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Electron microprobe dating of monazite

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Abstract

Because monazite is extremely rich in U and Th, radiogenic Pb (*Pb) accumulates very quickly, and reaches, in about 100 Ma, a level where it is possible to analyse it with the electron probe. Assuming that common Pb is negligible, and that partial loss of Pb has not occurred, the simultaneous measurement of U, Th, and Pb allows to obtain a geologically meaningful age from a single electron probe analysis. Here we present the results of two years of systematical investigations aiming to define both the limits and potential of this method. A specific statistical method to deal with the large number of data which can be obtained on a single sample is described, and several guidelines, illustrated by examples, are suggested to optimize the method. Electron probe measurements carried out on samples of known age, from 200 Ma to 3.1 Ga, yield ages that always fall inside the confidence interval of the isotopically determined age, demonstrating that this method is reliable. The younger age limit is approximately 100 Ma, although it can be younger in some favourable cases. In old monazites, extremely high *Pb contents have been found (up to 5 wt%) indicating that monazite can tolerate high radiation doses without experiencing lead loss. The final precision on the age, for a 'normal' monazite, is ± 30–50 Ma, for a total counting time of 600 s. A complete dating procedure can be completed in less than 1 h. First results indicate that old ages can be preserved in monazite, either in small relict cores in crystals, or by the coexistence of several generations of monazites in a sample. This method has all the advantages of the electron probe: it is non-destructive, has an excellent spatial resolution (monazites as small as 5 μm can be dated), and because it is possible to work on normal polished thin-sections, the petrographical position of the dated crystal is known. This method offers a large number of geologists access to an in-situ dating technique at moderate cost.

1. Introduction

The application of micro-analytical techniques to geochronology gave birth to a new discipline in earth sciences that can be called micro-geochronology or micro-dating. Since the beginning (Froude et al., 1983), the success of this type of work has been enormous, because it provides information that may not be obtained by any other method. For the moment, most results published in the field are U–Pb zircon ages obtained using the SHRIMP ion microprobe (e.g., Williams, 1992), but efforts are in progress in numerous laboratories to use other ion probes and minerals (De Wolf et al., 1993; Harrison et al., 1994; Zhu et al., 1994a and 1994b; Weinbeck

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and Goswami, 1994; Compston and Matthai, 1994), or other techniques like laser ablation coupled with Ar/Ar (Scaillet et al., 1990), or ICP–MS (Fryer et al., 1992).

In itself, micro-analysis is not new to the earth sciences. Since the 60's the use of electron probe has become routine to geologists, because it is close to being the perfect micro-analyser: it is non-destructive (except for fragile materials like glasses), has a spatial resolution of 1–3 μm, is relatively easy to use, and yields results in a few minutes. Because the interactions between electrons and atoms are well understood, it yields precise and accurate analyses. Most of the time, it is used to analyse major elements in minerals or glasses, and more rarely trace elements down to few tens of ppm. Occasionally the electron probe has been used to obtain 'chemical ages ' on uraniferous and thoriferous minerals such as uraninite or thorite (Cameron-Shiman, 1978; Cuney et al., 1982). Examples of the application of this method are rare because such minerals are uncommon, difficult to identify in thin-section, and very easily reset by a thermal event. Recently, studies have been carried out successfully using monazite, which is a more common mineral (Suzuki and Adachi, 1991; Suzuki and Adachi, 1994; Suzuki et al., 1994; Montel et al., 1994; Foret et al., 1994). The first results demonstrate that this method is very promising and could offer, to a large number of geologists, access to an in-situ micro-dating technique.

Here we present the results of two years of measurements, carried out in our laboratory to survey the possibilities and limits of the method, and to establish some general rules for optimum use.

2. The method

2.1. Theoretical basis

The theoretical basis for the method is the same as for the so-called chemical aging of U–Th minerals used in the first attempts of geochronology. It consists of measuring Th–U–Pb concentrations in a crystal and of calculating the age (τ) by solving the equation:

$$\mathrm{Pb} = \frac{\mathrm{Th}}{232}\left[\exp(\lambda^{232}\tau) - 1\right]208 + \frac{\mathrm{U}}{238.04}0.9928 \times \left[\exp(\lambda^{238}\tau) - 1\right]206 + \frac{\mathrm{U}}{238.04}0.0072 \times \left[\exp(\lambda^{235}\tau) - 1\right]207 \quad (1)$$

where Pb, U, Th are in ppm, and λ^{232}, λ^{235}, λ^{238} are the radioactive decay constants of ^{232}Th, ^{235}U, and ^{238}U, respectively. Because monazite is rich in Th (commonly 3–15 wt%, sometime up to 25%) and U (a few hundreds of ppm up to 5%) radiogenic lead (*Pb) accumulates very quickly, and in less than 100 million years reaches a level where a precise measurement can be performed with an electron microprobe. The age calculated by this method has a geological meaning if: (1) non-radiogenic lead is negligible, and (2) no modification of the U/Th/Pb ratios has occurred except by radioactive decay. The validity of these hypotheses, in the case of monazite, was discussed by Montel et al. (1994), but we review the main arguments here. Numerous analyses of monazites for conventional U–Pb dating have been performed routinely in geochronological laboratories for 20 years. The thorium content is generally not determined (see however Wang et al., 1994), but uranium and lead analyses indicate that the non-radiogenic lead content is always very low, less than 1 ppm according to Parrish (1990). The second assumption is simply the closed-system hypothesis used by most geochronological methods, such as Rb–Sr dating of biotite, or K–Ar dating. In the case of monazite, it finds justification in the usually concordant behaviour of monazite in the ^{235}U/^{207}Pb vs ^{238}U/^{206}Pb diagram (Albarède et al., 1985; Parrish, 1990). This indicates that the U–Pb system in monazite is either totally reset or totally unaffected by any subsequent geological event. The behaviour of the Th–Pb system, the most important for chemical dating of monazite, is poorly known, despite the fact that the theoretical basis for its interpretation is established (Steiger and Wasserburg, 1966; Allègre, 1967). Recently, Barth et al. (1994a,b) demonstrated that for allanite it is more stable than the U–Pb system. Thus we think that partial loss of lead from monazite (discordant monazites), which would produce chemical ages without any significance, is un-

common, but possible. This effect should be seriously considered in polymetamorphic samples.

2.2. Sample preparation

Until now we have worked with two types of preparation. For the most part, we have used polished thin-sections prepared for conventional electron microprobe analyses. The main advantage is that we know the petrographical position of each dated crystal, which may be helpful in age interpretation. Another advantage is that rock thin-sections are common and may have been extensively studied from a petrographic point of view. The main disadvantage is that it is sometimes hard to locate crystals using microscope optics. Most of the time we use both optical and scanning-electron microscope (SEM) systems. Location of monazite in thin-sections is not easy under the optical microscope, because although all the high-relief accessory minerals are readily located, it is difficult to distinguish monazite from zircon or xenotime (Montel, 1993). From our experience, we suggest the following criteria: (1) a crystal with two pyramids is certainly a zircon, because monazite displays only one pyramid, if any; (2) a crystal with a thin cleavage (100) is certainly a monazite because cleavage in zircon (if any) is coarse, similar to that of pyroxenes; (3) when the extinction angle can be measured, it is very distinctive because it is 0 in zircon but 5–10° in monazite; (4) if a crystal displays a very obvious zoning in plane polarized light or under cross polars it is likely to be a zircon; (5) the refractive index of monazite ($n = 1.77–1.85$) is lower than that of zircon ($n = 1.92–2.01$); (6) most of the time, monazite forms rounded grains while zircon forms better-shaped prisms.

The most efficient way to identify monazite is to use a scanning-electron microscope (SEM). If this is equipped with an analytical energy dispersive system (EDS), identification is virtually instantaneous. Without EDS, monazite can be identified in backscattered electron images (BSE) because it is extremely bright (high average atomic number), even more so than zircon. Only thorite, uraninite or some other less common minerals are brighter than monazite in BSE. SEM work is not always necessary, but has proven to be very useful for revealing the internal structure of monazite (Parrish, 1990; Montel, 1992, 1993; Wark and Miller, 1993). It is also useful for finding crystals not seen by optical microscopy, in particular very small crystals, or those included in minerals with a similar refractive index like garnet. We also use mineral separates mounted in epoxy. The advantage here is that grain location is easy and so the complete age determination can be made in a very short time (see below). The mineral separate does not need to be pure because it is simple with the electron probe to identify monazite through the intensity of its Th signal. The main disadvantages are that the petrographical position of the dated crystal is unknown, and that during mineral separation, crystals smaller than 20 or 50 μm are discarded. Our experience is that in granites the average size of monazite is about 20 μm (Montel, 1993) and that crystals enclosed in other minerals can be even smaller.

2.3. Analytical procedure

All the analyses were carried out using the Camebax Micro electron microprobe at Clermont-Ferrand. The relatively ancient design of this machine severely constrains the operating conditions. For example, the maximum regulated probe current is about 100 nA, but this value changes each time the probe-forming apertures are replaced, varying from 90 to 145 nA. The accelerating voltage we use is 15 kV. Initially (Montel et al., 1994) we used 25 kV because it gave a slightly higher peak/background ratio. The use of 15 kV allows a better spatial resolution, 1.5 μm in diameter according to the equation of Castaing (1960), and significantly reduce the ZAF correction factors. We use three PET crystals to analyse simultaneously U, Th, and Pb. The X-ray lines are Mα for Th and Pb and Mβ for U. The interference of K Kα with U Mβ is thought do be negligible because monazite contains virtually no K, and the Th Mγ line can be avoided. For Pb Mα the closest lines are the two Th Mζ lines, and the second-order La Lα line, which can be avoided too. The most serious problem arises from the Y Lγ line which is superimposed upon the Pb Mα line. We determined the effect of this interference by measuring the intensity of Y Lγ on synthetic YPO_4. Extrapolation down to the Y content of monazite (< 2 wt%) shows that this

creates a maximum overestimate of the Pb content of about 30 ppm. Standards are ThO_2, UO_2, and a synthetic glass ($CaO–Al_2O–SiO_2$) with 6200 ppm of lead made in our laboratory. The counting time for standards is 50 s on peak and 100 s on background for ThO_2 and UO_2, and 300 s on peak and 600 s on background for Pb glass. The peak–background ratio is imposed by the machine. For dating, the counting time governs the precision on the U, Th and Pb contents and thus the final precision on the age. It varies from 30 to 300 s, depending on the nature of the sample. At the moment, we use 100 s, repeating the analyses when necessary to get a longer total counting time. Only U, Th and Pb are measured, and the ZAF matrix effect correction procedure is carried out using an average monazite composition for elements other than U, Th, and Pb. The ZAF factor is about 1.00 for Pb, and 1.21 for U and Th.

Errors and detection limits are calculated using the statistical approach of Ancey et al. (1978), which is based on the fact, demonstrated experimentally, that photon counting in X-ray analysis follows Poisson's law. For each measurement, the confidence intervals for Th, U, Pb are determined, the age is computed from Eq. (1), and the uncertainty on the age (95% confidence) is calculated by propagating the uncertainties on Th, Pb, and U into Eq. (1). So each measurement yields an age with a statistical confidence interval. Hereafter age results are given with this statistical error only, to allow unbiased inter-sample age discrimination. Two other systematic sources of error must be considered. One results from the fact that we use an average monazite composition in the ZAF correction procedure, instead of the true composition. Montel et al. (1994) estimated by a Monte-Carlo simulation procedure that this produces an additional uncertainty on the final age of 1% relative. The other source of error lies in the uncertainty on the composition of the Pb standard (1% relative uncertainty on the final age). The precision in electron probe dating of monazite is less than by isotopic dating. On a single measurement, it varies from ± 30 Ma for a Th- and U-rich monazite with 300 s counting time to ± 100 Ma for an average monazite with 30 s. As an additional check of the quality of the analyses, we perform, at the beginning of any session, the complete procedure on a well-dated monazite (Madagascar sample, see below), that we use as an 'age standard'.

2.4. Data presentation

Numerous ages can be obtained on a single thin-section, e.g. 20 to 70 in less than 2 h. A simple graphical display of a data set is by histograms, but in this particular case, because the error on each measurement is large, histograms would require large classes (of about 200 Ma), which would give a rather crude representation of the age population. Instead of representing each age measurement by a point (as in the histogram presentation) we prefer to display its bell-shaped probability curve, as defined by the age and standard deviation. A 'weighted histogram' representation is then given by the sum of all n bell-shaped curves (Hurford et al., 1984). This graphical representation may allow to visualize whether the population of age data is consistent with a single geological event, or unambiguously defines two successive events, or is too broadly dispersed, or shows a few outliers, etc. However, this does not spare a quantitative modelling.

2.5. Modelling

It is a classical problem in statistics to find the best estimate a and standard deviation σ_a of some quantity (here the age of a geological event) from a collection of n independent estimates T_i with standard deviations σ_{T_i} (here the microprobe ages of n supposedly synchronous domains in some monazite crystal or crystals). The least-squares modelling is easily extended (see Appendix A to cover situations where there are domains of two or more geological ages (a, b, ...) with no a priori knowledge of which domains relate to which geological event. Whatever the supposed number of age components, m, the model is valid only if the so-called MSWD (mean square of weighted deviates; McIntyre et al., 1966) is sufficiently close to 1. More precisely, we require that the accumulated probability function of the MSWD, $P_f(x)$ (Wendt and Carl, 1991), taken at $f = n - m$ (the number of degrees of freedom) and $x =$ MSWD (see Appendix A, lie within some prescribed limits. In practice we impose $0.05 < P_f <$

0.95, which amounts to $|4\text{lmswd} - 1| < 2\sqrt{2/f}$, for $f > 3$ (Wendt and Carl, 1991).

In order to assess the number of distinct age components resolvable by the data, we begin the least-squares modelling with $m = 1$ (only one age). If this simple model is acceptable, the procedure stops here, and we deduce the age $a \pm 2\sigma_a$. If it fails, we proceed with $m = 2$, and so forth.

2.6. Comparison with previous work

Basically the method presented here is the same as proposed by Suzuki and Adachi (1991), but with three main differences. *(1) Ages and errors are not calculated in the same way.* Suzuki and Adachi (1991) calculated ages using an isochron-like method, that enables them to estimate the common lead content in the monazite. However, this method implicitly assumes that all the monazites in a single sample contain the same amount of common Pb, which is very unlikely. Because the common lead they found is always low this method, although valid, is not really useful. We think that the procedure we used allows a more detailed statistical discussion and is more efficient, specially for poly-aged samples. *(2) We principally analyse thin-sections.* This is a great advantage, because we know the petrographical position of each individual crystal. Moreover, in thin-section we can analyse small crystals, and crystals in inclusions in other minerals, which are lost during

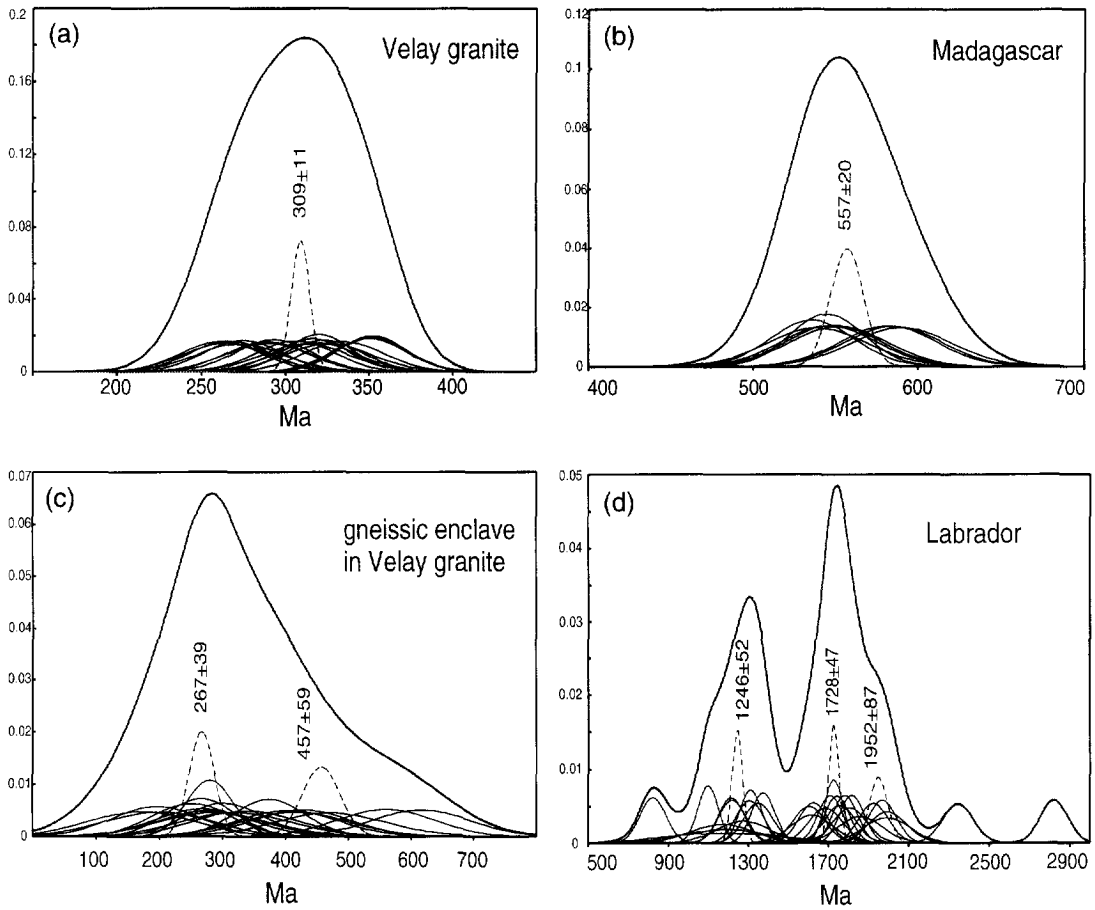

Fig. 1. Weighted-histogram representation of the data. Each small bell-shaped curve represents the probability density function for one measurement. The thick curve is the sum of all individual bell-shaped curves. The dashed curves represent the ages calculated by the statistical procedure.

Table 1
Analytical results for the Velay granite

N	Th	±	U	±	Pb	±	τ	±
1	50722	658	15959	376	1500	220	326	52
1	50830	660	16363	378	1234	219	265	51
1	41851	582	22649	420	1583	223	306	47
1	46695	624	21771	413	1705	221	323	46
1	61019	748	21060	409	2035	226	350	43
1	51891	669	19419	398	1375	222	267	47
1	118043	1238	7653	321	2258	225	352	40
1	48667	641	16918	380	1266	221	273	52
1	68438	811	10571	340	1437	217	312	52
1	79955	911	11061	344	1745	224	335	48
1	45733	616	23813	426	1746	224	316	44
1	39241	560	27670	451	2046	223	352	42
1	81612	926	11247	349	1455	225	275	46
1	62866	763	14336	364	1559	221	318	50
1	60226	741	16035	376	1319	221	262	48
1	45895	616	22708	419	1571	222	293	45
1	62831	763	15220	370	1454	221	289	48
1	48913	643	29388	462	2070	227	320	39
1	39246	560	19759	401	1341	217	290	51

N is the number of 100 s measurements at each point. Th, U, Pb in ppm, τ is the age in Ma. Printed errors limit the 95% confidence interval.

the mineral-separation procedure. *(3) We analyse only U, Th, and Pb.* With the Camebax it is not possible to change accelerating voltage and counting time during the analysis. So to obtain complete analysis of a monazite we should multiply the analytical time by 6, and work always at 25 kV accelerating voltage, which are not the best conditions for U, Th, and Pb. The method we use is a compromise, and we are aware that this part of our work could be improved.

3. Examples

3.1. Simple cases

Simple cases are samples where a single population has been found. Examples of such simple cases are given in Fig. 1 and in Tables 1 and 2. The fist sample is a grain mount of monazites from the Velay cordierite granite, dated at 298 ± 8 Ma by Rb–Sr

Table 2
Analytical results for the monazite from SE Madagascar

N	Th	±	U	±	Pb	±	τ	±
1	194393	1010	4247	368	5117	375	545	45
1	112781	741	2141	289	3191	294	590	61
1	115236	747	2700	291	3054	292	546	58
1	136512	819	2939	294	3531	302	536	51
1	110248	731	2353	288	2866	288	539	61
1	120645	766	2387	291	3373	297	582	58
1	116149	751	2459	290	3280	293	585	59
1	114779	746	2477	288	3060	293	552	59
1	116417	752	2719	290	3102	289	549	57

N is the number of 100 s measurements at each point. Th, U, Pb in ppm, τ is the age in Ma. Printed errors limit the 95% confidence interval.

whole rock isochron (Caen-Vachette et al., 1982). The monazites are very heterogeneous, containing 4.5 to 20% Th, 0.3 to 2.3% U and 800 to 2800 ppm Pb (Table 1). The crystal rims are richer in U and poorer in Th than the cores. The age population is homogeneous and gives 309 ± 11 Ma (MSWD = 1.52) for 19 × 100 s on 10 crystals (Fig. 1a). The second sample is a fragment of a massive monazite lens from the Manangoutry pass (SE Madagascar), dated at 545 ± 2 Ma (Paquette et al., 1994). The crystal is not perfectly homogeneous in composition (Table 2), but is homogeneous in age (Fig. 1b): 9 × 100 s gave 557 ± 20 Ma (MSWD = 0.46). We found that a unique population of ages is usual for granites, pegmatites, and in general, samples with simple geological histories.

3.2. A more complex case

An example of a sample displaying a bimodal age population is presented in Fig. 1c. The sample is a thin-section from a large gneissic enclave in the Velay granite. Large monazites and zircons are abundant in this sample, and 19 × 100 s measurements on 8 crystals were made (U = 0.23–0.90%; Th = 4–14%; Pb = 500–2300 ppm; Table 3). The bimodality of the age population can be suspected from the weighted-histogram representation (Fig. 1c), and is confirmed by the statistical calculations. The unique-age hypothesis ($\tau = 330 \pm 32$ Ma) is not statistically valid (MSWD = 2.32), although this age would be geologically acceptable. The program automatically separates the age populations into two groups, one with 12 measurements which gives 267 ± 39 Ma and the other with 7 measurements which gives 457 ± 59 Ma, with MSWD = 0.76. The late-Hercynian age is compatible with the Rb–Sr age of the Velay granite (see above). The older, Pan-African age, is similar to emplacement ages of orthogneisses in this area and could correspond to the age of a previous metamorphism (R'Khacham et al., 1990).

3.3. An even more complex case

One example of a complex population is presented in Fig. 1d. This sample is a grain mount of a mineral separate that has been dated by the conventional U–Pb method, as part of a detailed U–Pb geochronological study of a Proterozoic area in Labrador (Bertrand et al., 1993). The U, Th and Pb

Table 3
Analytical results for a gneissic enclave in the Velay granite

N	Th	±	U	±	Pb	±	τ	±
1	55767	931	7839	620	2063	503	560	153
1	61223	971	2306	572	1461	499	472	178
1	143094	1414	6779	614	2068	510	280	74
1	57703	945	4954	594	1337	496	403	162
1	50882	898	4412	590	581	478	199	172
1	52791	908	5136	599	1054	480	338	165
1	56816	939	5482	598	900	479	269	152
1	46105	864	5633	600	927	476	321	176
1	57275	944	7158	619	703	483	196	140
1	77963	1078	4632	596	1047	498	251	127
1	93007	1166	3910	591	1260	492	266	111
1	59222	957	3252	579	876	478	280	163
1	57804	951	2674	577	514	483	172	169
1	92969	1163	4589	596	1809	496	374	112
1	54672	924	3190	576	836	480	286	176
1	80673	1093	4101	590	1276	491	303	125
1	55155	928	7838	621	2255	513	616	158
1	45704	859	9386	637	1466	498	426	158
1	56436	937	4042	591	1297	499	415	174

N is the number of 100 s measurements at each point. Th, U, Pb in ppm, τ is the age in Ma. Printed errors limit the 95% confidence interval.

contents are highly variable (0.1–1.1% U; 0.2–13% Th; Table 4). The age population consists of 30 measurements on 9 crystals, which vary from 2.8 Ga to 824 Ma. The presence of Archaean ages is in agreement with the upper concordia intercept obtained on zircons from the same sample (2995 ± 24 Ma, Bertrand et al., 1993) or other samples from the same area (2767 ± 4 Ma, Scott and Machado, 1995). The 824 Ma age is isolated and will not be discussed further, although it could have some significance. For the rest of the population (26 ages) the statistical processing yields three populations: 1246 ± 52 Ma, $n = 10$; 1728 ± 40 Ma, $n = 11$; 1952 ± 87 Ma, $n = 5$; MSWD = 0.94. The intermediate age agrees with the conventional U–Pb monazite age (1739 ± 2 Ma; Bertrand et al., 1993). The 1952 ± 84 Ma age is probably related to the multiple events which occurred between 1.85 and 2.0 Ga in this area (Parrish, 1989, cited in Parrish, 1990; Bertrand et al., 1993; Scott and Machado, 1995). On the contrary, the 1246 ± 52 Ma age was not detected by the conventional U–Pb studies. From a regional point of view, this age is well-documented, and corresponds to the emplacement of the Elsonian igneous complexes at about 1.3 Ga (Ashwal et al., 1992). This example demonstrates that electron probe dating can give a detailed image of the geological history of a sample, although this image is less precise than conventional U–Pb dating, and more difficult to interpret. For example, the significance of the 2.4 Ga age is not clear. It can be the age of formation of this particular monazite (similar to the 2.56–2.66 Ga ages found by

Table 4
Analytical results for the sample from Labrador

N	Th	±	U	±	Pb	±	τ	±
1	92917	1180	1351	323	6055	542	1350	147
1	92179	1185	1184	499	5780	499	1308	139
1	82847	1125	1274	520	6487	520	1605	162
1	96117	1206	1295	494	5598	494	1216	130
1	94565	1195	1018	510	5479	510	1221	137
1	90963	1171	1916	555	8001	555	1759	156
2	25963	515	9198	561	4453	561	1619	215
2	24265	500	9970	364	4564	364	1627	148
2	19826	454	11285	383	5597	383	1926	149
2	38171	585	5047	362	4780	362	1808	167
1	126408	1354	4233	641	12005	641	1818	123
1	125351	1351	3349	623	10908	623	1711	123
1	119845	1321	3981	621	10879	621	1744	125
1	122049	1332	4521	631	11491	631	1784	123
2	51798	929	2813	510	5387	510	1859	223
1	51704	924	2648	527	5740	527	1992	234
1	84882	1130	9428	630	11082	630	1970	138
1	92024	1157	6956	676	13217	676	2347	152
2	53817	648	8115	484	11893	484	2823	138
2	95464	868	8240	441	10079	441	1728	93
1	77786	1101	1195	418	3057	418	824	130
2	75773	785	1296	333	4839	333	1313	111
2	76530	789	1240	316	4059	316	1101	103
2	73960	775	1716	352	5057	352	1375	117
1	82544	1122	1950	554	7010	554	1688	168
1	74580	1071	1784	561	7574	561	1995	191
1	5002	493	3722	433	970	433	1178	576
3	6095	308	4031	265	1079	265	1171	315
2	1909	341	4538	324	966	324	1189	427
3	3339	287	5841	271	3339	271	1275	267

N is the number of 30 s measurements at each point. Th, U, Pb in ppm, τ is the age in Ma. Printed errors limit the 95% confidence interval.

Scott and Machado, 1995) or an apparent, meaningless age, resulting from partial loss of lead in a 3 Ga monazite at 1.3, 1.7, or 1.9 Ga. Such complex, but information-rich, age populations are not rare in polymetamorphic samples (Suzuki and Adachi, 1991, 1994; Montel et al., 1994; Foret et al., 1994).

4. Working at the limits of the method

4.1. Young ages

The youngest age that can be measured is directly related to the detection limit for lead, which, with our apparatus, is about 200 ppm. When the measured lead content is lower than the detection limit, we can only say that the crystal is younger than a certain value (age detection limit), which depends on its Th and U content. In some exceptional cases, for very radioactive monazites (> 20% Th) it can be as low as 30 Ma, but most of the time, the limit is in the range 50–100 Ma. An example of this is given by the monazites from the Manaslu pluton for which we can only say that they are younger than 60 Ma (Montel et al., 1994). Although accurate, this result is not precise enough to be useful (Deniel et al., 1987). The youngest age we obtained is from the Jiouzou granite, Gungi province, China (Charoy et al., 1990). The sample is a polished thin-section, which contains numerous but small monazites (20–50 μm) that are moderately radioactive (5–8% Th, 0.13–0.4% U, Table 5). The Pb content is variable, but always less than 1000 ppm. 28 measurements were carried out on 15 crystals. Only 14 of them yield a lead content above the detection limit, for a final age of 213 ± 23 Ma, in agreement with the

Table 5
Analytical results for the Jiouzou granite

N	Th	±	U	±	Pb	±	τ	±
6	82202	494	1681	136	859	174	219	46
2	57846	696	2145	236	< 550		< 191	
2	61541	714	1582	233	603	293	202	102
2	70644	756	1327	233	601	299	180	93
2	76079	783	1935	236	807	297	219	84
2	71807	763	1970	233	< 549		< 161	
2	51675	660	4731	251	1015	298	338	182
2	52087	665	4642	248	< 543		< 183	
2	67971	746	2079	239	< 545		< 165	
2	51793	668	2201	238	< 548		< 210	
2	22825	491	3922	242	< 539		< 344	
3	29923	441	2443	193	< 443		< 265	
2	55817	687	1858	234	770	292	278	111
1	60158	984	1958	336	< 772		< 262	
2	68206	742	1915	230	< 534		< 162	
3	20442	387	2641	189	< 431		< 337	
2	70395	755	2422	234	839	301	240	90
2	75562	779	1847	232	705	295	193	84
2	62057	714	1135	227	616	295	209	104
2	61227	709	2113	233	559	288	183	98
2	70710	758	1504	226	676	284	199	87
2	68080	746	2322	233	538	289	159	88
3	50431	540	4642	199	682	235	232	84
4	57990	501	1896	163	642	207	224	75
3	55960	563	2145	190	< 442		< 158	
2	68620	747	2399	234	< 545		< 160	
2	74086	772	1783	230	< 549		< 157	
3	50529	541	2101	189	< 440		< 173	

N is the number of 30 s measurements at each point. Th, U, Pb in ppm, τ is the age in Ma. Printed errors limit the 95% confidence interval.

conventional U–Pb age of 230 Ma given by Chinese authors for the emplacement of the granite. The rest of the population is younger than about 160 Ma, suggesting that an event has reset part of the crystals since that time.

4.2. Old ages

From a theoretical point of view, there is no upper limit for this method. The only limitations may arise from two features: (1) the absence of monazite in old rocks, and (2) possible restriction to the ability of monazite to retain *Pb at very high concentrations. For the moment, the oldest sample we have studied is the 3.0 Ga Sinceni pluton (Trumbull, 1993) in Zwaziland. Monazites in this granite are moderately radioactive (0.04–0.45%, 2–14% Th), very old, and contain 0.2 to 2% lead (Montel et al., 1994). This is not the maximum Pb content of a monazite: in a 2.5 Ga sample, a crystal containing 11.5% Th and 9.6% U, has a Pb content of 5.6%. Actually, lead contents above 1% are very common in samples older than 1.5 Ga, but even such old monazites are most of the time optically clear, displaying no evidence of metamictization. On the contrary, the surrounding minerals are totally altered for more than 20 μm around the monazite, except if it is quartz or garnet. As the formation of 1 atom of lead requires the emission of 6, 7, or 8 α particles, it can be calculated that a 50 μm diameter monazite with 2% *Pb suffered 10^{15} α emissions. The fact that very old monazite crystals are presently clear and non-metamict suggests that this mineral can easily restore the crystal bonds broken by α particles. Thus, we think that the only upper age limit of the method is the absence of monazite in very old rocks, since peraluminous granites and metapelites, which are the rock types which contain monazite in abundance, are uncommon in old terranes.

4.3. Small crystals

The electron microprobe has an excellent spatial resolution, not only because the analysed volume is very small (less than 2 μm in diameter) but also because it is possible to localize very precisely the analysed area, either by normal optical view or by electronic imaging. Monazites in high-grade metamorphic rocks are usually rather big (100 μm), but monazites in granites are smaller, in the range 20–50

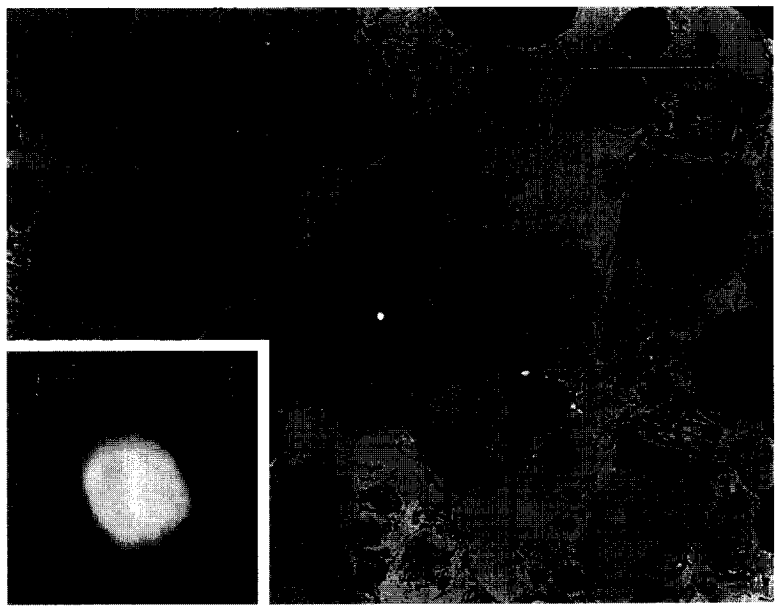

Fig. 2. Backscattered electron (BSE) image of an arkosic enclave in the Khartala volcano (Comoro Islands), showing bubbles (black), quartz and feldspars (medium grey), matrix (light grey), and the dated monazite, which is the bright spot in the quartz in the centre of the image. The scale bar in the enlarged portion is 10 μm.

μm. With the electron microprobe it is possible to make several measurements on a 20-μm crystal. The smallest monazites are 5–10 μm in diameter, and occur usually as inclusions in other minerals. An example of the use of the unsurpassed spatial resolution of the electron probe is detailed below. The sample is a thin-section from the Khartala Volcano, Comoro Islands. The thin-section is of a 10 × 15 mm arkosic enclave which contains several zircons and one 5-μm-diameter monazite (Fig. 2), included in a quartz. One measurement with 300 s counting time yielded Th = 93915 ± 801, U = 4188 ± 327, and Pb = 4815 ± 326 ppm, for a final age of 931 ± 52 Ma. Research is currently in progress to find a domain of similar age in the continental areas that surround the Comoro Islands, in order to identify the geographic origin of the arkose that the basaltic magma enclosed. At the present time, no technique other than electron probe could have dated this crystal.

4.4. Zoned crystals

On several occasions, we have found single crystals displaying two ages (Montel et al., 1994). Despite the primitive characteristics of the mapping and profiling ability of our relatively old Camebax Micro, we made U, Th, and Pb profiles across a monazite studied previously (Montel et al., 1994), known to display two ages on the same crystal. The sample is a thin-section from the southern granulite terrane of Madagascar. The U and Th contents vary from 0.03 to 0.6% and from 2.5 to 4.9%, respectively. In most of the crystal, the Pb content is in the range 600–1200 ppm, but in the centre lies a small area with Pb above 3000 ppm. Ages calculated at each point produced an age profile with a 1.5–2.0-Ga central domain surrounded by a larger 300–550-Ma domain (Fig. 3), in agreement with the previous measurements (Montel et al., 1994). The profile shown on Fig. 3 points out two interesting features. First, the spatial scale of age heterogeneities in a monazite can be as low as 10 μm. This means than any micro-dating technique that cannot analyse areas smaller than 10 μm may miss small crystal domains with different ages, and may yield intermediate ages by mixing informations from two or more domains of different ages. Secondly, the transition zone between the two domains is very small, only a few microns wide. This suggests that lead diffusion, in that particular case, has been very sluggish.

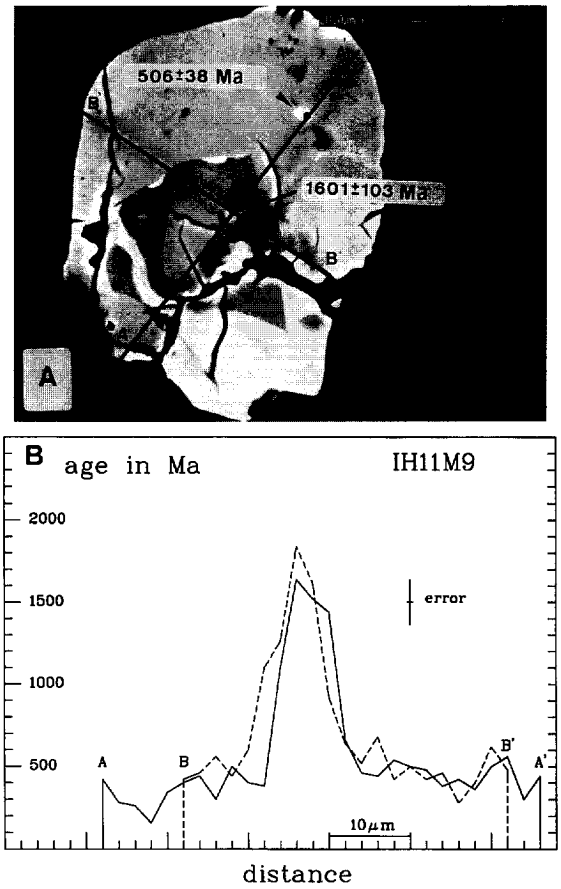

Fig. 3. (A) Backscattered electron (BSE) image of a monazite from the granulites of southern Madagascar. The two black lines represent profiles $A-A'$ and $B-B'$ shown below. In addition to the age profiles, two precise age determinations (300 s counting time), indicated by the arrows, were carried out. Variations of BSE intensity correspond to variations of average atomic number, mainly due to variations in Th content. (B) Age profiles along $A-A'$ and $B-B'$. Note the small size of the 1.6-Ga domain, and the sharpness of the transitions between the 1.6-Ga and 500-Ma domains.

5. Conclusions after two years of work

5.1. Reliability of the method

During two years we performed analyses on two types of samples: (1) those chosen to demarcate the

limits of the method, as discussed above; (2) samples of known age in order to definitively demonstrate its reliability. The comparison between the 'micro-chemical ages' and 'isotopic ages' obtained by other methods, is presented in Fig. 4. The agreement is excellent, always within the limits of analytical error. Frequently in polycyclic rocks we found multiple ages. In many cases, these ages correspond to previously recognized events in the same area (for example the micro-chemical age corresponds to the U–Pb zircon age, while another monazite population yields a younger age identical to the Rb/Sr biotite age on the same rock). The appearance of a previously unknown age was very rare. It can be an age older than any known age, which we consider to trace an old component, or an age younger than any other one, which we interpret as evidence for a younger event having reset some crystals. Finally, it can be an age intermediate between two well-documented ages, more difficult to interpret because it might be a meaningless age, resulting from lead diffusion (Suzuki et al., 1994).

5.2. Limits

The main limit of this method is the absence of monazite. Although relatively common, monazite is not as widely distributed as zircon or biotite which are used in other methods. Ca-rich rocks such as mafic and intermediate rocks of any type do not contain monazite, neither do metabasite or mafic greywackes (Cuney and Friedrich, 1987; Lee and Dodge, 1964). Low-grade metamorphic rocks also seem poor in monazite (Kingsbury et al., 1993). The lower limit to ages is at present about 100 Ma, but it is probably possible to lower this limit with another microprobe, in particular by using a higher probe current. Finally, this method is one order of magnitude less precise than isotopic methods.

5.3. Advantages

The advantages of this method were discussed above, but can be summarized as follows:

(1) It is accessible to many geologists because electron probes are widespread in earth sciences departments, and familiar to all petrologists.

(2) It is faster than conventional isotopic dating. When only dating is needed, work can be done either on mineral separates or on thin-sections. In the latter case the identification and dating of monazites can be done during the same session. Acquisition of 20×100 s measurements, with the complete statistical and age calculation procedure, is carried out in Clermont-Ferrand in less than 2 h. More detailed work is time-consuming, but even the most complex sample does not require more than 4 h of microprobe, 1 h of SEM, and 1 h of optical microscope work.

(3) It is non-destructive. This means that it is possible to re-analyse crystals when necessary, to

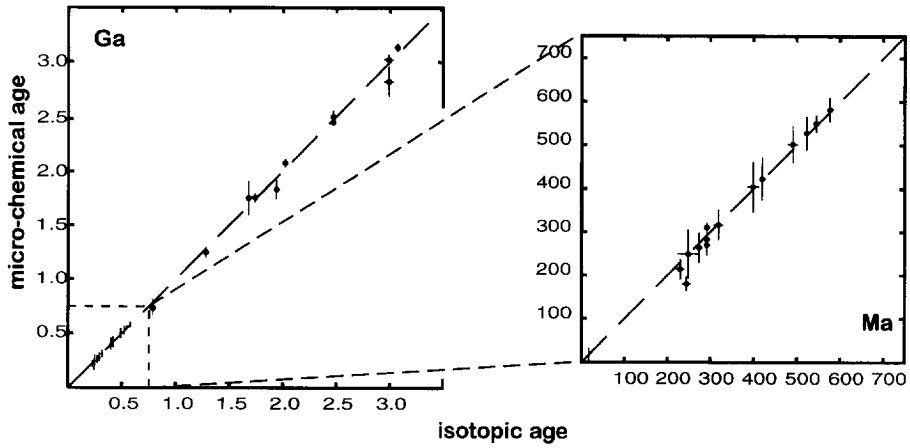

Fig. 4. Comparison between ages obtained with the electron probe ('micro-chemical age') and by conventional isotopic methods ('isotopic age').

analyse them for other elements, to do age mapping, or to analyse them by another method (an ion probe for example) after electron probe work.

(4) It is a truly in-situ dating technique that permits dating of crystals in thin-section knowing their exact petrographical position, even for very small crystals.

5.4. General guidelines

Field of application. The main fields of application of this method are:

(1) Exploration of a geochronologically unknown region, in order to define the most interesting targets that can be dated afterwards by conventional techniques.

(2) Work to solve a given problem, for instance to see rapidly if a granite is Pan-African or Archaean.

(3) Search of old terranes: because monazite has the ability to preserve old ages even through several geological events, it is possible to look systematically for old ages by investigating a large number of thin-sections.

(4) Dating of very small samples, like a granite pebble in a conglomerate, a small chip of dredged rock, a fragment of drill core, or an enclave in a basalt. A single grain as small as 5 μm can be dated.

Recommended procedure. For geochronological exploration, two methods can be used. In a polymetamorphic area it is possible to study in detail a small number of thin-sections, making numerous measurements (up to 70 can be done in a normal metapelite thin-section). The final age population will probably be complex, but the main geological events will emerge. The second method is to date a maximum of granites across the whole area. We found that, most of the time, the monazite age population in granites is unimodal, the analytical work easy and rapid, and the interpretation straightforward.

The quest of old terranes requires more detailed work. We recommend looking as systematically as possible for monazites included in garnet or in quartz, which seem to protect monazites efficiently (Foret et al., 1994; Zhu et al., 1994b; Montel et al., 1995).

A choice must also be made between dating a lot of points, with small counting times (hence with large errors), or making few but long and precise measurements. Numerous dates of low precision must be preferred for exploratory work. On the contrary, for precise dating, it is better to select the domains with the maximum Th- and U-contents, which will give the maximum precision.

5.5. The paradox of monazite concordant behaviour

There is an apparent contradiction between the first results obtained by in-situ dating of monazite (electron probe: Suzuki and Adachi, 1991, 1994; Montel et al., 1994; Foret et al., 1994; this work; ion probe: De Wolf et al., 1993; Zhu et al., 1994a, b; Harrison et al., 1994), which indicate that a single monazite crystal can be zoned in age, or that a single sample can display two age populations, and the usually concordant behaviour of monazite in the U–Pb system which suggests a simple geochronological behaviour. Several features may explain this discrepancy. First, the electron probe dating technique uses mainly the Th–Pb system, which produces 90% of *Pb. This system is, on the contrary, largely ignored in conventional dating based essentially on the U–Pb systems. Recently Barth et al. (1994a, b) demonstrated, in the case of allanite, that the Th–Pb system can be partially decoupled from the U–Pb systems. Second, during micro-dating procedures, old relicts are usually restricted to a few small domains. During a conventional isotopic study, the old signature is diluted, creating only a weak discordance, legitimately neglected. Finally, it must be recalled that with the electron microprobe, it is possible to analyse all crystals in a thin-section, including the smallest ones, and those included in other minerals, which are discarded during the mineral separation necessary for conventional analyses.

However, discordant monazites do exist (Gebauer et al., 1981; Black et al., 1984; Shärer et al., 1990; Parrish, 1990; Childe et al., 1993; Hawkins and Bowring, 1994) and we think that more detailed conventional work, considering in particular the Th–Pb system, will show that the geochronological behaviour of monazite is more complex than usually thought, as already demonstrated by Copeland et al. (1988) and Parrish (1990).

5.6. Future work

This method is in the early stages of its development. Because its field of application is large, and because it can easily be set up on any electron microprobe, we predict that it will become a routinely and widely used technique. It will make in-situ dating accessible to a large population of geologists, and numerous new results will be obtained, in particular for old cratons. The method can certainly be improved, in particular by other laboratories equipped with a more up-to-date machine. We think that it is possible to use probe currents as high as 300 nA, without damaging monazite, in order to get better precision and a lower detection limit. Progress can be made too by analysing elements other than U, Th, Pb, such as those with low atomic number (Si, Ca, P, Y), to obtain a more rigorous matrix effect correction. As modern microprobes have very elaborate mapping abilities, it is possible also to make age maps in crystals and this will give a lot of information on the behaviour of the U–Th–Pb system in monazite at the crystal scale (Suzuki et al., 1994). Finally, the best results will be obtained by combining the chemical data from the electron probe, and the isotopic data obtained with an ion probe.

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Appendix A. Least-squares modelling of multiple ages

A microprobe age T_i is obtained through an analytical procedure which introduces a random error $\delta_{0i} = T_i - t_{0i}$. At this stage the exact value t_{0i} is not implied to represent a true geological age, but just the translation into time units of an exact chemical quantity, the Pb–Th–U proportions. The error δ_{0i} is generated from a probability distribution $f_i(\delta)$, e.g. the Gaussian:

$$f_i(\delta) = \frac{1}{\sigma_{T_i}\sqrt{2\pi}} \exp\left[-\tfrac{1}{2}\left(\delta/\sigma_{T_i}\right)^2\right] \quad \text{(A-1)}$$

The most likely estimates of t_{0i} (t_i) are obtained by maximizing the joint probability distribution:

$$F(t_1, t_2, \ldots, t_n) = \prod_{i=1}^{n} f_i(T_i - t_i) \quad \text{(A-2)}$$

subject to the constraint that:

$$"t_1, t_2, \ldots, t_n \text{ verify the proposed model (exactly)}" \quad \text{(A-3)}$$

This formulation is just classical quantitative modelling, and would apply to any kind of data and any kind of theory. For Gaussian errors, maximizing Eq. A-2 is equivalent to minimizing:

$$S = \sum_{i=1}^{n} \left(\frac{T_i - t_i}{\sigma_{T_i}} \right)^2 \quad \text{(A-4)}$$

and the simplest theory that could apply to the microprobe ages is that all domains have evolved in closed system since some geological event of age a:

$$t_i - a = 0 \quad \forall i \in [1, n] \quad \text{(A-5)}$$

Minimizing Eq. A-4 subject to Eq. A-5 leads to the well-known weighted mean:

$$a = \frac{\sum_{i=1}^{n} \frac{T_i}{\sigma_{T_i}^2}}{\sum_{i=1}^{n} \frac{1}{\sigma_{T_i}^2}} \quad \text{(A-6a)}$$

$$\sigma_a = \frac{1}{\sqrt{\sum_{i=1}^{n} \frac{1}{\sigma_{T_i}^2}}} \quad \text{(A-6b)}$$

The next simplest theory is that some domains record a geological event of age a, and others (e.g., from inherited cores) another event of age b. Eq. A-5 should then be replaced by:

$$(t_i - a)(t_i - b) = 0 \quad \forall i \in [1, n] \quad \text{(A-7)}$$

and so forth for ($m > 2$) age components, if necessary.

A simple algorithm for minimizing Eq. A-4 subject to Eq. A-7 consists of partitioning the ordered set of T_i's into two consecutive subsets, then calculating (a, b) candidates as the weighted means of the two subsets, and storing the resultant S (Eq. A-4 with $t_i = a$ or b depending on i): the most likely partition is that with the smallest S, S_{min}. This algorithm is easily extended to $m > 2$ by considering the partitions into m consecutive subsets. The number of degrees of freedom, f, is equal to the number of constraints minus the number of free model parameters: $f = n - m$ (Eq. A-5, Eq. A-7 and so forth). Finally, a least-squares model is acceptable if S_{min} could have been generated from a χ^2-distribution with f degrees of freedom. One may carry out a classical χ^2-test, or resort to MSWD $= S_{min}/f$ (McIntyre et al., 1966; Wendt and Carl, 1991).

This modelling, which differs from previous approaches of the multi-component problem (e.g., Galbraith, 1988; Sambridge and Compston, 1994), is intended to be more orthodox. In particular, we do not consider that the m subsets result from a random sampling of m reservoirs with certain sampling probabilities, and we do not resort to a non-reversible likelihood function. However, we do not expect much quantitative difference between the various approaches; a detailed analysis will appear elsewhere.

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