Australian Government

Department of Education, Employment and Workplace Relations

MSL977009A Apply advanced ultraviolet, visible and near infra red spectroscopic techniques to analysis

Release: 1

MSL977009A Apply advanced ultraviolet, visible and near 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 ultraviolet, visible and near infra red (UV/VIS/NIR) 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 and forensic science (e.g. identification of paints), environmental monitoring (pollutants in air, soil and water), agricultural and mineral testing, industrial analysis (paints, inks, dyes, paper and petrochemical products), pharmaceutical products (active ingredients) and food and beverage (colour, rapid protein and moisture 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 UV/VIS/NIR 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. Measure analyte response for standards, validation checks and samples 3.6. Assess instrument performance in terms of response and resolution
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

ELEMENT	PERFORMANCE CRITERIA
5. Perform analysis	5.1. Measure analyte response for standards, validation 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 check and replace sample/waste tubing on peristaltic pump lines as appropriate 6.3. Replace lamps and 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)

ELEMENT	PERFORMANCE CRITERIA
	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 appropriate UV/VIS/NIR techniques and installing instrument accessories
- selecting, adapting and modifying standard test methods for unknown samples
- preparing samples and standards and 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 UV/VIS/NIR technique (e.g. cell, accessory) is best suited to which type of sample (e.g. gas, liquid, bulk solid, fibre, film and small quantity)
- sample preparation procedures including specialised techniques such as:
  - handling unstable/hazardous chemicals and samples, fragile/labile biological material
  - dilution without causing chromaphoric shifts
  - filtration or centrifugation to remove particulates
  - prevention of personal contamination of samples by exposure to analyst

REQUIRED SKILLS AND KNOWLEDGE

- cleaning and/or handling of optical elements:
  - cleaning techniques and handling of cells and flow cells
  - cleaning of reflectance standards and liquid standards
  - alignment techniques for source mirrors and accessories
- UV/VIS/NIR spectroscopic terms and concepts such as:
  - excitation, absorption, absorbance, transmittance, reflectance and fluorescence
  - electronic transitions during absorption/fluorescence and relationship to chemical properties, interference and quenching
  - concepts such as Beer's Law, Bouguer or Lambert's Law and molar absorptivity
  - deviations from Beers Law, polychromatic radiation and chemical reactions
  - chromaphores and auxochhromes
  - conjugation effects in organic molecules
  - pH effects in ionised samples
  - solvent effects
  - 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:
  - concentration and dilution
  - spectral matching
  - first and higher derivatives of spectra
  - multi-component quantitation
- operation, construction, selectivity, typical applications, troubleshooting and routine maintenance of UV/VIS/NIR systems including details such as:
  - sources, such as deuterium and tungsten filament
  - monochromators, such as prisms, gratings and filters
  - sample holders, such as quartz, glass and plastic cuvettes, cells and fibre optic dip probes
  - detectors, such as photomultipliers, photodiode, diode arrays, charge coupled devices, and semiconductor devices for NIR (e.g. PbS and InGaAs)
  - accessories that extend capabilities, such as autosamplers, thermostat controlled cell holders, multi-cell transports and diffuse and specular reflectance units
- calibration procedures for:
  - accuracy of wavelength and absorbance using hydrogen/deuterium, holmium oxide glass filters and holmium perchlorate solution
  - stray radiation using recommended liquids for each spectral range

REQUIRED SKILLS AND KNOWLEDGE

- photometric accuracy using potassium dichromate solution
- zero absorbance baseline flatness
- sources of spectral interferences such as:
  - particulates
  - fluorescence from heavy metals
  - interfering chromaphores
  - masking by chelating agents
- computer control software for operating and optimising instrument
- procedures for optimising instrument performance such as:
  - alignment of sub-systems (e.g. source mirror) and accessories
  - adjustment of slit and signal-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 with or without internal standardisation
  - 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 UV/VIS/NIR instrument consumables, such as lamps and purge gases • install UV/VIS/NIRaccessories such as optical fibre probes, microscopes 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 UV/VIS/NIR instruments to measure analytes • feedback from clients, peers and supervisors • oral or written questioning of relevant UV/VIS/NIR 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 workplacesetting. **Forensic science** A forensic science team is examining some fibre samples taken from a crime scene and a suspect. After a careful microscopic examination of the two sets of samples, one

EVIDENCE GUIDE	
	technician determines that both contain blue polyester fibres. Because of the problems associated with extracting dyes from fibres, the team decides to first use a non-destructive analytical technique. This involves obtaining the UV/VIS absorption spectra of single fibres using micro-spectrophotometry. Each fibre is mounted between a quartz slide and cover slip and examined in transmission. The laboratory's instrument enables the technician to obtain a several spectra from a 2µ x 2µ spot for a wavelength range of 190 nm to 900 nm. The repeat spectra for each fibre are first compared for internal consistency by overlaying the spectra in pairs. The spectra for each fibre are then compared with those obtained for the other fibres and with the reference spectra for a large number of known blue polyester fibres in an international spectral library. To provide enhanced discrimination, derivative spectra are used to reveal subtle inflections and changes of gradient in the broad convoluted absorption peaks. However, because of the spatial variations in dye uptake within any one fibre, these derivative spectra could not be relied on to provide conclusive identification. The team then attempted to measure each fibre's birefringence using a polarising microscope and employed a variety of FTIR transmission and reflectance techniques to shed more light on the chemical properties of the two sets of fibres. **Food processing** A laboratory technician's manager asks him/her to determine the concentration of Vitamin C in a sample of clear apple juice. The technician is very familiar with the analytical methods used for determining Vitamin C in bulk pharmaceutical preparations but soon realises that these methods do not give a reliable result in this case. He/she considers alternative spectroscopic methods. The technician is aware that the laboratory has a modern UV-VIS spectrometer with an advanced spectrum calculator capable of producing up to 4th order derivative spectra. After finding a suitable method, the technician determines the Vitamin C content by using the instrument's derivative capability to produce sharp peaks and overcome the broad UV absorption bands usually obtained with this kind of sample.

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
	  • AS3753-2001 Recommended practice for chemical analysis by ultraviolet/visible spectrophotometry
	  • ISO 22891:2007 Paper - Determination of transmittance by diffuse reflectance measurement
	  • 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 field of testing
	• Australian code of good manufacturing practice (GMP)
	• principles of good laboratory practice (GLP)

RANGE STATEMENT	
	• 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	
UV/VIS/NIR instruments and techniques	UV/VIS/NIR instruments and techniques may include: • peristaltic sample pumps for flow injection analysis and sipper cells • single/double beam instruments and single/double/triple monochromators • discrete analysers for specific analytes • gas, liquid and thermally jacketed cells • fibre optic probes to measure fluorescence from rocks, gems and minerals • diffuse reflectance, (variable angle) specular reflectance accessories • double aperture accessories • rear beam attenuators • tablet dissolution accessories • data systems such as recorders, electronic integrators, and software packages for peak detection and spectra manipulation
Testing that uses UV/VIS/NIR spectroscopy	Testing that uses UV/VIS/NIR spectroscopy may include: • medical (pathology) testing of whole blood and urine (e.g. phosphates, nitrates and creatine) • forensic testing to establish analyte 'fingerprint' and possible source of scene of crime samples (e.g. paint by specular reflectance) • environmental monitoring of pollution in air, water or soil (e.g. colour and oxidisable organics) • control of starting materials, in-process materials and final products in a wide range of industry sectors (e.g. paper) • food and beverage (e.g. colour and organic compounds), tri-stimulus analysis(determination of food components such as moisture and protein) • pharmaceuticals (e.g. limit tests for active ingredients) • geological testing (e.g. characterisation of rocks, gems and minerals by fluorescence)
Presumptive tests	Presumptive tests may include:

RANGE STATEMENT	
	• source of sample • type and quantity of sample • assessing suitability of sample and specified preparation for spectroscopic technique • presence of interfering substances • stability of chromophore

RANGE STATEMENT	
Sample and standard preparation	Sample and standard preparation may include: • identification of any hazards associated with the samples and/or analytical chemicals • grinding, dissolving, extraction, filtration, refluxing, 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 • cell positioning • cell matching • checking integrity and alignment of accessories • wavelength accuracy • detector signal to noise ratio • stray light levels • 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. absorption and emission) • wavelength range and start/finish • spectral bandwidth (slit width and signal to noise ratio) • scan speed/fixed wavelength • temperature control for kinetic studies
Common analytical procedure problems and remedies	Common analytical procedure problems and remedies may include: • cell cleanliness • mismatched optical cell pairs • poor selectivity • alignment of external attachments • photo-degeneration and sample instability

RANGE STATEMENT	
Common equipment problems	Common equipment problems may include: • adjustment of optical elements such as mirrors and lamps • alignment of external accessories • cleanliness/optical matching of cells
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, 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 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

RANGE STATEMENT	
	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		