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**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**

Natural mixture of illite-illite / smectite mixed layer
(FEED-2022-7690; CRL/220051)



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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: **FEED-2022-7690 - CRL/220051**

Name of Product / Feed Additive: ***Natural mixture of illite-illite/smectite mixed layer***

Active Agent (s): ***Illite and illite/smectite***

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

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Date: **05/09/2023**

Report approved by: **Christoph von Holst**
Date: **06/09/2023**

EXECUTIVE SUMMARY

In the current application an authorisation is sought under Article 4 for *natural mixture of illite and illite/smectite mixed layer* 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 clay composed of minimum of 35 % (w/w) of mixed layer of illite / smectite and minimum of 10 % (w/w) of illite. It also contains maximum levels of 20 % (w/w) of quartz, 15 % (w/w) of kaolinite and 5 % (w/w) of chlorite.

The *feed additive* is intended to be used in *premixtures* and *compound feed*. The Applicant proposed a minimum inclusion level of the *feed additive* of 50 g / kg *compound feed*.

For the characterisation of the *feed additive* the Applicant proposed to determine its mineralogical and elemental composition.

For the determination of the mineralogical composition of the *feed additive* the Applicant submitted a powder X-ray diffraction (XRD) method which is based on EN 13925 standard method. Furthermore, the *feed additive* was further characterised by the Applicant using X-ray fluorescence (XRF) spectrometry. This methodology is described in EN ISO 12677 standard method, which was recommended by the EURL for the characterisation of similar products in the frame of previous dossiers.

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).

The Applicant provided no analytical method or experimental data for the determination of the *natural mixture of illite and illite/smectite mixed layer* in *premixtures* and *compound feed*, 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 *natural mixture of illite and illite/smectite mixed layer* in *premixtures* and *compound feed*.

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

Natural mixture of illite-illite / smectite mixed layer, technological additives, binders, anticaking agents, all animal species.

1. BACKGROUND

In the current application an authorisation is sought under Article 4(1) (new feed additive) for *natural mixture of illite and illite/smectite mixed layer* 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 clay composed of minimum of 35 % (w/w) of mixed layer of illite / smectite and minimum of 10 % (w/w) of illite. It also contains maximum levels of 20 % (w/w) of quartz, 15 % (w/w) of kaolinite and 5 % (w/w) of chlorite [3].

The *feed additive* is intended to be used in *premixtures* and *compound feed*. The Applicant proposed a minimum inclusion level of the *feed additive* of 50 g / kg *compound feed* [4].

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 *natural mixture of illite-illite / smectite mixed layer* 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 current application. Furthermore, the Applicant provided no analytical method or experimental data for the determination of the *natural mixture of illite and illite / smectite mixed layer* in *premixtures* and *compound feed*, 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 *natural mixture of illite and illite / smectite mixed layer* in *premixtures* and *compound feed*.

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 proposed to determine its mineralogical and elemental composition [5].

For the determination of the mineralogical composition of the *feed additive* the Applicant submitted a powder X-ray diffraction (XRD) method [6] which is based on EN 13925 standard method [7]. The mineralogical compounds were identified by comparing their XRD patterns to the reference intensity ratio patterns published in the database of International Centre for Diffraction Data (ICDD) [8].

The Applicant analysed several batches of the *feed additive* and the following main mineralogical composition (in mass fractions) was derived: illite and illite / smectite mixed layer (70.6 ± 1.5 %), quartz (3.5 ± 0.9 %), calcite (7.7 ± 1.1 %), kaolinite (3.5 ± 0.9 %), feldspar (1.9 ± 0.2 %) and chlorite (0.8 ± 0.1 %) [6].

Furthermore, the *feed additive* was further characterised by the Applicant using X-ray fluorescence (XRF) spectrometry [6]. This methodology is described in EN ISO 12677 standard method [9], which was recommended by the EURL for the characterisation of similar products in the frame of several previous dossiers [10]. The following main elemental composition (in mass fractions) was derived from the analysis of several batches of the *feed additive* [6]: SiO₂ (54.6 ± 0.5 %), Al₂O₃ (16.7 ± 0.5 %), Fe₂O₃ (5.5 ± 0.2 %), CaO (6.1 ± 0.6 %), MgO (2.3 ± 0.1 %) and K₂O (2.8 ± 0.1 %) [6].

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

The Applicant provided no analytical method or experimental data for the determination of the *natural mixture of illite and illite / smectite mixed layer* in *premixtures* and *compound feed*, 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 *natural mixture of illite and illite / smectite mixed layer* in *premixtures* and *compound feed*.

Recommended text for the register entry (analytical method)

For the characterisation of the *feed additive*:

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

5. DOCUMENTATION AND SAMPLES PROVIDED TO EURL

In accordance with the requirements of Regulation (EC) No 1831/2003, reference samples of *Natural mixture of illite-illite / smectite mixed layer* 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] *Forwarding of applications for authorisation of feed additives in accordance with Regulation (EC) No 1831/2003 – E-Submission Food Chain platform – <https://webgate.ec.europa.eu/esfc/#/applications/9290>
<https://open.efsa.europa.eu/questions/EFSA-Q-2022-00840>
- [2] *Application – Annex 1
- [3] Technical dossier, Section II: 2.1.3. Qualitative and quantitative composition (active substance/agent, other components, impurities, batch to batch variation)
- [4] *Technical dossier, Section II: 2.5. Conditions of use of the additive
- [5] *Technical dossier, Section II: 2.6. Methods of analysis and reference samples
- [6] *Technical dossier, Section II – Annex _1
- [7] EN 13925-1,2:2003; EN 13925-3:2005 – Non-destructive testing. X-ray diffraction from polycrystalline and amorphous materials. General principles, procedures, instruments

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- [8] International Centre for Diffraction Data (ICDD) – <http://www.icdd.com/>
- [9] EN ISO 12677:2011 – Chemical analysis of refractory products by X-ray fluorescence (XRF) – fused cast-bead method
- [10] EURL reports:
https://joint-research-centre.ec.europa.eu/publications/fad-2010-0282_en
https://joint-research-centre.ec.europa.eu/publications/fad-2010-0284_en
https://joint-research-centre.ec.europa.eu/publications/fad-2019-0011_en

*Refers to Dossier no: FEED-2022-7690

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

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- Instytut Zootechniki — Państwowy Instytut Badawczy, Krajowe Laboratorium Pasz, Lublin (PL)
- Laboratoire de Rennes (SCL L35), Service Commun des Laboratoires DGCCRF et DGDDI, Rennes (FR)

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