SRI LANKA STANDARD 649:1984
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SPECIFICATION FOR

FOOD ADDITIVES, COLOURING MATTER, TARTRAZINE, FOOD GRADE

SRI LANKA STANDARDS INSTITUTION

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SLS 649:1984

Gr. 7

SRI LANKA STANDARDS INSTITUTION
53, Dharmapala Mawatha,
Colombo 3,
Sri Lanka.

SRI LANKA STANDARD

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FOREWORD

This Sri Lanka Standard was authorized for adoption and publication by the Council of the Sri Lanka Standards Institution on 1984-05-28, after the draft, finalized by the Drafting Committee on Food Additives, had been approved by the Agricultural and Food Products Divisional Committee.

This is one of a series of Sri Lanka Standards for food colours.

This specification is subject to the Food Act No. 26 of 1980 and the regulations framed thereunder, wherever applicable.

All standard values given in this specification are in SI units.

For the purpose of deciding whether a particular requirement of this specification is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with CS 102. The number of figures to be retained in the rounded off value shall be the same as that of the specified value in this specification.

In the preparation of this specification, the assistance derived from the publications of the Indian Standards Institution is gratefully acknowledged.

1 SCOPE

This specification prescribes the requirements and methods of sampling and test for Tartrazine used in the colouring of food stuffs.

2 REFERENCES

CS 102 Presentation of numerical values

SLS 394 Analysis of water soluble coal-tar dyes permitted for use in foods.

SLS 467 Labelling of prepackaged foods
Part 1 General guidelines
SLS 543 Sampling of food colours

3 DESCRIPTION

3.1 Common name

Tartrazine

3.2 Synonyms

C.I. Food Yellow 4

F.D. & C Yellow No. 5.

3.3 Colour index number and EEC number

Colour index number - 19140

EEC number - E 102

3.4 Class

Monoazo

3.5 Chemical name

Trisodium salt of 5-hydroxy-1-
(p-sulphophenyl)-4-(p-sulphophenylazo)
-pyrazole-3-carboxylic acid.

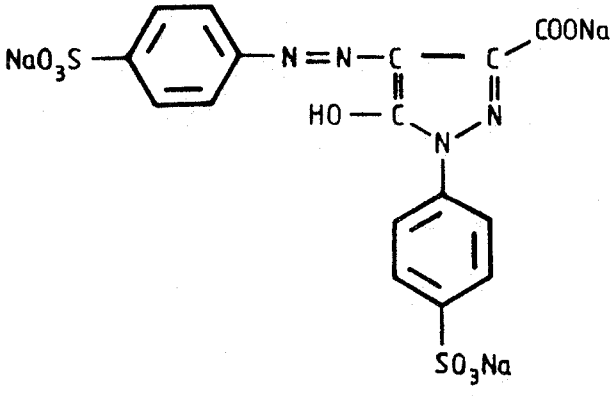
3.6 Empirical formula

$C_{16}H_9N_4O_9S_2Na_3$

3.7 Relative molecular mass

534.37

3.8 Structural formula



4 REQUIREMENTS

4.1 Composition

The colouring matter shall consist essentially of trisodium salt of 5-hydroxy-1-(p-sulphophenyl)-4-(p-sulphophenylazo)-pyrazole-3-carboxylic acid and shall not contain any extraneous matter injurious to health.

4.2 Special requirements

The colouring matter shall conform to the limits specified in Tables 1 and 2.

TABLE 1 - Requirements for tartrazine

Sl. No.	Characteristic	Requirement	Method of test Ref. to	
			Appendix in the standard	Clause Nos. in SLS 394: 1976
(1)	(2)	(3)	(4)	(5)
i	Total dye content corrected for sample dried at 105 ± 1 °C for 2 h, per cent by mass, min.	85	A	-
ii	Matter volatile at 135 °C, per cent by mass, max.	10	-	2.1
iii	Matter insoluble in water, per cent by mass, max.	0.3	-	2.2
iv	Chlorides and sulphates as sodium salts, total, per cent by mass, max.	5.0	-	2.5 and 2.6
v	Dye intermediates, per cent by mass, max.	0.5	B	-
vi	Subsidiary dyes, per cent by mass, max.	3.0	C	-
vii	Heavy metals as sulphides	Colour of reference standard	-	2.9

TABLE 2 - Tolerance limits for heavy metals

Sl. No. (1)	Trace element (2)	Tolerance limit mg/kg (3)	Method of test ref. to Cl. No. of SLS 394:1976 (4)
i	Copper (as Cu)	10	2.8
ii	Arsenic (as As)	1.0	
iii	Lead (as Pb)	10	

5 PACKAGING

Tartrazine shall be packed in suitable containers which shall in no way affect the nature and composition of the material within. The containers shall be strong enough to withstand damage in handling.

6 MARKING AND LABELLING

6.1 The following information shall be marked legibly and indelibly on the label of the container.

a) The words "Tartrazine, Food Grade";

b) Colour index number - No. 19140;

c) Name and address of the manufacturer;

d) Registered trade mark, if any;

e) Net mass, in grams; and

f) Batch number or code number.

6.2 Marking and labelling shall be carried out in accordance with **SLS 467.**

6.3 The containers may also be marked with the Certification Mark of the Sri Lanka Standards Institution illustrated below on permission being granted for such marking by the Sri Lanka Standards Institution.

NOTE - The use of the Sri Lanka Standards Institution Certification Mark (SLS Mark) is governed by the provisions of the Sri Lanka Standards Institution Act and the regulations framed thereunder.

The SLS mark on products covered by a Sri Lanka Standard is an assurance that they have been produced to comply with the requirements of that standard under a well defined system of inspection, testing and quality control, which is devised and supervised by the Institution and operated by the producer. SLS marked products are also continuously checked by the Institution for conformity to that standard as a further safeguard. Details of conditions under which a permit for the use of the Certification Mark may be granted to manufacturers or processors may be obtained from the Sri Lanka Standards Institution.

7 SAMPLING

7.1 Representative samples of the material shall be drawn as specified in the relevant clauses of **SLS 543**.

7.2 Tests for requirements specified in this specification shall be carried out on the composite sample obtained as in **7.1**.

8 METHODS OF TEST

Tests shall be carried out in accordance with **SLS 394**, and Appendices **A**, **B** and **C**.

9 CONFORMITY TO STANDARD

A lot shall be declared as conforming to the requirements of this specification if the test results obtained on the composite sample satisfy the relevant requirements.

APPENDIX A

DETERMINATION OF TOTAL DYE CONTENT

Two methods, a spectrophotometric method and the titanium trichloride method, are specified for the determination of the total dye content. The spectrophotometric method shall be used as the reference method.

A.1 SPECTROPHOTOMETRIC METHOD

A.1.1 Apparatus

A suitable spectrophotometer, with properly calibrated scales for both wave length and optical density. However, a suitable spectrocolorimeter may also be used for calibration against a spectrophotometer.

A.1.2 Procedure

Weigh, to the nearest milligram, approximately 250 mg of the dye sample and dissolve with 0.1 N hydrochloric acid in a 250-ml volumetric flask; dilute with the same solvent to make a final concentration of approximately 1 mg per 100 ml. Obtain the optical density of the dilute solution against 0.1 N hydrochloric acid as blank at 428 nm in a glass cell with 10.0-mm light path.

A.1.2.1 Simultaneously weigh to the nearest milligram approximately 2 g of the dye sample and dry this in an air oven at 105 ± 1 °C for two hours. Determine the loss of mass on drying and from this is calculated the dry mass of the sample (m) in the final solution taken for measurement of optical density.

A.1.3 Calculation

$$\text{Total dye content of sample, per cent by mass} = \frac{D \times 100}{m \times 475}$$

where,

D is optical density; and

m is dry mass, in grams, of the sample in 100 ml of solution.

NOTE - Tartrazine in 0.1 N hydrochloric acid has a maximum absorption $(E^{1\%}_{1cm})$ *of 475 at 428 nm.*

A.2 TITANIUM TRICHLORIDE METHOD

A.2.1 Reagents

A.2.1.1 *Sodium acid tartrate,* solid.

A.2.1.2 *Potassium dichromate,* standard solution, 0.1 N.

A.2.1.3 *Titanium trichloride,* standard solution, 0.1 N, prepared and standardized as in A.2.1.3.1 and A.2.1.3.2.

A.2.1.3.1 Prepare a 15 per cent (m/V) solution of titanium trichloride. Take 200 ml of this solution, add 150 ml of concentrated hydrochloric acid (rel. den. 1.16) and dilute to 2 litres. Make the solution approximately 0.1 N, place in a container provided with an arrangement to maintain it in an atmosphere of hydrogen and allow to stand for two days for absorption of residual oxygen.

A.2.1.3.2 Weigh 3 g of ferrous ammonium sulphate $FeSO_4.(NH_4)_2SO_4.6H_2O$ and transfer to a 500-ml flask. Introduce a stream of carbon dioxide and add 50 ml of freshly boiled water and 25 ml of sulphuric acid (40 per cent m/V). Then, without interrupting the current of carbon dioxide, rapidly add 40 ml of the standard potassium dichromate solution. Add the titanium trichloride solution until the calculated end point is nearly reached. Then add quickly 5 g of ammonium thiocyanate (NH_4CNS) and complete the titration. Carry out a blank titration on 3 g of ferrous ammonium sulphate using same quantities of water, sulphuric acid, ammonium thiocyanate and the current of carbon dioxide, without the addition of the standard potassium dichromate solution.

A.2.2 Procedure

Prepare a 1.0 per cent (m/V) aqueous solution of the material. Take a quantity of solution corresponding to about 20 ml of the standard titanium trichloride solution in a 500-ml Erlenmeyer flask. Add 15 g of sodium acid tartrate and dilute with water to a volume of 150 ml to 200 ml. Heat to boil and titrate with the standard titanium trichloride solution.

A.2.3 Calculation

Calculate the percentage of the pure dye from the following equation:

1 ml of 0.1 N $TiCl_3$ = 0.01336 g of tartrazine.

APPENDIX B

DETERMINATION OF DYE INTERMEDIATES

B.1 GENERAL

B.1.1 Tartrazine is prepared by diazotization of aniline-4-sulphonic acid and the diazo so formed is coupled with 1-p-sulphophenyl-5-pyrazolone-3-carboxylic acid. The intermediates are determined by ascending paper chromatography.

B.2 APPARATUS

B.2.1 *Chromatography tank and ancillary equipment*, as given in **2.4.2** of **SLS 394:1976.**

B.2.2 *Microsyringe*, capable of delivering 0.2 ml with a tolerance of ± 0.002 ml.

B.2.3 *Ultraviolet lamp*, with a wavelength of 365.5 nm.

B.2.4 *Filter paper*, Whatman No. 1 or equivalent.

B.3 REAGENTS

B.3.1 *Ammonium hydroxide solution*, relative density, 0.923.

B.3.2 *Aniline-4-sulphonic acid*

B.3.3 *1-p-sulphophenyl-1-5-pyrazolone-3-carboxylic acid*

B.3.4 *Developing solvent*, sodium bicarbonate solution, 10 per cent.

B.3.5 *Sodium nitrite*, crystals.

B.3.6 *Hydrochloric acid*, 1 N.

B.3.7 *Chromotropic acid*, 0.05 per cent.

B.3.8 *Sodium acetate solution*, 40 per cent.

B.3.9 *Sulphanilic acid solution*, 0.01 N, prepared as follows:

Take about 50 ml of ice cold water. Add 10 ml of 0.1 N sodium sulphanilate solution, 10.2 ml of 0.1 N sodium nitrite solution and 10 ml of 1 N hydrochloric acid. Shake well and adjust the volume to 100 ml in a measuring cylinder with ice cold water.

B.4 PROCEDURE

B.4.1 Preparation of solution

B.4.1.1 Prepare 2 per cent m/V solution of the dye in a mixture of nine parts of water and one part of the ammonium hydroxide.

B.4.1.2 *Reference substances*

a) Prepare a 0.01 per cent m/V solution of aniline-4-sulphonic acid using a solution containing nine parts of water and one part of the ammonium hydroxide.

b) Prepare a 0.01 per cent m/V solution of 1-p-sulphophenyl-5-pyrazolone-3-carboxylic acid using a solution containing a mixture of nine parts of water and one part of ammonium hydroxide.

B.4.2 Test solution

B.4.2.1 *Sample dye solution*

10 microlitres of the solution (B.4.1.1) shall be equivalent to 200 micrograms of the sample.

B.4.2.2 *Reference solution*

a) Aniline-4-sulphonic acid **(B.4.1.2 (a))**

5 microlitres shall be equivalent to 0.5 microgram corresponding to 0.25 per cent in the dye sample.

10 microlitres shall be equivalent to one microgram corresponding to 0.5 per cent in the dye sample; and

b) 1-p-sulphophenyl-5-pyrazolone-3-carboxylic acid **(B.4.1.2 (b))**

5 microlitres shall be equivalent to 0.5 microgram corresponding to 0.25 per cent in the dye sample.

10 microlitres shall be equivalent to one microgram corresponding to 0.5 per cent in the dye sample.

B.4.3 Mark out a sheet of chromatographic paper as shown in Fig. 3 of **SLS 394** and apply 10 microlitres of the dye solution as uniformly as possible with the help of a microsyringe. Also apply reference solution **(B.4.1.2)**. Mount the sheet together with a plain sheet to act as a blank in the frame *(C)*. Pour sufficient developing solvent into the tray *(D)* to bring the surface of the solvent about 10 mm below the base line of the chromatogram sheet. Place the frame *(C)* in the same position and replace the cover. Remove the filter paper after a 30-cm run. Dry in an oven at 70 °C to 75 °C.

B.4.4 Detection and semi-quantitative estimation

B.4.4.1 *Aniline-4-sulphonic acid*

Diazotize the chromatogram for the minutes under the nitrous acid vapours. Introduce the paper into a rectangular glass jar in which a beaker containing crystals of sodium nitrate have been kept. Cover and pour hydrochloric acid into the beaker, with the help of a pipette through the hole of the cover. Remove the paper and keep in air for ten minutes. Spray the chromatogram with the chromotropic acid solution in sodium acetate solution. Compare visually the intensity of the colour of the sample with the reference substances and report the content of aniline-4-sulphonic acid nearest to the intensity of the reference substance.

B.4.4.2 *1-p-sulphophenyl-5-pyrazolone-3-carboxylic acid*

Spray the chromatogram with the diazo solution of the sulphanilic acid after mixing it with the sodium acetate solution in 1:1 proportion. Compare visually the intensity of the colour of the sample with the reference substance and report the value nearest to the intensity of the reference substance.

APPENDIX C

DETERMINATION OF SUBSIDIARY DYES

The method given in **2.4** of **SLS 394:1976** shall be used except that the following solutions shall be employed in the test.

a) Atmosphere saturating and developing solvent

Methyl ethyl ketone	200 ml;
Acetone	300 ml;
Water	300 ml; and
Ammonia solution (rel.den.1.18)	2 ml

b) Extracting solvent

A mixture of equal volumes of acetone and water.

SLS CERTIFICATION MARK

The Sri Lanka Standards Institution is the owner of the registered certification mark shown below. Beneath the mark, the number of the Sri Lanka Standard relevant to the product is indicated. This mark may be used only by those who have obtained permits under the SLS certification marks scheme. The presence of this mark on or in relation to a product conveys the assurance that they have been produced to comply with the requirements of the relevant Sri Lanka Standard under a well designed system of quality control inspection and testing operated by the manufacturer and supervised by the SLSI *which includes surveillance inspection of the factory, testing of both factory and market samples.*

Further particulars of the terms and conditions of the permit may be obtained from the Sri *Lanka Standards Institution, 17, Victoria Place, Elvitigala Mawatha, Colombo 08.*

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SRI LANKA STANDARDS INSTITUTION

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The principal objects of the Institution as set out in the Act are to prepare standards and promote their adoption, to provide facilities for examination and testing of products, to operate a Certification Marks Scheme, to certify the quality of products meant for local consumption or exports and to promote standardization and quality control by educational, consultancy and research activity.

The Institution is financed by Government grants, and by the income from the sale of its publications and other services offered for Industry and Business Sector. Financial and administrative control is vested in a Council appointed in accordance with the provisions of the Act.

The development and formulation of National Standards is carried out by Technical Experts and representatives of other interest groups, assisted by the permanent officers of the Institution. These Technical Committees are appointed under the purview of the Sectoral Committees which in turn are appointed by the Council. The Sectoral Committees give the final Technical approval for the Draft National Standards prior to the approval by the Council of the SLSI.

All members of the Technical and Sectoral Committees render their services in an honorary capacity. In this process the Institution endeavours to ensure adequate representation of all view points.

In the International field the Institution represents Sri Lanka in the International Organization for Standardization (ISO), and participates in such fields of standardization as are of special interest to Sri Lanka.

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