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# **CURRENT STATE OF POST REGISTRATION QUALITY CONTROL OF PLANT PROTECTION PRODUCTS IN POLAND**

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## HISTORY OF PESTICIDE QUALITY CONTROL IN POLAND

- 1960** – establishing of a special analytical laboratory at the Plant Protection Institute, Sosnicowice Branch.
- 1965** – chemical analysis and biological control of expired pesticides.
- 1965-1995** – testing of expired pesticides for the purpose of claims, post-registration control – sampling performed by Plant Protection Institute.
- 1995 – (until present)** – expired pesticides, claims, other testing for clients, – post-registration control – sampling performed by the National Plant Protection and Seed Inspection Service.
- 2004 – (until present)** – control of products based on selected active ingredients – ~30% of the total number of samples.

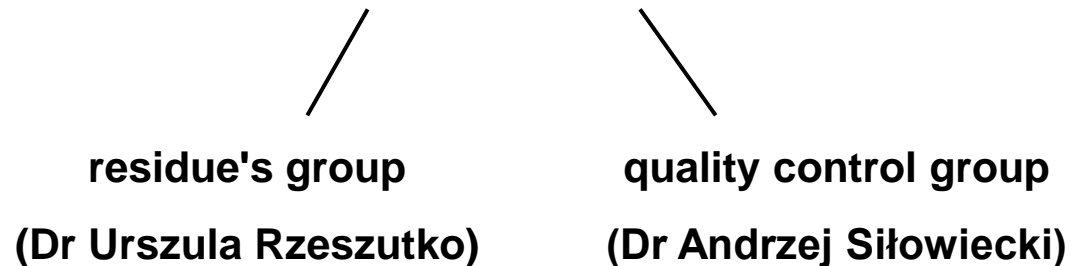


# PLANT PROTECTION INSTITUTE – NATIONAL RESEARCH INSTITUTE SOŚNICOWICE BRANCH

(part of Poznan's PPI since 1955)

– two scientific departments: Department of Pesticide Efficacy Testing  
(Dr Mariola Głazek)

Department of Analysis of Plant Protection Products  
(M.Sc. Stanisław Stobiecki)



49 people, 3 laboratory buildings, 15 ha experimental fields

## MAIN AREAS OF ACTIVITIES

- ❑ research on plant protection and analytical chemistry,
- ❑ post-registration quality control of plant protection products,
- ❑ investigations on efficacy of pesticides for registration purposes,
- ❑ obsolete pesticides and waste management – expertises and technical actions,
- ❑ residue analysis,
- ❑ expertises on quality of pesticides (generics, overterminated pesticides, claims from the users, technical concentrates, counterfeit products).



## TYPE OF ACTIVITIES

- ❑ **statutory** (scientific bases for plant protection programmes, analytics, waste disposal)
  
- ❑ **control** (quality of PPP and residues in plant material – monitorings)
  
- ❑ **technical** (quality testing, efficacy trials, waste management)



## QUALITY CONTROL – RESPONSIBILITIES

- ❑ quality control of PPP is an official duty of The Plant Health and Seed Inspection Service (PP&SI)

### PP&SI responsibilities

- ❑ providing funding for the entire program (sampling and lab testing until 2005; sampling only since 2006),
- ❑ country-wide sample collection,
- ❑ drafting guidelines and recommendations regarding inspecting,
- ❑ report approval,
- ❑ policy making.

### Plant Protection Institute responsibilities

- ❑ laboratory testing,
- ❑ preparation analytical certificates,
- ❑ annual reports (all data).



## LEGAL BASIS

- ❑ **Plant Protection Act of December 18, 2003**  
(Dz.U. [official legal register] of 2004, No 11, section 94 with subsequent amendments)

### Article 80, point 2

*The PPSI Service function of supervising the distribution and use of plant protection products shall include quality control of the plant protection products admitted for sale on the market...*

- ❑ **Council Directive No. 91/414/ EEC of July 15, 1991**  
regarding the placing of plant protection products on the market

### Article 17

*...plant protection products must be checked whether they meet the requirements defined in the registration process...*



## THE PURPOSE OF QUALITY CONTROL

- ❑ Perform quality control independent of producers and importers.
- ❑ Compare the level of technical parameters (product quality requirements) of plant protection products on the market with the requirements set in the registration process.
- ❑ Comply with Article 17 (Control Measures) of the Directive UE 91/414/EEC.
- ❑ Independent quality control of generic products, counterfeit products.
- ❑ Issue quality reports for disputed cases and claims.
- ❑ Preventing the use of products of poor quality, which, if used, could have an adverse effect on crops, humans and the environment.
- ❑ Maintain the technical capabilities for testing of all plant protection products admitted for use and distribution, as well as products withdrawn from the market (archival).





## QUALITY CONTROL SYSTEM

- ❑ **Samples collected by PP&SI Service inspectors according to annual plan**
- ❑ **Herbicides, insecticides, fungicides and other domestic and imported products (no recommendations – selection at inspector’s discretion)**
- ❑ **350-400 samples of plant protection products a year, including a reserve for interventional cases and counterfeit products**
- ❑ **computer system of registration and record keeping**
- ❑ **on-line information for PP&SI on sampled products**

**OFFICIAL CONTROL IS AN INDEPENDENT QUALITY MONITORING**



## CHANGES IN THE CONTROL SYSTEM (since 2004)

- introduction of scheduled, random and interventional control
  - **scheduled control** involves checking the quality of plant protection products containing selected active substances
  - **random control** involves checking the quality of plant protection products available on the market after random selection (all groups of products regardless of the container size, price, domestic or imported).
  - **interventional control** involves necessary control testing to process complaints and claims, **including cases of counterfeiting**
- testing the purity and compliance with plant protection product recipes (purity of technical concentrates and impurity profiles).



# LABORATORY INVESTIGATIONS

- ❑ chemical analysis of PPP – all samples
- ❑ biological efficacy – selected samples

## TESTING METHODS

**chemical testing methods are based on**

- ❑ industry standards,
- ❑ national Polish Standards (PN),
- ❑ study methods,
- ❑ FAO Specifications,
- ❑ scientific literature,
- ❑ methods by pesticide producers,
- ❑ internal PPI methods.

**biological testing methods are based on**

- ❑ laboratory tests :
  - fungicides – 11 tests,
  - insecticides – 11 tests,
  - herbicides – 6 tests,
  - phytotoxicity – greenhouse testing or field testing on selected crops



# CHEMICAL ANALYSIS

## a.i. content

- classic methods
- titration methods
- gas chromatography
- liquid chromatography
- spectrophotometry
- GC-MS
- TLC
- HPTLC (TLC scanner)
- NIR (near infrared spectroscopy)

## phys.-chem. properties

- emulsion stability
- suspension stability
- sieve testing
- water content
- wetting time
- pH
- specific gravity
- other



## CHEMICAL ANALYSIS – NEW POSSIBILITIES

EU PROJECT in PPI SOŚNICOWICE BRANCH

(closing the project – September 2008)

- ❑ total renovation of two laboratories
- ❑ purchase of modern analytical equipment
  - GC (detectors NPD, EC)
  - HPLC (DIODE-ARRAY-DETECTOR: DAD)
  - GC-MS system – 2 sets (FID, ECD, MS-EI, MS-Chem I )
  - LC/MS/MS System (triple quadrupole)

**all** – special dedicated to quantitative analysis and identification of impurities in plant protection products and residue analysis

**fully equipped** – special detectors, librarys etc.



## MULTI-YEAR PROGRAMME:

**„Protection of crops with consideration for the food safety and minimizing the yield losses and threats to human health, household animals and the environment”**

### Task 2.4.

**CONDUCTING QUALITY ANALYSES OF ACTIVE SUBSTANCES AND PLANT PROTECTION PRODUCTS – OFFICIAL QUALITY TESTING OF PLANT PROTECTION PRODUCTS PLACED ON THE MARKET**

**SECURED FUNDING 2006-2010**



## NUMBER OF OFFICIAL SAMPLES TESTED IN 2006-2007

| <b>2006</b>           |            | <b>2007</b>           |            |
|-----------------------|------------|-----------------------|------------|
| <b>Scheduled</b>      | <b>17</b>  | <b>Scheduled</b>      | <b>44</b>  |
| <b>Random</b>         | <b>293</b> | <b>Random</b>         | <b>245</b> |
| <b>Interventional</b> | <b>10</b>  | <b>Interventional</b> | <b>11</b>  |
| <b>TOTAL</b>          | <b>320</b> | <b>TOTAL</b>          | <b>300</b> |



## TESTING ANALYSES IN 2007

| Samples collected by PP&SI | Number of samples |
|----------------------------|-------------------|
| OFFICIAL SCHEDULED         | 44                |
| OFFICIAL RANDOM            | 245               |
| INTERVENTIONAL             | 11                |
| <b>TOTAL</b>               | <b>300</b>        |

| Number of determinations                   |             | Number of negative determinations |
|--|-------------|-----------------------------------|
| physical-chemical:                         | 91          | 1                                 |
| content of a.s.:                           | 76          | 4                                 |
| biological efficacy:                       | 2           |                                   |
| phytotoxicity:                             | 0           |                                   |
| physical-chemical:                         | 569         | 29                                |
| content of a.s.:                           | 349         | 18                                |
| biological efficacy:                       | 18          |                                   |
| phytotoxicity:                             | 0           |                                   |
| physical-chemical:                         | 18          |                                   |
| identification, a.s. content, impurities : | 32          | 3                                 |
| biological efficacy:                       | 0           |                                   |
| phytotoxicity:                             | 0           |                                   |
| <b>SUMA</b>                                | <b>1155</b> | <b>55</b>                         |





# RESULTS OF DETERMINATIONS OF PHYSICAL AND CHEMICAL PROPERTIES 2006 – 2007

| <b>Year</b> | <b>Total no. of determinations</b> | <b>No. of negative determinations</b> | <b>% of negative determinations</b> |
|-------------|------------------------------------|---------------------------------------|-------------------------------------|
| <b>2006</b> | <b>757</b>                         | <b>44</b>                             | <b>5.8</b>                          |
| <b>2007</b> | <b>660</b>                         | <b>30</b>                             | <b>4.5</b>                          |



## QUALITY CONTROL – DIFFERENT ORDERS FROM THE MARKET

| Year      | Assortment | Number of overterminated PPP | Number of negative results | Number of interventional PPP | Number of interventional PPP – bad quality negative results |
|-----------|------------|------------------------------|----------------------------|------------------------------|---|
| 2005      | 205        | 391                          | 7                          | 36                           | 15  |
| 2006      | 269        | 667                          | 12                         | 48                           | 12  |
| 2007      | 257        | 820                          | 7                          | 73                           | 18  |
| 2005-2007 | –          | 1878                         | 26                         | 157                          | 45  |



## QUALITY CONTROL – SUMMARY AND CONCLUSIONS

- ❑ In 2007 official control analysis were performed for 289 samples  
(in 2006 – 310 samples)
- ❑ In 2007 – 11 samples of interventional cases were processed  
(in 2006 – 10 samples)
- ❑ a.i. content:
  - 2006 – 13,6% of the results did not meet standard requirements
  - 2007 – 5,2% of the results did not meet standard requirements
- ❑ phys-chem. properties:
  - 2006 – 5,8% of the results did not meet standards
  - 2007 – 4,5% of the results did not meet standards

## QUALITY CONTROL – SUMMARY AND CONCLUSIONS (continued)

- ❑ In 2006, 10 interventional samples were tested. Negative reports were issued for 2 products: Grodyl 75 WG and Glean 75 WG. These were obvious counterfeit products.
- ❑ In 2007 – 11 interventional samples were tested.
- ❑ Based on the results we could conclude that the quality of plant protection products available on the market in Poland is very good. This is due to a relatively small number of discrepancies between the product and the requirements regarding the content of active ingredient and physical and chemical properties.
- ❑ The computer system designed to avoid testing products from the same batch functions well and fulfills its role.
- ❑ There is a need to continue the „quality monitoring” and, whenever justified, the producers should be notified if a products received a negative analytic certificate.



## **SUMMARY AND CONCLUSIONS (continued)**

- ❑ There is a justified need to pay more attention to the quality of generic products and selected preparations that contain the active substances.**
- ❑ The laboratory facility at the Plant Protection Institute is well equipped to handle all kinds of pesticide quality cases.**
- ❑ The independent quality control system in Poland that involves collaboration between the National Plant Protection and Seed Inspection Service and the Plant Protection Institute served well the interest of the consumers of products, producers and other participants in the sales and distribution of plant protection products.**
- ❑ It is worth emphasizing that it is possible to test a product suspected of poor quality free of charge (thanks to secured funding under the “Multi-year programme”) – counterfeit products also – by the plant protection inspectors action.**