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Updated 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

Kieselgur (diatomaceous earth, purified) (E 551c)

(FAD-2010-0284; CRL/100189)

European Union Reference Laboratory
Feed Additives

Updated 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-2010-0284 - CRL/100189**
Name of Feed Additive:	***Kieselgur (diatomaceous earth, purified) (E 551c)***
Active Agent (s):	**-**
Rapporteur Laboratory:	**European Union Reference Laboratory for Feed Additives (EURL-FA)** **JRC Geel, Belgium**
Report prepared by:	**Stefano Bellorini**
Report checked by: Date:	**Zigmas Ezerskis** **05/03/2020**
Report approved by: Date:	**Christoph von Holst** **05/03/2020**

EXECUTIVE SUMMARY

In the current application authorisation is sought under Article 10(2) for *kieselgur (diatomaceous earth, purified)* (E551c) as a feed additive under the category "technological feed additives", functional group 1(g) “binders” and 1(i) “anti-caking agents” according to the classification system of Annex I of Regulation (EC) No 1831/2003. The authorisation is sought to use the *feed additive* for all animal species and categories.

Kieselgur is available in natural (*diatomaceous earth*) or purified (*calcinated or flux calcinated diatomaceous earth*) forms. The *feed additive* is composed of several mineral constituents that jointly contribute to its activity. As purity criteria for the *feed additive*, the Applicant proposes the following minimum content of SiO_2: 50, 70 and 81 % (w/w), respectively, for *diatomaceous earth (natural), calcinated diatomaceous earth (purified)* and for *flux calcinated diatomaceous earth (purified).* The *feed additive* is intended to be included directly into *feedingstuffs* or through *premixtures* with no recommended minimum or maximum inclusion level.

Kieselgur does not contain any specific *active substance* to be determined. Furthermore, an unambiguous determination of *kieselgur (diatomaceous earth, purified)* 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 any specific *active substance* in *kieselgur (diatomaceous earth, purified)* and of the *feed additive* added to *premixture*s and *feedingstuffs*.

For the mineralogical characterisation of the *feed additive* the Applicant proposed to apply an X-Ray Diffraction (XRD) method equivalent to standard method EN 13925. The EURL considers the EN 13925 method based on XRD suitable for the mineralogical characterisation of the *feed additive.*

For the elemental characterisation of the *feed additive* the Applicant proposed to apply an X-Ray Fluorescence (XRF) method equivalent to the standard EN ISO 12677. The EURL recommends for official control the method EN ISO 12677 based on XRF spectrometry for the elemental characterisation of the *feed additive*.

Additionally, the EURL is aware of FAO JECFA and Food Chemical Codex (FCC) specific monographs for *diatomaceous earth* where additional tests (i.e. loss on drying, loss on ignition, non-siliceous substances quantification etc.) are described. The EURL considers these tests suitable for the additional characterisation of the *feed additive.*

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

Kieselgur, diatomaceous earth, purified, technological feed additives, binders, anti-caking agents, all animal species and categories

1. BACKGROUND

In the current application authorisation is sought under Article 10(2) (authorisation of an existing product) for *kieselgur (diatomaceous earth, purified)* (E551c) as a feed additive under the category "technological feed additives", functional group 1(g) "binders" and 1(i) "anti-caking agents" according to the classification system of Annex I of Regulation (EC) No 1831/2003 [1,2]. The authorisation is sought to use the *feed additive* for all animal species and categories [2].

According to the Applicant, the *feed additive* is a "*fine-grained siliceous sediment of biogenic origin*" [3]. *Kieselgur* is available in natural (*diatomaceous earth*) or purified (*calcinated or flux calcinated diatomaceous earth*) form [4]. The *feed additive* is composed of several mineral constituents that jointly contribute to its activity [3]. Silicon dioxide (SiO_2) is the main component of the *feed additive* [3]. As purity criteria for the *feed additive*, the Applicant proposes the following minimum content of SiO_2 in the three forms of *kieselgur* [2,5]:

- 50 % (w/w) for *diatomaceous earth (natural)*
- 70 % (w/w) for *calcinated diatomaceous earth (purified)*; and
- 81 % (w/w) for *flux calcinated diatomaceous earth (purified).*

The *feed additive* is intended to be included directly into *feedingstuffs* or through *premixtures* with no recommended minimum or maximum inclusion level [2,6].

Note: The EURL previously evaluated and recommended analytical methods for the characterisation of *diatomaceous earth* in the frame of dossier FAD-2019-0011 [7].

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 *kieselgur (diatomaceous earth, purified)* 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)

Kieselgur does not contain any specific *active substance* to be determined [3]. Furthermore, an unambiguous determination of *kieselgur (diatomaceous earth, purified)* 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 any specific *active substance* in *kieselgur (diatomaceous earth, purified)* and of the *feed additive* added to *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 mineralogical characterisation of the *feed additive* the Applicant proposed to apply [8]:

(i) an X-Ray Diffraction (XRD) method equivalent to standard method EN 13925 for the determination of the crystalline structure [9,10]; and

(ii) an analysis by optical microscopy or a Scanning Electron Microscopy (SEM) for the determination of the particle morphology [11].

Furthermore, the Applicant provided the EURL with the corresponding standard operating procedures (SOPs), the typical mineralogical composition of the three forms of the *feed additive* and a limited number of experimental data [9,11].

The optical microscopy and the SEM methods presented by the Applicant allow the visual identification of structured fragments. However, this type of analysis requires geochemical laboratories equipped with SEM and the availability of an experienced technician (i.e. geologist) which is not common in feed control laboratories [11]. Therefore, the EURL considers solely the EN 13925 method based on XRD suitable for the mineralogical characterisation of the *feed additive*.

For the elemental characterisation of the *feed additive* the Applicant proposed to apply [8]:

(iii) X-Ray Fluorescence (XRF) based on fused bead [12]; and/or

(iv) Atomic Absorption Spectrometry (AAS).

The Applicant did not present any SOP and/or any corresponding experimental data supporting the AAS technique proposed. On the other hand, the Applicant provided the EURL with the corresponding SOP for the submitted XRF method and supporting experimental data [12]. The method is based on the withdrawn British Standard 1902, replaced by the current standard method EN ISO 12677 already recommended by the EURL in a previous evaluation for *diatomaceous earth* [7,13,14].

When applying the SOP of EN ISO 12677, the powdered sample is fused with a suitable flux to destroy its mineralogical and particulate composition. The resultant melt is cast into the shape of a glass bead which is then introduced into an XRF spectrometer. The intensities of the fluorescent X-rays of the elements in the bead are measured and the chemical composition of the sample is determined by using calibration graphs or equations applying corrections for inter-elemental effects [14].

The Applicant analysed multiple batches of the three forms of the *feed additive* [15]. The experimental values obtained for the SiO_2 content are presented in Table 1.

The EURL recommends for official control the method EN ISO 12677 based on XRF spectrometry for the elemental characterisation of the *feed additive*.

Additionally, the EURL is aware of FAO JECFA and Food Chemical Codex (FCC) specific monographs for *diatomaceous earth* where additional tests (i.e. loss on drying, loss on ignition, non-siliceous substances quantification etc.) are described. The EURL considers these tests suitable for the additional characterisation of the *feed additive* [16,17].

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.

Table 1: SiO_2 content from multiple representative batches of *kieselgur (diatomaceous earth, purified)* obtained by XRF spectrometry. The results are expressed as average mass fraction ± standard deviation ($\bar{w} \pm s$). In addition the relative standard deviation is listed (RSD) [15].

kieselgur (E551c)	# batches	$\bar{w} \pm s$ (%)	RSD (%)
diatomaceous earth (natural)	2	70.5 ± 1.6	2.3
	3	59.6 ± 6.5	10.9
	7	82.5 ± 6.7	8.1
calcinated diatomaceous earth (purified)	3	76.9 ± 2.8	3.7
	10	88.7 ± 1.6	1.8
flux calcinated diatomaceous earth (purified)	20	86.2 ± 3.3	3.8

4. CONCLUSIONS AND RECOMMENDATIONS

In the frame of the current authorisation, the EURL recommends for official control the method EN ISO 12677 based on X-Ray Fluorescence (XRF) spectrometry for the elemental characterisation of the *feed additive*.

As an unambiguous determination of *kieselgur (diatomaceous earth, purified)* or the *feed additive* added to *premixtures* and *feedingstuffs* is not achievable experimentally, the EURL cannot evaluate nor recommend any method for official control for the determination of *kieselgur (diatomaceous earth, purified)* in *premixture*s and *feedingstuffs*.

Recommended text for the register entry (analytical method)

For the characterisation of the *feed additive*:

- 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 *kieselgur (diatomaceous earth, purified)* 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. 1831-0067-2019; FAD 2010-0284

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

[3] *Technical dossier, Section II: 2.1.3 Qualitative and quantitative composition

[4] *Technical dossier, Section II: 2.1.1 Name of the additive

[5] *Technical dossier, Section II: 2.1.4.3 Proposal for a specification of the additive

[6] *Technical dossier, Section II: 2.5 Conditions of use of the additive

[7] EURL Evaluation Reports: https://ec.europa.eu/jrc/sites/jrcsh/files/finrep-fad-2019-0011-sepiolitediatomaceous-earth.pdf

[8] *Technical dossier, Section II: 2.6 Methods of analysis and reference samples

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

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

[11] *Technical dossier, Section II: Annex II 2-6-2

[12] *Technical dossier, Section II: Annex II 2-6-3

[13] BS 1902-9.1:1987 - Methods of testing refractory materials. Chemical analysis by instrumental methods. Analysis of alumino-silicate refractories by X-ray fluorescence https://shop.bsigroup.com/ProductDetail/?pid=000000000000167091

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

[15] *Technical dossier, Section II: Annex II 2.1.3.4 Batch to batch variation

[16] FAO JECFA combined Compendium of Food Additive Specifications, "Diatomaceous Earth" Monograph 1 (2006) http://www.fao.org/food/food-safety-quality/scientific-advice/jecfa/jecfa-additives/detail/en/c/397/

[17] Food Chemical Codex monograph "Diatomaceous Earth", FCC 7 (2010), p.288

*Refers to Dossier no: FAD-2010-0284

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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- Państwowy Instytut Weterynaryjny, Pulawy (PL)
- Centro di referenza nazionale per la sorveglienza ed il controllo degli alimenti per gli animali (CReAA), Torino (IT)
- Laboratoire de Rennes (SCL L35), Service Commun des Laboratoires DGCCRF et DGDDI, Rennes (FR)
- Ústřední kontrolní a zkušební ústav zemědělský (ÚKZÚZ), Praha (CZ)
- Laboratori Agroalimentari, Departament d'Agricultura, Ramaderia, PESCA, Alimentació i Medi Natural. Generalitat de Catalunya, Cabrils (ES)
- [1] Wageningen Food Safety Research (WFSR) (NL)

[1] Name and address according to according Commission Implementing Regulation (EU) 2015/1761: RIKILT Wageningen UR, Wageningen.