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European Union Reference Laboratory

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

Potassium diformate
(FAD-2013-0021; CRL/130002)



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

Dossier related to: **FAD-2013-0021 - CRL/130002**

Name of Product **Potassium diformate**

Active Substance(s): **Potassium diformate**

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

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Date: **13/11/2013**

EXECUTIVE SUMMARY

In the current application authorisation is sought under article 4(1) for *potassium diformate* under the category/functional group 1(j) "technological additives"/"acidity regulators", according to the classification system of Annex I of Regulation (EC) No 1831/2003. The authorisation is sought for pigs for fattening, piglets (weaned), sows for reproduction and chickens for fattening. The *feed additive* is intended to be used in *premixtures* and/or *feedingstuffs*. The Applicant proposes a maximum concentration of 5, 12 and 18 g/kg in *feedingstuffs* for chickens for fattening, sows for reproduction and pigs for fattening and weaned piglets, respectively. The *feed additive* is an aqueous solution of $\text{CHKO}_2 \cdot \text{CH}_2\text{O}_2$ with a solid fraction of ca. 50%.

The Applicant submitted the following three international standards for the characterisation of *potassium diformate* in the *feed additive*:

- for the determination of *potassium* in the *feed additive*: EN ISO 6869 based on atomic absorption spectrometry (AAS) and EN 15510 based on inductively coupled plasma atomic emission spectrometry (ICP-AES),
- for the determination of *formate* in the *feed additive*: EN 15909 based on reverse phase high performance liquid chromatography with UV detection (RP-HPLC-UV) at 214 nm.

The EURL recommends for official control the above mentioned standards for the characterisation of *potassium diformate* in the *feed additive*.

Furthermore, the Applicant submitted the two methods already evaluated and recommended by the EURL for the determination of *diformate* as total formic acid in the *premixtures* and *feedingstuffs*:

- based on ion-exclusion high performance liquid chromatography with ultraviolet or refractive index detection (IEC-HPLC-UV/RI);
- based on the ion chromatography with electrical conductivity detection (IC-ECD).

The EURL recommends for official control the above mentioned methods for the determination of *diformate* as total formic acid in the *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

Potassium diformate, technological additives, acidity regulators, pigs for fattening, piglets (weaned), sows for reproduction and chickens for fattening.

1. BACKGROUND

In the current application authorisation is sought under article 4(1) for *potassium diformate* under the category/functional group 1(j) "technological additives"/"acidity regulators", according to the classification system of Annex I of Regulation (EC) No 1831/2003 [1]. The authorisation is sought for the use of the feed additive for pigs for fattening, piglets (weaned), sows for reproduction and chickens for fattening [1].

The *feed additive* is intended to be used in *premixtures* and/or *feedingstuffs*. The Applicant proposes a maximum concentration of 5, 12 and 18 g/kg in *feedingstuffs* for chickens for fattening, sows for reproduction and pigs for fattening and weaned piglets, respectively [2].

The *feed additive* is an aqueous solution of $\text{CHKO}_2 \cdot \text{CH}_2\text{O}_2$ with a solid fraction of ca. 50% [3].

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 *potassium diformate*, and their suitability to be used for official controls in the frame of the authorisation, were evaluated.

3. EVALUATION

Qualitative and quantitative composition of impurities in the feed additive

When required by EU legislation, analytical methods for official control of undesirable substances in the additive such as heavy metals (arsenic, cadmium, lead and mercury), dioxins, microbiological agents and mycotoxins are available from the respective European Union Reference Laboratories [4].

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

For the determination of *potassium* in the *feed additive* the applicant proposed two international standards [3]: - EN ISO 6869 based on atomic absorption spectrometry (AAS) and - EN 15510, based on inductively coupled plasma atomic emission spectrometry (ICP-

AES). The reported relative standard deviations for *repeatability* (RSD_T) and for *reproducibility* (RSD_R) range from 2 to 10 % when excluding mineral *premixtures* [5, 6],.

For the determination of *diformate* as *formate* in the *feed additive* the Applicant proposed another international standard (EN 15909) [3] based on the reverse phase high performance liquid chromatography with UV detection at 214 nm (RP-HPLC-UV). Quantification is performed by using external calibration with *formate* standard solutions. The following performance characteristics were reported: $RSD_T = 0.7\%$ and $RSD_R = 2.2\%$ [7].

The EURL recommends for official control all the three methods mentioned above for the characterisation of *potassium diformate* in the *feed additive*.

For the determination of *diformate* as total formic acid in the *premixtures* and *feedingstuffs* the Applicant proposed the two methods already evaluated and recommended by the EURL, based on:

- ion-exclusion high performance liquid chromatography with UV or RI detection (IEC-HPLC-UV/RI). The method developed by FEFANA is considered as candidate method in the CEN standardisation process. This method was recommended by the EURL in the previous report (FAD-2010-0188, FAD-2010-0303, FAD-2010-0312) [8] for the determination *formate* as formic acid in *premixtures* and *feedingstuffs*. RSD_R determined in the ring-trial ranges from 6.6 to 19.3% at the concentration levels of 4.5-44 g/kg. Furthermore, the Applicant provided supplementary experimental evidence confirming the suitability of the HPLC/RI method for determination of formic acid in *feedingstuffs* and reported a recovery rate of 99% and LOQ of 983 mg/kg *feedingstuffs* [10].
- ion chromatography with electrical conductivity detection (IC-ECD). This method was recommended by the EURL in the report of FAD-2009-0027 [9] for the determination of the formic acid in *premixtures* and *feedingstuffs*. A proficiency test was organised by VDLUFA in 2006 for the determination of organic acids including formic acid in *feedingstuffs*. Most of the laboratories used IC-ECD technique and the following performance characteristics were reported for formic acid: RSD_T ranging from 4 to 10%; and RSD_R ranging from 13 to 22% at the concentration levels of 7.2 - 506 g/kg *feedingstuffs*.

Based on the experimental evidence available and the information provided, the EURL recommends the two methods mentioned above for the determination of *diformate* as total formic acid 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 recommends for official control:

- EN ISO 6869, based on atomic absorption spectrometry (AAS) and EN 15510, based on inductively coupled plasma atomic emission spectrometry (ICP-AES), for the determination of *potassium* in the *feed additive*;
- EN 15909, based on reverse phase HPLC-UV for determination of *diformate* as *formate* in the *feed additive*; and
- the two single-laboratory validated and further verified by several laboratories based on ion-exclusion HPLC-UV/RI and on IC-ECD for the determination of *diformate* as total formic acid in *premixtures*, and *feedingstuffs*.

Recommended text for the register entry (analytical method)

For the determination of *potassium* in the *feed additives*:

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

For the determination of *diformate* as *formate* in the *feed additive*:

- reverse phase high performance liquid chromatography with UV (RP-HPLC-UV) - EN 15909

For the determination of *diformate* as total formic acid in *premixtures*, and *feedingstuffs*:

- Ion-exclusion high performance liquid chromatography with UV or refractive index detection (IEC-HPLC-UV/RI) or
- Ion chromatography method equipped with electrical conductivity detection (IC-ECD)

5. DOCUMENTATION AND SAMPLES PROVIDED TO EURL

In accordance with the requirements of Regulation (EC) No 1831/2003, reference sample of *potassium diformate* 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] * Reference SANCO/D/2 Forw. Appl. 1831/0014-2013
- [2] * Application, Proposal for Register Entry

- [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] EN ISO 6869: Animal feeding stuffs - Determination of the contents of calcium, copper, iron, magnesium, manganese, potassium, sodium and zinc - Method using atomic absorption spectrometry
- [6] EN 15510: 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 15909: Fertilizers - Determination of calcium and formate in calcium foliar fertilizers
- [8] #Final reports FAD-210-0188, FAD-210-0303 and FAD-210-0312
- [9] #Final report FAD-2009-0027
- [10] Technical dossier, Section II –Annex II.29
* Refers to Dossier No FAD-2013-0021
http://irmm.jrc.ec.europa.eu/EURLs/EURL_feed_additives/authorisation/evaluation_reports/Pages/index.aspx

7. RAPPORTEUR LABORATORY

The Rapporteur Laboratory for this evaluation was the 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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- RIKILT - Instituut voor Voedselveiligheid, Wageningen (NL)
- Österreichische Agentur für Gesundheit und Ernährungssicherheit (AGES), Wien (AT)
- Państwowy Instytut Weterynaryjny, Puławy (PL)

- Staatliche Betriebsgesellschaft für Umwelt und Landwirtschaft, Labore Landwirtschaft, Leipzig (DE)
- Thüringer Landesanstalt für Landwirtschaft (TLL), Abteilung Untersuchungswesen, Jena (DE)