers in the sample react with ferric iron in DEHA Reagent 2 Solution to produce ferrous ion in an amount that is equivalent to the DEHA concentration. This solution then reacts with DEHA 1 Reagent, which forms a purple color with ferrous iron that is proportional to the concentration of the oxygen scavenger. This method reacts with all oxygen scavengers and does not differentiate when the sample contains more than one type of oxygen scavenger. The measurement wavelength is 562 nm for spectrophotometers or 560 nm for colorimeters.

## Consumables and replacement items

### Required reagents

Description	Quantity/test	Unit	Item no.
Hydrochloric Acid Solution, 6.0 N (1:1)	varies	500 mL	88449
Water, deionized	varies	4 L	27256
Oxygen Scavenger Reagent Set	—	—	2446600

### Consumables and replacement items (continued)

Description	Quantity/test	Unit	Item no.
Includes:			
DEHA Reagent 1 Powder Pillows	2	100/pkg	2167969
DEHA Reagent 2 Solution	1 mL	100 mL	2168042

### Required apparatus

Description	Quantity/test	Unit	Item no.
Bottle, square, with 25-mL mark	2	each	1704200
Dropper, measuring, 0.5-mL and 1.0-mL plastic	1	20/pkg	2124720

### Optional apparatus

Description	Unit	Item no.
Thermometer, non-mercury, -10 to +225 °C	each	2635700



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