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METHODS FOR CHEMICAL ANALYSIS OF WATER AND WASTES 1971

ENVIRONMENTAL PROTECTION AGENCY

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PREFACE

This manual describes the analytical procedures selected for use in Water Quality Office laboratories for the chemical analysis of water and waste samples. The methods were chosen by a committee of senior chemists from within the Office, using Standard Methods for the Examination of Water and Wastewater, 13th Edition (1971) and ASTM Standards, Part 23, Water; Atmospheric Analysis (1970), and current water pollution control literature as basic references. When necessary, methods derived from these sources have been modified or replaced to more adequately meet the needs of the Office.

In order to provide reliable water quality and waste constituent data for use by the Office, these procedures will be used in all Office laboratories except under very unusual circumstances. Other agencies and individuals are encouraged to use these methods, in the interest of uniformity throughout the water pollution control effort.

David D. Dominick Acting Commissioner Water Quality

1. Scope and Application

- 1.1 This method is applicable to surface and saline waters in the range of turbidity from 0 to 40 Jackson units.
- 2. Summary of Method
 - 2.1 The method is based upon a comparison of the intensity of light scattered by the sample under defined conditions with the intensity of light scattered by a standard reference suspension. The higher the intensity of scattered light, the higher the turbidity. Readings, in Jackson units, are made in a nephelometer designed according to specifications outlined in Apparatus, 5. A standard suspension of Formazin, also prepared under closely defined conditions, is used to calibrate the instrument.
 - 2.1.1 Formazin polymer is used as the turbidity reference suspension for water because it is more reproducible than other types of standards previously used for turbidity standards.
- 3. Sample Handling and Preservation
 - 3.1 Samples taken for turbidity measurements should be analyzed as soon as possible. Preservation of samples is not recommended.
- 4. Interferences
 - 4.1 The presence of floating debris and coarse sediments which settle out rapidly will give false high readings. Finely divided air bubbles will also affect the results in a positive manner.
 - 4.2 The presence of true color, that is the color of water which is due to dissolved substances which absorb light will cause turbidities to be low, although this effect is generally not significant with finished waters.

- 5. Apparatus
 - 5.1 The turbidimeter shall consist of a nephelometer with light source for illuminating the sample and one or more photo-electric detectors with a readout device to indicate the intensity of light scattered at right angles to the path of the incident light. The turbidimeter should be so designed that little stray light reaches the detector in the absence of turbidity and should be free from significant drift after a short warm-up period.
 - 5.2 The sensitivity of the instrument should permit detection of turbidity differences of 0.02 unit or less in waters having turbidities less than 1 unit. The instrument should measure from 0 to 40 units turbidity. Several ranges will be necessary to obtain both adequate coverage and sufficient sensitivity for low turbidities.
 - 5.3 The sample tubes to be used with the available instrument must be of clear, colorless glass. They should be kept scrupulously clean, both inside and out, and discarded when they become scratched or etched. They must not be handled at all where the light strikes them, but should be provided with sufficient extra length, or with a protective case, so that they may be handled.
 - 5.4 Differences in physical design of turbidimeters will cause differences in measured values for turbidity even though the same suspension is used for calibration. To minimize such differences, the following design criteria should be observed:
 - 5.4.1 Light source: Tungsten lamp operated at not less than 85% of rated voltage or more than rated voltage.

(Turbidity)

5.4.2 Distance traversed by incident light and scattered light within the sample tube: Total not to exceed 10 cm.

5.4.3 Angle of light acceptance of the detector: Centered at 90°
to the incident light path and not to exceed ±30° from 90°.
5.4.4 Maximum turbidity to be measured: 40 units.

5.5 At the time of this writing, the only instrument commercially available with these specifications is the Hach Turbidimeter, Model 2100 and 2100A. This instrument is recommended.

6. Reagents

6.1 Turbidity-free water - Pass distilled water through a 0.45 μ pore size membrane filter if such filter and water shows a lower turbidity than the distilled water.

6.2 Stock turbidity suspension:

Solution 1: Dissolve 1.00g hydrazine sulfate, $(HN_2)_2 \cdot H_2SO_4$, in distilled water and dilute to 100 ml in a volumetric flask.

Solution 2: Dissolve 10.00g hexamethylenetetramine in distilled water and dilute to 100 ml in a volumetric flask.

In a 100-ml volumetric flask, mix 5.0 ml Solution 1 with 5.0 ml Solution 2. Allow to stand 24 hours at 25 \pm 3°C, then dilute to the mark and mix.

6.3 Standard turbidity suspension: Dilute 10.00 ml stock turbidity suspension to 100 ml with turbidity-free water. The turbidity of this suspension is defined as 40 units. Dilute portions of the standard turbidity suspension with turbidity-free water as required.

6.3.1 A new stock turbidity suspension should be prepared each month. The standard turbidity suspension and dilute turbidity standards

should be prepared weekly by dilution of the stock turbidity suspension.

7. Procedure

7.1 Turbidimeter calibration: The manufacturer's operating instructions should be followed. Measure standards on the turbidimeter covering the range of interest. If the instrument is already calibrated in standard turbidity units, this procedure will check the accuracy of the calibration scales. At least one standard should be run in each instrument range to be used. Some instruments permit adjustments of sensitivity so that scale values will correspond to turbidities.

Reliance on a manufacturer's solid scattering standard for setting overall instrument sensitivity for all ranges is not an acceptable practice unless the turbidimeter has been shown to be free of drift on all ranges. If a pre-calibrated scale is not supplied, then calibration curves should be prepared for each range of the instrument.

- 7.2 Turbidities less than 40 units: Shake the sample to thoroughly disperse the solids. Wait until air bubbles disappear then pour the sample into the turbidimeter tube. Read the turbidity directly from the instrument scale or from the appropriate calibration curve.
- 7.3 Turbidities exceeding 40 units: Dilute the sample with one or more volumes of turbidity-free water until the turbidity falls below 40 units. The turbidity of the original sample is then computed from the turbidity of the diluted sample and the dilution factor. For example, if 5 volumes of turbidity-free water were added to 1 volume of sample, and the diluted sample showed a turbidity of 30 units, then the turbidity of the original sample was 180 units.

7.3.1 The Hach Turbidimeters, Models 2100 and 2100A, are equipped with 5 separate scales: 0-.02, 0-1.0, 0-10.0, 0-100, and 0-1000 JTU. It is strongly recommended, however, that the upper scales be used as indications of required dilution volumes to reduce readings to less than 40 JTU. (NOTE: Comparative work performed in the AQC Laboratory indicates a progressive error on sample turbidities in excess of 40 units.)

8. Calculation

- 8.1 Multiply sample readings by appropriate dilution to obtain final reading.
- 8.2 Report results as follows:

| Jackson Turbidity Units | Record to nearest: |
|----------------------------|-----------------------|
| 0.0-1.0 | 0.05 |
| 1-10 | 0.1 |
| 10-40 | 1 |
| 40-100 | 5 |
| 100-400 | 10 |
| 400-1000 | 50 |
| >1000 | 100 |

9. Precision and Accuracy

9.1 Precision and accuracy data are not available at this time.