DR2004094

## Analytical techniques

### SAMPLE PREPARATION AND LA- ICP -MS ANALYTICAL INSTRUMENTATION AND PROCEDURES

Samples WR-10 and TB-1 were crushed with a jaw crusher and pulverised with a disc mill. Zircons were separated at the Complutense University of Madrid by heavy fraction enrichment on a Wilfley table followed by density separation using di-iodomethane ($CH_2I_2$) and magnetic separation in a Frantz isodynamic separator. Zircons were selected from the least magnetic fraction and hand-picked in alcohol under a binocular microscope. Zircon grains representing all sizes (except those whose breadth was shorter than *c.* 20μm) and morphological types present in both samples were selected for LA-ICP-MS analyses. Grains were set in synthetic resin mounts, polished to approximately half their thickness and cleaned in a warm $HNO_3$ ultrasonic bath. Analytical instrumentation consisted of a UP213 frequency quintupled Nd:YAG based laser ablation system (NewWave Research, Fremont, USA) coupled to a (Thermo Elemental) PQ3, quadrupole based ICP-MS instrument with enhanced sensitivity (S-Option) interface. Instrument and operating parameters used for individual zircon analyses are as given in Jeffries et al. (2003).

Samples and standard were ablated in an air-tight sample chamber flushed with Helium for sample transport. The laser was focused on the sample surface and energy density was kept constant for each analysis. The samples were rastered along lines ca. 30 to 60 microns long (depending on zircon size), using a constant raster speed for each analysis. Nominal beam diameter was 30μm for zircon analyses of sample WR-10 but all zircons from sample TB-1 were analysed with a nominal beam diameter of 18μm owing to their small size.

Data were collected in discrete runs of 20 analyses, comprising 12 unknowns bracketed before and after by 4 analyses of the standard zircon 91500

(Wiedenbeck et al. 1995). During the analytical sessions of samples WR-10 and TB-1 the standard 91500 yielded a weighted mean (n=56) of 1062.3 ±1.9 Ma (MSWD =1.2) for the $^{206}Pb/^{238}U$ age (certified ID-TIMS $^{206}Pb/^{238}U$ age: 1062.4 ± 0.4 Ma) and a weighted mean of 1065.3 ± 2 Ma (MSWD = 0.6) for the $^{207}Pb/^{206}Pb$ age (certified ID-TIMS $^{207}Pb/^{206}Pb$ age: 1065.4 ± 0.3 Ma).

Data for sample zircons were collected for up to 150 s per analysis with a gas background taken during the initial ca. 60 s. For each analysis, time-resolved signals were obtained and then examined to ensure that stable flat signal intervals (free from inclusions, core-rim features, zones of high common Pb or evidence of fractionation) were used in the age calculations. Preliminary selection of background and analysis signal intensities and data calculation was performed using 'LAMTRACE', a macro based spreadsheet written by Simon Jackson, Macquarie University, Australia. Background and mass bias corrected signal intensities and counting statistics were calculated for each isotope. Concordia age calculations, and concordia and cumulative probability plots were performed using Isoplot/Ex rev. 2.49 (Ludwig, 2001). Data treatment, assignment of final ages and errors for individual analyses, estimation of common Pb and criteria for rejection of analyses are those detailed in Jeffries et al. (2003).

*Common Pb*. The ages reported in Table 1 are not common-lead corrected as $^{204}Pb$ measurements are rendered useless by the isobaric interference from Hg, a contaminant present in the argon supply gas. $^{204}Hg$ interferes on $^{204}Pb$ and the $^{202}Hg$ peak is too small to allow a reliable overlap correction of acceptable precision. The $^{208}Pb$ correction method (Compston et al. 1984, Ludwig 2001) cannot be applied reliably to all U-Pb analyses as the assumption of concordance between the radiogenic $^{206}Pb/^{238}U$ and $^{208}Pb/^{232}Th$ ratios is not always justified. This is particularly obvious in U-Pb dating of detrital zircon populations where only one analysis per grain is obtained and extrapolations on the behaviour of the U-Th-Pb system cannot be applied as for instance in the case of U-Th-Pb dating of cogenetic magmatic zircons.

Andersen (2002) has proposed a common lead correction method that neither uses $^{204}$Pb nor assumes concordance, but relies instead on the assumption of coherent behaviour of the U-Pb and Th-Pb systems during Pb loss. Application of this correction algorithm revealed significant common lead content (i.e. age correction outside analytical error) in 6 discordant analyses. These analyses have been rejected and are not reported in Table 1. In addition, examination of the time-integrated U-Pb, Th-Pb and Pb-Pb isotope ratio plots allows the identification of analyses (or parts of an analysis) that contain significant amounts of common lead (e.g. Fernández-Suárez et al. 2002; Jeffries et al., 2003) and consequently either the whole analysis is rejected or the corresponding segment of the signal containing common Pb is not used in the age calculation. Therefore, we are confident that the effect of common Pb in the ages reported in Table 1 is well below total analytical uncertainty.

To date, we have no knowledge of any ICP-MS system/analytical protocol that can measure 204Pb in zircon with enough precision to allow a reliable and accurate correction. It is accepted by most U-Pb geochronologists that most zircons contain very small amounts of common lead and that most of it resides in altered/metamict zones, inclusions, fractures, that ideally are avoided by laser ablation sampling. Therefore we argue that when faced with vanishingly small amounts of common lead, the lack of 204Pb correction is not more inaccurate than the nearly inevitable over-correction derived from inaccurately measured 204Pb. As we show in the Jeffries et al. (2003) paper, a careful examination of the time-integrated isotope ratio plots will allow the analyst to detect zones of unusual high common Pb whose corresponding signal intervals are never used for age calculation. Our experience shows that the approach of very stringent signal selection/rejection guarantees that the effect of small amounts of common Pb does not affect the accuracy of analyses beyond their reported uncertainty. This latter point has been confirmed in many instances by analyses of zircons previously dated by ID-TIMS.

We must also state that in our analyses, most derived ages are based on the more robust 206Pb/238U ratios as they are concordant. It is only in the oldest (pre-Mesoproterozoic) discordant zircons (Table 1) that we report 207Pb/206Pb ages, and even in these cases the analyses are less than 10% discordant. Moreover, these analyses are the less relevant to the main geological issue addressed in this article.

The ages labelled as "*reported age*" in Table 1 are calculated as follows: For concordant analyses (ages whose corresponding isotope ratios have a 2$\sigma$ error ellipse that, to a greater or lesser extent, overlaps the concordia curve) we report concordia ages and errors as defined by Ludwig (1998). For normally discordant analyses we report the $^{207}$Pb/$^{206}$Pb age and 2$\sigma$ error. In cases where the error on the $^{207}$Pb/$^{206}$Pb ratio is large owing to low $^{207}$Pb or very short collection times (very small zircons) the error reported corresponds to the upper intercept of a discordia forced through 0 Ma. For slightly reversely discordant zircons younger than ca 900 Ma and whose $^{207}$Pb/$^{206}$Pb ages have large errors owing to small amounts of $^{207}$Pb we use the more precise $^{206}$Pb/$^{238}$U age and corresponding 2$\sigma$ error.

Further comments on analytical procedures:

1) Standard and sample are not mounted together in the same block as this would be wasteful of a standard in short supply, but the sample block and standard block are mounted together in the ablation cell. In our experience and with our analytical set up and protocol (described in Jeffries et al. 2003) there is no fractionation effect in moving between blocks already within the ablation cell just as there is none in moving from one side of a block to another. Concerning standardisation, we have opted for run standard

bracketing having found that the mass bias drift on our quadrupole instrument is insignificant during the course of a run and so there is no gain in using sample standard bracketing.

2) The uncertainties reported represent 2 sigma internal errors. In provenance studies, where the final goal is not one age but the statistical distribution of age clusters, we feel that propagating all sources of uncertainty simply produces larger uncertainties that finally make many data technically concordant (giving a false sense of security) owing only to the large resulting errors. We feel that 2 sigma internal errors alone give a better understanding of concordance of the U-Pb system. Particularly in this type of study, where it is the presence/absence and relative proportion of given age groups that provides the scientific arguments, the error propagation approach has no bearing on the conclusions. Full error propagation should be done when aiming at dating crystallisation of plutonic rocks, eruption of volcanic rocks or metamorphic events.

3) Signal interval selection. Signal interval selection is based on a careful and stringent examination of isotope ratio plots versus time. In this fashion, phenomena such as zones of high common Pb, fractures, inclusions, U-Pb elemental fractionation, inherited radiogenic lead, inconsistent behaviour of the Th-Pb and U-Pb systems are easily detected. Therefore the signal selected is not arbitrary but always the only interval that is free of suspect features. The practise of automatically matching signal intervals in standard and unknown may be required in cases where the laboratory opts for stationary spot analyses and then this becomes a necessity to correct for elemental fractionation. In our case, using line rasters and the analytical parameters and instrument conditions reported in Jeffries et al. (2003), we do not have detectable U-Pb elemental fractionation and therefore there is no reason to use matching intervals in sample and standard. That would lead in many cases to selection of parts of analysis that have any of the above suspect features.

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**Table 1 (continued). LA-ICP-MS U-Pb results**

| *Sample* ***TB-1*** | | ***Isotopic ratios and 2 σ (%) errors*** | | | | | | ***Ages and 2 σ absolute errors (Ma)*** | | | | | | ***Reported Age*** | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | | | | | | | | | | [see text for details] | | |
| **Anal. #** | **i.s. [s]** | **$^{206}$Pb/$^{238}$U** | ±2σ | **$^{207}$Pb/$^{235}$U** | ±2σ | **$^{207}$Pb/$^{206}$Pb** | ±2σ | **$^{206}$Pb/$^{238}$U** | ±2σ | **$^{207}$Pb/$^{235}$U** | ±2σ | **$^{207}$Pb/$^{206}$Pb** | ±2σ | **Age (Ma)** | ±2σ | **disc %** |
| | | | | | | | | | | | | | | *Dep. Age: Early Devonian* | | |
| fe06a05 | 29 | 0.0621 | 1.06 | 0.4588 | 2.36 | 0.0536 | 1.64 | 388 | 4 | 383 | 8 | 352 | 36 | **388** | **4** | -10.2 |
| fe06a15 | 35 | 0.0632 | 1.72 | 0.4852 | 2.30 | 0.0557 | 2.04 | 395 | 7 | 402 | 8 | 438 | 46 | **397** | **6** | 9.8 |
| fe06a08 | 27 | 0.0710 | 2.38 | 0.5515 | 3.08 | 0.0563 | 3.58 | 442 | 10 | 446 | 11 | 462 | 80 | **444** | **8** | 4.3 |
| fe06a16 | 19 | 0.0845 | 2.70 | 0.6760 | 2.82 | 0.0580 | 3.94 | 523 | 14 | 524 | 12 | 528 | 86 | **524** | **10** | 0.9 |
| fe06c07 | 33 | 0.0857 | 2.40 | 0.7121 | 5.00 | 0.0603 | 5.26 | 530 | 12 | 546 | 21 | 612 | 114 | **533** | **11** | 13.4 |
| fe06a12 | 19 | 0.0877 | 2.16 | 0.7134 | 1.94 | 0.0590 | 1.64 | 542 | 11 | 547 | 8 | 564 | 36 | **547** | **8** | 3.9 |
| fe06c15 | 59 | 0.0981 | 1.28 | 0.8063 | 1.48 | 0.0596 | 1.64 | 603 | 7 | 600 | 7 | 588 | 34 | **602** | **6** | -2.6 |
| fe06c16 | 23 | 0.1005 | 3.24 | 0.8169 | 4.48 | 0.0590 | 3.06 | 617 | 19 | 606 | 20 | 564 | 68 | **613** | **18** | -9.4 |
| fe06b16 | 34 | 0.1004 | 2.12 | 0.8370 | 4.68 | 0.0604 | 4.04 | 617 | 12 | 617 | 22 | 618 | 88 | **617** | **12** | 0.2 |
| fe06b07 | 27 | 0.1014 | 1.36 | 0.8580 | 1.78 | 0.0613 | 1.90 | 623 | 8 | 629 | 8 | 650 | 40 | **626** | **7** | 4.2 |
| fe06c05 | 56 | 0.1032 | 1.68 | 0.8598 | 1.90 | 0.0604 | 0.94 | 633 | 10 | 630 | 9 | 618 | 22 | **630** | **9** | -2.4 |
| fe06b11 | 25 | 0.1037 | 1.16 | 0.8888 | 1.76 | 0.0621 | 2.32 | 636 | 7 | 646 | 8 | 678 | 50 | **678** | **50** | 6.2 |
| fe06c06 | 46 | 0.1003 | 1.84 | 0.8687 | 2.52 | 0.0628 | 2.24 | 616 | 11 | 635 | 12 | 700 | 48 | **700** | **48** | 12.0 |
| fe06a11 | 21 | 0.1176 | 2.18 | 1.0559 | 2.86 | 0.0651 | 1.38 | 717 | 15 | 732 | 15 | 776 | 30 | **776** | **30** | 7.6 |
| fe06b05 | 19 | 0.1371 | 1.54 | 1.2550 | 1.44 | 0.0664 | 1.56 | 828 | 12 | 826 | 8 | 818 | 32 | **826** | **8** | -1.2 |
| fe06a06 | 50 | 0.1723 | 1.62 | 1.7179 | 1.98 | 0.0723 | 1.70 | 1025 | 15 | 1015 | 13 | 994 | 34 | **1018** | **12** | -3.1 |
| fe06a10 | 19 | 0.1797 | 1.92 | 1.8825 | 2.04 | 0.0760 | 1.86 | 1065 | 19 | 1075 | 14 | 1094 | 38 | **1073** | **13** | 2.7 |
| fe06b14 | 29 | 0.1841 | 1.76 | 2.0090 | 1.78 | 0.0791 | 1.94 | 1089 | 18 | 1119 | 12 | 1174 | 38 | **1174** | **38** | 7.2 |
| fe06b13 | 29 | 0.2399 | 2.22 | 3.0010 | 2.46 | 0.0907 | 1.48 | 1386 | 28 | 1408 | 19 | 1440 | 30 | **1440** | **30** | 3.8 |
| fe06c09 | 33 | 0.2995 | 1.40 | 4.4522 | 1.18 | 0.1078 | 0.96 | 1689 | 21 | 1722 | 10 | 1762 | 18 | **1762** | **18** | 4.1 |
| fe06c11 | 23 | 0.2881 | 1.54 | 4.3410 | 2.04 | 0.1092 | 2.14 | 1632 | 22 | 1701 | 17 | 1786 | 40 | **1786** | **40** | 8.6 |
| fe06b10 | 59 | 0.3303 | 1.24 | 5.1952 | 1.66 | 0.1140 | 1.12 | 1840 | 20 | 1852 | 14 | 1864 | 20 | **1864** | **20** | 1.3 |
| fe06c14 | 27 | 0.3299 | 1.68 | 5.6023 | 2.46 | 0.1231 | 1.42 | 1838 | 27 | 1916 | 21 | 2002 | 26 | **2002** | **26** | 8.2 |
| fe06b06 | 21 | 0.3830 | 1.92 | 6.7650 | 2.86 | 0.1281 | 1.50 | 2090 | 34 | 2081 | 25 | 2070 | 26 | **2078** | **25** | -1.0 |
| fe06c08 | 50 | 0.3793 | 1.48 | 6.9458 | 1.70 | 0.1328 | 1.32 | 2073 | 26 | 2105 | 15 | 2134 | 24 | **2134** | **24** | 2.9 |
| fe06c12 | 31 | 0.3746 | 1.68 | 7.0240 | 1.54 | 0.1360 | 0.90 | 2051 | 29 | 2114 | 14 | 2176 | 16 | **2176** | **16** | 5.7 |
| fe06b09 | 35 | 0.5252 | 1.40 | 13.6647 | 1.62 | 0.1887 | 1.24 | 2721 | 31 | 2727 | 15 | 2730 | 20 | **2727** | **15** | 0.3 |

i.s.= signal interval integrated for isotope-ratio and age calculation (in seconds)
disc%= percent discordance calculated from $^{207}$Pb/$^{206}$Pb and $^{206}$Pb/$^{238}$U ages (negative values: reverse discordance)

**Table 1. LA-ICP-MS U-Pb results**

| *Sample* ***WR-10*** | | ***Isotopic ratios and 2 σ (%) errors*** | | | | | | ***Ages and 2 σ absolute errors (Ma)*** | | | | | | ***Reported Age*** [see text for details] | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| **Anal. #** | **i.s. [s]** | **$^{206}Pb/^{238}U$** | ±2σ | **$^{207}Pb/^{235}U$** | ±2σ | **$^{207}Pb/^{206}Pb$** | ±2σ | **$^{206}Pb/^{238}U$** | ±2σ | **$^{207}Pb/^{235}U$** | ±2σ | **$^{207}Pb/^{206}Pb$** | ±2σ | **Age (Ma)** | ±2σ | **disc %** |
| | | | | | | | | | | | | | | *Dep. Age: Early Silurian* | | |
| fe05a05 | 27 | 0.0880 | 2.48 | 0.7024 | 3.08 | 0.0578 | 2.48 | 544 | 13 | 540 | 13 | 522 | 54 | **542** | **12** | -4.2 |
| fe05d09 | 21 | 0.0877 | 1.40 | 0.7037 | 2.92 | 0.0582 | 3.04 | 542 | 7 | 541 | 12 | 536 | 66 | **542** | **7** | -1.1 |
| fe05c14 | 31 | 0.0885 | 1.38 | 0.7017 | 1.86 | 0.0575 | 2.40 | 547 | 7 | 540 | 8 | 508 | 54 | **547** | **7** | -7.7 |
| fe05c07 | 29 | 0.0881 | 1.84 | 0.7263 | 2.62 | 0.0598 | 2.62 | 544 | 10 | 554 | 11 | 594 | 56 | **548** | **9** | 8.4 |
| fe05b16 | 34 | 0.0893 | 2.02 | 0.7208 | 3.50 | 0.0585 | 3.00 | 551 | 11 | 551 | 15 | 548 | 64 | **551** | **10** | -0.5 |
| fe05d08 | 34 | 0.0811 | 1.80 | 0.6577 | 2.24 | 0.0588 | 1.28 | 503 | 9 | 513 | 9 | 560 | 28 | **560** | **28** | 10.2 |
| fe05b08 | 57 | 0.0913 | 2.50 | 0.7435 | 3.12 | 0.0591 | 1.56 | 563 | 13 | 564 | 13 | 568 | 34 | **564** | **13** | 0.9 |
| fe05c09 | 31 | 0.0905 | 2.94 | 0.7454 | 2.00 | 0.0597 | 2.38 | 559 | 16 | 566 | 9 | 592 | 52 | **566** | **9** | 5.6 |
| fe05d14 | 33 | 0.0925 | 2.04 | 0.7572 | 2.98 | 0.0594 | 2.82 | 570 | 11 | 572 | 13 | 580 | 60 | **571** | **10** | 1.7 |
| fe05a16 | 40 | 0.0925 | 1.52 | 0.7612 | 1.82 | 0.0597 | 1.40 | 570 | 8 | 575 | 8 | 592 | 30 | **573** | **7** | 3.7 |
| fe05c08 | 25 | 0.0856 | 2.40 | 0.7009 | 2.42 | 0.0594 | 2.48 | 529 | 12 | 539 | 10 | 580 | 54 | **580** | **54** | 8.8 |
| fe05a08 | 17 | 0.0844 | 2.34 | 0.6965 | 2.68 | 0.0598 | 2.94 | 523 | 12 | 537 | 11 | 596 | 62 | **596** | **62** | 12.2 |
| fe05b05 | 25 | 0.0983 | 2.44 | 0.8027 | 2.02 | 0.0592 | 1.36 | 604 | 14 | 598 | 9 | 574 | 28 | **596** | **9** | -5.2 |
| fe05a10 | 42 | 0.0972 | 1.48 | 0.8028 | 1.82 | 0.0599 | 1.58 | 598 | 8 | 598 | 8 | 598 | 34 | **598** | **7** | 0.0 |
| fe05b11 | 40 | 0.0991 | 1.68 | 0.8196 | 2.42 | 0.0600 | 2.28 | 609 | 10 | 608 | 11 | 600 | 50 | **609** | **9** | -1.5 |
| fe05b14 | 33 | 0.1009 | 1.84 | 0.8386 | 5.18 | 0.0603 | 5.82 | 620 | 11 | 618 | 24 | 612 | 126 | **620** | **11** | -1.3 |
| fe05c10 | 31 | 0.1023 | 1.72 | 0.8752 | 3.38 | 0.0621 | 3.02 | 628 | 10 | 638 | 16 | 676 | 66 | **629** | **10** | 7.1 |
| fe05b07 | 27 | 0.1027 | 2.02 | 0.8744 | 3.12 | 0.0617 | 2.52 | 630 | 12 | 638 | 15 | 664 | 54 | **632** | **12** | 5.1 |
| fe05a12 | 33 | 0.1037 | 1.94 | 0.8669 | 2.16 | 0.0606 | 1.50 | 636 | 12 | 634 | 10 | 624 | 32 | **634** | **10** | -1.9 |
| fe05b13 | 40 | 0.1048 | 1.70 | 0.8662 | 3.04 | 0.0599 | 2.96 | 643 | 10 | 633 | 14 | 600 | 66 | **640** | **10** | -7.2 |
| fe05d11 | 27 | 0.1107 | 2.06 | 0.9575 | 1.82 | 0.0627 | 1.84 | 677 | 13 | 682 | 9 | 698 | 40 | **681** | **9** | 3.0 |
| fe05b09 | 27 | 0.1367 | 1.72 | 1.2651 | 1.42 | 0.0671 | 2.56 | 826 | 13 | 830 | 8 | 840 | 54 | **830** | **8** | 1.7 |
| fe05d12 | 25 | 0.1247 | 1.68 | 1.1533 | 2.48 | 0.0671 | 2.66 | 758 | 12 | 779 | 13 | 838 | 56 | **838** | **29** | 9.5 |
| fe05c16 | 29 | 0.1596 | 1.64 | 1.5862 | 1.42 | 0.0721 | 0.96 | 955 | 15 | 965 | 9 | 986 | 18 | **986** | **18** | 3.1 |
| fe05a11 | 40 | 0.1752 | 1.72 | 1.8004 | 1.76 | 0.0745 | 0.90 | 1040 | 17 | 1046 | 12 | 1054 | 18 | **1047** | **11** | 1.3 |
| fe05a14 | 19 | 0.2032 | 1.78 | 2.2156 | 1.64 | 0.0791 | 1.48 | 1192 | 19 | 1186 | 11 | 1172 | 30 | **1186** | **12** | -1.7 |
| fe05a09 | 40 | 0.2045 | 2.28 | 2.2587 | 2.28 | 0.0801 | 1.56 | 1200 | 25 | 1199 | 16 | 1198 | 32 | **1200** | **16** | -0.2 |
| fe05a07 | 27 | 0.2049 | 1.50 | 2.3055 | 1.80 | 0.0816 | 1.54 | 1202 | 16 | 1214 | 13 | 1234 | 30 | **1211** | **12** | 2.6 |
| fe05c12 | 31 | 0.2109 | 1.46 | 2.3737 | 1.96 | 0.0816 | 1.88 | 1234 | 16 | 1235 | 14 | 1234 | 38 | **1234** | **13** | 0.0 |
| fe05b15 | 27 | 0.3485 | 1.22 | 5.7528 | 1.18 | 0.1197 | 0.94 | 1927 | 20 | 1939 | 10 | 1950 | 16 | **1941** | **10** | 1.2 |
| fe05d05 | 23 | 0.3566 | 2.06 | 6.1414 | 2.00 | 0.1249 | 0.88 | 1966 | 35 | 1996 | 17 | 2026 | 16 | **2026** | **16** | 3.0 |
| fe05b06 | 27 | 0.3270 | 2.38 | 5.7972 | 2.42 | 0.1285 | 1.16 | 1824 | 38 | 1946 | 21 | 2078 | 22 | **2078** | **22** | 12.2 |
| fe05b12 | 27 | 0.3858 | 2.12 | 6.8431 | 2.14 | 0.1286 | 2.20 | 2103 | 38 | 2091 | 19 | 2078 | 40 | **2092** | **19** | -1.2 |
| fe05d06 | 27 | 0.3921 | 2.06 | 7.4040 | 2.08 | 0.1369 | 0.76 | 2133 | 38 | 2161 | 19 | 2188 | 14 | **2188** | **14** | 2.5 |

i.s.= signal interval integrated for isotope-ratio and age calculation (in seconds)

disc%= percent discordance calculated from $^{207}Pb/^{206}Pb$ and $^{206}Pb/^{238}U$ ages (negative values: reverse discordance)