GDAŃSK UNIVERSITY OF TECHNOLOGY

Subject card

Subject name and code	Technical Analytics and Quality Control, PG_00066035		
Field of study	Engineering and Technologies of Energy Carriers		
Date of commencement of studies	February 2025	Academic year of realisation of subject	2024/2025
Education level	second-cycle studies	Subject group	Obligatory subject group in the field of study Subject group related to practical vocational preparation
Mode of study	Full-time studies	Mode of delivery	at the university
Year of study	1	Language of instruction	Polish
Semester of study	1	ECTS credits	4.0
Learning profile	practical profile	Assessment form	exam
Conducting unit	Department of Process Engineering and Chemical Technology -> Faculty of Chemistry		
Name and surname of lecturer (lecturers)	Subject supervisor	dr inż. Patrycja Makoś-Chełstowska	
	Teachers	dr inż. Patrycja Makoś-Chełstowska dr inż. Karolina Kucharska dr inż. Edyta Słupek	

Lesson types and methods of instruction	Lesson type	Lecture	Tutorial	Laboratory	Project	Seminar	SUM
	Number of study hours	20.0	0.0	60.0	0.0	0.0	80
	E-learning hours included: 0.0						

Learning activity and number of study hours	Learning activity	Participation in didactic classes included in study plan	Participation in consultation hours	Self-study	SUM
	Number of study hours	80	5.0	15.0	100

Subject objectives	To acquire and comprehend the fundamental principles and specific methodologies of technical and industrial laboratory analytics, as well as the elements of process analytics and quality control for various types of fuels, including: • Gaseous fuels, • Liquid fuels, • Solid fuels, • Raw materials, • Process streams designated for further processing, • Components intended for blending, • Auxiliary materials such as reaction agents, catalysts, sorbents, auxiliary fluids (e.g., diluents), and process and exhaust gases.

Learning outcomes

Course outcome	Subject outcome	Method of verification
[K7_W07] selects analytical techniques appropriate for solving specific technological tasks in a production plant	Theoretical and practical knowledge about the physicochemistry of individual research techniques and methodologies.	[SW3] Assessment of knowledge contained in written work and projects [SW2] Assessment of knowledge contained in presentation [SW1] Assessment of factual knowledge
[K7_U03] uses analytical, simulation and experimental methods of chemistry, physics and chemical engineering and technology to formulate and solve complex engineering tasks, including non-standard tasks, as well as simple research problems	Practical ability to formulate research problems and select appropriate analytical methods for effective resolution, utilizing advanced computational and analytical tools. Capability to critically evaluate the obtained results.	[SU4] Assessment of ability to use methods and tools [SU2] Assessment of ability to analyse information [SU1] Assessment of task fulfilment
[K7_U02] is able to plan and conduct experiments, interpret the obtained results and draw conclusions	Theoretical and practical knowledge allowing verification of the correct implementation of complex processes and technologies on the basis of a planned cycle of research in the field of technical analytics and quality control.	[SU4] Assessment of ability to use methods and tools [SU1] Assessment of task fulfilment
[K7_U04] prepares a critical analysis of existing technical solutions and is able to propose their improvements (improvements).	Theoretical and practical knowledge on the selection of methodology for conducting qualitative and quantitative research on individual energy carriers. Ability to adapt existing techniques and methods to new applications.	[SU3] Assessment of ability to use knowledge gained from the subject [SU1] Assessment of task fulfilment

Subject contents

Technical analysis and quality control in the field of engineering and technology of energy carriers, encompassing various types of fossil fuels, biofuels, and fuel cells (including raw materials, process streams, finished products, auxiliary and process materials, flue gases, etc.):

- Gaseous fuels: natural gas, LPG, coke oven gas, gases from biomass conversion to gaseous fuels (bio-CH, bio-H)
- Liquid fuels: crude oil and its fractions, fuels from pyrolysis of wood, coal, and waste, bioethanol, biobutanol, petroleum condensate, liquid fractions from fluidized catalytic cracking (FCC), catalytic cracking, catalytic isomerization, catalytic reforming, etc.
- Solid fuels: coal, lignite, peat, charcoal, biomass (firewood, various types of straw, oilcakes), solid waste sludge from wastewater treatment, animal waste, etc.
- Fuel cells: hydrogen, methanol, ethanol, etc.

Techniques and methods in technical analytics and quality control include:

- Physical and physicochemical techniques and methodologies for studying basic properties, performance parameters, and the content of desirable and undesirable components in raw materials and fuels. Techniques include XR spectrophotometry (XRF/XRD), UV-VIS, MIR, H^1 and C^{13} NMR, ignition mineralization in synthetic air or a reducing air-hydrogen flame, with various detection and analytical methods such as IR, coulometric, potentiometric, and ion exchange.
- Sorption-desorption and ion exchange techniques, including K-GC, C-GC, HPLC/UPLC, TLC/HPTLC chromatography.
- Absorption and emission atomic spectroscopy and mass spectrometry (AAS, ICP-AE, ICP-MS).
- Classical or instrumental titration, including electrochemical methods (conductivity, potentiometry, voltammetry, coulometry), and determination of water content by the Karl Fischer method.

Additionally, the determination of repeatability/reproducibility in test procedures, analysis of error causes, measurement uncertainty, adherence to national or international measurement standards, validation of test procedures, and equipment supervision are critical aspects of technical analytics and quality control.

Prerequisites and co-requisites

Knowledge of inorganic and organic chemistry as well as physical, analytical chemistry and chemical technology.

Assessment methods and criteria

Subject passing criteria	Passing threshold	Percentage of the final grade
Laboratory	60.0%	30.0%
Exam	60.0%	70.0%

Recommended reading

Basic literature

J.G. Speight, Handbook of Petroleum Analysis, WILEY-Interscience, 2015

J.G. Speight, Handbook of Coal Analysis, WILEY-Interscience, 2005

Standard test methods PN/EN, ASTM, GLP/GMP; PN-EN-ISO 9001; PN-EN-ISO/IEC 17025

	Supplementary literature	1. Z. Witkiewicz, Podstawy chromatografii WNT, W-wa, 2005. 2. M. Kamiński (ed.) Chromatografia Cieczowa, CEEAM, Gdańsk, 2004. 3. J. Weiss, Handbook of ion chromatography, vol. 1,2, Willey-VCH 2004. 4. W. Zieliński, A. Rajca (red.): Metody spektroskopowe i ich zastosowanie do identyfikacji związków organicznych, WNT, W-wa, 1995. 5. J. Cazes (ed) Encyclopedia on Chromatography, Marcel Dekker, New York, 2001 (or newer edition) 6. J. Namieśnik, P. Konieczka, Kontrola i zapewnienie jakości wyników pomiarów analitycznych, PTIE, 2006.
	eResources addresses	Adresy na platformie eNauczanie:

Example issues/ example questions/ tasks being completed	**LECTURE** **Module I** - General principles of technical analytics and quality control I.1. Technical analytics - on-line, at-line and off-line measurements and testing; Process analytics - automatic, process analytics - laboratory analytics, quality control of raw materials and products, other technical analytics (test movements, testing of auxiliary materials - water, reagents, catalysts, sorbents, other auxiliary materials, working environment, etc.); I.2. Selected issues of test quality management systems - basic principles of PN-EN 9001, a reminder and the most important principles in practice according to: PN-EN ISO/IEC 17025, GLP and GMP; Supervision of test equipment; Inter-laboratory studies; Proficiency testing; Measurement consistency and its implementation in practice; Calibration; Standards; Certified reference materials; Full / simplified validation - concept, meaning, test procedure, most important parameters tested, reporting, conditions for authoritative validation; Statistical methods in supervision of test quality and in supervision of test equipment; I.3. . Sources of requirements for technical products and intermediates; process streams - legal regulations, standards, specifications, internal documentation, contracts, quotations, principles of rounding numbers, recording results, and comparing test results with requirements; statistical elaboration of test results - a reminder of basic principles; concepts and applications: repeatability, reproducibility; basic principles of the Law on Weights and Measures and their application in equipment supervistion/fuel testing; I.4 . organizational, technical, and material aspects of a modern technical analytical laboratory; I.5. Sample representativeness and randomness of sampling. Methods and equipment of collection, samples, safety of collection, transportation, handling, and preparation of fuel samples; principles of archiving and storage of archived samples. **Module II** The most important techniques and methodologies for the study of raw materials, process streams, finished products, auxiliary materials, process streams and materials, water, wastewater, flue gases, working environment, etc., fuels : • gas (natural gas, LPG, coke oven gas, gases from the conversion of biomass to gaseous fuels (bio-CH4, bio-H2)), • liquid (crude oil and fractions from its processing, gasolines, JET fuels, fuel oils, fuels from pyrolysis, wood, coal and waste, bio-ethanol, bio-butanol, petroleum condensate fuel liquid fractions from thermal catalytic cracking (FCC), catalytic hydro-cracking, catalytic isomerization, catalytic reforming, graphitization, ...), • solids (coal, lignite, peat, charcoal, biomass - firewood, straw of various types, oilcakes, solid waste sludge from wastewater treatment, animal waste, ...) • fuel cells (electrode materials, liquids, gases, and finished cells). II.3 Preparation for testing, samples: gaseous, liquid, solid-combustion, absorption, adsorption-desorption, extraction/leaching, incineration, grinding, pressing, digestion, microwave mineralization, precipitation with filtration, and drying to solids. II.4 . Methodologies for the study of physicochemical, utility, technical parameters in technical analysis and quality control - basic/advanced parameters: • gravimetric parameters, • temperature parameters, • distillation parameters (atmospheric distillation, vacuum distillation, SIMDIS, 15-shelf distillation), • thermal parameters (heat of combustion, calorific value, heat of phase / chemical transformation, sorption - desorption), • technical (numbers: octane, cetane, bromine, iodine, maleic, peroxide / oxidation resistance / resins / conductivity, flash point, ...), • refractometric, colorimetric, photometric, spectrophotometric, fluorescence, • average value and molecular weight distribution, • Determination of paraffinic, naphthenic and aromatic (Cp/Cn/Ca) groups using n-d-m / FTIR / H1NMR methodology, • crystal structure, • water content. II.5 General principles of chemical compound identification / chemical compound content determination / chemical compound groups II.6 . Techniques and methods of sample preparation and identification and determination of the content of elements (especially: C, H, N, S, X, O, P, Si, Al, Na, K, Ca, Mg, Fe, Ni, Mo, Mn, ...) by classical / instrumental techniques and methods: • X-ray fluorescence (XRF), • atomic absorption (AAS),

- ICP-OES / ICP-MS,
- ignition mineralization in an atmosphere of synthetic air / in a reducing hydrogen flame (Wickboldt),

II. 7 . Techniques and methods of identification/study of group / detailed composition (purity, content of selected chemical compounds / their groups / ions) and analyzers for such studies:

- classical techniques (S-2, Cl-, SO42-, TLC, TLC-FID, N-Kjehdala, water - K-F, water - acetylene method)
- instrumental: single and large-scale gas chromatography in the analytics of gases / volatile mixtures of organic compounds with FID, TCD, MS, PID, ECD detection,
- high performance liquid chromatography adsorption and ion exchange / ion chromatography with RID / UV-VIS-DAD, ELD, conductometric, FLD, MS ,
- spectroscopic techniques and methods MIR - FTIR, UV-VIS,
- coulometry, potentiometry, and volt amperometry inversion.

II.8. Chemometric techniques and methods, particularly in the near-infrared (NIR) range.

II.9. coupled techniques and methods (ignition digestion - IExchC / LC-GC / GC-LC)

Module III. Development and validation of new research methodologies.

II.1 Sources of sample preparation and testing methodologies and how to analyze them as inspiration to develop your own solutions - standardized methodologies, patent descriptions, scientific publications

II.2 Newly developed / adapted testing methodologies - case study

LABORATORY 12 exercises, 5-hour exercises

- Each exercise concerns the study of at least three parameters; each student performs the study of all parameters.

Topics of individual laboratory exercises

1. The basic parameters of the selected energy carriers (CH. D): density, refractive index, color, UV-VIS spectrum, kinematic viscosity, presence of water and impurities + determination of laboratory reproducibility of the test method/degree of contamination.

Study of the composition of gas mixtures by gas chromatography technique - gaseous energy carriers - packed / capillary columns of pora-plot type - 5A/3A, Q/N, SiO2/Al2O3, Carbopak/Cakt - GC-TCD; GC-FID; GC-FPD (CH. D) (hydrogen gas, LPG, digester gas, and flue gas compositions).

Liquid fuel/liquid process stream composition testing: gasoline/gasoline fractions/bioethanol-CGC-FID, CGC-FPD, CGC-PID, CGC-MS (CH. D);

Composition study of mixtures by high-performance liquid chromatography technique - NP-HPLC-BF-UV-DAD/RID/LLSD // TLC-FID (CH. C) (IP-391, EN-12-916:2010 - ON with FAME, TLC-FID, and heavy fuel oil.

Testing (determination) of sum parameters of complex mixtures: distillation temperature distribution (classical distillation/SIMDIS) - molecular weight distribution - GPC-SEC - UV-VIS-DAD/RID/LLSD - fractions A, C, D, TAG, and FAME (CH. C);

Combustion/microwave mineralization techniques as sample preparation techniques for the testing and determination of elements: Na, Fe, Ni, Si, Al-ASA/ion exchange chromatography (SO42-, Cl-, Na+ / NH4+, Ca+2, Mg+2, Fe+2, MEA, DEA, and MDEA (CH. D);

Group composition study by NP-TLC / RP-TLC technique with visualization at 365 nm / 254 nm / J2 / berberine sulfate // UV-VIS, FTIR (investigation of the presence and approximate content of saturated hydrocarbons/aromatic resins / FAME / acyl-glycerols (TAG/DAG/MAG), PAC / Glycerol in ON/FAME / ON+FAME (CH. C);

Testing the composition of acid / alkaline / enzymatic hydrolysate BMLC by RP-HPLC / HILIC-HPLC / IExclC- HPLC // RP-TLC / HILIC-TLC (CH.C)

	Testing of wastewater after petroleum refining processes: BOD, COD, and content of selected VOC groups (DLLME-HS-GC-MS, GC-FID, GC-NPD, GC-FPD). (CH.D); Testing of oxidation resistance of gasoline/ON, lubricating properties, injection pressure, ignition temperature in closed tyle-ON, presence of ON in gasoline-mirror method (CH.C); Determination of water in liquid fuels - by K-F method, moisture in BMLC - by drying method at 130C, ash in BMLC - in muffle furnace (CH.C) 12. Departure laboratory to Lotos LAB or other- principles of QMS according to PN-EN-9001 / 17025 + fuel laboratory - density / viscosity - vibrationally, pressure, oxidation resistance (induction period of gasolines, oxidation resistance of ON) LOB / LOM / cetane number, resins present, CGC - laboratory process analysis of gasoline fractions - PIONA, oxygen compounds in gasoline, SIMDIS fr. fuels, JETA1 (JEFTOT, flash point by TAG method, corrosion on silver, electrical conductivity, H2S and mercaptan sulfur - potentiometrically), S content in gasoline/ON-XRF, Clorg/SorgSorg / Norg content in petroleum gasoline fraction by ignition mineralization method with coulometric/fluorescence determination.
Work placement	Not applicable

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