1) Intro: Where are the geotherms?
   Geothermometry in basins by: vitrinite reflectance, bitumen refl.,
   graptolite refl., Raman spectroscopy, conodont alteration,
   spore colour, fluorescence, Rock-Eval, clay mineralogy

2) Fission track dating: - nuclear physics
   - age equation, statistics

3) Volcanic events (= formation ages)
   Basement exhumation (= cooling ages)

4) Complex T histories of basins & thermal modelling

5) Detrital geochronology (provenance by single-grain ages)

6) (U-Th)/He thermochronology

7) K/Ar, Ar/Ar, Luminescence, ESR and cosmogenic dating of sediments

8) U/Pb and U/Th dating of sediments
Geochronology

Radioactive elements for geochronology

$3^3$H, $14^4$C, $40^4$K, $87^{14}$Rb, $115^{11}$In, $138^{13}$La, $147^{14}$Sm, $176^{17}$Lu, $187^{18}$Re, $235^{23}$U, $238^{23}$U, $232^{23}$Th
Geochronology

Relative dating methods
A relative age is the age of a fossil organism, rock, or geologic feature or event defined relative to other organisms, rocks, or features or events rather than in terms of years. *(Paleontology, biostratigraphy, structural geology)*

Correlation methods in geochronology
Like fossils, the chemical and physical characteristics of rocks, minerals, and organic materials can be used for correlation. *(Paleomagnetic dating, tephrochronology, $^{87}\text{Sr} / ^{86}\text{Sr}$ geochronology, stable isotope records)*

Numeric Dating Methods (~radiometric dating) /absolute??? dating/
Numeric dating involves methods of determining the geologic age of a fossil, rock, or geologic feature or event given in units of time. [Ma, Myr, ka, kyr] *(40K/40Ar, 40Ar/39Ar, Rb/Sr, Sm/Nd, U/Pb, U-series methods, Pb-210 geochronology, tritium dating, radiocarbon geochronology, fission track, (U-Th)/He, luminescence geochronology)*
**Geochronology**

**Chronostratigraphy** is concerned with age of strata and time relations; it is an attempt to organize the sequence of strata into units that relate to time, determine local time relations, and correlate globally and thus establish a standard global chronostratigraphic scale. The nomenclature involves chronzone, stage, and series.

**Chronostratigraphic units** are bodies of rock that formed during a specific time interval; the chronostratigraphic unit represents all rock on globe that formed during the time interval. The boundaries of chronostratigraphic units are **isochronous surfaces** or time lines. The rank of chronostratigraphic units is based on time not thickness.

**Chronostratigraphic horizon** is an isochronous stratigraphic surface; theoretically it has no thickness. In the real world we use very thin distinctive intervals; called markers, marker horizon, marker bed, key bed, time surface, datum.

**Geochronologic units** are familiar names found on the geologic time scale; they relate time only and provide an arrangement of time units of worldwide scope for reference dates. The precision or effectiveness of geochronologic units decreases with size.
Thermochronology

closure temperature

Multimethod thermochronology

Elevation profile (1 method)

Exhuming mountains
Vertical age distribution

Apatite fission track age

Thermochronology closure temperature

Multimethod thermochronology

Elevation profile (1 method)

Exhuming mountains
Vertical age distribution

Apatite fission track age
Thermochronology: nowadays the result is not a (house)number, but a thermal path

Result of an unsupervised thermal modelling - described well the known thermal events.

[Danišík & Dunkl, 2004]
Thermochronometers

Typically apatite and zircon are used in low-T basin studies.
Selection guide

- U-containing accessory or rock-forming minerals (or glass)
  (apatite, zircon, sphene >> biotite, epidote, garnet.....)
YES: granitoid, intermediate-acid volcanites, tuff, sandstone, metapelites
NO: limestone, clay, marl, basalt, greenschist

***************

Good Question

Collection, ordering and evaluation of existing data:
geochemistry, stratigraphy, organic maturation, diagenesis, structural geology, hydrogeology
Occurrence of sphene
Typical densities of common minerals
At the beginning: separation of apatite and zircon fractions
Radioactive elements for geochronology

$^3$H, $^{14}$C, $^{40}$K, $^{87}$Rb, $^{115}$In, $^{138}$La, $^{147}$Sm, $^{176}$Lu, $^{187}$Re, $^{235}$U, $^{238}$U, $^{232}$Th

$^{238}$U

$0.7\% \quad ^{235}$U $\rightarrow$ $^{207}$Pb

$99.3\% \quad ^{238}$U $\rightarrow$ $^{206}$Pb

$^{238}$U

damaged zone = fission track
Fission tracks in an apatite crystal

(polished and etched by nitric acid – locality: Bohemian Massif)
Applicability depends on the track density (age & U content)
## History of fission track geochronology

<table>
<thead>
<tr>
<th>Year</th>
<th>Contributors</th>
<th>Technique</th>
</tr>
</thead>
<tbody>
<tr>
<td>1958</td>
<td>Young</td>
<td>etching tracks in LiF</td>
</tr>
<tr>
<td>1959</td>
<td>Silk &amp; Barnes</td>
<td>tracks in mica by TEM</td>
</tr>
<tr>
<td>1962</td>
<td>Price &amp; Walker (GE)</td>
<td>etching tracks in mica</td>
</tr>
<tr>
<td>(engineers of GE)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>1964</td>
<td>Fleischer &amp; Price</td>
<td>etching tracks in 29 minerals</td>
</tr>
<tr>
<td></td>
<td>Fleischer et al.</td>
<td>first dating</td>
</tr>
<tr>
<td></td>
<td>Naeser</td>
<td>apatite, sphene</td>
</tr>
<tr>
<td></td>
<td>Wagner 1972</td>
<td>thermochronology</td>
</tr>
</tbody>
</table>
### Fissionable isotopes and reactions

<table>
<thead>
<tr>
<th>natural fission</th>
<th>induced fission</th>
</tr>
</thead>
<tbody>
<tr>
<td>$^{238}\text{U}$</td>
<td>neutrons from $(\alpha,n)$ and $(\gamma,n)$ reactions</td>
</tr>
<tr>
<td>$^{235}\text{U}$</td>
<td>$^{238}\text{U}$</td>
</tr>
<tr>
<td>$^{232}\text{Th}$</td>
<td>$^{244}\text{Pu}$</td>
</tr>
<tr>
<td>$^{248}\text{Cm}$</td>
<td>$^{232}\text{Th}$</td>
</tr>
</tbody>
</table>

### Track production in an apatite crystal

| $^{238}\text{U}$ | 30.8 |
| $^{235}\text{U}$ | 0.01 |
| $^{232}\text{Th}$ | 0.0004 |
TEM (transmission electron microscope) image of a latent fission track in zircon

[Yada et al., 1987]
Cascade process creates "extended" and "point" defects along a track

[TEM image]

[Pellas & Perron, 1984]
"Thermal spike" model

[Chadderton, 1965]
### Some etchants

<table>
<thead>
<tr>
<th></th>
<th>conc.</th>
<th>time</th>
<th>temp. [°C]</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>apatite</strong></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>HNO3</td>
<td>0.25 - 65 %</td>
<td>4.5 m - 10 s</td>
<td>21</td>
</tr>
<tr>
<td><strong>sphene</strong></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>HCl</td>
<td>37%</td>
<td>30-90 m</td>
<td>90</td>
</tr>
<tr>
<td>HF-HCl-HNO3</td>
<td></td>
<td>6 m</td>
<td>20</td>
</tr>
<tr>
<td>NaOH</td>
<td>cc.</td>
<td>20-30 m</td>
<td>130</td>
</tr>
<tr>
<td><strong>zircon</strong></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>H3PO4</td>
<td>cc.</td>
<td>1 m</td>
<td>375</td>
</tr>
<tr>
<td>NaOH</td>
<td>100 N</td>
<td>15m - 4 h</td>
<td>220</td>
</tr>
<tr>
<td>HF-H2SO4</td>
<td></td>
<td>2-10 h</td>
<td>150</td>
</tr>
<tr>
<td>NaOH-KOH</td>
<td>eutekt</td>
<td>2-60 h</td>
<td>200</td>
</tr>
<tr>
<td>NaOH-KOH-LiOH</td>
<td>eutekt</td>
<td>2-40 h</td>
<td>190</td>
</tr>
<tr>
<td><strong>muscovite</strong></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>HF</td>
<td>40-48 %</td>
<td>4 - 120 m</td>
<td>21</td>
</tr>
</tbody>
</table>
Fission tracks in an apatite crystal

(polished and etched by nitric acid – locality: Bohemian Massif)
Figure 7.1. Hydrated, silicic glass shards from Banks Island tephra, western Arctic, with induced pre-annealed (3 months at 100°C) fission tracks. (Courtesy by J. Westgate.)
The etched tracks have blade shape in anisotropic crystals

[Etch pit]

[c-axis]

[Cystal surface]

[Rapid etching range]

[Slow etching range]

[Bulk etching velocity pattern]

[Yamada et al., 1993]

[Gleadow et al., 2002]
Dependence of etching time and track shape on metamictization

Gleadow et al. (1976)
Metamictization of zircon

Fig. 3. IR powder spectra in the range 1500–250 cm⁻¹ of three Sri Lanka gem zircon samples given in units of absorbance per mg as a function of wavenumber; cryst = Sri Lanka gem zircon 2916C, nonmetamict, 20 ppm eU. Absorption bands rep-

[Zhang et al., 2000] [Woodhead et al., 1991]
Flow-chart of fission dating method (external detector procedure)

- Accumulation of spontaneous fission tracks
- Polished section through crystal
- Spontaneous tracks etched
- External mica detector attached
- Thermal neutron irradiation
- Induced fission tracks register in detector
- Induced tracks etched only in detector

Plan view of several crystals

Grain mount showing spontaneous tracks in the individual grains

External detector mount showing induced tracks defining grain outlines

[Gallagher et al., 1998]
Latent tracks in U containing minerals from the natural fission of 238-U

After polishing and etching:

Number of spontaneous tracks
\( N_s = f(\text{Age, U content}) \)

Neutron irradiation produces induced fission tracks of 235-U

Number of induced tracks
\( N_i = f(\text{F}_n, \text{s}_{(n,F)}, \text{U content}) \)
ZIRCON INCLUSION IN APATITE

Spontaneous tracks

Induced tracks in external detector.

100 μm
Population method

External detector method

[Naeser et al., 1981]
Identification of etched fission tracks

1) Always straight line, pipe or cone.
2) Length max 20 µm, can not be more.
3) Random orientation.
4) Latent tracks disappear by annealing, while dislocations remain etchable.

\[ \rho_s = f(U, \lambda f, \text{time}) \]
Heterogeneous track density (uranium concentration) in apatite crystals and prints
**Etched crystal defects**
(~dislocations are curved, oriented and can be longer than a fission track)
Zeta method

needs: age standards + U-glasses

\[
\zeta = \frac{e^{\lambda_D T^{STD}} - 1}{\lambda_D \left( \frac{\rho_S}{\rho_I} \right)_{STD}} g \rho_D
\]

\[
Age = \frac{1}{\lambda_D} \ln \left[ 1 + \lambda_D \zeta \frac{\rho_S}{\rho_I} \rho_D \right]
\]

\[
\sigma(T) = T \sqrt{\frac{1}{N_s} + \frac{1}{N_I} + \frac{1}{N_D} + \left[ \frac{\sigma(\zeta)}{\zeta} \right]^2}
\]

[Hurford & Green, 1983]
Automatic track counting  Track Review software based on Coincidence mapping

Advantages: faster, no humans suffering

Problems:
- does not measure the track length and the straightness.
- relatively slow and needs a lot of checking
- does not estimate the parallel tracks and any other possible defects which seems like a track.

[Gleadow et al., 2009]
New age? Measuring spontaneous tracks by microscope and U content by laser ablation ICP-MS

\[ \varepsilon = \frac{\rho_s \lambda_D M}{\lambda_f N_A^{238} U \times 10^{-6} dR_{sp} K} \]

\[ t = \frac{1}{\lambda_D} \ln \left( 1 + \varepsilon \cdot \frac{\rho_s}{238U} \right) \]

Fig. 3. Single-grain FT apatite ages calculated by using \(^{238}\)U content determined by LA-ICP-MS plotted against FT ages determined by conventional zeta age calibration. Continuous lines represent reference age of each apatite standard (e.g. Hurford, 1998). Error bars are ±1 standard error.

[Hasebe et al., 2004]
New age? Measuring spontaneous tracks by microscope and U content by laser ablation ICP-MS
Real value

-1000%  -100%  +10%  +10%  +100%  +1000%

Sampling
Storage, Preparation
Analysis
Presentation of results
Interpretation

Source: BASF
“Hard rock” - one age cluster

"hard rock"

"soft rock"
### Recommended data presentation

dispersion (Galbraith & Laslett, 1993)

<table>
<thead>
<tr>
<th>Sample Code</th>
<th>Cryst.</th>
<th>Spontaneous $\rho_s$ (Ns)</th>
<th>Induced $\rho_i$ (Ni)</th>
<th>Dosimeter $\rho_d$ (Nd)</th>
<th>$P(\chi^2)$ [%]</th>
<th>Disp. [Ma]</th>
<th>FT age ± 1$\sigma$ [Ma]</th>
<th>U [ppm]</th>
<th>Track length (n) [µm]</th>
</tr>
</thead>
<tbody>
<tr>
<td>01-31</td>
<td>20</td>
<td>2.40 (244)</td>
<td>12.3 (1244)</td>
<td>5.30 (3106)</td>
<td>2</td>
<td>20.6 ± 2.0</td>
<td>17 11.8 ± 2.2</td>
<td>(62)</td>
<td></td>
</tr>
<tr>
<td>01-34</td>
<td>20</td>
<td>2.52 (295)</td>
<td>11.5 (1347)</td>
<td>5.24 (3106)</td>
<td>89</td>
<td>21.4 ± 1.5</td>
<td>24 14.5 ± 0.9</td>
<td>(50)</td>
<td></td>
</tr>
<tr>
<td>01-37</td>
<td>20</td>
<td>2.67 (349)</td>
<td>12.5 (1636)</td>
<td>5.48 (3889)</td>
<td>52</td>
<td>21.8 ± 1.4</td>
<td>7 13.1 ± 1.2</td>
<td>(102)</td>
<td></td>
</tr>
<tr>
<td>01-38</td>
<td>20</td>
<td>3.12 (341)</td>
<td>15.7 (1708)</td>
<td>5.41 (3889)</td>
<td>59</td>
<td>20.1 ± 1.3</td>
<td>34 13.1 ± 0.9</td>
<td>(104)</td>
<td></td>
</tr>
</tbody>
</table>

[after Hurford, 1990]
## Effect of the number of crystals

<table>
<thead>
<tr>
<th>Sample Code</th>
<th>$\rho_s(N_s)$, $10^6$cm$^{-2}$</th>
<th>$\rho_i(N_i)$, $10^6$cm$^{-2}$</th>
<th>$\rho_D(N_D)$, $10^6$cm$^{-2}$</th>
<th>n</th>
<th>r</th>
<th>Age($\pm\sigma$), Ma</th>
</tr>
</thead>
<tbody>
<tr>
<td>Zircon</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>RN11ZRO1</td>
<td>5.47(540)</td>
<td>5.15(509)</td>
<td>0.3077(4863)</td>
<td>2</td>
<td>1.000</td>
<td>55.6 ± 3.5</td>
</tr>
<tr>
<td>16ZRO1</td>
<td>9.72(937)</td>
<td>4.98(480)</td>
<td>0.1608(3738)</td>
<td>4</td>
<td>0.735</td>
<td>53.5 ± 3.1</td>
</tr>
<tr>
<td>17ZRO1</td>
<td>6.66(1083)</td>
<td>3.64(592)</td>
<td>0.1608(3738)</td>
<td>5</td>
<td>0.983</td>
<td>50.1 ± 2.7</td>
</tr>
<tr>
<td>18ZRO1</td>
<td>6.40(1421)</td>
<td>5.89(1307)</td>
<td>0.3077(4863)</td>
<td>6</td>
<td>0.700</td>
<td>57.0 ± 2.3</td>
</tr>
<tr>
<td>19ZRO1</td>
<td>5.89(2096)</td>
<td>5.43(1929)</td>
<td>0.3077(4863)</td>
<td>4</td>
<td>1.000</td>
<td>56.9 ± 2.0</td>
</tr>
<tr>
<td>22ZRO1</td>
<td>4.84(1855)</td>
<td>4.69(1799)</td>
<td>0.3077(4863)</td>
<td>5</td>
<td>0.987</td>
<td>54.0 ± 1.9</td>
</tr>
<tr>
<td>32ZRO1</td>
<td>5.36(436)</td>
<td>5.80(472)</td>
<td>0.3077(4863)</td>
<td>2</td>
<td>1.000</td>
<td>48.4 ± 3.3</td>
</tr>
<tr>
<td>Apatite</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>RN11APO1</td>
<td>1.943(921)</td>
<td>6.471(3068)</td>
<td>0.7265(3039)</td>
<td>6</td>
<td>0.164</td>
<td>37.2 ± 1.6</td>
</tr>
<tr>
<td>15APO2</td>
<td>1.479(1162)</td>
<td>4.220(3315)</td>
<td>0.7322(4339)</td>
<td>7</td>
<td>0.979</td>
<td>43.8 ± 1.6</td>
</tr>
<tr>
<td>17APO2</td>
<td>0.057(55)</td>
<td>0.569(549)</td>
<td>0.7322(4339)</td>
<td>11</td>
<td>0.760</td>
<td>12.5 ± 1.8</td>
</tr>
<tr>
<td>18AP</td>
<td>0.406(567)</td>
<td>5.426(7585)</td>
<td>0.7322(4339)</td>
<td>18</td>
<td>0.953</td>
<td>9.4 ± 0.4</td>
</tr>
<tr>
<td>19APO1</td>
<td>0.284(332)</td>
<td>2.937(3430)</td>
<td>0.7365(3081)</td>
<td>12</td>
<td>0.512</td>
<td>12.2 ± 0.7</td>
</tr>
<tr>
<td>20APO2</td>
<td>0.200(112)</td>
<td>2.307(1292)</td>
<td>0.7365(3081)</td>
<td>8</td>
<td>0.750</td>
<td>10.9 ± 1.1</td>
</tr>
<tr>
<td>21APO1</td>
<td>0.177(70)</td>
<td>2.488(983)</td>
<td>0.7365(3081)</td>
<td>5</td>
<td>0.984</td>
<td>9.0 ± 1.1</td>
</tr>
<tr>
<td>30APO2</td>
<td>0.679(56)</td>
<td>1.435(120)</td>
<td>0.7365(3081)</td>
<td>2</td>
<td>1.000</td>
<td>59 ± 10</td>
</tr>
<tr>
<td>32APO1</td>
<td>0.698(532)</td>
<td>1.611(1228)</td>
<td>0.7365(3081)</td>
<td>8</td>
<td>0.976</td>
<td>54.3 ± 3.0</td>
</tr>
</tbody>
</table>

- $\rho_s$, spontaneous track density; $N_s$, total number of spontaneous tracks counted; $\rho_i$, induced track density; $N_i$, total number of induced tracks counted; $\rho_D$, induced track density in a dosimeter glass; $N_D$, total track number to determine $\rho_D$; n, number of grains or fields counted; r, correlation coefficient between individual track counts.
Sources of extra-Poissonal error:

(A) Non-uniform age of crystals,

(B) Spatial variation of neutron fluence,

(C) Improper etching,

   Careless counting,

   Presence of track-like defects,

   Bad contact between mount and mica,

   Imprecise positioning,

(D) Vertical inhomegeneity of uranium.

[after Green, 1981]
The crystals have different track densities and thus ...
The crystals have different counts and thus different error.

<table>
<thead>
<tr>
<th>Number of tracks</th>
<th>FT age ± 1s [Ma]</th>
</tr>
</thead>
<tbody>
<tr>
<td>71</td>
<td>46 ± 8</td>
</tr>
<tr>
<td>45</td>
<td>34 ± 7</td>
</tr>
<tr>
<td>91</td>
<td>38 ± 6</td>
</tr>
<tr>
<td>45</td>
<td>28 ± 6</td>
</tr>
<tr>
<td>4</td>
<td>13 ± 7</td>
</tr>
<tr>
<td>66</td>
<td>49 ± 9</td>
</tr>
<tr>
<td>46</td>
<td>45 ± 9</td>
</tr>
<tr>
<td>420</td>
<td>42 ± 6</td>
</tr>
<tr>
<td>64</td>
<td>28 ± 5</td>
</tr>
<tr>
<td>18</td>
<td>21 ± 6</td>
</tr>
</tbody>
</table>
Average - but how?

**Pooled age:** the sum of spontaneous counts divided by the sum of induced counts

**Mean age:** arithmetic mean of the individual ratios of spontaneous to induced tracks

**Central age:** weighted mean of log normal distribution of single grain ages

**rejection**

To weight, or not to weight – that is the question:

To be, or not to be- that is the question

\[
\rho_{\text{RATIO}} = \frac{\sum w_j \rho_{s,j}}{\sum w_j \rho_{i,j}}
\]

**need: type of distribution**
What is an isochron plot?
The scatter comes from the low counts and from the "extra-Poissonal" error.
**RhoS vs. RhoI**

**A-303.APA**  
Dolomiti  DO-2

"Knödel" - type

Chi-sq.  P (%):
22.95   52.25  
Dispersion: 0.04
  a: 1.075  b: 0.447  r: 0.53

U (ppm):  V.C.:
13.87  (± 18 %)

**A-304.APA**  
Dolomiti  DO-7

"Spagetti" - type

Chi-sq.  P (%):
36.69   1.83  
Dispersion: 0.14
  a: 0.125  b: 0.535  r: 0.97

U (ppm):  V.C.:
58.28  (± 84 %)
Outlier? What to do?

need: type of distribution

dixon test $p < 0.5$
grubbs test $p < 1%$
"Zero track grains"
Allocate each measurement a Gaussian curve of unit area reflecting its uncertainties.
Radial plot
Usual presentation: radial plot + binned diagram + probability density plot

[Dunkl et al., 2010]