EUROPEAN COMMISSION
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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 Molybdate

(FAD-2010-0256; CRL/100164)

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-2010-0256 - CRL/100164**
Name of Feed Additive:	**Sodium Molybdate**
Active Agent (s):	**Sodium Molybdate**
Rapporteur Laboratory:	**European Union Reference Laboratory for Feed Additives (EURL-FA)** **Geel, Belgium**
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Report checked by: Date:	**Piotr Robouch (EURL-FA)** **26/04/2016**
Report approved by: Date:	**Christoph von Holst** **28/04/2016**

EXECUTIVE SUMMARY

In the current application authorisation is sought under article 10(2) for *sodium molybdate* 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 white crystalline powder, marketed in the form of sodium molybdate dihydrate (Na_2MoO_4 $2H_2O$), containing a minimum of 37% molybdenum. The *feed additive* is intended to be incorporated into *feedingstuffs* through *premixtures* with a maximum recommended level of 2.5 mg *total molybdenum* / kg complete *feedingstuffs*.

For the characterisation of the *feed additive* the Applicant did not propose any method. However, the EURL identified the European Pharmacopoeia monograph 1565, where: - identification is based on "loss on drying" and specific reactions of molybdate and sodium ions; while - quantitative assay is based on titration with lead nitrate in the presence of hexamethylenetetramine and nitric acid till the endpoint determined by 4-(2-pyridylazo) resorcinol monosodium salt as an indicator.

For the quantification of *total sodium* in the *feed additive*, the EURL identified two internationally recognised ring-trial validated CEN methods: (i) EN 15510, based on Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES); and (ii) EN ISO 6869, based on Atomic Absorption Spectrometry (AAS).

For the *quantification* of *total molybdenum* in the *feed additive*, *premixtures* and *feedingstuffs* the Applicant submitted the internationally recognised ring-trial validated CEN method (EN 15510) based on inductively coupled plasma atomic emission spectroscopy (ICP-AES). The Applicant applied the ICP-AES method to quantify *total molybdenum* in five batches of the *feed additive* and reported a relative standard deviation for *repeatability* (RSD_r) of 1.9% for samples containing 39% *total molybdenum*. The following performance characteristics were reported in the EN 15510 standard for *premixtures* and *feedingstuffs* samples containing from 1.06 to 16700 mg/kg *total molybdenum*: RSD_r ranging from 3.1 to 16%; and a relative standard deviation for *reproducibility* (RSD_R) ranging from 11 to 32%.

Based on the data available the EURL recommends for official control the above mentioned ring-trial validated CEN methods for the quantification of *total molybdenum* and *total sodium* in the *feed additive*; *premixtures* and *feedingstuffs* (only *total molybdenum*).

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 Molybdate, 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 molybdate* 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 white crystalline powder, marketed in the form of sodium molybdate dihydrate (Na_2MoO_4 $2H_2O$), containing a minimum of 37% molybdenum [2].

The *feed additive* is intended to be incorporated into *feedingstuffs* through *premixtures* with a maximum recommended level of 2.5 mg *total molybdenum* / kg complete *feedingstuffs* [2,3].

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 *sodium molybdate* 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].

For the characterisation of the *feed additive* the EURL identified the European Pharmacopoeia monograph 1565 [5], where:

- identification is based on "loss on drying" and specific reactions of molybdate and sodium ions; while
- quantitative assay is based on titration with lead nitrate in the presence of hexamethylenetetramine and nitric acid till the endpoint determined by 4-(2-pyridylazo)resorcinol monosodium salt as an indicator; where 1 ml of 0.05 M lead nitrate is equivalent to 10.30 mg of Na_2MoO_4.

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

For the quantification of *total sodium* in the *feed additive*, the EURL identified two internationally recognised ring-trial validated CEN methods:

i. EN 15510, based on Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES) [6]; and

ii. EN ISO 6869, based on Atomic Absorption Spectrometry (AAS) after dissolving in hydrochloric acid [7] for which relative precisions ranging from 4 to 27% were reported for *total sodium* content up to 237 g/kg feed related matrices.

For the *quantification* of *total molybdenum* in the *feed additive*, *premixtures* and *feedingstuffs* the Applicant submitted the internationally recognised ring-trial validated CEN method (EN 15510) [6] based on inductively coupled plasma atomic emission spectroscopy (ICP-AES).

When applying the EN 15510 method, a test portion of the sample is ashed and dissolved in hydrochloric acid (in the case of organic *feedingstuffs*) or wet digested with hydrochloric acid (in the case of mineral compounds) for further analysis by ICP-AES [6].

The Applicant applied the ICP-AES method to quantify *total molybdenum* in five batches of the *feed additive* and reported a relative standard deviation for repeatability (RSD_r) of 1.9% for samples containing 39% *total molybdenum* [3,8]. The following performance characteristics were reported in the EN 15510 standard for *premixtures* and *feedingstuffs* samples containing from 1.06 to 16700 mg /kg *total molybdenum*:

- RSD_r ranging from 3.1 to 16%; and
- a relative standard deviation for *reproducibility* (RSD_R) ranging from 11 to 32%.

Based on the data available the EURL recommends for official control the two ring-trial validated CEN methods mentioned above for the quantification of *total molybdenum* and/or *total sodium* in the *feed additive*; *premixtures* and *feedingstuffs* (only for *total molybdenum*).

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:

- the ring-trial validated CEN method (EN 15510) based on inductively coupled plasma atomic emission spectroscopy (ICP-AES) for the quantification of *total molybdenum* in the *feed additive*, *premixtures* and *feedingstuffs*; and
- two internationally recognised ring-trial validated methods for the quantification of *total sodium* in the *feed additive*: EN 15510, based on inductively coupled plasma atomic emission spectrometry (ICP-AES) and EN ISO 6869, based on atomic absorption spectrometry (AAS).

Recommended text for the register entry (analytical method)

For the quantification of *total molybdenum* content in *feed additive*, *premixtures* and *feedingstuffs*:

- inductively coupled plasma atomic emission spectroscopy (ICP-AES) – EN 15510

For the quantification of *total sodium* content in the *feed additive*:

- inductively coupled plasma atomic emission spectrometry (ICP-AES) – EN 15510; or
- atomic absorption spectrometry (AAS) – EN ISO 6869

5. DOCUMENTATION AND SAMPLES PROVIDED TO EURL

In accordance with the requirements of Regulation (EC) No 1831/2003, reference samples of *sodium molybdate* 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/G1: Forw. Appl.1831/0034-2015

[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] European Pharmacopoeia Monograph 01/2008:1565

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

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

[8] *Technical dossier, Section II – Annex II_1

*Refers to Dossier no: FAD-2010-0256

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 (EU) 2015/1761.

8. ACKNOWLEDGEMENTS

The following National Reference Laboratories contributed to this report:

- Laboratori Agroalimentari, Departament d'Agricultura, Ramaderia, PESCA, Alimentació i Medi Natural. Generalitat de Catalunya, Cabrils (ES)
- Laboratorio Arbitral Agroalimentario. Ministerio de Agricultura, Alimentación y Medio Ambiente, Madrid (ES)
- Centro di referenza nazionale per la sorveglienza ed il controllo degli alimenti per gli animali (CReAA), Torino (IT)
- Ústřední kontrolní a zkušební ústav zemědělský (ÚKZÚZ), Praha (CZ)
- Państwowy Instytut Weterynaryjny, Pulawy (PL)
- Thüringer Landesanstalt für Landwirtschaft (TLL). Abteilung Untersuchungswesen. Jena (DE)
- Österreichische Agentur für Gesundheit und Ernährungssicherheit (AGES), Wien (AT)
- Laboratoire de Rennes (SCL L35), Service Commun des Laboratoires DGCCRF et DGDDI, Rennes (FR)