The importance of the proficiency of laboratories carrying out halal analyses, and the role of reference material in verifying methods and ensuring the traceability of analyses

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Globalising the Trust in Halal Certification
6-7 October 2021 Ankara

I can detect non halal products!

Are you sure ?

Traceable?

Reliable?

Which method?

LOD?

Which standart?

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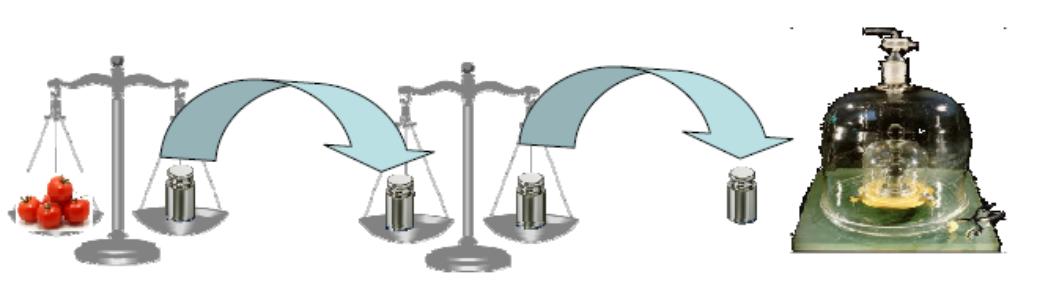
Do we get the same amount?

Is one Kg of tomato sold in enywhere equal to the 1 Kg of tomato sold in İstanbul?
What about the amounts bought from the bazaar or the supermarket?



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Metrological Traceability

"Once measured, everywhere accepted" requires Comparability through Traceability

Metrological traceability

Property of a **measurement result** whereby the result can be related to a reference **through an unbroken chain** of calibrations, each contributing to the **measurement uncertainty**

JCGM 200:2008 (VIM 3)

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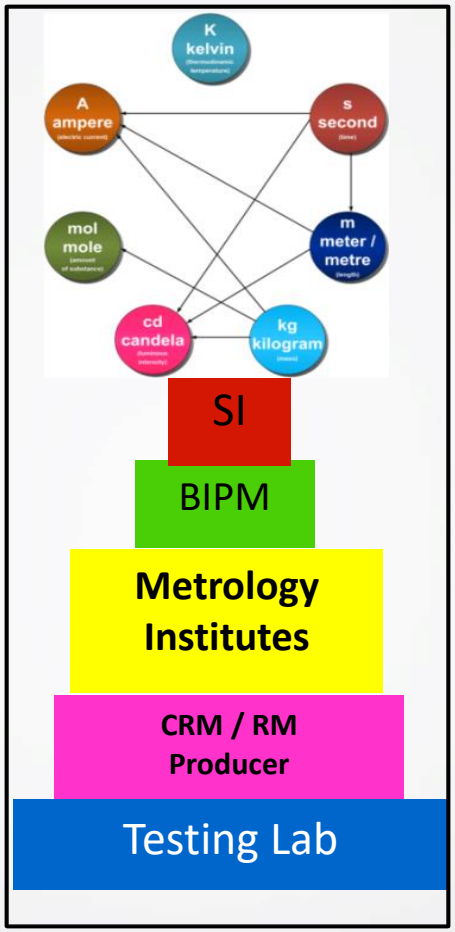
Metrological Traceability



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Quality Assurance in a Laboratory

Lab Staff & Instrument

+

(Standard) Methods

ISO

SMIIC
THE STANDARDS AND METROLOGY INSTITUTE FOR ISLAMIC COUNTRIES
OIC/SMIIC

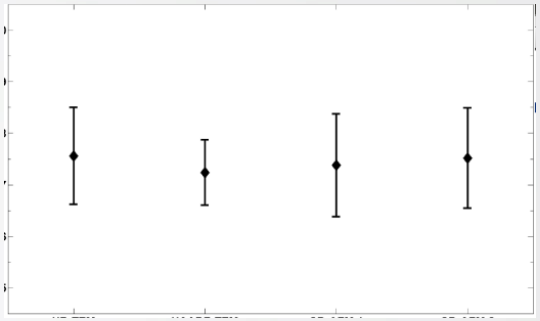
AOAC INTERNATIONAL

ASTM INTERNATIONAL

cen

=

Measurement Result



Competance

Training & Calibration

Validation/Verification & Fit for Purpose

Reliable & Traceable?

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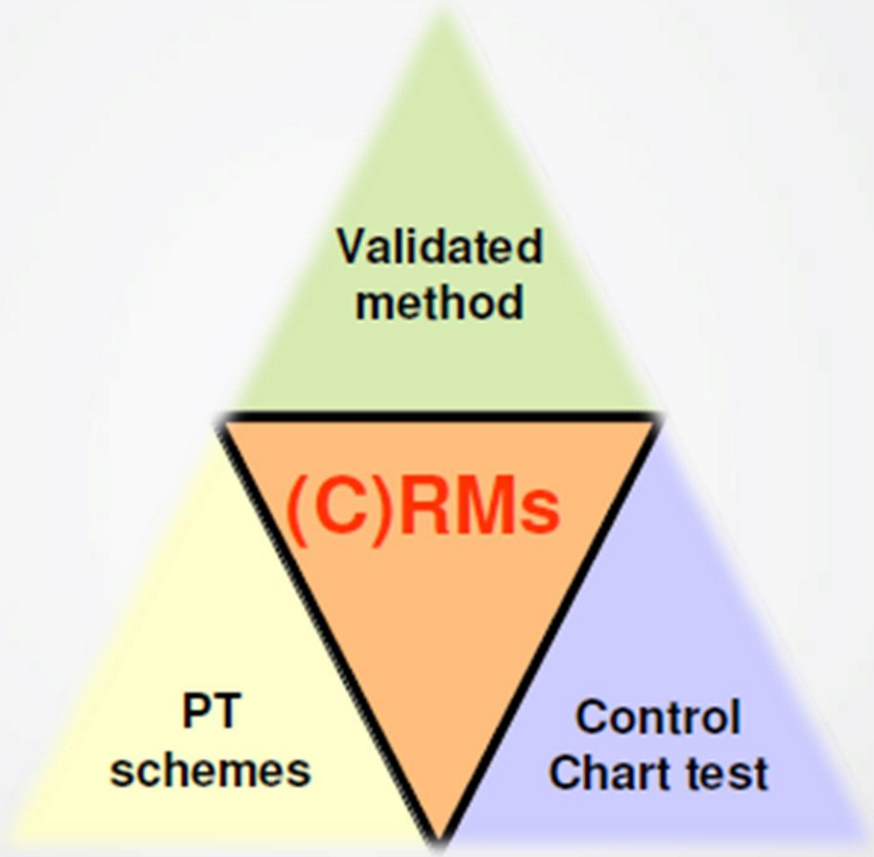
How to Demonstrate Competance



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Proving Lab Performance is Important

- Customers can trust the analytical results from the laboratory
- Comparability of measurement results
- Accreditation to an international standard (e.g. OIC/SMIIC 36:2020, ISO/IEC 17025)

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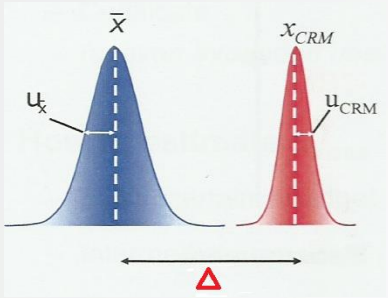
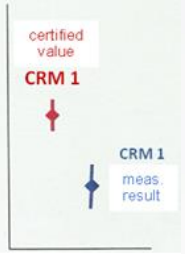
Improvement of Method Performance





Method performance is unacceptable

Reason found, Measurement repeated

Method Performance is acceptable

Detailed investigation needed to be done to find and correct the reason for unacceptable result

Despite all investigations the reason can not be found and the method performance is still unacceptable:

Further Check

1) **If the bias can be shown that it is systematic over time (eg. always 10% lower) (can much easily be seen by a control chart)**
2) **If the bias is constant or has been proven to have a mathematical relation with the concentration of the analyte in the sample**

Then,

- **The laboratory can either use a factor to correct the reported result for the bias or**
- **Without correcting the result, lab can add the influence of bias to the uncertainty**

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CRM & RM

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Definitions

Reference Material

Material, sufficiently homogeneous and stable with respect to one or more specified **properties**, which has been established to be fit for its intended use in a measurement process.

Properties can be quantitative or qualitative, e.g. identity of substances or species.

Certified Reference Material

Reference material characterized by a **metrologically valid procedure** for one or more specified properties, accompanied by an RM **certificate** that provides the **value** of the **specified property**, its associated **uncertainty**, and a statement of **metrological traceability**

ISO GUIDE 33:2015: Ref. Materials: Good Practice in using RMs

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CRM Types

1)Calibrator CRM's (pure materials): Characterized for chemical purity and/or trace impurity. They are used for calibration purposes.

2)Standard Solutions & Gas Mixtures: They are prepared from pure materials by gravimetrical mixing. They are used for calibration purposes.

3)Matrix CRM's (analyte in matrix): Characterized for major, minor and/or trace amount of chemical/biological constituents. Prepared from a matrix having the constituent or addition of constituents to the matrix or preparation of a synthetic matrix with major matrix components. Used for method development, validation and quality control purposes.

4)Physicochemical CRM's: Characterized for properties like melting point, viscosity and density.

5)Reference Objects and Human made artefacts: Characterised for functional properties such as taste, smell and hardness. Microscopy samples characterized for microbiological properties or samples characterized for fiber type are also belong to this group

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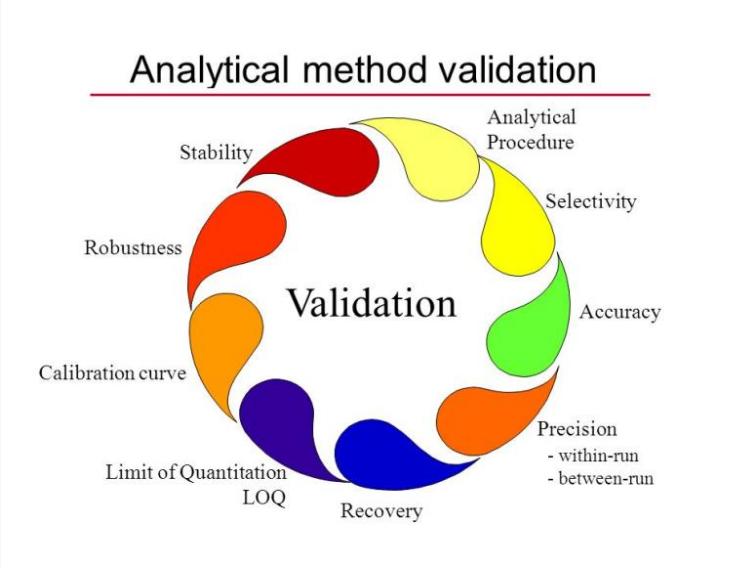
Application Areas of CRMs

- Method development
- Method validation
  - –Evaluation of trueness
  - –Estimation of uncertainty

  } Metrological Traceability
- Calibration
- Method performance charts
  - –Within Lab Quality Control (‘charts’)
  - –Evaluation of analyst or instrument performance
- Proficiency Tests
  - –Validation of Proficiency (external comparison)
- Assigning value to other materials



CRM’s serve for all these applications if they are properly selected and properly used!

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RM vs CRM

– homogeneous – stable	**Material Properties**	– homogeneous – stable
	Additional Investigation	Metrologically valid procedures for value and uncertainty assignment
Statements on homogeneity and stability	**Accompanying Information**	• **Traceability** of property value to a reference system • **Uncertainty** • Homogeneity & Stability • Intended use
➢ **QC applications (precision check: quality control charts)** ➢ **method development**	**Main Applications**	➢ **Calibration** ➢ **Trueness (Bias) control** ➢ **Full method validation** ➢ **All QC/QA applications**

ISO GUIDE 33:2015: Ref. Materials: Good Practice in using RMs

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CRM & RM Production

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Material Processing and Storage According to ISO Guide 34 / ISO 17034

Raw Material Selection and Supply

↓

Material Processing Condition
(Analyte addition, drying, milling, sieving, homogenisation)

↓

Material Filling and Labeling
(bottle cleaning, filling operation, capping, use of glove box, filling machine , gamma irradiation for further stability)

↓

Storage condition (Temperature, humidity, darkness)





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CRM Production

Planning

Material Processing*

Homogeneity Test*

Target?

yes

no

Short Term Stability Test* (Transfer)

Target?

yes

Long Term Stability Test* (Storage)

Target?

yes

Characterisation Study*

Target?

yes

Value & Uncertainty Assignment-
Certificate Preparation

* Activities can be Subcontracted

ISO 9001 · 17025 · Guide 34

TIME

1-3 YEARS

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CRM & RM Selection

Proper Selection

➢ Defined Intended Use	➢Calibration or Quality Control Pure Materials/ Matrix Materials
➢ Measurand and Range	➢Extractable pork, Total pork A CRM with lower property value then lab's LOQ!
➢ Matrix matching and potential interferences	➢Pork in gelatin, pork in protein, matrix differences: eg. Fat content)
➢ Uncertainty and Traceability	➢Is the uncertainty statement suitable acc. to GUM? (ISO/IEC Guide 98-3:2008)? Do homogeneity and stability taken into account?
➢ Certification Procedures	➢Are they in accordance with ISO Guide 34/ ISO 17034
➢ Quality Control Issues	➢Conformity of Laboratories participating in certification ISO/IEC 17025, OIC/SMIIC 36:2020 Accreditation or CIPM MRA CMC's(Calibration and Measurement Capabilities)?
➢ Clear and Transparent Information	➢CRM certificate, Certification Report?
➢ Availability and Price	➢Can the material be reached as quickly as possible to the customer under defined conditions ? (e.g: In dry Ice: < -70 °C)

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Proper Selection

Degree of manipulation of base/raw material
Processing information
(freeze drying, milling, sieving, mixing, bottling

Does it still sufficiently match your laboratory Sample?

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Do we need uncertainty?

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Why do we need uncertainty?

- Uncertainty of the result demonstrates **quality** of measurements
- Defines an appropriate **confidence range**
- It is mandatory for **compliance with limits**
- Provides **comparability** of the results
- It is required by ISO/IEC 17025 for accreditation
- Gives idea about measurement procedure
- Helps **documentation of measurement procedure**
- **Improves** measurement procedure

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Sources of uncertainty

SAMPLING

STORAGE CONDITIONS

INSTRUMENTS EFFECTS

CALIBRATION

REAGENT PURITY

ASSUMED STOICHIOMETRY

MEASUREMENT CONDITIONS

SAMPLE EFFECTS

DIFFERENT OPERATORS

BLANK CORRECTION

RANDOM EFFECTS

MATRIX EFFECTS

CLEAN-UP

DILUTION

Typical sources to consider for many chemical measurements:

Weighing, volumetric operations, purity of standards/RMs, molecular weights, recovery

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TÜBİTAK UME Reference Materials

https://rm.ume.tubitak.gov.tr/

Reference Materials for Meat analysis

A. Dana eti

B. Domuz eti

Şekil 1. Malzemelerden yağların uzaklaştırılması ve parçalama işlemleri

A. Dana eti

B. Do

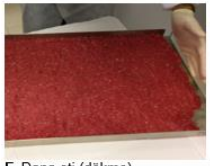
Şekil 2. Dana ve Domuz etlerinin kıyı

A. Dana eti

B. Domuz eti

C. Dana/Domuz eti karışımı

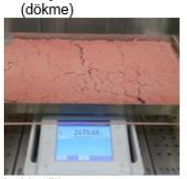
D. Dana/Domuz eti karışımı



E. Dana eti (dökme)

F. Karışım örnekleri (dökme)

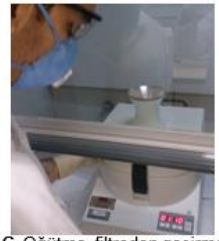
H. Liyofilizasyon



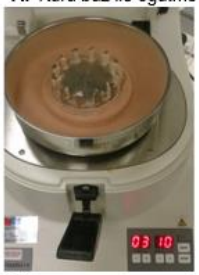
I. Liyofilizasyon sonrası tartım (Dana)

A. Kuru buz ile öğütme

B. Kuru buz ile öğütme



C. Öğütme, filtreden geçirme



D. Öğütme sonrası

E. Öğütme sonrası paketleme

F. Referans malzeme son hali

Şekil 4. Malzemenin liyofilizasyon sonrası toz haline getirme işlemleri ve şişelenmiş ürün

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Reference Materials for Meat analysis

UME RM 1003-2

Pork 10% in Beef Lyofilized Powder

UME RM 1003-1

Pork 1% in Beef Lyofilized Powder

UME RM 1004

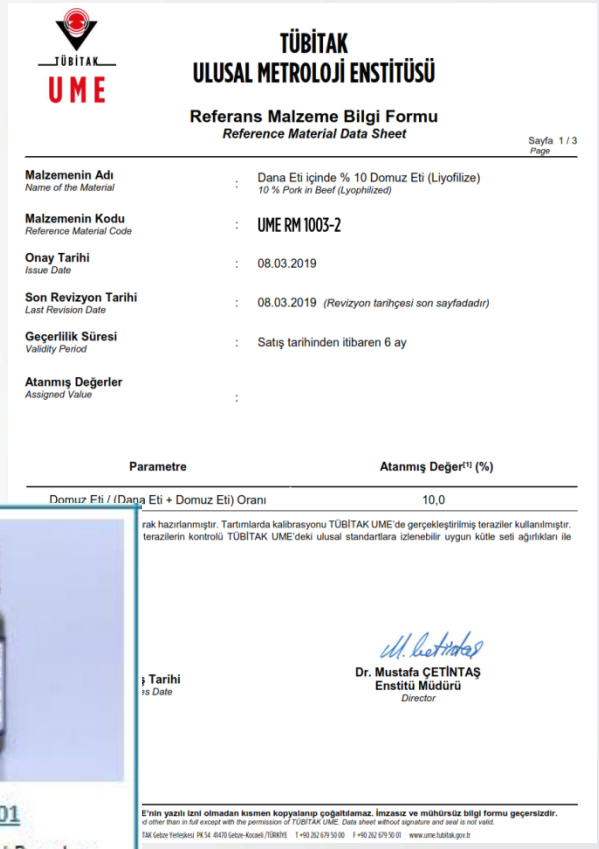
Horse 100% Lyofilized Powder

UME RM 1002

Pork 100% Lyofilized Powder

UME RM 1001

Beef 100% Lyofilized Powder



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ULUSAL METROLOJİ ENSTİTÜSÜ

Referans Malzeme Bilgi Formu
Reference Material Data Sheet

Sayfa 1 / 3
Page

Malzemenin Adı *Name of the Material*	:	Dana Eti içinde % 10 Domuz Eti (Liyofilize) *10 % Pork in Beef (Lyophilized)*
Malzemenin Kodu *Reference Material Code*	:	UME RM 1003-2
Onay Tarihi *Issue Date*	:	08.03.2019
Son Revizyon Tarihi *Last Revision Date*	:	08.03.2019 *(Revizyon tarihçesi son sayfadadır)*
Geçerlilik Süresi *Validity Period*	:	Satış tarihinden itibaren 6 ay
Atanmış Değerler *Assigned Value*	:	

Parametre	Atanmış Değer[1] (%)
Domuz Eti / (Dana Eti + Domuz Eti) Oranı	10,0

rak hazırlanmıştır. Tartımlarda kalibrasyonu TÜBİTAK UME'de gerçekleştirilmiş teraziler kullanılmıştır. terazilerin kontrolü TÜBİTAK UME'deki ulusal standartlara izlenebilir uygun kütle seti ağırlıkları ile

Tarihi
Date

Dr. Mustafa ÇETİNTAŞ
Enstitü Müdürü
Director

E'nin yazılı izni olmadan kısmen kopyalanıp çoğaltılamaz. İmzasız ve mühürsüz bilgi formu geçersizdir.
of other than in full except with the permission of TÜBİTAK UME. Data sheet without signature and seal is not valid.

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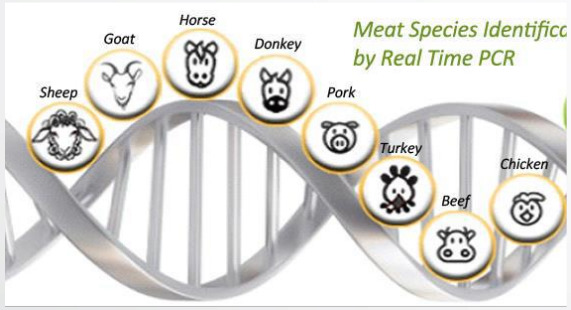
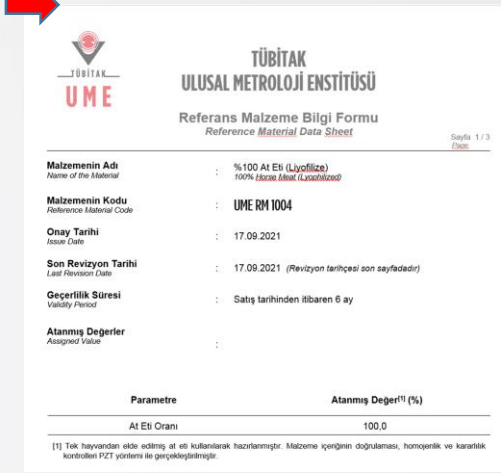
Reference Materials Under Development for Meat Species

Contaminant Level Meat	Main Matrix Meat: Turkey	Chicken	Sheep	Horse	Beef	Pork	Fish	Camel	Donkey
Turkey									
Chicken		100 % Chicken ISIRI/INSO							
Sheep			100 % Sheep ISIRI/INSO						
Horse									
Beef					100 % Beef				
Pork		Pork in chicken NMIM	Pork in sheep NMIM		1 %, 10 % Pork in Beef	100 % Pork	Pork in fish NMIM		
Camel								100 % Camel SASO	
Donkey									100 % Donkey SASO



Meat Species Project- II

- Horse – UME RM 1004
- Chicken
- Duck
- Camel
- Sheep
- Goat
- Salmon



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Referans Malzeme Bilgi Formu
Reference Material Data Sheet

Sayfa 1 / 3
Page

Malzemenin Adı *Name of the Material*	:	%100 At Eti (Liyofilize) *100% Horse Meat (Lyophilized)*
Malzemenin Kodu *Reference Material Code*	:	UME RM 1004
Onay Tarihi *Issue Date*	:	17.09.2021
Son Revizyon Tarihi *Last Revision Date*	:	17.09.2021 *(Revizyon tarihçesi son sayfadadır)*
Geçerlilik Süresi *Validity Period*	:	Satış tarihinden itibaren 6 ay
Atanmış Değerler *Assigned Value*	:	

Parametre	Atanmış Değer[1] (%)
At Eti Oranı	100,0

[1] Tek hayvandan elde edilmiş at eti kullanılarak hazırlanmıştır. Malzeme içeriğinin doğrulaması, homojenlik ve kararlılık kontrolleri PZT yöntemi ile gerçekleştirilmiştir.

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Reference Materials for Food Safety

UME RM 9921 Infant Formulae	UME CRM 1209 Elements in Seabass Fish Tissue	UME RM 9923 Red Papper Flakes	UME RM 9920 Tomato Paste	UME CRM 1312 Honey (unadulterated)	UME CRM 1311 Fructose	UME CRM 1310 Glucose	UME CRM 1309 Sucrose
UME RM 9912 Animal Feed	UME RM 9910 Mineral oil in edible oils	UME RM 9909 Aflatoxins and fat in hazelnut	UME RM 9907 Sulfur Dioxide in Apricot	UME CRM 1313 Honey (adulterated)	UME RM 9919 Red Pepper Flakes	UME CRM 1301 Chloramphenicol	UME CRM 1202 Elements in Hazelnut
UME RM 9905 Filtered Flower Honey	UME RM 9904 Black Tea	UME RM 9903 Sunflower Oil	UME RM 9902 Wheat Flour	UME CRM 1302 Aflatoxins in Dried Fig	UME RM 9906 Sunflower Oil	UME CRM 1201 Elements in Spring Water	

https://rm.ume.tubitak.gov.tr/urun_grup_en.aspx?p=3

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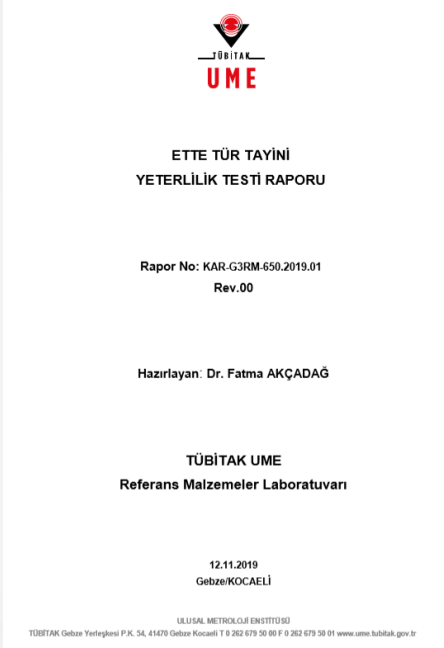
Proficiency Tests

- ✓ **Determination of Meat Species**
- ✓ **Determination of Species in Gelatine**
- ✓ **Det. of Caffeine, Acesulfame K, Benzoic Acid, Saccharin in Drinks**
- ✓ **Determination of Elements (Al, As, Cd, Cr, Cu, Fe, Hg, Mn, Ni, Pb, Sb, Zn, Ag, B, Ba and Se) in Drinking Water**
- ✓ **Determination of pH and PAH in Drinking Water**
- ✓ **Determination of Aflatoxins and Total Fat in Hazelnut Puree**
- ✓ **Det. of HMF, Glucose, Fructose, Sucrose, Diastase, Free Acid in Honey**



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ETTE TÜR TAYİNİ
YETERLİLİK TESTİ RAPORU

Rapor No: KAR-G3RM-650.2019.01
Rev.00

Hazırlayan: Dr. Fatma AKÇADAĞ

TÜBİTAK UME
Referans Malzemeler Laboratuvarı

12.11.2019
Gebze/KOCAELİ

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❖ 2 times a year

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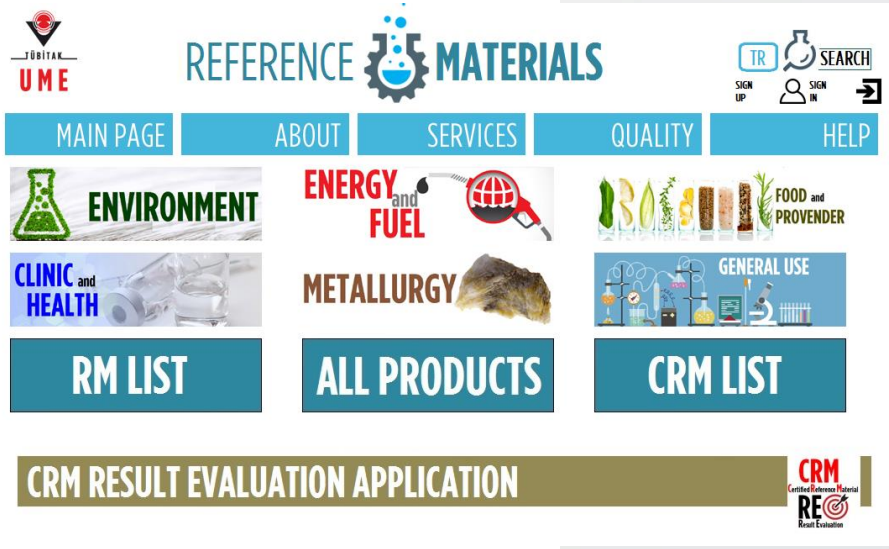
Databases, Producers, Useful Links

Databases:

COMAR	*www.comar.bam.de*
AOAC	http://tdrmdb.aoac.org/
Geological RM's	http://georem.mpch-mainz.gwdg.de/
Clinical CRM's	http://www.bipm.org/jctlm/
BIPM CMC's	http://kcdb.bipm.org/

Some RM Produces and Distributors:

NIST	https://www.nist.gov/srm
EC-JRC-IRMM	https://crm.jrc.ec.europa.eu/
BAM	https://www.bam.de
IAEA	https://nucleus.iaea.org/rpst
NMIJ	www.nmij.jp/english
KRISS	www.kriss.re.kr/eng
NRC	http://www.nrc-cnrc.gc.ca/eng
NMIA	http://www.measurement.gov.au
UME	https://rm.ume.tubitak.gov.tr
LGC	www.lgcstandards.com
Sigma-Aldrich	www.sigmaaldrich.com



Useful Links: *https://ec.europa.eu/jrc/en/reference-materials/useful-links*

THANK YOU

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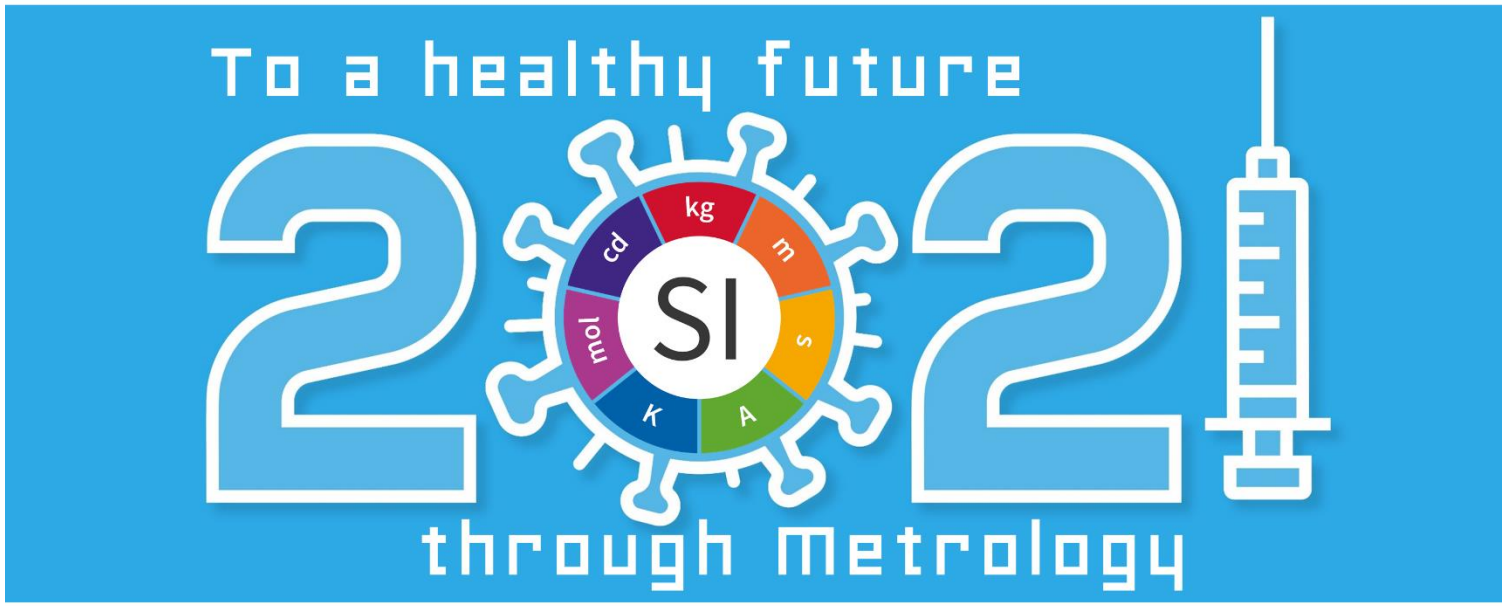
TÜBİTAK NATIONAL METROLOGY INSTITUTE



WE WILL DEFEAT THIS TOGETHER

TRUE MEASUREMENT EXCELLENCE

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