

Numerical ages of volcanic rocks and the earliest faunal zone within the Late Precambrian of east Poland

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Abstract: U–Pb age determinations by ion microprobe on zircons from two tuff samples within the Neoproterozoic–early Cambrian successions in Poland are presented. One sample, from the Kaplonosy borehole is within, or conformably below, rocks that contain the *Sabellidites–Vendotaenia* fossil assemblage of the Upper Vendian. The second sample is from the Książ Wielki IG-1 borehole, from rocks that were referred alternatively to the early Cambrian or to the Vendian on lithostratigraphic evidence.

The Kaplonosy zircons are euhedral and free of visible zircon cores, both optically and as back-scattered electron images, but they exhibit a range in ²⁰⁶Pb/²³⁸U ages that exceeds analytical error. The combined data-set can be resolved into three age-components in different proportions, which overlap in apparent age due to measurement errors. There is a well-defined principal age component at 551 ± 4 Ma (95% limits) and two minor detrital or inherited components at 588 ± 8 Ma and 635 ± 10 Ma. The age of the Kaplonosy tuff is interpreted as equal to that of the youngest and principal component, 551 Ma. This age allows a maximum time difference of 17 ± 4 Ma between the top of the Sławatycze Formation and Lower Cambrian strata of the *Heliosphaeridium dissimilare–Skiagia ciliosa* acritarch Zone, the latter being time-equivalent to Middle Tommotian strata in northeastern Siberia, recently dated as 534.6 ± 0.5 Ma.

The tuff from the Książ Wielki IG-1 borehole has a similar spectrum of zircon ages, but also contains several detrital grains that have concordant and separate early Proterozoic and Archaean ages. The age of the major component is 549 ± 3 Ma (72% of the total), and there are two older components at 578 ± 9 Ma (13%) and 619 ± 8 Ma (15%). The deposition of the tuff from the Książ Wielki Formation therefore occurred at 549 Ma, indicating that the Książ Wielki Formation is not early Cambrian but Upper Vendian, unless all zircons in the tuff are detrital.

Keywords: Poland, Vendian, absolute age, zircon, tuff.

Radiometric dating of minerals from volcanics interbedded with sediments offers the best current prospect for obtaining accurate numerical ages of sedimentary deposition. Examples are U–Pb dating of tuffs from the Tertiary (Oberli *et al.* 1990) and Triassic (Claoué-Long *et al.* 1991), and ⁴⁰Ar–³⁹Ar dating of K-feldspars (Deino & Potts 1990). Single grains must be analysed to identify xenocrystic or detrital feldspar and zircon. We consider that the latter cannot be recognized reliably by visual inspection prior to chemical processing, and that any age determination of a combined multi-grain sample might be too old. The results reported here reinforce this belief.

Recently, the acritarch succession and biostratigraphy of the Neoproterozoic to early Cambrian sequence of the East European Platform in Poland has been documented by Moczydłowska (1991). Interbedded tuffs that contain zircons occur in the Neoproterozoic of the Lublin slope of the East European Platform and in a supposedly early Cambrian succession south of the Holy Cross Mountains in the Nida Trough. Ion microprobe U–Pb analyses of single grains and of areas distributed within grains have shown that some of the zircons are much older xenocrysts or detrital grains. The remainder have been revealed as a dominant zircon

population that is mixed with one or more earlier populations that are only a few percent older. The presence of mixed age populations is indicated both by the total range of the data and by its frequency distribution. The means and uncertainties of the several populations have been estimated by a newly-developed method based on maximum-likelihood statistical techniques (Sambridge & Compston 1994). This type of mixture-modelling enhances the assessment of age results in general and especially that for ion microprobe ²⁰⁶Pb/²³⁸U ages, which for zircons younger than about 800 Ma usually cannot be corroborated by ²⁰⁷Pb/²⁰⁶Pb ages because of their insufficient precision.

The earliest fossil record of eastern Newfoundland (the global stratotype section for the Precambrian–Cambrian boundary) and Baltica display numerous similarities. Isotopic dating of a tuff layer at the top of the Sławatycze volcanogenic complex in the Lublin slope places a confident older limit on the numerical age of the late Vendian *Sabellidites–Vendotaenia* Zone. It also contributes to the interregional correlations between Baltica and Western Avalonia and to testing time-correlations based on secular changes in seawater chemistry recorded by stable isotopes and ⁸⁷Sr/⁸⁶Sr.

Geological background and sample locations

Tuff from the top of the Sławatycze Formation, PL-92-77

The Neoproterozoic–early Cambrian succession in the Lublin slope of the East European Platform in eastern Poland (Fig. 1) forms the basal portion of the platform cover overlying the early Proterozoic basement (Mansfield *et al.* 1993). Subsidence within the Lublin slope resulted in the formation of a basin whose initial infilling consists of feldspathic and quartzitic sandstones of the Polesie Formation (Fig. 2), which accumulated under continental and lagoonal conditions (Juskowiakowa 1974). The timing of this early depositional event is poorly constrained, but on uncertain lithostratigraphic grounds, the Polesie Formation is considered to have accumulated during late Riphean times (Areń *et al.* 1979). A second pulse of continental detrital deposition forms the base of the Sławatycze Formation (Fig. 2), and this is succeeded by a complex volcanogenic unit that constitutes the bulk of the Formation. The Sławatycze Formation rests disconformably either on the Polesie Formation or on the crystalline basement. It varies in thickness from 34 to 372 m over the Lublin slope, and

occupies a substantial area of the East European Platform (Fig. 1). On lithostratigraphic grounds alone, the age of both the Sławatycze Formation and its presumed lateral correlative, the volcanogenic Volhyn Group in the Ukraine, Belarus and Moldava, was regarded as early Vendian (Juskowiakowa 1971; Sokolov & Fedonkin 1990).

Until recently, the Sławatycze Formation was thought to be overlain disconformably by the Siemiatycze Formation and its lateral equivalent, the Białopole Formation (Areń *et al.* 1979; Lendzion 1983a) which developed in different facies in the northern and southern regions of the Lublin slope. However, examination by two of us (Vidal & Moczyłowska) in the Kaplonosy IG-1, Białopole IG-1 and Busówno IG-1 boreholes has shown that the tuffs of the Sławatycze Formation (Fig. 2) grade without break into the overlying mudstones at the base of the lower Białopole Formation in the Białopole IG-1 borehole (Vidal & Moczyłowska in press). The volcanogenic unit is overlain by a transgressive marine succession of siliciclastic rocks (Fig. 2) that span from the Upper Vendian to the Lower Cambrian (Moczyłowska 1991). The above relative ages are based on exhaustive palaeontological evidence (Lendzion 1983b; Paczeńska 1986; Moczyłowska 1991) that allows recognition of the Neoproterozoic/Cambrian boundary and

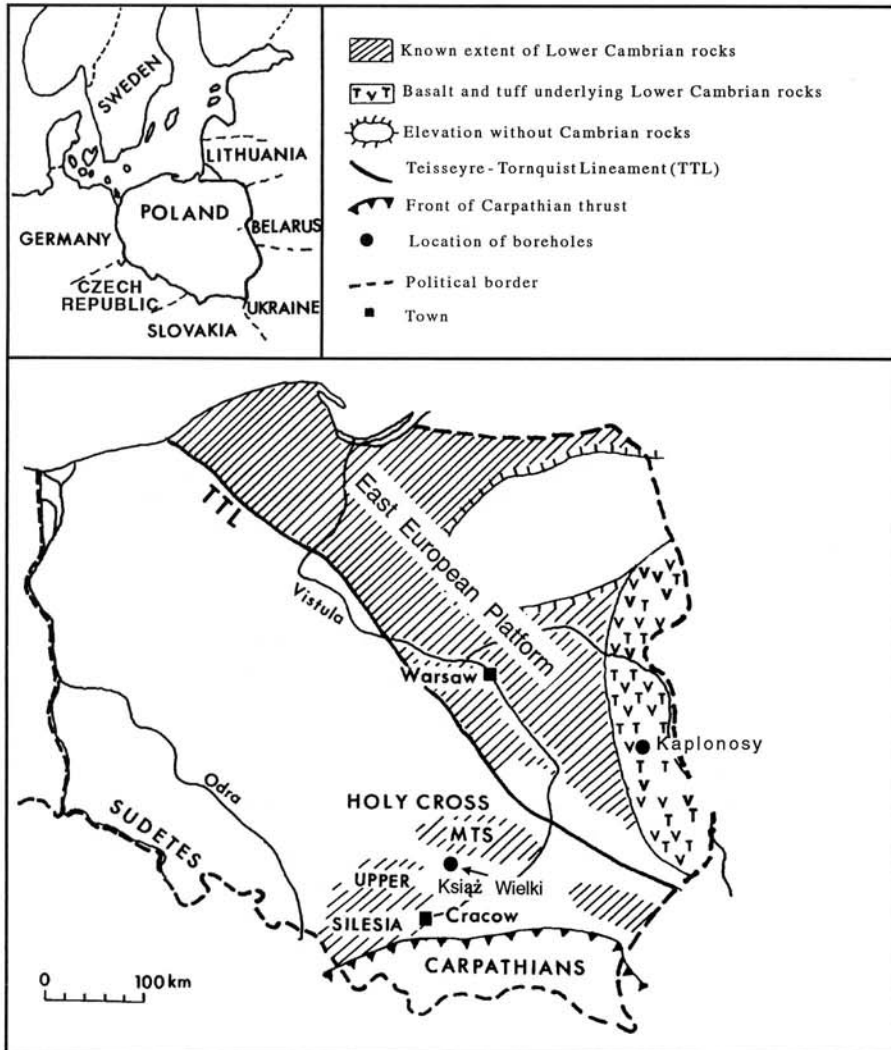


Fig. 1. Geological sketch-map showing occurrence of Lower Cambrian sedimentary rocks and Neoproterozoic volcanic rocks in Poland. The Kaplonosy borehole is located in the Lublin slope of the East European Platform. Modified after Juskowiakowa (1971) and Moczyłowska (1991).

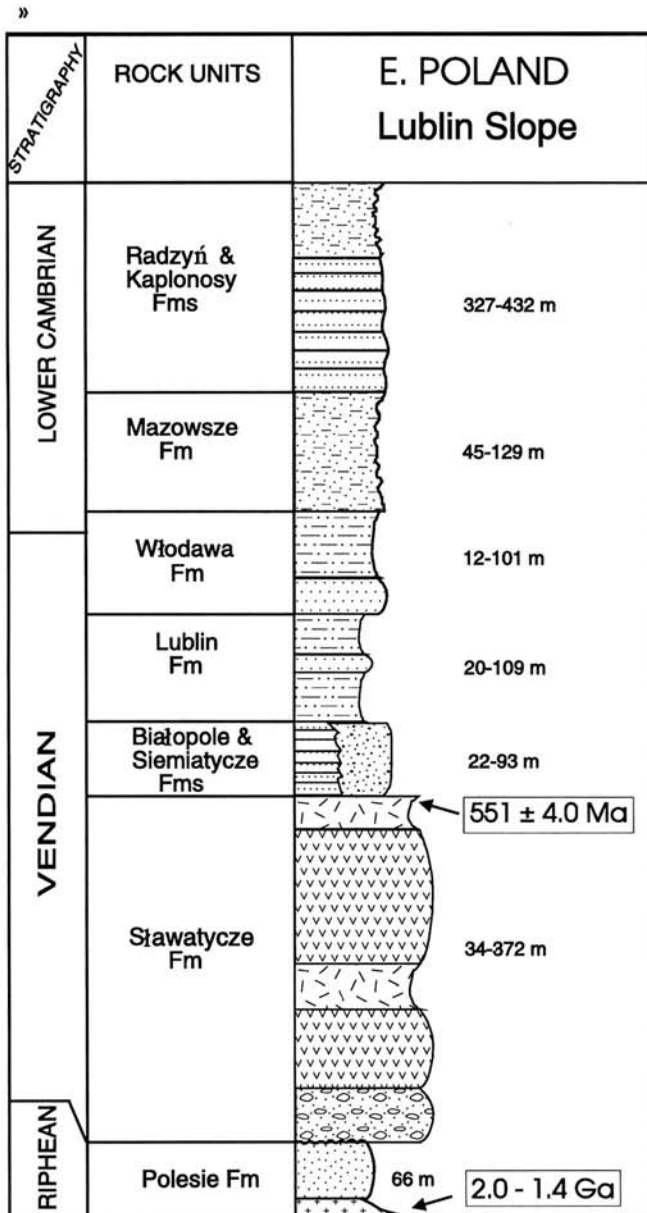


Fig. 2. Simplified lithostratigraphy of the Proterozoic–Lower Cambrian succession in the Lublin slope (East European Platform). Modified after Moczyłowska 1991. U–Pb ages on zircons from crystalline basement rocks are according to Mansfeld *et al.* (1993). The stratigraphic position of a dated tuff from the Sławatycze Formation (this paper) is indicated.

detailed biostratigraphic correlation with successions elsewhere in Baltica (Moczyłowska 1991).

The selected Vendian tuff is located about 113 m below the palaeontologically established Precambrian–Cambrian boundary (Moczyłowska 1991). The dated tuff sample (PL-92-77) was collected at the depth of 1455.5 m in the Kaplonosy IG-1 borehole from a 1 m thick layer of tuff overlying basalts and underlying the topmost portion of agglomerates and tuffs with thin interbeds of brecciated lavas (Fig. 3). Its major and trace element chemistry as determined by plasma-quad mass-spectrometry is shown in Table 1. A felsic to intermediate composition is indicated for

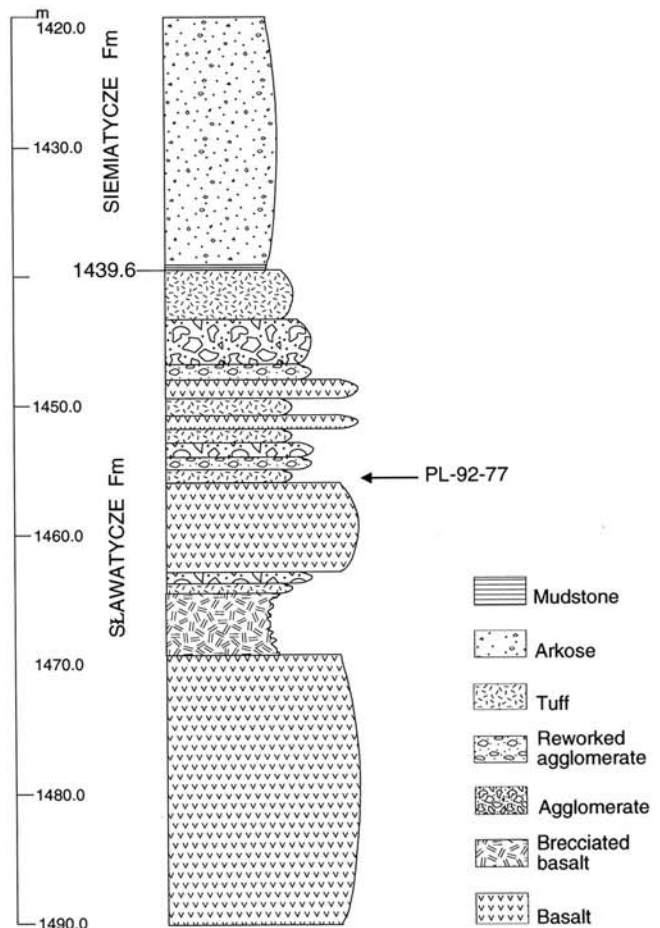


Fig. 3. Stratigraphic section of the upper Sławatycze Formation in the Kaplonosy IG-1 borehole showing sample of dated tuff layer (PL-92-77) at depth of 1455.5 m. Geological log prepared from data in Documentation of the Kaplonosy IG-1 borehole, State Geological Institute, Warsaw.

Table 1. Chemical composition of tuff sample 92-77 in the Sławatycze Formation, at 1455.5 m depth in the Kaplonosy IG-1 borehole

SiO ₂ (%)	61.0 ± 0.1	Ba (ppm)	203 ± 1	Sn	<11.8
TiO ₂	1.28 ± 0.01	Be	3.85 ± 0.07	Sr	38.9 ± 0.1
Al ₂ O ₃	15.1 ± 0.1	Co	20.4 ± 0.6	V	53.0 ± 0.1
Fe ₂ O ₃	11.2 ± 0.1	Cr	57.0 ± 3.0	W	12.2 ± 2.2
MnO	0.0194 ± 0.0001	La	110 ± 1	T	49.6 ± 0.2
MgO	2 ± 0.01	Mo	<5.9	Yb	6.72 ± 0.01
CaO	0.667 ± 0.002	Nb	54.9 ± 0.3	Zn	36.8 ± 0.1
Na ₂ O	0.515 ± 0.001	Ni	55.7 ± 1.8	Zr	421 ± 1
K ₂ O	6.34 ± 0.01	Sc	15.2 ± 0.1		
P ₂ O ₅	0.0971 ± 0.0009				

the tuff, so that precipitation of zircon from the tuff-magma as a liquidus phase would be possible.

Supposed early Cambrian tuff from the Książ Wielki Formation, PL-92-16

Tuffs and bentonites are frequently recorded within the Cambrian siliciclastic succession in the Holy Cross

Mountains and the Nida Trough in southern Poland (Fig. 1). The Cambrian age is either demonstrated by fossil evidence (in the Holy Cross Mountains) or inferred from lithostratigraphy (in the Nida Trough). Tuffs occur within various formations as thin tuffaceous-detrital layers a few cm in thickness. The source of the pyroclastic rocks is unknown, but it can be inferred that the source area of active Cambrian volcanism was situated south of the Holy Cross Mountains, at present buried under the Carpathian thrusts (Kowalczewski 1990). The East European Platform is excluded as a possible source area, as the volcanic activity there had definitely ended before the early Cambrian. But in view of the currently established late Vendian age of tuffs in the Książ Wielki Formation, the East European Platform might be a source area for at least late Vendian tuff units in the Nida Trough.

Thin interbeds of tuffs occur throughout a succession of greywackes in the Książ Wielki Formation in the Książ Wielki IG-1 borehole (Kowalczewski 1981; Fig. 4). The Książ Wielki Formation has a true dip thickness of about 350 m and consists of two informal members. The lower member comprises shales and sandstones that occupy the interval between 1773.0 and 1936.0 m, whereas the upper member consists of greywackes with tuffs at the depth of 1273.0 to 1773.0 m (Kowalczewski 1981). Recently an early Cambrian age was proposed for the Książ Wielki Formation

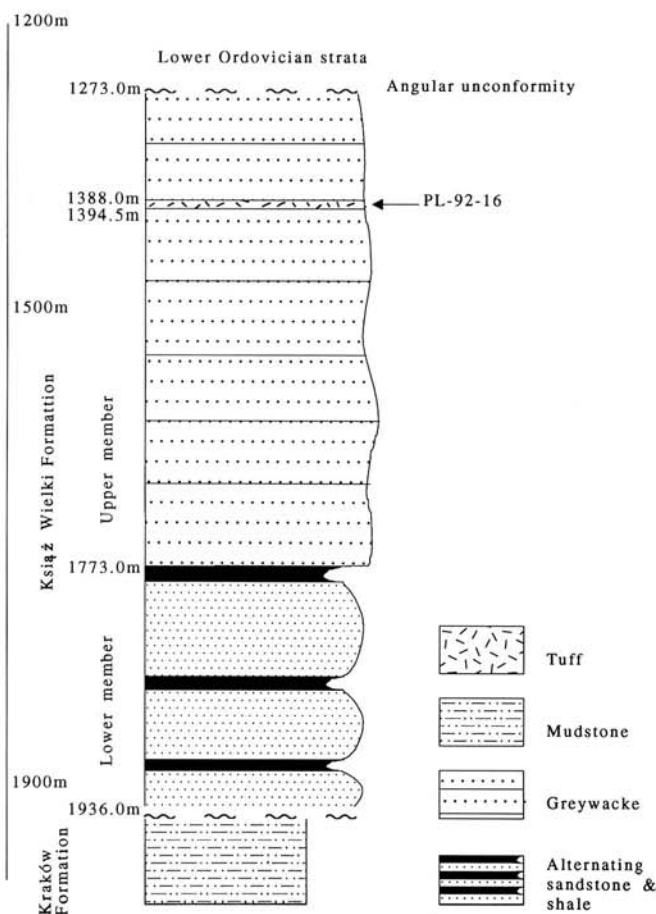


Fig. 4. Simplified stratigraphic section of the Książ Wielki Formation in the Książ Wielki IG-1 borehole and position of dated tuff sample (PL-92-16). Modified after Kowalczewski (1981).

(Kowalczewski 1990), based entirely on lithostratigraphic grounds. However, a Vendian age was formerly proposed for the formation on the grounds of inferred regional stratigraphic relationships (Kowalczewski 1979; Jurkiewicz 1980; Kowalski 1983) and is supported by our study.

The glassy and crystal tuffs of the Książ Wielki Formation are believed to derive from an intermediate dacite and andesite type of volcanism (Kowalczewski 1981, 1990). A sample for isotopic dating (PL-92-16) was collected from the fine-grained tuff interbedded with sandstones at the depth interval of 1388.0 to 1394.5 m in the Książ Wielki IG-1 borehole (Fig. 4).

Analytical procedures and data reduction

The data reported here were obtained using the SHRIMP II ion microprobe during the first months of its operation. The instrument was used over a range of conditions as regards elemental discrimination between Pb^+ , U^+ and Zr_2O^+ ; sensitivity for collected Pb^+ , both as total cps/ppm and cps/ppm/nA of primary beam; and choice of O_1^- or O_2^- for the primary beam. However, all values of reduced $^{206}Pb/^{238}U$ for the sample were referenced to interspersed analyses of the same fragment of the SL13 zircon standard, and operating conditions per single analytical session were kept constant. The age patterns observed between samples were found to be independent of operating conditions. The mass-resolution was between 5000 and 6000 (1% definition), and Pb hydrides were usually undetectable. Our final preferred conditions were probe diameter 15 μm , O_2^- primary beam at between 2 and 3 nA, and sensitivity 20 cps/ppm Pb/nAO_2^- .

Data-reduction procedures have been extended from those described for geological time-scale work by Compston *et al.* (1992), to deal with a sporadic dependence of the observed Pb^+/U^+ upon the Zr_2O^+ count-rate which was experienced during the initial adjustment of the SHRIMP II instrument, in addition to its usual dependence on UO^+/U^+ . These data for the standard per session (expressed as logarithms) were generally well-fitted to a plane, so that Pb^+/U^+ for the standard could be predicted for the co-ordinates of each sample analysis. The precision for the $^{206}Pb/^{238}U$ ages can be estimated from the Pb^+/U^+ residuals of the standard plane, or when the Zr_2O^+ effect was absent, from the dispersion of Pb^+/U^+ for the standard normalised to a fixed UO^+/U^+ . This includes the problem of identifying occasional 'outliers' in the data per session, due either to unknown analytical factors or to real variation within the SL13 standard caused by local Pb loss and local Pb inheritance. Cumulative probability plots for the ages of the standard were used to assist with this. Table 2 lists the supposed outliers, giving also the number of analysis points on the standard, the standard deviation in reduced $^{206}Pb/^{238}U$ per analysis, and the presence or absence of Zr_2O^+ discrimination. The pooled standard deviation for these nine sessions is 2.0%, which has been used in assessing all the samples.

Common Pb in the analysis is due both to surface-related Pb contamination acquired during polishing the grain-mount, and to common Pb within the zircon lattice and in sub-micron-sized inclusions within the grains. The former was minimized by rastering the primary beam over the selected area for several minutes prior to analysis. Many of the analysed grains have rather low U contents and hence low radiogenic Pb contents. Although the ratio of common ^{206}Pb to total ^{206}Pb varies widely, its median value is 3.6% and most $^{206}Pb/^{238}U$ ages are insensitive to error from this source. However the correction for common Pb is crucial for estimates of the radiogenic $^{207}Pb/^{206}Pb$.

The fraction of common ^{206}Pb in the total $^{207}Pb/^{206}Pb$ in each analysed area, denoted as f , has been measured in three separate ways: via ^{204}Pb , ^{207}Pb and ^{208}Pb isotopes respectively (Compston *et al.* 1984, 1992). Of these, the 204-method has proved to be the least precise owing to large counting-errors arising from the very low ^{204}Pb count-rate, but values for the 208-corrected and 207-corrected f for these samples are well-correlated. Consequently we employed the 208-method to obtain estimates of the radiogenic $^{207}Pb/^{206}Pb$

Table 2. Discrimination conditions and precision per analytical session for the 'calibration' of zircon standard SL13. The plane-fit procedure only was used for sessions showing correlation of age with Zr_2O^+ count-rate

Session	Correlated with Zr_2O^+ cps?	Adjustment methods	Analyses	'Outliers'	Standard deviation %
1 (24.9.92)	Yes	Normalized	13	6	2.5
		Plane-fit	13	0	2.8
2 (26.9.92)	Yes	Normalized	7	0	1.0
		Plane-fit	7	0	0.5
3 (28.9.92)	Yes	Normalized	10	3	2.8
		Plane-fit	10	0	2.4
4 (6.10.92)	Yes	Normalized	14	1	2.5
		Plane-fit	14	0	2.0
5 (20.6.93)	No	Power-law	12	1	1.3
6 (21.6.93)	No	Power-law	17	0	2.0
7 (22.6.93)	No	Power-law	11	4	1.7
8 (6.7.93)	No	Power-law	23	1	3.0
9 (27.7.93)	No	Power-law	19	1	2.1
Pooled standard deviation:					2.0

ratios of the analysed areas and their corresponding radiogenic $^{238}U/^{206}Pb$. We have used the 207-method as well, viewing it as probably the most reliable for $^{206}Pb/^{238}U$ ages because it does not depend on chemical closure of Th/U in the analysed areas.

It is essential to know the limits of the SHRIMP ion microprobe's reproducibility in $^{206}Pb/^{238}U$ age measurement, in order to assess the question of possible inheritance and Pb loss in the samples. This has been done here through the combined analyses of the SL13 standard zircon taken during the relevant analytical sessions. Figure 5a is a probability plot for the combined 125 measurements of $^{206}Pb/^{238}U$ in the SL13 zircon taken during nine separate analytical sessions, with no deletions for 'outliers'. As determined by isotope-dilution, the $^{206}Pb/^{238}U$ age of SL13 is uniform on the one milligram scale at 572 Ma. If its $^{206}Pb/^{238}U$ is uniform also on the one nanogram scale, the ion probe measurements will be dispersed solely by (Gaussian) experimental error, and the data in the probability plot should be well-fitted to a straight line. There is a well-defined linear central trend but the extremes are non-linear. The latter requires explanation, such as localized Pb loss in a few areas for the low ages or localized ^{206}Pb excess for the high ages. Both of these effects are known to occur very rarely in SL13, and will be documented elsewhere. If the extremes of the data are culled to exclude values older than 595 Ma and younger than 550 Ma, a good straight-line is found (Fig. 5b) with average value 572.1 Ma and standard deviation per analysis of 1.5%. Without culling, the Fig. 5a data-set gives a mean age of 570.8 Ma but with the standard deviation indicated as 2.9%. Outliers may be 'identified' during assessment of the ages in each session (Table 2) as those whose standardized residuals relative to the fitted line exceed say, 2.5.

The numerical uncertainties given within the main text will be the standard errors. Those in the abstract were given at the 95% confidence intervals (95% limits).

All data here are presented graphically but they are available also in the form of numerical tables as Supplementary Publication Number SUP 18095 (14 pp), which has been deposited with the Society Library and the British Library Document Supply Centre, Boston Spa, W Yorkshire, UK.

Results and interpretations

Sample PL-92-77, Kaplonosy drill-core

Initially, 66 $^{206}Pb/^{238}U$ ages were determined on 22 grains, which collectively showed a total range in value in excess of

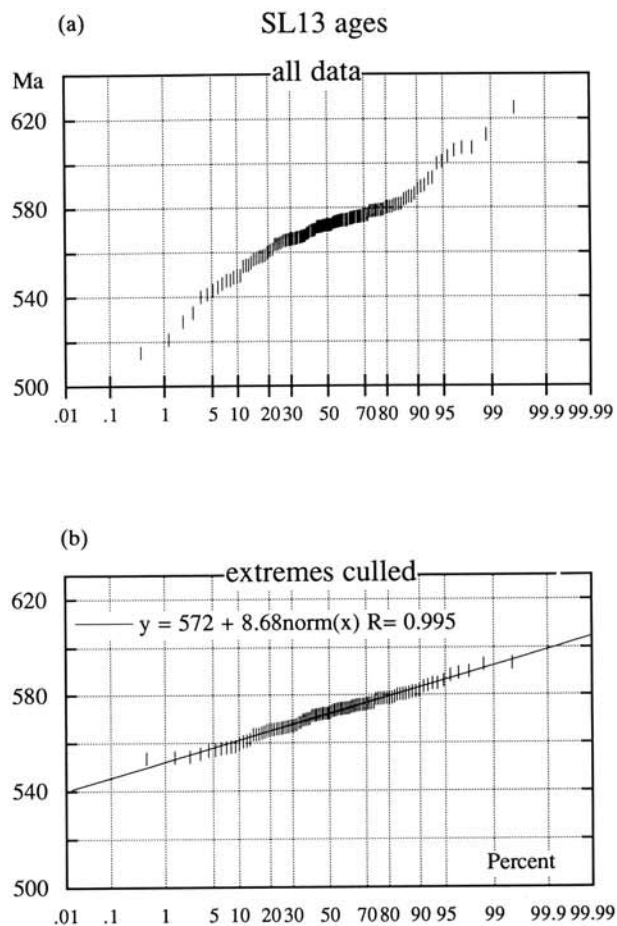


Fig. 5. (a) Probability plot for 125 $^{206}Pb/^{238}U$ analyses of the standard zircon SL13 taken during the measurements on samples 92-77 and 92-16. The non-linear extremes reflect uncommon but real variations in the internal zircon composition on a 20 μm spatial scale. (b) Probability plot for 103 $^{206}Pb/^{238}U$ analyses of zircon SL13 from the central part of Fig. 5a, with more dispersed data 'culled'. The good linearity is consistent with a single Gaussian distribution, and the 1.5% standard deviation observed is 'normal' for the measurement process.

that expected for a single age population (coefficient of variation per measurement *c.* 2%). This raised the hypothesis that the zircons contained a population of older detrital or xenocrystic grains in addition to the tuff-derived population. The zircon morphology was uniform and no internal structures were evident. Consequently, the whole data-set was subjected to detailed statistical assessment using the mixture-modelling method of Sambridge & Compston (1994).

This procedure represents an extension of the approach developed by Galbraith & Green (1990) for estimating multiple components from a set of measurements, in this case zircon ages, in the presence of experimental error. To apply the procedure to a data-set, one must assume a distinct number of age components and a distribution for the statistical errors involved. Since both of these quantities are unknown, Sambridge & Compston (1994) suggest repeating the estimation procedure (on the same data) assuming different numbers of components, n_c , and types of

Table 3. The results of applying mixture modeling to $66\ ^{206}\text{Pb}/^{238}\text{U}$ ages from the Kaplonosy drill-core. (Best solution from 50 trials)

Experiment	p value	Class	Age	σ	Proportion	σ	Misfit value
R1	2.0	1	574.7	2.1	1.0		93.61
R2	2.0	1	603.6	5.0	0.40		69.0
		2	558.2	3.5	0.60	0.1	
R3	2.0	1	630.8	10.6	0.10		
		2	551.0	6.7	0.39	0.2	
		3	585.1	6.8	0.51	0.2	
R4	2.0	1	550.3	–	0.00		66.32
		2	630.8	–	0.10	–	
		3	551.0	–	0.39	–	
		4	585.2	–	0.51	–	
R5	1.25	1	573.0	2.6	1.0		93.61
R6	1.25	1	605.1	5.3	0.40		69.00
		2	564.1	2.6	0.60	0.1	
R7	1.25	1	537.9	7.1	0.06		66.32
		2	607.4	4.4	0.25	0.1	
		3	567.1	2.7	0.69	0.1	
R8	1.25	1	641.0	17.0	0.05		66.32
		2	540.5	7.0	0.11	0.1	
		3	597.6	4.7	0.23	0.1	
		4	566.7	3.8	0.61	0.2	

error distribution, and then comparing the results. Maximum-likelihood ages and proportions were determined assuming up to four distinct age components, and the estimation procedure was performed for both an assumed Gaussian distribution (2.0 for p , Sambridge & Compston 1994) and for a more robust, longer-tailed distribution (1.25 for p). The parameter p ($1 \leq p \leq 2$) determines the nature of the error distribution. The two extreme values correspond respectively to a Gaussian ($p = 2$) and a more robust double exponential ($p = 1$) distribution. All intermediate values are possible and the character of the distribution varies accordingly. For each value of n_c and p , the estimation procedure was repeated from 50 random starting values, and a solution was obtained from each starting value after 50 iterations for the two assumed distributions. In all cases, 50 iterations was sufficient to ensure convergence.

The solution with the lowest misfit for each value of n_c and p is shown in Table 3. The misfit is a dimensionless number used to judge the relative fit to the data of different sets of ages and proportion parameters. A full statistical definition is given in Sambridge & Compston (1994). The proportion parameters are unknowns to be estimated and represent the proportion of the data associated with each age group. The starting points, solutions and confidence ellipses for each age/age pair are shown in Figs 6 & 7 for the Gaussian case. In each figure the widely spread set of starting points have all converged on the same region of the diagram (crosses). This indicates that the iterative numerical procedure has successfully identified the maximum-likelihood region of the ages and proportions. The misfit of the best of the 50 solutions is plotted against the number of age components in Fig. 8 (grey triangles). All 50 solutions have converged to the same values, and are indistinguishable from one another in Fig. 6. The 95% confidence ellipse about the two ages is small and does not intersect the equal-age line, indicating a high probability that the two age

components are distinct. When three components are assumed, the misfit is marginally improved (see Fig. 8), although the confidence ellipses are now larger and two are close to the diagonal equal-age line (Fig. 7). When four components are assumed, there is no improvement in misfit reduction and Table 3 (see runs labelled R3 and R4) shows that the best four-component solution is virtually identical to the three-component solution. (The first age has converged to the same as the third age but with a zero proportion parameter.) There is therefore no evidence for a fourth component, but there appears to be some support for three distinct components from the age/age diagram (Fig. 7). However the proportion value for the highest age (630 Ma) is only 0.1, which suggests that the third component is at best representing only 10% of the data. This has been confirmed using the procedure described in Sambridge & Compston (1994) to estimate which zircon ages belong to which age component. This classification procedure is separate from the initial estimation of the maximum-likelihood ages and proportions. For each measured age and age-component pair, it involves calculating the probability that the observation belongs to that component. Note that we cannot exactly determine to which component the measured age belongs, but only the probabilities associated with each component. The classification is made by assigning each observation to the component for which it has the highest probability. The data classified as belonging to the highest age component are the seven different measurements with age greater than 610 Ma (see the histogram in Fig. 9). This is too small a number to be fitted to any distribution and so the support for a third component is very weak. Figure 9 shows the classified data for the two-component case together with the estimated distributions (R2 in Table 3). Again the combined distribution in the lowest panel seems to provide a reasonable fit to the data. The peak in the distribution at around 560 Ma and

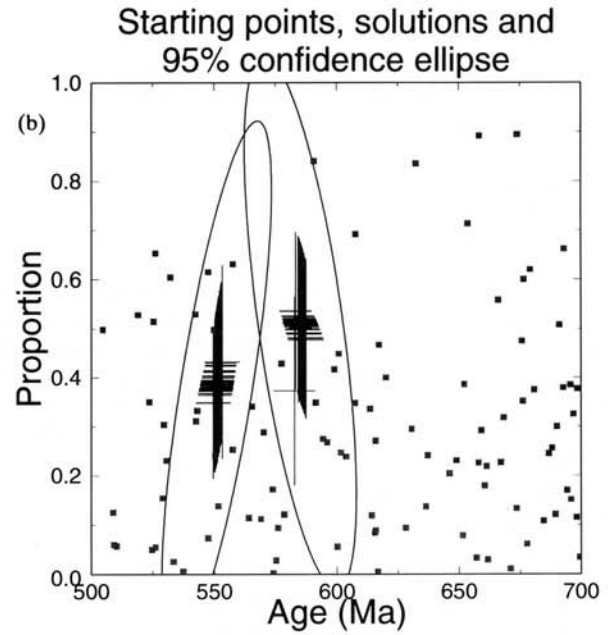
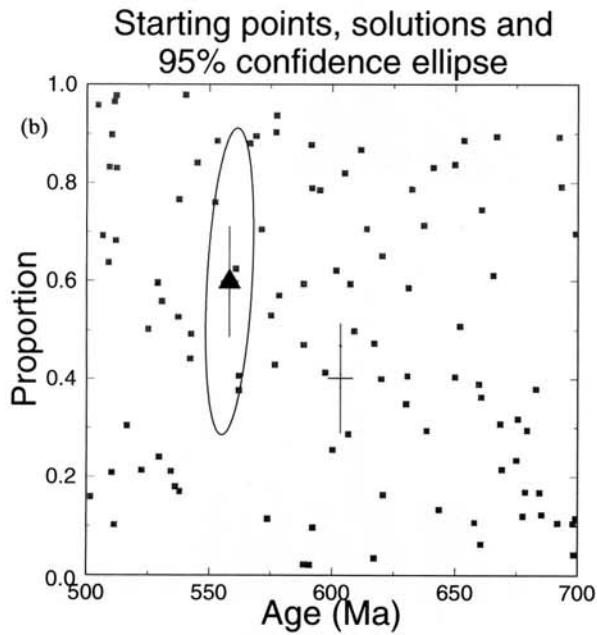
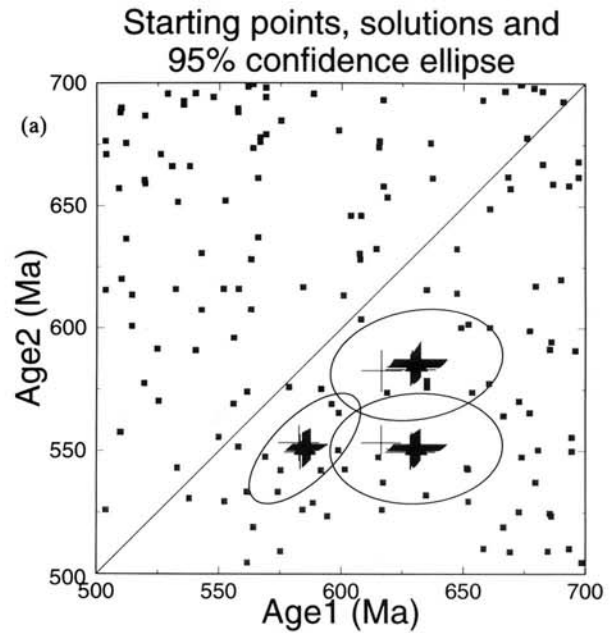
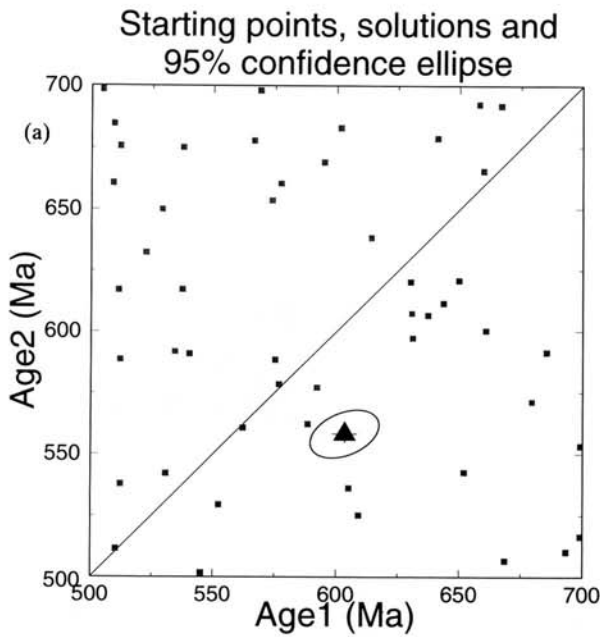


Fig. 6. (a) The starting points (grey squares), final solutions (crosses) and 95% confidence ellipse about the best of the 50 solutions (black triangle). Two age components have been assumed. All solutions lie on top of one another and are obscured by the triangle. The 95% confidence ellipse is clearly distinct from the equal age line indicating that the two ages are likely to be distinct. (b) Same as for (a) but showing both age-proportion pairs.

Fig. 7. (a) As for Fig. 6 but assuming three age components. The ellipses intersect but the crosses are tightly grouped. (b) Same as for (a) but showing both age-proportion pairs. The ellipses are inclined steeply and are unlikely to become distinct until the significance level is reduced considerably.

plateau between 580 and 600 Ma are both fitted well by the distribution.

The results for the more robust statistics (p at 1.25) are similar to the Gaussian case. For two components, the proportions are identical and the ages are very similar (compare R2 with R6 in Table 3). As the number of components is increased, the two main components are reproduced. The lower age in the two-component case

(564 Ma line labelled R6 in Table 3) reappears as 567 Ma in the three- and four-component cases (R7 and R8). A similar effect occurs with the higher age. For the three- and four-component cases, extra solutions are introduced that are not copies of the original pair, but lie instead at either end of the data range. They also have the largest standard errors and smallest proportion parameters. The role of the extra 'minor' solutions is merely to adjust the tails of the combined distribution in Fig. 9, which is still dominated by the two main solutions.

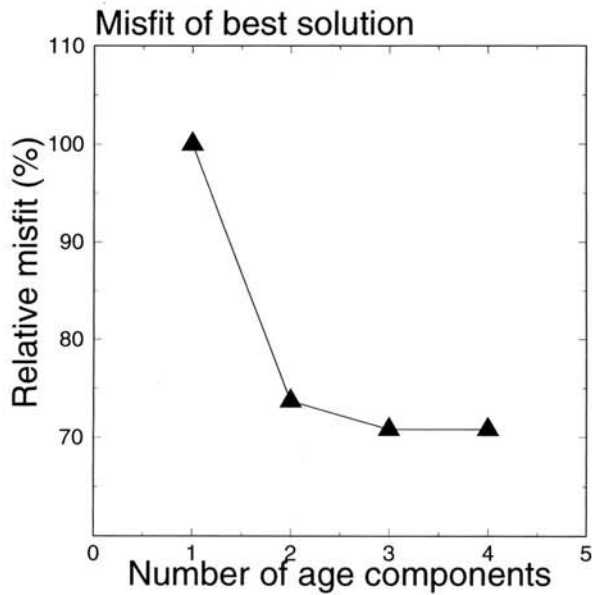


Fig. 8. Misfit of the best of 50 solutions against number of assumed age components for the data. There is only a slight improvement in the fit for more than two components.

Subsequent to the above statistical assessment, a further 37 $^{206}\text{Pb}/^{238}\text{U}$ ages were measured on grains having the higher U contents, which are the more favourable for precision. All 105 results are shown combined in Fig. 10a as the linearized cumulative probability distribution. The latter is an alternative and preferred presentation to a histogram plot. If there were a single age for the zircons dispersed only by Gaussian analytical error, the data would trend along a straight line in Fig. 10a with no non-analytical scatter. Instead, the correlation found is distinctly non-linear and the total range observed for the ages is well beyond that expected for the *c.* 2% standard deviation per analysis, both of which re-emphasize the previous conclusion that zircons having two or more different ages must be present. Application again of the mixture-modelling program yielded the same stable value of 551.0 ± 2.0 Ma for 57% of the population, 588.4 ± 3.5 Ma for 37%, and 635 ± 7 Ma for the remaining 6%. The reality of the latter remains subject to doubt as described in the detailed statistical assessment already given. The discrete age-groups are consistent with the simple picture of one and possibly two ages of detrital grains in addition to the tuff age at 551 Ma.

Examining the analyses on a grain-by-grain basis (Fig. 10b), several individual grains (#4, 19, 29 and 30) were found to contain almost the complete range of measured ages. The two inferred age-groups are therefore present within single grains, which requires a different geological interpretation than the simple mixing of detrital and tuff-related grains. Back-scattered electron imaging gave no sign of internal zircon cores, showing instead their generally uniform chemistry and unzoned structure. One grain only, #30, is structured with an inner euhedral zone overlain by a more massive outer zone. The implications of the age variation within grains will be discussed later.

The U and Th contents of the zircons are displayed on the probability plot Fig. 11, which shows a bimodal distribution for both elements. The latter does not correlate with the two principal $^{206}\text{Pb}/^{238}\text{U}$ ages.

Classification of zircon ages

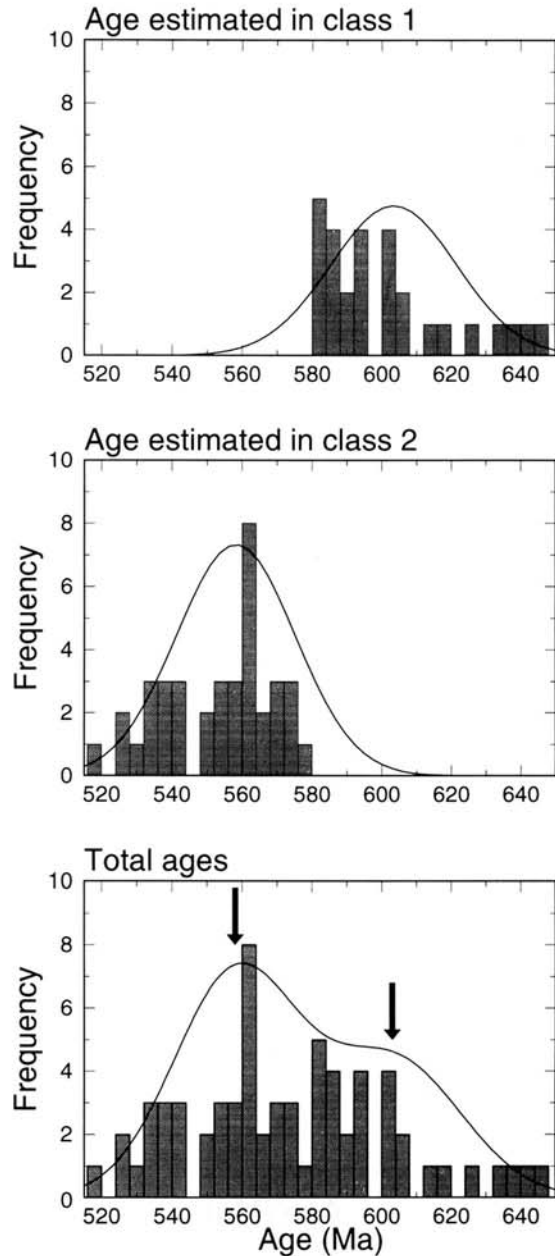


Fig. 9. Top and middle panels show histograms of the raw (synthetic) data classified into two groups according to the final solutions (shown as Gaussian distributions). The data are classified accurately except for the age range where the two overlap. Nevertheless the combined distribution (bottom panel) seems to model the combined histogram well. The arrows indicate the two true ages.

Concordia representation and $^{207}\text{Pb}/^{206}\text{Pb}$ ages for the Kaplonosy sample

Figure 12a shows the Kaplonosy zircon data, uncorrected for common Pb, plotted in terms of their $^{207}\text{Pb}/^{206}\text{Pb}$ and $^{238}\text{U}/^{206}\text{Pb}$ co-ordinates (Tera & Wasserburg 1972). Each point in Fig. 12a can be viewed as a blend between common

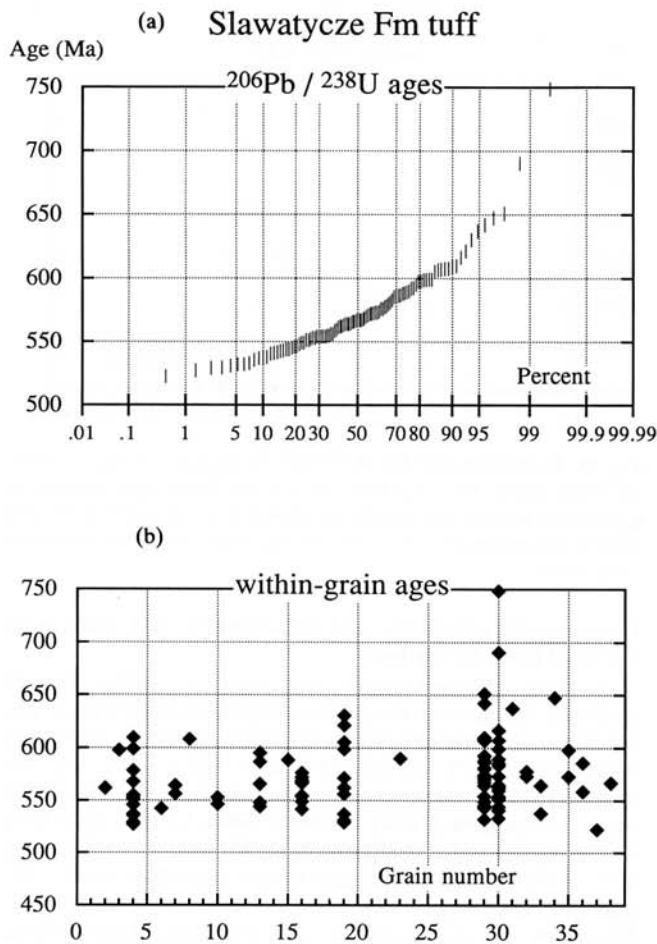


Fig. 10. (a) Probability plot for 104 $^{206}\text{Pb}/^{238}\text{U}$ analyses of zircons from the Kaplonosy borehole tuff within the Slawatyce Formation, sample 92-77. A well-fitted straight line is expected on this diagram if all grains have the same age and the data are dispersed solely by Gaussian errors of measurement. Statistical modelling finds two main age populations, at 551 Ma and 588 Ma. (b) Multiple $^{206}\text{Pb}/^{238}\text{U}$ analyses on single grains from sample 92-77 zircons, showing substantial dispersion in apparent age within particular grains.

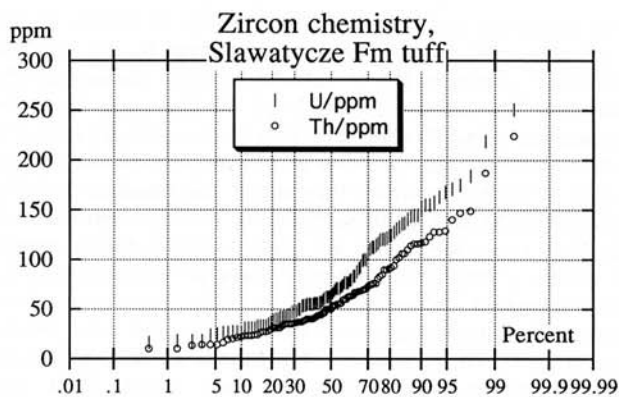


Fig. 11. Probability plots for the U and Th contents of the 92-77 zircons. The form of the curves indicates that there are two principal geochemical populations, one averaging c. 100 ppm U and the other 60 ppm. There is no correlation of these with age or zircon morphology.

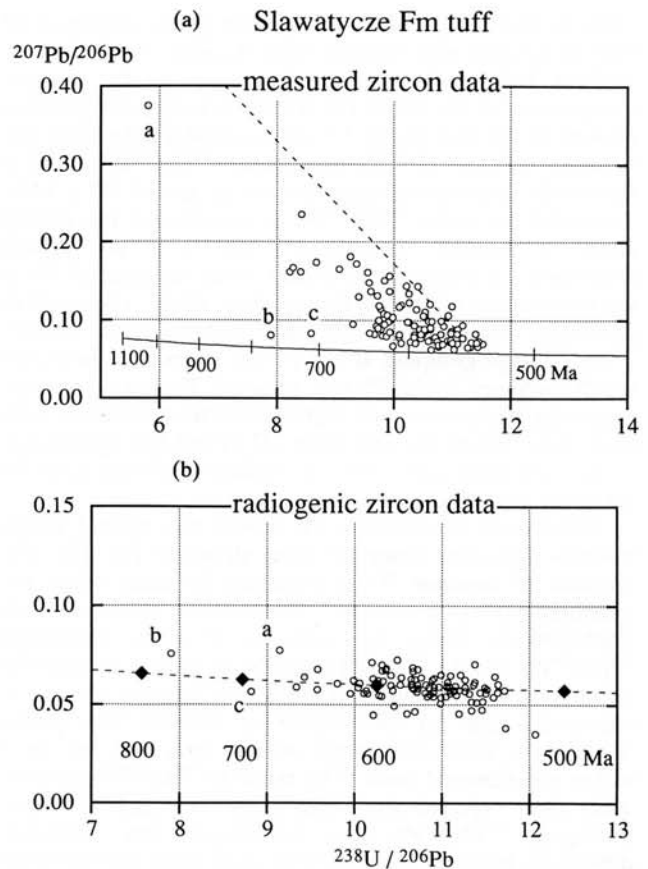


Fig. 12. (a) Tera-Wasserburg plot of the measured zircon data before subtraction of common Pb. The line shown is the locus for admixture of 'ordinary' common Pb with 540 Ma-old radiogenic Pb. Analyses b, grain 30.9, and c, 30.8, to the left of the main distribution must be older than the latter regardless of the common Pb subtraction. Analysis a, 34.1, will be older also, unless it contains an anomalous initial Pb. (b) Tera-Wasserburg plot after subtraction of 'ordinary' common Pb using the 208-method. All the data are within error of the Concordia curve and dispersed along it. Analyses a, b, and c represent the same samples as shown in (a).

Pb characterized by its value for $^{207}\text{Pb}/^{206}\text{Pb}$ at zero $^{238}\text{U}/^{206}\text{Pb}$, and the particular radiogenic Pb on the Concordia curve that specifies the age of the zircon. The locus for mixtures between 550 Ma radiogenic Pb and Broken Hill Pb is shown. Mixtures with any other 'crustal' common Pb, as modelled for example by Cumming & Richards (1975) will have a similar locus. For the simplest situation, in which all zircons have the same age but variable amounts of the same common Pb, all analyses would lie on a single mixing-line to within analytical error. The Kaplonosy data are obviously not simple. Figure 12a shows that those analyses such as 30.9 and 30.8, which are close to Concordia but well to the left hand side of the main group, must be older than the rest.

Grain 34 is the most influenced of all grains by admixture with common Pb. This is because it has the lowest U (16 ppm) and hence lowest radiogenic Pb content, and simultaneously the highest content of common ^{206}Pb (1.0 ppm). The latter is unlikely to be due to residual surface contamination because all other analyses for that session

(Day 3) were much lower in common ^{206}Pb , ranging from 0.15 to 0.3 ppm with median value 0.2 ppm. Consequently, analysis 34.1 provides valid information on the isotopic composition of the initial Pb. An error-weighted regression of most of the data in Fig. 12a gives a well-fitted mixing-line between 0.750 ± 0.027 for the initial $^{207}\text{Pb}/^{206}\text{Pb}$, and a Concordia intersection equivalent to an age of 547 ± 3 Ma. The value for initial $^{207}\text{Pb}/^{206}\text{Pb}$ is well beyond the normal range of 'crustal' common Pb but it is not grossly improbable: it would be described as an 'anomalous' Pb in the terminology of Russell & Farquhar (1960). The age is in permissive agreement with the slightly more precise $^{206}\text{Pb}/^{238}\text{U}$ age-grouping at 551 ± 2 Ma described above, for which 'ordinary' initial Pb was assumed. Excluded from the regression because of their high standardized residuals were four 'older' grains and one other (37.1) that lies significantly to the right hand side of the line indicative of radiogenic Pb loss after crystallization.

Subtraction of common Pb moves the plotted points towards Concordia along the locus shown in Fig. 12a. The amounts of common Pb as evaluated by using either the measured ^{204}Pb or measured ^{208}Pb determine their individual displacements along the locus, so that the radiogenic $^{207}\text{Pb}/^{206}\text{Pb}$ and the $^{238}\text{U}/^{206}\text{Pb}$ can be estimated without the assumption of age-concordance. Figure 12b shows the purely radiogenic data for the Kaplonosy zircons. The points straddle the Concordia curve without bias and fall on it within experimental error. The range in $^{238}\text{U}/^{206}\text{Pb}$ remains wider than expected for a single age, but their collective radiogenic $^{207}\text{Pb}/^{206}\text{Pb}$ does not suggest any significant differences between analyses. The mean value for the latter is equivalent to an age of 541 ± 17 Ma, although in view of the $^{206}\text{Pb}/^{238}\text{U}$ ages, this 'age' should be regarded as an imprecise estimate of a mixed age population rather than of a single event. The failure of the radiogenic $^{207}\text{Pb}/^{206}\text{Pb}$ data to distinguish mixed ages is due to the insufficient precision of the ion probe $^{207}\text{Pb}/^{206}\text{Pb}$ measurements for these low-Pb zircons.

Sample PL-92-16, Książ Wielki IG-1 drill-core

The zircons from this sample include six grains much older than the rest that show nearly concordant U–Pb ages in the range *c.* 750 Ma to *c.* 2775 Ma. Although there are no morphological signs of mechanical abrasion, the old ages label these grains conclusively as detrital in origin. Their existence in the sample warns that other detrital grains could be present, including some that might be only slightly older than volcanically-derived zircons in the tuff.

The cumulative probability plot (Fig. 13) shows that the dispersion and curvature of the $^{206}\text{Pb}/^{238}\text{U}$ data-set for 92-16 are as pronounced as those for 92-77, and the presence of two or more zircon ages may again be inferred. In this case, prior screening is warranted before applying the mixture-modelling program. One grain, #15, is distinctive in having a much higher and variable U content than the others (up to 1150 ppm). In addition, it is unique in showing open-system behaviour on the Th/U isochron diagram (not illustrated) and it contributes three of the four youngest $^{206}\text{Pb}/^{238}\text{U}$ ages. The remaining young age is one of two analyses of grain 22. These observations indicate that at least three of the four youngest ages in the distribution are due to post-crystallization Pb loss, not to the presence of an undisturbed

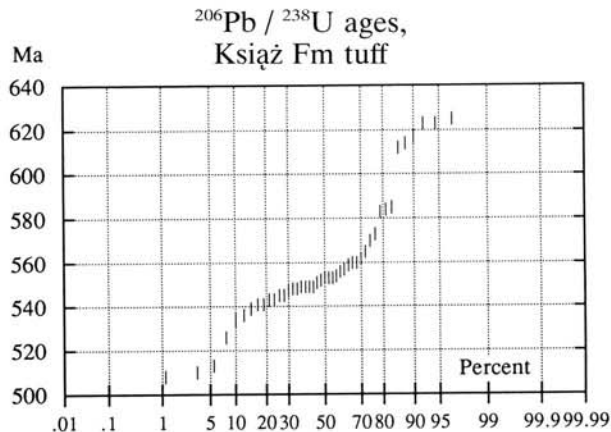


Fig. 13. Probability plot for 46 $^{206}\text{Pb}/^{238}\text{U}$ analyses of zircons from the Książ Wielki IG-1 borehole tuff, sample 92-16. Ages for several much older detrital zircons are not shown. The curvature of the plot reflects the mixing of a main 549 Ma age component with two older components.

young zircon age-group, and consequently they have been excluded from the data-set.

If two age-components are assumed within the 41 analyses, the mixture-modelling program rapidly converges, for all random starting points, on 550 ± 1.4 Ma for the major component (78% abundance) and 604 ± 3 Ma for the remainder. If three components are assumed, the age of the major component (72%) becomes 549 ± 1.6 Ma, and there would be two older components at 578 ± 4.5 Ma (13%) and 619 ± 4 Ma (15%). Regardless of the particular model, the principal zircon age component is close to 550 Ma. The deposition of the analysed tuff from the Książ Wielki Formation therefore occurred at or later than 550 Ma, dependent respectively on whether the dated zircons are cogenetic with the tuff magmatism or whether they are xenocrystic or detrital in origin. This result is not distinguishable from the Upper Vendian Kaplonosy sample.

Discussion

Zircon age interpretation

Our preferred age for the younger group of zircons in the Kaplonosy tuff is 551 Ma corresponding to the use of 'ordinary' common Pb in calculating their radiogenic Pb contents. The alternative result of 547 Ma based upon an anomalous initial Pb should be viewed as a younger limit. Of more concern is the question of how exactly do the zircons relate to the tuff in which they occur. Intermediate to felsic volcanic rocks such as those from the Nida Trough south of the Holy Cross Mountains are usually saturated with respect to zircon precipitation, whereas basaltic volcanics will be undersaturated. Zircons can certainly crystallize from chemically fractionated parts of thick basaltic flows where zircon saturation is achieved, but basalts are also known to contain zircon xenocrysts that are relict from the assimilation of older crustal rocks. Although being within a rapidly cooled lapilli-tuff rather than part of a massive flow, the volcanic sample studied here from the Kaplonosy Borehole has a felsic composition (Table 1). Nevertheless, it seems possible that many of the zircons found in it would

have formed prior to eruption. They could be older xenocrysts and/or precipitates from differentiated basaltic liquids in the magma chamber.

The zircon age interpretation must also account for the fact that some single grains have shown a wide range in $^{206}\text{Pb}/^{238}\text{U}$ ages (Fig. 10b). We therefore propose that many of the zircons are indeed older than the tuff but that they lost variable fractions of their radiogenic Pb by thermal diffusion during metamorphism at 551 Ma. Such thermal diffusion could accompany the incorporation of the zircons into basaltic magma chambers at that time, during assimilation of older felsic wall-rock. The low-abundance 635 Ma group could be a spurious 'group' produced by variable loss of Pb at 551 Ma from still-older primary ages. The sizeable 588 Ma group might represent the original age of earlier zircon crystallization, but none of the discontinuities in crystal form due to overgrowths or recrystallization to be expected for such a history have been detected.

Vendian time-scale

The age of 551 ± 4 Ma (95% limits) from tuff at the top of the Sławatycze Formation either determines the numerical age of the Vendian succession underlying the Sabellidites–Vendotaenia Zone within the Polish part of the East European Platform, or it sets a firm older limit for that succession if all the zircons in the tuff are detrital. As required, the age is older than recent results for zircon from early Cambrian volcanic rocks (Compston *et al.* 1992; Bowring *et al.* 1993).

Bowring *et al.* (1993) proposed that the Cambrian Period began at 543.9 Ma according to the age of pumice clasts within a breccia interpreted as phreatomagmatic from the Kessyusa Formation in northeast Siberia. If this interpretation is correct, the 551 Ma Vendian age allows a time-interval of 7 ± 4 Ma (95% limits) for the further c. 113 m of clastic sedimentation seen in the Kaplonosy core preceding the Cambrian/Precambrian boundary beds (Moczyłowska 1991), that are correlative of the global stratotype section. More conservatively, that time-interval becomes 17 ± 4 Ma (95% limits) relative to strata in the Kharaulakh Mountains (northeastern Siberia), dated as equal to or younger than 534.6 ± 0.5 Ma, that were originally believed to represent the lowermost Tommotian (Bowring *et al.* 1993), but are now known to underlie beds of the Middle Tommotian *D. regularis* Zone (Vidal *et al.* 1995a). The time-interval is also 21 ± 6 Ma (95% limits) relative to the revised Meishucun Bed 5 (upper Baiyanshao Member, corresponding to the *Anabarites–Protohertzina–Arthrochites* Assemblage) age of 530 ± 5 Ma (95% limits, Sambridge & Compston 1994).

It is important to remember that the *Anabarites–Protohertzina* Assemblage has a considerable stratigraphic range, as inferred from records in South Australia and Yukon (Quian Yi & Bengtson 1989). The Bowring *et al.* (1993) proposal of 543.9 Ma for the base of the Cambrian was inferred from stratigraphic relationships within the Kessyusa Formation to overlying rocks with fossils characterizing the *Anabarites trisulcatus* Zone. In this context, *A. trisulcatus* is not diagnostic for the base of the Cambrian (Quian Yi & Bengtson 1989). Furthermore, *A. trisulcatus* is referred either to the early Cambrian Manykaian Stage (Missarzewsky 1989) or to the Vendian Yudomian 'Stage' (Khomontovsky, *in* Sokolov & Fedonkin

1990). Bowring *et al.* (1993) indicated that chemostratigraphic data argued for placing the dated portion of the Kessyusa Formation in the earliest Cambrian (Manykaian Stage). However, the recently established co-occurrence of *A. trisulcatus* with the late Vendian shelly fossil *Cloudina* in Siberia (A. Zhuravlev, pers. comm. *in* Vidal *et al.* in press) opens an alternative interpretation in which the early part of the Manykaian Stage could be time equivalent with late Vendian *Cloudina*-bearing strata. It may then appear that the dated Kessyusa Formation is not proven to represent the base of the Cambrian System. By comparison with occurrences elsewhere, other fossil taxa within the Kessyusa Formation, such as *Sabellidites cambriensis* and the ichnofossil *Phycodes* sp., are *per se* not age-diagnostic either.

The zircons from the felsic tuff in the Książ Wielki IG-1 borehole are plausibly interpreted as dating the felsic magmatism in this area, rather than having a detrital or xenocrystic origin. Consequently, the felsic magmatism cannot be early Cambrian on current numerical control for the base of the Cambrian, and the lithostratigraphic correlation that was used previously to assign a Cambrian age for the Książ Wielki tuff cannot be supported.

Rates of biotic speciation in late Vendian times

Rates of speciation during late Vendian times can only be inferred against the background of few dated levels of Vendian and immediately succeeding Lower Cambrian strata. Recent biostratigraphy indicates that the bulk of the speciation-turnover represented by the radiation of faunas of early Cambrian small shelly fossils and trilobites falls within the lower and middle parts of the Tommotian and corresponds to two acritarch and trilobite biozones (Vidal *et al.* 1995), perhaps spanning no longer than 2–4 Ma each. As suggested by recent estimates, metazoan speciation levels during late Vendian and early Tommotian times were lower than during later portions of Cambrian time (Sepkoski 1992). Although the numerical time-frame (and indirectly the time-resolution) on which the original estimates were made is not substantial, the increasing number of reliable U–Pb ages allow the length of time involved in some critical intervals of the fossil record to be inferred. However, relative to the present date of tuffs within the Sławatycze Formation, most available dates for the Lower Cambrian apply to relatively 'young' portions of that Period, a condition that for the moment adds substantial difficulties to any attempt at comparing rates of speciation in the Vendian.

The timing of Neoproterozoic rifting in western Baltica

Neoproterozoic rock successions in Western Baltica are a record of deposition in which pre-Varangerian episodic emplacement of dolerite swarms ($870\text{--}1020$ Ma; 665 ± 10 Ma) and post-Varangerian basaltic volcanism in the late Vendian or Ediacaran at c. 551 Ma seem related to episodes of rifting and paroxysmal stages of faulting followed by erosion and basin infilling (Kumpulainen & Nystuen 1985; Vidal & Moczyłowska in press). Biostratigraphic correlation of successions in the western Baltoscandian basins and the Volhyn Aulacogen in the East European Platform support the idea that initial rifting, formation of embryonic fault basins within the core of Baltica, and their infilling might be regional isochronous events restricted to late

Riphean times (c. 800 to 750 Ma; Kumpulainen & Nystuen 1985).

Basaltic volcanic rocks occupy c. 140 000 km² (Juskowiakowa 1971) of the subsurface of the western margin of the East European Platform (Poland, Belarus, Russia, The Ukraine and Moldova). The U–Pb age of 551 Ma found for the dated tuff in the upper Sławatycze Formation suggests that this volcanic event is connected with the final rifting of the margin of Baltica prior to stabilization at the onset of the early Cambrian transgressions (Vidal & Moczyłowska in press). Organic matter preserved in late Vendian–early Cambrian rocks has recorded this as a thermal alteration event (Moczyłowska 1988) consistent with higher rates of heat flow associated with the opening of the Volhynian Aulacogen (considered as an aborted rift; Guterch 1977; Moczyłowska 1988). The 551 Ma age of the Sławatycze tuff sets an upper age constraint to the final rifting of Baltica (Vidal & Moczyłowska in press), relating to the opening of the Iapetus Ocean at the margin of the Ukrainian Shield, which is consistent with recent palaeogeographic reconstructions (Torsvik *et al.* 1992) assuming that the Lublin and Podolian slopes of the Ukrainian Shield were oriented towards Laurentia before rotation of Baltica which commenced in Vendian times.

On lithological grounds, the Sławatycze Formation has been correlated with the volcanogenic Volhyn Group in the Ukraine (Juskowiakowa 1971). Indirectly this sets constraints on the age of a succession overlying the latter, the Redkino 'horizon' (Sokolov & Fedonkin 1990), that yielded an Ediacaran-type fauna preceding the latest Vendian–Cambrian successions in Poland and the Ukraine (Vidal & Moczyłowska in press). The U–Pb age of the Sławatycze Formation is younger than K–Ar ages for glauconite from the Redkino 'horizon', as compiled by Sokolov & Fedonkin (1990), which yield ages in the range of 584–568 Ma (tuffites of the Kairovo subseries), 591–580 Ma (globular phyllosilicates of the Redkino Formation) and 575–546 (globular phyllosilicates of the Yaryshevo and Nagoryanka formations) in Podolia. Unless these datings represent the ages of detrital minerals, the present results would suggest an older age for the underlying Volhyn Group, generally regarded as ranging between 590 and 625 Ma (Sokolov & Fedonkin 1990).

The palaeogeographic position of the Holy Cross Mountains (Fig. 1) during Cambrian times in respect to Baltica is uncertain. The area was probably detached from Baltica along the Teisseyre–Tornquist Lineament. Despite evident faunal similarity with Baltoscandia during Cambrian times (Bergström 1984; Dadlez 1983), long-distance displacements prior to docking in late Palaeozoic times are inferred (Brochwicz *et al.* 1981, 1986; Pożaryski *et al.* 1982; Lewandowski 1993). The Cambrian succession in the Holy Cross Mountains consists of turbidites and has an estimated thickness of 2.0–2.5 km, as compared to 700–800 m of shallow shelf deposits in the East European Platform. Neoproterozoic deposits are not as yet documented in the Holy Cross Mountains, but their occurrence to the south in the Nida Trough (Książ Wielki Formation) was formerly suggested (Kowalczewski 1979; Jurkiewicz 1980; Kowalski 1983) and is supported by the present results. The late Vendian ages obtained for the Kaplonosy and Książ Wielki tuffs imply the possible proximity of the Nida Trough (and by default the southern Holy Cross Mountains) to Baltica in late Neoproterozoic and Cambrian times.

The southern Baltoscandian marginal areas and the East European Platform were viewed alternatively as part of a large single basin (of which the Holy Cross Mountains sequence is an integral part; Dadlez 1983; Bergström 1984), or as being on either sides of the Teisseyre–Tornquist Lineament. The latter view suggests that the southern Holy Cross Mountains basin was brought into its present vicinity to passive marginal basins of southern Baltoscandia and the East European Platform through extensive strike-slip displacement, mainly along the Teisseyre–Tornquist Lineament and the Hamburg–Kraków Lineament (Brochwicz *et al.* 1981, 1986; Pożaryski *et al.* 1982; Lewandowski 1993). These models propose strike-slip displacement along the Teisseyre–Tornquist Lineament, but disagree as for the sense of the displacement, which is proposed as either dextral (Lewandowski 1993) or sinistral (Brochwicz *et al.* 1981, 1986; Pożaryski *et al.* 1982). The Cambrian trilobite faunas of Baltoscandia, the East European Platform and the Holy Cross Mountains belong to the same fauna province and this feature alone could rule out the possibility of lateral displacement in the order of thousands of kilometers (Bergström 1984).

The age of 549 Ma for the tuff of the Książ Wielki Formation evidently indicates that deposition occurred at or later than 549 Ma (see above) and is here suggested to be of late Vendian age. In this context, the presence of substantially older zircon grains showing nearly concordant U–Pb ages in the range c. 750 Ma to c. 2775 Ma may be important. Conclusively labelled as detrital in origin, the late Archean age could suggest the incorporation in a Late Vendian volcanic event of Gondwana-derived zircons in an area where basement rocks are unknown.

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Non-biostratigraphical Methods of Dating and Correlation

Edited by R.E. Dunay (Mobil North Sea Ltd, UK) and E.A. Hailwood (Core Magnetics, UK)

Significant problems have arisen in the past with dating and correlating stratigraphic sequences impoverished in or barren of fossil remains, leaving major questions in stratigraphy for periods dominated by non-marine formations. These problems are exacerbated in dating and correlating barren sequences encountered in offshore exploration and appraisal drilling where the potential of alternative techniques becomes economically extremely important.

Brought together in this volume is a wide range of diverse techniques and disciplines, broadly grouped into mineralogical, chemical, isotopic, luminescence and cyclicity analyses, to explore their potential in solving difficulties in stratigraphy. The intention is also to introduce these techniques, already familiar to specialist researchers, to a wider audience of petroleum geologists who may find them useful in resolving their specific correlation problems.

The book will be of particular interest to hydrocarbon exploration and production geologists.

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