
**Natural gas — Gas chromatographic
requirements for hydrocarbon dewpoint
calculation**

*Gaz naturel — Exigences relatives à la chromatographie en phase
gazeuse pour le calcul du point de rosée hydrocarbures*

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Contents

Page

Foreword.....	iv
1 Scope	1
2 Normative references	1
3 Principle	2
4 Materials	2
5 Apparatus	2
6 Performance requirements	3
7 Sampling	3
8 Analytical procedure	4
9 Methods of test	7
10 Uncertainty in composition	9
11 Calculation of dewpoint temperature	9
12 Analytical uncertainty contribution to dewpoint temperature	9
Annex A (informative) Typical analytical conditions for C ₅ to C ₁₂ analysis	10
Annex B (informative) Validation of fraction data	12
Annex C (informative) Precision of area ratio	16
Annex D (informative) Recommendations on sample calibration gas introduction	20
Annex E (informative) Calculation of fraction quantities, boiling points and component uncertainties.....	22
Bibliography	26

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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ISO 23874 was prepared by Technical Committee ISO/TC 193, *Natural gas*, Subcommittee SC 1, *Analysis of natural gas*.

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Natural gas — Gas chromatographic requirements for hydrocarbon dewpoint calculation

1 Scope

This International Standard describes the performance requirements for analysis of treated natural gas of transmission or pipeline quality in sufficient detail so that the hydrocarbon dewpoint temperature can be calculated using an appropriate equation of state. It can be applied to gases that have maximum dewpoint temperatures (cricondentherms) between 0 °C and – 50 °C. The pressures at which these maximum dewpoint temperatures are calculated are in the range 2 MPa (20 bar) to 5 MPa (50 bar). Major components are measured using ISO 6974 (all parts) and the ranges of components that can be measured are as defined in ISO 6974-1. The procedure given in this International Standard covers the measurement of hydrocarbons in the range C₅ to C₁₂. *n*-Pentane, which is quantitatively measured using ISO 6974 (all parts), is used as a bridge component and all C₆ and higher hydrocarbons are measured relative to *n*-pentane.

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 6974-1, *Natural gas — Determination of composition with defined uncertainty by gas chromatography — Part 1: Guidelines for tailored analysis*

ISO 6974-2, *Natural gas — Determination of composition with defined uncertainty by gas chromatography — Part 2: Measuring-system characteristics and statistics for processing of data*

ISO 6974-3, *Natural gas — Determination of composition with defined uncertainty by gas chromatography — Part 3: Determination of hydrogen, helium, oxygen, nitrogen, carbon dioxide and hydrocarbons up to C₈ using two packed columns*

ISO 6974-4, *Natural gas — Determination of composition with defined uncertainty by gas chromatography — Part 4: Determination of nitrogen, carbon dioxide and C₁ to C₅ and C₆₊ hydrocarbons for a laboratory and on-line measuring system using two columns*

ISO 6974-5, *Natural gas — Determination of composition with defined uncertainty by gas chromatography — Part 5: Determination of nitrogen, carbon dioxide and C₁ to C₅ and C₆₊ hydrocarbons for a laboratory and on-line process application using three columns*

ISO 6974-6, *Natural gas — Determination of composition with defined uncertainty by gas chromatography — Part 6: Determination of hydrogen, helium, oxygen, nitrogen, carbon dioxide and C₁ to C₈ hydrocarbons using three capillary columns*

ISO 6975, *Natural gas — Extended analysis — Gas-chromatographic method*

ISO 10715, *Natural gas — Sampling guidelines*

3 Principle

For hydrocarbon gas mixtures such as natural gas, the phase boundary or dewpoint line distinguishing single-phase gas from gas-liquid mixtures is a complex function of pressure, temperature and composition. For a given composition, the pressure at which the dewpoint temperature is at a maximum is intermediate between those found in transmission and distribution operations. The analysis shall be comprehensive for inert components and for hydrocarbons up to C_{12} . It is not necessary to measure helium, hydrogen, water and sulfur compounds unless any of these is present at greater than 0,01 mole fraction.

The analysis shall be performed in two parts. Major components (nitrogen, carbon dioxide and hydrocarbons from C_1 to C_5) shall be analysed according to ISO 6974 (all parts). Higher hydrocarbons (C_5 to C_{12}) shall be analysed to satisfy the requirements given in this International Standard. This allows the traceability of measurements according to ISO 6974 (all parts) to be extended to the higher hydrocarbons.

It is not possible to identify all the measured higher hydrocarbons, nor is it possible to obtain a reference gas mixture that contains more than a few representatives of the higher hydrocarbons. The analytical data are, therefore, handled with a number of simplifying assumptions.

- Unidentified components are allocated a carbon number or molar mass according to their positions in the chromatogram with respect to identified *n*-alkanes.
- Alkanes of carbon number 7 and above are summed by carbon number and treated as fractions for input to the dewpoint calculation.
- Average boiling points and densities of fractions are calculated from the individual boiling points and quantities of the components that comprise them; individual component boiling points are calculated by interpolation between the bracketing *n*-alkanes [1].
- Sample components are quantified by comparison with *n*-pentane, which has been measured according to ISO 6974 (all parts), using relative response factors based on their allocated carbon numbers.

NOTE When using ISO 6974 (all parts), *n*-pentane can be measured as a direct component that is also present in the calibration-gas mixture, or as an indirect component using a response factor relative to a different component (for example, *n*-butane) in the calibration gas mixture. In either case, the uncertainty on the quantity of *n*-pentane can be calculated according to ISO 6974-2.

4 Materials

4.1 Certified-reference gas mixture for major components (CRM1), such as is required for ISO 6974 (all parts).

Depending upon the working range and the accuracy required, more than one CRM can be needed.

4.2 Certified-reference gas mixture, for higher hydrocarbons (CRM2), containing as a minimum, *n*-pentane, *n*-hexane, benzene, cyclohexane, *n*-heptane, toluene, methylcyclohexane and *n*-octane. Ideally, the CRM2 should also contain *n*-nonane, *n*-decane, *n*-undecane and *n*-dodecane in methane.

The mole fractions of components in CRM2 shall be chosen to be appropriate for the application, provided that the mixture is stable and free from the possibility of condensation in transport, storage and in use.

5 Apparatus

5.1 Measurement system for major components, comprised of a sample introduction and transfer unit, a separation unit, a detection unit, an integrator and a data reduction system, capable of meeting the analytical requirements described in 6.1.

ISO 6974 (all parts) describes equipment suitable for this part of the analysis.

5.2 Measurement system for higher hydrocarbons, comprised of a sample-introduction and transfer unit, a separation unit, a detection unit, an integrator and a data-reduction system, capable of meeting the analytical requirements described in 6.2.

Annex A describes a system that has been shown to be suitable. The user is responsible for demonstrating in each case that his/her chosen system is also suitable.

6 Performance requirements

6.1 Major components

The system for measurement of major components shall have performance as described in ISO 6974 (all parts).

6.2 Higher hydrocarbons

The system for measurement of higher hydrocarbons shall satisfy the following requirements:

- be capable of measuring alkanes up to and including *n*-dodecane;
- be capable of measuring individual alkanes at a concentration of 0,000 000 1 mole fraction (0,1 ppm¹⁾ or less;
- be able to distinguish and measure benzene, toluene, cyclohexane and methylcyclohexane as individual components;
- use a detection system that can, at least in principle, measure all hydrocarbons in the range C₅ to C₁₂;
- use a detection system that has a predictable response to hydrocarbons based on mass or carbon content, such that unidentified components of known molar mass or carbon number can be measured relative to other known components in the sample or in the calibration gas;

NOTE 1 It is most likely that the detection system is a flame ionization detector (FID).

- use a separation procedure such that the boiling points of unidentified components can be calculated by interpolation between those of known *n*-alkanes.

NOTE 2 Increasing the column temperature at a constant rate throughout the analysis (linear temperature programming) allows such interpolation.

NOTE 3 Annex A describes a configuration that has been found to be suitable for the requirements of 6.2.

7 Sampling

Carry out representative sampling in such a way that the sample represents the gas, particularly the higher hydrocarbons, at the time of sampling. Sampling and sample transfer shall be performed in accordance with ISO 10715.

1) ppm is a deprecated unit.

8 Analytical procedure

8.1 Major components

Follow the procedure specified in ISO 6974 (all parts).

8.2 Higher hydrocarbons

The analytical procedure consists of the following steps. All of these steps shall be carried out when the method is first introduced, and some of them when changes in equipment cause historical measurement data to be no longer valid. For normal use, steps may be left out provided that the procedures and equipment remain under statistical control.

8.2.1 Step 1 — Precision

Since quantitative information is derived by comparing the response of unknown components with that of *n*-pentane, which has been measured according to ISO 6974 (all parts), the precision information of interest is that of the ratio of component responses to that of *n*-pentane.

- Perform a number of repeat analyses on a typical natural gas and, for each analysis, calculate the ratio of the area of each peak to the area of *n*-pentane. For each peak, calculate the mean and the standard deviation of the ratios from all analyses.
- Where component data are to be summed into fractions or groups (see 9.3), calculate the ratio of the total peak area for that fraction or group to the area of *n*-pentane. For each fraction, calculate the mean and the standard deviation of the ratios.
- Convert both sets of mean and standard deviation data into natural logarithms, plot the natural logarithm of the standard deviation, $\ln s$, as a function of natural logarithm of the area ratio, $\ln R_A$, and perform a first-order regression analysis on the data. Annex C shows a worked example.
- Calculate the 95 % confidence limits for the regression line and plot these on the same graph. Select values of a and b in Equation (1) by trial and error such that

$$\ln s = a + b \times \ln R_A \quad (1)$$

results in a straight line that closely approximates to the upper 95 % confidence line (see Annex C). This line now defines the precision of measurement, as given in Equation (2):

$$s = \exp(a + b \times \ln R_A) \quad (2)$$

- This standard deviation is used as the standard uncertainty for each peak or fraction.

8.2.2 Step 2 — Relative response factors

When using a flame ionization detector, relative response factors, F_{RR} , are claimed to be proportional to carbon number. Under most circumstances this is true, but variations from ideal operating conditions can cause this assumption to be false, and so it is necessary that it be checked. This is all the more necessary for other types of detector. The procedure below uses the known composition of CRM2 to check the relative response factors. The most likely outcome is that the factors are directly related to carbon number, but the uncertainty with which this is validated shall be taken into account in the overall uncertainty calculation. Since *n*-pentane is the reference component, the response factor is conveniently expressed as a relative carbon response factor, $F_{RR,C}$, to that of *n*-pentane.

- Perform a number (not less than 5) of repeat analyses using CRM2. Measure the mean peak area response for each component and the equivalent standard deviations.

- Calculate the relative carbon response factor for each component, i , as given in Equation (3):

$$F_{RR,Ci} = \frac{5 \times \bar{A}_i \times c_{k,nC5}}{N \times \bar{A}_{nC5} \times c_{k,i}} \quad (3)$$

where

- \bar{A}_i is the average peak area of component i ;
- \bar{A}_{nC5} is the average peak area of n -C5;
- $c_{k,i}$ is the known concentration of component i ;
- $c_{k,nC5}$ is the known concentration of n -C5;
- N is the carbon number of component i ;
- 5 is the carbon number of n -C5 (n -pentane).

Equation (3) can also be expressed using area ratios, as given in Equation (4):

$$F_{RR,Ci} = \frac{5 \times \bar{R}_{A,i:nC5} \times c_{k,nC5}}{N \times c_{k,i}} \quad (4)$$

where $\bar{R}_{A,i:nC5}$ is the average peak area ratio of component i to n -C5. $F_{RR,C}$ values should be very close to 1.

- Calculate the average relative carbon response factor for each component, and the standard deviation of this value. The uncertainty of the $F_{RR,C}$ uses these standard deviation values and the uncertainties of the composition data for CRM2. The overall uncertainty of the $F_{RR,C}$ is calculated from the averages of the individual component values.

8.2.3 Step 3 — Validation of fraction data

Inert gases and individual hydrocarbons up to hexane can be measured unambiguously and used in the calculation software. Beyond this, n -alkanes up to C_{12} , benzene, toluene, cyclohexane and methylcyclohexane can be identified, but other hydrocarbons, in general, cannot be clearly identified and measured because of the large amount of overlap that occurs between isomers and the lack of reliable retention data. Even if individual isomers can be identified, it is unlikely that their critical properties, which are necessary for the calculation, are tabulated in the software.

Components that are measured but unidentified are summed into fractions or carbon number groups. Thus all components eluting after n -C₆ up to and including n -C₇, with the exceptions of benzene and cyclohexane, are summed as the C₇ fraction (FR7). The average boiling point and specific gravity of this summed group are calculated (see 9.3.2) and used by the software to calculate appropriate critical properties for the fraction. Two checks are made at this stage to ensure that the fraction data are as close an approximation as possible and to estimate the uncertainties involved. These are

- to compare attributed and “true” boiling points,
- to show that linear interpolation is valid.

8.2.3.1 Boiling-point comparison

The *n*-alkanes are easily distinguished in the separation and their properties are well known. They are, therefore, used as the basis for checking boiling point data. From an existing and comprehensive analysis, calculate the C₇ to C₁₂ fraction quantities, where measurable, according to 9.3.1. Identify these fractions as the equivalent *n*-alkanes and calculate the dewpoint on this basis. Now rename the C₇ group as the fraction, FR7, rather than *n*-C₇, and give it a boiling point and specific gravity that are those of the *n*-alkane. Recalculate the dewpoint.

This fraction dewpoint is likely to be slightly different from the *n*-alkane dewpoint. Adjust the boiling point value until the dewpoint agrees with that from the *n*-alkane. This adjusted boiling point, *T*_{BP,a}, provides the best fit when comparing the fraction property with that of the *n*-alkane. Repeat this substitution for each *n*-alkane individually, and note the adjusted boiling point in each case. Include the C₆ fraction and *n*-C₆. Even though the C₆ components are likely to be measured individually, the adjusted boiling point of *n*-C₆ is required for the calculation of the C₇ fraction properties in 9.3.2.

Having defined these adjusted boiling points (which can, in some cases, coincide with the “true” values), now define all carbon-number groups as fractions with these adjusted boiling points in each case. Recalculate the dewpoint. The difference between this last dewpoint value and that found when all groups are treated as *n*-alkanes is taken as the uncertainty value.

NOTE Annex B shows an example of such calculations.

8.2.3.2 Interpolation

Fraction boiling points are calculated on the assumption that the boiling points of individual unidentified components can be calculated by linear interpolation between the values for the bracketing *n*-alkanes. This is checked by using known data for *n*-alkanes. The boiling point of *n*-C₈ is calculated by interpolation between the known values (adjusted boiling points) for *n*-C₇ and *n*-C₉ and compared with the adjusted boiling point for *n*-C₈ found in 8.2.3.1. The calculated boiling point of the *n*-alkane is calculated as given in Equation (5):

$$T_{BP,cal,n-C_x} = T_{BP,a,n-C_{x-1}} + \frac{(t_{R,n-C_x} - t_{R,n-C_{x-1}}) \times (T_{BP,a,n-C_{x+1}} + T_{BP,a,n-C_{x-1}})}{(t_{R,n-C_{x+1}} - t_{R,n-C_{x-1}})} \quad (5)$$

where

*t*_{R,*i*} is the retention time of component *i* in the sample;

*T*_{BP,a,*n*-C_{*i*}} is the adjusted boiling point of *n*-alkane *i* in the sample.

The same is applied to other *n*-alkanes where interpolation is possible. For each *n*-alkane, the difference between the calculated boiling point and the adjusted boiling point from 8.2.3.1 is recorded. The boiling point uncertainty arising from interpolation is calculated as the mean of the absolute values of the differences between the calculated and adjusted boiling points for *n*-C₈, *n*-C₉, *n*-C₁₀ and *n*-C₁₁.

The sensitivity of the calculated dewpoint to these boiling point uncertainties is now determined. The analytical data used in 8.2.3.1 are used again but with fraction boiling-point data based on the adjusted boiling points for *n*-alkanes calculated as in 9.3.2. The dewpoint temperature is calculated from this analysis. Each fraction boiling point is then incremented by the boiling-point uncertainty calculated above and the dewpoint recalculated. The difference between the two dewpoint values is the uncertainty associated with interpolation.

NOTE Annex B shows an example of such calculations.

8.2.4 Step 4 — Sample introduction

Introduce the sample in accordance with 9.1.

8.2.5 Step 5 — Calculation of the composition

Calculate the composition of individual components and of fractions in accordance with 9.2.

8.2.6 Step 6 — Calculation of the composition uncertainty

Calculate the uncertainty of the composition in accordance with Clause 10.

9 Methods of test

9.1 Introduction of a typical sample of natural gas

To minimize surfaces on which higher hydrocarbons can be adsorbed, the connection to the sample cylinder can consist of a metering valve or needle valve fitted with low-dead-volume tubing. If this is not available, use a low-internal-volume pressure regulator with a stainless steel diaphragm and with no plastics material in contact with the sample gas.

If the temperature of the gas in the pipeline from which the sample was taken is known, ensure that the cylinder and the connecting tubing up to the pressure-reduction device or needle valve is heated to at least 10 °C above the gas temperature. The cylinder should be heated (if necessary) for not less than 4 h before use. The connecting tubing need only be heated at the time of use. Downstream of the pressure-reduction device or needle valve the gas is close to atmospheric pressure and heating is not necessary.

Purge the sample valve and connecting tubing with a low flow (about 10 ml/min) of sample gas for 5 min to 10 min. The volume flowed shall be at least 20 times the volume of the sample loop and connecting tubing. Inject the sample. If the analyser has provision to stop the sample flow before injection, then a means shall be provided to vent the gas to a by-pass line when using a needle valve or metering valve to control sample flow. If repeat analyses are going to be made, continue to flow the sample through the sample loop and connecting tubing while the analysis is proceeding.

NOTE Annex D shows examples of connections that have proved to be satisfactory.

9.2 Calculation of the composition

Components are measured relative to the peak for *n*-pentane. The quantity of *n*-pentane is derived from analysis in accordance with ISO 6974 (see 8.1). The quantity of each component, c_i , is calculated in accordance with Equation (6):

$$c_i = \frac{5 \times R_i \times c_{n-C5} \times F_{RR,C,i,n-C5}}{N \times R_{n-C5}} \quad (6)$$

where

- R_i is the instrument response to component i in the sample;
- R_{n-C5} is the instrument response to *n*-pentane in the sample;
- c_{n-C5} is the quantity of *n*-pentane in the sample, determined according to ISO 6974;
- $F_{RR,C,i,n-C5}$ is the relative carbon response factor of component i to that of *n*-pentane;
- N is the carbon number of component i .

9.3 Calculation of fraction quantities and properties

Other than the *n*-alkanes, benzene, toluene, cyclohexane and methyl cyclohexane, most peaks measured in the C₇ to C₁₂ part of the chromatogram are unidentified. They can be accounted for by using the widely

accepted assumption that (with the exception of aromatics and some cycloalkanes) all components eluting between the n -alkanes $n-C_x$ and $n-C_{x+1}$ are iso-alkanes of carbon number $x + 1$. This means that the same carbon number can be applied to those components as is used for $n-C_{x+1}$, and so quantitative values can be derived for unidentified components.

These unidentified components are summed as fractions by carbon number. Such fractions can be input into an equation of state used for dewpoint calculation, alongside individual identified components. Critical properties for individual components are available in the equation of state database, whereas those for fractions can be calculated from the average boiling points and densities of the fractions.

9.3.1 Calculation of fraction quantities

Individual peaks measured in the chromatogram are calculated according to 9.2, with the appropriate carbon-number values selected as described in 9.3. The calculated quantities for unidentified peaks that elute from immediately after $n-C_x$ up to and including $n-C_{x+1}$ are summed and the total allocated to fraction $x + 1$. Any peaks that are separately identified in this region, such as benzene and cyclohexane in the C_7 fraction, and toluene and methyl cyclohexane in the C_8 fraction, are not included in this summation, since they are accounted for individually.

NOTE Annex E shows a worked example of such a calculation.

9.3.2 Calculation of fraction properties

If the separation uses a linear temperature programme, then the boiling points of unidentified components can be inferred by linear interpolation between the values for the bracketing n -alkanes. Thus, the boiling point, $T_{BP,y}$, of a component y , which elutes between $n-C_x$ and $n-C_{x+1}$, is calculated by Equation (7).

$$T_{BP,y} = T_{BP,n-Cx} + \frac{(t_{R,y} - t_{R,n-Cx}) \times (T_{BP,n-Cx+1} - T_{BP,n-Cx})}{(t_{R,n-Cx+1} - t_{R,n-Cx})} \tag{7}$$

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where

- $t_{R,y}$ is the retention time of component y ;
- $t_{R,n-Cx}$ is the retention time of the n -alkane $n-C_x$;
- $t_{R,n-Cx+1}$ is the retention time of the n -alkane $n-C_{x+1}$;
- $T_{BP,n-Cx}$ is the boiling point of the n -alkane $n-C_x$;
- $T_{BP,n-Cx+1}$ is the boiling point of the n -alkane $n-C_{x+1}$.

The boiling point of the fraction, $T_{BP,FR}$, is then found by weighting the quantity of each component in the group by its boiling point, summing the total and dividing by the total quantity, according to Equation (8):

$$T_{BP,FR} = \frac{\sum (R_i \times T_{BP,i})}{\sum t_{R,i}} \tag{8}$$

The fraction density can be taken to be identical to that of the n -alkane within that fraction, since densities vary little among isomers.

NOTE 1 Calculated component concentrations can be used in Equation (8) in place of instrument responses, but can be more subject to rounding errors.

NOTE 2 Annex E shows a worked example of such a calculation.