Internal Quality Control for qualitative tests Identification of the active substance in tear gas weapons

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Overview

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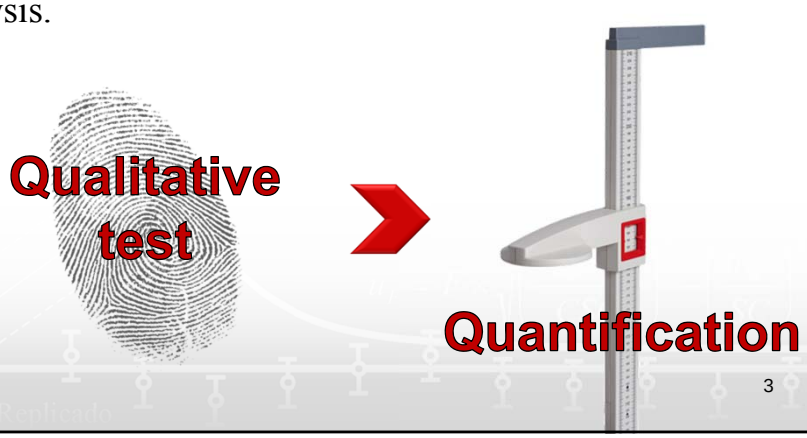
1. Introduction

Quantitative measurements involve a previous qualitative assessment (…)

This stage is more obvious in separation methods of analysis.



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1. Introduction

Accreditation of chemical tests

ISO/IEC 17025:2005 - General requirements for the competence of testing and calibration laboratories

The results of qualitative and quantitative tests must be proven valid.

5.4 Test and calibration methods and method validation
5.4.1 General
The laboratory shall use appropriate methods and procedures for all tests and/or calibrations within its scope. (...)
where appropriate, an estimation of the measurement uncertainty (...)

5.9 Assuring the quality of test and calibration results
5.9.1 The laboratory shall have quality control procedures for monitoring the validity of tests and calibrations undertaken. (...)

Some guidelines and references for the assessment of qualitative tests are available (…)

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1. Introduction

Accreditation of chemical tests

(…)

ILAC G17:2002: Introducing the concept of uncertainty of measurement in testing in association with the application of the standard ISO/IEC 17025

3. Only uncertainty of measurement in quantitative testing is considered for the time being. A strategy on handing results from qualitative testing has to be developed by the scientific community.

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2. Aim

Presentation of a strategy for the validation and quality control of the identification of the active substance in tear gas sprays.

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3. Portuguese legislation

Portuguese legislation – Lei no. 12/2011. Diário da República, 1ª Série – No. 81 – 27 de Abril de 2011, 2399-2439.

- Capsaicin is the only active substance allowed in Portugal (*);
- Capsaicin concentration ≤ 5 g/100 mL.

* - Qualitative test.

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4. Qualitative test procedure

Identification is performed by GC-MS:

Sample collection

GC-MS analysis:
- Retention time (RT);
- Ratio of abundance of characteristic fragments (RA).

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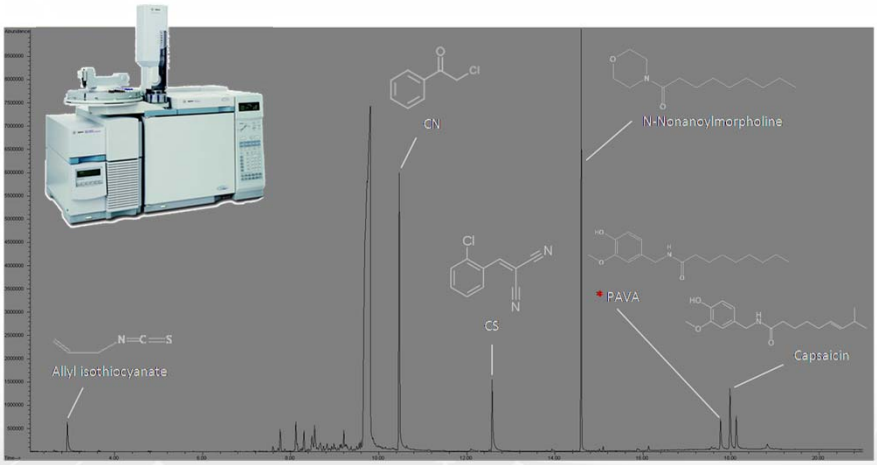
4. Qualitative test procedure

Identification is performed by GC-MS:



* PAVA - Pelargonic acid vanillylamide

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4. Qualitative test procedure

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GC-MS analysis:
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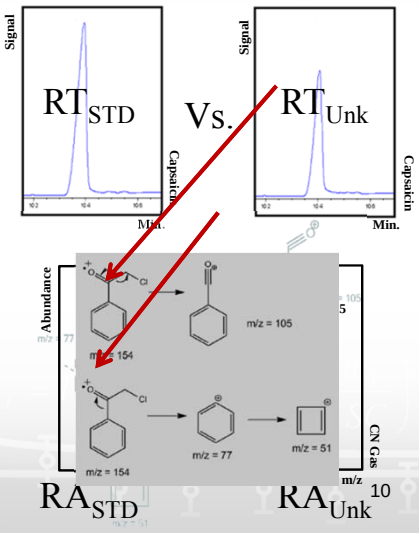
Standard (STD) vs. Unknown (Unk)



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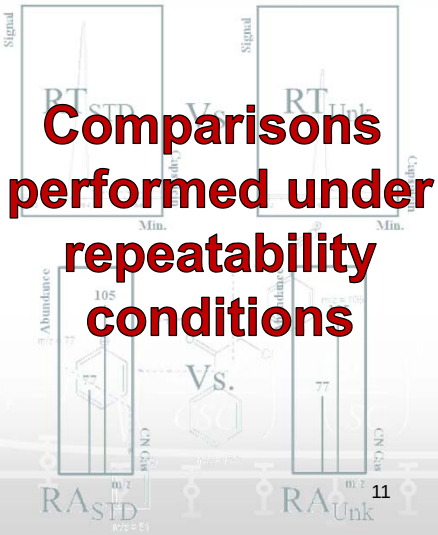

Comparisons performed under repeatability conditions

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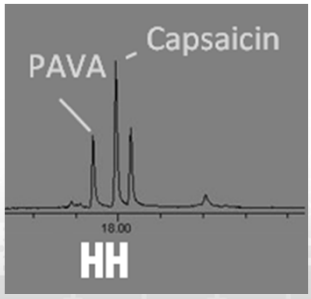
5.1. Qualitative test validation: Chromatography

Retention time (RT):

- Collection of pairs of RT obtained under repeatability conditions;
- Assessment of the normality of the differences (Kolmogorov–Smirnov test);
- Calculation of the repeatability of the difference (sd_{RT});
- Assessment of the fitness of sd_{RT} for the intended use:
  Non-overlapping RT tolerances for 99.7 % confidence level:
  RT±t(99.7%,n-1). sd_{RT}



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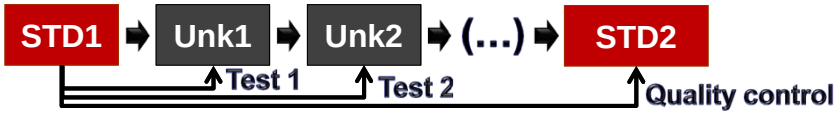
5.2. Qualitative test QC: Chromatography

Retention time (RT):

- Analysis sequence:



- Test criterion:

$$\left|RT_{STD1} - RT_{Unk1}\right| \leq t \cdot sd_{RT}$$

- QC criterion:

$$\left|RT_{STD1} - RT_{STD2}\right| \leq t \cdot sd_{RT}$$

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5.2. Qualitative test QC: Chromatography

Retention time (RT):

(...) - Test criterion:

$$\left|RT_{STD1} - RT_{Unk1}\right| \leq t \cdot sd_{RT}$$

- QC criterion:

$$\left|RT_{STD1} - RT_{STD2}\right| \leq t \cdot sd_{RT}$$

Analyte	Normal dist.	RT selectivity	$(t \cdot sd_{RT})/RT$
Allyl isothiocyanate	✔	✔	1.8 %
CN	✔	✔	0.39 %
CS	✔	✔	0.40 %
N-Nonanoylmorpholine	✔	✔	0.13 %
PAVA	✔	✔	0.60 %
Capsaicin	✔	✔	0.30 %

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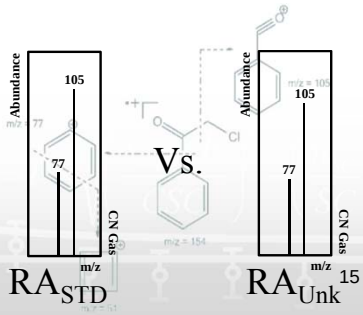
6.1. Qualitative test validation: MS

Ratio of abundances of MS fragments (RA):

- Collection of pairs of RA obtained under repeatability conditions;
- Assessment of the normality of the differences (Kolmogorov–Smirnov test);
- Calculation of the repeatability of the difference (sd_{RA});
- Assessment of the fitness of sd_{RA} for the intended use:

$$\frac{t(99.7\%,n-1).\ sd_{RA}}{RA} \leq 40\%$$

(for, at least, one ratio)



RA_{STD} RA_{Unk}

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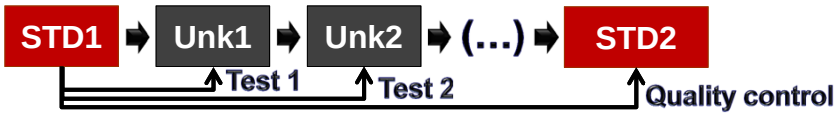
6.2. Qualitative test QC: MS

Ratio of abundances of MS fragments (RA):

- Analysis sequence:



- Test criterion:

$$\left|RA_{STD1} - RA_{Unk1}\right| \leq t \cdot sd_{RA}$$

- QC criterion:

$$\left|RA_{STD1} - RA_{STD2}\right| \leq t \cdot sd_{RA}$$

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6.2. Qualitative test QC: MS

Ratio of abundances of MS fragments (RA):

Analyte (m/z ratios; ❶ and ❷)	Normal dist.	Fit for use	$t \cdot sd_{RA}/RA$ ❶	$t \cdot sd_{RA}/RA$ ❷
Allyl isothiocyanate (❶-99/39;❷-99/72)	✔	✔	17 %	15 %
CN (❶-77/154;❷-105/154)	✔	✔	39 %	34 %
CS (❶-188/137;❷-153/188)	✔	✔	25 %	19 %
N-Nonanoylmorpholine (❶-86/227;❷-129/227)	✔	✔	69 %	35 %
PAVA (❶-293/43;❷-137/293)	✔	✔	80 %	24 %
Capsaicin (❶-305/122;❷-137/305)	✔	✔	40 %	64 %

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7. Conclusions

- The criteria of the identification of active substances can be supported by data collected under repeatability conditions;
- For three compounds (n-nonanoylmorpholine, PAVA and capsaicin), only one RA presents a relative tolerance not greater than 40%;
- Pragmatic and reliable strategy for the identification of active substances in tear gas weapons was presented.

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