Section A4.1 Analytical Methods for Detection and Identification

Annex Point IIA4.1/4.2 & IIIA-IV.1

Official use only

1 REFERENCE

1.1	**Reference**	De Jong, V. (2008) Measurement uncertainty for organic acids and oligomers. Purac Document no. VdJ2008097 Not GLP, Unpublished
1.2	**Data protection**	Yes
1.2.1	Data owner	Purac Biochem
1.2.2	Companies with letter of access	No
1.2.3	Criteria for data protection	Data submitted to the MS after 13 May 2000 on existing [a.s. / b.p.] for the purpose of its [entry into Annex I/IA / authorisation]

2 GUIDELINES AND QUALITY ASSURANCE

2.1	**Guideline study**	Internal method
2.2	**GLP**	No
2.3	**Deviations**	Not applicable

3 MATERIALS AND METHODS

3.1	**Preliminary treatment**	
3.1.1	Enrichment	This is a validation study [redacted]
3.1.2	Cleanup	This is a validation study [redacted]
3.2	**Detection**	
3.2.1	Separation method	[redacted] This is a validation study [redacted]
3.2.2	Detector	[redacted] This is a validation study [redacted]

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[redacted]

3.2.3 Standard(s)

This is a validation study [redacted]

3.2.4 Interfering substance(s)

Not applicable

3.3 Linearity

3.3.1 Calibration range

[redacted]:
Relevant range; note that this is a method for quantifying impurities in a technical product; as such, samples can be prepared to always fall within the required calibration range.

[redacted]
Relevant range; note that this is a method for quantifying impurities in a technical product; as such, samples can be prepared to always fall within the required calibration range.

3.3.2 Number of measurements

[redacted]
formic acid: 11
acetic acid: 11
[redacted]

[redacted]
lactic acid: 6
lactoyl-lactide: 6
lactide: 6
meso-lactide: 6

3.3.3 Linearity

[redacted]
formic acid: linear over entire calibration range ($r^2 = 0.9999$)
acetic acid: linear over entire calibration range ($r^2 = 0.9994$)
[redacted]

[redacted]
lactic acid: linear over entire calibration range ($r^2 \geq 0.9999$)
lactoyl-lactide: linear over entire calibration range ($r^2 \geq 0.9999$)
lactide: linear over entire calibration range ($r^2 \geq 0.9999$)
meso-lactide: linear over entire calibration range ($r^2 \geq 0.9999$)

3.4 Specifity: interfering substances

Not applicable

3.5 Recovery rates at different levels

[redacted]
lactic acid: 100.7% at method concentration
acetic acid: 101.0% at method concentration
[redacted]

[redacted]:

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[redacted]

3.5.1	Relative standard deviation	*See repeatability*
3.6	**Limit of determination**	
3.7	**Precision**	
3.7.1	Repeatability	[redacted] lactic acid: 3.1% acetic acid: 4.7% [redacted] [redacted] lactic acid: 5% lactoyl-lactide: 3.8% lactide: 2.8% meso-lactide: 2.0%
3.7.2	Independent laboratory validation	Not applicable

4 APPLICANT'S SUMMARY AND CONCLUSION

4.1	**Materials and methods**	[redacted]
4.2	**Conclusion**	The methods are suitable as an in-house quality control method. Method A4.1-07 is suitable as method to determine impurities in lactic acid samples.
4.2.1	Reliability	1
4.2.2	Deficiencies	No

Evaluation by Competent Authorities

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	Use separate "evaluation boxes" to provide transparency as to the comments and views submitted
	EVALUATION BY RAPPORTEUR MEMBER STATE
Date	*30/10/2014*
Materials and methods	
Conclusion	*The information given above is only in addition to the analytical methods for detection and identification presented in the DOC III A 4.1.07 and Doc III A4.1.08 documents.* *It would be more useful and comprehensible to merge all information on one analytical method for detection and identification and their validation in one DOC III A 4.1 document*
Reliability	
Acceptability	
Remarks	*Additional information.*
	COMMENTS FROM ...
Date	*Give date of comments submitted*
Results and discussion	*Discuss additional relevant discrepancies referring to the (sub)heading numbers and to applicant´s summary and conclusion.* *Discuss if deviating from view of rapporteur member state*
Conclusion	*Discuss if deviating from view of rapporteur member state*
Reliability	*Discuss if deviating from view of rapporteur member state*
Acceptability	*Discuss if deviating from view of rapporteur member state*
Remarks	