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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

Aluminosilicate of sodium, potassium, calcium and magnesium

(FAD-2017-0022; CRL/160049)

European Union Reference Laboratory
Feed Additives

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

Dossier related to:	**FAD-2017-0022 - CRL/160049**
Name of Product:	***Aluminosilicate of sodium, potassium, calcium and magnesium***
Active Agent (s):	**Aluminosilicate of sodium, potassium, calcium and magnesium**
Rapporteur Laboratory:	**European Union Reference Laboratory for Feed Additives (EURL-FA)** **JRC Geel, Belgium**
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Report checked by: Date:	**Piotr Robouch (EURL-FA)** **16/01/2018**
Report approved by: Date:	**Christoph von Holst** **06/02/2018**

EXECUTIVE SUMMARY

In the current application authorisation is sought under Article 4(1) for *aluminosilicate of sodium, potassium, calcium and magnesium* under the category/functional group (1i) "technological additives"/"anticaking agents", according to the classification system of Annex I of Regulation (EC) No 1831/2003. Specifically, authorisation is sought for the use of the *feed additive* for pigs.

The *feed additive* is a powder derived from crushed and milled natural aluminosilicate rocks, containing a minimum of 65.6 % (w/w) *chabazite* and other minerals such as phillipsite, K-feldspar, biotite, piroxene and volcanic glass.

The *feed additive* is intended to be used directly in *feedingstuffs* or through *premixtures* to ensure flowability within storage silos. The Applicant proposed a minimum inclusion level of the *feed additive* of 30 g/kg complete *feedingstuffs*.

For the determination of the mineralogical and chemical composition of the *feed additive* the Applicant applied (i) X-ray diffraction (XRD) and (ii) X-ray fluorescence spectrometry (XRF) based on the EN 13925 and ISO 29581-2 standard methods, respectively. Based on the experimental evidence available, the EURL recommends these two standard methods for official control for the characterisation of the *feed additive*.

As the accurate determination of the *aluminosilicate of sodium, potassium, calcium and magnesium* content added to *premixtures* and *feedingstuffs* is not achievable experimentally, the EURL cannot evaluate nor recommend any method for official control.

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

Aluminosilicate of sodium, potassium, calcium and magnesium, technological additives, anticaking agents, pigs

1. BACKGROUND

In the current application authorisation is sought under Article 4(1) (new *feed additive*) for *aluminosilicate of sodium, potassium, calcium and magnesium* under the category/functional group (1i) "technological additives"/"anticaking agents", according to the classification system of Annex I of Regulation (EC) No 1831/2003. Specifically, authorisation is sought for the use of the *feed additive* for pigs [1].

The *feed additive* is a powder derived from crushed and milled natural aluminosilicate rocks, containing a minimum of 65.6 % (w/w) *chabazite* [2, 3] and other minerals such as phillipsite, K-feldspar, biotite, piroxene and volcanic glass [3].

The *feed additive* is intended to be used directly in *feedingstuffs* or through *premixtures* to ensure flowability within storage silos [3]. The Applicant proposed a minimum inclusion level of the *feed additive* of 30 g/kg complete *feedingstuffs* [2].

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 *aluminosilicate of sodium, potassium, calcium and magnesium* 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, aflatoxin B1 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 the feed additive, premixtures and feedingstuffs

For the determination of the mineralogical composition of the *feed additive* the Applicant applied X-ray diffraction (XRD), where the identification of the individual minerals is based on the reference patterns from International Centre for Diffraction Data while the quantification of each mineral phase is performed using combined Rietveld and Reference Intensity Ratio (RIR) methods [5]. The XRD method based on the EN 13925 standard method [6] was previously recommended by the EURL in the frame of several aluminosilicate dossiers [7-9]. The following mineralogical composition was derived from the analyses of seven *feed additive* samples [3]:

chabazite:	68.1 ± 1.7 % (w/w)
volcanic glass:	12.6 ± 1.9 % (w/w)
K-feldspar:	6.3 ± 1.1 % (w/w)
biotite:	5.4 ± 2.2 % (w/w)
piroxene:	3.7 ± 0.8 % (w/w)
phillipsite:	3.6 ± 1.0 % (w/w)

The *feed additive* was further characterised using X-ray fluorescence spectrometry (XRF) [10]. This method based on the ISO 29581-2 standard method [11] was previously recommended by the EURL in the frame of a previous dossier [8]. The following main chemical composition was reported [3]:

SiO_2	55 ± 1.4 % (w/w)
Al_2O_3	15.0 ± 0.8 % (w/w)
K_2O	7.4 ± 0.5% (w/w)
CaO	6.2 ± 0.8 % (w/w)
MgO	1.7 ± 0.3 % (w/w)
Na_2O	0.5 ± 0.1 % (w/w)

Based on the experimental evidence available, the EURL recommends for official control the mineralogical characterisation by X-ray diffraction (XRD) and the element analysis by X-ray fluorescence spectrometry (XRF) described in the EN 13925 and the ISO 29581-2 standards for the characterisation of the *feed additive*, respectively.

Even though the Applicant states that these methods are suitable for the determination of *aluminosilicate of sodium, potassium, calcium and magnesium* in *premixtures* and *feedingstuffs* [3], the EURL considers that the accurate determination of the *aluminosilicate of sodium, potassium, calcium and magnesium* content added to these matrices is not achievable experimentally. Therefore, the EURL cannot evaluate nor recommend any method for official control.

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 X-ray diffraction (XRD) and X-ray fluorescence spectrometry (XRF) described in the EN 13925 and ISO 29581-2 standards for the characterisation of the *feed additive*.

As the accurate determination of the *aluminosilicate of sodium, potassium, calcium and magnesium* content added to *premixtures* and *feedingstuffs* is not achievable experimentally, the EURL cannot evaluate nor recommend any method for official control.

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 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 *aluminosilicate of sodium, potassium, calcium and magnesium* 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. 1831-0025-2017

[2] *Application, Proposal for Register Entry – Annex A

[3] *Technical dossier, Section II: Identity, characterisation and conditions of use of the feed 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 – Annex II_8 (A. F. Gualtieri, *J. Appl. Cryst.* (2000). 33, 267-278)

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

[7] #FAD-2010-0061 – JRC.D.5/SFB/CvH/JK /mds/Ares(2015)1318765

[8] #FAD-2010-0238 – JRC.D.5/SFB/CvH/ZE/Ares(2015)3651278

[9] #FAD-2016-0005 – JRC.D.5/CvH/ZE/mds/Ares(2016)2428070

[10] *Technical dossier – Annex II_8 (M. Franzini *et al* (1975). Revisione di una metodologia analitica per fluorescenza – X, basata sulia correzione complete degli effetti di matrice. Soc. Italiana di Mineralogia e petrologia – Vol XXXI (2), p.365-378)

[11] ISO 29581-2:2010 – Cement - Test methods - Part 2: Chemical analysis by X-ray fluorescence

*Refers to Dossier no: FAD-2017-0022

#https://ec.europa.eu/jrc/en/eurl/feed-additives/evaluation-reports

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)
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- Ústřední kontrolní a zkušební ústav zemědělský (ÚKZÚZ), Praha (CZ)
- RIKILT Wageningen UR, Wageningen (NL)
- Laboratoire de Rennes (SCL L35), Service Commun des Laboratoires DGCCRF et DGDDI, Rennes (FR)