Renewal Assessment Report

under Regulation (EC) 1107/2009

Zoxamide

Volume 3

Zoxium 240 SC

B.5 Methods of analysis

Rapporteur Member State: Latvia
Co-Rapporteur Member State: France

Version history

Date	Subject
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TABLE OF CONTENTS

B.5. ANALYTICAL METHODS

B.5.1. Methods for the generation of pre-authorisation data

B.5.1.1. Analysis of the plant protection product

(a) Methods for the determination of the active substance and/or variant in the plant protection product

Reference: **CP 5.1.1/01**
Report: **Fifi, A.P. (2011), Accelerated storage stability of product Zoxium 240 SC after 2 weeks at 54°C**
Guidelines: **SANCO/3030/99 rev. 4, EU Directive 91/414/EEC**
GLP: **Yes**

Principle of the method
Samples (290g) are added to a 100 mL graduated flask, dissolved and made up to volume with acetonitrile/ water, 70/30, v/v. The samples are syringe filtered (0.2 µm) and an aliquot (1 mL) is diluted with acetonitrile/ water, 70/30, v/v (9 mL). The samples are analysed by high performance liquid chromatography with ultra-violet detection (HPLC-UV) at 210 nm, using a Zorbax Eclipse Plus C_{18} column (100 x 2.1 mm, 1.8µm) and isocratic elution with a mobile phase of acetonitrile/ water (70/30). Quantification was performed using external standards.

Specificity
No significant interferences were observed at the retention time of the active substance.
Analyte identity was confirmed by retention match with an analytical standard and by comparison of UV spectra.

Linearity
Linearity of detector response was demonstrated for zoxamide using three concentrations (in triplicate) of reference standard across the concentration range of 25 mg/L to 101 mg/L, with a coefficient of determination (R^2) of 0.9999 (slope = 127.82, intercept = 162.81).

Precision (Repeatability)
Repeatability data was generated from five samples of Zoxium 240 SC. The relative standard deviation (RSD) obtained was within the guideline requirements and the results are presented in Table B.5.1.1-1.

Accuracy (Recovery)
Recovery data was generated using a standard addition procedure using five samples of Zoxium 240 SC which had been fortified with a known amount of zoxamide. The mean percentage recovery was within the guideline requirements and the results are presented in Table B.5.1.1-2 below.

Conclusion
The analytical procedure has been successfully validated in terms of specificity, linearity, precision and accuracy in accordance with all of the requirements of SANCO/3029/99 rev. 4, 11/07/2000.

Table B.5.1.1-1: Precision Data for Zoxamide

Sample	Analysed Conc. (% w/w)	Mean Conc. (% w/w)	RSD (%)	Acceptable RSD (%)
Fresh A	22.41	22.57	0.93	< 1.69
Fresh B	22.42			
Fresh C	22.45			
Fresh D	22.68			
Fresh E	22.89			

Table B.5.1.1-2: Accuracy Data for Zoxamide

Sample	Recovery (%)	Mean Recovery (%)	Acceptable Recovery (%)	RSD (%)
Recovery A	101.46	101.62	98 – 102	0.10
Recovery B	101.68			
Recovery C	101.62			
Recovery D	101.72			
Recovery E	101.63			

RMS comments and conclusion:
The method is acceptable for determination of zoxamide in the product Zoxium 240 SC.

(b) Methods for determination of relevant impurities identified in the technical material or which may be formed during manufacture of the plant protection product or from degradation of the plant protection product during storage

Not relevant, as neither zoxamide nor Zoxium 240 SC contains relevant impurities.

(c) Methods for the determination of relevant co-formulants or components of co-formulants, where required by the national competent authorities

Not relevant, as Zoxium 240 SC contains no relevant co-formulants or relevant components of co-formulants.

Methods for the determination of residues

a) **Methods In soil, water, sediment, air and any additional matrices used in support of environmental fate studies**

Please refer to Volume 3, B.3, CA, Point B.5.1.2 a.

b) Methods in soil, water and any additional matrices used in support of efficacy studies

Please refer to Volume 3, B.3, CA, Point B. 5.1.2 b.

c) Methods in feed, body fluids and tissues, air and any additional matrices used in support of toxicological studies

Please refer to Volume 3, B.3, CA, Point B 5.1.2 c.

d) Methods in body fluids, air, and any additional matrices used in support of operator, worker, resident and bystander exposure studies

Please refer to Volume 3, B.3, CA, Point B 5.1.2 d.

e) Methods in or on plants, plant products, processed food commodities, food of plant and animal origin, feed and any additional matrices used in support of residues studies

Please refer to Volume 3, B.3, CA, Point 5.1.2 e.

f) Methods in soil, water, sediment, feed and any additional matrices used in support of ecotoxicology studies

Please refer to Volume 3, B.3, CA, Point B 5.1.2 f.

g) Methods in water, buffer solutions, organic solvents and any additional matrices resulting from the physical and chemical properties tests

Please refer to Volume 3, B.3, CA, Point B.5.1.2 g.

Methods for Post-Authorisation Control and Monitoring Purposes

Please refer to Volume 3, B.3, CA, Point B. 5.2a-d.

B.5.2. References relied on

New studies

Data point	Author(s)	Year	Title Source (where different from company) Company, Report No GLP or GEP status (where relevant), Published or not	Vertebrate study Y/N	Data protection claimed (Y/N)	Justification if data protection claimed	Owner
KCP 5.1.1	Fifi A.P.	2011	Accelerated storage stability of product Zoxium 240 SC after 2 weeks at 54°C BioTecnologie BT SrL c/o Parco Tecnologico Agroalimentare dell'Umbria Report No. BT118/10 GLP, Not published	N	Y	Data to support new representative formulation	Gowan