



**NATIONAL STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA**

GB 1886.225-2016

National Food Safety Standard -- Food Additives -- Ethoxyquin

食品安全国家标准 食品添加剂 乙氧基喹

(English Translation)

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1 Scope

This standard applies to phenetidine and acetone as raw materials, the resulting catalyst under the reaction conditions and heating food additives ethoxyethyl Quinoline base.

2.1 Chemical Name

6-ethoxy-1,2-dihydro-2,2,4-trimethyl quinoline

2.2 Structure

Formula 2.3

C₁₄H₁₉NO

2.4 Molecular Weight

217.31 (according to 2011 international relative atomic mass)

3.1 Sensory requirements

Sensory requirements shall comply with the requirements in Table 1.

Table 1 Sensory requirements

Project requires test methods Color Light yellow to amber, light in a long time, the air After standing gradually turned dark brown Viscous liquid state Proper amount of sample is placed in clean, dry cuvette, in natural light the observed color and state

3.2 Physical and Chemical Indicators

Physical and chemical indicators should be consistent with the provisions of Table 2.

Table 2. Physical and chemical indicators

Item Index Test Method Ethoxyquin, w /% ≥ 98.0 Appendix A A.3 Phenetidide, w /% ≤ 0.2
 Appendix A A.4 Lead (Pb)/(mg/kg) ≤ 2.0 GB 5009.12 Arsenic (As)/(mg/kg) ≤ 2.0 GB
 5009.76 Appendix A Testing method A.1 General Provisions this standard reagents and
 water, in the absence of other requirements specified, refer to three analytical reagent
 water and GB/T 6682 provisions. test Test used in the standard solution, the standard
 solution, the determination of impurities, products and preparations, in the absence of other
 requirements specified, are by GB/T 601, GB/T 602 and GB/T 603 Preparation
 predetermined. The test does not indicate when the solution which is formulated with a
 solvent, refer to the aqueous solution.

A.2 Identification Test the sample was dissolved in 10mL of acetonitrile 1mg and observed
 under UV light, can show strong fluorescence.

Determination of ethoxyquin A.3 A.3.1 Reagents and materials A.3.1.1 glacial acetic acid.

A.3.1.2 methyl violet indicator solution. 10g/L. Weigh 1g Methyl Violet, dissolved in an
 appropriate amount of glacial acetic acid, transferred to 100mL flask with ice Acid volume.

Perchloric acid standard titration solution A.3.1.3. $c(\text{HClO}_4) = 0.1\text{mol/L}$.

A.3.1.4 acetic anhydride.

A.3.2 Analysis step

Weigh about 0.2g sample, accurate to 0.0001g, dried in a clean Erlenmeyer flask was
 added 50mL acetic acid was dissolved to make ice, plus Into 1mL of acetic anhydride
 mixed, add 2 drops of methyl purple indicator solution, titration with perchloric acid standard
 solution was titrated to green is the end.

At the same time a blank test.

A.3.3 calculation results Ethoxyquin mass fraction w_1 , according to formula (A.1) is
 calculated.

$$(V-V_0) \times c \times M$$

$$1000 \times m \times$$

$$100\% \text{ (A.1)}$$

Where.

V --- Sample consumption perchloric acid standard titrant volume in milliliters (mL);

--- consumption volume V_0 blank test standard solution of perchloric acid in milliliters (mL);

C --- perchloric acid concentration standard titration solution in units of moles per liter (mol/L);

--- M is ethoxyquin molar mass in grams per mole (g/mol) [M (C₁₄H₁₉NO) = 217.31];

1000 --- volume conversion factor;

--- m sample mass, in grams (g).

The arithmetic average of the results of the two replicates is reported results. The absolute difference determination results parallel to not more than 0.4%.

Determination of phenetidine A.4 A.4.1 Method summary by gas chromatography under the selected operating conditions, the respective components of the sample injector was isolated, detected by a hydrogen flame detector, with external Standard method.

A.4.2 Reagents and materials A.4.2.1 ethanol.

A.4.2.2 nitrogen. mass fraction of $\geq 99.99\%$.

A.4.2.3 diethyl phthalate internal standard solution. 100 μ g/mL. Weigh 1g diethyl phthalate, accurate to 0.0001g, to Dissolving a small amount of ethanol, into a volumetric flask, dilute to ethanol 100mL.

A.4.2.4 of standard aminophenyl ether solution. 10mg/mL. Weigh about 0.05g phenetidine (purity $\geq 99\%$), to the nearest 0.0001g, in stoppered glass vial, was added 5mL diethyl phthalate internal standard solution (A.4.2.3) pipetting with a pipette, cover Cork, shake.

A.4.2.5 sample solution. 60mg/mL. Weigh about 0.3g sample, accurate to 0.0001g, in stoppered glass vial, was added with a pipette Pipette tube in 5mL diethyl phthalate internal standard solution (A.4.2.3), stoppered, shaken.

A.4.3 Instruments and Equipment A.4.3.1 gas chromatograph. equipped with hydrogen flame ionization detector.

A.4.3.2 micro-syringe.

A.4.4 column and operating conditions the standard recommended chromatographic column and operating conditions are shown in Table A.1. Other to achieve the same degree of separation columns and LC operating conditions Member may also be used.

Table A.1 Recommended chromatographic column and operating conditions

Project Parameters

Column 5% phenyl - methyl polysiloxane Inside diameter x length x thickness of column 30m x 0.32mm x 0.25 μ m Column temperature/ $^{\circ}$ C Initial temperature 60 $^{\circ}$ C, temperature rising rate of 10.0 $^{\circ}$ C/min was raised to 200 $^{\circ}$ C, holding 6min, at a rate of 20 $^{\circ}$ C/min raised

to 280 °C, kept 10min Gasification chamber temperature/°C 240 Detector temperature/°C 280 Nitrogen carrier gas Flow rate/(mL/min) 40 A.4.5 Analysis step After the instrument is stable, the number of injection needles standard solution, until the adjacent base stable peak area ratio of the two needles, analyzed in the following order.

- a) phenetidine standard solution (A.4.2.4);
- b) the sample solution (A.4.2.5);
- c) sample solution (A.4.2.5);
- d) phenetidine standard solution (A.4.2.4).

The injection volume are 1μL.

A.4.6 calculation results the order a) -> b) -> c) -> d) of the injection, each injection peak area ratio f calculated according to the formula (A.2).

Where.

--- peak area S1 aminophenyl ether;

--- peak area S2 within the standard.

According to formula (A.2) obtained fa and fd, fb, and fc are averaged, to obtain f1 and f2.

Phenetidine quality score w2, calculated according to formula (A.3).

$f1 \times m1 \times w$

$f2 \times m2 \times$

100% (A.3)

Where.

F1 --- phenetidine standard solution averaged ratio of peak area of phenetidine and the internal standard;

M1 --- phenetidine quality standards, the unit is gram (G);

W --- mass fraction of phenetidine standard,%;

F2 --- averaged sample solution aminophenyl ether ratio of peak area of the internal standard;

--- M2 sample mass, in grams (g).

The arithmetic average of the results of the two replicates is reported results. The absolute difference determination results is not larger than the two parallel assay 6% of the arithmetic mean values.