DB43湖 南 省 地 方 标 准

DB43/T XXX—XXXX



饲料添加剂 苏氨酸锌螯合物

Feed additive Threonine zinc chelate

（征求意见稿）

XXXX - XX -XX发布 XXXX - XX -XX实施



湖南省市场监督管理局 发 布

DB43/T XXX—XXXX

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前 言

本文件按照GB/T 1.1-2020《标准化工作导则 第1部分：标准化文件的结构和起草规则》的规定起草。

本文件的某些内容可能涉及专利。本文件的发布机构不承担识别这些专利的责任。

本文件由湖南省市场监督管理局提出并归口。

本文件起草单位：长沙兴嘉生物工程股份有限公司。

本文件主要起草人：黄逸强、彭红星、洪双胜、钟芳、罗丹。

**饲料添加剂 苏氨酸锌螯合物**

**1 范围**

本标准规定了饲料添加剂苏氨酸锌螯合物的术语和定义、要求、试验方法、检验规则及标签、包装、运输、贮存和保质期。

本标准适用于饲料添加剂苏氨酸锌螯合物的生产、销售和使用。

**2 规范性引用文件**

下列文件对于本文件的应用是必不可少的。凡是注日期的引用文件，仅所注日期的版本适用于本文件。凡是不注日期的引用文件，其最新版本（包括所有的修改单）适用于本文件。

GB/T 5917 饲料粒度测定方法

GB/T 6435 饲料水分的测定方法

GB/T 6682 分析实验室用水规格和试验方法

GB 10648 饲料标签

GB 13078 饲料卫生标准

GB/T 13079 饲料中总砷的测定方法

GB/T 13080 饲料中铅的测定方法

GB 13082 饲料中镉的测定方法

GB/T 14699.1 饲料采样

GB/T 18823 饲料检测结果判定的允许误差

**3 化学名称、分子式、相对分子质量和结构式**

化学名称：苏氨酸锌螯合物

分子式：C8H20N2O8Zn

相对分子质量：337.63 (按2011年国际相对原子质量计)

结构式：(C4H8NO3)2Zn·2H2O

**4 术语和定义**

本文件没有需要界定的术语和定义。

**5 技术要求**

5.1 外观与性状

本品浅黄色或类白色结晶性粉末或细小颗粒，微溶于水，溶于酸，无刺激性气味。

5.2 技术指标

表 1

|  |  |  |
| --- | --- | --- |
| 项 目 | | 指 标 |
| 锌（以Zn计） % | ≥ | 18.5 |
| 总苏氨酸 % | ≥ | 67.0 |
| 砷（As） % | ≤ | 0.0005 |
| 铅（Pb） % | ≤ | 0.001 |
| 镉（Cd） % | ≤ | 0.0005 |
| 水分 % | ≤ | 10.00 |
| 粒度（孔径0.84mm试验筛通过率%） | ≥ | 95.0 |

**6 采样**

按GB/T 14699.1的规定进行采样。

**7 试验方法**

除非另有规定，本文件所用试剂和水，均指分析纯试剂和GB/T 6682中规定的三级水，所用标准溶液、制剂及制品，均按GB/T 601、GB/T 602、GB/T 603规定的方法制备。

7.1 感官检验

取约20g的试样置于清洁、干燥的白瓷盘内，在自然光下目视观察其色泽和状态。

7.2 鉴别实验

7.2.1 试剂或材料

7.2.1.1 盐酸溶液（1+10，v/v）：取100mL浓盐酸缓缓加入到1000mL水中，即得。

7.2.1.2 氨水溶液（1+1，v/v）：取水100mL加入到100mL浓氨水中，即得。

7.2.1.3 硫酸钠溶液：250g/L。

7.2.1.4 10g/L双硫腙四氯化碳溶液：取双硫腙1g加四氯化碳使其溶解至100mL，即得，使用期为两周。

7.2.1.5 三氯甲烷 AR。

7.2.1.6 茚三酮溶液：1g/L水溶液。

7.2.2 仪器设备

7.2.2.1 分析天平：感量0.0001g。

7.2.3 锌离子的鉴别

称取0.2g试样，加（1+10）10mL（7.2.1.1）盐酸溶液，加热至试样全部溶解，加5mL水，用氨水（7.2.1.2）调节试验溶液PH值为4～5，加两滴硫酸钠溶液（7.2.1.3），再加数滴双硫腙四氯化碳溶液（7.2.1.4），和1mL三氯甲烷（7.2.1.5），振摇后，有机层显紫红色。

7.2.4 苏氨酸的鉴别

7.2.4.1 茚三酮试验

称取试样0.1g，溶于100mL水中，取该溶液5mL，加1mL茚三酮溶液（7.2.1.6），加热，溶液呈蓝紫色，随温度升高颜色加重。

7.3 锌含量的测定

7.3.1 试剂或材料

7.3.1.1 盐酸溶液（1+3，v/v）：移取25 mL盐酸（优级纯或更高纯度）加入至75 mL水中，混匀。

7.3.1.2 20%六次甲基四胺溶液：称取200g六次甲基四胺试剂溶解于700mL水中，然后定容至1000mL容量瓶中，摇匀备用。

7.3.1.3 0.2%二甲酚橙指示剂：称取0.2g二甲酚橙指示剂于烧杯中，加适量水溶解，然后定容至100mL容量瓶中，摇匀，使用期限两周。

7.3.1.4 0.05mol/L EDTA-2Na标准滴定溶液。

7.3.2 仪器设备

7.3.2.1 分析天平：感量0.0001g。

7.3.2.2 酸式滴定管：50mL。

7.3.3 方法原理

将样品处理后，用酸溶解，加适量水，以六次甲基四胺调节溶液的pH值至5～6，以二甲酚橙为指示剂，用EDTA-2Na标准滴定溶液滴定至颜色由紫红色变为亮黄色即为终点。

7.3.4 测定步骤

准确称取样品0.2-0.5g（精确至0.0002g），加5mL（1+3）的盐酸（7.3.1.1），加入适量水加热溶解，加2滴二甲酚橙指示剂（7.3.1.3），然后滴加六次甲基四胺溶液（7.3.1.2）至溶液呈稳定的紫红色后再过量5mL，用0.05mol/L EDTA标准溶液（7.3.1.4）滴定至溶液由紫红色突跃为亮黄色或橙黄色即为终点。

7.3.5 结果计算：

试样中锌含量以计，质量分数用（%）表示，按以下式（1）计算：

┈┈┈┈┈┈┈（1）

式中：

c­­­­——乙二胺四乙酸二钠标准滴定溶液的浓度，mol/L；

V——滴定样品消耗乙二胺四乙酸二钠标准溶液的体积，mL；

m——称样试样的质量，g；

0.06539——与1.00mL乙二胺四乙酸二钠标准溶液（cEDTA-2Na=1.000mol/L）相当的锌的质量，g。

测定结果以两次平形测定的算术平均值表示，结果保留两位有效数字。

7.3.6 精密度

在重复性条件下，两次独立测定结果绝对差值不得超过0.3%。

7.4 苏氨酸及苏氨酸锌含量的测定

7.4.1 试剂或材料

7.4.1.1 硫酸：分析纯。

7.4.1.2 氢氧化钠：分析纯。

7.4.1.3 硼酸：分析纯。

7.4.1.4 蔗糖：分析纯。

7.4.1.5 硫酸铵：分析纯。

7.4.1.6 混合催化剂：称取0.4g五水硫酸铜、6.0g硫酸钾或硫酸钠，研磨混匀，或购买商品化的凯氏定氮催化剂片。

7.4.1.7 硼酸吸收液I：称取20g硼酸，用水溶解并稀释至1000mL。

7.4.1.8 硼酸吸收液II：1%硼酸水溶液1000mL,加入0.1%溴甲酚绿乙醇10mL，0.1%甲基红乙醇溶液7mL，4%氢氧化钠水溶液0.5mL，混匀，室温保存期为1个月（全自动程序用）。

7.4.1.9 氢氧化钠溶液：称取40g氢氧化钠，用水溶液，待冷却至室温后，用水稀释至100mL。

7.4.1.10 0.1mol/L盐酸标准滴定溶液。

7.4.1.11 甲基红乙醇溶液：称取0.1g甲基红，用乙醇溶液溶解并稀释至100mL。

7.4.1.12 溴甲酚绿乙醇溶液：称取0.5g溴甲酚绿，用乙醇溶解并稀释至100mL。

7.4.1.13 混合指示剂溶液：将甲基红乙醇溶液和溴甲酚绿乙醇溶液等体积混合。该溶液室温避光保存，有效期为3个月。

7.4.2 仪器设备

7.4.2.1 消煮炉或电炉。

7.4.2.2 消煮管。

7.4.2.3 凯氏蒸馏装置：常量直接蒸馏式或半微量水蒸气蒸馏式。

7.4.2.4 定氮仪：以凯氏原理制造的各类型半自动或全自动定氮仪。

7.4.2.5 酸式滴定管：50mL。

7.4.3 苏氨酸含量及苏氨酸锌含量的测定

7.4.3.1 方法原理

样品在催化剂的作用下，经硫酸消解，含氮化合物转化成硫酸铵，加碱蒸馏使氮逸出，用硼酸吸收后，再用盐酸标准滴定溶液滴定，根据氮含量计算出苏氨酸的含量，从而计算出苏氨酸锌的含量。

7.4.3.2 测定步骤

7.4.3.2.1 试样消煮

7.4.3.2.1.1 凯氏烧瓶消煮法：

称取试样1g（准确至0.0001g），置于凯氏烧瓶中，加入6.4g混合催化剂（7.4.1.6），混匀，加入12mL硫酸（7.4.1.1）和2粒玻璃珠，将凯氏烧瓶置于电炉上，开始于约200℃加热，待试样焦化、泡沫消失后，再提高温度至约450℃，直至呈透明的蓝绿色，然后继续加热至少2h。取出，冷却至室温。平行做两份试验。

7.4.3.2.1.2 消煮管消煮：

称取试样1g（准确至0.0001g），放入消煮管中，加入2片凯氏定氮催化剂片或6.4g混合催化剂（7.4.1.6），12mL硫酸（7.4.1.1），于450℃消煮炉上消化2h直至呈透明的蓝绿色，然后继续加热至少2h。取出，冷却至室温。平行做两份试验。

7.4.3.2.2 氨的蒸馏

待试样消煮液冷却，加入20mL蒸馏水，摇匀，冷却。将蒸馏装置的冷凝管末端浸入装有25 mL硼酸吸收液I（7.4.1.7）和2滴混合指示剂（7.4.1.13）的锥形瓶中。然后小心地向凯氏烧瓶中加入50mL 氢氧化钠溶液（7.4.1.9），摇匀后加热蒸馏，直至流出液体积约为100mL。降下锥形瓶，使冷凝管末端离开液面，继续蒸馏1min～2min，至流出液pH值为中性。用水冲洗冷凝管末端，洗液均需流入锥形瓶内，然后停止蒸馏。

采用半自动凯氏定氮仪时，将带消煮液的消煮管插在蒸馏装置上，以25mL硼酸吸收液I（7.4.1.7）为吸收液，加入2滴混合指示剂（7.4.1.13），蒸馏装置的冷凝管末端要浸入装有吸收液的锥形瓶内，然后向消煮管中加入50mL氢氧化钠溶液（7.4.1.9）进行蒸馏，至流出液pH值为中性。蒸馏时间以吸收液体积达到约100mL时为宜。降下锥形瓶，用水冲洗冷凝管末端，洗液均需流入锥形瓶内。

采用全自动凯氏定氮仪时，按仪器操作说明书进行测定。

7.4.3.2.3 滴定

用0.1mol／L盐酸标准滴定溶液（7.4.1.10）滴定吸收液，溶液由蓝绿色变成灰红色为终点。记录样品消耗盐酸标准滴定溶液的体积。

7.4.3.2.4 蒸馏步骤查验

精确称取0.2g硫酸铵（精确至0.0001g），代替试样，按7.4.3.2.2进行操作，测得硫酸铵含氮量应为（21.19±0.2）％，否则应检查加碱、蒸馏和滴定各步骤是否正确。

7.4.3.2.5 空白测定

精确称取0.5g蔗糖（精确至0.0001g），代替试样，按7.4.3.2.2进行空白测定，消耗0.1 mol／L盐酸标准滴定溶液的体积不得超过0.2mL。

7.4.3.2.6 苏氨酸及苏氨酸锌含量的结果计算：

试样中苏氨酸含量以计，质量分数用（%）表示，按以下公式（2）计算：

┈┈┈┈┈┈┈（2）

试样中苏氨酸锌含量以计，质量分数用（%）表示，按以下公式（3）计算：

┈┈┈┈┈┈┈（3）

式中：

c —— 盐酸标准溶液的浓度，单位为摩尔每升（mol/L）；

V1—— 样品消耗盐酸标准滴定溶液的体积，单位为毫升（mL）；

V0—— 空白样品消耗盐酸标准滴定溶液的体积，单位为毫升（mL）；

m1—— 试样的质量，单位为克（g）；

m2—— 试样的质量，单位为克（g）；

0.11912——与1.00mL盐酸标准溶液（cHCl=1.000mol/L）相当的苏氨酸的质量，g。

0.33763——与1.00mL盐酸标准溶液（cHCl=1.000mol/L）相当的苏氨酸锌的质量，g。

7.4.3.2.7 精密度

在重复性条件下，两次独立测定结果的绝对差值不超过0.5%。

7.5 粒度的测定

按GB/T 5917的规定执行。

7.6 水分的测定

按照GB/T 6435的规定执行。

7.7 砷的测定

按照GB/T 13079的规定执行。

7.8 铅的测定

按照GB/T13080的规定执行。

7.9 镉的测定

按照GB 13082的规定执行。

7.10 净含量及允许短缺量

按照JJF1070-2005执行。

**8 检验规则**

8.1 组批

产品以同一班组、同种原料生产的在规定限度内具有同一性质和质量产品为一批。

8.2 出厂检验

每批产品应进行出厂检验，出厂检验项目包括水分、粒度、锌、总苏氨酸。

8.3 型式检验

型式检验项目为本文件第5章规定的所有项目。在正常生产情况下，每半年至少进行1次型式检验。有下列情况之一时，亦应进行型式检验有下列情形之一时应进行型式检验：

a） 产品定型投产时；

b） 生产工艺、配方或主要原料来源有较大改变，可能影响产品质量时；

c） 停产3个月以上，重新恢复生产时；

d） 出厂检验结果与上次型式检验结果有较大差异时；

e） 饲料行政管理部门提出检验要求时。

8.4 判定规则

8.4.1 所有项目全部合格，判定为该批次产品合格。

8.4.2 检测结果中有任何指标不符合本部分规定时，可自同批产品中重新加倍取样进行复检。复检结果有一项指标不符合本部分规定，即判定该批产品不合格。

8.4.3 各项目指标的极限数值判定按GB/T 8170中修约值比较法执行。

**9 标签、包装、运输、贮存和保质期**

9.1 标签

标签应符合GB10648的规定。

9.2 包装

采用复合编织袋，内加塑料袋包装。净含量应符合《定量包装商品计量监督管理办法》的规定。

9.3 运输

产品在运输过程中应防潮、防高温、防包装破损，严禁与有毒有害物质混运。

9.4 贮存

产品应贮存于通风、干燥、无污染、无有毒有害物质处。

9.5 保质期

产品在规定的贮存条件下，从生产之日起保质期为12个月。