



IGC-SEA - 3000 Series

Inverse Gas Chromatography Surface Energy Analyzer





EPCC / PRODUCTS / APPLICATION / SOFTWARE / ACCESSORIES / CONSUMABLES / SERVICES

Analytical Technologies Limited

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SURFACE ENERGY

• The Key to Understanding Surface Properties

The factors which control the behavior and performance of many particulate solids, powders, fibers and films are often poorly understood. Such solids often display problems during manufacture, usage or storage across all industrial sectors.

Typically, particulate solids are subject to cursory characterization from a physical chemistry perspective, and often all that is known is the particle size or BET surface area of the solid. Contrast this with the detailed analytical chemical information, including the chemical structure and morphology as determined by NMR, FTIR, XRD, GC-MS and HPLC, which is routinely available. However, none of this information describes the thermodynamic state of the material. Researchers have now established that one of the most important properties of a powder, particulate material, film or fiber is its surface energy.

Surface energy Y, is the principle characteristic of solids measured by the Inverse Gas Chromatography-Surface Energy Analyzer (iGC- SEA). The surface energy of a solid is analogous to the surface tension of a liquid and it is a measure of attractive intermolecular forces on a solid surface.



High Surface Energy Low Surface Energy

It is the same intermolecular forces which are responsible for the attraction between powder particles and other solids, liquid and vapor molecules which can occur via long range van der Waals forces (dispersion forces) and short range chemical forces (polar forces). Thus, surface energy values (dispersive and polar) correlate to several key solid properties including wetting, dispersability, powder flowability, agglomeration, process-induced disorder, adhesion/ cohesion, static charge, adsorption capacity and surface chemistry.

The iGC-SEA probes the solid surface interface by exposing the solid sample to vapor probes of known properties. The intermolecular forces that result from this interaction can be analyzed to quantify the total surface energy of the sample.

Experimental Technique for Measuring Surface Energy





There are a range of techniques available for measuring the surface energy of solid particulate materials. Though contact angle measurement is by far the most common method, it is rarely used for particle and other non-planar materials due to experimental limitations leading to inaccurate and unreliable results. Inverse gas chromatogrphy is now the proven and preferred method for surface energy measurements, and surface energy heterogeneity in particular.

• Inverse Gas Chromatography (iGC)

Inverse Gas Chromatography (iGC) is a gas-solid technique for characterizing surface and bulk properties of powders, particulates, fibers, films and semi-solids. A series of vapor pulses are injected through a column packed with the sample of interest. Unlike traditional analytical gas chromatography, iGC is a physical chemistry technique using vapor probes with known properties to characterize the unknown surface/ bulk properties of the solid sample.

• The chart below shows different techniques and capabilities for measuring surface properties.

Inverse Gas Chromatography (iGC)	Atomic Force Microscope (AFM)	Contact Angle (CA)
Ok for flat surfaces.	Ok for flat surfaces.	Excellent for flat surfaces.
Excellent for particulates	Not well suitable for	Not suitable for
repeatable, no-hysteresis or roughness effects.	particulates - slow and poor data statistics.	particulates - swelling, hysteresis, dissolution, surface
Surface energy heterogeneity.	Theory for determining surface	roughness.
Can measure vapor adsorption isotherms as well as surface area.	energy can be complex.	Very few solutes possible.

Wetting Balance	
Excellent for flat surfaces. Not suitable for particulates - swelling, hysteresis, dissolution, surface roughness.	•
Very few solutes possible.	





IGC-SEA for Measuring Surface Energy

• What is iGC-SEA?

iGC- SEA or Inverse Gas Chromatography-Surface Energy Analyzer is an instrument that uses the iGC principle. The heart of its innovation is the patented injection manifold system which generates accurate solvent pulse sizes across a large concentration range, resulting in isotherms at unprecedented high and low sample surface coverages. This allows for the accurate determination of surface energy heterogeneity distributions.

iGC-SEA has a unique data analysis software, specifically designed to measure surface energy heterogeneity, isotherm properties and related physical termodynamic parameters. Further, bulk solid property experiments resulting from probe bulk interaction and using solubility theory are now possible. It automatically and directly provides a wide range of surface and bulk properties of the solid samples and gives more accurate and reliable data than manual calculations.

iGC-SEA also has a humidity control option, thereby the **impact of humidity** and temperature can be determined for the physico chemical properties of solids such as as moisture induced Tg, BET specific surface area, surface energy, wettability, adhesion and cohesion.

• iGC-SEA is used in characterizing particles, powders, fibers, films, nanomaterials, composite components and bulk phase.

• Industries which benefit from iGC-SEA:

Aerospace	Energy	Nuclear
Building Materials	Food	Pharmaceutical
Chemical	Composite Materials	Personal Care

Surface Energy Heterogeneity Profiling

Dispersive Surface Energy **Profiles** Budesonide Samples



Dispersive Surface Energy Distributions

Budesonide Samples

The surface energy distribution is the integration of the surface energy profile across the entire range at surface coverage and is analogous in principle to a particle size distribution.



>> Adsorption Isotherms, Heats of Adsorption & Henry Constants

Series of pulses for a multiple injection experiment (variable concentration) on M745 with hexane at 303 K.

Sorption isotherms of hexane by pulse injections on M745.



Dispersive and Acid-Base Surface Energy/Chemistry

Dispersive surface energy values for different proton exchange membranes as a function of background relative **humidity** conditions.



Gutmann acid (K_a) and base (K_b) values for different polymers along with their relative ranking on the **triboelectric series.**

Triboelectric Series Order	к _b /к _a
Polymethyl metharcrylate	1.33
Polycarbonate	1.10
Acrylonitirile	1.09
Polybutadiene-Styrene	
Polypropylene	0.63
Polyvinylchloride	0.02

Works of Adhesion and Cohesion

Work of adhesion and work of cohesion values for different nanofiller-polyurthane composites correlate directly with composite mechanical properties. There is a good blending performance with a W_{ad}/W_{coh} ratio near 1.

Sample	$W_{ad}^{\prime}/W_{coh}^{\prime}$	Tensile Strength at Break (MPa)
Polyurethane Alone		61+4 -
As Received	0.55	60+7 -
Multi-wailed Nanotube	L Catendary	92007-07
Oxidized Multi-walled	0.49	56-6
Nanotube		
As Received Nanoclay	0.47	54-11
Functionalized Nanoclay	0.86	71-7 -



>> Spray Dry (SD) and Freeze Dry (FD) Product Evaluation

The SD product/sample has an energetically more homogenous surface due to a more uniform particle size and shape. The FD product/sample exhibits a wide variation of surface energy sites.



Solubility Parameters (Hildebrand and Hansen)

Hildebrand solubility parameter for Polymethyl Methacrylate. Value of 19.08 MPa^{$\frac{1}{2}$} agrees with literature values (17.4-21.3 MPa^{$\frac{1}{2}$}).



"iGC-SEA represents a huge advance in inverse gas chromatography and physico-chemical measurement."

APPLICATION NOTES

201 Gas phase diffusion studies of cyclohexane by infinite dilution inverse gas chromatography.

202 Determination of the dispersive surface energy of Paracetamol by pulse inverse gas chromatography at infinite dilution.

203 Heat of sorption studies on micro crystalline cellulose by pulse inverse gas chromatography at infinite dilution.

204 Determination of the glass transition temperatures Tg of maltose and its dependence on relative humidity by infinite dilution inverse gas chromatography.



205 The determination of the solubility parameter of different starch types by infinite dilution inverse gas chromatography.

206 Determination of permeability coefficients of alkanes in polyethylene powder by infinite dilution inverse gas chromatography.

207 Characterization of drug polymorphs by inverse gas chromatography.

208 The measurement of isotherms by pulse inverse gas chromatography.

209 An investigation of Chromosorb silicas as support materials for inverse gas chromatography.

210 Investigation of the influence of bleaching conditions on surface properties of standard hair samples by inverse gas chromatography.

211 An investigation of minerals used in asphalt by inverse gas chromatography.

213 Determination of thermodynamic parameters by frontal inverse gas chromatography.

214 The determination of the permeability and the activation energy of diffusion of drug powders by infinite dilution inverse gas chromatography.

215 A sorption study on microporous materials by finite dilution inverse gas chromatography.

216 Characterization of surface properties of glass fibres by inverse gas chromatography.

227 Determination of Acid-Base Component of the Surface Energy by Inverse Gas Chromatography.

302 An Overview- Characterization of strong solid-vapour interactions by inverse gas chromatography.

303 An Overview of iGC-SEA - A new instrumental technique for characterizing the physico-chemical properties of polymers.

304 An Overview of Characterization of alumina and related surfaces by inverse gas chromatography.

PUBLICATIONS

"Surface characterization of standard cotton fibres and determination of adsorption isotherms of fragrances by IGC" (Surface and Interface Analysis, DOI: 10.1002/sia.5811)

"Effect of milling on particle shape and surface energy heterogeneity of needle-shaped crystals" (Pharm Res (2012) 29:2806-2816 DOI: 10.1007/s 11095-012-0842-1)

"Measuring surface roughness of pharmaceutical powders using vapor sorption methods" (AAPS PharmSciTech (2010) DOI: 10.1208/s12249-010-9571-0)

"Use of surface energy distributions by inverse gas chromatography to understand mechanofusion processing and functionality of lactose coated with magnesium sterate" (European Journal of Pharmaceutical Sciences 43 (2011) 325333)

"Influence of fines on the surface energy heterogeneity of lactose for pulmonary drug delivery" (International Journal of Pharmaceutics 388 (2010) 88-94)

"Determination of surface heterogeneity of D-Mannitol by sessile drop contact angle and finite concentration inverse gas chromatography" (International Journal of Pharmaceutics 387 (2010) 79-86)



"Inverse gas chromatographic method for measuring the dispersive surface energy distribution for particulates" (Langmuir 2008, 24, 9551-9557)

"Analysis of surface properties of cellulose ethers and drug release from their matrix tablets" (European Journal of Pharmaceutical Sciences 27 (2006) 375-383)

"Inverse Gas Chromatography of As-Received and Modified Carbon Nanotubes" (ACS- Langmuir Article, DOI: 10.1021/la900607s)

CASE STUDIES

603 Correlating drug-binder adhesive strengths measured by using Inverse Gas Chromatography with tablet performance.

605 The effect of primary particle surface energy on agglomeration rate in fluidised bed wet granualtion.

INSTRUMENT PLATFORM

Surface Measurement Systems continues as the world leader in inverse gas chromatographic instrumentation as illustrated by the iGC-SEA. Designed and manufactured in-house with extensive customer participation and feedback, the iGC-SEA meets and exceeds the need for a stable, reliable and easy to use surface energy heterogeneity and bulk property sorption solution.

iGC-SEA Hardware

Unique gas phase injection system with a 1:4000 injection volume ratio.

12 solvent reservoirs:

Easy accesss drawers. Temperature-controlled for vapor stability

Flame Ionization Detector (FID):

Adjustable gain

Fully integrated design

H₂ & Organic Vapor Leak Detector

2-sample column design:

Higher throughput

Sample column oven:

20°C to 150°C Fully integrated



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Minimized bench requirements:

System Dimensions: W:~490mm H:~650mm D:~564mm System Weight: 60kg

Optional:

Film/monolithic sample holder Background humidity control





iGC-SEA straight column design - simple sample packing and column loading



iGC-SEA Data Analysis Software

iGC-SEA data analysis software harnesses unsurpassed experimental flexibility, delivering extensive and user-friendly data analysis alongside one-click report generation. Cirrus Plus enables routine system operation and data analysis minimizing operator interaction time.



Standard features include:

- Isotherm determination/BET/Henry constant
- Surface energy analysis
- Surface heterogeneity mapping
- Competitive sorption measurement
- Acid-base chemistry analysis

Advanced features include:

- Glass Transition Temperature
- Hildebrand and Hansen solubility parameters
- Crosslink density
- · Work of adhesion/cohesion determination
- · Heats of adsorption/sorption measurement

IGC-SEA Specifications

1 Standard system information

The iGC-SEA is an advanced automated instrument specifically designed for carrying out inverse gas chromatography analysis, both surface and bulk, of particulate, film and fiber materials. The SEA system incorporates the following key design features:

1.1 Carrier Gas

The instrument can be operated with Helium or Nitrogen carrier gas. Please specify the end of the section 1, which carrier gas is requested.

1.2 Flame ionization detector

1.3 Probe vapor injection system

The automated probe vapor injection system includes twelve solvent reservoirs which allow the selection of twelve different probe vapors.



In addition, the following features come as standard:

- · Variable volume (1:4000) reproducible vapor pulse injections
- Integrated pneumatic valve actuation
- Inert solenoid valves, PEEK tubing and perfluoroelastomer o-rings, which support high repeatability, reliability and performance
- Thermoelectric heating and cooling of vapor reservoirs, which gives excellent temperature control (± 0.1°C) and allows for accurate probe concentrations
- Probe gas inlet port with inert solenoid valve allows injection of one non-condensable probe gas, such as methane for dead-time correction
- Three independent electronic mass flow controllers, each with a flow range of 0-50sccm, which provide accurate control and a wide range of solvent vapor injection sizes and surface coverages (Methane pulses are controlled by a flow valve)

1.4 Column oven

The SEA Column Oven is custom-designed with thermoelectric heating and cooling, allowing sub-ambient analysis temperatures.

- Range: 20°C-150°C
- Accuracy: 0.1°C
- Stability: 0.1°C

The oven is supplied with quick-fit column fittings designed to accommodate two 300mm x 6mm O.D. glass or stainless steel columns. They are specifically designed to eliminate leaks and allow quick sample changeovers.

1.5 Automated column packing device

Vibrational device for reproducible column packing.

1.6 Solvents calibrated as standard

The iGC-SEA is calibrated for use with a specific set of solvents as standard (listed below):

- 1-Propanol
- Heptane

Octane

Undecane

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HexaneNonane

- Acetonitrile
- Decane

Acetone

- Dichloromethane
- Ethanol
- Ethyl Acetate

• Water (for systems with the optional background humidity control)



1.6.1 Non-standard solvents

A solvent calibration wizard is included as standard the SEA control software (see below). T his allows the user to calibrate the iGC-SEA with additional solvents to those listed above.

Listed below are the additional solvents that can be calibrated using the wizard (other solvents not listed below can be agreed in consultation with SMS):

- 1,4-Dioxane
- 1-Butanol
- 1-Hexanol
- 1-Pentanol
- 2-Propanol
- Acetic Acid
- Benzene
- Benzyl-acetate
- Butyl Acetate

- CCI4
- Chloroform
- Cyclohexane
- Di-ethylamine
- Dodecane
- Formamide
- Heptadecane
- Hexadecane
- Hexanal

- Methanol
- Methyl salicylate
- Nitromethane
- Pentadecane
- Pentane
- Pyridine
- Tetradecane
- Toluene
- Tridecane

1.7 iGC-SEA data station

- Dell Desktop PC
- Microsoft Windows®7 or Windows®10 (requires 64 bit)
- Microsoft Office® 2016

Note: All SMS software must be used with English language versions of Microsoft Office® and Windows[®]. In addition, the regional settings in both Microsoft Office[®] and Windows[®] must be set to English. This applies to control computers supplied by SMS and/or the user, and to any computer using SMS analysis software.

Note: In general, all SEA software must be used with 64-bit versions of Microsoft Windows®.

1.8 Standard iGC-SEA software package

The standard SEA software package comprises the standard versions of both SEA Control software and SEA Analysis software.

Note: The standard SEA Control and SEA Analysis software programs cannot be purchased separately.

Note: The standard SEA software package is supplied as standard unless the advanced SEA software package is purchased. The advanced software package contains all the capabilities of the standard package with advanced features added. See Optional extras – software below.



1.8.1 SEA Control software

SEA Control provides complete system control, allowing the user to design, edit and run iGC experiments with ease.

Features include:

- Latest Windows®-based interface
- Solvent calibration wizard for calibrating solvents not included in the standard calibration (see above)
- Wide range of method variables, solvents, temperatures, surface coverage and flow rates
- · Real-time display of experiment progress
- Quick-set methods
- · Easy saving and restoring of methods
- Batch running of up to 10 methods
- Preconditioning methods
- User-programmable solvent database
- Solvent change wizard
- Also includes SEA calibration software
- Note: Supplied with 1 license as standard (3 users per PC per 1 license). Extra licenses are available at no additional cost. See below in Optional extras software.

1.8.2 SEA Analysis software

SEA Analysis software provides a powerful environment for rapid data processing and one-click report generation.

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Features include:

- Quick and easy data plotting
- Pulse analysis
 - o Dead-time correction
- Isotherm analysis:
 - o Peak max
 - o ECP tailing and fronting
 - o Henry constant



- BET specific surface area analysis
- Surface energy analysis:
 - o Dispersive component
 - o Specific (acid-base) component
 - o Total surface energy
- Surface heterogeneity mapping:
 - o Heterogeneity profile
 - o Heterogeneity distribution
- Acid-base free energy of desorption analysis
- Gutmann acid/base properties analysis
- · Determination of enthalpy of specific free energy
- · Display surface energy of multiple samples
- Merge experiments
- Method data storage and display including solvents used
- Note: Supplied with 1 license as standard (3 users per PC per 1 license). Extra licenses are available at no additional cost. See below in Optional extras software.

1.9 Installation/Warranty support

- On-site installation and qualification, including basic training and basic theory and applications
- 1 maintenance service visit is supplied within 6-12months of delivery.
- Emergency Breakdown visits and parts (excluding consumables) covered during the 12 month warranty period
- Additional advanced training available on request (chargeable see Optional extras advanced training section below)
- Note: min 1 month notice required in prior for the installation. Extra cost will be charged for installation within 4-2weeks and within 2weeks.

Requested Carrier Gas: Please indicate Helium or Nitrogen

2 Optional extras – instrumentation

2.1 Background humidity control

Addition of one extra mass flow controller (0-50sccm) to control the flow of water vapor from the existing vapor reservoirs. To be used for experiments which require controlled carrier gas humidification.



- Thermoelectric heating and cooling of the vapor reservoirs gives excellent temperature control (± 0.1°C) which allows accurate control of humidity
- Includes SEA Control Software functionality to set up additional humidity background

2.2 Film/Monolithic sample holder

A sample holder which accommodates a wide range of thin films, strips and wafers for surface and permeability studies.

The film cell is a modular unit which is plumbed into the SEA unit externally and is located adjacent to it on the laboratory bench.

- Flow-past mode for surface studies of the sample
- Flow-through mode for transmission and permeability studies
- Accommodates sample thicknesses of between 'zero' (i.e. thin films) to 12mm
- Accommodates rectangular and circular samples
- Wetted area of sample: 77cm² (11.9in²)

2.3 Large Column Design (10mm i.d.) (Part No. F-SEA-LRG)

A Large Column Accessory kit which allows the testing of oversize samples (up to 10mm in diameter). The Kit is comprised of the following:

- Large Column Fittings Set (x4) (P15MA099)
- O-Rings for the Column Fittings (x8) (C-MF-480)

3 Optional extras – software

3.1 Additional licences for standard software package

 he iGC SEA is supplied as standard with the latest versions of the SEA Control software and SEA Analysis software – 1 license for each (3 users per PC per 1 license). Extra licenses are available at no additional cost:

3.1.1 Additional SEA Control software licenses

Number of additional SEA Control Software licenses to be supplied:

3.2 Advanced SEA software package

The advanced SEA software package comprises the advanced versions of both SEA Control and SEA Analysis

Note: The advanced SEA Control and SEA Analysis software programs cannot be purchased separately.



Note: The advanced SEA software package contains all the capabilities of the standard SEA software package, with advanced features added. Therefore, if the advanced SEA software package is purchased, the standard package is not supplied.

3.2.1 Advanced SEA Control software

Advanced provides all of the capabilities listed above for the standard SEA Control software, plus these additional features:

- Injection Type option:
 - Target surface coverage
 - Target moles injected
- Variables option in method editor:
 - Change of temperature
 - Change of flow rate
- Note: Supplied with 1 license as standard (3 users per PC per 1 license). Extra licenses are available at no additional cost (see below).

3.2.1.1 Additional Advanced SEA Control software licences

Number of additional Advanced SEA Control Software licenses to be supplied:

3.2.2 Advanced SEA Analysis software

Advanced SEA Analysis provides all of the capabilities listed above for the standard SEA Anlaysis software, plus these additional features:

- Work of adhesion/cohesion analysis:
 - Work of cohesion
 - Work of adhesion (solid-solid)
 - Work of adhesion (solid-liquid)
- Heat of sorption analysis
- Glass transition temperature analysis
- Solubility parameter analysis:
 - Hildebrand (1D)
 - Hansen (3D)
- Degree of cross-linking analysis



Note: Supplied with 1 license as standard (3 users per PC per 1 license). Extra licenses are available at no additional cost (see below).

3.2.2.1 Additional Advanced SEA Analysis software licences

Number of additional Advanced SEA Analysis Software licenses to be supplied:

Optional extras – software validation documents

3.3 SEA Control and Advanced SEA control software

- Note: Validation documents are created cumulatively as new versions of the software are released. Therefore, the complete document set covering the entire version history of the software is required for a total validation of the current software.
- Note: Validation for the standard and advanced versions of the SEA Control software is covered in the same document set:
 - SEA Software 1.0.0 Software Acceptance Test Plan (Windows 7)
 - SEA Software 1.1.0 Software Acceptance Test Plan (Windows 7)
 - SEA Software 1.2.0 Software Acceptance Test Plan (Windows 7)
 - SEA Software 1.3.0 Software Acceptance Test Plan (Windows 7)

4 Optional extras – additional consumables

Note: The iGC SEA is supplied <u>as standard</u> with the following consumables in the quantities shown:

- 2mm ID (inner diameter) silanised glass columns (x20)
- 3mm ID silanised glass columns (x20)
- 4mm ID silanised glass columns (x10)
- 30ml solvent/water bottle (x15)
- Glass wool silane treated (x1 jar)



HPLC Servicing, Validation, Trainings and Preventive Maintenance :

HPLC Servicin	g:HPLC Servicing : We have team of service engineers who can attend to any make of HPLC promptly @the most
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Trainings	:We also take up preventive Maintenace to reduce downtime of HPLC's Trainings.
AMC's/CMC	:AMC's/CMC :We offer user training both in-House and at customer sites on HPLC principles, operations, trouble-
	shooting.
Validations	:Validations :We have protocols for carrying out periodic Validations as per GLP/GMP/USFDA norms.
• • •	

Instruments :Instruments :We offer instruments/Renting Services Modules like pumps, detector etc. on Rent.





About Analytical Technologies

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Optima Gas Chromatograph



3007

Semi Auto Bio Chemistry Analyzer



Chromatograph 2979 Plus



HEMA 2062 Hematology







Atomic Absorption

Spectrophotometer

URINOVA 2800

Urine Analyzer



Liquid Partical Counter



Total Organic Carbon 3800





Fully Automated CLIA

NOVA-2100 Chemistry Analyzer

PCR/Gradient PCR/ RTPCR

TOC Analyzer

Laser Particle Size Analyzer

Ion Chromatograph

Water purification system



Chromatograph



Micro Plate Reader/Washer







Regulatory compliances



Corporate Social Responsibility

Analytical Foundation is a nonprofit organization (NGO) found for the purpose of:



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2. Improving quality of life by offering YOGA Training courses, Work shops/Seminars etc.

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