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ANNEX

PART A

CHEMICAL PLANT PROTECTION PRODUCTS

SECTION 5

Analytical methods

Introduction

The provisions of this Section cover analytical methods used for the generation of pre-authorisation data and required for post-authorisation control and monitoring purposes.

Descriptions of methods shall be provided and include details of equipment, materials and conditions used.

On request, the following shall be provided:

- (a) analytical standards of the purified active substance and of the plant protection product;
- (b) samples of the active substance as manufactured;
- (c) analytical standards of relevant metabolites and all other components included in all monitoring residue definitions;
- (d) samples of reference substances for the relevant impurities.

In addition, the standards referred to in points (a) and (c) shall, where possible, be made commercially available and, on request, the distributing company shall be named.

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

5.1.1. Methods for the analysis of the plant protection product

Methods shall be provided, with a full description, for the determination of:

- (a) active substance and/or variant in the plant protection product;
- (b) 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;
- (c) relevant co-formulants or components of co-formulants, where required by the national competent authorities.

In the case of a plant protection product containing more than one active substance and/or variant a method capable of determining each, in the presence of the other, shall be provided. If a combined method is not submitted, the technical reasons shall be stated.

The applicability of CIPAC methods shall be assessed and reported. In case of use of a CIPAC method, further validation data shall not be required, but example chromatograms shall be submitted, where available.

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The specificity of the methods shall be determined and reported. In addition, the extent of interference by other substances present in the plant protection product (such as impurities or co-formulants), shall be determined.

The linearity of methods shall be determined and reported. The calibration range shall extend (by at least 20 %) beyond the highest and lowest nominal content of the analyte in relevant analytical solutions. Either duplicate determinations at three or more concentrations or single determinations at five or more concentrations shall be made. The equation of the calibration line and the correlation coefficient shall be reported and a typical calibration plot shall be submitted. In cases where a non-linear response is used, this shall be justified by the applicant.

The precision (repeatability) of the methods shall be determined and reported. A minimum of five replicate sample determinations shall be made, and the mean, the relative standard deviation and the number of determinations shall be reported. The accuracy of the methods shall be determined on at least two representative samples at levels appropriate to the material specification. The mean and the relative standard deviation of the recoveries shall be reported.

For relevant impurities and, where necessary, for relevant co-formulants the limit of quantification (LOQ) shall be determined and reported and shall be at a concentration of analyte, which is of toxicological or environmental significance, or at the concentration which is formed during storage of the product, where relevant.

5.1.2. *Methods for the determination of residues*

Methods shall be submitted, with a full description, for the determination of non-isotope-labelled residues in all areas of the dossier, as set out in detail in the following points:

- (a) in soil, water, sediment, air and any additional matrices used in support of environmental fate studies;
- (b) in soil, water and any additional matrices used in support of efficacy studies;
- (c) in feed, body fluids and tissues, air and any additional matrices used in support of toxicology studies;
- (d) in body fluids, air and any additional matrices used in support of operator, worker, resident and bystander exposure studies;
- (e) 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;
- (f) in soil, water, sediment, feed and any additional matrices used in support of ecotoxicology studies;
- (g) in water, buffer solutions, organic solvents and any additional matrices resulting from the physical and chemical properties tests.

The specificity of the methods shall be determined and reported. Validated confirmatory methods shall be submitted if appropriate.

The linearity, recovery and precision (repeatability) of methods shall be determined and reported.

Data shall be generated at the LOQ and either the likely residue levels or ten times the LOQ. The LOQ shall be determined and reported for each component in the residue definition.

5.2. **Methods for post-authorisation control and monitoring purposes**

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As far as practicable these methods shall employ the simplest approach, involve the minimum cost, and require commonly available equipment.

Analytical methods for the determination of the active substance and relevant impurities in the plant protection product shall be submitted, unless the applicant shows that these methods already submitted in accordance with the requirements set out in point 5.1.1 can be applied.

The provisions set out in point 5.1.1 shall apply.

Methods, with a full description, shall be submitted for the determination of residues:

- in or on plants, plant products, processed food commodities, food and feed of plant and animal origin,
- in body fluids and tissues,
- in soil,
- in water,
- in air, unless the applicant shows that exposure of operators, workers, residents or bystanders is negligible.

The applicant may deviate from such requirement by showing that the methods submitted in accordance with the requirements set out in point 4.2 of Part A of the Annex to Regulation (EU) No 283/2013 can be applied.

The specificity of the methods shall enable all components included in the monitoring residue definition to be determined. Validated confirmatory methods shall be submitted if appropriate.

The linearity, recovery and precision (repeatability) of methods shall be determined and reported.

Data shall be generated at the LOQ and either the likely residue levels or ten times the LOQ. The LOQ shall be determined and reported for each component included in the monitoring residue definition.

For residues in or on food and feed of plant and animal origin and residues in drinking water, the reproducibility of the method shall be determined by means of an independent laboratory validation (ILV) and reported.

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Changes and effects yet to be applied to the whole legislation item and associated provisions

- Signature words omitted by [S.I. 2019/556 reg. 22\(4\)](#)
- Annex Pt. B s. 11 words omitted by [S.I. 2019/556 reg. 22\(5\)\(c\)\(v\)](#)
- Art. 1(1) Art. 1 renumbered as Art. 1(1) by [S.I. 2019/556 reg. 22\(2\)\(a\)](#)
- Art. 1(2) inserted by [S.I. 2019/556 reg. 22\(2\)\(b\)](#)