

The background of the slide is a photograph of a modern, two-story laboratory building. The building has a mix of tan and light grey exterior walls. On the left side, the word "OMIC" is prominently displayed in large, dark letters. The building features large glass windows and a central entrance. In the foreground, there is a paved parking lot with several parking spaces, some marked with blue handicapped symbols. A red pickup truck is parked on the right side of the lot. The building is surrounded by green lawns and young trees. In the background, a dense forest of evergreen trees covers a hillside under a clear sky.

Pesticide Screening of Food Products with GC- AED for Import / Export Business

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Agenda

- **Regulation USA versus Japan**
- **AED Pesticide Screening Method**
- **AED OrganoTin Method**
- **AED Inorganic Bromide Method**

USDA-FDA

I General commodity monitoring program

- Enables enforcement of tolerances
- Determines incidences and level of pesticides in domestic and imported raw commodities, processed foods and animal feeds

USDA-FDA

II Total Diet Study (TDS) Program

- Testing food product using “market-basket” approach.
- Conducting nationwide surveys to test residue levels in selected commodities.

Japan “Positive List” Program

Prohibits the distribution of food that contains agricultural chemical above a certain level (usually 0.01 ppm) if maximum residue limit has not been established

Outline of Positive List System for Agricultural Chemical Residues

【 Current Regulation 】

Pesticides, Feed Additives,
and Veterinary Drugs

Chemicals for which
MRLs are established

283 substances

MRLs for 250 Pesticides and
33 Veterinary Drugs



Foods containing chemicals
above the MRLs are enjoined
from domestic distribution

Chemicals for which MRLs
are not established



Basically, even foods found to
contain chemicals are not
enjoined from distribution.

【 Enforcement of Positive List System (May 29, 2006~) 】

Pesticides, Feed Additives and Veterinary Drugs

Chemicals for which
MRLs are established

799 substances

Establishment of provisional
MRLs for agricultural
chemicals, considering Codex
standards, Japanese
registration withholding
limits, and other standards
established based upon
scientific evaluation

758 substances

Acceleration of the
establishment of MRLs

Foods containing chemicals
above the MRLs are enjoined
from domestic distribution.

Chemicals for which
MRLs are not
established

Establishment of a
certain level that is
determined to pose no
adverse health effects

0.01 ppm

Foods found to contain
chemicals above the
level are enjoined from
domestic distribution.

Chemicals
designated by
MHLW

Chemicals that do
not pose adverse
health effects

65 substances

Not subject to the
positive list system



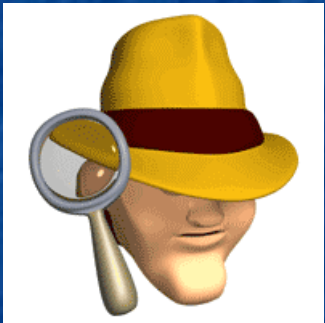


HOW?

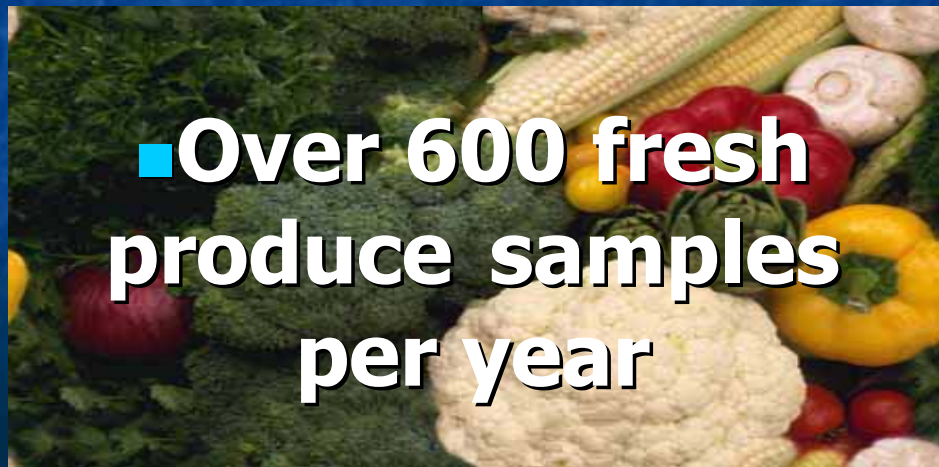
- Developing of multiresidue screening method to detect trace concentrations using a single procedure
- Utilizing selective detectors such AED to reduce bench-work time
- Process needs to be Rapid, Specific, and encompass a wide range of pesticides

OMIC USA Inc.

Export Inspection Business



Over 2,000 grain samples per year



■ Over 600 fresh produce samples per year

Multi Residue Pesticide Screening Method

Year	Nr. of Pesticide	Detectors	Method
1993	30	ECD NPD FPD	Acetone extraction Partition with MC Florisil Cleanup
1997	156	AED MSD	Acetonitrile extraction C ₁₈ and NH ₂ SPE cleanup
2006	241	AED ECD MSD	Acetonitrile extraction C ₁₈ and NH ₂ SPE cleanup

Multi Residue Extraction Method

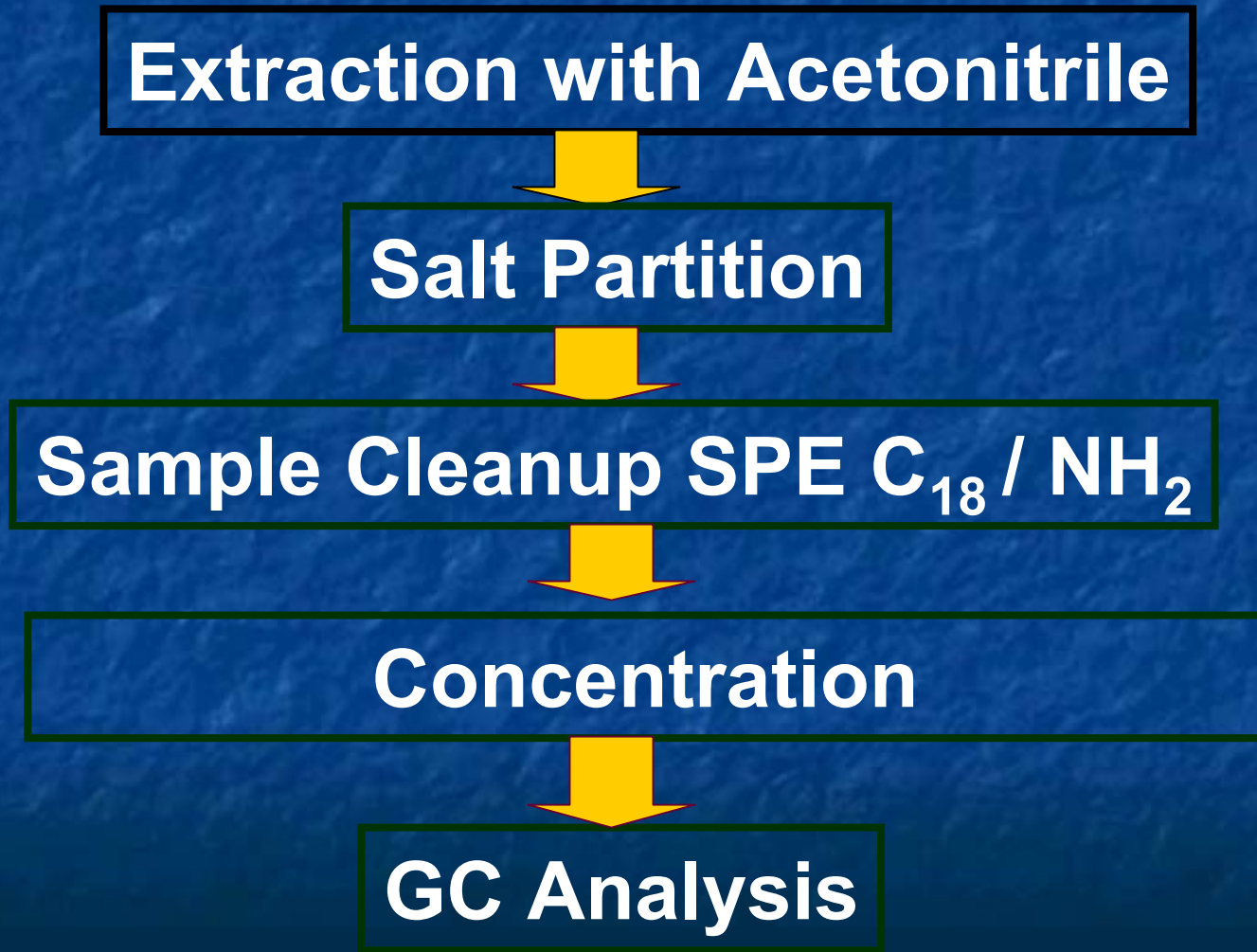
Extraction with Acetonitrile

Salt Partition

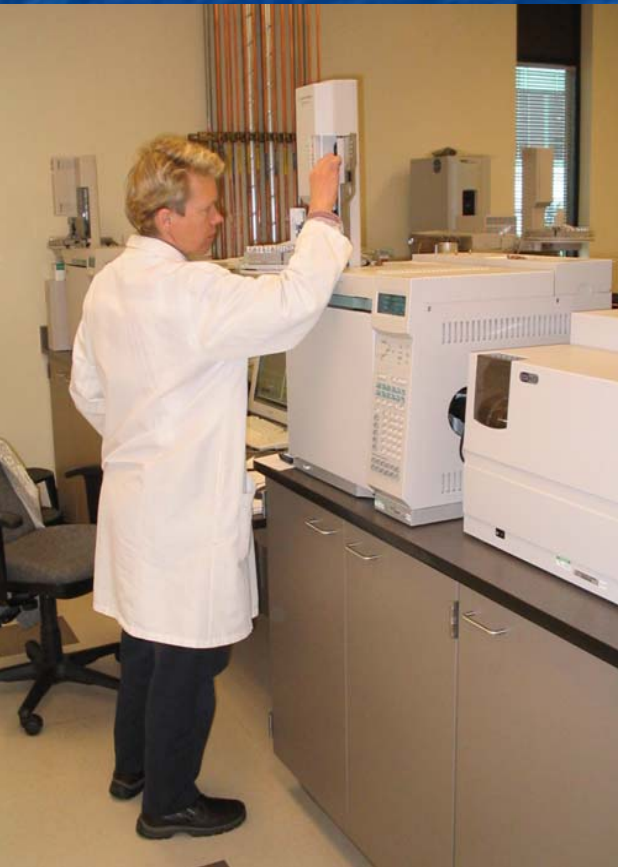
Sample Cleanup SPE C₁₈ / NH₂

Concentration

GC Analysis

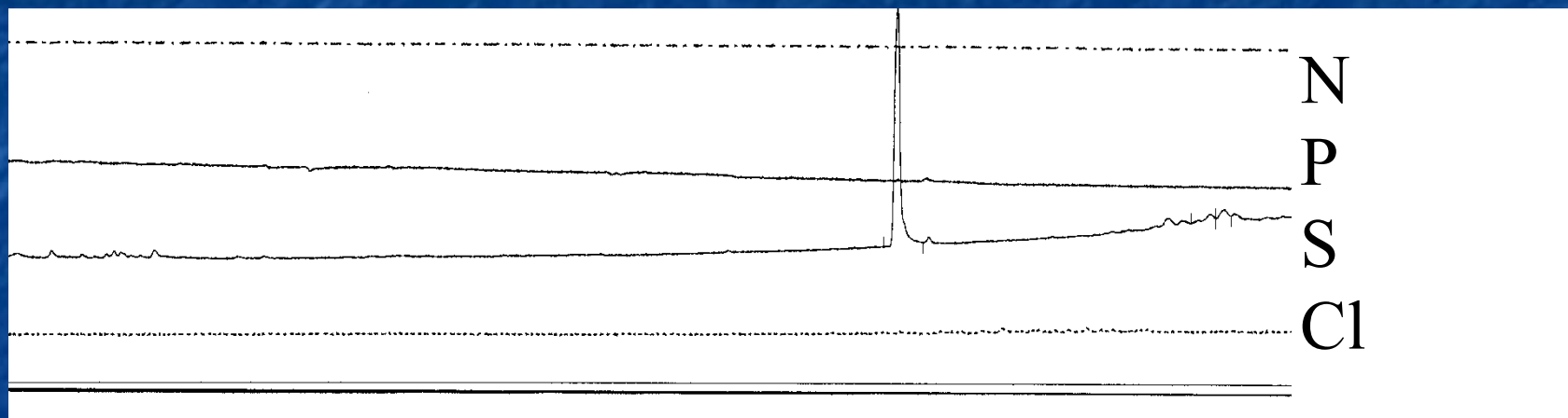


Multi Residue AED Method



- **Model: JAS G2350A**
- **Column: HP-5MS, 30 m
0.32 mm x 0.25 um**
- **Reference std:
M. Chlorpyrifos**
- **Retention Time Locking**
- **AED Library - 177 compounds**

Multi Residue AED Method



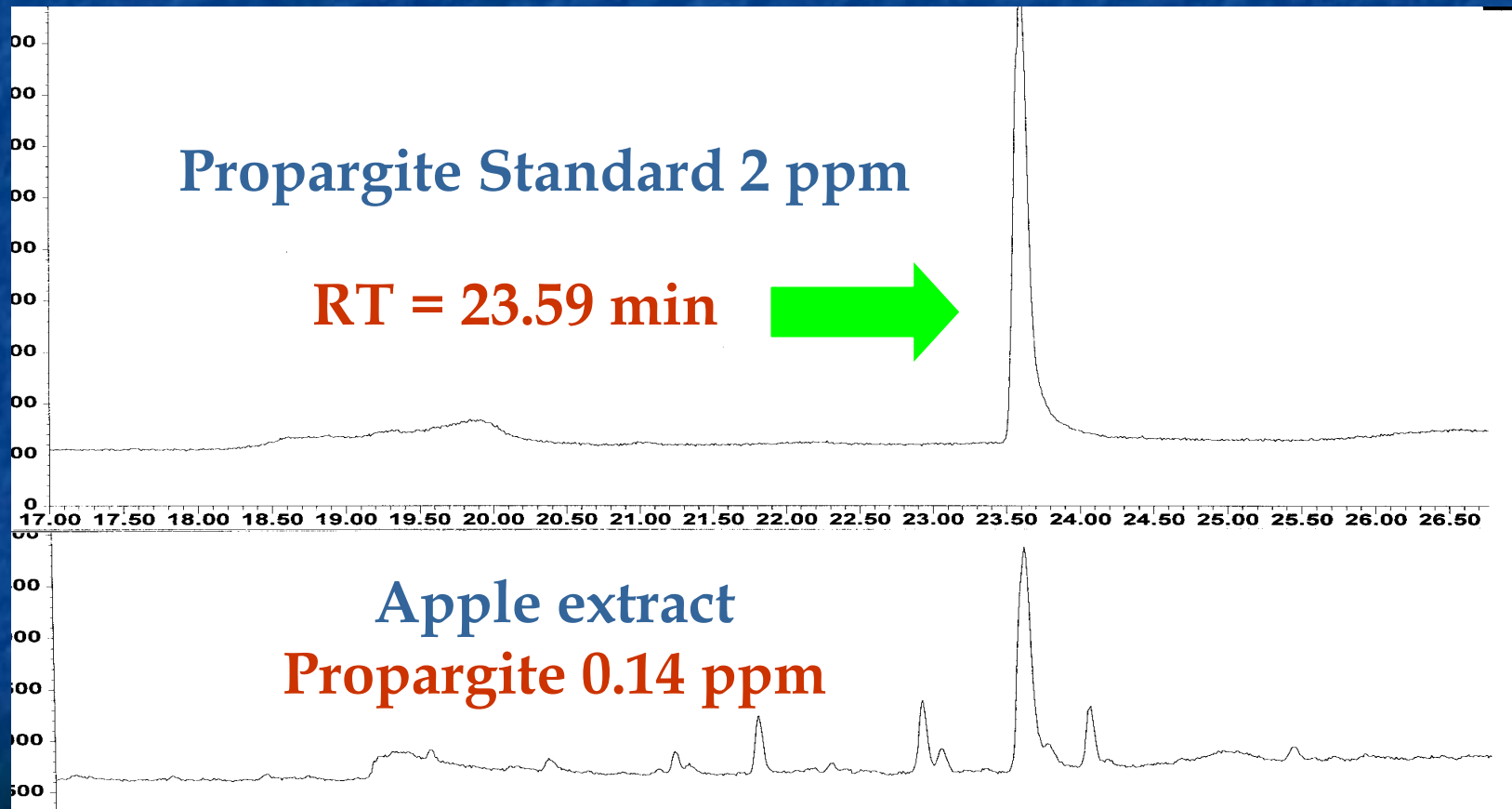
Type of sample: Apple

RT of unknown peak = 24.008 min; RRT = 1.358

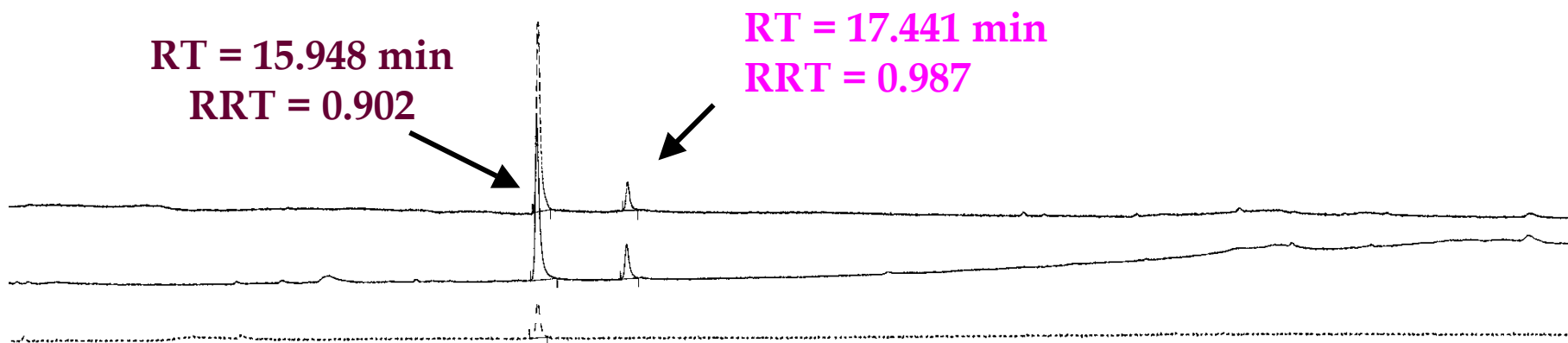
SULFUR TABLE

Captofol	Cl ₄ NS	S 1345
Propargite	S	S 1.359
Phosmet	NPS ₂	S 1.397

Multi Residue Method Confirmation MSD



Multi Residue AED Method



Type of sample: Wheat

<u>From Library</u>	<u>RRT P</u>	<u>RRT S</u>	<u>RRT Cl</u>
Parathion M.	0.900	0.900	---
M. Chlorpyrifos	0.902	0.902	0.902
Malathion	0.985	0.985	---
Fenthion	0.996	0.996	---

Multi Residue Method Confirmation MSD

M. Chlorpyrifos

Tgt Ion: 286

Q ion: 288

Result: 0.133 ppm

Malathion

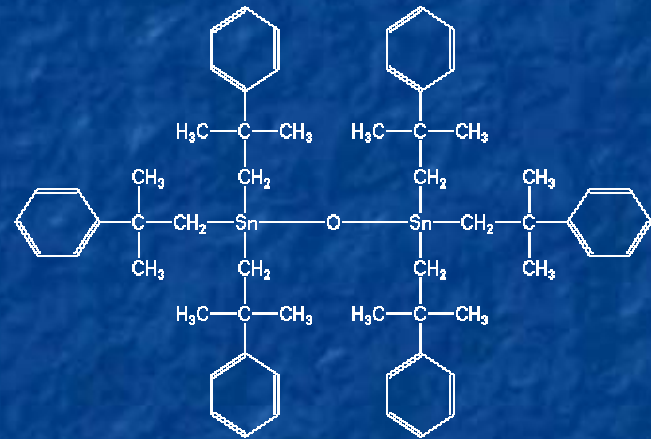
Tgt Ion: 125

Q ion: 173

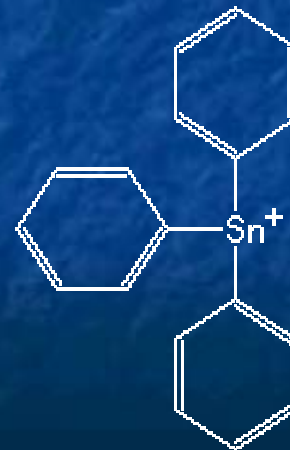
Result: 0.133 ppm

Organo Tin AED Method

- Fenbutatin Oxide

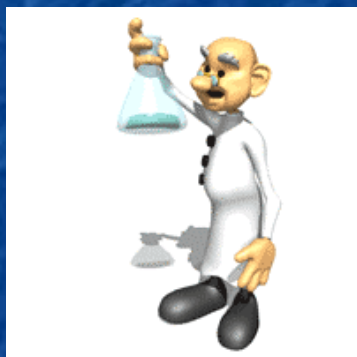


- Fentin



Organo Tin Method

■ Extraction:



- Hexane : Diethyl Ether (2:3)
- Alkylation:
Ethyl Magnesium Bromide
- SPE Si Gel clean up (for MSD only)

■ Detection:



- GC-AED (TIN 303) Channel
- Fentin Tgt Ion 351, Q1 349, Q2 350
- F. Oxide Tgt Ion 415, Q1 414, Q2 519

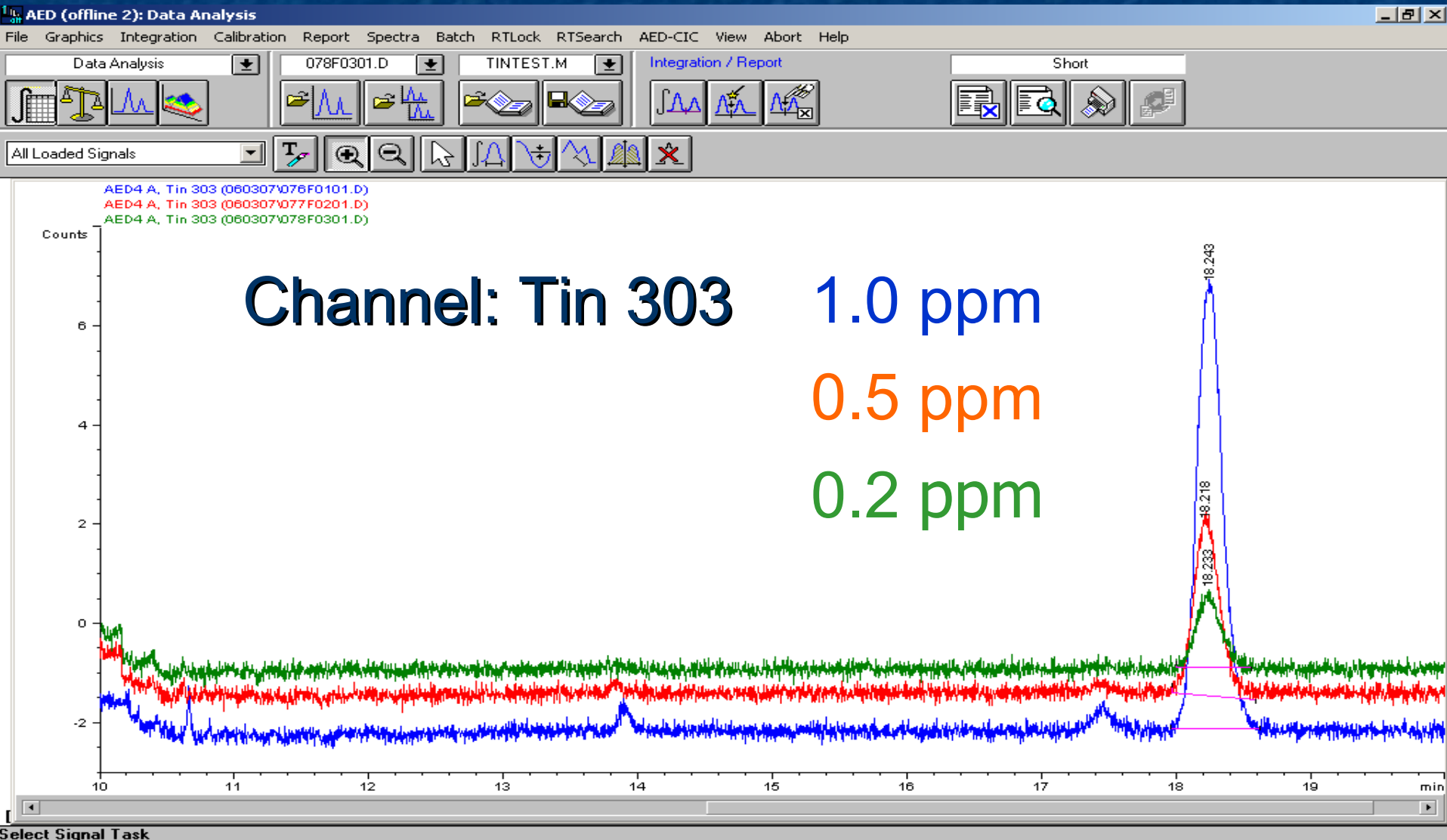
Organo Tin AED Method

- HP6890 + GC with AED Detector, Channel TIN 303
- Phenomenex ZB-WAX (15m X0.32mm X 0.50 μ m)
- Injector/Detector Temps: 230°C
- Injection Volume: 3 μ L
- Constant Helium Flow at 3.2 mL/min
- GC Temp Program:
100°C to 260°C at 15°C/min – Hold 10 min

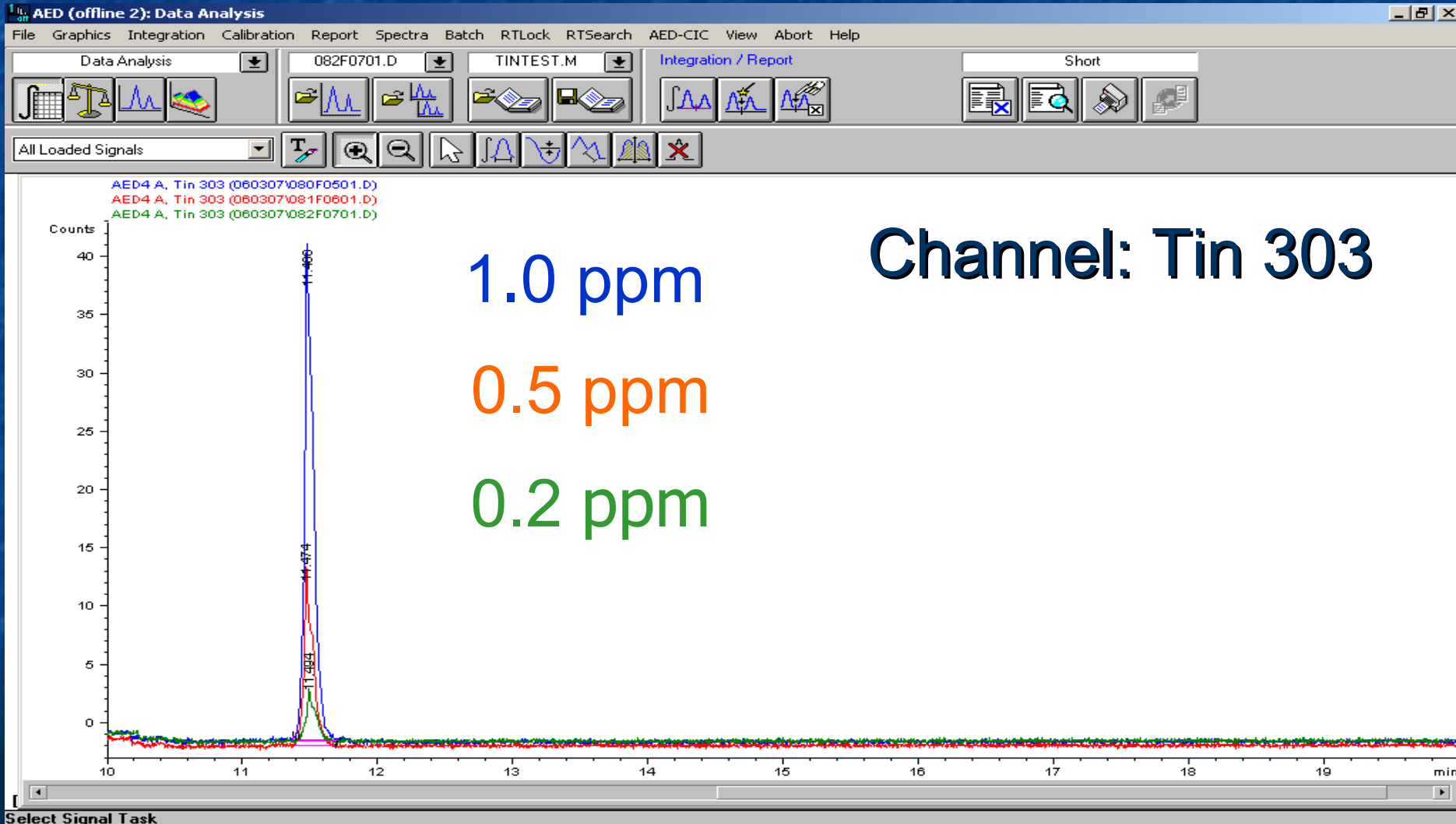


Total run time 20.67 min

Fenbutatin Oxide Standards



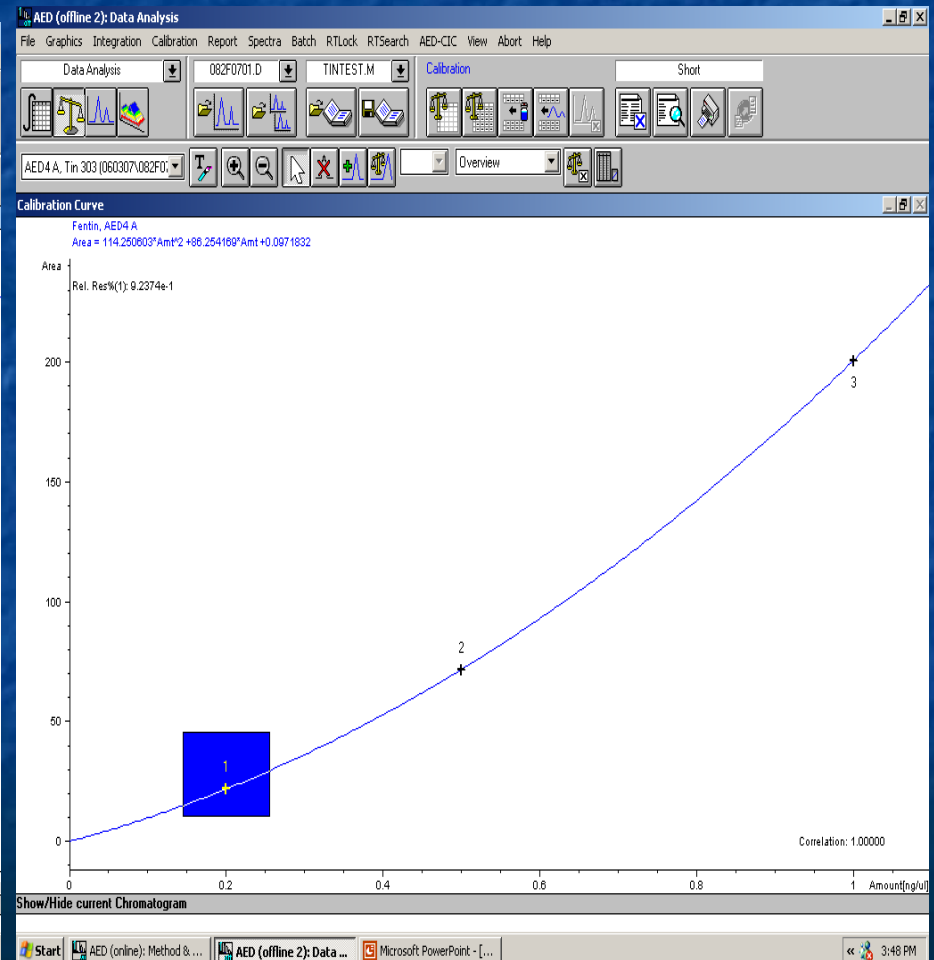
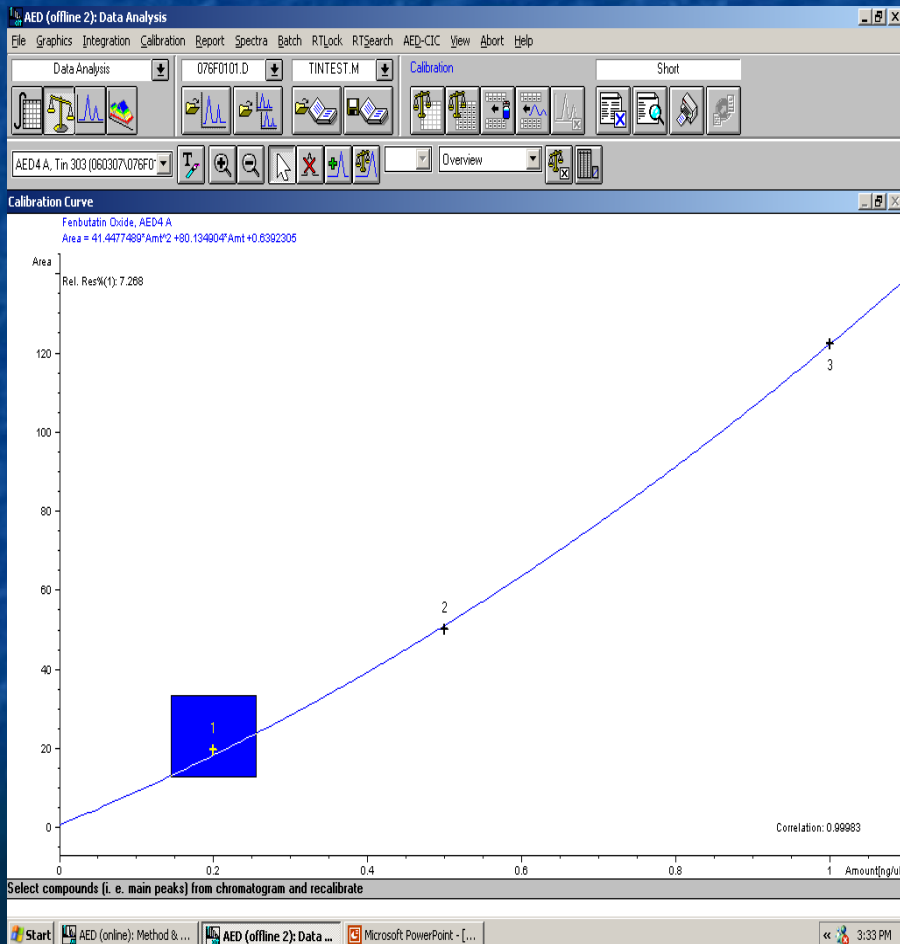
Fentin Standards



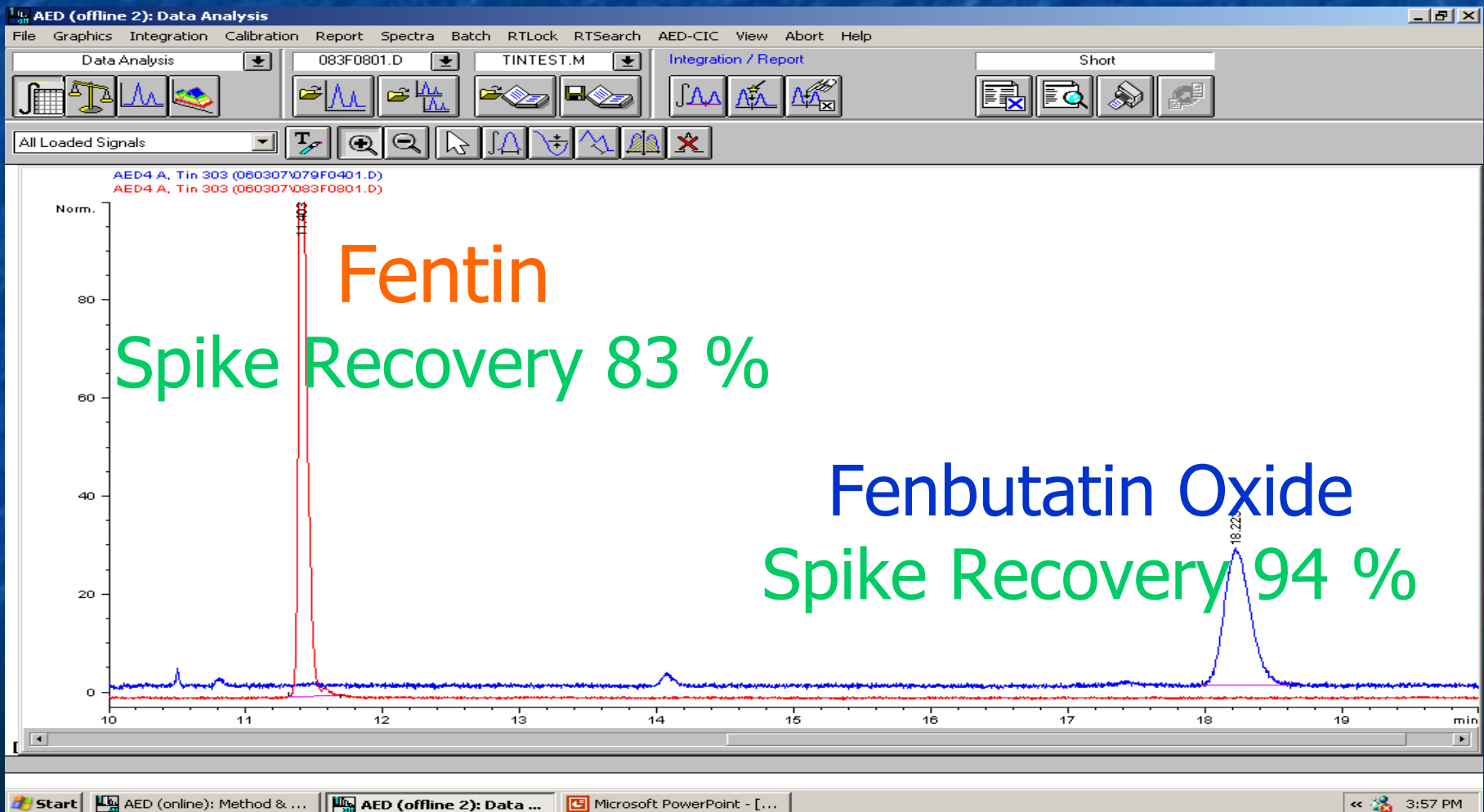
Calibration Curves

Fenbutatin Oxide

Fentin



Rice Sample Spiked at 0.1 ppm



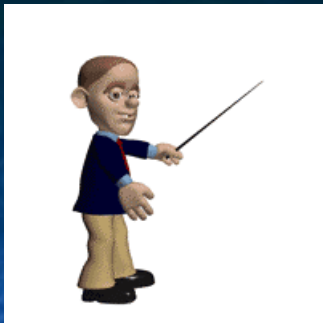
Screening for Inorganic Bromide

Wet Chemistry Method

- Static digestion at room temperature for **16 hours**
- Ashing in Muffle furnace for 3 hours
- Titration with 0.01 N Sodium Thiosulfate

AED Method

- Reaction with Propylene Oxide
- The resulting Bromopropanol is analyzed by AED Bromine 478 channel

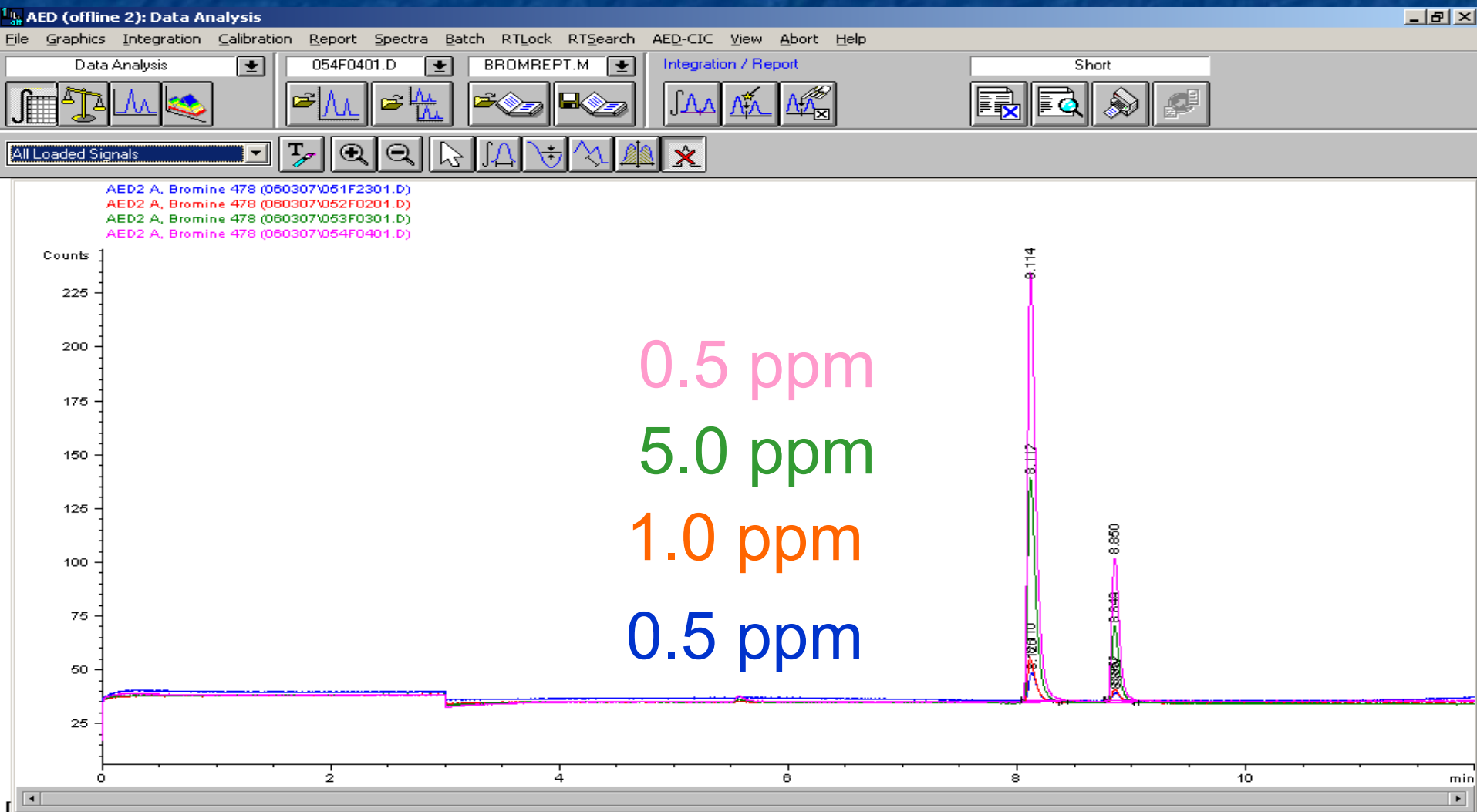


Method Conditions

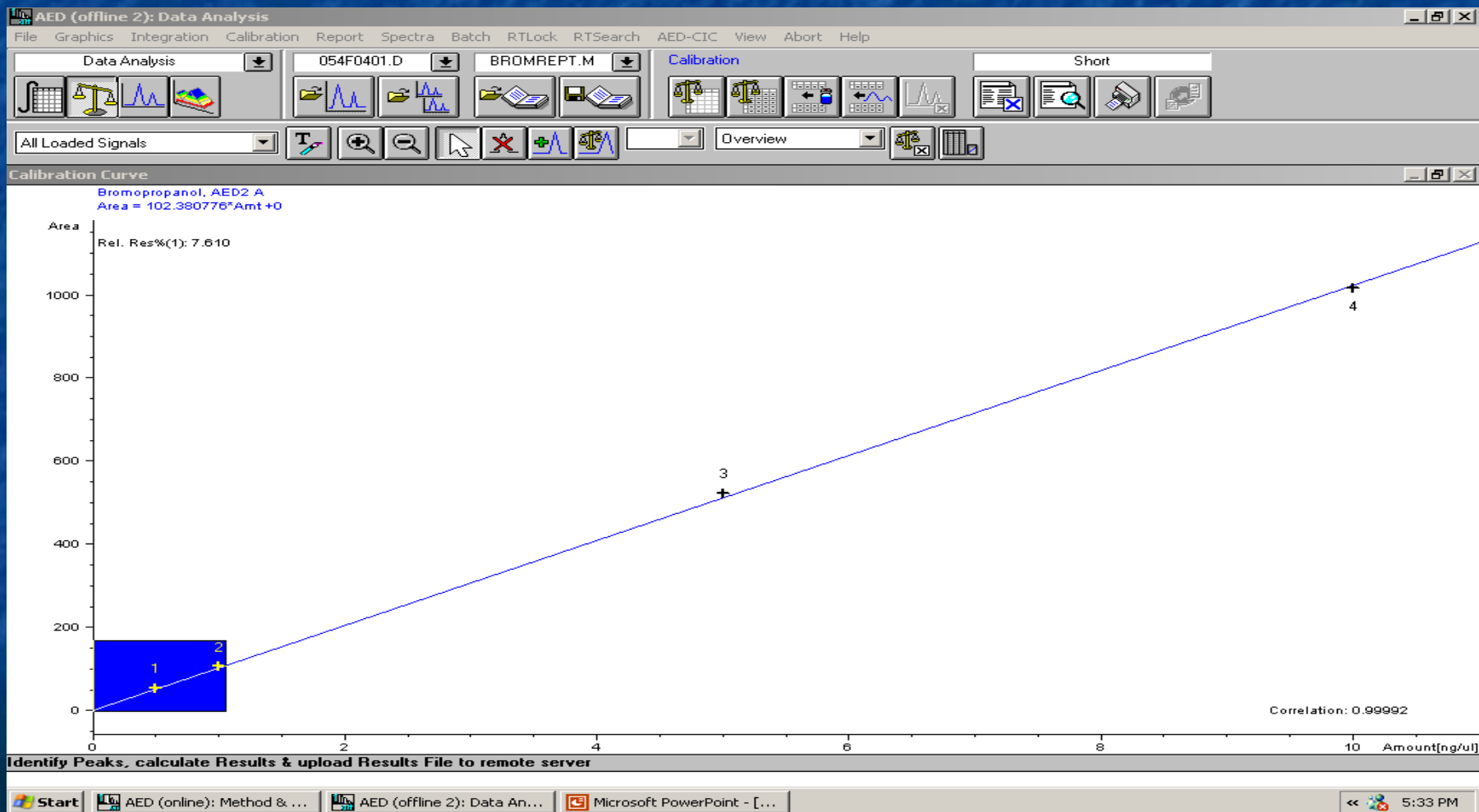
- HP6890 + GC with AED Detector, Channel Bromine 478
- Phenomenex ZB-WAX (15m X0.32mm X 0.50 μ m)
- Injector/Detector Temps: 120° C
- Injection Volume: 3 μ L
- Constant Helium Flow at 2.3 mL/min
- GC Temp Program:
50° C to 150° C at 10° C/min – Hold 1 min

Total run time 12 min

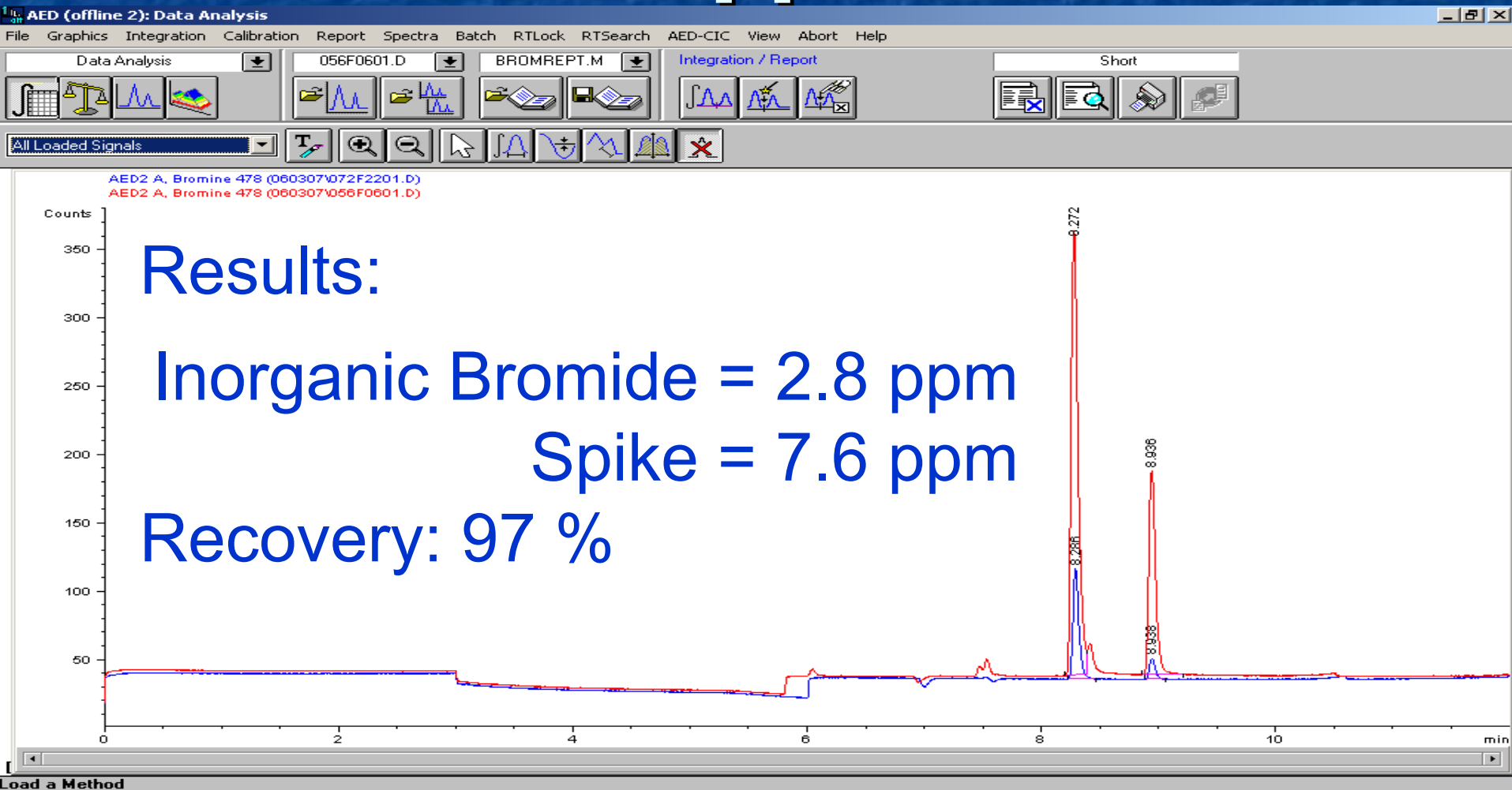
Bromopropanol Standards



Calibration Curve



Wheat Sample Spiked at 5 ppm



Conclusion

The GC/AED provides:

- Capability of screening for hundreds of pesticides regulated by different countries
- Quick identification of compounds using Retention Time Locking software
- Great selectivity reducing the matrix interferences
- Accurate quantification of targets at low levels in matrix