

# **U-Pb Detrital Zircon Geochronology Result for the Brennan Basin Member of the Duchesne River Formation, Duchesne 30' x 60' Quadrangle, Duchesne and Wasatch Counties, Utah**

*by*

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## INTRODUCTION

This report makes available raw analytical data from laboratory procedures completed to determine the age of rock samples collected during geologic investigations funded or partially supported by the Utah Geological Survey (UGS). The reference listed in table 1 provides additional information such as sample location, geologic setting, and significance or interpretation of the sample in the context of the area where it was collected. This report was prepared by Apatite to Zircon, Inc. under contract to the UGS. These data are highly technical in nature and proper interpretation requires considerable training in the applicable geochronologic techniques.

The data and methods are available at [http://geology.utah.gov/online/analytical\\_data.htm](http://geology.utah.gov/online/analytical_data.htm).

**Table 1.** Sample number and location.

Sample #	7.5' quadrangle	UTM Easting NAD83-12	UTM Northing NAD83-12	Reference
HC08132012-1	Hancock Cove	577949	4461548	Sprinkel (2014)

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## REFERENCE

Sprinkel, D.A., 2014, Interim geologic map of the east part of the Duchesne 30' x 60' quadrangle, Duchesne and Wasatch Counties, Utah (year 2): Utah Geological Survey Open-File Report 634, 27 p., 1 plate, scale 1:62,500.

# **UPbICP and Related Excel Workbooks: Software for Calculating U-Pb Zircon Ages and Presenting U-Pb Data Obtained by LA-ICP-MS**

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## **1. UPbICP Data Processing Software**

### **1.1. Background**

Two useful studies that demonstrate methods of U-Pb dating of zircon based on laser ablation-inductively coupled plasma-mass spectrometry (LA-ICP-MS) are Chang et al. (2006) and Gehrels et al. (2008). The U-Pb data analysis methods described here and the computer program UPbICP (written using Visual Basic 6.0, Microsoft Corporation) that implements them were developed by Apatite to Zircon, Inc. (A2Z) and remain the intellectual property of A2Z. A2Z offers no guarantees that the calculations performed by UPbICP are flawless but a good faith effort continues to be made to refine and verify the accuracy of the calculations. Original output data from the LA-ICP-MS system remain the property of the party that owns the sample from which they were obtained and UPbICP does not modify the original data.

*Dissemination and publication of the description provided here and the ideas and concepts contained herein are prohibited without the written permission of A2Z.*

### **1.2. Definitions**

A LA-ICP-MS session is characterized by a series of zircon *spots* (usually one per grain; **Figure 1**) that are ablated by a laser beam resulting in **laser ablation pits**. During laser ablation of a single spot, the ablated material is promptly transported from the deepening laser ablation pit into a mass spectrometer for a **scan** of the relative abundances of each target isotope. A series of scans is performed for each spot analyzed. A spot may be ablated for a zircon **age standard** having a known or accepted age, or for a zircon **unknown** having an unknown age. Spots for age standards are strategically interspersed with spots from unknowns. For each spot, measured **background** intensities for each isotope are obtained from scans performed prior to unblocking the laser. After the laser is unblocked, measured **background+signal** intensities are obtained from the remaining scans. During data analysis, **signal** intensities are calculated by subtracting respective background intensities from the background+signal intensities by a process referred to as **background correction**.

The U-Pb ages for a scan are deemed **concordant** if the  $^{207}\text{Pb}/^{235}\text{U}$  (where  $^{235}\text{U}$  is  $^{235}\text{U}$  calculated on the basis of measured  $^{238}\text{U}$ ),  $^{206}\text{Pb}/^{238}\text{U}$ , and  $^{207}\text{Pb}/^{206}\text{Pb}$  ages overlap each other at the  $2\sigma$  level. U-Pb ages that do not meet this criterion are deemed **discordant**. The **preferred age** for a spot is the U-Pb age for that spot deemed by UPbICP to be of highest quality where first preference is given to the

most precise weighted mean U-Pb age of the concordant scans (**Pb/U wmean conc.** for 206Pb/238U; **Pb/Pb wmean conc.** for 207Pb/206Pb) and second preference (used for spots exhibiting only discordant scans) is given to the most precise intercept age between the discordia and the concordia (**lower int.** for lower intercept; **upper int.** for upper intercept).

### 1.3. Data Collection and Age Calculation

Zircon grains prepared only for LA-ICP-MS U-Pb age dating are mounted in epoxide resin, cured at ~50°C for 3-10 hours, and polished to expose internal grain surfaces using 3.0µm Al<sub>2</sub>O<sub>3</sub> and 0.3µm Al<sub>2</sub>O<sub>3</sub> slurries on dedicated lap wheels. The polished zircon grains are then cleaned in reagent-grade 5.5M HNO<sub>3</sub> for 20s at 21°C. Zircon grains prepared for both zircon fission track (ZFT) and U-Pb age dating are mounted in FEP TEFLON and polished to expose internal grain surfaces using 3.0µm Al<sub>2</sub>O<sub>3</sub> and 0.3µm Al<sub>2</sub>O<sub>3</sub> slurries on dedicated lap wheels. Natural zircon fission tracks are revealed for optical viewing by immersing the grain mounts in an eutectic melt of NaOH+KOH (at ca. 220°C) for 12-72 hours. The polished and etched zircon grains are then cleaned in reagent-grade 48% HF for 15 minutes at 23°C.

Details concerning the LA-ICP-MS apparatus and data acquisition parameters routinely used by A2Z are summarized in **Table 1**. Images of spot 17103b\_20 are shown in **Figure 1** (the data in **Figures 2-15** except for **Figure 13b** pertain to spot 17103b\_20).

A2Z typically collects data from 255 scans per spot during ~40 seconds of acquisition time. Data for the isotopes used for U-Pb age calculation are collected during the first 250 scans. Data for 28Si and 91Zr are collected during the last 5 scans to verify zircon identity and to determine whether or not the laser has either dislodged the zircon grain from its mounting medium or passed through the zircon grain into the mounting medium. **Figure 2** presents 206Pb, 207Pb, and 238U data for spot 1713b\_20.

The laser is blocked from hitting the target zircon surface during the first ~ 45-55 scans and background intensities are recorded for the isotopes used to calculate U-Pb ages (background intensities are not collected for 28Si and 91Zr because background+signal intensities provide sufficient information for these isotopes). Background correction of the background+signal intensities for each isotope and scan and spot is performed by first fitting a polynomial equation to the background intensity versus scan number data (routine LFIT from Press et al., 1992):

$$B_{Fi} = \sum_{j=1}^{n+1} a_j^{j-1} i \quad \text{Equation 1}$$

where:

*i* = background intensity scan number

*B<sub>Fi</sub>* = fitted background value at scan *i*

*n* = order of polynomial; maximum=2

*a<sub>j</sub>* = fitted coefficient *j*

The polynomial is regressed from a selected range of scans (scans 1-40 are often used; linear regression is often used). The two highest background intensity values from the selected range of scans are eliminated prior to polynomial fitting to avoid outlier values (which are periodically observed; note the  $^{207}\text{Pb}$  outliers at scans 102 and 190 in **Figure 13b**; such spurious values may occur for any isotope at any scan and any spot). Acceptable polynomials are limited to those for which  $B_{Fnlast}$  (**Equation 1** where  $i = N_{last}$ , the last background intensity scan used for background correction) represents the minimum fitted value over the range of  $i$  values.

When the laser is unblocked and begins to ablate the surface of the zircon, background+signal intensities for the isotopes used for U-Pb age calculation are recorded through scan 250. For each isotope, a sum of multiple Gaussian equations is fitted to the background+signal intensity versus scan number data from a selected range of scans (scans 90-250 are often used; initial scan 90 is chosen to avoid complications that arise during the transition between background intensity data and background+signal intensity data):

$$BS_{Fi} = \sum_{j=1}^{N_{Gauss}} g_{1j} \exp \left\{ - \left[ \frac{(i - g_{2j})}{g_{3j}} \right]^2 \right\} \quad \text{Equation 2}$$

where:

$i$  = background+signal intensity scan number

$BS_{Fi}$  = fitted background+signal intensity at scan  $i$

$j$  = Gaussian equation number

$N_{Gauss}$  = number of Gaussian equations summed

$g_{1j}$  = amplitude of Gaussian equation  $j$

$g_{2j}$  = mean of Gaussian equation  $j$

$g_{3j}$  = standard deviation of Gaussian equation  $j$ .

The sum of Gaussian equations is comprised of a sufficient number of individual Gaussian equations to adequately fit most complicated background+signal intensity versus scan number data structures ( $N_{Gauss} = 10$  is usually the preferred number for **Equation 2**; numbers from 5-10 have been tested). Fitting is performed using the Levenberg-Marquardt method and chi-squared minimization (routine MRQMIN from Press et al., 1992). The  $\chi^2$  value from the Gaussian fitting is given by:

$$\chi^2 = \sigma_{SD}^2 N_{scan} \quad \text{Equation 3}$$

where:

$\chi^2$  = chi-squared statistic

$\sigma_{SD}$  = standard deviation of  $(BS_{Mi} - B_{Fnlast})$  about  $S_{Ci}$  (**Equation 6a** below)

$N_{scan}$  = number of signal intensity scans

The degrees of freedom of the fitted sum of Gaussian equations are given by:

$$\nu = N_{scans} - 3N_{Gauss}$$

**Equation 4**

where:

$\nu$  = degrees of freedom.

$N_{Gauss}$  = number of Gaussian equations summed

$N_{scan}$  = number of signal intensity scans

The probability of a worse fit ( $Q$  in **Equation 11** below) is calculated based on the number of degrees of freedom (**Equation 4**) and the  $\chi^2$  value from the Gaussian fitting (**Equation 3**) (routine GAMMQ from Press et al., 1992). A data filtering procedure is used by which all background+signal intensity values are first fitted and then those background+signal intensity values that fall outside of 2 standard deviations from their fitted counterparts are eliminated (e.g., note the spurious background+signal intensity values at scans 102 and 190 in **Figure 13b**). The process of fitting a sum of multiple Gaussian equations is performed a second time using the filtered background+signal intensity values

Background correction of the background+signal intensity data for an isotope is performed by subtracting the background intensity at the last background scan ( $B_{F40}$  for a scan range of 1-40; **Equation 1**) from the fitted background+signal intensity at each background+signal scan.

$$S_{Ci} = BS_{Fi} - B_{FNlast}$$

**Equation 5**

where:

$i$  = background+signal intensity scan number; signal intensity scan number after background correction

$S_{Ci}$  = calculated signal intensity at signal intensity scan  $i$

$BS_{Fi}$  = fitted background+signal intensity at scan  $i$  (**Equation 2**)

$N_{last}$  = scan number of the last background intensity scan

$B_{FNlast}$  = fitted background intensity at background intensity scan  $N_{last}$   
(**Equation 1** where  $i = N_{last}$ )

If  $S_{Ci} < 0$  due to this subtraction,  $S_{Ci}$  is set to 0. The standard error of the calculated signal intensity for each isotope for each spot is assumed constant for all regressed scans; this standard error is set equal to the standard error of the unfitted background+signal intensities about their fitted counterparts:

$$\sigma_{SD} = \left( \left( \frac{1}{N_{scan}} \right) \sum_{i=1}^{N_{scan}} ((BS_{Mi} - B_{FNlast}) - S_{Ci})^2 \right)^{1/2}$$

**Equation 6a**

where:

$i$  = background+signal intensity scan number; signal intensity scan number after background correction

$\sigma_{SD}$  = standard deviation of  $(BS_{Mi} - B_{FNlast})$  about  $S_{Ci}$

$N_{scan}$  = number of signal intensity scans

$S_{Ci}$  = calculated signal intensity at signal intensity scan  $i$  (**Equation 5**)

$BS_{Mi}$  = measured background+signal intensity at scan  $i$

$N_{last}$  = scan number of the last background intensity scan

$B_{Fnlast}$  = fitted background intensity at background intensity scan  $N_{last}$

(**Equation 1** where  $i = N_{last}$ )

$$\sigma_{SE} = \frac{\sigma_{SD}}{\sqrt{N_{Gauss}}}$$
 **Equation 6b**

where:

$i$  = background+signal intensity scan number; signal intensity scan number after background correction

$\sigma_{SE}$  = standard error of  $(BS_{Mi} - B_{Fnlast})$  about  $S_{Ci}$

$\sigma_{SD}$  = standard deviation of  $(BS_{Mi} - B_{Fnlast})$  about  $S_{Ci}$  (**Equation 6a**)

$N_{Gauss}$  = number of Gaussian equations summed

$N_{scan}$  = number of signal intensity scans

After scan 250, five scans of background+signal intensities are recorded for 28Si and 91Zr. For a given spot, the median value for each isotope from the values recorded is used to avoid spurious values. Under typical analytical conditions, zircon yields a 91Zr/28Si ratio of median background+signal intensities in the range 0.5-2.0. For a whole LA-ICP-MS session, the 91Zr/28Si ratios of median background+signal intensities exhibit a standard deviation in the range 0.1-0.2. If the laser has passed through a zircon into the mounting medium, this ratio decreases due to the faster decay rate of the 91Zr background+signal intensity relative to the decay of the 28Si background+signal intensity. A median 91Zr/28Si ratio for all primary age standard spot ratios for a session is determined and the standard deviation about this median (excluding the two minimum and two maximum values to avoid any possible problematic spots) is calculated. Only secondary age standard spots and unknown spots exhibiting 91Zr/28Si ratios within 3 standard deviations of the median of the medians for the primary age standard are considered acceptable spots for U-Pb age calculation. The mineral baddeleyite yields 91Zr/28Si ratios of median background+signal intensities in the range 20-40. Other non-zircon mineral species encountered yield 91Zr/28Si ratios less than 0.2.

The common Pb correction in UPbICP is performed as described in **Appendix 1**. This correction is not routinely invoked, however. Instead, spots are rejected from further data analysis if sufficient 204Pb is encountered in at least one scan (see section **2.2.1** below).

UPbICP calculates U-Pb ages using the following conventions (Steiger and Yäger, 1977):

$$235\text{U}/238\text{U} = 1 / 137.88$$

$$\lambda_{235\text{U}} = 0.00098485 \text{ Ma}^{-1}$$

$$\lambda_{238\text{U}} = 0.000155125 \text{ Ma}^{-1}$$

$$\lambda_{232\text{Th}} = 0.000049475 \text{ Ma}^{-1}$$

Two types of age standards are used during each LA-ICP-MS session (**Table 2**). A **primary age standard** is used to characterize isotopic fractionation factors for all scans and all spots for which U-Pb age calculations are to be performed. A series of **secondary age standards** are used to correct fractionation factors from the primary age standard for the effect of relative  $\alpha$ -radiation damage on laser penetration rate at each spot. The primary age standard, usually FC-1 obtained from the University of Minnesota student Geology Club (Paces and Miller, 1993), is analyzed in pairs at the beginning and end of each session and every 20-30 spots during a session. The isotopic ratios used to calculate fractionation factors for primary age standard FC-1 are (**Table 2**; obtained from the University of Minnesota student Geology Club; Paces and Miller, 1993):

$$\begin{aligned} \text{207Pb/235U} &= 1.951607 \text{ (accepted)} \\ \text{206Pb/238U} &= 0.185877 \text{ (accepted)} \\ \text{207Pb/206Pb} &= 0.076149 \text{ (accepted)} \\ \text{208Pb/232Th} &= 0.055878 \text{ (assumed)} \end{aligned}$$

Fractionation factors for the isotopic ratios  $207\text{Pb}/235\text{U}_c$ ,  $206\text{Pb}/238\text{U}$ , and  $207\text{Pb}/206\text{Pb}$  are calculated for every signal scan and spot position for which primary age standard data are measured. For  $206\text{Pb}/238\text{U}$  (similar equations can be written for  $207\text{Pb}/235\text{U}_c$  and  $207\text{Pb}/206\text{Pb}$ ):

$$FF_{C206/238i,k} = \frac{\left( \frac{^{206}\text{Pb}}{^{238}\text{U}} \right)_{\text{accepted}}}{\left( \frac{S_{C206i,k}}{S_{C238i,k}} \right)} \quad \text{Equation 7}$$

where:

$i$  = signal intensity scan number

$k$  = primary age standard spot position in session

$FF_{C206/238i,k}$  =  $206\text{Pb}/238\text{U}$  fractionation factor at signal intensity scan  $i$   
based on individual calculated signal intensities and spot position  $k$

$S_{C206i,k}$  = calculated  $206\text{Pb}$  signal intensity at signal intensity scan  $i$  and  
spot position  $k$  (**Equation 5**)

$S_{C238i,k}$  = calculated  $238\text{U}$  signal intensity at signal intensity scan  $i$  and spot  
position  $k$  (**Equation 5**)

At fixed signal scan number  $i$ , a polynomial equation is fitted to spot number (abscissa) versus fractionation factor (ordinate) for each isotopic ratio over the range of primary age standard spot positions throughout the session. For  $206\text{Pb}/238\text{U}$  (similar equations can be written for  $207\text{Pb}/235\text{U}_c$  and  $207\text{Pb}/206\text{Pb}$ ):

$$FF_{F206/238i,k} = \sum_{j=1}^{n+1} f_{i,j}^{j-1} k \quad \text{Equation 8a}$$

where:

$k$  = spot number in session

$i$  = fixed signal scan number

$FF_{F206/238i,k}$  = fitted  $206\text{Pb}/238\text{U}$  fractionation value at signal scan  $i$  and spot number  $k$

$n$  = order of polynomial; maximum 4

$f_{i,j}$  = fitted coefficient  $j$  for signal scan  $i$

$$\sigma_{t206/238} = \left( \left( \frac{1}{N_{PAS}} \right) \sum_{j=1}^{N_{PAS}} \left( t_{206/238j} - \frac{\sum_{j=1}^{N_{PAS}} t_{206/238j}}{N_{PAS}} \right)^2 \right)^{1/2} \quad \text{Equation 8b}$$

where:

$j$  = primary age standard number

$\sigma_{t206/238}$  = standard deviation of  $206\text{Pb}/238\text{U}$  ages for the primary age standard

$N_{PAS}$  = number of primary age spots

$t_{206/238j}$  =  $206\text{Pb}/238\text{U}$  age for primary age standard  $j$

A data filtering procedure is used whereby all points are fitted and those points that fall outside of 2 standard deviations (function STEYX; Excel 2007) are eliminated and the remaining points are re-fitted and the resulting regression used. Relative errors of the  $207\text{Pb}/235\text{Uc}$ ,  $206\text{Pb}/238\text{U}$ , and  $207\text{Pb}/206\text{Pb}$  fractionation factors are calculated as follows: a) the primary age standard is treated as an unknown and ages for each isotopic ratio are calculated for each spot, b) the mean preferred ages and their respective standard deviations for all spots for each isotopic ratio are calculated, c) the relative error of each fractionation factor is set equal to its respective standard deviation divided by its respective mean preferred age, and d) fractionation factor relative errors are assumed constant throughout the session.

Fractionation factors are adjusted for variable laser penetration rate (and hence variable ablation pit depth at any given scan number from spot to spot) due to variable  $\alpha$ -radiation damage using the following scheme:

- A secondary age standard is analyzed in pairs along with the primary age standard (recent sessions use TR as this secondary age standard; see **Table 2**). An additional series of secondary age standards is analyzed at the beginning and again at the end of a LA-ICP-MS session. Recent sessions include 10 spots each (5 near the beginning and 5 near the end of each session) for four additional secondary age standards (recent sessions use FC-5z, MD, IFCan, and TEM2; see **Table 2**). Preferred ages are calculated for each secondary age standard spot using uncorrected fractionation factors from the primary age standard. Relative  $\alpha$ -radiation damage is calculated for each spot by integrating function *dAlphaEnergy* (**Appendix 2**) over all signal scans.

- For each secondary age standard, the weighted mean of preferred ages from the youngest, concordant population of spots is calculated and the mean relative  $\alpha$ -radiation damage for the population of spots is calculated. Weighted mean ages calculated for the secondary age standards using uncorrected fractionation factors from the primary age standard are observed to deviate linearly from their accepted ages as a function of the relative  $\alpha$ -radiation damage, with fractionation being greater in older, more  $\alpha$ -radiation damaged zircons (**Figure 3**). Presumably, this is due to greater penetration rates of the laser into softer, more highly  $\alpha$ -radiation damaged zircon. To permit the correct ages of the secondary age standards to be calculated, a fractionation factor correction is applied as shown in *sub AlphaEnergyFF* in **Appendix 2** and discussed further in **Section 2.5**.

U-Pb preferred ages are calculated for a spot using the following scheme:

- For age standards, UPbICP characterizes signal intensities of each isotope at each scan and each spot for which U-Pb age calculations are to be performed. Fractionation factors from the primary age standard are corrected for  $\alpha$ -radiation using secondary age standards.
- For unknowns, UPbICP characterizes signal intensities of each isotope at each scan and each spot for which U-Pb age calculations are to be performed. As such, each scan can be considered an unique U-Pb analysis and each spot can be considered composed of a series of downhole U-Pb analyses.
- The  $207\text{Pb}/206\text{Pb}$  fractionation factor for the primary age standard pertaining to each scan is forced to be 1 at all scans and all spots.
- The combination of isotopic ratios for unknowns and fractionation factors from age standards at each scan and each spot permits the calculation of three ages for each scan for each unknown; these are: 1)  $207\text{Pb}/235\text{Uc}$  age, 2)  $206\text{Pb}/238\text{U}$  age, and 3)  $207\text{Pb}/206\text{Pb}$  age.
- A discordia ( $207\text{Pb}/235\text{Uc}$  abscissa,  $206\text{Pb}/238\text{U}$  ordinate) can be characterized for each spot from an unknown. Ages corresponding to the upper and lower intercepts of the discordia with the concordia are calculated by: a) linear regression of the ( $207\text{Pb}/235\text{Uc}$ ,  $206\text{Pb}/238\text{U}$ ) points for the spot and b) Monte Carlo simulation of the discordia using 1000 randomly generated lines based on the regression-derived slope and intercept values and their respective errors. The quality of these ages increases with increasing number of discordant scans, and increasing range of discordance of the scans.
- *Preferred age based on the weighted mean isotopic ratios of concordant scans:* This strategy seeks the weighted mean of all isotopic ratios from concordant scans. A scan is concordant if its  $207\text{Pb}/235\text{Uc}$  age,  $206\text{Pb}/238\text{U}$  age, and  $207\text{Pb}/206\text{Pb}$  age overlap each other at the  $2\sigma$  level (see below). According to this strategy, if at least one scan is concordant, the weighted mean of the  $207\text{Pb}/235\text{Uc}$  ratios from all concordant scans is used to calculate the  $207\text{Pb}/235\text{Uc}$  age (function *Age207235* in **Appendix 3**), the weighted mean of the  $206\text{Pb}/238\text{U}$  ratios from all concordant scans is used to calculate the  $206\text{Pb}/238\text{U}$  age (function *Age206238* in **Appendix 3**), and the weighted mean of the  $207\text{Pb}/206\text{Pb}$  ratios from all concordant scans is used to calculate the

$^{207}\text{Pb}/^{206}\text{Pb}$  age for the spot (function  $\text{Age}207206$  in **Appendix 3**). The error for each of these ages is based on the weighted mean error of its respective weighted mean ratio combined with the relative error of its respective fractionation factor. For  $^{206}\text{Pb}/^{238}\text{U}$  ages, the equations are:

$$R_{206/238i,k} = FF_{F206/238i,k} \left( \frac{S_{C206i,k}}{S_{C238i,k}} \right) \quad \text{Equation 9a}$$

where:

$k$  = spot number in session

$i$  = fixed signal scan number

$R_{206/238i,k}$  =  $^{206}\text{Pb}/^{238}\text{U}$  ratio based on calculated  $^{206}\text{Pb}$  and  $^{238}\text{U}$  signal intensities at signal intensity scan  $i$  and spot position  $k$

$FF_{F206/238i,k}$  = fitted  $^{206}\text{Pb}/^{238}\text{U}$  fractionation value at signal scan  $i$  and spot number  $k$  (**Equation 8a**)

$S_{C206i,k}$  = calculated  $^{206}\text{Pb}$  signal intensity at signal intensity scan  $i$  and spot position  $k$  (**Equation 5**)

$S_{C238i,k}$  = calculated  $^{238}\text{U}$  signal intensity at signal intensity scan  $i$  and spot position  $k$  (**Equation 5**)

$$t_{206/238i,k} = \text{Age}206238(R_{206/238i,k}) \quad \text{Equation 9b}$$

where:

$k$  = spot number in session

$i$  = fixed signal scan number

$\text{Age}206238$  = function for age calculation (**Appendix 3**)

$t_{206/238i,k}$  =  $^{206}\text{Pb}/^{238}\text{U}$  age at signal intensity scan  $i$  and spot position  $k$

$R_{206/238i,k}$  =  $^{206}\text{Pb}/^{238}\text{U}$  ratio based on calculated  $^{206}\text{Pb}$  and  $^{238}\text{U}$  signal intensities at signal intensity scan  $i$  and spot position  $k$  (**Equation 9a**)

$$\sigma_{R206/238i,k} = R_{206/238i,k} \sqrt{\left( \frac{\sigma_{SE206}}{S_{C206i,k}} \right)^2 + \left( \frac{\sigma_{SE238}}{S_{C238i,k}} \right)^2 + \left( \frac{\sigma_{t206/238}}{\sum_{j=1}^{N_{PAS}} t_{206/238j}} \right)^2} \quad \text{Equation 9c}$$

where:

$k$  = fixed spot number in session

$i$  = signal scan number

$j$  = primary age standard number

$\sigma_{R206/238i,k}$  = absolute error for  $R_{206/238i,k}$

$R_{206/238i,k}$  = 206Pb/238U ratio based on calculated 206Pb and 238U signal intensities at signal intensity scan  $i$  and spot position  $k$  (**Equation 9a**)

$\sigma_{SE206}$  = standard error of  $(BS_{Mi} - B_{Fnlast})$  about  $S_{Ci}$  for 206Pb (**Equation 6b**)

$\sigma_{SE238}$  = standard error of  $(BS_{Mi} - B_{Fnlast})$  about  $S_{Ci}$  for 238U (**Equation 6b**)

$S_{C206i,k}$  = calculated 206Pb signal intensity at signal intensity scan  $i$  and spot position  $k$  (**Equation 5**)

$S_{C238i,k}$  = calculated 238U signal intensity at signal intensity scan  $i$  and spot position  $k$  (**Equation 5**)

$\sigma_{t206/238}$  = standard deviation of 206Pb/238U ages for the primary age standard

$N_{PAS}$  = number of primary age standard spots

$t_{206/238j}$  = 206Pb/238U age for primary age standard  $j$  (**Equation 8b**)

$$\sigma_{wmcR206/238k} = \left( \sum_{i=1}^{N_{CScan}} \frac{1}{(\sigma_{R206/238i,k})^2} \right)^{-1/2} \quad \text{Equation 9d}$$

where:

$k$  = fixed spot number in session

$i$  = concordant scan number  $i$

$\sigma_{wmcR206/238k}$  = weighted mean error of  $R_{206/238i,k}$  values for all concordant scans at fixed spot number  $k$

$\sigma_{R206/238i,k}$  = absolute error for  $R_{206/238i,k}$  (**Equation 9c**)

$N_{CScan}$  = number of concordant scans at fixed spot number  $k$

$$R_{wmcR206/238k} = \frac{\sum_{i=1}^{N_{CScan}} \frac{R_{206/238i,k}}{(\sigma_{R206/238i,k})^2}}{\sum_{i=1}^{N_{CScan}} \frac{1}{(\sigma_{R206/238i,k})^2}} \quad \text{Equation 9e}$$

where:

$k$  = fixed spot number in session

$i$  = concordant scan number  $i$

$R_{wmc206/238k}$  = weighted mean of  $R_{206/238i,k}$  values for all concordant scans at fixed spot number  $k$

$\sigma_{wmcR206/238k}$  = weighted mean error of  $R_{206/238i,k}$  values for all concordant scans at fixed spot number  $k$  (**Equation 9d**)

$R_{206/238i,k}$  = 206Pb/238U ratio based on calculated 206Pb and 238U signal intensities at signal intensity scan  $i$  and spot position  $k$  (**Equation 9a**)

$\sigma_{R206/238i,k}$  = absolute error for  $R_{206/238i,k}$  (**Equation 9c**)

$N_{CScan}$  = number of concordant scans at fixed spot number  $k$

$$t_{wmc206/238k} = Age206238(R_{wmc206/238k})$$

**Equation 9f**

where:

$k$  = spot number in session

$Age206238$  = function for age calculation (**Appendix 3**)

$t_{wmc206/238k}$  = weighted mean concordant 206Pb/238U age at spot position  $k$

$R_{wmc206/238k}$  = weighted mean of  $R_{206/238i,k}$  values for all concordant scans at fixed spot number  $k$

$$\sigma_{twmc206/238k} = Age206238(\sigma_{wmcR206/238k})$$

**Equation 9g**

where:

$k$  = spot number in session

$\sigma_{twmc206/238k}$  = weighted mean concordant 206Pb/238U age error at spot position  $k$

$\sigma_{wmcR206/238k}$  = weighted mean error of  $R_{206/238i,k}$  values for all concordant scans at fixed spot number  $k$  (**Equation 9d**)

The above equations apply to the 207Pb/235Uc system as well. For the 207Pb/206Pb system, the 207Pb/206Pb ratio at 0 Ma is approximately 0.046003. The weighted mean concordant 207Pb/206Pb age error is calculated as follows:

$$\sigma_{twmc207/206k} = Age207206(R_{wmcR206/238k} + \sigma_{wmcR207/206k}) - Age207206(\sigma_{wmcR207/206k})$$

**Equation 9h**

where:

$k$  = spot number in session

$Age207206$  = function for age calculation (**Appendix 3**)

$\sigma_{twmc207/206k}$  = weighted mean concordant 207Pb/206Pb age error at spot position  $k$

$R_{wmc207/206k}$  = weighted mean of  $R_{207/206i,k}$  values for all concordant scans at fixed spot number  $k$  (**Equation 9e** for 207Pb/206Pb)

$\sigma_{wmcR207/206k}$  = weighted mean error of  $R_{207/206i,k}$  values for all concordant scans at fixed spot number  $k$  (**Equation 9d** for 207Pb/206Pb)

The preferred age for a spot exhibiting at least one concordant scan is either the 206Pb/238U or the 207Pb/206Pb age, whichever exhibits the lowest relative error.

- *Preferred age based on discordia intercepts with the concordia:* A discordia (207Pb/235Uc abscissa, 206Pb/238U ordinate) is characterized for each spot from an unknown by linear regression of the (207Pb/235Uc, 206Pb/238U) points for the spot, giving a regression intercept and regression intercept error, and a regression slope and regression slope error (routine LFIT in Press et al., 1992). The lower intercept age is the lowest age consistent with a point of intersection between the discordia and the concordia. The upper intercept age is the highest of

two ages consistent with two points of intersection between the discordia and the concordia. Sometimes, for example when the laser ablates zircon containing a mixture of two distinct age domains, the number of concordant scans is zero. For these spots, the preferred age is set equal to either the age of the upper or lower intercept between the discordia and the concordia. The error for each of these ages is obtained from the standard deviation of the respective intercept ages derived from the Monte Carlo simulation of the discordia combined with the relative errors of the  $^{206}\text{Pb}/^{238}\text{U}$  and the  $^{207}\text{Pb}/^{206}\text{Pb}$  fractionation factors.

$$\sigma_{t_{\text{int}}} = t_{\text{int}} \left( \left( \frac{\sigma_{MC_{\text{int}}}}{t_{\text{int}}} \right)^2 + \left( \frac{\sigma_{t_{206/238}}}{\sum_{j=1}^{N_{PAS}} t_{206/238j}} \right)^2 + \left( \frac{\sigma_{t_{207/235c}}}{\sum_{j=1}^{N_{PAS}} t_{207/235cj}} \right)^2 \right)^{1/2}$$

**Equation 10**

where:

$j$  = primary age standard number

$\sigma_{t_{\text{int}}}$  = intercept age error

$t_{\text{int}}$  = intercept age

$\sigma_{MC_{\text{int}}}$  = standard deviation of Monte Carlo-derived intercept ages about  $t_{\text{int}}$

$N_{PAS}$  = number of primary age spots

$\sigma_{t_{206/238}}$  = standard deviation of  $^{206}\text{Pb}/^{238}\text{U}$  ages for the primary age standard

$t_{206/238j}$  =  $^{206}\text{Pb}/^{238}\text{U}$  age for primary age standard  $j$  (**Equation 8b**)

$\sigma_{t_{207/235c}}$  = standard deviation of  $^{207}\text{Pb}/^{235}\text{Uc}$  ages for the primary age standard

$t_{207/235cj}$  =  $^{207}\text{Pb}/^{235}\text{Uc}$  age for primary age standard  $j$  (**Equation 8b**)

The age chosen as the preferred age is the intercept age that exhibits the lowest relative error.

## 2. Workbook: UPbICPReport\_

### 2.1. General

LA-ICP-MS-based U-Pb zircon analysis of a single geological sample is comprised of a series of spots analyzed on a series of zircon grains (usually one spot per grain). An Excel 97-2003 workbook **UPbICPReport\_** is provided containing 7 worksheets that present summary data from UPbICP for this series of grains. It is necessary to ‘enable macros’ for this worksheet to work properly. If a crash occurs during any code execution, it is necessary to either ‘reset’ Visual Basic execution prior to trying again or to close and then reopen the workbook.

- **Worksheet: Data** provides access to and permits the setting of data quality filters; and it presents a single line of calculated ages and related data for each spot analyzed.
- **Worksheet: Calculate** presents options and results related to age population and age spectrum calculations.
- **Worksheet: Plots** presents age spectra plots, age data plots, and several plots involving U and Th concentrations.
- **Worksheet: Standard\_Statistics** presents data from the primary and secondary age standards used to calculate ages.
- **Worksheet: A2Z\_Standards** presents preferred ages calculated for internal age standards used at A2Z from the LA-ICP-MS session(s) indicated.
- **Worksheet: Comment** is used to verify whether or not data have been loaded into arrays prior to the initiation of various calculations in **Calculate**.
- **Worksheet: Results\_Settings** presents a summary of the age data for the preferred grain age population as specified in **Calculate** and the data quality filters as specified in **Data**.

## 2.2. Worksheet: Data (Figure 4)

**2.2.1. Data quality filters.** Several data quality filters are employed for objective filtering of preferred ages. The purpose of these quality filters is to parse the data from the series of spots analyzed and distinguish zircon spots from non-zircon spots, or zircon spots that show evidence of problematic data.

- **Min Fit Quality** (cell B4)

The  $\chi^2$  value (**Equation 3**) and number of degrees of freedom (**Equation 4**) for the fit of the sum of Gaussian equations to each isotope permits the probability of worse fit to be calculated (routine GAMMQ in Press et al.,1992). If the probability of worse fit  $Q$  for any one of the isotopes used to calculate ages (206Pb, 207Pb, 208Pb, 232Th, and 238U) is less than this parameter (<0.0500 is commonly used; cell B4 shows the value of this data quality filter; columns Y and AH show the values for each spot), the preferred age is rejected from further analysis. This parameter can be set by the user.

$$QF_{\text{fit}} = \min(Q_{206\text{Pb}}, Q_{207\text{Pb}}, Q_{208\text{Pb}}, Q_{232\text{Th}}, Q_{238\text{U}}) \quad \text{Equation 11}$$

where:

$QF_{\text{fit}}$  = minimum  $Q$  value (columns Y and AH); maximum probability of worse fit value

$Q_{206\text{Pb}}$ , etc. =  $Q$  value from GAMMQ for the sum of Gaussian equations fitted to the 206Pb background+signal intensities, etc.

- **Min Clean Quality** (cell D4)

During the fitting of the sum of Gaussian equations for an isotope, a first pass is performed and then outliers greater than 2 standard deviations from the sum of fitted Gaussian equations are rejected and the fitting process is repeated. In some cases, this filtering process causes a sequence of consecutive and potentially useful scans to be

rejected. If the inverse of the number of consecutive, rejected scans for any one of the isotopes used to calculate ages (206Pb, 207Pb, 208Pb, 232Th, and 238U) is less than this parameter (<0.3333 is commonly used; cell D4 shows the value of this data quality filter; columns Z and AI show the values for each spot), the preferred age is rejected from further analysis. This parameter can be set by the user.

$$QF_{clean} = \min\left(\left(\frac{1}{CO_{206Pb}}\right), \left(\frac{1}{CO_{207Pb}}\right), \left(\frac{1}{CO_{208Pb}}\right), \left(\frac{1}{CO_{232Th}}\right), \left(\frac{1}{CO_{238U}}\right)\right)$$

**Equation 12**

where:

$QF_{clean}$  = minimum  $CO^{-1}$  value (columns Z and AI); maximum number of consecutive background+signal intensities cleaned for each isotope  
 $CO_{206Pb}$ , etc. =  $CO$  value for 206Pb background+signal intensities, etc.

If this parameter is <0.3333 for a spot (a maximum of 3 consecutive rejected outliers is acceptable), the preferred age is rejected from further analysis. This parameter can be user set.

- **Min 204Pb Quality** (cell F4)

The presence of significant 204Pb indicates the presence of significant common Pb in the target zircon. For each scan, the ratio of the background corrected 204Pb signal intensity to the background 204Pb intensity is determined and the maximum value of this ratio for scans for the spot is determined. If this maximum ratio is less than the set value of this data quality parameter (<0.3333 is commonly used; cell F4 shows the value of this data quality filter; columns AA and AJ show the values for each spot), the preferred age is rejected from further analysis. This parameter can be set by the user.

$$QF_{204Pb} = \max\left(\frac{S_{C204Pbi}}{B_{F204PbNlast}}\right) \text{ for } i=1 \text{ to } N_{\text{scan}}$$

**Equation 13**

where:

$QF_{204Pb}$  = minimum signal intensity divided by background correction intensity (columns AA and AJ)

$S_{C204Pbi}$  = 204(Hg+Pb) signal intensity at signal intensity scan  $i$  (**Equation 5**)

$N_{\text{last}}$  = scan number of the last background intensity scan

$B_{F204PbNlast}$  = fitted 204(Hg+Pb) background intensity at background intensity scan  $N_{\text{last}}$  (**Equation 1** where  $i = N_{\text{last}}$ )

- **Min Uppm Quality** (cell H4)

This quality parameter was originally designed to eliminate spots in grains that were likely not zircon, using an old LA-ICP-MS data collection protocol that did not permit use of the **Mineral (or “n.a.”)** filter (see next paragraph). Spots that exhibit

Uppm values (column I) < 9 are eliminated from further analysis. This parameter can be user set.

- **Mineral (or “n.a.”)** (cell J4)

The current LA-ICP-MS data collection protocol used by A2Z includes the analysis of 28Si and 91Zr near the bottom of a laser ablation pit. A spot that exhibits a 91Zr/28Si ratio within  $3\sigma$  of the median value obtained for all zircon spot analyses of the primary age standard during a LA-ICP-MS session are deemed to be zircon.

These analyses are indicated by the extension ‘\_Zrn’ at the end of the Analysis\_# in column A. Non-zircon spots, or spots for which the laser has either blasted the grain out of the mounting medium or passed through the grain into the mounting medium (the latter causing a faster decay of the 91Zr background+signal intensity relative to the 28Si background+signal intensity), do not have this extension added. This parameter can be user set to “n.a.” to eliminate this filter entirely.

**2.2.2. Column contents.** The contents of each column are described below.

### Measured Isotopic Ratios

- **Analysis\_#** (column A)

Extension ‘\_Zrn’ indicates a spot that exhibits a 91Zr/28Si ratio within  $3\sigma$  of the median value obtained for all zircon spot analyses of the primary age standard during a LA-ICP-MS session.

- **207Pb/235Uc\*** (column B)
- **207Pb/235Uc\* ( $\pm 2\sigma$ )** (column C)
- **206Pb/238U** (column D)
- **206Pb/238U ( $\pm 2\sigma$ )** (column E)
- **207Pb/206Pb** (column F)
- **207Pb/206Pb ( $\pm 2\sigma$ )** (column G)

The ratios are weighted means of the signal ratios for all concordant scans. A scan is concordant if its 207Pb/235U<sub>c</sub>, 206Pb/238U, and 207Pb/206Pb ages overlap each other at the  $2\sigma$  level. The ratio errors represent the errors of the respective weighted mean ratios combined with the relative errors for the respective fractionation factors. A value of ‘undefined’ appears when there are no concordant scans. A value of ‘poor data’ or ‘-’ appears when the data do not permit the calculation of realistic U-Pb ages.

### U and Th

- **U (ppm)** (column I)
- **Th (ppm)** (column J)
- **U/Th** (column K)

The average U and Th concentrations for primary age standard FC-1 are assumed to be equal to the weighted mean values shown in **Table 3** (data from Paces and Miller, 1993). A median value of average 235Uc signal intensity per scan and a median value of average 232Th signal intensity per scan are determined for all FC-1 primary age standard spots. The assumed average U and Th concentrations for the primary age standard FC-1 are divided by these ratios giving Uppm\*scan/235Uc signal intensity

and Thppm\*scan/232Th signal intensity factors. These factors are then used to convert 235Uc signal intensity/scan and 232Th signal intensity/scan to Uppm and Thppm for all scans (age standards and unknowns). For unknown and secondary age standard spots exhibiting at least 1 concordant scan, the average Uppm and Thppm concentrations for the concordant scans is reported here. For spots exhibiting no concordant scans, the average Uppm and Thppm concentrations for the discordant scans are reported here.

## Apparent Ages

- **Analysis\_#** (column M)

Unlike column A, no extension is added to analysis number to indicate zircon.

- **207Pb/235Uc\*** (column N)
- **207Pb/235Uc\* ( $\pm 2\sigma$ )** (column O)
- **206Pb/238U** (column P)
- **206Pb/238U ( $\pm 2\sigma$ )** (column Q)
- **207Pb/206Pb** (column R)
- **207Pb/206Pb ( $\pm 2\sigma$ )** (column S)

These ages and errors are based on the **Measured Isotopic Ratios** (columns B-G) described above.

## Preferred Ages

- **Analysis\_#** (column U)

Unlike column A, no extension is added to analysis number to indicate zircon.

- **Preferred Age (Ma)** (column V)

*Preferred age based on the weighted mean isotopic ratios of concordant scans:* This strategy seeks the weighted mean of all isotopic ratios from concordant scans. The preferred age for a spot exhibiting at least one concordant scan is either the 206Pb/238U or the 207Pb/206Pb age (based on weighted mean isotopic ratios from concordant scans), whichever exhibits the lowest relative error. *Preferred age based on discordia intercepts with the concordia:* For spots exhibiting no concordant scans, the preferred age is set equal to either the age of the upper or lower intercept between the discordia and the concordia. The error for each of these ages is obtained from the standard deviation of the respective intercept ages derived from the Monte Carlo simulation of the discordia combined with the relative errors of the 206Pb/238U and the 207Pb/206Pb fractionation factors. The age chosen as the preferred age is the intercept age that exhibits the lowest relative error. A value of ‘undefined’ appears when a discordia cannot be fitted (e.g., all points lying above the concordia). A value of ‘undefined’ appears specifically for the upper intercept age and error if there is no upper intercept between the discordia and concordia.

- **Absolute Error ( $\pm 2\sigma$ )** (column W)

The error, including fractionation factor error, of the preferred age described above.

- **Age Type** (column X)

Types include a) ‘Pb/U wmean conc.’= 206Pb/238U weighted mean concordant, b) ‘Pb/Pb wmean conc.’= 207Pb/206Pb weighted mean concordant, c) ‘lower int.’=age

based on the lower intercept between the discordia and the concordia, and d) ‘upper int.’ = age based on the upper intercept between the discordia and the concordia. A value of ‘poor data’ or ‘-’ appears when the data do not permit the calculation of realistic U-Pb ages.

- **Fit Quality 1=best** (column Y)

This value indicates the minimum probability of worse fit for all of the isotopes used for calculate ages (206Pb, 207Pb, 208Pb, 232Th, and 238U).

- **Clean Quality 1=best** (column Z)

This value indicates the maximum value of [1 / (maximum number of consecutive rejected outliers)] for all of the isotopes used to calculate ages (206Pb, 207Pb, 208Pb, 232Th, and 238U).

- **204Pb Quality 1=best** (column AA)

This value indicates the minimum value of [(204(Hg+Pb)\_background / (204(Hg+Pb)\_signal + 204(Hg+Pb)\_background)] for all data scans from a spot.

- **2σ Conc. Scans** (column AB)

This value indicates the number of concordant scans for the spot.

## Preferred Ages - Sorted

- **Analysis\_#** (column AD)
- **Preferred Age (Ma)** (column AE)
- **Absolute Error ( $\pm 2\sigma$ )** (column AF)
- **Age Type** (column AG)
- **Fit Quality 1=best** (column AH)
- **Clean Quality 1=best** (column AI)
- **204Pb Quality 1=best** (column AJ)
- **2σ Conc. Scans** (column AK)

These are the same data as provided in columns U-AB; however, the data here have been sorted in ascending order of the value in column AE.

## Fitted Discordia

- **Age-Lower Intercept (Ma)** (column AM)

A discordia (207Pb/235Uc abscissa, 206Pb/238U ordinate) is characterized for each spot from an unknown by linear regression of the (207Pb/235Uc, 206Pb/238U) points for the spot, giving a regression intercept and regression intercept error, and a regression slope and regression slope error. The regression is performed using routine LFIT in Press et al. (1992). The lower intercept age is the lowest age consistent with a point of intersection between the discordia and the concordia. If the regression intercept is <0 then a value of ‘undefined’ appears here. If the lower intercept age is <0 Ma or >4560 Ma then a value of ‘undefined’ appears here.

- **Age-Lower Intercept ( $\pm 2\sigma$ )** (column AN)

See comments for column AM above. The errors for the upper and lower intercepts of the discordia with the concordia are calculated by Monte Carlo simulation of the discordia using 1000 randomly generated lines based on the regression-derived slope and intercept values and their respective errors. If a value other than ‘undefined’

appears in column AM, the standard deviation of the Monte Carlo-derived lower intercepts is calculated about the lower intercept age, ignoring any intercepts  $<0$  Ma or  $>4560$  Ma. The  $\pm 2\sigma$  error on the lower intercept age is calculated based on the relative errors of the lower intercept age and the  $206\text{Pb}/238\text{U}$  and  $207\text{Pb}/206\text{Pb}$  fractionation factors ( $\sigma = \text{lower\_intercept\_age} * \sqrt{\text{relative\_error\_lower\_intercept\_age}^2 + \text{relative\_error\_206Pb/238U\_fractionation\_factor}^2 + \text{relative\_error\_207Pb/206Pb\_fractionation\_factor}^2}$ ).

- **Age-Upper Intercept (Ma)** (column AO)

See comments for column AM above. The upper intercept age is the highest of two ages consistent with points of intersection between the discordia and the concordia. If the discordia fails to intersect the concordia twice, the upper intercept age does not exist and a value of ‘undefined’ appears here. If the upper intercept age is  $>4560$  Ma and zero upper intercepts  $<4560$  Ma are encountered during the Monte Carlo simulation of the discordia, the upper intercept age does not exist and a value of ‘undefined’ appears here. If the upper intercept age is  $>4560$  Ma and at least one upper intercept  $<4560$  Ma is encountered during the Monte Carlo simulation, the upper intercept age does exist and a value of ‘4560’ appears here.

- **Age-Upper Intercept ( $\pm 2\sigma$ )** (column AP)

See comments for column AM above. The errors for the upper and lower intercepts of the discordia with the concordia are calculated by Monte Carlo simulation of the discordia using 1000 randomly generated lines based on the regression-derived slope and intercept values and their respective errors. If a value other than ‘undefined’ appears in column AO, the standard deviation of the Monte Carlo-derived upper intercepts is calculated about the upper intercept age, ignoring any intercepts  $<0$  Ma or  $>4560$  Ma. The  $\pm 2\sigma$  error on the upper intercept age is calculated based on the relative errors of the upper intercept age and the  $206\text{Pb}/238\text{U}$  and  $207\text{Pb}/206\text{Pb}$  fractionation factors ( $\sigma = \text{upper\_intercept\_age} * \sqrt{\text{relative\_error\_upper\_intercept\_age}^2 + \text{relative\_error\_206Pb/238U\_fractionation\_factor}^2 + \text{relative\_error\_207Pb/206Pb\_fractionation\_factor}^2}$ ).

- **Regression Intercept** (column AQ)

This value is the y-axis intercept of the best-fit line through all ( $207\text{Pb}/235\text{Uc}$  abscissa,  $206\text{Pb}/238\text{U}$  ordinate) points for all scans, calculated using routine LFIT in Press et al. (1992).

- **Regression Intercept ( $\pm 1\sigma$ )** (column AR)

This value is calculated using LFIT in Press et al. (1992).

- **Regression Slope** (column AS)

This value is the slope of the best-fit line through all ( $207\text{Pb}/235\text{Uc}$  abscissa,  $206\text{Pb}/238\text{U}$  ordinate) points for all scans, calculated using routine LFIT in Press et al. (1992).

- **Regression Slope ( $\pm 1\sigma$ )** (column AT)

This value is calculated using LFIT in Press et al. (1992).

## Analysis Details

- **% Minimum Discordance** (column AV)

Discordance here is defined as  $100 * (207\text{Pb}/206\text{Pb}_\text{age} - 206\text{Pb}/238\text{Pb}_\text{age}) / 207\text{Pb}/206\text{Pb}_\text{age}$  (%). If either  $207\text{Pb}/206\text{Pb}_\text{age}$  or  $206\text{Pb}/238\text{Pb}_\text{age}$  is zero, this discordance value is set to -200.00%. The minimum value of this discordance for all scans is reported here. Any value <-999.99% is arbitrarily set to -999.99%. If the value reported here is -200.00%, then at least one scan yielded either a  $207\text{Pb}/206\text{Pb}_\text{age}$  or a  $206\text{Pb}/238\text{Pb}_\text{age}$  value equal to zero and all other discordance values for the scans were >-200.00%.

- **% Maximum Discordance** (column AW)

The maximum value of this discordance for all scans is reported here. If the value reported here is -200.00%, then at least one scan yielded either a  $207\text{Pb}/206\text{Pb}_\text{age}$  or a  $206\text{Pb}/238\text{Pb}_\text{age}$  value equal to zero and all other discordance values for the scans were <-200.00%.

- **Preferred Scan #** (column AX)

The scan number that yielded the discordance value closest to zero.

- **Discordia Scans** (column AY)

The number of scans used to calculate the discordia.

- **$2\sigma$  Conc. Scans** (column AZ)

This value indicates the number of concordant scans for the spot. This is the same as columns AB and AK (column AK is sorted with other data).

- **Relative  $\alpha$ -Radiation** (column BA)

For each spot, this number is relative to the median  $\alpha$ -radiation value for all primary age standard spots. It represents the sum of the U and Th signal values for a spot, integrated over the preferred age of the spot, using the decay constants for various isotopes. Information regarding its calculation and usage is provided in **Appendix 2**.

- **Analysis\_#** (column BB)

Unlike column A, no extension is added to analysis number to indicate zircon.

### 2.3. Worksheet: Calculate (Figure 5)

**Calculate** requires the following operations be performed so that the various features work properly:

- **Step 1:** Enable macros.
- **Step 2:** Set ‘xmax\_plot’ (cell H5), ‘xbinwidth\_plot’ (cell H6), and ‘xstep\_plot’ (cell H7). Copy H5:H7 and paste to I5:I7. These parameters have the following meaning: xmax\_plot = maximum plot x-axis value in Ma (see **Plots**); xbinwidth\_plot = histogram bin width for distribution of grain ages into bins; xstep\_plot =  $\Delta x$  value for probability distribution function points (xmax\_plot/xstep\_plot must be  $\leq 5000$ ).
- **Step 3:** Press ‘transfer data’ button. This operation invokes several Visual Basic routines that read data into arrays and performs the following preliminary calculations:
  - the grain ages in column A are distributed into histogram bins (histogram x and y values are in columns N and O, respectively),

- the grain ages and their  $2\sigma$  errors in columns A and B, respectively, are converted to individual Gaussian distributions and added together to form the raw data probability distribution function (probability distribution function x and y values are in columns P and Q, respectively),
- peaks and troughs are determined for the raw data probability distribution function and following parameters related to the Gaussian deconvolution are set:
  - **ngauss:** Cell H14 is the number of Gaussians into which **Calculate** prefers to deconvolve the probability distribution function. The maximum number is 20. The minimum number is 3. For a counted number of probability distribution function peaks between 1 and 16, the peaks are ranked in order of decreasing area and the largest two are split into 3 Gaussians of area equal to 1/3 of their parent Gaussian (by dividing the amplitude by 3) and positions  $x=\text{mean}-1\sigma$ ,  $x=\text{mean}$ ,  $x=\text{mean}+1\sigma$ . For a counted number of probability distribution function peaks  $>16$ , only the largest 16 are used. Means are specified as the peak x-position (in Ma); standard deviations are specified as equal to  $\text{area}/(\text{amplitude}^*1.772415)$ ; areas are then normalized to the area of the raw data probability distribution function.
  - **gauss:** Cells G22:G41
  - **amplitude:** Cells H22:H41
  - **mean:** Cells I22:I41
  - **std dev:** Cells J22:J41
  - **data fraction:** Cells K22:K41
- These parameters represent the initial guesses for the Gaussians after pressing ‘transfer data’.
- d) all of the grains are combined into a single, preferred population (data in *red italics*) and the following data are calculated for the preferred population:
  - **# ages:** Cell H2 contains the number of ages total; cell I2 contains the number of ages in the red preferred population.
  - **maximum row:** Cell J11 contains the maximum row for the age to be included in the preferred population. The ages in column A are ranked from youngest to oldest (and below the grains with acceptable ages are the rejected grains with the following codes: F C Pb U M; \_ \_ Pb \_ \_ indicates rejection for failure to pass the quality test for Pb, and so on). Upon pressing ‘transfer data’, maximum row is set to the row of the oldest, acceptable grain and all grains are included in the population.
  - **wmean:** Cell L6 contains the weighted mean age of the grains in preferred population.
  - **$2\sigma+\text{FFerr}$ :** Cell L8 contains the  $2\sigma$  error of the weighted mean age of the grains in the preferred population; this error also includes the fractionation factor error.

- **$2\sigma$** : Cell L10 contains the  $2\sigma$  error of the weighted mean age of the grains in the preferred population; this error does not include the fractionation factor error.
- **MSWDw**: Cell L12 contains the weighted mean square of the weighted deviates of the grain ages in the preferred population.
- **mean**: Cell M6 contains the mean age of the grains in preferred population.
- **$2\sigma$  stddev**: Cell M8 contains the 2 standard deviation error of the mean age of the grains in the preferred population.
- **$2\sigma$  stderr**: Cell M10 contains the 2 standard error of the mean age of the grains in the preferred population.
- **P(MSWDw)**: Cell M12 contains MSWDw probability (probability of a greater MSWDw value) of the grain ages in the preferred population.
- **start\_row**: Cell L14 contains the row with lowest age in the preferred population.
- **end\_row**: Cell M14 contains the row with highest age in the preferred population.
- **# 1 $\sigma$  Absolute Errors**: Cell L16 contains the number of  $1\sigma$  absolute errors used for overlap to determine concordance between different grains ages within the preferred population. The grain age errors in column B are  $2\sigma$ . This parameter is very important when using button **maximum row** (see below).
- **Uppm**: Cell M22 contains the mean Uppm value (**Data** column I) for the grains in the preferred population.
- **Thppm**: Cell M23 contains the mean Thppm value (**Data** column J) for the grains in the preferred population.
- **U/Th**: Cell M24 contains the mean U/Th value (**Data** column K) for the grains in the preferred population.
- **alpha**: Cell M25 contains the mean relative  $\alpha$ -radiation damage value (**Data** column BA) for the grains in the preferred population.
- **Step 4**: Press ‘rescale plots’ button. This operation rescales the plots in **Plots** to the specified xmax\_plot value.

**Calculate** is now ready to work. The grain age columns are described below:

- **Preferred Age (Ma)** (column A)  
From **Data** column AE.
- **Absolute Error ( $\pm 2\sigma$ )** (column B)  
From **Data** column AF.
- **Age Type** (column C)  
From **Data** column AG.
- **Analysis #** (column D)  
From **Data** column AD.
- **Product of Qualities** (column E)

Product of the values from columns **Data AH, AI, AJ**.

Suppose one desires the age of the largest population for which all grains are concordant, overlapping by the # of  $1\sigma$  absolute errors set in cell L16. Simply ensure that the maximum row parameter in cell J11 indicates the row number of the oldest grain and press the button ‘maximum row’. All of the preferred population parameters (in *red italics*) will update.

Suppose one desires the age of the youngest population containing 2 or more grains. Set the maximum row in cell J11 to 5 and press button ‘maximum row’. If the grain in row 5 is NOT part of the population in *red italics* but grains in rows 2-4 are, then this is the youngest population. If this fails, try another value of maximum row in cell J11 and press button ‘maximum row’. Repeat until the data of interest appear for the preferred population.

Suppose one desires the age of the population for grains in rows 11-17, regardless of their ages (whether or not they are concordant) and regardless of the ages for grains in rows 2-10 and 18+. Change parameter start\_row in cell L14 to 11 and parameter end\_row in cell M14 to 17 and press button ‘statistics’. The population in *red italics* will update to reflect on these grains in this row interval.

Finally, press button ‘deconvolve’ to deconvolve the probability distribution function into the number of Gaussians indicated by parameter ngauss in cell H14. Cells H11, H12, I12, H16 (current mean  $\chi^2$  per nodal point deconvolved), H17 (best mean  $\chi^2$  per nodal point deconvolved), and G22:K41 will update as the deconvolution progresses and finally finishes. Parameters dym\_factor in cell H15 and chisqtol in cell H18 may be user set but this is not recommended. The significance of these parameters pertains to the chi-squared minimization process employed during the deconvolution and the may be ascertained directly by viewing the source code and searching for ‘dymfactor’ and ‘chisqtol’.

The curious user can investigate the meaning of the remaining columns and cells in this worksheet.

## 2.4. Worksheet: Plots (Figures 6a and 6b)

The plots are self-explanatory:

- **U-Pb Age (Ma) vs. Relative Age Frequency**

U-Pb age on x-axis vs. raw data probability distribution function and histogram bins on y-axis.

- **U-Pb Age (Ma) vs. Relative Age Frequency/U/Th**

U-Pb age on x-axis vs. raw data probability distribution function and U/Th on y-axis.

- **U-Pb Age (Ma) vs. Relative Age Frequency**

U-Pb age on x-axis vs. raw data probability distribution function and Gaussian deconvolution probability distribution function on y-axis.

- **U-Pb Age (Ma) vs. U/Th**

U-Pb age on x-axis vs. U/Th on y-axis.

- **U-Pb Age (Ma) vs. Frequency**

Gaussian deconvolution peak mean x-axis vs. Gaussian deconvolution peak data fraction on y-axis.

- **U-Pb Age (Ma) vs. U ppm**

U-Pb age on x-axis vs. U on y-axis.

- **Upper-Age Intercept (Ma) vs. Lower-Age Intercept (Ma)**

U-Pb upper-age intercept on x-axis vs. U-Pb lower-age intercept on y-axis.

- **U-Pb Age (Ma) vs. Th ppm**

U-Pb age on x-axis vs. Th on y-axis.

## 2.5. Worksheet: Standard\_Statistics (Figure 7)

This sheet contains information pertaining to the primary age standard FC-1 (indicated by age standard index 6 in UPbICP code; cell B2 here; age standard name F in UPbICP code; cell D2 here) of accepted age 1099 Ma (cell B3) and the secondary age standard TR run in pair along with the primary age standard (indicated by age standard index 2 in UPbICP code; cell B1 here; age standard name T in UPbICP code; cell D11 here) of accepted age 61.23 Ma (cell B12). Cells B5:B9 display mean preferred U-Pb ages for the primary age standard FC-1, calculated when FC-1 is treated as an unknown. Cells C5:C9 present  $1\sigma$  relative errors on the ages in cells B5:B9; these relative errors are used as the relative errors of the various isotopic ratio fractionation factors for the session.

During a first-pass run of UPbICP, the weighted mean age for the secondary age standard is given as the uncorrected age in cell B13 (uncorrected meaning that the fractionation factors were not corrected for relative  $\alpha$ -radiation damage). Parameter measAgeStdPreferred2 in **Appendix 2** subroutine AlphaEnergyFF is the accepted age of this secondary age standard in cell B12. Parameter std207235Age(nAgeStdPreferred2) (and for the other isotopic ratios) in **Appendix 2** subroutine AlphaEnergyFF is the uncorrected age of this secondary age standard in cell B13.

Cell B17 gives the median 91Zr/28Si (neither background corrected) value for all primary age standard spots and cell B18 gives the  $1\sigma$  standard deviation about this median ratio. All spots during the session within  $3\sigma$  of this median value are deemed zircon and the extension ‘\_Zrn’ (in cell B15) is added to the Analysis\_# name in **Data** column A. The values in cells C17:D20 represent statistics calculated for non-background corrected counts for the primary age standard.

## 2.6. Worksheet: A2Z\_Standards (Figure 8)

These columns are self-explanatory and provided for information purposes only. This is a compilation of U-Pb zircon ages (column C) and their  $2\sigma$  errors (column D) for all secondary age standards in the session from which the data in worksheet **Data** were derived. A value near zero for cell I2 and a value near one for cell K2 is desired.

## 2.7. Worksheet: Comment (Figure 9)

This sheet is required to provide a necessary comment in the event calculations are attempted in **Calculate** prior to pressing the ‘transfer data’ button.

## **2.8. Worksheet: Results\_Settings (Figure 10)**

These data are described above and they are simply combined here, minus all of the other data, for easy viewing. Click on each value to see the source of the value in the status bar.

## **3. Workbook: UPbICPViewGrain\_**

### **3.1. General**

This workbook is provided as a tool to permit the user to quickly view the raw data, sum of Gaussian equation fits, and discordia for individual spots analyzed. The workbook is composed of 5 worksheets:

- **Worksheet: Raw\_Data** allows the raw data from the ICP-MS system to be viewed, along with data pertaining to the background values and fitted Gaussians for each isotope.
- **Worksheet: Raw\_Data\_Plots** shows plots of the raw data.
- **Worksheet: bkg\_Corrected\_Data\_Plots** shows plots of the background corrected data and several relevant background corrected isotopic ratio plots.
- **Worksheet: Discordia** shows age and discordance versus scan number plots as well as discordia plots on the concordia diagram.
- **Worksheet: Standard\_Statistics** presents data from the primary and secondary age standards used to calculate ages.

### **3.2. Worksheet: Raw\_Data (Figure 11)**

Note that it is important to exit active cells containing data being set for the program to operate properly. Prior to pressing any of the buttons described below, an empty cell should be selected as the active cell. The following steps must be taken in order for this program to work:

1. Select the **Raw\_Data** tab at the bottom of the workbook.
2. File location examples from A2Z session z031110:
  - Location of run details file (\*.csv):
    - Folder: c:\UPbICP\UPbZrSiZ031110\
    - File: UPbZrSiZ031110.csv
  - Location of ICP-MS data files (\*.txt):
    - Folder: c:\UPbICP\UPbZrSiZ031110\
    - Files: \*.\* where \* indicates sample root name and grain number (sample root name, cell U14, is case sensitive; cases can be verified in the run details file UPbZrSiZ031110.csv or by checking the names of the \*.txt files themselves)
  - Location of UPbICP output files (\*.txt):
    - Folder: c:\UPbICP\ UPbZrSiZ031110\
    - Files:

- UPbZrSiZ031110StdBestGrainAge.txt
  - UPbZrSiZ031110StdBkgFit.txt
  - UPbZrSiZ031110StdScanAge.txt
  - UPbZrSiZ031110StdSigDelete.txt
  - UPbZrSiZ031110StdSigFit.txt
  - UPbZrSiZ031110StdFF.txt (not used at the time of this writing)
  - UPbZrSiZ031110UnkBestGrainAge.txt
  - UPbZrSiZ031110UnkBkgFit.txt
  - UPbZrSiZ031110UnkScanAge.txt
  - UPbZrSiZ031110UnkSigDelete.txt
  - UPbZrSiZ031110UnkSigFit.txt
3. Set run details file (cell U1) and press the upper ‘refresh run details file’ button. Cells U2:U9 are updated by this operation. Data transfer is complete when cell U15 is made the active cell.
  4. Set U11 through U17 and U20 (the spin up/down button may not work well, if this happens, set U15 manually). **Cell U14 is case sensitive.** Only ‘Std’ or ‘std’ are valid for age standard; only ‘Unk’ or ‘unk’ are valid for unknown in cell U20. Cell U17 is the maximum grain number for which an ICP-MS data file is available for the root filename in cell U14 in the specified folder location. Press ‘refresh grain’ button. Five files are read during data transfer (progress reported in cells U24 and U25). Data transfer and the calculations are complete when cell Q20 on worksheet **Discordia** is made the active cell.

Brief descriptions of various cells and columns follow:

- Columns A-R are read directly from, or calculated based on, data contained in the \*.txt file for the spot of interest (17103b\_20.txt in cell U18 in **Figure 11**).
- Cells AA2:AI10 are updated by reading the data in \*BkgFit.txt pertaining to the spot of interest.
- Cells AA15:AI112 are updated by reading the data in \*SigFit.txt pertaining to the spot of interest.
- Cells W22:W31 represent the area/10000 of the deleted background-corrected, measured signal intensities. Cells X22:X31 represent the area/10000 of the background-corrected, measured signal intensities to which the sum of Gaussian equations is fitted. Cells Y22:Y31 represent the area/10000 of the sum of Gaussian equations fitted to the background corrected, measured signal intensities.
- Column pairs AM and AN, AO and AP...BC and BD contain background corrected signal data (value in AM91 equals bkg+signal value in C91 minus the bkg value in AH2; see the formula in cell AM91), and calculated signal data fitted values (value in AN91 is the value indicated by the sum of the Gaussians detailed in cells AG15:AI24 minus the bkg value in AH2), respectively.
- Columns BF and BG represent the calculated 207Pb/235Uc and 206Pb/238U ratios, respectively for the signal scan indicated by the discordia index value in column BH, based on the sum of Gaussian equations for the respective isotopes (discordia index 1 equals scan 90, the first bkg corrected signal scan).

- The number 500 in column BH indicates a scan that is not acceptable for calculating the discordia. These data come from \*ScanAge.txt.
- Columns BN, BO, and CG represent bkg corrected signal data for the  $^{207}\text{Pb}/^{235}\text{Uc}$ ,  $^{206}\text{Pb}/^{238}\text{U}$ , and  $^{207}\text{Pb}/^{206}\text{Pb}$  ratios for the scans shown in column BM and CF.
- Columns BP, BQ, and CH represent the fractionation factors for the  $^{207}\text{Pb}/^{235}\text{Uc}$ ,  $^{206}\text{Pb}/^{238}\text{U}$ , and  $^{207}\text{Pb}/^{206}\text{Pb}$  ages, respectively for the scans shown in columns BM and CF. These data come from \*ScanAge.txt. Note the  $^{207}\text{Pb}/^{206}\text{Pb}$  fractionation factors are all set to 1. This is a fundamental feature of UPbICP.
- The number 500 in column BI indicates a scan that is not either acceptable or not concordant. These data come from \*ScanAge.txt.
- Column BS, BT, and BU represent the ages indicated for the scan shown in column BM. These data come from \*ScanAge.txt.
- Column BV represents the discordance defined as  $100 \times (\text{207Pb/206Pb\_age} - \text{206Pb/238Pb\_age}) / \text{207Pb/206Pb\_age} (\%)$ . These data come from \*ScanAge.txt.
- Cells BX2:BY3 show the minima maxima (the latter set to  $1.5 \times$  the maxima of columns BN and BO) of the measured data for plotting the concordia diagram on worksheet **Discordia**.

### 3.3. Worksheet: Raw\_Data\_Plots (Figure 12)

1. Select the **Raw\_Data\_Plots** tab at the bottom of the workbook.
2. The plots are self-explanatory.

### 3.4. Worksheet: bkg\_Corrected\_Data\_Plots (Figure 13a and Figure 13b)

1. Select the **bkg\_Corrected\_Data\_Plots** tab at the bottom of the workbook.
2. The plots are self-explanatory.

### 3.5. Worksheet: Discordia (Figure 14)

1. Select the **Discordia** tab at the bottom of the workbook.
2. The following plots are self-explanatory.

- **207Pb/235Uc vs. 206Pb/238U discordia (small scale)**  
 $^{207}\text{Pb}/^{235}\text{Uc}$  on x-axis vs.  $^{206}\text{Pb}/^{238}\text{U}$  on y-axis.
- **207Pb/235Uc vs. 206Pb/238U discordia (large scale)**  
 $^{207}\text{Pb}/^{235}\text{Uc}$  on x-axis vs.  $^{206}\text{Pb}/^{238}\text{U}$  on y-axis.
- **Scan vs. U-Pb Age (Ma)**  
Scan number on x-axis vs.  $^{207}\text{Pb}/^{235}\text{Uc}$ ,  $^{206}\text{Pb}/^{238}\text{U}$ , and  $^{207}\text{Pb}/^{206}\text{Pb}$  U-Pb ages on y-axis.
- **Scan vs. Discordance (%)**  
Scan number on x-axis vs.  $^{206}\text{Pb}/^{238}\text{U}$  vs.  $^{207}\text{Pb}/^{206}\text{Pb}$  discordance on y-axis.
- **Scan vs. U/Th**

Scan number on x-axis vs. U/Th on y-axis.

- **Scan vs. U ppm**

Scan number on x-axis vs. U ppm on y-axis.

- **Scan vs. Th ppm**

Scan number on x-axis vs. Th ppm on y-axis.

The ‘refresh’ button does very little at this point. Ultimately, it will calculation of the weighted mean concordant age for a sub-population of user-set scans (user will set Q20 and Q21 and the ‘refresh’ button will perform calculations to update all fields in *red italics*.

### **3.6. Worksheet: Standard\_Statistics (Figure 15)**

See the discussion in Section 2.5 above.

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## **Appendix 1: Common Pb Correction**

202Hg and the combination of 204Hg+204Pb are measured for purposes of common Pb correction. In the analysis below, no fractionation is assumed for the various isotopes; as such, the current A2Z preference is to avoid applying this correction until the fractionation of Hg and Pb isotopes in the apparatus used is better understood. The isotopic abundances of the Hg isotopes measured are (Friedlander et al., 1981):

$$202\text{Hg fraction} = 0.298$$

$$204\text{Hg fraction} = 0.069$$

Common Pb isotopic ratios are (Chen and Wasserburg, 1983):

$$206\text{Pb}/204\text{Pb}_{\text{common}} = 9.3066$$

$$207\text{Pb}/204\text{Pb}_{\text{common}} = 10.293$$

$$208\text{Pb}/204\text{Pb}_{\text{common}} = 29.475$$

Common 204Pb is calculated as follows:

$$204\text{Pb}_{\text{common}} = (204\text{Hg}204\text{Pb}) - [(204\text{Hg fraction}/202\text{Hg fraction}) * 202\text{Hg}] \text{ (if } <0 \text{ for a scan then set to 0)}$$

Common Pb corrected isotopic ratios are calculated as follows:

$$206\text{Pb} = 206\text{Pb\_background\_corrected} - (204\text{Pb}_{\text{common}} * 206\text{Pb}/204\text{Pb}_{\text{common}}) \text{ (if } <0 \text{ for a scan then set to 0)}$$

$$207\text{Pb} = 207\text{Pb\_background\_corrected} - (204\text{Pb}_{\text{common}} * 207\text{Pb}/204\text{Pb}_{\text{common}}) \text{ (if } <0 \text{ for a scan then set to 0)}$$

$$208\text{Pb} = 208\text{Pb\_background\_corrected} - (204\text{Pb}_{\text{common}} * 208\text{Pb}/204\text{Pb}_{\text{common}}) \text{ (if } <0 \text{ for a scan then set to 0)}$$

## Appendix 2: Primary Age Standard Fractionation Factor Correction for $\alpha$ -Radiation Damage

Function dAlphaEnergy (below) is used to calculate the relative  $\alpha$ -radiation damage. Th232, U235c, and U238 represent the signal intensities for these isotopes for the current scan. Parameters ageTh232, ageU235c, and ageU238 represent the respective UPb ages for the current scan. Values of dAlphaEnergy are summed for all scans for a spot.

```
Function dAlphaEnergy(U235c, U238, Th232, ageU235c, ageU238, ageTh232, ffage)
    'dim ageU235c, ageU238, ageTh232, ffage(5) implicit
    'U235c
    dAlphaEnergy = (7 * 5.94 * U235c / ffage(2)) * (Exp(lamdaU235 * ageU235c) - 1)
    'U238
    dAlphaEnergy = dAlphaEnergy + (8 * 5.36 * U238 / ffage(3)) * (Exp(lamdaU238 * ageU238) - 1)
    'Th232
    dAlphaEnergy = dAlphaEnergy + (6 * 5.83 * Th232 / ffage(5)) * (Exp(lamdaTh232 * ageTh232) - 1)
End Function
```

Corrections of the fractionation factors for the primary age standard are performed using the following subroutine. Several important parameters have the following meanings:

nAgeStdPreferred1=index for the primary age standard (FC-1 in most cases)  
nAgeStdPreferred2=index for the secondary age standard (TR in most cases)  
StdAlphaEnergy(nAgeStdPreferred1)=relative  $\alpha$ -radiation damage for the primary age standard  
StdAlphaEnergy(nAgeStdPreferred2)=relative  $\alpha$ -radiation damage for the secondary age standard  
SAlphaEnergy=average StdAlphaEnergy(nAgeStdPreferred1) per scan multiplied by the scan number i  
UAlphaEnergy=sum of dAlphaEnergy values (function dAlphaEnergy) for scans 1 through i  
std207235Age(nAgeStdPreferred2)=accepted 207Pb/235Uc age for the secondary age standard (same for std208238... and std207206...)  
measAgeStdPreferred2=measured age for the secondary age standard when calculated *without* fractionation factor correction

Several conditional statements limit the amount of fractionation factor corrections that are permitted. a) If UAlphaEnergy $\geq$ SAlphaEnergy then no correction is performed.  
b) No fractionation factor is permitted to be set less than the minimum value observed for the respective isotopic ratio for the primary age standard.

```
Sub AlphaEnergyFF(SAlphaEnergy, UAlphaEnergy, ffage, minff)
    'dim ffage(5),minff(5) implicit
    Dim xl, x2, y1, y21, y22, y2235, y2238, y2232, m, b
    If UAlphaEnergy < SAlphaEnergy Then
        'calibration for acceptable 207/206
        xl = 1 'FC-1/FC-1
        x2 = (StdAlphaEnergy(nAgeStdPreferred2) / StdAlphaEnergy(nAgeStdPreferred1)) 'IFCT/FC-1
        y1 = 1 'FC-1/FC-1
        '235Uc
        y2235 = std207235Age(nAgeStdPreferred2) / measAgeStdPreferred2
        m = (y2235 - y1) / (x2 - xl)
        b = y2235 - x2 * m
        ffage(2) = ffage(2) * (m * UAlphaEnergy / SAlphaEnergy + b)
        If ffage(2) < minff(2) Then
            ffage(2) = minff(2)
        End If
    '238U
```

```

y2238 = std206238Age(nAgeStdPreferred2) / measAgeStdPreferred2
m = (y2238 - y1) / (x2 - x1)
b = y2238 - x2 * m
ffage(3) = ffage(3) * (m * UAlphaEnergy / SAlphaEnergy + b)
If ffage(3) < minff(3) Then
    ffage(3) = minff(3)
End If
'232Th
y2232 = std208232Age(nAgeStdPreferred2) / measAgeStdPreferred2
m = (y2232 - y1) / (x2 - x1)
b = y2232 - x2 * m
ffage(5) = ffage(5) * (m * UAlphaEnergy / SAlphaEnergy + b)
If ffage(5) < minff(5) Then
    ffage(5) = minff(5)
End If
End If
End Sub

```

### Appendix 3: Functions for Calculating U-Pb Ages

The following functions are used by UPbICP to calculate U-Pb ages for their respective isotopic ratios (input parameter *ratio*).

```
Function Age207235(ratio)
    'dim ratio,Age207235 implicit
    If ratio < 0 Then
        Age207235 = 0
    Else
        Age207235 = (1 / lamdaU235) * Log(ratio + 1)
    End If
End Function

Function Age206238(ratio)
    'dim ratio,Age206238 implicit
    If ratio < 0 Then
        Age206238 = 0
    Else
        Age206238 = (1 / lamdaU238) * Log(ratio + 1)
    End If
End Function
```

For  $^{207}\text{Pb}/^{206}\text{Pb}$  ratios, U-Pb ages must be calculated by iteration. To avoid this, UPbICP reads an array *ratio207206\_data* indexed from  $i = 1$  to 45600 and containing  $^{207}\text{Pb}/^{206}\text{Pb}$  ratios corresponding to  $i \times 10^5$  years for each index  $i$ .

```
Function Age207206(ratio)
    'dim ratio,Age207206 implicit
    Dim i, m, b
    If ratio <= Pb207206init Or ratio > ratio207206_data(45600) Then
        Age207206 = 0
    Else
        For i = 1 To 45600
            If ratio < ratio207206_data(i) Then
                m = 0.1 / (ratio207206_data(i) - ratio207206_data(i - 1))
                b = CSng(i - 1) * 0.1 - ratio207206_data(i - 1) * m
                Age207206 = m * ratio + b
                Exit Function
            End If
        Next i
    End If
End Function
```

**Table 1.** ICP-MS and laser ablations system operating conditions and data acquisition parameters.

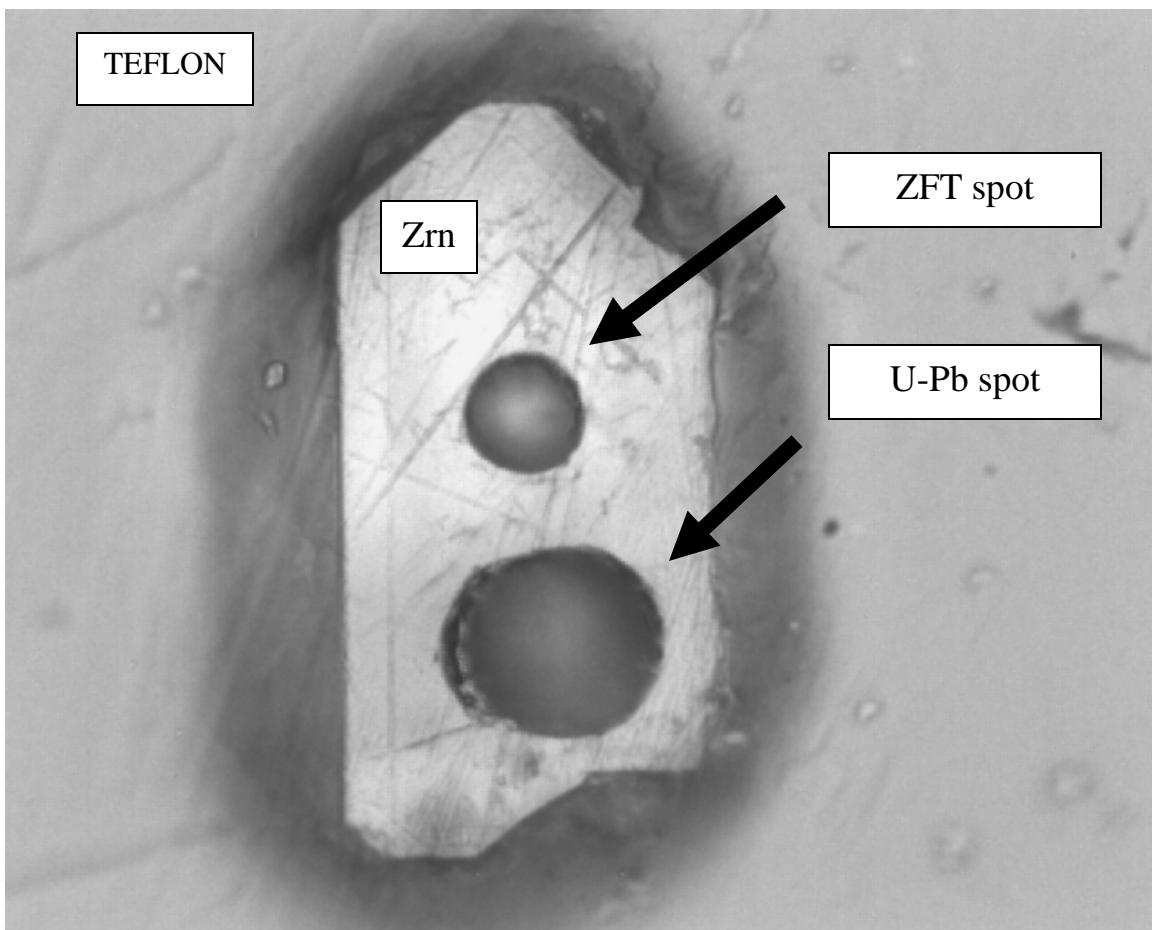
<i>ICP-MS: operating conditions</i>	
Instrument	Finnigan Element II Magnetic Sector ICP-MS
Forward power	1.25 kW
Reflected power	<5 W
Plasma gas	Ar
Coolant flow	15 l/min
Carrier flow	1.0 l/min (Ar) 0.8 l/min (He)
Auxiliary flow	1.0 l/min
<i>ICP-MS: data acquisition parameters</i>	
Dwell time	24 milliseconds per peak point
Points per peak	3
Mass window	5%
Scans	255
Data acquisition time	38.5 sec
Data acquisition mode	E scanning
Isotopes measured	<sup>202</sup> Hg, <sup>204</sup> Pb, <sup>206</sup> Pb, <sup>207</sup> Pb, <sup>208</sup> Pb, <sup>232</sup> Th, <sup>235</sup> U, <sup>238</sup> U (scans ~60-250) <sup>28</sup> Si, <sup>91</sup> Zr (scans 251-255)
<i>Laser ablation system: operating conditions</i>	
Laser type	New Wave Neodymium: YAG
Wavelength	213 nm
Laser mode	Q switched
Laser output power	10 J/cm <sup>2</sup>
Laser warm up time	6 sec
Shot repetition rate	5 Hz
Sampling scheme	spot (20 µm)

**Table 2.** Age standards used at A2Z.

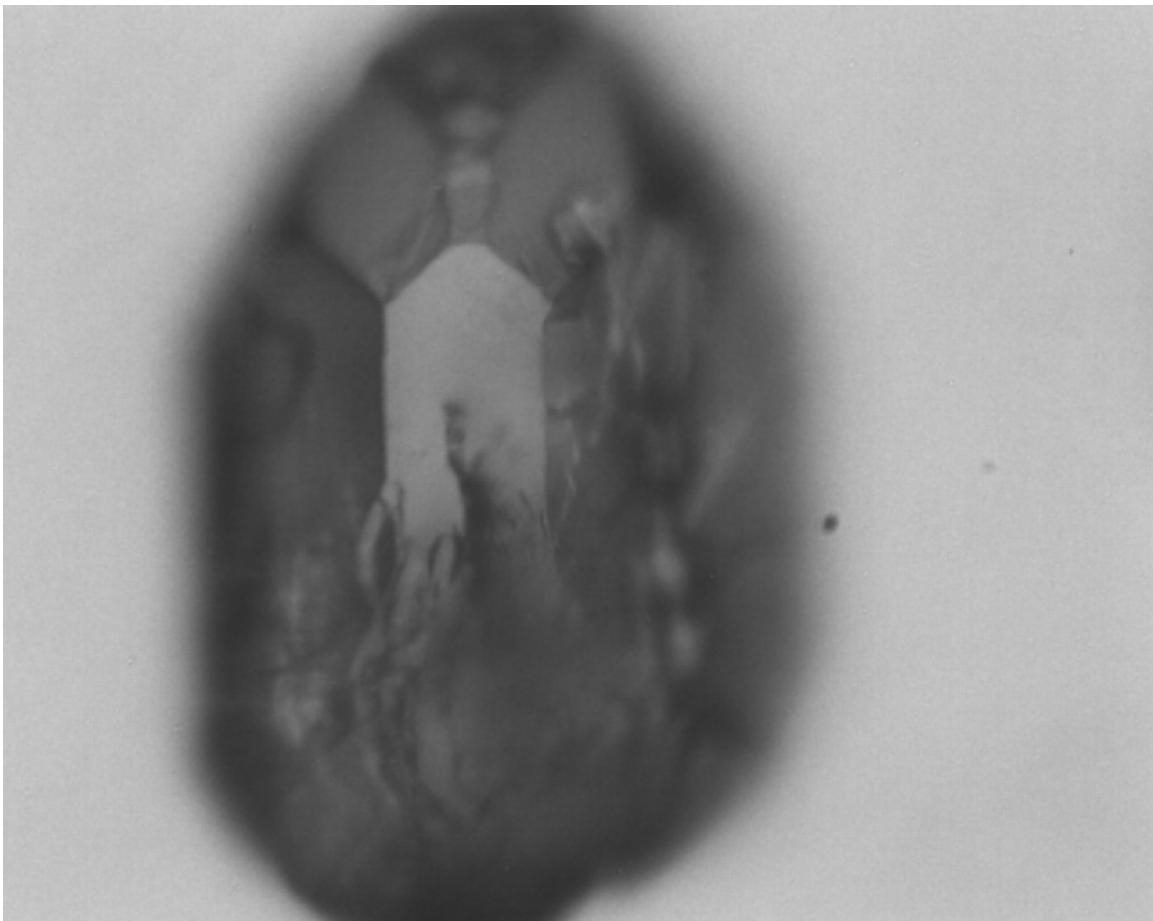
Standard	Standard	U-Pb age ( $\pm 2\sigma$ )	Reference
FC-1	Duluth complex	$1099.0 \pm 0.6$ Ma	Paces and Miller, 1993
FC-5z	Duluth complex	$1099.0 \pm 0.6$ Ma (assumed equal to FC-1)	Paces and Miller, 1993
TR	Tardree Rhyolite	$61.23 \pm 0.11$ Ma	Dave Chew, personal communication
IFCan	Fish Canyon Tuff	$28.201 \pm 0.012$ Ma	Kuiper et al., 2008
R33	Braintree complex	$418.9 \pm 0.4$ Ma	Black et al., 2004
TEM2	Temora 2, Middledale gabbroic diorite	$416.78 \pm 0.33$ Ma	Black et al., 2004
MD	Mount Dromedary	$99.12 \pm 0.14$ Ma	Renne et al., 1998
200-01	Tioga Bed K bentonite	$391.4 \pm 1.8$ Ma	Tucker et al., 1998

**Table 3.** Weighted mean U and Th ppm values assumed for FC-1 (Paces and Miller, 1993)

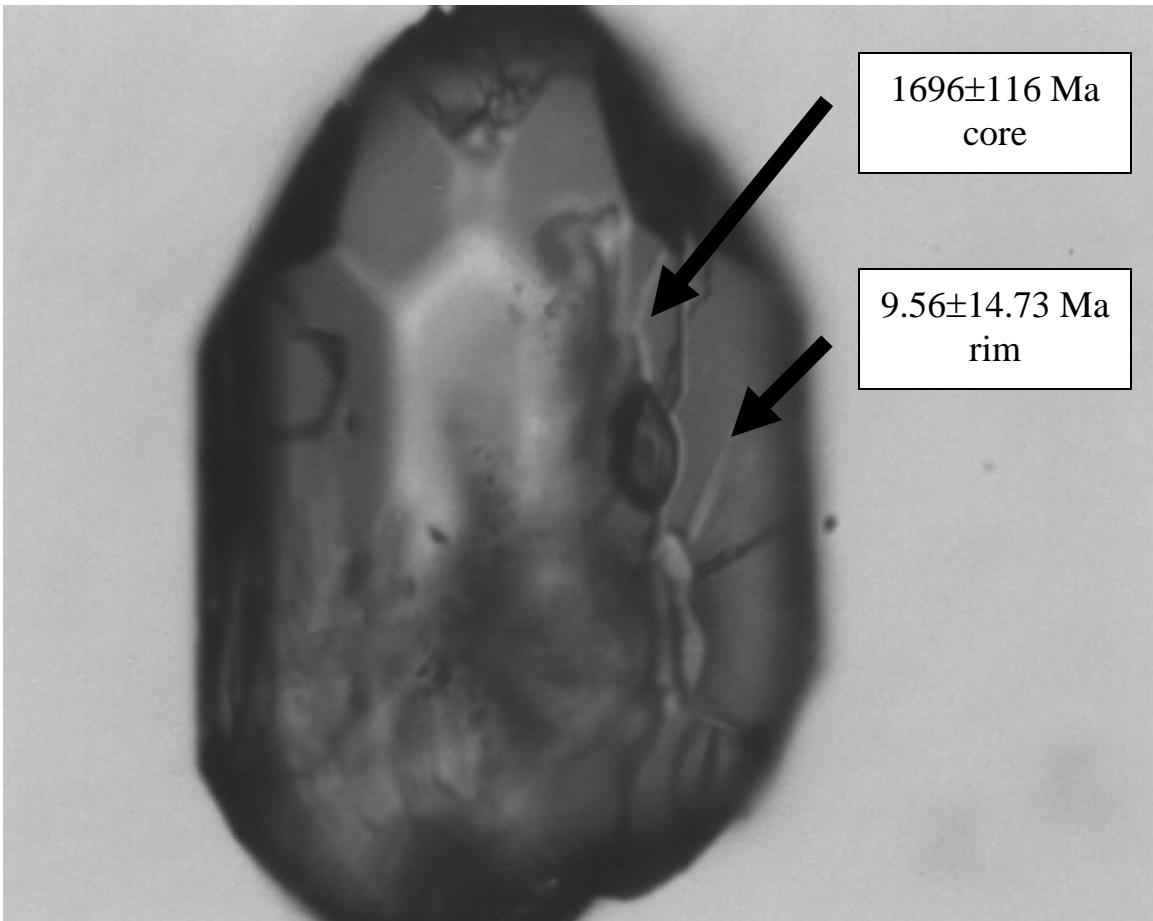
mg	Uppm	Thppm	mg*Uppm	mg*Thppm
125	420	258	52500	32250
120	1510	905	181200	108600
155	299	170	46345	26350
118	218	115	25724	13570
133	292	176	38836	23408
sum mg			mg*Uppm/sum mg	mg*Thppm/sum mg
651		weighted mean	529.35	313.64



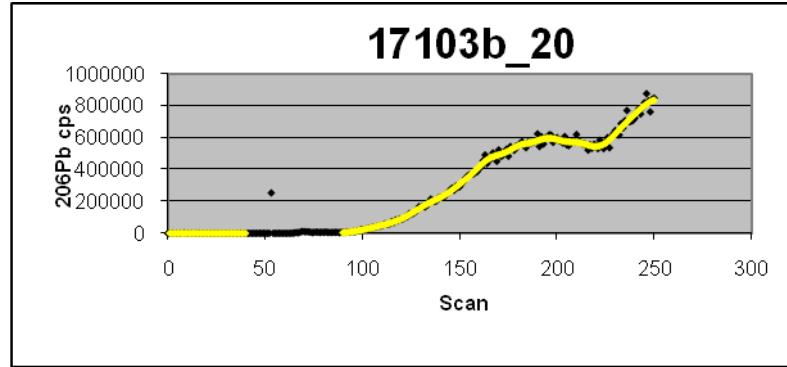
**Figure 1a.** Transmitted and reflected light image of spot 1713b\_20 at 625x magnification.  
The plane of focus is on the polished and etched zircon surface.



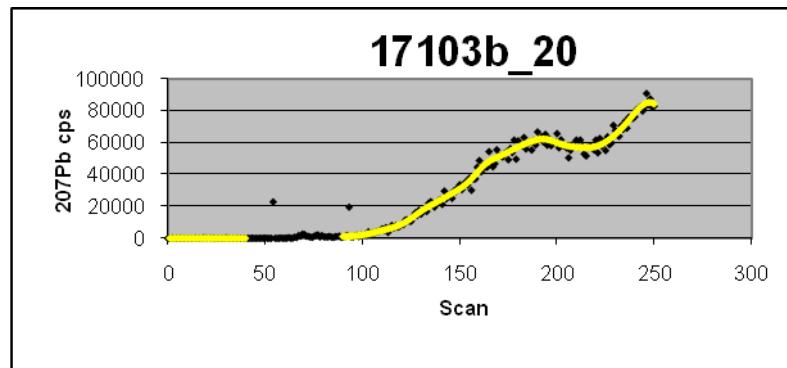
**Figure 1b.** Transmitted light image of spot 1713b\_20 at 625x magnification. The plane of focus is on the lower prismatic crystal surface of the zircon grain.



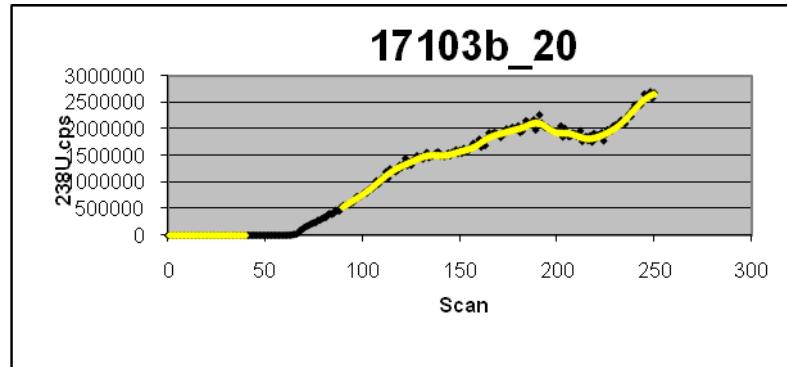
**Figure 1c.** Transmitted light image of spot 1713b\_20 at 625x magnification. The plane of focus is on the interior of the zircon grain and it shows the boundary between the old core and young rim.



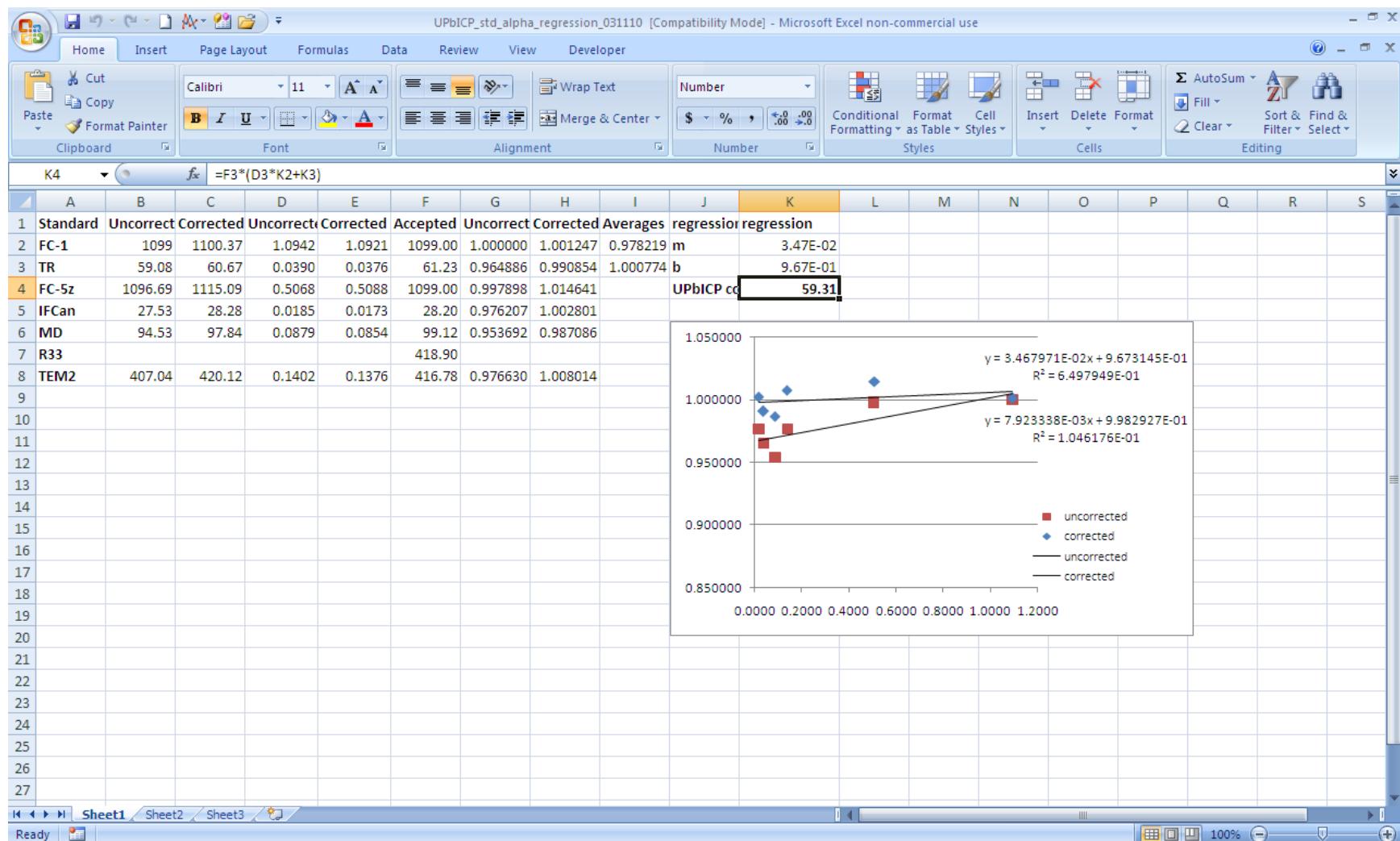
**Figure 2a.**  $206\text{Pb}$  data for spot 1713b\_20. Left-hand yellow curve represents the polynomial fitted to the background intensities. Right-hand yellow curve is represents the sum of Gaussian equations fitted to the signal intensities.



**Figure 2b.**  $207\text{Pb}$  data for spot 1713b\_20.



**Figure 2c.**  $238\text{U}$  data for spot 1713b\_20.



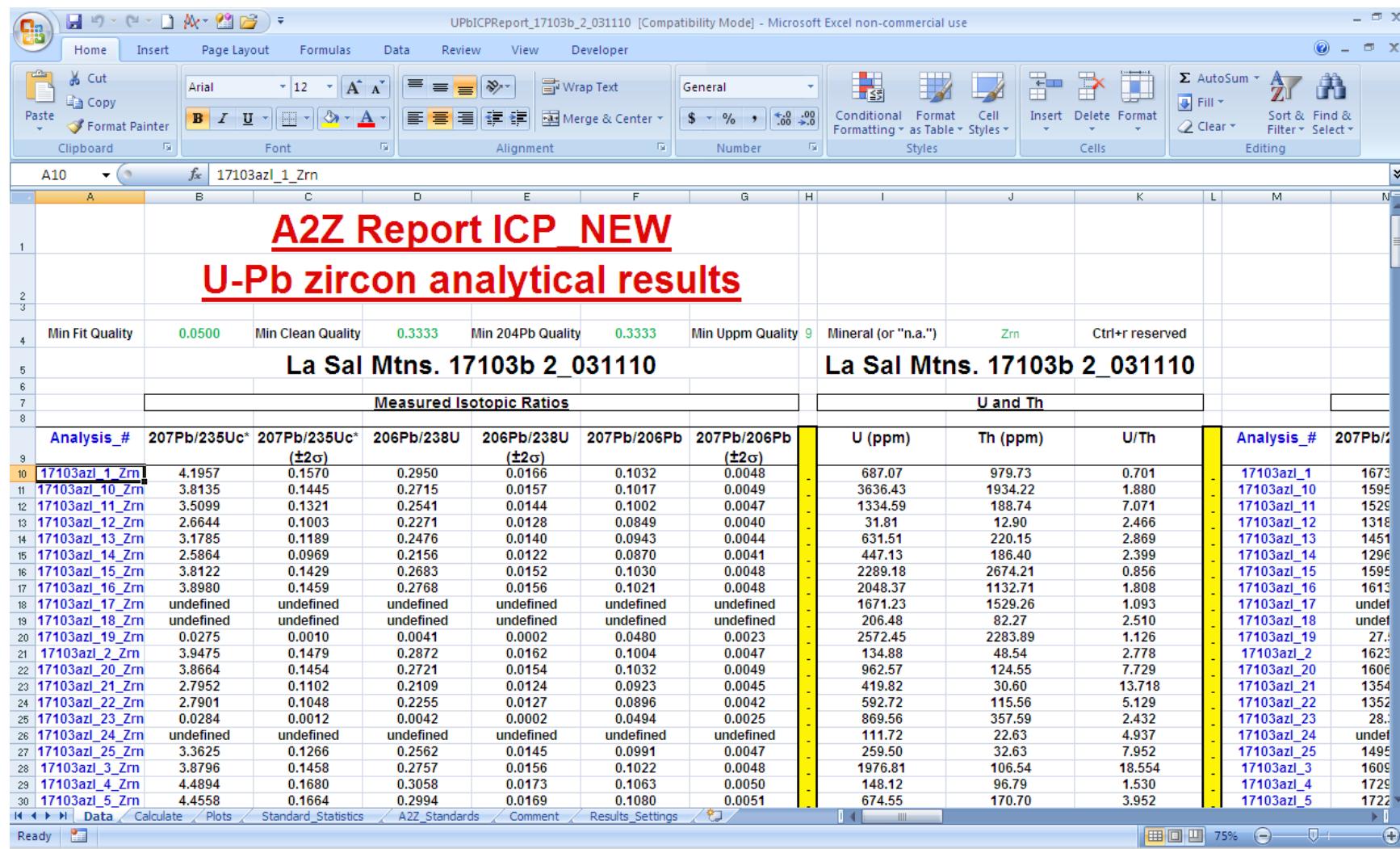


Figure 4. Worksheet: Data

UPbICPReport\_17103b\_2\_031110 [Compatibility Mode] - Microsoft Excel non-commercial use

The screenshot shows a Microsoft Excel spreadsheet titled "UPbICPReport\_17103b\_2\_031110 [Compatibility Mode] - Microsoft Excel non-commercial use". The ribbon is visible at the top with tabs for Home, Insert, Page Layout, Formulas, Data, Review, View, and Developer. The formula bar at the top says "not ready". The main area contains a table with data and formulas. The columns are labeled A through N. The rows are numbered 1 to 31. Some cells are highlighted in gray or red. The data includes various parameters and their values, such as "Preferred Age (Ma)", "Absolute Error ( $\pm 2\sigma$ )", "Age Type", "Analysis #", "Product of Qualities", and various workspace and progress-related variables. The status bar at the bottom shows "Ready" and "100%".

	A	B	C	D	E	F	G	H	I	J	K	L	M	N
1	Preferred Age (Ma)	Absolute Error ( $\pm 2\sigma$ )	Age Type	Analysis #	Product of Qualities		workspace	workspace	workspace	workspace	workspace	workspace	workspace	x hist age
2	21.27	1.90	lower int.	17103azs_45	0.8004		# ages	72	28	transfer data				0.00
3	21.74	1.24	Pb/U wmean conc.	17103azs_27	0.7766		xmin_data	18.42			rescale plots			0.00
4	23.38	1.33	Pb/U wmean conc.	17103azl_9	0.4314		xmax_data	2060.58	preferred					20.00
5	23.43	1.76	lower int.	17103azs_36	0.2769	set	xmax_plot	2000.00	2000.00		wmean	mean		20.00
6	23.71	1.82	lower int.	17103b_3	0.5676	set	xbinwidth	20.00	20.00		26.35	26.27		20.00
7	23.72	1.57	Pb/U wmean conc.	17103b_14	0.8145	set	xstep_plot	0.5	0.5		2σ+FFerr	2σ stddev		40.00
8	24.00	2.04	lower int.	17103azs_32	0.6877					Progress	1.27	2.65		40.00
9	24.25	2.34	lower int.	17103azs_41	0.5054				not ready	Maximum Row	2σ	2σ stderr		40.00
10	25.15	1.66	Pb/U wmean conc.	17103b_9	0.8431		area histog	1440.00			0.31	0.50		60.00
11	25.60	1.45	Pb/U wmean conc.	17103b_23	0.5022		area pdf	1440.01			MSWDw	P(MSWDw)		60.00
12	26.06	1.49	Pb/U wmean conc.	17103azs_49	0.8162		area gauss	1430.29	1430.26		2.12	0.004676		60.00
13	26.10	1.57	Pb/U wmean conc.	17103azs_43	0.5089		ngauss	12		maximum row	start_row	end_row		80.00
14	26.23	1.52	Pb/U wmean conc.	17103b_24	0.9797	set	dym_factor	2.25		set	5	32		80.00
15	26.32	2.06	lower int.	17103azs_35	0.5219		chisqm	0.0035316			# of 1σ Absolute Errors			80.00
16	26.42	1.68	Pb/U wmean conc.	17103b_8	0.9977		chisqbestm	0.0035316			3.00			100.00
17	26.44	1.51	Pb/U wmean conc.	17103b_2	0.8305	set	chisqtol	0.1000		deconvolve		statistics		100.00
18	26.44	1.52	Pb/U wmean conc.	17103b_5	0.6612									100.00
19	26.60	1.51	Pb/U wmean conc.	17103azl_19	0.6268									120.00
20	26.78	1.86	lower int.	17103b_15	0.7152									120.00
21	26.79	1.54	Pb/U wmean conc.	17103azl_23	0.5228		gauss	amplitude	mean	std dev	data fraction	mean		120.00
22	26.86	1.53	Pb/U wmean conc.	17103b_18	0.7283		1	27.132	21.590	0.837	2.81%	Uppm	631.77	140.00
23	26.92	1.53	Pb/U wmean conc.	17103azs_28	0.8270		2	64.663	23.589	0.830	6.65%	Thppm	467.29	140.00
24	27.00	1.54	Pb/U wmean conc.	17103azs_33	0.7205		3	30.524	25.489	1.506	5.70%	U/Th	1.975	140.00
25	27.01	1.54	Pb/U wmean conc.	17103azs_29	0.8514		4	176.130	26.810	1.114	24.32%	alpha	0.0254	160.00
26	27.15	1.56	Pb/U wmean conc.	17103azs_37	0.7874		5	42.509	29.318	2.251	11.86%	Q Filters		160.00
27	27.48	1.57	Pb/U wmean conc.	17103b_4	0.9975		6	7.091	44.150	1.591	1.40%	Fit	0.0500	160.00
28	27.54	1.57	Pb/U wmean conc.	17103b_6	0.6853		7	0.263	981.700	42.878	1.40%	Clean	0.3333	180.00
29	27.59	1.58	Pb/U wmean conc.	17103b_12	0.8529		8	1.226	1290.650	56.656	8.61%	204Pb	0.3333	180.00
30	27.63	1.62	Pb/U wmean conc.	17103b_13	0.5923		9	0.723	1465.360	99.559	8.92%	U ppm	9	180.00
31	27.82	1.60	Pb/U wmean conc.	17103b_25	0.3160		10	0.447	1615.032	30.051	1.66%	Mineral	Zrn	200.00

Figure 5. Worksheet: Calculate

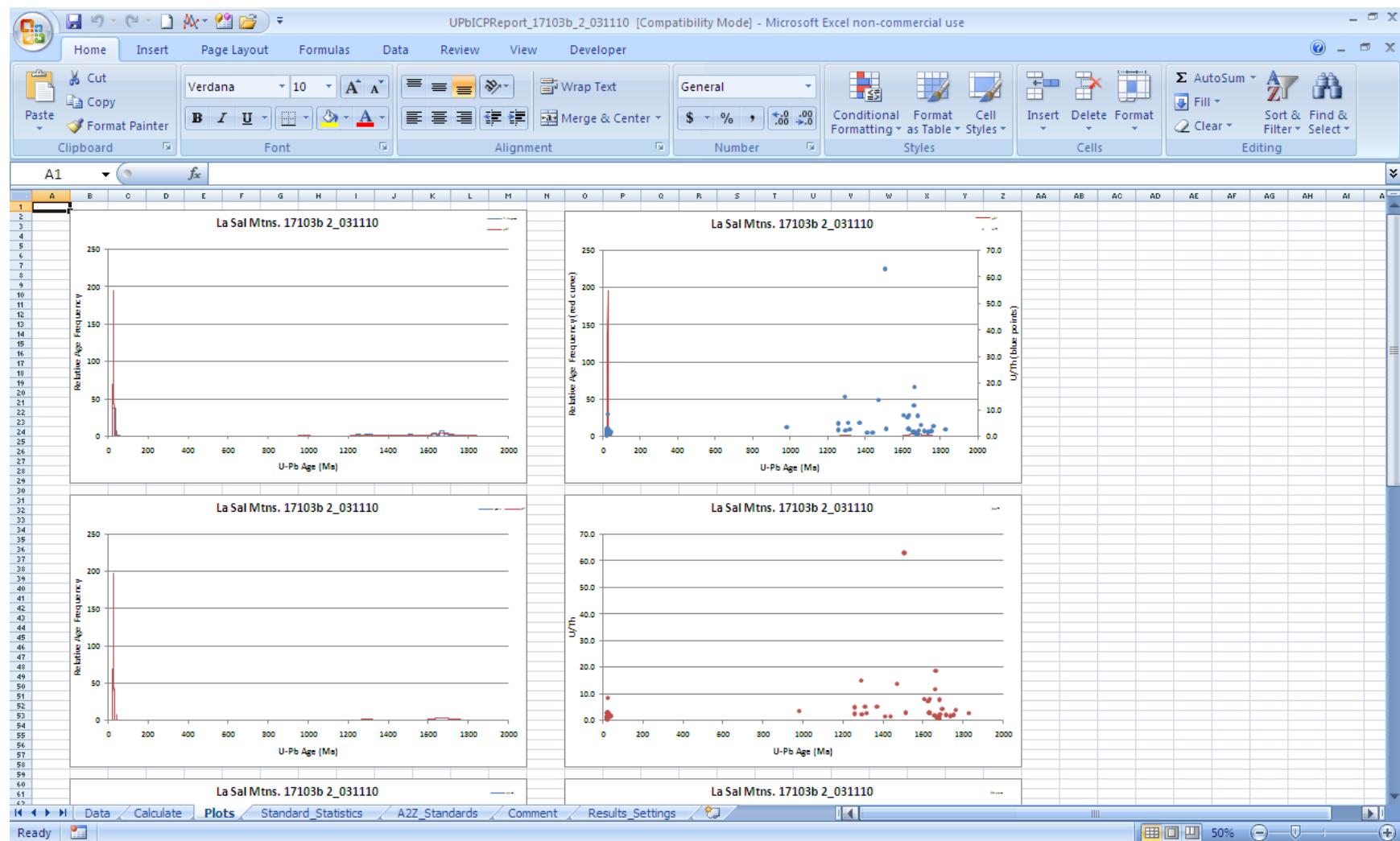


Figure 6a. Worksheet: Plots

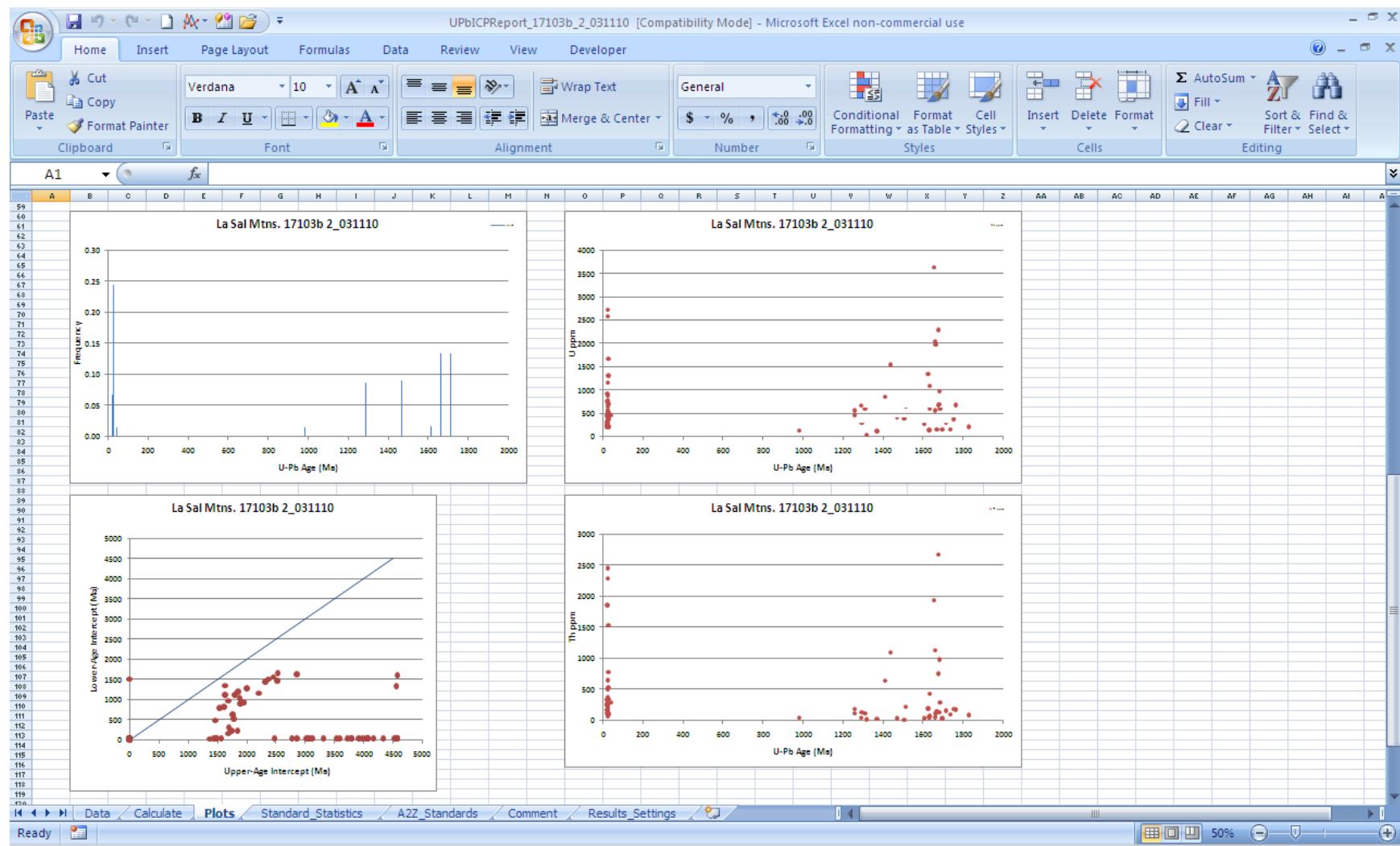


Figure 6b. Worksheet: Plots

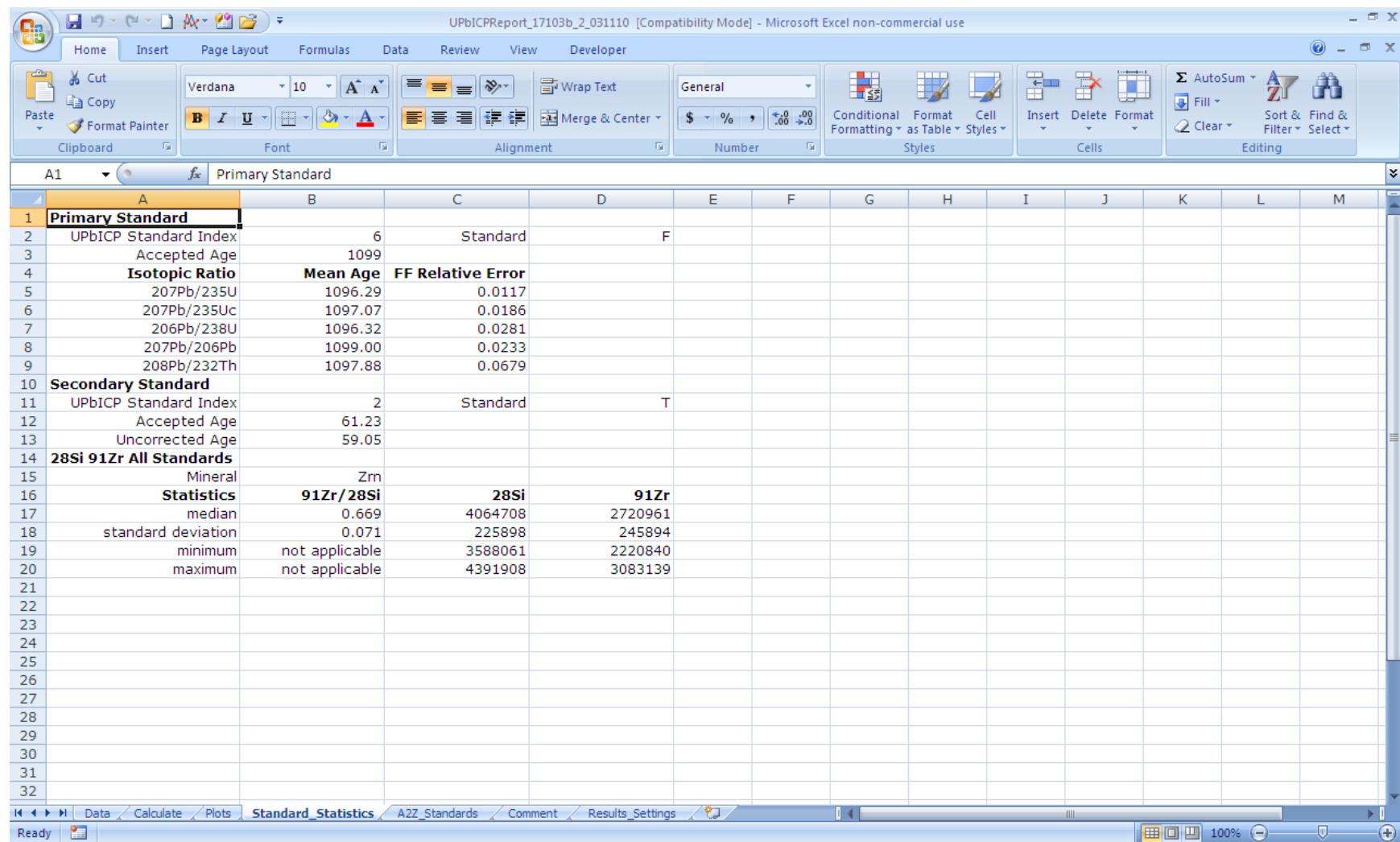
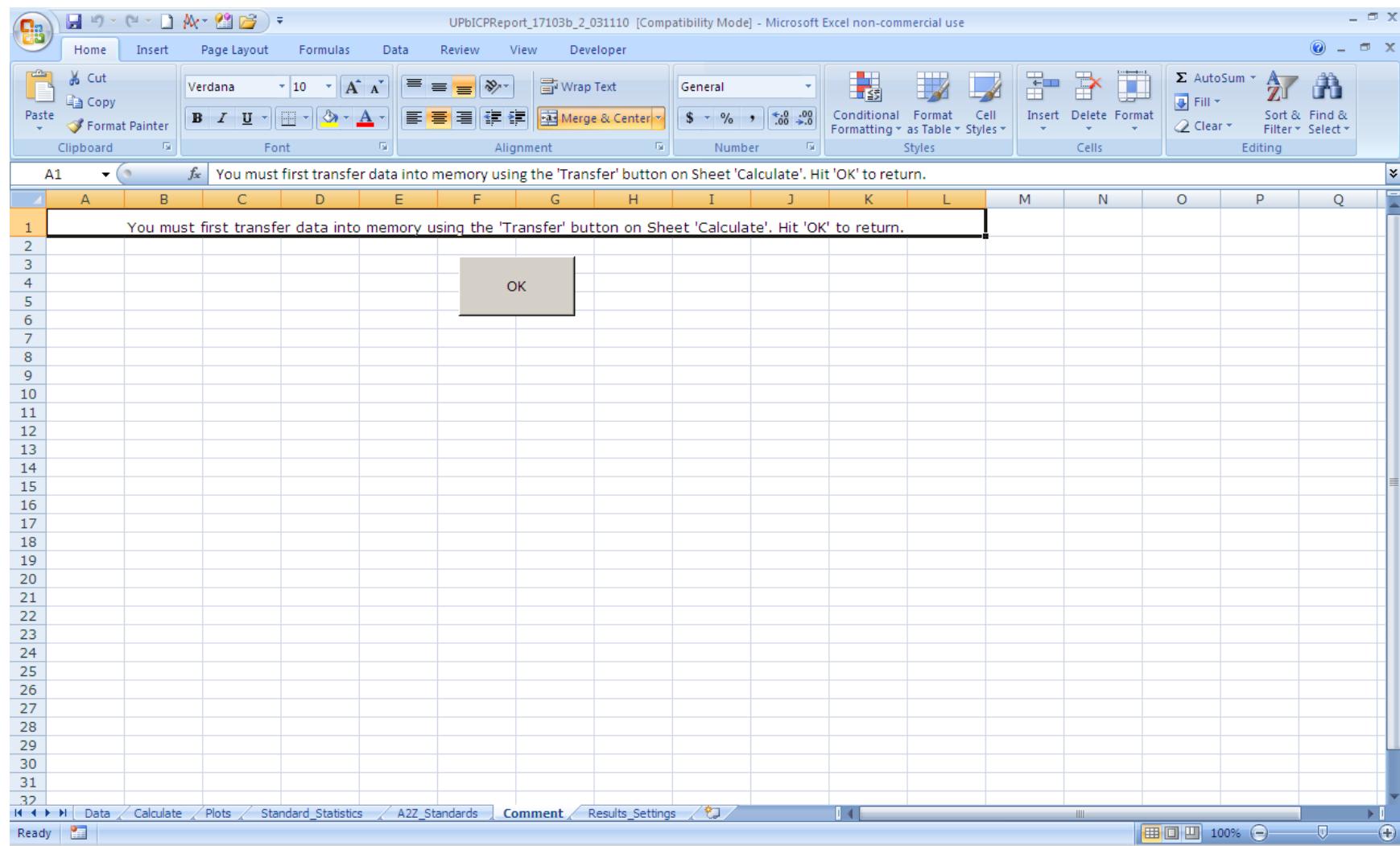


Figure 7. Worksheet: Standard\_Statistics

UPbICPReport\_17103b\_2\_031110 [Compatibility Mode] - Microsoft Excel non-commercial use

	A	B	C	D	E	F	G	H	I	J	K	L	M	N
1	Session	Grain	Preferred (Measured) Age (Ma)	Absolute Error (±2σ)	Age Type	Accepted Age (Ma)	Absolute Error (±2σ)	(Accepted - Preferred) / Accepted	Average (perfect=0)	Accepted / Measured	Average (perfect=1)			
2	z031110	TR_11	54.00	4.15	lower int.	61.23	0.11	0.1181	-0.0004	1.1339	1.0006			
3	z031110	TR_15	57.30	3.41	Pb/U wmean conc.	61.23	0.11	0.0641			1.0685			
4	z031110	TR_10	59.01	3.37	Pb/U wmean conc.	61.23	0.11	0.0362			1.0376			
5	z031110	TR_12	59.10	3.41	Pb/U wmean conc.	61.23	0.11	0.0347			1.0360			
6	z031110	TR_16	59.65	3.41	Pb/U wmean conc.	61.23	0.11	0.0259			1.0266			
7	z031110	TR_18	60.07	3.43	Pb/U wmean conc.	61.23	0.11	0.0190			1.0194			
8	z031110	TR_21	60.10	3.43	Pb/U wmean conc.	61.23	0.11	0.0185			1.0188			
9	z031110	TR_22	60.13	3.43	Pb/U wmean conc.	61.23	0.11	0.0180			1.0184			
10	z031110	TR_19	60.51	3.46	Pb/U wmean conc.	61.23	0.11	0.0117			1.0119			
11	z031110	TR_8	60.68	3.46	Pb/U wmean conc.	61.23	0.11	0.0090			1.0090			
12	z031110	TR_13	60.88	3.47	Pb/U wmean conc.	61.23	0.11	0.0057			1.0057			
13	z031110	TR_6	60.91	3.54	Pb/U wmean conc.	61.23	0.11	0.0053			1.0053			
14	z031110	TR_17	60.95	3.48	Pb/U wmean conc.	61.23	0.11	0.0046			1.0046			
15	z031110	TR_20	61.06	4.12	Pb/U wmean conc.	61.23	0.11	0.0028			1.0028			
16	z031110	TR_7	61.33	3.50	Pb/U wmean conc.	61.23	0.11	-0.0017			0.9983			
17	z031110	TR_3	62.09	3.61	Pb/U wmean conc.	61.23	0.11	-0.0140			0.9862			
18	z031110	TR_9	62.45	3.58	Pb/U wmean conc.	61.23	0.11	-0.0199			0.9805			
19	z031110	TR_1	62.50	4.49	lower int.	61.23	0.11	-0.0207			0.9797			
20	z031110	TR_5	63.25	3.63	Pb/U wmean conc.	61.23	0.11	-0.0330			0.9681			
21	z031110	TR_4	63.86	3.67	Pb/U wmean conc.	61.23	0.11	-0.0430			0.9588			
22	z031110	TR_14	64.72	3.69	Pb/U wmean conc.	61.23	0.11	-0.0570			0.9461			
23	z031110	FC5_8	1078.60	66.15	Pb/U wmean conc.	1099.00	0.60	0.0186			1.0189			
24	z031110	FC5_9	1087.72	66.69	Pb/U wmean conc.	1099.00	0.60	0.0103			1.0104			
25	z031110	FC5_6	1096.77	67.27	Pb/U wmean conc.	1099.00	0.60	0.0020			1.0020			
26	z031110	FC5_7	1105.31	67.84	Pb/U wmean conc.	1099.00	0.60	-0.0057			0.9943			
27	z031110	FC5_1	1129.00	69.40	Pb/U wmean conc.	1099.00	0.60	-0.0273			0.9734			
28	z031110	FC5_2	1132.16	69.61	Pb/U wmean conc.	1099.00	0.60	-0.0302			0.9707			
29	z031110	FC5_3	1132.50	69.66	Pb/U wmean conc.	1099.00	0.60	-0.0305			0.9704			
30	z031110	FC5_4	1137.66	69.98	Pb/U wmean conc.	1099.00	0.60	-0.0352			0.9660			

Figure 8. Worksheet: A2Z\_Standards



**Figure 9.** Worksheet: Comment

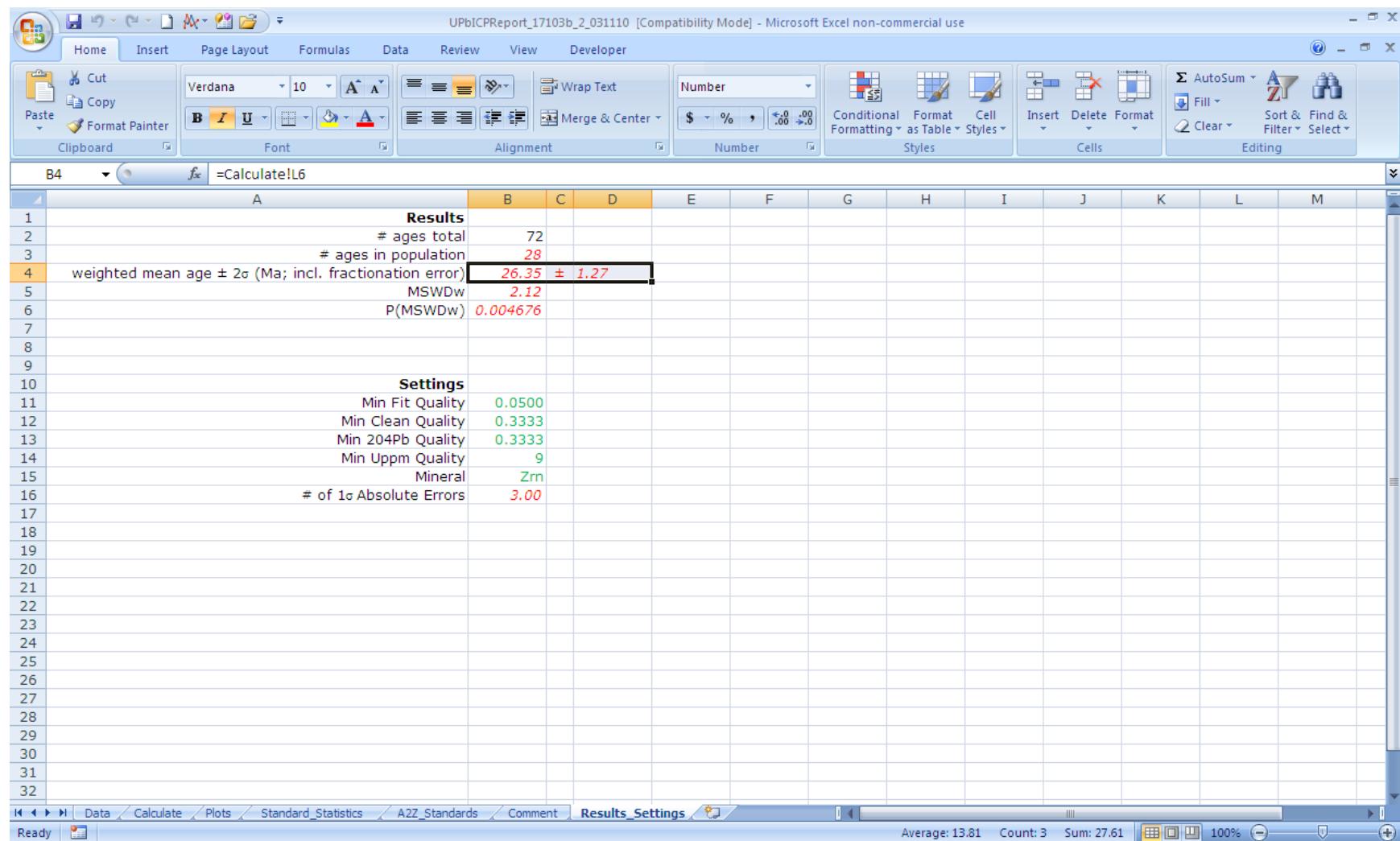


Figure 10. Worksheet: Results\_Settings

UPbICPViewGrain\_17103b\_20\_031110 [Compatibility Mode] - Microsoft Excel non-commercial use

Home Insert Page Layout Formulas Data Review View Developer

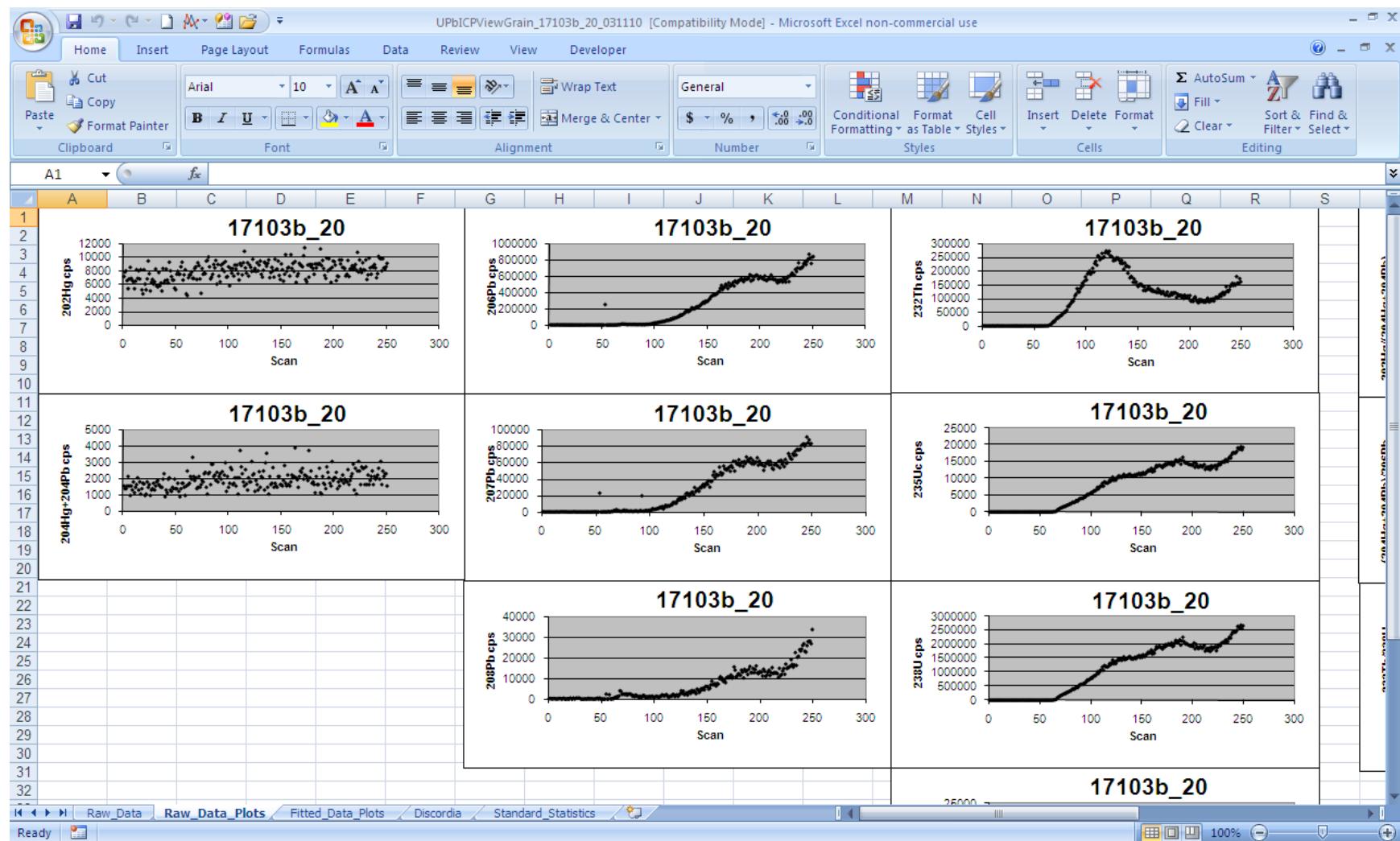
Cut Copy Paste Format Painter Clipboard Font Alignment Number Conditional Formatting as Table Styles Insert Delete Format Cells AutoSum Fill Clear Sort & Find & Filter Select

U15 f2 20

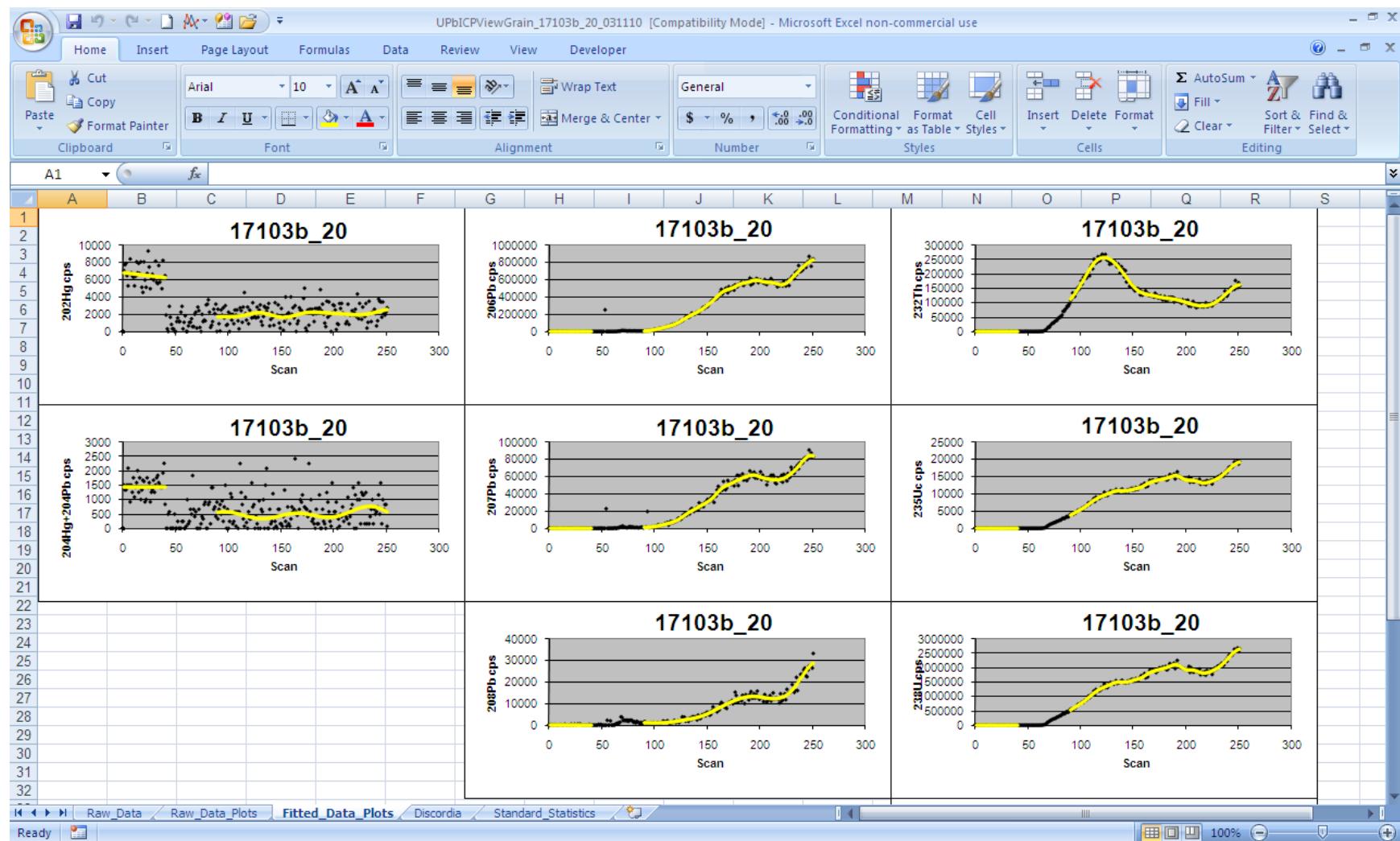
S	T	U	V	W	X	Y	Z	AA	AB	AC	AD	AE	AF	AG	AH	AI	AJ	
1	set	run file	c:\UPbICP\UPbZrSiZ031110.csv		bkg_Fit	isotope	coefficient	coefficient	coefficient	1σ coefficient	1σ coefficient	1σ coefficient	1σ coefficient	value at n	raw standard devi			
2		file type	2				1	6834.43	-14.5273	0	345.5884	14.5238	1	6253.336	1040.544			
3		bkg order	1				2	1438.32	0	0	48.73386		1	1438.32	296.4365			
4		std sig order	0				3	381.6464	-7.66632	0	80.51627	3.351477	1	74.99352	239.106			
5		session order	3				4	80.02804	-1.19234	0	29.18907	1.234741	1	32.33433	86.39104			
6		unk sig order	5				5	205.6142	-2.30106	0	49.15406	2.112682	1	113.5719	152.4879			
7		bkg begin	1				6	175.2195	-0.1235	0	49.41291	2.05779	1	170.2793	144.6663			
8		bkg end	40				7	0	0	0	0	0	0	0	0	1		
9		sig begin	90				8	789.8837	-16.3902	0	160.8436	7.093056	1	134.2737	492.2503			
10		sig end	250				9	5.728761	-0.11887	0	1.166546	0.051444	1	0.973854	3.570136			
11																		
12	set	disk:\folder	c:\UPbICP\															
13	set	session	PbZrSiZ031110															
14	set	root (CASE!)	17103b															
15	set	scroll grain number	20					bkg+sign	isotope	degrees	chisq	Q	1σ (stdde	Gaussian	amplitude	mean (sc	standard deviation	
16	set	extension	.txt					202Hg		1	121	102.6774	0.884875	93.03654	1	3650.319	70.62784	49.08384
17	set	grains	25												2	3522.134	85.35723	58.29498
18	filename	c:\UPbICP\UPbZrSiZ031110\17103b_20.txt													3	2622.897	122.9659	38.15323
19	scans	250													4	2188.742	134.2201	21.56248
20	set	Std/Unk	Unk												5	2257.01	166.2178	24.68312
21								deleted	measured	fit					6	2579.289	176.3416	29.32299
22		title	17103b_20	202Hg	0.65	133.23	133.23								7	2190.113	203.6227	26.52433
23				204Hg+204Pb	0.97	31.28	31.28								8	3085.601	228.7843	71.82386
24		reading file		6	206Pb	0.00	6397.83	6397.80							9	3330.49	268.7222	54.38105
25		status		ready	207Pb	1.82	664.27	664.27							10	3219.355	271.0428	69.00866
26					208Pb	0.00	155.85	155.85	204Hg+20	2	125	102.6614	0.928427	48.17649	1	518.6675	57.24311	78.13519
27					232Th	0.00	2430.60	2430.63							2	442.5729	88.18353	42.61623
28					235U	0.00	198.36	198.36							3	702.3949	95.76696	42.05452
29					238U	0.00	27217.30	27217.62							4	541.595	138.892	80.49998
30					235Uc	0.00	197.40	197.39							5	462.0818	167.9482	35.20547
31					sum	n.a.	37426.13	37426.45							6	456.0786	162.751	35.11963
32															7	425.6167	217.004	79.70284

Raw\_Data Raw\_Data\_Plots Fitted\_Data\_Plots Discordia Standard\_Statistics

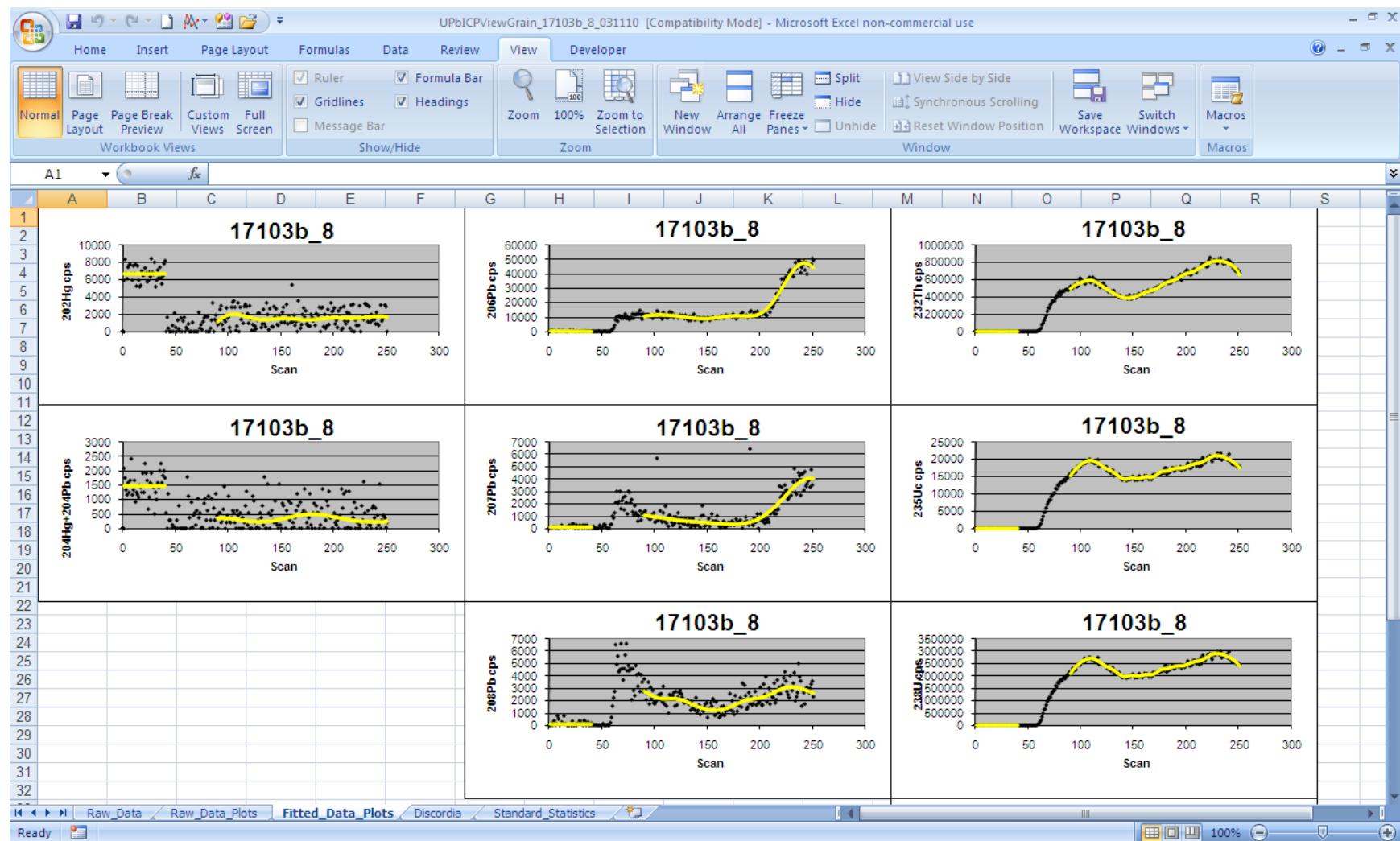
Figure 11. Worksheet: Raw\_Data



**Figure 12.** Worksheet: Raw\_Data\_Plots



**Figure 13a.** Worksheet: bkg\_Corrected\_Data\_Plots for spot 17103b\_20.



**Figure 13b.** Worksheet: bkg\_Corrected\_Data\_Plots for spot 17103b\_8.

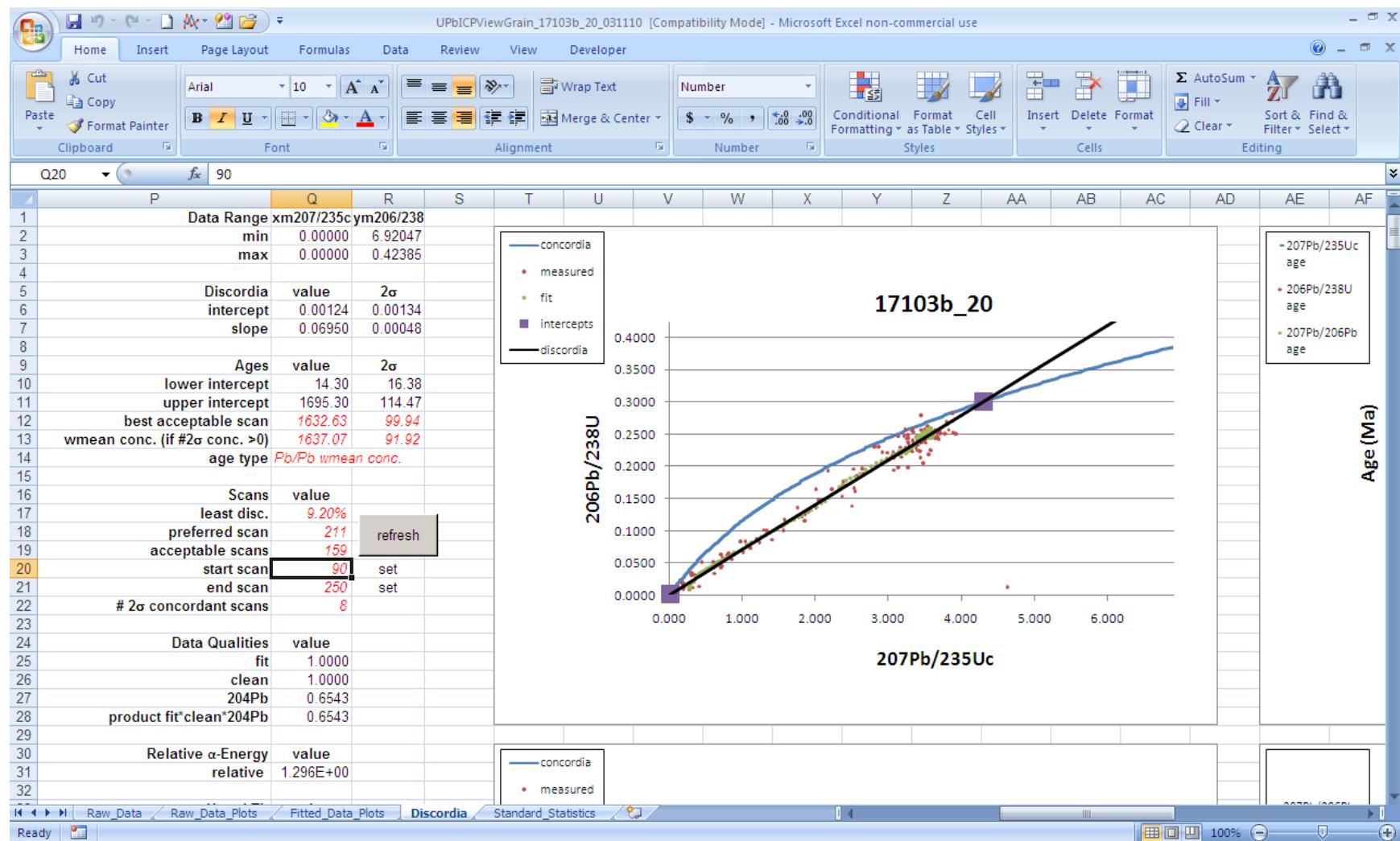
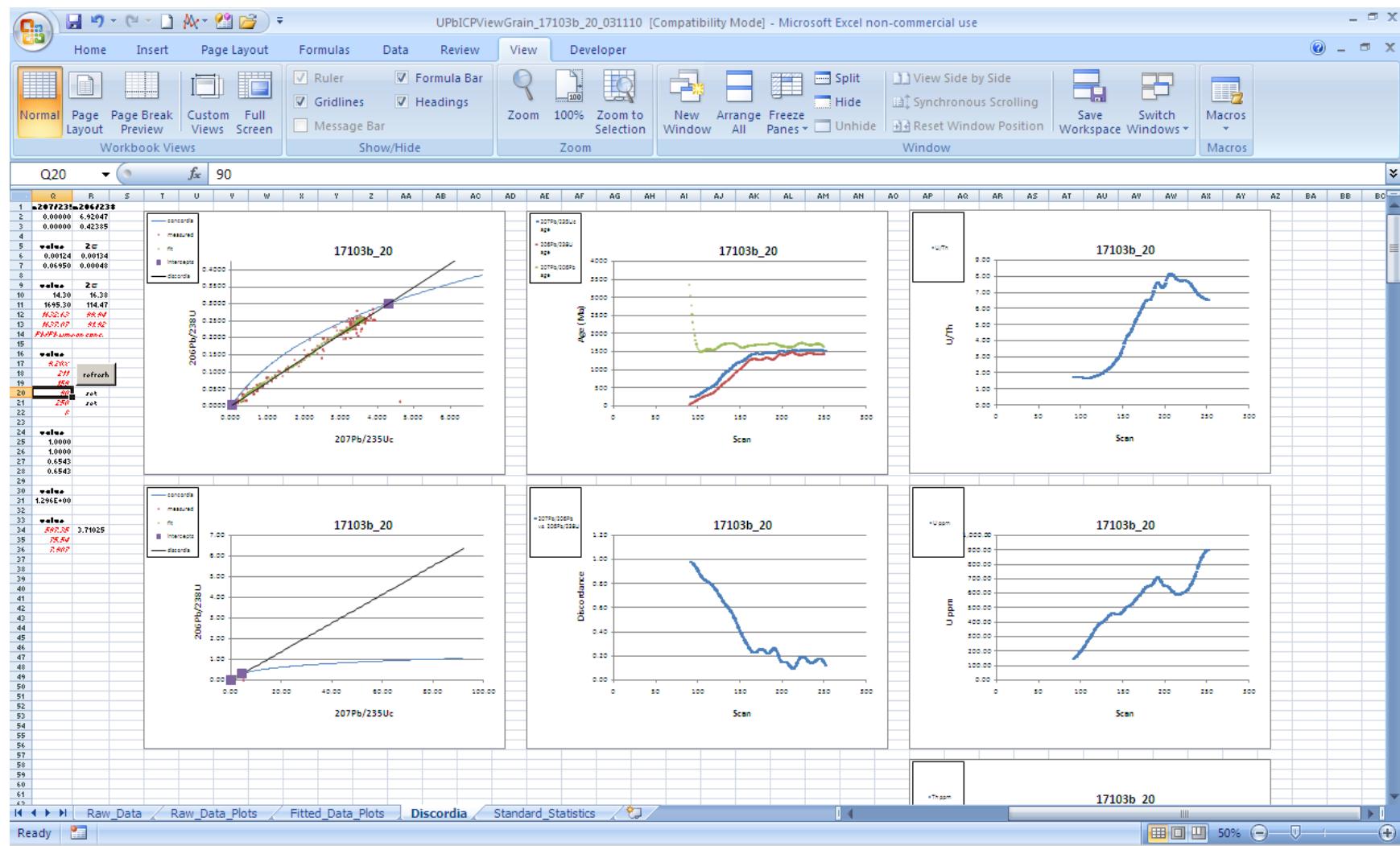


Figure 14a. Worksheet: Discordia



The screenshot shows a Microsoft Excel spreadsheet titled "UPbICPViewGrain\_17103b\_20\_031110 [Compatibility Mode] - Microsoft Excel non-commercial use". The ribbon tabs are Home, Insert, Page Layout, Formulas, Data, Review, View, and Developer. The Home tab is selected.

The data is organized into several sections:

- Primary Standard:**

	A	B	C	D	E	F	G	H	I	J	K	L	M	N
1	<b>Primary Standard</b>													
2	UPbICP Standard Index	6	Standard											
3	Accepted Age	1099												
4	<b>Isotopic Ratio</b>	<b>Mean Age</b>	<b>FF Relative Error</b>											
5	207Pb/235Uc	1096.40	0.0112											
6	207Pb/235Uc*	1096.83	0.0185											
7	206Pb/238U	1095.96	0.0279											
8	207Pb/206Pb	1099.00	0.0232											
9	208Pb/232Th	1097.62	0.0679											
- Secondary Standard:**

	A	B	C	D	E	F	G	H	I	J	K	L	M	N
10	<b>Secondary Standard</b>													
11	UPbICP Standard Index	2	Standard											
12	Accepted Age	61.23												
13	Uncorrected Age	59.31												
- 28Si 91Zr All Standards:**

	Mineral	Zrn												
15														
16	<b>Statistics</b>	<b>91Zr/28Si</b>	<b>28Si</b>	<b>91Zr</b>										
17	median	0.669	4064708	2720961										
18	standard deviation	0.071	225898	245894										
19	minimum	not applicable	3588061	2220840										
20	maximum	not applicable	4391908	3083139										

The status bar at the bottom shows "Ready" and "100%".

Figure 15. Worksheet: Standard\_Statistics