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## Sample contamination explains the Pb isotopic composition of some Rurutu island and Sasha seamount basalts

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Received October 15, 1990; revision accepted May 7, 1991

### ABSTRACT

Palacz and Saunders [1] reported Pb isotopic compositions for Rurutu island basalts that form a trend oblique to the main oceanic basalt array and interpreted them as representing a mixture of HIMU and DUPAL (or EM II) mantle sources. Detailed leaching experiments on aliquots of the same rock powders as those measured by Palacz and Saunders [1] demonstrate that these powders have been heavily contaminated by a foreign Pb component. Two contaminants with different Pb isotopic compositions are identified. They represent together more than 80% of the total Pb present in the sample. The residues display a single coherent Pb isotopic trend consistent with more recent measurements [2,3] of Rurutu island basalts and with the main oceanic basalt array.

Pb isotopic compositions similar to those of Rurutu basalts [1] were reported by Fornari et al. [4,5] for Lamont seamount basalts. They interpreted them as reflecting a Rapa or Rurutu-type (EM II) hotspot influence in their sources. Strong leaching of glass chips from one of the Fornari et al. [4,5] samples demonstrates that these glass chips were contaminated by a foreign Pb component similar to one of the Rurutu contaminants.

In both of these cases the contamination of the samples occurred, most likely, prior to the initiation of isotope analyses. These findings highlight the importance of careful sample preparation procedures and we suggest using acid-washed rock chips for even the freshest looking lavas for Pb isotopic analyses.

### Introduction

The island of Rurutu is part of the Austral islands in French Polynesia and is considered to be of hotspot origin. It is made up of two main sequences of basalts ranging in age from 12 to 8 Ma and 2 Ma to the present [6]. Rurutu basalts with high  $^{206}\text{Pb}/^{204}\text{Pb}$  values [2,3] have Sr, Pb and Nd isotopic compositions equivalent to other HIMU (high  $\mu$  or high  $^{206}\text{Pb}/^{204}\text{Pb}$ ) basalts such as those from St. Helena, Tubuai and Mangaia islands [1,7–9]. The data of Palacz and Saunders [1] for Rurutu basalts, however, display an unusual variation of Pb isotopic compositions [ $^{206}\text{Pb}/^{204}\text{Pb}$  ranges from 19.9 to almost 21.0 at a near constant  $^{207}\text{Pb}/^{204}\text{Pb}$  value of 15.77 (Fig. 1)]

and form a trend that extends from the Northern Hemisphere Reference Line (NHRL, [10]) towards the field of DUPAL basalts (basalts which plot above the NHRL, [10]). These data were interpreted [1] as a mixture of two or more distinct mantle components, specifically a HIMU component and a DUPAL or EM (Enriched Mantle) component (see discussions of [11,12] for the nature and character of these mantle components). Because such a trend in  $^{206}\text{Pb}/^{204}\text{Pb}$  versus  $^{207}\text{Pb}/^{204}\text{Pb}$  space is very uncommon among OIB (ocean island basalts), and because data from Palacz and Saunders [1] are not comparable to other data [2,3], we have re-analysed the samples previously measured by Palacz and Saunders [1]. Nakamura and Tatsu-moto [2] also raised some suspicion concerning the data of Palacz and Saunders [1] for Rapa island basalts (see p. 2917 of [2]).

The Lamont seamounts are located immediately west of the East Pacific Rise and consist

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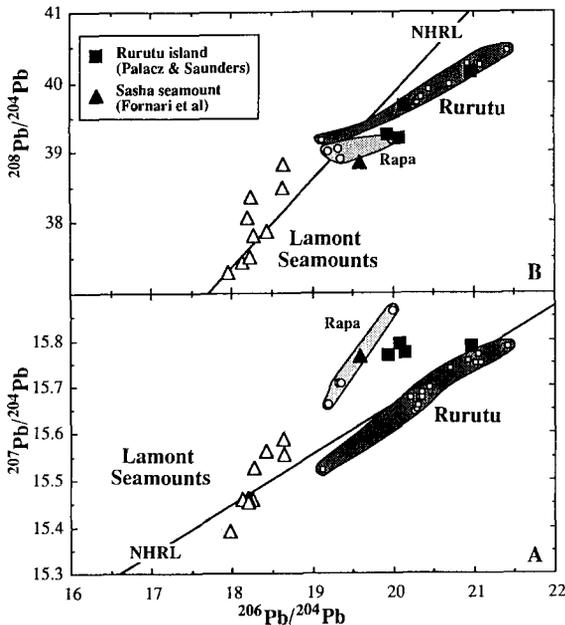


Fig. 1. (a)  $^{206}\text{Pb}/^{204}\text{Pb}$ – $^{208}\text{Pb}/^{204}\text{Pb}$ , and (b)  $^{206}\text{Pb}/^{204}\text{Pb}$ – $^{207}\text{Pb}/^{204}\text{Pb}$  isotope diagrams showing the northern hemisphere reference line (NHRL) [10], the fields of Rurutu [2,3] and Rapa [1] island basalts and the data for the Lamont seamounts (triangles) [4,5]. The reported Pb isotopic compositions of Rurutu island basalts are represented by two different symbols: filled squares represents the data from Palacz and Saunders [1], whereas smaller open squares represents the data from Nakamura and Tatsumoto [2] and Chauvel et al. [3]. Two distinct isotopic trends can be seen for the Rurutu island basalts, especially in the  $^{206}\text{Pb}/^{204}\text{Pb}$ – $^{207}\text{Pb}/^{204}\text{Pb}$  diagram the data of Palacz and Saunders [1] show a relatively constant  $^{207}\text{Pb}/^{204}\text{Pb}$  composition over a range of  $^{206}\text{Pb}/^{204}\text{Pb}$  values, while other Rurutu island data [2,3] are subparallel to the NHRL. The Pb isotopic composition of a Sasha seamount basalts reported by Fornari et al. [4,5] is represented as a filled triangle. Note this sample from the Lamont seamount chain with a high  $^{207}\text{Pb}/^{204}\text{Pb}$  composition plots in the field of Rapa island basalts.

of a series of small (1–2 km above the seafloor) off-axis volcanoes [4,5]. Fornari et al. [4,5] showed that the Lamont seamounts are exclusively tholeiitic lavas with N-MORB-like trace elements and Sr isotopic characteristics however, the Pb isotopic compositions of some of these basalts fall outside the range of N-MORB [12,13]. They found that two of these seamounts (Sasha and MOK) had relatively high  $^{207}\text{Pb}/^{204}\text{Pb}$  ratios extending into the field of basalts from Rapa and Rurutu islands (Fig. 1) as reported in Palacz and Saunders [1].

Given the large range of Pb isotopic compositions for Sasha seamount basalts, we decided to re-analyse the sample that has a particularly high  $^{207}\text{Pb}/^{204}\text{Pb}$  value [4,5], comparable to those of basalts from Rurutu and Rapa islands [1].

We report here a series of leaching experiments conducted on Rurutu island and Sasha seamount basalts. Differences in Pb isotopic compositions between leached and unleached portions of geologically recent basalt (< 10Ma) are not expected unless the samples have; (1) unusually high  $^{238}\text{U}/^{204}\text{Pb}$  values; (2) been altered *in situ* by a process which has significantly affected the Pb isotopic compositions of the rock; or (3) been contaminated by a foreign Pb component which was introduced during collection and/or handling. Because the Rurutu and Sasha basalts cannot be expected to have an extremely high  $^{238}\text{U}/^{204}\text{Pb}$  and do not show signs of severe alteration, we suspect that any difference in Pb isotopic composition between leached and unleached samples must be attributed to contamination during handling and/or crushing. Our aims were therefore to evaluate leaching procedures used in the preparation of young basalts for Pb isotopic analyses and to understand the Pb isotopic composition of these two volcanoes.

## 2. Sample description

### 2.1. Rurutu island basalts

Aliquots of the same powders as those analysed by Palacz and Saunders were measured; rock chips were not available to us. In contrast to the original paper, all samples in this study were leached prior to dissolution and the residues and leachates were analysed for their Pb isotopic compositions.

### 2.2. Sasha Seamount basalt

Hand-picked glass chips were used in this and the earlier studies [4,5]. In the earlier study selected glasses were acid washed in 2.5M HCl in an ultrasonic cleaner (M. Perfit, pers. commun., 1990). Basalt chips selected for analyses in this study were small, clear, dark green to black, glass fragments showing no signs of alteration. Glass chips were hand picked under a binocular microscope.

**3. Results of leaching experiments**

Two different types of leaching procedures have been used in this study:

(1) strong acid leach under boiling conditions (1–2 h) to ensure maximum elimination of contamination Pb and to obtain the true Pb isotopic composition of the basalts

(2) gradual leaching experiments starting with H<sub>2</sub>O and finishing with strong acid in order to understand the nature of the contaminant and distinguish the presence of more than one contaminant.

*3.1. Strong leaching experiment*

We conducted two strong leaching experiments on the rock powders and glass chips. The first involved adding 6M HCl to beakers with sample

(about 200 mg of sample), capping the beakers and heating them for at least two hours under boiling conditions. The second experiment involved the addition of 6M HCl and concentrated HF to beakers with the Sasha seamount glass chips (about 200 mg), capping the beakers and heating it for at least two hours under boiling conditions. The residues were rinsed more than four times in distilled H<sub>2</sub>O and then dissolved. Pb was isolated from both leachates and residues according to standard procedures [14]. During this part of the study the maximum total Pb blank, as measured by repeating the experiments (including column chemistry) without sample material, was less than 220 picograms.

*3.1.1. Rurutu island basalts*

A total of eleven dissolutions of the four samples analysed by Palacz and Saunders [1] were

TABLE 1

Pb Isotopic Compositions of strong leachates and residues of Rurutu and Sasha Basalts

	<sup>206</sup> Pb/ <sup>204</sup> Pb	<sup>207</sup> Pb/ <sup>204</sup> Pb	<sup>208</sup> Pb/ <sup>204</sup> Pb
<i>Rurutu island basalts</i>			
199(4) published value	20.151	15.772	39.672
199(4) residue a	20.324	15.681	39.758
199(4) residue b	20.394	15.717	39.807
199(6) published value	20.972	15.784	40.148
199(6) residue a	21.203	15.785	40.305
199(6) residue b	21.019	15.770	40.201
199(6) leachate b	20.925	15.780	40.101
199(7) published value	19.946	15.765	39.247
199(7) residue a	20.012	15.812	39.076
199(7) residue b	20.807	15.737	40.061
199(7) leachate b	19.936	15.756	39.143
199(7) residue c	20.529	15.729	39.753
199(7) leachate c	19.883	15.762	39.140
199(11) published value	20.091	15.791	39.192
199(11) residue a	20.823	15.775	39.974
199(11) residue b	20.503	15.730	39.507
199(11) leachate b	20.157	15.811	39.192
199(11) residue c	20.575	15.748	39.639
199(11) leachate c	20.157	15.811	39.192
<i>Sasha seamount basalt</i>			
Sample 1558–2014			
published value	19.572	15.766	38.854
residues after:			
6.0M HCl + HF	17.926	15.429	37.282
6.0M HCl	17.922	15.444	37.314

a, b and c refers to replicate leaching experiments. Pb isotopic ratios are corrected for mass fractionation of 0.11 ± 0.03% per atomic mass unit (amu) based on analyses of National Bureau of Standards (NBS) standard 982. All samples were measured in duplicate.

performed using the strong leaching technique (Table 1). Pb isotopic compositions of the leachates are, within errors, comparable to the values reported for the powders by Palacz and Saunders [1]. In contrast, the composition of residues are drastically different from their results [1] (see Table 1 and Fig. 2). Since results obtained on the leached residues are in the range of values obtained for other basalts from Rurutu [2,3], we conclude that the observed shifts in Pb isotopic compositions between leached and unleached powders are due to contamination by a foreign Pb component.

The introduction of a foreign Pb component to the Rurutu rock powders probably occurred during or before the rock crushing stage because our analyses of the leachate fractions (Table 1) using hot 6M HCl are, within uncertainties, identical to the values reported by Palacz and Saunders [1] for the rock powders. Our data demonstrate that the powders provided by A. Saunders had the same Pb isotopic compositions as those previously analysed by them—therefore this rules out the possibility that results published by Palacz and Saunders [1] were primarily affected by a laboratory Pb contamination. Clearly, the rock powder must have been contaminated prior to their isotopic investigation. Furthermore, one of our analyses of the residue of sample 199(7) (see Table

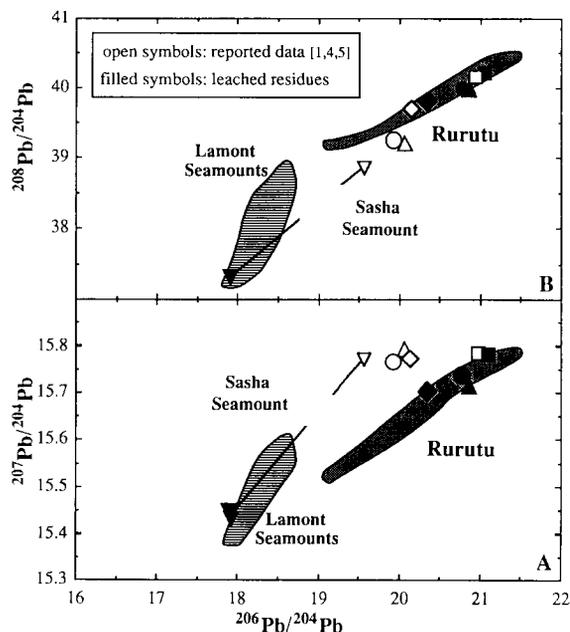


Fig. 2. (a)  $^{206}\text{Pb}/^{204}\text{Pb}$ – $^{207}\text{Pb}/^{204}\text{Pb}$ , and (b)  $^{206}\text{Pb}/^{204}\text{Pb}$ – $^{208}\text{Pb}/^{204}\text{Pb}$  isotope diagrams for selected Rurutu island basalts and a Sasha seamont basalt. The earlier data for Rurutu island basalts from Palacz and Saunders [1] (open symbols) are compared with data for the same basalt powders after leaching (filled symbols) (Table 1). The earlier data [4,5] for the Sasha seamont basalt (open inverted triangle) is compared with the data obtained after leaching (filled inverted triangles) (Table 1). See Fig. 1 for the data fields of Rurutu and Lamont seamonts.

TABLE 2

Results of step leaching experiments on two of Rurutu basalts

	$^{206}\text{Pb}/^{204}\text{Pb}$	$^{207}\text{Pb}/^{204}\text{Pb}$	$^{208}\text{Pb}/^{204}\text{Pb}$	ng Pb *	% of total Pb recovered *
<i>Sample 199(7)</i>					
H <sub>2</sub> O	19.553	15.735	38.966	4	0.4
1M HCl	19.612	15.723	38.873	392	39.4
3M HCl	20.136	15.783	39.171	378	37.9
6M HCl cold	20.221	15.801	39.314	87	8.76
6M HCl hot	20.101	15.710	39.380	79	7.9
residue	20.778	15.736	39.997	57	5.7
<i>Sample 199(11)</i>					
H <sub>2</sub> O	20.064	15.833	39.195	88	6.0
1M HCl	20.062	15.820	39.161	1010	69.5
3M HCl	19.988	15.776	39.272	189	13.0
6M HCl cold	20.087	15.754	39.323	66	4.5
6M HCl hot	20.621	15.766	39.741	64	4.4
residue	20.876	15.714	39.996	36	2.5

\* The reported Pb contents (in nanograms) were determined on aliquots of solutions that were spiked after sample dissolution and column chemistry. These values are, therefore, only minimum contents for each fraction. The % of total Pb recovered (last column) expresses these concentrations in relative terms (summed to 100%).

1: residue a) has a Pb isotopic composition similar to the value reported previously [1] and is different from analyses of the other three residues (Tables 1 and 2). This suggests that even hot 6M HCl could not eliminate the contaminant Pb in this specific case. Moreover, replicate analyses of the residues (Table 1) show a range of values for each of the Pb isotopic ratios. The range is larger than analytical uncertainties (few% versus < 1‰) and must represent variations in the composition of the powder after leaching. This suggests that the rock powders are intimately contaminated by foreign Pb and that the real Pb isotopic composition of the basalt might not be obtainable.

### 3.1.2. Sasha seamount basalt

The Pb isotopic compositions of the two residues, leached in two different ways, are given in Table 1 for sample 1558–2014 from Sasha seamount. The two residues have essentially identical isotopic compositions (Fig. 2), but both of them are very different from the value reported by Förnari et al. [4,5]. Our measurements of the residues of this sample give low  $^{206}\text{Pb}/^{204}\text{Pb}$ ,  $^{207}\text{Pb}/^{204}\text{Pb}$  and  $^{208}\text{Pb}/^{204}\text{Pb}$  ratios relative to values reported by Fornari et al. [4,5], but comparable to those reported for other seamounts from the same area [4,5] (see Fig. 2).

The introduction of a foreign Pb component to the Sasha seamount rock is somewhat difficult to envisage since glass chips and not powders were analysed in both this and the original work. However, it was probably introduced during a handling stage. M. Perfit (pers. commun.) reported that each sample was hand picked and acid leached in warm 2.5M HCl in an ultrasonic cleaner for two hours prior to dissolution. He repeated this procedure with the same Sasha seamount split twice and found that these results agreed with the previously reported data [4,5]. A further test on another glass separate by M. Perfit (pers. commun.), where he has leached hand-picked glass chips in warm 6M HCl, yielded results comparable to ours. Based on our findings, we predict that the MOK seamount basalts with high  $^{207}\text{Pb}/^{204}\text{Pb}$  ratios may well have been affected by a foreign Pb component. Recent work by Perfit [15] shows that the two MOK samples with the highest  $^{207}\text{Pb}/^{204}\text{Pb}$  isotopic compositions also shifted to lower values with stronger acid leaching procedures.

### 3.2. Incremental leaching experiment

A more detailed, incremental leaching experiment was carried out on two of the Rurutu island basalts: 199(7) and 199(11). This latter experiment was devised in order to understand the nature of the leaching process and to better characterize the composition of the contaminant. In the first leaching step H<sub>2</sub>O was added to the powders, these mixtures were vigorously stirred and then allowed to stand at room temperature for approximately one hour. After decanting and separating off the leachate, the same procedure was repeated using sequentially cold 1M HCl, 3M HCl and 6M HCl. In the final leaching step 6M HCl was added to the residue and the beaker was placed on a hotplate at boiling temperature for one hour. Lead from all different leachate solutions and residue was isolated using the same standard procedure as above [14]. After separation and purification of the Pb fraction, a 10% aliquot of the liquid was taken and spiked in order to determine the relative amount of Pb present in each leachate and in the residue. Improvements in the acid chemistry led to a reduction in the total procedural Pb blank. Thus, the maximum total Pb blank, as measured by repeating these experiments (including column chemistry) without sample material, was less than 60 pg for this stage of the experiment.

Pb isotopic compositions as well as Pb contents of the various fractions are reported in Table 2. Both Pb contents and Pb isotopic compositions are extremely variable among fractions. The proportion of Pb removed by the initial cold H<sub>2</sub>O leaching step is very low ( $\leq 6\%$ , see Fig. 3) and so are the Pb contents of the two 6M HCl steps (< 9% for each step). In contrast, high Pb contents are obtained for the 1M HCl and 3M HCl leaching steps (Table 2 and Fig. 3), these two fractions representing about 80% of the total Pb in the rock powder. The analysed residues contain only a very small proportion of the total Pb content (< 6%). For both samples,  $^{206}\text{Pb}/^{204}\text{Pb}$  and  $^{208}\text{Pb}/^{204}\text{Pb}$  ratios vary systematically with increasing leaching strength, whereas the  $^{207}\text{Pb}/^{204}\text{Pb}$  ratios change in a more complicated way (see Fig. 3). In addition, the Pb removed in the initial leaching steps has different isotopic compositions for these two samples, whereas the re-

sidues have virtually indistinguishable Pb isotopic compositions (Fig. 4).

The difference in behaviour of these two samples suggests that they have been contaminated by two different contaminants. The first contaminant (labelled contaminant 1 in Fig. 4) has a  $^{206}\text{Pb}/^{204}\text{Pb}$  ratio of 20, a  $^{207}\text{Pb}/^{204}\text{Pb}$  of 15.83 and a  $^{208}\text{Pb}/^{204}\text{Pb}$  of 39.1. It is worth noting that the isotopic composition of this contaminant is almost identical to that reported by Moorbath et al. [16] for a contaminant introduced into granitic rocks

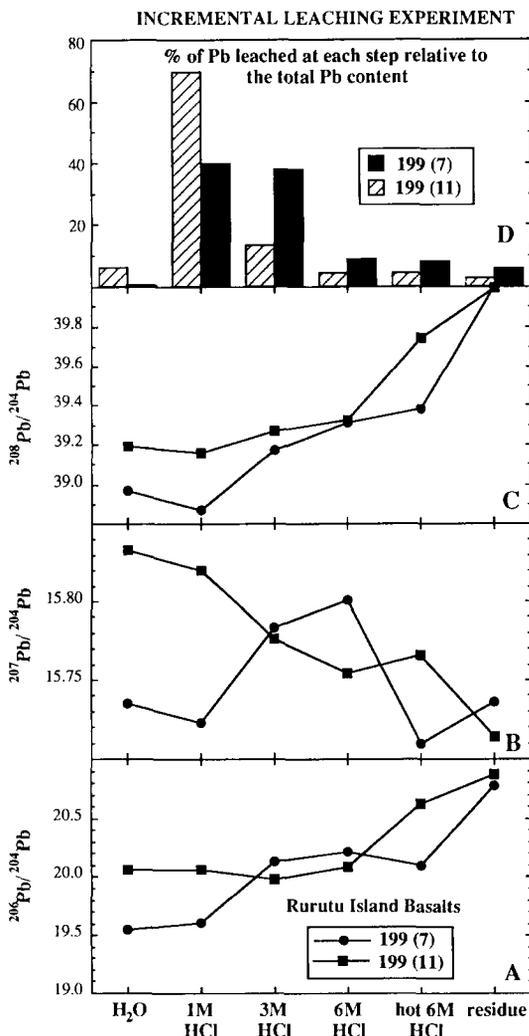


Fig. 3. Changes in (a)  $^{206}\text{Pb}/^{204}\text{Pb}$ , (b)  $^{207}\text{Pb}/^{204}\text{Pb}$ , (c)  $^{208}\text{Pb}/^{204}\text{Pb}$  ratios of the leachate solutions and residues, and (d) the relative fraction of Pb in the leachate solutions and residue of two Rurutu island basalts. Some 80% of the total Pb is removed in the 1M and 3M HCl leaching steps and its isotopic composition is distinct from that of the residue.

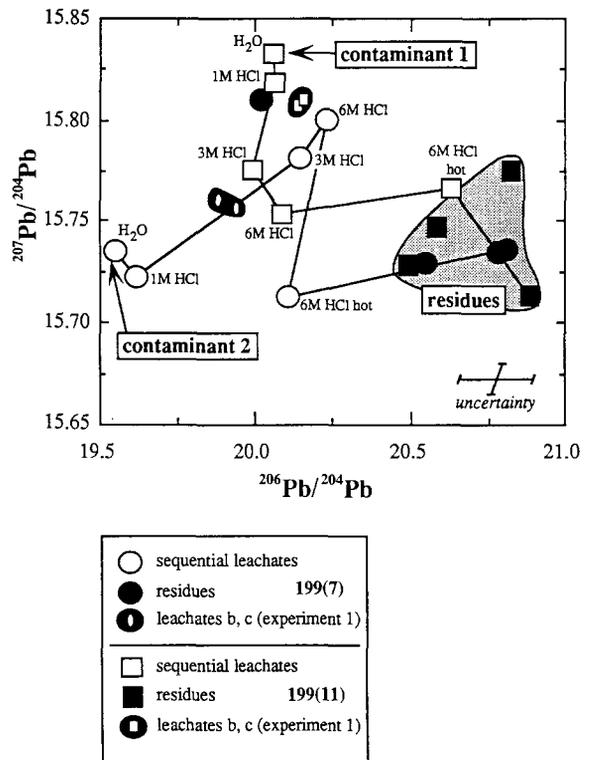


Fig. 4.  $^{206}\text{Pb}/^{204}\text{Pb}$ – $^{207}\text{Pb}/^{204}\text{Pb}$  diagram showing the changes in isotopic composition as leaching progresses for two Rurutu island basalts originally reported in [1]. Also shown are the compositions of other analyses of residues and strong leachates from Table 1. See text for further discussion on the character of contaminants 1 and 2.

from Greenland. Moorbath's samples were pickled in leaded petrol, where the Pb added to the petrol had an isotopic composition similar to that of Mississippi Valley Lead [17]. The second contaminant (labelled contaminant 2 in Fig. 4) has lower  $^{206}\text{Pb}/^{204}\text{Pb}$ ,  $^{207}\text{Pb}/^{204}\text{Pb}$  and  $^{208}\text{Pb}/^{204}\text{Pb}$  ratios. Its isotopic composition could be that of Broken Hill-type (Proterozoic) Pb ores [17], where all Pb isotopic ratios are low compared to values measured on present-day basalts. Although we have identified two contaminants, the exact nature (fluid, aerosol, powder, etc.) of these contaminants and how they were introduced cannot be precisely determined.

#### 4. Pb isotopic composition of Rurutu and Lamont basalts

These findings lead us to conclude that differences in Pb isotopic compositions between

leached and unleached portions are due to contamination during shipping, storage, handling or crushing of these samples. Saunders (pers. commun., 1990) points out that contamination most probably occurred prior to their experiments, i.e., during rock collection, shipping or storage at the British Museum, because all samples reported in their paper [1] were treated in a similar manner from the crushing procedure to the chemical separation. Only samples collected and stored by the British Museum since 1927, are affected by the contamination whereas others, in particular Mangaia basalts display normal Pb isotopic compositions. The contamination of the Sasha seamount glass chips is also likely to have occurred prior to the stage when the glasses were selected for analyses. However, like the Rurutu example, the exact nature (fluid, aerosol, powder, etc.) of these contaminants and how they were introduced cannot be precisely determined.

Based on our findings, we suggest that the unusually high  $^{207}\text{Pb}/^{204}\text{Pb}$  composition of one of the Rapa basalts (Fig. 1) reported by Palacz and Saunders [1] can be explained by the same contamination of the rock powder as in the Rurutu case. Saunders (pers. commun., 1990) notes that these samples were also stored at the British Museum since 1927 with the Rurutu samples. Other results for Rapa island basalts [2,9] have lower  $^{206}\text{Pb}/^{204}\text{Pb}$ ,  $^{207}\text{Pb}/^{204}\text{Pb}$  and  $^{208}\text{Pb}/^{204}\text{Pb}$  ratios similar to the ratios reported by Palacz and Saunders for their least radiogenic sample 198(6). These data fall within the general oceanic basalt trend.

The Pb isotopic compositions of the residues for the Sasha seamount basalt are similar to those of other nearby Lamont seamounts, so that it is not necessary to invoke a HIMU or DUPAL component in the source of these basalts. Likewise, our data for the residues of the four Rurutu samples are comparable to other available data for that island [2,3] (Fig. 2). Here again, there is no need to invoke mixing of hypothetical end members such as HIMU and DUPAL components in the source of Rurutu island basalts.

## 5. Observations and recommendations

The problems encountered during the acquisition of Pb isotopic data documented here are not

unique. Discrepancies in Pb isotopic compositions in other young basalts are found in the literature (Dupuy et al. [18] versus Duncan et al. [19] and Sun [7] versus Oversby et al. [20,21]). Unless erroneous Pb isotopic measurements are detected, they may lead to false interpretations, for example the creation of fictitious mantle reservoirs. Our study, in addition to these examples, emphasizes the importance of careful sample preparation procedures. We recommend using rock or glass chips, coupled with strong acid leaching, for Pb isotopic analyses of young basalts.

## Acknowledgments

We very much appreciate the generosity and cooperation of Andy Saunders, Mike Perfit and Zenon Palacz. Without their help and samples this study would not have been possible. Suggestions made by Steve Goldstein, Al Hofmann, Mike Perfit, Andy Saunders and Shen-su Sun improved the nature and content of the paper. We thank them for fruitful discussions and their comments on the manuscript. The review comments of Drs. Moorbath, Tatsumoto and Prof. Begemann are appreciated.

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