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Institute for Reference Materials and
Measurements
**Community Reference Laboratory for Feed
Additives**

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

Dossier related to: **FAD-2009-0017
CRL/090015**

Name of Additive: **Clinoptilolite of sedimentary origin (E 568)**

Rapporteur Laboratory: **Community Reference Laboratory for Feed
Additives (CRL-FA)**

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14/07/2010**

EXECUTIVE SUMMARY

In the current application authorisation is sought for *Clinoptilolite* under article 4(1) and article 10(2), under the category 'technological additives' functional group (1f) 'binders' according to Annex I of Regulation (EC) No 1831/2003. Specifically, authorisation is sought for the *feed additive* to be placed on the market in the form of powder containing a minimum of 80% Clinoptilolite and a maximum of 20% clay. The intended use of the current application is for all animal species. The additive can be used in pelleted mixed feeds and in feeds in the form of meal at a maximum content of 20 g/kg.

For the determination of *Clinoptilolite* in the *feed additive* the applicant proposes X-ray diffraction method. The sample diffractograms are compared to the standard pattern of the Hector material, containing 90% *clinoptilolite*. The CRL recommends for official control the method submitted by the applicant for the determination of *Clinoptilolite* in the *feed additive*.

The unambiguous determination of the *Clinoptilolite* content added to *premixtures* or *feedingstuffs* is not achievable by analysis. The Applicant did not provide any experimental method or data on this matter. Furthermore, no international standard methods of analysis could be identified. Therefore the CRL cannot evaluate nor recommend any method for official control to determine *Clinoptilolite* in *premixtures* or *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

Clinoptilolite, technological additives, binder, all species

1. BACKGROUND

Clinoptilolite (E.568) is a *feed additive* for which authorisation is sought under Article 4(1) (new use) and Article 10(2) (re-evaluation of an authorized additive by European Regulation (EC) No 1810/2005) the category of 'technological additives' functional group (1f) 'binders' according to Annex I of Regulation (EC) No 1831/2003 [1]. Specifically, authorisation is sought for the *feed additive* to be placed on the market in the form of powder containing a minimum of 80% *Clinoptilolite* and maximum of 20% clay [2]. The gross formula of *Clinoptilolite* is $(\text{NaK})_4 \text{CaAl}_6 \text{Si}_{30}\text{O}_{72} \cdot 24 \text{H}_2\text{O}$ [3]. The intended use of the current application is for all animal species. The additive can be used in pelleted mixed feeds and in feeds in the form of meal in a maximum content of 20 g/kg [2].

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 tasks of the Community Reference Laboratory concerning applications for authorisations of feed additives, the CRL is requested to submit a full evaluation report to the European Food Safety Authority (EFSA) for each application or group of applications. For this dossier, the methods of analysis submitted in connection with the *Clinoptilolite* dossier 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

Quantitative and qualitative composition of impurities in the additive

For undesirable substances (i.e. arsenic, cadmium, mercury, lead and selenium) internationally recognised standard methods are available at the respective Community Reference Laboratories, in accordance with Commission Regulation (EC) No 776/2006.

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

For the determination of *Clinoptilolite* in *feed additive* the applicant proposes X-ray diffraction method, where the sample diffractograms [4] are compared to the standard pattern of the Hector

material, with a *Clinoptilolite* content close to 90% [5]. To determine the content of *Clinoptilolite* in the *feed additive*, the mean values of ratios of peak intensity were calculated [4].

The CRL recommends for official control the method submitted by the applicant for the determination of *Clinoptilolite* in the *feed additive*.

The *feed additive* was further characterised by AAS to derive the following composition [7]:

- $\text{SiO}_2 = 70\%$
- $\text{Al}_2\text{O}_3 = 13\%$
- $\text{Fe}_2\text{O}_3 = 1.5\%$
- $\text{CaO} = 3.5\%$

The unambiguous determination of the content of exogenous *Clinoptilolite* added to *premixtures* or *feedingstuffs* is not achievable by analysis. The Applicant did not provide any experimental method or data on this matter. Furthermore, no international standard methods of analysis could be identified. Therefore the CRL cannot evaluate nor recommend any method for official control to determine *Clinoptilolite* in *premixtures* or *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

In the frame of this authorisation the CRL recommends for official control the X-ray diffraction method submitted by the applicant for the determination of *Clinoptilolite* in the *feed additive*. No methods are recommended by the CRL for the determination of *Clinoptilolite* 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.

Recommended text for the register entry, fourth column (Composition, chemical formula, description, analytical method)

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

- X-ray diffraction method.

5. DOCUMENTATION AND SAMPLES PROVIDED TO CRL

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

6. REFERENCES

- [1] *Application, Reference SANCO/D/2 Forw. Appl. 1831/025-2009
- [2] *FAD-2009-0017_RegEntry.
- [3] *Technical Dossier, section II; Identity, Description of the method of production
- [4] *Supplementary information, Annex 1, "X-ray diffraction"
- [5] *Supplementary information, "FAD-2009-0014 Clinoptilolite"
- [6] *Supplementary information, Annex 3, "X-ray analysis"
- [7] *Technical Dossier, section II, "Chemical composition"

* Refers to Dossier No: FAD-2009-0017

7. RAPPORTEUR LABORATORY

The Rapporteur Laboratory for this evaluation was Community 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.

8. ACKNOWLEDGEMENTS

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- Österreichische Agentur für Gesundheit und Ernährungssicherheit (AGES), Austria
- Centro di referenza nazionale per la sorveglianza ed il controllo degli alimenti per gli animali (CReAA), Italia