

Single-lab validation approach in practice:

Determination of acrylamide in crisp bread by LC-MS

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Single-lab validation approach in practice: Determination of acrylamide in crisp bread by LC-MS

- Concentration level 998 µg/kg
- Laboratory has analysed two certified reference materials (CRMs) with similar matrixes
 - Potato chips and crisp bread
 - The crisp bread CRM is also used as a control sample

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Certified reference material (CRM)

- The **crisp bread** CRM has the following acrylamide content:

$$C_{\text{acrylamide}} = (1179 \pm 68) \mu\text{g/kg} \quad (k = 2, \text{norm.})$$

- The **potato chips** CRM has the following acrylamide content:

$$C_{\text{acrylamide}} = (860 \pm 42) \mu\text{g/kg} \quad (k = 2, \text{norm.})$$

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Measurements with the CRMs

Crisp bread

Days	C (µg/kg)
5.01.2008	1172
6.03.2008	1186
3.04.2008	1153
8.01.2009	1151
18.03.2009	1181
3.04.2009	1147
11.04.2009	1097
16.04.2009	1102
25.04.2009	1162
3.08.2009	1138
28.08.2009	1122
27.11.2009	1191

Mean: 1150 µg/kg
Std Dev: 31 µg/kg

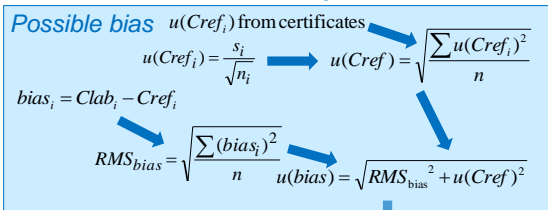
Potato chips

Days	C (µg/kg)
3.04.2008	845
3.04.2008	832
3.04.2008	802
27.04.2008	829
27.04.2008	851
27.04.2008	834

Mean: 832 µg/kg
Std Dev: 17 µg/kg

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Roadmap:



Uncertainty due to random effects

$$u(R_w) = s_{RW}$$

Combined standard uncertainty

$$u_c = \sqrt{u(R_w)^2 + u(bias)^2}$$

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Finding $u(R_w)$

$$u(R_w) = s_{RW} = 31 \mu\text{g/kg}$$

$$u(R_w)_{\text{rel}} = s_{RW_rel} = 31/1150 \cdot 100 = 2.70 \%$$

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Finding $u(\text{bias})$

Ref value $\mu\text{g/kg}$	$U (k=2)$ $\mu\text{g/kg}$	u_c $\mu\text{g/kg}$	Lab result $\mu\text{g/kg}$	u_{c_rel} %	bias_i $\mu\text{g/kg}$	bias_rel %
1179	68	34	1150	2.88	-29	-2.45
860	42	21	832	2.44	-28	-3.24
$u(\text{Cref})_{rel} = 2.67\%$						
$RMS_{\text{bias_rel}} = 2.87\%$						
$u(\text{bias})_{rel} = 3.92\%$						

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Result:

$u_{c_rel} =$	4.8 %
$u_c =$	48 $\mu\text{g/kg}$
$U_{rel} (k=2) =$	9.5 %
$U (k=2) =$	95 $\mu\text{g/kg}$

- Acrylamide content in the sample

$$C_{\text{acrylamide}} = (998 \pm 95) \mu\text{g/kg} \quad (k = 2, \text{norm.})$$

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