
Dossier related to: FAD-2010-0134
CRL/100065

Name of Product: Fumaric acid (E297)

Active Substance(s): Fumaric acid

Rapporteur Laboratory: European Union Reference Laboratory for Feed Additives (EURL-FA)
Geel, Belgium

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EXECUTIVE SUMMARY

In the current application authorisation is sought for fumaric acid (E297), under article 10, category/functional group 1(a) “technological additives/preservatives”, according to the classification system of Annex I of Regulation (EC) No 1831/2003. Fumaric acid is already authorised as feed additive under Commission Directive 70/524/EEC.

According to the Applicant, the feed additive is an odourless white crystalline powder with a minimum purity of 99.5% fumaric acid. Specifically, authorisation is sought for the use of fumaric acid as preservative for all animal species and categories. The feed additive is intended to be mixed into premixtures or feedingstuffs with a proposed maximum level of 20 g/kg in complete feedingstuff.

For the determination of fumaric acid in the feed additive, the Applicant submitted the Food Chemical Codex 7 (FCC) methods, based on infrared absorption spectrophotometry and acid/base titration. Even though no performance characteristics of these methods are provided, the EURL recommends for official control the internationally recognised FCC methods based on infrared absorption spectrophotometry and titrimetry to determine fumaric acid in the feed additive.

For the determination of fumaric acid in premixtures and feedingstuff the Applicant proposed a multi-analyte method based on High Performance Liquid Chromatography with UltraViolet detection (HPLC-UV). This method does not distinguish between fumaric acid and its salts. In addition the Applicant stated that the method is also applicable to pure acid and acid blends. The following performance characteristics for the quantification of total fumaric acid, were derived from the single-laboratory validation study:

- a relative standard deviation for repeatability (RSDr) ranging from 2.8% to 11.7%, for concentrations ranging from 0.01 to 1000 g/kg;
- a recovery rate (Rrec) ranging from 90.9 to 107.5%; and
- a limit of quantification (LOQ) of 6.5 mg/kg in feedingstuffs.

Furthermore, the method was ring trial validated with four laboratories and a relative standard deviation for reproducibility (RSDR) ranging from 1.6% to 8.1% was determined for premixtures and feedingstuffs containing 8.8 to 66 g fumaric acid/kg, respectively.

Based on the performance characteristics presented, the EURL recommends for official control the ring trial validated method based on ion-exclusion HPLC-UV to determine fumaric acid (expressed as total fumaric 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.

KEYWORDS

Fumaric acid, E297, technological additives, preservatives, all animal species and categories.

1. BACKGROUND

In the current application authorisation is sought for fumaric acid (E297), under article 10, category/functional group 1(a) “technological additives/preservatives”, according to the classification system of Annex I of Regulation (EC) No 1831/2003 [1,2]. Fumaric acid is already authorised as feed additive under Commission Directive 70/524/EEC [3].

According to the Applicant, the feed additive is an odourless white crystalline powder with a minimum purity of 99.5 % fumaric acid [3,4]. Fumaric acid is synthesised, under specific conditions, by isomerisation of an aqueous solution of maleic acid followed by several purification steps [5].

Specifically, authorisation is sought for the use of fumaric acid as preservative for all animal species and categories. The feed additive is intended to be mixed into premixtures or feedingstuffs [1,6]. Furthermore, the Applicant proposed a maximum level of 20 g/kg in complete feedingstuff [2,6].

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 fumaric acid 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, aflatoxin B1 and dioxins) are available from the respective European Union Reference Laboratories [7].

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

For the determination of fumaric acid in the feed additive, the Applicant submitted the Food Chemical Codex 7 (FCC) methods based on infrared absorption spectrophotometry and acid/base titration with 0.5 N sodium hydroxide and phenolphthalein as indicator, to determine fumaric acid in the feed additive [8,9].

Even though no performance characteristics of these methods are provided, the EURL recommends for official control the internationally recognised FCC methods based on infrared absorption spectrophotometry (for identification) and titrimetry (for quantification) to determine fumaric acid in the feed additive.

For the quantification of fumaric acid in premixtures and feedingstuffs the Applicant proposed a multi-analyte method based on High Performance Liquid Chromatography with UltraViolet detection (HPLC-UV) [10]. This method is applicable for the determination of total organic acids and their salts and therefore it does not distinguish between fumaric acid and its salts. In addition the Applicant stated that the method is also applicable to pure acid and acid blends.

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, fumaric acid is quantified by spectrophotometry at 217 nm, using external calibration.

The following performance characteristics for the quantification of total fumaric acid, were derived from the single-laboratory validation study [10]:

- a relative standard deviation for repeatability (RSDr) ranging from 2.8 % to 11.7%, for concentrations ranging from 0.01 to 1000 g/kg;
- a recovery rate (Rrec) ranging from 90.9 to 107.5%; and
- a limit of quantification (LOQ) of 6.5 mg/kg in feedingstuffs.

Furthermore, the method was ring trial validated with four laboratories and a relative standard deviation for reproducibility (RSDR) ranging from 1.6 % to 8.1 % was determined for premixtures and feedingstuffs containing 8.8 to 66 g fumaric acid/kg, respectively [10].
Based on the performance characteristics presented, the EURL recommends for official control the ring trial validated method based on ion-exclusion HPLC-UV to determine fumaric acid (expressed as total fumaric 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:

- the internationally recognised FCC method based on infrared absorption spectrophotometry and titrimetry to determine fumaric acid in the feed additive;

- the ring trial validated method based on ion-exclusion HPLC-UV to determine fumaric acid (expressed as total fumaric acid) in premixtures and feedingstuffs.

**Recommended text for the register entry (analytical method)**

For the determination of fumaric acid in the feed additive:

- infrared absorption spectrophotometry and titration with sodium hydroxide (Food Chemical Codex 7)

For the determination of fumaric acid (expressed as total fumaric acid) in premixtures and feedingstuffs:

- ion exclusion High Performance Liquid Chromatography with UV detection (HPLC-UV)

5. DOCUMENTATION AND SAMPLES PROVIDED TO EURL

In accordance with the requirements of Regulation (EC) No 1831/2003, reference samples of fumaric acid 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

[2] *Application, Proposal for Register Entry – Annex A
[4] *Technical dossier, Section II, 2.1.3. Qualitative and quantitative composition
[6] *Technical dossier, Section II, 2.5. Conditions of Use
* Refers to Dossier No. FAD-2010-0134

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

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