

# Evaluation of lead isotope compositions of NIST NBS 981 measured by thermal ionization mass spectrometer and multiple-collector inductively coupled plasma mass spectrometer

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Received 21 December 2015; revised 8 April 2016; accepted 12 April 2016

Available online 6 September 2016

## Abstract

Because Pb isotopes can be used for tracing, they are widely used in many disciplines. The detection and analysis of Pb isotopes of bulk samples are usually conducted using thermal ionization mass spectrometer (TIMS) and multiple-collector inductively coupled plasma mass spectrometer (MC-ICP-MS), both of which need external reference materials with known isotopic compositions to correct for the mass discrimination effect produced during analysis. NIST NBS 981 is the most widely used reference material for Pb isotope analysis; however, the isotopic compositions reported by various analytical laboratories, especially those using TIMS, vary from each other. In this study, we statistically evaluated 229 reported TIMS analysis values collected by GeoReM in the last 30 years, 176 reported MC-ICP-MS analysis values, and 938 MC-ICP-MS analysis results from our laboratory in the last five years. After careful investigation, only 40 TIMS results were found to have double or triple spikes. The ratios of the overall weighted averages,  $^{206}\text{Pb}/^{204}\text{Pb}$ ,  $^{207}\text{Pb}/^{204}\text{Pb}$ , and  $^{208}\text{Pb}/^{204}\text{Pb}$ , obtained from 40 spiked TIMS reports and 1114 MC-ICP-MS results of NIST NBS 981 isotopes were  $16.9406 \pm 0.0003$  (2s),  $15.4957 \pm 0.0002$  (2s), and  $36.7184 \pm 0.0007$  (2s), respectively.

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**Keywords:** NIST NBS 981; Lead isotope; Thermal ionization mass spectrometer; Multiple-collector inductively coupled plasma mass spectrometer; GeoReM

## 1. Introduction

Because of the unique geochemical properties of Pb, its isotopes can be used for various purposes such as tracing of different magmatic processes, ore-forming material sources, raw material sources of bronzeware and chinaware, and biological processes. Thus, Pb isotopes are widely applied in different fields, including earth sciences, archaeological science, material science, biological science, chemistry, and environmental science (Dreyfus et al., 2007; Kate Souders and Sylvester, 2008; Schultheis et al., 2004; Sjastad et al., 2011; Teresa de la Cruz et al., 2009). Accurate analysis of Pb isotopes is the prerequisite for these studies.

*Abbreviations:* ESA, electrostatic analyzer; ETV, electrothermal vaporization; IC, ion-counting multiplier; LA, laser ablation; MC-ICP-MS, multiple-collector inductively coupled plasma mass spectrometer; TIMS, thermal ionization mass spectrometer; PFA, perfluoroalkoxy.

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Peer review under responsibility of Guangzhou Institute of Geochemistry

<http://dx.doi.org/10.1016/j.sesci.2016.04.001>

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Currently, there are mainly three methods for Pb isotope analysis: secondary ionization mass spectrometer (SIMS), thermal ionization mass spectrometer (TIMS) and multiple-collector inductively coupled plasma mass spectrometer (MC-ICP-MS) (Baker et al., 2004; Belshaw et al., 1998; Chen et al., 2014; Hirata, 1996; Ortega et al., 2012; Pomiès et al., 1998; Taylor et al., 2015; Whitehouse et al., 2005). The SIMS technique needed solid state reference materials for external calibration since it is an in situ analytical technique, and the Pb isotopic compositions of the solid state reference materials was normally ascertained by TIMS or MC-ICP-MS techniques. TIMS uses a thermal ionization ion source, has a stable ion production rate, and can achieve highly accurate Pb isotope composition when a double or triple spike is added, making it the preferred method for the certification of standard samples (Galer, 1999; Powell et al., 1998; Ruiz Encinar et al., 2001; Taylor et al., 2015). MC-ICP-MS uses ICP as the ion source and an interface composed of a sampler cone and skimmer cone. Both ICP and interface had a kinetic energy spread which led to a defocusing of the ion beam (Becker, 2007). Although the subsequent electrostatic analyzer (ESA) can compensate for the kinetic energy spread, the final Pb isotope accuracy is usually poorer than TIMS. However, the MC-ICP-MS has a simple sample preparation process, provides fast analysis speeds, and can be easily connected with third-party sample injection systems (such as desolvator, electrothermal vaporization (ETV), and laser ablation (LA)) to increase sensitivity and achieve in situ microanalysis. Additionally, the accuracy of the analysis meets the requirements of modern studies; therefore, MC-ICP-MS is commonly used in mainstream Pb isotope analysis. Unlike TIMS that corrects quality discrimination of MS by adding spikes, MC-ICP-MS uses an external reference (NIST NBS 981 is commonly used) or a combination of external reference and internal isotope pairs ( $^{203}\text{Tl}$  and  $^{205}\text{Tl}$  are commonly used because their mass numbers are close to that of Pb) to correct the mass discrimination of MS, so as to achieve an accuracy of analysis that is approaching TIMS measurement using double or triple spikes (Taylor et al., 2015).

Many laboratories have analyzed and reported the Pb isotopic composition of NIST NBS 981. We compared all the reported data included in the GeoReM database (<http://georem.mpch-mainz.gwdg.de/>). A database for all kinds of reference materials and isotopic standards of geochemical and mineralogical interest up to May 2015 (Jochum et al., 2005), and found that the NIST NBS 981 ratios used by different laboratories as the external reference are not the same and that there are significant system deviations in TIMS analysis results. Since different laboratories use NIST NBS 981 as the external reference to obtain Pb isotopic compositions of their real samples, any difference in this reference value will directly affect the analytical accuracy of the real samples. Therefore, it is necessary to certify the NIST NBS 981 ratios, so that the Pb isotope ratios analyzed by different laboratories are comparable. Although some researchers have discussed Pb isotope ratios of NIST NBS 981, none of them has systematically analyzed the published reference ratios (Baker et al.,

2004; Collerson et al., 2002; De Muynck et al., 2008; Sarkar et al., 2015; Taylor et al., 2015; Todt et al., 1996).

In this study, Pb isotopic composition of NIST NBS 981 was statistically evaluated based on the composition obtained through MC-ICP-MS analyses at the State Key Laboratory of Continental Dynamics in the last five years, and by using previously reported TIMS and MC-ICP-MS results.

## 2. Experimental

### 2.1. Reagents and materials

Nitric acid ( $\text{HNO}_3$ ) used in this study was purified twice through DST-1000 sub-boiling stills (Savillex Corporation, Eden Prairie, MN USA). The ultrapure water (electrical conductivity  $> 18.2 \text{ M}\Omega \text{ cm}^{-1}$ ) used in the experiment was produced by using a water purifier (Millipore, USA) and was further purified by using a quartz sub-boiling distiller. The Pb background concentration was  $< 200 \text{ pg g}^{-1}$ . Both NIST NBS 981 (Pb) and NIST SRM 997 (Tl) were purchased from the National Institute of Standards and Technology (NIST).

### 2.2. Instrumentation

All analyses were conducted at the State Key Laboratory of Continental Dynamics at Northwest University. Two MC-ICP-MSs (Nu Plasma I (NP-I) and Nu Plasma II (NP-II)) from Nu Instruments (Wrexham, UK) were used. NP-I has 12 F cups (FC) and 3 full-sized discrete dynode ion-counting multipliers (IC), while NP-II has 16 FCs and 5 ICs.  $^{204}\text{Pb}$ ,  $^{206}\text{Pb}$ ,  $^{207}\text{Pb}$ ,  $^{208}\text{Pb}$ ,  $^{202}\text{Hg}$ ,  $^{203}\text{Tl}$ , and  $^{205}\text{Tl}$  isotopes were analyzed in static state using the FCs.  $^{202}\text{Hg}$  was used to monitor the Hg level in the gas background, and to correct for the isobaric interference on  $^{204}\text{Pb}$  according to the natural abundance ratio. The resistances of the pre-amplifiers were  $10^{11} \Omega$ .  $^{203}\text{Tl}$  and  $^{205}\text{Tl}$  were used to correct for the mass discrimination of Pb and Hg isotopes using exponential law ( $^{205}\text{Tl}/^{203}\text{Tl} = 2.3889$ ,  $^{204}\text{Hg}/^{202}\text{Hg} = 0.229883$ ) (Chen et al., 2014; Yuan et al., 2013). NIST NBS 981 (Pb) and NIST SRM 997 (Tl) were mixed at a Pb:Tl ratio of  $\sim 1$ , and the mixture was then injected using two desolvators (DSN-100 for Nu Plasma I; Aridus II for Nu Plasma II). The nebulizer used was the PFA micro-flow nebulizer ( $100 \mu\text{L min}^{-1}$ ). Sensitivities of NP-I and NP-II were larger than 300 and 600 V  $\text{ppm}^{-1}$  (total Pb beam), respectively. Instrument parameters are shown in Table 1.

### 2.3. Data source

We statistically analyzed the Pb isotope data collected by the GeoReM website from published papers in the last 30 years (Jochum et al., 2005), as well as Pb isotopic compositions obtained through MC-ICP-MS analyses at the State Key Laboratory of Continental Dynamics in the last five years, and three reported TIMS results with lead spikes that were not present in GeoReM bibliographic lists (Galer, 1998; Hamelin et al., 1985; Thirlwall, 2000; Woodhead et al., 1995). GeoReM has collected NIST 981 data from 1985 to 2015, including 229

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