# BROWN FK

*Prepared at the 30th JECFA (1986), published in FNP 37 (1986) and in FNP 52 (1992). Metals and arsenic specifications revised at the 59th JECFA (2002). No ADI was allocated at the 30th (JECFA 1986)*

**SYNONYMS** CI Food Brown 1; INS No. 154

**DEFINITION** A mixture of six mono-, bis- and trisazo colours (see 'Chemical names' below) and subsidiary colouring matters together with water, sodium chloride and/or sodium sulfate as the principal uncoloured components.

This product as manufactured, and to which these specifications apply, is often diluted with sodium chloride to a total colouring matters content of about 50% to meet the needs of users.

Chemical names

A mixture of
I Sodium 4-(2,4-diaminophenylazo) benzenesulfonate
II Sodium 4-(4,6-diamino-m-tolylazo) benzenesulfonate
III Disodium 4,4'-(4,6-diamino-1,3-phenylenebisazo)- di(benzenesulfonate)
IV Disodium 4,4'-(2,4-diamino-1,3-phenylenebisazo)- di(benzenesulfonate)
V Disodium 4,4'-(2,4-diamino-5-methyl-1,3-phenylene- bisazo)di(benzene-sulfonate)
VI Trisodium 4,4',4''-(2,4-diaminobenzene-1,3,5- trisazo)tri-(benzene-sulfonate)

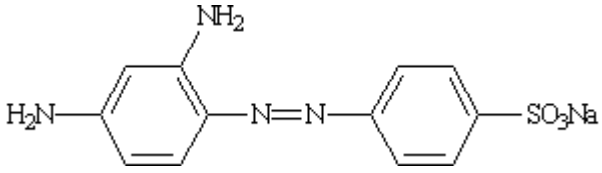
C.A.S. number 8062-14-4

Chemical formula

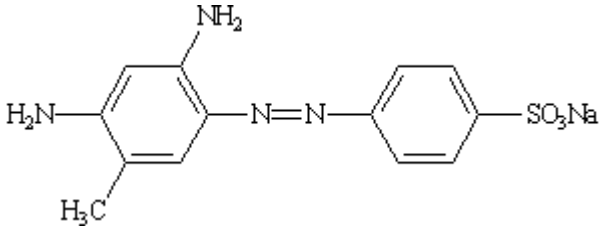
I $C_{12}H_{11}N_4NaO_3S$
II $C_{13}H_{13}N_4NaO_3S$
III $C_{18}H_{14}N_6Na_2O_6S_2$
IV $C_{18}H_{14}N_6Na_2O_6S_2$
V $C_{19}H_{16}N_6Na_2O_6S_2$
VI $C_{24}H_{17}N_8Na_3O_9S_3$

Structural formula

I




II

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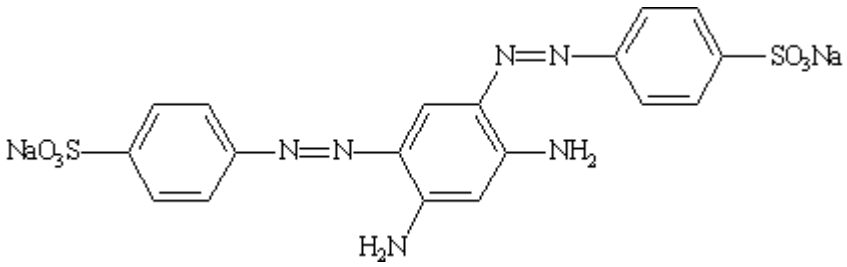

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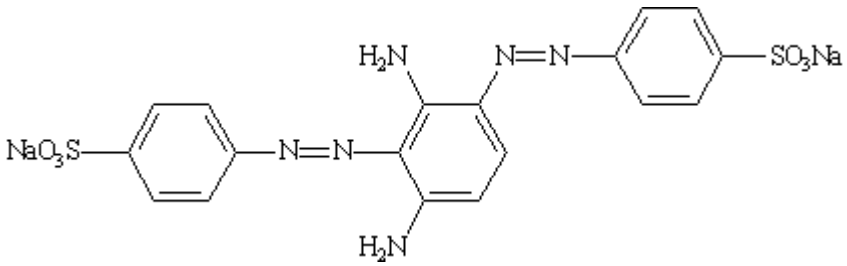

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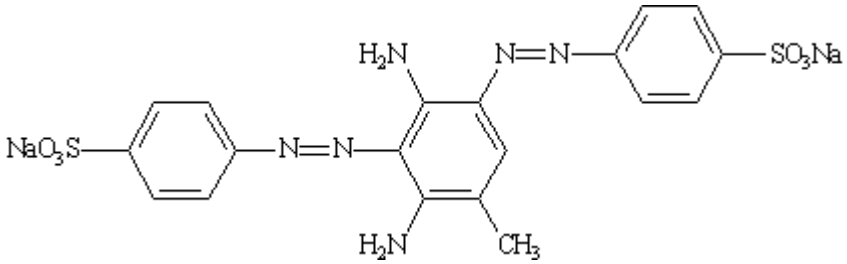
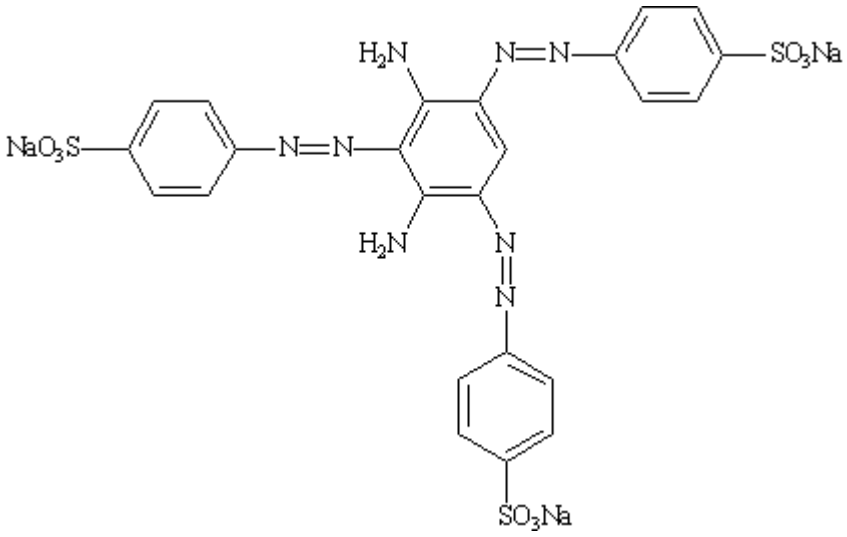


VI



Formula weight

I 314.30
II 328.33
III 520.46
IV 520.46
V 534.47
VI 726.59

Assay

Not less than 70% total colouring matters. Of the total colouring matters present the proportions of the components shall not exceed:

I 26%
II 17%
III 17%
IV 16%
V 20%
VI 16%

**DESCRIPTION** Red-brown powder or granules

**FUNCTIONAL USES** Colour

**CHARACTERISTICS**

IDENTIFICATION

| | |
|---|---|
| Solubility (Vol. 4) | Soluble in water; sparingly soluble in ethanol |
| Identification of colouring matters (Vol. 4) | Passes test |

PURITY

| | |
|---|---|
| Loss on drying at 135° (Vol. 4) | Not more than 30% together with chloride and sulfate calculated as sodium salts |
| Water insoluble matter (Vol. 4) | Not more than 0.2% |
| Lead (Vol. 4) | Not more than 2 mg/kg<br>Determine using an atomic absorption technique appropriate to the specified level. The selection of sample size and method of sample preparation may be based on the principles of the method described in Volume 4, "Instrumental Methods." |
| Subsidiary colouring matters | Not more than 3.5%<br>See description under METHOD OF ASSAY |
| Organic compounds other than colouring matters | Not more than<br>- 0.7% of 4-aminobenzene-1-sulfonic acid<br>- 0.35% of - m-phenylenediamine and 4-methyl-m-phenylenediamine<br>See description under TESTS |
| Unsulfonated primary aromatic amines (Vol. 4) | Not more than 0.007% calculated as aniline (other than m-phenylene diamine and 4-methyl-m-phenylene diamine) |
| Ether extractable matter (Vol. 4) | Not more than 0.2% |

TESTS

| | |
|---|---|
| Organic compounds other | Use *liquid chromatography* (see Volume 4) under the following conditions: |

than colouring matters

Detector: A UV HPLC detector recording absorbances at 254 nm
Column: 250 x 4 mm Li Chromosorb RP8, 7 µm
Solvent system
A: 0.075 M sodium acetate solution adjusted to pH 6.0 using glacial acetic acid.
B: A:methanol (2:3)
Flow rate: 1 ml/min
Gradient:
0 min: 100% (A), 0% (B)
15 min: 0% (A), 100% (B)
20 min: 0% (A), 100% (B)
25 min: 100% (A), 0% (B)

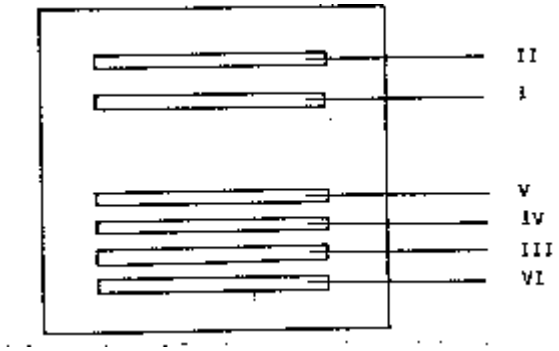
**METHOD OF ASSAY**

Determination of the amount of component colouring matters and subsidiary colouring matters.

Apparatus
- Glass tank capable of holding 20 cm x 20 cm glass TLC plates.
- Micrometer syringe capable of delivering 0.10 ml with a tolerance of ± 0.002 ml
- Spectrophotometer

Reagents:
All of recognized analytical grade
- Chromatography solvent: A mixture of 4 parts phenol and 1 part water by volume
- Diethyl ether
- Kieselgel G
- Extraction solvent: Mix 100 ml 10% sodium carbonate solution with 500 ml methanol and dilute to 1 L with water

Procedure
Prepare a 20 x 20 cm TLC plate with a 0.5 mm thick coating of Kieselgel G. Using the micrometer syringe apply a solution containing 0.4 mg Brown FK as evenly as possible in an area near the bottom of the plate. Develop the chromatogram in the phenol/water mixture allowing the solvent to ascend the full height of the plate; then remove it from the tank and allow it to dry.
The diagram shows a typical Brown FK chromatogram with the bands numerically identified.




Remove each band from the plate and transfer it to a small beaker. The subsidiary colouring matters are located in the area between bands I and V. Remove this area of Kieselgel G. Transfer it to a beaker and wash it with a

small quantity of ether to remove the phenol. Allow the residual ether to evaporate.

Add 10 ml of extraction solvent to each beaker and swirl to extract the colour. Filter through a small filter paper and measure the absorbance versus extraction solvent in 10 mm cells at the wavelength of maximum absorption. Calculate the concentration of each component colouring matter and of the subsidiary colouring matters using the absorptivity figures given in the table below.

| Component | Wavelength(nm) | Absorptivity |
|---|---|---|
| I | 453 | 55.1 |
| II | 464 | 59.6 |
| III | 355 | 88.2 |
| IV | 412 | 70.5 |
| V | 448 | 60.3 |
| VI | 410 | 59.7 |
| Subsidiary Colouring Matters | 425 | 74.2 |

Calculation of

(i) Total colouring matters content
(ii) Percentages of component colours
(iii) Percentage of subsidiary colours

Note: Component colours are expressed as percentages of the total colouring matters present, i.e. as percentages of the sum of component colours and subsidiary colours. Subsidiary colours are expressed for the purposes of the specification limit as a percentage of the sample.
Let the absorptivities of the component colours be $a_1$, $a_2$....$a_6$. and the absorbances of the extracts of the component colours be $A_1$, $A_2$..... $A_6$.
Let the Absorptivity of the subsidiary colours be $a_7$ and the absorbance of the extract of the subsidiary colours be $A_7$.

The weight (in mg) of component colour I in the 10 ml extract is calculated from the expression:

$$\frac{A_1}{a_1} \times 10\,mg = W_1$$

In a similar manner calculate the weights ($W_2$, $W_3$.....$W_7$) of the remaining component colours and the subsidiary colours.

(i) Total colouring matters content (%) =

$$\frac{W_1 + W_2 + W_3 + W_4 + W_5 + W_6 + W_7}{0.4} \times 100\,\%$$

(ii) The percentage of component colour 1 =

$$\frac{W_1}{W_1 + W_2 + W_3 + W_4 + W_5 + W_6 + W_7} \times 100\ \%$$

In a similar manner calculate the percentages of the other component colours.

(iii) The percentage of subsidiary colours =

$$\frac{W_7}{0.4} \times 100\ \%$$