Standing Committee of Analysts

The Determination of Colour and Turbidity in Raw and Drinking Waters 2017

Methods for the Examination of Waters and Associated Materials

The determination of colour and turbidity in raw and drinking waters (2017)

Methods for the Examination of Waters and Associated Materials

This booklet contains details one manual method for the determination of colour and one manual method for the determination of turbidity.

A separate SCA document covers the automation of these tests.

Whilst this booklet may report details of the materials actually used, this does not constitute an endorsement of these products but serves only as an illustrative example. Equivalent products are available and it should be understood that the performance characteristics of the method might differ when other materials are used. It is left to users to evaluate methods in their own laboratories.

This bluebook updates and replaces: Colour and Turbidity of Waters 1981. ISBN 0 11 751955 3.

Whilst specific commercial products may be referred to in this document, this does not constitute an endorsement of these products. They serve only as illustrative examples of the types of products available. Equivalent products may be available and it should be understood that the performance of the method might differ when other materials are used and all should be confirmed by validation of the method.

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About this series

Introduction

This booklet is part of a series intended to provide authoritative guidance on recommended methods of sampling and analysis for determining the quality of drinking water, ground water, river water and sea water, waste water and effluents as well as sewage sludges, sediments, soils (including contaminated land) and biota. In addition, short reviews of the most important analytical techniques of interest to the water and sewage industries are included.

Performance of methods

Ideally, all methods should be fully evaluated with results from performance tests. These methods should be capable of establishing, within specified or pre-determined and acceptable limits of deviation and detection, whether or not any sample contains concentrations of parameters above those of interest.

For a method to be considered fully evaluated, individual results from at least three laboratories should be reported. The specifications of performance generally relate to maximum tolerable values for total error (random and systematic errors) systematic error (bias) total standard deviation and limit of detection. Often, full evaluation is not possible and only limited performance data may be available.

In addition, good laboratory practice and analytical quality control are essential if satisfactory results are to be achieved.

Standing Committee of Analysts

The preparation of booklets within the series “Methods for the Examination of Waters and Associated Materials” and their continuing
revision is the responsibility of the Standing Committee of Analysts (established 1972 by the Department of the Environment). At present, there are eight working groups, each responsible for one section or aspect of water quality analysis. They are

1 General principles of sampling and accuracy of results
2 Microbiological methods
3 Empirical, Inorganic and physical methods
4 Metals and metalloids
5 Solid substances
6 Organic impurities
7 Biological, biodegradability and inhibition methods
8 Radiochemical methods

The actual methods and reviews are produced by smaller panels of experts in the appropriate field, in co-operation with the working group and main committee. The names of those members principally associated with these methods are listed at the back of this booklet.

Publication of new or revised methods will be notified to the technical press. If users wish to receive copies or advanced notice of forthcoming publications or obtain details of the index of methods then contact the Secretary on the SCA’s web-page:- http://www.standingcommitteeofanalysts.co.uk/Contact.html

Every effort is made to avoid errors appearing in the published text. If, however, any are found, please notify the Secretary. Users should ensure they are aware of the most recent version they seek.

Rob Carter
Secretary
April 2017

Warning to users

The analytical procedures described in this booklet should only be carried out under the proper supervision of competent, trained analysts in properly equipped laboratories.

All possible safety precautions should be followed and appropriate regulatory requirements complied with. This should include compliance with the Health and Safety at Work etc. Act 1974 and all regulations made under the Act, and the Control of Substances Hazardous to Health Regulations 2002 (SI 2002/2677). Where particular or exceptional hazards exist in carrying out the procedures described in this booklet, then specific attention is noted.

Numerous publications are available giving practical details on first aid and laboratory safety. These should be consulted and be readily accessible to all analysts. Amongst such resources are; HSE website HSE: Information about health and safety at work ; RSC website http://www.rsc.org/learn-chemistry/collections/health-and-safety ,"Safe Practices in Chemical Laboratories" and "Hazards in the Chemical Laboratory", 1992, produced by the Royal Society of Chemistry; "Guidelines for Microbiological Safety", 1986, Portland Press, Colchester, produced by Member Societies of the Microbiological Consultative Committee; and "Biological Agents: Managing the Risks in Laboratories and Healthcare Premises", 2005 and "The Approved List of Biological Agents" 2013, produced by the Advisory Committee on Dangerous Pathogens of the Health and Safety Executive (HSE).

A The manual determination of Colour in Raw and Treated Waters

A1 Introduction - Colour

When viewed by transmitted light through a depth of several feet, pure water exhibits a variety of colours. The most common colour is light blue but this may be may be modified by the presence of organic matter to greenish blue, green, greenish yellow, yellow or brown.

The standard analytical colour test is "true colour." The sample is filtered through a 0.45µm cellulose acetate filter. Any measurable colour is due to dissolved matter only.

When measuring "apparent colour" the sample is not filtered. Any measurable colour is due to both dissolved material and suspended material.

It is important to note that when considering the optical properties of waters that colour and turbidity values can change with time. pH values can affect determined colour values. Samples may precipitate during the time between sampling and analysis affecting turbidity values. Users of this method must establish stability times for their own samples. Factors to consider would include:

- Sampling arrangements
- Sample container used
- Sample storage arrangements

Colour results are expressed as mg/l Pt/Co or “Hazen” units.

One “Hazen” unit is defined as the colour produced by 1mg/l platinum in the presence of 2mg/l cobaltous chloride.

A2 Scope

The method is suitable for the examination of surface and ground waters and drinking waters.

A3 Performance characteristics of the method

A3.1	Substances determined	Colour value expressed as mg/l Pt/Co or "Hazen" Units.
A3.2	Type of sample	Raw waters, drinking waters and process waters.
A3.3	Basis of method	The absorbance value of samples at a wavelength of 400nm is compared to a previously constructed calibration graph using Pt/Co standards.
A3.4	Range of application	Typically, up to 30 Hazen. The range may be extended (see section A4.3).
A3.5	Standard deviation	See Table A1.
A3.6	Limit of detection	Typically 0.7 Hazen based on 5 x S_W of deionised water "blanks."
A3.7	Bias	See Table A1.
A3.8	Interferences	"True" colour values – none. "Apparent" colour values will increase with increasing turbidity levels.
A3.9	Sample stability	See table A2

A4 Principle

The absorbance values at 400nm of a series of Pt/Co standards are determined using a spectrophotometer.

A calibration graph is then constructed of “absorbance value” in absorbance units verses colour value (mg/l Pt/Co). The graph is typically linear.

The absorbance values obtained for samples are then referenced to the previously constructed calibration graph.

Colour values are expressed as mg/l Pt/Co or “Hazen” Units.

A5 Hazards

Refer to the manufacturers Safety Data Sheet for full details of the hazards associated with these materials before use. Wear appropriate PPE.

Potassium hexachloroplatinate (IV) is toxic and an eye and skin irritant. Repeated exposure may cause sensitisation.

Cobalt (II) chloride hexahydrate is harmful, a suspect carcinogen, may cause genetic defects and damage fertility, an irritant and highly damaging to the environment. It may cause asthma or an allergic response if inhaled.

Concentrated Hydrochloric acid (d_{20}1.18) is corrosive and gives off irritant or corrosive vapours. This material must be handled as far as possible in a fume cupboard.

A6 Reagents

Where possible, all reagents must be of analytical grade.

A6.1 Deionised water (conductivity <2µS/cm is suitable)

A6.2 500 Hazen Colour Calibration Stock

This solution may be prepared or can alternatively be bought in.

Using a 4d.p. analytical balance, weight out 1.245 ± 0.001g potassium hexachloroplatinate ($K,PtC1_6$) and 1.000 ± 0.001 g cobalt (II) chloride hexahydrate ($CoC1_26H_20$).

Dissolve in about 500 ml of water.

Using a suitable measuring cylinder, add 100 ± 1ml of hydrochloric acid (d_{20}1.18) and dilute with water to 1-litre in a calibrated flask. This prepares a colour stock of 500 Hazen units. This standard must be stored in an amber bottle and is stable for 6 months.

A6.3 Colour Working Standards

Prepare suitable working colour standards appropriate to the range of analysis required. Typically 4-6 standards are prepared. Calibration standards of 0-30 Hazen are suggested below. Pipette appropriate volumes of 500 Hazen Stock (A4.2) into a series of 100ml calibrated flasks. Make up to the mark with DI water and mix well before use.

The analytical range can be extended by using cuvettes of shorter optical path length. Care

must be taken to ensure that the range is not extended to the point that the calibration graph ceases to be linear. Using a 30 Hazen calibration range a cuvette with 4cm optical path length is suitable.

Volume of 500 Hazen Stock (A4.2) Added to 100ml Calibrated Flask	Colour Standard Value
0.0	0.0 Hazen
2.0	10.0 Hazen
4.0	20.0 Hazen
6.0	30.0 Hazen

A6.4 500 Hazen Colour AQC Stock

This solution may be prepared or can alternatively be bought in.

This stock must be prepared using chemicals supplied from a different supplier, or from a different batch to that used for the calibration stock (A4.2)

Using a 4d.p. analytical balance, weight out 1.245 ± 0.001g potassium hexachloroplatinate ($K,PtC1_6$) and 1.000 ± 0.001 g cobalt (II) chloride hexahydrate ($CoCl_26H_20$).

Dissolve in about 500 ml of water.

Using a suitable measuring cylinder, add 100 ± 1ml of hydrochloric acid ($d_{20}1.18$) and dilute with water to 1-litre in a calibrated flask. This prepares a colour stock of 500 Hazen units. This standard must be stored in an amber bottle.

A6.5 20 Hazen AQC Solution

Pipette 10 ± 0.05ml of 500 Hazen Colour Stock (A4.4) into a 250ml calibrated flask. Make up to the mark with deionised water and mix well before use.

The standard is stable for a maximum of two days if stored in the dark.

A6.6 Glassware cleaning solution made up to the manufacturers recommended strength.

A7 Apparatus

In addition to normal laboratory glassware, the following apparatus will be required.

A7.1 Spectrophotometer capable of measuring absorbance at 400nm.

A7.2 Cuvette of suitable optical path length for the calibration range selected.

A7.3 0.45µ cellulose acetate filters (either filter discs or syringe type filters) plus associated sample filtration equipment.

A8 Sample collection and preparation

Samples may be taken in glass or plastic bottles of suitable size. Approximately 50ml will be required for each determination.

It is recommended samples are stored refrigerated in the dark prior to analysis.

It is important to note that users must establish the stability time of their own samples.

A9 Analytical procedure

Step	Procedure	Notes
A9.1	Turn on the spectrophotometer and set it up to read absorbance units at 400nm wavelength.	(a) Allow the spectrophotometer to warm up for the recommended time before use. Wavelengths within the range 400 nm may be suitable but users should establish that the method sensitivity has not been compromised.
A9.2	Rinse and then fill the cuvette with deionised water. Wipe the optical faces of the cuvette with tissue.	
A9.3	Insert the cell into the cell holder within the spectrophotometer and close the instrument lid.	(b) In the event of using a “dual beam” spectrophotometer ensure that the cell is located in the “measurement” position.
A9.4	“Zero” the spectrophotometer.	(c) The displayed absorbance value should now read 0.000 ± 0.001
A9.5	Insert the cuvette into the spectrophotometer. Close the lid and note the absorbance value displayed.	
A9.6	In a similar manner read the absorbance values obtained for the other calibration standards.	

A9.7	Construct a calibration graph of Absorbance value at 400nm Verses Colour concentration in Hazen using the values obtained for the calibration standards. The graph must be linear and go through the origin.	(e) The calibration graph can either be constructed manually or using a spreadsheet application such as MS Excel. Many modern spectrophotometers can also plot calibration graphs directly.
A9.8	Filter approximately 50ml of the first sample through the 0.45µm filter, discarding the first 20ml approx. of solution.	
A9.9	Read and note the absorbance of the solution.	
A9.10	In a similar manner, filter and determine the absorbance values of all samples to be analysed.	(d) As part of the run also analyse "Blanks" (DI water) and 20 Hazen AQC samples (A4.5) at the frequency defined by the laboratories internal quality procedures. This will typically be one "Blank" and AQC per 20 samples in randomised positions.
A9.11	Any samples which exceed the absorbance value of the top standard must be appropriately diluted with DI water and then reanalysed.	(e) Alternatively, samples could be analysed on a higher calibration range.
A9.12	Using the calibration graph, determine the colour values corresponding to the recorded absorbance values of the samples.	Report colour results as mg/l Pt/Co or in "Hazen" Units.

A10 Calculation

For most samples no additional calculation is required.

For diluted samples:

Colour result = C x F

C = colour value of diluted sample

F = dilution factor

Table A1 Typical Performance data: Colour

Concentration (nominal value) (Hazen)	Total standard deviation (degrees of freedom) (Hazen)	Relative standard deviation (%)	Recovery (%)	Bias (%)
5.00 (4.805)	0.326 (16)	6.78	N/A	-3.90
25.0 (24.536)	0.684 (12)	2.79	N/A	-1.86
20.0 (19.695)	0.684 (11)	3.47	N/A	-1.52
Borehole sample (1) spiked to 20 Hazen	0.700 (11)	3.51	98.02	
Surface treated sample (2) spiked to 20 Hazen	0.636 (11)	3.21	98.31	
Surface raw sample (3) spiked to 20 Hazen	0.662 (12)	3.32	97.66	

LOD = 0.71 Hazen

(3x s_w for sample 1)

Performance data provided by Thames Water Utilities, Spencer House Laboratory, Reading, Berks

B The determination of Turbidity in raw and Drinking Waters Using a Nephelometric Technique

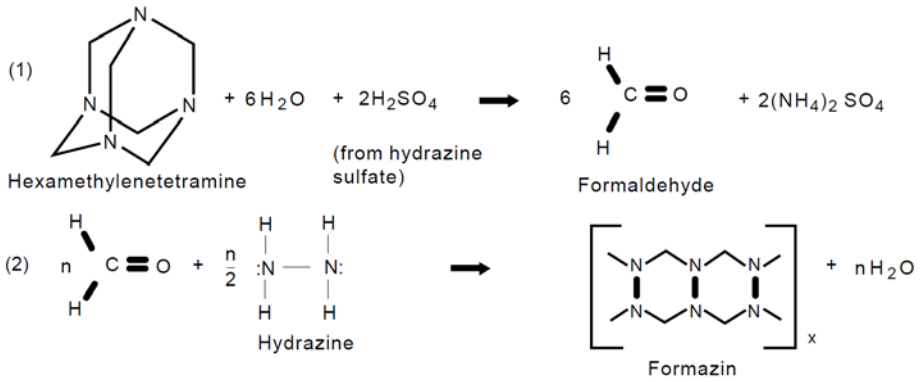
B1 Introduction – Turbidity

Turbidity is an expression of the optical property of a liquid that causes light to be scattered and absorbed rather than transmitted in straight lines through the sample. Turbidity is the measure of particulate and colloidal matter suspended in a solution and is used to measure the clarity of water for public health and aesthetic reasons. The turbidity of a sample is measured nephelometrically in relation to the turbidity produced by Formazin suspensions. Results are expressed as NTU (Nephelometric Turbidity Units) or FTU (Formazin Turbidity Units)

Formazin is a milky white polymer that is used because of its reproducible optical properties.

Formazin is made by reacting hexamethylenetetramine with hydrazine (Refer to Figure 1)

Figure 1 – The structure of Formazin*



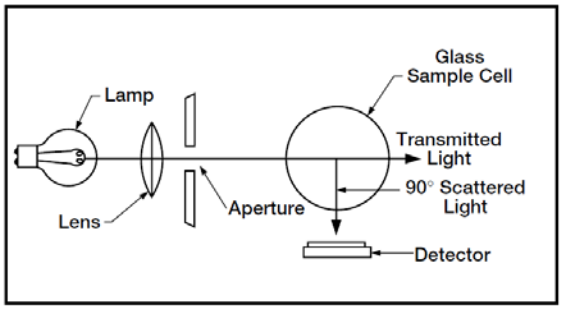
* diagram used by kind permission of Hach Lange UK ltd

Formazin is typically prepared as a concentrated stock that is diluted down to provide working standards. The formazin stock can be prepared or bought in.

Nephelometry is a method of measuring turbidity. In a nephelometric turbidity meter light from a light source shines on a cell containing the sample. The cell has a detector at 90º to the incident light beam. The detector measures light reflected by cloudiness and particulate matter present in the sample.

The internal layout of a typical nephelometric turbidity meter is illustrated in figure 2.

Figure 2: The internal layout of a typical nephelometric turbidity meter* -



* diagram used by kind permission of Hach Lange UK ltd

B2 Performance characteristics of the method

B2.1	Substances determined	Turbidity - measured in NTU or FTU (1NTU=1FTU)
B2.2	Type of sample	Raw waters (surface and ground waters) and drinking waters.
B2.3	Basis of method	The turbidity of the sample is measured using a nephelometric turbidity meter with reference to formazin calibration standards.
B2.4	Range of application	Typically up to 4,000 NTU
B2.5	Standard deviation	See Table B13.
B2.6	Limit of detection	Typically 0.06 NTU (5x s_w Blank or 3x s_w Sample)
B2.7	Bias	See Table B13.

B3 Principle

Turbidity is measured using a nephelometric turbidity meter. The sample is shaken and poured into a glass measurement cell that is inserted into the meter. The sample cell is illuminated with a light source. Turbidity is measured by the amount of light scattered by particulate matter and cloudiness within the sample. The meter is calibrated using formazin standards.

B4 Interferences

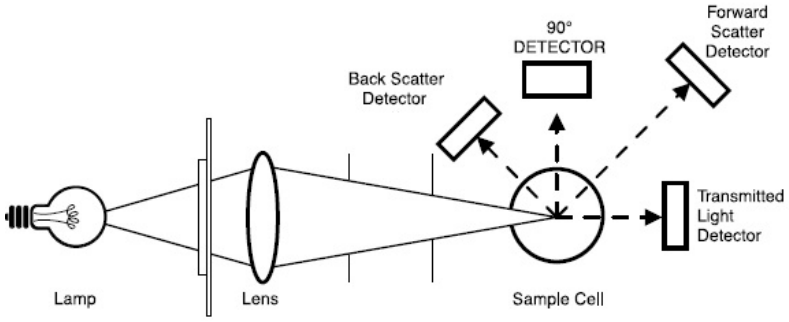
Excessive levels of colour can interfere with turbidity measurement. This is unlikely to be an issue with most potable samples.

If colour is known to be an issue some manufacturers supply turbidity meters with an LED light source which generates light at a longer wavelength than conventional tungsten lamps. The light from an LED source is typically in the wavelength range 850-880nm. Few natural materials absorb light in this wavelength range and hence colour interference is substantially reduced.

Another technique for reducing colour interference when measuring turbidity is through the use of “ratio detection systems.”

A “ratio” turbidimeter has a series of detectors at varying angles around and behind the measurement cell. Refer to figure 3

Figure 3 – typical optical design of a “ratio” turbidimeter*



* diagram used by kind permission of Hach Lange UK ltd

The transmitted light detector behind the cell detects light losses due to turbidity but also detects light losses due to sample colour.

The expressed turbidity value is calculated from an algorithm that utilises a “ratio” of the outputs from the different sensors. This can correct for colour interference as well as other measurement issues.

B5 Hazards

Refer to the manufacturers SDS sheet for full details of the hazards associated with these materials before use. Wear appropriate PPE.

Hexamethylenetetramine is highly flammable and may cause skin sensitisation.

Hydrazinium sulphate is toxic, causes severe skin burns, may cause skin allergies, may cause cancer and is very toxic to aquatic life.

Formazin Stock Solution may cause skin irritation or sensitisation.

B6 Reagents

The range of formazin standards required to calibrate a turbidity meter is likely to vary dependent on the meter manufacturer. Calibration standards are prepared by dilution of a stock solution that can be prepared (see B5.2 to B5.4) or bought in.

Formazin readily settles out. Formazin standards must be well shaken prior to use.

Some manufacturers supply calibration standards in the form of stabilised vials of formazin ready for immediate use.

B6.1 Deionised water (Typically <2µS/cm) quality is generally satisfactory.

If the turbidity of the DI water is suspected of having an unsatisfactorily high turbidity value it should be filtered before use through a 0.1µ membrane filter.

B6.2 1.0% $^{w}/_{v}$ Hydrazinium Sulphate Solution

Weigh out 1 ± 0.0010g of hydrazinium sulphate and dissolve in approximately 60ml of DI water. Transfer to a 100ml calibrated flask and make up to the mark. Shake well. Transfer to an amber glass bottle. This solution is stable for one month.

B6.3 10.0% $^{w}/_{v}$ hexamethylenetetramine Solution

Weigh out 10.0 ± 0.01g of hexamethylenetetramine and dissolve in approximately 60ml of DI water. Transfer to a 100ml calibrated flask and make up to the mark. Shake well. Transfer to an amber glass bottle. This solution is stable for one month.

B6.4 400 NTU Stock Turbidity Standard

Pipette 5 ± 0.03ml of both hydrazinium sulphate solution (B5.2) and hexamethylenetetramine solution (B5.3) into a 100ml calibrated flask.

Mix well and allow to stand for 24 hours at 25 ± 3ºC. After 24 hours the solution will be milky white. Make up to the mark with DI water and mix well. Transfer to an amber glass bottle. This solution is stable for one month.

B6.5 Glassware cleaning solution made up to the manufacturers recommended strength.

B7 Apparatus

In addition to normal laboratory glassware, the following may be required.

B7.1 Nephelometric turbidity meter.

B7.2 Glass cells for turbidity measurement.

It is essential that measurement cells are kept scrupulously clean by periodic soaking in glassware cleaning solution. After soaking, the cell must be thoroughly rinsed with tap and then DI water.

Some manufacturers supply a special “polishing” cloth and silicon oil to help keep cells clean and scratch free. The silicon oil has a similar optical density to the glass used in the cell. Applying a thin layer of oil can hide minor scratches.

Cells that are permanently soiled or scratched must be discarded.

Some measurement cells have a specific “mark” on the glass. This has to be aligned with a mark on the meter. This is to ensure that the cells are inserted into the meter in a consistent manner.

Before a cell is inserted into the meter it must be dried and all grease removed by wiping with soft tissue paper.

B8 Sample collection and preparation

Samples can be taken in either glass or plastic bottles of suitable size. It is essential that the bottles used are scrupulously clean and free from dust.

Samples are recommended to be stored refrigerated in the dark, prior to analysis.

It is important to note that users must establish the stability time of their own samples.

In hard water areas where samples are high in calcium salts, the sample stability time for some samples may be reduced once the bottle is opened. Exposure to the air can result in calcium precipitation leading to increasing turbidity values.

B9 Analytical procedure

Step	Procedure	Notes
B9.1	Turn the turbidity meter on.	Allow the meter to warm up for the recommended time or leave the meter permanently switched on.
B9.2	Calibrate the meter according to the manufacturer's instructions. Turbidity meters do not require calibration every day. Typically meters are calibrated at 6 or 12 monthly intervals. The meter calibration must however be verified daily by analysing one or more formazin response standards appropriate to the turbidity values of the samples being analysed.	
B9.3	Rinse the cell thoroughly and then fill with deionised water to provide a "cell/stray light blank" sample. Measure the turbidity of the solution.	The "cell/stray light blank" value requires to be at an acceptable level for the results being reported prior to further analysis commencing
B9.4	Mix the first "response" standard well. Measure the turbidity of the solution. Repeat for all "response" standards.	Ensure that the values obtained for response solutions are within the acceptance limits set. Values outside of the limits must be investigated before proceeding further.

B9.5	Rinse the cell well. Mix the first sample And fill the cell. Wipe the sides of the cell with tissue and insert into the meter.	Care must be taken to insure that samples are not vigorously shaken as this can aerate the sample leading to unstable values.
B9.6	Wait for the turbidity value to stabilise. Note the turbidity value obtained.	See B12
B9.7	"AQC" solutions and "blanks" must be included in the batch of analysis at a recommended frequency of approximately one within 20 samples.	"blanks" are comprised of DI water. AQC standards are formazin standards prepared completely independently to any standards used for calibration or response checking. AQC standards are prepared at a concentration appropriate to the samples being analysed.

B10 Calculation

Turbidity result (NTU) = T_m - T_b

where

T_m = measured turbidity of sample (NTU)

T_b = "cell blank" or "stray light" value (NTU)

At higher levels of turbidity users may deem the "cell blank" or "stray light" correction insignificant and not subtract it.

B11 Interferences

B11.1 The presence of floating debris and coarse sediments which settle rapidly may give low readings.

B11.2 Dirty measuring cells may produce inaccurate readings and so must be kept scrupulously clean. For this reason the measurement of samples containing traces of oil should be avoided.

B11.3 Fine air bubbles in the sample may give high readings.

B11.4 Colour in the sample may give rise to low results depending on the particular instrument used. Although no specific tests of the qualitative effect of colour have been made, the effect is considered usually unimportant for treated waters. Each application should be given individual consideration depending on the instrument used and the nature of the samples.

B11.5 Light absorbing materials such as activated carbon may cause low readings.

B11.6 Sample cell variations can produce inaccurate readings. This can be minimised by the use of matching and indexing techniques. The application of silicon oils can reduce reflections due to scratches on the cell surface.

B11.7 Stray light in the instrument may give high readings. Generally speaking, interferences resulting in a negative bias are associated with measurements greater than 1 NTU and can become more significant as the value increases. Positive turbidity interferences are typically associated with very low turbidity measurements which are values below 0.1 NTU. These effects may make achieving an appropriate limit of detection to meet the requirement above problematic.

B12 Timing of measurement

B12.1 Air bubbles should be allowed to surface and easily settleable solids to settle, for this reason the meter should be allowed to stabilise before the reading is taken.

B12.2 The reading must remain stable for 3 seconds before it is recorded. If the nature of the turbidity in the sample gives an unstable reading then it should be taken at 60 seconds.

B13: Typical Performance Data: Turbidity

Concentration (nominal value) (NTU)	Total standard deviation (degrees of freedom) (NTU)	Relative standard deviation (%)	Recovery (%)	Bias (%)
1.00 (1.06)	0.030 (15)	2.85	N/A	6.00
4.00 (3.982)	0.052 (22)	1.31	N/A	-0.45
Surface water treated sample spiked to 1NTU	0.128 (18)	12.22	100.12	
spiked to 4NTU	0.057 (20)	1.39	96.52	
Treated Borehole supply				
Spiked to 1NTU	0.086 (18)	8.62	99.22	
Spiked to 4NTU	0.102 (18)	2.48	97.37	
Surface raw sample spiked to				
spiked to 4NTU	0.164 (21)	3.99	96.69	

LOD = 0.06 NTU

5x s_w for Blank

Performance data provided by Thames Water Utilities, Spencer House Laboratory, Reading, Berks

C The determination of Colour and Turbidity in raw and drinking waters using an Automated Technique

C1 Performance characteristics of the method

C1.1	Substances determined	Turbidity - measured in NTU Colour – measured in mg/l Pt/Co
C1.2	Type of sample	Raw waters (surface and ground waters) and drinking waters.
C1.3	Basis of method	The turbidity of the sample is measured using a nephelometric turbidity meter with reference to formazin calibration standards. Colour is determined from the absorbance value of samples at a wavelength of 400nm compared to a previously constructed calibration graph using Pt/Co standards.
C1.4	Range of application	Typically Turbidity to 5.0 NTU Colour to 25 mg/l Pt/Co
C1.5	Standard deviation	See Table B13.
C1.6	Limit of detection	Typically 0.06 NTU (5x s_w blank)
C1.7	Bias	See Table B13.

C2 Principle

An Automated Robotic system is designed to perform Turbidity and Colour measurements on potable water samples. A typical system comprises of an auto sampler that is capable of sampling either from the original sample bottle or a suitable vessel containing a sub sample. The sample is pumped through tubing by peristaltic pumps into flow cells contained within a spectrophotometer and turbidimeter. These instruments may be of the same type used to carry out manual determination of colour and turbidity.

The system may use a barcode reader to identify the samples analysed or alternatively the samples can be pre-programmed into the systems' software. A racking list can then be produced to determine the order in which samples are placed on the auto sampler and analysed.

The turbidity of a sample is measured nephelometrically in relation to the turbidity produced by Formazin suspensions. The units of measurement are Nephelometric Turbidity Units (NTU). Any turbidity in the samples is re-suspended by introducing a short air purge down the sampling tubes to the base of the sample bottles, or alternatively by using a stirring device to agitate the sample.

True Colour is measured spectrophotometrically; True Colour is the colour due to dissolved matter in the liquid and is measured using a spectrophotometer at a wavelength of 400nm with a path length of either 10mm or 40mm. Before the sample is analysed it is filtered through a 0.45 µm cellulose acetate membrane. The Colour of a sample is measured in mg/l Pt/Co.

C3 Hazards

Refer to the manufacturers SDS sheet for full details of the hazards associated with these materials before use. Wear appropriate PPE.

Formazin Stock Solution may cause skin irritation or sensitisation.

Potassium Hexachloroplatinate (IV) is toxic and can be fatal if swallowed. Can cause breathing difficulties if inhaled. Can cause serious damage to the eyes.

Hydrochloric acid causes severe burns to skin and eye damage. May also cause respiratory irritation.

Potassium chromate (VI) is harmful and toxic to the environment. May cause genetic defects, cancer, allergic skin reactions, eye irritation and skin irritation.

Cobalt (II) Chloride hexahydrate may damage fertility, cause genetic defects, damage internal organs, cause cancer and allergic skin reactions. Harmful if swallowed. It is also toxic to the environment.

C4 Reagents

C4.1 Deionised Water

The water must have a concentration of the analytes of interest below the method detection limits. This water should be used in the preparation of all standards and controls in this test. This water is also used for system blanks.

C4.2 0.5M Sulphuric Acid

Transfer to a 500 ml volumetric flask containing approximately 200 ml of deionised water, 50 ± 1ml of the 5M sulphuric acid standard solution and dilute to volume with deionised water. Mix thoroughly. This solution is stable for 3 months when stored in a plastic bottle at room temperature.

C4.3 Calibration standards

C4.4 4000 NTU Formazin Turbidity Stock Calibration Standard

Use as supplied. The suspension is stable until the manufacturer’s expiry date or for one year from opening if an expiry date is not supplied. Store at room temperature.

C4.5 Turbidity Working Calibration Standards

Using an auto-pipette or glass bulb pipette, prepare a set of Calibration standards appropriate to the range of application required. Typical concentrations are 0.5, 2.5 and 5.0NTU. Formazin suspensions should be prepared daily and stored at room temperature.

C4.6 Colour Stock Calibration Standard - 2000 mg/l Pt/Co

Weigh 4.9800 ± 0.0010 g of potassium hexachloroplatinate (IV) and 4.0000 ± 0.0010 g of cobalt (II) chloride hexahydrate and dissolve in approximately 500ml of deionised water in a 1L volumetric flask. Add 100 ± 1 ml of concentrated hydrochloric acid. Dilute to volume with deionised water. This solution is stable for 6 months when stored in an amber glass bottle at room temperature. Mix thoroughly.

C4.7 Colour Working Calibration Standards

Using an auto-pipette or glass bulb pipette, prepare a set of Calibration standards appropriate to the range of application required. Typical concentrations are 5, 10 and 25mg/l Pt/Co. These solutions should be prepared daily and stored in an amber glass bottle at room temperature. When transferred to the automated system, colour standard solutions should be analysed immediately or stored in bottles or pots that exclude light to prevent chemical degradation. An example of this is to wrap bottles in aluminium foil.

C4.8 4000 NTU HACH Formazin Turbidity Stock AQC Standard

Use as supplied. The solution is stable for one year after opening or until the manufacturer's expiry date whichever is the sooner. Store at room temperature.

C4.9 Turbidity AQC Working Standards

Using an auto-pipette or glass bulb pipette, prepare an AQC standard appropriate to the area of interest required. Typical concentrations are 1.0 and 4.0NTU due to the current PCVs for drinking water being set at these values. Formazin suspensions should be prepared daily and stored at room temperature.

C4.10 Colour Stock AQC Standard – 2030 mg/l

Weigh 2.0300 ± 0.0005g of potassium chromate (previously dried at 105 ± 5oC for at least an hour and allowed to cool in a desiccator) and dissolve in approximately 500ml of deionised water in a 1L volumetric flask. Dilute to volume with deionised water. Mix thoroughly.

C4.11 Colour Working AQC Standard – equivalent to 20 mg/l Pt/Co

Using a 25 ml glass bulb pipette, add 25 ml of the 2030mg/l colour AQC stock and using an auto-pipette add 5 ml of 0.5M sulphuric acid solution into a 5 L volumetric flask containing approximately 1 L of deionised water. Dilute to volume with deionised water. Mix thoroughly.

C5 Apparatus

(All temperature ranges are nominal)

C5.1 A Robotic system comprising of an autosampler and appropriate analytical instruments; a Nephelometric Turbidimeter and suitable Spectrophotmeter capable of measuring at 400nm. The system shall include a device to re-suspend any turbidity within the sample such as a stirrer or air bubbler. If the system analyses Turbidity and Colour from separate subsamples, the subsample to be analysed for Colour may be manually filtered prior to being placed on the autosampler. If the same subsample is to be analysed for Turbidity and Colour then the system must be capable of filtering samples before colour analysis is performed.

C5.2 Assorted Volumetric flasks

C5.3 Drying oven typically measuring at 105°C.

C5.4 Assorted weighing boats / beakers

C5.5 Analytical balance with a capability of measuring to 0.1mg.

C5.6 0.45um cellulose acetate membrane

C5.7 Various types and sizes of pump tubing

C5.8 Flow cells compatible with the analytical instruments used

C5.9 Assorted Auto-pipettes and associated pipette tips.

C5.10 Assorted glass bulb pipettes

C5.11 Desiccators containing self-indicating silica gel.

C6 Sample collection and preparation

If analysis cannot be immediately undertaken, samples should be stored refrigerated prior to analysis. Samples should be equilibrated to room temperature prior to analysis and analysed as soon as possible after collection.

C7 Analytical procedure

Step	Procedure	Notes
C7.1	Ensure the Automated Robotic system and associated instruments are switched on. Check that the air supply to the system is working and that the air pressure is correct (if applicable). If a stirring device is used, check that it is working correctly. Check the pump tubing for any damage and	Trials should be carried out using samples of known concentrations during method development in order to assign suitable settings within the instrument software. These settings will typically relate to

	change if necessary. Check the filtration mechanism and filter paper to ensure all samples will be filtered prior to colour analysis.	result stability times, the total read time and the number of readings carried out in order to obtain a stable result.
C7.2	Prepare the calibration standards and place them in the correct containers on the auto sampler.	
C7.3	Ensure a calibration is carried out in which the calibration standards are sampled and processed by the Automated Robotic system.	
C7.4	Once the calibration is finished, examine the calibration graphs and make a note of any system suitability check values. If any values are outside the quoted tolerances do not proceed with sample analysis. Investigate why the checks are not satisfactory, resolve any problems found and repeat the calibration(s) that failed. If all of the System Suitability checks are within tolerance levels, commence analysis.	Examples of suitable System Suitability Checks include the correlation coefficient and the absorbance of one or more of the colour calibration standards.
C7.5	If required, prepare a worksheet and racking list to determine the order in which samples will be positioned on the auto sampler.	
C7.6	Prepare the AQC standards required and place them in suitable containers on the auto sampler along with the samples and blanks (recommended frequency of one in every 20 samples). Should samples be subsampled from another container prior to being placed on the auto sampler, ensure that any Turbidity in the sample is re-suspended and that the sample is homogenous before subsampling is carried out.	
C7.7	Some robotic systems are equipped with a barcode reader. If barcodes are used, the sample numbers may be automatically stored in the correct position in the run sequence.	

C7.8 The analytical run may be started when all samples and standards are in position. No further operator input is required until the analytical run is complete.

C7.9 When complete the sample results can be entered onto the laboratory's results database provided that all system suitability checks, calibrations, AQC and blank results are acceptable.

C7.10 All automated systems should be well maintained in order to ensure method performance does not deteriorate. Typically, issues can occur due to restricted sample flow through the system. This can be due to 'flattened' peristaltic pump tubing, the build-up of Formazin or lime scale in the tubing and reduced performance from any motors responsible for pumping sample through the system. A suitable preventative maintenance programme can ensure these issues are rarely encountered. A daily wash with a laboratory cleaning agent can ensure tubing is kept free from any build-up. It is advisable to carry out regular preventative maintenance on any analytical instrumentation in line with the manufacturer's recommendations.

C8 Interferences

C8.1 The presence of floating debris and coarse sediments which settle rapidly may give low readings. It is advised that samples of this type are not analysed using an automated system and are analysed manually.

C8.2 Dirty measuring cells may produce inaccurate readings and so must be kept scrupulously clean. For this reason the measurement of samples containing traces of oil should be avoided.

C8.3 Fine air bubbles in the sample may give high readings. This issue should be considered when setting appropriate read times in the instrument software. An initial delay may be considered in order to enable any bubbles to disperse before readings are taken.

C8.4 Sample cell variations and cell movement can produce inaccurate readings. This can be minimised by the use of matching and indexing techniques. The application of silicon oils can reduce reflections due to scratches on the cell surface. Consideration should be taken that the location of any instrument is not susceptible to vibrations caused by movement of the automated system.

C8.5 See section B11 for other interferences associated with both manual and automated Turbidity analysis.

Address for correspondence

However well procedures may be tested, there is always the possibility of discovering hitherto unknown problems. Analysts with such information are requested to contact the Secretary of the Standing Committee of Analysts at the address given below. In addition, if users wish to receive advance notice of forthcoming publications, please contact the Secretary.

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Standing Committee of Analysts

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