THERMOMETRY BETWEEN 0.5 K AND 30 K

MEASUREMENTS RELATED TO THE "1976 PROVISIONAL 0.5 K TO 30 K TEMPERATURE SCALE"

A. E. EL SAMAHY

THERMOMETRY BETWEEN 0.5 K AND 30 K

MEASUREMENTS RELATED TO THE "1976 PROVISIONAL 0.5 K TO 30 K TEMPERATURE SCALE"

THERMOMETRY BETWEEN 0.5 K AND 30 K

MEASUREMENTS RELATED TO THE "1976 PROVISIONAL 0.5 K TO 30 K TEMPERATURE SCALE"

PROEFSCHRIFT

TER VERKRIJGING VAN DE GRAAD VAN DOCTOR IN DE
WISKUNDE EN NATUURWETENSCHAPPEN
AAN DE RIJKSUNIVERSITEIT TE LEIDEN,
OP GEZAG VAN DE RECTOR MAGNIFICUS DR. A. A. H. KASSENAAR,
HOOGLERAAR IN DE FACULTEIT DER GENEESKUNDE,
VOLGENS BESLUIT VAN HET
COLLEGE VAN DEKANEN TE VERDEDIGEN OP
DINSDAG 11 DECEMBER 1979 TE KLOKKE 16.15 UUR

DOOR

AHMED EZ-ELARAB ABDEL-AZIZ EL SAMAHY
geboren te Koum Hamada, Egypte

NKB OFFSET B.V. BLEISWIJK

Promotor : Prof. dr. R. de Bruyn Ouboter

Co-promotor: Dr. M. Durieux.

to Mervat and our
daughter Nahla.

CONTENTS

CHAPTER 1. INTRODUCTION

1.1. International practical temperature scales 11

1.2. Derivation of the EPT-76 12

1.3. Summary of contents 14

CHAPTER 2. MEASUREMENTS WITH GERMANIUM AND RHODIUM-IRON RESISTANCE THERMOMETERS BETWEEN 0.5 K AND 30 K

2.1. Introduction

2.1.1. *Germanium and rhodium-iron resistance thermometers* 18

2.1.2. *Resistance thermometers used in the thermometry group* 19

2.1.3. *Summary of contents* 20

2.2. Resistance versus temperature characteristics of the thermometers

2.2.1. *Germanium resistance thermometers* 21

2.2.2. *Rhodium-iron resistance thermometers* 24

2.3. Experimental arrangement

2.3.1. *The resistance measurements* 26

2.3.2. *Cryostats* 28

2.4. Experimental data on germanium thermometers

2.4.1. *Self-heating* 35

2.4.2. *Calibration and reproducibility* 37

2.5. Experimental data on rhodium-iron thermometers

2.5.1. *Self-heating* 41

2.5.2. *Calibration and reproducibility* 43

2.6. Comparison of temperature scales maintained with calibrated germanium and rhodium-iron thermometers 45

CHAPTER 3. EXPERIMENTAL DATA ON THE USE OF THE SUPERCONDUCTIVE TRANSITION POINTS OF Pb, In, Al, Zn AND Cd AS THERMOMETRIC REFERENCE POINTS

3.1. Introduction 50

3.2. Experimental arrangement

3.2.1. *The SRM 767 device* 51
3.2.2. *The resistance thermometers* 53
3.2.3. *The cryostat* 53
3.3. Measurements procedure 54
3.4. Results 55
3.5. Relation between transition temperatures and widths of transitions 60
3.6. Measurements in 1973 (Pb, In) and 1974 (Pb, In, Al) 62
3.7. Comparison with the NPL data for three SRM 767 devices 63
3.8. Results of NML 66
3.9. Conclusions 67

CHAPTER 4. MEASUREMENTS OF VAPOUR PRESSURES OF LIQUID ^{4}He AND ^{3}He BELOW 2 K

4.1. Introduction

4.1.1. *Vapour pressures of liquid ^{4}He and ^{3}He and thermometry* 72
4.1.2. *Vapour pressures of liquid ^{4}He* 72
4.1.3. *Vapour pressures of liquid ^{3}He* 74
4.1.4. *Summary of contents* 75

4.2. Vapour pressure measurements of liquid ^{4}He and ^{3}He below 2 K with the first cryostat

4.2.1. *Experimental arrangement* 76
4.2.2. *Procedure of measurement* 78
4.2.3. *Experimental results for ^{4}He* 81
4.2.4. *Discussion of ^{4}He vapour pressures* 82
4.2.5. *Experimental results for ^{3}He* 83

4.3. Vapour pressure measurements of liquid ^{3}He between 0.5 K and 1.8 K with the second cryostat

4.3.1. *The cryostat* 84

4.3.2. *Vapour pressure measurements* 85

4.3.3. *Experimental results and discussion* 89

4.3.4. *Thermodynamic calculations of the vapour pressure equation* 91

4.4. Conclusion 92

APPENDIX 95

SAMENVATTING 104

STUDIEOVERZICHT 106

NAWOORD 107

CHAPTER 1.

INTRODUCTION.

1.1. INTERNATIONAL PRACTICAL TEMPERATURE SCALES.

As long as there is no simple direct way for measuring thermodynamic temperatures it will be necessary to define international temperature scales for use in science and industry. Such scales define practical temperatures which are close to the thermodynamic temperature and relatively easy to measure.

During the last decade a considerable amount of work has been done in several laboratories for establishing a new practical temperature scale between 0.5 K and 30 K. The new scale was in principle adopted by the International Committee of Weights and Measures in 1976 under the name "1976 Provisional 0.5 K to 30 K Temperature Scale" or "EPT-76"1). The final text of the scale was published under the auspices of the Advisory Committee for Thermometry2) in 19783,4). In Fig. 1.1 differences are shown between the EPT-76 and older international practical scales (the 1958 ^{4}He Scale 5), the 1962 ^{3}He Scale6) and the International Practical Temperature Scale of 19687)).

This thesis deals with experimental work which was done during the last four years in the thermometry group of the Kamerlingh Onnes Laboratory for the derivation of the new temperature scale and for its realization in the laboratory. For easy reference the complete English text of the scale is given in the Appendix at the end of the thesis.

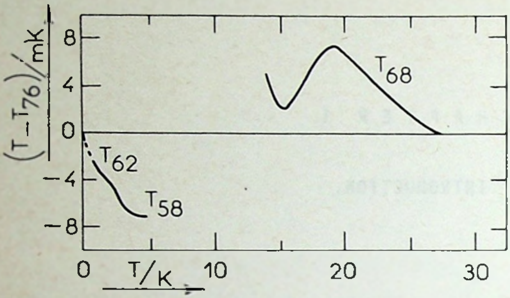

Fig. 1.1.

Differences between the EPT-76 (T_{76}) and older international practical temperature scales between 0.5 K and 30 K (T_{58}, T_{62} and T_{68}).

1.2. DERIVATION OF THE EPT-76.

A detailed account of the derivation of the EPT-76 is given in ref.8. Only a short description of the background of the scale will be given here.

Between about 1964 and 1974 a number of low temperature scales was established in various laboratories. Among these were the "NBS Provisional Temperature Scale 2-20 K of 1965 (NBS 2-20 scale)" based on an acoustic thermometer by Plumb and Cataland[9)] and the magnetic scales of Cetas and Swenson at Iowa State University[10)], Van Rijn at the Kamerlingh Onnes Laboratory[11)], Cetas at the National Measurement Laboratory in Sydney and Astrov at the Institute for Physico-Technical and Radiotechnical Measurements in Moscow (see ref. 8 and Appendix). In order to avoid a proliferation of scales, plans were made[12)] for a generally accepted low temperature scale and, as a first step, an international comparison of scales was organized: each laboratory which had established a scale sent calibrated germanium thermometers to the National Measurement Laboratory in Sydney where they were intercompared[13)].

One of the results obtained from the comparisons was that the magnetic scales, when reduced to the same reference points, agreed within 1 mK[8,13]. Because of this consistency between magnetic scales, one of them, the $T_{XAc'}$-scale, was chosen as the basis for the new international scale. The $T_{XAc'}$-scale was chosen because it was defined in a wider temperature range than the other magnetic scales and, more important, because it was at its lower end based on cerium magnesium nitrate for which the deviation from Curie's law is small and well-known. It was, however, decided later that the new scale should join smoothly with the IPTS-68 at 27.1 K (the boiling point of neon); therefore a slight correction, $\Delta T = - 2.5 \times 10^{-6}\ T^2/K$, was added to $T_{XAc'}$ (see Fig. 1.2).

After these scale comparisons the results became available of two experiments with primary thermometers: Isotherm and gas thermometer measurements of Berry[14] at the National Physical La-

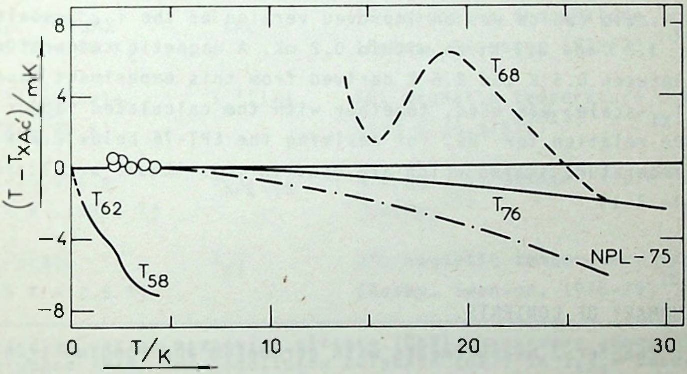

Fig. 1.2. Relation of the EPT-76 (T_{76}) to the magnetic $T_{XAc'}$-scale, the NPL-75 gas thermometer scale and noise thermometer data of Klein et al. (o). Also the older international scales (T_{58}, T_{62} and T_{68}) are indicated.

boratory in Teddington resulted in the NPL-75 scale between 2.6 K and 27.1 K. Klein, Klempt and Storm[15)] at the University of Münster determined with a modified classical noise thermometer temperature differences $T_{noise} - T_{58}$ at six temperatures between 2.1 K and 4.2 K. At temperatures between 2.6 K and 4.2 K the noise thermometer data agreed with the NPL-75 scale within 0.4 mK (see Fig. 1.2). The difference between the NPL-75 scale and T_{76}, as defined by the relation $T_{76} = T_{XAc'} - 2.5 \times 10^{-6}\ T^2/K$ appeared to be a very smooth function of the temperature (see Fig. 1.2). (If the calibration constants for the magnetic $T_{XAc'}$-scale would be changed slightly the magnetic scale would agree with the NPL-75 scale within the precision of the measurements, see ref. 8).

A new magnetic thermometer experiment, with cerium magnesium nitrate as the paramagnetic salt, was made by Rusby and Swenson[16)] at the National Physical Laboratory. The magnetic data could be fitted to the NPL-75 scale between 2.6 K and 3.1 K, and to the T_{XISU75}-scale (which was an improved version of the $T_{XAc'}$-scale) between 1.3 K and 3.1 K, to within 0.2 mK. A magnetic temperature scale between 0.5 K and 2.6 K derived from this experiment (and named T_{X1}-scale) was used, together with the calculated vapour pressure relation for ^{4}He, for deriving the EPT-76 below 2.6 K[8)]. (The temperature scales which are used in this thesis are listed in Table 1.1).

1.3. SUMMARY OF CONTENTS.

In Chapter 2 measurements with germanium and rhodium-iron resistance thermometers are described. These thermometers are the main carriers of the various laboratory scales mentioned in the preceeding section: The NBS 2-20 scale and the five magnetic scales between 1 K and 30 K were all maintained with calibrated germanium thermometers. The NPL-75 scale and the T_{X1}-scale are maintained with rhodium-iron thermometers. At the end of Chapter 2

Table 1.1. List of temperature scales used in this thesis.

Scale and range	Temperature	Description
1958 ^{4}He scale (1 K - 5.2 K)	T_{58}	^{4}He vapour pressure vs temperature table5)
1962 ^{3}He Scale (0.5 K - 3.3 K)	T_{62}	^{3}He vapour pressure vs temperature equation6)
IPTS-68 (above 13.81 K)	T_{68}	International Practical Temperature Scale of 1968; reference temperatures and platinum resistance thermometer7)
EPT-76 (0.5 K - 30 K)	T_{76}	Echelle Provisoire de Température de 1976 entre 0,5 K et 30 K
$T_{XAc'}$-scale (0.9 K - 30 K)	$T_{XAc'}$	ISU magnetic temperature scale* (Cetas, Swenson, 1972-73)10)
T_m(III)-scale (2 K - 30 K)	T_m(III)	KOL magnetic temperature scale** (Van Rijn, 1971)11)
NPL-75 scale (2.6 K - 27.1 K)	T_{NPL-75}	NPL gas thermometer scale (Berry)14)
T_{X1}-scale (0.5 K - 2.6 K)	T_{X1}	NPL magnetic temperature scale*** (Rusby, Swenson, 1976-79)16)

*Based on cerium magnesium nitrate (CMN), manganese ammonium sulphate (MAS) and gadolinium sulphate (GS). In $T_{XAc'}$ subscript X denotes that it is a magnetic scale (X is mutual inductance bridge reading), Ac that the scale was fitted to acoustic thermometer data9), and the dash that it was slightly adjusted thereafter (ISU: Iowa State University).

**Based on MAS and GS.

***Based on CMN. Two magnetic temperatures were defined T_{X1} and T_{X2} (see section 4.4) (NPL: National Physical Laboratory, Teddington, England).

the agreement is shown between realizations of the EPT-76 by means of the KOL magnetic scale, the ISU magnetic scale and the NPL gas thermometer scale together with the scale differences given in Table 5 of the EPT-76 (see Appendix).

The superconductive transition points of Pb, In, Al, Zn and Cd are reference points of the EPT-76 (Appendix, Table 1). Chapter 3 deals with the precise measurement of these transition points and the determination of their temperatures on well-defined temperature scales.

In Chapter 4 measurements of ^{4}He and ^{3}He vapour pressures below 2 K, in which the T_{X1}-scale was used as the temperature scale, are described and the relation between the thermodynamic vapour pressure equations and the T_{X1}-scale is discussed. The results are of importance for deriving vapour pressure vs temperature relations for ^{4}He and ^{3}He on the EPT-76.

Finally, it is gratifying that work described in this thesis could contribute to maintaining existing, or establishing new facilities for calibrations of thermometers. In nearly all of the experiments described, there were, in addition to the standard thermometers, a number of germanium thermometers in the cryostat which were calibrated for groups in and outside the laboratory.

REFERENCES.

1) Procès-Verbaux du Comité International des Poids et Mesures 44(1976)11.

2) Comité Consultatif de Thermométrie, 12^{e} Session (1978), to be published.

3) Echelle provisoire de température de 1976 entre 0,5 K et 30 K, Bureau International des Poids et Mesures (Sèvres, France, 1978).

4) The 1976 Provisional 0.5 K to 30 K Temperature Scale, Metrologia 15(1979)65. (The French text of the scale appeared also in J. de Phys. 39(1978) p. C-1631).

5) F.G. Brickwedde, H. van Dijk, M. Durieux, J.R. Clement and J.K. Logan, J. Res. Natl. Bur. Stand. (Wash.) 64A(1960)1.

6) R.H. Sherman, S.G. Sydoriak and T.R. Roberts, J. Res. Natl. Bur. Stand. (Wash.) 68A(1964)579.

7) The International Practical Temperature Scale of 1968, Amended Edition of 1975, Metrologia 12(1976)7.

8) M. Durieux, D.N. Astrov, W.R.G. Kemp and C.A. Swenson, Metrologia 15(1979)57.

9) H.H. Plumb and G. Cataland, Metrologia 2(1966)127.

10) C.A. Swenson, Metrologia 9(1973)99.

11) C. van Rijn and M. Durieux, Temperature, its measurement and control in science and industry 4(Instrum. Soc. of America, Pittsburgh, 1972) Part 1, p. 73.

12) Procès-Verbaux du Comité Consultatif de Thermométrie, 9^{e} Session (1971) T21.

13) L.M. Besley and W.R.G. Kemp, Metrologia 13(1977)15.

14) K.H. Berry, Metrologia 15(1979)89.

15) H.-H. Klein, G. Klempt and L. Storm, Metrologia 15(1979)143.

16) R.L. Rusby and C.A. Swenson, Metrologia, to be published.

CHAPTER 2.

MEASUREMENTS WITH GERMANIUM AND RHODIUM-IRON RESISTANCE THERMOMETERS BETWEEN 0.5 K AND 30 K.

2.1. INTRODUCTION.

2.1.1. *Germanium and rhodium-iron resistance thermometers.*

After Kunzler et al. demonstrated in 1961[1)] that certain doped germanium crystals mounted strainfree in a copper capsule could be used as sensitive stable thermometers, germanium thermometers soon became available from several manufacturers and were widely used in standards thermometry below 30 K and in practical temperature measurements below 100 K.

However, as experience with many germanium thermometers was obtained, it appeared that small changes sometimes occur in their calibrations when the thermometers are cycled between room temperature and low temperatures[2,3,4)]. These changes, of the order of 0.5 mK at 4.2 K to 3 mK at 30 K make the thermometers unsuitable for use in standards thermometry and other temperature measurements of the highest accuracy (e.g. in comparisons of temperature scales or of realizations of thermometric reference points in which a 0.1 mK accuracy is nowadays desirable, see Chapters 3 and 4).

The use of a rhodium-iron alloy as the sensitive element in a resistance thermometer was first suggested by Coles[5)]. The thermometer was developed by Rusby[6,7,8)] at the National Physical Laboratory (NPL) in Teddington and is now commercially available from H. Tinsley & Co. Ltd, London. A RhFe-wire is encapsulated in a thin-walled cylindrical platinum tube, 5 mm in diameter and 50 mm in length, filled with ^{4}He gas of a pressure of about 30 kPa at room temperature, the leads being brought out through glass seals. RhFe-thermometers have been used by at least three thermometry groups[9,4,10)] and their calibrations have been found to be stable, between 0.5 K and 30 K, within the measurement precision of 0.1 mK. This makes that RhFe-thermometers are rapidly replacing germanium thermometers in standards thermometry. For many practical temperature measurements, however, the larger size, lower resistance and lower sensitivity of RhFe-thermometers compared to germanium thermometers are real disadvantages.

2.1.2. *Resistance thermometers used in the thermometry group.*

In the thermometry group at KOL germanium thermometers were used by Van Rijn et al. in establishing a magnetic temperature scale between 2 K and 30 K[2,11)]. The magnetic scale was maintained in terms of calibrated germanium thermometers, in particular by the average of four germanium thermometers which had proved to be stable within ± 1 mK between 2 K and 30 K.

These four thermometers were included in 1974-1976 in the international comparison of low temperature scales at the National Measurement Laboratory in Sydney by Besley and Kemp[12)] (see Chapter 1) and are referred to as the four international germanium thermometers. Since this comparison these thermometers carried also calibrations in terms of the $T_{XAc'}$-scale, the magnetic temperature scale of Cetas and Swenson, which later became the basis of the 1976 Provisional 0.5 K to 30 K Temperature Scale (EPT-76)* .

Two RhFe-thermometers, calibrated by R.L. Rusby at the Na-

---

* See Table 1.1 for a list of temperature scales used in this thesis.

tional Physical Laboratory on the NPL-75 gas thermometer scale[9)] between 2.6 K and 27.1 K and on the NPL magnetic CMN scale (the T_{X1}-scale)[10)] between 0.5 K and 2.6 K, were obtained in 1976. Two other RhFe-thermometers of an older type, which had been obtained uncalibrated, were calibrated against the first two. The four international germanium thermometers were compared with the RhFe-thermometers in 1976-1977. Since that time the RhFe-thermometers are the primary carriers of the various temperature scales in our group.

The consistency of the scales maintained by the resistance thermometers was checked on various occasions by comparisons with platinum resistance thermometers calibrated at the reference points of the IPTS-68 and, at the lower temperatures, by measuring the superconductive transition temperatures of Pb, In, Al, Zn and Cd (Chapter 3) and helium vapour pressures (Chapter 4).

Since Van Rijn's measurements, about sixty germanium thermometers were calibrated; some of these were used in the thermometry group but most of them were supplied to other groups in the laboratory and at other institutes in The Netherlands. For about fifteen of the thermometers a history of their stabilities over time intervals up to twenty years is available.

2.1.3. *Summary of contents.*

In section 2.2 resistance versus temperature characteristics of germanium and rhodium-iron thermometers are given.

Section 2.3 deals with the experimental arrangement for thermometer comparisons: the resistance measuring equipment is discussed in section 2.3.1 with emphasis on the requirements that had to be met in the change-over from germanium to RhFe-thermometers; the cryogenic apparatus used for thermometer comparisons is decribed in section 2.3.2 (the apparatus described in this section was also used in the measurements presented in Chapters 3 and 4).

Experimental data on the self-heating and on the reproducibilities of germanium and RhFe-thermometers are given in sections 2.4 and 2.5.

In section 2.6 experimental results are presented on the comparison of the temperature scales carried by the germanium thermometers with the scale carried by the RhFe-thermometers.

2.2. RESISTANCE VERSUS TEMPERATURE CHARACTERISTICS OF THE THERMOMETERS.

2.2.1. *Germanium resistance thermometers.*

The thermometers were almost exclusively obtained from CryoCal Inc., St. Paul, U.S.A. In Fig. 2.1 resistances of CryoCal thermometers types CR 1000, CR 250, CR 100, CR 50 and CR 30 are shown between 0.5 K and 30 K (the type numbers give nominal resistances at 4.2 K). In Fig. 2.2 the sensitivity of a type CR 250 thermometer is shown.

The choice of the type of thermometer depends on the temperature range for which it has to be used. In general, it is advantageous to use a thermometer with a high resistance because, for a constant voltage over the thermometer, the energy dissipation in the thermometer is inversely proportional to its resistance. Moreover, a thermometer with a higher resistance has a higher sensitivity (see Fig. 2.1). Resistances should, however, not much exceed a value of about 10 kΩ in order to avoid loss of sensitivity of the electronic amplifier in the potentiometer circuit (see section 2.3) and difficulties with insulation resistances. Type CR 1000 is the most suitable for temperatures between about 100 K and 1.5 K. For measurements at temperatures down to 0.5 K type CR 250 is more suitable.

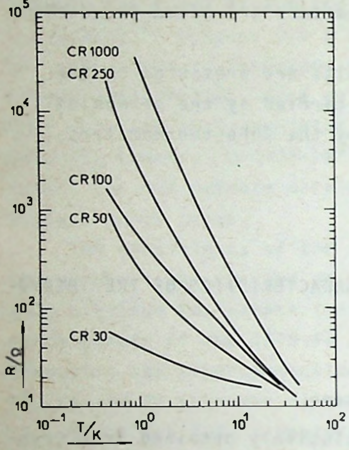

Fig. 2.1.
Resistances of germanium thermometers types Cryo-Cal CR 1000, CR 250, CR 100, CR 50 and CR 30 versus temperature.

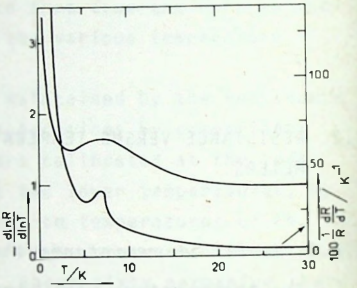

Fig. 2.2.
Sensitivities d ln R/d ln T and (1/R)dR/dT versus temperature for a germanium thermometer type CR 250.

In Table 2.1 characteristics are given of types CR 250 and CR 1000 germanium thermometers. The data for the self-heating are taken from section 2.4.1. It can be seen that the accuracy in the resistance measurements that is required for a 0.1 mK accuracy in the temperature measurements varies from 650 ppm at 0.5 K to 4 ppm at 30 K for a type CR 250 thermometer and from 600 ppm at 1 K to 8 ppm at 30 K for a type CR 1000 thermometer. In case measuring currents are used which produce a self-heating of 0.05 mK,

Table 2.1. Approximate values of resistances and sensitivities dR/dT *and* $(\frac{1}{R})\, dR/dT$ *for germanium thermometers.* P *is the power dissipated by a measuring current corresponding to a self-heating of 0.05 mK;* i *and* V *are the corresponding measuring current and voltage across the thermometer. The last column gives the corresponding voltage sensitivity of the thermometer.*

T (K)	R (Ω)	$-\frac{dR}{dT}$ (ΩK^{-1})	$-\frac{1}{R}\frac{dR}{dT}$ (K^{-1})	P (nW)	i (µA)	V (mV)	$\frac{dV}{dT}$ (µV/0.1 mK)
type CR 250							
0.5	18300	119000	6.5	1.3	0.27	4.9	3.2
1	2700	6200	2.3	3.0	1.1	3.0	0.7
2	680	560	0.85	6.3	3.1	2.1	0.18
5	160	53	0.32	14	9.4	1.5	0.05
10	54	8	0.15	26	22	1.2	0.018
20	23	1.2	0.052	45	45	1.0	0.005
30	15	0.6	0.035	56	60	0.9	0.003
type CR 1000							
1	30000	180000	6.0	3.0	0.33	10	6
2	5000	11000	2.1	6.3	1.1	5.5	1.2
5	760	560	0.74	14	4.3	3.3	0.26
10	160	56	0.35	26	13	2.1	0.07
20	44	5	0.12	45	32	1.4	0.017
30	25	2	0.08	56	47	1.2	0.010

the precision in the voltage measurements required for a 0.1 mK precision in the temperature measurements varies from 3μV at 0.5 K to 3 nV at 30 K for a type CR 250 thermometer and from 6 μV at 1 K to 10 nV at 30 K for a type CR 1000 thermometer. Thus, if type CR 250 thermometers are used at the lower temperatures and type CR 1000 at the higher temperatures, an accuracy in the resistance measurements between 650 ppm (at 0.5 K) and 8 ppm (at 30 K) and a precision in the voltage measurements between 3 μV and 10 nV are required for a 0.1 mK accuracy in the temperature measurements.

2.2.2. *Rhodium-iron resistance thermometers.*

In Fig. 2.3 the resistance and the sensitivity of a RhFe-thermometer are shown between 0.5 K and 30 K. The data are for a thermometer of the common type having an ice-point resistance of 100 Ω. Further characteristics of the thermometer are given in Table 2.2 (self-heating data are taken from section 2.5.1).

It follows from the table that for a 0.1 mK accuracy in the temperature measurements an accuracy of 14 ppm in the resistance

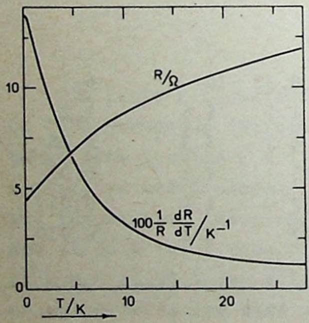

Fig. 2.3. Resistance R and sensitivity (1/R)dR/dT for a rhodium-iron thermometer ($R_{0^\circ C}$ = 100 Ω).

Table 2.2. Resistances and sensitivities dR/dT and $(\frac{1}{R})$ *dR/dT for a rhodium-iron thermometer (R(0° C) ≈ 100 Ω). P is the power dissipated by a measuring current corresponding to a self-heating of 0.05 mK; i and V are the corresponding measuring current and voltage across the thermometer. The last column gives the corresponding voltage sensitivity of the thermometer.*

T (K)	R (Ω)	$\frac{dR}{dT}$ (ΩK^{-1})	$\frac{1}{R}\frac{dR}{dT}$ (K^{-1})	P (μW)	i (mA)	V (mV)	$\frac{dV}{dT}$ (nV/0.1 mK)
0.5	4.6	0.65	0.14	0.031	0.08	0.37	5
1	4.9	0.60	0.12	0.5	0.3	1.5	18
2	5.5	0.57	0.10	0.12	0.15	0.83	8
5	7.0	0.43	0.061	0.33	0.22	1.5	9
10	8.7	0.28	0.032	0.52	0.24	2.1	7
20	10.8	0.16	0.015	0.75	0.26	2.8	4.2
30	12.2	0.13	0.011	0.89	0.27	3.3	3.6

measurements is required at 0.5 K and 1 ppm at 30 K. For measuring currents, which produce a self-heating of 0.05 mK, the precision in the voltage measurements corresponding to 0.1 mK in the temperature measurements is 5 nV at 0.5 K and 4 nV at 30 K. (For RhFe-thermometers with an ice-point resistance of 50 Ω (our thermometers RhFe-3 and RhFe-4) the corresponding figures are 3 nV at 0.5 K and 2 nV at 30 K).

2.3. EXPERIMENTAL ARRANGEMENT.

2.3.1. *The resistance measurements.*

Resistances were measured by using a dc current through a number of thermometers in series with a standard resistance and by comparing the voltages over the thermometers and the standard resistance with a classical potentiometer. Thermal EMF's were cancelled in the usual way by measuring in two directions of the thermometer and potentiometer currents. In general, three circuits were used, one for the RhFe-thermometers, one for the high resistance germanium thermometers and one for the low resistance germanium thermometers.

A four dial potentiometer was used developed by Dr. H. van Dijk and constructed by Bleeker Company in Zeist (The Netherlands); the first, second, third and fourth dial have 20, 100, 100 and ± 10 steps, respectively, so that a precision of 1 ppm could be obtained in the setting of the potentiometer (if the first dial was on step 10 or higher). However, in practice, only the first two dials of the potentiometer were used for compensating voltages to within about 0.1 %; the unbalance of the potentiometer was then measured with a Keithley 140 dc amplifier and a digital voltmeter. A low-pass filter with variable rise time was used at the input of the amplifier.

The Keithley amplifier has a maximum amplification factor of 10^5 and is linear to within 10 ppm of the full scale. At a rise time of about 5 seconds, the detection sensitivity of the system was about 3 nV if the external resistance in the input circuit did not exceed about 100 Ω. For higher external resistances, the detection sensitivity decreased to about 0.01 μV at 1 kΩ and 0.1 μV at 10 kΩ. (The external resistance is mainly determined by the resistance of the thermometer, the potentiometer output resistance being only 35 Ω).

Ordinary lead batteries were used as the thermometer and potentiometer current sources. An attempt to use an electronic power supply was not successful due to electrical interference with the Keithley amplifier. However, when using the batteries, care had to be taken that the currents were not interrupted during current reversals. (If currents were interrupted it took more than one minute before they reached stable values within 1 ppm).

For germanium thermometers, the detection sensitivities mentioned above were sufficient for obtaining a precision in the temperature measurements, using currents corresponding to a self-heating of 0.05 mK, which ranged from 0.01 mK at 0.5 K to 0.1 mK at 30 K (see data in Table 2.1). However, at high resistances of the germanium thermometers, stray rf-fields caused voltages of the order of 15 mV across the potential leads which affected the detection sensitivity of the amplifier and heated the thermometers and the copper block in which the thermometers were mounted. A 0.01 μF capacitor across the potential leads, where they enter the potentiometer, sufficiently suppressed rf pick-up.

For the RhFe-thermometers of the common type ($R(0^{o}\ C) \approx 100\ \Omega$) the detection sensitivity was sufficient for obtaining a 0.05 mK to 0.1 mK precision in the temperature measurements between 0.5 K and 30 K for currents which caused a self-heating of 0.05 mK (compare data in Table 2.2). (Even for the RhFe-thermometers of the older type, with $R(0^{o}\ C) \approx 50\ \Omega$, it was possible to obtain a 0.1 mK precision, corresponding to 2 nV, by averaging several measurements). For obtaining a 0.1 mK accuracy in the temperature measurements at 30 K with RhFe-thermometers, a 1 ppm accuracy in the resistance measurements is required. Resistance ratio's could be measured with this accuracy (provided that the resistances did not differ by more than a factor of two). The determining factor for the accuracy is the linearity of the potentiometer, which was checked by internal calibrations every two years; it appeared that voltage ratio's from the potentiometer did not change by more than 0.5 ppm of the full range per year.

The standard resistances (10 Ω in the case of RhFe thermometers; 10 Ω, 100 Ω, 1 kΩ or 10 kΩ in the case of germanium thermometers) were calibrated at intervals of about five years at the Van Swinden Laboratory of the Dutch Institute for Calibrations in Delft. This guarantees an absolute accuracy of 5 ppm. A high absolute accuracy in the resistance measurements was required when temperatures at reference points measured with RhFe-thermometers in different laboratories were compared. From the measurements described in this thesis the demands are most severe at the superconductive transition point of Pb (see Chapter 3) where a 6 ppm accuracy in the resistance measurement is required for a 0.1 mK accuracy in the temperature measurement.

2.3.2. *Cryostats.*

Two cryostats were used for the comparisons of resistance thermometers described in this chapter and for the measurements presented in Chapters 3 and 4.

a) The first apparatus was the same as used by Tiggelman$^{13)}$ for thermometer comparisons between 1.2 K and 300 K but a few modifications were made. It is sketched in Fig. 2.4. In the copper block R with a mass of about 2 kg, there were ten holes P to accommodate RhFe-thermometers or platinum thermometers and ten holes S for germanium thermometers. (The small copper cylinders U were sometimes used as binding posts for the germanium thermometer leads). The block was suspended by three thin-walled stainless steel capillaries O from the top I of the thermal shield K which surrounded the block. Both I and K were made of copper. The shield was suspended by three stainless steel capillaries H from the brass top of the stainless steel vacuum can J. The can was sealed with an indium O-ring G. The pumping line C had an inner diameter of 9 mm. When the apparatus was constructed, a provision was made for insertion of a vapour pressure thermometer into the block through the central openings in the top plates of the vacuum can and the shield.

The apparatus was originally designed for measurements above 1.2 K but it could be used for measurements at temperatures down to 0.5 K when the combination of the ^{3}He vapour pressure thermometer Q and the small ^{3}He cryostat L was inserted; this is described in detail in Chapter 4, dealing with the ^{3}He vapour pressure measurements (see Fig. 4.1).

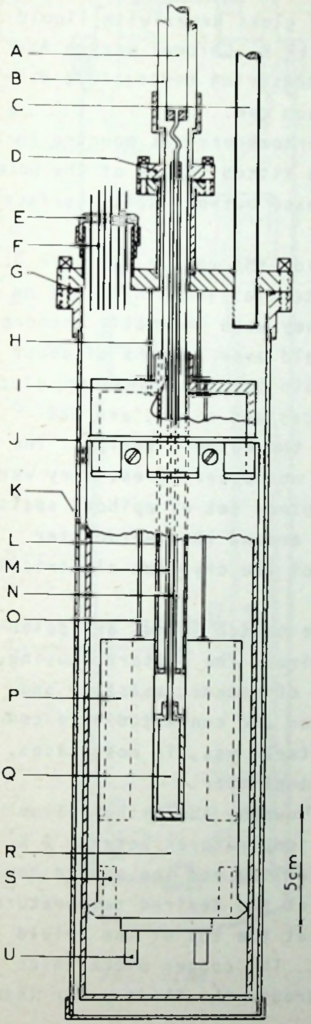

Fig. 2.4.

The first apparatus, used for comparisons of germanium and RhFe-resistance thermometers between 1.2 K and 30 K and for preliminary measurements on superconductive reference points (Chapter 3) and helium vapour pressures (Chapter 4). The insert for the ^{3}He cryostat and the vapour pressure thermometer is described in detail in Chapter 4.

The vacuum can was surrounded by a glass dewar with liquid helium which could be pumped to about 1.2 K. Chromel versus Au-0.03 % Fe thermocouples were used for measuring temperature differences between block, shield and vacuum can.

Each of the germanium and RhFe-thermometers was mounted in a cavity in a small copper cylinder which fitted in one of the holes in the copper block, with Apiezon N grease between metal surfaces for thermal contact.

The leads of the thermometers inside the vacuum can were 0.1 mm diameter manganin wires, for the potential leads, and 0.1 mm copper wires, for the current leads. They were thermally anchored to the block and to the top of the shield over lengths of about 100 mm using GE 7031 varnish, with a thin layer of paper for electrical insulation between copper surfaces and wires, and passed at F out of the vacuum can through the epibond seals E. The leads outside the vacuum can were 0.15 mm copper wires; they were brought out of the cryostat through another set of epibond seals. The vacuum can, aluminium foil wrapped around the thermometer leads in the dewar, and the brass top of the cryostat electrically shielded the circuit.

The leads from the cryostat to the switch boards and potentiometer were single shielded copper wires. The battery housing, potentiometer, and the various chassis of decade resistors and amplifier were all electrically shielded and connected to a central ground. The thermometer circuit itself was, in most cases, grounded at the input of the Keithley amplifier.

The copper block could be cooled down by admitting helium exchange gas into the vacuum can. For temperatures between 2 K and 30 K the can was evacuated after cooling and the shield temperature was electronically regulated at the desired temperature using a germanium thermometer mounted at the top of the shield and an auxiliary potentiometer circuit. The copper block which was, except for the heat conduction through the thermometer leads

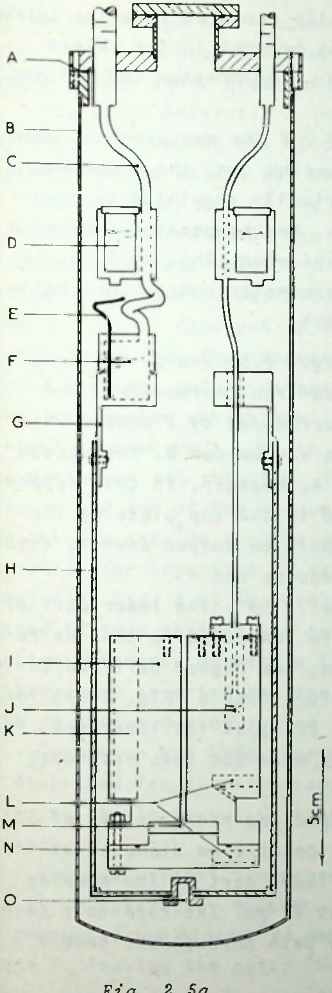

Fig. 2.5a.

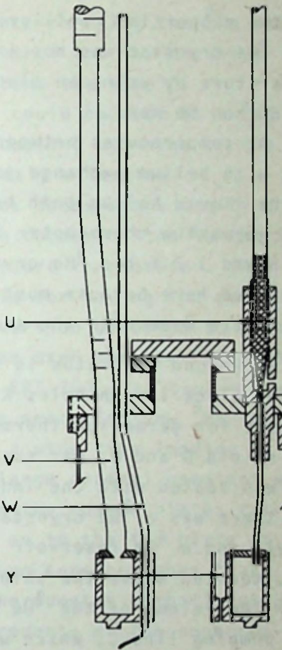

fig. 2.5b.

Fig. 2.5.

The second apparatus, used for comparisons of resistance thermometers between 0.5 K and 30 K and for the measurements in Chapters 3 and 4. (The pumping tube of the vacuum can is not indicated).

and the supporting capillaries, thermally isolated from the shield (the ^{3}He cryostat was not inserted) was brought to the shield temperature by using an electric heater. Thermometer comparisons could then be made.

At temperatures between 1.2 K and 2 K the measurements were made with helium exchange gas in the vacuum can, the temperature of the liquid helium bath being electrically regulated by means of a germanium thermometer in the bath. For temperatures between 0.5 K and 1.2 K the ^{3}He cryostat was inserted. This will not be discussed here because most of the thermometer comparisons below 1.2 K were made with the second cryostat.

b) The second apparatus is shown in Figs. 2.5a and b. Again, a copper block I with holes K for rhodium-iron thermometers and holes L for germanium thermometers, surrounded by a copper thermal shield G and H, was suspended in a vacuum can B. The vacuum can was sealed with the indium O-ring A. However, in this apparatus there was a ^{3}He cryostat F mounted in the top plate of the shield and a ^{4}He reservoir D, which could be pumped down to about 1 K, mounted above the shield in the vacuum can.

The volume of the ^{3}He cryostat was 7 cm^3. The lower part of the pumping line C, which was thermally connected to the ^{4}He reservoir, had an inner diameter of 4 mm; the higher part had an inner diameter of 9 mm. (There was a return line W (Fig. 2.5b) for the ^{3}He, with flow resistance E (Fig. 2.5a) at its lower end, for using the ^{3}He cryostat in a continuous mode but this was never done).

The ^{4}He reservoir D was ring-shaped and had a volume of 60 cm^3. The pumping line had a constriction of 2 mm diameter at its lower end; the inner diameter of the lower part of the pumping tube V was 7 mm and of the higher part 12 mm. The reservoir could be filled with liquid helium from the bath through the needle valve U.

Germanium thermometers were mounted at the top and bottom O of the shield and at Y in the copper plate carrying the ^{4}He reservoir. They were used for temperature control and for checking whether temperature gradients existed along the shield.

The small reservoir J in the block could be used as a ^{3}He cryostat for cooling the block in case cooling by means of the ^{3}He cryostat in the shield was not sufficiently effective.

At the lower end of the copper block there was a cavity M, closed with an indium O-ring seal by the copper bottom N, which served as the reservoir of a vapour pressure thermometer. (The bottom of the cavity was made demountable for having the possibility to put a catalyst in for the conversion of ortho to para hydrogen, in case the apparatus was to be used for realizing the liquid hydrogen reference points of the EPT-76). The vapour pressure thermometer was primarily meant for measuring low ^{3}He vapour pressures down to 0.5 K, in which case a wide tube leading to the manometer was required. Therefore, large central openings were made in the top of the vacuum can and in the copper plates carrying the ^{3}He and ^{4}He reservoirs, as well as in the top plate of the cryostat, for insertion of vapour pressure sensing tubes (see Chapter 4, Fig. 4.5). However, in the thermometer comparisons discussed in this chapter and in the measurements of the superconductive reference points in Chapter 3 a vapour pressure sensing tube was not inserted and the top of the vacuum can was closed with an indium O-ring sealed flange.

Germanium and RhFe-thermometers were mounted in the block, as described for the first apparatus. (The epibond seals for bringing the leads out of the vacuum can are not drawn in the figure).

The measurement procedure was similar to that described for the first apparatus. However, temperatures below 4.2 K were reached by pumping at the liquid ^{4}He and, below 2.5 K, the ^{3}He in the reservoirs D and F, leaving the outer ^{4}He bath at 4.2 K. In the beginning, measurements were made with the copper block isolated from the

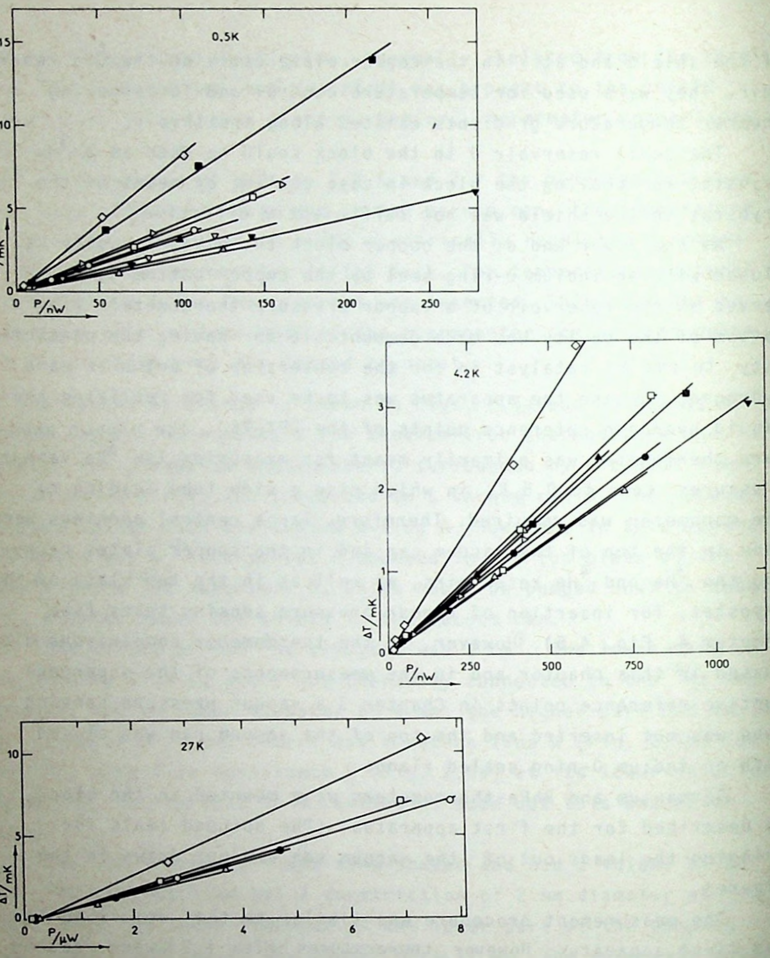

Fig. 2.6. Self-heating ΔT of germanium thermometers at 0.5 K, 4.2 K and 27 K versus dissipated power P in the thermometers.

○ △ ◇ ● type CR 250 thermometers
▼ ■ " CR 100 "
◆ " CR 50 "
▽ ▲ " CR 30 "

shield, as in the first apparatus. Later on, a weak thermal link was made between block and shield so that the block slowly followed the shield temperature. In some of the measurements presented in Chapter 4 the thermal contact between block and shield was increased even more, by using strips of copper foil, so that block and shield formed one isothermal unit. This facilitated the attainment of temperature equilibrium considerably, but put higher demands on the shield temperature regulation.

2.4. EXPERIMENTAL DATA ON GERMANIUM THERMOMETERS.

2.4.1. *Self-heating.*

Rather extensive measurements were made of the self-heating of germanium thermometers by the measuring current. Temperatures indicated by the germanium thermometers at different measuring currents were compared with temperatures measured with one of the RhFe-thermometers in the copper block. Measurements were made at 0.5 K, 2 K, 4.2 K, 13.8 K and 27 K. The experimental data for three temperatures are shown in Fig. 2.6. ΔT is the increase in temperature of the germanium thermometer with respect to the temperature at the normal measuring current. (Normal currents correspond to dissipated powers of about 2 nW, 5 nW and 200 nW at 0.5 K, 4.2 K and 27 K, respectively). Besides giving information on the self-heating of the thermometers, the data also showed that measurements at different currents, in general, gave consistent results within ± 0.1 mK which proved that the resistance measurements at the normal currents were accurate to within this limit. The self-heating coefficient, defined as the self-heating ΔT divided by the dissipated power P, vs temperature for the various thermometers is shown in Fig. 2.7. As could be expected,

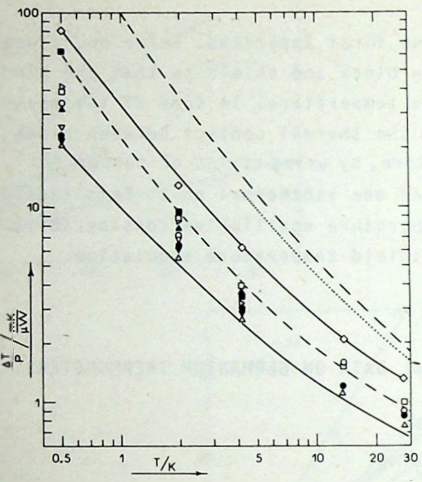

Fig. 2.7. Self-heating ΔT divided by the dissipated power P in the thermometers vs temperature between 0.5 K and 27 K for various germanium thermometers. Points are indicated as in Fig. 2.6. The full drawn curves indicate upper and lower limits. The dashed lines are upper and lower limits of comparable data on the self-heating of germanium thermometers by Besley and Kemp[12]*. The dotted curve is derived from data of Anderson and Swenson for their germanium thermometer 803*[4]*.*

there are no systematic differences between the various types of thermometers, CR 1000, CR 250, CR 100, CR 50 and CR 30, which are of the same construction. Results for individual thermometers differ by a factor of 2 to 4; this is probably due to differences in the thermal anchoring of the thermometers and their leads to the block. This may also be the reason for the differences between the present results and those of Besley and Kemp[12)] and Anderson and Swenson [4)] (see Fig. 2.7). In practice, one wants to keep the self-heating of the thermometers below about 0.05 mK; it follows from Fig. 2.7 that, according to the present measurements, for an average CryoCal germanium thermometer the dissipated power should then be kept below 1.5 nW at 0.5 K, 5 nW at 2 K, 15 nW at 4.2 K, 30 nW at 13.8 K and 50 nW at 27 K. It was discussed in section 2.2.1 that with these restrictions for the power dissipation in the thermometers temperatures can still be measured with a precision between 0.01 mK at 0.5 K and 0.1 mK at 27 K.

2.4.2. *Calibration and reproducibility.*

Calibrations of germanium thermometers are usually represented by polynomials[14)]

$$\ln R = \Sigma_{n=0}^{m} a_n (\ln T)^n \tag{2.1}$$

The order of the polynomial depends on the temperature range which has to be covered and on the required accuracy of the calibration. For example, in the range from 2 K to 30 K a twelfth degree polynomial fits calibration data within 0.5 mK, provided, of course, that the data are sufficiently accurate and the temperature scale which is used is sufficiently smooth[2)]. In the course of the present work, ten germanium thermometers types CR 30, CR 50, CR 100 and CR 250 were calibrated on the T_{X1}-scale (see Table 1.1) between 0.5 K and 2.6 K; fifth degree polynomials fitted the data within 0.2 mK (see Fig. 2.8).

The reproducibility of good germanium thermometers is illustrated in Fig. 2.9 for four thermometers (all CryoCal type CR 1000). The thermometers are the international germanium thermometers which were calibrated by Van Rijn in 1970 on his magnetic

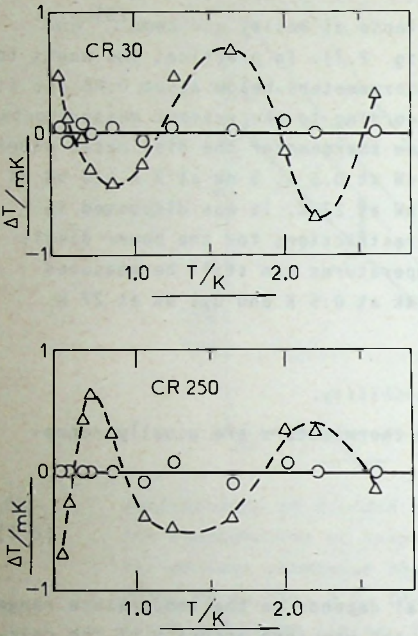

Fig. 2.8.

Polynomial fits of calibrations of germanium thermometers types CryoCal CR 30 and CR 250 between 0.5 K and 2.6 K. The triangles and the circles represent the residuals of the third and the fifth degree polynomials respectively.

temperature scale between 2 K and 30 K and the average of which has defined this temperature scale between that time and 1977 (see sections 2.1.2 and 2.6). The points in the figure were derived from comparisons of the resistances of the thermometers, temperatures being obtained from the 1970 calibration polynomials. (The systematic wiggles exhibited by several series indicate that the calibration polynomials did not give a sufficiently accurate representation of the resistance vs temperature data).

Fig. 2.10 gives an example of an unstable germanium thermometer. It is shown that the changes in the calibration of the

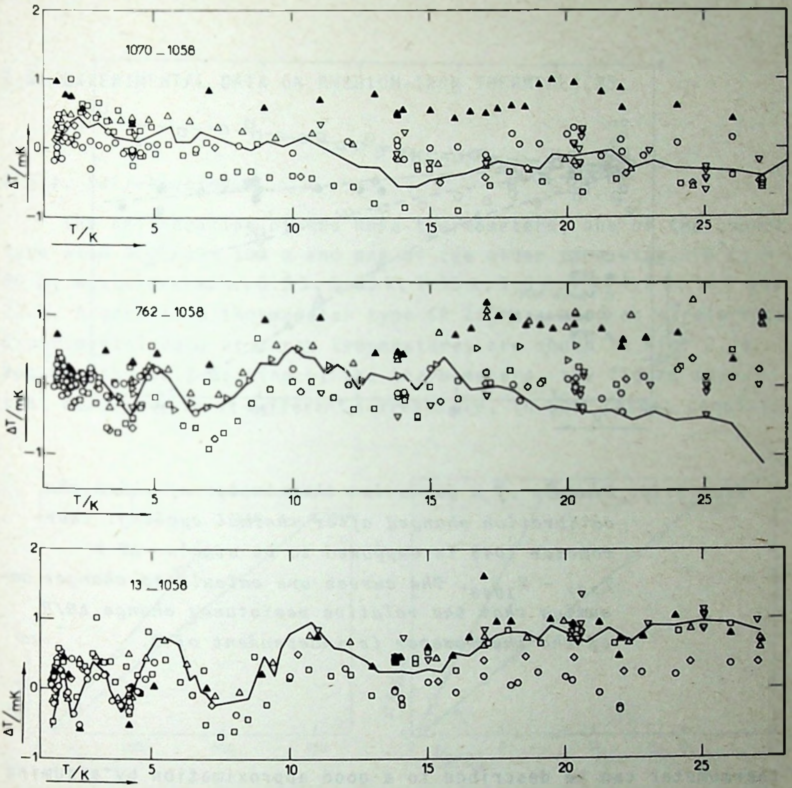

Fig. 2.9. Reproducibility of temperature measurements with relatively stable germanium thermometers (thermometers 1058, 762, 1070 and 13).

○ original calibration by Van Rijn in 1970.

▲ 1970 comparison series □ 1972 comparison series

▽ 1973 comparison series

——— comparison data from NML (Besley and Kemp, 1974)

▷ ◇ 1976 comparison series △ 1977 comparison series.

(Part of the figure has been published also in ref. 15).

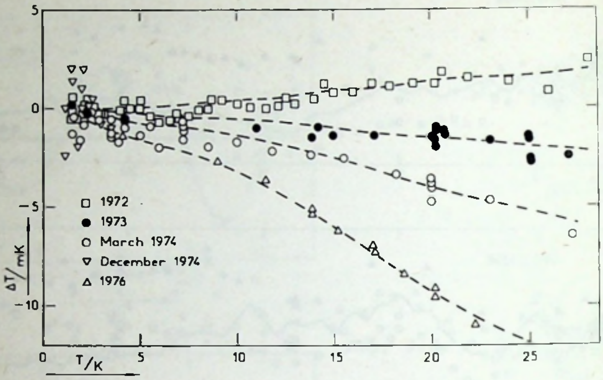

Fig. 2.10. Example of a germanium thermometer of which the calibration changed after thermal cycling. Thermometer 1048 is supposed to be stable. $\Delta T = T_{777} - T_{1048}$. *The curves are calculated changes assuming that the relative resistance change* $\Delta R/R$ *of the thermometer is independent of* T.

thermometer can be described to a good approximation by assuming a temperature independent percentage change in the resistance. Thus, if the thermometer calibration can be checked occasionally at one reference temperature, preferably the highest temperature of the range, a corrected calibration can easily be derived. This feature, and the fact that if the calibration of the thermometer is not corrected the error which is made in the temperature measurements is a smooth function of temperature, makes that (small) changes in the calibration are, in many cases, tolerable[2,4].

2.5. EXPERIMENTAL DATA ON RHODIUM-IRON THERMOMETERS.

2.5.1. *Self-heating.*

The self-heating of two RhFe-thermometers, one of the common type with $R(0^oC) \approx 100\ \Omega$ and one of the older type with $R(0^oC) \approx 50\ \Omega$, was measured at 0.5 K, 0.85 K, 1.15 K, 1.5 K, 2 K, 4.2 K, 14 K and 27 K. A germanium thermometer type CR 250 was used as a reference. Experimental data at three temperatures are shown in Fig. 2.11. Besides the self-heating of the thermometers, the figure shows that measurements at different currents are, in most cases, consist-

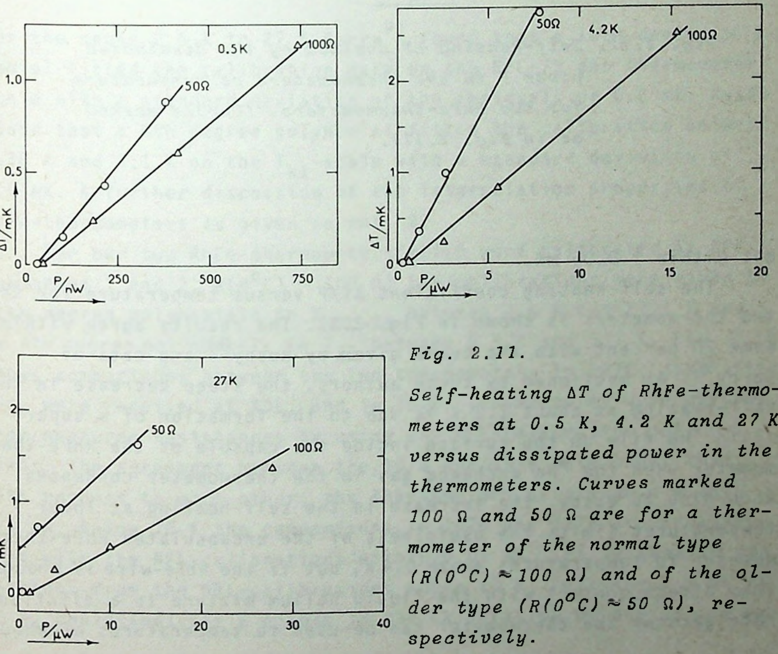

Fig. 2.11.

Self-heating ΔT of RhFe-thermometers at 0.5 K, 4.2 K and 27 K versus dissipated power in the thermometers. Curves marked 100 Ω and 50 Ω are for a thermometer of the normal type ($R(0^oC) \approx 100\ \Omega$) and of the older type ($R(0^oC) \approx 50\ \Omega$), respectively.

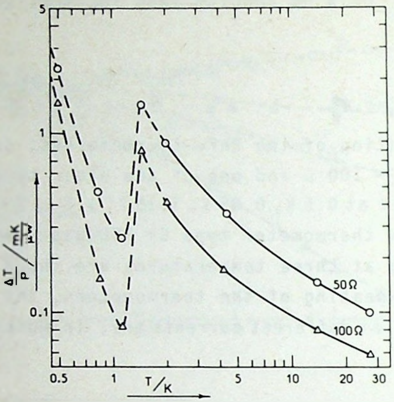

Fig. 2.12. Self-heating ΔT devided by the dissipated power P in the thermometers vs temperature for two RhFe-thermometers. (Curves marked as in Fig. 2.11).

ent within ± 0.05 mK.

The self-heating coefficient ΔT/P versus temperature for the two thermometers is shown in Fig. 2.12. The results agree within some 20 percent with the curve given by Rusby[8] and data of Berry[9]. As mentioned by these authors, the steep decrease in the self-heating at about 1.2 K is due to the formation of a superfluid ^{4}He film on the surface inside the capsule of the RhFe-thermometer when the ^{4}He exchange gas in the thermometer condenses. According to Rusby, the increase in the self-heating at lower temperatures limits the usefulness of the encapsulated RhFe-thermometer to temperatures above 0.4 K, but if the RhFe-wire is brought into direct contact with the liquid helium mixture in a dilution refrigerator the thermometer can be used to temperatures of about

0.1 K$^{8)}$.

The results plotted in Fig. 2.12 were used in Table 2.2 and in the conclusion about the precision of the temperature measurements with RhFe-thermometers in section 2.3.1.

2.5.2. *Calibration and reproducibility.*

Following Rusby$^{6)}$, the calibrations of RhFe-thermometers are represented by polynomials

$$T = \Sigma_{n=0}^{m} b_n R^n$$

For the range 2.6 K to 27 K Berry$^{9)}$ found that a 10th degree polynomial fitted the calibration data on the NPL-75 gas thermometer scale with a standard deviation of the residuals of 0.2 mK. Rusby found that a 4th degree polynomial fitted the calibration between 0.58 K and 3.1 K on the T_{X1}-scale with a standard deviation of 0.1 mK. A further discussion of the interpolation properties of RhFe-thermometers is given in ref. 8.

For our two RhFe-thermometers which were calibrated at NPL (numbered 1 and 2, $R(0^oC) \approx 100\ \Omega$) the calibrations were given by 11th degree polynomials in T_{NPL-75} between 2.6 K and 27.1 K and by 4th degree polynomials in T_{X1} between 0.5 K and 3.1 K. Fig. 2.13 shows comparisons between the two thermometers in 1976, just after they were received at KOL, and in 1978. Temperatures were derived from measured resistances by using the NPL-calibration polynomials. The agreement between the 1976 and 1978 data shows that, with respect to each other, the thermometers were stable within 0.1 mK. Below 10 K the comparisons in 1976 and 1978 are in agreement with the NPL-calibrations within 0.05 mK. The systematic deviations from the NPL-calibrations above 10 K, reaching 0.4 mK at 27 K, could indicate a change in the calibration of one of the two

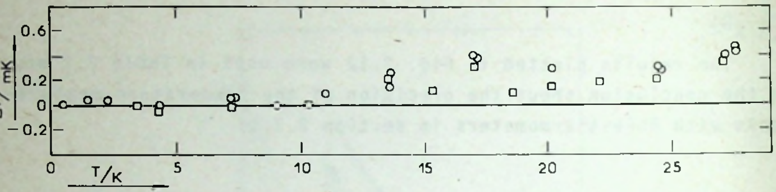

Fig. 2.13. Comparisons of RhFe-thermometers 1 and 2. Temperatures were derived from measured resistances by using the NPL-calibration polynomials.
○ 1976 comparison series
□ 1978 comparison series.

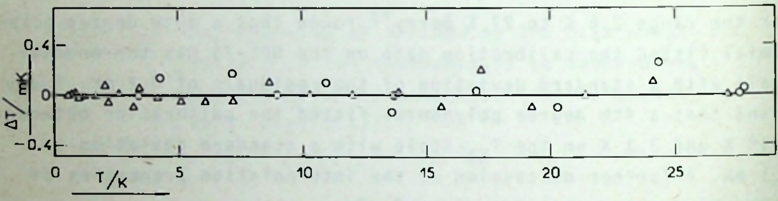

Fig. 2.14. Calibration of RhFe-thermometer 4 ($R(0^{o}C) \approx 50\ \Omega$) against RhFe-thermometer 2. △ 1979 calibration series; points give residuals of the least squares fits using 11th and 4th degree polynomials between 2.6 K and 27 K and between 0.5 K and 2.6 K, respectively.
○ 1978 comparison series (temperatures derived from the 1979-polynomials).

thermometers during the transport from NPL to KOL.

The two other RhFe-thermometers used in our group (numbered 3 and 4, both with $R(0^{o}C) \approx 50\ \Omega$) were calibrated on the NPL-75 and T_{X1}-scales by comparisons with thermometers 1 and 2 and with each

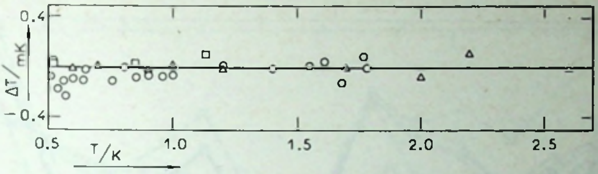

Fig. 2.15. Calibration of RhFe-thermometers 3 and 4 (both with $R(0^{\circ}C) \approx 50\ \Omega$) between 0.5 K and 2.6 K.

△ $T_2 - T_4$ *1979 calibration series of thermometer 4 against 2.*

□ $T_2 - T_4$ *1978 comparison series*

○ $T_3 - T_4$ *1979 calibration series of thermometer 3 against 4.*

other (see Figs. 2.14 and 2.15). The standard deviations of the residuals, using 11th degree polynomials between 2.6 K and 27 K and 4th degree polynomials between 0.5 K and 2.6 K, were about 0.05 mK. Also for the thermometers 3 and 4 the calibrations were stable within 0.1 mK between 1976 and 1978.

2.6. COMPARISON OF TEMPERATURE SCALES MAINTAINED WITH CALIBRATED GERMANIUM AND RHODIUM-IRON THERMOMETERS.

It has been mentioned in section 2.1.2 that the four international germanium thermometers (nos. 1058, 762, 1070 and 13*) define, since 1970, the KOL magnetic scale (T_m(III)-scale) and carry, since 1976, also the T_{XAc}'-scale. Figs. 2.16 and 2.17 show

---

* The thermometer no. 13 was kindly given in 1965 by Dr. Harmon H. Plumb of the National Bureau of Standards, with a calibration on the NBS 2-20 scale.

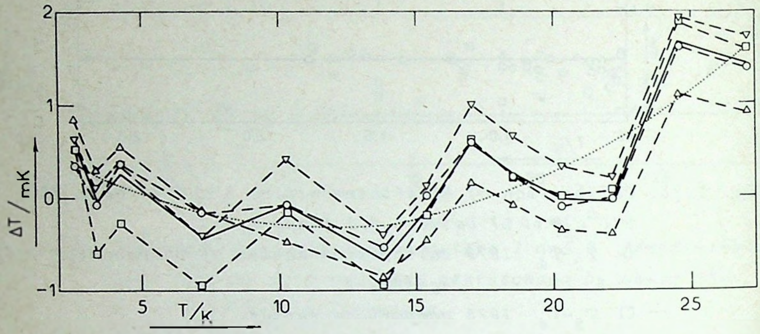

Fig. 2.16. Comparison of temperature scales carried by germanium and RhFe-thermometers. $\Delta T = T_{76,mIII} - T_{76,NPL-75}$ *(see text) Different points indicate that* $T_{76,m(III)}$ *was derived from* $T_m(III)$ *for the different germanium thermometers. The full drawn curve gives the average for the four germanium thermometers.* $T_{76,NPL-75}$ *was derived from* T_{NPL-75} *as given by RhFe-thermometer 2. The dotted curve is explained in the text.*

the result of a comparison of these thermometers in 1977 with RhFe-thermometer 2 carrying the NPL-75 gas thermometer scale and the T_{X1}-scale. Temperatures $T_m(III)$, $T_{XAc'}$ and T_{NPL-75} were recalculated to T_{76} using the scale differences in Table 5 of the EPT-76 (see Appendix), the recalculated temperatures being denoted $T_{76,m(III)}$, $T_{76,XAc'}$ and $T_{76,NPL-75}$, respectively.

The differences between the curves for the four thermometers in the figures show how far the thermometers drifted apart in seven years since their original calibration on the $T_m(III)$-scale (Fig. 2.16) or in three years after their calibration on the $T_{XAc'}$-scale (Fig. 2.17). The full drawn curve in Fig. 2.16 gives the difference between the EPT-76 as realized by means of the $T_m(III)$-scale and the NPL-75 and T_{x1}-scales; the full drawn curve in

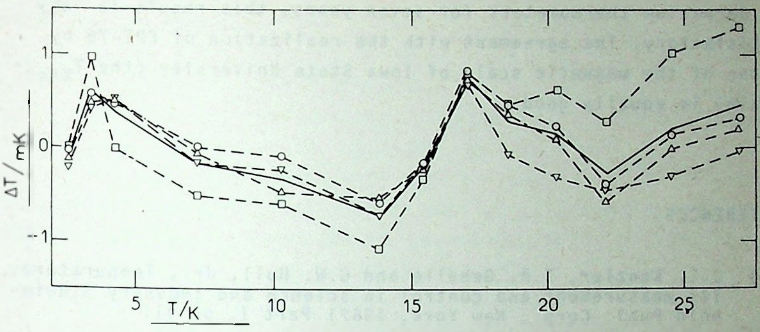

Fig. 2.17. Comparison of temperature scales carried by germanium and RhFe-thermometers.

$\Delta T = T_{76,XAc'} - T_{76,NPL\text{-}75}$ *(see text).*

Points indicate results for different germanium thermometers; the full drawn curve gives the average for the four thermometers.

Fig. 2.17 the comparable differences for the $T_{XAc'}$-scale. Between 2 K and 22 K the differences are within ± 0.7 mK in both cases. For the case of the T_m(III)-scale the differences are large above 22 K.

From Fig. 2.16 it can be seen that an equation $\Delta T=a+bT+cT^2$ (the dotted line in the figure) fits the experimental points $T_{76,m(III)} - T_{76,NPL\text{-}75}$ with a maximum deviation of 0.6 mK (and a standard deviation of 0.3 mK). This means that if the scale difference $T_m(III) - T_{76}$ in Table 5 of the EPT-76 is slightly revised, the realizations of the EPT-76 with the KOL magnetic scale and with the NPL-75 gas thermometer scale agree to within 0.6 mK between 2.6 K and 27 K. In view of the different basis of the two scales and the fact that the KOL magnetic scale has been retained on germanium thermometers for seven years, this result is very satisfactory. The agreement with the realization of EPT-76 by means of the magnetic scale of Iowa State University (the T_{XAc}'-scale) is equally good.

REFERENCES.

1) J.E. Kunzler, T.H. Geballe and G.W. Hull, Jr., Temperature, its measurement and control in science and industry 3(Reinhold Publ. Corp., New York, 1962) Part 1, p. 41.

2) C. van Rijn, J.L. Tiggelman, M.C. Nieuwenhuys-Smit, M. Durieux and J.E. van Dijk, Temperature, its measurement and control in science and industry 4(Instrum. Soc. of America, Pittsburgh, 1972) Part 2, p. 75.

3) L.M. Besley and H.H. Plumb, Rev. Sci. Instr. 49(1978)68.

4) M.S. Anderson and C.A. Swenson, Rev. Sci. Instr. 49(1978) 1027.

5) B.R. Coles, Phys. Lett. 8(1964)243.

6) R.L. Rusby, Temperature, its measurement and control in science and industry 4(Instrum. Soc. of America, Pittsburgh, 1972) Part 2, p. 865.

7) R.L. Rusby, J. Phys. F: Metal Phys. 4(1974)1265.

8) R.L. Rusby, Inst. Phys. Conf. Ser. No. 26(London 1975) p. 125.

9) K.H. Berry, Metrologia 15(1979)89.

10) R.L. Rusby and C.A. Swenson, Metrologia, to be published.

11) C. van Rijn and M. Durieux, Temperature, its measurement and control in science and industry 4(Instrum. Soc. of America, Pittsburgh, 1972) Part. 1, p. 73.

12) L.M. Besley and W.R.G. Kemp, Metrologia (1977)35.

13) J.L. Tiggelman, Ph.D. Thesis, Leiden, 1973.

14) J.S. Blakemore, J. Winstel and R.V. Edwards, Rev. Sci. Instr. 41(1970)835.

15) M. Durieux, Inst.Phys.Conf.Ser. No. 26(London, 1975) p. 17.

CHAPTER 3.

EXPERIMENTAL DATA ON THE USE OF THE SUPERCONDUCTIVE TRANSITION POINTS OF Pd, In, Al, Zn AND Cd AS THERMOMETRIC REFERENCE POINTS.

3.1. INTRODUCTION.

The use of the superconductive transition points of Pb, In, Al, Zn and Cd in zero magnetic field as thermometric reference points was first investigated by Schooley1). Later studies2,3) confirmed the applicability of these transition points in thermometry. The transition points of the five metals are now reference points of the EPT-764) (see Appendix). Except for one experiment, in the Moscow Standards Laboratory5), all recent thermometric measurements of the transition points are made with the National Bureau of Standards SRM 767 device6) (Standard Reference Materials 767), see section 3.2. More than one hundred of these devices have been distributed by NBS; they are used in many laboratories for calibrations of resistance thermometers or magnetic thermometers. However, at the time that the EPT-76 was established little precise information on the behaviour of SRM 767 devices, such as small differences in transition temperatures between devices, shapes of transition curves and long time stability, was published7,8,9). Therefore, it was proposed10) that the National Physical

Laboratory in Teddington (NPL), the National Measurement Laboratory in Sydney (NML) and the thermometry group at KOL, which together had used eight SRM 767 devices, should jointly publish their results on the realization of the superconductive reference points[11)] as a supplementary paper to the EPT-76.

At KOL new measurements were made on three devices. It was a considerable advantage, compared to earlier experiments, that stable rhodium-iron thermometers were available for the temperature determinations, the KOL thermometers being calibrated on the same scales as the NPL thermometers (see Chapter 2). In sections 3.2, 3.3, 3.4 and 3.5 the new KOL measurements and results are presented. In section 3.6 an attempt is made to compare older KOL measurements[7)] on the three devices with the new data. In section 3.7 the NPL data on three devices are compared with the KOL data.

In section 3.8 the NML data on two devices are compared with the NPL and KOL results.

A conclusion is given in section 3.9.

3.2. EXPERIMENTAL ARRANGEMENT.

3.2.1. *The SRM 767 device.*

The SRM 767 device is shown in Fig. 3.1. The device is described in detail in ref. 6. Samples of the five metals Pb, In, Al, Zn and Cd, small rods of about 30 mm long and 1.5 mm in diameter, are varnished into a copper stud which terminates in a thread for thermally connecting the device to, e.g., a copper block. The five metal rods are surrounded by two coils forming a mutual inductance. The change in the mutual inductance when passing from the superconductive to the normal state of each of the metals is used for the detection of the transition. The transition temperature T_c is defined as the temperature at the midpoint of the induc-

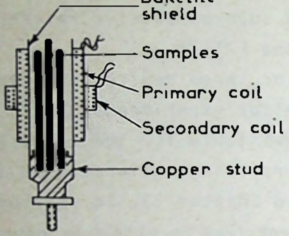

Fig. 3.1.

The Standard Reference Material (SRM) 767 device of NBS for measuring the superconductive transition points of Pb, In, Al, Zn and Cd as thermometric reference points (only three metal samples are indicated in the figure) (see ref. 6).

tance change. The inductance change is about 10 μH for each metal. The primary current in the mutual inductance was 20 μA, corresponding to a magnetic induction of 0.4 μT inside the coils. This induction has a negligible effect on the transition temperatures (see below). The frequency of the current was 227 Hz. The inductance changes were measured by balancing, before each measurement, the mutual inductance of the SRM 767 device with a Hartshorn mutual inductance bridge and recording the bridge unbalance, after amplification with a PAR preamplifier and lock-in amplifier (type 220). The total voltage change at the input of the amplifier when passing a transition was about 0.3 μV, one percent of this could easily be detected. At each measurement the earth magnetic induction, and stray magnetic inductions which might be present, must be compensated to within about 0.5 μT (the dependences of the transition temperatures on the magnetic induction are 49 K/T, 64 K/T, 56 K/T, 86 K/T and 106 K/T for Pb, In, Al, Zn and Cd, respectively[12]). This was accomplished by trimming the currents in three perpendicular sets of coils outside the cryostat so that a maximum value for T_c was obtained.

Three SRM 767 devices, numbered 102, 103 and 108 were used.

3.2.2. *The resistance thermometers.*

Two rhodium-iron resistance thermometers, RhFe 2 and RhFe 4, were used for the temperature measurements. Both thermometers were calibrated on the NPL-75 scale between 2.6 K and 27 K and on the T_{X1}-scale between 0.5 K and 2.6 K (see Chapter 2). Thermometer RhFe 2 was considered to give the most accurate results because its resistance was twice that of RhFe 4. There were also 15 germanium resistance thermometers of several types in the cryostat of which five were calibrated earlier; the other ten were calibrated in these experiments for use in other groups in the laboratory. One calibrated germanium thermometer (type CR 250) was used in the measurements of the transition curves (see below), because its sensitivity is higher than that of the RhFe thermometers (see Chapter 2).

3.2.3. *The cryostat.*

The cryostat described in section 2.3.2b, Fig. 2.5 was used. The three SRM 767 devices were mounted on top of the copper block with the lead wires for the primary and secondary coils thermally anchored to the block and the shield. The RhFe and germanium resistance thermometers were mounted in the copper block as described in section 2.3.2.

The measurements were made with the weak thermal link between block and surrounding shield attached, so that the block slowly followed temperature variations of the shield. By appropriate settings of the electronically regulated shield temperature, the block temperature could be kept constant, or varied, with a resolution of 0.01 mK.

3.3. MEASUREMENTS PROCEDURE.

The measurements at each transition point consisted of:
1) A preliminary recording of the inductance change and the block temperature, as indicated by one of the germanium resistance thermometers (type CR 250), versus time on a two-channel recorder during a passage of the transition, in order to determine the transition midpoint. At the midpoint the currents in the three perpendicular sets of coils outside the cryostat were adjusted such that the inductance of the SRM 767 device had its minimum value, i.e. the transition temperature had its maximum value and the magnetic induction at the site of the sample was zero. In practice, the values of the compensating induction were always between about the earth magnetic induction of 48 μT and a value which is 10 percent higher.

2) A measurement of the transition curve, i.e. the inductance change versus temperature, with increasing and decreasing temperature. The transition curves were derived from recordings of the inductance change and the block temperature versus time. An example of these recordings is given in Fig. 3.2. The corresponding transition curve is included in Fig. 3.5. The transition midpoint and the width of a transition, which is defined here as the temperature interval centered around the transition midpoint in which 80 percent of the transition takes place, were determined from the transition curves.

The rate of temperature change was usually 1 mK in 30 to 90 min during the passage of the central 80 percent of the transition increasing to 1 mK per 5 min in the tails of the transition.

3) Measurements of the transition temperatures. The temperature of the block was adjusted to the transition midpoint (by adjusting the shield temperature) and, after checking the currents in the field compensator for maximum T_c, the resistances of the two rhodium-iron thermometers and of several of the germanium thermometers were measured. Two determinations of T_c were made for each

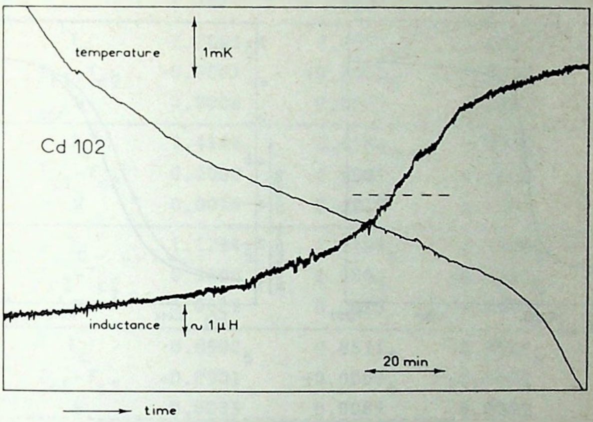

Fig. 3.2. Inductance change and temperature (as measured with a germanium thermometer) versus time during the passage of the superconductive transition point of Cd (device 102). The dashed line indicates the midpoint of the transition.

sample, one with the midpoint approached from the low temperature side and one approached from the high temperature side.

3.4. RESULTS.

Experimental results are given in Table 3.1 and Figs. 3.3, 3.4 and 3.5. The T_c-values given in Table 3.1 are those measured with RhFe-thermometer 2, and are averages of the two measurements at the midpoint of the transition, i.e. with the midpoint approach-

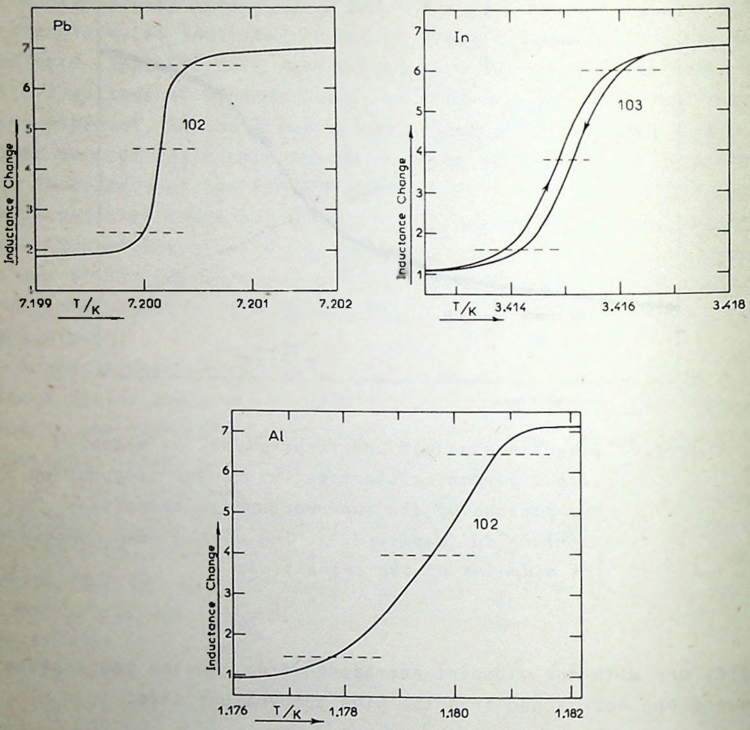

Fig. 3.3. Inductance change versus temperature during the passage of the superconductive transition for Pb, In and Al (zero field). The dashed lines indicate the midpoints ±40% of the transitions. (The small difference between the curves for increasing and decreasing temperature for In is probably not a real hysteresis but due to a slight inconsistency in the temperature measurement with the germanium thermometers). The inductance change is in arbitrary units.

Table 3.1. Transition temperatures T_c and widths of transition W measured, for three SRM 767 device (nos 102, 103 and 108). Temperatures are on the NPL-75 scale for Pb, In and Al and on the T_{X1}-scale for Zn and Cd.

Device no.		102	103	108	δT
Pb	T_c	7.2001_5K	7.2001_5K	7.2001 K	0.0_5 mK
	$T_{c1}-T_{c2}$	-0.0001	$+0.0000_5$	0.0000	
	W	0.0005	0.0009	0.0003	
In	T_c	3.4145_5	3.4148_5	3.4147_5	0.2_5
	$T_{c1}-T_{c2}$	0.0000	-0.0001	0.0000	
	W	0.0014	0.0019	0.0009	
Al	T_c	1.1794_5	1.1794_5	1.1796_5	0.2
	$T_{c1}-T_{c2}$	0.0000	0.0000	$+0.0000_5$	
	W	0.0029	0.0025	0.0025	
Zn	T_c	0.8502_5	0.8511_5	0.8505_5	0.9
	$T_{c1}-T_{c2}$	-0.0001	$+0.0000_5$	-0.0001	
	W	0.0033	0.0089	0.0061	
	T_c	0.8503	0.8510		
	$T_{c1}-T_{c2}$	0.0000_5	0.0000_5		
	W	0.0030	0.0086		
Cd	T_c	0.5200	0.5203_5	0.5196_5	0.6_5
	$T_{c1}-T_{c2}$	0.0000	-0.0000_5	0.0000	
	W	0.0029	0.0062	0.0006	
100 μA	T_c	0.5198_5			
	$T_{c1}-T_{c2}$	0.0000_5			
	W	0.0026			
without earth field compensation	T_c	0.5146			
	$T_{c1}-T_{c2}$	0.0002			
	W	0.0029			

T_c is the temperature at the midpoint of the transition and is the average of two determinations T_{c1} and T_{c2} (see text), δT is the spread in T_c values for the three devices, W is the temperature interval centered around the midpoint, in which 80% of the inductance change occurs. All measurements were in zero field and for a primary current of 20 μA except where indicated.

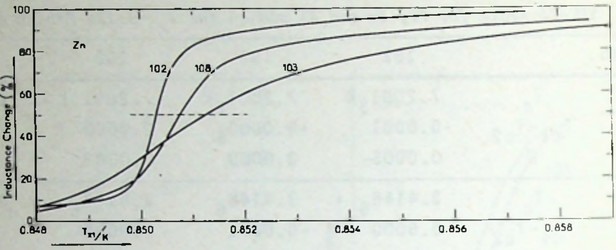

Fig. 3.4. Inductance change versus temperature for the superconductive transition of Zn measured with the three SRM 767 devices (zero field).

ed from the low temperature side, T_{c1}, and approached from the high temperature side, T_{c2}. Differences $T_{c1}-T_{c2}$ are between -0.12 mK and + 0.08 mK and are believed to be due to experimental inaccuracies and not to hysteresis.

The temperatures measured with RhFe-thermometer 4 were within 0.1 mK equal to those obtained from thermometer 2, except in two cases where the differences were 0.2 mK. Temperature differences between devices derived from the germanium thermometer readings, agreed with those derived from RhFe-2 within 0.1 mK (except in two cases where the disagreement was 0.2 mK).

Temperatures are on the NPL-75 scale for Pb, In and Al and on the T_{X1}-scale for Zn and Cd.

The spread in T_c-values between the three devices was smaller than 0.3 mK for Pb, In and Al but considerably larger for Zn and Cd (see Table 3.1).

The widths of the transitions, W, are also given in Table 3.1. The transition curves for Pb, In and Al are quite similar for the three devices. Examples are shown in Fig. 3.3. For Zn and Cd large differences in widths between devices occur; the transition curves

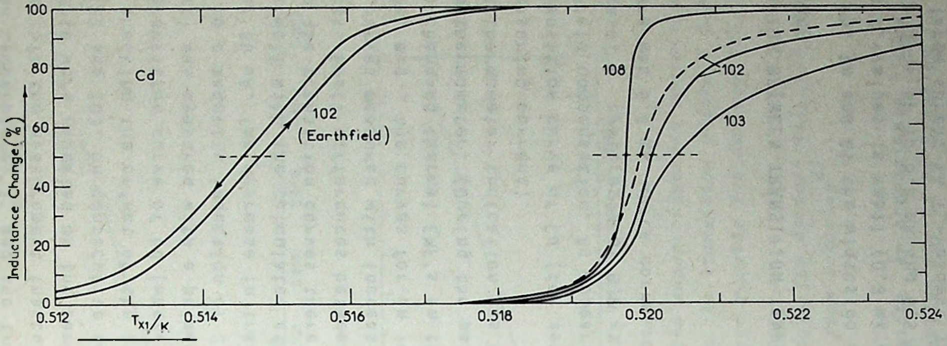

Fig. 3.5. *Inductance change versus temperature for the superconductive transition of Cd measured with the three SRM 767 devices with field compensation (zero field). For device 102 the transition was also measured with a primary current of 100 μA (ac induction about 2 μT) instead of the usual current of 20 μA (dashed line) and without field compensation (curve marked earth field).*

for all three devices are given in Figs. 3.4. and 3.5. Both the transition temperatures T_c and the transition curves were reproducible within the measurement inaccuracy (0.05 to 0.1 mK) when measurements were repeated on the same or, in the case of Zn for devices 102 and 103, on consecutive days.

For checking the effect of the primary current in the inductance a transition curve of Cd (where the magnetic field effect is largest) was measured with a primary current of 100 μA (corresponding to a magnetic induction of 2 μT) instead of the usual current of 20 μA. The decrease in transition temperature of 0.1_4 mK agrees well with the calculated effect of 0.17 mK.

From the transition curves there was no evidence of hysteresis effects. Small differences between the inductance versus temperature curves measured with increasing and decreasing temperatures (see, e.g., the curves for In in Fig. 3.3) are believed to be due to changing thermal EMF's in the potential leads of the germanium thermometer. (During the passage of the transition the germanium thermometer indication was recorded, without reversal of the measuring current).

A transition curve of Cd (for device 102) was also measured without field compensation. As appears from Fig. 3.5 there was, in this case, a hysteresis of 0.2 mK. The decrease in transition temperature was 5.4 mK, which corresponds to a magnetic induction of 51 μT.

3.5. RELATION BETWEEN TRANSITION TEMPERATURES AND WIDTHS OF TRANSITIONS.

For Pb, In and Al the widths do not exceed 3 mK and the spread in T_c-values is small (0.3 mK or less). For Zn and Cd, however, widths up to 8.9 mK and 6.9 mK occur and there is a relatively large spread in T_c-values. In Fig. 3.6 the transition temperatures are plotted versus the widths for Zn and Cd. Within the inaccuracy of the measurements there is a linear relation be-

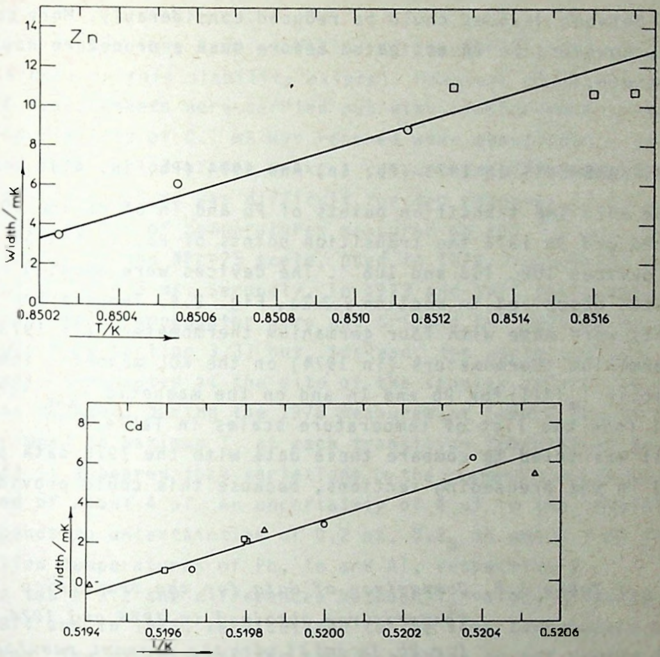

Fig. 3.6. Dependence of the superconductive transition temperatures T_c on the transition widths W for Zn and Cd.
○ *present results for devices 102, 103 and 108.*
□ *NPL data for devices 111, 112 and 113.*
△ *data from Schooley3).*

tween T_c and the transition width. An approximate linear relation between T_c and W was found earlier for Cd by Schooley3).

Fig. 3.6, and also the transition curves in Figs. 3.4 and 3:5, suggest that by defining T_c as the transition temperature extrapolated to zero width (or recalculated to the same width) the spread between devices could be reduced considerably. More devices should, however, be investigated before such a procedure could be recommended.

3.6. MEASUREMENTS IN 1973 (Pb, In) AND 1974 (Pb, In, Al).

In 1973 the transition points of Pb and In of device 108 were measured and in 1974 the transition points of Pb, In and Al of the three devices 102, 103 and 108$^{7)}$. The devices were mounted in the apparatus described in section 2.3.2a, Fig. 2.4. Temperature measurements were made with four germanium thermometers (in 1973) and two germanium thermometers (in 1974) on the KOL magnetic temperature scale T_m(III) for Pb and In and on the magnetic $T_{XAc'}$-scale for Al (see the list of temperature scales in Table 1.1).

It was tried to compare these data with the 1978 data presented in the preceeding sections, because this could provide in-

Table 3.2. Comparison of data for the transition temperatures obtained in 1973 and 1974 for Pb, In and Al with the present results (T_c(1978)). $\Delta T=T_c$ (1973,1974)$-T_c$(1978).

Device no.	102 ΔT/mK (1974)	103 ΔT/mK (1974)	108 ΔT/mK (1973)	 (1974)
Pb	-0.8	-0.7	-0.3	-0.8
In	-0.4	-0.5	+0.4	+0.5
Al	+0.3	+0.8		+0.4

formation on the long term stability of the SRM 767 devices. (Little data on this stability exists). However, although the 1973-74 measurements were carried out with considerable care and a reproducibility of 0.1 mK was reached when measurements were repeated in a period of one week, a comparison with the present results within 0.5 mK was difficult for two reasons: Firstly, the recalculation of temperatures measured on the T_m(III)- and $T_{XAc'}$-scales to the NPL-75 scale, used in 1978, introduced uncertainties of 0.3 mK. Secondly, in 1973 and 1974 the currents in the earth field compensator were not trimmed for getting maximum T_c-values (see section 3.3) but, instead, the earth magnetic field was compensated at the site of the samples before the cryostat was mounted. During the 1978 measurement (where the currents were trimmed to maximum T_c at each transition temperature measurement) it appeared that variations in the compensating inductance occurred of about 4 µT. An uncertainty of 4 µT in the induction corresponds to uncertainties of 0.2 mK, 0.2_5 mK and 0.4 mK in the transition temperatures of Pb, In and Al, respectively.

In table 3.2 the differences between T_c-values measured in 1973-1974 and in 1978, recalculated to the same temperature scale, are given. The differences for the In samples do not exceed the sum of the uncertainties mentioned above; for the Pb samples and for Al, device 103, they are larger.

3.7. COMPARISON WITH THE NPL DATA FOR THREE SRM 767 DEVICES.

In Fig. 3.7 the T_c-values measured at NPL by Rusby for the SRM 767 devices 111, 112 and 113 are compared with the KOL data for devices 102, 103 and 108. The widths of the transitions measured at NPL and KOL are also shown.

The temperatures measured at NPL and KOL are on the same scales: The NPL-75 scale for Pb and In and the T_{X1}-scale for Al, Zn and Cd. The excellent reproducibility of the rhodium-iron thermometers with which these scales are maintained, makes that the realizations of these scales at NPL and KOL are equal within 0.2 mK and probably within 0.1 mK (see Chapter 2). There are no systematic differences between the NPL and the KOL data, but in some cases, significant differences between the devices occur.

For Pb the T_c-values of five devices are within 0.2_5 mK, but there is one device (111) of which T_c is 1.0 mK higher than the average of the others. The width for device 111 (1.5 mK) is also higher than for the other devices. For In the T_c's of all devices are within 0.3_5 mK. For Al five devices give T_c-values within

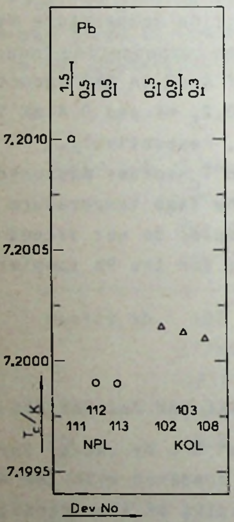

Fig. 3.7a.

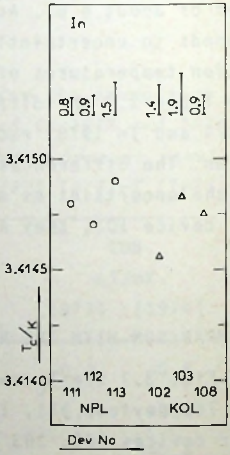

Fig. 3.7b.

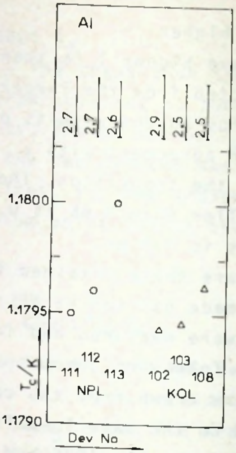

Fig. 3.7c.

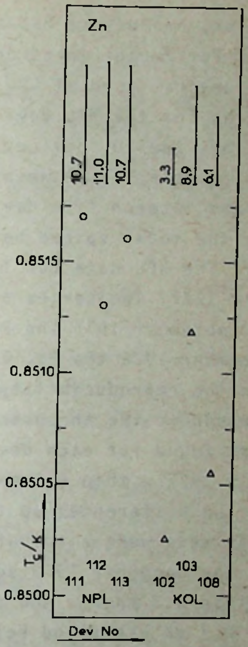

Fig. 3.7d.

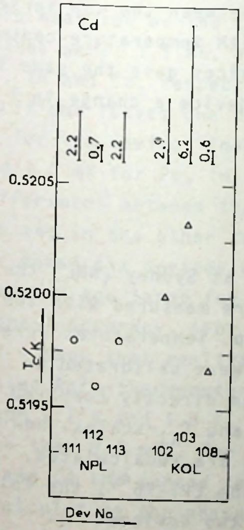

Fig. 3.7e.

Fig. 3.7.

NPL and KOL data for the superconductive transition temperatures T_c obtained for in total six SRM 767 devices. Temperatures are on the NPL-75 scale for Pb and In and on the T_{X1}-scale for Al, Zn and Cd. The measured transition widths, in millikelvins, are indicated in the upper parts of the figures.

0.2 mK, one device has a T_c which is 0.4 mK higher.

For Zn the three devices used at NPL have higher T_c's than the devices used at KOL which could be expected from the larger widths for the NPL devices (see Fig. 3.7). For Cd the results of the NPL and KOL devices are within 0.1 mK in agreement with a linear relation between T_c and the width of the transition. The spread between five devices with widths smaller than 3 mK is 0.4 mK, the total spread between the six devices is 0.7 mK.

The NPL data for T_c shown in Fig. 3.7 are those obtained in July 1977. Two series of measurements were made earlier at NPL: in September 1976 the Pb, In and Al points were measured and in December 1976 the Pb, Zn and Cd points. The following conclusions for the reproducibility of the T_c-values were drawn from the comparison of the three series. For In, Al and Zn the same T_c-values were found for each device in the series of September 1976 and July 1977 within 0.2 mK and for all but two samples within 0.1 mK. For Cd differences up to 0.4 mK were found between the two series but these were attributed to difficulties with temperature control in the December 1976 series. For Pb, two devices gave the same T_c within 0.3 mK for the three series for one device a change in T_c of 0.7 mK was found between two series.

3.8. RESULTS OF NML.

At the National Measurement Laboratory at Sydney (NML) the transition temperatures of Pb, In and Al were measured with two SRM 767 devices by R.C. Kemp and W.R.G. Kemp. Temperatures were measured with germanium thermometers which were calibrated on the T_{XISU75} - scale* so that they cannot be directly compared with the NPL and KOL results on the NPL-75 and T_{X1}-scales. However, when the NML data for the two devices are recalculated, using the scale differences in Table 5 of the EPT-76[4)], the following results for T_c are obtained for the two devices: Pb: 7.1994 K and 7.1993 K; In: 3.4150 K and 3.4145 K; Al: 1.1797 K and

---

[*] a slightly revised version of the $T_{XAc'}$-scale.

1.1804 K.

The transition temperatures were measured several times with thermal cycling to room temperature between successive measurements. The spread in T_c values found in five or more measurements of the two devices was for the lead point 0.4 mK for each of the devices, for the indium point 0.0 mK and 0.6 mK and for the aluminium point 0.4 mK and 0.8 mK.

3.9. CONCLUSIONS.

The NPL and NML data and the KOL data presented in sections 3.4 - 3.6 have provided precise information on the realization of the superconductive transition points for eight SRM 767 devices. The following conclusions can be drawn:

a) For the six devices of NPL and KOL the transition temperatures were measured on the same temperature scales. The spreads in T_c-values were: 1.1 mK, 0.4 mK, 0.6 mK, 1.5 mK and 0.7 mK for Pb, In, Al, Zn and Cd, respectively. (For Pb the spread is reduced to 0.2_5 mK if one leaves one device out). With the NML data on Pb, In and Al for two devices included the spreads increase to 1.7 mK, 0.5 mK, and 1.0 mK for Pb, In and Al, respectively, but this may be due to differences between the (recalculated) temperature scales used at NML and in the other two laboratories. Although spreads as found for these six devices will not be serious for most of the users, they are too large for using the devices in thermometry of the highest accuracy. (For comparison, data presented in Chapters 2 and 4 show that realizations of a scale defined in terms of calibrated RhFe-thermometers or of the ^{3}He vapour pressure scale between 1.2 K and 1.8 K, in different laboratories do not differ by more than 0.2 mK). The data presented suggest, however, that for Zn and Cd the spread could be reduced by a factor of 2 or 3 by recalculating the measured T_c-values to a certain standard transition width.

It may be mentioned here that at NBS superconductive transitions in pure single crystals have been studied; the transitions were found to be narrower than for polycrystalline material[13)]. Suggestions have been made for making a few SRM 767 devices with single crystal material of the five metals. These devices could then be circulated among various thermometry groups[13)] for testing and for comparisons of laboratory temperature scales.

b) Regarding the stability of the devices, it was found that changes in the transition temperatures were less than 1 mK for

Table 3.3. Assigned EPT-76 values of the superconductive transition temperatures of Pb, In, Al, Zn and Cd and average experimental values for T_{NPL-75} and T_{X1} deduced from measurements with six SRM 767 devices at NPL and KOL. The last column gives the average experimental values recalculated to T_{76}.

	assigned value	average value for six devices		
	T_{76}/K	T_{NPL-75}/K	T_{X1}/K	T_{76}/K
Pb	7.1999	7.2000*		7.2003*
In	3.4145	3.4148		3.4149
Al	1.1796		1.1794	1.1794
Zn	0.851		0.8511	0.8511
Cd	0.519		0.5199	0.5199

* average with device 111 not included.

three devices after one year (NPL), for two devices on repeated cycling between room temperature and low temperatures (NML) and for three devices after four years (KOL). Most of the changes that have been found could be attributed to experimental uncertainties, but for one Pb sample a real change of 0.7 mK between two measurements, with a half year interval, was found.

c) When the EPT-76 was established, best values had to be assigned to the five superconductive transition points$^{9)}$. This task was besieged with some difficulties because transition temperatures were measured in various laboratories on different temperature scales. The present NPL and KOL data provide accurate data of T_c for six devices on the NPL-75 scale (Pb, In) and the T_{X1}-scale (Al, Zn, Cd). In table 3.3 the assigned values for T_c in the EPT-76 are given, together with the average T_{76}-values for T_c derived from the new NPL and KOL data. It can be seen that the assigned values agree within 0.4 mK with the new average values, except for Cd where the difference is 0.9 mK. (The assigned T_{76}-value for the cadmium point, however, was not determined as an average best value of T_c on the T_{X1}-scale, but as the average of the NPL data for their three devices on the T_{X1}- scale (0.5197 K ± 0.0005 K) and a determination on a noise thermometer scale by Soulen (0.5190 K ± 0.0006 K)$^{9)}$.

Acknowledgement.

Our sincere thanks are due to Dr. R.L. Rusby from the National Physical Laboratory in Teddington and Drs. R.L. Kemp and W.R.G. Kemp from the National Measurement Laboratory in Sydney for allowing us to include their results in this chapter before the joint publication of the NPL, NML and KOL data appears.

REFERENCES.

1) J.F. Schooley and R.J. Soulen, Jr., Temperature, its measurement and control in science and industry 4 (Instrument Society of America, Pittsburgh, 1972), Part 1, p. 169.

2) R.J. Soulen, Jr. and J.H. Colwell, J. Low Temp. Phys. 5(1971)325.

3) J.F. Schooley, J. Low Temp. Phys. 12(1973)421.

4) The 1976 Provisional 0.5 K to 30 K Temperature Scale (EPT-76), Metrologia 15(1979)65.

5) D.N. Astrov, private communication (1978).

6) J.F. Schooley, R.J. Soulen, Jr. and G.A. Evans, Jr., Preparation and use of Superconductive Fixed Point Devices, SRM 767 (NBS Special Publication 260-44, 1972).

7) J.E. van Dijk and M. Durieux, Comité Consultatif de Thermométrie, 10^{e} Session (1974) p. T119.

8) L.M. Besley and W.R.G. Kemp, Metrologia 13(1977)35.

9) M. Durieux, D.N. Astrov, W.R.G. Kemp and C.A. Swenson, Metrologia 15(1979)57.

10) Comité Consultatif de Thermométrie, Report of 12th Session (1978), section 7.

11) A.E. Samahy, M. Durieux, R.C. Kemp, W.R.G. Kemp and R.L. Rusby, An evaluation of the SRM 767 superconductive fixed point device, Metrologia, to be published.

12) J.F. Schooley, R.J. Soulen, Jr. and G.A.Evans, Jr., Natl. Bur. Stand., Washington, Certificate SRM 767 device.

13) R.J. Soulen, Jr., private communication (1979).

CHAPTER 4.

MEASUREMENTS OF VAPOUR PRESSURES OF LIQUID ^{4}He AND ^{3}HE BELOW 2 K.

4.1. INTRODUCTION.

4.1.1. *Vapour pressures of liquid ^{4}He and ^{3}He and thermometry.*

Vapour pressure relations of ^{3}He and ^{4}He provide between about 0.5 K and 5.2 K a continuous range of calibration points for calibrations of resistance thermometers, low temperature magnetic thermometers and other practical thermometers. The "1958 ^{4}He Scale"$^{1)}$ and the "1962 ^{3}He Scale"$^{2-5)}$ gave internationally agreed tables for deriving temperatures, T_{58} and T_{62}, from measured vapour pressures. The scales were replaced (i.e. corrected) in 1978 by the "1976 Provisional 0.5 K to 30 K Temperature Scale" (EPT-76)$^{6)}$. Differences T_{58}-T_{76} and T_{62}-T_{76} are given in Table 3 of the EPT-76 (see Appendix) and are shown in Fig. 1.1.

4.1.2. *Vapour pressures of liquid ^{4}He.*

The 1958 ^{4}He Scale was based on gas thermometer and magnetic thermometer data and, below 2 K, also on a thermodynamic calculation of the vapour pressure equation$^{1)}$.

Recently, new vapour pressure vs temperature measurements for liquid ^{4}He were made between 1.2 K and 5.2 K by Rusby and Swenson$^{7)}$ using above 2.6 K the NPL-75 (gas thermometer) scale and below 2.6 K the T_{X1}-scale (a magnetic scale based on cerium magnesium nitrate) (see Table 1.1 for a list of temperature scales used in this thesis). Klein et al.[8] determined ^{4}He vapour pressures on a noise thermometer scale at six temperatures between 2.1 K and 4.2 K. The noise thermometer based vapour pressures agreed at 4.2 K with those measured on the NPL-75 scale within the equivalent of 0.1 mK and between 2 K and 4.2 K with those on the NPL-75 and T_{XI}-scales within 0.4 mK. Both experiments confirmed earlier conclusions that T_{58} was at 4.2 K about 7 mK lower than the thermodynamic temperature[9].

The thermodynamic vapour pressure relation of liquid ^{4}He can be written as[1]

$$\ln p = i_o - \frac{L_o}{RT} + \frac{5}{2}\ln T - \frac{1}{RT}\int_o^T S_L dT + \frac{1}{RT}\int_o^p V_L dp + \varepsilon \qquad (4.1)$$

where $i_o = \ln (2\pi m)^{3/2}\ k^{5/2}/h^3$

and $$\varepsilon = \ln \frac{pV_G}{RT} - \frac{2B}{V_G} - \frac{3C}{2V_G{}^2} \qquad (4.2)$$

p is the vapour pressure, L_o is the molar heat of vaporization at T = 0 K, R is the molar gas constant, S_L and V_L are the molar entropy and volume of the liquid under saturation pressure, m is the mass of a helium atom, k is Boltzmann's constant, h is Planck's constant, V_G is the molar volume of the vapour at saturation pressure, B and C are the molar second and third virial coefficients of the gas.

The last three terms in eq. (4.1) are usually referred to as the correction terms. The correction terms and their uncertainties increase rapidly with temperature which makes eq. (4.1) less useful for the calculation of vapour pressures above about 2 K. Below 2 K the equation is quite useful because the correction terms and their uncertainties are small so that there is only one adaptable parameter, L_o, which can be determined by fitting the equation to experimental vapour pressure vs temperature data.

Calculations of the vapour pressure equation for ^{4}He below 2 K using eq. (4.1) were made by J.E. van Dijk et al.[10] for different values of L_0. Best available experimental data for the heat capacity and the density of the liquid and an extrapolation of experimental values for B and C above 2.6 K were used. With these data (and values for m, k, h, and R) the calculations were made exact. Once L_0 has been chosen, the uncertainties in the calculated vapour pressures depend mainly on the uncertainties in the data for the heat capacity of the liquid and the virial coefficients of the gas. From an estimate of these uncertainties it may be expected that the uncertainties in the calculated vapour pressure relation are less than 0.1 mK below 1.3 K, about 0.1 mK at 1.5 K, 0.2 mK at 1.8 K and 0.4 mK at 2 K (see ref. 10 and also Table 1 of ref. 7) if a value of L_0 has been adapted.

4.1.3. *Vapour pressures of liquid ^{3}He.*

The vapour pressure equation which defined the 1962 ^{3}He Scale[5] was between 0.9 K and 3.3 K derived from comparisons of ^{3}He and ^{4}He vapour pressures[2] and the 1958 ^{4}He scale. Thus, within the accuracy of these vapour pressure comparisons, the scales should be equal in this temperature range.

At the time that the 1962 scale was derived, vapour pressure measurements on a temperature scale which was equal to, or could be related to, the thermodynamic temperature were not available below 0.9 K[3]. Therefore, a thermodynamic vapour pressure equation was used for the calculation of vapour pressures below 0.9 K. It was not practical to use eq. (4.1) because, in the case of ^{3}He, the entropy of the liquid phase is appreciable even at 0.5 K and uncertainties in experimental heat capacities of the liquid below 0.5 K would introduce appreciable uncertainties in the calculated vapour pressures at higher temperatures. For this reason, the vapour pressure equation was written in the form[3]

$$\ln p = i - \frac{a}{RT} - \frac{b}{R} + \frac{5}{2} \ln T$$

$$- \frac{1}{RT} \int_{T_m}^{T} dT' \int_{T_m}^{T'} \frac{C_L}{T''} dT'' + \frac{1}{RT} \int_0^P V_L dp + \varepsilon \qquad (4.3)$$

where $a = L_0 - \int_0^{T_m} C_L \, dT$

and $b = S_L(T_m)$.

C_L is the molar heat capacity of the liquid at saturation. T_m can be chosen arbitrarily (for $T_m = 0$ eq. (4.3) is identical with eq. (4.1)). In the derivation of the 1962 scale the quantities a and b were derived by fitting eq. (4.3) to the experimental vapour pressure data (on T_{58}) between 0.9 K and 2 K. Eq. (4.3) was then used for deriving vapour pressures below 0.9 K. It will be clear that the advantage of using eq. (4.3) instead of eq. (4.1) is that experimental heat capacity data for the liquid phase are required only in the temperature range where the equation is used; the disadvantage is that two adaptable parameters occur (a and b) in stead of one (L_0).

New measurements of ^{3}He vapour pressures vs temperature between 3.2 K and 0.5 K were made by Rusby and Swenson[7)] at NPL using the NPL-75 and T_{X1}-temperature scales.

4.1.4. *Summary of contents.*

The vapour pressure measurements of ^{4}He and ^{3}He below 2 K presented in this chapter were set up for two reasons. Firstly, vapour pressure measurements of ^{3}He and ^{4}He form the most reliable way of realizing a temperature scale (e.g. the EPT-76) between

0.5 K and 4.2 K. Secondly, in the experiments of Rusby and Swenson vapour pressures of ^{4}He (below 1.2 K) and ^{3}He (below 1 K) vs T_{X1} were found which were inconsistent with thermodynamically calculated vapour pressure equations. Also, a preliminary analysis of the ^{4}He vapour pressure vs temperature data near 1.5 K suggested an irregularity with respect to T_{58} 11). Dr. R.L. Rusby came to our laboratory for one month at the end of 1976. It was planned to make during that time new ^{4}He and ^{3}He vapour pressure measurements below 2 K, using for the temperature measurements three RhFe-thermometers which Rusby brought with him and which he had calibrated on the NPL-75 and T_{X1}-scales. Because it turned out to be impossible in the available time to install a new cryostat which was under construction (the cryostat described in section 2.3.2b), an existing apparatus (the cryostat described in section 2.3.2a) was adapted for vapour pressure measurements down to 0.5 K. The experiments with this apparatus and a discussion of the ^{4}He vapour pressure data are presented in section 4.2.

In section 4.3 ^{3}He vapour pressure measurements in 1979 with the new cryostat are described and a discussion of the ^{3}He vapour pressure relation below 2 K is given. Concluding remarks are given in section 4.4.

4.2. VAPOUR PRESSURE MEASUREMENTS OF LIQUID ^{4}He AND ^{3}He BELOW 2 K WITH THE FIRST CRYOSTAT.

4.2.1. *Experimental arrangement.*

The combination of a vapour pressure thermometer and a small ^{3}He cryostat sketched in Fig. 4.1 was inserted in the apparatus described in section 2.3.2a (see Fig. 2.4). The copper reservoir of the vapour pressure thermometer Q fitted in the central hole of the copper block. The vapour pressure sensing tube consisted at its lower end of the stainless steel capillary N with an inner diameter of 1.5 mm and, above W, of the stainless steel tube A with an inner diameter of 7 mm.

The copper ^{3}He cryostat LL' was of the same outer diameter as the reservoir Q and fitted in the central holes of the copper block and of the shield (so that ^{3}He cryostat, block and shield were thermally connected). The pumping tube of the ^{3}He cryostat consisted of the stainless steel tubes X and B with inner diameters of 9 and 16 mm, respectively. At the flange D the insert was sealed to the vacuum can with an indium O-ring. In order to prevent the occurrence of a cold spot where the vapour pressure sensing tube passed the ^{3}He cryostat, this tube (N) was isolated from the ^{3}He cryostat by the space M, between N and the inner wall of the ^{3}He cryostat, which was evacuated together with the can. The ^{3}He cryostat could be pumped to about 0.5 K with a rotatory pump with a capacity of 4 m^3 per hour. Constant temperatures were obtained by manual adjustment of valves in the pumping line.

For ^{3}He vapour pressures higher than

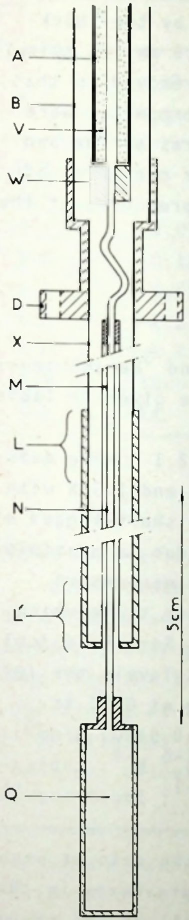

Fig. 4.1. The insert with the ^{3}He cryostat and vapour pressure thermometer used in the first cryostat (Fig. 2.4).

about 20 Torr* (T > 1.2 K) oscillations occurred in the gas column in the 7 mm diameter vapour pressure sensing tube A. For this reason, temperatures above 1.2 K were measured in a separate series with this tube partially filled up by the thick-walled glass tube V with an inner diameter of 2.5 mm. In this series of measurements the vacuum can had been removed so that the copper block and the ^{3}He vapour pressure thermometer were directly immersed in the bath and vapour pressures of ^{3}He and ^{4}He could be measured simultaneously: ^{3}He vapour pressures at the vapour pressure thermometer and ^{4}He vapour pressures at the bath.

4.2.2. *Procedure of measurement.*

For orientation, vapour pressures of ^{4}He and ^{3}He and sensitivities of the vapour pressure thermometers are given in Table 4.1.

Vapour pressures of ^{3}He between 1.2 K and 2.1 K were measured with a mercury manometer and between 0.5 K and 1.5 K with oil manometers (for pressures below 10 cm oil a short-legged oil manometer was used in order to minimize errors due to possible temperature differences between the legs of the manometer). Menisci were read with a cathetometer on an invar scale which was mounted vertically close to the manometers. For the calculation of pressures from measured mercury or oil levels the following quantities were used: density of mercury at 0 ^{0}C is 13.5951 g/cm^3; density of Octoil-S at 25 ^{0}C is 0.91903 g/cm^3; cubic expansion coefficient of mercury 182 x 10^{-6} K^{-1}; cubic expansion coefficient of Octoil-S 785 x 10^{-6} K^{-1}; local accele-

---

* Throughout this chapter the Torr is used as the unit of pressure, instead of the pascal, because vapour pressures in the 1958 ^{4}He Scale and the 1962 ^{3}He Scales, as well as the calibration of the diaphragm gauges which were used, are given in Torr.

Table 4.1. Vapour pressures of liquid ^{4}He and ^{3}He, and sensitivities of vapour pressure thermometers, below 2 K.

T_{58}, T_{62}	^{4}He			^{3}He		
K	p Torr	$\frac{dp}{dT}$ Torr/mK	$\frac{1}{p}\frac{dp}{dT}$ mK^{-1}	p Torr	$\frac{dp}{dT}$ Torr/mK	$\frac{1}{p}\frac{dp}{dT}$ mK^{-1}
0.5				0.16	0.0023	0.014
0.6				0.54	0.0058	0.011
0.8	0.011	0.00016	0.014	2.9	0.019	0.007
1.0	0.12	0.0012	0.010	8.8	0.042	0.0047
1.2	0.63	0.0044	0.007	20	0.073	0.0036
1.4	2.2	0.012	0.0054	39	0.11	0.0029
1.6	5.7	0.025	0.0043	65	0.16	0.0024
1.8	12.5	0.044	0.0035	103	0.21	0.0021
2.0	24	0.070	0.0029	151	0.27	0.0018

ration of gravity 9.8126 m s^{-2} *.

Corrections were applied for the weight of the ^{3}He gas in the pressure sensing tube (aerostatic head correction) and the thermomolecular pressure effect. The first correction was estimated from the temperatures of the pressure sensing tube at the reservoir Q, at the flange D (bath temperature) and at the top of the cryostat (room temperature). The thermomolecular pressure effect was calculated using the Weber-Keesom-Schmidt equation 12);

---

* We wish to thank Mr. G.L. Strang van Hees, from the Technical University in Delft, who determined this value of the acceleration of gravity in our laboratory in 1973.

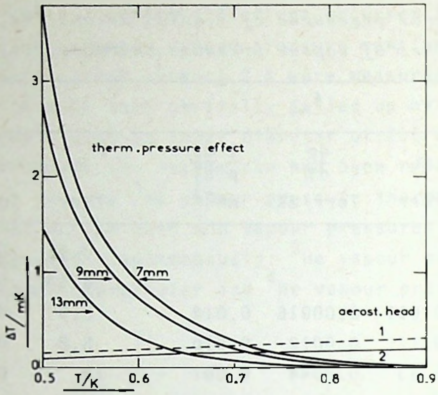

Fig. 4.2. Corrections applied to temperatures derived from ^{3}He vapour pressures for the aerostatic head in the first and second apparatus (curves marked 1 and 2) and for the thermomolecular pressure effect for three tube diameters.

only the pressure difference over the upper part (A) of the vapour pressure sensing tube had to be taken into account since the lower part (L) was always below 4 K. The estimated aerostatic head correction and the calculated thermomolecular pressure effect for the 7 mm diameter tube A are shown in Fig. 4.2.

The ^{3}He gas used for the vapour pressure measurements was taken from the laboratory stock; a mass spectrometrical analysis made at the Laboratory for Isotope Geology* in Amsterdam

---

* We thank Dr.E.E.Hebeda from this laboratory for making the analysis.

gave a ^{4}He content of 0.03 volume percent. The correction to be applied for this ^{4}He impurity varied from 0.15 mK at 2.1 K to less than 0.05 mK at 0.5 K$^{4)}$.

The ^{4}He vapour pressures between 1.2 K and 2.1 K were measured with two separate oil manometers (a normal one and a short one for the low pressures) and, below a pressure of 10 Torr, also with a diaphragm pressure gauge, type Baratron 145 BH·(MKS Instruments Inc., Burlington, Mass., U.S.A.), range 10 Torr, which was calibrated against a pressure balance (see section 4.3). The pressures were measured at the liquid helium bath by means of stainless steel tubes of 11.5 mm inner diameter (for temperatures between 1.2 K and 1.4 K) and of 8.5 mm inner diameter (for temperatures between 1.4 K and 2.1 K) which were inserted in the cryostat so that their lower ends were about 10 cm above the liquid surface. The corrections for the thermomolecular pressure effect was 0.4 mK at the lowest pressure (1.2 K) for the 11.5 mm inner diameter tube; the estimated aerostatic head correction was 0.2 mK at 2.1 K and less than 0.1 mK at 1.2 K.

In all measurements, temperatures T_{X1} were measured with two RhFe-thermometers which were in the copper block (thermometers nos. 1 and 2, see Chapter 2).

4.2.3. *Experimental results for ^{4}He.*

Experimental results, expressed as $T_{X1} - T_{58}$, T_{58} being derived from the measured vapour pressures and T_{X1} from the RhFe-thermometers, are shown in Fig. 4.3.

At temperatures between 1.7 K and 1.3 K the points referring to vapour pressures measured with the oil manometer differ by 0.1 mK to 0.3 mK from those for the diaphragm gauge. In view of the sensitivity of the oil manometer, which is only 0.05 mm oil/0.1 mK at 1.7 K and 0.01 mm oil/ mK at 1.3 K, the points measured

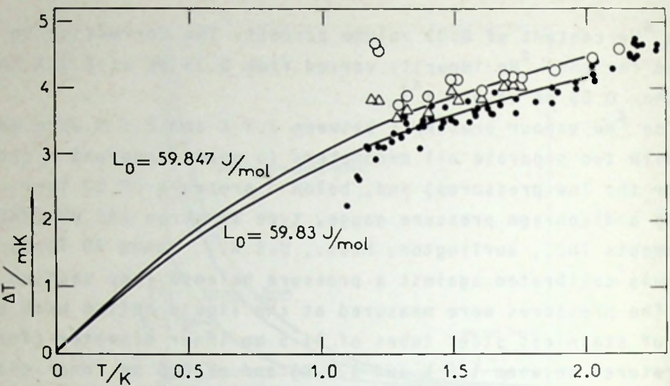

Fig. 4.3. Differences $T_{X1}-T_{58}$. T_{X1} *was derived from RhFe-thermometers which were calibrated on the* T_{X1}*-scale,* T_{58} *from* ^{4}*He vapour pressure measurements.*

- △ *present data, diaphragm pressure gauge*
- ○ *present data, oil manometer*
- • *data from Rusby and Swenson*[7)].
- ——— *thermodynamic vapour pressure equations for two values of* L_o.

with the diaphragm gauge should be given the highest weight. For the same reason the oil manometer point at 1.2 K, which is 0.8 mK (or 0.05 mm oil) above the diaphragm gauge point may be neglected.

4.2.4. *Discussion of* ^{4}*He vapour pressures.*

In Fig. 4.3 also the experimental points $T_{X1}-T_{58}$ of Rusby and Swenson[7)] are given. The differences between these results and the present data are 0.2 mK to 0.3 mK between 2.1 K and 1.3 K, which is only slightly larger than the scatter of the data points in each of the two experiments.

The curves marked L_0 = 59.847 J/mol and L_0 = 59.830 J/mol indicate vapour pressures calculated using eq. (4.1), for these two values of L_0. The curves were derived from Van Dijk's calculations (see section 4.1.3) by slightly adjusting the value of L_0. The curve L_0 = 59.830 J/mol was chosen by Rusby and Swenson as the best fit to their data below 2 K. The present data would indicate the slightly larger value L_0 = 59.847 J/mol.

The consistency of the T_{X1}-scale with the thermodynamically calculated vapour pressure equations is excellent for the data of Rusby and Swenson as well as for the present data above 1.2 K and 1.3 K, respectively. The systematic deviation below 1.2 K between the experimental data of Rusby and Swenson and the calculated equation was attributed by these authors to a possible systematic error in their measurements of low vapour pressures (see section 4.3.3). The present data could not be extended below 1.2 K because the cryostat could not be pumped to a lower temperature.

4.2.5. *Experimental results for* ^{3}*He*.

The experimental results for ^{3}He are shown in Fig. 4.4. At temperatures between 1.2 K and 1.5 K the mercury manometer points are probably less accurate, since the sensitivity of the mercury manometer is only 0.007 mm Hg/0.1 mK at 1.2 K and 0.013 mm Hg/0.1 mK at 1.5 K.

The dashed line in Fig. 4.4 indicates an average of the data of Rusby and Swenson$^{7)}$. A discussion of the results, together with those obtained with the second cryostat, is given in section 4.3.

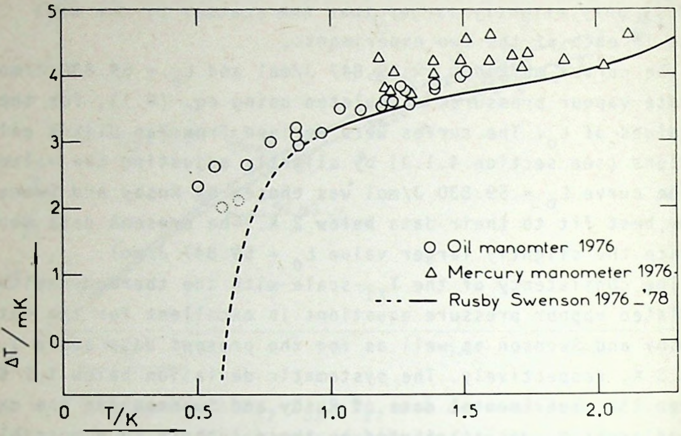

Fig. 4.4. Differences $T_{X1}-T_{62}$*. (*T_{X1} *from RhFe-thermometers calibrated on the* T_{X1}*-scale;* T_{62} *from* ^{3}He *vapour pressures).*

4.3. VAPOUR PRESSURE MEASUREMENTS OF LIQUID ^{3}He BETWEEN 0.5 K AND 1.8 K WITH THE SECOND CRYOSTAT.

4.3.1. *The cryostat.*

The cryostat has been described in section 2.3.2b (Fig. 2.5). The insert for the vapour pressure measurements is shown in Fig. 4.5. The vapour pressure sensing tube consisted of the tube B (inner diameter 9 mm), and the capillaries F (inner diameter 2.5 mm) and I (inner diameter 1.8 mm), all made of stainless steel. At the copper connecting pieces D and H, each of which contained a radiation trap, the vapour pressure tube was thermally anchored to the liquid helium bath and to the copper plate G, respectively. Plate G contained the small ^{4}He cryostat indicated by D in Fig. 2.5. (The vapour pressure tube was not connected to the ^{3}He cryostat on the shield (Fig. 2.5) in order to avoid a cold spot). At the flange J the vapour pressure tube was sealed with an indium O-ring to the copper block.

Tube B was surrounded by the stainless steel tube A (inner diameter 16 mm). The space between A and B could be evacuated. The assembly was sealed together and to the flange E of the vacuum can with two indium O-rings.

4.3.2. *Vapour pressure measurements.*

It was decided to abandon mercury and oil manometers altogether in the new measurements and to use the Baratron capacitive diaphragm gauge system. Three different diaphragms were used with ranges of 100 Torr, 10 Torr and 1 Torr, type Baratron 145 BH (10 and 100 Torr ranges) and 310 BH (1 Torr range). Pressure gauges of this type were used before in gas thermometry by Berry$^{13)}$ and for vapour pressure measurements by Rusby and Swenson$^{7)}$.

The sensitivity of these diaphragm

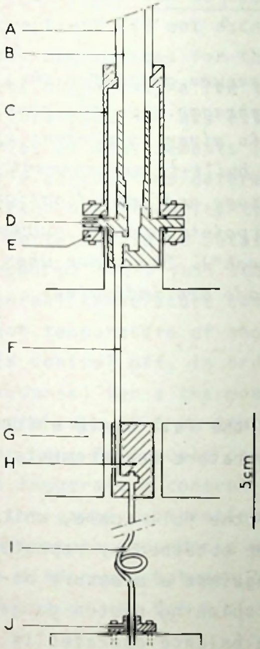

Fig. 4.5. The insert for the vapour pressure sensing tube in the second cryostat (Fig. 2.5). (The temperature gradient along the tube B was measured with five chromel versus Au-0.03% Fe thermocouples).

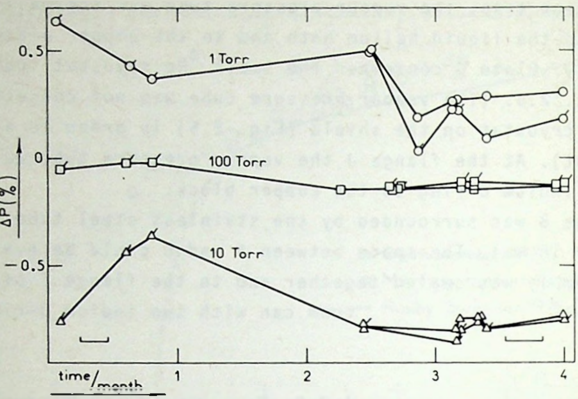

Fig. 4.6. Calibrations of the diaphragm pressure gauge for the 100 Torr, 10 Torr and 1 Torr diaphragms. Δp, the correction at full scale pressure, is given in percent of the full scale. (The gauge has a built-in arrangement for checking the internal bridge voltage and amplification factor ("system check"). The dashed points were not corrected for changes in the "system check"). The times when the pressure measurements were made are indicated.

manometers was in practice about 10 ppm of the full scale which was sufficient for a precision in the temperature measurements of 5 μK.

The accuracy of the order of 0.01 % of the full scale, which was required (see Table 4.1), could only be obtained by repeated calibrations. The calibrations were made against a pressure balance (Bell and Howell Type b-201, a free spinning piston gauge). The minimum pressure at which the pressure balance operates is 16

Torr. The calibrations at lower pressures were made by having a reference pressure of about 16 Torr at the reference side of the diaphragm gauge. The reference pressure was kept constant by using an 8 dm^3 thermostated volume connected to the diaphragm gauge.

Results of the calibrations for the three diaphragms are shown in Figs. 4.6 and 4.7. Fig. 4.6 shows the change in the correction at full scale pressures, during five months. Figs. 4.7a, b and c show the non-linearity of the three diaphragms. In these figures also the accuracies which are required for a 0.1 mK accuracy in the temperatures derived from the measured vapour pressures are indicated. It can be seen from the figures that the non-linearity corrections did not exceed 0.04 % of the full scale. The full scale corrections for the 10 Torr and 1 Torr diaphragms sometimes changed by a few tenths of a percent between successive calibrations. It was estimated that by taking for each of the two series of measurements (see Fig. 4.6) the average of the full scale corrections determined before and after the series and an average non-linearity correction, an accuracy equivalent to 0.1 mK or better could be obtained when the diaphragms were not used below 15 percent of their full scales. The diaphragms can be used with an internal temperature control, which keeps the diaphragm at a constant temperature of about 50 ^{o}C, on or off. They were used with this control off, in order to avoid corrections (at the lowest pressures) for a thermomolecular pressure difference over the entrance tube of 6 mm inner diameter of the diaphragm heads. However, one full scale calibration was made for each diaphragm with the temperature control on, in order to determine the dependence of the gauge calibration on the room temperature. The maximum temperature dependence was found for the 10 Torr diaphragm where the difference between calibration with the temperature control off and on was 0.6 % of the full scale.

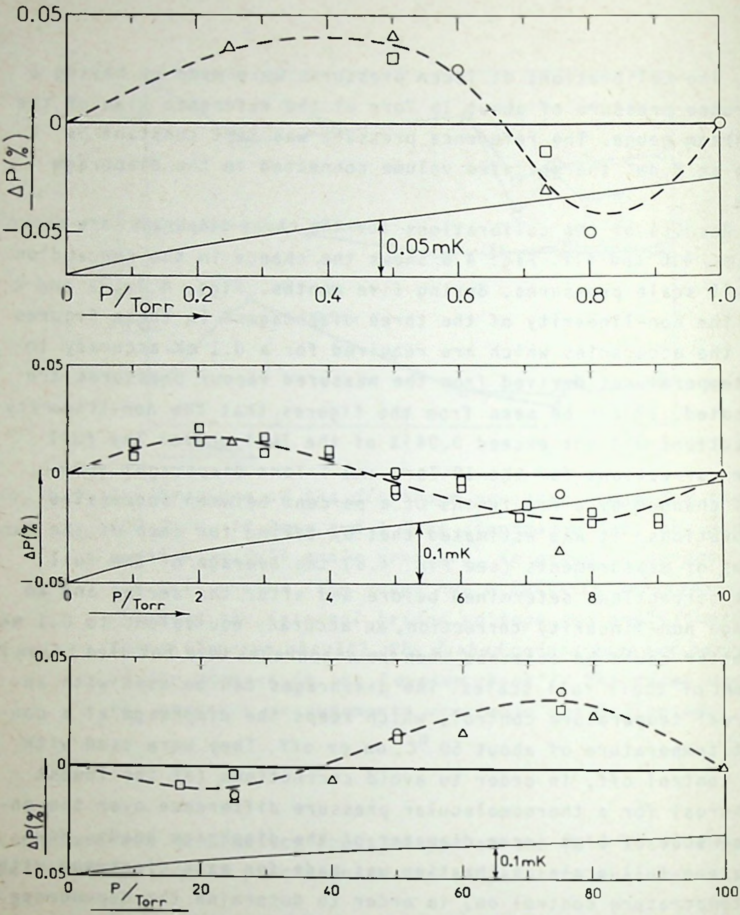

Fig. 4.7. Non-linearity of the diaphragm pressure gauges. Δp is the non-linearity correction in percent of the full scale of each diaphragm. The accuracy required for a 0.1 mK accuracy in the temperatures derived from ^{3}He vapour pressures is indicated.

4.3.3. *Experimental results and discussion.*

The experimental results expressed as T_{X1}-T_{62}, with T_{X1} derived from temperature measurements with RhFe-thermometers 2 and 4 and T_{62} from the measured vapour pressures, are shown in Fig. 4.8 (measurements of June 1979). For comparison, also the experimental points of Rusby and Swenson$^{7)}$ are plotted.

A steep decline in T_{X1}-T_{62} near 0.5 K, as measured by Rusby and Swenson, if correct, would almost certainly mean that T_{X1} is in error, since T_{62} was based on a thermodynamic equation. Rusby and Swenson attributed the decline to errors in the low pressure vapour pressure measurements, in fact, they suggested that a flow into the cryostat of gaseous impurities evolved from copper tubing used in the vapour pressure line at room temperature might be responsible for the effect.

In the present experiment, the vapour pressure line between the cryostat and the diaphragm gauge consisted of a flexible stainless steel tube (150 cm long, 6 mm inner diameter), a copper tube (50 cm, 4 mm diameter) and 3 Saunders valves. In order to check whether a further increase in the diameter of the vapour pressure sensing line had any effect, the measurements were repeated with a vapour pressure sensing tube in the cryostat of an inner diameter of 13 mm (instead of 9 mm), and a shorter and wider stainless steel flexible tube (about 30 cm long and 12 mm in diameter) and Leyboldt bellows valves between the cryostat and the diaphragm gauge. The results obtained with this arrangement are also shown in Fig. 4.8 (measurements of October 1979).

By comparison of Figs. 4.4 and 4.8, it can be seen that the October 1979 data agreed between 0.5 K and 1.0 K within 0.2 mK with those obtained in 1976 with the first apparatus and the oil manometer (if the two lower points near 0.6 K in Fig. 4.4, which were considered to be less precise, were neglected). This very good agreement was somewhat surprising in view of the low sensitivity of the oil manometer (see Table 4.1) and the sizable correction for the thermomolecular pressure effect in the first appara-

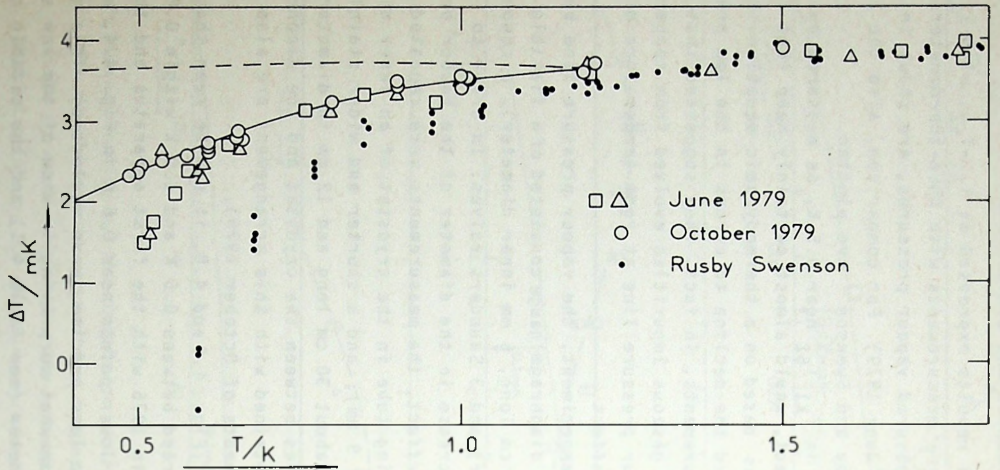

Fig. 4.8. Differences $T_{X1}-T_{62}$. T_{X1} derived from RhFe-thermometers calibrated on the T_{X1}-scale; T_{62} derived from measurements of ^{3}He vapour pressures. Measurements in October 1979 were made with a wider vapour pressure sensing line. Curves indicate calculated vapour pressure equations (see text).

tus (Fig. 4.2, 7 mm tube). Also, in the 1976 measurements no very special precautions were taken against degassing of the tubing connecting the cryostat to the oil manometer (a stainless steel flexible tube of 1 m length and 6 mm diameter and glass tubes of 2 m length and 8 mm diameter). It may be possible that degassing in the copper tubing, or in the diaphragm gauge itself, was responsible for the deviation of the June 1979 data from those of October.

It can be seen in Fig. 4.8 that the experimental data of October 1979 follow very closely the shape of calculated vapour pressure equations (see next section), also between 0.6 K and 0.5 K in which range the vapour pressure decreases by a factor of three; the October data do not show the systematic deviations from the calculated equations as are apparent for the earlier data.

As a conclusion, we estimate that the October 1979 data represent ^{3}He vapour pressures vs temperature on the T_{X1}-scale with an uncertainty of 0.2 mK above 1 K, increasing to 0.5 mK at 0.5 K.

4.3.4. *Thermodynamic calculations of the vapour pressure equation.*

Thermodynamic calculations of the vapour pressure equation of ^{3}He between 0.5 K and 1 K were made using the same equation as was used by Sydoriak et al.$^{3)}$ in the derivation of the 1962 ^{3}He Scale (eq. (4.3)). Firstly, the effect of the change in the temperature scale above 1 K was calculated. If all other input data in eq. (4.3) are left unchanged, only the constants a and b will change. Thus,

$$\Delta T(T) = \left(\frac{\Delta a}{RT} + \frac{\Delta b}{R}\right) dT/d\ln p \tag{4.4}$$

Δa and Δb were derived by fitting eq. (4.4) to the T_{X1}-T_{62} data between 1 K and 1.8 K. The result is given by the full drawn curve in Fig. 4.8.

In view of the uncertainties in the data for the heat capacity of the liquid and the virial coefficients of the gas which are used in eq. (4.3), and the uncertainties in T_{X1} (see below), the nearly exact agreement between the calculated curve and the experimental points measured with the wider vapour pressure line is fortuitous. This is illustrated by a second calculation, in which values of the second virial coefficient according to Boyd et al.[14)] were used instead of the values used by Sydoriak et al. The result of this calculation is given by the dashed line in Fig. 4.8. It was estimated in 1962 that the uncertainty in the data for the heat capacity of the liquid introduced an uncertainty in the calculated vapour pressure equation of about 0.4 mK at 0.5 K. The total uncertainty in the thermodynamic extrapolation of the vapour pressure equation is about 1.5 mK at 0.5 K.

4.4. CONCLUSION.

Measurements of ^{4}He vapour pressures were made between 1.2 K and 2.1 K and of ^{3}He vapour pressures between 0.5 K and 1.8 K. The CMN magnetic T_{X1}-scale of NPL, carried on rhodium-iron resistance thermometers was used as the temperature scale.

The ^{4}He vapour pressure data agree with those of Rusby and Swenson within 0.3 mK; both sets of data show that the T_{X1}-scale is consistent with the calculated vapour pressure equation for ^{4}He in this range.

The ^{3}He vapour pressure data between 1.1 K and 1.8 K agree with those of Rusby and Swenson within 0.2 mK, which shows the excellent reproducibilities of the vapour pressure measurements in this range and of the temperature scale carried by the RhFe-thermometers. The results below 1.1 K suggest that ^{3}He vapour pressures were measured down to 0.5 K with the same reproducibility, but a further study with different vapour pressure sensing tubes should verify this. No inconsistencies have been found between the T_{X1}-scale and the thermodynamic vapour pressure equation for ^{3}He.

Rusby and Swenson estimated the uncertainty (with respect to the thermodynamic temperature) in the T_{X1}-scale to be 0.5 mK above 1.1 K increasing to 1.5 mK at about 0.5 K. The larger uncertainty at 0.5 K was assumed because a second single crystal of cerium magnesium nitrate yielded a (second) magnetic temperature scale (denoted T_{X2}-scale), which was identical with the T_{X1}-scale at temperatures above 1.3 K but differed from this scale by 0.7 mK at 0.7 K (the lowest temperature at which the T_{X2}-scale was determined), T_{X2} being the highest. Rusby and Swenson have chosen to use the T_{X1}-scale on the ground that it originated from a more successful experiment[7)]. Also, a determination of the superconductive transition point of Cd with a noise thermometer by Soulen gave the value 0.5290 K ± 0.0006 K[7,9)] which is in good agreement with the value on the T_{X1}-scale (see Chapter 3). Because the thermodynamic extrapolation of the ^{3}He vapour pressure equation has also an uncertainty of 1.5 mK at 0.5 K, ^{3}He vapour pressure measurements cannot resolve whether T_{X1} or T_{X2} is correct.

At present, the full drawn curve in Fig. 4.8 seems to be the best choice as a basis of a new ^{3}He vapour pressure equation between 0.5 K and 1 K.

REFERENCES.

1) F.G. Brickwedde, H. van Dijk, M. Durieux, J.R. Clement and J.K. Logan, J. Res. Natl. Bur. Stand. (Wash.) 64A(1960)1.

2) S.G. Sydoriak and R.H. Sherman, J. Res. Natl. Bur. Stand. (Wash.) 68A(1964)547.

3) S.G. Sydoriak, T.R. Roberts and R.H. Sherman, J. Res. Natl. Bur. Stand. (Wash.) 68A(1964)559.

4) T.R. Roberts, R.H. Sherman and S.G. Sydoriak, J. Res. Natl. Bur. Stand. (Wash.) 68A(1964)567.

5) R.H. Sherman, S.G. Sydoriak, T.R. Roberts, J. Res. Natl. Bur. Stand. (Wash.) 68A(1964)579.

6) The 1976 Provisional 0.5 K to 30 K Temperature Scale, Metrologia 15(1979)65.

7) R.L. Rusby and C.A. Swenson, Metrologia, to be published.

8) H.-H. Klein, G. Klempt and L. Storm, Metrologia 15(1979)143.

9) M. Durieux, D.N. Astrov, W.R.G. Kemp and C.A. Swenson, Metrologia 15(1979)57.

10) J.E. van Dijk, H. ter Harmsel and M. Durieux, Comité Consultatif de Thermométrie, 11e Session(1976) p. 83.

11) R.L. Rusby and C.A. Swenson, private communication (1976).

12) S. Weber, W.H. Keesom and G. Schmidt, Commun. Kamerlingh Onnes Lab., Leiden, No. 246a(1936).

13) K.H. Berry, Metrologia 15(1979)89.

14) M.E. Boyd, S.Y. Larsen and J.E. Kilpatrick, J. Chem. Phys. 50(1969)4034.

APPENDIX.

THE 1976 PROVISIONAL 0.5 K TO 30 K TEMPERATURE SCALE.*†

Evidence has been increasingly available that the currently used helium vapour pressure scales[1,2] and the lower temperature end of the International Practical Temperature Scale of 1968 (IPTS-68)[3] depart substantially from thermodynamic temperature and, moreover, are not consistent with each other. These problems with the current international practical temperature scales have been recognized and discussed by the Comité Consultatif de Thermométrie (CCT). As a result, the CCT proposed to the Comité International des Poids et Mesures (CIPM) in 1976 that a new provisional practical temperature scale be recommended for international use between 0.5 K and 30 K until a new International Prac-

---

*This document is the translation of the official French text of the "Echelle Provisoire de Température de 1976 entre 0,5 K et 30 K".

†This document has been prepared by the members of the Working Group 4 of the Comité Consultatif de Thermométrie: M. Durieux, Chairman, Kamerlingh Onnes Laboratorium (Netherlands), W.R.G. Kemp, National Measurement Laboratory (Australia), C.A. Swenson, Iowa State University (U.S.A.), D.N. Astrov, Physico-Technical and Radio-Technical Measurements Institute (U.S.S.R.). The French version can be obtained from the Bureau International des Poids et Mesures F-92310 Sèvres, France.

tical Temperature Scale can be adopted4). The CIPM authorized the CCT to promulgate the "1976 Provisional 0.5 K to 30 K Temperature Scale"* when it became available in its final form5,6). This scale is described in the following.

The objectives in deriving the EPT-76 were that it should be thermodynamically smooth**, that it should be continuous with the IPTS-68 at 27.1 K, and that it should agree with thermodynamic temperature T as closely as these two conditions allow.

The EPT-76 is defined in terms of the reference points given in Table 1. They have been derived as far as possible to conform with the objectives laid out above, using recent results in thermometry. A realization of the EPT-76 is obtained by interpolation between the reference points, but in contrast with the International Practical Temperature Scale of 1968, a variety of methods of interpolation is allowed, and it is also permissible to obtain values of T_{76} from certain existing laboratory scales.

Approved methods of realizing of the EPT-76 over its full range or part of its range are:

(a) by use of a thermodynamic interpolating instrument, such as a

---

*Temperatures on this scale are denoted T_{76} and EPT-76 is used as an abbreviation for the French title: "Echelle Provisoire de Température de 1976 entre 0,5 K et 30 K".

**The term "thermodynamically smooth" as used here implies that the second and higher derivatives of T_{76} with respect to T should be small. In a heat capacity measurement, for instance, the structure which appears due to a lack of smoothness of the temperature scale should be less than 0.1%. This same smoothness criterion should apply to thermometer calibrations which span the temperature region near 27.1 K where EPT-76 and the IPTS-68 join.

Table 1. Reference points of the 1976 provisional 0.5 K to 30 K temperature scale (EPT-76).

Reference point	Assigned temperature T_{76} (K)
Superconducting transition point of cadmium	0.519
Superconducting transition point of zinc	0.851
Superconducting transition point of aluminium	1.1796
Superconducting transition point of indium	3.4145
Boiling point of ^{4}He[a]	4.2221
Superconducting transition point of lead	7.1999
Triple point of equilibrium hydrogen[b]	13.8044
Boiling point of equilibrium hydrogen at a pressure of 33.330.6 pascal (25/76 standard atmosphere)[b]	17.0373
Boiling point of equilibrium hydrogen[a,b]	20.2734
Triple point of neon[c]	24.5591
Boiling point of neon[a,b,c]	27.102

Superconducting transition point: the transition temperature between the superconducting and the normal state in zero magnetic field as given by NBS-SRM 767.

[a] Boiling point under a pressure p_o = 101325 Pa(1 standard atmosphere).

[b] These are the four lower defining points of the IPTS-68. (Note: the values of temperature assigned to these points in EPT-76 are not the same as those assigned in IPTS-68).

The term equilibrium hydrogen means here that the hydrogen should have its equilibrium ortho-para composition at the relevant temperature.

[c] The two neon points are for neon with the natural isotopic composition of 2.7 mmol of ^{21}Ne and 92 mmol of ^{22}Ne per 0.905 mol of ^{20}Ne[3].

gas thermometer or a magnetic thermometer, calibrated at one or more reference points of Table 1;

(b) for temperatures above 13.81 K by use of the IPTS-68 together

Table 2. Differences between the EPT-76 (T_{76}) and the IPTS-68 (T_{68}).

T_{68} (K)	$T_{68}-T_{76}$ (mK)	T_{68} (K)	$T_{68}-T_{76}$ (mK)	T_{68} (K)	$T_{68}-T_{76}$ (mK)
13.81	5.6	19.0	7.4	24.5	2.1
14.0	4.6	19.5	7.3	25.0	1.6
14.5	3.0	20.0	6.9	25.5	1.1
15.0	2.0	20.5	6.4	26.0	0.7
15.5	2.2	21.0	5.8	26.5	0.3
16.0	2.6	21.5	5.3	27.0	0.0
16.5	3.6	22.0	4.8	27.1	0.0
17.0	4.6	22.5	4.2	28.0	0.0
17.5	5.6	23.0	3.7	29.0	0.0
18.0	6.5	23.5	3.2	30.0	0.0
18.5	7.2	24.0	2.7		

with the differences given in Table 2;

(c) for temperatures below 5 K by use of the vapour pressure scale of ^{4}He, the 1958 ^{4}He scale, or of ^{3}He, the 1962 ^{3}He scale, together with the differences given in Table 3;

(d) by use of any of the laboratory scales for which the differences from the EPT-76 are given in Tables 4, 5 and 6.

The different procedures for realizing the EPT-76 may, through a lack of internal consistency, introduce slight ambiguities between realizations. These would not be acceptable for

Table 3. Differences between the EPT-76 (T_{76}) and the helium vapour pressure scales (the 1958 ^{4}He scale (T_{58}) and the 1962 ^{3}He scale (T_{62})).

T_{vp} (K)	$T_{vp}-T_{76}$ (mK)	T_{vp} (K)	$T_{vp}-T_{76}$ (mK)	T_{58} (K)	$T_{58}-T_{76}$ (mK)
0.5	-1.9	1.8	-3.9	3.2	-6.6
0.6	-2.1	2.0	-4.1	3.4	-6.8
0.8	-2.5	2.2	-4.4	3.6	-7.0
1.0	-2.9	2.4	-4.9	3.8	-7.0
1.2	-3.2	2.6	-5.4	4.0	-7.1
1.4	-3.5	2.8	-5.9	4.2	-7.1
1.6	-3.7	3.0	-6.3	4.5	-7.1
		3.2	-6.6	5.0	-7.1

T_{vp} means an average of T_{62} and T_{58} up to 3.2 K.

Table 4. Differences between the NBS provisional temperature scale 2-20 K(1965)($T_{NBS\ 2\text{-}20}$) and the EPT-76 (T_{76}).

$T_{NBS\ 2\text{-}20}$[a] (K)	$T_{NBS\ 2\text{-}20}-T_{76}$ (mK)	$T_{NBS\ 2\text{-}20}$[a] (K)	$T_{NBS\ 2\text{-}20}-T_{76}$ (mK)
2.3	2.1	11.0	-1.0
2.8	-1.1	12.0	0.2
3.2	1.0	13.0	-1.8
4.2	2.5	14.0	-2.2
5.0	3.0	15.0	-0.6
6.0	1.7	16.0	0.9
7.0	4.8	17.0	1.7
8.0	2.2	18.0	-0.9
9.0	-1.5	19.0	-0.2
10.0	-2.1	20.0	-0.8(+7[b])

[a] These temperatures are very close to the actual temperatures (acoustic points) at which the NBS 2-20 scale is defined.

[b] With $T_{NBS\ 2\text{-}20}$ as given in early NBS calibrations.

Table 5. Differences between the magnetic scales of ISU, KOL, NML, PRMI, NPL, the NPL-75 gas thermometer scale and the EPT-76.
Coefficients are given for relationship

$$T_i - T_{76} = a + bT_i + cT_i^2 + d/T_i,$$

where T_i represents temperatures on the various laboratory scales.

Laboratory	Scale[a]	a $(10^{-3}K)$	b (10^{-3})	c $(10^{-3}/K)$	d $(10^{-3}K^2)$	Range (K)
ISU	$T_{XAc'}$	0	0	0.0025	0	1.1-30
KOL	$T_m(III)$	- 8.0	1.5	- 0.0413	8.3	2 -27
NML	T_{XNML}	- 1.5	0.41	- 0.0109	0	1.1-30
NML	T_{MAS}	- 1.5	0.49	- 0.0125	0	1.1-30
PRMI	T_{XPRMI}	0	0.51	- 0.0125	0	4.2-27
NPL	T_{X1}	0	0	0	0	0.5- 3.1
NPL	T_{NPL-75}	0	0	- 0.0056	0	2.6-27.1

[a] These scales are discussed in Ref. (9).

an International Practical Temperature Scale, but the advantages gained by the introduction of the EPT-76 as a working scale are considered to outweigh this disadvantage. The EPT-76 does not replace the IPTS-68 in the region of overlap, from 13.81 K to 30 K, but its use will be preferred in applications requiring smoothness with respect to thermodynamic temperature.

Tables 4, 5 and 6 contain references to scales which are discussed in the CCT-promoted comparisons of platinum resistance thermometers by Ward andCompton$^{7)}$ and germanium resistance thermometers by Besley and Kemp$^{8)}$. In particular, the latter authors'

Table 6. Differences between the NBS version of the IPTS-68 (T_{NBS-68}) and the EPT-76 (T_{76}).

T_{NBS-68} (K)	$T_{NBS-68}-T_{76}$ (mK)	T_{NBS-68} (K)	$T_{NBS-68}-T_{76}$ (mK)	T_{NBS-68} (K)	$T_{NBS-68}-T_{76}$ (mK)
13.8	1.7	19.0	4.9	24.5	2.9
14.0	1.8	19.5	4.9	25.0	2.5
14.5	2.1	20.0	4.9	25.5	2.1
15.0	2.0	20.5	4.8	26.0	1.7
15.5	2.2	21.0	4.7	26.5	1.3
16.0	2.4	21.5	4.6	27.0	1.1
16.5	3.0	22.0	4.5	27.5	1.1
17.0	3.5	22.5	4.2	28.0	1.1
17.5	3.9	23.0	4.0	29.0	1.1
18.0	4.4	23.5	3.7	30.0	1.0
18.5	4.8	24.0	3.4		

$T_{XAc'}$-scale is related to the EPT-76 by

$$T_{XAc'} - T_{76} = 2.5 \times 10^{-6}\ T^2/K.$$

Supplementary information on the background, derivation, realization and estimated thermodynamic accuracy of the EPT-76 is given elsewhere[9)].

REFERENCES.

1) Brickwedde, F.G., Van Dijk, H., Durieux, M., Clement, J.R., Logan, J.K.: J. Res. NBS 64A, 1(1960).

2) Sherman, R.H., Sydoriak, S.G., Roberts, T.R.: J. Res.: NBS 68A, 579(1964).

3) Comptes rendus des séances de la 15e Conférence Générale des Poids et Mesures, A 1, 1975 et Comité Consultatif de Thermométrie, 10e session, A 1, 1974. Metrologia 12, 7(1976).

4) Comité Consultatif de Thermométrie, 11e session, T 1, 1976.

5) Procès-Verbaux CIPM 44, 11(1976).

6) Terrien, J: Metrologia 13, 53(1977).

7) Compton, J.P., Ward, S.D.: Temperature Measurement 1975, Institute of Physics Conference Series No. 26, p. 91. London: Institute of Physics. Also to be published in Metrologia by Ward, S.D. and Compton, J.P.

8) Besley, L.M., Kemp, W.R.G.: Metrologia 13, 35(1977).

9) Durieux, M., Astrov, D.N., Kemp, W.R.G., Swenson, C.A.: Metrologia 15, 57-63(1979).

SAMENVATTING.

De totstandkoming in 1978 van een nieuwe internationale praktische temperatuurschaal, de "Echelle provisoire de température entre 0,5 K et 30 K" of "EPT-76", was het resultaat van een aantal experimenten in verschillende laboratoria. In dit proefschrift worden metingen beschreven die in het Kamerlingh Onnes Laboratorium zijn gedaan ter voorbereiding van de nieuwe schaal en om de schaal in het laboratorium te realiseren.

Ter inleiding worden in het eerste hoofdstuk de verschillen tussen de nieuwe temperatuurschaal en oudere internationale temperatuurschalen gegeven en wordt aangegeven hoe de nieuwe schaal is tot stand gekomen.

Weerstandsthermometers, met name germanium-weerstandsthermometers en rhodiumijzer-weerstandsthermometers, worden gebruikt om temperatuurschalen die in verschillende laboratoria zijn ontwikkeld vast te leggen en onderling te vergelijken. In hoofdstuk 2 van dit proefschrift wordt de apparatuur beschreven die voor de weerstandsmetingen en voor het onderling vergelijken van de thermometers wordt gebruikt. Uit metingen van de weerstanden van de thermometers met verschillende meetstromen blijkt, dat een nauwkeurigheid wordt bereikt die overeenkomt met 0,1 mK in de temperatuur. Aangetoond wordt dat met behulp van geijkte rhodiumijzerweerstandsthermometers een temperatuurschaal tussen 0,5 K en 30 K kan worden vastgesteld met een reproduceerbaarheid van 0,2 mK en dat een realisering van de EPT-76 uitgaande van een in 1969 in het laboratorium ontwikkelde magnetische temperatuurschaal, die sindsdien is vastgelegd met behulp van vier germanium-weerstandsthermometers, binnen 1 mK overeenstemt met realiseringen uitgaande van in andere instituten ontwikkelde schalen.

In de nieuwe temperatuurschaal worden de supergeleidende overgangspunten van lood, indium, aluminium, zink en cadmium als referentiepunten gebruikt. De overgangspunten worden gemeten aan

"standaard-referentiematerialen" die door het National Bureau of Standards in Washington ontwikkeld zijn. Uit een onderzoek, dat samen met het National Physical Laboratory in Teddington, Engeland, en het National Measurement Laboratory in Sydney is uitgevoerd, blijkt dat verschillen tot 1 mK optreden tussen de overgangstemperaturen gemeten aan verschillende specimen van de metalen en dat er voor zink en cadmium een duidelijke relatie is tussen de overgangstemperatuur en de breedte van de overgang. Deze metingen worden in het derde hoofdstuk beschreven.

In het vierde hoofdstuk worden metingen van de dampspanning van vloeibaar ^{4}He tussen 1,2 K en 2,1 K en van vloeibaar ^{3}He tussen 0,5 K en 1,8 K beschreven. Deze metingen zijn van belang omdat de dampspanningsmeting één van de meest nauwkeurige methodes is om een praktische temperatuurschaal te realiseren en omdat de dampspanningsrelaties thermodynamisch kunnen worden berekend, waardoor meting van de dampspanningsrelaties op een bepaalde temperatuurschaal een controle van deze temperatuurschaal geeft. De in het vierde hoofdstuk beschreven metingen vormen een aanvulling op soortgelijke metingen die in het National Physical Laboratory in Teddington waren verricht en waarbij bij lage dampspanningen tegenstrijdigheden tussen de experimentele en de thermodynamisch berekende dampspanningen werden gevonden. De metingen, waarin een reproduceerbaarheid in de dampspanningsmeting overeenkomende met 0,2 mK in de temperatuur werd bereikt, tonen aan dat de experimentele dampspanningsrelaties voor ^{4}He en ^{3}He in overeenstemming zijn met de thermodynamisch berekende. In het bijzonder de resultaten van ^{3}He beneden 1 K zijn van nut voor het afleiden van een nieuwe dampspanningsrelatie voor ^{3}He, die als praktische temperatuurschaal zal worden gebruikt.

STUDIEOVERZICHT.

Teneinde te voldoen aan het verzoek van de Faculteit der Wiskunde en Natuurwetenschappen volgt hier een beknopt overzicht van mijn studie.

Na in 1961 het eindexamen van de "Damanhour Secondary School" in Damanhour, Egypte, te hebben afgelegd, begon ik mijn studie in de Wis- en Natuurkunde aan de Universiteit van Alexandrië. In 1965 behaalde ik het diploma "Bachelor of Science (BSc)" met de aantekening "distinction, honours". Hierna was ik tot 1974 bij het Natuurkundig Practicum aan de Universiteit van Alexandrië werkzaam als wetenschappelijk assistent.

In 1970 verkreeg ik de "Master's Degree (MSc)" op een onderwerp uit de kernphysica, dat werd bestudeerd onder leiding van Prof. dr. Y.S. Selim.

In 1974 verkreeg ik van de Egyptische regering een beurs voor een studie op het gebied van de physica bij lage temperaturen, in welk gebied de Universiteit van Alexandrië in de toekomst werkzaam wil zijn. Vanaf die tijd heb ik in het Kamerlingh Onnes Laboratorium gewerkt op de afdeling Thermometrie, die onder leiding staat van Dr. M. Durieux en Dr. H. ter Harmsel.

Gedurende zeven maanden had ik een aanstelling als doctoraal assistent.

NAWOORD.

Bij de totstandkoming van dit proefschrift zijn velen betrokken geweest. In de eerste plaats wil ik de leden en oud-leden van de werkgroep Thermometrie noemen: Dr. M. Durieux, Dr. H. ter Harmsel, J. Mooibroek, Drs. J.E. van Dijk, Drs. C.L. Herbschleb, Drs. E.J. Kruithof, M.J. Betlem en Drs. P.P.M. Steur. Zij allen hebben hun onontbeerlijke steun op enthousiaste wijze gegeven in alle fasen van het onderzoek; de zorgvuldige voorbereiding van de metingen die vaak afhing van het werk van J. Mooibroek en de computerberekeningen door J.E. van Dijk noem ik als voorbeelden van hun medewerking.

Daarnaast wil ik noemen: J.A. van der Zeeuw, electronicus; A.J.J. Kuyt, G. Vis, J.P. Hemerik, A.J. Kamper, J. van der Waals, technici, die de apparatuur, of onderdelen daarvan, vervaardigden; B. Kret, P.J.M. Vreeburg, P. van Biemen, die het glastechnische deel van de apparatuur verzorgden, en J.D. Sprong, J.A. Th. van Schooten en J. de Vink, die de cryogene vloeistoffen verschaften. Hun voortdurende steun maakte de uitvoering van de metingen mogelijk.

Mijn erkentelijkheid gaat ook uit naar al diegenen van de administratieve, huishoudelijke, technische en wetenschappelijke staven van het Kamerlingh Onnes Laboratorium, die mij hebben geholpen; hun vriendschap heeft de tijd, die ik in het laboratorium heb doorgebracht, voor mij onvergetelijk gemaakt.

Prof. dr. K.W. Taconis, als hoofd van de Vakgroep waarin ik werkzaam was, Prof. dr. R. de Bruyn Ouboter, als promotor, waren steeds bereid mijn belangen in het laboratorium te behartigen.

Veel medewerking ondervond ik ook van Drs. C.M. van Dam en B. van der Geest.

Ik betuig mijn dank aan de Leidse Universiteit voor de mij geboden gelegenheid tot het doen van een promotie-onderzoek en aan de Egyptische Regering en haar culturele vertegenwoordiging in

Bonn, voor de steun gedurende mijn studie in Leiden.

Met veel toewijding is het type-werk voor dit proefschr verzorgd door Mevr. M.G. de Jong-Van der Meijde, de tekening door J. Bij en de foto's door W.F. Tegelaar.

STELLINGEN.

1. Het is gewenst experimenteel te onderzoeken, welk deel van het warmtecontact van germanium-weerstandsthermometers met de omgeving tot stand komt via de toevoerdraden en welk deel via de capsule waarin het germaniumkristal is gemonteerd.

   Dit proefschrift, hoofdstuk 2.

2. De overgangscurven die bij de bepaling van het supergeleidende overgangspunt van cadmium volgens de inductiemethode zijn gevonden, wijzen er op, dat de verbreding van de overgang die in enkele preparaten optreedt, een gevolg is van een mechanisme dat de kritische temperatuur verhoogt.

   Dit proefschrift, p. 59.

3. De detectie van massatransport in onverzadigde superfluïde helium-films volgens de thermische methode biedt aanzienlijke voordelen ten opzichte van detectie op capacitieve wijze.

4. Metingen van de diëlectriciteitsconstante van een fluïdum in het kritische gebied geven in principe informatie over de kritische exponenten α, β, γ, δ en ϕ.

5. De conclusie van Morishita, dat de frequentie waarbij de differentiële susceptibiliteit wordt gemeten invloed heeft op het al of niet eerste orde zijn van de magnetische faseovergang in $ErCrO_3$, is aanvechtbaar.

   T. Morishita, J. Phys. Soc. Jap. 46(1979)1748.

6. De verklaring die Sichel e.a. geven voor de afhankelijkheid van de weerstand in koolstof-polyvinylchloride van het elektrische veld, is noch de enig mogelijke, noch de meest logische.

   E.K. Sichel, J.I. Gittleman en Ping Sheng, Phys. Rev. B18(1978)5712.

7. Het is mogelijk de werkruimte in een dubbele helmholtzspoel aanzienlijk te vergroten, zonder de homogeniteit van het magnetische veld aan te tasten, door de stroomrichting in twee van de spoelen tegengesteld aan die in de andere twee te kiezen.

8. Uit het waargenomen maximum in de soortelijke warmte van verdunde PdDy legeringen en uit de electronspin-resonantie-spectra van PdDy en van PdEr als functie van de concentratie van Dy en Er moet de conclusie worden getrokken, dat in deze legeringen de zeldzame-aardatomen een axiaal electrisch veld ondervinden, waarvan de grootte toeneemt met de concentratie.
   R.A.B. Devine, W. Zingg en J.M. Moreti, Solid State Comm. 11 (1972)233.
   R.A.B. Devine, J.M. Moreti, J. Ortelli, D. Shaltiel, W. Zingg en M. Peter, Solid State Comm. 10(1972)575.
   L.L. Isaacs, Phys. Rev. B9(1974)2228.

9. Het verdient aanbeveling te onderzoeken of voor de vervaardiging van rhodiumijzer-weerstandsthermometers draad kan worden gebruikt met een kleinere diameter dan thans gebruikelijk is.

10. De dampspanningsrelatie van vaste waterstof kan worden gebruikt voor temperatuurmetingen tussen 14 K en 8 K met een precisie van 1 millikelvin.

11. Bij de samenwerking op het gebied van de natuurwetenschappen tussen universiteiten in ontwikkelingslanden en in "westerse" landen zijn eenvoudige hulpmiddelen en wederzijds begrip veelal van meer belang dan het zonder meer overbrengen van dure en ingewikkelde apparaten.

A.E. El Samahy   Leiden, 11 december 1979.

Toegang tot de promotie wordt in verband met de beperkte ruimte in de Senaatskamer uitsluitend verleend op vertoon van een toegangskaart, die u op verzoek wordt toegezonden.

Na de promotie is er voor iedereen een receptie in het academiegebouw, Rapenburg 73, Leiden.

N.B. Met parkeerproblemen bij het academiegebouw moet rekening worden gehouden.