MITTATEKNIIKAN KESKUS

CENTRE FOR METROLOGY AND ACCREDITATION

Julkaisu J1/2000

INTERCOMPARISON OF TEMPERATURE STANDARDS OF LITHUANIA AND FINLAND

Thua Weckström

Helsinki 2000

ABSTRACT

The Temperature Standard Laboratory of Lithuania is a new laboratory with 4 fixed point cells (H_2O, Sn, Zn and Al). Within the PRAQIII Fast project: Short term advice on ”Examination and Quality Evaluation of the National Temperature Standard Calibration Procedure” held at the State Metrology Service, Vilnius, Lithuania, an intercomparison of two fixed-point calibrated standard platinum resistance thermometers was made in the fixed point cells of the Temperature Standard Laboratory.

The standard platinum resistance thermometer (SPRT) of the Temperature Standard Laboratory of Lithuania laboratory was a Tinsley thermometer, calibrated by NPL (National Physical Laboratory, United Kingdom) and the SPRT of the Centre for Metrology and Accreditation (MIKES) was a Chino thermometer, calibrated by MIKES. The measurements were made in October 1999.

The results agree with the manufacturer’s specifications for the fixed point cells.

TIIVISTELMÄ

The Temperature Standard Laboratory of Lithuania on Liettuan uusi kansallinen mittanormaalilaboratorio, jolla on neljä kiintopistekennoa (H_2O, Sn, Zn ja Al). Kahden kiintopistekalibroidun platinavastusanturin vertailumittaus suoritettiin laboratorion kiintopistekennoissa PRAQIII Fast projektin yhteydessä: Short term advice on ”Examination and Quality Evaluation of the National Temperature Standard Calibration Procedure” held at the State Metrology Service, Vilnius, Lithuania.

Vertailumittauksessa käytettiin Liettuan kansallisen mittanormaalilaboratorion Tinsley standardiplatinavastuslämpömittaria, joka oli kalibroitu NPL:ssä (National Physical Laboratory, Yhdistynyt Kuningaskunta) ja Chino-anturia, joka oli kalibroitu MIKESissä. Mittaukset suoritettiin lokakuussa vuonna 1999.

Tulokset ovat yhtäpitävät kennojen valmistajan spesifikaatioiden kanssa.

CONTENTS

1. INTRODUCTION

The Temperature Standard Laboratory of Lithuania is a new laboratory with 4 fixed point cells (H_2O, Sn, Zn and Al). The laboratory intends to apply for accreditation some time in the future, and for that reason a comparison measurement with the Centre for Metrology and Accreditation (MIKES) was decided upon. The laboratory has two standard platinum resistance thermometers calibrated at the National Physical Laboratory in Teddington, UK. One of them is calibrated at the fixed points above zero and the other one below zero.

The laboratory has one furnace for the metal fixed point cells, and two baths, one for the triple point of water cell and the other one for the standard resistors. Thus it was difficult to decide what kind of a comparison could be made with the available equipment. In the end it was decided that both laboratories should measure the temperature of the metal fixed point cells of the Vilnius laboratory using the calibration certificates of NPL and MIKES, respectively.

2. EQUIPMENT USED IN THE INTERCOMPARISON

The metal fixed point cells, the furnace and the baths were manufactured by Hart Scientific, USA. The bridge was from Measurement International Ltd in Canada, and the standard resistors were from Tinsley & Co in England. The water triple point cell was manufactured by NPL.

Reference Standards	***Manufacturer/model***	***Serial No***	***Calibration certificate/date***
H_2O triple point cell	NPL 32	788	Q9TM1T31/N97/1998
Other equipment used			
Standard resistor	Tinsley 5658A, 25 Ω	274878	ED.02/99/084/Rm 10.27
Bridge	MIL 6010T	980109	ES 98-33397/10.3.1998
Scanner	MIL 4220A	980303	
Furnace	Hart 9114	8B023	
Resistor bath	Hart 7037		
WTP bath	Hart 7012		

Cells used in the intercomparison	***Manufacturer/model***	***Serial No***	***Calibration certificate/date***
Tin freezing point cell	Hart Scientific 5905*	8016	Sn-8016/3.12.1998
Zinc freezing point cell	Hart Scientific 5906*	8015	Zn-8015/3.12.1998
Aluminium freezing point cell	Hart Scientific 5907*	8025	Al-8025/3.12.1998

*The cells are closed.

Thermometers used in the intercomparison	***Manufacturer/model***	***Serial No***	***Calibration certificate/date***
SPRT (Vilnius)	Tinsley 5187SA	274259	Q9TM1T12/FN97/095/ 23.2.1998
SPRT (MIKES)	Chino R800-2	RS916-3	M-99T151/2.11.1999

3. MEASUREMENT PROCEDURES

The thermometers were calibrated in the triple point of water (TPW) cell, and in each of the fixed point cells, each measurement followed by a TPW measurement.

The TPW cell was prepared on October 1st , but the thermometer well tube was filled with water in the evening of October 4th(Monday). As this was done so late after the preparation, the size of the ice crystals in the ice mantle had begun to grow. This does not generally affect the temperature of the cell, but it is an indication of that the mantle will melt faster than if the cell had been handled in the usual way, filling the thermometer well with water as the last stage of the preparation of the cell.

The Aluminium cell was melted in the furnace over night with a fixed point calibrated type S thermocouple monitoring the temperature of the cell. When the metal had melted on Tuesday October 5th, the temperature of the furnace was lowered. At the beginning of the freezing plateau the inner part of the cell was cooled by inserting a quartz rod into the central tube. After that, the Tinsley thermometer was inserted. The plateau was measured with the software for the bridge. The temperature of the furnace was lowered to 400 °C and the thermometer was withdrawn from the cell. At the end of the day the thermocouple was inserted in the cell, and a new melt was started. On the next day, Wednesday October 6th, the Chino thermometer was inserted and measured in the Aluminium point. It was removed at 400 °C on Thursday October 7th, the set point value of the furnace was decreased, and the cell was removed at the end of the day while still hot. The Hart cells can be lifted from the furnace with a long handle that can be screwed into the lid of the cell. The furnace was still cooled, and at night the Zinc cell was placed in the furnace. The cell was melted with the thermocouple monitoring the temperature. The measurements with both thermometers were made on the same freezing plateau. The cell was then replaced with the Tin cell, this time with a Hart SPRT as a monitoring device. On Monday October 11th the Tin cell was lifted out from the furnace to induce the freezing plateau, and then the measurements on the plateau were started, first with the Tinsley thermometer and then with the Chino thermometer.

The fixed point plateaux are shown in Figs. 1 to 6. The temperature values in the graphs are arbitrary.

The breaks in the Aluminium plateau in Fig. 1 arise from problems with the measurement software.

The shape Aluminium plateau in Fig. 2 arises from the fact that the Chino thermometer should have been annealed at 670 °C after the transportation from Helsinki to Vilnius. However, the laboratory did not have any annealing furnace.

Fig. 6 shows that the Tin plateau was almost ended when the preheated Chino thermometer was put into the cell.

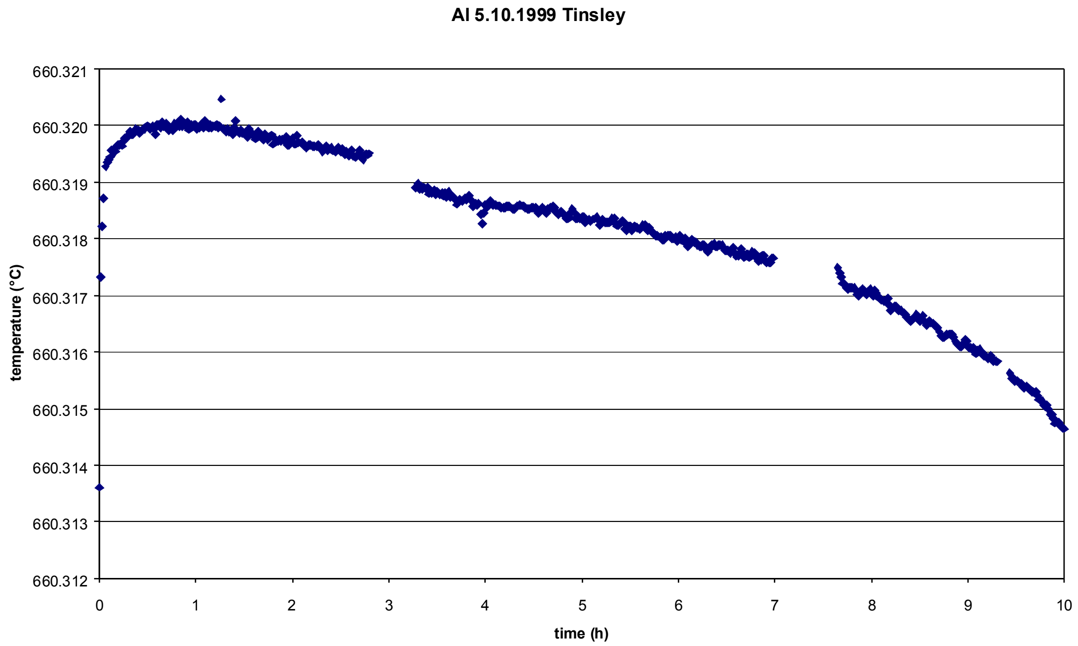

Fig. 1.

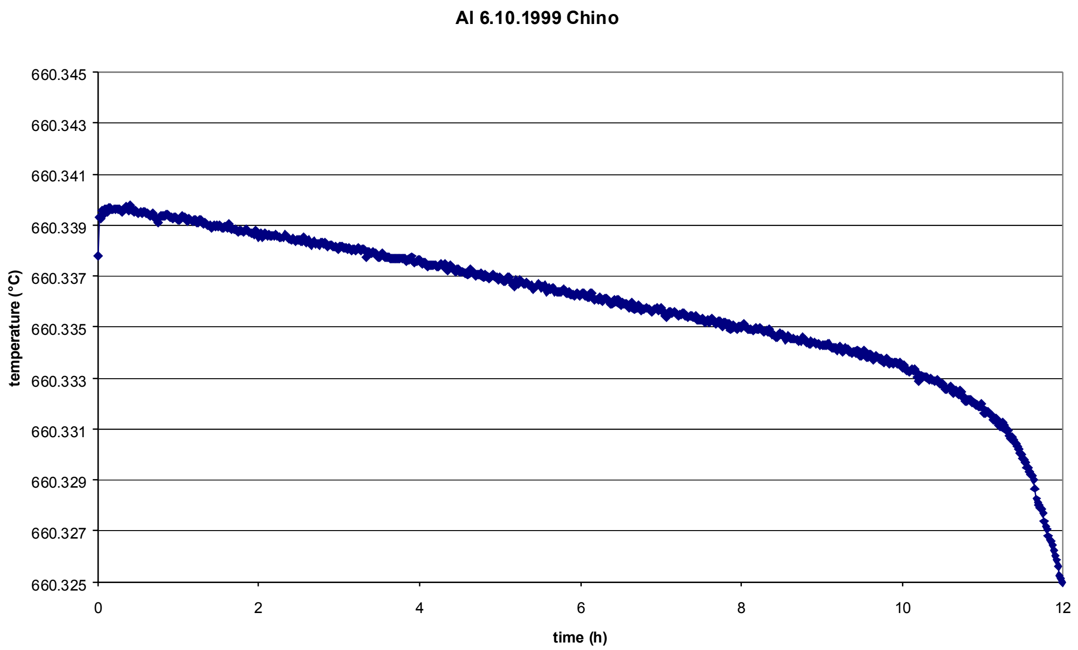

Fig. 2. The values used for the calculation are between 0,05 h and 0,68 h.

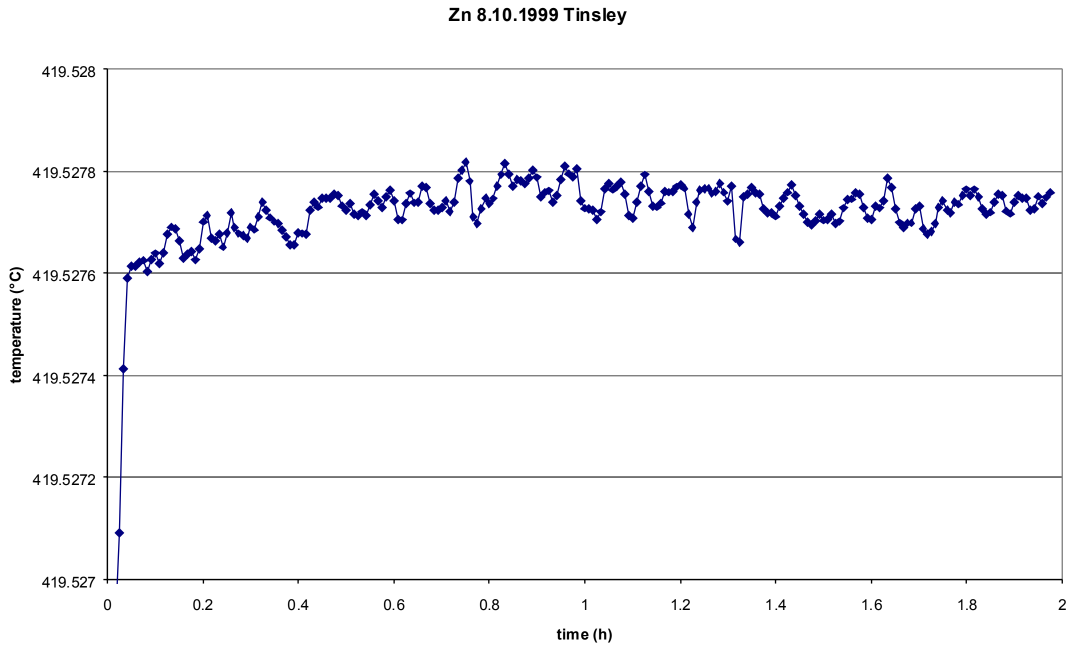

Fig. 3

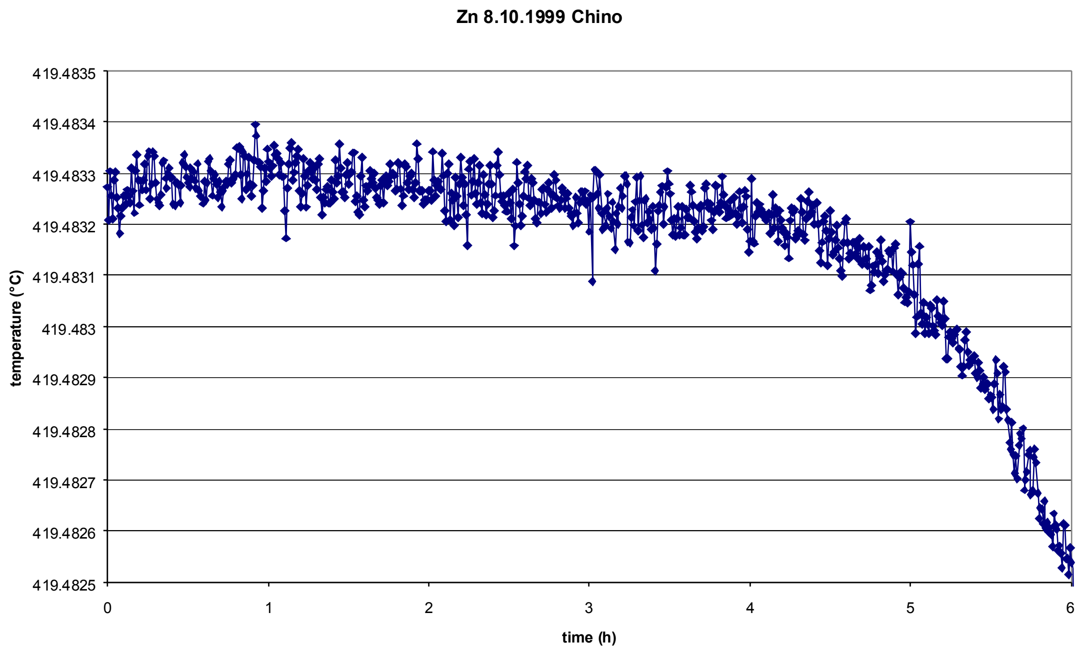

Fig. 4. The values used in the calculation are between 0 h and 2,08 h.

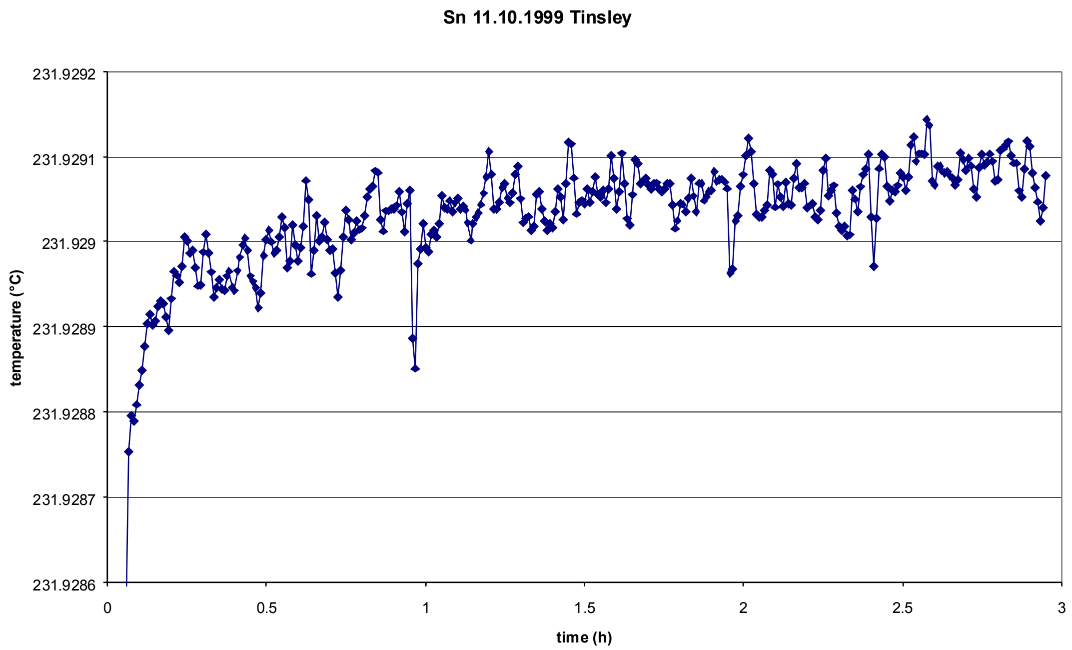

Fig. 5

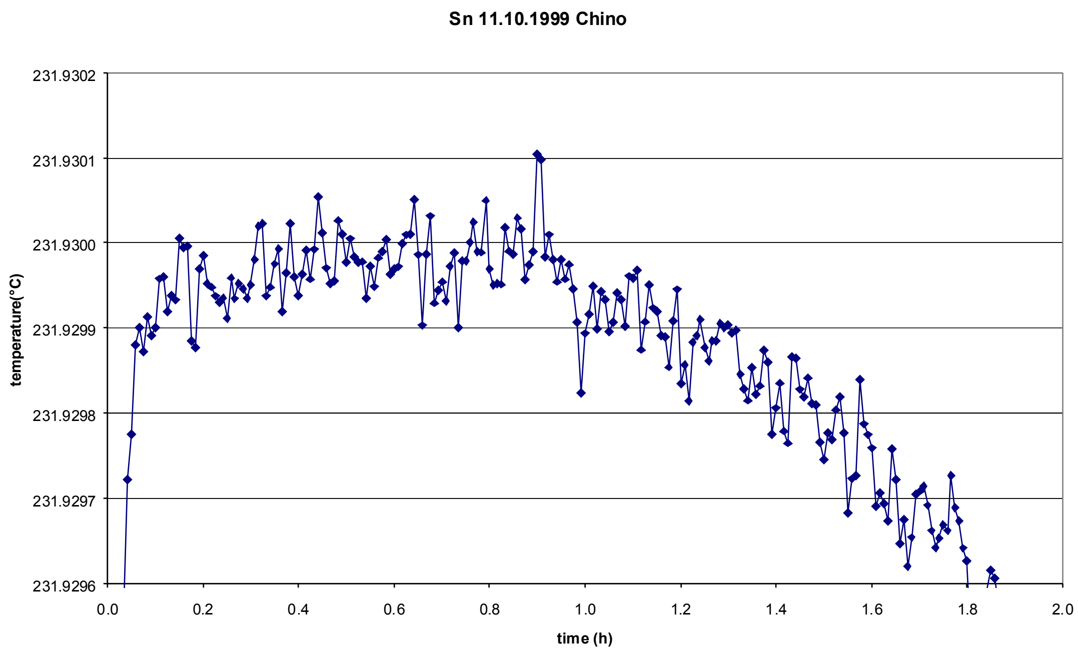

Fig. 6. The values used in the calculation are between 0,11 h and 1,13 h.

The stability of the thermometers can be deduced from the TPW values.

Thermometer	Tinsley	Chino
before Al	25,483 506 Ω	24,811 989 Ω
after Al	25,483 320 Ω	24,811 841 Ω
before Zn	25,483 302 Ω	24,811 843 Ω
after Zn	25,483 285 Ω	24,811 846 Ω
before Sn	25,483 295 Ω	24,811 846 Ω
after Sn	25,483 344 Ω	24,811 899 Ω

The values in this table are not corrected for hydrostatic pressure. From this table it can be seen that both thermometers would have needed an anneal before the measurement, but this was not possible, because the laboratory does not have a annealing furnace.

The resistance of both thermometers increased after the Tin point. This follows from the oxidation of the platinum wire in the thermometers.

4. THE RESULTS OF THE MEASUREMENT

The Vilnius laboratory has calculated the temperatures of the fixed point cells. The results are given in the calibration certificate in Appendix A. The results of MIKES are given in the calibration certificate in Appendix B.

The MIKES thermometer was recalibrated in Helsinki, using an other Aluminium cell than in the calibration made before the comparison.

The results are given in Table 1.

Table 1: The results of the intercomparison

ITS-90 temperature	Hart value (°C)[1]	Hart value (°C)[2]	Tinsley thermometer (Vilnius) (°C)[3]	Chino thermometer (MIKES) (°C)[3]
660,323 °C	660,3222 ± 0,0025	660,3211	660,3210 ± 0,0065	660,3246 ± 0,0072
419,527 °C	419,5268 ± 0,0010	419,5261	419,5250 ± 0,0020	419,5261 ± 0,0021
321,928 °C	231,9278 ± 0,0010	231,9272	231,9270 ± 0,0021	231,9280 ± 0,0022

[1]These values are corrected to a pressure of 101,325 kPa and an immersion depth of 180 mm.
[2]These values are not corrected for pressure, but for an immersion depth of 180 mm.
[3]These values are corrected for an immersion depth of 180 mm.

5. SUMMARY

The result of the intercomparison shows, that the temperature assigned to the Hart fixed point cells is compatible with the values measured with two different kinds of thermometers, the Tinsley thermometer that was calibrated by NPL, and the Chino thermometer that was calibrated by MIKES.

The E_n-values for a comparison are calculated using the deviation E_n normalised with respect to the stated uncertainty, i.e.

$$E_n = \frac{X_{lab} - X_{ref}}{\sqrt{u_{lab}^2 + u_{ref}^2}}$$

X_{lab} is the result of a participating laboratory as given in its calibration certificate, X_{ref} is the reference value assigned to the measurement device on the date when X_{lab} was obtained, u_{lab} is the uncertainty of X_{lab} as given in its calibration certificate. u_{ref} is the uncertainty of X_{ref} which should include an allowance for the performance of the measurement device over the course of the comparison (and may be time-dependent).

The E_n-values calculated by using the Hart values in column 3 of Table 1 as reference values are given in Table 2.

Table 2. The E_n-values of the intercomparison

ITS-90	E_n (Vilnius)	E_n (MIKES)
660,323 °C	0,02	-0,46
419,527 °C	0,49	0
231,928 °C	0,09	-0,33

The result of the comparison is satisfactory, as the absolute values of all E_n-values are smaller than 1. (An E_n-value close to zero can be obtained either if the values measured by the laboratory are the same as those measured by the reference laboratory, or if the uncertainties are large.)

Thus, the measured values of the freezing points of the cells are in accordance with the ones indicated in the certificates of the cells (Table 1).

A more accurate comparison of the temperature produced by the metal fixed point cells should be done with transfer cells and it should be repeated several times during a longer time period.

References:

EA-P7: EA Interlaboratory Comparisons

Hart Scientific: Certification of the Freezing Point of Aluminium Cell Al-8025, of the Freezing Point of Zinc Cell Zn-8015 and of the Freezing point of Tin Cell Sn-8016

MITTATEKNIIKAN KESKUS
PL 239
00181 HELSINKI
Puh. (09) 616 761 (vaihde)
Telekopio (09) 616 7467

CENTRE FOR METROLOGY AND ACCREDITATION
P.O. Box 239
FIN-00181 HELSINKI
Tel. +358 9 616 761
Telefax +358 9 616 7467

ISBN 952-5209-46-6
ISSN 1235-5704