GB

ICS 65.120
B 46

National Standard

of the People's Republic of China

GB/T 13883-2008

Replacing GB/T 13883-1992

Determination of selenium in feeds

饲料中硒的测定

Issued on: August 1, 2008 **Implemented on: November 1, 2008**

Issued by: General Administration of Quality Supervision, Inspection and Quarantine of the People's Republic of China;

Standardization Administration Committee.

Table of Contents

Foreword

This Standard replaces GB/T 13883-1992 "Determination of selenium in feeds."

Compared with GB/T 13883-1992, the main changes in this Standard are as follows:

— INTRODUCE hydride generation atomic fluorescence spectrometry as arbitration method;

— SPECIFY that the excitation wavelength of 2,3-diamino-naphthalene fluorescence is 376mm; the emission wavelength is 520nm.

This Standard was proposed and shall be under the jurisdiction of the National Standardization Technical Committee of Feed Industry.

Drafting organizations of this Standard: China Feed Industry Association, the National Feed Quality Supervision and Inspection Center (Wuhan).

Main drafters of this Standard: He Yifan, Xin Shengpeng, Su Shenglan, Xu Jinping, Zou Daqiong, Liu Xiaomin, Gao Lihong.

This Standard was first-time released in 1991 as the national standard GB 13883-1992. It was adjusted to non-mandatory standard in 1997, and renumbered as GB/T 13883-1992. This revision is the first revision of this Standard.

Determination of selenium in feeds

1 Scope

This Standard specifies the determination method of selenium in compound feeds, concentrated feeds, and premixed feeds.

This Standard applies to the determination of selenium in compound feeds, concentrated feeds, and premixed feeds.

Quantitative limit of hydride generation atomic fluorescence spectrometry is 0.01mg/kg; quantitative limit of fluorescence is 0.02mg/kg.

2 Normative references

The provisions in following documents become the provisions of this Standard through reference in this Standard. For dated references, the subsequent amendments (excluding corrections) or revisions do not apply to this Standard, however, parties who reach an agreement based on this Standard are encouraged to study if the latest versions of these documents are applicable. For undated references, the latest edition of the referenced document applies..

GB/T 6682 Water for analytical laboratory use - Specification and test methods (GB/T 6682-1992, neq ISO 3696: 1987)

GB/T 14699.1 Animal feeding stuffs - Sampling (GB/T 14699.1-2005, ISO 6497: 2002, IDT)

GB/T 20195 Animal feeding stuffs - Preparation of test samples (GB/T 20195-2006, ISO 6498: 1998, IDT)

3 The first method -- Hydride generation atomic fluorescence spectrometry (Arbitration method)

3.1 Principle

After the sample is heated and digested by acid, in hydrochloric acid medium, REDUCE the hexavalent selenium in the sample TO tetravalent selenium. USE sodium borohydride as the reducing agent; REDUCE the tetravalent selenium to hydrogen selenide in hydrochloric acid medium; then it is carried by carrier gas into the atomizer to atomize. Under the irradiation of selenium hollow cathode lamp,

the relative deviation ≤20%;

When the mass fraction of selenium is more than 0.40mg/kg, the relative deviation ≤12%.

4 The second method -- 2,3-diamino-naphthalene fluorescence

4.1 Principle

DIGEST the sample by mixed acid, so as to make the selenium free. In acidic solution, selenium (Se^{4+}) and 2,3-diamino-naphthalene (DAN) generate 4,5-phenylbenzo selenadiazole. USE cyclohexane to directly extract in the solution of which the acidity is the same as that of generated complex. USE fluorescence spectrophotometer to determine the fluorescence intensity at an excitation wavelength of 376nm and an emission wavelength of 520nm. And then calculate selenium content in the sample.

4.2 Reagents

The following reagents, unless otherwise stated, are of analytical purity. The water shall meet the Grade-2 water specified in GB/T 6682.

4.2.1 Perchloric acid: premium-grade pure.

4.2.2 Nitrate: premium-grade pure.

4.2.3 Ammonia solution: 1 + 1.

4.2.4 Hydrochloric acid solution: *c* (HCl) = 3mol/L.

4.2.5 Hydrochloric acid solution: *c* (HCl) = 0.1mol/L.

4.2.6 Cyclohexane: if there are fluorescent impurities, it shall be re-evaporated before being used.

4.2.7 Hydroxylamine hydrochloride-ethylene diamine tetraacetic acid (EDTA) solution: WEIGH 10g of EDTA and dissolve them in 500mL of water. ADD 25g of hydroxylamine hydrochloride and make them dissolved. DILUTE with water to 1L.

4.2.8 2,3-diamino-naphthalene (DAN) solution: WEIGH 10g of DAN in a 250-mL beaker. ADD 100mL of hydrochloric acid solution (4.2.5) to make them dissolved. TRANSFER into a 250-mL separating funnel. ADD 20mL of cyclohexane (4.2.6) and SHAKE for 1min. REMOVE cyclohexane after stratification. USE cyclohexane to repeatedly process aqueous phase for 2 to 3 times. PUT the aqueous phase into a brown bottle and cover with 1-cm-thick cyclohexane. KEEP in the dark. This solution can be used for several weeks.

4.5.1.2 Premixed feeds

For premixed sample, WEIGH 1.0g of sample (accurate to 0.0001g). PLACE in a 100-mL high beaker. ADD 10mL of water and 15mL of nitric acid (4.2.2). COVER with a watch glass. PLACE on an electric hot plate and boiling at a low temperature for 30min. TAKE down and cool it down. USE water to transfer into a 100-mL volumetric flask. DILUTE to the scale. SHAKE well. MESURE some supernatant (Se≤0.4μg) into a 100-mL high beaker. ADD 5mL of perchloric acid (4.2.1). The following steps are conducted according to 4.5.1.1 - the analysis steps after perchloric acid is added.

4.5.2 Preparation of standard curves

Accurately MEASURE 0.00, 0.50, 1.00, 2.00, 3.00, 4.00mL of selenium standard working solution (4.2.9) respectively into 50-mL colorimetric tubes with stoppers. ADD 2 drops of cresol red indicator (4.2.10). The following steps are conducted according to 4.5.1.1 - the analysis steps after “neutralize with aqueous ammonia solution (4.2.3)”.

4.5.3 Sample determination

ABSORB the cyclohexane solution on the upper layer of the solution to be tested (4.5.1.1) INTO a 1-cm quartz cup. Use fluorophotometer to respectively determine the fluorescence intensity at the positions where excitation wavelength is 376nm and emission wavelength is 520nm. At the same time, DETERMINE the standard curves; DRAW the standard curves. OBTAIN the selenium content in the solution from the standard curves. Determination results of selenium in samples are calculated according to 4.6.1.

4.6 Calculation and presentation of analysis results

4.6.1 Calculation results

Selenium content X in the sample, in mass fraction, of which the numerical value is expressed in milligrams per kilogram (mg/kg), is calculated according to equation (2).

$$X = \frac{(m_1 - m_2) \times V_0 \times 1\,000}{m_0 \times V_1 \times 1\,000} = \frac{(m_1 - m_2) \times V_0}{m_0 \times V_1} \quad \cdots\cdots\cdots\cdots\cdots\cdots (2)$$

Where:

m_1 — Mass fraction of selenium in samples, obtained from the standard curve, in units of micrograms (μg);

m_2 — Mass fraction of selenium in blank, obtained from the standard curve, in units of micrograms (μg);

V_0 — Total volume of sample digestion solution, in units of milliliters (mL);

m_0 — Sample mass, in units of grams (g);

V_1 — Volume of dispensed solution, in units of milliliters (mL).

The determination results are expressed in the arithmetic mean after parallel determination. The calculation results accurate to 0.01mg/kg.

4.6.2 Repeatability

The results of two parallel determinations obtained in the same laboratory and by the same analyst shall meet the following requirements for relative deviation:

When the mass fraction of selenium is less than or equal to 0.10mg/kg, the relative deviation ≤40%;

When the mass fraction of selenium is more than 0.10mg/kg and less than 0.40 mg/kg, the relative deviation ≤20%;

When the mass fraction of selenium is more than 0.40mg/kg, the relative deviation ≤15%.

_____ END _____

This is an excerpt of the PDF (Some pages are marked off intentionally)

------ The End ------