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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

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(CRL-FA)

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## 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<sub>r</sub>) 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<sub>r</sub> 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<sub>r</sub> 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<sub>r</sub> 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 - Ameisensaeuemethode 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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- RIKILT-Instituut voor Voedselveiligheid, Wageningen, Nederland
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