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Directorate F - Health, Consumers & Reference Materials (Geel/Ispra)
European Union Reference Laboratory for Feed Additives

JRC F.5/CvH/ZE/AS/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

Kaolinitic clays, free of asbestos (E 559)

(FAD-2010-0282; CRL/100186)

European Union Reference Laboratory
Feed Additives

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Dossier related to:	**FAD-2010-0282 - CRL/100186**
Name of Feed Additive:	***Kaolinitic clays, free of asbestos (E 559)***
Active Agent (s):	**Kaolinite**
Rapporteur Laboratory:	**European Union Reference Laboratory for Feed Additives (EURL-FA)** **JRC Geel, Belgium**
Report prepared by:	**Zigmas Ezerskis**
Report checked by: Date:	**Stefano Bellorini** **16/12/2019**
Report approved by: Date:	**Christoph von Holst** **16/12/2019**

EXECUTIVE SUMMARY

In the current application an authorisation is sought under Article 10(2) for *kaolinitic clays, free of asbestos (E 559)* under the category / functional group 1(g) and 1(i) "technological additives"/"binders" and "anticaking agents", according to the classification system of Annex I of Regulation (EC) No 1831/2003. Specifically, the authorisation is sought for the use of the *feed additive* for all animal species.

The *feed additive* is a naturally occurring mixture of complex hydrated aluminium silicates with a minimum content of 50 % (w/w) of *kaolinite*. The *feed additive* also contains other minerals such as mica (muscovite), feldspar, quartz and smectite.

The *feed additive* is intended to be used in *premixtures* and *feedingstuffs*. The Applicant did not propose any limits of the *feed additive* in *feedingstuffs*. However, the suggested inclusion levels of the *feed additive* are ranging from 10 to 50 g/kg *feedingstuffs*.

For the characterisation of the *feed additive* the Applicant suggested to determine its mineralogical and elemental composition. For the determination of the mineralogical composition of the *feed additive* the Applicant submitted the method EN 13925 based on X-ray diffraction (XRD). Furthermore, for the determination of the elemental composition the Applicant submitted the method EN ISO 12677 based on X-ray fluorescence (XRF) spectrometry.

Based on the experimental evidence available, the EURL recommends for official control of the *feed additive* the mineralogical characterisation by X-ray diffraction (XRD) (EN 13925) together with the elemental analysis by X-ray fluorescence (XRF) (EN ISO 12677).

The Applicant provided no analytical method or experimental data for the determination of the *kaolinitic clays, free of asbestos (E 559)* 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 *caolinitic clays, free of asbestos (E 559)* in *premixture*s 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, as last amended by Regulation (EU) 2015/1761) is not considered necessary.

KEYWORDS

Kaolinite, kaolinitic clays, free of asbestos (E 559), technological feed additives, binders, anticaking agents, all animal species

1. BACKGROUND

In the current application an authorisation is sought under Article 10(2) (re-authorisation of additives already authorised under the provisions of the Council Directive 70/524/EEC) for *kaolinitic clays, free of asbestos (E 559)* under the category / functional group 1(g) and 1(i) "technological additives"/"binders" and "anticaking agents", according to the classification system of Annex I of Regulation (EC) No 1831/2003 [1]. Specifically, the authorisation is sought for the use of the *feed additive* for all animal species [2].

The *feed additive* is a naturally occurring mixture of complex hydrated aluminium silicates with a minimum content of 50 % (w/w) of *kaolinite* [2,3]. The *feed additive* also contains other minerals such as mica (muscovite), feldspar, quartz and smectite [3].

The *feed additive* is intended to be used in *premixtures* and *feedingstuffs*. The Applicant did not propose any limits of the *feed additive* in *feedingstuffs*. However, the suggested inclusion levels of the *feed additive* are ranging from 10 to 50 g/kg *feedingstuffs* [2,3].

2. TERMS OF REFERENCE

In accordance with Article 5 of Regulation (EC) No 378/2005, as last amended by Regulation (EU) 2015/1761, 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 *kaolinitic clays, free of asbestos (E 559)* and their suitability to be used for official controls in the frame of the authorisation were evaluated.

3. EVALUATION

Description of the analytical methods for the determination of the active substance in the feed additive, premixtures, feedingstuffs and when appropriate water (section 2.6.1 of the dossier - Annex II of Commission Regulation (EC) No 429/2008)

An evaluation of corresponding methods of analysis is not relevant for the present application. Furthermore, the Applicant provided no analytical method or experimental data for the determination of the *kaolinitic clays, free of asbestos (E 559)* 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 *kaolinitic clays, free of asbestos (E 559)* in *premixture*s and *feedingstuffs*.

Methods of analysis for the determination of the residues of the additive in food (section 2.6.2 of the dossier - Annex II of Commission Regulation (EC) No 429/2008)

An evaluation of corresponding methods of analysis is not relevant for the present application.

Identification/Characterisation of the feed additive (section 2.6.3 of the dossier - Annex II of Commission Regulation (EC) No 429/2008)

For the characterisation of the *feed additive* the Applicant suggested to determine its mineralogical and elemental composition [3].

For the determination of the mineralogical composition of the *feed additive* the Applicant submitted the EN 13925 method based on X-ray diffraction (XRD) [4]. The mineralogical compounds were identified by comparing their XRD patterns to the corresponding reference patterns published in the International Centre for Diffraction Data (ICDD) [5]. Furthermore, quantification was performed by using an internal standard and the established intensity factors for the different minerals [6]. The Applicant analysed several batches of the *feed additive* and a relative standard deviation for *repeatability* (RSD_r) ranging from 0.6 to 9.5 % was derived for a *sepiolite* content ranging from 79 to 91 % (w/w) [3].

Furthermore, for the determination of the elemental composition the Applicant submitted the EN ISO 12677 method based on X-ray fluorescence (XRF) spectrometry [7]. An indicative main elemental composition (in mass fractions) was derived from the analysis of the *feed additive* [8]: Al_2O_3 (35.4 %), SiO_2 (48.9 %), K_2O (1.5 %), Fe_2O_3 (1.1 %), TiO_2 (0.4 %), MgO (0.2 %) and CaO (0.1 %).

Based on the experimental evidence available, the EURL recommends for official control the crystallographic characterisation of the *feed additive* by X-ray diffraction (XRD) (EN 13925) together with the elemental analysis by X-ray fluorescence (XRF) (EN ISO 12677).

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, as last amended by Regulation (EU) 2015/1761) is not considered necessary.

4. CONCLUSIONS AND RECOMMENDATIONS

In the frame of the current authorisation the EURL recommends for official control the crystallographic characterisation of the *feed additive* by X-ray diffraction (XRD) (EN 13925) together with the elemental analysis by X-ray fluorescence (XRF) (EN ISO 12677).

The Applicant provided no analytical method or experimental data for the determination of the *kaolinitic clays, free of asbestos (E 559)* 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 *kaolinitic clays, free of asbestos (E 559)* in *premixture*s and *feedingstuffs*.

Recommended text for the register entry (analytical method)

For the characterisation of the *feed additive*:

- X-ray diffraction (XRD) (EN 13925) and
- X-ray fluorescence (XRF) (EN ISO 12677)

5. DOCUMENTATION AND SAMPLES PROVIDED TO EURL

In accordance with the requirements of Regulation (EC) No 1831/2003, reference samples of *kaolinitic clays, free of asbestos (E 559)* 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 SANTE_E5_FWD. APPL. 1831-0045-2019 & Annex I – submission number 1288257599740-1222

[2] *Application, proposal for Register entry – Annex A

[3] *Technical dossier, Section II: Identify, characterisation and conditions of use of the additive; methods of analysis

[4] EN 13925-1,2:2003; EN 13925-3:2005 – *Non-destructive testing. X-ray diffraction from polycrystalline and amorphous materials. General principles, procedures, instruments*

[5] International Centre for Diffraction Data (ICDD) – http://www.icdd.com/

[6] *Technical dossier, Section II – Annex II_2-1-6

[7] EN ISO 12677:2011 – *Chemical analysis of refractory products by X-ray fluorescence (XRF) – fused cast-bead method*

[8] *Technical dossier, Section II – Annex II_2-2-2

*Refers to Dossier no: FAD-2010-0282

7. RAPPORTEUR LABORATORY & NATIONAL REFERENCE LABORATORIES

The Rapporteur Laboratory for this evaluation is the European Union Reference Laboratory for Feed Additives, JRC, 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 (EU) 2015/1761.

8. ACKNOWLEDGEMENTS

The following National Reference Laboratories contributed to this report:

- Państwowy Instytut Weterynaryjny, Pulawy (PL)
- Centro di referenza nazionale per la sorveglienza ed il controllo degli alimenti per gli animali (CReAA), Torino (IT)
- Ústřední kontrolní a zkušební ústav zemědělský (ÚKZÚZ), Praha (CZ)
- [1]Wageningen Food Safety Research (WFSR) (NL)
- Laboratori Agroalimentari, Departament d'Agricultura, Ramaderia, PESCA, Alimentació i Medi Natural. Generalitat de Catalunya, Cabrils (ES)

[1] Name and address according to according COMMISSION IMPLEMENTING REGULATION (EU) 2015/1761: RIKILT Wageningen UR, Wageningen.