



Biopharmaceutical Development Program

## Standard Operating Procedure

---

### Title: HACH Free Chlorine Tests

SOP Number: 22125

Revision Number: 02

Supersedes: Revision 01

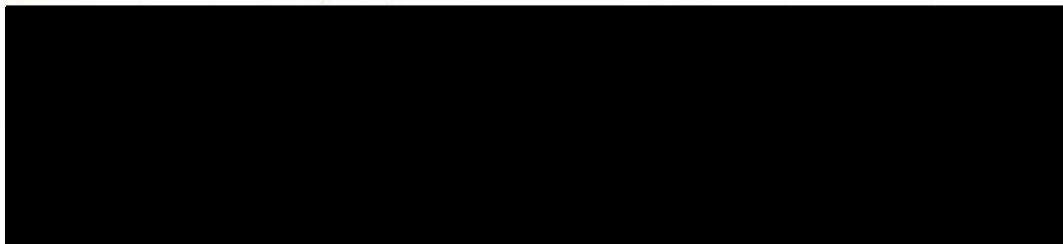
Effective Date: NOV 18 2013

---

Originator/Date:

Approval/Date:

Approval/Date:



### Table of Contents

- 1.0 Purpose
- 2.0 Scope
- 3.0 Authority and Responsibility
- 4.0 Equipment and Reagents
- 5.0 Preparations and Precautions
- 6.0 Instrument
- 7.0 Procedure
- 8.0 Calibration
- 9.0 Documentation
- 10.0 References and Related Documents
- 11.0 Attachments

#### 1.0 Purpose

This procedure describes how free chlorine concentration in a solution can be measured with the Hach Free Chlorine Test Kit.

#### 2.0 Scope

This procedure is to be performed by members of the Process Analytics (PA) lab.

#### 3.0 Authority and Responsibility

3.1 The Head, Process Analytics (PA) has the authority to define this procedure.

- 3.2 PA personnel are responsible for the implementation of this procedure.
- 3.3 Biopharmaceutical Quality Assurance (BQA) is responsible for quality oversight of this procedure.

#### 4.0 Equipment and Reagents

- 4.1 Free Chlorine Kit Using the Pocket Colorimeter Analysis System.
  - 4.1.1 DPD Free Chlorine Reagent, BDP PN 30459.
  - 4.1.2 Sample Cells, 10 mL w/ caps, Hach Catalog Number 24276-06.
  - 4.1.3 Pocket Colorimeter Instrument Kit, Chlorine, Hach Catalog Number 46700-00.
  - 4.1.4 DPD Free Chlorine Reagent Standard, 25-30 mg/L, BDP PN 30505.

#### 5.0 Preparations and Precautions

- 5.1 High concentrations of monochloramine combined with chlorine will interfere in the free chlorine determination. This does not apply to the total chlorine test.
- 5.2 Glassware should be rinsed with purified water after each test. Contamination from previous chlorine tests may cause errors in subsequent chlorine tests.
- 5.3 Failure to clean the sample cells after each test may result in a brown film on the glass surface. This can be removed with a damp, soft cloth and a mild detergent, followed by a clean-water rinse. Tubes may be cleaned best with a brush and soapy water.

**NOTE:** The chemicals in this kit may be hazardous to the health and safety of the user if inappropriately handled.

#### 6.0 Instrument

- 6.1 Do not allow liquids to enter any part of the instrument. Failure to follow these instructions can result in damage to the instrument.
- 6.2 Wipe outside of the cell dry before inserting into the instrument.
- 6.3 If the foam inside the carrying case lid becomes wet, allow it to dry before storing the instrument in a closed case.
- 6.4 Should liquids enter the instrument, thoroughly clean and dry before storing in the closed case.
- 6.5 There are four AAA batteries in the back of the pocket colorimeter. Change the batteries every six months or sooner, if the results are not consistent, by removing the plate screw on the back and replacing the batteries. Record your actions in the comments section of Form 22125-01.

## 7.0 Procedure

- 7.1 Free Chlorine Test Instructions Using the Pocket Colorimeter Kit (Range 0-2.00 mg/L).
  - 7.1.1 Rinse the sample cell w/ cap thoroughly with the water to be tested. Fill the cell to the 10 mL mark with the water sample.
  - 7.1.2 Open one DPD Free Chlorine Reagent. Add the contents to the sample cell, and cap. Shake gently for 20 seconds to mix. This is the prepared sample. Complete Steps 5.1.3 through 5.1.6 within one minute.
  - 7.1.3 Fill the other sample cell with cap with untreated sample. This is the blank.
  - 7.1.4 Insert the blank sample cell into the opening of the colorimeter with the diamond marker facing the front.
  - 7.1.5 Place the cover over the sample with the ribbed side to the rear. Press the ZERO button on the colorimeter to blank the instrument.
  - 7.1.6 Remove the blank sample and replace it with the prepared sample. Replace the cover and press the READ button. Read the value within one minute of addition of the DPD reagent.
  - 7.1.7 Report the results as free chlorine (mg/L or ppm).

## 8.0 Calibration

- 8.1 Perform the calibration verification on the first week of each month and record on Form 22125-01.
- 8.2 Obtain a DPD Free Chlorine Reagent Standard.
- 8.3 Pipet 2.00 mL of the 25-30 mg/L (ppm) chlorine standard into a 100 mL volumetric flask.
- 8.4 Dilute the standard to 100 mL with purified water quality water.
- 8.5 Proceed to test the standard following the procedure in Step 5.1.
- 8.6 The result must be 0.45 - 0.64 ppm

## 9.0 Documentation

- 9.1 Record the test results on Form 22125-01 and on the Water Test Form, or the BQC Test Request Form 22002-01, if appropriate.

## 10.0 References and Related Documents

10.1 Pocket Colorimeter Analysis System (Chlorine) Manual. HACH Company.

## 11.0 Attachments

11.1 **Attachment 1** Free Chlorine (Cl<sub>2</sub>) Assay, Form 22125-01

**Attachment 1**

NCI- Frederick  
Form No.: 22125-01  
SOP No.: 22125  
Revision 02: NOV 18 2013

**Free Chlorine Assay Form**

| <b>Assay Materials</b>    |                       |                        |
|---------------------------|-----------------------|------------------------|
|                           | <b>BDP Lot Number</b> | <b>Expiration Date</b> |
| DPD Free Chlorine Reagent |                       |                        |

| <b>Monthly Calibration</b>         |                       |                         |
|------------------------------------|-----------------------|-------------------------|
|                                    | <b>BDP Lot Number</b> | <b>Expiration Date</b>  |
| DPD Free Chlorine Reagent Standard |                       |                         |
|                                    |                       |                         |
|                                    | <b>Result (ppm)</b>   | <b>Acceptable Range</b> |
| DPD Free Chlorine Reagent Standard |                       | 0.45ppm- 0.64 ppm       |
|                                    |                       |                         |
| <b>Performed By/Date</b>           |                       |                         |

| <b>Assay Results</b> |                  |                     |
|----------------------|------------------|---------------------|
| <b>QC Number</b>     | <b>Sample ID</b> | <b>Result (ppm)</b> |
|                      |                  |                     |
|                      |                  |                     |
|                      |                  |                     |
|                      |                  |                     |
|                      |                  |                     |

|                                |
|--------------------------------|
| <b>Assay Performed By/Date</b> |
|--------------------------------|

|                 |
|-----------------|
| <b>Comments</b> |
|-----------------|

|                            |
|----------------------------|
| <b>PA Reviewed By/Date</b> |
|----------------------------|