Australian Government

Department of Education, Employment and Workplace Relations

MSL977006A Apply specialised knowledge of gas chromatography techniques to analysis

Release: 1

MSL977006A Apply specialised knowledge of gas chromatography techniques to analysis

Modification History

Not applicable.

Unit Descriptor

Unit descriptor	This unit of competency covers the ability to analyse samples using advanced gas chromatography (GC) instruments including GC-MS. The unit also includes establishing client needs for routine and non-routine samples, optimising enterprise procedures and instruments for specific samples, obtaining valid and reliable data and reporting test results. Personnel are required to recognise atypical test data/results and troubleshoot common analytical instrument and procedure problems and perform routine instrument maintenance.

Application of the Unit

Application of the unit	This unit of competency is applicable to experienced laboratory technical officers/technicians, laboratory supervisors and technical specialists who conduct instrumental analysis in laboratories providing consultancy, research and development and quality assurance services. These services may be provided for a wide range of industry sectors, such as forensic science (e.g. fire debris, toxicology and explosives), biomedical (e.g. isotopic labelling of metabolites), environmental monitoring (e.g. organics), food and beverage processing (e.g. aromatics, pesticide residues) and pharmaceuticals (e.g. active ingredient) testing. Industry representatives have provided case studies to illustrate the practical application of this unit of competency and to show its relevance in a workplace setting, at the end of this unit of competency under the section 'This competency in practice'.

Licensing/Regulatory Information

Not applicable.

Pre-Requisites

Prerequisite units		

Employability Skills Information

Employability skills	This unit contains employability skills.

Elements and Performance Criteria Pre-Content

Elements describe the essential outcomes of a unit of competency.	Performance criteria describe the performance needed to demonstrate achievement of the element. Where bold italicised text is used, further information is detailed in the required skills and knowledge section and the range statement. Assessment of performance is to be consistent with the evidence guide.

Elements and Performance Criteria

ELEMENT	PERFORMANCE CRITERIA
1. Determine sample characteristics and appropriate analytical methods	1.1. Interpret client request and/or identify sample characteristics that may affect sample preparation and/or analysis 1.2. Liaise with client or sample provider to review client needs, testing requirements and sample history. if necessary 1.3. Identify analytical standards, reference materials, test methods and enterprise procedures that may be applicable 1.4. Select the most appropriate standard test method that is consistent with testing requirements and instrument availability 1.5. If no standard method exists, adapt or modify a test method to suit the sample characteristics 1.6. If necessary, seek advice from supervisor about any proposed variations and document all approved changes to test methods 1.7. Schedule analysis using enterprise procedures
2. Prepare samples and standards	2.1. Log sample into instrument software 2.2. Obtain a representative analytical portion of the laboratory sample 2.3. Prepare sample in accordance with selected test method 2.4. Prepare validation checks and/or calibration standards for analytical portions 2.5. Use specialised procedures for ultra trace sample and standard preparation as required
3. Set up instrument and perform trial analysis	3.1. Configure the gas flow, injector, column, oven and detector sub-systems according to the selected test method 3.2. Perform pre-use, calibration and safety checks using enterprise procedures 3.3. Set instrumental parameters in accordance with those specified in selected test method 3.4. Check and optimise each instrument sub-system 3.5. Select an appropriate internal standard, if required 3.6. Conduct performance tests using (internal standards), standards and samples 3.7. Assess instrument performance in terms of response, resolution and run-time
4. Optimise instrument performance	4.1. Apply an understanding of analyte and column chemistry, temperature control and gas flow rate to determine strategies for enhancing separation and

ELEMENT	PERFORMANCE CRITERIA
	detection of required species 4.2. Adjust instrumental parameters in a logical and efficient sequence to optimise performance 4.3. When optimum separation is achieved, check that the detector and system software can correctly identify and quantify the required species

ELEMENT	PERFORMANCE CRITERIA
5. Perform analysis	5.1. Measure analyte (and internal standard) response for standards, validation checks and samples using optimised instrument settings 5.2. Conduct sufficient measurements to obtain reliable data 5.3. Regularly check for calibration drift and take appropriate action as necessary 5.4. Use system software to produce calibration graphs, chromatographs and/or mass spectra, confirm data quality and calculate uncertainties 5.5. Check that results are consistent with estimations and expectations 5.6. Analyse trends in data and/or results and report out of specification or atypical results promptly to appropriate personnel 5.7. Return instrument to standby or shutdown condition in accordance with enterprise procedures 5.8. Report results with the appropriate accuracy, precision, uncertainty and units
6. Perform routine maintenance and troubleshoot instruments	6.1. Regularly check the condition of gas cylinders, filters and traps and replace as necessary 6.2. Regularly check that the injector, column and detector sub-systems are clean/undamaged and replace consumable items as necessary 6.3. Change columns in accordance with manufacturer's instructions and ensure that the system is free of leaks and properly conditioned before re-use 6.4. Investigate possible causes for the absence of peaks and presence of ghost peaks, split peaks or distorted peak shapes and apply recommended remedial actions 6.5. Investigate possible causes for baseline instability and non-reproducible retention times and apply recommended remedial actions 6.6. Identify the need for repairs or servicing and determine whether local repair/maintenance is technically possible and economic 6.7. Arrange for repair or servicing from an accredited agent or other appropriate personnel in accordance with enterprise procedures
7. Maintain a safe work environment	7.1. Identify risks, hazards, safety equipment and control measures associated with sample handling/preparation and test method 7.2. Use personal protective equipment and safety procedures specified for test method and materials to

ELEMENT	PERFORMANCE CRITERIA
	be tested 7.3. Minimise the generation of wastes and environmental impacts 7.4. Ensure the safe collection/disposal of laboratory wastes 7.5. Clean, care for and store equipment and consumables in accordance with enterprise procedures
8. Maintain laboratory records	8.1. Enter approved data and results into laboratory information management system (LIMS) 8.2. Maintain logs of instrument calibration checks, use and maintenance in accordance with enterprise procedures 8.3. Maintain security, integrity and traceability of samples, results and documentation 8.4. Communicate results to appropriate personnel in accordance with enterprise procedures

Required Skills and Knowledge

REQUIRED SKILLS AND KNOWLEDGE

This section describes the skills and knowledge required for this unit.

Required skills

Required skills include:

- establishing client needs for routine and non-routine samples
- interpreting client requests, test methods and procedures accurately
- selecting, adapting and modifying standard test methods for unknown samples (including consideration of suitable stationary phase, support, solvent, temperature, flow rate, column type, column length and detection)
- preparing samples and standards, optimising procedures and equipment to suit sample/test requirements
- setting up, starting up and shutting down equipment
- checking the calibration/qualification status of equipment
- selecting, configuring, checking and optimising instrument sub-systems
- performing routine instrument maintenance and replacement of consumables
- obtaining valid and reliable data
- calculating analyte concentrations with appropriate accuracy, precision, uncertainty and units
- recognising atypical data/results and troubleshooting common analytical procedure and equipment problems
- recording and reporting data/results using enterprise procedures
- maintaining security, integrity and traceability of samples and documentation
- assessing risks, applying specified control measures and working safely
- minimising waste and ensuring safe collection and disposal of waste materials
- applying relevantprinciples of good laboratory practice (GLP) procedures
- maintaining technical knowledge by accessing journals, technical updates, suppliers' product notes and test methods

Required knowledge

Required knowledge includes:

- scope of samples that can be tested using gas chromatography GC techniques
- sample preparation procedures including specialised techniques such as:
  - handling unstable/hazardous chemicals and samples, and fragile/labile biological material
  - liquid-liquid extraction, solid-phase micro-extraction, derivatisation and diluting/concentrating
- GC principles for separation of analytes such as:
  - separation modes, chemical structure of stationary phase and its interaction with

REQUIRED SKILLS AND KNOWLEDGE

the analyte
  - order of elution based on analyte volatility and polarity
  - predicting effect of condition changes
- chromatography concepts and calculations involving:
  - retention times, peak widths, peak asymmetry, capacity factor k' and resolution
  - column selectivity, column efficiency (plates/m), optimum flow rate, minimum theoretical plate height, Van Deemter and related equations
  - limit of detection, limit of quantitation and their application to quality control procedures
- types of gases and requirements for purity and pre-treatment of gases, such as drying, use of oxygen/moisture/hydrocarbon traps and filters
- operation, construction, typical applications, troubleshooting and routine maintenance of injectorssuch as:
  - head space sampling
  - hot direct
  - split/splitless
  - solid phase micro-extraction (SPME)
  - program temperature vaporisation (PTV)
  - gas desorption
  - purge and trap
  - on column
  - choice, use and maintenance of syringes
- operation, construction, selectivity, sensitivity, linear range, typical applications, troubleshooting and routine maintenance of GC systems, including details such as:
  - packed columns
  - capillary columns including megabore
  - column conditioning and replacement
  - gas inlets, septum, septum purge, injector insert, heater block, tubing materials, column connection, split valve and vent and compatibility of connectors
  - checking for leaks and system conditioning
- operation, construction, selectivity, sensitivity, linear range, typical applications, troubleshooting and routine maintenance of GC detectors such as:
  - thermal conductivity detectors (TCD)
  - flame ionisation detectors (FID)
  - electron capture detector (ECD)
  - flame photometric detector (FPD)
  - mass spectrometry (GC-MS) (GC-MS-MS) using full scan or selective ion monitoring (SIM)
- routine quality control procedures such as use of manual/computer calibration charts and/or standards

REQUIRED SKILLS AND KNOWLEDGE

- computer control software for operating and optimising instrument (peak detection and integration, drift parameters, baseline correction and instrument/integrator zero)
- procedures for optimising instrument performance such as:
  - investigation of elution order
  - optimising separation by changing injection technique, sample size and sample preparation
  - effects on instrumental outputs and analytical results by fine tuning injection temperature, gas flow rate, column pressures and changing column type and detector type
  - use of temperature, flow gradient and pressure programming
- steps in identifying and quantifying analytes, including relative retention data, peak area normalisation and response factors, internal standards and spiking
- calculation steps (e.g. dilution steps) to give results in appropriate units and precision
- troubleshooting and maintenance procedures recommended by instrument manufacturer
- enterprise and/or legal traceability requirements
- relevant health, safety and environment requirements

Evidence Guide

EVIDENCE GUIDE	
The Evidence Guide provides advice on assessment and must be read in conjunction with the performance criteria, required skills and knowledge, range statement and the Assessment Guidelines for the Training Package.	
Overview of assessment	
Critical aspects for assessment and evidence required to demonstrate competency in this unit	Assessors should ensure that candidates can: • interpret client requests, test methods and procedures accurately • select, operate and maintain a variety of GC injectors, columns and detectors • install injectors and columns • safely set up, start up and shut down instrument using enterprise procedures • prepare samples and calibration standards in accordance with test method • check calibration/qualification status of equipment • optimise instrument sub-systems and procedures and equipment to suit sample/test requirements • operate equipment to obtain valid and reliable data • use software to identify analytes and calculate concentrations with appropriate accuracy, precision and units • recognise atypical data/results • troubleshoot common analytical procedure and equipment problems • record and report data/results using enterprise procedures • maintain security, integrity and traceability of samples and documentation • follow OHS procedures and principles of GLP.
Context of and specific resources for assessment	This unit of competency is to be assessed in the workplace or simulated workplace environment. This unit of competency may be assessed with: • *MSL976003A Evaluate and select appropriate test methods and procedures* • *MSL977003A Contribute to the validation of test methods* • *MSL977004A Develop or adapt analyses and procedures*.

EVIDENCE GUIDE	
	Resources may include: • laboratory with specialised analytical instruments • laboratory reagents and equipment • SOPs and test methods.
Method of assessment	The following assessment methods are suggested: • review of test data/results/calibration graphs obtained by the candidate over time to ensure accuracy, validity, precision and timeliness of results • inspection of results and technical records (e.g. maintenance schedules and quality controllogbooks) completed by the candidate • observation of candidate using GC instruments to measure analytes • feedback from clients, peers and supervisors • oral or written questioning of relevant gas chromatography concepts, chemical principles underpinning sample preparation and separation of species,instrument design and optimisation, analytical techniques and enterprise procedures. In all cases, practical assessment should be supported by questions to assess underpinning knowledge and those aspects of competency which are difficult to assess directly. Where applicable, reasonable adjustment must be made to work environments and training situations to accommodate ethnicity, age, gender, demographics and disability. Access must be provided to appropriate learning and/or assessment support when required. The language, literacy and numeracy demands of assessment should not be greater than those required to undertake the unit of competency in a work like environment.
This competency in practice	Industry representatives have provided the case studies below to illustrate the practical application of this unit of competency and to show its relevance in a workplace setting. **Environmental testing** A laboratory routinely analyses samples of foodstuffs for pesticide residues. Traditionally, they have used GC combined with ECD and FID detectors to quantify one or two classes at a time. Recently, the laboratory has

EVIDENCE GUIDE	
	commissioned a new GC-MS instrument that is capable of quantifying low level pesticides using the SIM mode while simultaneously performing quantification of higher concentrations using full-scan (SIFI single ion and full ion) acquisition. The technician sets up for a typical run of samples. He/she uses the programmable split/splitless injector to provide 1µL samples. The injection port temperature is set at 275°C (isothermal) and the capillary column uses a phase specifically designed for separation of pesticides. The helium carrier gas is programmed with a constant velocity of 30 cm/s. The oven temperature program is initially set to 80°C with no hold and ramped to 290°C at 20°C/min with a hold of 4.5 min. The total oven program is 15 min, with an injection to injection time of less than 20 min. The MS method contains multiple SIM functions overlapped by a *m/z 40* to *m/z 450* full scan function and the mass spectrometer transfer line and ion source are heated to 275°C. **Pharmaceutical testing** A laboratory technician working for a major cosmetics company is given a retail sample of a competitor's perfume and asked to determine its composition. He/she has to start somewhere and decides to use one of the laboratory's GC-MS instruments to simplify the identification process and to avoid the need for multiple spikes. The GC-MS is already set up to measure the company's own perfume and so the technician uses standard instrumental parameters as a starting point. However, he/she finds that many peaks are not resolved and some take a long time to elute. The technician realises that he/she should either modify the temperature and/or pressure programming or change columns. Sensibly, the technician chooses to modify the easiest parameter (i.e. temperature) before changing the column. This overcomes the problem of slow elution but does not resolve all peaks. He/she then varies the pressure programming which provides better resolution but does not completely separate all peaks. The technician then uses the instrument's spectral matching software to identify as many peaks as possible, including the ones that overlap.

Range Statement

RANGE STATEMENT	
The range statement relates to the unit of competency as a whole. It allows for different work environments and situations that may affect performance. Bold italicised wording, if used in the performance criteria, is detailed below. Essential operating conditions that may be present with training and assessment (depending on the work situation, needs of the candidate, accessibility of the item, and local industry and regional contexts) may also be included.	
Codes of practice	Where reference is made to industry codes of practice, and/or Australian/international standards, it is expected the latest version will be used

Standards, **codes**, **procedures and/or enterprise requirements**	Standards, codes, procedures and/or enterprise requirements may include:
	• Australian and international standards, such as:
	  • AS ISO 17025-2005 General requirements for the competence of testing and calibration laboratories
	  • AS/NZS 2243 Set:2006 Safety in laboratories set
	  • AS/NZS ISO 9000 Set:2008 Quality management systems set
	  • AS 2830.1 Good laboratory practice - Chemical analysis
	  • ASTM E355 Standard practice for gas chromatography terms and relationships
	  • ASTM E516 Standard practice for testing thermal conductivity detectors used in gas chromatography
	  • ISO 6889 Natural gas rapid analysis by gas chromatography
	  • BS 5443 Recommendations for standard layout for methods of chemical analysis by gas chromatography
	  • ISO/IEC Guide 98-3:2008 Uncertainty of measurement - Part 3 Guide to the expression of uncertainty in measurement (GUM)
	• Eurachem/CITAC Guide CG4 Quantifying uncertainty in analytical measurement
	• National Association of Testing Authorities (NATA) supplementary requirements for the

RANGE STATEMENT	
	field of testing • Australian code of good manufacturing practice (GMP) • principles of good laboratory practice (GLP) • material safety data sheets (MSDS) • national measurement regulations and guidelines • enterprise procedures, standard operating procedures (SOPs) and operating manuals • quality manuals, equipment and procedure manuals • equipment startup, operation and shutdown procedures • calibration and maintenance schedules • cleaning, hygiene and personal hygiene requirements • data quality procedures • enterprise recording and reporting procedures • material, production and product specifications • production and laboratory schedules • quality system and continued improvement processes • safety requirements for equipment, materials or products • sampling procedures (labelling, preparation, storage, transport and disposal) • schematics, work flows and laboratory layouts • statutory and enterprise occupational health and safety (OHS) requirements • stock records and inventory • test procedures (validated and authorised) • waste minimisation, containment, processing and disposal procedures

RANGE STATEMENT	

GC instruments and techniques	GC instruments and techniques may include:
	• sample introduction, such as:
	  • micro-syringe, split/splitless
	  • head space analysis
	  • thermal desorption
	  • purge and trap
	  • on column injection
	• packed columns, capillary columns, support coated open tubular (SCOT), wall coated (WCOT) and porous layer (PLOT) types
	• ovens including temperature stability and temperature programming capability
	• detectors, such as:
	  • thermal conductivity (TCD)
	  • flame ionisation (FID)
	  • electron capture (ECD
	  • atomic emission (AED)
	  • flame photometric (FPD)
	  • photo ionisation (PID)
	  • fourier transform infra red (FTIR)
	• 'hyphenated' mass spectrometry (GC-MS) (GC-MS-MS)
	  • ionisation - chemical, plasma/glow discharge, electron impact, electro spray, electric field and laser ionisation
	  • design layout - ion cyclotron resonance fourier transform, ion trap, magnetic-sector, quadrupole and time of flight
	  • ion detectors - channeltron, Daly, electron multiplier tube and micro-channel plate
	  • full scan or selective ion monitoring (SIM)
	• replaceable items, such as gas inlets, septum, septum purge, injector insert, heater block, tubing materials, column connectors, split valve and vent, and lamps
	• data systems such as recorders, electronic integrators, and software packages for peak detection and integration

Testing that uses GC instruments	Testing that uses GC instruments may include: • forensic testing, such as toxicology of

RANGE STATEMENT	
	biological samples, identification of fire and explosive residues • medical testing, such as using isotopic labelling of metabolic compounds • testing of athletes for performance enhancing drugs • environmental cleanup and monitoring of pollution in air, water or soil (e.g. organochloride pesticides) • control of starting materials, in-process materials and final products in a wide range of industry sectors (pharmaceuticals, biotechnology, petroleum and manufacturing) • multi-analyte determination • testing for contaminants in food and beverages • analysis of flavour and fragrance

RANGE STATEMENT	
Sample characteristics that may affect analysis	Sample characteristics that may affect analysis may include: • presence of non-volatiles, such as carbohydrates
Sample preparation	Sample preparation may include: • conversion to small volumes (1ÂµL) • derivatisation • identification of any hazards associated with the samples and/or analytical chemicals • grinding, dissolving, extraction, filtration, refluxing, centrifuging, evaporation, washing and drying • solid-phase micro-extraction • determination of, and if appropriate, removal of any contaminants or impurities or interfering substances • ultra-trace procedures requiring high purity solvents, clean rooms, ultra clean glassware and specialised glassware

Instrumental parameters	Instrumental parameters may include
	• GC parameters:
	  • injection mode (direct, split/splitless, on column)
	  • manual/auto sample (injector volume, speed and time)
	  • pre- and post-sample washes
	  • gas flow controls
	  • isothermal versus temperature programming
	  • isobaric versus pressure programming
	• detector/source parameters and single/split system
	• MS parameters:
	  • vacuum pressures and gas flows
	  • nebuliser gas flow
	  • ionisation control
	  • interface cone alignment
	  • ion lens voltage
	  • mass analyser control

RANGE STATEMENT	
	• solvent delay • scan, mass start/end, scan time and inter-scan delay • selective ion monitoring (SIM)

RANGE STATEMENT	
Common analytical procedure and equipment problems	Common analytical procedure and equipment problems may include: • system leaks • syringe blockage or incorrect type and inappropriate septum • column overloading • contamination of sample, gas or solvents, lines or other system elements and out gassing of traps • overcoming problems with interfering substances by using SIM • lack of suitable reference standards • poor separation due to inappropriate selection of column or operating parameters (temperature and flow) • poor sensitivity • absence of peaks and presence of ghost peaks, split peaks or distorted peak shapes and broad solvent peaks • baseline instability and non-reproducible retention times
Hazards	Hazards may include: • electric shock • biohazards, such as microbiological organisms and agents associated with soil, air, water, blood and blood products, and human or animal tissue and fluids • corrosive chemicals • sharps and broken glassware • hot surfaces • flammable liquids and gases • fluids under pressure sources of ignition • disturbance or interruption of services
Addressing hazards	Addressing hazards may include: • use of MSDS • accurate labelling of samples, reagents, aliquoted samples and hazardous materials • personal protective equipment such as gloves, safety glasses and coveralls • use of fumehoods, direct extraction of vapours

RANGE STATEMENT	
	and gases • use of appropriate equipment such as biohazard containers, laminar flow cabinets, Class I, II and III biohazard cabinets • handling and storage of all hazardous materials and equipment in accordance with labelling, MSDS and manufacturer's instructions
Occupational health and safety (OHS) and environmental management requirements	OHS and environmental management requirements: • all operations must comply with enterprise OHS and environmental management requirements, which may be imposed through state/territory or federal legislation - these requirements must not be compromised at any time • all operations assume the potentially hazardous nature of samples and require standard precautions to be applied • where relevant, users should access and apply current industry understanding of infection control issued by the National Health and Medical Research Council (NHMRC) and State and Territory Departments of Health

Unit Sector(s)

Unit sector	Testing

Competency field

Competency field	

Co-requisite units

Co-requisite units		