

# QUANTITATIVE ANALYSIS OF TOBACCO-SPECIFIC N-NITROSAMINES IN TOBACCO, TOBACCO PRODUCTS, FIBRE-BASED MATRICES AND TOBACCO DERIVED PRODUCTS WITH (b) (4)

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## Purpose

To determine the concentration of the tobacco-specific N-nitrosamines (TSNA): N-nitrosoanabasine (NAB); N-nitrosoanatabine (NAT); 4-(methyl nitrosamines)-1-(3-pyridyl)-1-butanone (NNK) and N-nitrososnormicotine (NNN) in tobacco, tobacco products, fibre-based matrices and tobacco derived products (also called Purified Products) via (b) (4)

## Applies to

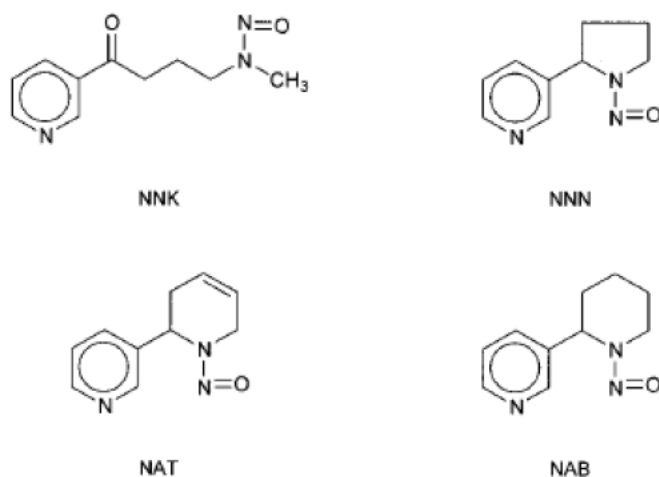
APS

## General information

### Principle of the method

TSNA (see Figure 1) is extracted from the sample matrix into (b) (4). The extract is then filtered to vials, whereupon the sample preparation is complete. (b) (4). Separation and detection are performed with (b) (4).

Calculations are performed (b) (4). The capacity per instrument and person is (b) (4) single samples per week (b) (4) otherwise it is (b) (4) single samples per week.



**Figure 1.** Chemical structural formula for NNK, NNN, NAT and NAB.

This method (APS) is based on the CORESTA Recommended Method No 72 (CRM 72), but uses (b) (4) as in CRM 72.

Additionally, (b) (4)

The differences between CRM 72 and the APS method are presented in [Table 1a](#).

**Table 1a.** The differences between CRM 72 and the APS method.

CRM 72	APS method
(b)	(4)

Note: All reference documents and additional information stated “available upon request” are in Swedish. They are available upon request but need to be translated into English first.

#### Method scope, measurement range and measurement uncertainty

(b)	(4)
-----	-----

(b) (4)

#### Literature references

(b) (4)

#### Internal reference documents (available upon request)

(b) (4)

#### Risk assessment and safety instructions

#### Summarised risk assessment of the method

(b) (4)

(b) (4)

#### **Substances hazardous to the environment**

No substances are classified as environmentally hazardous.

#### **Flammable substances**

(b) (4)

#### **Hazard and precautionary statements**

(b) (4)

#### **Equipment**

##### **Apparatus**

Two different (b) (4) systems are used for the analysis. See the relevant attachment for the settings of individual systems.

Appendix 1. (b) (4)

Appendix 2. (b) (4)

##### **Other equipment and laboratory utensils**

(b) (4)

(b) (4)

(b) (4)

**Check samples**

(b) (4)

**Preparation of stock and standard solutions**

(b) (4)



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(b) (4)





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(b) (4)



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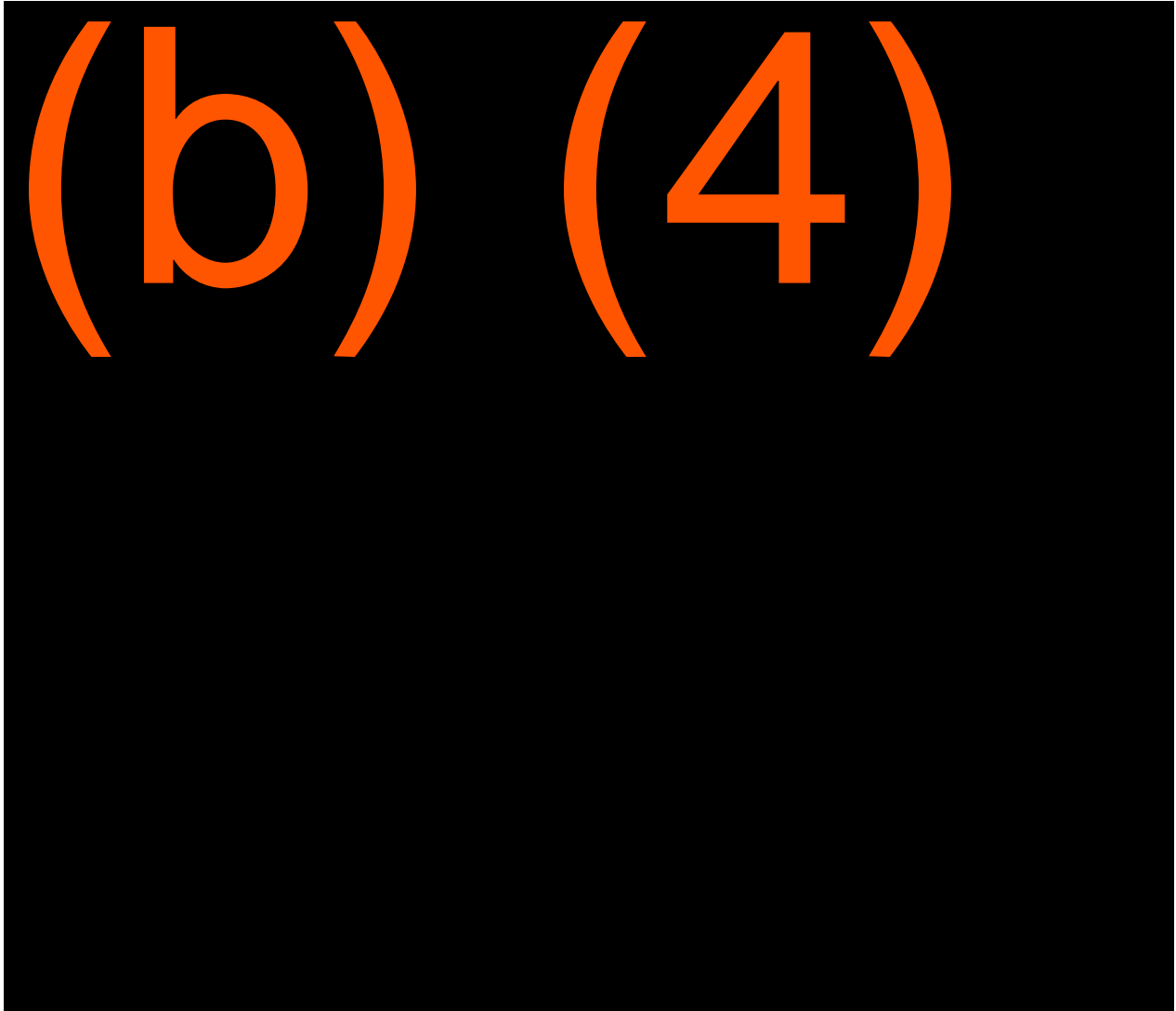
(b) (6)

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(b) (4)

### Preparation of other solutions



### Sample handling

#### Sample storage and preparation

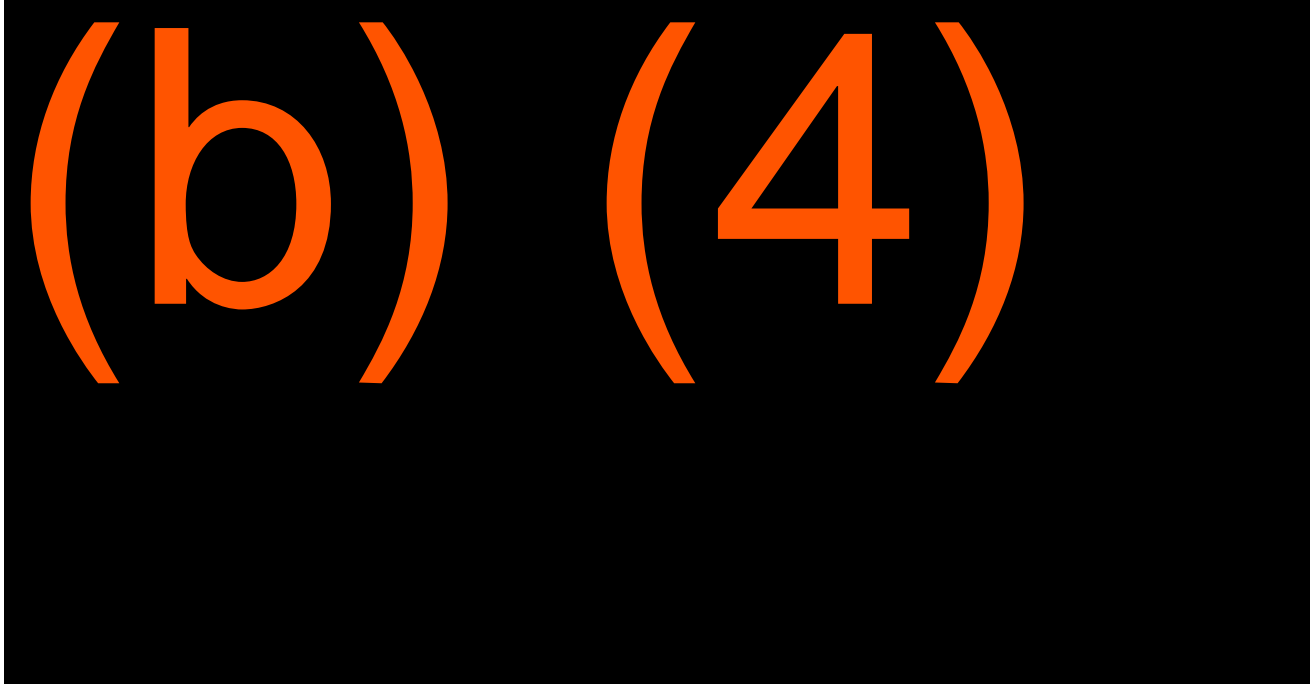
Samples are stored and prepared in accordance with (b) (4) ”.

#### Sample amount

The minimum required sample size for duplicate analysis and re-analysis is 10 g. The minimum amount of sample for performing a simple analysis is 10 g.

## Analysis

### Calibration and verification of apparatus

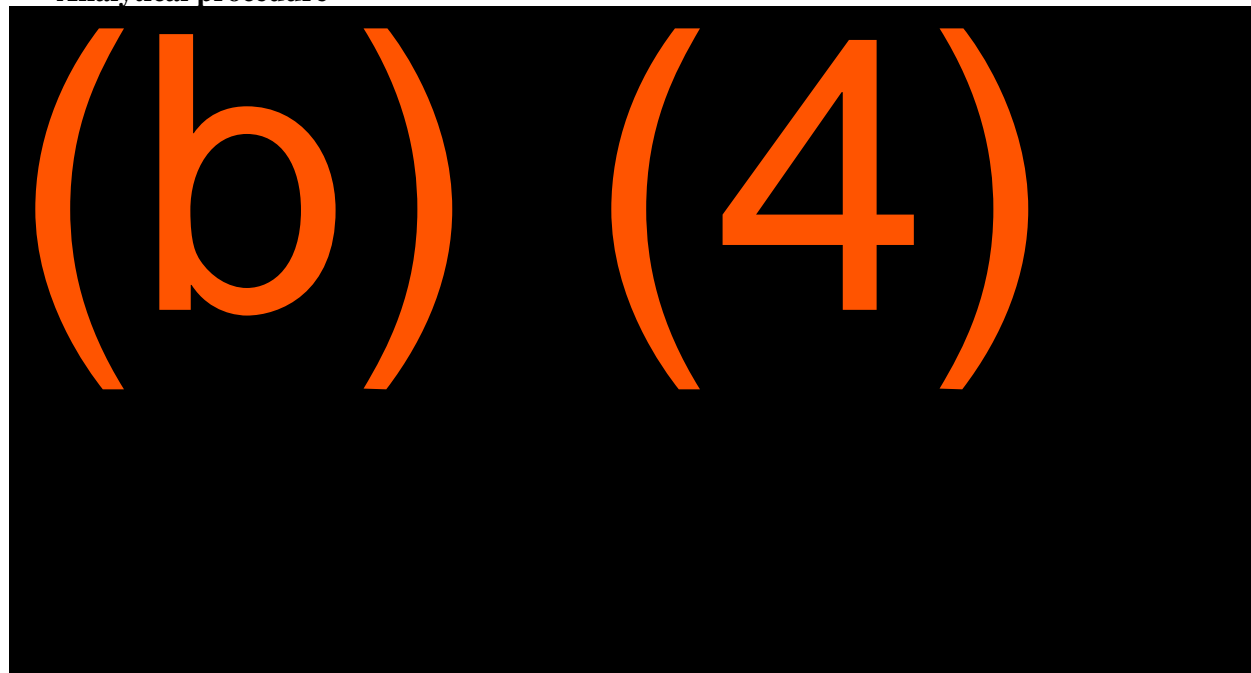


### Sample stability

Prepared sample extracts in a vial are stored in a refrigerator (b) (4)

. Extracts have a shelf life of (b) (4) in the refrigerator.

### Analytical procedure



(b) (4)

#### Special instructions

(b) (4)

#### Documentation

(b) (4)

#### Data


##### Collection and storage of data

(b) (4)

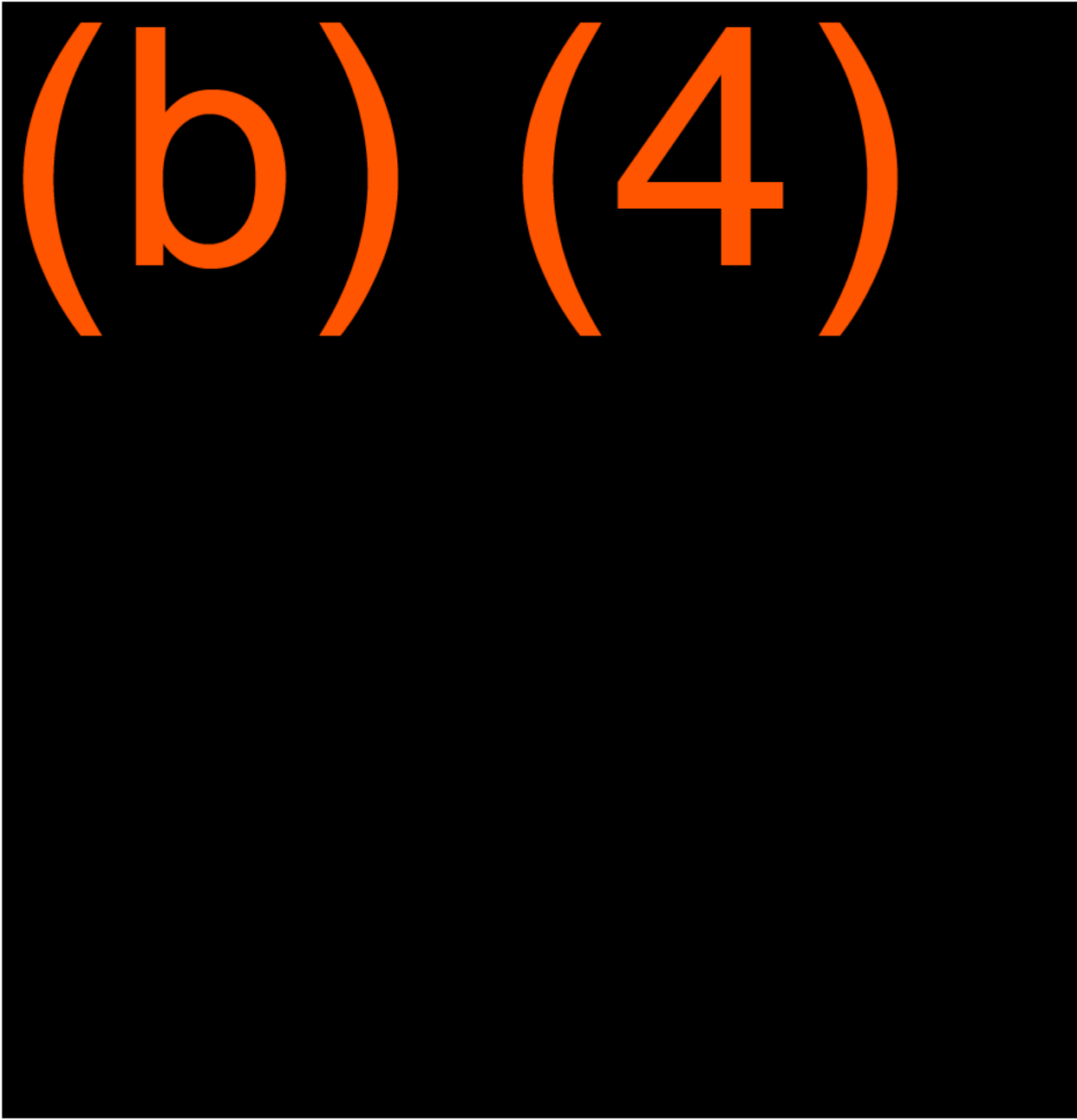
##### Calculations

(b) (4)

(b) (4)




**Quality assurance**




(b) (4)

(b) (4)




**A response greater than the highest calibration standard**

(b) (4)



**Reporting of analysis results**


(b) (4)



**Revision history**


2018

(b) (4)




2017

(b) (4)



2015/2016

(b) (4)



2015

(b) (4)

2014

(b) (4)

2013

(b) (4)

**Person responsible**

Director APS



## Validation

### Validation report

(b) (4)

### Specificity

(b) (4)



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(b) (4)



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(b) (4)

(b) (4)

#### Cross talk

(b) (4)

#### Carry-over

(b) (4)

#### Repeatability

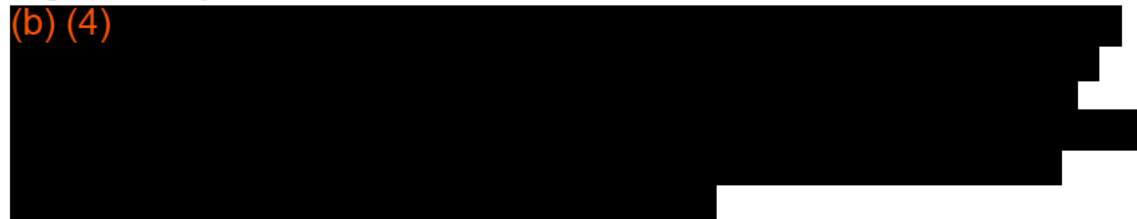
(b) (4)

#### Precision within the laboratory

(b) (4)


### Reproducibility

(b) (4)



### Evaluation of sample comparisons with other laboratories

(b) (4)



### Accuracy

(b) (4)



### Bias from accuracy data

(b) (4)



(b) (4)

**Assessment of matrix effects and extraction yield (ME PE RE)**

(b) (4)


(b) (4)

**Limit of detection (LOD)**

(b) (4)

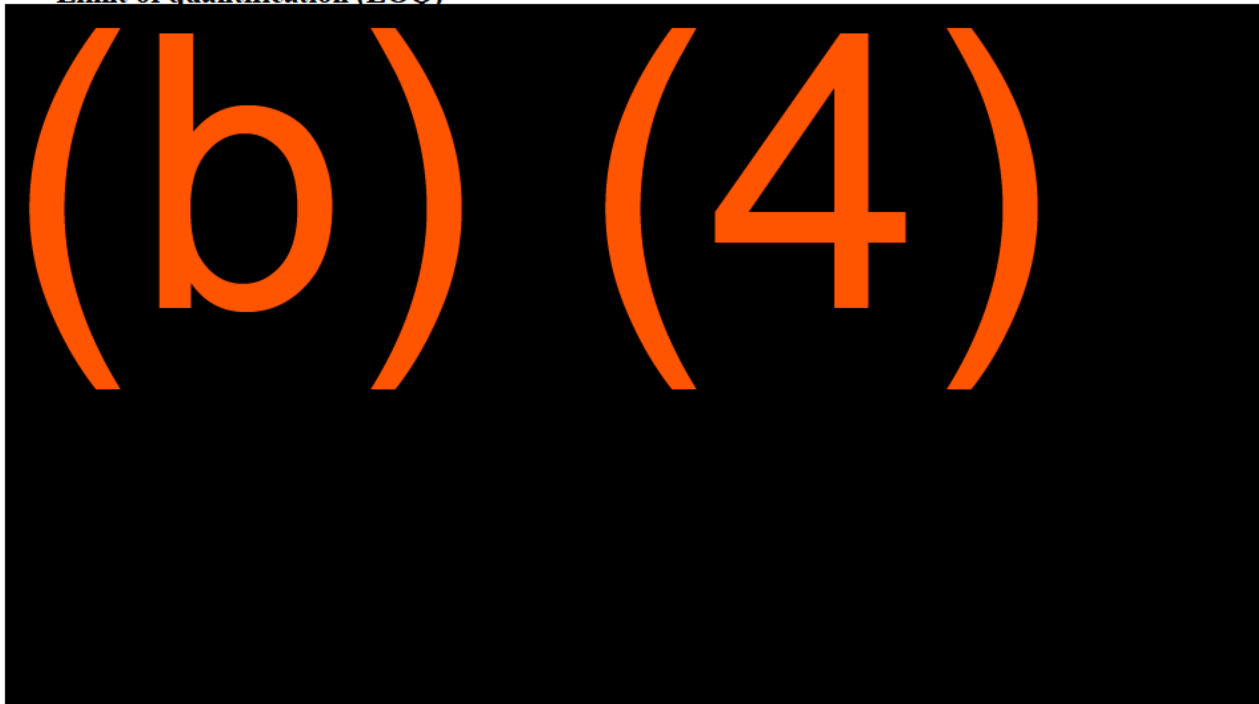


(b) (4)

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#### Limit of quantification (LOQ)

(b) (4)

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#### Linearity

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#### Robustness

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(b) (4)

**Measurement range and measurement uncertainty**

(b) (4)



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(b) (4)

(b) (4)

### Conclusion

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### Appendices

(b) (4)

Appendix 1. (b) (4)

(b) (4)



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(b) (4)



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(b) (4)



Appendix 2. (b) (4)

(b) (4)



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