HCl To remove $CaCO_3$

for chemistry, is also contaminated, having $^{10}Be/^{7}Be$ ratio of 10^{-5} shortly after manufacture (12,13).

EXPERIMENTAL SECTION

Reagents: All reagents used are of analytical grade. High purity quartz distilled water is used for dilution. Current total-chemistry blanks (reflecting contamination from reagents, Teflon ware, baking crucible and handling) are on the order of 1 to 5 $\times$ 10^4 atoms of ^{10}Be.

The spike is added to the sample as a weighed amount of $^{9}BeF_2$ in 0.1M HCl. The spike concentration was determined with inductively coupled plasma spectrometers at the National Bureau of Standards and the Geological Survey, Denver; the two independent determinations agreed to 0.1%. To ensure isotopic equilibration, the ^{9}Be spike is the first reagent added to the sample powder which is then allowed to digest in HF-$HClO_4$ or HF-HNO_3 mixture at room temperature for a minimum of 5 days, with occasional swirling. The sample is then heated at ~60°C for several days in a partially closed Teflon jar, before the cover is further loosened to expel the excess acid. In the case of HF-$HClO_4$ mixture the temperature is gradually increased till $HClO_4$ is completely expelled at ~230°C. Expulsion of HNO_3 at low temp. is dictated by the volatility of $Be(NO_3)_2$.

Primary standards containing known atomic concentrations of ^{10}Be and ^{9}Be are not available in quantities large enough for monitoring the accelerator's performance, so we dissolved a large amount of pelagic sediment from Deep Sea Drilling Project Core 482C to form a stock solution as a secondary standard. Beryllium is extracted from this as needed. This high level house standard is compared with β-counted standards furnished by Dr. James Arnold of the University of California, San Diego. All results reported here have been normalized to Arnold's standard. In order to examine the experimental uncertainties at the low count rates typical of lavas, we prepared a low-level house standard by adding

ΔE pulses, are proportional to the rate of energy loss in the counter gas; pulses from element 3, called E pulses, are proportional to the total energy minus that lost in element 2. Display in an E-ΔE two dimensional spectrum allows certain particle identification. For such display the energy lost in element 2 is added by the computer to that obtained from element 3.

Chemical Separation:

a) *Rocks*: Fig. 2 is a schematic outline for the procedure as applied to silicate samples. Heavy arrows pointing upwards and downwards indicate supernates and precipitates, respectively. Rectangles on the right hand side indicate discarded components at various steps enumerated in Arabic. Five grams of the rock powder is mixed with 10 ml H_2O and 10 ml conc. $HClO_4$. ^9Be spike is added followed by the slow addition of 20 ml conc. HF. The loosely covered sample is digested as described earlier. The following is a brief step by step outlining of the method.

(1) The dry cake resulting from HF-$HClO_4$ digestion is treated with 20 ml 4M HCl and dried at ~200°C. This cake is completely dissolved in 150 ml 2M HCl.

(2) About 30 ml concentrated NH_4OH are added to the sample solution to bring the pH to 8, then the pH is further increased to 9 with 6 ml of 10M NaOH. The resulting brown precipitate usually contains >98% of the Be. The supernate is discarded.

(3) The hydroxide precipitate is dissolved into a clear solution with 170 ml of 2M HBr.

(4) About 20 ml of concentrated NH_4OH are added in small portions while stirring till dense precipitation of Fe commences. At this point NH_4OH is added in smaller and smaller portions till the fading color of the supernate is straw-yellow. This generally occurs around pH 4. Under this condition Fe

and Ti are precipitated with no more than 2% of Be lost to the precipitate. The precipitate is discarded. Overshooting to higher pH causes larger Be losses. In this case it is advisable to redissolve the precipitate by addition of 2M HBr, then reprecipitate more cautiously. Exact adjustment of the pH is not required; the general rule is that no Be loss of consequence (i.e., >5%) takes place before the small trace of Fe (imparting a straw-yellow color to the supernate) is removed.

(5) The supernate of the above step contains Al+Be and traces of Fe and sometimes traces of Ti. These are coprecipitated by the addition of 5 ml concentrated NH_4OH followed by 10 ml of 10M NaOH. The discarded supernate usually contains <0.5% of total Be.

(6) The $Al(OH)_3$, tinted with traces of Fe and containing ~95% of the Be, is suspended in concentrated ammonia in capped polyethylene tubes (on the average, four 50 ml tubes per sample, each containing ~10 ml hydroxide) agitated by a vibrating device. The volume of NH_4OH used is about 3 times that of the precipitate. Once the hydroxide is disseminated the tubes are centrifuged and the supernate, containing <0.5% of the Be, is discarded.

(7) The above step is repeated once more, causing the hydroxide to shrink by ~30% of its original volume.

(8) The gel (wet with NH_4OH) is disseminated in 5% HF about twice the volume of the gel. More 5% HF is added till pH 5 is reached. Again exact adjustment of the pH is not required. In most cases, a convenient indication of this pH is the sudden color change of the suspended gel from brown to light beige. At this stage over 85% of the Be is extracted to the liquid phase together with minor amounts of Al and traces of iron (and occasionally traces of Ti as well). Overshooting to pH <<5 increases the amounts of Al and Fe contaminants in the liquid phase.

(9) Concentrated NH_4OH is added dropwise to the light beige syrup until pH 8 is reached. This is conveniently indicated by the reappearance of the brown color of Fe. The contents are centrifuged and the precipitate (Al+Fe) is discarded.

(10) The supernate is evaporated to dryness and the white NH_4F residue is expelled at ~200°C in a venting oven in a hood.

(11) The resulting residue containing >85% of the Be and traces of Al and occasionally traces of Fe and Ti is redissolved in 10 ml of 2.5% HF. Again NH_4OH is added till pH 8 to precipitate the last traces of Al and Fe. The supernate containing >75% of Be is evaporated to dryness and NH_4F is expelled.

The residue from the above step is evaporated twice from ~1 ml of 20% $HClO_4$ to oxidize minor amounts of organic matter (often unseen) suspected of being the major source of ^{10}B interference in the accelerator. The boron background is further reduced, often to a negligible level, by carrying out the separation in isolation of the turbulent laboratory air. The resulting residue is converted to chloride by evaporation with ~1 ml 2M HCl twice. This residue is dissolved in 10 ml of 1M HCl with occasional minor turbidity, owing to minor amounts of Ti, may be centrifuged away. $Be(OH)_2$ is precipitated from this solution, by addition of conc. NH_4OH in drops, at pH ~ 8. No adjustment of pH is required; a faint smell of ammonia is a good enough indicator. $Be(OH)_2$ is then heated in a small alumina crucible, at 550°C for 1 hr to obtain the oxide. Heating at a much higher temperature is avoided as a safeguard against ^{10}Be memory in the crucible, which is reused after thorough cleaning in boiling aqua regia and prolonged soaking in that solution. It has long been known (15) that the energy content of BeO is greater (and thus its decomposition is easier) the lower the temperature of its preparation.