# Electron Spin Resonance Studies of Gammairradiated Tertiary Strata in the Huai Luang Formation in the Mae Moh Basin, Northern Thailand

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## ABSTRACT

Electron spin resonance studies were done on bedding plane sediment samples from the Middle Miocene Huai Luang Formation in the Mae Moh Basin in northern Thailand. Electron spin resonance spectra data that were recorded included those of $Fe^{3+}$, $Mn^{2+}$, and $E_1'$ center in iron oxide and quartz. The radicals produced by gamma irradiation in these sediment samples, whose $g$ factors were near $g \cong 2$, are attributed to $E_1'$ center. The electron spin resonance signal intensity of these radicals gradually increased as the Gy dose increased. Electron spin resonance analyses produce reliable results that can be used for age dating rocks. The results from the Huai Luang sediment samples can be used as reference data.

**KEYWORDS:** Electron spin resonance, Huai Luang Formation, Mae Moh Basin

## INTRODUCTION

During the Early Tertiary, the crust of northern Thailand was subjected to high tension that caused folding, faulting, and warping in many places (Griscom and Beltrán-López, 2002). Most of the Tertiary basins that originated during this time were the result of extensional rifting related to the collision of the Indian and Asian plates. In Thailand, there are 61 Cenozoic basins in both onshore and offshore areas. Those that are in northern Thailand have lignite and oil shale deposits. One of these, the Mae Moh Basin, has the largest lignite deposits and is the biggest coal mine in Thailand (Songthama and others, 2005). The Mae Moh Basin has become the key to biostratigraphic correlation in Thailand. Considering the philosophy of Ikeya (Ikeya, 1993), electron spin resonance was investigated for being able to provide additional geological insights not apparent from the more traditional methods of geology.

Electron spin resonance occurs whenever a system, such as an atom, a molecule, an ion, or a defect and impurities in a solid, possesses unpaired electrons. In many cases this condition is brought about by irradiation of a solid with energetic radiation, such as ultraviolet, x-ray, or nuclear radiation. Nearly all substances, if irradiated long enough, will show a resonance spectrum. The first successful experiment to observe electron spin resonance was done by Zavoisky (1945). Since then, the electron spin resonance technique has found a wide variety of applications in chemistry, physics, and biology. It is also used in process control and clinical analysis. Zeller and others (1967), Zeller (1968), and Levy (1968) suggested that electron spin resonance could also be used in geology and archaeology. (Engin and others, 2006).

This paper presents the electron spin resonance spectra results of $Fe^{3+}$, $Mn^{2+}$, and $E_1'$ center in iron oxide and quartz from some Middle Miocene claystone deposits of the Mae Moh Basin. These results were compared to radicals produced by gamma irradiation in sediments whose *g* factors are near $g \cong 2$. This study has great importance since it is the first one being used as reference data for electron spin resonance dating analysis for the additive irradiation method for Middle Miocene rocks.

## GEOLOGY

The Mae Moh Basin is situated in Mae Moh District of Lampang Province and is about 26 kilometers east of Lampang city. It has is an area of approximately 135 square kilometers, being 7 kilometers east-west and 16 kilometers north-south (Figure 1). The basin floor is 320 to 340 meters above mean sea level. The basin is an intermountain graben basin and is filled with Tertiary and Quaternary strata. These sedimentary beds include the Mae Moh Group (Songthama and others, 2005).


**Figure 1.** Map of northern Thailand showing location of the Mae Moh Basin.

The Tertiary strata in the Mae Moh Basin have been named the Mae Moh Group . This group has three formations, the Huai King, Na Khaem, and Huai Luang. The combined thickness of these formations is nearly 1,000 meters (Figure 2). However, this paper only concerns sediment samples from a bedding plane of the Middle Miocene Huai Luang Formation (Ratanasthien, 2002). The Huai Luang Formation is composed of claystone and siltstone and has some sandstone and conglomerate lenses. Its characteristic color is red to brownish red, though there are some gray interbeds. Gypsum and selenite are abundant in this formation. The I-coal zone is in the middle of the formation (Songthama and others, 2005).

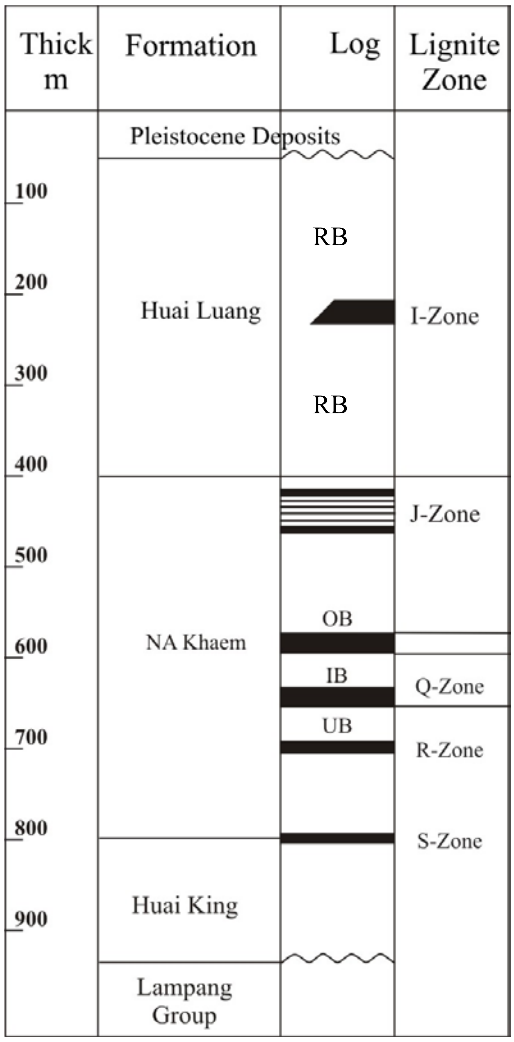


**Figure 2.** Schematic lithostratigraphic units of the Mae Moh Group.

## SAMPLES AND EXPERIMENTAL DETAIL EVALUATION

The Huai Luang samples collected from the Mae Moh Basin were washed and cleaned in an ultrasonic bath using distilled water. After that, the samples were gently ground with a mortar and the resultant grains were separated into several sizes by sieving. Grain sizes of 75 to 250 micrometers were retained. These retained grains were etched by 5 percent hydrochloric acid for 1 day and then rinsed with water. Quartz grains were separated using 20 percent hydrofluoric acid for 2 hours and were then rinsed with water. These grains were washed repeatedly in distilled water and then dried at a temperature of 40°C. Following this, the samples were divided into two parts, half for electron spin resonance spectra and half for x-ray diffraction analysis. The samples that were prepared for electron spin resonance spectra procedures were performed in dim red light.

The samples for x-ray diffraction analysis were characterized by an x-ray powder diffractometer to identify the phases. These analyses were done on powdered samples using a Philips diffractometer that used monochromatized $CuK_{\alpha}$ radiation. The x-ray tube was operated at 30 kilovolts and 25 milliamperes. The samples were scanned in the $2\theta$ range of 10° to 80° with a scanning speed of 2° per minute.

The samples for electron spin resonance analysis were divided into six aliquots, approximately 300 to 500 milligrams each. Three aliquots were irradiated for electron spin resonance measurements. The other aliquot were not irradiated. Artificial gamma irradiations were made using a cobalt-60 source (GC-220E), which delivered 3.404 gray per second, at the cobalt 60 gamma ray irradiation laboratory of the Office of Atoms for Peace. Artificial dosages of gray that were used were levels of 20, 50, 70, 100, 120, 150, 200, 300, and 500 gray. The sediment powders were packed in cylindrical containers for irradiation and then transferred to tubes for electron spin resonance measurements. These samples weighed 300 to 500 milligrams. The electron spin resonance spectra were obtained using a Bruker spectrometer operating in the X band (TE011 mode) microwave range. The spectrometer operating conditions used during the experiment were: 349 millitesla central magnetic field; 350, 100, and 10 millitesla scan ranges; 0.1 millitesla field modulation amplitude; 100 kilohertz modulation frequency; 0.63 milliwatts microwave power; 5.12 millisecond conversion time; and 163.84 millisecond time constant. DPPH with a g-value of 2.0036 was used as an internal standard for g factor calculations. All data points are the average of at least three different aliquots of the samples.

## RESULT AND DISCUSSION

The electron spin resonance signals of the Huai Luang Formation red bed layer in Figures 3 and 4 correspond with $Fe^{3+}$ at $g$ = 4.3 (Ikeya, 1993) and sextet hyperfine signals of $Mn^{2+}$ organic radicals around $g$ = 2.0, respectively. In Figure 5, electron spin resonance signals at $g$ = 2.0018 and $g$ = 2.0003 correspond to $E_1'$ center (Zeller, 1968; Levy, 1968; Taylor and Martin, 1997) in the sedimentary layer.

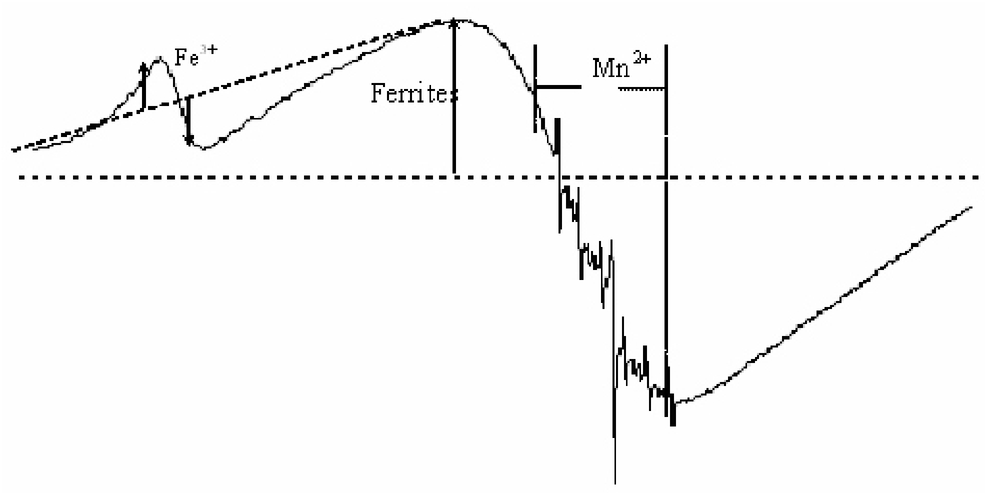


**Figure 3**. Red bed signals of $Fe^{3+}$ at $g$ = 4.3 and the sextet hyperfine of $Mn^{2+}$; scan range is 350 mT.

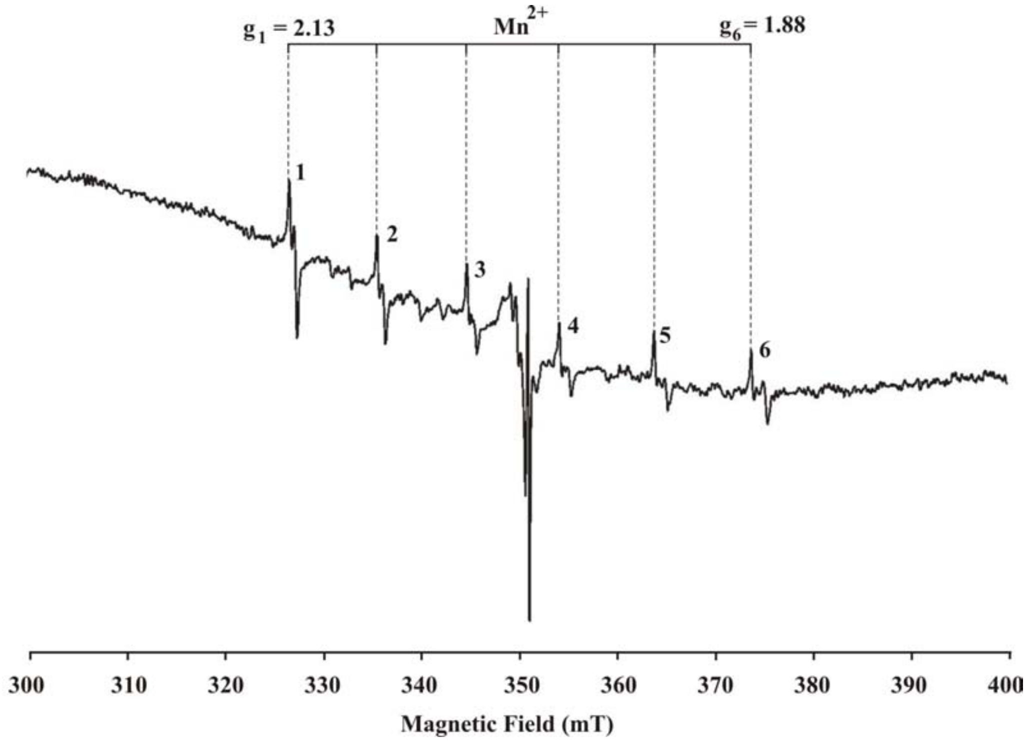


**Figure 4**. Red bed sextet hyperfine signals of $Mn^{2+}$ organic radicals around $g$ = 2.00; scan range is 100 mT.

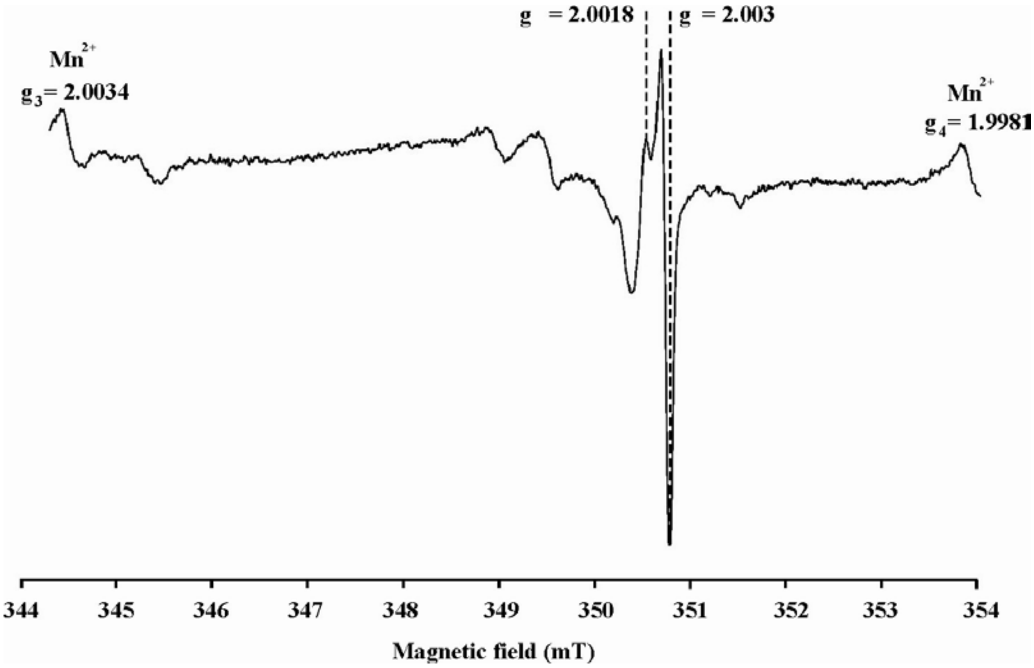


**Figure 5.** Electron spin resonance spectra of the $E_1'$ center at $g$ = 2.0018 and $g$ = 2.0003; scan range is 10 mT.

The electron spin resonance signal in Figure 4 shows a clear sextet hyperfine of $Mn^{2+}$. This signal occurs mostly in calcium carbonate material and, thus, indicates that the red bed layer has some calcium carbonate. The electron spin resonance spectrum of the red bed layer in Figure 5 is associated with $E_1'$ center at $g$ = 2.0018 and $g$ = 2.0003. This indicates that the red bed layer contains iron oxide and quartz. Confirmation of this is the x-ray diffraction results shown in Figure 6. Figure 7 shows electron spin resonance spectra of a natural sample γ-irradiated with a dose of 500 gray. The $E_1'$ center of this sample increases with increased γ-radiation. The increase with γ-radiation of electron spin resonance signals intensity of $E_1'$ center can be used to determine the age of a sample. Also, most research work on electron spin resonance dating in quartz uses $E_1'$ center to determine age.

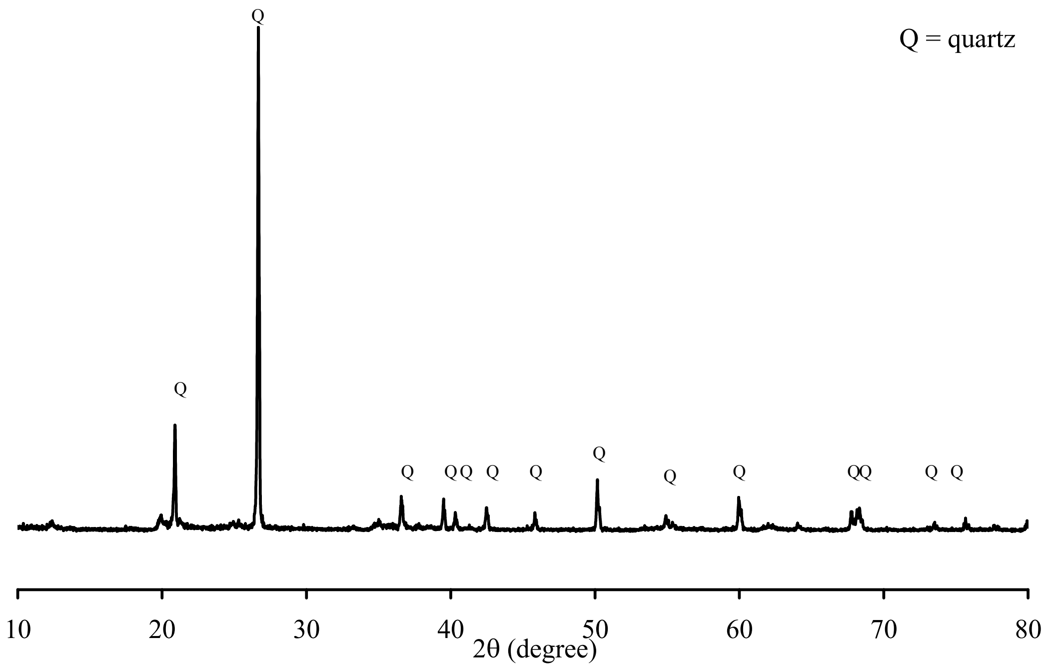


**Figure 6.** X-ray powder diffraction patterns of red bed layer sediment.

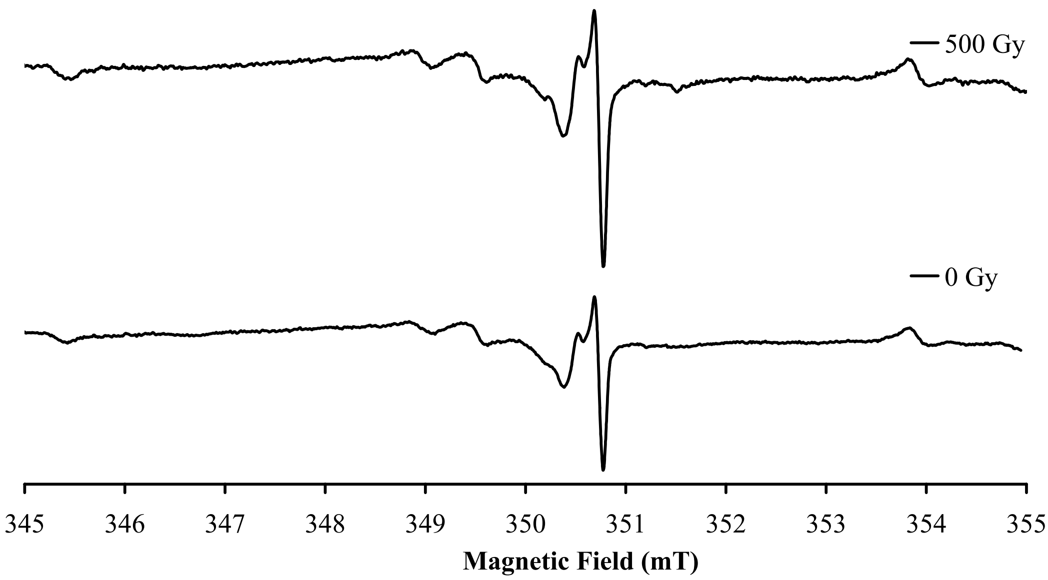


**Figure 7.** Electron spin resonance spectra comparison between a natural sample and a 500-Gy . γ-irradiated sample.

## CONCLUSION

Electron spin resonance signals of gammairradiated red bed sediment samples from the Huai Luang Formation are associated with quartz and calcium carbonate and can indicate the signal of $E_1'$ center and $Mn^{2+}$. When known, the $E_1'$ center signal can be used to determine age because electron spin resonance signals of this center increase with increasing irradiation. This research and analyses have proven the reliability and dependability of electron spin resonance dating and are important for use as reference data for electron spin resonance dating analysis of Middle Miocene rocks (Ratanasthien, 2002).

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