COMMISSION DIRECTIVE 1999/76/EC

of 23 July 1999

establishing a Community method of analysis for the determination of lasalocid sodium in feedingstuffs

(Text with EEA relevance)

THE COMMISSION OF THE EUROPEAN COMMUNITIES,

Having regard to the Treaty establishing the European Community,

Having regard to Council Directive 70/373/EEC of 20 July 1970 on the introduction of Community methods of sampling and analysis for the official control of feedingstuffs [1], as last amended by the Act of Accession of Austria, Finland and Sweden, and in particular Article 2 thereof,

(1) Whereas Directive 70/373/EEC stipulates that official controls of feedingstuffs for the purpose of checking compliance with the requirements arising under the laws, regulations and administrative provisions governing their quality and composition must be carried out using Community methods of sampling and analysis;

(2) Whereas Council Directive 70/524/EEC of 23 November 1970 concerning additives in feedingstuffs [2], as last amended by Commission Regulation (EC) No 866/1999 of 26 April 1999 [3] stipulates that the lasalocid sodium content must be indicated on the labelling where these substances are added to premixtures and feedingstuffs;

(3) Whereas Community methods of analysis must be established for checking these substances;

(4) Whereas the measures provided for in this Directive are in accordance with the opinion of the Standing Committee for Feedingstuffs,

HAS ADOPTED THIS DIRECTIVE:

Article 1

The Member States shall provide that analyses conducted with a view to official controls of the lasalocid content of feedingstuffs and premixtures are carried out using the method set out in Annex hereto.

Article 2

The Member States shall bring into force, not later than 31 January 2000, the laws, regulations or administrative provisions necessary to comply with the provisions of this Directive. They shall immediately inform the Commission thereof.

They shall apply the measures from 1 February 2000.

When Member States adopt these measures, they shall contain a reference to this Directive or shall be accompanied by such reference at the time of their official publication. The procedure for such reference shall be adopted by Member States.

Article 3

This Directive shall enter into force on the 20th day following its publication in the *Official Journal of the European Communities*.

Article 4

This Directive is addressed to the Member States.

Done at Brussels, 23 July 1999.

For the Commission

Franz FISCHLER

Member of the Commission

[1] OJ L 170, 3.8.1970, p. 2.
[2] OJ L 270, 14.12.1970, p. 1.
[3] OJ L 108, 27.4.1999, p. 20.

ANNEX

DETERMINATION OF LASALOCID SODIUM

Sodium salt of a polyether monocarboxylic acid produced by Streptomyces lasaliensis

1. **Purpose and scope**

The method is for the determination of lasalocid sodium in feedingstuffs and premixtures. The limit of detection is 5 mg/kg, the limit of determination is 30 mg/kg.

2. **Principle**

Lasalocid sodium is extracted from the sample into acidified methanol and determined by reversed-phase high performance liquid chromatography (HPLC) using a spectrofluorimetric detector.

3. **Reagents**

3.1. Potassium dihydrogen phosphate (KH_2PO_4)

3.2. Orthophosphoric acid, w = 85 %

3.3. Orthophosphoric acid solution, σ = 20 %

Dilute 23,5 ml of orthophosphoric acid (3.2) to 100 ml with water.

3.4. 6-Methyl-2-heptylamine (1,5-dimethylhexylamine), w = 99 %

3.5. Methanol, HPLC grade

3.6. Hydrochloric acid, ρ_{20} 1,19 g/ml

3.7. Phosphate buffer solution, c = 0,01 mol/l

Dissolve 1,36 g of KH_2PO_4 (3.1) in 500 ml of water (3.11), add 3,5 ml of orthophosphoric acid (3.2) and 10,0 ml of 6-methyl-2-heptylamine (3.4). Adjust the pH to 4,0 with orthophosphoric acid solution (3.3) and dilute to 1 000 ml with water (3.11).

3.8. Acidified methanol

Transfer 5,0 ml of hydrochloric acid (3.6) into a 1 000 ml graduated flask, make up to the mark with methanol (3.5) and mix. This solution must be prepared freshly before use.

3.9. HPLC mobile phase, phosphate buffer-methanol solution 5 + 95 (V + V)

Mix 5 ml of phosphate buffer solution (3.7) with 95 ml of methanol (3.5).

3.10. Lasalocid sodium standard substance with guaranteed purity, $C_{34}H_{53}O_8Na$ (sodium salt of a polyether monocarboxylic acid produced by Streptomyces lasaliensis), E763

3.10.1. Lasalocid sodium stock standard solution, 500 µg/ml

Weigh to the nearest 0,1 mg, 50 mg of lasalocid sodium (3.10) into a 100 ml graduated flask, dissolve in acidified methanol (3.8), make up to the mark with the same solvent and mix. This solution must be freshly prepared before use.

3.10.2. Lasalocid sodium intermediate standard solution, 50 µg/ml

Pipette 10,0 ml of stock standard solution (3.10.1) into a 100 ml graduated flask, make up to the mark with acidified methanol (3.8) and mix. This solution must be prepared freshly before use.

3.10.3. Calibration solutions

Into a series of 50 ml graduated flasks transfer 1.0, 2.0, 4.0, 5.0 and 10.0 ml of the intermediate standard solution (3.10.2). Make up to the mark with acidified methanol (3.8) and mix. These solutions correspond to 1.0, 2.0, 4.0, 5.0 and 10.0 µg of lasalocid sodium per ml respectively. These solutions must be prepared freshly before use.

3.11. Water, HPLC grade