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Screening method validation of pesticide residues in cereals using GC-QTOF

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Introduction: GC-QTOF is a new detection technique in the field of screening for pesticides residue in food samples. The technique will enable screening of many pesticides compared to the MS/MS analyses commonly used. With the purpose to develop generic screening methods, the EURL-CF has purchased an Agilent QTOF 7200. Information on instrumentation can be seen in the box to the left.

Library: GC-QTOF presents other challenges than LC-QTOF, especially because the molecular ions typically do not survive. The compounds are fragmented in the ion source and currently no libraries with exact masses of the fragments are available. The EURL-CF will in cooperation with the EURL-FV create a library with exact masses of GC amendable pesticides. MS-interpreter in the NIST MS Library is one of the tools to identify the fragments and exact masses, see Figure 1

Method: Barley, rice, and wheat cereal samples with no pesticide residues were spiked at 0.01 and 0.05 mg/kg with a mixture of more than 300 pesticides. Six replicates of each cereal type were spiked (Figure 2). The samples were extracted by QuEChERS method and analysed by GC-QTOF. Together with the 24 spiked samples, 4 blank cereal samples (same cereals types) and 4 EUPT tests materials were extracted and cleaned up. Only 38 of the compounds have currently been evaluated.

Validation criteria: According to SANCO at least 95% of the recoveries samples should be detected, meaning that only 1 out of 20 spiked samples are allowed to be non-detected. The criteria to detect a compound in this validations was a slightly different from those listed in SANCO/12571/2013. See Figure 3.

Validation - spike: A screen dump of pirimiphos-methyl result showing the software and chromatogram at 0.01 mg/kg is shown in Figure 4. The validation results showed that of 36 of the 38 evaluated compounds was validated, 19 with Screening Detection Limit, SDL, at 0.05 mg/kg and 17 with SDL 0.01 mg/kg. The SDL was defined as the lowest concentration in which a pesticide could be detected with only one non-detect or less out of the 24 samples. All processing of data was done automatically by Mass Hunter in the Quantitative software. No manual assessment was done. See Table 1.

EUPT-C test materials: Results from the EUPT test materials showed very good agreement with the validation. The test materials contained 46 residues of 27 different pesticides in the range of 0.012-2.180 mg/kg. All pesticides were detected apart from one residue of lambda-cyhalothrin. However, this residue was below the SDL of 0.05 mg/kg. No false positives were seen. See Table 2

Table 1: Typical examples on validation data. Pink colored numbers are spiked spike level

Table 2: Screening result of EUPT-C2, C4, C5 and C8.