EUROPEAN COMMISSION
JOINT RESEARCH CENTRE

Institute for Reference Materials and Measurements (Geel)
Standards for Food Bioscience

JRC.D.5/SFB/CvH/JK/msd/Ares

European Union Reference Laboratory

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, montmorillonite and kaolinite

(FAD-2012-0025; CRL/120016)

European Union Reference Laboratory
Feed Additives

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

Dossier related to:	**FAD-2012-0025 - CRL/120016**
Name of Product	**Natural mixture of illite, montmorillonite and kaolinite**
Active Substance(s):	**-**
Rapporteur Laboratory:	**European Reference Laboratory for Feed Additives (EURL-FA)**
Report prepared by:	**Johanna Keltti**
Report revised by: Date:	**Piotr Robouch (EURL-FA)** **12/09/2013**
Report approved by: Date:	**Christoph von Holst** **12/09/2013**

EXECUTIVE SUMMARY

In the current application authorisation is sought under article 4(1) for Velay green clay, a *natural mixture of illite, montmorillonite* and *kaolinite* (MIMK), 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. The authorisation is sought for the use of the *feed additive* for all animal species. According to the Applicant, the *feed additive* contains a minimum of 40 % *illite*. The *feed additive* is intended to be used in *premixtures* and *feedingstuffs*. The Applicant did not specify any maximum or minimum concentration of MIMK in *feedingstuffs* but recommends a dosage of 2-5 % for all animal species.

For the determination of mineralogical composition of the *feed additive* the Applicant submitted experimental data obtained using X-ray diffraction (XRD) method. Furthermore, the chemical composition of the *feed additive* was characterised by the Applicant using X-Ray fluorescence (XRF). Based on the experimental evidence provided, the EURL recommends for official control the two methods (XRD and XRF) for the characterisation of *Velay green clay.*

As the quantification of *MIMK* in *premixtures* and *feedingstuffs* is not achievable experimentally, the EURL cannot recommend any method for official control in these matrices. However, the addition of *Velay green clay* in *feedingstuffs* could be monitored indirectly by determining the increased iron and aluminium content, if identical *feedingstuffs* without the product is available for comparison.

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

Argile verte du velay, Velay green clay, illite, montmorillonite, kaolinite, technological additives, binders, anticaking agents, all animal species.

1. BACKGROUND

In the current application authorisation is sought under article 4(1) for Velay green clay, a *natural mixture of illite, montmorillonite,* and *kaolinite* (later referred as *MIMK*), 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]. The authorisation is sought for the use of the *feed additive* for all animal species [2].

Velay green clay is natural clay obtained by crushing and milling natural rocks. The *feed additive* consists mainly of *illite, montmorillonite, kaolinite* and other minerals such as calcite and sanidine (feldspar). According to the Applicant, the *feed additive* contains a minimum of 40 % *illite* [3].

The *feed additive* is intended to be used in *premixtures* and *feedingstuffs*. The Applicant did not specify any maximum or minimum concentration of *MIMK* in *feedingstuffs* but recommends a dosage of 2-5 % for all animal species [3].

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 *Velay green clay,* and their suitability to be used for official controls in the frame of the authorisation, were evaluated.

3. EVALUATION

Qualitative and quantitative composition of impurities in the feed additive

When required by EU legislation, analytical methods for official control of undesirable substances in the additive such as heavy metals (arsenic, cadmium, lead and mercury), dioxins, microbiological agents and mycotoxins are available from the respective European Union Reference Laboratories [4].

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

For the determination of mineralogical composition of the *feed additive* the Applicant submitted experimental data obtained using X-ray diffraction (XRD) method [5]. The mineralogical compounds are identified comparing the XRD patterns to the Reference Intensity Ratio values published in the International Centre Diffraction Data reference cards. The following results are derived from the analyses of 10 samples:

illite:	53.5 ± 5.7%
montmorillonite:	16.2 ± 4.8%
kaolinite:	16.5 ± 3.5%
calcite:	7.8 ± 3.9%
sanidine (feldspar):	6.2 ± 3.0%

Furthermore, the *feed additive* was characterised by the Applicant using X-Ray fluorescence (XRF) and the following chemical composition was derived from the analysis of elements in 6 samples [6]:

SiO_2	41.0 ± 3.0 %
Al_2O_3	24.4 ± 1.9 %
Fe_2O_3	14.9 ± 2.9 %
CaO	8.5 ± 3.0 %
K_2O	7.9 ± 1.0 %
MgO	3.2 ± 0.4 %

Based on the experimental evidence provided, the EURL recommends for official control the crystallographic characterisation by X-ray diffraction (XRD) together with the chemical analysis by X-Ray fluorescence (XRF) for the characterisation of *Velay green clay.*

The Applicant provided no experimental data or any analytical method for the determination of the *MIMK* in *premixtures* or *feedingstuffs* as the unambiguous determination of *Velay green clay* content added to these matrices is not achievable experimentally. Therefore, the EURL cannot evaluate nor recommend any method for official control for the direct determination of *MIMK* in *premixtures* or *feedingstuffs*.

However, during the review process one NRL noticed that including 2% of *Velay green clay* in *feedingstuffs* would result in high levels of iron and aluminium (ca. 2 g/kg). According to the NRL, such levels could be monitored applying (1) the Community method based on Atomic Absorption Spectrometry (AAS) and (2) Inductively Coupled Plasma Atomic Emission Spectroscopy (ICP-AES), for the determination of iron and aluminium in *feedingstuffs*, respectively.

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 X-ray diffraction (XRD) together with the X-Ray fluorescence (XRF) for the characterisation of *Velay green clay* (*feed additive)*.

As the quantification of *MIMK* in *premixtures* and *feedingstuffs* is not achievable experimentally, the EURL cannot recommend any method for official control in these matrices. However, the addition of *Velay green clay* in *feedingstuffs* could be monitored indirectly by determining the increased iron and aluminium content, if identical *feedingstuffs* without the product is available for comparison.

Recommended text for the register entry (analytical method)

Characterisation of *Velay green clay* (*feed additive)*:

- X-ray diffraction (XRD) together with
- X-Ray fluorescence (XRF)

5. DOCUMENTATION AND SAMPLES PROVIDED TO EURL

In accordance with the requirements of Regulation (EC) No 1831/2003, reference sample of *Velay green clay* 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] *Reference SANCO/D/2 Forw. Appl. 1831/0001-2013

[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] *Technical dossier, Section II – Annex II 2-1-5

[6] *Technical dossier, Section II – Annex II 2-1-6

* Refers to Dossier No: FAD-2012-0035

7. RAPPORTEUR LABORATORY

The Rapporteur Laboratory for this evaluation was the 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:

- Fødevarestyrelsen, Ringsted (DK)
- Schwerpunktlabor Futtermittel des Bayerischen Landesamtes für Gesundheit und Lebensmittelsicherheit (LGL), Oberschleißheim (DE)
- Ústřední kontrolní a zkušební ústav zemědělský (ÚKZÚZ), Praha (CZ)
- Państwowy Instytut Weterynaryjny, Puławy (PL)
- RIKILT - Instituut voor Voedselveiligheid, Wageningen (NL)
- Centro di referenza nazionale per la sorveglianza ed il controllo degli alimenti per gli animali (CReAA), Torino (IT)
- Laboratoire de Rennes, SCL L35, Service Commun des Laboratoires, Rennes (FR)
- Thüringer Landesanstalt für Landwirtschaft (TLL), Abteilung Untersuchungswesen, Jena (DE)
- Staatliche Betriebsgesellschaft für Umwelt und Landwirtschaft, Labore Landwirtschaft, Leipzig (DE)