ETHIOPIAN STANDARD

ES 3343:2021

Second edition
xx-xx-2021

Specification for Vat Green 1 for Textile Industry

ICS: 59.080.01

Published by Ethiopian Standards Agency

Foreword

This Ethiopian Standard has been prepared under the direction of Technical Committee for Sizing & Finishing material,Dye stuff & Pigments (TC 71) and published by the Ethiopian Standards Agency (ESA).

This Ethiopian Standard cancels and replaces ES 3343:2007(Reaffirmed 2018) *Specification for vat 1 for textile industry*.

In preparing this Ethiopian Standard reference has been made to the following:

IS 12547:1988 “Specification for Vat green 1 for textile Industry", published by Indian Standard Institution.

Acknowledgment is made for the use of information from the above publication.

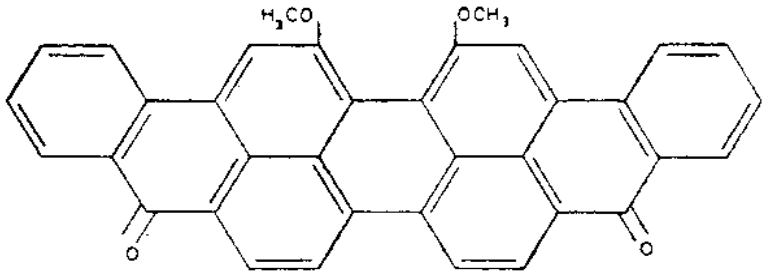
Specification for Vat Green 1 for Textile Industry

1. Scope

This Ethiopian Standard specifies the quality requirements for Vat Green 1 used in textile industry.

2 Chemical constitution

2.1 The chemical structure of the dyestuff shall be as given below:



Note - The method for the preparation of the dye, chemically known as 16, 17-dimethoxydibenzanthrone, is given according to DRP 411,013 in the book titled 'Chemistry of Synthetic Dyes' by K. Venkataraman, Vol II, P 971.

3 Physical state

3.1 The dyestuff shall be a dispersed mixture of the dye with auxiliary substances supplied in the form of a dustless powder with the dye content value as agreed to between the buyer and the seller when tested by the method prescribed in Annex B. The colour of the dye shall be brilliant green.

4 Identification

4.1 The color of sodium salt of leuco vat dye prepared as prescribed in Annex C shall be blue, and the colour of vat acid prepared as prescribed in Annex C shall be bright red.

4.2 With concentrated sulphuric acid, the dye shall give a reddish violet coloured solution which on further dilution gives a green precipitate.

4.3 The dye shall be soluble in warm tetrahydronaphthalene and insoluble in alcohol, chloroform and toluene.

5 Standard dyestuff

5.1 In order to compare the strength, tint, wettability and spreading in water; and rate of development and rate

of fixation of the dyestuff under test, a standard sample of the dyestuff shall be sealed according to the agreement between the buyer and the seller. The strength of the standard sample of dyestuff shall be assumed to be 100 percent. The strength of the dyestuff under test shall be determined by the method prescribed in IS 4394: 1988.

5.1.1 The standard sample shall be kept in hermetically closed glass vessels in a dry dark place and shall be replaced by fresh one once every year.

6 Requirements

6.1 The dyestuff shall meet the physico-chemical requirements given in Table 1 and 7.2 to 7.5.

6.2 Colour fastness properties

The minimum colour fastness ratings of the dye on cotton yarn hanks dyed and after-treated by the method A/QI prescribed in IS 4394: 1988 shall satisfy the requirements given in Table 2.

6.3 Migration properties of dye

The color fastness ratings to staining of adjacent white cotton yarn hank, when tested by the method prescribed in Annex H shall be as follows:

Temperature	Rating for Staining
50 ^{o} C	1
80 ^{o}C	3 - 4

6.4 Strike of dye

The difference in colour of the hanks, when tested by the method prescribed in Annex J, shall be equal to grey scale grade 2-3.

6.5 Leveling properties of dye

The difference in colour of the hanks, when tested by the method prescribed in Annex K, shall be equal to grey scale grade 3-4.

Sl No	Characteristic	Requirement	Method of Test
i)	Strength of dye with respect to standard specimen, percentage	100 ± 2	ES 3321
ii)	Tint	Like the standard specimen	Visual examination on dyed yarn samples (see NOTE)
iii)	Filtering capacity limit of water suspension, Min, ing/100ml	0.6	Annex D
iv)	Wettability and spreading of dye in water	Corresponding to the standard specimen	Annex E
v)	Rate of development and fixation of dye on cotton fibre	Corresponding to the standard specimen	Annex F
vi)	Redox potential of dye in mV, Min	742	Annex G
	NOTE :- The assessment of tint may also be done instrumentally, wherever facilities are available		

7 Packing and marking

7.1 For smaller packing, the dyestuff in powder form shall be packed in polyethylene bags which shall be

sealed and put in rustproof mild steel containers of 0.5kg or 1.00 kg capacity and having an opening of 7'5 cm or 13'0 cm (circumference). Each container shall be made leakageproof by proper sealing of the end or corners, each container shall be provided with a temper proof, spillproof and pilferproof sealing so as to avoid adulteration at intermediate stages. Such sealing device may consist of a mild steel ring and a plastic seal to be fitted around the neck of the container after seaming the capseal. The seal may also bear the monogram, the serial number and different columns at'intervals making the replacement not very easy. A suitable number of such containers shall then be packed in wooden cases or HDPE containers lined with cushioning of corrugated paper board/synthetic cushion materials so as to mitigate shock and/or to protect surfaces from abrasion and/or to position an article in a receptacle.

7.2 The dyestuff in emulsion or paste form shall be packed in 5.0 or 10.0 kg capacity container/ drum made of HDPE or polypropylene or corrugated fibre board lined with polyethylene or polyester film resins or with Kraft paper combined with thermoplastic adhesives. The containers shall have an opening of 18.0 cm or 28.0 cm (circumference) for convenient scooping out of the dyestuff at the time of use. Each container shall have a tamperproof, spillproof and pilfer proof sealing as described for small containers.

7.3 For medium packing of dyestuff in the range of 25 to 200 kg, drums or containers as described for dyestuff in emulsion or paste form shall be used.

7.4 For bulk packing of dry 'flowable' powder of dyestuff to contain up to 2000 kg of dyestuff, bulk bags made of HDPE or bulk boxes made from triple wall or two or more thicknesses of double wall corrugated fibre board laminated in the side walls by polyethylene or polyester film resin shall be used.

7.5 In cases of packing of dyestuff for export, in addition to the above, the packing shall be such that it meets the requirements laid in the contract and also the requirements of shipping corporations (national and international) and the insurance obligations to safeguard against unfortunate incidents. The packing shall be such that it is not damaged during transhipment, airfreight or transport, and during loading and unloading.

7.6 Each package shall be marked with the following:

a) Name and class of the dyestuff according to colour index (3rd edition);

b) Dye content of dyestuff;

c) Strength of dyestuff;

d) Month and year of manufacture and product batch number;

e) Trade name and for trade-mark of the manufacturer, if any; and

f) Net mass of each package.

Table 2 Minimum Colour Fastness Rating of the Dye on cotton Fibre (Clause 6.2)

Percent Dye	Light	Weathering by Xenon Arc Lamp	water	Washing Test 3	Washing Test 5	Preparation Acidic and Alkaline	Hot Pressing	Rubbing		Bleaching with Hypochlorite	Bleaching with Peroxide	Mercerizatoin	Soda Boil
								Dry	Wet			150^0 c	180^0 c
(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)	(9)	(10)	(11)	(12)	(13)	(14)
0.3	5-6	4-5	5(5)	5(5)	4-5 (4)	4-5(4)	4-5(4)	(5)	(4)	4	4-5(4)	4-5(4)	4-5(4)
1.8	6-7	5-6	5(5)	5(5)	4-5 (4)	4-5(4)	4-5(4)	(4)	(4)	4-5	4-5(4)	4-5(4)	5(5)
3.6	7	6	5(5)	5(5)	4(4)	5(4)	5(4-5)	(4)	(4)	4-5	4-5(4)	5(4)	5(5)
Method of Test	IS2454: 1985	IS 6152: 1985	IS 767: 1988	IS 764: 1979	IS 3417:1979	IS 971:1983	IS 689: 1988	IS 766:1988		IS 762:1988	IS 763:1988	IS 979:1988	IS 973: 1988

NOTE: The figures without bracket indicate the colour fastness rating for change in colour while those with bracket indicate the colour fastness rating for staining of adjacent fabrics.

8.6.1 Each package may also be marked with the information required by any other law in force, for example, excise regulations.

8.6.2 Each package may also be marked with the Standard Mark.

9 SAMPLING 9.1 Lot

All the packages of the same dye having the same dye content delivered to a buyer against one despatch note shall constitute a lot.

9.2 Unless otherwise agreed to between the buyer and the seller, the number of containers to be selected at random from a lot shall be as given in Table 3

Table 3 Sample Size and Crtieria for Conformity

Clause 8.2, 8.3, 8.4 and 8.5

Lot size (Packages)	Sample Size (Packages)	Permissible Number of Non-conforming Packages	Sub-sample Size (Packages)
1	2	3	4
Up to 100	10	1	5
101 to 300	15	2	6
301 to 500	25	3	7
501 to 800	35	4	8
801 to 1300	50	5	9
1301 and above	75	6	10

ANNEX A

(Clause 3.1)

DETERMINATION OF DYE CONTENT

A-1 Take a test specimen of the dyestuff of about 1 g in a weighing bottle and dry it to constant mass in a drying oven at 105 ±3°C. Cool the specimen in a desiccator charged with self-indicating silica gel or calcium chloride and obtain the oven-dry mass of the specimen to an accuracy of 0.000 2 g with the help of an analytical balance.

A-2 Prepare leuco vat of the dye as prescribed in Annex C and filter the solution on a Buchner funnel through Whatman filter paper No. 41 under low pressure of 320 mm of mercury column. Repeat the procedure twice more with the residue left and collect all the filtered solution in a beaker.

A-3 Oxidize the solution obtained in A - 2 by bubbling a strong current of air through it for about 20 minutes and filter the solution through a weighed sintered filter crucible. Repeat the procedure twice more with the solution left and collect and the residues on sinteredfilter crucible. Dry the crucible and the residue to constant mass in a drying oven at 105± 3°C. Cool in a dessicator charged with self-indicating silica gel and weigh them to obtain the oven dry mass of the residue.

7-4 Calculate the dye content, percent (P) by the formula

$$P = \frac{m_1}{m_2} \times 100$$

Where

m_1 is the oven-dry mass of the residue, and

m_2 is the oven–dry mass of the specimen taken.

ANNEX B
(Clause 4.1)
PREPARATION OF LEUCO V AT AND ACID V AT OF THE DYE

B-I PREPARATION OF LEUCO VAT

Prepare a paste of 0.1g of dye in Turkey red oil or a suitable wetting agent in a beaker. Add 20 ml of hot water (50°C) to the paste with constant stirring. Then add the required quantity of sodium hydroxide solution and sodium hydrosulphite *(see* IS 4394: 1988) with constant stirring and allow to stand for 5 to 10 min.

B-2 PREPARATION OF ACID VAT

Prepare a leuco vat of the dye. Add an acid stable dispersing agent and glacial acetic acid slowly with constant stirring for about 5 minutes, which gives acid vat of the dye in which the dye is precipitated in very finely dispersed state.

NOTE - Instead of preparing the leuco vat and then precipitating the dye by oxidation, the dye may be dissolved in cold concentrated sulphuric acid and then precipitated by dilution with water.

ANNEX C

(Table 1)

DETERMINATION OF FILTERING CAPACITY LIMIT OF WATER SUSPENSION OF THE DYE

C-1 Take 0.5 to 1.0 g samples of the dye in steps of 0.1 g and weigh them correct to 0.01 g. Introduce the samples cups of 150 ml capacity containing 50ml of distilled water heated to 70°C. Under stirring, add the dye in each cup and then make the volume in each cup up to 100 ml by adding distilled water at 10°C with constant stirring. Place the suspension in each cup for 5 minutes on a water-bath at 70°C with constant stirring. Then cool the suspension and filter on a Buchner funnel through a "Whatman filter paper No. 41 under low pressure of 320 mm of mercury column.

C-2 Take that mass of the sample as filtering capacity limit for which the filter paper is uniformly coloured and the residues of the dye are visible in the place over the holes of the funnel.

ANNEX D

(Table 1)

DETERMINATION OF WETTABILITY AND SPREADING OF DYE IN WATER

D-1 Introduce 0.02g of the test dye and the same amount of the standard sample of dye in separate glass *cups* of 200 ml capacity, in each of which 100 ml of distilled water has been added. The test dye shall get wet and spread in the water at least as much as the standard sample of dye.

ANNEX E

(Table 1)

DETERMINATION OF RATE OF DEVELOPMENT AND FIXATION OF DYESTUFF

E-I Carry out dyeing of a hank with the dye as prescribed in Method A/QI of ES 3321

E-2 Take another five hanks and (dye as given in E-I except that the amount of dye and chemicals shall De double to that taken in E-l, and times of dyeing shall be 5, 10, 15, 20 and 25 minutes for each hank respectively.

E.3 Match the samples-obtained in E-2 with that of E-1 and not the time of dyeing for the sample obtained in F.2 which exactly matches' with the sample in F-1.

E-4 Record the time of dyeing noted in E- 3 as the time of half dyeing for the dyestuff.

NOTE – The time of half dyeing is a measure of rate of development and fixation of the dyestuff

ANNEX F

(Table 1)

DETERMINATION OF CHARACTERISTIC REDOX POTENTIAL OF DYE

F-I PRINCIPLE

The fully reduced vat dye in aqueous alkaline solution in the presence of known amount of sodium hydrosulphite powder is titrated potantiometrically against a standard solution of potassium ferricyanide. The 'value of potential is calculated and termed as 'characteristic redox potential'.

F-2 TITRATION APPARATUS

F-2.1 The potentiometer titration is carried but in the apparatus described in- Pig. 1. It consists of a round-bottomed Pyrex flask of one 'liter capacity provided with a central neck *A* and six other narrow necks Al to *A6* disposed symmetrically around *A.* The central neck carries the socket of a B-24 quick fit joint in which is fitted a standard metal gland carrying a link stirrer, *L,* through a reducer (B-24-B-19). The six narrow side necks carry sockets of B-19 joint. In *Al* is fitted a microburette B, graduated to 0.02 ml and complete with side reservoir for the standard solution of the oxidizing agent, potassium ferricyanide, In A_2 and A_3, fit two electrode containers through which two platinum electrodes E_1 and E_2 are passed (A_3 and E_2 cannot be seen in the figure), The electrode consists of 2 cm length of bright platinum wire (2 mm in diameter) used at the end of a long, narrow, and soft glass tube, Mercury is added to the tube to facilitate connection. A_4 contains the connecting salt bridge of a mercuric oxide half-cell, The emf of the half-cell is experimentally determined using a saturated calomel electrode using 0.1 N potassium chloride bridge and is found to be 168 m V at 30°C, *A s* in the figure contains an inlet tube for nitrogen gas, and an outlet for the gas is provided, A_6 (not seen) is provided for facilitating the addition of sodium hydrosulphite required for reducing the dye and is provided with a stopper, The flask is immersed in a thermostat kept at 30 ± 0.1°C.

F-3 REAGENTS

F-3.1 Caustic Soda Solution

0.1 N.

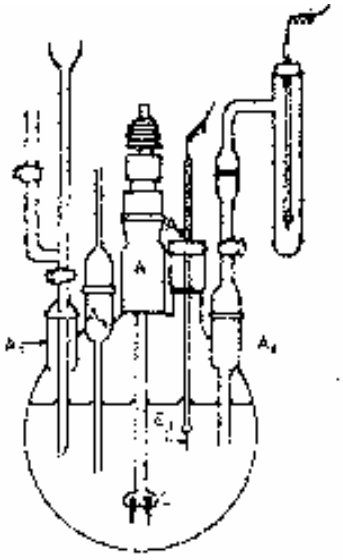


FIG 1 TITRATION APPARATUS

F-3.2 Neutral Potassium Ferricyanide Solution 0.2 N (ANALAR Grade).

F-3,3 Sodium Hydrosulphite Powder- Of minimum 85 percent active matter content.

F-3.4 Concentrated Sulphuric Acid

F-3.5 Vat Green 1 Solution (0.5 Percent m/v) Prepared in concentrated sulphuric acid.

F-4 PROCEDURE

F-4.1 Assemble the apparatus as indicated in Fig, 1. Remove all the air from the apparatus by a rapid current of oxygen-free nitrogen. Transfer 600 ml of air-free distilled water to the titration apparatus, Add to it 1 ml of 0'5 percent solution of the dye in concentrated sulphuric acid while stirring. Then add required quantity of sodium hydroxide solution so that the final solution is 0.1 N, Bubble oxygen-free nitrogen rapidly through this dispersion for about one hour, after this period, add 200 mg of sodium hydrosulphite powder. Titrate this solution with potassium ferricyanide solution by adding 0.0, 0.5, 0.7, 0.8, 0.9, 1.0, 1.10, 1.20, 1.70, 2.70, 3.70, 4.70, 5.70 and 6.70 ml of it, and measure the potential of the cell consisting of the mercuric oxide half-cell and the platinum electrode after each addition.

F-4.2 Construct a titration curve of the observed potential and the volume of potassium ferricyanide solution used as indicated in Fig. 2.

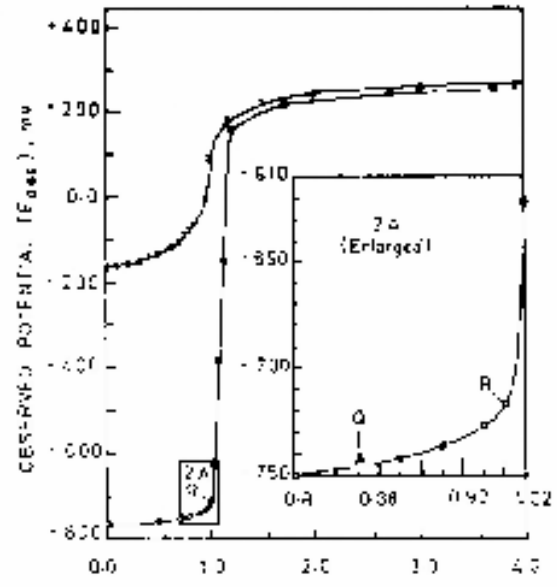


FIG.2 Volume of $K_aF_e(CN)_6$ Solutions ADDED, ml

F.5 CALCULATIONS

F-5.1 Calculate the concentration of leuco vat dye by the formula:

$$[A] = (Concentration\ \ of\ \ dye\ in\ moles/litre) \times \frac{V_1}{V_2}$$

Where

[A] is the Concentration of leuco vat dye in moles/litre,

V_1 is the volume of K_3 (CN) $_6$ used for unoxidized leuco dye, and

V_2 is the volume of K_3 (CN) $_6$ used for complete oxidation f total leuco vat dye.

F.5.2 Calculate the characteristic redox potential of the dye by the formula:

$$E_c = E_{obs} + \frac{RT}{2F} In[A]$$

Where

E_c is the characteristic Redox potential of dye in mV,

E_{obS} is the observed potential of dye in mV,

R is the universal gas constant and its value is 8.31 joule $k^{-1}mole^{-1}$

T is the absolute temperature, and

F is the number of coulombs in one Faraday and its value is equal to 96500.

ANNEX G

(Clause 7.3)

DETERMINATION OF MIGRATION PROPERTIES

G-1 Carry out the treatments of an already dyed and a white hank of cotton yarn at 50°C for 40 minutes- at a liquor ratio of 30: 1 in a blank dye bath simultaneously by the method A/QI as prescribed in ES 3321. Also after treat the hanks as prescribed in ES 3321.

NOTE - The blank dye bath contains all other chemicals except the dyestuffs and the common salt.

G.2 Similarly carry out treatment of another pair of a dyed and a white hank of cotton yarn at 80°C in a blank bath for 40 minutes at a liquor ratio of 30: 1 and after-treat as given in H-I.

G.3 Assess the staining of the white-treated hank by comparing with the dyed-treated hank with the help of grey scale for staining *(see* ES ISO 105-A02 and ES ISO 105-A03: 2005).

ANNEXH

(Clause 6.4)

DETERMINATION OF STRIKE

H-I Prepare dye bath of 5 percent depth for a 5 g cotton yarn hank as prescribed in ES 3321. Enter the hank in dyebath, and turn steadily and continuously. After 2 minutes, enter a second 5 g hank in the dyebath and turn the hanks in the same way. After next 3 minutes, remove both the hanks and after-treat them by the procedure given in IS 4394: 1988.

H.2 Assess the difference in colour of the two hanks by the greedy scale for change in color *(see* ES ISO 105-A02 and ES ISO 105-A03: 2005).

ANNEX I

(Clause 6.5)

DETERMINATION OF LEVELLING PROPERTIES

I-1 Prepare a dyebath as given in **J-1**. Enter a 5 g hank of cotton yam' in the dye bath and turn steadily and continuously. After 2 minutes, enter a second 5 g hank and turn both the hanks in the same way for next 3 minutes. Thereafter turn both the hanks for 1 min after every 5 min. After further 35 min (that is a total of 40 min), remove both the hanks and after-treat them as given in ES 3321.

I.2 Assess the difference in colour of the two hanks by the grey scale for change in colour (*see* ES ISO 105-A02 and ES ISO 105-A03: 2005).

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Ethiopian Standards Agency

Organization and Objectives

The Ethiopian Standards Agency (ESA) is the national standards body of Ethiopia established in 2010 based on regulation No. 193/2010.ESA is established due to the restructuring of Quality and Standards Authority of Ethiopia (QSAE) which was established in 1998.

ESA's objectives are:-

- ❖ Develop Ethiopian standards and establish a system that enable to check whether goods and services are in compliance with the required standards,
- ❖ Facilitat the country's technology transfer through the use of standards,
- ❖ Develop national standards for local products and services so as to make them competitive in the international market.

Ethiopian Standards

The Ethiopian Standards are developed by national technical committees which are composed of different stakeholders consisting of educational institutions, research institutes, government organizations, certification, inspection, and testing organizations, regulatory bodies, consumer association etc. The requirements and/ or recommendations contained in Ethiopian Standards are consensus based that reflects the interest of the TC representatives and also of comments received from the public and other sources. Ethiopian Standards are approved by the National Standardization Council and are kept under continuous review after publication and updated regularly to take account of latest scientific and technological changes. Orders for all Ethiopian Standards, International Standard and ASTM standards, including electronic versions, should be addressed to the Documentation and Publication Team at the Head office and Branch (Liaisons) offices. A catalogue of Ethiopian Standards is also available freely and can be accessed in from our website.

International Involvement

ESA, representing Ethiopia, is a member of the International Organization for Standardization (ISO), and Codex Alimentarius Commission (CODEX). It also maintains close working relations with the international Electro-technical Commission (IEC) and American Society for Testing and Materials (ASTM).It is a founding member of the African Regional Organization for standardization (ARSO).

<u>More Information</u>?
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