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Activation Analysis for Selected Elements in Micrometeorites and Hypervelocity Projectiles

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"Investigation of suitable catcher materials for hypervelocity projectiles, and the analysis of Na, Mn, W, Fe, Co, Sc, Zn, and Cr in simulated samples."

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ABSTRACT

In an attempt to find a suitable catcher for micrometeorites, several likely candidates have been investigated. Of these "99.99999% pure" KCl crystals and ultra high purity silicon appear to be the most advantageous materials for such a purpose.

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INTRODUCTION

The possibility of many theories of origin make the composition of micrometeorites of great interest to the geochemist and cosmochemist.

One possibility exists that dust may have existed in galactic space prior to and concomitant with the formation of our solar system, resulting from expulsion of gases into space by stars of unstable composition such as novas. This implies that at least some micrometeorites might be the most primitive samples of solid matter subject to analysis in our solar system, perhaps predating even the sun's formation. Such an analytical finding would present strong evidence that the formation of our planetary system and the sun itself was due to a condensation process whereby gases and dust particles gravitated into dense matter in a central region of galactic space and thereby created our solar system.

Secondly, some micrometeorites must surely be the result of a grinding process from collisions in space of large meteorites. Therefore, it would be of interest to correlate the composition of micrometeorites with the meteorites which have been recovered on earth.

Third, the origin of some solar dust might be from expulsion of matter from a planet or moon by volcanic activity or by collision of large meteorites such as those thought responsible for most craters on the moon.

Thus, if these tiny dust particles could be collected and analyzed, evidence pro or con could be presented for each of the differing theories of origin.

Although there are incredibly large numbers of micrometeorites in space, the flux density is very low due to the immense volume they occupy. A further complication in obtaining enough material for analysis is the rapid decrease in particle density with increasing mass of the micrometeorite, such that a square meter sized catcher orbited for 126 years has a 65% probability of capturing a particle larger than 100 micrograms.

When dealing with such small quantities of matter, two requirements must be met -- a sufficient analytical tool to detect elements at the nanogram level, and a material serving as a catcher free of the elements sought after. High flux activation analysis is the proper tool, and our investigations are aimed at finding a suitable catcher. Thus, we are determining the contamination levels of a number of elements of interest to us in the most convenient catchers available.

EXPERIMENTAL: CATCHER MATERIALS INVESTIGATED

ALUMINUM:

This was the first catcher material proposed and studied. From the analytical results of activating aluminum foils, the conclusion was reached that the production of ^{27}Mg and ^{24}Na by (n, p) and (n, α) reactions, respectively, from ^{27}Al completely ruined any hope of determining these elements in the sample itself. Thus, further investigation of aluminum was abandoned.

POLYETHYLENE:

This material has the advantage of being chemically constituted of only carbon and hydrogen, both elements which produce negligible activity upon irradiation with neutrons.

The elements Mn, Na and W were analyzed in polyethylene samples that had been bombarded by hypervelocity particles without difficulty in the OSU TRIGA reactor at a flux of 7×10^{11} neutrons/cm^2 sec. The analysis of iron by the comparator technique using iron wire standards was performed in a high flux reactor (the complete report is contained in the third quarterly report for the period January 1 to March 31, 1970). The iron content was found to vary from 16 micrograms to less than one microgram depending on whether the projectile was fayalite or olivine. The sensitivity for scandium was less than a nanogram in this high flux irradiation while chromium and cobalt sensitivities were less than 10 nanograms.

Although the high flux irradiation showed great promise, the polyethylene chemically decomposes in the high neutron flux with subsequent release of H_2. The second progress report for the period October 1 - December 31, 1969 presents a calculation which indicates that one gram of plastic would completely decompose into H_2 and C in 470 minutes at 10^{14} neutrons/cm^2 sec (i.e. complete decomposition for irradiation of a micrometeorite-containing sample).

In a simulated study of micrometeorites, three samples each of polyethylene containing glass, olivine, and fayalite which had been impacted into the plastic were analyzed by activation at 10^{13}/cm^2 sec for 6 hours. The amount of iron in each sample (save one) was fairly easily determined by counting on a 30 cc Ge(Li) detector, even at this low flux by the comparator technique; and scandium, chromium, cobalt and zinc were deter-

mined as upper limits by taking three times the standard deviation of the area at the energy at which the photopeak for the particular element occurred (3σ calculation). Knowing the maximum activity due to the element, use of known cross sections, isotopic fractions, etc., allowed calculation of the maximum amount of the element in question present. It is apparent from the results of calculations performed in the above manner, exhibited in Table I, that the sensitivity of the method is very high.

BERYLLIUM:

Many low Z elements have the following nuclear characteristics making them ideal catcher matrices:

1. Short half life from (n, γ) reactions.

2. Low cross sections for fast neutron reactions, and short half lives of products.

3. Stable solid states which do not release gases upon irradiation.

Beryllium metal, obtained from U.S. Bureau of Mines, Albany, Oregon, thus appeared ideally suited. The purest sample obtainable was however, so grossly contaminated that a "forest" of gamma lines and an intense bremstrahlung activity was the result of counting on a Ge(Li) detector. Due to the toxic nature of beryllium salts no effort was made to further investigate beryllium compounds.

CARBON:

Inquiries were made of all major carbon suppliers to find the purest carbon available. Again, as with beryllium metal, "pure" carbon proved quite impure for our purposes, and no purpose would have been served in identifying the gamma lines and quantizing the results.

KCl CRYSTALS:

The physical chemistry department has kindly provided us with KCl crystals which are 99.99999% pure. Activation of six samples of the pure crystals was performed in the high flux reactor at Missouri for two weeks at an average flux of 7.0×10^{13} neutrons/cm^2 sec. The samples were acti-

vated in pure aluminum foil along with an iron wire standard and then encapsulized in a standard aluminum container which was welded shut so that no leakage of liquid under pressure was possible. After activation the samples were cooled for three weeks and shipped back for analysis.

It was immediately apparent from the spectrum obtained with a 30 cc Ge(Li) detector that bremstrahlung activity from a high energy beta emitter was so large as to completely eliminate any hope of instrumental analysis. A beta absorption curve identified the extremely high activity as due to ^{32}P and ^{35}S, the former produced from ^{35}Cl (n, α), and the latter by ^{35}Cl (n,p).

An attempt to separate the interfering elements by an iron hydroxide co-precipitation of the desired elements, while the ^{32}P and ^{35}S remain in solution, failed, since the ^{32}P also precipitated along with the iron. We then attempted to remove the ^{35}S and ^{32}P activities by ion exchange techniques.

After some trial and error, it was found that the ^{32}P activity could be removed by passing a neutral solution of the KCl crystal through a column charged with Dowex 1 x 8 resin. Experiments with standard solutions verified that all cations of interest passed easily through the column while the ^{32}P (presumably as PO_4^{3-})remained on the resin. However, the ^{35}S could be removed only by passing the element from the Dowex column through a column loaded with Ag 50 W-4X cation exchange resin, which retains the cations of interest while passing through the ^{35}S. The cations are then recovered by elution with a strong HCl solution.

The results of analyses of two crystals are presented in Table II. These results verify that these crystals, especially KCl, are of exceptional purity with sensitivities for the elements reported in the picogram to nanogram range. Apparently iron is much higher in NaCl than KCl. One may note the high Rb contamination for KCl.

SILICON:

Samples of pure silicon were obtained from three major suppliers and are being investigated at present for contamination levels. Although the work is still in its infancy and will be covered more thoroughly in the next project report; the initial activations are encouraging with very low contamination levels.

SUMMARY AND CONCLUSION:

It appears that an excellent candidate for a catcher material is the phenomenally pure KCl "home grown" here at OSU. However, being unsatisfied with tedious and dangerous radiochemical procedure necessary for separation of the high levels of ^{32}P and ^{35}S activities arising from fast neutron reactions on ^{35}Cl, we are continuing the search for the ideal catcher. Polyethylene is suitable only for micrometeorite analysis in the microgram range due to the problems with H_2 pressure buildup during long irradiations at high flux. We have hopes that zone refined Silicon is the answer for analysis of micrometeorites in the nanogram range, despite the fact that three major elements, Si, Al and Mg are undeterminable.

TABLE I

Amounts of Selected Elements Imbedded in Polyethylene Catchers as a Result of Hyper-Velocity Impact, Micrograms

Material	NASA Number	Fe	Sc	Cr	Co	Zn
Glass	B-2	1	0.0002	0.01	0.003	0.1
	B-14-1,2	4 ± 1	0.0002	0.01	0.005	0.1
	B-15-4,5	5 ± 1	0.0003	0.02	0.004	0.1
Olivine	#7	9 ± 1	0.0003	0.02	0.004	0.1
	B-6-4	1.7 ± 0.7	0.0002	0.02	0.004	0.1
	B-13-1,2	0.7 ± 0.2	0.0003	0.02	0.004	0.1
Fayalite	#6	12 ± 1	0.0005	0.02	0.005	0.3
	B-8-2,3	4 ± 1	0.0002	0.02	0.003	0.1
	B-12-1	17 ± 1	0.0003	0.02	0.003	0.1

Masses listed are from 3 σ calculations except for Fe values.

All values are in micrograms

TABLE II

Contamination Levels in Pure Crystals, ppb

Crystal Type	Thermal Neutron Flux	Fe	Rb	Sc	Cr	Co	Zn	Sr
KCl	7.0×10^{13}	*10 ± 2	*8740 ±110	0.007	0.07	.01	.01	8
NaCl	7.1×10^{13}	*127 ±4	12	0.04	0.5	0.05	0.3	37

*determined by comparator technique.

all other determinations by 3 σ calculations, and thus is an upper limit.

all values reported as ppb.