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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

Sodium Selenate

(FAD-2010-0149; CRL/100198)

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-2010-0149 - CRL/100198**
Name of Feed Additive:	**Sodium Selenate**
Active Agent (s):	**Sodium Selenate**
Rapporteur Laboratory:	**European Union Reference Laboratory for Feed Additives (EURL-FA)** **Geel, Belgium**
Report prepared by:	**Zigmas Ezerskis**
Report checked by: Date:	**Piotr Robouch (EURL-FA)** **23/02/2015**
Report approved by: Date:	**Christoph von Holst** **24/02/2015**

EXECUTIVE SUMMARY

In the current application authorisation is sought under article 10(2) for *sodium selenate* under the category / functional group (3b) "nutritional additives"/"compounds of trace elements", 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 all categories and species. The *feed additive* is a white powder containing at least of 98% of *sodium selenate* (Na_2SeO_4) based on anhydrous weight, which corresponds to a minimum content of 41% *selenium* and 24% *sodium*. The *feed additive* is intended to be incorporated into *feedingstuffs* through *premixtures* with maximum levels of 0.5 mg *total selenium* /kg *feedingstuffs*.

For the quantification of *total selenium* in the *feed additive* the Applicant suggested an assay based on the volumetric method of iodometric analysis. The EURL considers this method suitable for the official control.

For the quantification of *total sodium* in *feed additive* the EURL identified two ring-trial validated methods: - EN ISO 6869:2000, based on Atomic Absorption Spectrometry (AAS) after dilution in hydrochloric acid, and - EN 15510:2007, based on Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES) after dilution in hydrochloric acid.

For the quantification of *total selenium* in *premixture* samples, the Applicant applied flame atomic absorption spectrometry (AAS) in the frame of homogeneity studies. Furthermore, the Applicant submitted for the quantification of *total selenium* in *premixtures* and *feedingstuffs* the EN 16159:2012 method, based on Hydride Generation Atomic Absorption Spectrometry (HGAAS) after microwave digestion with HNO_3/H_2O_2. This method was already evaluated and recommended by the EURL in the frame of previous *selenium* dossiers.

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

Sodium Selenate, Selenium, nutritional additives, compounds of trace elements, all animal species and categories

1. BACKGROUND

In the current application authorisation is sought under article 10(2) (re-evaluation of the already authorised additives under provisions of Council Directive 70/524/EEC) for *sodium selenate* under the category / functional group (3b) "nutritional additives"/"compounds of trace elements", 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 all categories and species [1].

The *feed additive* is a white powder containing at least of 98 % of *sodium selenate* (Na_2SeO_4) based on anhydrous weight, which corresponds to a minimum content of 41% *selenium* and 24% *sodium* [2,3].

The *feed additive* is intended to be incorporated into *feedingstuffs* through *premixtures* with maximum levels of 0.5 mg *total selenium* /kg *feedingstuffs* [2,3], thus complying with legal requirements; no minimum dose was proposed by the Applicant.

Note: The Applicant requested the withdrawal of the current application and the Commission services accepted this request [4].

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 *sodium selenate* 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 and mercury) are available from the respective European Union Reference Laboratories [5].

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

For the quantification of *total selenium* in the *feed additive* the Applicant suggested an assay based on titraton [3]. The determination of the assay is based on the volumetric method of iodometric analysis wherein standard thiosulphate solution is used as titrant and starch as the indicator. Potassium iodide is added and the endpoint is reached when the blue complex disappears. The EURL considers this method suitable for the official control.

For the quantification of *total sodium* in the *feed additive* the EURL identified the following two ring-trial validated methods: - EN ISO 6869:2000, based on Atomic Absorption Spectrometry (AAS) after dilution in hydrochloric acid [6], and - EN 15510:2007, based on Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES) after dilution in hydrochloric acid [7], for which relative precisions ranging from 4 to 27 % were reported.

For the quantification of total selenium in *premixture* samples, the Applicant applied flame atomic absorption spectrometry (AAS) in the frame of homogeneity studies [8]. Furthermore, the Applicant submitted for the quantification of *total selenium* in *premixtures* and *feedingstuffs* the EN 16159:2012 method [9], based on Hydride Generation Atomic Absorption Spectrometry (HGAAS) after microwave digestion with HNO_3/H_2O_2 – a method that was originally developed and ring-trial validated by the "Association of German Agricultural Analytical and Research Institutes" (VDLUFA, Germany) [10]. This method was already evaluated by the EURL in the frame of previous selenium dossiers. The following performance characteristics were reported for feed samples:

- a relative standard deviation for *repeatability* (RSD_r) ranging from 3.4 to 10 %;
- a relative standard deviation for *reproducibility* (RSD_R) ranging from 15 to 23 %; and
- a limit of quantification of 0.125 mg/kg, clearly below the maximum legal limit of 0.5 mg Se /kg feed.

Based on the performance characteristics presented the EURL considers the EN 16159:2012 method suitable for the official control to determine *total selenium* 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

In the frame of this authorisation the EURL considers the following methods suitable for official control:

- titration, for the quantification of *total selenium* in the *feed additive*;
- the ring trial validated methods EN ISO 6869:2000, based on AAS or EN 15510:2007 based on ICP-AES, for the quantification of *total sodium* in the *feed additive*;
- the ring-trial validated method (EN 16159:2012), based on HGAAS for the quantification of *total selenium* in *premixtures* and *feedingstuffs.*

5. DOCUMENTATION AND SAMPLES PROVIDED TO EURL

In accordance with the requirements of Regulation (EC) No 1831/2003, reference samples of *sodium selenate* 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.1831/0041-2014

[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] *Withdrawal of application, Reference FAD-2010-0149 / SANTE-0041-2014

[5] 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

[6] EN ISO 6869:2000 – Animal feedingstuffs – Determination of the contents of calcium, copper, iron, magnesium, manganese, potassium, sodium and zinc - Method using atomic absorption spectrometry

[7] EN 15510:2007 – Animal feedingstuffs – Determination of calcium, sodium, phosphorus, magnesium, potassium, iron, zinc, copper, manganese, cobalt, molybdenum, arsenic, lead and cadmium by ICP-AES

[8] *Technical dossier, Section II: Identity, characterisation and conditions of use of the feed additive; methods of analysis – Annex II-17

[9] EN 16159:2012 – Animal feeding stuffs – Determination of selenium by hydride generation atomic absorption spectrometry (HGAAS) after microwave digestion (digestion with 65% nitric acid and 30% hydrogen peroxide)

[10] VDLUFA Methodenbuch III (2003), 11.6.1 – Selen

*Refers to Dossier no: FAD-2010-0149

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

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:

- Österreichische Agentur für Gesundheit und Ernährungssicherheit (AGES), Wien (AT)
- Staatliche Betriebsgesellschaft für Umwelt und Landwirtschaft. Geschäftsbereich 6 - Labore Landwirtschaft, Nossen (DE)[1]
- Państwowy Instytut Weterynaryjny, Puławy (PL)
- Ústřední kontrolní a zkušební ústav zemědělský (ÚKZÚZ), Praha (CZ)
- 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)
- Instytut Zootechniki w Krakowie, Krajowe Laboratorium Pasz, Lublin (PL)
- Thüringer Landesanstalt für Landwirtschaft (TLL), Abteilung Untersuchungswesen, Jena (DE)

Name and addresses according to Regulation (EC) No 885/2009:

[1] Sächsische Landesanstalt für Landwirtschaft. Fachbereich 8 – Landwirtschaftliches Untersuchungswesen, Leipzig