Revision 2023

GalChimia

Analytical Services

Introduction

Our Analytical Department provides support to our synthesis teams as an integral part of our Discovery and Process Chemistry services, but it can also be contracted as a separate service. Years of experience working with complex molecules have built a strong know-how allowing us to provide solutions to a wide range of analytical challenges. We can provide full characterization of chemical products, as well as services on Method Development and Identification and Characterization of impurities and metabolites.

GalChimia
passion for chemistry

GalChimia is a CRO that offers specialized services in Discovery, Process, and Analytical Chemistry for the pharma, biotech, and agrochemical industries. The passion for chemistry of GalChimia's team has become a referent throughout the years, transforming the company into a European leader in Chemical Synthesis. We not only make molecules, we foster innovation and provide a dynamic environment to continually push the boundaries of organic chemistry and help you succeed in your R&D projects.

1. Equipment and Services

1.1. Chromatography Service

From routine testing, characterization, and purification of complex chemical molecules to impurity determination, as well as method development or specific analytical studies, our team of dedicated and experienced Analytical Chemists will provide results on a fast timescale.

Method verification

The analytical method is provided by the sponsor and GalChimia verifies that it complies with the intended purpose of use.

This service includes assessment of the following parameters:

- Specificity
- Linearity and LLOQ (where applicable)
- Accuracy
- Repeatability

Development of a new method

Development of a new analytical method for the intended purpose of use based on molecular structure information provided by the sponsor.

This service includes assessment of the following parameters:

- Specificity
- Linearity and LLOQ (where applicable)
- Accuracy
- Repeatability
- System suitability
- Preparation of draft method for validation

Type 1 Validation. Quantitative test for a chemical product

Validation of an analytical method to quantify a chemical product in a sample, based on the draft method defined previously in the verification or development stage.

This service includes assessment of the following parameters:

- Specificity
- System suitability
- Linearity
- Accuracy
- Repeatability
- Precision
- Robustness

Type 2 Validation. Quantitative test for impurity content

Validation of an analytical method to quantify the impurity content in a sample, based on the draft method defined previously in the verification or development stage.

This service includes assessment of the following parameters:

- Specificity
- System suitability
- Linearity
- Determination of LOD (where applicable) and LOQ
- Accuracy
- Repeatability
- Precision
- Robustness

Type 3 Validation. Limit test for the control of impurities

Validation of an analytical method for the control of impurity threshold, based on the draft method defined previously in the verification or development stage.

This service includes assessment of the following parameters:

- Specificity
- System suitability
- Determination of LOD
- Robustness

1.2. Chromatography Equipment

High Performance Liquid Chromatography

- Waters 717 plus Autosampler
- Waters 2996 Photodiode Array Detector (PDA)
- Waters 600 Pump and Controller

High Performance Liquid Chromatography / Mass Spectrometry

- Waters Alliance HT 2795 Separation Module
- Waters 2996 Photodiode Array Detector (PDA)
- Waters Micromass ZQ 2000 (Single Quadrupole) with Electrospray Ionization (ESI)

Ultra Performance Liquid Chromatography

- Acquity H-Class UPLC system
- Acquity PDA Detector
- Acquity QDa Detector with Electrospray Ionization (ESI)
- Waters 2424 ELS detector

Gas Chromatography / Mass Spectrometry

- Agilent 7820A GC System
- Agilent G4513A Automatic Liquid Sampler
- Agilent G4331-60516 Flame Ionization Detector (FID)
- Agilent 5977E Mass Detector (Single Quadrupole) with Electron Impact Ionization (EI).

High Performance Semipreparative Liquid Chromatography

- UltiMate 3000 HPLC Semipreparative
- UltiMate 3000 DAD Detector
- Teledyne Isco Foxy R1 Fraction Collector

Supercritical Fluid Chromatography

- Agilent Shimadzu Nexera UC system
- Shimadzu SPD-M40 PDA (Photodiode Array Detector)
- Shimadzu CTO-40C 6-column oven

Preparative Supercritical Fluid Chromatography

- Shimadzu Nexera UC Prep system
- Shimadzu SPD-M40 PDA equipped with a preparative flow cell
- Shimadzu FRC-40 fraction collector, SFC work mode

1.3. Titration Service and Equipment

Determination of water level or Humidity (volumetric Karl-Fischer titration): amperometric titration used for the determination of the percentage of water in a sample, either as part of a hydrated molecule or as free water.

Determination of acidity or basicity: potentiometric acid-base titration used to determine the acidity or basicity of a compound bearing acidic or basic groups.

Determination of richness (with perchloric acid in glacial acetic acid): potentiometric non-aqueous acid-base titration used to determine the richness of basic compounds. It can be applied to amine evaluation.

Determination of chloride (argentometry): potentiometric precipitation titration used to determine the amount of chloride (Cl^-) present in a sample. It can be applied to hydrochloride evaluation.

pKa determination: potentiometric acid–base titration performed to determine the pKa of a compound. The methodology is selected based on the solubility of the substance in water.

Metrohm 888 Titrando

- Metrohm 806 Exchange Unit (10 mL buret) × 4 units
- Metrohm 806 Exchange Unit (1 mL buret) × 1 unit
- Metrohm 801 Magnetic Stirrer

1.4. Thermogravimetric Service and Equipment

Volatile content: measurement of sample weight loss under specific values of temperature, typically 105 °C.

Inorganic residue content: measurement of sample weight loss after a calcination process carried out under oxidative atmosphere at a minimum temperature of 850 °C.

PerkinElmer TGA 4000

- Temperature range: ambient to 1000 °C
- Atmosphere: static or dynamic, including nitrogen and air
- Balance sensitivity: 1 µg
- Balance precision: ± 0.01%
- Balance accuracy: ± 0.02%

2. Details

2.1. Method development and validation

Development of analytical LC or GC methods	
Method verification	**Scope**: verification of the method to comply with its intended purpose of use.
Development of a new method	**Scope:** evaluation of a new method based on structure information provided by the sponsor.
Type 1 Validation. Quantitative test for a chemical product	**Scope:** validation of an analytical method to quantify a chemical product in a sample.
Type 2 Validation. Quantitative test for impurity content	**Scope:** validation of an analytical method to quantify the impurity content in a sample.
Type 3 Validation. Limit test for control of impurities	**Scope:** validation of an analytical method for the control of the impurity limit.

2.2. Studies

	Scope
Kinetic solubility	Evaluation of the solubility grade of a compound in a selected medium.
Thermodynamic solubility	Evaluation of the solubility of a compound as a saturated solution in equilibrium.
Accelerated degradation	Evaluation of the compound stability under forced conditions. Low, room and high temperature (-18 to 75 ºC); Basic, neutral and acidic conditions (pH 1,1 to 9); Presence of oxidant agents (H_2O_2).
Custom study	To be determined

2.3. Analyses

Technique	Detection / Methodology	Delivery*	Required quantity per test**
HPLC - achiral	PDA MS (ESI)	2-3 days	1-5 mg
HPLC - chiral	PDA	3-5 days	3-8 mg
UPLC	PDA MS (ESI) ELSD	2-3 days	1-5 mg
GC	FID MS (EI)	2-3 days	1-5 mg
SFC - chiral	PDA	3-5 days	3-8 mg
Semipreparative HPLC - achiral	DAD	Based on complexity and sample amount	up to 1 g
Semipreparative HPLC - chiral	DAD	Based on complexity and sample amount	up to 1 g
Preparative SFC - chiral	PDA	Based on complexity and sample amount	up to 1 g
TGA	Volatile content Inorganic residue content	2-3 days	20 mg 40 mg
Automatic titration	Karl-Fischer Chloride content Acid content Basic content Richness	2-3 days	0,5-1 g 30 mg 300 mg 300 mg 300 mg
	pKa determination *Method 1: Water soluble compounds* pKa determination *Method 2: Co-solvent determination*	2-3 days	30-200 mg*** 150-800 mg***

* From sample reception date.
** In the case of duplicate or triplicate testing, the price will increase accordingly.
*** Required quantity depends on the molecular weight of the compound. The reported quantities have been calculated considering compounds with molecular weight ranging 150–800 Da.