

# **REDUCING ERRORS THAT LEAD TO UNCERTAINTIES IN THERMAL CONDUCTIVITY MEASUREMENT**

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## **ABSTRACT**

Thermal conductivity ( $k$ -value or  $\lambda$ -factor) is the fundamental driver of subsea thermal insulation system dimensions. It follows that an accurate determination of this parameter is a prerequisite for proper thermal design. Poor  $k$ -value data inevitably lead to inadequate system designs, which in turn can limit economic viability.

This paper provides an overview of the error sources inherent to a representative heat flow meter (HFM) method for measuring  $k$ , and their contribution to the accuracy and precision obtainable for the final experimental value. The paper also documents dramatic improvements in accuracy and reproducibility obtained using this method.

## **Section 1 INTRODUCTION**

Thermal insulation of oil and gas production and transport has become a tried and trusted flow assurance tool for subsea (and land-based) pipelines. Many hundreds of kilometres of insulated pipelines, based on single pipe and dual pipe designs, have enabled the reliable development and exploitation of hydrocarbon reserves. The current trend is toward greater dependence on thermal insulation, based either on a passive system or an active system supported by e.g. DC electrical power supply<sup>1</sup>. This enables progressively longer tiebacks and production to land, but greatly increases the need for proper system design.

The fundamental material property required for simple, steady state thermal design is thermal conductivity ( $k$ -value or  $\lambda$ -factor); a basic schematic of the steady state test is shown in Figure 1. For thermal design in transient conditions, density and specific heat capacity are also required; and to produce a comprehensive end-of-life design, taking into account factors such as hydrostatic pressure, creep collapse and water absorption, it becomes necessary to have a variety of other material properties. Nevertheless, the fact remains that the fundamental driver of subsea insulation system dimensions is thermal conductivity.

It follows that accurate  $k$ -value data are a prerequisite for thermal design; indeed, given the potential operational risk of an underperforming system, its importance can

hardly be overstated. On this basis, it would be natural to assume that  $k$ -value data, and the apparatus used to generate them, are subject to stringent critical review.

In fact, historically, there has been very little criticism of  $k$ -value data. This is partly because most of the errors associated with the test tend to produce an artificially low, and therefore pleasing, test result. Conversely, there may be considerable reluctance to accept genuinely accurate data, which tend to look disappointing by comparison.

The primary problem, however, seems to be a general, ongoing perception in the industry that the test is a straightforward one, and cannot be that difficult to carry out. The reality is that the relatively simple principles of the basic concept are greatly complicated by surface phenomena and non-linear heat flow (see Figure 1), making steady state  $k$ -value testing far more complex than it looks. Performing the test in such a way as to obtain genuinely accurate data is actually very demanding.

Furthermore, merely recognising that a set of  $k$ -value data may be inaccurate often requires considerable expertise, as there is no obviously intuitive way to assess a thermal conductivity measurement. Results obtained from a faulty methodology that would fairly quickly be identified as unacceptable in other areas of testing may go unquestioned for years provided the data are reasonably self-consistent.

It is therefore timely to review the sources of error associated with the steady-state technique used for measuring thermal conductivity, and the levels of uncertainty they tend to contribute. This paper will focus primarily on the heat flow meter (HFM) method, but makes reference to the guarded hot-plate (GHP) method.

This paper also documents significant advances in specimen preparation technique and instrument calibration, based on studies of error sources and error mitigation techniques, that have enabled thermal conductivity to be determined accurately and precisely to the 1mW (0.001W/mK) level.

## **Section 2 ERROR SOURCES IN K-VALUE DETERMINATION**

### **2.1 General Comments**

A widespread practice adopted over the years by groups offering  $k$ -value measurement services is to associate wide error bands with the results obtained: tolerances of 2.5-3.0% are often quoted, based largely on round robin studies. For a  $k$ -value in the 0.10-0.20W/mK range, a 3.0% error equates to an absolute uncertainty approaching  $\pm 0.005$ W/mK, making it essentially meaningless to cite the third decimal place. For the purposes of subsea insulation design, this is unfortunate: rounding up to the nearest 0.01W/mK translates to an increase in system cost of up to 10%; it also masks the effect of temperature, further impairing attempts at accurate system design.

In fact, the evidence from commercially obtained measurements is that a tolerance of 3.0% is optimistic: while not readily acknowledged, it is by no means unusual for the experimental error to exceed 10%. For a  $k$ -value in the 0.10-0.20W/mK range, this

equates to an absolute uncertainty capable of exceeding  $\pm 0.02\text{W/mK}$ , which is wholly unacceptable for any sort of quantitative design.

Equally, the assumption that  $k$  is a universal constant over an entire group of materials is equivalent to associating an error band of up to 10% with the experimental result. The  $k$ -value of polypropylene, for example, can vary from  $0.20\text{W/mK}$  to  $0.25\text{W/mK}$  depending on the commercial grade; hence, the assumption that  $k_{\text{PP}} = 0.22\text{W/mK}$  ‘creates’ an absolute uncertainty of  $\pm 0.02\text{W/mK}$ . Again, this is unacceptable.

## 2.2 Selection of Test Apparatus

The need to avoid unjustifiable assumptions about the value of  $k$  (see above) highlights a particular difficulty in the determination of  $k$ -values for subsea insulation materials. Obtaining an  $8'' \times 8'' \times 1''$  specimen from an insulation pipe coating is typically impossible due to the curvature of the pipe. Preparing a separate coupon in a mould may impart different properties to the material, including a different  $k$ -value. What is required is a test method capable of accurately testing small specimens.

It is generally accepted that the most reliable values of  $k$  are obtained by measuring in steady state conditions (transient methods allow somewhat faster measurement and do not require as much in the way of sample preparation). The steady state technique most commonly used for commercial  $k$ -value measurement is the heat flow meter (HFM) method; the guarded hot-plate (GHP) method, an absolute technique, is both slower and more expensive, and tends to be used to characterise reference materials.

An HFM apparatus capable of testing 5.0mm thick disc specimens 50mm in diameter, and suitable for testing in the  $0.1\text{-}10\text{W/mK}$  range<sup>(1)</sup>, has been commercially available since 1999 and has acquired an increasing track record over the last five years. HFM error sources quoted in the remainder of this paper are appropriate to this instrument only; furthermore, unless the text specifically states otherwise, all subsequent references to  $k$ -value measurement apparatus refer to this instrument.

## 2.3 Sources of Experimental Error

When measuring the heat flow through a planar specimen of material in steady state conditions (see Figure 1),  $k$  can be determined using a rearranged form of the integrated Fourier equation:-

$$Q/A = \frac{\Delta T}{(\Delta x/k)} \dots\dots\dots(1)$$

where:

- $k$  = thermal conductivity<sup>(2)</sup> calculated for the test specimen ( $\text{Wm}^{-1}\text{K}^{-1}$ )
- $Q/A$  = heat flow per unit area (also defined as the *heat flux*,  $q$ ) ( $\text{Wm}^{-2}$ )

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<sup>(1)</sup> the FOX50 HFM, developed by Lasercomp Incorporated, Saugus MA, USA

<sup>(2)</sup> the parameter  $(\Delta x/k)$  is defined as the *thermal resistance* ( $\text{m}^2\text{KW}^{-1}$ ) encountered by the instrument

$\Delta x$  = thickness of the test specimen (m)

$\Delta T$  = temperature difference across the test specimen (K)

In order to calculate  $k$ , there are three separate variables to measure:  $Q/A$ ,  $\Delta x$  and  $\Delta T$ . Hence, for an HFM instrument, there are four basic sources of experimental error:  $Q/A$ ,  $\Delta x$ ,  $\Delta T$  and loss of steady-state conditions.

### 2.3.1 Error Sources associated with Loss of Steady-State Conditions

The problem of deviation from steady-state conditions is entirely instrument related. The specific error sources are temperature variation and non-linear heat flow.

Temperature variation at contact surfaces can occur in two forms: (a) a progressive drift in the overall temperature, and (b) a temporary fluctuation across the platen surface. For the instrument under discussion, it is stated that, at 20°C, each platen is able to maintain an isothermal contact temperature to a resolution of  $\pm 0.01^\circ\text{C}$ .

Assuming a specimen of uniform material, with both contact surfaces maintained at isothermal temperatures, non-linear heat flow is only likely to occur around the edge of the specimen (see Figure 1). For  $k \geq 0.1\text{W/mK}$ , the thermal resistance of the test material will also be relatively low in comparison to the surroundings, making that material the preferred route for a flux of heat energy. In an absolute technique such as the GHP method, edge losses resulting from non-linear heat flow would still be considered a major source of error under these circumstances. However, the normalising effect of the calibration makes edge losses far less important for HFM instruments, assuming that the reference material has a similar heat flow pattern; and edge losses for the apparatus under discussion are usually considered negligible.

For our application however, where the third decimal place was considered important, it proved to be necessary to upgrade the insulation material inside the guard cylinders (see Section 4.1) in order to reduce edge losses when testing above 40°C.

### 2.3.2 Error Sources associated with $Q/A$

HFM instruments measure  $Q/A$  using thin heat flux sensors, one embedded in each instrument platen. The heat flux sensor designs for this particular instrument are proprietary, making it difficult to comment in detail. However, the raw sensitivity of the sensors is a simple matter of analogue/digital signal resolution:  $\pm 1\mu\text{V}$  per sensor, equating to a total relative error of just over 0.02%, which is negligible.

The primary “error” associated with  $Q/A$  is that the sensor output is proportional to the heat flux, and requires a conversion factor,  $S_{\text{cal}}$  ( $\text{Wm}^{-2}\mu\text{V}^{-1}$ ). Generating  $S_{\text{cal}}$  requires a reference material of known  $k$ -value, with which to interpret the microvolt signals (i.e. calibrate the instrument). The reference material itself will have to have been previously characterised (using the GHP method), so a numerical value for  $S_{\text{cal}}$  effectively contains *two whole experiments*, with all the uncertainty that implies; the cumulative uncertainty contributed to the final  $S_{\text{cal}}$  value can easily exceed  $2 \times 5.0\%$ .

### 2.3.3 Error Sources associated with $\Delta x$

$\Delta x$  is the one source of error that is entirely specimen related, and thus entirely within the control of the operator. Obtaining an average  $\Delta x$  value for a given specimen is straightforward enough; the problem is the standard deviation around that average. The various sources of error, whether macroscopic (lack of planarity, in the form of undulations or hollows, or lack of parallelism) or microscopic (lack of smoothness) can be gathered together under a single heading: specimen preparation tolerance.

For a  $\Delta x$  value of 5.0mm, a tolerance of  $\pm 0.05$ mm (which would normally be considered 'good' for the polymer resins on which subsea thermal insulation systems are based) creates a relative error of 1.0%. The specimen preparation tolerance therefore constitutes a significant source of error when measuring the  $k$ -value of relatively thin specimens of material removed from a pipe coating. This subject is sufficiently important that it will be dealt with in its own right (see Section 3).

### 2.3.4 Error Sources associated with $\Delta T$

There are a number of error sources associated with the measurement of  $\Delta T$ , all of them basically related to the Type E thermocouples used by the instrument under discussion to measure  $T_1$  and  $T_2$  ( $\Delta T = T_1 - T_2$ ).

The first, and most fundamental, error source is metallurgical. Relative metallurgical variation is virtually eliminated by using adjacent lengths of wire from the supplied spool – paired thermocouples prepared in this way should have the exact same offset (if any) from the 'ideal' composition. Regarding absolute accuracy, the official figure given for the material is  $\pm 1.5^\circ\text{C}$ . Assuming a relatively large temperature dependence for  $k$  (e.g. 0.003W/mK over  $20^\circ\text{C}$ ), this figure yields a nominal relative error of 0.15%, equating to an absolute uncertainty that rounds to  $\pm 0.00025$ W/mK.

The second error source is sensitivity: the instrument has an analogue/digital resolution of  $\pm 0.01^\circ\text{C}$ , which, for a  $\Delta T$  of  $20^\circ\text{C}$ , yields a relative error of 0.05% per thermocouple; the use of one thermocouple per platen doubles this error, leading to a total absolute uncertainty that rounds to  $\pm 0.00015$ W/mK. Temperature sensitivity is considered to be the limiting factor in current instrument performance.

When using externally mounted thermocouples to measure  $\Delta T$ , a third error source to consider is the drop in temperature,  $\delta T_r$ , associated with  $r$ , the thermocouple radius. In order to control this error, it is recommended that a wire diameter of no more than 0.2mm be used<sup>2</sup>; if possible, this should be flattened to a thickness of only 0.1mm. The error contribution can be considerable: in the process of generating the results presented in this paper, it was found that using a cylindrical thermocouple diameter of 0.65mm to measure  $\Delta T$  in GHP testing produced a 5.0% distortion in the absolute experimental  $k$ -value (see Section 4.2). Embedding the thermocouples within instrument platens for physical protection (the normal practice in the HFM method – externally mounted 0.1mm thick thermocouples are *extremely* fragile) is equivalent to significantly increasing the value of  $r$  and, hence, the error contribution from  $\delta T_r$ .

When using embedded thermocouples, a fourth error source to consider is the imperfect physical contact between the specimen and instrument platen surfaces. This creates a further temperature drop,  $\delta T_{SCR}$ , described as the Surface Contact Resistance (SCR); the phenomenon may be thought of as a thin layer of ‘foam’ ( $k \approx 0.03\text{W/mK}$ ) occurring between the bulk specimen material and the bulk platen material. As such,  $\delta T_{SCR}$  is negligible for most land-based insulation materials, where  $k \approx 0.03\text{W/mK}$ ; the threshold value above which it starts to become significant is thought to be  $0.1\text{W/mK}$ . Comparative studies on a syntactic material with  $k = 0.15\text{W/mK}$  suggest a relative experimental error of 2.0-4.0%, depending on the surface finish of the test specimen.

## 2.4 The HFM “Two-Thickness” Method<sup>3</sup>

Where embedded thermocouples have been employed in the HFM method, distinguishing between the effect of surface contact and the effect of the thermocouple radius is not normally practical; all that can be said is that there is an overall drop in temperature from the specimen surface to the centre of the embedded thermocouple. The contribution from  $\delta T_{SCR} + \delta T_r$  is far more significant than that of any other measurement error associated with  $\Delta T$ , and can also be far more difficult to assess, being intimately bound up with the method.

The Two-Thickness method treats the aggregate error associated with  $\delta T_{SCR} + \delta T_r$  as a single quantity,  $R$ , defined as the additional thermal resistance occurring between the specimen surface and the embedded thermocouple:-

$$Q/A = \frac{\Delta T}{[(\Delta x/k) + 2R]} \dots\dots\dots(2)$$

where:

- $Q/A$  = heat flow per unit area (heat flux) ( $\text{W.m}^{-2}$ )
- $\Delta T$  = temperature difference between the instrument platens (K)
- $\Delta x/k$  = thermal resistance of the bulk specimen material ( $\text{m}^2.\text{K.W}^{-1}$ )
- $R$  = additional thermal resistance ( $\text{m}^2.\text{K.W}^{-1}$ )

In order to distinguish  $\Delta x/k$  from  $R$ , it is first of all necessary to assume identical values of  $R$  for either side of the test specimen (i.e.  $2R$  as opposed to  $R_1 + R_2$ ). For this assumption to be valid, it is necessary to ensure firstly that the specimen has the same reproducible surface finish on both contact surfaces, and secondly that each thermocouple is as similar as possible to its twin embedded in the opposite platen.

Assuming that two specimens with significantly different thicknesses,  $\Delta x_1$  and  $\Delta x_2$ , can be prepared from a given material, with the same reproducible finish on both contact surfaces, testing these specimens separately using the same  $\Delta T$  will result in significantly different heat flux values,  $Q_1/A$  and  $Q_2/A$ , for each specimen. An experimental value of  $2R$  can then be isolated by mathematically solving a system of simultaneous equations based on Equation 2, above, allowing a truly accurate value of  $k$  to be back-calculated. For a properly prepared specimen of foamed polypropylene ( $\rho = 700\text{kg/m}^3$ ,  $k = 0.17\text{W/mK}$ ), the contribution from  $2R$  is approximately 7.0%.

## Section 3 SPECIMEN PREPARATION TOLERANCE

### 3.1 General Comments

The very considerable difficulty of machining polymers is not universally appreciated.

The problems begin with thermal sensitivity, which is far more acute for polymers than it is for metals: a temperature rise of only 40-50°C can relieve internal stresses in polypropylene, whereas a stress relieving operation for a carbon steel requires a heat input of 400-500°C. Polymers also have thermal expansion coefficients roughly 10 times higher than metals and are 100 times slower to disperse a build up of heat. Moreover, given that the coolants used in traditional operations have a tendency to cause polymers to swell, despite the above problems it is preferable to machine dry.

Mechanically, polymer fracture behaviour is such that there is a tendency for the tool to tear rather than cut, or for cut material to remain adherent to the surface, forming a rag that has to be scraped away before further cutting can take place. Unreinforced polymers also have moduli approximately two orders of magnitude lower than that of steel. Conventional gripping techniques, which assume the material is fairly stiff, are therefore likely to cause them to distort (a bulging surface that has been successfully machined flat will become hollowed once the pressure is released, the worst form of deviation from planarity when designing an HFM  $k$ -value test specimen).

Finally, the main commercial and operational drivers in polymer processing tend to be cost reduction. This has led to advances in moulding technology rather than machining (machining typically being viewed as an expensive overhead). As such, the technology and techniques developed for machining polymers generally lag behind their metal equivalents by a significant margin.

The upshot of all these problems is that, for polymer specimens, it is commercially impractical to expect a machined thickness tolerance to be significantly better than  $\pm 0.05\text{mm}$  (conversely, for 1-2"  $\phi$  hardened steel specimens, a tolerance of  $\pm 0.001\text{mm}$  is easily obtainable). As stated in Section 2.3.3, a tolerance of  $\pm 0.05\text{mm}$  creates a measurement error in excess of 1.0% for a specimen thickness of 5.0mm.

### 3.2 Overview of Specimen Preparation Methodology

For the most part, specimen preparation was carried out at a chosen testing facility<sup>(3)</sup> with specialised experience in machining unreinforced polymers.

Based on the theoretical requirements of the "Two-Thickness" method, thickness values of 5.0mm and 12.0mm respectively were arbitrarily selected for  $\Delta x_1$  and  $\Delta x_2$ . A tolerance of  $\pm 0.005\text{mm}$  (5 microns) was set in each case, in order to restrict the relative measurement error to a maximum of 0.1%.

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<sup>(3)</sup> Materials Engineering Ltd, Aberdeen, UK

In order to meet the above tolerance, the problem of lateral distortion due to excessive pressures applied while securing the sample had to be overcome. A proprietary gripping technique was developed that applied an even pressure across the sample equivalent to a force of 40N (4kg), sufficient to prevent displacement during machining from shear forces brought about by contact with the cutting tool. This technique produced essentially zero distortion in the final, machined specimen.

In order to remove the tiny quantities of material required to meet such a tolerance, it was also necessary to use a parallel cutting technique. Flat, parallel surfaces were achieved using a removal rate of 0.25mm per pass. Once parallel, specimen blanks were reduced to the final thickness using a removal rate of 0.05-0.01mm per pass. Proprietary cutting tools had to be developed from first principles for this purpose; these would be unsuitable for machining metals.

Temperature measurements carried out between passes showed the heat input to be approximately 1-2°C. Careful inspection of the final machined surfaces revealed them to be essentially free of mechanical distortion, with a highly reproducible finish.

### 3.3 Worked Example: Preparation of PMMA Reference Specimens

Heat flow meters are calibrated using machined specimens of previously characterised reference materials. Cast polymethyl methacrylate (PMMA) was adopted for use as a subsea insulation reference, based on its relatively low  $k$ -value (see also Section 4).

A 5.0mm disc specimen and a 12.0mm disc specimen were machined from a single 305mm × 305mm × 31mm PMMA block, initially using a conventional technique. The resulting disc specimens, although unusually good bearing in mind the difficulties encountered when machining polymers (see Section 3.1), had to be rejected on the basis of poor data reproducibility. Both specimens were therefore re-machined using the proprietary technique. The challenge in this case, given that the specimens had *already* been machined to size, was the need to restrict the overall thickness reduction to no more than 0.2mm. Final thickness dimensions, before and after re-machining, are shown in Tables 1a and 1b respectively.

## Section 4 CHARACTERISATION AND DEVELOPMENT OF A SUBSEA INSULATION REFERENCE MATERIAL

### 4.1 Background

The HFM method is not absolute, and requires the instrument to be calibrated using previously characterised reference materials (see also Section 2.3.2). The materials originally offered by the instrument supplier were 'Pyrocera 9606' ( $k = 4.0\text{W/mK}$ ), 'Pyrex 7740' ( $k = 1.1\text{W/mK}$ ) and 'Vespel SP1' ( $k = 0.37\text{W/mK}$ ). None of these were considered to be suitable reference materials for HFM-based characterisation of subsea insulation materials, partly because, in each case, the  $k$ -value lay outwith the

desired range ( $k = 0.1\text{-}0.2\text{W/mK}$ ), and partly because, in each case, the uncertainty inherent to the official values was not accepted to be better than 3-5%<sup>(4)</sup>.

At the time this work commenced, the UK National Measurement Institute (NMI)<sup>(5)</sup> had recently qualified, and made available for reference purposes, a speciality grade of PMMA, cast as a panel between sheets of high quality glass. This material had not yet been characterised as part of an international round robin between NMIs, and offered only a relatively limited temperature range, being too soft at 80°C to maintain its original dimensions. However, the  $k$ -value as characterised by the UK NMI lay within the desired 0.1-0.2W/mK range ( $k = 0.19\text{W/mK}$ ), meaning that the heat flow pattern would be similar to that observed in subsea insulation materials.

The UK NMI PMMA was therefore adopted as a candidate reference material. Note that, as with other polymers, PMMA can display a variety of  $k$ -values, depending on the commercial grade and processing history; the following discussion is necessarily limited to material traceable to the original UK NMI casting.

## 4.2 GHP Characterisation of Cast PMMA

The UK NMI was commissioned to formally characterise two blocks of PMMA cut from the original cast panel (identified respectively as QM315A and QM315B) using their GHP apparatus. The Canada NMI<sup>(6)</sup> was then commissioned to re-characterise the blocks, using their own GHP apparatus that had been constructed to a different design, so that the raw variation in absolute experimental data could be compared. The values generated are cited in Table 2, and documented in separate reports<sup>4,5</sup>.

The discrepancy in the initial values, while disappointing, was at least too large to be written off as unavoidable experimental error. The Canada NMI data was suspect, being the lower of the two results, and the problem was identified as the 0.65mm diameter thermocouples used in the Canada NMI GHP (see also Section 2.3.4.4). The Canada NMI performed two re-tests, see Table 2, using 0.1mm flattened thermocouples supplied by the UK NMI. This yielded values indistinguishable from the UK NMI data when measuring to three decimal places; this level of precision had not been achieved before in round robin GHP testing.

## 4.3 Preparation of Machined HFM PMMA Test Standards

The next stage in development was to generate correct HFM  $S_{\text{cal}}$  values (see also Section 2.3.2) using machined specimens removed from one of the PMMA blocks. Initial attempts to generate an aggregate  $S_{\text{cal}}$  from a series of five in-house calibrations highlighted disappointing levels of reproducibility compared to that achieved in the round robin GHP testing. This was unacceptable, as the impressive reproducibility of the GHP testing would effectively be lost if it could not be effectively transferred to the HFM calibration process. The problem was believed to be the specimen tolerance

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<sup>(4)</sup> based on the variation in results observed in round robin GHP testing between various NMIs

<sup>(5)</sup> the UK NMI is known as the National Physical Laboratory (NPL)

<sup>(6)</sup> the Canada NMI is known as the National Research Council Canada (NRCC)

(a conventional machining technique had been used to save time), and the decision was therefore taken to have the specimens re-machined (see also Section 3.3).

The use of re-machined specimens produced a dramatic improvement in reproducibility, confirming the importance of an exacting thickness tolerance. The test results for a second aggregate  $S_{cal}$ , based on a repeat series of five in-house calibrations, are documented in Table 3.

As can be seen in Table 3, although the improved  $S_{cal}$  was acceptable for use at 20°C, the experimental results showed a non-linear variation with temperature. This finding was confirmed by a comparison of interim  $k$ -value test results based respectively on (a) the improved PMMA  $S_{cal}$ , and (b) a traditional ‘Vespel SP1’  $S_{cal}$ .

The above shortcoming could not realistically be attributed to either the original GHP values (see Section 4.1) or the specimen tolerance (see Section 3.3); hence, the performance of the HFM instrument had to be brought into question. Given that the problem was mostly occurring at 60°C, a deviation from steady-state conditions due to non-linear heat flow – i.e. edge losses – was suspected (see also Section 2.3.1). The instrument supplier therefore offered to upgrade the insulation in the upper and lower guard cylinders (designed to isolate the platen/specimen/platen test system from the outside environment) as part of a general upgrade/maintenance programme.

At this time, the supplier was also asked to amend the test software to include a fourth decimal place in the experimental result, the aim being to monitor statistical variance in the in-house data (it was not meaningful to quote a standard deviation when viewing the result to only three decimal places, as there was essentially no variation).

Bearing in mind the minute levels of precision now being aimed for, the instrument supplier recommended using an individual calibration to generate  $S_{cal}$ , as opposed to a statistical average that would naturally include one or two outliers. A further series of five in-house calibrations was therefore performed, but this time each one was individually assessed for accuracy<sup>(8)</sup>, beginning with the most likely candidate as judged by the supplier. The results of the overall survey are documented in Table 4.

As can be seen from Table 4, the fourth calibration produced acceptable  $S_{cal}$  values for 40°C and 60°C, but did not produce an acceptable  $S_{cal}$  value for 20°C; fortunately, the third calibration was subsequently found to be appropriate for this temperature.

When bringing experimental data under this degree of scrutiny, the question that will always be asked is, to what degree is that data reproducible. In fact, as shown in Table 5, while assessing the third calibration, the variation in  $k$ -value data at 20°C was revealed to be no more than 0.0001W/mK, rising to 0.0003W/mK at 60°C. This number variation has been repeatedly confirmed; indeed, recent indications are that it has been improved upon, due to further refinements in the test methodology.

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<sup>(8)</sup> given that the official GHP data for the PMMA had only been expressed to three decimal places, it was felt best to arbitrarily add a ‘0’ to each value (e.g. 0.189W/mK → 0.1890W/mK)

## Section 5 DISCUSSION OF TEST METHOD, AND WORKED EXAMPLES RELEVANT TO THERMAL DESIGN

### 5.1 Discussion of Test Method

A combination of precision to four decimal places and accuracy to three decimal places points to a negligible contribution from most of the error sources described in Sections 2 and 3 of this paper, at least compared to historical levels of uncertainty. Subsea insulation design requires data accurate to three decimal places in order to minimise the effect of  $k$ -value on system cost, and this has now been achieved.

Making more specific reference to Section 2.3.4 of this paper, the official figure for the metallurgical accuracy of the thermocouple material has to be conservative, at least for the thermocouples in this particular HFM instrument. An absolute variation of  $\pm 1.5^\circ\text{C}$  could feasibly lead, in itself, to an uncertainty of  $\pm 0.0002\text{W/mK}$ ; the reality is that the cumulative uncertainty of the entire test is slightly less than  $\pm 0.0001\text{W/mK}$ .

Making reference to Section 2.4, the Two-Thickness method manages to sidestep the error contributed by the thermocouple radius, by combining it with surface errors into a single variable,  $R$ , the additional thermal resistance. However, the following cautionary points need to be made:-

- for the technique to be physically valid, the  $\Delta x_1$  and  $\Delta x_2$  specimens must each have the same tolerance and surface finish on both contact surfaces
- using  $2R$  as a mathematical input to isolate a ‘true’ value for  $k$  effectively adds an extra layer of experimental error to the final, calculated result, making error mitigation techniques essential to avoid cumulative uncertainty
- if external thermocouples (considered to be the ‘best practice’ for both HFM and GHP methods) are used instead of an artificial system of simultaneous equations, then the thermocouple radius error,  $\delta T_r$ , must not be ignored

### 5.2 Worked Examples Relevant to Subsea Insulation Design

Two commercial grades of polypropylene have been characterised using the HFM method described in this paper. The data are presented in Figure 2.

## Section 6 REFERENCES

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- 3 A. Brzezinski, A. Tleoubaev: “EFFECTS OF INTERFACE RESISTANCE ON MEASUREMENTS OF THERMAL CONDUCTIVITY OF COMPOSITES AND POLYMERS” Proceedings of the 30th North American Thermal Analysis Society Conference, September 23-25, 2002, Pittsburgh, PA, USA, K. J. Kociba, ed., Lubrizoil Corp. (pp.512-517)
- 4 NPL Certificate of Calibration: “THERMAL CONDUCTIVITY OF PERSPEX”; Reference PR44/E03120068, issued January 2004
- 5 NRCC Client Report: “THERMAL PROPERTIES OF ONE (1) PAIR OF ‘PERSPEX’ PMMA SPECIMENS”; Reference B-1166.1, issued August 04, 2004

5.0mm & 12.0mm $\Delta x$ DIMENSIONS BEFORE RE-MACHINING					
<b>5.0mm (<math>\Delta x_1</math>) Specimen Dimensions (mm)</b>					
Side ( $x_a$ )	Side ( $y_a$ )	Metering ( $C_a$ )	Side ( $x_b$ )	Side ( $y_b$ )	
5.286	5.282	<b>5.268</b>	5.285	5.282	
<b>12.0mm (<math>\Delta x_2</math>) Specimen Dimensions (mm)</b>					
Side ( $x_a$ )	Side ( $y_a$ )	Metering ( $C_a$ )	Side ( $x_b$ )	Side ( $y_b$ )	
<b>12.031</b>	12.044	12.040	<b>12.054</b>	12.047	
5.0mm specimen unacceptably concave : $\Delta v.(x_a x_b y_a y_b) - C_a =$ <b>16 microns</b>					
12.0mm specimen unacceptably off-parallel : $x_b - x_a =$ <b>23 microns</b>					

5.0mm & 12.0mm $\Delta x$ DIMENSIONS AFTER RE-MACHINING					
<b>5.0mm (<math>\Delta x_1</math>) Specimen Dimensions (mm)</b>					
Side ( $x_c$ )	Side ( $y_d$ )	Metering ( $C_b$ )	Side ( $x_c$ )	Side ( $y_d$ )	
5.007	5.006	5.007	5.008	5.006	
<b>12.0mm (<math>\Delta x_2</math>) Specimen Dimensions (mm)</b>					
Side ( $x_c$ )	Side ( $y_d$ )	Metering ( $C_b$ )	Side ( $x_c$ )	Side ( $y_d$ )	
12.014	12.016	12.016	12.015	12.014	
5.0mm specimen total thickness variation = <b>2 microns</b> (tolerance = $\pm 1$ micron)					
12.0mm specimen total thickness variation = <b>2 microns</b> (tolerance = $\pm 1$ micron)					

**Tables 1a & 1b:  $\Delta x$  dimensions before and after re-machining**

Test Temperature	Experimentally Obtained Values (W/mK)		
	UK NMI	Canadian NMI	
	First Value	First Value	Re-test Value
20°C	0.189	0.179	<b>0.189</b>
40°C	0.191	0.181	–
60°C	0.193	0.182	<b>0.193</b>

**Table 2: NMI values for speciality grade cast PMMA**

Temperature	Experimental Result	'Correct' (GHP) Value
20°C	0.189 W/mK	0.189 W/mK
40°C	0.191 W/mK	0.191 W/mK
60°C	<b>0.196</b> W/mK	0.193 W/mK

**Table 3: experimental results for re-machined PMMA specimens**

Test Temperature	20°C	40°C	60°C
<b>GHP Value</b>	<b>0.1890 W/mK</b>	<b>0.1910 W/mK</b>	<b>0.1930 W/mK</b>
1 <sup>st</sup> Calibration	0.1883 W/mK	0.1905 W/mK	0.1926 W/mK
4 <sup>th</sup> Calibration	0.1895 W/mK	<b>0.1910 W/mK</b>	<b>0.1929 W/mK</b>
5 <sup>th</sup> Calibration	0.1881 W/mK	Not Required	Not Required
3 <sup>rd</sup> Calibration	<b>0.1889 W/mK</b>	Not Required	Not Required
2 <sup>nd</sup> Calibration	Not Required	Not Required	Not Required

**Table 4: experimental data obtained using individual calibration factors**

Test Temperature	20°C	40°C	60°C
Replicate #1	0.1887 W/mK	0.1903 W/mK	0.1927 W/mK
Replicate #2	0.1888 W/mK	0.1902 W/mK	0.1924 W/mK
Reproducibility	<b>+0.0001 W/mK</b>	<b>-0.0001 W/mK</b>	<b>-0.0003 W/mK</b>

**Table 5: reproducibility for 5.0mm ( $\Delta x_1$ ) specimen test results (3<sup>rd</sup> calibration)**

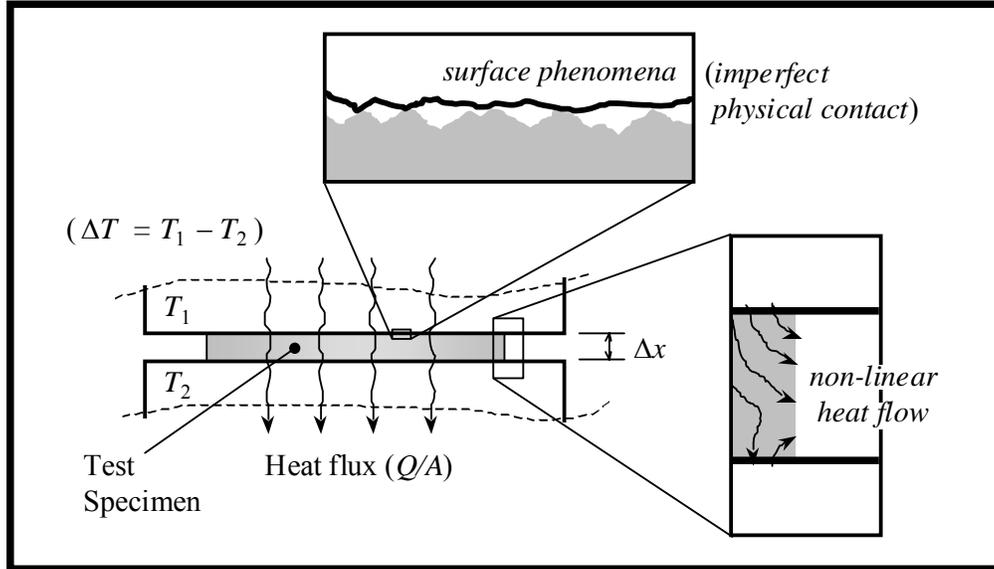


Figure 1: schematic of steady state  $k$ -value test, plus complicating factors

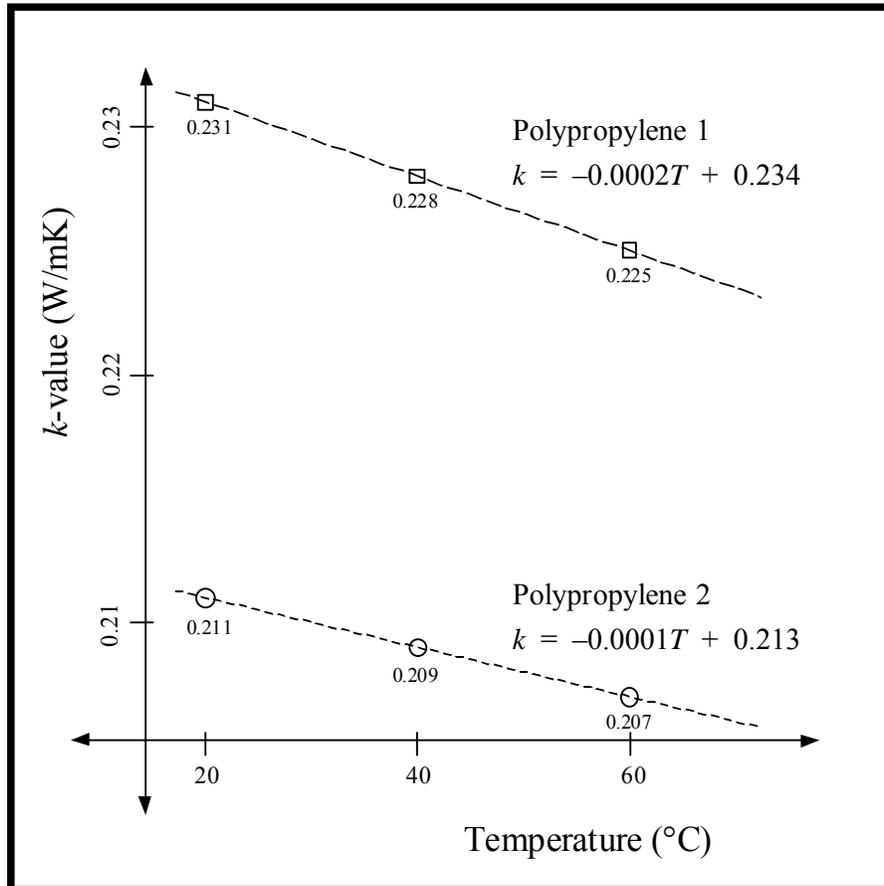


Figure 2: temperature behaviour of two commercial PP grades