The Determination of Colour of Waters and Wastewaters 1988

A Supplementary Method based upon the "CIELAB" Colour Difference Measurement System

Methods for the Examination of Waters and Associated Materials

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Contents


P.B.O

About This Series

This booklet is part of a series intended to provide both recommended methods for the determination of water quality, and in addition, short reviews of the more important analytical techniques of interest to the water and sewage industries.

In the past, the Department of the Environment and its predecessors, in collaboration with various learned societies, have issued volumes of methods for the analysis of water and sewage culminating in "Analysis of Raw, Potable and Waste Waters". These volumes inevitably took some years to prepare, so that they were often partially out of date before they appeared in print. The present series will be published as series of booklets on single or related topics; thus allowing for the replacement or addition of methods as quickly as possible without need of waiting for the next edition. The rate of publication will also be related to the urgency of requirement for that particular method, tentative methods and notes being issued when necessary.

The aim is to provide as complete and up to date a collection of methods and reviews as is practicable, which will, as far as possible, take into account the analytical facilities available in different parts of the Kingdom, and the quality criteria of interest to those responsible for the various aspects of the water cycle. Because both needs and equipment vary widely, where necessary, a selection of methods may be recommended for a single determinand. It will be the responsibility of the users—the senior technical staff to decide which of these methods to use for the determination in hand. Whilst the attention of the user is drawn to any special known hazards which may occur with the use of any particular method, responsibility for proper supervision and the provision of safe working conditions must remain with the user.

The preparation of this series and its continuous revision is the responsibility of the Standing Committee of Analysts (to review Standard Methods for Quality Control of the Water Cycle). The Standing Committee of Analysts is a committee of the Department of the Environment set up in 1972. Currently it has 9 Working Groups, each responsible for one section or aspect of water cycle quality analysis. They are as follows:

1.0 General principles of sampling and accuracy of results
2.0 Microbiological methods
3.0 Empirical and physical methods
4.0 Metals and metalloids
5.0 General nonmetallic substances
6.0 Organic impurities
7.0 Biological methods
8.0 Sewage Works Control Methods
9.0 Radiochemical methods

The actual methods and reviews are produced by smaller panels of experts in the appropriate field, under the overall supervision of the appropriate working group and the main committee.

The names of those associated with this method are listed inside the back cover. Publication of new or revised methods will be notified to the technical press, whilst a list of Methods in Print is given in the current HMSO Sectional Publication List No 5.

Whilst an effort is made to prevent errors from occurring in the published text, a few errors have been found in booklets in this series. Correction notes and minor additions to published booklets not warranting a new booklet in this series will be issued periodically as the need arises. Should an error be found affecting the operation of a method, the true sense not being obvious, or an error in the printed text be discovered prior to sale, a separate correction note will be issued for inclusion in that booklet.

L R PITTWELL
Secretary

1 July 1987

Warning to Users

The analytical procedures given in this booklet should only be carried out by competent trained persons, with adequate supervision when necessary.

Local Safety Regulations must be observed.

Laboratory procedures should be carried out only in properly equipped laboratories.

Field Operations should be conducted with due regard to possible local hazards, and portable safety equipment should be carried.

Care should be taken against creating hazards for one's self, one's colleagues, those outside the laboratory or work place, or subsequently for maintenance or waste disposal workers. Where the Committee have considered that a special unusual hazard exists, attention has been drawn to this in the text so that additional care might be taken beyond that which should be exercised at all times when carrying out analytical procedures. Reagents of adequate purity must be used, along with properly maintained apparatus and equipment of correct specifications. Specifications for reagents, apparatus and equipment are given in manufacturers' catalogues and various published standards. If contamination is suspected, reagent purity should be checked before use.

Lone working, whether in the laboratory or field, should be discouraged.

The best safeguard is a thorough consideration of hazards and the consequent safety precautions and remedies well in advance. Without intending to give a complete checklist, points that experience has shown are often forgotten include: laboratory tidiness, stray radiation leaks (including ultra violet), use of correct protective clothing and goggles, removal of toxic fumes and wastes, containment in the event of breakage, access to taps, escape routes, and the accessibility of the correct and properly maintained first-aid, fire-fighting, and rescue equipment. Hazardous reagents and solutions should always be stored in plain sight and below face level. Attention should also be given to potential vapour and fire risks. If in doubt, it is safer to assume that the hazard may exist and take reasonable precautions, rather than to assume that no hazard exists until proved otherwise.

There are numerous handbooks on first aid and laboratory safety. Among such publications are: "Guide to Safe Practices in Chemical Laboratories" and "Hazards in the Chemical Laboratory" issued by the Royal Society of Chemistry, London; "Safety in Biological Laboratories" (Editors Hartree and Booth), Biochemical Society Special Publication No 5, The Biochemical Society, London, which includes biological hazards; and "The Prevention of Laboratory Acquired Infection", Public Health Laboratory Service Monograph 6, HMSO, London.

It cannot be too strongly emphasised that prompt first aid, decontamination, or administration of the correct antidote can save life; but that incorrect treatment can make matters worse. It is suggested that both supervisors and operators be familiar with emergency procedures before starting even a slightly hazardous operation, and that doctors consulted after any accident involving chemical contamination, ingestion, or inhalation, be made familiar with the chemical nature of the injury, as some chemical injuries require specialist treatment not normally encountered by most doctors. Similar warning should be given if a biological or radio-chemical injury is suspected. Some very unusual parasites, viruses and other micro-organisms are occasionally encountered in samples and when sampling in the field. In the latter case, all equipment including footwear should be disinfected by appropriate methods if contamination is suspected. If an ambulance is called or a hospital notified of an incoming patient give information on the type of injury, especially if poisoning is suspected, as the patient may be taken directly to a specialized hospital.

The Determination of Colour of Waters and Wastewaters 1988

A Supplementary Method based upon the "CIELAB" Colour Difference Measurement System

Introduction

The water industry has long used Hazen or the related Platinum-Cobalt units for colour comparison measurements. Workers in many other fields have their own special scales such as the US Geological Survey scale for mineral and rock colours, the Gibbon's scale for stamp colours, etc. The basis of all such scales is comparison with appropriate "standard" coloured materials. Whilst the water industry scales are reproducible, they are only strictly applicable to yellow—brownish waters. Many examples of green, blue and even red natural waters are known, due to the presence of dissolved minerals or algal pigments. Also, industrial wastewaters can be of almost any colour. When evaluating the colour of such waters it is therefore necessary to use some more widely applicable measurement if accurate assessments are to be made.

The 1980 booklet in this series "Colour and Turbidity of Waters" which is still valid, suggests that for such waters a full, visible absorption spectrum be plotted. However, several schemes have been devised for assessing colour quantitatively based upon such spectral measurements. The most favoured internationally is the CIE 1976 (L*a*b*), usually written "CIELAB", system which is explained below.

The perceived colour of a water is dependent upon the colour in solution, the colour of any suspended matter present and the relative amounts of each. To make a proper assessment of perceived colour, it is necessary to separate and quantify the colour of the suspended solids if present in any appreciable amount in addition to measuring the colour in solution, even though this does introduce a risk of colour absorption by the filter medium, which must be checked and avoided if necessary, by appropriate choice of filter medium.

Every colour, no matter what the material, can be quantified by the "Tristimulus Values" X, Y & Z, defined by the International Commission on Illumination (CIE). These values are based on the amounts of monochromatic red, green, and blue lights which when mixed together would match the colour. These XYZ values may be transformed mathematically into L*a*b* values which then define a uniform, 3-dimensional "colour space", CIELAB, (See Fig I), in which the distance between any two points is linearly related to the perceived difference in colour between the corresponding samples. A comprehensive account of the development of the CIE methods of colour and colour difference measurements is given by McLaren[1].

The method below specifies the use of colour difference in CIELAB ΔE units between a water sample and distilled/deionised water to derive a measure of the colour of the sample. Because the colour of the water has historically been expressed in Hazen Units, conversion factors have been determined so that colour differences may also be expressed in Colorimetric Hazen Units, CHU. The method is more precise than visual assessment of colour in Hazen Units and is equally applicable to water samples whose hue is significantly different from those of Hazen Standard Solutions. ΔE is a measure of the perceived intensity of colour, and gives a single, unambiguous figure for this intensity, which is independent of hue.

The method also specifies a procedure for the determination of the colour of separated suspended material based upon "reflectance" XYZ measurements. The associated "Reflectance ΔE" values cannot however be related to Hazen Units as in the case of colour in solution, above.

1. Performance Characteristics of the Method

1.1	Type of sample	Raw, Potable and Wastewaters (See Section 3, below).
1.2	Basis of Method	The colour difference between a filtered water sample and distilled/deionised water is determined by transmission measurements, in CIELAB Units, and may be subsequently converted to Colorimetric Hazen Units (CHU) if required. The colour difference between the filter paper on which the suspended material from the original sample has been collected, and a white standard is determined in CIELAB Units by reflectance measurements.

2. Principle

The sample is filtered through an appropriate filter (paper) and the transmittance values of the clear solution at a minimum of 16 wavelengths between 400 and 700 nm, are determined. The colour difference, ΔE in CIELAB Units between the sample and distilled/deionised water is calculated, and converted to Colormetric Hazen Units (CHU).

The reflectance values of the suspended material collected on the filter (paper) and of a titanium dioxide standard deposited on a similar filter (paper) are determined at a minimum of 16 wavelengths between 400 and 700 nm. The colour difference, ΔE, in CIELAB Units between the specimen and the white standard is calculated.

3. Field Application

The method is applicable to the measurement of colour in all raw, potable and wastewaters. It is particularly applicable in cases where the hue of the sample(s) concerned varies significantly from that of Hazen (Platinum—Cobalt) standards.

4. Hazards

All apparatus used for vacuum filtration should be inspected for flaws and star-cracks before use. Ideally, vacuum filtration should be carried out behind a safety screen. Alternatively, the apparatus can be covered with a suitably protective mesh.

5. Apparatus

5.1 A spectrophotometer capable of measuring transmittance values in cells of 10, 20, 30, 40 or 50 nm path length, at a MINIMUM of 16 wavelengths (20 nm intervals) between 400 and 700 nm should be employed. Ideally the measurement range (and hence the number of individual measurements) should be extended to cover 360–780 nm, and an instrument which gives a direct readout of XYZ (transmittance) values should be used, (but see note (j) and the appendix below).

5.2 A spectrophotometer capable of measuring reflectance values at a minimum of 16 wavelengths (20 nm intervals) between 400 and 700 nm should be employed. Ideally the measurement range and hence the number of individual measurements) should be extended to cover 360–780 nm, and an instrument which gives a direct readout of XYZ (reflectance) values should be used, (but see note (j) and the appendix below).

[Note—Many commercially available spectrophotometers can be used to measure both transmittance and reflectance.

The smaller the interval between readings over the spectral measurement range, the more accurate will be the derived data (eg 10 nm interval data will give more accurate results than those obtained from 20 nm interval data. Refer to the appendix and tables 1 and 2 below).

The use of tristimulus colorimeters as an alternative to spectrophotometers in these procedures is not recommended and can only be justified if it has been shown that comparable results can be obtained with such instruments].

5.3 Filtration Apparatus

5.3.1 *Borosilicate glass Buchner type filtration apparatus* of an appropriate size (left to the discretion of the analyst). The Buchner funnels should be of the type comprising a glass frit support, rather than a perforated support. (This is not necessary if only "filtrate" colour is to be measured).

5.3.2 *Glass fibre papers* of an appropriate grade (GF/C) and a diameter for use with the funnels above, or an alternative if necessary. (See note (h) below).

5.4 Reagents

5.4.1 *A 10% v/v solution of glycerol* (analytical Reagent Grade) in water.

5.4.2 *Titanium Dioxide Food Grade powder*, for preparation of the reflectance standard.

5.5 Cleanliness

Maintenance of laboratory glassware in scrupulously clean condition is of utmost importance. Store the base of the filter holder in an inverted position with the neck full of dilute chromic acid, and the Buchner flask filled with 20% v/v hydrochloric acid. Before use pour the cleaning solutions away and thoroughly rinse with water.

Reagent bottles should be cleaned with a 2% v/v solution of a laboratory surfactant, rinsed with 20% v/v hydrochloric acid and thoroughly rinsed with water prior to use.

Store the spectrophotometer cells in a 2% v/v solution of a laboratory surfactant. Empty and rinse thoroughly with water prior to use. Polish the optical surfaces of the cells with an optical polishing cloth.

6. Sample Collection

Collect the samples in borosilicate glass bottles which have been previously cleaned with dilute chromic acid cleaning solution and thoroughly rinsed with water. Rinse the bottle two or three times with sample before filling.

The samples must be analysed as soon as possible after collection.

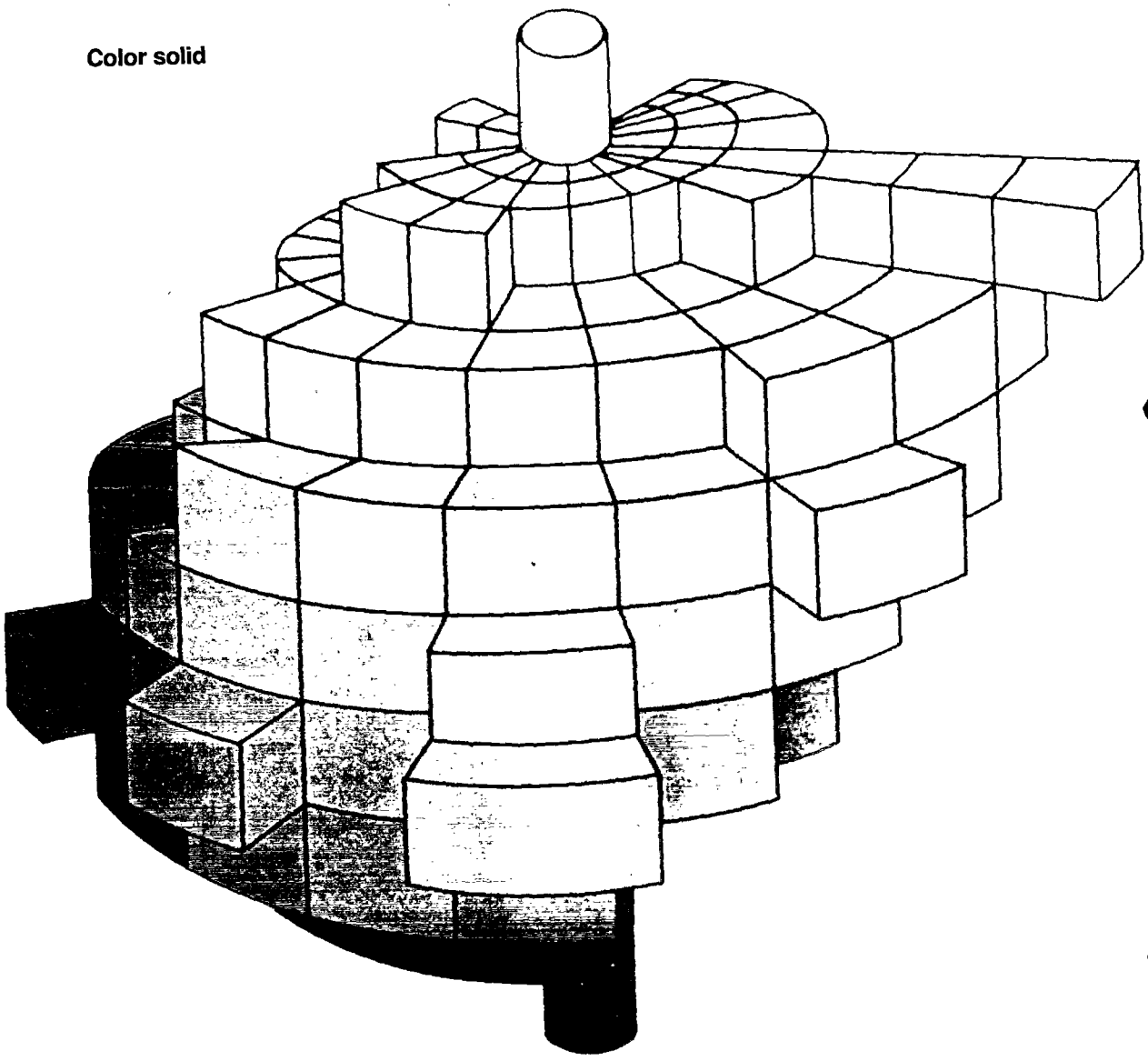
7. Analytical Procedures

Step	Procedure	Notes
	Filtration Method	
7.1	Assembled the clean, dry filtration apparatus complete with glass fibre filter paper, and attach to a vacuum line. (Note (a)).	(a) The procedure outlined assumes that both the colour of the filtered (clear) liquid and that of the solid material retained on the filter are to be evaluated. If the latter is not the case the analyst should disregard the inapplicable parts of the procedure.
7.2	A well mixed aliquot of sample is introduced to the filter funnel and gentle suction applied to effect filtration. Fore-runnings should be collected with the filtrate and NOT rejected. See step 7.4 below if the filtrate is not clear. (Notes (b), (c), (d) and (g)). Continue the filtration until all of the aliquot has been filtered (but not materially longer) and the retained solids on the filter paper appear liquid free.	(b) The volume of the aliquot taken is left to the discretion of the analyst. (But see (d) below). (c) Colour is frequently pH dependent. If pH correction is to be applied, then this should be done before filtration. (d) If the colour of the retained solids is to be measured, sufficient sample must be taken to give a dense, coherent layer on the filter paper.

Step	Procedure	Notes
7.2.1	For reflectance measurements prepare a white standard by similarly filtering an aqueous suspension of an appropriate quantity of food grade titanium dioxide powder, to completely and uniformly cover the filter paper (See 7.3.1).	
7.3	When all the aliquot has been filtered, turn off the vacuum and leave the apparatus until the internal pressure equilibrates with atmospheric. Carefully disassemble the apparatus retaining both filtrate and filter paper as required. (Note (e)).	(e) Care must be taken when removing the filter paper from the funnel, not to break or crack the surface of the film of retained solids, if its colour is to be evaluated. The filter paper and retained solids must not be allowed to dry out completely before colour measurements are made on it, as this also can destroy or alter the nature of the surface to be measured. (It may sometimes be useful in order to produce a suitable solids film for measurement, to "wash" the filter paper and film, mounted in a SEPARATE Buchner apparatus from that use in the original separation, with a small volume of 10% solution of glycerol in water—do not collect this filtrate if this is done).
7.3.1	For reflectance measurements process the filter paper from step 7.2.1 as above and Note (e); that filtrate may be discarded.	
7.4	Visually examine the filtrate. If it is not completely clear (ie $<$ 1FTU), it may be refiltered as above using a new glass fibre paper. Retain the clear filtrate for colour measurements. (Notes (f) and (g)).	(f) Omission of the small amount of "fines" (collected on refiltration) from the solids for colour evaluation is usually without effect on the measured colour of the solids. (g) Occasionally, loss of carbon dioxide from the sample may occur during filtration; this may cause decomposition of bicarbonates or carbonato complexes and lead to the formation of a turbidity which though usually of the same colour as the bulk of the material being filtered off, MAY be of a different colour. In this latter event filtration of a fresh sample under a slight positive pressure from an inert gas (eg nitrogen) is suggested. Since oxidation can also cause colour change and turbidity, especially with chalybeate waters, air should not be used. Use this fresh filtrate instead of the original one if there is a perceptible difference in colour between them.
7.5	Examine the underside of the filter paper. It should not have been materially stained (dyed) by the passage of liquid through it. (Note (h)).	(h) If staining (dyeing) has occurred to any extent, the analyst should consider whether the degree of loss of colour from the sample is acceptable. If it is not, an alternative filter medium (eg Cellulose acetate type filters) should be sought and the determination restarted. The use of any form of "filter aid" materials should be avoided.

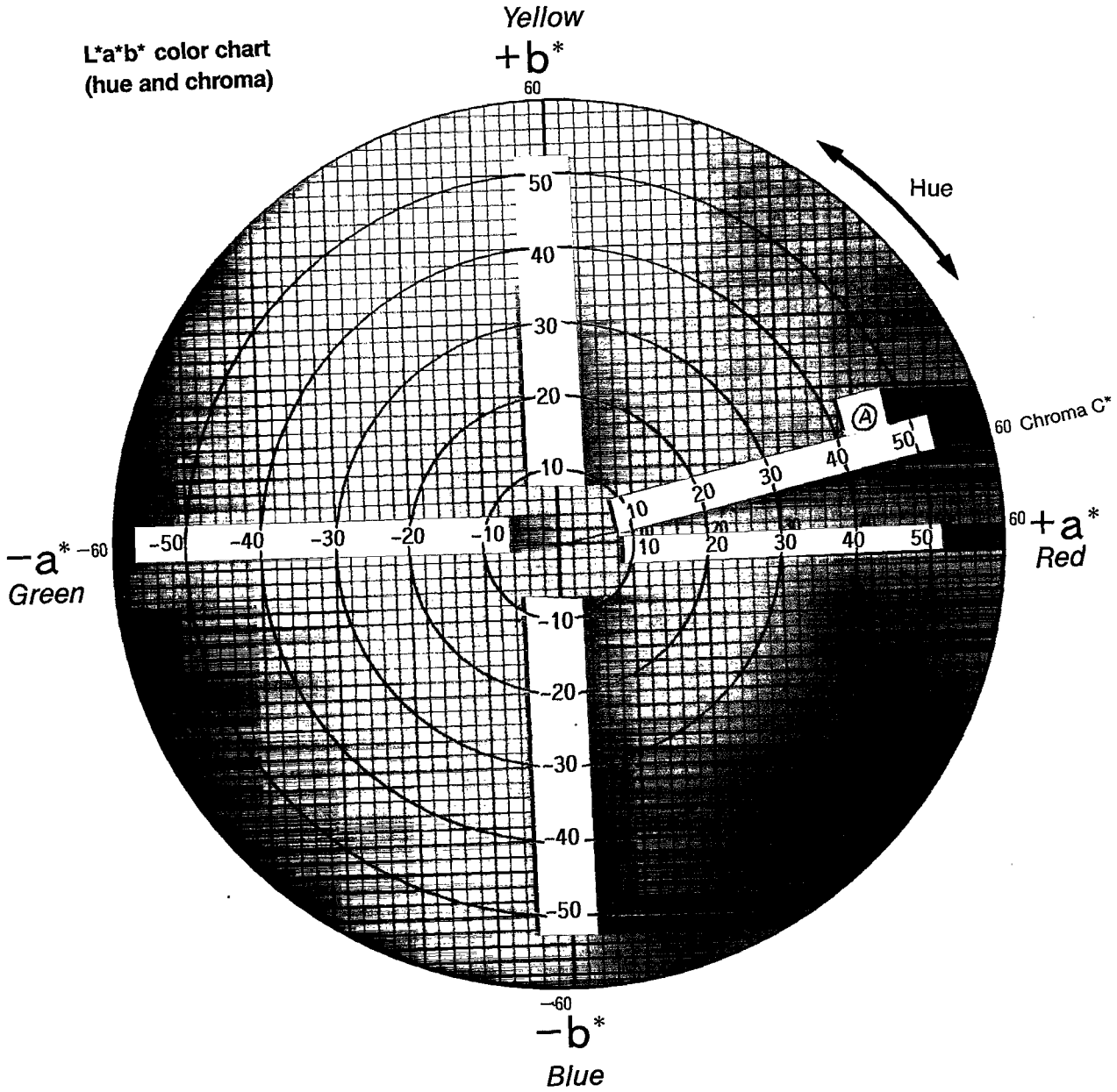
Step	Procedure	Notes
	Measurement of Colour	
7.6	**Filtered Water Samples**	
7.6.1	Calibrate the spectrophotometer for transmittance measurements according to the manufacturer's instructions.	
7.6.2	Fill the cell (cuvette) with distilled/deionised water and measure the transmittance values.	
7.6.3	Fill the cell (cuvette) with filtered sample (note (i)) and measure the respective transmittance values.	(i) As a precaution, fill and empty the cell (cuvette) three times before final filling and measurement.
7.6.4	For both samples and distilled/deionised water calculate the XYZ tristimulus values for the CIE 1964 supplementary standard colorimetric observer under Illuminant D65 (Note (j)).	(j) If the spectrophotometer does not generate these XYZ values automatically, the method described in the appendix shall be used for their derivation.
7.6.5	Convert the XYZ values for both samples and distilled/deionised water into L*a*b* values by applying the equations:— $L^* = 116\ (0.01Y)^{\frac{1}{3}} - 16$ $a^* = 500\ [(0.0105X)^{\frac{1}{3}} - (0.01Y)^{\frac{1}{3}}]$ $b^* = 200\ [(0.01Y)^{\frac{1}{3}} - (0.0093Z)^{\frac{1}{3}}]$	(Distilled/Deionised water has the co-ordinates $L^* = 100$, $a^* = 0$, $b^* = 0$ If this is not the case, prepare fresh distilled water as contamination has occurred. If the water was deionised, the resin is "bleeding" and needs changing or washing.)
7.6.6	Calculate the colour difference, ΔE from:— $\Delta E = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{0.5}$ ΔE is then in CIELAB units and may be used as such to quantify the colour of the water sample (Note (k)).	$\Delta L^* = L^*$ (sample)$-L^*$ (water) $\Delta a^* = a^*$ (sample)$-a^*$ (water) $\Delta b^* = b^*$ (sample)$-b^*$ (water) (k) ΔE values must be accompanied by the optical path length used in their generation, since they are inter-dependent.
7.6.7	ΔE values can be converted to the equivalent Colorimetric Hazen Units by reference to Fig II (attached). This shows that colour differences (ΔE's) between standard Hazen solutions and distilled/deionised waters are linearly related to Hazen Units (for any given path length). Colour differences in CIELAB, (ΔE) units can therefore be converted into Colorimetric Hazen Units (CHU) using $CHU = f \times E$ (Note (1)).	where:— $f = 33.8$ for a 10 mm cell 17.1 20 11.5 30 8.7 40 7.1 50 (l) Colorimetric Hazen Units (CHU) are independent of the path length employed during measurement.

Color solid



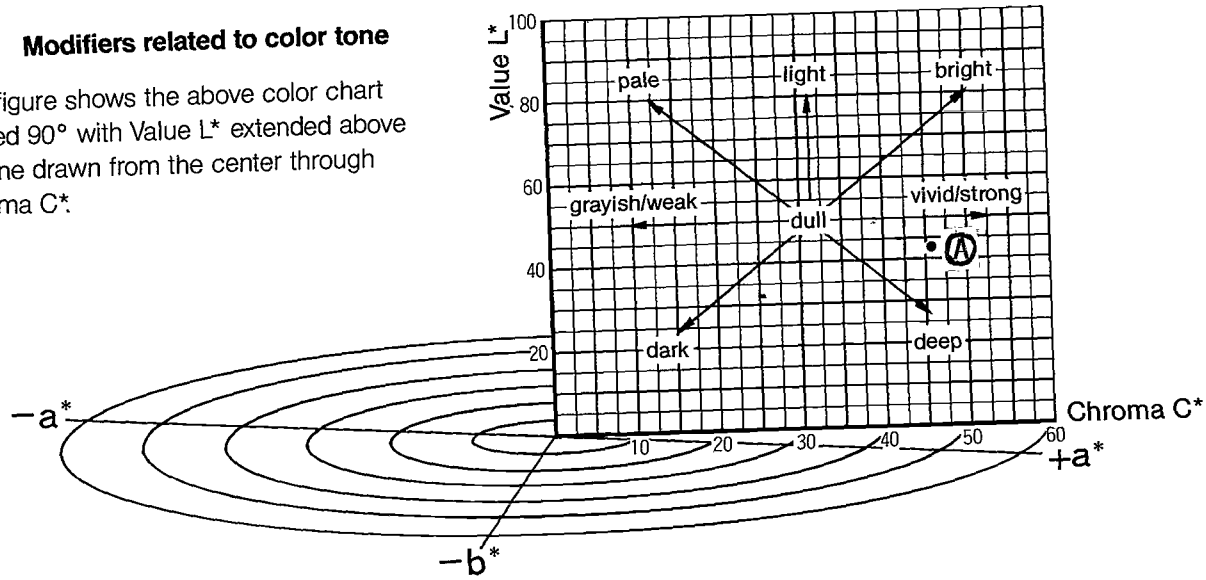
Reproduced from *Precise Color Communication*
courtesy of Minolta UK

L*a*b* color chart (hue and chroma)



Modifiers related to color tone

This figure shows the above color chart rotated 90° with Value L* extended above the line drawn from the center through Chroma C*.



Step	Procedure	Notes
7.7	**Retained Suspended Solids**	
7.7.1	Calibrate the spectrophotometer for reflectance measurements according to the manufacturers instructions.	
7.7.2	Measure the Reflectance XYZ values for the retained solids on the filter paper.	
7.7.3	Measure the Reflectance XYZ values for the titanium dioxide standard (white) paper prepared in steps 7.2.1 and 7.3.1 above.	
7.7.4	From the two sets of reflectance data, calculate the Colour Difference, ΔE, in CIELAB units as described in 7.6.5 and 7.6.6 above.	
	ΔE (reflectance) values may be used as such to quantify the colour of solids retained by filtration from a water sample (Note (m)).	(m) CIELAB, ΔE (reflectance) values CANNOT be converted to equivalent Colorimetric Hazen Units, as can be done with transmittance values relating to coloured solutions.

Appendix

If the spectrophotometer does not generate XYZ values for Illuminant D65 and the 10° observer they can be calculated as follows:—

Multiply each spectral transmittance or spectral reflectance factor (on a 0–1 scale) by the weights given in Table 1 for 10 nm interval data or Table 2 for 20 nm interval data. These tables are contained in ASTM Designation: E 308–85. If data are not available for the full wavelength range (360–780 nm), add the weights at the wavelengths for which data are not available to the weights at the shortest and longest wavelength for which spectral data are available before multiplication with the extremes of spectral (transmittance on reflectance) data. As a minimum, data must be available over the range 400–700 nm. The sum of the products using the X weights is the X tristimulus value. The Y and Z tristimulus values are the sums of the products using the Y and Z weights respectively.

Table 1 Weights for Illuminant D65*, 1964 Observer†, 10 nm Interval

	X	Y	Z
360	0.000	0.000	0.000
370	0.000	0.000	0.000
380	0.000	0.000	−0.002
390	0.008	0.001	0.033
400	0.137	0.014	0.612
410	0.676	0.069	3.110
420	1.603	0.168	7.627
430	2.451	0.300	12.095
440	3.418	0.554	17.537
450	3.699	0.890	19.888
460	3.064	1.290	17.695
470	1.933	1.838	13.000
480	0.802	2.520	7.699
490	0.156	3.226	3.938
500	0.039	4.320	2.046
510	0.347	5.621	1.049
520	1.070	6.907	0.544
530	2.170	8.059	0.278
540	3.397	8.668	0.122
550	4.732	8.855	0.035
560	6.070	8.581	0.001
570	7.311	7.951	0.000
580	8.291	7.106	0.000
590	8.634	6.004	0.000
600	8.672	5.079	0.000
610	7.930	4.065	0.000
620	6.446	2.999	0.000
630	4.669	2.042	0.000
640	3.095	1.290	0.000
650	1.859	0.746	0.000
660	1.056	0.417	0.000
670	0.570	0.223	0.000
680	0.274	0.107	0.000
690	0.121	0.047	0.000
700	0.058	0.023	0.000
710	0.028	0.011	0.000
720	0.012	0.005	0.000
730	0.006	0.002	0.000
740	0.003	0.001	0.000
750	0.001	0.001	0.000
760	0.001	0.000	0.000
770	0.000	0.000	0.000
780	0.000	0.000	0.000
Sum	94.811	100.000	107.304

Table 2 Weights for Illuminant D65*, 1964 Observer†, 20 nm Interval

	X	Y	Z
360	−0.001	0.000	−0.007
380	−0.043	−0.004	−0.200
400	0.378	0.035	1.667
420	3.138	0.320	14.979
440	6.701	1.104	34.461
460	6.054	2.605	35.120
480	1.739	4.961	15.986
500	0.071	8.687	4.038
520	2.183	13.844	1.031
540	6.801	17.327	0.229
560	12.171	17.153	0.002
580	16.465	14.150	−0.003
600	17.230	10.118	0.000
620	12.872	6.012	0.000
640	6.248	2.593	0.000
660	2.126	0.832	0.000
680	0.544	0.210	0.000
700	0.105	0.041	0.000
720	0.023	0.009	0.000
740	0.005	0.002	0.000
760	0.001	0.000	0.000
780	0.000	0.000	0.000
Sum	94.811	100.000	107.304

Notes for Tables 1 and 2

*Illuminant D65 is the daylight standard recommended by the CIE and defines the light from a lightly overcast sky having a correlated colour temperature of 6504°K.

†The 1964 observer data are based on the average amounts of monochromatic red, green and blue lights required to match visual stimulus between 390 and 830 nm, by 76 observers viewing a split field subtending an angle of 10° at the eye.

Reference

(1) McLaren K. "The Colour Science of Dyes and Pigments", Second Edition, (Adam Hilger, Bristol and Boston).

Address for Correspondence

However thoroughly a method may be tested, there is always the possibility of a user discovering a hitherto unknown problem. Users with information this method are requested to write to:

The Secretary
The Standing Committee of Analysts
The Department of the Environment
2 Marsham Street
LONDON SW1P 3PY
England.

Figure I

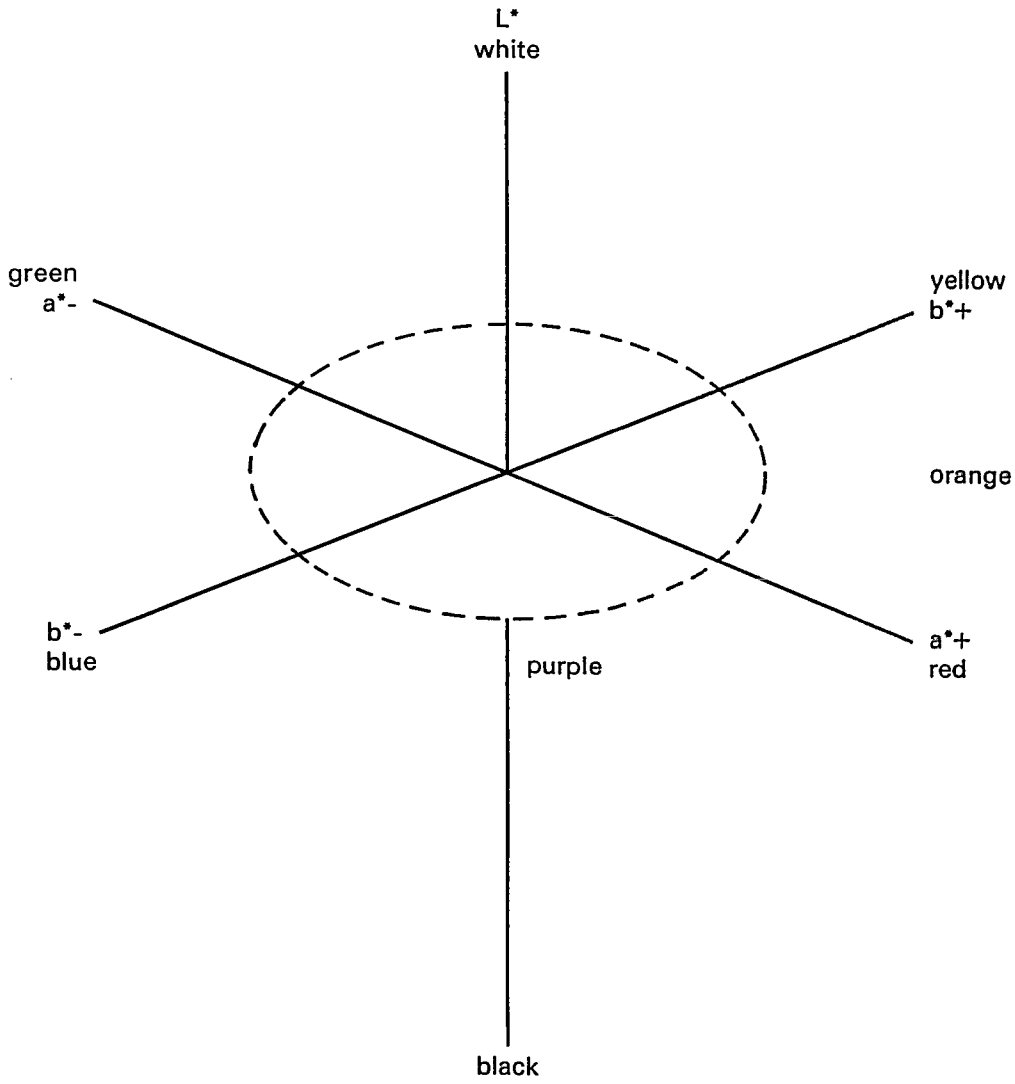
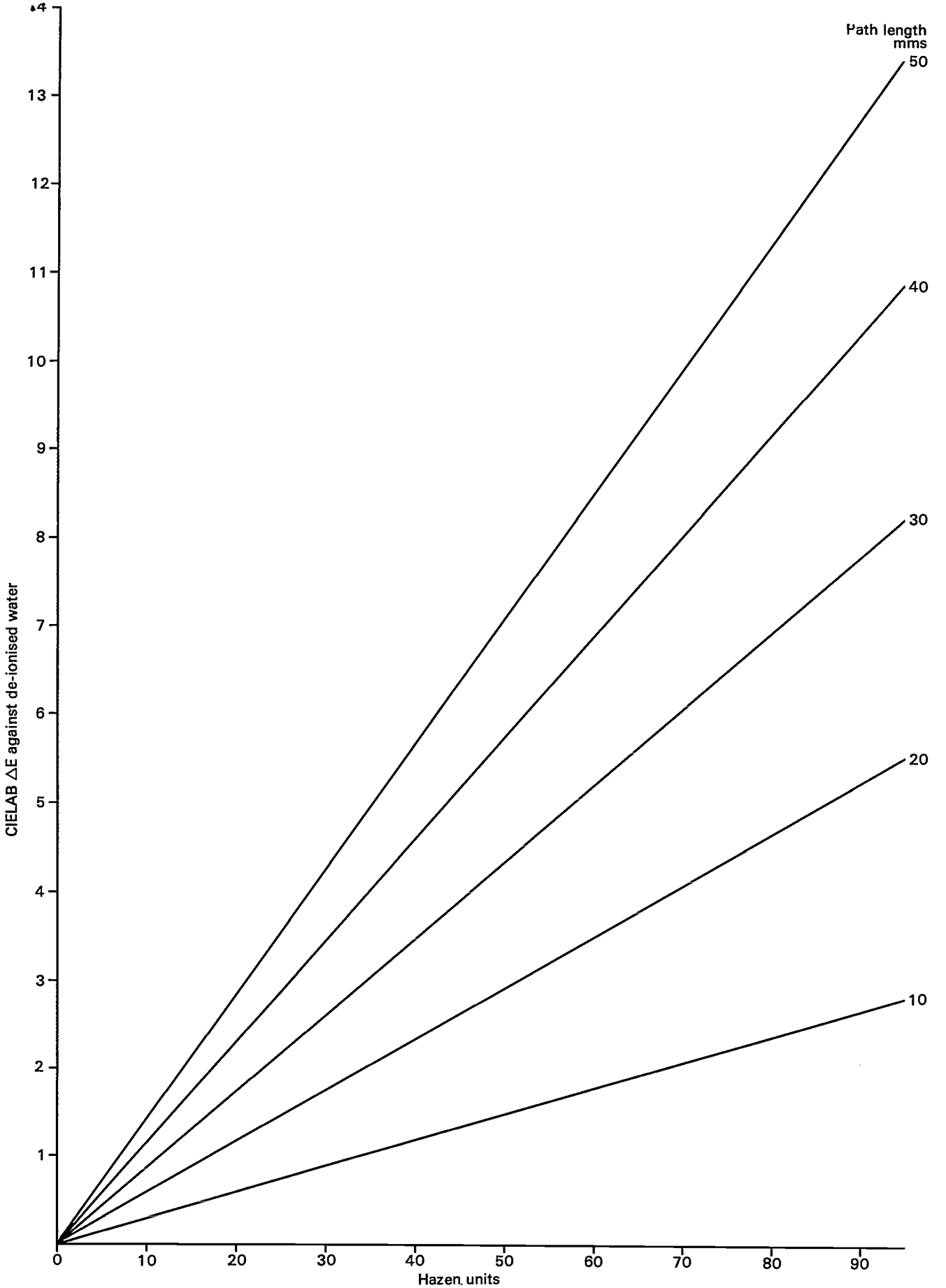


Figure II


Path length mms
50
40
30
20
10
CIELAB ΔE against de-ionised water
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
10
20
30
40
50
60
70
80
90
Hazen units

Department of the Environment

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