

# Laboratoire national de métrologie et d'essais

Dr Jean-Rémy FILTZ











### **Thermal and Optical Metrology**

# A panorama of Key recent developments at LNE

Metrology for Health, Environment and Energy



#### **Outline**



- 1 Main actions for sustaining the Industry
- 2 New triggers driving developments in metrology
- 3 4 examples in 3 domains
- 4 Conclusions



### Main actions for sustaining the Industry





National traceability chain

**ISO/CEI 17025** 

**ISO/CEI 17025** 

**LNM** National standards LNE Associate Transfer laboratories to users Accredited laboratories calibration Industrial measurements



### Main actions for sustaining the Industry



- ☐ Perform missions devoted to National Metrology Institute
  - Develop National Standards for basic units
  - Maintain National standards and develop sub units
  - Guaranty traceability to industry
  - □ Provide technical assistance to industry in supporting and developing Metrology



#### **Applied Research**



 Development of standards and specifications Studies for innovative technical solutions



 Design, development and fine adjustment of test and calibration facilities • Experiments and statistical analysis programmes



### New triggers driving developments in metrology

Sustainable development

**Global warming** 

**Kyoto Protocol** 

**European Directives, Regulation,...** 

Grenelle de l'environnement

Agenda 21...

International Issues

le Grenelle Environnement

National or European Issues



## New triggers driving developments in metrology



- ☐ Help governmental agencies to prevent Society from risks
- ☐ Support policies related to public health
- Support French and European industry...
- ☐ Act for constructing a European Metrology infrastructure
- Support and take part in advanced metrology
- Disseminate Metrology and Knowledge Transfer



#### **Environment: developments at LNE**







Indoor air quality
NO<sub>2</sub>, VOCs
Aldehydes
GhG
Heavy metals,
HAP, PCB,
Endocrine disruptors
Speciation
Pesticides, OCP

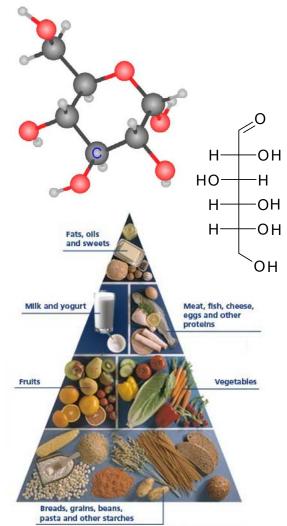




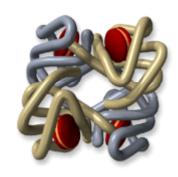


#### **Health: developments at LNE**

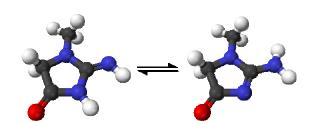




Traceability of bio-medical analysis
Glycosylated hemoglobin - Diabetes
Measuring out Glucose concentration
Blood pressure metrology
Ethylometry
Creatinine
Tonometry, Spirometry
UV radiometry
Metrology to prepare for a potential
pandemic influenza
Metrology supporting Nano-particles







**Diabetes food pyramid** 



Santiago de Querétaro, México, 2008-10-23

#### **Energy: developments at LNE**

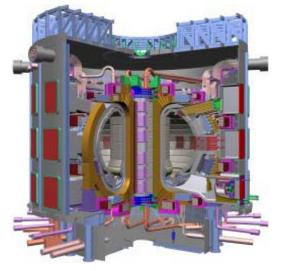








Help saving energy
Power and Energy,
Energy conversion and
Metrology;
LEDs highlighting
Solar
Biofuels...



Iter: International thermonuclear experimental reactor



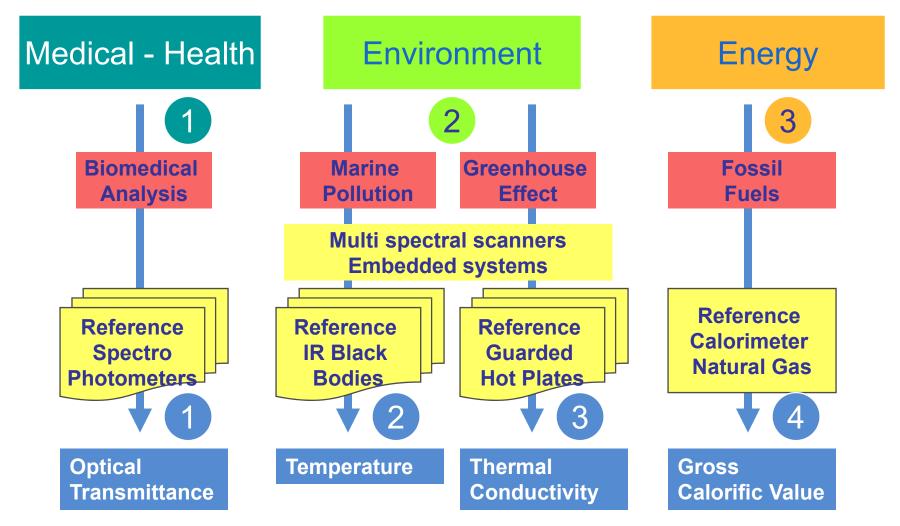




## Developments: 4 examples in 3 domains



In the field of Thermal and Optical Metrology

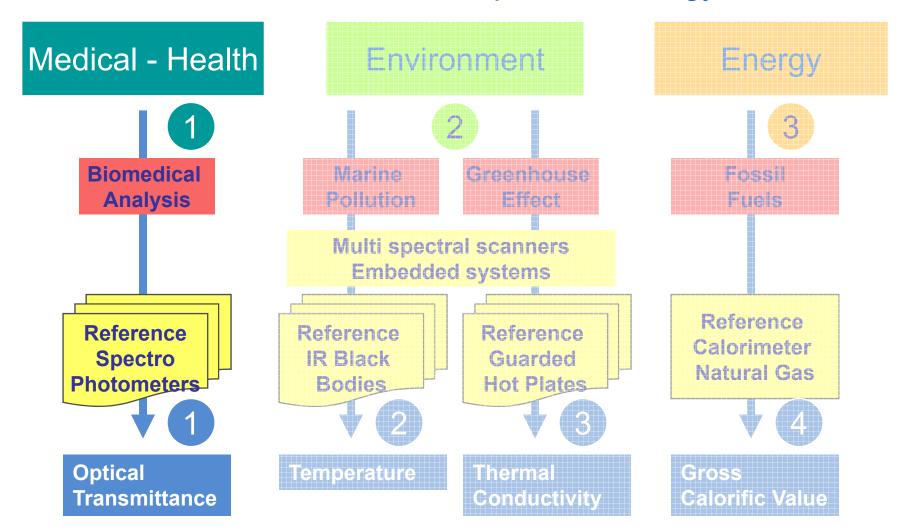




#### **Medical - Health**



#### In the field of Thermal and Optical Metrology

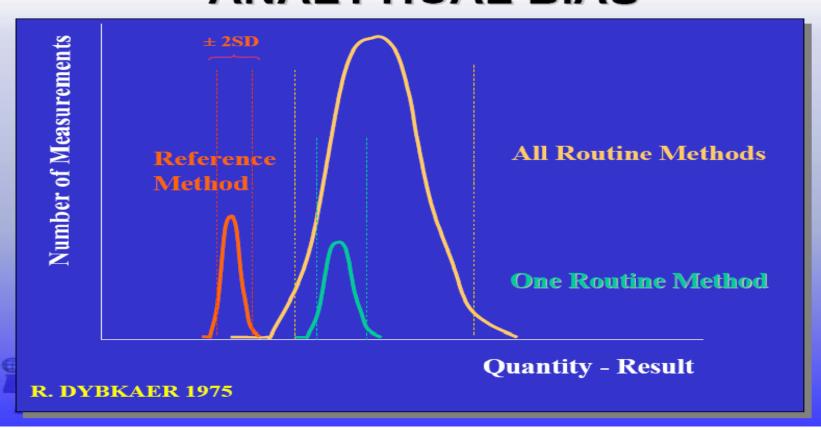




### **Biomedical analysis and Traceability**







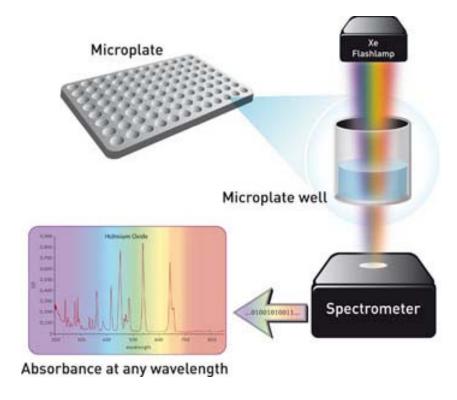
Traceability to references: a common requirement Directive 98/79 DIV, ISO 17511, ISO 15195



## Biomedical laboratory and 'Optical properties of liquids



Testing laboratories using optical instruments such as microplates readers for making biological and bacteriological analyses in the field of health, food or the environment need to be traceable to SI



Source :www.bmglabtech.com



# Biomedical laboratory and Optical properties of liquids



For quality assurance, the sets of filters

fitted into quality control plates,

used to check the accuracy of

microplates readers, require



### calibration in spectral optical density



# Biomedical laboratory and Optical properties of liquids

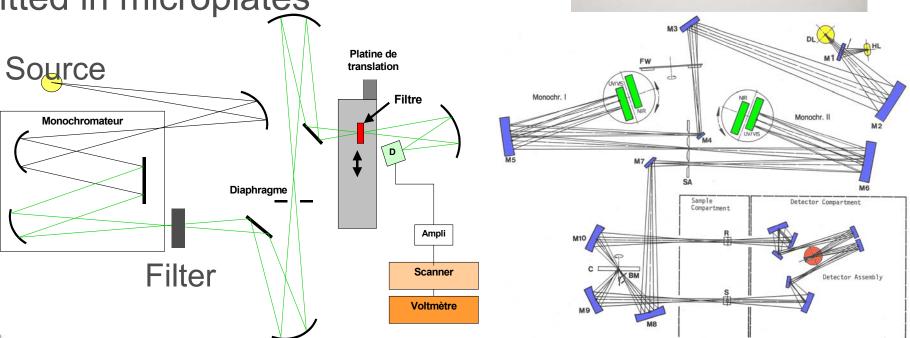
Kkg S SIMPOSIO Metrología Metrología Metrología

LNE has developed specific ways to calibrate

in regular spectral transmittance

the very small reference filters

fitted in microplates





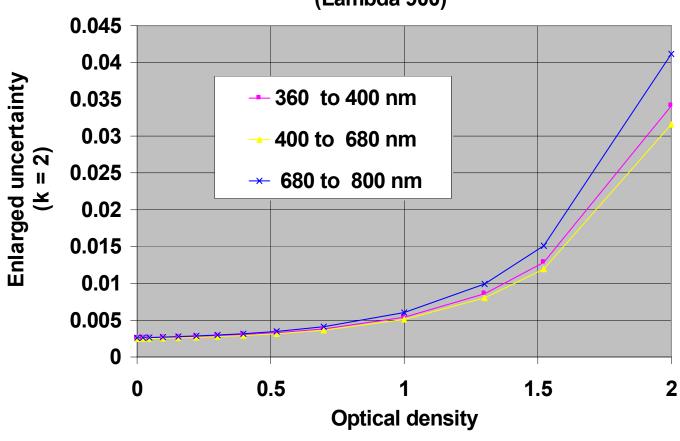
Santiago de Querétaro, México, 2008-10-23 Thermal and Optical Properties of Materials

A Panorama of Key Recent Developments at LNE

# Biomedical laboratory and Optical properties of liquids



# Enlarged uncertainty on spectral optical density (Lambda 900)

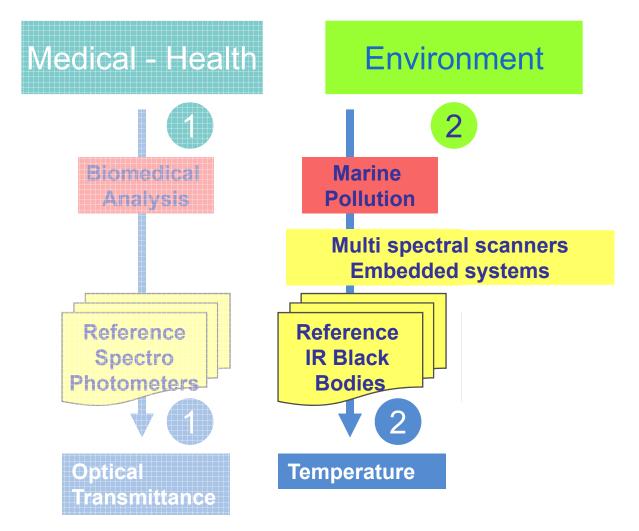




#### **Environment**



#### In the field of Thermal and Optical Metrology





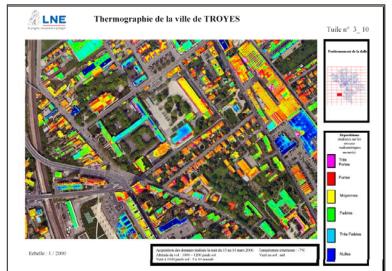
# Help public agencies and local communities to take decision



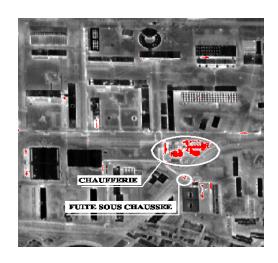
Monitoring water cooling of electrical power plants



Design and set up of multi-spectral embedded systems for french coast guards (Polmar)



Diagnosis of heating network and leak detection (Lyon)

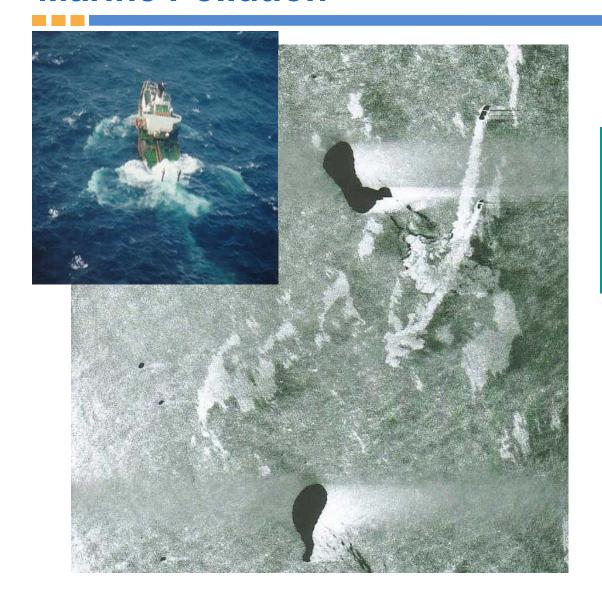


Airborne thermography of many towns (Marseille, Troyes,...)









# After the sinking of cargo Prestige

# Prestige infrared image of split spills

Name: Prestige

Date: 13/11/2002

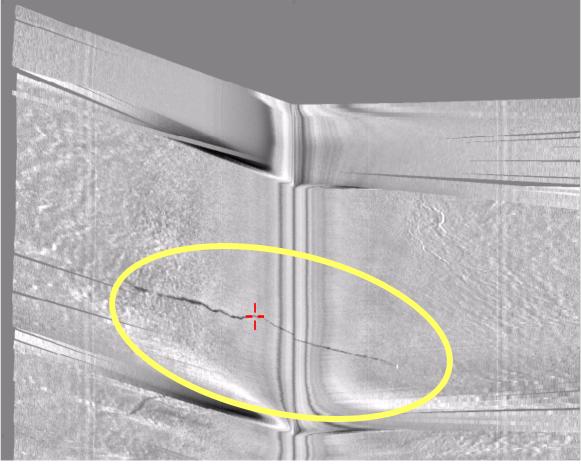
Place: Spain

Pavilion : Bahamas Product: crude oil Quantity : 77 000 t

Oil spill : Spain, France, Portugal







# TYPICAL Pollution Detection System Functions Developed

**DETECT: SLAR Image** 

Polluted wake (black line) is detected.

It is 20 nautical miles long.

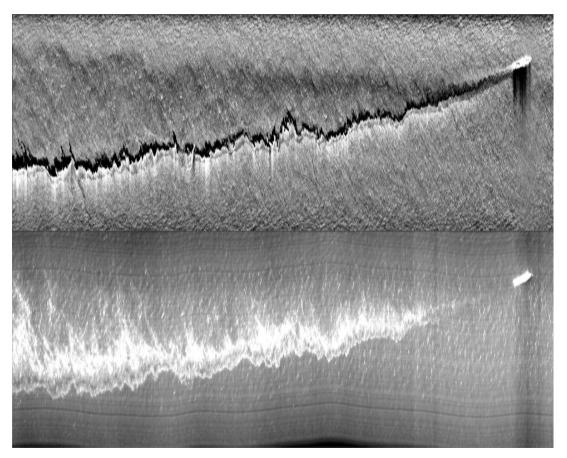
This image and all data are recorded in the data base.



SLAR Electronic units head.







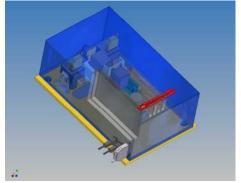
México, 2008-10-23

#### **CONFIRM: SCANNER Image**

The pollution is confirmed.

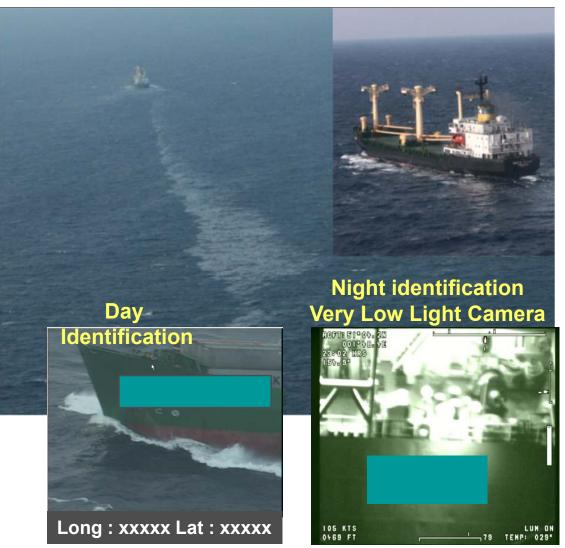
If necessary, the oil spill surface is measured and the volume is computed.

#### **Scanner Head**









# IDENTIFY : Photos or Camera Images

The ship is identified and all views are recorded in the data base.



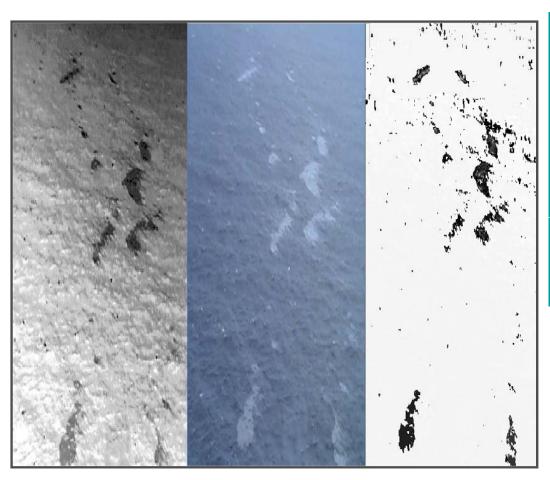








#### **Scanner integrated functions**

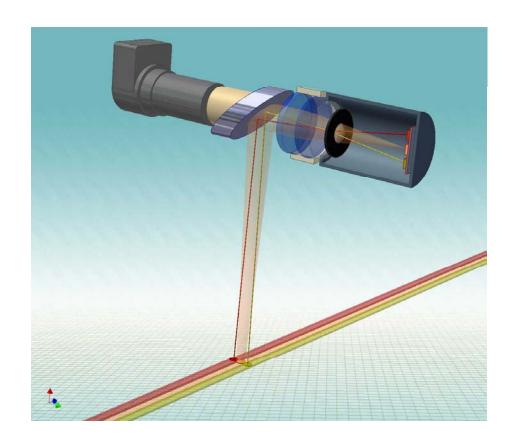


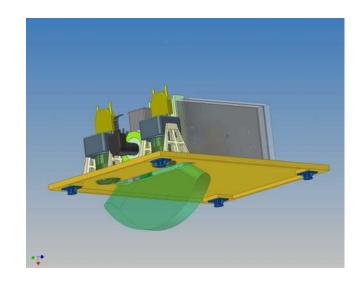
# Multi channel Display and processing

- Channel combinations
- Arithmetic operations
- Neural Network classification (development)







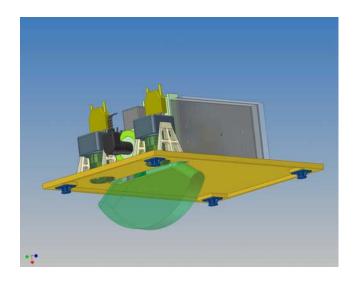


### **STORM Multispectral scanner**



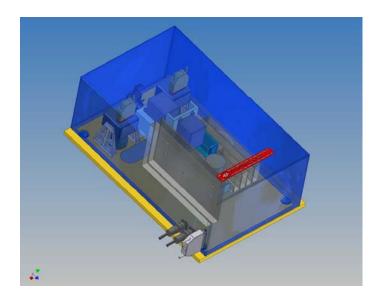
## **STORM Multispectral scanner**





#### Scanner Head





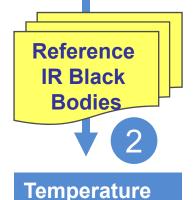






**Metrology of the infrared systems** based on Standard IR Black Body

**Cavities** 







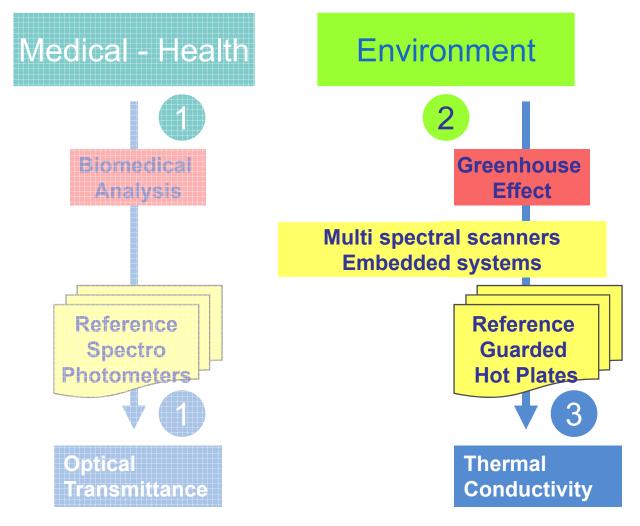


México, 2008-10-23

#### **Environment**



#### In the field of Thermal and Optical Metrology



Santiago de Querétaro,

México, 2008-10-23



#### **Environment: Greenhouse Effect**



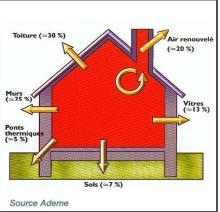
### Saving energy in existing houses

Kyoto protocol has been ratified in 2005 and is now applied.

One objective: increase energy efficiency

Construction = sector most energy consumer in

**France** 





#### **Environment: Greenhouse Effect**



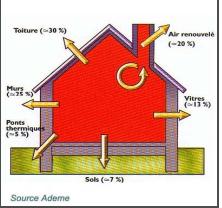
#### Saving energy in existing houses

The LNE's answer: airborne infrared thermography

Convince citizen to renovate houses and to improve thermal insulation

For instance, the proportion of heat loss through the roof is around 25-30 %, 30-35% through the walls, 10% through the

windows...





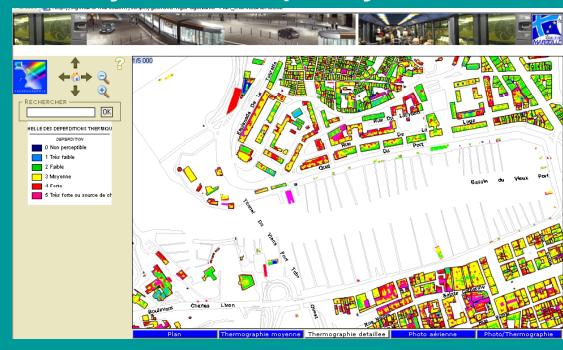
#### **Environment: Greenhouse Effect**



One cost-effective way to save energy and reduce GHG emission

# Service to Local Communities or Public Authorities Mapping of heat losses Marseille, Troyes, Blois, Epernay...







#### **Thermal Properties of Materials**



#### Context

The thermal properties of materials are studied at LNE for a long time...

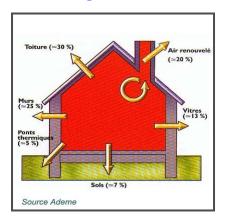
#### Main concerned industrial sectors

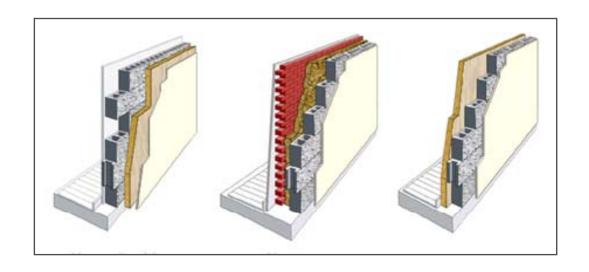
**Automotive industries** 

**Aeronautics / Defence** 

**Energy / Nuclear** 

#### **Building**







#### **Thermal Properties of Materials**

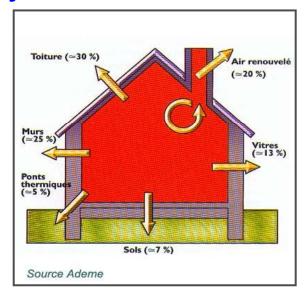


#### LNE has been developing several facilities to study:

- Thermal conductivity and thermal diffusivity
- Specific heat and enthalpy

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- Thermal Expansion
- Emissivity, Reflectance, Transmittance





#### **Thermal Properties of Materials – Range and Methods**



#### **Direct Method**

**Guarded Hot Plate** 

- 10 °C / 60 °C

23 °C / 200 °C

#### **Indirect Method**

 $\lambda = \mathbf{a} \cdot \mathbf{p} \cdot \mathbf{c}_{\mathbf{p}}$ 

23 °C / 800 °C

#### **Drop Calorimetry**

23 °C / 1000 °C

#### DSC

- 100 °C / 800 °C

#### **Laser pulse Method**

23 °C / 1500 °C

+

**Dilatometry** 

- 100 °C / 1000 °C

**Emissivity** 

- 20 °C / 800 °C

**Immersion Method** 

23 °C

**Dilatometry** 

- 100 °C / 1000 °C



α

 $\alpha$ 

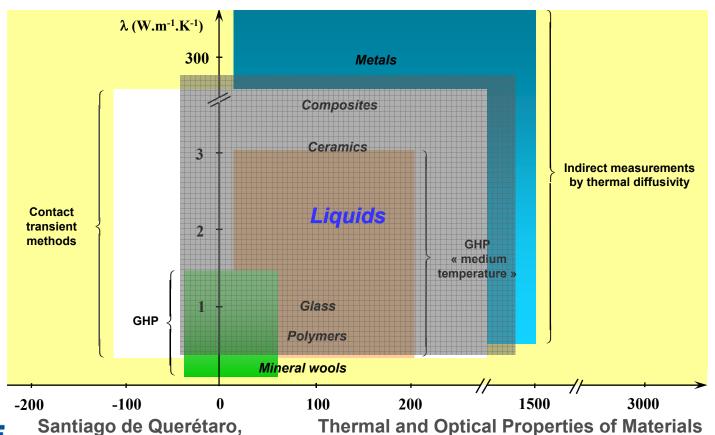
#### **Thermal Properties of Materials**



#### **Thermal Conductivity - Materials and Means**



Measurement means of LNE cover a large temperature range





Santiago de Querétaro México, 2008-10-23

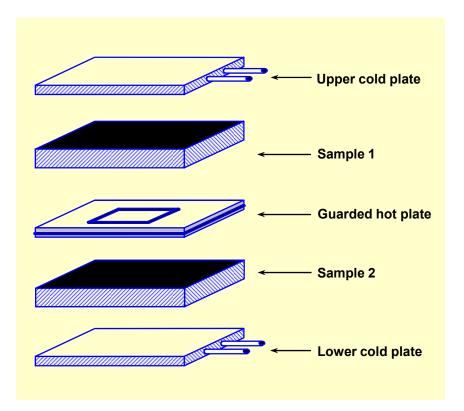
Thermal and Optical Properties of Materials

A Panorama of Key Recent Developments at LNE

### **Thermal Properties of Materials**



### Thermal Conductivity - Guarded Hot Plate (GHP)



- Measurement by guarded hot plate (ISO 8302)
- Temperature range from 5 °C to + 50°C
- Low conductive materials (λ<0.5 w/m.K)</li>
- Uncertainty (k=2) : from  $\pm$  0.5% to  $\pm$  2 %



Development of a new GHP for medium temperatures (up to 500 °C)



### Insulation Materials – a new GHP

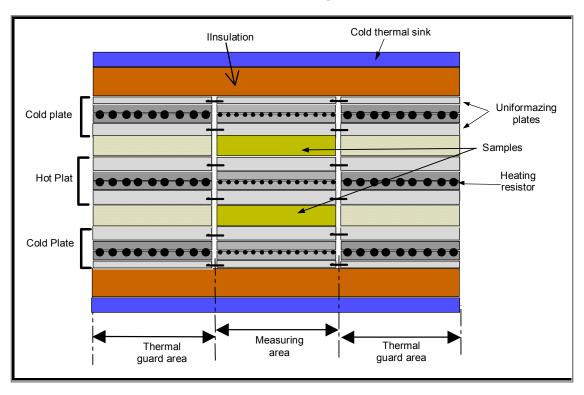


Thermal conductivity range: 0.1 to 10 W.m<sup>-1</sup>.K<sup>-1</sup>

Temperature range: 23 to 500 °C

The goal is to measure thermal conductivity with a relative uncertainty

from 1to 5%.



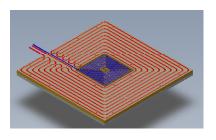
Cross section view of the "stacking"



### **GHP Reference set-up**







Patterns of the heating resistors of the measuring area and the thermal guard ring



General views of the LNE set-up and of the heating plates.



### **GHP Reference set-up**

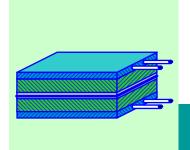


Material	Specimen thickness	Temperature (°C)	Thermal conductivity (W·m <sup>-1</sup> ·K <sup>-1</sup> )	Uncertainty (k=2) (W·m <sup>-1</sup> ·K <sup>-1</sup> )
Rubber	20 mm	20	0.2822	0.0068
		70	0.3168	0.0074
Silicone	10 mm	20	0.2211	0.0037
		70	0.2156	0.0035
PVC	20 mm	20	0.1403	0.0068
		70	0.1815	0.0074

Materials used for checking







# Pilot study for limited comparison on thermal conductivity measurement by Guarded Hot Plate

Jean-Rémy FILTZ - Bruno HAY - Benoît DOUCET **Jacques HAMEURY Thermal and Optical Division** 



México, 2008-10-23



### The main characteristics of the measurement protocol are the following:

- Method: ISO 8302
- - Expanded polystyrene board (specific batch produced by Lafarge - France) - thickness = 35 and 70 mm
- Programme of measurements:
  - ► 1 measurement at 10 °C
  - ► 4 measurements at 23 °C
  - ▶ 1 measurement at 40 °C

- MW thickness = 35 mm
  - EPS thickness = 35 mm
  - **EPS** thickness = 70 mm



Measurements performed successively by all the participants on the same specimens

with  $\Delta T = 20 \text{ K}$ 





NMI's involved in this pilot study.



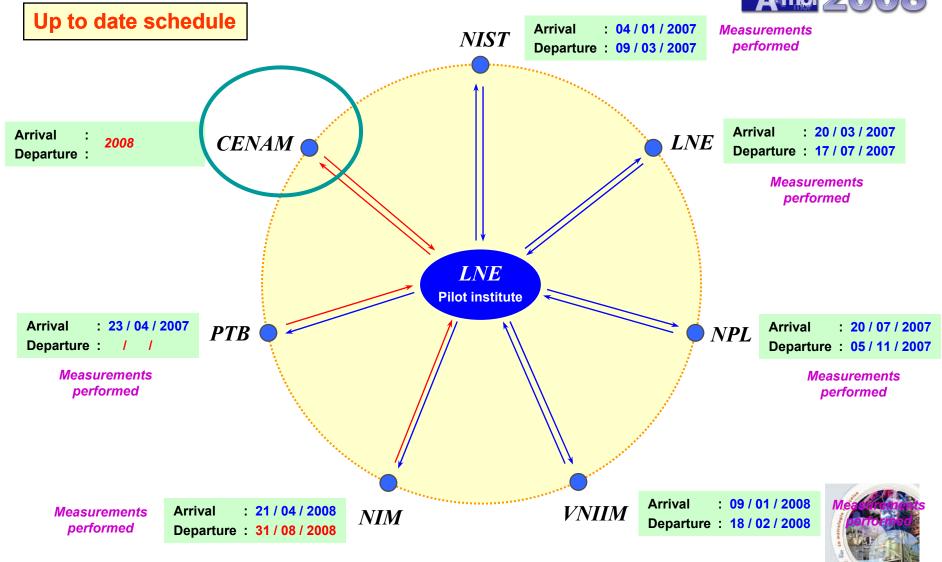




### **Summary of the measurement program**

Laboratory	GHP	Dimensions (mm)	Program of measurements	Materials and thicknesses (mm)
NIST (USA)	Double	φ 1016	10 °C (x1) 23 °C (x4) 40 °C (x1)  23 °C (x4) 40 °C (x1)	MW (35 mm) EPS (35 mm) EPS (70 mm)
LNE (FR)	Double	610 x 610		
NPL (UK)	Single	610 x 610		
VNIIM (RU)	Double	φ 330		WIW (35 mm) EPS (35 mm)
NIM (CN)	Single	φ 330		MW (35 mm) EPS (35 mm) EPS (70 mm)
PTB (DE)	Single	φ 100		EPS (20 mm) EPS (25 mm)
	Transient Hot Bridge	105 x 50	40 °C (x1)	MW (35 mm) EPS (35 mm) EPS (70 mm)





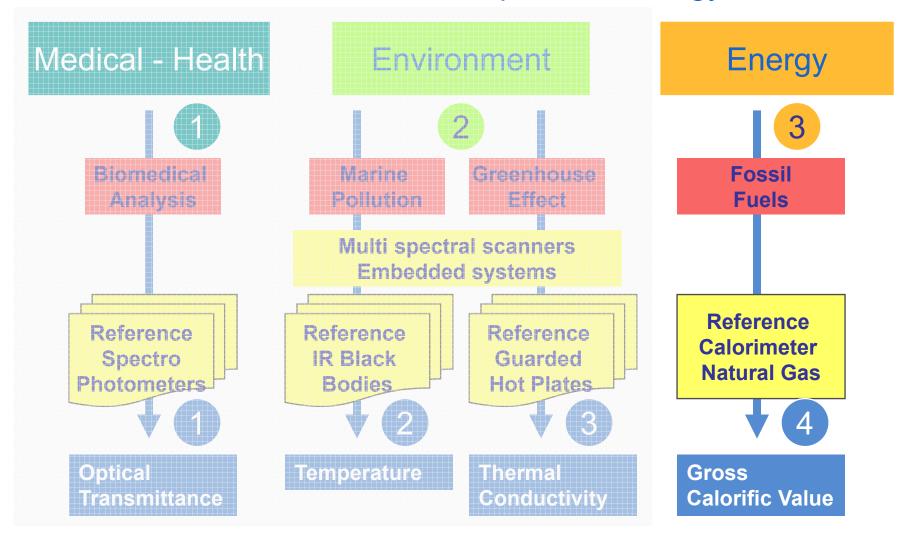


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### **Energy**



### In the field of Thermal and Optical Metrology



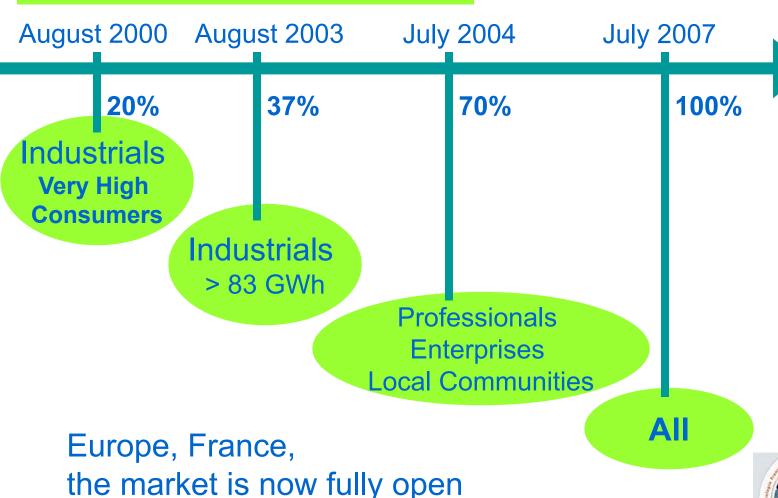


# **Background**











# **Background**







Today 2008







# **Background**

### Metrology & Natural Gas:



## What does it mean in Europe and in France?



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- Opening of the natural gas market in Europe
  - Non constant natural gas composition flowing in the pipes
  - Accurate energy measurements \* needed

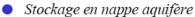
Improvement of the transported and distributed gas transactions by more accurate measurement of natural gas gross calorific value (GCV)

•GCV = Quantity of heat released by complete combustion of a specified gas quantity

# France: Natural Gas, Pipelines and Transportation







O Stockage en cavités salines

Station de compression

🗱 Gisement de gaz naturel

— Canalisation GDF

— Canalisation GSO

— Canalisation Total

— Canalisation CFM

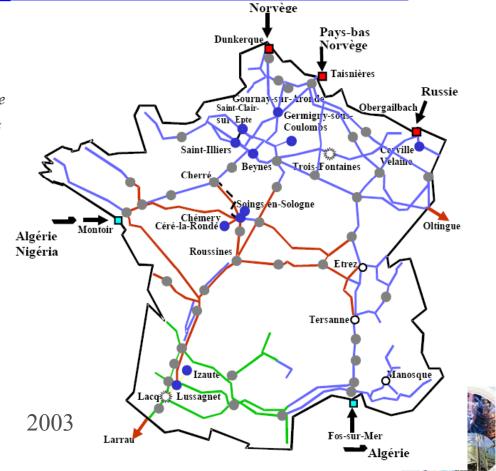
Canalisation en projet

→ Arrivée de gaz naturel

Méthanier

Terminal méthanier

Terminal de réception





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### México: Natural Gas network



PROSPECTIVA DEL MERCADO DE GAS NATURAL 2007-2016





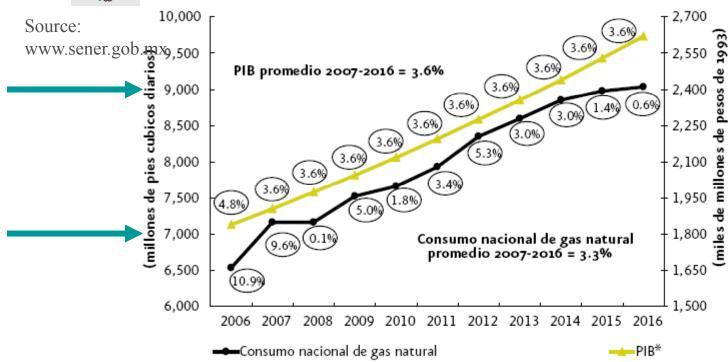
### Gas Natural: Consumo nacional 2007-2016

Kkg SIMPOSIO
Metrología
Metrología
Mol 2008

PROSPECTIVA DEL MERCADO DE GAS NATURAL 2007-2016



### Crecimiento de la demanda de gas natural y el PIB en México, 2006-2016



<sup>\*</sup>Las cifras están referidas al año base de 1993. Fuente: CAPEM e IMP.



kg SIMPOSIO
Metrología
Amol 2008

Prospectiva del mercado de gas natural 2007-2016



www.sener.gob.mx

...Desagregación de tipos de combustible y transformación a unidades comunes (Btu's, GWh, Joules, etc.)....

-> Breakdown of fuel rates and conversion to common units (Btu's, GWh, Joules, etc.)...



### **Gas Calorimetry**





### **NG: GCV Determination**

**Direct Method Calorimetry** 



**Reference Calorimeter** 

Non-Direct Method
Gas Chromatography
+ GCV values (ISO 6976)

**Reference Gas Mixtures** 



Santiago de Querétaro, México, 2008-10-23

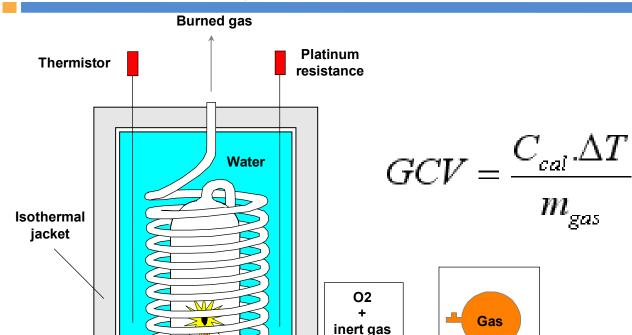
# **Gas Calorimetry – Non-Direct Method** Natural Gas pipeline Metrology **Flow Temperature Pressure** Chromatography **Calculation** ISO 6976 **Correction factors** Data: flow, t, p, GCV, Energy Data Checking exploitation Santiago de Querétaro, **Thermal and Optical Properties of Materials**



México, 2008-10-23

A Panorama of Key Recent Developments at LNE

# **Gas Calorimetry - Reference Method**





www.nap.edu



Frederick X Rossini

**Frederick Dominic Rossini** 

1899 - 1990

1928 - 1950 (NBS)

1966 - 1970 ( 1<sup>st</sup> Pdt CODATA)

C<sub>cal</sub> is the heat capacity of the calorimeter (J.K<sup>-1</sup>)

Gas

Weight measurement of gas

before and after combustion

with a mass comparator

m<sub>gas</sub> is the mass of burned gas (kg)



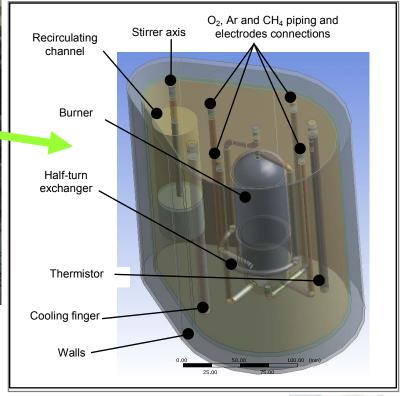
Santiago de Querétaro, México, 2008-10-23

**During combustion** 

### **Gas Calorimetry - Reference Method**







### New design of the LNE Calorimeter

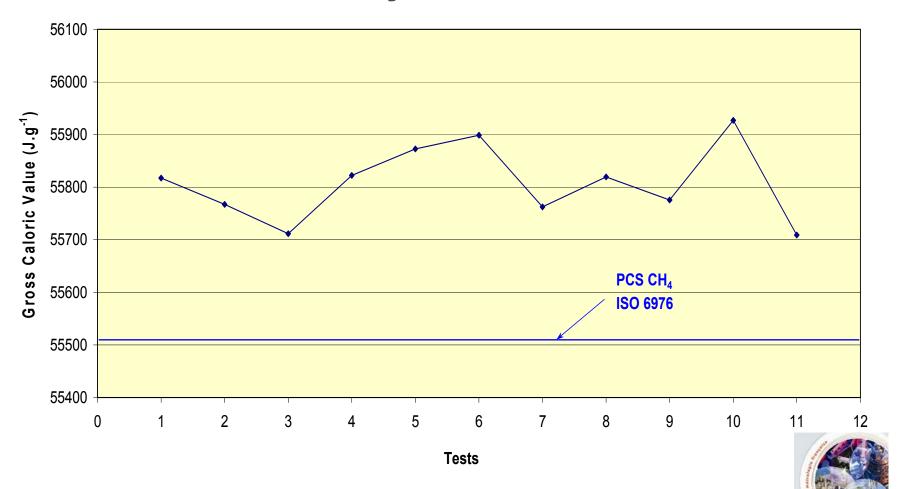
México, 2008-10-23



# **Gas Calorimetry - Reference Method**



# **Preliminary results**





### **Conclusions**



Mainly with regards to the Metrology at the transfer level but not only...:

- ☐ On the basis of its regular missions, LNE as all NMIs can provide a useful support to the industry or relatively to the needs of the society. We do it!
- New triggers driving developments in metrology have recently arisen. They lead our institute to strengthen our links with the Industry, the society and the governmental agencies. We try to answer to these different challenges in a practical way. We do it!
- ☐ As you have seen through the different illustrations, there is no barrier for Metrology. Basic Competencies of NMIs are a real power to be exploited for constructing innovative solutions useful for meeting a sustainable development. We think it, we try to do it and we would like to share it with you!





Laboratoire national de métrologie et d'essais (LNE)

Organisme de référence pour la métrologie française avec le concours de :

### 3 autres laboratoires nationaux

LNE-INM

Conservatoire National des Arts et Métiers

LNE-LNHB

Commissariat à l'Energie Atomique

LNE-SYRTE

Observatoire de Paris

#### et 6 laboratoires associés

- Centre Technique des Industries Aérauliques et Thermiques (CETIAT)
- Ecole Nationale Supérieure d'Arts et Métiers de Paris (ENSAM)
- Franche-Comté, Electronique, Mécanique, Thermique et Optique - Sciences et Technologies (FEMTO-ST)
- Institut de Radioprotection et de Sûreté Nucléaire (IRSN)
- Laboratoire Associé de Débitmétrie Gazeuse (LADG)
- Observatoire de Besançon

### www.lne.fr

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