14.1.1 Introduction

The K/Ar dating scheme, using the conventional K/Ar method, and its variant, the 40Ar/39Ar technique, have become arguably the most utilized methods for providing precise and accurate numerical ages for igneous rocks or igneous-derived rocks in the age range from ~50 ka to >10 Ma, and indeed to much older rocks in the geological record. As will be explained, the techniques commonly yield ages of precision of 1% or better in many cases, but with decreasing precision at the younger end of the time scale because the proportion of radiogenic argon (40Ar*) compared with the amount of atmospheric 40Ar present progressively decreases. Provided that igneous rocks are interbedded within a stratigraphic succession, it is commonly possible to provide numerical ages for fossils within the sequence by extrapolating from the measured ages. Indeed, many of the ages now quoted for fossils, especially hominins, are constrained by 40Ar/39Ar ages on interbedded volcanic rocks or tuffaceous equivalents.

Historically, the K/Ar dating method has been of considerable importance in providing age information related to a variety of studies over the last six decades since Aldrich and Nier (1948) identified radiogenic argon. These studies include the delineation and calibration of the geomagnetic polarity time scale, one of the essential foundations for the development of plate tectonic concepts; the numerical calibration of the relative geological time scale, as well as calibration of the evolution of flora and fauna; and the determination of rates of geological processes, and the age of individual geological events.

In this chapter, the basis for the application of the K/Ar and 40Ar/39Ar dating techniques will be outlined, together with their limitations, followed by an example of what has been achieved in the Omo-Turkana Basin of northern Kenya and southern Ethiopia, with reference to examples elsewhere, especially in the Afar region of Ethiopia. It is emphasized that dating by these techniques is confined principally to measurements on igneous rocks or their explosive products, so that they are not directly applicable to fossiliferous sequences that consist primarily of detrital sediments or other sedimentary deposits, or to the fossils themselves. For example, the sequences that are found in the important hominin-bearing cave deposits in the Malapa–Sterkfontein region north of Johannesburg in Gauteng Province, South Africa, are dominated by clastic sediments and limestones in caves, where other dating techniques must be employed, including U/Pb measurements directly on limestone flowstones, which in particularly favorable circumstances yield ages at high precision (cf. Berger et al., 2010).

14.1.2 Basis of the K/Ar and 40Ar/39Ar Dating Techniques

An isotope of potassium, 40K, is naturally radioactive with a total decay constant (λ) of about 5.543 × 10^-10 year^-1 (Steiger and Jäger, 1977; error at 1σ from Min et al., 2000) or a half life of 1250 Ma. Renne et al. (2010, 2011) recently derived a slightly different total decay constant of 5.5305 ± 0.0135 × 10^-10 year^-1, based upon a comparison of 40Ar/39Ar ages with 238U/206Pb ages on the same rocks, as the 238U decay constant is very well determined. For the present, it is not necessary to discuss this in detail; generally, the recommended values of parameters as given by Steiger and Jäger (1977) will be followed, because there is still uncertainty as to the correct values of the 40K partial decay constants (see Renne et al., 2011; Schwarz et al., 2011). The radioactive K isotope is present in small amounts in all terrestrial K-bearing materials; it has an abundance in terrestrial samples of 1.167 × 10^-4 mol mol^-1 K^-1 (±0.34%), that is, ~1 atom in ~8570 atoms of K (Garner et al., 1975). The main isotopes of K, both stable, are 39K (93.3%) and 41K (~6.7%) (see Figure 1). The radioactive 40K has a dual decay (Figures 1 and 2) to 40Ca (~89.5% of decays) and 40Ar (~11.5% of decays). The decay to 40Ca can be utilized as the basis for a dating method, but it is rarely used, simply because Ca is common in nature and 40Ca is the dominant isotope (96.9%) (cf. Marshall and DePaolo, 1982). However, the branch of the decay yielding radiogenic argon (40Ar*) is the basis for a particularly useful geochronometer, the K/Ar method and its variant the 40Ar/39Ar technique. How does the K/Ar clock work? It is particularly applicable to igneous
Figure 1  Chart of the naturally occurring isotopes of chlorine, argon, potassium, and calcium (boxes with solid outlines), together with a number of the radioactive isotopes that are produced artificially (boxes with dashed outlines) and are relevant to the 40Ar/39Ar dating technique. Arrows indicate the decay paths for the radioactive nuclides. For each isotope, the mass number is shown as a superscript to the chemical symbol; the natural abundance (in atomic percent) is listed below the symbol, and the half life for each unstable isotope is given at the bottom of each relevant box. Reproduced from McDougall I and Harrison TM (1999) Geochronology and Thermochronology by the 40Ar/39Ar Method, 2nd edn. New York: Oxford University Press.

Figure 2  Decay scheme for 40K, illustrating the dual decay to 40Ca and 40Ar. This is adapted from the recommended values of Steiger and Jäger (1977), but note that some revisions have been advocated by Renne et al. (2010, 2011), partly following Min et al. (2000). Reproduced from McDougall I and Harrison TM (1999) Geochronology and Thermochronology by the 40Ar/39Ar Method, 2nd edn. New York: Oxford University Press.
rocks, including tuffaceous products, because at the high temperature of a magma, any preexisting $^{40}$Ar* is normally lost from the system as, at low pressure, argon is a gas so that it preferentially degasses to the atmosphere. Thus, the magma on eruption generally has equilibrated with atmospheric argon, and the clock is set to zero, noting that the K remains in the magma. Following eruption and cooling, the K continues to decay and in favorable circumstances the $^{40}$Ar* is retained within the sample, accumulating with the passage of time. The basic age equation for the K/Ar system is as follows:

$$t = \frac{1}{\lambda_c} \ln \left( 1 + \frac{\lambda_c}{\lambda e}^{40} \frac{\text{Ar}}{39} \right)$$

where $t$ is the calculated age, $\lambda_c$ is the total $^{40}$K decay constant, $\lambda e$ is the partial decay constant for the $^{40}$K to $^{40}$Ar* branch ($\sim 0.581 (\pm 0.008) \times 10^{-10}$ year$^{-1}$, where the 1σ error is after Min et al. (2000)), $^{40}$Ar* is the measured radiogenic argon, $^{40}$K is the derived amount of $^{40}$K in the sample, as usually the total K is measured (by flame photometry, atomic absorption spectrometry, or isotope dilution) and the $^{40}$K is calculated using the abundance determined by Garnier et al. (1975). This can be done because Humayun and Clayton (1995) have shown that the $^{39}$K/$^{40}$K ratio is constant in terrestrial materials to better than 0.1%, and it is assumed that this also applies to the $^{39}$K/$^{40}$Ar ratio. The decay constants often utilized have been derived from counting experiments on K. Thus, we are dealing with an accumulative clock, whereby the $^{40}$Ar* increases with time from the eruption. In the K/Ar method, the $^{40}$Ar* is measured on a separate aliquot of the sample usually by isotope dilution, normally using $^{38}$Ar as the calibrated tracer added to the argon extracted from the sample under high-vacuum conditions, and following purification of the gas on getters, the isotopic composition of the argon is measured in a mass spectrometer. In well-calibrated systems, it is possible to measure the amount of argon without the presence of an isotopic tracer (cf. Cassignol and Gillot, 1982; Charbit et al., 1998), detecting small variations in isotopic composition of argon from that of atmospheric argon and hence detecting small variations in isotopic composition of argon spectrometer. In well-calibrated systems, it is possible to measure the amount of argon without the presence of an isotopic tracer (cf. Cassignol and Gillot, 1982; Charbit et al., 1998), detecting small variations in isotopic composition of argon from that of atmospheric argon and hence detecting small variations in isotopic composition of argon, and the clock is set to zero, noting that the K remains in the magma. Following eruption and cooling, the K continues to decay and in favorable circumstances the $^{40}$Ar* is retained within the sample, accumulating with the passage of time. The basic age equation for the K/Ar system is as follows:

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The basic age equation for the $^{40}$Ar/$^{39}$Ar technique is as follows:

$$t = \frac{1}{\lambda} \ln \left( 1 + \frac{J}{\lambda}^{40} \frac{\text{Ar}}{39} \right)$$

where $J$ is an irradiation parameter, defined below, and $^{39}$Ar* is the Ar* produced from $^{39}$K during the irradiation. To determine $J$, a sample of precisely known K/Ar age (the fluence monitor) is irradiated together with the unknown from which the irradiation parameter is obtained via the following equation:

$$J = \frac{\exp \left( \frac{\lambda t}{\lambda} \right) - 1}{\lambda^{40} \frac{\text{Ar}}{39} \text{K}}$$

where $\lambda$ is the total $^{40}$K decay constant, $t$ is the K/Ar age of the fluence monitor, and $^{40}$Ar*/$^{39}$Ar* is the derived ratio from the measured $^{40}$Ar*/$^{39}$Ar in the gas extracted from the fluence monitor. Thus, for the irradiation in fast neutrons, the fluence monitor crystals are placed in known geometric relationship to the unknown samples, either in small packets in a tube or in machined wells in an aluminum disc (cf. Renne et al., 1998; Vasconcelos et al., 2002). After irradiation in a facility in a nuclear reactor which has a suitable fast nuclear flux with minimal flux gradients, the individual fluence monitor samples, often as single crystals, are fused to release their Ar in a high-vacuum extraction system, and, after gas purification, the Ar is measured isotopically in a mass spectrometer. Typically, with careful attention to geometry, the neutron flux gradients observed are small to negligible, and in any case can be readily allowed for. The unknowns can then be measured, utilizing the determined $J$, so that the age of a sample can be determined, often with a precision of better than 0.5%. As it is necessary to measure only isotope ratios, from which an age can be determined, this approach is inherently more precise than the conventional K/Ar age measurement, which requires measurement of the amount of $^{40}$Ar* on one aliquot of the sample and of K on another aliquot. In addition, the ability to measure ages on single crystals of unknowns is a major bonus in the $^{40}$Ar/$^{39}$Ar technique. With automated extraction systems and mass spectrometers, now typical of $^{40}$Ar/$^{39}$Ar laboratories, it is also possible to undertake a number of measurements on single crystals to verify that a single juvenile population is present, or to differentiate between older crystals, such as xenocrysts, and a juvenile component.

It is stressed that many of the same assumptions must be satisfied for either technique to yield a geologically realistic age. These are as follows:

1. The rock or mineral being dated was fully degassed of pre-existing $^{40}$Ar* at the time of eruption, so that the clock was set to zero. For a volcanic rock, or for a magma erupted near the Earth’s surface, rapid cooling to ambient temperature will have occurred. In general, no $^{40}$Ar* is trapped in the rock or mineral at the time of crystallization, but this assumption needs to be verified.
2. The $^{40}\text{Ar}^*$ generated from $^{40}\text{K}$ since the rock or mineral crystallized has been fully retained in the sample.
3. No change in K concentration has occurred subsequent to crystallization, apart from very small changes caused by the decay of $^{40}\text{K}$.
4. Appropriate correction is made for nonradiogenic $^{40}\text{Ar}$ and for interfering isotopes in the $^{40}\text{Ar}/^{39}\text{Ar}$ technique (see below).

It almost goes without saying that both techniques require that the measurements are made properly and well, and that the various constants (decay constants, $^{40}\text{K}$ abundance, etc.) are known with appropriate precision.

In many cases, these assumptions can be checked, usually by consistency arguments, and it is often possible to be more thorough in this respect using the $^{40}\text{Ar}/^{39}\text{Ar}$ approach. This is because there is an extra dimension in what can be done with the latter technique, using the step-heating procedure, whereby the argon is released progressively at steps of increasing temperature. The gas released at each step can be analyzed and a so-called age spectrum produced in which the age determined on each gas aliquot is plotted against the proportion of the release. This uses atmospheric argon as the composition of the trapped component. If the age spectrum is essentially flat, as is often the case, then it can usually be asserted with confidence that the sample has not been disturbed or reset by a subsequent geological event, such as by significant reheating. In addition, the data can be plotted on an isochron or isotope correlation diagram, usually $^{36}\text{Ar}/^{40}\text{Ar}$ versus $^{39}\text{Ar}/^{40}\text{Ar}$, and the best-fit straight line can yield an age estimate as well as an independent measure of the trapped argon composition (cf. McDougall and Harrison, 1999).

In the $^{40}\text{Ar}/^{39}\text{Ar}$ dating technique, it is the $^{40}\text{Ar}^*/^{39}\text{Ar}_K$ ratio that needs to be determined (see earlier equation), and this is derived from the isotopic ratios measured on the argon gas extracted from the sample. The equation is listed in McDougall and Harrison (1999, p. 91) and is as follows:

$$
{^{40}\text{Ar}/^{39}\text{Ar}}_K = \frac{^{40}\text{Ar}/^{39}\text{Ar}}{^{40}\text{Ar}/^{39}\text{Ar}} = \frac{(^{40}\text{Ar}/^{39}\text{Ar})_m - 295.5{(^{36}\text{Ar}/^{39}\text{Ar})}_m}{295.5{(^{40}\text{Ar}/^{39}\text{Ar})}_m - (^{40}\text{Ar}/^{39}\text{Ar})_K}
$$

where the subscript $m$ refers to measured ratios, the subscript $Ca$ refers to the production ratios of $^{39}\text{Ar}$ and $^{36}\text{Ar}$ from calcium in the nuclear reactor, and $(^{40}\text{Ar}/^{39}\text{Ar})_K$ is the production ratio for $^{40}\text{Ar}$ from $^{40}\text{K}$ in the nuclear reactor relative to the $^{39}\text{Ar}_K$. The Ca correction factors are referred to the production of $^{37}\text{Ar}$ because this isotope is produced via the nuclear reaction $^{40}\text{Ca}(n,\gamma)^{37}\text{Ar}$, and $^{36}\text{Ar}$ and $^{39}\text{Ar}$ are produced by reactions $^{40}\text{Ca}(n,\alpha)^{36}\text{Ar}$ and $^{42}\text{Ca}(n,\gamma)^{39}\text{Ar}$, respectively (Bereton, 1970; Turner, 1971). Fortunately, these interferences can be allowed for by measuring the $^{37}\text{Ar}$ in the gas after irradiation, as this isotope is essentially only produced from Ca, so that it is used as the reference isotope to correct for $^{36}\text{Ar}$ and $^{39}\text{Ar}$ produced from Ca in the sample. A complication is that $^{37}\text{Ar}$ is radioactive, and decays with a half-life of 35.1 days (Stoennner et al., 1965), but this is readily corrected for provided times of irradiation and the time elapsed between irradiation and measurement are kept track of. Note that after about 1 year since the irradiation, the $^{37}\text{Ar}$ will have decayed away to less than one thousandth of its original concentration, indicating that a reliable Ca correction can only be made if no more than about 6 months have elapsed since irradiation. The production ratios on Ca measured on pure calcium salts generally are very similar to one another, irrespective of the reactor used, simply because they are also products of nuclear reactions on Ca requiring fast neutrons, and all nuclear reactors are based upon U as the fuel and have similar neutron energy distributions. Thus, the mean of many production ratios determined in a number of reactors for $(^{40}\text{Ar}/^{37}\text{Ar})_\text{Ca}$ and $(^{39}\text{Ar}/^{37}\text{Ar})_\text{Ca}$ are 2.65 ($\pm 0.41$) x $10^{-4}$ and 7.7 ($\pm 1.6$) x $10^{-4}$, respectively (McDougall and Harrison, 1999, pp. 58–61). The $(^{40}\text{Ar}/^{39}\text{Ar})_K$ correction factor is much more variable, because the reaction $^{40}\text{K}(n,p)^{40}\text{Ar}$ requires only thermal neutrons for it to proceed (Tetley et al., 1980), and as the thermal neutron fluence varies greatly from neutron irradiation facility to facility, so does the measured value, usually measured on a high-K salt or artificial glass. The use of Ca shielding is employed in some neutron irradiation facilities to absorb the bulk of the thermal neutrons, and this results in low values for the $(^{40}\text{Ar}/^{39}\text{Ar})_K$ correction, often $< 20 \times 10^{-4}$. The Ca correction factors result in only minor corrections when argon is extracted from low-Ca minerals, such as many alkali feldspars, but can be of considerable significance when measuring samples of plagioclase or basalt with relatively high Ca content. Note that $^{39}\text{Ar}$ is also radioactive and decays with about a 269-year half life (Stoennner et al., 1965); a correction for this decay also is routinely made in data reduction programs.

A critical factor in using either variant of the K/Ar dating scheme is that there is always some contamination of the argon sample gas by atmospheric argon, because of equilibration with atmospheric argon during eruption or derived in part from the extraction system and mass spectrometer. This correction always has to be made, and is done by using the measured $^{36}\text{Ar}$ in the argon gas, after correction for any neutron-induced $^{36}\text{Ar}$ from Ca in the case of the $^{40}\text{Ar}/^{39}\text{Ar}$ method. Effectively, what is done is to take the measured or corrected $^{40}\text{Ar}$ and multiply it by the $(^{40}\text{Ar}/^{36}\text{Ar})_\text{atmos}$ of ~295.5, the ratio observed in atmospheric argon (Nier, 1950), to yield the amount of atmospheric $^{40}\text{Ar}$ in the sample gas. Indeed, it is the proportion of atmospheric argon in the gas sample that ultimately limits how young a sample can be measured at an appropriate or stated precision. As the proportion of atmospheric $^{40}\text{Ar}$ approaches 100%, the error in the determination of the $^{40}\text{Ar}^*$ necessarily increases dramatically (Figure 3), so that it may only be possible to place approximate limits on the age at high proportions of atmospheric argon in a run. Because the amount of inherent atmospheric argon in a sample varies enormously, it is very difficult to give a younger limit for detection of radiogenic argon and hence age. Thus, it was found empirically that fresh crystals of alkali feldspar have much lower inherent atmospheric argon than platy micas, often by more than an order of magnitude, and that unaltered and well-crystallized samples of volcanic rocks often also generally had similar atmospheric argon contents as alkali feldspar crystals. It was this characteristic that made it possible to successfully measure K/Ar ages on quite youthful volcanic rocks from the Hawaiian island chain (McDougall, 1964). In addition, the higher the K content, with relatively low inherent atmospheric argon, the younger the age that can be successfully measured. In the extreme case, it may be possible to measure...
ages into the historic era, as shown by Renne et al. (1997), who demonstrated using isochron analysis of samples of sanidine with a high K content (~12.5 wt%) from the Vesuvius eruption of AD 79 that agreement within error was obtained between the 40Ar/39Ar age of 1925 ± 94 years and the historically recorded eruption that overwhelmed Pompeii. A major factor in the measurement of young rocks by the 40Ar/39Ar method has been the ability to measure small ion beams in modern mass spectrometers, generally using multiplier detectors, but increasingly using multibeam collection of isotopes on Faraday cups fitted with O or higher value feedback resistors (cf. Mark et al., 2009). This technology, together with the use of laser beams to fuse individual crystals or small multigrain samples, essentially without heating any of the surrounding high-vacuum vessel, has resulted in major improvements in the ability to measure young samples at high precision.

It should also be noted that the mass spectrometer being used needs to be calibrated. Normally, this is done by measuring atmospheric argon, and utilizing the measured 40Ar/36Ar to calculate the mass spectrometer discrimination per unit argon mass, which is then applied to all other runs. Traditionally, the value of this ratio in atmospheric argon is taken as 295.5 (Steiger and Jäger, 1977; derived from Nier, 1950), but even if the more recent and more precise value for this ratio of 298.56 ± 0.31 of Lee et al. (2006) is used, the results obtained are essentially identical. It is emphasized that whichever ratio is chosen for the normalization, it must be used for determination of discrimination of the mass spectrometer, and that this discrimination must also be utilized on every measurement of argon, whether this be a blank, fluence monitor standard, or an unknown.

14.1.3 Suitable Materials for Dating

Generally, any potassium-bearing mineral or rock sample can be utilized for K/Ar and 40Ar/39Ar dating, but in practice the range of samples that can be successfully used is more restricted. Minerals most suitable for dating are those in which the potassium is located in lattice sites as an essential cation, but some minerals that are useful for dating contain relatively minor amounts of potassium substituting for other cations. In the Pliocene–Pleistocene, most successful age measurements are made on relatively high-potassium minerals and whole rocks, because materials low in potassium in many cases will not have generated sufficient radiogenic argon to be measured effectively.

As previously mentioned, these methods are most useful when applied to igneous rocks, although age information related to cooling often can be obtained from metamorphic rocks. Rarely are the methods applicable to the direct dating of the time of deposition of sedimentary rocks themselves.

It is emphasized that careful examination of all samples under the microscope prior to analysis is very important to determine whether they are suitable for age determination. In the case of larger samples, a thin section provides the means by which examination under a microscope can help an observer to decide which potassium-bearing minerals
might be separated, or whether the sample might be measured as a whole rock if it is too fine-grained to undertake mineral separation. Indeed, many igneous rocks fall into the latter category, and their degree of freshness and crystallinity are major factors in assessing whether such a rock may have retained all its radiogenic argon since crystallization. Lavas that have altered groundmass material or glass replaced by alteration minerals are unlikely to retain all their radiogenic argon since crystallization, leading to variably young apparent ages. Alteration also usually has the effect of increasing the amount of atmospheric argon within a sample, making it more difficult to detect $^{40}$Ar* over atmospheric $^{40}$Ar. Microscopic examination also is an indispensable part of any mineral separation procedure.

Age measurements by the K/Ar and $^{40}$Ar/$^{39}$Ar methods are made on minerals such as leucite, alkali feldspar in both high- and low-temperature forms, muscovite, biotite, nepheline, amphibole, and plagioclase, listed approximately in order of decreasing potassium content. Mineral concentrates are obtained from the crushed sample by heavy liquid and magnetic methods at the coarsest grain size, consistent with obtaining material free of composites. In the case of tuffaceous samples, there is always the possibility of contamination of any mineral separated by xenocrysts or by detrital materials, although the single-crystal $^{40}$Ar/$^{39}$Ar dating technique enables these factors to be directly evaluated. For volcanic rocks, especially basalts and basaltic andesites, the use of whole rock samples for dating is very common; provided these are fresh and well crystallized they commonly yield what appear to be good ages, reflecting the time of their cooling to ambient temperature. In some cases, even fresh basaltic glass has yielded reliable ages (Renne et al., 1999).

### 14.1.4 Size Limitations

The ability to measure the argon composition isotopically depends upon a number of factors, including the inherent atmospheric argon amount in the sample, the argon blank from the extraction system, as well as the sensitivity and background in the mass spectrometer. Thus, when conventional K/Ar dating was the main approach used, the ion beams were often detected on a Faraday cup and measured using a feedback resistor of $1 \times 10^{11} \Omega$. An alkali feldspar from the Silbo Tuff in the Omo-Turkana Basin, northern Kenya, of age about 0.73 Ma, with 5.6 wt% K, has $^{40}$Ar* of about 7.4 atm. Omo-Turkana Basin, northern Kenya, of age about 0.73 Ma, with $^{40}$Ar* of about 7.4 atm.

Assuming a sensitivity of the mass spectrometer, a VG1200, of $10^{11}$ atm, an alkali feldspar from the Silbo Tuff in the Omo-Turkana Basin, northern Kenya, of age about 0.73 Ma, with 5.6 wt% K, has $^{40}$Ar* of about $7.4 \times 10^{-15}$ mol g$^{-1}$. Assuming a sensitivity of the mass spectrometer, a VG1200, of $10^{11}$ atm, an alkali feldspar from the Silbo Tuff in the Omo-Turkana Basin, northern Kenya, of age about 0.73 Ma, with 5.6 wt% K, has $^{40}$Ar* of about $7.4 \times 10^{-15}$ mol g$^{-1}$ using the Faraday detector. 50% of the beam radiogenic argon, and the blank and background argon as small to negligible, the beam of total $^{40}$Ar measured would be about 8.4 V for a sample of 2 g, with about 14 mV of $^{36}$Ar, utilized for the correction of atmospheric argon. These measurements were made with an overall precision of around 1% (McDougall, 1985). McDougall also reported a number of $^{40}$Ar/$^{39}$Ar step-heating experiments using a similar measurement technique, on bulk samples of alkali feldspar on the order of 1.0 g in size. The youngest of these was on an alkali feldspar from pumice in the Silbo Tuff, and with 15 temperature increments using a resistance furnace to release the argon, an age spectrum was produced that was essentially flat (Figure 4). The argon in 10 of the 15 steps exceeded 50% radiogenic, and gave an overall age with uncertainty of about ±2%. Note that the correlation diagram indicates a similar age and a trapped argon component that is indistinguishable from that in atmospheric argon (Figure 4). The disadvantage of using the multigrained approach is that the sample has to be entirely juvenile, with no xenocrystic or detrital grains present, in order to obtain the correct cooling age. However, this can readily be checked using the $^{40}$Ar/$^{39}$Ar single-crystal approach, with detection of the ion beams on a multiplier. Usually, the crystals are individually fused using a continuous wave laser. A 1 mg crystal of alkali feldspar from the Silbo Tuff pumice clast, about 0.73 Ma old and with 5.6% K content, contains about $7.4 \times 10^{-15}$ mol $^{40}$Ar*, which yields a beam of about 285 mV on the multiplier in a VG3600 mass spectrometer operated at about 100 times the sensitivity of the Faraday cup using a feedback resistor of $1 \times 10^{11} \Omega$ (McDougall and Brown, 2006), so the overall sensitivity was about $2.6 \times 10^{-17}$ mol mV$^{-1}$. For such a measurement, the amount of radiogenic argon was found to be variable from crystal to crystal, but usually in the range of 20% to over 80%, with atmospheric $^{36}$Ar between about 0.2 and 4.5 mV, which could be measured on the multiplier at an uncertainty of about 1–2% (McDougall and Brown, 2006). Taking an average of about 50% radiogenic argon, and a measured blank from the extraction system without the presence of a sample of about 2.5 ($±$~1) $\times 10^{-16}$ mol $^{40}$Ar, calculation indicates that the blank contribution makes up less than about 2% of the total $^{40}$Ar beam.

As a further example as to what can be achieved using the $^{40}$Ar/$^{39}$Ar total fusion technique, reference is made to results on alkali feldspars separated from small pumice clasts in the Aliyo Tuff of Member III of the Kibish Formation in southern Ethiopia and reported by McDougall et al. (2005, 2008). This unit is shown to have an age close to 100 ka from both single-crystal measurements and determinations on more than one crystal because of small crystal size. Even at this age, the alkali feldspars commonly have more than 30%, and up to over 70%, of radiogenic argon, but because of small sample size and the youthfulness of the eruption, the ages have very variable uncertainties (standard deviations) ranging from about 3% to as much as 15%. This clearly is a case where it is desirable to report the age as a weighted mean, with a standard error. Measurements were generally made on at least ten individual crystals or multiple crystals from individual pumice clasts, with near concordance of results from four pumice clasts, yielding an overall mean age of $103.7 \pm 0.9$ ka, as shown in a probability plot in Figure 5.

These examples indicate not only the ability to measure precise ages on appropriate igneous materials at quite youthful ages, but that it is not usually possible to state what the younger limit might be. The advent of mass spectrometers with multicollectors, using Faraday cups with feedback resistors of $1 \times 10^{12}$ or even $1 \times 10^{13} \Omega$, will mean that even more precise ages are likely to be determined in the future. And for very small or very young samples, detection of the ion beams on a single electron multiplier with ion counting will continue to make age determinations feasible, recognizing that the overall limitation is the detection of $^{40}$Ar* above the atmospheric argon component.
14.1.5 The Omo-Turkana Basin Sequence

An excellent example of what can be achieved by utilizing initially the K/Ar dating method, followed by the application of the $^{40}$Ar/$^{39}$Ar technique on multigrained samples, then on single crystals, is the continuing study of rocks from the Omo-Turkana Basin that were deposited over the last ~4.3 Ma during the Pliocene and Pleistocene (Figures 6 and 7). This stratigraphic sequence, shown to be up to 800 m thick in subaerial exposure, is of considerable significance in part because of the large number of hominin fossils (>400) that have been recovered over the last 4 decades mainly by the Leakeys when associated with the National Museums of Kenya (Feibel et al., 1989; Leakey and Leakey, 1978; Leakey et al., 1998; Wood, 1991) providing a remarkable picture of the complexity of the evolution of hominins. Specimens assigned to *Australopithecus anamensis*, *A. aethiopicus*, *Africanus*, *boisei*, *Kenya-thropus platyops*, *Homo habilis*, *H. rudolfensis*, *H. erectus*, and *H. sapiens* have been recorded (e.g., McDougall et al., 2012; Wood, 1991). Likewise, it should be emphasized that many other vertebrate fossils have been collected and described from which much information on the evolution of many genera has been obtained (Harris, 1983). Indeed, it has been the continuing new fossil finds that have driven much of the detailed stratigraphic and geochronological work over a number of decades. Clearly, a well-constrained stratigraphic and temporal framework is desirable in deciphering vertebrate evolution independent of the perceived evolutionary sequence.

The Omo-Turkana Basin in northern Kenya and southern Ethiopia comprises the depositional environment around and to the north of the present-day closed basin of Lake Turkana (Figure 7). Dating of tuffaceous products within the sedimentary sequences shows that deposition within the basin was initiated around 4.24 Ma ago (McDougall and Brown, 2008). The basin has been the focus of deposition since initiation. Much of the sedimentary material deposited in the basin was brought in by the Omo River, which drains part of the Ethiopian highlands. The basin extends at least 350 km N–S and up to 90 km E–W (Figure 7). It lies within the East African Rift system where the NNE–SSW trending Ethiopian Rift intersects the N–S trending Kenya Rift. Initially it was thought that the various regions mapped comprised separate depocenters, and this is reflected in the stratigraphic nomenclature. Thus, the Shungura Formation was defined and mapped north of Lake Turkana in southern Ethiopia (de Heinzelin, 1983), with the Koobi Fora Formation east of Lake Turkana (Brown and Feibel, 2000).
followed by the Nachukui Formation west of Lake Turkana (Harris et al., 1988); all are now collectively placed in the Omo Group of de Heinzelin (1983).

The detrital sediments themselves are not particularly useful for correlation as there are major facies variations from fluvial to deltaic and lacustrine over quite short distances. In contrast, tephra or tuffaceous sediments, with their distinctive elemental signatures of the contained volcanic glass, have been the key to stratigraphical correlations from area to area. These tephra and tuffaceous sediments have been mapped and named as tuffs. Chemical analysis of glass shards in these tuffs has shown that some are widespread (Brown et al., 2006). As many of the tuffs have been deposited by water, it has become increasingly obvious that the sediments of the Omo-Turkana Basin are all part of a single depositional system. Thus, through successive improvements in correlation of tuffs within the basin, a robust stratigraphic framework has been developed (cf. Brown et al., 2006, and references therein). Some tuffs locally contain pumice clasts, which usually have glass of the same composition as the enclosing tuff, indicating derivation from the same volcanic eruption. The pumice clasts commonly have alkali feldspar (anorthoclase) phenocrysts, which are ideal for isotopic dating by the K/Ar method and by the $^{40}$Ar/$^{39}$Ar dating technique. Thus, not only have the tuffs facilitated correlations throughout the basin, but some have been isotopically dated, from which a numerical time framework has been developed for the basinal sequences. With concerted efforts over the last 3 decades, more than 35 units in the Omo-Turkana Basin have been dated, with most now having been measured using single-crystal $^{40}$Ar/$^{39}$Ar total fusion techniques (McDougall and Brown, 2006, 2008; McDougall et al., 2012). Virtually all these ages are consistent with the stratigraphic order, not only indicating that the ages record the time of eruption, but also that deposition occurred very shortly after eruption. The isotopic dating results already available mean that fossils can usually be assigned ages to better than 100 ka, without further direct dating, provided that the fossils can be placed within the stratigraphic sequence of tuffs. However, in restricted parts of the section, gaps still exceed 400 ka between dated tuffs, so that in some cases, considerable interpolation is required to assign an age to an individual fossil, or to derive an age for a particular stratigraphic level.

In the initial stages, starting in 1978, alkali feldspar crystals were separated from pumice clasts in a number of the tuffaceous beds and K/Ar ages were measured on multigrained samples (McDougall, 1985; McDougall et al., 1980) using between 0.5 and 2 g of material for each argon extraction, subsequently followed by $^{40}$Ar/$^{39}$Ar step heats on selected multigrained samples (McDougall, 1981, 1985), and then by...
multiple single-crystal \(^{40}\text{Ar}/^{39}\text{Ar}\) total fusion analyses (McDougall and Brown, 2006, 2008; McDougall et al., 2012). A summary of these results for the composite stratigraphic column for the Koobi Fora Formation is given in Figure 8, together with a K/Ar whole rock age on basalt at the base of the section. The fluence monitor used in the step-heating experiments was GA1550 biotite with a K/Ar age of 97.9 Ma (McDougall and Roksandic, 1974; Steiger and Jäger, 1977), although with a revised K content, the K/Ar age is now given as 98.5 Ma (McDougall and Wellman, 2011). The step-heating experiments all yielded essentially flat age spectra, providing increased confidence that the derived ages are those of the igneous eruption, and have not been subsequently disturbed (Figure 4). The single-crystal \(^{40}\text{Ar}/^{39}\text{Ar}\) ages are reported versus the Fish Canyon Tuff sanidine fluence monitor age of 28.1 Ma (Spell and McDougall, 2003). The diagram shown in Figure 8 highlights the remarkably good agreement between the various measurements on the volcanic-derived tuffs, and also the concordancy of the results with the stratigraphy, younging upward, and additionally shows several of the more important hominin finds in their approximate stratigraphic positions within the sequence. The concordancy of the ages with the stratigraphy provides great confidence in their veracity, not only giving precise estimates of the age of the volcanic eruptions that produced the tuffs and enclosed pumice clasts, but also providing confidence that the deposition age of the tuffs is very soon after the explosive eruptions that produced them. It is by interpolation between the dated
tuffs and the stratigraphic position of the fossils that an age is derived for the individual fossils. As already mentioned, in general, ages can be assigned to better than 100 ka for fossils on the basis of these interpolations.

Figure 9 provides an overall summary for much of the Omo-Turkana Basin stratigraphy and the ages that have been measured, mainly by the single-crystal technique, now on over 35 different volcanic levels. In all these cases, the Fish Canyon Tuff sanidine with assigned reference age of 28.1 Ma (Spell and McDougall, 2003) was utilized as the fluence monitor (McDougall and Brown, 2006, 2008; McDougall et al., 2012). This figure also shows the links between the various identified formations based upon correlations using the distinctive chemical compositions of the volcanic glass comprising the bulk of the tuffs (Brown et al., 2006). As previously discussed, it is these correlations that resulted in the recognition that the Omo-Turkana Basin was a single depositional basin, despite the very large facies changes that can occur over quite short distances. In addition, some ages also can be derived from paleomagnetic polarity boundaries, and a few
Figure 9  Composite stratigraphic sections of the main formations in the Omo-Turkana Basin, together with the results of \(^{40}\)Ar/\(^{39}\)Ar dating of mainly alkali feldspar from pumice clasts within tuffs, where the quoted error is the standard deviation of the population (McDougall and Brown, 2006, 2008; McDougall et al., 2012). The main tuffaceous beds are shown, often named, and correlations between the stratigraphic columns are indicated by linking lines. To the left of the stratigraphic columns for the Koobi Fora and Shungura formations are the measured magnetic polarities, where gray shading represents normal polarity and unshaded indicates reversed polarity. Ages assigned to the polarity boundaries are from Gradstein et al. (2004). For the dating, the Fish Canyon Tuff sanidine fluence monitor was assigned a reference age of 28.1 Ma (Spell and McDougall, 2003). Diagram courtesy of F. H. Brown, University of Utah.
ages are imported from other regions. The bigger picture, incorporating the whole basin, shows that the ages are fully consistent with the stratigraphy, giving increasingly great confidence in the ages. The ages recorded at Kanapoi, southwest of Lake Turkana, are on alkali feldspar separated from weathered pumice clasts in mudstones for the earliest two beds, whereas the younger age is on alkali feldspar from small pumice clasts in the Kanapoi Tuff, higher in the sequence. These ages were initially reported by Leakey et al. (1995, 1998) and were recalculated by McDougall and Brown (2008) using the slightly greater age of 28.1 Ma for the Fish Canyon sanidine fluence monitor. The significance of these rather precise ages is that many fossil hominins assigned to *Australopithecus anamensis* were found in this stratigraphic section, mainly below the Kanapoi Tuff, so their age is very tightly controlled between 4.2 and 4.1 Ma. However, Leakey et al. (1995, 1998) also included hominin fossils in this species from Allia Bay, on the east side of Lake Turkana, where they are shown to be just below the Moiti Tuff, which has been dated at 3.97 ± 0.03 Ma, thus extending the age range for *A. anamensis* significantly.

Noteworthy is that within the individual stratigraphic sequences, not all tuffs are present, and in some cases, there are significant breaks in deposition that are evident. The most conspicuous of the latter is that so far there are no recorded sediments in the Omo-Turkana Basin between the ages of about 0.7 and 0.2 Ma. It is likely that there is a record of deposition under Lake Turkana itself, but no deposition is known subaerially. Similarly, in the Koobi Fora Formation, it has long been noted that there is an absence of sedimentary strata in an interval of perhaps 0.5 Ma between the lower and upper Burgi Member, and yet sediments are known from this interval in the Nachukui Formation and the Shungura Formation.

About 100 km to the north of Lake Turkana, the Kibish Formation was deposited between about 200 ka and nearly the present time (Brown and Fuller, 2008; McDougall et al., 2005, 2008). Four members were defined in the Kibish Formation, each reflecting a very high stand of Lake Turkana at the time of deposition, with Members I to III showing evidence of having been deposited in deltaic environments, whereas the youngest member, Member IV, is thought to have been formed as a lake margin deposit, during the Holocene, equivalent to the Galana Boi Formation recognized further south around the present Lake Turkana. Each of the members is thought to have been deposited over very short intervals, with much of the time represented by the disconformities usually evident between members. From Member I, several hominin fossils were recovered in the original expedition to the region (Leakey, 1969), and Day (1969) described two of these crania as belonging to somewhat archaic *H. sapiens*. The more recent expeditions to the region confirmed the provenance of these fossils, and also were able to use 40Ar/39Ar date alkali feldspar from the Nakaa’kire Tuff from just below the level of Omo II, one of the fossil hominins. The age of the tuff was determined to be 198 ± 14 ka, where the quoted error is the standard deviation of the population, or 196 ± 2 ka if a weighted mean age is preferred. It was argued by McDougall et al. (2005, 2008) that this age was very close to that of hominin Omo II, because of the deduced rapid deposition of each member, and because there was a quite unexpected correlation with the deposition of sapropel 7 in the Mediterranean Sea, some 3000 km to the northwest. The link was that high lake levels required for deposition of the members of the Kibish Formation as delta deposits of the earlier Omo River, some 100 km north of the present Lake Turkana, were brought about by very high rainfall in the Ethiopian Highlands, the source of the Omo River, and the Blue Nile, and this resulted in the overflow of Lake Turkana across the divide into the White Nile. These very large flows of the Nile River system were implicated in the formation of the sapropels in the Mediterranean Sea (Rossignol-Strick et al., 1982). In Member III of the Kibish Formation occurs the Aliyo Tuff, from which small pumice clasts yielded alkali feldspar crystals that were also dated, yielding a weighted mean age of 104 ± 1 ka (Figure 5). This age is consistent with its stratigraphic position, and also shows that the hominins Omo I and II are older. Although Millard (2008) argued that the age on these hominins is only limited to between about 190 and 100 ka, the approximate isotopic ages on tuffs in Members I and III, respectively, we argue on the basis of the stratigraphy and the presence of major disconformities that the age of the *H. sapiens* fossils at Kibish is very close to the age of the Nakaa’kire Tuff of Member I, which is more like 195 ka, yielding the oldest currently known age for *H. sapiens*.

### 14.1.6 Results from Afar, Ethiopia

Studies in the Afar region of Ethiopia, east of Addis Ababa, have been of considerable significance and most helpful in providing age constraints on the many hominin fossils recovered from these sequences that range in age from Miocene to Pleistocene. The sequences have produced a prolific vertebrate fauna, including fossils assigned to a number of hominins; these include *Ardipithecus ramidus*, *Australopithecus afarensis*, *Homo sapiens*, among others. The sedimentary rocks occur over a wide region, with geological and paleontological studies concentrated in particular areas.

An older sequence of considerable relevance is that described by Renne et al. (1999) comprising the Miocene–Pliocene Sagantole Formation, in which many vertebrate fossils, including the early hominin *Ardipithecus ramidus*, occur (White et al., 1994, 1995). The Renne et al. (1999) paper is an update on the stratigraphy and geochronology of the area, following the earlier studies by WoldeGabriel et al. (1994, 1995). The Sagantole Formation consists of nearly 300 m of sedimentary rocks, mapped as seven members, variously comprising sands, silts, and clays, together with basalt lavas in the lower part of the sequence and many basaltic and more silicic tuffs throughout the sequence. Twelve intercalated volcanic rock units have been dated utilizing the 40Ar/39Ar technique, either as step-heating experiments or total fusion analyses. All the ages were reported relative to a Fish Canyon sanidine reference age of 27.84 Ma for the fluence monitor, and the errors are weighted standard deviations of the mean. Paleomagnetic measurements were also made on the sequence, enabling unequivocal links to the geomagnetic polarity time scale boundaries in the Gilbert Chron, so that seven ages were derived from this additional information. Thus, there are about 19 levels in the Sagantole Formation for which direct or indirect ages are available, making the sequence one of the best dated in East Africa.
Africa. Of major significance is that the direct dating of 12 units, using a wide variety of samples, shows that the sequence was deposited between about 5.6 and 3.9 Ma, noting that all the measured ages are consistent with the stratigraphy. This is all the more remarkable as the ages were determined on a variety of samples including whole rock basalts, glass lapilli from basaltic tufts, plagioclase from several tufts, and sanidine separated from a number of more silicic tufts. In the latter cases, ages were measured using total fusion of single crystals, commonly revealing the presence of much older xenocrystic crystals, often about 23–24 Ma old. This further demonstrated the enormous advantage of the single-crystal approach, with the juvenile or eruptive age of the unit being recognized from the youngest peak of the probability plot. That all these samples yielded such consistent results, with the whole rock basalts, the glass lapilli, and the plagioclase yielding flat age spectra, provides great confidence in the veracity of the results.

Comparison between the directly determined \(^{40}\text{Ar}/^{39}\text{Ar}\) ages and ages determined from the geomagnetic polarity time scale indicates further consistency, particularly when errors are taken into account. The fossils of \(A. \text{ramidis}\) were derived from the Aramis Member of the Sagantole Formation in the narrow interval of \(\sim 4\) m between the Ga`ala Tuff and the Daam Aatu Basaltic Tuff (WoldeGabriel et al., 1994). Renne et al. (1999) reported an age for sanidine of \(4.39 \pm 0.03\) Ma for the Ga`ala Tuff and an age of \(4.39 \pm 0.05\) Ma from a flat age spectrum on lapilli glass from the overlying Daam Aatu Basaltic Tuff. This excellent agreement means that the age control on \(Ardipithecus ramidus\) is among the best determined anywhere, and indicates that this species predates the fossils of \(Australopithecus anamensis\) found and dated mainly from Kanapoi, southwest of Lake Turkana.

About 50 km to the north of the Middle Awash area discussed above, within the Afar Depression, lies the area known as Hadar, the site from which were recovered many of the specimens used to describe and define \(Australopithecus afarensis\). Within the Hadar Formation, fossils of this hominin come from a minimum of 35 individuals and include a famous partial skeleton (A. L. 288-1) (Johanson and White, 1979; Johanson et al., 1978, 1982). The sedimentary sequence comprising the Hadar Formation is up to 270 m thick and consists of near flat-lying beds of pebbly gravel, sand, silt, and clay, with some interbedded basalts and tuffaceous beds (Taieb and Tiercelin, 1979; Taieb et al., 1976). Near the base of the sequence is the Sidi Hakoma Tuff, shown to be correlated with the Tulu Bor Tuff of the Omo-Turkana Basin (Brown, 1982; Sarna-Wojcicki et al., 1985), and dated at \(\sim 3.4\) Ma (McDougall and Brown, 2008; Walter and Aronson, 1993). There have been considerable difficulties in dating the younger part of the Hadar Formation, but it now appears that the Kadada Moumou Basalt in the upper part of the Sidi Hakoma Member of the Hadar Formation has an age of \(3.28 \pm 0.04\) Ma from whole rock \(^{40}\text{Ar}/^{39}\text{Ar}\) step-heating experiments (Renne et al., 1993), consistent with the slightly older age of the Sidi Hakoma Tuff. Single-crystal alkali feldspar measurements by the \(^{40}\text{Ar}/^{39}\text{Ar}\) technique, reported by Walter (1994), showed that the Triple Tuff 4, stratigraphically above the Kadada Moumou Basalt and near the base of the Denen Dora Member, has an age of \(3.22 \pm 0.01\) Ma, using Fish Canyon Tuff sanidine as the fluence monitor with reference age of \(27.84\) Ma, where the error quoted is the standard deviation of the mean. Similar \(^{40}\text{Ar}/^{39}\text{Ar}\) ages on alkali feldspar crystals from the Kada Hadar Tuff, the defined base of the overlying Kada Hadar Member, yielded an age of \(3.175 \pm 0.012\) Ma (Walter, 1994). An air-fall tuff, one of the Bouroukie tufts, BKT-2, lies in the upper part of the Kada Hadar Member and contains fairly low K (\(\sim 1.6\%\)) anorthoclase crystals. Walter and Aronson (1982) reported \(K/Ar\) ages on anorthoclase separates from this tuff with a best estimate of \(2.88 \pm 0.08\) Ma. Subsequently, DiMaggio et al. (2008) reported nearly flat \(^{40}\text{Ar}/^{39}\text{Ar}\) age spectra for anorthoclase separated from tufts BKT-2L and U. Average plateau ages of \(2.963 \pm 0.017\) and \(2.970 \pm 0.026\) Ma, respectively, are calculated using \(28.01\) Ma as the reference age for Fish Canyon Tuff sanidine. These ages are more precise than the \(K/Ar\) ages reported previously and are thus preferred. Note that Brown et al. (2012) in their review of the age ranges for \(Australopithecus\) species have recalculated all these ages to a common reference age of \(28.1\) Ma reference age for the Fish Canyon Tuff sanidine fluence monitor. Ages are fully consistent with the stratigraphy, giving close control on the ages of vertebrate fossils in the sequence. As shown by Johanson et al. (1982) and Walter and Aronson (1993), hominin fossils were recovered mainly from the Sidi Hakoma and Denen Dora members of the Hadar Formation. Thus, the \(A. afarensis\) fossils in the Hadar Formation are constrained to lie between the ages of \(\sim 3.1\) and \(3.4\) Ma. The definition of \(A. afarensis\) also includes specimens from Laetoli in Tanzania from the Upper Laetolil Beds that have been securely dated by \(^{40}\text{Ar}/^{39}\text{Ar}\) measurements on alkali feldspar and biotite from contemporaneous tufts by Deino (2011). His measurements show that the \(A. afarensis\) fossils derive from beds that are between \(3.63\) and \(3.85\) Ma old, and are thus older than the Hadar Formation in Ethiopia. On this basis, \(A. afarensis\) has age limits from \(3.85\) to \(\sim 3.1\) Ma, and would appear to be slightly younger than the \(A. anamensis\) fossils from Kanapoi in the Omo-Turkana Basin. This is in keeping with the view that \(A. anamensis\) could well have been ancestral to \(A. afarensis\) as suggested by Leakey et al. (1995, 1998), White et al. (2006), and Kimbel et al. (2006). Although this \(0.7\) Ma of existence of \(A. afarensis\) seems long for a hominin species, it should be pointed out that a further hominin has been described from this temporal interval. Thus, \(Kenyanthropus platyops\), documented by Leakey et al. (2001) from the Kataboi Member of the Nachukui Formation of the Omo Group in the Omo-Turkana Basin, has an intercalated age of about \(3.5\) Ma. It is inferred from the fossil assemblage in this part of the Nachukui Formation that in fact multiple hominin species existed between \(3.5\) and \(3.0\) Ma.

### 14.1.7 Conclusions

In this contribution, a review of the place of \(K/Ar\) and \(^{40}\text{Ar}/^{39}\text{Ar}\) isotopic dating techniques as applied to young volcanic rocks and related products, and how these results have provided well-constrained ages for a wide range of hominin fossils, especially those recovered from sites in East Africa. In the majority of cases, this has enabled workers to place hominin fossils in an independent time scale, unrelated to the perceived evolutionary stage exhibited by the fossils. It is hoped that the explanations given are clear, and that this chapter also shows why these dating techniques have become...
so important in dating hominin fossils. It has been stressed that most numerical ages assigned to hominins and other vertebrates are from interpolation between dated igneous-derived samples, rather than by directly dating the fossils themselves. In earlier times, K/Ar and indeed even 40Ar/39Ar ages were often the subject of dispute, but, especially with continued development of the latter technique, the results generally have become well accepted by the geological and paleoanthropological community.

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