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JRC.D.5/SFB/CvH/ZE/Ares

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

Natrolite-phonolite
(FAD-2010-0238; CRL/100234)



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in connection with the Application for Authorisation of a
Feed Additive according to Regulation (EC) No 1831/2003**

Dossier related to: **FAD-2010-0238 - CRL/100234**

Name of Feed Additive: **Natrolite-phonolite E566**

Active Agent (s): **Natrolite-phonolite**

Rapporteur Laboratory: **European Union Reference Laboratory for
Feed Additives (EURL-FA)
Geel, Belgium**

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Date: **03/09/2015**

Report approved by: **Christoph von Holst**
Date: **04/09/2015**

EXECUTIVE SUMMARY

In the current application authorisation is sought under articles 10(2) for *natrolite-phonolite*, under the category/functional group 1(i) 'technological additives/'anticaking agents', according to the classification system of Annex I of Regulation (EC) No 1831/2003. The authorisation is sought for the use of the *feed additive* for all animal species.

According to the Applicant the *feed additive* is an odourless grey-brown powder obtained by grinding mineral-based substances derived from volcanic rocks. The *feed additive* consists of natural mixture of aluminium silicates, natrolite, feldspar and alkaline, alkaline-earth and aluminium hydro-silicates. According to the Applicant, the content of natrolite (zeolite) in the *feed additive* is ranging from 43 to 46.5 %. The *feed additive* is intended to be used directly in *feedingstuffs* or through *premixtures* to ensure flowability within the storage silos. The Applicant proposed a maximum inclusion level of the *feed additive* in complete *feedingstuffs* of 25 g/kg.

For the characterisation of the *feed additive* (*natrolite-phonolite*) the Applicant suggested X-Ray diffraction (XRD) analysis to determine the main mineralogical components. This method - described in the EN 13925 standard - was previously recommended by the EURL in the frame of the evaluation of FAD-2010-0061. Four main mineralogical compositions were reported for four typical natrolite-phonolite samples: natrolite, K-feldspar, ägirinaugit, and wollastonite. In addition, the Applicant applied X-Ray fluorescence spectrometry (XRF) as described in the ISO 29581-2 standard and reported elemental composition for the *feed additive* consisting of SiO₂, Al₂O₃, CaO, Na₂O, K₂O and Fe₂O₃.

Based on the available experimental data, the EURL recommends for official control the XRD and XRF methods described in the EN 13925 and the ISO 29581-2 standards for the characterisation of the *feed additive*.

The unambiguous determination of the *feed additive* added to *premixtures* and *feedingstuffs* is not achievable experimentally. Therefore, the EURL cannot evaluate nor recommend any method for official control for the determination of *natrolite-phonolite* in *premixtures* and *feedingstuffs*.

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

Natrolite-phonolite, technological additives, anticaking agents, all animal species

1. BACKGROUND

In the current application authorisation is sought under articles 10(2) (re-evaluation of the already authorised additives under provisions of Council Directive 70/524/EEC) for *natrolite-phonolite*, under the category/functional group 1(i) 'technological additives'/anticaking agents', according to the classification system of Annex I of Regulation (EC) No 1831/2003. The authorisation is sought for the use of the *feed additive* for all animal species [1,2].

According to the Applicant the *feed additive* is an odourless grey-brown powder obtained by grinding mineral-based substances derived from volcanic rocks [3]. The *feed additive* consists of natural mixture of aluminium silicates, natrolite, feldspar and alkaline, alkaline-earth and aluminium hydro-silicates [2,3]. According to the Applicant, the content of natrolite (zeolite) in the *feed additive* ranges from 43 to 46.5 % [3]. The *feed additive* is intended to be used directly in *feedingstuffs* or through *premixtures* to ensure flowability within the storage silos. The Applicant proposed a maximum inclusion level of the *feed additive* in complete *feedingstuffs* of 25 g/kg [2].

Note: The EURL evaluated another natrolite-phonolite dossier (FAD-2010-0061).

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 *natrolite-phonolite* 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, mycotoxins and dioxins) are available from the respective European Union Reference Laboratories [4].

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

For the characterisation of the *feed additive (natrolite-phonolite)* the Applicant suggested using an X-Ray diffraction (XRD) analysis to determine its main mineralogical components [2,3]. This method - described in the EN 13925 standard [5] - was previously recommended by the EURL in the frame of the evaluation of FAD-2010-0061 [6].

The following main mineralogical compositions were reported for four typical natrolite-phonolite samples [7]:

<i>natrolite</i>	43.1 - 46.3%;
<i>K-feldspar</i>	27.1 - 35.2%;
<i>ägirinaugit</i>	9.6 - 11.6%;
<i>wollastonite</i>	6.6 - 12.2%;

In addition, the Applicant applied X-Ray fluorescence spectrometry (XRF) as described in the ISO 29581-2 standard [8] and derived the following "observed" elemental composition for the *feed additive* [3], which are compared to "typical" natrolite-phonolite compositions [7] in the table hereafter:

Composition	Observed [3]	Typical [7]
SiO ₂	47.2 - 50.8 %	48.2 - 50.6 %
Al ₂ O ₃	16.3 - 18.6 %	17.4 - 18.6 %
CaO	6.2 - 8.7 %	6.7 - 8.5 %
Na ₂ O	5.3 - 7.5 %	5.5 - 8.2 %
K ₂ O	4.4 - 6.1 %	4.1 - 5.7 %
Fe ₂ O ₃	4.1 - 4.5 %	3.9 - 4.3 %

Based on the available experimental data, the EURL recommends for official control the XRD and XRF methods described by the EN 13925 and the ISO 29581-2 standards for the characterisation of the *feed additive*.

The Applicant provided no experimental data or any analytical method for the determination of the *natrolite-phonolite* in *premixtures* and *feedingstuffs* as the unambiguous determination of the *feed additive* added to the matrices is not achievable experimentally. Therefore, the EURL cannot evaluate nor recommend any method for official control for the determination of *natrolite-phonolite* in *premixtures* and *feedingstuffs*.

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

The EURL recommends for official control two internationally recognised standards: - EN 13925, based on X-ray diffraction (XRD); and - ISO 29581-2, based on X-ray fluorescence spectrometry (XRF) for characterisation of the *feed additive (natrolite-phonolite)*.

The unambiguous quantification of the *feed additive* added to *premixtures* and *feedingstuffs* is not achievable experimentally. Therefore, the EURL cannot evaluate nor recommend any method for official control for the quantification of *natrolite-phonolite* in *premixtures* and *feedingstuffs*.

Recommended text for the register entry (analytical method)

Characterisation of the *feed additive*:

- X-ray diffraction (XRD) – EN 13925; and
- X-ray fluorescence spectrometry (XRF) – ISO 29581-2

5. DOCUMENTATION AND SAMPLES PROVIDED TO EURL

In accordance with the requirements of Regulation (EC) No 1831/2003, reference samples of *natrolite-phonolite* 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, Reference SANCO/G1: Forw. Appl. 1831/0027-2014
- [2] *Application, Proposal for Register Entry
- [3] *Technical dossier, Section II: Identity, characterisation and conditions of use of the additive; methods of analysis
- [4] 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
- [5] EN 13925-1,2:2003; EN 13925-3:2005 – Non-destructive testing. X-ray diffraction from polycrystalline and amorphous materials. General principles, procedures, instruments
- [6] FAD-2010-0061 – JRC.D.5/SFB/CvH/JK/mds/Ares (2015)1318765
- [7] *Technical dossier, Section II – Annex_II_1_8
- [8] ISO 29581-2:2010 – Cement - Test methods - Part 2: Chemical analysis by X-ray fluorescence

*Refers to Dossier no: FAD-2010-0238

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

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- Ústřední kontrolní a zkušební ústav zemědělský (ÚKZÚZ), Praha (CZ)
- Centro di referenza nazionale per la sorveglianza ed il controllo degli alimenti per gli animali (CReAA), Torino (IT)
- Państwowy Instytut Weterynaryjny, Pulawy (PL)