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European Union Reference Laboratory for Feed Additives



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**EURL Evaluation Report on the Analytical Methods  
submitted in connection with the Application for the  
Authorisation of Feed Additives according to  
Regulation (EC) No 1831/2003**

**Dossier related to:** **FAD-2010-0209**  
**CRL/100272**

**Name of feed additive:** ***Lignosulphonate***

**Active Substance(s):** ***Lignosulphonate***

**Rapporteur Laboratory:** **European Union Reference Laboratory  
for Feed Additives (EURL-FA)  
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**Date:** **19/04/2011**

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**Date:** **19/04//2011**

## EXECUTIVE SUMMARY

In the current application authorisation is sought under articles 4(1) and 10(2) for *Lignosulphonate* under the category of 'technological additives' functional groups 'g) binders' and 'k) silage additives' according to Annex I of Regulation (EC) No 1831/2003. Specifically, authorisation is sought for the use of *Lignosulphonate* in *premixtures*, *feedingstuffs*, *water* and *silage* for all animal species. It is intended to be marketed either as spray dried powder or as an aqueous solution. *Lignosulphonate* is intended to be mixed as *binder* in *premixtures* and *feedingstuffs*, as *silage* additive, or used directly in *water*. Unlike the previous regulation, where no condition of use was set, the Applicant proposed the following concentration levels: 50 to 1000 mg/kg for *silage* and 1 to 3 g/kg for *feedingstuffs*.

For the determination of *Lignosulphonate* in the *feed additive* the Applicant proposed an indirect determination based on reducing sugars (derived from hemicellulose) and total ash (including calcium, sodium, magnesium, sulphate and sulphite), as described in the JECFA monograph (40-65 - new specification for calcium lignosulfonate). No performance characteristics of these methods are provided. However, the EURL considers these methods suitable to be used within the frame of official control.

The Applicant did not provide any experimental method or data for the determination of *Lignosulphonate* in *premixtures*, *feedingstuffs*, *silage* and *water*. Furthermore, the unambiguous determination of the content of *Lignosulphonate* added to *premixtures*, *feedingstuffs* and *silage* cannot be determined analytically. Therefore the EURL cannot evaluate nor recommend any method for official control to determine *Lignosulphonate* in *premixtures*, *feedingstuffs*, *silage* and *water*.

Further testing or validation of the methods to be performed through the consortium of National Reference Laboratories as specified by article 10 (Commission Regulation (EC) No 378/2005) is not considered necessary.

## KEYWORDS

*Lignosulphonate*, technological additives, binders, silage additives, all animal species.

## 1. BACKGROUND

In the current application authorisation is sought under articles 4(1) (new use in water and silage) and 10(2) (re-evaluation of additives already authorised under council directive 70/524/EEC) for *Lignosulphonate* under the category of 'technological additives' functional groups 'g) binders' and 'k) silage additives' according to Annex I of Regulation (EC) No 1831/2003 [1]. Specifically, authorisation is sought for the use of *Lignosulphonate* in *premixtures, feedingstuffs, water and silage* for all animal species [2]. *Lignosulphonate* is a complex light-brown, amorphous natural polymer, derived from lignin; composed of phenylpropane monomers, which are covalently linked through a variety of chemical bonds [3]. It is intended to be marketed either as spray dried powder or as an aqueous solution. Unlike the previous regulation, where no condition of use was set [4], the Applicant proposed the following concentration levels: 50 to 1000 mg/kg for *silage* and 1 to 3 g/kg for *feedingstuffs* [2, 5].

## 2. TERMS OF REFERENCE

In accordance with Article 5 of Regulation (EC) No 378/2005, as last amended by Regulation (EC) No 885/2009, on detailed rules for the implementation of Regulation (EC) No 1831/2003 of the European Parliament and of the Council as regards the duties and the tasks of the European Union Reference Laboratory concerning applications for authorisations of feed additives, the EURL is requested to submit a full evaluation report to the European Food Safety Authority for each application or group of applications. For this particular dossier, the methods of analysis submitted in connection with *Lignosulphonate*, and their suitability to be used for official controls in the frame of the authorisation, were evaluated.

## 3. EVALUATION

### *Identification/Characterisation of the feed additive*

#### *Qualitative and quantitative composition of impurities in the additive*

When required by EU legislation, analytical methods for official control of undesirable substances in the additive (e.g. arsenic, cadmium, lead, mercury and dioxins) are available from the respective European Union Reference Laboratories [6].

***Description of the analytical methods for the determination of the active substance in feed additive, premixtures and feedingstuffs***

For the identification of *Lignosulphonate* in the *feed additive* the Applicant proposed UV spectroscopy at 280 nm as described in JECFA monograph (40-65 - new specification for calcium lignosulfonate) [7]. For the determination of *Lignosulphonate* in the *feed additive* the Applicant proposed an indirect determination based on (i) reducing sugars (derived from hemicellulose) and (ii) total ash (including calcium, sodium, magnesium, sulphate and sulphite), as described in above mentioned JECFA monograph [7].

For the determination of reducing sugars, the samples are dissolved and diluted with deionized water and injected in flow injection analyzer. The solutions are then dialyzed through a cellulose membrane and 1M NaOH, CaCl<sub>2</sub> and p-hydroxybenzoic hydrazide (PHBH) are added. The mixture is heated at 90 °C and analyzed at 410 nm, using standard glucose solutions as calibration.

For the determination of total ash the samples are weighed in a special platinum crucible, previously cleaned with potassium bisulfate and dried at 105 °C, and heated over a flame. The samples are ignited at 550 °C for 1 hour and at 900 °C for approximately 10 minutes, until ash is white. The weight of the residue is determined, after cooling in dessicator.

No performance characteristics of these methods are provided. However, the EURL considers these methods suitable to be used within the frame of official control.

The Applicant did not provide any experimental method or data for the determination of *Lignosulphonate* in *premixtures*, *feedingstuffs*, *silage* and *water*. Furthermore, the unambiguous determination of the content of *Lignosulphonate* added to *premixtures*, *feedingstuffs* and *silage* cannot be determined analytically. Therefore the EURL cannot evaluate nor recommend any method for official control to determine *Lignosulphonate* in *premixtures*, *feedingstuffs*, *silage* and *water*.

Further testing or validation of the methods to be performed through the consortium of National Reference Laboratories as specified by article 10 (Commission Regulation (EC) No 378/2005) is not considered necessary.

#### 4. CONCLUSIONS AND RECOMMENDATIONS

In the frame of this authorisation the EURL recommends for official control the indirect method, described in the JECFA monograph (40-65 - new specification for calcium lignosulfonate), based on the determination of reducing sugars and total ash, for the determination of *Lignosulphonate* in the *feed additive*.

The Applicant did not provide any experimental method or data for the determination of *Lignosulphonate* in *premixtures*, *feedingstuffs*, *silage* and *water*. Furthermore, the unambiguous determination of the content of *Lignosulphonate* added to *premixtures*, *feedingstuffs* and *silage* cannot be determined analytically. Therefore the EURL cannot evaluate nor recommend any method for official control to determine *Lignosulphonate* in *premixtures*, *feedingstuffs*, *silage* and *water*.

#### *Recommended text for the register entry (analytical method)*

For the determination of *Lignosulphonate* in the *feed additive*:

- indirect determination by total ash and reducing sugars from JECFA monograph (40-65 - new specification for calcium lignosulfonate)

#### 5. DOCUMENTATION AND SAMPLES PROVIDED TO EURL

In accordance with the requirements of Regulation (EC) No 1831/2003, samples of the additive *Lignosulphonate* have been sent to the European Union Reference Laboratory for Feed Additives. The dossier has been made available to the EURL by EFSA.

#### 6. REFERENCES

- [1] \*Application/Ref: SANCO/D/2: Forw.Appl.1831/00125 (10057)- 2010
- [2] \*Application, Annex A, Proposal for register entry
- [3] \*Technical Dossier, Section II.2.1 Identity of the additive
- [4] COUNCIL DIRECTIVE 70/524/EEC of 23 November 1970 concerning additives in feedingstuffs
- [5] \*Technical Dossier, Section II.2.5.1. Proposed mode of use in animal nutrition
- [6] Commission Regulation (EC) No 776/2006 amending Annex VII to Regulation (EC) No 882/2004 of the European Parliament and of the Council as regards to Community Reference Laboratories

[7] \*Technical Dossier, Annex 2.2.2.a JECFA Calcium\_Lignosulfonate\_40\_65 New specification

\*Refers to Dossier No: FAD-2010-0209

## **7. RAPPORTEUR LABORATORY & NATIONAL REFERENCE LABORATORIES**

The Rapporteur Laboratory for this evaluation was European Union Reference Laboratory for Feed Additives, IRMM, Geel, Belgium. This report is in accordance with the opinion of the consortium of National Reference Laboratories as referred to in Article 6(2) of Commission Regulation (EC) No 378/2005, as last amended by Regulation (EC) No 885/2009.

## **8. ACKNOWLEDGEMENTS**

The following National Reference Laboratories contributed to this report:

- Centro di referenza nazionale per la sorveglianza ed il controllo degli alimenti per gli animali (CReAA), Torino (IT)
- Plantedirektoratet, Laboratorium for Foder og Gødning, Lyngby (DK)
- Ústřední kontrolní a zkušební ústav zemědělský (ÚKZÚZ), Praha (CZ)
- Kmetijski inštitut Slovenije, Ljubljana (SI)
- Laboratoire de Rennes, SCL L35, Service Commun des Laboratoires, Rennes (FR)