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ESR DATING OF THE DONGGUTUO PALAEOLITHIC SITE IN THE NIHEWAN BASIN, NORTHERN CHINA

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Abstract: The fluvio-lacustrine sequences in the Nihewan Basin, northern China, provide important terrestrial archives about Palaeolithic settlements and, therefore, about early human occupation in high northern latitude in East Asia. Here we present detailed ESR dating of the Donggutuo Palaeolithic site, located in this basin. Four levels A, B, C and E of the Donggutuo archaeological layer yield ESR ages ranging from 1060±129 ka to 1171±132 ka with a mean of 1119±132 ka. The ages are consistent with the paleomagnetic data, which show that the Donggutuo Palaeolithic site lies just below the onset of the Jaramillo normal subchron (0.99-1.07 Ma). Furthermore, our results indicate that the reliable ESR dating range of bleached quartz using Ti-Li centre can be effectively extended to 1100 ka and the Ti-Li centre was zeroed before the last deposition, which requires improvement of the understanding of the bleaching mechanism conditions.

Keywords: ESR dating, fluvial sediment, Early Pleistocene, signal bleaching, Palaeolithic site.

1. INTRODUCTION

The chronology of East Asian Palaeolithic and/or early human fossil sites is crucial for the understanding of early human occupation in this region and the overall framework of human origin and migration in the Old World (Zhu *et al*., 2004, 2008; Shen *et al*., 2009). The Nihewan Basin (**Fig. 1**), in temperate northern China, has significantly contributed to our understanding of early human adaptability to high-latitude in East Asia (Zhu *et al*., 2001, 2004; Deng *et al*., 2006, 2007), as most of the rare Early Pleistocene archaeological sites of this region were found in this basin. The age of the Palaeolithic sites in mainland East Asia has long been controversial due to the absence of suitable material for accurate isotopic dating. Since the pioneering paleomagnetic work of Zhu *et al*. (2001) on the dating of the famous Xiaochangliang Palaeolithic site, considerable progress has been made during the past decade towards paleomagnetism applied on the Palaeolithic sites in this basin (Zhu *et al*., 2001, 2003, 2004; Wang *et al*., 2004; Wang *et al*., 2005; Deng *et al*., 2006, 2007; Li *et al*., 2008; Liu *et al*., 2010).

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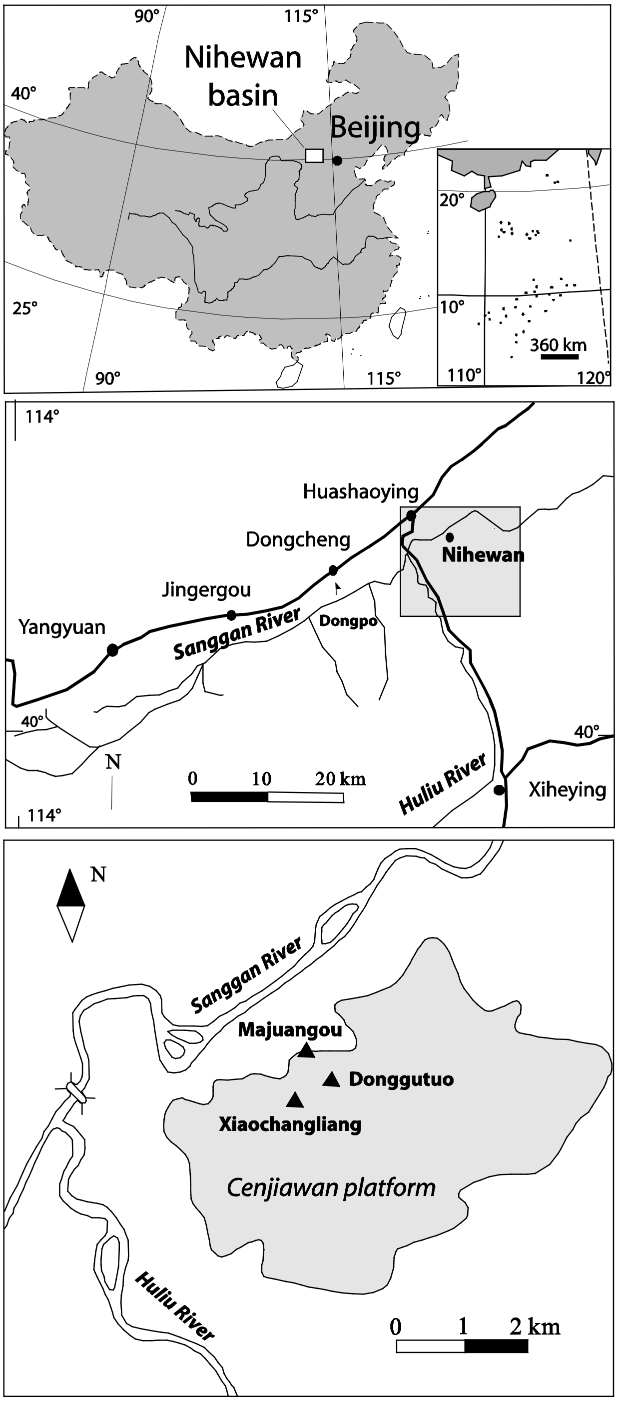

Fig. 1. *Location of the Nihewan Basin, China, and of the main sites of this area, including the Donggutuo site (after Wang* et al.*, 2005).*

The Nihewan basin is not only rich in mammalian fauna but also in Palaeolithic sites. The deposition timing of the Nihewan Formation is critical to understand the chronology of a series of important issues, such as early human adaptability to the hostile environments in high northern latitudes, sedimentary infilling process into the Nihewan Basin, and biostratigraphical sequence of the North China (Zhu *et al.*, 2007). Therefore, it is an important area for the interdisciplinary study of Quaternary geology, palaeoenvironments, palaeontology, geochronology and Palaeolithic archaeology, which can be as remarkable as the famous as Olduvai Gorge in Tanzania for its importance of the understanding of human evolution (Zhu *et al.*, 2004; Qiu, 2000; Deng *et al.*, 2008).

Magnetic polarity stratigraphy has proved to be useful in establishing chronological control of terrestrial sedimentary sequences in the Nihewan basin. However, in order to strengthen the chronology of early human sites in eastern Old World, the dating of the early and middle Pleistocene fluvial deposits in this basin by other methods, such as $^{26}Al/^{10}Be$ and ESR, remain required. Recently, many successful applications of ESR dating for fluvial sediments were carried out (e.g. Voinchet *et al.*, 2004, 2010; Tissoux *et al.*, 2007; Liu *et al.*, 2010; Moreno *et al.*, 2012), thus offering a good opportunity to date early and middle Pleistocene sediments including early human sites.

The Donggutuo site is one of the early Palaeolithic sites located in the eastern of the Nihewan basin. In this study, we tried to obtain Electron Spin Resonance (ESR) ages for the Donggutuo site using the quartz Ti-Li centre signal to verify the reliability of the ESR dating method for fluvio-lacustrine sediments in the Nihewan basin, which are attributed to the late Early Pleistocene, i.e., older than the Brunhes/Matuyama (B/M) boundary. An independent age obtained for the Donggutuo site should hence contribute to a better understanding of the chronological framework of early human occupation at high northern latitudes in East Asia.

2. GEOLOGICAL AND ARCHAEOLOGICAL SETTINGS AND SAMPLING

Geological setting

The Nihewan Basin, Hebei Province, North China, is a down-faulted basin, located in the transition zone between the North China Plain and the Inner Mongolian Plateau. Nihewan Basin is well known for its late Cenozoic lacustrine and fluvial strata, the so-called Nihewan Beds (Barbour, 1924), which are among the most famous and well-preserved palaeontological Quaternary strata in East Asia.

The Cenjiawan (Cheng-chia-wan) platform in the eastern margin of the Nihewan Basin, predominantly consisting of lacustrine sediments, covers an area of 20 km^2 and has a relative altimetry of 120 m (**Fig. 1**). Its exploration has led to the discovery of numerous palaeontological or archaeological localities, including the Donggutuo site, one of the most famous early Palaeolithic sites in China and one of the most studied localities in the Nihewan Basin (Li and Wang, 1985; Schick *et al.*, 1991; Qiu, 2000; Zhu *et al.*, 2001, 2004; Wang *et al.*, 2005).

A regional chronological framework of the Nihewan basin was built recently using magnetostratigraphy (Zhu *et al*., 2004; Deng *et al*., 2008) and permits to date the geological and archaeological levels, furnishing hence an independent geochronological control to check the reliability of dating methods, such as ESR dating of bleached quartz. A previous ESR dating study in this region indicated that the age of fluvial sediment younger than 780 ka can be determined using the quartz Ti-Li centre ESR signal (Liu *et al*., 2010). It is worth testing the ESR dating method for the sediments being older than 780 ka. In the Nihewan basin, the Donggutuo site seems to be a convenient site for such purpose, because magnetostratigraphic results show that the artifact layer is located below sediments with normal magnetization attributed to Jaramillo subchron (Li and Wang, 1985).

Archaeological setting and sampling

The Donggutuo site (**Fig. 1**) is one of the most extensively excavated localities in the Nihewan Basin (Schick *et al*., 1991). Among the five excavated trenches, Trench 1 (Tr.1) is the largest and the most prolific one (Wei, 1985; Schick *et al*., 1991) and it was chosen as a sampling site for the present study. In this trench, the Donggutuo archaeological layer can be divided into five levels (indexed from the top to the bottom A to E, Xie, 2006) on the basis of the sediment characteristics (**Fig. 2**). Four ESR dating samples were taken on the Donggutuo profile from A, B, C and E layers.

Paleomagnetic studies of the Donggutuo sites

As an extensively excavated site, several paleomagnetic studies were carried out (Zhu *et al*., 2003). The work done by Li and Wang (1985) and supported by Schick and Dong (1993) and by the high resolution paleomagnetic sequence constructed by Wang *et al*. (2005) addressed that the artifact layer is located about 5 m below the Jaramillo normal subchron (0.99-1.07 Ma) as in **Fig. 2**. The age of the Donggutuo site can be definitively estimated to be about 1.1 Ma (Wang *et al*., 2005; Zhu *et al*., 2007).

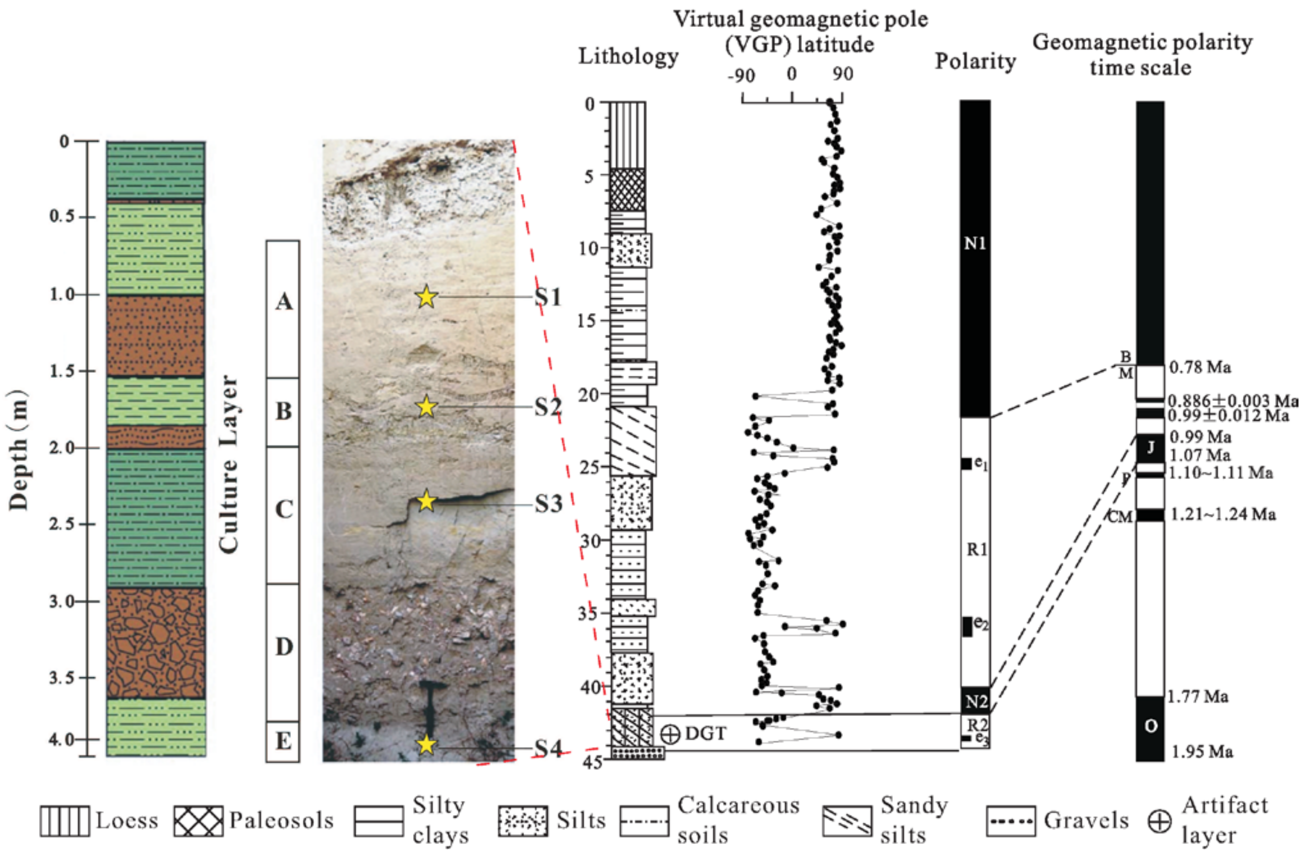

Fig. 2. *Lithostratigraphy, magnetic polarity record and detailed stratigraphic division of the Donggutuo section (as "DGT" in the profile) and its correlations with the geomagnetic polarity timescale (after Wang* et al*., 2005; Han, 2011). A: Grey-yellow silty fine sand with small gravel, stone artifacts and bone fragments 0.50-1.10 m thick; B: Brown fine sandy silt with spots, strips, unearthed stone artifacts and bone fragments 0.40-1.00 m thick; C: Light grey sandy silt with large gravel, stone artifacts and bone fragments, ~0.90 m thick; D: Gravel layer with brown sand and silt, with stone artifacts, ~0.70 m thick; E: Grey sandy silt, contain stone artifacts, ~0.60 m thick.*

3. EXPERIMENTAL METHODS

Quartz extraction and irradiation

Each sediment sample was dried at low temperature. The 105-200 μm grain size fractions were separated by sieving and pure quartz was obtained through chemical separation techniques detailed by Liu *et al.* (2010). After rising with distilled water and drying, the quality of the mineral separation was checked under a microscope. Each sample of pure quartz grains was then divided into 200 mg subsamples and these aliquots have received additional gamma doses ranging from 0 to 10 kGy using the ^{60}Co source of Peking University, Beijing.

Measurement of the dose rate

In this study, we did not perform in-situ measurements of the dose rate (D). The dose rate (D) was calculated from the concentrations of uranium, thorium and potassium of each sample (Aitken, 1998). Uranium and thorium contents were obtained using a thick source Daybreak 530 Model alpha counter. The potassium oxide content was determined by atomic absorption. The water content was estimated as 10 wt % for every sample since they were kept in the dry environment for a long time. Cosmic dose rates were calculated on the burial depth and altitude and latitude of the sample (Prescott and Hutton, 1994).

ESR measurement

The quartz Ti-Li centre ESR intensity was measured at low temperature with a Bruker ER-041-XG X-band spectrometer in a finger dewar cooled to 77 K with liquid nitrogen, in the ESR laboratory of the Institute of Geology, China Earthquake Administration, Beijing, using a microwave power of 5 mW and a modulation amplitude of 0.16 mT. The Ti-Li centre intensity was measured from the top of the peak at g=1.979 (T1) to the bottom at g=1.913 (T2) (Rink *et al.*, 2007; Liu *et al.*, 2010). **Fig. 3** showed the natural S1 sample ESR spectrum at low temperature (liquid nitrogen). The angular dependence of the ESR signal due to the sample heterogeneity was taken into account. Each aliquot was measured six times after a rotation of 60° angle in the cavity to obtain the average intensity.

4. RESULTS AND DISCUSSION

Resetting rate of the ESR centres in quartz extracted from fluvial sediments plays a key role for dating samples. Recently, improvements in the understanding of the bleaching mechanism on the quartz ESR signals resetting, have confirmed the potential of dating bleached quartz by ESR. Sunlight exposure was initially considered as the first driver for quartz bleaching in fluvial sediments and several experiments claimed that the Ti-Li centre can be completely bleached after the equivalent of a few days of sunlight exposure (Toyoda *et al.*, 2000; Tissoux *et al.*, 2007, 2008; Gao *et al.*, 2009) while the Al centre can only be bleached to a stable residual level (varying from one sample to another) after natural sunlight exposure of 2-4 months (Tissoux *et al.*, 2007) or perhaps even 6 months (Voinchet *et al.*, 2003). Fieldwork realized by Voinchet *et al.* (2007) led them to claim that the Ti-Li centre of quartz extracted from river sediments can be completely reset and the Al centre could be reduced to a stable level only after 1 km of transport in natural present-day conditions. The study of the ESR dating from the Dongpo site, another locality of Nihewan Basin (**Fig. 1**, Liu *et al.*, 2010), demonstrated that the Nihewan fluvial sediments sampled near the Brunhes/Matuyama (B/M) boundary were bleached to zero before the last deposition.

Bleaching experiments have shown that the Ti centre can be completely bleached after the equivalent of a few days of sunlight exposure (Toyoda *et al*., 2000; Tissoux *et al*., 2007, 2008; Gao *et al*., 2009). Considering that optical dating of river sediments requires bleaching times in the second to minute range for complete bleaching and as incomplete bleaching is a serious problems in geological context (Olley *et al*., 2004), one would expect that ESR dating results on river sediments could be severely overestimated. Voinchet *et al*. (2007) collected sediment samples of the Creuse River from its spring to 170 km downstream and showed that all modern sediments had completely bleached Ti-Li centres. Recent experiment results showed hence that the fluvio-mechanical action could be another non-negligible bleaching driver, leading to obvious partial resetting with short tumbling times (Liu and Grün, 2011). Therefore, the equivalent dose values (D_e), determined from the quartz Ti-Li centre ESR signals of our four samples collected from the Donggutuo site, were obtained using a complete bleaching assumption.

Single saturating exponential (SSE) function, traditionally used to describe dose response curves of quartz grains (e.g. Liu *et al*., 2010; Voinchet *et al*., 2010), does not fully fit the experimental data points obtained from the Donggutuo site samples, leading to a significant overestimation of the D_e value. Duval *et al*. (2009) had fit the D_e values for Lower Pleistocene teeth samples using a (SSE + LIN) function, because it describes better the

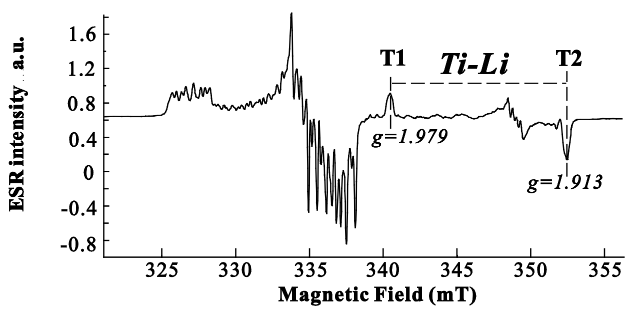

Fig. 3. *Quartz ESR spectrum observed in the natural sample S1 at low temperature using the finger dewar (at liquid nitrogen temperature, 77 k).*

experimental data points:

$$I(D) = I_{sat}(1 - e^{-\mu(D+De)}) + B(D + D_e) \qquad (1)$$

where I is the intensity of the ESR signal of a sample irradiated at the dose D, I_{sat} the saturation intensity, μ the coefficient of sensitivity of the sample and D_e the equivalent dose.

This kind of function was also recently used to describe quartz sample growth curves (Moreno *et al.*, 2012). Therefore, we used a fitting by a linear function coupled with single saturating exponential (LIN + SSE). The growth curve of the sample S3 is shown in **Fig. 4**.

The quartz ESR ages and associated data obtained from the Donggutuo artifact layer samples are shown in **Table 1** and **Fig. 5**.

The ESR ages of the four samples range from 1060±129 to 1171±132 ka, with a mean age of 1119±132 ka (**Fig. 5**). These ESR results are consistent with the lithostratigraphy and previous magnetostratigraphic results, which indicated that the date of all four samples should be older than the Jaramillo normal subchron (0.99-1.07 Ma) (Wang *et al.*, 2005; Li *et al.*, 2008). These ESR results indicate then that the Ti-Li centre in quartz can be used to set up the time scale for Lower Pleistocene samples (i.e., older than the B/M boundary). The new results from the Donggutuo site extend hence the reliable dating range of ESR method and seem to confirm that the ESR signal of the Ti-Li centre in quartz has been initially zeroed before the sediments deposition.

5. CONCLUSION

Four sediment samples collected in the Donggutuo site below the Jaramillo normal subchron record were dated by ESR dating method on bleached quartz. The ESR dates obtained using Ti-Li centre range from 1060±129 ka to 1171±132 ka and are consistent with geomagnetic polarity study of this profile. The range of the ESR dating by Ti-Li centre in quartz was efficiently extended. This study provides another proof of the probable initial zeroing of the Ti-Li centre signal in the fluvial sediments before deposition and encourages us to improve the understanding of the resetting mechanism in fluvial system.

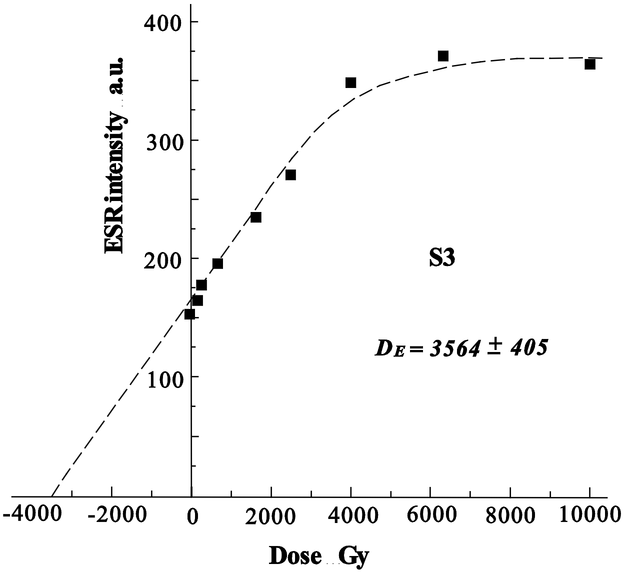

Fig. 4. *Growth curve of sample S3 obtained from the quartz ESR Ti-Li centre signal.*

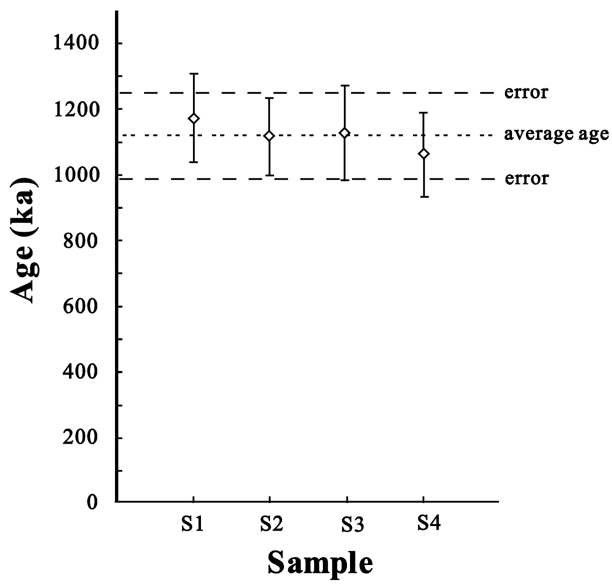

Fig. 5. *Quartz Ti-Li centre ESR ages of the Donggutuo artifact layer, Nihewan Basin, China.*

Table 1. *ESR ages and associated data obtained from the Donggutuo samples, Nihewan Basin, China.*

Sample No.	Level	Depth (m)	Water (%)	U and Th (counts/ks)	K_2O (%)	Cosmic dose rate (Gy/ka)	D (Gy/ka)	D_E (Gy)	ESR age (ka)
S1	A	1.0	10±5	6.02±0.30	2.20±0.11	0.21±0.01	2.763±0.164	3224±307	1171±132
S2	B	1.7	10±5	7.79±0.39	2.35±0.12	0.20±0.01	3.103±0.186	3452±307	1116±120
S3	C	2.4	10±5	7.85±0.40	2.45±0.13	0.18±0.01	3.174±0.195	3564±405	1127±146
S4	E	4.1	10±5	9.84±0.49	2.50±0.13	0.14±0.01	3.432±0.210	3624±380	1060±129

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