

Validating Methods Outside of Accreditation

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Validating Methods Outside of Accreditation

Why validate methods outside of accreditation?

Validating Methods Outside of Accreditation

- 1) The laboratory may not be accredited
- 2) The laboratory may not want to accredit the test

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The laboratory is not be accredited

Does it need to be?

Small food business internal laboratories

Only enforcement/control laboratories have to!

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The laboratory may not want to accredit the test

Rarely used

Excessive costs

Responding to an issue

Beware two tier systems

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Often analysis required for unusual analyte at short notice

Sudan Dyes - Food

MCPD – Processed Foods

MITC (fungicide) – Wine

Phthalates – Food seals

Monomers – Plastics

Results of investigation/complaint

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Cost of Validation and tests

How much development work will be needed?

How many samples are there?

How much validation work will be needed?

How long is the test (Man hours)?

Will we need to invest? (Is it a one off scare?)

Can we subcontract?

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- 1) Methods rarely used
- 2) New method to laboratory (But in use elsewhere)
- 3) Method developed from basics

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Methods rarely used

Probably already have some old performance data

need to show you can recreate (Or better) the performance

Laboratories have documented policies and procedures to follow for validation

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New method to laboratory (But in use elsewhere)

Documented methods may have performance data

Need to show you can achieve this

Laboratories have documented policies and procedures to follow for validation

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Overall requirement – Fit for purpose

Detection limits

Reliability

Interferences

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Method developed from basics

a) Half a method

Part of the analytical procedure may already exist,

i.e. Same analyte in a different matrix.

Similar compounds extracted from similar matrices

Some idea of performance will be available.

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Method developed from basics

b) Full Method

No method exists and/or no performance data is available

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First steps

Can you get the analyte in a standard (Certified) form?

Is it readily available?

Are any reference materials available?

How was the issue originally identified?

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Literature search

Has this ever been done before?
Similar compounds?
Same compound different matrix?
Instrument companies?
Universities?
Internet

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Don't forget the science!

Solubility

Volatility

Properties

Diffusion constants

Equilibrium

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Could take piece meal approach

Show detection and quantification works before worrying about extraction

Give some information on concentrations required.

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Example

MITC in wine

3 labs did work but expensive
Obviously guard their methods

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Looked up solvents that were immiscible with water and that MITC was readily soluble in.

Made up MITC standards in solvent and ran them via GC with Nitrogen detector to ensure we could separate and detect/quantify

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Matrix

Made up standards in artificial sample matrix (Water and alcohol)

Check for recovery and linearity

is recovery at this stage critical?

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Actually only about 20%

BUT – Consistent

Not as sensitive as it could be, but still low level.

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Try running some wines and spiking them at increasing levels

Prove zero at origin

Show still linear

When you find some in sample also spike to show correct additional recovery

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Don't forget method development and validation was around before ISO17025 and similar requirements

These brought in a more disciplined/structured approach

Harmonized Guidelines for Single-Laboratory Validation of Methods of Analysis, (IUPAC Technical Report)

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What validation required?

Are we seeking accreditation?

Could we defend our results?

Was the test fit for purpose?

Full test or screen?

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Low level spikes – Calculate limit of detection

Recovery checks and additional recoveries

(On every positive)

Matrix checks – recoveries on changed matrix

(Foods are variable)

Multiple analysis of positives – precision

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When should validation be done?

Ideally prior to analysis

Alongside analysis (Good for generating data)

After analysis (To give extra confidence)

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Finally – After all this effort don't forget

Keep detailed method notes

Keep all records

Someone else should be able to follow your work

Ultimately

Write up method including QC requirements

Ensure it fits within your quality system

Add to scope if desired