DATING BASIC ROCKS FOR USE IN
PALAEOMAGNETISM

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ABSTRACT

Potassium-argon age measurements on young rock samples depend on the accurate measurement of the extremely small volumes of argon-40 that are trapped in the rock. In this dissertation, the construction of an Omegatron, a mass spectrometer of high sensitivity and low background noise, is described. It is shown to be ideally suited for this work, the characteristics and properties being discussed in relation to those of a theoretical model.

An account is given of the construction of an all-metal argon line for the purification of the argon evolved from the rock, and the methods of measurement of the gas sample using the Omegatron are discussed. Results of age measurements on basic rocks previously used in Palaeomagnetism are compared to the ages expected on geological evidence and from an examination of their thin sections.

From Palaeomagnetic studies on rocks from Ethiopia, no significant movement of the continent of Africa was found to have taken place since the Upper Eocene. Some rocks from the Sudan with anomalous directions of magnetisation are described. These rocks have a large reversed component which is removed on heating, and one particular sample reversed completely.
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CHAPTER I
INTRODUCTION

A rock on cooling from above the Curie point of its magnetic constituents will in general acquire a direction of magnetisation parallel to that of the ambient geomagnetic field at the time of cooling. Palaeomagnetism is concerned with the study of this remanent magnetisation retained in the rock and the past history of the earth's magnetic field.

With the development by Blackett\(^1\) of an astatic magnetometer of high sensitivity and the improvement of the spinner type first constructed by Johnson and McNish\(^2\) interest in this subject has grown enormously in the last decade. The direction of magnetism of many thousands of rocks of differing ages and location have now been determined. Basaltic lavas are perhaps the best rocks for this study as they have a strong permanent magnetisation (intensity of \(10^{-2}\) to \(10^{-3}\) electromagnetic units) and have been proved to have a high degree of magnetic stability and consistent direction of magnetisation within one continent in any one geological period. Red sandstone with less strong magnetisation (\(10^{-5}\) to \(10^{-7}\) electromagnetic units) have also been used successfully.
In investigations of recent lava flows by Roche\(^{(3)}\) and Hospers\(^{(4)}\) it was found that the flows were magnetised on the average not along the present earth's field but in the direction of the theoretical dipole field, which is that due to a supposed magnetic dipole at the centre of the earth and directed along its rotational axis. The secular variation about the direction of the theoretical dipole field was assumed to have been smoothed out.

However, when the direction of magnetisation of older rocks is determined two distinct phenomena are observed. The first is that many rocks have directions of magnetisation which are widely oblique to the direction of the earth's theoretical field and as the formation of these rocks took the best part of a geological period, the cause of this oblique magnetisation is certainly not the secular variation of the earth's field. The second phenomenon is that approximately half the rocks have a direction of magnetisation which is opposed to the present direction of the earth's field and are termed reversed rocks.

The phenomenon of this widely divergent direction of magnetisation from the present earth's field has been ascribed to a drift of the continents relative to each other. This theory of Continental Drift was originally proposed by Wegener who noticed the similarity of the
West Coast of Africa and the East Coast of South America. The chief difficulty has been in providing an adequate physical process which can bring about these wholesale tangential movements of large pieces of the earth's crust. The current theory to account for this was originally postulated by Vening Meinesz\(^{(5)}\) and more recently discussed by Runcorn\(^{(6)}\) and supposes the idea of convection currents in the earth's mantle.

From a study of the direction of magnetisation of rocks of different ages it is possible to trace the movement of a particular continent over the surface of the earth. Unfortunately basalts which are the most suited for this work are scarce in fossils and consequently the determination of the geological age to any accuracy very much shorter than a geological period has until recently been rather difficult. For a particular continent a mean rate of change in the direction of magnetisation of about one-third of a degree per million years is indicated; hence it is possible that there could be discrepancies of up to 20 or 30 degrees in the palaeomagnetic direction of rocks from a geological period.

The fact that about 50\% of all rocks are found to be reversely magnetised leads to the idea that perhaps the earth's field reverses periodically. Matuyama\(^{(7)}\) was the
first to suggest this and for many years his idea was accepted. Graham(3), however, in examining some sedimentary rocks which had normal and reversed magnetisation came to the conclusion that a more likely explanation could be that of a self-reversing mechanism and asked Neel to consider this possibility. Neel(9) proposed a number of physical methods by which rocks might become magnetised in the direction opposite to that of the earth's field in which they were cooled. Since then Nagata(10), Carmichael(11) and myself have found rocks which self-reverse in the laboratory and many other natural and synthetic samples have been found with self-reversing mechanisms.

Recently Ade-Hall(12) has found a marked petrological correlation between the normal and reversed lavas of Mull. Unexsolved homogeneous opaque grains are related to the normal lavas and exsolved ilmenite is found to be related to the reversed. Field reversal could have occurred during local reheatings causing exsolution of the ilmenite and a sort of "exsolution remagnetisation" along this reversed direction. Alternatively, self-reversal in a normal field could have been a result of the process of exsolution. Wilson(13) in studying the general magnetic properties of these lavas failed to distinguish between the two petrologically distinct groups.
Perhaps the most convincing evidence for field reversal has been found in examining the directions of magnetisation of baked rocks and the baking igneous body. A rock which has been previously magnetised, if heated above its Curie point, will on cooling become magnetised in the direction of the field acting at that time. Consequently, if self-reversing mechanisms are rare, one would expect that the heating body and the country rock at the point of contact would have the same direction of magnetisation. Wilson\(^{14}\) collected information relating to 52 baked contacts sampled throughout the world and of widely differing ages. He found that of these, 49 showed agreement in the direction of magnetisation, whereas only 3 showed disagreement. This evidence indicates that the occurrence of self-reversing mechanisms in the magnetic materials in rocks is a relatively rare phenomenon.

The only conclusive way to resolve the problem of the reversal of the earth's field is by accurate age determinations on rocks of known polarity throughout the world. This can only be carried out successfully if the fraction of self-reversing rocks is so small that their numbers can be statistically ignored. We can never be sure that any rock is not self-reversing, for the physical investigation may not have been thorough enough to reveal it.
Various estimates have been made for the frequency of this polarity change but large variation can be found. For instance, the duration of the Permian period is predominantly reversed and the Tertiary basalt lavas of Northern Ireland indicate a long period of reversal. Hospers (15) found that in a sequence of recent lavas in Iceland polarity zones occur and it appears that in the Cainozoic reversals are frequent.

Since the development by Reynolds (16) of a mass spectrometer of high sensitivity and low background noise it has become possible to date these recent polarity epochs using the potassium argon method. From work done by McDougall and Tarling (17, 18) on recent lava flows from Hawaii and by Cox et al (19) on the volcanic plugs and flows from Sierra Nevada it seems likely that the most recent reversal of the earth's field occurred around 1 million years. The length of this reversed epoch is at present uncertain owing to the small number of age measurements made.

From the previous discussion it will have become apparent that accurate age determinations are an essential part of any palaeomagnetic study. The potassium-argon age determination/depends on the measurement of argon-40 evolved from the decay of potassium-40 and which is retained in the rock. For very young rocks this volume of radiogenic
argon-40 is extremely small and consequently a very highly sensitive mass spectrometer is needed for its measurement. This thesis is concerned with the construction of a mass spectrometer, known as an Omegatron, which works on the cyclotron principle and is extremely suited for this study.
CHAPTER 2

THE OMEGATRON

2.1. Principle of the Omegatron

The Omegatron device was first described by Sommer et al. (20) and later modified by Alpert and Buritz (21) in 1951 to give a simpler design. Several other modifications have been described e.g. Brubaker and Perkins (22) in which the sensitivity is increased by suitable electrostatic and magnetic fields.

Ions formed by electron bombardment in a magnetic field will describe circles whose natural frequency of revolution is given by the cyclotron frequency. A radio-frequency field applied perpendicular to the magnetic field will accelerate ions with the same frequency of oscillation and their path will become an Archimedes spiral. These resonant ions can be trapped by a suitably placed collector and detected by a sensitive amplifier. Non resonant ions describe spiral paths with pulsing radii and do not reach the collector.

2.2. Theory

The theory of an idealized omegatron (neglecting electrostatic fields and space charge effects) was formulated by Sommer et al. (20) who gave the expression for the resolving power, time of flight, path length and final energy of the
ions. Berry\(^{(23)}\) derived and discussed the equation of motion of the particles in a two dimensional system.

A uniform magnetic field along the z-axis and a uniform alternating electric field with positive direction along the positive x-axis will be assumed. This implies zero forces in the z direction so that only motions in the x-y plane need be considered.

The following symbols are used, the M.K.S. system of units being used throughout.

- \(B\) = the magnetic field strength
- \(\omega_0\) = the angular frequency of the applied electric field \(E\sin\omega_0 t\)
- \(E\) = the peak amplitude of the electric field
- \(M\) = the mass of the ion
- \(e\) = the electronic charge
- \(u, v\) are the initial velocities of the particle in the x and y direction respectively which can be represented by the vector \(V = u + iv\)
- \(\phi\) = the phase angle relating the time of origin of the ion to the phase of the applied electric field.
- \(\omega = \frac{eB}{M}\) = the cyclotron frequency \(2.1\)
The differential equations of motion in the x-y plane of a charged particle are

\[ Mx = eE \sin \omega_0 t + eBy \]

and \[ My = -eBx \]

the dots represent time derivatives.

If we substitute \( a = \frac{eE}{M} \) & \( \omega \) the cyclotron frequency, these equations become

\[ x = a \sin \omega_0 t + \omega y \]

\[ y = -\omega x \]

The solutions are considerably simplified using complex notation & for the initial condition \( x = y = 0, x = u, y = v \) for \( \omega_0 t = \varphi \) the complete solution is

\[
x + iy = i \frac{V}{\omega} \left[ e^{-i \omega (t - \varphi/\omega)} - 1 \right] - i \frac{a}{\omega^2 - \omega^2} \left[ \frac{\omega}{\omega_0} \cos \omega_0 t - i \sin \omega_0 t \right]
+ i \frac{a}{\omega_0^2 - \omega^2} \left[ \frac{\omega_0}{\omega_0} \cos \varphi - i \sin \varphi \right] e^{-i \omega (t - \varphi/\omega_0)} - i \frac{a}{\omega_0} \cos \varphi
\]

(2.2)

The first term of this equation represents a circle of radius \( \frac{|V|}{\omega} \). The second term represents an elliptical motion with angular frequency \( \omega_0 \) which is independent of the phase angle. The third term represents a circular motion with angular frequency \( \omega \), the amplitude of and phase of which depend on the relative magnitudes of \( \omega, \omega_0 \) & \( \varphi \). The fourth term is simply a static shift along the y-axis of the centre of rotation.

At resonance, \( \omega = \omega_0 \) and to find the path of a resonant particle we must find the limit of equation (2.2) as \( \omega_0 \to \omega \)
This will also give us the expression for the path of a particle near resonance.

Letting $\epsilon = \omega - \omega_0$ be the difference frequency and re-arranging equation (2.2)

$$x + iy = \left[ iV_{\omega_0} + \epsilon \right] \left[ e^{-i(\omega_0 + \epsilon)(t - \varphi/\omega_0)} - 1 \right]$$

$$+ \frac{i \alpha}{2\omega_0 + \epsilon} \left[ 1 - \left( 1 - \frac{\epsilon e^{i\varphi}}{\omega_0 + \epsilon} \right) e^{i\epsilon(t - \varphi/\omega_0)} \right] e^{-i\omega_0 t}$$

$$+ \left( \epsilon/\omega_0 \right) (\cos \omega_0 t) - \left[ (2\omega_0 + \epsilon) \epsilon/(\omega_0 + \epsilon) \omega_0 \right] (\cos \varphi) \quad (2.3)$$

By expanding $\exp[-i\epsilon(t - \varphi/\omega_0)]$ in a power series in $\epsilon$ the equation for a resonant particle is found to be

$$\left[ x + iy \right]_{\omega = \omega_0} = \left( iV_{\omega_0} \right) (e^{-i\omega_0(t - \varphi/\omega_0)} - 1)$$

$$- \left[ \frac{i \alpha}{2\omega_0^2} \right] \left[ \omega_0 t - \varphi - ie^{i\varphi}\omega_0 \right] e^{-i\omega_0 t}$$

$$+ \left( \frac{i \alpha}{2\omega_0^2} \right) (\cos \omega_0 t) - \left( \frac{i \alpha}{\omega_0^2} \right) (\cos \varphi)$$

The first and last terms are identical to those in equation (2.2). The second term represents a spiral motion due to the appearance of $t$ in the amplitude of the exponential. After a few cycles the other terms in the brackets of the second term become negligible compared to $\omega_0 t$.

The third term is simply a small sinusoidal oscillation of the centre of rotation along the $y$-axis; after a few cycles these terms become negligible also. Neglecting the initial velocities, the motion of a particle in resonance closely approaches an Archimedes spiral.
2.3. **Construction of the head**

The head used is one designed by Philips for use in measuring partial pressures. When adjusted properly it gives a constant sensitivity and a complete collection of the resonant ions is achieved. In an idealised omegatron no electric fields exist and the collection is complete, but in a real omegatron there are always fields present which can cause some of the resonant ions to be lost.

These fields are caused by

(a) Space charge effects due to the electron beam and the ions present.

(b) The electrodes of the omegatron which can become charged.

(c) Leakage fields from the accelerating plates.

(d) The small positive field which must be applied to prevent the drift of ions in the direction of the magnetic field.

These fields can be of the same order of magnitude as the applied electric field and hence large variations in the ion collection can result.

The Philips head is identical to that designed by Sommer et al (20) when viewed in a direction perpendicular to the magnetic field (Fig. 2.1), but in addition it has two side plates CC' to which a large negative voltage can be applied. This negative voltage extracts non-resonant
ions, reducing the space charge effect, and can be adjusted to obtain a maximum ion current and give the tube a reproducible sensitivity. The value of this voltage depends on the number of guard rings and the positive voltage \( V_R \) which prevents the drift of ions in the direction of the magnetic field.

The electrode assembly forms a cube of side about 2.5 cm. and is arranged with the electrodes as in Fig. 2.1. It can be degassed to 400°C and the metal electrodes to 900°C by induction heating. In actual practice a temperature of 200°C is found quite sufficient to give pressures of \( 1-2 \times 10^{10} \) mm of mercury. A platinum iridium alloy is used to prevent any oxide layer forming and the directly heated cathode is barium oxide coated tungsten to minimise the rates of reaction with any reactive gases which may be present.

2.4. **Components**

2.4.(a) **The Oscillator**

The TF 1370 Marconi R-C oscillator supplies the radio frequency field and covers the range 10 cycles per second to 10 megacycles per second in six decade bands. Square waves are available but are not needed.

The sine wave output has a range of 1mV to 3.16 volts via an attenuator switch and a direct range of up to 31.6
volts at frequencies up to 100 Kc/s across loads of 2 kilohms or greater. The zero attenuated output is used which in practice gives a range of 0-1.8 volts R.m.s. value. After an hours warm up, the frequency is stable to ±0.5% up to 10 Mc/s and is free of harmonics which is essential to avoid the collection of resonant ions with that harmonic frequency.

2.4.(b) Magnet supply

A Newport instrument 7 inch electro-magnet Type E is used with plane pole-pieces. The pole gap can be adjusted over the range zero to six inches but was set at 43 mm as this gave a reasonable clearance for adjusting the line up of the magnet, while providing a sufficiently high field for good resolution of the peaks. A C224 power supply with a maximum power rating of 6K.watts provides the power for the magnet and has a stability of 1 in 10^4 for one hour.

The magnet supply is stabilised in two stages:
(a) A special 3 phase motor metadyne generator converts the mains supply into a controllable, very low ripple d.c. supply. The motor is insensitive to variations of mains voltage and its inertia makes it immune to fast transients
(b) A feedback loop controls the output voltage of the generator so that the voltage developed by the magnet current on a standard resistance is constant, against a reference voltage derived from mercury cells. There is
a coarse, numbered control and a fine current control. An adjustable sweep unit is incorporated with the controls but as the omegatron is run 'statically' this is not used.

The strength of the magnetic field was measured using a Hall effect probe for different values of the magnet current. A field of just over 10 kilo-gauss could be achieved when the magnet current was at its maximum value of 26 amps. (Fig. 2.2). The homogeneity of the field was found to be better than 1 part in 5000 at a field of 5 kilo-gauss up to a distance of 4 cm. from the centre (Fig. 2.3). The pole pieces were supplied with adjustable shims but there appeared in this instance to be no advantage in using them as neither the homogeneity nor the absolute field attainable were altered appreciably. It was also easier to remove the connections to the head when the shims were retracted.

The magnet itself was mounted on a steel trolley which could be racked away from the head and clamped in any position. Facilities were available for adjusting the magnet in two directions at right angles.

2.4.(c) The current amplifier

The current from the ion collector is measured using a vibrating Reed amplifier - the 51A Vibron Mass Spectrometer amplifier. It will discriminate currents as low as
10^{-10} amperes quickly and reliably against a random noise of 5 \times 10^{-17} amperes.

The overall gain between 1 and 11,000 is useful in scanning the mass spectrum to equalise the heights of successive pulses and hence obtain a much more accurate determination of the relative abundance. A 10^{10} ohm input resistor makes currents of 10^{-14} amperes easily discernable and was sufficient for our present requirements. A 10^{12} ohm input resistor can be used which will increase the sensitivity by a factor of a hundred.

Using an extended gain control consisting of precision decade resistances, the sensitivity can be continuously varied over four decades and with this arrangement gain ratios of 1000 to 1 can be measured to an accuracy of better than 1%. Two variable time constants are incorporated, the adjustment of which permit a compromise between the conflicting requirements of fast response, low noise level and the absence of overshooting on the meter or the recorder. An input capacitance control is provided which is continuously variable over the range 1 -10\text{pF}. Coarse adjustment is effected by fitting a suitable capacitor in the compartment of the head unit. An output time constant at the end of the first amplifying stage is variable from a nominal 0.5 to 60 seconds. For beam currents greater than 10^{-12}
amperes the amplifier has a 10% to 90% response time of 100 m.sec. but increases to about 5 secs. with an input current of $5 \times 10^{-17}$ amperes.

The vibron head is mounted horizontally on the welded steel frame supporting the magnet. A copper tube about 1 ft. long with a brass flange and 'O' ring provides an airtight extension to the vibron case and enables the magnet to be racked away from the omegatron without removing the vibron. The vibron casing and extension tube are connected to the backing pump of the argon line to reduce the random noise level as much as possible.

2.4.(d) The Chart Recorder

This is an A.E.I. model type 105. The operation is based on a D.C. potentiometer, a fixed voltage being maintained across a slide-wire by means of a constant current supply unit. The D.C. input signal to be measured is compared with the voltage across the slide wire and any difference in voltage serves to drive the potentiometer to balance and at the same time to move the indicating pointer and recording pen. The 10 inch wide chart can be traversed in one second. Input signals of less than 1 microvolt can be measured and ranges changed by means of plug-in units. A 10 m. volt full-scale deflection plug-in unit is used and as the output of the vibron is 10 volts full scale a chart
selector switch (Fig.2.4) was incorporated to attenuate the output by 1000; there are also facilities for attenuating by 1, 10, and 100. The chart recorder can also be connected directly by means of the switch to the fore-line Pirani or the backing line Pirani of the argon line, an external jack plug, or an ion gauge whose output can be dropped by 1, 2 or 10.

2.4(e) The Pumping system

This is a Balzers ultra-high vacuum system Type PST-26-U.H.V. It is supplied complete with bake-out ovens with the aid of which the entire plant above the cabinet including the adsorption traps and metal valve can be baked to a temperature of 450°C. The bakeable experimental area is 40 x 80 cm. and the argon line is situated here. After a few hours of baking a pressure of $1-2 \times 10^{-10}$ mm. of mercury is obtained on cooling.

Connected to the pumps are the ceramic adsorption trap, the Bayard-Alpert ionisation gauge IM-8, and the bakeable valve UV-8H. The adsorption trap is of the labyrinth-baffle type and is filled with activated alumina which reduces the back streaming of oil into the vacuum chamber to such an extent that the ultimate pressure obtained can be maintained over several months. The omegatron head is isolated from the pumps by the high vacuum valve UV-8H.
It is a manually operated valve of the Alpert type and in the fully open position has a conductance of at least 0.5 litres/sec. and when closed about \(10^{-10}\) litres/sec. It is advisable when baking to dismantle the tap and clamp it open as this will greatly prolong its life.

Pressures in the high and ultra high vacuum ranges are measured with the Bayard-Alpert tube IM-8. With the ion collector surface reduced to a minimum the effect of the generated soft x-rays is so small that pressures in the range of \(1 \times 10^{-11}\) mm. of mercury can be measured. The IM-8 is fused to the bake-able part of the apparatus and connected to the gauge control IMG-UI. This control also accommodates the medium vacuum part KV303 for the thermo-couple gauge head NV2. The range of the highly sensitive ultra high vacuum part is sub-divided into seven decades and the tube emission can be varied in three steps, 0.1-1.0-10.0 microamps.

The pumps, all indicator controls and safety devices are housed in a completely enclosed cabinet whose top is covered with heat insulating and heat-resisting 'Eternit' plates. An air insulation space equipped with cooling pipes provides additional protection for the pumps and control area against transfer of heat during bake-out.

Plate (1) shows the high vacuum bake-able area
including the adsorption trap, ion gauge head and all metal tap.

A rotary two stage backing pump with gas ballast provision is followed by a solenoid valve and booster pump. The booster pump is a two stage, all metal diffusion pump which provides the necessary working conditions for the all-glass oil diffusion pump and prevents possible backstreaming of oil vapours from the backing pump into the final pump.

The all-glass diffusion pump is a two stage fractionating, water cooled pump and has a water cooled baffle above it. From the baffle the vacuum duct runs to the adsorption trap in the workable area. The operating sequence of the pump unit is controlled by push buttons and can be made to proceed either manually or automatically. The pump unit is protected automatically at any stage in the pumping operation against failure in the cooling water supply by a water flow switch; against poor vacuum by a backing pressure relay and against breakdown in the current supply by a phase guard. These safety devices switch off the pump unit either partially or wholly according to the nature of the failure, and the type of failure is indicated by continuous flashing of the appropriate pilot light in the push button. Plate (2) shows the pumps fitted in the cabinet.
The bake-out control is fully automatic and the temperature of bake set at 450°C. The omegatron head which protrudes out of the end panel of the oven and between the magnet pole-pieces has a specially made oven of asbestos board and steel which will fit over it when the magnet is racked away. The bake-out temperature of the head is controlled by a Variac connected to two electric fire elements in the oven. A Venner time switch was incorporated with the pumping unit to control the bake-out time. This switch can operate the bake-out relay and at the same time provide the power for the oven around the head.

Plate (3) shows the bake-out ovens in position.

2.4.(f) The Emission Regulator

This was built from a circuit supplied by Dr. Klopfer of Philips in Aarchen and is shown in Fig. 2.5.

Basically the power for the filament is supplied by two double tetrodes QQV03 working on separate halves of a 2 x 600 volt mains transformer. The output of these valves was stepped down to about 10 volts and smoothed to remove any a.c. ripple. Stabilisation of the emission is achieved by amplifying the trap current using the EF 40 pentode and feeding the output to the two tetrodes. This provides negative feedback which can be varied by switching the trap current through four different input loads, giving
emission currents of 1, 3, 10 and 30 micro amps. An emission current of 3 micro amps is normally used. An unregulated position of the control switch bypasses the amplifying valve.

The various voltages to the electrodes are supplied by using rectifiers, L-C smoothing circuits and voltage stabilisers in the normal way.

A trip is incorporated in the output of the two power valves which can be set to trip out the filament transformer, operated by a push-on switch, if the current to the filament exceeds 5 amps. The external trip of the IM-8 gauge is also connected in to the power for the filament transformer.

2.4.(g) Connections to the head

An eight core screened cable is taken from a plug at the back of the emission control unit to a small brass box mounted on the vibron casing. This box serves as a plug-in point for the leads to the head and is useful for test purposes. All electrical connections are made as in Fig.2.6. Meter J₂ is a very sensitive galvanometer which is used in the setting up procedure and when the omegatron is used it is replaced by a jumper. This ties the screen of the oscillator, the anode and the casing of the vibron to earth. J₁ (see Fig 2.1.) is the meter on the emission control unit
which registers the trap current. \(V_\text{R}\) supplies the small positive bias to the four guard rings which are connected at the junction of 5 x 200 ohm carbon resistors. Two 1\(\mu\)F condensers allow A.C. to pass to these guard rings.

A thin walled perspex tube which would slip over the glass support for the ion collector is covered with copper foil to provide adequate and necessary screening of the collector. To facilitate connections to the head, small 'chocolate block' connections were made of 1/16 inch brass rod, these could be screwed on to stiff copper wire mounted loosely in holes drilled into a perspex disc so that they were aligned with the appropriate pins of the head. The perspex disc was fixed to the tube (Fig. 2.7. and Plate 4) and the 5 x 200 ohm resistors mounted directly behind the disc on to the appropriate copper connections to the guard rings. The ion collector was connected to the vibron by a piece of stiff copper wire and it could be screened with a sliding brass tube. It was found essential that the anode, copper foil and brass tube were all earthed to the vibron casing for removing unnecessary pick-up noise.

Plate 5 shows the control panel for the omegatron.

2.5. **The setting up procedure**

All the correct voltages being on the electrodes as in Fig. 2.1, a 100 micro-amp meter is connected into the lead to grid \(G_1\). The filament current 'on' button is pressed
with the emission switch in the unregulated position. A graph of filament current against meter reading is plotted to ensure that sufficient emission is possible.

The electron beam must then be lined up with the magnet field so that no electrons impinge on the anode as this would create secondary electrons and alter the characteristic of the head.

The magnet is first adjusted in two directions at right angles for maximum trap current, measured with \( J_1 \), keeping the emission switch in the unregulated position. A fine adjustment is then made by using \( J_2 \), which is plugged into the box on the vibron. This must register no electron current, i.e. less than \( 10^{-8} \) ampere.

The small positive voltage \( V_R \) and the large negative voltage \( V_{cc} \) must be adjusted for the optimum operating conditions so that a maximum ion current can be obtained. It is the property of an omegatron that an ion current can be measured in the absence of an r-f field. This occurs when the potential of the ion source becomes equal to the potential of the ion collector due to space charge effects. This residual ion current is plotted for variations of \( V_{cc} \) with no oscillating voltage and with \( V_R \) set at zero. Similarly \( V_R \) is plotted against the residual ion current with \( V_{cc} \) set at -55 volts. Figs 2.8 and 2.9 show the working ranges when no residual ion current is observed.
The oscillator is then switched on to give an output of about 1 volt r.m.s. and the frequency of the generator tuned on to the argon - 38 peak, the gas being obtained from the spike bulb of the argon line.

From equation (2.1) the resonance frequency

\[ f = \frac{1533 B}{M} \]  

where B is now in Gauss and M is in Mass units.

The ion current must then be regulated to a maximum value by limited variations of \( V_R \) and \( V_{CC} \) within the working ranges and by adjustment of \( V_T \), the trap voltage.

Figs. 2.10, 2.11 and 2.12 show the working ranges are

\[ 0 \leq V_R \leq 0.2 \text{ volts} \]
\[ -65 \leq V_{CC} \leq -55 \text{ volts} \]
\[ 16 \leq V_T \leq 22 \text{ volts} \]

In the range of values of \( V_R \) where there is a large residual current, it is interesting to note that non-resonant and resonant ions can reach the collector.

Brubaker and Perkins (22) have shown that there is a critical lower limit of the oscillator voltage at which the resonant ions just manage to reach the ion collector after passing two potential barriers. A sample of atmospheric argon was prepared and used to optimise the values of the oscillator voltage. The argon-40 and argon-36 peak heights were measured for different values of the oscillator.
voltage (Fig. 2.13). At 1.05 volts r.m.s., a saturation point is reached where all the argon-40 and argon-36 ions reach the collector and an atmospheric ratio \( \frac{A^{40}}{A^{36}} \) of 296 is obtained.

2.6. **Method of use**

If we have a gas sample in the argon line there are two possible ways of measuring the argon isotope ratios.

2.6.(a) **Dynamical method**

The gas sample is let into the head through a small leak, the tap to the pumps being open. The peaks decay away exponentially and are scanned with the magnetic field, keeping the oscillator frequency fixed. Isotopic fractionation occurs, lighter masses being pumped away faster. The initial isotope ratios can be determined by fitting the best exponential curve to the peak heights and extrapolating back to time zero, when the trap was opened (Miller\(^\text{(24)}\)).

2.6.(b) **Static Method**

This method was the one adopted and it consists of letting the sample into the head with the tap to the pumps closed. The mass spectrum could then be scanned either by hand or automatically. After some experience with the location of the peaks, it was found that the numbered scale of the magnet dial could be used as a setting for the different masses. Using a field of 9 Kilogauss and a
frequency of 350 Kc/s, the peaks were quite stable, but for any higher fields the magnet would overheat, and the resonance condition would no longer occur.

If the gas sample was insufficiently clean and the pressure below $4 \times 10^{-5}$ mm$^3$, the characteristic of the omegatro would change. Non-resonant ions would be collected causing an upward drift of the base line resulting in inaccurate measurements of peaks at high gain. Using the static method, manual scanning was found to be preferable to automatic scanning as the peaks were stable in the resonance condition and could be measured for a considerable time removing any errors due to fluctuations in the emission.

2.7. Resolving power

This is defined as $\frac{M}{\delta M}$ or $\frac{\omega}{\delta \omega}$ where $\delta M$ and $\delta \omega$ correspond to the base width of a peak in appropriate units. Ions of a given mass will be collected throughout the frequency range $(\omega_0 - \epsilon)$ to $(\omega_0 + \epsilon)$ so long as the maximum amplitude of their motion is equal to or greater than the distance $R_0$ to the collector.

From equation (2.3), in the vicinity of resonance where $\omega_0 \approx \omega_i e \epsilon \ll \omega_0$, if we consider particles of zero initial velocity,

$$x + iy \mid \epsilon \ll \omega_0 = \left\{ \frac{1}{\alpha/[2\omega_0 + \epsilon + \epsilon]} \right\} \left( 1 - e^{i \epsilon (\omega_0 - \omega)} \right) x e^{-i \omega_0 t}$$
This has a maximum amplitude of \( \frac{2a}{(\omega_o + \epsilon)} \)

and as \( \delta \omega \approx 2\epsilon \)

\( \delta \omega \) is determined by

\[
R_0 = 2a (\omega_o + \epsilon) \epsilon = \frac{2a}{\omega_o} \delta \omega
\]

So the resolving power \( \frac{\omega}{\delta \omega} = R_0 \omega_o / 2a \)

\[
= R_0 \frac{eB^2}{2BM}
\]

For our omegatron this should give complete separation of one mass unit at mass 40 with an oscillator voltage of 1 volt peak to peak for a field of 5 Kilogauss. Klopfer and Schmidt (25) state that for this type of omegatron complete separation is possible up to mass 30. It is not possible to increase the resolution much by decreasing the oscillator voltage as this must be kept within the saturation range. Increasing the magnetic field does increase the resolution as predicted.

For argon isotope measurements it is only necessary to have complete separation of 2 mass units at mass 40 provided the sample is clean and there are no intermediate peaks. With fields of 9 Kilogauss masses 38 and 40 have complete separation, the tailing of the 38 on the 40 peak being less than 1 part in 50,000 and completely undetectable. A typical scan of atmospheric argon mixed with 'spike' 38 clearly shows this complete separation (Fig. 2.14). The frequency of the oscillator was 350 Kc/s giving a theoretical
field of 9 Kilogauss for the resonance condition of argon-40.

2.8. Sensitivity

The system was not adjusted for maximum sensitivity as the volume of the apparatus could be much smaller. With 3 micro-amps emission, in the present system, a gas sample of $10^{-3}$ mm when let into the head from the last section of the argon line will give an ion current of $1 \times 10^{-10}$ amperes. Using a $10^{10}$ ohm input resistor in the vibron head this represents a voltage drop of one volt or ten divisions at unit gain on the amplifier. At gain 10,000; ten divisions are easily detectable against the random background noise level. A volume of $10^{-7}$ cu.mm. is thus the smallest volume of gas that can be easily detected with the omegatron adjusted in its present condition.

2.9. Response with pressure

A sample of atmospheric argon sufficient to give a reasonable deflection at low gain was prepared and isolated in the last two sections of the argon line. The last section of the line was opened to the head with the tap to the pumps closed. A pressure of $3 \times 10^{-5}$ mm. of mercury was indicated by the ion gauge. The controls were set for the resonance condition of argon-40 and when the chart
recorder had reached its maximum value the height and gain noted. The argon in the head and the last section of the argon line was pumped away, and the sample remaining let into the two end sections as before. This smaller volume was then let into the head and the process repeated until too little gas was left for an accurate measurement, 21 times in all. A pressure of \(2 \times 10^{-6}\) mm. of mercury was then registered by the ion gauge.

A typical scan is shown of the fourteenth shot being let into the head and pumped away (Fig. 2.15).

A graph of the logarithm of the deflection against the shot number (Fig. 2.16) clearly shows the linearity of response of the head within the pressure range \(3 \times 10^{-5}\) to \(2 \times 10^{-6}\) mm. of mercury.

This then leads to the possibility of calibrating the peak heights in terms of the volume of gas admitted to the head.

2.10. Mass discrimination

If \(A^{40}, A^{30}\) and \(A^{36}\) are the omegatron recorder deflections at unit amplifier sensitivity for argon samples from a rock, then the number of ions per unit time arriving at the collector and the number of atoms or the volumes of gas in the head will be proportioned to these. These proportionality constants may be different for the different isotopes if there is any mass discrimination.
A sample of atmospheric argon was prepared by purifying a small air shot of volume about 1 cc. and expanding it into a 1 litre flask connected to the spike tap. One shot from the spike tap then gave a reasonable deflection at gain 4 for argon-40. The atmospheric ratios were then determined by scanning the three isotopes of argon for a period of 20 minutes. The mean of 10 peak heights of each isotope and its root mean square error were determined.

For two different atmospheric samples the ratios obtained were:

\[
\begin{align*}
A^{40}/A^{36} &= 298.5 \pm 2.9 \\
&\quad 294.3 \pm 2.0 \\
A^{36}/A^{38} &= 5.27 \pm 0.15 \\
&\quad 5.25 \pm 0.07
\end{align*}
\]

Weighting the values according to their respective errors \(\text{Bond}^{(26)}\), the mean values and their errors are found to be:

\[
\begin{align*}
A^{40}/A^{36} &= 295.6 \pm 1.4 \\
A^{36}/A^{38} &= 5.26 \pm 0.06
\end{align*}
\]

Nier\(^{(27)}\) determined the atmospheric ratios for the isotopes of argon and obtained values of

\[
\begin{align*}
A^{40}/A^{36} &= 296.0 \pm 0.53 \\
A^{36}/A^{38} &= 5.32 \pm 0.01
\end{align*}
\]

From a comparison of these results, no observable mass discrimination effects are apparent.
SCHEMATIC DIAGRAM OF THE OMEGATRON

FIG. 2.1
VARIATION OF MAGNET FIELD WITH CURRENT

FIELD K. GAUSS

FIG. 2.2

POLE PIECES 43 MM. APART
SHIMS RETRACTED
SHIMS OUT

CURRENT IN AMPS.
VARIATION OF FIELD AT 5K.GAUSS WITH DISTANCE FROM CENTRE

FIELD K.GAUSS

POLE PIECES 43 MM. APART
SHIMS RETRACTED
SHIMS OUT

DISTANCE FROM CENTRE CM.

FIG 2.3
FIG. 2.4
The Omegatron
Emission Control Circuit

**FIG. 2.5**
JUNCTION BOX AND TEST POINTS

TO EMISSION CONTROL

10
9
8
7
6
5
4
3
2
1

1μF
1μF

V_RF

J_2

100K

BRASS BOX

PERSPEX DISC
R=150Ω CARBON RESISTORS

TO OMEGATRON

R

R

R

R

FIG. 2.6
CONNECTIONS TO THE OMEGATRON

FIG. 2.7
RESIDUAL ION CURRENT
(ARBITRARY UNITS)

FIG. 2.8

$V_{cc} = 60$ VOLTS
FIELD = 8.5 K. GAUSS
FIG. 2.9

RESIDUAL ION CURRENT (ARBITRARY UNITS)

VR = 0 VOLTS
FIELD = 8.5 K.GAUSS

WORKING RANGE
WORKING RANGE

$V_{cc} = 55 \text{ VOLTS}$

$\text{FIELD} = 7.5 \text{ K.GAUSS}$
RESONANT IONS OF ARGON -38

RESONANT ION CURRENT
(ARBITRARY UNITS)

FIG. 211

\[ V_R = 0 \text{ VOLTS} \]
\[ \text{FIELD} = 7.5 \text{ K.GAUSS} \]
FIG. 2.12

$V_R = 0 \text{ VOLTS}$
$V_{CC'} = -55 \text{ VOLTS}$
FIELD = 7.5 K.GAUSS
VARIATION OF ARGON-36,40 PEAK HEIGHTS AND ATMOSPHERIC RATIOS WITH R-F VOLTS

FIG. 2.13
RESOLUTION SCAN OF ATMOSPHERIC ARGON MIXED WITH 'SPIKE' ARGON

FIG. 2.14
ARGON-40 BEING LET INTO THE HEAD
AND PUMPED AWAY

FIG. 2.15

PEAK HEIGHT
OF A-40

TAP TO PUMPS
OPEN

GAIN CHANGE OF 10

SAMPLE IN

TIME (MINS.)

0 1 2
LINEARITY EXPERIMENT

FIG. 2.16

SHOT NUMBER

Log$_{10}$ deflection at gain 1000
PLATE 1
THE HIGH VACUUM SIDE
PLATE 2

THE PUMPING UNITS

FORE-LINE PIRANI GAUGE

ALL GLASS DIFFUSION PUMP

OMEGATRON DIFFUSION PUMP

OMEGATRON ROTARY PUMP

ARGON-LINE DIFFUSION PUMP
PLATE 3

THE BAKE-OUT OVENS IN POSITION
PLATE 4

CONNECTIONS TO THE HEAD
PLATE 5
THE CONTROL PANEL
PLATE 6
THE ARGON-LINE
CHAPTER 3

THE ARGON LINE

3.1. General

For efficient practical use it is essential that an argon purification line be as small as possible. This minimises absorption on the walls of the apparatus and also permits the partial pressure of the gases in the line to be as high as possible allowing quicker and better cleaning. The compactness of the line also facilitates the detection of leaks. It is also necessary for the line to be robust and easy to handle.

Such a line has been built, similar in construction to that used by Miller(24) but with several modifications. Fig.31 shows a schematic diagram of the line.

The entire plant is constructed on a 6 by 14 inch stainless steel plate which is screwed on to the bake-able area above the cabinet containing the pump units for the head. Plate 6 shows the layout of the line which is separated from the bake-able area of the head-side by an aluminium partition filled with glass fibre. This allows a lower bake-out temperature for the argon line, controlled by a Simmerstat. The line is connected to the head by
by tap (4) making leak detection easy and swift. When a leak becomes apparent, the controls of the omegatron are adjusted until mass 2 comes into resonance, and a jet of hydrogen is played on to the possible area of the leak. With tap (4) open to the head a leak is indicated by a sudden rise in the peak height of mass 2.

3.2. The Pumping System

A Leybold-Elliott oil diffusion pump fitted immediately below the line is backed by a rotary pump placed on the floor below the pump controls. They are connected by a \( \frac{3}{4} \) inch bore copper tube to provide good conductance and fast pumping speeds. The air ballast control of the backing pump extends through the blank panel near the floor.

A push button control circuit was built to operate the two pumps, an air admittance valve fitted directly on the rotary pump and a magnetic valve between this and the fore pump. The system could be operated either manually or automatically by pushing the appropriate buttons; the control panel is fitted below the control panel for the head pumps (see Plate 5). Fig 3.2 shows this control circuit.

When the 'automatic' button is pressed, a thermal delay provides a time lapse between the backing pump and the diffusion pump coming on. A water flow switch incorp-
orated in the diffusion pump cooling supply operates a buzzer and switches off the pump in the event of a water failure. Should the mains fail, the magnetic valves will close in the correct sequence and when the power is returned the cycle will start up automatically.

A water recycling system was developed for the cooling water to the diffusion pumps and the magnet. Fig. 3.3 shows the schematic layout of this system. A large tank filled from the mains water supply supplies a constant head of water and has a float switch incorporated in it. If the mains supply fails this switch will operate a small recycling pump connected to a small tank at the outlet of the cooling supply. Water will thus be pumped to the main tank until it reaches its original level, when the float switch will switch off the recycling pump. This pump can also be operated manually.

Owing to the low head of water available, it was found necessary to fit a booster pump at the outlet of the main tank so that the pressure switch in the cooling supply of the omegatron pumping system would not operate. This pump is run continuously.

A 250 micro amp meter, marked '0-25, Water Reserve, gallons' indicates the level of the water in the main tank.
These automatic safety devices were considered essential for the continual running of the apparatus and saved endless time in re-baking the vacuum system and pumping down.

It was found that a bake-out temperature of $100^\circ C$ was quite sufficient to produce a pressure of less than $10^{-6}$ mm. of mercury, if the system was baked out over night. With this pressure, no background can be seen in the mass range 29-43. A time switch is used to switch off the baking during the night, allowing sufficient time for cooling and immediate use in the morning. A higher bake-out temperature was considered inadvisable as this would be liable to cause loss of radiogenic argon from the rock sample which was loaded in the bake-out zone, in the furnace.

3.3. Pressure Indicators

A fore-line pirani gauge blown on a glass metal seal is connected between taps (2) and (3) by a $\frac{1}{4}$ inch bore copper tube. As the gauge was not bake-able it was fitted below the bake-out zone. This gauge is used to indicate the state of the cleaning process. A similar pirani gauge is fitted to the large bore tubing of the backing line.
These two gauges are incorporated in a bridge circuit shown in Fig. 3.4. A three way switch enables one to watch the complete cleaning process or the pumping down of the backing line by switching either pirani gauge to a variable resistance in units of 1 and 10 ohms. While one process is being watched the other gauge is switched to a fixed resistance which causes the gauge to zero at a good vacuum position. A sensitivity control was built into the circuit so that the meters indicating the good vacuum position would stay on scale over the whole range of pressure. When this control is not in the maximum sensitivity position, a red light is showing. The centre position of this three way switch brings in the fixed resistance, indicating a good vacuum position in both pirani gauges, when the respective meters are in the zero position. An internal external switch can be used to bypass the meters and connect the gauges directly to a chart recorder.

An Elliott ion gauge connected in the vacuum line between taps (3) and (4) indicates the pressure in the line and is necessary to ensure the pressure is low enough for the omegatron to work satisfactorily. The situation of the control panel for the gauge is shown in plate (5).
3.4. Furnace Units

Initially two furnace units 8 inches high were employed. They were built by Wesley Coe Ltd. of Cambridge and were mounted on $3\frac{3}{4}$ inch diameter stainless steel flanges by hard soldering a 1½ inch copper glass seal. They could be sealed on to the argon line by bolting them down on to a diamond section copper ring on a similar stainless steel flange. The copper rings can be used many times before they need resharpening or replacing. The sample fusion furnace is in the section between taps (1) and (2) and the titanium cleaning furnace between taps (2) and (3). Both these units are cooled by water running through a glass outer jacket, which is sealed, using a rubber 'O' ring, on to the metal of the glass-metal seal.

In the process of using the line, it became apparent that much atmospheric contamination came from the metal crucible and its quartz support. An automatic loader was then constructed (see plate 6) whereby this source of atmospheric contamination could be removed by pre-heating the crucible and support, before dropping the samples in. The original furnace units were shortened slightly and an off-centre ½ inch bore glass tube joined on to the top of the unit. Five narrow bore side arms were fitted and contained stainless steel rods. These rods could be drawn
across the vertical tube by means of a magnet.

Five samples can be loaded at once by dropping the samples in one at a time, with the furnace unit inverted, and then drawing the rods across. The crucible can then be preheated to drive off the absorbed gases and when the crucible and support have cooled, the first sample can be dropped in by withdrawing the first rod. This automatic loading device is particularly useful for whole rock measurements but can also be adapted for using separated minerals, by making small sachets to contain the sample.

3.5. Vacuum Taps

These all-metal taps are identical to those used by Miller\textsuperscript{24} and were developed by Mr. L. H. Flavill. The taps 1–4 and the spike tap 5 are connected together by hard soldering \(\frac{1}{4}\) bore copper tubing. For details of their construction see Miller\textsuperscript{24} and Mudie\textsuperscript{28}.

3.6. The Spike Tap

In the isotope dilution technique a known volume of enriched argon-38 is mixed with the argon obtained by the fusion of the sample, the volume of argon-40 being estimated by measurements of the argon 40/36 and 36/38 ratios of the mixture.

It is essential to know the isotopic composition of
the enriched argon-38 and also the exact volume of the spike added. The former can be measured to an accuracy of 1% by comparison of the peak heights of the isotopes, using the omegatron. The greatest difficulty had been to devise some way of supplying constant volumes of the spike. This is achieved by using a double vacuum tap with a common stainless steel base (Müdie(28)). A hole was drilled through this base and when both taps are shut, a known volume, approximately \( \frac{1}{2} \) cc. is enclosed and constitutes the spike volume. The stainless steel base is water cooled by copper pipes joined to it, and an asbestos box was constructed to fit over the tap during bake-out of the argon-line. When admitting the 'spike' it is essential that the temperature of the tap and the reservoir are the same, otherwise differences in volume of the spike will result.

Suppose the volume of the spike is \( v \) and the volume of the reservoir at a temperature \( T \) is \( V \) where \( V/v > 2000 \).

Then when the spike is open to the reservoir the number of molecules in the spike will be

\[
\frac{V \times n}{V + v} \approx \frac{v \times n}{V}
\]

where \( n \) is the total number of molecules present

Now suppose the volume of the spike is increased to \( v + v_s \) and the volume of the reservoir to \( V + V_R \) by increases in temperature of \( T_s \) and \( T_R \) respectively.
Then the pressure in the spike is given by

\[ P_s = \frac{n_s k (T + T_s)}{v + v_s} \]

where \( n_s \) is the number of molecules in the spike and \( k = \) Boltzmann's constant and the pressure in the reservoir is given by

\[ P_R = \frac{n_R k (T + T_R)}{V + V_R} \]

(\( n_R \) is the number of molecules in the reservoir) but these pressures must be equal and therefore

\[ \frac{n_s}{n_R} \approx \frac{n}{n} = \frac{v + v_s}{V + V_R} \times \frac{(T + T_R)}{(T + T_s)} \]

i.e. the number of molecules in the spike is

\[ \frac{n (v + v_s)}{V + V_R} \times \frac{(T + T_R)}{T + T_s} \]

Now for a temperature change of one degree centigrade, the fractional change in \( v \) is equal to the coefficient of subical expansion of stainless steel which is \( \sim \frac{3}{10^5} \) per \( ^oC \) and similarly for the glass reservoir \( \sim \frac{1}{10^5} \) per \( ^oC \).

Thus the expression for the number of molecules in the spike volume reduces to

\[ n_s = \frac{v}{V} \times n \frac{T + T_R}{T + T_s} \]

As \( T \) is around \( 300^oK \) a temperature difference \( T_s - T_R \) of only \( 3^oC \) will result in a 1% change in volume of the spike.
3.7. Method of use

When the argon line is leak tight and the pressure better than $1 \times 10^{-6}$ mm. of mercury, indicated by the ion gauge, the stainless steel trap (see Mudie(28)) is cooled in liquid nitrogen to remove any oil vapour which may have back-streamed from the diffusion pump.

The induction heater coil used to fuse the sample is placed around the furnace unit, and the molybdenum crucible made from 0.002 inch sheet is heated to about $1400^\circ$C. Tap (2) is shut while the crucible is being heated to keep the other two sections clean. The hot crucible also heats up the quartz tube acting as a radiation shield and support. A bad pressure is indicated by the backing line pirani, and it may be necessary to switch off the diffusion pump. When the backing line pirani reaches its equilibrium position and no more gas is being driven off the crucible and support, the heater is switched off.

Liquid nitrogen is then placed round glass trap (2) and around the charcoal trap (1). Tap (1) is then shut and the first sample released into the crucible. The sample is then heated slowly at first and then more strongly until a temperature of about $1300^\circ$C is reached and the sample is fused. The charcoal trap (1) serves to absorb most of the
gas evolved, reducing the pressure in the line and hence heat losses by conduction, thus making fusion easier. The induction coil is then placed around the second furnace unit, containing titanium sponge, and it is heated to a bright red heat at a temperature of 900°C with taps (2) and (3) closed.

Liquid nitrogen is then removed from trap (1), and tap (2) is opened. The absorbed gases then come into contact with the titanium sponge, which is slowly cooled by progressively lowering the power of the induction heater. Any volatile gases are condensed on to the trap (2). If an isotope dilution determination is to be made, a sample of spike argon-38 is prepared in the spike tap by opening it to the reservoir. Assuming the titanium has cleaned the sample so that the pirani gauge is reading less than 1 Ohm from the zero position, the sample is let into the last section of the argon line together with the spike. Liquid nitrogen is then placed around the charcoal trap (3) and the system left for 10 minutes. Fig. 3.5 shows the process of the argon being absorbed on to the charcoal.

Tap 3 is then closed and the titanium reheated to 1000°C with the line open to the pumps and the absorbed gases pumped away. The tap to the head is opened and
hydrogen pumped off for 20 minutes while the titanium is being cleaned. When the pressure in the fore-line has reached a pressure indicated by about 7 ohms from the fore pirani zero position with the titanium still hot, the tap to the head is closed and the liquid nitrogen removed from (3) allowing the argon to come off the charcoal. When the charcoal has reached room temperature, tap (2) is closed and tap (3) opened for a second cleaning. On complete cooling a pressure of less than $4 \times 10^{-5}$ mm. of mercury must be indicated by the ion gauge otherwise the cleaning process has to be repeated.

The argon evolved from the rock and the spike argon-38 are then isolated in the last two sections of the line. Tap 3 is then shut and the sample in the last section allowed to flow into the head, the tap to the pump being closed. An analysis of the spectrum in the mass range 36-40 is made, scanning with the magnetic field. When suitable deflections were obtained by adjusting the gain controls on the amplifier, the sample was then pumped away by opening the head tap. The gas sample left is then expanded into the two end sections as before and the process repeated six times.
3.8. The cleaning process

Unlike the total volume method (Miller(24)) for measuring the argon sample, it is not essential that it should be perfectly clean. It is essential however that the final pressure should be better than $4 \times 10^{-5}$ mm. of mercury, otherwise the characteristics of the head will change and incomplete or excessive collection may result. Thus it is advisable to get the argon sample as clean as possible.

Water vapour, carbon dioxide and any volatile gases, e.g. oil vapour which may have by-passed the stainless steel trap will be condensed out by the liquid nitrogen trap (2). Evernden and Curtis(29) maintain that more than 90% of the argon can be lost by the removal of carbon dioxide at liquid nitrogen temperatures. This phenomenon has not been observed and may be due to the large difference in sample size used. By far the greater proportion of the gas evolved is nitrogen and oxygen. Miller (24) has investigated the clean up of an air sample by red-hot titanium sponge and has shown that in one clean-up process 99% of the gases are absorbed. Fortunately the absorption process is reversible and the titanium can be rejuvenated by outgassing to a temperature of $1000^\circ \text{C}$. This rejuvenation
process normally takes somewhere between $\frac{1}{2}$ and $\frac{3}{4}$ of an hour. The titanium does not appear to lose its absorption efficiency after many hundreds of runs and can be used over and over again.

When the molybdenum crucible is heated for outgassing at the start of a run, large volumes of gas are evolved but after the initial burst has died down a clean-up process is observed even though the taps to the pumps are closed. This is due to the removal of oxygen by its chemical combination with molybdenum to form a variable oxide. M. R. Westcott\(^{(30)}\) has identified Mo\(_{40}\) by x-ray diffraction. The remainder of the oxygen is removed by the titanium sponge.

Hydrogen is removed by absorbing the gases on to charcoal at liquid nitrogen temperature, and then opening the system to the pumps. A trap down time of 10 minutes was found to be sufficient for absorbing the argon completely (Fig. 3.5). The hydrogen has a higher vapour pressure at this temperature and most of it can be pumped away. In one pumping process of $\frac{1}{2}$ hour, Miller\(^{(24)}\) has shown that 90% of a volume of 5.26 mm\(^3\) of hydrogen was removed by pumping.
CONTROL CIRCUIT FOR PUMPS

Fig. 3.2
THE OMEGATRON AUTOMATIC WATER RECYCLING SYSTEM

Float Switch

Earth on tank

Water Tank

Level Indicator

250 microamps f.s.d.

To Booster Pump

FIG. 3.3
THE ARGON LINE PIRANI GAUGE CIRCUIT

FIG. 3.4
TRAPPING TIME ONTO CHARCOAL

PEAK HEIGHT

TRAP ON

GAIN CHANGE OF 100

FIG. 3.5
CHAPTER 4

DATING BASIC ROCKS

4.1. Suitability of the decay of K-40 for dating purposes

Sawyer and Wiedenback (31, 32) and Johnson (33) established the decay scheme of K-40. It decays in two ways, by K-electron capture to argon-40 and by β emission to calcium 40.

For a decay scheme to be suitable for geological dating, it is essential that no measurable amount of the daughter product should be present in the parent material initially, and none of the daughter product should be gained or lost once it has been formed. The decay of K-40 to A-40 satisfies the first condition, the validity of the second condition will be discussed later.

A half-life that is comparatively short compared to geological time will limit its application, and should the half-life be extremely long, very little of the daughter product will be produced which will make determinations at low ages difficult and inaccurate. Potassium-40 with a half-life of $1.3 \times 10^9$ years is extremely suited for age determinations from a few tens of thousands of years to
the beginning of geological time.

It is also useful to have the parent element commonly found in rocks for it to have wide application. The decay of K-40 is ideally suited for age determination in all these respects.

4.2. Theory of the decay

Fig. (4.1) shows the decay scheme of K-40.

The decay constants have been determined by counting techniques and by making estimates of the potassium/argon content of rocks of known ages.

We use the decay constants

\[ \lambda_\beta = 4.72 \times 10^{-10} \text{ yr}^{-1} \]
\[ \lambda_e = 0.584 \times 10^{-10} \text{ yr}^{-1} \]

\[ \frac{dN_{K40}}{dt} = - (\lambda_e + \lambda_\beta)N_{K40} \]

and \[ \frac{dN_A}{dt} = \lambda_e N_{K40} \]

\( N_{K40} \) and \( N_A \) are the number of atoms of \( K^{40} \) and of radiogenic \( A^{40} \) per gm. of rock.

\[ N_{K40} = N_0 e^{-(\lambda_e + \lambda_\beta)t} \]
\[ N_A = N_0 \left( 1 - e^{-(\lambda_e + \lambda_\beta)} \right) \frac{\lambda_e}{\lambda_e + \lambda_\beta} \]

The ratio of argon-40/potassium-40 is then given by
Therefore the age is given by

\[
t = \frac{1}{\lambda_e + \lambda_\beta} \ln \left[ 1 + \frac{\lambda_e}{\lambda_\beta} \frac{N_A}{N_{K40}} \right]
\]

Now suppose the fraction of K-40 atoms present in potassium is

\[N_{K40} = a \text{ } N_K\]

and \[N_K = \frac{N_p}{W_{K20}}\]

\(N\) is Avogadro's number, \(W_{K20}\) is the molecular weight of \(K_2O\) and \(p\) is the proportion in the rock of \(K_2O\) in gm/gm; the number of potassium-40 atoms present

\[N_{K40} = \frac{N \cdot a \cdot p}{W_{K20}}\]

and the number of argon-40 atoms present

\[N_A = \frac{V_R}{V} \text{ } N\]

\(V_R\) is the vol. in \(cm^3\) of the radiogenic argon at N.T.P. in 1 gm. of rock and \(V\) is the volume of 1 gm molecule of the gas in \(cm^3\).

\[\frac{N_A}{N_{K40}} = \frac{W_{K20}}{V \cdot a} \cdot \frac{V_R}{p}\]

The age may now be expressed in terms of the volume and
K₂O content.

\[ t = \frac{1}{\lambda_e + \lambda_\beta} \ln e \left[ 1 + \frac{\lambda_\beta + \lambda_e}{\lambda_\beta}, \text{C. } \frac{V}{P} \right] \]

where C is \( \frac{W}{dV} \) and is set at 17.6581

4.3. Measurement of potassium by the Bel photometer

The accuracy of the age determinations depends on the accuracy of the potassium content in the rock as well as the argon volume measurement.

Metallic salts when introduced into a non-luminous flame will burn with colours characteristic of the metal. A flame photometer utilises this phenomena to measure the concentration of alkali salts in a solution. The solution under test is atomised and introduced into a non-luminous flame burning under controlled conditions. This flame becomes coloured and the intensity of the light emitted is measured by means of a photo-cell. An optical filter allows only the light in the region of the spectrum appropriate to potassium through. The intensity of the light emitted by the sample is then compared with that emitted by a prepared solution of known concentration.

About 1 gm. of the rock to be analysed is weighed out accurately into a platinum crucible and then 1cc. of 50% sulphuric and 2cc of hydrofluoric acid are added.
The mixture is warmed gently at about 150°C on a sand bath until the fuming ceases, then heated strongly to dryness.

After cooling, 4 drops of 50% sulphuric acid are added to the solid residue and the crucible half-filled with distilled water. The crucible and contents are then heated on a water bath for at least one hour until all the material goes into solution.

The contents of the crucible are then transferred without loss to a one litre standard flask which is half-filled with de-ionised water.

A solution of 10 gm. of powdered ammonium carbonate (A.R.) in 50 cc. of water is prepared and 50 cc. of 0.88 ammonia solution added to it. Ten ccs. of this mixture are added to the solution in the standard flask which is shaken vigorously and allowed to stand for a few minutes. It is then made up to the mark with distilled de-ionised water and allowed to stand for some hours. This procedure causes precipitation of the heavy metals present. The solution is filtered through a dry filter paper and flask to ensure that no further dilution takes place. After discarding the first few ccs. of filtrate, the remainder is collected into a polythene bottle and samples taken for the flame photometer.
4.4. Gain of argon

The main source of extra argon is from the atmosphere, but fortunately this can be determined.

A certain amount of argon-36 is always found when a rock sample has been fused, even though the apparatus appears to be completely leak tight and a pressure of less than $1 \times 10^{-6}\text{mm}$ of mercury has been maintained in the argon line for several days. It can only be assumed to have come from absorption on the sample, molybdenum crucible and quartz support.

An experiment was carried out to determine how much of this argon came from these sources and to see if it was advantageous to devise a scheme for removing this source of contamination.

The crucible was heated for 10 minutes at a temperature of about $1400^\circ\text{C}$, sufficient to fuse a sample. The same procedure as for an age determination was used to clean the gas sample given off and prepare it for admission to the head. During this early part of experimentation no spike -38 was prepared and the volumes of gas were measured by comparison with the heights obtained from samples of known radio-genic argon content.

It was found that a volume of approximately $5 \times 10^{-4}\text{mm}^3$ of argon was evolved from the crucible and its quartz
support. An argon-40/36 ratio of 296 was found indicating that the source of contamination was solely atmospheric.

A basic igneous rock, e.g. a basalt, has on average about 1% of K₂O present and a rock of 30 million years will only evolve a volume of \(1 \times 10^{-3}\) cu.mm. when a sample of mass 1 gm. is fused. If there is no argon absorbed on the sample a typical age determination will have at best a 30% atmospheric contamination. Consequently an automatic loader was built as described in Section 3.4. to remove this source of atmospheric contamination.

4.5. Measurement of radio-genic argon-40 by the isotope dilution technique

In this technique the volume of argon evolved from the rock is determined by mixing a known volume of enriched argon-38 with the sample, and using it as an internal standard. This 'spike'-38 is admitted to the system from the spike-tap after the first cleaning process with the titanium. The procedure is carried out this way so that the clean walls of the system will absorb the oxygen and nitrogen, which are the initial gases evolved from the rock, and no fraction of the spike will be absorbed. The volume of the spike is calibrated using a known volume of argon-40 from a prepared air-shot. Initially it was calibrated using a known age rock sample.
4.6. Spike Calibration

4.6(a) Spike calibration using a known age sample

The particular sample chosen was of shap biotite, reference number 1875-20512, KA/8. Several determinations have been made on this rock by both the Oxford and Cambridge group (Dodson et al\(^{(34)}\)).

These results show that \(10.95 \pm 0.27 \times 10^{-2}\) mm.\(^3\) of radiogenic argon-40 is evolved per gm. of sample.

About 0.05 gm. of this sample was weighed out accurately and the sample fused and cleaned as for an age determination. The argon was isolated in the last two sections of the argon line and six progressive fractionations of this sample carried out, the \(A^{40}, A^{38}, A^{36}\) peak heights being measured each time as described in 2.9. In this way the average of six isotope ratios was obtained.

If \(\alpha = \frac{S^{38}}{S^{36}}\), \(\beta = \frac{S^{38}}{S^{40}}\) are the spike isotope ratios, and \(\alpha, \beta\) are of the order of 10,000, it can be assumed that all the 38 peak has come from the spike.

Suppose the absolute peak heights measured at unit gain of the amplifier are \(H^{36}, H^{38}\), and \(H^{40}\).

Of the 36 peak, \(\frac{H^{38}}{\alpha}\) is due to the spike and hence \(H^{36} - \frac{H^{38}}{\alpha}\) represents the amount of argon-36 of atmospheric origin.

Hence \(296 \left[ H^{36} - \frac{H^{38}}{\alpha} \right]\) is then the atmospheric contrib-
ution of the argon-40 peak.

\[ R^{40} = H^{40} - 296 \left[ \frac{H^{36} - H^{38}}{\alpha} \right] \]

and

\[ \frac{R^{40}}{H^{38}} = \frac{H^{40}}{H^{38}} - 296 \left[ \frac{H^{36}}{H^{38}} \frac{1}{\alpha} \right] \]

but the heights \( R^{40} \) and \( H^{38} \) will be proportional to the volumes of radiogenic argon-40 and spike respectively.

Therefore

\[ \frac{\text{volume of radiogenic}}{\text{volume of spike}} = \frac{H^{40}}{H^{38}} - 296 \left[ \frac{H^{36}}{H^{38}} \frac{1}{\alpha} \right] \]

Two measurements of the spike-isotope gave values of

\[ \alpha = 205,000 \pm 10\% \quad \beta = 11,000 \pm 10\% \]

As the contribution of the spike to the 36 peaks is normally around 2%, the error due to the inaccuracies of the isotope ratios is only about 0.2%.

Two spike calibrations were made and using equation 4.2 the volume of the spike was found to be

\[ (1.47 \pm 0.04) \times 10^{-3} \text{ cu.mm.} \]

and \[ (1.44 \pm 0.04) \times 10^{-3} \text{ cu.mm.} \]

4.6 (b) Spike calibration using an air shot

The smallest size air shot which can be prepared accurately has a volume of about 0.1 cc. and contains too much argon-40 to be measured on the omegatron.
A gas pipette was constructed by M. R. Westcott and myself whereby an air shot of known argon-40 content could be expanded into a large 1 litre flask and a known volume of approximately 25 cc. drawn off and used for the calibration of the spike.

Fig. 4.2 shows the schematic layout of this gas pipette. Mercury cut-offs were used as shown to isolate the different sections, and the argon line was kept free of mercury by surrounding a U-tube connected to the gas pipette with liquid nitrogen. This liquid nitrogen trap was kept cool continually to prevent any mercury condensing in the argon line.

The entire system was left pumping for several days and checked for any leaks. When the system was found to be leak-free, mercury was allowed to flow up arm II, to the mark as shown and hence isolate the system. The air shot was then broken, using the steel ball, and expanded into both the large and small reservoirs. When equilibrium was reached the small reservoir was isolated by raising the level of the mercury in arm 1. The spike shot was then prepared and let into the argon line together with the air shot. The mixture was cleaned and the argon-40 and argon-38 peak heights were measured.

This procedure was repeated 5 times using progressively
smaller volumes of the air shot. The volume of the larger
reservoir was measured by filling it with water, and measur-
ing the volume, and the smaller reservoir by weighing it
empty and full of mercury.

Except for the first calibration where the emission was
unsteady, each isotope ratio was measured at least six times.

They gave for the volume of the spike:

\[ 1.38 \pm 0.01 \times 10^{-3} \text{ cu.mm.} \]
\[ 1.46 \pm 0.01 \times 10^{-3} \text{ cu.mm.} \]
\[ 1.46 \pm 0.01 \times 10^{-3} \text{ cu.mm} \]
\[ 1.470 \pm 0.004 \times 10^{-3} \text{ cu.mm.} \]

The three consistent results give the volume of the spike to be

\[ 1.463 \pm 0.004 \times 10^{-3} \text{ cu.mm} \]

This agrees very closely with the results obtained using
the shap biotite.

4.7. Errors

Experimental errors, as distinct from the discrepancy
introduced by gain or loss of argon in response to geolog-
ical environment, arise from errors involved in the measure-
ment of the potassium and argon contents of the rock.

The magnitude of such errors can be estimated by making
repeated measurements on the same material, but it is more
satisfactory to evaluate the actual experimental errors
incurred in the measurements of the physical quantities
involved and determine the effect on the age.

Usually six determinations of potassium oxide content are made on a number of separate samples of the same specimen, a mean value and percentage error can then be determined. To eliminate any systematic errors involved, checks against standard samples are made prior to making the potash determinations.

Errors involved in the radiogenic argon measurement, using the isotope dilution technique, arise from inaccuracies in the spike volume and in measuring the isotope ratios of the final mixture. The former can be determined by making several consecutive measurements of the spike volume. As the volume of the spike tap is approximately $\frac{1}{2}$ cc. and that of the spike bulb 1 litre, the volume of the spike will only decrease by 1% in 200 age determinations. The errors involved in the measurement of the isotope ratios are determined by making at least six measurements of these ratios and calculating the mean and percentage error.

Overall errors also depend upon the condition of the sample and cannot be assessed quantitatively. The geological factors which influence the apparent ages of whole rock samples are considered in the next section.
4.8. **Factors influencing dates of basic rocks**

Potassium age determinations can be made on either individual minerals or whole rock samples. The micas have been used very successfully as they have conveniently large potassium content and under favourable conditions can retain over one hundred per cent of their radiogenic argon-40. Several other minerals, e.g. amphiboles and pyroxenes have been used with success.

The possibility of dating basic volcanics and minor intrusives, which have very low potash content, by the whole rock method, has been investigated in Cambridge (Miller and Mussett⁴⁵), and elsewhere (Brockson and Kulp⁴⁶). Successful results have been obtained by McDougall and Tarling⁴⁷,⁴⁸ in Canberra and by Evernden and Curtis⁴⁷ in California.

Miller and Mussett⁴⁵ carried out initial experiments on the validity of the potassium argon whole-rock method applied to igneous rocks using samples from the Whin Sill of Northern England. Apparent ages ranging from $305 \pm 20$ M.yrs. to $130 \pm 9$ M.yrs. were obtained. A marked correlation was found between the apparent ages of the samples and the relative amount of alteration shown by their plagioclase felspars. The apparently older samples were found to have the least amount of alteration. Electron
probe measurements demonstrated that potassium was relatively abundant in the small region corresponding to the potash felspars of the ground mass but was virtually absent from fresh plagioclase. In the secondary alteration products, within the plagioclase felspars, potassium was found to be present in some abundance.

Discrepant high ages can be obtained from rocks which contain zeolites. Taylor and Urey\(^{(38)}\) suggest that the zeolites cause the chemical fractionation of the potassium isotopes by a cation exchange process. It is possible that the passage of the volatiles continued over a time long enough for the zeolite constituents of the rock to capture excess radiogenic argon because of their high surface area. Miller and Fitch\(^{(39)}\) have obtained results from dolerites which gave impossibly high ages. They have also described rocks in which a subsequent mineralisation event occurred and caused all the previously accumulated argon to be lost.

It is concluded that whole rock samples of completely unaltered basic igneous rock can be dated successfully but doubt should be expressed of results obtained where there was any noticeable alteration of the felspars. Results from rocks containing any zeolites should be viewed with the greatest suspicion.

Before the meaning of potassium-argon dating can be
properly interpreted, it is necessary that full geological and petrological information on the material analysed should be available. Determinations of the initial ages of the rock cannot be accepted as accurate unless all the possible sources of discrepancy have been investigated and shown to be unimportant.
DECAY SCHEME FOR POTASSIUM-40

FIG. 4.1
THE GAS PIPETTE

TO ARGON LINE

LIQUID NITROGEN TRAP

25CC. BULB

ARM2

ARM1

1 LITRE BULB.

BALL BEARING

AIR SHOT

MERCURY RESERVOIR

AIR INLET

TO BACKING PUMP

FIG 4.2
CHAPTER 5

PALAEOMAGNETIC RESULTS FROM ETHIOPIA AND THE SUDAN

5.1. Results from Ethiopia

5.1(a) Ethiopian Geology

Some time after the Mesozoic formation, Eritrea and the remaining parts of Ethiopia underwent a considerable uplift, as much as 3,000 metres in places. The greater part of this high plateau is covered by a series of lavas, similar to those of the Deccan Traps of India, but unfortunately no suitable fossil zones can be found for dating the series. Abul-Hāggag (40) who has made a comprehensive study of the area believes the uplift occurred in the Upper Eocene; that is, it did not coincide with or occur immediately after the retreat of the Mesozoic sea as had been generally supposed by many previous authors.

The traps are definitely post Jurassic since they overlie sedimentary rocks that belong to Upper Jurassic times. It seems likely that the eruption was initiated by the local deformation which accompanied the Upper Eocene uplift and thus caused a number of breaks in the continuity of the crystalline substratum and the overlying sedimentary series. Through these breaks or fissures the internal magma rose to the surface of the uplifted country and continued to rise
after the uplifting movement had come to an end.

5.1 (b) **Sampling**

47 samples were collected by an Imperial College expedition to Ethiopia in the summer of 1962. 21 of these were collected from a series of plateau lavas in the Blue Nile Gorge on the Addis Ababa–Debra Marcos road. The remaining samples were from scattered sites throughout the country.

All specimens were marked in situ with a horizontal line, whose bearing was noted; and also a line at right angles, whose tilt was measured using a standard clinometer compass instrument.

In the Blue Nile Gorge, 6 or 7 lava flows outcropped from a height of 6,700 ft. to 7,900 ft., and these flows were sampled from the bottom of the section up. Figure 5.1 shows a profile of the gorge and the sites of the samples collected.

5.1 (c) **Measurement of samples**

Each specimen was set in plaster with the same orientation as it was found in the field and two cores cut from each. These cores $\frac{3}{8}$" in diameter and approximately 1" long were measured for their direction and intensity of remanent magnetisation using an astatic magnetometer of similar pattern to that described by Blackett\(^1\). The
mean direction of the specimens from the two groups are shown in figures (5.2) and (5.3) by plotting on an equatorial Schmidt equal area projection.

The 21 specimens from the Blue Nile Gorge show good agreement and as they were taken from several lava flows, the mean direction of magnetisation of all cores from this site will provide a reliable pole position for Africa at the time the rocks were laid down. The remaining samples, however, are unsuitable palaeomagnetically as they were from isolated and unrelated sites.

An attempt was made to reduce the scatter of the specimens from the Blue Nile Gorge by thermal demagnetisation in the manner described by Wilson (41), using his furnace. The magnetic moment of a specimen was plotted on a Schmidt equal area projection as a function of the temperature. In this way the progressive elimination of any secondary components can be seen, until at high temperatures only the original moment is left. Thermal demagnetisation was considered preferable to alternating demagnetisation because components with low blocking temperatures which could perhaps acquire viscous moments from the ambient field would be difficult to remove completely by the use of alternating fields.
5.a (d) Results of measurements

Table 5.1 shows the directions of magnetisation of the cores from the Blue Nile Gorge before and after heating. Figure (5.4) is a Schmidt plot of the directions after heating. The mean direction and circle of confidence was determined using Fischer's method. This was found to be 3° East of True North with a dip of 14° down, the 95% circle of confidence being 7°.

As the theoretical dipole field at the site where the rocks were collected has a dip of 19° down, the results thus indicate, that since the rocks were laid down, probably in the late Eocene, there has been no significant movement of the land mass of Africa as a whole.
Table 5.1
Directions of Magnetisation of Cores from the Blue Nile Gorge

<table>
<thead>
<tr>
<th>Sample No.</th>
<th>Direction &amp; Intensity before heating</th>
<th>Direction after heating</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Declination East of Magnetic North</td>
<td>Dip Down</td>
</tr>
<tr>
<td>19</td>
<td>10°</td>
<td>30°</td>
</tr>
<tr>
<td>20</td>
<td>14°</td>
<td>13°</td>
</tr>
<tr>
<td>21</td>
<td>0°</td>
<td>15°</td>
</tr>
<tr>
<td>22</td>
<td>18°</td>
<td>-2°</td>
</tr>
<tr>
<td>23</td>
<td>0°</td>
<td>-1°</td>
</tr>
<tr>
<td>24</td>
<td>15°</td>
<td>20°</td>
</tr>
<tr>
<td>25</td>
<td>16°</td>
<td>19°</td>
</tr>
<tr>
<td>26</td>
<td>16°</td>
<td>17°</td>
</tr>
<tr>
<td>27</td>
<td>4°</td>
<td>20°</td>
</tr>
<tr>
<td>28</td>
<td>301°</td>
<td>25°</td>
</tr>
<tr>
<td>29</td>
<td>2°</td>
<td>17°</td>
</tr>
<tr>
<td>30</td>
<td>331°</td>
<td>21°</td>
</tr>
<tr>
<td>31</td>
<td>0°</td>
<td>5°</td>
</tr>
<tr>
<td>32</td>
<td>335°</td>
<td>14°</td>
</tr>
<tr>
<td>33</td>
<td>354°</td>
<td>7°</td>
</tr>
<tr>
<td>34</td>
<td>268°</td>
<td>9°</td>
</tr>
<tr>
<td>35</td>
<td>0°</td>
<td>9°</td>
</tr>
<tr>
<td>36</td>
<td>0°</td>
<td>9°</td>
</tr>
<tr>
<td>37</td>
<td>1°</td>
<td>13°</td>
</tr>
<tr>
<td>38</td>
<td>329°</td>
<td>-9°</td>
</tr>
<tr>
<td>39</td>
<td>317°</td>
<td>1°</td>
</tr>
</tbody>
</table>
5.2. Results from the Sudan

5.2 (a) Natural Remanent Magnetisation

Ten samples were collected from a recent basalt exposure situated about 10 miles N.W. of Khartoum. This small outcrop was a source of building material and road metal for the surrounding districts. Each sample was marked as described in 5.1.(b).

The ten specimens were set in plaster and their natural remanent magnetisation measured as a whole rock sample. Fig. 5.5 shows a plot of these directions on a Schmidt equal area projection and they can be seen to lie in three distinct groups, approximately at right angles to each other. At first sight the reason for this seemed to be due to an error in marking the samples in the field. However, it was decided to take cores from one sample of each of the three groups and investigate the effect of heating on their directions of magnetisation. The particular samples chosen were S.18, S.23 and S.24.

5.2 (b) Results of heating experiments

The direction of magnetisation of each core was measured every 50°C using the Wilson furnace, until the Curie point was reached. The directions of each core are plotted every 50°C on a Schmidt projection (Fig. 5.6) and their intensities on Fig. 5.7.
Specimen S.23 reversed completely between $100^\circ$C and $150^\circ$C. S.24 and S.18 gradually changed their directions of magnetisation immediately heating commenced until at $150^\circ$C they became stable at a common direction close to that of the reversed rock S.23. When S.23 was reheated, after being cooled from $600^\circ$C in $\frac{1}{2}$ oersted, it was found to lose its reversing property completely (Fig. 5.7).

5.2 (c) Discussion of results

If magnetic particles with relaxation times less than the geological age of the rocks are present, a secondary component can be acquired, different in direction from the primary one, if the direction of the geomagnetic field has changed since the latter was acquired. This is termed a viscous magnetisation. Rocks with very short relaxation times comparable with the time of storage of the rocks can acquire a viscous component which can cause the direction of the specimens to change by as much as $180^\circ$ (e.g. Irving et al.(43)). This viscous magnetisation will be removed at low temperatures and is a possible explanation of the anomalous results obtained. Until many more heating and storage experiments are carried out on these rocks, the correct explanation will remain uncertain.
BASALT SAMPLES FROM
THE BLUE NILE GORGE

FIG. 5.1

ANERIOD READING

Basalt on plateau 20Km from Gorge
33 39

Columnar Basalt
34 35

Obsidian
36 37

Vesicular Basalt
32 33

Pillow Lava silicious
31
tree root found

Volcanic Ash

Basalts
25 26
Columnar but not
27 28
vertical
29 30
several
flows

Columnar Basalt
21 22
No Exposure
23 24

Columnar Basalt
19 20
No Exposure

Oolitic Limestone
with Shelly bands

Precambrian outcropping in the base of the
Gorge

Precambrian outcropping in the base of the
Gorge

FIG. 5.1
THE BLUE NILE GORGE SAMPLES

OTHER SITES

FIG. 5.2

FIG. 5.3

THE BLUE NILE GORGE SAMPLES AFTER HEATING

THEORETICAL DIPOLE FIELD - □
DIP UP - ○
DIP DOWN - x

FIG. 5.4
NATURAL MAGNETISATION DIRECTIONS OF SUDAN SAMPLES

DIRECTION CHANGES ON HEATING

DIP UP — o
DIP DOWN—x
THEORETICAL DIPOLE FIELD □

Fig. 5.5

Fig. 5.6
HEATING CURVES FOR SUDAN SAMPLES S18, S23, & S24

Intensity (emu/gm x 10^{-3})

- S18
- S24

Reheating of S23 after cooling from 600ºC in 1/2 oersted. (Intensity - half scale)

FIG. 5.7
6.1. Basalts from the Northern part of the Mid-Atlantic Ridge

Eight samples of basalt from the Rift zone of the Mid-Atlantic ridge were selected for age determination. These samples were collected during the autumn cruise of R.R.S. Discovery II in 1960 by Drs. M. N. Hill and D. H. Matthews of the Department of Geodesy and Geophysics, Cambridge. The dredging was carried out at station 4519 (45° 44'N, 27° 43' W) and the samples collected from the deep median rift valley, a typical feature of any traverse of the ridge and first reported by Hill (44). This central valley and the adjacent rift mountains contain the epicentres of nearly all the shallow focus earthquakes recorded.

Our knowledge of the ridge is derived mainly from the study of the rocks forming the volcanic islands that are scattered at wide intervals along the crest zone of the ridge. In both the North and South Atlantic the rift zone is still volcanically active, but in the South there is more evidence of Quarternary volcanic activity and the mid-ocean ridge is characterised by greater relief and a more youthful appearance, and the rocks here are of a more
alkaline character. Some of the 'Atlantis' cruise rocks have been dated by the potassium argon method and gave ages of a few tens of millions of years (Heezen and Ewing[45]).

Dr. I. D. Muir and Professor C. E. Tilley of the Department of Mineralogy and Petrology, Cambridge, who made a detailed study of the rocks dredged, selected the eight samples as they covered the range of composition met with among the specimens dredged. The potassium analyses were made by J. H. Scoon of the same department. The samples ranged from the olivine-basalt type to those with a distinctly alkaline affinity.

Thin sections were made of all the specimens measured. All appeared to be virtually unaltered except in some cases where the margins of micro-phenocrysts of olivine and plagioclase were composed of secondary chlorite, serpentine or calcite.

A brief description of each thin section is given in Table 6.1. All these samples should give very good ages due to their virtually unaltered nature.
Table 6.1

<table>
<thead>
<tr>
<th>Reference</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>665; 4519-56</td>
<td>A fresh holocrystalline alkali-olivine basalt. It should give a good age.</td>
</tr>
<tr>
<td>666; 4519-34</td>
<td>A very similar rock.</td>
</tr>
<tr>
<td>668; 4519-11</td>
<td>A virtually fresh olivine-dolerite.</td>
</tr>
<tr>
<td>669; 4519-55</td>
<td>A basaltic variolite with beginnings of alteration. Only slightly discrepant ages to be expected.</td>
</tr>
<tr>
<td>670; 4519-46</td>
<td>A virtually unaltered basaltic variolite.</td>
</tr>
<tr>
<td>671; 4519-65</td>
<td>A very similar rock.</td>
</tr>
<tr>
<td>672; 4519-54</td>
<td>A virtually fresh olivine-basalt.</td>
</tr>
<tr>
<td>673; 4519-21</td>
<td>This rock contains cores of almost unaltered basaltic glass with microphenocrysts within spheroids, the margins of which are composed of secondary chlorite, serpentine or calcite. The age is expected to be slightly low depending on how much of the altered material is included in the sample.</td>
</tr>
</tbody>
</table>

The results of age determinations on these samples are given in Table 6.2.

The ages ranging from Miocene to Late Pleistocene indicate the very youthful nature of the rocks measured as might have been expected from their tectonic setting in the Rift-zone.
Table 6.2
Mid-Atlantic Ridge Samples

<table>
<thead>
<tr>
<th>Reference</th>
<th>K$_2$O%</th>
<th>Radiogenic Argon-40 per gm. per K$_2$O%</th>
<th>Atmospheric Contamination</th>
<th>Age M.yrs</th>
<th>Error M.yrs</th>
</tr>
</thead>
<tbody>
<tr>
<td>665, 4519-56</td>
<td>0.613 ± 0.007</td>
<td>5.1 x 10$^{-5}$ cu.mm.</td>
<td>95%</td>
<td>1.6</td>
<td>4.5</td>
</tr>
<tr>
<td>666, 4519-34</td>
<td>0.57</td>
<td>4.3 x 10$^{-5}$ cu.mm.</td>
<td>-</td>
<td>1.3</td>
<td></td>
</tr>
<tr>
<td>668, 4519-11</td>
<td>0.330 ± 0.023</td>
<td>9.6 x 10$^{-5}$ cu.mm.</td>
<td>96%</td>
<td>3.0</td>
<td>± 1.5</td>
</tr>
<tr>
<td>669, 4519-55</td>
<td>0.490 ± 0.012</td>
<td>1.6 x 10$^{-4}$ cu.mm.</td>
<td>97.5%</td>
<td>4.8</td>
<td>± 2.7</td>
</tr>
<tr>
<td>670, 4519-46</td>
<td>0.427 ± 0.007</td>
<td>2.8 x 10$^{-4}$ cu.mm.</td>
<td>94%</td>
<td>8.5</td>
<td>± 1.5</td>
</tr>
<tr>
<td>671, 4519-65</td>
<td>0.297 ± 0.040</td>
<td>6.1 x 10$^{-4}$ cu.mm.</td>
<td>96%</td>
<td>1.8</td>
<td>± 6.0</td>
</tr>
<tr>
<td>672, 4519-54</td>
<td>0.59</td>
<td>9.4 x 10$^{-5}$ cu.mm.</td>
<td>92%</td>
<td>3.0</td>
<td>± 2.0</td>
</tr>
<tr>
<td>673, 4519-21</td>
<td>0.77</td>
<td>0.8 x 10$^{-5}$ cu.mm.</td>
<td>99%</td>
<td>0.23</td>
<td>1.1</td>
</tr>
</tbody>
</table>
6.2. The Deccan Traps

In 1955, Clegg et al.\(^{(46)}\) published a paper describing results of their palaeomagnetic studies on some Deccan Traps of India. These studies were confined to two regions about 550 miles apart; the first in the neighbourhood of Linga near the district capital of Chhindwara in the Central Province, and the second close to Khandala on the Bombay-Poona highway. Five samples from the Khandala region were selected for age measurement. They were thought to belong to the upper part of the middle section of the Deccan Traps, and are younger than the Linga rocks, probably being laid down during the early Eocene period (Fermor and Fox\(^{(47)}\)).

All the samples, olivine dolerites, were examined carefully in thin sections and were found to have some degree of alteration to a greater or lesser extent. K.13 was the only rock in which the alteration was confined to minor chloritization of the pyroxene and partial serpentinitization of the olivine. The other rocks had all been affected by a strong subsequent hydrothermal event that had caused leaching, resulting in chloritization and zeolitization in patches. Some showed a later oxidation of the chlorite due to weathering, and this is possibly the reason for the anomalous group of directions in Clegg's results. A later oxidation remagnetisation would have a direction of
magnetisation closer to the present earth's field.

During the early part of the experimentation, no spike was available and the volumes of argon evolved were determined by calibrating the peak heights using a known volume of radio-genic argon from a sample of shap-biotite. This is termed the 'deflection' method. Table 6.3 shows the results of the age determinations on these samples.

As was expected from an examination of the thin sections, the ages were widely scattered as rocks containing any zeolites can have discrepantly high ages. K.13 is the only sample in which the ages determined by Miller and Mussett (unpublished) agrees, and this is the only dolerite which looks reasonably fresh in thin section. In the others, radiogenic argon must have been leached out, have migrated and been accumulated in a very variable way, even within a small volume of the rocks, and consequently the ages must be discrepant.

It would seem that the only reliable age is that obtained from K.13 and that the age of the Deccan Traps in the Khandala region is Early Eocene as Fermor and Fox suggest.
<table>
<thead>
<tr>
<th>Reference</th>
<th>K₂O%</th>
<th>Method of measurement</th>
<th>Volume of Radiogenic argon-40 per GM of sample, per K₂O%</th>
<th>Atmospheric contamination %</th>
<th>Age M.yrs</th>
</tr>
</thead>
<tbody>
<tr>
<td>K13, 218</td>
<td>0.70</td>
<td>Deflection</td>
<td>1.99 x 10⁻³ cu.mm.</td>
<td>63.5</td>
<td>59 ± 2</td>
</tr>
<tr>
<td>K13, 218</td>
<td>0.70</td>
<td>Spike</td>
<td>2.03 x 10⁻³ cu.mm.</td>
<td>36.0</td>
<td>60 ± 2</td>
</tr>
<tr>
<td>K121,220</td>
<td>1.22</td>
<td>Deflection</td>
<td>2.55 x 10⁻³ cu.mm.</td>
<td>33.0</td>
<td>84 ± 2</td>
</tr>
<tr>
<td>K123,221</td>
<td>0.55</td>
<td>Deflection</td>
<td>1.62 x 10⁻³ cu.mm.</td>
<td>48.0</td>
<td>48.5 ± 1.5</td>
</tr>
<tr>
<td>K123,221</td>
<td>0.55</td>
<td>Spike</td>
<td>1.44 x 10⁻³ cu.mm.</td>
<td>27.0</td>
<td>43 ± 1</td>
</tr>
<tr>
<td>K128,222</td>
<td>0.86</td>
<td>Deflection</td>
<td>2.46 x 10⁻³ cu.mm.</td>
<td>48.5</td>
<td>72 ± 3</td>
</tr>
<tr>
<td>K128,222</td>
<td>0.86</td>
<td>Spike</td>
<td>1.69 x 10⁻³ cu.mm.</td>
<td>84.0</td>
<td>50.5 ± 8</td>
</tr>
<tr>
<td>K133,223</td>
<td>0.84</td>
<td>Deflection</td>
<td>1.77 x 10⁻³ cu.mm.</td>
<td>55.5</td>
<td>52 ± 2</td>
</tr>
<tr>
<td>K133,223</td>
<td>0.84</td>
<td>Spike</td>
<td>1.58 x 10⁻³ cu.mm.</td>
<td>17.0</td>
<td>47 ± 2</td>
</tr>
</tbody>
</table>
6.3. The Isle of Mull

Five samples of plateau basalts from the Isle of Mull, Scotland, were selected for age determination. All of these rocks came from the middle part of the early plateau group of lavas, the earliest flows in North Mull.

Thin sections were made of these olivine basalts and all were found to be altered to a varying extent. Sample 103/6 was the least altered and its age is probably somewhere near the correct value. 105/5 and 105/6 were moderately badly altered, and 104/5 and 106/3 badly altered.

The age determinations were all made using the 'deflection' method, the results are shown in Table 6.4.

Table 6.4

<table>
<thead>
<tr>
<th>Ref.</th>
<th>K₂O%</th>
<th>Volume of Radio-</th>
<th>Atmospheric Contamination</th>
<th>Age M.years</th>
</tr>
</thead>
<tbody>
<tr>
<td>106/3, 230</td>
<td>0.66</td>
<td>$1.20 \times 10^{-3}$</td>
<td>45%</td>
<td>36.0 ± 1.5</td>
</tr>
<tr>
<td>104/5, 227</td>
<td>0.40</td>
<td>$1.22 \times 10^{-3}$</td>
<td>73%</td>
<td>36.0 ± 2.0</td>
</tr>
<tr>
<td>103/6, 225</td>
<td>0.50</td>
<td>$2.20 \times 10^{-3}$</td>
<td>91%</td>
<td>65.0 ± 1.5</td>
</tr>
<tr>
<td>105/5, 228</td>
<td>0.44</td>
<td>$2.21 \times 10^{-3}$</td>
<td>50%</td>
<td>65.0 ± 2.5</td>
</tr>
<tr>
<td>105/6, 229</td>
<td>0.32</td>
<td>$1.80 \times 10^{-3}$</td>
<td>43.5%</td>
<td>54.0 ± 4.0</td>
</tr>
</tbody>
</table>
The results suggest that the period of volcanism occurred in the Early Palaeocene and that the period of alteration (chloritization etc.) in the Early Oligocene or later.

6.4. Antrim, Northern Ireland

In 1959, Wilson\(^{(48)}\) published a paper describing results of his palaeomagnetic studies in Antrim, Northern Ireland. This work was an extension of that done by Charlesworth and Hospers\(^{(49)}\) in the same region. The igneous rocks in this region were generally accepted as Eocene in age, but are divided into three groups.

(A) The older olivine basalts
(B) The younger tholeiitic basalts
(C) The intrusive bodies, dykes and sills

Six samples were selected from these three groups for age determinations and thin-sections made from each rock to determine the degree of alteration. A brief description of each sample is given.

A.J.I - An olivine basalt from the base of (A) with patches of alteration. The result must be discrepant, but not to any great extent.

G.VIII - A basalt from the base of (B) with very little alteration; it should give a good age.
H.III, R I - Basalts from the base of (B) with a moderate amount of subsequent alteration. The age should be discrepant.

H.II - A badly altered olivine-basalt, supposedly from the base of (B), but it would seem likely to belong to (A). Its age should be discrepant.

A.Q IIa - A very fresh olivine-dolerite sill from group (C) which should give a good age.

Table 6.5 shows the results of age determinations on these samples by the 'deflection' method.

<table>
<thead>
<tr>
<th>Ref.</th>
<th>Rock Group</th>
<th>K₂O%</th>
<th>Volume of Radiogenic Argon per gm. per K₂O%</th>
<th>Atmospheric Contamination</th>
<th>Age M.yrs</th>
</tr>
</thead>
<tbody>
<tr>
<td>AJ,I, 265</td>
<td>A</td>
<td>0.18</td>
<td>$2.22 \times 10^{-3}$ cu.mm,</td>
<td>70.5%</td>
<td>66 ± 12</td>
</tr>
<tr>
<td>G.VIII 266</td>
<td>B</td>
<td>0.76</td>
<td>$1.30 \times 10^{-3}$ cu.mm</td>
<td>63.0%</td>
<td>39 ± 1</td>
</tr>
<tr>
<td>H.III 271</td>
<td>B</td>
<td>0.57</td>
<td>$1.20 \times 10^{-3}$ cu.mm</td>
<td>34.5%</td>
<td>36 ± 3</td>
</tr>
<tr>
<td>R.I, 210</td>
<td>B</td>
<td>0.37</td>
<td>$1.04 \times 10^{-3}$ cu.mm</td>
<td>66.0%</td>
<td>31 ± 2</td>
</tr>
<tr>
<td>H.II 267</td>
<td>B?</td>
<td>0.69</td>
<td>$1.70 \times 10^{-3}$ cu.mm</td>
<td>64.0%</td>
<td>51 ± 1.5</td>
</tr>
<tr>
<td>A.QIIa 270</td>
<td>C</td>
<td>0.36</td>
<td>$0.86 \times 10^{-3}$ cu.mm</td>
<td>34.5%</td>
<td>26 ± 3</td>
</tr>
</tbody>
</table>

Group (A) - The older olivine basalts
(B) - The younger, tholeiitic basalts
(C) - The intrusives
From these results it would appear that the period of olivine-basalt volcanism occurred in the late Cretaceous to Early Palaeocene. Then followed a quiescent interval, the interbasaltic period, which was followed in the Late Eocene to Early Oligocene by the tholeiitic basalt volcanism. The period of intrusion probably occurred later, in the Early Miocene.
6.5. Recent reversed and normal basalts from Iceland

As has previously been discussed, Hospers (15) found that in a sequence of recent lavas in Iceland polarity zones occurred. Binarsson & Sigurgeirsson (50) extended the work of Hospers and collected many thousands of samples from all over Iceland. Their observations indicated that there were at least three periods during which there was a reversion of the earth's field. The last period seemed to cover the Pliocene-Pleistocene boundary as had been found by Roche (51) in France. 24 samples were supplied by Sigurgeirsson covering these three periods, and it is hoped to date these polarity epochs. The samples are designated in terms of their polarity epochs; N1 being the group corresponding to the most recent lavas which are normal, and R1 corresponding to the first most recent period of reversal. In all, 6 samples have been dated but unfortunately the samples taken from the boundary of the most recent reversal are too low in potassium for accurate measurement of the radiogenic argon content. However, in the older rocks, more argon is evolved and two transition samples from R1-N2 have been dated with consistent results. Two samples from the second period of reversal R2 have also been dated successfully.

All the samples in the Groups N1 and R1 were found to
be fresh in thin-section except for sample number 14 which was not dated. Although the groups N2 and R2 were not examined in this way, in view of the consistency of the results it would seem that no radiogenic argon had been lost from the samples measured.

Table 6.6. gives results on the rocks measured.

<table>
<thead>
<tr>
<th>Polarity Group</th>
<th>Sample No.</th>
<th>K$_2$O%</th>
<th>Volume of Radiogenic argon per Gm. of sample per K$_2$O%</th>
<th>Atmospheric contam. %</th>
<th>Age M.Yrs</th>
</tr>
</thead>
<tbody>
<tr>
<td>N1</td>
<td>1-7</td>
<td>No accurate age determinations as potassium content too low</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>R1</td>
<td>13*</td>
<td>0.312 ± 0.005</td>
<td>4.9 x $10^{-5}$ cu.mm</td>
<td>92.5</td>
<td>1.5 ± 0.4</td>
</tr>
<tr>
<td></td>
<td>13*</td>
<td></td>
<td>6.9 x $10^{-5}$ cu.mm</td>
<td>91.5</td>
<td>2.1 ± 0.3</td>
</tr>
<tr>
<td>N2</td>
<td>16*</td>
<td>0.531 ± 0.010</td>
<td>5.9 x $10^{-5}$ cu.mm</td>
<td>93.0</td>
<td>1.8 ± 0.2</td>
</tr>
<tr>
<td>R2</td>
<td>19</td>
<td>0.237 ± 0.005</td>
<td>9.6 x $10^{-5}$ cu.mm</td>
<td>93.0</td>
<td>2.9 ± 0.4</td>
</tr>
<tr>
<td>R2</td>
<td>20</td>
<td>0.584 ± 0.012</td>
<td>1.1 x $10^{-5}$ cu.mm</td>
<td>93.0</td>
<td>3.2 ± 0.5</td>
</tr>
</tbody>
</table>

*Transition sample R1-N2*
These results suggest that a reversal of the earth's field occurred at 1.8 M.yrs. as three consistent results were obtained for the transition samples R1-N2. This substantiates the results obtained by Cox et al.\(^{(52)}\) who found only two normal rocks at 1.9 M.yrs, amongst 30 other reversed rocks ranging in age from 1.0 to 2.5 M.yrs. Until many more dates have been obtained from these Iceland samples, it will be impossible to determine the lengths of the polarity epochs and compare them with the results of Cox et al.\(^{(52)}\).
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30. Westcott, M.R; Private communication.


48. Wilson, R.L; 1959, Phil. Mag. 42, 750.
ACKNOWLEDGEMENTS

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