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Natural gas — Calibration of chilled mirror type instruments for hydrocarbon dewpoint (liquid formation)

*Gaz naturel — Étalonnage des instruments du type à miroir refroidi pour
points de rosée hydrocarbures (formation de liquide)*

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

In exceptional circumstances, when a technical committee has collected data of a different kind from that which is normally published as an International Standard ("state of the art", for example), it may decide by a simple majority vote of its participating members to publish a Technical Report. A Technical Report is entirely informative in nature and does not have to be reviewed until the data it provides are considered to be no longer valid or useful.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

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Introduction

Under certain conditions, higher hydrocarbons present in natural gas or similar gases can condense and the hydrocarbon liquids formed can cause difficulties in the operation of gas transport and distribution systems.

Hydrocarbon dew-point measurements, by condensation on a mirror, can give an indication of the conditions under which condensation starts. Theoretically, the hydrocarbon dew point is the temperature at which the first small droplets of liquid are formed at a fixed pressure. In practice, all dew-point-measurement methods are based on the observation of the formation of a film of hydrocarbon liquids on the surface of an illuminated, cooled mirror. The observation can be done visually (manual mirror) or by an electronic sensor (automatic chilled mirror). The cooling can be achieved in two ways: expansion of natural gas, compressed air or carbon dioxide or by applying a Peltier-cooling device. Either manual or automatic chilled mirrors can be applied to measure the hydrocarbon dew point of natural gas.

It is not possible to calibrate commercially available hydrocarbon dew point analysers in a traceable way, because no hydrocarbon dew-point reference material or reference instrument is available. Because of differences in working principles, analysers from different manufacturers can give different values for the hydrocarbon dew point for a given gas. In practice, the dew point of an automatic dew point monitor is often "tuned" to match the value measured by a manual chilled mirror, or "tuned" to the value calculated from a known gas composition using a thermodynamic model.

Modern automatic hydrocarbon-dew-point chilled-mirror instruments have the possibility of adjusting the value of the presented hydrocarbon dew point. Sometimes this adjustment is carried out by changing a physical setting in the instrument itself; in other cases the setting can be changed by entering a new value into the instrument's controller. The availability of such an adjustable parameter is a prerequisite for using the calibration procedure described in this Technical Report.

NOTE 1 Changing the setting of such a parameter can result in a major change in the presented hydrocarbon-dew-point value. For example, a study carried out under the auspices of the National Physical Laboratory^[2] shows from real measurements that the hydrocarbon dew point for a natural gas measured at the same pressure varies between $-6,28\text{ }^{\circ}\text{C}$ and $8,54\text{ }^{\circ}\text{C}$ [Gas B, with pressure equal to 3,5 MPa (35 bars), detector sensitivity varying between 110 mV and 275 mV].

In 2007, the results of a comprehensive study^[2] of the measurement of the hydrocarbon dew point of real and synthetic natural gases was published. In this study, six analytical methods were examined: one automatic chilled-mirror instrument, one manual chilled-mirror instrument, two laboratory gas chromatographs and two process gas chromatographs. In this study, it is concluded that the role of accurate synthetic gas mixtures in the calibration of chilled-mirror instruments is limited. Furthermore, it is stated that a standard composed of *n*-butane in nitrogen is indeed a straightforward and inexpensive way to calibrate a chilled-mirror instrument, yet forms an atypical, rapidly condensing hydrocarbon film. Therefore, such a calibration gas has limited use in calibrating a hydrocarbon-dew-point instrument.

NOTE 2 Several studies showed the importance of not only the component concentration in a calibration mixture but also knowledge of the nature of the components used. For example, a GERG study^[4] shows that adding aromatic and/or cyclic hydrocarbons has a significant influence on the hydrocarbon dew point.

Based on the working principle of chilled-mirror devices, there are five major sources that can be responsible for significant systematic errors in the measured hydrocarbon dew point and for which no adjustment can be made because no proper calibration method exists. These five sources are

- a) the often significant amount of liquid that it is necessary to form before the instrument is able to detect the dew point temperature;
- b) the cooling rate, which is often too fast to ensure that the temperature measured by a temperature sensor somewhere in the mirror equals the temperature of the mirror surface and the temperature of the gas in the measuring cell;

- c) the way the gas flow passes through the measurement cell during the actual measuring phase (continuous versus stop-flow principle);
- d) the measurement of the mirror temperature, which doesn't take place at the mirror surface itself but near the mirror surface;
- e) the hydrocarbon dew point that, when measured at a certain pressure setting, doesn't necessarily corresponds to the cricondentherm pressure valid for the actual gas composition.

In this Technical Report, a calibration procedure is presented which allows the adjustment and even the calibration of a hydrocarbon-dew-point chilled-mirror analyser against the indirect automatic weighing method according to ISO 6570. By using this procedure, the measured hydrocarbon dew point corresponds unambiguously to a given value for the potential hydrocarbon liquid content (PHLC) at this measured dew-point temperature. In this way, a traceable and much more objective measurement of the hydrocarbon dew point is possible. By doing an on-site comparison/calibration against the indirect automatic weighing method, it is even possible to correct for the gas-dependent performance of the hydrocarbon-dew-point analyser, which exists to some extent for different natural gases.

Measurements carried out according to ISO 6570 consist of cooling down a well defined gas flow to a specified and accurately measured temperature. The gas has enough time to form liquid and to establish a gas-liquid equilibrium at a certain pressure and temperature. This process is similar to the process that occurs in practice, when in a pipeline the pressure and/or temperature are reduced, the complete gas flow is cooled down and depending on the gas quality in the worst case can result in hydrocarbon liquid drop out. By calibrating a dew point analyser against ISO 6570, it is made sure that a dew point is obtained, which is related to the process which actually occurs in the pipeline upon pressure reduction, although the measurement technique is quite different to the process in the pipeline.

According to ISO 6570, the detection limit of the automatic weighing method is 5 mg/Nm^3 . Theoretically it can be argued that the temperature corresponding to a potential hydrocarbon liquid content of 5 mg/m^3 is not a correct estimate for the hydrocarbon-dew-point temperature. However, taking into account the measuring capabilities of the existing hydrocarbon-dew-point measurement devices, this effect can be neglected.

Based on the geometry of the measurement cell of a chilled mirror instrument, it is estimated by Cowper^[3] that it is necessary to condense approximately 70 mg/Nm^3 onto a mirror surface to register a hydrocarbon-dew-point temperature. Depending on the gas composition, this effect results in a bias of up to $-1,5 \text{ }^\circ\text{C}$ in the measured hydrocarbon dew point value.

NOTE 3 EASEE-gas (European Association for the Streamlining of Energy Exchange) recently agreed upon harmonized values of the hydrocarbon dew point throughout Europe (Common Business Practice 2005-001/01)^[1]. The required harmonized measuring method, which it is still necessary to identify, can clearly benefit from the proposed traceable calibration procedure presented in this Technical Report.

Natural gas — Calibration of chilled mirror type instruments for hydrocarbon dewpoint (liquid formation)

WARNING — The use of this Technical Report can involve hazardous materials, operations and equipment. This Technical Report does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this Technical Report to establish appropriate safety and health practices and to determine the applicability or regulatory limitations prior to use.

1 Scope

This Technical Report describes the principles of, and general requirements for, the traceable calibration of automatic hydrocarbon-dew-point chilled-mirror instruments using the indirect automatic weighing method (method B) described in ISO 6570:2001 to determine the potential hydrocarbon liquid content of natural gas, or similar gas. The calibration procedure is intended for use by chilled-mirror instruments in downstream applications transferring processed natural gas.

If the gas composition is constant, the manual weighing method (method A) described in ISO 6570:2001 is also applicable.

NOTE 1 Whether or not a gas composition is constant is difficult to establish. A process gas chromatograph (GC) measuring calorific values within 0,1 % is absolutely no guarantee for a constant hydrocarbon liquid drop-out content. Information up to C₁₂ is required to verify that the gas composition is constant.

NOTE 2 The application of this calibration procedure in the upstream area is not excluded *a priori*, however, currently there is no experience using this procedure in an upstream environment.

The usability of data on the potential hydrocarbon liquid content of natural gas for verification, adjustment or calibration of hydrocarbon-dew-point chilled-mirror instruments is based on the condensation behaviour of natural gases. Information on the condensation behaviour of natural gases and the various measuring techniques to determine properties, like hydrocarbon dew point and potential hydrocarbon liquid content, related to the condensation behaviour of natural gases are given in Annex A.

NOTE 3 Unless otherwise specified, gas volumes are in cubic metres at 273,15 K and 101,325 kPa.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 6570:2001, *Natural gas — Determination of potential hydrocarbon liquid content — Gravimetric methods*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1

potential hydrocarbon liquid content

PHLC

property of natural gas defined as the amount of the condensable liquid (in milligrams) at the pressure, p , and temperature, T , of the measurement per unit volume of gas at normal conditions, that is, at a temperature of 0 °C and a pressure of 101,325 kPa, by passing a representative sample of the gas through an apparatus where it is first brought to the pressure, p , and then cooled to the temperature, T

NOTE It is necessary to take care that only gas, not a two-phase mixture, has been withdrawn from the pipeline.

3.2

hydrocarbon dew point

HCDP

property of natural gas defined as the temperature, T_{HCDP} , at which an automatic chilled-mirror instrument detects the first presence of hydrocarbon liquid drop out at the pressure, p

NOTE In chemical thermodynamics, the “true” hydrocarbon dew point is the temperature (at a stated pressure) at which the enthalpy of the gas and liquid phases is identical. Since measurement of the dew point involves reduction of the system temperature, this equates to the temperature at which the first appearance of the liquid phase occurs. At this point, the quantity of liquid phase is infinitesimally small. Since no instrument is able to detect this infinitesimally small amount, the aforementioned definition of the hydrocarbon dew point is adopted in this Technical Report. However, depending on the gas composition and the sensitivity of the detection system of the hydrocarbon-dew-point chilled-mirror device, the measured hydrocarbon dew point can be considerably lower than the “true” hydrocarbon dew point.

3.3

PHLC reference value

parameter in the calibration procedure chosen by the user

NOTE At the PHLC reference value, the hydrocarbon dew point temperature and the temperature of the ISO 6570 measurement are equal.

4 Symbols

p pressure at which PHLC and hydrocarbon dew point measurement are carried out

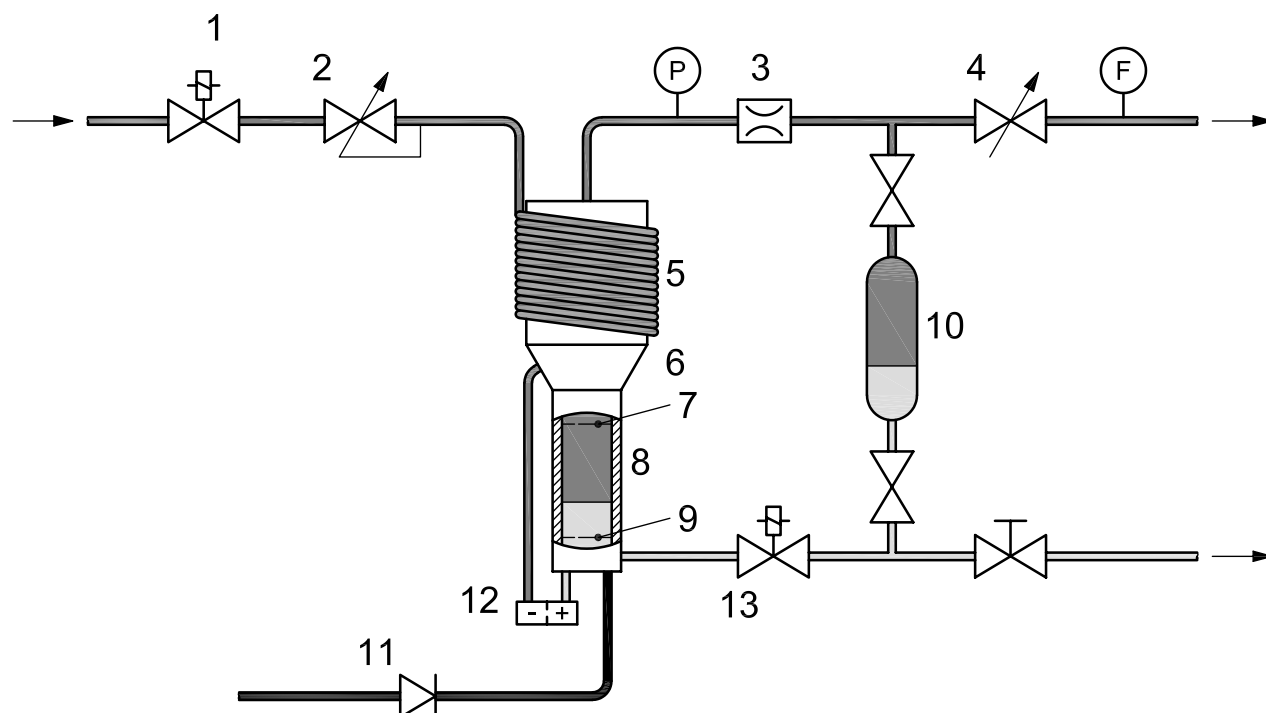
T temperature at which the PHLC measurement is carried out




5 Performance characteristics of automatic weighing method in accordance with ISO 6570:2001

WARNING — The instrumentation used for this method shall comply with local legal and safety regulations, especially with the local regulations for application of the instrumentation in hazardous areas.

5.1 Working principle

The principle of the indirect automatic weighing method (method B) is described in ISO 6570:2001. An example of an implementation of this method is shown in Figure 1. ISO 6570 states that the quantity of hydrocarbon liquids that can be formed at a certain pressure and temperature is determined by passing a representative sample of the gas through the apparatus where it is first brought to the required pressure and then cooled to the required temperature. The liquids formed during cooling are separated from the gas flow and collected by means of a cyclone separator into a measuring tube. The measuring tube is automatically drained when it is totally filled up with liquid. The liquid from the measuring tube is collected in a hydrocarbon-liquids drum. In the indirect automatic weighing method, the determination of the amount of hydrocarbon liquids formed is derived from the differential pressure measured along the measuring tube.



-  Natural gas
-  Condensate
-  Calibration liquid

Key

- 1 relieve valve
- 2 pressure controller
- 3 restriction
- 4 flow controller
- 5 heat exchanger
- 6 gas/liquid separator
- 7 maximum level
- 8 measuring tube
- 9 minimum level
- 10 condensate drum
- 11 injection point for calibration liquid
- 12 differential pressure sensor
- 13 drain valve

**Figure 1 — Implementation of indirect automatic weighing method according to ISO 6570
(GACOM[®] of N.V. Nederlandse Gasunie)¹⁾**

1) GACOM is an example of a suitable product available commercially. This information is given for the convenience of users of ISO 12148 and does not constitute an endorsement by ISO of this product.

5.2 Functional requirements

5.2.1 General

To ensure the correct functioning of the indirect automatic weighing method, it is necessary to fulfil the requirements given in 5.2.2 to 5.2.8.

5.2.2 Gas sampling

The sampling point at the pipeline should be situated as described in ISO 10715 [6]. The sampling system should be such that a continuous gas withdrawal at a pressure and temperature considerably above the cricondentherm is possible. No liquids or aerosols should be withdrawn together with the gas sample. The sample line should be heat traced up to at least 35 °C. The sampling system should be clean to prevent or at least to minimize the adsorption of higher hydrocarbons.

NOTE (Excessive) adsorption of higher hydrocarbons in the sampling system results in a systematic error in the measured hydrocarbon dew point value.

5.2.3 Calibration of the differential pressure sensor

The indirect automatic weighing method requires calibration of the differential pressure sensor. The differential pressure transmitter is calibrated by introducing known amounts of a calibration liquid, such as *n*-decane, into the measuring tube at the temperature and pressure at which the PHLC measurement will be performed. Such calibration shall be carried out at least at the start of a new measurement or calibration series and after (large) adjustments of the measuring pressure, *p*, or measuring temperature, *T*.

NOTE Frequent calibration of the differential pressure sensor is required to exclude the possible interference of the static pressure and/or temperature on the raw output of the sensor. The measured differential pressures are small and, therefore, any small interferences can considerably disturb the measurement results.

5.2.4 Pressure controller

In ISO 6570, the pressure controller is allowed to introduce a maximum variation of 10 kPa in the gas/liquid cyclone separator. For this particular application, it is advised to use a pressure regulator that introduces variations in the pressure of less than 10 kPa. Either the pressure controller should be electrically heated or the sample line and controller should be heat traced up to at least 40 °C.

NOTE Commercially available, high-quality pressure controllers introduce variations in the pressure of less than 10 kPa only for gas flows of approximately 1 Nm³/hr.

5.2.5 Temperature control

In accordance with ISO 6570, the cooling bath shall be capable of maintaining the temperature of the cooling liquid at any point of the bath with a variation of less than 0,25 K.

NOTE For temperatures below –15 °C, the specification in this subclause is difficult to maintain without taking special precautions. Especially with high environmental temperatures, such as occur during summertime, regular thermostats are not able to meet the specified criterion.

5.2.6 Measurement of pressure and temperature

The pressure and temperature sensors shall be calibrated at least once a year.

5.2.7 Flow measurement

The device for measuring the gas flow through the instrument, which is normally operated with a gas flow of 1 Nm³/hr, shall be checked at regular intervals against a calibrated gas meter. Such verification shall be carried out at least at the start of a new measurement or calibration series.

The automatic measuring device is operating with a gas flow of $1\text{ Nm}^3/\text{hr}$ at a fixed pressure, most often between 2,7 MPa and 3,0 MPa [(27 and 30 bar) (at the cricondentherm)], and a temperature (e.g. $-3\text{ }^\circ\text{C}$) at which the hydrocarbon liquid content (often 5 mg/Nm^3) is specified in some contracts.

5.2.8 PHLC measurement period

Due to the nature of the PHLC measurement, measurement values become available at a certain time interval. Since a short cycle period results in PHLC measurements with a high uncertainty, it is advised to use a cycle time of at least 30 min.

NOTE For a proper calibration of a hydrocarbon-dew-point chilled-mirror instrument, it is advisable to compare data for a period of at least 24 hours or more.

5.3 Measurement uncertainty

The uncertainty in the PHLC value depends on the pressure and temperature set points and the condensation behaviour of the gas being measured. PHLC-values above 5 mg/m^3 shall be detected unambiguously. At low PHLC values (less than 30 mg/Nm^3), the uncertainty shall be equal or less than 5 mg/Nm^3 .

As an example, the results of two individual, indirect automatic weighing instruments are presented in Figure 2. Both devices measure the potential hydrocarbon liquid content of the same gas and are operating at the same pressure and temperature. These results give an indication of the achievable reproducibility, which is within $\pm 5\text{ mg/Nm}^3$. During other experiments with a stable, type H-gas (high calorific value), an even smaller random error (2σ) of $\pm 2\text{ mg/Nm}^3$ was found.

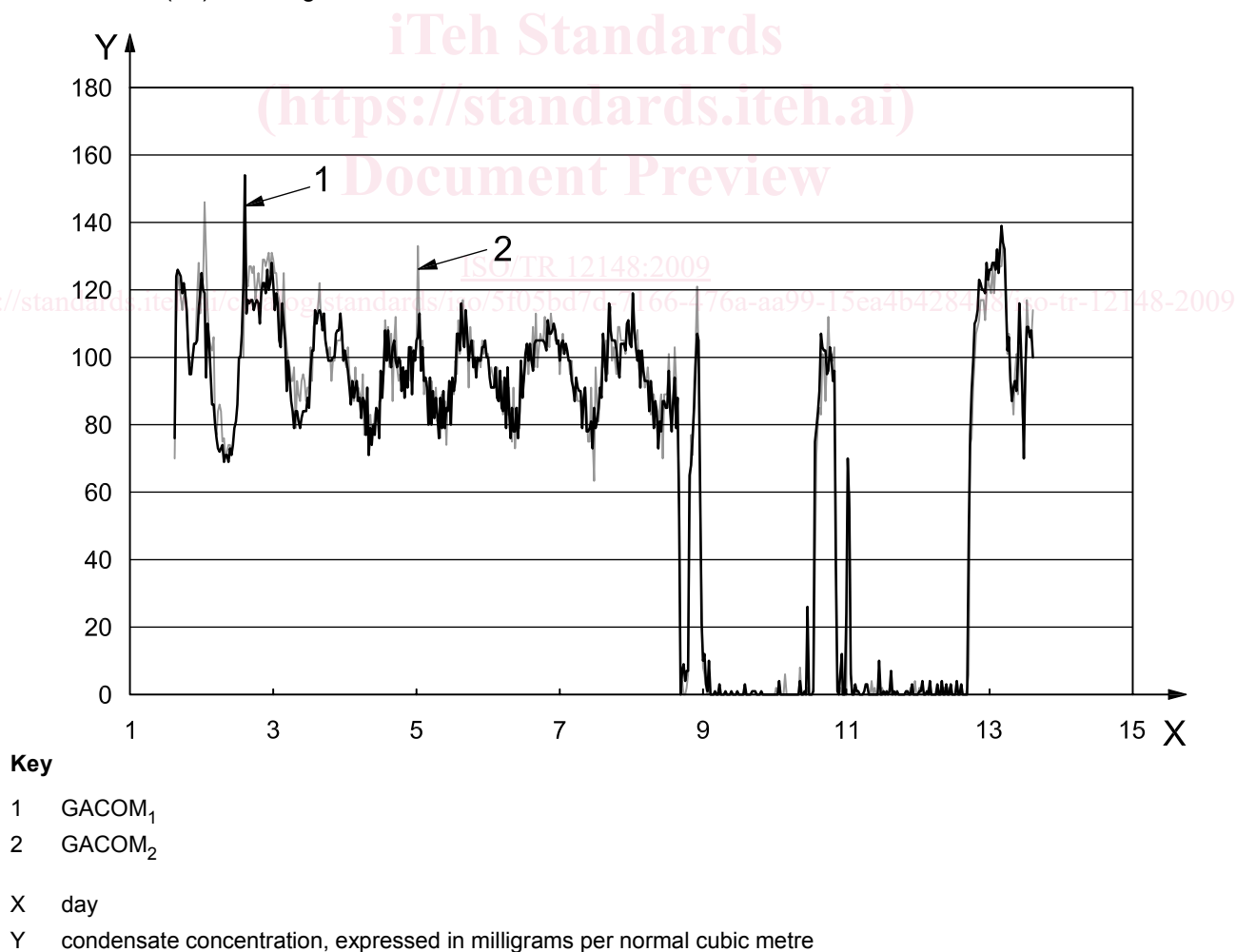


Figure 2 — PHLC measurements using 2 GACOM[®] units for an L-type gas at 2 700 kPa and $-3\text{ }^\circ\text{C}$