



OSL DATING USING QUARTZ FINE GRAINS EXTRACTED FROM LOESS IN UPPER PALAEOOLITHIC SITES OF NIHEWAN BASIN, NORTHERN CHINA

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Abstract: Although radiocarbon (¹⁴C) dating, uranium-series dating, and optically stimulated luminescence (OSL) dating have been conducted for Upper Palaeolithic sites in the Nihewan Basin, northern China, there is room for constructing a detailed chronological framework. In this study, loess sediments collected from two Upper Palaeolithic sites, Youfang site and Hutouliang site, were dated using the single-aliquot regenerative-dose (SAR) OSL protocol. OSL measurements for palaeodoses estimation used fine-grained quartz samples extracted from loess. OSL dating results were obtained as 10-17 ka. These OSL ages were consistent with the related stratigraphy of Palaeolithic sites, archaeological evidence and independent ¹⁴C ages.

Keywords: OSL dating, Upper Palaeolithic sites, Loess, Nihewan Basin.

1. INTRODUCTION

Optically stimulated luminescence (OSL) dating using fine-grained quartz is widely accepted as a reliable tool for use in dating of loess sediments (Watanuki *et al.*, 2003; 2005; Narama *et al.*, 2007; Timar *et al.*, 2010). The chronology of Chinese loess using OSL dating methods has been ascertained (Wang *et al.*, 2006; Lai, 2010).

In this study, OSL dating was performed on loess from Palaeolithic sites of the Nihewan Basin in northern China. The basin is situated 150 km west of Beijing, covering a wide area of the northwestern part of the Hebei Province and the northeastern part of the Shanxi Province (Fig. 1) (Wei *et al.*, 2011). Many Palaeolithic sites have been discovered in the Nihewan Basin, yielding important information related to archaeology and anthro-

pology. Most geochronological studies relevant to the Lower Palaeolithic sites discovered in the Nihewan Basin employed uranium (U) series dating (Chen *et al.*, 1984), electron spin resonance (ESR) dating (Liu *et al.*, 2010a), geomagnetostratigraphic dating, and sedimentology (Zhu *et al.*, 2001; 2004; Liu *et al.*, 2010b; Zuo *et al.*, 2011). The established chronological framework of the Lower Palaeolithic has elucidated the importance of early human evidence in the Nihewan Basin. Nevertheless, few attempts have been made to construct a chronological framework for the Upper Palaeolithic sites in the Nihewan Basin. In the Upper Palaeolithic sites, U-series ages have been obtained for the Xibaimaying site (Xie and Yu, 1989). In addition, ¹⁴C ages have been obtained for the Shenquansi site, Nanmo site, Erdaoliang site (Wei *et al.*, 2011), and Hutouliang site (Gai, 1991). Nagatomo *et al.* (2009) and Zhao *et al.* (2010) recently conducted OSL dating in this area. However, datasets for a chronological

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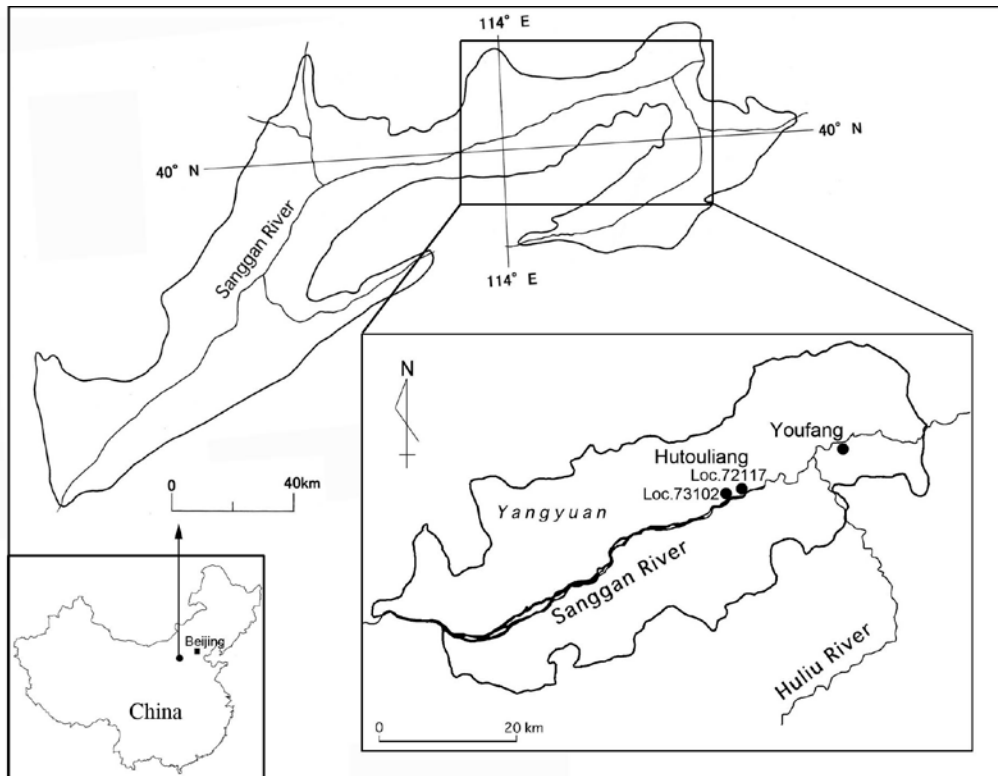


Fig. 1. Location map showing the Nihewan Basin and the Palaeolithic sites described in this paper.

framework of the Upper Palaeolithic sites in the Nihewan Basin are insufficient. Age determination demands accuracy for construction of a chronological framework. Data of Nagatomo *et al.* (2009) are of especially insufficient accuracy for IRSL age, without fading correction using the multiple aliquot protocol. OSL dating using the single-aliquot regenerative-dose (SAR) protocol (Murray and Wintle, 2000) was used for this study. The SAR protocol using sedimentary quartz is sufficiently accurate for sensitivity correction and estimation of the spread of the palaeodose. The ages obtained as results of this study can be expected to contribute to determination of the detailed chronological framework of the Upper Palaeolithic sites in the Nihewan Basin as key sites exhibiting evidence of microblade technique development.

2. SAMPLE DETAILS

The Nihewan Basin, located in the Sanggan River drainage, is filled with Pliocene to Holocene lacustrine, fluvial, and aeolian loess deposits (Wei, 1994). The cultural horizons of the Upper Palaeolithic sites, discovered in the Nihewan Basin, are formed mainly within the aeolian loess deposits (Xie, 2006; Wei *et al.*, 2011).

All samples for OSL dating were collected from loess sediments associated with two Upper Palaeolithic sites, namely Youfang (Xie and Cheng, 1989) and Hutouliang (Gai and Wei, 1977), situated along the eastern margin of

the Nihewan Basin (Fig. 1). At the Youfang site, two samples were collected from above (sample YF_No. 1) and below (sample YF_No. 2) (Fig. 2a). Two samples (Fig. 2b) of Loc.73102 were ca. 1.4 m (73102_No. 1) and 2.1 m (73102_No. 2), both of which are from above the cultural layer. Two samples (Fig. 2c) of Loc.72117 were taken. Sample 72117_No. 1 was taken from above the cultural layer. Sample 72117_No. 2 was from the cultural layer.

3. OSL DATING

Sample preparation

Minerals in each collected sample were separated into coarse and fine grains (FG) by wet sieving in water. After fine silt (approx. 4-10 μm diameter) was separated by Stokes' law of settling in acetone from FG (Zimmerman, 1971), it was treated with 10% hydrogen peroxide for 16 hr and with 10% hydrochloric acid for 90 min. To separate the fine-grained quartz, the fine silt samples were immersed in 20% hydrofluorosilicic acid (H_2SiF_6) for 9 days. The quartz extract purity was verified using infrared stimulation. We took sample grains showing less than 10% IRSL compared with blue-stimulated ones. If an infrared signal was measured, indicating the presence of feldspar, then the H_2SiF_6 treatment was repeated.

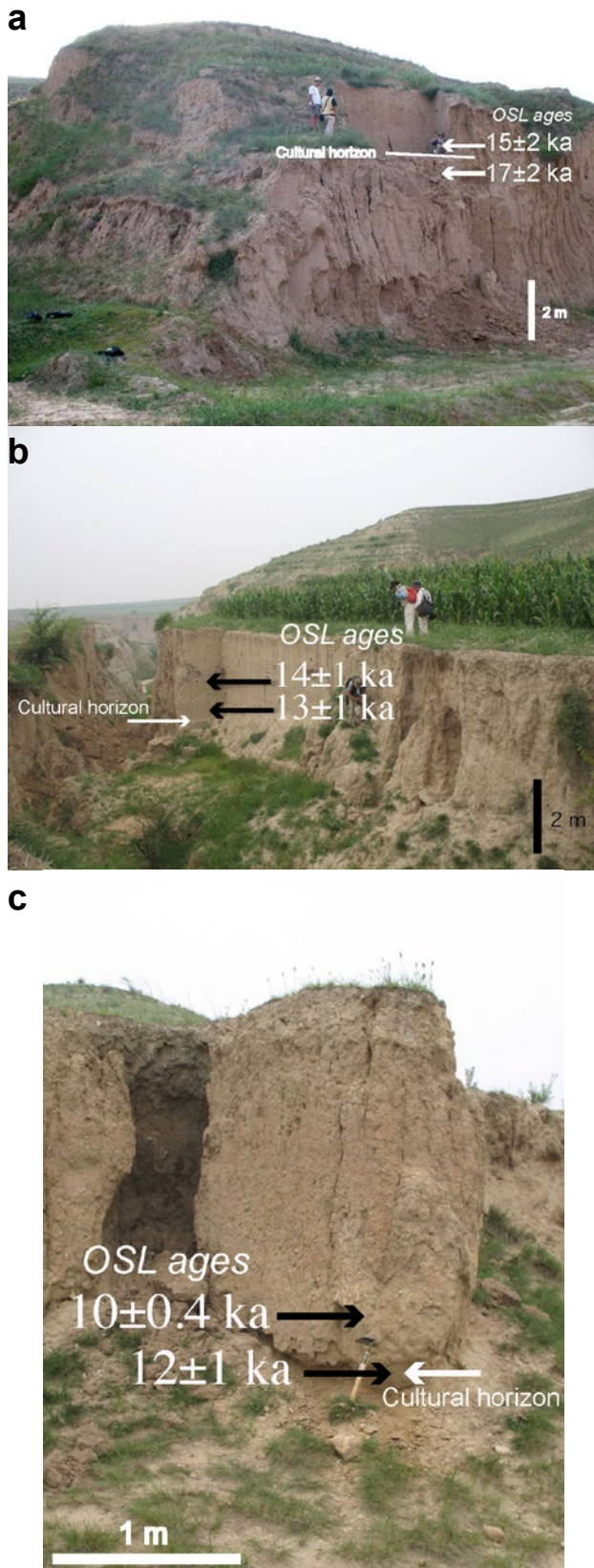


Fig. 2. Photographs of a representative section of each of the Palaeolithic sites: a) Youfang site; b) Loc.73102 of the Hutouliang site; c) Loc.72117 of the Hutouliang site.

Instrumentation and Palaeodose estimation

All measurements of fine-grained quartz OSL were performed using an OSL/TL reader (NRL-99-OSTL) (Nagatomo *et al.*, 2007; 2009) equipped with an array of 32 blue LEDs (470 nm) or 32 infrared LEDs (890 nm). The diameter of each sample disc was 10 mm. An aliquot was 2 mg. The OSL from a sample was detected using a photomultiplier tube (R1140P; Hamamatsu Photonics K.K.) housed in a cooling unit, through two condensing lenses and a glass filter (2 mm, Hoya U-340; Kenko Tokina Corp.). Irradiation was conducted using a Risø $^{90}\text{Sr}/^{90}\text{Y}$ beta source (dose rate 0.07 Gy/s) built into the OSL reader.

Dose response curves from which palaeodoses could be estimated were constructed using the SAR shown in **Table 1**. To select appropriate preheating conditions using the SAR protocol, a preheat temperature plateau and dose recovery test was conducted. Preheat temperatures between 200 and 300°C were used for 60 s at intervals of 20°C. The cut-heat was 160°C.

Annual dose estimation

Annual doses (dose rate) were measured using a high-resolution gamma-ray spectrometer. The concentrations of ^{238}U , ^{232}Th , and ^{40}K in the samples were analyzed using a Ge gamma-ray detector (EGSP 8785; Eurisys Mesures). The annual dose was calculated using the dose-rate conversion factors reported by Adamiec and Aitken (1998). An alpha efficiency of 0.1 was used, as suggested by Aitken (1998). Present water contents of each sample were used. Contributions of the cosmic dose rate to the annual dose were assumed according to methods proposed in previous reports by Prescott and Hutton (1994), Nagatomo *et al.* (2009), and Shitaoka *et al.* (2012).

4. RESULTS AND DISCUSSION

A preheat plateau for sample 72117_No. 1 is shown in **Fig. 3**. A plateau was obtained across the entire preheat range from 200 to 300°C. Therefore, we selected a preheat temperature of 220°C or 240°C for routine palaeodose determination in this study. A recuperation test (Wintle and Murray, 2006) was performed for each sample. Recuperation was <5% for all samples. The meas-

Table 1. SAR protocol used for this study

Step	Treatment	Observed
1 ^(a)	Give dose, D_i	-
2	Preheat (220-240°C for 60 s)	-
3	Stimulate for 100 s at 120°C	L_i
4	Give test dose, 3 Gy or 5 Gy	-
5	Cut heat (160°C)	-
6	Stimulate for 100 s at 120°C	T_i
7	Return to 1	-

^(a)Step1: For the natural sample, $i = 0$ and $D_0 = 0$ Gy.

ured doses in the dose recovery test ($n=3$ for each sample) were within $\pm 10\%$ of the administered dose. The result of the dose recovery test for sample 72117_No. 1 is portrayed in Fig. 4. The result suggests that the SAR procedure can recover a laboratory dose.

Each sensitivity-corrected OSL for doses was calculated by dividing L_i by the respective T_i (i.e., L_i / T_i). The SAR protocol consists of a series of regenerative and test-dose measurements to create a sensitivity-corrected growth curve onto which the sensitivity-corrected natural OSL signal ($L_{\text{natural}} / T_{\text{natural}}$) is projected. The palaeodose value is obtained from the point of intercept, which is then interpolated onto the dose axis. The value of palaeodose and its error was estimated using a Monte Carlo method (Duller, 2007). For most aliquots, recycling ratios fell within the range of 0.9-1.1. A few aliquots for each sample with a recycling ratio falling outside this range were rejected. Palaeodoses of each sample calculated using the central age model (Galbraith *et al.*, 1999). Radial plots (Vermeesch, 2009) for all samples are presented in Fig. 5. The overdispersion values of YF_No.1 and YF_No.2 were, respectively, 17% and 12%. Hutouliang samples obtained overdispersion values of 10-15%. Following Olley *et al.* (2004), a sedimentary sample with overdispersion of less than 20% was fully bleached. It is assumed that Youfang and Hutouliang samples have been well-bleached sediments.

All OSL ages are presented in Table 2. The OSL ages of the sites are consistent with their respective stratigraphic positions (Fig. 2). For the samples from Youfang site, OSL ages support the archaeological evidence, which is represented by microblade technology that appeared earlier than industry in Hutouliang (Xie and Cheng, 1989). The OSL ages obtained for Hutouliang Loc.73102 were consistent with the calibrated ^{14}C ages of 16,310-15,280 cal BP (13,080 \pm 120 BP; Xie (2006)).

5. CONCLUSION

OSL dating was performed on fine-grained quartz extracted from loess at Palaeolithic sites in the Nihewan Basin of northern China. The OSL-estimated ages give ages of 15-17 ka for the Youfang site and give ages of

10-14 ka for the Hutouliang site. These OSL ages are stratigraphically coherent. Additional dating studies of Upper Palaeolithic sites in the Nihewan Basin would help to further clarify the chronological framework of the Upper Palaeolithic in this area.

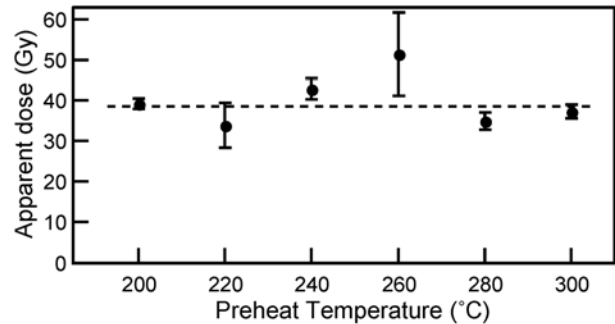


Fig. 3. Preheat temperature plateau dataset of sample 72117_No. 1. Each point represents the average of three aliquots at each temperature.

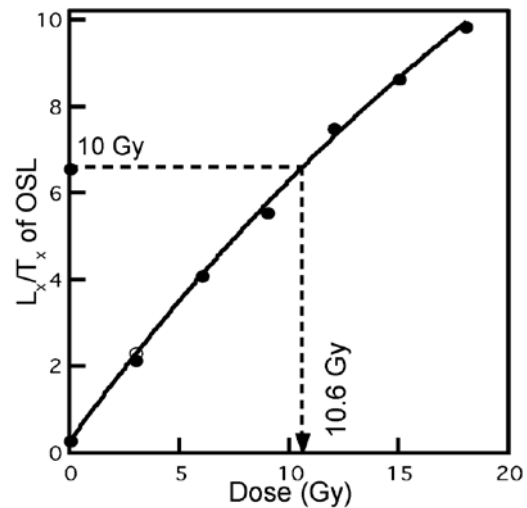


Fig. 4. Dose recovery test of sample 72117_No. 1. The administered dose was 10 Gy. The measured dose was 10.6 Gy.

Table 2. Palaeodose, annual dose and OSL ages for all samples measured in this study.

Site	Sample No.	Palaeo-dose (Gy)	Aliquots (n)	U (ppm)	Th (ppm)	^{40}K (wt%)	Water content (%)	Cosmic dose (mGy/a)	Annual dose (mGy/a)	OSL age (ka)
Youfang	1	68 \pm 4	7/8	2.07 \pm 0.12	10.63 \pm 0.44	1.96 \pm 0.36	1	0.15	4.60 \pm 0.46	15 \pm 2
	2	87 \pm 4	8/11	2.61 \pm 0.15	10.48 \pm 0.48	2.18 \pm 0.42	1	0.15	5.05 \pm 0.55	17 \pm 2
Hutouliang Loc.73102	1	61 \pm 2	14/16	2.90 \pm 0.16	10.03 \pm 0.43	1.65 \pm 0.10	4	0.15	4.42 \pm 0.10	14 \pm 1
	2	65 \pm 2	15/19	3.00 \pm 0.17	10.45 \pm 0.48	2.07 \pm 0.12	2	0.15	5.02 \pm 0.13	13 \pm 1
Hutouliang Loc.72117	1	40 \pm 1	15/15	2.64 \pm 0.15	8.14 \pm 0.43	1.44 \pm 0.10	1	0.15	3.96 \pm 0.15	10 \pm 0.4
	2	63 \pm 2	15/19	3.34 \pm 0.18	9.56 \pm 0.51	2.00 \pm 0.12	1	0.15	5.04 \pm 0.16	12 \pm 1

n is the number of individual aliquots contributing to the Palaeodose estimation against the number of aliquots measured.

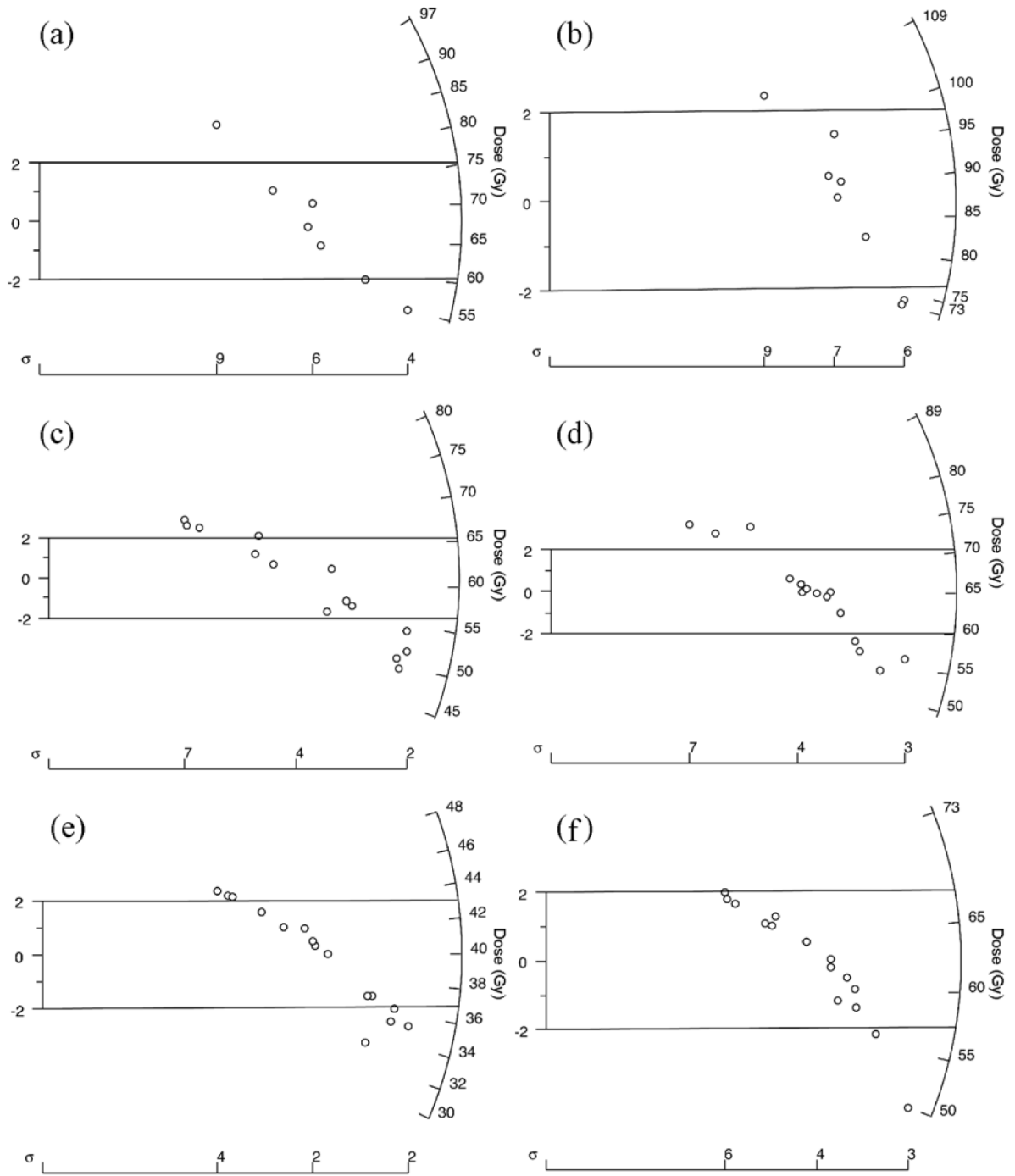


Fig. 5. Dose distributions for all samples are shown using the radial plots: a) YF_No. 1; b) YF_No. 2; c) 73102_No. 1; d) 73102_No. 2; e) 72117_No. 1; f) 72117_No. 2.

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