



Subject: Addendum to the EURL evaluation report

References:

FAD-2010-0188 + FAD-2010-0303 + FAD-2010-0312 (*ammonium formate, sodium formate, calcium formate and potassium diformate*) – JRC.DG.D.6/CvH/ZE/mdS/Ares(2011)1267744

Upon the recent publication of new ring-trial validated methods EN 17294 [1] and EN 17298 [2] 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 [3] considered appropriate to perform a new evaluation of the methods of analysis for official control of *ammonium formate, sodium formate, calcium formate and potassium diformate* in the *feed additives, premixtures, feedingstuffs and water* in the frame of the above-mentioned *feed additive* dossiers. 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 [4], the EURL also updates in this amendment the relevant methods for the metals (*sodium, calcium and potassium*) and *ammonium*.

For the determination of *ammonium formate, sodium formate, calcium formate and potassium diformate* (as total *formic acid*) in the *feed additives, premixtures, feedingstuffs and water* the EURL evaluated ring-trial validated EN 17294 method based on ion chromatography coupled to conductivity detection (IC-CD) [1]. 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, compound feed and water [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 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 total *formic acid* in *premixtures*, *feedingstuffs* (feed material, complimentary feed and compound feed) and *water* are presented in Table 1. In addition, a limit of quantification (LOQ) of 200 mg for *formic acid* acid /kg *feedingstuffs* is reported [1].

Based on the performance characteristics available 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 *ammonium formate*, *sodium formate*, *calcium formate* and *potassium diformate* (as total *formic acid*) in the *feed additives*, *premixtures*, *feedingstuffs* and *water*.

For the determination of total *sodium*, *calcium*, *potassium* and *ammonium* in the *feed additives* (*sodium formate*, *calcium formate*, *potassium diformate* and *ammonium formate*) the EURL previously evaluated and recommended for official control [5] the three ring-trial validated methods, namely i) EN ISO 6869 based on atomic absorption spectrometry (AAS) [6]; ii) EN15510 based on inductively coupled plasma-atomic emission spectrometry (ICP-AES) [7]; and iii) ISO 5664 based on distillation and titration of ammonia, respectively [8]. These recommendations are also valid in the frame of this amendment.

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

	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*.

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

For the determination of *ammonium formate*, *sodium formate*, *calcium formate* and *potassium diformate* (as total formic acid) in the *feed additives* (*ammonium formate*, *sodium formate*, *calcium formate* and *potassium diformate*), *premixtures*, *feedingstuffs* and *water*:

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

For the determination of total *sodium*, *calcium* and *potassium* in the *feed additives* (*sodium formate*, *calcium formate* and *potassium diformate*):

- Atomic absorption spectrometry (AAS) – EN ISO 6869; or
- Inductively coupled plasma-atomic emission spectrometry (ICP-AES) – EN15510

For the determination of total *ammonium* in the *feed additive* (*ammonium formate*):

- Distillation and titration – ISO 5664

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] EN 17298 Animal feeding stuffs: Methods of sampling and analysis – Determination of benzoic and and sorbic acid by High Performance Liquid Chromatography (HPLC)
- [3] 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
- [4] 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
- [5] EURL evaluation report:
<https://ec.europa.eu/jrc/sites/jrcsh/files/FinRep-FormateGroup.pdf>
- [6] 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
- [7] 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
- [8] ISO 5664:1984 Water quality - Determination of ammonium - Distillation and titration method

Addendum

- Prepared by Zigmas Ezerkis

- Reviewed and approved by María José González de la Huebra and Christoph von Holst (EURL-FA), respectively, Geel, 27/05/2021



JRC.DG.D.6/CvH/ZE/mds/ARES(2011)1267744

**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-2010-0188 - CRL/100246
FAD-2010-0303 - CRL/100200
FAD-2010-0312 - CRL/100045
to be "grouped" with the previous report
FAD-2009-0027 - CRL/090012

Name of Feed Additive: -

Active Substance(s): Ammonium formate (E295)
Sodium formate (E237)
Potassium diformate (237a)
Calcium formate (E238)

Rapporteur Laboratory: European Union Reference Laboratory
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Date: 25/11/2011

EXECUTIVE SUMMARY

In the current application authorisation is sought under articles 4(1)¹ and 10(2)^{1,2,3} for *ammonium formate* (E295)¹, *sodium formate* (E237)¹, *calcium formate* (E238)^{1,2} and *potassium diformate* (237a)³ under the category of "technological additives" functional group 1(a) "preservatives" (for all) and 1(k) "silage additives" (not for *calcium formate* and *potassium diformate*) according to the classification system of Annex I of Regulation (EC) No 1831/2003.

Ammonium formate (E295) is a liquid consisting of a minimum of 35 % of ammonium formate and a maximum of 64 % of free formic acid, the rest being water. *Sodium formate* (E237) is either a solid with a minimum purity of 98 % or a liquid containing a minimum of 15 % of sodium formate and a maximum of 75 % of free formic acid, the rest being water. Authorisation is sought for the use of these two *feed additives* for all animal species and categories. Both *feed additives* are intended to be used in *premixtures*, *feedingstuffs*, *water* and *silage*. The Applicant proposes a maximum concentration (expressed as formic acid) of 5 g/L in *water*, 10 g/kg in *silage* and 20 g/kg in *feedingstuffs*.

Calcium formate (E238) is a colourless solid with a minimum purity of 98 %. Authorisation is sought for the use of the *feed additive* for all animal species and categories. The *feed additive* is intended to be used in *premixtures* and *feedingstuffs*. The Applicant proposes a maximum concentration (expressed as formic acid) of 30 g/kg in *feedingstuffs*.

Finally, *potassium diformate* (237a) is a colourless aqueous solution with a minimum of 50 % of potassium diformate in water. Authorisation is sought for the use of the *feed additive* for all animal species and categories. The *feed additive* is intended to be used in *premixtures* and *feedingstuffs*. The Applicant proposes a maximum concentration (expressed as formic acid) of 9, 18 and 12 g/kg in fish for feed, in *feedingstuffs* for weaned piglets and sows & pigs for fattening, respectively.

The following international ring-trial validated standards were submitted by the Applicants and/or identified by the EURL:

- EN ISO 6869 and EN 15510, based on atomic absorption spectrometry (AAS) and inductively coupled plasma atomic emission spectrometry (ICP-AES), respectively, for the determination of total calcium, potassium and sodium in the *feed additives* (*calcium formate* - E238; *potassium diformate* - 237a ; *sodium formate* - E237);
- ISO 5664, based on the distillation & titration of ammonia and the Community method (R152/2009, Annex III, C) based on Kjeldahl for the determination of nitrogen

¹ FAD-2010-0312; ² FAD-2010-0303; ³ FAD-2010-0188

and calculation the ammonium content in the *feed additive* from the measured nitrogen content (*ammonium formate* - E295);

- EN 15909, based on the EDTA complexometric reaction for the determination of total calcium in the *feed additive* (*calcium formate* - E238); and
- EN 15909, based on reverse phase high performance liquid chromatography with UV detection (RP-HPLC-UV) at 214 nm, for the determination of total formate in all the *feed additives* of concern (E295, E237, 237a and E238).

The EURL recommends for official control all the above mentioned methods for the determination of *feed additives* of concern. The relevant details about the principles of analysis and the corresponding performance characteristics are provided in the report.

For the determination of total formate (originating from *ammonium, sodium, calcium formates* and *potassium diformate*) in *premixtures* and *feedingstuffs* the Applicant¹ proposed a single laboratory validated method based on ion-exclusion high performance liquid chromatography with UV or refractive index detection (HPLC-UV/RI). This method does not distinguish between *formic acid* and its salts. The following performance characteristics for the quantification of total formate (expressed as total formic acid), were derived from the single-laboratory validation study in *premixtures* and *feedingstuffs*: - a relative standard deviations for *repeatability* (RSD_r) ranging from 1.5 to 6.5 % for the concentration ranging from 1 to 1000 g/kg; - a *recovery rate* (R_{rec}) ranging from 89 to 99 %; and - a limit of quantification (LOQ) of 90 mg *formic acid*/kg *feedingstuffs*. The HPLC-UV/RI method was further ring trial validated with three to five laboratories and a relative standard deviation for *reproducibility* (RSD_R) ranging from 6.6 to 19.3 % was determined for *premixtures* and *feedingstuffs* containing from 4.5 to 44 g *formic acid*/kg.

Furthermore, the EURL wishes to recall the conclusions drawn in the frame of the FAD-2009-0027 "Formic acid" dossier, dated 21/05/2010:

For the determination of *formic acid* in *feedingstuffs*, the Applicant (FAD-2009-0027) suggested a single-laboratory validated method based on ion chromatography with electrical conductivity detection (IC-ECD). Approximately 1g of sample is extracted with 80mL of water for 30 minutes and then filled up to 100 mL. After filtration through 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 formic acid one. The following performance characteristics were reported: - a limit of detection (LOD) and quantification (LOQ) of 100 and 500 mg/kg *feedingstuffs*, respectively; - a recovery rate (R_{rec}) close to 100%; and - a repeatability relative standard deviation (RSD_r) of ca. 3.5%. The validation experiments were performed

¹ FAD-2010-0312; ² FAD-2010-0303; ³ FAD-2010-0188

with a set of different feed samples covering a formate content ranging from 3.6 to 10 g/kg. These samples were also analysed by a second independent expert laboratory and all the results were in agreement. Furthermore, the validation report included summary information related to an inter-laboratory comparison organised by VDLUFA in 2006 for the determination of organic acids in *feedingstuffs*, including formic acid, to which six laboratories participated. The following performance characteristics were reported, for sample formic acid concentrations ranging from 7.2 to 506 g/kg *feedingstuffs*: - RSD_r ranging from 4 to 10 %; and - a reproducibility relative standard deviation (RSD_R) ranging from 13 to 22 %.

Based on the performance characteristics presented, the EURL recommends for official control the ring trial validated and submitted by Applicant¹ method which is based on ion-exclusion HPLC-UV/RI to determine total formate (expressed as total formic acid) in *premixtures* and *feedingstuffs* containing *ammonium formate*, *sodium formate*, *calcium formate* or *potassium diformate*. In addition, the EURL recommends for official control the IC-ECD method submitted by the Applicant (FAD-2009-0027) for the determination of formic acid (and total formate) in *feedingstuffs*. Furthermore, the EURL considers that maximum concentration levels (5g/kg) of *sodium* and *ammonium formates* in *water* could be monitored using the two methods (HPLC-UV/RI or IC-ECD) recommended above.

None of the Applicants provided analytical method or experimental data for the quantification of *ammonium formate* and *sodium formate* in *silage*. Therefore, the EURL cannot evaluate nor recommend any method for official control to determine *ammonium formate* and *sodium formate* in *silage*.

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.

The conclusions/recommendations presented in this report for the various *formate* salts are to be combined with those presented in the FAD-2009-0027 for the *formic acid*.

KEYWORDS

Ammonium formate, sodium formate, calcium formate, potassium diformate, technological additives, preservatives, silage additives, all animal species and categories.

¹ FAD-2010-0312; ² FAD-2010-0303; ³ FAD-2010-0188

1. BACKGROUND

In the current application authorisation is sought under articles 4(1)¹ and 10(2)^{1,2,3} for *ammonium formate* (E295)¹, *sodium formate* (E237)¹, *calcium formate* (E238)^{1,2} and *potassium diformate* (237a)³ under the category of "technological additives" functional group 1(a) "preservatives" and 1(k) "silage additives" (except for *calcium formate* and *potassium diformate*) according to the classification system of Annex I of Regulation (EC) No 1831/2003 [1-3].

Ammonium formate (E295) is a liquid consisting of a minimum of 35 % of ammonium formate and a maximum of 64 % of free formic acid, the rest being water [4, 5]. *Sodium formate* (E237) is either a solid with a minimum purity of 98 % or a liquid containing a minimum of 15 % of sodium formate and a maximum of 75 % of free formic acid, the rest being water [4, 5]. Authorisation is sought for the use of these two *feed additives* for all animal species and categories [1, 4]. Both *feed additives* are intended to be used in *premixtures*, *feedingstuffs*, *water* and *silage* [4, 5]. The Applicant proposes a maximum concentration (expressed as formic acid) of 5 g/L in *water*, 10 g/kg in *silage* and 20 g/kg in *feedingstuffs* [4, 5].

Calcium formate (E238) is a colourless solid with a minimum purity of 98 % [4, 6]. Authorisation is sought for the use of the *feed additive* for all animal species and categories [1, 2]. The *feed additive* is intended to be used in *premixtures* and *feedingstuffs* [5, 7]. The Applicant proposes a maximum concentration (expressed as formic acid) of 30 g/kg in *feedingstuffs* [6].

Potassium diformate (237a) is a colourless aqueous solution with a minimum of 50 % of potassium diformate in *water* [8, 9]. Authorisation is sought for the use of the *feed additive* for all animal species and categories [3, 8]. The *feed additive* is intended to be used in *premixtures* and *feedingstuffs* [8, 9]. The Applicant proposes a maximum concentration (expressed as formic acid) of 9, 18 and 12 g/kg in fish for feed, in *feedingstuffs* for weaned piglets and sows & pigs for fattening, respectively [8, 9].

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

¹ FAD-2010-0312; ² FAD-2010-0303; ³ FAD-2010-0188

Safety Authority for each application or group of applications. For this particular dossier, the methods of analysis submitted in connection with *ammonium formate*, *sodium formate*, *calcium formate* and *potassium diformate* 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 and dioxins) are available from the respective European Union Reference Laboratories [10].

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

For the determination of total calcium, potassium and sodium in the *feed additives* (*calcium formate*, *potassium diformate* and *sodium formate*), EURL identified two ring-trial validated methods - EN ISO 6869:2000, based on atomic absorption spectrometry (AAS) after dilution in hydrochloric acid [11], and - EN 15510:2007, based on inductively coupled plasma atomic emission spectrometry (ICP-AES) after dilution in hydrochloric acid [12]. Relative standard deviation for *repeatability* (RSD_T) and for *reproducibility* (RSD_R) ranging from 2 to 8 % were reported [11, 12].

For the determination of ammonium in the *feed additive*, EURL identified the ring-trial validated method (ISO 5664:1984) based on distillation and titration of ammonia [13]. RSD_R ranging from 1.4 to 9 % was reported, for the concentration ranging from 2.3 to 51.5 mg/l for *ammonium* [13]. Furthermore, the community method (R152/2009, Annex III, C) for the determination of nitrogen content could be used for the quantification of ammonium in the feed additive.

For the quantification of calcium formate in the *feed additive* the Applicant² submitted the CEN ring-trial validated method (EN 15909:2010) [14], in which:

- the total calcium content in the *feed additive* is determined complexometrically using 0.1 M EDTA (ethylenedinitrilotetraacetic acid disodium salt dehydrate) in alkaline media in the presence of calconcarboxylic acid, a calcium-specific indicator;

¹ FAD-2010-0312; ² FAD-2010-0303; ³ FAD-2010-0188

- the total *formate* content in the *feed additive* is determined isocratically by reverse phase high performance liquid chromatography with UV detection (RP-HPLC-UV) at 214 nm. Quantification is performed by using external calibration with standard solutions.

The following performance characteristics are reported [14]: RSD_r of 0.3 %* and 0.7 %** ; and RSD_R of 0.8 %* and 2.2 %** [for (*) *calcium* and (**) *formate*, respectively]. The EURL considers that this CEN method is also applicable for the determination of total *formate* in the other feed additives of concern.

Based on the performance characteristics presented, the EURL recommends for official control all the above mentioned international standard CEN and ISO methods for the determination of total *sodium*, *potassium*, *ammonium*, *calcium* and total *formate* (including *diformate*) in the various *feed additives* of concern.

For the determination of total formate (originating from *ammonium*, *sodium*, *calcium formates* and *potassium diformate*) in *premixtures* and *feedingstuffs* the Applicant¹ proposed a single laboratory validated method based on ion-exclusion high performance liquid chromatography with UV or refractive index detection (HPLC-UV/RI) [15]. This method does not distinguish between *formic acid* and its salts.

The sample is extracted with 0.005 M sulphuric acid at a pH ranging from 2 to 3.5. The solution is then centrifuged or filtered and used for the HPLC measurement. After ion-exclusion chromatography, *formate* is quantified as *formic acid* by spectrophotometry at 217 nm or by the refractive index, using external calibration.

The following performance characteristics for the quantification of total formate (expressed as total formic acid), were derived from the single-laboratory validation study in *premixtures* and *feedingstuffs* [15]:

- a relative standard deviations for *repeatability* (RSD_r) ranging from 1.5 to 6.5 % for the concentration ranging from 1 to 1000 g/kg;
- a *recovery* rate (R_{rec}) ranging from 89 to 99 %; and
- a limit of quantification (LOQ) of 90 mg *formic acid*/kg *feedingstuffs*.

The HPLC-UV/RI method was further ring trial validated with five laboratories and a relative standard deviation for *reproducibility* (RSD_R) ranging from 6.6 to 19.3 % was determined for *premixtures* and *feedingstuffs* containing from 4.5 to 44 g *formic acid*/kg [15].

Furthermore, the EURL wishes to recall the conclusions drawn in the frame of the FAD-2009-0027 "Formic acid" dossier, dated 21/05/2010 [16]:

¹ FAD-2010-0312; ² FAD-2010-0303; ³ FAD-2010-0188

For the determination of *formic acid* in *feedingstuffs*, the Applicant (FAD-2009-0027) suggested a single-laboratory validated method based on ion chromatography with electrical conductivity detection (IC-ECD). Approximately 1g of sample is extracted with 80mL of water for 30 minutes and then filled up to 100 mL. After filtration through 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 formic acid one. The following performance characteristics were reported: - a limit of detection (LOD) and quantification (LOQ) of 100 and 500 mg/kg *feedingstuffs*, respectively; - a recovery rate (R_{Rec}) close to 100%; and - a repeatability relative standard deviation (RSD_r) of ca. 3.5%. The validation experiments were performed with a set of different feed samples covering a formate content ranging from 3.6 to 10 g/kg. These samples were also analysed by a second independent expert laboratory and all the results were in agreement. Furthermore, the validation report included summary information related to an inter-laboratory comparison organised by VDLUFA in 2006 for the determination of organic acids in *feedingstuffs*, including formic acid, to which six laboratories participated. The following performance characteristics were reported, for sample formic acid concentrations ranging from 7.2 to 506 g/kg *feedingstuffs*: - RSD_r ranging from 4 to 10 %; and - a reproducibility relative standard deviation (RSD_R) ranging from 13 to 22 %.

Based on the performance characteristics presented, the EURL recommends for official control the ring trial validated and submitted by Applicant¹ method which is based on ion-exclusion HPLC-UV/RI to determine total formate (expressed as total formic acid) in *premixtures* and *feedingstuffs* containing *ammonium formate*, *sodium formate*, *calcium formate* or *potassium diformate*. In addition, the EURL recommends for official control the IC-ECD method submitted by the Applicant (FAD-2009-0027) for the determination of formic acid (and total formate) in *feedingstuffs*.

Furthermore, the EURL considers that maximum concentration levels (5 g/kg) of *sodium* and *ammonium formates* in *water* could be monitored using the two methods (HPLC-UV/RI or IC-ECD) recommended above.

None of the Applicants provided analytical method or experimental data for the quantification of *ammonium formate* and *sodium formate* in *silage*. Therefore, the EURL cannot evaluate nor recommend any method for official control to determine *ammonium formate* and *sodium formate* in *silage*.

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.

¹ FAD-2010-0312; ² FAD-2010-0303; ³ FAD-2010-0188

4. CONCLUSIONS AND RECOMMENDATIONS

In the frame of this authorisation the EURL recommends for official control:

- the ring trial validated CEN methods (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 total calcium, sodium and potassium in the *feed additives* (*calcium* and *sodium formates* and *potassium diformate*);
- an alternative ring-trial validated CEN method (EN 15909), based on the complexometric titration for the determination of calcium in the *feed additive* (*calcium formate*);
- the ring-trial validated ISO 5664 method, based on distillation and titration of ammonia (ISO 5664:1984), for the determination of ammonium in the *feed additive* (*ammonium formate*) together with the Community method (R152/2009, Annex III, C)
- the ring-trial validated CEN method (EN 15909), based on reverse phase HPLC-UV for determination of formate in all the *feed additives* under investigation (*ammonium*-, *calcium*-, *sodium formates* and *potassium diformate*);
- the ring trial validated method based on ion-exclusion HPLC-UV/RI (FAD 2010-0312) and the single-laboratory validated and further verified method based on IC-ECD (FAD-2010-0027) for the determination of total formate in *premixtures*, *feedingstuffs* and *water* - for the relevant *active substances*.

None of the Applicants provided analytical method or experimental data for the quantification of *ammonium formate* and *sodium formate* in *silage*. Therefore, the EURL cannot evaluate nor recommend any method for official control to determine *ammonium formate* and *sodium formate* in *silage*.

The conclusions/recommendations presented in this report for the various *formate* salts are to be combined with those presented in the FAD-2009-0027 for the *formic acid*.

Recommended text for the register entry (analytical method)

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

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

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

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- EN ISO 6869: atomic absorption spectrometry (AAS) or
EN 15510: inductively coupled plasma atomic emission spectrometry (ICP-AES); or
EN 15909: Complexometrical titration with EDTA

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

- ISO 5664: distillation and titration
- COM EC R152/2009 - Kjeldahl method

For the determination of total *formate* in the *feed additives*:

- EN 15909: reverse phase HPLC-UV

For the determination of total *formate* in *premixtures, feedingstuffs* and *water*:

- Ion-exclusion high performance liquid chromatography with UV or refractive index detection (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 samples of *ammonium formate, sodium formate, calcium formate* and *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] *Application, Reference SANCO/D/2 Forw. Appl. 1831/00151/(10179)/2010
- [2] +Application, Reference SANCO/D/2 Forw. Appl. 1831/7148-2010
- [3] ±Application, Reference SANCO/D/2 Forw. Appl. 1831/7075-2010
- [4] *Application, Proposal for Register Entry - Annex A
- [5] *Technical dossier, Section II: Identity, characterisation and conditions of use
- [6] +Application, Proposal for Register Entry - Annex A
- [7] +Technical dossier, Section II: Identity, characterisation and conditions of use
- [8] ±Application, Proposal for Register Entry - Annex A
- [9] ±Technical dossier, Section II: Identity, characterisation and conditions of use
- [10] 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

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- [11] 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
- [12] 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
- [13] ISO 5664:1984 Water quality - Determination of ammonium - Distillation and titration method
- [14] EN 15909:2010 Fertilizers - Determination of calcium and formate in calcium foliar fertilizers
- [15] *Technical dossier, Section II - Annex II_4
- [16] <http://irmm.jrc.ec.europa.eu/SiteCollectionDocuments/FinRep-FAD-2009-0027.pdf>

* Refers to Dossier No. FAD-2010-0312

+ Refers to Dossier No. FAD-2010-0303

± Refers to Dossier No. FAD-2010-0188

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

The following National Reference Laboratories contributed to this report:

- Plantedirektoratet, Laboratorium for Foder og Gødning, Lyngby (DK)
- Schwerpunktlabor Futtermittel des Bayerischen Landesamtes für Gesundheit und Lebensmittelsicherheit (LGL), Oberschleißheim (DE)
- Państwowy Instytut Weterynaryjny, Puławy (PL)
- Ústřední kontrolní a zkušební ústav zemědělský (ÚKZÚZ), Praha (CZ)
- RIKILT - Instituut voor Voedselveiligheid, Wageningen (NL)
- Laboratoire de Rennes, SCL L35, Service Commun des Laboratoires, Rennes (FR)