# SPECIFIC PROTOCOL

## for the 7th EU Proficiency Test on Pesticides requiring Single Residue Methods

## EUPT – SRM7 (2012)

(last updated: 08.05.2012)

## Introduction

This protocol is complementary to the **"General Protocol for EU Proficiency Tests for Pesticide Residues in Food and Feed"** covering all EUPTs.
The EUPT-SRM7 is organised by the EU Reference Laboratory for pesticides requiring Single Residue Methods (EURL– SRM) and deals with the analysis of SRM-pesticides in dry lentils. This EUPT is to be performed by all National Reference Laboratories for Single Residue Methods (NRL-SRMs) as well as by all official EU laboratories (OfLs) involved in official pesticide residue controls as far as their scope overlaps with that of the EUPT-SRM7. The commodity "dry lentils" is to be considered as representative for commodities with "high starch and/or protein content and low water and fat content" (see **SANCO document 12495/2011**). A **Tentative List of obliged labs for EUPTs in 2012** has been published in the EURL-website. Obliged labs not intending to participate in this EUPT were requested to state the reasons for non-participation.

## Test Items (Test Materials)

This EUPT deals with the analysis of pesticide residues in **dry lentils**.

Participants will receive two bottles containing:

1) ca. 400 g **Spiked-Test Item**, containing spiked pesticides from the **Target Pesticide List**
2) ca. 400 g **Blank-Test Item**, that can be used for recovery experiments as well as for the preparation of matrix-matched calibration standards

Using randomly chosen bottles, the Organizers will check the spiked Test Item for sufficient homogeneity as well as for stability under conditions representing sample shipment and storage during the duration of the test. The blank Test Item will be also checked to prove that the target analytes are not contained at any relevant levels. All these tests will be conducted by the EURL-SRM that is ISO 17025 accredited.

## Analytical parameters

The Test Item contains several pesticides from the **Target Pesticide List**.
Laboratories should carefully read the Target Pesticides List, where important information about reporting of results, as well as the **Minimum Required Reporting Levels (MRRLs)** is given.
The MRRL values will be used to help identify false positive and false negative results and for the calculation of z-scores for false negatives.
It should not be assumed that only pesticides registered for use on lentils are present in the Test Item.

For practical reasons, the residue definitions listed in the Target Pesticides List, in some cases, do not fully match with the legal ones.

## Shipment of Test Items

**The Test Materials are planned to be shipped on 23 April, 2012.**
Test Material will be shipped frozen and packed in thermo-boxes together with a freeze gel pack. The organisers will aim to ensure that all participating laboratories will receive their shipments on the same day.

Prior to shipment a reminder will be sent to the participating laboratories by e-mail.

Laboratories must make their own arrangements for the receipt of the package. They should inform the Organiser of any public holidays in their country/city during the week of the shipment, and must make the necessary arrangements to receive the shipment, even if the laboratory is closed.

## Instructions on handling of Test Items

Once received, the Test Items should be stored deep frozen (at -18°C or lower) until analysis to avoid any possible deterioration/spoilage and to minimize pesticide degradation.

**The Test Material should be mixed thoroughly in its entirety before a portion is taken for analysis.**

All participants should use their own routine standard operating procedures for extraction, clean-up and analytical measurement as well as their own reference standards for identification and quantification purposes. Considering the available amount of Test Item, laboratories employing methods requiring large analytical portions are advised to scale them down. As the test material is already milled and sufficiently homogeneous, method sensitivity is the only major factor to consider when deciding about the size of the analytical portion.

The homogeneity tests will be conducted using 2-5 g of Test Item depending on the analyte. As variability increases with decreasing analytical portion size, sufficient homogeneity can only be guaranteed for sample portions that are equal or larger than those employed for the homogeneity test.

## Results submission website

Sample receipt acknowledgement, analytical results and method information are to be submitted via the **EUPT-SRM7 Result Submission Website** (**http://thor.dfvf.dk/eupt-SRM7**) that will be **accessible from 24 April 2012 onwards for sub-page 0 and 27 April 2012 onwards for sub-pages 1-3**.

This website also contains a link to specific instructions on how to enter the required data in the various submission forms (sub-pages).

**To access the data submission forms participants must use their unique login data (username and password) given in the confirmation e-mails sent to the laboratories upon registration.**

**The deadline for result submission is 29 May 2012 at 15.00 CET**

## - Sample Receipt and Acceptance (Sub-Page 0)

Once the laboratory has received the Test Items it must report to the organiser, via the **EUPT-SRM7 Result Submission Website** (sub-page 0) the date of receipt, the condition of the Test Item, and its acceptance. The deadline for acceptance is the 26 April 2012. If a laboratory does not respond by this deadline the Organisers will assume that the Test Items have been received and accepted. If any participants have not received the Test Items by the 27 April at noon, they must inform the Organiser immediately by e-mail (**EUPT-SRM@cvuas.bwl.de**), so that a new shipment can be managed.

## - Reporting Qualitative and quantitative Results (Sub-Page 1 and 2)

To report their results, laboratories must access the **EUPT-SRM7 Result Submission Website**.

**All results must be reported on the above website by the 29.May 2012 at 15:00 (3 p.m.) CET, at the latest.** The website will not be accessible after this deadline and all results submitted afterwards will be not included in the statistical treatment or in the final report.

Before entering the results, please study the **Target Pesticide List**. The residue definitions are not given on the Result Submission Website carefully.

The following fields will be available for reporting the quantitative results:

- "**Concentration in mg/kg":** the pesticide concentrations that would be reported in routine work. Recovery-corrected results should be reported only where this reflects the normal lab procedure, otherwise the non-recovery-corrected result should be reported. Results should not be reported where a pesticide was not detected, or was detected below the RL (Reporting Limit) of the laboratory or the MRRL. Results reported as "<RL" will be considered as „Not Detected".

  The residue levels of the pesticides must be reported in mg/kg using the following **significant figures**:
  - Levels <0.010 mg/kg to be expressed to 2 significant figures, e.g. 0.0058 mg/kg;
  - Levels ≥ 0.010 mg/kg to be expressed to 3 significant figures, e.g. 0.156, 1.64, 10.3 mg/kg.

- **"Conc. in blank in mg/kg":** concentration values of any pesticides from the Target Pesticides List determined in the blank (even at levels below the MRRL).

- **"Experience with this compound".** Use the dropdown-menu to indicate for how many years you have been analysing for each compound using the method applied in this EUPT.

- **"Is your result recovery-corrected?":** Please specify, via dropdown-menu, whether the reported result was recovery-corrected and the of recovery-correction approach used.

- **"Recovery figure (in %)":** Here labs can report any recovery figures (in %) obtained for the analyte in question. If a recovery factor was used to correct for recovery, the recovery figure (in %) used for the calculation MUST be reported.

Additional information on how each recovery figure was derived will be asked in separate fields.

### - Reporting Information on Analytical Methodology (Sub-Page 3)

In **sub-page 3 within of** the "**EUPT-SRM7 Result Submission Website** all laboratories must provide information on the analytical method(s) employed to analyze the pesticides for which results where reported.

The laboratories are urged to thoroughly fill-in this important information in order to minimize the administrative burden of collecting this information a posteriori.

### - Reporting missing information after result submission deadline (Sub-page 4)

In case of false negative results the affected laboratories will be asked to provide details on the methodology used after the deadline for result submission. This can be done by accessing sub-page 4 within the **EUPT-SRM7 Result Submission Website**.
The dates sub-page 4 will be accessible will be announced in due time. Where sub-page 4 is empty when accessed, no further information is needed from you the lab.
**If no sufficient information on the methodology used is provided, the Organiser reserves the right not to accept the analytical results reported by the participant.**

## Follow-up actions

According to instructions by the Commission, the "**Protocol for management of underperformance in comparative testing and/or lack of collaboration of National Reference Laboratories (NRLs) with Community reference laboratories (CRLs) activities**" will be followed for NRLs.

## Documents

All documents relating to EUPT–SRM7 can be found in the **EURL-Document Repository (CIRCA/FIS-VL).**
Links to the documents can be found in the **EUPT-SRM7 Website.**
For further information please contact the organizers **EUPT-SRM@cvuas.bwl.de**

## Participation fees and payment details

To cover the costs of production, handling and shipment of the Test Materials the following participation fees will be charged to each participating laboratories,:

- OfLs (including NRLs) from EU countries, EU-candidate countries and EFTA countries: 175 €
- Labs based in third countries: 250 €

All laboratories that have been accepted to participate will be sent an invoice to the "invoice address" stated in the registration form.

Payment is expected to be made prior to the scheduled shipment date. **No Test Material will be sent to labs from which no payment has been received by the shipment date.**
Details of payment will be given in the invoices.

To facilitate tracking of money transfer mind to include your special payee identification text (= invoice number) as shown in the invoice. **Payments without this identification text may not be considered as paid!**

**Bank Details:**

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| **Bank account holder:** | **CVUA Stuttgart** |
| **Bank Name :** | **Baden Wuerttembergische Bank** |
| **IBAN:** | **DE 72 6005 0101 7469 5341 03** |
| **BIC/SWIFT:** | **SOLADEST** |
| **Payee identification text:** | **See invoice (VERY IMPORTANT!)** |

## Calendar

(see also **http://www.crl-pesticides.eu/library/docs/srm/EUPT_SRM7_Calendar.pdf** )

| Activity | Who ? | Dates |
|---|---|---|
| Opening of the **EUPT-SRM7 Website** with links to all relevant documents and to the **EUPT-General Protocol** | EURL-SRM | Jan 2012 |
| Distribution of "**EUPT-SRM7-Calendar**" | EURL-SRM | Jan 2012 |
| Distribution of "**Target Pesticides List**", | EURL-SRM | Jan 2012 |
| Distribution of "**Announcement/Invitation-Letter**" | EURL-SRM | Jan 2012 |
| Accessibility of "**EUPT-Registration Website**"<br>To **sign up for EUPT-SRM7** and to **explain the reasons for non-participation** | **- Obliged OfLs from EU-MSs** (regardless if not participating)<br>- **OfLs from EFTA countries** & **EU-candidate countries** | **8 Feb – 27 Feb 2012** |
| Distribution of "**EUPT-SRM7-Specific Protocol**" | EURL-SRM | Mar 2012 |
| Preparation of EUPT-SRM7-Test Material | EURL-SRM | Oct 2011 - Mar 2012 (preliminary tests) |
| | | Mar 2012 (Spiking / Homogenization) |
| Homogeneity tests | EURL-SRM | Mar-Apr 2012 |
| Stability tests | EURL-SRM | Apr-May 2012 |
| **Distribution of EUPT-SRM7 Test Material** (Reminder to the labs about upcoming shipment) | EURL-SRM | **23 Apr 2012** |
| **Deadline for Receipt and Acceptance of Test Material:** Online Submission of Form 0 (sub-page 0) | **Participating Labs** | **within 48 hr of receipt** |
| Activation of "**Result Submission Website!** | EURL-SRM | **27 Apr 2012** |
| **Deadline for Result Submission**<br>**Pesticide scope, Results, Method Information**<br>Submission of Form 1 – 3 (sub-pages 1 – 3) | **Participating Labs** | **29 May 2012**<br>**(at 15:00 CET)** |
| EUPT Evaluation Meeting | EUPT-Scientific Committee, DG-SANCO | Jul 2012 |
| Preliminary Report (only compilation of results) | EURL-SRM | Jul 2012 |
| Final Report | EURL-SRM | Nov 2012 |

# Contact information

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**Quality Control Group**

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