# INTERNATIONAL INTERCOMPARISON OF FIXED POINTS

BY MEANS OF SEALED CELLS

(13.81 K to 90.686 K)

(1978 - 1984)

Final Report of the Pilot Laboratory

by

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# FINAL REPORT

# on the

INTERNATIONAL INTERCOMPARISON OF FIXED POINTS BY MEANS OF SEALED CELLS

(13.81 K to 90.686 K)

(1978 - 1984)

F. Pavese Editor

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### I. FOREWORD

This is the Final Report of the International Intercomparison of Fixed Points by Means of Sealed Cells, which has been held under the auspices of the Comité Consultatif de Thermométrie (CCT) between 1978 and 1984.

Forty sealed cells, realizing the triple point of seven different substances, defining both primary fixed points of the IPTS-68 and secondary fixed points in the temperature range from 14 K to 90 K, were supplied by nine Laboratories. They were measured in eleven National Laboratories around the world, against the fixed points realized in these Laboratories (both in open cryostats or in other sealed cells). Some 150 independent series of data were originated, from almost 300 melting experiments, representing some 2300 equilibrium temperature values.

This work involved a large staff of experts in every Laboratory. The role of IMGC, after the initial proposition of the Intercomparison and apart from its own measurements, has been first to work out a comparison scheme tailored on the requests of each participating Laboratory, secondly to co-ordinate the circulation of the cells between Laboratories in the whole world, and finally to collect, compile and digest the measurement data obtained in the Laboratories, write and edit the present Report.

This could be done also through three Meetings of the participating Laboratories held on the day before CCT Meetings in 1980, 1982 and 1984.

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### II. INTRODUCTION

The calibration of platinum resistance thermometers of specified quality (SPRT) on the International Practical Temperature Scale (IPTS) in the cryogenic region consists of the measurement of their resistance values at a certain number of reference temperatures, corresponding to the boiling and triple points of substances that are gaseous at room temperature. Although the Scale is based on thermodynamic states uniquely defined and on platinum of a specified quality, interlaboratory comparisons are necessary to check the degree of uniformity internationally achieved in the realization of the Temperature Scale.

In fact, the practical implementation of both temperature fixed points and the interpolating instrument may perceivably affect the physical property being measured; with PRTs it brings about a dispersion of the W versus T characteristic, which affects the uniqueness of the Scale definition between fixed point temperatures; with fixed points, it produces different temperature values for the same physical thermodynamic state in different measurements.

Several international intercomparisons have been promoted to control the uniformity of IPTS realizations in different laboratories 1.4. An intercomparison requires travelling standards: only SPRTs, calibrated on laboratory realizations of the IPTS (LAB-IPTS), were available for this purpose in the past, although they are very delicate instruments liable to instability when transported.

In the last 10-year period, extensive studies were made on the realization of fixed points in transportable sealed cells, down to solid hydrogen temperatures (see below for references). Since the beginning, the cells made at IMGC and INM have been compared with conventional realizations at BIPM (1975)<sup>65</sup>, NRC (1976)<sup>66.67</sup> and NPL (1975-78)<sup>52</sup>, and this immediately demonstrated the superior results that could be obtained transporting the cells $^{66.52}$ , with respect to the use of travelling thermometers $^{65.52}$ , as pointed out in Ref.66. Consequently, in 1978 IMGC proposed to undertake under the auspices of the Comité Consultatif de Thermométrie (CCT) an intercomparison of fixed-point realizations in National Laboratories by using fixed points in small sealed cells, instead of capsule PRTs, as travelling standards. This was decided in the 12th Meeting of the CCT in June 1978 <sup>5</sup>. Since these devices are both strong and stable in time, one set of them (one cell for each substance) would have been sufficient for the comparison (only triple points and solid-to-solid transitions could be studied in sealed cells). However, as it was quite a recent device, it was preferred to circulate more than one cell for each substance, in order to check also for the quality of the standards used.

The definition of the goals of the intercomparison required an extensive discussion (see Section III.3); as a result, it was decided that this exercise should generate information in three main areas:

a) Intercomparison of different models of sealed cells.

- b) Intercomparison of candidate substances for reference points.
- c) Relationship between the LAB-IPTS-68 at the reference temperatures considered in the intercomparison.

#### III. INTERCOMPARISON CONFIGURATION

### 1. Standards used in the intercomparison: sealed cells and gases

The travelling standards used to compare the fixed point realizations of the Laboratories were sealed-cell devices, in which a sample of a gaseous substance is permanently enclosed in order to provide a permanent realization of its triple point (and of the solid-to-solid transitions, if any: this possibility has not been used).

As sealed cells from many of the participant Laboratories were available at the time where the comparison started in 1978 (and more became available later), cells from nine of them were circulated between Laboratories. The physical appearance of each different cell model taking part in this exercise is shown in Fig. III.1.

Five different gases were initially selected to be sealed into the cells: argon, oxygen, e-hydrogen, methane and neon; two more were added subsequently: nitrogen and e-deuterium. Table III.1 gives the set of 41 cells and 7 gases involved in the studies. The following references should be consulted for information about cell fabrication and performances: ASMW<sup>55</sup>, BIPM<sup>6</sup>, IMGC<sup>7,8</sup>, INM<sup>9,10</sup>, NBS<sup>11,68</sup>, NIM<sup>18</sup>, NRLM<sup>12</sup>. Cell ageing after sealing ranged from few months (at the beginning of the circulation) to more than eight years (at the end).

## 2. Participating Laboratories

Eleven Laboratories took part in the Intercomparison at different times and with different involvements.

Table III.2 lists the dates when the measurements were made by each Laboratory: some relevant measurements made before 1979 are also included; 151 independent sets of measurements are included, corresponding to each circulating cell being measured by an average of 4-5 Laboratories. Some 300 meltings were performed during these studies.

Since the cells are stable devices, a regular pattern was not required for the circulation scheme. However, the circulation plan was designed so as to send at least two models of cells for each of the substances and at the same time. The circulation started at the beginning of 1979 and was completed by the beginning of 1984. Although a variety of unexpected events has randomly disturbed the regularity of the circulation pattern (involving 31 of the devices and 11 countries in

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Fig. III.1 a - A S M W models 1 and 2

<u>Cell parts</u>: A: body (copper); B: block for one thermometer (copper); C: "pinch-off" tube (copper).

<u>Cell assembling</u>: silver brazing.

Cell volume: mod.1 2.5 cm<sup>3</sup> mod.2 5 cm<sup>3</sup>

Volume for condensed sample (surrounding the block): mod.1 0.6 cm<sup>3</sup> mod.2 2 cm<sup>3</sup>

Filling method: argon, cryogenic condensation; neon, high pressure at 78 K.





Fig. III.1 b - <u>B I P M</u>

 Cell parts:
 A: well for one thermometer; B:"pinch-off" tube (copper);

 C: sample wells (6); D: body (stainless steel).

 Cell assembling:
 arc welding, except for the "pinch-off" tube, which is silver brazed.

 Cell volume:
 74 cm<sup>3</sup>

 Volume for condensed sample:
 (in the wells):
 9 cm<sup>3</sup>

 Filling method:
 cryogenic condensation.

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Fig. III.1 c - IMGC models A and B

<u>Cell parts</u>: A: well for one (mod.A) or three (mod.B) thermometers; B: indium seal; C: thermometer block with vertical fins (copper); D: body (stainless steel).

Cell assembling: arc welding.

Cell volume: mod.A 33 cm<sup>3</sup> mod.B 22 cm<sup>3</sup>

Volume for condensed sample (surrounding the block): mod.A 5 cm<sup>3</sup> mod.B 4 cm<sup>3</sup>

Filling method: cryogenic condensation.





Fig. III.1 d - IMGC model C

<u>Cell parts</u>: A: large conical well for thermometer adaptor; B: indium seal; C: thermometer block with helical thread (copper); D: body (stainless steel).

Cell assembling: arc welding.

<u>Cell volume:</u> 20 cm<sup>3</sup>

<u>Volume for condensed sample</u> (surrounding the block): 2.5 cm<sup>3</sup>

Filling method: cryogenic condensation.





Fig. III.1 e - IMGC model M

<u>Cell parts</u>: B: indium seal; D: body (stainless steel); F:body (copper with horizontal fins) to transfer interface temperature to the external thermometer block (not shown).

Cell assembling: arc welding.

Cell volume: 8 cm<sup>3</sup>

Volume for condensed sample (surrounding the copper body): 1 cm<sup>3</sup>

Filling method: cryogenic condensation.





Fig. III.1 f - <u>INM</u> model A

 <u>Cell parts</u>:
 A: well for one thermometer; B:"pinch-off" tube (copper);

 C: sample wells (6); D: body (stainless steel).

 <u>Cell assembling</u>:
 arc welding, except for the "pinch-off" tube, which is silver brazed.

 <u>Cell volume</u>:
 60 cm<sup>3</sup>

 <u>Volume for condensed sample</u> (in the wells):
 9 cm<sup>3</sup>

 <u>Filling method</u>:
 cryogenic condensation.





INM mod. BCM

Fig. III.1 g - I N M model BCM (Multicomponent cell)

<u>Cell parts</u>: A: well for one thermometer; B: body (stainless steel); C: "pinch-off" tube (copper: one for each chamber); D: sample chambers with heat exchanger.

<u>Cell assmbling</u>: arc welding, except for the "pinch-off" tubes, which are silver brazed.

Cell volume (each chamber): 7.5 cm<sup>3</sup>

Volume for condensed sample (in the thread): 0.9 cm<sup>3</sup>





NBS (Ar)

Fig. III.1 h N B S model M1

<u>Cell parts</u>: A: large well for one thermometer; B: "pinch-off" tube (stainless steel); C: sample chamber; D: body (stainless steel); E: isothermal thin shell (copper).

Cell'assembling: arc welding.

<u>Cell volume:</u> 50 cm<sup>3</sup>

Volume for condensed sample (surrounding the well): 4 cm<sup>3</sup>





Fig. III.1 i - NBS model M2

<u>Cell parts</u>: A: large well for one thermometer; B: "pinch-off" tube (stainless steel); C: sample chamber; D: body (stainless steel); E: isothermal thin shell (copper).

Cell assembling: arc welding.

<u>Cell volume</u>: n°2-1: 50 cm<sup>3</sup>; n° 2-3: 16 cm<sup>3</sup> <u>Volume for condensed sample</u> (surrounding the well): 2 cm<sup>3</sup> <u>Filling method</u>: cryogenic condensation.





NIM mod. BC-INM

Fig. III.1 j - N I M model BC(INM)

<u>Cell parts:</u> A: well for one thermometer; B:"pinch-off" tube (copper); C: heat exchanger; D: body (stainless steel).

<u>Cell assembling</u>: arc welding, except for the "pinch-off" tube, which is silver brazed.

Cell volume: 15 cm<sup>3</sup>

Volume for condensed sample (in the thread): 0.9 cm<sup>3</sup>





NIM

Fig. III.1 k - <u>N I M</u> model 1

<u>Cell parts</u>: A: large well for one thermometer (copper); B:"pinch-off" tube (copper); C: body (copper).

Cell assembling: silver brazing.

<u>Cell volume</u>: 7.5 cm<sup>3</sup>

Volume for condensed sample (surrounding the well): 1 cm<sup>3</sup>





NRC

Fig. III.1 1 - <u>NRC</u>

<u>Cell parts</u>: A: well for one thermometer; B: indium seal; C: thermometer block (copper) with horizontal thin baffles (15 copper disks, 0.5 mm spacing); D: body (copper, with external stainless steel jacket). Hydrogen cell: all stainless steel.

Cell assembling: arc welding and silver brazing.

Cell volume: 35 cm<sup>3</sup>

Volume for condensed sample (between the baffles): 5 cm<sup>3</sup>





NRLM

Fig. III.1 m - NRLM

<u>Cell parts</u>: A: large well for one thermometer (copper); B:"pinch-off" tube (copper); C: body (copper).

<u>Cell assembling</u>: silver brazing.

Cell volume: 15 cm<sup>3</sup>

<u>Volume for condensed sample</u> (around the well): 0.9 cm<sup>3</sup>

<u>Filling method</u>: (supposed) Ar,  $0_2$ , CH<sub>4</sub>, cryogenic condensation; H<sub>2</sub>, Ne high pressure, room temperature.





Fig. III.1 n - PRMI Multicomponent cell

<u>Cell parts</u>: A: thermometer well (copper tube); B<sub>lto6</sub>: sample chambers (copper coil).

<u>Cell assembling</u>: silver brazing.

<u>Cell volume</u> (each chamber): 20 cm<sup>3</sup>

Volume for condensed sample: non definable.

Filling method:

# Table III.1 : Sealed cells involved in the intercomparison

# Substances sealed in the cells

Laboratories	ARGON	OXYGEN	HYDROGEN	METHANE	NITROGEN	NEON	e-DEUTERIUM	
supplying the cells	°83.798 K	°54.361 K	°13.81 K	<b>^90.7</b> К	^63.1 К	^24.6 K	^18.7 K	
ASMW	4Ar(May 81)					1Ne (Nov 81)		
BIPM	3Ar(Feb 77)*			7CH4(Sep 77)				
IMGC	<u>1Ar(Jul 75)</u> 2Ar(May 78)	102(Sep 76) 802(Nov 78)	1H2(Aug 80) 2H2(Jan 83)*	2CH4(Aug 76) 12CH4(Apr79)	2N2(Feb 80)	1Ne(Jun 77) 3Ne(Feb 79)	1eD2(Nov 80)	
INM	1Ar(Sep 75) XXI(Dec 78) BCM4(Jan 82)	802(Feb 76) BCM4(Jan 82)			BCM4(Jan 82)	BCM4(Jan 82)		
NBS	MlAr(Feb 78)	M2-102 (Feb83	)* )+					
NIM	113(Dec 80)*	PP11(Aug81)* PP07(Ju181)*	)*					
NRC	10Ar(May 79) 14Ar(May 79)	1502(Jun 79)	23H2(Aug 79)	18CH4 (Aug79)	33N2(Jul 82)	12Ne(Jun 79)	31eD2(Dec81)*	
NRLM	7803(Jun 78)	7802 (Jun78)* 7801 (Jun78)	7801(Jun 78)			Ne01(Jul 78) Ne02(Jul 78)		
PRMI	MC(Dec 78)*	MC(Dec 78)*	MC(Dec 78)*			MC(Dec 78)*		

\* cell not circulated; reference cell; °IPTS-68 value; ^approximate value.

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111.2	2			CELLS	available f	'rom:			I
 ies	А S M И I	BIPM	INGC	! INN	NBS	NIH	INRC I	NRLH I	PRMI
Ат 11 02 11 H2 11	Jun 81		Nov 79   Feb 80   Mar 81   Dec 79	1	     		Feb 81	Nav 80   I Dec 80	
Ne i	Dec 82		Mar 81			u.	Jan 81	i	
Ar II CH4 II		i   Feb BO   Feb-Arr BOI	i Dec 79   Arr 80	   Nov 79   	l Feb 80 l I		  Feb-Har 80    Jan-Feb 80   	l Nov-Dec 791 I	
Ar   02   H2   CH4   N2	Oct 82		Sep75-Hay80     Sep76-Nov81     Oct80-Jun83     Aus76-Oct81     Aus 80		1		Apr 80     Sep 80     Oct 80     Jul 80     Jun 83	Sep 80   Nov 80   Mar 79     	
Ne li D2 l	l May 83		Jun77-Dec81   Dec 80		1		Oct 80   	Sep 80	ъ.
Ar   02   N2		     	APT 80 1 Oct 80 1 Sep 83	  Apr80-Feb821  Hay60-Feb821   Feb 82	ב		1    Apr 80     May 80   		
Ne      	   	   	Sep 83 	Mar 82		   	 		
Ar   02   	[ ] 	   	Aus 79   Nar 84 	   	Feb-Har 791 Mar-Oct 831	t 1 1	Feb 81   	Jul 81   	
Ar   02   CH4		   	Feb 81   Feb 81   Mar 81	Aug 81     Aug 81   		Aug 81   Jul 81 	Aus 81   	1	
Ar 1 OZ 1 H2 1 Na 1	1 1 1	1	Hay 79   Nov 78     Dec 87	Hay 79     Sep 79   	2	[   	Nov 79   Nov 79   Feb 83   Dec 79	Jul 79       Oct 79	
Ar i 02 i H2 i	     	   	I I Jan 78 I Feb 78 I	     		     	   Nov 81   Nov 81   Feb 80	     May 79	
CH4 I N2 I Ne I D2 I	     	1 1 1	Apr 78   Nov 80   Jan 80   Nov 81 	1 1 1 1		     	l I Feb 80 I	    May79-Feb80    	e.
Ar 1 02 1 H2 1 CH4 1	[ ] ] ]	     Aus:79	1 Dec 76 1Dec76-Oct79 1 1Dec75-Aus79	1 Dec 76-Jul 82 1 Jun 76-Jul 82 1 1	1 Aus: 79 1 1 1	     	Jun79-Ju180   Jun 79   Sep 79   Aus 79	1 May 79 1 Oct 79 1 Jun 80 1	
N2   Ne   D2		   	Nov 81   Jun 79   Nov 81	Jul 82   Jul 82   		1	Jul 82  Ser79-Feb82   Dec 81	    Oct 79	
Ar   02   H2	1	1 1 1	Nav 81   Nav 81 	1 1 1	May 82   	   1	1 1	Oct78-Nov81  Oct78-Nov81  Ju178-Nov81	1
CH4 1 Ne 1	1	1	l Nav 81 I Nav 81	1	1	1	1	i lAus78-Nov81 l	1
Ar   02   H2   CH4	     	1 1 1	Dec 81   Dec 81   Dec 81   Dec 81   Dec 81	       	       	       	1 1 1	1	Nov 81   Nov 81   Nov 81   Nov 81 
	LII.2         ies         ies         ifes         ies         ifes         ifes	II	II	III.2         III.4 S M W       B I P M       I M G C         II.4 S M W       B I P M       I M G C         II.4 Jun B1       I Nov 79         Q2       I       I Feb 80         H2       I       I Feb 80         H2       I       I Dec 79         Ne       II Dec 82       I Mar 81         II       I Feb 80       Dec 79         CH4       I Feb-Apr 80       Apr 80         II       I Sep75-Mar90         Q2       I I       I Sep76-Nov81         H2       I I       I Gct80-Jun83         CH4       I I       I Aws 83       I Jun77-Dec81         N2       II       I Aws 79       Q2         Q2       II	C E L L S         II       A S M W       B I P M       I M G C       I N M         II       Jun 61       I Nov 73       I       I         Ar II       Jun 61       I Nov 73       I       I         Ar II       I Dec 73       I       I       I         CH4 II       I Dec 73       I       I       I         II       I Dec 82       I Mar 81       I       I         II       I Feb 80       Dec 73       Nov 73       I         II       I Feb 73-Mar80       Dec 73       Nov 73       I         II       I Feb 73-Mar80       Dec 73       Nov 73       I         II       I Sep75-Mar80       I       I       I         II       I Sep75-Mar80       I       I       I         II       I Sep75-Mar80       I       I       I         II       I Sep73-Mar80       I       I       I         II       I Sep73-Mar80       I       I       I         II       I Sep73       I       I       I       I         II       I Aus 70       I       I       I       I         II       I Aus 83	CII.2       CELLS       available f         ies       A S M W       B I P M       I M G C       I N H       N 8 S         Ar       Jun 81       Feb 80       I       I       I         Ar       I Jun 81       Feb 80       I       I       I         Ar       I Jun 81       Pec 79       I       I       I         Ar       I Dec 82       Mar 81       I       I       I         Ar       I Dec 82       Mar 81       I       I       I         Ar       I Dec 82       ISer75-Mar90       I       I       I         Ar       I Oct 82       ISer76-Mar90       I       I       I         Ar       I Oct 82       ISer76-Mar90       I       I       I         Ar       I Mar 83       Jun77-Dec81       I       I       I         D2       II       Apr 80       Apr 80       I       Apr 80       I         Ar       I Apr 80       Apr 80       Apr 80       Apr 80       I       Apr 80       I         Z       II       Apr 80       Apr 80       IApr 80       I       Apr 80       I         Z       II       A	III.2       CELLS       evailable from:         ies       ASMA       BIPH       IMGC       INM       NBS       NIM         ies       ASMA       BIPH       IMGC       INM       NBS       NIM         ies       Jun 61       Feb 80       Ier 73       Ier 73       Ier 73       Ier 73         ies       I       Dec 73       Nov 73       Feb 80       Ier 73       Ier 74       Ier 74         ies       I       Dec 73       Nov 73       Feb 80       Ier 74       Ier 74       Ier 74         ies       I       Dec 73       Nov 73       Feb 80       Ier 74       Ier 74 <td< td=""><td>III.2       CELLS       available Free:         ies       ASMA       BIPM       INGC       INM       NBS       NIM       NRC         ies       ASMA       BIPM       INGC       INM       NBS       NIM       NRC         ies       Jan BI       Feb B0       Feb B0       Feb B1       Feb B1       Feb B1         IZ       Mar B1       Mar B1       Jan B1       Jan B1       Jan B1         IA       Mar B1       Dec 73       Nou 73       Feb B0       Feb Har B0         IA       Feb A0       Dec 73       Nou 73       Feb B0       Feb Har B0         IA       IOct 82       ISer75-Mar B0       IAerr B0       IAerr B0         IZ       IOct 82       ISer75-Mar B0       IAerr B0       IAerr B0         IZ       IOct 82       ISer75-Mar B0       IAerr B0       IAerr B0         IZ       IOct 82       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    IA       Feb A0       Dec 73       Nou 73       Feb B0       Feb Har B0         IA       IOct 82       ISer75-Mar B0       IAerr B0       IAerr B0         IZ       IOct 82       ISer75-Mar B0       IAerr B0       IAerr B0         IZ       IOct 82       ISer75-Mar B0       IAerr B0       IAerr B0         IZ       IOct 82       ISer75-Mar B0       IAerr B0       IAerr B0         IZ       IOct 82       ISer75-Mar B0       IAerr B0       IAerr B0         IZ       IOct 82       ISer75-Mar B0       IAer B0       IAer B0         IZ       IAar B0       IAer B0       IAer B0       IAer B0         IZ	LII.2       C C L L S       available Free:         iss       I A S M M       B I P M       I N G C       I N M       N B S       N I M       N R C       N R L M         ar       J an B1       Ner 73       Feb 80       Feb 81       Jae 83       Jae 83

the whole world), loss of substantial information could almost entirely be avoided, at the expense of an increase in the total time initially planned for the exercise.

The stability of each cell was controlled by the originating Laboratory at the end of the circulation.

## 3. Purposes of the intercomparison

The meaning of the direct comparison of fixed points made possible by the sealed cell devices has been subject of extensive discussion among the Laboratories involved.

At the beginning, the intercomparison had been proposed only to complement the intercomparison of National IPTS-68 realizations, performed at NPL since 1975, where calibrated SPRTs were used as travelling standards. As these standards are delicate instruments liable to instability in transportation, the use of transportable fixed points, insensitive to normal transportation, should have permitted a comparison of national fixed-point realizations with higher reliability (± 0.1 mK).

Subsequently, the availability of many models of cells in 1978, suggested to extend the comparison to the realization of fixed points in different types of cells.

As a result of the discussion, the comparison was set to provide information in the following areas:

a) Intercomparison of different cell models.

This exercise comprised direct comparison of the devices received by each Laboratory. This comparison does not involve either Scale realization or the quality of the thermometer used, except its short-term stability, since the same thermometer must be used with all cells. This rule was strictly followed, with only few exceptions.

The aim is to understand if these devices are suitable for accurate realization of fixed points, and how much the results on each single cell are dependent on the ancillary equipment.

b) Intercomparison of candidate substances for reference points.

The gases included in this exercise are all the substances with triple-point temperature lower than 90 K. They were already previously studied in some of the participating Laboratories. However the use of common devices for all the Laboratories made it possible to study the quality of each substance as a candidate for a reference temperature in a more uniform way. In fact, the merits of a candidate-substance do not only consist of a flat and reproducible melting plateau, but also on the possibility of a realization which should be simple, reliable and largely insensitive to the quality of the thermal equipment and to the details of the experimental method used.

c) Relationship between LAB-IPTS-68s at reference temperatures.

The intercomparison makes it possible first, to measure the systematic differences between the LAB-IPTS-68 realizations and the reference travelling standards at three definition temperatures; secondly, to assign a temperature value to four secondary fixed points. This latter goal involves the use of the National Scales; hence, some precautions must be taken to limit the influence of the scale non-uniqueness.

## IV. EXPERIMENTAL CONDITIONS

### 1. Measurement equipment

To carry out the measurements, the Laboratories used their routine equipment for the realization of the IPTS-68. The following references should be consulted for more information on each Laboratory realization:

- <u>A S M W</u> (26) The cells are suspended in an adiabatic cryostat by means of plastic wires or a rod. Shield is controlled to less than 5 mK to the cell temperature. Residual heat leak corresponds to a drift of the cell temperature less than 5 mK/h. Resistance measurements are made with a R-348 dc potentiometer, with a temperature-equivalent resolution of about 0.1 mK at Ar, 0.3 mK at Ne and 0.4 mK at H<sub>2</sub> triple point.
- <u>BIPM</u> (6,14) The cells are suspended in an adiabatic cryostat by means of a plastic rod. The shield is controlled so that the residual heat exchange (positive or negative) corresponds to a temperature drift of the cell of few millikelvins per hour. Resistance measurements are made with a dc Kusters Comparator bridge, with a temperature-equivalent resolution of 0.01 mK at Ar and CH<sub>L</sub> triple points.
- <u>I M G C</u> (8) Two adiabatic cryostats have been used for the work respectively below and above 54 K; the one used for higher temperatures is a much simplified one. The cells are suspended by means of a plastic rod. Thermal drift rates (always positive) generally do not exceed 10 mK/h. Resistance measurements are made with a dc Kusters Comparator bridge, with a temperature-equivalent resolution of 0.01 mK above 50 K, decreasing to 0.15 mK at 14 K. All plateaux are continuously recorded on paper.

- <u>INM</u> (15,10) An adiabatic calorimeter with one isothermal shield has been used, regulated to the same temperature of the cell by means of a differential thermocouple; he cell is suspended with nylon threads. The typical thermal drift rate is less than 1 mK/h. Resistance measurements are made with a dc Kusters Comparator bridge, connected to an automatic data acquisition system. This system calculates the response time of the cell using a statistical criterion that always leads to larger values than the adopted criterion of recovery of temperature within 0.1 mK (see later on).
- <u>N B S</u> (17) An adiabatic calorimeter built for specific heat measurements has been used. The cells were enclosed in an auxiliary copper shell suspended inside the shield with plastic threads, so that the residual temperature drift rate is of the order of 1 mK/h. Resistance measurements are made with a dc Kusters Comparator bridge, with a temperature-equivalent resolution of 0.01 mK.
- <u>N I M</u> (18,53) Two cryostats have been used for the work below and above 84 K; both are of the adiabatic type, but that used at the higher temperature is a much simplified one. The cells are suspended by means of plastic wires or rod. Shield is controlled to closer than 10 mK to the cell temperature. Residual heat leak corresponds to a drift of the cell temperature of less than 10 mK/h. Resistance measurements are made with a dc Kusters Comparator bridge with a temperature-equivalent resolution of 0.01 K above 50 K.
- <u>N M L</u> (19) The cells are suspended from the shield of a flow adiabatic cryostat with a low-conductance thermal path made of stainless steel. Residual thermal drift rate was kept below 2 mK/h. Resistance measurements are made with a dc Kusters Comparator bridge, or with an automatic data acquisition system with a temperature-equivalent resolution of 0.01 mK.
- <u>N P L</u> (3) The adiabiatic cryostat of the 1975 intercomparison has been used, where residual thermal drift rates less than 1 mK/h were generally obtained. Resistance measurements are made with a dc Kusters Comparator bridge.
- <u>N R C</u> (20) An adiabatic cryostat has been used with residual thermal drift rate lower than 1 mK/h. Resistance measurements are made with a dc Kusters Comparator bridge; before 1979 a Mueller G4 bridge has been used.

- <u>N R L M</u> (21) An adiabatic calorimeter has been used, with the temperature of the isothermal shield controlled so as to be the same as that of the sample cell, using a differential thermocouple. The residual thermal drift rate is of the order of a few millikelvins per hour. Resistance measurements are made with a dc Kusters Comparator bridge with a temperature-equivalent resolution of 0.02 mK.
- <u>P R M I</u> (22) An adiabatic calorimeter has been used with two adiabatic shields. The outer one is controlled through a differential thermocouple. The inner one is regulated in an absolute way with a resistance thermometer or,relative to the cell, with another differential thermocouple. The residual heat leak is less than 0.1 mW at 14 K and 3 mW at 90 K. IMGC cells were suspended by means of a thinwalled stainless-steel tube; the PRMI cell is held by three needle points away from a further shield. Thermal drift rates do not exceed 5 mK/h. Resistance measurements are made with a potentiometer R-348, calibrated with a precision of ± 3 ppm. The resistance standards are certified to be accurate within ± 1 ppm. Temperature resolution is never worse than 0.1 mK.

Each Laboratory used SPRTs of their own for the measurements. As a rule, the same thermometer was always used in each Laboratory, at least for all the cells containing the same substance. Each Laboratory was also asked to use the thermometers which had taken part in the 1975 NPL intercomparison, in order to establish a relationship between the previous and the present exercise (see section VII.2). These rules have been followed, except in a few cases, which will be listed later on.

### 2. Cell measurement method

Some Laboratories supplied a reference procedure for the use of their cells as, at the beginning of the exercise, it was thought that the method of using the cells might sensibly affect cell performances and results. This was eventually proved not to be the case.

The cells were measured with methods differing in many details but one: the calorimetric method of heating the cell in discrete amounts and waiting for temperature re-equilibration , under adiabatic conditions, was always followed.

#### The differences in method concerned mainly:

rate of cooling to freeze the sample; temperature distribution in the cell during cool-down, owing to different cryostat geometries; minimum temperature allowed in the solidified sample; stabilization time before melting; heating rate to warm the sample to the melting temperature; residual heat exchange; temperature drift-rate value considered as the limit during re-equilibration, for definition of the equilibrium conditions.

A uniform criterion was used in order to evaluate the triple-point temperature: the experimental equilibrium temperature values at different melted fractions F were plotted as temperature (actually resistance) versus 1/F and the extrapolated temperature value at 1/F=1 was defined as the triple-point temperature.

3. Measurement accuracy

It is necessary to make a distinction between the part of the Intercomparison which involves the definition of temperature values and that where only differences between resistance values are required.

3.1 Accuracy in cell intercomparison

This concerns the comparison of the different cell models for each substance (section VII.1), where it is only necessary to define a resistance value at 1/F=1 for each melting and to compare it with the corresponding values of other melting plateaux.

In this case, uncertainty derives from the following sources:

- a) reproducibility of measurements in each laboratory. It was obtained from direct assessment of the Laboratory or from reproducibility of data from several melting plateaux made with the same cell;
- b) fitting of the melting plateau curve. The plateaux were all made by intermittent melting; therefore each plateau consists of a number of experimental equilibrium temperatures obtained at different liquid-to-solid ratios F. By plotting these values versus l/F one obtains the usual melting plot which shows a negative slope. The temperature values at the definition value F=1 must be obtained usually by extrapolation from lower F values and the difficulty in defining a smooth curve through the experimental points may limit the precision of the triple-point temperature value assigned to that plateau.
- c) thermometer calibration. In a few cases, where the cells were measured with different thermometers (or with the same thermometer over a large time interval during which the R(0°C) value could have changed), additional uncertainty derives from thermometer calibration.

It must be pointed out that, owing the small number of measurements generally involved, a curve through the experimental points on a plateau could not be easily fitted mathematically. Therefore, the uncertainties associated with each difference between cells are entirely related to the scatter range of the values available for this analysis, or to source a). However, the resulting figure was <u>set</u> at a value never less than  $\pm$  0.1 mK. Considering that the difference values come from a pair of independent measurements of comparable precision, the uncertainty associated with these differences will be 1.41 times larger than that of the single measurement.

The uncertainty values for these differences can be summarized as follows for each Laboratory:

- <u>A S M W</u>: values assigned by the Laboratory (1  $\sigma$ ): ± 0.15 mK above 54 K; ± 0.25 mK at 25 K; ± 0.5 mK at 14 K.
- <u>B I P M</u>: essentially limited by the "definability" of the melting plateau: ± 0.3 mK.
- <u>I M G C</u>: values assigned by the Laboratory on the basis of the internal consistency of a large number of measurements on melting plateaux:  $\pm$  0.15 mK, except with methane ( $\pm$  0.2 mK) and hydrogen ( $\pm$  0.3 mK).
- INM : value assigned: ± 0.15 mK.
- <u>N B S</u> : value assigned: ± 0.15 mK. The measurements show a better reproducibility.
- N I M : limited by the "definability" of the melting plateau: ± 0.4 mK
- N M L : value assigned: ± 0.15 mK.
- <u>N P L</u> : value assigned: ± 0.15 mK, in consideration of the accuracy figure reported in Ref.3.
- <u>N R C</u> : value assigned : ± 0.15 mK, in consideration of the accuracy figures stated in most of the published studies on the same substances.
- N R L M : value assigned : ± 0.15 mK.
- P R M I : value assigned by the Laboratory: ± 0.3 mK.

The figures given above still apply to the comparison of the sealed cells against the conventional realizations of both definition and secondary fixed points in each Laboratory, provided again that the same thermometer is used for all the measurements.

3.2 Accuracy in assigning temperature values

When temperature values are to be stated, the whole Scale realization in each Laboratory is involved (as in goal c), Sections VI.2 and VII.2). Hence, the accuracy associated with these temperature values depends not only on the uncertainty figures given above, but also on the contributions introduced by the Scale definition itself.

The latter are twofold: i) systematic errors in the definition fixed points; ii) scale non-uniqueness between fixed points. For temperatures above 54 K, this intercomparison included a check of item i). However, most of the national IPTS-68 realizations are still obtained by using the condensation point of oxygen instead of the triple point of argon, and it is known that the two realizations are not unique within  $\pm$  0.1 mK between 80 K and 90 K (see later for methane).

The non-uniqueness of the IPTS-68 between fixed points is known to be considerably larger than ± 0.1 mK; for this reason, the temperature value assigned to each non-definition fixed point sensibly depends on the thermometer used and there is no way to by-pass the problem. It can only be limited by using a single thermometer for all the measurements or by using a "virtual" unique thermometer. The first solution would have implied shipping one thermometer with the cells: as it was completely unpractical, this solution has been discarded. On the contrary, it was decided to take advantage of the exercise made at NPL in the previous intercomparison, when thermometers from all the laboratories participating in the present exercise were compared at several temperatures in the range 14 K to 273 K. With them it was possible to cancel out the effect of Scale non-uniqueness, and that group could be virtually considered as a single thermometer, available to all the Laboratories and not needing further transportation. The level of uniqueness was stated at NPL to be  $\pm$  0.1 mK in 1975 <sup>3</sup>; however, the present traceability to those measurements has been lowered in the years by the degree of stability of each thermometer with time.

Appendix I summarizes the data of the thermometers used in the measurements of cells in different Laboratories. Relevant comments about the quality of the thermometers are given here for the individual Laboratories.

- <u>A S M W</u> : thermometer 217990, 217997 and 207278. All these thermometers were measured at NPL in 1975; R(0°C) values drifted since then by -140, -30 and +80  $\mu\Omega$ , but original W values are assumed to be still retained. However, below 30 K some disagreement seems to occur between 217990 and 217997, up to 1 mK level.
- <u>B I P M</u> : thermometer 226321 and 226322. This Laboratory did not take part in the NPL exercise but, later, NPL calibrated both thermometers and stated that this calibration can be traced to the intercomparison within  $\pm$  0.1 mK <sup>23</sup>. Both thermometers showed some R(0°C) changes after that calibration: from BIPM and NPL calibrations, thermometer 226322 showed a R(0°C) difference of  $\pm$  150  $\mu\Omega$  in the period 1976-1980. BIPM is supplying R(0°C) values during the intercomparison time, ranging randomly by about 80  $\mu\Omega$  with thermometer 226322 and steadily increasing by about  $\pm$  200  $\mu\Omega$  with thermometer 226321.
- <u>I M G C</u> : thermometer PL01-6, PL02-6 and 45. The IMGC thermometer 838, of the NPL group, was very unstable after return to IMGC. Therefore IMGC used the three PRMI thermometers of the NPL group, which had been available since 1978. Thermometer PL01-6 showed, shortly after arrival at IMGC, a R(0°C) instability equivalent to about a 1 mK change, while the other two remained stable within 0.2 mK; after this R(0°C) change of thermometer PL02-6 reported to have occurred at NPL, PRMI calibrations of PL01-6 and PL02-6 agreed very well below 54 K, differing from that of thermometer 45 by about 1 mK, while above 60 K, thermometer PL02-6 disagreed by about 1 mK with respect to the other two<sup>24</sup>. On the other hand, NPL calibration of thermometer

PLO1-6 seemed to have suffered from the  $R(0^{\circ}C)$  change, below 30 K. In addition, thermometer 45 showed a very large overheating, as it was also observed at NPL, which limits its reliability. In conclusion, traceability to NPL measurements in certainly worse than the required  $\pm$  0.1 mK level.

- <u>INM</u> : thermometer 232788 and 1812283. The former was used for many of the measurements. The relationship with thermometer 1812283 of the NPL group was supplied at Ar and  $O_2$  triple-point temperatures. However, R(0°C) for the latter thermometer was observed to change with time, by + 280  $\mu\Omega$  between 1975 and 1979, recovering back by 100  $\mu\Omega$  in 1982. These facts certainly lower the traceability to NPL measurements.
- <u>N B S</u> : thermometer 1774095. It was used for all the measurements. The relationship to NBS thermometer 1812282 of the NPL group was supplied at the triple points of argon  $(0.2 \pm 0.1 \text{ mK})$  and oxygen  $(0.1 \pm 0.1 \text{ mK})$ . Both thermometers are reported to have been stable within  $\pm 0.1 \text{ mK}$  for years.
- <u>N I M</u> : thermometer 7709, 7703 and 188640. The last-mentioned thermometer holds a NIM-IPTS-68 calibration above 54 K. Relationship to NPL measurements could be obtained only for thermometer 7709, through a comparison performed in 1982 with thermometer 1812283.
- <u>N M L</u> : thermometer 1731676. This thermometer was used for all the measurements and belongs to the NPL group. It is reported to have been stable within  $\pm$  0.1 mK.
- <u>N P L</u> : thermometer 1728839. This thermometer is the "master" of the NPL group and it is reported to have been stable within  $\pm$  0.1 mK.
- <u>N R C</u> : thermometer 1521389 and 1872179. The latter has been used only for the last measurements and it is reported to agree with the former within  $\pm$  0.1 mK at 63 K and  $\pm$  0.25 mK at 18 K. Changes in calibration of the former (up to 300  $\mu$ 0 of R(0°C) since 1976) have been traced back. None of these thermometers belongs to the NPL group but the relationship with NRC thermometer 1158062 of the NPL group is provided at the relevant reference temperatures. NRC also pointed out that the systematic differences found at NPL between thermometers 1158062 and 1158066 were not confirmed by measurements made at NRC before and after the NPL comparison, although both were perfectly stable in time<sup>25</sup>. These facts lower the traceability to NPL measurements.
- $\frac{N \ R \ L \ M}{1978}$ : thermometer 7681 (1981). No reference to thermometers used in 1978 was possible. Neither thermometer 6601 nor 6803 of the NPL group could be used. Thermometer 7681 was compared with NPL-IPTS-68 in 1982; traceability is stated to be ± 0.3 mK.

<u>P R M I</u> : thermometer 1842381. Since PRMI thermometers of the NPL group were at IMGC at the time of the intercomparison, PRMI used a thermometer calibrated at ASMW against thermometer 217997 of the NPL group, with a stated accuracy of  $\pm$  0.5 mK. However, W(100°C) has only been estimated<sup>26</sup>: at IMGC this value has been set, in the calibration table, to 1.39270, using Seifert's criterion<sup>54</sup>. These facts lower the traceability to NPL measurements; scale non-uniqueness is also involved for the secondary-point temperature calculations. At PRMI thermometers 8, 14 and 17 were also used: results on cell differences will use a mean value from the four thermometers.

A detailed discussion on the resulting estimated accuracy of temperature values is given in Section VII.2.

- 4. LAB-IPTS-68 realizations (status during the intercomparison)
- <u>A S M W</u>: The Scale is maintained above 84 K, using the triple point of argon realized in a conventional way. In the future, sealed cells will be used for the Scale definition.
- <u>B I P M</u>: The Scale is maintained above 84 K, using the triple point of argon in a sealed cell.
- <u>IMGC</u>: The Scale is maintained above 54 K, using the triple point of argon and sealed-cell realizations of the fixed points. Below 54 K triple points are available in sealed cells.
- <u>INM</u>: The Scale is maintained above 54 K, using the triple point of argon and sealed-cell realizations of the fixed points. Below 54 K triple points are available in sealed cells.
- <u>N B S</u> : The Scale is defined over the whole range by a group of standard thermometers. The Scale is NBS-1955 adjusted according to its relationship to IPTS-68 prescribed by the CCT  $^{64}$ .
- <u>NIM</u>: The Scale is maintained above 54 K with an accuracy of 1 to 3 mK.The condensation point of oxygen is used. In the future sealed-cell realizations will be used.
- <u>N M L</u> : The Scale is maintained over the whole range on a set of standard thermometers, calibrated on recent conventional realizations of the fixed points. The condensation point of oxygen is used.
- <u>N P L</u> : The Scale is maintained over the whole range on a set of standard thermometers, calibrated on recent conventional realizations of the fixed points. The condensation point of oxygen is used.
- <u>NRC</u> : The Scale is maintained over the whole range on a set of standard thermometers, calibrated on recent conventional and sealed-cell realizations of fixed points. The condensation

point of oxygen is used. A set of fixed points in sealed cells (triple points) is also available.

- <u>N R L M</u>: The Scale NRLM-80 is defined by a set of calibrated thermometers. The condensation point of oxygen is used. A set of fixed points in sealed cells (triple points) is also available.
- <u>PRMI</u>: The Scale is maintained over the whole range on a set of standard thermometers, calibrated on recent conventional realizations of the fixed points. The condensation point of oxygen is used. For some triple points a sealed-cell realization is also available.

### V. RESULTS ON CELL INTERCOMPARISON

Some 300 meltings (with 2300 equilibrium temperatures) have been originated by the 11 Laboratories, each Laboratory having measured several cells for each substance, each cell having been measured by several Laboratories and each substance having been sealed in several cells.

This Section will supply the whole original data, so that everyone may have access to them as they were available to the Editor and will be able to make his own calculations and speculations.

However, it was decided not to present merely a collection of the graphs and tables provided by the Laboratories, especially as they were originally not given in a uniform way.

Consequently, data on cell intercomparison have been organized in two different ways:

The first consists of a series of 57 data sheets wherein the 151 sets of independent data originated by the Laboratories are collected. There is one sheet for each of the 40 cells (one for each substance, in the case of the multicomponent cells), with an additional sheet in the case when there are more than five Laboratories having measured the cell. Each sheet contains a drawing of the cell and a Table with the physical and filling characteristics. Then, data of a typical melting plateau from each Laboratory (selected, when more then one was available) are given in the form W versus the melted fraction F: the W(100%) value at F=1 is taken as the definition value for the triple-point measurements of that Laboratory on that cell and substance, and it is used in the subsequent analysis of results. Sometimes, W(100%) does not come from extrapolation of all the experimental points, since there was evidence of steady overheating of the liquid phase or of anomalous reversal of slope for high melted fractions; this evidence comes only from analysis of the plot of the melting plateau as W versus 1/F, or from comparison of different meltings.

Therefore, a second representation of the data is given which is complementary to the former. Each data sheet is followed by a plot of <u>all</u> the meltings made with that cell by <u>all</u> the Laboratories. Plots are given as <u>differences</u> to the definition W(100%) value, so that systematic differences between Laboratories are cancelled out. These plots present the variability in the shape of meltings, which represents one of the limiting reasons for accuracy in the determination of the triple-point temperature.

The data sheets contain also some ancillary data on thermal behaviour of the equipment and cell (which will be defined later on and discussed in Section VII.1.2.b-c) and collect some information useful for calculations, such as  $R(0^{\circ}C)$  of the thermometer used and the corresponding temperature values in the LAB and NPL Scales, which will be examined in Section VI.

The data sheets (and associated plots) are grouped for each gas,

giving realizations of the definition points of the IPTS-68 first, then of secondary points, for decreasing temperatures. The cells for each substance are presented in the alphabetical order of the Laboratories.

Definition of parameters in the data sheets

- <u>Cell drawing</u>: The drawing is intended only to show the design geometry of the cell, with its overall dimensions.
- Sealing date: indicates the ageing of the sample in the cell; the gas could have been bottled by the manufacturer in the cylinder used by the Laboratory significantly earlier.
- <u>Cell total mass</u>: is the mass of the filled cell: when no wires are permanently glued on the body, it is reproducible within l mg and can be used to check for leaks with time, by weighing.
- Sample mass: is the mass of the sealed sample; with some kind of sealed cells, it is possible to obtain this value very accurately by difference of the weights of the filled and empty cell.
- Impurity analysis: Nominal purity means purity specification given by the manufacturer's catalogue; most of the reported analyses are actually batch analyses, not specific to the bottle used.
- Enthalpy of melting: is the value calculated from the mass of the sample and literature data on enthalpy of melting.
- Thermometer: is the thermometer actually inserted in the cell for the measurements; the R(0°C) value indicated is the actual value at the time of the measurements and has been used in order to obtain W values.
- Typical melting plateau: This has been selected from the meltings reported by each Laboratory (when more than one). The number of points has been sometimes limited to eleven for practical reasons.
- Temperature values: They are included in the sheets in order to complete the collection of data received from the Laboratories. For definition of their meaning see Section VI.
- <u>Average drift</u>: is the average value of thermal drift observed by the thermometer inserted in the cell when the cell is left unheated just before and after melting. It is determined by the residual heat exchange of the cell with the cryostat: this heat flows through the cell all the time during the melting.
- Recovery time: is defined here as the time required for the temperature of the cell (as measured by the thermometer) to recover within 0.1 mK its equilibrium value, after a heating cycle. Since overheating depends on the heating power, recovery time slightly depends on it, especially for small values (fast cells). Part of the reason for the quite large variability observed for this parameter is the lack of uniqueness in the interpretation of this parameter in different laboratories.
- <u>Overheating</u>: indicates the rise in temperature during the heating periods, as observed by the thermometer. Since it is strongly dependent on the melted fraction (it increases with the melted fraction), the value at F = 50% is considered and it is referred to the heating power, as it depends on that too.
- Enthalpy of melting (end of the sheet): refers to the value actually obtained by the Laboratories during the melting experiments.

#### GRAPHS

- Temperature differences: For each of the melting plateaux the plots show the difference between the temperature of each equilibrium point and the value at 1/F = 1; this value corresponds to the W(100%) value given in the sheets. Therefore, systematic differences between Laboratories are cancelled out.
- Symbols: One symbol has been used for each Laboratory: it is a letter taken from its acronym and it is used rotated to distinguish between different meltings made by the same Laboratory: W (ASMW); B (BIPM); G (IMGC); N (INM); S (NBS); M (NIM); L (NML); P (NPL); R (NRLM); C (NRC); V (PRMI).

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# DATA SHEETS FOR THE CELLS

Definition points: 1. argon

2. oxygen

3. e-hydrogen

Secondary points: 1. methane

2. nitrogen

3. neon

4. e-deuterium

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# CELL TYPE: 4 AR ASMW

International Intercomparison CCT: 1979-1983



Manufacturer: ASMW

Sealing date: May 1981

cell total mass: 80 g

sample mass: 0.014 mol

Filling gas type: R 50 Technische Gase

impurity analysis: < 10 ppm</pre>

Enthalpy of melting: 16.5 J

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Measurements at Resistance Ratio (W)

LABORATORY	ASMW	IMGC		
Date	Jun 81	Oct 82		
thermometer N°	217278	PL01-6		
Ro (ohm)	25.346860	25.271140		
Typical melting plateau	10%      2237        20%      2308        30%      2324        40%      2328        50%      2348        60%      2356	6 % 2654 12% 2689 19% 2709 25% 2719 38% 2733 50% 2749		
melted fraction F	70% 2356 80% 2415	77% 2780		
W(100%)	0.21602447	0.21602772		
T (K) LAB NPL	83.79758 83.79731	83.79806 83.79729		
average drift (mK/h)	<3	5		
to 0.1mK (min)	1-3	1-3		
overheating at 50% (mK/mW)	0.26	1		
enthalpy of melting (J)	16.5	15		



#### CELL TYPE: 3 AR BIPM

International Intercomparison CCT: 1979-1983



Sealing date: Feb 1977

cell total mass: 315 g

sample mass: 0.18 mol

Filling gas type: Air Liquide

impurity analysis: nom. 99.9995%
 nitrogen 3 vppm
 oxygen <1
 methane <0.5</pre>

Enthalpy of melting: 240 J

Resistance Ratio (W)

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-135-	
	-C
	-^

at	
LABORATORY	BIPM
Date	Feb 80
thermometer N°	226321
Ro (ohm)	25.369110

Measurements

21%	3852
38%	3872
62%	3872
80%	3872
	21% 38% 62% 80%

melted
fraction
F

	W(1	.00%)	0.21603920
Т	(K)	LAB NPL	ref 83.79670

average drift	
(mK/h)	-1.5
recovery time	
to 0.1mK (min)	2-100
overheating	
at 50% (mK/mW)	0.3
enthalpy	
of melting (J)	241

Notes:

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International Intercomparison CCT: 1979-1983 pag.1

Manufacturer: IMGC

Sealing date: Jul 1975

cell total mass: 190 g

sample mass: 0.17 mol

Filling gas type: SIO-Air Liquide

impurity analysis: nom. 99.9997%
nitrogen a) 1.85; b) 2.8 vppm
oxygen 0.95 0.5
carb.dioxide <0.5 none
hydrocarbons none</pre>

Enthalpy of melting: 195 J

Measurements at

-023

140

Mod. A : 1 well

Mod.B: 3 wells

Resistance Ratio (W)

LABORATORY	ASMW	IMGC	NIM	NPL	NRC
Date	Nov 79	Sep 75-May 80	Feb 81	Jan 78	Dec 76
thermometer N°	217990	PL01-6	188640	1728839	1521389
Ro (ohm)	24.186290	25.271140	24.164330	25.559570	25.523332
Typical melting plateau melted fraction F W(100%)	5.5% 0770 9.5% 0810 19% 0865 27% 0880 37% 0880 50% 0875 63% 0880 76% 0880 91% 0890 0.21600890	6 % 2528 10% 2551 15% 2579 18% 2595 23% 2603 27% 2634 32% 2662 36% 2682 44% 2694 53% 2702 62% 6710 0.21602745	12% 0312 23% 0346 35 0321 46% 1099* 58% 1182* 70% 1463* 81% 1641* * not used 0.21650500 <sup>(1)</sup>	4 % 0063 6 % 0083 23% 0171 29% 0183 38% 0182 46% 0187 55% 0175 59% 0184 64% 0196 74% 0175 87% 0196 0.21610195	11% 7823 40% 7921 80% 7960 92% 8003
T (K) LAB NPL	83.79770 83.79745	ref 83.79723	83.79960	83.79690 83.79690	83.79776 83.79666
average drift (mK/h) recovery time (min) overheating at 50% (mK/mW)	2 2-10 0.05	2 2-3 0.04	5 2-25 0.8	<15	
enthalpy of melting (J)	197	195	190		

Notes: (1) 0.21652080 using all data (NIM).

	С	E	L	L	Т	Y	Ρ	E:	1	AR	IMG	3
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International Intercomparison CCT: 1979-1983 pag.2



Manufacturer: IMGC

Sealing date: Jul 1975

cell total mass: 190 g

sample mass: 0.17 mol

Filling gas type: SIO-Air Liquide

impurity analysis:	nom. 99	9.9997%
nitrogen a)	1.85 ; 1	) 2.8 vppm
oxygen	0.95	0.5
carb.dioxide	<0.5	none
hydrocarbons		none

Enthalpy of melting: 195 J

Resistance Ratio (W)

LABORATORY NRLM	
Date Nov 81	
thermometer N° 7681	
Ro (ohm) 25.3632	54

Measurements

at

Typical malting	6 % 18% 44% 48%	1174 1290 1362
plateau	79%	1384
Plateau	83%	1386
melted fraction F		
W(100%)	0.216	511388
	07 70	070

Ľ	(K)	LAB	83.79878
		NPL	83.79828

average drift				
(mK/h)	2.5			
recovery time				
to 0.1mK (min)	3-8			
overheating				
at 50% (mK/mW)				
enthalpy				
of melting (J)	202			

Notes:



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2	2 AR IMGC			International Intercomparison CCT: 1979-1983 pag.l
		Manufacturer:	IMGC	

Sealing date: May 1978

167.419 g <sup>(1)</sup> cell total mass:

sample mass: 0.08915 mol <sup>(1)</sup>

Filling gas type: SIO-Air Liquide

impurity analysis: nom. 99.9997% nitrogen a) 1.9; b) 2.4 vppm 1.0 0.2 oxygen carb.dioxide <0.5 none hydrocarbons none

Enthalpy of melting: 106 J

Resistance Ratio (W)

LABORATORY BIPM IMGC INM NBS NML Date Dec 79 May 78-May 80 Apr 80 Aug 79 May 79 thermometer N° 226321 PL01-6 232788 1774095 1731676 Ro (ohm) 25.369005 25.271140 25.087300 25.560840 25.522800 7 % 8 % 3685 2607 10% 3297 13% 4819 2.5% 0126 26% 3863 12% 2627 20% 3333 28% 4883 12% 0149 48% 3882 2647 30% 3349 44% 4918 19% 0152 20% Typical 2663 60% 4931 melting 67% 3969 29% 40% 3361 22% 0162 plateau 90% 3980 36% 2679 50% 3369 75% 4946 27% 0170 50% 2702 60% 3381 91% 4980 44% 0192 59% 2710 70% 3401 65% 0198 2734 melted 74% 80% 3429 80% 0198 fraction 90% 3461 95% 0198 F W(100%) 0.21603980 0.21602738 0.21603380 0.21604960 0.21610195 T (K) LAB 83.79814 83.79798 83.79805 83.80030 83.79670 NPL 83.79680 83.79722 83.79732 83.79700 83.79698 average drift (mK/h)10 1 recovery time to 0.lmK (min) 15 5 60 40-60 overheating 24 (2) (2) 5 at 50% (mK/mW)0.02 enthalpy 106 of melting (J) 115 104

Notes: (1) this cell was returned to IMGC without sealing nut in May 1980, with no apparent contamination of the sample. (2) cell heated from inside the block.



CELL TYPE:

Measurements

at

## CELL TYPE: 2 AR IMGC

International Intercomparison CCT: 1979-1983 pag.2

Manufacturer: IMGC

Sealing date: May 1978

cell total mass: 167.419 g  $^{(1)}$ sample mass: 0.08915 mol <sup>(1)</sup>

Filling gas type: SIO-Air Liquide

impurity analysis: nom. 99.9997% oxygen a) 1.9 ; b) 2.4 1.0 0.2 nitrogen carb.dioxide <0.5 none hydrocarbons none

Enthalpy of melting: 106 J

Resistance Ratio (W)



at	
LABORATORY	PRMI
Date	Dec 81
thermometer N°	1842381
Ro (ohm)	25.544950

Measurements

			10%	9262
			21%	9264
	Typic	cal	31%	9269
1	melt:	ing	41%	9292
	plate	eau	52%	9321
			72%	9325
			93%	9308
	melto fract F	ed tion		
	W(10	0%)	0.21	589320
T	(K)	LAB NPL	83.7	9693 <sup>(2)</sup>

average drift	
(mK/h)	<1
recovery time	
to 0.1mK (min)	20
overheating	
at 50% (mK/mW)	0.06
enthalpy	
of melting (J)	98.8

Notes: (1) this cell was returned to IMGC without sealing nut in May 80, with no apparent contamination of the sample. (2) through ASMW thermometer. 46 -



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Manufacturer: INM

Sealing date: Sep 1975

cell total mass: 265 g

sample mass: 0.268 mol

Filling gas type: Air Liquide

impurity analysis: nom. 99.9995%
 oxygen <1 vppm
 nitrogen 3
 hydrocarbons <0.5
 carbon oxide <0.5</pre>

Enthalpy of melting: 276 J

Resistance Ratio (W)

LABORATORY	BIPM	INM	NML	NRC
Date	Nov 79	Apr 80	May 79	Dec 76
thermometer N°	226321	232788	1731676	1521389
Ro (ohm)	25.368966	25.087300	25.522800	25.523332
Typical melting plateau melted fraction F	7 % 3450 20% 3837 34% 3955 67% 3971 90% 3979	10%334120%334530%334940%335350%335360%335370%335380%336590%3373	5 % 0329 10% 0317 16% 0305 30% 0321 40% 0344 50% 0364 65% 0372 75% 0364 85% 0337	3 % 7940 10% 7979 25% 7979 49% 7991 74% 8042 89% 8026 94% 8050
W(100%)	0.21603990	0.21603355	0.21610340	0.21598038
T (K) LAB NPL	83.79817 83.79690	ref 83.79727	83.79710 83.797362	83.79793 83.79683
average drift (mK/h) recovery time	<1	<2		
to 0.1mK (min)	22	10-20		
at 50% (mK/mW)	1.8	2.7		
enthalpy of melting (J)	162	276		301





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Measurements at



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### CELL TYPE: XXI AR INM

Manufacturer: INM

Sealing date: Dec 1978

cell total mass: 284.36 g

sample mass: 0.130 mol

## Filling gas type:

impurity analysis: oxygen <1 vppm nitrogen <3 methane <0.5 carbon oxide <0.5</pre>

Enthalpy of melting: 160 J

Measurements at

128

Resistance Ratio (W)

LABORATORY	INM	NIM
Date	Jan 79	Aug 81
thermometer N°	1812283	7703
Ro (ohm)	25.494778	24.899378

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. 8

C

	0 %	6234 6253	9 % 18%	4668
Typical	20%	6273	27%	4709
melting	30%	6297	36%	4721
plateau	40%	6309	45%	4737
	50%	6321	55%	4798
	60%	6329	64%	4821
melted	70%	6333	73%	4829
fraction	80%	6340	82%	4841
F	90%	6344	91%	4845
W(100%)	0.21	596344	0.216	514760
T (K) LAB	83.79	9802		
NPL	83.7	9729		

average drift		
(mK/h)	<2	<10
recovery time		
to 0.1mK (min)	6	1-3
overheating		
at 50% (mK/mW)		0.2
entnaipy		
of melting (J)	160	120

Notes:



#### CELL TYPE: BCM4 INM (Ar)

International Intercomparison CCT: 1979-1983

Manufacturer:INM<br/>Multicomponent cell<br/>(argon,oxygen,neon,nitrogen)Sealing date:Jan 1982

cell total mass: 369.83 g

sample mass: 0.0218 mol

### Filling gas type:

impuri	ity analys:	is:	
	oxygen	0.2	vppm
	nitrogen	0.5	
	methane	0.1	
	water	0.5	
	water	0.5	

Enthalpy of melting: 25.7 J

Measurements at

5

Resistance Ratio (W)

LABORATORY	INM	NRC
Date	Feb 82	Jul 82
thermometer N°	1812283	1521389
Ro (ohm)	25.494713	25.523332

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INM mod. BCM

C :

			0 %	6258	3.1%	7867
			10%	6316	7.6%	7907
	Typic	al	20%	6328	16%	7987
	melti	ing	30%	6324	34%	8027
	plate	au	40%	6348	70%	8067
			50%	6360		
			60%	6360		
	melte	ed	70%	6369		
	fract	tion	80%	6369		
	F		90%	6405		
	W(100	0%)	0.21	596405	0.215	98077
т	(K)	T.AB	83.79	9816	83.79	802
Ť		NPL	83 70	3743	83 79	695
		السلك ملك 7 مل	JJ. / .			0/2

average drift		
(mK/h)	3	
recovery time		
to 0.1mK (min)	8.5	30
overheating		
at 50% (mK/mW)		
enthalpy		
of melting (J)	25.7	26.8

Notes:



International Intercomparison CCT: 1979-1983

Manufacturer: NBS

Sealing date: Feb 1978

cell total mass: 357 g

sample mass: 0.21 mol

Filling gas type:

impurity analysis: nom. 99.9999%

Enthalpy of melting: 250 J

Resistance Ratio (W)

at					
LABORATORY	BIPM	INM	NBS	NRC	NRLM
Date	Feb 80	Apr 80	Feb 79	Aug 79	May 82
thermometer N°	226321	232788	1774095	1521389	1781356
Ro (ohm)	25.369062	25.087300	25.560840	25.523332	25.525818
Typical melting plateau melted fraction F	8 % 3952 27% 3991 45% 4039 67% 4031 90% 4070	10%336120%336930%339340%339350%339360%339370%341780%344590%3461	3.1% 4908 14% 4924 24% 4939 35% 4953 46% 4951 57% 4961 67% 4971 78% 4968 89% 4965	5 % 7912 10% 7936 20% 7947 41% 7959 60% 7947	7.3% 9594 16% 9661 25% 9665 30% 9688 37% 9676 42% 9684 67% 9665 77% 9696
W(100%)	0.21604070	0.21603395	0.21604965	0.21597961	0.21599700
T (K) LAB NPL	83.79834 83.79700	83.79809 83.79736	83.80032 83.79700	83.79775 83.79665	83.79885 83.79835
average drift (mK/h) recovery time	0.4				0.1
to 0.1mK (min)	30	1-6			30-180
at 50% (mK/mW)	1	0.1			
enthalpy of melting (J)	222		250	255	253

#### Notes:



NBS (Ar)

Measurements



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International Intercomparison CCT: 1979-1983

### Manufacturer: NIM

Sealing date: Dec 1980

cell total mass: 193 g

sample mass: 0.06 mol

# Filling gas type:

impurity analysis: nitrogen 4 vppm oxygen 1 carb.diox.<1 hydrocarb.<1; hydrogen <1</pre>

Enthalpy of melting: 57 J

Resistance Ratio (W)



NIM

LABORATORY	NIM
Date	Aug 81
thermometer N°	7703
Ro (ohm)	24.899378

Measurements at

			5%	4661
			10%	4685
	Typic	cal	19%	4705
	melt:	ing	30%	4721
	plate	eau	48%	4737
			60%	4745
			71%	4753
	melte	ed	82%	4757
	fract	tion	88%	4761
	F		3	
	W(10	0%)	0.21	614755
T	(K)	LAB NPL		

average drift	
(mK/h)	<10
recovery time	
to 0.1mK (min)	1-3
overheating	
at 50% (mK/mW)	0.15
enthalpy	
of melting (J)	57

Notes:



International Intercomparison CCT: 1979-1983 pag.1

# Manufacturer: NRC

Sealing date: May 1979

cell total mass: 200 g

sample mass: 0.05943 mol

Filling gas type: Matheson

impurity analysis: research grade

Enthalpy of melting: 70 J

Resistance Ratio (W)

at					
LABORATORY	BIPM	IMGC	INM .	NBS	NML
Date	Mar 80	Apr 80	Apr 80	Feb 81	Nov 79
thermometer N°	226321	PL01-6	232788	1774095	1731676
Ro (ohm)	25.369098	25.271140	25.087300	25.560840	25.522800
Typical melting plateau	4.5% 3850 14% 3992 24% 4044 90% 3969	10%274515%277319%278524%279327%279531%279742%2793	10% 3218 20% 3301 30% 3313 40% 3317 50% 3321 60% 3325 70% 3329	6.5% 4868 18% 4926 32% 4935 50% 4952 58% 4955 72% 4954 85% 4955	5 % 0246 10% 0297 15% 0321 20% 0323 30% 0340 40% 0344 50% 0352
melted fraction F		55% 2795 62% 2797 85% 2808	80% 3337 90% 3345	98% 4953	70% 0359 75% 0376 80% 0399
W(100%)	0.21604000	0.21602805	0.21603335	0.21604955	0.21610370
T (K) LAB NPL	83.79818 83.79690	83.79814 83.79736	83.79795 83.79722	83.80028 83.79696	83.79717 83.79739
average drift (mK/h) recovery time	0.4	1			
to 0.1mK (min)	1	1-2	0.5-5		
overheating at 50% (mK/mW)	0.13	0.35	0.25		
enthalpy of melting (J)	81	65		70	

NRC

Measurements

International Intercomparison CCT: 1979-1983 pag.2

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Manufacturer: NRC

Sealing date: May 1979

cell total mass: 200 g

sample mass: 0.05943 mol

Filling gas type: Matheson

impurity analysis: research grade

# Enthalpy of melting: 70 J

Measurements at		Resistance	Ratio	(W)
LABORATORY	NPL	NRC		
Date	Nov 81	Jun 79		
thermometer N°	1728839	1521389		
Ro (ohm)	25.559570	25.523332		
Typical melting plateau melted fraction F	7 %    0190      14%    0210      21%    0210      28    0208      35%    0210      45%    0210      60%    0210      76%    0210      97%    0210	5 % 7779 10% 7873 22% 7912 31% 7947 48% 7959 74% 7959 90% 7959		
W(100%)	0.21610210	0.21597959		
T (K) LAB NPL	83.79695 83.79695	83.79775 83.79665		
average drift (mK/h) recovery time to 0.1mK (min) overheating at 50% (mK/mW)	0.3 5	6		
enthalpy of melting (J)		70		

Notes:



NRC



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Manufacturer: NRC

Sealing date: May 1979

cell total mass: 228 g

sample mass:

Filling gas type: Matheson

impurity analysis: research grade

Enthalpy of melting: 72 J

(W)

Measurements at		Resistance	Ratio
LABORATORY	NIM	NRC	
Date	Aug 81	Jul 80	
thermometer N°	7703	1521389	
Ro (ohm)	24.899378	25.523332	
Typical melting plateau melted fraction	5 % 4617 10% 4713 19% 4738 27% 4758 51% 4797 60% 4801 68% 4809 76% 4814 84% 4814	2.7% 7751 3.6% 7786 5.4% 7822 7.2% 7849 11% 7896 22% 7970 43% 7970 86% 7970	
W(100%)	0.21614820	0.21597970	
T (K) LAB NPL		83.79775 83.79665	
average drift (mK/h) recovery time	<10		
to 0.lmK (min) overheating	1-2	<16	
at 50% (mK/mW)	0.3		
enthalpy of melting (J)	63	71.5	

Notes:



NRC



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Manufacturer: NRLM

Sealing date: Jun 1978

cell total mass: 170 g

sample mass: 0.063 mol

Filling gas type:

impurity analysis: tot. 0.8 ppm

Enthalpy of melting: 74.6 J

Measurements at		Resistance	Ratio	(W)
LABORATORY	NBS	NRLM	2	
Date	Jul 81	Jul 78		
thermometer N° Ro (ohm)	1774095 25.560840	(1)		

			5.6%	4829	16%	6210
			20%	4959	28%	6210
	Typic	cal	31%	4958	41%	6220
	melt:	ing	41%	4959	54%	6280
	plate	eau	52%	4952	67%	6240
			63%	4957	81%	6280
			74%	4958	93%	6280
	melt	ed	84%	4962		
	fract	tion	95%	4967		
	F					
	W(10	0%)	0.216	04960	0.216	506270
т	(K)	I.AB	83.80	030		
		NPT.	83.79	698		
		1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1		~~~		

average drift (mK/h)		
recovery time		
co U.IMK (MIN) overheating	2	
at 50% (mK/mW)		
enthalpy		
or metting (J)	15	74.6

Notes: (1) 1978 data are reported only for record.

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NRLM



## CELL TYPE: 7803 AR NRLM

International Intercomparison CCT: 1979-1983 pag.1

### Manufacturer: NRLM

Sealing date: Jun 1978

cell total mass: 170 g

sample mass: 0.06 mol

Filling gas type:

impurity analysis: tot. 0.8 ppm

NRLM

Enthalpy of melting: 67 J

Measurements at		Resista	nce Ratio (W)	)	
LABORATORY	ASMW	BIPM	IMGC	INM	NML.
Date	Nov 80	Dec 79	Sep 80	Apr 80	Jul 79
thermometer N°	217997	226321	PL01-6	232788	1731676
Ro (ohm)	25.418540	25.368956	25.271140	25.087300	25.522800
Typical melting plateau melted fraction F	10%    0210      30%    0281      50%    0320      70%    0352      80%    0352      90%    0332      95%    0360      98%    0340	9 % 3893 27% 3900 45% 3963 67% 4023 90% 3987	5 % 2690 8 % 2725 12% 2757 17% 2773 21% 2785 25% 2797 31% 2801 45% 2824 67% 2836	10%    3297      20%    3313      30%    3329      40%    3361      50%    3389      60%    3389      70%    3389      80%    3389      90%    3401	5 % 0325 10% 0333 15% 0356 20% 0364 30% 0395 40% 0431 50% 0446 65% 0454 75% 0474 90% 0501
W(100%)	0.21600360	0.21604000	0.21602825	0.21603400	0.21610510
T (K) LAB NPL	83.79760 83.79735	83.79818 83.79690	83.79817 83.79739	83.79811 83.79738	83.79730 83.79760
average drift (mK/h) recovery time	2.5	1	5		
to 0.1mK (min) overheating		2	1	2-23	
at 50% (mK/mW)	0.4	0.6	0.1-0.5	0.8	
enthalpy of melting (J)	55.4	63	60		

Notes:



### CELL TYPE: 7803 AR NRLM

International Intercomparison CCT: 1979-1983 pag.2

### Manufacturer: NRLM

Sealing date: Jun 1978

cell total mass: 170 g

sample mass: 0.06 mol

Filling gas type:

impurity analysis: tot. 0.8 ppm

NRLM

-8

Enthalpy of melting: 67 J

Measurements at		Resista	nce Ratio (W)	
LABORATORY	NRC	NRLM	NRLM	
Date	May 79	Oct 78	Nov 81	
thermometer N°	1521389	(1)	7681 25.362937	
Ro (ohm)	25.523332			
Typical melting plateau melted fraction F	5 % 7838 10% 7885 20% 7912 40% 7959 60% 7959 80% 7955	9 %    6170      27%    6230      36%    6220      56%    6280      68%    6290      77%    6300      91%    6270      94%    6310	9.8% 1305 17% 1354 46% 1377 53% 1379 82% 1359 89% 1377	
W(100%)	0.21597961	0.21606280	0.21611377	
T (K) LAB NPL	83.79775 83.79665		83.79875 83.79825	
average drift (mK/h) recovery time to 0.1mK (min) overheating at 50% (mK/mW)	8			
enthalpy of melting (J)	64	66.7		

Notes: (1) 1978 data are reported only for record.



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### CELL TYPE: PRMI (Ar)

International Intercomparison CCT: 1979-1983



PRMI

<u>Manufacturer</u>: PRMI Multicomponent cell (argon,oxygen,neon,hydrogen) <u>Sealing date</u>: Dec 1978

cell total mass:

sample mass:

Filling gas type:

impurity analysis: nitrogen <10 vppm oxygen < 2 carb.dioxi.< 3</pre>

Enthalpy of melting: 102.5 J

Resistance Ratio (W)

Measurements at	
LABORATORY	PRMI
Date	Nov 81
thermometer N°	1842381
Ro (ohm)	25.544950

	15%	9169
	30%	9277
Typical	45%	9254
melting	61%	9258
plateau	• 76%	9248
	91%	9263
melted		

fraction F

W(100%)		0%)	0.21589295	
Т	(K)	LAB	(1)	

NPL 83.79672<sup>(1)</sup>

average drift	
(mK/h)	1
recovery time	
to 0.1mK (min)	15
overheating	
at 50% (mK/mW)	0.09
enthalpy	
of melting (J)	102.5

Notes: (1) through ASMW thermometer.



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International Intercomparison CCT: 1979-1983 pag.1

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Manufacturer: IMGC Sealing date: Sep 1976 cell total mass: 190 g sample mass: 0.22 mol Filling gas type: SIO-Air Liquide impurity analysis: nom. 99.998% nitrogen 8 vppm argon <10 krypton+xenon <1</pre>

Enthalpy of melting: 96 J

Measurements at Resistance Ratio (W)

LABORATORY	ASMW	IMGC	NIM	NPL	NRC
Date	Feb 80	Sep 76-Nov 81	Feb 81	Feb 78	Dec 76
thermometer N°	217997	PL01-6	7709	1728839	1521389
Ro (ohm)	25.418580	25.271140	25.352963	25.559570	25.523332
Typical melting plateau melted fraction F	12% 7380 30% 7860 48% 7840 72% 7850 90% 7790	7 % 1655 10% 1675 12% 1698 17% 1717 20% 1730 30% 1750 45% 1754 65% 1758	5 % 3116 10% 3155 15% 3175 20% 3274 30% 3274 40% 3273 51% 3286 71% 3290	17%    6389      18%    6393      28%    6387      48%    6377      59%    6378      77%    6380      88%    6382	8 % 4526 16% 4734 46% 4746 77% 4746 96% 4757
W(100%)	0.09187880	0.09191770	0.09183293	0.09196382	0.09184746
T (K) LAB NPL	54.36090 54.36093	ref 54.36132	54.36360 54.36125	54.36072 54.36072	54.36108 54.36091
average drift (mK/h) recovery time to 0.lmK (min) overheating at 50% (mK/mW)	2-12 0.35	5 1-10 0.3	3 2-5 0.7	<5	
enthalpy of melting (J)	96	105	91		
### CELL TYPE: 1 02 IMGC

International Intercomparison CCT: 1979-1983 pag.2



Manufacturer: IMGC

Sealing date: Sep 1976

cell total mass: 190 g

sample mass: 0.22 mol

Filling gas type: SIO-Air Liquide

impurity analysis: nom. 99.998%
 nitrogen 8 vppm
 argon <10
 krypton+xenon <1</pre>

Enthalpy of melting: 96 J

Resistance Ratio (W)

LABORATORY	NRLM			
Date	Nov 81			
thermometer N°	7681			
Ro (ohm)	25.363060			
	22% 1188			

Measurements at

Ο

Typical melting plateau	25% 52% 57% 82% 89%	1179 1196 1191 1205 1206
melted fraction F		
W(100%)	0.092	201210
T (K) LAB NPL	54.30 54.30	5160 5170
average drift (mK/h)	1.	. 6
to 0.1mK (min) overheating	1.	-8

at 50% (mK/mW) enthalpy of melting (J) 95.5



### CELL TYPE: 8 02 IMGC

International Intercomparison CCT: 1979-1983 pag.1

# Manufacturer: IMGC

Sealing date: Nov 1978

cell total mass: 168.316 g

sample mass: 0.10610 mol

Filling gas type: SIO-Air Liquide

impurity analysis: nom. 99.998%
 nitrogen 1.8 vppm
 argon 3.0
 methane 0.7

Enthalpy of melting: 47 J

Resistance Ratio (W)

Measurements at

.

LABORATORY	IMGC	INM .	NBS	NML	NRC
Date	Nov 78-May 81	Oct 80	Mar 84	Nov 78	Oct 79
thermometer N° Ro (ohm)	PL01-6 25.271140	232788 25.087300	1812282 25.510280	1731676 25.522800	1521389 25.523332
Typical melting plateau melted fraction F	11% 1900 18% 1914 24% 1924 32% 1932 63% 1952	10%332920%335330%336940%336950%336960%336970%340580%340090%3400	16% 4154 40% 4196 63% 4189 87% 4194	5 % 6561 10% 6573 17% 6581 25% 6593 30% 6597 37% 6601 50% 6605 60% 6613 80% 6585 95% 6581	4.5% 4756 5% 4840 8% 4820 10% 4847 18% 4851 20% 4867 36% 4890 40% 4902 71% 4910 90% 4918
W(100%)	0.09191956	0.09193405	0.09184211	0.09196593	0.09184920
T (K) LAB NPL	54.36148 54.36180	54.36163 54.36194	54.36196 54.36149	54.36143 54.36133	54.36153 54.36136
average drift (mK/h) recovery time to 0.1mK (min) overheating at 50% (mK/mW)	2 2-10 <sup>.</sup> 0.02	5-10 0.09			
enthalpy of melting (J)	46				48



CCT: 1979-1983

Measurements

Manufacturer: IMGC

Sealing date: Nov 1978

cell total mass: 168.316 g

sample mass: 0.10610 mol

Filling gas type: SIO-Air Liquide

impurity analysis: nom. 99.998%
 nitrogen 1.8 vppm
 argon 3.0
 methane 0.7

Enthalpy of melting: 47 J

Resistance Ratio (W)

, at		
LABORATORY	PRMI	3
Date	Dec 81	
thermometer N°	1842381	
Ro (ohm)	25.544950	
<u>Typical</u> melting plateau	10%469019%473029%475039%484048%476067%4780	
melted fraction F		
W(100%)	0.09174820	
T (K) LAB NPL	54.36059	
average drift (mK/h)	3	
to 0.1mK (min)	20	
at 50% (mK/mW)	0.01	
enthalpy of melting (J)	46.3	



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International Intercomparison CCT: 1979-1983

Manufacturer: INM

Sealing date: Feb 76

cell total mass: 265 g

sample mass: 0.279 mol

Filling gas type: Air Liquide

impurity analysis: nom. 99.998%
 nitrogen 5 vppm
 argon 12
 krypton 3

Enthalpy of melting: 124 J

Resistance Ratio (W)

										4
LABORATORY	INM		NIM		NML		NRC		NRC	
Date	May 80-Fe	eb 82	Aug 8	1	Sep 7	9	Jun	76	Nov 7	9
thermometer N°	232788		7709		17316	76	15213	89	15213	89
Ro (ohm)	25.0873	00	25.35	2963	25.52	2800	25.52	3332	25.52	3332
Typical melting plateau melted fraction F	10%       3         20%       3         30%       3         40%       3         50%       3         60%       3         70%       3         80%       3         90%       3	145 149 153 153 153 149 145 149 149	10% 20% 30% 50% 60% 70% 80% 90%	3211 3250 3293 3297 3297 3274 3297 3297 3297	5 % 10% 15% 20% 30% 40% 50% 60% 70% 80% 90%	6413 6456 6464 6475 6487 6495 6499 6464 6482 6498 6484	9.2% 25% 48% 72% 92%	4808 4840 4875 4901 4914	5 % 10% 20% 25% 30% 50% 56% 60% 62% 90%	4695 4730 4734 4742 4746 4738 4722 4710 4726 4734 4702
W(100%)	0.09193	155	0.091	83300	0.091	196500	0.091	84906	0.091	.84734
T (K) LAB NPL	ref 54.3613	1	54.36 54.36	360 127(1)	54.30 54.30	5112 5102	54.36 54.36	149 132	54.30 54.30	5105 5088
average drift (mK/h)	1.5		10	)						
to 0.1mK (min)	2-14		1-4	•						10
overheating at 50% (mK/mW)			0.	03	5.	. 8				
enthalpy										

of melting (J) 124

120

Notes: (1) through thermometer 1812283 (1982).



Measurements at



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С	Ε	L	L	Т	Y	Ρ	E:	BCM4	INM	(02)
0	-	_	-	-	-	-		DOLL		10-

International Intercomparison CCT: 1979-1983



INM mod. BCM

Manufacturer: INM Multicomponent cell (argon, oxygen, neon, nitrogen) Sealing date: Jan 1982 cell total mass: 369.83 g sample mass: 0.0222 mol Filling gas type: impurity analysis: 5 vppm; methane <0.2 nitrogen argon 12 ; hydrogen<0.1 krypt.+xenon 3 carbon diox. 0.2

Enthalpy of melting: 9 J

Measurements at Resistance Ratio (W)

LABORATORY	INM	NRC .
Date	Feb 82	Jul 82
thermometer N°	1812283	1521389
Ro (ohm)	25.494713	25.523332
Typical melting plateau melted	0 % 0958 10% 1103 20% 1099 30% 1087 40% 1111 50% 1111 60% 1111 70% 1111	6 % 4652 14% 4674 24% 4730 45% 4738 67% 4738 88% 4754
fraction F	80% 1123 90% 1146	
W(100%)	0.09181120	0.09184740
T (K) LAB NPL	54.36104 54.36135	54.36107 54.36090
average drift (mK/h) recovery time	-12	20
overheating at 50% (mK/mW)	6	30
enthalpy of melting (J)	9	8.9



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### CELL TYPE: M2 O2 NBS



Manufacturer: NBS

<u>Sealing date</u>: n.1 Feb 1983 n.3 Jul 1983 cell total mass: 240 g; 190 g

sample mass: 0.08 mol; 0.12 mol

Filling gas type: home-made from decomp. of KMnO4

impurity analysis:

Enthalpy of melting: 35 J; 53 J

Measurements at		Resistance	Ratio	(W)
LABORATORY	NBS (n°l)	NBS (n°3)		2
Date	Apr 1983	May 1983		
thermometer N°	1774095	1812282		
Ro (ohm)	25.560840	25.570340		
Typical melting plateau melted fraction F	6.7% 1904 16% 2087 25% 2098 34% 2099 45% 2096 67% 2088 93% 2074	2.1% 3631 12% 3913 23% 3967 33% 3963 43% 3960 53% 3958 63% 3953 73% 3950 83% 3939 93% 3943		
W(100%)	0.09192112	0.09183948		
T (K) LAB NPL	54.36145 54.36099	54.36130 54.36083		

average drift (mK/h) recovery time to 0.1mK (min) overheating at 50% (mK/mW)		
enthalpy of melting (J)	35	53



Manufacturer: NIM

Sealing date: Aug 1981

cell total mass: 156.39 g

sample mass: 0.020 mol

### Filling gas type:

impurity analysis: nitrogen 3.1 vppm 0.15 argon carb.diox.< 0.5 methane 0.15

Enthalpy of melting: 9 J

Resistance Ratio (W)



NIM mod. BC-INM

NIM

Date	Aug 81
thermometer N°	7709
Ro (ohm)	25.352963

Measurements at

LABORATORY

			11%	3234
			22%	3254
	Typic	cal	28%	3254
	melt:	ing	39%	3254
	plat	eau	50%	3254
			61%	3254
			72%	3293
	melt	ed	83%	3293
	frac	tion	94%	3293
	F			
	W(10	0%)	0.091	83293
т	(K)	LAB		(1)
		NPL	54.36	5125 <sup>(1)</sup>

average drift	
(mK/h)	
recovery time	
to 0.1mK (min)	1-2.5
overheating	
at 50% (mK/mW)	0.04
enthalpy	

of melting (J)

Notes: (1) through thermometer 1812283 (1982).



Manufacturer: NIM

Sealing date: Aug 1981

cell total mass: 176.38 g

sample mass: 0.020 mol

# Filling gas type:

impurity analysis: nom. 99.999%
 nitrogen 4.2 vppm
 argon 0.8
 methane 0.83
 carb.diox.0.5

Enthalpy of melting: 9 J

Resistance Ratio (W)



NIM mod. BC-INM

at	
LABORATORY	NIM
Date	Aug 81

Measurements

thermo	meter	N°	7709	
Ro	(ohm)		25.35296	3

			6.2%	3136
			12%	3163
	Typi	cal	19%	3187
	melt	ing	25%	3203
	plat	eau	31%	3211
			37%	3222
			44%	3222
	melt	ed	50%	3230
	frac	tion	56%	3234
	F		69%	3238
			87%	3246
	W(10	0%)	0.091	83246
Т	(K)	LAB NPL	54.36	113(1)

average drift	
(mK/h)	9
recovery time	
to 0.1mK (min)	1-4
overheating	
at 50% (mK/mW)	0.03
enthalpy	
of melting (J)	9

Notes: (1) through thermometer 1812283 (1982).



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International Intercomparison CCT: 1979-1983 pag.1

### Manufacturer: NRC

Sealing date: Jun 1979
cell total mass: 202.586 g
sample mass: 0.063 mol
Filling gas type: Matheson
impurity analysis:

Enthalpy of melting: 28 J

Measurements at Resistance Ratio (W)

LABORATORY	ASMW	IMGC	INM	NML	NPL
Date	Feb 81	Sep 80	May 80	Jun 79	Nov 81
thermometer N°	217997	PL01-6	232788	1731676	1728839
Ro (ohm)	25.418540	25.271140	25.087300	25.522800	25.559570
Typical melting plateau melted fraction F	8 % 7849 16% 7912 24% 7888 42% 7912 58% 7919 75% 7880	8 % 1786 15% 1809 20% 1821 26% 1837 32% 1849 42% 1861 50% 1884 60% 1896	10%315720%316930%316140%316550%317360%316570%315380%314990%3138	5 % 6424 15% 6448 20% 6452 25% 6456 30% 6460 40% 6460 60% 6460 80% 6460 95% 6460	5 % 6420 10% 6424 18% 6430 25% 6440 35% 6440 50% 6442 65% 6444 80% 6450 95% 6460
W(100%)	0.09187939	0.09191885	0.09193165	0.09196460	0.09196444
T (K) LAB NPL	54.36107 54.36110	54.36148 54.36167	54.36103 54.36134	54.36102 54.36092	54.36088 54.36088
average drift (mK/h) recovery time to 0.1mK (min)	2 0.5-4	5	2-3		2
overheating at 50% (mK/mW)	0.7	0.5	2 0		-
enthalpy of melting (J)	27.8	21			

Notes:



NRC

### CELL TYPE: 15 02 NRC

International Intercomparison CCT: 1979-1983 pag.2

Manufacturer: NRC

Sealing date: Jun 1979

cell total mass: 202.586 g

sample mass: 0.063 mol

Filling gas type: Matheson

impurity analysis: research grade

NRC

5

n

# Enthalpy of melting: 28 J

Resistance Ratio (W)

Measurements at	
LABORATORY	NRC
Date	Jun 79
thermometer N° Ro (ohm)	1521389 25.523332
<u>Typical</u> melting plateau	4 % 4729 8 % 4764 25% 4772 67% 4772
melted fraction F	
W(100%)	0.09184772
T (K) LAB NPL	54.36116 54.36099
average drift (mK/h) recovery time to 0.1mK (min) overheating at 50% (mK/mW)	10



enthalpy

of melting (J)

27



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International Intercomparison CCT: 1979-1983

Manufacturer: NRLM

Sealing date: Jun 1978

cell total mass: 170 g

sample mass: 0.06 mol

Filling gas type:

impurity analysis: nom. 99.99%

NRLM

Enthalpy of melting: 28 J

Measurements Resistance Ratio (W) at LABORATORY IMGC INM NRC NRLM NRLM (cell 7802) Oct 78 Date Nov 80 May 80 Oct 79 Nov 81 thermometer N° PL01-6 232788 1521389 7681 (1)Ro (ohm) 25.271140 25.087300 25.523332 25.362928 6415 8 % 5 % 8 % 2351 10% 4050 4930 1.7% 1276 17% 2498 20% 4066 10% 5165 11% 6515 15% 1592 Typical 25% 2545 30% 4034 30% 5321 18% 6630 27% 1789 melting 33% 2581 40% 4110 60% 5306 26% 6705 41% 1750 42% 50% 5400 35% 6740 52% 1746 plateau 2513 4114 83% 54% 2585 60% 4110 49% 6740 66% 1700 59% 67% 2573 70% 3995 6735 77% 1718 melted 90% 2561 80% 4174 67% 6745 92% 1710 fraction 90% 4190 72% 6735 F 91% 6775 W(100%) 0.09192560 0.09194095 0.09185354 0.09196770 0.09201750 T (K) LAB 54.36300 54.36337 54.36264 54.3631 54.36368 NPL 54.36335 54.36247 54.3632 average drift 20 1.5 (mK/h)recovery time to 0.1mK (min) 2 2-8 2 - 4overheating at 50% (mK/mW) 0.4 enthalpy

of melting (J) 20	28.1	20.7
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Notes: (1) 1978 data reported only for record.





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# CELL TYPE: PRMI (02)

International Intercomparison CCT: 1979-1983

Manufacturer: PRMI Multicomponent cell (argon, oxygen, neon, hydrogen) Sealing date: Dec 1978

cell total mass:

sample mass:

Filling gas type: home-made from decomp. of KClO4

impurity analysis:

Enthalpy of melting: 32.5 J

Resistance Ratio (W)

at		
LABORATORY	PRMI	
Date	Nov 81	
thermometer N°	1842381	
Ro (ohm)	25.544950	
Typical melting plateau	17% 4520 33% 4680 50% 4780 67% 4690 83% 4710	
melted fraction F		

W(100%)		0%)	0.09174780
Т	(K)	LAB NPL	54.36049

NPL	54.3604

average drift	
(mK/h)	-3
recovery time	
to 0.1mK (min)	20
overheating	
at 50% (mK/mW)	0.15
ontholow	
enchalpy	
of melting (J)	32.5

Notes:



PRMI

Measurements



### CELL TYPE: 1 H2 IMGC

Manufacturer: IMGC

Sealing date: Aug 1980

cell total mass: 164.422 g (with 1.000 g catalyst) sample mass: 0.11 mol

Filling gas type: Precision Gas Products

impurity analysis: nom. 99.9999%

Enthalpy of melting: 13 J

Measurements at		Resista	nce Ratio (W)
LABORATORY	ASMW	IMGC	PRMI
Date	Mar 81	Aug 80	Dec 81
thermometer N°	207278	PL01-6	1842381
Ro (ohm)	25.346860	25.271140	25.544950
Typical melting plateau melted fraction F	9 % 4358 18% 5358 27% 5354 36% 5358 45% 5362 52% 5362 57% 5358 91% 5358	5 % 0983 10% 0985 20% 0988 40% 0991 75% 0992	22% 8814 44% 8823 67% 8821 89% 8828
W(100%)	0.001353580	0.001409930	0.001188280
T (K) LAB NPL	13.81109	ref 13.81567 <sup>(2)</sup>	13.81030
average drift (mK/h) recovery time	4	5	<1
to 0.lmK (min) overheating	1-3	1	10
at 50% (mK/mW)	1.5	1	0.2
enthalpy of melting (J)	14.9	13.2	3.7 (1)

Notes: (1) the cell began to leak at PRMI. (2) with PL02-6 T(NPL)= 13.81070 K.





## CELL TYPE: 2 H2 IMGC

International Intercomparison CCT: 1979-1983

# Manufacturer: IMGC

Sealing date: Jan 1983

cell total mass: 163.343 g

sample mass: 0.055 mol

Filling gas type: Precision Gas Products

impurity analysis: nom. 99.9999%

Enthalpy of melting: 6.5 J

Resistance Ratio (W)

ŝ

Measurements at	
LABORATORY	IMGC
Date	Jun 83
thermometer N°	PL01-6
Ro (ohm)	25.271140
<u>Typical</u> melting plateau	7.5% 09822 12% 09836 23% 09869 59% 09910 80% 09934
melted fraction F	
W(100%)	0.001409922
T (K) LAB NPL	13.80997 13.81564
average drift (mK/h) recovery time to 0.1mK (min) overheating at 50% (mK/mW)	<10 1 10
enthalpy of melting (J)	5.9
Notes:	





Т

# Manufacturer: NRC

Sealing date: Aug 1979

cell total mass: 127.808 g

sample mass: 0.04556 mol

Filling gas type:

impurity analysis:

Enthalpy of melting: 5.33 J

Measurements at		Resistan	nce Ratio (W)	)
LABORATORY	IMGC	NML	NPL	NRC
Date	Oct 80	Feb 83	Feb 80	Sep 79
thermometer N°	PL01-6	1731676	1728839	1521389
Ro (ohm)	25.271140	25.522800	25.559570	25.523332
Typical melting plateau melted fraction F W(100%)	15% 0983 30% 0985 37% 0986 45% 0987 62% 0989 90% 0991	5 % 0022 7 % 0034 9 % 0051 13% 0057 16% 0069 25% 0081 36% 0086 50% 0086 61% 0092 71% 0098 86% 0110 0.001350095	7 % 5059 14% 5060 23% 5061 33% 5061 47% 5062 67% 5063 88% 5063	4 % 1490 7 % 1650 14% 1650 42% 1690 71% 1730
T (K) LAB NPL	13.80995 13.81562	13.80962 13.80993	13.81008 13.81008	13.80881 13.80927
average drift (mK/h) recovery time to 0.1mK (min) overheating at 50% (mK/mW)	5 10 1.5		0.3 1	
enthalpy of melting (J)	4.2			5.0

Notes:



NRC



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International Intercomparison CCT: 1979-1983 pag.1

Manufacturer: NRLM

Sealing date: Jun 1978

cell total mass: 174 g

sample mass: 0.038 mol

Filling gas type:

impurity analysis: nom. 99.99999%
 tot. 0.6 vppm

NRLM

Measurements

Enthalpy of melting: 4.42 J

Resistance Ratio (W)

at		<ul> <li>4</li> </ul>			
LABORATORY	ASMW	IMGC	NML	NPL	NRC
Date	Dec 80	Mar 79	Oct 79	May 79	Jun 80
thermometer N°	217997	PL01-6	1731676	1728839	1521389
Ro (ohm)	25.418540	25.271140	25.522800	25.559570	25.523332
Typical melting plateau melted fraction F	5 % 1963 10% 1982 22% 1986 30% 1982 50% 1982 57% 1990 70% 1982 90% 1986	5 % 0982 10% 0987 20% 0987 30% 0985 50% 0987 65% 0989 75% 0987	5 %       5004         10%       5006         20%       5006         30%       5006         40%       5006         50%       5006         60%       5006         70%       5006         80%       5006         95%       5006	4 % 5060 6 % 5061 9 % 5061 19% 5062 41% 5063 45% 5063 51% 5063 59% 5063 87% 5063	11% 1810 14% 1850 28% 1850 55% 1810 69% 1770 83% 1810
W(100%)	0.001319900	0.001409870	0.001350063	0.001350630	0.001301830
T (K) LAB NPL	13.81057	13.80975 13.80542	13.80950 13.80981	13.81008 13.81008	13.80914 13.80960
average drift (mK/h) recovery time	4	5			
to 0.1mK (min) overheating	0.5-3	3	1	1	
at 50% (mK/mW)	0.3	2			
enthalpy of melting (J)	4.3	4.17			4.64

```
Notes:
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# CELL TYPE: 7801 H2 NRLM

International Intercomparison CCT: 1979-1983 pag.2

Manufacturer: NRLM

Sealing date: Jun 1978

cell total mass: 174 g

sample mass: 0.038 mol

Filling gas type:

Enthalpy of melting: 4.42 J

Measurements at	2	Resistance	Ratio	(W)
LABORATORY	NRLM	NRLM		
Date	Jul 78	Nov 81		
thermometer N° Ro (ohm)	(1)	7681 25.362929		
Typical melting plateau melted fraction F	8 % 34863 10% 34875 16% 34882 28% 34887 46% 34891 67% 34891 78% 34895 82% 34891 92% 34898	1.7% 398435 20% 400794 21% 00784 41% 00784 49% 00802 61% 00783 79% 00827 81% 00783		
W(100%)	0.001334895	0.001400810		

Т	(K)	LAB	13.8115	
		NPL	13.8134	

average drift (mK/h)		2.6
recovery time		
to 0.1mK (min)		
overheating		
at 50% (mK/mW)		
199 in 1996 Families Constructions Construction		
enthalpy		
of melting (J)	4.42	4.37

Notes: (1) 1978 data reported only for record.



NRLM



### CELL TYPE: PRMI (H2)

International Intercomparison CCT: 1979-1983

 Manufacturer: PRMI Multicomponent cell (argon,oxygen,neon,hydrogen) Sealing date: Dec 1978 cell total mass: sample mass: Filling gas type: commercial source purif.

through palladium filter impurity analysis:

PRMI

# Enthalpy of melting:

Resistance Ratio (W)

Measurements at

LABORATORY		PRMI	
Date		Nov	81
thermometer	N°	1842	2381

Ro	<b>b</b> (	ohm)	25.	544950

Typical melting plateau melted fraction F	9 % 19% 28% 37% 56% 74% 93%	8822 8821 8822 8827 8837 8838 8837
W(100%)	0.001	18830
T (K) LAB NPL	13.81	033
average drift (mK/h) recovery time to 0.lmK (min) overheating at 50% (mK/mW)	2 10 0	6
enthalpy of melting (J)	0.	U



### CELL TYPE: 7 CH4 BIPM

Manufacturer: BIPM

Sealing date: Sep 1977

cell total mass: 230 g

sample mass: 0.15 mol

Filling gas type: Air Liquide

impurity analysis: nom. 99.9995%
 nitrogen 2 vppm
 oxygen 0.5
 carb.diox. 0.1
 hydrogen <0.1; hydroc. <0.1</pre>

Enthalpy of melting: 140 J

Measurements

SEI

at

Resistance Ratio (W)

Ţ	ABOR	ATORY	BIPM		NRC	
	Dat	te	Apr 80	D	Aug 79	
th	Ro	neter N° (ohm)	226321 25.369150		152138 25.523	39 3332
		-				
	Typic melt: plato melto frac: F	ed	9 % 20% 50%	3847 3934 3946	4 % 8 % 15% 24% 44% 58% 70% 73% 88%	7934 7954 7973 7993 8051 8091 8091 8071 8101
	W(10	0%)	0.245	93950 <sup>(1)</sup>	0.245	88103
Т	(K)	LAB NPL	90.68 90.68	661 402	90.68 90.68	539 464

average drift	10 mil	
(mK/h)	<3	
recovery time		
to 0.1mK (min)	30-40	20
overheating		
at 50% (mK/mW)	0.3	
enthalpy	5 . 15° ber	
of melting (J)	143	

Notes: (1) in oct 77/nov 78 the same cell and thermometer gave W(100%)= 0.2459363 = T(LAB)= 90.68586 K: no explanation available.



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International Intercomparison CCT: 1979-1983 pag.1



Measurements at Manufacturer: IMGC

Sealing date: Aug 1976

cell total mass: 195 g

sample mass: 0.23 mol

Filling gas type: Matheson

impurity analysis: nom. 99.995%

Enthalpy of melting: 220 J

Resistance Ratio (W)

LABORATORY	ASMW	IMGC	NIM	NPL .	NRC
Date	Dec 79	Aug 76	Mar 81	Apr 78	Dec 76
thermometer N°	217990	PL01-6	188640	1728839	1521389
Ro (ohm)	24.186290	25.271140	24.164330	25.559570	25.523332
Typical melting plateau melted fraction F	10% 8901 22% 8982 40% 9013 58% 9023 60% 9016 76% 9024 82% 9031 89% 9026	5 % 0740 7.5% 1000 10% 1240 25% 1780 42% 2000 62% 2140	7 % 3716 14% 3794 20% 3799 27% 3827 41% 3839 55% 3858 68% 3868	9 % 59850 16% 9900 22% 9930 31% 9940 41% 9960 53% 9970 87% 9990	5.4% 5662 11% 6504 15% 6782 16% 6813 26% 7182 33% 7244 44% 7464 65% 7624 78% 7664
W(100%)	0.24590300	0.24592185	0.24638710	0.24600010	0.24587703
T (K) LAB NPL	90.68350 90.68350	90.68518 90.68386	90.68370	90.68372 90.68372	90.68447 90.68373
average drift (mK/h) recovery time	2	10		15	
overheating at 50% (mK/mW)	0.3	40-20 0.04		15	
enthalpy of melting (J)	224.5	215			215
International Intercomparison CCT: 1979-1983 pag.2



Manufacturer: IMGC

Sealing date: Aug 1976

cell total mass: 195 g

sample mass: 0.23 mol

Filling gas type: Matheson

impurity analysis: nom. 99.995%

Enthalpy of melting: 220 J

Resistance Ratio (W)

LABORATORY	NRLM
Date	Nov 81
thermometer N°	7681
Ro (ohm)	25.363524

Measurements

at

	Typic melt: plate	cal ing eau	19% 21% 23% 49% 77% 79% 87%	599131 9252 9406 9930 600263 0241 0112
	merro	ed		
	frac F	tion		
	W(10	0%)	0.24	4600250
Т	(K)	LAB NPL	90.0 90.0	58811 58798
av re	verag (mK/)	e drift h) rv time		4

recovery time	
to 0.1mK (min)	10-40
overheating	
at 50% (mK/mW)	
enthalpy	
of melting (J)	218



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### CELL TYPE: 12 CH4 IMGC

Manufacturer: IMGC

Sealing date: Apr 1979

cell total mass: 165.573 g

sample mass: 0.11489 mol

Filling gas type: Matheson

impurity analysis: nom. 99.995% oxygen+argon 0.25 vppm carbon dioxide 0.1 nitrogen 2.3

Enthalpy of melting: 108 J

/ . . . Resistance Ra

lati	lo	(W)	

LABORATORY	BIPM	IMGC	NRC	PRMI
Date	Mar 80	Apr 79-Oct 81	Aug 79	Dec 81
thermometer N°	226321	PL01-6	1521389	1842381
Ro (ohm)	25.369150	25.271140	25.523332	25.544950
Typical melting plateau melted fraction F	14% 1880 48% 3300 77% 3450	5 % 89927 10% 90422 20% 0877 29% 1154 38% 1392 48% 1550 59% 1787 67% 1905 77% 1985	2.5% 5897 10% 6739 20% 7052 30% 7307 60% 7621 80% 7699 85% 7738	5 % 7941 9 % 8003 14% 8283 19% 8509 29% 8829 40% 9099 50% 9241 74% 9349
W(100%)	0.24593500	0.24592120	0.24587753	0.24579400
T (K) LAB NPL	90.68554 90.68298	90.68503 90.68371	90.68459 90.68384	90.67968
average drift (mK/h) recovery time to 0.1mK (min)	-2.6	10	30	<1 90
overheating at 50% (mK/mW)	5 <sup>(1)</sup>	0.02	50	0.3
enthalpy of melting (J)	76	107	105	117

Notes: (1) cell heated from inside the block.



Measurements

at



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Manufacturer: NRC

Sealing date: Aug 1979

cell total mass: 197.291 g

sample mass:

Filling gas type: commercial source purified at NRC

impurity analysis:

Enthalpy of melting: 60 J

Measurements at				Resistan	nce R	latio	(W)
LABORATORY	BIPM		IMGC		NRC		
Date	Feb 80	)	Jul 80	С	Aug 7	'9	
thermometer N° Ro (ohm)	226321 25.369	088 9088	PL01-6	6 1140	15213 25.52	389 23332	
Typical melting plateau melted fraction F	7 % 20% 38% 59% 90%	2977 3245 3415 3513 3434	12% 17% 22% 32% 38% 45% 53% 62% 80%	1922 2005 2056 2092 2124 2151 2175 2207 2223 2254 2261	2.5% 5 % 10% 20% 40% 60% 80%	7229 7425 7523 7640 7738 7875 7933	
W(100%)	0.2459	93430 <sup>(1)</sup>	0.245	92280	0.24	587955	
T (K) LAB NPL	90.685 90.683	566 310	90.68 90.68	540 408	90.68 90.68	3505 3430	
average drift (mK/h) recovery time	-6		5				
to 0.1mK (min)	5		5-1	0	30	C	
at 50% (mK/mW)	0.1	12	0.	2			
enthalpy of melting (J)	84		51		60	C	

Notes: (1) suggested BIPM value: 0.2459354.



NRC



Manufacturer: IMGC

Sealing date: Feb 1980

cell total mass: 78.429 g

sample mass: 0.03596 mol

Filling gas type: Matheson

Enthalpy of melting: 25.9 J

Measurements at		Resista	nce Ratio (	W)
LABORATORY	IMGC	INM	NPL	NRC
Date	Aug 80	Sep 83	Nov 80	Nov 81
thermometer N°	45	1812283	1728839	1872179
Ro (ohm)	25.679410	25.494711	25.559570	25.582650
Typical melting plateau melted fraction F W(100%)	7 % 8549 13% 8587 20% 8617 33% 8636	7.7% 1753 15% 1780 23% 1792 31% 1796 38% 1796 46% 1796 54% 1796 62% 1796 69% 1804 91% 1804	7 % 6650 12% 6660 18% 6610 27% 6650 35% 6670 47% 6680 58% 6680 70% 6680 88% 6680	0.5% 3356 1.1% 3759 4.5% 3793 13% 3805 30% 3832 56% 3856 82% 3879
T (K) LAB NPL	63.14627 63.14626	63.14596 63.14671	63.14611 63.14611	63.14637 63.14562
average drift (mK/h) recovery time	2	-1.5 to +3	0.3	
to U.1mK (min) overheating at 50% (mK/mW)	10 0.1	30	10	20
enthalpy of melting (J)	23			





### CELL TYPE: BCM4 INM (N2)

International Intercomparison CCT: 1979-1983

Manufacturer: INM Multicomponent cell (argon,oxygen,neon,nitrogen) Sealing date: Jan 1982

cell total mass: 369.83 g

sample mass: 0.0218 mol

### Filling gas type:

Enthalpy of melting: 13 J

Resistance Ratio (W)

Measurements	
at	

LABORATORY	INM	NRC
Date	Feb 82	Jul 82
thermometer N°	1812283	1872179
Ro (ohm)	25.494713	25.582657

Typical melting plateau	0 % 144 10% 165 20% 166 30% 17 40% 17 50% 17 60% 17	80       5.5%         37       9.7%         84       18%         31       35%         31       52%         31       68%         31       01	3665 3715 3754 3793 3793 3793
melted	70% 17	31	
fraction	80% 17	31	
F	90% 17	31	
W(100%)	0.127417	<u>31</u> <u>0.12</u>	743793
T (K) LAB NPL	63.14579 63.14654	63.1 63.1	4633 4558

average drift (mK/h)	-12 to 3	
recovery time to 0.1mK (min) overheating at 50% (mK/mW)	10	30
enthalpy of melting (J)	13	

Notes:

B Construction Construction

INM mod. BCM



Manufacturer: NRC

Sealing date: Jul 82

cell total mass: 205.100 g

sample mass: 0.05236 mol

Filling gas type: MG Scientific Gases

impurity analysis: research grade

NRC

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# Enthalpy of melting: 37.4 J

Measurements at		Resistance	Ratio	(W)
LABORATORY	IMGC	NRC		
Date	Jun 83	Jul 82		
thermometer N°	45	1872179		
Ro (ohm)	25.679410	25.582651		
Typical melting plateau melted fraction F	5% 8430 10% 8528 20% 8603 30% 8622 40% 8630 50% 8626 70% 8632	2.6% 3430 4.2% 3555 7.4% 3664 14% 3715 27% 3738 53% 3731 79% 3715 99% 3754		
W(100%)	0.12758642	0.12743740		
T (K) LAB NPL	63.14622 63.14621	63.14620 63.14545		
average drift (mK/h) recovery time to 0.1mK (min) overheating at 50% (mK/mW)	2 1 0.02	13		
enthalpy of melting (J)	37.2	37.4		





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Ned. 1: @ 10 Mod. 2: @ 20

Measurements

Manufacturer: ASMW

Sealing date: Nov 1981

cell total mass: 105 g

sample mass: 0.0375 mol

Filling gas type: Linde AG type R5.0

impurity analysis: nom. 99.999%

Enthalpy of melting: 12.2 J

Resistance Ratio (W)

at				5
LABORATORY	ASMW		IMGC	
Date	Dec 8	32	May 8	3
thermometer N°	20727	8	PL01-	6
Ro (ohm)	25.34	6860	25.27	1140
Typical melting plateau melted fraction F	10% 20% 30% 40% 50% 60% 70% 80%	3317 3329 3369 3357 3357 3369 3369 3369 3369 3373	6.1% 10% 20% 31% 41% 53% 69%	8061 8069 8089 8093 8097 8097 8099
W(100%)	0.00863370		0.00868097	
T (K) LAB NPL	24.56	5312 <sup>(1)</sup>	24.56	308
average drift (mK/h) recovery time	5		2	
to 0.1mK (min) overheating	1		1	
at 50% (mK/mW)	0.	55	0.7	
enthalpy of melting (J)	12.	. 2	11.	8

Notes: (1) in Ref.55: T(NPL) = 24.5627 K.



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Manufacturer: IMGC

Sealing date: Jun 1977

cell total mass: 200 g

sample mass: 0.32 mol

### Filling gas type: Matheson

impurity analysis: nom. 99.995%
 hydrogen 25 vppm
 nitrogen 11.3
 oxygen+argon 2.7

Enthalpy of melting: 106 J

Measurements at

Resistance Ratio (W)

LABORATORY	IMGC	NRLM
Date	Jun 77-Dec 81	Nov 81
thermometer N° Ro (ohm)	PL01-6 25.271140	7681 25.362939
<u>Typical</u> melting plateau	11%802620%803030%804050%804470%805280%8054	16%         30288           17%         30345           40%         30475           41%         30495           64%         30607           65%         30600           87%         30608
melted fraction F		89% 30633
W(100%)	0.008680620	0.008730630
T (K) LAB NPL	ref 24.56281 <sup>(1)</sup>	24.5615 24.5622
average drift (mK/h) recovery time	10	1.6
to 0.1mK (min)	1	3-9
at 50% (mK/mW)	0.1	
enthalpy of melting (J)	106	104.8

Notes: (1) with PL02-6 T(NPL) = 24.5617 K.



	THRC
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International Intercomparison CCT: 1979-1983 pag.1

Manufacturer: IMGC

Sealing date: Feb 1979

cell total mass: 165.449 g

sample mass: 0.11128 mol

Filling gas type: Matheson

impurity ana	alysis:	nom.	99.	995%
hydrog	gen a)	-	b)	<25
nitrog	gen	5.5		6.3
oxyger	n+argon	2.5		6.3

Enthalpy of melting: 37 J

Resistance Ratio (W)

LABORATORY	ASMW	IMGC	INM	NML	NPL
Date	Mar 81	Feb 79-Dec 81	Sep 83	Dec 82	Jan 80
thermometer N°	207278	PL01-6	1812283	1731676	1728839
Ro (ohm)	25.346860	25.271140	25.494711	25.522800	25.559570
Typical melting plateau melted fraction F	8 % 3239 21% 3266 33% 3254 50% 3262 70% 3270	10%804812%805017%805220%805433%805648%805867%805880%8060	14% 8468 28% 8471 42% 8476 56% 8492 70% 8495 84% 8508	5 % 4581 7 % 4585 10% 4588 15% 4592 20% 4594 24% 4596 32% 4600 41% 4605 51% 4607 80% 4607 95% 4613	6 % 4510 12% 4524 20% 4536 29% 4543 40% 4545 52% 4548 67% 4550
W(100%)	0.008632780	0.008680620	0.008484935	0.008646070	0.008645500
T (K) LAB NPL	24.56236	24.56281	24.56195	24.56250 24.56190	24.56163 24.56163
average drift (mK/h) recovery time	3.5	15			0.3
to 0.1mK (min) overheating	1-5	1-5	12	1	2
at 50% (mK/mW)	0.15	0.18		0.02	
enthalpy of melting (J)	36.3	35.7		37.5	

Notes:



Measurements

at

2	IMGC	NE	3	E:	Ρ	Y	Τ	L
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International Intercomparison CCT: 1979-1983 pag.2

Manufacturer: IMGC

Sealing date: Feb 1979

cell total mass: 165.449 g

sample mass: 0.11128 mol

Filling gas type: Matheson

impurity analysis: nom. 99.995%
hydrogen a) - b) <25 vppm
nitrogen 5.5 6.3
oxygen+argon 2.5 6.3</pre>

Enthalpy of melting: 37 J

Measurements at		Resistance	Ratio	(W)
LABORATORY	NRC .	PRMI	đ	
Date	Jun 79	Dec 81		
thermometer N°	1521389	1842381		
Ro (onm)	25.523332	25.544950		
Typical melting plateau melted fraction F	2 % 7282 4 % 7302 9 % 7314 25% 7314 50% 7310 75% 7306 82% 7302 90% 7306	5 % 5735 14% 5741 23% 5751 32% 5737 41% 5742 59% 5750 77% 5759		
W(100%)	0.008573080	0.008457550		
T (K) LAB NPL	24.56346 24.56125	24.56346 24.56125 24.56187		
average drift (mK/h) recovery time to 0.1mK (min)		<1		
overheating at 50% (mK/mW)		0.02		
enthalpy				

of melting (J)



CEL



C	ਸ਼	Т	T	T	V	P	F •	BCM4	TNM	(Ne)	ì.
0	<u>.</u>	-		-	- <b>-</b>	-	1.4 .	DOLLA	TT1TT	(LIC)	,

International Intercomparison CCT: 1979-1983

Manufacturer: INM Multicomponent cell (argon, oxygen, neon, nitrogen) Sealing date: Jan 1982 cell total mass: 369.83 g sample mass: 0.0262 mol Filling gas type: impurity analysis: nitrogen 15 vppm oxygen 3 helium 80 hydrogen 3

Enthalpy of melting: 9.1 J

Resistance Ratio (W) Measurements at LABORATORY INM NRC Jan 82 Jul 82 Date thermometer N° 1812283 1872179 Ro (ohm) 25.494713 25.582650 0 % 8470 4.1% 1655 8.2% 1811 11% 8466 Typical 22% 8479 18% 1890 33% 8485 39% 1968 melting

8493

59%

2007

melted fraction F	55% 66% 77% 88%	8501 8503 8513 8495	80%	2085	
W(100%)	0.00	8484920	0.008	3552124	
r (k) lai NPI	3 24.5	6194	24.56 24.56	5336 5115	

44%

average drift (mK/h) recovery time to 0.1mK (min) overheating at 50% (mK/mW)	-5 6	18
enthalpy of melting (J)	9.1	

Notes:

plateau





INM mod. BCM



International Intercomparison CCT: 1979-1983 pag.1

### Manufacturer: NRC

Sealing date: Jun 1979 cell total mass: 206.507 g sample mass: 0.051 mol

Filling gas type:

impurity analysis:

Enthalpy of melting: 17 J

Resistance Ratio (W)

LABORATORY	ASMW	IMGC	NML	NPL	NRC
Date	Jan 81	Oct 80	Dec 79	Feb 80	Sep 79
thermometer N°	217997	PL01-6	1731676	1728839	1521389
Ro (ohm)	25.418540	25.271140	25.522800	25.559570	25.523050
Typical melting plateau melted fraction F	9 % 1198 18% 1210 27% 1222 45% 1230 58% 1230 76% 1222	5 % 7990 10% 8003 19% 8029 29% 8027 40% 8029 50% 8027 70% 8029 90% 8027	5 % 4516 10% 4518 20% 4524 30% 4535 40% 4537 50% 4535 60% 4537 80% 4547	5 % 4502 9 % 4512 15% 4518 22% 4521 29% 4524 43% 4522 59% 4520 73% 4522 86% 4522 97% 4523	5 % 7274 10% 7282 30% 7298 60% 7302 90% 7302
W(100%)	0.008612220	0.008680300	0.008645370	0.008645220	0.008573030
T (K) LAB NPL	24.56234	24.56255	24.56287 24.56166	24.56141 24.56141	24.56342 24.56121
average drift (mK/h) recovery time to 0.1mK (min) overheating at 50% (mK/mW)	3.5 1-5	15 2		0.3 1	
enthalpy of melting (J)	17.2	15.5			17

```
Notes:
```



NRC

Measurements at

### CELL TYPE: 12 NE NRC

International Intercomparison CCT: 1979-1983 pag.2

## Manufacturer: NRC

Sealing date: Jun 1979

cell total mass: 206.507 g

sample mass: 0.051 mol

Filling gas type:

impurity analysis:

Enthalpy of melting: 17 J

Measurements at		Resistance	Ratio	(W)
LABORATORY	NRC			
Date	Feb 82			
thermometer N°	1872179			
Ro (ohm)	25.582651			
<u>Typical</u> melting plateau	6% 1303 11% 1538 23% 1616 34% 1733 57% 1772 80% 1890			
melted fraction F				
W(100%)	0.008551811			
T (K) LAB NPL	24.56311 24.56090			
<pre>average drift   (mK/h) recovery time to 0.lmK (min) overheating at 50% (mK/mW) enthalpy</pre>				
of melting (J)				

Notes:



NRC

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Manufacturer: NRLM

Sealing date: Jul 1978

cell total mass: 170 g

sample mass: 0.050 mol

Filling gas type:

impurity analysis: nom. 99.99% tot. 2.5 vppm

Enthalpy of melting: 16.7 J

Measurements at		Resistance Ratio (W)
LABORATORY	NPL (1)	NRLM
Date	May 79	Sep 78
thermometer N°	1728839	(2)
	23:337570	
Typical melting plateau	8 % 4529 14% 4537 27% 4543 38% 4548 49% 4550 63% 4552 78% 4551	
melted fraction F	90% 4551	
W(100%)	0.008645520	
T (K) LAB NPL	24.56165 24.56165	24.56305
average drift (mK/h) recovery time to 0.lmK (min) overheating at 50% (mK/mW)	1 2	
enthalpy of melting (J)		16.7
Notes: (1) the (2) 1978	cell lost the data reported	gas after the measurements at NPL. I only for record.



NRLM



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Manufacturer: NRLM

Sealing date: Jul 1978

cell total mass: 168 g

sample mass: 0.052 mol

Filling gas type:

impurity analysis: nom. 99.99% tot. 2.5 vppm

Enthalpy of melting: 17.5 J

Measurements at	<i>2</i>	Resista	nce Ratio (W	)	
LABORATORY	IMGC	NPL	NRC	NRLM	NRLM
Date	Sep 80	Feb 80	Sep 79	Nov 81	Aug 78
thermometer N°	PL01-6	1728839	1521389	7681	(1)
KO (OIIII)	23.271140	23.33370	23.22332	23.302923	
Typical melting plateau melted fraction F	9 % 8000 14% 8010 22% 8026 32% 8046 43% 8052 68% 8058	7       %       4548         11%       4552         16%       4558         20%       4559         29%       4562         38%       4562         56%       4559         70%       4558         87%       4557	4       %       7278         8       %       7298         16%       7310         26%       7314         31%       7314         43%       7314         55%       7325         60%       7318         70%       7333         77%       7325         88%       7325	8.5%3020411%3013318%3042130%3038137%3053949%3050056%3050068%3046075%3044087%30460	¥ V
W(100%)	0.008680640	0.008645600	0.008573290	0.0087305000	
T (K) LAB NPL	24.56283	24.56172 24.56172	24.56363 24.56142	24.5614 24.5621	24.56307
average drift (mK/h) recovery time	15	0.3		2.3	
to 0.1mK (min)	1-5	2		1.4-3	
at 50% (mK/mW)	0.8				
enthalpy of melting (J)	17		19	20.1	

Notes: (1) 1978 data reported only for record.



NRLM



### CELL TYPE: PRMI (Ne)

International Intercomparison CCT: 1979-1983



PRMI

<u>Manufacturer</u>: PRMI Multicomponent cell (argon,oxygen,neon,hydrogen) Sealing date: Dec 1978

cell total mass:

sample mass:

### Filling gas type:

impurity analysis: nitrogen 16 vppm oxygen 3 carb.dioxi.<0.7 hydrog.,helium none

Enthalpy of melting:

Resistance Ratio (W)

at		
LABORATORY	PRMI	
Date	Nov 81	
thermometer N°	1842381	
Ro (ohm)	25.544950	
	11% 5717	
	23% 5741	
DOULD' AND MO		

Measurements

Typical melting plateau	23% 34% 46% 57% 69%	5741 5750 5748 5763 5762
melted fraction F	01%	5789
W(100%)	0.008	345771
T (K) LAB NPL	24.56	5200
average drift (mK/h) recovery time	-1,	. 5
to 0.1mK (min)	10	
overheating at 50% (mK/mW)	0.	. 07
enthalpy		

of melting (J)



International Intercomparison CCT: 1979-1983



Manufacturer: IMGC

Sealing date: Nov 80

cell total mass: 169.195 g (incl. 1.5 g catalyst) sample mass: 0.013 mol

Filling gas type: C.E.A.

impurity analysis: nom. 99.86%

Enthalpy of melting: 20.5 J

Measurements at		Resista	nce Ratio (W)
LABORATORY	IMGC	NPL	NRC
Date	Dec 80	Jan 82	Nov 81
thermometer N°	PL01-6	1728839	1872179
Ro (ohm)	25.271140	25.559570	25.582651
Typical melting plateau melted fraction F	6.5% 8208 11% 8235 16% 8295 23% 8331 28% 8358 39% 8390 48% 8429 59% 8469 90% 8520	5.5% 2965 11% 2999 22% 3033 39% 3095 55% 3156 71% 3210 88% 3264	5 % 3520 20% 3704 30% 3813 40% 3899 50% 3993 60% 4079 70% 4141 80% 4227
W(100%)	0.00338515	0.00333290	0.00324300
T (K) LAB NPL	18.6778	18.6753 18.6753	18.6764 18.6777
average drift (mK/h)	5		bad thermal control
to 0.1mK (min)	5		
at 50% (mK/mW)	0.3		
enthalpy of melting (J)	20.5		20.8



Manufacturer: NRC

Sealing date: Dec 1981

cell total mass: 204.954 g

sample mass: 0.0484 mol

Filling gas type: Monsanto

impurity analysis: nom. 99.6%

NRC

Measurements

Enthalpy of melting: 4.9 J

Resistance Ratio (W)

at		
LABORATORY	NRC	
Date	Dec 81	
thermometer N°	1872179	
Ro (ohm)	25.582651	
Typical melting plateau	5.2% 10% 21% 36% 63% 78%	3043 3203 3258 3305 3363 3398
melted fraction F		
W(100%)	0.003	23380
T (K) LAB NPL	18.66 18.66	10 23
average drift (mK/h)		

(mK/h)
recovery time
to 0.lmK (min)
overheating
at 50% (mK/mW)
enthalpy
of melting (J) 4.9 J





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#### VI. RESULTS ON SCALE REALIZATIONS

This section collects the temperature data that could be obtained from the measurements made during the Intercomparison. These data are also contained in the data sheets of the preceeding Section.

In this Section, they are grouped in three sets of Tables (VI.1.x, VI.2.x and VI.3.x); each set contains seven Tables, one for each of the three definition points (a., b. and c.) and one for each of the secondary fixed points (d. to g.).

Tables VI.1.x collect the W(100%) values used in order to calculate the temperature values (and the temperature differences of Section VII, Tables VII.1.x).

Tables VI.2.x collect the temperature values, calculated on the National realizations of the IPTS-68 (LAB-IPTS-68), when available (see information about these realizations at p.13-14).

Tables VI.3.x collect the temperature values, calculated with calibrations on NPL-IPTS-68, when available on the thermometers used by the Laboratories.

#### Captions of the Tables

Tables VI.1.x: W(100%) values

Thermometer: in the first row the reference thermometers are indicated, to which the underlined W values pertain. In the subsequent lines, the thermometers actually used in the measurements at the Laboratories are indicated (when different from the reference thermometer): the corresponding W values are reported in a subsequent line below the reference W values in the Table. Underlining of the thermometers indicates that a NPL calibration is available.

W values: - underlined (reference) values refer to the reference thermometers. - not-underlined values refer to the other indicated thermometers; in this case, the reference value is calculated from the former, using the calibration tables of both the reference (1) and non-reference (2) thermometers, at the temperature indica-

> ted by thermometer (2). - some Laboratories have not measured the reference cell , but another IMGC cell. In this case, the reference value for the reference cell has been calculated from the difference between the two cells, as measured at IMGC. With hydrogen, the PRMI value for the NRLM cell has been obtained through IMGC cell.

Tables VI.2.x: T(LAB-IPTS-68) values

- Thermometers: only the thermometers used for temperature calculation are indicated. They are underlined when a NPL calibration is also available.
- Temperature values: (ref.) indicates that this cell has been considered as the reference realization for the Laboratory. In a few cases, two independent determinations of the same Laboratory, made at different times, are available for the same cell.
- Tables VI.3.x: T(NPL-IPTS-68) values
- Thermometer: only thermometers with NPL calibrations are indicated. The calibration may be one of two types: a) calibration made at NPL during the International Intercomparison of thermometers in 1975 (underlined; referred as "international group"); b) calibration made subsequently either at NPL (e.g. the BIPM thermometer) or by comparison with other thermometers calibrated at NPL (e.g. the NRLM and PRMI thermometers . For the latter the comparison, and calibration table, has been done at the argon triple point, instead of at the NPL calibration point (normal condensation of oxygen)). At NBS and NRC, the reference thermometer of Tables VI.1 and VI.2 (here indicated in second line), has a known relationship with the thermometers of the "international group" reported in the first line.
- Temperature values: owing to thermometer instability, in a few cases temperature values obtained at the same Laboratory with different calibrated thermometers are reported, when differences between them greatly exceeded IPTS-68 non-uniqueness.
|                  | (Reference cell: 1 IMGC)     |                          |                          |                                 |                                 |                          |                                 |                   |                   |                   |                          |  |
|------------------|------------------------------|--------------------------|--------------------------|---------------------------------|---------------------------------|--------------------------|---------------------------------|-------------------|-------------------|-------------------|--------------------------|--|
|                  | ASMW                         | BIPM                     | IMGC                     | INM                             | NBS                             | NIM                      | NML                             | NPL               | NRC               | NRLM              | PRMI                     |  |
| Thermometer:     | 217997<br>217990<br>(217278) | 226321                   | <u>PL01-6</u>            | 232788<br>1812283               | 1774095                         | 7703<br>188640           | 1731676                         | 1728839           | <u>1521389</u>    | 7681<br>1781356   | 1842381                  |  |
| Cells:           |                              |                          |                          |                                 | Resistance                      | ratio at F               | =100%                           |                   |                   |                   |                          |  |
| 4 ASMW<br>3 BIPM | 0.21600349<br>(0.21602447)   | 0.21603920               | 0.21602772               |                                 |                                 |                          |                                 |                   |                   |                   |                          |  |
| 1 IMGC<br>2 IMGC | 0.21600380<br>0.21600890     | 0.21603987<br>0.21603980 | 0.21602745<br>0.21602738 | <u>0.21603387</u><br>0.21603380 | <u>0.21604967</u><br>0.21604960 | 0.21614787<br>0.21650500 | <u>0.21610202</u><br>0.21610195 | <u>0.21610195</u> | <u>0.21597964</u> | <u>0.21611388</u> | 0.21589327<br>0.21589320 |  |
| 1 INM            |                              | 0.21603990               |                          | 0.21603355                      |                                 |                          | 0.21610340                      | ×                 | 0.21598038        |                   |                          |  |
| XXI INM          |                              |                          |                          | $\frac{0.21603360}{0.21596344}$ |                                 | 0.21614760               |                                 |                   |                   |                   |                          |  |
| BCM4 INM         |                              |                          |                          | 0.21603421<br>0.21603421        |                                 |                          |                                 |                   | 0.21598077        |                   |                          |  |
| M1 NBS           |                              | 0.21604070               |                          | 0.21596405                      | 0.21604965                      |                          |                                 |                   | 0.21597961        | 0.21611416        |                          |  |
| 113 NIM          |                              |                          |                          |                                 |                                 | 0.21614755               |                                 |                   |                   | 0.21599700        |                          |  |
| 10 NRC           |                              | 0.21604000               | 0.21602805               | 0.21603335                      | 0.21604955                      |                          | 0.21610370                      | 0.21610210        | 0.21597959        |                   |                          |  |
| 14 NRC           |                              |                          |                          |                                 |                                 | 0.21614820               |                                 |                   | 0.21597970        |                   |                          |  |
| 7801 NRLM        |                              |                          |                          |                                 | 0.21604960                      |                          |                                 |                   |                   |                   |                          |  |
| 7803 NRLM        | 0.21600360                   | 0.21604000               | 0.21602825               | 0.21603400                      |                                 |                          | 0.21610510                      |                   | 0.21597961        | 0.21611377        |                          |  |
| Ar PRMI          |                              |                          |                          |                                 |                                 |                          |                                 |                   |                   |                   | 0.21589295               |  |

Table VI.1.a : Results of the intercomparison between sealed cells filled with: A R G O N (Reference cell: 1 IMGC)

	A S M W°	BIPM°	IMGC°	INM	NBS	NIM	NML	NPL	NRC	NRLM	PRMI
Thermometer:	217997	226321	<u>PL01-6</u>	1812283	1774095	7703	1731676	1728839	<u>1521389</u>	7681	1842381
					The second second						
<u></u>					Temperatu	re in LAD-1	<u>(K)</u>				
4 ASMW	83.79758		83.79806								
3 BIPM	ós.	83.798 §									
1 IMGC	83.79770		83.798 §			83.79960		83.79690	83.79776	83.79878	
2 IMGC		83.79814	83.79798	83.79805	83.80030		83.79670				
1 INM		83.79817		83.798 §			83.79710		83.79793		
XXI INM				83.79802							
BCM4 INM				83.79816					83.79802		
M1 NBS		83.79834		83.79809	83.80032				83.79775	83.79885	
113 NIM				181							
10 NRC		83.79818	83.79814	83.79795	83.80028		83.79717	83.79695	83.79775		
14 NRC									83.79775		
7801 NRLM					83.80030						
7803 NRLM	83.79760	83.79818	83.79817	83.79811			83.79730		83.79775	83.79875	
Ar PRMI											

Table VI.2.a : Results of measurements on sealed cells filled with: A R G O N

°) Laboratories using argon triple point in the IPTS-68 definition. §) exact by definition (reference cell).

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	ASMW	BIPM	IMGC	INM	NBS	NIM	NML	NPL	NRC	NRLM	PRMI
Thermometer: (°)	217997	226321	PL01-6	1812283	<u>1812282</u> 1774095		<u>1731676</u>	1728839	<u>1158062</u> 1521389	7681	1842381
<u>Cells</u> :					Temperatur	e in NPL-IP	<u>TS-68</u> (K)				
4 ASMW	83.79731		83.79729								
3 BIPM		83.79670									
1 IMGC	83.79745		83.79723					83.79690	83.79666	83.79828	
2 IMGC		83.79680	83.79722	83.79732	83.79700		83.79698				83.79693
1 INM		83.79690		83.79727			83.79732		83.79683		
XXI INM				83.79729							
BCM4 INM				83.79743					83.79695		
M1 NBS		83.79700		83.79736	83.79700				83.79665	83.79835	
113 NIM											
10 NRC		83.79690	83.79736	83.79722	83.79696		83.79739	83.79695	83.79665		
14 NRC									83.79665		
7801 NRLM					83.79698						
7803 NRLM	83.79735	83.79690	83.79739	83.79738			83.79760		83.79665	83.79825	
Ar PRMI											83.79672

Table VI.3.a : Results of measurements on sealed cells filled with: A R G O N

°) underlining indicates thermometers of the "international group".

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(Reference cell: 1 IMGC)											
Thermometer:	<u>ASMW</u> 217997	<u>BIPM</u>	<u>I M G C</u> PL01-6	<u>I N M</u> <u>1812283</u> 232788	<u>N B S</u> 1812282	<u>N I M</u> 7709	<u>N M L</u> 1731676	<u>N P L</u> 1728839	<u>N R C</u> 1521389	<u>N R L M</u> 7681	<u>P R M I</u> 1842381
Cells:					Resistance	ratio at F	=100%				
1 IMGC	0.09187880		0.09191770	0.09181177	0.09184025	0.09183293	0.09196407	0.09196382	0.09184746	0.09201210	0.09174634
8 IMGC §			0.09191956	0.09193219	0.09184211		0.09196593		0.09184920		0.09174820
8 INM				0.09181113		0.09183300	0.09196500	i.	0.01984906 0.09184734 0.00184740		
BCM4 INM				0.09181120	1	*			0.09184740		
M2 NBS					0.09183952						
PPO7 NIM						0.09183293					
PP11 NIM						0.09183246					
15 NRC	0.09187939		0.09191885	0.09181123 0.09193165			0.09196460	0.09196444	0.09184772		
7801 NRLM			0.09192560	0.09182053					0.09185354	0.09201750	
O2 PRMI				0.07174095							0.09174780

Table VI.1.b : Results of the intercomparison between sealed cells filled with: 0 X Y G E N

\*) mean of two cells. \$) Cell actually measured at INM, NBS, NML and PRMI instead of the reference cell, whose values are calculated.

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Thermometer:	<u>ASMW</u> 217997	BIPM	<u>I M G C</u> PL01-6	<u>I N M</u> 1812283	<u>N B S</u> <u>1812282</u> 1774095	<u>N I M</u> 7709	<u>N M L</u> 1731676	<u>N P L</u> 1728839	<u>N R C</u> 1521389	<u>N R L M</u> 7681	<u>P R M I</u> 1842381
Cells:					Temperatur	e in LAB-IP	<u>TS-68</u> (K)	1			
1 IMGC	54.36090		ref.			54.36360		54.36072	54.36108	54.36160	
8 IMGC			54.36148	54.36163	54.36190		54.36143		54.36153		
8 INM				ref.		54.36360	54.36112		54.36149		
BCM4 INM				54.36104					54.36105		
M2 NBS					54.36131 *						
PPO7 NIM					54.50145 *						
PP11 NIM											
15 NRC	54.36107		54.36148	54.36103			54.36102	54.36088	54.36116		
7801 NRLM			54.36300	54.36337					54.36264	54.36310	
02 PRMI			ν.								

Table VI.2.b : Results of measurements on sealed cells filled with: O X Y G E N

\*) mean of two cells.

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	ASMW	BIPM	IMGC	INM	NBS	NIM	NML	NPL	NRC	NRLM	PRMI
Thermometer: (°)	<u>217997</u>		<u>PL01-6</u>	<u>1812283</u>	<u>1812282</u> 1774095	7709	<u>1731676</u>	1728839	<u>1158062</u> 1521389	7681	1842381
<u>Cells</u> :					Temperatur	e in NPL-IP	<u>IS-68</u> (K)				
1 IMGC	54.36093		54.36132			54.36125		54.36072	54.36091	54.36167	
8 IMGC			54.36180	54.36194	54.36144		54.36133		54.36136		54.36059
8 INM				54.36131		54.36127	54.36102		54.36132		
BCM4 INM				54.36135					54.36090		
M2 NBS					54.36085						
PPO7 NIM					J4.30077 *	54.36125					
PP11 NIM						54.36113					
15 NRC	54.36110		54.36167	54.36134			54.36092	54.36088	54.36099		
7801 NRLM			54.36335	54.36368					54.36247	54.36320	
O2 PRMI											54.36049

Table VI.3.b : Results of measurements on sealed cells filled with: O X Y G E N

°) underlining indicates thermometers of the "international group". \*) this value would be 54.36087 K if the 1976 (NPL-NBS) comparison is used. The mean difference (NPL-NBS) = +0.58 mK for the three PRTs of the "international group".

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(Reference cell: 7801 NRLM)													
Thermometer:	<u>ASMW</u> 217997 207278	BIPM	<u>I M G C</u> PL01-6	INM	N B S	<u>NIM</u>	<u>N M L</u> 1731676	<u>N P L</u> 1728839	<u>N R C</u> 1521389	<u>N R L M</u> 7681	PRMI 1842381		
Cells:					Resistance	ratio at F	=100%						
1 IMGC	0.00132003 0.00135358		<u>0.00140993</u>	2							0.00118828		
23 NRC			0.00140991	<u>-</u> <u>7</u>			0.00135009	0.00135063	0.00130175				
7801 NRLM	0.00131990		0.00140987				0.00135006	0.00135063	0.00130183	0.00140081	0.00118822		
H2 PRMI							T.				0.00118830		

Tab	Table VI.l.c : Results of the intercomparison between sealed cells filled with:e - H Y D R O G E N(Reference cell: 7801 NRLM)													
SMW	BIPM	IMGC	INM	NBS	NIM	NML	NPL	NRC	NRLM	PR				
7997		PL01-6				1731676	1728839	1521389	7681	184				

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A S M W Thermometer: 217997	BIPM	<u>I M G C</u> PL01-6	INM	NBS	NIM	<u>N M L</u> 1731676	<u>N P L</u> 1728839	<u>N R C</u> 1521389	<u>N R L M</u> 7681	<u>P R M I</u> 1842381
<u>Cells</u> :	3.			Temperatu	re in LAB-II	<u>?TS-68</u> (K)				
1 IMGC		13.81 §								
2 IMGC		13.80997								
23 NRC		13.80995				13.80962	13.81008	13.80810		
7801 NRLM		13.80975				13.80950	13.81008	13.80914	13.81150	
H2 PRMI										

Table VI.2.c : Results of measurements on sealed cells filled with: e - H Y D R O G E N

§) exact by definition (reference cell).

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Thermometer:	<u>ASMW</u> 2 <u>217997</u> ( <u>207278</u> )	<u>BIPM</u>	<u>IMGC</u> PL01-6 (PL02-6)	INM	NBS	<u>NIM</u>	<u>N M L</u> 1731676	<u>N P L</u> 1728839	<u>N R C</u> <u>1158062</u> 1521389	<u>N R L M</u> 7681	<u>P R M I</u> 1842381
<u>Cells</u> :				3	Temperatu	re in NPL-II	<u>2TS-68</u> (K)				
1 IMGC 2 IMGC	(13.81109)		13.81567 (13.81070) 13.81564								13.81030
23 NRC			(13.81067) 13.81562 (13.81065)			3	13.80993	13.81008	13.80927		
7801 NRLM mc PRMI	13.81057		13.81542 (13.81045)				13.80981	13.81008	13.80960	13.81340	13.81033

Table VI.3.c : Results of measurements on sealed cells filled with: e - H Y D R O G E N

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") underlining indicates thermometers of the "international group".

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(Reference cell: 2 IMGC)												
Thermometer:	<u>ASMW</u> 217990	<u>в і р м</u> 226321	<u>I M G C</u> PL01-6	INM	<u>N B S</u>	<u>N I M</u> 188640	<u>N M L</u>	<u>N P L</u> 1728839	<u>N R C</u> 1521389	<u>N R L M</u> 7681	<u>P R M I</u> 1842381	
<u>Cells</u> :					Resistance	ratio at F	=100%					
7 BIPM		0.24593950							0.24588103			
2 IMGC	0.24590300	0.24593565	0.24592185			0.24638710		0.24600010	0.24587703	0.24600250	0.24579465	
12 IMGC		0.24593500	0.24592120						0.24587753		0.24579400	
18 NRC		0.24593430	0.24592280						0.24587955			

Table VI.1.d : Results of the intercomparison between sealed cells filled with: M E T H A N E

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# Table VI.2.d : Results of measurements on sealed cells filled with: M E T H A N E

Thermometer:	<u>ASMW</u> ° 217990	<u>B I P M</u> ° 226321	<u>I M G C</u> ° PL01-6	<u>INM</u> °	<u>N B S</u>	<u>N I M</u> 188640	<u>N M L</u>	<u>N P L</u> 1728839	<u>N R C</u> 1521389	<u>N R L M</u> 7681	<u>P R M I</u> 1842381
<u>Cells</u> :					Temperatur	e in LAB-IP	<u>TS-68</u> (K)				
7 BIPM	(1978)	90.68661 90.68586							90.68539		
2 IMGC	90.68350		90.68518		ж.	90.68370		90.68372	90.68447	90.68811	
12 IMGC		90.68554	90.68503						90.68459		
18 NRC		90.68566	90.68540						90.68505		

\*

°) Laboratories using the argon triple point in the IPTS-68 definition.

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Thermometer: (°)	<u>ASMW</u> 217990	<u>B I P M</u> 226321	<u>I M G C</u> PL01-6	INM	<u>N B S</u>	<u>NIM</u>	<u>N M L</u>	<u>N P L</u> 1728839	<u>N R C</u> <u>1158062</u> 1521389	<u>N R L M</u> 7681	<u>P R M I</u> 1842381
<u>Cells</u> :					Temperatur	e in NPL-IP	<u>FS-68</u> (K)				
7 BIPM	(1070)	90.68402							90.68464		
2 IMGC	90.68350	(90.68325)	90.68386					90.68372	90.68373	90.68798	
12 IMGC		90.68298	90.68371						90.68384		90.67968
18 NRC		90.68310	90.68408						90.68430		

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Table VI.3.d : Results of measurements on sealed cells filled with: M E T H A N E

°) underlining indicates thermometers of the "international group".

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	Table VI.l.e : <u>Results of the intercomparison between sealed cells filled with</u> : N I T R O G E N (Reference cell: 2 IMGC)													
	ASMW	BIPM	IMGC	INM	NBS	NIM	NML	NPL	NRC	NRLM	PRMI			
Thermometer:			<u>45</u>	1812283				1728839	1872179					
						•								
Cells:					Resistance	ratio at F	=100%							
2 IMGC			0.12758665	0.12741804				0.12756680	0.12743810					
BCM4 INM				0.12741731					0.12743793					
33 NRC			0.12758642						0.12743740					

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Thermometer:	A S M W	BIPM	<u>I M G C</u> <u>45</u>	<u>I N M</u> <u>1812283</u>	<u>N B S</u>	<u>NIM</u>	<u>N M L</u>	<u>N P L</u> 1728839	<u>N R C</u> 1872179	NRLM	PRMI
<u>Cells</u> :					Temperatur	e in LAB-II	<u>?TS-68</u> (K)		<i></i>		
2 IMGC			63.14627	63.14596				63.14611	63.14637		
BCM4 INM				63.14579					63.14633		
33 NRC			63.14622						63.14620		

Table VI.2.e : Results of measurements on sealed cells filled with: N I T R O G E N

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Thermometer:	ASMW	<u>BIPM</u>	<u>I M G C</u> <u>45</u>	<u>I N M</u> 1812283	NBS	NIM	<u>NML</u>	<u>N P L</u> 1728839	<u>N R C</u> <u>1158062</u> 1872179	NRLM	PRMI
<u>Cells</u> :					Temperatu	re in NPL-I	PTS-68 (K)				
2 IMGC			63.14626	63.14671				63.14611	63.14562		
BCM4 INM				63.14654					63.14558		
33 NRC			63.14621						63.14545		

Table VI.3.e : Results of measurements on sealed cells filled with: N I T R O G E N

") underlining indicates thermometers of the "international group".

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Thermometer:	<u>ASMW</u> 217997 207278	BIPM	<u>I M G C</u> PL01-6	<u>I N M</u> 1812283	<u>N B S</u>	<u>NIM</u>	<u>N M L</u> 1731676	<u>N P L</u> 1728839	<u>N R C</u> 1521389 1872179	<u>N R L M</u> <u>7681</u>	<u>P R M I</u> 1842381
<u>Cells</u> :					Resistance	ratio at F	=100%				
1 ASMW	0.00861321	e -	0.00868097								
1 IMGC	0.0000000000000000000000000000000000000		0.00868062							0.00873063	
3 IMGC	0.00861229		0.00868062	0.00848493			0.00864607	0.00864550	0.00857308	0.00873063	0.00845755
BCM4 INM	0.00863278			0.00848492					0.00857334		
12 NRC	0.00861222		0.00868030				0.00864537	0.00864522	0.00855212		
1 NRLM								0.00864552	0.00855181		
2 NRLM			0.00868064					0.00864560	0.00857329	0.00873050	
Ne PRMI											0.00845771

# Table VI.1.f : Results of the intercomparison between sealed cells filled with:N E O N(Reference cell: 3 IMGC)

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Thermometer:	<u>ASMW</u> 217997	BIPM	<u>I M G C</u> PL01-6	<u>I N M</u> 1812283	<u>N B S</u>	<u>NIM</u>	<u>N M L</u> 1731676	<u>N P L</u> 1728839	<u>N R C</u> <u>1521389</u> 1872179	<u>NRLM</u> <u>PR</u> <u>7681</u> other(1978)	<u>M I</u> 381
<u>Cells</u> :					Temperatur	ce in LAB-IP	<u>TS-68</u> (K)			v	
1 ASMW											
1 IMGC			ref.							24.56150	
3 IMGC			same T			1	24.56250	24.56163	24.56346		
BCM4 INM									24.56336		
12 NRC							24.56225	24.56141	24.56342 24.56311		
1 NRLM								24.56165			
2 NRLM								24.56172	24.56363	24.56140	
Ne PRMI										,	

# Table VI.2.f : Results of measurements on sealed cells filled with: N E O N

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	ASMW	BIPM	IMGC	INM	NBS	NIM	NML	NPL	NRC	NRLM	PRMI
Thermometer: (°)	<u>217997</u> ( <u>207278</u> )		<u>PL01-6</u> (PL02-6)	<u>1812283</u>			<u>1731676</u>	<u>1728839</u>	<u>1158062</u> 1521389	7681	1842381
<u>Cells</u> :					Temperatu	ce in NPL-IP	<u>TS 68</u> (K)				
1 ASMW 1 IMGC	(24.56312) 24.5627 §		24.56308 (24.5620) 24.56281 (24.5617)							24.56220	
3 IMGC BCM4 INM	24,56236		(24.5617) 24.56281 (24.5617)	24.56195 24.56194			24.56190	24.56163	24.56125 24.56115		24.56187
12 NRC 1 NRLM	24.56234		24.56255 (24.5615)				24.56165	24.56141 24.56165	24.56121 24.56090		
2 NRLM Ne PRMI			24.56283 (24.5617)				15	24.56172	24.56142	24.56210	24.56200

## Table VI.3.f : Results of measurements on sealed cells filled with: N E O N

§) in Ref.55. °) Underlining indicates thermometers of the "international group".

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	Tabl	e – D E U	TERIUM								
Thermometer:	ASMW	<u>BIPM</u>	<u>I M G C</u> PL01-6	INM	<u>N B S</u>	<u>NIM</u>	<u>N M L</u>	<u>N P L</u> 1728839	<u>N R C</u> 1872179	NRLM	PRMI
<u>Cells</u> :					Resistan	ce ratio at	F=100%				
1 IMGC 31 NRC			<u>0.0033851</u>	5				<u>0.0033329</u>	0.0032430 0.0032338	<u>00</u> 10	

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	Tabl	le VI.2.g :	Results of	measureme	nts on seale	d cells fi	lled with:	e – D E U T	ERIUM			
Thermometer:	ASMW	BIPM	<u>I M G C</u> PL01-6	INM	<u>N B S</u>	<u>NIM</u>	<u>N M L</u>	<u>N P L</u> 1728839	<u>N R C</u> 1872179	NRLM	PRMI	
<u>Cells</u> :					Temperatu	re in LAB-	<u>LPTS-68</u> (K)					
1 IMGC			ref.					18.6753	18.6764			
31 NRC									18.6610			

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	Tabl	e VI.3.g :	Results of	measurement	s on sealed	cells fill	ed with: e	- D E U T	ERIUM		
3	ASMW	BIPM	IMGC	INM	N B S	NIM	NML	NPL	NRC	NRLM	PRMI
Thermometer:			$\frac{PL01-6}{(PL02-6)}$					1728839	1158062		
			(1102-0)								
Cells:					Temperatur	e in NPL-IP	<u>TS-68</u> (K)				
1 IMGC			18.6778					18.6753	18.6777		
31 NRC			(18.6763)						18.6623		

°) Underlining indicates thermometers of the "international group".

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#### VII. DISCUSSION OF RESULTS

#### 1. On cell intercomparison

This part of the Intercomparison fulfilled goals a) and b) of Section III.3. We shall examine the results by considering, in turn, three separate features:

- 1) differences between fixed-point realizations in different cells;
- 2) measurability of the sealed-cell devices;
- 3) merits of the different gases as candidate substances for temperature reference points.

#### 1.1 Differences between fixed-point realizations in different cells

These differences can be obtained directly from the W values in Tables VI.1. They are shown as temperature differences in Tables VII.1.x for the three definition points and for the four secondary fixed points. These calculations do not involve errors related to scale realization or thermometer calibration, provided that the same thermometer is used. Therefore, some small increase in the uncertainty levels, indicated in Section IV.3.1 for the comparison measurements, may occur in the cases indicated in Tables VI.1, where additional calculations were needed, when thermometers or cells different from the reference ones have been actually used.

However, the usual representation of these differences as a single value with associated uncertainty limits can be misleading, because all the values in the uncertainty interval are, in most cases, equally probable, since most of the uncertainty comes from what has been called in Section III "definability" of the triple point temperature, due to the shape of the melting plateau.

The single difference values in Tables VII.1.x came only from the need of representing a "typical" plateau in a Table of the data sheets, but the analysis of the data and their statistical significance should be performed on the whole uncertainty interval, seen as an "indeterminacy" of the assignable temperature value.

Therefore, Figs. VII.1.x have been drawn, which represent such intervals, centred on the value given in Tables VII.1.x and as wide as defined in Section III.3.1.

From these figures the overall distribution of the deviations shown in Figs. VII.2.x has been obtained, summing up the number of cells found at each deviation value (taken in 0.01 mK steps); from these figures the mean value has been defined as the deviation value dividing the area into two equal parts, and the standard deviation as the deviation limit which contains 66.7% of the total area of the pattern. Let us now examine the gases separately.

A R G O N (Table VII.1.a; Figs. VII.1.a and VII.2.a)

This gas has been the pivot of the intercomparison and, in fact, it has been measured by all the participating Laboratories; 14 cells have been involved and some 48 measurements resulted, leading to 36 values of differences between them.

The cell chosen as a reference is shown to well represent the average temperature value reproduced by all the cells; actually, no systematic differences are evident for any cell.

However, some asymmetry is equally evident, as all the outliers reproduced a higher temperature value. Of the 6 difference values higher than  $\sigma$ , the three obtained at NML have actually been discarded in Fig. VII.2.a: the analysis of temperature data (Section VII.2) shows that the high values are not due to an anomalously low value for the measurement on the reference cell, but to high temperature values obtained with INM, NRC and NRLM cells.

The standard deviation of the resulting distribution is  $\pm$  0.15 mK, not significantly different from the uncertainty limit of most Laboratories and from the weighted combined uncertainty of all the participating Laboratories ( $\pm$  0.21 mK).

#### O X Y G E N (Table VII.1.b; Figs. VII.1.b and VII.2.b)

This gas, which exhibits one of the flattest and more reproducible melting plateaux (using the same sample), is generally considered as a very good definition point for the IPTS. However, in the past years evidence has grown in several Laboratories of difficulties in its realization, resulting essentially from systematic differences between samples that could not be predicted from the certification of the gas purity or from thermal analysis (see Section VII.1.3).

Two of the cells involved in the Intercomparison showed definite systematic deviations: 8 IMGC, which was known to deviate from the reference cell by  $+ 0.48 \pm 0.15 \text{ mK}^8$ , and 7801 NRLM, which was found to have an average deviation of + 1.8 mK (with its companion 7802 NRLM, measured only at NRLM). The rise in systematic deviation resulted in a rise of the measurement scatter for the latter. For cell 8 IMGC, this was not evident. In fact, in addition to the fact that reproducibility of the deviation from 1 IMGC was found to be  $\pm 0.15 \text{ mK}$  at IMGC, no systematic difference appears between the two sets of measurements: the ones made at ASMW, IMGC, NIM, NPL using cell 1 IMGC as a reference, and the ones made at INM, NBS, NML, NRC, PRMI using reference cell 8 IMGC.

The two cells 1 IMGC and 7801 NRLM have been excluded from consideration in Fig. VII.2.b; nevertheless, the resulting standard deviation of the distribution is  $\pm$  0.23 mK, considerably larger than that found with argon. e – HYDROGEN

With this gas, the NRLM cell was selected as a reference since it has circulated in all the Laboratories, except PRMI. The number of measurements for this definition point of IPTS-68 is much more limited than with argon and oxygen but some interesting features of the results can be pointed out.

First of all, the reference cell appears to deviate systematically from the others: in fact, all the differences in Table VII.l.c, except for the NRC measurement on its cell, are positive. From Fig. VII.2.c a mean deviation value + 0.31 mK is obtained.

There is also a large standard deviation ( $\pm$  0.30 mK), which is certainly related to the larger uncertainty limit given by most Labora-tories. The weighted combined uncertainty is now  $\pm$  0.29 mK.

#### METHANE (Table VII.1.d; Fig. VII.1.d)

This substance has been included in the Intercomparison since its triple-point temperature value is very close to that of the condensation point of oxygen. The intercomparison of the four cells available showed some difficulties. First, the IMGC cells showed a melting range larger than the others; cell 2 IMGC was used as a reference, since it has been circulated around all the Laboratories (four of them actually measured only this cell, therefore only temperature data are available in these cases). However, some increase in the resulting uncertainty can be due to this fact, since it has been shown<sup>56</sup> that the spread of values with different melting ranges is larger at 1/F=1 than at 1/F=0 and that the values themselves are systematically lower. This may be also a reason for the fact that most of the differences are positive.

Another problem arose with the BIPM cell, which was found to present quite a high temperature value: this was apparently due to a shift of the triple-point temperature of the cell, observed at BIPM since 1978 (Table VII.1.d; also the companion cell 6 BIPM is reported to have suffered the same drift). This instability has been the only one reported for a cell taking part in this exercise, but some instability in the realization of the triple point of methane in sealed cells had been previously reported by NRLM<sup>41</sup>, and explained by spin conversion.

For these reasons, Fig. VII.2.d has not been drawn; when no problems are evident, differences between cells could be limited within about  $\pm$  0.3 mK.

#### NITROGEN (Table VII.1.e; Fig. VII.1.e)

This substance was introduced into the exercise at a very late stage, so that only three cells were involved, with a limited circulation. For this reason Fig. VII.2.e has not been drawn.

However, the results available are very good. This fixed point

appeared to be easily achieved and showed quite a limited melting range; the difference values in Table VII.1.e are small, so that an uncertainty limit of about  $\pm$  0.15 mK can be stated, similar to that of argon.

### <u>N E O N</u> (Table VII.1.f; Figs. VII.1.f and VII.2.f)

The neon triple-point temperature is quite close to that of the normal boiling point and can therefore be considered for substitution for the latter in the Scale definition, but some problems can, in principle, arise from normal neon being an isotopic mixture.

A comprehensive batch of cells has been available, filled with gas of different manufacturers from all the continents of the world. The difference values are collected in Table VII.1.f; there is evidence from Table VI.3.f that the correct value of the difference for the ASMW cell measured by the same Laboratory is the lower one.

Figure VII.2.f shows no deviation from the reference cell and a small standard deviation ( $\pm$  0.20 mK).

However, the figures reveal small systematic differences between some of the cells. The mean deviation for each cell is given in Fig. VII.1.f: the extreme values are  $\pm 0.30 \pm 0.04$  mK for the ASMW cell and  $- 0.20 \pm 0.15$  mK for the NRC cell. Therefore, the distribution of Fig. VII.2.f is biased by these systematic differences, which broaden it to some extent, though they are of low statistical significance. For each cell, a standard deviation of  $\pm 0.15$  mK could be probably more appropriate.

#### e - D E U T E R I U M (Table VII.l.g)

Its temperature value is situated very usefully in between those of the hydrogen and neon triple points, so that it could be considered for a Scale definition. It has been included in this exercise in a very late stage and only two cells were available.

For this reason and because large differences have been found (see Table VI.2.g and VI.3.g) the discussion will be postponed to Section VII.2.

Thermometer:	<u>ASMW</u> <u>217997</u> 217990 (207278)	<u>в і р м</u> 226321	<u>I M G C</u> PL01-6	<u>I N M</u> 232788 <u>1812283</u>	<u>N B S</u> 1774095	<u>N I M</u> 7703 188640	<u>N M L</u> 1731676	<u>N P L</u> 1728839	<u>N R C</u> 1521389	<u>N R L M</u> <u>7681</u> 1781356	<u>P R M I</u> 1842381
Cells:				Temperatur	e differenc	<u>es</u> : cell(L	AB)-cell(re	f) (mK)			
4 ASMW	-0.07		+0.06								
3 BIPM		-0.15				13					
1 IMGC	ref.	ref.*	ref.	ref.*	ref.*	ref.	ref.*	ref.	ref.	ref.	ref.*
2 IMGC			-0.02								
1 INM		+0.07		-0.07			+0.32		+0.17		
XXI INM				-0.06		-0.06					
BCM4 INM				+0.08		(assumed)			+0.26		
M1 NBS		+0.19		+0.02	-0.00				-0.01	+0.07	
113 NIM						-0.07					
10 NRC		+0.03	+0.14	-0.12	-0.03		+0.39	+0.03	-0.01		
14 NRC						+0.08			-0.01		
7801 NRLM					-0.02					-0.03	
7803 NRLM	-0.05	+0.03	+0.18	+0.04			+0.71		-0.01	-0.03	
mc PRMI										(mean)	-0.08 -0.02

Table VII.1.a:	Results	of	the	intercomparison	between	sealed	cells	filled	with:	Α	RG	ON
	-			(Reference cell	1: 1 IMG0	C)			11.17			

\* through cell 2 IMGC.

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CE	ELLS:	Measur	ed at:
4	ASMW	2000000000000	ASMW
-	<u></u>	200000000000000000000000000000000000000	IMGC
3	BIPM	x0000000000000000000000000000000000000	BIPM
2	IMGC	xaaaaaaaxQxaaaaax	IMGC
1	INM	200000000000000000000000000000000000000	BIPM
		νασοσοσοαα	INM
		X20202020200000K	NML
		20000000000000	NRC
XXI	INM	xxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxx	INM
BCM4	INM	χαατΟιαοσοασασασαα	INM
		30000000000000000000000000000000000000	NRC
Ml	NBS	30000000000000000000000000000000000000	BIPM
		300000X()3000000X	INM
		xxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxx	NBS
		χαροροαχΟχορορακ	NRC
		xccccCrccccccc	NRLM
113	NIM	200000000000000000000000000000000000000	NIM
10	NRC	x2222222222220220222222222	BIPM
		χθασσοσσοσασα	IMGC
		x0xxxxxxxxxxx	INM
		χαροσοροα	NBS
		XXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXX	NML
		xxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxx	NPL
		200000000000000000000000000000000000000	NRC
14	NRC	200000000000000000000000000000000000000	NIM
		200000000000000000000000000000000000000	NRC
7801	NRLM	xxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxx	NBS
		300000000000000000000000000000000000000	NRLM
7803	NRLM	x02020202020X0X202X	ASMW
	· · · · · · · · · · · · ·	x0200000000000000000000000000000000000	BIPM
		20000000000000	IMGC
		xxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxx	INM
		XXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXX	NML
		xxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxx	NRC
		200000000000000000000000000000000000000	NRLM
	PRMI	xxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxx	) PRMI
	8	6 4 2 0 2 4 6 8	
	(-)	(x0.1 mK)	
	<b>D</b> 4 - 1	ITT 1 of Differences between collective state A D C C N	
	rig. \	II.I.a. Differences between cells filled with A K G U N	

and the reference cell 1 IMGC.



Fig. VII.2.a: Distribution of the differences between cells filled

with ARGON and reference cell 1 IMGC.

5.	Table VII.1.b: <u>Results of the intercomparison between sealed cells filled with</u> : O X Y G E N (Reference cell: 1 IMGC)											
	ASMW	BIPM	IMGC	INM	NBS	<u>NIM</u>	<u>N M L</u>	<u>N P L</u>	NRC	NRLM	PRMI	
Thermometer:	<u>217997</u>		<u>PL01-6</u>	1812283 232788	1812282	<u>7709</u>	1731676	<u>1728839</u>	1521389	7681	<u>1842381</u>	
Cells:				Temperature	e differenc	es: cell(L	AB)-cell(re	f) (mK)				
1 IMGC	ref.		ref.	ref.*	ref.*	ref.	ref.*	ref.	ref.*	ref.	ref.	
8 IMGC			+0.48						+0.45			
8 INM				-0.15		+0.02	+0.24		-0.03			
BCM4 INM				-0.14					-0.01			
NBS					•••							
PPO7 NIM						-0.01						
PP11 NIM						-0.12						
15 NRC	+0.15		+0.29	-0.12			+0.14	+0.16	+0.08			
7801 NRLM			+2.02	+2.23					+1.56	+1.40		
mc PRMI			E,							(mean)	+0.39 +0.09	

\* through cell 8 IMGC.

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\*7

8	IMGC						*****	XXXXXXX		IMGC		
						х	000000000000000000000000000000000000000	XXXXX		NRC		
8	INM			200000000	0,0202020					INM		
			2000	20000000000	xxxxxx0xxxxxx	x000000000x	XXXXXXXXXXX			NIM		
					x	******	xxx			NML		
				2000	200000000000000000000000000000000000000	xx				NRC		
BCM4	INM			20000000	x0xxxxxx					INM		
				200	xxxxxx0xxxxxx	xxx				NRC		
	NBS		2		xxxxx					NBS		
PP07	NIM		xxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxx									
PP11	NIM	22	000000000		xxxxxx0xxxxxx	2000000000				NIM		
15	NRC				20000000		xx			ASMW		
						200000000	2000000			IMGC		
				XXXXXXXXXX	200000000000000000000000000000000000000					INM		
					x0xxxxx	xxxxxxxxxxx				NML		
					20000	*****	:			NPL		
					200000000000000000000000000000000000000	2000000				NRC		
	PRMI			20000000	xxxxxx0xxxxxx	20000000000	x		(mean)	PRMI		
	8	6	4	22	0	2	4	6	8			
	(-)				0		(w0 1 -	(·	+)			
							(XU+1 I					



Fig. VII.1.b: Differences between cells filled with 0 X Y G E N and the <u>reference cell 1 IMGC</u>.



Distribution of mean difference values of Table VII.1.b:

(0)

X XXXX	X	Х	X	XX	XXX	Х	X
X		Х					

Fig. VII.2.b: Distribution of the differences between cells filled

with OXYGEN and reference cell 1 IMGC.

Table VII.1.c: Results of				the inter (Ref	comparison b erence cell:	etween seal 7801 NRLM)	ed cells fi	lled with:	е-НҮД		
	ASMW	BIPM	IMGC	INM	NBS	NIM	NML	NPL	NRC	NRLM	PRMI
Thermometer:	<u>217997</u> 207278		<u>PL01-6</u>				1731676	1728839	1521389	<u>7681</u>	<u>1842381</u>
Cells:				Temperat	ure differen	ces: cell(	LAB)-cell(r	ef) (mK)			
1 IMGC	+0.52*		+0.24								+0.24
2 IMGC			+0.21								(assumed)
23 NRC			+0.19				+0.13	0.00	-0.33		
7801 NRLM	ref.		ref.				ref.	ref.	ref.		ref.
mc PRMI										(mean)	+0.34 +0.57

\* difference of NPL temperature values of thermometers 217997 and 207278: there is a possible error (see Neon, Table VII.1.f).

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Distribution of values of Table	<pre>mean difference VII.1.c:</pre>	(0)			
	X	x x	XX X	x	X

Fig. VII.2.c: Distribution of the differences between cells filled

with e - HYDROGEN and reference cell 7801 NRLM.

Table VII.l.d: <u>Results of the intercomparison between sealed cells filled with</u> : M E T H A N E (Reference cell: 2 IMGC)											
Thermometer:	<u>A S M W</u> 217990	<u>в і р м</u> 226321	<u>I M G C</u> <u>PL01-6</u>	INM	<u>N B S</u>	<u>N I M</u> 188640	<u>NML</u>	<u>N P L</u> 1728839	<u>N R C</u> 1521389	<u>N R L M</u> <u>7681</u>	<u>P R M I</u> 1842381
Cells:				Temperature	e difference	es: cell(LA	B)-cell(ref	E) (mK)			
7 BIPM 2 IMGC	(1978) ref.	+0.89 +0.14 ref.*	ref.			ref.		ref.	+0.91 ref.	ref.	ref.
12 IMGC 18 NRC		-0.31	-0.15 +0.22						+0.11		

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\* through cell 12 IMGC.

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(x0.1 mK)

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Table VII.l.e: <u>Results of the intercomparison between sealed cells filled with</u> : N I T R O G E N (Reference cell: 2 IMGC)											
	ASMW	BIPM	IMGC	INM	NBS	NIM	NML	NPL	NRC	NRLM	PRMI
Thermometer:			<u>45</u>	1812283			1	1728839	<u>1872179</u>		
<u>Cells</u> :				Temperatu	re differe	nces: cell(	(LAB)-cell()	cef) (mK)			
2 IMGC			ref.	ref.				ref.	ref.		
BCM4 INM				-0.17					-0.04		
33 NRC			-0.06						-0.17		

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Fig. VII.1.e: Differences between cells filled with N I T R O G E N and the reference cell 2 IMGC.

Table VII.1.f: <u>Results of the intercomparison between sealed cells filled with</u> : N E O N (Reference cell: 3 IMGC)											
	ASMW	BIPM	IMGC	INM	NBS	NIM	NML	NPL	<u>N R C</u>	NRLM	PRMI
Thermometer:	<u>217997</u> 207278		<u>PL01-6</u>	1812283			1731676	1728839	<u>1521389</u> 1872179	<u>7681</u>	<u>1842381</u>
Cells:				Temperatur	e differenc	es: cell(L	AB)-cell(re	f) (mK)			
1 ASMW	+0.74		+0.26								
1 IMGC	+0.34 9		0.00								
3 IMGC	ref.		ref.	ref.			ref.	ref.	ref.	ref.*	ref.
BCM4 INM				-0.03					-0.10		
12 NRC	-0.06		-0.27				-0.25	-0.24	-0.04		
1 NRLM								+0.03	-0.35	-0.13	
2 NRLM			+0.03					+0.08	+0.17	-0.11	
mc PRMI										(mean)	+0.13 +0.06

\* through cell 1 IMGC. §) with thermometer 207278 (see Table VI.3.f).

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# Measured at:

CELLS:

1	<u>ASMW</u>	+ 0.30 <u>+</u> 0.04	200000000000000000000000000000000000000	A S M W I M G C							
1	IMGC		x2222222CXQx222222C	IMGC							
BCM4	INM	- 0.07 <u>+</u> 0.04	300000000CC000000CCC	I N M N R C							
12	<u>N R C</u>	- 0.20 <u>+</u> 0.15	- 0.20 <u>+</u> 0.15 xxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxx								
1	NRLM	- 0.05 <u>+</u> 0.08	x200000000x200000000 x0x000000000x	N P L N R L M							
2	<u>NRLM</u>	+ 0.04 <u>+</u> 0.15	x22222222222222222 x222222222222222 x222222	IMGC NPL NRC NRLM							
	PRMI	<u>I</u> x000000000000000000000000000000000000									
	8(	-) 6	4 2 0 2 4 6 0 (x0.1 mK)	8 8							

# Fig. VII.1.f: Differences between cells filled with N E O N. and the reference cell 3 IMGC.



Distribution of mean values of Table VII.	n differend .l.f:	e		(0)				
x x	X X	x	X XX	X X	X X X	x	X	x

Fig. VII.2.f: Distribution of the differences between cells filled

with N E O N and reference cell 3 IMGC.

	Table VII.l.g: <u>Results of the intercomparison between sealed cells filled with</u> : e - D E U T E R I U M (Reference cell: 1 IMGC)										
	ASMW	BIPM	IMGC	INM	NBS	NIM	NML	NPL	NRC	NRLM	PRMI
Thermometer	:		PL01-6					1728839	1872179		
								-			
<u>Cells</u> :				Temperatu	ire differe	nces: cell	(LAB)-cell(	ref) (mK)			
1 IMGC			ref.					ref.	ref.		
31 NRC									-15.4		

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1.2 Measurability of the sealed-cell devices.

The Intercomparison permitted one to study the realization of the same thermodynamic state in cells of different design, as has been shown so far, but also to check the performance of the same cell when measured by different users. This is important, since a measurement with a sealed cell should no longer be considered an "experiment" but a test, the performance of a device instead of the running of an experimental apparatus.

For the former statement to be true, the results of the measurements should not depend critically on details of the test: this quality is called here "measurability", that is, the disposition of a cell to reproduce the same temperature value of the fixed point when used in as wide a variety of situations as possible (or convenient).

The Intercomparison tested this quality, both by confronting different cell designs and by using different cryostats and measurement procedures.

Sixteen models of cells were considered, showing quite different geometries and using copper and stainless steel for the fabrication in many possible combinations. The weight of the devices varied over the range 80-370 g, sealing a sample of mass ranging from 0.02 to 0.3 mol. Ageing of the sample in the cell after sealing ranged from one month to eight years.

The apparatuses used for the measurements were all vacuum adiabatic calorimeters, since this was mandatory, but the design was extremely varied; both flow and bath cryostats were included, with one or two isothermal shields. The cells were suspended in the experimental chamber either with nylon threads or with plastic or stainless steel fittings. The overall quality of the thermal equipment ranged from purposedly simple experiments (e.g. ASMW, IMGC above 54 K) to extremely careful calorimetry (e.g. NBS). Some experiments were computer assisted (INM, NML). Usually, the apparatuses used for testing these devices have been the same as are used in the Laboratories to reproduce or transfer LAB-IPTS-68, so that this exercise allowed also a test of the traceability of temperature measurements between Laboratories.

The answer to the question whether the results were affected or not by this variety of experimental conditions and cell design is evident from the preceeding Chapter. As can be seen particularly from Figs. VII.1.x, in most cases the mean value is within (or not significantly outside) the uncertainty limits of the differences measured by each Laboratory (strip boundaries), which indicates a small probability for experimental circumstances to produce systematic errors exceeding the limit of  $\pm \sigma$  (as a matter of fact, almost all the difference values shown in Tables VII.1.x, and reported at the bottom of Figs. VII.2.x, are within these limits). This seems to be the actual limit of uniformity in temperature measurements at these fixed points around the world, which is about twice or three times larger, depending on the substance, than the reproducibility limit for most Laboratories, which is close to  $\pm 0.1$  mK.

Within these limits, cell design did not appear to have any syste-

matic influence on the quality of the results. This means that no better or worse reproducibility for the differences from the reference cell could have been obtained with different cell models. Consequently it is possible to state that every model tested was equally good in reproducing the correct value for the thermodynamic state temperature (of course, differences due to sample contamination are not under consideration here).

Nevertheless, not every model allowed one to obtain this result with the same ease or, sometimes, with the same confidence. There are three the main parameters which allow one to check the quality of the cell performance in a melting experiment:

- a) melting range;
- b) overheating when heating the cell;
- c) recovery time to equilibrium after overheating.

The values obtained during each experiment of the Intercomparison are recorded in the data sheets of Section V. Let us consider them separately.

a) <u>melting range</u> - from a thermodynamic point of view, this is defined only by sample purity and, consequently, it should be as reproducible as the value of the triple-point temperature. The shape of the melting plateau, too, should be quite similar (though not necessarily a straight line) for samples of comparable purity of the same substance. This has not been observed during the Intercomparison. The Figures associated with the data sheets of Section V show all the melting plateaux obtained in different Laboratories: it is evident that the same cell can produce curves of quite different shapes. That applies to all cell models, no one showing sensible reduction of the dispersion: large differences of the melting range for different cells are not due to cell design but to the purity of the sample.

Therefore, it is impossible, as a rule, to correlate the melting behaviour with parameters such as the materials of the cell body or the geometry of the sample interface. Speculations have been attempted in many Laboratories to guess about the distribution of the liquid-solid interface in the cell during melting, which depends on the distribution of the impurities, on the thermal map both during heating and at equilibrium and on the internal geometry of the cell. However, except when melting behaviour is dominated by relevant sample contamination (cells 2 CH4 IMGC, 12 CH4 IMGC, 1 eD2 IMGC, 33 NRC; 7801 02 NRLM is a special case that will be examined in the next Chapter), the melting pattern appears to depend completely on the thermal conditions of the experiment, which essentially means non-adiabaticity for the environment or not a true-equilibrium state for the sample. Since the sensitivity of a cell design to these conditions can be qualitatively related to an overheating factor and to the thermal reponse time, the discussion of this point is postponed to the following sections.

b) <u>overheating factor</u> - When the sample of condensed substance enclosed in the cell is crossed by a heat flux a temperature-gradient distribution develops and the thermometer, which is thermally linked to the sample, indicates an overall change of the temperature value. In addition, gradients will develop also in the cell body. Therefore, when the cell is heated with a heater mounted on the cell body, in order to melt the sample, an increase of temperature is observed and when heating is stopped, the temperature recovers to a lower value (which is supposed to be an equilibrium one). For this reason a melting plateau must be made by steps and not with the continuous heating method, if maximum accuracy is the aim. The overheating of the cell for heating at constant rate is known to always rise with the melted fraction: this is explained by an increase of overheating in the liquid phase, since there is experimental evidence that the solid phase tends to be shielded by the liquid one.

The size of the overheating produced by the cell heater for a given heating power fully depends on the details of each model design. Since it is essentially the same as that produced by any kind of heat exchange of the cell with its surroundings, no matter how imposed, the smallest is the temperature change measured by the thermometer, the best should the thermal behaviour of the cell be considered, because the least will be the sensitivity of the cell behaviour to heat exchanges with the surroundings. Although a study of the overheating behaviour versus heating power is possible for each of the cell models, this specific measurement has only been made recently for the ASMW cell<sup>55</sup>; in a few other cases the whole overheating profile (what one could define as the "overheated plateau") has been reported. During the Intercomparison only a few Laboratories supplied the overheating profile (ASMW, BIPM, IMGC, NPL (few)), so that it has not been included in this Report, but only a single point has been selected to represent the sensitivity of each cell to overheating. An overheating factor has been defined at F=50%, referred to the unit power P (in mW); this is not exactly true if overheating is not proportional to P. It must be pointed out that the sensitivity of the cell to freezing is different, but it is generally agreed that heat leaks should be kept positive to avoid the risk of condensation of substance in parts of the cell sufficiently decoupled from the thermometer to produce a lowering of temperature due to evaporation (though this effect in a sealed cell is much less dramatic than in a conventional apparatus).

The reason for selecting the point at F=50% can be better explained with Fig. VII.3. Melting begins with no overheating if a proper procedure has been followed for solidification of the sample and the subsequent warm up, so that the beginning of melting has been reached slowly. Then, if P is such that the energy supplied per minute is of the order of a hundredth of the total enthalpy of sample melting, overheating increases to quickly reach an almost steady value, only slowly increasing with the melted fraction. Eventually, past 60% to more than 90% of total melted fraction, depending on cell model and on substance, it increases again rapidly. In several cases it is so high, close to the end of melting, that it is difficult to locate the very end of the plateau, resulting in an uncertainty of few percent on the total length of melting. The selection of F=50% for the definition of typical overheating brings into consideration the steady portion of the "overheated plateau", after the initial transient and before the variable final portion.



Fig.VII.3: Definitions of overheating factor and recovery time.

It is possible now to discuss the statement at the end of point a). In fact, the shape of the "overheated plateau" shows the evolution of the sensitivity of the cell to an external heat flux, and consequently the shape of the equilibrium plateau can be considered as the limit of the former for very small heat flux. As a matter of fact, also the equilibrium plateaux often show a curvature upward, sharply increasing above F=50-90% (particularly evidenced in the 1/F plot), which corresponds to the sharp increase of overheating. This correlation is easily explained by a loosening of the thermal coupling between thermometer and solid-liquid interface. The temperature of the cell (as indicated by the thermometer) becames more and more influenced by the residual heat exchange with the cryostat; therefore, after heating, the sample does not return to equilibrium but to a steady state, corresponding to a temperature higher than the equilibrium one (for heat intake). This error may be different from model to model and be relevant beginning from different F values.

The extent of non-adiabaticity during each experiment has been controlled in two ways. The first, by asking the Laboratories to report the temperature drift observed just before beginning and after completion of melting: therefore, from the heat capacity of the cell (approximately calculable from its total mass) the residual heat leak could be calculated. Since the two drift rates were in general quite closetogether, only the average value is reported in the data sheets. The second control is through the <u>measured</u> value of the enthalpy of melting. This parameter is a constant of each cell: therefore the deviations from the calculated value (or assumed value, when the mass of the sample must be assumed) are due only to lack of adiabaticity of the calorimeter. The data sheets often show large discrepancies, up to about 30%, in these values, indicating bad thermal control: most of the wrong values are too small, which means large heat inflow.

The overheating factor varies widely from cell to cell, in the range 0.04-0.3 mK/mW for CH<sub>4</sub> (but 5 mK/mW at BIPM, where the cell heater has been mounted on the thermometer block of cell 12 IMGC, instead of externally on the body), 0.02-3 mK/mW for Ar (but 5 and 24 mK/mW respectively at BIPM and INM, for the same reason, on cell 2 IMGC), 0.01-0.1 mK/mW for N<sub>2</sub>, 0.01-6 mK/mW for 0<sub>2</sub>, 0.02-0.7 mK/mW for Ne and 0.2-10 mK/mW for H<sub>2</sub>. Moreover, large differences (up to more than one order of magnitude) were found for this parameter when the same cell was measured in different Laboratories.

In conclusion, although the collection of these secondary parameters was incomplete, a picture arises from the data sheets of a quality of thermal control that is very different from experiment to experiment, and often less effective than expected.

The discussion on overheating does not fully help in explaining the large variability observed in the melting-onset temperature. The Intercomparison was unable to provide any enlightenment on this point, which seems to be related, for a given sample, to the possibility of gradients inside the sample depending on its thermal history (freezing and warming rates, stabilization times, annealing), or to the distribution of impurities in it.

c) recovery time - The return of the cell to equilibrium after each heating pulse proceeds exponentially, but two time constants are involved: a shorter one related to the cell body and a longer one due to the sample, whose thermal diffusivity is always low. For this reason, and because not all the Laboratories recorded (on chart or by computer) the whole pattern of the plateaux, it was preferred not to define recovery time by means of a time constant, but in a simple and uniform operational way: as the recovery of temperature to within 0.1 mK of the value considered as the equilibrium one by each Laboratory (Fig. VII.3). A much tighter limit could have been selected (e.g. 0.02 mK), since many of the cells are fast and show small overheating, but this was not possible in every case. On the other hand, for the purposes of the Intercomparison, recovery within the uncertainty limit and expressed in minutes was a sufficient goal. Nevertheless, there has been some lack of uniformity in the Laboratory reports in applying the criterion, especially concerning the definition of equilibrium temperature. This is particularly evident in the case of INM, whose statistical criterion always led to larger values.

The values of recovery time are recorded in the data sheets: when two figures are given, the first refers to small melted fractions (when overheating is small), the second to very high melted fractions (where overheating is high). Recovery time was generally observed to increase up to one order of magnitude from beginning to the very end of melting (but it remains almost stable at the smaller value for most of plateau).

Several data sheets show also a dramatic variety of values obtained in different Laboratories for the same cell, up to more than two orders of magnitude, quite outside the possible spread due to different interpretations of the definition. As a matter of fact, the thermal coupling of the cell with the cryostat, pointed out in b), also affects thermal recovery time.

Screening the results from these artifacts, the Intercomparison has shown that recovery time depends for each cell model on the substance, and for each substance on cell design. Among the substances investigated here, methane is the slowest, followed by nitrogen and argon; re-equilibration is quite fast in oxygen, neon and hydrogen. Among the cell models, considering the average behaviour, the all-copper cells (e.g. NRC, NRLM) seem to have the faster response, while the massive NBS argon cell was the slowest. However, differences are smaller than the systematic differences between Laboratories. As a rule, at 54 K or less, after 10 minutes the temperature can be considered to be at equilibrium well within the uncertainty limits, while one should wait twice as long as that for argon and nitrogen (and methane, when the purity is such that the melting range is within 1 mK; when it is around 5 mK, 30-40 minutes are required).

There are two reasons why it is important for cells to have a reasonably fast thermal recovery. The first is the opportunity to limit the time required to perform a full melting, since this limits non-adiabaticity errors: the effect of a slow cell is worse than proportional to the time constant, since these cells necessarily show also high overheating factors, slow recovery being a symptom of bad thermal coupling with the <u>whole</u> sample. The second is to have more reliably true equilibrium states: in fact, a slow cell can average fluctuating residual heat exchanges with the surroundings and stabilize itself in a steady overheated state instead of in the true equilibrium state.

#### 1.3 Gases as candidate substances for reference points.

The Intercomparison also collected useful information about the merits of the different substances for their use in the realization of temperature reference points. All these gases have already been studied in several Laboratories, and, on the other hand, there is a very limited choice of fixed points below 100 K. However, the Intercomparison allowed one for the first time to study the same sample of gas in different Laboratories, for a better understanding of how much the measured properties could be influenced by the Laboratory equipment and procedures (as in a round-robin test); and different samples in different cells, which is important to check for differences in gases produced by different manufacturers in the world (particularly for neon). Only the influence of the cell model on the measured properties could not be resolved in this exercise, as the same model of cell was never filled with gases of different countries (but this has already been done during other studies in some Laboratories). Of course, no check of the effect of impurities was possible, the devices being sealed, but it was possible, on the other hand, to test the quality of research-grade gases (though the Laboratories generally circulated devices known to work properly).

#### ARGON

No problems were found in reproducing very accurately the triplepoint temperature of this gas. The melting range was always a few tenths of a millikelvin and the reponse time of the devices quite fast, allowing one to perform a full melting in few hours. This gas is therefore extremely good and reliable for realizing a reference point of a temperature Scale.

#### OXYGEN

The Intercomparison has confirmed the systematic differences between cells which are possible with this gas, already observed at  $\rm IMGC^8$ ,  $\rm INM^{13}$ ,  $\rm NBS^{13}$ ,  $\rm NIM^{13}$  and  $\rm NRC^{36}$ , where differences higher than a millikelvin have been found.

The effect consists in a <u>rise</u> of the triple-point temperature with no apparent change in melting range (or recovery time), so that the systematic errors cannot be detected otherwise than by comparison of the device with a certified cell. It has been observed that this error never occurred with oxygen home-made by decomposition of KClO<sub>4</sub> or KMnO<sub>4</sub> (NBS, NRC); no data are available yet about the behaviour of commercial research-grade oxygen obtained by electrolysis of water versus that obtained by distillation of air (most common).

In particular, two of the cells taking part in the Intercomparison suffered from a systematic deviation: 8 IMGC which was higher by + 0.48 mK and 7801 NRLM which was higher by 1.8 mK.

The problem is puzzling, since there has been so far no experimental evidence of the reason for these systematic errors. The only known phenomenon that could cause a rise in the triple-point temperature of that kind is contamination with argon (see Ref.36 and references in Ref.52). It forms with oxygen a mixture showing a peritectic around 10% Ar in 0, at a temperature 0.2 K higher than that of pure oxygen. The initial slope of the mixture diagram is such that 100 ppm of argon are necessary to raise the triple-point temperature by 1.5 mK, and there is no separation of the liquidus and solidus lines, leaving the melting range unaffected. Unfortunately, there is not a single confirmation, so far, of such a contamination with argon for the samples used in the Laboratories<sup>8,13</sup> all the analyses, batch or especially made on the bottles used for filling the cells, gave argon contamination never exceeding 10 ppm (see also data sheets).

Consequently, this gas still requires careful analysis for further use in a temperature Scale: the problem needs to be eliminated or a reliable procedure must be found to avoid these systematic errors. Actually, there is not even experimental evidence at present, but only a high probability of occurrence, that the lower value reproduced by most of the cells is the right one to assign to the triple-point temperature of pure oxygen.

The other characteristics of melting are extremely good: melting range is very narrow and recovery to equilibrium very fast.

#### e – HYDROGEN

The main problem in assessing the quality of this gas does not come from the limited number of measurements during the Intercomparison but from the broadening of the uncertainty limits for many of the measurements, which can be due to increasing difficulties both in resistance measurements (1  $\mu$ Ω corresponds to about 0.15-0.20 mK) and in thermal control. As a matter of fact, the very narrow melting range and the very fast recovery to equilibrium are the prerequisites of a very good fixed point, but it has been impossible to compare the cells and to check reproducibility at a convenient accuracy level. Therefore, it is quite possible that this fixed point reproduces better than could be inferred from Fig. VII.2.c.

No problems have been found with the catalyst contained in the cells. It must be pointed out that the mass of the sample cannot be known accurately if cryogenic condensation is used to fill the cell, since the catalyst is losing weight in the activation process.

#### METHANE

This substance confirmed that it <u>may</u> be good for the realization of a fixed point but this is not certain at present. In fact, as happens with oxygen, there is not enough expertise at present to avoid systematic errors. In this case, unstable temperature values have been found, although limited to two Laboratories (BIPM<sup>13</sup> and NRLM<sup>41</sup>): the cells involved were completely different in design, the first being all made of stainless steel, the second all of copper. Also, large melting-range differences have been found with gases of the same nominal purity (confirmed by specific analyses, for some of them); gas with better nominal purity gave even worse results both at IMGC and BIPM<sup>13</sup>. Finally, there is some evidence that the geometry of the cell can be an influence parameter for that.

Considering also the slower recovery time of this gas, the superior performance of the nearby argon triple point leaves very little interest in considering this fixed point as a candidate definition-point of a temperature Scale.

#### NITROGEN

Contrary to the considerations with methane, the limited number of measurements made with this substance were sufficient to reveal the very high quality of this fixed point, which appears to be very much the same as that of argon. Probably, more work should be done to ascertain the influence of impurities (especially argon) on sample-to-sample reproducibility of the triple-point temperature.

#### NEON

Quite a lot of interest was focused on this substance, since it was expected to show problems connected with isotopic composition and distillation during melting<sup>6</sup>. The measurements made during the Intercomparison on a comprehensive set of cells showed a melting plateau and a thermal behaviour of very high quality, so that a reproducibility of the same level as argon cells should be expected for each device. However, there has been evidence, though not conclusive, that some systematic differences between cells may occur: a scatter of the mean values of the cells close to half a millikelvin has been found (Fig. VII.1.f). Again, as with oxygen and methane, one must conclude that, although these results could represent an extreme situation, a reliable assessment of the accuracy of the realization of this fixed point must include, at present, a statement that differences up to 0.5 mK can be found from cell to cell, probably due to differences in isotopic composition of the gas. If a better accuracy is aimed at, each new device should be compared with certified cells.

However, the correlation between these differences and the isotopic composition has not been demonstrated so far; consequently, it is impossible, at present, even to guess which one of the cells taking part in the Intercomparison most closely approaches the realization of the triple point of neon of natural composition (as defined by IPTS-68).

#### e – D E U T E R I U M

An evaluation of this substance was attempted in the Intercomparison on the only two cells available. In contrast to all other gases considered here, the purity of the samples has been very poor (nominal purity 99.86% for the IMGC and 99.6% for the NRC cell). Measurements on the IMGC cell, made in three Laboratories, demonstrated that it is possible to obtain an agreement compatible with the quality of the cell (melting range of about 5 mK): the temperature values (Tables VI.2.g and VII.3.g) actually show an agreement limited to about 2 mK, due to thermometer calibration problems at IMGC and NRC.

On the other hand, the difference between the two cells is very large (-15 mK): it is large also compared with another recent (conventional) realization at NML:  $\pm 15 \, \text{mK}^{50}$ . In addition, the differences between the triple-point temperature of normal and equilibrium deuterium were widely different, being 18 mK, 51 mK and 51 mK at NML, IMGC and NRC respectively. Recent discussions with specialists of deuterium production and handling seem to indicate that the problem consists of a contamination with  $\mathrm{HD}^{16}$ , and that this is almost unavoidable. The only hope of avoiding it resides in a very special fabrication of the cell and in a direct filling at the production plant, where HD contamination is known to be limited (before any handling) to a few tens of parts per million.

#### 2. On Scale realizations

The results discussed in this Chapter should in no way be confused with those of the former Chapter. In fact, the former results do not depend on assigned temperature values, except in the limited number of cases where they were obtained indirectly, involving some calculations indicated earlier.

Although it was not necessary for cell intercomparison, Laboratories were asked to use calibrated thermometers for the measurements, since some additional useful information can be obtained, and in order to fulfill goal c) of the Intercomparison (see Sect.III.3 and IV.3.2). First of all, for Laboratories where the Scale is not based on sealedcell realizations of the fixed points it is possible to obtain a relationship between the cells and the conventional realizations (or LAB-IPTS-68 for a specified thermometer, when secondary points are not realized). Secondly, it is possible to obtain a relationship between these measurements and the exercise at NPL in 1975 when thermometers belonging to what has been called here "international group" were used. Finally, it is possible to relate the measurements on some cells to the others, even in the (few) cases where only one cell of a substance was measured by a Laboratory.

Tables VI.2.x and VI.3.x collect the same information on temperature values as are in the data sheets of Section V, for LAB-IPTS-68 and NPL-IPTS-68 respectively, and for all cells. They show a dispersion of values which depends both on differences between cells, already analyzed in Tables VII.1.x, and on differences due to the thermometers or to Scale realizations. If the latter contributions to total uncertainty were small, the set of results would present the same dispersion of values in Tables VI.2-3 and in Tables VII.1.x. This was not the case, as one will see later for each substance.

In addition to the inaccuracy of the thermometer calibrations and to differences between National Scales, with secondary fixed points there is also a contribution due to IPTS-68 non-uniqueness. For the thermometers of the "international group" the amount of this contribution is known, since they were not only calibrated at NPL but also compared to the "master" thermometer 1728839 in the whole temperature range. Since many of the Laboratories used them, it is possible for most of the temperature data of Tables VI.3.x to apply the non-uniqueness correction and obtain the temperature values on NPL-IPTS-68(1728839). They are reported in Tables VII.2.a,d,e,f for the secondary fixed points, excluding e-deuterium and including argon, which was a secondary point of the NPL exercise. Non-uniqueness corrections are reported in Appendix I (data from Ref.3).

In the following chapter, the analysis of the temperature values for <u>all</u> the cells will be made on the basis of the three sets of data, collected in Tables VI.2.x, VI.3.x and VII.2.x. From each of these Tables a mean temperature value can be obtained for the whole set of cells and a standard deviation calculated. Table VII.3 allows a compact comparison of these data, which can be compared also with the dispersion of the differences between cells obtained from Figs. VII.2.x: the latter figure represents the width of the dispersion histogram at 2/3 of the total area. The last column of the Table allows one to compare the former experimental uncertainties of the intercomparison with the limit represented by the Laboratory-weighted total uncertainty, that is, the mean square of the uncertainties defined in Sect.IV.3.1, weighted according to the number of measurements done by each Laboratory.

Finally, a collection of data on IPTS-68 National realizations will be done in the second chapter. In this case, only <u>one</u> cell value for each substance is needed: the reference cell has been used, since it generally represents well the average value (Figs. VII.2.x).

2.1 On the temperature values of the cells.

Let us consider the substances separately.

#### ARGON

Systematic differences of more than 1 mK are visible between LAB-IPTS-68 values. Some are certainly related to Scale realizations, as the well-known case of NBS, others could be due only to the thermometer actually used (NRLM, NIM). It must be noticed that temperature values for BIPM, IMGC and INM are relative to a Laboratory reference cell, which is supposed to reproduce T = 83.798 K exactly (actually, these Laboratories use the argon triple point for IPTS-68 definition). The mean temperature value and standard deviation reported in Table VII.4 are calculated excluding NBS values.

Concerning NPL-IPTS-68 values with the Laboratory thermometers, Table VI.3.a shows a rather good agreement ( $\pm$  0.27 mK) among these old (circa 1975) calibrations. Only the newer calibration of n°7681 (NRLM) shows quite an anomalous value, which has been excluded from the calculated values reported in Table VII.3. After the non-uniqueness corrections are applied (Table VII.2.a), the standard deviation ( $\pm$  0.27 mK) does not improve and remains higher than the cell-comparison uncertainty ( $\pm$  0.15 mK) and the total uncertainty ( $\pm$  0.21 mK) (Table VII.4).

#### OXYGEN

With oxygen, the systematic differences in LAB-IPTS-68 values of Table VI.2.b come only from cell differences, except for the NIM measurements. The values in Table VII.4 have been obtained excluding both NIM values and deviating cells (8 IMGC and 7801 NRLM). The same calculation has been performed in Table VI.3.b on NPL-IPTS-68 values, discarding only the values of the deviating cells.

The standard deviation is worse ( $\pm$  0.29 mK) than with argon (of course there are no uniqueness corrections here) and significantly worse than the cell-comparison dispersion ( $\pm$  0.23 mK) and total uncertainty ( $\pm$  0.21 mK).

#### e – HYDROGEN

Among the few values available on LAB-IPTS-68, it must be noticed that the IMGC ones are relative to the 1H2IMGC cell, chosen to represent the definition value T = 13.81 K. Concerning NPL-IPTS-68 values, there is evidence of the large calibration drift of thermometer n°PL01-6, which has been discarded from the calculations, together with the NRLM value.

Still, the standard deviation is worse  $(\pm 0.49 \text{ mK})$  than the cellcomparison dispersion  $(\pm 0.31 \text{ mK})$  and the total uncertainty  $(\pm 0.29 \text{ mK})$ (again no non-uniqueness corrections to apply).

#### METHANE

Large systematic deviations are evident between Laboratories in LAB-IPTS-68 (Table VI.2.d). They generally reflect the systematic differences at the argon triple point<sup>56,63</sup>, and the fact that the two definitions of IPTS-68, using argon triple-point or oxygen condensation-point, give rise to a systematic difference  $(T(Ar)-T(O_2))$  at the methane triple point, amounting in this Intercomparison to 0.7  $\pm$  0.7 mK (where NRLM values were again discarded).

In fact, values are better reproduced using NPL-IPTS-68, as shown in Table VI.3.d. After NRLM and PRMI values are discarded, standard deviation settles to a lower value ( $\pm$  0.48 mK), and to a still better value after non-uniqueness corrections are applied( $\pm$  0.35 mK) (Table VII.2.d). The last value is practically coincident with the results of cell comparison ( $\pm$  0.3 mK) and the total uncertainty level ( $\pm$  0.25 mK). However, cell 7 BIPM had also to be excluded, since it was found to be unstable (Table VII.1.d).

#### NITROGEN

Results with nitrogen suffer from being only a small number. This results in a random pattern for changes of standard deviation values in Table VII.3. In fact, the very good agreement between LAB-IPTS-68 values (Table VI.2.e) and the poor one for NPL-IPTS-68 values (Table VI.3.e) are quite contradictory; the latter improves only a little when applying the non-uniqueness corrections (Table VII.2.e). Consequently, the quality of temperature measurements ( $\pm$  0.35 mK) turns out to be much worse than the agreement between cells ( $\pm$  0.15 mK) and compared with the total uncertainty ( $\pm$  0.15 mK).

#### NEON

From Sect.VII.1 it is known that some systematic differences may exist between cells. This results, of course, in some spread also for the temperature values. However, a spread much larger than justified by that may be observed between LAB-IPTS-68 values in Table VI.2.f  $(\pm 0.86 \text{ mK})$ , though only four Laboratories are involved. It may be mainly attributed to differences between the National realizations, because the NPL-IPTS-68 values of Table VI.3.f, though extended to many more Laboratories, give a better agreement  $(\pm 0.41 \text{ mK}, \text{ excluding n}^{\circ}\text{PLOI-}6)$ , with no change after the non-uniqueness corrections are applied (± 0.41 mK); this value is still quite a bit higher than the dispersion of cell-comparison measurements (+ 0.20 mK) and the total uncertainty (+ 0.25 mK) .

#### e – D E U T E R I U M

As pointed out in Sect.VII.1.3, the quality of the two cells available was much worse than for the others, the gas being quite impure. No evaluation was possible in Sect.VII.1.1; here some considerations come from the measured temperature values.

The IMGC cell was measured in three Laboratories: the agreement between measurements was about 2 mK. This is much worse than the published reproducibility figure  $(\pm 0.3 \text{ mK})^{48,49}$ , but is compatible with the difficulties found at NPL and NRC in obtaining a good shape for the melting plateau and with the differences in calibration of the thermometers (e.g. see hydrogen, Table VI.3.c). By the way, it is much better than the systematic difference found with respect to the NRC cell (15 mK): a possible reason for that is given in Sect.VII.1.3.

	Table VII.2.a : Results of measurements on sealed cells filled with: A R G O N										
	ASMW	BIPM	IMGC	INM	NBS	NIM	<u>N M L</u>	NPL	NRC	NRLM	PRMI
<u>Cells</u> :					Temperatu (thermo	re in NPL-I meter: 1728	<u>PTS-68</u> (K) 839)				
4 ASMW	83.79758		83.79746								
3 BIPM											
1 IMGC	83.79772		83.79740					83.79690	83.79685		
2 IMGC			83.79739	83.79742	83.79719		83.79705				
1 INM				83.79737			83.79730		83.79702		
XXI INM				83.79739							
BCM4 INM				83.79753					83.79714		
M1 NBS				83.79746	83.79719				83.79684		
113 NIM											
10 NRC			83.79753	83.79732	83.79715		83.79737	83.79695	83.79684		
14 NRC									83.79684		
7801 NRLM					83.79717						
7803 NRLM	83.79762		83.79756	83.79748			83.79758	- 542	83.79684		
mc PRMI											

 $T_{mean}$  = 83.79723 K,  $\sigma$  = 0.27 mK.

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Tabl	e VII.2.d :	Results of	measuremen	nts on seal	ed cells fi	lled with:	метнан	I E		
ASMW	BIPM	IMGC	INM	NBS	NIM	<u>N M L</u>	NPL	NRC	NRLM	PRMI
	24 1021 1020104			Temperatu (thermo	re in NPL-I meter: 1728	PTS-68 (K) 839)				
								90.68462°		
90.68348		90.68385					90.68372	90.68372		
		90.68370						90.68383		
		90.68407		•				90.68429		
-	Tabl	Table VII.2.d :         A S M W       B I P M         90.68348	Table VII.2.d : Results of         A S M W       B I P M       I M G C         90.68348       90.68385         90.68348       90.68370         90.68407	Table VII.2.d : Results of measurement         A S M W       B I P M       I M G C       I N M         90.68348       90.68385       90.68370       90.68407	Table VII.2.d : Results of measurements on seal         A S M W       B I P M       I M G C       I N M       N B S         A S M W       B I P M       I M G C       I N M       N B S         90.68348       90.68385       90.68370       90.68407	Table VII.2.d : Results of measurements on sealed cells fi         A S M W       B I P M       I M G C       I N M       N B S       N I M         A S M W       B I P M       I M G C       I N M       N B S       N I M         A S M W       B I P M       I M G C       I N M       N B S       N I M         A S M W       B I P M       I M G C       I N M       N B S       N I M         A S M W       B I P M       I M G C       I N M       N B S       N I M         A S M W       B I P M       I M G C       I N M       N B S       N I M         M W       B I P M       I M G C       I N M       N B S       N I M         M W       B I P M       I M G C       I N M M       N B S       N I M         M W       B I P M       I M G C       I N M M       N B S       N I M         M W       B I P M       I M G C       I M M M       N B S       N I M         M W       B I P M       I M G C       I M M G C       I M M M       N B S       N B S         90.68348       90.68370       90.68407       .       .       N B S       .       N B S       .       N B S       .       N B S	Table VII.2.d : Results of measurements on sealed cells filled with:         A S M W       B I P M       I M G C       I N M       N B S       N I M       N M L         A S M W       B I P M       I M G C       I N M       N B S       N I M       N M L         B I P M       I M G C       I N M       N B S       N I M       N M L         G I N M       B I P M       I M G C       I N M       N B S       N I M       N M L         B I P M       I M G C       I N M G C       I N M       N B S       N I M       N M L         B I P M       I M G C       I N M G C       I N M M       N B S       N I M       N M L         B I P M       I M G C       I M G C       I N M       N B S       N I M       N M L         G I M G C       I M G C       I M G C       I N M G C       I M G C       I M G C       I M G C         90.68348       90.68385       90.68370       90.68407       I M G C       I M G C       I M G C	Table VII.2.d : Results of measurements on sealed cells filled with: METHAN         ASMW       BIPM       IMGC       INM       NBS       NIM       NML       NPL         ASMW       BIPM       IMGC       INM       NBS       NIM       NML       NPL         METHAN       MEC       Inmethan       NBS       NIM       NML       NPL         90.68348       90.68385       90.68370       90.68407       90.68407       90.68407	Table VII.2.d : Results of measurements on sealed cells filled with: METHANE         ASMW       BIPM       IMGC       INM       NBS       NIM       NML       NPL       NRC         ASMW       BIPM       IMGC       INM       NBS       NIM       NML       NPL       NRC         Image: Asymptotic colstance       Image: Asymptotic colstance       Image: Asymptotic colstance       90.68462°         90.68348       90.68385       90.68372       90.68372       90.68372         90.68407       90.68429       90.68429	Table VII.2.d : Results of measurements on sealed cells filled with: METHANE         ASMW       BIPM       IMGC       INM       NBS       NIM       NML       NPL       NRC       NRLM         ASMW       BIPM       IMGC       INM       NBS       NIM       NML       NPL       NRC       NRLM         ASMW       BIPM       IMGC       INM       NBS       NIM       NML       NPL       NRC       NRLM         Temperature in NPL-IPTS-68 (K) (thermometer: 1728839)       90.68462°         90.68348       90.68385       90.68372       90.68372       90.68372         90.68370       90.68407       90.68429       90.68429

Excluding °):  $T_{mean} = 90.68392 \text{ K}$ ,  $\sigma = 0.35 \text{ mK}$ .

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	Table VII.2.e : <u>Results of measurements on sealed cells filled with</u> : N I T R O G E N											
	ASMW	BIPM	IMGC	INM	NBS	<u>NIM</u>	<u>N M L</u>	NPL	<u>N R C</u>	NRLM	PRMI	
Cells:					Temperat (therm	ure in NPL- ometer: 172	<u>IPTS-68</u> (K) 8839)					
2 IMGC			63.14629	63.14686				63.14611	63.14599			
BCM4 INM				63.14669					63.14593			
33 NRC			63.14624						63.14590			

£.

 $T_{mean}$  = 63.14625 K ,  $\sigma$  = 0.35 mK.

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	Table VII.2.f : Results of measurements on sealed cells filled with: N E O N												
	ASMW	BIPM	IMGC	INM	<u>N B S</u>	<u>NIM</u>	NML	<u>N P L</u>	<u>N R C</u> (*)	NRLM	PRMI		
<u>Cells</u> :					Temperat (therm	ure in NPL- ometer: 172	<u>IPTS 68</u> (K) 8839)						
1 ASMW 1 IMGC	24.56298°		24.56292° 24.56185 24.56284°										
3 IMGC BCM4 INM	24.56213		24.56155 24.56284° 24.56155	24.56208 24.56207			24.56191	24.56163	24.56105 24.56095				
12 NRC 1 NRLM	24.56211		24.56258° 24.56135				24.56166	24.56141 24.56165	24.56101 24.56070				
2 NRLM mc PRMI			24.56286° 24.56155					24.56172	24.56122				
* through 1	1521389. σ <sub>to</sub>	= 0.55 m	K. Exclu	ding °): T	= 24.5 nean	6156 K , σ	= 0.41 mK.						

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Table VII.3: Uncertainties in the International Intercomparison. (mK)

	(1)	(2)	(3)	(4)	(5)
	<u>On t</u>	emperature va	alues	On cell Inter-	Laboratory weighted
	LAB-IPTS-68	NPL-IPTS-68 lab.therm.	NPL-IPTS-68 1728839	comparison	total uncertainty
Substance	σ	σ	σ		
Argon	83.79793 K	83.79707 K	83.79727 K		
	<u>+</u> 0.59	<u>+</u> 0.27	<u>+</u> 0.27	<u>+</u> 0.15	<u>+</u> 0.21
Oxygen	54.36113 K	54.36109 K	54.36109 K		
	<u>+</u> 0.24	<u>+</u> 0.29	<u>+</u> 0.29	<u>+</u> 0.23	<u>+</u> 0.21
e-Hydrogen	13.80977 K	13.81025 K	13.81025 K		
	<u>+</u> 0.85	<u>+</u> 0.49	<u>+</u> 0.49	<u>+</u> 0.31	<u>+</u> 0.29
Methane	90.68485 K	90.68372 K	90.68392 K		
	<u>+</u> 0.79	<u>+</u> 0.48	<u>+</u> 0.35	∿ <u>+</u> 0.3	<u>+</u> 0.25
	90.68517 K + 0.79	(Ar)*			
	90.68449 K <u>+</u> 0.68	(0 <sub>2</sub> )*			
Nitrogen	63.14625 K	63.14606 K	63.14625 K		
	. <u>+</u> 0.20	<u>+</u> 0.45	<u>+</u> 0.35	∿ <u>+</u> 0.15	<u>+</u> 0.15
Neon	24.56248 K	24.56176 K	24.56156 K		
	<u>+</u> 0.86	<u>+</u> 0.41	<u>+</u> 0.41	<u>+</u> 0.20	<u>+</u> 0.25

\* for laboratories using the two different IPTS-68 definitions.

(1) from Tables VI.2.x
(2) from Tables VI.3.x
(3) from Tables VII.2.x
(4) from Figs. VII.2.x
(5) from Sect. IV.3.1 and Tables VII.1.x

#### 2.2 On differences between Scale realizations

The possibility of making this analysis suffers from some restrictions, due to limitations in the availability of data.

Tables VII.4.x summarize the relevant data. On the left side of each Table, there is a summary of the data available from the literature and information is given about the thermometer(s) used (when available). On the right side, the reference cell of the intercomparison is compared with the Laboratory realization. The NPL-IPTS-68 value (column 1) is reported only as a memo. The LAB-IPTS-68 value measured with the Laboratory thermometer (column 2) is corrected for non-uniqueness, leading to the value for thermometer n°1728839 of column 3. That is made using again the corrections given in Appendix I: the error due to differences of this correction using NPL instead of LAB calibration are supposed to be immaterial. The last column (4) gives, for secondary points, the temperature value for the Laboratory realization, again calculated on LAB-IPTS-68(1728839); unfortunately, some of these data are lacking.

Therefore, the difference between the last two values in a row gives the difference between the same device (the reference cell) and the Laboratory realization of the fixed point and, consequently, the differences between Laboratory <u>fixed-point realizations</u> may be obtained. From column (3), on the other hand, the differences between <u>Scale realizations</u> can be obtained. The two sets of differences are coincident at the definition points of IPTS-68. The absolute temperature values are relative to the reference cell used here and consequently would change slightly if other cells were used in Tables VII.4.x, according to the dispersion shown in Tables VI.2.X. Finally, one has to notice that all the values in columns (1),(2) and (3) are affected by the uncertainty specified in Sect.VI.3.1, and the values in column (4) by the error indicated in the literature.

The two sets of differences are collected, for convenience, in Table VII.6 (differences between LAB-IPTS-68 and NPL-IPTS-68) and Table VII.5 (differences between Laboratory and NPL realizations). With methane and nitrogen, IMGC-IPTS-68 has been used instead as a reference, since NPL realizations of these fixed points are not available.

The analysis of these differences will not be made in this Report.

# Table VII.4.a: Summary on Scale realizations

. triple point of. A R G O N

				Publis realiza	hed tions		Reference cellLaboratory(results of therealizatioIntercomparison)				
La	abo	ora	atory	open	cell	<u>Ref</u> .	(1) NPL lab.th	(2) (IPTS LAB nermom.	(3) 5-68) LAB 1728839	(4) LAB 1728839	
A	s	м	W		°83.7976	26	83.7975	83.7977	83.7977	83.7979	
B	I	P	М	definitio	n point	6,14	83.7968	83.7982	ş	83.7980*	
I	М	G	С	-	defin.	7,8	83.7972	refer.	refer.	83.7980*	
I	N	м		-	defin.	9	83.7973	83.7981	83.7982	83.7980*	
N	в	S		<sup>β</sup> 83.7996 <sup>+</sup>	<sup>Y</sup> 83.8001	27	83.7970	83.8003	83.8005		
N	I	M			83.7996	18		83.7996	§	§	
N	M	L		<sup>δ</sup> 83.7974	-	28	83.7970	83.7967	83.7968	83.7976	
N	P	L		<sup>ε</sup> 83.7971	-	29	83.7969	83.7969	83.7969	83.7971	
N	R	С		<sup>n</sup> 83.7973	-	30	83.7967	83.7978	83.7981	83.7975	
N	R	L	м	-			83.7983	83.7988	§	§	
P	R	М	I	<sup>ĸ</sup> 83.7975		32,33	83.7960				
<u>0t</u>	he	er	Natio	nal Laborat	ories						
K	0	L		-	-						
P	т	в		<sup>λ</sup> 83.7972	×	42				83.7977	
Tł	Thermometers used and Notes: *) definition value. \$) not available. +) low-purity sample.										
C	x ,	1°	20727	8 (NPL-IPTS	-68); <sup>β,γ</sup>	n°17	74095 (N	BS-IPTS-	68); <sup>δ</sup> τ	°1654278	
(Ì к	(NML-IPTS-68); <sup>c</sup> n°1728839 (NPL-IPTS-68); <sup>n</sup> n°1521389 (NRC-IPTS-68); <sup>c</sup> average of n°874, 876 and F (PRMI-IPTS-68); <sup>λ</sup> n° 188682 (PTB-IPTS-68;										

thermom. n°188682 belongs to the "international group": T<sub>NPL</sub>= 83.7976 K).

# Table VII.4.b: <u>Summary on Scale realizations</u>

# triple point of: O X Y G E N

				Publis realiza	shed ations		Reference cellLaboratory(results of therealizationIntercomparison)realization				
La	abo	ora	atory	open	cell	<u>Ref</u> .	(1) NPL lab.th	(2) (IPTS LAB hermom.	(3) 5-68) LAB 1728839	(4) LAB 1728839	
A	s	м	W	defin.			54.3609	54.3609	54.3609	54.3610*	
B	I	P	М	-	-						
I	M	G	C		defin.	8,52	54.3613	refer.	refer.	54.3610*	
I	N	M		-	defin.		54.3615	54.3612	54.3612	54.3610*	
N	B	S		defin.		-	<sup>+</sup> 54.3609	54.3614	54.3614	54.3610*	
N	I	M			<sup>α</sup> 54.3636	62	54.3612	54.3636	ş	54.3610*	
N	M	L		defin.	-	34	54.3608	54.3609	54.3609	54.3610*	
N	P	L		defin.	-	35	54.3607	54.3607	54.3607	54.3610*	
N	R	С		defin.		36	54.3608	54.3611	54.3611	54.3610*	
N	R	L	м	defin.			54.3617	54.3616	ş	54.3610*	
P	R	М	I	defin.			54.3606			54.3610*	
0	the	er	Nation	al Laborat	tories						
K	0	L		defin.	-	46,58					
P	Т	B		defin.	-						
Τ α	he: n	rm( °7	ometers 709 (NI	used and M-IPTS-68)	Notes: +	*) def: H) usi:	inition ng NPL co	value. § ompariso	) not av n data.	ailable.	

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#### Table VII.4.c: Summary on Scale realizations

triple point of: e - H Y D R O G E N

		Reference cellLaborato(results of therealizatIntercomparison)					
Laboratory	open	cell	<u>Ref</u> .	(1) NPL lab.th	(2) (IPTS LAB mermom.	(3) 5-68) LAB 1728839	(4) LAB 1728839
ASMW	-	-		13.8106			
B I P M I M G C	-	αdefin.	8,49	13.8105	13.8097	13.8097	13.8100*
INM NBS	defin.	-					
N I M N M L	defin.	-	37	13.8098	13.8095	13.809	5 13.8100*
N P L N R C	defin. defin.	-	38 39	13.8101 13.8110	13.8101 13.8106	13.810 13.810	13.8100* 5 13.8100*
N R L M P R M I	defin.		40	13.8134 13.8103	13.8115		13.8100* 13.8100*
Other Nationa	l Laborat	cories					
KOL							
РТВ							
Thormometers	used and	Notoci					

Thermometers used and Notes: \*) definition value.

 $^{\alpha}$  n° 1722205, PL01-6 and PL02-6; using the NBS, NPL and PRMI-IPTS-68 calibrations, values for T(H<sub>2</sub>) of 13.8082 K, 13.8108 K and 13.8167 K respectively have been obtained.

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Table VII.4.d: Summary on Scale realizations

#### triple point of: METHANE

Published realizations							Reference cellLaboratory(results of the Intercomparison)realization				
							Ref.	(1)	(2) (IPTS	(3) 5-68)	(4)
Laboratory			ator	<u>y</u>	open	cell		NPL lab.th	LAB nermom.	LAB 1728839	LAB 1728839
	s	м	W		-	-		90,6835	90,6835	90, 6836	_
B	I	P	м		°90.6858	<sup>α</sup> 90.6854	6,14	90.6847	90.6857	ş	ş
I	M	G	С		-	<sup>β</sup> 90.6856	8,56	90.6839	90.6855	90.6856	90.6856
I	N	M			-	-					
N	B	S			-	-					
N	I	M			-	-			90.6837		-
N	M	L			-	-					
N	P	L			-	-		90.6821	90.6821	90.6821	-
N	R	С			-			90.6841	90.6849	90.6850	
N	R	L	М		-	<sup>Y</sup> 90.685	41	90.6880	90.6881	\$	§
P	R	М	I		-	-		90.6797			-
01	the	er	Nat	tio	nal Labora	tories			a.		
K	0	L			-	-					
P	T	B			<sup>δ</sup> 90.6846	-	42				90.6851

Thermometers used and Notes: §) not available.

<sup>α</sup> n°226321 (BIPM-IPTS-68); <sup>β</sup>average of n°646, 838, 1754792, 1722205, 45, 1761201, PL01-6, PL02-6 (IMGC-IPTS-68); <sup>γ</sup> not specified; <sup>δ</sup> n° 188682 (PTB-IPTS-68(0<sub>2</sub>); using Ar the the Scale definition: T = 90.6854 K; thermom. n°188682 belongs to the "international group":  $T_{NPL} = 90.6851$  K).

Table VII.4.e: Summary on Scale realizations

triple point of: NITROGEN

	Reference cellLaboratory(results of therealizationIntercomparison)						
			Ref.	(1)	(2) (IPTS	(3) 5-68)	(4)
Laboratory	open	cell		NPL lab.th	LAB nermom.	LAB 1728839	LAB 1728839
ASMW							
BIPM	<u>×</u>						
IMGC	-	<sup>α</sup> 63.1458	8,60	63.1463	63.1463	63.1463	63.1463
INM	-			63.1467	63.1460	63.1461	63.1459
NBS							
NIM							
NML	<sup>β</sup> 63.1459	-	28				
NPL	-	-		63.1461	<b>62.</b> 1461	63.1461	L –
NRC	<sup>Y</sup> 63.1464	-	47	63.1456	63.1464	63.1460	63.1467
NRLM							
PRMI							
Other National Laboratories							
KOL	<sup>δ</sup> 63.148	-	61				
РТВ	-	-					

Thermometers used and Notes:

<sup> $\alpha$ </sup> average of n°1722205, 45 (IMGC-IPTS-68); <sup> $\beta$ </sup> n°459 (NML-IPTS-68); <sup> $\gamma$ </sup> n°1521389 (NRC-IPTS-68); <sup> $\delta$ </sup> not specified.

#### Table VII.4.f: Summary on Scale realizations

### triple point of: N E O N

				Publi: realiz	shed ations	hed tions		erence of sults of ercompar	the ison)	Laboratory realization
						Ref.	(1)	(2) (IPTS	(3) 5-68)	(4)
Laboratory		atory	open	cell		NPL lab.th	NPL LAB lab.thermom.		LAB 1728839	
A	s	м	W	-	°24.5627	55	24.5621	-	-	-
в	I	P	м							
I	M	G	С	-	<sup>β</sup> 24.562	8,56	24.5625	-	-	<b>-</b> t:
I	N	M		-		57	24.5616 24.5619	-	-	-
N	B	S		Υ <sub>24.5611</sub>	-	43				
N	I	M								
N	M	L		$\delta^{24.5631}$	-	28	24.5619	24.5625	24.5625	5 24.5623
N	P	L		<sup>η</sup> 24.5619	-	29	24.5616	24.5616	24.5616	5 24.5619
N	R	С		<sup>0</sup> 24.5620	-	44	24.5617	24.5640	24.5628	3 24.5618
N	R	L	М	-	<sup>µ</sup> 24.5630		24.5622	24.5615	§	§
P	R	M	I	<sup>ĸ</sup> 24.5627		32,33	24.5619			
Ot	he	er	Natio	nal Labora	tories					
ĸ	0	L		<sup>λ</sup> 24.5618	-	46,58	ġ.			
P	Т	B		3						
	-									

Thermometers used and Notes: §) not available.

<sup>α</sup> n° 207278 (NPL-IPTS-68); <sup>β</sup> average of n°1722205, 1761201, PL01-6, PL02-6, 45 (platinum calibrated on NBS, NPL, PRMI-IPTS-68) and n°2551, 2863 (germanium calibrated on XAC'); <sup>γ</sup> n°1692598 (NBS-IPTS-68 from NBS-55; 0.9 mK lower on NPL-IPTS-68); <sup>δ</sup> n°459 (NML-IPTS-68); <sup>ε</sup> n°1731676 (NML-IPTS-68); <sup>n</sup> n°1728839 (NPL-IPTS-68); <sup>θ</sup> n° 1521389 (NRC-IPTS-68); <sup>K</sup> average of n°874, 876, F (PRMI-IPTS-68); <sup>λ</sup> average of n°164956(B2) and T4 calibrated on KOL-IPTS-68; <sup>μ</sup> not specified.

	<u>Publi</u> realiz	shed ations		(re Int	eference sults of ercompan	cell 1 the 1 rison)	Laboratory realization
			Ref.	(1)	(2) (IPTS	(3) 5-68)	(4)
Laboratory	open	cell		NPL lab.th	LAB nermom.	LAB 1728839	LAB 1728839
	trip	le point (	of: e	- D E U	TERI	UM	
ASMW							
ΒΙΡΜ		_					
IMGC		a18.678	8,49	18.6778	-	-	-
INM							
NBS							
NIM							
NML	<sup>β</sup> 18.6909	÷	50				
NPL				18.6753	18.6753	18.6753	-
NRC	-	<sup>Y</sup> 18.662	51	18.6786	18.6773	18.6778	18.6620
NRLM							
PRMI							
	trip	le point	<u>of</u> : n	- D E U	TERI	UM	
IMGC	-	<sup>8</sup> 18.729	48,49				
NML	<sup>β</sup> 18.709	-	50				
NRC	-	Υ <sub>18.712</sub>	51				

## Table VII.4.g: Summary on Scale realizations

Thermometers used and Notes:

<sup>α</sup> average of n°1722205, PL01-6, PL02-6 calibrated on NBS, NPL, PRMI-IPTS-68. <sup>β</sup> n° 373 (NML-IPTS-68); <sup>γ</sup> n°1872179 (NRC-IPTS-68); <sup>δ</sup> same as <sup>α</sup> plus n°1761201 and n°45.

		( (		,		
Laboratory	Ar	°2	e-H <sub>2</sub>	сн <sub>4</sub>	<sup>N</sup> 2	Ne
ASMW	+0.02	-0.2				
вірм°						
IMGC°	(+0.0) *	-0.3	+0.4	( <u>+0.0</u> )*	( <u>+0.0</u> )*	
INM°	-0.2	-0.5			+0.2	
NBS		-0.7				
NIM						
NML	+0.8	-0.2	+0.6			-0.5
NPL	+0.2	( <u>+0.3</u> )*	( <u>-0.1</u> )*	×		(+0.3)*
NRC	-0.5	-0.4	-0.5		-0.7 -	-1.3
NRLM						
PRMI			1			
* (ref.Lab.)	- (ref.ce	11); °)	Argon is	used for	IPTS-68	definition.
Table	VII.6: Dif	ferences	between R	EF-IPTS-6	8 and LAE	B-IPTS-68.
	(ref	erence the	ermometer	: 1728839	; mK)	÷
Laboratory	Ar	0 <sub>2</sub>	e-H2	сн <sub>4</sub>	N <sub>2</sub>	Ne
ASMW	-0.8	-0.2		+2.0		
BIPM°						
IMGC°	-1.1	-0.3	+0.4	REF.	REF.	
I N M°	-1.3	$\frac{-0.2}{-0.5}$ \$			-0.2	
NBS	-3.6	-0.7				
NIM		<u>+0.5</u> §				
NML	+0.1	-0.2	+0.6			-0.9
NPL	REF.	$\frac{+0.1}{\text{REF.}}$ §	$\frac{-0.3}{\text{REF.}}$ §	-3.5	+0.2	REF.
NRC	-1.1	-0.4	-0.5	+0.6	+0.3	-1.2
NRLM	.±	0.2+0.7 9	<u>+2.3</u> §			
PRMI						
	- <u>+</u>	<u>0.1-0.3</u> §	<u>+1.1</u> §			
§ NPL-IPTS-6	8 - LAB-IP	TS-68 dif	ferences	measured	at NPL in	n 1975 (Ref.3)

Table VII.5: Differences between national fixed-point realizations. (NPL(or IMGC) - LAB, mK)

#### VIII. CONCLUSIONS

Most of the goals of the International Intercomparison have been fulfilled during the five years of circulation of cells. Only the comparison of the National Scale realizations at the fixed-point temperatures suffered from some lack of results. This was generally due to a fact that may be considered also as a result of this exercise: when thermometer calibrations were involved in the calculations, i.e. when one was dealing with temperature values, data generally showed a larger dispersion of values.

This is equivalent to stating that this intercomparison of fixed points in sealed devices allowed one to check for uniformity of realization of these fixed points among laboratories, with an accuracy better than was possible with thermometers; or, at least, that the reliability in assessing the accuracy level of fixed-point intercomparison was higher.

As a matter of fact, the agreement found between the values obtained when comparing the participating cells was much better than it was possible to conclude from literature data, based on LAB-IPTS-68. On the other hand, the level of inaccuracy was found to be between  $\pm$  0.15 and  $\pm$  0.3 mK, higher than the one ( $\pm$  0.1 mK) which was anticipated. This may be due either to the actual state of the art in thermal experiments in the laboratories, or to the present limit of measurability of the sealed-cell devices.

Apart from the few cases of inaccuracy ascertained for some cells, values of differences between cells up to about 0.5 mK were occasionally found. This limit is higher than most of the melting ranges observed for the samples enclosed in the cells: in addition, quite large differences from laboratory to laboratory have been observed in the shape of the melting plateau. These facts seem to indicate that there is a real lack of control of the thermal process at a ± 0.1 mK level. A better knowledge and control of the behaviour of the sample inside the cell seems to be necessary to reach a reproducibility level of the order of few tens of microkelvins, needed to limit the total inaccuracy to within ± 0.1 mK. Some of the results of this exercise seem to indicate that it is not an impossible goal, since for most of the substances the melting range could be limited well within 0.5 mK. On the other hand, there was also evidence of some increase in the spread of values for lower temperatures, especially in the range where platinum resistance thermometers exhibit decreasing sensitivity: this could be due to increasing practical difficulties, more in the measurement process with the experimental apparatus than in the control of the thermal process in the cell.

However, the present agreement between the realizations of the fixed points that is shown in the figures and tables may be already considered adequate for the uniqueness requirements of an improved future IPTS. It has been proved that it can be obtained using the technique of small transportable sealed cells, whose design turned out to be largely non-critical.

The temperature of the thermodynamic state reproduced by each of the cells will be accurately conserved and reproduced for many years to come (no tendency to change has been observed so far), allowing immediate availability for realization to any new Scale definition, provided only that it uses triple points exclusively, and that a suitable value is assigned to each temperature reproduced by them.

# ACRONYMS OF THE LABORATORIES

A	S	ΜW	Ξ	Amt für Standardisierung Messwesen und Warenprüfung, G.D.R.
B	I	ΡM	=	Bureau International des Poids et Mesures, Sèvres.
I	М	GC	=	Istituto di Metrologia "G.Colonnetti", Italy.
I	N	M	=	Institut National de Métrologie, France.
K	0	L	=	Kamerling Onnes Laboratorium, The Netherlands.
N	B	S	=	National Bureau of Standards, U.S.A.
N	I	М	æ	National Institute of Metrology, China.
N	M	L	=	National Measurements Laboratory, Australia.
N	P	L	=	National Physical Laboratory, U.K.
N	R	C	=	National Research Council, Canada.
N	R	LM		National Research Laboratory of Metrology, Japan.
P	Т	В	=	Physikalisch-Technische Bundesanstalt, F.R.G.
P	R	MI	=	Physicotechnical and Radiotechnical Measurement Institute, U.S.S.R.

#### IX. REFERENCES

- M.P.Orlova, D.I. Sharevskaja, D.N.Astrov, I.G.Krutikova, C.R.Barber and J.C.Hayes, Metrologia <u>2</u>, 6 (1966).
- 2. C.R.Barber and J.C.Hayes, Metrologia 2, 11 (1966).
- 3. S.D.Ward and J.C.Compton, Metrologia 15, 31 (1979).
- 4. L.M.Besley and W.R.G.Kemp, Metrologia 13, 35 (1977).
- Comptes Rendus of the 12th Session and Report to CIPM, Comité Consultatif de Thermométrie (BIPM, 1978).
- 6. J.Bonhoure and R.Pello, Metrologia 16, 95 (1980).
- 7. F.Pavese, G.Cagna and D.Ferri, Proceeding ICEC 6, IPC Science and Technology Press, Guildford 1976, 205.
- 8. F.Pavese and D.Ferri, TMCSI 5, 217 (1983).
- 9. G.Bonnier and R.Malassis, Bulletin BNM n.22, 19 (1975).
- 10. G.Bonnier and Y.Hermier, TMCSI 5, 231 (1983).
- 11. G.T.Furukawa, Comité Consultatif de Termométrie, Doc. CCT/80-34 (1980); TMSCI 5, 239 (1983).
- K.Mitsui and A.Inaba, Comité Consultatif de Thermométrie, Doc. CCT/80-28 (1980).
- 13. Private Communication to the Editor.
- 14. J.Bonhoure and R.Pello, Metrologia 14, 175 (1978).
- 15. G.Bonnier and A.Moser, Measurement (IMEKO) 1, 143 (1983).
- 16. G.T.McConville, private communication to the Editor.
- 17. G.T.Furukawa and M.L.Reilly, J.Research NBS 74A, 617 (1970).
- Li Zhiran and Li Zhongyue, Acta Phys.Temp.Humilis Sinica <u>5</u>, 67 (1983).
- 19. J.A.Cowan, R.C.Kemp and W.R.G.Kemp, Metrologia 12, 87 (1976).
- 20. J.Ancsin, letter to the Editor.
- 21. O.Tamura and H.Sakurai, Jap.J.Appl.Physics 22, 356 (1983).
- V.M.Khnykov, M.P.Orlova and D.N.Shakevskaya, Izmeritelnaja Tekhnika: Supplement Metrologia 12, 3 (1973).
- 23. R.L.Rusby, letter to the Editor.
- F.Pavese, Proc. Symposium on Temperature Measurement, Dom Techniki CSSR, Karlovy Vary 1981, 35.
- 25. J.Ancsin, letter to the Editor.
- 26. H.Maas, letter to the Editor.
- 27. G.T.Furukawa, W.R.Bigge and J.R.Riddle, TMSCI 4, 231 (1972).
- 28. R.C.Kemp and W.R.G.Kemp, Metrologia 14, 83 (1978).
- 29. S.D.Ward, Comité Consultatif de Thermométrie, Doc. CCT/80-51 (1980)
- 30. J.Ancsin, Metrologia 9, 147 (1973).
- T.Shiratori, M.M.Mehdi and K.Mitsui, Comité Consultatif de Thermométrie, Doc. CCT/78-23 (1978) T103.
- 32. V.M.Khnykov, L.I.Rabukh, L.B.Beliansky, T.S.Pantin, M.P.Orlova and D.N.Astrov, Comité Consultatif de Thermométrie, Doc. CCT/76-39 (1976).
- 33. D.N.Astrov, letter to the Editor.
- 34. R.C.Kemp, W.R.G.Kemp and J.A.Cowan, Metrologia 12, 93 (1976).
- 35. J.P.Compton and S.D.Ward, Metrologia 12, 101 (1976).
- 36. J.Ancsin, Metrologia 9, 26 (1973).
- 37. R.C.Kemp and W.R.G.Kemp, Metrologia 15, 155 (1979).
- 38. J.P.Compton, TMSCI 4, 195 (1972).
- 39. J.Ancsin, Metrologia 13, 79 (1977).
- M.P.Orlova, L.B.Belyansky, Ya.E.Razhba, R.V.Philonchik and V.M.Khnykov, Comité Consultatif de Thermométrie, Doc. CCT/71-56 (1971).
- 41. A.Inaba and K.Mitsui, Jpn.J.Appl.Physics 18, 1193 (1979).
- 42. W.Blanke and W.Thomas, Measurement (IMEKO) 1, 105 (1983).
- G.T.Furukawa, W.G.Saba, D.M.Sweger and H.H.Plumb, Metrologia <u>6</u>, 35 (1970).
- 44. J.Ancsin, Metrologia 14, 1 (1978).
- 45. H.Sakurai, communication to the Editor.
- 46. J.P.Tiggelman, Ph.D. Thesis, Kamerling Onnes Laboratorium, Leiden 1973.

- 47. J.Ancsin, Can.J.Phys. <u>52</u>, 1521 (1974).
- 48. F.Pavese and C.Barbero, Cryogenics 19, 255 (1979).
- 49. F.Pavese, Physica 107B, 333 (1981) (Proceedings LT 16).
- 50. R.C.Kemp, TMSCI 5, 249 (1983).
- 51. J.Ancsin, Comité Consultatif de Thermométrie, Doc. CCT/82-(1982).
- 52. F.Pavese, Metrologia 14, 93 (1978).
- 53. Li Zhiran and Li Zhongyue, Comité Consultatif de Thermométrie, Doc. CCT/82-33.
- 54. P.Seifert, Comité Consultatif de Thermométrie, Doc. CCT/80-46
- P.Seifert, Comité Consultatif de Thermométrie, Doc. CCT/82-6 (1982).
- 56. F.Pavese, Metrologia 15, 47 (1979).
- 57. F.Pavese and C.Barbero, Proc. XV Int. Congress Refrig., IIR, 1979 vol.1, 155.
- 58. J.L.Tiggelman, C.Van Rijn and M.Durieux, TMSCI 4, 137 (1972).
- 59. R.C.Kemp, letter to the Editor.
- 60. F. Pavese, Comité Consultatif de Thermométrie, Doc. CCT/80-24.
- 61. J.L.Tiggelman and M.Durieux, TMSCI 4, 149 (1972).
- 62. Wu Biqiu, Wang Zilin and Huan Ninsheng, Comité Consultatif de Thermométrie, Doc. CCT/82-35.
- 63. R.E.Bedford, G.Bonnier, H.Maas and F.Pavese (Working Group 2 of the Comité Consultatif de Thermométrie), Doc. CCT/84-40.
- R.E.Bedford, M.Durieux, R.Muijlwijk and C.R.Barber, Metrologia <u>5</u>, 47 (1969).
- F.Pavese, G.Bonnier and J.Bonhoure: Comité Consultatif de Thermométrie, Doc. CCT/76-32 (1976).
- 66. J.Ancsin: Metrologia 14, 79 (1978).
- 67. J.Ancsin and G.Bonnier: Comité Consultatif de Thermométrie, Doc. CCT/76-40 (1976).
- 68. G.T.Furukawa, to be published on Metrologia.

## APPENDIX I

## Non-uniqueness <u>corrections</u> for thermometers of the "international" group (after Compton & Ward, Ref.3)<sup>23</sup>

Thermometer	Argon 83.798 K	Methane 90.6855 K	Nitrogen 63.1462 K	Neon 24.5622 K
217997	+ 0.27	- 0.02	+ 0.49	- 0.23
207278	+ 0.33	- 0.03	+ 0.58	- 0.14
PL01-6	+ 0.17	- 0.01	+ 0.47	- 0.16
PL02-6	+ 0.24	- 0.01	+ 0.51	- 0.15
45	- 0.15	+ 0.01	+ 0.03	- 0.08
1812283	+ 0.10	- 0.01	+ 0.15	+ 0.13
1812282	+ 0.19	- 0.02	+ 0.19	+ 0.19
1731676	+ 0.07	- 0.01	+ 0.06	+ 0.01
1158062	+ 0.19	- 0.02	+ 0.35	- 0.20

## T<sub>1728839</sub> - T<sub>x</sub> (mK)

APPENDIX II: Calibration data of thermometers.

Thermometer:	ASMW	ASMW	ASMW	BIPM	BIPM	BIPM	BIPM	IMGC	IMGC
	207278	217997	217990	226322	226322	226321	226321	PL01-6	PL01-6
(calibration:)	(NPL)	(NPL)	(NPL)	(NPL)	(BIPM)	(NPL)	(BIPM)	(NPL)	(IMGC)
Fixed point (K)				(ohm)	)				
373.15	35.298875	35.398895	33.682996	34.694699	34.694776	35.329705	35.329733	35.193550	35.193626
273.15	25.346730	25.418300	24.186290	24.913330	24.913392	25.369090	25.369110	25.271030	25.271140
90.188	·6.178722	6.195642	6.139226	6.074291		6.184647		6.160324	
83.798					5.383077		5.480737		5.459260
54.361	2.329294	2.335410	2.314124	2.290773				2.322823	2.322865
27.102	0.307643	0.308040	0.305102	(1976)				0.307860	
20.28	0.111906	0.111587	0.110502					0.112862	
17.042	0.062608	0.062036	0.061468			3		0.063771	
13.81	0.034302	0.033546	0.033312					0.035595	0.035630
90.6855	6.233440	6.250517	6.189085	6.128075	6.127892	6.239421	6.239227	6.214877	6.214807
83.798	5.475589	5.490516	5.488947	5.383224		5.480956		5.459321	
63.1462	3.231584	3.240213	3.272673	3.177652				3.222258	3.222314
24.5622	0.218808	0.218904	0.219989		1 <u>7</u>			0.219350	

Thermometer:	IMGC	IMGC	INM	INM	INM	NBS	NBS	NBS	NIM
	45	45	1812283	1812283	232788	1774095	1812282	1812282	7703
(calibration:)	(NPL)	(IMGC)	(NPL)	(INM)	(INM)	(NBS)	(NPL)	(NBS)	(NIM)
Fixed point (K)			Ŷ	(ohm)	)				
		÷							
373.15	35.760497	35.760401	35.504765	35.505337			35.526435	35.526518	
273.15	25.679390	25.679381	25.494500	25.494711	24.087300	25.560840	25.510280		24.899378
90.188	6.262076		6.213374			6.231297	6.218080	6.217717	
83.798		5.549490		5.505919	5.480579				
54.361	2.362255	2.362190	2.340650	2.340685		2.349529	2.342869	2.342825	
27.102	0.313479		0.305813			0.310304	0.306376	0.306335	
20.28	0.114979		0.108619			0.112833	0.108937	0.108971	
17.042	0.064974	-	0.058992			0.063049	0.059228	0.059246	
13.81	0.036262		0.030629			0.034498	0.030805	0.030835	
90.6855	6.317517		6.268424	6.268291			6.273159	6.272805	
83.798	5.549724		5.505983				5.510296	5.509901	
63.1462	3.276340		3.248407	3.248509			3.251288	3.251053	
24.5622	0.223397		0.216327				0.216794	0.216734	

APPENDIX	II:	Calibration	data	of	thermometers.
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Thermometer:	NIM	NIM	NPL	NML	NML	NRC	NRC	NRC	NRC
	188640	7709	1728839	1731676	1731676	1521389	1158062	1158062	1872179
(calibration:)	(NIM)	(NIM)	(NPL)	(NPL)	(NML)	(NRC)	(NPL)	(NRC)	(NRC)
Fixed point (K)				(ohm)	)				
373.15	2		35.593541	35.542413	35.542413	35.545198	35.467637	35.467457	35.628083
273.15	24.164330	25.352963	25.559570	25.522800	25.522800	25.523332	25.469660	25.469547	25.582651
90.188			6.232672	6.223657	6.223646	6.220654	6.213066	6.212952	6.234894
83.798									
54.361			2.350583	2.347195	2.347185	2.344245	2.345837	2.345804	2.349500
27.102			0.310722	0.310276	0.310263	0.308205	0.314670	0.314567	0.308485
20.28			0.112961	0.112801	0.112814	0.111212	0.117890	0.117888	0.110721
17.042			0.063120	0.063020	0.063024	0.061615	0.068250	0.068243	0.060859
13.81			0.034521	0.034459	0.034460	0.033232	0.039643	0.039623	0.032318
		1							
90.6855			6.287854	6.278764	6.278752	6.275768	6.268040	6.267923	6.290120
83.798			5.523596	5.515606	5.515595	5.512551	5.506668	5.506546	5.525160
63.1462			3.260563	3.255856	3.255844	3.252812	3.252284	3.252204	3.260186
24.5622			0.220997	0.220691	0.220679	0.218775	0.225380	0.225308	0.218749

## APPENDIX II: Calibration data of thermometers.

Thermometer:	<u>NRLM</u> 7681	<u>NRLM</u> 7681	<u>NRLM</u> 1781356	<u>PRMI</u> 1842381	PRM1 1842381
(calibration:)	(NPL)	(NRLM)	(NRLM)	(NPL <sup>+</sup> )	(PRMI)
Fixed point (K)				(ohu	m)
373, 15				35, 576452	2*
273.15		25.362925	25.52581	8 25.544950	0
90.188					
83.798				5.515116	6
54.361				2.343744	4
27.102				0.305632	2
20.28				0.108293	3
17.042				0.058672	2
13.81				0.030353	3
90.6855				6.279004	4
83.798				5.515116	6
63.1462				3.253298	8
24.5622				0.216070	0

\* assumed (see text). + triple point of argon used instead of condensation point of oxygen.