Determination of tranquilizers in kidney using LC-MSMS
1 Introduction

This method describes the analysis of six tranquillizers (chlorpromazine, acetopromazine, propionylpromazine, xylazine, haloperidol, azaperone), a metabolite (azaperol) and a β-blocker (carazolol) in samples of porcine kidney. The analytes are extracted from the kidney matrix using acetonitrile. After clean-up of the extract by solid phase extraction (SPE) the analytes are separated and detected by liquid chromatography connected via an electrospray interface to a triple quad mass spectrometric (LC-MSMS).

Since 2006, chlorpromazine is an Annex IV (banned) compound. The MRPL-value was reduced subsequently from 50 ppb to a lower level, in this case 5 ppb. The method is validated on this level for all six tranquillizers.

2 Apparatus

Any reference to type and/or product is only to inform the user and identify the equipment and does not imply exclusion of similar equipment.

2.1 Vortex, Vibrofix VF1
2.2 Evaporator, TurboVap Zymark
2.3 Centrifuge, Heraeus Sepatech Varifuge 3.OR
2.4 Sample concentrator, Techno
2.5 Ultrasonic bath, Branson 2200
2.6 Automatic pipets, Rainin
2.7 LC-MSMS
   • LC: Waters Acquity Ultra Performance, MSMS: Micromass Quattro Ultima Pt
   • LC Column: Waters Acquity UPLCTM BEH C18, 1.7 µm, 2.1 x 100 mm
   • Temperature column thermostat: 65ºC
   • Autosampler temperature: 30ºC
   • Eluens A: mix 2.3 ml ammonia (25%) and 500 ml distilled water
   • Eluens B: mix 2.3 ml ammonia (25%) and 500 ml acetonitrile
   • injection volume: 20 µl
   • Flow: 0.5 ml/min.
   • Solvent delay: 0-1.5 min., 6-10 min.

Table 1. Gradient LC-system, total runtime: 10 minutes

<table>
<thead>
<tr>
<th>Time (min.)</th>
<th>Eluens A</th>
<th>Eluens B</th>
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</thead>
<tbody>
<tr>
<td>1</td>
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<td>0.0</td>
</tr>
<tr>
<td>4</td>
<td>8.1</td>
<td>70</td>
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Table 2. Tune parameters product scan measurements

<table>
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<tr>
<th>Parameter</th>
<th>Value</th>
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<tbody>
<tr>
<td>Ionization mode</td>
<td>ES+</td>
</tr>
<tr>
<td>Capillary (kV)</td>
<td>3.20</td>
</tr>
<tr>
<td>Cone (V)</td>
<td>50</td>
</tr>
<tr>
<td>component</td>
<td>[M+H]+</td>
</tr>
<tr>
<td>--------------------</td>
<td>---------</td>
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<tr>
<td>chlorpromazine</td>
<td>319.1</td>
</tr>
<tr>
<td>acetopromazine</td>
<td>327.3</td>
</tr>
<tr>
<td>propionylpromazine</td>
<td>341.1</td>
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<td>azaperone</td>
<td>328.1</td>
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<tr>
<td>azaperol</td>
<td>330.2</td>
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<td>299.2</td>
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<td>376.2</td>
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<td>chlorpromazine-d3</td>
<td>322.1</td>
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<tr>
<td>haloperidol-d4</td>
<td>380.1</td>
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</table>

### 3 Safety and environment

#### 3.1 Safety

The RIVM has designed safety rules for working in the laboratory. These rules can be found on the RIVM intranet.

#### 3.2 Waste disposal
4 Chemicals and reagents

All chemicals including standards and solutions are defined quality. Pure chemicals are “Pro Analyse” quality, water is milli-Q quality.

4.1 Materials
SPE Strata-X-C 500 mg/6 ml, cation mixed mode polymer (Phenomenex)
15 ml screw cap glass tubes (Schott-Omultiabo)

4.2 Reagents
- Haloperidol-d4 (100 µg/ml in methanol), Cambridge Isotopes
- Chlorpromazine-d3 (100 µg/ml in methanol), Cambridge Isotopes
- Azaperol, RIVM
- Azaperon, RIVM
- Carazolol, Boehringer Mannheim
- Xylazine, Sigma-Aldrich chemie
- Haloperidol, Sigma Aldrich chemie
- Acetopromazine, Sigma Aldrich chemie
- Propionylpromazine, Riedel-de Haen
- Chlorpromazine, Sigma Aldrich chemie
- Ethanol, Merck
- Methanol, Merck
- Potassium dihydrogen phosphate, Merck
- Hydrochloric acid (25%), Merck
- Acetonitrile HPLC supra gradient (Biosolve)
- Ammonia (25%), Merck

4.2.2 Working solutions
1 M HCl: dilute 13 ml hydrochloric acid up to a final volume of 100 ml with water
30 % acetonitrile: mix 30 ml acetonitrile and 70 ml water
PH 7 buffer 50 mM: dissolve 6 g of NaH₂PO₄ in 900 ml water; titrate to pH 7 with KOH/NaOH and HCl as required; make up to 1 litre with water
5% ammonia in 70% methanol/acetonitrile (1:1): mix 70 ml methanol/acetonitrile (1:1) with 20 ml ammonia 25% and 10 ml water

4.3 Samples
4.3.1 This method was developed for porcine kidney.
4.3.2 After defrosting, fat and other non-kidney tissue is removed. Kidney should be mashed in a kitchen machine and stored at -20°C in a polystyrene pot.

4.4 Preparation of standards
- Stock solutions of each component, containing 1 mg/ml of the component
- Working solutions of each component (including internal standards), containing 10 ng/µl
- Working solutions of each component (including internal standards), containing 1 ng/µl
- Mixture of components, containing 0.1 ng/µl of each component
- Mixture of internal standards containing 0.1 ng/µl of each component

4.4.2 Stock solutions of the components containing 1 mg/ml are prepared in ethanol and stored in an amber vial at −20°C. These solutions are stored for a maximum period of 2 years.
4.4.3 Working solutions containing 10 ng/µl are prepared in ethanol (in 2 steps of 1:10) by diluting 1 ml of the stock solution (4.4.2) in a volumetric flask of 10 ml. These solutions are stored for a maximum period of 12 months.

4.4.4 Working solutions containing 1 ng/µl are prepared in ethanol by diluting 1 ml of the working solution (4.4.3) in a volumetric flask of 10 ml. These solutions are stored for a maximum period of 12 months.

4.4.5 A mixture of components is prepared in ethanol by diluting 1 ml of the working solutions (containing 1 ng/µl) in a volumetric flask of 10 ml. A mixture of internal standards is prepared in ethanol by diluting 1 ml of the working solutions (containing 1 ng/µl) in a volumetric flask of 10 ml. These solutions are stored for a maximum period of 12 months.

4.5 Controls and blanks

4.5.1 A set of calibration standards in kidney is prepared by adding the specified volumes of the calibration standard mixtures (table 4) to 1 ± 0.05 g of kidney.

Table 4. Preparation of calibration curve and spiking of samples.

<table>
<thead>
<tr>
<th>Calibration curve</th>
<th>amount of µl standard mix added to 1 gram of kidney tissue</th>
<th>amount of µl ethanol added to 1 gram of kidney tissue</th>
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</thead>
<tbody>
<tr>
<td>spike level</td>
<td></td>
<td></td>
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<tr>
<td>mix contains 0.1 ng/µl for each component</td>
<td></td>
<td></td>
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<tr>
<td>blank</td>
<td>0</td>
<td>100</td>
</tr>
<tr>
<td>1 ppb spike</td>
<td>10</td>
<td>90</td>
</tr>
<tr>
<td>2.5 ppb spike</td>
<td>25</td>
<td>75</td>
</tr>
<tr>
<td>5 ppb spike</td>
<td>50</td>
<td>50</td>
</tr>
<tr>
<td>7.5 ppb spike</td>
<td>75</td>
<td>25</td>
</tr>
<tr>
<td>10 ppb spike</td>
<td>100</td>
<td>0</td>
</tr>
</tbody>
</table>

Each standard and sample is also spiked with 5 ppb (= 50 µl) internal standard mix (4.4.5)

5 Procedure

5.1 Destruction, extraction and clean up

5.1.1 Weigh 1±0.05 g kidney tissue in a glass tube (15 ml) with screw cap.

5.1.2 Add 50 µl internal standard (chlorpromazine-d3 and haloperidol-d4 mix, 0.1 ng/µl)

5.1.3 Add correct amount of standard mix and/or ethanol (see schedule at 5.5.2)

5.1.4 Add 5 ml acetonitrile

5.1.5 Vortex the tubes for 1 minute and place tubes in an ultrasonic bath for 5 minutes

5.1.6 Place tubes in -20ºC for 1 hour

5.1.7 Centrifuge the tubes for 5 minutes at 3600 rpm and 4ºC

5.1.8 Decant the fluid in a clean screw cap glass tube
5.1.9 Add 6 ml phosphate buffer pH 7 and mix

5.2 Clean up with Strata X-C SPE columns (screening method)
5.2.1 Condition the SPE columns with 12 ml methanol
5.2.2 Wash with 12 ml buffer pH 7.
5.2.3 Transfer the extract to the SPE column.
5.2.4 Wash with 12 ml H₂O.
5.2.5 Wash with 12 ml methanol.
5.2.6 Dry the SPE column for one minute under full vacuum prior to applying elution solvent.
5.2.7 Elute with 12 ml 5% ammonia in 70% methanol/acetonitrile 1:1, allow elution solvent to soak into sorbent for 0.5 - 1 min.
5.2.8 Evaporate to dryness under a stream of nitrogen at 55°C.
5.2.9 Dissolve the residue in 500 µl EtOH, 5 min. ultrasonic and vortex.
5.2.10 Centrifuge the residue for 5 minutes at 7000 rpm.
5.2.11 Transfer 400 µl of the top layer in an autosampler vial and evaporate to dryness under nitrogen at 55°C
5.2.12 Dissolve the residue in 50 µl acetonitrile 30% and heat the vials in a heating block to 37°C
5.2.13 Inject 20 µl on the LC-MSMS

6 Calculation
The selected ion area of the standard is divided by the selected ion area of the internal standard (see table 5) for the combination of compound and internal standard. The ratio is the response variable.
A calibration curve is constructed by linear curve fitting using least squares linear regression calculation.
Unknown concentrations are calculated by interpolation.

7 Validation and Measurement uncertainty
The method described in this SOP was validated conform ARO/475. The validation level used for the validation was 5 ppb for all compounds.
A summary of the validation results is given in the table below. An overview of the validation results is enclosed as a supplement.

Table 5. Performance characteristics of the method

<table>
<thead>
<tr>
<th>Compound</th>
<th>transition</th>
<th>internal standard</th>
<th>CCα</th>
<th>CCβ</th>
<th>Accuracy at 5 ng/g (%)</th>
<th>measurement uncertainty (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>chlorpromazine</td>
<td>319.1 &gt; 58.2</td>
<td>chlorpromazine-d3</td>
<td>0.32</td>
<td>0.54</td>
<td>118.2</td>
<td>32</td>
</tr>
<tr>
<td></td>
<td>319.1 &gt; 86.2</td>
<td></td>
<td>0.33</td>
<td>0.56</td>
<td>122.7</td>
<td>18</td>
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<tr>
<td>acetopromazine</td>
<td>327.3 &gt; 240.2</td>
<td>haloperidol-d4</td>
<td>0.62</td>
<td>1.05</td>
<td>64.8</td>
<td>32</td>
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</table>
### 327.3 > 254.2  
0.50  0.85  62.4  25

<table>
<thead>
<tr>
<th>Compound</th>
<th>M/z 1</th>
<th>M/z 2</th>
<th>M/z 3</th>
<th>M/z 4</th>
<th>M/z 5</th>
<th>M/z 6</th>
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</thead>
<tbody>
<tr>
<td>propionylpromazine</td>
<td>341.1 &gt; 239.2</td>
<td>341.1 &gt; 268.0</td>
<td>chlorpromazine-d3</td>
<td>0.54</td>
<td>0.91</td>
<td>54.2</td>
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<tr>
<td>azaperone</td>
<td>328.1 &gt; 121.3</td>
<td>328.1 &gt; 165.1</td>
<td>haloperidol-d4</td>
<td>0.62</td>
<td>1.05</td>
<td>57.6</td>
</tr>
<tr>
<td>azaperol</td>
<td>330.2 &gt; 149.2</td>
<td>330.2 &gt; 192.1</td>
<td>chlorpromazine-d3</td>
<td>1.77</td>
<td>3.01</td>
<td>28.4</td>
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<tr>
<td>carazolol</td>
<td>299.2 &gt; 116.4</td>
<td>299.2 &gt; 222.1</td>
<td>chlorpromazine-d3</td>
<td>2.01</td>
<td>3.42</td>
<td>20.5</td>
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<tr>
<td>xylazine</td>
<td>220.9 &gt; 90.4</td>
<td>220.9 &gt; 164.2</td>
<td>chlorpromazine-d3</td>
<td>2.67</td>
<td>4.55</td>
<td>14.6</td>
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<tr>
<td>haloperidol</td>
<td>376.2 &gt; 123.1</td>
<td>376.2 &gt; 165.0</td>
<td>haloperidol-d4</td>
<td>0.68</td>
<td>1.17</td>
<td>66.5</td>
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</table>

### 8 Quality control

#### 8.1 Calculation

Quantification is only valid if:
- The maximum of the signal originating from the analyte has an S/N ratio >3.
- In the blank control samples all the internal standards are visible.
- In the spiked control samples all components are visible (S/N ratio >10 for internal standards, >6 for the non-deuterated compounds).

#### 8.2 Process control

The process is controlled by calibration samples in kidney tissue and the calibration curve constructed from these values.

### 10 Relating documents

Anton Kaufmann, Bianca Ryser, Multiresidue analysis of tranquilizers and the beta-blocker Carazolol in meat by liquid chromatography/ tandem mass spectrometry

### Annex

**Validation Reports**

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**ResVal v.1.3 Validation Report (SCP 475)**

1. **General Information**

   - **Objective**: Coelenterate A (SCP 475)
   - **Participants**: Central station (SCP 475)
   - **Instruments**: 18

2. **Calibration Information**

   - **Calibration Line**: $y = mx + b$
   - **Regression**: 0.0012, 0.002, 0.003, 0.004
   - **Correlation**: 0.998, 0.998, 0.998, 0.998

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<th>Sample</th>
<th>Y-Intercept (b)</th>
<th>Slope (m)</th>
<th>Correlation</th>
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<tr>
<td>1</td>
<td>0.0012</td>
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<td>0.998</td>
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<tr>
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<td>0.002</td>
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<tr>
<td>3</td>
<td>0.003</td>
<td>0.0003</td>
<td>0.998</td>
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<tr>
<td>4</td>
<td>0.004</td>
<td>0.0004</td>
<td>0.998</td>
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3. **Accuracy Means, Standard Deviations and Covariances**

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4. **Critical levels according to EC/2002/157**

5. **Results from ISO 5725-2: 1994**

6. **Accuracy Means & Standard deviations experiment 4**

7. **Measurement Uncertainty**

---

**Initials**

- [Initials]
- [Initials]
- [Initials]
- [Initials]

---

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ResVal v 2.2 Validation Report (SCP 475)

1 General Information

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<th>Analysis Method</th>
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2 Calibration Information

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<th>Deviation</th>
<th>Correlation</th>
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<td>Ref. 2</td>
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<td>Ref. 3</td>
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<th>Standard (µg / mL)</th>
<th>Deviation</th>
<th>Correlation</th>
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3 Accuracy Means, Standard Deviations and Covariances

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<th>Accuracy (%)</th>
<th>Exp. 1</th>
<th>Exp. 2</th>
<th>Exp. 3</th>
<th>Exp. 1</th>
<th>Exp. 2</th>
<th>Exp. 3</th>
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<tbody>
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<td>Covariance</td>
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4 Critical levels according to EC 2002/657

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<thead>
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<th>Critical level</th>
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<th>0.006</th>
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5 Results from ISO 5715-2: 1994

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<td>CV (%)</td>
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6 Accuracy Means & Standard Deviations experiment 4

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<thead>
<tr>
<th>Accuracy (%)</th>
<th>S.D.</th>
<th>CV (%)</th>
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</thead>
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<td>S.D.</td>
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<td>CV (%)</td>
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7 Measurement uncertainty

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<tr>
<th>Sources</th>
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Initial: Technical: Initial: Study Director:
ResVal v. 2.1 Validation Report (SCP 475)

1 General Information

- Instrument: [Name of instrument]
- Method: [Description of method]
- Project: [Project details]
- Lab: [Laboratory details]
- Author: [Author's name]
- Date: [Date of report]
- Revision: [Version of report]

2 Calibration Information

<table>
<thead>
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<th>Standard (mg/l)</th>
<th>Standard (mg/l)</th>
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<td>1.000</td>
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3 Accuracy Means, Standard Deviations and Covariances

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<th>Exp 2</th>
<th>Exp 3</th>
<th>Exp 1</th>
<th>Exp 2</th>
<th>Exp 3</th>
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4 Critical levels according to EC260/2/157

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<th>Repeatability</th>
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5 Results from ISO 5725: 1994

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<th>RSD (%)</th>
<th>RSD (%)</th>
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<td>2</td>
<td>4.0</td>
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6 Accuracy Means & Standard deviations experiment 4

<table>
<thead>
<tr>
<th>Scenario</th>
<th>Accuracy</th>
<th>RSD (%)</th>
<th>RSD (%)</th>
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<td>1</td>
<td>2.0</td>
<td>2.0</td>
<td>2.0</td>
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</table>

7 Measurement uncertainty

- Precision:
  - Random uncertainty:
  - Systematic uncertainty:
  - Total uncertainty:
- Accuracy:
  - Random uncertainty:
  - Systematic uncertainty:
  - Total uncertainty:

Initial draft: [Initials]
Final draft: [Initials]
ResVal v. 2.1 Validation Report (SCP 475)

1 General Information

2 Calibration Information

<table>
<thead>
<tr>
<th>Sample</th>
<th>Result (a)</th>
<th>Yielded (b)</th>
<th>Correlation (r)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Exp 1</td>
<td>0.012</td>
<td>0.094</td>
<td>0.961</td>
</tr>
<tr>
<td>Exp 2</td>
<td>0.013</td>
<td>0.075</td>
<td>0.886</td>
</tr>
<tr>
<td>Exp 3</td>
<td>0.027</td>
<td>0.103</td>
<td>0.972</td>
</tr>
<tr>
<td>Exp 4</td>
<td>0.021</td>
<td>0.093</td>
<td>0.854</td>
</tr>
</tbody>
</table>

3 Accuracy Means, Standard Deviations and Covariances

<table>
<thead>
<tr>
<th>Experiment</th>
<th>Exp 1</th>
<th>Exp 2</th>
<th>Exp 3</th>
<th>Exp 1</th>
<th>Exp 2</th>
<th>Exp 3</th>
</tr>
</thead>
<tbody>
<tr>
<td>Accuracy</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Exp 1</td>
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<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Exp 2</td>
<td></td>
<td></td>
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<td></td>
<td></td>
</tr>
<tr>
<td>Exp 3</td>
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</tr>
<tr>
<td>Exp 4</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

4 Critical levels according to EC2602/157

5 Results from ISO 5755-2: 1994

6 Accuracy Means & Standard deviations experiment 4

7 Measurement uncertainty

Footnote: The measurement uncertainty is expressed as a relative value with an absolute value of 0.5% or higher according to section 3.2.

Printdatum: 2-Oct-08
ResVal v. 2.1 Validation Report (SCP 475)

1 General Information

Instrument: Type 4484

2 Calibration Information

3 Accuracy Means, Standard Deviations and Covariances

4 Critical levels according to EC2602/157

5 Results from ISO 5715-2: 1994

6 Accuracy Means & Standard deviations experiment 4

7 Measurement uncertainty

Results are given as if matrix used in table 7 for the determination of the measurement uncertainty

To the results of this experiment are used in table 7 for the determination of the matrix effect

The measurement uncertainty is expressed as a relative value and is given as an absolute value as in section 2.2.

The relative uncertainty is the relative average value of 0.1, 1 and 10.

By using the results of table 7 measured concentrations should be reported as ±2.5% (U5).
### 1 General Information

**Instrument:**
- **Model:**
- **Serial number:**

**Calibration Information**

<table>
<thead>
<tr>
<th>Exp 1</th>
<th>Exp 2</th>
<th>Exp 3</th>
</tr>
</thead>
<tbody>
<tr>
<td>100.0</td>
<td>101.0</td>
<td>99.0</td>
</tr>
</tbody>
</table>

**Accuracy Means, Standard Deviations and Covariances**

<table>
<thead>
<tr>
<th>Exp 1</th>
<th>Exp 2</th>
<th>Exp 3</th>
</tr>
</thead>
<tbody>
<tr>
<td>100.0</td>
<td>101.0</td>
<td>99.0</td>
</tr>
</tbody>
</table>

**Critical levels according to EC2002/157**

<table>
<thead>
<tr>
<th>CCl4</th>
<th>CCl3</th>
<th>CCl2</th>
</tr>
</thead>
<tbody>
<tr>
<td>100</td>
<td>100</td>
<td>100</td>
</tr>
</tbody>
</table>

**Results from ISO 5725-2: 1994**

<table>
<thead>
<tr>
<th>Exp 1</th>
<th>Exp 2</th>
<th>Exp 3</th>
</tr>
</thead>
<tbody>
<tr>
<td>100.0</td>
<td>101.0</td>
<td>99.0</td>
</tr>
</tbody>
</table>

**Accuracy Means & Standard Deviations experiment 4**

<table>
<thead>
<tr>
<th>Exp 1</th>
<th>Exp 2</th>
<th>Exp 3</th>
</tr>
</thead>
<tbody>
<tr>
<td>100.0</td>
<td>101.0</td>
<td>99.0</td>
</tr>
</tbody>
</table>

**Measurement Uncertainty**

<table>
<thead>
<tr>
<th>Category</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Relative uncertainty</td>
<td>0.005</td>
</tr>
</tbody>
</table>

**ResVal v. 2.2 Validation Report (SCP 475)**

**Printdatum:** 2-Oct-08

**Pagina:** 13 van 24
ResVal [v. 1.2] Validation Report (SCP 475)

1 General Information

2 Calibration Information

3 Accuracy Means, Standard Deviations and Covariances

4 Critical levels according to EC/2002/557

5 Results from ISO 5715-2: 1994

6 Accuracy Means & Standard deviations experiment 4

7 Measurement uncertainty

[Table and text content]

Printdatum: 2-Oct-08
ResVal v. 2.0 Validation Report (SCP 475)

1 General Information

2 Calibration Information

3 Accuracy Means, Standard Deviations and Covariances

4 Critical levels according to EC2602/957

5 Results from ISO 5715-2: 1994

6 Accuracy Means & Standard Deviations experiment 4

7 Measurement uncertainty

---

Pagina 15 van 24
### ResVal v. 2.2 Validation Report (SCP 475)

#### 1 General Information

<table>
<thead>
<tr>
<th>Instrument</th>
<th>Model</th>
<th>Series</th>
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<tbody>
<tr>
<td></td>
<td></td>
<td></td>
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</tbody>
</table>

#### 2 Calibration Information

<table>
<thead>
<tr>
<th>Step (a)</th>
<th>Yielded (b)</th>
<th>Correlation (c)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Exp 1</td>
<td>0.12</td>
<td>0.991</td>
</tr>
<tr>
<td>Exp 2</td>
<td>0.139</td>
<td>0.991</td>
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<tr>
<td>Exp 3</td>
<td>0.146</td>
<td>0.991</td>
</tr>
<tr>
<td>Exp 4</td>
<td>0.152</td>
<td>0.991</td>
</tr>
</tbody>
</table>

#### 3 Accuracy Means, Standard Deviations and Covariances

<table>
<thead>
<tr>
<th>Exp 1</th>
<th>Exp 2</th>
<th>Exp 3</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

#### 4 Critical levels according to EC/2002/457

<table>
<thead>
<tr>
<th>Level 1</th>
<th>Level 2</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.01</td>
<td>0.002</td>
</tr>
<tr>
<td></td>
<td></td>
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</tbody>
</table>

#### 5 Results from ISO 5725-2: 1994

<table>
<thead>
<tr>
<th>Repeatability</th>
<th>%R.D.</th>
</tr>
</thead>
<tbody>
<tr>
<td>2.6</td>
<td>1.9</td>
</tr>
</tbody>
</table>

#### 6 Accuracy Means & Standard deviations experiment 4

<table>
<thead>
<tr>
<th>Exp 1</th>
<th>Exp 2</th>
<th>Exp 3</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

#### 7 Measurement uncertainty

<table>
<thead>
<tr>
<th>Contribution</th>
<th>%U</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>0.005</td>
</tr>
<tr>
<td></td>
<td>0.005</td>
</tr>
<tr>
<td></td>
<td>0.005</td>
</tr>
</tbody>
</table>

**Initial:** [Signature]
**Study director:** [Signature]

### SOP ARO/504 v 1
Printdatum: 2-Oct-08
### 1 General Information

**Instruments:**

- Name: GCMS
- Project: Analytical method development
- Endpoints: Validation of methods

**Validation:**

- Method: SNI 8522.04
- Reaction: 1000 ppm

**Calibration Information**

<table>
<thead>
<tr>
<th>Sample</th>
<th>Reaction (ppm)</th>
<th>Deviation (ppm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Exp. 1</td>
<td>1000</td>
<td>0.1</td>
</tr>
<tr>
<td>Exp. 2</td>
<td>1000</td>
<td>0.2</td>
</tr>
<tr>
<td>Exp. 3</td>
<td>1000</td>
<td>0.3</td>
</tr>
<tr>
<td>Exp. 4</td>
<td>1000</td>
<td>0.4</td>
</tr>
</tbody>
</table>

**Accuracy:**

<table>
<thead>
<tr>
<th>Exp. 1</th>
<th>Exp. 2</th>
<th>Exp. 3</th>
</tr>
</thead>
<tbody>
<tr>
<td>Accuracy (%)</td>
<td>Accuracy (%)</td>
<td>Accuracy (%)</td>
</tr>
<tr>
<td>99.5</td>
<td>100.5</td>
<td>99.0</td>
</tr>
</tbody>
</table>

### 3 Accuracy Means, Standard Deviations and Covariances

<table>
<thead>
<tr>
<th>Exp. 1</th>
<th>Exp. 2</th>
<th>Exp. 3</th>
</tr>
</thead>
<tbody>
<tr>
<td>Accuracy (%)</td>
<td>Accuracy (%)</td>
<td>Accuracy (%)</td>
</tr>
<tr>
<td>99.5</td>
<td>100.5</td>
<td>99.0</td>
</tr>
</tbody>
</table>

### 4 Critical levels according to EC 2002/157

<table>
<thead>
<tr>
<th>Level</th>
<th>Accuracy (%)</th>
<th>Standard Deviation (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Level 1</td>
<td>99.0</td>
<td>1.0</td>
</tr>
<tr>
<td>Level 2</td>
<td>98.0</td>
<td>1.5</td>
</tr>
</tbody>
</table>

### 5 Results from ISO 5725: 1994

<table>
<thead>
<tr>
<th>Level</th>
<th>Accuracy (%)</th>
<th>Standard Deviation (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Level 1</td>
<td>99.0</td>
<td>1.0</td>
</tr>
<tr>
<td>Level 2</td>
<td>98.0</td>
<td>1.5</td>
</tr>
</tbody>
</table>

### 6 Accuracy Means & Standard deviations experiment 4

<table>
<thead>
<tr>
<th>Level</th>
<th>Accuracy (%)</th>
<th>Standard Deviation (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Level 1</td>
<td>99.0</td>
<td>1.0</td>
</tr>
<tr>
<td>Level 2</td>
<td>98.0</td>
<td>1.5</td>
</tr>
</tbody>
</table>

### 7 Measurement Uncertainty

- Estimated: 1.0%
- Calculation: 1.0%
- Other uncertainties: 0.5%
- Overall uncertainty: 1.5%

**Intake technician:**

<table>
<thead>
<tr>
<th>Intake director</th>
<th>Director</th>
</tr>
</thead>
<tbody>
<tr>
<td>John Doe</td>
<td>Jane Doe</td>
</tr>
</tbody>
</table>

**Printdatum:** 2-Oct-08

**Pagina 18 van 24**
### General Information

<table>
<thead>
<tr>
<th>Instrument</th>
<th>Name of the instrument</th>
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</thead>
<tbody>
<tr>
<td>Value 1</td>
<td>Value 2</td>
</tr>
<tr>
<td>Value 3</td>
<td>Value 4</td>
</tr>
</tbody>
</table>

### Calibration Information

<table>
<thead>
<tr>
<th>Calibration Point</th>
<th>Standard Value (a)</th>
<th>Obtained Value (b)</th>
<th>Correlation (c)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Exp 1</td>
<td>123.4</td>
<td>123.5</td>
<td>0.999</td>
</tr>
<tr>
<td>Exp 2</td>
<td>124.6</td>
<td>124.7</td>
<td>0.998</td>
</tr>
<tr>
<td>Exp 3</td>
<td>125.8</td>
<td>125.9</td>
<td>0.997</td>
</tr>
</tbody>
</table>

### Accuracy Means, Standard Deviations and Covariances

<table>
<thead>
<tr>
<th>Experiment</th>
<th>Exp 1</th>
<th>Exp 2</th>
<th>Exp 3</th>
</tr>
</thead>
<tbody>
<tr>
<td>Accuracy (%)</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Standard Deviation (%)</td>
<td></td>
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</table>

### Critical levels according to EC/2002/157

<table>
<thead>
<tr>
<th>Level</th>
<th>Critical Value (%D)</th>
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</thead>
<tbody>
<tr>
<td>Exp 1</td>
<td>1</td>
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<tr>
<td>Exp 2</td>
<td>2</td>
</tr>
<tr>
<td>Exp 3</td>
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</table>

### Results from ISO 5725-2: 1994

<table>
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<tr>
<th>Experiment</th>
<th>Accuracy (%)</th>
<th>Standard Deviation (%)</th>
<th>Relative Error (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Exp 1</td>
<td>4.5</td>
<td>3.5</td>
<td>0.2</td>
</tr>
<tr>
<td>Exp 2</td>
<td>5.6</td>
<td>4.7</td>
<td>0.3</td>
</tr>
</tbody>
</table>

### Accuracy Means & Standard Deviations experiment 4

<table>
<thead>
<tr>
<th>Experiment</th>
<th>Accuracy (%)</th>
<th>Standard Deviation (%)</th>
<th>Relative Error (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Exp 1</td>
<td>6.7</td>
<td>5.8</td>
<td>0.4</td>
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</tbody>
</table>

### Measurement Uncertainty

<table>
<thead>
<tr>
<th>Source of Uncertainty</th>
<th>Uncertainty</th>
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</thead>
<tbody>
<tr>
<td>Sources of uncertainty</td>
<td>0.004</td>
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<tr>
<td>Measurement uncertainty</td>
<td>0.004</td>
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</tbody>
</table>

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Note: The results are given as % of the reference results.
### 1 General Information

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<thead>
<tr>
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<th>Setting</th>
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<tr>
<td>Value</td>
<td>CASN</td>
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<tr>
<td>Project</td>
<td>Code</td>
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</tbody>
</table>
| Method | Identification of substances in food by analysis of fingerprints in the ResVal [v. 2.2 Validation Report (SCP 475)]
| Instrument | OPERA 4111 (R 10081) |

### 2 Calibration Information

<table>
<thead>
<tr>
<th>Sample</th>
<th>Y Intercept (a)</th>
<th>Y Intercept (b)</th>
<th>Correlation (r)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Exp 1</td>
<td>0.000</td>
<td>0.000</td>
<td>0.999</td>
</tr>
<tr>
<td>Exp 2</td>
<td>0.000</td>
<td>0.000</td>
<td>0.999</td>
</tr>
<tr>
<td>Exp 3</td>
<td>0.000</td>
<td>0.000</td>
<td>0.999</td>
</tr>
<tr>
<td>Exp 4</td>
<td>0.000</td>
<td>0.000</td>
<td>0.999</td>
</tr>
</tbody>
</table>

### 3 Accuracy Means, Standard Deviations and Covariances

#### a) Univariate

<table>
<thead>
<tr>
<th>Sample</th>
<th>Exp 1</th>
<th>Exp 2</th>
<th>Exp 3</th>
<th>Exp 1</th>
<th>Exp 2</th>
<th>Exp 3</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Accuracy (%)</td>
<td>Accuracy (%)</td>
<td>Accuracy (%)</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Exp 1</td>
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<td>90.5</td>
<td>92.3</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Exp 2</td>
<td>90.5</td>
<td>92.3</td>
<td>90.5</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Exp 3</td>
<td>92.3</td>
<td>90.5</td>
<td>92.3</td>
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</tr>
</tbody>
</table>

#### b) Multivariate

<table>
<thead>
<tr>
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<th>Exp 1</th>
<th>Exp 2</th>
<th>Exp 3</th>
<th>Exp 1</th>
<th>Exp 2</th>
<th>Exp 3</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Accuracy (%)</td>
<td>Accuracy (%)</td>
<td>Accuracy (%)</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Exp 1</td>
<td>92.3</td>
<td>90.5</td>
<td>92.3</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Exp 2</td>
<td>90.5</td>
<td>92.3</td>
<td>90.5</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Exp 3</td>
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<td>90.5</td>
<td>92.3</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

### 4 Critical levels according to EC260/2015

- Value: 0.000
- Value: 0.000
- Value: 0.000

### 5 Results from ISO 5725-2: 1994

#### a) Linearity

<table>
<thead>
<tr>
<th>Sample</th>
<th>Linearity (n)</th>
<th>Linearity (s)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Regression</td>
<td>Correlation</td>
</tr>
<tr>
<td>Exp 1</td>
<td>0.990</td>
<td>0.999</td>
</tr>
<tr>
<td>Exp 2</td>
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<td>0.999</td>
</tr>
<tr>
<td>Exp 3</td>
<td>0.999</td>
<td>0.999</td>
</tr>
</tbody>
</table>

### 6 Accuracy Means & Standard deviations experiment 4

<table>
<thead>
<tr>
<th>Sample</th>
<th>Accuracy (%)</th>
<th>Standard (%)</th>
<th>CV (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Regression</td>
<td>Covariance</td>
<td>Total</td>
</tr>
<tr>
<td>Exp 1</td>
<td>92.3</td>
<td>90.5</td>
<td>92.3</td>
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<tr>
<td>Exp 2</td>
<td>90.5</td>
<td>92.3</td>
<td>90.5</td>
</tr>
<tr>
<td>Exp 3</td>
<td>92.3</td>
<td>90.5</td>
<td>92.3</td>
</tr>
</tbody>
</table>

### 7 Measurement uncertainty

The measurement uncertainty is expressed as a relative value of the standard concentration and a standard deviation in the measurement uncertainty, given in the table below. The measurement uncertainty in the table is the average value of 0.05, 1.0, and 1.5.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Accuracy (%)</th>
<th>Standard (%)</th>
<th>CV (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Regression</td>
<td>Covariance</td>
<td>Total</td>
</tr>
<tr>
<td>Exp 1</td>
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<td>90.5</td>
<td>92.3</td>
</tr>
<tr>
<td>Exp 2</td>
<td>90.5</td>
<td>92.3</td>
<td>90.5</td>
</tr>
<tr>
<td>Exp 3</td>
<td>92.3</td>
<td>90.5</td>
<td>92.3</td>
</tr>
</tbody>
</table>

Signatures:

- [Signature 1]
- [Signature 2]
- [Signature 3]
### Validation Report (SCP 475)

#### 1 General Information

<table>
<thead>
<tr>
<th>Instrument</th>
<th>LC</th>
<th>IC</th>
<th>GC</th>
<th>FT-IR</th>
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<tbody>
<tr>
<td>Method</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

#### 2 Calibration Information

<table>
<thead>
<tr>
<th>Sample</th>
<th>Blank</th>
<th>100 ppm</th>
<th>200 ppm</th>
<th>300 ppm</th>
<th>400 ppm</th>
<th>500 ppm</th>
<th>600 ppm</th>
<th>700 ppm</th>
<th>800 ppm</th>
<th>900 ppm</th>
<th>1000 ppm</th>
</tr>
</thead>
<tbody>
<tr>
<td>Value</td>
<td>0.015</td>
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<td>0.016</td>
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#### 3 Accuracy Means, Standard Deviations and Covariances

<table>
<thead>
<tr>
<th>Assay 1</th>
<th>Exp 1</th>
<th>Exp 2</th>
<th>Exp 3</th>
<th>Exp 1</th>
<th>Exp 2</th>
<th>Exp 3</th>
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<tbody>
<tr>
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#### 4 Critical Levels according to EC/2802/1997

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#### 5 Results from ISO 5725-2: 1994

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#### 6 Accuracy Means & Standard Deviations experiment 4

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#### 7 Measurement Uncertainty

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Analytisch laboratorium voor voedings- en residue onderzoek

SOP ARO/504 v 1

Printdatum: 2-Oct-08
### 1 General Information

**Instrument:**
- Name: GC/MS
- Project: Contaminant/Reagent (250.6)

**Methodology:**
- Sample preparation: 
  - Instruments (GC/MS)
  - Procedures (GC/MS)

**Quality Assurance:**
- Calibration
- Accuracy
- Reproducibility

### 2 Calibration Information

<table>
<thead>
<tr>
<th>Sample</th>
<th>Y (x = 1)</th>
<th>Y (x = 1.00)</th>
<th>Correlation (r)</th>
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<td>0.012</td>
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<tr>
<td>Exp. 3</td>
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<td>0.015</td>
<td>0.012</td>
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<tr>
<td>Exp. 4</td>
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<td>0.012</td>
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### 3 Accuracy Means, Standard Deviations and Covariances

<table>
<thead>
<tr>
<th>Sample</th>
<th>Exp. 1 (Accuracy) (%)</th>
<th>Exp. 2 (Accuracy) (%)</th>
<th>Exp. 3 (Accuracy) (%)</th>
<th>Exp. 1 (Stdev. (%)</th>
<th>Exp. 2 (Stdev. (%)</th>
<th>Exp. 3 (Stdev. (%)</th>
<th>Correlation (r)</th>
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<td>Exp. 2</td>
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### 4 Critical levels according to EC/2002/157

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### 5 Results from ISO 5755-2: 1994

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### 6 Accuracy Means & Standard deviations experiment 4

<table>
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<th>CV (%)</th>
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<tr>
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### 7 Measurement uncertainty

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Intake technician: [Signature]  
Study director: [Signature]  
Mentor: [Signature]
Documentbeheer

Algemeen

Invoerdatum: 23 augustus 2006
Wijzigingsdatum: 16 januari 2007
Controledatum: 31 maart 2011
Publicatiedatum: 18 oktober 2006

Wijzigingen ten opzichte van vorige versie:

Beoordelaars

Saskia Sterk (afdelingshoofd)

Hyperlinks