Dispersive or column SPE for better cleaning up of cereal extracts for further GC-MS determination

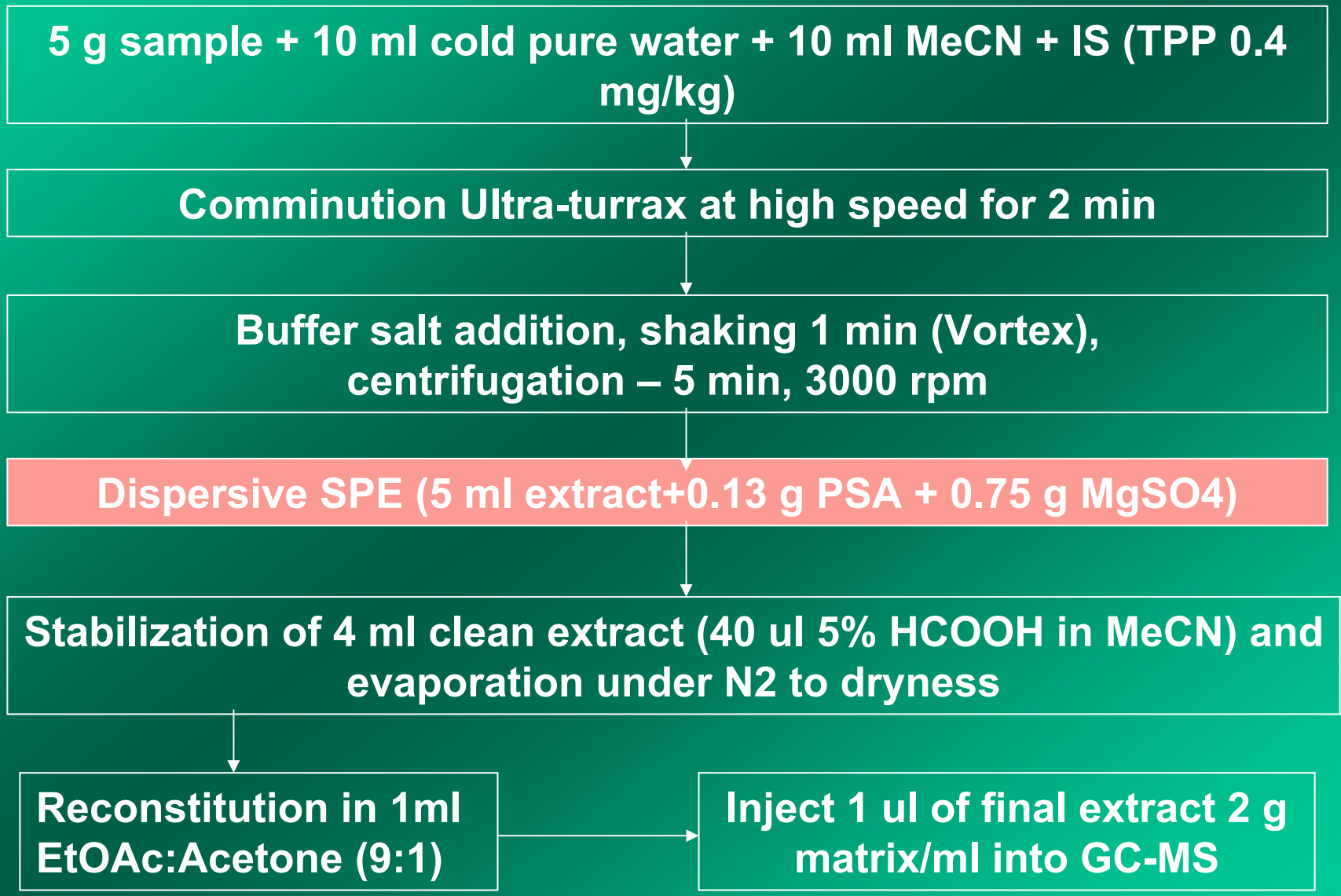
BG experience in EUPT-C2

Rositsa Mladenova, PhD

Plant Protection Institute

National Service for Plant Protection

Procedure used during PT participation



Gas chromatographic system

Thermo Finnigan Trace GC ultra with Finnigan Trace DSQ MS

T injector 230°C

T transfer line 250°C

T ion source 220°C

1 ml/min const carrier flow

Splitless time – 1 min

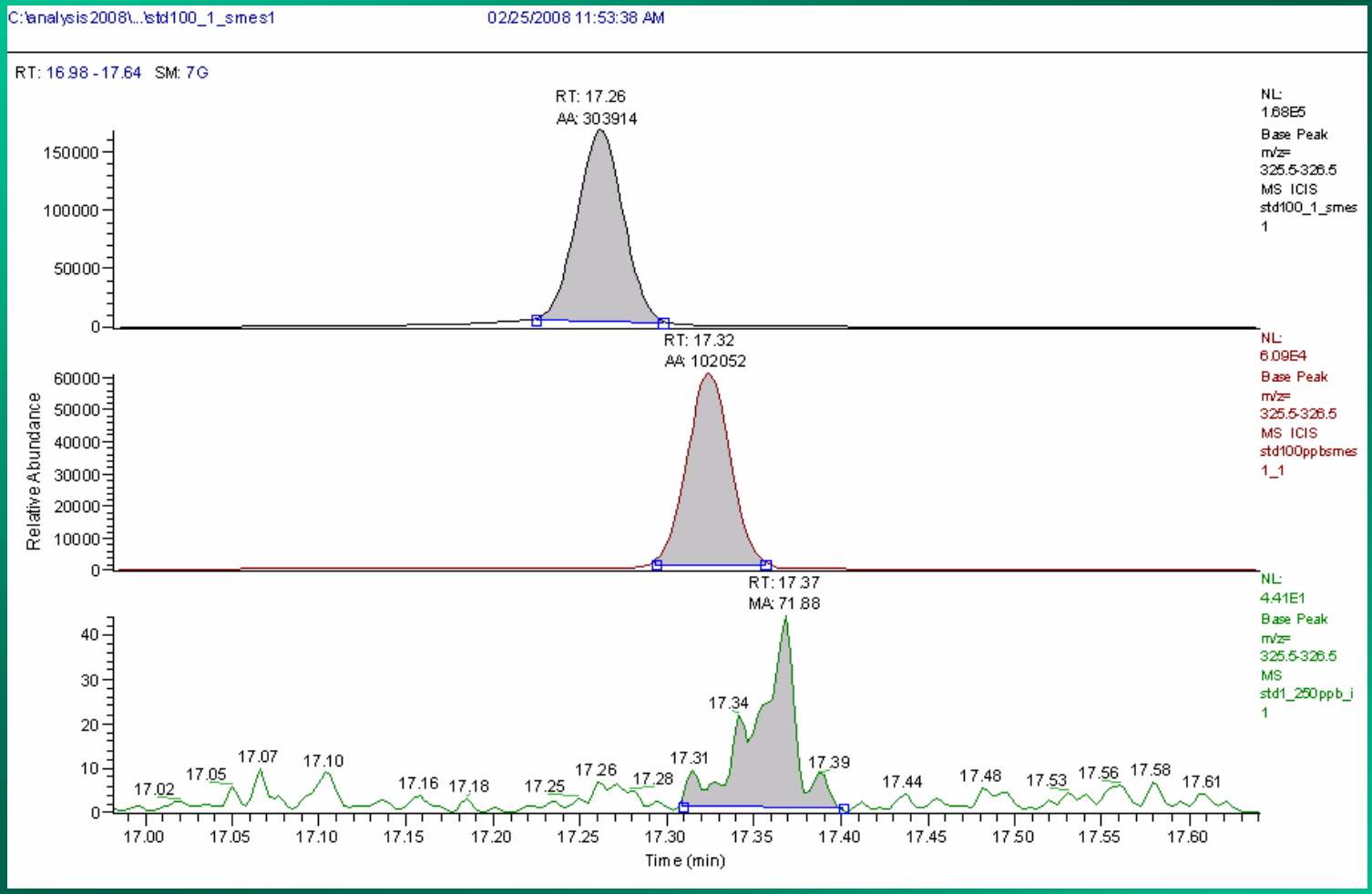
Oven temperature program

70°C /1 min $\xrightarrow{30°C/min}$ 190°C /1 min $\xrightarrow{5°C/min}$ 235°C $\xrightarrow{15°C/min}$ 280°C /6 min $\xrightarrow{30°C/min}$ 295°C /4 min

Results obtained by the lab

Pesticide	Assigned value, mg/kg	Submitted value, mg/kg	Z-score
Azoxystrobine	0.239	0.284	0.8
Bifenthrin	0.087	0.087	0.0
Alpha-cypermethrin	0.079	0.069	-0.5
Chlorpyrifos-methyl	0.130	0.143	0.4
Difenoconazole	0.169	0.191	0.5
Epoxyconazole	0.176	0.169	-0.2
Malathion	0.162	0.161	0.0
Pirimicarb	0.038	0.042	0.5
Prochloraz	0.239	0.303	1.1
Spiroxamin	0.075	0.169	5.0
Trifloxystrobin	0.439	0.528	0.8

MS pollution



At the beginning of PT

After 60 injections

After 130 injections - end

Further changes in the method for cereals

CEN/TC 275/WG 4 N 0236 - QuEChERS method

1.Removal of co-extracted fat, wax, sugars (e.g. for cereals, citrus fruits) – by Freezing out

2.Cleanup with amino-sorbent („Dispersive SPE“ with PSA) - For each 1 ml of extract 150 mg PSA and 900 mg $MgSO_4$ for additional cleaning

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Andrea Hercegová
Milena Dömötörová
Dáša Kružlicová
Eva Matisová

Institute of Analytical Chemistry, Slovak University of Technology, Faculty of Chemical and Food Technology, Radlinského, Bratislava, Slovak Republic

Original Paper

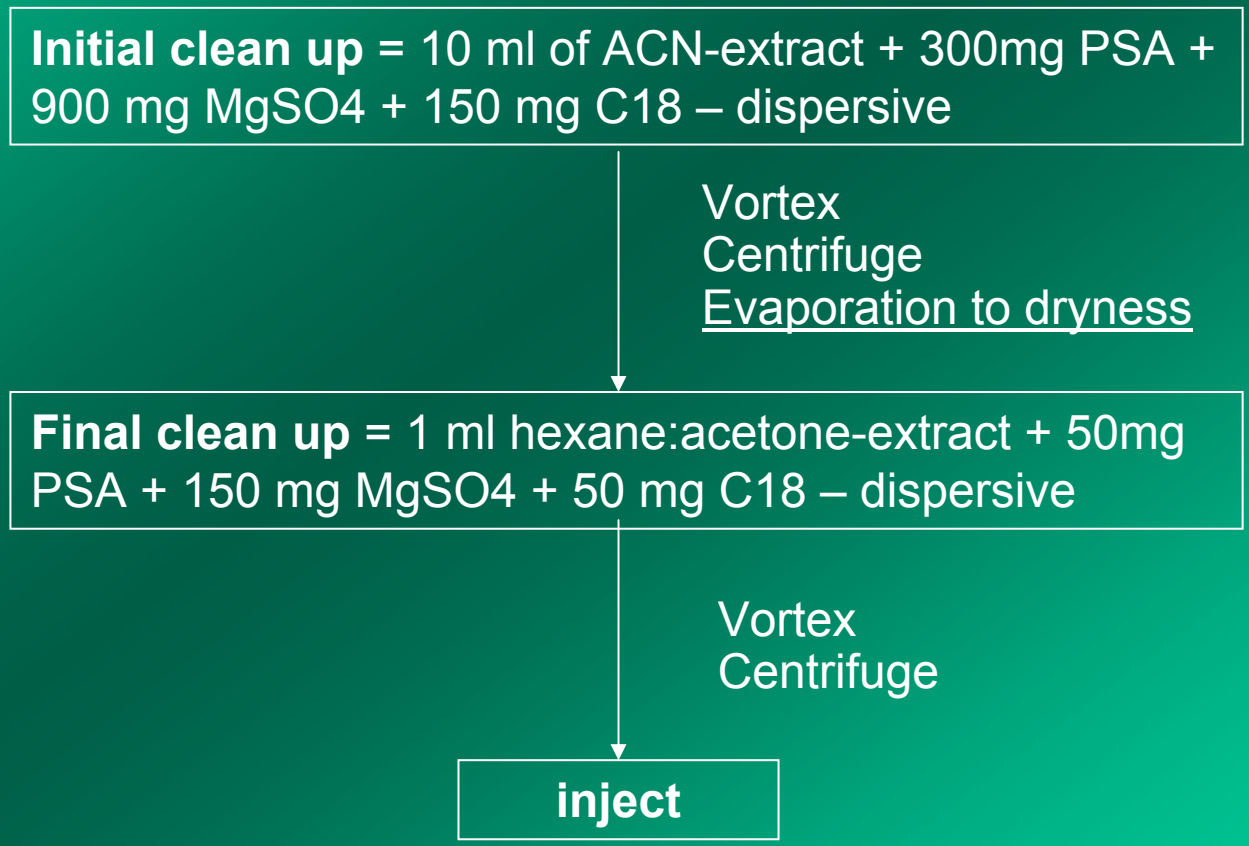
Comparison of sample preparation methods combined with fast gas chromatography – mass spectrometry for ultratrace analysis of pesticide residues in baby food

Four sample preparation techniques were compared for the ultratrace analysis of pesticide residues in baby food: (a) modified Schenck's method based on ACN extraction with SPE cleaning; (b) quick, easy, cheap, effective, rugged, and safe (QuEChERS) method based on ACN extraction and dispersive SPE; (c) modified QuEChERS method which utilizes column-based SPE instead of dispersive SPE; and (d) matrix solid phase dispersion (MSPD). The methods were combined with fast gas chromatographic-mass spectrometric analysis. The effectiveness of clean-up of the final extract was determined by comparison of the chromatograms obtained. Time consumption, laboriousness, demands on glassware and working place, and consumption of chemicals, especially solvents, increase in the following order QuEChERS < modified QuEChERS < MSPD < modified Schenck's method. All methods offer satisfactory analytical characteristics at the concentration levels of 5, 10, and 100 μg/kg in terms of recoveries and repeatability. Recoveries obtained for the modified QuE-

<u>Modified QuEChERS</u> – after first extraction of the upper layer was transferred onto an SPE column filled with acetone-conditioned 0.5 g of NH2-sorbent covered with 1 cm layer of MgSO4

Multi-residue Pesticide Analysis in Lettuce by a Modified QuEChERS Extraction and Ion Trap GC/MS/MS Analysis

David Steiniger, Jessie Crockett Butler, Eric Phillips
Thermo Fisher Scientific, Austin, Texas USA



Comparison of recovery by Hercegova et al. and Thermo modified procedures (at level 0.1 mg/kg)

pesticide	Hercegova et al.	Thermo
Chlorpyrifos-methyl	91	83
Vinclozoline	86	88
Epoxiconazol	89	88
Bifenthrin	89	90
Metconazol	79	85
Pyrazophos	86	90
Permethrin	90	86
Cypermethrin	81	85
Difenconazol	71	86

Modification of the analytical procedure

5 g sample + 10 ml cold pure water + 10 ml MeCN + IS (TPP 0.4 mg/kg)

↓

Comminution Ultra-turrax at high speed for 2 min

↓

Buffer salt addition, shaking 1 min (Vortex), centrifugation – 5 min, 3000 rpm

↓

Freezing out – overnight, column SPE (5 ml extract+0.5 g PSA + 1 cm layer MgSO4) elution with 10 ml acetone

↓

Evaporation under N2 to dryness

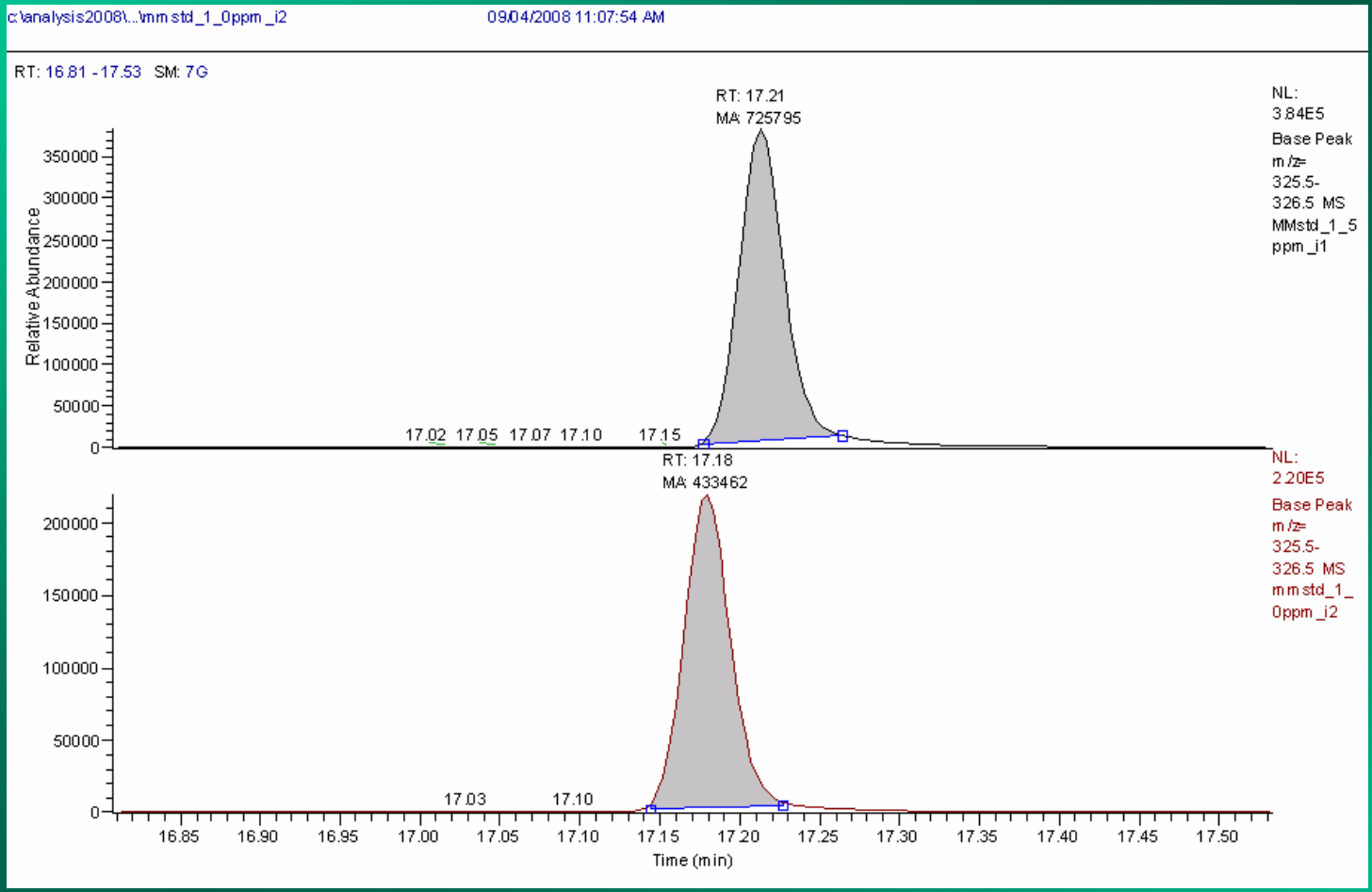
↓

Reconstitution in 1ml toluene

→

Inject 1 ul of final extract 2.5 g matrix/ml into GC-MS

MS status with modified procedure



After MS cleanning

After 60 injections

Comparison of MS pollution after 60 injections

Correlation of TPP peak intensity (I_0/I_{60})	Original QuEChERS	Modified QuEChERS
	3.0	1.7