Australian Government

Department of Education, Employment and Workplace Relations

MSL977010A Apply advanced infra red spectroscopic techniques to analysis

Revision Number: 1

MSL977010A Apply advanced infra red spectroscopic techniques to analysis

Modification History

Not applicable.

Unit Descriptor

Unit descriptor	This unit of competency covers the ability to analyse samples using specialist techniques and modern accessories that extend the capability of fourier transform infra red (IR or FTIR) spectrometers. The unit 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 biomedical (e.g. bacterial screening), forensic science (e.g. fingermark residues, paints and fibres), environmental monitoring (pollutants in air, soil and water), industrial analysis (textile fibres, polymers, lubricant condition, defect and surface contamination), pharmaceutical products (drug analysis) and food (e.g. nutrition supplement analysis). 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 perform presumptive tests to identify sample characteristics and determine the most appropriate IR spectroscopic technique 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
3. Set up instrument and perform trial analysis	3.1. Configure the appropriate instrument accessories to perform the selected test method 3.2. Perform other 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. Conduct performance tests using standards and samples 3.6. Assess instrument performance in terms of response, resolution and number of scans required
4. Optimise instrument performance	4.1. Adjust instrumental parameters in a logical and efficient sequence to optimise performance 4.2. When optimisation is achieved, check that the detector and system software can correctly identify and quantify the required species
5. Perform analysis	5.1. Measure analyte response for standards, validation

ELEMENT	PERFORMANCE CRITERIA
	checks and samples using optimised instrument settings 5.2. Conduct sufficient measurements to obtain reliable data 5.3. Use system software to produce calibration graphs, spectra, confirm data quality and calculate uncertainties 5.4. Check that results are consistent with estimations and expectations 5.5. Analyse trends in data and/or results and report out of specification or atypical results promptly to appropriate personnel 5.6. Return instrument to standby or shutdown condition in accordance with enterprise procedures 5.7. Report results with the appropriate accuracy, precision, uncertainty and units
6. Perform routine maintenance and troubleshoot instruments	6.1. Regularly check the spectral and photometric performance of the instrument 6.2. Regularly clean sample cell and/or reflectance crystal faces and replace consumables as appropriate 6.3. Confirm optical matching for cell pairs as necessary 6.4. Identify the need for repairs or servicing and determine whether local repair/maintenance is technically possible and economic 6.5. 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 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

ELEMENT	PERFORMANCE CRITERIA
	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 appropriate IR techniques and installing instrument accessories
- selecting, adapting and modifying standard test methods for unknown samples
- 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, ensuring safe collection and disposal
- applying relevant principles 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:

- criteria for determining which IR technique (e.g. cell and accessory) is best suited to which type of sample (e.g. gas, liquid, bulk solid, fibre, film, small quantity)
- sample preparation procedures including specialised techniques such as:
  - handling unstable/hazardous chemicals and samples, fragile/labile biological material and hygroscopic samples
  - filtration or centrifugation to remove particulates
  - prevention of personal contamination of samples by exposure to analyst
- cleaning and/or handling of optical elements:
  - cleaning techniques and handling of transmission cells and flow cells

REQUIRED SKILLS AND KNOWLEDGE

- cleaning of reflectance crystals and solid/liquid standards
- alignment techniques for source mirrors and accessories

- IR spectroscopic terms and concepts such as:
  - absorption, absorbance, transmittance, diffuse and specular reflectance and attenuated total reflectance
  - fourier transform of interferograms to produce spectra
  - fast fourier transform (FFT)
  - Fourier Transform advantages (Jacquinot, Felgett, Connes)
  - electronic, vibronic, vibrational and rotational transitions during absorption and relationship to chemical properties
  - concepts such as Beer's Law, Bouguer or Lambert's Law and molar absorptivity
  - deviations from Beers Law, polychromatic radiation and chemical reactions
  - spectral resolution, spectral bandwidth and linear dispersion
  - limit of detection, limit of quantitation and their application to quality control procedures
  - derivative spectra
  - multi-component analysis
  - analysis of reaction kinetics
- calculations and data processing involving:
  - baseline correction and spectral smoothing
  - Kubelka-Munk conversion (to linearise diffuse reflectance data)
  - Kramer-Kronig transformation (to remove refraction effects)
  - Concentration and dilution
  - spectral matching
  - spectral subtraction and spectral deconvolution
  - first and higher derivatives of spectra
  - multi-component quantitation techniques such as Classic Least Squares (CLS), Inverse Least Squares (ILS), Partial Least Squares (PLS), Principal Component Regression (PCR)
- operation, construction, selectivity, typical applications, troubleshooting and routine maintenance of IR systems including details such as:
  - scan settings (source/detector/beam-splitter combination, number of scans, scan range, scan mode, aperture, resolution, apodisation, zero filling, optical alignment, sensitivity, threshold, optical path difference and velocity)
  - sources (e.g.tungsten-halogen, ceramic, mercury arc, glow bars and Nernst glowers)
  - interferometer components (e.g. beam splitters (KBr, CsI, Mylar and metal mesh) and mirrors
  - sample transmittance (KBr, ZnSe and CsI cells), sample reflectance mounts and troughs, fibre optic probes and microscope attachments

REQUIRED SKILLS AND KNOWLEDGE

- mid IR detectors (e.g. HgCdTe or MCT, DLaTGS), far IR detectors (e.g. DLaTGS, liquid helium cooled Si bolometers)
- IR sensitive charge coupled array devices and focal plane array detectors for imaging

- operation, construction, typical applications, troubleshooting and routine maintenance of IR accessories such as:
  - attenuated total reflectance, single and multi-reflection unit and grazing angle
  - specular and diffuse reflectance units
  - single point microscope and spectrochemical imaging microscope
  - photoacoustic spectroscopy (PAS)
  - thermogravimetric analysers (TGA-IR)
  - polariser
- calibration procedures for:
  - accuracy of wavelength using transmission and reflectance standards, such as polystyrene, germanium and rare earth oxide glasses such as holmium oxide
  - photometric accuracy using for example, polystyrene or polyethylene terephthalate and potassium dichromate
  - zero absorbance baseline flatness
- sources of spectral interferences such as:
  - water and carbon dioxide
  - strongly absorbing matrix components
- computer control software for operating and optimising instrument
- procedures for optimising instrument performance such as:
  - alignment of sub-systems (e.g. source mirror and beam splitter) and accessories
  - adjustment signal to noise ratio to obtain satisfactory spectral resolution
- use of manual/computer calibration charts and/or standards to identify and quantify analytes such as:
  - external calibration
  - multi-component analysis
  - semi-quantitative analysis
  - library searching for spectral matching
  - derivative spectrum analysis
- calculation 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 • replace standard IR/FTIR instrument consumables such as lamps, desiccants and purge gases • install IR/FTIR accessories, such as sample cells/probes, microscope and reflectance units • 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*

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	procedures. 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 control logbooks) completed by the candidate • observation of candidate using IR/FTIR instruments to measure analytes • feedback from clients, peers and supervisors • oral or written questioning of relevant IR/FTIR spectroscopy 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. **Forensic science** A forensic science team is examining some motor vehicle paint samples taken from a crime scene. After a careful microscopic examination of the topcoat and

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	undercoat layers, one technician narrows the sample's origin down to a small range of possible vehicle makes, models and years of manufacture. To aid identification, another technician prepares a small paint chip for examination in the laboratory's FTIR spectrometer. After referring to the recommended analytical methods, he/she decides to use a transmittance technique because all the sampling wavelengths are subjected to the same path lengths and most of the reference data for paints, binders, pigments and additives consist of transmittance spectra. The technician cuts a cross-section sample with a microtome to expose an edge of the multi-layered sample and then mounts it using the microscope accessory. He/she adjusts the microscope aperture to minimise stray light and diffraction effects before obtaining spectra for each layer. After comparing his/her results with the spectral library data for binders, pigments and additives used by the manufacturers of interest, the origin of the paint is identified. Some weeks later, confirmation is obtained when samples taken from a suspect's vehicle match the physical and chemical features and spectra determined from the crime scene samples and diffuse reflectance measurements on the two sets of samples show identical weathering and surface contamination.

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 E168 - 06 Standard practices for general techniques of infra red quantitative analysis
	  • ASTM E334 - 01(2007) Standard practice for general techniques of infra red microanalysis
	  • ASTM E573 - 01(2007) Standard practices for internal reflection spectroscopy
	  • ASTM E2224 - 02 Standard guide for forensic analysis of fibers by infra red spectroscopy
	  • 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 (MSDSs) • 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	
IR/FTIR instruments and techniques	IR/FTIR instruments and techniques may include: • grating or interferometer as the dispersive element • gas sampling cells • liquid autosamplers and flow cells • beam condensers for micro-samples • reflection/transmission holders for FTIR microscopes • specialised infra red windows for FTIR microscopes • attenuated total reflectance ATR units (e.g. variable pressure, horizontal HATR trough plates, multiple internal reflectance MIR, single reflection ATR units and universal UATR units) • fixed/variable angle specular reflectance units • polarisers • single point microscopes and array-based imagers • discrete analysers (e.g. oil and SO_2) • data systems such as recorders, electronic integrators, and software packages for peak detection and spectra manipulation • spectral library matching
Testing that uses IR/FTIR spectroscopy	Testing that uses IR/FTIR spectroscopy may include: • medical (pathology) testing (e.g. bacterial screening) • forensic testing to establish analyte 'fingerprint' and possible source of scene of crime samples (e.g. paint layers by specular reflectance, fingermark residues by ATR and fibres by polarisable ATR) • environmental monitoring of pollution in air, water or soil (e.g. multi-organic components in industrial emissions) • control of starting materials, in-process materials and final products in a wide range of industry sectors (e.g. surface defects in polymers, semiconductor contamination and quantitation of biodiesel components) • materials testing (e.g. lubricant condition)

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	• food testing (e.g. moisture and protein determination in grain) • pharmaceuticals (e.g. drug testing of athletes) • geological testing (e.g. oil inclusions in rocks by grazing angle ATR)

RANGE STATEMENT	
Presumptive tests	Presumptive tests may include: • source of sample • type and quantity of sample • assessing suitability of sample and specified preparation for spectroscopic technique (e.g. pH and aqueous content)
Sample and standard preparation	Sample and standard preparation may include: • identification of any hazards associated with the samples and/or analytical chemicals • grinding, mulling and preparation of disks • dissolving, extraction, centrifuging, evaporation, washing and drying • determination of, and if appropriate, removal of any contaminants or impurities or interfering substances
Pre-use, **calibration and safety checks**	Pre-use, calibration and safety checks may include: • cleanliness of cells and dip/or probes • condition of desiccant packs and purge gas flows • detector coolant • optical alignment (e.g. beam splitter alignment, mirrors and accessories) • cell positioning and cell matching • checking integrity and alignment of accessories • wavelength accuracy using polystyrene • amplifier gain adjustment, signal to noise ratio • stray light levels • 100% transmittance baseline flatness • spectral bandwidth (sharpness of peak, peak intensity and resolution of adjacent peaks)
Instrumental parameters	Instrumental parameters may include: • spectral lamp selection • measurement mode (i.e. transmittance, reflectance) • wavelength range, start/finish • spectral bandwidth (slit width and signal to noise ratio) • scan speed/fixed wavelength • number of scans

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	• temperature control for kinetic studies

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Common analytical procedure problems and remedies	Common analytical procedure problems and remedies may include: • cell cleanliness • mismatched optical cell pairs and incorrect cell path length • alignment of external attachments • full scale absorption and too much sample • non-homogeneity of samples • air gap between solid sample and reflectance crystal
Common equipment problems	Common equipment problems may include: • system leaks • adjustment of optical elements such as mirrors and lamps • alignment of external accessories • cleanliness/optical matching of cells and cell window degradation • cell blockages • contamination of reflectance crystal
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 • flammable liquids and gases • fluids under pressure and 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 and gases • use of appropriate equipment such as biohazard

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	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		