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ON-LINE VALIDATION OF DENSITOMETERS BY PRESSURE PYKNOMETRY

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INTRODUCTION

In a Mass transfer accounting system the measurement of the density of the fluid is of equal importance to the measurement of the volume of fluid transferred. It is therefore of equal importance to have the capability to check the calibration of on-line density meters as it is to prove the volumetric meters and to calibrate the master provers.

Since all density measurement devices in the North Sea rely on the variation in fundamental frequency of vibration of a tube or vane with changing density it is important to have an independent means of determining the fluid density. High pressure Pyknometry provides such a method fundamentally different from that used on-line.

Using purpose built, mobile, pyknometry systems, Moore Barrett and Redwood have carried out the validation of densitometers calibrations on Fiscal systems throughout the North Sea. The method has also been used successfully on crude oil, condensate, NGL and petroleum products.

This paper reviews the progress in equipment design and construction and improvements in the methods of obtaining repeatable and representative results under adverse conditions. It also reviews the method of pyknometer calibration adopted by MB+R which allows the overall uncertainty of the method to be reduced.

SUMMARY OF METHOD

To validate the calibration of a density meter at its operating conditions by pyknometry, a sub-sample of the fluid passing through the densitometer must be captured within the pyknometer. Co-ordination of the sampling and the reading of the densitometer must be precise.

Conditioning of the pyknometers to line conditions of temperature and pressure are of the utmost importance. Therefore accurate measurement of pressure and temperature within the conditioning skid are important.

As the density meter sees only a small portion of the total fluid in the main line and the pyknometer captures a small sub-sample of the fluid flowing through the densitometer, the importance of representative samples within the pyknometers cannot therefore be overstressed.

The filled pyknometers are removed to a suitable site laboratory where the outside surfaces are thoroughly cleaned and dried. The pyknometer is then weighed. Having previously determined the weight of the empty pyknometer at local conditions, and knowing the internal volume and expansion coefficients for the pyknometers, the density at line conditions can be calculated.

EQUIPMENT

The original equipment designed and built by Stanhope Seta for the sample conditioning was envisaged to be permanently sited on a platform or installation metering station. In order to render it portable considerable redesign of the pipework had to be undertaken. Consideration had to be given to the weight of the cabinet and the pipework and supports within.

In order to minimise heat loss between the line connections and the pyknometers all the pipework and hose connections are lagged. The internal pipework is valved so that the system can be flushed with fluid without contaminating the pyknometers. This is particularly useful on initial start-up of an exercise when the sample point is first opened.

The by-pass system also allows the pyknometers to be isolated from the main flow to facilitate draining of the internal pipework to allow easy disconnection of the Hanson couplings. In the case of high pressure NGL systems the drain system doubles up as a means of pressurising the pyknometers with inert gas to avoid chilling from flashing of the NGL.

Monitoring of the temperature across the system is carried out by means of a PRT upstream of the pyknometer and two temperature shoes fitted into the lagged, insulated pyknometer carry case which contact the skin of the pyknometers. The temperature across the system must balance to within 0.5 deg C.

Pressure gauges are connected to 3 points across the system to ensure that line pressure is achieved at the time of closing the outlet valve when sampling. Flow through the pyknometers is monitored by a variable area flow meter.

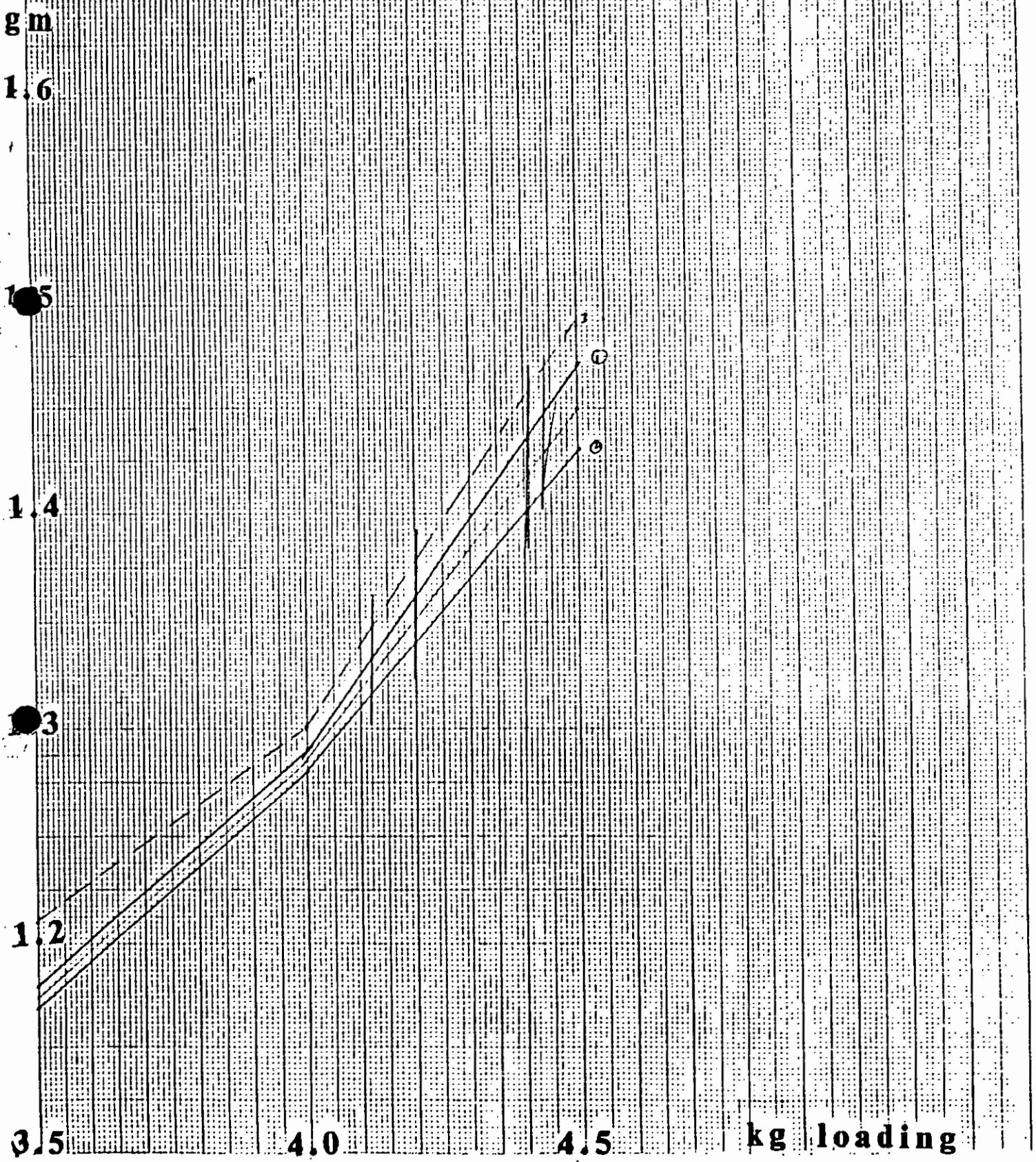
The balance used on site to weigh the pyknometer is a Sartorous MP 1507 Electronic balance fitted with the "Animal Weighing Program". This software is able to compensate for the vibration and motion of the platform and produces weights accurate to the desired 0.01g.

Since local conditions vary from place to place, the weights of the pyknometers, full and empty, are referenced to a series of masses which have been certified by a UK recognised National Calibration Laboratory. It has been found that these electronic balances exhibit nonlinearity in the range 3.5 kg - 5.0 kg. This is especially relevant on offshore platforms when vibration and motion are exaggerated.

To compensate for this lack of linearity a series of weights at 3.5 kg, 4.0 kg and 4.5 kg are plotted against the load difference. This permits the correction of the full and empty weights of pyknometers to be applied accurately. All calculations can be performed on a standard base.

The graph below shows the extent of this non-linearity.

BALANCE NON-LINEARITY



SAMPLING

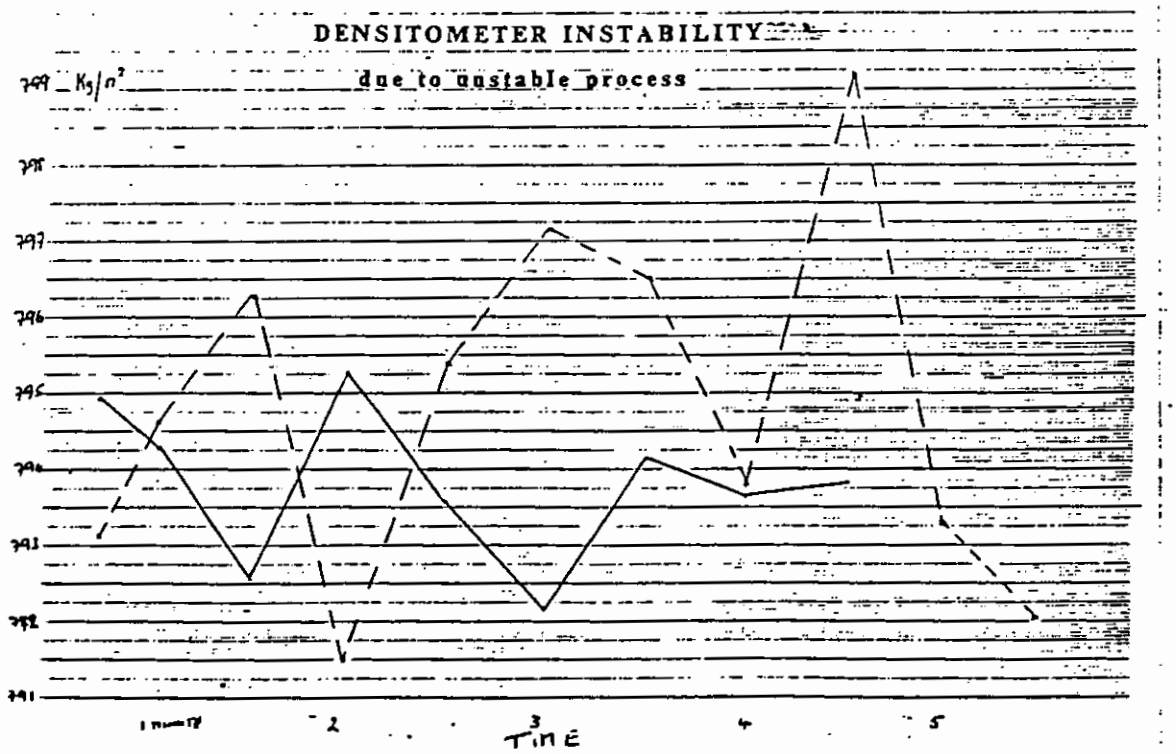
The technique of obtaining representative samples in the pyknometers, under line process conditions, can be an art form. The co-ordination of density meter readings and the catching of samples are the most important factors in validating the calibration of the density meters.

A complete understanding, by the personnel performing the validations, of the engineering and laboratory procedures used in obtaining density measurements is imperative if accurate and meaningful results are to be obtained. This level of understanding provides an overall degree of certainty that the complete density measurement system is being assessed and not just the performance of the densitometer tubes.

Pyknometry is therefore providing a means of validating the the total density measurement system, the temperature and pressure measuring devices, the flow computer/density convertor and the densitometer device. It also gives the assurance that the positioning of the ancilliary equipment in the system gives the most accurate and representative measurements of temperature and pressure in the system at the densitometer. It is current usual practice to use values obtained from the main line or the volume metering lines for the calculation of line densities.

Stability of process conditions is important to the successful validation of densitometers. The IP Code of Practice used as guidelines requires stability of process density to be better than 0.05% over 2 minutes. In installations where it is not possible to obtain this stability, plots of the density against time must be used to obtain the density at the exact time of sampling.

The graphic examples below illustrate the methods of relating line density to the pyknometer density under unstable conditions.



Where the conditions of line density are stable an average of the density over the two minutes prior to the sampling can be used as representative of the density of the fluid in the pyknometer.

Flow rate through the pyknometers is critical in obtaining duplicate samples representative of the fluid. This is particularly true of NGL systems and where there are crudes with high water contents, there is a tendancy for the heavier components to accumulate within the pyknometers at low velocities.

The IP code requires duplication between pyknometers better than 0.05% for the determination to be acceptable. In practice it is found that, with experience, duplication of better than 0.02% can be achieved for crude oil. For NGL and condensate systems this level of duplication is more difficult to achieve and the 0.05% is more realistic.

Experience has shown that NGL and condensate homogeneity systems offshore pose considerably greater problems due to the non homogeniety of the fluid, when glycol or other contaminents are present. The ability to achieve duplication is adversely affected by this and the achievement of duplication better than the 0.05% is therefore more difficult.

The accumulation of the heavier components in the pyknometers is a function of the flow rate obtainable. This has an effect on the representativity of the sample and hence on the validation of the density meters. By controlling the flow rate and the length of time to achieve temperature stability it is possible to minimise the effect on some systems and eliminate it on others.

Calibration of Pyknometers

The procedures, developed by Moore, Barrett and Redwood, for calibrating the base volume and mass of the pyknometer follow closely the original calibration procedures of NPL and those proposed in API Chapter 14.6.

The mass of the pyknometer body and of the valve components are determined independently using a specially modified bullion balance and a Sartorius research grade balance to 0.1 mgm. The pyknometer is reassembled and weighed, the total weight should agree to within 0.5 mgm of the total of body and fittings weights.

The initial volume at base conditions and the coefficients of expansion due to pressure and temperature are determined using water. Base volume at 25 deg C and 1 bar A is determined. Further volumes at elevated pressures of 10, 20, 60 and 120 bar are determined to establish pressure coefficients. Volumes at 5 deg C and 50 Deg C are determined at constant pressure to establish temperature expansion coefficients.

Thermometry is carried out using an ASL F26 bridge fitted with a Rosemount 100 ohm platinum resistance sensor and a Cropico 100 ohm standard. A Tinsley 5187 SA 25 ohm sensor complete with 30 ohm standard is currently undergoing calibration at the National Physical Laboratory, and when this is brought into use they should allow temperature measurement with uncertainties of the order of +/- 2mK and produce a subsequent improvement in all the volumetric uncertainties quoted below.

A Budenberg 380L dead weight tester with a stated accuracy of 0.04% along with a Fig 28 oil seal is used. The manufacturers state that the error introduced by the oil seal is no greater than 0.014 bar at any applied pressure

Weighing is carried out on a purpose-built bullion balance with a total capacity of 5kg and a nominal sensitivity of 2 mg per division. Weights up to and including 100 g are of E2 standard, above 100 g, are of F1 standard, but have been recalibrated using several sets of E2 weights to achieve uncertainties similar to E2 standard.

Valve plugs and fittings are weighed on a Sartorious research grade balance with has a resolution of 50 microgrammes at this load.

All weighings are carried out using Gauss's method, and air buoyancy corrections are always applied. Air densities are calculated from air temperature, pressure and humidity observed during the weighing.

All water used is drawn from a Barnstead water purifier, which gives a quality exceeding 18 Megohm centimetre resistivity or 0.055 microSiemen per centimetre conductivity.

Water densities at temperature and pressure are taken from Kell, Journal Chemical and Engineering Data Vol.20 No. 1 (1975).

We currently believe our uncertainties to be as follows:

Mass of pyknometer body	+/- 7mg.
Mass of complete pyknometer	+/- 7mg.
Base volume	+/- 0.02 ml
Change with pressure	+/- 0.0002 ml/bar
Change with temperature	+/- 0.001 ml/deg C

Using state of the art thermometry and pressure control equipment and a balance capable of weighing to mgms it is possible to reduce the uncertainty of calibration on each pyknometer to 0.004%.

Uncertainty

Practical experience, by Moore, Barrett and Redwood, shows that the uncertainty of performing the pyknometry exercise in the field is 0.013%. For duplicate pyknometers this uncertainty is 0.010% under field conditions. This takes account of the weighing uncertainty and that of the pressure and temperature measurement devices.

To ensure that any systematic uncertainty associated with the calibration of individual pyknometers is minimised we use random pairings of the pyknometers during an exercise.In this way any systematic error in a pyknometer is made obvious to the operator and can be investigated. The tight control of the repeatability between duplicate pyknometers on each run is a further controlling measure.

In arriving at our quoted uncertainty values we have only considered those variables which affect directly the conditioning of the pyknometer and the density calculated from it. This makes the statistical approach a direct comparison to the proving of volume meters by pipe provers. To compare directly with the densitometer we must consider the repeatability achieved under field conditions between the pyknometers and the densitometers on successive runs and the reproducibility on successive visits.

Calculating overall repeatability from practical experience as detailed in Table B. From the validation exercises using 6-8 individual determinations per validation gives us an overall figure of 0.024% for crude oil.

This can further be broken down to show overall repeatability associated with installations with Twin Tube densitometers and single tube densitometers.

On Twin Tube systems	0.012%
On Single Tube systems	0.033%

The above figures are based on the use of duplicate pyknometer results.

TABLE B

Repeatability Between Densitometer And Pyknometer Densities

PLATFORM	FISCAL		TRACKING	
	% DIFFERENCE	STD. DEV.	% DIFFERENCE	STD. DEV.
A	0.003	0.004	0.018	0.014
A	0.010	0.008	0.025	0.018
A	0.038	0.022	0.019	0.007
A	0.018	0.012	0.025	0.013
B	0.078	0.026	0.038	0.023
B	0.101	0.008	0.087	0.008
C	0.014	0.010	0.030	0.018
D	0.024	0.019	0.022	0.011
E	0.020	0.013	0.104	0.021
F	0.036	0.044	0.073	0.028
F	0.066	0.022	0.130	0.018
F	0.100	0.030	0.033	0.019
F	0.045	0.027	0.087	0.024
G	0.270	0.070	0.090	0.043
G	0.024	0.020	0.285	0.048

NGL,Condensate and Petroleum Products

This paper deals mainly with the use of pyknometry for validating crude oil systems. However we have had success in using the method to validate densitometers used in N.G.L., Condensate and Petroleum Products. This has however been restricted by the number of offshore and onshore systems where we have had practical experience.

Several problems have been encountered in the use of pyknometry on high pressure condensate analysis. The seals on the pyknometer valves is the most critical of these. To overcome this problem it has been necessary to use only new valve seats and to ensure that the valve stems are not scored in any way. This has led to a considerable improvement in the duplication achievable and the number of acceptable determinations.

The current pyknometers have a mass of about 4 kg and this ratio of mass of pyknometer to mass of fluid contained within the pyknometer means that the achievement of duplication between pyknometer is much more critical than for crude oil to obtain the same level of accuracy. Development of less dense material for pyknometer construction would result in improvements in this ratio.

To achieve acceptable levels of uncertainty for NGL and light fluids the number of determinations is often considerably higher than the 6-8 used for crudes. This may also be a factor of the densitometers and sampling systems used.

Conclusions

In the light of experience gained in the field over the last two years, Moore, Barrett and Redwood are quite convinced that pyknometry is a practical, first principles method for routine validation of fiscal systems and this method gives results compatable with the requirements of the Institute of Petroleum Part 7 section II as currently defined. The pyknometry method, with improvements in the current temperature and pressure measurement equipment, can be extended to calibrate densitometers either offline under laboratory conditions or on line in field situations.

While being recognised as a relatively expensive method of confirming densitometer calibrations consideration must be given to the independence of methodology and the high level of personnel required which gives an equally high level of confidence in the results. This must be weighed against the relative costs of the exercise.

Over and above the routine applications pyknometry can be considered as a tool with considerably wider applications in the field of oil industry measurement. Some of these are summarised here:

1. Development of confidence in density measurements performed by new online instruments and in new pipework configurations.

2. Investigations of imbalances in density measurement between Fiscal stations, providing an independant referee method.

3. Development of laboratory based calibration techniques for online and inline instruments. Under laboratory conditions the uncertainties can be reduced considerably.

4. Developments which are currently under consideration for the construction of the pyknometers from less dense materials which will improve the body weight to volume ratio. This will permit more accurate weighing and allow pyknometry to be used on light liquified products and eventually on gas systems.

References

[1] Paper presented at the North Sea Flow Measurement Workshop, a workshop arranged by NFOGM & TUV-NEL

Note that this reference was not part of the original paper, but has been added subsequently to make the paper searchable in Google Scholar.