



JRC.DG.D.6/CvH/GB/mds/ARES(2011)722146

**EURL Evaluation Report on the Analytical Methods
submitted in connection with the Application for the
Authorisation of Feed Additives according to
Regulation (EC) No 1831/2003**

Dossier related to: FAD-2010-0037
CRL/100030

FAD-2010-0242
CRL/100294

Product Name:

Active Substance(s): Ammonium chloride

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Date: 04/07/2011

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EXECUTIVE SUMMARY

In the current applications authorisation is sought: - under articles 4(1) and 10(2) for *ammonium chloride* (E510) under the category "technological additives", functional group 1j "acidity regulators" by Applicant I (FAD-2010-0242), and - under articles 4(1) for *Amoklor* (*ammonium chloride*) under the category "zootechnical additives", functional group 4d "other zootechnicals", by Applicant II (FAD-2010-0037) according to the classification system of Annex I of Regulation (EC) No 1831/2003. According to Applicants I and II, the *feed additive* is a white, crystalline powder with a minimum purity of 99.0 and 99.5%, respectively.

Specifically, authorisation is sought for the use of the *feed additive* for bovines, sheep, cats and dogs by Applicant I and lambs for fattening by Applicant II. The *feed additive* is intended to be used in *feedingstuffs*. Applicant I and II suggested for ammonium chloride a concentration in *feedingstuffs* ranging from 2 to 20 g/kg and from 5 to 10 g/kg, respectively, while no limits were set in previous regulations.

For the quantification of *ammonium chloride* in the *feed additive*, Applicant I proposes a method similar to the method described in the European Pharmacopoea Monograph 0007. The EURL recommends for official control the internationally recognised European Pharmacopoeia method or the internationally recognised JECFA method to determine *ammonium chloride* in the *feed additive*.

The unambiguous quantification of *ammonium chloride* in *feedingstuffs* is not achievable by analytical methods. Hence, the EURL does not recommend for official control any methods for the quantification of *ammonium chloride* in *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

ammonium chloride, AMOKLOR, technological additive, acidity regulators, zootechnical additive, other zootechnicals, bovines, sheep, cats, dogs, lambs for fattening

1. BACKGROUND

In the current applications authorisation is sought:

- under articles 4(1) and 10(2) for *ammonium chloride*(E510) under the category "technological additives", functional group 1j "acidity regulators" (Applicant I, FAD-2010-0242 [1]); and
- under article 4(1) for *Amoklor* (*ammonium chloride*) under the category "zootechnical additives", functional group 4d "other zootechnicals" (Applicant II, FAD-2010-0037 [2]),

according to the classification system of Annex I of Regulation (EC) No 1831/2003.

According to Applicants I and II, the *feed additive* is a white, crystalline powder with a minimum purity of 99.0 % [3, 4] and 99.5% [5, 6], respectively.

Specifically, authorisation is sought for the use of the *feed additive* for bovines, sheep, cats and dogs (by Applicant I [3]) and lambs for fattening (by Applicant II [5]).

The *feed additive* is intended to be used in *feedingstuffs*. Applicants I and II suggested concentrations in *feedingstuffs* ranging from 2 to 20 g/kg [3] and 5 to 10 g/kg [5], respectively; while no limits were set in the previous regulations [7].

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 *Ammonium chloride & Amoklor*, 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

For the identification of *ammonium chloride* the EURL recommends the internationally recognised European Pharmacopoeia method [8] or JECFA method [12], based on (i) selective reactions with *chloride* ions and (ii) the selective reactions with *ammonium* ions.

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, PAHs, dioxins) are available from the respective European Union Reference Laboratories [9].

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

For the quantification of *ammonium chloride* in the *feed additive*, Applicant I proposes a method [10] similar to the method described in the European Pharmacopoeia Monograph. The EURL recommends instead for official control the internationally recognised European Pharmacopoeia method (Monograph 0007 [8]), based on acid/base titration with 1 M sodium hydroxide and phenolphthalein as indicator, or the internationally recognised JECFA method (monograph 'ammonium chloride') [12], based on titration with 0.1 N silver nitrate to quantify *ammonium chloride* in the *feed additive*.

For the quantification of *ammonium chloride* in *feedingstuffs*, Applicant I submitted two methods for the determination of (i) ammonia content and (ii) chloride content. The sample is extracted with water, and the solution is filtered. Ion-chromatography with conductivity detector (IC-CD) after a Kjeldahl distillation is used for the determination of ammonia, while potentiometric titration with 0.1 M silver nitrate solution [11] is used for the determination of chloride. However, neither validation nor verification data were provided by the Applicant.

The quantification of added *ammonium chloride* in *feedingstuffs* is only possible when identical untreated feed (control) samples are available, thus allowing corrections for endogenous *ammonium* and *chloride* present in the feed; such cases are not frequent. When untreated feeds are *not* available, the estimate of the total contents of "ammonia" and "chloride" would be meaningless, due to the endogenous contributions from other sources than *ammonium chloride*. In consequence, the determination of the individual ions (i.e. ammonia and chloride) does not allow for the quantification of ammonium chloride. Therefore, the EURL does not recommend for official control any methods for the quantification of *ammonium chloride* in *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 European Pharmacopoeia Monograph methods (Monograph 0007) or JECFA Monograph methods ('ammonium chloride') for the identification and the quantification of *ammonium chloride* in *feed additive*

Recommended text for the register entry (analytical method)

For the quantification of the *ammonium chloride* in the *feed additive*:

- titration with sodium hydroxide (European Pharmacopoeia monograph 0007) or titration with silver nitrate (JECFA monograph 'ammonium chloride')

5. DOCUMENTATION AND SAMPLES PROVIDED TO EURL

In accordance with the requirements of Regulation (EC) No 1831/2003, reference samples of *Ammonium chloride* and *Amoklor* 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/D/2 Forw. Appl. 1831/00152(10267)/2010
 - [2] +Application, Reference SANCO/D/2 Forw. Appl. 1831/073-2010
 - [3] *Application, Proposal for Register Entry – Annex A
 - [4] *Technical dossier, Section II
 - [5] +Application, Proposal for Register Entry – Annex A
 - [6] +Technical dossier, Section II
 - [7] Council Directive 70/524/EEC of 23 November 1970 concerning additives in feeding-stuffs
 - [8] European Pharmacopoeia Monograph 0007
 - [9] 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
 - [10] *Technical dossier, Section II – Annex-"Determination of ammonium chloride"
 - [11] *Technical dossier, Section II – Annex-"Determination of ammonium chloride in animal feed"
 - [12] JECFA Monograph 'ammonium chloride'
- * Refers to Dossier No. FAD-2010-0242
+ Refers to Dossier No. FAD-2010-0037

7. RAPPORTEUR LABORATORY & NATIONAL REFERENCE LABORATORIES

The Rapporteur Laboratory for this evaluation was European 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
- Plantedirektoratet, Laboratorium for Foder og Gødning, Lyngby, DK