EUROPEAN COMMISSION
JOINT RESEARCH CENTRE
Institute for Reference Materials and Measurements
**European Union Reference Laboratory for Feed Additives**

**JRC.DG.D.6/CvH/RM/ag/ARES(2011)869041**

# 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

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| Dossier related to: | FAD-2010-0297<br>CRL/100273 |
| Feed additive: | Titanium dioxide (anatase and rutile structure) (E 171) |
| Active Substance(s): | Titanium dioxide |
| Rapporteur Laboratory: | European Union Reference Laboratory for Feed Additives (EURL-FA)<br>Geel, Belgium |
| Report prepared by: | Roberto Molteni (EURL-FA) |
| Report revised by:<br>Date: | Piotr Robouch (EURL-FA)<br>10/08/2011 |
| Report approved by:<br>Date: | Christoph von Holst<br>10/08/2011 |

## EXECUTIVE SUMMARY

In the current application authorisation is sought under articles 4(1) and 10(2) for *Titanium dioxide* in anatase and rutile structure, under the "sensory additives", functional group 2(a) "colourants", according to the classification system of Annex I of Regulation (EC) No 1831/2003. Authorisation is sought for the use of the *feed additive* for all animal species and categories. The *feed additive* is intended to be incorporated directly in *feedingstuffs*, with no recommended minimum or maximum levels.

The Applicant used X-Ray Diffraction (XRD) to identify the anatase and rutile structure in the *feed additive*. Furthermore, the Applicant proposed the internationally recognised European Pharmacopoeia method, based on a redox titration with ammonium and cerium nitrate for the determination of *Titanium dioxide* in the *feed additive*. Even though no performance characteristics are provided, the EURL recommends for official control the European Pharmacopoeia method (Ph. Eur. 7.0, method 01/2011:0150), for the identification and quantification of *Titanium dioxide* in the *feed additive*.

The Applicant did not provide any experimental method or data for the determination of *Titanium dioxide* in *premixtures* and *feedingstuffs*. However, EURL identified several analytical methods to determine *Titanium* in food and feed, based on sample digestion followed by spectrophotometry or by multi-elemental techniques, such as Inductively Couple Plasma Optical Emission Spectrometry (ICP-OES) or Mass Spectrometry (ICP-MS). The EURL considers these methods suitable for quantification of *Titanium* 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**
*Titanium dioxide*, sensory additive, colourants, all animal species and categories

## 1. BACKGROUND

In the current application authorisation is sought under articles 4(1) (new use) and 10(2) (re-evaluation of additives already authorised under the provisions of the Council Directive 70/524/EEC) for *Titanium dioxide* in anatase and rutile structure, under the "sensory additives", functional group 2(a) "colourants" [1], according to the classification system of Annex I of Regulation (EC) No 1831/2003. Authorisation is sought for the use of the *feed additive* for all animal species and categories [2].

The *feed additive* is produced by reacting titanium ores with chlorine gas or sulphuric acid, resulting in *Titanium dioxide* [3]. The Applicant states that the purity criteria set in the Commission Directive 2008/128/EC for the food additive apply to the requirement for the *feed additive* [3]. *Titanium dioxide* is intended to be incorporated directly in *feedingstuffs*, with no recommended minimum or maximum levels [2].

## 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 *Titanium dioxide in anatase and rutile structures)*, 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, mycotoxins, and dioxins) are available from the respective European Union Reference Laboratories [4].

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

For the identification of the anatase and rutile structure in the *feed additive*, the Applicant used X-Ray Diffraction (XRD) pattern [5]. For the determination of *Titanium dioxide* in the *feed additive*, the Applicant proposed the internationally recognised European Pharmacopoeia method, based on a redox titration with ammonium and cerium nitrate [6].

Even though no performance characteristics are provided, the EURL recommends for official control the European Pharmacopoeia method (Ph. Eur. 7.0, method 01/2011:0150), for the identification and quantification of *Titanium dioxide* in the *feed additive*.

The Applicant did not provide any experimental method or data for the determination of *Titanium dioxide* in *premixtures* and *feedingstuffs*. However, EURL identified several analytical methods to determine *Titanium* in food and feed [7], based on sample digestion followed by spectrophotometry or by multi-elemental techniques, such as Inductively Couple Plasma Optical Emission Spectrometry (ICP-OES) or Mass Spectrometry (ICP-MS). The EURL considers these methods suitable for quantification of *Titanium* 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 European Pharmacopoeia method – Ph. Eur. 7.0, method 01/2011:0150, based on a redox titration with ammonium and cerium nitrate for the identification and quantification of *Titanium dioxide* in the *feed additive*.

The Applicant did not provide any experimental method or data for the determination of *Titanium dioxide* in *premixtures* and *feedingstuffs.* However, EURL identified several analytical methods to determine *Titanium* in food and feed, based on spectrophotometry or ICP-OES or ICP-MS. The EURL considers these methods suitable for quantification of *Titanium* in *premixtures* and *feedingstuffs.*

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

For the quantification of *Titanium dioxide* in the *feed additive*:

- Redox titration with ammonium and cerium nitrate
  (Ph. Eur. 7.0, method 01/2011:0150)

## 5. DOCUMENTATION AND SAMPLES PROVIDED TO EURL

In accordance with the requirements of Regulation (EC) No 1831/2003, reference samples of *Titanium dioxide* 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. 1831/00175 (10381)-2010

[2] *Application, Proposal for Register Entry – Annex A

[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] *Supplementary information provided by the Applicant upon request EURL

[6] *Technical dossier, Section II – Annex II 9 TiO2 Ph.Eur. monograph

[7] Scotter M.J. 2011. Methods for the determination of European Union-permitted added natural colours in foods: a review. Food Additives and Contaminants, Vol. 28, No. 5, May, 527–596

* Refers to Dossier No. FAD-2010-0297

## 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:

- 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)
- Istituto Superiore di Sanita' - Dipartimento di Sanita' alimentare ed animale, Roma (IT)
- Instytut Zootechniki w Krakowie, Krajowe Laboratorium Pasz, Lublin (PL)