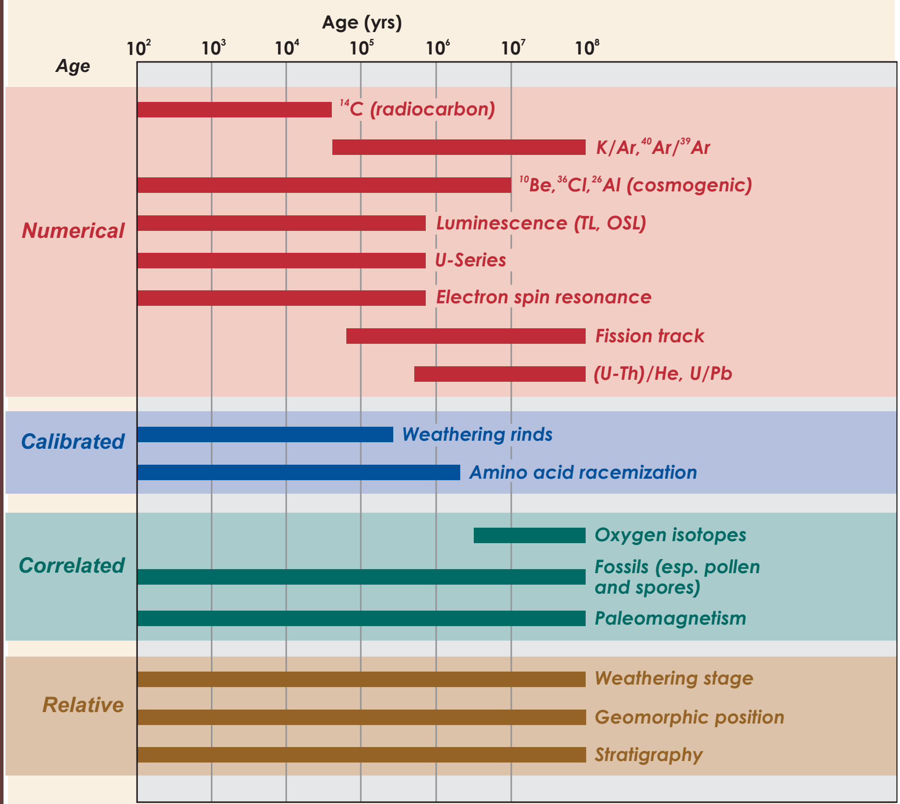
Cooperative Research Centre for Landscape Environments and Mineral Exploration

Regolith dating methods: A guide to numerical dating techniques in Australia

Brad Pillans



REGOLITH DATING METHODS: A GUIDE TO NUMERICAL DATING TECHNIQUES IN AUSTRALIA

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Regolith Dating Methods: A Guide to Numerical Dating Techniques in Australia

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PREFACE TO THE SECOND EDITION

The decade since the publication of the first edition of this guide (Pillans 1998) has seen a marked upsurge in the application of geological dating methods to Australian regolith. A significant driver of this upsurge has been the research programs of CRC LEME (see Pillans 2005, 2006).

The format and content of the first edition was well received, so changes to this edition largely reflect updates to the reference lists and laboratory contact details. However, some techniques that were only briefly mentioned in the first edition, such as (U-Th)/He and SHRIMP U/Pb dating, are now fully described.

INTRODUCTION

Before the twentieth century, notions of geological age and time were founded on the principles of geological correlation and superposition. In other words, geologists erected a relative time scale, which was expressed in the broadest sense by the now standard geological time scale of eras (e.g. Mesozoic), periods (e.g. Cretaceous), stages (e.g. Albian), and so on. However, since 1905, when Ernest Rutherford first proposed that radioactive decay could form the basis of dating minerals, a wide variety of geological dating techniques have been discovered and refined. Consequently, geologists increasingly are able to assign numerical ages (and age uncertainties) to particular strata and events.

The purpose of this guide is to provide information on existing dating techniques that may be useful in determining numerical ages of Australian regolith materials. It does not contain detailed information on how each method works, but rather is intended as a guide to selecting appropriate methods for particular dating problems. In almost every case close consultation with dating practitioners regarding sampling strategies and general methodologies, is recommended.

For each dating method, the following headings are used:

METHOD:	Name of technique, e.g. fission-track.
TYPE:	Classified according to type of result: e.g. calibrated age or type of method; e.g. isotopic (Colman *et al.* 1987). See *Table 1*.
AGE RANGE:	Age range over which the method may be applied (*Figure 1*).
PRECISION:	Uncertainty of age determination.
MATERIALS:	Types of minerals, rocks, etc which can be dated.
DESCRIPTION:	Summary of how the method works.
APPLICATIONS:	General summary of applications in regolith studies.
PROBLEMS:	Complications, tricks, assumptions etc in applying the method.

FIELD SAMPLING: Sampling equipment, sample size etc.

LABS: Names of laboratories where the measurements are carried out.

COSTS: Indicative costs for age determinations.

REFERENCES: Key papers illustrating methodology, applications, etc.

SIDEREAL[1]	ISOTOPIC	RADIOGENIC	CHEMICAL/ BIOLOGICAL	GEOMORPHIC	CORRELATION
Historical records	**Radiocarbon**	**Fission-track**	**Racemization**	**Weathering rinds**	**Paleomagnetism**
Dendro-chronology	**K/Ar and Ar/Ar**	**Luminescence**	Obsidian hydration	Soil development	**Stable isotopes**
Varve chronology	**Cosmogenic isotopes**	**ESR**	Lichenometry	Geomorphic position	**Spores & pollen**
	(U+Th)/He		Soil chemistry	Rate of deposition	Other fossils
	U/Pb			Rate of deformation	Orbital variations
					Lithostratigraphy
					Tektites
[1]SIDEREAL: based on solar time.					Tephrochronology
					Rock varnish

Table 1: Classification of dating methods according to type method employed (modified from Colman et al. 1987).

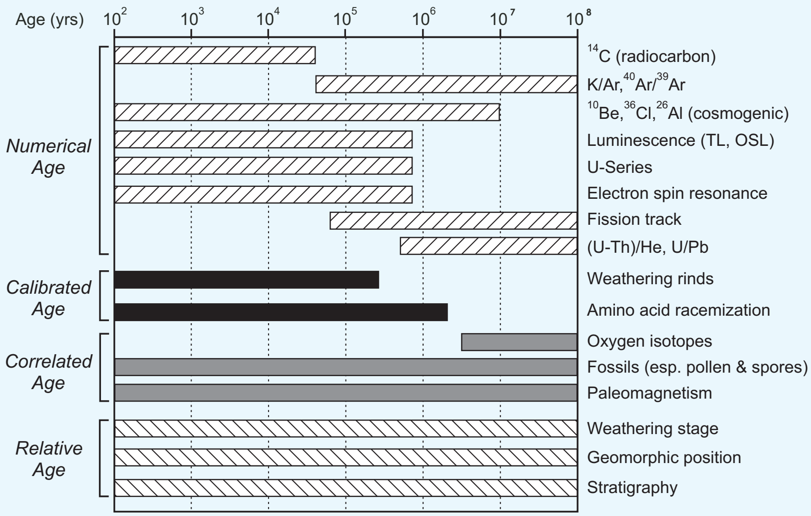

Figure 1: Age ranges over which regolith dating methods can be applied. Methods are grouped according to type of age result produced (see Colman et al. 1987).

TERMINOLOGY

Wherever appropriate, the recommendations of Colman *et al.* (1987) are followed:

"A date is a specific point in time, whereas an age is an interval of time measured back from the present. The use of 'date' as a verb to describe the process of producing age estimates is generally accepted. However, in geologic applications, 'date', when used as a noun, carries a connotation of calendar years and a degree of accuracy that is seldom appropriate. Most 'dates' are better described as 'age estimates' or simply 'ages'. Exceptions include dates derived from historical records, and some ages derived from tree rings or varves. In spite of its connotations, we recognize that the use of 'date' is firmly entrenched, that alternatives are sometimes awkward, and that the verb or its derivatives are acceptable. For these reasons we use the phrase 'dating methods' and we do not expect phrases such as 'radiocarbon dating' to be abandoned."

In terms of abbreviations used to express ages, Colman *et al.* (1987) followed the North American Commission on Stratigraphic Nomenclature in distinguishing between ages determined by geochronologic methods and other intervals of time. They recommend "the use of the SI-derived abbreviations ka and Ma (thousand and million years, respectively, measured from the present) for ages, and informal abbreviations such as yr, k.y. and m.y. for time intervals. Time measured from the present is implicit in ka and Ma; neither 'before present' nor 'ago' should be added to these abbreviations. Radiocarbon dating has established the use of the phrase 'yr B.P.' to indicate ^{14}C ages measured from 1950 A.D. To avoid confusion, the use of yr B.P. should be restricted to radiocarbon ages."

Colman *et al.* (1987, p. 315) strenuously object to the use of the word absolute to describe any dating method. As they say: "this term is more appropriate for despots and deities than for dating methods." Absolute is commonly used to describe the results of isotopic dating methods; however, analytical uncertainties, undetected contamination, and geological uncertainties—such as the temporal relationship between a geological event and time zero of a dating method—all mean that isotopic ages are less than absolute. It is recommended that the term absolute should be replaced with the term numerical, for ages.

GENERAL REFERENCES ON DATING METHODS

A number of books, book chapters and journal articles deal with a range of dating methods, rather than a single method. For a general, non-specialist introduction to dating methods, these sources are often the best way to become familiar with applications and methodologies of dating methods. These publications are included in the reference list below.

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METHODS

Radiocarbon

TYPE:	Numerical age; isotopic.
AGE RANGE:	0–50 ka.
PRECISION:	Better than ± 1% for samples 2–20 ka; lower precision in younger and older samples.
MATERIALS:	Organic materials including shell, charcoal, wood, peat, carbonates, etc.
DESCRIPTION:	Radioactive decay of ^{14}C with half life 5730 years. By convention, ages are reported as years B.P. (before present, i.e. 1950 AD) using the 'Libby' half-life of 5568 years. Because the amount of ^{14}C in the atmosphere is not constant, conversion of radiocarbon age to calendar age uses calibration curves from dated tree rings.
APPLICATIONS:	Numerous applications in 'young' sedimentary sequences.
PROBLEMS:	Age limited; contamination; organics uncommon in some sedimentary environments; radiocarbon ages need calibration before comparison with results from other techniques.
FIELD SAMPLING:	Sample size depends on material: a few grams of charcoal is recommended for a conventional age determination, whereas more than 1 kg may be required to date soil materials with low (< 1%) carbon contents. Milligram- to microgram-size samples may be dated by accelerator mass spectrometry (AMS).
LABS:	Australian National University (Dr S Fallon). Australian Nuclear Science and Technology Organisation, Lucas Heights (Dr D Fink). Waikato University, New Zealand (Dr A Hogg).

COSTS: Typically around $400–450 for conventional ages; $700–900 for AMS ages.

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Potassium–argon and argon–argon

TYPE: Numerical age; isotopic.

AGE RANGE: Typically 100 ka+; occasionally as young as 20 ka or younger in favourable circumstances (e.g. high K-bearing minerals).

PRECISION: Typically 1% for K/Ar and better than 1% for Ar/Ar in cases where proportion of radiogenic argon > 10%.

MATERIALS: Potassium-bearing minerals in igneous and metamorphic rocks; well crystallised whole rock samples, especially volcanic rocks; secondary alunite and K-Mn oxides; glauconite.

DESCRIPTION: Based on radioactive decay of ^{40}K to ^{40}Ar. In the conventional K–Ar dating method, total K is measured on an aliquot of the sample, and the amount of ^{40}K determined from the essentially constant $^{40}K/K$ ratio in terrestrial materials; the amount of ^{40}Ar is determined on a separate aliquot by isotope dilution. In the $^{40}Ar/^{39}Ar$ method, the argon and potassium are determined on a single aliquot of the sample by isotope analysis in a mass spectrometer, after irradiation in a nuclear reactor to convert a proportion of the ^{39}K atoms to ^{39}Ar atoms. Because the $^{40}K/^{39}Ar$ ratio is essentially constant in nature, the $^{40}Ar/^{40}Ar$ ratio, and hence the age, can then be calculated. Although it is more time consuming and more expensive, the $^{40}Ar/^{39}Ar$ method has advantages over the conventional K–Ar method, such as smaller sample size and greater precision; also, stepwise heating release of Ar from a sample yields a spectrum of apparent ages, which provides better understanding of the thermal history of a sample.

APPLICATIONS: Wide applications in dating igneous rocks and secondary minerals.

PROBLEMS: Argon loss, excess and inherited argon.

FIELD SAMPLING: Rock samples must be fresh and unaltered; alteration of rock samples and purity of secondary mineral phases should be verified in the laboratory. Sample size varies with age, material and K content.

LABS: Australian National University (Dr M Forster).

Melbourne University (Dr D Foster).

University of Queensland (Dr P Vasconcelos).

COSTS: For collaborative studies, typically $650 for K/Ar and $1000 for $^{40}Ar/^{39}Ar$; commercial rates available on request.

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Cosmogenic isotopes (e.g. ^{3}He, ^{10}Be, ^{26}Al, ^{21}Ne, ^{36}Cl)

TYPE:	Numerical age; isotopic.
AGE RANGE:	0–5 Ma.
PRECISION:	3–10%, depending on age, sample type and measured isotope.
MATERIALS:	Quartz; carbonates; Ca-, K-, Cl-bearing whole rocks; soils.
DESCRIPTION:	Cosmic ray interactions produce ^{3}He, ^{10}Be, ^{21}Ne, ^{26}Al and ^{36}Cl, in the atmosphere and lithosphere. Accumulation reflects the duration of cosmic ray exposure within the upper 1–2 metres of the Earth's surface.
APPLICATIONS:	Rock surface exposure ages and erosion rates.
PROBLEMS:	Uncertainties in isotope production rates and altitude/latitude corrections. Ability to determine surface age often compromised by erosion: resulting in a minimum age estimate.
FIELD SAMPLING:	Stable rock surfaces, which must have been created by the event to be dated. Collect surface sample up to ~5 cm thick. Near-horizontal surfaces most appropriate; otherwise corrections for cosmic-ray obstruction are required. Sample altitude and latitude are required for age calculation.
LABS:	Research Schools of Earth Sciences and Physical Sciences and Engineering, Australian National University (Prof. K Fifield). Australian Nuclear Science and Technology Organisation, Lucas Heights (Dr D Fink).
COSTS:	$600–1800 per isotopic determination, depending on preparation costs.

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(U/Th)/He

TYPE:	Numerical age, isotopic.
AGE RANGE:	1 Ma+.
PRECISION:	Typically ± 5% or better
MATERIALS:	Iron oxides, apatite.
DESCRIPTION:	Radioactive decay of U and Th produces ^{4}He which is trapped in crystal lattices. Ages for Fe oxides are minimum ages unless corrected for He diffusion.
APPLICATIONS:	Potential wide applications in weathered regolith materials containing Fe oxides.
PROBLEMS:	He diffusion.
FIELD SAMPLING:	Contact lab for details.
LABS:	University of Melbourne (Dr B Kohn)
	The Australian National University (Dr M Honda)
	California Institute of Technology (Prof. K Farley)
COSTS:	Negotiable.

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Stock GM, Ehlers TA and Farley KA, 2006. Where does sediment come from? Quantifying catchment erosion with detrital apatite (U-Th)/He thermochronometry. *Geology* 34, 725–728.

Uranium–lead

TYPE:	Numerical age; isotopic.
AGE RANGE:	100 yrs–4.5 Ga+.
PRECISION:	Varies with age and material—typically 0.1–1% in zircon; around 2% in speleothems reported by Woodhead *et al.* (2006).
MATERIALS:	Zircon is most common mineral dated, but monazite, titanite, xenotime and baddeleyite are also suitable; carbonates (e.g. speleothems) can also be dated.
DESCRIPTION:	Based on radioactive decay of ^{238}U to ^{206}Pb (half life ~4.5 Ga) and ^{235}U to ^{207}Pb (half life ~700 Ma).
APPLICATIONS:	The sensitive high-resolution ion micro-probe (SHRIMP) designed and operated in the Research School of Earth Sciences, ANU, allows U/Pb dating of single zircons. Although normally applied to rocks of great antiquity (e.g. Froude *et al.* 1983), this method is widely used for provenance studies of detrital zircon.
PROBLEMS:	Non-radiogenic Pb in carbonates; Pb loss in zircon
FIELD SAMPLING:	Sample size varies with lithology
LABS:	Australian National University (Dr IS Williams)
	Melbourne University (Dr J Woodhead)
	Curtin University (Dr M McWilliams)
COSTS:	Available on request.

REFERENCES:

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Woodhead J. Hellstrom J, Maas R, Drysdale R, Zanchetta G, Devine P and Taylor E, 2006. U-Pb geochronology of speleothems by MC-ICPMS. *Quaternary Geochronology* 1, 208–221.

Luminescence (including TL and OSL)

TYPE:	Numerical age; radiogenic.
AGE RANGE:	10 yrs–1 Ma.
PRECISION:	± 5–15% (OSL), ± 10–20% (TL).
MATERIALS:	Quartz, feldspars and gypsum in aeolian sediments.
DESCRIPTION:	Natural radiation (α, β, γ and cosmic) produces electron and hole pairs, which can be trapped at defects in a crystal lattice. Luminescence is emitted when electrons liberated from the traps by heating (thermoluminescence or TL) or exposure to visible - wavelength light (optically stimulated luminescence or OSL) or infra-red light (infra-red stimulated luminescence or IRSL) recombine with trapped holes. The greater the radiation dose accrued, the greater the resulting luminescence. Age is found by calibrating the luminescence with known radiation doses, in combination with measurements of the environmental dose rate.
APPLICATIONS:	Wide application in Australian landscapes, especially aeolian deposits.
PROBLEMS:	Incomplete bleaching during sediment transport
FIELD SAMPLING:	Samples cannot be exposed to light. Usually, steel tubes are hammered into the outcrop, sealed in black polythene and only subsequently opened under darkroom conditions in the luminescence lab. Extra samples are taken for radioisotope assays and water content determinations. Sediment should ideally be of uniform composition within a 30 cm radius of the sample (the limit of the γ radiation dose).
LABS:	Australian National University (Dr K Fitzsimmons). University of Wollongong (Prof. RG Roberts). University of Adelaide (Prof. J Prescott). University of Melbourne (Dr M Cupper) CSIRO (Dr Jon Olley)

COSTS: $700–2000 depending on nature of sample.

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Uranium series

TYPE:	Numerical age; isotopic.
AGE RANGE:	0–1 Ma.
PRECISION:	Better than 0.5% using mass spectrometry.
MATERIALS:	Carbonates (speleothems, corals), U-bearing oxides, bone, volcanic rocks; opal; peat is sometimes suitable.
DESCRIPTION:	Based on decay or accumulation of various parent/daughter isotopes in the U-series decay chains, especially $^{234}U/^{238}U$ and $^{230}Th/^{234}U$.
APPLICATIONS:	Commonly used for dating cave speleothems, and fossil corals in shoreline deposits.
PROBLEMS:	Uranium mobility; detrital Th contamination. For example, dating pedogenic calcrete is generally unreliable because of detrital Th contamination.
FIELD SAMPLING:	Check corals for recrystallisation from aragonite to calcite.
LABS:	Australian National University (Prof M McCulloch).
	Melbourne University (Dr J Woodhead, Dr J Hellstrom).
	ANSTO (Dr T Esat).
	University of Queensland (Prof. K Collerson).
COSTS:	Negotiable.

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Electron spin resonance

TYPE:	Numerical age; radiogenic.
AGE RANGE:	2 ka–1 Ma+.
PRECISION:	Best values around 5–7%.
MATERIALS:	Carbonates (shell, coral), teeth, quartz, gypsum, silcrete.
DESCRIPTION:	Radiation produces unpaired electrons in crystal lattices. The concentration of unpaired electrons increases with time and radiation dose.
APPLICATIONS:	Widely used overseas for dating vertebrate fossils, including human fossils. Has been used to date silcrete in Queensland.
PROBLEMS:	Dose response curves not well defined; U migration in teeth.
FIELD SAMPLING:	Contact lab for details.
LABS:	Quaternary Dating Research Centre, ANU (Dr R Grün).
COSTS:	Only available for collaborative research projects. Costs will be detailed for specific projects.

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Fission track

TYPE:	Numerical age
AGE RANGE:	150 ka+.
PRECISION:	Typically better than 5–10%.
MATERIALS:	Uranium-bearing minerals such as zircon, sphene, apatite, volcanic glass; possibly gypsum.
DESCRIPTION:	Spontaneous fission of ^{238}U produces two fragments that disrupt the crystal lattice to create damage tracks. The number of tracks is proportional to the age of the U content and thermal history. Dates the time of cooling below a mineral-dependent track annealing temperature.
APPLICATIONS:	Widely used overseas for dating tephras. Major application in Australia to thermochronology of landscape evolution. If gypsum proves to be a suitable mineral (c.f. Li 1991), then many new applications will become possible in Australia.
PROBLEMS:	Interpretation of fission-track data in terms of denudation history is complex.
FIELD SAMPLING:	Sample size depends on mineral content. Contact lab for details.
LABS:	Melbourne University (Prof A Gleadow; Assoc Prof B Kohn).
COSTS:	$400 per mineral sample for collaborative studies; commercial rates available on request.

REFERENCES:

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Amino acid racemisation

TYPE: Calibrated age.

AGE RANGE: 100 yrs –1 Ma+, depending on temperature and nature of material.

PRECISION: Better than ± 10% in favourable circumstances.

MATERIALS: Organic materials containing fossil proteins including marine shell, wood, eggshell, speleothems, teeth, bone, snail shell.

DESCRIPTION: Living organisms contain dominantly L-amino acids. After death, the amino acids undergo diagenetic changes, including racemisation, which converts L-amino acids into D-amino acids. The D/L ratio increases over time at a rate that is dependent on temperature.

APPLICATIONS: Widely used for correlation and dating of coastal deposits containing molluscan shells. Can be used to calculate palaeotemperatures when age is independently known.

PROBLEMS: Temperature dependence of racemisation; requires calibration using other numerical dating methods; contamination and degradation of proteins.

FIELD SAMPLING: Avoid excessive handling of samples; seal in plastic bags. Samples from depths of less than 1 m may be adversely affected by ground-temperature fluctuations.

LABS: University of Wollongong (Dr C Murray-Wallace).

CSIRO, Adelaide (Dr R Kimber).

COSTS: Contribution to costs depending on nature of collaboration.

REFERENCES:

Bowen DQ, Pillans B, Sykes GA, Beu AG, Edwards AR, Kamp PJJ and Hull AG, 1998. Amino acid geochronology of Pleistocene marine sediments in the Wanganui Basin: a New Zealand framework for correlation and dating. *Journal of the Geological Society, London* 155, 439–446.

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Weathering rinds

TYPE:	Calibrated age.
AGE RANGE:	100 yrs–1 Ma.
PRECISION:	Better than ± 10% in favourable circumstances.
MATERIALS:	Rocks of relatively uniform composition and grain size; may be exposed rock surfaces or pebbles and boulders that are buried below the ground surface.
DESCRIPTION:	The depth of chemical weathering on the surfaces of rocks progressively increases over time, to produce a weathering skin or rind.
APPLICATIONS:	Used overseas for dating and correlation of glacial moraines.
PROBLEMS:	Requires calibration.
FIELD SAMPLING:	Samples are obtained by fracturing weathered clasts with a heavy hammer so as to obtain small rock chips with complete sections through the rinds. At least 50 rind chips are recommended for each dated site.
LABS:	No specialised lab facilities required. See McSaveney (1992) for details of measurement techniques, etc.
COSTS:	Nil.

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Palaeomagnetism

TYPE: Correlated age.

AGE RANGE: 100 yrs–100 Ma+.

PRECISION: Varies with age. Typically ± 10 Ma for Cenozoic age estimates determined using the Australian Apparent Polar Wander Path (AAPWP).

MATERIALS: Most igneous, metamorphic and sedimentary rocks, palaeosols, oxidised regolith materials.

DESCRIPTION: Palaeomagnetism uses past changes in the Earth's magnetic field on three broad time scales:

1. Secular variation, on time scales of 10 yrs–10 ka.

2. Reversal stratigraphy, on time scales of 10 ka–100 Ma+.

3. Apparent polar wander, on time scales of 5–100 Ma+.

APPLICATIONS: Wide application in regolith studies, including sedimentary sequences and weathered regolith materials.

PROBLEMS: There is some uncertainty regarding the Cenozoic apparent polar wander path for Australia. Reversal stratigraphy must be tied to independent ages.

FIELD SAMPLING: Rocks are sampled using a portable rock drill; unconsolidated samples are obtained using 6 cm3 plastic boxes. Samples must be oriented using a magnetic compass (corrected for local field variation) or sun compass.

LABS: Australian National University (Prof. B Pillans)

CSIRO, North Ryde (Dr P Schmidt)

University of Western Australia

COSTS: Available on request.

REFERENCES:

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Stable isotopes (^{18}O, ^{13}C)

TYPE: Correlated age.

AGE RANGE: 5 Ma+.

PRECISION: Provides broad age estimates only, e.g. Late, Middle and Early Cenozoic.

MATERIALS: Clay minerals, especially kaolinite; Fe oxides

DESCRIPTION: Systematic variations in the isotopic composition of meteoric waters—and therefore of the authigenic regolith materials that formed in equilibrium with them—are the result of the drift of the Australian continent from high to low latitudes and changes in global climate.

APPLICATIONS: Potential wide applications in weathered regolith materials containing kaolinite and/or Fe oxides.

PROBLEMS: Lacks precision.

FIELD SAMPLING: Contact lab for details.

LABS: University of Wollongong (Prof. A Chivas)

University of Queensland

COSTS: Negotiable.

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Pollen spores, dinoflagellates and acritarchs

TYPE:	Correlated age
AGE RANGE:	0–400 Ma
PRECISION:	Resolution varies with age and location—from less than 1 Ma for Cenozoic sections in southeast Australia to greater than 10 Ma for Palaeozoic rocks in northwest and central Australia.
MATERIALS:	Most fine-textured sediments that have not been subjected to prolonged weathering.
DESCRIPTION:	Usually based on a combination of first and last appearances of selected fossil species (concurrent range zones); less often using relative abundance data or associations between pairs of species (Opel zones).
APPLICATIONS:	Widely used for age control, correlation and palaeoenvironmental analysis in onshore sedimentary basins and analogous depositional environments, e.g. palaeochannels incised into Archean rocks.
ADVANTAGES:	Provides a cheap, quick and reliable method for correlating within and between widely separated sedimentary basins.
PROBLEMS:	Contamination due to downhole caving (rotary chip samples); bioturbation and reworking of older sediments (all depositional environments); oxidative destruction of organic matter by weathering processes (most areas).
FIELD SAMPLING:	Preferably core or cuttings from fully flushed drill holes. Strongly weathered or jointed outcrops should be avoided.
LABS:	Geoscience Australia (Assoc. Prof. C Foster) Australian National University (Prof. G Hope) Primary Industries and Resources, Adelaide (L Stoian)

Monash University (Prof. AP Kershaw)

CoreLab (Australia) Pty Ltd, Perth

COSTS: $100–350 per sample (including processing), depending on requirements, including age determination and detailed report.

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