Field Dewpoint Measurement – How Good Are We ?

Recently BJ PPS Leeds dried a client system with nitrogen. Dewpoint was measured using a Michell ADM dewpoint meter which was connected to the system at different locations via a one metre long rubber hose. The instrument was allowed to stabilise for approximately five minutes before each reading was taken. Average result was in the region of -27.5 °C. A check of our nitrogen supply and the client's nitrogen supply showed a dewpoint of -30 °C. Clearly the nitrogen supply readings indicated that it was highly likely that the measurement of dryness was not correct as nitrogen is usually much drier than -30 °C and in fact we had dried the client system to a lower level than we could demonstrate !

The above project details were recorded just before BJ PPS Leeds were due to carry out dewpoint measurement trials at Moisture Control and Measurement and highlighted the need to raise our awareness of what actually happens when we measure dryness and how accurate our readings actually are!

For the purpose of testing MCM calibration house facilities utilising dewpoint generators that are calibrated to National Physical Laboratory and International Standards were employed. Reference standards used were a 1.8 ppm by volume (- 70 °C) air supply and generated set dewpoints of - 60 °C, - 40 °C and - 20 °C.

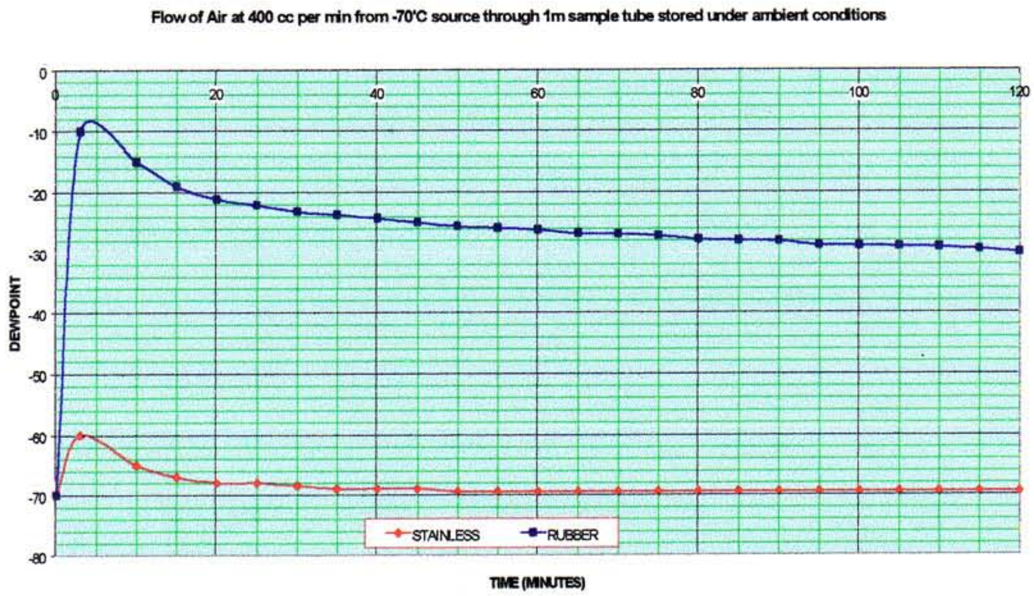
Please note that all measurements were made under laboratory conditions. We actually take measurements in the field where conditions are far from ideal and uncertainties are far greater.

Areas of investigation were limited to the following :-

- The effect on dewpoint measurement created by sample line composition.
- The performance of six different dewpoint meters through a dry to wet to dry cycle.

<u>The Effect on Dewpoint Measurement created by Sample Line Composition</u>.

One metre lengths one of rubber sampling hose and small bore stainless steel tubing that had been stored under ambient conditions were each (in turn) connected to the same -70 °C air supply at a flow of 400 cc per minute and the outlet dewpoints temperatures were logged. The results obtained are detailed in the graph below.



The rubber hose took two hours to achieve a dryness of -30 °C. In practical terms in the field it means that it is virtually impossible to read dewpoints less than –30 °C using rubber hose even after two hours of continuous sampling.

In addition it is a fact that ambient temperature and humidity can greatly influence the effective permeation rate of the rubber and made its performance in any situation a lottery. Rubber hoses are both hygroscopic and permeable.

If flow rates are increased through rubber hose then the contribution to overall dewpoint by the moisture produced from the rubber is less and dewpoint readings may fall – but they will increase again on reduction of flow (a confirmation that outgassing is taking place ie the rubber is adding water to the measurement). Also excessive flow will be more likely to contaminate the moisture sensor by incresing the rate of deposition of any contaminants to the sensor.

The stainless steel sample tube dried to –67 °C in 10 minutes and is to be recommended for field operations.

The above and other sample hose materials and their effectiveness are detailed in "A Guide to the Measurement of Humidity" produced by the National Physical Laboratory and the results we obtained were in line with the Guide's published results.

Conclusions

In practical terms for field applications the following recommendations apply

- ❑ Do not use rubber hose for sampling (or nylon as it is almost as bad).
- ❑ Stainless steel tubing (pig tail) or hose is ideal.
- ❑ PTFE lined stainless steel braided tube or PTFE tubing is also acceptable.
- ❑ Sample flow rates should be as low as practical.
- ❑ Sample lines should be as short as possible.

<u>The Performance of Dewpoint Meters through a Dry to Wet to Dry Cycle.</u>

Instruments tested

Michell MD10 (aluminium oxide sensor)
Michell ADM (aluminium oxide sensor)
Shaw type (aluminium oxide sensor)
MCM (push purge facility and heated silicon sensor)
Michell Cermax (aluminium oxide sensor)
Michell Transmet (aluminium oxide sensor)

Performance Assessment Method

Each instrument was connected to the same source and subjected to the same cycle of events detailed below. Instrument readings were recorded at five minute intervals after the initial dry down and the test cycle lasted for 19 hours for each instrument.

Dry down to best attainable in minimum 12 hours when connected to a – 72 °C source.
Exposure to –60 °C generated dewpoint for 60 minutes.
Exposure to –40 °C generated dewpoint for 60 minutes.
Exposure to –20 °C generated dewpoint for 30 minutes.
Exposure to ambient for 5 minutes.
Exposure to –70 °C generated dewpoint for 25 minutes.
Exposure to ambient for 45 minutes.
Exposure to –20 °C generated dewpoint for 45 minutes.
Exposure to –40 °C generated dewpoint for 60 minutes.
Exposure to –60 °C generated dewpoint for 60 minutes.
Exposure to –70 °C generated dewpoint to monitor final dry down for 30 minutes.

Durations of each hold were generally set to allow as long as needed at each step change for readings to stabilise

Results obtained were as detailed in the graph overleaf.

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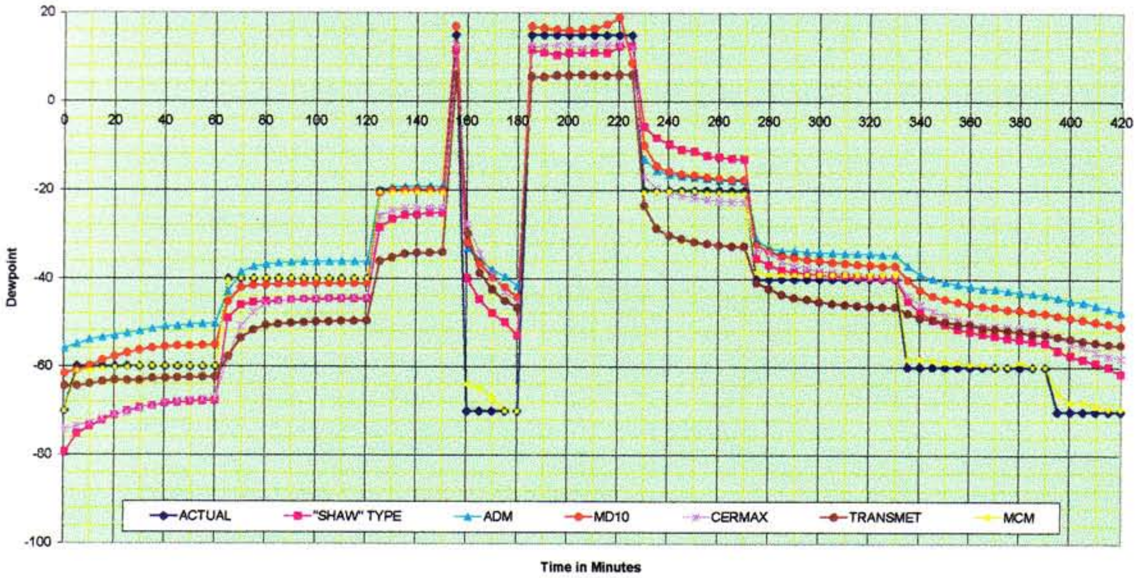


First impression from the graph shows there is wide spread of readings both from instrument to instrument. The range of spread is dependent on whether instrument sensors are going from dry to wet or from wet to dry (hysteresis). For instance, at a true - 40 °C, when going from dry to wet, readings vary from - 49 °C to -36.1 °C, a range of 13 °C. At a true -20 °C, when going from wet to dry, readings vary from -13 °C to –32.6 °C, a range of nearly 20 °C. (In terms of actual water present in PPM (V) in the latter instance it means that for an actual water content of 1020 ppm - the indicated level is anything from 1960 ppm down to 290 ppm – an astounding range by any standards – reading indicated is from double to one third the actual water content!!)

All instruments carried calibration certificates yet readings obtained appear to be a lottery.

The performance characteristics of any measuring instrument (in this case dewpoint) can be demonstrated by looking at

Speed of Response
Stability
Repeatability
Accuracy

Other important factors include drydown time, hysteresis, time to equilibrium, resistance to contaminants and diagnostic capability.

Under closer scrutiny, it is apparent that one instrument tested above is an order of magnitude superior to the rest. This is the one using silicon technology.

Aluminium oxide technology is still currently used by the majority of suppliers because they have become locked into that technology over a long period starting when dewpoint measurement was merely a measurement of relative humidity and the large drift characteristics resulting from lack of temperature stability of aluminium oxide was less relevant at these higher moisture levels.However as the need for more accurate readings down to lower levels of moisture developed, the usefulness of such non temperatur controlled devices has been pushed to the limit.

To understand the variations in perfomance figures we have obtained it is important to review aluminium oxide and silicon sensors' characteristics in more detail and look how silicon sensors have been adapted to optimise their performance under practical field conditions.

Aluminium Oxide Sensors

Properties change as material ages due to changes in crystalline form causing change in electric properties. This produces a change in sensitivity and the lack of response to moisture increases, reducing sensitivity.

Sensors left on dry gas tend to produce a steady a steady downward drift leading to a false idea of how dry a gas is (one supplier even includes a disclaimer in his literature that after 8 hours on line readings can become inaccurate).

Aluminium oxide sensors have been scientifically been shown to be accurate only on the day of calibration and a second calibration the next day is likely to produce a significant zero shift. In addition there is scientific proof that new calibrated aluminium oxide sensors when stored under dry nitrogen conditions for six weeks have shown differences in their zero calibration point ranging from 20% to a massive 70%. The National Bureau of Standards in Washington published a report concluding that for aluminium oxide humidity sensors "the drift in the calibration curve is too large for these sensors to be useful to study the behaviour of water inside experimental test packages over extended periods." We store sensors for months and expect them to perform accurately!

Aluminium oxide sensors produced by the likes of Michell, Shaw and Panametrics are calibrated at a set temperature. However they all fail to tell you that when you are measuring dew points in the region of –20°C (approx 1000 ppm by volume) that every 1°C in temperature variance from the calibration temperature can produces a change of 1°C in actual reading. This effect happens throughout the measuring range to some extent or another and is because the molecular structure of the sensor material is such that the effect of temperature and moisture on the structure inhibits or sometimes accentuates its sensing ability as the molecular structure changes with time and temperature.

Aluminium oxide sensors are inherently slow and their speed of reaction is linked to the a) the starting condition of the sensor and b) the amount of contamination present following direct deposition from produced flow (and in our case the added back migration of hydrocarbon molecules from our vacuum pumps) and the amount of condensation deposited on the probe from the product being measured.

The starting condition of the sensor depends on how wet or dry the sensor is and the magnitude of the difference between the actual sensor dew point and the product dew point to be measured. This difference determines the amount of time needed for the unit to stabilise and give consistent readings.

This is normally in the region of hours and days rather than minutes at the drier levels encountered. Contamination of the probe also effects the reading and stabilisation of the instrument with the more contamination the slower the response time. The temperature and humidity will also affect the stabilisation level to above or below the actual dew point measurement depending on the effect on the molecular structure of the aluminium oxide.

Aluminium oxide is inherently more hygroscopic than silicon so natural drying time is longer.

Heating of aluminium oxide sensors increases its ageing rate and can cause surface cracking.

Operation at ambient temperatures increases the tendency for vapour phase materials to adsorb on the surface and sensor is wetted immediately by any condensing atmosphere. Water on the surface can damage the surface which is also readily attacked by acidic gases CO_2 etc.

In operation it impossible to know if readings are being affected by contamination or oxidation though the ageing process. Brief exposures to ambient air conditions is a practical reality when connecting equipment a system, so is longer term storage at ambient. As investigated, very long drydown times are required if instruments are stored wet.

Silicon Sensors

This material is inherently more stable due to the lack of change with time, which avoids loss of sensitivity and major drift problems.

Natural tendency is to dry down faster than aluminium oxide. This is assisted further by the sensor's small physical size and low mass and its inbuilt temperature control which holds the sensor above normal ambient temperatures.

Inherent stability of silicon means it can be heated without detrimental effects which produces massive advantages over aluminium oxide sensors.

Sensor in unit tested was constantly heated to 46 °C at all times. The instrument was calibrated at this temperature.Readings are therefore taken with the sensor at a constant elevated temperature ensuring stability of readings and eliminating drift due to changes in sample gas temperatue or ambient conditions.

In addition there was no possibility of condensation forming on the sensor during operation and a reducting of the tendency for contaminants in the vapour phase to adsorb to the sensor surface.

Also due to the thermal stability of the sensor the unit tested had the facility to heat the sensor to 130 °C during operation to drive off and vapourise any volatile contaminants. This ability meant that readings could be taken relatively quickly with limited stabilisation required. The unit always read from dry to wet in the same way it was calibrated, and hysteresis effects were virtually eliminated. Also drydown from wet to dry was achieved within a few minutes.

In addition the ability to heat the sensor to 130 °C at any time means the unit can be used as a diagnostic tool to confirm validity of readings at any time throughout the measuring process.

Units are available fully certified as intrinsically safe. Even though sensors incorporate a heater, the low amount of heat generated and the small surface area means that they are within standards for Zone 1 operation.

Conclusion

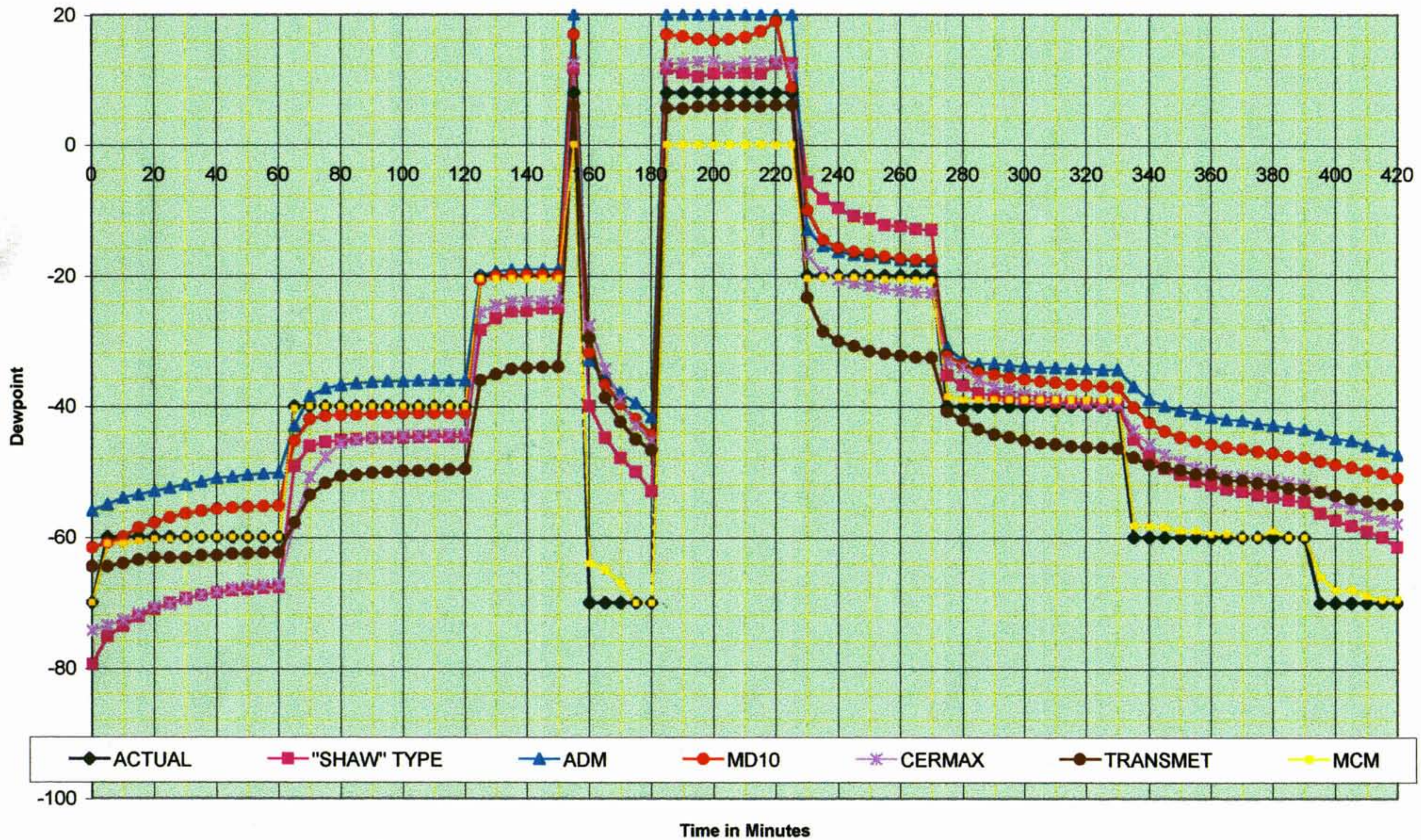
From the above explanations it is apparent why we are inconsistent with the measurement of dew point. We are locked into the same technology as our main suppliers where the product used is not exactly the best. Fortunately the majority of our clients are equally ignorant. Switching on a dew point meter, taking a reading and giving the results to the client with a calibration certificate keeps him happy.

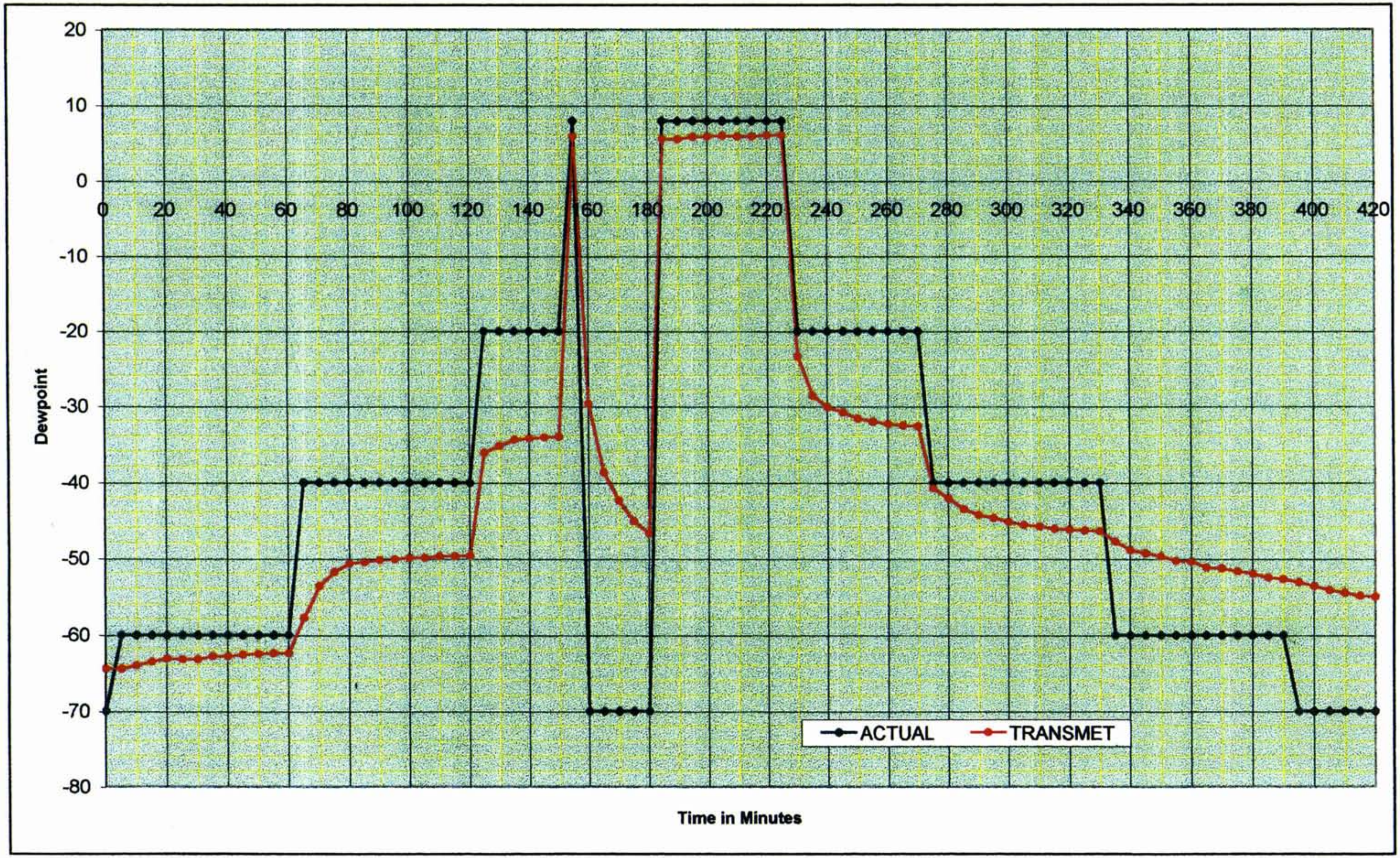
However , there is an alternative technology available that gives us the opportunity to break away from the less reliable systems. We should move forward to utilise that technology in order to :-

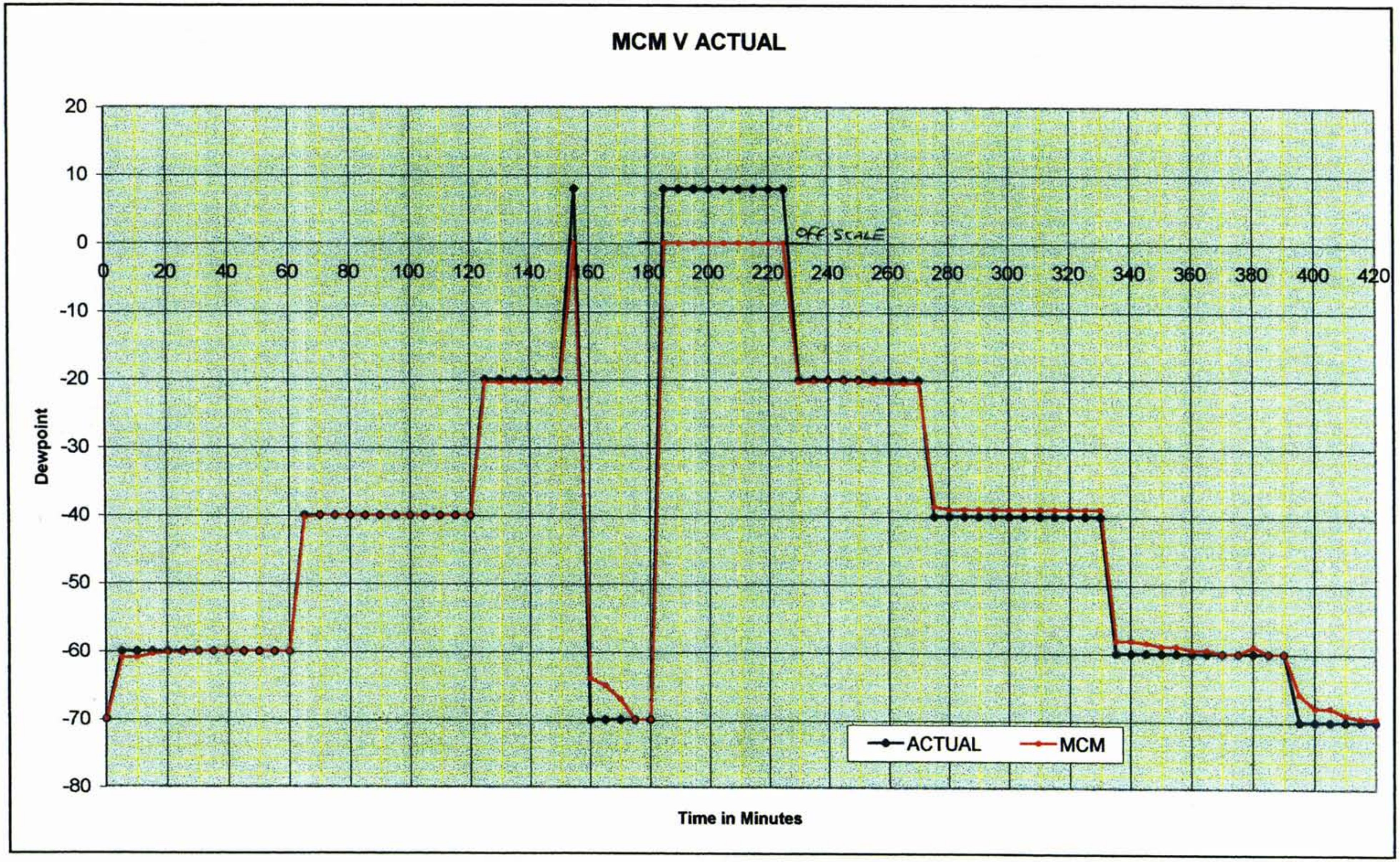
- **Improve the reliability and accuracy of dewpoint readings in the field.**
- **Have the choice of faster measurement times or improved accuracy as required by the job.**
- **Raise client awareness of our superior technology**
- **Provide a value added product**
- **Provide a competitive edge over our opposition by highlighting the weaknesses of less reliable technologies.**

Leeds base is currently about to envelop silicon technology and is negotiating for the supply of units for use in the field.

MCM DEWPOINT TRIALS 01




Dewpoint
20
10
0
-10
-20
-30
-40
-50
-60
-70
-80
0
20
40
60
80
100
120
140
160
180
200
220
240
260
280
300
320
340
360
380
400
420
ACTUAL
TRANSMET
Time in Minutes



MCM V ACTUAL
Dewpoint
20
10
0
-10
-20
-30
-40
-50
-60
-70
-80
0
20
40
60
80
100
120
140
160
180
200
220
240
260
280
300
320
340
360
380
400
420
OFF SCALE
ACTUAL
MCM
Time in Minutes

Flow of Air at 400 cc per min from -70'C source through 1m sample tube stored under ambient conditions