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TECHNICAL SPECIFICATION
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CEN/TS 15439

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English Version

**Biomass gasification - Tar and particles in product gases -
Sampling and analysis**

Gazéification de biomasse - Goudron et particules dans les
gaz produits - Échantillonnage et analyse

Biomassevergasung - Teer und Staub in Produktgasen -
Probenahme und analytische Bestimmung

This Technical Specification (CEN/TS) was approved by CEN on 21 February 2006 for provisional application.

The period of validity of this CEN/TS is limited initially to three years. After two years the members of CEN will be requested to submit their comments, particularly on the question whether the CEN/TS can be converted into a European Standard.

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Foreword

This Technical Specification (CEN/TS 15439:2006) has been prepared by Working Group CEN/BT/TF 143 “Measurement of organic contaminants (tar) in biomass producer gases”, the secretariat of which is held by NEN.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to announce this CEN Technical Specification: Austria, Belgium, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland and United Kingdom.

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Introduction

The main contaminants in the product gases of biomass gasification are dust and soot particles, tars, alkali metals, acid gases and alkaline gases. Measuring techniques for these contaminants allow determination of the functioning of the gasifier itself, of the efficiency of the gas cleaning process and of the quality of the cleaned gas to be used in, for instance, a gas engine or gas turbine.

The development of this Technical Specification started out of the need for a reliable method for the measurement of tars. For most contaminants in product gases of biomass gasification, well-developed measurement techniques exist that are similar to techniques used for related technologies, such as coal combustion and coal gasification. For tars, however, no well-developed and widely used measurement techniques existed in these related technology fields. As some of the tars were (and are) seen as the major problem causing contaminants in biomass gasification, manufacturers and other workers in this field used a number of different sampling and analysis methods to determine the level of tars. As a result, comparison of data and definition of clear maximum allowable concentrations for tars was problematic. This formed an obstacle for market introduction of biomass gasification systems, as tars can cause damage or require an unacceptable level of maintenance.

This Technical Specification gives methods for sampling and analysis of tars and particles in product gases from biomass gasifiers operating under atmospheric or pressurised conditions. The sampling and analysis methods in this Technical Specification differ from most of the methods used for sampling organic compounds present in the gaseous emissions from various industrial processes such as flue gases or automobile exhaust gases. The differences are related to the fact that the levels of the organic compounds present in the gasification product gases exceed the levels found in flue gases generally by more than three orders of magnitude. Hence the methods described in this Technical Specification are not intended to be applicable for sampling organic compounds in trace concentrations (sub-ppm range).

The tar-containing biomass gasification product gas is formed by thermal decomposition of biomass at sub stoichiometric conditions (pyrolysis, gasification) and is typically used to produce electricity, heat, or gaseous or liquid biofuels. As tars from pyrolysis or gasification of coal are similar in nature compared to (high temperature) biomass gasification tars, coal tars can also be sampled and analysed with this Technical Specification.

Biomass in this Technical Specification is defined as material of biological origin excluding material embedded in geological formations and transformed to fossil¹⁾. The Technical Specification is developed for uncontaminated biomass, a term being defined in Clause 3 "Terms and definitions". Tests on accuracy and repeatability of the Technical Specification have been performed with uncontaminated biomass. The Technical Specification may also be used for tars produced from gasification of contaminated biomass and for tars produced from gasification of fossil fuels, however, in this case it is up to the user to assess to what extent the concentration and composition of the tars differ from gasification of uncontaminated biomass. Biomass gasifiers, as referred to in this Technical Specification, can be updraft fixed bed gasifiers, downdraft fixed bed gasifiers, stage divided gasifiers, fluidised bed gasifiers, entrained flow gasifiers and other types of gasifiers. Updraft and downdraft fixed bed, fluidised bed and entrained flow gasifiers are described in more detail in a background Technical Report [1].

¹⁾ This definition is the same as the definition of biomass in CEN TC 335 Solid Biofuels

1 Scope

This Technical Specification gives methods for sampling and analysis of tars and particles in order to determine the load of these contaminants in flowing biomass gasification product gases. The Technical Specification is applicable to sampling and analysis of tars and particles in the concentration range typically from 1 mg/m_n^3 to 300 g/m_n^3 (tars) and from 20 mg/m_n^3 to 30 g/m_n^3 (particles) at all relevant sampling point conditions (0°C to 900°C and 60 kPa to 6000 kPa ($0,6 \text{ bar}$ to 60 bar))²⁾.³⁾ Particle concentrations lower than 20 mg/m_n^3 are outside the scope of this Technical Specification and can be measured according to EN 13284-1.

Application of this Technical Specification allows determination of five different parameters:

- A. The concentration of gravimetric tar in mg/m_n^3 ;
- B. The concentration of individual organic compounds in mg/m_n^3 . This Technical Specification gives data on repeatability and reproducibility for the compounds listed in Annex B. The Technical Specification is also applicable for other organic compounds (e.g. those mentioned in Annex A), but repeatability and reproducibility have not been assessed for compounds other than those in Annex B;
- C. The sum of concentrations of identified GC-detectable compounds listed in Annex B;
- D. The sum of concentrations of all GC-detectable compounds with retention times in the range of benzene to coronene calculated as naphthalene (benzene excluded), given that this sum of concentrations can be determined.
- E. The concentration of particles in mg/m_n^3 .

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 9096

Stationary source emissions – Manual determination of mass concentration of particulate matter

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3 Terms and definitions

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

3.1

aerosol

suspension of solid or liquid particles in a gas

NOTE The term aerosol includes both the particles and the suspending gas. The particle size may range from about $0,002 \mu\text{m}$ to more than $100 \mu\text{m}$.

3.2

biomass

material of biological origin, excluding material embedded in geological formations and transformed to fossil

NOTE This definition is the same as the definition of biomass in CEN TC 335 Solid Biofuels [7].

²⁾ In fact it is not correct to give a concentration or to give concentration ranges for “tar” (see also its definition in Clause 3). This Technical Specification is designed and has been evaluated for measurement of tar and particles in the following concentration ranges:

Gravimetric tar	500 mg/m_n^3 to 300 g/m_n^3
Sum of GC-detectable tars	1 mg/m_n^3 to 300 g/m_n^3
Particles	20 mg/m_n^3 to 30 g/m_n^3

The concentration range mentioned for gravimetric tar is a range based on a sampling time of 2 h. Lower concentration limits can be attained with proportionally longer sampling times (e.g. 100 mg/m_n^3 with a sampling time of 10 h). Due to the determination method, it is recommended not to determine gravimetric tar below the concentration limit.

³⁾ The performance characteristics in Clause 14 are determined under atmospheric conditions. Under pressurised conditions, the Technical Specification as a whole has not been evaluated. However, sampling under pressurised conditions is based on relevant experience (relevant construction details of probes are mentioned in a background document [1]) and the analysis of the liquid samples is identical for atmospheric and pressurised gasification. Therefore, pressurised conditions are included in this Technical Specification.

CEN/TS 15439:2006 (E)**3.3****contaminated biomass**

biomass not being uncontaminated biomass

NOTE See 3.20.

3.4**downdraft gasification**

gasification process in which a bed of solid carbon-based material moves slowly downward under gravity as it is gasified, whilst the gasification agent (e.g. air) passes through the bed co-currently⁴⁾. The product gas leaves the gasifier at the base

3.5**entrained flow gasification**

gasification process in which carbon-based material is fed co-currently with the gasification agent (e.g. air, oxygen or steam) and where the velocity of the gasification agent is sufficiently high to maintain entrainment of the carbon-based material

3.6**fluidised bed gasification**

gasification process in which carbon-based material is fed into a suspended (bubbling) or circulating hot bed of inert particles (e.g. sand or ash), the suspension or circulation being created by the gasification agent (e.g. air, oxygen or steam)

3.7**gasification**

thermal conversion of carbon-based materials into a product gas composed primarily of CO, H₂, methane and lighter hydrocarbons in association with CO₂, H₂O and N₂ depending on the specific gasification process considered

NOTE Gasification can be accomplished by direct internal heating provided by partial oxidation using e.g. substoichiometric air or oxygen. Alternatively, concepts based on either indirect heating methods or autothermal methods using exothermic reduction reactions may be applied.

3.8**GC detection limit**

lowest concentration that can be detected by the GC equipment.

NOTE This concentration depends on the quality of the equipment and is defined as twice the noise level in the GC chromatogram.

3.9**GC-detectable tar**

tar that can be measured as a gaseous mixture of individual components according to standard (or state of the art) gas chromatographic practice

3.10**gravimetric tar**

evaporation/distillation residue from particle free sampling solution(s) determined by gravimetric analysis

3.11**isokinetic sampling**

sampling at a flow rate such that the velocity and direction of the gas entering the sample nozzle are the same as they are in the gas in the duct at the sampling point

3.12**normal conditions**

conditions with a temperature of 273,15 K, pressure of 101 325 Pa (1,013 25 bar) and on a dry basis

⁴⁾ Co-current does not automatically mean downdraft but can also mean updraft, although co-current updraft designs are uncommon.

3.13**particles**

solid residue collected by a particle filter after solvent extraction or solid residue from filtration of sampling solution(s)

NOTE Solid residue may contain a small amount of insoluble polymerised tar.

3.14**Petersen column**

alternative to impinger bottles in the sampling train. The Petersen column is a piece of glass equipment with two containers for liquid, which act as washing stages to remove soluble compounds from a gas. The two containers are separated by a glass frit. A description of the Petersen column is given in Annex C

3.15**product gas**

gas resulting from gasification

NOTE The product gas from biomass gasification can be used as a fuel (then also named fuel gas, producer gas or water gas) in internal and external combustion engines, fuel cells, and other prime movers for heat and mechanical or electrical power. Alternatively, the product gas may be used as a feedstock (then also named synthesis gas or syngas) for the petrochemical and refining industries, e.g. for the production of liquid fuels or chemicals.

3.16**pyrolysis**

thermal decomposition in the absence of an oxygen source such as air, oxygen, H₂O or CO₂

3.17**sampling train**

equipment used for sampling particles and tars, consisting of the particle filter, the impinger bottles or Petersen column, the pump and flow measuring equipment and all connecting tubes

3.18**soxhlet extraction**

batch extraction method at the boiling point of the used solvent and atmospheric pressure

3.19**tar**

generic (unspecific) term for entity of all organic compounds present in the gasification product gas excluding gaseous hydrocarbons (C1 through C6)

3.20**uncontaminated biomass**

biomass from the following sources:

- Products from agriculture and forestry;
- Vegetable waste from agriculture and forestry;
- Vegetable waste from the food processing industry;
- Wood waste, with the exception of wood waste that may contain halogenated organic compounds or heavy metals as a result of treatment with wood preservatives or coatings, and including in particular wood derived from construction and demolition waste

NOTE This definition reflects the biomass categorised as “solid biofuels“ under the scope of CEN TC 335 Solid Biofuels [7].

3.21**updraft gasification**

gasification process in which a bed of solid carbon-based material moves slowly downward under gravity as it is gasified, whilst the gasification agent (e.g. air or oxygen) passes through the bed counter-currently⁵⁾, where the product gas leaves the gasifier at the top

⁵⁾ Counter-current does not automatically mean updraft but can also mean downdraft, although counter-current downdraft designs hardly exist.

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4 Symbols and abbreviations

BTX	:	Benzene, Toluene and Xylenes
GC-FID	:	Gas Chromatography – Flame Ionisation Detector
GC-MS	:	Gas Chromatography – Mass Spectrometer
id	:	Internal diameter
ISTD	:	Internal Standard
m_n^3	:	Cubic metre at normal conditions
M/z	:	Molar mass-to-charge ratio
MW_{th}	:	Mega Watt based on the thermal input of the biomass (lower heating value)
Pa	:	Pascal
PAH	:	Polycyclic Aromatic Hydrocarbons
PTFE	:	Polytetrafluoroethene

NOTE The designation of the variables RF, M and A is explained just below the equations in which they are used.

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5 Principle of the measurement method

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5.1 Introduction

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The principle of the measurement method is based on the discontinuous extractive sampling of a representative part of a gas stream containing particles and organic compounds (tar) under isokinetic conditions. The determination of particles and tars in biomass gasification product gases is carried out in two steps: sampling and analysis.

5.2 Sampling

Samples of tars and particles are taken at a gasifier sample port, which is gas tight so that neither can gas escape from the system nor can oxygen enter it. The sampling train is configured as a heated probe with a heated particle filter to remove solid matter. The volatile tars are trapped in heated or chilled impinger bottles containing an organic liquid absorbent. The sampled gas volume is measured under dry conditions by means of a gas meter. The samples are prepared on-site and are stored until they can be analysed.

5.3 Analysis**5.3.1 Introduction**

The samples are analysed in a laboratory. The particle filter containing the particle sample is Soxhlet extracted in order to remove adsorbed tars. Subsequently the amount of particles is determined gravimetrically. The tars from the Soxhlet extraction are added to the liquid tar samples. If required, the liquid tar samples are filtered for removal of solid matter.

For the analysis of tars two methods are given in this Technical Specification, analysing respectively gravimetric tar and gas chromatographable tar. The user is free to choose either one or both methods, depending on the kind of information needed. The users attention is drawn to the fact that the two resulting values are not supplementary, but that there is an overlap between the two tar values. The user's attention is also drawn to the

fact that the gravimetric method is not suitable for clean gases with low tar concentrations and that its reproducibility is significantly poorer than the GC analysis method.

5.3.2 Gravimetric determination

Part of the homogeneous liquid sample is evaporated under well-defined conditions and the evaporation residue is weighed. The result is then recalculated to give the amount of gravimetric tar in mg per m_n^3 of product gas.

5.3.3 Gas chromatography

Part of the liquid sample is injected into a gas chromatograph (GC). This analysis results in concentrations of individual tar compounds and of the sum of GC-detectable tars, both in mg per m_n^3 of product gas.

Positive identification of the condensed material as biomass tar is performed using GC-MS. The presence of tar is indicated typically by the presence of the US-EPA suite of PAH compounds, phenols and BTX. Quantitative determination of each compound is performed either by GC-MS or GC-FID analysis using internal standards.

Please note that not all of the tar constituents are amenable to GC analysis because of the presence of high molecular weight material, thermal labile or extreme polar compounds. GC analysis will usually determine more than 80% of the tar, the remainder being e.g. high-molecular weight material of >350 g/mol. However, for updraft gasifier tars the fraction determined may be considerably lower.

6 Reagents

6.1 Solvent for tar collection and Soxhlet extraction

Isopropanol (2-propanol) shall be used as the solvent for tar collection, for Soxhlet extraction and for sample preparation and analysis. The minimum purity of the isopropanol is 99%. It shall be verified with a blank GC determination that the solvent does not contain GC detectable amounts of relevant tar compounds.

Ethanol shall be used in gravimetric analysis procedure to remove trace water from evaporation residue (gravimetric tar). The required minimum purity is 99%.

Other solvents like ethanol or dichloromethane shall only be used for rinsing of the glass and PTFE equipment when cleaning with isopropanol leaves tar residues that can be visually observed.

6.2 Carrier gas in gas chromatography

The carrier gas shall be helium of acceptable purity or a suitable alternative as specified by the GC manufacturer.

6.3 Calibration standards

All reagents shall be of recognised analytical reagent quality, preferably analytical or "pro analysis" grade. The tar compounds are named in Annex A and Annex B.

7 Equipment

7.1 Equipment for sampling

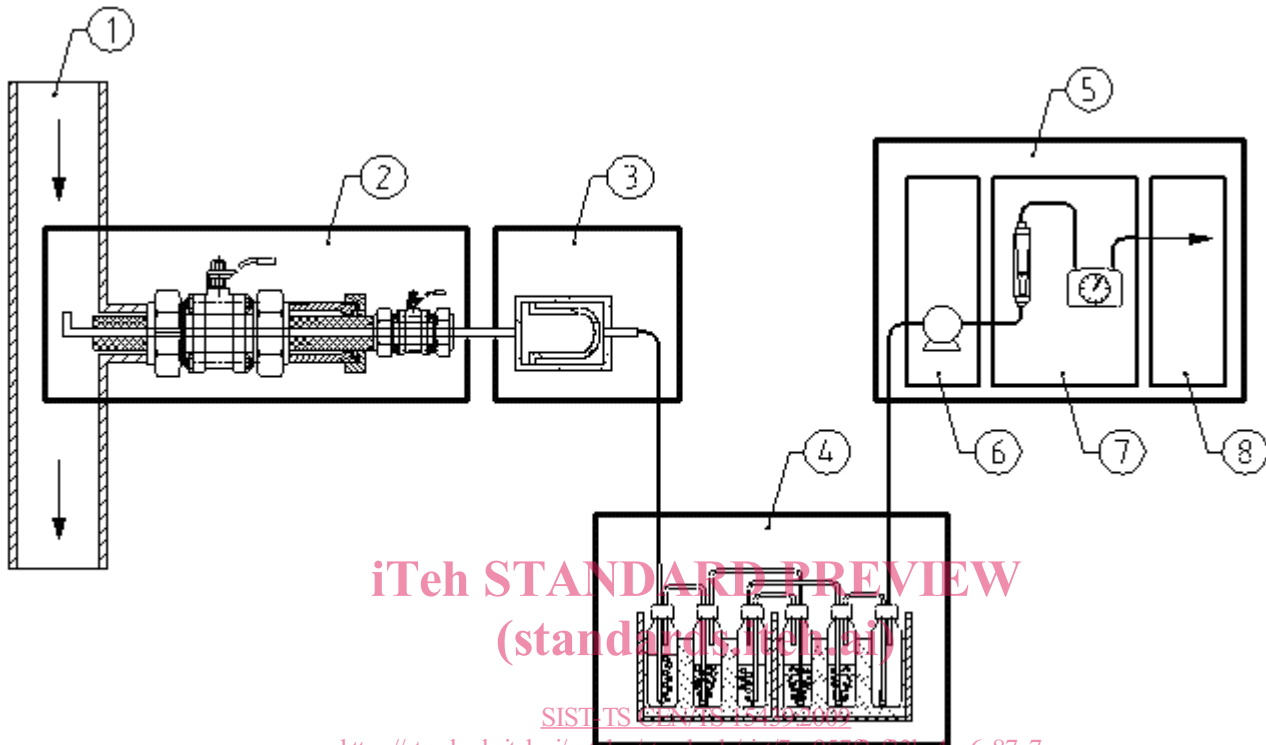
7.1.1 Description of the sampling equipment

The equipment for sampling consists of a heated probe, a heated particle filter, a condenser, a series of impinger bottles containing a solvent for tar absorption, and equipment for pressure and flow rate adjustment and measurement. Upstream of the condenser the tubes connecting these parts are heated in order to prevent tar condensation. Temperatures for heating the probe, filter and tubes are specified to avoid both condensation of tars and thermal decomposition of tars. Temperatures for the condenser and the impingers are specified to

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ensure quantitative collection of the tars. An explanation for the choice of equipment and conditions is given in a separate document entitled "Rationale for setup" [2].

The sampling equipment consists of four main modules and respective sub-modules. The main modules are gas preconditioning, particle collection, tar collection and volume measurement. These modules are shown in Figure 1. The basic equipment of these modules is mentioned in the next paragraph in Table 1. A more detailed description of each module is found in a background Technical Report [1].



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Key

- 1 Gasflow
- 2 Module 1
- 3 Module 2
- 4 Module 3
- 5 Module 4
- 6 Sub-module 4.1
- 7 Sub-module 4.2
- 8 Sub-module 4.3

Figure 1 — Concept of the modular sampling train

The equipment needed for sampling is mentioned in Table 1. Examples and details on construction are described in a background Technical Report [1], including the adaptations needed for module 1 if tars and particles have to be sampled from pressurised gasifiers.

Table 1 — General description of modules and sub-modules with purpose and equipment used

	Function	Main equipment	
Module 1 (Gas preconditioning)	Gas cooling, pressure reduction	Nozzle, valves, sampling lines	
Module 2 (Particle collection)	Separation and collection of solids	Heated filter (high temperature)	
Module 3 (Tar collection)	Moisture collection and partial tar condensation	<u>Option 1</u> Condenser with solvent (1 impinger bottle)	<u>Option 2</u>
	Tar collection	Impingers with solvent (4 impinger bottles, some with glass frits)	Petersen column (see Annex C)
	Drop collection	Empty impinger (1 bottle with a glass frit)	
Module 4 (Volume registration)			
Sub-module 4.1	Gas suction	Gas drying, pump	
Sub-module 4.2	Gas volume integration	Gas meter, needle valve (adjustment and control of flow rate), rotameter (flow indication), indicators for temperature and differential pressure, barometer	
Sub-module 4.3	Off-gas handling	Outdoor ventilation	

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7.1.2 Requirements for sampling equipment

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The sampling equipment shall meet the following functional requirements:

- When using impinger bottles of standard design (NS 29/32-250), flow rates through the impinger bottles shall be between 0,1 m_n³/h and 0,6 m_n³/h. For high gas velocities, requiring a flow rate through the nozzle higher than 0,6 m_n³/h to meet isokinetic conditions, this requirement plus the requirements on the minimum nozzle diameter and on isokinetic sampling shall be met by splitting the gas flow between the nozzle and the impinger bottles.
- The sampling equipment allows isokinetic sampling according to ISO 9096. If only tars are analysed, isokinetic sampling is still required in all cases with the following exceptions: (1) for pressurised gasifiers; (2) for large-scale gasifiers with large pipe diameters (for example in ISO 9096 duct diameter shall be < 0,35 m for only one sampling point); and (3) if only tar is sampled and the temperature of the gasification product gas at the sampling site exceeds 350 °C. Isokinetic sampling demands an undisturbed gas flow. Recommendations on how to measure isokinetically according to ISO 9096 are given in a background Technical Report [1].
- For pressurised gasifiers, the product gas temperature at the location of sampling shall exceed 350 °C.
- The equipment shall be gas tight.
- The minimum nozzle diameter shall be 4 mm according to ISO 9096. For isokinetic sampling, the nozzle shall be designed according to ISO 9096.
- Temperatures of the sampling line and the particle filter shall be:
 - between 100 °C and 125 °C for updraft gasification;
 - between 300 °C and 350 °C for downdraft, fluidised bed and entrained flow gasification.
- Gas velocities from the nozzle to the particle filter shall be higher than 25 m/s to avoid particle deposition.