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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**

**Precipitated and dried silicic acid
Colloidal silica
(FAD-2010-0226; CRL/100247)**



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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-2010-0226 - CRL/100247**

Name of Feed Additives: **Precipitated and dried silicic acid**
Colloidal silica

Active Agent (s):

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

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Date: **18/08/2015**

Report approved by: **Christoph von Holst**
Date: **18/08/2015**

EXECUTIVE SUMMARY

In the current application authorisation is sought under article 10(2) for *precipitated and dried silicic acid* (E 551a) and *colloidal silica* (E 551b) under the category / functional group 1(i) 'technological additives' / 'anticaking agents', according to Annex I of Regulation (EC) No 1831/2003. Specifically, the authorisation is sought for the use of the *feed additives* for all animal species.

The *feed additives* are synthetic amorphous high purity silicon dioxides produced as precipitated silica or silica gel, or as fumed pyrogenic silica. All these products appear as a fluffy white powder containing a minimum of 94 % SiO₂. The Applicant states that the purity criteria/specifications set in Commission Regulation (EU) 231/2012 for the food additive (silicon dioxide) apply also to the *feed additives*.

The *feed additives* are intended to be included in *premixtures* or added into *feedingstuffs* with recommended maximum inclusion level of 50 g/kg complete *feedingstuffs*.

For the characterisation of the *feed additives*, the Applicant referred to the Commission Regulation (EU) 231/2012 and submitted the FAO JECFA '*Silicon dioxide, amorphous*' monograph of compendium for food additives, and the dedicated Food Chemicals Codex (FCC) monograph '*Silicon dioxide*'. According to the Commission Regulation (EU) 231/2012 the following tests/methods have to be applied for the characterisation of *silicon dioxide*: - test for silica; - loss on drying; - loss on ignition; - assay of silicon dioxide content; and - assay of soluble ionisable salts. All these tests are also described in the FAO JECFA and FCC monographs mentioned above. Even though no performance characteristics are provided, the EURL recommends for official control the methods required by Commission Regulation (EU) 231/2012 and described in the FAO JECFA and Food Chemicals Codex (FCC) monographs for the characterisation of the *feed additives*.

As stated by the Applicant, the quantification of synthetic amorphous silicon dioxide added to *premixtures* or *feedingstuffs* is not achievable experimentally. Therefore, the EURL cannot recommend any methods for official control for the quantification of the *feed additives* in *premixtures* or *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

Precipitated and dried silicic acid, colloidal silica, silicon dioxide, technological additives, anticaking agents, all animal species

1. BACKGROUND

In the current application authorisation is sought under article 10(2) (authorisation of an existing product) for *precipitated and dried silicic acid* and *colloidal silica* under the category / functional group 1(i) 'technological additives' / 'anticaking agents', according to Annex I of Regulation (EC) No 1831/2003 [1]. Specifically, the authorisation is sought for the use of the *feed additives* for all animal species [1,2].

The *feed additives* are synthetic amorphous high purity silicon dioxides produced as precipitated silica or silica gel, or as fumed pyrogenic silica. All these products appear as a fluffy white powder containing a minimum of 94 % SiO₂. [2,3]. The Applicant states that the purity criteria/specifications set in Commission Regulation (EU) 231/2012 for the food additive (silicon dioxide) apply also to the *feed additives* [3].

The *feed additives* are intended to be included directly into *feedingstuffs* or through *premixtures* with recommended maximum inclusion level of 50 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 (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 *precipitated and dried silicic acid* and *colloidal silica* 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, 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, premixtures and feedingstuffs

For the characterisation of the *feed additives*, the Applicant referred to Commission Regulation (EU) 231/2012 and submitted the FAO JECFA '*Silicon dioxide, amorphous*' monograph of compendium for food additives [5], and the dedicated Food Chemicals Codex (FCC) monograph '*Silicon dioxide*' [6].

According to the Commission Regulation (EU) 231/2012 the following tests/methods have to be applied for characterisation of *silicon dioxide*: - test for silica; - loss on drying; - loss on ignition; - assay of silicon dioxide content; and - assay of soluble ionisable salts. All these tests are described in the FAO JECFA and FCC monographs mentioned above [5,6].

The amount of volatile silicon tetrafluoride (SiF_4) is determined in the "test for silica" [5].

Performed at 105 °C for 2 h, the "loss on drying" is determined from the difference in weights of the initial and the dried samples [5,6].

For "loss on ignition" the dried sample (1 to 2 g) is further heated at 900-1000 °C for 1 h, cooled in a desiccator and weighed to obtain the ignited sample weight [5,6].

For the quantification of SiO_2 , the ignited sample obtained after the "loss on ignition" test is further treated with alcohol, sulphuric and hydrofluoric acids, evaporated to dryness, ignited over Meker burner to red heat, cooled in a desiccator and weighed to obtain the residual weight. The difference in weight between the ignited and the residual sample represents the amount of SiO_2 in grams in the ignited sample [5,6].

For the quantification of the soluble ionisable salts, 5 g of the dried sample is stirred in 150 ml of water for 5 min and filtered. The filtrate is further diluted before the conductivity measurement with a suitable conductance bridge assembly. The quantity of soluble ionisable salts is expressed as percentage of sodium sulphate content [6].

Even though no performance characteristics are provided, the EURL recommends for official control the methods required by Commission Regulation (EU) 231/2012 and described in the FAO JECFA and Food Chemicals Codex (FCC) monographs mentioned above for the characterisation of the *feed additives (precipitated and dried silicic acid and colloidal silica)*.

As stated by the Applicant, the quantification of synthetic amorphous silicon dioxide added to *premixtures* or *feedingstuffs* is not achievable experimentally. Therefore, the EURL cannot recommend any methods for official control for the quantification of the *feed additives* in *premixtures* or *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

In the frame of this authorisation the EURL recommends for official control of the *precipitated and dried silicic acid and colloidal silica (feed additives)* the methods required by Commission Regulation (EU) 231/2012 and described in FAO JECFA '*Silicon dioxide, amorphous*' monograph and Food Chemicals Codex (FCC) monograph for '*Silicon dioxide*'.

As stated by the Applicant, the quantification of synthetic amorphous silicon dioxide added to *premixtures* or *feedingstuffs* is not achievable experimentally. Therefore, the EURL cannot recommend any methods for official control for the quantification of the *feed additives* in *premixtures* or *feedingstuffs*.

Recommended text for the register entry (analytical method)

For the characterisation of *precipitated and dried silicic acid* or *colloidal silica (feed additives)*:

- Commission Regulation (EU) No 231/2012 and the methods described in the FAO JECFA '*Silicon dioxide, amorphous*' and Food Chemicals Codex (FCC) '*Silicon dioxide*' monographs

5. DOCUMENTATION AND SAMPLES PROVIDED TO EURL

In accordance with the requirements of Regulation (EC) No 1831/2003, reference samples of "*precipitated and dried silicic acid and colloidal silica*" 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. 1831/0024-2014
- [2] *Application, Proposal for Register Entry, Annex A
- [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] FAO JECFA Combined Compendium of Food Additive Specifications, '*Silicon dioxide, amorphous*', Monograph No. 1 (2006)
<http://www.fao.org/ag/agn/jecfa-additives/specs/Monograph1/Additive-385.pdf>
(last visited on 09/07/2015)
- [6] Food Chemicals Codex, '*Silicon dioxide*' monograph. Seventh Edition (2010 - 2011)

*Refers to Dossier no: FAD-2010-0226

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.

8. ACKNOWLEDGEMENTS

The following National Reference Laboratories contributed to this report:

- Centro di referenza nazionale per la sorveglianza ed il controllo degli alimenti per gli animali (CReAA), Torino (IT)
- Fødevarestyrelsens Laboratorier Aarhus (kemisk) (DK)¹
- Laboratoire de Rennes (SCL L35), Service Commun des Laboratoires DGCCRF et DGDDI, Rennes (FR)²
- Thüringer Landesanstalt für Landwirtschaft (TLL). Abteilung Untersuchungswesen, Jena (DE)
- Ústřední kontrolní a zkušební ústav zemědělský (ÚKZÚZ), Praha (CZ)
- Państwowy Instytut Weterynaryjny, Pulawy (PL)
- Univerza v Ljubljani. Veterinarska fakulteta. Nacionalni veterinarski inštitut. Enota za patologijo prehrane in higieno okolja, Ljubljana (SI)
- Staatliche Betriebsgesellschaft für Umwelt und Landwirtschaft. Geschäftsbereich 6 - Labore Landwirtschaft, Nossen (DE)³

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