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Incorporating
Amendment No. 1

Record inks —

Part 1: Specification for blue-black inks

Committees responsible for this British Standard

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Association of Metropolitan Authorities
British Office Systems and Stationery Federation
HMSO
Public Record Office
Scottish Record Office
Society of Archivists
Society of Dyers and Colourists
Writing Instruments Association

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Foreword

This Part of BS 3484 has been prepared under the direction of the Paper and Printing Standards Policy Committee. It supersedes BS 3484:1962 which is withdrawn.

This specification calls for the use of substances and/or test procedures that may be injurious to health if adequate precautions are not taken. It refers only to technical suitability and in no way absolves the user of this standard from statutory obligations relating to health and safety at any stage of manufacture or use. In particular, attention is drawn to the preparation of the standard reference ink in Appendix A and the determination of colour fastness to light in Appendix E.

A specification for permanent record inks is given in BS 3484-2.

Summary of pages

This document comprises a front cover, an inside front cover, pages i and ii, pages 1 to 6, an inside back cover and a back cover.

This standard has been updated (see copyright date) and may have had amendments incorporated. This will be indicated in the amendment table on the inside front cover.

1 Scope

This Part of BS 3484 specifies performance and certain features of the composition of blue-black ink for archival purposes. Appendix A to Appendix E give details of tests for compliance of the ink with this Part of BS 3484.

NOTE 1 The long-term durability of any document will also depend on the composition of the paper, etc. on which it is written.

NOTE 2 The titles of the publications referred to in this standard are listed on the inside back cover.

2 Definition

For the purposes of this Part of BS 3484 the following definition applies.

2.1
archival record ink

an ink suitable for creating records which are required to remain legible for long periods of time

3 Ink description

The ink shall be of the ferrous gallo-tannate type and shall write with an initial blue colour which develops to an intense black within 14 days. It shall conform in all respects to the standard reference ink described in Appendix A.

4 Ink requirements

4.1 Iron content

When tested in accordance with one of the methods described in Appendix B, the ink shall contain not less than 4.5 g/L and not more than 5.5 g/L of iron, calculated as metallic iron.

4.2 Freedom from sediment

When assessed by the method described in Appendix C, the ink shall show no greater sediment than freshly prepared standard reference ink (see Appendix A).

4.3 Stability

When tested in accordance with the method described in Appendix D, the ink shall be free from deposits, surface growths and scales and shall be at least equal to freshly prepared standard reference ink (see Appendix A) in these respects.

4.4 Performance

When tested in accordance with the methods described in Appendix E, the ink shall show:

a) colour depth and intensity by incident and transmitted light at least equal to freshly prepared standard reference ink (see Appendix A) immediately after drying (see Appendix E);

b) density of colour at least equal to that shown by freshly prepared standard reference ink after development, when tested in accordance with item a) of **E.4**;

c) resistance to water at least equal to that of freshly prepared standard reference ink, when tested in accordance with item b) of **E.4**;

d) resistance to fading not less than that of freshly prepared standard reference ink, when tested in accordance with item c) of **E.4**.

5 Packaging

Each bottle or container shall be legibly marked with the following information:

a) the name, trademark or other means of identifying the manufacturer;

b) the date or date code of manufacture;

c) the number and date of this British Standard, i.e. BS 3484-1:1991[1].

NOTE 1 It is recommended that bottles made from alkali free amber or plain glass should be used. Filled bottles when made from alkali free plain glass should be supplied in cartons or other containers which exclude light. The bottle should be kept in its container and care should be taken that the bottle is not left open after use: the container should carry printed instructions to this effect.

NOTE 2 If stored in accordance with these recommendations the ink will conform with the requirements of this standard for a period of not less than 12 months.

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[1] Marking BS 3484-1:1991 on or in relation to a product represents a manufacturer's declaration of conformity, i.e. a claim by or on behalf of the manufacturer that the product meets the requirements of the standard. The accuracy of the claim is therefore solely the responsibility of the person making the claim. Such a declaration is not to be confused with third party certification of conformity, which may also be desirable.

Appendix A Standard reference ink

A.1 Reagents and apparatus

NOTE All reagents are general purpose grades.

A.1.1 *3,4,5-trihydroxybenzoic acid (gallic acid)* ($C_7H_6O_5$).

A.1.2 *Tannic acid* ($C_{14}H_{10}O_9$).

A.1.3 *Sulphuric acid* $c(H_2SO_4)$ = 18 mol/L.

A.1.4 *Iron(II) sulphate* ($FeSO_4.7H_2O$).

A.1.5 *Phenol* (C_6H_5OH).

A.1.6 *Blue dye*, essentially free from organic and inorganic diluents or impurities [Colour Index[2] 42755 (acid blue 22) or 42780 (acid blue 93)].

A.1.7 *Distilled water*.

A.1.8 *Ordinary laboratory apparatus*

A.2 Preparation

Dissolve 1 g of phenol and 3 g of the blue dye in about 100 mL of distilled water. Prepare 500 mL of hot distilled water and add, while stirring, 6.4 g of gallic acid and 19.5 g of tannic acid. Dissolve 25 g of iron(II) sulphate in 300 mL of distilled water. Carefully add 3 g of sulphuric acid to the gallic and tannic acid solution and then, while stirring, add the iron(II) sulphate solution. Let the mixture stand for 24 h, filter, add the dye solution followed by sufficient distilled water to make up to 1 L. Allow to stand for another 24 h and refilter.

Appendix B Determination of iron content

B.1 Atomic absorption spectrometric method

B.1.1 ***Principle***

A test solution of ink is prepared and aspirated into an acetylene-air flame. The absorption of the 248.3 nm line emitted by an iron hollow-cathode lamp is measured.

B.1.2 ***Reagents***

B.1.2.1 *Standard iron solution*, corresponding to 0.2 g/L of iron.

This solution may be prepared by dissolving 0.200 g of pure iron wire in the smallest possible quantity of hydrochloric acid, with a density of 1.19 g/mL, in a 1 000 mL one-mark volumetric flask, making up to the mark with distilled water, followed by thorough mixing.

1 mL of this solution contains 0.2 mg of iron.

B.1.2.2 *Hydrochloric acid solution*, approximately c(HCl) = 6 mol/L.

B.1.2.3 *Distilled water*

B.1.3 ***Apparatus***

B.1.3.1 *Atomic absorption spectrometer*, fitted with an acetylene and air burner.

B.1.3.2 *Iron hollow-cathode lamp*

B.1.3.3 *Ordinary laboratory apparatus*

B.1.4 ***Preparation of the calibration graph***

B.1.4.1 *Preparation of the standard matching solutions*

In each of a series of six 50 mL one-mark volumetric flasks, place 10 mL of the hydrochloric acid solution and then, respectively, the volumes of the standard iron solution given in Table 1. Dilute to the mark with distilled water and mix.

Table 1 — Standard solutions for calibration graph

Volume of standard iron solution	Corresponding mass of iron
mL	mg
0[a]	0
15	3.0
20	4.0
25	5.0
30	6.0
35	7.0

[a] Blank test on reagents for calibration graph.

B.1.4.2 *Adjustment of the apparatus*

Fit the iron hollow-cathode lamp in the apparatus, switch on the current and allow to stabilize. Adjust the current, sensitivity and the aperture of the slit according to the characteristics of the apparatus. Adjust the waveband in the region of 248.3 nm to maximum absorbance. Adjust the pressure of the acetylene and of the air according to the characteristics of the burner. Adjust the aspiration rate to between 2 mL/min and 4 mL/min.

B.1.4.3 *Spectrometric measurement*

Aspirate the series of standard matching solutions (see Table 1) in succession into the flame and measure the absorbance for each. Take care to keep the aspiration rate constant. Spray water through the burner after each measurement.

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[2] Colour Index, Vol. 4, 3rd edition 1971, published by the Society of Dyers and Colourists, P.O. Box 244, Perkin House, 82 Grattan Road, Bradford BD1 2JB, West Yorks., United Kingdom, and by the American Association of Textile Chemists and Colorists, P.O. Box 12215, Research Triangle Park, North Carolina 27709, USA.

B.1.4.4 *Plotting the graph*

Plot a graph, having the masses of iron in milligrams, see Table 1, (contained in 50 mL portions of the standard matching solutions) as abscissae and the corresponding values for the measured absorbance, reduced by the value of the absorbance measured in the blank test, as ordinates.

B.1.5 ***Blank test***

Carry out a blank test at the same time as the determination following the same procedure and using the same amount of all reagents, but omitting the ink portion.

B.1.6 ***Determination***

To each of two 50 mL one-mark volumetric flasks add 1 mL of the ink, 10 mL of the hydrochloric acid solution and make up to the mark with distilled water. Mix thoroughly and if the solution contains any suspended matter allow this to settle.

Carry out the spectrometric measurements in accordance with **B.1.4.3**, after having adjusted the instrument to zero absorbance against the blank test solution.

B.1.7 ***Expression of results***

From the calibration graph, read the iron content equivalent to the mean absorbance determined in **B.1.6**, multiply by 1 000 and report the result in grams per litre to one decimal place.

B.2 Titrimetric method

B.2.1 ***Principle***

An accurately known quantity of ink is decolourized, titrated with disodium EDTA and the iron content calculated from the titre.

B.2.2 ***Apparatus***

B.2.2.1 *pH meter*

B.2.2.2 *Ordinary laboratory apparatus*

B.2.3 ***Reagents***

B.2.3.1 *Concentrated sulphuric acid,* $c(H_2SO_4) = 18$ mol/L.

B.2.3.2 *Sodium hydroxide solution,* $c(NaOH) = 4$ mol/L.

B.2.3.3 *Sodium acetate solution,* ($NaC_2H_3O_2.3H_2O$) 10 % *m*/*m*.

B.2.3.4 *Potassium permanganate solution* ($KMnO_4$) 2 % *m*/*m*.

B.2.3.5 *5-sulphosalicylic acid solution*, 2 % *m*/*m*.

B.2.3.6 *Disodium EDTA solution,* $c(Na_2C_{10}H_{14}N_2\,O_8) = 0.05$ mol/L.

B.2.3.7 *Distilled water*

B.2.4 ***Procedure***

Weigh accurately to three decimal places about 5 g of ink into a tared 250 mL conical flask. Add 50 mL of distilled water and 2 mL of concentrated sulphuric acid. Add the potassium permanganate solution drop by drop until the flask contents turn a pale yellow colour, i.e. the ink has been decolourized. If the mixture is not clear, heat until any solid dissolves.

Add the sodium hydroxide solution until the pH is approximately 1.8 then add the sodium acetate solution until the pH is in the range 2 to 3.
Add 10 mL of the 5-sulphosalicylic acid solution (the mixture should turn purple) and heat to 40 °C to ensure the complex formation is complete.

Allow to cool then titrate with the disodium EDTA solution to a yellow end-point.

Repeat the procedure for another portion of ink.

B.2.5 ***Calculation and expression of results***

Calculate the iron content of the ink, *I*, in grams per litre from the equation

$$I = \frac{T}{m}\rho \times 2.7925$$

where

T is the mean titre of the two results, (in mL);

m is the mean mass of the two ink samples (in g);

ρ is the density of the ink (in g/mL).

Express the result to one decimal place.

Appendix C Determination of sediment

C.1 Apparatus and reagent

C.1.1 *Test paper holder of acid resistant material*, fitted with stainless steel clips, such that the chromatographic paper is held firmly but not in contact with the surface of the holder and of a size suitable for mounting a test paper 200 mm long and 20 mm wide. Figure 1 illustrates a suitable design of holder.

C.1.2 *Chromatographic paper*[3)]

C.1.3 *pH meter*

C.1.4 *Coverable, acid resistant container*, of sufficient size to contain the test paper holder.

C.1.5 *Ordinary laboratory apparatus*

C.1.6 *Sulphuric acid solution*, adjusted to the pH of the ink.

[3)] Whatman 3MM has been found to be suitable.

C.2 Preparation of chromatograms

C.2.1 Cut a strip of chromatographic paper about 200 mm long and about 20 mm wide. About 30 mm from one end lightly mark a cross in the centre with a blunt, soft leaded pencil, so as not to damage the surface of the paper. Make a mark 70 mm from the cross by lightly drawing a line across the paper. Mount the paper in the holder with the cross towards the lower clip.

C.2.2 Shake the ink under test thoroughly and by means of a suitable pipette or eye dropper draw up approximately 1 mL of the ink sample. Without actually touching the paper surface apply one drop of approximately 0.1 mL of the ink to the cross marked on the paper. Immediately place the holder and sample in the vessel, filled previously with the sulphuric acid solution to a depth almost reaching the marked cross. Cover the container and leave until the liquid front reaches the upper mark on the paper. Remove the holder from the acid solution and unclip and lightly blot the test strip with clean tissue or blotting paper. Repeat this procedure with the standard reference ink and compare the chromatograms for evidence of sediment.

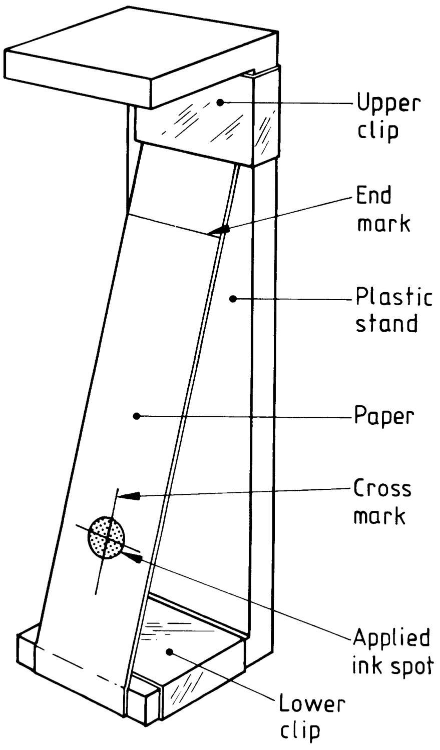

Figure 1 — Design of suitable holder

Appendix D Determination of stability

D.1 Apparatus

D.1.1 *Cellulosic filter paper*

D.1.2 *Ordinary laboratory aparatus*

D.2 Method

Take 25 mL of the ink and place in a 50 mL unlipped beaker of approximately 4 cm diameter. Cover the beaker with a 5.5 cm cellulosic filter paper[4] and place a 5 g to 10 g mass on it to maintain the filter paper flat and in good contact with the lip of the beaker. Expose to the standard atmospheric conditions of 23 °C and 50 % r.h. (see BS EN 20187) for 14 days.

Carry out a simultaneous procedure with freshly prepared standard reference ink (see Appendix A).

At the end of the 14 day period examine both samples for mould growth, scale on the surface of the fluid and deposits on the side and bottom of the beaker.

Appendix E Determination of ink performance

E.1 Apparatus

E.1.1 *Woodfree bond paper*

E.1.2 *Smooth board and drawing pins*

E.1.3 *Ordinary laboratory apparatus*

E.2 Preparation of test samples

Take a piece of white 80 g/m^2 woodfree bond paper, in a size of approximately 250 mm × 200 mm cut so that the machine direction runs parallel to the longer side. Place it on a piece of smooth board with the wire side of the paper against the board and secure it with two drawing pins. Tilt the board at an angle of 45° with the short edge resting on the bench and support it in this position for the duration of the test.

Measure 0.5 mL of the ink in a graduated 1 mL pipette, allow the tip of the pipette just to touch the paper at the top edge and release the ink so that it can flow down the paper to give a stripe of uniform width without cockling or causing waviness in the paper. The stripe of ink shall be similar in width to that of the standard reference ink. When dry, remove the paper from the board, cut off and discard the bottom 25 mm of paper and cut the remainder into 50 mm strips across the lines of ink. Make similar tests with the standard reference ink.

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[4] Whatman No. 1 has been found to be suitable.

E.3 Visual examination before development

The stripes of ink shall be visually examined immediately for colour depth and intensity by incident light and by transmitted light.

E.4 Performance after development

Expose the stripe for 14 days to air, out of direct sunlight, at room temperature, to allow full development to a dense black colour. Then examine for the following.

a) Density of colour. The fully developed stripes shall be examined visually by incident and transmitted light.

b) Resistance to immersion in water for 24 h.

c) Resistance to fading after exposing the stripes for 48 h in accordance with method B02 of BS 1006:1990.

blank

Publication(s) referred to

BS 1006, *Methods of test for colour fastness of textiles and leather*.

BS EN 20187, *Paper, board and pulps — Standard atmosphere for conditioning and testing and procedure for monitoring the atmosphere and conditioning of samples*.

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