Appendix-A Methods[*]

To investigate the geological occurrence, spatial distribution and the geological relationships between the high-pressure and ultrahigh-pressure mafic eclogitic rocks and their host rocks in the Yuka and Xitieshan terranes, North Qaidam Orogen, Western China, extensive field work was carried out at the beginning of the PhD project in July 2009, by a research collaboration of staff and students from the VU University Amsterdam and the Guangzhou Institute of Geochemistry, Chinese Academy of Sciences. Important and representative samples from the Yuka and Xitieshan terranes for further metamorphic, geochemical and geochronological studies were collected.

Petrographic examination was undertaken on about 100 thin-sections, in order to recognize different mineral assemblages and metamorphic stages. Representative thin-sections were selected for mineral compositional analysis using Electron Probe Mirco-Analysis (EMPA), to determine mineral chemistry to be used for calculation of P-T conditions, and further to reconstruct the tectono-metamorphic history of the study region (See Chapters 3 and 7). Bulk geochemical analysis of major elements was carried out by X-ray fluorescence (XRF) on several mafic eclogite samples from the Yuka terrane, which is used to discuss the petrogenesis and possible tectonic environment of these rocks (See Chapter 3). In order to constrain the cooling/exhumation process of the UHP metamorphic rocks in north Qaidam Orogen, laser stepwise heating $^{40}Ar/^{39}Ar$ analyses were performed on metamorphic minerals (amphibole, white mica, biotite and K-feldspar) from different rock types (See Chapters 4 and 8). *In vacuo* crushing is an accepted technique to release selectively gas trapped in fluid inclusions in crystalline material and along cracks and cavities in crystals. To decipher the genesis of extraneous ^{40}Ar in metamorphic amphibole, using a combination of *in vacuo* crushing and stepwise heating, the $^{40}Ar/^{39}Ar$ technique has been applied to amphiboles from two Yuka garnet amphibolites (See Chapter 5). To address the origin of fluid flow and constrain the age of quartz veining directly, preliminary micro-thermometric measurements combined with $^{40}Ar/^{39}Ar$ *in vacuo* crushing dating were carried out on quartz veins in eclogite-facies HP/UHP metamorphic rocks from the Yuka terrane (See Chapter 6).

A.1 Electron Probe Micro-Analysis

Chemical compositions of mineral phases were analysed using a JEOL JXA-8800M electron microprobe at the Department of Earth Sciences of VU University Amsterdam.

The analyses were performed at an accelerating voltage of 15.0 kV and a beam current of approximately 20 nA, a counting time of 20-30 seconds for peaks and 10-15 seconds for background. The data was regressed using an oxide-ZAF correction program supplied by JEOL. Natural minerals were used as standards (see Table A.0.1). The approach of calculation of

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[*] This chapter was part of the Chinese version of the thesis.

Fe^{3+} in garnet and clinopyroxene is based on the scheme of Droop (1987). The molecular formula for amphibole was calculated according to Holland and Blundy (1994). Total Fe is reported as Fe^{2+} in white mica and as Fe^{3+} in epidote group minerals.

Table A.0.1 Standards used for mineral analysis by the electron microprobe

Series 1:Clinopyroxene, Amphibole, Plagioclase, Mica, Zoisite				Series 2:Garnet		
Element	Standard	Spot Size		Element	Standard	Spot Size
Si	Diopside	1 µm		Si	Almandine	1 µm
Al	Corundum	1 µm		Al	Almandine	1 µm
Mg	Diopside	1 µm		Mg	Diopside	1 µm
Fe	Olivine	1 µm		Fe	Almandine	1 µm
Mn	Tephroite	1 µm		Mn	Tephroite	1 µm
Ti	Ilminite	1 µm		Ti	Ilminite	1 µm
K	Orthoclase	10 µm		K	Orthoclase	10 µm
Na	Jadeite	5 µm		Na	Jadeite	5 µm
Ca	Diopside	1 µm		Ca	Diopside	1 µm

A.2 Whole rock chemistry

Whole rock chemistry of the samples was determined by X-ray fluorescence (XRF) technique. To prepare fused beads for major element analyses by XRF, rocks were crushed and powdered at the Department of Earth Sciences, VU University. Fine rock powder was made by first removing the weathered rims of the rock samples and cutting the remainder of the rock into small pieces. These pieces were then put into the jaw crusher, the disk mill and the planet mill creating a final homogeneous powder of ~2µm grain size. The material was dried overnight in a stove at 100 °C. The dry material was then weighed and put in the oven at 1000 °C for more than one hour to burn off organic carbon. From the heated material one gram was weighed and mixed with four grams of a lithium tetraborate flux to a homogenous substance. This was put into a Switch Perlx3 apparatus, which melts the material and shapes it into beads. These beads were then analyzed in a PANalytial Magix Pro XRF instrument for the following major components: Fe_2O_3, Na_2O, CaO, K_2O, MgO, Al_2O_3, TiO_2, MnO and SiO_2. The percentages of major elements were subsequently recalculated for ferrous iron values, assuming that only a very minor fraction of iron in the eclogite was ferric.

A.3 $^{40}Ar/^{39}Ar$ analysis

A.3.1 Sample preparation and irradiation facility

Sample preparation has been conducted at the mineral separation laboratory of VU University Amsterdam. Samples were crushed with the jaw crusher and sieved to obtain a size fraction of 200–500 µm. The Faul vibration table was used to separate the flat minerals (mica) from the non-flat ones. Afterwards, standard mineral separation methods (i.e. Franz

magnetic and heavy liquid separation; garnet density: 3.7–4.1g/cm^3, amphibole density: 3.1–3.3 g/cm^3, K-feldspar density: 2.54–2.59 g/cm^3, quartz density: 2.64–2.66 g/cm^3,) were used. At the end of these procedures, all the samples were handpicked under a binocular microscope. Before and after hand-picking, the crystals were ultrasonically cleaned in H$_2$O. The samples were wrapped in aluminum foil and put into quartz tubes together with the monitor standards for irradiation. The packet thicknesses of all samples and monitors were recorded. Afterwards, the *J*-value of each sample was calculated by interpolation from the line (or curve) of *J*-value *vs.* height based on the monitor data in each sample tube. The *J*-value uncertainty of 0.15% (1σ) was propagated into the age calculations.

The irradiation facilities used for this project were the cadmium-lined CICLIC facility of the TRIGA reactor at the Oregon State University Reactor Centre, USA (batch VU83); the HFR reactor at the NRG (Nuclear Research and Consultancy Group) operated facility in Petten, The Netherlands (batch VU89); and the pool of the 49-2 reactor in China Institute of Atomic Energy, Beijing, China (batch GZ17). Irradiation times were 20 hrs and 12 hrs for irradiations VU83 and VU89. Correction factors for interferences of Ca and K isotopes are: $(^{39}Ar/^{37}Ar)_{Ca}$ = 0.000673, $(^{36}Ar/^{37}Ar)_{Ca}$ = 0.000264, $(^{40}Ar/^{39}Ar)_K$ = 0.00086 and $(^{38}Ar/^{39}Ar)_K$ = 0.01211. These values were determined using zero age K-feldspar and anorthite glass. The flux monitor standard for this project was DRA sanidine, with an age of 25.26 ± 0.2 Ma (Wijbrans et al., 1995). These values are compatible with the set of Renne et al. (1998), based on biotite GA1550 (at K/Ar age of 98.79 ± 0.69 Ma). Irradiation times were 50 hrs for GZ17. Correction factors for interfering argon isotopes derived from Ca and K isotopes are: $(^{39}Ar/^{37}Ar)_{Ca}$ = 0.0008984, $(^{36}Ar/^{37}Ar)_{Ca}$ = 0.0002673, $(^{40}Ar/^{39}Ar)_K$ = 0.00597 and $(^{38}Ar/^{39}Ar)_K$ = 0.01211. The flux monitor standards for *j*-value calculation for this project were ZBH-25 biotite with a K-Ar age of 132.7 ± 1.2 Ma (Wang, 1983). The biotite analyzed by $^{40}Ar/^{39}Ar$ stepwise heating forms a flat age spectra, with plateau age of 132.7 ± 0.1 Ma recalculated with the GA-1550 reference age of 98.79 ± 0.69 Ma (Renne et al., 1998) and the ^{40}K decay constant of Steiger and Jager (1977).

A.3.2 Laser stepwise heating

The $^{40}Ar/^{39}Ar$ laser stepwise heating experiments were carried out on a MAP-215-50 mass spectrometer at VU University Amsterdam and a GVI-5400$^{®}$ mass spectrometer at the State Key Laboratory of Isotope Geochemistry, Guangzhou Institute Geochemistry, Chinese Academy of Sciences (GIG-CAS), respectively. In order to correct the backgrounds, the experiments always begin with a line background measurement and additional line backgrounds were analysed after every four to six gas extraction steps.

In the VU laboratory, the MAP-215-50 had been fitted with a SEV-216 electron multiplier with the amplifier equipped with three resistors of 10^9, 10^8 and 10^{7}Ω that automatically switch according to the ^{40}Ar signal intensities during peak-centering on the gas before analysis. Laser step heating was performed with a 50W Synrad$^{©}$ 48-5 series CO$_2$ laser, using a Raylase industrial scan head as a beam diffuser by application of a triangular current

on the Y-axis motor drive of the scan head. The gases released were purified by a Fe/V/Zr getter pump operated at 250 °C and a Zr/Al pump operated at 450 °C (Wijbrans et al., 1995; Qiu and Wijbrans, 2006; Wijbrans et al., 2011).

In the Guangzhou laboratory, a COHERENT[©] 50-watt CO_2 laser is used for stepwise heating. An in-house designed and manufactured sample chamber with the ZnS 50mm-viewport mounted on a NW63CF flange is connected to the purification line through stainless steel flexible tubing. The gases were purified for 5–8 minutes by two Zr/Al getter pumps operated at room temperature and at about 450 °C respectively (Qiu and Jiang, 2007; Qiu and Wijbrans, 2008). The GVI-5400[®] mass spectrometer is equipped with a high Faraday cup and a SEV-217 electron multiplier. The Faraday feedback resistor is $10^{11}\Omega$. The source trap current is set at 200μA during measurement. The signal intensity ratio of the multiplier to the Faraday cup is about 0.84 (Bai et al., 2013).

A.3.3 In vacuo crushing and stepwise heating

In vacuo crushing experiments and stepwise heating of the powder residues were carried out in an in-house designed crushing apparatus that was connected to a three stage extraction line and a quadrupole mass spectrometer (Schneider et al., 2009) in the argon isotope laboratory at VU University Amsterdam. The analytical procedure is described by Qiu and Wijbrans (2006; 2008). Samples were loaded into a 40 cm long, 4 cm diameter Inconel[®] tube. The tube has spherical curvature on the inside base. The base is made of a 10 cm long, 4 cm diameter solid rod of Inconel[®] that was welded to the tube. Crushing is achieved by moving a magnet steel dead-weight, with a machined spherical curvature on the one end, grooves on the side and a threaded nipple on the top end (for easy removal). The curvature on the dead-weight is of slightly smaller radius when compared to that of the crusher base, allowing concentration of powder near the tube central axis during crushing. Prior to analysis, blanks were reduced by heating the whole apparatus to 150 °C using heating tape and the sample tube to 300 °C using a furnace for *ca.* 12 hrs.

The loaded samples were crushed by an iron pestle that is lifted and dropped with a frequency of one time per second (1 Hz) using external electromagnet-control. The extracted gases were exposed to a cold trap filled with liquid nitrogen (kept at *ca.* − 90 °C), and then purified at 250 °C by a Fe/V/Zr getter pump and 450 °C by a Zr/Al getter pump. The purification process lasted for around 16 minutes before the gas was analyzed for the argon isotopes in the mass spectrometer. Experiments began and ended with cold blank analyses to correct for system blanks with the procedure described above but without moving the pestle. To analyze residues in stepwise heating experiments, the crusher tube was rotated 90 degrees and horizontally put into an externally temperature-controlled oven. The pestle was moved to the end of the tube to avoid degaussing it during stepwise heating. System blanks were measured in the initial steps at room temperature.

A.4 Microthermometry

Fluid inclusion heating and freezing measurements were performed on doubly polished thick sections of four high pressure quartz vein samples using a Linkam MDS 600 freezing/heating stage coupled to a BX51 Olympus polarizing microscope, with nitrogen as the cooling medium, at the State Key Laboratory of Geological Processes and Mineral Resources, China University of Geosciences, Wuhan, China. The lower and upper temperature limits of the Linkam MDS 600 instrument are -195 °C and +600 °C, respectively. For temperatures below +30 °C precision and accuracy are ±0.2 °C, for temperatures between +30 and +300 °C precision and accuracy are ±1 °C, and about ±2 °C above +300 °C. The heating/freezing stage was calibrated with pure CO_2 in natural fluid inclusions. The rate of heating and cooling is normally ~10 °C/min and reduced to 2 °C/min near phase changes. Phase transitions in aqueous inclusions are best defined by the terminology proposed by Roedder (1984). The homogenization temperatures (T_h) of aqueous fluid inclusions that homogenize to the liquid phase and the temperatures of ice-melting (T_m) were measured. Homogenization temperatures are the minimum trapping temperatures, whereas ice-melting temperatures provide a measure of the fluid salinity (Hall et al., 1988).