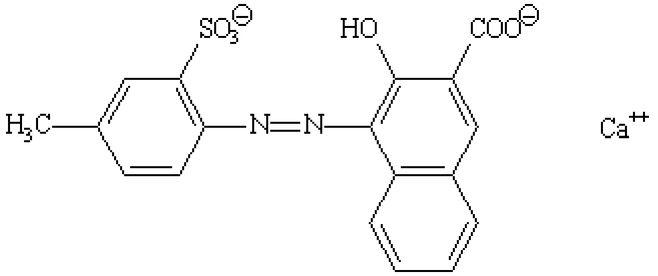
LITHOL RUBINE BK

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 (1987)

SYNONYMS	CI Pigment Red 57, FD & C Red No. 7, Brilliant Carmine 6B, Rubinpigment, Carmine 6B, Litholrubintoner BKL, Permanent Rubin L6B, CI (1975) No. 15850, INS No. 180
DEFINITION	Consists essentially of calcium 3-hydroxy-4-(4-methyl-2-sulfonatophenylazo)-2-naphthalenecarboxylate and subsidiary colouring matters together with water, calcium chloride and/or calcium sulfate as the principal uncoloured components.
Chemical names	Calcium 3-hydroxy-4-(4-methyl-2-sulfonatophenylazo)-2-naphthalene-carboxylate
C.A.S. number	5284-04-9
Chemical formula	$C_{18}H_{12}CaN_2O_6S$
Structural formula	
Formula weight	424.45
Assay	Not less than 90% total colouring matter
DESCRIPTION	Red powder
FUNCTIONAL USES	Colour
CHARACTERISTICS	
IDENTIFICATION	
Solubility (Vol. 4)	Slightly soluble in hot water (90^o); insoluble in cold water; insoluble in ethanol
Spectrophotometry (Vol. 4)	Absorbance maximum at about 442 nm (in dimethylformamide)
PURITY	
Loss on drying at 135^o (Vol. 4)	Not more than 10% together with chloride and sulfate calculated as calcium salts

Lead (Vol. 4)	Not more than 2 mg/kg 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 0.5% See description under TESTS
Organic compounds other than colouring matters	Not more than 0.2% of 2-Amino-5-methylbenzenesulfonic acid, calcium salt Not more than 0.4% of 3-hydroxy-2-naphthalenecarboxylic acid, calcium salt See description under TESTS
Unsulfonated primary aromatic amines	Not more than 0.01% calculated as aniline See description under TESTS
Ether extractable matter (Vol. 4)	Not more than 0.2% Dry 2 g of the sample in a desiccator (sulfuric acid) for 24 h and extract using Method II

TESTS

PURITY TESTS

Chlorides (Vol. 4)	Mix 1 g of the sample with 100 ml of water and let stand for 30 min, shaking occasionally. Filter and wash the residue with a small amount of water. Combine the washings with the filtrate. Acidify with 5 ml of 1.5 N nitric acid and titrate with 0.1 N silver nitrate solution as directed under *Determination of Chloride as Sodium Chloride*. Each ml of 0.1 N silver nitrate solution is equivalent to 0.00555 g of calcium chloride.
Sulfates	Weigh accurately about 1 g of the sample, mix 100 ml of water and heat on a water bath for 10 min. Cool, filter and wash the residue with a small amount of water. Combine the washings with the filtrate. Dilute to 150 ml with water and acidify with hydrochloric acid, adding 1 ml in excess. Heat the solution to boiling and add an excess of 0.25 N barium chloride drop by drop, with stirring. Allow the mixture to stand on a hot plate for 4 h, or leave it overnight at room temperature and then bring it to about 80^{o} and allow the precipitate to settle. Filter off the precipitated barium sulfate, wash with hot water and ignite at a dull red heat in a tared crucible until a constant weight is obtained. Carry out a blank determination and apply any necessary correction. Calculate the % of sulfate as calcium sulfate from: $\frac{\text{weight of } BaSO_4 \text{ (g) x } 0.583}{\text{weight of sample (g)}} \text{ x } 100$
Subsidiary colouring matters	Boil 0.10 g of sample gently with 100 ml of glacial acetic acid and with 75 ml of 8 N hydrochloric acid until the colour has dissolved. Cool and transfer the solution to a 1000 ml separatory funnel, washing any residual

solution into the funnel with small portions of acetic acid. Extract the acidic solution with 150 ml of ether and separate the miscible solution formed by adding about 150 ml of water. Transfer the lower layer to a second funnel and extract with another 100 ml of ether.
Combine the ether extracts and wash with 100 ml portions of water until the washings are colourless, and twice more after the last colourless washings. Remove the subsidiary colour from the ether layer by extraction with 20 ml portions of 2% sodium hydroxide. Warm to expel the ether. Determine the colour concentration spectrophotometrically at 505 nm.

Standard:
3-Hydroxy-4-(4-methylphenylazo)-2-naphthalenecarboxylic acid has an absorptivity of 0.032 L mg^{-1} cm^{-1} at 505 nm.

Organic compounds other than colouring matters (Vol. 4)

Use 10% ammonium sulfate in place of the 25% ammonium sulfate listed in the method. Add 100 ml eluant to 5 g cellulose, stir, allow to settle and decant. Place 0.100 g of the colour sample in a beaker and add 5 ml ethanol. Stir to ensure complete wetting of the sample. Transfer the 5 g cellulose to the beaker containing the sample. Add 10 g ammonium sulfate and stir thoroughly. Transfer the mixture to the column. Rinse the beaker with 25 ml eluant, adding the rinse to the column. Then follow the procedure detailed in the *Determination by Column Chromatography.*

Unsulfonated primary aromatic amines (Vol. 4)

Mix 2 of the sample well with 150 ml of toluene and boil gently for 5 min. Filter after cooling and wash the residue with a small amount of toluene. Combine the washing with the filtrate and extract with three 10-ml portions of N hydrochloric acid and dilute the combined extracts to 100 ml with water. Follow the procedure detailed in the method *Unsulfonated primary aromatic amines*.

METHOD OF ASSAY

Place about 0.2 g of the sample, accurately weighed, in a 500-ml Erlenmeyer flask and add 5 ml of sulfuric acid. Mix well and add 100 ml of ethanol. Shake well, heat on a water bath. Add a solution made by dissolving 20 g of sodium hydrogen tartrate in 100 ml of boiling water and mix with 20 ml of 30% sodium hydroxide solution, shaking vigorously. Titrate with 0.1 N titanous chloride. Each ml of 0.1 N titanous chloride is equivalent to 10.61 mg of $C_{18}H_{12}CaN_2O_6S$.