Glass–PTFE stopcocks for use at elevated temperatures and pressures

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Abstract We describe a modification to commercial glass–PTFE stopcocks to permit their use at elevated temperatures and pressures.

Many operations in the physical sciences are facilitated by the use of greaseless stopcocks. For many years, greaseless taps with neoprene or Viton diaphragms have been used (G Springham and Co., Harlow, CM20 2AZ). These have a fairly good vacuum performance and can be operated satisfactorily in a hot-box by means of easily fitted extension handles (e.g. Dabbagh *et al* 1975, Haszeldine *et al* 1970). The diaphragms are not immune from attack, however, and we have experienced considerable difficulties, both with perishing by corrosive gases and with absorption and subsequent release of relatively innocuous compounds; the latter problem completely vitiates the use of diaphragm taps in some analytical systems.

More recently, the use of greaseless taps has spread rapidly with the advent of the cheaper and less cumbersome taps using a PTFE plug, or PTFE O-rings on a glass plug, the plug being forced onto its seating by a plastic cap screwing onto a glass thread cast on the body of the tap. These overcome virtually all problems of chemical reactivity but can be operated as purchased at only a moderately elevated temperature, such as 50°C, since above this temperature the caps distort and slip on the glass thread, particularly when the internal pressure exceeds atmospheric. Furthermore, if the taps are situated in a hot-box, or inside a protective cabinet for use under pressure, they have to be operated by extension handles, and these are not readily fitted to the plastic caps. Replacement of the plastic caps by metal versions is not in itself satisfactory, since the glass thread is found to chip or crack within a short period of use.

We have overcome these problems by using glass-plug/PTFE-O-ring taps (J C Young (Scientific Glassware) Ltd, London W3 8BS) modified by the provision of a brass cap which operates, not on the glass thread, but on a sleeve of glass-reinforced PTFE material, 'Rulon'†, as shown in figure 1. The Rulon sleeve A is threaded on both sides, the inside gripping the tap body B tightly, and being bonded to the glass with Araldite epoxy resin G. The outside is threaded to enable the metal cap C to screw smoothly onto the tap; a suitable thread is ½ in BS pipe thread, and it is desirable to rethread the outside after cementing the sleeve in place. Extensions for remote operation are easily connected to the metal cap by a flexible coupling.

Taps with these modifications have been tested hydraulically to 5 MPa without any case of rupture and have been used successfully for over a year in a hot-box at 130°C with gas pressures up to 1·3 MPa. One limb of the tap is connected to glassware in a furnace at *ca* 400°C, so that the sealing O-ring (not shown in the figure) operates at 250°C and needs replacing monthly; the upper O-rings (at 130°C) have not yet needed changing. The taps retain good vacuum performance and have a leakage rate of only 0·4 mPa s^{-1} from a volume of 0·3 l at 1·3 MPa internal pressure.

† Manufactured under licence from the Dixon Corporation and supplied by Henry Crossley (Packings) Ltd, Bolton BL1 6FB.

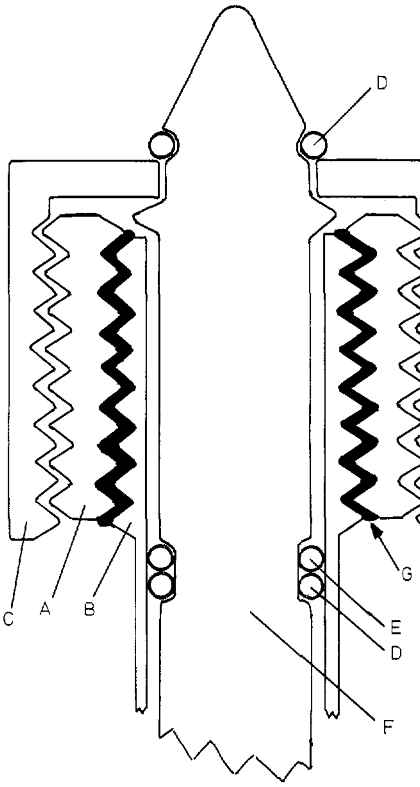

Figure 1 Section of modified tap head: A, Rulon sleeve; B, glass body of tap; C, brass cap; D, PTFE O-ring; E, Viton O-ring; F, glass plug of tap (gas seal at lower end, not shown); G, Araldite

References

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A simple conversion formula for type 'T' (copper–constantan) thermocouple readings

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Abstract A simple formula is given for the calculation of junction temperature from the measured EMF of a type 'T' (copper–constantan) thermocouple. The formula is accurate to better than 0·1°C over the range −200°C to +400°C, and requires only eight arithmetic operations for its evaluation.

Thermocouples of copper with a copper–nickel alloy are convenient for temperature measurement in the range −200°C to +400°C. The relationship between junction temperature and measured EMF is given in standard reference tables (BS 4937, BS 1828).

The direct use of these tables is especially inconvenient when the conversion is to be done automatically on a computer. If the interpolation algorithm is kept simple, a large table must be held in storage. Alternatively, if fewer points are stored, a more complex interpolation algorithm must be used.

A neater solution employs a mathematical function which adequately approximates the conversion table. Polynomials are often used for this purpose, since over a limited range the table may be approximated to any desired accuracy by a polynomial of sufficiently high order. The coefficients for a least-squares fit can be calculated explicitly.

Many functions, however, although smooth, do not resemble polynomials in their behaviour. Very high-order polynomials are needed to approximate them, and the terms in the power expansion must be calculated to high precision to obtain accurate cancellation of oscillating terms.

In these cases it is better to look for simple functions other than polynomials which more nearly resemble the tabulated relationship. The form of the calibration curve for type 'T' thermocouples, shown in figure 1, suggested hyperbolic terms.

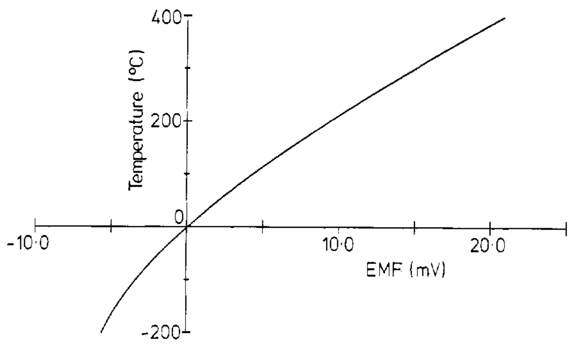

Figure 1 Calibration curve for type 'T' thermocouple (reference at 0°C)

Some trial and error work using a flexible least-squares minimization routine† led to the function given in table 1. Two sets of coefficients have been computed, one corresponding to the international table for type 'T' thermocouples (BS4937 part 5) and the other to the older BS1828 for copper–constantan, which differs from BS4937 slightly but significantly. (The BS4937 tables are identical to those published in the 1973 American Bureau of Standards Monograph 125.) Many thermocouples still in use were manufactured to the older specification. The error of the approximating function with respect to BS4937 is shown in figure 2 and is everywhere less than 0·1 degree in the range −200°C to +400°C. There is little point in calculating to a greater accuracy, since larger errors arise from manufacturing tolerances in the thermocouple alloy. The errors in the fit to BS1828 are even smaller. Below −200°C, the errors rapidly increase.

To achieve similar accuracy over the same range using a polynomial requires a formula of at least 12th order which must be evaluated with an internal precision at least four orders greater than the precision of the result. The formula of table 1, in contrast, requires only eight arithmetic operations per evaluation, each with precision only slightly greater than the result precision. The formula is simple enough to evaluate on a pocket calculator and presents a trivial task to a computer.

† NAG Library routine E04FBA, Numerical Algorithms Group, Oxford University Computing Laboratory.

Table 1 Simple formula and coefficients for type 'T' (copper–constantan) thermocouples

	BS4937 (part 5) ($-200°C<T<+400°C$)	BS1828 ($-190°C<T<+400°C$)
A	238·29	257·65
B	13·364	13·346
C	4776·4	5597·9
D	21·867	24·061
E	154·83	201·35
F	7·8242	8·0390

$$T=A+BV-C/(V+D)-E/(V+F)$$

where T is the junction temperature in °C, V is the junction EMF in mV (with respect to reference junction at 0°C).

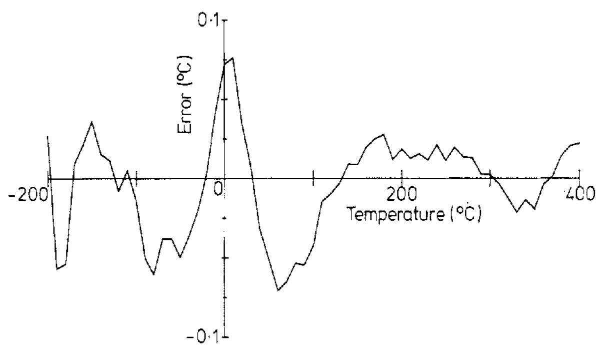

Figure 2 Error of the formula given in table 1 with respect to BS4937 part 5

Acknowledgment
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References

British Standards Institution 1961 BS1828

British Standards Institution 1974 BS4937