



ÚSTREDNÝ KONTROLNÝ A SKÚŠOBNÝ ÚSTAV POĽNOHOSPODÁRSKY V BRATISLAVE



# Zdravá pôda, Zdravé rastliny, Zdravý život Healthy soil, Healthy plants, Healthy life

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CEUREG FÓRUM XXIV, Mojmírovce, SLOVAK REPUBLIC

23<sup>th</sup> – 24<sup>th</sup> September 2024



#### The role of the CCTIA

**Through performance** 

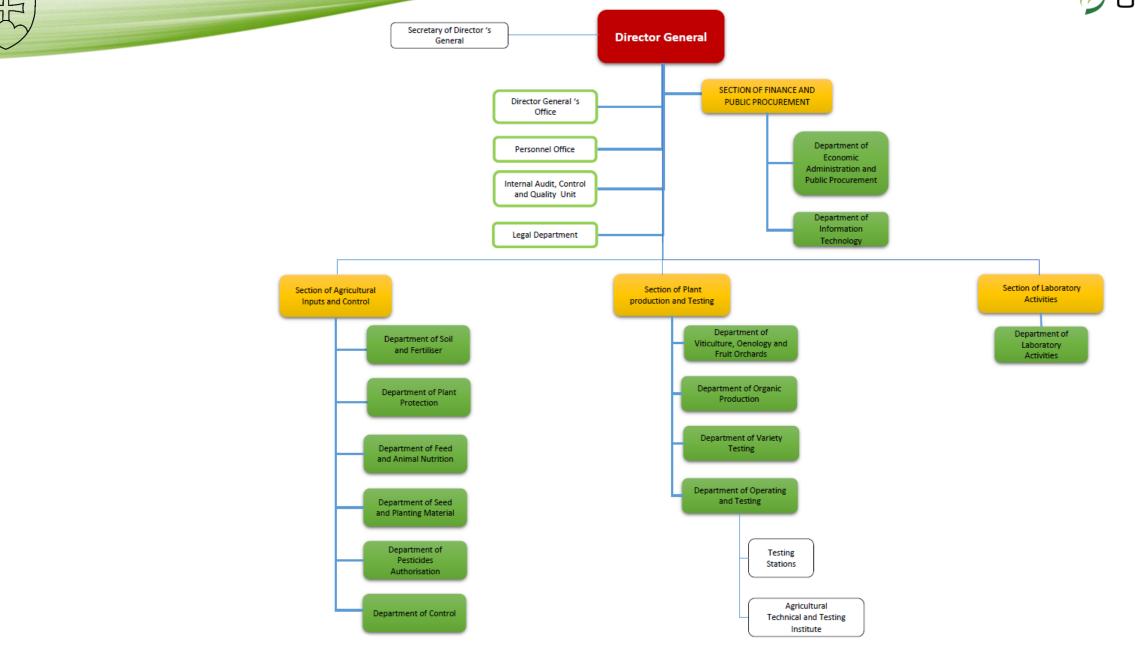
#### of official control and other official activities, state professional control, testing, laboratory activities and certification

inputs into agricultural primary production and in the field of viticulture, winemaking, fruit growing, organic agricultural production and application equipment

ensure safe sources for food and feed production and thereby protect the health of people, animals and the environment









#### **Activity of CCTIA**

#### **Department of Plants Protection**

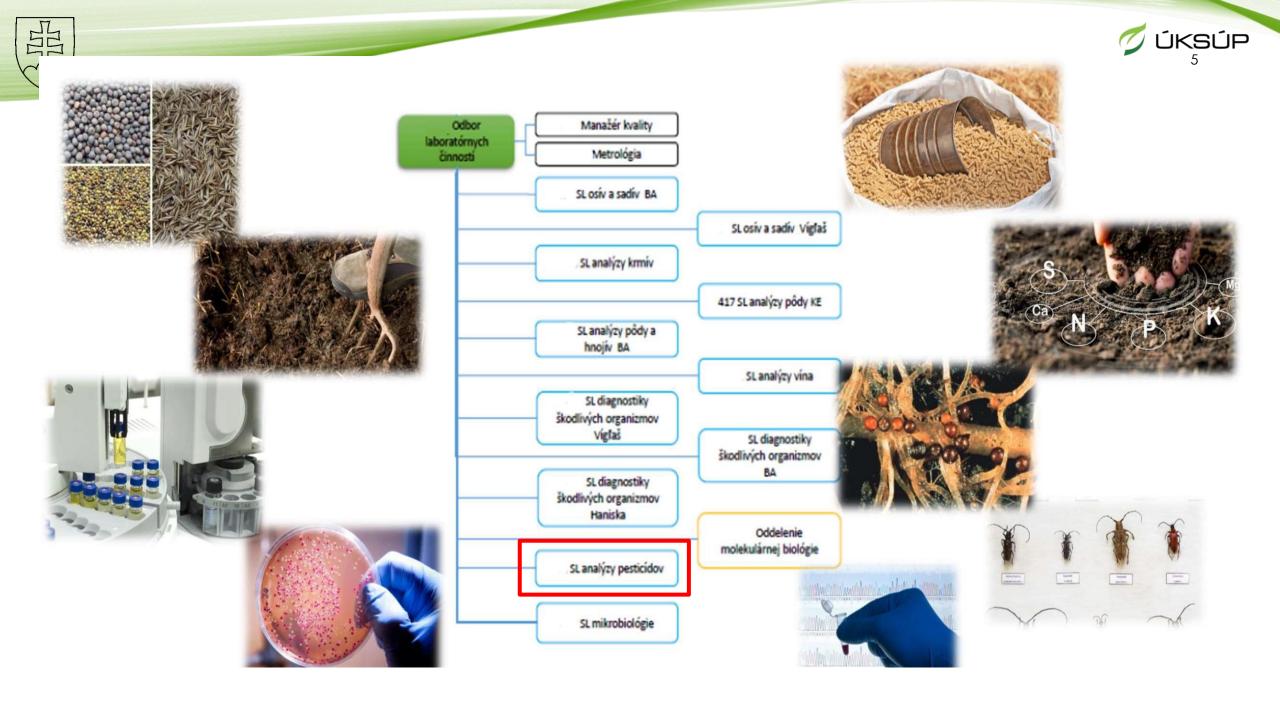


- Keeps a register of professional operators with plants, plant protection products (PPPs) and other items
- Official controls on registered professional operators
- Official controls of quarantine stations and isolation facilities
- Control of imports and exports of plants, plant products and other objects
- Control of illegally grown poppy plants and hemp plants (cannabis sativa etc.)
- Performs official control of PPPs
- PPPs trainings
- Professional trainings in the field of PPPs

#### **Department of Pesticides Authorisation**



- Evaluation of active substances, safeners, synergents, adjuvants or basic substances
- Issuing decisions on the authorisation of PPPs
- Evaluation and testing of biological efficiency of PPPs
- Keeping and processing of sales data for PPPs under the EU Regulation for statistics





#### **Quality systems**





MINISTERSTVO PODHOSPODÁRSTVA A ROZVOJA VIDIEKA SLOVENSKEJ REPUBLIKY

> receiptor processing estimates a regergie with Sciencerulus) regulation count direct

> > V Bratislave 13.06.2023 Č. s.: 7354/2023-810 Č. z.: 13131/2023-1

Na rálade dl. 100 santadenia EF a R(El)č. 2017/625 EF a R $\sigma$  imstrijeli kontralski a iných tinadných članostiach vykonkvatých na zabezprední a platňovania porovinového a krmioveňo prísna a paradike pre adravic zvistná a dobří žirevné podmisnéh visinat, pre zdravic razliti a pre príprevky na ochrana razlila, Ministerstvo půdobnojodánista a rozvoja vůleka SR

#### urbajem

Ústredný kontrolný a skúlobný ústav poľnohospodársky v Bratislave, Odbor laboratémych činností

Pracovisku: Skulobné laboratérium analýzy pesticidov, Matiškova 21, 833 16 Bratislava

ako Národné referenéné laboratórium pre úradnú kontrolu chemických prípravkov na ochranu rastlín

Poveneuje sa vydáva na dobu utčítá do doby platnosti osvedženiu o aktoditácii.



Príloha 1: Povinnosti Národního referencinho laboradnia Príloha 2: Výkon sudita na národných referenčných laboradelách Príloha 2: Podmienky zrušenia menovania Národného referencichho laboratéria



### The Pesticide Analysis Testing Laboratory (PATL, Slovak abbr. as "SLAPe")

- The Pesticide Analysis Test Laboratory (SLAPe) specializes in chromatographic analyses.
- It is the National Reference Laboratory for the Official Control of PPPs, National Reference Laboratory for the Analysis of Feed Additives, Official Laboratory for the Analysis of Pesticide Residues of Feed and Plant Material, performs soil monitoring for persistent pesticide residues and persistent organic pollutants (POPs).
- Collaborates on non-clinical studies with the Slovak Academy of Sciences and the Slovak Medical University.



#### The Pesticide Analysis Test Laboratory SLAPe

 SLAPe has two quality systems in place. It has a flexible and fixed accreditation according to EN ISO 17025 and a system of good laboratory practice (GLP). Physicochemical and analytical studies are carried out in accordance with Act No. 67/2010 on the conditions for placing chemical substances and chemical mixtures on the market and on amendments to certain acts (Chemical Act) and in accordance with the principles of OECD GLP and Directive 2004/10/EC of the European Parliament and of the Council.





#### Inspection and control of PPPs in the Slovak Republic

Laboratory control of PPPs in SLAPe consists of:

- analyses of the active substances, safeners, impurities and co-formulants 662 indicators were analysed and carried out in 2023,
- determination of physico-chemical parameters (persistent foam, pH, specific gravity, emulsification characteristics, wet sieve test, suspension, wettability, dispersibility/spontaneous dispersion, macroscopic assessment - color, ...) – in 2023, a total of 986 parameters were determined,
- At the workplace, PPPs profiles are analyzed on HPLC, GC/FID and GC/MS in order to create profile libraries of PPPs. For profile analyses of polar PPPs, Fourier transform infrared spectroscopy instrumentation (so-called Raman with FTIR) was supplied to make it more effective and move the work with data processing, especially for false and counterfeit PPPs, to a higher level in the laboratory.





## Official control of the content of co-formulants in PPPs intended to be placed on the market

Before the authorization of each new PPP is granted, an administrative check of the content of unacceptable coformulants listed in Commission Regulation (EU) 2021/383 of 3 March 2021 amending Annex III to the Regulation of the European Parliament and Council (EC) no. 1107/2009, which includes a list of co-formulants whose inclusion in PPPs was not accepted.

In the area of coformulants, methods for the determination of toluene, 1,2-dichloroethane, phenol, propylene glycol, ethylene glycol and dimethyladipate are tested. However, these parameters are not yet prepared and incorporated into the scope of accreditation of the testing laboratory, so the performance of these tests in samples is only informative.

In the matter of official controls of PPPs, the Ministry of Agriculture and Rural Development of the Slovak Republic commissioned the Pesticide Analysis Testing Laboratory (SLAPE) of the Central Control and Testing Institute of Agriculture in Bratislava (CCTIA) as the only one in the Slovak Republic. The laboratory performs its activities in accordance with the requirements of the ISO IEC 17025:2017 (accreditation) and also in the Good Laboratory Practice (GLP) system. Regarding the testing/detection of co-formulants with toxicological, ecotoxicological or ecological significance in the sense of Regulation (EC) no. 1107/2009, the SLAPE laboratory currently does not have accredited analyzes for the determination of co-formulants.

CCTIA performs the control of co-formulants using as yet non-accredited methods in the form of monitoring.



SLAPe analysis is appointed by the Ministry of Agriculture and Rural Development of the Slovak Republic as the **National Reference Laboratory for the Official Control of Chemical Plant Protection Products**. SLAPe has been accredited since 2007 in SNAS, has implemented and works in two quality systems, namely:

- accreditation in a flexible and fixed scope according to EN ISO 17025, based on the requirements of Regulation (EU) 2017/625 of the European Parliament and of the Council. The number of accredited analyses of active ingredients and safeners in terms of flexible scope is gradually expanding according to customer requirements. As of 2020, SLAPe has validated and accredited 134 analytes.
- is a holder of the Certificate of Compliance with Good Laboratory Practice No. G-040 according to Act No. 67/2010 (Chemical Act) and in accordance with the principles of OECD GLP, is included in the National GLP Monitoring Program and in the Bulletin of the Office of Standardization, Metrology and Testing of the Slovak Republic. The results of the studies are valid in all EU countries and OECD countries.







#### **Determination of physicochemical parameters of PPPs**

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#### **ANALYTICAL TECHNIQUES**

- Application of Multi-Component Analytical Methods for the Determination of PPP Active Substances
  - HPLC/UV/FLD
  - GC/FID

- Determination of co-formulants and additives
  - HPLC/DAD
  - HS-GC/FID; GC-MS
  - FTIR Raman spectroscopy



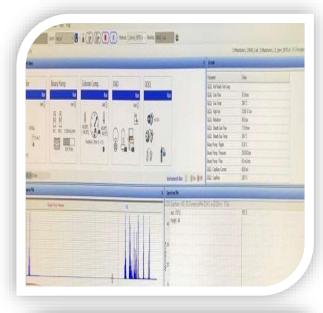


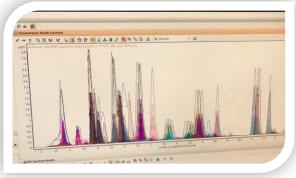


#### LC/MS-MS ANALYSIS OF PPPs AND THEIR RESIDUES



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#### **CHROMATOGRAPHIC ANALYSIS OF PPPs**

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#### **CHROMATOGRAPHIC ANALYSIS OF PPPs**

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#### MEASUREMENT CONTROL AND INTERNATIONAL ACTIVITIES OF SLAPE IN THE FIELD OF PPPs

- long-term and successfully participates in international CIPAC collaborative exams (*Collaborative International Pesticides Analytical Council*) for the validation of new analytical methods for the determination of active substances
- regularly participates in interlaboratory tests organized by European laboratories. By actively participating in the proficiency tests, we confirm the correctness of our analyses. In 2023, SLAPe successfully participated in the following international proficiency tests (MPS) for the analysis of PPPs
- Agence Fédéralepour la Sécurité de la Chaîne Alimentaire (AFSCA), Belgium - Proficiency testing on Physicochemical Properties of a Pesticide Formulation: 11 analytes analyzed
- Italian Laboratory of National Institute of Health ITPT 2023
  Plant Protection Products : 2 active substances

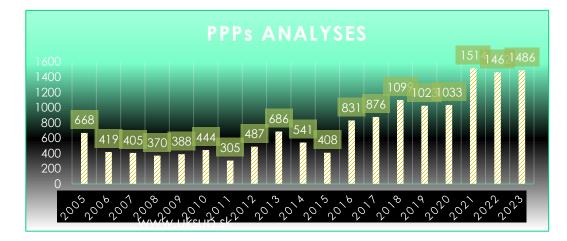




Official control of chemical plant protection products is directly related to consumer protection and food and feed safety. In SLAPe, official inspections of PPPs are carried out in the following scope:

- identification of active substances of safeners, co-formulants and impurities by the GC, HPLC
- determination of the content of active substances, safeners, co-formulants
- and impurities
- determination of physicochemical parameters
- GC/MS profiles of PPPs, illegal PPPs, identification of active substances in treated seeds
- Comparison of FTIR and RAMAN spectra

When checking products, the analysis of active substances alone is not enough, which is why the number of analyzed parameters is increasing:







- Reduced active ingredient content
- unsatisfactory physicochemical parameters (pH, density, foaminess, wettability...)
- composition of co-formulants and impurities
- contamination of spiked seeds
- Determining co-formulants and impurities is like looking for a "needle in a haystack" and requires formulation data that is often unavailable.
- Chromatogram 1 a sample of a PPP from the MPS test where we identified 4 undesirable co-formulants
- Chromatogram 2 contaminated spiked seed with declared active substances imazalil, ipconazole
- Chromatogram 3 a sample of a PPP with **isobutanol impurity**



#### **THANK YOU FOR YOUR ATTENTION**

