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CEUREG FORUM XVII

THE PROBLEM OF ILLEGAL PESTICIDES - POLISH EXPERIENCES ON THEIR IDENTIFICATION

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CEUREG FORUM XVII organised by National Food Chain Safety Office

Budapest, Hungary 14th-15th October 2013

INSTITUTE OF PLANT PROTECTON - NATIONAL RESEARCH INSTITUTE

PESTICIDE QUALITY TESTING LABORATORY

Head of the Laboratory: Marek Miszczyk, Ph. D.

- ✓ **Andrzej Siłowiecki, Ph. D.**
- ✓ **Marlena Płonka, M. Sc.**
- ✓ **Jarosław Ścipień, M. Sc.**
- ✓ **Ewa Ledzińska, M. Sc.**
- ✓ **Zuzanna Foks**
- ✓ **Iwona Knapik**
- ✓ **Aleksandra Ciuman**
- ✓ **Mirela Wiśniewska**
- ✓ **Gizela Harensa**

QUALITY SYSTEM **- first GLP Certificate: 2008**

GLP Cerificate No 15/2012/DPL in the range of: - composition testing of pesticides
- phys.-chem. testing

PPP's QUALITY TESTING IN POLAND

- ✓ **Official quality checking of pesticides for State Plant Health and Seed Inspection Service – 1107/2009, Chapter VIII, Art. 68**
- ✓ **Testing of expired products (1960-2013) – finished May 2013 because of new pesticide regulation**
- ✓ **Testing of interventional pesticides (claims, complaints, prosecutor's and police orders, others from users and distributors)**
- ✓ **Chemical and phys.-chem. analysis of ppp's and technical concentrates for registration purposes**

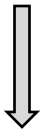
ORGANIZATION OF QUALITY TESTING OF PPP'S IN POLAND

OFFICIAL CONTROL

Ministry of Agriculture and Rural Development

and

National Plant Protection and Seed Inspection Service



Institute of Plant Protection – National Research Institute

Multi-year national programme for 2011-2015

„CROP PROTECTION ACCOUNTING FOR FOOD SAFETY AND PREVENTION OF YIELD LOSSES AND HAZARDS TO HUMAN HEALTH, LIFESTOCK AND THE ENVIRONMENT"

Task 1.9: „Quality analysis of active substances and plant protection products"

Funding provided from the national budget since 2006 till 2012

ORGANIZATION OF QUALITY TESTING IN POLAND

OFFICIAL CONTROL

Task 1.9

1) **National Plant Protection and Seed Inspection Service**

SCOPE:

- ✓ draft plans
- ✓ collect control samples
- ✓ provide substantive supervision of the task
- ✓ receive and approve reports

2) **Institute of Plant Protection – National Research Institute Sośnicowice Branch**

SCOPE:

- ✓ conduct analytic testing
- ✓ perform analyses of the results
- ✓ issue certificates of analysis
- ✓ draft detailed annual reports
- ✓ create databases

LABORATORY OF QUALITY CONTROL OF PPP's

- ✓ gas chromatography: FID, ECD, HEAD SPACE, GC-MS (EI, CHEM)
- ✓ liquid chromatography: UV, DAD, RR, LC-MS/MS
- ✓ spectrophotometry: NIR
- ✓ phys-chem methods:
  - stability of emulsion/suspension
  - sieve analysis
  - water content
  - wetting time
  - pH, density
  - stability

NUMBER OF PPP'S SAMPLES TESTED OFFICIAL CONTROL (2009-2013)

Year	Number of samples - official control	Number of samples - interventional control
2009	261	45
2010	271	78
2011	268	40
2012	275	79
2013	227	34
Total	1302	276

NUMBER OF PRODUCTS AND ACTIVE SUBSTANCES TESTED – OFFICIAL CONTROL 2012

Different products tested – 157

Active substances tested – 115

EXPIRED AND INTERVENTIONAL SAMPLES FROM THE MARKET (2012-2013)

Type of control samples	Number of samples		Number of „negative" results	
	2012	2013	2012	2013
Expired	738	585	17	13
Intervention	37	16	29	14

THE OFFICIAL QUALITY CONTROL IN 2012

Type of control	Number of samples	Type of determination	Number of determinations	Number of negative certificates
Basic	275	phys.-chem. prop	528	11
		content of a.s.	445	
		additional det.	18	
Interventional	79	phys.-chem. prop	186	40
		content of a.s.	167	
		additional det.	14	
Total	354		1358	51

THE OFFICIAL QUALITY CONTROL IN 2012

Type of control	Number of "negative" results	Reason (1)	Reason (2)	Reason (3)	Reason (4)
Basic	11	5	5	1	0
Interventional	40	8	11	7	14

(1) - samples does not comply the requirements (a.s., phys-chem prop.)

(2) - differences in a formulation (does not comply the declared content)

(3) - differences in impuritie's profile

(4) - others (proper labeling, containers)

THE OFFICIAL QUALITY CONTROL IN 2013

in the process of analysis

Type of control	Number of "negative" results	Reason (1)	Reason (2)	Reason (3)	Reason (4)
Basic	0	0	0	0	0
Interventional	6	0	6	0	0

(1) - samples does not comply the requirements (a.s., phys-chem prop.)

(2) - differences in a formulation (does not comply the declared content)

(3) - differences in impuritie's profile

(4) - others (proper labeling, containers)

„NEGATIVE” RESULTS (CERTIFICATES) 2012-2013

Type of control	Total number of "negative" results	Reason (1)	Reason (2)	Reason (3)	Reason (4)
Basic	11	5	5	1	0
Interventional	46	8	17	7	14

(1) - samples does not comply the requirements (a.s., phys-chem prop.)

(2) - differences in a formulation (does not comply the declared content)

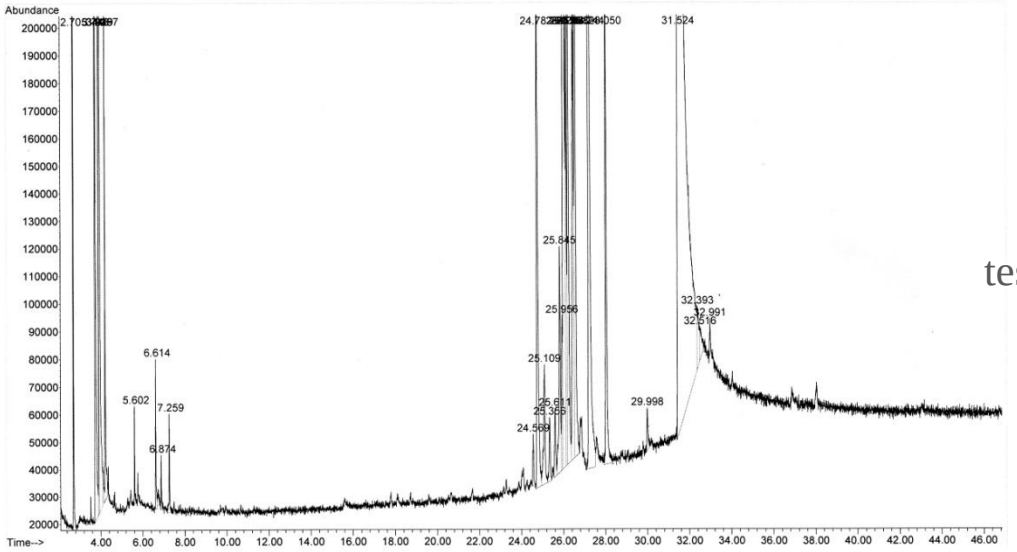
(3) - differences in impuritie’s profile

(4) - others (proper labeling, containers)

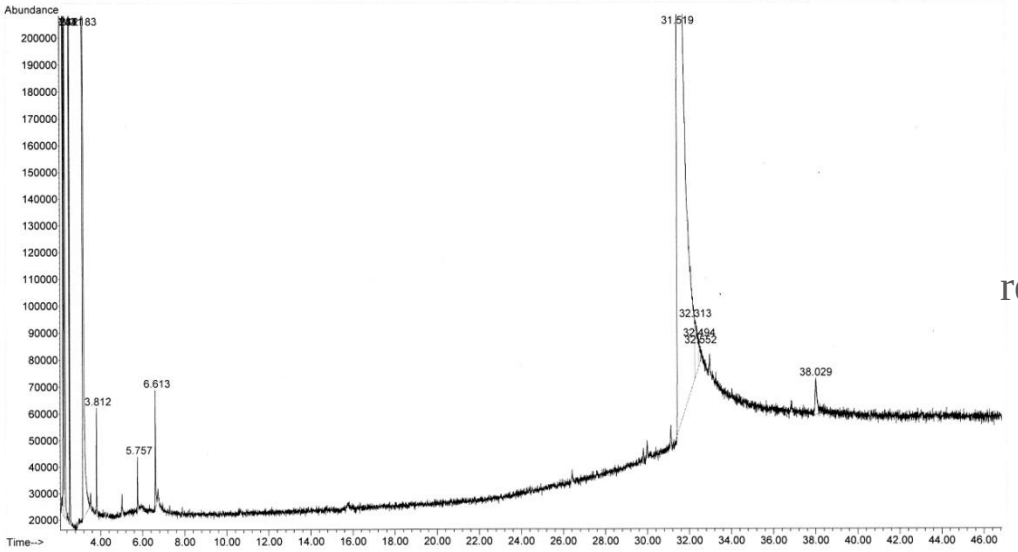
THE PARALLEL TRADE PESTICIDES IN „NEGATIVE" RESULTS IN 2012-2013

Type of control	Number of "negative" certificates	Number of "negative" parallel trade
Basic	11	5
Interventional	46	14
Total	57 in 2010-2011 – **94**	19 in 2010-2011 – **25**

BIG DIFFERENCES (GC-MS)

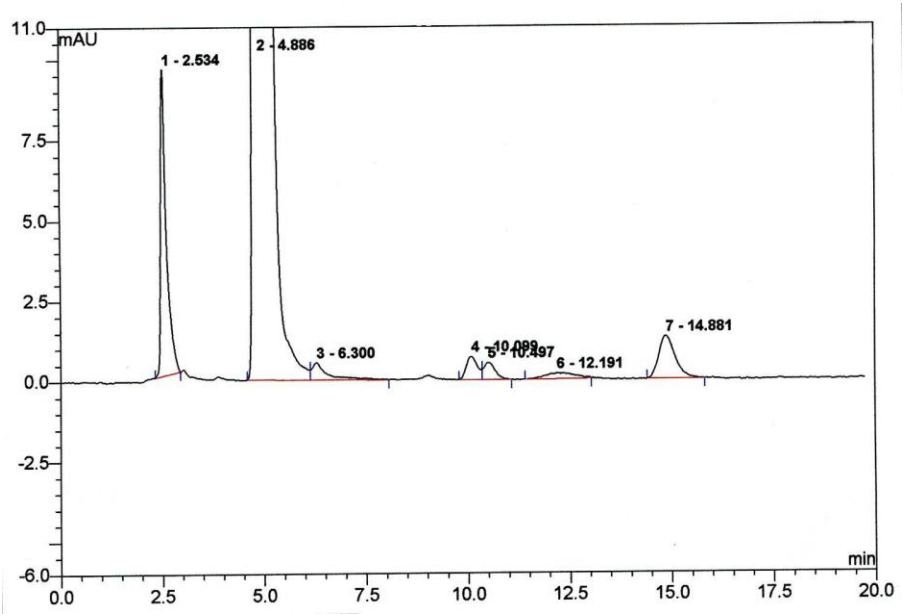


tested product

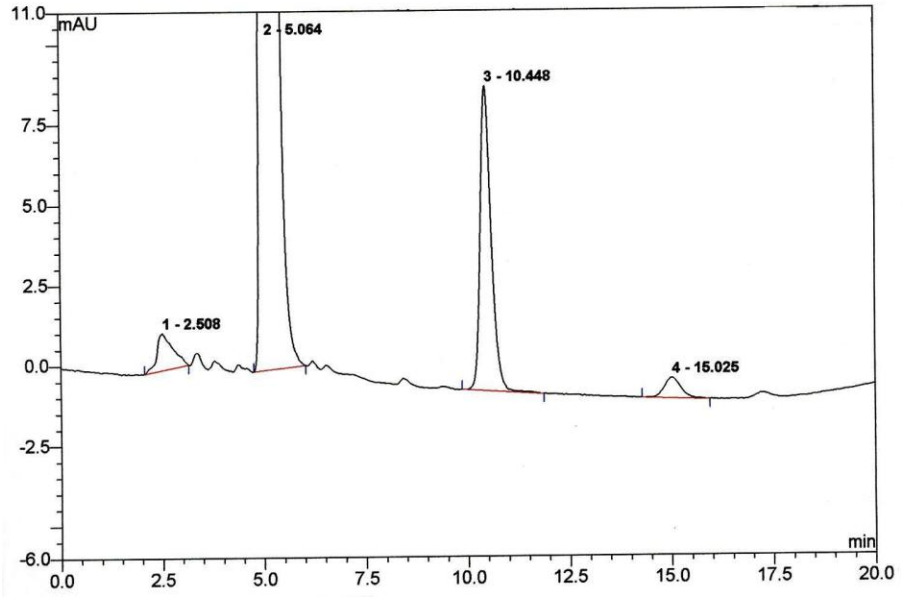


reference product

BIG DIFFERENCES (HPLC)

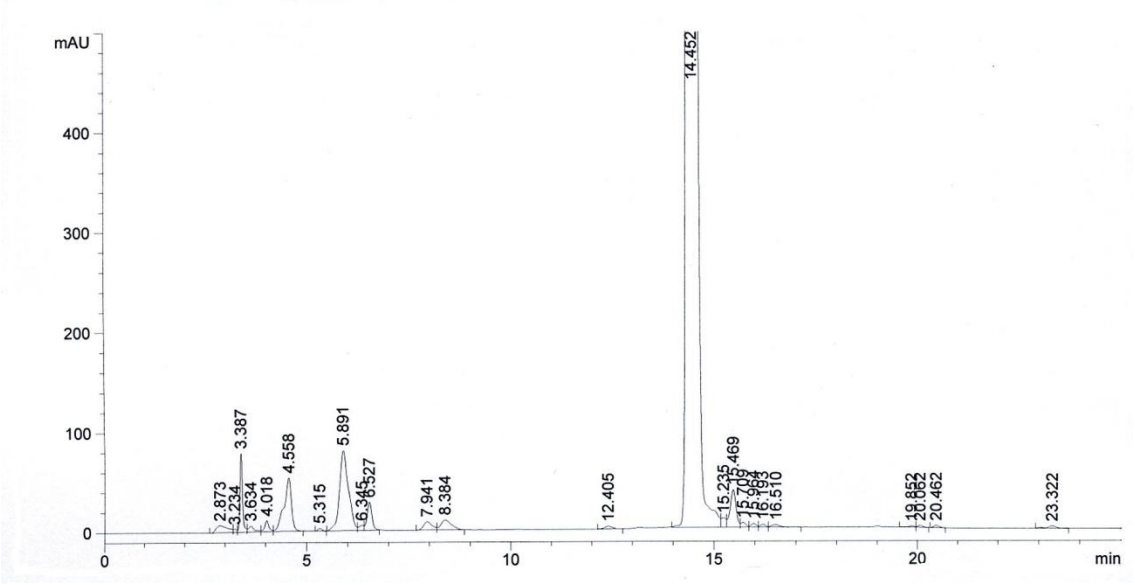


tested product

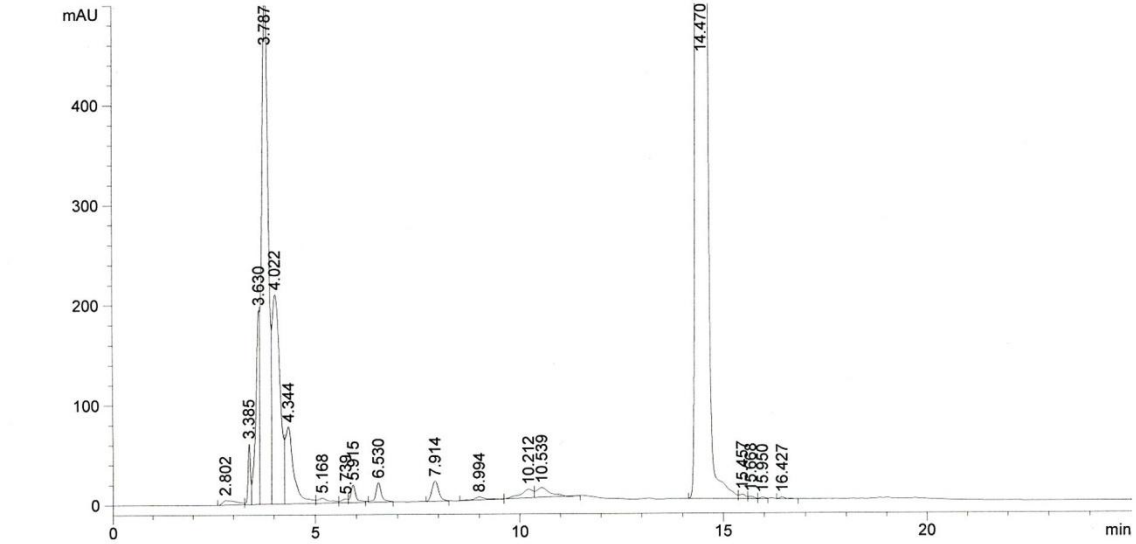


reference product

BIG DIFFERENCES (HPLC)



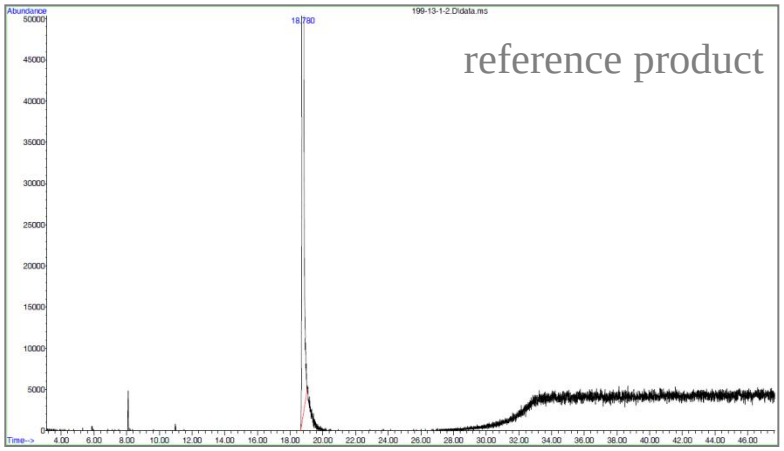
tested product

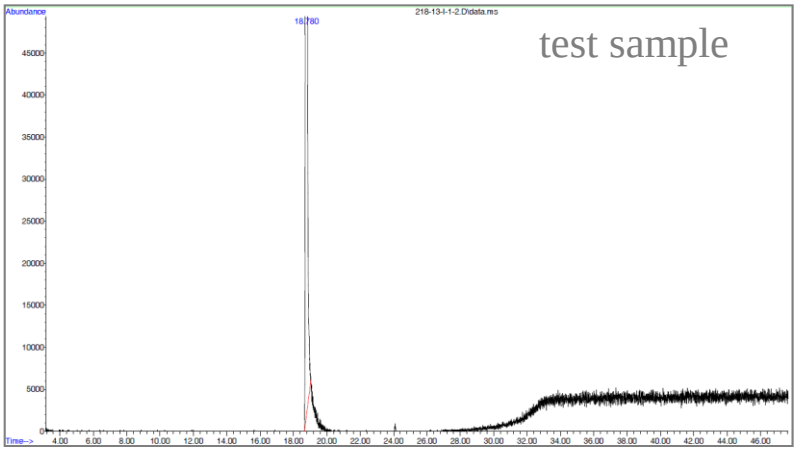


reference product

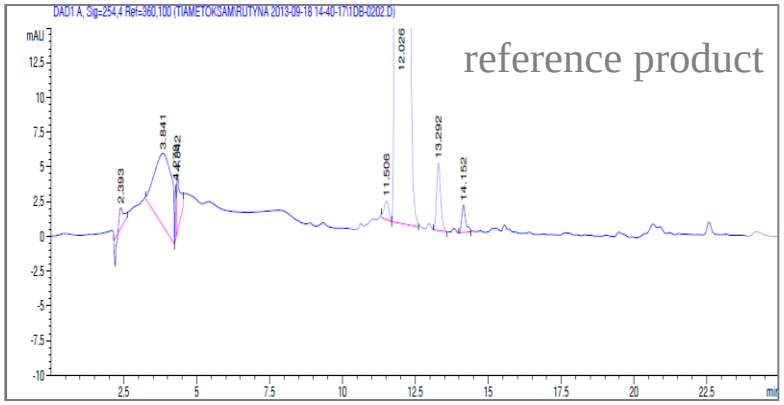
USAGE OF HPLC and GC-MS

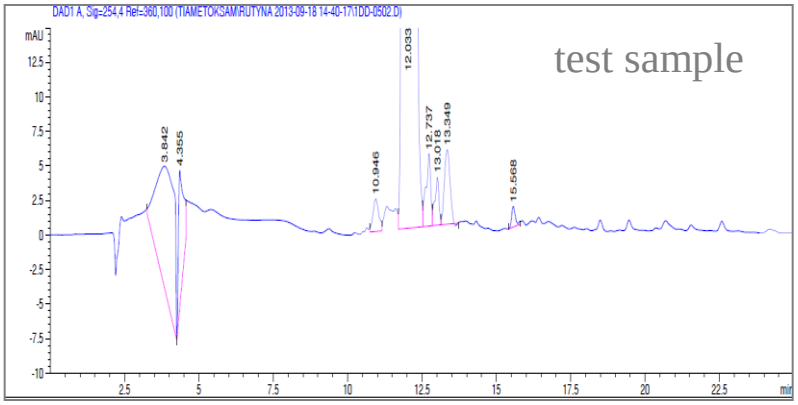
Analysis by GC-MS technique





Analysis by HPLC technique

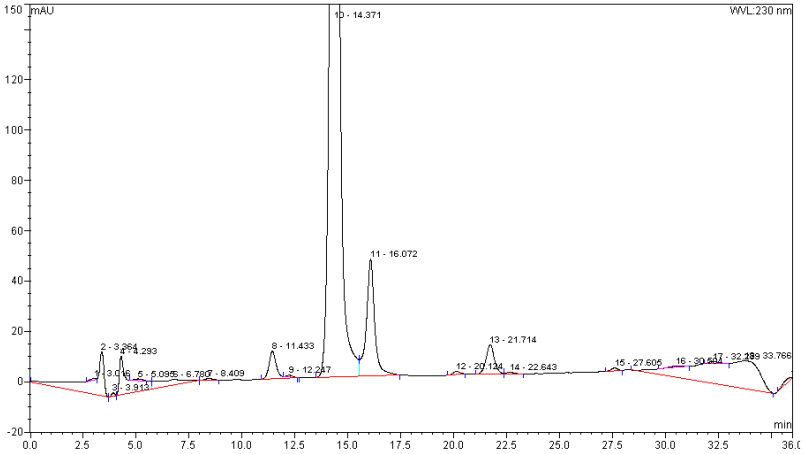




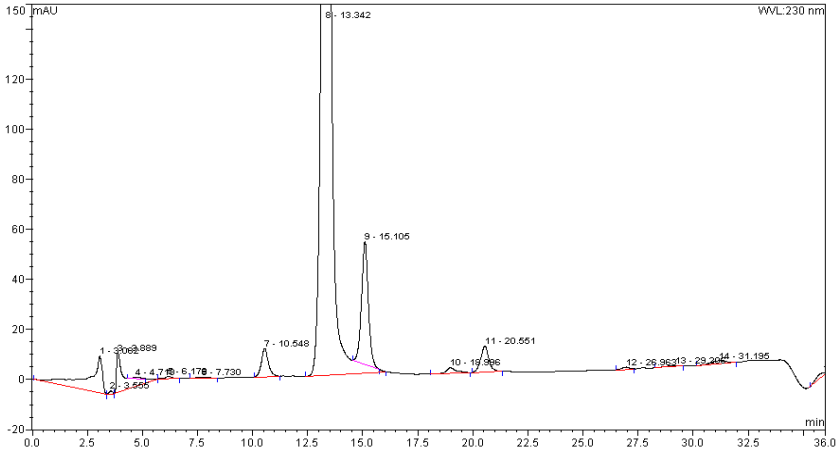
EXAMPLE OF IDENTITY TESTING IN PARALLEL TRADE

- ✓ **Tested sample**
- ✓ **Reference sample derived directly from the producer with its certificate of origin (certificate of analysis)**
- ✓ **Sample from parallel trade delivered by authorisation holder**

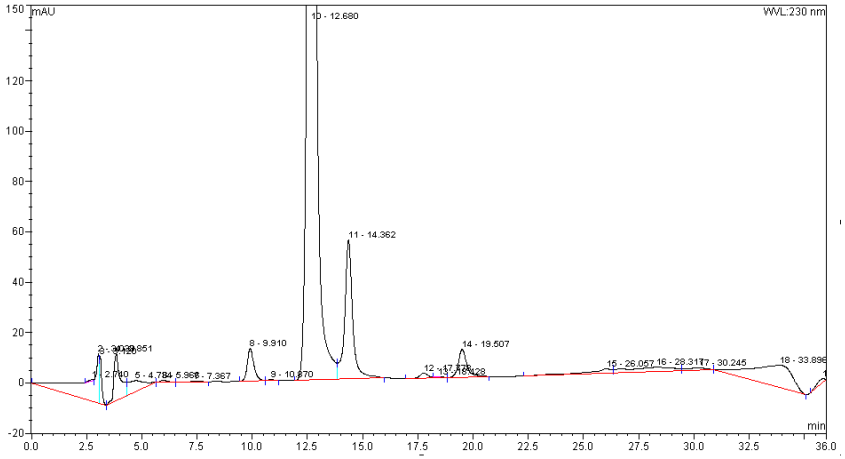
AN EXAMPLE OF THE FULL ANALYSIS OF IDENTICALITY OF PARALLEL TRADE PESTICIDE



1. tested product



3. standard sample of parallel product



2. reference product

DIFFICULTIES OF THE CONTROL LAB

- ✓ **Incomplete documentation (product composition, results of 5-batch analysis, analytical methods for a.s. and impurities)**
- ✓ **Lack of reference material of adequate quality**

> **Reference material must come directly from the product manufacturer and be supplied with the Certificate of Analysis**

- ✓ **Original manufacturer does not know the acceptable variability limits between different batches of the product**
- ✓ **Overengagement of technical means (equipment capacity, time)**
- ✓ **Constantly changing subject matter – variety of topics**

RECOMMENDATIONS - SUGGESTIONS

1. To perfect the definition of identity in parallel trade
2. To establish rules on packaging and labeling of parallel trade products
3. To supply documentation to the control lab, lack of product quality requirements, 5-batch analyses of active substances
4. EC to create an international team of experts to fight against counterfeit crop protection products
5. Poland offers support the initiative towards creating legislation concerning control of crop protection products
6. To improve exchange of information between the different institutions and countries involved in the issue