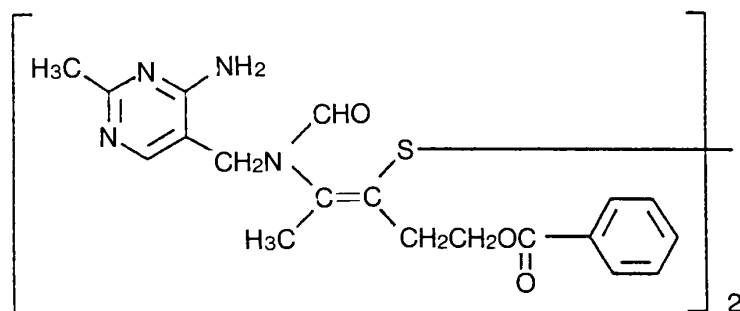


## D. MONOGRAPHS

### Bisbentiamine

Benzoylthiamine Disulfide



$C_{38}H_{42}N_8O_6S_2$

Mol. Wt. 770.93

*N,N*- [ dithiobis [ 2- [ 2-(benzoyloxy) ethyl ] -1-methyl-2,1-ethenediyl ] ] bis [ *N*- [ (4-amino-2-methyl-5-pyrimidinyl)methyl ] formamide ] [ 2667-89-2 ]

**Content** Bisbentiamine, when dried, contains 98.0 - 102.0% of bisbentiamine ( $C_{38}H_{42}N_8O_6S_2$ ).

**Description** Bisbentiamine occurs as white crystals or crystalline powder. It is odorless and has a slightly bitter taste.

**Identification** (1) To 50 mg of Bisbentiamine, add 5 ml of methanol, and dissolve while warming. Add 2 ml of a mixture (1 : 1) of hydroxylamine hydrochloride solution (3 20) and sodium hydroxide solution (3 20), and warm in a water bath at 50 - 60 for 2 minutes. To this solution, add 0.8 ml of hydrochloric acid and 0.5 ml of ferric chloride solution (1 10), and add 8 ml of water. A red-purple color develops.

(2) To 5 mg of Bisbentiamine, add 1 ml of methanol to solve while warming, add 2 ml of water, 2 ml of cysteine hydrochloride solution (1 100), and 1 ml of sodium hydroxide solution (1 25), shake, and allow to stand for 5 minutes. To this solution, add 1 ml of freshly prepared potassium ferricyanide solution (1 10) and 5 ml of isobutyl alcohol, shake vigorously for 2 minutes, allow to stand, and observe under ultraviolet light. The isobutyl alcohol layer emits a blue-purple fluorescence, which disappears when the solution is made acidic, and reappears when returned to alkaline.

**Purity** (1) Melting point 140 - 145 (decomposition).

(2) Clarity and color of solution Colorless and clear (0.10 g, methanol 20 ml).

(3) Heavy metals Not more than 20  $\mu\text{g/g}$  as Pb (1.0 g, Method 2, Control solution 2.0 ml).

## D. MONOGRAPHS

**Loss on Drying** Not more than 0.50% (24 hours).

**Residue on Ignition** Not more than 0.20%.

**Assay** Weigh accurately about 0.5 g of Bisbentiamine, previously dried, dissolve in 50 ml of acetic acid, and titrate with 0.1 mol/l perchloric acid (indicator: 1 ml of crystal violet - acetic acid TS) until the color of the solution changes from purple through blue to green. Perform a blank test in the same manner, and make any necessary correction.

1 ml of 0.1 mol/l perchloric acid = 38.547 mg of  $C_{38}H_{42}N_8O_6S_2$