

THAI INDUSTRIAL STANDARDS
Synthetic Dyestuff: Vat Dye

1. Scope

- 1.1 This standard covers synthetic dyestuff: vat dye, hereinafter called “vat dye”.

2. Definition

For the purpose of this standard, the following definitions apply:

- 2.1 “Synthetic dyestuffs” means synthesised colouring substances that can be applied to material directly or through the reactive dyeing or printing process.
- 2.2 “Vat dye” means a synthetic dyestuff, with the composition of the keto group, generally insoluble. Before dyeing, it shall be changed into a water-soluble form through an alkali reduction process and subsequently oxidised to an insoluble form.
- 2.3 “Colour difference” (ΔE) means the variation of a colour from its reference standard specified by the manufacturer.

3. Requirements

- 3.1 Safety requirements
- 3.1.1 Hazardous aromatic amines
No hazardous aromatic amine (as listed in Table 1) may be split out.

Table 1 List of hazardous aromatic amines
(Clause 3.1.1)

No.	CAS number	Substances
1	92-67-1	Biphenyl-4-ylamine 4-aminobiphenyl xenylamine
2	92-87-5	benzidine
3	95-69-2	4-chloro-o-toluidine
4	91-59-8	2-naphthylamine
5	97-56-3	o-aminoazotoluene 4-amino-2',3-dimethylazobenzene 4-o-tolylazo-o-toluidine
6	99-55-8	5-nitro-o-toluidine
7	106-47-8	4-chloroaniline
8	615-05-4	4-methoxy-m-phenylenediamine
9	101-77-9	4,4'-methylenedianiline 4,4'-diaminodiphenylmethane
10	91-94-1	3,3'-dichlorobenzidine 3,3'-dichlorobiphenyl-4, 4'-ylenediamine
11	119-90-4	3,3'-dimethoxybenzidine o-dianisidine
12	119-93-7	3,3'-dimethylbenzidine 4,4'-bi-o-toluidine
13	838-88-0	4,4'-methylenedi-o-toluidine
14	120-71-8	6-methoxy-m-toluidine p-cresidine
15	101-14-4	4,4'-methylene-bis-(2-chloro-aniline) 2,2'-dichloro-4,4'-methylene-dianiline
16	101-80-4	4,4'-oxydianiline
17	139-65-1	4,4'-thiodianiline
18	95-53-4	o-toluidine 2-aminotoluene
19	95-80-7	4-methyl-m-phenylenediamine

Table 1 List of hazardous aromatic amines (as above)

No.	CAS number	Substances
20	137-17-7	2,4,5-trimethylaniline
21	90-04-0	o-anisidine 2-methoxyaniline
22	60-09-3	4-aminoazobenzene p-aminoazobenzene
23	95-68-1	2,4-xylidine
24	87-62-7	2,6-xylidine

3.1.2 Free aromatic amines

These shall not exceed 150 mg/kg.

The test method shall be in compliance with Clause 7.1.1.

3.1.3 Heavy metal contamination

Heavy metal contamination shall not exceed the criteria set in Table 2.

The test method shall be in compliance with Clause 7.1.2.

Table 2 Volume of heavy metal contamination
(Clause 3.1.3)

Heavy metal	Criteria mg/kg
Lead	100
Mercury	4
Cadmium	20
All Chromium	100
Chromium (VI)	25
Copper	250
Nickel	200
Cobalt	500

Note: In the event that the vat dye has a complex heavy metal composition, the manufacturer shall notify the Thai Industrial Standards Institute and shall be exempted from testing for heavy metals.

3.2 Moisture content (if the substance is a solid)

The moisture content shall not exceed 3%.

The test method shall be in compliance with Clause 7.2.

3.3 Colour difference

The colour difference shall not exceed 0.5 CMC units.
The test method shall be in compliance with Clause 7.3.

3.4 Colour fastness to light (xenon arc lamp)

Upon testing under Clause 7.4, the colour fastness to light shall be in compliance with the provisions set out in the manufacturer's instruction manual, with the maximum deviation not exceeding half the specified value*.

*Note: * If the specified value is 3, the level obtained from the test shall not be less than 2-3 or, if the specified value is 3-4, the level obtained from the test shall not be less than 3.*

3.5 Colour fastness to washing

Upon testing under Clause 7.5, the colour fastness to washing shall be in compliance with the provisions set out in the manufacturer's instruction manual, with the maximum deviation not exceeding half the specified value*.

*Note: * If the specified value is 3, the level obtained from the test shall not be less than 2-3 or, if the specified value is 3-4, the level obtained from the test shall not be less than 3.*

3.6 Colour fastness to bleaching agents

Upon testing under Clause 7.6, the colour fastness to bleaching agents shall be in compliance with the provisions set out in the manufacturer's instruction manual, with the maximum deviation not exceeding half the specified value*.

*Note: * If the specified value is 3, the level obtained from the test shall not be less than 2-3 or, if the specified value is 3-4, the level obtained from the test shall not be less than 3.*

3.7 Colour fastness to rubbing

Upon testing under Clause 7.7, the colour fastness to rubbing shall be in compliance with the provisions set out in the manufacturer's instruction manual, with the maximum deviation not exceeding half the specified value*.

*Note: * If the specified value is 3, the level obtained from the test shall not be less than 2-3 or, if the specified value is 3-4, the level obtained from the test shall not be less than 3.*

4. Packing

4.1 Vat dye shall be packed in a clean, dry and tightly sealed container that does not react with vat dye.

4.2 The net weight of vat dye in each container shall not be less than that specified on the label.

5. Marking and labelling

- 5.1 Each container shall bear as a minimum numbers, letters or marks indicating the following information clearly and legibly:
- (1) The name of the product that is subject to this standard or another name referring to the product that is subject to this standard
 - (2) The name of the dye, consisting of its trade name and colour shade, such as Augus Yellow 3R
 - (3) Dye concentration as a percentage (if appropriate)
 - (4) Net weight in kilograms
 - (5) Serial number and year of production
 - (6) Name of manufacturer or factory with its location or registered trademark
 - (7) Hazard warnings as appropriate, such as “Containing toxic substance”, “Do not consume”.
- 5.2 The manufacturer’s instruction manual shall detail as a minimum the following properties of the dye:
- (1) Whether the dye is in powder, solid, liquid, or any other form
 - (2) Samples of fibre showing the level of dye attached to fibres at different concentrations
 - (3) Colour fastness to light
 - (4) Colour fastness to washing
 - (5) Colour fastness to bleaching agents
 - (6) Colour fastness to rubbing
 - (7) The user instructions shall contain, as a minimum, the dyeing method.
- 5.3 If a foreign language is used, the meaning shall correspond to the meaning in Thai.

6. Sampling and criteria for conformity

- 6.1 Sampling and criteria for conformity shall comply with Appendix A.

7. Testing

7.1 Safety requirements

7.1.1 Free aromatic amines

The specimen of 0.1 g vat dye shall be tested in accordance with the method specified in BS EN 14362-1.

7.1.2 Heavy metal contamination

The specimen shall be prepared in accordance with EPA Method 3050B and analysed to detect the volume of heavy metal using Atomic Absorption Spectrometry (AAS) or Inductively Coupled Plasma Atomic Emission Spectrometry (ICP), Spectrophotometry technique.

7.2 Moisture content (if the substance is a solid)

7.2.1 Equipment

7.2.1.1 Measuring equipment with 0.0001 g resolution

7.2.1.2 Temperature-controlled oven

7.2.1.3 Measuring bottle

7.2.1.4 Desiccator

7.2.2 Method

The measuring bottle shall be heated at a temperature of $60 \pm 2^\circ\text{C}$ until the volume is stable. It shall then be measured to read the exact weight. A 10 g vat dye specimen shall be put in and measured before heating at $60 \pm 2^\circ\text{C}$ until the volume is stable. Leave to cool in the desiccator before weighing.

7.2.3 Calculating method

The moisture content shall be calculated using the following formula:

$$\text{Moisture content \%} = \frac{m_0 - m_1}{m_0} \times 100$$

Where m_0 is the weight of the vat dye specimen before heating (in grams)

m_1 is the weight of the vat dye specimen after heating (in grams)

7.3 Colour difference

7.3.1 Equipment

7.3.1.1 Measuring equipment with 0.0001 g resolution

7.3.1.2 Dyeing device with at least 2 containers capable of dyeing the fibre specimens at the same time and controlling the temperature of the dyeing liquid (with a deviation not exceeding $\pm 1^\circ\text{C}$)

7.3.1.3 Colorimeter which shall use the Illuminant D65 with an opening for a specimen with a diameter or width and length of not less than 2 cm and which shall be capable of indicating colour differences in the specimen to a resolution of 0.1 CMC units.

7.3.1.4 White cross-pattern cotton woven with cotton yarn sized 9.8 text or 14.8 text, bleached, washed, and free from starch and contaminants. In each test, specimens of the same cotton type shall be used.

7.3.1.5 A sample of vat dye chosen as the standard by the manufacturer shall be given to the Thai Industrial Standards Institute at the beginning for use as a standard reference for colour comparison with the specimen.

7.3.2 Dyeing method

7.3.2.1 A piece of cotton fibre shall be dyed with standard vat dye in accordance with the manufacturer's instruction manual to achieve the depth of standard colour 1/1 according to ISO 105 A 06 or any other equivalent standard and it shall be ironed.

7.3.2.2 The other specimen shall be dyed in the other container in parallel with the first specimen dyed in accordance with Clause 7.3.2.1, using the same vat dye volume and fibre weight, and under the same conditions and method. Then the dyed fibre shall be ironed.

7.3.3 Test method

The colorimeter in Clause 7.3.2.1 and 7.3.2.2 shall be used to determine colour difference by folding the fibre into various thicknesses until light cannot penetrate it.

7.4 Colour fastness to light (xenon arc lamp)

A piece of cotton fibre shall be dyed in accordance with Clause 7.3.2.2 and tested in compliance with ISO 105 B 02 or the test method as stated in the manufacturer's instruction manual.

7.5 Colour fastness to washing

A piece of cotton fibre shall be dyed in accordance with Clause 7.3.2.2 and tested in compliance with ISO 105 C 01 to 06 or the test method as stated in the manufacturer's instruction manual.

7.6 Colour fastness to bleaching agents

A piece of cotton fibre shall be dyed in accordance with Clause 7.3.2.2 and tested in compliance with ISO 105 N 01 or the test method as stated in the manufacturer's instruction manual.

7.7 Colour fastness to rubbing

A piece of cotton fibre shall be dyed in accordance with Clause 7.3.2.2 and tested in compliance with ISO 105 X 12 or the test method as stated in the manufacturer's instruction manual.

Appendix A
Sampling and Criteria for Conformity
(Clause 6.1)

- A.1 "Lot" means vat dye with the same name and produced in the same period of time.
- A.2 Sampling and acceptance shall comply with the sampling plan below or another technically equivalent plan.
 - A.2.1 Sampling and acceptance for testing on packing as well as marking and labelling
 - A.2.1.1 Samples shall be taken at random from the same lot for 3 container units.
 - A.2.1.2 Samples complying with Clauses 4 and 5 shall be deemed to meet the criteria.
 - A.2.2 Sampling and acceptance for testing of the requirements
 - A.2.2.1 Samples shall be taken in accordance with Clause A.2.1.1 from each container at 3 different levels with equivalent volume and added together to obtain a total weight of not less than 500 grams. If the weight of the samples is insufficient, additional samples shall be taken at random to ensure that the specified weight is reached.
 - A.2.2.2 Samples which comply with Clause 3 shall be deemed to meet the criteria.
- A.3 Criteria for conformity
Vat dye samples which comply with Clause A.2.1.2 and Clause A.2.2.2 shall be deemed to meet the criteria.
