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접촉방법에 의한
고온의 정밀측정기술개발
(제 2 차년도)

Precision Measurement of High Temperature by Contact Method

연구기관

재단법인 한국표준과학연구원

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13. 초록(주요 참고문헌이나 관련문헌이 있을 경우 여기에 명시한다)

2차년도에는 962 ℃~ 1554 ℃ 온도영역에서 열전대의 온도눈금 실현을 위하여 고온 금속응고점(Cu, Ni, Pd) 셀을 설계, 제작하고 고온 금속응고점을 실현하기위한 수직형 고온전기로를 설계, 제작하였다. CCT관련연구로 NIST 및 6개의 주요 측정표준연구기관과 공동으로 ITS-90 에 준한 S 형열전대의 기준함수 및 기준테이블을 제정하였고, 이 기준테이블은 1993년 부터 IEC Publication 584-1로 공포되어 전세계적으로 공통으로 사용될 예정이다.

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13. ABSTRACT

To realize the temperature scale of thermocouple in the temperature range of 962 °C to 1554 °C, high temperature freezing point cells(Cu, Ni, Pd) have been designed and constructed in the second year project. And high temperature furnace for the realization of high temperature metal freezing points were designed and constructed. By the recommendation of the CCT, we produced the reference functions and reference tables for type S thermocouples based on the ITS-90 through the collaboration of eight national standard laboratories including NIST. This reference table will be published as the IEC Publication 584-1 and will be used internationally.

14. KEYWORDS

high temperature freezing point cell, high temperature furnace, noble metal thermocouple, ITS-90, CCT, reference function for the type S thermocouple

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KOREA RESEARCH INSTITUTE OF STANDARDS AND SCIENCE

제 출 문

과학기술처장관 귀하

본 보고서를 "접촉방법에 의한 고온의 정밀측정기술개발" 사업의 2차년도 최종보고서로 제출합니다.

1993년 1월 일

연구기관명 : 한국표준과학연구원
연구책임자 : 감 기 술
연 구 원 : 송 창 호
이 영 희

요 약 문

Ⅰ. 제 목

접촉방법에 의한 고온의 정밀측정기술개발

Ⅱ 연구의 목적 및 중요성

600 ℃~1554 ℃온도영역에서 접촉방법에의한 고온의 정밀측정기술을 개발하고 기준기급열전대 표준기준물을 제작한다.

Ⅲ. 연구의 내용 및 범위

1. 550 ℃~1100 ℃ 온도영역에서 동작되는 고온 PRT 와 열전대비교교정용 정밀전기로를 설계, 제작하고 마이크로컴퓨터를 이용한 온도제어 및 데이타 동시수집 시스템을 제작하였다. S형열전대의 e.m.f. 를 국제온도눈금- 90에 의거하여 575 ℃~962 ℃ 온도영역에서 측정하였으며,새로운 열전대인 Au-Pd 및 Au-Pt 열전대의 e.m.f. 를 600 ℃~962 ℃온도영역에서 측정하였다.

2. 2차년도에는 962 ℃~ 1554 ℃ 온도영역에서 열전대의 온도눈금실현을 위하여 고온 금속응고점(Cu,Ni,Pd)셀을 설계, 제작하고 고온 금속응고점을 실현하기위한 수직형고온전기로를 설계, 제작하였다. CCT 관련연구로 NIST 및 6개의 주요 측정 표준연구기관과 공동으로 ITS-90에 준한 S형열전대의 기준함수 및 기준테이블을 제정하였

고, 이 기준테이블은 1993년 부터 IEC Publication 584-1로 공포되어 전세계적으로 공통으로 사용될 예정이다.

3. 3차년도에는 복사온도계를 기준으로 한 열전대교정시스템을 구축하고 아울러 고온에서의 열전대의 안정도를 비롯한 특성조사를 수행한다. 고온백금저항온도계와 열전대 비교측정 데이타 및 복사온도계에 의한 열전대 비교측정 데이타와 고온 금속응고점 데이타를 분석하여 600 ℃~ 1554 ℃ 온도영역에서의 열전대의 내삽공식을 유도한다. S형열전대를 비롯한 귀금속열전대를 고온측정용 표준기준물로 국내에 보급 할 것이다.

IV. 연구의 결과 및 활용

1. 연구개발 결과

- 고온금속응고점실현용 수직형 정밀전기로 설계 및 제작(0~1600 ℃)
- 고온금속응고점(Pd, Ni, Cu)셀 설계,제작
- ITS-90에 준한 S형열전대의 기준함수 및 기준테이블제정

2. 활용에 대한 건의

- 고온용 온도계들의 정밀비교측정기술 확립
- 0~1600 ℃ 온도영역의 열전대 비교교정기술 확보
- Au-Pt 및 Au-Pd 열전대등 귀금속열전대의 기본적인 데이타 축적
- 고온금속응고점의 실현방법 확보

SUMMARY

I. Title of the Study

Precision measurement of high temperature by contact method.

II. Objectives and Importance of the Study

The objective and importance of this study is to develop the precision measurement technology of high temperature by contact method and fabricate the reference grade thermocouple as standard reference meterial in the temperature range of 600 °C to 1554 °C.

III. Contents and Scope of This Study

1. Precision heat pipe furnace which can be operated in the temperature range of 550 °C to 1100 °C was designed and constructed for a intercomparison between high temperature PRTs and thermocouples. Temperature control and data acquisition system was constructed using microcomputer. Thermal e.m.f.'s of S-type thermocouples were measured based on the ITS-90 in the temperature range of 575 °C to 962 °C, and e.m.f.'s of Au-Pt and Au-Pd thermocouples were measured in the range of 600 °C to 962 °C.

2. To realize the temperature scale of thermocouple in the temperature range of 962 °C to 1554 °C, high temperature freezing point cells(Cu, Ni, Pd) have been designed and constructed in the second year project. And high temperature furnace for the realization of high temperature metal freezing points were designed and constructed. By the recommendation of the CCT, we produced the reference functions and reference tables for type S thermocouples based on the ITS-90 through the collaboration of eight national standard laboratories including NIST. This reference table will be published as the IEC Publication 584-1 and will be used internationally.

3. The thermocouple calibration system will be designed and constructed by using the radiation thermometer as a reference thermometer and the characteristics of noble thermocouples will be investigated in the high temperature in the third year project. Based on the inter- comparison data between the radiation thermometer, and between the high temperature PRT and thermocouple, simple interpolating equations for thermocouples in the temperature range of 600 °C to 1554 °C will be devised. The calibrated thermocouples will be used as a standard reference material for high temperature measurement by contact method in this country.

IV. Results and Applications

1. Results

- Design and fabrication of precision electric furnace for intercomparison measurement (600 °C~1100 °C).
- Design and construction of high temperature metal freezing point cells(Pd, Ni, Cu).
- Production of the reference functions and reference tables for type S thermocouples based on the ITS-90.

2. Application

- Intercomparison technique of high temperature thermometers.
- Calibration technique of thermocouples in the range of 600 °C to 962 °C.
- Accumulation of basic data of thermoelectric power of Au-Pt and Au-Pd thermocouples.
- Realization method of high temperature metal freezing points.

CONTENTS

목 차

제 1 장 서 론

국제온도눈금-90 (International Temperature Scale of 1990, ITS-90)(1) 에서는 0 ℃~962 ℃ 온도영역의 표준은 PRT 를 Ag, Al, Zn, Sn, In 의 응고점 및 Ga 의 녹는점에서 교정하여 사용토록 하고있으며, 962 ℃ 이상의 영역은 금,은 혹은 구리의 응고점흑체를 사용하여 플랑크 복사법칙에 의거하여 복사온도계를 교정하여 사용하도록 정해져 있다. 고온백금저항온도계는 1000 ℃ 까지 사용할 수 있으나 온도감지부가 응력이 작용되지 않도록 (strain free 구조) 만들어져 있기 때문에 진동, 급격한 온도변화등 환경이 열악한 분위기를 가진 중화학공업등의 생산현장에서는 사용하기에 부적절한 온도계로 알려져 있다. 복사온도계는 측정온도영역에 제한을 받지 않고 광범위한 온도영역에 걸쳐 사용할수 있으나 근본적으로 측정방법이 비접촉식이기 때문에 측정물체 표면의 복사율 및 광로에서의 오차요인이 많아, 접촉식온도계에 비하여 측정정확도가 떨어진다. 열전대는 고온 PRT에 비하여 정확도는 나쁘나 사용하기가 간편하고 고온에서 접촉방법으로 온도를 측정할수 있는등 많은 장점을 가지고 있어서 고온측정이 많이 요구되는 생산현장에서 가장 많이 사용되는 온도계이다. 특히 최근에는 고온 PRT를 대신할수 있을 정도로 정확도가 높은 새로운 열전대의 개발이 외국의 표준기관에서 시도되고 있으며(2), 기존의 열전대도 새로운 온도눈금인 ITS-90에 준한 온도대 열기전력표 제정 및 교정방법에 대하여 많은 연구가 진행되고 있다.(3-6) 본 연구에서는 중화학공업에서 많이 요구되는 온도영역인 600 ℃~1554 ℃ 에서의 ITS-90에 의거한 열전대 교정방법을 개발하는 것을 목표로 하고 있으며 1 차년도 연구에서는 600 ℃~1000 ℃ 온도영역에서 고온 PRT와 열전대의 비교교정을위한 히트파이프 정밀전기로 제작 및 온도제어용 컴퓨터프로그램 개발, 그리고 이들 장치를

이용한 고온PRT와 열전대의 비교교정을 실시한 바 있으며(7), 2차년도에서는 S형 열전대의 기준테이블 제정 및 팔라듐, 니켈, 구리의 응고점 셀과 이들 응고점을 실현할 수 있는 고온전기로를 개발하여 금속응고점에 의한 고온열전대의 교정을 위한 준비를 완료하였다. 3차년도에는 복사온도계를 기준으로 열전대를 교정하는 시스템을 제작하고 금속응고점에서의 측정데이타와 복사온도계에 의한 측정데이타를 비교,분석하여 열전대의 교정에 필요한 기준함수를 유도하고자 하며, 이를 바탕으로 고온에서의 열전대의 교정방법확립과 기준열전대의 보급을 활성화 할 계획이다.

제2장 ITS-90 에 준한 S 형 열전대의 기준함수 및 기준테이블 제정

ITS-90이 발효된 이후 새로운 온도눈금에 의한 열전대의 기준함수 및 기준테이블이 제정되어 있지 않아서, 1991년 CCT의 요청으로 NIST가 주관하는 S형 열전대의 기준함수 및 기준테이블 제작프로그램에 참여하였다. 1991년도에는 주로 고온백금저항온도계를 기준으로 한 S형 열전대의 온도에 따른 열기전력 측정에 집중하였으며, 그 결과는 전년도 보고서(KRISS-92-011-IR)에 잘 정리되어 있으며, 1992년도에는 측정된 데이타를 이용한 기준함수 및 기준테이블 제정에 여러 국가표준기관과 공동으로 참여하여 최종적인 결과를 제 7차 "Temperature Symposium"에서 3편의 논문으로 발표하였다.(부록 1참조) 새로운 S형 열전대의 기준함수는 -50 °C에서 1768.1 °C(백금의 용융점)까지 적용되며, -50 °C부터 1064.18 °C(금의 응고점)까지는 이번의 측정결과를 토대로 만들어 졌으나 1064.18 °C이상의 온도영역은 IPTS-68의 기준함수를 IPTS-68과 ITS-90간의 온도차를 수학적으로 변환하여 얻었다.(8-10) S형 열전대의 새로운 기준함수는

$$E=\sum_{i=0}^{n}a_i(t_{90})^i \qquad \text{...} \qquad (2\text{-}1)$$

이며, 여기서 t_{90}은 °C 으로, E는 μV의 단위를 갖는다. 표2-1은 각 온도영역별로 정해진 계수를 나타내고 있으며, 표2-2는 식(2-1)을 사용하여 ITS-90에서 정의되어있는 여러 고정점에서의 S형 열전대의 열기전력과 1,2차 미분값을 표로 정리한 것이다. 이번에 정해진 ITS-90을 기준으로한 S형 열전대의 열기전력표를 IPTS-68의 열기전력표와 비교한 결과가 그림 2-1에 나타나 있다.

열기전력을 온도로 환산하기 위해서는 역함수가 요구되며 식(2-2)는 식

(2-1)의 역함수로서, 이 두식으로 계산되는 온도는 ±0.02 °C이내에서 서로 일치한다.

$$t_{90}=\sum_{i=0}^{n}b_i(E)^i \qquad \text{...}(2\text{-}2)$$

표2-3은 역함수의 계수를 각 온도영역별로 나타낸 것이다. 현재 ITS-90에 의거한 S형 열전대의 기준함수는 CCT와 IEC Sub-committe 65B를 통과하였으며 1993년도에는 IEC Publication 584-1로 공표될 예정이다. 아울러 S형 열전대이외의 다른 형의 열전대에대한 기준함수 및 테이블은 S형 열전대를 기준으로하여 비교측정한 후 새로운 기준함수가 제정될 것이다.

Table 2-1. Coefficients of the reference functions for type S thermocouples for the indicated temperature ranges.

-50 °C to 1064.18 °C		1064.18 °C to 1664.5 °C	
a_1	5.40313308631	a_0	$1.32900444085 \times 10^{3}$
a_2	$1.25934289740 \times 10^{-2}$	a_1	3.34509311344
a_3	$-2.32477968689 \times 10^{-5}$	a_2	$6.54805192818 \times 10^{-3}$
a_4	$3.22028823036 \times 10^{-8}$	a_3	$-1.64856259209 \times 10^{-6}$
a_5	$-3.31465196389 \times 10^{-11}$	a_4	$1.29989605174 \times 10^{-11}$
a_6	$2.55744251786 \times 10^{-14}$		
a_7	$-1.25068871393 \times 10^{-17}$	1664.5 °C to 1768.1 °C	
a_8	$2.71443176145 \times 10^{-21}$		
		a_0	$1.46628232636 \times 10^{5}$
		a_1	$-2.58430516752 \times 10^{2}$
		a_2	$1.63693574641 \times 10^{-1}$
		a_3	$-3.30439046987 \times 10^{-5}$
		a_4	$-9.43223690612 \times 10^{-12}$

Table 2-2. Values of E and the first and second derivatives of E with respect to t_{90} computed from equation (2-1) at selected fixed points of the ITS-90.

t_{90}(°C)	E(μV)	dE/dt_{90}(μV/°C)	d^2E/dt^2_{90}(nV/°C^2)
-38.8344	-189.40	4.312	31.23
0.000	0.00	5.403	25.19
0.01	0.05	5.403	25.19
29.7646	171.39	6.094	21.36
156.5985	1082.27	8.045	10.69
231.928	1715.00	8.711	7.24
419.527	3446.89	9.638	3.50
630.615	5552.64	10.303	3.16
660.323	5860.13	10.398	3.23
961.78	9148.38	11.418	3.22
1064.18	10334.20	11.743	3.27
1084.62	10574.80	11.798	2.55
1664.5	17535.96	11.681	-2.94
1768.1	18693.54	10.311	-23.52

Table 2-3. Coefficients of the inverse functions for the type S thermocouples for the indicated temperature ranges.

	-50 °C to 250 °C		250 °C to 1200 °C
b_1	$1.84949460 \times 10^{-1}$	b_0	$1.291507177 \times 10^{1}$
b_2	$-8.00504062 \times 10^{-5}$	b_1	$1.466298863 \times 10^{-1}$
b_3	$1.02237430 \times 10^{-7}$	b_2	$-1.534713402 \times 10^{-5}$
b_4	$-1.52248592 \times 10^{-10}$	b_3	$3.145945973 \times 10^{-9}$
b_5	$1.88821343 \times 10^{-13}$	b_4	$-4.163257839 \times 10^{-13}$
b_6	$-1.59085941 \times 10^{-16}$	b_5	$3.187963771 \times 10^{-17}$
b_7	$8.23027880 \times 10^{-20}$	b_6	$-1.291637500 \times 10^{-21}$
b_8	$-2.34181944 \times 10^{-23}$	b_7	$2.183475087 \times 10^{-26}$
b_9	$2.79786260 \times 10^{-27}$	b_8	$-1.447379511 \times 10^{-31}$
		b_9	$8.211272125 \times 10^{-36}$

	1064 °C to 1664.5 °C		1664.5 °C to 1768.1 °C
b_0	$-8.087801117 \times 10^{1}$	b_0	$5.333875126 \times 10^{4}$
b_1	$1.621573104 \times 10^{-1}$	b_1	$-1.235892298 \times 10^{1}$
b_2	$-8.536869453 \times 10^{-6}$	b_2	$1.092657613 \times 10^{-3}$
b_3	$4.719686976 \times 10^{-10}$	b_3	$-4.265693686 \times 10^{-8}$
b_4	$-1.441693666 \times 10^{-14}$	b_4	$6.247205420 \times 10^{-13}$
b_5	$2.081618890 \times 10^{-19}$		

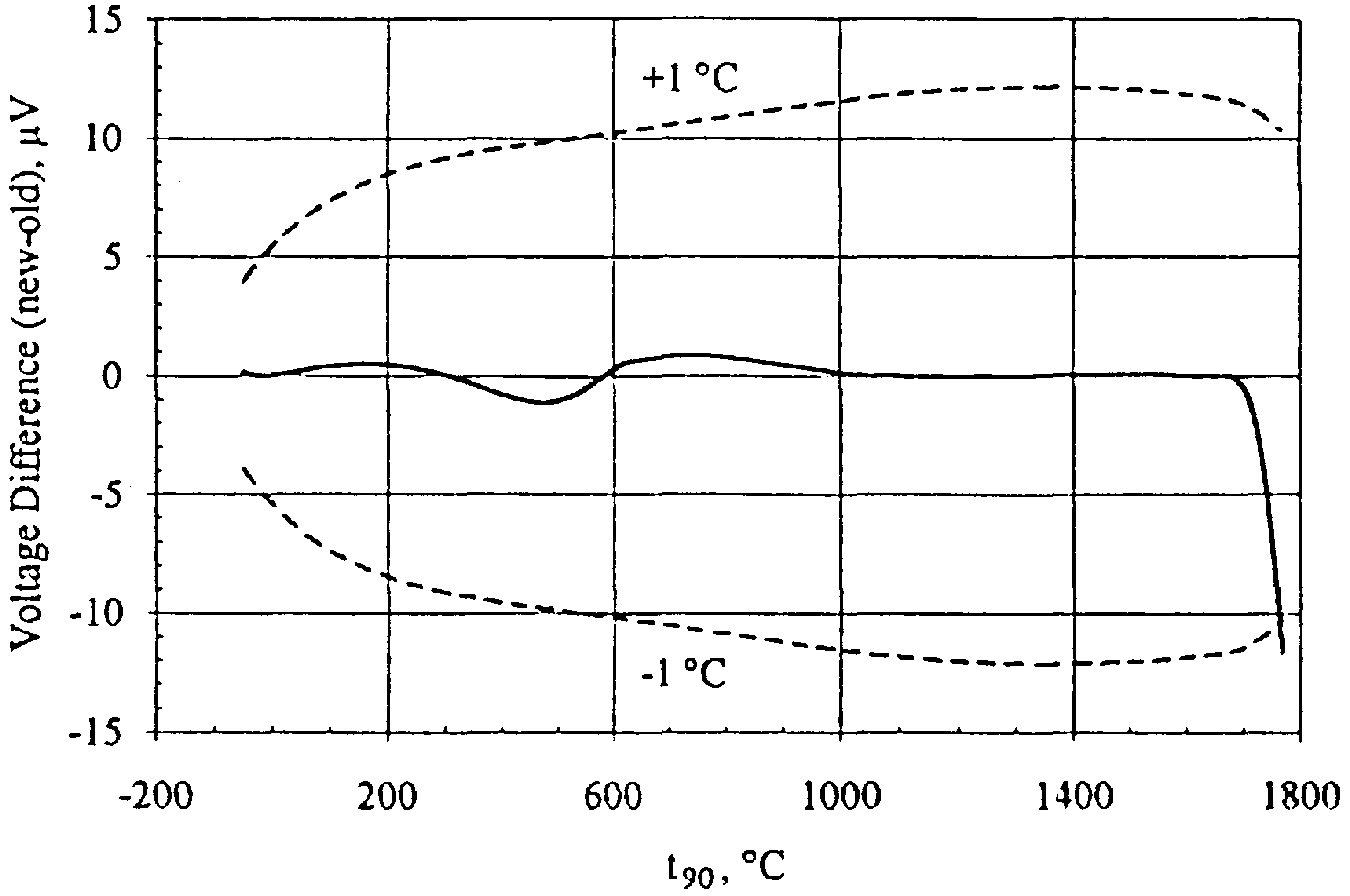

Fig.2-1. Differences between the new ITS-90 reference functions and the old IPTS-68 reference functions for the type S thermocouples. The dashed lines indicate an emf deviation equivalent to ±1 °C.

제3장 고온금속응고점 실현용 고온전기로 설계 및 제작

고온금속응고점(Pd, Ni, Cu)을 실현하기 위하여 그림3-1과 같은 고온전기로를 설계,제작하였다.(부록2. 고온전기로 부품도면참조) 고온전기로는 사용최고온도가 1600 ℃으로서 로듐이 30 %함유된 백금선(선경 0.5 mm)을 열선으로 사용하였으며, 백금열선의 오염을 방지 및 고온단열재로 마그네시아 벽돌을 사용하였다. 열선은 고순도알루미나튜브(o.d.59 mm, i.d.46 mm, length 50 cm)외부에 magnetic noise를 줄이기 위하여 2중으로 감았으며,총 turn수는 32회로 셀의 도가니가 위치하는 부분에 15 cm를 가열할 수 있도록 설계하였다. 백금선은 온도에 따른 저항변화가 크기때문에 전기로의 열선으로 사용한 5 m의 경우 실온에서 5.7 Ω의 저항을 가지나 1600℃에서는 42.0 Ω의 저항을 가져서 220 V의 교류전원을 사용할 경우 실온에서 8.5 kW의 출력을 낼 수 있으나 백금선의 최대허용전류인 15 A를 넘지않도록 전압을 강하하여 사용하여야 한다. 복사에 의한 과도한 열손실을 방지하기위하여 마그네시아 벽돌과 외피사이에 스테인레스 310S로 제작된 복사방지판을 설치하였으며, 복사방지판과 외피사이에는 fiber frax를 채웠다. 외피 및 아래판의 외부에는 냉각을 위하여 구리냉각파이프를 용접하여 부착하였다.

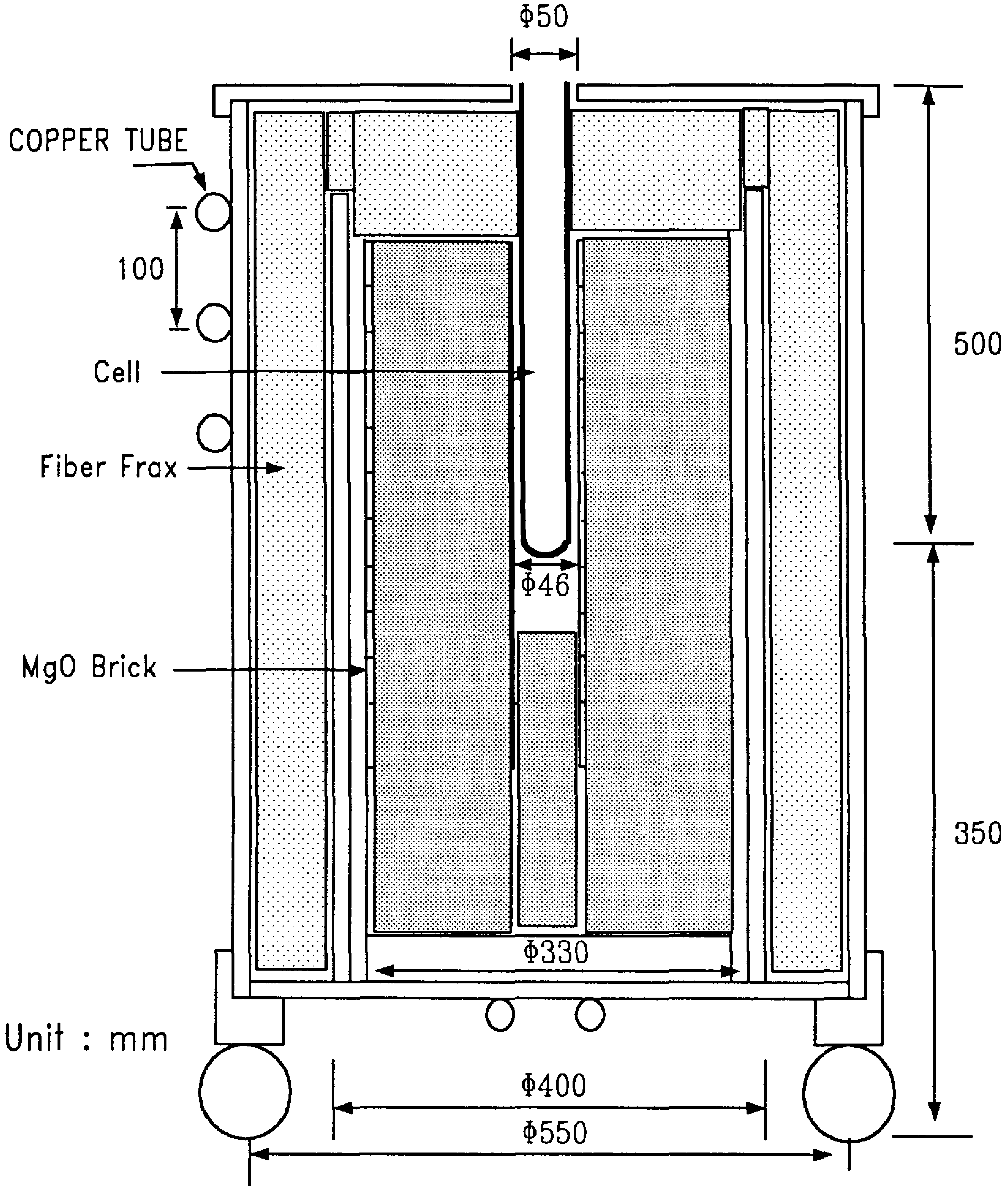

Fig.3-1. Cross section of high temperature furnace for the realization of the freezing points of Pd, Ni, Cu.

제4장 고온금속응고점(Pd,Ni,Cu)셀 설계,제작

열전대 정점교정용 고온금속응고점셀은 1차년도에 설계를 완료하였으며 (KRISS-92-011-IR참조), 2차년도에는 고순도 금속(Pd, Ni, Cu)을 각각 400 g,300 g, 500 g구입하고 흑연도가니에 충진하였다. Pd의 순도는 99.99 %로서 일본의 고순도연구소제품을 구입하였고, Cu 및 Ni은 각각 99.9999 % 및 99.995 %의 순도를 가진것으로 Johnson-Matthey사에서 제조된것이다. Pd 및 Ni응고점셀은 용적이 28.3 cc인 I형셀구조로 개방형으로 제작되었으며, Cu응고점셀은 용적이 49.6 cc로 설계된 II형셀구조이며 흑연도가니 및 온도계구멍을 석영유리로 seal함으로서 밀봉형 셀로 제작하였다. 밀봉에 사용된 silica glass는 복사선의 도파로 인한 열손실을 차단하기 위하여 셀외부 및 온도계센서well의 외부면을 sand-blasting 처리를 하였다. 충진된 고순도금속은 Pd 400 g, Ni 300 g, Cu 500 g으로서 Cu셀의 경우는 고온백금저항온도계의 사용도 가능하도록 온도계삽입구의 내경을 14.5 mm로 하였으며, 충분한 담금효과특성을 부여하기위하여 흑연도가니의 높이를 165 mm로 하여 제작하였다. 그림4-1은 PTB에서의 sealed cell 제조방법을 따른(11) Cu응고점셀의 밀봉화과정을 나타내고 있으며, 개방형 응고점셀의 제작방법은 과거의 연구결과보고서(KSRI-89-26-IR)(12)에 자세히 기술되어있다.

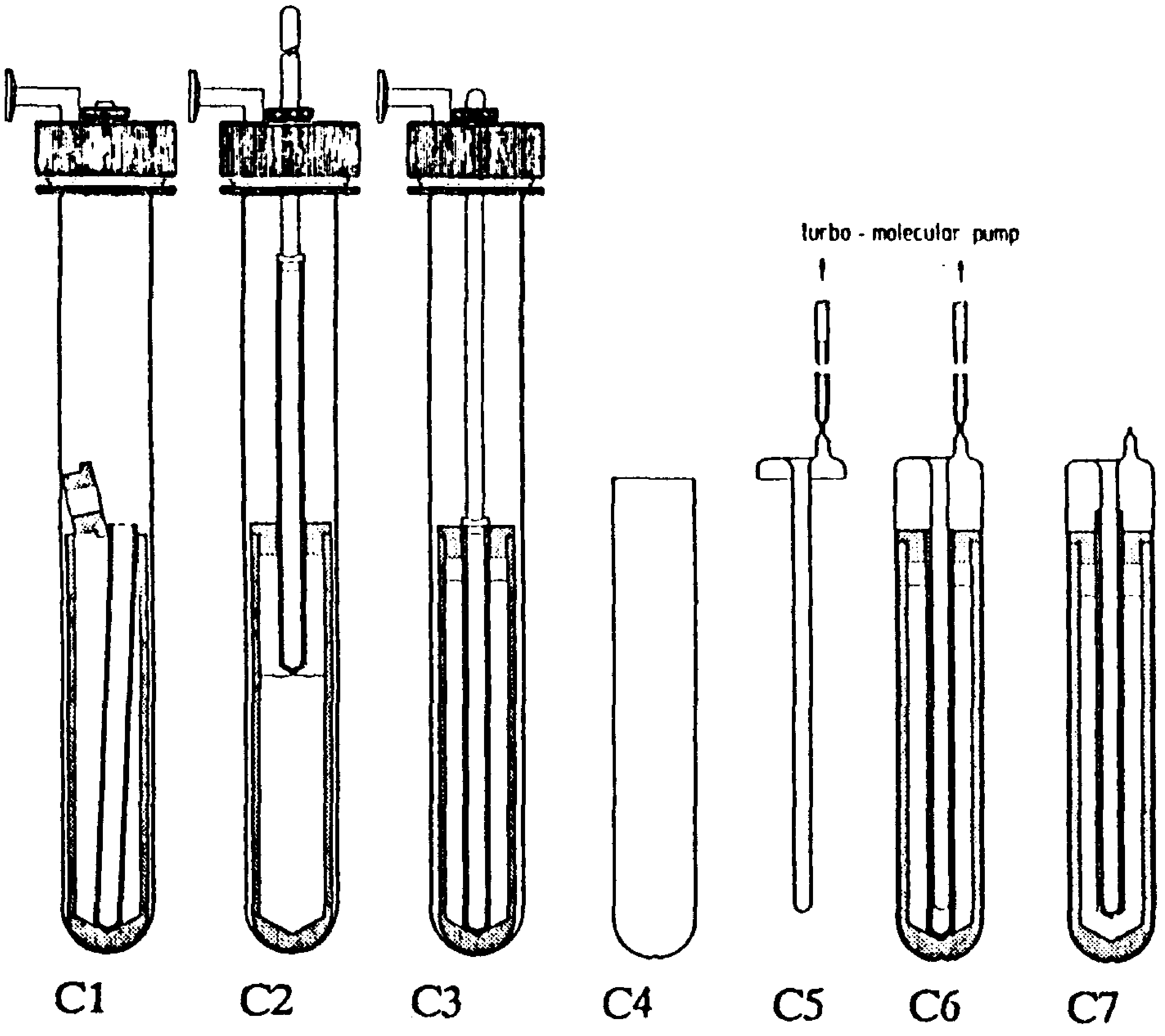

Fig.4-1. The steps of preparation of sealed freezing point cells. C1:Bake-out of high purity graphite components inside an auxiliary vessel. C2:Insertion of high purity metals and alumina press rod. C3:Melting of the metal samples and immersion of graphite well. C4:Cleaning of silica glass sheath. C5:Cleaning of the silica glass well with the cap. C6:Insertion of the filling graphite crucible from no.(C3) into the silica glass sheath(C4) and fusing the cap (C5) onto the it. C7:Melting the metal samples until the graphite well is floated upwards.

제5장 귀금속열전대의 제작 및 고정점측정

제1절 열전대 제작

S 형 열전대를 대체할 수 있는 새로운 귀금속열전대 개발을 위하여 선경이 0.5 mm인 Pt, Pd, Au 3종의 고순도 귀금속 소선을 Johnson-Matthey 및 고순도회사에서 구입하여 Au-Pt, Au-PD, Pt-Pd 열전대를 구성하였다. 표5-1은 이번에 제작된 귀금속 열전대의 구성과 순도를 정리한 것이다. 열전대소선의 표면은 손기름등 유기물을 제거하기 위하여 trichloroethylene 용액 및 ethylalcohol 로 개끗이 딱은후에(13) water welder로 접점을 형성하였다.

제2절 열처리 및 고정점에서의 열기전력 측정

열전대는 제작과정 및 접점용접과정에서 소선내에 잔류응력이 존재하게 될 뿐만 아니라 합금소선인 경우 첨가불순물의 농도구배가 존재하여 비균질한 특성을 보인다. 이러한 오차요인을 최소로 줄이기 위하여 소선을 소둔열처리 하게된다. S형 열전대는 IPTS-68 에 열처리방법이 명시되어 있으므로 그 절차에 따라 1450 °C에서 1시간이상 AC전류를 흘려 열처리 하였으며, Pd소선은 1300 °C에서 1시간 열처리 하였다. Au 소선은 silica glass 튜브내에 소선을 장치하여 1000 °C의 수평전기로에서 1시간열처리하였다. 열전대의 열처리에 대한 연구는 카나다NRC 및 호주NML에서 많이 수행되었으며 앞으로 그 결과를 이용한 KRISS식 열처리 방법을 개발해야 할 것으로 생각된다. 각 고정점에서의 열전대의 열기전력 측정은 먼저 S형 열전대를 금의 녹는점에서 소선용융법으로 측정한 후 은,알루미늄,안티모니,아연,주석,인듐

의 응고점 순으로 높은온도 에서 낮은 온도방향으로 측정하였다. 열전대의 열기전력은 수십 mV정도로 미약한 신호이기 때문에 빙점에서의 열전대소선과 voltmeter(Keithley Model 182)의 연결선으로 선경 1.2 mm인 구리선을 사용하였으며, voltmeter 단자 및 내부에서 발생되는

Table 5-1. Designation and purity of the noble metal thermocouples.

Designation of noble thermocouples	Suppliers	Purity of wires
KSTC 1	Sigmund-Cohen	Pt:5N up
KSTC 2	"	"
Pt-Pd 1	Johnson-Matthey	Pt:6N Pd:4N5
Pt-Pd 2	"	"
Au-Pd	"	Au:5N5 Pd:4N5
Au-Pt 1	"	Au:5N5 Pt:6N
Au-Pt 2	"	"
STC-131	Sigmund-Cohen	Pt:5N up
JAu-Pt 1	Kosundo Lab.	Au:5N Pt:4N
JAu-Pt 2	"	"
JAu-Pd 1	"	Au:5N Pd:4N
JAu-Pd 2	"	"
JPt-Pd 1	"	Pt:4N Pd:4N
JPt-Pd 2	"	"

열기전력 잡음을 상쇄시키기 위하여 모든 측정은 polarity 를 바꾸어 가면서 2 번씩 측정하여, 그 평균값을 데이타로 취하였다. 표5-2는 각 고정점에서 측정한 열전대의 열기전력을 종합한 것이다. 3종의 새로운 열전대중 Au-Pt열전대는 S형 열전대에 비하여 1000 ℃근처에서 1.6배의 분해능을 갖고있으며, 안정도도 ±10mK이내로 우수한 것으로 알려져 있다(6). Au-Pd열전대는 3종의 귀금속 열전대중 가장 분해능이 우수한 열전대로서 S형 열전대의 2.6배정도로 나타났으며, 앞으로 안정도등의 제반특성이 S형 열전대보다 우수할 경우 새로운 고온용 정밀온도계로 각광을 받을것으로 기대된다. 3차년도에는 특별히 이 열전대에 대한 특성조사연구를 추가로 수행 할 예정이며, 고온백금저항온도계 대체용 표준온도계로서의 가능성도 있는 것으로 생각된다. Pt-Pd열전대는 S형 열전대와 비슷한 특성을 보였으나 특별한 장점이 발견되지 않아 더 이상 연구할 가치가 없는 것으로 판단된다.

Table 5-2. Calibration data of the noble metal thermocouples at the metal freezing points.

T/C Designations	Freezing Points e.m.f.(μV)						
	E(Au)	E(Ag)	E(Al)	E(Sb)	E(Zn)	E(Sn)	E(In)
KSTC 1	10315.0	9134.1	-	5545.0	3442.5	1715.8	1083.5
KSTC 2	10314.7	9134.8	-	5544.6	3442.9	1715.3	1083.3
Pt-Pd 1	-	10807.4	5776.2	5372.9	2961.8	1427.8	-
Pt-Pd 2	-	10807.1	5776.8	5373.2	2962.8	1428.3	-
Au-Pd	-	26874.0	15058.9	14078.9	7888.6	3651.1	-
Au-Pt 1	-	16056.5	9262.7	8707.6	4906.7	2208.9	1330.0
Au-Pt 2	-	16065.6	9282.0	8708.5	4927.8	2224.5	-
STC-131	-	9119.5	5852.5	-	3437.5	1704.4	1080.1
JAu-Pt 1	-	15841.1	9132.2	-	4829.6	2176.3	1311.8
JAu-Pt 2	-	15853.7	9136.8	-	4859.2	2186.6	1317.6
JAu-Pd 1	-	26676.6	14935.7	-	7811.0	3613.0	2237.4
JAu-Pd 2	-	26684.9	14937.0	-	7830.8	3618.7	2240.3
JPt-Pd 1	-	10837.8	5805.1	-	2978.2	1435.1	925.3
JPt-Pd 2	-	10838.8	5803.9	-	2980.5	1435.8	925.7

제6장 결 론

열전대는 고온 PRT에 비하여 정확도는 나쁘나 사용하기가 간편하고 고온에서 접촉방법으로 온도를 측정할수 있는등 많은 장점을 가지고 있어서 고온측정이 많이 요구되는 생산현장에서 가장 많이 사용되는 온도계이다. 본 연구에서는 중화학공업에서 많이 요구되는 온도영역인 600 ℃~1554 ℃ 에서의 ITS-90에 의거한 열전대 교정방법을 개발하는 것을 목표로 하고 있으며 1 차년도 연구에서는 600 ℃~1000 ℃ 온도영역에서 고온 PRT와 열전대의 비교교정을위한 히트파이프 정밀전기로 제작 및 온도제어용 컴퓨터프로그램 개발, 그리고 이들 장치를 이용한 고온PRT와 열전대의 비교교정을 실시한 바 있으며, 2차년도에는-50 ℃에서 1768.1 ℃ (백금의 용융점)까지 ITS-90을 기준으로한 새로운 S형 열전대의 기준함수를 얻었다. 현재 ITS-90에 의거한 S형 열전대의 기준함수는 CCT와 IEC Sub-committe 65B를 통과하였으며 1993년도에는 IEC Publication 584-1로 공표될 예정이다. 아울러 S형 열전대이외의 다른형의 열전대에대한 기준함수 및 테이블은 S형 열전대를 기준으로하여 비교측정한 후 새로운 기준함수가 제정될 것이다.

고온에서의 열전대정점교정용 팔라듐,니켈,구리의 응고점셀과 이들 응고점을 실현할 수 있는 고온전기로를 개발하여 금속응고점에 의한 고온열전대의 교정을 위한 준비를 완료하였으며, 3종의 귀금속열전대(Au-Pt, Au-Pd, Pt-Pd)를 3군데의 공급회사에서 구입한 귀금속 소선으로 제작하여 열처리,정점교정을 통하여 분해능을 측정하고, S형 열전대와 고온백금저항온도계를 대체할 수 있는 새로운 열전대의 개발을 시도하였으며,3종의 열전대중 Au-Pd열전대는 분해능이 S형 열전대에 비하여 2.6배 정도 우수하여 앞으로 장기안정도 등의 제반특성을 더 조사할 예정이다. 3차년도에는 복사온도계를 기준으로 열전대를 교정하는 시스템을 제작하고 금속응고점에서의 측정데이타와 복사온도계에 의한 측정데이타를 비교,분석하여 열전대의 교정에 필요한 기준함수를 유도하고자 하며, 이를 바탕으로 고온에서의 열전대의 교정방법확립과 기준열전대의 보급을 활성화 할 계획이다.

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여 백

제 8 장 부 록

부록1. 7th Temperature Symposium 게제논문(3편)

부록2. 고온전기로 부품도면

부록1. 7th Temperature Symposium 게재논문(3편)

New reference function for platinum-10% rhodium versus platinum (type S) thermocouples based on the ITS-90. Part I: Experimental procedures

G. W. Burns, G. F. Strouse, and B. W. Mangum
Process Measurements Division, National Institute of Standards and Technology (NIST), Gaithersburg, Maryland 20899

M. C. Croarkin and W. F. Guthrie
Statistical Engineering Division, National Institute of Standards and Technology (NIST), Gaithersburg, Maryland 20899

P. Marcarino and M. Battuello
Istituto di Metrologia "G. Colonnetti" (IMGC), Torino, Italy

H. K. Lee, J. C. Kim, K. S. Gam, and C. Rhee
Temperature Laboratory, Korea Research Institute of Standards and Science (KRISS), Taejon, Republic of Korea

M. Chattle
Division of Quantum Metrology, National Physical Laboratory (NPL), Teddington, United Kingdom

M. Arai and H. Sakurai
Thermometry Section, National Research Laboratory of Metrology (NRLM), Ibaraki, Japan

A. I. Pokhodun, N. P. Moiseeva, and S. A. Perevalova
D. I. Mendeleyev Institute for Metrology (VNIIM), St. Petersburg, Russia

M. J. de Groot
Van Swinden Laboratorium (VSL), Delft, The Netherlands

Jipei Zhang, Kai Fan, and Shuyuan Wu
Shanghai Institute of Process Automation Instrumentation (SIPAI), Shanghai, China

The Comité Consultatif de Thermométrie requested its Working Group 2 to collaborate with national laboratories in the production of new reference tables and functions for thermocouples based on the International Temperature Scale of 1990 (ITS-90). Pursuant to this recommendation, eight national laboratories have obtained new data on type S thermocouples obtained from several sources. The thermoelectric voltages of those thermocouples have been measured as a function of t_{90} in the range −50 °C to 1070 °C, with temperatures obtained from standard platinum resistance thermometers that were calibrated in accordance with the ITS-90 to 962 °C and extrapolated to 1070 °C. Also, in the range from 710 °C to 1065 °C, temperatures were measured with a radiation thermometer. In addition, the thermoelectric voltages of the thermocouples have been determined at various thermometric fixed points. The experimental procedures, apparatuses, and materials used for the measurements are described in this part.

INTRODUCTION

The adoption of the International Temperature Scale of 1990 (ITS-90) (1), which supersedes the International Practical Temperature Scale of 1968, amended edition of 1975 (IPTS-68) (2), requires that the reference functions and tables for the thermocouples incorporated in various national and international standards (3, 4, 5) be revised to give the electromotive force (*emf*) as a function of t_{90}. Mathematical conversions of the previous thermocouple functions (6) were performed at NIST using the temperature scale differences tabulated in Ref. 1, but they produced unsatisfactory results due to the slope discontinuity at t_{68} = 630.74 °C (7) that was inherent in the IPTS-68. Consequently, Working Group 2 of the Comité Consultatif de Thermométrie circulated a request (July 1990) to national laboratories inviting an international collaborative effort to generate new experimental data for Pt-10%Rh vs. Pt (type S) thermocouples. Pursuant to this request, eight national laboratories obtained new data for the determination of new reference functions and tables for the type S thermocouple based on the ITS-90. In this part, we describe the experimental procedures, apparatuses, and materials used in obtaining these data. Part II of this paper presents the results obtained from this international collaboration and gives the new reference and inverse functions. In addition, data from seven of the participating laboratories were used for a new determination of (t_{90} - t_{68}) values over the range 630 °C to 1064 °C and the results are presented in Part II.

EXPERIMENTAL PROCEDURES, APPARATUSES, AND MATERIALS

The *emf*-t_{90} relationships of type S thermocouples were measured at the eight laboratories using different thermocouples, different experimental procedures, and, of course, different apparatuses. Altogether, such measurements were obtained for 37 thermocouples acquired from several sources. At all the laboratories, measurements were made of the *emf* of the thermocouples as a function of t_{90} over the range 630 °C to 962 °C, with t_{90} being determined with high temperature standard platinum resistance thermometers (HTSPRTs) calibrated according to the ITS-90. Henceforth in this paper, this will be referred to as a comparison measurement.

Similar measurements with standard platinum resistance thermometers (SPRTs) were conducted within the range -50 °C to 630 °C at SIPAI and NIST. At IMGC, temperatures from 710 °C to 1065 °C were measured also with an infrared pyrometer, while at NIST temperatures up to 1070 °C were measured with an HTSPRT. In addition, the thermocouples were calibrated at various thermometric fixed points as realized either in metal freezing-point cells or by the melting-wire method. The methods used for the measurements and the number of thermocouples tested at each laboratory are summarized in Table I.

The experiments conducted at KRISS, NIST, NRLM, SIPAI, and VNIIM are described in the following sections. Those performed at IMGC, NPL, and VSL are described in another paper at this Symposium (8). In all of the experiments, the reference junctions of the thermocouples were maintained at 0 °C in an ice bath when measurements were made.

Table I. Type S thermocouple measurements performed at the participating laboratories.

Laboratory	**Thermocouples**		**Measurement Methods and Temperature Ranges**		
	Number Tested	Number of Manufacturers	Comparison with SPRT	Fixed-Point Cell	Comparison with Pyrometer
			(range, °C)	(points)	(range, °C)
IMGC	4	3	600 to 962	Sb, Al, Ag, Au	710 to 1065
KRISS	2	1	575 to 962	Zn, Sb, Ag, Au[1]	
NIST	5	3	-50 to 1070	In, Sn Cd, Zn Al, Ag, Au	
NPL	4	2	600 to 963	In, Sn, Zn, Al, Ag, Au	
NRLM	5	4	628 to 962	Sn, Zn, Al, Ag, Au	
SIPAI	13	4	0 to 962		
VNIIM	2	1	595 to 962	Zn, Al, Ag, Au	
VSL	2	1	600 to 970	Sn, Zn, Al, Ag, Au, Pd[1]	

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[1] By melting-wire method

KRISS Measurements

Two thermocouples (KSTC 1 AND KSTC 2) were compared with an HTSPRT in the range 575 °C to 962 °C. The experimental procedure was first to calibrate the HTSPRT and thermocouples at fixed points, then compare them in a graphite-block comparator cell, and finally recalibrate them at the same fixed points.

Both thermocouples were made from the same lots of reference grade Pt and Pt-10% Rh thermocouple wire, 0.5 mm diam, supplied by a manufacturer in the USA. The procedures for annealing, insulating, and protecting the thermocouples were essentially the same as those used by Evans and Wood (9). Calibrations of the thermocouples were made first at the melting point of Au by the melting-wire method and then at the freezing points of Ag, Sb, and Zn. The values of *emf* were measured with a calibrated digital multimeter (DMM, Hewlett Packard model 3458A[a]). During measurements, the polarity of the connections to the DMM was reversed to cancel out residual voltages. At each of the freezing points *emf* measurements were taken at 10 s intervals for 10 min. The standard deviations (1 σ) of such measurements were less than 50 nV. High purity (99.9999%) Au wire, 0.5 mm diam, was used for the calibrations at the Au melting point. The melting temperature of the Au wire was taken as the median of its melting curve (10). Typically, the melting range was about 0.1 °C. The reproducibility of the melting-point measurements was estimated to be ±1 μV.

A 0.25 Ω, bird-cage type HTSPRT purchased from Rosemount, Inc. was used in the comparison. Before the comparison measurements, the HTSPRT was annealed at 1000 °C for various lengths of time and then calibrated at the freezing points of Ag, Al, Zn, and Sn, in that order. The freezing-point cells were the same ones used in the calibration of the thermocouples. Throughout its calibration, the HTSPRT was cooled slowly (at about 1 °C/min) in a furnace down to 450 °C after being heated at high temperature. Its resistance was determined at the triple point of water (TPW) before and after the measurements in each of the freezing-point cells. The resistance of the HTSPRT was measured with a Guildline model 9975 current comparator resistance bridge using a measuring current of 10 mA, both in the fixed-point cells and during the comparison measurements. During the calibration of the HTSPRT performed after the comparison, it was given no further high temperature annealing. The total changes in the HTSPRT during the comparison measurements were +33.5 m°C, +34.6 m°C, +19.9 m°C, and +7.2 m°C at the Ag, Al, Zn, and Sn points, respectively.

The uniform, high-temperature environment needed for the comparison was achieved in a sodium heat-pipe furnace containing a graphite-block comparator. This furnace also was used for realizing the Ag and Al freezing points. The graphite-block comparator has 4 wells, 2 for thermocouples and 2 for HTSPRTs. The thermocouples and HTSPRT were protected by closed-end silica-glass tubes which were roughened on the outside. The thermocouples were positioned so that their measuring junctions were close to the midpoint of the Pt resistor of the HTSPRT.

The comparator cell was brought to the desired temperature by using a programmable controller having a resolution of 1 °C. Simultaneous measurements of resistance and *emf* were made when three successive observations, covering a period of 5 min, differed by not more than 10 m°C. Comparison measurements were made at about 50 °C intervals from 575 °C up to about 962 °C, and then from 900 °C down to 600 °C. Temperatures were calculated from the HTSPRT measurements made during the comparison using the average of the calibrations performed before and after the comparison measurements. In those calculations, corrections were applied to account for the hydrostatic head in all fixed-point cells, but no corrections were made for self-heating effects in the HTSPRT.

NIST Measurements

Five thermocouples (S1, S2, S3, S4, and S5), made from 0.5 mm diam wires, were compared with an SPRT over the range -50 °C to 550 °C and with an HTSPRT from 500 °C to 1070 °C. Thermocouple S4 was from the same wire lots used by Bedford *et al.* (11) as the basis for the IPTS-68 based reference functions. S4 was from the manufacturer denoted by A in Ref. 11. S1, S2, and S3 were from the wire lots obtained by Bedford *et al.* (11) from the manufacturer denoted by C in Ref. 11. S5 was from wire purchased in 1989.

The thermocouple wires were cleaned with ethyl alcohol and then annealed electrically in air. The Pt wire of each thermocouple and the Pt-10%Rh wire of S3 were annealed for 1 h at about 1450 °C, cooled rapidly (quenched) to room temperature and then annealed for 1 h at about 450 °C. The Pt-10%Rh wires of the other 4 thermocouples were annealed for 1 h at about 1450 °C, followed by 1 h at about 700 °C and then several minutes at 450°C. Next, the wires were mounted in twin-bore, alumina insulating tubes (4 mm in diameter, 1 mm bores, and 76 cm long) and further annealed in a 1.1 m long horizontal tube furnace. S3 was annealed for 20 h at 450 °C and then removed from the furnace. The other 4 thermocouples were annealed for 1 h at 1100 °C, cooled in about 3.5 h to 450 °C, held at 450 °C for 20 h and then removed from the furnace.

The comparison measurements between the thermocouples and the platinum resistance thermometers (PRT's) were made in a cryostat below 0 °C, in stirred-liquid baths from 10 °C up to 550 °C, and in a sodium heat-pipe furnace with an Inconel-block comparator from 500 °C up to 1070 °C.

The comparator had a cylindrical Inconel block, 25 cm long and 4.9 cm in diameter, with 6 wells for thermocouples equally spaced on a 3.1 cm diameter circle and a central, axial well for the HTSPRT. Each of the thermocouple wells contained an alumina protecting tube (5 mm i.d., 6.5 mm o.d.). The HTSPRT was protected from contamination from metal ions by inserting it into a platinum test tube (56 cm long, with a wall thickness of 0.13 mm) that was located between two 56 cm long fused-silica test tubes (12).

The automated measurement system of this experiment include an ASL F-18 ac resistance-ratio bridge, a DMM (HP 3458A), a scanner (HP 3495A) with low thermal switches, and a computer. Different-valued ac/dc reference resistors are used with the F-18 in order to increase the resolution and to minimize measurement error from non-linearity. The resolution of our measurements was the equivalent of 0.01 m°C for the 25.5 Ω SPRT and 0.04 m°C for the 0.59 Ω HTSPRT. The DMM was calibrated by the NIST Electricity Division twice before the experiment and then about every three months. Internal calibration of the DMM was conducted every 24 hours and additionally, whenever the internal temperature of the DMM changed by more than 1 °C. The thermocouple and platinum resistance thermometer data were taken automatically via a computer-controlled IEEE-488 bus and logged to a data file for later analysis.

The data-acquisition system also used the PRTs to determine when the comparison baths had reached thermal equilibrium. Measurements were taken only when a bath was drifting at a rate of less than 2 m°C/min. The thermocouple measurements were bracketed by the PRT measurements; at each temperature a thermocouple was measured four times at about 5 min intervals, reversing the order of readings each time, at each temperature. To correct for any drift in the DMM zero, thermocouple measurements were bracketed by measurements of a short on a scanner channel. Additionally, the data-acquisition system was used to automatically change the temperature of the sodium heat-pipe furnace containing the inconel-block comparator. An isolated digital/analog programmable power supply permitted a change in the reference voltage for the furnace control thermocouple to set a new temperature after each set of measurements was completed.

Both the SPRT (25.5 Ω Chino model R800-2) and the HTSPRT (0.59 Ω VNIIM, designated HTSPRT I in Ref. 12) were calibrated (13) on the ITS-90. The interpolation method used for determining temperatures with the HTSPRT above the freezing point of silver is discussed in Ref. 12. The 25.5 Ω SPRT was used for measurements over the range from -50 °C to 550 °C. After each measurement sequence, the SPRT was also measured at the TPW. The equivalent temperature change at the TPW during the comparison measurements was about 0.6 m°C. The difference between the calibrations of the SPRT performed before and after the comparison measurements was not more than 0.5 m°C. The HTSPRT was calibrated (see I1 and I2 in Ref. 12) before (I1) and after (I2) the three comparison runs. After each run, it was measured at the TPW in order to track its stability; the equivalent temperature change for the three runs was about 3.5 m°C (after 180 h above 1000 °C). The change in the HTSPRT between calibrations I1 and I2 was not more than 12 m°C and that being at the gold freezing point.

The measurement sequence for the thermocouples was as follows: 1) water bath (10 °C to 95 °C); 2) cryostat bath (-50 °C to -10 °C); 3) oil bath (95 °C to 180 °C); 4) ice bath (0 °C); 5) freezing points of In, Sn, Cd and Zn; 6) overnight furnace anneal at 450 °C (OFA); 7) salt bath (275 °C to 550 °C);, 8) OFA; 9) freezing point of Zn; 10) sodium heat-pipe furnace with inconel-block comparator (500 °C to 1070 °C); 11) 1450 °C and 450 °C wire anneal and then 450 °C furnace anneal for S3; and for the other four thermocouples, an 1100 °C and then a 450 °C furnace anneal (A2); 12) freezing points of Zn and Al; 13) A2; 14) freezing point of Ag; 15) A2; 16) freezing point of Au; 17) A2; and 18) Pt-67 comparison.

Repetitive measurements of S4 and S5 were made in order to establish reproducibility. Measurements of S4 and S5 were made in two comparator runs (Run01 and Run03). They were given an overnight 450 °C furnace anneal before the second run. Repetitive measurements of S4 and S5 were made also at the freezing points of In, Sn, Cd, and Zn. Immersion tests in the Zn freezing-point cell were used to yield results on the homogeneity of each thermocouple as a function of time.

The measured values of *emf* at 1064 °C for the platinum wires of thermocouples S1, S2, S3, S4, and S5 versus the NIST platinum thermoelectric reference standard, Pt-67 (6), were +9.9 μV, +10.5 μV, +9.9 μV, +6.5 μV, and +4.1 μV, respectively. The *emf* values for the Pt wires of S1, S2, S3, and S4 agree to within 1 μV with those reported by Bedford *et al.* (11) for samples from the same wire lots.

231.928 °C, 419.527 °C, 660.323 °C, and 961.78 °C. The thermocouples were then calibrated with decreasing temperature at 800 °C, 660 °C, and 500 °C. The means of the differences between values measured with increasing and decreasing temperature for the thirteen thermocouples were 0.44 µV, 0.31 µV, and 0.43 µV at 800 °C, 660 °C, and 500 °C, respectively. Additionally, thermocouple No. 81103 was calibrated six times at 600 °C and 800 °C on different days to estimate the reproducibility of the measurements. Four of the calibrations were performed with increasing temperature and two were conducted with decreasing temperature. The standard deviations (1 σ) of these measurements were found to be 0.25 µV at 600 °C and 0.36 µV at 800 °C.

VNIIM Measurements

Two type S thermocouples (325 and 326) were calibrated by comparison with HTSPRTs from 595 °C to 962 °C. Both thermocouples were made from the same lot of 0.5 mm diam. wire manufactured in Russia. The thermocouple wires were mounted in ceramic insulating tubes that were 50 cm long and 5 mm in diameter. Before the comparison, the thermocouples were annealed in a vertical tube furnace for 5 h at 1100 °C, and then they were calibrated in freezing-point cells of Zn, Al, Ag, and Au.

Two 0.6 Ω HTSPRTs (12) manufactured at VNIIM and a 0.25 Ω HTSPRT, model WZPB-5, acquired from the Yunnan Instrument Factory in the PRC were used in the comparison. Prior to their use, the 0.6 Ω HTSPRTs were subjected to a series of cyclic annealing treatments up to 1100 °C. They were inserted in a vertical tube furnace, heated to 1100 °C, held for 5 h at 1100 °C, and then cooled slowly in the furnace. Before the next annealing cycle, their resistances were determined at the TPW. The change in their resistances at the TPW after 12 such annealing cycles (60 h) did not exceed 1.5 m°C per cycle. The 0.25 Ω HTSPRT was annealed in the same manner but at 1000 °C. Its instability after annealing for 40 h did not exceed 1.0 m°C per cycle. After these annealing treatments, the HTSPRTs were calibrated at the ITS-90 fixed points.

The comparisons between the thermocouples and the HTSPRTs were made in an Inconel-block comparator. A cylindrical Inconel block, 15 cm long and 5.8 cm in diameter, was centrally located within a vertical tubular furnace having a sodium heat-pipe liner. The block had four, 10.8 cm deep, thermometer wells for 2 thermocouples and 2 HTSPRTs, which were equally spaced on a 3.2 cm diameter circle. A closed-end, silica glass tube was inserted in each well to protect the thermometers. Cylindrical graphite heat shunts were placed at intervals in the region above the Inconel block and the space between them was packed with insulation. The temperature of the furnace was maintained automatically with an electronic regulator. During the comparisons, the vertical non-uniformity of temperature within the furnace at a distance 25 cm above the bottom of the Inconel block did not exceed 0.5 °C.

A series of 10 HTSPRT-thermocouple comparisons were performed in which simultaneous measurements of thermocouple *emf* and HTSPRT resistance were taken at about 30 °C intervals from 595 °C to 962 °C, and in several cases at 10 °C intervals in the subrange 595 °C to 675 °C. At each temperature, the measurements were carried out simultaneously within 10 s and during this time the temperature inside the Inconel block did not change by more than 2 m°C. Both thermocouples and two HTSPRTs were included in each comparator run. In this manner both thermocouples were compared with the 0.25 Ω HTSPRT and with one of the 0.6 Ω HTSPRTs at least twice. A Guildline model 9975 current comparator bridge was used to measure the resistance, and a Soviet-made voltage comparator, type P-3017, was used for the *emf* measurements.

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[a]Certain commercial equipment, instruments, or materials are identified in this paper in order to adequately specify the experimental procedure. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

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INTRODUCTION

The adoption of the International Temperature Scale of 1990 (ITS-90) (1), which supersedes the International Practical Temperature Scale of 1968, amended edition of 1975 (IPTS-68) (2), requires that the reference functions and tables for the thermocouples incorporated in various national and international standards (3, 4, 5) be revised to give the electromotive force (*emf*) as a function of t_{90}. Mathematical conversions of the previous thermocouple functions (6) were performed at NIST using the temperature scale differences tabulated in Ref. 1, but they produced unsatisfactory results due to the slope discontinuity at t_{68} = 630.74 °C (7) that was inherent in the IPTS-68. Consequently, Working Group 2 of the Comité Consultatif de Thermométrie circulated a request (July 1990) to national laboratories inviting an international collaborative effort to generate new experimental data for Pt-10%Rh vs. Pt (type S) thermocouples. Pursuant to this request, eight national laboratories obtained new data for the determination of new reference functions and tables for the type S thermocouple based on the ITS-90. This part of the paper presents the results obtained from this international collaboration and gives the new reference and inverse functions. Data from seven of the participating laboratories were used for a new determination of (t_{90} - t_{68}) values over the range 630 °C to 1064 °C and the results are presented.

The experimental procedures, apparatuses, and materials used in obtaining the data were described in Part I of this paper. The *emf*-t_{90} relationships of type S thermocouples were measured at the eight laboratories using different thermocouples, different experimental procedures, and, of course, different apparatuses. Altogether, such measurements were obtained for 37 thermocouples acquired from several sources. At all the laboratories, measurements were made of the *emf* of the thermocouples as a function of t_{90} over the range 630 °C to 962 °C, with t_{90} being determined with high temperature standard platinum resistance thermometers (HTSPRTs) calibrated according to the ITS-90. Henceforth in this paper, this will be referred to as a comparison measurement. Similar measurements with standard platinum resistance thermometers (SPRTs) were conducted within the range -50 °C to 630 °C at SIPAI and NIST. At IMGC, temperatures from 710 °C to 1065 °C were measured also with an infrared pyrometer, while at NIST temperatures up to 1070 °C were measured with an HTSPRT. In addition, the thermocouples were calibrated at various thermometric fixed points as realized either in metal freezing-point cells or by the melting-wire method. The methods used for the measurements and the number of thermocouples tested at each laboratory are summarized in Part I (see Table I).

Computation of (t_{90} - t_{68}) values

Since the type S thermocouple was the standard interpolating instrument on the IPTS-68 (2) in the range from t_{68} = 630.74 °C to t_{68} = 1064.43 °C, values of the temperature differences between the ITS-90 and the IPTS-68 in this range were derived from our *emf*-t_{90} measurements. On the IPTS-68, the temperature, t_{68}, determined from type S thermocouples, is defined by the relation

$$E = a + bt_{68} + ct_{68}^2 \ , \tag{1}$$

where E is the *emf* when one of the junctions of the thermocouple is at 0 °C and the other is at t_{68}. The coefficients a, b, and c are calculated from values of E at t_{68} = 630.74 °C ± 0.2 °C, as determined by an SPRT, and at t_{68} = 961.93 °C and t_{68} = 1064.43 °C, the freezing points of Ag and Au, respectively. It should be noted that at these three temperatures, the adopted differences (1,7) for (t_{90} - t_{68}) are −0.125 °C, −0.15 °C, and −0.25 °C, respectively , and that the corresponding values of t_{90} at these points are 630.615 °C, 961.78 °C, and 1064.18 °C. We accepted the adopted differences between the scales at these temperatures in deriving the (t_{90} - t_{68}) values presented here.

The procedure for calculating (t_{90} - t_{68}) values from the *emf*-t_{90} data consisted of a three-step process. As a first step, the IPTS-68 quadratic for each thermocouple was determined. Next, for each of the measured values of *emf* within the range 630.615 °C to 1064.43 °C, the corresponding value of t_{68} was obtained from the IPTS-68 quadratic by iteration. The calculated values of t_{68} were then subtracted from the measured values of t_{90}. Such calculations were carried out for the 24 thermocouples (see Table I in Part I), whose calibrations included a measurement at the Au point.

The *emf* values at 630.615 °C, 961.78 °C, and 1064.18 °C for determining the IPTS-68 quadratic were obtained from the comparison and fixed-point data in the following ways. Values of *emf* at 630.615 °C and 961.78 °C were obtained for all 24 thermocouples from the HTSPRT-thermocouple comparison data by interpolation. For the 5 NIST thermocouples, *emf* values at 1064.18 °C were interpolated from the comparison data. Similarly, for the four thermocouples that were compared with an infrared pyrometer at IMGC, *emf* values were interpolated at 961.78 °C and 1064.18 °C from those comparison data. For the thermocouples that were measured at fixed points both before and after comparison with the HTSPRT, the mean of the *emf* values obtained before and after the comparison was computed at each fixed point, except for the two VSL thermocouples. Both of the VSL thermocouples, as discussed in Ref. 8, changed appreciably during the experiment, and the fixed-point data obtained prior to the comparison measurements differed substantially from the comparison data; hence, the initial fixed-point measurements were not used.

All of the thermocouples, except VSL-A, met the *emf* requirements for standard thermocouples on the IPTS-68 (see Eqs. (13), (14), (15) in Ref. 2) The values of *emf* for VSL-A did not satisfy Eq. (14) in Ref. 2.

Trial calculations of (t_{90} - t_{68}) values were made using various combinations of the *emf* values obtained from the comparison and fixed-point data to determine the IPTS-68 quadratic. It was clear that the best estimate for a particular thermocouple was realized by using data obtained in the same apparatus. For 15 thermocouples it was necessary, however, to use the fixed-point measurement at 1064.18 °C. In this instance we believe that better continuity in the results can be realized by using the mean of the fixed-point and comparison values at 961.78 °C, together with the *emf* value at 630.615 °C interpolated from the comparison data, to determine the IPTS-68 quadratics. Hence, the quadratics used to compute the (t_{90} - t_{68}) values presented here for those thermocouples, as well as for the four IMGC thermocouples that were compared with an HTSPRT, were determined in this manner. A second set of quadratics was calculated for the IMGC thermocouples using the *emf* values at 1064.18 °C and 961.78 °C interpolated from the infrared pyrometer comparison data and *emf* values at 630.615 °C obtained by computing the means of the HTSPRT-comparison and fixed-point (Sb). values. For the NIST thermocouples the IPTS-68 quadratics were determined by using the *emf* values interpolated from the comparison data at all three temperatures.

Because of chemical and physical inhomogeneities in the thermocouples and the different temperature gradients that existed in the comparison and fixed-point apparatuses, a given thermocouple was likely to produce a different value of *emf* at the same temperature in the two apparatuses. Analysis showed that at 961.78 °C, the difference between the *emf* value interpolated from the comparison data and the value obtained from fixed-point measurements was less than the equivalent of 0.1 °C for 19 of the 24 thermocouples. The difference between the comparison and fixed-point values at 1064.18 °C was less than 55 m°C for 8 of the 9 NIST and IMGC thermocouples. Such surprisingly close agreement was achieved, we believe, because the same furnace was used at NIST for the fixed-point and comparison measurements, while the blackbody comparator used at IMGC was designed (8) to have very nearly the same immersion conditions as the fixed-point apparatus.

The (t_{90} - t_{68}) values calculated from the IMGC, KRISS, NIST, NPL, NRLM, VNIIM, and VSL thermocouple data are shown in Figs. 1, 2, 3, 4, 5, 6, and 7, respectively. The previously published values (CIPM, see Ref. 1) for the temperature differences are shown for comparison. Statistical analysis of the (t_{90} - t_{68}) values was performed using iteratively reweighted least squares regression to obtain a consensus model for the difference between the two temperature scales and is described in another paper at this Symposium (17). The 5th degree polynomial which describes this consensus model for (t_{90} - t_{68}) is

$$\begin{aligned}\Delta t\,(t_{90}) = \;& (7.8687209 \times 10^{1}) \\ & -(4.7135991 \times 10^{-1})t_{90} \\ & \;\;(1.0954715 \times 10^{-3})t_{90}^{2} \\ & -(1.2357884 \times 10^{-6})t_{90}^{3} \\ & \;\;(6.7736583 \times 10^{-10})t_{90}^{4} \\ & -(1.4458081 \times 10^{-13})t_{90}^{5}\end{aligned} \qquad (2)$$

Figure 8 shows the (t_{90} - t_{68}) values computed from this polynomial, the values calculated from type S thermocouple data of the seven laboratories, and the previously published (CIPM) values (1).

Thermocouple reference functions

The new reference function giving the *emf* as a function of t_{90} over the range from −50 °C to 1064.18 °C is based upon the experimental data for NIST thermocouple S5. The rationale for this choice and the analysis of the data are given in Ref. 17. An 8th degree polynomial was fitted to the *emf*-t_{90} data for thermocouple S5 by the method of least squares. The residual standard deviation was 0.063 µV with 436 degrees of freedom. The polynomial was then adjusted quadratically, as described in Ref. 17, to obtain the reference function. As a consequence of this adjustment, the reference function gives the same value of *emf* at the freezing point of gold as the previous reference function (6), after the latter was corrected to account for the 1 January 1990 change in the volt (18).

Above 1064.18 °C, the new reference functions are based upon mathematical conversions of the IPTS-68 based reference functions (6). The previous functions consist of two cubics which join at t_{68} = 1665 °C. The use of two functions was necessary to accommodate the rapid decrease of the Seebeck coefficient above 1700 °C. Direct substitution of $t_{68} = t_{90} - \Delta t$ in the cubics, where Δt is given by Eq. (42) in Ref. 19, produced two 6th degree polynomials that give the *emf* as a function of t_{90}. The coefficients of both polynomials were multiplied by 0.999990736 to account for the change in the volt. These polynomials were then modified to obtain the reference functions as follows.

The 6th degree polynomial for the range 1064.18 °C to 1664.5 °C was truncated to a 4th degree polynomial. The coefficients of the 4th degree polynomial were then adjusted to obtain a polynomial that produces the same values of *emf* (E) and $\mathrm{d}E/\mathrm{d}t_{90}$ at 1064.18 °C as the reference function of the preceding range and the same values of E and $\mathrm{d}E/\mathrm{d}t_{90}$ at 1664.5 °C as the 6th degree polynomial before it was truncated. The resulting bias in the adjusted 4th degree polynomial relative to the 6th degree polynomial is ≤ 0.047 µV, in absolute terms. The adjusted 4th degree polynomial is used as the reference function in this range.

It should be noted that the previous reference function in the range above 1664.5 °C was based on t_{68} = 1767.6 °C (11) for the freezing point of Pt. More recent determinations of the Pt freezing-point temperature have resulted in a recommended value (20) of t_{68} = 1768.7 °C for this point.

The corresponding value of t_{90}, according to Eq. (42) in Ref. 19, is 1768.117 °C. Hence, in order for the new and old reference functions to give the same values of E at the freezing point of Pt, a corrective function was added in this range. First, the 6th degree polynomial for this range was truncated to a 4th degree polynomial, thereby incurring a bias of ≤ 0.022 µV. Then a cubic correction was made so that the resulting 4th

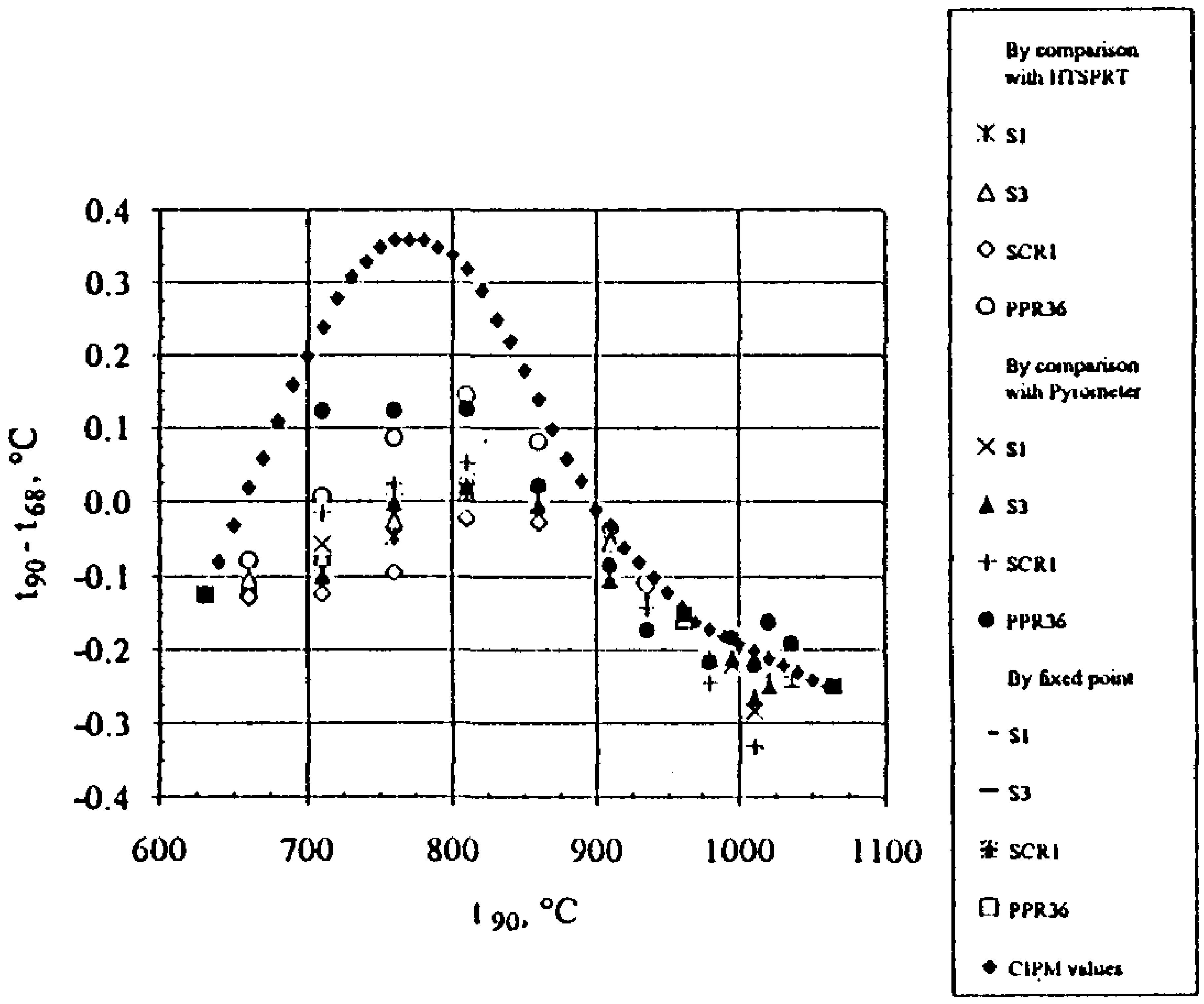

Figure 1. Values of t_{90} - t_{68} computed from IMGC type S thermocouple data compared with previously published CIPM values.

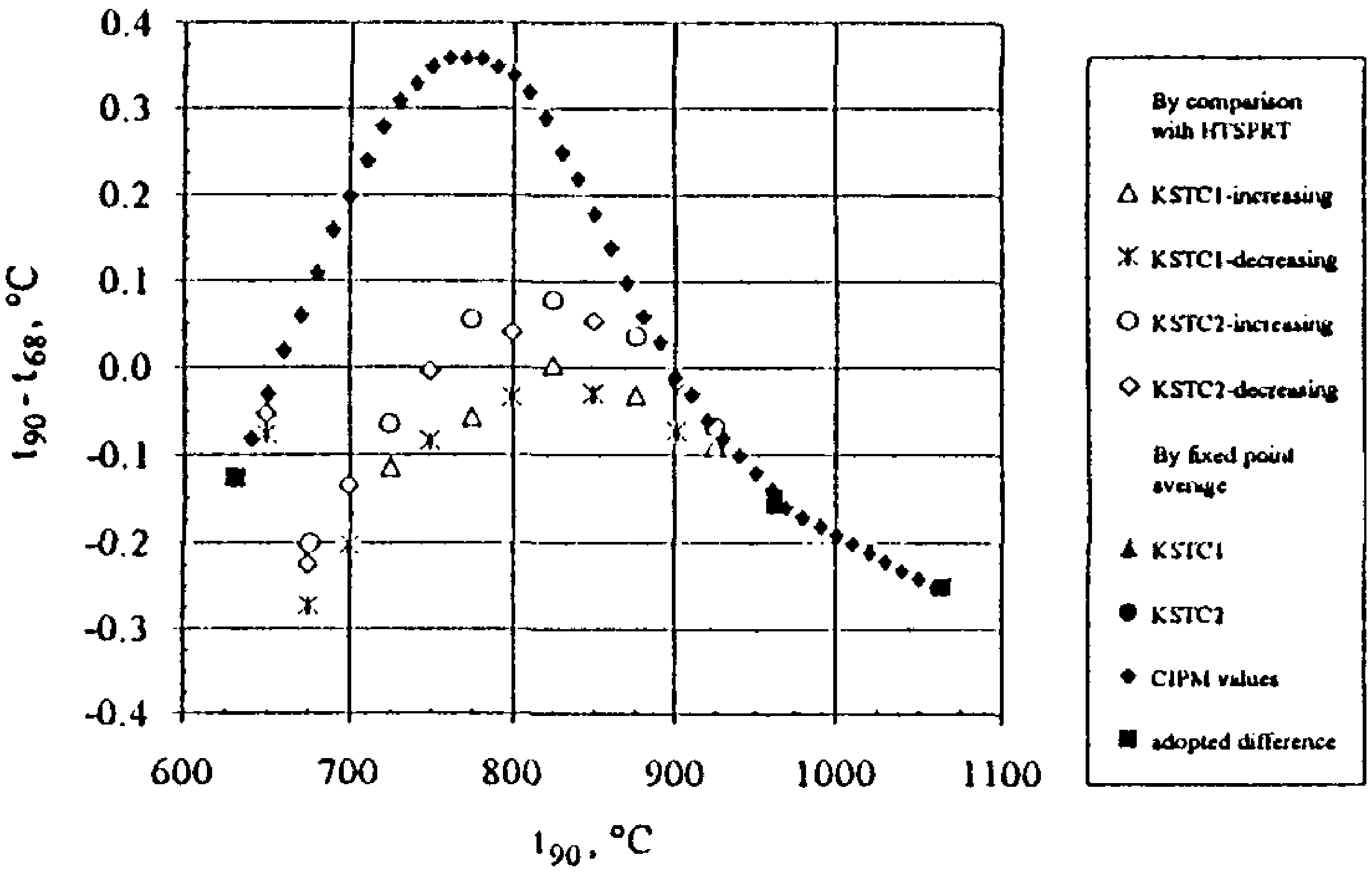

Figure 2. Values of t_{90} - t_{68} computed from KRISS type S thermocouple data compared with previously published CIPM values.

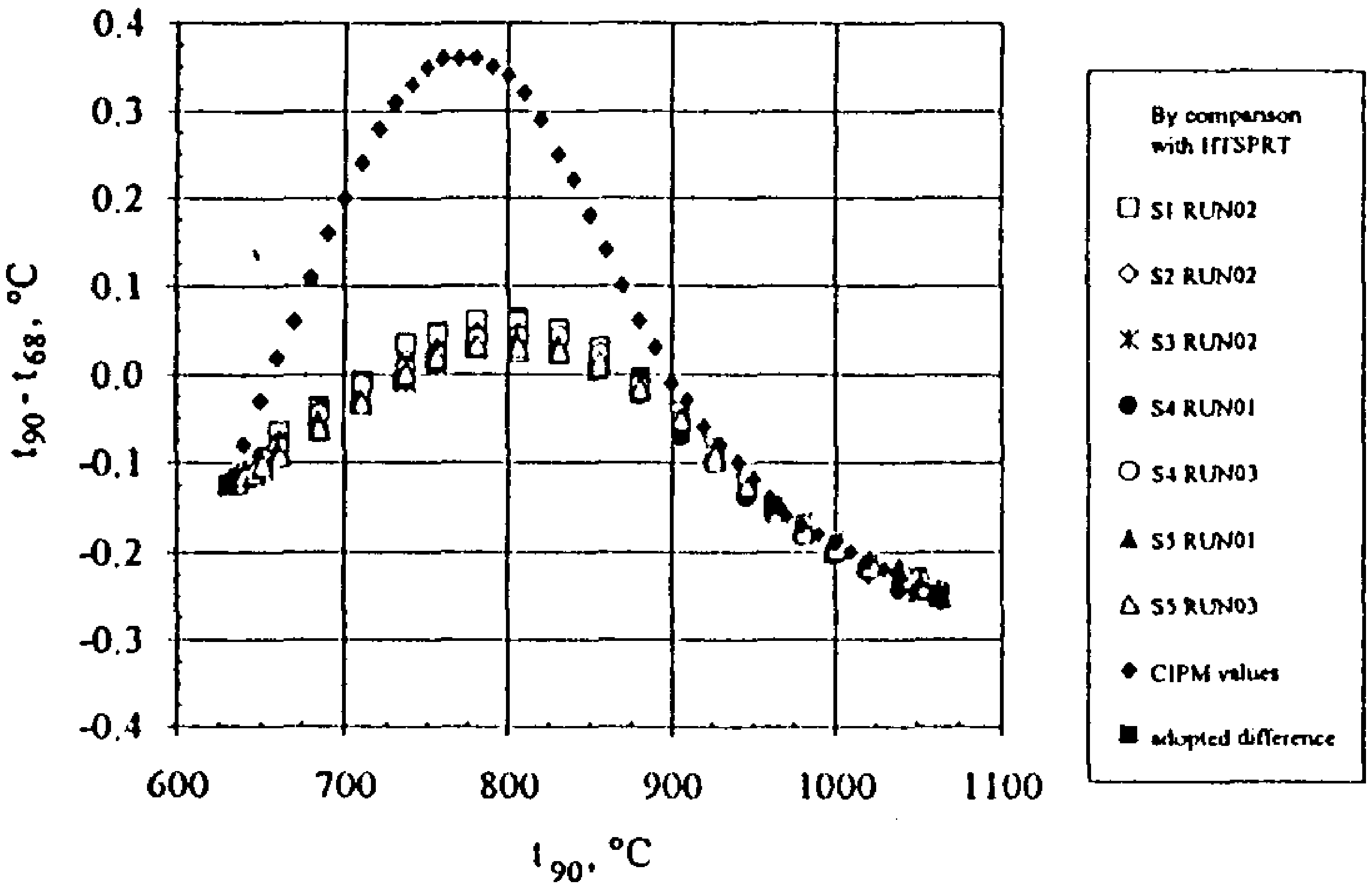

Figure 3. Values of t_{90} - t_{68} computed from NIST type S thermocouple data compared with previously published CIPM values.

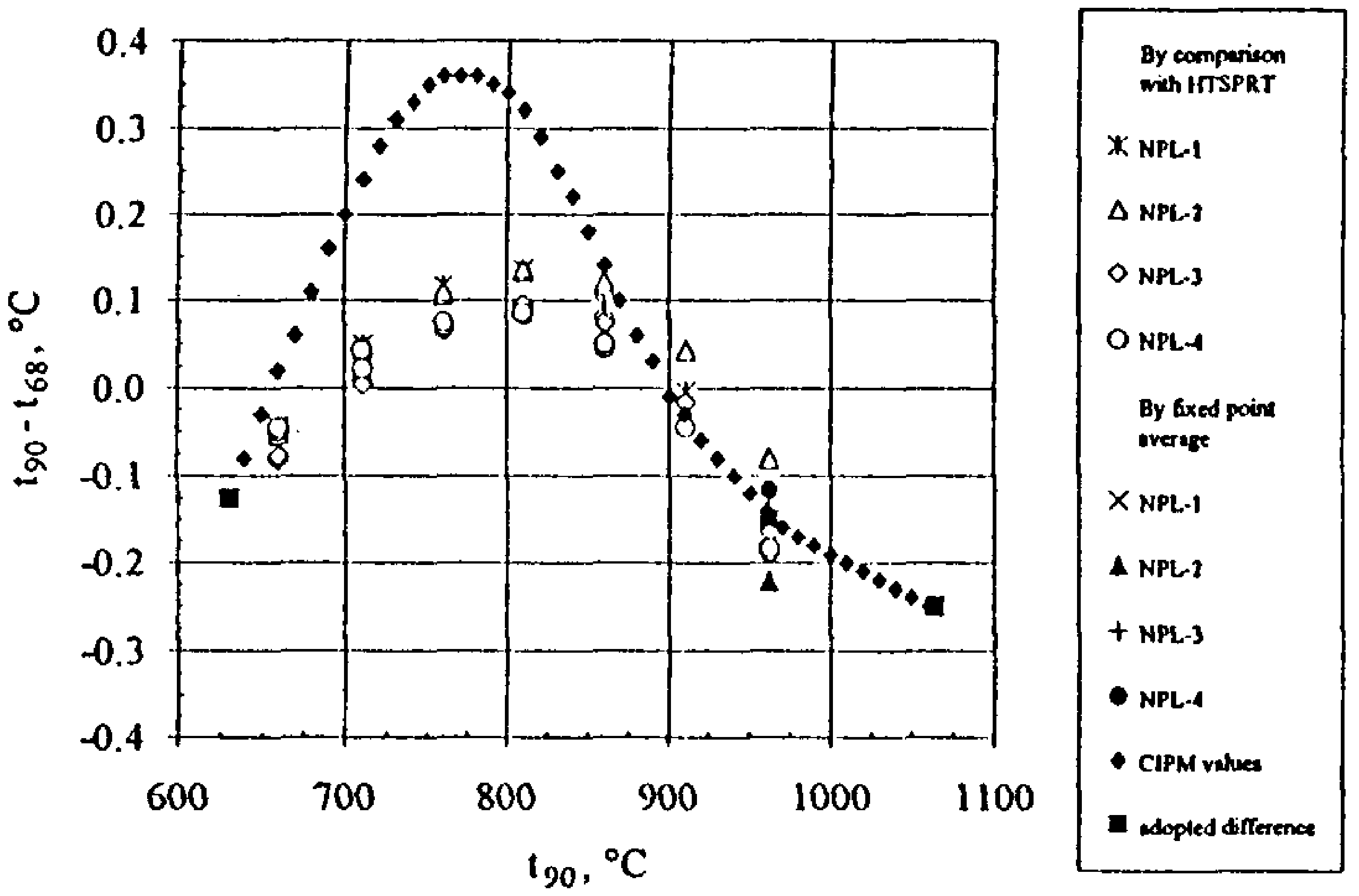

Figure 4. Values of t_{90} - t_{68} computed from NPL type S thermocouple data compared with previously published CIPM values.

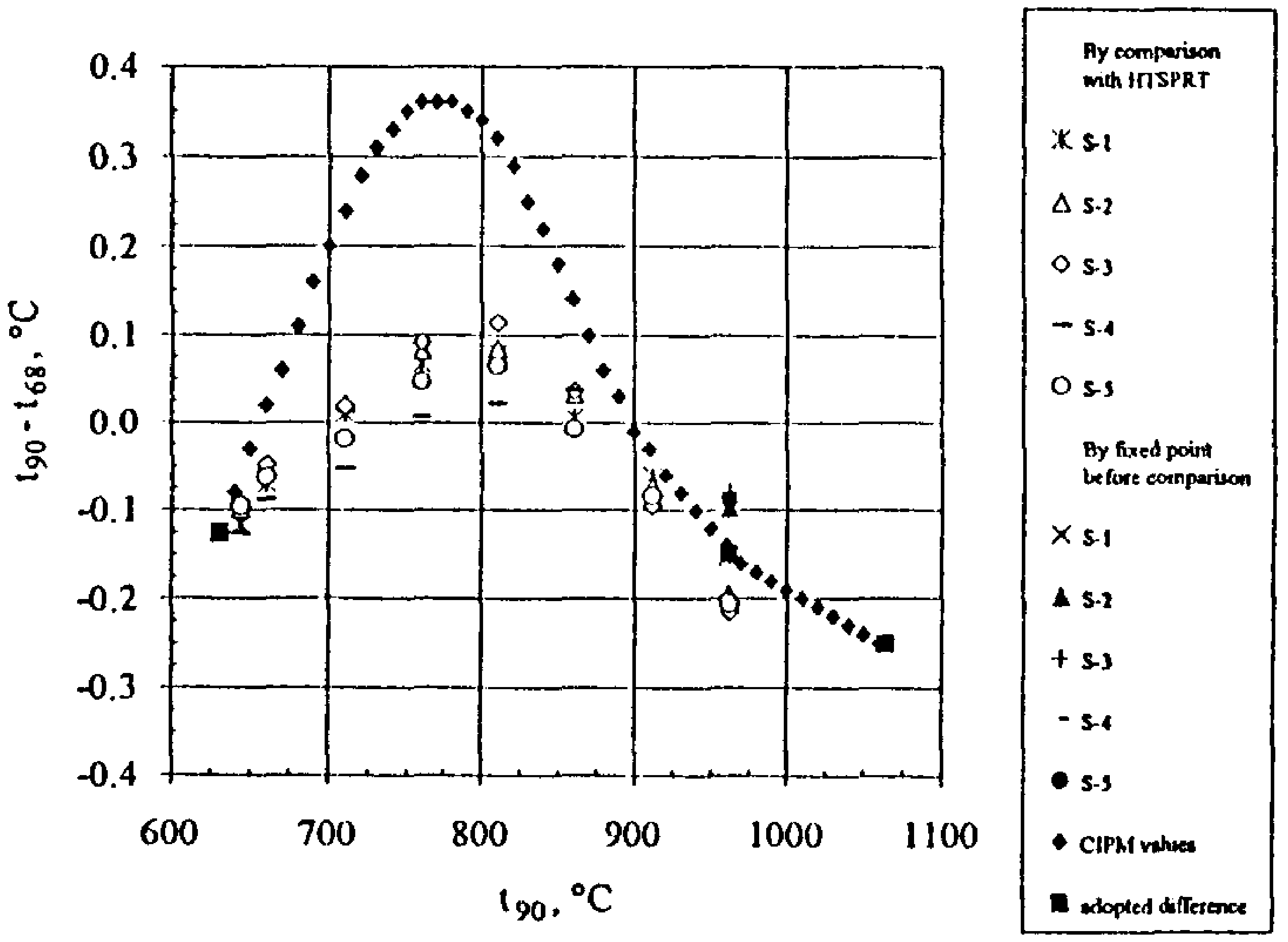

Figure 5. Values of t_{90} - t_{68} computed from NRLM type S thermocouple data compared with previously published CIPM values.

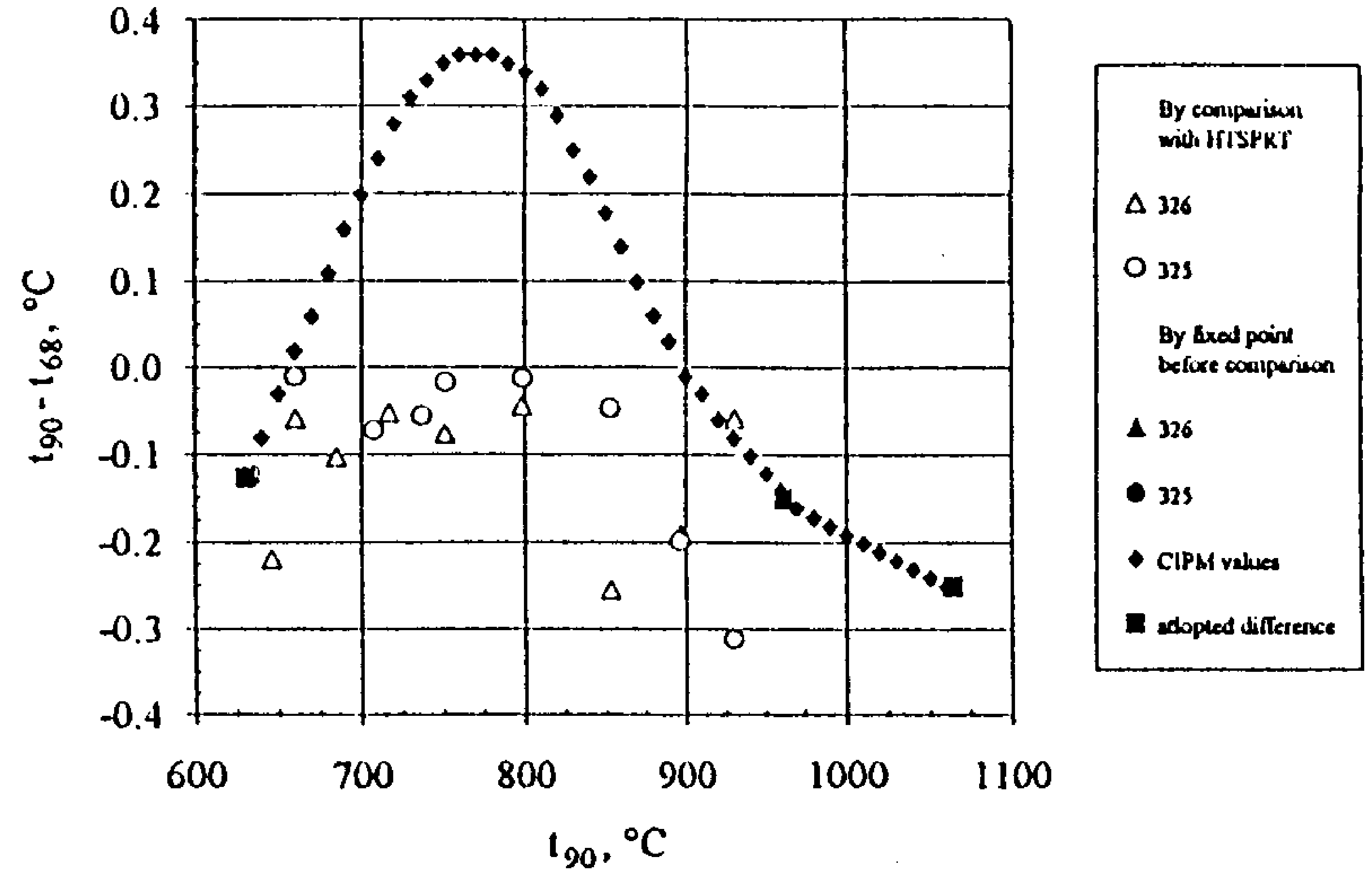

Figure 6. Values of t_{90} - t_{68} computed from VNIIM type S thermocouple data compared with previously published CIPM values.

degree polynomial produces the same values of E, dE/dt_{90}, and d^2E/dt_{90}^2 at 1664.5 °C as the reference function of the preceding range, and it also gives the same value of E at 1768.117 °C that the IPTS-68 based cubic gives at t_{68} = 1767.6 °C, after the latter is corrected to account for the change in the volt.

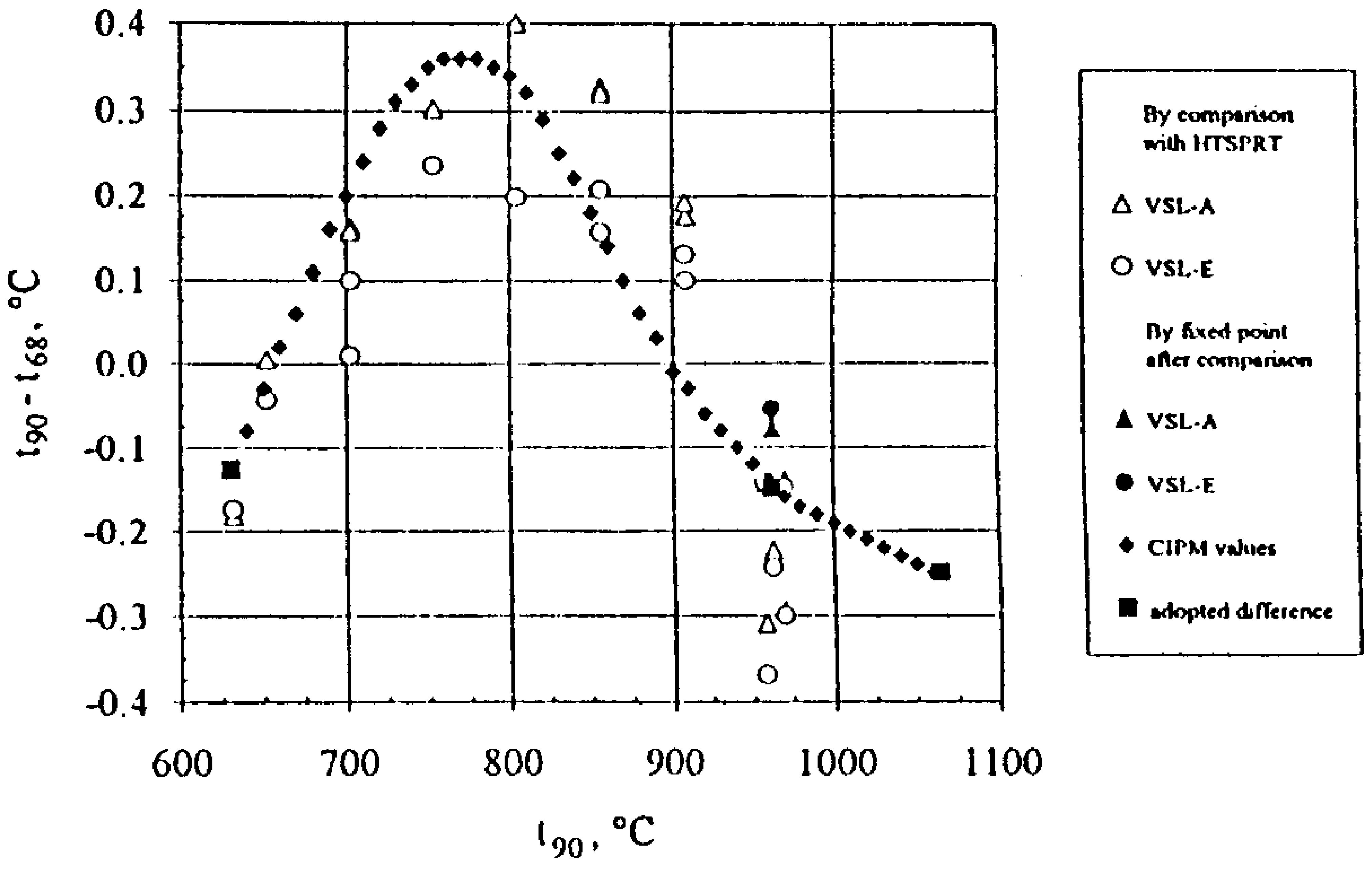

Figure 7. Values of t_{90} - t_{68} computed from VSL type S thermocouple data compared with previously published CIPM values.

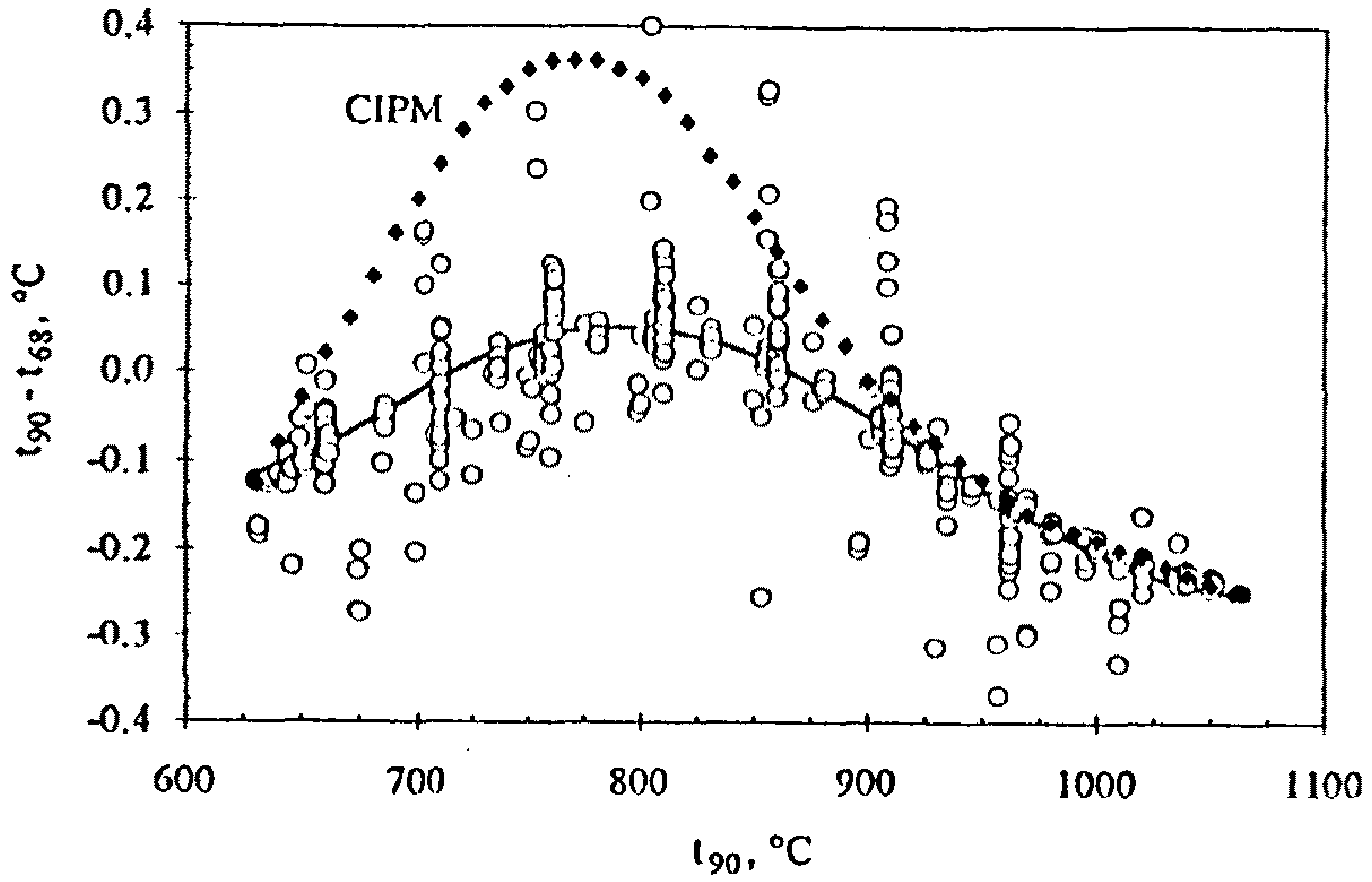

Figure 8. Values of $t_{90} - t_{68}$ computed from type S thermocouple data (open circles) from all of the seven national laboratories compared with previously published CIPM values (solid diamonds). The line represents the 5th degree polynomial fitted to the data by iteratively reweighted least-squares regression.

The new reference functions for the type S thermocouples are of the form:

$$E = \sum_{i=0}^{n} a_i (t_{90})^i , \tag{3}$$

where t_{90} is in degrees Celsius and E is in microvolts. The coefficients of Eq. (3) for the various temperature ranges are given in Table I.

Table I. Coefficients of the reference functions for type S thermocouples for the indicated temperature ranges.

−50 °C to 1064.18 °C		1064.18 °C to 1664.5 °C	
a_1	5.40313308631	a_0	$1.32900444085 \times 10^{3}$
a_2	$1.25934289740 \times 10^{-2}$	a_1	3.34509311344
a_3	$-2.32477968689 \times 10^{-5}$	a_2	$6.54805192818 \times 10^{-3}$
a_4	$3.22028823036 \times 10^{-8}$	a_3	$-1.64856259209 \times 10^{-6}$
a_5	$-3.31465196389 \times 10^{-11}$	a_4	$1.29989605174 \times 10^{-11}$
a_6	$2.55744251786 \times 10^{-14}$		
a_7	$-1.25068871393 \times 10^{-17}$	1664.5 °C to 1768.1 °C	
a_8	$2.71443176145 \times 10^{-21}$		
		a_0	$1.46628232636 \times 10^{5}$
		a_1	$-2.58430516752 \times 10^{2}$
		a_2	$1.63693574641 \times 10^{-1}$
		a_3	$-3.30439046987 \times 10^{-5}$
		a_4	$-9.43223690612 \times 10^{-12}$

Values of E and the first and second derivatives of E with respect to t_{90} computed from the reference functions (see Eq. (3) and Table I) at selected values of t_{90} are given in Table II.

Table II. Values of E and the first and second derivatives of E with respect to t_{90} computed from equation (3) at selected values of t_{90}.

t_{90}, °C	E, μV	dE/dt_{90}, μV/°C	d^2E/dt_{90}^2, nV/°C^2
-38.8344	-189.40	4.312	31.23
0.000	0.00	5.403	25.19
0.01	0.05	5.403	25.19
29.7646	171.39	6.094	21.36
156.5985	1082.27	8.045	10.69
231.928	1715.00	8.711	7.24
419.527	3446.89	9.638	3.50
630.615	5552.64	10.303	3.16
660.323	5860.13	10.398	3.23
961.78	9148.38	11.418	3.22
1064.18	10334.20	11.743	3.27
1084.62	10574.80	11.798	2.55
1664.5	17535.96	11.681	-2.94
1768.1	18693.54	10.311	-23.52

Figure 9 shows the *emf* difference between the new ITS-90 based reference functions and the IPTS-68 based reference functions. The IPTS-68 based reference functions were converted to the ITS-90 and corrected for the change in the volt. The conversion of the IPTS-68 based reference function in the range 630.615 °C to 1064.18 °C is based on Eq. (2). The dashed line in the figure labelled +1 °C is the Seebeck coefficient as a function of t_{90}.

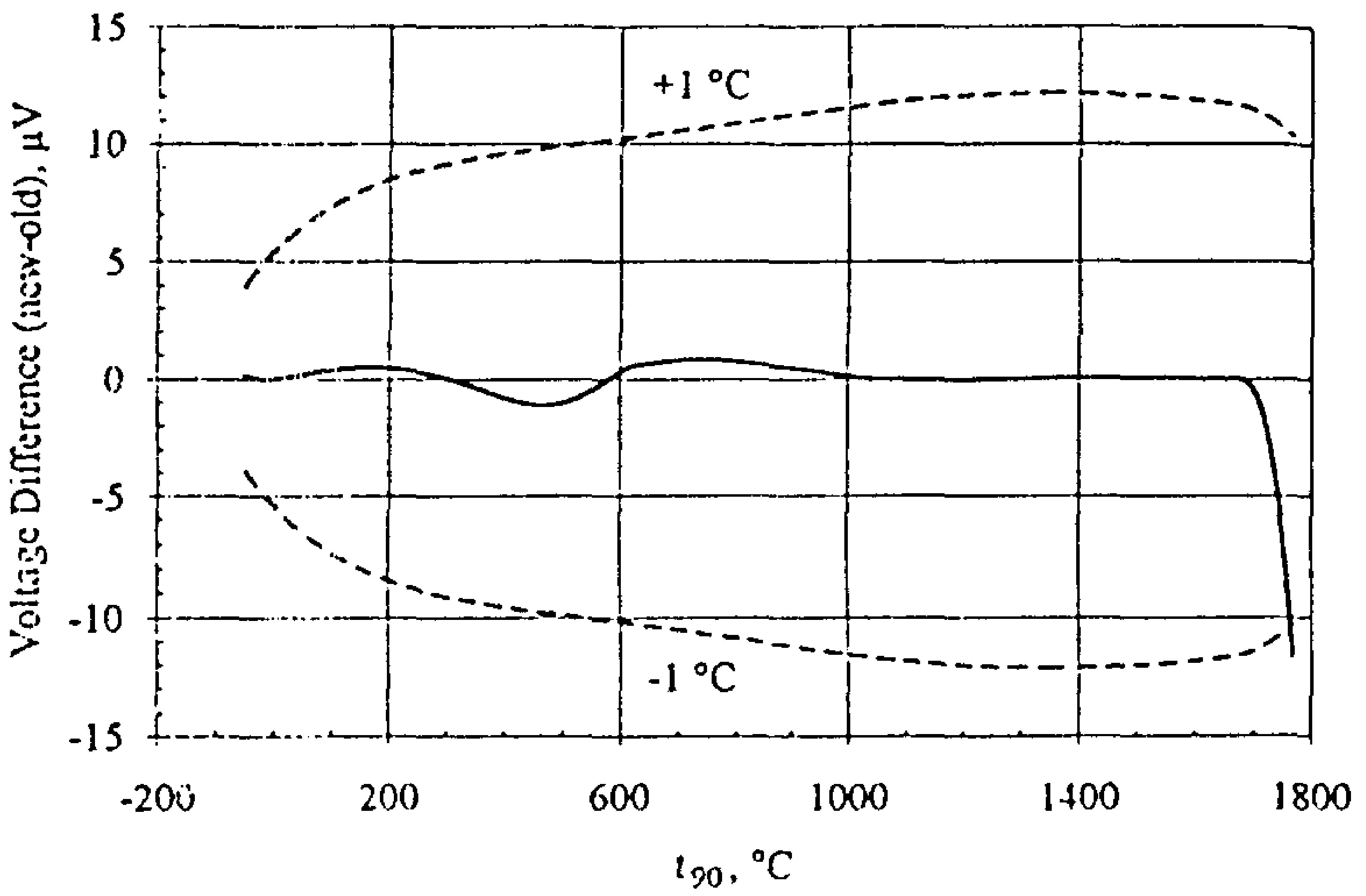

Figure 9. Differences between the new ITS-90 reference functions and the old IPTS-68 reference functions for type S thermocouples. The values of the old type S reference functions are adjusted to the ITS-90 and corrected for the change in the volt. The dashed lines indicate an *emf* deviation equivalent to ± 1 °C.

The deviations of the IMGC, KRISS, NIST, NPL, NRLM, SIPAI, VNIIM, and VSL thermocouple data from the new reference function are shown in Figs. 10, 11, 12, 13, 14, 15, 16, and 17, respectively. The dashed lines in the figures, which indicate an *emf* deviation equivalent to ±1 °C, represent the Class 1 manufacturing tolerance for type S thermocouples as given in IEC standard 584-2 (21). Twenty eight of the 37 thermocouples used in this effort satisfy the Class 1 tolerance.

Since the reference functions given above are not well suited for calculating values of temperature from values of *emf*, a set of inverse functions, based on the ITS-90, is included here for that purpose. These inverse functions give values of temperature that agree with values obtained from the respective reference function to at least ±0.02 °C. The inverse functions are of the form:

$$t_{90} = \sum_{i=0}^{n} b_i (E)^i \, , \tag{4}$$

where t_{90} is in degrees Celsius and E is given in microvolts. The coefficients of Eq. (4) for various temperature and *emf* ranges are given in Table III.

Table III. Coefficients of inverse functions for the type S thermocouples for the indicated temperature and *emf* ranges.

	−50 °C to 250 °C −236 μV to 1874 μV		250 °C to 1200 °C 1874 μV to 11951 μV
b_1	$1.84949460 \times 10^{-1}$	b_0	$1.291507177 \times 10^{1}$
b_2	$-8.00504062 \times 10^{-5}$	b_1	$1.466298863 \times 10^{-1}$
b_3	$1.02237430 \times 10^{-7}$	b_2	$-1.534713402 \times 10^{-5}$
b_4	$-1.52248592 \times 10^{-10}$	b_3	$3.145945973 \times 10^{-9}$
b_5	$1.88821343 \times 10^{-13}$	b_4	$-4.163257839 \times 10^{-13}$
b_6	$-1.59085941 \times 10^{-16}$	b_5	$3.187963771 \times 10^{-17}$
b_7	$8.23027880 \times 10^{-20}$	b_6	$-1.291637500 \times 10^{-21}$
b_8	$-2.34181944 \times 10^{-23}$	b_7	$2.183475087 \times 10^{-26}$
b_9	$2.79786260 \times 10^{-27}$	b_8	$-1.447379511 \times 10^{-31}$
		b_9	$8.211272125 \times 10^{-36}$

	1064 °C to 1664.5 °C 10332 μV to 17536 μV		1664.5 °C to 1768.1 °C 17536 μV to 18694 μV
b_0	$-8.087801117 \times 10^{1}$	b_0	$5.333875126 \times 10^{4}$
b_1	$1.621573104 \times 10^{-1}$	b_1	$-1.235892298 \times 10^{1}$
b_2	$-8.536869453 \times 10^{-6}$	b_2	$1.092657613 \times 10^{-3}$
b_3	$4.719686976 \times 10^{-10}$	b_3	$-4.265693686 \times 10^{-8}$
b_4	$-1.441693666 \times 10^{-14}$	b_4	$6.247205420 \times 10^{-13}$
b_5	$2.081618890 \times 10^{-19}$		

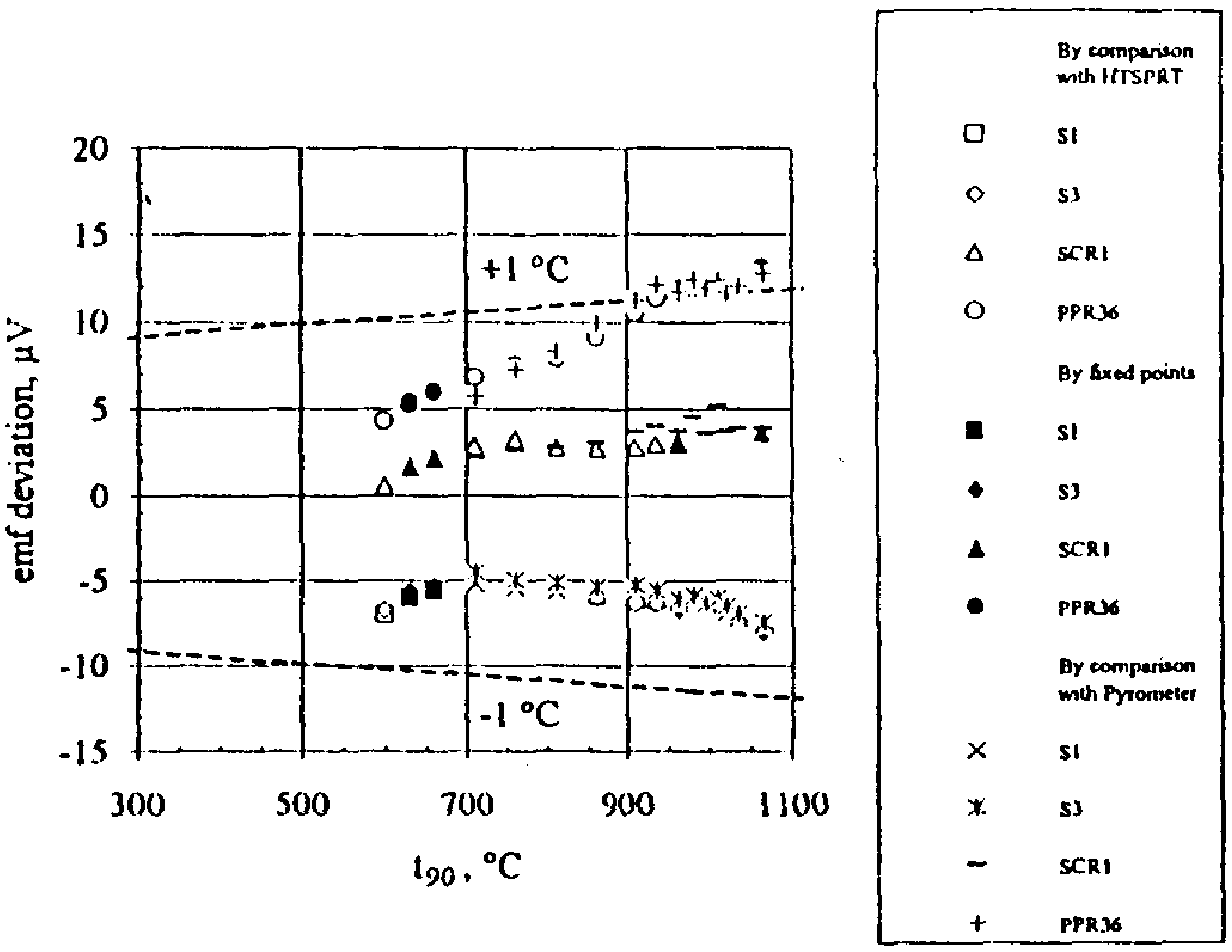

Figure 10. Deviations of IMGC type S thermocouple data from the reference function (deviation = measured *emf* values - reference function). The dashed lines indicate an *emf* deviation equivalent to ±1 °C.

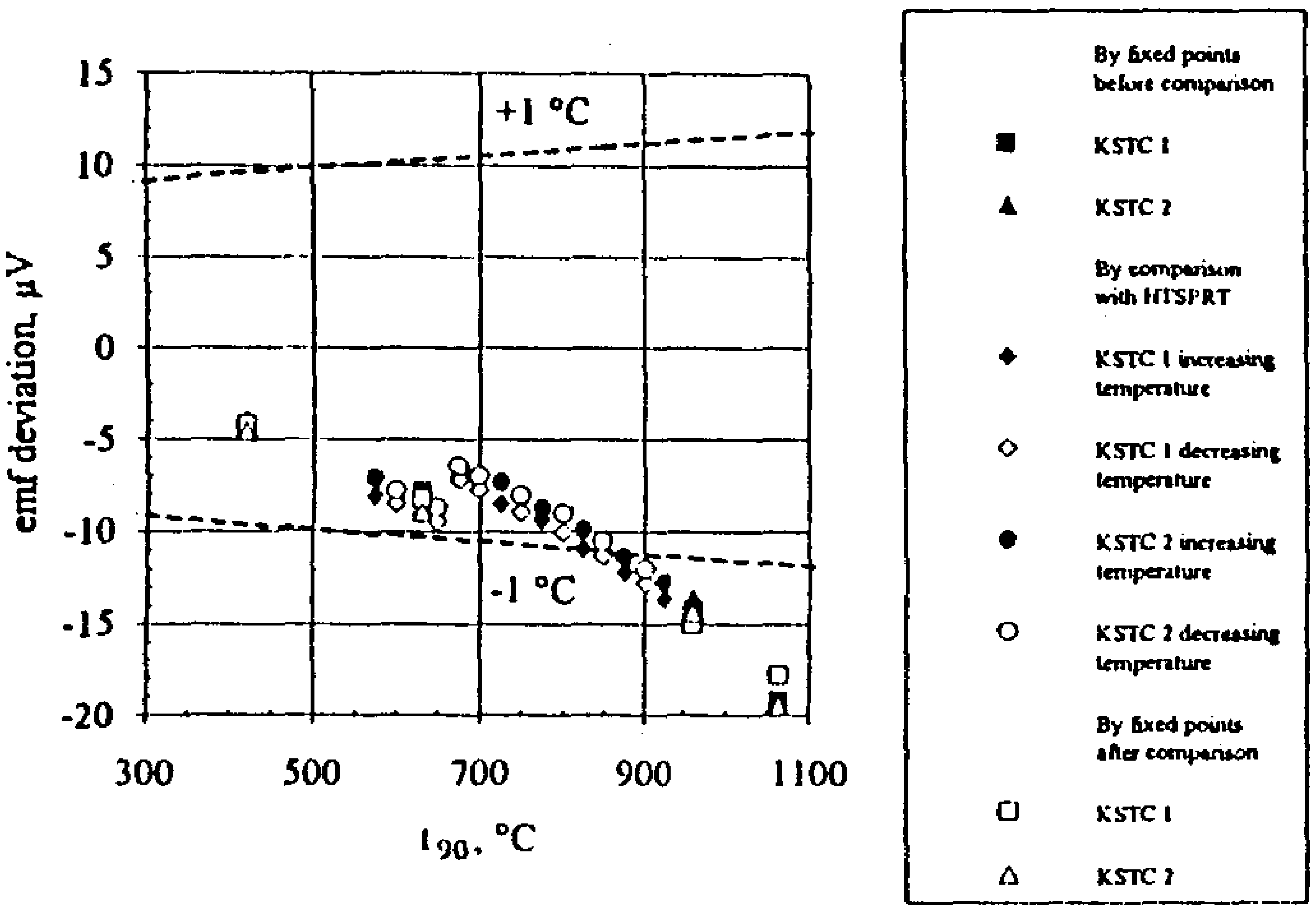

Figure 11. Deviations of KRISS type S thermocouple data from the reference function (deviation = measured *emf* values - reference function). The dashed lines indicate an *emf* deviation equivalent to ±1 °C.

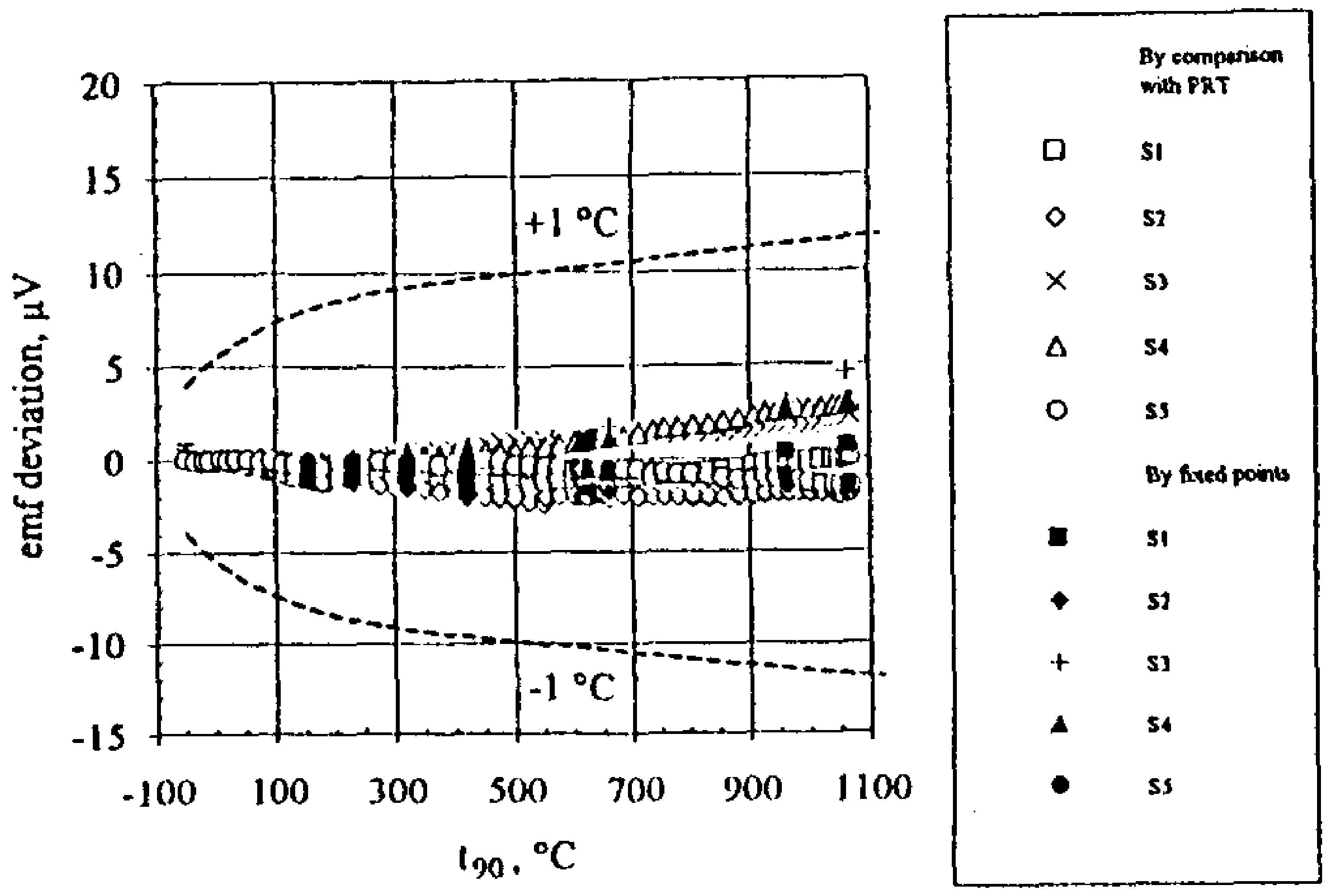

Figure 12. Deviations of NIST type S thermocouple data from the reference function (deviation = measured *emf* values - reference function). The dashed lines indicate an *emf* deviation equivalent to ±1 °C.

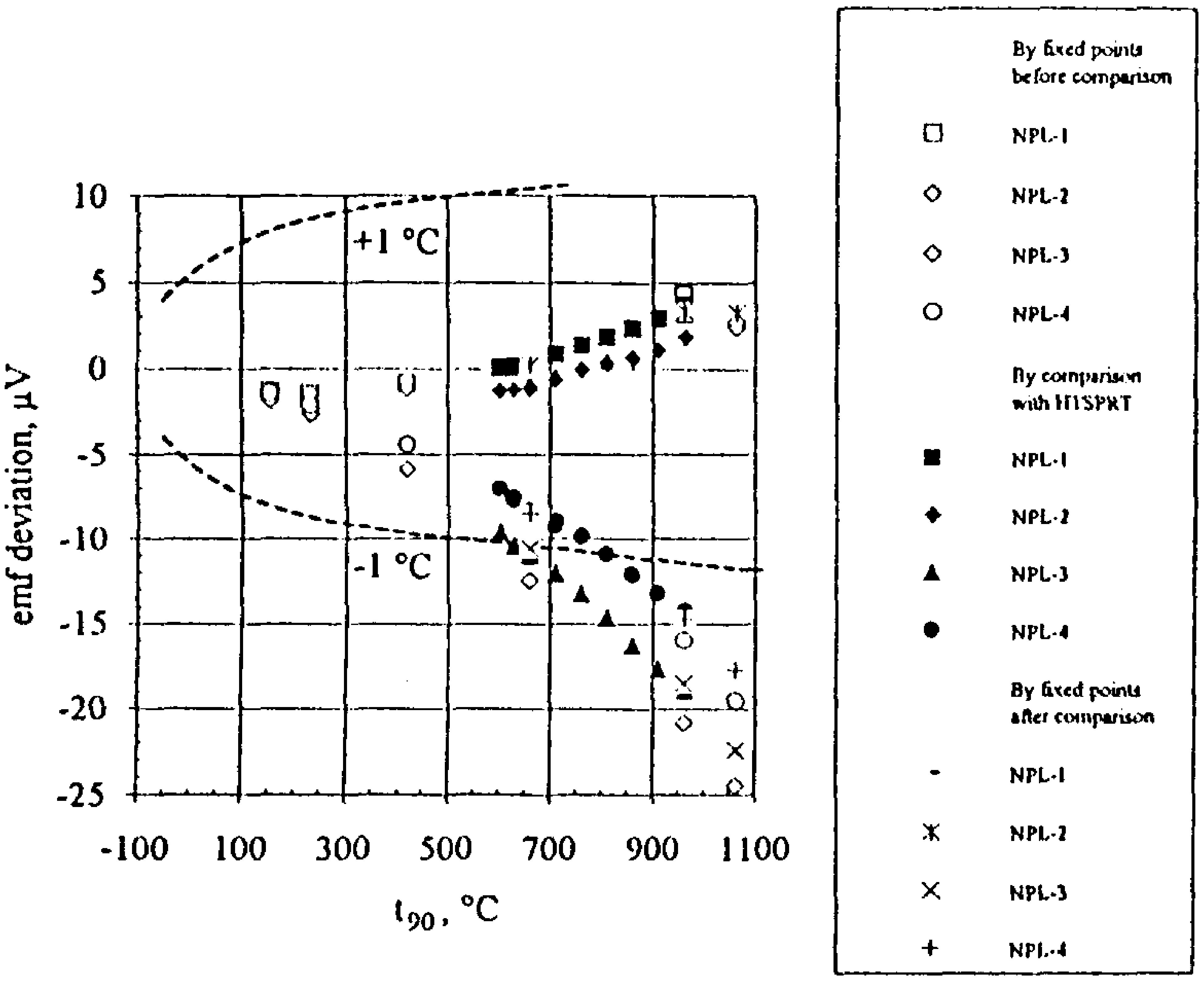

Figure 13. Deviations of NPL type S thermocouple data from the reference function (deviation = measured *emf* values - reference function). The dashed lines indicate an *emf* deviation equivalent to ±1 °C.

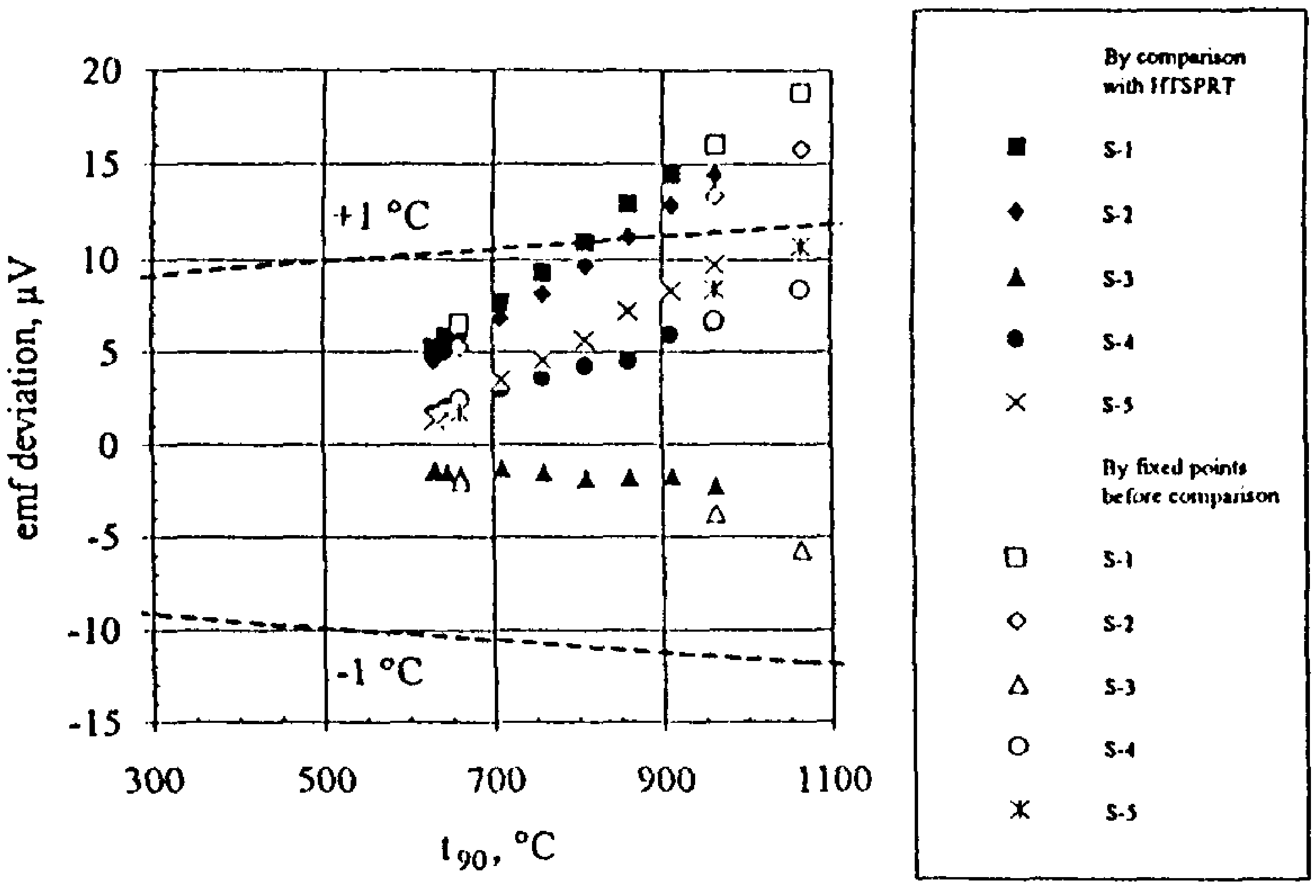

Figure 14. Deviations of NRLM type S thermocouple data from the reference function (deviation = measured *emf* values - reference function). The dashed lines indicate an *emf* deviation equivalent to ±1 °C.

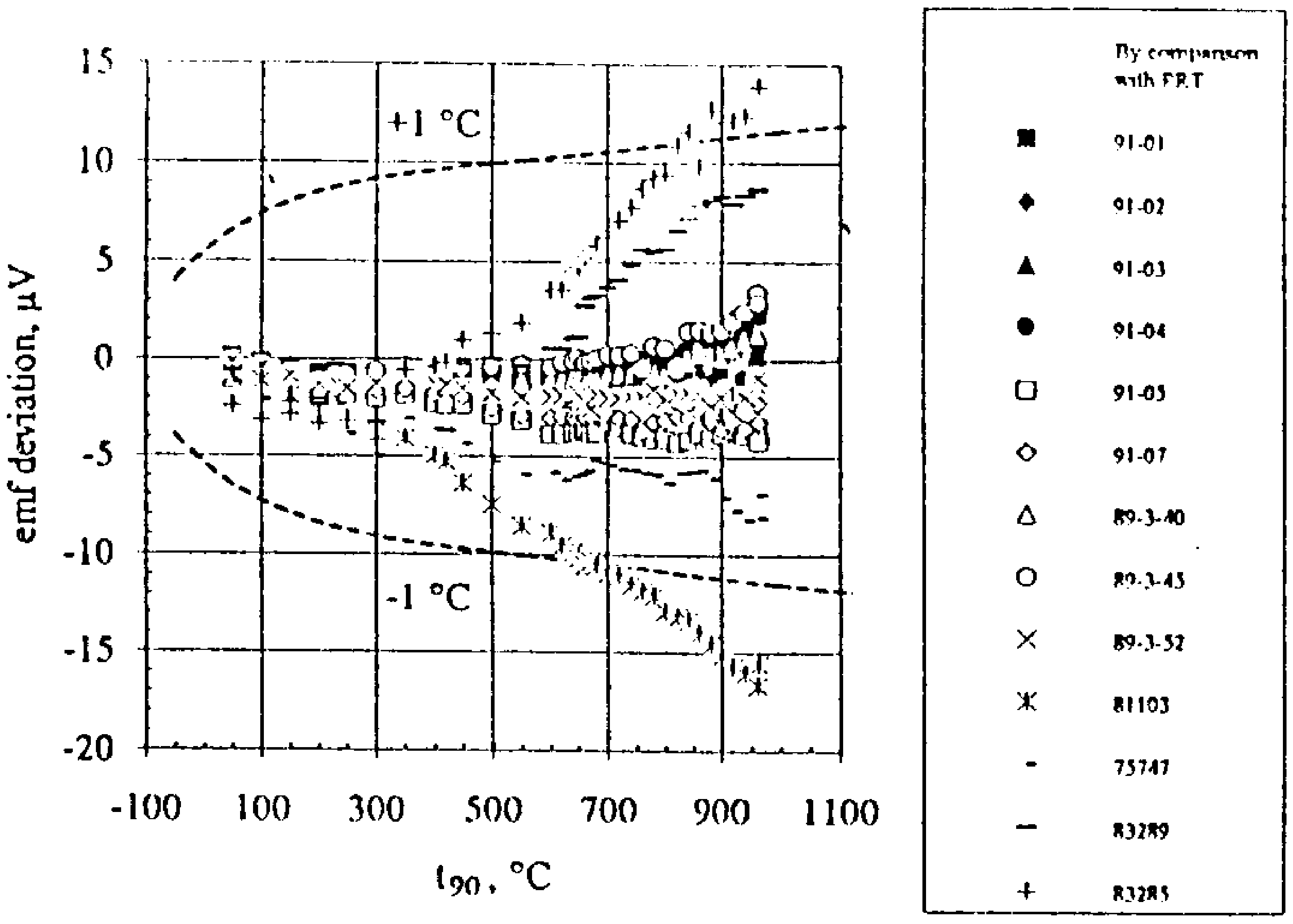

Figure 15. Deviations of SIPAI type S thermocouple data from the reference function (deviation = measured *emf* values - reference function). The dashed lines indicate an *emf* deviation equivalent to ± 1 °C.

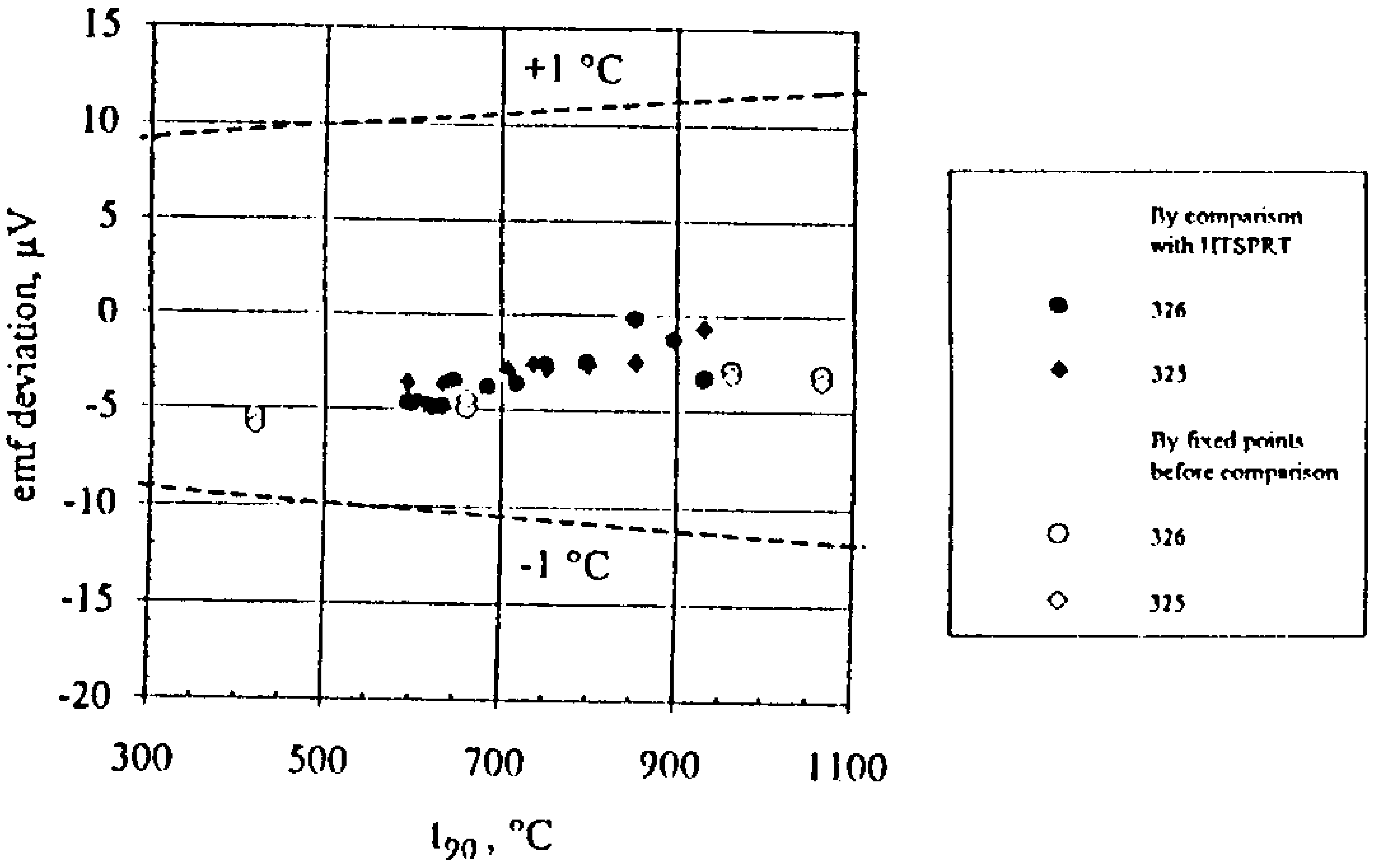

Figure 16. Deviations of VNIIM type S thermocouple data from the reference function (deviation = measured *emf* values - reference function). The dashed lines indicate an *emf* deviation equivalent to ± 1 °C.

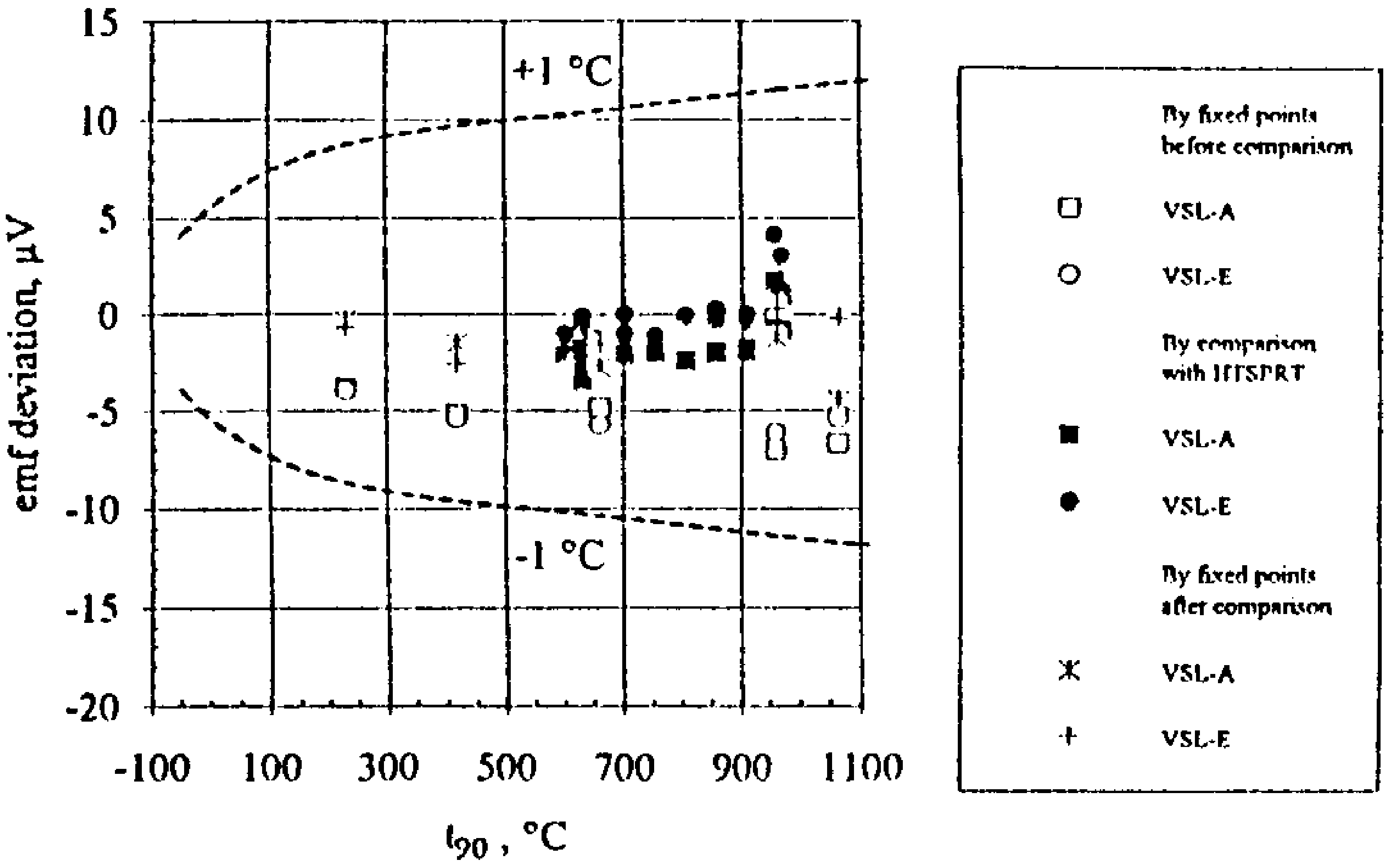

Figure 17. Deviations of VSL type S thermocouple data from the reference function (deviation = measured *emf* values - reference function). The dashed lines indicate an *emf* deviation equivalent to ± 1 °C.

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Statistical analysis of type S thermocouple measurements on the International Temperature Scale of 1990

W. F. Guthrie, M. C. Croarkin, G. W. Burns, and G. F. Strouse
National Institute of Standards and Technology (NIST), Gaithersburg, Maryland 20899

P. Marcarino and M. Battuello
Istituto di Metrologia 'G. Colonnetti' (IMGC), Torino, Italy

H. K. Lee, J. C. Kim, K. S. Gam, and C. Rhee
Korea Research Institute of Standards and Science (KRISS), Taejon, Republic of Korea

M. Chattle
National Physical Laboratory (NPL), Teddington, United Kingdom

M. Arai and H. Sakurai
National Research Laboratory of Metrology (NRLM), Ibaraki, Japan

A. I. Pokhodun, N. P. Moiseeva, and S. A. Perevalova
D. I. Mendeleyev Institute for Metrology (VNIIM), St. Petersburg, Russia

M. J. de Groot
Van Swinden Laboratory (VSL), Delft, The Netherlands

Jipei Zhang, Kai Fan, and Shuyuan Wu
Shanghai Institute of Process Automation Instrumentation (SIPAI), Shanghai, China

Data collected by scientists from eight national laboratories were used to estimate the differences between temperatures on the International Temperature Scale of 1990 and the International Practical Temperature Scale of 1968, Amended Edition of 1975 between 630.615 °C and 1064.18 °C. A new reference function for type S thermocouples was determined for the temperature range −50 °C to 1064.18 °C. The new reference function was combined with two other reference functions for temperatures between 1064.18 °C and 1768.1 °C to provide a complete set of reference functions on ITS-90 for type S thermocouples. This paper describes the modeling procedures used to determine the reference functions and statistical analyses used to estimate differences between the two temperature scales. Issues addressed include: variability within laboratories, form of the new reference functions, and the uncertainty associated with the reference functions.

INTRODUCTION

This paper describes how type S thermocouple measurement data were used to estimate the difference between temperatures on the International Temperature Scale of 1990 (ITS-90) (1) and the International Practical Temperature Scale of 1968, Amended Edition of 1975 (IPTS-68) (2) for temperatures in the range 630.615 °C to 1064.18 °C. The development of a new reference function for type S thermocouples for the temperature range −50 °C to 1064.18 °C is also described. The data were collected by eight national laboratories as part of an international experiment to develop ITS-90 based thermocouple reference functions (3).

Throughout the paper, t_{90} and t_{68} denote temperatures measured in degrees Celsius on the ITS-90 and IPTS-68, respectively. The difference between the two scales is denoted by $\Delta t = t_{90} - t_{68}$.

ESTIMATION OF $\Delta t = t_{90} - t_{68}$

The data used for estimating the temperature differences between the ITS-90 and the IPTS-68 are shown in Figure 1. Data from each lab are denoted by a unique symbol. The temperature differences were obtained by taking the differences of the temperatures measured directly on the ITS-90 and the corresponding IPTS-68 temperatures calculated using the associated emf measurement and the IPTS-68 defining quadratic function for each thermocouple. The details of the temperature difference computations are discussed in (3).

Figure 1 makes it clear that there are systematic errors between sets of temperature differences derived from measurements made by different laboratories using different thermocouples. In addition to the systematic errors in each set of temperature differences there are, of course, random measurement errors as well.

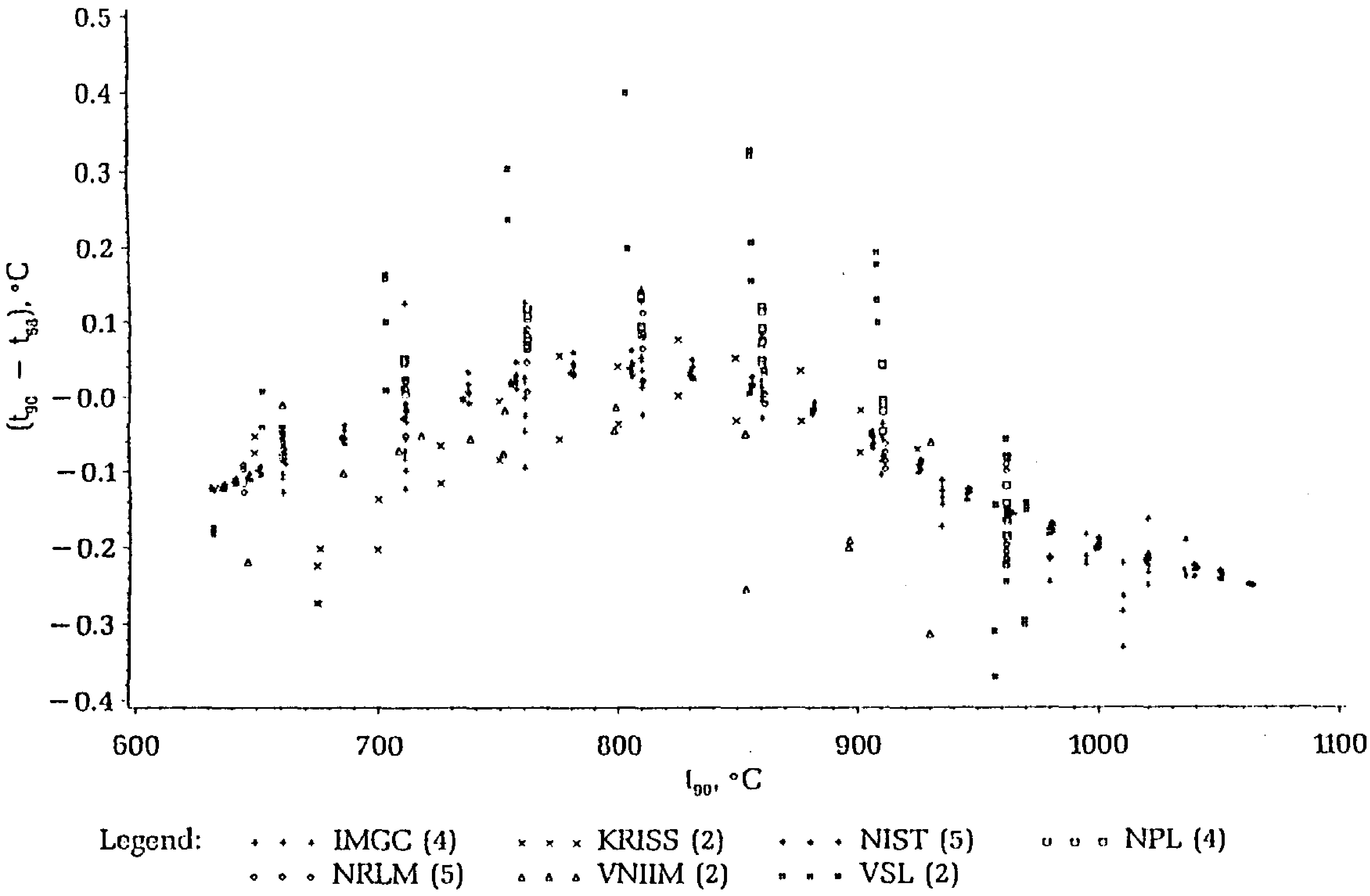

Figure 1: Measured temperature differences vs. t_{90}. The number of thermocouples tested by each laboratory is shown in parentheses.

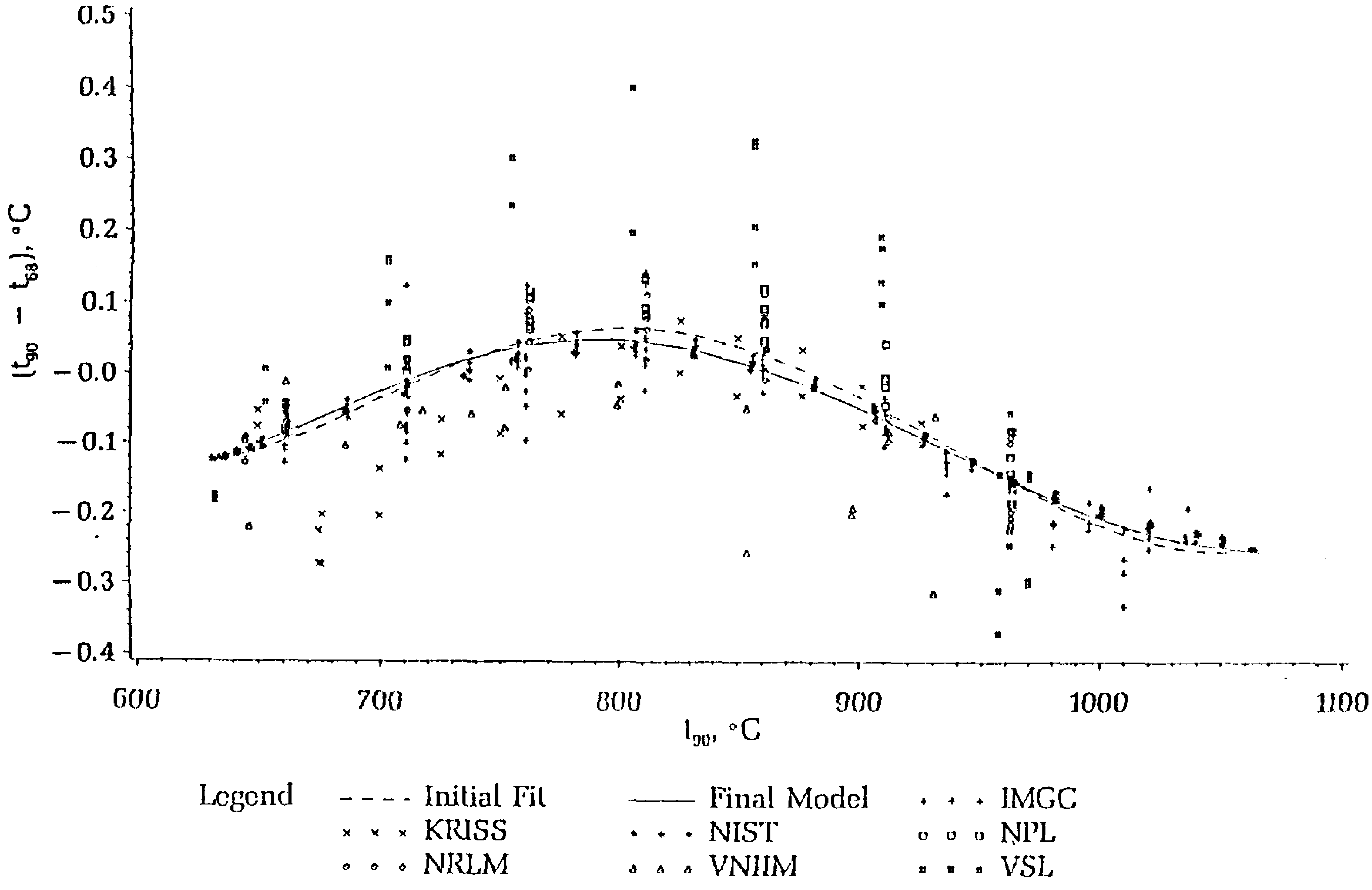

Figure 2: Measured temperature differences, initial model, and final model vs. t_{90}.

Because the data have a complicated error structure, and because there is no way to determine which set of temperature differences is closest to the actual difference between the two temperature scales, the difference between the scales is estimated by the consensus of data from different sources. In this paper, we assume that the systematic errors associated with each lab/thermocouple combination are randomly distributed across laboratories and thermocouples, and that the distribution of the systematic errors is centered at zero. Under these assumptions, the consensus value gives an unbiased estimate of the difference between temperatures on the ITS-90 and the IPTS-68.

To develop a consensus model for the difference between temperatures on the two scales, we fitted a 5^{th} degree polynomial in t_{90} to the temperature difference data using least squares regression. The polynomial model was chosen because no theoretical model relating Δt to t_{90} is known. The 5^{th} degree polynomial provided a reasonable summary of the data. Polynomials of lower degree did not adequately describe the difference in temperatures on the two scales as a function of t_{90}. Polynomials of higher degree did not significantly improve the fit to the data, so use of additional terms in the model was unwarranted. The regression function is shown in Figure 2 and is labeled 'Initial Model' in the legend.

To reduce the influence of isolated data points on the estimation procedure, we standardized the residuals and then weighted each point in the data set using weights inversely related to the magnitude of the standardized residual. Then, we refitted the model using weighted least squares regression to obtain estimates of the regression parameters that were less prone to effects of outlying data. This process of weighting the residuals after fitting the model and then refitting the model was repeated until the regression function converged to a final model, which better reflects the consensus of the data than the initial model does. This repeated fitting/weighting/fitting procedure is known as iteratively reweighted least squares regression (IRLS regression) in statistical literature (4).

Equation 1 shows how the i^{th} standardized residual is computed for the j^{th} iterative fit.

$$u_{i,j} = \frac{d_i - \hat{d}_{i,j}}{cS_j} \tag{1}$$

The standardized residuals are denoted by $u_{i,j}$, the measured temperature difference by d_i, and the predicted temperature difference by $\hat{d}_{i,j}$. The denominator of $u_{i,j}$ is the product of a tuning constant, c, and S_j, a robust measure of the variability of the residuals. The median of the absolute residuals was used as the measure of residual variability, S_j, for this analysis. The tuning constant chosen was $c = 6$. For this experiment $i = 1, 2, \ldots, 466$ and $j = 1, 2, \ldots, 5$.

We used Tukey's biweight function (4) to compute the weights for each data point.

$$w(u_{i,j}) = \begin{cases} (1 - u_{i,j}^2)^2 & \text{if } |u_{i,j}| \leq 1 \\ 0 & \text{otherwise} \end{cases} \tag{2}$$

The biweight function produces weights between 0 and 1. Observations near the regression function receive a weight close to 1 and the weights progressively decrease as the observations get further away from the regression function. Points that are more than c units away from the regression function (measured in scale units of size S_j) receive a weight of 0 and are omitted from the next model fit. However, these points could receive positive weight again in a later step.

Figures 3 and 4 show some of the details of the IRLS regression analysis. Figure 3 shows the weights that were used for the final fit. In the final fit, 414 out of 466 data points had weights greater than zero.

Figure 4 shows the convergence of the model, as measured by the sample residual standard deviation, to its final form. The sample residual standard deviation from each fit is plotted versus iteration number in this figure. The sample residual standard deviation from the initial fit is inflated by outlying points in the data set. As the data are reweighted in successive steps, the residual standard deviation shrinks towards a value which more accurately reflects the random measurement error in the data. When all of the data have been appropriately weighted, the sample residual standard deviation remains unaltered by further reweighting and refitting. In this experiment, the initial model had a residual standard deviation of 62.6 m°C. After five iterations, the residual standard deviation converged to 17.5 m°C.

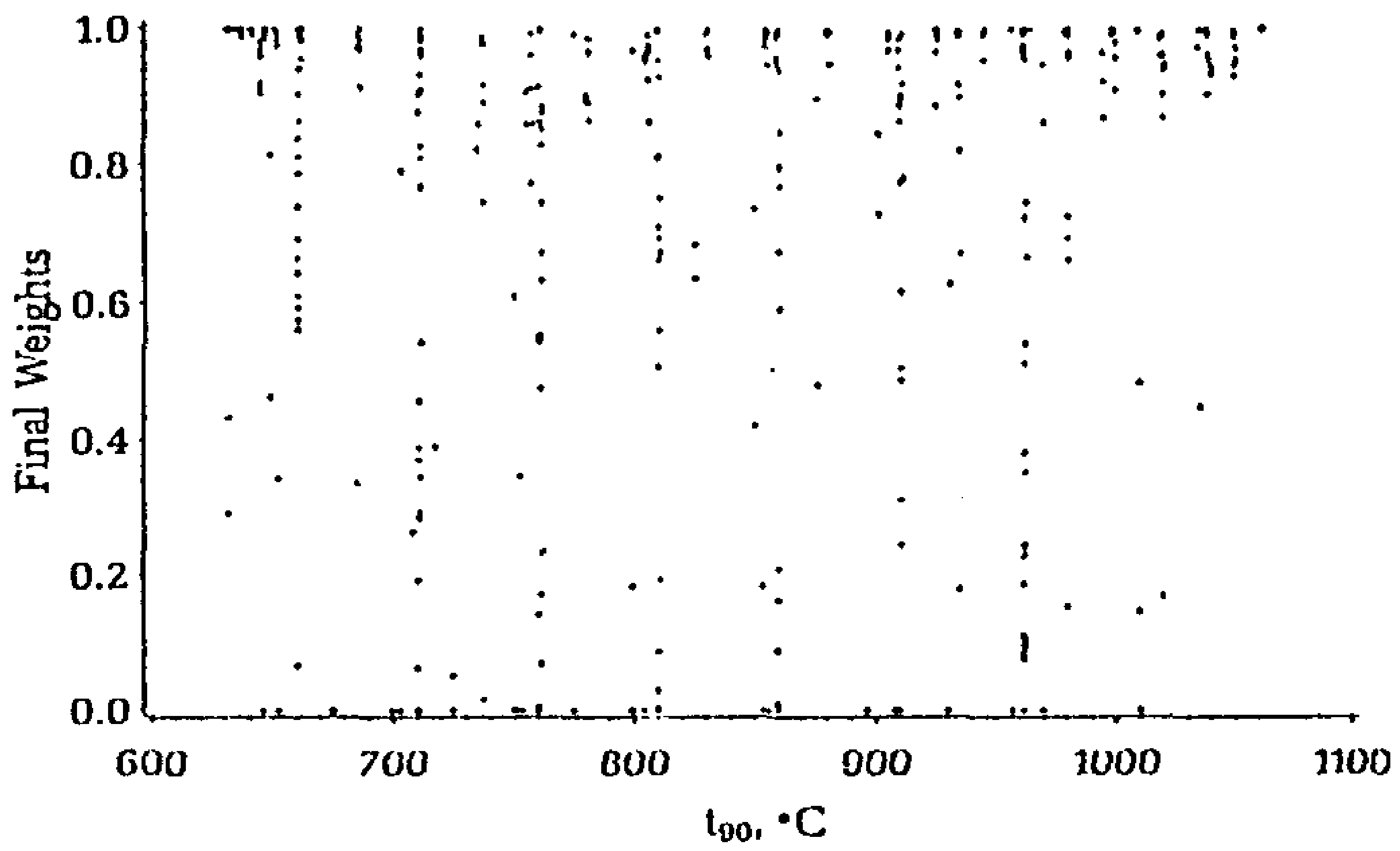

Figure 3: Weights used to fit the final model.

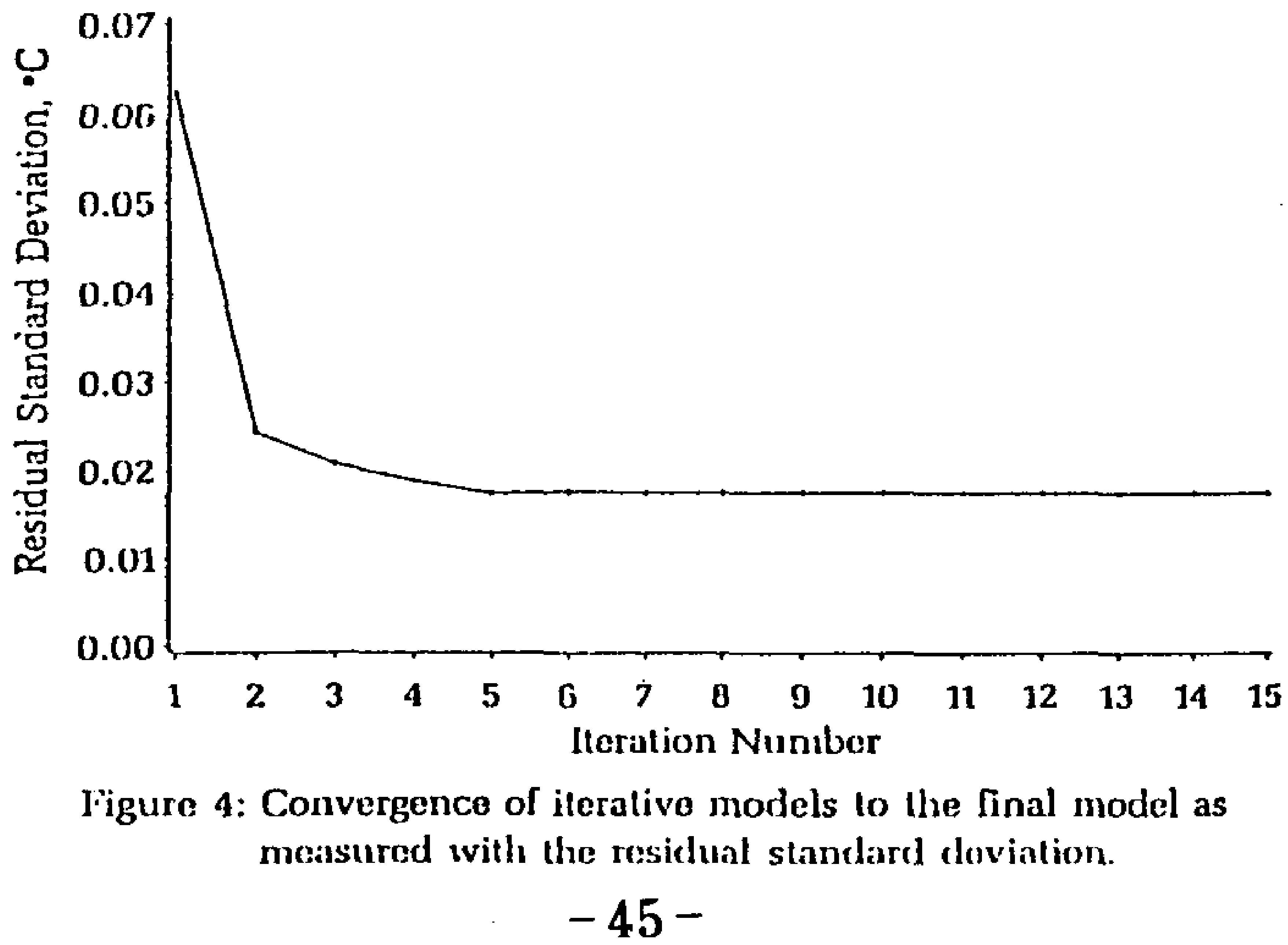

Figure 4: Convergence of iterative models to the final model as measured with the residual standard deviation.

As mentioned above, the final regression function should describe the consensus of data and should not be affected by outlying data points in the data set. The final model from the iteratively reweighted least squares analysis is also shown in Figure 2. Comparing the initial and final models with the data from the the different laboratories makes it clear that the iterative weighting and fitting reduced the influence of the data isolated from the bulk of the measured temperature differences.

In addition to using regression analysis techniques that reduce the effect of outliers in the data, the function $\Delta t(t_{90})$ was constrained to match the adopted temperature differences (5) of −0.125 °C, −0.15 °C, and −0.25 °C at t_{90} = 630.615 °C, t_{90} = 961.78 °C, and t_{90} = 1064.18 °C, respectively. $\Delta t(t_{90})$ matches the adopted temperature differences to at least 0.1 m°C at these three points.

$\Delta t(t_{90})$ was not constrained to have the same first derivative (or slope) that the published scale difference (1,5) has at either t_{90} = 630.615 °C or at t_{90} = 1064.18 °C. The discontinuity in the first derivative at t_{90} = 630.615 °C is approximately 0.14%. This discontinuity is smaller than the 0.51% reported in (6), but this estimate of the discontinuity has a magnitude that is similar to estimates from earlier experiments (7,8,9). The discontinuity at t_{90} = 1064.18 °C is about 0.008%, which is negligible relative to the errors in the determination of $\Delta t(t_{90})$.

The final regression model which describes the temperature differences between the ITS-90 and the IPTS-68 is

$$\begin{aligned}\Delta t(t_{90}) \;=\; & (7.8687209 \times 10^{1}) \\ & -(4.7135991 \times 10^{-1})t_{90} \\ & +(1.0954715 \times 10^{-3})t_{90}^{2} \\ & -(1.2357884 \times 10^{-6})t_{90}^{3} \\ & +(6.7736583 \times 10^{-10})t_{90}^{4} \\ & -(1.4458081 \times 10^{-13})t_{90}^{5}.\end{aligned} \tag{3}$$

Figure 5 compares the differences between temperatures on the ITS-90 and the IPTS-68 as published in (1) and as estimated from this experiment. In the table of differences in (1), the maximum difference between the two temperature scales in the range t_{90} = 630.615 °C to t_{90} = 1064.18 °C is 0.36 °C. The maximum is attained at t_{90} = 760 °C, t_{90} = 770 °C, t_{90} = 780 °C (this is a non-unique maximum). The estimate of the difference between the two scales obtained from this experiment has a maximum of 0.051 °C at t_{90} = 790.916 °C. The maximum disagreement between these two estimates of Δt is 0.318 °C at t_{90} = 760 °C.

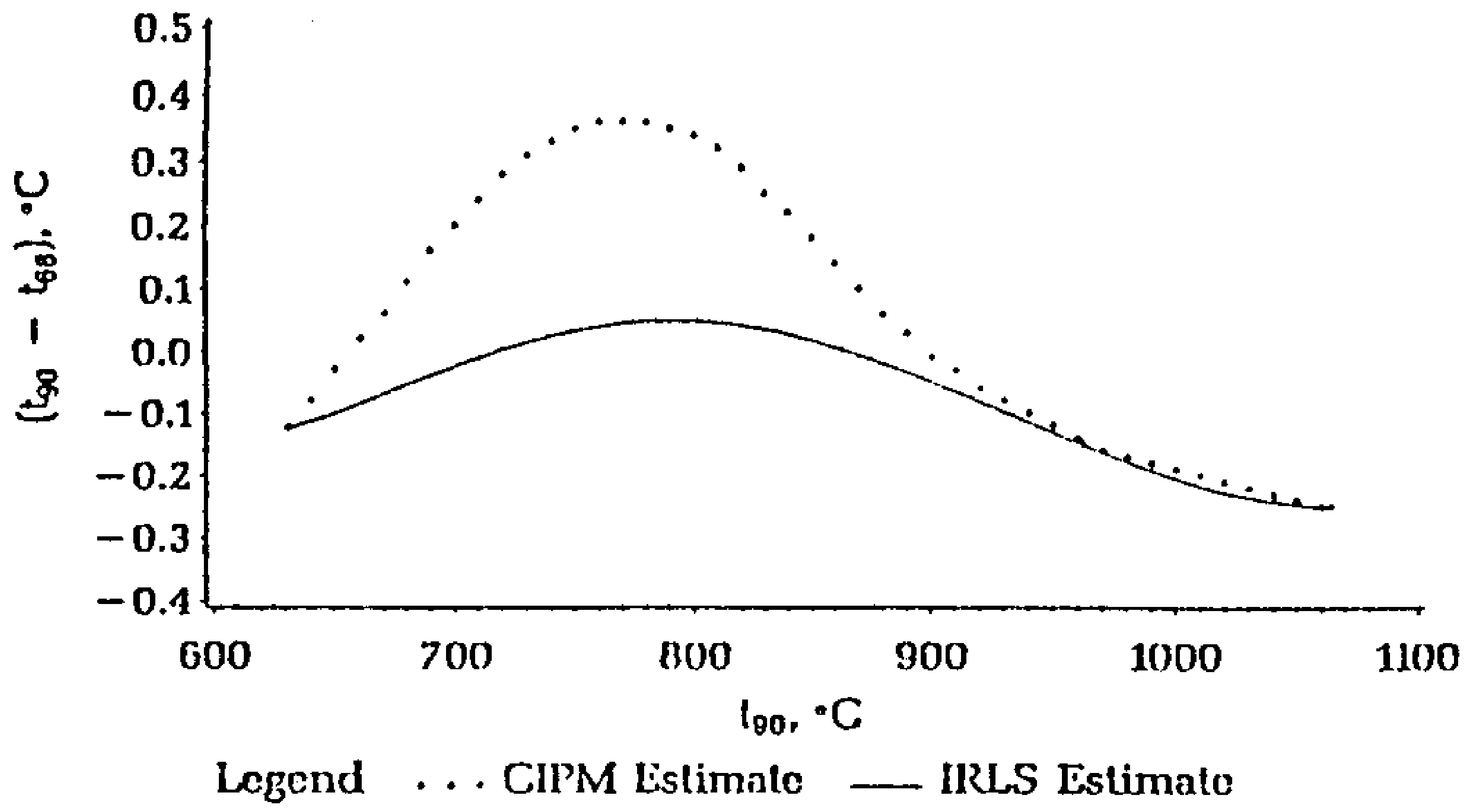

Figure 5: Comparison of IRLS estimate of ($t_{90} - t_{68}$) with previously published CIPM estimate of ($t_{90} - t_{68}$).

Clearly there is significant disagreement between the table of differences in (1) and the data and estimate of Δt presented here. This disagreement should be taken into consideration in any use of the temperature differences between the ITS-90 and the IPTS-68. The estimate of Δt presented here is based on results from a large experiment with the participation of many laboratories. The polynomial describing the temperature differences between the two scales was derived using robust regression techniques which protect against biases caused by isolated, outlying data points. Assuming that the systematic errors present in the data are randomly distributed with zero mean across different lab/thermocouple combinations, the estimate of Δt presented here should be an accurate reflection of the actual difference between temperatures on the ITS-90 and the IPTS-68.

REFERENCE FUNCTION ON ITS-90 FOR TYPE S THERMOCOUPLES

The reference function for type S thermocouples based on the IPTS-68 consisted of four functions, $g_i(t_{68}), (i = 1, \ldots, 4)$, with breaks at $t_{68} = 630.74$ °C, $t_{68} = 1064.43$ °C and $t_{68} = 1665$ °C (7). The reference function based on the ITS-90 consists of a single function, $f_1(t_{90})$, in the range −50 °C to 1064.18 °C and two functions, $f_2(t_{90})$ and $f_3(t_{90})$, in the range above 1064.18 °C (3). The latter are the result of substitution of $t_{90} - \Delta t$ for t_{68} in the IPTS-68 reference functions with modifications as described in Burns, Strouse, et al. (3).

The purpose of this section is to describe the derivation of $f_1(t_{90})$ from the data of the current experiment. The experiment was undertaken because satisfactory reference functions on ITS-90 in the range −50 °C to 1064.18 °C could not be produced by substitution in $g_1(t_{68})$ and $g_2(t_{68})$. Furthermore, an assessment of uncertainty was not possible without a new experiment because the earlier data on type S thermocouples were not archived. Analysis of the present data produced a single function based on the ITS-90 to replace $g_1(t_{68})$ and $g_2(t_{68})$.

The only candidates for determining $f_1(t_{90})$ were NIST thermocouples, S1, S2, S3, S4, and S5 because they were the only thermocouples measured over the full range between −50 °C and 1070 °C. Thermocouple S3 was not considered because it was annealed in a different manner from the other thermocouples, and it was not as stable. There were no repeated runs with thermocouples S1 and S2; consequently, there were fewer data for them than for S4 and S5. Thermocouple S5 was preferred over S4, although S4 was made from the same material that was the basis for the IPTS-68 reference function, because the S5 emf-temperature relationship agreed more closely with the IPTS-68 reference function. Also, S5 was more thermoelectrically homogeneous; its fixed point data agreed more closely with its comparison data than was the case for S4. The data for all five NIST thermocouples and thermocouples from other laboratories are compared in (3).

The data on S5 consisted of a relatively few fixed point determinations and a large number of comparisons with standard platinum resistance thermometers calibrated in accordance with the ITS-90. Comparison measurements were made in five apparatuses as shown in the table below. Below 0 °C, comparisons were made at 10 °C intervals; above 0 °C, comparisons were made at roughly 20 °C intervals except in the neighborhoods of 630 °C and 1064 °C where temperatures were closely spaced. Measurements were made with both increasing and decreasing temperatures with multiple readings at each temperature; however, only data with increasing temperature contributed to the reference function. Data were reduced to four points at each nominal temperature, and the entire sequence was repeated on one or two occasions. The experimental plan for S5 is shown in Table I.

Table I: Experimental Plan for Thermocouple S5.

Apparatus	Range			Occasions
Cryostat	−50 °C	to	−10 °C	2
Water bath	0 °C	to	95 °C	2
Oil bath	95 °C	to	180 °C	1
Salt bath	275 °C	to	550 °C	1
Comparator	500 °C	to	1070 °C	2

The reference function in the range −50 °C to 1064.18 °C is an *8th* degree polynomial, $f_1(t_{90})$, of the form

$$f_1(t_{90}) = \sum_{i=1}^{8} \beta_i t_{90}^i \tag{4}$$

where the coefficients, β_i ($i = 1, \ldots, 8$), were initially determined from a least squares fit, $p(t_{90})$, to 444 measurements of emf as a function of temperature on the ITS-90. The residual standard deviation was 0.0629 μV with 436 degrees of freedom. The residuals from the fit are shown in Figure 6.

An *8th* degree polynomial was chosen as the reference function for the following reasons. Lower degree polynomials did not prove adequate in that the residuals from the fit, which should be randomly distributed about zero, showed cyclic structure. A test statistic for each β coefficient was formed as the ratio of β to its standard error. All coefficients for the *8th* degree polynomial tested significantly different from zero at the 0.0001 significance level, indicating the need for at least an *8th* degree fit. Furthermore, the *8th* degree fit proved successful in removing cyclic structure across different pieces of equipment, although retaining some structure within an apparatus. Polynomials of higher degree were successful to some extent in removing within apparatus structure from the residuals; however, the reduction in the mean squared error, achieved by going from an *8th*

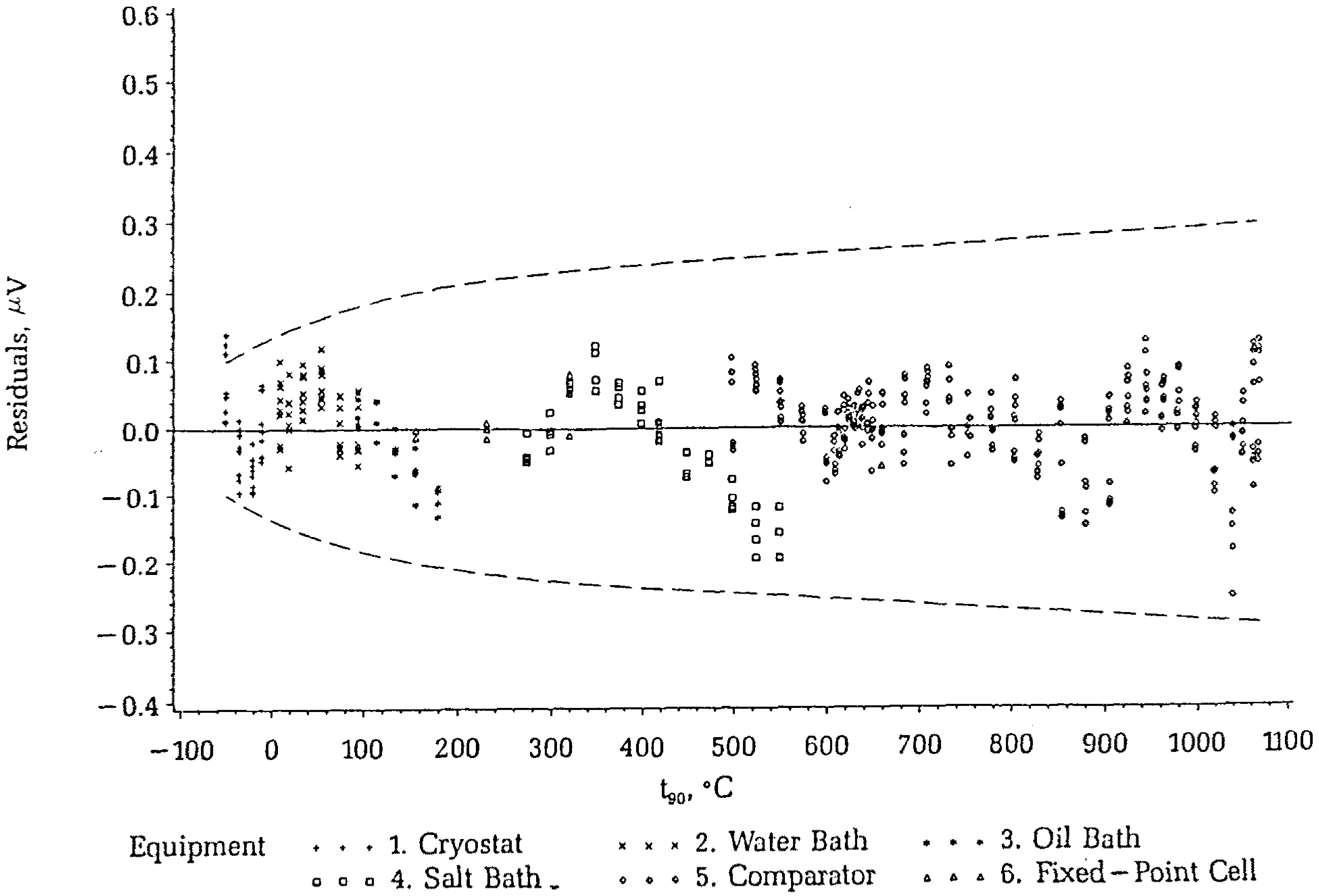

Figure 6: Residuals from an 8th degree polynomial model vs. t_{90} for NIST thermocouple S5. The dashed lines are ±25 m°C bounds on the residuals in terms of emf.

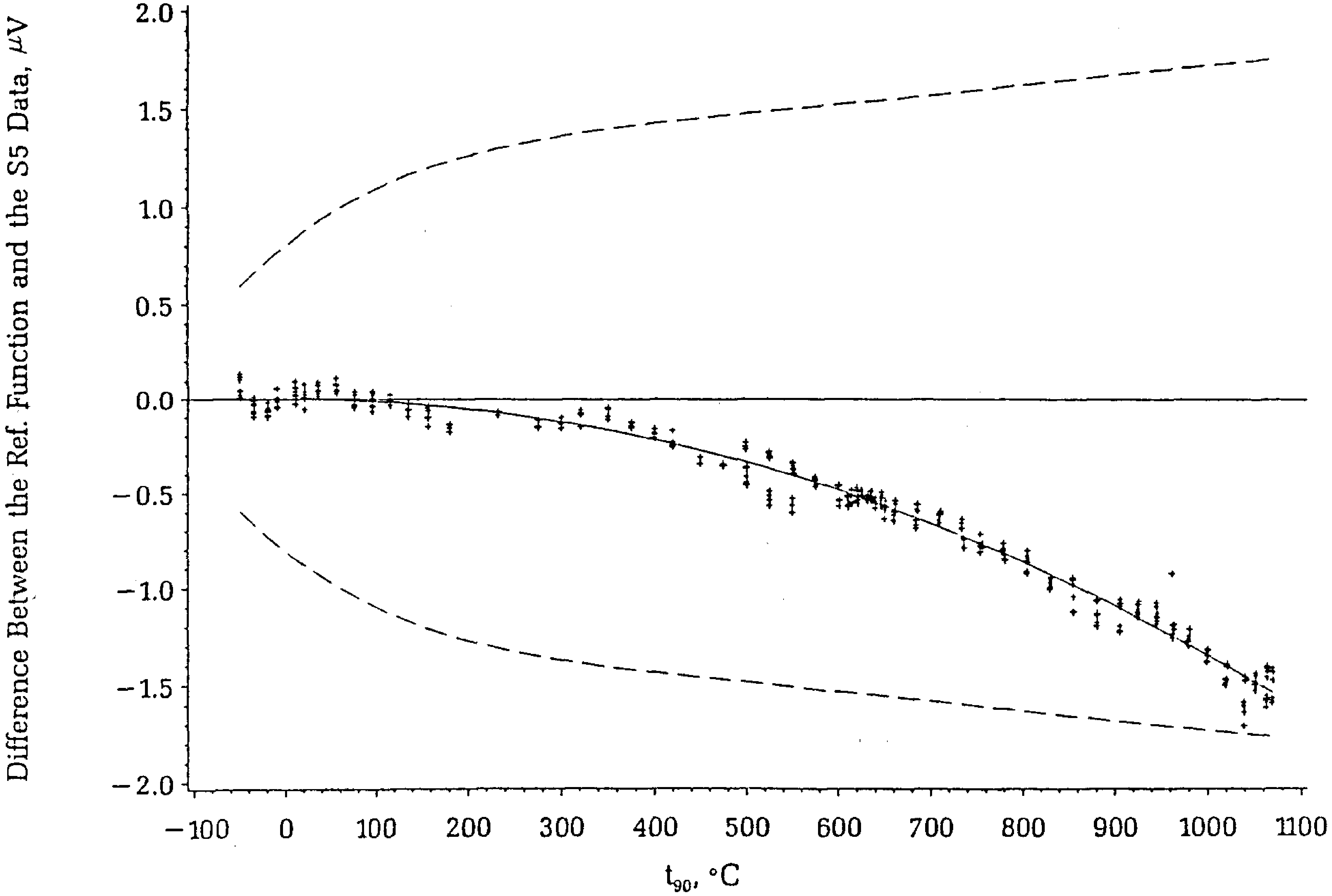

Figure 7: Deviation of S5 data and the polynomial fit to the data from the reference function as published in (3). The solid line indicates the 8th degree polynomial fit. The dashed lines indicate deviations equivalent to ±0.15 °C.

degree polynomial to a higher degree polynomial, did not prove substantial enough to warrant increasing the order of the reference function. Also, the structure in the residuals suggests that small changes occurred in the thermocouple with increasing temperature. These changes should not be reflected in the reference function. Therefore, the model does not account for this type of structure.

The random component of uncertainty for $p(t_{90})$ is calculated using Working-Hotelling confidence bands (10). The upper and lower 95% confidence bands at temperature t_h are $p(t_h) \pm v(t_h)$ where

$$v(t_h) = \sqrt{8F_{0.95}(8,436)}s_h. \tag{5}$$

The critical value $F_{0.95}(8,435) = 1.96$ is the upper 95 percent point of the F distribution with 8 and 436 degrees of freedom, and s_h is the standard deviation of $p(t_h)$ at temperature t_h. The Working-Hotelling confidence bands are appropriate for unlimited use of the reference function. Representative values are shown in Table II.

Table II: Random uncertainties (μV) for $p(t_{90})$ from 95% Working-Hotelling confidence bands

t °C	$p(t_{90})$	$v(t_{90})$
-50.00	-235.56	0.07
0.00	0.00	0.00
100.00	645.90	0.04
200.00	1440.73	0.04
300.00	2322.92	0.05
400.00	3259.14	0.04
500.00	4232.96	0.03
600.00	5238.21	0.02
700.00	6274.59	0.03
800.00	7344.12	0.03
900.00	8448.15	0.03
1000.00	9585.75	0.04
1064.18	10332.68	0.05

The final reference function, $f_1(t_{90})$, is derived from an adjustment of the quadratic coefficient, β'_2, of $p(t_{90})$ to make $f_1(t_{90}) = f_2(t_{90})$ at $t_{90} = 1064.18$ °C. The adjusted coefficient is

$$\beta_2 = \frac{f_2(1064.18) - (p(1064.18) - \beta'_2 1064.18^2)}{(1064.18)^2}. \tag{6}$$

The magnitude of the adjustment at 1064.18 °C is 1.52 μV. Figure 7 shows the deviation of the S5 data from the reference function $f_1(t_{90})$. The adjustment guarantees that the reference function is *continuous* at 1064.18 °C. The coefficients for $f_1(t_{90})$ are reported by Burns, Strouse et al. (3).

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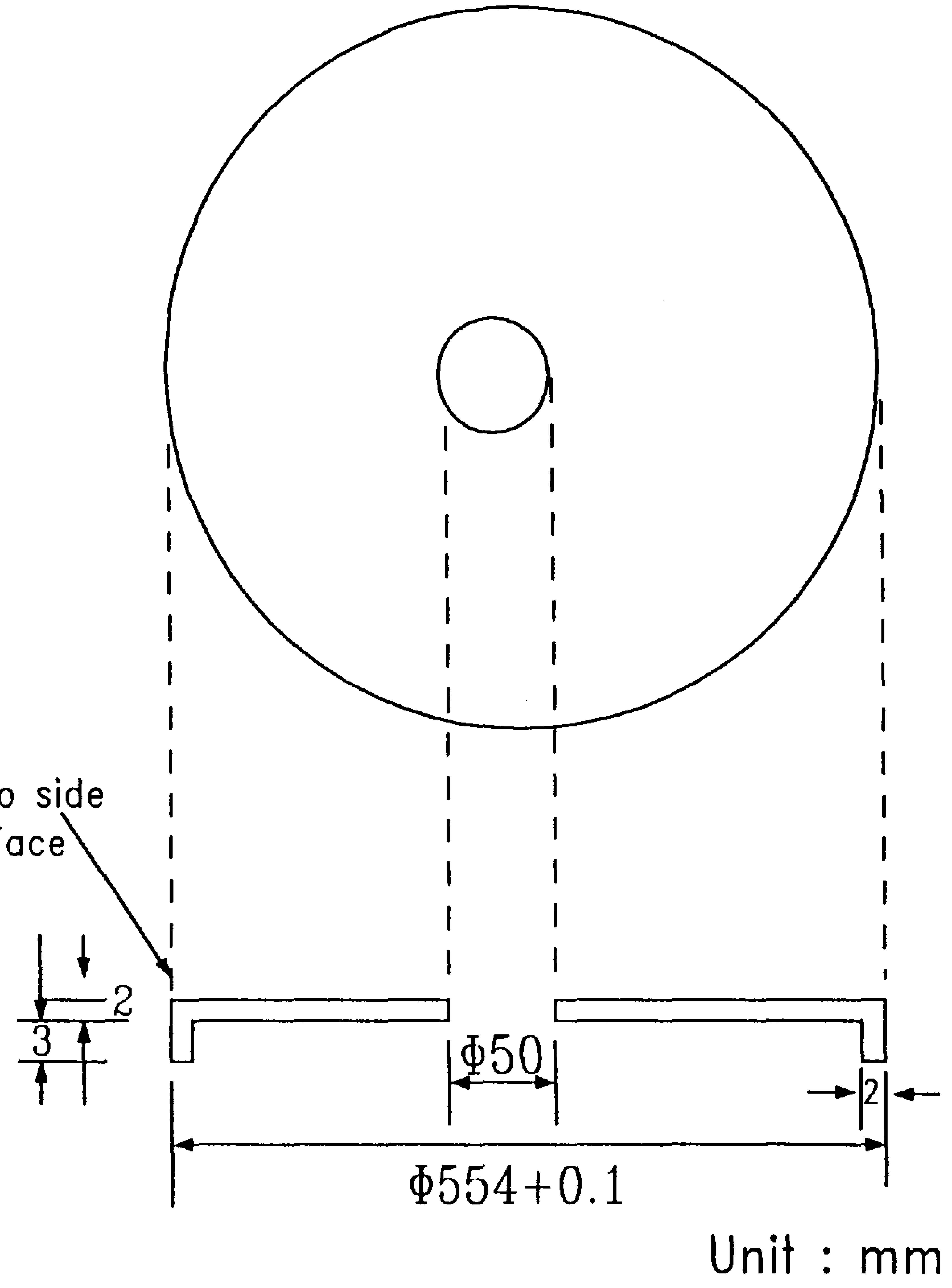
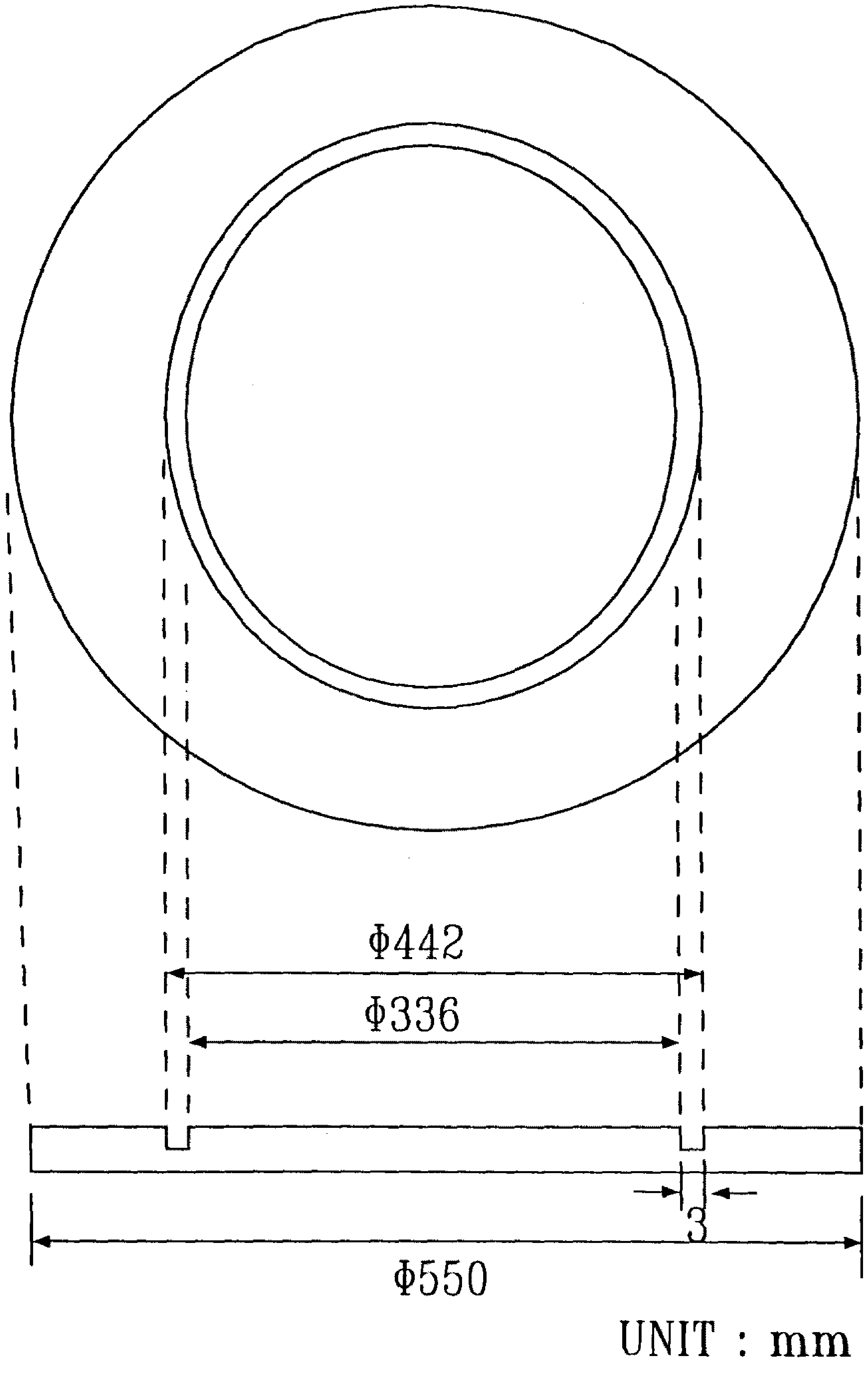
부록2. 고온전기로 부품도면

Upper flange



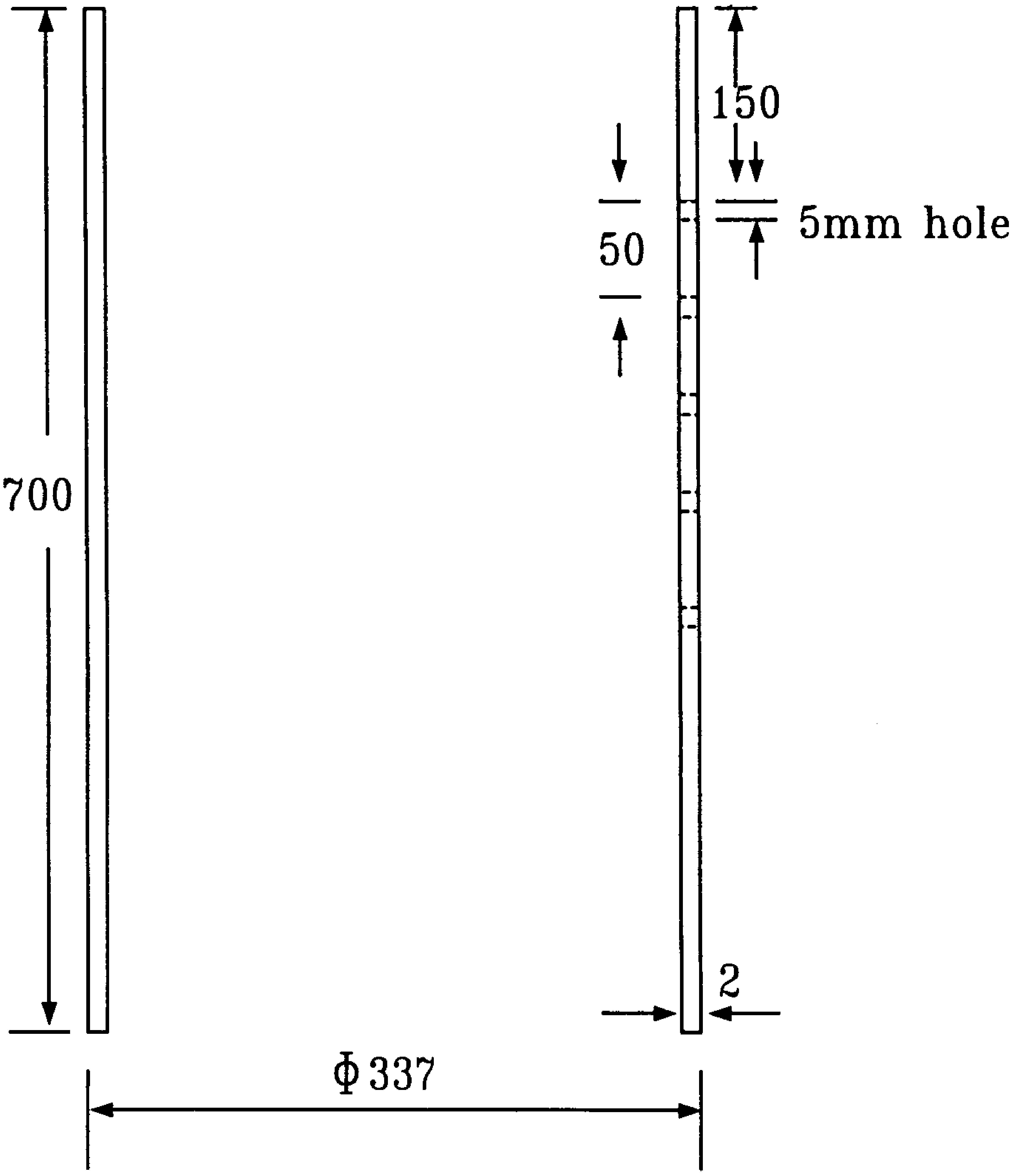
Material : Stainless
steel 304

Down flange



Material : Stainless
steel 304

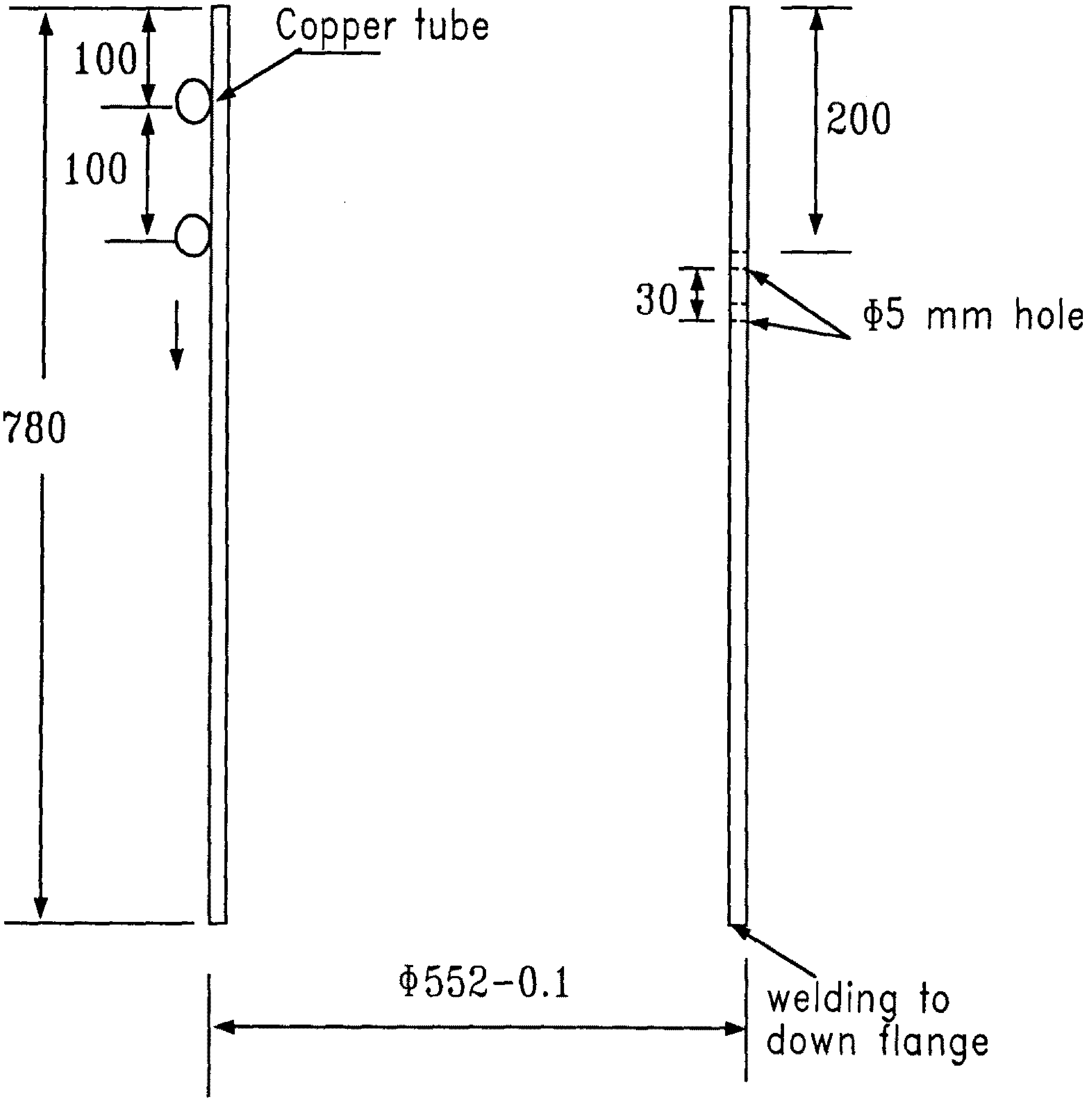
Radiation shield



UNIT : mm

Material: Stainless
steel 310S

Surface of furnace

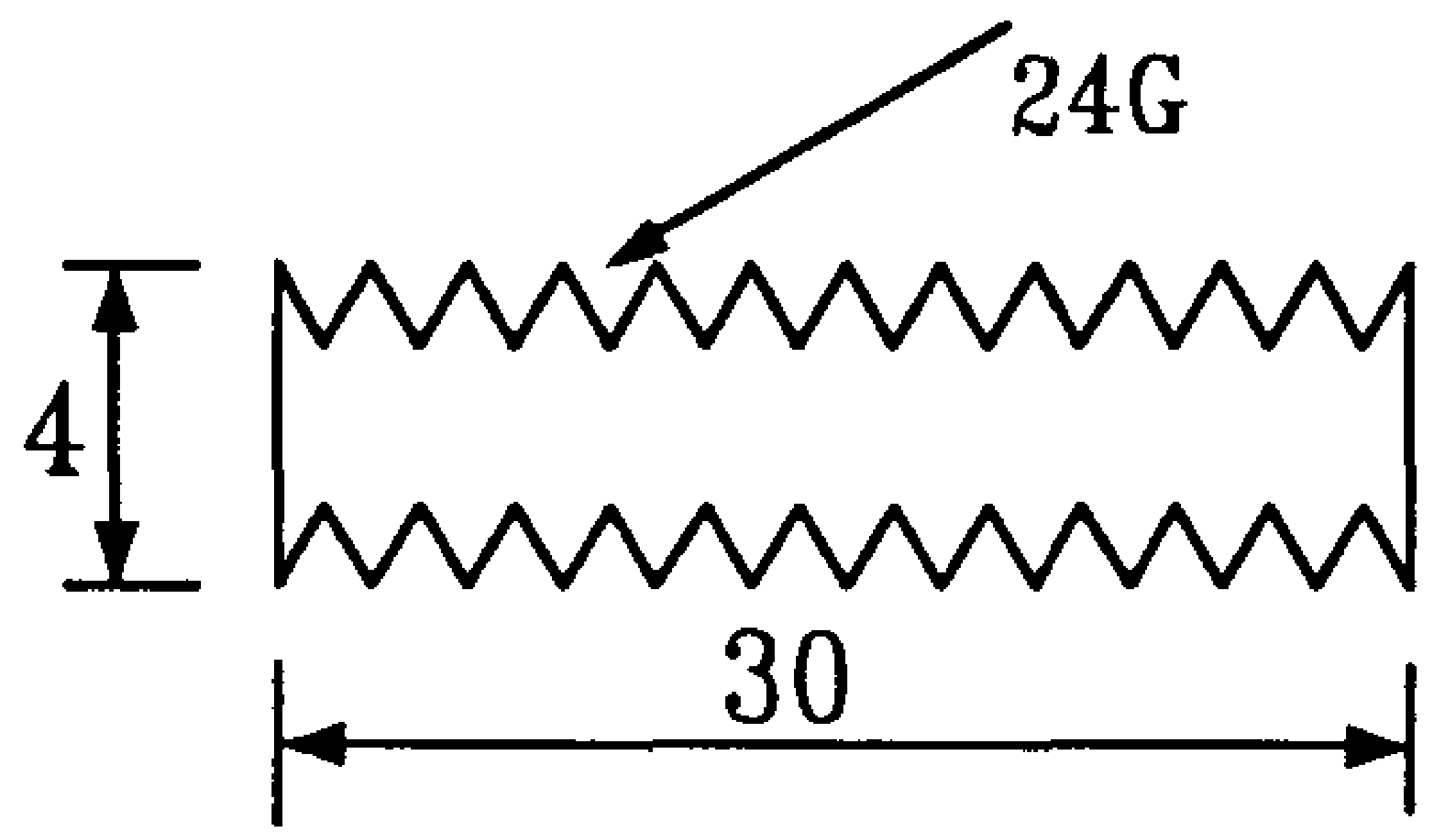


Unit : mm

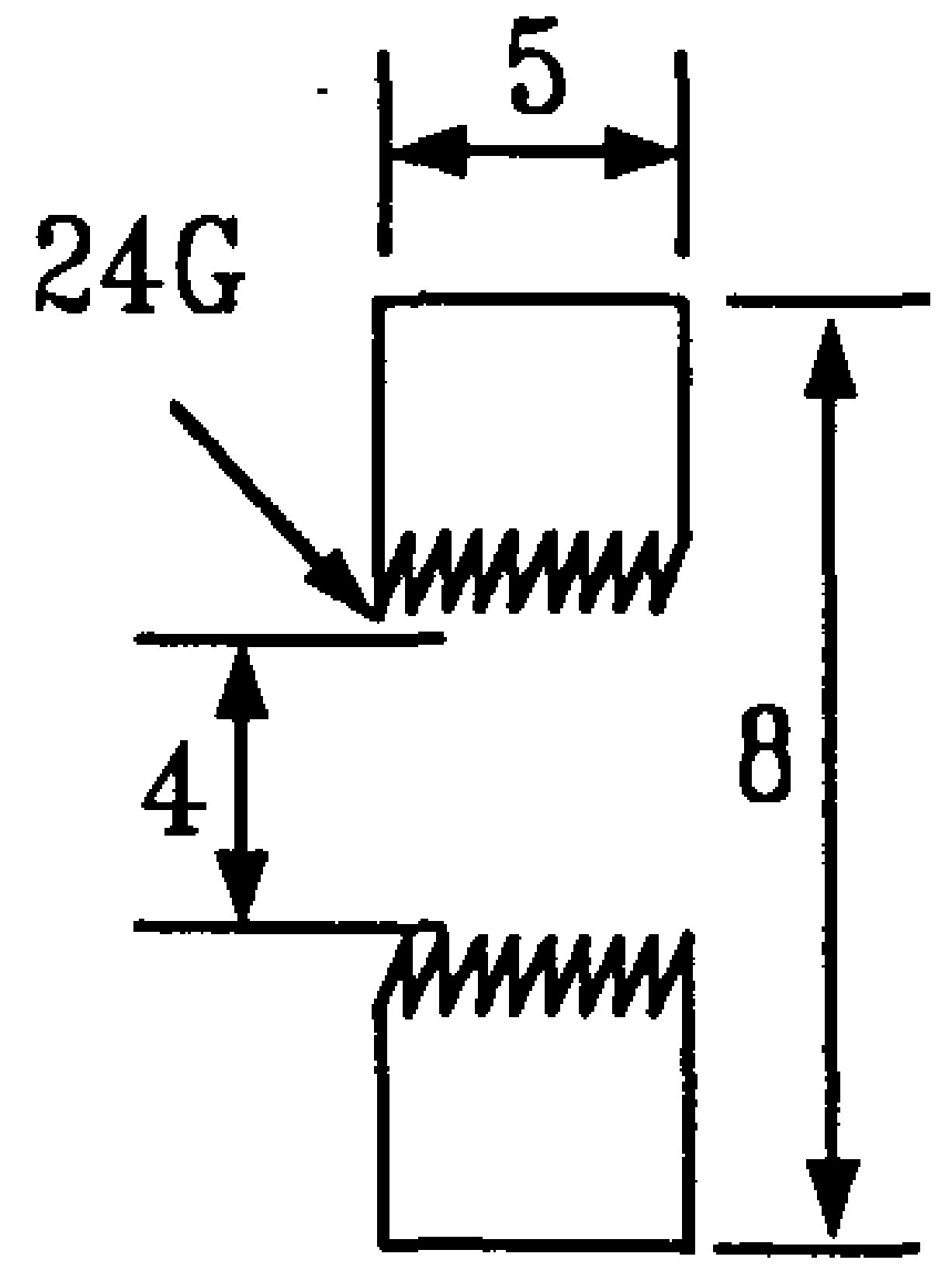
Material : Stainless
steel 304

Parts of electric terminal

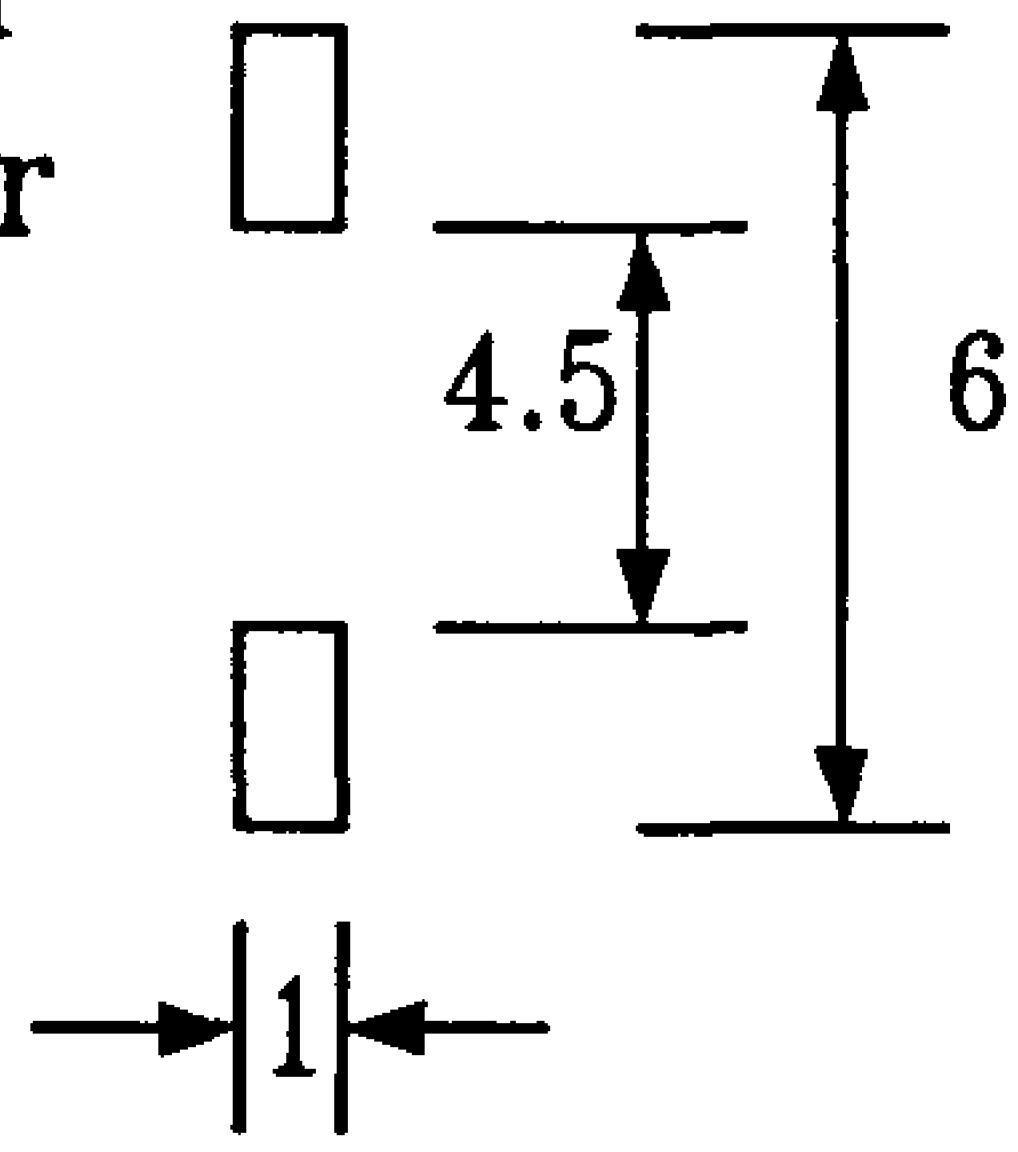
A. Copper screw



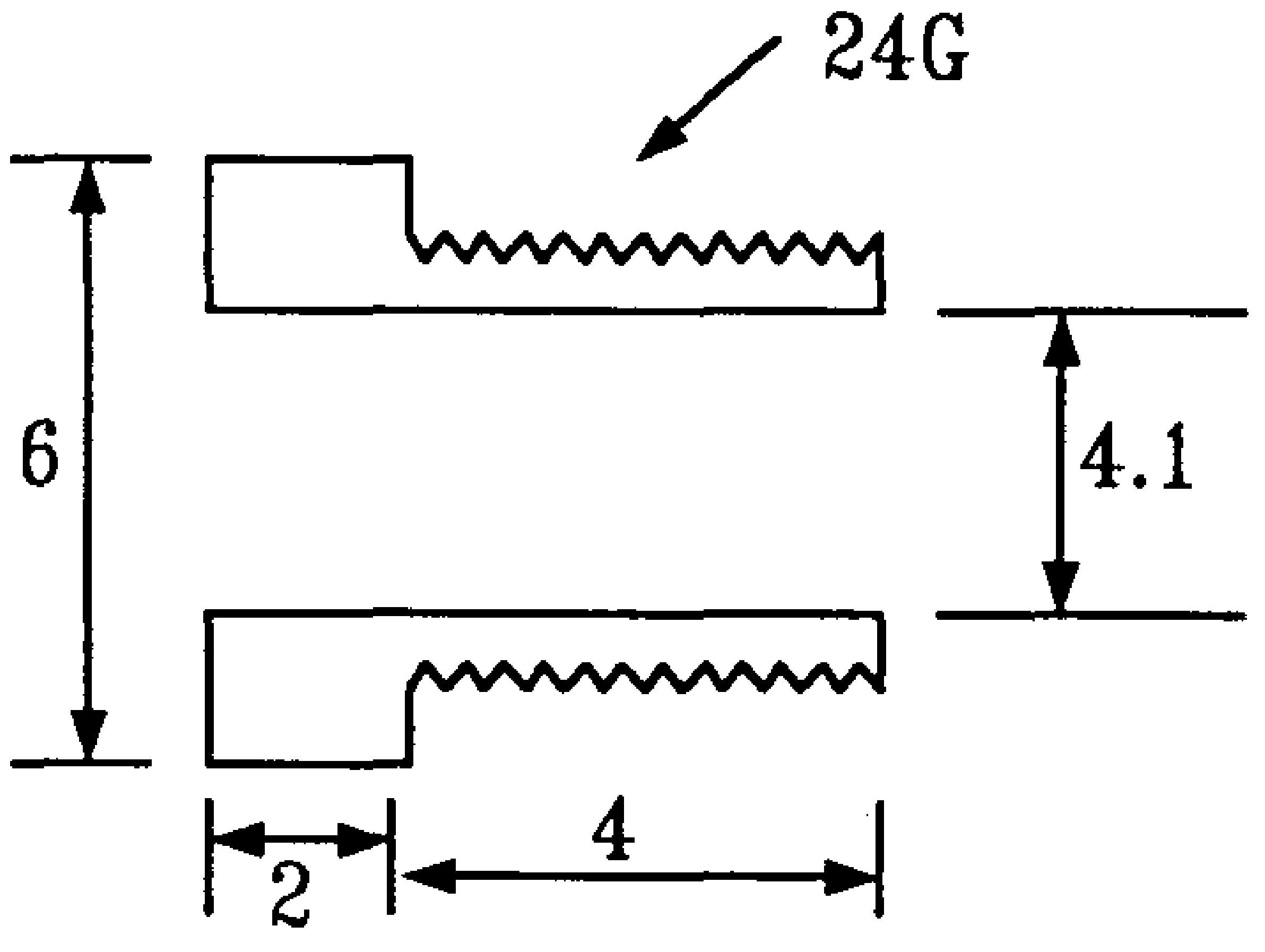
B. Copper female screw



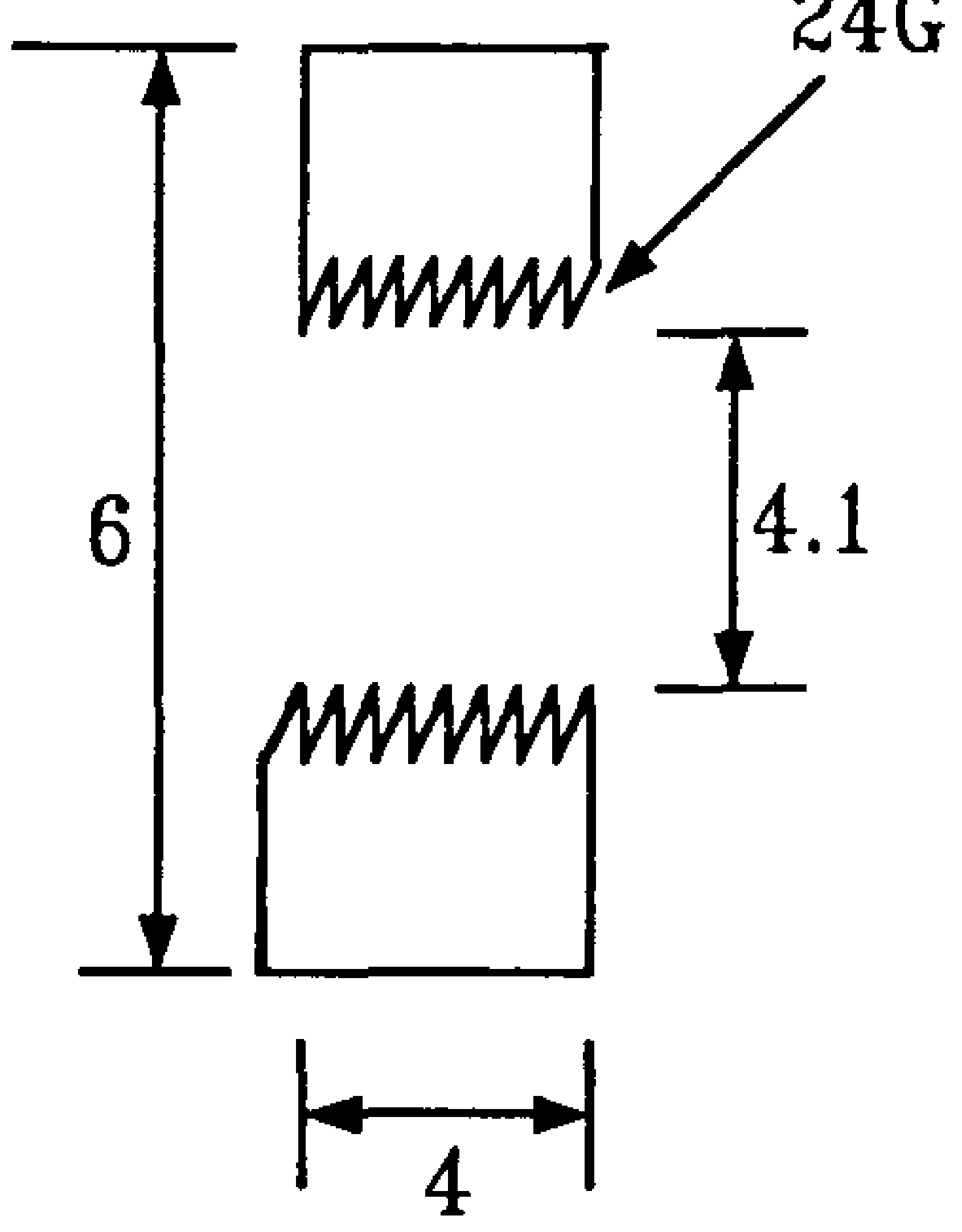
C. Copper washer



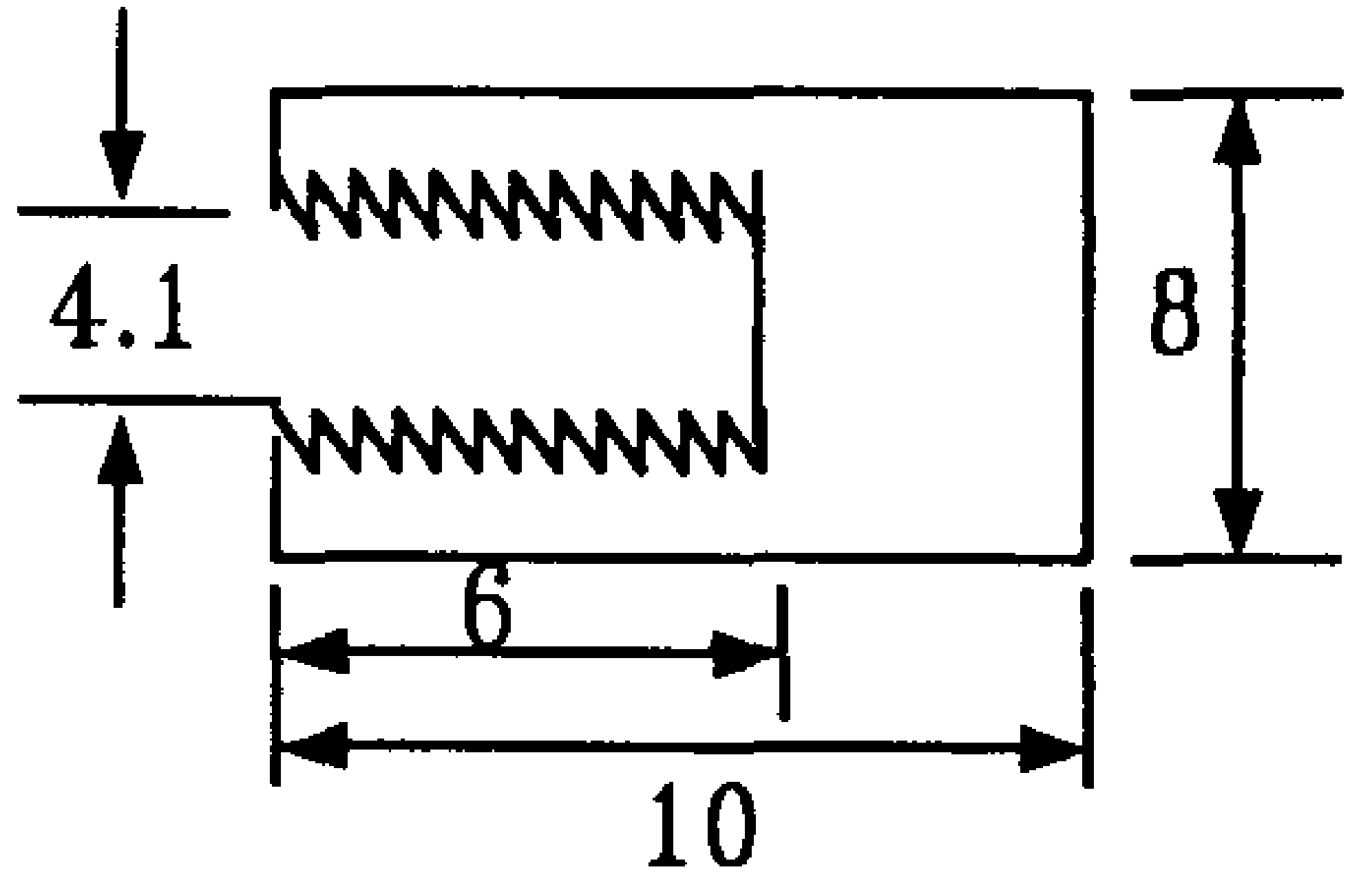
D. Teflon insulator



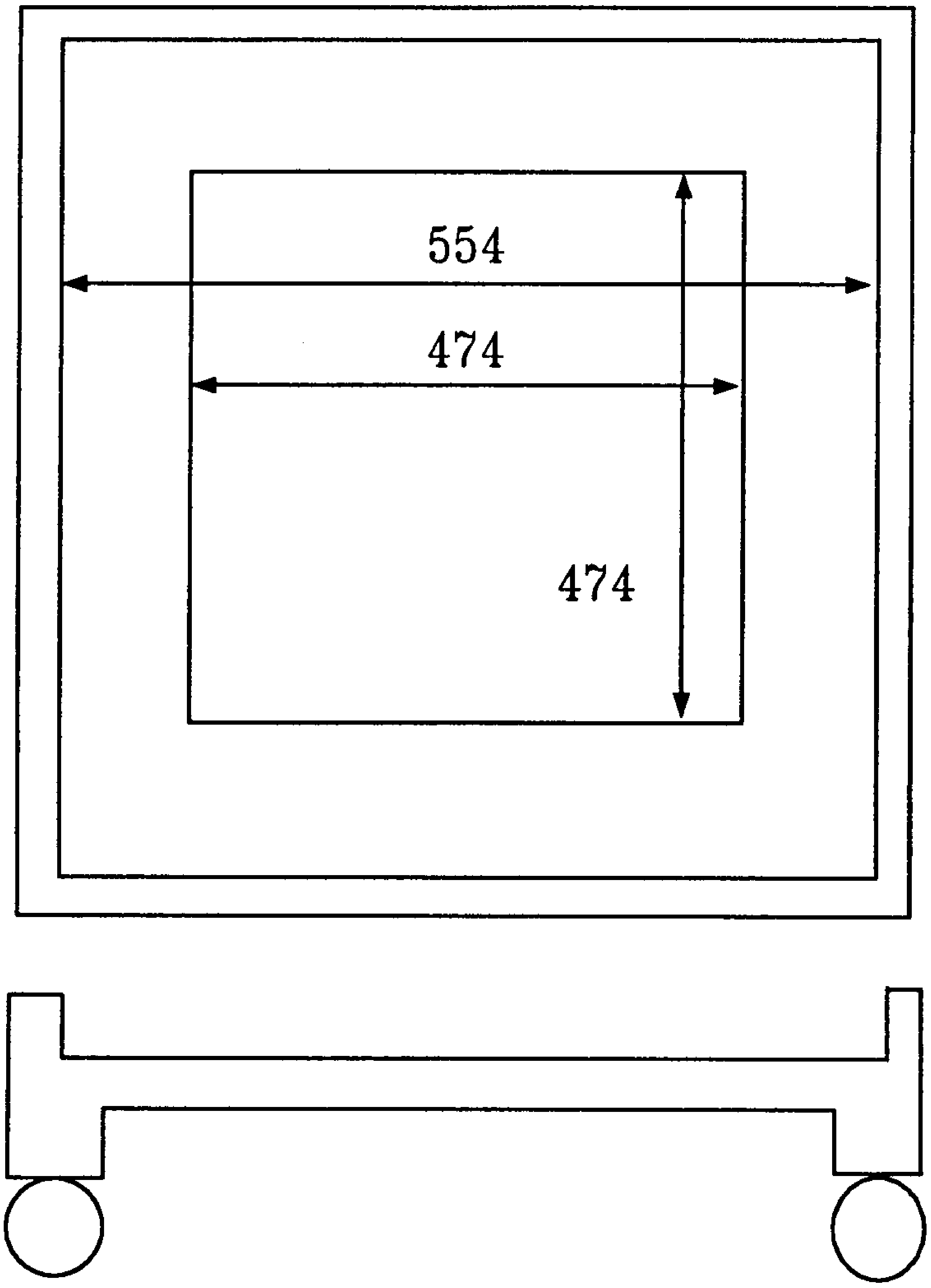
E. Teflon washer



F. Teflon screw



Moving stage of the furnace



Unit : mm

Material : Steel angle