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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**

Phyllite

(FAD-2016-0005; CRL/150031)



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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: **FAD-2016-0005 - CRL/150031**

Name of product: **Phyllite**

Active Agent (s): **-**

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

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Date: **23/05/2016**

Report approved by: **Christoph von Holst**
Date: **23/05/2016**

EXECUTIVE SUMMARY

In the current application authorisation is sought under article 4(1) for *phyllite*, under the category/functional group 1(i) 'technological additives/'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* is a finely ground powder derived from mountain rocks. The *feed additive* consists of a natural mixture of minerals of metamorphic origin containing a minimum of 40 % of phyllo- and alumo-/alumohydrosilicates such as muscovite, illite, chlorite and talc. The *feed additive* is intended to be used directly in *feedingstuffs* or through *premixtures* to ensure flowability within the storage silos. The Applicant proposed a maximum inclusion level of the *feed additive* of 25 g/kg complete *feedingstuffs*.

For the characterisation of the *phyllite* the Applicant submitted the EN 13925 standard method based on X-Ray diffraction (XRD) to determine its mineralogical composition. Furthermore, the Applicant applied X-Ray fluorescence spectrometry (XRF) as described in the EN ISO 12677 standard to determine the elemental composition in the *feed additive*.

Based on the experimental data provided, the EURL recommends for official control the XRD and XRF methods described in the EN 13925 and EN ISO 12677 standards for the characterisation of the *feed additive*.

The Applicant provided no experimental data or any analytical method for the quantification of *phyllite* 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 *phyllite* 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.

KEYWORDS

Phyllite, technological additives, anticaking agents, all animal species

1. BACKGROUND

In the current application authorisation is sought under article 4(1) (new feed additive) for *phyllite*, under the category/functional group 1(i) 'technological additives'/'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 [1,2].

According to the Applicant the *feed additive* is a silky, light green-grey finely ground powder derived from mountain rocks [3]. The *feed additive* consists of a natural mixture of minerals of metamorphic origin containing a minimum of 40 % of phyllo- and alumo-/alumohydro-silicates such as muscovite, illite, chlorite and talc [2]. *Phyllite* also contains quartz, sodium and potassium feldspar [3].

The *feed additive* is intended to be used directly in *feedingstuffs* or through premixtures to ensure flowability within the storage silos. The Applicant proposed a maximum inclusion level of the *feed additive* of 25 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 *phyllite* 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, mycotoxins 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 feed additive

For the characterisation of *phyllite* the Applicant submitted the EN 13925 standard method based on X-Ray diffraction (XRD) to determine its mineralogical composition [5]. The following composition was reported for five typical *phyllite* samples [3]:

Mica/muscovite and illite	30 – 36.5 %
Chlorite and talc	10 – 16 %
Quartz	30 – 33 %
Potassium feldspar	2 – 4 %
Sodium feldspar	7 – 11.5 %
Calcite	1 – 2 %

Furthermore, the Applicant applied X-Ray fluorescence spectrometry (XRF) as described in the EN ISO 12677 standard [6] and reported the following elemental composition of the *feed additive* [3]:

Composition	Concentration range
SiO ₂	54 – 61 %
Al ₂ O ₃	18 – 21 %
Fe ₂ O ₃	7.5 – 9.5 %
MgO	1.5 – 3.0 %
K ₂ O	4.0 – 5.0 %
Na ₂ O	0.4 – 1.4 %
CaO	0.7 – 2.2 %
TiO ₂	0.95 – 1.3 %
P ₂ O ₅	0.08 – 0.12 %
MnO	0.04 – 0.17 %

Based on the experimental data provided, the EURL recommends for official control the XRD and XRF methods described by the EN 13925 and the EN ISO 12677 standards for the characterisation of the *feed additive*.

In addition, the Applicant applied the method for determination of loss on ignition in the *feed additive* as described in the DIN 51081 standard [7] and the values from 3.5 to 4.5 % were reported [3].

The Applicant provided no experimental data or any analytical method for the quantification of the *phyllite* 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 *phyllite* 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.

4. CONCLUSIONS AND RECOMMENDATIONS

The EURL recommends for official control two internationally recognised standards: - EN 13925, based on X-ray diffraction (XRD); and – EN ISO 12677, based on X-ray fluorescence spectrometry (XRF) for characterisation of *phyllite*.

The unambiguous determination of the *feed additive* added to *premixtures* and *feedingstuffs* is not achievable experimentally. Therefore, the EURL cannot evaluate nor recommend any method for official control for the determination of *phyllite* in *premixtures* and *feedingstuffs*.

Recommended text for the register entry (analytical method)

Characterisation of the *feed additive*:

- X-ray diffraction (XRD) – EN 13925; and
- X-ray fluorescence spectrometry (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 *phyllite* 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 SANCO/G1: Forw. Appl. 1831/0007-2016
- [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

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- [5] EN 13925-1,2:2003; EN 13925-3:2005 – Non-destructive testing. X-ray diffraction from polycrystalline and amorphous materials. General principles, procedures, instruments
- [6] EN ISO 12677:2011 – Chemical analysis of refractory products by X-ray fluorescence (XRF) – fused cast-bead method
- [7] DIN 51081:1996-07 – Testing of oxidic raw materials and material – Determination of change in mass on ignition

*Refers to Dossier no: FAD-2016-0005

7. RAPPORTEUR LABORATORY & NATIONAL REFERENCE LABORATORIES

The Rapporteur Laboratory for this evaluation was 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, as last amended by Regulation (EU) 2015/1761.

8. ACKNOWLEDGEMENTS

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- RIKILT Wageningen UR, Wageningen (NL)
- Instytut Zootechniki — Państwowy Instytut Badawczy, Krajowe Laboratorium Pasz, Lublin (PL)