



Subject: Addendum to the EURL evaluation report

Reference: FAD-2008-0044 (*potassium diformate*) – D08/FSQ/CVH/MDr(2009)192341

Upon the recent publication of the new ring-trial validated method EN 17294 [1] for the analysis of organic acids in *feed additives*, *premixtures*, feed materials, *compound feed* and water, the EURL under the frame of article 5 of Regulation (EC) No 378/2005 [2] considered appropriate to perform a new evaluation of the methods of analysis for official control of *potassium diformate* in the *feed additive*, *premixtures* and *compound feed* in the frame of the above-mentioned *feed additive* dossier. In this line, aiming to recommend the available analytical methods complying with the highest requirements as stated in Annex II of Regulation (EC) No 429/2008 [3], the EURL also updates in this amendment the relevant method for the determination of *potassium* in the *feed additive*.

For the determination of *potassium diformate* (*as total formic acid*) in the *feed additive*, *premixtures*, *compound feed* and *water* the EURL evaluated the ring-trial validated EN 17294 method based on ion chromatography coupled to conductivity detection (IC-CD). This method is designed for the determination of *formic*, lactic, propionic, citric, fumaric, malic and acetic acids and their salts (*as total individual acids*) in *feed additives*, *premixtures*, feed materials and *compound feed* [1].

According to the method, 5 g of sample is mixed with 100 ml of water and the mixture is stirred for 60 min (or sonicated for 30 min). The resulting extract is filtered using ash free paper filter or centrifuged at 5000 g for 3 min. The filtrate or the supernatant after the dilution is filtered through a membrane filter before the chromatographic analysis. The individual analytes are detected by ion conductivity detection and the quantification is performed using an external standard calibration curve prepared from the standard solutions of the above-mentioned acids [1].

The performance characteristics obtained in the frame of the ring-trial validation studies of the EN 17294 method for the quantification of *formic acid* in *premixtures*, *feedingstuffs* (feed material, complementary feed, compound feed) and *water* are presented in Table 1. In addition, a limit of quantification (LOQ) of 200 mg for *formic acid*/kg *feedingstuffs* is reported [1].

Table 1. The performance characteristics obtained in the frame of the ring-trial validation studies of the EN 17294 method for the quantification of *formic acid* in *premixtures* and *feedingstuffs* (feed materials, complementary feed and *compound feed*) and water [1].

	Premixtures	Feedingstuffs	Water
Mass fraction, mg/kg	39935 - 269745	5669 - 26309	999
RSD _r , %	3.6 – 4.6	0.9 – 5.3	0.6
RSD _R , %	8.5 – 9.5	4.5 – 21.2	5.9
Reference	[1]		

RSD_r and RSD_R: relative standard deviations for *repeatability* and *reproducibility*, respectively.

Based on the performance characteristics presented and the scope of the method in terms of matrices, the EURL recommends for official control the ring-trial validated EN 17294 method based on ion chromatography coupled to conductivity detection (IC-CD) for the determination of *potassium diformate* (as total *formic acid*) in the *feed additive*, *premixtures* and *compound feed*.

In similar dossiers [4], for the determination of total *potassium* in the *feed additive* the EURL previously evaluated and recommended for official control ring-trial validated methods, namely: i) EN ISO 6869 based on atomic absorption spectrometry (AAS) [5] and ii) EN 15510 based on inductively coupled plasma-atomic emission spectrometry (ICP-AES) [6]. These recommendations are considered also valid in the frame of the present amendment.

Recommended text for the registry entry (analytical methods) (replacing the previous recommendations)

For the determination of *potassium diformate* (as total *formic acid*) in the *feed additive*, *premixtures* and *compound feed*:

- Ion chromatography with conductivity detection (IC-CD) – EN 17294

For the determination of total *potassium* in the *feed additive*:

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

References

- [1] EN 17294 Animal feeding stuffs: Methods of sampling and analysis – Determination of organic acids by Ion Chromatography with Conductivity Detection (IC-CD) – Complementary element
- [2] Commission Regulation (EC) No 378/2005 of 4 March 2005 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 tasks of the Community Reference Laboratory concerning applications for authorisations of feed additives, OJ L 059 5.3.2005, p. 8
- [3] Commission Regulation (EC) No 429/2008 of 25 April 2008 on detailed rules for the implementation of Regulation (EC) No 1831/2003 of the European Parliament and of the Council as regards the preparation and the presentation of applications and the assessment and the authorisations of feed additives, OJ L 133 22.5.2008, p. 1
- [4] EURL evaluation report:
<https://joint-research-centre.ec.europa.eu/system/files/2021-07/amended-finrep-formategroup.pdf>
- [5] 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
- [6] EN 15510:2017 - Animal feeding stuffs: Methods of sampling and analysis - Determination of calcium, sodium, phosphorus, magnesium, potassium, iron, zinc, copper, manganese, cobalt, molybdenum and lead by ICP-AES

Addendum

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- Reviewed and approved by Zigmantas Ezerskis and Christoph von Holst (EURL-FA),
respectively, Geel, 26/04/2023



D08/FSQ/CVH/MDr(2009)192341

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

Dossier related to: FAD-2008-0044
CRL/080019

Product name: Potassium diformate (Formi LHS)

Active Substance(s): Potassium diformate

Rapporteur Laboratory: Community Reference Laboratory
(CRL-FA)

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Date: 30/07/2009

Report approved by: Christoph von Holst

Date: 31/07/2009

EXECUTIVE SUMMARY

In the current application authorisation is sought for *potassium diformate (Formi LHS)* under the category "zootechnical additives", group 4(d) - "other zootechnical additives", according to the classification system of Annex I of Regulation (EC) No 1831/2003. Specifically, authorisation is sought to use Formi LHS as an additives for sows. The additive is intended to be marketed as a crystalline dry product containing 98% *potassium diformate*, 1.5% silica and water up to 0.5%.

The active agent of *Formi LHS* is *potassium diformate*. The product is intended to be incorporated into premixtures and/or complete feedingstuffs. The minimum and maximum content of *potassium diformate* in complete feedingstuffs for sows is 8000 and 12000 mg/kg, respectively.

For the determination of *potassium diformate* in the *feed additive*, the applicant proposes a method based on the quantification of total formate. The measured formate content allows the calculation of potassium diformate content in the sample. The method is based on oxidation with potassium permanganate followed by iodometric titration. The following acceptable performance characteristics for the determination of total formate content obtained from the in-house validation study were reported: - a recovery rate ranging from 99 to 101 % and a relative standard deviation of repeatability (RSD_r) of 0.1 %.

Based on acceptable performance characteristics, the proposed method is recommended for official control purposes for the determination of *potassium diformate* in *feed additives* in the frame of authorisation.

For the determination of *potassium diformate* in *premixtures* and *feedingstuffs*, the applicant proposes an ion chromatography method equipped with electrical conductivity detection (IC/ECD). The method is based on the principle that *potassium diformate* dissociates into formate under the conditions of the analysis. From the measured formate content the potassium diformate content is then calculated. On request of the CRL the applicant provided in-house validation results for the determination of *potassium diformate* in *feedingstuffs* only. As no results were reported for *premixtures* the CRL could not evaluate the suitability of the proposed method for official control purposes.

The following acceptable performance characteristics obtained from the in-house validation study were reported for *feedingstuffs*: - a limit of detection (LOD) of 100 mg/kg; - a limit of quantification (LOQ) of 500 mg/kg; - a recovery rate close to 100 %; and - RSD_r ranging from 3.2 to 3.5 %. The validation experiments were performed with a set of different feed samples covering a formate content ranging from 3600 to 10000 mg/kg. These samples were also analysed by a second independent expert laboratory and all the reported results were in good agreement. Furthermore, the validation report included summary information related to a proficiency test (PT) organised by VDLUFA in 2006. Upon request from the CRL, the organiser of the PT provided the raw data together with the statistical assessment of the trial, showing a relative standard deviation of reproducibility of 16%.

Based on the acceptable performance characteristics mentioned above, the CRL recommends the proposed method for official control purposes for the determination of *potassium diformate* in *feedingstuffs* in the frame of authorisation.

Further testing or validation is not considered necessary.

KEYWORDS

Potassium diformate, Formi LHS, zootechnical additives, sows

1. BACKGROUND

Formi LHS is a feed additive containing *potassium diformate* for which authorisation is sought under the category "zootechnical additives", functional group 4(d) "other zootechnical additives" according to the classification system of Annex I of Regulation (EC) No 1831/2003 [1].

The additive is intended to be marketed as a crystalline dry product containing 98 % *potassium diformate*, 1.5 % silica and water up to 0.5 %. The active agent of *Formi LHS* is *potassium diformate* with a purity of 98 %. The product is intended to be incorporated into premixtures and/or complete feedingstuffs to obtain a minimum and maximum *potassium diformate* content of 8000 and 12000 mg/kg of *feedingstuffs* for sows, respectively [2].

2. TERMS OF REFERENCE

In accordance with Article 5 of Regulation (EC) No 378/2005 on detailed rules for the implementation of Regulation (EC) No 1831/2003, the CRL is requested to submit a full evaluation report to the European Food Safety Authority for each application. For this particular dossier, the methods of analysis submitted in connection with *Formi LHS*, were evaluated for their suitability for official controls.

3. EVALUATION

Identification/Characterisation of the feed additive

Quantitative and qualitative composition of impurities in the additive

When required by EU legislation, analytical methods for official control of undesirable substances (e.g. arsenic and other heavy metals) in the *additive* are available at the respective Community Reference Laboratories [3].

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

The *potassium diformate* content in *feed additive* is calculated from content of total formate and controlled by determination of formic acid and/or potassium [4]. For the determination of total formate in *feed additive*, a method based on oxidation of formate ion with potassium permanganate followed by iodometric titration is proposed by applicant.

Approximately 18 ml of 1 M sodium carbonate solution and 50 ml of 0.02 M potassium permanganate solution are added to a suitable weight of *feed additive* sample. The sample is kept at 80°C for 5 minutes and during this time is shaken several times. After cooling 1 g of potassium iodide and 15 ml of 2 M sulphuric acid are added. The solution is then titrated by 0.1 M sodium thiosulfate with a starch indicator. The following acceptable performance characteristics for the determination of total formate in *feed additive* obtained from the in-house validation study were reported [5]: - a recovery rate ranging from 99 to 101 %; and a seemingly under estimated RSD_r value of 0.1 %.

Based on acceptable performance characteristics, the CRL recommends the proposed method for official control purposes for the determination of *potassium diformate* in *feed additives* in the frame of authorisation.

For the determination of *potassium diformate* in premixtures and feedingstuffs, the applicant suggested IC/ECD method [6]. Approximately 1 g of sample is extracted with 80 ml of water for 30 minutes and then filled up to 100 ml. After filtration through the paper and membrane filters, the solution is injected into the ion chromatograph. External standard calibration is used for the quantification of the formate content. The measured formate content allows the calculation of the potassium diformate one.

On request of the CRL the applicant provided in-house validation results for the determination of *potassium diformate* in feedingstuffs only. As no results were reported for *premixtures* the CRL could not evaluate the suitability of the proposed method for official control purposes.

The following acceptable performance characteristics obtained from the in-house validation study on *feedingstuffs* were reported [6]: - LOD = 100 mg/kg; - LOQ = 500 mg/kg; - a recovery rate close to 100 %; and RSD_r ranging from 3.2 to 3.5 %.

The validation experiments were performed with a set of different feed samples covering a formate content ranging from 3600 to 10000 mg/kg. These samples were also analysed by a second independent expert laboratory and all the reported results were in good agreement. Furthermore, the validation report includes summary information related to a proficiency test (PT) organised by VDLUFA in 2006. Upon request from the CRL, the organiser of the PT provided the raw data together with the statistical assessment of the trial, showing a relative standard deviation of reproducibility of 16% [7].

Based on the acceptable performance characteristics mentioned above, the CRL recommends the proposed method for official control purposes for the determination of *potassium diformate* in *feedingstuffs* in the frame of authorisation.

Further testing or validation is not considered necessary.

4. CONCLUSIONS AND RECOMMENDATIONS

In the frame of the *Formi LHS* authorisation the CRL recommends the in-house validated methods suggested by applicant for official control purposes in *feed additive* and *feedingstuffs*.

Further testing or validation by the CRL is not considered necessary.

Recommended text for the register entry, fourth column (Composition, chemical formula, description, analytical method)

- Determination of the active substances in the *feed additives*: oxidation-reduction titration for determination of *potassium diformate*.
- Determination of the active substances in the *feedingstuffs*: ion chromatography method equipped with electrical conductivity detection (IC/ECD).

5. DOCUMENTATION AND SAMPLES PROVIDED TO CRL

In accordance with the requirements of Regulation (EC) No 1831/2003, reference samples of have been sent to the Community Reference Laboratory for Feed Additives Authorisation. The dossier has been made available to the CRL by EFSA.

6. REFERENCES

- [1] * Reference SANCO/D/2 Forw. Appl. 1831/027-2008.
- [2] * Annex III: Proposal for Registry Entry
- [3] 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 Community Reference Laboratories, Official Journal of the European Union L 136. 24.5.2006.
- [4] * Annex Section II-25_apaa55e_ Determination of potassium diformate
- [5] * Annex Section II-28_Evaluation of analytical methods
- [6] * Supplementary information - Ameisensaeuremethode SKMBT_C25009042014310

[7] * Supplementary information – "Ring trial ameisensaure", provided by J. Danier

* Refers to Dossier No: FAD-2008-0044.

7. RAPPORTEUR LABORATORY

The Rapporteur Laboratory for this evaluation was the Community Reference Laboratory (CRL-FA).

8. ACKNOWLEDGEMENTS

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- Plantedirektoratets Laboratorium, Lyngby, Danmark
- RIKILT-Instituut voor Voedselveiligheid, Wageningen, Nederland
- Państwowy Instytut Weterynaryjny, Puławy, Polska
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- Sächsische Landesanstalt für Landwirtschaft, Fachbereich 8 — Landwirtschaftliches Untersuchungswesen, Leipzig, Deutschland
- Thüringer Landesanstalt für Landwirtschaft (TLL), Abteilung Untersuchungswesen. Jena, Deutschland

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