PREAGING TECHNIQUES AS A MEANS OF STABILISING THERMOELECTRIC DRIFT IN NICKEL-CHROMIUM/NICKEL-ALUMINIUM THERMOCOUPLES FOR USE IN AN ALUMINIUM HEAT TREATING FURNACE

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Abstract

This dissertation is primarily concerned with investigating and improving the degree of accuracy and precision that may be achieved from temperature measurements made utilising nickel-chromium/nickel-aluminium (Type K) thermocouples.

The practice of heat treating extruded aluminium section creates specific metallurgical properties within the section. Development of specialised aluminium alloys has necessitated the use of treatment temperatures, close to the limit beyond which the alloy experiences undesirable, permanent, metallurgical change. This situation has demanded urgent attention to, in quality assurance terms, the, "fitness for purpose", of primary temperature sensors.

The most established of these sensors, the Type K thermocouple, has known problems relating to calibration stability and drift. The substantial amount of furnace control instrumentation and cabling dedicated to measurement from Type K sensors precludes the simple conversion to an alternate sensor type. The more practical option of applying calibration correction factors to existing measuring systems is only feasible if sensor stability characteristics permit measurement traceability to be established within required uncertainty limits.

This project investigates the employment of a preaging technique as a means to stabilise the calibration of Type K thermocouples intended for use in conditions specifically applicable to the heat treatment of aluminium. The investigation includes evaluation and assessment of the preaged thermocouple calibration characteristics through industrial process trials. The most severe process application, solution heat treatment, is selected for the evaluation trials as it is the treatment requiring the most demanding control limit (+/-3^{o}C on the aluminium).

Essential throughout the investigation is the aspect of traceability of the calibration data to the national standard. Frequently misunderstood, these aspects of traceability and calibration, within the context of temperature metrology, are discussed in the dissertation.

A consequence of the project's requirement for fast, traceable calibration data, produced with a low level of uncertainty, in an industrial process environment, is the development of the automated calibration system. Employing a unique algorithm to ensure stability at time of calibration, the system, after loading, runs unattended.

Dedication

To my wife

Janet

Quote

The ability to measure
accurately and with repeatability
therefore is, if not the most important
then certainly a very, very important element of
improved productivity

(Fulton, 1990)

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CHAPTER 1

THE PROBLEM AND ITS SETTING

1.1 INTRODUCTION

In the solution heat treatment furnaces (figure 1.1) at Hulett Aluminium Profiles (Pty) Ltd. in Pietermaritzburg four sets of thermocouples are used to record and control the temperature within the electrically heated furnaces. The first set, the **control thermocouples** (figure 1.2), provide the input signals to the furnace temperature controllers; these thermocouples are situated, one per furnace zone, within the airstream of the heated air as it enters the furnace work chamber. The second set of thermocouples, the **air thermocouples**, are either constructed in duplex with the control thermocouples, or alternately, are individual thermocouples located in close proximity to the control thermocouples. These thermocouples provide the input signals to the furnace temperature chart recorder. This is a multiple input device providing a trend plot of temperature in relation to time with each zone's temperature trace being overlaid on a common section of chart. The plot of the furnace air temperature is used to provide a record of the furnace performance. The third set of thermocouples, the **extreme temperature thermocouples**, are also positioned within the air flow of each heating zone

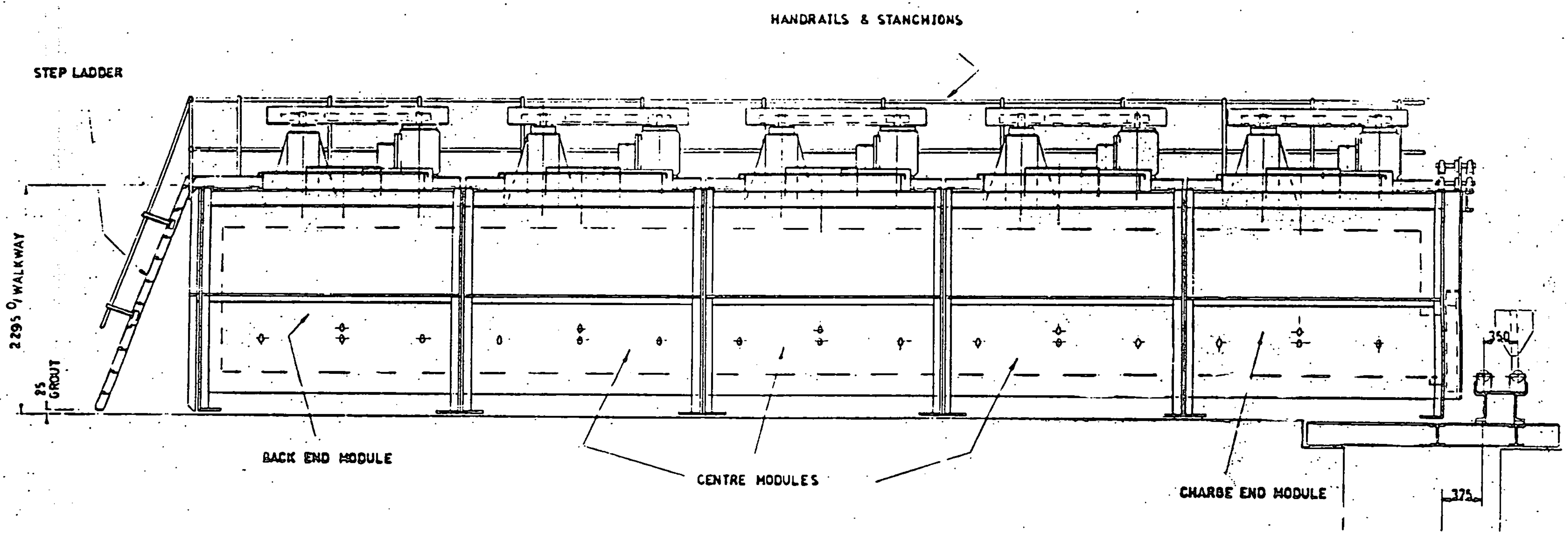


Figure 1.1 Typical furnace layout. Side view of five zone aluminium heat treatment furnace. Air, control and excess temperature thermocouple entry ports shown on each zone side module. (Mechatherm Eng.Ltd.,1980,Drg.No.1049-38A)

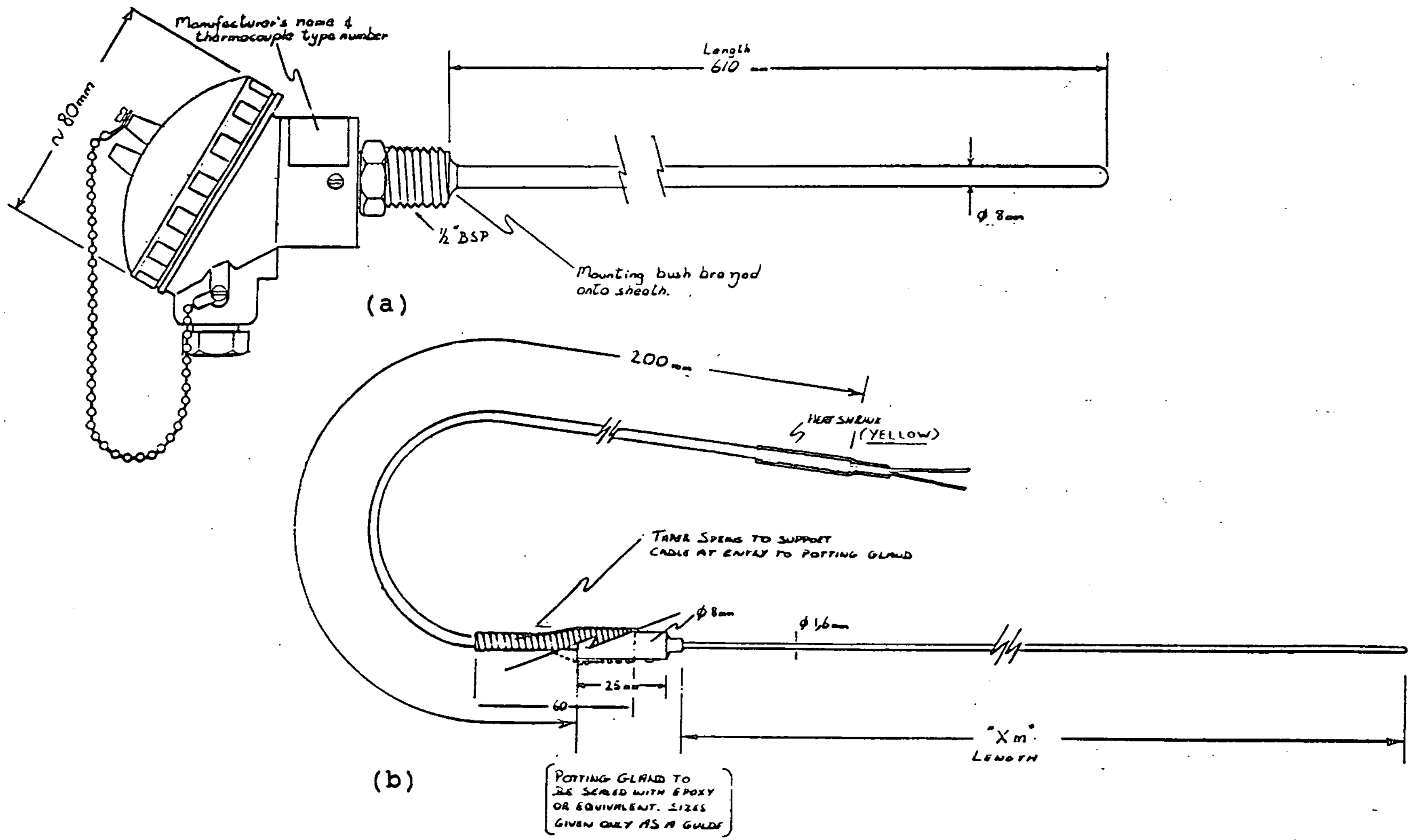
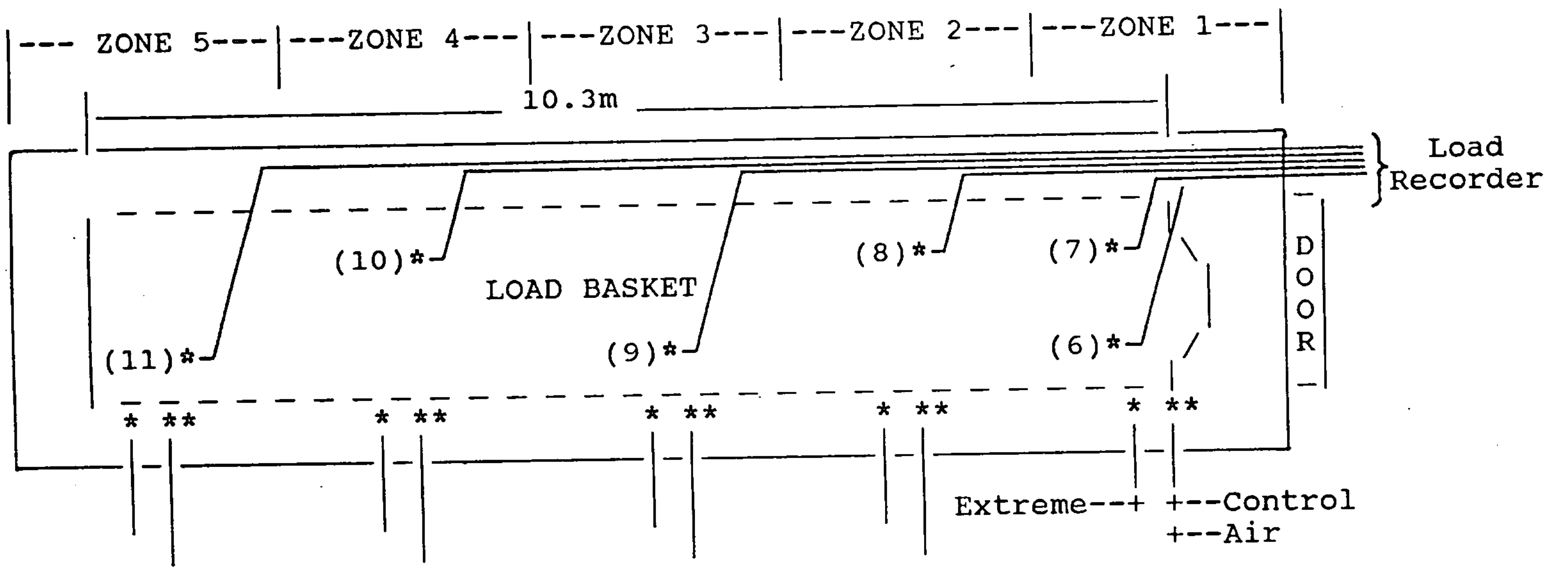


Figure 1.2 Thermocouple description. (a) Control type thermocouple. (Type: HA/K/8/610/A). (b) Load type thermocouple. (Type: HA/H/1.6/Xm/LA). (Hulett Aluminium Standard Temperature Sensor and Material Specification, 1990)

and provide the input signal to the overtemperature controllers, the function of which is to shut off all electrical power to the elements in the event of the temperature reaching a preset maximum value. The fourth set of thermocouples are flexible **load thermocouples** (figure 1.2) whose function is to record the actual temperature of the aluminium load within the furnace. For this purpose these thermocouples are mechanically bonded to various sections of aluminium which are then positioned among the load across the length of the furnace (figure 1.3). These thermocouples provide a second set of input signals to the furnace chart recorder, or in certain cases, to a separate recorder, providing a trend plot of the temperature of the aluminium load. This trend will lag the furnace air temperature trend by some degree, dependent, among other factors, on the mass of the load and the furnace efficiency. It is the temperature provided by the set of load thermocouples that determines the start of the heat treatment cycle and determines also whether the heat treatment criteria has been met.

It is well known that Type K thermocouples exhibit thermoelectric instability and drift related both to time and temperature. This instability and drift causes an error in measurement which affects the accuracy of both the control and recorded temperatures. Control temperature error effectively means that the furnace operates at some temperature other than the required temperature and error on the load temperature record results in an incorrect soak period.



Load Thermocouple lengths: (6) = 4.5m (9) = 9.0m
(7) = 5.0m (10) = 11.5m
(8) = 7.0m (11) = 13.0m

(*) Indicates approximate positioning of thermocouple measuring junction.

Figure 1.3 Load thermocouple positioning for a five zone furnace.

The control, air and extreme air temperature thermocouples are normally held at temperatures which vary between 200 and 500^{o}C. The load thermocouples are, in the case of solution heat treatment, subjected to the same quenching as the load (Hulamin Standard Practice,1983, 9.K.3). This rapid cooling causes an apparent inhomogeneity in the temperature gradient zone of the thermocouple, and since the thermocouple is flexible, the positioning of this zone may vary to some extent on successive loads.

Many investigations into the cause, including inhomogenenity, and extent of this instability and drift (Burley,Hess,Howie,Coleman,1982) have been made since Dahl's first detailed study was presented in 1939 (Dahl,1941). A new thermo-element alloy, nicrosil/nisil - Type N, was developed specifically to minimise this drift (Starr and Wang,1976; Wang and Starr,1980). A problem still exists in that:

(i) Much of industry is committed, through the dedicated nature of existing thermometry installations, to the use of Type K thermocouples in their processes.

(ii) There remains insufficient data covering the rate of drift and extent of instability of these thermocouples to enable the user of an industrial solution heat treatment furnace to control the temperature of the furnace to such a degree that the temperature across the furnace load may be certified as conforming to within (+/-)3^{o}C of the true temperature traceable to the National Standard.

1.2 STATEMENT OF THE SUBPROBLEMS

1.2.1 The First Subproblem

The first subproblem is to determine the temperature and length of soak to which the thermocouple or thermocouple material should be preaged in order to obtain a satisfactory level of stability prior to operational use.

1.2.2 The Second Subproblem

The second subproblem is to determine the lowest level of total uncertainty that may be stated for the load temperature values as corrected and recorded during the operation of a heat treatment furnace in order to specify the number of hours or cycles of use at which that level of uncertainty will hold good for a 95% level of confidence.

1.3 THE HYPOTHESES

1.3.1 The First Hypothesis

The first hypothesis is that after a large initial drift in thermoelectric output, Type K thermocouples become relatively stable, and that by heat treating the thermocouple material prior to use, firstly, this state of relative stability might be produced, and secondly, that the extent of inhomogeneity created by subsequent useage may be reduced.

1.3.2 The Second Hypothesis

The second hypothesis is that by applying a calibration correction factor to the furnace temperature controllers and recorders, and by adhering to a practice of periodic recalibration, the load temperature may be shown statistically to be within an accuracy of (+/-)3^{o}C of the true temperature traceable to the National Standard within a 95% level of confidence.

1.4 THE DELIMITATIONS

The delimitations of this project were determined by the operating parameters and the thermocouple specification for the heat treatment furnaces at Hulett Aluminium Profiles (Pty) Ltd.

1.4.1 Temperature Range

This study is confined to the temperature range:
100 to 550^{o}C, this being the range that covers the following aluminium heat treatment practices:

(i) Aging: 100 to 300^{o}C.
(ii) Annealing: 300 to 450^{o}C.
(iii) Solution heat treatment: 450 to 550^{o}C.

1.4.2 Load Thermocouples

The work on load thermocouples is specifically on 3mm diameter, Type K, stainless steel sheathed mineral insulated thermocouples.

1.4.3 Control and Air Thermocouples

The work on control and air thermocouples is specifically on 8mm diameter, Type K, stainless steel sheathed mineral insulated thermocouples.

1.4.4 Other Thermocouple Types

In this study comparisons of thermocouple Types other than Type K are obtained under specific test circumstances, and are included to add perspective to the current study and should not be considered as conclusive evidence of the characteristics noted in these other thermocouple Types.

1.5 THE DEFINITIONS OF TERMS

Bias: The arithmetic difference between the average of a set of data and the required value.

Calibrate: To determine the indication or output of a measuring device with respect to that of a reference.

Calibrate (in regard to thermocouples): To determine the emf developed by a thermocouple with respect to a temperature established by a standard (ASTM:1990,p.248).

Control thermocouple: A thermocouple measuring the air temperature in a furnace zone and supplying that zone temperature controller with an input signal.

Instantaneous value: The temperature value in degrees Celsius of a thermocouple being logged on the calibrating system without any statistical manipulation of the value.

Load thermocouple: A thermocouple that has its hot junction in firm mechanical contact with one of the aluminium sections being heat treated.

Reference thermocouple: A noble metal thermocouple of the same order of quality and calibration integrity as a standard thermocouple but which is used as a reference to corroborate the results of the standard thermocouple during an automatic calibration procedure.

Standard thermocouple: A noble metal thermocouple that has been calibrated and certified by the CSIR in accordance with their procedures for providing traceability to the National Standard. The standard thermocouple is utilised as the standard of highest quality within the secondary calibrating laboratory.

Soak: The period for which the load of aluminium within a furnace, having reached the specified temperature range, is required to be held within that range to ensure that the specified properties will be developed (MIL-H-6088F,1981, 3.7.3.1)

Survey thermocouple: A load thermocouple that is used in determining the furnace calibration and which is one of a set of thermocouples which is calibrated prior to and following the evaluation of the furnace calibration.

Thermocouple: A thermoelement formed by the junction of two dissimilar metals. For the purpose of this study a thermocouple shall refer only to the element up to and including the first connector and shall not include the compensating cable linking the element to the monitoring equipment, neither shall it include what is commonly termed the cold junction reference circuit.

Type K thermocouple: Nickel-10% chromium versus Nickel-5% (aluminium, silicon) alloy thermocouple as defined in NBS monograph 125.

Type N thermocouple: Nicrosil versus Nisil alloy thermocouple as defined in NBS monograph 161.

1.6 ASSUMPTIONS

1.6.1 The First Assumption

Hulett Aluminium Profiles (Pty) Ltd. is a SABS 0157 Quality listed Company and the purchase of thermocouple materials is controlled by the following means:

(i) A limited number of approved local (South African) suppliers are selected and utilised.

(ii) The description and specification for each item ordered is identified in a controlled document the "Standard Temperature Sensor and Material Specification" (Hulett Aluminium, 1990) which is issued to each of the approved suppliers.

(iii) All thermocouple materials are, prior to acceptance, checked by the Hulett Aluminium Temperature Laboratory.

These controls are considered adequate in securing a satisfactory level of consistency in the quality of material supplied. All thermocouple wire is currently imported and the above system provides no control over the sourcing of the bulk thermocouple material which may originate from any one of a number of countries. The first assumption is that the thermocouple material used during the test is representative of the same grade, type and gauge from other sources. This assumption will be tested by a comparative study of material from three independant sources.

Further support for the validity of the above assumption is provided by an investigation conducted by Sanders (1974,p.204). Using an analysis of variance on data obtained over a five month period, Sanders examined the effects that various factors, including suppliers, had on thermocouple wire. His findings indicate that only the gauge of the wire has any effect on precision, bias or variation. A total of 42 reels of wire from 6 different vendors was considered in this analysis (Sanders,1974, p.204).

1.6.2 The Second Assumption

The second assumption is that the initial inhomogeneity of the thermocouple is negligible in comparison to the effects caused by the controlled preaging of the thermocouples (BURLEY,et.al.,1982,p.1159).

1.6.3 The Third Assumption

The third assumption is that for control and air thermocouples, the depth of immersion will not be changed after initial installation. The validity of this assumption will be assured by thermocouple mounting design and an identification system which excludes the possibility of interchanging the thermocouples.

1.7 THE IMPORTANCE OF THIS PROJECT

1.7.1 Thermocouple Accuracy

Due to the close tolerance on temperature required for the heat treatment of certain aluminium alloys the use of Type K termocouples as temperature sensing elements has become unsatisfactory in terms of accuracy. The project addresses this immediate problem and provides industry with:

(i) An indication of the feasibility of using the proposed preaging and calibration correction techniques, thereby dispensing with the necessity of a changeover to an alternative temperature sensing device which may have an equally unproven record of performance under the previously noted operating conditions.

(ii) A previously unobtainable, statistical estimate of load thermocouple accuracy related to use, thereby directly improving on certifiable temperature tolerance levels.

(iii) A base, established within the aluminium heat treatment field, against which a future comparison of alternative temperature sensing elements may be made.

1.7.2 Derived Benefit

As a direct result of the increased integrity of furnace control and load temperature measurement introduced through this project:

(i) The intercomparison of furnaces and their efficiencies will be more accurately assessed.

(ii) Time taken for load to reach soak temperature, a prime factor in determining furnace throughput, and a direct function of measurement accuracy, will be positively influenced.

(iii) Increased confidence in measurement results, in conjunction with physical properties records, will enable metallurgists to optimise heat treatment practices.

1.7.3 Groundwork for Future Development

Insight gained through this project indicates that much technological development remains to be done in the heat treatment field in order to keep pace with the requirements of producing specialised aluminium alloys in production quantities. It is envisaged that this development, specifically with respect to temperature control, will take place in the following areas:

(i) Furnace survey techniques.

(ii) Statistical process control.

(iii) Predictive control of load temperature through analysis of power utilization.

1.7.4 Commitment to the Advancement of Technology

In an attempt to deal with the immediate difficulties experienced in the measurement and control of temperature within aluminium heat treating furnaces, and for Hulett Aluminium Profiles (Pty) Ltd. to maintain the technological lead in the field of special alloy production, this project is considered to be part of this Company's commitment to the continued updating of its technology.

1.8 OVERVIEW OF THE CHAPTERS

1.8.1 Chapter 1: The Problem and its Setting

Chapter one introduces the origin of the researched problem, and provides the background necessary for understanding the problem. To this background, the hypotheses are introduced and the delimitations, definition of terms, and assumptions, are stated.

1.8.2 Chapter 2: Review of the Related Literature

Chapter two provides an overview of the research and available literature related to the problem of instability and drift in the calibration of Type K thermocouples. This research spans the years 1939 to 1990. A manufacturers investigation into manufacturing drift, the effect of short-range ordering, and thermocouple manufacturer's specifications are reviewed.

1.8.3 Chapter 3: Temperature Metrology

Chapter three defines the aspects of traceability and temperature calibration within their context of temperature metrology on a national basis. The aspect of calibration introduces some of the complications encountered in the field of measurement.

1.8.4 Chapter 4: Calibration

Chapter four deals with the aspect of calibration that forms the core of this research investigation. Consequently calibration is dealt with in depth, with the automated calibration technique, developed as a consequence of the project, described in detail. The employment of a new calibration technique necessitated the establishment and verification of calibration uncertainty, a procedure that is covered in this chapter.

1.8.5 Chapter 5: General Procedures

Chapter five describes the preliminary informal aging trials which form the basis for determining the procedures set for conducting the formal trials, the scheduling of which, is described later in the chapter.

1.8.6 Chapter 6: The Results - Preaging

Chapter six reports and discusses the results of the trials undertaken to determine the most suitable temperature and soak period for the preaging procedure.

1.8.7 Chapter 7: The Results - Calibration Compensation

Chapter seven discusses the procedures used to conduct the process evaluation of the calibration compensation technique by evaluating the effect of work aging on thermocouple calibration. The results of these trials are presented and discussed.

1.8.8 Chapter 8: General Discussion

Chapter eight presents the final discussion of the findings, providing indication for the practical application of the proposals as well as considering the possibility for further research in this field.

CHAPTER 2

REVIEW OF THE RELATED LITERATURE

2.1 HISTORICAL OVERVIEW

It is of interest to note that for many years there has been an awareness of the problem of drift in Type K thermocouples. As early as 1939, Dahl presented a detailed paper on, "The Stability of Base Metal Thermocouples in Air from 800 to 2000^{O}F" at the Symposium on Temperature in New York (Dahl,1941). Dahl states that some processes require that a given temperature be maintained within narrow limits for extended periods, and in order to meet these requirements a more complete knowledge of thermoelectric stability of base metal thermocouple materials is necessary. Dahl's research is extensive and the results he obtains provided an adequate indication of the problem of drift. Dahl notes that whilst thermocouples are given a stabilisizing heat treatment by the manufacturer this treatment does not prove sufficient where continuous high accuracy is required. In his paper however, he does not pursue this stabilizing effect of heat treatment prior to use. Many thermocouple users in our current era, searching for inexplicable changes in their production quality, may do well to note Dahl's following remark:

> In many industrial processes, thermocouples when placed in service, are left undisturbed until there is evidence of either mechanical

> failure or serious error in the temperature indicated. However long before this occurs, the thermocouple may have changed to such an extent as to make it unreliable for accurate temperature measurement (Dahl,1941).

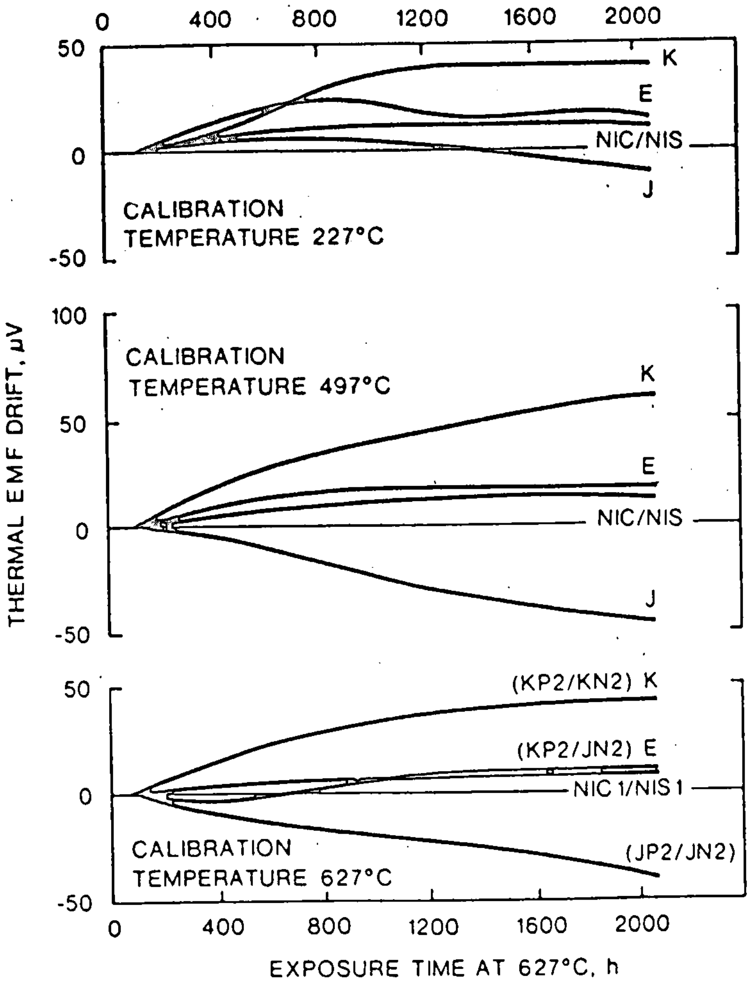
Possibly of equal historical interest is the immediate response to Dahl's paper by the Chief Engineer of Hoskins Manufacturing, W.A.Gatward and G.C.Stauffer of the Driver Harris company, thermocouple wire manufacturers. Gatward (Gatward,1941), suggests that Dahl's work is limited since it considers only thermocouples in oxidizing conditions. He suggests that in practice the opposite to Dahl's findings is true and that Chromel-Alumel thermocouples usually read low due to the presence of reducing gasses. Gatward then proceeds to present the (generalized) expectancy that the tendency to read high as suggested by Dahl will, in many instances, be "partially, wholly or excessively compensated by the tendency to read low" (Gatward,1941,p.1260).

Stauffer in like manner comments that commercial practice over the years shows that "properly installed" thermocouples do not deteriorate at the rate shown by Dahl but that their actual error value falls, (again the generalisation), "between the ideal of no change and the rapid change shown under oxidising conditions" (Stauffer,1941,p.1266).

Since both Gatward and Stauffer stress the compensating tendency of reducing conditions it is fitting to note that the current "Thermocouple Alloys" data sheet issued by British Driver-Harris Company Ltd. specifies that Type K thermocouples should be used in "fully oxidising conditions" (Driver-Harris,[sa],p.16). This requirement being to avoid the preferential oxidation of chromium which results in a form of corrosion known as "green-rot" which can be the cause of large negative errors (ASTM STP470,1981,p.24).

In a more recent statistical analysis of the accuracy of Type K thermocouple wire below 260°C, (Sanders,1974) concludes that accuracies far beyond the nominal 0.6°C could be achieved by calibration. His statistical model shows that without calibration all Type K thermocouples are not within 0.6°C below 260°C. In his summary Sanders suggests that below 260°C heat treatment would stabilize thermocouples over prolonged periods of use.

Possibly the most recent study concerning long term drift in Type K thermocouples is that done by the Australian Department of Defences Materials Research Laboratory (Burley, Hess, Howie and Coleman,1982). In a comparitive study of the new Nicrosil/Nisil thermocouple with the ANSI standard letter-designated base metal thermocouples, Burley's results reveal a 1.5°C positive drift at 497°C in a 14 AWG Type K thermocouple after a 2000 hour soak at 627°C. The equivalent drift in the Nicrosil/Nisil



NOTE: The values shown are changes from emf output values existent <u>after a preaging period of 100 hours</u> at a temperature of 627 ^{o}C.

Figure 2.1 **Calibration drift versus time. Burley's comparison of drift in thermocouple types: E,J,K and N.** (Burley *et al*.,1982,p.1162).

thermocouple is 0.4^{o}C. The study is based on a review of the various factors causing thermoelectric instability in base metal alloys. Burley summarises these factors from his earlier work with other researchers in this field:

> (i) a gradual and generally cumulative drift in thermal emf on long exposure at elevated temperatures ... due mainly to oxidation ...
>
> (ii) a short-term cyclic change in thermal emf on heating in the temperature range from 250^{o}C to 650^{o}C ... believed to be due to some structural or electronic phenomenon ...
>
> (iii) a time-independant perturbation in thermal emf in specific temperature ranges ... due to magnetic transformations which perturb the thermal emfs in Type KN in the range from 25^{o}C to 225^{o}C ... (Burley,et.al.,1982, p.1159)

With minor variations these effects are substantiated by Nicholas and White of the New Zealand Department of Scientific and Industrial Research (Nicholas and White,1982,p.107).

2.2 MANUFACTURING DRIFT INVESTIGATION

An interesting investigation of drift in Type K thermocouples during manufacture was undertaken by the Technical Department of BICC Pyrotenax Limited (Cairns, Moriarty and Wayman,1987). In the summary of this investigation it is concluded that whilst the extent of such drift is small, there is a significant difference in behaviour between differing sheath types. As this difference is most likely due to the differing stress levels set up in the core materials during drawing, it is improbable that sheath type would have any influence on

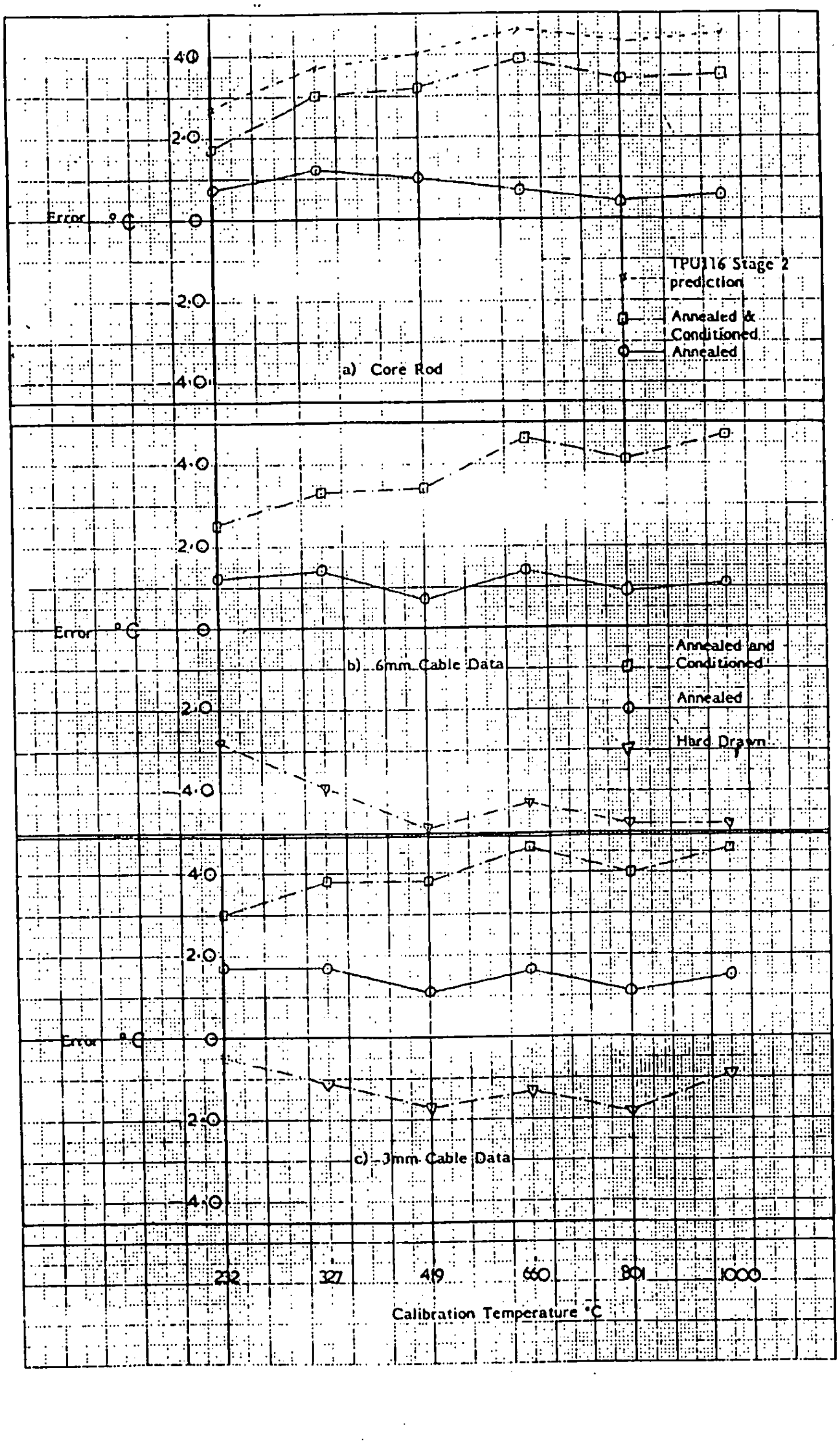


Figure 2.2 BICC test results showing effect of conditioning, (aging for 4 hours at 400°C), at three stages of the drawing process. (Cairns et al.,1987,fig.2.4).

subsequent drift. However, the consideration should be noted, and in the case of this project, the sheath material will be confined to 316 and 310 stainless steel. Of particular interest in the BICC investigation is the comprehensive set of calibration data produced concerning the thermocouple material in the hard drawn, annealed ($1050^{o}C$), and conditioned (aged, $400^{o}C$ for 4 hrs) states. In every instance at the respective calibration reference points, the conditioned thermocouple material shows an increased positive calibration offset above the equivalent annealed state calibration, which itself displays a significant positive offset above the calibration in the hard drawn state. At the $419^{o}C$ reference point the conditioned calibration value is respectively more than 2 and $4^{o}C$ greater than the equivalent annealed and hard drawn states. After each of eleven stages of drawing, the thermocouple material was annealed to relieve stress, and up to $660^{o}C$, the variance between all eleven calibration sets was less than $1^{o}C$. Whilst it must be remembered that the annealing in this case was carried out to relieve mechanically induced stress (through drawing) it is still significant that:

(i) Annealing is able, within reason, to return a thermocouple to its original state of calibration.

(ii) A very short work period will induce a significant calibration offset.

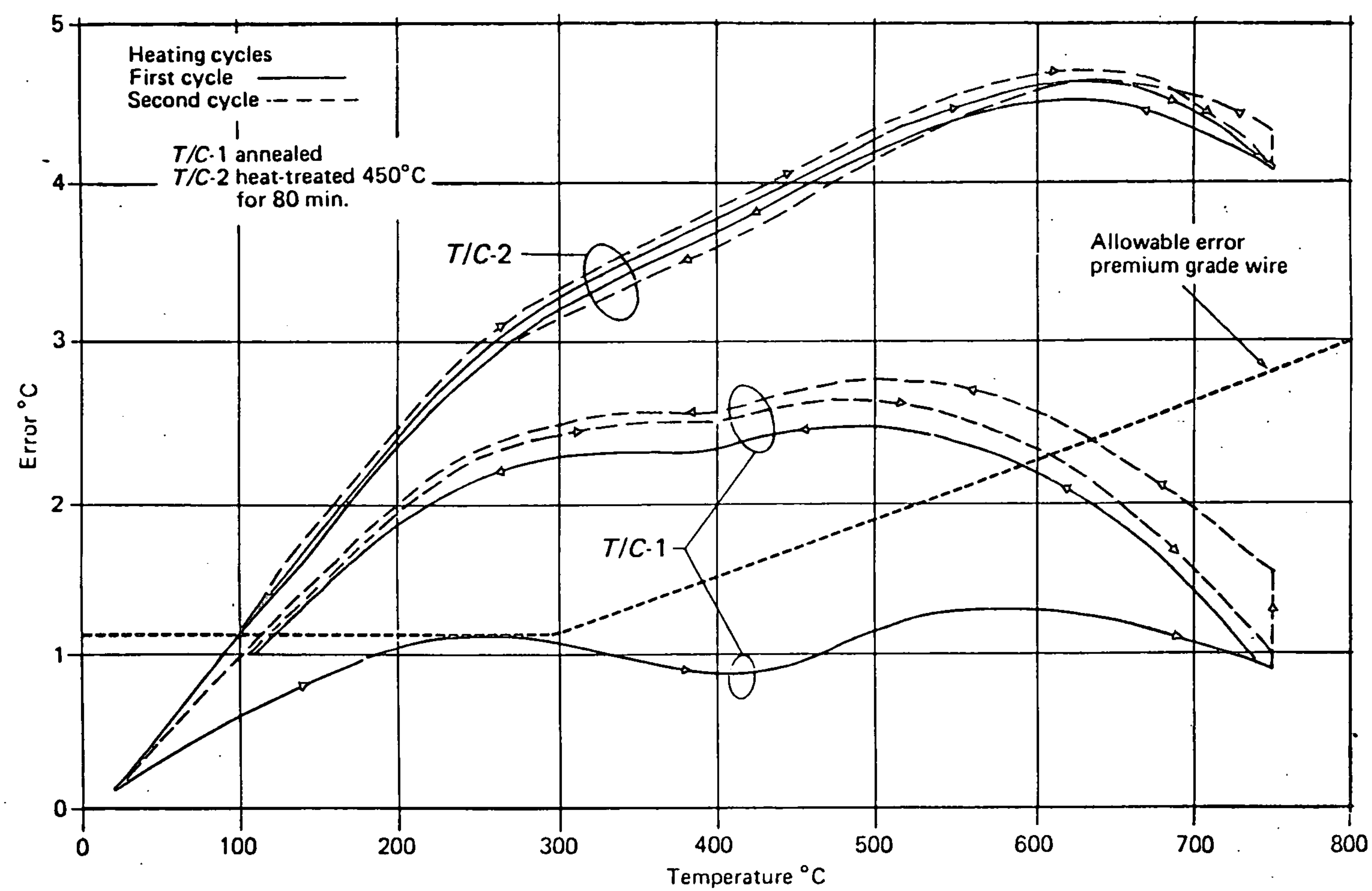


Figure 2.3 Effects of short-range ordering on the calibration of type K thermocouples. (Kerlin and Shepard,1982,p.131).

It is of further interest to note that BICC recognise the important influence that conditioning the thermocouple cable at $400^{\circ}C$ has on the emf output. A proposed amendment to BICC's purchasing specification utilises this conditioned (aged) state calibration to determine the acceptability of core material.

2.3 SHORT-RANGE ORDERING

Of particular relevance to this project is an investigation into "Temperature Measurement Errors in Type K Thermocouples due to Short-Ranged Ordering in Chromel" by Kollie, Horton, Carr, Herskovitz and Mossman referenced in the ISA Industrial Temperature Measurement course manual (Kerlin and Shepard,1982,pp.131-132). The results of Kollie's investigation into the effects of short range ordering on calibration of Type K thermocouples are depicted graphically in figure 2.3. As in the BICC investigation, reference is made to the calibration of both annealed and heat-treated ($450^{\circ}C$ for 80 min.) thermocouples. Of significance is the considerable difference between initial and second calibrations of the annealed thermocouple (T/C-1) compared to the relative repeatability of the aged thermocouple (T/C-2), figure 2.3. From these results the conclusion may be drawn that the calibration process alone is sufficient to cause a substantial drift in an annealed thermocouple, and, in this case, initial calibration data has little value other than to identify the calibration status of the thermocouple in a freshly annealed condition.

Short-range ordering is the tendency of the nickel and chromium atoms in the Chromel leg of the thermocouple to form a crystalline, ordered arrangement which changes the Seebeck coefficient of the Chromel wire. In considering the application of load thermocouples at Hulett Aluminium Profiles Pty. Ltd. where these thermocouples emerge from the furnace at differing points along their lengths on successive jobs it is of significance to note the following comment that:

> Short-range ordering introduces a thermoelectric inhomogeneity into the wires of a thermocouple where they emerge from a furnace at higher temperatures and can produce temperature errors of the order of 1 to 1.5% depending on the steepness of the temperature gradient (Kerlin and Shepard,p.132).

Considering that 1 to 1.5% represents 5 to 7.5^{O}C at 500^{O}C the potential effect this could have on the repeatability and accuracy of load thermocouples can be clearly seen.

Appendix X1 to ASTM Standard E 839 (1990 Annual Book of ASTM Standards, Volume 14.03) indicates that ordering is likely to result in a calibration shift of about 0.5% although it is noted that up to 1% has been reported. In clarifying the position concerning short-range ordering this Appendix states that:

(i) The effects of ordering are almost immediate and occur in the temperature range 320 to 540^{O}C. The ordering effect is removed when the thermoelement is heated above 600^{O}C. (X1.2.1).

(ii) Individual calibration is not generally recommended for Type K thermocouples (X1.3.1).

(iii) The Type K thermocouple is considered a stable and reliable thermocouple, however where repeatability and bias of 0.5% or better are required, the effects of ordering are a serious consideration (X1.4.1).

(iv) Ordering effects are most serious in those applications where thermocouples are subjected to temperatures in the range from 320 to 540°C. (X1.4.1.2).

2.4 MANUFACTURERS SPECIFICATION

Manufacturers specification (tolerance) for the two grades of Type K thermocouples in the temperature range, 0 to 1250°C is given as:

Standard grade: 2.2°C or 0.75% of temperature

Special grade : 1.1°C or 0.4% of temperature

(Driver-Harris,[sa],p.18; 1990 Annual Book of ASTM Standards, Volume 14.03, E 839, Appendix X2, Table X2.6). At 500°C these tolerances relate to 3.75°C and 2°C for standard and special grade respectively. It is significant to note that a number of publications relate these tolerances to a materials tolerance rather than to values that might be realistically expected during use. In the following quotes this expectancy is specifically excluded:

> Tolerances apply to new material as produced and do not allow for calibration drift during use (ASTM E 839, X2.6.2).

> The tolerances given ... are valid for new thermocouple wires at time of first heating. Output may drift significantly outside of these limits with subsequent usage (SABS 1488,1989, p9).

> It should be noted that these limits of error only apply to new, clean, annealed thermocouples prior to exposure to temperatures or conditions found in many applications. Limits of error for thermocouples are, in reality, materials tolerances ... These limits do not indicate the accuracy of the thermocouple in service (Kerlin and Shepard, p.109).

Pollock (1985,p.167) emphasises the need for periodic checks on the accuracy of thermocouples. He recommends immediate replacement the moment doubt arises as regards their accuracy. The questions this consideration gives rise to are:

(i) At what limit is the Type K thermocouple considered inaccurate?

(ii) Should the thermocouple be discarded when earlier research shows evidence of the anomaly that as the Type K thermocouple approaches its least accurate state (after prolonged use) it is also in its most stable condition?

Thermocouples are inevitably supplied in the fully annealed condition condition (Driver-Harris,[sa],p.19). For use above 320°C and below 600°C there is certain to be a short-range ordering effect of the order of 0.5% which, together with the manufacturers "materials" tolerance allowance of 0.4% for special grade, provides an expected starting accuracy of 4,5°C at 500°C. Using these values and considering a further 2°C inaccuracy due to drift and inhomogeneity, realistically combined, using the root of the sum of squares method, provides an uncertainty of around 4°C.

A realistic expectancy for the role of calibration is provided by the SABS 1488 Standard Specification for Temperature measuring sensors, Part II, which states:

> In cases where tolerance is a critical factor, the purchaser is advised to request calibration, traceable to the National Measuring Standard, for the range within which the thermocouple is expected to be used. The tolerance values given ...[manufacturers specifications]... are then only valid after the calibration factors of the calibrated thermocouple have been added to the original readings. Furthermore, the overall tolerance is then subject to the calibrating authority's stated limit of uncertainty (SABS 1488,p.9).

The stated limit of uncertainty quoted by any Temperature Laboratory providing traceability to the National Standard for a Type K thermocouple at 500°C for industrial use is unlikely to be better than +/-3°C and as continued drift must still be taken into account a 3 to 4°C uncertainty appears to be the best tolerance that may be expected from a Type K thermocouple for use in the 320 to 540°C range.

From the above considerations the need for establishing a means of providing an improved uncertainty of measurement when employing Type K thermocouples within the 320 to 540°C temperature range can be seen. This project attempts through a simple preaging technique to establish such a means specifically applicable to the aluminium heat treatment industry which has critical temperature requirements within this 320 to 540°C range.

2.5 SELF CALIBRATING THERMOCOUPLES

Isothermal Technology Ltd. have since 1985 listed among their product range a "self calibrating thermocouple". Essentially this is standard thermocouple with its hot junction surrounded by a capsule of metal with an established melting point. As the process temperature reaches this melting point the change of phase causes the temperature about the thermocouples hot junction to remain constant for a period. The difference between the indicated temperature at this point and the specified melt or calibration temperature is then the thermocouple error (Isothermal Technology Ltd.,1985,p.15). More recently an article in R&D Magazine describes seven new temperature sensor types being developed by engineers at the University of Tennessee, and hailed as having the ability to; " ... check their own accuracy and warn users of faulty measurements ..." (Katauskas,1990,p.152). One of these sensors described is based on the same principle as the Isotech thermocouple. The University of Tennessee have built and tested only one of the models. Practical considerations and the availability of the more stable Type N thermocouple are likely to preclude the general acceptance of these types of sensor for use in industrial application. Katauskas quotes ALCOA plant

What's new in thermocouples

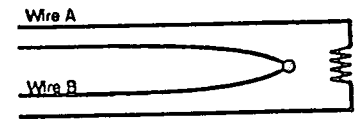
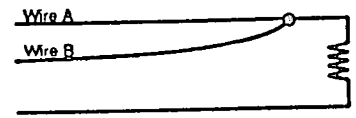
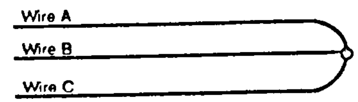
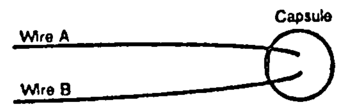
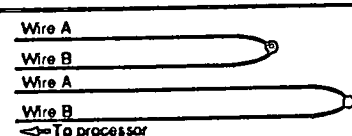
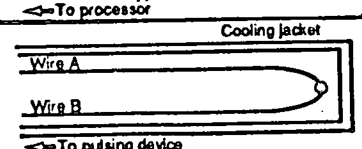
Principle	Description	Schematic
Calibration	This version of the dual/diverse sensor combines a separate thermocouple and resistance thermometer to produce two distinct temperature measurements, enabling the user to cross check results for disagreement.	
Calibration	This version of the dual/diverse sensor connects a thermocouple with a resistance thermometer in the same circuit to produce two distinct measurements which also can be cross checked for disagreement.	
Calibration	The multilead thermocouple joins three or more wires instead of the usual two to increase the number of simultaneous instrument readings. This is the only experimental sensor built by Univ. of Tennessee so far.	
Calibration	A metal with a known melting point changes phase in the capsule of the melt/freeze thermocouple, enabling the operator to calibrate the device. UT is designing a program to automate measurement.	
Performance	Two sensors are installed at different points along the dynamic compensation thermocouple to measure how long it takes heat to travel from one point on the device to another. This can increase the speed of measurement.	
Performance	A microprocessor and a sensor are attached to a gradient thermocouple to separate heat transfer effects from the actual temperature reading. This eliminates false readings caused by a temperature change in the instruments.	
Performance	A pulsed thermocouple uses a cooling jacket to protect the device from damage during measurement at high temperatures. The liquid cooling system is turned off in a brief pulse to obtain measurement.	

Figure 2.4 Proposed new thermocouple designs from the University of Tennessee. (Katauskas,1990,p.154).

engineer, Bert Kissick as considering that the understanding of error and calibration of thermocouples is very limited. Kissick points out the difficulty of determining where to start on the problem (Katauskas, p.154).

2.6 VALIDITY OF APPROACH

As noted in the historical overview, research work has frequently indicated the stabilizing effect of heat treatment upon thermocouples (Sanders,1974; Kerlin and Shepard,1982; Burley,et.al.,1982; Cairns,et.al.,1987). Burley, in plotting thermal emf drift versus exposure time, specifies that the drifts are expressed as changes from emf output values existent after 100 hours of exposure at a constant aging temperature of 627°C. This method is used to effectively eliminate the initial calibration differences and short-term emf changes due to factors such as the relief of residual internal stresses. These graphs also clearly indicate a gradual stabilizing of drift in the Type K thermocouple in relation to exposure time (Burley,et.al,1982,p.1162). Preliminary work done in the Hulett Aluminium Temperature Laboratory also demonstrates a change in both drift and short term cyclic changes related to heat treatment (Hart,1986). From this it may be said that aging and heat treatment has an effect on the drift pattern of a Type K thermocouple. The extent and predictability of this effect has, in turn, a marked influence on the level of confidence invested in any calibration procedure. It is essentially the measure of this confidence that this project examines.

CHAPTER 3

TEMPERATURE METROLOGY

3.1 INTRODUCTION

A criterion of this project is that the drift and uncertainty values established relate traceably to the national measuring standard for temperature. This criterion provides the following assurances:

(i) That a reliable indication of the extent of deviation from an absolute value is provided. Throughout this project temperature values will be given with respect to the IPTS-68 temperature scale.

(ii) That the errors in every element of the measuring chain are considered.

(iii) That future comparisons may be made directly against these data.

An essential aspect of this project is the comparison of calibration data sets, obtained prior to a thermocouple's use, to equivalent data sets, obtained up to several months later, after continued exposure to the operating environment. Consequently the integrity of the calibration data is of primary importance to this project. Temperature metrology, although a well established branch of science, has been limited in its scientific application in South Africa to the larger institutions and industry. There are at present only eight temperature laboratories in the country that have established traceable links to the national measuring standard for temperature. The implementation of Quality Assurance programs such as SABS 0157 (ISO 9000) has generated an awareness of the necessity of having the accuracy of calibration traceable to national measuring standards (National Calibration Service Directory,1989,p2). However there are still misconceptions regarding certain aspects of calibration and traceability. This chapter outlines the main elements of two of the most important aspects of the field of temperature metrology which relate to the project and more specifically to the criteria governing the admissibility of the data. The two aspects to be dealt with are traceability and thermocouple calibration techniques.

3.2 TRACEABILITY

3.2.1 CSIR

The CSIR is South Africa's legally appointed custodian of the national standards (Measuring Units and National Measuring Standards Act, Act 76 of 1973) and as such provides the legal source for traceability of measurements at all levels of accuracy. The National Calibration Service (NCS), a network of approved metrology laboratories established within industry and commerce, is managed and operated by the CSIR's Engineering Metrology Programme, Division of Production Technology. The NCS facilitates the national measuring system for calibration by providing industry with a means of obtaining traceability at the desired levels of accuracy and demonstrable by authorised, legally acceptable calibration

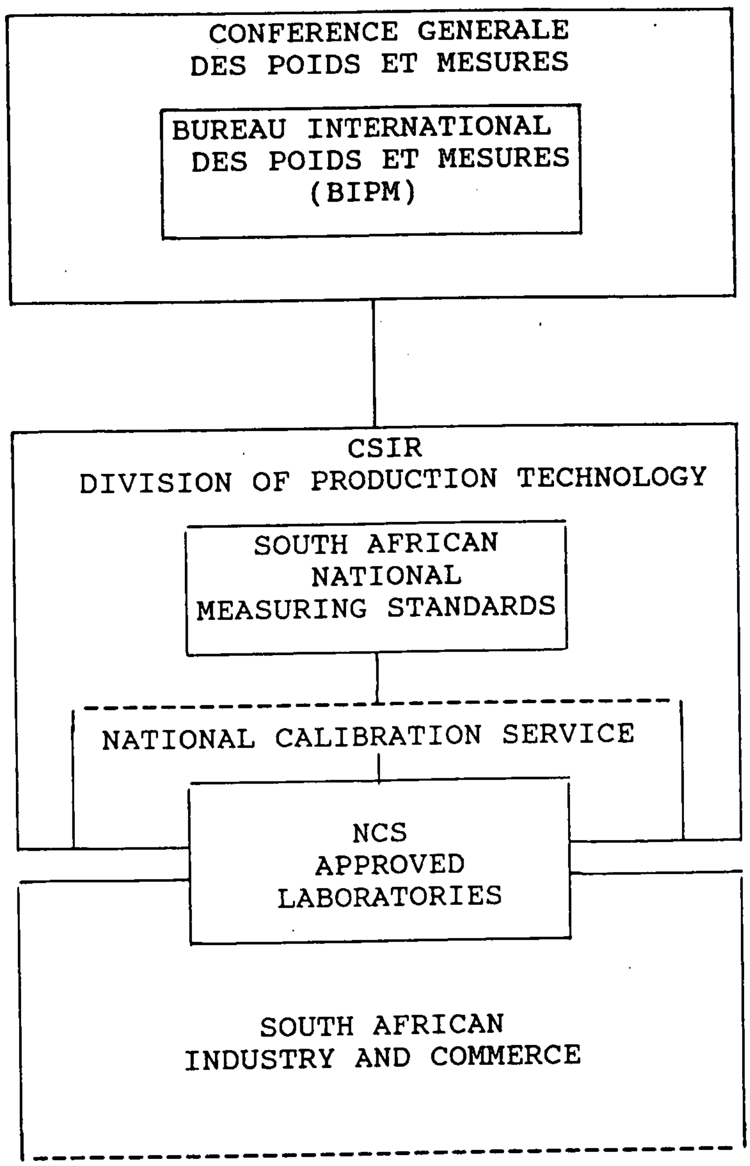


Figure 3.1 Traceability of accuracy of calibration from international standards to local industry.

certificates (National Calibration Service Directory, 1989,p.2). Figure 3.1 indicates the chain of traceability from the international authority, the Bureau International des Poids et Mesures (BIPM), through the CSIR and NCS approved laboratories, to industry and commerce.

3.2.2 Definition

The NCS defines the term "traceability to national measuring standards" (National Calibration Service Manual, NCS(1),1981,p.4) as meaning:

(i) That each measuring standard used for calibration has itself been calibrated against a standard of higher quality up to the level at which the higher quality instrument is the accepted national measuring standard.

(ii) That the frequency of such calibration is such as to establish reasonable confidence that its value will not move outside the limits of its specification between successive calibrations.

(iii) That calibration of any instrument (includes thermocouples) against a measuring standard is valid in exact terms only at the time of calibration and that instrument's performance thereafter must be inferred from a knowledge of the quality and stability of the instrument together with knowledge of its application and operating environment.

3.2.3 The Practical Transfer

The transfer between the national measuring standard for temperature and the Hulett Aluminium Temperature Laboratory (NCS laboratory approval number 301) is established by means of the Laboratory's standard thermocouple. This is a noble metal, Type S thermocouple that has been calibrated by the CSIR against a standard platinum resistance thermometer and at a number of fixed points. In the temperature range covered by this project the best measurement capability for the calibration of thermocouples, approved by the CSIR and expressed as an uncertainty, is (+/-)1^{o}C. Whilst the uncertainty of comparisons made in this project, is claimed to be in the order of (+/-)0.5^{o}C., traceability to the national measuring standard for temperature is not claimed closer than the aforementioned (+/-)1^{o}C.

3.3 THERMOCOUPLE CALIBRATION TECHNIQUES

Three basic models exist for the calibration of thermocouples and their associated instrumentation. A brief description of each of the three models is provided below. These descriptions are followed by a discussion of the specific difficulties underlying thermocouple calibration.

3.3.1 Individual Calibration

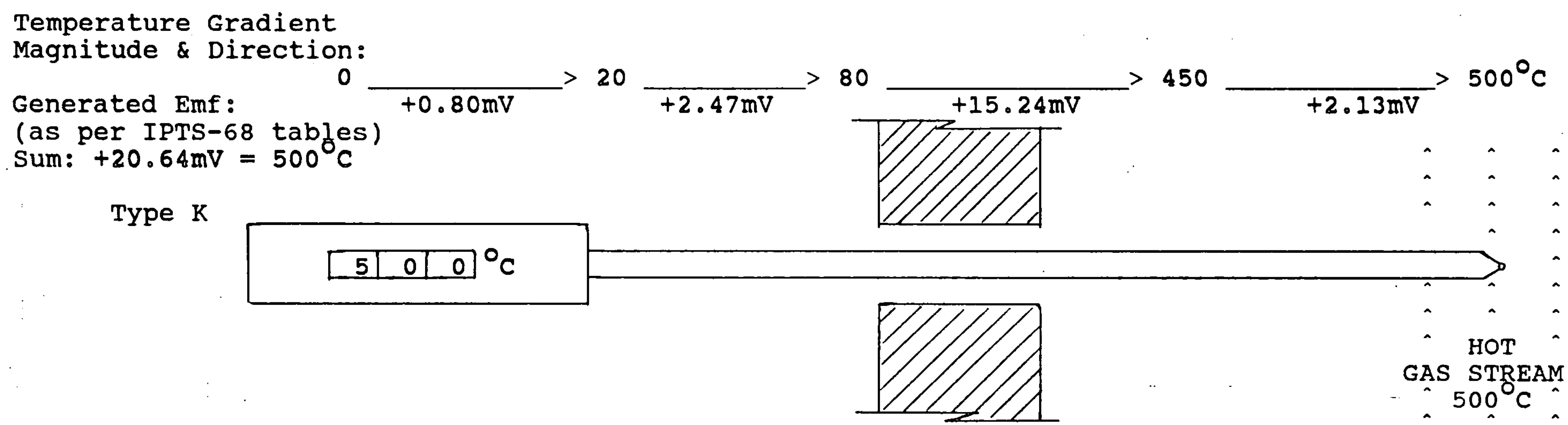
In this first calibration model, thermocouples are calibrated either by fixed point or comparison method and their individual emf/temperature relationship obtained. From this relationship a table or formula may be developed which provides the thermocouple error in relation to the relevant reference, i.e. IPTS-1968 or ITS-90. Associated instrumentation is calibrated by electrical means independently of the sensor (thermocouple).

3.3.2 Thermometry System Calibration

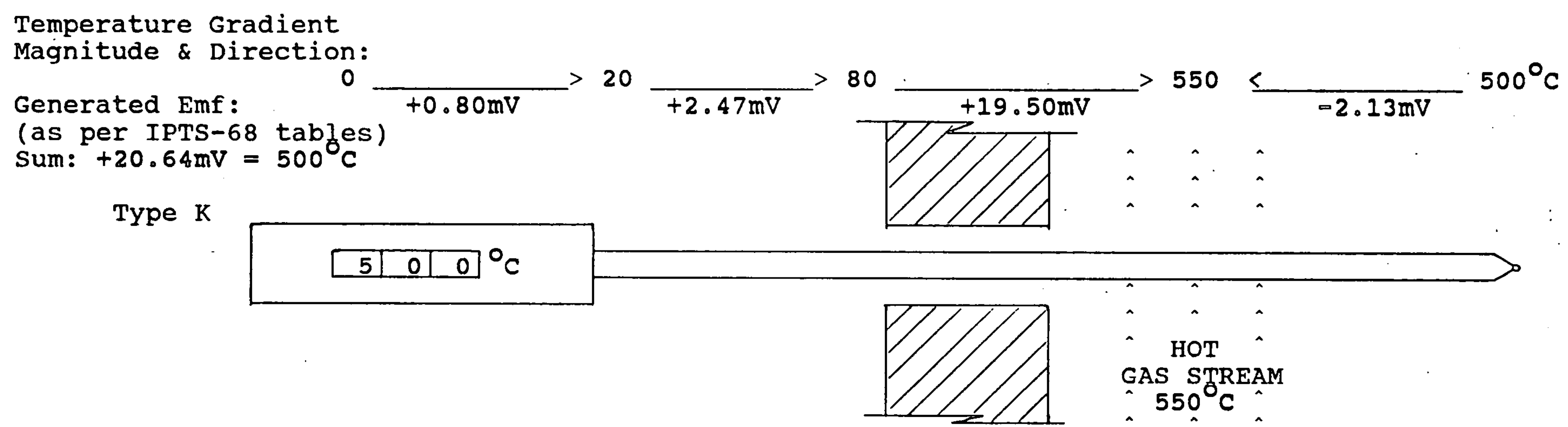
In the second model the thermocouple is calibrated together with its associated instrumentation. The calibration data then refers to this specific combination and is not valid for the individual elements. The advantage of this model is that due to there being a single calibration procedure, the total system uncertainty of calibration may be lower than the combined uncertainties of individual calibration . Calibration according to this model provides either a table of errors related to the system indication at specific temperatures, or a statement of uncertainty for the complete system covering a specific temperature range.

3.3.3 Temperature or Thermometry Installation Calibration

The third model for thermocouple calibration considers the thermocouple together with its associated instrumentation *in situ* and under representative working (loading) conditions. Calibration is by comparison to working standard thermocouples using the process as the transfer medium. For this model uncertainty is usually greater than for either of the previous models due to the variability of transfer through the process medium. This model does however provide a realistic expectation of control uncertainty. The problem of thermocouple inhomogeneity that confronts the other two calibration models is avoided by the *in situ* nature of this calibration model.



(a) Thermocouple exposed to varying gradients in one direction.



(b) Thermocouple exposed to opposing gradients.

Figure 3.2 Emf developed across thermocouple is generated in the areas of thermal gradient and not at the junction. It is still true, as the calculation shows, that the thermocouple indicates the temperature of its measuring junction.

3.3.4 The Thermocouple under Calibration

The main problem regarding the thermocouple under calibration relates to the temperature gradient zone and homogeneity in the thermocouple. Several important aspects concerning the generation of thermoelectricity within the thermocouple are the following:

(i) The thermal emf developed across a thermocouple is not generated at the hot junction (measuring point). The thermal emf is generated in any area of the thermocouple across which a thermal gradient exists.

(ii) The thermal emf developed across the thermocouple is equal to the sum of the thermal emf's generated across each thermal gradient along each leg of the thermocouple.

(iii) The value of the sum of the thermal emf's along the thermocouple is equivalent to the value that would be generated in a single temperature gradient equal to the temperatures of the hot and cold junctions of the thermocouple.

In simplified terms the above indicates that whilst the output of a thermocouple is equivalent to the temperature across its junctions, the emf is not generated at the junctions but in the areas of thermal gradient. Figure 3.2 demonstrates this principle. It must be appreciated that precisely the same gradients of temperature on the same areas of thermocouple that exist under operating conditions cannot be reproduced in the calibration laboratory. In the case of a homogeneous thermocouple, and in considering point (iii) above, this presents no problem. In the case of inhomogeneity, the thermoelectric characteristics (Seebeck coefficient) of the thermocouple wires may vary within the temperature gradient causing an error which is specific to the degree and positioning of the gradient, a situation that may cause any calibration technique, other than in situ calibration, to yield irrelevant results. The difficulty is compounded by stress and aging, likewise affecting the thermoelectric characteristics. The affect is such that the inhomogeneity, stress and aging produced during the calibration process alone is capable of introducing a calibration change in the order of 3^{o}C, (at 500^{o}C), along the heated length of the thermocouple under test. Each successive heating causes a thermoelectric signature, unique to a specific thermocouple, to be developed along the length of the thermocouple. The effect of this signature is demonstrated in the results of the preliminary aging trials presented in Chapter 5, General Procedures.

Further references are made to the above aspects of calibration in following chapters where method and calibration are discussed in more detail. Specifically these references will relate to the techniques employed by this project to avoid the problems caused by the above characteristics.

CHAPTER 4

CALIBRATION

4.1 INTRODUCTION

The primary criteria governing the selection of calibration method were determined both by the requirements of this project and by the needs related to the development of the thermocouple calibration facility within the Hulett Aluminium Temperature Laboratory. The Laboratory first established for instrument maintenance purposes was extended to service the requirement for a lower tolerance coupled to a higher level of confidence in the heat treatment practices at the Hulett Aluminium manufacturing plant in Pietermaritzburg. These criteria were determined as follows:

(i) The required calibration range is 50 to 550°C.

(ii) The uncertainty of calibration intercomparisons to be no greater than 0.5°C throughout the required calibration range. This uncertainty is a function of system repeatability and transfer capability.

(iii) The uncertainty of traceability to the National Standard to be no greater than 1°C at 500°C.

(iv) Due to the volume of thermocouples to be calibrated a facility with the capacity to carry six test thermocouples was envisaged.

(v) The multiple calibration steps required for each calibration run set the need for a facility with a fast heat up rate. Cooling rate was not considered critical as calibration would only be conducted on rising temperature.

The above criteria proved challenging and the establishment of a laboratory facility to meet these needs developed into a major aspect of this project. The foremost element in achieving the high volume, tight tolerance, calibration requirement was the use of a modified FB-08 fluidised furnace coupled with a unique automatic control algorithm. The development of the control algorithm, which permits full calibration cycles to run unattended whilst maintaining the highest level of integrity, required the largest investment in terms of resources allocated to the joint fulfillment of this research project and the establishment of the calibration facility.

The following paragraph "Calibration Method" describes the traditional component of the method selected. A further section "Calibration Techniques" discusses the more debatable elements of the calibration technique. A full description of the hardware and software employed is covered under the same section. A flowchart and description of the above mentioned control algorithm are included in the section concerning the software. The final paragraph of this section describes the trials undertaken to test the integrity of the system and to establish the calibration uncertainty.

4.2 CALIBRATION METHOD

The calibration method used follows the standard method for calibration by comparison as described in ASTM Standard E 220-86 (ASTM Standards on Thermocouples,1986,pp.5-21). In this method thermocouple calibration is based on the comparison of the indication of the thermocouple under test with the corrected value of a reference (standard) thermocouple at the same temperature.

The basic description of the calibration method that follows is guided by the arrangement of the method as detailed by ASTM Standard E 220-86 and is given to indicate which of the options provided for by this Standard have been selected for use in the calibration method employed by this project.

4.2.1 Apparatus

The comparator bath used to provide the medium by which the measuring junctions of the thermocouple under test and the reference thermocouple are brought to the same temperature is an air fluidized, aluminium oxide powder, bath having an operating range extending from ambient to $700^{o}C$.

A reference junction temperature of $0^{o}C$ for the thermocouples under test and for the reference thermocouple is achieved through the ice point prepared in a reference bath according to ASTM Standard E 563-73 (Reapproved 1987)(Annual Book of ASTM Standards,Volume,14.03,1990, pp.254-257).

A Group C emf-measuring instrument, a digital voltmeter, directly coupled to an automatic scanning unit located on the copper portion of the circuit is provided for the acquisition of the thermocouple signals.

In the case of preliminary calibration work on bare wire thermocouples an "Alsint" two hole protective support tube is utilised. The three millimetre mineral insulated stainless steel sheathed thermocouples do not require additional protection nor support.

4.2.2 Reference Thermometer

For the purpose of this section (paragraph 4.1) of chapter 4 the use of the term "reference thermometer" defined as, "a thermometer whose calibration is known within a certain specified accuracy", by ASTM Standard E 220-86 has been retained. In this project's definition of terms (chapter 1), the equivalent term more specifically defined is "standard thermocouple".

The reference thermometer used for comparison is a Type S platinum-10% rhodium/platinum thermocouple calibrated by the CSIR for the temperature range 0 to 1065^{o}C.

4.2.3 General Procedure

ASTM Standard E 220-86 general procedure method B (ASTM Standard on Thermocouples,1986,p.9) is used. Each thermocouple is connected in sequence to the measuring instrument through an automatic scanner. Figure 4.1 illustrates this basic arrangement.

4.2.4 Calibration Procedure

As ASTM Standard E 220-86 provides no specific procedure for the calibration of thermocouples in a fluidized bath, a full description of the procedure used is given in paragraph 4.4, "Calibration Procedure".

4.2.5 Calculations

Details of the calculations utilised in the calibration method used are given in paragraph 4.3, "Calibration Technique".

4.2.6 Accuracy

When employing interpolated values ASTM Standard E 220-86 suggests that uncertainty will be in the order of $1^{o}C$ (ASTM Standards on Thermocouples,1986,E 220-86,Table 1). For the purpose of avoiding this relatively substantial uncertainty all comparative calibrations will be carried out at the same reference temperatures without the need for interpolation tables. This approach allows for an uncertainty in the order of $0.5^{o}C$.

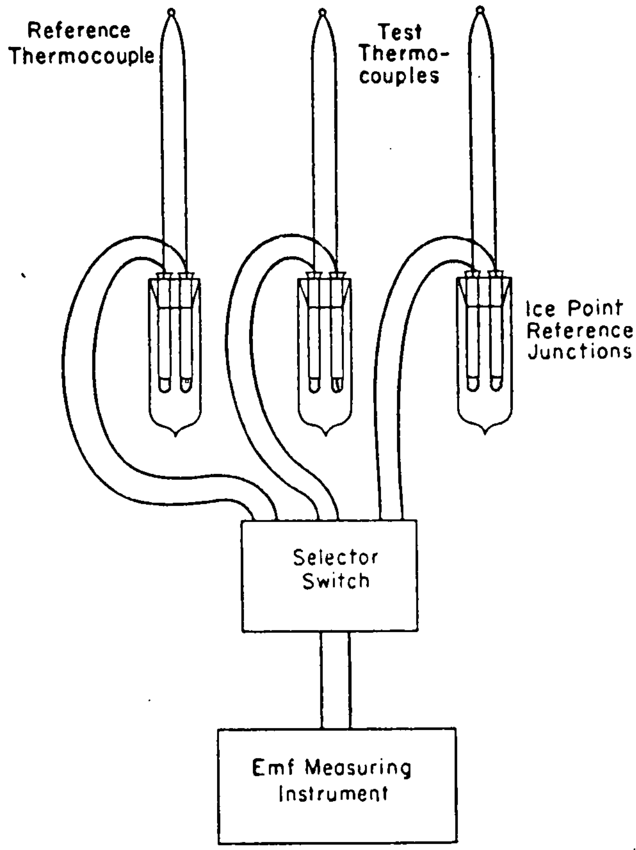


Figure 4.1 Schematic of thermocouple connection. Arrangement showing selector switch with single measuring instrument. (ASTM Standard 220-86,fig.2).

4.3 CALIBRATION TECHNIQUE

A modified Techne FB-08 fluidised bath is used to provide the transfer medium for calibration by comparison of the thermocouple under test to the standard thermocouple. The accuracy attained at each calibration point depends, other than on the accuracy of the standard, upon the degree to which the standard and test thermocouples are maintained at the same temperature (ASTM STP 470B,1981,paragraph 8.1.7.2). Whilst the heat transfer rate in the fluidised bath is inferior to both oil and salt baths it is vastly superior to the heat transfer obtained in air as shown by the following figures as compiled by Edwards (1980,p.3):

Heat transfer in kcal/hr/mm/deg.C	
Air circulation furnace	2.5 X 10E-5 to 5 X 10E-5
Fluidised bath	40 X 10E-5 to 60 X 10E-5
Oil bath	100 X 10E-5 to 150 X 10E-5
Salt bath	120 X 10E-5 to 180 X 10E-5

The manufacturers, Techne Cambridge Limited, of the Techne FB-08 fluidised bath that was used in this project specify a short term stability of 0.01^{o}C using the "dead bed" calibration technique and 0.5^{o}C using a freely fluidised bath (Techne Cambridge Limited,1986). The "dead bed" calibration technique is unique to fluidised baths and was discovered accidentally by engineers at the British Central Electricity Generating Board, (CEGB), and developed jointly by the Board and Techne Cambridge Limited. The technique involves stabilising the bath temperature, collapsing the fluidised bed and then taking a series of comparison readings. The concept as described by Fairburn(1975) is that in collapsing the bed the fluidised medium is converted from a medium with good heat transfer characteristics to one with good insulation in a stable isothermal state (Fairburn,1975,pp.3-4). Where thermocouples differ in construction to the reference and consequently have differing heat transfer characteristics and conduction losses the determining of the correct "calibration point" within the short stability period may be problematic. An investigation by Edwards[1980] found that the response of sensors subsequent to de-fluidising the bath varied with the type of sensor (Edwards,[1980], p.4). For this reason a "live bed" technique utilising a principle similar to that described by Edwards in his paper on "The Design and Manufacture of Equipment for Temperature Calibration" was used. Edwards describes a computer controlled system which ensures a stability of 0.1^{o}C, and it is relevant to note Edwards' comment relating to the CEGB calibration practice:

> As now operating, the system gives such consistent results that CEGB have been able to revert to "live bed" calibration and achieve better results than were previously possible using the dead bed technique, with a consequent reduction in overall calibration time ... (Edwards,[1980],p.6).

Further support is provided for the "live bed" technique by consideration of the concept of what constitutes an "equilibrium state", the state ideally prevailing within the working zone of a calibration furnace. Nicholas and White (1982) consider that the definition of an "equilibrium state" commonly given as, a state in which the physical variables are all constant in time, is not completely adequate and consider it more appropriate to look for the absence of systematic trends in the time averages of the physical variables (Nicholas and White, 1982,p.154).

In an adaption of the "live bed" technique and in an attempt to further decrease the uncertainty of measurement an averaging technique that compensates for any symmetrical systematic trends in the time averages of the standard and test thermocouples, coupled with variance detection to delimit the average data set has been used. This technique is described in detail under the "Software" subsection of this chapter.

4.3.1 The Hardware

A description of the calibration furnace and calibration measuring equipment together with a functional description and schematic of this interconnection is itemised under each respective element as follows:

a. Calibration Furnace

1. Specification

Maker: Techne Cambridge Limited.

Model: Techne FB-08.

Temperature range: 50 to 700^{o}C.

Heater power: 3 kW.

Heat up rate: 45 minutes from 20 to 700^{o}C.

2. Modifications

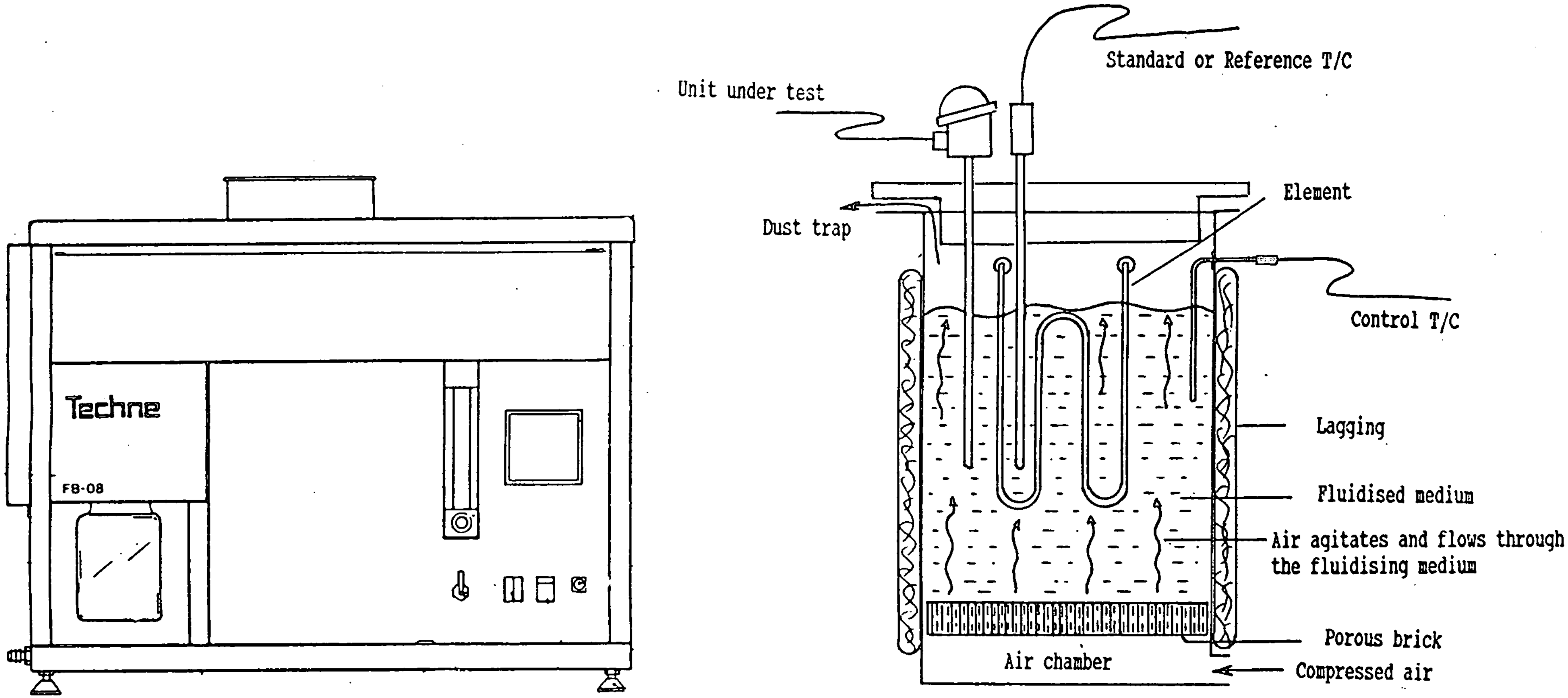
Three major modifications to the standard bath were made:

(a) **The control system**

A Eurotherm Limited, model 818P temperature controller with communications capability was fitted as the furnace controller. A thyristor power regulator controlling in the phase angle control mode was fitted to provide smooth switching of the furnace elements.

(b) **The fluidising air system**

The fluidising air stepped flow control system was replaced by a linear system providing a rate of fluidising air flow continuously proportional to bath temperature.



(a) Front view

(b) Basic arrangement of fluidising chamber

Figure 4.2 Techne FB-08 fluidised furnace.

(c) **The dust extraction system**

A ducting system with extractor fan replaced the standard filter and provided a negative pressure to the removal vent. This system enabled the venturi air flowrate to be lowered with a subsequent reduction in noise. A purge rotameter was fitted to provide manual control and indication of the venturi air flow.

(d) **Operation**

The bed of fine chromatographic grade dry alumina oxide powder is fluidised to a controlled extent by low pressure compressed air (figure 4.2). The resulting fluidised medium is heated by immersion heaters to a temperature controlled to a resolution of 0.1^{o}C by the control circuit. Thermocouples to be calibrated together with the standard and reference thermocouples are immersed to a fixed depth in the bath.

b. **Scanner**

1. **Specification**

Maker: John Fluke Manufacturing Company.

Model: 2300A Scanner.

Fitted option: Thermocouple scanner card.

Resolution: 1 microvolt.

2. **Function**

The function of the scanner is to accept analog signals from the copper side of the thermocouple ice reference junctions and to supply these signals, in sequence, to the measuring instrument.

3. **Operation**

The scanner is set to continuously scan 10 channels at an interval of 2 seconds per channel. No thermocouple linearisation nor cold junction compensation is performed. The scanner is coupled by dedicated bus to the 2190A digital thermometer.

c. **Digital Thermometer**

1. **Specification**

Maker: John Fluke Manufacturing.

Model: 2190A Digital Thermometer.

Resolution: 1 microvolt.

Range: 99.999 millivolts.

A/D Conversion: Dual-slope.

2. **Function**

The function of the Digital Thermometer is to convert the analog signal received from the Scanner into a digital signal, to display this signal and to further prepare a transmission package containing the respective channel number and digitised signal for transmission on the RS-232 link to the process computer.

3. **Operation**

The Digital Thermometer operates in the voltage mode, consequently the thermocouple linearization functions are not employed. Measured value is displayed and retransmitted directly in microvolts.

d. **Process Computer**

1. **Specification**

Maker: John Fluke Manufacturing.

Model: 1722A Instrument Controller

2. **Function**

The Process Computer functions as a supervisory controller performing the following tasks according to software programmed requirements:

(i) Accepts calibration program and thermocouple details input via keyboard and touch-screen.

(ii) Calls for and processes thermocouple data from digital thermometer.

(iii) Communicates with fluidised bath via Eurotherm 818P controller on dedicated RS-232 port.

(iv) Outputs calibration reports and certificates via printer and plotter linked via common IEEE port.

3. **Operation**

Calibration software is loaded by mini disc. Program and thermocouple details are entered by keyboard and touchscreen. Thereafter the calibration cycle is fully automatic.

e. **Peripheral Devices**

1. **Printer**

An Epson Corporation, model FX-85 dot matrix printer provides hard copy reports of thermocouple, program and calibration details.

2. **Plotter**

A Hewlett-Packard Company, model HP 7470A two pen graphics plotter provides a graphic plot of calibration details on a certificate format.

f. **Interconnection**

The hardware interconnection is illustrated in the schematic diagram, figure 4.3. Functionally the thermocouple signals are processed as follows:

1. **Calibration Furnace**

Thermocouple measuring junctions are placed in the furnace and heated to the required temperature.

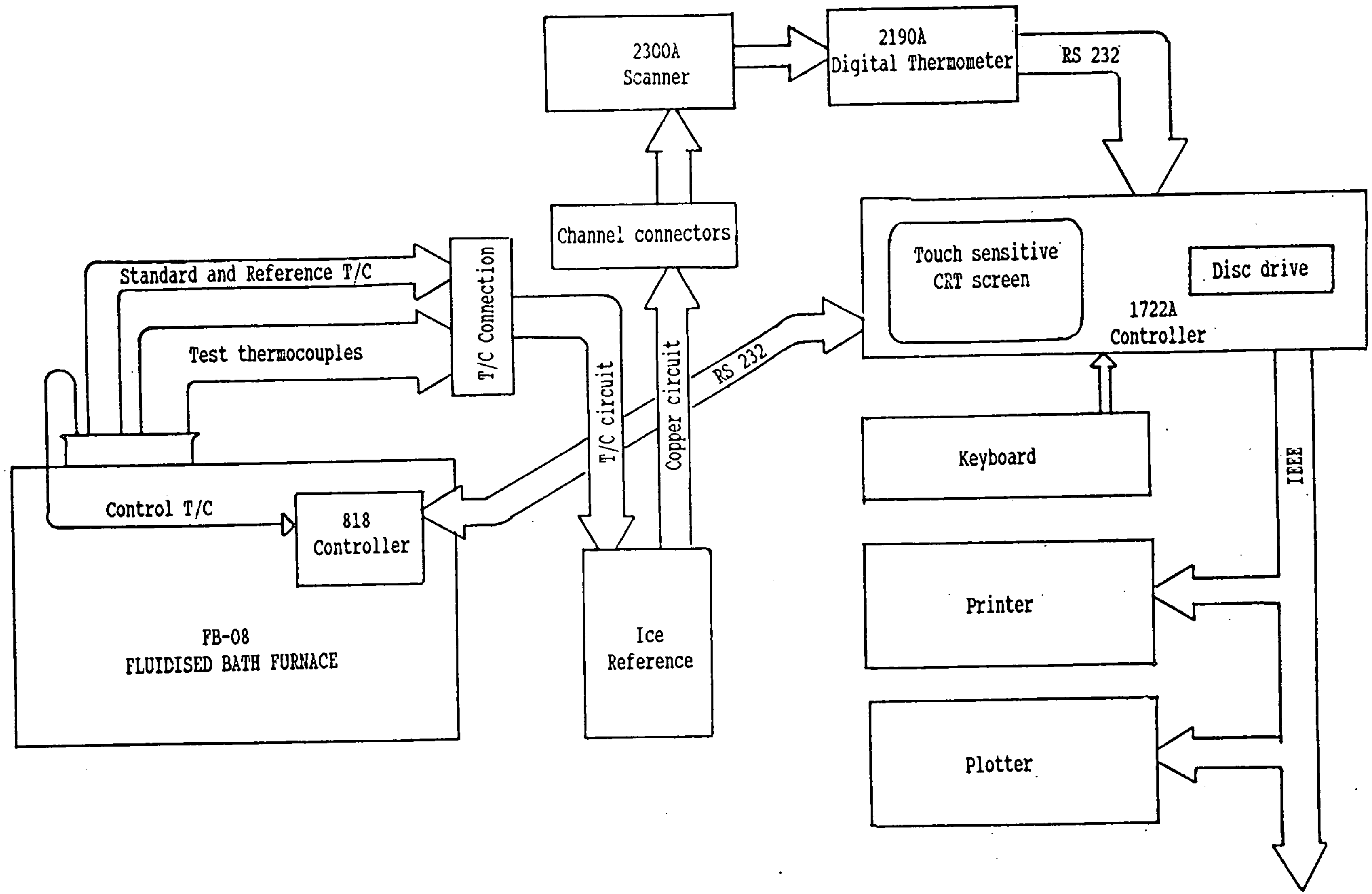


Figure 4.3 Calibration hardware interconnection for automatic thermocouple calibration facility.

2. **Ice Bath**

The thermocouples are connected by matching thermocouple wires to the reference junctions located in an ice bath. Emf developed across the thermocouple is a function of the difference in temperature between the measuring and reference junctions. The signal wires emanating from the reference junction are termed the "copper" side of the circuit and act purely as conductors.

3. **Scanner**

All thermocouple signals enter the scanner individually. The scanner selects the signals in sequence at 2 second intervals and transmits the analog signal selected to the digital thermometer.

4. **Digital Thermometer**

The digital thermometer converts the incoming analog signal into a digitally expressed microvolt value. This signal is transmitted via an RS232 link to the process computer.

5. **Process Computer**

From the microvolt values transmitted to the process computer the computer selects the value linked to the standard thermocouple measuring channel and using calibration data unique to the standard thermocouple, computes the equivalent temperature. The microvolt values linked to the thermocouples under test are treated in similar manner, the emf/temperature reference used for these thermocouples, being the relevant standard IPTS-68 table. Subtracting the temperature of the standard thermocouple from that of the thermocouple under test provides the deviation value for that thermocouple at that specific temperature. The process computer files these respective deviation values on disc in addition to providing further processing for translating the data into suitable report or graphics format for exporting to the printer or plotter.

4.3.2 The Software

All software referred to in this section refers to software run on the process computer. The automation program called LAB87 was written in Basic to provide the process computer with capability of conducting a full calibration procedure. This procedure as commonly conducted by a metrologist involves:

(i) Setting the calibration furnace temperature.

(ii) Determining the point at which furnace temperature stability is achieved.

(iii) Recording consecutively the indicated temperatures or emf values of the thermocouple under test and the standard thermocouple.

(iv) Repeating the above readings until the consistency of the recorded data is established.

(v) Repeating steps (i) to (iv) for as many calibration points as are required.

(vi) Screening the recorded data and rejecting any datum with suspected gross error.

(vii) Averaging or in other ways statistically treating the data.

(viii) Evaluating the data against the respective calibration references.

(ix) Issuing a unique calibration identification number and filing a report.

Due to the limited memory capability of the standard version of the computer used, the program LAB87, was divided into five sub-programs. A brief description of the function of each the secondary sub-programs is provided with a detailed description and flow diagram given of the program containing the control algorithm.

a. LAB87.BAS

The initial program is termed LAB87.BAS and performs the primary function of assembling the data file containing thermocouple and calibration details. This is achieved by prompting the operator for input and screening the entered data. Should the operator persist in attempting to load invalid data a help function is automatically engaged. The help function details the valid options available. The sequence of the program is as follows:

(i) Prompt for and screen operator authorisation code.

(ii) Allocate a sequentially selected "Test number" and real time date.

(iii) Prompt for and screen the input of the following thermocouple details:
Channel number
Thermocouple serial number
Thermocouple Type
Data sheet record number
Thermocouple description
Thermocouple supplier

(iv) Prompt for and screen the input of the following test details:
Starting temperature
Final temperature
Calibration interval

(v) Transfer the data to the virtual array file named "TEST.VRT".

(vi) Access the printer to provide a hardcopy record of assembled data (figure 4.4).

(vii) Load the following sub-program.

b. TC.BAS

The sub-program TC.BAS runs as a continuation of LAB87.BAS and fulfills the function of checking that channels selected have valid signals attached. This check is accomplished by the following procedure:

(i) Record signals from all selected channels.

(ii) Increment furnace control setpoint to 5^{o}C above present measured variable value.

(iii) After one minute again record signals from all selected channels.

(iv) Return furnace setpoint to original setting.

(v) Subtract first data set from second. Evaluate results as follows:
Result positive: Connection good
Result negative: Reverse polarity
Result equals zero: Short circuit
No signal or signal overange: Open circuit
No result: Channel selected but not logged

(vi) Access printer and print results (figure 4.4). If no errors exist load following sub-program. If errors exist provide audible response and wait for program to be rerun.

HULETT ALUMINIUM

CALIBRATION LISTING
TEST No. 10294

PROGRAM: LAB87 Version Dev 4.0

07-Dec-90 10:19

CHANNEL	T/C No.	TYPE	DATA SHEET	DESCRIPTION	MANUFACTURER
Std.	01-90	S			
Ref.	13-85	S			
0	2185	K	2086020790	KK/8/700/A	REPUBLIC
1	3246	J	0008040690	J/1.6/5m/LA	REPUBLIC
2	3776	J	7500031090	HA/J/3/Z-(J1)	TEMP.CONTROLS
3	3778	J	7500031090	HA/J/3/Z-(J2)	TEMP.CONTROLS
4	3782	K	7500041090	HA/K/3/Z-(K1)	TEMP.CONTROLS
5	3786	K	7500041090	HA/K/3/Z-(K2)	TEMP.CONTROLS
6	3852	K	0001011190	HA/K/30/G-G	GILECTRIC

Calibration from: 50°C to: 550°C at: 50°C intervals.

PROGRAM AUTHORIZED: R.W.W.Hart

Signed: ______________

Figure 4.4 **LAB87 Calibration test listing of thermocouple and program details. Unique test number, date of calibration and person responsible for authorising the test are shown.**

c. FB08.BAS

FB08.BAS comprises the main elements of LAB87, the primary element being the control algorithm. The function of the program is to supervise the control of the furnace (actual control is handled by the discrete temperature controller), to process the incoming data and to evaluate the conditions pertaining to stability, thereby ensuring the integrity of the calibration data. The following description provided, explicates the attendant schematics, figures 4.5 to 4.8.

1. **Stage 1 (Figure 4.5)**

(a) Startup routine

The startup routine opens the required files, loads relevant thermocouple Type, channel and program details, loads registers with thermocouple coefficients required for computing temperature/emf characteristics and clears the data storage files. Finally the startup routine sets up the loop that determines the number of calibration points through which the program runs.

Table 4.1 TABLE.VRT Correction table. Original values.

T/C TABLE -- ORIGINAL LISTING --

07-Dec-90 09:06 Page 1

TEMP	0	1	2	3	4	5	6	7	8	9
0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
10	-0.0	-0.0	-0.0	-0.0	-0.0	-0.0	-0.0	-0.0	-0.0	-0.0
20	-0.1	-0.1	-0.1	-0.1	-0.1	-0.1	-0.1	-0.1	-0.1	-0.1
30	-0.2	-0.2	-0.2	-0.2	-0.2	-0.2	-0.2	-0.2	-0.2	-0.2
40	-0.4	-0.4	-0.4	-0.4	-0.4	-0.4	-0.4	-0.4	-0.4	-0.4
50	-0.5	-0.5	-0.5	-0.5	-0.5	-0.5	-0.5	-0.5	-0.5	-0.5
60	-0.7	-0.7	-0.7	-0.7	-0.7	-0.7	-0.7	-0.7	-0.7	-0.7
70	-0.8	-0.8	-0.8	-0.8	-0.8	-0.8	-0.8	-0.8	-0.8	-0.8
80	-1.0	-1.0	-1.0	-1.0	-1.0	-1.0	-1.0	-1.0	-1.0	-1.0
90	-1.1	-1.1	-1.1	-1.1	-1.1	-1.1	-1.1	-1.1	-1.1	-1.1
100	-1.3	-1.3	-1.3	-1.3	-1.3	-1.3	-1.3	-1.3	-1.3	-1.3
110	-1.5	-1.5	-1.5	-1.5	-1.5	-1.5	-1.5	-1.5	-1.5	-1.5
120	-1.7	-1.7	-1.7	-1.7	-1.7	-1.7	-1.7	-1.7	-1.7	-1.7
130	-1.8	-1.8	-1.8	-1.8	-1.8	-1.8	-1.8	-1.8	-1.8	-1.8
140	-2.0	-2.0	-2.0	-2.0	-2.0	-2.0	-2.0	-2.0	-2.0	-2.0
150	-2.2	-2.2	-2.2	-2.2	-2.2	-2.2	-2.2	-2.2	-2.2	-2.2
160	-2.3	-2.3	-2.3	-2.3	-2.3	-2.3	-2.3	-2.3	-2.3	-2.3
170	-2.5	-2.5	-2.5	-2.5	-2.5	-2.5	-2.5	-2.5	-2.5	-2.5
180	-2.6	-2.6	-2.6	-2.6	-2.6	-2.6	-2.6	-2.6	-2.6	-2.6
190	-2.8	-2.8	-2.8	-2.8	-2.8	-2.8	-2.8	-2.8	-2.8	-2.8
200	-2.9	-2.9	-2.9	-2.9	-2.9	-2.9	-2.9	-2.9	-2.9	-2.9
210	-3.0	-3.0	-3.0	-3.0	-3.0	-3.0	-3.0	-3.0	-3.0	-3.0
220	-3.2	-3.2	-3.2	-3.2	-3.2	-3.2	-3.2	-3.2	-3.2	-3.2
230	-3.3	-3.3	-3.3	-3.3	-3.3	-3.3	-3.3	-3.3	-3.3	-3.3
240	-3.4	-3.4	-3.4	-3.4	-3.4	-3.4	-3.4	-3.4	-3.4	-3.4
250	-3.5	-3.5	-3.5	-3.5	-3.5	-3.5	-3.5	-3.5	-3.5	-3.5
260	-3.6	-3.6	-3.6	-3.6	-3.6	-3.6	-3.6	-3.6	-3.6	-3.6
270	-3.6	-3.6	-3.6	-3.6	-3.6	-3.6	-3.6	-3.6	-3.6	-3.6
280	-3.7	-3.7	-3.7	-3.7	-3.7	-3.7	-3.7	-3.7	-3.7	-3.7
290	-3.8	-3.8	-3.8	-3.8	-3.8	-3.8	-3.8	-3.8	-3.8	-3.8
300	-3.8	-3.8	-3.8	-3.8	-3.8	-3.8	-3.8	-3.8	-3.8	-3.8
310	-3.9	-3.9	-3.9	-3.9	-3.9	-3.9	-3.9	-3.9	-3.9	-3.9
320	-3.9	-3.9	-3.9	-3.9	-3.9	-3.9	-3.9	-3.9	-3.9	-3.9
330	-3.9	-3.9	-3.9	-3.9	-3.9	-3.9	-3.9	-3.9	-3.9	-3.9
340	-3.9	-3.9	-3.9	-3.9	-3.9	-3.9	-3.9	-3.9	-3.9	-3.9
350	-3.9	-3.9	-3.9	-3.9	-3.9	-3.9	-3.9	-3.9	-3.9	-3.9
360	-4.0	-4.0	-4.0	-4.0	-4.0	-4.0	-4.0	-4.0	-4.0	-4.0
370	-4.0	-4.0	-4.0	-4.0	-4.0	-4.0	-4.0	-4.0	-4.0	-4.0
380	-3.9	-3.9	-3.9	-3.9	-3.9	-3.9	-3.9	-3.9	-3.9	-3.9
390	-3.9	-3.9	-3.9	-3.9	-3.9	-3.9	-3.9	-3.9	-3.9	-3.9

Table 4.2 TABLE.VRT Correction table. Revised values. Only values utilised in prior calibration runs are affected.

T/C TABLE -- REVISED LISTING --

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TEMP	0	1	2	3	4	5	6	7	8	9
0	0.0	0.0	0.0	0.0	0.0	0.0	-0.0	-0.0	-0.0	-0.0
10	-0.0	-0.0	-0.1	-0.1	-0.1	-0.1	-0.1	-0.1	-0.1	-0.1
20	-0.1	-0.1	-0.2	-0.2	-0.2	-0.2	-0.2	-0.2	-0.2	-0.2
30	-0.2	-0.3	-0.3	-0.3	-0.3	-0.3	-0.3	-0.3	-0.3	-0.4
40	-0.4	-0.4	-0.4	-0.4	-0.4	-0.4	-0.5	-0.5	-0.5	-0.5
50	0.0	-0.5	-0.5	-0.6	-0.6	-0.6	-0.6	-0.6	-0.6	-0.6
60	-0.7	-0.7	-0.7	-0.7	-0.7	-0.7	-0.7	-0.8	-0.8	-0.8
70	-0.8	-0.8	-0.8	-0.9	-0.9	-0.4	-0.9	-0.9	-0.9	-1.0
80	-0.1	-1.0	-1.0	-1.0	-1.0	-1.1	-1.1	-1.1	-1.1	-1.1
90	-1.1	-1.2	-1.2	-1.2	-1.2	-1.2	-1.2	-1.3	-1.3	-1.3
100	-0.6	-1.3	-1.3	-1.4	-1.4	-1.4	-1.4	-1.4	-1.5	-1.5
110	-1.5	-1.5	-1.5	-1.5	-1.6	-1.6	-1.6	-1.6	-1.6	-1.6
120	-0.4	-1.7	-1.7	-1.7	-1.7	-0.7	-1.8	-1.8	-1.8	-1.8
130	-1.8	-1.8	-1.9	-1.9	-1.9	-1.9	-1.9	-1.9	-2.0	-2.0
140	-1.1	-2.0	-2.0	-2.0	-2.1	-2.1	-2.1	-2.1	-2.1	-2.1
150	-1.7	-2.2	-2.2	-2.2	-2.2	-2.2	-2.3	-2.3	-2.3	-2.3
160	-1.6	-2.3	-2.4	-2.4	-2.4	-2.4	-2.4	-2.4	-2.5	-2.5
170	-2.5	-2.5	-2.5	-2.5	-2.5	-1.8	-2.6	-2.6	-2.6	-2.6
180	-1.7	-2.6	-2.7	-2.7	-2.7	-2.7	-2.7	-2.7	-2.7	-2.8
190	-2.8	-2.8	-2.8	-2.8	-2.8	-2.8	-2.9	-2.9	-2.9	-2.9
200	-1.8	-2.9	-2.9	-3.0	-3.0	-3.0	-3.0	-3.0	-3.0	-3.0
210	-3.0	-3.1	-3.1	-3.1	-3.1	-3.1	-3.1	-3.1	-3.1	-3.2
220	-3.2	-3.2	-3.2	-3.2	-3.2	-2.3	-3.2	-3.2	-3.3	-3.3
230	-3.3	-3.3	-3.3	-3.3	-3.3	-3.3	-3.3	-3.3	-3.4	-3.4
240	-3.2	-3.4	-3.4	-3.4	-3.4	-3.4	-3.4	-3.4	-3.5	-3.5
250	-2.4	-3.5	-3.5	-3.5	-3.5	-3.5	-3.5	-3.5	-3.5	-3.6
260	-2.8	-3.6	-3.6	-3.6	-3.6	-3.6	-3.6	-3.6	-3.6	-3.6
270	-2.9	-3.6	-3.7	-3.7	-3.7	-2.5	-3.7	-3.7	-3.7	-3.7
280	-3.2	-3.7	-3.7	-3.7	-3.7	-3.7	-3.7	-3.7	-3.8	-3.8
290	-2.8	-3.8	-3.8	-3.8	-3.8	-3.8	-3.8	-3.8	-3.8	-3.8
300	-2.8	-3.8	-3.8	-3.8	-3.8	-3.8	-3.8	-3.8	-3.9	-3.9
310	-2.9	-3.9	-3.9	-3.9	-3.9	-3.9	-3.9	-3.9	-3.9	-3.9
320	-3.3	-3.9	-3.9	-3.9	-3.9	-2.0	-3.9	-3.9	-3.9	-3.9
330	-2.9	-3.9	-3.9	-3.9	-3.9	-3.9	-3.9	-3.9	-3.9	-3.9
340	-2.8	-3.9	-3.9	-3.9	-3.9	-2.4	-3.9	-3.9	-3.9	-3.9
350	-2.2	-3.9	-3.9	-4.0	-4.0	-4.0	-4.0	-4.0	-4.0	-4.0
360	-3.0	-4.0	-4.0	-4.0	-4.0	-4.0	-4.0	-4.0	-4.0	-4.0
370	-2.8	-3.9	-3.9	-3.9	-3.9	-2.3	-3.9	-3.9	-3.9	-3.9
380	-2.7	-3.9	-3.9	-3.9	-3.9	-3.9	-3.9	-3.9	-3.9	-3.9
390	-2.8	-3.9	-3.9	-3.9	-3.9	-3.9	-3.9	-3.9	-3.9	-3.9

(b) **Control calibration correction**

The initial required value (first calibration point) temperature is checked against a setpoint correction table, (refer table 4.2), in a file called TABLE.VRT. This table contains the calibration offsets for all unit values of temperature from 0 to 700^{o}C for the control thermocouple which is part of the calibration furnace discrete control loop. This correction is necessary since the control thermocouple signal will be linearised directly by the furnace temperature controller according to the standard IPTS-68 tables. This calibration factor is added to the required value temperature and transmitted as the setpoint value from the process computer to the temperature controller.

As an example of the above procedure for a required actual temperature of 300^{o}C the corresponding calibration offset, found in table 4.2, is -2.8^{o}C. The setpoint value to be transmitted to the controller is 297.2^{o}C.

Table 4.3 Polynomial expression and constants supplied as a means of computing the unique temperature / emf relationship for the thermocouple specified. (CSIR Certificate Temp/203, 1989).

CALIBRATION OF A THERMOCOUPLE TYPE-S
SERIAL NO: WS- 02-81

In the equation:

$$T = b_0 + b_1 V + \ldots + b_n V^n$$

where: T = temperature (°C)
V = e.m.f. (mV)
n = 11,

The following values were obtained for the constants:

b_0 = -1,581042690E+00 K
b_1 = 1,832145563E+02 $K.V^{-1}$
b_2 = -6,092525911E+01 $K.V^{-2}$
b_3 = 3,736788567E+01 $K.V^{-3}$
b_4 = -1,713309397E+01 $K.V^{-4}$
b_5 = 5,493804984E+00 $K.V^{-5}$
b_6 = -1,207293879E+00 $K.V^{-6}$
b_7 = 1,797304664E-01 $K.V^{-7}$
b_8 = -1,776615706E-02 $K.V^{-8}$
b_9 = 1,115112124E-03 $K.V^{-9}$
b_{10} = -4,019728139E-05 $K.V^{-10}$
b_{11} = 6,334600650E-07 $K.V^{-11}$

Data transfer to the controller follows the routine by which the change setpoint instruction is succeeded by a setpoint enquiry instruction. A simple comparison of transmitted and received values confirms succesful transfer.

(c) **The data file**

Data from the thermocouple channels are continuously entering the process computer on a sequential basis. These data are matched against their respective thermocouple Type and fitted to the appropriate polynomial equation (table 4.3) that converts the millivolt readings directly to temperature. The temperature values are added to the thermocouple data file. The data file (table 4.4) stores the fifteen most recent logged temperature values of each channel. Data are stepped through successive rows of the file on a "first-in-first-out" basis. After each full log sequence (10 channels) the individual average for each column of the file is calculated and stored. This value equates to a 7.8 minute time average for the respective channel.

Table 4.4 Calibration data record. Record is printed after attainment of full stability conditions at the respective calibration temperature.

---------------- 500 ----------------

TEST No. 10263

30-Jul-90 21:47 TARGET TEMP: 500 SET TEMP: 497.8

CHANNEL	S/Nuaber	TEMP	CORRECTION
STD	WSBu13-85	500.0	
REF	SSY 01-90	500.1	-0.1
0	3442	501.7	-1.7
1	3445	502.9	-2.9
2	3447	502.8	-2.8
3	3452	505.7	-5.7
4	3455	505.2	-5.2
5	3457	505.0	-5.0

ROW: 10 STB: 0.23 CNT: 23 3Sx: 0.7: 0.3 CNTR: 0 DLY: 3 ALIGN: 0 REF:-0.1

	0	1	2	3	4	5	6	SP	STD	REF
0	501.70	502.90	502.80	505.70	505.20	505.00	0.00	0.00	500.00	500.10
1	501.52	502.89	502.63	505.40	505.26	505.00	0.00	0.00	500.07	500.07
2	501.41	502.53	502.61	505.49	505.05	504.79	0.00	0.00	499.97	499.97
3	501.64	502.92	502.86	505.78	505.26	505.02	0.00	0.00	499.97	500.07
4	501.78	503.15	502.79	505.71	505.28	505.12	0.00	0.00	500.07	500.07
5	501.60	502.84	502.72	505.68	505.19	504.95	0.00	0.00	500.07	499.97
6	501.76	502.79	502.96	505.82	505.35	505.12	0.00	0.00	500.07	500.07
7	501.92	503.19	502.91	505.82	505.40	505.24	0.00	0.00	500.17	500.18
8	501.76	502.82	502.75	505.68	505.28	505.09	0.00	0.00	500.07	500.07
9	501.64	502.68	502.44	505.52	504.95	504.81	0.00	0.00	499.87	499.97
10	501.57	502.84	502.56	505.56	505.09	504.93	0.00	0.00	499.77	499.97
11	501.88	503.38	502.96	505.82	505.42	505.17	0.00	0.00	500.17	500.18
12	501.64	502.72	502.65	505.68	505.24	505.00	0.00	0.00	500.07	500.18
13	501.57	502.68	502.68	505.61	505.17	504.88	0.00	0.00	499.97	500.07
14	501.55	502.75	502.84	505.68	505.17	504.95	0.00	0.00	500.07	500.07
15	501.76	503.03	502.93	505.75	505.38	505.05	0.00	0.00	499.97	500.18
16	0.40	0.70	0.50	0.40	0.40	0.40	0.00	0.00	0.30	0.30

NOTE:

Data file: Row (0) contains the average of rows (1) to (15).

Row (16) contains the 3Sx values of rows (1) to (15).

Column (STD) contains the Standard T/C data, the average of this column is, at the recorded point of calibration equal to the calibration temperature (500.0 ^{o}C).

The row of data between the thermocouple identification data and the data average file, contains information relating to the status of the stability controls at the time of logging.

2. Stage 2 (Figure 4.6)

(a) **Bath stability count**

Following each successive log sequence the last instantaneous value of the standard thermocouple is subtracted from the data file average for the standard channel. If the difference calculated above is within the stability limit, the stability counter is incremented and the process repeated until there has been a stability between the standard average and instantaneous readings for a set number of counts. When the counter contains the required count the program proceeds to the next parameter check. Table 4.5 provides a listing of all adjustable program limits. In the example shown a stability limit of 0.4^{o}C is specified for a count of 6, these settings provide a stability period of 4.7 minutes. The stability limit and count routine ensures that the bath temperature is "physically" stable within the set parameters.

Table 4.5 FB08.BAS Algorithm limit settings (program parameters). The particular parameter values shown in this version are not necessarily the same as given in the text. Refer to text for explanation of the function of the parameter limits.

```
10   !////\\\\////\\\\////\\\\////\\\\////\\\\////\\\\////\\\\/
20   !
30   !
40   !  PROGRAM NAME : FB08.BAS        Version: 7
50   !  AUTHOR       : Rod Hart
60   !  DATE         : JULY 1990
70   !  COMPANY      : HULETT ALUMINIUM - PIETERMARITZBURG.
80   !  REVISIONS    : * Updated version of FB.BAS containing data storage
90   !                   Present stage: Channel 0 & STD stores both inst and
100  !                   ave. Channel 0 stores correction factor.
110  !                 * Second stability using standard deviation
120  !  PROGRAM PARAMETERS: AVERAGE FILE: 15 X 2sec X 10 ch : 7.8min ave.
130  !                      STABILITY    : 0.4 Deg.C  (Inst-Ave)
140  !                 *    3Sx          : 0.4 (higest): 0.4 (Std.)
150  !                      COUNTER      : 6          (Stable logs : 4.7 min)
160  !                      CONTROL      : 0.3 Deg.C  (SP-PV)
170  !                      ALIGNMENT    : 0          (RV-Ave)
180  !                      DELAY        : 6          (After resetting control)
190  !                      REFERENCE    : 0.5 Deg.C  (Std-Ref)
200  !
210  !\\\\////\\\\////\\\\////\\\\////\\\\////\\\\////\\\\////\
220  !
```

(b) **Standard deviation**

The previous check does not test the complete data file, for this the standard deviation of all fifteen values for each data channel is calculated. Because of the differing levels of importance the standard thermocouple is evaluated separately from the thermocouples under test. In the example provided in table 4.5 the standard thermocouples data set is required to meet a three standard deviation (99.7%) criteria of 0.4^{o}C. A far more lenient 3^{o}C on test thermocouples prevents the program from locking up in the case of minor instability in one of the test thermocouples.

(c) **Control stability**

Whilst the criteria above tests the physical stability of the bath and integrity of the data file it does not necessarily follow that the control is stable, for this the control thermocouple actual value (process variable, PV) is subtracted from the setpoint value (SP) in order to determine if the bath has reached the control point. Experience has shown this point to be highly active especially in the higher temperature range. The thyristor control and advanced control features of the furnace controller have, however, enabled this criteria to be set as low as 0.3^{o}C.

3. **Stage 3 (Figure 4.7)**

(a) **Required value alignment**

Although the furnace controller receives a calibration corrected setpoint value there may, after stabilisation, be an offset remaining between the actual bath temperature as determined by the standard thermocouple and the control point as determined by the control thermocouple. A discrepancy in the order of points of a degree is normal and may be caused by drift in the control thermocouple calibration or by the difference in immersion depth or other disturbances within the bath caused by the positioning of the thermocouples under test. Because realignment of the control setpoint necessitates disturbing the bath, when this is required, the stability counter is reset to force the stability period to be reassessed. The control setpoint to the furnace controller is incremented or decremented by 0.1^{o}C in the direction required to align the temperatures. The stability counter now performs a second function in determining the minimum delay between possible changes to the setpoint thus avoiding the possibility of "windup".

(b) **Reference check**

The fifth and final of the parameter checks confirms the integrity of the standard thermocouple by comparison to the reference thermocouple. This comparison is carried out by subtracting the data file average for the reference thermocouple from the equivalent average for the standard. If the result of this check exceeds the set limit the check is again repeated after a further cycle of stability checks. If the discrepancy remains the program is permitted to continue but an error warning flag is set which causes a warning to be printed out with the calibration data.

4. **Stage 4 (Figure 4.8)**

(a) **Correction table update**

Subtracting the required value from the final setpoint yields a revised correction factor for the furnace control thermocouple, for that specific temperature. This correction factor is now filed in the correction table file, "TABLE.VRT", replacing the original value and thereby ensuring that the table is continuously updated. Tables 4.1 and 4.2 provide examples of the difference between original and revised correction tables.

(b) Calibration data report

With the completion of the fifth parameter check at the end of stage 3 the calibration procedure is complete. At this stage the average of the standard data set held in the data file is equal to the required value to within one decimal place. The calibration data report (table 4.4) is now generated with the differences between the standard and the individual thermocouples under test being expressed in terms of correction values. In addition to the thermocouple identification and calibration details the entire data file and parameter statuses are printed out.

(c) Next calibration point

Where there are further calibration points required the program loops back to the start. After the final calibration the furnace controller setpoint is set to 50^{o}C and the shutdown sequence instituted.

(d) **Shutdown sequence**

The shutdown sequence closes off all files prints "End of program" message and loads the following program.

d. **PLOT.BAS**

PLOT.BAS is a menu driven data management program which permits access to and management of either the current test data or previously stored data. The main elements of this program are briefly indicated below:

1. **Display Directory**

   Displays the directory of test numbers on the current disc.

2. **Plot Deviation Curve**

   This option directs the user through the options available in plotting a deviation (calibration) certificate (figure 4.9). Thermocouple identification, Type, customer, certificate number, calibration date in addition to the data used in the plot is accessed from the stored file.

3. **Calibration Report Summary**

   The calibration report summary lists the entire contents of the selected test file (table 4.6).

4. **Copy Data File**

The copy data file sequence guides the user through the necessary steps in copying a test file onto a second storage disc.

5. **New Test Number**

Provides the facility to individually access the available test files.

e. **TABLE.BAS**

Identified with the previously introduced "TABLE.VRT" this program, which is not directly linked to LAB87, enables "TABLE.VRT" to be accessed, printed (table 4.2), and if necessary adjusted by having a new set of values fitted.

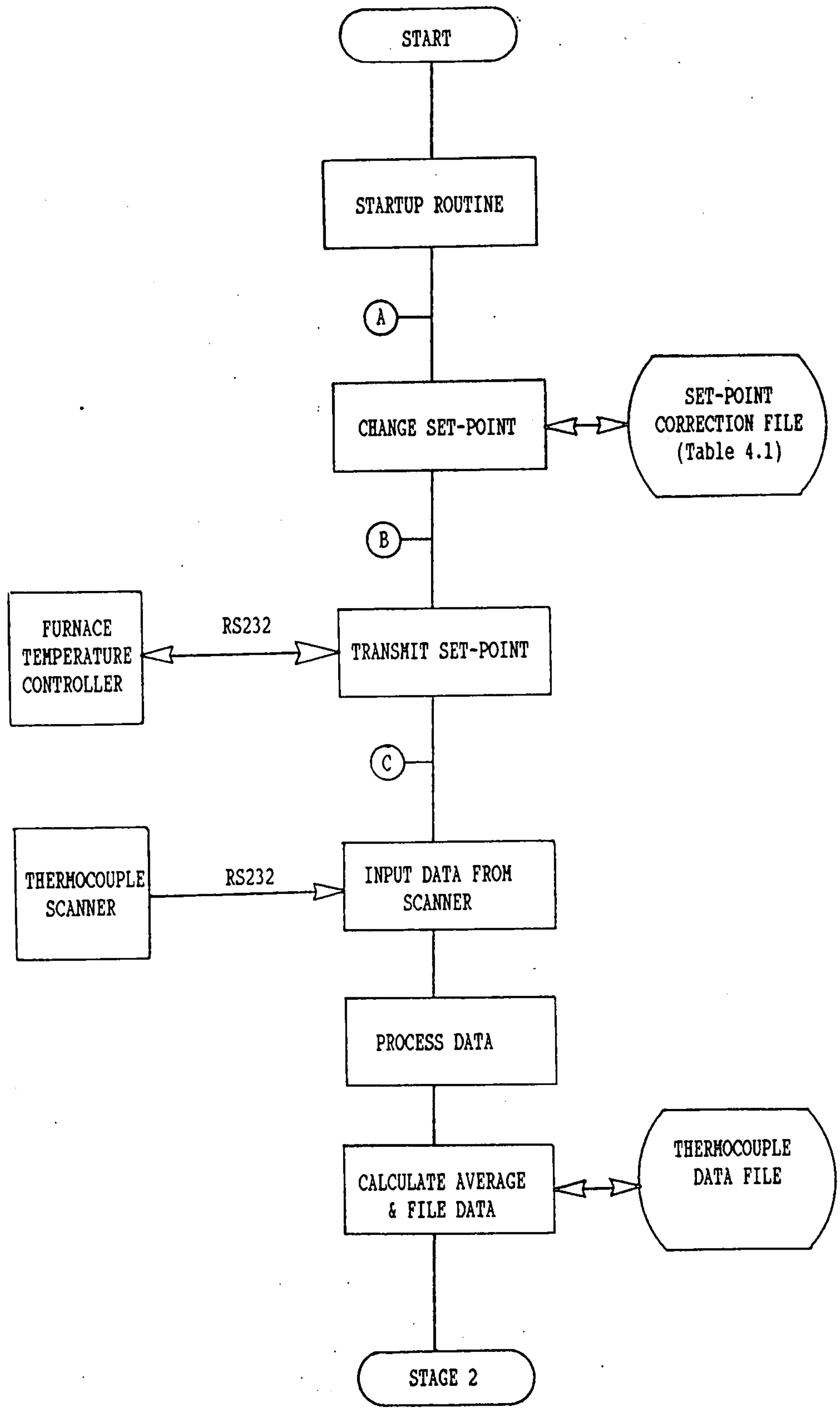
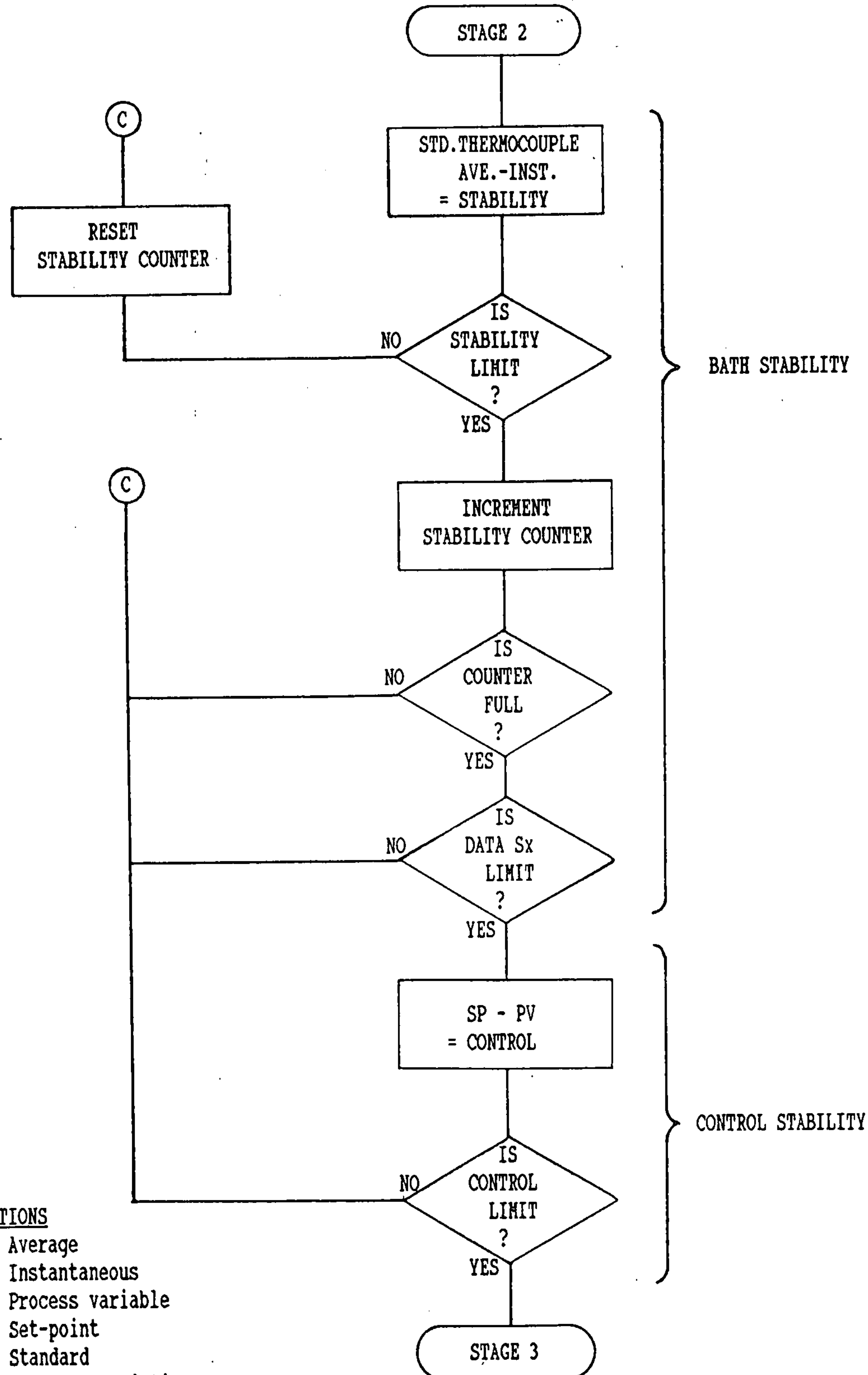


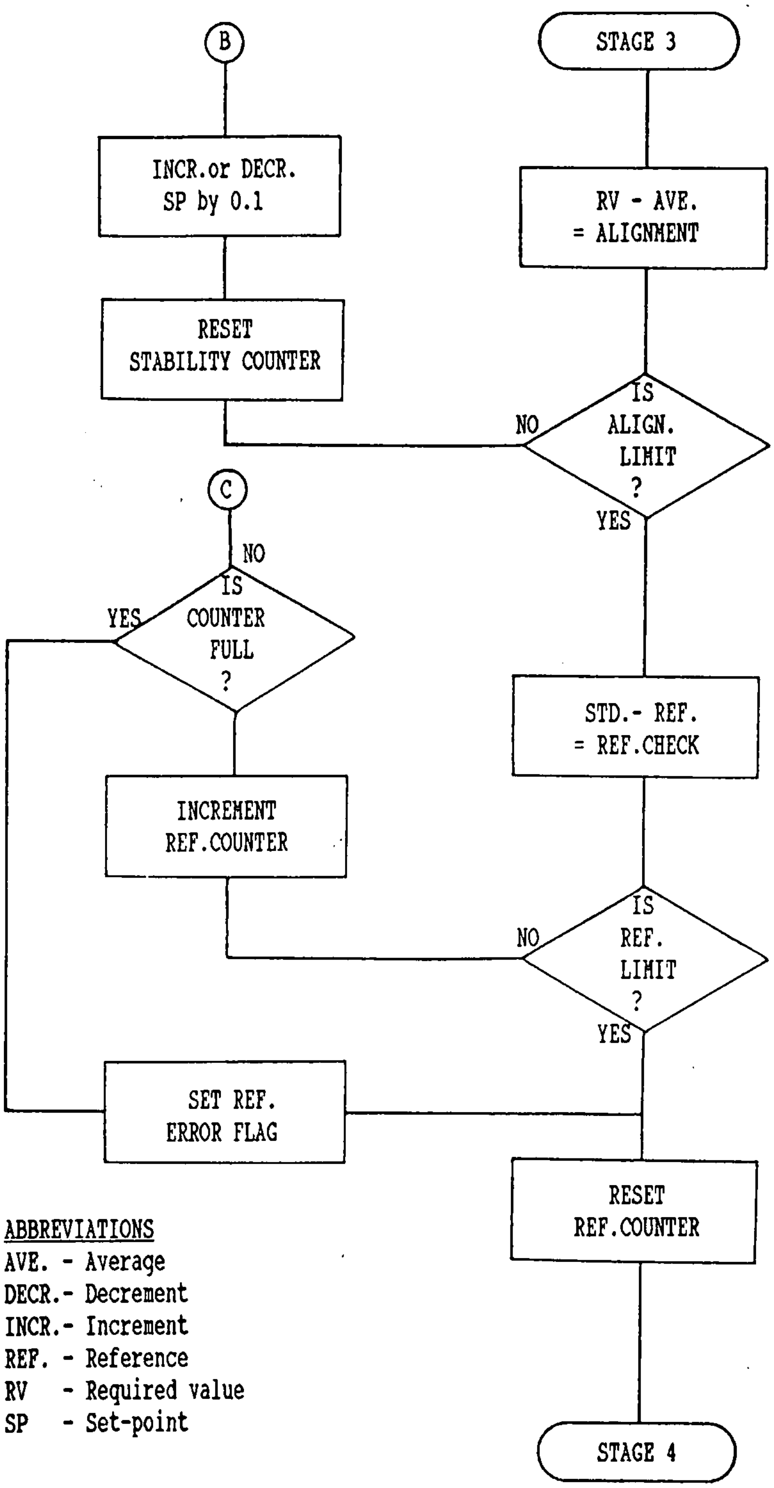
Figure 4.5 LAB87 Program software schematic.
Stage 1: Setpoint correction and data averaging.



ABBREVATIONS

AVE. - Average
INST.- Instantaneous
PV - Process variable
SP - Set-point
STD. - Standard
Sx - Standard deviation

Figure 4.6 LAB87 Program software schematic.
Stage 2: Bath stability and control stability.



ABBREVIATIONS
AVE. - Average
DECR.- Decrement
INCR.- Increment
REF. - Reference
RV - Required value
SP - Set-point

Figure 4.7 LAB87 Program software schematic.
Stage 3: Required temperature alignment and reference check.

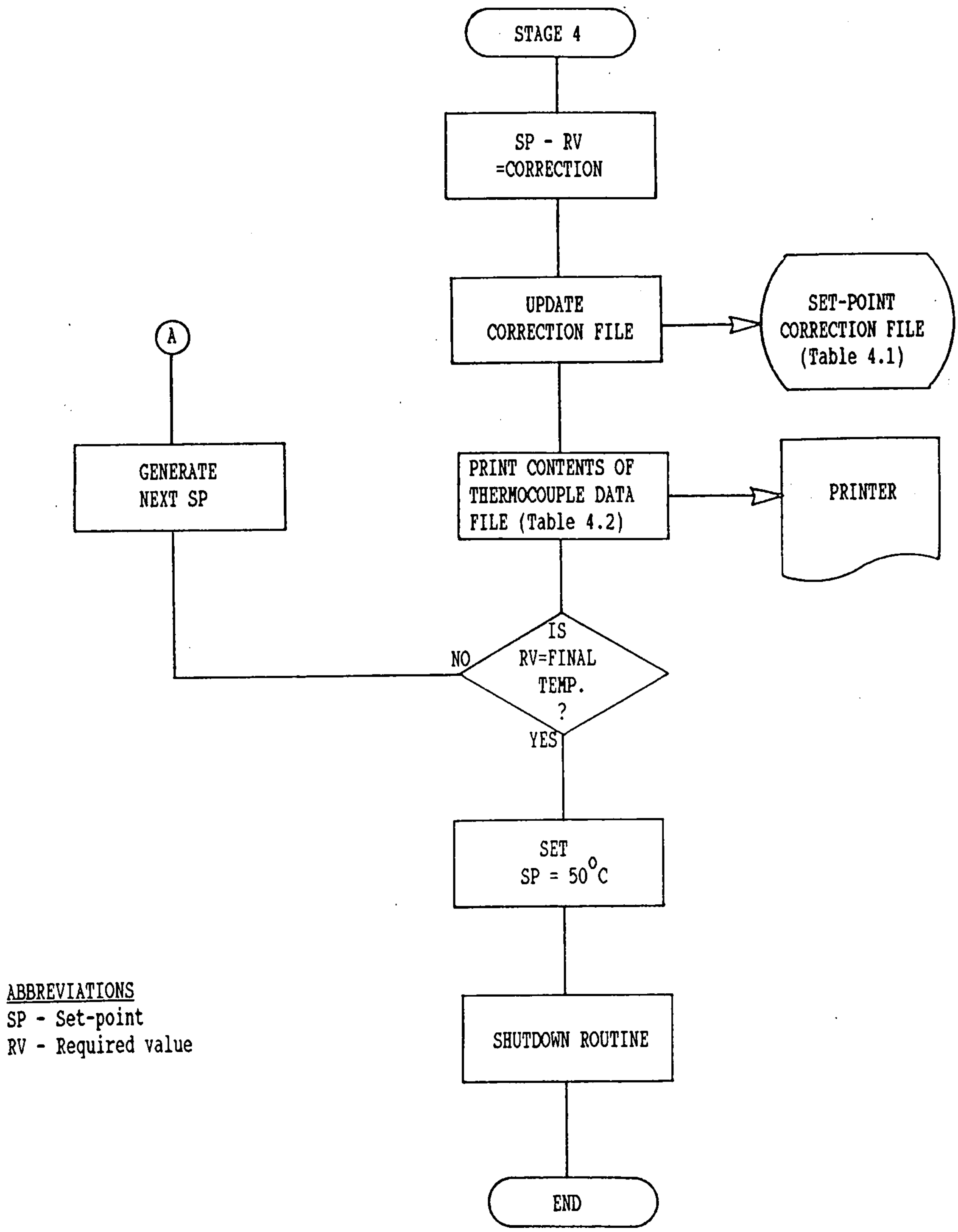


Figure 4.8 LAB87 Program software schematic.
Stage 4: Correction file update, report printout and shutdown routine.

Table 4.6 Calibration report summary. Columns reflect deviation from Standard thermocouple. Column showing calibration temperatures is actual Standard record. The column on the extreme right reflects the difference between the Standard and Reference thermocouples.

CALIBRATION REPORT SUMMARY

TEST No: 10263

3442	K	3844030790	K/3/[AF7X6]/LA	TEMP.CONTROLS
3445	K	3844030790	K/3/[AF7X6]/LA	TEMP.CONTROLS
3447	K	3844030790	K/3/[AF7X6]/LA	TEMP.CONTROLS
3452	K	3844040790	K/3/[AF7X6]/LA	TEMP.CONTROLS
3455	K	3844040790	K/3/[AF7X6]/LA	TEMP.CONTROLS
3457	K	3844040790	K/3/[AF7X6]/LA	TEMP.CONTROLS
0000	0000	0000	0000	0000
0000	0000	0000	0000	0000
STD.	0000	0000	0000	0000
REF.	0000	0000	0000	0000
11	50	550	50	30-Jul-90

DEVIATION FROM STANDARD [+/- Deg.C]:

3442	3445	3447	3452	3455	3457	0000	0000		
0.4	0.4	0.4	0.5	0.5	0.5			50.0	-0.2
0.7	0.9	1.0	1.3	1.3	1.2			100.0	-0.2
1.4	1.8	2.0	2.4	2.3	2.3			150.0	-0.1
1.2	1.8	2.0	2.6	2.6	2.6			200.0	-0.1
1.1	2.0	2.2	3.1	3.1	3.0			250.0	0.1
1.1	2.1	2.2	3.6	3.5	3.4			300.0	0.2
1.0	2.0	2.2	4.0	3.9	3.7			350.0	0.1
0.9	2.0	2.3	4.3	4.2	3.9			400.0	0.0
1.2	2.4	2.5	4.9	4.6	4.4			450.0	0.0
1.7	2.9	2.8	5.7	5.2	5.0			500.0	0.1
1.7	2.8	2.7	5.9	5.5	5.2			550.0	0.1

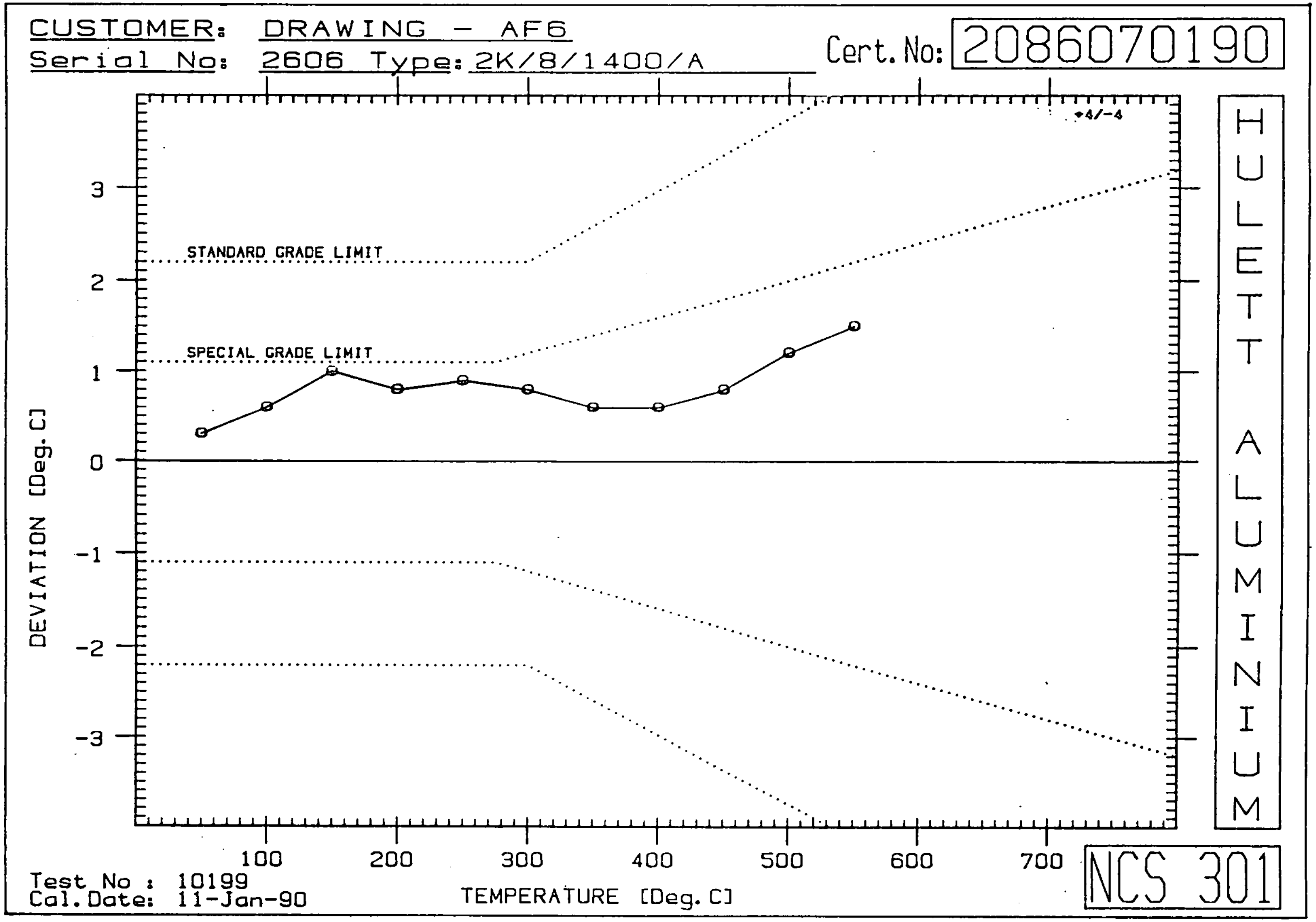


Figure 4.9 Example of calibration certificate. Standard and special grade limits, shown dotted, refer to manufacturer specification. Indices,(o), locate the calibration datum points.

4.4 CALIBRATION PROCEDURE

Due to the automated calibration technique, calibration procedure is greatly simplified. Each step of the full procedure is indicated below:

(i) The level of powder in the fluidised bath is checked. With the the bed collapsed the powder level is to be 100 mm, (+/-)5 mm, below the lower face of the thermocouple entry cover plate.

(ii) With electrical power and supply air pressure on, the exhaust and fluidising air controls are set. The temperature controller is set to a temperature value not greater than the first calibration temperature required and shall, for safety, in no event be set higher than 100°C during setting up procedures. Prior to the loading of the test thermocouples the furnace temperature shall not exceed the control setpoint by more than 10°C.

(iii) The ice reference bath is prepared and the relevant reference junctions fitted and connection made to the scanner.

(iv) The required depth of immersion is marked on the test thermocouples. The marking of the depth of immersion is relative to the thermocouple entry cover plate. Thermocouples are immersed and clamped in position. The cover plate provides entry holes suitable for the adequate separation of the thermocouples (figure 4.10).

(v) Thermocouple connections are made to the respective reference junctions.

(vi) Program LAB87 is loaded on the process computer and the required detail entered as prompted. After the thermocouple connection program has confirmed that all signals are good the program may be left unattended.

(vii) On completion of the calibration program the PLOT.BAS management menu is displayed and may be utilised to provide a full calibration report (table 4.6) and individual or combination calibration curve plots (figure 4.9).

(viii) It is desirable, for safety, to leave the thermocouples in the fluidised bath until the temperature of the bath has reached such a point as to enable the safe handling of the thermocouples. Under no circumstances are the standard and reference thermocouples to be disturbed whilst the temperature of the bath exceeds 100°C.

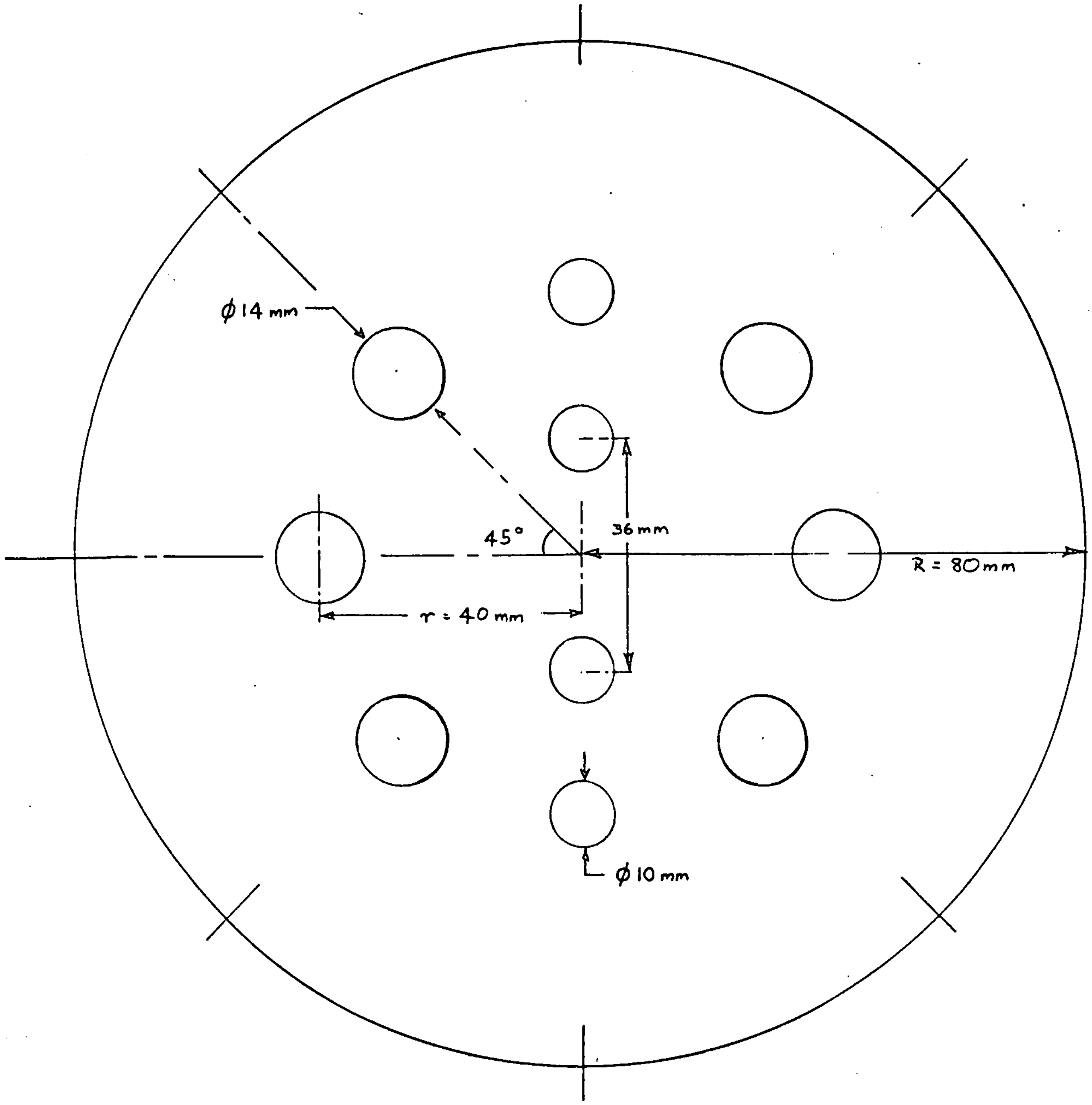


Figure 4.10 Fluidised bath cover plate. Plan view, actual size, showing detail of thermocouple entry ports.

4.4.1 Immersion Depth

One aspect of procedure relating specifically to calibration in a fluidised bath furnace that can lead to misinterpretation of results, and that needs to be fully understood, is that of immersion depth.

This aspect relates to the central problem of this project, that of instability of the Type K thermocouple during the initial period of use. Except under specific circumstances where conditions of use are well defined it is common practice for temperature laboratories to either advise against the recalibration of base metal thermocouples or to issue wide uncertainties, in the order of (+/-)3^{o}C. ASTM Standard E220-86, which prescribes the calibration method employed in this project, states that base metal thermocouples should be calibrated in the "as-received" condition and that the changes they undergo with use at high temperature render them unfit for recalibration (1990 Annual Book of ASTM Standards, Volume 14.03,1990,E 220-86, par.10.3). It is therefore of paramount importance to understand the reasoning behind the precautions taken during this project in determining the immersion depth in order to accept the admissibility of these calibration data at the uncertainty levels claimed.

Cognisance was taken of the effect that the calibrating temperatures have on the Type K thermocouple, and in taking precautionary steps to avoid these changes affecting the calibration results, it was found possible to utilise this effect positively. The following points itemise the precautions and findings concerning this aspect of the procedure:

a. **Initial Calibration**

Initial calibration data quoted is always given for calibration carried out on first calibration on rising temperatures only, on thermocouples in the "as supplied" condition.

b. **Short Term Aging Effects**

By identifying the immersion depth in relation to a reference point on the bath it is possible to repeat the calibration at either of two points. By raising the thermocouple 20 mm, a second calibration run produces calibration results consistent with that of a thermocouple after a short work cycle at what was the highest calibrating temperature. This calibration can be utilised in predicting the effect of short term aging on that specific material batch.

c. **Confirmation of Initial Calibration**

By lowering the thermocouple 60 mm deeper than the initial immersion depth it is possible to reproduce, within the claimed tolerance limits, the initial calibration results. The reason for this is that the fluidised bath has excellent vertical temperature uniformity. The aged section of the thermocouple, together with the interface with unaged material, is completely immersed, due to the greater immersion depth, at what is a relatively uniform temperature whilst the section found in the area of the thermal gradient, where the emf is generated, is unaged material.

d. **Successive Calibration Runs**

On trials where a number of calibration runs are required, the effective immersion depth is always lowered to ensure that the cumulative aging effect is obtained.

e. **Level of Fluidised Medium**

A further aging effect that interferes with calibration and that is unique to the live bed calibration technique is that caused by the change of level of the fluidised bed. As the temperature of the bath rises, air flow to the bath is decreased proportionally in order to maintain the same level of fluidisation. Whilst this is largely successful, the increased fluidity of the bath at each successive temperature does result in a raising of the level of the powder, hence the necessity in (c) above to lower the thermocouple 60 mm to be certain of obtaining unaged material; whereas it is only considered necessary to raise the thermocouple 20 mm to be certain of aged material. This positive change of level does not present a problem. The problem is caused by the increased amount of powder lost from the bath to the dust extraction system due to the raised level and increased (apparent) volatility. The maximum level of the bath at the temperature concerned is achieved at the moment that the bath reaches that temperature. It may however be some time before calibration stability is reached. During this time the level of the bath may lower, due to loss of powder to the extraction system, causing a section of aged material to be exposed to the area of thermal gradient. For Type K thermocouples up to 550°C this would result in an increased positive calibration deviation error. Above 600°C the same condition may produce a negative effect on the natural error. Throughout this project calibration stabilisation times are recorded and data comparisons made in an effort to detect any inconsistancies that may have influenced specific data sets.

f. Overfluidisation

A similar effect to that in (e) above is caused by overfluidisation of the bath. In this case the overactive medium causes a variable area of thermal gradient that is continuously rising and falling. Here the thermocouples are likely to be affected individually according to their position within the bath. The resultant effect may be poor correlation between calibration results across a batch of like material. Whilst the automatic fluidising air control system adequately ensures against excessive fluidisation it remains possible, under the relatively active normal fluidising condition, for this effect to disturb the natural calibration. The effect under controlled fluidisation is considered moderate and is additionally limited by the time period averaging technique used. Up to 550^{o}C, the highest calibration temperature considered under the specified uncertainty limits pertaining to this project, any deviation in calibration caused by spurious instability in the level of the fluidised medium would be in the same direction as the thermocouples' natural drift at the same temperature. This deviation is thus not considered to violate data integrity but remains a valid aspect of calibration that has been included in the overall estimation of uncertainty.

Where it is considered that the effects identified in either (e) or (f) above have significantly influenced data, due consideration is given to explanation in the accompanying text.

g. **Comparison with "Dead Bed" Technique**

It should be noted that the effects detailed in (e) and (f) above would not be found in the "dead bed" technique since the collapsed bed ensures that the area of thermal gradient is consistently located in a uniformly heat treated area of the thermocouple.

Although these results may prove more uniform than equivalent "live bed" results they cannot be considered as representative of initial calibration. The "dead bed" technique, through this consideration alone, proves unsuitable for the specific calibration requirements of this project.

The above considerations do not detract from the integrity of the calibration data produced but rather accentuate the fact that any calibration measurement is made under a specific set of conditions and is valid only at the time of measurement. A thorough comprehension of the effects influencing calibration is required in order, through analysis, to extend the validity of the data within the bounds of an established uncertainty limit. It is thus established that the repeatability of the results claimed is inseparably linked to the calibration method and procedures employed.

4.5 ESTABLISHING CALIBRATION UNCERTAINTY

In temperature metrology traditional uncertainty budgets have limited application. Specifically in comparative calibration the factors affecting transfer are ill defined and whilst best measurement capability (bmc) may be delimited by equipment specification, effective uncertainty is ultimately determined by metrologist and technique. The most appropriate manner of establishing uncertainty is by audit sample providing traceability to a higher authority.

The uncertainties claimed in this project (chapter 3, par.3.2.3), unless otherwise specified, are:

(i) Precision : (+/-)0.5^{o}C

(ii) Traceability: (+/-)1.0^{o}C

Several approaches, indicated briefly below and discussed in detail in the following subsections, are provided to support these uncertainty claims.

Commencing with method the primary consideration is to evaluate control technique in order to assess the degree of control maintained over the standard and test thermocouple temperatures. This assessment is carried out through an evaluation of the standard and test thermocouple temperature response trends, specifically over the stabilising period prior to and during the filling of the thermocouple data average file (containing the fifteen values used to

Table 4.7 Temporary data storage array.

02-Oct-89
ARRAY NAME: std1.vrt LENGTH: 1000

Position	Time	Std.Ave.	Std.	CH.0 Ave.	CH.0
0	0.00	0.00	0.00	0.00	0.00
1	6.50	62.70	85.32	56.00	87.83
2	7.00	65.90	87.58	60.70	91.82
3	7.50	69.10	89.40	65.40	93.49
4	8.00	72.10	91.07	69.90	94.94
5	8.50	75.00	92.61	74.30	96.16
6	9.00	77.80	93.99	78.50	97.32
7	9.50	80.50	96.20	82.40	98.15
8	10.00	83.00	97.03	85.70	99.58
9	10.50	85.40	97.86	88.10	100.16
10	11.00	87.50	98.41	90.30	100.60
11	11.50	89.40	99.09	92.30	100.99
12	12.10	91.20	99.91	94.00	101.33
13	12.60	92.80	100.32	95.50	101.74
14	13.10	94.10	100.60	96.80	101.91
15	13.60	95.30	100.73	97.90	102.08
16	14.10	96.40	100.87	98.80	102.15
17	14.60	97.30	101.01	99.50	102.23
18	15.20	98.00	101.01	100.10	102.20
19	15.70	98.70	100.87	100.60	102.20
20	16.20	99.30	100.87	101.00	102.15
21	16.70	99.70	100.73	101.30	102.11
22	17.20	100.00	100.60	101.60	102.03
23	17.70	100.20	100.46	101.70	101.86
24	18.30	100.40	100.46	101.80	101.77
25	18.80	100.50	100.32	101.90	101.67
26	19.30	100.60	100.32	101.90	101.62
27	19.80	100.60	100.32	101.90	101.52
28	20.30	100.60	100.46	101.90	101.43
29	20.80	100.60	100.32	101.90	101.40
30	21.40	100.60	100.46	101.80	101.38
31	21.90	100.60	100.46	101.80	101.38
32	22.40	100.50	100.32	101.70	101.38
33	22.90	100.50	100.32	101.70	101.40
34	23.40	100.40	100.32	101.60	101.40
35	23.90	100.40	100.32	101.60	101.40
36	24.40	100.40	100.19	101.50	101.38
37	25.00	100.30	100.05	101.50	101.35
38	25.50	100.30	99.91	101.50	101.28
39	26.00	100.30	99.91	101.40	101.23
40	26.50	100.20	99.78	101.40	101.18
41	27.00	100.20	99.78	101.40	101.18
42	27.50	100.20	99.91	101.30	101.16
43	28.10	100.10	99.91	101.30	101.13
44	28.60	100.10	99.91	101.30	101.11
45	29.10	100.10	100.05	101.30	101.06
46	29.60	100.00	100.05	101.20	101.08
47	30.10	100.00	100.32	101.20	101.13
48	30.60	100.00	100.19	101.20	101.23
49	31.20	100.00	100.05	101.20	101.23
50	31.70	100.00	100.19	101.20	101.23

compute stability and final calibration values). The control under which calibration temperatures are approached provides an indication of the degree of precision that may be expected of the system.

Following the above assessment are a number of repetitive comparison trials providing positive indication of precision and repeatability. Finally the result of a CSIR assessment audit is provided as an indication of the calibration systems' capability of providing traceability within the specified uncertainty.

4.5.1 Evaluating the Automated Live Bed Technique

a. Instantaneous/Average Data Comparison

Program FB08.BAS creates a temporary array that captures selected processed data enabling the evaluation of a completed calibration run. Due to the volume of disc memory utilised in storing this data, the file is overwritten by the succeeding program run. The following columns of data for each log cycle are captured throughout the entire calibration program:

(i) **Row number (Position):** Indicates the sequential position of the log cycle. Row capture and numbering starts once the data average file is full.

(ii) **Time:** Indicates minutes since the initiation of the calibration run.

(iii) **Standard Average:** Indicates the running average of the standard thermocouple channel as calculated from the data average file. It is this value that is used by the program to ascertain the standard temperature.

(iv) **Standard (Instantaneous value):** Indicates the current logged value of the standard thermocouple. This value represents the most recent entry into the data average file for that specific log cycle.

(v) **Channel 0 Average:** Indicates the running average of the test thermocouple connected through channel 0.

(vi) **Channel 0 (Instantaneous):** Indicates the current logged value of channel 0.

Table 4.7 provides an example of the construction of the above data array. In this example the calibration target temperature is 100^oC. At row 22, after 17.2 minutes, the running average of the standard thermocouple achieved the required target of 100.0^oC. At this time the standard deviation requirement for the instantaneous data is not met. Three times the standard deviation (3Sx) for the data concerned, the fifteen values in column "Std." (the instantaneous values) between rows 8 and 22, is equal to 3.8^oC. LAB87 program standard deviation criteria permits a maximum 3Sx of 0.4^oC. In row 22 the calibration value of the thermocouple under test is 101.6^oC. In row 46 after 29.6 minutes the standard again achieves the required target, an analysis of the respective instantaneous data from rows 32 to 46 reveals three times the standard deviation to be 0.6^oC. By row 50 the 3Sx value decreased to 0.4^oC. From row 46 through 50 the calibration value indicated for the thermocouple under test is 101.2°C. Full stability requirements for this example were met after 41.4 minutes on row 69 (not shown in example) providing a calibration result of 101.1°C for the thermocouple under test. In this example, taken from an actual test (test number 10187, dated 89.10.02), both standard and test thermocouples were stable within 0.2°C of their final temperature for a period of 14 minutes prior to acceptance of the test thermocouple's calibration value. The above data is summerised in the following table:

Row	Minutes	Std.Ave.	3Sx	Test Ave.
22	17	100.0	3.84	101.6
46	30	100.0	0.60	101.2
50	32	100.0	0.48	101.2
69	41	100.0	0.40	101.1

Evaluation of the instantaneous/average data is simplified by applying the data to a temperature versus time plot. In this plot the thermocouple response curves provide clear indication of control and stability levels. Figures 4.11 through 4.14 present such an application of the data captured during an early test run of the program (dated 89.02.06). In these figures the graphs feature that portion of the temperature curve falling within (+/-)0.5°C of the respective final temperatures. For conformity the X-axis, in each case, represents a period of 30 minutes with indicated values representing time, in minutes, from start of the calibration run. Due to the nature of the variables involved and the degree of detail expressed by the 0.1^{o}C temperature and 1 minute interval resolution, no single response may be considered typical of a calibration range or point. The calibration plots selected for evaluation, with 100^{o}C, 200^{o}C, 300^{o}C and 700^{o}C calibration targets, do provide a range of responses that are considered representative of actual performance.

In each of the figures 4.11 through 4.14, markers on the instantaneous temperature trends indicate the values comprising the data average file at the point of calibration. In figure 4.11 the temperature response curves commence on the sixth minute once the data average file has accumulated fifteen values. For the sake of clarity of presentation, text in the following subsections, relating to the figures 4.11 through 4.14, is in tabular rather than discursive format.

1. **Data Comparison at 100°C Target (Figure 4.11).**

Refering to figure 4.11:

(a) **Standard thermocouple instantaneous trend.**

Lowest value in data average file : 99.7°C

Highest value in data average file: 100.1°C

Average at point of calibration : 100.0°C

Confidence interval at 99.7% (3Sx):+/-0.4°C

(b) **Standard thermocouple average trend.**

Average is within (+/-)0.2°C of 100.0°C for a period of 10 minutes prior to calibration.

Average lags instantaneous by 2 minutes.

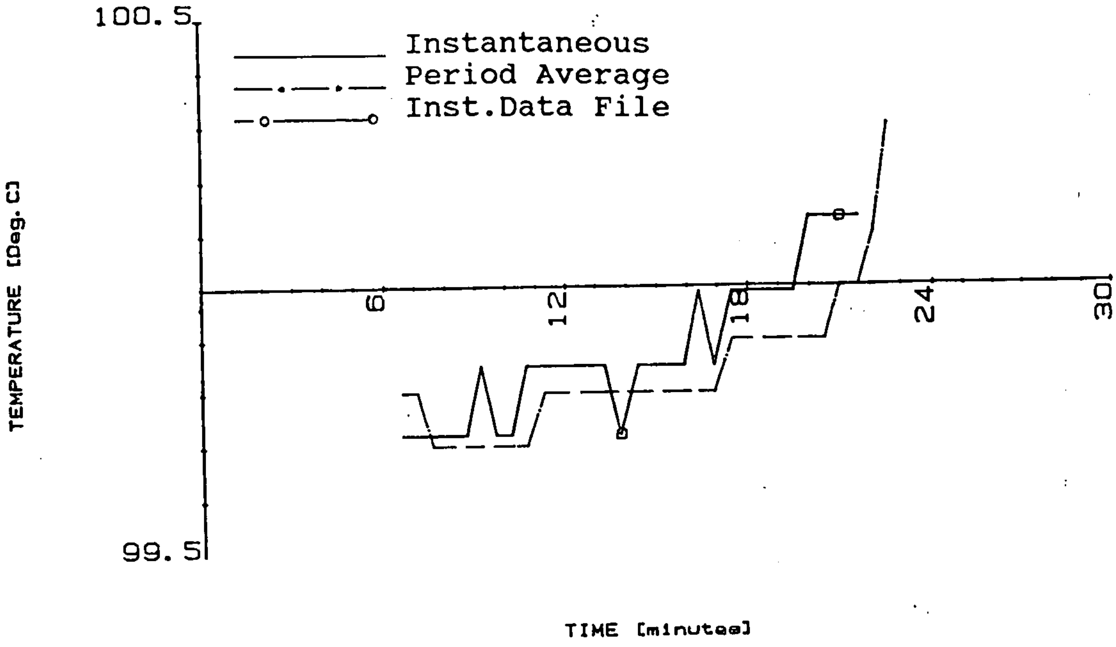
(c) **Test thermocouple instantaneous trend**

Lowest value in data average file : 100.8°C

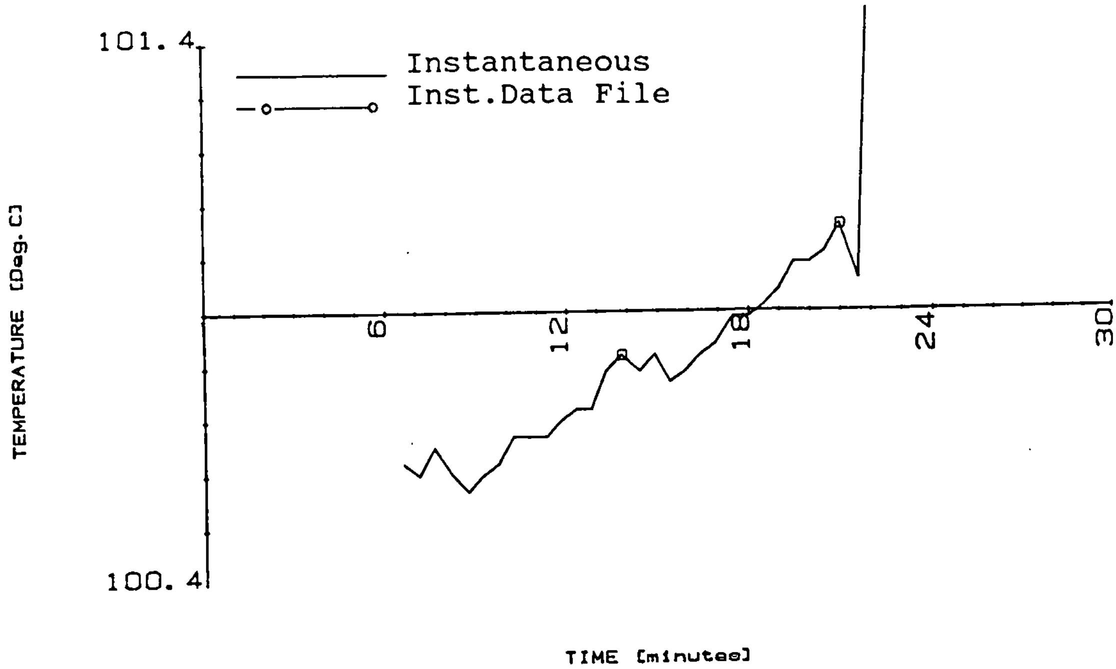
Highest value in data average file: 101.1°C

Average at point of calibration : 100.9°C

Confidence interval at 99.7% (3Sx):+/-0.3°C



(a) STANDARD THERMOCOUPLE TEMPERATURE



(b) TEST THERMOCOUPLE TEMPERATURE

Figure 4.11 Thermocouple response curves: 100°C. Time indicates minutes expired since start of calibration run. Standard T/C X-axis intersects at 100.0°C. Test T/C axis intersects at 100.9°C.

2. **Data Comparison at 200°C Target (Figure 4.12).**

Refering to figure 4.12:

(a) **Standard thermocouple instantaneous trend.**

Lowest value in data average file : 199.8°C

Highest value in data average file: 200.3°C

Average at point of calibration : 200.0°C

Confidence interval at 99.7% (3Sx):+/-0.4°C

(b) **Standard thermocouple average trend.**

Average is within (+/-)0.2°C of 200.0°C for a period of 4 minutes prior to calibration. Average lags instantaneous by 3 minutes.

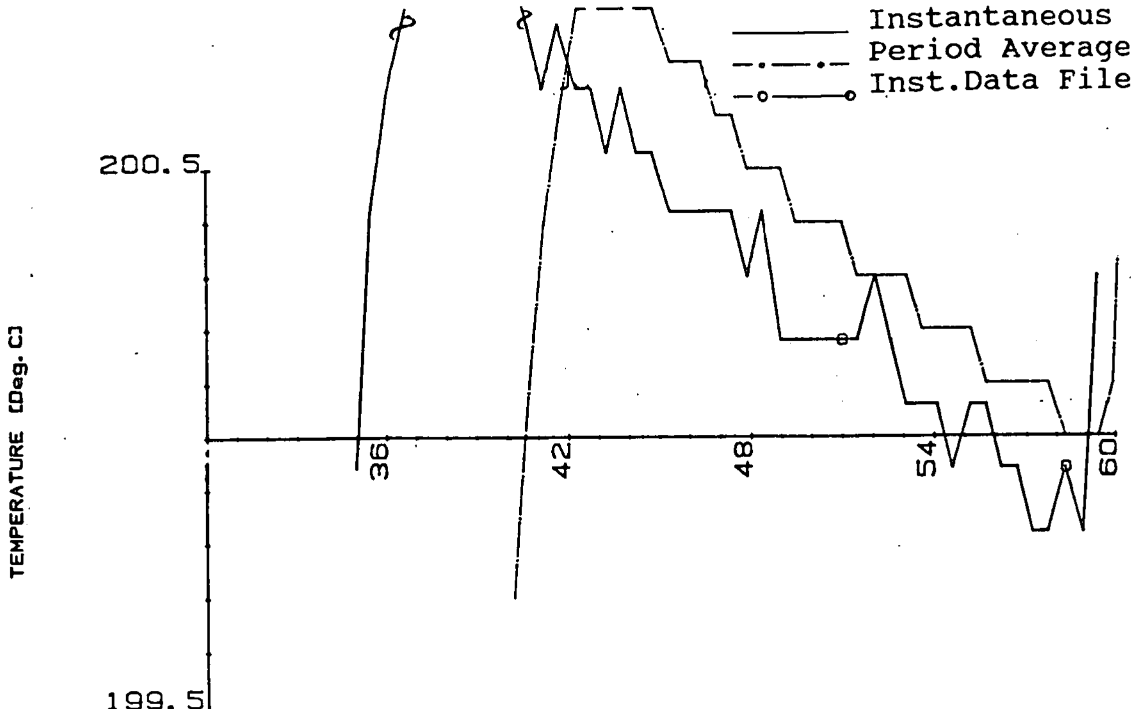
(c) **Test thermocouple instantaneous trend**

Lowest value in data average file : 201.9°C

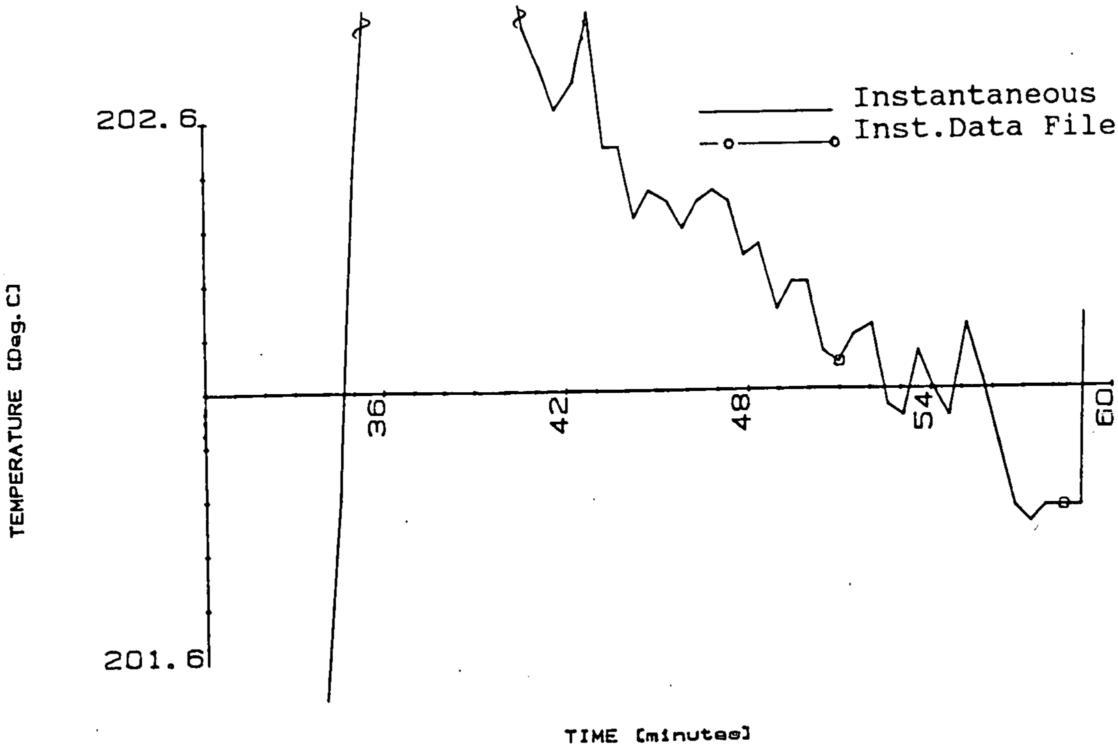
Highest value in data average file: 202.2°C

Average at point of calibration : 202.1°C

Confidence interval at 99.7% (3Sx):+/-0.4°C



(a) STANDARD THERMOCOUPLE TEMPERATURE



(b) TEST THERMOCOUPLE TEMPERATURE

Figure 4.12 Thermocouple response curves: 200°C. Time indicates minutes expired since start of calibration run. Standard T/C X-axis intersects at 200.0°C. Test T/C axis intersects at 202.1°C.

3. **Data Comparison at 300^{o}C Target (Figure 4.13).**

Refering to figure 4.13:

(a) **Standard thermocouple instantaneous trend.**

Lowest value in data average file : 299.8^{o}C

Highest value in data average file: 300.3^{o}C

Average at point of calibration : 300.0^{o}C

Confidence interval at 99.7% (3Sx):+/-0.4^{o}C

(b) **Standard thermocouple average trend.**

Average is within (+/-)0.2^{o}C of 300.0^{o}C for a period of 6 minutes prior to calibration. Average lags instantaneous by 5 minutes.

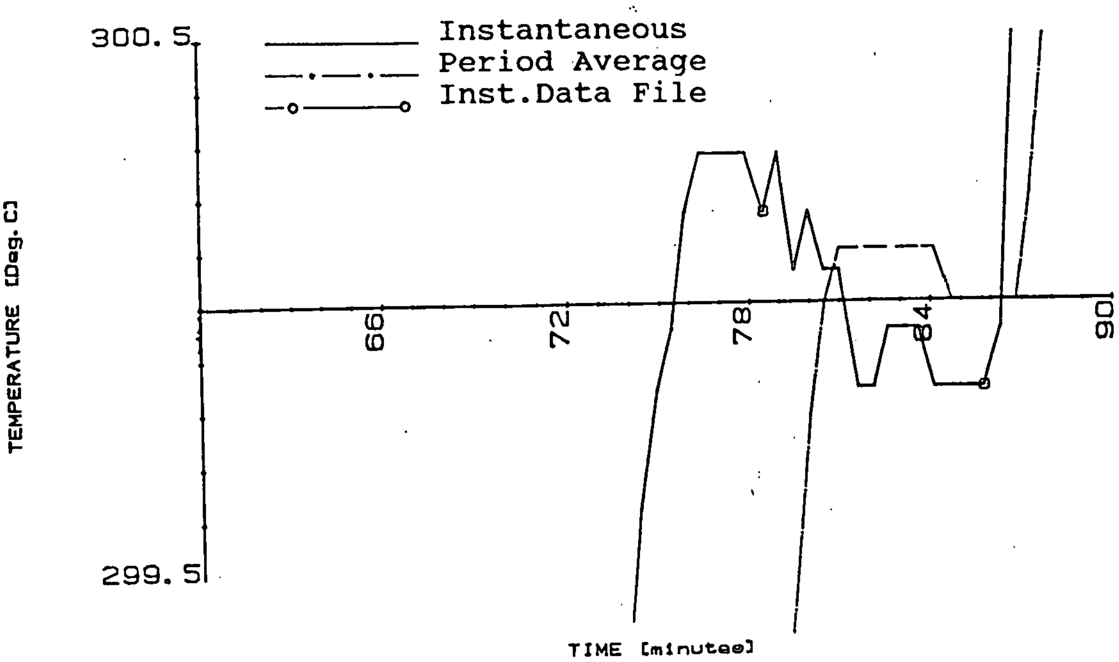
(c) **Test thermocouple instantaneous trend**

Lowest value in data average file : 302.1^{o}C

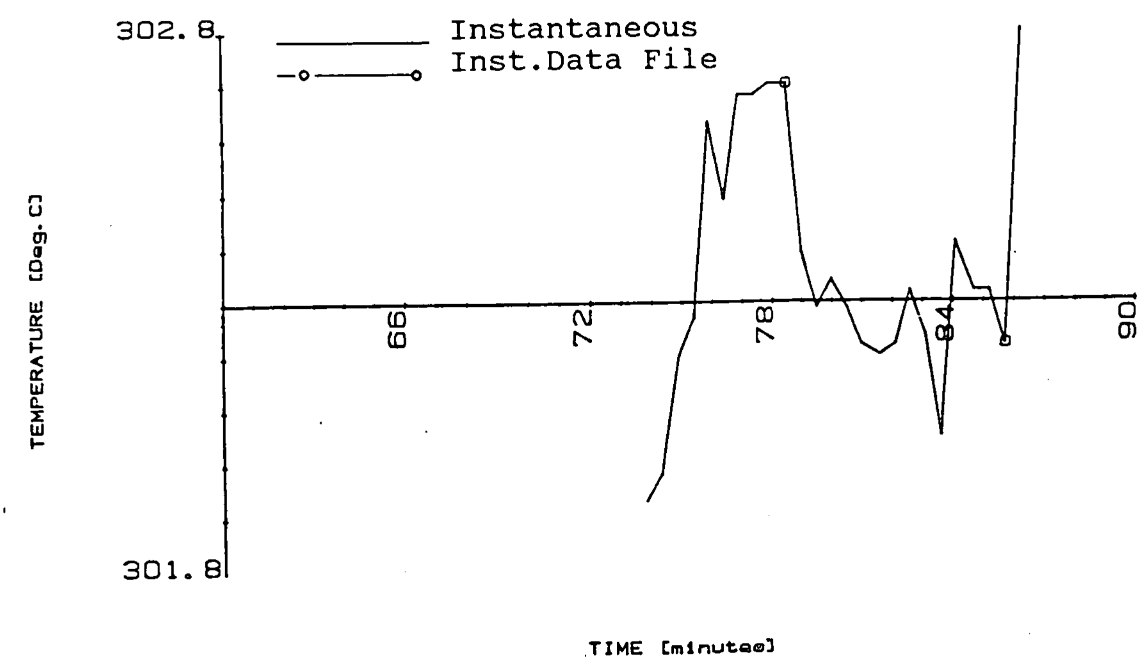
Highest value in data average file: 302.7^{o}C

Average at point of calibration : 302.3^{o}C

Confidence interval at 99.7% (3Sx):+/-0.4^{o}C



(a) STANDARD THERMOCOUPLE TEMPERATURE



(b) TEST THERMOCOUPLE TEMPERATURE

Figure 4.13 Thermocouple response curves: 300°C. Time indicates minutes expired since start of calibration run. Standard T/C X-axis intersects at 300.0°C. Test T/C axis intersects at 302.3°C.

4. **Data Comparison at 700^{o}C Target (Figure 4.14).**

Refering to figure 4.14:

(a) **Standard thermocouple instantaneous trend.**

Lowest value in data average file : 699.8^{o}C

Highest value in data average file: 700.2^{o}C

Average at point of calibration : 700.0^{o}C

Confidence interval at 99.7% (3Sx):+/-0.4^{o}C

(b) **Standard thermocouple average trend.**

Average is within (+/-)0.2^{o}C of 700.0^{o}C for a period of 12 minutes prior to calibration.

Average lags instantaneous by 4 minutes.

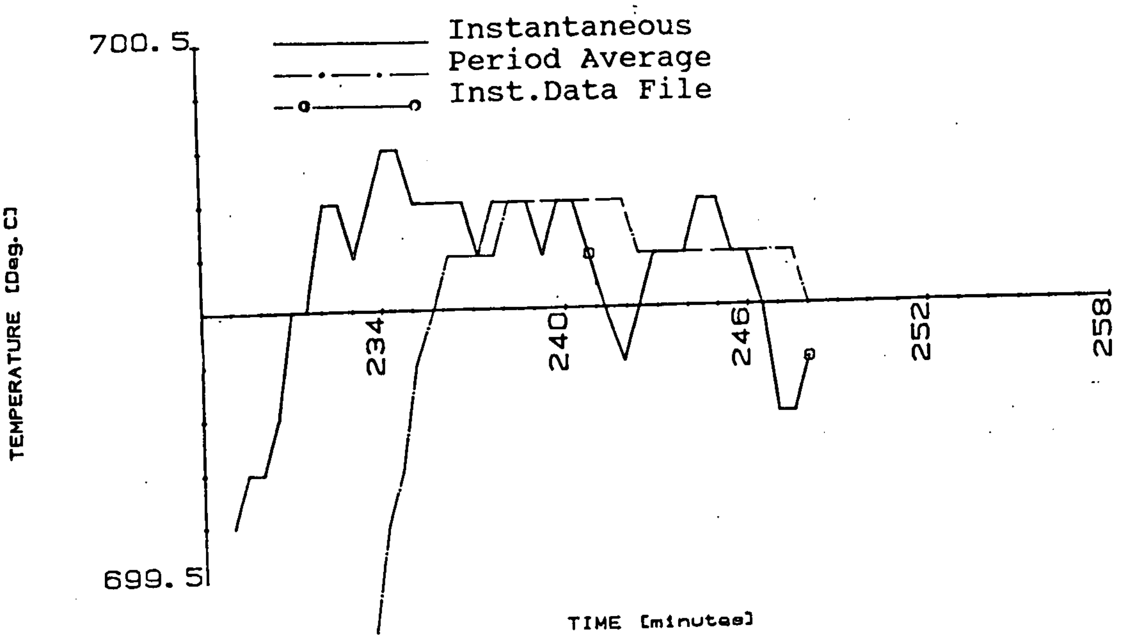
(c) **Test thermocouple instantaneous trend**

Lowest value in data average file : 700.2^{o}C

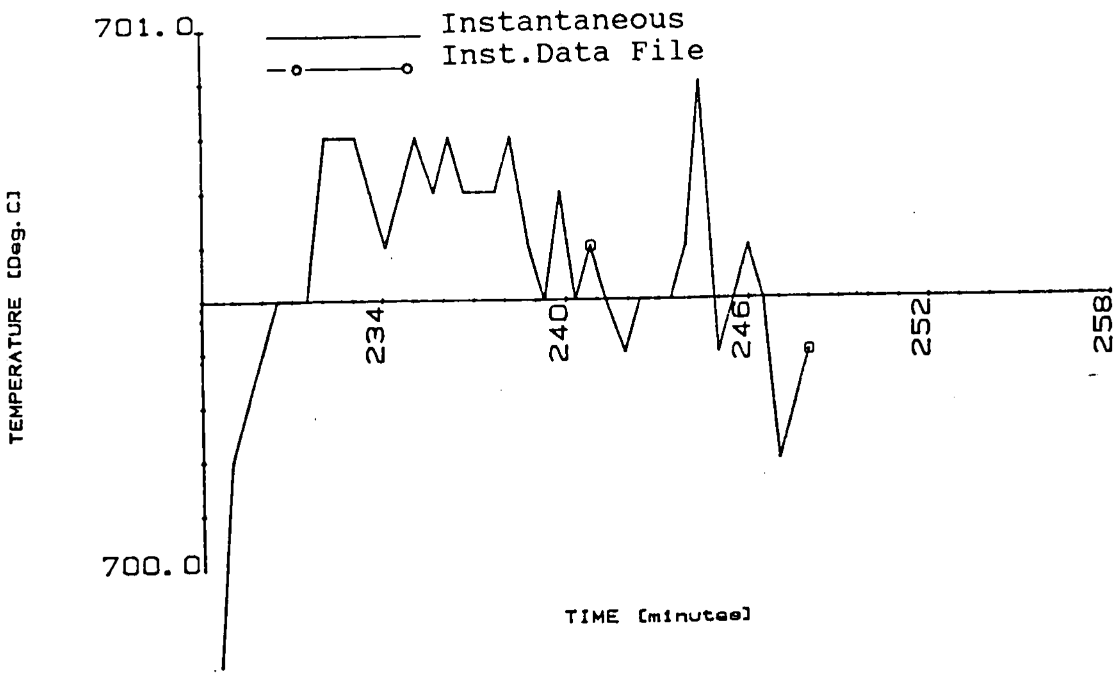
Highest value in data average file: 700.9^{o}C

Average at point of calibration : 700.5^{o}C

Confidence interval at 99.7% (3Sx):+/-0.5^{o}C



(a) STANDARD THERMOCOUPLE TEMPERATURE



(b) TEST THERMOCOUPLE TEMPERATURE

Figure 4.14 Thermocouple response curves: 700°C. Time indicates minutes expired since start of calibration run. Standard T/C X-axis intersects at 700.0°C. Test T/C axis intersects at 700.5°C.

5. **Response Time**

Figure 4.15 displays the trend of the standard thermocouple's averaged value for the complete calibration cycle (test dated 89.02.06). The trend plots the temperature cycle from 100 to 700°C. Indication of the stabilising periods at the calibration intervals of 100°C, is apparent in the flattening of the trend at these points. As with the foregoing temperature response curves, this graph is plotted directly from the array data file and as such represents, within the constraints of available resolution, precise detail of the original logged data. The particularly brief stabilisation period at 300°C, displayed in detail in figure 4.12, can be seen in perspective in figure 4.15. Total calibration run time is 248 minutes, which for seven calibration points, produces an average of 36 minutes per point. Actual intervals between calibration points are tabled below:

100 to 200°C: 37 minutes
to 300°C: 28 minutes
to 400°C: 35 minutes
to 500°C: 39 minutes
to 600°C: 34 minutes
to 700°C: 54 minutes

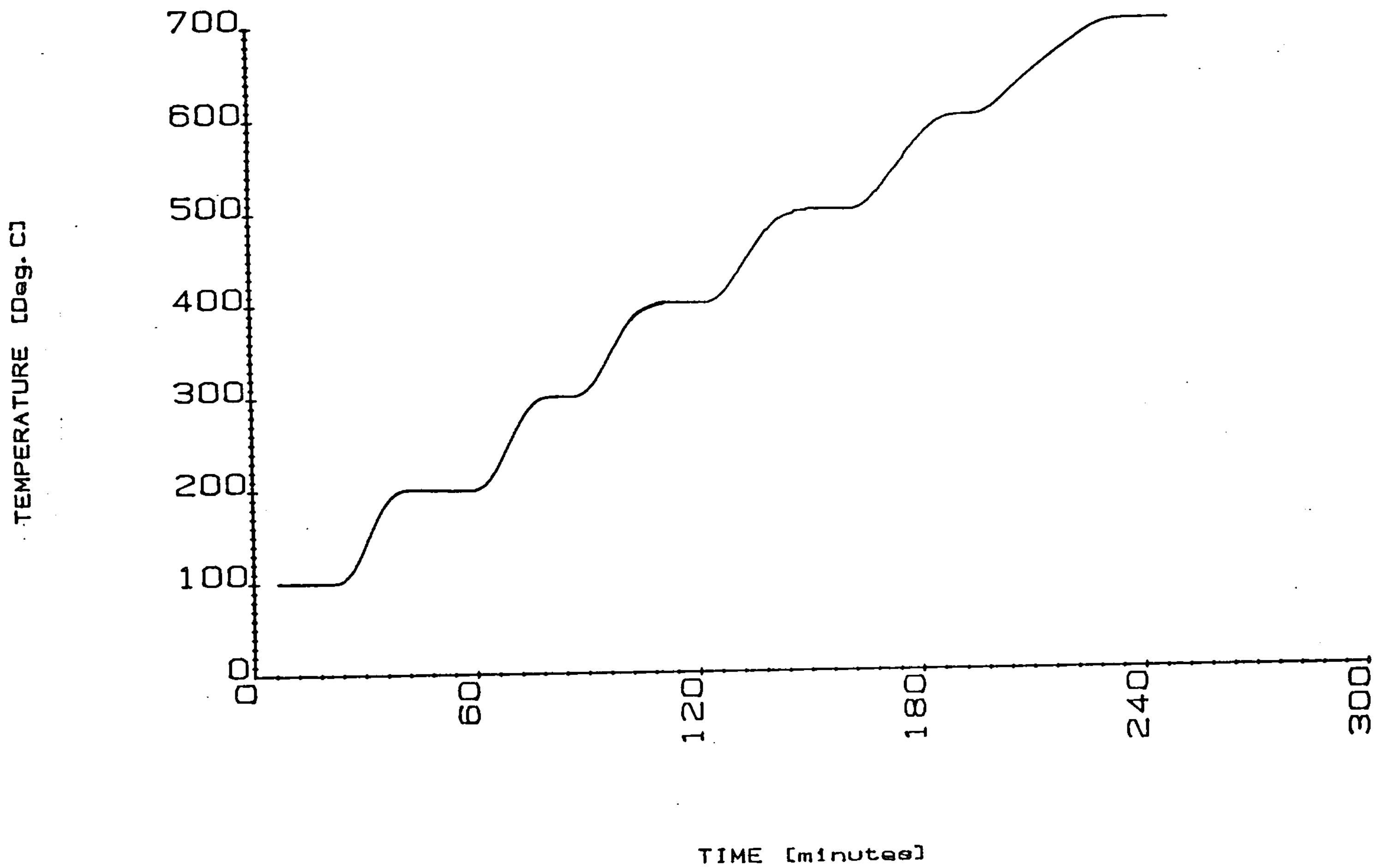


Figure 4.15 Standard thermocouple response during calibration trial run. Program set to run from 100 to 700 ^{o}C at 100 ^{o}C intervals. Plot represents the running average of the standard thermocouple as computed from the data average file.

Targeted response time is from 30 to 60 minutes per calibration point with a full calibration run average of 45 minutes per point. Response times less than 30 minutes run some risk of compromising the integrity of the data through an inadequate stabilisation period. Times in excess of 60 minutes risk bath level fluctuation. Whilst the LAB87 algorithm makes it possible to "tune" response time from one extreme to the other, the foregoing calibration cycle is representive of the program utilised for the comparative tests conducted in this project. Due to the number of variables affecting the algorithm it can, on occasion, be expected to "hang-up" as the program waits for a specific criterion to be met. Figure 4.16 provides an example of such an occurence. In this figure the respective response times are:

400 to 500°C: 108 minutes
to 600°C: 38 minutes
to 700°C: 60 minutes

6. Conclusion

The foregoing evaluation provides positive indication of the calibration system's capability of producing comparative calibrations within a (+/-)0.5°C level of uncertainty throughout the calibrating range (0 to 700°C). The study of the data file establishes also the system capability to provide the required fast calibration cycle

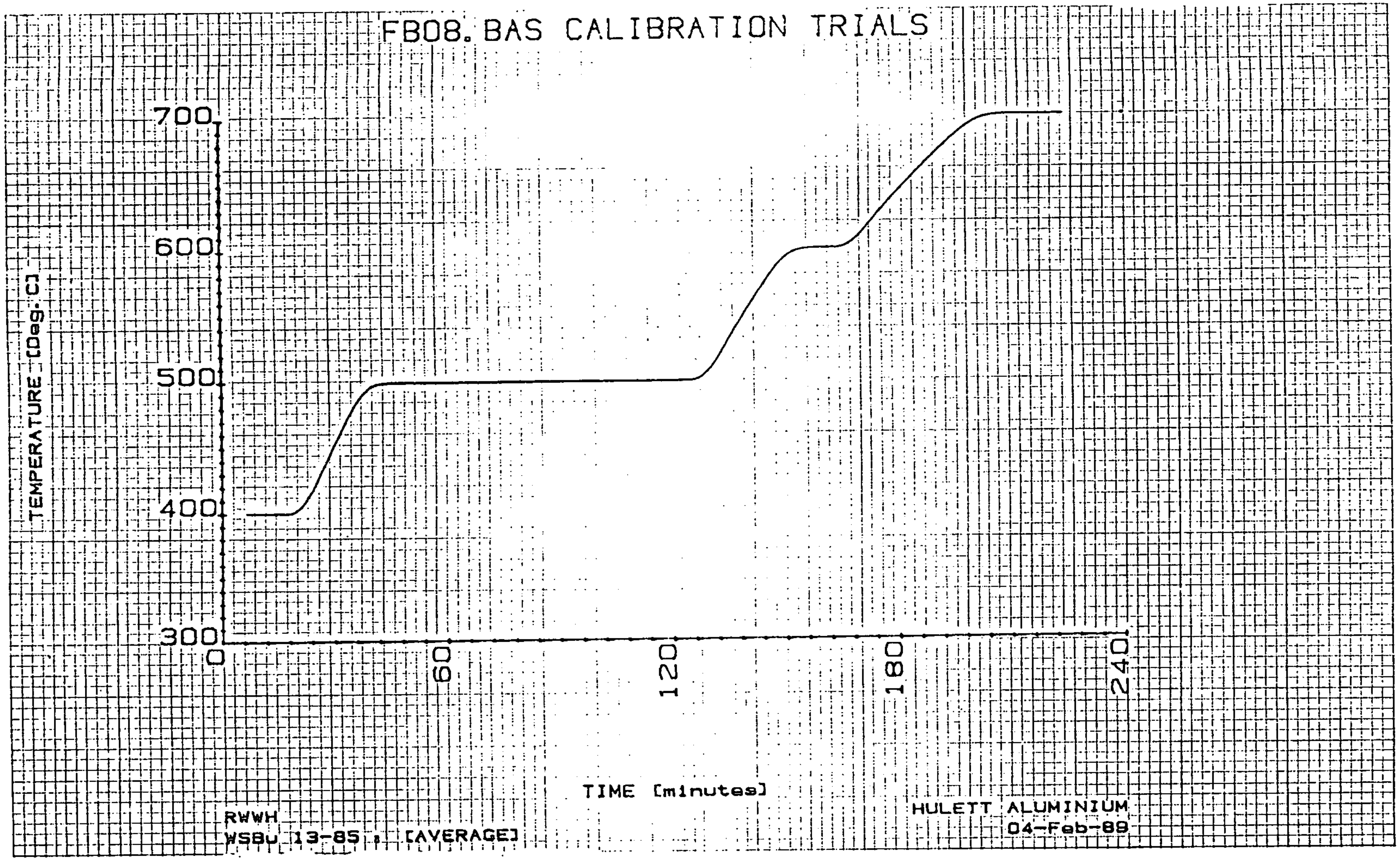


Figure 4.16 Calibration response trial. Standard thermocouple response curve showing excessive delay in reaching stabilisation at 500°C.

whilst maintaining data integrity. The ensuing subsection is complementary in extending the evaluation into further assessment of precision and repeatability.

b. **Standard/Reference Calibration Trials**

In a series of twelve calibration runs the control system's reference thermocouple is calibrated against the standard. Both thermocouples, serial numbers WSBu 13-85 and WSR 14-85, are certified traceable to the national standard and are of known stability. The thermocouples are both Type S and are of identical construction. The advantages of employing the reference thermocouple in these comparative trials are:

(i) The stability is such that any variance in calibration may be attributed to the system and not the thermocouple under test.

(ii) As the system reference thermocouple, computed temperatures are corrected by the thermocouple's unique calibration coefficients and values are thus directly traceable. Any deviation from standard may thus be viewed as system error.

(iii) The Type S thermocouple has an emf/temperature relationship of the order: 1 microvolt/ 0.1°C. This ensures that the calibration system's measuring circuits are evaluated at their highest level of sensitivity.

The twelve calibration cycles were each to run from 100 to 700°C with a calibration interval of 50°C. Of the twelve cycles, run (10), due to incorrect program instructioning ran with a 100°C calibration interval and run (11) aborted after 300°C due to a power failure on the furnace controller supply. There are

Table 4.8 Comparison test details for uncertainty trial. Twelve sequential calibration runs from 100°C to 700°C at 50°C calibration intervals.

Test	Serial	Start date & Time		Finish date & Time		Hours	Ave/Cal
(1)	10175	15 Sept.89	08.07	15 Sept.89	20.17	12hr10min	61min
(2)	10176	16 Sept.89	13.24	17 Sept.89	01.41	12hr12min	61min
(3)	10177	17 Sept.89	10.01	17 Sept.89	23.50	13hr49min	69min
(4)	10178	18 Sept.89	09.17	19 Sept.89	01.58	16hr41min	83min
(5)	10182	22 Sept.89	10.48	23 Sept.89	01.24	14hr36min	73min
(6)	10183	23 Sept.89	15.16	24 Sept.89	01.22	10hr 6min	51min
(7)	10184	24 Sept.89	19.02	25 Sept.89	13.48	18hr46min	94min
(8)	10185	25 Sept.89	20.08	26 Sept.89	09.23	13hr15min	66min
(9)	10186	27 Sept.89	08.24	27 Sept.89	20.48	12hr24min	52min
(10)	10187	30 Sept.89	18.06	1 Oct. 89	01.27	(7hr21min)*	(73min)*
(11)	10189	2 Oct. 89	17.45	2 Oct. 89	20.33	(2hr48min)*	(42min)*
(12)	10192	3 Oct. 89	12.40	3 Oct. 89	23.13	10hr33min	53min

NOTE:

1. Start time: Indicates time of first calibration i.e. 100°C.
2. Hours : Indicates time elapsed between first and last calibrations.
3. Ave/Cal : Indicates the average interval between calibrations.
4. Serial : Breaks in sequential numbering due to software trial runs. Normal calibration procedure not affected.

(*) Test number (10) - incorrect calibration interval (100°C).
(11) - incomplete data, test aborted after 300°C due to power failure.

several occurences of the system "hanging up" for extended periods, runs (7) and (8). All available data as regards calibration and response is presented and considered. From run (6) to run (12) a concurrent calibration trial was run with a 3 mm diameter bare wire Type K thermocouple. The results of this trial are treated separately and follow the standard/reference and response evaluation.

1. **Standard/Reference Comparison**

The twelve comparison calibration runs were set up according to standard procedure. Table 4.8 records the details of the test numbers, date of test and test duration. After initial setup all tests ran unsupervised and the data presented in this document is as captured. Over the 144 hour duration of the twelve tests more than 34000 polynomial equations are solved, 4200 of these results are used in computing the 140 standard and reference calibration values which are further reduced to 70 deviation values. After further statistical analysis the overall average deviation and uncertainty for a 99.7% confidence level is computed to be:

$\underline{0.1^{o}C}$ with an uncertainty of $\underline{(+/-)0.4^{o}C}$

Table 4.9 Standard/Reference Comparison.

Number of calibration runs: 12
Calibration program : 100 to 700 °C at 50 °C intervals.
Serial number of standard : WSBu 13-85

Values shown are deviation between reference and standard in °C
(Std-Ref)= Deviation value

Temp °C	Run number (1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)	(9)	(10)	(11)	(12)
100	-0.1	-0.2	0.0	-0.1	-0.1	-0.1	-0.1	-0.2	-0.1	-0.3	-0.2	-0.1
150	-0.2	-0.2	-0.1	-0.2	-0.1	-0.1	-0.1	-0.2	-0.2		-0.3	-0.2
200	0.0	-0.1	-0.1	-0.1	-0.1	0.0	0.0	-0.1	-0.1	0.0	0.0	0.0
250	0.0	0.0	0.0	0.0	0.2	0.1	0.2	0.1	0.0		0.1	0.1
300	0.0	0.0	0.2	0.1	0.3	0.2	0.2	0.2	0.2	0.2	0.2	0.2
350	0.1	0.0	0.1	0.1	0.1	0.2	0.2	0.2	0.3			0.2
400	0.0	0.0	0.0	0.1	0.1	0.0	0.1	0.1	0.2	0.0		0.1
450	0.1	0.0	0.1	0.0	0.1	0.1	0.0	0.0	0.1			0.1
500	0.1	0.1	0.2	0.0	0.0	0.0	0.1	0.1	0.1	0.0		0.0
550	0.1	0.1	0.2	0.1	0.1	0.1	0.1	0.3	0.2			0.0
600	0.1	0.1	0.2	0.3	0.1	0.2	0.2	0.2	0.2	0.2		0.2
650	0.1	0.2	0.1	0.2	0.1	0.1	0.2	0.2	0.1			0.0
700	0.1	0.2	0.2	0.1	0.1	0.1	0.0	0.1	0.0	0.1		0.1

Average deviation: <u>0.1 °C</u> with <u>(+/-)0.4 °C</u> uncertainty
(99.7% confidence interval).

Note: 1. Run (10) Incorrect program entry. Incomplete data.
2. Run (11) Aborted after 300 °C due to power supply failure.

Table 4.10 Statistical treatment of Standard/Reference deviation data.

All availaible data from the twelve 12 Standard/Reference calibration runs is utilised in the following average and standard deviation analysis.

A three standard deviation (99.7% confidence interval) is quoted.

Temp. °C	Average deviation °C	3Sx (+/-) °C
100.0	-0.1	0.2
150.0	-0.2	0.2
200.0	-0.1	0.2
250.0	0.1	0.2
300.0	0.2	0.3
350.0	0.2	0.3
400.0	0.1	0.2
450.0	0.1	0.2
500.0	0.1	0.2
550.0	0.1	0.3
600.0	0.2	0.2
650.0	0.1	0.2
700.0	0.1	0.2
Overall:	0.1	0.4

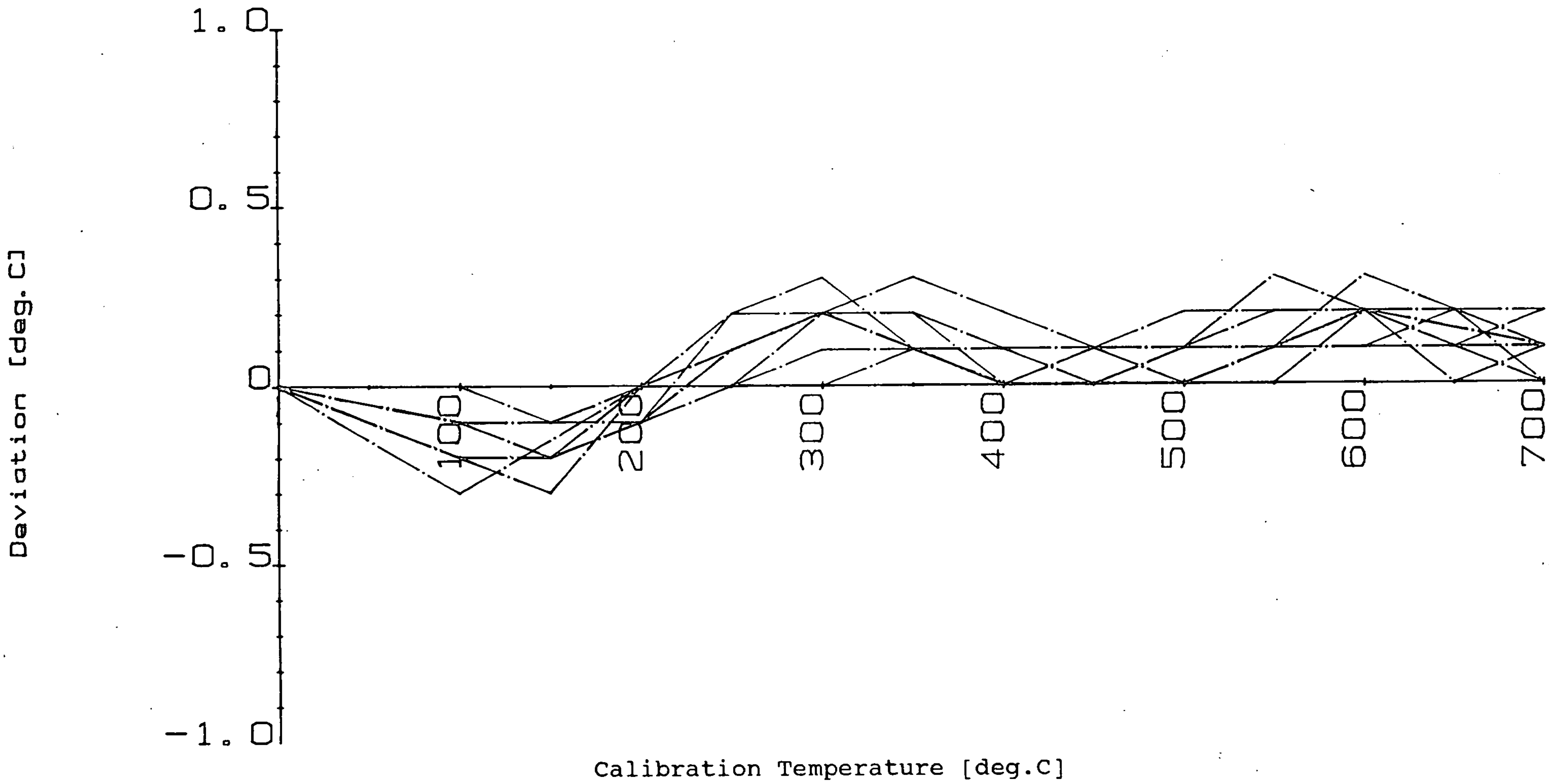


Figure 4.17 Standard/Reference comparison trial. Deviation results of twelve calibration runs shown overlaid.

Table 4.9 lists the complete set of deviation values, table 4.10 the results of deviation analysis of the data at each calibration temperature. Figure 4.17 illustrates a graphical overlay of the deviation data presented in table 4.9.

2. **Response Times**

Response time is considered as the time expired between the establishment of two consecutive calibration points. In the data being considered these points follow at 50^{o}C intervals. Table 4.11 lists the appropriate data set. An analysis of these data is performed in order to ascertain the system's average response in minutes per calibration point. In the treatment of these data the following delimitations are set:

(i) Only responses falling within the 100 to 550^{o}C range as applicable to this project are considered.

(ii) Trials (10) and (11) having incomplete data sets are omitted from the evaluation.

(iii) The response on trial (7) for the interval 500 to 550^{o}C (454 minutes) is omitted as it is considered to have been influenced by an abnormal situation. Calibration test listings record the following:
500^{o}C logged at 01.06 on 25 Sept.89
550^{o}C logged at 08.40 on 25 Sept.89
It is considered probable in this case that the delay is due to an elememt fuse failure, that is rectified at the start of the following shift without otherwise disrupting the program.

(iv) Although falling outside of the range specified in (i) above it is noted that at 700^{o}C reponse time is severely influenced by available power (refer to values in table

Table 4.11 **Response times for Standard/Reference comparison calibration trials. Times in minutes per interval.**

Interval °C	Test No. (1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)	(9)	(12)
100 to 150	33	50	**65**	52	**76**	**87**	58	32	**62**	33
150 to 200	30	48	**110**	**172**	**119**	27	52	35	41	49
200 to 250	60	32	60	**82**	32	29	24	25	54	**70**
250 to 300	**158**	37	54	52	39	48	30	50	**91**	27
300 to 350	33	**61**	56	**70**	35	40	32	33	51	26
350 to 400	24	55	**71**	**65**	49	40	43	**101**	39	29
400 to 450	44	38	**68**	57	28	30	45	37	45	**61**
450 to 500	32	58	**63**	**61**	**74**	38	**80**	47	52	44
500 to 550	40	59	**66**	52	55	53	(454)*	38	**63**	49
550 to 600	56	**63**	**79**	**78**	52	**62**	**102**	43	47	**70**
600 to 650	54	**125**	57	**69**	**73**	**65**	**72**	51	**74**	59
650 to 700	**166**	**111**	**80**	**191**	**244**	**87**	**134**	**303**	**125**	**116**

Average response: <u>53 minutes</u> (Standard deviation <u>26 minutes</u>)

NOTE:

1. Tests (10) and (11) omitted due to incomplete data sets.

2. Data exceeding 60 minutes is printed in bold typeset.

* Datum for test (7), interval 500 to 550, not used in analysis, (refer to text).

4.11). Improvement may be made by insulating the thermocouple entry cover plate, by providing a stabilised power source or by increasing element rating.

Within the above delimitations the average reponse time computed from these data is:

<u>53 minutes</u> (standard deviation: <u>26 minutes</u>).

Whilst a high standard deviation may be expected from an evaluation of data that is determined as the result of a process dependent on a number of interactive variables, the above value is considered excessive. The average value also exceeds the targeted 45 minutes. An investigation of the control system determined that an "adaptive" tuning feature of the furnace controller had generated over-sensitive control tuning. Locking out the "adaptive" feature and refitting original control parameters led to a lowering of average response time and improved data variance. Table 4.12 provides a listing of response times compiled from recent working calibration runs. Over this data set the average response time is computed as:

<u>32 minutes</u> (standard deviation: <u>8 minutes</u>).

As the furnace control is handled independantly of the LAB87 system control algorithm it does not, other than affecting the response time,

Table 4.12 **Calibration response times under working load conditions, after correction of furnace control tuning.**

Interval °C	(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)	(9)	(10)
	Test No.									
50 to 100	**46**	26	33	19	38	28	34	21	39	41
100 to 150	34	32	33	**50**	37	28	24	20	35	29
280 to 240	28	24	34	22	26	25	**47**	21	35	28
200 to 250	30	32	24	40	24	26	26	40	26	**53**
250 to 300	27	26	26	29	29	24	36	28	24	33
300 to 350	42	24	**47**	29	27	24	**50**	27	21	22
350 to 400	24	30	31	41	28	29	30	28	24	28
400 to 450	31	(171)*	**53**	**47**	45		36	24	33	34
450 to 500	36		34	42	43		**49**	34	34	28
500 to 550	30		33	35	**49**		35	24	33	45

Average response: 32 minutes (Standard deviation 8 minutes).

NOTE:

1. Tests (2) and (6) have incomplete data sets due to failure to complete the test runs.
2. Data exceeding 45 minutes is printed in bold typeset.
3. Test details provided in table 4.13.

* Datum for test (2), interval 400 to 450, not used in analysis, as the delay is considered to be linked to the fault which resulted in the subsequent failure of the test run.

Table 4.13 **Further Standard/Reference comparison data, captured under work load conditions.**

Number of calibration runs: 10
Calibration program : 50 to 550 ^{o}C at 50 ^{o}C intervals.
Serial number of standard : WSBu 13-85
Serial number of reference: Tests(1-9): WSB 14-85
Test (10) : SSY 01-90

Values shown are deviation between reference and standard in ^{o}C
(Std-Ref)= Deviation value

Temp ^{o}C	Run number (1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)	(9)	(10)
50	-0.3	-0.3	-0.3	-0.4	-0.1	-0.1	-0.3	-0.2	-0.3	-0.1
100	-0.2	-0.2	-0.2	-0.2	-0.2	-0.1	-0.3	-0.2	-0.2	-0.1
150	-0.1	-0.2	-0.2	-0.3	-0.2	-0.2	-0.1	-0.2	-0.2	-0.1
200	0.0	-0.1	-0.1	-0.1	-0.1	-0.1	-0.1	0.0	-0.1	-0.1
250	0.1	0.0	0.1	0.0	0.0	0.1	0.0	0.0	0.1	0.1
300	0.1	0.0	0.1	0.0	0.2	0.1	0.0	0.1	0.1	0.2
350	0.1	0.1	0.0	0.1	0.1	0.1	0.0	0.1	0.0	0.1
400	-0.1	0.0	0.0	0.0	-0.1	0.1	0.0	-0.1	-0.1	0.1
450	0.0	0.0	-0.1	0.0	0.0		0.1	-0.1	-0.1	0.1
500	0.0		0.0	0.0	0.0		0.0	0.1	0.0	0.1
550	0.2		0.1	0.2	0.0		0.1	0.1	0.1	0.2

Average deviation: 0.0 ^{o}C with (+/-)0.4 ^{o}C uncertainty
(99.7% confidence interval)

Note: 1. Runs (2) and (6): Incomplete data.
2. A new reference thermocouple,(SSY 01-90), was used in run (10).

TEST DETAILS:
Selection criteria: Tests with the required program, (50-550/50), were sequentially selected from the project files.

Identification:

Test	Serial	Date
(1)	10216	6 May 90
(2)	10217	8 May 90
(3)	10219	9 May 90
(4)	10225	10 Jun 90
(5)	10226	11 Jun 90
(6)	10227	11 Jun 90
(7)	10236	14 Jun 90
(8)	10240	16 Jun 90
(9)	10241	16 Jun 90
(10)	10252	2 Jul 90

cause any interference with the calibration data. This consideration is confirmed by the correlation of the deviation results produced by three sets of calibration runs, (1) the twelve Standard/Reference comparison runs considered above, (2) a set of six similar comparisons run with the same Standard and Reference thermocouples during the development stage of LAB87, (3) the ten working calibration runs considered above. The deviation data and test details for these ten runs are presented in table 4.13. The summary of the analyses of the deviation results produced by these three data sets, and quoted for a 99.7% (3Sx) confidence interval is given below:

Data set	Average deviation	Uncertainty
	^oC	$(+/-)\,^oC$
(1): 12 runs	0.1	0.4
(2): 6 runs	0.0	0.3
(3): 10 runs	0.0	0.4

3. **Conclusion**

In terms of the foregoing empirically established uncertainty levels, a precision claim of (+/-)0.5^{o}C is considered justified. The traceability claim of (+/-)1.0^{o}C makes allowance for the full precision uncertainty budget as well

Table 4.14 CSIR audit results: annual assessment of Hulett Aluminium Temperature Laboratory, July 1989.

Audit Results

Type S thermocouple SSR 06-89

Hulett Aluminium °C	Output (mV)	NMS Calc. °C	(NMS - Hulett) °C	'bmc' °C
100	645	99,96	-0,04	± 1
200	1440	200,06	+0,06	± 1
300	2323	300,14	+0,14	± 1
400	3259	400,04	+0,04	± 1
500	4233	500,03	+0,03	± 1
600	5238	599,97	-0,03	± 1
700	6277	699,84	-0,16	± 1
800	7349	799,75	-0,25	± 1
900	8453	899,67	-0,33	± 1
1000	9592	999,84	-0,16	± 1

NOTE: NMS refers to National Measuring Standards as allowing for an additional transfer uncertainty due to difference in construction between the standard thermocouple and the thermocouples under test.

Final verification of the appropriateness of the claimed precision and uncertainties is provided by the CSIR, 1989 audit results (table 4.14) . The seven calibration points, from 100 to 700°C, calibrated, unattended, according to normal LAB87 procedures in a period of 8 hours 4 minutes (test number: 10163), produced the following overall deviation and uncertainty (99.7%):

Deviation: <u>0.0°C</u>. Uncertainty:<u>(+/-)0.3°C</u>

The calibration points from 800 to 1000°C were calibrated manually, by comparison method, in a Leeds and Northrup vertical thermocouple testing furnace.

c. **Type K Calibration Trials**

In concluding the evaluation of the Automated Live Bed Technique, the deviation results of a series of seven calibration trials on a Type K thermocouple are considered. These trials run concurrently with the final seven [runs (6-12)] Standard/Reference comparison tests (table 4.8). The results of these trials serve to reintroduce the central problem of initial drift in calibration in Type K thermocouples.

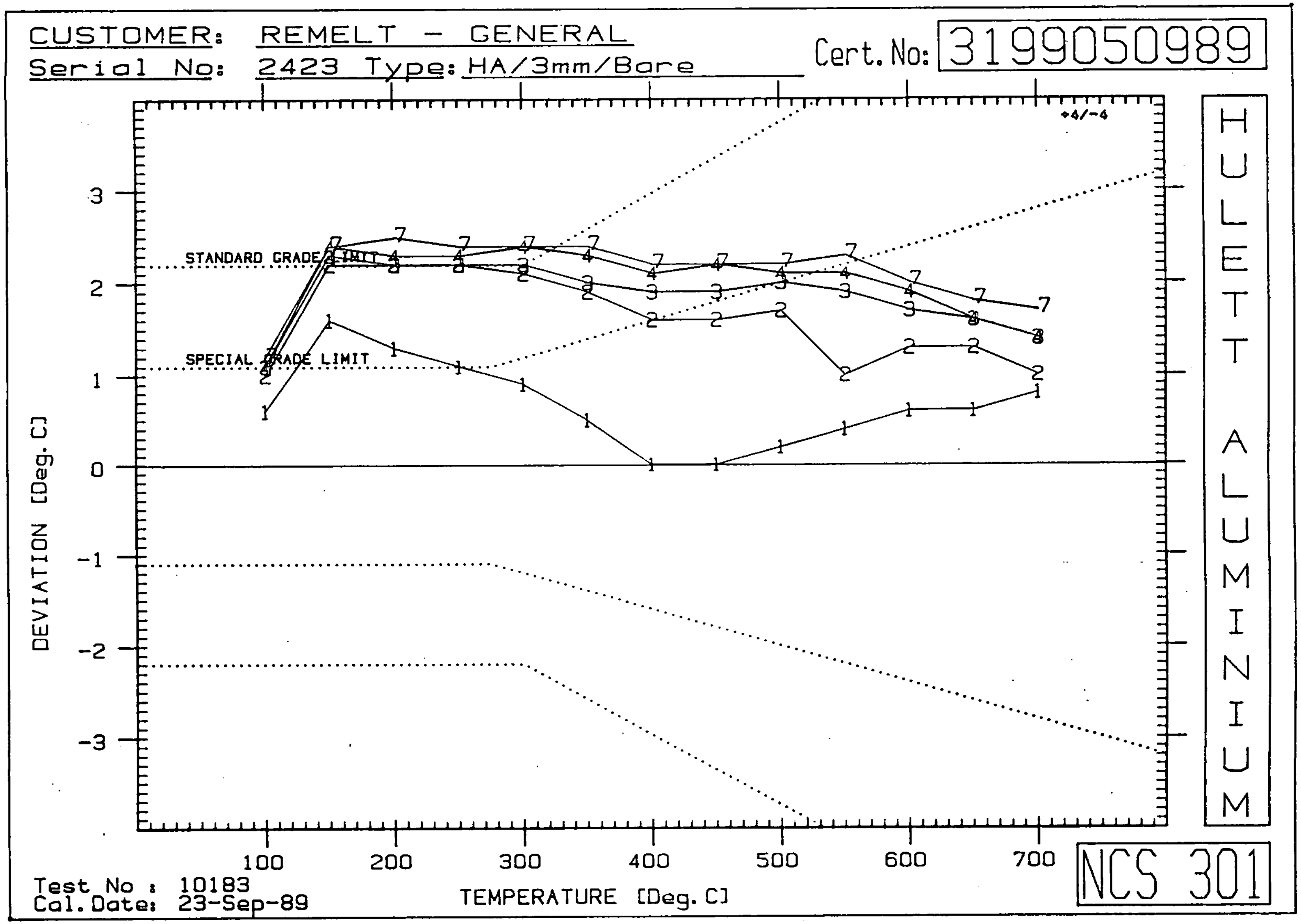


Figure 4.18 Type K calibration trial. Graphical representation of deviation data from calibration runs; (1),(2),(3),(4) and (7) overlaid on common axes. Test number and date refer to run (1).

The evaluation of these data provides perspective for the premise that, for the Type K thermocouple, uncertainties claimed are valid, at the time of calibration, without regard to lack of assurance in terms of subsequent repeatability of the result.

1. **Comparison of the Calibrations**

Figure 4.18 provides a graphical overlay of the deviation results of five of the calibration runs. Runs (5) and (6) [corresponding to Standard/Reference comparison tests (10) and (11)] are omitted due to incomplete data sets. In figure 4.18 the numbering of the calibration point indices represents the calibration run sequence. The extent of initial drift [between runs (1) and (2)] and the continuing more gradual drift [run (2) to (3), (3) to (4) and (4) to (7)] is apparent from the data plots on this graphical representation. The variant datum point at 550°C on run (2) is linked to the excessive response (454 minutes) dealt with under the Standard/Reference comparison response evaluation, and it is most probable that the deviant datum is associated with an alteration of immersion level.

Table 4.15 Type K calibration data set.

Number of calibration runs: 7
Calibration program : 100 to 700 °C at 50 °C intervals.
Serial number of standard : WSBu 13-85
Serial number of thermocouple under test: 2423

Values shown are deviation between thermocouple under test and Standard in °C. (Std-test t/c)= Deviation value

Temp °C	Run number (1)	(2)	(3)	(4)	(5)	(6)	(7)	3Sx (1-7)	3Sx (3-7)
100	0.6	1.0	1.1	1.1	1.1	1.2	1.2	0.6	0.2
150	1.6	2.2	2.3	2.4		2.4	2.4	0.9	0.2
200	1.3	2.2	2.2	2.3	2.4	2.5	2.5	1.3	0.4
250	1.1	2.2	2.2	2.3		2.4	2.4	1.5	0.3
300	0.9	2.1	2.2	2.4	2.4	2.5	2.4	1.7	0.3
350	0.5	1.9	2.0	2.3			2.4	2.3	0.6
400	0.0	1.6	1.9	2.1	2.1		2.2	2.5	0.4
450	0.0	1.6	1.9	2.2			2.2	2.8	0.5
500	0.2	1.7	2.0	2.1	2.2		2.2	2.3	0.3
550	0.4	1.0	1.9	2.1			2.3	2.4	0.6
600	0.6	1.3	1.7	1.9	1.9		2.0	1.6	0.4
650	0.6	1.3	1.6	1.6			1.8	1.4	0.4
700	0.8	1.0	1.4	1.4	1.6		1.7	1.1	0.5
Average uncertinty (+/-)								1.7	0.4

Note: 1. Run (5) Incorrect program entry. Incomplete data.
2. Run (6) Aborted after 300 °C due to power supply failure.

2. Data Evaluation

Table 4.15 tables the Type K deviation result data set for the complete seven runs. Runs (5) and (6) calibration data are incomplete. Whilst this data set does not contain sufficient data to purport to be a full investigation into the drift characteristics of the Type K thermocouple under calibration conditions, it does support the drift related findings discussed in detail in the review of the related literature. In addition each test run ranging from 100 to 700°C with calibration points every 50°C (13 calibration points per run) is considered comprehensive in providing a representative calibration curve. In the evaluation of these data, two sets of uncertainty figures for each calibration point are produced:

(i) The first set of uncertainty figures are computed utilising the entire data set from run (1) through (7). The uncertainties representative of a 99.7% confidence interval range from:

Lowest : (+/-)0.6°C at 100°C
Highest: (+/-)2.8°C at 450°C
Average: (+/-)1.7°C.

(ii) The second set of uncertainty figures omit runs (1) and (2), thus evaluating data after the "preaging" effect of the first two calibration runs. With this evaluation the uncertainty ranges from:

Lowest : (+/-)0.2°C at 100°C
Highest: (+/-)0.6°C at 350 and 550°C
Average: (+/-)0.4°C.

3. Conclusion

The foregoing data set (table 4.15) and accompanying evaluation provides indication (figure 4.18) of the extent of the problem of drift occuring subsequent to initial calibration. The second set of deviation uncertainty results, whilst based on a limited set of data, indicate the feasibility of securing reliable calibration results, within an uncertainty of (+/-)0.5^{o}C for Type K thermocouples, providing that the calibration results are always viewed within the correct perspective in terms of past calibration history and future calibration expectancy.

CHAPTER 5

GENERAL PROCEDURES

5.1 PRELIMINARY AGING TRIALS OF TYPE K THERMOCOUPLES

5.1.1 Introduction

During 1985 the Hulett Aluminium Temperature Laboratory began investigating the extent and cause of discrepancies detected in the calibration of Type K thermocouples after short periods of use. Through simulating working conditions in the laboratory it could be observed that after a substantial, (3 to 4°C), initial shift in calibration the thermocouple attained a degree of stability with a comparatively slight, continuing positive drift in calibration. The extent of this subsequent drift appeared to be contained within a 1°C margin of the initial offset.

Preliminary investigations are carried out on 20 gauge bare thermocouple wire. Limited trials, during this preliminary stage, using 3 mm diameter, mineral insulated (MI), thermocouples indicate similar performance (figure 5.4). In introducing the general procedures adopted for conducting the final aging trials, two examples from the preliminary trials as well as the results of a batch calibration trial on 3 mm MI thermocouples are discussed.

Table 5.1 **Preliminary aging trial, September 1985.**
Thermocouple wire batch serial number: 0037.

(a) Calibration status (relative to IPTS-1968) at each successive stage of the trial.

Calibration	Temperature (°C)						
	100	200	300	400	500	600	700
Acceptance ("as received"): A	-0.3	-0.4	-0.9	-1.4	-1.0	-1.3	
After preaging (200 hr at 500°C):B	1.0	2.6	3.1	3.5	4.1	4.5	4.3
After use (83 hr):C		2.9	3.5	3.7	4.2		

NOTE: Values not recorded are not given on original records.

(b) Comparison between calibration results.

Comparison	Temperature (°C)			
	200	300	400	500
Final : Initial (Calibration shift): (C-A)	3.3	4.4	5.1	5.2
Final : Expected(IPTS-68) (Indicated error): (C)	2.9	3.5	3.7	4.2
Final : Preaged (Possible limitation): (C-B)	0.3	0.4	0.2	0.1

5.1.2 Preliminary Trials

a. December 1985

Table 5.1 presents the data related to these trials. Figure 5.1 provides graphical representation of the calibration in the, "as received", preaged, and used conditions. For this trial lengths of thermocouple wires are taken from bulk reels (serial number 0037) of matched nickel-chromium and nickel-aluminium thermocouple wire and wound onto individual steel spools. These spools and wire with the serial number 0193, are aged for a period of 200 hours at a soak temperature of 500^{o}C, (+/- 10^{o}C). A batch of four load thermocouples together with a 2 m "test" thermocouple is manufactured from this preaged wire.

The load thermocouples are subjected to a period of 83 hours of process use (approximately 20 work cycles), in a solution heat treatment furnace with a work cycle that includes the quenching of the load and thermocouples from soak temperature of 503^{o}C. Following this period the thermocouples are withdrawn from service and calibrated.

In the presentation of the results, table 5.1, the term "acceptance calibration", refers to the initial calibration of a sample taken from the bulk reels, (serial number 0037), on receipt of the material

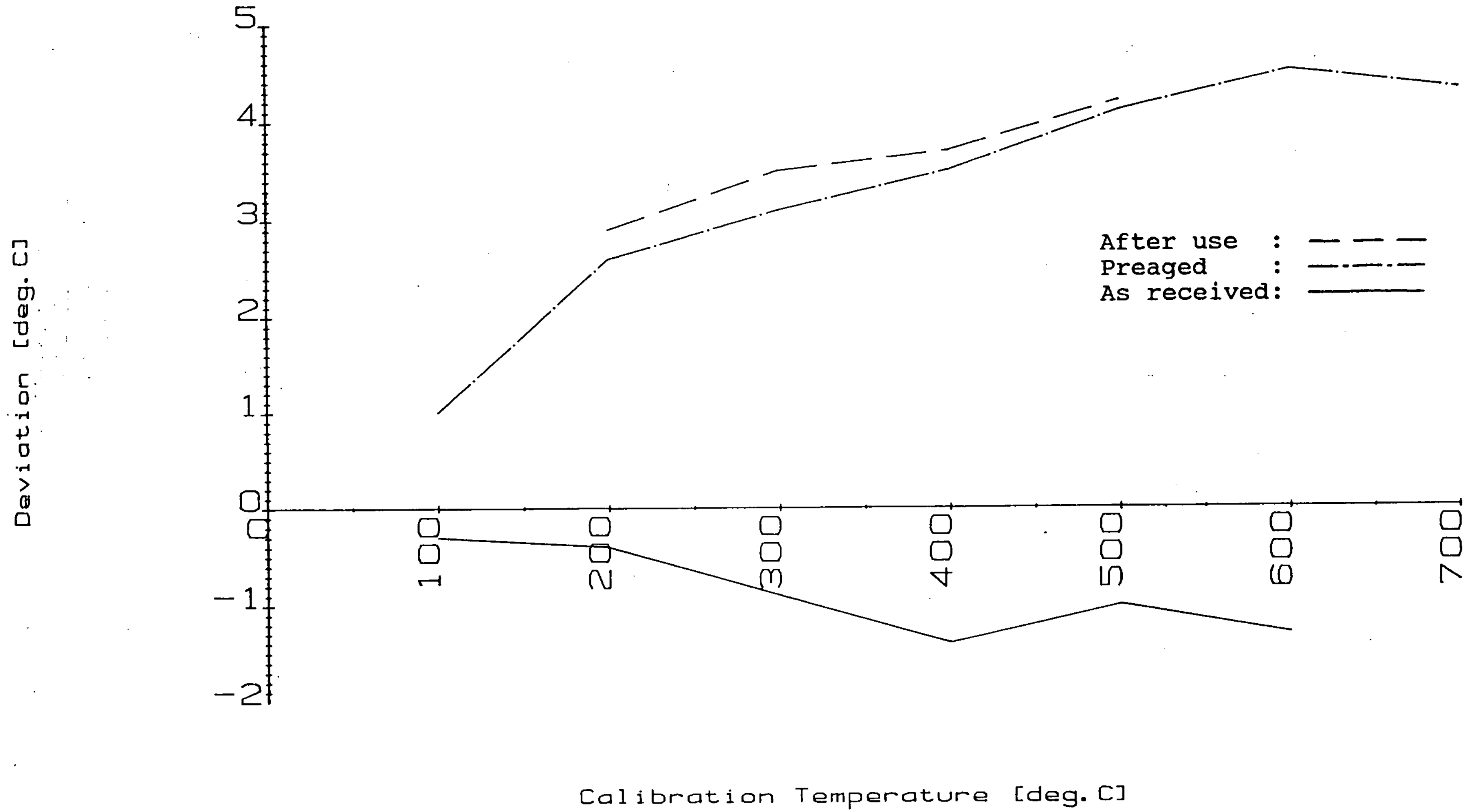


Figure 5.1 Preliminary aging trial results, September 1985. The plots indicate the correlation of the preaged and after use calibrations relative to the "as received" calibration.

prior to its acceptance for process use. This calibration is representative of the "as received" calibration for those matched reels of thermocouple wires.

A comparison of the calibration results for each of the three stages, (figure 5.1), shows the association between the preaged and used condition calibrations. That the used calibration (83 hours) is more positive than the preaged calibration (200 hours) is indicative that cycling has a greater influence on the aging process than has duration. The severity of the offset produced during the work cycle and the potential for balancing this effect through a controlled preaging and calibration procedure is apparent from consideration of table 5.1 (b). Row 1 of this table provides indication of the full extent of the shift in calibration between the "as received" and used thermocouple conditions. A more practical evaluation is given in row 2 of the table which indicates the differences between the expected calibration (following IPTS-1968) and the used condition calibration. This row may be considered as indicating the effective error of the thermocouples after the 20 work cycles. At the critical temperature of 500^{o}C the error is in the order of 4^{o}C. Row 3 of the table indicates the difference in calibration between the preaged thermocouple and the used thermocouple. These

Table 5.2 Manufacturer's details for thermocouple wire batch serial number: 1405.

Manufacturer: Driver Harris (France)

Supplier: Republic Thermocouples (Pty.) Ltd.

Description: Standard grade, 0.8 mm diameter, type K, bare thermocouple wire.

Nickel-chromium (+) : Melt number 2985.
Net mass 6.008 kg.

Nickel-aluminium (-): Melt number 1183.
Net mass 5.130 kg.

Manufacturer's calibration data. *(mV/Pt 67):

Calibration Temp.(^{o}C)	*T1	*T2	Combined uV	Indicated Temp.(^{o}C)
200	5.988	2.193	8181	201.1
400	12.744	3.640	16384	399.7
600	19.600	5.296	24896	599.9
800	26.213	7.077	33290	800.3

Hulett Aluminium acceptance test thermocouple s/n: 1406.

differences provide indication of the degree of correction that may be achieved through implementation of controlled preaging and application of calibration offset.

b. **July 1988**

In this trial five thermocouples, each 2 m long, are manufactured from the bulk source, serial number 1405, Type K thermocouple wires. Table 5.2 furnishes details of this source. Each of these five thermocouples is individually heat treated according to the following procedures:

Serial number 1418: No heat treatment. To be used as " control" thermocouple.
Serial number 1420: Heated to 500°C and quenched at intervals ranging from 1 to 4 hours for a total of ten cycles. Effective soak time at 500°C is 20 hours.
Serial number 1423: Soaked at 500°C for 52 hours.
Serial number 1421: Soaked at 500°C for 100 hours.
Serial number 1422: Soaked at 500°C for 250 hours.

The heat treatment of thermocouple serial number 1420 provides simulation of actual work cycling. The three soak periods of 52, 100, and 250 hours respectively are used to provide indication of the degree of drift occuring at 500°C. With trials of this nature only one calibration run at the conditioned state is possible since the calibration procedure itself

Table 5.3 Final preliminary preaging trial details, July 1988. Thermocouple wire batch S/N: 1405.

(a) Manufacturers, Hulett Aluminium acceptance, and trial control thermocouple calibration deviation data given as deviation from IPTS-68 in degrees Celsius.

Calibration	Temperature (°C)							
	100	200	300	400	500	600	700	800
Manufacturer:		1.1		-0.3		-0.1		0.3
Acceptance:		0.2	-0.5	-0.8	-0.5	-0.3	0.1	
Control:	0.1	0.2	-0.3	-1.2	-0.5			

NOTE: Values not recorded are not given on original records.

(b) Summary of preaging trial calibration results. (Deviation from IPTS-68 in degrees Celsius).

S/N	Preaging	Temperature (°C)				
		100	200	300	400	500
1418	Control	0.1	0.2	-0.3	-1.2	-0.5
1420	10 X Quench (20 hr)	1.1	2.5	2.6	3.0	3.3
1423	52 hr at 500°C	1.0	2.6	2.7	3.1	3.6
1421	100 hr at 500°C	1.2	2.5	3.1	3.5	3.5
1422	250 hr at 500°C	1.3	3.0	3.5	3.7	4.2

(c) Difference in calibration between preaged thermocouples S/N's 1421 to 1423, and the control thermocouple S/N 1418 as shown in (b).

S/N	Preaging	Temperature (oC)				
		100	200	300	400	500
1423	52 hr at 500°C	0.9	2.4	3.0	4.3	4.1
1421	100 hr at 500°C	1.1	2.3	3.4	4.7	4.0
1422	250 hr at 500°C	1.2	2.8	3.8	4.9	4.7

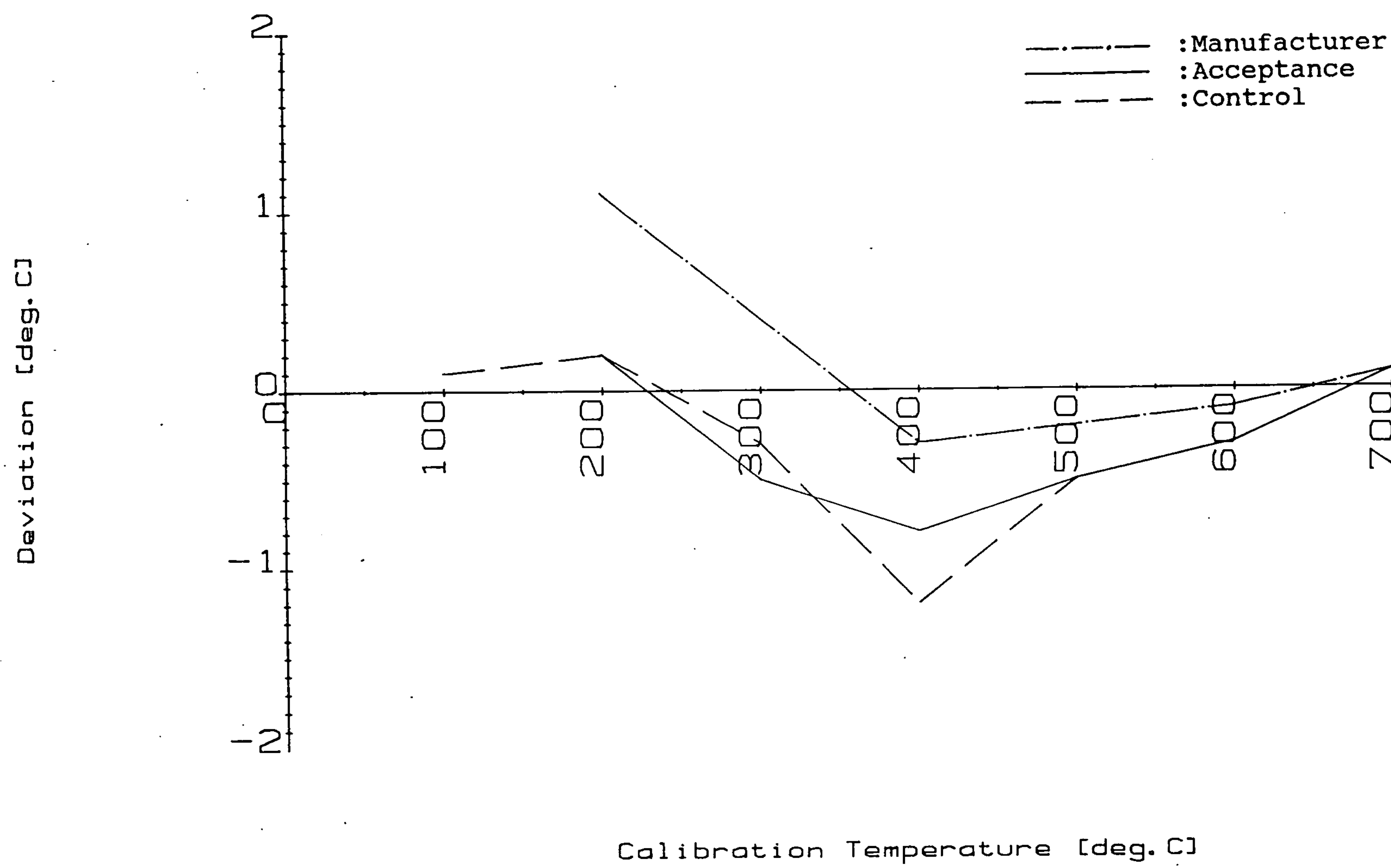


Figure 5.2 Initial calibration detail. Plots show manufacturer's calibration, Hulett Aluminium acceptance calibration, and preaging trial control thermocouple calibration.

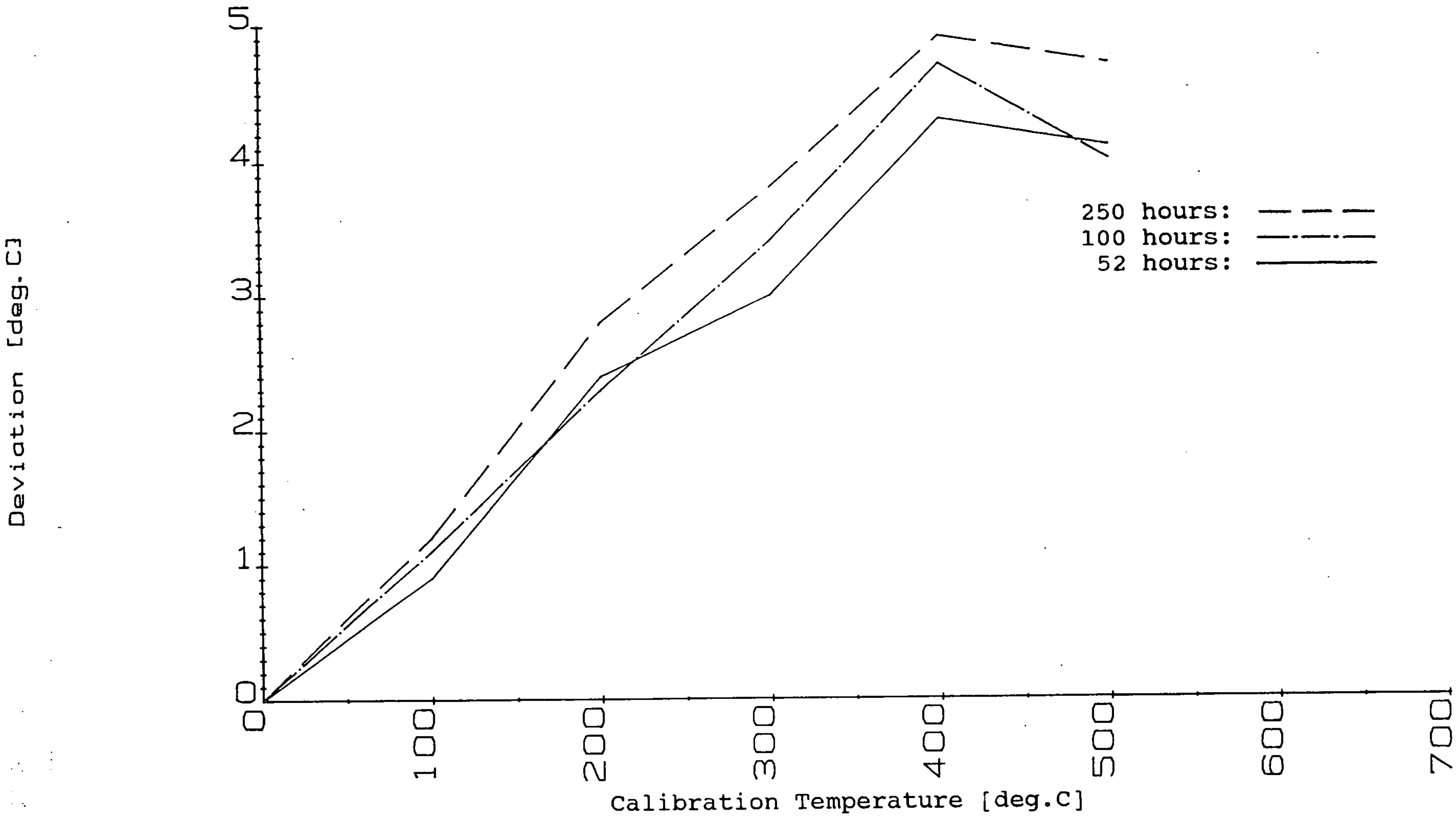


Figure 5.3 Calibration deviation from control, for preliminary preaging trials, July 1988. Calibration drift is apparent from a comparison of the plots.

introduces an additional aging factor. The purpose of the "control" thermocouple is to provide assurance of the integrity of that specific calibration run by providing a means of comparison with previously established calibration records. The control thermocouple and test are calibrated together in a single calibration run following the preaging procedures. The results of this calibration run are tabled in table 5.3(b).

Table 5.3(a) presents a comparison between the manufacturer's calibration , the Hulett Aluminium acceptance calibration (thermocouple S/N 1406), and the control thermocouple, (S/N 1418), calibration. The discrepancy between the manufacturer's calibration and the two Hulett Aluminium calibrations, as shown in table 5.3(a), is due to the different calibration techniques used. Both sets of calibration data are valid for the condition of the wire at the time of calibration. The Hulett calibrations capture the calibration status closer to the "as supplied" condition as they are conducted on the actual thermocouple, not on a sample. Figure 5.2 provides graphcal representation of these calibration data. Table 5.3(b) provides a listing of the calibration results for the control and four test thermocouples after preaging. The deviation values given in this table are relative to IPTS-1968. The differences between the control and the three duration test thermo-

Table 5.4 **Batch trial preaging details and calibration results.**

Thermocouple type: 3 mm diameter, stainless steel sleeved, mineral insulated, type K.

Serial numbers: 0195 to 0199

Lengths: 5.5 m, 6.8 m, 8.5 m, 10.5 m, and 15.5 m.

Preaging detail: 50 hours soak at 500°C (+/- 10°C).

The following sets of calibration data given in deviation from IPTS-68 in degrees celsius represent the highest, average, and lowest deviation values, at each of the indicated calibration temperatures:

Details	Temperature (°C) 100	200	300	400	500
Initial calibration					
highest:	0.2	-0.1	-0.5	-0.8	-0.7
average:	0.1	-0.2	-0.9	-1.3	-1.0
lowest :	-0.1	-0.4	-1.1	-1.8	-1.4
Relative spread:	0.3	0.3	0.6	1.0	0.7
Preaged calibration					
highest:		1.6	1.8	1.8	2.4
average:		1.3	1.6	1.6	2.1
lowest :		1.1	1.4	1.2	1.6
Relative spread:		0.5	0.4	0.6	0.8
Difference between initial and preaged ave:		1.5	2.5	2.9	3.1

NOTE: Values not recorded are not given on original records.

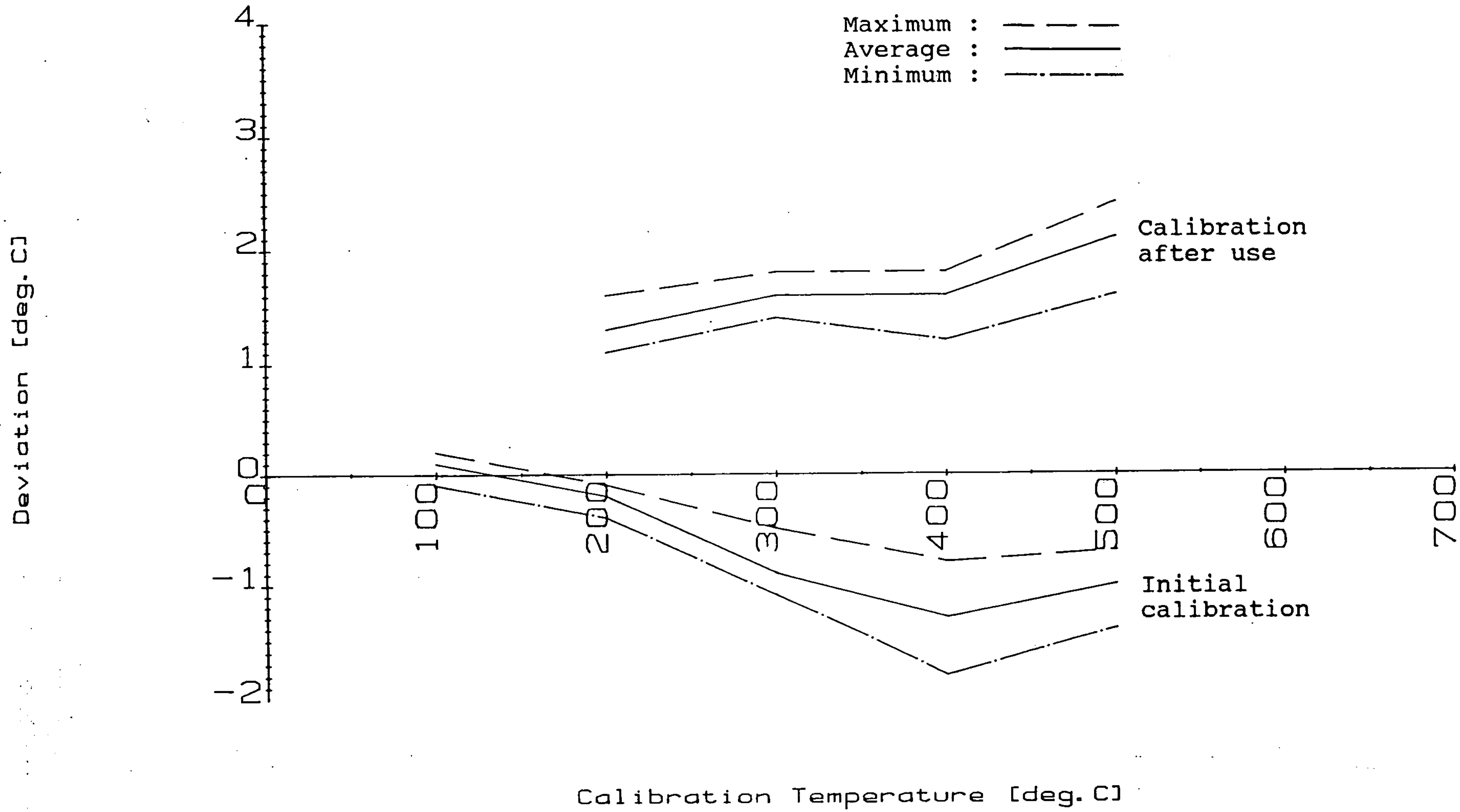


Figure 5.4 Batch trial calibration summary. Maximum, minimum, and average datum points plotted for initial and after use data sets.

couples, (S/N's 1421-3), are given in table 5.3(c) and graphically represented in figure 5.3. Consideration of the foregoing data provides indication that whilst there is a significant offset from initial calibration in all four preaged thermocouples, subsequent drift, although apparent, is limited (less than 1^{o}C between thermocouples).

c. **Batch Trial**

The batch trial, conducted on a batch of five, 3 mm diameter, MI, Type K thermcouples provides indication of the spread in calibration that may be expected across a batch of thermocouples, supplied from a common bulk source. The data pertaining to this trial is given in table 5.4. These data indicate that the initial spread, with a maximum difference of 1^{o}C across the batch, is contained within similar limits after preaging at 500^{o}C for 50 hours. Differences in calibration between the averages of each data set indicate comparable deviation performance with the 20 gauge bare thermocouple wire trials. Figure 5.4 provides graphical representation of these data with the high, average, and low limits of both the initial and preaged data sets plotted.

5.1.3 Conclusion

From the results of the preliminary aging trials the following conclusions are reached:

(i) Initial instability, (up to 250 hours), in Type K thermocouples, under the conditions considered, can be identified as attributable to two major factors. Short-form ordering is responsible for the substantial initial "step" deviation from the "as received" calibration status. Drift due to continued aging of the wire is revealed in a gradual positive shift in calibration.

(ii) Relatively short term preaging (less than 20 hours) is sufficient to generate a significant initial offset.

(iii) The characteristic positive drift in calibration, if proven to be consistent, could be compensated for by provision of an estimated correction factor added to the calibrated calibration offset.

(iv) Thermocouples from the same bulk source and preaged as a batch may have a common average calibration offset applied without significantly lowering the integrity of the calibration procedure (difference from average is within calibration uncertainty).

5.2 TRIAL PROCEDURE

5.2.1 Introduction

The aging trials evaluate two aspects of thermocouple aging. Firstly the initial effect on calibration that is effected through preaging is evaluated. Secondly the effect of aging under working conditions in a solution heat treatment furnace is considered. In determining the trial procedure primary consideration is given to the fact that this project aims to provide a practical solution to a current problem. Thus whilst the preliminary trials indicate extended preaging soak periods (greater than 250 hours) it is considered that this is impractical and as a consequence the trials concentrate on evaluating shorter preaging periods.

Table 5.5 Preaging trial thermocouple identification. Sets identified in (a) and (b) are manufactured from the same source material.

(a) Aging Furnace number 6 load thermocouple batches:

Trial A S/N	Trial B S/N	Length (m)
2535	2545	4.5
2536	2546	5.0
2537	2547	7.0
2538	2548	9.0
2539	2549	11.5
2540	2550	13.0

(b) Aging Furnace number 5 load thermocouple batches:

Trial A S/N	Trial B S/N	Length (m)
2541	2551	6.0
2542	2552	8.5
2543	2553	11.0
2544	2554	14.5

(c) Wellman Aging Furnace load thermocouple batch:

S/N	Length (m)
3058	10
3059	10
3060	10

5.2.2 Thermocouples

Thermocouples used in the trial are selected from normal batch specifications. In order to allow comparison of calibration results across batches the thermocouples of each batch originate from the same source material. Thermocouples are of the type: 3mm diameter, 316 stainless steel sheathed, mineral insulated, Type K with flexible compensation cable tail and male Type K compensation connector. Serial numbers and individual thermocouple lengths are indicated in table 5.5.

Four batches comprising a total of twenty thermocouples, manufactured from the same source material, are used in two preaging and work aging trial runs, designated "Trial A" and "Trial B". A further three thermocouples, from a different source material, are utilised in an additional work aging trial.

5.2.3 Preaging

A muffle furnace with a vertical depth of 390 mm is used to preage the test thermocouples. Temperature variation across the furnace is less than +/-10^{o}C and control stability is within +/-2^{o}C at the soak temperature.

The thermocouples being aged are immersed at the hot junction end to a depth of 390 mm, the remaining lengths of the thermocouples are at ambient temperature and receive no preaging. Calibration of the preaged thermo-

Table 5.6 Trial A, preaging schedule. The ten thermocouples used are divided into two sets according to their calibration grouping.

Calibration Set: 1

			Furnace Schedule										
S/N	Data Sheet	Aging	Mon.	Tue.	Wed.	Thur.	Fri.	Sat.	Sun.	Mon.	Tue.	Wed.	Thur.
2535	2086011289	10hr at $500^{\circ}C$	In: 08.00 Out: 18.00										
2536	2086021289	20hr at $500^{\circ}C$	In: 12.00	Out: 08.00									
2537	2086031289	40hr at $500^{\circ}C$	In: 16.00	-----	Out: 08.00								
2538	2086041289	80hr at $500^{\circ}C$	In: 08.00	-----	-----	Out: 16.00							
2539	2086051289	160hr at $500^{\circ}C$	In: 08.00	-----	-----	-----	--*--	-----	-----	-----	Out: 16.00		
2540	2086061289	Control (1)											

Calibration Set: 2

			Furnace Schedule										
S/N	Data Sheet	Aging	Mon.	Tue.	Wed.	Thur.	Fri.	Sat.	Sun.	Mon.	Tue.	Wed.	Thur.
2541	4476011289	10hr at $600^{\circ}C$										In: 08.00 Out: 18.00	
2542	4476021289	20hr at $600^{\circ}C$									In: 17.00	Out: 13.00	
2543	4476031289	40hr at $600^{\circ}C$									In: 17.00	-----	Out: 09.00
2544	4476041289	Control (2)											

(*) Furnace temperature dropped to $400^{\circ}C$ over a period of 30 min due to a power failure.

couples is carried out at a reference depth of 300 mm (effective immersion depth is 240 mm) thereby ensuring that the preaged portion of the thermocouple is calibrated.

The preaging schedule, listing the soak periods and temperatures for the respective thermocouples, for Trial A is given in table 5.6. This table also identifies the calibration grouping of the thermocouples. Two soak temperatures, 500 and 600°C, are selected for Trial A in order to ascertain whether an accelerated aging effect is achieved at the higher temperature. The soak periods selected at the 500°C soak temperature are intended to provide comprehensive indication of the degree of drift occuring at this temperature.

5.2.4 Calibration

With each calibration run an, "as received", control thermocouple is included in the calibration in order to provide assurance of the comparable calibration conditions produced during that run. The degree of correlation between the calibration results of the control thermocouples is indicative of the confidence that may be placed on an intercomparison of the results.

5.2.5 Procedure

The procedure employed for both Trial A and Trial B is as follows:

Hulett Aluminium

FURNACE: AF5

4	4	7	6	0	1	0	1	9	0

DATA SHEET NUMBER

Cal 10201

recal 10216

LOAD THERMOCOUPLE

WORK LOG SHEET

Identification: S/N 2541 - 2544

Temperature	200°C	300°C	400°C	500°C
THERMOCOUPLE CORRECTION FACTOR	—	—	—	—

TTL CONTROL 01

CONTROL:

DATE	BATCH / TIME	TEMP.	HOURS	REMARKS
29/01	T332	465±5	2Hrs ..min	S.H.T
01/02	H6B	465°±5°	3Hrs 45min	S.H.T
08/02	H720E	520°±5°	1Hrs 55.	S.H.T
14/02	Ref S/Mill	465 +2/−3	4Hr 10Min	S.H.T
21/02/90	T205	465°±5°	2Hrs 25min	S.H.T.
22/02/90	T314	465°±5°	3Hrs 15	S.H.T.
22/02/90	T335	520°±5	1Hr 40	S.H.T
26.02.90	T147P	465±5	3Hrs 25min	SHT.
26.02.90	T147A	465±5	3 Hrs 30min	SHT.
26.02.90	T147B	465°±5°	3Hrs 10min	S.H.T.
27.02.90	T345A	520±5	3 Hrs 35min	SHT.
28.02.90	T345B	520±5°	2 Hrs 50min	SHT.
01.03.90	Ref S/Mill	465 +5/−2	5 Hrs	SHT.
02.03.90	"	465 +5/−2	5 Hrs 15min	SHT.
02.03	Ref S/Mill	465° +5°/−3	5 Hrs 30mins	S.H.T

DATE	BATCH / TIME	TEMP.	HOURS	REMARKS

Figure 5.5 Load Thermocouple Work Log Sheet, example of work record for Trial A, Aging Furnace 5, thermocouples.

(i) Identification of thermocouples.

(ii) Preaging according to schedule.

(iii) Calibration of preaged thermocouples.

(iv) Thermocouples issued for work trials.

(v) Thermocouples retrieved and recalibrated.

(vi) Total calibration offset and offset attributable to work trials tabled for each calibration temperature.

5.2.6 Work Trials

Difficulty is experienced in determining the extent of aging that a thermocouple undergoes during the working period. This can be attributed to the three main factors:

(i) Several sets of thermocouples are available at each furnace. Total furnace work load may be shared unequally between these sets.

(ii) During the "time up to temperature" thermocouples are not at a constant temperature.

(iii) Soak temperatures vary from load to load.

Due to these factors only an estimation of the extent of aging is ascertainable. In order to ensure that this estimation is confined within acceptable parameters a system is provided requiring the furnace operator to record details of load thermocouple usage on a log sheet. This log sheet, the "Load Thermocouple Work Log Sheet" (figure 5.5), provides a record of the soak temperature and period as well as indication of the number of quench cycles. Evaluation from this record disregards time spent coming up to temperature and for practical consideration all temperatures above 450^{o}C and below 550^{o}C are considered in common.

5.3 SUMMARY

The procedures scheduled for Trial A are determined directly through consideration of the preliminary aging trials. At the stage of Trial A the intention is to determine:

(i) A suitable preaging temperature ($500^{o}C$ or $600^{o}C$).

(ii) The necessary soak time to ensure consistent results.

(iii) The extent of continued drift after preaging due to work induced aging.

The procedure for Trial B is determined by the result of Trial A and is consequently presented following the discussion of the Trial A initial calibration results. In both trials the work trial component provides data for a statistical evaluation of the projected calibration uncertainty. The three additional "Wellman furnace" load thermocouples are employed following Trial B in a full application trial that tests the hypotheses under operational conditions.

CHAPTER 6

THE RESULTS: PREAGING

6.1 INTRODUCTION

6.1.1 The Data

Each thermocouple calibration comprises eleven datum points across the range 50 to 550°C. Each datum point indicates the deviation of the respective thermocouple's calibration from IPTS-68 at a specific calibration temperature. Deviation values are commonly quoted either in microvolts, which relate directly to the IPTS-68 tables, or in degrees Celsius. For convenience of comparison of deviation values, degrees Celsius, expressed to one decimal place, are used throughout the presentation of the results.

6.1.2 Manner of Presentation

Because of the difficulty in evaluating the non-linear calibration trends, two means of presenting the data are utilised:

(i) The full data set for a specific calibration run is presented in tabular format enabling precise evaluation of these data, as required.

(ii) Graphical representation is provided of specific calibration trends enabling visual intercomparison of the selected plots.

Each calibration run is treated individually according to the above means of presentation prior to crosscorrelating the data between other calibration runs. Discussion concerning the evaluation of the data follows each presentation.

In graphical representations the following conventions have, unless otherwise indicated, been applied:

(i) Calibration data points for initial calibration of a thermocouple in a specific condition ("as received", preaged or after use), are identified by the marker, "0". Calibration data points plotted on the same graph, for each recalibration are identified by successive numerical value markers, "1", "2", "3".

(ii) Where data from several calibration test runs, or different thermocouples, are overlaid on a common frame, identifiers (test number, calibration date, serial number, Type, certificate number), shall refer to the thermocouple or calibration being referenced.

(iii) Where calibration data from a batch of thermocouples is overlaid on a common frame, individual calibration plots are identified by successive numerical markers. Marker "1" indicates the plot of the thermocouple with the lowest serial number. Graph identifiers refer to the same thermocouple.

(iv) Manufacturers specification limit plots, for standard and special grade thermocouples, are included on each graph as additional lines of reference.

Table 6.1 Calibration test number 10200. Thermocouple identification and results.

(a) Thermocouple identification:

Thermocouple Serial No.	Conditions pertaining to calibration
2535	Preaged 10 hours at 500°C
2536	Preaged 20 hours at 500°C
2537	Preaged 40 hours at 500°C
2538	Preaged 80 hours at 500°C
2539	Preaged 160 hours at 500°C
2540	Control: as received

(b) Results. Deviation from standard in degrees Celsius:

	Temperature of standard (°C)										
S/n.	50	100	150	200	250	300	350	400	450	500	550
2535	0.4	0.7	1.4	1.4	1.5	1.8	1.8	1.8	2.0	2.2	2.4
2536	0.4	0.6	1.4	1.4	1.5	1.8	1.8	1.9	2.0	2.2	2.4
2537	0.4	0.6	1.4	1.3	1.5	1.7	1.7	1.8	2.0	2.2	2.4
2538	0.4	0.7	1.4	1.4	1.5	1.8	1.8	1.9	2.1	2.4	2.6
2539	0.4	0.6	1.4	1.3	1.5	1.7	1.7	1.7	1.9	2.2	2.4
2540	0.2	0.1	0.6	0.1	-0.2	-0.4	-0.7	-0.9	-0.6	-0.5	-0.2

(c) Maximum variation between preaged thermocouples:

Temperature of standard (°C)										
50	100	150	200	250	300	350	400	450	500	550
0.0	0.1	0.0	0.1	0.0	0.1	0.1	0.2	0.2	0.2	0.2

(d) Variation between preaged thermocouples averaged deviations and equivalent control thermocouple deviation:

Temperature of standard (°C)										
50	100	150	200	250	300	350	400	450	500	550
0.2	0.5	0.8	1.3	1.7	2.2	2.5	2.7	2.6	2.7	2.6

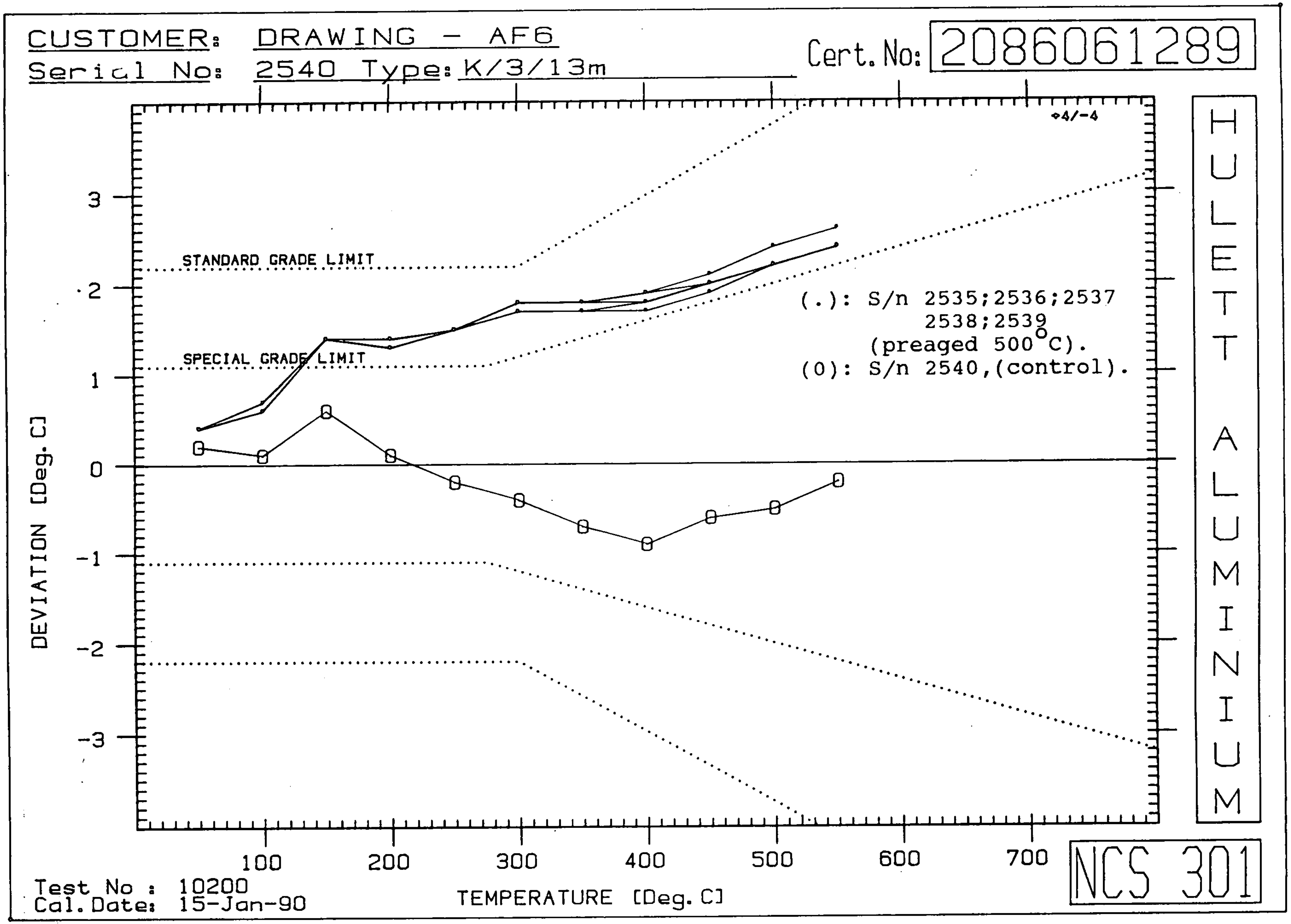


Figure 6.1 Calibration test number 10200. Plots show thermocouple calibration deviation from standard.

6.2 RESULTS

6.2.1 Trial A: Preaging Calibration

a. Calibration Test: 10200

Calibration results are tabulated in table 6.1. From these results the following observations are made:

1. Drift

Contrary to expectation no significant age related drift is discernable between the five thermocouples representing aging times from 10 to 160 hours. Differences between preaged thermocouples, across the temperature range, are no greater than 0.2°C [table 6.1(c)].

2. Offset

Offset from "as received" calibration followed the anticipated pattern varying from 0.2°C at 50°C, to 2.6°C at 550°C [table 6.1(d)]. Between 400 and 550°C offset is constant within 0.1°C of the 550°C value (figure 6.2).

3. Graphical comparison

Figure 6.1 provides graphical comparison of the calibration results for preaged thermocouples S/N's 2535 to 2539 and control thermocouple

Table 6.2 Calibration test number 10201. Thermocouple identification and results.

(a) Thermocouple identification:

Thermocouple Serial No.	Conditions pertaining to calibration
2541	Preaged 10 hours at 600°C
2542	Preaged 20 hours at 600°C
2543	Preaged 40 hours at 600°C
2544	Control(2): as received
2540	Control(1): first recalibration

(b) Results. Deviation from standard in degrees Celsius:

	Temperature of standard (°C)										
S/n.	50	100	150	200	250	300	350	400	450	500	550
2541	0.0	0.3	0.8	0.4	0.4	0.2	-0.1	-0.2	-0.1	0.1	0.1
2542	0.0	0.2	0.9	0.5	0.5	0.2	0.0	-0.2	-0.1	0.1	0.2
2543	0.0	0.3	0.9	0.4	0.5	0.2	0.0	-0.2	-0.2	0.1	0.2
2544	0.0	0.2	0.6	0.0	-0.1	-0.5	-0.8	-0.5	-0.2	0.1	0.0
2540	0.1	0.5	1.2	1.0	1.1	1.0	1.0	0.9	1.2	1.4	1.5

(c) Maximum variation between preaged thermocouples:

Temperature of standard (°C)										
50	100	150	200	250	300	350	400	450	500	550
0.0	0.1	0.1	0.1	0.1	0.0	0.1	0.0	0.1	0.0	0.1

(d) Variation between preaged thermocouples averaged deviations and equivalent control thermocouple deviation:

Temperature of standard (°C)										
50	100	150	200	250	300	350	400	450	500	550
0.0	0.1	0.3	0.4	0.6	0.7	0.8	0.3	0.1	0.0	0.2

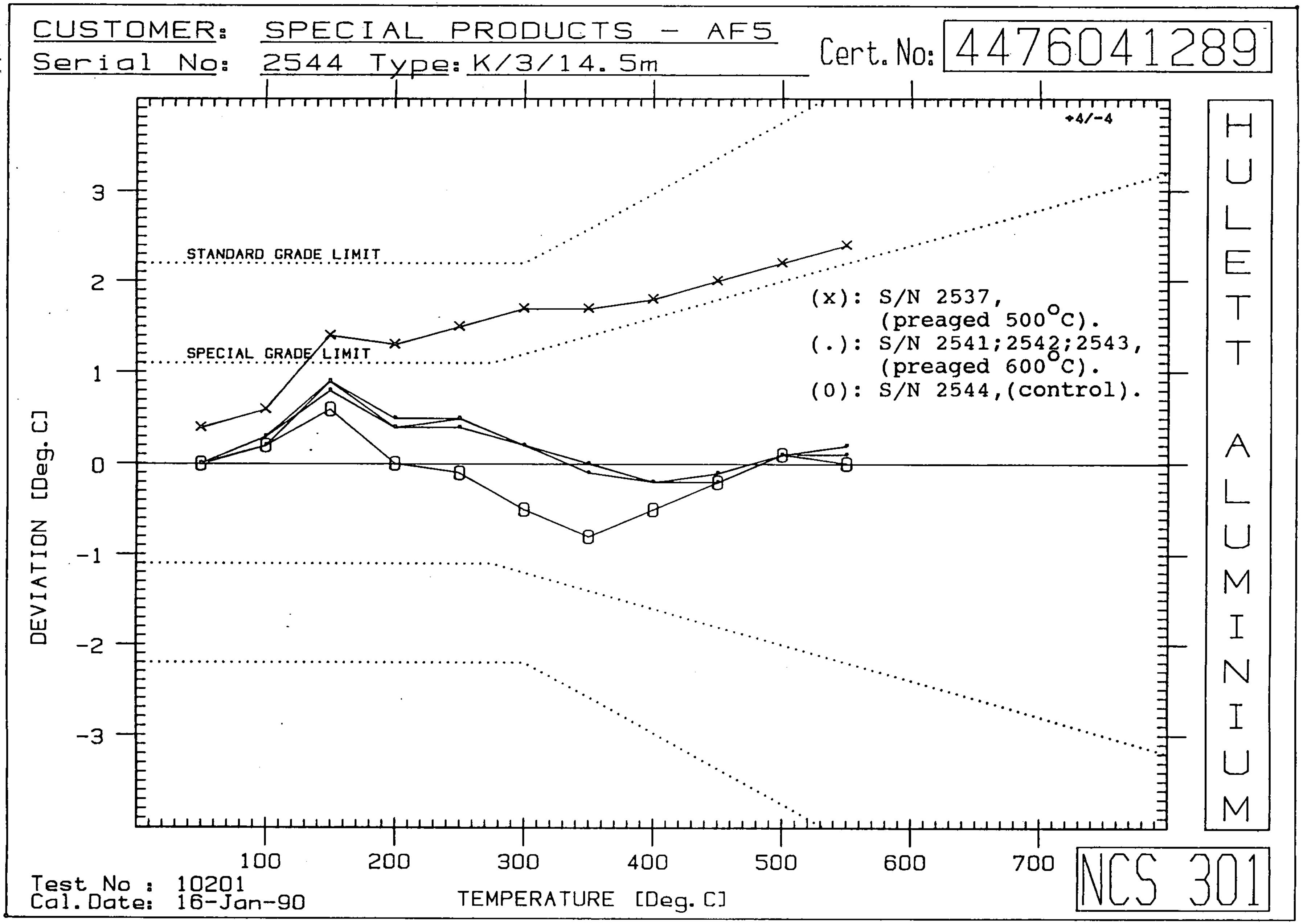


Figure 6.2 Calibration test number 10201. Plots show thermocouple calibration deviation from standard. Data for S/N 2537 is from test number 10200.

S/N 2540. Because of the overlapping of the preaged thermocouple plots, no individual identification is given. The control thermocouple plot is identified by the reference "0" markers.

b. Calibration Test: 10201

Calibration results are tabulated in table 6.2(b). Control thermocouple (1), S/N 2540 was carried over from test 10200 to receive its first recalibration.

1. Drift

As with the previous calibration, no age related drift is discernable between the preaged thermocouples. Differences between the calibrations of the three thermocouples aged for periods of 10, 20 and 40 hours respectively at $600^{o}C$, do not exceed $0.2^{o}C$ [table 6.2(c)].

2. Offset

Offset of the $600^{o}C$ preaged thermocouples does not follow the expected positive increase relative to the $500^{o}C$ preaged thermocouples. At each calibration temperature, offset deviation, although positive, is less than for the equivalent $500^{o}C$ preaged thermocouples. The only significant (greater than $0.5^{o}C$) deviation from control(2) occurs between 200 and $400^{o}C$ [table 6.2(d)], and is limited to a maximum of $0.8^{o}C$.

Table 6.3 Deviation between control thermocouples (1) and (2). Deviation in degrees Celsius.

Calibration Temperature °C										
50	100	150	200	250	300	350	400	450	500	550
0.2	0.1	0.0	0.1	0.1	0.1	0.1	**0.4**	**0.4**	**0.6**	0.2

Table 6.4 Calibration response times for tests 10200 and 10201. Excessive calibration stabilising period at 400°C in test 10201 is highlighted in bold print. Response times in minutes per interval.

Interval °C	Response Time 10202	10201
50 to 100	57	46
100 to 150	57	29
150 to 200	33	38
200 to 250	69	38
250 to 300	54	51
300 to 350	52	56
350 to 400	37	**94**
400 to 450	46	53
450 to 500	39	54
500 to 550	54	47
Average:	54	51
Sx:	11	17

NOTE: Because of critical nature of these tests LAB87 stability control parameter, 3Sx for thermocouples under test, is set at 0.4°C (equal to the standard). This parameter is responsible for the average response times exceeding the 45 minute target.

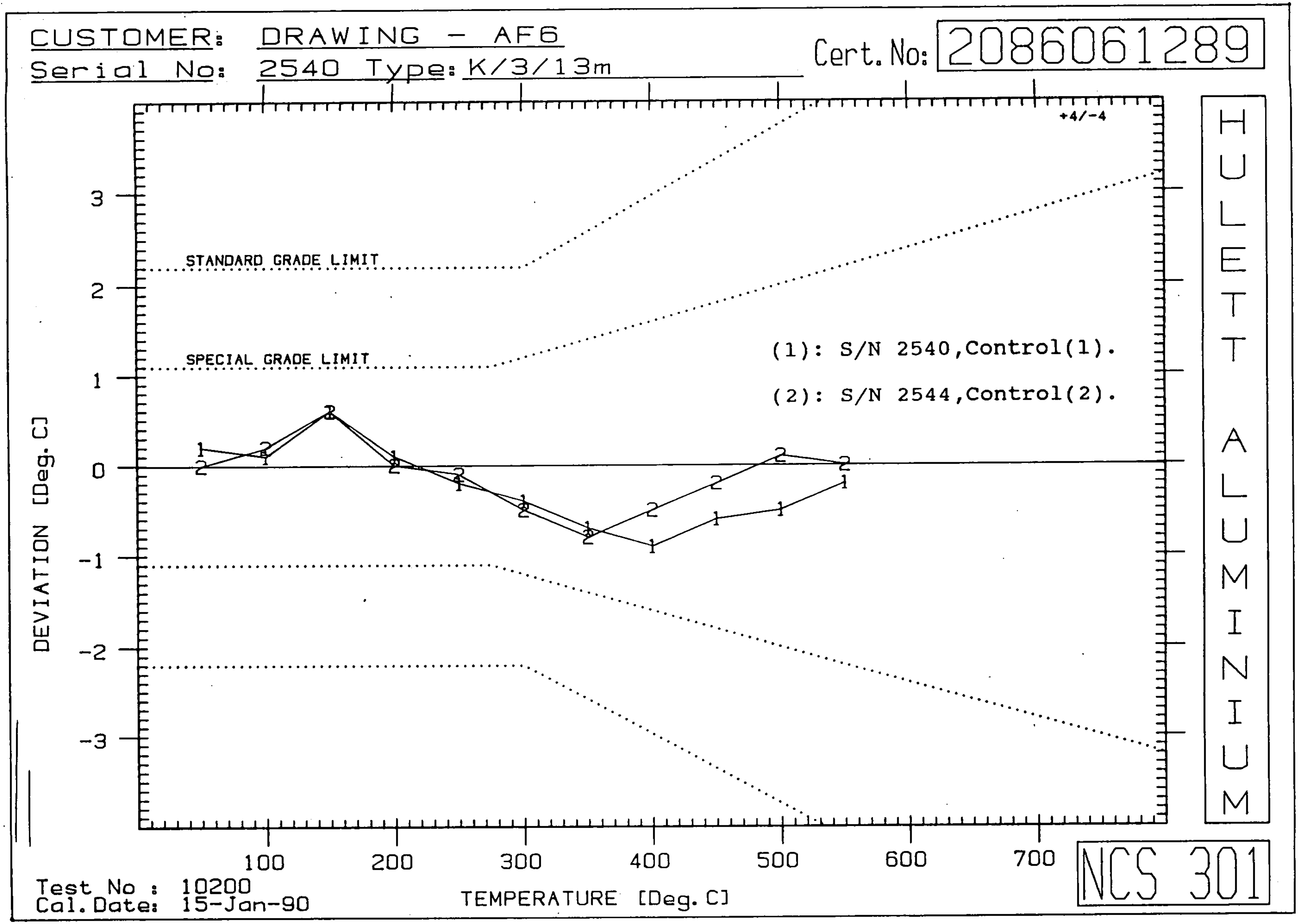


Figure 6.3 Control thermocouple comparison. Control(1) from test 10200, control(2) from test 10201.

3. **Graphical Comparison**

Figure 6.2 provides graphical comparison of the calibration results for the preaged thermocouples S/N's 2541 to 2543 and control(2) thermocouple, S/N 2544. Control(2) plot is identified by the reference "0" markers. Calibration plot for thermocouple S/N 2537 from the previous calibration run is included for comparison and is identified by the marker, "x".

4. **Control Comparison**

Figure 6.3 provides comparison of the, "as received", calibrations of the two control thermocouples. Control thermocouples (1) and (2) are identified by the markers, "1" and "2", respectively. Deviation between the two calibrations is indicated in table 6.3. From $50^{o}C$ through $350^{o}C$ correlation between the two calibrations is within $0.2^{o}C$. At $500^{o}C$ correlation ($0.6^{o}C$) falls outside of the acceptable confidence limit of $0.5^{o}C$. A comparison of the calibration response times for the calibration runs 10200 and 10201 (table 6.4), reveals an excessive calibration stabilising period at $400^{o}C$ (94 minutes). This excessive stabilising period accounts for a lowering of the effective calibration depth (due to additional loss of fluidising powder). The consequential difference in calibration is due to the lower calibration

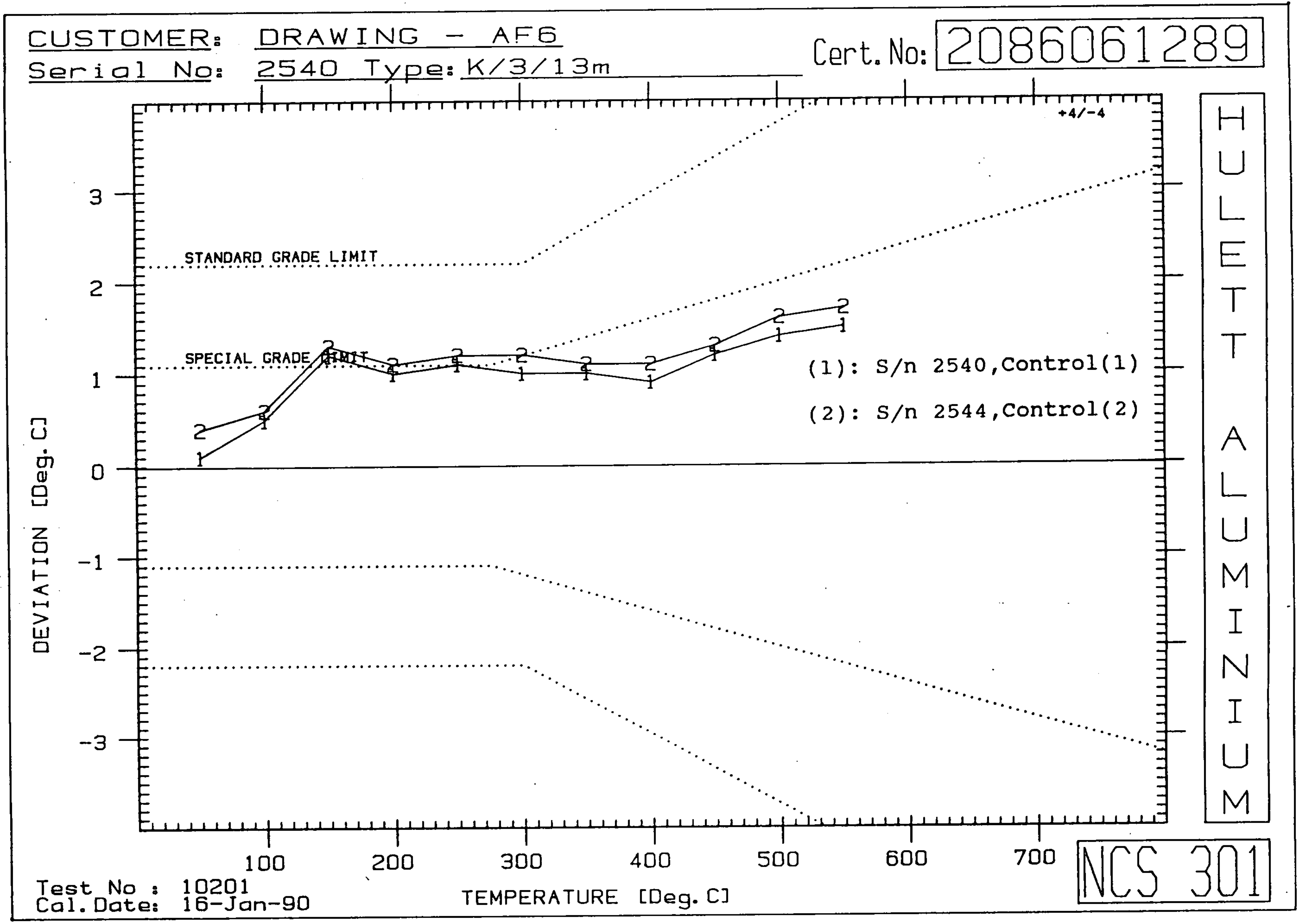


Figure 6.4 First recalibration comparison of control thermocouples (1), from test 10201, and (2), from test 10202.

point causing the main thermal gradient to include an area of thermocouple that has been exposed to an additional element of aging. From these considerations, the following conclusions concerning the integrity of the second calibration run (10201), are drawn:

(i) From 50°C to 350°C the data is valid within the project's stated confidence limit.

(ii) The effect of the lowered calibration point is expected to be less on the preaged thermocouples than on the "as received" control thermocouple. This consideration is supported by a comparison of the subsequent recalibration of control(2) in test 10202, to the first recalibration of control(1) during the test in question (10201). In this comparison (figure 6.4), deviation between the two plots does not exceed 0.3°C, (0.2°C above 350°C).

(iii) Any effect on the preaged thermocouples would be in a positive direction. Thus uncertainty of measurement of all calibration recordings above 350°C in test 10201 is increased to +0.5/-1.0°C. Uncertainty of comparisons between measurements would, to a degree be self correcting, and are considered adequately covered by the +-0.5°C uncertainty specification.

The increased uncertainty of measurement effected above does not narrow the margin between the calibration results achieved from the two preaging temperatures. Any adjustment would widen the margin and it is considered reasonable within the stated limits of error to accept the calibrations as they stand.

c. **Conclusion**

From consideration of the results of calibration test runs numbers, 10200 and 10201, the following conclusions are drawn:

1. **Drift**

No significant time related drift is discernable at up to 160 hours aging at 500^{o}C or 40 hours at 600^{o}C. It is therefore concluded that preaging times exceeding 200 hours would be neccessary to induce time related drift.

2. **Offset**

Preaging at 500^{o}C creates the expected (+3^{o}C,+/-1^{o}C), offset in calibration due to the effects of short-range ordering. The ordering effect, which occurs in the temperature range 320 to 540^{o}C, is removed when the thermocouple is heated above 600^{o}C (1990 Annual Book of ASTM Standards, Volume 14.03, Standard E 839, Appendix X1, par. X1.2.1), accounting for the lesser offset derived from the 600^{o}C preaging trials. It is therefore concluded that preaging temperature should be between 500 and 540^{o}C.

6.2.2 Trial B

a. Preaging schedule

The preaging schedule for Trial B is arranged to confirm and extend the findings of Trial A. Four thermocouples (S/N's 2545 to 2548), are used for preaging at 500°C, the first is given a one hour soak, which is at this stage considered to be the recommended practice. A second thermocouple is removed after one hour, allowed to cool to ambient, and then taken up to 500°C for a further hour in order to evalute the influence of limited cycling. The same procedures are carried out on a further two thermocouples with the soak periods extended to ten hours.

Two thermocouples (S/N's 2549 and 2550) are preaged at 600°C, for one and eight hours respectively. One thermocouple (S/N 2551) is preaged at 700°C for three hours; this thermocouple is, however, not calibrated after preaging. Thermocouple identification and preaging practices are detailed in table 6.5(a).

Three thermocouples (S/N's 2552 to 2554) are retained for use as control thermocouples numbers (3),(4) and (5) respectively. Control(3) is used in the calibration of the above preaging trial and controls (4) and (5) are used in the recalibration of Trial A thermocouples returned from service.

Table 6.5 **Calibration test number 10213. Thermocouple identification and results.**

(a) Thermocouple identification:

Thermocouple Serial No.	Conditions pertaining to calibration
2545	Preaged 1 hr at 500°C
2546	Preaged 1 hr at 500°C/ambient/ 1 hr at 500°C
2547	Preaged 10 hr at 500°C
2548	Preaged 10 hr at 500°C/ambient/10 hr at 500°C
2549	Preaged 1 hr at 600°C
2550	Preaged 8 hr at 600°C
2551	Preaged 3 hr at 700°C: **NOT CALIBRATED**
2552	Control(3): as received
2553	Control(4): used as control in test 10215
2554	Control(5): used as control in test 10216

(b) Results. Deviation from standard in degrees Celsius:

	Temperature of standard (°C)										
S/n.	50	100	150	200	250	300	350	400	450	500	550
2545	0.3	0.6	1.5	1.4	1.5	1.6	1.6	1.8	2.0	2.3	2.3
2546	0.3	0.7	1.5	1.4	1.6	1.7	1.6	1.7	1.8	2.3	2.4
2547	0.3	0.6	1.5	1.3	1.5	1.7	1.7	1.8	2.0	2.4	2.5
2548	0.3	0.6	1.4	1.3	1.5	1.7	1.6	1.8	2.0	2.3	2.4
2549	0.2	0.3	0.8	0.3	0.3	0.1	-0.2	-0.2	-0.2	0.2	0.3
2550	0.2	0.3	0.8	0.5	0.1	-0.1	-0.3	-0.5	0.0	0.3	0.5
2552	0.1	0.1	0.6	0.1	-0.1	-0.3	-0.8	-0.9	-0.8	-0.5	-0.5

Table 6.6 Analysis of calibration test number 10213 results.

(a) Maximum variation between 500°C preaged thermocouples:

Temperature of standard (°C)										
50	100	150	200	250	300	350	400	450	500	550
0.0	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.2	0.1	0.2

(b) Maximum variation from average of all nine, 500°C preaged thermocouples, in calibration tests; 10200 and 10213:

Temperature of standard (°C)										
50	100	150	200	250	300	350	400	450	500	550
0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.2	0.1	0.2

(c) Variation between control thermocouples (1) and (3) from calibration tests 10200 and 10213 respectively:

Temperature of standard (°C)										
50	100	150	200	250	300	350	400	450	500	550
0.1	0.0	0.0	0.0	0.1	0.1	0.1	0.1	0.2	0.0	0.3

b. **Calibration Test: 10213**

Calibration results for Trial B, initial calibration after preaging, are tabulated in table 6.5. From these results the following observations are made:

1. **Preaged at 500°C**

   Calibrations of the four thermocouples preaged at 500°C concurred with the previous trial's results. Calibration results indicate that the offset induced is repeatable and is not significantly influenced by soak time nor by limited cycling. Table 6.6(a) shows the correlation between calibration values at the respective calibration temperatures. Table 6.6(b) shows the correlation between all thermocouples preaged at 500°C in both Trial A and B, covering a range of soak periods from 1 hour to 160 hours. Out of 99 calibration measurements, only two varied from the respective averages by more than 0.1°C (in both cases the variation is 0.2°C).

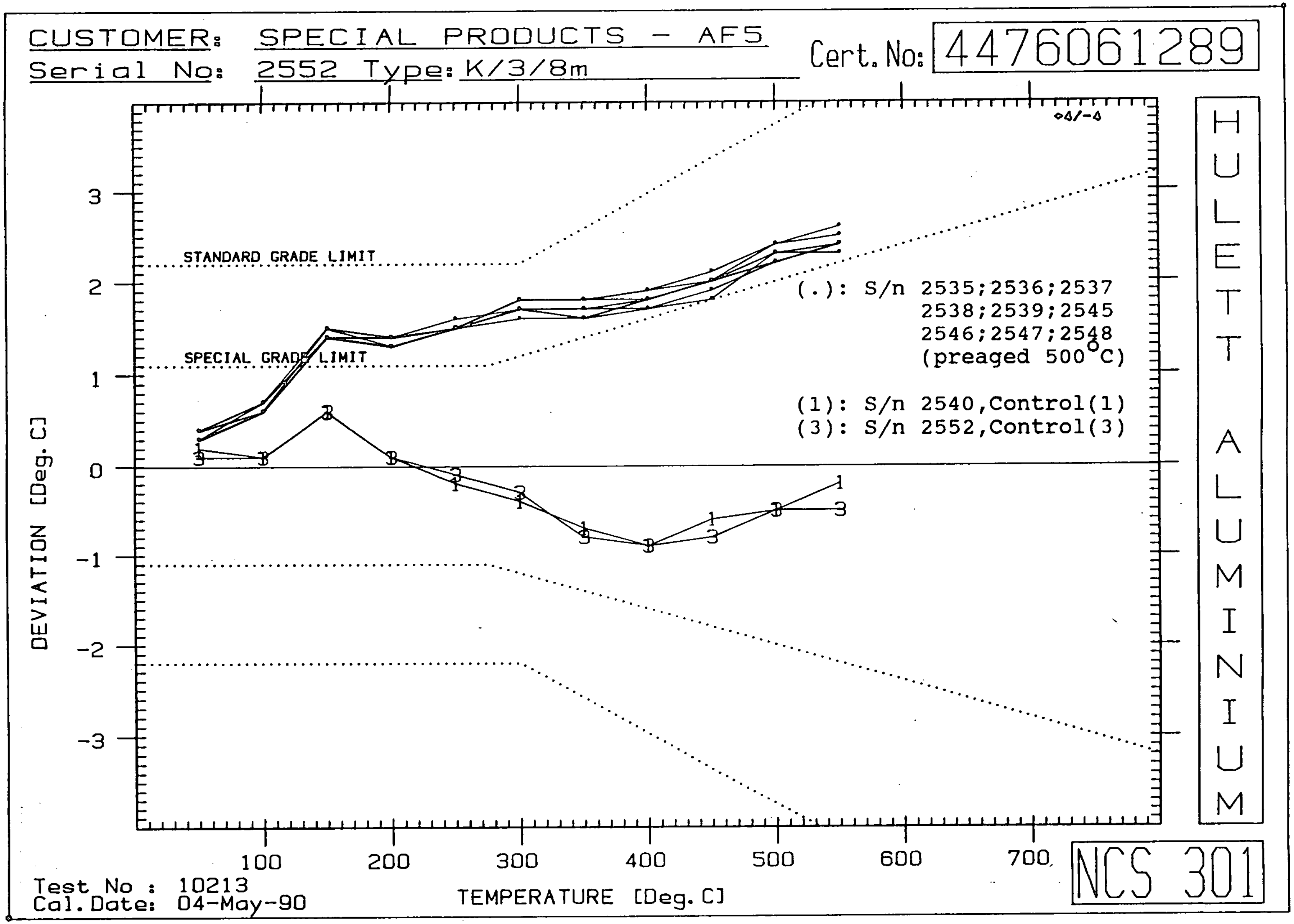


Figure 6.5 Calibration test number 10213. Composite overlay of all 500°C preaged thermocouple calibrations from tests 10200 and 10213. Control thermocouples (1) and (3) from the same tests respectively, are included.

2. **Preaged at 600^{o}C**

Calibration for the two thermocouples preaged at 600^{o}C follows a similar trend to the equivalent test in Trial A, confirming the earlier conclusions given concerning the effect of aging at 600^{o}C. No further consideration is given to preaging at this temperature.

3. **Control Comparison**

Table 6.6(c) shows the correlation between control thermocouples (1): test 10200, and (3): test 10213. The greatest difference between the two control thermocouples does not exceed 0.3^{o}C and the correlation is seen to represent a satisfactory calibration run with results acceptable within the specified confidence limits.

4. **Graphical Comparison**

Figure 6.5 provides a composite graphical overlay of the calibrations of all 500^{o}C preaged thermocouples (9 off), together with control(1) and control(3) calibrations overlaid.

6.3 SUMMARY

6.3.1 Preaging: Conclusion

a. Preaging Practice

Results of the two preaging trials indicate that a preaging soak period of one hour at a temperature of 500°C provides a satisfactory practice for the preaging of Type K thermocouples for use in an aluminium heat treatment furnace.

b. Effect of Preaging

Preaging according to the above practice creates short-range ordering within the thermocouple resulting in a positive offset in calibration.

c. Repeatability

Offset in calibration is repeatable on thermocouples manufactured from the same source material batch.

d. Contra-indication

The above preaging technique is not advised where thermocouple usage is:

(i) to exceed 550°C

(ii) to not exceed 300°C.

6.3.2 Calibration Offset

In preparation for the work aging trials the apportioning of calibration offsets is given (table 6.7) for the relevant thermocouples. In apportioning the offsets, due regard is given to the likelihood of further drift, as well as to the risk of extending work soak times through application of too great an offset. The following guide is used:

(i) Below $300^{o}C$ the first decimal place up to and including ".7", in calibration values, is rounded down in providing the equivalent offset.

(ii) From $300^{o}C$ up the first decimal place, up to and including ".7", in calibration values, is rounded up in providing the equivalent offset.

(iii) For calibrations of thermocouple batches the average calibration value is used to determine the offset.

Table 6.7 Apportioning calibration offset prior to use. Calibration deviation from standard, for 500°C preaged thermocouples is shown below together with the apportioned correction factors.

S/n.	50	100	150	200	250	300	350	400	450	500	550
	Temperature of standard (°C)										
2535	0.4	0.7	1.4	1.4	1.5	1.8	1.8	1.8	2.0	2.2	2.4
2536	0.4	0.6	1.4	1.4	1.5	1.8	1.8	1.9	2.0	2.2	2.4
2537	0.4	0.6	1.4	1.3	1.5	1.7	1.7	1.8	2.0	2.2	2.4
2538	0.4	0.7	1.4	1.4	1.5	1.8	1.8	1.9	2.1	2.4	2.6
2539	0.4	0.6	1.4	1.3	1.5	1.7	1.7	1.7	1.9	2.2	2.4
2545	0.3	0.6	1.5	1.4	1.5	1.6	1.6	1.8	2.0	2.3	2.3
2546	0.3	0.7	1.5	1.4	1.6	1.7	1.6	1.7	1.8	2.3	2.4
2547	0.3	0.6	1.5	1.3	1.5	1.7	1.7	1.8	2.0	2.4	2.5
2548	0.3	0.6	1.4	1.3	1.5	1.7	1.6	1.8	2.0	2.3	2.4
OFFSET:	0°C		-1°C			-2°C					

CHAPTER 7

THE RESULTS: CALIBRATION COMPENSATION

7.1 INTRODUCTION

Calibration compensation is effected through application of calibration correction factors to furnace instrumentation. Because correction is not a linear function, but varies across the temperature range, it is seldom possible to make the correction directly to the furnace instrumentation. Correction is thus typically applied manually through the use of correction tables or lists. The relevance of calibration compensation is considered through the comparison of three calibration data sets:

(i) Initial calibration ("as received" condition).

(ii) Preaged calibration (from which the correction factor is determined).

(iii) Calibration after use.

These comparisons provide indication of the extent of error occuring under normal conditions of use, and of the degree to which this error may be limited, through use of the preaging technique and application of calibration compensation.

Table 7.1 Work aged calibration results for trial A. Results from calibration tests 10215 and 10216.

(a) Thermocouple identification:

Thermocouple Serial No.	Conditions pertaining to aging: Range of soak temp.: 465 to 520°C	
Test 10215	Hours	Work cycles
2535	30	14
2536	189	82
2537	189	82
2538	189	82
2539	189	82
2540	30	14
Test 10216		
2541	46	15
2542	46	15
2543	46	15
2544	46	15
2553	Control(4): as received. Test 10215.	
2554	Control(5): as received. Test 10216.	

(b) Results. Deviation from standard in degrees Celsius:

	Temperature of standard (°C)										
S/n.	50	100	150	200	250	300	350	400	450	500	550
2535	0.4	0.7	1.5	1.6	1.9	2.0	2.1	2.2	2.6	2.8	3.1
2536	0.2	0.8	1.6	1.6	1.7	1.9	2.0	2.1	2.3	2.7	2.8
2537	0.2	0.7	1.6	1.5	1.8	1.8	1.9	1.9	2.2	2.6	2.7
2538	0.3	0.6	1.4	1.3	1.4	1.5	1.5	1.5	1.9	2.2	2.2
2539	0.2	0.9	1.7	1.6	1.9	2.0	2.1	2.2	2.3	2.7	2.9
2540	0.2	0.7	1.6	1.6	1.7	1.9	2.0	2.2	2.5	2.9	3.0
2541	0.2	0.8	1.8	1.8	2.0	2.0	2.1	2.2	2.6	3.0	3.1
2542	0.1	0.7	1.7	1.6	1.8	1.9	2.0	2.1	2.5	3.0	3.1
2543	0.2	0.8	1.7	1.7	2.0	2.1	2.1	2.2	2.5	2.9	3.1
2544	0.2	0.6	1.5	1.5	1.8	1.7	1.8	1.9	2.6	3.1	3.2
Ave:	0.2	0.7	1.6	1.6	1.8	1.9	2.0	2.1	2.4	2.8	2.9
3Sx:	0.2	0.3	0.4	0.4	0.5	0.5	0.6	0.7	0.7	0.8	0.9
2553	0.0	0.2	0.6	0.1	-0.2	-0.5	-0.8	-1.0	-0.8	-0.6	-0.4
2554	0.0	0.1	0.6	0.1	-0.3	-0.6	-0.9	-1.1	-0.8	-0.5	-0.3

7.2 WORK AGING PRACTICE

For work aging trials the four sets of thermocouples making up the twenty thermocouples alloted to Trials A and B, are issued for process use as normal load thermocouples. Recording of the number of work cycles, together with the soak times are recorded, as standard practice, on the Load Thermocouple Work Log Sheet. Thermocouples sets are returned from use when any one thermocouple of the set fails.

7.3 RESULTS

7.3.1 Trial A: Work Aged Calibration

a. Condition of Thermocouples

Five of the ten test thermocouples returned from service damaged. In Aging Furnace number 6 (AF 6), load thermocouple batch two thermocouples snagged and snapped whilst withdrawing a load from the furnace. These thermocouples were removed (after 30 hours service) and the remainder of the batch continued to complete 189 hours of service. In Aging Furnace number 5, (AF 5), an aluminium plate buckled severely on quenching, shearing three of the four load thermocouples batch after 46 hours of service.

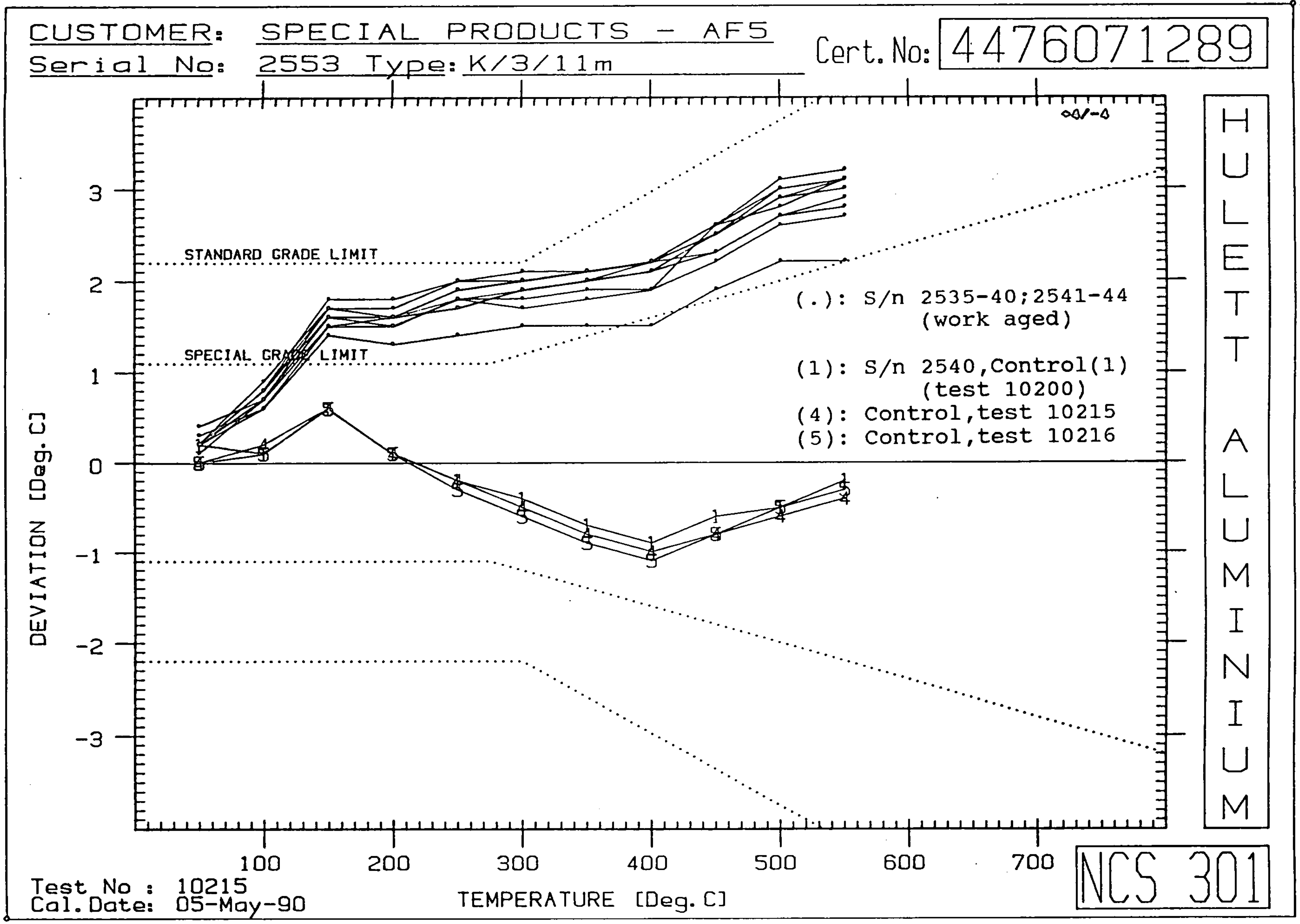


Figure 7.1 Calibration tests numbers 10215 and 10216. Composite overlay of all thermocouple calibrations after work aging. Control thermocouples couples (4) and (5) shown in comparison to control(1) from test 10200.

Recovery of all sections of the critical sections of the damaged thermocouples enabled provision to be made for their repair and recalibration. The damaged thermocouples, together with details of the repairs effected, are identified below:

2535: Snapped 1.7 m from hot junction. Remade cold end connection on 1.7m length.

2540: Snapped 3 m from potting seal. Remade cold end connection.

2541, 2542, 2544: Sheared within 60 mm of hot junction Remade hot junctions.

Table 7.1(a) lists the identification of all ten test thermocouples together with the record of the number of work cycles completed.

b. **Calibration**

Calibration is performed in two consecutive tests, 10215 and 10216. Each calibration run is conducted together with a control thermocouple, providing reference back to the "as received" calibration. All of the preaged thermocouples are recalibrated within the 300 mm original preaged segment (not applicable to control thermocouples (1) and (2), S/N's 2540 and 2544).

c. **Results**

The results of calibration tests 10215 and 10216 are presented in table 7.1(b). In this table the control thermocouple results have been placed at the end of the table to permit uninterrupted scanning of the

Table 7.2 Analysis of work aging results, trial A. Extent of error shown for thermocouples at time of issue (rows A and C), and on return from service (rows B and D).

(a) Deviation from standard after application of correction factor:
A. After preaging (average of: 2535-39).
B. After use (average of: 2535-44).

	Temperature (°C)										
	50	100	150	200	250	300	350	400	450	500	550
A:	0.4	0.6	0.4	0.4	0.5	-0.3	-0.3	-0.2	0.0	0.3	0.4
B:	0.2	0.7	0.6	0.6	0.8	-0.1	0.0	0.1	0.4	0.8	0.9

(b) Deviation from standard without preaging and compensation:
C. As received [Control(1): 2540].
D. After use (Average of: 2535-44).

	Temperature (°C)										
	50	100	150	200	250	300	350	400	450	500	550
C:	0.2	0.1	0.6	0.1	-0.2	-0.4	-0.7	-0.9	-0.6	-0.5	-0.2
D:	0.2	0.7	1.6	1.6	1.8	1.9	2.0	2.1	2.4	2.8	2.9

(c) Change of calibration during use. Difference between, "as received", and final, "after use", calibrations:

Temperature (°C)										
50	100	150	200	250	300	350	400	450	500	550
0.0	0.6	1.0	1.5	2.0	2.3	2.7	3.0	3.0	3.3	3.1

work aged results. Figure 7.1 provides a composite graphical overlay of the calibration data presented in table 7.1(b). In this figure the separation between the control thermocouple plots, (1), (4) and (5), and the work aged calibrations is representative of the total shift in calibration between "as received" and "as used" states.

1. **Preaged Thermocouples**

The preaged thermocouples S/N's 2535-39 and 2541-43 show an increased positive calibration, in the order of 0.5^{o}C at 500^{o}C. As the final calibrations of the original control(1) and (2) thermocouples, S/N's 2540 and 2544 are of the same order as the preaged thermocouples, their calibration data are included in the analysis showing average (Ave.) and three times standard deviation (3Sx). The result of this analysis is included in the presentation of the results in table 7.1(b).

2. **Control Thermocouples**

Control thermocouples (4) and (5), S/N's 2553 and 2554 respectively, provide traceability to the "as received" calibration. A comparison of these calibration results to control(1), S/N 2540, "as received" calibration is provided graphically in figure 7.1. Difference between the three sets of calibration results at each calibration point does not exceed 0.2°C, thereby allowing for a high degree of confidence in the respective calibration tests results.

d. **Comparison Analysis**

Table 7.2(a) lists the calibration deviation from standard, after the application of the correction factor, for the average of the 500°C preaged thermocouple data set (row A), and the equivalent average after use (row B). Figure 7.2 provides graphical representation of these averages plotted within the boundary of three times the standard deviation of the data points used to generate the averages. In plotting the boundary, the highest and lowest limit values, from the two data sets have been selected to provide the upper and lower boundary limits respectively. This boundary represents the level of uncertainty holding good, throughout the 14 to 82 cycles of use, of the preaged thermocouples for a 99.7% level of confidence. Limitation on establishing traceability (+/-1°C), forces an extension of this boundary to +/-1°C of the respective averages. In the region of 400 to 550°C, where the 3Sx values are from 0.7 to 0.9°C, the effect is not significant, and the uncertainty remains traceable to within +2/-1°C of the national standard with a 99.7% level of confidence.

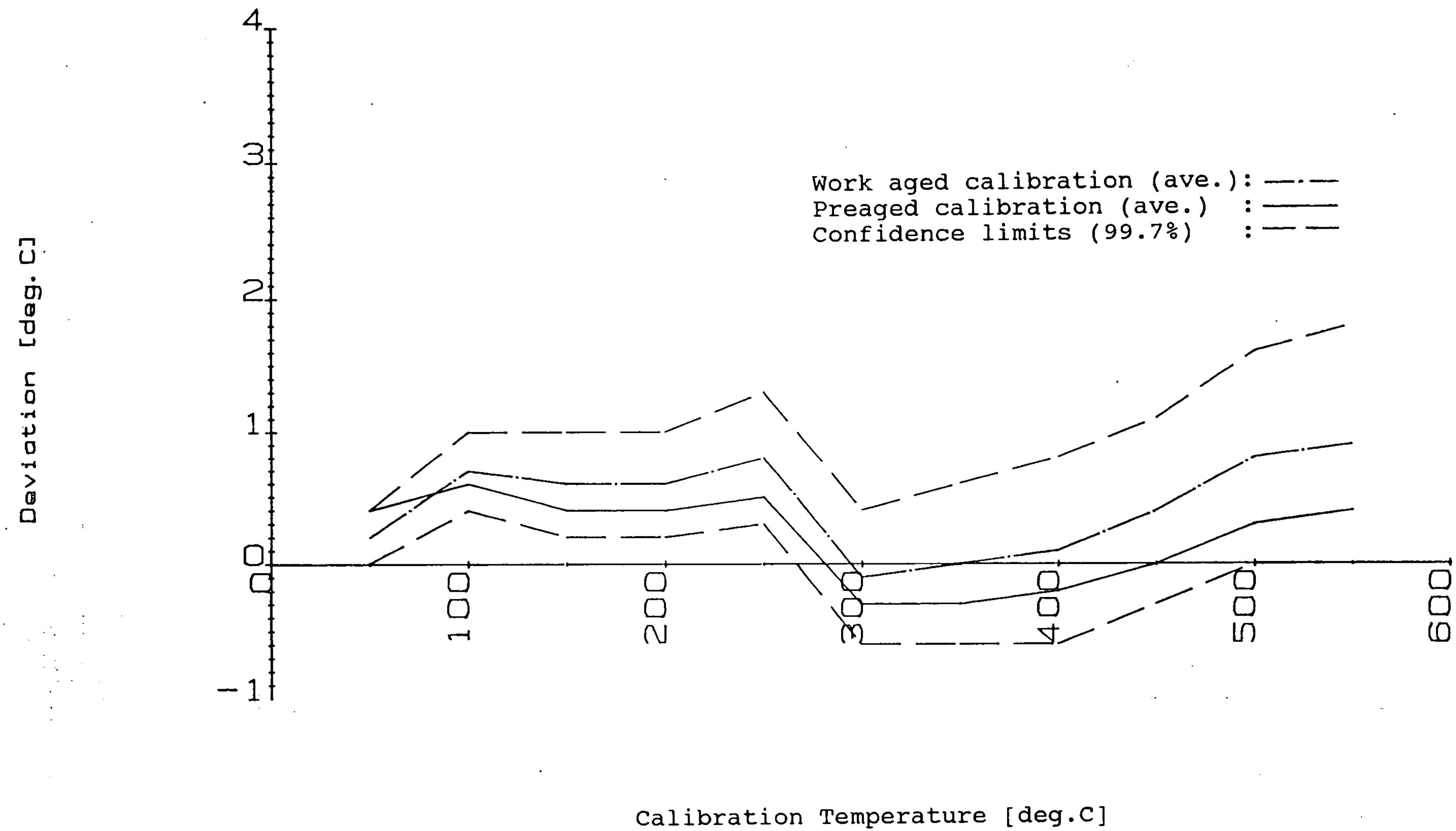


Figure 7.2 Uncertainty limits on preaged furnace load thermocouples. Derived from an evaluation of ten "trial A" thermocouples.

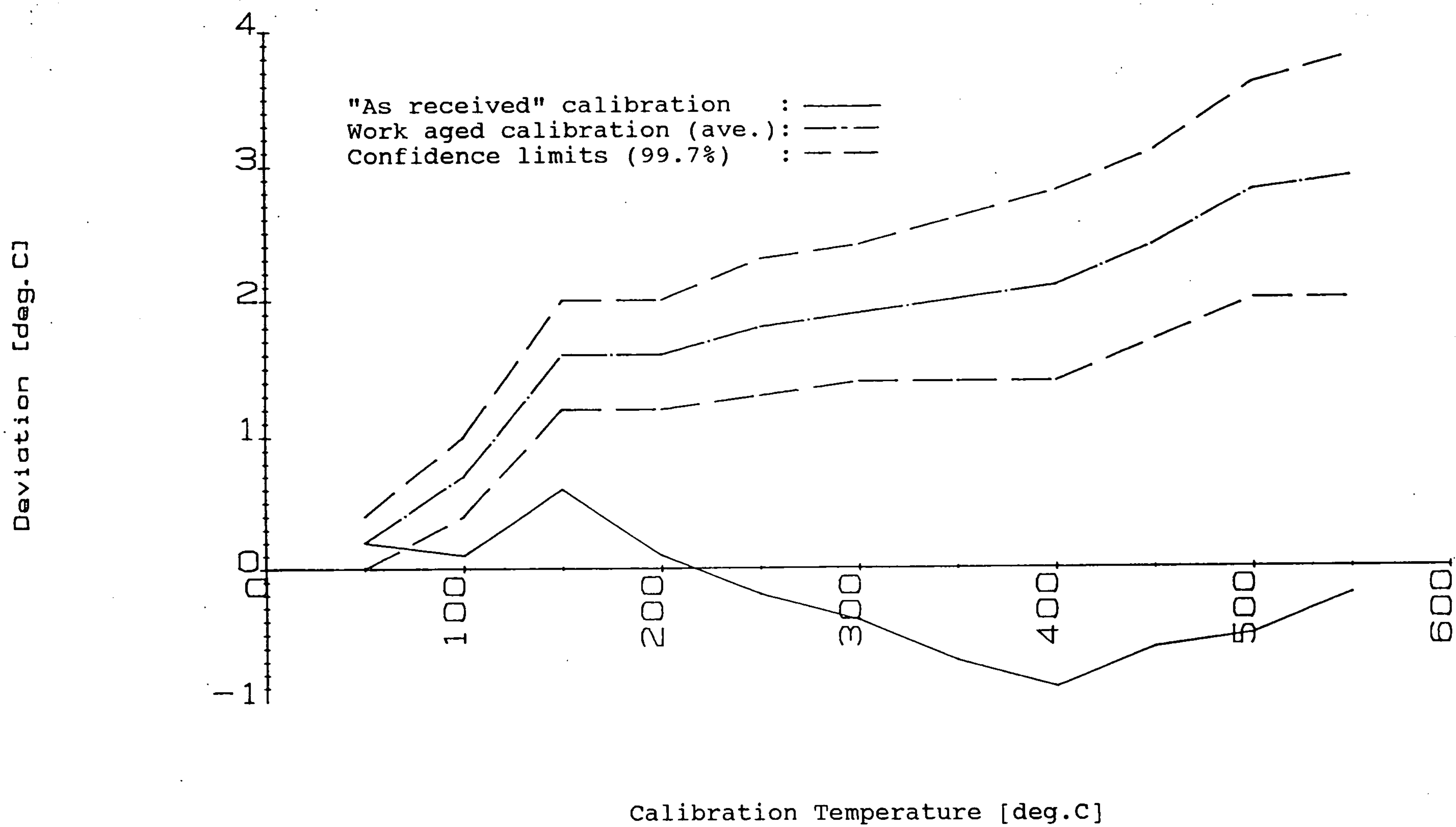


Figure 7.3 Variation in calibration between "as received" and work aged states. Data from "trial A" tests. Limits of uncertainty are shown for the work aged data set.

Table 7.2(b) lists the calibration deviation from standard for the "as received" and "after use" calibrations. This comparison indicates the extent of variation that occurs which, without calibration, is not able to be compensated for. Figure 7.3 provides graphical representation of this comparison. The 3Sx boundary for the "as used" calibration is included in the figure. Table 7.2(c) records the absolute difference between the two calibration states.

7.3.2 Trial B: Work Aged Calibration

a. Condition of Thermocouples

Three of the ten thermocouples returned from service damaged. Recalibration after repair of the damaged thermocouples provided unstable results, presumed due to the ingress of moisture into the mineral insulated sleeving. Calibration of these thermocouples is consequently not considered in these results. Identification of the ten thermocouples together with the record of work cycles, and respective calibration runs is provided in table 7.3(a).

b. Calibration

Calibration is carried out in two separate tests numbered 10244 and 10252. The results of both tests are considered together. Although no control thermo-

Table 7.3 Trial B work aged calibration results. Results from calibration tests 10244 and 10252.

(a) Thermocouple identification:

Thermocouple Serial No.	Conditions pertaining to aging: Range of soak temp.: 465 to 520°C	
Test 10252	Hours	Work cycles
2545	63	33
2546	63	33
2547	63	33
2548	---	--- (Damaged)
2549	63	33
2550	63	33
Test 10244		
2551	---	--- (Damaged)
2552	74	19
2553	---	--- (Damaged)
2554	74	19
	No control thermocouples	

(b) Results. Deviation from standard in degrees Celsius:

	Temperature of standard (°C)										
S/n.	50	100	150	200	250	300	350	400	450	500	550
2545	0.4	0.7	1.7	1.5	1.8	1.9	2.0	2.1	2.4	2.8	3.0
2546	0.4	0.8	1.8	1.6	1.8	1.9	2.0	2.1	2.4	2.8	3.0
2547	0.4	0.8	1.8	1.6	1.8	2.0	2.1	2.2	2.5	2.8	3.0
2548	Unstable										
2549	0.3	0.7	1.7	1.6	1.7	1.9	2.0	2.2	2.5	3.0	3.2
2550	0.3	0.7	1.7	1.6	1.8	2.0	2.1	2.2	2.5	2.9	3.1
2551	Unstable										
2552	0.5	0.7	1.4	1.3	1.5	1.6	1.6	1.8	1.9	2.2	2.2
2553	Unstable										
2554	0.5	0.6	1.3	1.2	1.3	1.4	1.4	1.4	1.6	1.9	2.0
Ave:	0.4	0.7	1.6	1.5	1.7	1.8	1.9	2.0	2.3	2.6	2.8

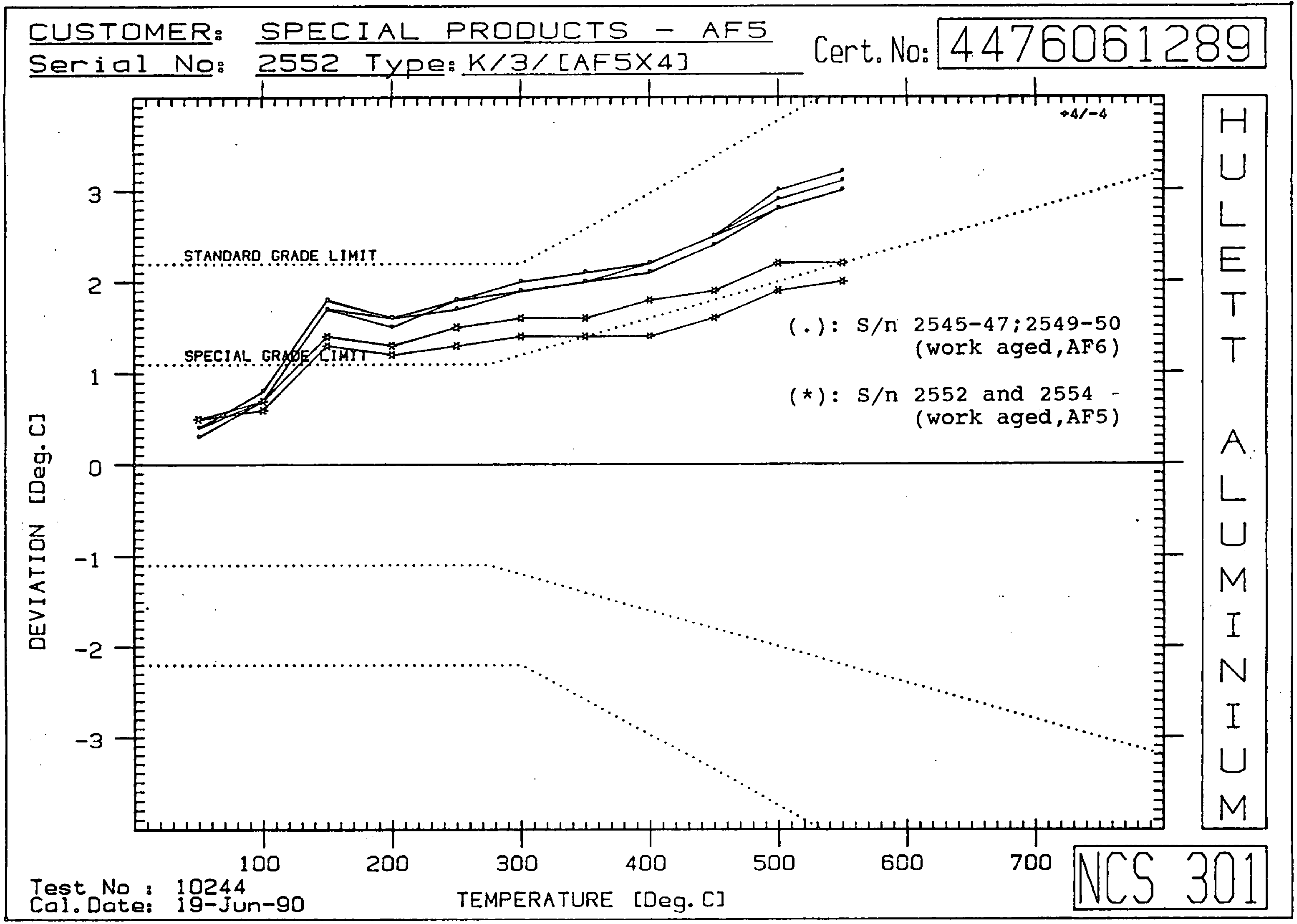


Figure 7.4 Calibration tests numbers 10244 and 10252. Composite overlay of trial B thermocouple calibrations after work aging.

couple calibrations are run with these tests, calibration results are supportive of Trial A findings and there is no reason to question the standard confidence levels for comparison and traceability.

c. **Results**

The results of the above calibrations are presented in table 7.3(b). Average and three times standard deviation (3Sx), for the data set, are presented in the same table. Figure 7.4 provides a composite graphical overlay of the full calibration data set. Data from the thermocouples work aged in AF 5 are identified by the markers (*), thermocouple work aged in AF 6, by the markers (.).

d. **Comparative Analysis with Trial A**

Table 7.4 provides comparison of Trial A and B work aged thermocouple averages, and comparison of the minimum and maximum datum points occuring at each calibration point in Trial B data set, with the three standard deviation limits established from Trial A data. Whilst the Trial B data transgresses the 3Sx limits at three points, the infringement is, in each case, limited to 0.1^{o}C.

Application of the apportioned calibration correction factors to the minimum and maximum values extracted from Trial B data demonstrates that these data fall

Table 7.4 Trial A and B comparison of data. Trial B minimum and maximum values are shown referenced to Trial A, 3Sx limits. Values expressed as deviation from standard in degrees celsius.

	Calibration Temperature										
	50	100	150	200	250	300	350	400	450	500	550
3Sx High Limit (trial A)	0.4	1.0	2.0	2.0	2.3	2.4	2.6	2.8	3.1	3.6	3.8
Highest t/c trial B	0.5*	0.8	1.8	1.6	1.8	2.0	2.1	2.2	2.5	3.0	3.2
Average trial B	0.4	0.7	1.6	1.5	1.7	1.8	1.9	2.0	2.3	2.6	2.8
Average trial A	0.2	0.7	1.6	1.6	1.8	1.9	2.0	2.1	2.4	2.8	2.9
Lowest t/c trial B	0.3	0.6	1.3	1.2	1.3	1.4	1.4	1.4	1.6*	1.9*	2.0
3Sx Low Limit (trial A)	0.0	0.4	1.2	1.2	1.3	1.4	1.4	1.4	1.7	2.0	2.0

(*) Out of Limits

within the +2/-1°C limits established by Trial A, and no further analysis is considered necessary. An example of the application of the correction factor at 500°C is given as follows:

Correction factor at 500°C = -2°C
Highest t/c at 500°C:
S/N 2549 at 3.0°C + (-2°C) = 1.0°C calibration error

Lowest t/c at 500°C:
S/N 2554 at 1.9°C + (-2°C) = -0.1°C calibration error

At 500°C in Trial B calibration data with correction factor applied falls within the limits: +1.0/-0.1°C.

7.4 WELLMAN FURNACE WORK AGING TRIALS

7.4.1 Introduction

This test represents the practical application of the preaging technique and calibration correction factors. Three Type K, 3mm, mineral insulated, stainless steel sheathed load thermocouples, S/N's 3058-60, each 8 m long, are employed in this final evaluation trial. The furnace used for the evaluation, the Wellman furnace, is a vertical solution heat treatment furnace, with a nominal aging temperature of 465°C.

7.4.2 The Practical Trial

a. Preaging

The thermocouples are preaged within the Wellman furnace with as much of their length as possible (6 to 7 m) within the furnace chamber. This practice ensures that the areas normally positioned across the

Table 7.5 Wellman trials calibration results from calibration tests 10219 ,10236, 10298, and 10299.

(a) Calibration test identification:

Calibration Test No.	Conditions pertaining to aging at time of calibration: Hours	Work cycles
10299	As received (refer to text)	
10219	Preaged (500^{o}C, 1 hour 15 minutes)	
10236	64	23
10298	211	70 (total)

(b) Results. Deviation from standard in degrees Celsius:

	Temperature of standard (^{o}C)										
S/n.	50	100	150	200	250	300	350	400	450	500	550
Test 10299:											
3058	0.3	0.6	1.2	1.0	0.7	0.6	0.5	0.8	1.2	1.6	1.6
3059	0.3	0.6	1.3	1.0	0.8	0.7	0.7	1.0	1.4	1.9	1.9
3060	0.3	0.5	1.1	0.9	0.6	0.4	0.4	0.9	1.2	1.5	1.5
Ave:	0.3	0.6	1.2	1.0	0.7	0.6	0.5	0.9	1.3	1.7	1.7
Test 10219:											
3058	0.4	0.9	1.8	2.0	2.3	2.4	2.6	2.9	3.3	3.8	4.2
3059	0.4	1.0	2.0	2.2	2.6	2.7	2.8	3.1	3.3	3.6	3.5
3060	0.3	0.9	1.9	2.1	2.5	2.6	2.9	3.0	3.2	3.5	3.5
Ave:	0.4	0.9	1.9	2.1	2.5	2.6	2.8	3.0	3.3	3.6	3.7
Test 10236:											
3058	0.4	1.0	2.1	2.2	2.5	2.8	3.0	3.3	3.7	4.0	4.1
3059	0.4	1.0	2.1	2.3	2.6	2.8	3.0	3.3	3.7	4.1	4.2
3060	0.4	1.0	2.1	2.3	2.7	3.0	3.2	3.4	3.8	4.2	4.3
Ave:	0.4	1.0	2.1	2.3	2.6	2.9	3.1	3.3	3.7	4.1	4.2
Test 10298:											
3058	0.6	1.3	2.3	2.4	2.6	2.8	3.1	3.5	3.9	4.3	4.4
3059	0.5	1.3	2.4	2.6	2.9	3.1	3.4	3.9	4.2	4.6	4.7
3060	0.5	1.2	2.3	2.4	2.7	2.9	3.2	3.5	4.0	4.3	4.4
Ave:	0.5	1.3	2.3	2.5	2.7	2.9	3.2	3.6	4.0	4.4	4.5

Table 7.6 Comparison of averages from Wellman trials. Averages derived from calibration of "as received", preaged, intermediater work aged and fully work aged, conditions. The error columns provide indication of the extent of error remaining after correction.

		Calibration Temperature										
		50	100	150	200	250	300	350	400	450	500	550
As received (10299)	A.	0.3	0.6	1.2	1.0	0.7	0.6	0.5	0.9	1.3	1.7	1.7
Preaged (10219)	B.	0.4	0.9	1.9	2.1	2.5	2.6	2.8	3.0	3.3	3.6	3.7
Intermediate (10236)	C.	0.4	1.0	2.1	2.3	2.6	2.9	3.1	3.3	3.7	4.1	4.2
Final (10298)	D.	0.5	1.3	2.3	2.5	2.7	2.9	3.2	3.6	4.0	4.4	4.5
Initial offset :	(B-A)	0.1	0.3	0.7	1.1	1.8	2.0	2.3	2.1	2.0	1.9	2.0
Work ageing drift:	(D-B)	0.1	0.4	0.4	0.4	0.3	0.3	0.4	0.6	0.7	0.8	0.8
Correction factors		------------		-2		-2		-3		-3		
Error:												
Preaged - On issue		*	*	-0.1	0.1	0.5	0.6	-0.2	0.0	0.3	0.6	0.7
Final - On return		*	*	0.3	0.5	0.7	0.9	0.2	0.6	1.0	1.4	1.5

(*) No correction factor provided in this temperature range.

main thermal gradient, as well as the areas extending into the ambient condition, are fully preaged. Furnace air temperature for preaging is 500°C and soak time is 1 hour 15 minutes.

b. **Procedure**

The test thermocouples are calibrated in the "as received" condition prior to preaging. After preaging, correction factors are apportioned, and the thermocouples issued for use. After 23 work cycles, major maintenance to the furnace, provided the opportunity to recalibrate the thermocouples. After recalibration new correction factors are provided and the thermocouples replaced in service. For the purpose of evaluation, only the initial correction factors are referenced.

c. **Results**

Details of the four relevant calibration runs are provided in table 7.5(a). The original "as received" calibration record was not located on the LAB87 calibration data storage discs. In order to recapture this data the load thermocouples are cut back to within 500 mm of the cold end pot seal, the hot junction remade, and the unaged material in this area calibrated (test 10299). Calibration deviation results from each of the runs are tabulated in table

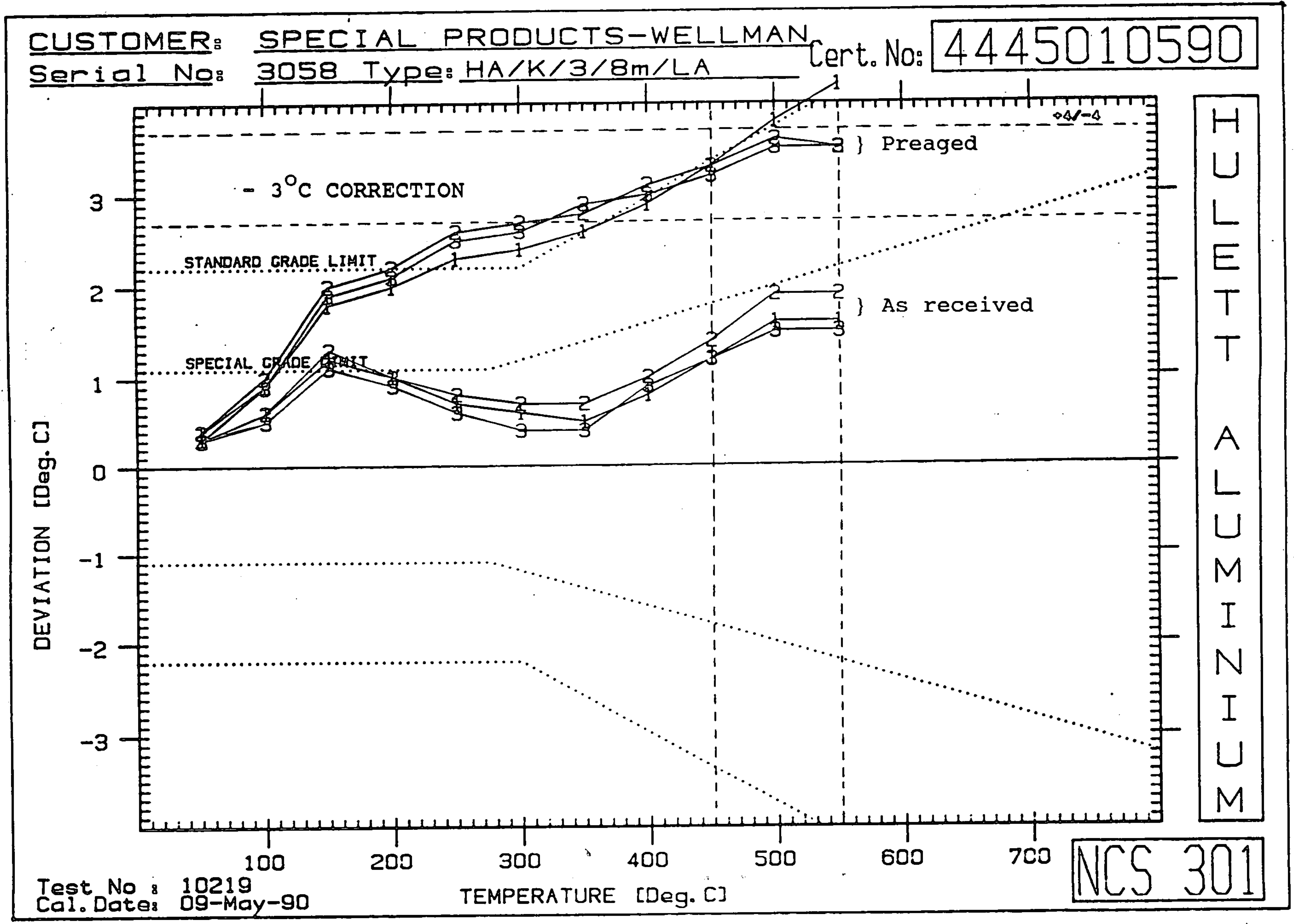


Figure 7.5 Calibration tests, numbers 10219 and 10299. Composite overlay of thermocouple calibrations, as received, and preaged. Grid demonstates apportioning of calibration correction factor from 450 to 550°C.

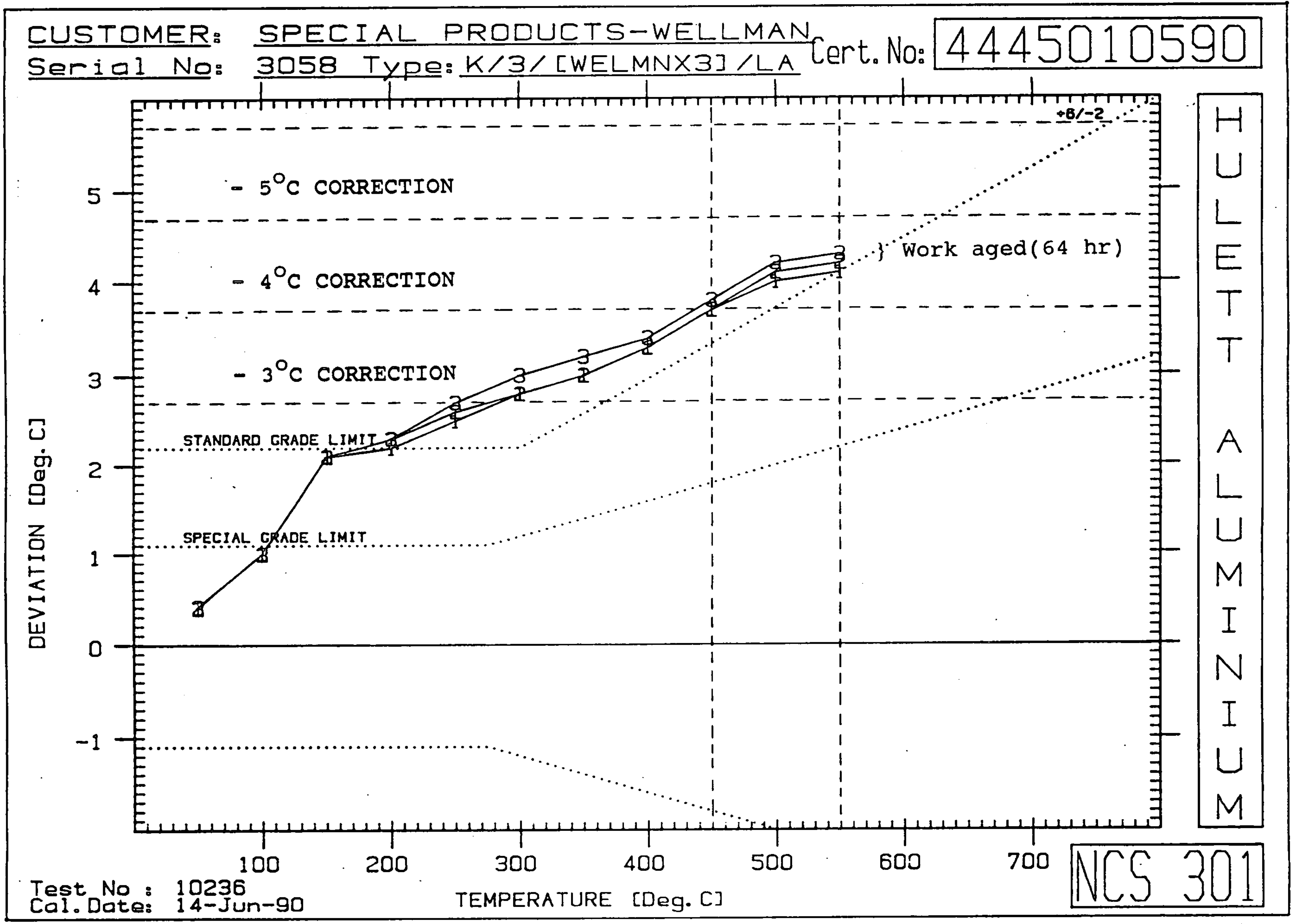


Figure 7.6 Calibration test number 10236. Calibration correction grid shows work aged thermocouple calibration in the region 450 to 550°C, as having drifted into the -4°C correction segment.

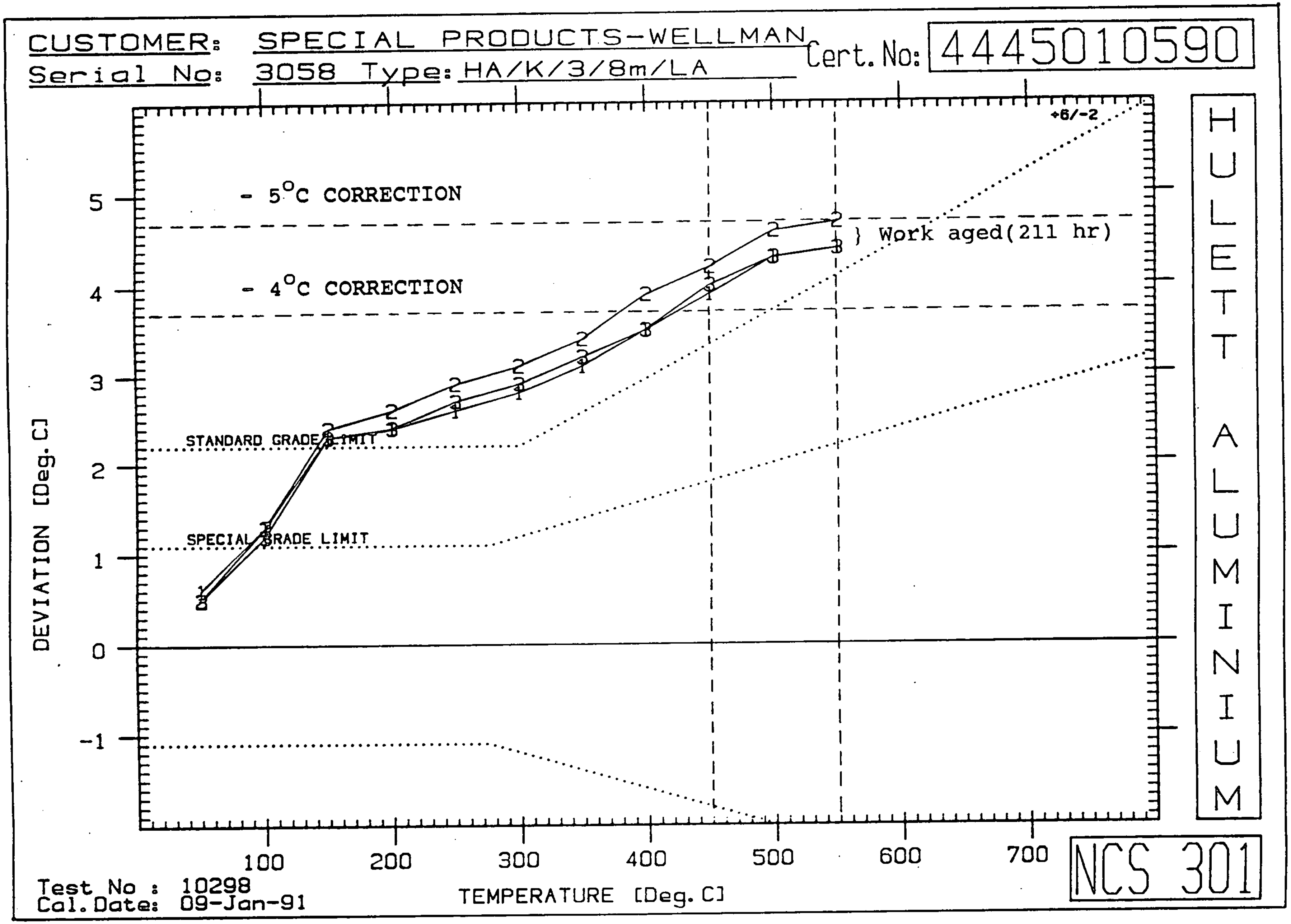


Figure 7.7 Calibration test number 10298. Calibration correction grid shows work aged thermocouple calibration, in the region 450 to 550°C, as having experienced positive drift within the -4°C correction segment.

7.5(b). Table 7.6 presents a comparison of the averages of the data from each run, providing indication of the extent of initial offset and work aged associated drift.

Figure 7.5 presents a graphical overlay of the data from calibration runs 10299 ("as received"), and 10219 (preaged). In each data set the thermocouples are identified by their individual markers (1), (2) and (3), allocated consecutively according to their serial number in ascending order. For clarity the graphs of the two subsequent calibrations are not overlaid but presented independently in figures 7.6 (test 10236) and figure 7.7 (test 10298).

d. Load Thermocouple Work Log Sheet

The Load Thermocouple Work Log Sheets, figures 7.8 and 7.9, (copies of the working documents), provide a record of the work cycles, and soak times. These records provide indication of the type of work scheduling experienced by load thermocouples in a solution heat treatment furnace. Unless indicated as "anneal" or "survey" all cycles include a water quench from the indicated soak temperature.

e. Evaluation of results

Table 7.6 includes a presentation of the error remaining after the application of calibration correction factors. As there is insufficient data to prod-

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FURNACE: Wellman

4 4 4 5 3 1 3 5 9 C

DATA SHEET NUMBER

LOAD THERMOCOUPLE WORK LOG SHEET

FULL CALIBRATION

cal 10217/10219

Identification: 3058 ; 3059 ; 3060

Temperature	200°C	300°C	400°C	500°C
THERMOCOUPLE CORRECTION FACTOR	−2	−2	−3	−3

CONTROL: (TTL CONTROL 01)

DATE	BATCH – TIME	TEMP.	HOURS	REMARKS	DATE	BATCH – TIME	TEMP.	HOURS	REMARKS
90/05/18	Dummy Load 1	465°C	1¼	465 +0°C −2°C					
90/05/19	Dummy 2	465°C	1¼	465 +0 −3°C					
90:05:20	Dummy Load 3.	465°C	1¼	465° +0° −2°C					
90:05:21	T330	465	1¼	465 +0 −2°C					
90:05:21	T170	465°C	1¼	465° +0 −2°C					
90:05:22	T170	465°	1¼	465° ±0 −2°C					
90:05:23	T170	465	1¼	465° +0° −2°C					
90:05:23	T170	465	1¼	465 +0 −3°C					
90:05:24	T170	465°	1¼	465 +0° −2°C					
90:05:25	T171	465°C	1¼	465 +0° −3°C					
90:05:26	T171	465°C	1¼	465° +0 −2°C					
90:05:27	T171	465°	1¼	465° ±0° −2°C					
90:05:29	T171	465°	1¼	465° ±0 −2°C					
90:05:29	T172	465°	1¼	465° ±0 −2°C					
90:05:30	T172	465°C	1¼	465° +0° −2°C					
90:05:30	T172	465°C	1¼	465° ±0 −2°C					
90:06:01	T172	465°C	1¼	465° +0 −2°C					
90:06:04	T173.	465°C	1¼	465° +0° −2°C					
90:06:05	T173	465°C	1¼	465° +0 −2°C					
90:06:06	T173	465°C	1¼	465° +1 −2°C					
90:06:07	T173	465°C	1¼	465° +2 −2°C					
90:06:07	T173	465°	1¼	465° ±0 −2°C					
90:6.9	Dummy	465	36 h						
removed for recal									
	cal 10236								

Figure 7.8 Work Log Sheet, 90.05.18 to 90.06.09 covering first part of Wellman load thermocouple work aging trials.

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FURNACE: Wellman

47450105 90

LOAD THERMOCOUPLE

WORK LOG SHEET

DATA SHEET NUMBER

FULL CALIBRATION CONTINUATION

CAL 10236

Identification: S/N 3058. 3057. 3060.

Temperature	200°C	300°C	400°C	500°C
Thermocouple Correction Factor	-2	-3	-3	-4

CONTROL: TM CONTROL 01

DATE	BATCH / TIME	TEMP.	HOURS	REMARKS
21.06.90	T173	465±5°	3hrs 35mins	S.H.T.
21.06.90	T173	465±5°	4hrs 45mins	S.H.T.
22/06/90	T173.	465±5	2H 40m	SHT
22/06/90	Furnace Survey	465±5°	4HRS 05mins	
04.07.90	T351	465±5°	1¼ Hrs	+1 -2
05.07.90	T351	465±5°	1¼ Hrs	+0 -2
90.07.13		465	1 h	SURVEY
90.07.14		465	12.5hrs	SURVEY
90.07.15		465	2.5h	SURVEY
17.07.90	T351	465°	1¼ Hrs	+3 -1
18/07/90	T351	465±5°	1¼ Hrs	+6 -2
19/07/90	T351	465±5	1¼	+4 -2
[illegible]	[illegible]	[illegible]	1¼	+4 -2
23/07/90	T351	465±5	1¼.	
24.07.90	T174	465±5°	1¼	+4 -2
25.07.90	T174	465±5°	1¼	+4 -2
26.07.90	T174 Re-SHT	465±5°	1¼.	+4 -2
27.07.90	190	465±5°	1½	+3 -3 & SHT.
28.07.90	199	350°±10°	2	+5 -0 ANNEAL.
30.07.90	T174 Re-SHT	465°±5°	1¼	
31/07/90	T460	465±5	3½	3H 50m SHT
08/08/90	T174	465°±5°	3hrs 30mins.	S.H.T.
10.08.90	T174	465°±5	1¼	+2 -3 SHT.
21.08.90	Circles.	340°±10°	2HRS.	05min ANNEAL.
29.08.90	199	350°±10°	5HRS	55min ANNEAL.
31/08/90	199	350°±10°	6Hrs	55min Anneal
			6Hrs 55min	Anneal
10.09.90	T432	420°±10°	8 HRS	15min ANNEAL
10/09/90	T452A	465°±5°	3Hrs 40min	S.H.T.
11/09/90	T452A	465±5	2Hrs 45min	S.H.T.

DATE	BATCH / TIME	TEMP.	HOURS	REMARKS.
14.09.90	Furnace Survey	465±5	2Hrs 20	S.H.T.
18.09.90	Ref Sheet Mill	430±5°	5 Hrs	SHT.
18/09/90	Ref S/Mill	430±5	4Hrs [illegible]	SHT
[illegible]	T452A	465±5	2Hrs	[illegible] SHT.
[illegible]	T452A	465±5	2Hrs	15min S.H.T.
21/09/90	T440	465±5	2Hrs	35min S.H.T.
[illegible]	T440	465±5	2Hrs	10min S.H.T.
22.09.90	T440	465±5	2 HRS	55min SHT.
22.9.90	T440	465±5	3 HRS	35min SHT
24.9.90	Pre Survey	500	2h	C1
25/09/90	T438	500°C	2HRS	15mins S.H.T.
26/09/90	T438	500	2HRS	[illegible] SHT
26.10.90	Ref Sheet Mill	450±5	3HRS	[illegible] min SHT.
27.10.90	Ref S/Mill	450±5	5 HRS	05mins SHT
27.10.90	Ref, S/Mill	450±5	4Hrs	
28.10.90	Ref Sheet Mill	450±5°	4HRS	35min SHT.
29.10.90	Ref Sheet Mill	450±[illegible]	3Hrs	

Figure 7.9 Work Log Sheet, 90.06.21 to 90.10.29 covering second part of Wellman load thermocouple work aging trials.

uce a meaningful standard deviation, the 99.7% confidence uncertainty limit of +/-1^{o}C, for traceability, is applied. From data in Trial A this limitation is shown to be justifiable. The results of the Wellman trials indicate that error is contained within the uncertainty limit +3/-2^{o}C for a 99.7% confidence interval. Without calibration correction, error ranges from the "as received" value to the final calibration values, a range of +0 to +5^{o}C, with uncertainty expressed as, +6/-1^{o}C.

7.5 SUMMARY

7.5.1 Calibration Compensation: Conclusion

Results of the work aging trials support the hypothesis that, using preaged thermocouples and applying calibration correction factors, it is possible to record the load temperature within an accuracy of (+/-)3^{o}C of the true temperature traceable to the National Standard within a 95% level of confidence. Whilst supporting this hypothesis, further evaluation of the following areas is necessary before this level of confidence may be statistically claimed:

(i) Longer term characteristics of work related drift.

(ii) Effects of inhomogeneity caused through quenching.

(iii) Variance of calibration within a thermocouple batch after work aging.

(iv) Precision and accuracy of recording instrumentation.

(v) Furnace temperature uniformity.

(vi) Furnace control stability.

7.5.2 Consistency of Offset

The first assumption of this project is that thermocouples used during the test are considered representative of the same grade, Type and gauge from other sources. From the results of the preliminary and formal trials it is clear that the degree of offset produced during preaging is unique to a specific batch of material and may not be considered representative. The phenomenon of initial offset occurring under the stated conditions is consistent, but the extent of offset is required to be ascertained for individual batches. An investigation is required into sourcing material from a manufacturer whose material sources and manufacturing techniques are such that the thermocouples produced provide a "post offset" calibration close to ITS-90 specification.

7.5.3 Recalibration Interval

From consideration of the load thermocouple drift after preaging, as graphically represented in figures 7.5 to 7.7, it is apparent that a practice of recalibrating thermocouples after a period of work aging could significantly lower the level of uncertainty. From the Wellman trials data, recalibration after the first 100 hours of production soaking is indicated. Recalibration after a further interval is then required to maintain this lower level of uncertainty. Data captured in the Wellman trials is not extensive enough to consider proposing post work aging recalibration techniques, and where lower levels of uncertainty are required, this area is open to investigation.

CHAPTER 8

GENERAL DISCUSSION

8.1 INTRODUCTION

In the light of the completed trials the general discussion evaluates the combined result and reviews certain of the procedures. Where applicable, difficulties experienced and deviations from the intended line of research are identified, and explained. Consideration and discussion is given to related literature, published subsequent to the running of the trials. An application of the findings, as implemented at the Hulett Aluminium Profiles heat treatment plant in Pietermaritzburg, is presented.

8.2 EVALUATION OF THE COMBINED RESULTS

8.2.1 General Remarks

Correlation between the final calibration results produced by the thermocouples employed in Trials A and B is of significance when considering the range of preaging and work cycling. Of the seventeen thermocouples to survive through to final calibration the largest variation between calibration results is 1.2^{o}C occurring at 550^{o}C. The following list summarises the details down to the "bottom line" for the critical temperature of 500^{o}C:

(i) Range of preaging: none (control thermocouples)

160 hr at 500°C (S/N 2539)
40 hr at 600°C (S/N 2543)

(ii) Range of work aging: 30 hr 14 cycles
189 hr 82 cycles

(iii) Range in calibration deviation (500°C): 1.9 to 3.0°C
Variation : 1.1°C

(iv) Range in indicated error (at 500°C)
with application of correction factor :-0.1 to 1.0°C

The twofold significance of these results is that whilst, for a specific batch of thermocouples, calibration remains within a defined band of error, the width of this band (1°C) is such that cognisance must be taken of this variance in any practical uncertainty claims.

In view of the above, the evaluation of the results in chapter seven, where an uncertainty limit of +3/-2°C is derived, is reasonable and realistic.

8.2.2 Hypothesis 1: Preaging

a. Calibration Drift

The investigation confirms the positive effect that preaging has on the stability of Type K thermocouples. Preaging does not significantly influence drift as initially expected. Preaging rather enables the large initial calibration offset, which from the data collected, appears to be a result of short-range ordering, to be accounted for. This results in an immediate benefit of 2 to 3°C decrease in uncertainty at 500°C. As the findings indicate, the preaging soak time is not critical and a soak time of one to two hours at the determined temperature of 500°C is recommended as a satisfactory preaging procedure.

b. **Homogeneity**

The homogeneity of the trial thermocouples is not tested in the calibration procedures used in this investigation. Calibration of the trial thermocouples is conducted on a section of each thermocouple located within 300 mm of the hot junction, an area not exposed to the varying gradients likely to cause inhomogeneity. The close correlation in calibration results between thermocouples exposed to varying degrees of work cycling, provides indication that inhomogeneity caused through uneven heat treatment would, after preaging, not produce severe interference in measurements. This consideration does however remain untested, and is an element for consideration in further research.

8.2.3 Hypothesis 2: Calibration Correction

a. Level of Uncertainty

Establishing the level of uncertainty resulting through employment of preaging procedures and application of the calibration correction technique, is dealt with comprehensively in chapter seven and it only remains to state that the uncertainty of $+3/-2^{o}C$ for a 99.7% level of confidence positively addresses the second hypothesis. The intent of the hypothesis is that the final, indicated or recorded load temperature value, be statistically traceable within the stated accuracy. This project has, however, confined its investigation to the primary sensor alone. Uniformity of heat distribution within the furnace, recorder calibration, effects of cabling and connections, have not been considered. An evaluation of the aforementioned factors requires further investigation. The automated calibration, preaging and calibration compensation techniques dealt with by this project make such further investigation possible within a production environment.

The original statement of the hypothesis, in line with current National Calibration Service recommended practice, considers a 95% level of confidence. The low level of uncertainty established by the results suggests the higher 99.7% level of confidence as more appropriate. Consequently three standard deviation values are employed in the statistical evaluation.

b. Application of Correction Factor

Whilst application of calibration correction involves only the simple addition or subtraction of a single digit figure, some complexity is generated when it is necessary to apply a factor to both "set value" and "measured" or "process values". Correction values for the traditional positive deviation in Type K thermocouples, require the subtraction of the relative factor from the measured value. However, in the case of setting a controller, the same correction value is required to be added to the required temperature. In practice this is found to cause a degree of confusion and an automated method of correction is preferable, and recommended in any application of calibration correction to a production unit.

8.2.4 Conclusion

The findings of the investigation are summarised as follows:

Thermocouples: Type K
Mineral insulated
3 mm Diameter stainless steel sheath

Preaging: 1 to 2 hours at 500^{o}C (+/-10^{o}C)

Calibration: After preaging to traceability of +/-1^{o}C

Correction factor: Unitary value derived from calibration deviation at the relevant temperatures

Uncertainty attainable: +3/-2^{o}C for sensor only at a confidence level of 99.7%

8.3 PROBLEMS ENCOUNTERED DURING THE PROJECT

8.3.1 Establishing Calibration Uncertainty Trials

During the running of the calibration uncertainty trials, 15 September 1989 through 3 October 1989 (table 4.8), a problem with the furnace control program resulted in a control situation with less than desirable stability. Evaluation data file records make it clear that the situation affected the data average file. Average response stabilising times for the runs are 53 minutes compared to the targeted 36 to 45 minutes. Repeating tests run over this duration is not feasible due to the disruption of the laboratory's normal workload. Evaluation of the trial's data set reveals that, regardless of the instability, an acceptable result is produced. Average deviation is 0.1^{o}C with (+/-)0.4^{o}C uncertainty for a 99.7% confidence interval. This result enables the data to be utilised for establishing calibration uncertainty. The problem with the furnace control is identified and cleared. A modification to LAB87 enables the program to utilise work load runs to capture comparative data between the standard and reference thermocouples. Analysis of ten such runs (table 4.13) with greatly improved response times, yields an identical evaluation of uncertainty.

This exercise resulted in an extensive evaluation of all data involved in the test run and the report in chapter four is fully comprehensive. The problem highlighted the capability of the control algorithm to deal positively with abnormal situations without compromising the integrity of the calibration results.

During the same trials a power failure resulted in the abortion of one test run after 300^{o}C. An incorrect program instruction resulted in the a further run having an interval of 100 instead of 50^{o}C. Sufficient runs had, however, been planned to allow for the possible loss of data as such problems, particularly with loss of power, are common in industrial situations.

8.3.2 Preaging Soak Trials

During the elaborately planned, trial A soak schedule (table 5.6), an explosion on an inadvertantly overloaded electrical distribution board resulted in loss of power to the furnace being used to age the thermocouple (S/N 2539). A temperature of 400^{o}C is, at the time, considered the maximum permissable drop below which the specific test, having completed 80 of the 160 hour soak period, would be considered invalidated. By utilising a limited capacity uninterruptable power source the temperature was able to be maintained above 400^{o}C until the reinstatement of the power. Subsequent evaluation of results reveal that the interruption has no effect.

8.3.3 Control Thermocouple Deviation

Control thermocouples are used throughout the Trial A and B calibration tests in order to provide a common reference point directly related to the thermocouple batch under test. Five of the twenty test thermocouples in these trials are allocated as control thermocouples. The particular importance of the control thermocouple is highlighted in calibration run 10201 (figure 6.3) where control thermocouple (2) exceeds an acceptable difference from the previous control thermocouple (1) (table 6.3). This information leads to a full investigation of the calibration program. An excessive response time at the 400^{o}C calibration point (table 6.4), matching with the start of the deviation between control thermocouples, identifies this as the source of the problem. Possible causes are investigated, supplementary checks programmed for following calibration runs, and the effect of the problem on the integrity of the trial results is evaluated, taken account of and reported on (par.6.2.1, b.,4).

8.4 DEVIATION FROM INTENDED INVESTIGATION

8.4.1 Control Thermocouples

Informal investigation of the effects of aging are currently being carried out on control thermocouples from the solution heat treating furnaces and provide results similar to those for load thermocouples reported in this study. The reasons for not associating the two investigations are:

(i) The difficulty in conducting preaging evaluation on the shorter control thermocouples.

(ii) The difference in work cycling. Control thermocouples seldom drop below 200°C. The longer time at operating temperatures, combined with a longer life expectancy, results in the extended effect of aging being of more consequence than with load thermocouples.

(iii) The difficulty in accessing control thermocouples for recalibration.

The circumstances of control thermocouples are such that they warrant full investigation with the accompanying aspects of furnace control (instrumentation, temperature distribution, operating practice). For this reason, it is considered preferable to omit the partial data obtained from this report.

8.4.2 Comparison of Thermocouples from Different Sources

From the preliminary trials it becomes apparent that it is necessary to treat each batch of thermocouple material individually. Any one batch may not be considered representative of that thermocouple Type's initial calibration offset characteristics. The comparative study of material from different sources is not necessary in terms of this project's objective. Although no specific comparison is made, the Trial A and B thermocouples originate from a different source to those used in the Wellman trials.

8.5 RELATED LITERATURE

8.5.1 Thermocouple Calibration Stability

Daneman, in his paper "Thermocouple Calibration Stability", subtitled "The Choice of Metal Sheathing for Mineral-insulated Thermocouples", dated December 17, 1990 (Daneman ,1990), suggests that sheath material is a primary factor in thermocouple instability. Daneman supports this supposition through consideration of the factors contributing to instability and drift. The compositional changes resulting from contamination, as referred to in the paper, are likely to be more active at the temperature quoted (1100 and 1200°C) than at aluminium heat treating temperatures (maximum 550°C).

In his discussion Daneman refers to several interesting aspects of hysteresis in Type K thermocouples that are similar to the findings reported in this dissertation. The points noted by Daneman are:

(i) Hysteresis occurs when the thermocouple is cycled between 200 and 600°C.

(ii) Maximum hysteresis of approximately 5°C occurs at 450°C.

(iii) Where used below the peak hysteresis temperature (450°C), the degree of hysteresis is reduced by annealing overnight at 500°C.

(iv) Crystallographic investigations do not support the hypothesis that hysteresis is a form of short-range ordering.

The hysteresis resulting through heating to 600°C is observed in the preaging investigation at this temperature, reported in chapter six. The points Daneman notes are not directly comparable with this investigation which is concerned with deviation from standard rather than hysteresis. The similarity of the stabilising temperature (500°C) is, however, worth noting, as is the point that peak hysteresis occurs at 450°C. Temperatures employed in the work aging trials rise to 520°C and may account for the degree of spread in calibration.

Throughout this research project calibration has been considered on rising temperature. Although, in the heat treatment of aluminium, absolute values are not critical on the cooling cycle (the rate of cooling may be critical), an investigation into the effects of hysteresis will be necessary in any consideration of the control thermocouples where the operating temperature of a furnace may be significantly lowered between runs.

8.5.2 Selection and Maintenance of Thermocouples

In a paper titled "Selection and Maintenance of Thermocouples for Industrial Applications" (Wang,1990,pp.599-613), presented at the Aluminium Association's 1990 Technology Seminar, held in San Antonio, Texas, T.P. Wang mentions two important points with respect to the maintenance of thermocouple accuracy:

(i) A good maintenance program starts, he suggests, with good knowledge of the thermocouple and the measuring/control equipment.

(ii) For the most accurate requirement, the base metal thermocouples are discarded after a single use.

Point (ii) is considered a valid alternative to the pre-aging method. The procedure of using thermocouples once only, ensures that they are used in the "as received" state, which, as each of this projects calibration runs show is the closest to standard. There are several drawbacks to the method:

(i) Cost. Offset partially by the lower gauge wire and type of insulation.

(ii) For most accurate work calibration must still be taken into consideration.

(iii) Because of "single use" application calibration is required to be carried out on a sample of the thermocouple wire. The most cost effective method is to purchase bulk reels and calibrate samples periodically through the reel as it is used.

(iv) If calibration is not carried out the system is open to significant error and there is no support for claims of increased accuracy.

The above application remains a valid alternative for the Hulett Aluminium Profiles heat treating plant, as the calibration facilities existing on site would enable the highest possible accuracy to be achieved. Current

Standard Operating Procedures, based on the conditions of International Standards, specify the number and positioning of thermocouples to be used on each heat treatment load. Under these procedures the application of "single use" thermocouples is not viable on cost. It is expected that further investigation into furnace performance, will enable the high number of thermocouples used to be reduced and the "single use" thermocouple may become a cost effective proposition.

8.6 APPLICATION AT HULETT ALUMINIUM PROFILES

8.6.1 Preaging Technique

As a result of the findings of this project the following technique is currently in operation at the Hulett Aluminium Profiles factory in Pietermaritzburg.

(i) Thermocouple material (3 mm, stainless steel sheathed, mineral insulated, Type K) is ordered in bulk batches (+/-300 m).

(ii) On receipt of the imported material the thermocouple manufacturer (importer) manufactures two test sample thermocouples from each reel of thermocouple wire to be supplied.

(iii) One sample is calibrated by the Hulett Temperature Laboratory in the "as received" condition. Recalibration of the same thermocouple provides indication of the thermocouples expected performance after preaging. If the results are satisfactory the manufacturer is advised, he then despatches the full batch of thermocouple material to Hulett Aluminium. The second test thermocouple is retained by the Hulett Temperature Laboratory in the "as received" condition, to be used in event of later queries.

(iv) After receipt of the bulk batch of material it is preaged in a complete batch, for 2 hours at 500 °C. The sample thermocouple already tested undergoes the same preaging with the batch.

(v) On completion of the preaging procedure the bulk batch of thermocouple material is returned to the manufacturer for manufacture of the required length load thermocouples. The sample thermocouple is re-calibrated to provide "preaged" calibration.

(vi) On delivery the completed thermocouples are checked at several temperatures to check conformance with the "preaged" calibration as established by the sample thermocouple. If acceptable a Work Log Sheet containing calibration correction factors is completed for the specific set of thermocouples.

(vi) The thermocouples then await issue on demand to the furnace centres.

(vii) Completed Work Log Sheets returned to the Temperature Laboratory provide record of usage.

(viii) Used thermocouples may be recovered and recalibrated in order to facilitate the collection of "work aged" drift data.

8.6.2 Calibration Compensation

At present calibration compensation on load thermocouples is done manually, by subtracting the correction factor from the indicated value. A system utilising automatic compensation has been proposed and is currently awaiting development.

8.6.3 Extension of the Concept

One of the considerations awaiting development is the proposal to grade thermocouples, simply, according to their basic calibration curve. In this proposal a Type K thermocouple having a calibration matching specific reference limits would be designated: Type K1, K2, K3 or K4, according to the particular limits matched.

8.7 CONCLUSION

The primary aim of the research into the use of preaging techniques as a means of stabilising drift in the Type K thermocouples used in the heat treatment furnaces at Hulett Aluminium Profiles has been successfully filled by the application of the proposed method. This research, in providing a means of accessing more accurate data relating to furnace performance under load conditions, has opened a number of avenues for future development leading to improved productivity.

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