Asia-Pacific
Economic Cooperation

Asia-Pacific
Legal Metrology Forum

Handbook on Metrology in Food Safety, Agricultural Products Workshop

APEC/APLMF Training Courses in Legal Metrology
(CTI 12/2008T)

Sept. 23-25, 2009
at the Rex Hotel in Ho Chi Minh City, Viet Nam

APEC Secretariat
35 Heng Mui Keng Terrace
Singapore 119616
Tel: +65-6775-6012
Fax: +65-6775-6013
E-mail: info@apec.org
Website: www.apec.org

APLMF Secretariat
Department of Metrology, AQSIQ
No. 9 Madiandonglu, Haidian District, Beijing, 100088, P. R. China
Tel: +86-10-8226-0335
Fax: +86-10-8226-0131
E-mail: sec@aplmf.org
Website: www.aplmf.org

APEC#209-CT-03.4 ISBN 978-7-5026-2988-5

Workshops on Metrology in Food Safety, Agricultural Products and Product Safety
Sept. 23 – 25, 2009 at the Rex Hotel in Ho Chi Minh City, Viet Nam

Photos taken at the workshop in Ho Chi Minh City, Viet Nam

Contents

1 Foreword ····· 1

2 Summary Report ····· 3

3 Program ····· 6

4 Participants List ····· 15

5 Lectures

Section 1: Legal Metrology Infrastructure for Quality Measurements of Agricultural Products (Chair: Dr. Matsumoto, Co-Chair: Dr. Osman Zakaria)

1. Traceability, Uncertainty Analysis and Current Situation of OIML R59 on Rice Moisture Meters by Dr. Tsuyoshi Matsumoto (NMIJ, Japan) ····· 17
2. Evaluation of Uncertainty of the Drying Method by Mr. Hideyuki Tanaka (NMIJ, Japan) ····· 23
3. Grain Moisture Content Measurement Methods and Traceability in China by Ms. Zhang Weiwei (Heilongjiang Institute of Measurement and Verification, P. R. China) ····· 29
4. Rice Moisture Meters by Device by Mr. Sakchai Hasamin (CBWM, Thailand) ····· 35
5. Grain Moisture Measurements in Mexico by Mr. Enrique Martines Lopez (CENAM, Mexico) ····· 40
6. The Traceability of Rice Moisture Meter in Indonesia by Ms. Sri Astuti (Metrological Training Centre, Indonesia) ····· 47
7. Checking the Calibration of Rice Moisture Meters by Mr. Warachai Triarun (CBWM, Thailand) ····· 50

Session 2: Measurement Method and CRMs Underpinning Food Safety (Chair: Dr. Laly Samuel, Co-Chair: Dr. Wong YC)

1. Preparation of Ref. Material of Melamine in Milk Powder by Dr. Yiu-chung WONG (Hong Kong Government Laboratory, Hong Kong, China) ····· 56
2. Assurance of Measurement of Foods and Agricultural Products in China by Prof. Zhang Qing-he (NIM, P. R. China) ····· 62
3. Overview of the Legal Metrology System in Food Safety, Agricultural Products and Product Safety in the Philippines by Ms. Marilyn Fos (ITDI, Philippines) ····· 71

4. Development of Domestic Infrastructure to Support Legal Control of Food Safety, Agricultural Products and Product Safety In Malaysia by Dr. Osman Zakaria (SIRIM, Malaysia) ····· 78
5. Papua New Guinea Domestic Agricultural Research Institute by Mr Peter Corbett (NARI, Papua New Guinea) ····· 89
6. Metrology, Standardization, Testing and Quality Management as tools in Food Safety: Status at the FoodDevelopment Center of the Domestic Food Authority, Department of Agriculture by Dr. Amelia W. Tejada (Food Development Center, Philippines) ····· 95
7. A Collaborative Approach to Establish Traceability in Chemical Measurement and Food Safety in New Zealand by Dr. Laly Samuel (Measurement Standards Laboratory, New Zealand) ····· 104
8. Overview of Measurement Standards in Agriculture Facilities in Papua New Guinea by Ms. Edna Egu (NISIT, Papua New Guinea) ····· 118
9. Domestic Metrology Infrastructure for Food Safety in Mexico by Dr. Norma Gonzalez (CENAM, Mexico) ····· 122

Session 3: Summary and Discussion (Dr. Tsuyoshi Matsumoto, Dr. Zhang Chao)

6 Training on Moisture Meters
(by Mr. Hiroshi Yamahira, Kett Electric Lab., Japan) ····· 127

Foreword

This booklet is one of outcomes of the APEC Seminars and Training Courses (APEC TILF projects, CTI 12/2008T) in Legal Metrology entitled "Workshops on Metrology in Food Safety, Agricultural Products" held on Sept. 23-25 2009 in Ho Chi Minh City, Viet Nam.

Two workshops on Metrology in Food Safety, Agricultural Products and Product Safety were held in Thailand and Hangzhou in February 2007 and June 2008, respectively. This workshop is to review the outcomes of the previous two workshops and also to develop the concrete action plan which would be executable for legal metrology experts in the food safety area. It was organized by APLMF and supported by Directorate for Standards & Quality (STAMEQ), Viet Nam. In parallel, the moisture meter training course supported by Kett. Elec. Co. Japan was conducted on the demand of some member economies. Having this result, I would like to extend my sincere gratitude to all the staffs of STAMEQ who have made efforts for the outstanding preparation and all the speakers from member economies who contributed to this workshop. I would also like to thank Kett. Elec. Co., Japan for supporting the moisture meter training program. Also, special thanks should be extended to the APEC Secretariat for their great contributions.

This workshop/training program was identified after the survey conducted among the APEC member economies to find their prior needs as well as possible resources available in the region. The main goal of this workshop was to bring together experts from APEC/APLMF member economies in both legal metrology and scientific metrology field to build on the outputs from the first two workshops. The speakers have presented the case studies that review existing metrological infrastructure within APEC/APLMF economies with a view to identifying what is working and possible solutions to issues yet to be addressed. The contents of the workshop were classified into two key topics: Legal metrology infrastructure for quality measurements of agricultural products and Measurement method and CRMs underpinning food safety. Presentations on each topic were delivered by the speakers that are experienced in the fields.

In this view, the workshop provided an important opportunity for the experts in the Asia-Pacific region to clarify the present situation on the development of legal metrology infrastructure of food safety, agricultural trading in the member economies, share information on metrological requirements for food safety set in the region, introduce methods for quality evaluation, explain technical procedures for verification of measuring instruments and discuss the issues that exist in the region. I would like to say that this workshop have laid the solid foundation for the development of concrete and executable action plan and also taken a valuable step

to promote the establishment and development of robust legal metrological infrastructure for food safety, agriculture product in the developing economies.

Meanwhile, a successful training course on moisture meters was conducted. On one hand, the participants acquired further knowledge on the measurement principle of moisture meters. More importantly, they obtained skills on the key steps in the practical operations and consequently improved their hands – on experience. The participants were satisfied this training arrangement.

I am really pleased to have these fruitful outcomes from the workshop and short term training course. And again the APEC Secretariat's generosity in contributing to the development in legal metrology among the APLMF member economies is highly appreciated.

Oct. 15, 2009

Pu Changcheng
APLMF President

Summary Report

According to the increasing international trade of agricultural products across borders within the Asia – Pacific region, people have more concern about the quality and safety of foods including agricultural products. In order to respond such concerns as well as to remove unnecessary technical barriers to trade (TBT), establishment of a robust metrological infrastructure underpinning the quality and safety of such products becomes a critical issue for the APLMF and APEC economies. However, authorities and manufacturers in the economies exporting agricultural products are still facing difficulty in constructing a reliable infrastructure including measurement standards for food quality and food safety.

With an aim to facilitate these requirements in the region, two APEC/APLMF workshops on metrology of agricultural products and food safety were held in February 2007 in Chiang Mai, Thailand, and June 2008 in Hangzhou, P. R. China. The workshop in 2008 also included a session for product safety other than foods.

The present workshop was a follow – up workshop of these workshops and organized with a unique design, in which a workshop on food quality / safety and a technical training on rice moisture measurement were held jointly within three days. This form is a tentative solution in reply to two different requirements from the region, i. e. (1) a need for open and free discussion on food safety / quality between the experts including those outside from legal metrology, and (2) a traditional training program on rice moisture measurement that is one of important fields in legal metrology requested from the APLMF members.

This workshop titled as "Workshop on Metrology in Food Safety and Agricultural Products" was held from 23 – 25 September, 2009 at the Rex Hotel in Ho Chi Minh City, Viet Nam organized by APLMF and APEC. This workshop was also supported by (1) Directorate for Standards & Quality (STAMEQ) in Viet Nam, and (2) Kett Electric Laboratory Co. Ltd. in Japan.

A total of 25 participants including the 16 speakers and one trainer attended the workshop from the following 11 economies: P. R. China (2), Hong Kong, China (1), Indonesia (3), Japan (3), Malaysia (1), Mexico (2), New Zealand (1), Papua New Guinea (2), Philippines (2), Thailand (2) and Viet Nam (1), in which the number of participants indicated in parentheses. In addition, two staffs attended from the APLMF secretariat and more than 10 local staffs from STAMEQ and QUATEST3 (Center for Quality Assurance and Testing 3 in Ho Chi Min City) to support the workshop. Some of the travel fund of speakers and expenses by the host economy were supported by the APEC TILF (Trade and Investment Liberalization and Facilitation) fund (CTI – 12/2008T).

On Wednesday 23rd, the workshop started off with the opening ceremony, where

Mr. Tran Van Vinh (Deputy Director General, STAMEQ) delivered an opening address on behalf of the host economy and Dr. Zhang Chao (APLMF secretary) delivered an address on behalf of APLMF. After the opening ceremony, the topics listed in Table 1 were presented by the speakers and discussed with all participants for one and half days.

At the end of all presentations, a summary session was arranged and it was chaired by Dr. Matsumoto and Dr. Zhang to summarize all topics and to discuss future planning for the next workshop. In the summary discussion, a lot of valuable suggestions and requests for the future were proposed. The following list provides some of the suggestions.

1. Needs for traceability system for food quality measurement including rice moisture
2. Needs for alternative standard method for rice moisture with low cost
3. Legal control on rice moisture meters for reliable verification
4. Legal control on chemical measuring instruments / analyzers
5. Uncertainty analysis for rice moisture measurements
6. Role of CRM for quality of agricultural products and food safety
7. Priority of CRM in consideration of requests from users and suppliers of CRM
8. International sharing system of CRMs including database
9. Cooperation between legal metrology and scientific metrology
10. Cooperation with food organizations (CODEX, etc.)
11. Cooperation with other regional organizations (APEC, ASEAN, APLAC, PASC, etc.)
12. Continue to provide the guide documents to be utilized in APLMF
13. Harmonization with the OIML Recommendations, ISO/IEC standards and CODEX
14. Future programs for food quality measurement. There are two extreme ways: (1) expand more on food safety/product safety, or (2) go back to original background in legal metrology (rice moisture).

In the afternoon on Thursday, the main target of the workshop was switched to the technical training on rice moisture measurement. Mr. Hiroshi Yamahira of Kett Electric Laboratory served as the trainer, and most of the participants to the workshop session continuously attended this training session. Mr. Yahamira firstly gave lectures on basic understanding of grain moisture, standard measurement method using a dry oven, principle of moisture meters, traceability and calibration of moisture meters, and practical measurement procedure.

After the lectures, all participants started practical training using real moisture meters. Twenty sets of three kinds of brand new resistance / capacitance type moisture meters were supplied with the courtesy by the Kett Laboratory. The practical training was conducted using the three different kinds of moisture meters on several test samples of rice, corn and beans. Mr. Yamahira firstly demonstrated basic handling of the instrument, on - site calibration procedure using a standard resisters/capacitors, and practical measurements by taking average of several sample. Then, all participants practiced by following the demonstration even if some participants had never used a moisture meter before. This training session was useful for

the participants to obtain a practical view on what moisture meters looks like and how it works, which is difficult to obtain only through a discussion in a simple workshop.

On Friday 25th, the entire workshop was concluded with a closing ceremony. Firstly Dr. Ngo Tat Thang (Deputy Director, International Cooperation Department, STAMEQ) delivered a closing address from the host economy. Additional closing remarks from Mr. Guo Su (APLMF secretary) followed his speech.

Besides the workshop, the APLMF and host economy provided activities to encourage further discussion and friendship among the participants. A welcome dinner and a farewell dinner were held on Wednesday and Thursday, respectively. On Friday afternoon, the host economy provided a city tour to Ho Chi Minh City. The participants visited the Reunification Palace, Handcraft Center, War Remnants Museum and Notre – Dame Cathedral.

In conclusion, as the chair of the WG coordinated this workshop, I would like to express my deepest gratitude to the hard work and dedicated support provided by the staffs of the host economy, APLMF secretariat and Kett Electric Laboratory. I also appreciate participating APLMF economies for providing informative presentations and valuable suggestions in the discussion. Besides the participants, I appreciate the support by Mrs. Marian Haire in Australia in organizing this workshop although she could not attend the workshop. The structure, timing and place of a follow – up workshop will be discussed at the APLMF forum meeting in November 2009 in Chiang Mai, Thailand.

Dr. Tsuyoshi Matsumoto
Chair, Working Group on Quality Measurements
of Agricultural Products

APEC
Asia-Pacific
Economic Cooperation

APLMF
Asia-Pacific
Legal Metrology Forum

APEC/APLMF Seminars and Training Courses in Legal Metrology
(CTI-12/2008T)

Workshop on Metrology in Food Safety, Agricultural Products

Sept. 23-25 2009

at the Rex Hotel in Ho Chi Minh City, Viet Nam

Program

1. Organizers:

1. Asia-Pacific Economic Cooperation (APEC)
2. Asia-Pacific Legal Metrology Forum (APLMF)

2. Supporting Organizations:

1. Directorate for Standards & Quality (STAMEQ) Viet Nam
2. Kett Elec. Co, Japan

3. Objective:

These days, huge amounts of products are traded across borders within the Asia-Pacific region. Increasingly, more people have become anxious about the quality and safety of such internationally traded products and are interested in developing a reliable system to evaluate the quality and safety of such products.

According to the requirement to realize international trade without technical barriers to trade (TBT), developing economies producing agricultural products for export are requested to achieve reliable technical infrastructure including measurement standards for both product quality and food safety in order to obtain international competitiveness. A robust metrological infrastructure plays a critical role in the removal of technical barriers to trade by underpinning the quality and safety of export products.

In order to facilitate these objectives, two APEC/APLMF workshops on metrology of agricultural products and food safety were held in 2007 in Thailand and 2008 in P. R. China respectively. At the second workshop in 2008, the range of topics was extended to cover product safety other than foods, and Small Working Groups (SWGs) meetings to draft APLMF guideline documents were also held jointly. This proposed workshop is a follow-up meeting of the two workshops and aims to bring together experts from APEC/APLMF member economies to build

on the outputs from these workshops by:

a) Presenting the member economies an opportunity to learn about current situation of measurement standard and traceability system for quality measurements of agricultural products,

b) Presenting an opportunity reviewing present metrological infrastructure within the member economies and identifying practical needs in the future that is executable for APLMF. This activity may include the fields on quality of agricultural products and food safety.

4. Agenda

The program will include one-day training course on moisture meters and one and a half day workshop.

4.1. Workshop:

1 Legal metrology infrastructure for quality measurements of agricultural products

1.1 Primary method, measurement standard and traceability system for grain moisture, protein, fat and saccharimetry, etc.

1.2 Measurements of special products such as starch, cane, coffee, milk, wine, water, etc.

1.3 Legal control of measuring instruments

1.4 Needs for training and/or intercomparisons

1.5 Issues on packaging and labeling

1.6 The role of national legal metrology authorities

1.7 …

2 Measurement method and CRMs underpinning food safety

2.1 Measurement standards and traceability for CRMs

2.2 Legal control on CRMs

2.3 Need for new CRMs

2.4 New measurement method

2.5 …

4.2. Training Course on moisture meters will cover the following topics:

1. Infrastructure elements supporting rice moisture measurements
2. Other Moisture Measurements
3. "Grain and Oilseeds" and "Protein Measuring Instruments for Cereal Grain and Oil Seeds"
4. …

5. Program（Venue：Rex Hotel，Ho Chi Minh City）

Day 1（September 23，Wed）	08：30-09：30	*Registration*
	Opening ceremony	
	09：30-10：00	Welcome Address from the Host
		Welcome Address from APLMF
		Take a group photo
	10：00-10：30	*Coffee break*
	Workshop：Session 1. Legal Metrology Infrastructure for Quality Measurements of Agricultural Products（Chair：Dr. Matsumoto，Co-Chair：Dr. Osman Zakaria）	
	10：30-10：50	**Traceability，uncertainty analysis and current situation of OIML R59 on rice moisture meters**（Tsuyoshi Matsumoto，Japan）
	10：50-11：10	**Techniques of Measuring Grain Moisture Content in China and its Development Trend**（Zhang Weiwei，China）
	11：10-11：30	**Rice Moisture Meters**（Sakchai Hasamin，Thailand）
	11：30-11：50	**Grain moisture measurements in Mexico**（Enrique Martines-Lopez，Mexico）
	12：00-13：30	*Lunch break*
	Workshop：Session 1.（Continue）	
	13：30-13：50	**The traceability system of rice moisture meter in Indonesia**（Sri Astuti，Indonesia）
	13：50-14：10	**Checking the Calibration of Rice Moisture Meters**（Warachai Triarun，Thailand）
	14：10-14：30	Local speaker
	14：30-15：00	*Coffee break*
	Workshop：Session 2. Measurement method and CRMs underpinning food safety（Chair：Dr. Laly Samuel，Co-Chair：Dr. Wong YC）	
	15：00-15：20	**Preparation of reference material of melamine in milk powder**（Wong YC，Hong Kong，China）
	15：20-15：40	**Assurance of Measurement of Foods and Agricultural products in China by Chemical Metrology**（ZHANG Qinghe，China）

Day 1 **(September 23, Wed)**	15：40-16：00	**Overview of the Legal Metrology System in Agricultural Products, Food Safety and Product Safety in the Philippines** (Marilyn C. Fos, Philippines)
	16：00-16：20	**Development of National Infrastructure to Support legal Control of Food safety, Agricultural products and Product safety in Malaysia** (Osman Zakaria, Malaysia)
	16：20-16：40	**Agriculture and food testing in Papua New Guinea** (Peter Corbett, Papua New Guinea)
	16：40-17：00	**Metrology, Standardization, Testing and Quality Management as tools in Food Safety: Status at the Food Development Center of the National Food Authority, Department of Agriculture** (Amelia W. Tejada, Philippines)
	19：00-21：00	*Welcome dinner*
Day 2 **(September 24, Thur)**	**Workshop: Session 2.** (Continue)	
	09：20-09：40	**A collaborative approach to establish traceability in chemical measurement and food safety in New Zealand** (Laly Samuel, New Zealand)
	09：40-10：00	**Measurement Standards in Agriculture Facilities** (Edna Egu, Papua New Ginuea)
	10：00-10：20	Local Speaker
	10：20-10：40	**National Metrology Infrastructure for Food Safety in Mexico** (Norma Gonzalez-Rojano, Mexico)
	10：40-11：10	*Coffee break*
	Workshop: Session 3. Summary and Discussion (Dr. Tsuyoshi Matsumoto, Dr. Zhang Chao)	
	11：00-12：30	Presentations and discussions
	12：30-14：00	*Lunch break*
	Training: Infrastructure elements supporting rice moisture measurements (Trainer: Mr. Hiroshi Yamahira, Japan)	
	14：00-15：30	Practical training
	15：30-16：00	*Coffee break*
	16：00-17：00	Practical training
	19：00-21：00	*Farewell Dinner by APLMF*

Day 3 (September 25, Fri)	**Training: Other Moisture Measurements**	
	09：00-10：30	Practical training
	10：30-11：00	*Coffee break*
	Closing ceremony	
	11：15-11：45	Closing Address from the Host
		Closing Address from APLMF
	12：00-13：30	*Lunch break*

6. Registration

6.1. Categories of participants

The participants to the present workshop are grouped into the four categorizes below.

1. **Organizers of the workshop** (APEC Experts): Organizers who will cooperatively lead and organize the entire workshop.
2. **Primary chairpersons of workshop** (APEC Experts): Primary chairpersons of **topics** in the **workshop** (4.1). They will actively lead and organize discussions in the workshop. They are required to submit **a summary report** of the discussion after the workshop.
3. **Trainers of the moisture meters** (APEC Experts): They will lead the training program on moisture meters (4.2).
4. **Speakers** (APEC active participants): Active participants who provide a presentation on their knowledge or situation in their economy to support the workshop or training program.

6.2. How to Make a Registration

If you wish to participate in the workshop and/or training course, please complete the attached **registration form** by indicating the **category** you wish to participate and send the form to the **APLMF secretariat** to the contact address shown on the form. The **deadline** of registration is Aug. 23, 2009.

If you wish to provide **a presentation** in the workshop, the registration form must be accompanied with an **abstract** within one page (A4) outlining:

1. How your metrological infrastructure / technique supports the topics you have an interest in the workshop,
2. Issues and problems encountered within national metrological infrastructures in your economies, and
3. Possible solutions that could be implemented within the region.

It is recommended if the abstract also highlight how your presentation builds on the 2nd Workshop held in 2008 in PR China (see "Handbook on Metrology of Agricultural Products and Foods" on the APLMF website).

6. 3. Selection of Chairpersons and Speakers

The chairpersons and speakers will be selected by the APLMF secretariat in cooperation with the relevant APLMF WGs on Quality Measurements of Agricultural Products, and Training Co-ordination. The selected chairpersons and speakers will be informed from the secretariat before the workshop.

7. Travel Support:

- **APEC travel support (1)**, comprising a roundtrip economy-class airfare and per diem including accommodation, is available for the **APEC Experts** in the categories **1, 2 and 3** of the chapter 6. 1.
- **APEC travel support (2)**, comprising a roundtrip economy-class airfare and per diem including accommodation, is available for the **APEC active participants** in the categories 4 of the chapter 6. 1. The eligible participants must belong to one of the economies; PR China, Chile, Indonesia, Malaysia, Mexico, Papua New Guinea, Philippines, Peru, the Russian Federation, Viet Nam and Thailand.
- **APLMF travel support** is available for the participants from one of the **non-APEC economies** but belong to one of the **APLMF full member economies**.

APEC and APLMF travel support will cover **one** speaker **per economy** unless otherwise permitted by the APLMF secretariat in order to organize the workshop. Participant (s) approved for travel support will be finally decided by the APEC/APLMF Secretariat. We welcome more self-funded speakers will join the workshop.

8. Visa Assistance:

If you need a visa to enter Viet Nam, please complete the bottom section of the Registration Form under "Visa information". This information will be forwarded to the host by the APLMF Secretariat. On receipt, the host economy will send an official letter of invitation to support visa applications.

9. Venue and Accommodation:

Rex Hotel

Add: 141 Nguyen Hue Blvd, District 1, HCM city, Viet Nam

Tel: +84 8 38292185, Fax: +84 8 38296536

http://www. rexhotelvietnam. com

Dao Duy Truc Lam (Ms.)

Sales Manager-Corporate

Email: lamddt. rex@ sgtourist. com. vn

Cell phone: 0938 087 108

Tel：(848) 3829 9084
Fax (848) 3824 8962

If you wish to reserve a room at the venue, please complete the Hotel Reservation Form and send it to the Rex hotel by lamddt. rex@ sgtourist. com. vn or nguyentt. rex@ sgtourist. com. vn and the host in Viet Nam by htqt@ tcvn. gov. vn or bandoluong@ tcvn. gov. vn (1 month before).

10. Access Information：

Ideally located in the center heart of Ho Chi Minh City, next door to the People Committee House, within steps of the famous Ben Thanh Market and historical Opera. The guests can take any kinds of transportation to reach the hotel.

Transport：

The Rex in Ho Chi Minh City is only 5 kilometers from the Saigon railway. The Tan Son Nhat Airport is also 7 kilometers far away from the Rex in Ho Chi Minh City.
The taxi fare from the airport to the Rex Hotel Hotel is less than $5 per trip (70 000 VND-100 000 VND).

Weather：

Temperature between 20℃ to 33℃ and Relative Humidity between 70% to 90%.
There are 2 main seasons in HCM city, rainy and dry seasons. In September, it's monsoon season, the weather is warm and humid.

Currency：

USD 1 is about 18 000 VND.

11. Submission of documents and slides：

If you hope your documents and/or slides would be distributed at the workshop in a hard copy, please be advised to send your documents (soft copy) to the secretariat < APLMF@ aqsiq. gov. cn > by **August 28**.

12. Contacts for the APEC/APLMF Meetings：

1. APLMF Secretariat (registration and travel support)
Dr. ZHANG Chao & Mr. GUO Su
Department of Metrology, General Administration of Quality Supervision, Inspection and Quarantine of the People's Republic of China (AQSIQ)
No. 9, Madiandonglu, Haidian District, Beijing 100088, P. R. China
Tel：+86-10-8226-0335, Fax：+86-10-8226-0131, E-mail：sec@ aplmf. org

2. Host in Viet Nam (visa assistance, accommodation and venue)

Ms. Nguyen Thi Minh Nguyet
International Cooperation Department
Directorate for Standards, Metrology and Quality (STAMEQ)
Tel: +84 4 37911630; Fax: +84 4 37911605; Email: htqt@ tcvn. gov. vn
Address: No. 8 Hoang Quoc Viet Street, Cau Giay District, Ha Noi, Viet Nam

Or

Ms. Tran Thi Thuy Ha
Metrology Department
Directorate for Standards, Metrology and Quality (STAMEQ)
Tel: +84 4 37911632; Fax: +84 4 37911631; Email: bandoluong@ tcvn. gov. vn
Address: No. 8 Hoang Quoc Viet Street, Cau Giay District, Ha Noi, Viet Nam

Participants List
APEC/APLMF Seminar and Training Courses in
Legal Metrology (CTI-12/2008T)
Workshop on Metrology in Food Safety,
Agricultural Products

No.	Category	Economy	Name	Organization
1	APLMF	China, PR	Dr. ZHANG Chao	APLMF Secretary, Department of Metrology, AQSIQ
2	APLMF	China, PR	Mr. GUO Su	APLMF Secretary, Department of Metrology, AQSIQ
3	Trainer	Japan	Mr. Hiroshi Yamahira	International Marketing, Kett Electric Laboratory
4	Participant	Malaysia	Mr. Osman ZAKARIA	National Metrology Laboratory, SIRIM Berhad
5	Participant	New Zealand	Dr. Laly ASAMUEL	Measurement Standards Laboratory of New Zealand, Industrial Research
6	Participant	Indonesia	Ms. Sri Astuti	Metrological Training Centre
7	Participant	Mexico	Dr. Norma Gonzalez-Rojano	Centro Nacional de Metrologia
8	Participant	Mexico	Mr. Enrique Martines-Lopez	Centro Nacional de Metrologia (CENAM)
9	Participant	Philippine	Dr. Amelia Tejada	Food Development Center, NFA
10	Participant	Philippine	Ms. MARILYN Fos	National Metrology Laboratory-Industrial Technology Development Institute
11	Participant	PNG	Mr. Peter Corbett	Papua New Guinea National Agriculture Research Institute (NARI)

12	Participant	PNG	Ms. Edna Egu	National Institute of Standards & Industrial Technology
13	Participant	China, PR	Dr. ZHANG Qinghe	National Institute of Metrology, China
14	Participant	China, PR	Ms. ZHANG Weiwei	Helongjiang Provincial Institute of Measurement and Verification
15	Participant	Thailand	Mr. Warachai Triarun	Central Bureau of Weights and Measures
16	Participant	Thailand	Mr. Sakchai Hasamin	Central Bureau of Weights and Measures
17	Participant	Japan	Mr. Tsuyoshi Matsumoto	National Metrology Institute of Japan (NMIJ)
18	Participant	Hong Kong, China	Dr. Yiu Chung WONG	Hong Kong Government Laboratory
19	Participant	Japan	Mr. Ryoichi Ishii	JT Engineering Inc
20	Local participant	Viet Nam	Ms. Pham Thi Kim Ngoc	Quality Assurance and Testing Centre (QUATEST 3), Viet Nam
21	Local participant	Viet Nam	Mr. Huynh Trong Nghia	Quality Assurance and Testing Centre (QUATEST 3), Viet Nam
22	Host	Viet Nam	Mr. Tran Van Vinh	Directorate for Standards, Metrology and Quality (STAMEQ)
23	Host	Viet Nam	Mr. Nguyen Hung Diep	Directorate for Standards, Metrology and Quality (STAMEQ)
24	Host	Viet Nam	Mr. Tran Quy Giau	Directorate for Standards, Metrology and Quality (STAMEQ)
25	Host	Viet Nam	Dr. Ngo Tat Thang	Directorate for Standards, Metrology and Quality (STAMEQ)

AIST

Traceability, Uncertainty Analysis and Current Situation of OIML R59 on Rice Moisture Meters

Dr. Tsuyoshi Matsumoto, Dr. Hideyuki Tanaka, Mr. Issei Akamatsu*
National Metrology Institute of Japan (NMIJ)
National Institute of Advanced Industrial Science and Technology (AIST)
and
Mr. Hiroshi Yamahira
Kett Electric Laboratory, Japan

*former staff of NMIJ

AIST

CONTENTS

1. Basic understanding of rice moisture meters
2. Traceability for rice moisture measurement
3. Current situation in OIML TC17/SC1 and overview of OIML-R59
4. Activities of APLMF WG on Quality Measurements of Agricultural products
5. Evaluation of uncertainty of the drying method for rice moisture (provided by Dr. H. Tanaka)

AIST

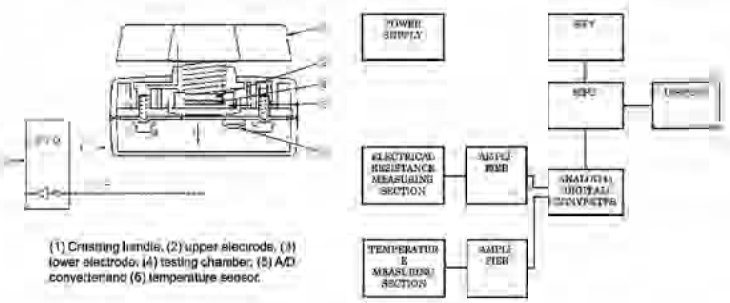
1. Basic understanding of rice moisture meters

- **Rice (grain) moisture meters** form an important category in agricultural measurements that is closely related to fare trade and health / welfare of people.
- Moisture is an essential property to determine the **value of grains** as well as stability in long-time storage.
- Because of the importance, this category of instruments is controlled under **legal metrology** in some economies. In particular, economies exporting grains have much concern on **grain moisture measurements**.
- Typical measurement methods for grain moisture are:
  1. Drying oven method (direct & absolute)
  2. Electric resistance method (indirect)
  3. Electric capacitance method (indirect)
  4. Near infrared method (indirect)

AIST

1. Basic understanding of rice moisture meters

Electric Resistance Grain Moisture Meter

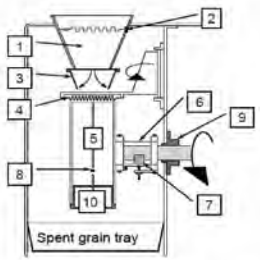


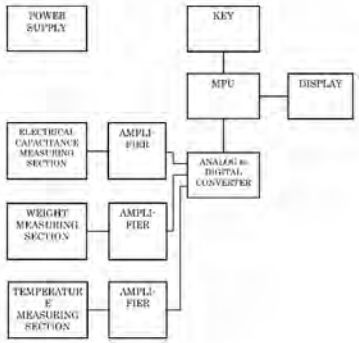
(1) Crossing handle, (2) upper electrode, (3) lower electrode, (4) testing chamber, (5) A/D converter and (6) temperature sensor.

Small sample size & handheld
About 70% of market in Asia

1. Basic understanding of rice moisture meters

Electric Capacitance Grain Moisture Meter

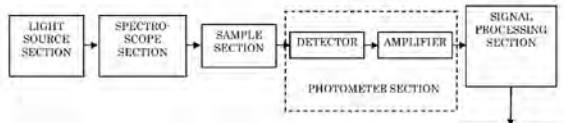


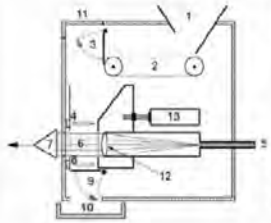


(1) Hopper, (2) sensor for grain amount, (3) (4) loading mechanism, (5) test cell, (6) flexors, (7) weight sensor, (8) temperature sensor, (9) axle supports and (10) A/D converter.

1. Basic understanding of rice moisture meters

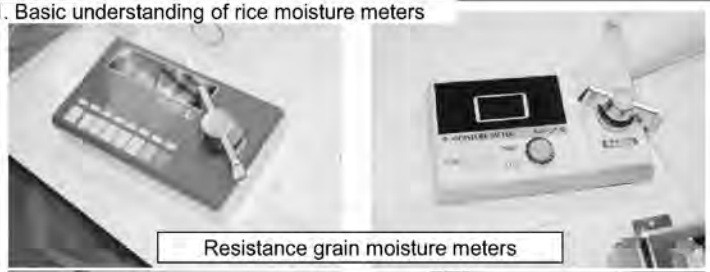
Near Infrared Grain Moisture Meter





(1) Hopper, (2) (3) loading mechanism, (5) scanning monochromator, (6) sample chamber, (7) detector, (9) (10) drain mechanism and (12) lens.

1. Basic understanding of rice moisture meters



Resistance grain moisture meters

Capacitance grain moisture meters

1. Basic understanding of rice moisture meters

Rice inspection at the Yamagata Food Office (Japan)

AIST

2. Traceability for Rice Moisture Measurement

Needs for Traceability in Asian Countries

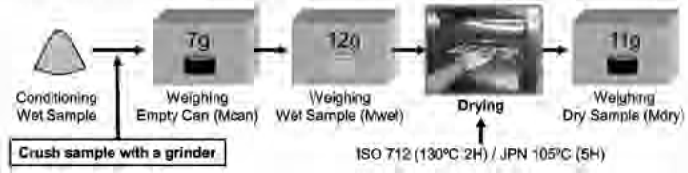
1. A lot of **rice-producing** and **rice-exporting** countries.
2. International and domestic **fair trade** of rice is requested.
3. No **common calibration system** in Asian countries.
4. Few countries have their **own traceability system.**
5. Lack of **practical traceability system** applicable to moisture meters in service.

Requirements for Practical System

1. Harmonization with existing **international standards**: ISO-712, ISO-7700 and OIML R59
2. Applicable to **popular moisture meters**
3. Low cost
4. Easy to adopt / operate

2 Traceability for Rice Moisture

Calibration of Moisture Meters with a Dry Oven Method



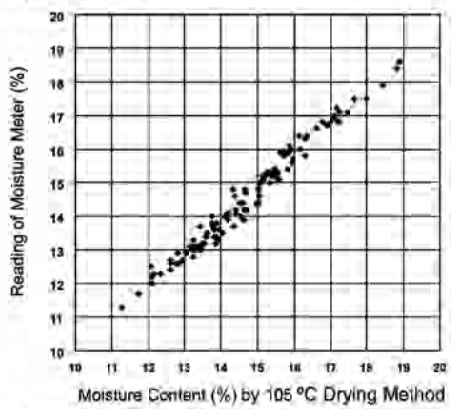
Moisture Content = (Mwet - Mdry) / (Mwet - Mcan)
= (12g - 11g) / (12g - 7g) = 0.2

Absolute & Direct Measurement

Indirect Measurement (electric, capacitance & infrared meters)

Standard (20%) ⟷ In-service (19%)

2 Traceability for Rice Moisture

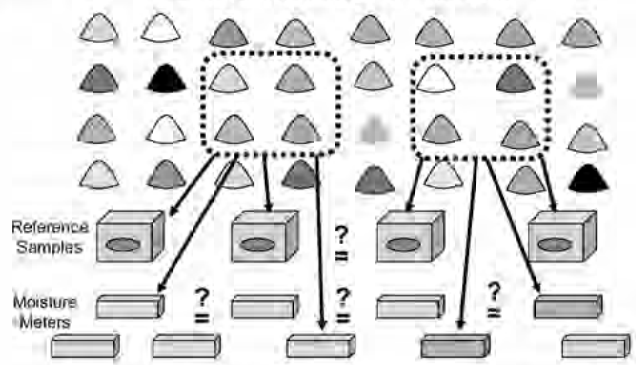


An Example of Calibration Curve of a Resistance Moisture Meter
(105 °C drying method, 2001FY brown rice, 113 samples / 30 varieties)

2 Traceability for Rice Moisture

Rice Moisture Measurement without Traceability

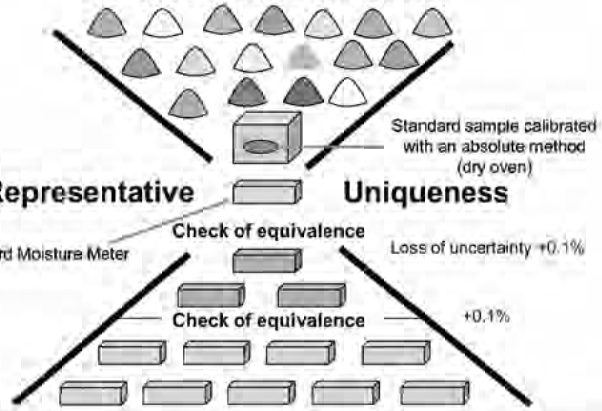
Several Hundreds of Products



AIST

2. Traceability for Rice Moisture

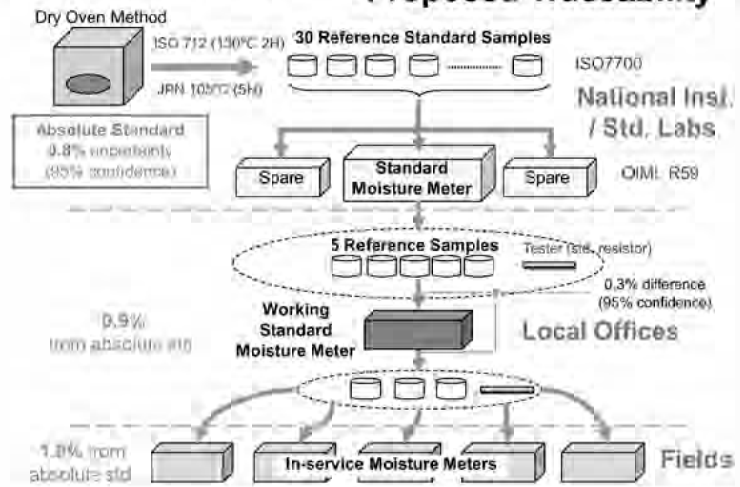
Proposed Traceability



AIST

2. Traceability for Rice Moisture

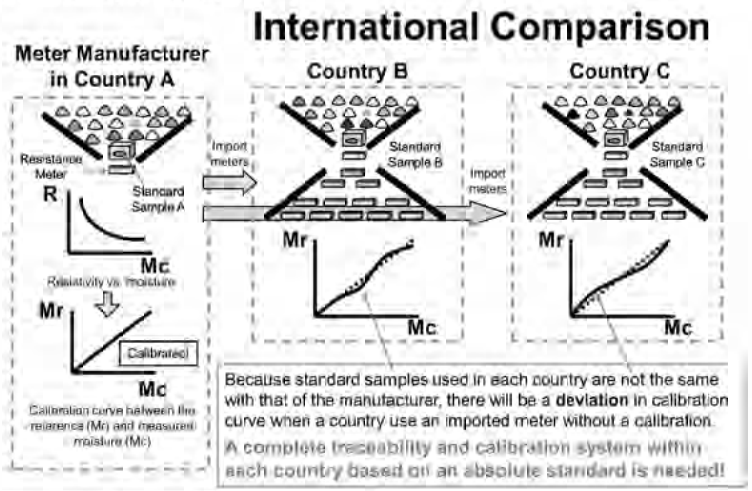
Proposed Traceability



AIST

2. Traceability for Rice Moisture

International Comparison



Because standard samples used in each country are not the same with that of the manufacturer, there will be a **deviation** in calibration curve when a country use an imported meter without a calibration.

A complete traceability and calibration system within each country based on an absolute standard is needed!

AIST

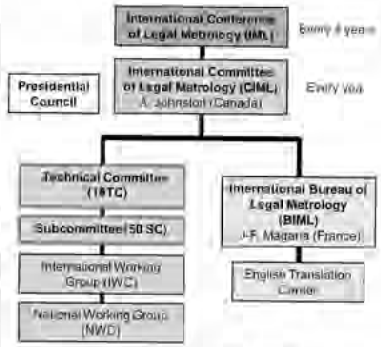
3. Current Situation in OIML TC17/SC1 (humidity) and Overview of OIML R59 (moisture meters for cereal grains and oilseeds)

3. Current Situation in OIML

OIML

(International Organization of Legal Metrology)

- Established in **1955** following the conclusion of OIML Convention.
- **58** member states and **56** corresponding member states (January 2009).
- A **standard setting organization** under the agreement on **WTO/TBT** in together with ISO and IEC.
- Dedicated to harmonize **national metrology regulations** of member states to remove **technical barriers to trade**.
- To help developing countries to set up sound **national legal metrology systems**.



OIML Organization Chart

3. Current Situation in OIML

OIML Recommendations (R)

- **Model technical regulations** of measuring instruments consisting of: (1) technical requirements, (2) test procedures for conformity to the requirements, and (3) test report format.
- Developed by **TC/SC**, approved by CIML
- Member states are **morally recommended** to implement in their national systems.
- **More than 100** recommendations (R7-R142) have been published and be downloaded freely from the OIML website.

3. Current Situation in OIML

Outline of OIML R59 (1984)

"Moisture meters for cereal grains and oilseeds"
Developed by TC17/SC1

1. **Scope:** measuring moisture (or volatile) contents of grains and oilseeds
2. **Field of Application**: applicable only to static samples in categories A & B
3. **General (terminology)**: moisture content, conversion tables, etc.
4. **General technical requirements**: construction, indication device, etc
5. **Moisture meters of category A** (automatic)
6. **Moisture meters of category B** (non-automatic)
7. **Inscription**: markings, identifications and supervision
8. **Maximum permissible errors**: in type approval, initial verification and in-service
9. **Seals for protection and guarantee, table certification marks**
10. **Sanction of the controls – Stamping**
11. **Provisions to assure fairness of the measurement operations**
12. **Appendix I**: Practical reference methods for the verification
13. **Appendix II:** Routine reference method for the verification
14. **Appendix III:** Metrological controls

Close relationships with:
ISO 712 (Cereals and cereal products - Determination of moisture content - Routine reference method) and
ISO 7700 (Check of the calibration of moisture meters - Part 1: Moisture meters for cereals).

3. Current Situation in OIML

History of Revision of R59

2001: TC17/SC1 meeting was held in **PTB, Berlin** to discuss major revisions to the existing OIML R59 (1984). The committee agreed that the U.S. National Institute of Standards and Technology (**NIST**) would prepare the first committee draft (**1CD**)

2002: The U.S. completed a first Committee Draft (1CD) of R59. TC17/SC1 secretariat in PR China circulated the **1CD** to the member countries for comment

2003: The U.S. developed the **2CD** based on the comments on 1CD. The secretariat circulated **2CD** to the member countries for comment.

2003: TC17/SC1 meeting was held in **Beijing** to review the comments and revisions to 2CD. **Two concerns** expressed by Japan about temperature requirements and sample size for resistance meters.

2004: The U.S. drafted the **3CD** of OIML R 59 based on the comments at the meeting in 2003 The secretariat in PR China circulated the **3CD** and then, **4CD** for comments

September 2007: TC17/SC1 meeting was held at **NIST in U.S.** with other OIML meetings. After this meeting, the co-secretariat in NIST reviewed the comments to 4CD and developed **5CD**.

March 2009: 5CD was circulated from the co-secretariat in NIST to the member economies Comments to the **5CD** were submitted from the member countries in May, 2009

2010: Draft Recommendation?

AIST

4. Activities of **APLMF Working Group on Quality Measurements of Agricultural Products**

1. This WG was set up in **2001** as **'WG on Rice Moisture Meters'** with a chairperson, Mr. Issei Akamatsu (NMIJ).
2. In 2001-2005, the WG conducted **training courses** on rice moisture meters with support by APLMF/APEC, Japanese Gov. and Kett Lab. These courses contributed **capacity building** in verification and proposed a **traceability system**.

3. In 2005, the WG was renamed as '**WG on Measurement of Moisture Content of Agricultural Commodities**' and the chairperson was taken over by Mr. Hiroshi Kitano (NMIJ).
4. In 2007, the WG was renamed as present and the chairperson was taken over by **Matsumoto** (me). Two workshops were conducted with support by APLMF/APEC and WG on Training Coordination (Australia).

AIST

4. Activities of APLMF WG

APLMF Training Courses on Rice Moisture Meters and Quality of Agricultural Products

Course Title	Date (d/m/y)	Place (Host)	Trainers	Trainees
Study Tour for Rice Moisture Measurement*	30/06-5/10/2001	Japan including NMIJ (NMIJ)	I. Akamatsu (NMIJ) and others (JPN)	9 from 7 econ.
Training on Calibration of Rice Moisture Meters*	19-30/08/2002	Khon Kaen, Thailand (CBWM)	I. Akamatsu, H. Tanaka (NMIJ), T. Watanabe, N.Yoshida (Kett Co.)	23 from 7 econ.
Training on Calibration of Rice Moisture Meters*	30/08-10/09/2004	Bien-hoa, Viet Nam (STAMEQ)	I. Akamatsu (NMIJ), T. Watanabe, N. Yoshida, T. Suzuki (Kett Co.)	About 20
Training on Calibration of Rice Moisture Meters*	15-29/11/2004	Chiang Mai, Thailand (CBWM)	I. Akamatsu, H. Tanaka (NMIJ), T. Watanabe, N.Yoshida (Kett Co.)	About 23 from ASEAN
Training on Calibration of Rice Moisture Meters*	15-26/08/2005	Manila, Philippines (ITDI)	I. Akamatsu, H. Tanaka (NMIJ), T. Watanabe, N.Yoshida (Kett Co.)	From ASEAN
Workshop on Metrology of Agricultural Products and Foods	7-9/02/2007	Chiang Mai, Thailand (CBWM)	24 from 11 econ.	About 80 including speakers
Workshop on Metrology in Food Safety, Agricultural Products and Product Safety	4-6/06/2008	Hangzhou, PR China (AQSIQ)	24 from 14 econ.	About 70 including speakers

* These courses were supported by Japanese fund. They were not organized by APLMF secretariat but conducted in cooperation with APLMF.

AIST

4. Activities of APLMF WG

Summary of the Workshop on Metrology in Food Safety, Agricultural Products and Product Safety (2008)

Metrological Infrastructure
1. Need for sound metrological infrastructure.
2. Important roles of national authorities including NMI.
3. Need for primary methods for measurement.
4. Need for a traceability system with uncertainty.
5. Consideration of the difference between scientific metrology and legal metrology.
6. Concern on other foods (coffee, tea, starch, milk, etc.)

CRMs (Certified Reference Materials)
7. Collaboration between economies for developing CRMs.
8. Need for regionally or internationally certified CRMs.
9. Need for regional or international CRM database.
10. Requirement to CRMs from legal actions.
11. Need for organic CRMs for biological measurement / analysis.
12. Need for CRMs for GMO (Genetically Modified Organisms).
13. Need for CRMs for product safety.

Food Safety
14. Need for primary methods in biological measurement.
15. Evaluation of uncertainty in chemical measurement.
16. Issues on containers in food preparation.
17. Legal requirement to biological measurements.
18. Issues on PT (Proficiency Test) scheme.
19. Consider the difference between physical / biological measurements.

Product Safety
20. Legal requirements to the product safety.
21. International trend to control hazardous materials.
22. Need for technical support to product safety assessments.
23. Share test results on product safety internationally.
24. Collaboration between product safety and food safety.
25. Need for infrastructure for product safety assessment.

Others
26. Need for training and knowledge base.
27. Issues on labeling on products/foods.
28. Issues on accreditation schemes.

Future work of APLMF and other regional organizations
29. Assist economies to develop infrastructure in metrology.
30. Enhance cooperation between metrology and other fields.
31. Enhance collaboration between APLMF and APMP by paying attention to the difference in targets and interests.
32. Complete the three guide documents by the SWGs.
33. Carry out a survey to prioritize the needs for CRMs.
34. Organize or participate in international intercomparisons in chemical measurements.
35. Organize training programs or intercomparison on rice moisture meters.
36. Organize regional project on starch content in cassava.
37. Investigate the situation on product safety.
38. Consider guideline on the information on the website.

AIST

4. Activities of APLMF WG

Objectives of the Present Workshop

Workshops in 2007 & 2008.
Target field was expanded and many requests & problems were highlighted. But APLMF solely can not be a

Proposals in the APLMF Meeting in 2008 (Sydney)
1. Go back to our original field and select the executable targets.
2. Continue the training course on rice moisture measurements.

Objectives of the Present Workshop
1. Provide a training course on rice moisture to focus on the need within the region.
2. Examining the outputs from the previous workshops and determining how APLMF can provide support within the region for any issue identified.

Evaluation of Uncertainty of the Drying Method

Hideyuki TANAKA
Metrology Institute of Japan
National Institute of Advanced Industrial Science and Technology
E-mail: tanaka-hideyuki@aist.go.jp

Measurement procedure of the drying method

- 1: Prepare the samples of adjusted moisture.
- 2: Grind the samples in a grinder.
- 3: Measure the constant weight of the weighing can to be used.

  Repeat once.
- 4: Transfer the crushed samples into the weighing can and weigh them.

  Repeat once.

  Samples…Two samples are taken from the same lot.
- 4: Dry the samples.
- 5: Weigh the samples after drying.

  Repeat once.
- 6: Calculate the moisture content.

The source of uncertainty of the drying method

- Uncertainty caused by the **distribution of temperature** in the dryer.
- The uncertainty of this distribution is evaluated from the deviation between the samples' moisture content which are measured when the same samples are placed at **different regions**.

The source of uncertainty of the drying method

- Uncertainties caused by the **repeatability** and the **deviation** between samples.
- This is calculated simultaneously from the above experiment.

AIST

The source of uncertainty of the drying method

- Uncertainty caused by the reproducibility of the **grinders**.
- Prepare the grinders, which is the **same type**. And evaluate the **deviation** between grinders.

AIST

The source of uncertainty of the drying method

- Uncertainty in **mass measurement**.
  - Uncertainty of the mass of a weighing can.
  - Uncertainty of the mass of a weighing can and the samples.
  - Uncertainty in the calibration of a weighing machine.

AIST

Model equation of the drying method

- Model equation

$$M = \frac{m_0 - m_1}{m_0 - m_c} \times 100 + e_T + e_R + e_G$$

M: The moisture content in the sample.
m_0: The mass of the sample before drying + the mass of the weighing can.
m_1: The mass of the sample after drying + the mass of the weighing can.
m_c: The mass of the weighing can.
e_T: Moisture dispersion caused by the distribution of temperature in the dryer.
e_R: Moisture dispersion caused by the repeatability and the deviation between the samples.
e_G: Moisture dispersion caused by the reproducibility of the grinders.

AIST

Model equation of the drying method

- The law of propagation of uncertainty is applied in the above equation

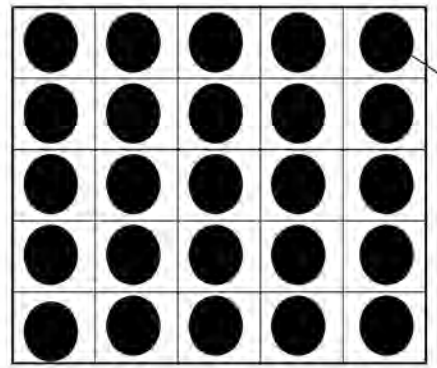
$$\frac{\partial M}{\partial m_0} = \frac{100(m_1 - m_c)}{(m_0 - m_c)^2} \qquad \frac{\partial M}{\partial m_1} = -\frac{100}{m_0 - m_c} \qquad \frac{\partial M}{\partial m_c} = \frac{100(m_0 - m_1)}{(m_0 - m_c)^2}$$

$$u_c^2(M) = \left[\frac{100(m_1 - m_c)}{(m_0 - m_c)^2}\right]^2 u_{m_0}^2 + \left[-\frac{100}{m_0 - m_c}\right]^2 u_{m_1}^2 + \left[\frac{100(m_0 - m_1)}{(m_0 - m_c)^2}\right]^2 u_{m_c}^2 + u_T^2 + u_R^2 + u_G^2$$

Evaluation of several standard uncertainty

- Uncertainty caused by the **distribution of temperature** in the dryer.
- The interior of the dryer is divided into **25 regions**. The moisture content of 25 samples that are sampled from the same lot and placed in several regions are measured. This measurement is **repeated twice**.

Uncertainty caused by the distribution of temperature in the dryer.



Weighing can with sample which derived from same lot.

The interior of the dryer

Uncertainty caused by the **distribution** of **temperature** in the dryer.

	n_1	n_2
P_1	13.97	13.98
P_2	13.89	13.98
P_3	13.96	13.98
P_4	13.88	13.92
P_5	13.99	13.95
P_6	13.97	13.96
P_7	13.94	13.94
P_8	13.97	13.96
P_9	13.96	13.99
P_{10}	13.91	13.97
P_{11}	13.90	13.92
P_{12}	13.98	13.98
P_{13}	13.95	13.98
P_{14}	13.96	13.93
P_{15}	13.96	13.99
P_{16}	13.97	13.96
P_{17}	13.88	13.94
P_{18}	13.97	13.97
P_{19}	13.94	13.96
P_{20}	13.90	13.96
P_{21}	13.96	13.93
P_{22}	13.98	13.97
P_{23}	13.88	13.92
P_{24}	13.92	13.97
P_{25}	13.97	13.95

ANOVA*

Factor	Sum of square, S	Deg. free, f	Variance, V	Expectation of variance, $E(V)$
Place	0.03071200	24	0.00127967	$\sigma_e^2 + 2\sigma_P^2$
Error	0.01665000	25	0.00066600	σ_e^2
Total ST	0.04736200	49		

*ANOVA: **Analysis of variance** developed by Sir Ronald Aylmer Fisher

Uncertainty caused by the **distribution of temperature** in the dryer.

- Therefore, in actual measurement, two samples taken from the same lot are measured and the result is calculated from the mean of the values as follows:

$$u_T = \frac{0.01752}{\sqrt{2}} = 0.01239\ \%$$

(σ_P = 0.01752; 2 times repeat)

AIST

Uncertainties caused by the repeatability and the deviation **between samples**.

- The variance of the error calculated in 1) represents the combination of repeatability and the deviation between the samples and the measurement result is calculated from the mean of the values as follows:

$$u_R = \frac{0.02581}{\sqrt{2}} = 0.01825\ \%$$

(σ_B: 0.02581; 2 times repeat)

AIST

Uncertainty caused by the reproducibility of the **grinders**

Specification of the **grinder** is the one of the most influential factors to change the rice moisture in the drying method. Therefore, we recommend that specifications of the grinders are determined by several country standards.

Example: Japanese Standard

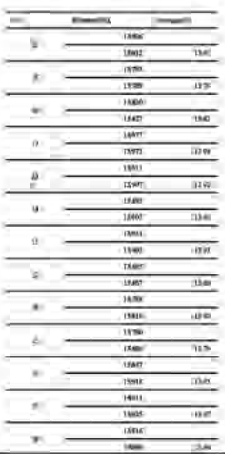
AIST

Uncertainty caused by the reproducibility of the **grinders**

Although, the specifications of the grinders are determined, there is an uncertainty caused by the **reproducibility** of the **grinders**.
The samples that are sampled from the same lot are crashed by the **13 grinders**, and the moistures are measured. This measurement is **repeated twice**.

AIST

Uncertainty caused by the reproducibility of the **grinders**



ANOVA

Factors	S	f	V	$E(V)$
Reproducibility of the grinders	0.10355988	12	0.00862999	$\sigma_e^2 + 2\sigma_B^2$
Error	0.00735825	13	0.00056602	σ_e^2
Total	0.11091813	25		

AIST

Uncertainty caused by the reproducibility of the **grinders**

The following values are taken from the ANOVA table:

$$\hat{\sigma}_G = 0.06350\%$$

In actual measurement, only one grinder is used

$$u_G = \hat{\sigma}_G = 0.06350\%$$

AIST

Uncertainty in **mass measurement**.

- Repeatability of the mass of a weighing can.
  Five weighing cans are repeatedly measured 5 times.

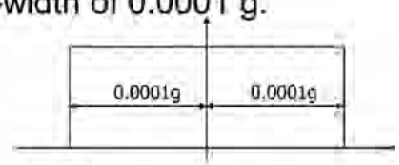
Weighing can (Number)	Mass of the weighing can				
	1	2	3	4	5
25	11.2966	11.2966	11.2966	11.2966	11.2966
26	11.0373	11.0374	11.0374	11.0373	11.0373
28	10.7781	10.7780	10.7779	10.7780	10.7780
27	11.0427	11.0427	11.0426	11.0426	11.0427
39	10.7202	10.7202	10.7201	10.7201	10.7200

AIST

Repeatability of the mass of a weighing can.

- All the measurement results include a mean value of 0.0001 g. Therefore, we assume the rectangular distribution of the possible values of mass of the weighing can with a half-width of 0.0001 g.

We assume:



$$u_{CAN} = \frac{0.0001}{\sqrt{3}} = 0.0000577\,\mathrm{g}$$

AIST

Repeatability of the mass of a weighing can and the sample

One weighing can and sample is repeatedly measured 10 times.

No.	Mass (Can+Sample) [g]
1	16.7741
2	16.7743
3	16.7741
4	16.7742
5	16.7742
6	16.7743
7	16.7743
8	16.7742
9	16.7744
10	16.7744
S.D.	0.0001080

For this result, standard deviation of the repeatability of the mass of a weighing can and the sample is 0.0001080(g). In actual measurement, the weighing can and sample is weighed one time.

$$u_{mc} = 0.0001080(\mathrm{g})$$

Uncertainty in the calibration of a weighing machine.

- From the calibration certificate of the weighing machine:

Calibration Certificate

Expanded Uncertainty
(k=2)
0.00005g

$$u_S = \frac{0.00005}{2} = 0.000025\,(\mathrm{g})$$

Uncertainty of the weighing can

- u_{mc} which represents the uncertainty of the weighing can, is evaluated using a combination of u_{CAN} and u_S.

$$u_{mc} = \sqrt{u_{CAN}^2 + u_S^2} = \sqrt{0.0000577^2 + 0.000025^2} = 0.0000629\,(\mathrm{g})$$

Uncertainty caused by the **mass of a sample** before and after drying + the mass of a weighing can

- u_{m_0} and u_{m_1} are evaluated using a combination of u_S and u_{mc}.

$$u_{m_0} = u_{m_1} = \sqrt{u_S^2 + u_{mc}^2} = \sqrt{0.000025^2 + 0.0001080^2} = 0.0001109\,(\mathrm{g})$$

Budget Sheet

Symbol	Source	Standard Uncertainty	Sensitivity Coefficient	Standard Uncertainty(%)
u_1	Uncertainty caused by the distribution of temperature in the dryer	0.01239 (%)	1	0.01239
u_2	Uncertainties caused by the repeatability and the deviation between samples	0.01825 (%)	1	0.01825
u_3	Uncertainty caused by the reproducibility of the graders	0.06350 (%)	1	0.06350
u_{m_0}	Uncertainty caused by the mass of a sample before drying + the mass of a weighing can	0.0001109 (g)	17.1715 (%/g)	0.001904
	u_S: Uncertainty in the calibration of a weighing machine	0.000025 (g)		
	u_{mc}: Uncertainty of the weighing can and sample	0.0001080 (g)		
u_{m_1}	Uncertainty caused by the mass of a sample after drying + the mass of a weighing can	0.0001109 (g)	19.9605 (%/g)	0.002214
	u_S: Uncertainty in the calibration of a weighing machine	0.000025 (g)		
	u_{mc}: Uncertainty of the weighing can and sample	0.0001080 (g)		
u_{mc}	Uncertainty of the weighing can	0.0000629 (g)	2.78894 (%/g)	0.0001754
	u_S: Uncertainty in the calibration of a weighing machine	0.000025 (g)		
	u_{CAN}: Uncertainty of the weighing can	0.0000577 (g)		
	Combined Standard Uncertainty(%)			0.06729
	Expanded Uncertainty(%) (k=2)			0.13

Measurement Results

- The mass of sample A before drying + the mass of weighing can A.....15.8234 g
- The mass of sample B before drying + the mass of weighing can B.....15.9631 g
- The mass of weighing can A.....10.8135 g
- The mass of weighing can B.....11.2915 g
- The mass of sample A after drying + the mass of weighing can A.....15.1234 g
- The mass of sample B after drying + the mass of weighing can B.....15.3112 g
- The moisture content in sample A.....13.97%
- The moisture content in sample B.....13.95%
- Mean value of the samples.....13.96%
- The moisture content in the lot of the rice is:

$$M = 13.96(\%) \pm 0.13(\%) \quad (k = 2)$$

Grain Moisture Content Measurement Methods and Traceability in China

Weiwei Zhang

Heilongjiang Institute of Measurement and Verification
P. R. China

OUTLINE

- Introduction
- Importance of Measurement
- National Standards
- Existing Methods
- Traceability of Moisture Meter
- Arising Problems
- Future Trends
- Conclusions

Introduction

- Grain moisture content (MC) has been an important quality criterion controlled by the Department of Food for a long time, both at home and abroad.
- MC plays an important role, not only in storage of grain but also in grain marketing and handling.
- Therefore, it is of great demand to determine the MC via correct and precise procedures.

Importance of Measurement

- The level of grain MC is an important factor influencing storage safety, grain color and quality.
- Measurement of grain moisture is not just a technical issue, but also associate with the vital interests of most Chinese farmers.

National Standards

- **GB/T 21305-2007**
- **Cereals and cereal products—Determination of moisture content—Reference method**
- Method: Heating (oven) methods (130± 3) ℃.
- Applies to: wheat, rice (paddy, milled), barley, millet, rye, oats, triticale, sorghum in the form of grains, milled grains, semolina or flour.
- Not applicable to: maize and pulses.
- Equivalent to: ISO712:1998 "Cereals and cereal products — Determination of moisture content — Reference method"

Existing Methods

Direct Methods

- Heating method
  - Based on mass loss through heating in a heating oven
  - **(105±2)℃ method**
    - a standard method with a single measurement time of (2~3)h
    - used as calibration for other methods
  - **(130±3)℃** electric heating method
    - most commonly used in lab

Direct Methods

- **Uncertainty**:
  U=0.4%~0.6% (k=2)
- **Advantages**:
  - Direct method
  - Traceable to a balance of 1mg resolution
  - High accuracy
  - Good reliability
- **Disadvantages**:
  - Long time (at least 3 hours)
  - High demand for operator

Direct Methods

- Others

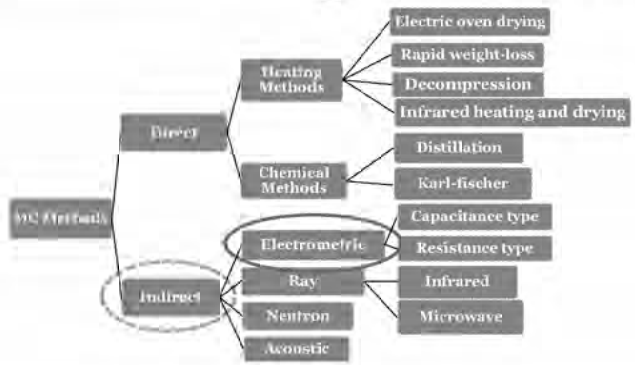
Decompression method

- Not influenced by the shape of grains
- Free of special pre-treatment
- Ease of operation
- High reliability
- Suitable for quality control

Infrared heating method

- Using infrared lamps
- High precision
- Easy to use
- Wide rang of measurement
- Time consuming (1030min)

Existing Methods



Indirect methods–Electrometric method

Resistance Method

Principle

- Determined according to the changes of conductance

Advantages

- Fast response
- Simple structure
- Easy operation

Disadvantages

- Temperature influences the MC results
- Not suitable for high MC samples and trace water

Capacitance Method

Principle

- Determined by measuring the dielectric constant of the grain

Advantages

- High reliability
- Low cost
- Easy operation and maintenance
- Online determination
- For high MC samples

Disadvantages

- Lots of influencing factor and complex data

OUTLINE

- Introduction
- Importance of Measurement
- domestic Standards
- Existing Methods
- Traceability of Moisture Meter
- Arising Problems
- Future Trends
- Conclusions

Typical Moisture Range

Typical Moisture Range of Different Grains

Name	Range of MC (%)	Primary Range of MC (%)
Paddy	9~20	12~17
Rice	10~20	12~17
Wheat	8~20	11~16
Maize	10~22	12~17
Cereal	8~20	10~15
Millet	10~18	12~15
Sorghum	8~20	10~16
Sorghum rice	12~20	12~16

Traceability of Moisture Meter

- JJG 891—1995
- Verification Regulation of Instruments for Measuring the Moisture Content of Grain.
  - Requirement of techniques
  - Conditions of verification
  - Methods for verification
  - Period of verification

Traceability of Moisture Meter

- **JJG 891–1995**
  - **Requirement of techniques**
    - **Safety requirement**
      - Resistance between source circuit and insulation > 7 MΩ
      - The instrument shall endure 50Hz, 1500V sine wave AC, 5mA, 1min between source circuit and insulation without breakdown and arc
    - **Measurement time < 5min**
    - **Error indication**

Traceability of Moisture Meter

- **JJG 891–1995**

Requirement on indication error of equipments

Grade of Moisture Meter	Primary Range of MC		Other Range of MC	
	Indication Error	Repeatability	Indication Error	Repeatability
Grade 0.2	±0.2%	≤0.1%	±0.4%	≤0.2%
Grade 0.5	±0.5%	≤0.2%	±1.0%	≤0.5%
Grade 1	±1.0%	≤0.5%	±2.0%	≤1.0%

Traceability of Moisture Meter

- **JJG 891–1995**
  - **Requirements on conditions of verification**
    - **Equipments demanded (105 ℃)**
      - Balance: resolution 0.1mg
      - Electric Heated Ventilation Constant Temperature Chamber: control accuracy ±2℃
      - Dryer and allochroic silicagel: blue
      - Aluminium case
    - **Other equipments (other methods)**
      - Uncertainty should be lower than 1/3 of tested instrument

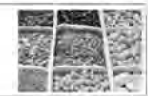
Traceability of Moisture Meter

- **JJG 891–1995**
  - **Measurement of standard moisture by the verification instrument:**
    - Sample preparation (add moisture to the sample that moisture is lower than measured points)
    - Determination of standard method
      a) 7%~18%
      pulverization, heat, result calculation
      b) >18%
      pulverization, 1st heat, 2nd heat, result calculation

Arising Problems

- Disadvantages of standard routine reference method
  - **Time consuming**
  - **Require pretreatment**
- Disadvantages of new methods
  - **Influenced by temperature, moisture density, shape of sample and other factors**
  - **Difficult in handling**
  - **Expensive and difficult to expand applications**
  - **Limited within certain type of grains**
  - **Time consuming and laborious**

Future Trends



- Because determination of MC is affected by temperature, density, mass and other factors, drawing the integrated effects of physical quantities on the determination of MC will fix this problem. Finally, accurate results of MC can be obtained.
- New determination techniques for MC with higher accuracy, less influencing factors and simple handling need to be developed.
- General-purpose rapid moisture meter of low-cost will be in high demand.

Conclusions

- MC plays an important role not only in storage of grain but also in grain marketing and handling.
- Measurement of grain moisture is not just a technical issue, but also associate with the vital interests of most Chinese farmers.
- Standard method (GB/T 21305–2007)
- Verification regulation for instruments (JJG 891–1995)
- Existing methods
- Therefore, more accurate, high throughput, more efficient and easier handling methods are in great demand.

Rice Moisture Meters

APEC/APLMF
Workshops on Metrology in Agricultural Products, Food Safety and Product Safety

September 23-25, 2009
Ho Chi Minh City, Viet Nam

Sakchai Hasamin
Thailand

Contents

- Regulation
- Traceability
- Reference Moisture Determination
- Primary Standard Calibration
- Secondary Standard Calibration
- Initial and Subsequent Verification
- In-Service Inspection

Regulation

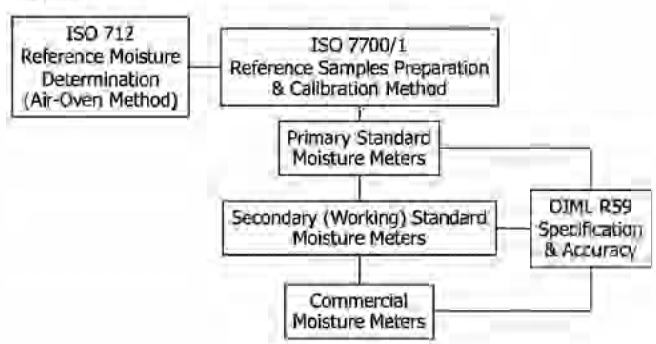
Ministerial regulation No.2 (A.D.2004)

- Article 55/6 Maximum permissible errors for rice moisture meters are prescribed as follows

Standards		MPE	
		Initial and subsequence verification	Inspection
Reference sample	Moisture content not exceeding 16%	0.8%	1.0%
	Moisture content exceeding 16%	0.05 x moisture value (%)	0.06 x moisture value (%)
Moisture meter		0.8%	1.0%

- Article 89 Rice moisture meters are required to be re-verified every 2 years.

Traceability



Reference Moisture Determination

Preparation of reference samples

- Procedure
  - ISO 7700/1: Check of the calibration of moisture meters — Part 1: Moisture meters for cereals
    - Selection of samples
      - Type: Paddy
      - Variety: Jasmine 105 (Khoa Dawk Mali 105)
      - Pathum Thani 1, Suphan Buri 1 and Chai Nat 1
      - Moisture range: 10% ~ 25%

draft version OIML RSR April 2005, 6. Technical recommendations - Grains and moisture meters

Reference Moisture Determination

- Cleaning of samples
  - Sieve and Mechanical separator
- Conditioning of samples
  - decreasing moisture content of fresh paddy samples by drying at the room condition and in the oven at temperature not exceed 30°C (or 60°C in case lower than 16 % of moisture content is needed)
- Homogenization of samples
  - Rolling machine

Reference Moisture Determination

Determination of moisture content of reference samples

- Procedure
  - ISO 712: Cereals and cereal products — Determination of moisture content (Air-oven method)
    - oven
    - 120 min ± 5 min
    - 130°C ± 3°C
    - readability of weighing scale 1 mg
- Uncertainty
  - 0.3% (95% confidence interval)

Fan-forced oven

Grinding mill

Dessicator

Thermocouple

Thermo-hygrograph

Primary Standard Calibration

- Calibrate primary standard moisture meter by reference samples at least 30 samples which their moisture contents are known
  - Procedure
    - ISO 7700/1: Check of the calibration of moisture meters — Part 1: Moisture meters for cereals
  - Uncertainty
    - 0.8% (95% confidence interval)

Secondary Standard Calibration

- Calibrate secondary (working) standard moisture meter against primary standard
- Using reference samples at least 5 samples
  - Procedure
    - ISO 7700/1: Check of the calibration of moisture meters — Part 1: Moisture meters for cereals
  - Uncertainty
    - 0.9% (95% confidence interval)

Initial and Subsequent Verification

- Verify commercial moisture meter against working standard
- Using reference samples at least 3 samples
  - Maximum Permissible Error
    - 0.8%

Collecting samples at high moisture content in the harvesting time

Removing impurities such as straw, chaff and weed by winnower

Decreasing moisture content of samples by crying at the room condition

Homogenize the moisture content of samples after moisture content adjustment by the rolling machine

Importer prepares the moisture meters before test

Importer prepares the moisture meters before test

Keeping the moisture meters and test samples to reach thermal equilibrium under the laboratory temperature before the test

Verification of rice moisture meters

In-Service Inspection

- Check in-service moisture meter against working standard
- Using reference samples at least 3 samples
  - Maximum Permissible Error
    - 1.0%

Thanks you for your attention.

CENAM

Asia-Pacific Economic Cooperation

Grain moisture measurements in Mexico

Enrique Martines-Lopez
Moisture measurements laboratory/CENAM

CENAM

Content

Asia-Pacific Economic Cooperation

- Introduction
- Gravimetric (drying oven) method
- Implementation of National Standard of Moisture Measurements
- Grain moisture conditioning systems
- Final comments

Introduction

- Water is present in any kind of solid materials, in particular in grain and cereals
- Its presence could affect to the properties of grain (mechanical, physical-chemical, biological, nutritional, etc.)
- The excess of water could deteriorate the grain and too dry grain affects to the flowing and nutritional properties

  (grain should not be too wet or too dry)
- Pricing depends of the moisture content

Introduction

- The grain moisture measurements have been growing during the last few years.
- Moisture measurements have an economic impact on the international and domestic grain trade.
- These measurements will be more important in use of transgenic grains for food and fuel production.

Main uses of grain

- Human consumption
  45,9%, Mexico 2007

- Animal consumption
  54,1 %, Mexico 2007

- Fuel
  0 %, Mexico 2007

Trade of grain in Mexico

- Mainly grain for human consumption: corn, bean, wheat and rice
- Production of grain (2007): 28×10^9 kg
- Imports (per year):

  Corn: 9×10^9 kg

  Wheat: 4×10^9 kg

  Rice: 1×10^9 kg

  Sorghum: 2.5×10^9 kg

Needs of measurement

Mexico has about 2000 grain moisture meters used for

- Official inspection organizations (PROFECO, SEMARNAP, SAGARPA, etc.)
- Producers, packers, handlers, stores
- Food processing companies
- Importers, exporters

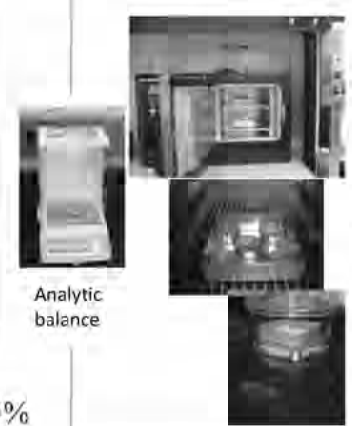
without a suitable metrological support

Gravimetric (drying oven) method

- It is an absolute method; It is internationally recognized as a primary method
- It could be applied to many kinds of solid materials
- It is compatible with international standards
- It is widely used in national laboratories in the implementation of the national measurement standard

Gravimetric method

- Wet mass (m_w)
- Drying (T, time)
- Dry mass (m_d)



$$MC_{wb} = \frac{m_w - m_d}{m_w} \cdot 100\%$$

Implementation of the National Standard of Moisture Measurements

- Facilities
- Training
- Equipment
- Characterisation and calibration of equipment
- Evaluation of uncertainties

Implementation of the National Standard of Moisture Measurements

- Development and characterisation of moisture conditioning method
- International comparisons
- Report of National Standard

Facilities and equipment

- Laboratory with controlled ambient conditions (T, RH)
- Fume extracting hood
- Dry oven with forced convection (45 °C to 325 °C)
- 2 Analytical balances
  - range: 220 g, resolution 0.1mg, U=0.4 mg
  - range: 3.1 kg, resolution 0.1 g, U=0.2 g
- Grain moisture meter (capacitance; commercial)
- Saturated salt solution chamber with 3 salts

Facilities and equipment

- Temperature measurement system
- Sampling system
- Glass dryers
- Moisture conditioning system
- Sieves
- Manual grinding mill

Characterisation of dry oven using forced convection

Calibration of equipments

The instruments involved in the implementation of the gravimetric method were calibrated by CENAM with traceability to domestic standards

Main uncertainties with drying oven method

- Mathematical model (MC_{wb})
- Drying time
- Drying temperature
- Sample mass
- Ambient conditions

Combined uncertainty of the standard

$$u(\%MC) = \sqrt{u^2_{\text{model}} + u^2_{\text{drying-temp}} + u^2_{\text{drying-time}} + u^2_{\text{sample-mass}}}$$

Uncertainty evaluation for some grain

Uncertainty source	Rice	Corn	Sorghum
	MC(%)		
u_{model}	0.01	0.01	0.01
$u_{\text{drying-temp}}$	0.06	0.11	0.04
$u_{\text{drying-time}}$	0.02	0.06	0.03
u_{mass}	0.03	0.10	0.03
u_c	0.07	0.16	0.06

Grain moisture conditioning methods

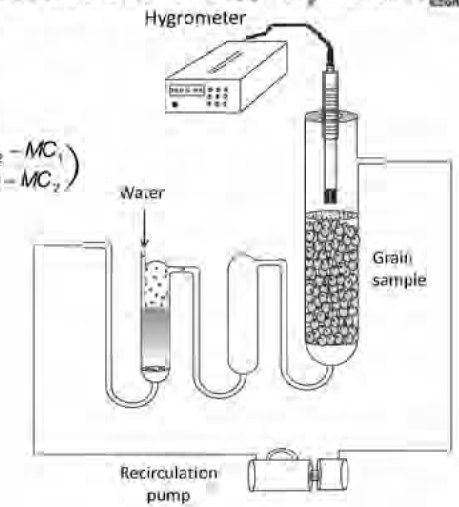
- Direct methods (grain ± water)
- ISO 7700/1 (84)
- Moisturizing system

- Indirect methods (MC ~%RH)

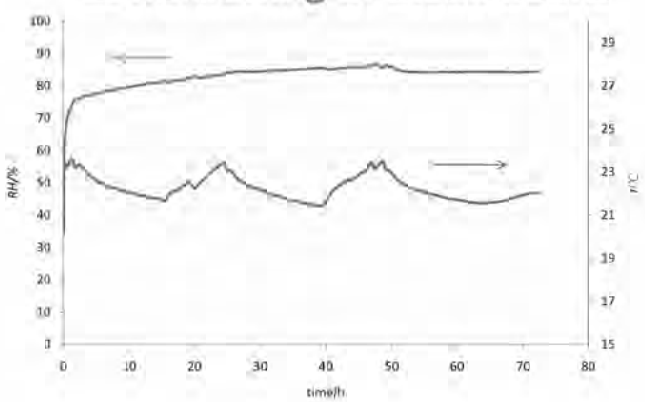
- Saturated salt solutions
- Humidity chamber with recirculation

Moisturizing system

$$m_{H_2O} = m_W \bullet \left(\frac{MC_2 - MC_1}{100 - MC_2}\right)$$



Conditioning of black beans



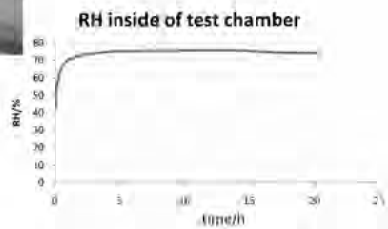
Grain conditioning

	Pinto bean
mw(before)	300.0 g
MC_1	10.8 g
m_{H_2O}	5 g
mh (after)	304.8 g
H_2 calculated	12.20 %
H_2 measured	12.1 %
Difference/m_{H_2O}	0.2 g
Difference in MC/%	0.10 %
u/%	0.09 %

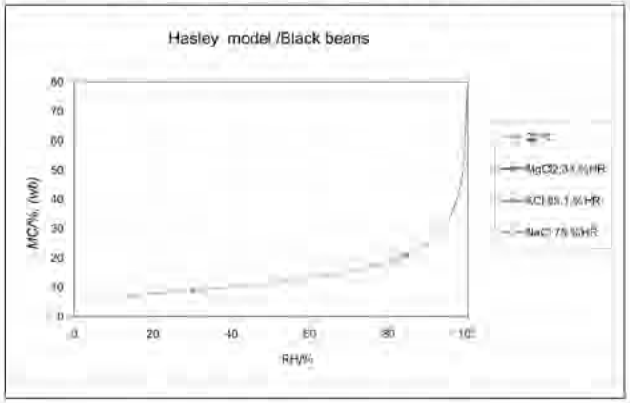
Saturated salt solution

NaCl:75,5 %HR @ 25 °C
Water: 100 mL
Salt: 335 g

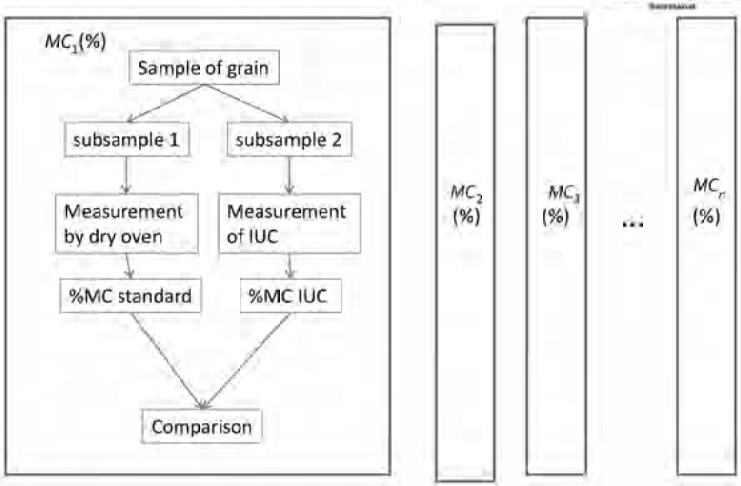
Grain conditioning time:
2 weeks



Conditioned grain at three moisture values



Grain moisture meters calibration procedure



Example of calibration results of a moisture meter

Type of grain	Standard			Moisture meter	Correction	Coverage factor	Uncertainty
	Drying temperature / °C	Drying time/h	MC(%)	MC(%)	MC(%)	k	U(%)
Corn (popcorn)	129.8	4	12.9	12.9	0.0	2	0.4
	129.9	4	13.6	13.5	0.1	2	0.5
	129.8	4	14.4	14.3	0.1	2	0.5

MC: moisture content wet basis

Final comments

- Large amounts of grain is bought and sold around the world.
- The price depends on its moisture content. Therefore, it is necessary to have a standard to give confidence in its measurement.
- At this time, at CENAM we are implementing the National Moisture Standard, which will provide traceability and confidence to the national measurements.
- It will also give metrological support to grain exporters and importers.

THANK YOU!

THE TRACEBILITY OF RICE MOISTURE METER IN INDONESIA

by:
Metrology of Indonesia

INDONESIA

RICE

- Rice is main of agricultural commodity in Indonesia
- Production of rice had affected of field economic, employment, ecology, social and politics
- Thus, we need government policy to stabilize the prices, sufficient of national production and product safety on rice market.

MOISTURE METER

- Moisture is amount of water content in food product that concerned in percentage
- Moisture content in rice can affected the safety of rice because the high level of moisture content can produce bacteria, fungus and khamir easily when its stored in chamber

so.....

need a reliable rice moisture meter

BULOG

- National logistical supply of rice organization in Indonesia
- BULOG has responsibility:

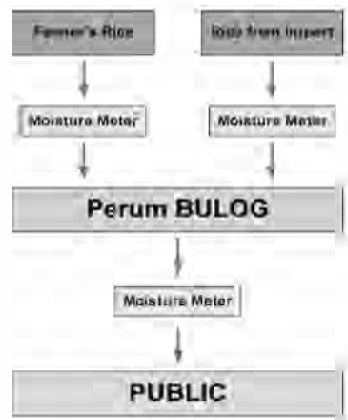
a. Procurement,

b. Distribution,

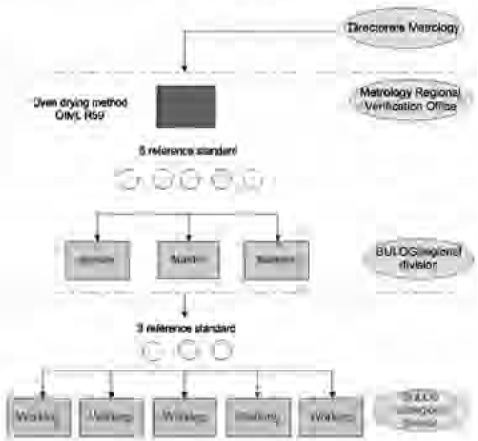
c. Government rice reserve

- BULOG infrastructure is divided into regional division (DIVRE) located in province and sub regional division (SUBDIVRE) in sub province

TRANSACTION OF RICE



TRACEABILITY SYSTEM OF RICE MOISTURE METER



REFERENCE

- OIML R 59 : Moisture Meters for Cereal Grains and Oilseed
- Keputusan Direktorat Metrologi Nomor 921/Dirmet-1/III/1997 tentang Syarat-syarat Teknis Khusus Meter Kadar Air

CONCLUSION

- Rice is main agricultural commodity that had effected in many field because of that need government policy in aimed to market of rice;
- Moisture meter subject to legal control because used for the public domain, custody transfer, trade transaction and safety;
- Traceability of rice moisture meter is responsible of BULOG and referred to national traceability system.

Checking the Calibration of Rice Moisture Meters

APEC/APLMF
Workshops on Metrology in Agricultural Products, Food Safety and Product Safety

September 23-25,2009
Rex Hotel, Ho Chi Minh City, Viet Nam

Warachai Triarun
Thailand

Contents

- Introduction
- Selection and cleaning of samples
- Preparation of test samples
- Determination of moisture content
- Checking the moisture meter
- Expression of results
- Result of checking the calibration curve

Introduction

- This test is objective to check the calibration curve of rice moisture meters before access to legal metrological control
- This test is carried out conforming to international standard ISO 7700/1:1984 (E): Check of the calibration of moisture meters—Part 1: Moisture meters for cereals
- This test is checking the calibration curve of jasmine paddy mode of Kett PM-410 (4053) and EE-KU 60th Anniversary by jasmine 105 variety of paddy samples collected from northeastern region of Thailand in crop year 2008

Manufacturer: Kett, Model: PM-410 (4053)

Manufacturer: EE-KU, Model: 60th Anniversary

Selection and cleaning of samples

- Collection of samples
  - In-season paddy
  - Jasmine 105 variety
  - High moisture content
  - Harvesting time
    - Nov 2008
  - Date of collection
    - Nov 15-18,2008
  - Place of collection
    - Northeastern region (4)

Selection and cleaning of samples

- Cleaning of samples
  - Remove lighter impurities such as straw, chaff and weed by winnower
  - Remove undersize and larger impurities such as small rock and soil by sieve and hand

Preparation of test samples

- Procedure when checking several values
  - Each sample having a mass of ~1 kg
  - Various moisture contents between 10%~25%
  - Samples should have moisture contents in their natural state
  - If necessary, samples specially conditioned by the procedure specified in ISO 7700/1

Preparation of test samples

- Conditioning of samples
  - Using decreasing moisture content of samples by drying at the room condition or in the oven at the temperature not exceeding 30°c and 60°c in case of desired moisture content below 16%
  - Homogenize moisture content of samples after moisture content adjustment by put on the rolling machine about 1~2 days

Determination of moisture content

- Reference method
  - ISO 712: Cereals and cereal products — Determination of moisture content—Routine reference method
- Apparatus
  - Usual laboratory apparatus and, in particular, the following.
    - Analytical balance
    - Grinding mill
    - Metal dish
    - Constant-temperature oven
    - Desiccator

Determination of moisture content

- Preparation of test sample
  - Grinding without pre-conditioning
    - The moisture content is between 9%~15%, carry out grinding without pre-conditioning

Nominal aperture size of sieve in accordance with ISO 3310-1

Requirement of particle size distribution of products after grinding

Determination of moisture content

- Preparation of test sample
  - Grinding with pre-conditioning
    - The moisture content is more than 15%, carry out grinding with pre-conditioning to bring the moisture content to between 9%~15% before grinding
      - Carry out a pre-dying about(7~10)min and cooling down in the laboratory temperature with the dish uncovered and without a desiccator for at least 2h

Determination of moisture content

- Procedure
  - Carry out two single determinations at the same time
    - if the absolute difference between the two results is more than repeatability limit r ($r = 0.013m$-0.06,where m is the mean of the two test results) repeat determination until the result meets this requirement

Determination of moisture content

- Rapidly weigh all the grindings obtained to the nearest 0.001 g in the dish previously dried and tared together with its lid to the nearest 0.001 g

Determination of moisture content

- Drying the open dish containing the test portion together with the lid in the oven and leave 120 min ± 5 min from the moment when the oven temperature is again 130°c ± 3°c

Determination of moisture content

- Rapidly take the dish out of the oven, cover it and place it in the desiccator when the dish has cooled to laboratory temperature about(30～45)min then weigh it to the nearest 0.001 g

Determination of moisture content

- Calculation
  - Without pre-conditioning

$$w = \left(1 - \frac{m_1}{m_0}\right) \times 100\%$$

  - With pre-conditioning

$$w = \left(1 - \frac{m_1 \cdot m_3}{m_0 \cdot m_2}\right) \times 100\%$$

Where:
W is the moisture content expressed as percentage
m_0 is the mass, in grams, of the test portion
m_1 is the mass, in grams, of the test portion after drying
m_2 is the mass, in grams, of sample taken before pre-conditioning
m_3 is the mass, in grams, of the preconditioned sample

Checking the moisture meter

- Procedure when checking several values
  - Keep the test samples under the laboratory conditions (temperature 25℃ ± 2℃ and humidity 50% ± 10%) as the moisture meter before the test to allow them to reach thermal equilibrium with the moisture meter
  - Reject the test samples that emit an odor of fermentation or are moldy

Checking the moisture meter

- Determine the moisture content by the routine reference method specified in ISO 712
  - Take as the result the arithmetic mean of two determinations
- Using the moisture meter, carry out 4 successive measurements using 4 test portions taken from the test samples

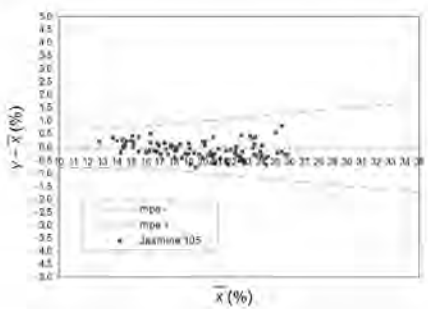
Expression of results

- Procedure when checking several values
  - For each test sample, the following values are available
    - Two results obtained by the routine reference method, x
      - The difference between these two results shall not exceed 0.15% for the test samples not requiring pre-conditioning and 0.20% for the test samples requiring pre-conditioning
      - Otherwise, repeat the test
    - Four measurements carried out with the moisture meter, y

Expression of results

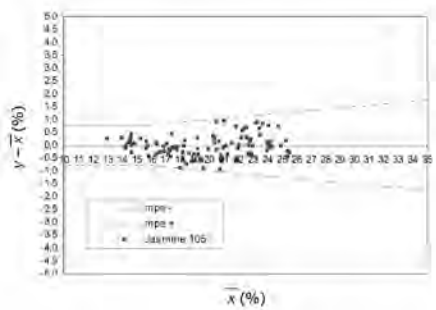
- For each test sample, calculate the difference between the result of each measurement carried out with the moisture meter, y, and the mean of the two results obtained by the routine reference method, $\bar{x}$, i.e. $y - \bar{x}$
  - The values $y - \bar{x}$ shall be less than Maximum Permissible Errors
    - MPE = 0.8% for moisture content of test sample not exceeding 16%
    - MPE = 0.05 x moisture value% for moisture content of test sample exceeding 16%

Result of checking the calibration curve



Rice moisture meter
- Kett
- PM-410 (4053)
- Jasmine paddy mode
- Testing samples
  - In-season Paddy 2008
  - Jasmine 105 variety
  - 103 samples

Result of checking the calibration curve



Rice moisture meter
- EE-KU
- 60th anniversary
- Jasmine paddy mode
- Testing samples
  - In-season Paddy 2008
  - Jasmine 105 variety
  - 103 samples

Thanks you for your attention

PREPARATION OF REF. MATERIAL OF MELAMINE IN MILK POWDER

APEC/APLMF Seminars, HCMC, Viet Nam
23-25 September 2009

Yiu-chung WONG, Hong Kong,China Government Laboratory

www.govtlab.gov.hk

Role of CRM

- Unique metrological property: Traceability link (SI / national)
- Excellent external quality control mechanism:
  - Establish measurement confidence
  - Identify problems; self-improvement
  - Determine measurement bias (accuracy) and give good MU estimate

www.govtlab.gov.hk

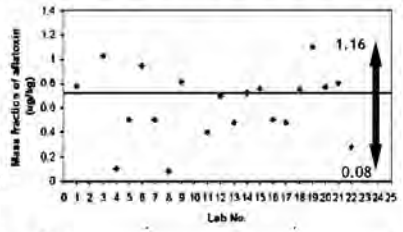
Accuracy & Precision

Good Precision but not good accuracy

CRM

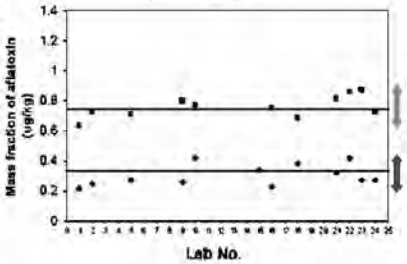
Good Precision and good accuracy

Eg. Application of CRM



- No CRM

large data dispersion

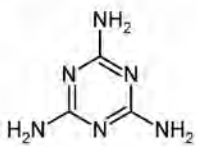


- With CRM

dispersion improved

Melamine Crisis 07-08

- In 2007, melamine was found in pet food in the US, caused fatal renal failure of animals
- In Sept. 2008, cases of kidney stones affecting infants who have consumed melamine-tainted infant formula were reported in China, >54,000 children needed medical treatment.



- To protect public health, the local government amended the Harmful Substances in Food Regulation (Cap. 132AF) on 23 Sept 2008:
  Infant milk: <1 mg/kg
  Other foods: <2.5 mg/kg

Melamine Crisis

- Recognized as one of the largest food incidents, resulted in extensive recall & monitoring worldwide
- Laboratories experienced a sudden & huge demands for melamine tests
- New in-house methods such as GC-MS; LC-MS/MS, ELISA, CE, etc. were developed
- No interlaboratory comparison programs and CRM for validation & to evaluate technical competence

Local Program

- A melamine interlaboratory comparison organized in late Sept 2008 for local labs:
  —ascertain their testing capabilities
  —assist their accreditation application
- Preparation of milk samples (4 different conc.)

Date	Action
6 Oct 08	Sample prep^{n} + homogeneity test
8 Oct 08	Sample dispatch to 14 participants
26 Oct 08	Submission deadline
29 Oct 08	Stability test
6 Nov 08	Issuance of Report

Assigned Values

- Gravimetrical spike of melamine at the concentrations of 0, 0.05, 1.2 and 4.5 mg/kg
- Confirmed by a validated LC-MS/MS method
- Estimation of MU:

Sample	Assigned value ± MU (mg/kg)
1	0.0500 ± 0.0089
2	1.200 ± 0.072
3	0
4	4.497 ± 0.261

Performance Assessment

Participant's Value

Assigned Reference Value: Determined using gravimetric method

$$Z = \frac{X - \bar{X}}{\sigma}$$

Performance SD: Horwitz equation

Results

- The *z*-score results indicated that **9** labs. were able to detect melamine at the legal limit, ie. 1 mg/kg, & at 4.5 mg/kg.
- **4** labs gave false positive results for the blank sample (Sample 3).
- Only **3** labs. have the capabilities to accurately quantify low level of melamine (0.05 mg/kg).

*Food Additives & Contaminants, 2009 (26) 1450-1458.

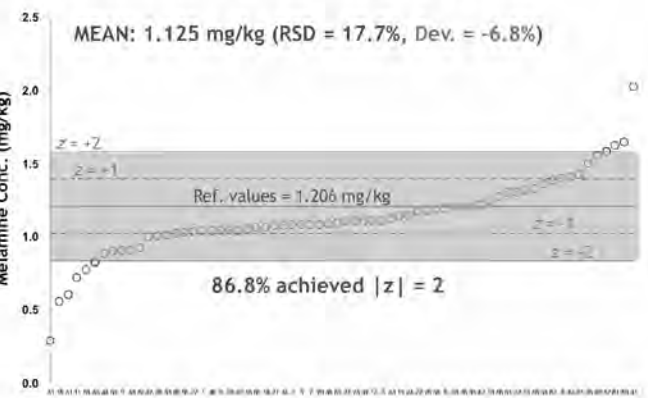
Interlaboratory Comparison Programs

Program	No. of Participants / Economies	Completion
Melamine in Animal Feed (APLAC T069) - One sample	52 / 20	Aug 2009
Melamine in Milk Powder (APLAC T071) - Two samples	76 / 28	Oct 2009

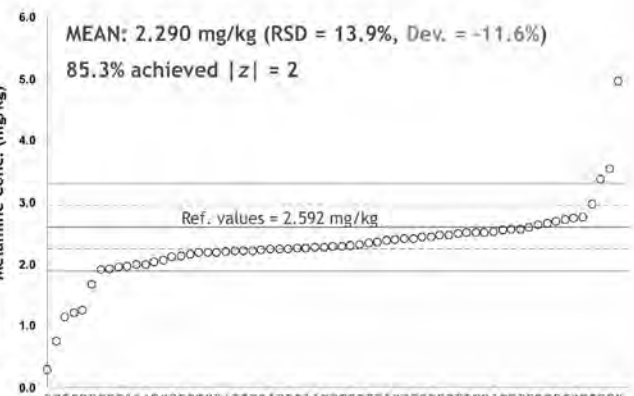
Participants

ECONOMY	No.	ECONOMY	No.
Australia	4	Indonesia	4
Brazil	2	Jamaica	1
Brunei	1	Japan	5
Canada	3	Korea	1
Chile	1	Malaysia	1
China	5	Netherland	1
Costa Rica	1	New Zealand	4
Croatia	1	Philippines	2
Czech Republic	2	Poland	1
Denmark	1	Sri Lanka	1
Estonia	1	Switzerland	4
France	1	Thailand	5
Germany	7	USA	3
Hong Kong,China	8	Viet Nam	4

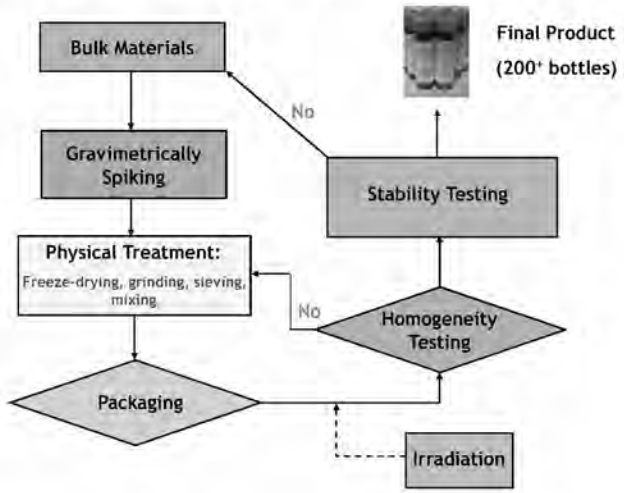
Performance (Sample I)



Performance (Sample II)



Preparation of CRM (ISO G-34)



Statistical Analysis

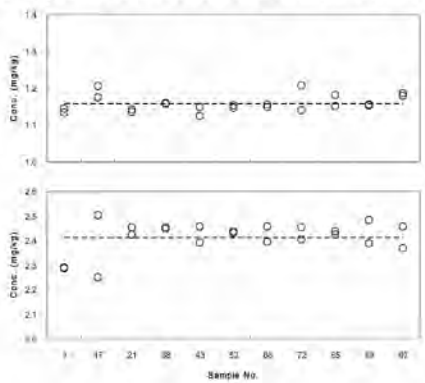
- Assigned values are based on the gravimetric values
- One-way ANOVA to evaluate CV_w & CV_b (variance within & between bottles)
- Sample inhomogeneity (u_{bb}):

$$u_{bb} = \sqrt{(CV_b - CV_w)/2}$$

$$\text{or} = \sqrt{\frac{CV_w}{n}} \cdot \sqrt[4]{\frac{2}{df}} \qquad \text{if } CV_w > CV_b$$

Homogeneity Test

- Using an accredited LC-MS/MS method



Stability Test

- One year study at 25°C will be performed (June 2009 to Jun 2010)

  RSD = 1.6% to 2.9%

- No statistical difference (till Sept 2009)

Purity Assessment

- Moisture content:

  Karl Fisher titration & GC-TCD

- Impurity by LC-MS/MS & LC-UV:

  melame
  meleme
  ammeline
  ammelide
  ureidomelamine
  methylmelamine
  oxytriazine
  cyanuric acid
  melamine cyanurate
  dicyanamide

Way Forward

- Continue to provide reference materials in food safety area to field laboratories
- Assigned reference values are traceable to SI units or determined using primary methods (eg. IDMS)
- Seek for RM Producer accreditation in mid 2010

THANK YOU

Assurance of Measurement of Foods and Agricultural products in China by Chemical Metrology

Prof. Zhang Qing-he
National Institute of Metrology, P. R. China
Sept . 23, 2009

Outline

1. Food Quality and Safety system in China
2. Status of Chemical Metrology in Foods and Agricultural products
   - ① Traceability Establishment
   - ② International intercomparisons and mutual recognition
   - ③ Certified reference materials
   - ④ Measurement instruments
3. Future Work Plan

Food Safety Law Regime

- **Food Hygiene Law （2009 June 1st）**
- **Metrology Law**
- the Quality and Safety of Agricultural Products
- Import and Export Commodity Inspection
- Animal and Plant Entry and Exit Quarantine
- Frontier Health and Quarantine
- Animal Disease Prevention
- The Product Quality Law
- These laws are then translated into rules and regulation of specific departments

Food Safety Regulatory System in China

Department	Field
The agriculture department	The production of primary agricultural products
The quality supervision and inspection department (AQSIQ)	◆The quality of food processing ◆The imported and exported agricultural products and other foodstuffs
The department of industry and commerce	The food circulation and distribution
The health department	◆The catering industry and canteens ◆The integrated food-safety supervision and coordination

Food Quality and Safety Standard System

- Over 1,800 standards concerning food and agricultural products, and over 2,900 for the food industry
- among which 634 national standards are compulsory.
  - the place of origin of agricultural products
  - animal and plant quarantine
  - good agricultural practices (GAP)
  - maximum amount of pesticides, veterinary drugs, pollutants and spoilage organisms allowed in food
  - food additives, packaging materials, special dietary food, signs or labels
  - testing methods concerning food

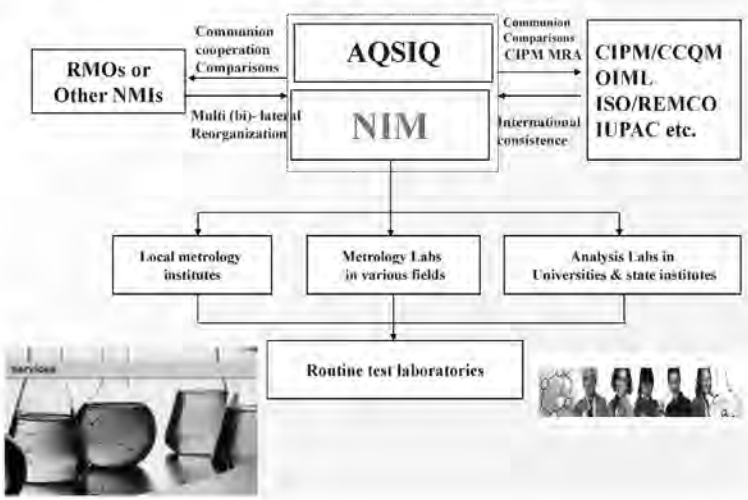
Food Safety Inspection and Testing Framework

- foodstuffs :
  - 48 state-level quality inspection centers
- agricultural products
  - 323 state- and ministerial-level quality inspection centers
- import and export foodstuffs
  - 35 state-level key laboratories
- 3,913 food testing laboratories have passed CNAS.
- These laboratories can detect all kinds of food-borne pathogens and 786 safety or hygienic items, such as residue of pesticides and veterinary drugs, additives and heavy metals.

Outline

1. Food Quality and Safety system in China
2. Status of Chemical Metrology in Foods and Agricultural products
   - ① Traceability Establishment
   - ② International intercomparisons and mutual recognition
   - ③ Certified reference materials
   - ④ Measurement instruments
3. Future Work Plan

Status & Role of NIM



Main Works of NIM

Sketch Map of Measurement Traceability in Chemistry Drawn by ISO/REMCO

1. Traceability Establishment
2. International intercomparisons and mutual recognition
3. Certified reference materials
4. Primary reference instruments

1:Traceability Establishment on Measurement of Foods and Agricultural products in China

Traceability pyramid

Traceability using CRMs

- The use of CRMs is very important for traceability of many measurement activities
- NIM is responsible that both measurement results and the property values of CRMs provided to the customer should be traceable to national or international standard.

Establishment of traceability

◆NIM by law responsible for implementation of national measurement system, Source of traceability to the SI for all users in the laboratories

◆Nucleus for national metrological infrastructure of accredited calibration and testing laboratories

◆International recognition of national measurement standards and traceability obtained from that NIM (calibration/measurement certificates, CRMs)

◆International/regional cooperation with other NMIs

2. International intercomparisons and mutual recognition

➢NIM participated activities of CIPM MRA, including intercomparisons of CCQM, APMP in food and agricultural products measurements:

- 45 key intercomparisons
- 50+ pilot study intercomparisons
- 41 key intercomparisons related to Food Safety
- 4 intercomparisons organized by NIM

International intercomparisons

CCQM-	Name	CCQM-	Name
P10	Gamma-HCH in Fish Oil	K39	Chorinated Pesticides in Solution
P21	p,p' -DDT in Fish Oil	P39.1	Methyl-mercury in salmon fish
K5	p,p' -DDE in Fish oil	K43	DiButylTin in sediment
P35	Ethanol in aqueous matrix (for. & commod. levels)	P44	DNA Quantification
P12	Pb in wine	P53	DNA Profiling
K24	Cd in Rice	P64	Trace elements in soyabean powder
K27	Ethanol in aqueous matrix	P20.E	Purity Series: Theophylline
K21	p,p' -DDT in Fish Oil	P78	Nutrients in infant/ adult formula
P40	Organic Contaminants in Mussel Tissue	P86	Analysis of total Se and Se methionine in pharmaceutical supplements

International intercomparisons

CCQM-P20A	TBT Chloride	CCQM-P54	DNA primary quantification
CCQM-P31.A	Organic Calibration Solutions (PAHs)	CCQM-P55	Peptide / protein quantification
CCQM-P31.B	Organic Calibration Solutions (PCBs)	CCQM-P58	Fluorescence in ELISA
CCQM-P31.C	Organic Calibration Solutions (Chlorinated pesticides)	CCQM-P59	Protein structural measurements by CD
CCQM-P20C	Atrazine	CCQM-P60	DNA extraction - reference method
CCQM-P20D	Chlorpyrifos	CCQM-K49	Toxic and essential elements in bovine liver
CCQM-K38	PAHs in Solution	CCQM-K30	Pb in wine
CCQM-K40	PCB Congeners in Solution	CCQM-K56&P64.1	Trace elements in Soybean powder
CCQM-P57	PCB Congeners in Tissue Extract	CCQM-K59	Determination of nitrite and nitrate in calibration solutions and natural water
CCQM-P61	Volatile organic compounds (VOCs) in solution	CCQM-P39.1	Methyl-mercury in salmon fish
CCQM-P67	PCBs Congeners in Tissue		

Mutual recognition: CMC

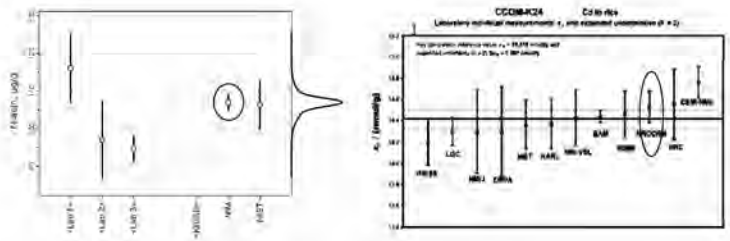
NIM has established primary reference measurement methods in food and agricultural products, gained CMCs.

◆BIPM website: NIM CHINA
- ➢Food：18

◆Related
- ➢High Purity Materials：12
- ➢Inorganic Solutions：37
- ➢Organic Solutions：19

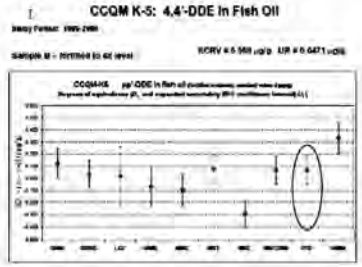
Support International Mutual Recognition in Food Safety

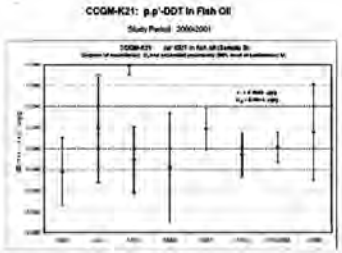
Intercomparison Graphs



CCQM- K62/P78.1 Nutrients in Infant/Adult Formula: Niacin.

Intercomparison Graphs





domestic Proficiency Tests Provided

- Investigated the testing capabilities of relevant labs in China.
- Provided an opportunity to the testing labs for their method validation.
- Improved the measurement capabilities of relevant labs through using CRMs and NIM technical training.

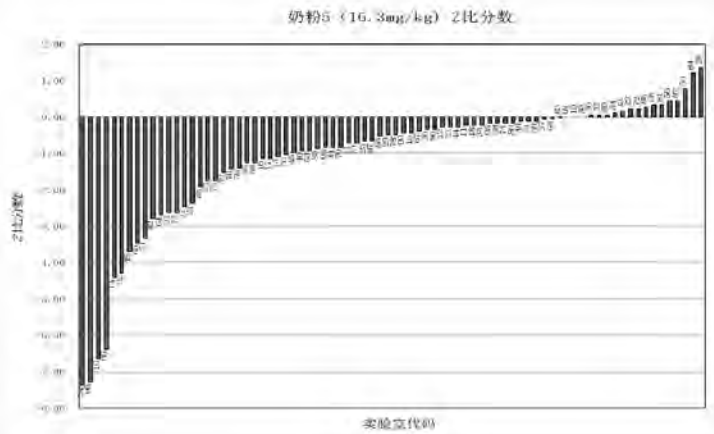
Proficiency Tests Organized

Year	Code	Name	Measurands
2003	CNAL T0131	Determination of anions in water	NO_3^-, Cl^-, SO_4^{2-}
2004	CNAL T0087	Organochlorine pesticides analysis	γ-BHC , P,P'-DDE
2004	CNAL T0248	Analysis of Organochlorine pesticides and PCBs in fish tissue	Hexachlorobenzene, Lindane, DDE, DDT, PCB28, PCB52, PCB101, PCB118, PCB138, PCB153, PCB180
2005	CNAL T0186	Determination of nutrient component in soybean powder	Protein , Fat , Fibre
			Ca , Fe , Zn
2006	CNAL T0250	Analysis of synthesis coloring matters in drink	Tartrazine
2006	CNAS T0330	Determination of acesulfame-K in beverage	Acesulfame-K

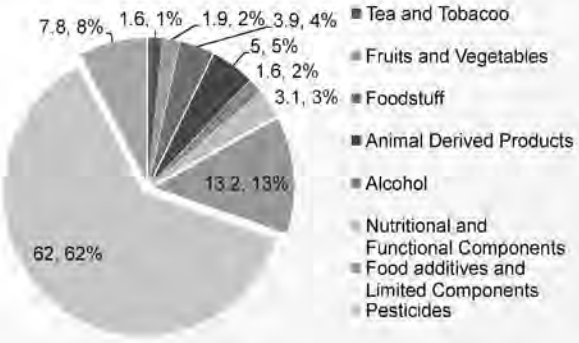
Proficiency Tests Organized

Year	Code	Name	Measurands
2007	CNCA-2007-B11	Determination of six pyrethrioids in concentrated apple juice	Bifenthrin, permetrhin, Lambda-Cyhalothrin, Fenvalerate, Deltamethrin, Fenpropathrin
2007	CNAS T0249	Quantification of cholesterol in egg yolk powder	cholesterol
2008	/	Melamine in milk	Melamine
2008	**T0402**	**Determination of Pb and Cd in wheat powder**	**Pb , Cd**
2009	/	Pesticides in tea	Pesticides

Determination of melamine in milk powder



2 CRMs for Foods and Agricultural products



pure, solution and matrix of inorganic and organic compounds

Foodstuff，Tea and Tobacco CRM

GBW08510	Cadmium in rice
GBW08511	Cadmium in rice
GBW08512	Cadmium in rice
GBW10010	Rice flour
GBW10011	Wheat flour
GBW10012	Corn flour
GBW10013	Soybean flour
GBW(E)080684	Rice flour
GBW(E)100009	Nutritious compositions in rice flour
GBW(E)100010	Nutritious compositions in wheat flour

No.	Name
GBW07605	Tea
GBW08516	Fluoride composition in tea
GBW08514	Tobacco
GBW08515	Tobacco

Nutritional and Functional Components Fruits and Vegetables CRM

Nutritional and Functional Components	Fruits and Vegetables
Melatonin	Laminaria japonica aresch
Genistein	Cabbage
Daidzein	Spinach
17 amino acid mixture	Tea
Glycine	Apple
Iodine in refined salt	
Germanium in ganoderma lucidum	
niacinamide	

CRM for Animal Derived Products

13 organochlorinated pesticides in salmon tissue
7 PCBs in salmon tissue
18 organochlorinated pesticides in tuna tissue
14 organochlorinated pesticides in tuna tissue
7 PCBs in tuna tissue
7 PCBs in tuna tissue (Arochlor fortified)
16 organochlorinated pesticides in salmon oil
7 PCBs in salmon oil

Food additives and Limited Components

Food pigment-tartrazine, amaranth, sunset yellow, ponceau 4R, brilliant blue in water, tetraiodofluorescein sodium salt , Allura Red AC, tetraiodofluorescein sodium salt ; Sudan I, Sudan II, Sudan III, Sudan IV, Sudan red 7b

Food sweetener: sodium saccharin，acesulfame potassium， sodium cyclamate

Food preservative: benzoic acid in water，benzoic acid in petroleum ether，sorbic acid in water，benzoic acid and sorbic acid in water，Sodium saccharin,benzoic acid and sorbic acid in water

Methyl p-hydroxy benzoate，Ethyl p-hydroxybenzoate，Propyl p-hydroxybenzoate，Butyl p-hydroxybenzoate

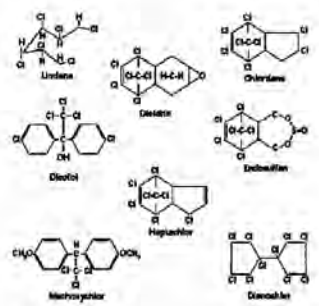
Calcium propionate, Sodium propionate

bromate in Water　　Caffeine

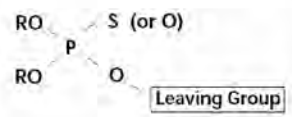
Aflatoxin B1 standard solution　　Sodium nitrite in water

CRM of Pesticides

Over 140 CRMs



Organochlorine

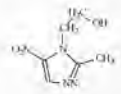


Organophosphate

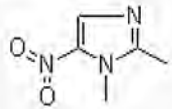
CRM of Veterinary Drugs

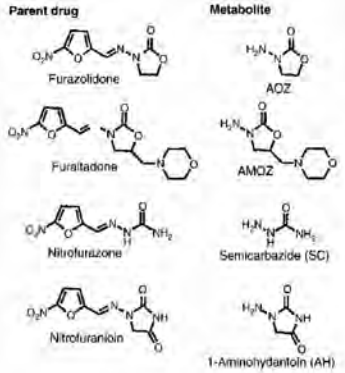
- Nitroimidazoles
- Nitrofurans

Metronidazole

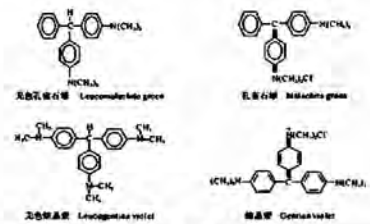


Dimetridazole





Malachite green and crystal violet



	Purity (%)	*U* (%)
Malachite green oxalate salt	99.1	0.8
Leucomalachite green	99.2	0.6
Crystal violet	98.9	0.9
Lecuocrystal violet	99.7	0.5

Doping Control **CRMs**

14 β-Agonists CRM

components	purity (%)	U_p (%)	C (mg/ml)	U_c (%)
Clenbuterol-HCl	99.3	0.7	1.01	3
Fenoterol hydrobromide	99.2	1.2	0.88	3
Tulobuterol	99.6	1.1	0.89	3
Pentubutolol sulfate	99.6	0.8	1.00	3
Salmeterol Xinafoate	99.5	0.9	0.10	3
Cimaterol	99.4	0.6	0.90	3
Salbutamol	99.4	0.8	0.89	3
Ractopamine hydrochloride	97.8	1.4	1.05	3
3-Hydroxytyramine hydrochloride	99.0	0.7	0.98	3
3-Methoxytyramine hydrochloride	99.3	1.0	0.99	3
Terbutaline hemisulfate salt	99.8	0.4	1.00	3
Clorprenaline hydrochloride	99.8	0.6	1.05	3
(±)-Propranolol hydrochloride	99.8	0.4	1.22	3
(±)-Epinephrine	99.4	0.6	—	—

CRMs database of China

4 Primary reference instruments

- Combustion Heat
- Viscosity
- Gravimetric Hygrometer/ Two-pressure Standard Humidity Generator
- pH
- Electrolytic Conductivity
- purity of chemical reagent
- FPD Primary measurement apparatus

Verification Regulation on Analytical Instruments

- Over 50 verification regulation on analytical instrument have been published
- Ultraviolet, visible, near-infraction spectrophotometeris
- HPLC, Ion chromatograp, GC, GC-mass, capillary electrophoresis
- AAS, AFS, AES, ICP-MS
- ELISA analytical instruments
- Total organic carbon analyzer
- Water quality synthetical analyzer

Establishment of national testing standards

- Rapid determination of melamine in raw milk-HPLC method
- Determination of 5 biogenic amines including histamine-HPLC Method
- Co-60 γ-ray standard irradiation field
- ESR technology to determine irradiation processing method for food of bone, meat and fish
- Thermoluminescence method to determine irradiation processing method for spice and dehydrated vegetables

Outline

1. Food Quality and Safety system in China
2. Status of Chemical Metrology in Foods and Agricultural products
   - ① Traceability Establishment
   - ② International intercomparisons and mutual recognition
   - ③ Certified reference materials
   - ④ Measurement instruments
3. Future Work Plan

Future research highlight

- Based on the legal metrology infrastructure, study the chemical metrology science and technology on food and agricultural products
- CRMs for Limited components and urgent components in food safety monitoring:
  - Pesticides, Veterinary drugs, Food Additives, Heavy Metal Elements, Food Nutrients, Food Packaging materials, Cosmetics.
- Construct emergency system of food safety
- Strengthen emergency processing capability of food safety

Thank you
for your attention !

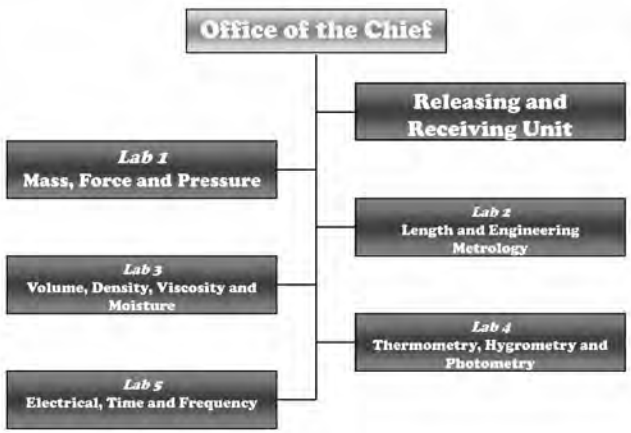
Overview of the Legal Metrology System in Agricultural Products, Food Safety and Product Safety in the Philippines

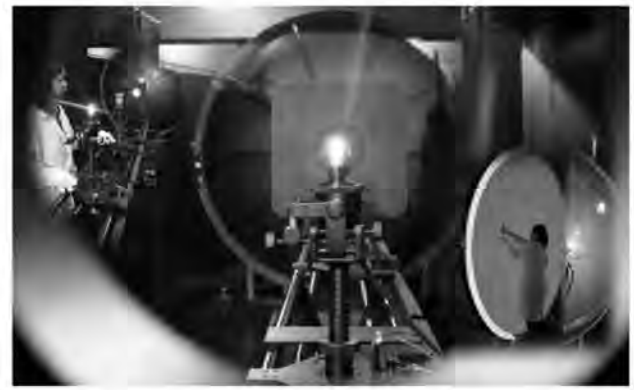
*I am **Marilyn C. Fos,** Senior Science Research Specialist working at the Viscosity, Density, Moisture, Volume and Flow Standards Section of the National Metrology Laboratory (NML) of the Industrial Technology Development Institute (ITDI) an agency under the Department of Science and Technology (DOST).*

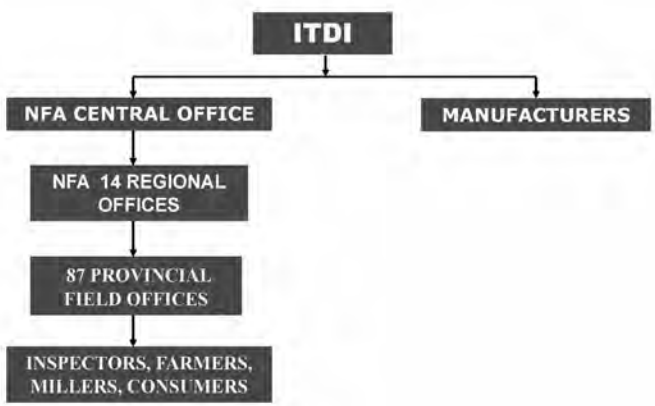
I have already spent 20 years in government service, 19 years of which in Metrology.

NML Goal - Provide accurate international traceability of the physical measurements undertaken in the country.

The NML, ITDI provides traceability to grains moisture meters and other moisture meters used in the industry and trade and other applications by performing verification on working standards of end users periodically. The ITDI applies International Standards ISO 712, ISO 7700 and OIML R59 as appropriate. The ITDI also conducts training / seminar – workshops upon request for end users on the verification of moisture meters.







- is primarily responsible for developing international recognition of the measurement capabilities of the region's national and territorial measurement laboratories. It is the oldest continually operating metrological grouping in the world.

- is a grouping of legal metrology authorities from the Asia Pacific Economic Co-operation (APEC) and other Pacific Rim economies.

The task of the BIPM is to ensure world-wide uniformity of measurements and their traceability to the International System of Units (SI).

Department of Agriculture (DA)

The principal government department responsible for the promotion of agricultural development and growth. It provides the policy framework, helps direct public investments, and in partnership with local government units (LGUs) provides the support services necessary to make agriculture and agri-based enterprises profitable and to help spread the benefits of Agricultural development to the poor and needy, particularly those in rural areas.

National Food Authority (NFA)

The National Food Authority was created through Presidential Decree No. 4 dated September 26, 1972, under the name National Grains Authority, (NGA) with the mission of promoting the integrated growth and development of the grains industry covering rice, corn, feed grains and other grains like sorghum, mongo, and peanut. This decree abolished two agencies, namely, the Rice and Corn Board (RICOB) and the Rice and Corn Administration (RCA) but absorbed their respective functions.

National Food Authority (NFA)

As of this date there are about 1.575m metric tons of imported rice from VietNam, Thailand and Pakistan for food security purposes in times of calamity and emergency and for stabilization purposes in deficit areas and during lean periods.

Philippine Rice Research Institute (PhilRice)

PhilRice is also an agency under the DA which is responsible to sustain rice self-sufficiency and to build a competitive rice economy through research, technology promotion, and policy advocacy.

International Rice Research Institute (IRRI)

An International Organization supported by different international organizations and many foreign governments to generate and disseminate rice-related knowledge and technology of short- and long-term environmental, social, and economic benefit and to help enhance national rice research and extension systems. With the assistance from many foreign researchers, it conducts research and development on different varieties of rice under different environmental conditions at different locations for different soil conditions.

These units are periodically calibrated using ISO 712.

Aug. 15 ~ 26, 2005 → Training on Traceability of Rice Moisture Meters, sponsored by NIST, NMIJ and Kett Japan

Participants : VietNam, Thailand, China, Malaysia, Myanmar, Indonesia, Lao PDR and Philippines

June 23 – 24 & 26 – 27, 2008

→ Training on Calibration of Moisture Meters

→ Participants: NFA Regional and Central Office Staff

→ Sponsored by: Aspen Philippines & Kett Japan

Harmonization of International Standards used in verification of moisture meters. Different countries produce different grains (rice) quality and use different standards on verifying moisture meters. This is a problem between importer and exporter of the product.

Local scene

Local regulatory bodies, suppliers and traders do not have the capabilities in terms of knowledge and laboratory standards and facilities regarding verification of moisture meters. Because of which, The ITDI is loaded with requests for verification of moisture meters.

a) Farmers need to be provided accurate and reliable moisture meters.

b) Drying systems should be improved. Use of Solar, or other dryers using non- conventional energy and storage systems for palay and other grain products which will assure control of quality especially during rainy season should be explored and encouraged and they should be made available to farmers.

c) Equip the necessary instruments and technical knowledge to calibration/ verification laboratories of moisture meters in local regulatory offices.

d) Perform the intercomparison between verification laboratory of ITDI and laboratories of testing offices or inspection bodies.

e) To implement a National Grains Standards through-out the country through continuing advocacy campaigns particularly among farmers, millers, rice grains businessmen and consumers on grains quality, labeling, weighing and packaging.

An archipelago of approximately 7,107 islands, the Philippines stretches from the south of China to the northern tip of Borneo. The country has over a hundred ethnic groups and a mixture of foreign influences which have molded a unique Filipino culture

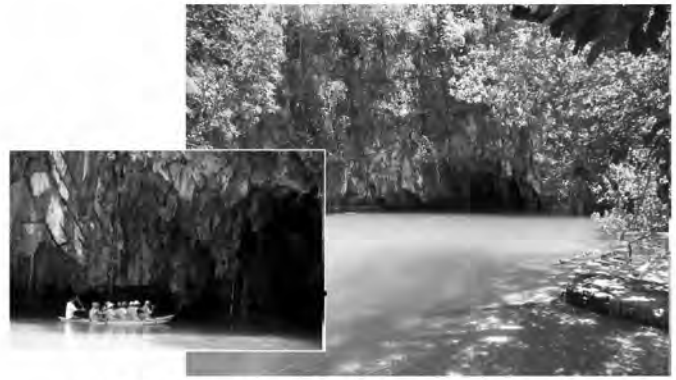
Total Land Area: 115,600 sq. miles / 299,404 sq. kms

Capital City: MANILA

Population: approximately 92 million people

Banaue - This majestic man-made wonder looks like a giant stairway leading to the sky.

Palawan group of islands



The Underground River or Subterranean River National Park of Puerto Prinsesa is now one of the New 7 Wonders of the World

Hundred Islands - This National Park covers a land area of 1,884 hectares with 123 islands.

Thank You!!!

DEVELOPMENT OF NATIONAL INFRASTRUCTURE TO SUPPORT LEGAL CONTROL OF FOOD SAFETY, AGRICULTURAL PRODUCTS AND PRODUCT SAFETY IN MALAYSIA

Dr Osman Zakaria
National Metrology Laboratory
SIRIM Berhad

WORKSHOPS ON METROLOGY IN AGRICULTURAL PRODUCTS, FOOD SAFETY AND PRODUCT SAFETY
23-25 SEPTEMBER, 2009
REX HOTEL, HO CHI MINH CITY, VIET NAM

NML....YOUR SOLUTION TO INTERNATIONAL TRACEABILITY

Contents of Presentation

- Introduction
- National Measurement System Act 2007
- Implications of the Act
- Development of Metrological Traceability for Chemical Measurements
- The Way Forward for Legal Measurements

What is the National Measurement System?

The totality of administrative and technical arrangements within a country which enables an individual or organization to have the means to make accurate and traceable measurements.

The NMS infrastructure consists of :

- Measurement standards
- Knowledge of measurement methods and how they may be used to obtain valid results
- The necessary organization to ensure that there are practitioners competent to provide measurement and calibration services which pass on accuracy and traceability to their recipients.

Why legislate the National Measurement System?

The mandate of the State is essential to :

- ensure conformity to measurement requirements
- suppress fraud
- provide trust and confidence to the measurement system

National Measurement System Act

- To provide for uniform Units of Measurement based on SI Units
- To provide for the establishment of national measurement standards
- To provide for measurement traceability to national standards
- To provide for the coordination of Malaysia's national measurement system

National Measurement System Act

- Non Application

This Act shall not apply to the use of units of measurements in:

- any international conventions;
- any agreements between governments in the fields of navigation by sea, air traffic and rail transport;
- the armed forces.

National Measurement System Act

- UPON THE COMING INTO OPERATION OF THIS ACT, EVERY MEASUREMENT SHALL BE MADE IN COMPLIANCE WITH THE REQUIREMENTS UNDER THIS ACT - if not so made the measurement shall be considered void

National Measurement System Act

- Realization and Maintenance of National Measurement Standards

"NATIONAL MEASUREMENT STANDARD" MEANS A MEASUREMENT STANDARD OR REFERENCE MATERIAL ESTABLISHED, MAINTAINED OR CAUSED TO BE MAINTAINED BY THE NATIONAL MEASUREMENT STANDARDS LABORATORY (NMSL) OR ORGANIZATION APPOINTED UNDER SECTION 12 (1) TO SERVE AS A BASIS FOR ASSIGNING VALUES TO A PARTICULA MEASURABLE QUANTITY.

National Measurement System Act

Traceability of Measurement

- ANY MEASUREMENT MADE FOR THE PURPOSE OF ANY WRITTEN LAW SHALL BE TRACEABLE TO THE NATIONAL MEASUREMENT STANDARDS

NATIONAL MEASUREMENT SYSTEM ACT

TRACEABILITY OF MEASUREMENT OUTSIDE MALAYSIA

- IF THERE IS NO TRACEABILITY TO THE NATIONAL MEASUREMENT STANDARDS, THE MEASUREMENT MAY BE TRACEABLE TO A MEASUREMENT STANDARDS LAB. OF ANOTHER COUNTRY OR TO A CALIBRATION LAB. IN ANOTHER COUNTRY RECOGNIZED BY NMSL.

National Measurement System Act

NATIONAL MEASUREMENT STANDARDS LABORATORY:

- APPOINTMENT OF A LABORATORY TO BE THE NATIONAL MEASUREMENT STANDARDS LABORATORY FOR CARRYING INTO EFFECT THE PROVISIONS OF THIS ACT

NML SIRIM Berhad appointed as the National Measurement Standards Laboratory on 15 February 2008

National Measurement System Act

FUNCTIONS OF NMSL

- To realize, establish and maintain or caused to be maintained, the National Measurement Standards
- To disseminate Units of Measurement that are traceable to the National Measurement Standards
- To maintain or caused to be maintained the Coordinated Universal Time (UTC)

National Measurement System Act

FUNCTIONS OF NMSL *(cont.)*

- To carry out research and to develop measurement technology and measurement standards
- To approve the patterns of measuring instruments
- To coordinate and promote the national measurement system

National Measurement System Act

FUNCTIONS OF NMSL *(cont.)*

- To assist the Council in matter relating to measurement technology and measurement standards
- To publish and disseminate technical information on measurement technology and measurement standards

National Measurement System Act

POWERS OF NMSL

NMSL may:

- Undertake international comparison of standards measurement standards
- Cooperate and collaborate with other measurement laboratories and institutions of higher learning
- Represents Malaysia in international measurement activities

National Measurement System Act

POWERS OF NMSL *(cont.)*

NMSL may:

- Impose fees and other charges as the Minister may prescribe by regulations
- Issue a certificate in respect of any reference material or recognize a certificate issued by any other person or body

National Measurement System Act

IMPLICATIONS OF ACT

- NMSA 2007 – Not conferred Power of Enforcement.
  - such enforcement powers remain with the respective enforcement
  - no restructuring of existing enforcement agencies necessary

National Measurement System Act

IMPLICATIONS OF ACT

- Existing enforcement legislations related to measurements need to be aligned to provisions of NMSA 2007.
  - use of SI units,
  - legal measurements to be traceable to national measurement standards.

National Measurement System Act

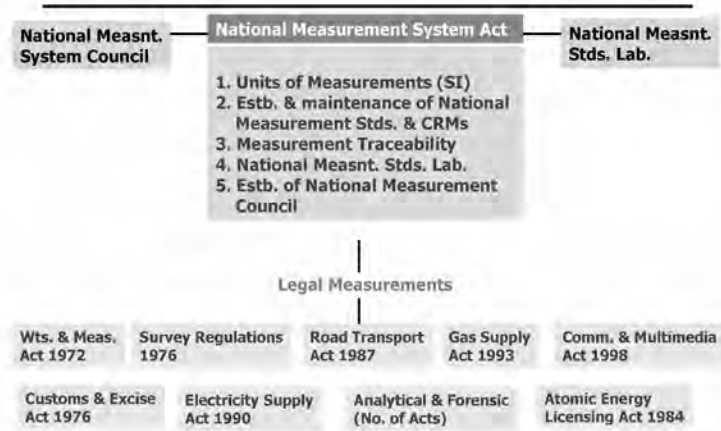
IMPLICATIONS OF ACT

- NMSL entrusted to realize and establish national measurement standards which are equivalent and comparable to international standards.
  - need for greater involvement in R&D activities and collaboration with international measurement laboratories,
  - need for continued government funding support.

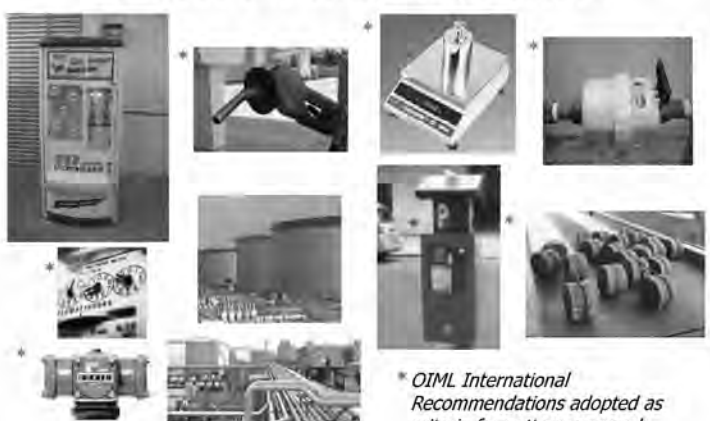
National Measurement System Act

IMPLICATIONS OF ACT

- NMSA 2007 – an "umbrella Act" for all legislations related to measurements.



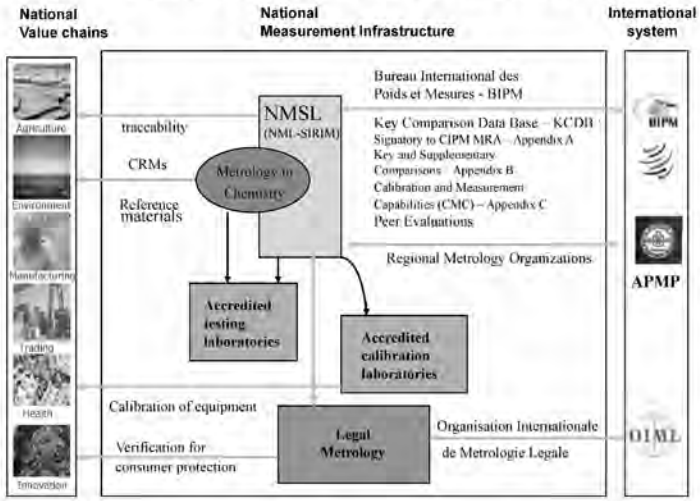
Regulated Measuring Instruments for trade and consumer protection



* *OIML International Recommendations adopted as criteria for pattern approval*

Medical Measuring Instruments regulated to ensure safety of users/patients

METROLOGY SYSTEM IN MALAYSIA



Food Sectors

- The government's aspiration is to be a world leader in food production and net exporter of food items by the year 2010.
- Malaysia economic growth prospect shows that agriculture sector is expected to grow at an annual average rate of 3.0 percent while the food sector at an average rate of 6.2 percent.
- The plan for for the development of the national food-agro products into a modernised, profitable and commercial entity.
- Malaysia has allocated a sum of RM 2.8 billion primarily for agriculture, animal husbandry, fishery and forestry for 2006.

Networking -Government Agencies

- Integrated several authorities include Ministry of Health (Food Quality Control Division, Pharmacy Division, and Disease Control Division), and Ministry of Agriculture (Department of Agriculture, Department of Veterinary Services, Department of Fisheries, and Federal Agricultural Marketing Authority); other ministries include the Ministry of International Trade and Industry, Ministry of Domestic Trade and Consumer Affairs, Ministry of Housing and Local Government, and Department of Royal Customs and Excise
- SIRIM Berhad is responsible to establishes standards for various food products where everybody can carry SIRIM mark as an indicator of the quality of their products

Standardisation and Accreditation

- SIRIM Berhad, appointed by the Department of Standards Malaysia as the National Standards Developing Agency continues to develop relevant Malaysian Standards through the industry Standards Committee on Food and Agriculture with the assistance of its respective Technical Committees and Working Groups (more than 491 Malaysian Standards have been developed)
- Developing standards that can protect consumer needs and at the same time ensure fair practices in food trade need the support from various parties including food industry e.g manufacturers, distributors, regulatory authorities, academicians, etc
- ISC A has established various technical committee and working groups to develop Malaysia Standards. ISC A will continue to oversea the development of Malaysian Standards on food and agriculture to support the standardisation in Malaysia

Cont'd

- To ensure the reliability quality and safe operation of products.
- The existence of these non-harmonised standards can contribute to the so-called "technical barriers to trade" which can partially impede the international trade flow.
- Malaysian Standard MS 1500 : 2004 Halal Food - Production, Handling and Storage - General Guidelines
- Malaysian Standard MS 1480 : 1999 Food safety according to hazard analysis and critical control point (HCCP) system.
- Malaysia Standard MS 1514 : 2001 General Principles of food hygiene.

Cont'd

- Skim Akreditasi Ladang Malaysia (SALM) run by the Department of Agriculture. The scheme is introduces to accredit the farms that impliment Good Agriculture Practice (GAP).
- Department of Agriculture and Federal Agricultural Marketing Authority (FAMA) have also introduce a national brand called Malaysia's Best. This is to ensure that our product is safe for consumption, for example, free from pesticide residue or heavy metal content.
- Ministry of Health (MOH) launched the national Hazard Analysis and Critical Control Point (HACCP)Certification Scheme for the identification, assessment and control of hazards during production, processing, manufacturing, preparation, delivery and use of food to ensure that the food is safe when consumed.
- SIRIM Berhad has also launched its own HACCP and combined HACCP/ISO 9001:2000 Certification Scheme.

National Infrastructure for Metrology in Chemistry

- The national strategies of Metrology in Chemistry (MiC) infrastructure will include the setting up the network partner to meet the need for a national platform as outlined in NMSA Act 2007
- All government's/competence laboratories enable to promote and provided with guidance on internationally recognised and accepted equivalence of measurement results in the field of metrology in chemistry and traceability to appropriate measurement standards

MAIN OBJECTIVES

- Promoting the concept of traceability of measurement results to the International System of Units (SI) or, where necessary, to other internationally agreed references;
- Promote close links between Government's Laboratories and National Metrology Institute (NMI);
- Coordinating and giving guidance in the establishment of Reference Measurement Systems with respect to regulatory needs;

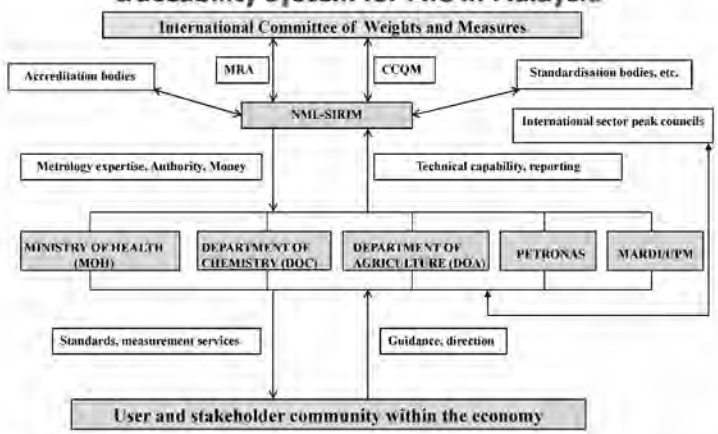
Cont'd..

- Identifying and prioritizing the measurands requiring international traceability and comparability and thereby encouraging appropriate organizations to accept responsibility for the development of suitable reference methods and measurement procedures and certified reference materials;
- Publicising widely relevant information to interested parties;
- Providing scientific and organizational expertise to the parties involved;

Cont'd..

- Permanent, direct contact to national expert institutes gives fast access to the relevant information on metrological needs in a subject field.

Proposed partnership model for the national traceability system for MiC in Malaysia



The network: selecting the partners

Coordinator

NML-SIRIM

Our competence:

- Inorganic analysis
- Surface analysis
- Electrochemistry
- Gas metrology
- Organics analysis
- Bio-metrology

selection process:

acquaintance process

intercomparisons

Participation in international comparisons (e.g. pilot studies of APMP, CCQM)

Network partner

Competence:

- ✓ pure materials
- ✓ gases
- ✓ gas mixtures
- ✓ gases for environmental measurements
- ✓ clinical analytes
- ✓ residues in food

New Building for Metrology in Chemistry

- The construction started in January 2007 and expected to be ready by December 2009
- Total cost about USD 5 millions was funded by the Government of Malaysia under Ninth Malaysia Plan
- To accommodate the high priority areas including gas metrology, organic analysis, inorganic analysis, Electrochemistry, surface analysis and biotechnology

Calibration and Measurement Services

- Evidential Breath Analyzer (EBA) to support Road Transport Department (Transport Act 1987)
- Vehicle Emission Devices and Chlorofluorocarbon Analyzer to support Department of Environment (Environmental Quality Act 1991)
- Gas detectors and other devices from industry

Method Development for Gas Analysis

- To develop a Primary Reference Materials (PRM) by gravimetric methods and participate in Key Comparison conducted by CCQM
- Area of interest : Green house gases, natural gases, IT gases, VOC's in air, ethanol in air and CRM's production
- Impurity analysis: GC-ICP/MS, GC-ECD, GC-TCD, GC-Methanator, GC-SCD, GC-FID and GC-MSD
- One staff from NML-SIRIM had attached to NPL, UK for 6 month in 2009 for Gas Standard Production

Method Development for Inorganic Analysis

- To develop a Primary Reference Method (IDMS) and participating in Key Comparison conducted by CCQM
- Area of interest: Food and health products, pharmaceutical products, construction materials and CRM's production
- To develop new capabilities for the analysis of RoH in semiconductor and electronic sectors
- Current facilities: ICP-MS/MS, Capillary Electrophoresis (CE), Ion Chromatograph (IC) and Microwave Digestion System

Method Development for Organic Analysis

- To develop a Primary Reference Method (IDMS) and participate in Key Comparison conducted by CCQM
- To develop a method for the analysis of pesticide residues in tea leaves and characterisation of the structure for pure substances using HR-NMR
- Analysis of malachite green, chloramphenicol and melamine in food products using LC-MS/MS
- Other facilities include were LC-ELSD, TGA, FT-IR, and GC-MS/MS

Method Development for Electrochemistry Analysis

- To develop a Primary Reference Method (Harned Cell) and participate in Key Comparison conducted by CCQM
- Area of interest: pH, Conductivity, Coloumetry and CRM's
- To develop and produce pH buffer using High Precision Glass Electrode

New Building For Chemistry Section

The new building is proposed for Chemistry Section with cost about USD 5 millions under the nine Malaysia plan. Equipped with gas laboratory, organic laboratory and clean room are ready, gas analysis, electrochemistry, surface analysis, trace metals, biotechnology, characterization of pure organic substance and CRM's production. The new building for Chemistry Section expected ready by January 2009.

- **First phase will be focused on the production of gas productions**
- **Second phase will be focused on the characterization of pure substance such as drugs, natural products, DNA profiling, pharmaceutical products, bio-metrology etc.**
- **Third phase will be focused on the trace metal analysis, electrochemistry, nanotechnology, biotechnology, surface analysis etc.**

Metrology in Chemistry – The Way Forward

- Bioanalysis – Biotechnology, DNA profiling, drug, steriod, hormone, protein analysis, nucleic acid/gene and cell measurement.
- Surface Analaysis – Nanotechnology, semiconductor, polimer coating, thin-film composition and corrosion.
- Nanomaterials and Nanotechnology – bio-materials, electronics sectors and etc
- Omics Analysis – protein, peptide, etc

Papua New Guinea
National Agricultural Research Institute
(NARI)
Chemistry Laboratory

by: Mr Peter Corbett

Overview

Designation

NARI is a statutory body coming under the Ministry of Higher Education, Research and Science Technology

The chemistry laboratory is part of NARI's technical services division

Operational funding covered from user pay testing

The Laboratory

As the National Agricultural Chemistry Laboratory, its mission is to provide agricultural, food and environmental diagnostic support to those requiring this service including:

- Laboratory services to rural farmers
- Commercial agriculture
- Quality advice to food and natural product producers
- Water providers and environmental monitors
- Laboratory training and advisory services

Laboratory Technical Sections

- NACL – soils, leaves, environment
- NFL – food, feed, natural products
- NRSL –rubber export certification testing
- QC – all sections

Analytical Work - 2009

- Drinking water and environmental testing
- Leaf nutrient analysis & plant health status
- Soils fertility testing & interpretation
- Food testing - inorganic nutrients, contaminants, vitamin A, energy, fibre, cyanide in cassava, aflatoxins, histamines, ochratoxins, caffeine
- Livestock feed testing
- PNGCR rubber quality certification, DM testing
- Pyrethrum & Vanilla quality testing
- Carbon, biomass studies

Instrumentation

- Core instruments
  - ICP
  - AAS, Flame &VGA
  - FIA
  - HPLC
  - CS Combustion analyser
  - Bomb calorimeter
  - Soxtec, keltec, fibertec
- Special rubber test equipment
- Standard general test instruments

Dry matter Testing

- Samples tested on DM content, method as requested by client or procedure
- Standard 105 degrees for 4 hours
- Fan assisted fresh air intake ovens
- NISIT certified ovens and balances

Quality Control

- ISO 17025 compliance. Accreditation to be applied for
- QC Manual
- SOP Manual– all procedures
- Lab method manual based on standard methods of analysis, AOAC, APHA etc
- Primary standards, reference standards, equipment calibration standards
- Quality of chemicals assessed
- QC check samples
- Routine QC performance control charts
- Equipment performance checks, ovens, balances, spectrometers, electrochemical meters, SOP's
- Good test record keeping and traceability of results
- Records – calibration, maintenance
- Audit checks
- Soils and plant Inter-laboratory sample exchanges
- Safety Manual

Quality Assurance

- In house reference materials
- Equipment operational calibration
- Comparative test methods and procedures for result validation and backup
- In house equipment maintenance, instrument repair, good electronics ability for instrument fault analysis
- Central UPS and power protection
- RO DI water supply
- Lab temperature, humidity, air control
- LAN
- Spare parts and consumables stock

Metrological Services

- National Institute for Standards and Industrial Technology (NISIT) – PNGLAS ISO 17025 lab certification accreditation inspectorate, industry standards, reference library, QA/QC training
- Measurement Standards Laboratory (NISIT) - instrument certified calibration
- NISIT Metrological Survey – identification of metrological needs including APMP &APLMF participation
- South Pacific Agricultural Chemistry Laboratory Network (SPACNET) – QC/QA training for SP island economy labs

Metrological Needs

- **ISO 17025 laboratory accreditation for:**
  - **Water testing**
  - **Food testing and quality certification**
  - **Rubber quality certification**
  - **Pyrethrum quality certification**
- **Organic laboratory certification**
- **Analytical instrument operational certification**
- **Standards for calibration of food test methods**
- **CRM's for foods and natural products**
- **Participation in inter-laboratory sample exchange programs for foods, natural products and water, especially APEC region**
- **Food and QC chemist to provide onsite training**
- **Attendance of metrological and QC training**
- **Up to date references, AOAC, APHA**

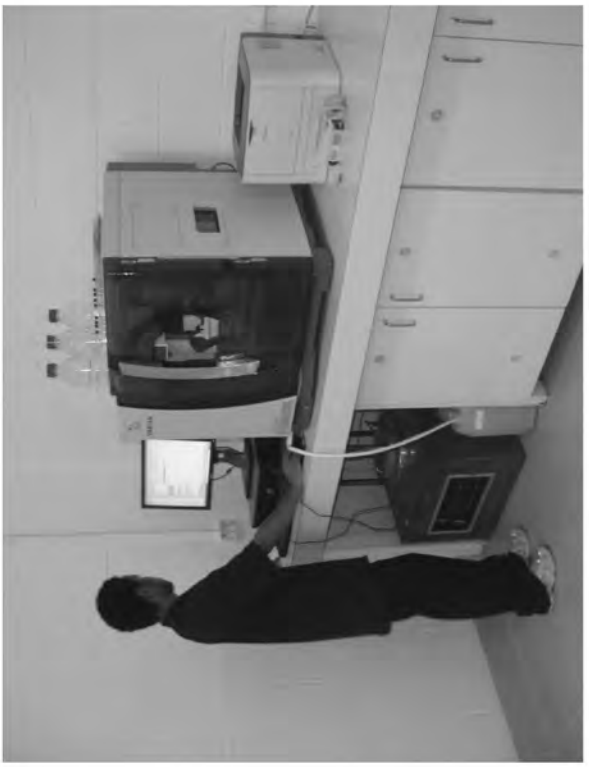
Development funding sources

EU – building renovation 2008

NADP – major equipment replacement 2009

AusAID – equipment, extensive infrastructure support 1996 to 2000

FAO – Accreditation and food lab enhancement assigned for implementation 2010



THE END

Thank you

Metrology, Standardization, Testing and Quality Management as tools in Food Safety: *Status at the Food Development Center of the National Food Authority, Department of Agriculture*

AMELIA W. TEJADA, Ph.D

INTRODUCTION

- Nowadays, harmonization of food standards within the country, among ASEAN countries, and even in the international arena is very important to promote fair trade practices and ensure food safety of the consumers

INTRODUCTION

- In the World Trade Organization (WTO), the CODEX Alimentarius serves as the reference points for these food standards along with other international organizations involved in Metrology, Standardization, Testing and Quality management (MSTQ).

Metrology, Standards, Testing, and Quality

- MSTQ infrastructure is in progress.
- It needs to be fast-tracked in order for the country to keep pace with its ASEAN neighbors.

MSTQ Working Group Consists of representatives from:

- National accrediting body
- National standardization bodies
- National metrology laboratory
- Bureaus on fisheries and aquatic resources
  and on food and drugs
- Government and private institutions conducting research and testing activities

Activities of MSTQ

- Upgrading of capabilities in a number of testing and analytical work
- Product certification
- HACCP assessment

National Metrology Laboratory

- In the Philippines, the NML of the Industrial Technology Development Institute, DOST (per National Metrology Act of 2003) establishes and disseminates national standards of units of measurements to local calibration laboratories. The service facilities have served 495 clients to date.

NML Philippines maintains five laboratories

1) mass, force, and pressure
2) length and engineering
3) viscosity, volume, density and flow
4) thermometry, hydrometry and photometry
5) electricity and frequency

Philippine Accreditation Office (PAO)

- PAO was created in May 18, 2009 under Executive Order 802 "Strengthening and Recognizing the PAO of DTI as the national accreditation body"
- Task of PAO : accredit inspection, testing and certifying bodies, and other bodies offering conformity assessment services needed by the country.

Composition of PAO

- DTI Secretary
- Representative of the Department of Science and Technology (DOST), Department of Health (DOH), Department of Agriculture (DA), Department of Environment and Natural Resources (DENR), Department of Public Works and Highways (DPWH), Department of Energy (DoE) and Department of National Defense (DND).
- Three representatives from private sectors

CAC & the World Trade Organization (WTO)

WTO: Administers multilateral agreements on trade

-Forum for trade negotiations

-Handles trade disputes

CODEX standards were identified as key reference points in the WTO "Agreement on the Application of Sanitary and Phytosanitary Standards (SPS) and Technical Barrier to Trade (TBT)"

Codex Alimentarius Commission

- **Protecting the health of consumers**
- **ensure fair practices in food trade**

". . . to guide and promote the elaboration and establishment of definitions and requirements for foods, to assist in their harmonization and, in doing so, to facilitate international trade".

http://www.codexalimentarius.net/

Codex Alimentarius Commission (CAC)

The CAC was founded in 1963 by FAO and WHO to develop standards/ guidelines and other documents for foods.

→ >180 Member States, representing 99% of the world's population

The SPS Agreement

recognizes, as the international reference, the standards, guidelines and recommendations established by **the Codex Alimentarius Commission**

- As long as a country employs these standards, its measures are presumed to be consistent with the provisions of the SPS Agreement

National CODEX Organization

- November 25, 2005 (DA-AO No. 01 S2005/DOH-AO No. 2005-0028)
- To provide the organizational link between the concerned government and non-government organizations whose activities affect the development and implementation of food safety and quality standards

Philippines National Codex

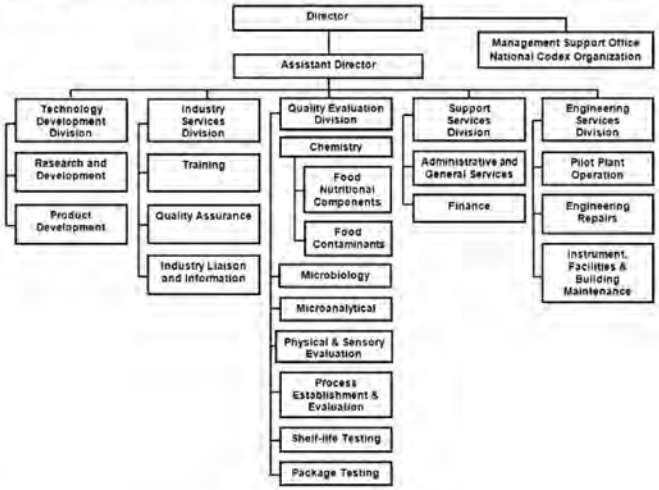
- serves as an advisory body on various issues arising from the work of CODEX Alimentarius Commission to ensure food safety and fair trade practices.
- composed of the Sec of Agriculture (chair) and the Dept of Health (Vice Chair) with members from government and non government institutions.
- about twenty four Technical Committees and subcommittees of the National Codex Organization on various agricultural commodities

- The Bureau of Agricultural and Fisheries Product Standards of the Dept. of Agriculture (BAFPS) is the Codex Contact Point in the country on all food standards that has national concern.
- The Food Development Center (FDC) houses the Management Support Office of the National Codex Organization(NCO). FDC is the Secretariat of NCO.

The FOOD DEVELOPMENT CENTER

FDC

Organizational Structure of FDC



QUALITY POLICY

- NFA-FDC aims to be a recognized center for appropriate technologies, training, product and process evaluation, inspection and standards development.
- NFA-FDC is committed to timely delivery of all products and services that consistently and reliably comply with customer requirements and applicable regulatory and statutory requirements.
- NFA-FDC is committed to the continual improvement of its quality management system.

SERVICES

- Product Testing Evaluation (Chem, Micro, physical and sensory, others)
- Plant Product Inspection and Certification
- Pilot Plant Scale Production
- Product and Process Development
- Training-e.g. HACCP, BPCS, Thermal processing Predictive Micro, Shelf life testing, Food packaging , QA, Filth analysis, food labels, others

Status of Food Safety at NFA-FDC, DA

- On 20 July 2009, the Secretary of Agriculture, under AO No.13, designated the Food Development Center of the National Food Authority (NFA), DA as the Official Laboratory for Contaminants Analysis in agricultural, fisheries and animal foods and food products.

Status at NFA-FDC, DA cont'd

- The Republic Act 8976 establishing the Philippine Fortification Program on rice and other foods where NFA is responsible for ensuring that rice dealers comply with the rice fortification program in the country
- FDC then, analyzes and certifies the iron content of iron rice premixed (IRP) and cooked iron fortified rice (IFR)

Status Cont'd

Rice- Iron Premixed

- Moisture Analysis (gravimetric method)
- Iron Content (gravimetric method)
- Physical characteristics
- Acceptability – Sensory Evaluation
- Packaging (stress test)

Status Cont'd

Chemical Contaminants

Heavy metals, Pesticide and veterinary drug residues, Antibiotics residues, melamine, acrylamide, PAH, Food additives (Vitamin A, Sulfite, Benzoic acid, Sorbic acid,Sodium Nitrite

Microbial Contaminants

Aflatoxins B and G , Salmonella, *S. aureus, L. Monocytogenes SalmonellaV, choleraV, parahaemolyticus,*

E,g.Calibration of Equipments

- Test weights-ITDI, TROEMNER-USA, Mettler- Toledo, Scientific Standards Services
  FPFI, Metrology lab.
- Micrometer,feeler gauge-ITDI
- Top loading balance,Vaccum oven, Incubator, furnace, thermohygrometer, dial caliper, pressure gauge–MIRDC-DOST
- pH meter- MettlerToledo
- Major equipments-AA Spectrometer, GC, HPLC, HPTLC, Elisa reader- External maintenance and calibration from sole distributor

FDC's Needs

- With the new developments at FDC, there is a need to strengthen the laboratory in accordance to ISO 17025 to build up its market confidence.
- Acquisition of highly sophisticated equipment such as LC MS/MS is in progress but needs special training.
- Measurement with high degree of sensitivity, accuracy to meet international standards.

Way Forward

- Continuous upgrading of facilities and training of personnel
- Continual Quality Management
  NFA-FDC maintains its certification to **ISO 9001:2000** to ensure its services to be at par with other foreign or local institutions providing the same services
- Participation in proficiency testing through inter-laboratory test comparisons

Way Forward Cont'd

- Regular calibration of the equipments
- Validation of method (including accuracy, precision)
- Traceability/uncertainty measurement of results
- Certification of the Quality Assurance Section of NFA-FDC to ISO/IEC 17021 for Hazard Analysis Critical Control Point (HACCP) certification activities

CONCLUSION

- Metrology, Standardization, Testing and Quality Management are tools in Food Safety.
- At FDC-NFA, developments on food safety has been the priority to cater the increasing needs of various Authority of the Department of Agriculture as well as the industries.

Conclusion cont'd

- FDC is developing, to its fullest , the capabilities of the Center to extend services in accordance to ISO 9000, ISO/IEC 17021 and ISO/IEC 17025 to build up the market confidence of the country.

Conclusion Cont'd

- In the Philippines, infrastructure and acquisition of highly sophisticated equipment is still in progress. It needs to be fast-tracked in order for the country to keep pace with its ASEAN neighbors as well as meet international standards.

Conclusion

- FDC needs the capacity building of analyst on all tests (MSTQ) necessary for tools in Food Safety to ensure compatibility with generally accepted conformity assurance and conformity assessment concepts.
- thereby, facilitate and promote acceptance of the calibration and test results between countries to avoid technical barriers to trade.

MSL Measurement Standards Laboratory of New Zealand

A collaborative approach to establish traceability in chemical measurement and food safety in New Zealand

Dr. Laly Samuel

Project Leader, Metrology in Chemistry and Biology

Measurement Standards Laboratory of New Zealand

MSL is New Zealand's national metrology institute operating within Industrial Research under the authority of the Measurement Standards Act 1992.

IRL

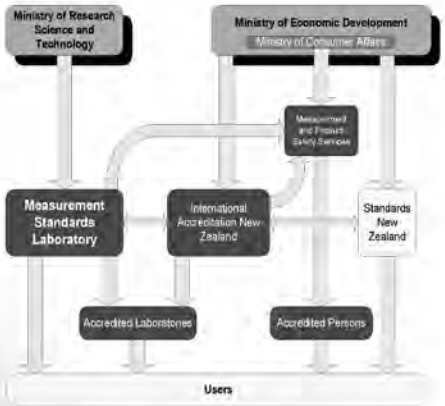
Measurement Standards Laboratory of New Zealand (MSL)

MSL New Zealand

- MSL is the National Metrology Institute and is responsible for the provision of measurement standards in New Zealand
- Our Team - Electricity, Length, Light, Mass, Pressure, Temperature, Humidity, Time and Frequency and Chem-bio
- Represents NZ at:
  - Asia Pacific Metrology Programme (APMP)
  - BIPM/CIPM

2

New Zealand Measurement System



Measurement and Product Safety Service (MAPSS)

- Operational Unit in the Ministry of Consumer Affairs
- Responsible for administration and enforcement of:
  - Trade Measurement System (Weights and Measures used for trade)
  - Safety of general consumer products
  - Monitoring of retail fuel quality
- Represents NZ at:
  - International Organisation of Legal Metrology (OIML)
  - Asia-Pacific Legal Metrology Forum (APLMF)
  - International Consumer Products Health and Safety Organisation

NZ Product Safety System

Product	Relevant organisation	Contact details
General consumer products	Measurement and Product Safety Service, Ministry of Consumer Affairs	www.mca.govt.nz
Products subject to a mandatory product safety standard or unsafe goods notice	Commerce Commission	www.comcom.govt.nz
Food	New Zealand Food Safety Authority (NZSFA)	www.nzfsa.govt.nz
Medicines and therapeutic goods	Ministry of Health	www.moh.govt.nz
Motor vehicles	Ministry of Transport Land Transport New Zealand	www.mot.govt.nz www.ltsa.govt.nz
Gas and electrical products	Energy Safety (part of the Ministry of Economic Development)	www.energysafety.govt.nz
Hazardous products and products used in the workplace	Occupational Safety and Health (a division of the Department of Labour)	www.osh.govt.nz
Hazardous substances and organisms	Environmental Risk Management the Authority (ERMA)	www.erma.govt.nz
Products used in building and construction	Department of Building and Housing	www.dbh.govt.nz

Importance of Agricultural measurements in New Zealand

International issues

Growers face chemicals ban

International Trade

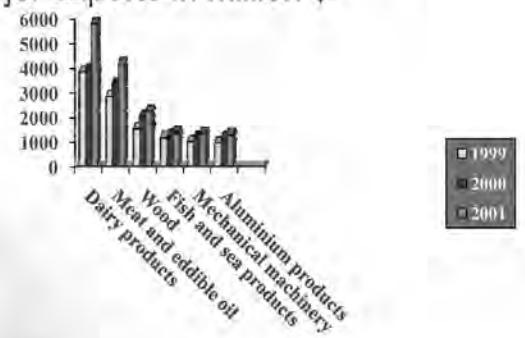
- International trade is vital for our primary production sector
- Removal and avoidance of potential barriers to trade is critical
- Border security is important to protect our flora and fauna
- Accurate measurements are key to our product safety
- Public Health and social well-being are important for our low population

Key Exports

- Dairy products
- Timber
- Meat
- Seafood
- Horticultural products (including organics)

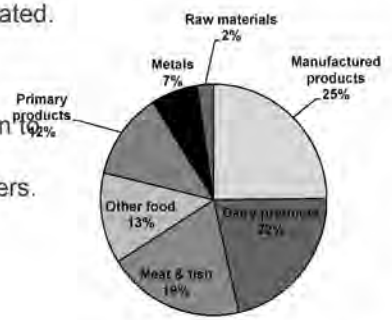
Exports by commodity

Major exports in million $



Economic impact

- Measurement activity.
  - All ~$800 million (NZ$) .
  - ~$120 million lab related.
- Exports.
  - $30 billion (2006).
  - Additional $1.2 billion to win from resolving technical trade barriers.



Market

- More than 90 per cent of all dairy products produced in New Zealand are exported.
- 90 per cent of all lamb, and 80 per cent of all beef, are exported.
- Market access matters - if New Zealand loses access to key markets, we all suffer."

Product safety and sustainability

- **Animal Welfare**
- GM – retailers and brand owners don't want it
- **Timber** –demand for certified product
- **Seafood** –key sustainability issue – demand for certified product
- **Dairy products** – NZ's clean green image definite selling point
- **Meat and fresh produce** – increasing demand for farm assurance schemes including environment and community welfare

Relevance to New Zealand

- About 527 accredited laboratories
- About 70% are dealing with chemical or biological products, and/or measurements
- Those accredited labs include testing laboratories, calibration laboratories, radiology services and inspection services

Present situation ....

- NZ laboratory's technical competence is accredited by International Accreditation New Zealand (IANZ)
- IANZ accreditation is common to most activities, and is increasingly relied on by regulators
- Accreditation programmes undertaken by IANZ are overseen by Accreditation Advisory Committee (AAC)
- Professional Advisory committee (PAC) are formed within IANZ to provide technical advice and review of specific areas of technology

Present Status

- MSL has an MoU with IANZ and advises through professional advisory committees (Metrology and Calibration, Chemistry Biology and Dairy)
- Some regulators utilising different paths
- Overseas regulators not using harmonised systems

Food

- **Food is big business in New Zealand. The food industry produces 23% of New Zealand's manufacturing GDP, and represents half the total value of merchandise exports.**
- **According to a recent Massey University study, *value-added food and beverage exports made up 54 per cent of NZ total food exports in 2004.***

Food measurement

- Food commodities from New Zealand are now exported to over 125 countries around the globe, with key markets being Australia, Japan, the United Kingdom, European Union and the USA.
- Traditionally these export markets have concentrated on raw commodities (fruit and vegetables) and finished products (cheese and butter)
- More recently, there has been increased attention given to the production of functional food ingredients and hence the separation, isolation and characterisation of these ingredients for export to the global market.
- The dairy industry of New Zealand (which accounts for over 50% of the export income for New Zealand) has embraced this change in focus by being pioneers in the utilisation of whey protein isolates, milk proteins, fats and additional components.

Cost effective way of implementation

- Virtual Institute for Metrology in Chemistry –
- Linking Nationally
- Linking Internationally

19

Virtual Institute for Metrology in Chemistry (VIMC)

- VIMC as the major source for dissemination of traceability in chemical and biological measurements.
- Work together to increase awareness of metrology in Chemistry and Biology among NZ measurement group.

20

New Strategy

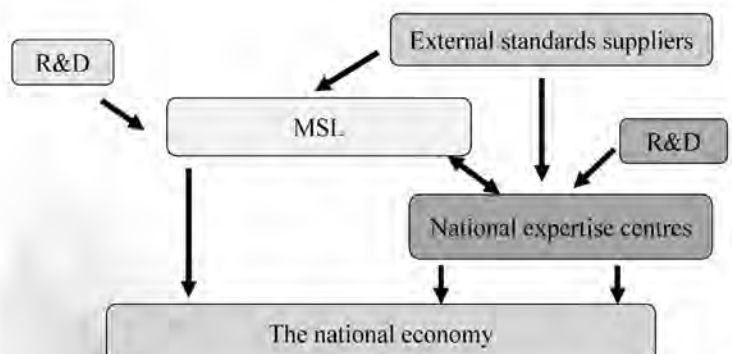
Virtual Institute for Metrology in Chemistry (VIMC)

What is this ?

- This is a network structure of existing arrangements and organizations without walls running by using available resources and facilities in New Zealand.
  - Bringing all expertise together
  - Bringing all laboratories together
- VIMC is the major source for dissemination of information related to traceability in chemistry.
- Provides all the services that a metrology institute could provide.
- Work together to increase awareness of metrology in Chemistry and Biology among NZ measurement group.

21

A Virtual Institute for Metrology in Chemistry



22

Characteristics of VIMC Partners

- True expertise in specialist area
  - appropriate depth
  - appropriate scope
- Willingness to work cooperatively within VIMC
- Metrological systems in place
- Strong linkages to user community

VIMC members

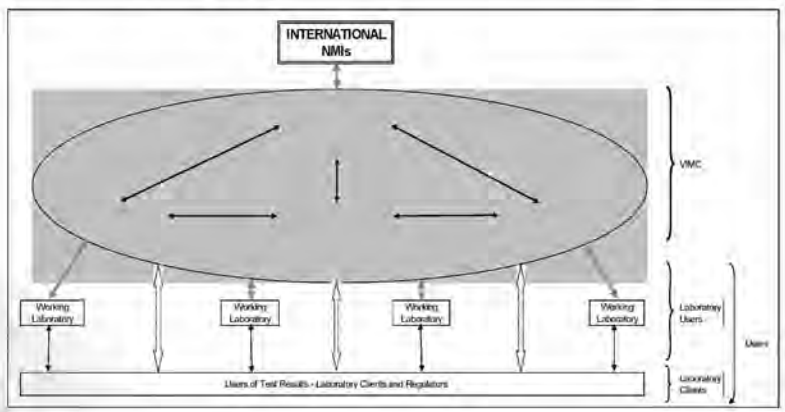
- MSL as the signatory
- Hill Laboratories
- AgriQuality
- Watercare
- ESR (Environmental Science and Research)
- IANZ (International accreditation NZ)
- About 70 members joined in the awareness group

Expectations of VIMC Partners

- Standards Development and Maintenance
  - long-term commitment
  - within VIMC, setting of priorities for new work
- Standards Dissemination to NZ community
  - proficiency testing schemes
  - reference materials
  - provide IANZ with assessors and advice
  - provide uncertainty training

Linkage to global standards
- representation of New Zealand at specialist international metrology forums
- participation in regional, global measurement comparisons

Relationships of the VIMC TO NMIs and New Zealand user groups



NZ specific problems

- Proficiency programmes
- National Standards Development
- Reference Method Development
- CRM Development

MSL Collaborative Projects under VIMC

- DNA reference standard project -ESR
- Value assigned PT project - AgriQuality
- Norovirus Quantification using LCMS –Crop and food, ESR
- Marine Toxin Reference standards - Cawthron Institute
- CRMs for Toxins in honey- IRL
- CRMs for colostrum based IgG - Cawthron

1. DNA reference standard project

- Development of two potential calibration standards for the PCR quantification of the microorganism *Campylobacter jejuni*.
- Development of PCR calibration standards
  - equivalent standard to those in other areas of chemistry
  - Calibration standards must be of known and documented composition
  - this must include demonstrated stability from time of characterisation until time of use.

Campylobacter

- Quantification of *C. jejuni* is of interest as:
  - it is a common cause of gastrointestinal illness and
  - is of public health significance in many countries and
  - is of particular concern in New Zealand

Table 1 showing recent recorded incident rates for six developed countries

Country	Rate (per 100,000)
New Zealand (2001)	**271.5**
England and Wales (1998)	**111**
Australia, excluding NSW (2000)	**107**
Denmark (1999)	**78**
Canada (1986~1998)	**39~54**
USA (2000)	**20.1**

Source of data: Lake et al. (2003).

31

2.Value assigned PT scheme

- To obtain reference samples for testing the chemical residues of 1080, paraquat, diaquat and problematic anions (nitrates, nitrites and phosphates) in potable water
  - specifically samples containing various pesticides 1080 (sodiumfluoroacetate), and herbicides (diuron, triclopyr, 2,4-dichlorophenoxy acetic acid(2,4-D), benzo[a]pyrene and tri halo methanes (chloroform, bromoform, dibromo-chloromethane, bromodichloromethane) at concentration close to the maximum allowable values of potable water requirement by the NZFSA
- storage of the reference samples
- effect of transport on the stability of the reference samples
- An assessment on the development of a limited proficiency programme for New Zealand laboratories to support the systems for efficient handling and analysing of trace contaminants as compared to the high costs programmes from traditional procedures.

32

3.Norovirus project -ESR

- Aim of the project was to develop an ID-LCMS method to detect and quantify norovirus from environmental samples in collaboration with ESR and Crop and Food Research.
- An LC-MS/MS Method was developed and validated to detect and identify MS-2 phage virus in environmental water samples and to evaluate the method performance and fitness for purpose to screen environmental samples.
- ESR provided digested phage protein purified from gel for method validation, initially prepared according to the ESR standard protocol with modifications as required.
- ESR provided river water samples prepared according to the selected protocol(s) to improve the limits of detection and specificity of the method. The samples were seeded with FRNA phage and an isotopically labelled peptide (provided by MSL) prior to processing.

33

Norovirus project – CFR

- To set up instrument conditions and determine instrument detection limit with tryptic digests of standard reference material as well as pure and matrix MS-2 phage samples
- Develop and optimise an LC-MS/MS method to detect and quantify MS-2 phage in environmental samples
- Validate the method for MS-2 phage proteins based on the developed methodology
- Validate ID-LC-MS/MS method to detect norovirus from environmental samples

34

4.Shellfish toxin CRMs

MEDIA MONITORS

Food Technology in NZ
October, 2008
Page: 6
Section: General News
Region: National Circulation: 2,883
Type: Magazines Trade
Size: 289.82 sq.cms.
Published: Monthly

Brief: IRL(W)
Page 1 of 2

New Zealand leads drive for new standard in shellfish testing

A precise and powerful technique for detecting toxins in shellfish is a step closer to being accepted as a worldwide standard after validation by IRL's Measurement Standards Laboratory (MSL) and endorsement from the United States Food and Drug Administration (FDA).

Neurotoxin Shellfish poisoning (NSP)

- Neurotoxin Shellfish poisoning or NSP is a syndrome caused by the consumption of shellfish contaminated with a class of natural toxins called brevetoxins
- Consumers are protected from NSP by the regular testing of shellfish and since the 1950's this testing has been via a mouse bioassay
- very slow to perform (shellfish was often already eaten by the time a positive result was returned)
- The industry was also concerned that animal rights pressures, particularly in Europe, would see the test banned
- The NSP mouse bioassay protocol is unethical (it requires 5 mice of specified body weight) and is not validated
- The mouse bioassay protocol is also labourious and requires the use of large amounts of diethyl ether which is dangerous and difficult to handle in the laboratory

Shellfish Toxins

- Aim - Quantification of five key brevetoxins in a single lab validation (SLV) and
- Incorporate brevetoxins into a multi-toxin LC-MS method for a wide range of lipophilic marine toxins from several classes and algal sources.

Method

- This method was approved by NZFSA in 2004
- USFDA is collaborating with Cawthron Institute to develop this method
- CRM will be available through Institute of Marine Biosciences (IMB), Canada

Method

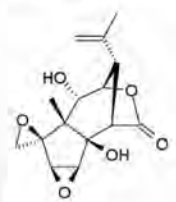
- NSP is screened using an LCMS method for two brevetoxins (PbTx-2 and PbTx-3) when either compound is detected the regulatory mouse bioassay is performed.
- The new method tests a total of 29 toxins in a single LCMS run including two brevetoxins (BTX-B2 and desoxy BTX-B2).

5.MSL pH standard project

- Developing Harned cell based national pH reference standard
  - Develop a traceability chain for pH measurement in NZ

6. NZ honey Problem

- Tutu is a native plant species found throughout New Zealand.
- Vine hopper insects feed on the sap of the tutu plant and produce honeydew containing tutin and hyenanchin.



NZ Specific Problem

43

Tutin

- Toxic honey is produced as a result of bees gathering this honeydew.
- Risk managed through guidance to beekeepers
  - but 22 reported cases of poisoning from tutin in honey in March 2008

44

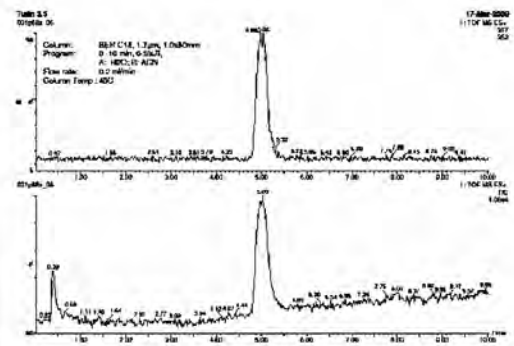
Response

- Research to define acceptable levels
  - interim limit set of 0.1mg/kg
  - contracted locally for purified tutin and hyenanchin samples
  - 3 laboratories accredited for testing, LCMS
- MSL developing reference standard
  - international sourcing for higher purity
  - accessing various institutes for capabilities
  - intend IDMS for validation

45

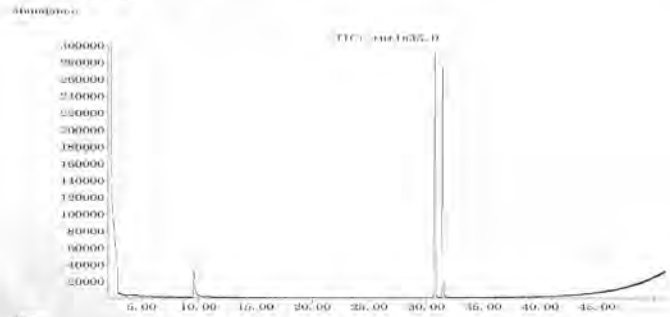
LC-MS of Tutin

1. Chromatogram for ion 317 (M^+ + Na)
2. TIC



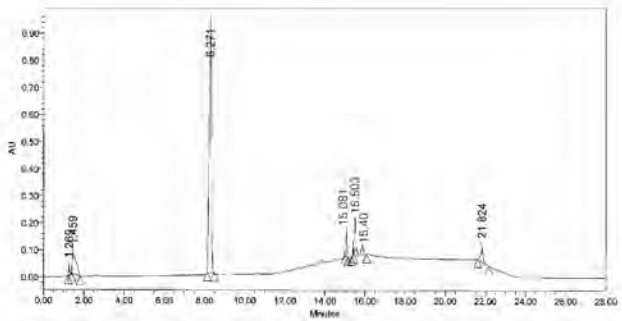
46

GC-MS TIC of tutin in EtOH with injector temperature at 250°C



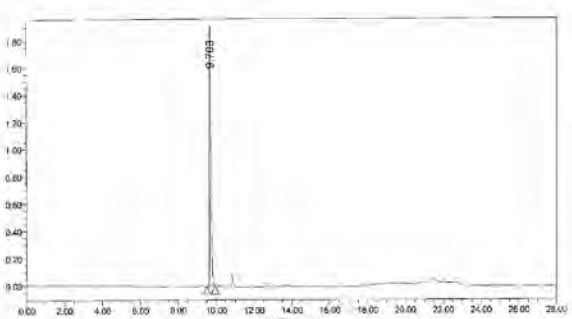
47

UPLC of tutin in EtOH with solvent programme 1 and detection at 195nm



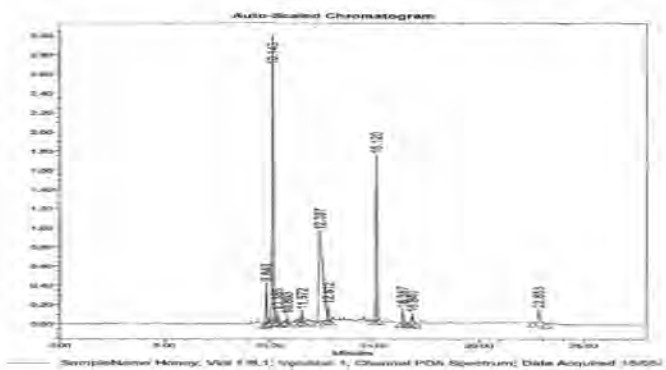
48

UPLC of hyenanchin in water with solvent programme 2 and detection at 200nm



49

UPLC of EtOAc extract of toxic honey



50

7. Colostrum based IgG CRM

- Colostrum is an important specialty product for the NZ dairy industry.
- Colostrum contains a range of bioactive compounds but the most active group is the immunoglobulins, dominated by IgG in bovine colostrum.
- IgG content (5%~40%) is the key parameter for colostrum quality and several analytical techniques are available for quantitative determination.
- These methods all require calibration and IgG from bovine plasma (Sigma-Aldrich) is the most widely used standard material at present.
- Sigma standard is plasma derived and is principally the IgGa variant whereas colostrum principally contains the IgGb variant which has some differences in properties

IgG CRM

- Looking for 99%~100% pure monomer sample
- Project is still in the development stage

8. Uncertainty Training

- We provide uncertainty training course to regulatory agencies and field laboratories

Summary

- NZ is adopted a collaborative approach to demonstrate traceability
- Specific NZ issues are taken into consideration
- Public health and food safety are our prime importance in the selection of chemical metrology projects
- New measurement methods are developed and validated

Acknowledgements

- **Stephen O'Brien***

Co-author

Manager, Measurement and Product Safety Service, Ministry of Consumer Affairs, New Zealand

- APEC

  Travel funding

- VIMC members and project collaborators

55

Thank You

56

Asia-Pacific Economic Cooperation

APLMF

APEC/APLMF

Workshops on Metrology in Agriculture Products, Food Safety and Product Safety

September 23 – 25th ,2009

Introduction

Overview of Measurement Standards in Agriculture facilities in Papua New Guinea

- Presentation By

Edna Egu

Metrologist– Metrology Division

NISIT

PNG – National Institute of Standards and Industrial Technology

- Responsible for overseeing to standardization and conformance activities in Papua New Guinea.

In summary, NISIT is obligated under NISIT Act-1993 to perform the following:

- Standards Development and Publication
- Standards Information Dissemination and Sales of Standards or Publications
- Calibration, Verification and Testing of Measuring Equipment and Artifacts
- Laboratory Accreditation
- Management System Certification
- Conduct Professional Training programs on standardization and quality assurance

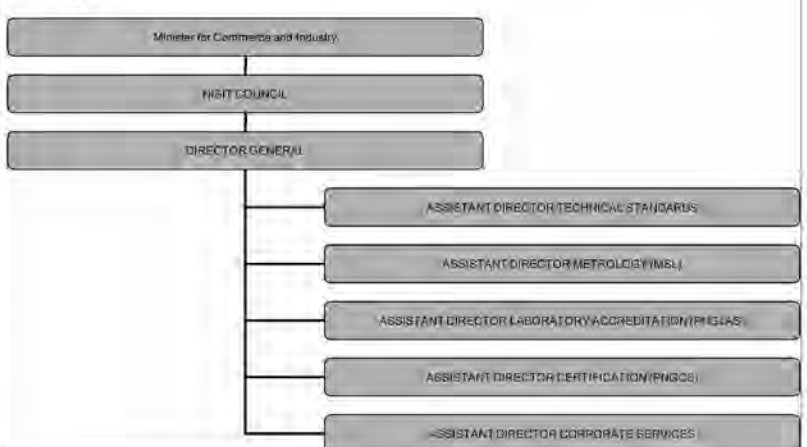
PNG – National Institute of Standards and Industrial Technology

As per the NISIT Act 1993, the roles and objectives:

- Ensure the NISIT becomes the National Technical Infrastructure that is essential to provide technical support to Commerce, Trade and Industrial Developments on matters of standardization, quality assurance and conformity assessment.
- To provide technical support to effect the enforcement of government technical regulations and standards pertaining to Quality, safety, health environmental protection and consumer protection.

NISIT is mandated to have legal custody and maintain all the National Reference and Physical Measurement Standards in Papua New Guinea.

Standards and Conformance Infrastructure (National Institute of Standards & Industrial Technology)



Measurement Standards Laboratory (MSL)

•Metrology Division is the national body in charge of Legal and Physical Metrology in PNG.

•The Measurement Standards Laboratory in its current capacity and capability building is responsible for the provision of Calibration and Verification Services in Papua New Guinea.

The services offered by MSL to date include:

- Calibration Services
- Verification Services
- Training Services
- Advisory & Consultation Services (Measurement)

Measurement Standards Laboratory

CALIBRATION AND VERIFICATION SERVICES OF MSL:

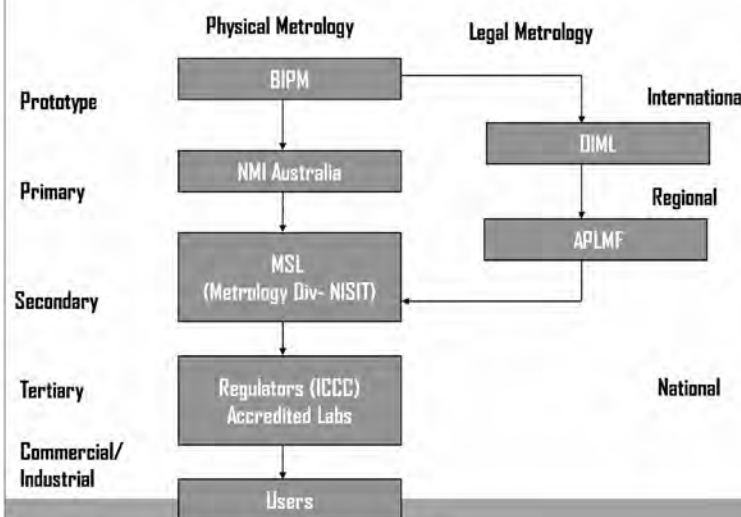
- MASS —— eg. Weights, Balances, NAWI
- VOLUME CAPACITY —— eg. Povers, flow-meters
- TEMPERATURE —— eg. Mercury in glass, Ovens
- DENSITY —— eg. Hydrometers, Density meters
- TORQUE —— eg. Torque wrenches
- FORCE —— eg. Force Machines, Load cells
- PRESSURE —— Pressure Gauges, Safety Valves
- LENGTH —— Vernier Calipers, micrometers

➢ELECTRICAL——Undergoing construction

Measurement Standards Laboratory

Measurement Standards Laboratory

Traceability of Measurement



Metrology and its Contribution to the Agriculture Sector.

Measurement Standards Laboratory	→	AGRICULTURE RESEARCH
Measurement Standards Laboratory	→	PRODUCTION
Measurement Standards Laboratory & Trade Measurement Branch, ICCC	→	TRANSACTION

MEASUREMENT STANDARD FACILITY

• Current calibration services MSL provides to the Agricultural Sector to assist in removing technical barriers to trade internationally:

➢Balances, Masses, Density hydrometers ,Temperature sensors & Ovens etc.

➢ Moisture analyzers (Mettler Toledo, HB43-S)

-Used widely in all testing laboratories, agriculture research station, food manufacturing plants and commodity boards etc.
-Provide Adjustments for its weight measurement on Moisture analyzers using instruction manual method.
-No appropriate establishment of calibration service on the instrument.

• CONTRAINS!
➢Technical Expertise
➢ Appropriate Measurement Standards etc.

Measurement Standards Laboratory (MSL) WAY FORWARD!

• Looking at Agricultural Sectors.

——Needs improvement and development
➢Such as control and coordination of CRM's/RM's used in chemical testing Labs
——Begun a National Survey for Chemical Testing Laboratories
➢Assess the needs and prioritize the servicing the Chem Labs in PNG that certify food products and safety there of
➢Establishment of Chemical Metrology Infrastructure and measurement traceability
✓ Help support greater market access for our Domestic and International Food Market and most importantly Health of its people.

-Metrology Division Maintains a strong course in building appropriate metrological infrastructure to support this sector for years to come.

THE END !

THANK YOU FOR YOUR ATTENTION.

15 aniversario CENAM
CENTRO NACIONAL DE METROLOGÍA

National Metrology Infrastructure for Food Safety in Mexico

APEC/APLMF Workshop on Metrology in Agricultural Products, Food Safety, and Product Safety
23-25 Sep 2009, Ho Chi Minh city, Viet Nam
Norma Gonzalez

Presentation outline

- Metrological infrastructure
- Proposal to support the national system for food safety
- Traceable measurement infrastructure
- Other way to support our national system
- Considerations

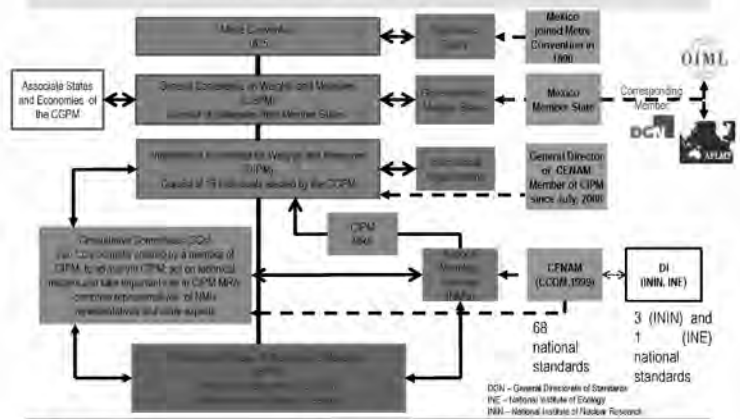
Metrology

- Is the science of measurement and its anplication

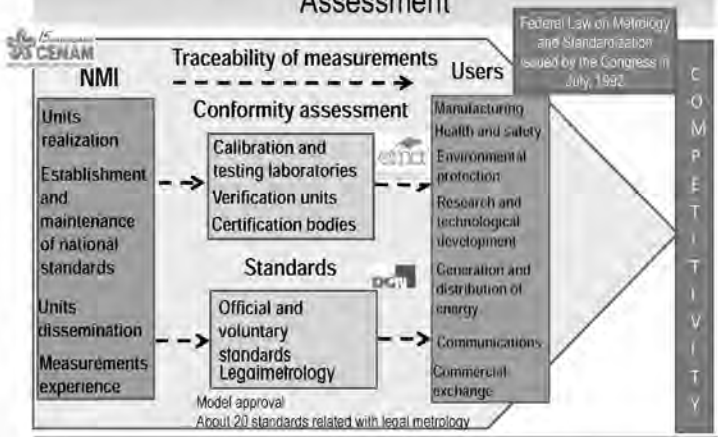
- To ensure all measurements are reliable and equivalents

12 TLCs with 42 countries

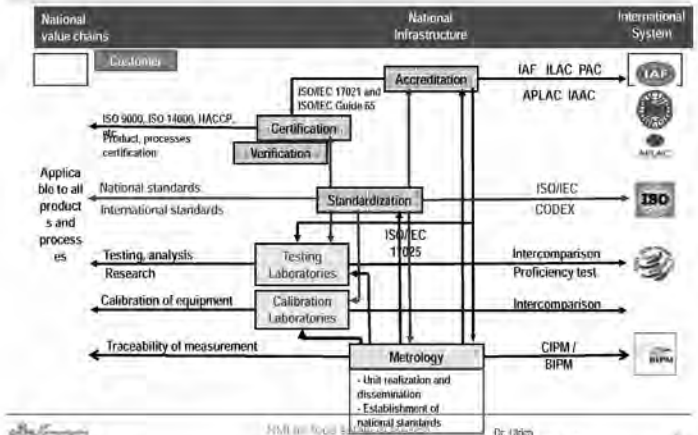
Metrology Infrastructure



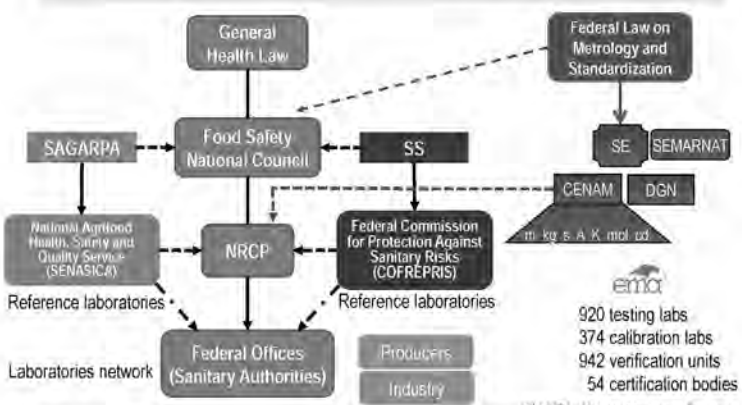
Metrology, Standardization and Conformity Assessment



National Infrastructure

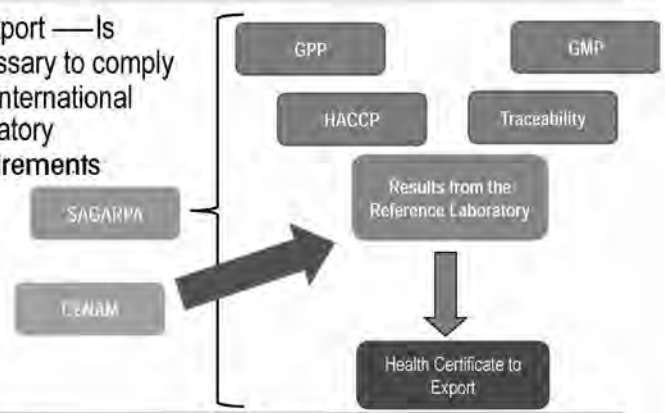


Proposal of a National Infrastructure for Food Safety

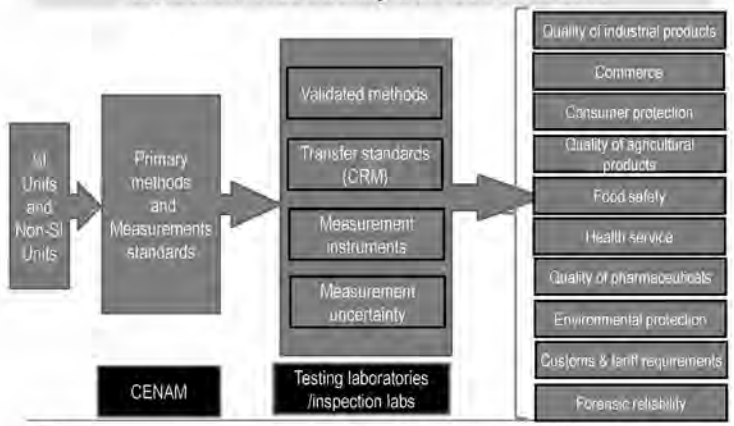


Requirements to Export

- To export —Is necessary to comply with international regulatory requirements



National Chemical Measurement Infrastructure to Establish Traceability of Measurements

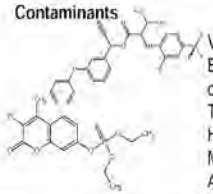


CRM Needs

Pharmaceuticals

Nutritional constituents
Fat
Protein
Ash
Moisture
Minerals

Contaminants



Veterinary drugs
Environmental contaminants
Toxins
Hormones
Metals
Antibiotics
OP, ON, OC, Py

Physical standards

Calibrants
Different matrices:
- Vegetables/Fruits
- Meat: poultry, beef, pork, game, equidae, goat, sheep
- Fishing and aquaculture products
- Eggs
- Milk
- Honey
- Grains
- Pharmaceuticals

Standardization

International Standards

National Standards

NOM (Mexican official standards)

NMX (Mexican standards)

DGN

NOM – SAGARPA		150
	Harmonized	1
	SS	191
	Harmonized	16
NMX – Food		576
	Harmonized	67
	Agricultural	340
	Harmonized	0

December, 2007

SEMARNAT | SECTUR | SALUD | STPS | SAGARPA | SE | SCT | SENER | SEDESOL | SEGOB | SSP | SEP

International Collaboration within APEC Region

Interlaboratory comparison——Determination of pesticides residues in Chinese Cabbage

- Objectives of study
  - Conduction of a comparative study, to identify existing measurement capability in safety and quality of food exports.
  - Workshop to present the study results as well as to conduct cause-effect analysis, to discuss on action plans of capability building for each economy, by defining activities to be incorporated in a project aimed at developing fundamental capability of chemical metrology in each APEC member economy.
- Participants
  - Analytical laboratories at inspection level, in collaboration with an NMI

Sample Materials

- Materials provided by KRISS
- Matrix: Chinese Cabbage (Freeze-dried powder)
- Analytes: Diazinon and Chlorpyrifos
  - —— Representatives of Organophosphorous Pesticides
  - —— 0.2 to 20 μg/g on dry mass basis (0.01 to 1 μg/g on raw material basis)

Participants

- Number of participants: 16 laboratories from 11 countries
  - —— 2 NMIs
  - —— 14 field laboratories

*4 laboratories collaborated with their NMIs, 2 of them were Mexican labs and collaborated with CENAM

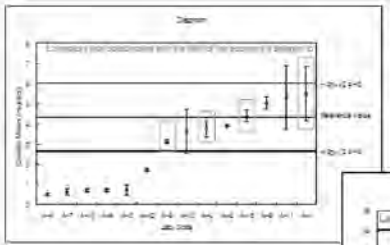
Study Scheme

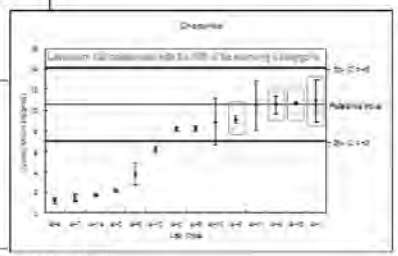
- Method to be Applied by Participants
  - —— Method regularly used in the laboratory for Food inspection
  - —— Validation/verification of the method in collaboration with a NMI (or by using CRMs) was recommended
- Measurement Scheme
  - —— Four measurements per day and repeated for three different days, total of 12 measurement (within-day repeatability + among-days reproducibility)
- Reporting Results
  - —— Separate results for each of three days with uncertainty
  - —— Summary of results of three days
  - —— Uncertainty budgeting (in collaboration with a NMI)
  - —— Details of analytical method
  - —— Calibration method, CRMs if used
- KRISS certified value was used as reference values of this intercomparison study

Information of Laboratories

Field Laboratories	NMIs
Calibration solution was prepared in house	CIPM CCQM member or observer
Use of manufacturer chemical purities except two laboratories that use purity assay results provided by the NMI	Participating in CCQM intercomparisons
GC, GC/MS, LC/MS/MS	Used an higher-order method (IDMS method)
They validated/verified their methods as is required by their QS	Laboratory is accredited by Mexican Accreditation Entity (ema) in conformance with ISO/IEC 17025: 2005 (NMX-EC-17025-IMNC-2006).
All laboratories except one were accredited based on ISO/IEC 17025	
All laboratories reported that they participate in external PTs	Participate in PTs at least once per four year accreditation period
Mexican laboratory used GC-PFPD, internal standard method and substances purity were evaluated by CENAM	CENAM gave support to estimate the uncertainty of measurements

Results





- Laboratories that collaborated with NMIs of their economy showed better performance than those that did not

Summary of Results and Conclusions

- All participating laboratories are accredited based on ISO/IEC 17025 (Except two laboratories that are currently under assess for ISO/IEC 17025 accreditation).
- The reference values of this study certified by KRISS are supported by results of two NMIs.
- Within- and among-day(s) repeatability and reproducibility are very good for all participants. However, many laboratories' results showed large biases from the reference values.
- *z*-scores (based on the standard deviation estimated by Horwitz model) of many laboratories exceed ± 3, indicating that they have uncorrected bias sources.
- Laboratories that collaborated with NMIs showed *z*-score within ±3, indicating that collaboration with NMI can help reducing bias sources and making the results traceable to SI.
- Implementation of quality system based on ISO/IEC 17025 itself is not enough to make measurement results of a laboratory successful. Laboratories have to implement strategy to fulfill and maintain the following technical requirements in ISO/IEC 17025.
  - Validation of methods
  - Estimation of uncertainty of measurement
  - Measurement traceability

Final Considerations

- Food and agricultural products are important field for Mexico
- It is necessary to establish a national infrastructure for food safety and agricultural products to get traceable measurements
- In this process is necessary the participation of different parties: Government, industry, laboratories, NMI...
- To define the mechanism to develop CRM for legal use
- There is still several activities to do...

Thank you very much

Moisture Adjustment of Reference Samples for the Development of Calibration Curves and Evaluation of Grain Moisture Meters

1. **Objectives**
2. **Collection and Preconditioning of Grain Reference Samples**
3. **Arrangement of Moisture Content**
4. **Homogenization and Low Temperature Storage**
5. **Precautions for Use**

1. Objectives

- To describe procedures of preconditioning for adjusting the moisture content of reference samples

Samples are to be used for
(1) development of calibration curves of grain moisture meters and
(2) verification of commercial grain moisture meters.

2. Collection and Preconditioning of Grain Reference Samples (1)

Usually minimum number of samples are prepared.
(1) Requirements for reference samples
① Cover more than 70% of the varieties.
② Cover more than 70% of growing places.
③ Be harvested within one year.
④ Circulation of grain in the market be taken into account.
⑤ Kind, variety, type, place of sampling, place of product and year of harvest be recorded.
⑥ More than 4 times of the minimum weight required be collected.
⑦ Be collected immediately after harvesting.

2. Collection and Preconditioning of Grain Reference Samples (2)

(2) Preliminary Procedures for Conditioning
Get rid of foreign substances, check original moisture content and divide for measurements and storage.

① Husker and sieve.
② Hands and sieve.
③ Pick out foreign substances.
④ Original moisture content is measured by three or five times average.
⑤ Mixing well and equally divided.

3. Arrangement of Moisture Content (1)

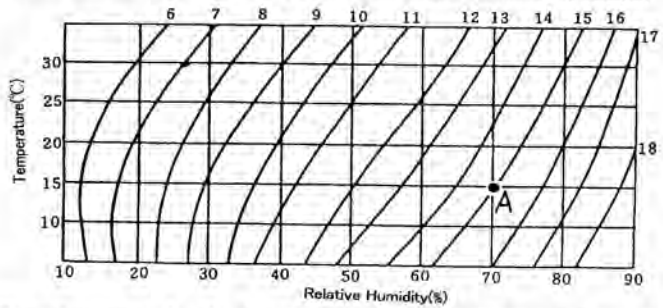
Development and evaluation of wide range moisture content reference samples

(1) Adjustment of Moisture Content by Drying

① Check the original moisture content
② Dry samples (in the room or by the oven)
③ Check moisture content by weighing
④ Mix sample well to homogenize
⑤ Put data label

Drying Process on Paddy

Equilibrium Moisture Content of Paddy under Certain Ambient Thermo-Humid Condition



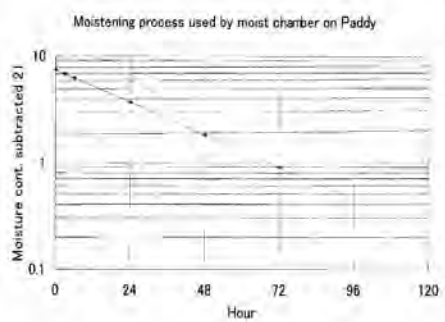
The moisture content of paddy set at 15℃ and 70%rh, which is revealed as "A", gradually move to 15%.

3. Arrangement of Moisture Content (2)

(2) Adjustment of Moisture Content by Moistening

① Check original moisture content
② Moisten samples by a moist bath
③ Elevate moisture content up to 2% higher than the target by checking the weight
④ Dry sample slightly by an oven
⑤ Mix sample well to make homogenize
⑥ Put data label

Moistening Process

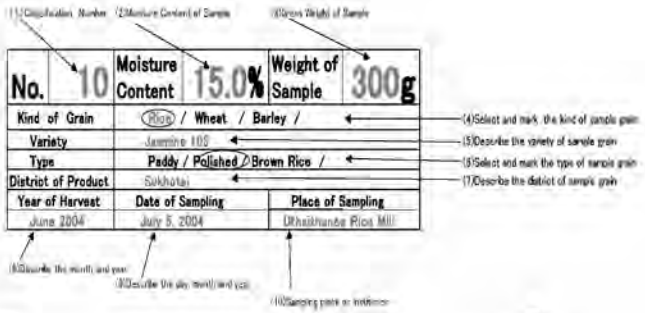


The moistening process is ruled by the exponential phenomenon.

The moisture content is subtracted 2%.

Sample information (label)

An Example of label description



4. Homogenization and Low Temperature Storage

- Minimize dispersion of the moisture content and keep the sample quality.

(1) Homogenization

(2) Low temperature storage

4. 1 Homogenization

To minimize the dispersion among grain kernels of the samples by diffusion of water molecule.

Water molecules are exchanged among grain via ambient water vapor.

It is necessary to seal the bag containing a sample.

4. 2 Low Temperature Storage

The lower temperature the quality of the samples is stored, the better the quality is obtained.

Freezing should be avoided since the ice crystal will damage the grain tissue and will change the physical properties.

It is impossible to maintain the quality of sample forever even if they are stored at 5℃.

Moisture
Adjustment

5. Precautions for Use

When a sample stored for a long time at a low temperature is to be used for the measurement of moisture content at room temperature, the sample bag should not be opened immediately after taking the sample out of the refrigerator.

Keep the sample bag at the room temperature for 2 to 3 hours without opening it.

13

International
Standard

ISO 712

Cereals and cereal products ——
Determination of moisture content ——
Routine reference method

1. **Definition of moisture content**
   Loss in mass, expressed as a percentage, undergone by the sample under the condition specified in this standard
2. **Specifications of apparatus and conditions for this standard**
   Analytical balance, Grinding mill, Metal dish, Oven, Desiccator
3. **Preparation of samples**
   Grinding to adjust particle size, Pre-conditioning
4. **Procedures**
   Dry for 2h at (130±3)°C. Weight to 1mg
5. **Calculation and expression of results**

1

ISO 712

1. Definition of moisture content

- Loss in mass, expressed as a percentage, undergone by the product under the conditions specified in this standard
- Test portion is dried for 2h at (130±3)°C
- Laboratory apparatus
  Analytical balance, Grinding mill, Metal dish, Oven, Desiccator

2

2. Specification of apparatus

1) Analytical balance
   Capable of weighing to an accuracy of ±1mg
2) Grinding mill
   Irrelevant to moisture, easy to clean, without heat, homogeneous, at 20 °C and 40% to 70% R.H.
3) Metal dish
   Non-corrodible, tight-fitting, an effective surface area
4) Oven
   Constant and stable for 2h at(130±3)°C
5) Desiccator
   Containing an effective desiccant

3. Preparation of test sample (1) grinding

Table 1 — Particle size distribution of products not requiring grinding

Particle size (mm)	Proportion (%)
≤1.7(1.8)[a]	100
>1.0(1.0)[b]	10
<0.5(0.56)[a]	50

a Sieve through which this particle size passes.

b Sieve through which this particle size does not pass.

- Products having the particle size distribution given in Table 1 do not need to be ground before the determination

3. Preparation of test sample (2-1) pre-conditioning

- If the sample does not have the particle size characteristics specified in Table 1, it shall be ground either without pre-conditioning or with pre-conditioning

1) Grinding products without pre-conditioning when its moisture content is between 7% and 15%
2) Products shall be pre-conditioned so as to bring their moisture content to between 7% and 15% if possible between 9% and 15% before grinding

3. Preparation of test sample (2-2) pre-conditioning

- If the moisture content is in excess of 15%,
  1) weigh, to 1mg, provide the test sample slightly greater than 5g on each sample,
  2) carry out a pre-drying operation, at 130℃±3℃.
  3) except that the time of heating shall be 7min to 10min,
  4) the cooling of pre-drying sample to laboratory temperature shall be carried out with dish uncovered, for at least 2h.
- If the moisture content is less than 7%, carry out a pre-wetting operation in suitable atmosphere until a moisture content within the limits.

ISO 712

4. Procedure

1) Number of determination

More than two determinations are required when the absolute difference between the two results is more than the repeatability limit r.

2) Test portion

In case of products to be ground, provide a test sample slightly greater than 5g before grinding, and weigh all the grindings obtained to 1mg.

3) Drying

Do not open the oven door during drying.

Place the open dish containing the test portion together with the lid, in the oven and leave for 120min ±5min from the moment when the oven temperature is again 130°C±3°C.

4) Weighing

When the dish has cooled to laboratory temperature, weigh it to the nearest 1mg.

ISO 712

5. Calculation and expression of results

- The moisture content, m_x, expressed as percentage by mass of products as received, is given by the following equations.

  1) Without pre-conditioning

  $$m_x = (1 - w_1/w_0) \times 100\%$$

  where w_0 and w_1 are the weights, in grams, of the test portion and the dried test portion, respectively.

  2) With pre-conditioning

  $$m_x = \{1 - (w_1 w_3)/(w_0 w_2)\} \times 100\%$$

  where w_2 and w_3 are the weights of before and after pre-conditioned, respectively.

- The result is the arithmetic mean of two single determinations which meet the repeatability requirement. It is expressed to two decimal places.

ISO 712

6. Precision

1) Interlaboratory test
When the interlaboratory tests are carried out, the evaluation is concerned with the following affairs of the precision.

2) Repeatability, r;
obtained using the same method on identical test material
in the same laboratory by the same operator
using the same equipment
carried out within a short interval of time;
$r = 0.013m - 0.06$, where m is the mean of the two test results.

3) Reproducibility, r_d;
obtained using the same method on identical test material
in different laboratories with different operators using different equipment;
$r_d \leq 0.59\%$

International Standard

ISO 7700

Calibration of moisture meters
Part 1: Moisture meters for cereals

1. **Calibration of moisture meters by reference samples**
2. **Selection and cleaning of samples**
3. **Conditioning of samples**
   - **Quantity of distilled water to adjust moisture content**
   - **Shaking**
4. **Calibration**
   - **Determination of moisture content by ISO 712**
   - **Calibration of moisture meters**
5. **Expression of results**

1. Introduction

- For stable samples in ideal measuring conditions, calibration of moisture meters would be satisfactory.
- But actual results can be affected by cultivation, ripeness, humidity, temperature, harvesting, transportation and impurities, particularly for cereals of high moisture content.

2. Scope

- ISO7700 specifies a method for checking the calibration of grain moisture meters in service, by checking some values or a range covering all the values for which the moisture meter is used.

3. Apparatus

(1) Storage

- **Sample bag**
  - **Airtight, thick enough**
  - **Clean**
- **Refrigerator**
  - **Constant temperature at 5deg**
  - **Uniform temperature**

ISO 7700

3. Apparatus

(2) Reference method

- Analytical balance
- Grinding mill
- Metal dish
- Constant-temperature oven
- Desiccator
  (see ISO 712)

ISO 7700

3. Apparatus

(3) Cleaning samples

- Manual sieves
  Slot apertures of 1.80, 2.00 and 2.24 mm width
  Round holes of 4.50mm diameter
- Mechanical separator

ISO 7700

4. Procedures

(1) Selection and cleaning samples

- Cleaning the samples by removing undersize material and shrivelled grains.
- Manual sieving using appropriate sieves and removing larger impurities by hand or using a mechanical separator.

ISO 7700

4. Procedures

(2) Preparation of test samples

- Procedures for checking several moisture contents
  1) Prepare more than 10 samples.
     approximately 1kg each
     moisture contents between 10 to 25%
  2) Choose moisture contents where the moisture meters are commonly used.
  3) Drying or wetting is usually allowed unless instrument characteristics are affected.

4. Procedures

(3) Conditioning test samples

To arrange moisture contents with equal intervals

1) Dry samples very gradually at a temperature lower than 30° C, ventilating if necessary.
2) Wet samples and calculate moisture content as follows:

$$\Delta w = w_0 \times (m_x - m_0)/(100 - m_x)$$

where

Δw is the increase weight during wetting;

w_0 and m_0 are original weights and moisture content, respectively

m_x is the desired moisture content.

3) In all cases, the sample bags should be kept at a temperature of approximately 5 °C, for example in a refrigerator.

4. Procedures

(4) Checking the moisture meter

- After conditioning, take out the sample bags from the refrigerator at least 16h (usually overnight) before the test, to make them thermal equilibrium with the moisture meter.

1) Procedures for checking the moisture content of a sample
Reject samples emitting odour of fermentation or molding.

Perform the following operations:
① Routine reference method
② Carry out four successive measurements by the moisture meter.
③ Repeat ①.

2) Procedures for checking the range of moisture contents

On each test sample, carry out the same operations specified in 1) .

That is, repeat the operations at an interval of 24h using the same test samples for cereals other than maize.

5. Expression of results

(1) Procedures for checking a moisture content

- For each test sample, the following values are available :

1) Average of two results obtained by the routine reference method, <*x*>.

Maximum Permissible Error (MPE) of each *x*;

0.15% —— without pre-conditioning,

0.20% —— with pre-conditioning.

Exceeding MPE, repeat the test.

2) Each measurement by the moisture meter, *y*.

- Each value $y - \langle x \rangle$ shall be less than MPE such as those specified in the above 1).

5. Expression of results

(2) Procedures for checking a range of moisture contents

- Deal separately with the two series of measurements carried out at an interval of 24h and compare them.
- For each measurements, the following values are available:

1) Two results obtained by the routine reference method, x.
The maximum permitted errors (abbr. in "m.p.e.") are
0.15%—— in the case without pre-conditioning,
0.20%——in the case with pre-conditioning.
If the error excess the m.p.e, repeat the test.
2) Four measurements carried out with the moisture meter, y.

- The value $|m(y)-m(x)|$ shall be less than the m.p.e. such as those specified in the annex. Otherwise, repeat the measurements on the corresponding test sample.

Annex

Maximum permitted errors

- A.1 Class I moisture meters

0.8 (absolute) for a moisture content, $m(x)$, less than 10%;
0.4 (absolute)+4%(relative) for moisture content, $m(x)$, greater than 10%.

- A.2 Class II moisture meters

0.9 (absolute) for a moisture content, $m(x)$, less then 10%;
0.4 (absolute)+5%(relative) for moisture content, $m(x)$, greater than 10%.

Moisture Measurement in Agricultural products
&
Moisture Testers

Contents

1. Scope

2. Air oven method (105 degrees centigrade air oven method)

3. Factors affecting the precision of moisture measurement by the air oven methods

4. Electronic moisture testers for grain

5. Resistance type moisture tester

6. Capacitance type moisture tester

7. Accuracy check for moisture testers (Riceter J/m, PM-400)

Scope

Classification of the methods employed to determine the moisture content of agricultural products

1. Oven methods
2. Chemical methods
3. Distillation methods
4. Others
   * Electronic moisture testers
   * Infrared moisture determination balances
   * Microwave methods
   * Near Infrared methods (Transmittance and Reflectance type)

Air oven methods

Products \ Organization	Japan	USDA	ISO	AOAC	ASAE
Cereal grain	105℃ 5hrs	130℃ 1hr	130℃ 2hrs	135℃ 2hrs	103℃ 20hrs for barley 19hrs for wheat
Beans	105℃ 5hrs	130℃ 1hr	130℃ 2hrs	135℃ 2hrs	103℃ 72hrs
Peas and lentils	105℃ 5hrs	130℃ 1hr	130℃ 2hrs	135℃ 2hrs	
Maize [Food]	105℃ 5hrs	103℃ 72hrs	(130-133)℃ 4hrs		103℃ 72hrs
[Feed]	135℃				
Grain Sorghum [Food]	105℃ 5hrs	130℃ 1hr	130℃ 2hrs	135℃ 2hrs	103℃ 18hrs
[Feed]	135℃ 2hrs				
Soybeans	105℃ 5hrs	130℃ 1hr	130℃ 2hrs	135℃ 2hrs	103℃ 72hrs

105℃ air oven method in Japan

1.5g grinded sample.

2.Two dishes are prepared and weighed with grind sample.

3.All dishes should be placed on a single shelf in the oven.

4.Put all sample into a desiccator.

5.Weight the sample dishes and determine the weight loss.

$$\text{Moisture}(\%) = \frac{M-M_1}{M} \times 100$$

Where M : Weight of the original sample

M_1 : Weight of the sample after drying

6.Moisture contents of two sample dishes should have difference within 0.2%.

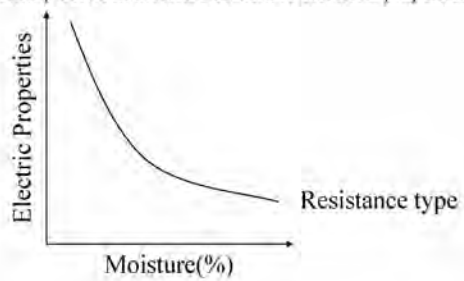
Source of errors in the oven method

1. Grind (when the method requires grinding before drying)
2. Grinding
   * Grinding methods
   * Moisture content
   * Distribution of particle size
   * Thermo-humid condition of the laboratory
3. Sample weights and drying containers
4. Oven
5. Thermometer

Electronic Moisture Testers for grain (Kett models in 2004)

1. Electric Resistance type (Conductance type)—Riceter J & m series
2. Dielectric Constant type (Capacitance type)—PM-400

Relationship between moisture content & electrical properties of grain

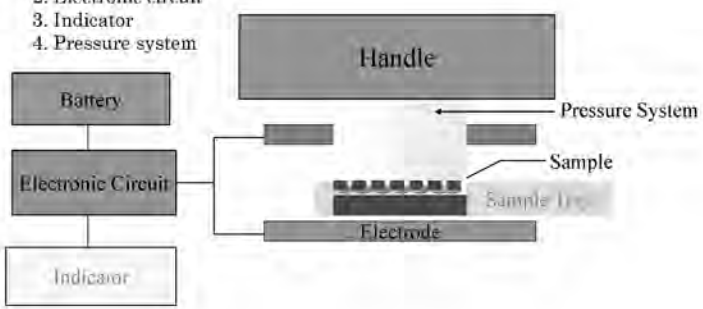


Notice of Electronic Moisture Testers

1. Calibration curve against an appropriate basic method. (i.e. Air Oven drying method)
2. The electrical properties of grain. (Varieties and crop growing conditions)
3. Temperature compensation. (i.e. 25 degree centigrade = - 0.5% correction)
4. Sample mass.
5. Electronic moisture tester should be sued with correctly.
   (Sample mass, pressure, rotating handle, installing the sample to the main unit and so on)

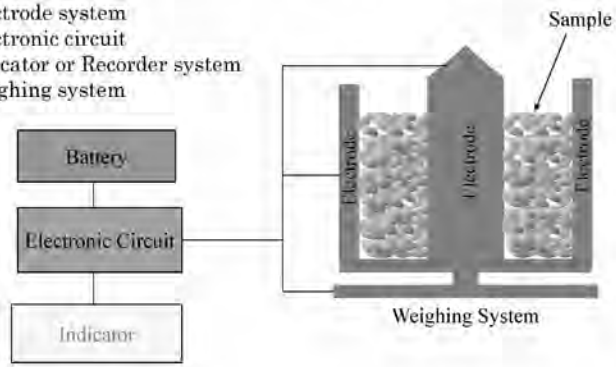
Electric Resistance type Moisture Tester

1. Electrode system
2. Electronic circuit
3. Indicator
4. Pressure system



Dielectric Constant (Capacitance) type Moisture Tester

1. Electrode system
2. Electronic circuit
3. Indicator or Recorder system
4. Weighing system



Checking procedure for model Riceter J/m series (Japanese example)

Need as following tool and reference sample;

1. Checker for Riceter J/m
2. Actual Reference sample (Brown Rice)

Notice :

1. Cleaning : Sample tray, Handle and inside the main unit.
2. Calibration check points : 13.0 & 18.0 by Checker for Riceter J/m.
3. Actual reference sample must be same moisture content.
4. The temperature of moisture tester and reference sample should be same condition.

How to use checker for Riceter J/m

How to use checker for Riceter J/m

1.Check display on the instrument.
2.Check for temperature of instrument.
3.Check for battery voltage.
4.Make the top point of crushing handle and testing chamber clean.
5.Insert Checker into the testing chamber completely.
6.Check 13% calibration.
7.Check 18% calibration.

How to use checker for Riceter J/m

1.Check display on the instrument.
2.Check for temperature of instrument.
3.Check for battery voltage.
4.Make the top point of crushing handle and testing chamber clean.
5.Insert Checker into the testing chamber completely.
6.Check 13% calibration.
7.Check 18% calibration.

How to use checker for Riceter J/m

1.Check display on the instrument.
2.Check for temperature of instrument.
3.Check for battery voltage.
4.Make the top point of crushing handle and testing chamber clean.
5.Insert Checker into the testing chamber completely.
6.Check 13% calibration.
7.Check 18% calibration.

How to use checker for Riceter J/m

1.Check display on the instrument.
2.Check for temperature of instrument.
3.Check for battery voltage.
4.Make the top point of crushing handle and testing chamber clean.
5.Insert Checker into the testing chamber completely.
6.Check 13% calibration.
7.Check 18% calibration.

How to use checker for Riceter J/m

1.Check display on the instrument.
2.Check for temperature of instrument.
3.Check for battery voltage.
4.Make the top point of crushing handle and testing chamber clean.
5.Insert Checker into the testing chamber completely.
6.Check 13% calibration.
7.Check 18% calibration.

How to use checker for Riceter J/m

1.Check display on the instrument.
2.Check for temperature of instrument.
3.Check for battery voltage.
4.Make the top point of crushing handle and testing chamber clean.
5.Insert Checker into the testing chamber completely.
6.Check 13% calibration
7.Check 18% calibration.

How to use checker for Riceter J/m

1.Check display on the instrument.
2.Check for temperature of the instrument.
3.Check for battery voltage.
4.Make the top point of crushing handle and testing chamber clean.
5.Insert Checker into the testing chamber completely.
6.Check 13% calibration.
7.Check 18% calibration

How to use Riceter J/m

Actual moisture measurement for Riceter J/m

1.Check sample tray.
2.Mix the sample grain.
3.Take one layer of rice sample on the sample.
4.Insert the sample tray into the testing chamber completely.
5.Rotate crushing handle quickly until stop.
6.Take measurement five times or more for one sample and record.
7.Make final judge. (Detail refer to OIML or ISO7700)

Actual moisture measurement for Riceter J/m

1.Check sample tray.
2.Mix the sample grain.
3. Take one layer of rice sample on the sample.
4.Insert the sample tray into the testing chamber completely.
5.Rotate crushing handle quickly until stop.
6.Take measurement five times or more for one sample and record.
7.Make final judge. (Detail refer to OIML or ISO7700)

Actual moisture measurement for Riceter J/m

1.Check sample tray.
2.Mix the sample grain.
3.Take one layer of rice sample on the sample.
4.Insert the sample tray into the testing chamber completely.
5.Rotate crushing handle quickly until stop.
6.Take measurement five times or more for one sample and record.
7.Make final judge. (Detail refer to OIML or ISO7700)



Actual moisture measurement for Riceter J/m

1.Check sample tray.
2.Mix the sample grain.
3.Take one layer of rice sample on the sample.
4.Insert the sample tray into the main unit completely.
5.Rotate crushing handle quickly until stop.
6.Take measurement five times or more for one sample and record.
7.Make final judge. (Detail refer to OIML or ISO7700)

Actual moisture measurement for Riceter J/m

1.Check sample tray.
2.Mix the sample grain.
3.Take one layer of rice sample on the sample.
4.Insert the sample tray into the testing chamber completely.
5.Fully rotate the crushing handle quickly until stop is reached.
6.Take measurement five times or more for one sample and record.
7.Make final judge. (Detail refer to OIML or ISO7700)


Actual moisture measurement for Riceter J/m

1.Check sample tray.
2.Mix the sample grain.
3.Take one layer of rice sample on the sample.
4.Insert the sample tray into the testing chamber completely.
5.Rotate crushing handle quickly until stop.
6.Take measurement five times or more for one sample and record.
7.Make final judge. (Detail refer to OIML or ISO7700)

Actual moisture measurement for Riceter J/m

1.Check sample tray.
2.Mix the sample grain.
3.Take one layer of rice sample on the sample.
4.Insert the sample tray into the testing chamber completely
5.Rotate crushing handle quickly until stop.
6.Take measurement five times or more for one sample and record.
7.Make final judge. (Detail refer to OIML or ISO7700)

Checking procedure for model PM-400 (Japanese example)

Need as following tool and reference sample
1. Checker set for PM-400 (2 pcs. checker, Weight and remover)
2. Actual reference sample (Soybean)

Notice :
1. Cleaning : Inside of main unit
2. Calibration check points : 9.6 & 28.0 by Checker for PM−400.
3. Load cell check : 200g weight
4. Actual reference sample must be same moisture content.
5. The temperature of moisture tester and reference sample should be same condition.

How to use checker for PM−400

How to use checker for PM−400

1.Check by Calibration standards
2.Load cell Check
3.Temperature check

How to use checker for PM–400

1.Check by Calibration standards
2.Load cell Check
3.Temperature check


How to use checker for PM–400

1.Check by Calibration standards
2.Load cell Check
3.Temperature check



Verification
for Grain Moisture Testers

1. Model

- 1. Model: Riceter–m411
  m999
  m401
- 2. Model: PM–410
- 3. Model: PM–600

2. Model:
Riceter—m

- (1) Display Check
  Turn "POWER" on and confirm
  If all segments are complete.
- (2) Instrument temperature check
  Keep "Ave" depressed, turn "Power" on.
- (3) Battery check
  Depress "SELECT" and confirm If battery voltage is
  over 5V Otherwise replace all batteries.
- (4) Turn "POWER" off

2. Model:
Riceter—m

- (5) Electricity check by Calibration Standard.
  Insert Calibration standard into Electrode firmly and turn
  Measurement Handle until stop.
- (6) Keep "SELECT" depressed, Turn "POWER" on.
- (7) 13% check
  Put the lever of Calibration Standard to upper position.