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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**

Manganese hydroxychloride
(FAD-2012-0040; CRL/120030)



**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-2012-0040 - CRL/120030**

Name of Product: **Manganese hydroxychloride**

Active Agent (s): **Manganese hydroxychloride**

Rapporteur Laboratory: **European Union Reference Laboratory
for Feed Additives (EURL-FA)
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Date: **30/01/2014**

Report approved by: **Christoph von Holst**
Date: **30/01/2014**

EXECUTIVE SUMMARY

In the current application authorisation is sought under article 4(1) for *manganese hydroxychloride* 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 species and categories. *Manganese hydroxychloride* ($Mn_2(OH)_3Cl$) is a brown free-flowing crystalline powder with a minimum content of 44 % *total manganese* and a *chloride* content ranging from 16 to 19 %. The *feed additive* is intended to be mixed into *premixtures* and *feedingstuffs*. The Applicant suggested maximum levels ranging from 100 to 150 mg *total manganese* /kg *feedingstuffs*.

For the quantification of *chloride* in the *feed additive*, the Applicant suggested the ion chromatography method described in the U.S. Environmental Protection Agency (EPA method 300.0, Rev.2.1). However, the EURL recommends instead the Community method, based on titration, for determination of *chlorine* in the *feed additive*.

For the quantification of *total manganese* content in the *feed additive*, *premixtures* and *feedingstuffs* the Applicant submitted the ring-trial validated CEN method (EN 15510) based on Inductively Coupled Plasma Atomic Emission Spectroscopy (ICP-AES). In addition to this method, the EURL already recommended - in the frame of the evaluation of the manganese group (cf. FAD-2010-1169, FAD-2010-0088 and FAD-2010-0235) - two other ring-trial validated methods: EN 15621, based on ICP-AES after pressure digestion; and - EN ISO 6869 based on Atomic Absorption Spectrometry (AAS), together with the Community method, further ring-trial validated by the UK Food Standards Agency.

Based on the acceptable method performance characteristics available the EURL recommends for official control all the methods mentioned above for the quantification of *total manganese* in the *feed additive*, *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

Manganese hydroxychloride, nutritional additives, compounds of trace elements, all animal species and categories

1. BACKGROUND

In the current application authorisation is sought under article 4(1) (new feed additive) for *manganese hydroxychloride* 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 [1]. Specifically, authorisation is sought for the use of the *feed additive* for all species and categories [1, 2].

Manganese hydroxychloride ($Mn_2(OH)_3Cl$) is a brown free-flowing crystalline powder [3] with a minimum content of 44 % *total manganese* and a *chloride* content ranging from 16 to 19 % [2, 3].

The *feed additive* is intended to be mixed into *premixtures* and *feedingstuffs* [3]. The Applicant suggested maximum levels ranging from 100 to 150 mg *total manganese* /kg *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 *manganese hydroxychloride* 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].

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

The Applicant did not provide any method for the identification of *manganese hydroxychloride*, however, the EURL considers X-ray diffraction (XRD) method described in the generic European Pharmacopoeia monograph [5] for the characterisation of the orthorhombic crystalline structure of $Mn_2(OH)_3Cl$. The X-Ray diffraction pattern is to be compared to the reference one published in the Handbook of Mineralogy [6].

For the quantification of the *chloride* content, the Applicant submitted the ring-trial validated, U.S. Environmental Protection Agency (EPA) method based on ion chromatography for the determination of inorganic anions [7]. However, the EURL recommends instead the Community method for determination of *chlorine* (from chlorides) in the *feed additive* [8]. The method is based on titration by silver nitrate and ammonium thiocyanate, and the content of *chlorine* is expressed as % of NaCl.

For the quantification of total manganese content in the *feed additive, premixtures and feedingstuffs* the Applicant submitted the CEN method (EN 15510) based on Inductively Coupled Plasma Atomic Emission Spectroscopy (ICP-AES) [9].

Additionally, the EURL already evaluated in the frame of the evaluation of the manganese group (cf. FAD-2010-1169; FAD-2010-0088; FAD-2010-0235 [10]) two ring-trial validated methods: - EN 15621 based on ICP-AES after pressure digestion [11] and EN ISO 6869 based on Atomic Absorption Spectrometry (AAS) [12], together with the Community method [13] based on AAS method (for feed samples only). The later was further verified by the UK Food Standards Agency (FSA) recently [14], using samples such as dog biscuits, layer pellets, beef nuts, sow rolls or rabbit pellets. The performance characteristics reported by the four ring-trials mentioned above are summarised in Table 1.

Table 1: Performance characteristics for the quantification of *total manganese* in *premixtures and feedingstuffs*

	EN 15510 [9]	EN 15621 [11]	EN 6869 [12]	FSA [14]
Method	ICP-AES	ICP-AES	AAS	AAS
Content (mg/kg)	127 – 3527	99 – 3440	16 – 13200	30 – 128
RSD _r (%)	1.9 – 6.2	2.5 – 10	1.0 – 4.2	2.7 – 4.3
RSD _R (%)	5.0 – 9.5	10 - 15	3.4 – 20	5.2 – 7.1
LOQ (mg/kg)	3	1	5	20

RSD_r and RSD_{ip}: relative standard deviation for *repeatability* and *intermediate precision*;

LOQ: limit of quantification

Based on the acceptable method performance characteristics available the EURL recommends for official control the four ring-trail validated methods mentioned above for the quantification of *total manganese* in the *feed additive*, *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 recommends for official control:

- the Community method based on titration, for the quantification of *chlorine* in the *feed additive*;
- three CEN methods (EN 6869, EN 15510 and EN 15621) for the quantification of *total manganese* content in the *feed additive*, *premixtures* and *feedingstuffs*; or
- the Community method based on AAS, for the quantification of *total manganese* in *feedingstuffs*

Recommended text for the register entry (analytical method)

For the quantification of *chlorine* in the *feed additive*:

- Titration - Commission Regulation (EC) No 152/2009

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

- Atomic Absorption Spectrometry (AAS) - EN ISO 6869; or
- Inductively Coupled Plasma – Atomic Emission Spectrometry (ICP-AES) - EN 15510; or
- Inductively Coupled Plasma – Atomic Emission Spectrometry after pressure digestion (ICP-AES) - EN 15621; or
- Atomic Absorption Spectrometry (AAS) (for *feedingstuffs* only) - Commission Regulation (EC) No 152/2009

5. DOCUMENTATION AND SAMPLES PROVIDED TO EURL

In accordance with the requirements of Regulation (EC) No 1831/2003, reference samples of *manganese hydroxychloride* 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.1831/0069-2012
- [2] *Application, Proposal for Register Entry – Annex A
- [3] *Technical dossier, Section II
- [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 01/2008:20933
- [6] John W. Anthony, Richard A. Bideaux, Kenneth W. Bladh, and Monte C. Nichols, Eds., Handbook of Mineralogy, Mineralogical Society of America, Chantilly, VA 20151-1110, USA. <http://www.handbookofmineralogy.org/>
- [7] *Technical dossier, Section II – Annex 2.6.3.b (U.S. EPA method 300.0 Rev 2.1 – Determination of inorganic anions by ion chromatography)
- [8] Commission Regulation (EC) No 152/2009 of 27 January 2009 laying down the methods of sampling and analysis for the official control of feed (cf. Annex III-Q)
- [9] 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*
- [10] <http://irmm.jrc.ec.europa.eu/SiteCollectionDocuments/FinRep-SANCO-Manganese.pdf>
- [11] EN 15621:2012 – *Animal feeding stuffs – Determination of cadmium, sodium, phosphorus, magnesium, potassium, sulphur, iron, zinc, copper, manganese, cobalt and molybdenum after pressure digestion by ICP-AES*
- [12] 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*
- [13] Commission Regulation (EC) No 152/2009 of 27 January 2009 laying down the methods of sampling and analysis for the official control of feed (cf. Annex IV-C)
- [14] Supplementary Information - FAD-2010-0046: Food Standards Agency – Information Bulletin on Methods of Analysis and Sampling for Foodstuffs, No 102; March 2010

*Refers to Dossier No. FAD-2012-0040

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

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- Instytut Zootechniki w Krakowie, Krajowe Laboratorium Pasz, Lublin (PL)
- Schwerpunktlabor Futtermittel des Bayerischen Landesamtes für Gesundheit und Lebensmittelsicherheit (LGL), Oberschleißheim (DE)
- 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)
- Państwowy Instytut Weterynaryjny, Puławy (PL)
- Laboratorio Arbitral Agroalimentario, Ministerio de Agricultura, Alimentación y Medio Ambiente, Madrid¹ (ES)
- Österreichische Agentur für Gesundheit und Ernährungssicherheit (AGES), Wien (AT)
- Laboratoire de Rennes, SCL L35, Service Commun des Laboratoires, Rennes (FR)

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