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The Allende Meteorite Reference Sample

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EDITORS

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ABSTRACT

Jarosewich, Eugene, Roy S. Clarke, Jr., and Julie N. Barrows, editors. The Allende Meteorite Reference Sample. *Smithsonian Contributions to the Earth Sciences,* number 27, 49 pages, 32 tables, 1986.—A reference material for comparative analytical studies and standardization was prepared from fresh, clean specimen material from the Allende, Mexico, Type CV3 carbonaceous chondrite fall of 8 February 1969. Fragments weighing 4 kg were powdered, homogenized, and split into 1 g and 5 g subsamples. Analytical results for a total of 74 elements were provided by 24 analysts or groups of analysts. A variety of techniques were used, and many elements were determined by more than one technique. Reports from contributors of data outline their procedures and give their results in detail. Sample homogeneity has been evaluated in terms of this body of data, and "recommended values" are suggested for 43 elements.

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The Allende Meteorite Reference Sample

Editors' Introduction

Eugene Jarosewich, Roy S. Clarke, Jr., and Julie N. Barrows

The fundamental significance of meteorites for such diverse scientific fields as cosmochemistry, geochemistry, and planetary dynamics has become increasingly apparent during the past three decades. It is now generally accepted that meteorites are our most primitive rocks, providing a record of extraterrestrial events as ancient as the collapse of the solar nebula and as contemporary as recent solar flares. These characteristics have made meteorites sought-after subjects for interdisciplinary study, a practice that is now commonplace.

Geochemists have been historically attracted to meteorites as a source of information on the distribution of chemical elements within the earth and the planetary system as a whole. Our concept of the distribution of the nonvolatile chemical elements in the planetary system is based on chemical analyses of the most abundant group of meteorites, the chondrites. The analysis of chondrites, however, presents problems beyond those normally encountered in the study of terrestrial rocks. Difficulties result from the mineral assemblages present in most chondritic meteorites. In addition to major amounts of the common rock-forming minerals olivine, pyroxene, and feldspar, they contain significant quantities of troilite (FeS) and the metallic phases kamacite (low-Ni Ni,Fe) and taenite (high-Ni Ni,Fe). The association of sulfide and metallic phases with silicates presents both sampling and analytical problems. Early researchers recognized these problems; they analyzed meteorites with care using chemical methods that at the time were still in their infancy.

During the 19th and early 20th centuries large numbers of chondritic meteorite analyses were reported in the literature in widely scattered sources. As mineralogical and chemical knowledge accumulated, it became obvious that many of these analyses were seriously flawed. Urey and Craig (1953), in their comprehensive paper on the composition of stone meteorites, reviewed the older literature and established the modern approach to the evaluation of meteorite analyses. Mason (1965) modified the Urey and Craig criteria for acceptable analyses by introducing more rigorous mineralogical considerations. The discussion continues with no end in sight. It is now conducted, however, in an established climate of awareness of the need for constant critical evaluation of analytical techniques, procedures, and results.

Interpreters of meteorite analyses have required progressively more accurate data as the problems they address have become more sophisticated. The use of standardized techniques that are monitored periodically by reference samples is a common strategy for increased accuracy and for interlaboratory comparisons. Such reference materials are necessary for modern instrumental techniques because of their comparative approach.

Two of the most widely used geologic reference samples have been the rock samples G-1 and W-1, which have been used primarily for the study of precision and accuracy of chemical and spectrochemical methods as summarized by Fairbairn et al. (1951). Stevens et al. (1960) reviewed the eariler analyses of these two rocks, compiled new data, and suggested "recommended values." Fleischer (1965) updated this work with new analyses and new recommended values.

At the time our work was undertaken in late 1969 there were several types of geological reference samples available that covered the compositional range of most common terrestrial materials (Webber, 1965; Abbey, 1972; Flanagan, 1973, 1976; to list a few). For meteorites, however, such a reference sample was not available, and terrestrial reference samples were not always suitable for comparison with the analyses of meteorites. Typically, both the level of trace elements in meteorites and the concentration relations of a specific element to concentrations of other elements with which it is associated differ greatly from those present

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in terrestrial materials. The differences are such that even mixtures of different reference samples will not approximate the composition of meteorites.

There are two primary reasons why a meteoritic reference material had not been prepared previously. The first and most important is that a suitable meteorite had not been available in adequate amount for preparation of sufficient powder for subsequent distribution. Some meteorite finds that might have provided enough material were considered too contaminated by their terrestrial surroundings to be useful. Secondly, the problem of preparation of homogeneous samples had not been solved. Meteorites that had been considered were primarily ordinary chondrites containing 5–15% metal. It is very difficult, and in fact almost impossible, to pulverize a large sample containing metal of various grain sizes to less than 100 mesh particles so that a reasonable homogeneity may be assured.

The Allende, Mexico, meteorite fall of 8 February 1969 (Clarke et al., 1970) provided a solution to these problems. The meteorite was a fresh fall available in large quantity. Early work established that it was a rare Type III carbonaceous chondrite that contained very little metal and was easy to homogenize. The availability of this meteorite led us to undertake the preparation and distribution of a meteoritic reference sample. An important additional stimulus was the need of the scientific community for a reference material for the analyses of returned lunar samples.

ACKNOWLEDGMENTS.—Several participants at the Apollo 11 Lunar Science Conference in Houston in 1971, W.D. Ehmann, P.A. Baedecker, J.W. Morgan, G.H. Morrison, and A.A. Smales, discussed with one of the authors (E.J.) the need for a meteoritic reference material for chemical analyses. Their suggestions and enthusiastic support for this project are acknowledged.

We wish to thank Mrs. P. Brenner for her diligent and meticulous preparation of the sample and cataloging of the individual splits. Also, we thank F.J. Flanagan of the U.S. Geological Survey for sharing his experience and advising on the details of sample preparation, for his suggestions on statistical evaluation of the results, and for a general critique of the paper. This work was made possible by the Smithsonian Research Foundation Grant 413616 and by partial support from NASA Grant NGR-015-146 (B. Mason, Principal Investigator).

Sample Preparation

Two pieces from a 35 kg Allende meteorite specimen (NMNH 3529), one of 2.4 kg and the other of 1.6 kg, were selected for preparation of the sample powder. Fusion crust covered approximately 35 percent of the surfaces of both pieces and was removed by sandblasting. The pieces were then cleaned of entrapped dust using a jet of compressed air followed by brushing with a nylon brush. These pieces were powdered separately and combined into one 4 kg sample powder.

Fragmentation and powder preparation were carried out in a clean room in which no other laboratory activities were conducted. The two large pieces were broken to fragments of about 1 cm using a hardened steel pestle and a steel plate placed in a plastic tray. The few small chips that were projected beyond the tray were excluded from the sample to avoid contamination. The cm-size fragments were further broken down in a diamond mortar, and they were then ground by hand in an agate mortar to fine powder. The powder was passed through a 100-mesh nylon sieve with the aid of a nylon brush and collected in a 2-gallon polyethylene bottle previously cleaned with dilute nitric acid and distilled water. No metal particles large enough to be retained by the 100-mesh sieve were found in the 4 kg of powdered sample, but the powder does contain small particles (<100 mesh), which are attracted by a magnet. Special care was taken in the preparation of the sample to minimize contamination, thus keeping the sample compositionally as close as possible to the fresh meteorite.

The sample powder was homogenized in the large polyethylene bottle, which was rolled on a jar-mill rolling machine. Twenty-nine portions weighing either 32 g or 160 g were taken from this powder and split into 1 g or 5 g subsamples using the 32-position U.S. Geological Survey stainless steel conical splitter (Flanagan, 1967). Small plastic vials, also previously cleaned with dilute nitric acid and distilled water, were used to collect the subsamples. Each vial was given split and position numbers. Two subsamples, selected on the basis of a table of random numbers, were made available to participating laboratories for analyses.

Evaluation of Homogeneity

Homogeneity at the subsample level is a prerequisite for a useful reference powder. When a large quantity of material (for instance, 1 g) is used to calibrate an instrument or to check an analytical method, a representative sample may be comparatively easily obtained. However, if very small subsamples are used (10–100 mg), as is common in trace element analyses, it may be difficult to obtain a representative sample. If the powder is not ground sufficiently fine, for instance, the presence or absence of an element residing in a specific over-sized grain may significantly affect the results on that aliquot. To avoid problems of this nature our material was finely ground, sieved to assure particle size of less than 100 mesh, thoroughly mixed, and carefully split. The particle size distribution of the powder was determined on a 15 g subsample with the following results:

Mesh Size		*Percent*
<100	>160	1.5
<160	>200	11.1
<200	>300	18.0
<300		69.4

More than 85% of this subsample passed the 200-mesh sieve and less than 2% of it was retained on the 160-mesh sieve.

Summary of Results

The contributed papers in this report were prepared during the early to mid 1970s by the authors who present their analytical data on the Allende reference material and describe their methods. Major, minor, and trace elements were determined by a variety of techniques. Included among them were gravimetric (grav), colorimetric (color), and titrimetric (titr) analyses; flame and/or atomic absorption spectrophotometry (flame); x-ray fluorescence spectrophotometry (XRF); neutron activation analysis (NAA); mass spectrometry (MS); emission spectrometry with various sources (ES); and isotopic dilution analysis (ID). Although these techniques have been described extensively in the literature, we asked contributors to present brief summaries of their methods so that readers may evaluate specific results. Vincent (1952), among others, commented on the importance of giving this type of detailed background material.

Table 1 presents a summary of the data. Major and minor elements were tabulated as oxides in the conventional order for reporting meteorite analyses; results for the other elements are listed by atomic number (in parentheses). Table 1 also gives an overview of the methods that can be successfully used in the determination of a desired element, and it can be seen that different analytical methods provide acceptable results. For example, neutron activation analysis (NAA), a method used primarily in trace element work gives satisfactory results for some major and minor elements.

"Recommended values" were derived for 43 of the elements for which there were sufficient data in the following way: the mean and standard deviation of all analyses was calculated; those analytical values occurring beyond one standard deviation from this mean were excluded (marked by asterisk), and the second mean was calculated. This second mean is the recommended value. This admittedly arbitrary device excluded widely varying results from the calculation of recommended values and resulted in small standard deviations. An exception to this procedure was made for Fe and Ni, for which all neutron activation results were excluded. The wide variation in these results (21.1%–26.7% for Fe) would have seriously biased the recommended value in favor of a technique that is known to be much less precise than titrimetric and x-ray fluorescence techniques. Means and recommended values were not calculated for those elements for which only a few varying results were available, although the data are listed.

The homogeneity of the reference powder was estimated from data provided by contributors. For those sets of data that consist of an independent determination of an element in two portions of sample from each of two vials, the set of four data was considered as an experimental design with a single variable of classification (the two vials). The calculations for the analysis of variance for this design are given in statistical texts, including Dixon and Massey (1951). The calculations result in two mean squares: one for the variation in the data attributable to the means of the data in the vials and the other for the variation within the vials (also called the error mean square).

These mean squares, frequently abbreviated MS(V) and MS(E) respectively, are used to form the F ratio, MS(V)/MS(E). We have used the 95 percentile of the F distribution (95% confidence level) for the value not to be exceeded by the ratio calculated from the data and the appropriate value is $F_{0.95}$ (d.f. 1,2) = 18.5 where there is one degree of freedom (d.f.) for the vials and two (d.f.) for the error term. If the calculated F ratio does not exceed the value of 18.5 then the variation due to the vial means is not significantly larger (NS) than the variation within the two vials; and the element or oxide may be said to be homogeneously distributed between the two vials. These conclusions from the analysis of variance are listed in Table 2 as NS (homogeneous) or S (heterogeneous). Of the 75 such tests made, the element or oxide could be said to be distributed homogeneously among the vials for 71 tests. Based on the estimates that are listed in Table 2, we conclude that the Allende reference powder is homogeneous at the 95% confidence level for 44 of the 48 elements. The apparent significant inhomogenity of O, Cs, Ce, and Tb may instead represent analytical problems.

A more elaborate statistical evaluation of sample homogeneity would be desirable but the required data are not available. Most elemental values determined by different techniques and in different laboratories establish a high level of homogeneity for the reference powder. Nevertheless, individual users are encouraged to evaluate critically the available data in the context of the use they contemplate.

TABLE 1.—Summary of analytical results on the Allende meteorite reference sample drawn from 24 laboratories (means and recommended values have been calculated and are given; contributors are identified by the number of their paper (see "Contents"); several results reported in contributions as elements (in ppm) are given as oxides (in percent) in this table; asterisk indicates result not included in calculation of "recommended value." Abbreviations for methods: color = colorimetric analysis; ES = emission spectrometry; flame = flame or atomic absorption spectrometry; grav = gravimetric analysis; ID = isotopic dilution analysis; LECO = carbon by LECO; MS = mass spectrometry; NAA = neutron activation analysis; Pnfld = Penfield water determination; SSMS = spark source mass spectrometry; titr = titrimetric analysis; XRF = x-ray fluorescence spectrometry).

Constituent	Results	Method	Contributor	Split/ Position
	PERCENT			
SiO_2	*35.5 (av of 2)	NAA	5	13/2
	34.15	grav	6	19/11
	34.48	grav	6	10/31
	34.11	grav	11	20/2
	34.26	grav	11	20/7
	34.07 (av of 2)	XRF	12	5/10
	34.12 (av of 2)	XRF	12	9/18
	34.28	grav	19	20/1
	34.27	grav	19	22/10
	34.70	NAA	22	19/14
	34.53	NAA	22	8/26
	34.10	grav	23	20/20
	*33.65	XRF	24	1/3
	*33.62	XRF	24	4/22
	34.27±0.46 (mean)			
	34.28±0.21 (recommended value)			
TiO_2	0.16	color	6	19/11
	0.17	color	6	10/31
	0.17	NAA	8	6/25
	0.13	NAA	8	7/4
	0.16	color	11	20/2
	0.16	color	11	20/7
	0.15 (av of 2)	XRF	12	5/10
	0.15 (av of 2)	XRF	12	9/18
	0.15	NAA	15	6/24
	0.13	NAA	15	12/29
	*0.19	color	19	20/1
	*0.18	color	19	22/10
	0.13	color	23	20/20
	0.140	XRF	24	1/3
	0.141	XRF	24	4/22
	0.15±0.02 (mean)			
	0.15±0.01 (recommended value)			
Al_2O_3	3.21	NAA	1	16/10
	3.36	NAA	1	16/32
	3.18	grav	6	19/11
	3.19	grav	6	10/31
	3.40	NAA	8	6/25
	3.36	NAA	8	7/4
	3.27	grav	11	20/2
	3.29	grav	11	20/7
	3.22 (av of 2)	XRF	12	5/10
	3.23 (av of 2)	XRF	12	9/18
	*3.6	NAA	15	6/24
	*3.6	NAA	15	12/29
	*3.71	grav	19	20/1
	*3.67	grav	19	22/10
	3.27 (av of 2)	NAA	20	10/3
	3.23 (av of 2)	NAA	20	12/32
	3.43	NAA	23	20/20
	3.20	XRF	24	1/3
	3.30	XRF	24	4/22
	3.35±0.17 (mean)			
	3.28±0.08 (recommended value)			
Cr_2O_3	0.5363	NAA	1	16/10
	0.5203	NAA	1	16/32
	0.5391 (av of 9)	ES	2	1/32
	0.5407 (av of 7)	ES	2	8/31
	0.5350 (av of 9)	ES	2	20/5
	0.5294 (av of 9)	ES	2	20/3
	0.50 (av of 2)	NAA	5	13/32
	0.56 (av of 2)	NAA	5	1/24
	*0.43	color	6	19/11
	*0.45	color	6	10/31
	0.4709 (av of 4)	NAA	7	7/10
	*0.4648 (av of 2)	NAA	7	4/25
	0.528	NAA	8	6/25
	0.539	NAA	8	7/4
	0.54	color	11	20/2
	0.52	color	11	20/7
	0.54 (av of 2)	XRF	12	5/10
	0.54 (av of 2)	XRF	12	9/18
	0.53	NAA	15	6/24
	0.51	NAA	15	21/29
	0.53	color	19	20/1
	0.54	color	19	22/10
	*0.6263 (av of 2)	NAA	20	10/3
	*0.6087 (av of 2)	NAA	20	12/32
	*0.44	ES	21	10/13
	*0.44	ES	21	11/11
	*0.59	color	23	20/20
	0.55	NAA	23	20/20
	0.55	XRF	24	1/3
	0.54	XRF	24	4/22
	0.52±0.05 (mean)			
	0.53±0.02 (recommended value)			
MnO	0.1800	NAA	1	16/10
	0.1756	NAA	1	16/32
	0.2043 (av of 7)	ES	2	1/32
	0.2050 (av of 7)	ES	2	8/31
	0.2097 (av of 7)	ES	2	20/5
	*0.2213 (av of 5)	ES	2	20/3
	*0.1614 (av of 6)	NAA	4	3/15, 9/31
	*0.12	color	6	19/11
	*0.12	color	6	10/31
	0.2053 (av of 4)	NAA	7	7/10

TABLE 1.—Continued.

Constituent	Results	Method	Contributor	Split/ Position
	PERCENT			
	0.1999 (av of 2)	NAA	7	4/25
	0.192	NAA	8	6/25
	0.192	NAA	8	7/4
	0.20	color	11	20/2
	0.19	color	11	20/7
	0.21 (av of 2)	XRF	12	5/10
	0.21 (av of 2)	XRF	12	9/18
	0.187	NAA	15	6/24
	0.187	NAA	15	12/29
	0.18	color	19	20/1
	0.18	color	19	22/10
	0.1821 (av of 2)	NAA	20	10/3
	0.1801 (av of 2)	NAA	20	12/32
	0.193	color	23	20/20
	0.198	XRF	24	1/3
	0.199	XRF	24	4/22
	0.19±0.02 (mean)			
	0.19±0.01 (recommended value)			
MgO	24.50	grav	6	19/11
	24.92	grav	6	10/31
	*25.5	NAA	8	6/25
	24.2	NAA	8	7/4
	24.54	grav	11	20/2
	24.69	grav	11	20/7
	24.52 (av of 2)	XRF	12	5/10
	24.47 (av of 2)	XRF	12	9/18
	*23	NAA	15	6/24
	*23	NAA	15	12/29
	24.50	grav	19	20/1
	24.51	grav	19	22/10
	*25.65	grav	23	20/20
	24.79	XRF	24	1/3
	24.81	XRF	24	4/22
	24.51±0.72 (mean)			
	24.59±0.20 (recommended value)			
CaO	2.7	NAA	1	16/10
	2.5	NAA	1	16/32
	*2.71	grav	6	19/11
	2.58	grav	6	10/31
	*2.8	NAA	8	6/25
	2.5	NAA	8	7/4
	2.65	grav	11	20/2
	2.63	grav	11	20/7
	2.60 (av of 2)	XRF	12	5/10
	2.60 (av of 2)	XRF	12	9/18
	2.7	NAA	15	6/24
	2.5	NAA	15	12/29
	2.60	grav	19	20/1
	2.53	grav	19	22/10
	2.5 (av of 2)	NAA	20	10/3
	*2.4 (av of 2)	NAA	20	12/32
	2.58	grav	23	20/20
	2.56	XRF	24	1/3
	2.57	XRF	24	4/22
	2.59±0.09 (mean)			
	2.58±0.07 (recommended value)			
	PERCENT			
Na_2O	0.456	NAA	1	16/10
	0.453	NAA	1	16/32
	*0.42 (av of 2)	NAA	5	13/32
	0.46 (av of 2)	NAA	5	1/24
	0.47	flame	6	19/11
	0.47	flame	6	10/31
	*0.4966 (av of 4)	NAA	7	7/10
	0.4798 (av of 2)	NAA	7	4/25
	0.458	NAA	8	6/25
	0.457	NAA	8	7/4
	*0.43	flame	11	20/2
	*0.43	flame	11	20/7
	0.48 (av of 2)	flame	12	5/10
	0.48 (av of 2)	flame	12	9/18
	0.47	NAA	15	6/24
	0.47	NAA	15	12/29
	0.4489 (av of 3)	NAA	17	5/1
	0.4516 (av of 3)	NAA	17	8/27
	0.45	flame	19	20/1
	0.45	flame	19	22/10
	0.436 (av of 2)	NAA	20	10/3
	0.451 (av of 2)	NAA	20	12/32
	0.47	NAA	23	20/20
	0.46	XRF	24	1/3
	0.45	XRF	24	4/22
	0.46±0.02 (mean)			
	0.46±0.01 (recommended value)			
K_2O	0.036	NAA	1	16/10
	0.033	NAA	1	16/32
	0.048	flame	6	19/11
	0.05	flame	6	10/31
	0.034	NAA	8	6/25
	0.043	NAA	8	7/4
	0.03	flame	11	20/2
	0.03	flame	11	20/7
	0.04 (av of 2)	XRF	12	5/10
	0.04 (av of 2)	XRF	12	9/18
	0.04	NAA	15	6/24
	0.04	NAA	15	12/29
	0.0355 (av of 3)	NAA	17	5/1
	0.0348 (av of 3)	NAA	17	8/27
	0.03	flame	19	20/1
	0.03	flame	19	22/10
	*0.0265 (av of 2)	NAA	20	10/3
	*0.0265 (av of 2)	NAA	20	12/32
	0.046	NAA	23	20/20
	0.033	XRF	24	1/3
	0.034	XRF	24	4/22
	0.04±0.01 (mean)			
	0.04±0.01 (recommended value)			
P_2O_5	0.23	color	6	19/11
	0.23	color	6	10/31
	0.24	color	11	20/2
	0.25	color	11	20/7
	0.25 (av of 2)	XRF	12	5/10
	0.25 (av of 2)	XRF	12	9/18
	0.26	color	19	20/1

TABLE 1.—Continued.

Constituent	Results	Method	Contributor	Split/ Position
	PERCENT			
	0.26	color	19	22/10
	*0.21	color	23	20/20
	0.230	XRF	24	1/3
	0.244	XRF	24	4/22
	0.24±0.02 (mean)			
	0.24±0.01 (recommended value)			
Fe**	21.7	NAA	1	16/10
	22.1	NAA	1	16/32
	22.5 (av of 4)	NAA	5	13/32
	23.8 (av of 2)	NAA	5	1/24
	23.55 (av of 2)	titr	6	19/11
	*23.76 (av of 2)	titr	6	10/31
	22.99 (av of 4)	NAA	7	7/10
	22.52 (av of 2)	NAA	7	4/25
	22.1	NAA	8	6/25
	22.3	NAA	8	7/4
	23.53	titr	11	20/2
	23.62	titr	11	20/7
	23.45 (av of 2)	XRF	12	5/10
	23.57 (av of 2)	XRF	12	9/18
	23.5	NAA	15	6/24
	23.5	NAA	15	12/29
	23.56	titr	19	20/1
	23.50	titr	19	22/10
	26.7 (av of 2)	NAA	20	10/3
	25.9 (av of 2)	NAA	20	12/32
	23.16	titr	23	20/20
	23.67	XRF	24	1/3
	23.68	XRF	24	4/22
	23.55±0.16 (mean)			
	23.57±0.08 (recommended value)			
Ni**	1.3	NAA	1	16/10
	1.4	NAA	1	16/32
	1.43 (av of 2)	NAA	3	1/13
	1.45 (av of 2)	NAA	3	6/23
	*1.47	grav	6	19/11
	1.45	grav	6	10/31
	1.44	NAA	8	6/25
	1.52	NAA	8	7/4
	1.39	grav	11	20/2
	1.42	grav	11	20/7
	*1.27 (av of 2)	XRF	12	5/10
	*1.27 (av of 2)	XRF	12	9/18
	1.42	grav	19	20/1
	1.42	grav	19	22/10
	1.40	grav	23	20/20
	1.39±0.07 (mean)			
	1.42±0.02 (recommended value)			

** All NAA results for Fe and Ni excluded. See "Summary of Results."

Constituent	Results	Method	Contributor	Split/ Position
Co	0.0670	NAA	1	16/10
	0.0677	NAA	1	16/32
	0.0569 (av of 6)	ES	2	1/32
	0.0557 (av of 6)	ES	2	8/31
	0.0628 (av of 6)	ES	2	20/5

Constituent	Results	Method	Contributor	Split/ Position
	PERCENT			
	0.0639 (av of 5)	ES	2	20/3
	0.0606 (av of 4)	NAA	5	13/32
	0.0652 (av of 2)	NAA	5	1/24
	0.071	color	6	19/11
	*0.076	color	6	10/31
	0.0600 (av of 4)	NAA	7	7/10
	0.0579 (av of 2)	NAA	7	4/25
	0.0695	NAA	8	6/25
	0.0692	NAA	8	7/4
	0.06	color	11	20/2
	0.06	color	11	20/7
	0.0565 (av of 2)	XRF	12	5/10
	0.0567 (av of 2)	XRF	12	9/18
	0.0600	SSMS	15	6/24
	0.0610	SSMS	15	12/29
	0.0749 (av of 2)	NAA	20	10/3
	0.0728 (av of 2)	NAA	20	12/32
	0.074	ES	21	10/13
	0.070	ES	21	11/11
	0.069	NAA	23	20/20
	0.06±0.01 (mean)			
	0.06±0.01 (recommended value)			
S	*2.20	grav	6	19/11
	*2.01	grav	6	10/31
	2.14	grav	11	20/2
	2.13	grav	11	20/7
	2.08 (av of 2)	XRF	12	5/10
	2.11 (av of 2)	XRF	12	9/18
	*2.04	grav	19	20/1
	2.10	grav	19	22/10
	2.07	XRF	24	1/3
	2.07	XRF	24	4/22
	2.10±0.05 (mean)			
	2.10±0.03 (recommended value)			
H_2O	<0.1	Pnfld	11	20/2
	<0.1	Pnfld	11	20/7
	0.17	Pnfld	19	20/1
	0.16	Pnfld	19	22/10
	0.00	Pnfld	23	20/20
	0.096	Pnfld	24	1/3
	0.131	Pnfld	24	4/22
C	0.23	MS	9	4/11
	0.25	MS	9	8/23
	0.27	LECO	11	20/2
	0.26	LECO	11	20/7
	0.27	LECO	14	19/21
	*0.29	LECO	14	19/22
	0.22	grav	19	20/1
	0.22	grav	19	22/10
	0.28	grav	23	20/20
	0.25±0.03 (mean)			
	0.25±0.02 (recommended value)			
O	36.6 (av of 2)	NAA	5	13/32
	36.37 (av of 4)	NAA	22	19/14
	36.64 (av of 4)	NAA	22	8/26

TABLE 1.—Continued.

Constituent	Results	Method	Contributor	Split/ Position
	PARTS PER MILLION			
Li (3)	<1	ES	2	1/32
	<1	ES	2	8/31
	<1	ES	2	20/5
	<1	ES	2	20/3
	1.3	SSMS	15	6/24
	1.4	SSMS	15	12/29
	4	ES	21	10/13
	3 (av of 2)	ES	21	11/11
Be (4)	0.03	SSMS	15	6/24
	0.03	SSMS	15	12/29
B (5)	1.0	SSMS	15	6/24
	1.0	SSMS	15	12/29
	<5	ES	21	10/13
	<5	ES	21	11/11
F (9)	56	SSMS	15	6/24
	53	SSMS	15	12/29
Cl (17)	316	NAA	8	6/25
	265	NAA	8	7/4
Sc (21)	11.8	NAA	1	16/10
	12.3	NAA	1	16/32
	12 (av of 7)	ES	2	1/32
	11 (av of 6)	ES	2	8/31
	10 (av of 7)	ES	2	20/5
	11 (av of 7)	ES	2	20/3
	9.8 (av of 4)	NAA	4	3/15, 9/31
	10.7 (av of 4)	NAA	5	13/32
	12.0 (av of 2)	NAA	5	1/24
	10.9 (av of 4)	NAA	7	7/10
	10.1 (av of 2)	NAA	7	4/25
	12.0	NAA	8	6/25
	11.8	NAA	8	7/4
	10	NAA	15	6/24
	10	NAA	15	12/29
	12.1 (av of 2)	NAA	20	10/3
	11.9 (av of 2)	NAA	20	12/32
	10	NAA	23	20/20
	11±1 (mean)			
	11±1 (recommended value)			
V (23)	100	NAA	1	16/10
	*120	NAA	1	16/32
	84 (av of 4)	ES	2	1/32
	85 (av of 4)	ES	2	8/31
	84 (av of 4)	ES	2	20/5
	87 (av of 4)	ES	2	20/3
	93	NAA	8	6/25
	96	NAA	8	7/4
	91.4 (av of 2)	XRF	12	5/10
	91.6 (av of 2)	XRF	12	9/18
	*77	SSMS	15	6/24
	89	SSMS	15	12/29
	89	NAA	15	6/24
	89	NAA	15	12/29
	*113 (av of 2)	NAA	20	10/3
	*111 (av of 2)	NAA	20	12/32
	100	ES	21	10/13
	*73 (av of 2)	ES	21	11/11
	104 (av of 2)	ES	23	20/20
	94±12 (mean)			
	92±6 (recommended value)			
Cu (29)	137 (av of 10)	ES	2	1/32
	135 (av of 9)	ES	2	8/31
	151 (av of 10)	ES	2	20/5
	139 (av of 10)	ES	2	20/3
	115 (av of 6)	NAA	4	3/15, 9/31
	97	NAA	8	6/25
	101	NAA	8	7/4
	95.3 (av of 2)	XRF	12	5/10
	96.1 (av of 2)	XRF	12	9/18
	*230	NAA	15	6/24
	*230	NAA	15	12/29
	120	ES	21	10/13
	120	ES	21	11/11
	125 (av of 2)	ES	23	20/20
	135±44 (mean)			
	119±19 (recommended value)			
Zn (30)	112 (av of 2)	ES	2	1/32
	114 (av of 2)	ES	2	8/31
	114 (av of 2)	ES	2	20/5
	113 (av of 2)	ES	2	20/3
	112 (av of 3)	NAA	3	1/13
	116 (av of 3)	NAA	3	6/23
	*125 (av of 4)	NAA	4	3/15, 9/31
	107	NAA	8	6/25
	*77	NAA	8	7/4
	*130.5 (av of 2)	XRF	12	5/10
	*130.5 (av of 2)	XRF	12	9/18
	100	NAA	15	6/24
	100	NAA	15	12/29
	110	ES	21	10/13
	110	ES	21	11/11
	111±13 (mean)			
	110±5 (recommended value)			
Ga (31)	9.0	NAA	1	16/10
	7.0	NAA	1	16/32
	7 (av of 4)	ES	2	1/32
	7 (av of 4)	ES	2	8/31
	8 (av of 4)	ES	2	20/5
	8 (av of 4)	ES	2	20/3
	6.0 (av of 3)	NAA	3	1/13
	5.9 (av of 3)	NAA	3	6/23
	5.60 (av of 6)	NAA	4	3/15, 9/31
	6.0	NAA	8	6/25
	4.6	NAA	8	7/4
	5.4 (av of 2)	XRF	12	5/10
	5.5 (av of 2)	XRF	12	9/18
	5	NAA	15	6/24
	5	NAA	15	12/29
	*25	ES	21	10/31
	*23 (av of 2)	ES	21	11/11
	8±6 (mean)			
	6±1 (recommended value)			

TABLE 1.—Continued.

Constituent	Results	Method	Contributor	Split/ Position
	PARTS PER MILLION			
Ge (32)	17.6 (av of 3)	NAA	3	1/13
	17.9 (av of 3)	NAA	3	6/23
	11	SSMS	15	6/24
	11	SSMS	15	12/29
As (33)	1.9 (av of 4)	NAA	4	3/15, 9/31
	0.87	NAA	8	6/25
	0.94	NAA	8	7/4
	3	NAA	15	6/24
	3	NAA	15	12/29
Se (34)	10.5 (av of 4)	NAA	4	3/15, 9/31
	12.2	NAA	4	3/15
	7.4 (av of 2)	NAA	13	6/11
Br (35)	1.54 (av of 2)	NAA	17	5/1
	1.52 (av of 2)	NAA	17	8/27
Rb (37)	1.3	ES	2	1/32
	1.3	ES	2	8/31
	1.3	ES	2	20/5
	1.3	ES	2	20/3
	1.3 (av of 4)	NAA	4	3/15, 9/31
	*0.86	NAA	8	6/25
	*0.77	NAA	8	7/4
	1.32 (av of 2)	XRF	12	5/10
	1.26 (av of 2)	XRF	12	9/18
	1.2	SSMS	15	6/24
	*1.5	SSMS	15	12/29
	1.2	NAA	17	5/1
	1.2	NAA	17	8/27
	<5	ES	21	10/13
	<8 (av of 2)	ES	21	11/11
	1.0	XRF	24	1/3
	1.3	XRF	24	4/22
	1.2±0.2 (mean)			
	1.2±0.1 (recommended value)			
Sr (38)	13 (av of 3)	ES	2	1/32
	10 (av of 3)	ES	2	8/31
	11 (av of 3)	ES	2	20/5
	12 (av of 3)	ES	2	20/3
	<20	NAA	8	6/25
	<20	NAA	8	7/4
	14.69 (av of 2)	XRF	12	5/10
	14.71 (av of 2)	XRF	12	9/18
	*27	SSMS	15	6/24
	*27	SSMS	15	12/29
	8	ES	21	10/13
	8 (av of 2)	ES	21	11/11
	14.2	XRF	24	1/3
	14.1	XRF	24	4/22
	14±6 (mean)			
	12±3 (recommended value)			
Y (39)	<10	ES	2	1/32
	<10	ES	2	8/31
	<10	ES	2	20/5
	<10	ES	2	20/3
	3.2 (av of 2)	XRF	12	5/10
	PARTS PER MILLION			
	3.2 (av of 2)	XRF	12	9/18
	3.2	SSMS	15	6/24
	3.0	SSMS	15	12/29
	*2.3	SSMS	16	1/27
	3.0	SSMS	16	13/23
	2.9 (av of 2)	NAA	20	10/3
	3.0 (av of 2)	NAA	20	12/32
	≃3	XRF	24	1/3
	3.0±0.3 (mean)			
	3.1±0.1 (recommended value)			
Zr (40)	14.8	NAA	4	3/15
	6.63	NAA	5	13/32
	7.5 (av of 2)	XRF	12	5/10
	7.7 (av of 2)	XRF	12	9/18
	11	SSMS	15	6/24
	10	SSMS	15	12/29
	11	NAA	15	6/24
	11	NAA	15	12/29
	6.4	SSMS	16	1/27
	6.4	SSMS	16	13/23
	*48	ES	21	10/13
	*51 (av of 2)	ES	21	11/11
	8.3	XRF	24	1/3
	8.0	XRF	24	4/22
	15±15 (mean)			
	9±3 (recommended value)			
Nb (41)	<1 (av of 2)	XRF	12	5/10
	<1 (av of 2)	XRF	12	9/18
	0.72	SSMS	15	6/24
	0.76	SSMS	15	12/29
	0.48	SSMS	16	1/27
	0.53	SSMS	16	13/23
	<2.5	XRF	24	1/3
	<2.5	XRF	24	4/22
Mo (42)	2.5 (av of 4)	NAA	4	3/15, 9/31
	<2	ES	21	10/13
	<2 (av of 2)	ES	21	11/11
Ru (44)	0.85 (av of 4)	NAA	4	3/15, 9/31
Pd (46)	0.66 (av of 2)	NAA	13	6/11
	0.62 (av of 2)	NAA	13	13/20
Ag (47)	<1	ES	21	10/13
	<1 (av of 2)	ES	21	11/11
Cd (48)	0.433	NAA	3	1/13
	0.432 (av of 2)	NAA	3	6/23
	0.58 (av of 2)	NAA	20	10/3
	0.57	NAA	20	12/32
	<2	ES	21	10/13
	<2 (av of 2)	ES	21	11/11
In (49)	0.0289 (av of 3)	NAA	3	1/13
	0.0289 (av of 3)	NAA	3	6/23
	<0.01 (av of 4)	NAA	4	3/15, 9/31
	0.031	NAA	8	6/25
	*0.041	NAA	8	7/4

TABLE 1.—Continued.

Constituent	Results	Method	Contributor	Split/ Position
	PARTS PER MILLION			
	0.028	NAA	20	10/3
	0.027 (av of 2)	NAA	20	12/32
	0.031±0.005 (mean)			
	0.029±0.001 (recommended value)			
Sn (50)	0.30 (av of 4)	NAA	4	3/15, 9/31
	<20	ES	21	10/13
	<20	ES	21	11/11
Sb (51)	0.04 (av of 4)	NAA	4	3/15, 9/31
	0.085	NAA	15	6/24
	0.088	NAA	15	12/29
Te (52)	1.35	NAA	4	3/15
	1.1 (av of 2)	NAA	13	6/11
	1.1 (av of 2)	NAA	17	5/1
	1.1 (av of 2)	NAA	17	8/27
Cs (55)	<1	ES	2	1/32
	<1	ES	2	8/31
	<1	ES	2	20/5
	<1	ES	2	20/3
	0.10 (av of 4)	NAA	4	3/15, 9/31
	<0.1	NAA	8	6/25
	<0.1	NAA	8	7/4
	0.1	SSMS	15	6/24
	0.09	SSMS	15	12/29
	0.10	SSMS	16	1/27
	0.092 (av of 3)	NAA	17	5/1
	0.096 (av of 3)	NAA	17	8/27
Ba (56)	5 (av of 2)	ES	2	1/32
	4 (av of 2)	ES	2	8/31
	4 (av of 2)	ES	2	20/5
	4 (av of 2)	ES	2	20/3
	<10	NAA	8	6/25
	<10	NAA	8	7/4
	*12	SSMS	15	6/24
	*12	SSMS	15	12/29
	5.1	SSMS	16	1/27
	5.7	SSMS	16	13/23
	<2	ES	21	10/13
	<2 (av of 2)	ES	21	11/11
	3	XRF	24	1/3
	4	XRF	24	4/22
	6±3 (mean)			
	4±1 (recommended value)			
La (57)	0.48	NAA	1	16/10
	0.47	NAA	1	16/32
	0.56	NAA	4	3/15
	0.47	NAA	7	7/10
	*0.38	NAA	7	4/25
	0.46	NAA	8	6/25
	0.48	NAA	8	7/4
	0.56	SSMS	15	6/24
	0.56	SSMS	15	12/29
	0.6	NAA	15	6/24
	*0.7	NAA	15	12/29
	0.53	SSMS	16	1/27
	0.52	SSMS	16	13/23
	0.5121 (av of 2)	ID	18	2/20
	0.5149 (av of 2)	ID	18	5/4
	0.50 (av of 2)	NAA	20	10/3
	0.51 (av of 2)	NAA	20	12/32
	0.52±0.07 (mean)			
	0.52±0.04 (recommended value)			
Ce (58)	*1.45	NAA	1	16/10
	1.40	NAA	1	16/32
	1.32	NAA	4	3/15
	1.23	NAA	5	13/32
	1.21	NAA	8	6/25
	1.30	NAA	8	7/4
	1.2	SSMS	15	6/24
	1.3	SSMS	15	12/29
	*1	NAA	15	6/24
	*1	NAA	15	12/29
	1.43	SSMS	16	1/27
	*1.45	SSMS	16	13/23
	1.328 (av of 2)	ID	18	2/20
	1.335 (av of 2)	ID	18	5/4
	1.35 (av of 2)	NAA	20	10/3
	1.43 (av of 2)	NAA	20	12/32
	1.30±0.14 (mean)			
	1.33±0.08 (recommended value)			
Pr (59)	*0.26	NAA	4	3/15
	0.22	SSMS	15	6/24
	0.21	SSMS	15	12/29
	0.19	SSMS	16	1/27
	0.20	SSMS	16	13/23
	0.21 (av of 2)	NAA	20	10/3
	0.21 (av of 2)	NAA	20	12/32
	0.21±0.02 (mean)			
	0.21±0.01 (recommended value)			
Nd (60)	*0.92	SSMS	15	6/24
	0.94	SSMS	15	12/29
	1.00	SSMS	16	1/27
	0.99	SSMS	16	13/23
	1.009 (av of 2)	ID	18	2/20
	1.009 (av of 2)	ID	18	5/4
	1.01 (av of 2)	NAA	20	10/3
	*1.08 (av of 2)	NAA	20	12/32
	0.99±0.05 (mean)			
	0.99±0.03 (recommended value)			
Sm (62)	0.31	NAA	1	16/10
	0.35	NAA	1	16/32
	*0.18	NAA	4	3/15
	0.33 (av of 3)	NAA	7	7/10
	0.37 (av of 2)	NAA	7	4/25
	0.324	NAA	8	6/25
	0.336	NAA	8	7/4
	0.36	SSMS	15	6/24
	0.35	SSMS	15	12/29
	0.34	NAA	15	6/24

TABLE 1.—Continued.

Constituent	Results	Method	Contributor	Split/ Position
	PARTS PER MILLION			
	0.36	NAA	15	12/29
	0.30	SSMS	16	1/27
	0.32	SSMS	16	13/23
	0.3284 (av of 2)	ID	18	2/20
	0.3269 (av of 2)	ID	18	5/4
	0.334 (av of 2)	NAA	20	10/3
	0.361 (av of 2)	NAA	20	12/32
	0.33±0.04 (mean)			
	0.34±0.02 (recommended value)			
Eu (63)	0.106	NAA	1	16/10
	0.109	NAA	1	16/32
	0.136	NAA	4	3/15
	0.14 (av of 4)	NAA	7	7/10
	*0.15	NAA	8	6/25
	*0.16	NAA	8	7/4
	0.1	SSMS	15	6/24
	*0.09	SSMS	15	12/29
	0.1	NAA	15	6/24
	0.1	NAA	15	12/29
	0.10	SSMS	16	1/27
	0.11	SSMS	16	13/23
	0.1133 (av of 2)	ID	18	2/20
	0.1125 (av of 2)	ID	18	5/4
	0.116 (av of 2)	NAA	20	10/3
	0.116 (av of 2)	NAA	20	12/32
	0.12±0.02 (mean)			
	0.11±0.01 (recommended value)			
Gd (64)	*0.54	NAA	4	3/15
	0.42	SSMS	15	6/24
	0.42	SSMS	15	12/29
	*0.36	SSMS	16	1/27
	0.39	SSMS	16	13/23
	0.4134 (av of 2)	ID	18	2/20
	0.4050 (av of 2)	ID	18	5/4
	0.45 (av of 2)	NAA	20	10/3
	0.44 (av of 2)	NAA	20	12/32
	0.43±0.05 (mean)			
	0.42±0.02 (recommended value)			
Tb (65)	0.08	NAA	1	16/10
	0.08	NAA	1	16/32
	0.075	NAA	4	3/15
	0.090	NAA	8	6/25
	0.075	NAA	8	7/4
	0.077	SSMS	15	6/24
	0.075	SSMS	15	12/29
	0.1	NAA	15	6/24
	0.1	NAA	15	12/29
	0.07	SSMS	16	1/27
	0.07	SSMS	16	13/23
	0.078 (av of 2)	NAA	20	10/3
	0.084 (av of 2)	NAA	20	12/32
	0.081±0.010 (mean)			
	0.081±0.010 (recommended value)			
Dy (66)	0.408	NAA	4	3/15
	*0.34	NAA	8	6/25
	PARTS PER MILLION			
	0.38	NAA	8	7/4
	0.41	SSMS	15	6/24
	0.41	SSMS	15	12/29
	0.44	SSMS	16	1/27
	0.47	SSMS	16	13/23
	*0.5056	ID	18	2/20
	*0.5020	ID	18	5/4
	0.43 (av of 2)	NAA	20	10/3
	0.45 (av of 2)	NAA	20	12/32
	0.43±0.05 (mean)			
	0.42±0.03 (recommended value)			
Ho (67)	0.114	NAA	4	3/15
	0.088	NAA	8	6/25
	0.092	NAA	8	7/4
	*0.12	SSMS	15	6/24
	*0.12	SSMS	15	12/29
	0.1	NAA	15	6/24
	0.1	NAA	15	12/29
	0.09	SSMS	16	1/27
	0.10	SSMS	16	13/23
	0.114 (av of 2)	NAA	20	10/3
	0.113 (av of 2)	NAA	20	12/32
	0.10±0.01 (mean)			
	0.10±0.01 (recommended value)			
Er (68)	*0.344	NAA	4	3/15
	*0.27	SSMS	15	6/24
	0.28	SSMS	15	12/29
	0.28	SSMS	16	1/27
	0.28	SSMS	16	13/23
	0.3031	ID	18	2/20
	0.3031	ID	18	5/4
	0.30 (av of 2)	NAA	20	10/3
	0.30 (av of 2)	NAA	20	12/32
	0.30±0.02 (mean)			
	0.29±0.01 (recommended value)			
Tm (69)	0.053	SSMS	16	1/27
	0.064	SSMS	16	13/23
	0.056 (av of 2)	NAA	20	10/3
	0.055 (av of 2)	NAA	20	12/32
Yb (70)	0.26	NAA	1	16/10
	0.30	NAA	1	16/32
	*0.33	NAA	4	3/15
	*0.33	NAA	7	7/10
	*0.22	NAA	8	6/25
	*0.25	NAA	8	7/4
	*0.33	SSMS	15	6/24
	0.28	SSMS	15	12/29
	0.29	SSMS	16	1/27
	0.31	SSMS	16	13/23
	0.3161	ID	18	2/20
	0.3133	ID	18	5/4
	0.30 (av of 2)	NAA	20	10/3
	0.30 (av of 2)	NAA	20	12/32
	0.29±0.03 (mean)			
	0.30±0.02 (recommended value)			

TABLE 1.—Continued.

Constituent	Results	Method	Contributor	Split/ Position
	PARTS PER MILLION			
Lu (71)	*0.041	NAA	1	16/10
	0.048	NAA	1	16/32
	0.053	NAA	4	3/15
	*0.065	NAA	8	6/25
	*0.039	NAA	8	7/4
	0.06	NAA	15	6/24
	0.06	NAA	15	12/29
	0.05	SSMS	16	1/27
	0.06	SSMS	16	13/23
	0.0468	ID	18	2/20
	0.0462	ID	18	5/4
	0.048 (av of 2)	NAA	20	10/3
	0.050 (av of 2)	NAA	20	12/32
	0.051±0.008 (mean)			
	0.052±0.006 (recommended value)			
Hf (72)	*0.35	NAA	1	16/10
	*0.30	NAA	1	16/32
	*0.13	NAA	4	3/15
	0.201	NAA	5	13/32
	0.21	NAA	8	6/25
	0.24	NAA	8	7/4
	0.20	SSMS	15	6/24
	0.19	SSMS	15	12/29
	0.2	NAA	15	6/24
	0.2	NAA	15	12/29
	0.20	SSMS	16	1/27
	0.22	SSMS	16	13/23
	0.22±0.06 (mean)			
	0.21±0.01 (recommended value)			
W (74)	0.20	NAA	8	6/25
	0.29	NAA	8	7/4
	0.2	NAA	15	6/24
	0.2	NAA	15	12/29
Re (75)	0.020 (av of 4)	NAA	4	3/15, 9/31
	0.063 (av of 2)	NAA	13	6/11
	0.064 (av of 2)	NAA	13	13/20
Os (76)	0.40 (av of 4)	NAA	4	3/15, 9/31
	0.73 (av of 2)	NAA	13	6/11
	0.77 (av of 2)	NAA	13	13/20
Ir (77)	0.80 (av of 3)	NAA	3	1/13
	0.83 (av of 2)	NAA	3	6/23
	*0.51 (av of 4)	NAA	4	3/15, 9/31
	0.6 (av of 2)	NAA	5	13/32
	0.6 (av of 2)	NAA	5	1/24
	*0.89 (av of 2)	NAA	7	7/10
	0.79	NAA	8	6/25
	0.76	NAA	8	7/4
	0.79 (av of 2)	NAA	13	6/11
	PARTS PER MILLION			
	0.74	NAA	13	13/20
	0.73±0.12 (mean)			
	0.74±0.09 (recommended value)			
Au (79)	*0.22	NAA	1	16/10
	*0.25	NAA	1	16/32
	0.14	NAA	3	1/13
	0.17	NAA	3	6/23
	0.15 (av of 4)	NAA	4	3/15, 9/31
	0.14 (av of 6)	NAA	4	3/15, 9/31
	0.16	NAA	7	7/10
	*0.11 (av of 2)	NAA	13	6/11
	0.13 (av of 2)	NAA	13	13/20
	0.16±0.04 (mean)			
	0.15±0.01 (recommended value)			
Hg (80)	0.035 (av of 4)	NAA	4	3/15, 9/31
Pb (82)	1.138	ID	10	4/26
	1.538	ID	10	9/14
	1.6	SSMS	15	6/24
	1.0	SSMS	15	12/29
	1.57	SSMS	16	1/27
	1.49	SSMS	16	13/23
	*12	ES	21	10/31
	*12 (av of 2)	ES	21	11/11
	4.04±4.92 (mean)			
	1.39±0.25 (recommended value)			
Bi (83)	0.028 (av of 2)	NAA	13	6/11
	<2	ES	21	10/13
	<2 (av of 2)	ES	21	11/11
Th (90)	0.5	NAA	1	16/10
	0.5	NAA	1	16/32
	<0.3	NAA	8	6/25
	<0.3	NAA	8	7/4
	2.2 (av of 2)	XRF	12	5/10
	1.6 (av of 2)	XRF	12	9/18
	0.070 (av of 2)	NAA	13	6/11
	0.063	SSMS	16	1/27
	0.063	SSMS	16	13/23
	PARTS PER BILLION			
Tl (81)	53.1	ID	10	4/26
^{204}Pb	20.30	ID	10	4/26
	26.24	ID	10	9/14
U (92)	<0.07	NAA	8	6/25
	<0.07	NAA	8	7/4
	16 (av of 2)	NAA	17	5/1
	16	NAA	17	8/27

TABLE 2.—Summary of the analysis of variance for 48 elements based on 75 replicate analyses (mean = average of replicate analyses as given in contributed paper in percent or ppm; conclusion (at 95% confidence level): NS = not significant (homogeneous); S = significant (heterogeneous); contributor = contributed paper as listed in Contents; method = analytical method using abbreviations as listed in Table 1).

Constituent	Mean	Conclusion	Contributor	Method
	PERCENT			
SiO_2	34.09	NS	12	XRF
TiO_2	0.15	NS	12	XRF
Al_2O_3	3.22	NS	12	XRF
	3.25	NS	20	NAA
Cr_2O_3	0.534	NS	2	ES
	0.54	NS	12	XRF
	0.62	NS	20	NAA
MnO	0.206	NS	2	ES
	0.21	NS	12	XRF
	0.181	NS	20	NAA
MgO	24.49	NS	12	XRF
CaO	2.60	NS	12	XRF
	2.5	NS	20	NAA
Na_2O	0.44	NS	5	NAA
	0.48	NS	12	flame
	0.450	NS	17	NAA
	0.443	NS	20	NAA
K_2O	0.04	NS	12	XRF
	0.035	NS	17	NAA
	0.027	NS	20	NAA
P_2O_5	0.25	NS	12	XRF
Fe	23.51	NS	12	XRF
Ni	1.27	NS	12	XRF
Co	0.0584	NS	2	ES
	0.0566	NS	12	XRF
	0.0738	NS	20	NAA
S	2.09	NS	12	XRF
O	36.50	S	22	NAA
	PARTS PER MILLION			
Sc	11	NS	2	ES
	12.0	NS	20	NAA
V	85	NS	2	ES
	91.5	NS	12	XRF
	112	NS	20	NAA
Cu	142	NS	2	ES
	95.7	NS	12	XRF
Zn	113	NS	2	ES
	PARTS PER MILLION			
	130.5	NS	12	XRF
Ga	7	NS	2	ES
	5.4	NS	12	XRF
Br	1.53	NS	17	NAA
Rb	1.29	NS	12	XRF
Sr	12	NS	2	ES
	14.70	NS	12	XRF
Y	3.2	NS	12	XRF
	3.0	NS	20	NAA
Zr	7.6	NS	12	XRF
Pd	0.64	NS	13	NAA
Te	1.1	NS	17	NAA
Cs	0.094	S	17	NAA
Ba	4	NS	2	ES
La	0.5135	NS	18	ID
	0.50	NS	20	NAA
Ce	1.331	NS	18	ID
	1.39	S	20	NAA
Pr	0.21	NS	20	NAA
Nd	1.009	NS	18	ID
	1.05	NS	20	NAA
Sm	0.3276	NS	18	ID
	0.347	NS	20	NAA
Eu	0.1129	NS	18	ID
	0.116	NS	20	NAA
Gd	0.4092	NS	18	ID
	0.44	NS	20	NAA
Tb	0.081	S	20	NAA
Dy	0.44	NS	20	NAA
Ho	0.113	NS	20	NAA
Er	0.30	NS	20	NAA
Tm	0.055	NS	20	NAA
Yb	0.30	NS	20	NAA
Lu	0.049	NS	20	NAA
Re	0.063	NS	13	NAA
Os	0.75	NS	13	NAA
Ir	0.6	NS	5	NAA
Au	0.12	NS	13	NAA
Th	1.9	NS	12	XRF

1. Trace Element Analyses of the Allende Meteorite Reference Sample by Neutron Activation

Ralph O. Allen, Jr.

Two splits of the Allende meteorite reference sample were analyzed for 22 major, minor, and trace elements by instrumental neutron activation analysis. A 500 mg sample of each specimen in a polyethylene vial was irradiated for 1 minute in the University of Virginia nuclear reactor at a flux of $\sim 10^8$ neutrons cm^{-2} sec^{-1}. The γ-ray spectra were taken within minutes of the irradiation with a 20 cm^3 Ge(Li) detector and a 512-channel analyzer for the analysis of Al, Ca, and V. The samples were counted about 8 hours after the irradiation for the determination of Mn and Na. This procedure is similar to that described by Wakita et al. (1970).

The same samples were then irradiated for 2 hours at a flux of $\sim 10^{13}$ neutrons cm^{-2} sec^{-1} along with aqueous solutions of the standards. Each polyethylene vial was wrapped with a weighed iron wire. The specific activities of these iron wires were used to correct for any variations in flux between the vials. After allowing the radioactive samples to cool for ~12 hours, they were transferred to new polyethylene vials for counting. Gamma-ray spectra were taken with either a 40 cm^3 or a 70 cm^3 Ge(Li) detector coupled to a 4096-channel analyzer. The counting procedure was similar to that described by Gordon et al. (1968). The following elements were measured: Fe, Na, K, Ni, Mn, Cr, Co, Sc, V, Hf, Au, Ga, Th, La, Ce, Sm, Eu, Tb, Yb, and Lu. For most elements two or more photopeaks were used for the calculations and in some cases two different isotopes were used.

The results are summarized in Table 3. Sample B-10 is Split 16/Position 10 and sample B-11 is Split 16/Position 32 of the Allende meteorite reference sample. Sample B-9 is Split 7/Position 4 of the USGS Standard BCR-1, which was analyzed at the same time as the Allende samples. Within the analytical uncertainties there is no great difference between the two Allende samples.

TABLE 3.—Neutron activation analyses of two subsamples of the Allende meteorite reference sample (B-10 = Split 16/Position 10; B-11 = Split 16/Position 32) and USGS Reference Sample BCR-1 (dash indicates not determined).

Constituent	B-10 Allende 16/10	B-11 Allende 16/32	B-9 BCR-1 7/4
PERCENT			
Fe_2O_3*	31.0±0.5	31.6±0.5	3.13±0.06
Na_2O	0.456±0.001	0.453±0.009	3.30±0.03
K_2O	0.036±0.006	0.033±0.006	1.68±0.03
Al_2O_3	3.21±0.05	3.36±0.15	13.6±0.3
CaO	2.7±0.3	2.5±0.4	7.0±0.6
Ni	1.3±0.1	1.4±0.1	–
PARTS PER MILLION			
Mn	1394±27	1360±30	1352±30
Cr	3670±63	3560±52	16±4
Co	670±10	677±10	38.7±0.8
Sc	11.8±0.2	12.3±0.4	38.6±0.6
V	100±40	120±30	440±50
Hf	0.35±0.15	0.30±0.15	4.1±0.3
Au	0.22±0.05	0.25±0.5	–
Ga	9.0±0.7	7.0±0.7	21.8±1.4
Th	0.5±0.2	0.5±0.2	7.8±1.4
La	0.48±0.04	0.47±0.04	24.2±0.2
Ce	1.45±0.14	1.40±0.10	52.5±2.8
Sm	0.31±0.04	0.35±0.04	6.95±0.05
Eu	0.106±0.007	0.109±0.007	2.10±0.05
Tb	0.08±0.02	0.08±0.02	1.15±0.10
Yb	0.26±0.03	0.30±0.03	3.44±0.10
Lu	0.041±0.008	0.048±0.006	0.523±0.010

* Total Fe reported as Fe_2O_3.

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2. Emission Spectrographic Analyses of Trace Elements in the Allende Meteorite Reference Sample

C.S. Annell

Emission spectrographic analyses were performed for 14 elements in four splits of the Allende meteorite reference sample. Each sample split was quartered twice and one quarter of the original sample was ground in an agate mortar. A 200 mg portion of each finely ground sample was mixed with 50 mg of graphite powder by grinding together in an agate mortar, and the mixtures were stored in polyethylene capsules.

Three methods of d.c. arc emission spectroscopy were used. *Method 1*: A 15 A arc in air vaporized a 25 mg sample-graphite mixture to completion. First order spectra from 2300 to 4800 Å were photographed using a 3.4 m Ebert spectrograph. The spectra were examined for 38 elements. *Method 2*: A 25 A arc in an atmosphere of argon was used selectively to volatilize and determine nine elements: Ag, Au, Bi, Cd, Ge, In, Pb, Tl, and Zn. To do this, a 25 mg sample-graphite was spectrochemically buffered with 30 mg of Na_2CO_3. Second order spectra in the 2400–3650 Å region were recorded with a 3 m Eagle spectrograph. *Method 3*: A 12.5 mg portion of the sample-graphite mixture, buffered with 20 mg of K_2CO_3, was vaporized in a 15 A arc in air for the determination of Cs, Rb, and Li. The 3 m Eagle spectrograph was used to record first order spectra in the 6500–9000 Å region.

Based on data for the general composition of the Allende meteorite, a matrix of high Si, Fe, and Mg containing proportionate amounts of Al, Ca, Na, and Ti as oxides or carbonates was prepared and sintered. This matrix was used for dilution of other standards and mixtures to provide spectra and inter-element reactions comparable to those for meteorites.

For those elements determined by emission spectroscopy (Method 1), a coefficient of variation of 15% of the amount present is assigned. Methods 2 and 3, specifically designed for a selective group of elements, both have a coefficient of variation of 10% of the amount present.

The results of the analyses are listed in Table 4. The following elements were looked for but not detected; if present, their concentrations are below those indicated in parenthesis (in ppm): Ag (0.2), As (4), Au (0.2), B (10), Bi (1), Cd (8), Ce (100), Cs (1), Ge (1), Hf (20), Hg (8), In (1), Mo (2), Nd (100), P (2000), Pb (1), Pt (3), Re (30), Sb (100), Sn (10), Ta (100), Te (300), Th (100), Tl (1), U (500), and W (200).

TABLE 4.—Emission spectrographic analyses of trace elements (ppm) in four subsamples from the Allende meteorite reference sample (elements listed in order of decreasing volatility in the d.c. arc.).

Constituent	Split/Position							
	1/32		8/31		20/5		20/3	
Zn	120	103	110	117	110	117	115	110
Cu	113	170	150	123	185	125	165	125
	120	160	121	143	165	173	120	143
	150	133	135	175	120	175	135	185
	125	106	106	135	123	172	103	105
	150	145	127		162	106	157	155
Ga	6	8	8	7	7	7	7	8
	7	8	7	7	8	8	8	8
Cs	<1		<1		<1		<1	
Rb	1.3		1.3		1.3		1.3	
Li	<1		<1		<1		<1	
Mn	1425	1500	1425	1470	1500	1600	1550	1720
	1500	1550	1500	1720	1450	1820	1400	2300
	1650	1950	1400	1900	1300	2150	1600	
	1500		1700		1550			
Cr	3700	3200	4400	3250	3600	4200	3800	3650
	3300	3250	3250	4350	3950	3300	3950	3500
	3350	3800	3300	3550	3350	3750	3550	3800
	4200	3900	3800		3800	3000	4200	3150
	4500				4000		3000	
Co	615	675	575	700	810	650	780	675
	535	590	530	510	775	510	725	510
	525	475	515	510	515	505	505	
Ba	5	4	4	4	4	4	4	4
Sr	14	12	11	10	13	9	12	12
	12		10		10		13	
V	65	90	68	87	78	83	73	88
	90	90	94	91	93	82	99	87
Sc	11	11	12	9	11	8	10	9
	12	12	13	10	10	14	11	12
	11	12	9	10	10	10	14	11
	15				10		12	
Y	<10		<10		<10		<10	

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3. Abundances of Eight Elements in the Allende Meteorite Reference Sample Determined by Neutron Activation Analysis

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Two splits of the Allende meteorite reference sample were analyzed for Ni, Zn, Ga, Ge, Ir, Au, Cd and In by radiochemical neutron activation analysis. Approximately 500 mg aliquots of each sample were packaged in fused silica vials and irradiated along with appropriate flux monitors in the Ames Laboratory reactor at a flux of 10^{13} neutrons cm^{-2} sec^{-1} for 66 hours. After cooling for 4 days the samples were re-irradiated in the UCLA reactor for 3 hours at a flux of 2×10^{11} neutrons cm^{-2} sec^{-1} to produce the short-lived activities that are utilized in our procedure. Details of our radiochemical separation procedure can be found in Baedecker et al. (1971), Baedecker et al. (1972), Baedecker et al. (1973), Müller et al. (1971), and Wasson and Baedecker (1970).

The precision (one standard deviation on a single determination) of our method based on replicate analyses of Allende and Orgueil meteorites is Ni, ±3%; Zn, ±9%; Ga, ±4%; Ge, ±6%; Cd, ±4%; In, ±4%; Ir, ±4%; Au, ±12%. The accuracy of our results was evaluated by Chou et al. (1976) based on a comparison of our Allende and Orgueil data with those of other laboratories for the same meteorites. Our data for Ni, Zn, Ge, Cd, and In fell within ±5% of the values obtained by other workers. Much larger interlaboratory biases were observed for Au and Ir, and although our results were bracketed by those of other groups, the accuracy of our data could not be adequately assessed. Our data for the Allende meteorite reference sample (previously published by Chou et al., 1976) are reported in Table 5. A combined mean as well as means for the two separate bottles are reported. The error limits reported are 95% confidence limits on the mean. The agreement between the bottles is well within our estimated error limits and shows no indication of inhomogeneity for the reference material for any of the elements determined, in spite of substantial variations between bulk Allende and that meteorite's spheroidal Ca-Al-rich inclusions (Chou et al., 1976).

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TABLE 5.—Radiochemical neutron activation analyses of eight trace elements in two subsamples of the Allende meteorite reference sample (dash indicates only one reading obtained).

Constituent	Split 1/ Position 13	Mean	Split 6/ Position 23	Mean	Overall mean
PARTS PER THOUSAND					
Ni	14.0	14.3	14.0	14.5	14.4±0.8
	14.6		15.0		
PARTS PER MILLION					
Zn	107	112	125	116	114±14
	98		103		
	132		121		
Ga	6.2	6.0	5.7	5.9	5.9±0.3
	6.2		6.2		
	5.6		5.7		
Ge	16.9	17.6	16.9	17.9	17.8±0.8
	18.1		18.3		
	17.8		18.6		
Ir	0.78	0.80	0.80	0.83	0.81±0.04
	0.81		0.86		
	0.80		–		
Au	0.14	0.14	0.17	0.17	0.16
PARTS PER BILLION					
Cd	433	433	429	432	432±7
In	27.0	28.9	27.5	28.9	28.9±5.1
	29.5		30.4		
	30.4		28.7		

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4. Determination of Trace Elements by Neutron Activation Analysis in the Allende Meteorite Reference Sample

R. Becker, P. Koller, P. Morschl, W. Kiesl, and F. Hermann

Trace elements in two splits of the Allende meteorite reference sample were determined by neutron activation analysis (NAA) followed by chemical separations of the elements under consideration. Final counting of nuclides was carried out by γ-ray spectrometry, using NaI(Tl) or Ge(Li) detectors and an Intertechnique Company multichannel analyzer.

Rare earth elements (REE) were determined by irradiating 1 g of Split 3/Position 15 in the TRIGA Mark II reactor for 7 hours at a thermal neutron flux of 10^{13} neutrons cm^{-2} sec^{-1}. The sample was then treated with HF, HNO_3, and $HClO_4$ for 2 hours in a teflon beaker at 250° C. ^{144}Ce was added for yield determination and 400 μg of a REE-mixture as a carrier. A multi-step group separation (Becker et al., 1974) was used, and the separated REE-solution as well as the standards were counted at several different decay times using a 40 cm^3 Ge(Li) detector.

For Zr and Hf, 500 mg samples of Split 3/Position 15 were irradiated together with standards for 2 hours in the TRIGA Mark II reactor. The basic principle was the determination of Zr via its daughter product ^{97}Nb, which originates from ^{97}Zr. ^{97}Nb was isolated by selective extraction with methylisobutylketone (MIBK) in an aqueous solution of HF-$HClO_4$. Hf was determined using its isotope ^{181}Hf after two extractions with thenoyltrifluoracetone (TTA) (Koller, 1971). Nuclides were counted by means of a 3 × 3 inch NaI(Tl) crystal.

The 300 mg of Split 3/Position 15 was sealed in a quartz vial for the determination of Se and Te. The sample together with standards was irradiated for 3 days in the core of the ASTRA reactor using a flux of 4×10^{13} neutrons cm^{-2} sec^{-1}. Te was determined indirectly via the 8.05-day ^{131}I daughter of 1.2-day ^{131m}Te and 25-min ^{131}Te. The iodine was oxidized and extracted into CCl_4, stripped, and precipitated as AgI for yield determination. Se was precipitated

TABLE 6.—Neutron activation analyses of trace elements (ppm) in the Allende meteorite reference sample. Means of values obtained from Split 9/Position 31 and Split 3/Position 15 are reported as there was almost no deviation of concentrations between splits.

Constituent	Mean values
	Single Analysis (3/15)
La	0.56
Ce	1.32
Pr	0.26
Sm	0.18
Eu	0.136
Gd	0.54
Tb	0.075
Dy	0.408
Ho	0.114
Er	0.344
Yb	0.33
Lu	0.053
Zr	14.8
Hf	0.13
Se	12.2
Te	1.35
	4 Analyses (3/15)
Mn	1250±90
Cu	115±12
Ga	5.60±0.45
Au	0.14±0.02
	6 Analyses (3/15 and 9/31)
Se	10.5±1.0
As	1.9±0.2
Sb	0.04±0.01
Sn	0.30±0.1
Re	0.020±0.002
Hg	0.035±0.004
Au	0.15±0.01
Mo	2.5±0.2
Os	0.40±0.08
Ru	0.85±0.08
Zn	125±6
Ir	0.51±0.1
Sc	9.8±0.9
Rb	1.3±0.3
Cs	0.10±0.02
In	<0.01

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by $Na_2S_2O_5$, filtered, washed, and dried. The samples were measured 6 days after the end of irradiation on a 3 × 3 inch Na(Tl) crystal (Morschl, 1972).

For Mn, Cu, Au, and Ga, 100 mg of Split 3/Position 15 was sealed in a quartz vial. The sample and standards were simultaneously irradiated for 7 minutes in the core of the ASTRA reactor using a flux of 3×10^{13} neutrons cm^{-2} sec^{-1}. After removal of silica with HNO_3 and HF and dissolution of insoluble fluorides by fuming with H_2SO_4, the elements were separated (Morschl, 1972) on an anion-exchange column and measured by means of a 3 × 3 inch NaI(Tl) crystal.

Each 300–500 mg of Split 9/Position 31 and Split 3/Position 15 was irradiated with standards for 5 days in the ASTRA reactor for the determination of Se, As, Sb, Sn, Re, Hg, Au, Mo, Os, Ru, Cr, Sc, Cs, Rb, Zn, In, and Ir. After a cooling period of 1 day the elements were separated by distillation and ion exchange steps according to an analytical separation scheme that has been developed at the Analytical Institute of the University of Vienna (Hermann et al., 1971). The radiochemical determinations of the nuclides were carried out by using a 40 cm^3 Ge(Li) detector and a 400-channel γ-spectrometer (Intertechnique Company Selecteur Sa-40). Results of these determinations are given in Table 6.

5. Some Elemental Abundances in the Allende Meteorite Reference Sample Determined by Neutron Activation Analysis

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Eleven major, minor, and trace elements were determined using 14 MeV and thermal neutron activation analysis. O and Si were determined by 14 MeV instrumental neutron activation analysis (INAA), according to the method of Morgan and Ehmann (1970). The data presented here are based on six replicate O and Si analyses of each powder aliquant. The remaining elements were determined by reactor INAA, or RNAA, employing high resolution Ge(Li) spectrometry. The RNAA determinations of Zr and Hf are based on a radiochemical separation procedure by Kumar et al. (1977). All Zr data are corrected for the U fission contribution to the indicator radionuclide ^{95}Zr. Primary standards were used for most of the elements determined in this study, complemented by USGS Standard Rock BCR-1 as a secondary standard.

Data for the 11 elements determined in this study are presented in Table 7. The lettered columns present data obtained on separate powder aliquants of each split/position sample. Means for each split/position and the overall means are also given. The data for Fe exhibit an appreciable variation among the several aliquants analyzed. However, there appears to be a positive correlation between the Fe and Co data, which would suggest that the variation noted here is due to inhomogeneity with respect to the metallic phase content in the ~0.3 g aliquants used for these determinations. Only a single analysis is reported for both Zr and Hf in Split 13/Position 32. However, we have independently (Kumar et al., 1977) determined Zr and Hf abundances in eight powder splits derived from a bulk specimen

TABLE 7.—Some elemental abundances in the Allende meteorite reference sample (dash indicates not reported).

Constituent	Estimated analytical rel. error* (%)	Split 13/Position 32					Split 1/Position 24			Overall mean for both splits
		A	B	C	D	Mean	A	B	Mean	
					PERCENT					
O	1–2	36.8	36.4	–	–	36.6±0.2	–	–	–	36.6
Si	2	16.6	16.5	–	–	16.6±0.1	–	–	–	16.6
Fe	3–5	23.4	23.1	22.2	21.2	22.5±0.5	24.0	23.6	23.8±0.2	23.2
Cr	3–5	–	–	0.35	0.34	0.34±0.01	0.38	0.37	0.38±0.01	0.36
Na	2–3	–	–	0.32	0.30	0.31±0.01	0.35	0.34	0.34±0.01	0.33
					PARTS PER MILLION					
Co	5	601	619	614	590	606±7	664	640	652±12	629
Sc	5	11.3	11.1	10.0	10.4	10.7±0.3	12.7	11.3	12.0±0.7	11.2
Zr**	5	6.63	–	–	–	6.63	–	–	–	6.63
Ce**	5–10	1.23	–	–	–	1.23	–	–	–	1.23
Ir	10–15	–	–	0.6	0.6	0.6	0.7	0.6	0.6±0.1	0.6
Hf**	5–8	0.201	–	–	–	0.201	–	–	–	0.201

* Typical values based on experience in our laboratory.

** Data obtained on a fifth aliquant of Split 13/Position 32, but are here listed in the column for aliquant A.

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of Allende from our research collection. The values of Zr = 6.29±0.29 (1σ) ppm and Hf = 0.200±0.014 (1σ) ppm obtained from these bulk sample splits are in good agreement with the standard reference powder data presented here. These Zr data are lower than we previously reported (Morgan et al., 1969) for several bulk Allende samples. As noted in later work from our laboratory (Ehmann and Chyi, 1974), laboratory glassware contaminated with the highly charged Zr^{4+} and Hf^{4+} ions could introduce low levels of contamination, which would appreciably alter the results obtained for relatively small meteorite samples that contain very low concentrations of Zr and Hf. Results obtained for typically larger samples of standard rocks with much higher Zr and Hf contents would not be significantly affected. Segregation of expensive glassware which must be reused into categories according to expected Zr and Hf abundance levels and extensive cleaning procedures have eliminated these problems, which apparently affected a number of the earlier low level RNAA studies.

6. Bulk Chemical Analyses of the Allende Meteorite Reference Sample

C.J. Elliott

Bulk chemical analyses of Split 19/Position 11 and Split 10/Position 31 of the Allende meteorite reference sample were performed employing the chlorination technique for separation of the phases and classical chemical methods. It was intended to analyze the meteoritic splits by the method of Moss et al. (1967) but a magnetic separation could not be achieved, apparently owing to the fineness of the powders and the intimate mixing of the minerals. Accordingly, the meteorite powders as received were chlorinated, and the resulting modifications to the method are indicated in the scheme given in Table 8.

A good separation of metal and sulfides from the silicate seems to have been achieved since only 0.12% Ni (calculated on the whole meteorite) was detected in the insoluble residue. This result indicates also that the amounts of awaruite and taenite, which are not attacked by chlorine, are small. Chlorination did, however, release 3% of the total MgO. The reason for this is not clear unless it can be shown that some of the magnesium-bearing silicate phases are attacked

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TABLE 8.—Flow chart for chemical analyses of the Allende meteorite reference sample.

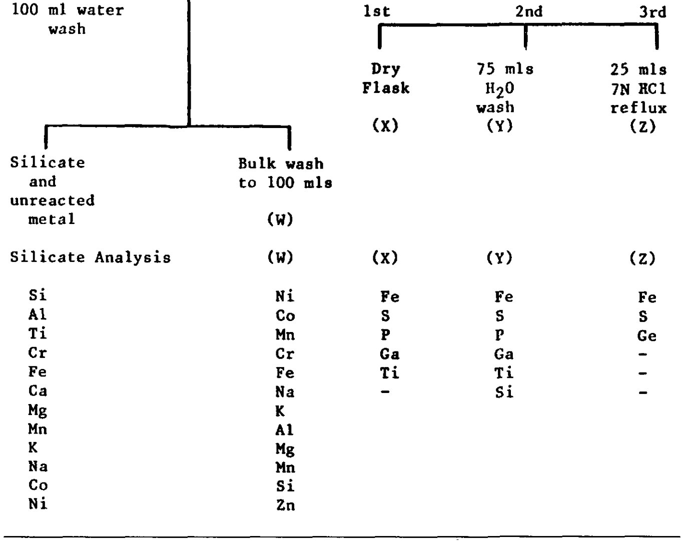

TABLE 9.—Bulk chemical analyses and analyses of elements in silicate, sulfide, and metal phases of the Allende meteorite reference sample.

Constituent	Split 19/ Position 11	Split 10/ Position 31
	PERCENT	
SiO_2	34.15	34.48
TiO_2	0.16	0.17
Al_2O_3	3.18	3.19
Cr_2O_3	0.43	0.45
FeO	22.45	22.81
MnO	0.12	0.12
MgO	24.50	24.92
CaO	2.71	2.58
Na_2O	0.47	0.47
K_2O	0.048	0.05
P_2O_5	0.23	0.23
Fe	6.17	6.28
Ni	1.47	1.45
Co	0.071	0.076
S	2.20	2.01
Total	98.36	99.29
	SULFIDE AND METAL PHASES (ppm)	
Cu	68.0	76
Zn	14	9
Cr	13	19
Mn	300	230
Ti	10	8
P	18	13
Ge	≪0.9	≪0.5
Si	<85	≪47

TABLE 10.—Wahl norms for bulk chemical analyses of the Allende meteorite reference sample.

Constituent	Split 19/ Position 11	Split 10/ Position 31	Jarosewich (1970:45, col. 3)
Olivine	70.02	71.20	78.20
Hypersthene	4.38	4.70	.00
Diopside	3.86	3.32	3.20
Perovskite	0.27	0.29	0.26
Anorthite	6.43	6.44	6.95
Chromite	0.63	0.64	0.76
Nepheline	2.32	2.32	2.16
Chlorapatite	0.55	0.55	0.55
Iron			1.17

by chlorine; previous analytical work of Moss et al. (1967) has established that chlorine does not release Mg from olivine-hypersthene and olivine-bronzite chondrites.

The bulk compositions of the meteorite powders are given in Table 9, together with the proportions of trace elements in the metal and sulfide phases. Trace elements in the silicate phase have not been determined because of the analytical difficulties encountered. However, work is in hand to establish the mechanism of the reaction between chlorine and the silicate phases of this meteorite.

If our results are compared with those of Jarosewich (in Clarke et al., 1970), it will be seen that, whereas the determinations of total iron content are in agreement, the distribution of this iron is different. The absence of hypersthene and the excess FeO in the norm indicate that the FeO content of Jarosewich's analysis is too high (Table 10).

No explanation can be advanced for the low summation of the analysis of Split 19/Position 11; repeat analyses did not reveal any major discrepancy in the composition of the silicate.

7. Instrumental Neutron Activation Analysis of the Allende Meteorite Reference Sample

Lawrence Grossman and D.P. Kharkar

We have used instrumental neutron activation analysis to determine the concentrations of twelve elements in two splits of the Allende meteorite reference sample—Split 4/Position 25 and Split 7/Position 10. The abundances of Mn, Na, Sm, La, Co, Fe, Sc, and Cr were determined in both bottles. In addition, Yb, Eu, Ir, and Au were determined in only one bottle. Slight sample heterogeneities within a single bottle were indicated by the Co, Fe, and Sc analyses. No significant differences between the two bottles were observed for any of the elements determined in both.

For the analyses, the powdered samples were further ground in an agate mortar and four 25 or 50 mg portions of Split 7/Position 10 and two 50 mg portions of Split 4/Position 25 along with five duplicate silicate standards were accurately weighed into quartz vials, which were previously cleaned by boiling in hot concentrated HNO_3. Duplicate Au and Ir standards were prepared by pipetting standard solutions of these elements onto crushed quartz substrates inside quartz vials of the same size as those used for the samples and evaporating to dryness. A "blank" quartz vial was also included with the samples and standards. The vials were heat-sealed and packed radially in an aluminum cylinder and irradiated in the Union Carbide "swimming pool" reactor at Sterling Forest, New York.

The irradiation times and counting sequence used here are similar to those described by Turekian and Kharkar (1970) and Kharkar and Turekian (1971). The first irradiation lasted 30 minutes in a highly thermalized portion of the reactor at a flux of about 4.5×10^{12} neutrons cm^{-2} sec^{-1}. Three hours after removal from the pile the vials were unpacked, washed with HNO_3, and counted vertically in a bored lucite holder resting on the top of a 25 cm^3 coaxial Ge(Li) detector coupled to a 2048-channel analyzer. All spectra were punched onto paper tape for data reduction by an IBM 7094–7040 computer. In all the counting, the distance from the bottom of the vials to the detector was adjusted with lucite blocks so as to give a maximum dead time of 14%–16% for the "hottest" sample.

Each sample and standard and the blank vial were counted for 200 seconds or 500 seconds for the first analysis to measure ^{56}Mn ($t_{1/2}$ = 2.58 hr). The samples were then allowed to "cool" for a day to reduce the background interference of ^{56}Mn and other short-lived activities and then each was counted for 2000 seconds to measure ^{24}Na ($t_{1/2}$ = 15 hr).

After the determination of Mn and Na, the samples, standards, and blank were repacked and sent for a three-day irradiation at a flux of 2×10^{13} neutrons cm^{-2} sec^{-1}. The samples, standards, and blank, after another acid wash, were counted for 5000 seconds each from 5 to 8 days after removal from the pile for short-lived nuclides (^{140}La, ^{153}Sm, ^{175}Yb, ^{198}Au). Then the samples were re-counted 13 days later to obtain data for long-lived nuclides (^{152}Eu, ^{60}Co, ^{59}Fe, ^{46}Sc, ^{51}Cr, ^{192}Ir). The counting times were 5000 or 10,000 seconds for each sample, 5000 or 20,000 seconds for each standard, and 5000 seconds for the blank.

The nuclides, γ-ray peaks, and half-lives of elements analyzed and their concentrations in the standards chosen for each element are shown in Table 12. The concentrations of Eu, Co, Fe, Sc, and Cr are based on the "best" values in

TABLE 11.—Elemental abundances in the Allende meteorite reference sample. (Values in ppm unless otherwise indicated; errors due to counting statistics are Mn (0.5–1.0%), Na (0.6–1.0%), Sm (13–14%), Yb (95%), La (14–18%), Eu (60–93%), Co (0.2%), Fe (0.4%), Sc (0.8%), Cr (2%), Ir (3.7%), Au (6.9%); ND = not determined).

Constituent	Split 7/ Position 10	Split 4/ Position 25
Mn	1564, 1590, 1574, 1633	1533, 1564
Na	3668, 3608, 3686, 3774	3556, 3563
Sm	0.22, 0.36, 0.41	0.27, 0.46
Yb	0.35, 0.30	ND
La	0.47	0.38
Eu	0.15, 0.17, 0.12, 0.13	ND
Co	622, 611, 578, 590	570, 587
Fe(%)	23.28, 22.89, 22.69, 23.10	22.24, 22.80
Sc	11.6, 11.5, 10.2, 10.4	10.0, 10.2
Cr	3308, 3205, 3137, 3238	3140, 3220
Ir	0.87, 0.91	ND
Au	0.16	ND

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TABLE 12.—Nuclides counted and standards used for each element in neutron activation analyses of the Allende meteorite sample (SP = standard pottery (Perlman and Asaro, 1969); dash indicates either no data or not repeated).

Constituent	Product nuclide	γ-ray peak (keV)	Half-life (hrs)	Best values for standard chosen (ppm)				
				BCR-1	SP	W-1	G-1	GSP-1
Mn	^{56}Mn	874	2.58	1320	–	1320	–	317.5
Na	^{24}Na	1369	15.00	24,000	2610	–	24,200	20616
Sm	^{153}Sm	103	47.00	–	5.78	–	–	–
Yb	^{175}Yb	396	101.04	–	2.80	–	–	–
La	^{140}La	1596	40.22	25.20	44.90	–	104	–
Eu	^{152}Eu	1408	108624	2.26	1.43	–	–	2.79
Co	^{60}Co	1332	45991	35.15	13.10	–	–	–
Fe	^{59}Fe	1100	1080	92955	9286	73300	–	30200
Sc	^{46}Sc	889	2016	33.10	19.85	–	–	–
Cr	^{51}Cr	320	667	–	114	–	–	–
Ir	^{192}Ir	468	1776	–	–	–	–	–
Au	^{198}Au	412	64.8	–	–	–	–	–

the silicate standards as determined by Katz and Grossman (1976). For the remaining elements, the data for the Standard Pottery were taken from Perlman and Asaro (1969) and the data for the rest of the standards from the compilations of Fleischer (1969) and Flanagan (1969).

The values obtained for Allende samples are shown in Table 11, along with 1σ uncertainties due to counting statistics.

The 317 keV peak of ^{192}Ir interferes with the 320 keV peak of ^{51}Cr. Using the spectrum of our Ir monitor, we found that 10–12% of the counts under the Cr peak were due to the presence of Ir in Allende and the Cr counts were corrected accordingly.

For each of the elements Co, Fe, and Sc, the concentration range determined within a single bottle exceeds that expected on the basis of 2σ counting statistics. We attribute these wide ranges to sample heterogeneity resulting probably from the small sample sizes employed here.

The test for a difference between the means of the determinations in material from Split 7/Position 10 and that from Split 4/Position 25 failed at the 95% confidence level for Mn, Sm, Co, Fe, Cr, Sc, and Na, the elements for which at least duplicate determinations are available for each split. For the remaining elements, La, Eu, Yb, Ir, and Au, insufficient data were available to evaluate the similarity of the two bottles.

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8. Neutron Activation Analyses of Seven Elements in the Allende Meteorite Reference Sample

K.S. Heier, A.O. Brunfelt, E. Steinnes, and B. Sundvoll

Elemental abundances were obtained by neutron activation analysis (Brunfelt et al., 1977). The samples were determined relative to USGS standard sample BCR-1. The assigned values are listed in Table 13.

TABLE 13.—Bulk concentrations of 38 elements in the Allende meteorite reference sample (Al corrected for contribution from the $^{28}Si(n,p)^{28}$ Al reaction).

Constituent	Split 6/ Position 25	Split 7/ Position 4	Constituent	Split 6/ Position 25	Split 7/ Position 4
	PERCENT			PARTS PER MILLION	
Na	0.340	0.339	Rb	0.86	0.77
Mg	15.4	14.6	Sr	<20	<20
Al	1.80	1.78	In	0.031	0.041
K	0.028	0.036	Cs	<0.1	<0.1
Ca	2.0	1.8	Ba	<10	<10
Ti	0.10	0.08	La	0.46	0.48
Cr	0.361	0.369	Ce	1.21	1.30
Mn	0.149	0.149	Sm	0.324	0.336
Fe	22.1	22.3	Eu	0.15	0.16
Ni	1.44	1.52	Tb	0.090	0.075
	PARTS PER MILLION		Dy	0.34	0.38
			Ho	0.088	0.092
Cl	316	265	Yb	0.22	0.25
Sc	12.0	11.8	Lu	0.065	0.039
V	93	96	Hf	0.21	0.24
Co	695	692	Ta	<0.1	<0.1
Cu	97	101	W	0.20	0.29
Zn	107	77	Ir	0.79	0.76
Ga	6.0	4.6	Th	<0.3	<0.3
As	0.87	0.94	U	<0.07	<0.07

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9. Determination of $\delta^{13}C$ and Total Noncarbonate Carbon in Two Splits of the Allende Meteorite Reference Sample by Mass Spectrometry

J.M. Herndon and W.M. Sackett

Two splits from the Allende meteorite reference sample were analyzed for $\delta^{13}C$ and total noncarbonate carbon by mass spectrometry. The values reported are expressed in terms of

$$\left[\delta^{13}C = \frac{(^{13}C/^{12}C \text{ sample}}{(^{13}C/^{12}C) \text{ standard}} - 1\right] \times 1000$$

relative to the Chicago PDB material used by Craig (1953). The measurements were actually made relative to NBS carbon isotope reference sample #21. A value of −28.25 ‰ relative to Chicago PDB standard was used to obtain the results reported here. The total percent noncarbonate carbon was determined from the volume of CO_2 produced after sample preparation.

About 0.8 g of each sample was treated with 3M HCl and evaporated to dryness; the residue was heated in a closed system to 850° C with pure O_2 in the presence of CuO catalyst. SO_2 was removed by passing the gases over silver wool at elevated temperatures. The CO_2 was collected in a trap at liquid nitrogen temperature. Isotopic measurements were made with a nuclide model RMS-6-60 isotope ratio mass spectrometer (Nuclide Associates, State College, Pennsylvania) equipped with a tungsten ribbon (0.001 × 0.003 inch) electron impact source. This instrument is equipped with dual, room temperature, balanced capillary inlets for sample and reference, with switching between the samples accomplished by a solenoid. Before each run, small adjustments in the inlet pressure are made with a mercury piston to give identical nuclide electrometer amplifier voltages for the sample reference. The ratio of the ion current of the beam focused on the Faraday cup to that falling on the collector plates is read from the 4-dial General Radio 1454 A Kelvin-Varley voltage divider with the last digit in the ratio interpolated from a recorder tracing of the remaining imbalance from the null. The vibrating reed electrometer is operated to measure the approach to null on the 100 mV full scale range. A separate voltage divider is used for the reference sample and is switched into the circuit when the sample and reference compounds are alternated. The switching time interval is approximately 2 minutes. Identical ratios are obtained on the reference when the divider is used and also when the reference is introduced from both sides.

TABLE 14.—Mass spectrometric determination of $\delta^{13}C$ and total noncarbonate carbon in the Allende meteorite reference sample. $\delta^{13}C$ reported relative to Chicago PDB standard (Craig, 1953).

Constituent	Split 4/ Position 11	Split 8/ Position 23
$\delta^{13}C$ vs. PDP (ppt)	−18.4±0.1	−17.9±0.1
Total noncarbonate carbon (%)	0.23±0.04	0.25±0.04

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10. Mass Spectrometric Isotope Dilution Analysis of Lead and Thallium in the Allende Meteorite Reference Sample

J.M. Huey and T.P. Kohman

The determination of lead and thallium in the Allende meteorite reference sample was performed by the mass spectrometric isotope dilution technique. In the first sample, Split 4/Position 26 (our No. M-85), both elements were determined by volatilization in our standard heating schedule. In the second sample, Split 9/Position 14 (our No. M-86), several volatile fractions were collected at different temperatures and analyzed separately and the total contents determined by addition. The data on elementary contents are summarized in Table 15. We have listed total Pb and ^{204}Pb separately because the latter is the nonradiogenic isotope and indicates primordial lead. We feel that the differences between the two samples are much greater than the uncertainties of measurement and are real sample differences.

TABLE 15.—Lead and thallium in the Allende meteorite reference sample (ND = not determined).

Constituent	Split 4/ Position 26	Split 9/ Position 14
Total Pb (ppm)	1.138±0.014	1.538±0.014
^{204}Pb (ppb)	20.30±0.25	26.24±0.23
Tl (ppb)	53.1±0.8	ND

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11. Bulk Chemical Analysis of the Allende Meteorite Reference Sample

Eugene Jarosewich

Bulk chemical analysis of two splits of the Allende meteorite reference sample was performed in essentially the same manner as described earlier (Jarosewich, 1966) with one modification. The sample for major and minor elements was not separated for analysis into acid soluble and acid insoluble portions.

About 1 g of powdered sample was used for the determination of major and some minor elements. Since the sample contains small quantities of metal, it was first treated with dilute HCl to remove the metal and prevent its reaction with the platinum crucible. The undissolved portion was fused with Na_2CO_3 and the melt dissolved in HCl and combined with the earlier HCl filtrate. This solution was then analyzed for silicon, aluminum, iron, calcium, magnesium, and titanium employing modified classical methods of Hillebrand et al. (1953), Peck (1964), and Sandell (1959).

Silica was separated from the HCl solution by double dehydration and filtration and determined by evaporation with HF. From the filtrate the R_2O_3 group was precipitated twice with NH_4OH. The precipitate was dissolved in dilute H_2SO_4 and evaporated to fumes and the residual silica separated. An aliquot of the R_2O_3 solution was used in the determination of iron by reduction in a silver reductor and titration with $K_2Cr_2O_7$. A second aliquot, from which interfering elements were removed by a mercury cathode, was used for the determination of aluminum and titanium. From one portion of this solution aluminum was precipitated with 8-hydroxyquinoline; a second portion was used for the colorimetric determination of titanium employing Tiron. Manganese was removed with zirconyl chloride from the filtrate obtained after separation of the R_2O_3 group (Peck, 1964), and from the resulting filtrate calcium was doubly precipitated with oxalic acid and magnesium with dibasic ammonium phosphate.

For the determination of nickel, sulfur, phosphorus, cobalt, and manganese, 1 g of sample was dissolved in bromine water and HNO_3 and treated with HF to remove silica. The residue was filtered off, fused with Na_2CO_3, and combined with the main solution. Nickel was precipitated with dimethylglyoxime and sulfur with $BaCl_2$ after reduction of Fe^{3+} with zinc in HCl solution. Phosphorus was determined colorimetrically with molybdovanadate, chromium with diphenylcarbazide, cobalt with nitroso-R-salt, and manganese as permanganate.

Sodium and potassium were determined flame photometrically after dissolving a 100 mg sample in HF and H_2SO_4. Combined water was determined by the Penfield method, using PbO and $PbCrO_4$ as a flux. Carbon was determined employing a LECO carbon analyzer. Metallic iron was dissolved with $HgCl_2$–NH_4Cl and titrated with $K_2Cr_2O_7$.

The results of the two splits and their averages are given in Table 16, as well as the bulk composition derived by assignment of appropriate oxidation states.

TABLE 16.—Bulk chemical analysis (percent) of the Allende meteorite reference sample (* = calculated on the basis of microprobe data for metallic phase: 68% Ni, 31% Fe, and 1.6% Co; metallic Fe was determined chemically and metallic Ni and Co assigned on the basis of this analysis; remaining Ni and Co were calculated as sulfides; remaining S was calculated as FeS; remaining Fe was calculated as FeO).

Constituent	Split 20/ Position 2	Split 22/ Position 7	Average	Bulk composition
SiO_2	34.11	34.26	34.19	34.19
TiO_2	0.16	0.16	0.16	0.16
Al_2O_3	3.27	3.29	3.28	3.28
Cr_2O_3	0.54	0.52	0.53	0.53
FeO				26.84
MnO	0.20	0.19	0.20	0.20
MgO	24.54	24.69	24.62	24.62
CaO	2.65	2.63	2.64	2.64
Na_2O	0.43	0.43	0.43	0.43
K_2O	0.03	0.03	0.03	0.03
P_2O_5	0.24	0.25	0.25	0.25
$H_2O(+)$	<0.1	<0.1	<0.1	<0.1
$H_2O(-)$	0.01	0.02	0.02	0.02
C	0.27	0.26	0.27	0.27
S	2.14	2.13	2.14	
FeS				4.11*
NiS				1.73*
CoS				0.08*
Fe	0.15	0.11	0.13	0.13
Ni	1.39	1.42	1.41	0.29*
Co	0.06	0.06	0.06	0.01*
Total				99.80
Total Fe	23.53	23.62	23.58	23.58

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12. X-Ray Fluorescence Spectrometric Analysis of the Allende Meteorite Reference Sample

M.J. Kaye and B.W. Chappell

Two splits of the Allende Meteorite Reference Sample have been analyzed in duplicate for major, minor, and trace elements by x-ray fluorescence spectrometry. Methods of analysis are essentially similar to those used by us for lunar samples (Compston et al., 1970). Details of techniques are set out more fully here.

Trace elements were determined by measuring emission of characteristic radiation from 1.5 g of sample and correcting for matrix effects by either measuring or calculating mass absorption coefficients (Norrish and Chappell, 1977). Absorption coefficients were measured directly for Rb Kα and Sr Kα radiations (Table 17), after which the powdered material was recovered and used in emission measurements. The Sr Kα coefficient was used for Sr and extrapolated for Y, Zr, and Nb measurements; the Rb Kα coefficient was used for Rb, Th, Ni, Cu, Zn, and Ga measurements. For elements emitting at longer wavelengths than the Fe Kα absorption edge (V, Cr, Co), a calculated Fe Kα absorption coefficient was used. This was based on the major element analysis and the mass absorption coefficient data of Heinrich (1966), a correction factor (× 0.929) being applied, based on the difference between measured and calculated absorption coefficients in terrestrial basalts.

Pellets for emission measurements were prepared using the method of Norrish and Chappell (1977). Boric acid *pellets, produced in this way, do not allow complete sample* recovery. For other less abundant meteorite samples and for lunar samples we prepare emission samples in a plastic covered Mylar sleeve as described by Compston et al. *(1970). The present method is capable of producing slightly* more reproducible results.

Intensities were corrected for nonlinearity of background using pellets of Spex Al_2O_3 and SiO_2 and Herasil glass (Chappell et al., 1969) and for spectral overlap (Norrish and Chappell, 1977). Calibrations were made against synthetic standards prepared by grinding pure salts or oxides with quartz (Compston et al., 1970). All measurements were made on a Philips PW 1220 spectrometer; instrumental conditions are given in Table 20.

Trace element results are given in Table 19. Stated precision limits represent 95% confidence based on variation in observed emission measurements. Data for the USGS standard reference basalt BCR-1, obtained concurrently, are also listed.

TABLE 17.—Mass absorption coefficients of the Allende meteorite reference sample for Rb Kα and Sr Kα radiations (error limits represent 95 percent confidence of mean of four mass absorption measurements; split numbers are those of authors).

Constituent	No. 138 Split 5/Position 10	No. 274 Split 9/Position 18
Rb	25.65±0.35	25.64±0.22
Sr	22.04±0.29	22.01±0.21

TABLE 18.—X-ray spectrometric analysis (percent) of Allende meteorite reference sample (total excludes H_2O and C; split numbers are those of authors; a and b are duplicate analyses of each split).

Constituent	No. 138 Split 5/ Position 10 (a)	No. 138 Split 5/ Position 10 (b)	No. 274 Split 9/ Position 18 (a)	No. 274 Split 9/ Position 18 (b)	Average of 4 analyses
SiO_2	*34.10*	*34.04*	*34.13*	*34.10*	*34.09*
TiO_2	0.15	0.15	0.15	0.15	0.15
Al_2O_3	3.24	3.20	3.20	3.25	3.22
FeO	30.33	30.00	30.34	30.31	30.25
MnO	0.21	0.21	0.21	0.21	0.21
MgO	*24.46*	*24.57*	*24.51*	*24.42*	*24.49*
CaO	2.60	2.59	2.60	2.60	2.60
Na_2O	0.48	0.48	0.48	0.48	0.48
K_2O	0.04	0.03	0.03	0.04	0.04
P_2O_5	0.25	0.25	0.25	0.25	0.25
S	2.06	2.09	2.11	2.11	2.09
Cr_2O_3	0.54	0.54	0.54	0.54	0.54
NiO	1.62	1.62	1.62	1.62	1.62
Sum of trace elements as oxides	0.12	0.12	0.12	0.12	0.12
Less O = S	1.03	1.04	1.05	1.05	1.04
Total	99.17	98.85	99.24	99.15	99.11

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Sodium was determined on 0.1 g of sample by flame photometry using a lithium internal standard. Other major and minor elements were measured on fused samples by x-ray fluorescence spectrometry using the specific technique of Norrish and Hutton (1969). Additional sodium nitrate was added to give the equivalent of 10% Na_2O and 90% Allende powder in a 0.28 g sample. This was necessary to achieve oxidation and retention of sulfur during fusion. Instrumental conditions corresponded to those of Norrish and Hutton (1969) (Table 20), except that a LiF(200) crystal was used for potassium measurements, and Mg, Al, Si, P, and S were measured with x-ray tube operating at 2 kW.

Calibration was made against the USGS rock standards GSP-1, AGV-1, BRC-1, PCC-1, and W-1. These had been calibrated previously using synthetic primary standards. Trace element results are given in Table 19 and major element results in Table 18.

TABLE 19.—Trace element results (in ppm) for the Allende meteorite reference sample and USGS BCR-1 reference basalt (error limits represent 95 percent confidence of mean; split numbers are those of authors; a and b are duplicate analyses of each split).

Constituent	No. 138 Split 5/Position 10 (a)	No. 138 Split 5/Position 10 (b)	No. 247 Split 9/Position 18 (a)	No. 247 Split 9/Position 18 (b)	Mean	BCR-1
Rb	1.30±0.08	1.34±0.10	1.26±0.10	1.26±0.10	1.29	46.7
Sr	14.65±0.13	14.72±0.14	14.56±0.14	14.85±0.10	14.70	329
Th	3.0±1.0	1.4±0.7	1.4±0.5	1.7±0.8	1.9	5.7
Zr	7.3±0.4	7.6±0.4	7.6±0.5	7.8±0.3	7.6	186
Nb	<1	<1	<1	<1	<1	11
Y	3.1±0.1	3.2±0.1	3.1±0.1	3.2±0.1	3.2	33
V	91.7±0.7	91.1±0.9	91.1±0.6	92.0±0.6	91.5	376
Co	565±2	564±2	567±3	567±2	566	38
Cu	95.3±1.1	95.2±1.1	96.6±0.9	95.5±0.6	95.7	14
Zn	130.9±0.7	130.0±0.9	130.8±0.5	130.2±0.8	130.5	123
Ga	5.5±0.4	5.3±0.2	5.4±0.3	5.5±0.4	5.4	21

TABLE 20.—Instrumental conditions for x-ray emission measurements of trace elements in the Allende meteorite reference sample. Conditions refer to Philips PW 1220 spectrometer; pulse height selector set to 1–2 V window for flow proportional counter (FPC) and 0.8–2.2 V for scintillation counter (SC); cycles refer to multiples of 200 second counts on peak and 100 second counts on each background position.

Element	Line	X-ray tube target	kV, mA	Collimator (μm)	Crystal	Detector	Backgrounds (°2θ)	Cycles
V	Kα	W	55,36	160	LiF(220)	FPC	±1.75	10
Cr	Kα	W	55,36	160	LiF(200)	FPC	±0.90	10
Co	Kα	W	55,36	480	LiF(200)	SC	±0.40	10
Ni	Kα	Au	62,32	480	LiF(200)	SC	±0.40	10
Cu	Kα	Au	62,32	480	LiF(200)	SC	±0.40	10
Zn	Kα	Au	62,32	480	LiF(200)	SC	±0.40	10
Ga	Kα	Mo	100,20	480	LiF(200)	SC	±0.40	35
Rb	Kα	Mo	100,20	480	LiF(200)	SC	±0.30	50
Sr	Kα	Mo	100,20	480	LiF(200)	SC	±0.35	35
Y	Kα	Mo	100,20	480	LiF(200)	SC	±0.40	35
Zr	Kα	W	100,20	480	LiF(200)	SC	±0.40	40
Nb	Kα	W	100,20	160	LiF(200)	SC	±0.35	40
Th	Lα_1	Mo	100,20	160	LiF(200)	SC	±0.23	15

13. Neutron Activation Analysis of Some Trace Elements in the Allende Meteorite Reference Sample

J.F. Lovering and R.R. Keays

Two splits from the Allende meteorite reference sample were analyzed for nine trace elements by neutron activation analysis. Results are summarized in Table 21.

TABLE 21.—Neutron activation analysis of the Allende meteorite reference sample (dash indicates results not reported).

	Split 6/Position 11		Split 13/Position 20	
Constituent	Aliquot 1	Aliquot 2	Aliquot 1	Aliquot 2
	PARTS PER MILLION			
Re	0.063±0.001	0.062±0.001	0.064±0.001	0.063±0.001
Os	0.76±0.01	0.70±0.01	0.79±0.01	0.75±0.01
Ir	0.80±0.01	0.78±0.01	0.74±0.01	-
Au	0.10±0.01	0.12±0.01	0.10±0.01	0.15±0.01
Pd	0.59±0.01	0.72±0.01	0.56±0.01	0.67±0.01
	PARTS PER BILLION			
Se	7600	7200	-	-
Te	1100	1100	-	-
Th	77	63	-	-
Bi	27	29	-	-

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14. Carbon Analysis of the Allende Meteorite Reference Sample by Standard Rapid Combustion Method

Carleton B. Moore

Carbon analysis was performed on two splits of the Allende meteorite reference sample employing the standard rapid combustion method with a LECO carbon apparatus: for Split 19/Position 21, C = 0.26_8%; for Split 19/Position 22, C = 0.28_5%.

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15. Multielement Analyses of the Allende Meteorite Reference Sample by Neutron Activation and Spark Source Mass Spectrometry

G.H. Morrison, N.M. Potter, A.M. Rothenberg, E.V. Gangadharam, and S.F. Wong

Multielement analyses were performed for 45 elements on two splits of the Allende meteorite reference sample employing neutron activation analysis (NAA) (Morrison et al., 1969) and spark source mass spectrometry (SSMS) (Morrison and Kashuba, 1969). These techniques were applied earlier in the analyses of USGS rock standards (Morrison et al., 1969; Morrison and Kashuba, 1969) and lunar samples, meteorites, and tholeiites (Morrison et al., 1970a, 1970b; Morrison et al., 1971).

Two 5 g aliquots of powdered meteorite were received bearing the identification numbers: Split 6/Position 24 and Split 12/Position 29. For NAA, duplicate 200 mg samples of each split were weighed directly into suitable containers for irradiation. For SSMS, 2 g aliquots of each split were ground in an agate ball and mortar mill for 4 hours, and two 1 g aliquots of each were mixed in 1:1 ratio with National Carbon Company ultrapure graphite. The mixes were briquetted into disks, which were quartered, and the quarters used as electrodes in the mass spectrometer.

For nondestructive NAA, the 200 mg aliquots of samples were placed in clean polyethylene vials and irradiated in the Cornell TRIGA Mark II reactor successively at the following flux levels: 6×10^{10} neutrons cm^{-2} sec^{-1} for 45 seconds for Al and V; 2×10^{11} neutrons cm^{-2} sec^{-1} for 40 seconds for Mg; and 2×10^{12} neutrons cm^{-2} sec^{-1} for 90 seconds for Mn. Following these irradiations and gamma-spectrometric measurement, the same samples were sealed in high purity quartz ampules and irradiated at a flux of 3.5×10^{12} neutrons cm^{-2} sec^{-1} for 8.5 hours for radiochemical NAA involving chemical group separations after a decay of 15 hours. Gamma-spectrometry was employed for measurement of the radionuclides.

The counting system consisted of a 30 cm^3 coaxial Ge(Li) detector (Nuclear Diodes) coupled to a 4096-channel analyzer (Northern Scientific). The spectra were read directly onto computer-compatible magnetic tape, which was then processed by an IBM 360/65 computer. Details of the activation procedures have been given by Morrison et al. (1969).

For mass spectrometry, the electrodes were sparked in a GRAF-2 double focusing mass spectrograph (Nuclide Associates). Different settings of magnet currents and charge collections were used to reduce interferences. USGS standard diabase W-1 was used as a comparative standard, and sensitivity factors were calculated for use in computing the quantitative results. A Jarrell-Ash microdensitometer was used to read the photoplates and the data were processed on a PDP-11 computer.

Table 22 lists the results obtained from duplicate analyses of each of the two splits received. The means and average deviations (AD) for each split are presented, as well as an overall mean and relative standard deviation (RSD) based on all values for each element.

With regard to the precision of the measurements, the average RSD for the 26 elements determined by NAA is 6%; for the 28 elements determined by SSMS it is 7%. Only Be, Cs, Ce, Eu, Lu, W, and Pb have RSDs above 10% and these can be attributed to analytical problems associated with their determinations. Since the reproducibility of most of the elements determined is within the 10% precision capability of the techniques employed, results for the duplicate analyses of each split as well as the results for both splits are essentially the same, so that the material can be considered homogeneous.

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TABLE 22.—Results of duplicate analyses on two samples of the Allende meteorite reference sample (Split 6/Position 24, Split 12/Position 29) (AD = average deviation; RSD = relative standard deviation).

Constituent	Method	Mean Split 6	AD (%)	Mean Split 12	AD (%)	Overall mean	RSD (%)
			PERCENT				
MAJORS							
Na	NAA	0.35	3.7	0.35	4.3	0.35	4.7
Mg	NAA	14	1.8	14	1.6	14	2.3
Al	NAA	1.9	1.6	1.9	3.4	1.9	3.1
K	NAA	0.03	3.2	0.03	0	0.03	3.6
Ca	NAA	1.9	5.2	1.8	2.7	1.9	5.9
Ti	NAA	0.09	0.33	0.08	2.4	0.09	4.3
Cr	NAA	0.36	0.01	0.35	0.56	0.35	0.51
Mn	NAA	0.145	0.24	0.145	1.1	0.145	0.96
Fe	NAA	23.5	1.1	23.5	3.0	23.5	2.5
			PARTS PER MILLION				
TRACE ELEMENTS							
Li	SSMS	1.3	0.80	1.4	1.1	1.3	6.8
Be	SSMS	0.03	14	0.03	9.1	0.03	14
B	SSMS	1.0	5.5	1.0	8.0	1.0	7.8
F	SSMS	56	0.09	53	1.3	55	3.3
Sc	NAA	10	2.4	10	3.4	10	3.4
V	SSMS	77	7.0	89	4.8	83	11
	NAA	89	4.6	89	3.5	89	4.7
Co	SSMS	600	0.08	610	1.3	610	1.7
Cu	NAA	230	1.3	230	0.22	230	1.2
Zn	NAA	100	5.0	100	1.6	100	4.3
Ga	NAA	5	1.3	5	2.0	5	2.7
Ge	SSMS	11	2.3	11	3.1	11	3.8
As	NAA	3	3.4	3	10	3	8.6
Rb	SSMS	1.2	3.3	1.5	1.7	1.4	11
Sr	SSMS	27	1.1	27	3.7	27	3.3
Y	SSMS	3.2	2.7	3.0	6.0	3.1	6.1

Constituent	Method	Mean Split 6	AD (%)	Mean Split 12	AD (%)	Overall mean	RSD (%)
			PARTS PER MILLION				
Zr	SSMS	11	2.8	10	5.2	11	5.0
	NAA	11	0.05	11	–	11	0.095
Nb	SSMS	0.72	0.69	0.76	0.86	0.74	2.8
Sb	NAA	0.085	4.3	0.088	0.11	0.086	4.1
Cs	SSMS	0.1	0	0.09	18	0.1	19
Ba	SSMS	12	2.1	12	2.5	12	2.6
La	SSMS	0.56	1.4	0.56	1.4	0.56	1.7
	NAA	0.6	3.3	0.7	10	0.6	5.6
Ce	SSMS	1.2	5.2	1.3	1.2	1.2	6.6
	NAA	1	14	1	7.5	1	14
Pr	SSMS	0.22	8.8	0.21	0	0.22	8.0
Nd	SSMS	0.92	1.1	0.94	1.2	0.93	1.7
Sm	SSMS	0.36	1.6	0.35	2.1	0.36	3.0
	NAA	0.34	1.6	0.36	7.9	0.35	6.9
Eu	SSMS	0.1	4.5	0.09	11	0.1	11
	NAA	0.1	15	0.1	3.0	0.1	16
Gd	SSMS	0.42	1.3	0.42	1.7	0.42	1.7
Tb	SSMS	0.077	0.65	0.075	2.7	0.076	2.3
	NAA	0.1	0.49	0.1	0.91	0.1	7.3
Dy	SSMS	0.41	6.5	0.41	3.1	0.41	5.9
Ho	SSMS	0.12	8.3	0.12	4.2	0.12	8.0
	NAA	0.1	6.2	0.1	1.8	0.1	5.6
Er	SSMS	0.27	1.1	0.28	11	0.28	9.1
Yb	SSMS	0.33	0.15	0.28	1.4	0.30	10
Lu	NAA	0.06	18	0.06	13	0.06	18
Hf	SSMS	0.20	2.3	0.19	2.9	0.19	3.5
	NAA	0.2	–	0.2	–	0.2	12
W	NAA	0.2	4.6	0.2	17	0.2	14
Pb	SSMS	1.6	2.1	1.0	6.7	1.3	27

16. Spark Source Mass Spectrometer Analysis of the Allende Meteorite Reference Sample

P.E. Muir, S.R. Taylor, and Brian Mason

Two splits from the Allende meteorite reference sample were analyzed for 22 trace elements by spark source mass spectrometry. The analytical technique for the spark source mass spectrometer has been described by Taylor (1971) and Taylor and Gorton (1977). The method utilizes the rare earth element Lu as an internal standard. The samples were mixed in equal amounts with a graphite mix containing 50 ppm Lu as the oxide. Two electrodes were then prepared inside polyethylene slugs, using a steel die and a hydraulic press. The electrodes were mounted in the source of an AEI MS7 spark source mass spectrometer and subjected to a high-voltage spark. The ion beam was recorded on an Ilford Q-2 photographic plate. A series of graded exposures was obtained, which ensured that a wide range of element concentration was covered. The intensity of nuclide lines for the various elements was determined with a photodensitometer. The ratio of this intensity to the intensity of the internal standard line on the same exposure is directly related to element concentration. Calibration of element to internal standard intensity ratio to elemental abundance was obtained by the use of well-analyzed rock standards, such as W-1, G-1, BCR-1, and AGV-1. Accuracy and precision of the technique are dependent upon the total number of exposures used to calculate the abundance of each element. For this project three photoplates were exposed for each sample, each with about 14 exposures. This resulted in the abundance of each element being based on 10 to 15 determinations. Lutetium is an ideal internal standard for the lanthanide elements, so excellent precision and accuracy is obtained for these elements. Accuracy decreases for elements with mass number below 100 because of increasing background. There is also a greater possibility of interference from metal carbides and oxides in this mass region. The precision obtained for most elements is about ±5% and accuracy is about ±5%, but this varies somewhat according to the element in question. Results are listed in Table 23.

TABLE 23.—Spark source mass spectrometer analysis (in ppm) of two samples of the Allende meteorite reference sample (dash indicates no data).

Constituent	Split 1/ Position 27	Split 13/ Position 23
Y	2.3	3.0
Zr	6.4	6.4
Nb	0.48	0.53
Cs	0.10	–
Ba	5.1	5.7
La	0.53	0.52
Ce	1.43	1.45
Pr	0.19	0.20
Nd	1.00	0.99
Sm	0.30	0.32
Eu	0.10	0.11
Gd	0.36	0.39
Tb	0.07	0.07
Dy	0.44	0.47
Ho	0.09	0.10
Er	0.28	0.28
Tm	0.053	0.064
Yb	0.29	0.31
Lu	0.05	0.06
Hf	0.20	0.22
Pb	1.57	1.49
Th	0.063	0.063

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17. Determinations of Na, K, Rb, Cs, Br, Te, and U by Neutron Activation Analysis in the Allende Meteorite Reference Sample

O. Müller

Seven minor and trace elements have been determined in the Allende meteorite reference sample. Replicate analyses were performed on Split 5/Position 1 and Split 8/Position 27 of the reference powder using neutron activation analysis and radiochemical separations.

The irradiations of the Allende samples were mainly performed in the core of the TRIGA Mark I reactor of the Deutsches Krebsforschungszentrum, Institut für Nuklearmedizin, Heidelberg, at a neutron flux of $\sim 10^{13}$ neutrons $cm^{-2}\ sec^{-1}$. Samples were also irradiated in the high flux reactor FR2 of the Gesellschaft für Kernforschung, Karlsruhe, at a flux of $\sim 8 \times 10^{13}$ neutrons $cm^{-2}\ sec^{-1}$. Contributions to (n,γ)-produced nuclides by interfering (n,p), (n,α), and U(n,f) reactions have been determined or estimated. For most nuclides analyzed in this work the correction was small or negligible. We observed, however, a distinct interference of $^{24}Mg(n,p)^{24}Na$, the amount of which was considered in the calculation of the sodium concentration. Mg is a major element in Allende, about 15%.

Four different schemes for chemical analysis were used for the following groups of elements: (1) Na, K, and Cs; (2) Na, K, Rb, and Cs; (3) Br and Te; and (4) U.

For the determination of Na, K, and Cs, 100 mg of Allende powder and Na, K, and Cs monitors were irradiated for 4 hours in the core of the TRIGA reactor. The samples were dissolved with carriers in HF and $HClO_4$. The dry residue was dissolved in 1N HCl. Na, K, and Cs were separated by passing the solution through a 25 × 0.8 cm Dowex 50, X-8, 50–100 mesh cation exchange column and eluting with 1N HCl. For the Na fraction no additional chemical processing was necessary. Potassium was precipitated with sodium tetraphenylboron, the precipitate filtered and dissolved in 6N HCl, the solution scavenged twice with Fe and Mn carriers, and K was again precipitated as above and converted to $KClO_4$. The purification of the Cs fraction was similar to that for K; the final step was precipitation as $Cs_3Bi_2I_9$, by which complete separation from K was achieved. Monitor solutions of Na were weighed on Mylar foil and those of K and Cs on highly pure aluminum foil before irradiation. The monitors were chemically processed as described above. The γ-activities of ^{24}Na, ^{42}K, and ^{134m}Cs were counted using 3 × 3 inch NaI(Tl) scintillation counters and a 400-channel pulse height analyzer. Na and K were counted in solution (well counter), and Cs was mounted as $Cs_3Bi_2I_9$ and counted within the solid crystal. The chemical yields of Na and K were measured by atomic absorption and the Cs yield was determined gravimetrically as $Cs_3Bi_2I_9$.

For the determination of Na, K, Rb, and Cs, 90 mg of Allende powder was irradiated in the high flux reactor FR2 of the Gesellschaft für Kernforschung, Karlsruhe, for 24 hours at a flux of $\sim 8 \times 10^{13}$ neutrons $cm^{-2}\ sec^{-1}$.

Na, K, Rb, and Cs monitors and USGS standard diabase W-1 as a reference sample, were irradiated simultaneously. The high neutron flux is necessary to produce sufficient activity of ^{86}Rb ($t_{1/2}$ = 18.7 day) and ^{134}Cs ($t_{1/2}$ = 2.05 yr). Cesium, measured as ^{134m}Cs in the first procedure, was determined now as ^{134}Cs. The meteorite and diabase samples were dissolved with carriers in HF and $HClO_4$. The dry residue of perchlorates was ground with pure ethanol. The perchlorates of K, Rb, and Cs remained undissolved and were collected on filter paper. The alcoholic filtrate containing the Na was evaporated, the residue dissolved in 1N HCl, and Na separated by passing the solution through a Dowex 50 column as before. The perchlorates of K, Rb, and Cs were dissolved in H_2O and the solution scavenged twice with Fe and Mn carriers (hydroxide precipitations). K, Rb, and Cs were precipitated with sodium tetraphenylboron and converted to perchlorates, which were rinsed with 5 ml of H_2O into a plastic counting tube. Because of the different γ-energies of ^{42}K, ^{86}Rb, and ^{134}Cs, these nuclides can be measured together; the resolution of the NaI(Tl) detector is sufficient. ^{42}K was counted first and, after its decay, ^{86}Rb and ^{134}Cs. A Na monitor solution was weighed on Mylar foil and equilibrated with the Na carrier after irradiation. This solution was used for counting after

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TABLE 24.—Results of neutron activation analysis of the Allende meteorite reference sample (* activation in the high flux reactor FR2, Karlsruhe; all other values from activation in the TRIGA reactor, Heidelberg).

Constituent	Split 5/ Position 1	Mean with error	Split 8/ Position 27	Mean with error	Average of splits
		PARTS PER MILLION			
Na	3420	3330±90	3410	3350±100	3340
	3260		3230		
	3310*		3420*		
K	301	295±7	291	289±2	292
	288		287		
	297*		288*		
Rb	1.2*	1.2	1.2*	1.2	1.2
Br	1.52	1.54±0.02	1.54	1.52±0.02	1.53
	1.56		1.50		
Te	1.0	1.1±0.1	1.0	1.1±0.1	1.1
	1.2		1.2		
		PARTS PER BILLION			
Cs	91	92±2	96	96±1	94
	94		96		
	92*		95*		
U	16	15.5	16	16	16
	15				

appropriate dilution with water. A mixed K, Rb, and Cs monitor solution was weighed on highly pure aluminum foil and dissolved with carriers in HCl after irradiation. The heavier alkali elements were chemically processed and counted as described above without separation. The chemical yields of Rb and Cs were determined in an aliquot of the alkali solution, by reactivation and the yields of Na and K by atomic absorption spectrometry.

For the Br and Te analysis, 100 mg of Allende powder and Br and Te monitors were irradiated for 14 hours in the core of TRIGA. Chemical processing was started 15 hours after the end of irradiation. A waiting period of at least 4 hours is necessary to allow for the decay of ^{131}Te to ^{131}I ($t_{1/2}$ = 25 min). The monitors, which were pipetted on highly pure aluminum foil, were dissolved in the presence of NaBr and NaI carriers with 2N NaOH in a nickel crucible, dried, and fused with Na_2O_2. The fusion cake was treated as described below for the meteorite samples. Each meteorite powder was fused with Na_2O_2 in the presence of NaBr and NaI carriers in a nickel crucible. The fusion cake was dissolved in H_2O and the solution boiled to destroy excess H_2O_2. Na_2SO_3 was added and the solution weakly acidified with 4N H_2SO_4 to reduce BrO_3^- and IO_3^- to Br^- and I^-. The whitish residue was filtered off and discarded. The filtrate was transferred to a separatory funnel and I^- was oxidized to I_2 by dropwise addition of $NaNO_2$ solution in the presence of CCl_4. Br^- remained in the aqueous phase. I_2 was extracted into CCl_4, the violet solution transferred to another funnel, and I_2 back-extracted into H_2O by adding Na_2SO_3 solution. The I^--I_2 extraction cycle was repeated twice. The final aqueous I^- solution was heated to expel excess SO_2 and I^- precipitated as AgI, which was weighed for chemical yield. ^{131}I was counted on a 3 × 3 inch solid NaI(Tl) crystal and the 0.365 meV γ-peak was used for evaluation. The Br^- solution was acidified to at least 3N by adding 8N H_2SO_4. Br^- was oxidized to Br_2 by dropwise addition of $KMnO_4$ solution in the presence of CCl_4. Br_2 was extracted into CCl_4 by intense shaking, the yellow-brown solution transferred to another funnel, and Br_2 back-extracted into 3N H_2SO_4 by adding Na_2SO_3 solution. The Br^--Br_2 extraction cycle was repeated twice. The final Br^- solution was heated to expel excess SO_2 and Br^- was precipitated as AgBr, which was weighed for chemical yield. ^{82}Br was counted on a 3 × 3 inch solid NaI(Tl) crystal and the 0.55 meV γ-peak was used for evaluation.

A sample of the Allende powder weighing 100 mg, and a U monitor were irradiated for 2 hours in the core of TRIGA for the determination of uranium. The chemical processing of samples and monitor was started 20 minutes after irradiation. Because of the short-lived ^{239}U, a rapid chemical procedure for the separation of U must be used. Each Allende powder was fused in Na_2O_2 with 15 mg U carrier in a nickel crucible. The fusion cake was dissolved in 6N HCl and the solution filtered through a plug of glass wool to remove undissolved silica and part of the NaCl. A new ml of concentrated HCl was added to the filtrate to make it at least 6N. The filtrate was passed through an 8 × 1.2 cm Dowex 1, X-8, 100–200 mesh anion exchange column to absorb the $(UO_2)^{2+}$-chloro complex. The column was washed several times with a few ml of 6N HCl. Then $(UO_2)^{2+}$ was eluted with 40 ml 0.4N HNO_3 and the eluate collected in a beaker containing 40 g $Ca(NO_3)_2 \cdot 4H_2O$ and 4 ml concentrated HNO_3. U was extracted into diethyl ether and the ether phase washed three times with 15 ml of a solution containing 40 ml 0.4N HNO_3, 4 ml concentrated HNO_3, and 40 g $Ca(NO_3)_2 \cdot 4H_2O$. U was back-extracted into H_2O and the ether still present evaporated. To the acidic U solution 1 ml 0.1N EDTA solution was added to complex Ca^{2+}. Then NH_4OH was added until a pale yellow color appeared. U was precipitated by adding 4 ml 2% 8-hydroxyquinoline in 3% acetic acid solution and by buffering the solution to pH 7 with a 10% ammonium acetate solution. The U precipitate was filtered, washed with H_2O, and dissolved with 2N HCl, and the U solution was transferred to a plastic counting tube. The γ-ray spectrum of ^{239}U was counted using a 3 × 3 inch NaI(Tl) scintillation well counter and a 400-channel pulse height analyzer. The 74 keV line of ^{239}U was used for evaluation of the intensity. This line has a high peak height to background ratio and is therefore especially valuable for the detection of uranium amounts in the nanogram range. In addition, the half-life of ^{239}U ($t_{1/2}$ = 23.5 min) was controlled by counting the U solutions of samples and monitors at least twice. The chemical yield of U was determined by transferring the U solution to a beaker quantitatively and by re-precipitating U with 8-hydroxyquinoline. To achieve complete precipitation 2 ml of the reagent was added to the 2N HCl-U solution and the acid neutralized as above. The U precipitate was collected in a weighed glass filter crucible, washed with H_2O, dried at 105° C, and weighed. The yield was ~50%. The U monitor, weighed as solution on aluminum foil, was dissolved with 15 mg U carrier in 10 ml 6N HCl and 1 ml concentrated HNO_3. The solution was passed through a Dowex-1 ion exchange column and the chemical separation, counting, and yield determination performed as described above. The chemical yield was ~60%.

The results of the replicate analyses are compiled in Table 24. All elements except Rb and U (only one U run for Split 8/Position 27), were determined at least twice in both splits. The agreement between analyses of each split is satisfactory as shown in Table 24. The errors are about ±3% for most elements, but are somewhat higher for Te and U. In comparing the mean values of Split 5/Position 1 and Split 8/Position 27, it appears that these splits are derived from a well-homogenized powder, because no obvious differences with respect to the analyzed elements exist.

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18. Analysis of Rare Earth Elements in the Allende Meteorite Reference Sample by Stable Isotope Dilution

Noboru Nakamura and Akimasa Masuda

Rare earth elements were determined by the stable isotope dilution technique in two splits of the Allende meteorite reference sample. Results are given in Table 25.

TABLE 25.—Stable isotope dilution analysis (in ppm) of rare earth elements in the Allende meteorite reference sample (a and b are duplicate analyses of each split; dash indicates data not available).

Constituent	Split 2/Position 20 (a)	(b)	Split 5/Position 40 (a)	(b)
La	0.5052	0.5190	0.5077	0.5220
Ce	1.330	1.325	1.320	1.350
Nd	1.004	1.014	1.010	1.008
Sm	0.3288	0.3280	0.3303	0.3234
Eu	0.1125	0.1141	0.1130	0.1119
Gd	0.4130	0.4138	0.4138	0.3961
Dy	0.5056	–	0.5020	–
Er	0.3031	–	0.3031	–
Yb	0.3161	–	0.3133	–
Lu	0.0468	–	0.0462	–

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19. Bulk Chemical Analysis of the Allende Meteorite Reference Sample

J.H. Scoon

Two samples of the Allende meteorite reference sample, identified as Split 20/Position 1 and Split 22/Position 10, were analyzed for major and minor elements using wet chemical methods (Table 26).

Silica was determined by dehydration with hydrochloric acid and volatilization with hydrofluoric acid. The silica remaining in solution after this operation was recovered by dehydration of the solution obtained from the pyrosulfate fusion of the R_2O_3 precipitate. Alumina was obtained by difference from the R_2O_3 precipitate. Total iron was determined by titration with ceric sulfate solution after treatment of an aliquot of the R_2O_3 solution in a silver reductor. Ferrous iron was determined by the modified Pratt method. Manganese was determined colorimetrically as permanganate after oxidation with potassium periodate. Titanium was estimated similarly using hydrogen peroxide. Calcium was precipitated as oxalate and ignited and weighed as oxide. Magnesium was precipitated as magnesium ammonium phosphate and ignited and weighed as $Mg_2P_2O_7$. Sodium and potassium were determined using an E.E.L. flame photometer. Total water was obtained by the Penfield method. H_2O^- was obtained from loss in weight at 105° C. Phosphorus was determined colorimetrically by the vanadomolybdate method. Chromium was estimated colorimetrically using diphenyl carbazide. Nickel was precipitated as the red dimethylglyoxime complex, dried, and weighed. Sulfur was precipitated as $BaSO_4$ after sodium carbonate fusion and extraction; allowance was made for reagent blank. Carbon was determined by wet oxidation with chromic acid and phosphoric acid. The carbon dioxide formed was weighed in absorption tubes. Any carbonate present was first decomposed by heating with phosphoric acid alone.

TABLE 26.—Bulk chemical analysis of two samples of the Allende meteorite reference sample (in %).

Constituent	Split 20/ Position 1	Spit 22/ Position 10
SiO_2	34.28	34.27
Al_2O_3	3.71	3.67
Cr_2O_3	0.53	0.54
Fe_2O_3	nil	nil
FeO	30.31	30.23
MnO	0.18	0.18
MgO	24.50	24.51
CaO	2.60	2.53
Na_2O	0.45	0.45
K_2O	0.03	0.03
H_2O^+	0.11	0.10
H_2O^-	0.06	0.06
TiO_2	0.19	0.18
P_2O_5	0.26	0.26
NiO	1.81	1.81
S	2.04	2.10
C	0.22	0.22
Total	101.28	101.14
Less S = O	0.88	0.90
Total	100.40	100.24
Total Iron as Fe	23.36	23.32

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20. Abundances of the 14 Rare Earth Elements and 12 Other Major, Minor, and Trace Elements in the Allende Meteorite Reference Sample by Neutron Activation Analysis

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Two splits of the Allende meteorite reference sample, Split 10/Position 3 and Split 12/Position 32, were analyzed for the 14 rare earth elements (REE) and 13 major, minor, and trace elements. Two 1 g aliquants of each sample split were subjected to sequential instrumental neutron activation analysis (INAA), followed by radiochemical neutron activation analysis (RNAA), as outlined by Schmitt et al. (1970) and Rey et al. (1970).

The sequence began with a determination of the Al abundance via INAA by a 1 minute activation in a low thermal neutron flux of $\sim 5 \times 10^{6}$ neutrons cm^{-2} sec^{-1}, followed by counting with NaI(Tl) γ-ray spectrometry. Ca, V, Na, and Mn were also determined by INAA with a 5 minute activation ($\sim 8 \times 10^{11}$ neutrons cm^{-2} sec^{-1}); spectra were taken with a Ge(Li) detector coupled to a 2048-channel analyzer. Samples and standards were then subjected to a 3 hour activation in a high thermal neutron flux ($\sim 1.6 \times 10^{12}$ neutrons cm^{-2} sec^{-1}) and the abundances of Cr, Sc, Fe, and Co were obtained by counting with Ge(Li) γ-ray spectrometry. After about one month's radioactive decay, the samples and standards were activated for 3 hours at a thermal neutron flux of $\sim 1.6 \times 10^{12}$ neutrons cm^{-2} sec^{-1}, allowed to decay for 2 to 3 days, and again reactivated for 1 hour at the same thermal neutron flux. This procedure was employed in order to maximize the long-lived REE activities and to minimize the level of 15-hour ^{24}Na during radiochemical processing of the activated samples. The samples were allowed to decay for ~30 minutes after activation before chemical separations were started. The abundances of K, In, Cd, Y, and the 14 REE were determined by RNAA.

Analytical results are listed in Table 27. The associated errors represent the statistical counting uncertainties plus the overall estimated error, which includes the estimated errors in counting geometries and the conventional chemical standardization of the activation elemental standards. Column six lists the average abundance values for the four samples analyzed in this work and column seven gives the corresponding Allende data of other workers.

For the major, minor, and trace elements, Al to Cd in Table 27, the average precision was ±4.0%, with a range from 0.7 to 16%. The precision was above 7% for only one element (Ca, 16%) and is the upper limit for the other ten elements of this group. This large Ca uncertainty is entirely due to the low counting statistics obtained for 8.8-minute ^{49}Ca, which was counted together with 5.8-minute ^{51}Ti and 3.77-minute ^{52}V. The precision for Ca could be improved to below ±10% if V were excluded from the joint INAA for Ca and V. Despite the large Ca error, the average value agrees with other workers (King et al., 1969, and Clarke et al., 1970) who used other techniques.

As noted in the last two columns of Table 27, the agreement between the averages of this work and others for Al, Ca, Mn, K, Sc, and In indicates that these elements are homogeneously distributed, at least for the three or more fragments that were sampled and analyzed by various groups.

Apparently the elements Fe, V, Cr, Co, and Cd are heterogeneously distributed among the various fragments. Variations for V, Cr, and Co that were obtained from various Allende fragments are probably real. The same INAA technique in our laboratory yielded 26.3+0.5% Fe (this work with 4 g) and 21.9±0.4% (Wakita and Schmitt, 1970, with a 2 g interior piece) for two different Allende fragments. Emery et al. (1969), also using INAA, obtained 27.8% Fe, which agrees within 2ρ of this work and which abundance value is above the 23.8%, 23.6%, and 24.4%

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obtained by Clarke et al. (1970), King et al. (1969), and Morgan et al. (1969), respectively. At present we are not able to explain why the Fe values obtained via INAA are about 10% higher than those obtained by conventional techniques. Furthermore, we believe that the Fe value obtained by the standard methods are more accurate than the INAA results.

The very uniform In abundances observed in this work and that by Wakita and Schmitt (1970) and the similar chalcophilic character of In and Cd tend to cast some doubt on the validity of either the lower value of Cd, 0.19±0.01 ppm (Wakita and Schmitt, 1970), or the average higher value of 0.58±0.04 ppm of this work. Perhaps the large variations in Cd abundances are real and represent sampling problems. Assuming trace elemental uniformity within each carbonaceous meteoritic type, the close agreement of the Cd of 0.58 ppm of this work with 0.52 ppm Cd found in Mokoia, another Type III carbonaceous chondrite (Schmitt et al., 1963), the reliability of the lower Cd value, 0.19 ppm, may be questioned. To settle this point, Cd should be determined in many additional Allende fragments.

REE and Y abundances of this work agree in general with those determined in another fragment by Wakita and Schmitt (1970), who have discussed the relationships of the REE abundances in Allende with other carbonaceous classes. The average precision, expressed as a sample standard deviation, is ±3.5% for the REE + Y, with a precision range of ±1 to 10%. The element Nd yielded the poorest precision of ±10%, this resulted mainly from counting statistics. The light REE, La to Bd, are enriched by an

TABLE 27.—Composition of the Allende meteorite reference sample (two 1 g aliquants of each split were taken for analysis).

	Split 10/Position 3		Split 12/Position 32			
Element	10-1	10-2	12-1	12-2	Average	Other work
			PERCENT			
Al	1.74±0.05	1.72±0.05	1.70±0.05	1.72±0.05	1.72±0.02	1.71±0.05[c], 1.73[e], 1.75[f]
Ca	1.4±0.3	2.2±0.4	1.6±0.3	1.8±0.3	1.8±0.3	2.0±0.2[c], 1.87[e], 1.8[f]
Fe	27.0±1.3	26.4±1.3	26.1±1.3	25.7±1.3	26.3±0.5	21.9±0.4[c], 24.4±0.4[d], 23.8[e], 23.6[f], 27.8[g]
			PARTS PER MILLION			
Mn	1410±35	1410±35	1390±35	1400±35	1400±10	1450±40[c], 1400[e], 1300[f], 1700[g]
Na	3260±160	3210±160	3280±160	3410±170	3290±90	3370±100[c], 3300±100[d], 3300[e], 3000[f]
K	210±20	230±20	220±20	220±20	220±10	200[e], 250[f], 1800±1200[g]
V	115±20	110±20	112±20	110±20	112±3	130±22[c], 70[e], 170[f]
Cr	4330±130	4240±130	4190±130	4140±130	4230±80	3680±100[c], 3900±100[d], 3600[e], 4200[f], 4200[g]
Co	753±38	744±37	734±36	721±36	738±14	640±20[c], 650±40[d], 600[e], 700[f], 600[g]
Sc	12.2±0.6	12.0±0.6	11.8±0.6	11.9±0.6	12.0±0.2	11.0±0.5[c], 12.2±0.2[d], 11[e], 10[g]
In	0.028±0.001	[a]	0.027±0.001	0.027±0.001	0.027±0.001	0.027±0.001[c]
Cd	0.62±0.06	0.54±0.05	0.57±0.06	[b]	0.58±0.04	0.19±0.01[c]
Y	3.0±0.1	2.8±0.1	2.9±0.1	3.1±0.1	3.0±0.1	3.0±0.1[c], 2[e]
La	0.49±0.02	0.50±0.02	0.49±0.02	0.53±0.02	0.50±0.02	0.44±0.02[c], 0.7[e]
Ce	01.36±0.06	1.34±0.06	1.42±0.06	1.44±0.06	1.39±0.05	1.25±0.06[c], 1[e]
Pr	0.21±0.01	0.21±0.01	0.21±0.01	0.21±0.01	0.21±0.00	0.20±0.01[c], 0.2[e]
Nd	0.98±0.05	1.04±0.05	1.18±0.06	0.98±0.05	1.05±0.10	0.91±0.05[c], 0.9[e]
Sm	0.341±0.013	0.327±0.012	0.362±0.014	0.359±0.014	0.347±0.016	0.29±0.01[c], 0.5[e]
Eu	0.117±0.005	0.115±0.005	0.116±0.005	0.116±0.05	0.116±0.001	0.107±0.005[c], 0.1[e]
Gd	0.45±0.02	0.44±0.02	0.43±0.02	0.44±0.02	0.44±0.01	0.43±0.02[c], 0.6[e]
Tb	0.077±0.005	0.079±0.005	0.084±0.005	0.083±0.005	0.081±0.003	0.074±0.005[c], 0.09[e]
Dy	0.45±0.02	0.41±0.02	0.45±0.02	0.45±0.02	0.44±0.02	0.42±0.02[c], 0.6[e]
Ho	0.113±0.004	0.115±0.004	0.112±0.004	0.113±0.004	0.113±0.001	0.12±0.1[c], 0.1[e]
Er	0.30±0.01	0.29±0.01	0.30±0.01	0.29±0.01	0.30±0.01	0.31±0.02[c], 0.3[e]
Tm	0.053±0.002	0.058±0.002	0.056±0.002	0.053±0.002	0.055±0.003	0.049±0.001[c]
Yb	0.30±0.01	0.30±0.01	0.30±0.01	0.30±0.01	0.30±0.00	0.32±0.02[c], 0.4[e]
Lu	0.048±0.002	0.048±0.002	0.050±0.002	0.049±0.002	0.049±0.001	0.058±0.002[c]

[a] The In value determined for 10-2 is 0.02±0.01; the large error limit is imposed due to an analyzer malfunction during counting.

[b] The Cd value for 12-2 could not be determined because of a laboratory accident during the chemical processing.

[c] Wakita and Schmitt, 1970; specimen NMNH 3496.

[d] Morgan et al., 1969; specimens NMNH 3610 and ASU S-5211.

[e] Clarke et al., 1970; specimens NMNH 3509 and 3511.

[f] King et al., 1969.

[g] Emery et al., 1969.

average of ~12% in the Allende meteorite reference sample over the corresponding REE abundances in fragment NMNH 3496 (Wakita and Schmitt, 1970), while the average ratio of the heavy REE (Tb to Lu plus Y) abundances in the Allende meteorite reference sample to fragment NMNH 3496 is 1.00; i.e., essentially identical heavy REE abundances were obtained in two different Allende fragments. The relative enrichment of light REE may be ascribed to a larger content of accessory minerals such as plagioclase in the Allende meteorite reference sample relative to fragment NMNH 3496.

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This contribution is dedicated to the memory of our deceased friend, Richard H. Smith. In the field of meteoritics he was one of the collaborators who first measured accurately the fundamental rare earth elemental abundances in chondritic, achondritic, and other types of meteorites.

21. Analyses of Trace Elements in the Allende Meteorite Reference Sample by Emission Spectrometry

G. Thompson

Two splits from the Allende meteorite reference sample were analyzed for trace element composition. Results were obtained by direct-reading optical emission spectrometry with d.c. arc excitation. Details of the technique and precision and accuracy (±5–10%) are given by Thompson and Bankston (1969). Results are listed in Table 28.

TABLE 28.—Trace element analysis of the Allende meteorite reference sample (in ppm) (standard matrix used for analyses of Split 10/Position 13 and Split 11/Position 11(a) had the composition: SiO_2 39.4%, Al_2O_3 11.3%, Fe_2O_3 30.4%, MgO 6.7%, $CaCO_3$ 10.0%, NaCl 1.1%, KCl 1.1%; standard matrix used for analysis of Split 11/Position 11(b) had the composition: SiO_2 48.2%, Al_2O_3 13.9%, Fe_2O_3 9.3%, MgO 7.4%, $CaCO_3$ 16.5%, Na_2CO_3 2.8%, KCl 1.0%, TiO_2 0.9%; dash indicates not determined).

Constituent	Split 10/ Position 13	Split 11/ Position 11 (a)	(b)
Ag	<1	<1	<1
B	<5	<5	–
Ba	<2	<2	<2
Bi	<2	<2	<2
Cd	<2	<2	<2
Co	740	700	>500
Cr	3000	3000	>2000
Cu	120	120	–
Ga	25	25	20
Li	4	4	2
Mo	<2	<2	<2
Ni	>1000	>1000	>2000
Pb	12	10	13
Rb	<5	<5	<10
Sn	<20	<20	–
Sr	8	8	8
V	100	85	60
Zn	110	110	–
Zr	48	54	47

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22. Analyses of Oxygen and Silicon in the Allende Meteorite Reference Sample by Neutron Activation

A. Volborth

Two splits from the Allende meteorite reference sample have been analyzed for oxygen and silicon by neutron activation analysis. Each split was analyzed for oxygen by comparison to four carefully prepared standards. The actual number of counts selected per determination varied from 1,000,000 to 1,400,000 depending on the total neutron flux during each experiment. For silicon, results of each split represent the mean and actual deviation of two independent determinations of 300,000 counts each or 600,000 counts total; thus a theoretical standard deviation of 775 counts or 0.13% applies for each of these determinations. Details of the technique are given in Vincent and Volborth (1967), Volborth and Vincent (1967), and Volborth et al. (1975, 1977a,b), and the results are given in Table 29.

TABLE 29.—Neutron activation analysis (%) of oxygen and silicon in two subsamples of the Allende meteorite reference sample.

Constituent and standard no.*	Split 19/ Position 14 (Rabbitt 190)	Split 8/ Position 26 (Rabbitt 191)
OXYGEN		
157	36.30	36.74
183	36.43	36.69
184	36.32	36.65
187	36.41	36.47
mean	36.37±0.06	36.64±0.12
SILICON	16.22±0.00	16.14±0.03

* Composition of standard numbers: 157 = 100% SiO_2, quartz; 183 = 50% Al_2O_3, 50% SiO_2; 184 = 30% Al_2O_3, 70% SiO_2; 187 = 20% Al_2O_3, 60% SiO_2, 20% Fe_2O_3.

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23. Bulk Chemical Analysis of the Allende Meteorite Reference Sample

H.B. Wiik

Split 20/Position 20 from the Allende meteorite reference sample was analyzed for major, minor, and trace elements. Results and methods used for the analysis are given in Table 30.

TABLE 30.—Bulk chemical analysis of the Allende meteorite reference sample.

Constituent	Split 20/ Position 20	Method
	PERCENT	
Fe	0.1	extraction with $HgCl_2$
Ni	1.40	gravimetric from main analysis
Co	0.069	neutron activation
FeS	6.17	calculated from S (as $BaSO_4$)
(S)	(2.25)	
SiO_2	34.10	gravimetric + colorimetric
TiO_2	0.13	colorimetric
Al_2O_3	3.43	neutron activation
FeO	24.62	calculated from total Fe, metallic Fe, and FeS
MnO	0.193	colorimetric
MgO	25.65	gravimetric
CaO	2.58	gravimetric, oxalate to carbonate at 520°
Na_2O	0.47	neutron activation
K_2O	0.046	neutron activation
P_2O_5	0.21	colorimetric
$\pm H_2O$	0.00	gravimetric
Cr_2O_3	0.59	colorimetric
	0.55	neutron activation
C	0.28	gravimetric
Total	100.04	
Total Fe	23.16	
	PARTS PER MILLION	
Sc	10	neutron activation
V	100–108	emission spectroscopy
Cu	120–130	emission spectroscopy

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24. X-Ray Fluorescence Spectrometric Analysis of the Allende Meteorite Reference Sample

J.P. Willis

Two splits of the Allende meteorite reference sample were analyzed for major, minor, and trace elements by x-ray fluorescence spectrometry. The two 5 g samples received for analysis were ground separately for 1 hour in a specially cleaned automatic agate mortar. Of the finely ground powder 4 g was briquetted with a Bakelite/H_3BO_3 backing and used for the determination of Na, S, Nb, Zr, Y, Sr, Rb, and Ba. The remaining 1 g portions were dried at 120° C in preheated Vitreosil silica crucibles then heated to constant weight at 1050° C in a furnace. Three 0.28 g portions of each sample were fused, cast as disks following the method of Norrish and Hutton (1969), and used for determining Fe, Mn, Cr, Ti, Ca, K, P, Si, Al, and Mg. Each disk was counted twice and each briquette three times, and the results averaged.

All determinations were carried out by x-ray fluorescence spectrometry using a Philips 2 kW PW 1220 semi-automatic x-ray spectrometer. The major elements (except Na and S) were determined using the technique and correction factors of Norrish and Hutton (1969). Mass absorption corrections were made in determining S and Ba using the data of Birks (1963) and using Cr $K\alpha$ as the effective primary radiation. No absorption corrections were made for Na. Mass absorption coefficients for the determination of the remaining trace elements were measured by the method of Reynolds (1963). The analytical techniques have been described elsewhere (Willis et al., 1969; Cherry et al., 1970). Instrumental variables are listed in Table 31. Results are listed in Table 32.

TABLE 31.—Instrumental variables in analyses of the Allende meteorite reference sample by x-ray fluorescence (FPC = flow proportional counter; SC = scintillation counter).

Constituent	X-ray tube target	Crystal	Detector	Collimator
Fe, Mn, Cr	W	LiF(220)	FPC	Fine
Ti, Ca, K	Cr	LiF(200)	FPC	Fine and coarse
Si, Al	Cr	EDDT and PET	FPC	Coarse
Mg	Cr	ADP	FPC	Coarse[1]
P, S	Cr	Ge	FPC	Coarse
Ba	Cr	LiF(220)	FPC	Fine
Na	Cr	Gypsum	FPC	Coarse
Sr, Rb	Mo	LiF(220)	SC	Coarse[2]
Nb, Zr, Y	W	LiF(220)	SC	Coarse[2]

[1] An asymmetric PHA window was used to limit the contribution of P fluorescence from the ADP crystal.

[2] An additional 4-inch secondary collimator was used in front of the scintillation counter.

TABLE 32.—Analyses of two subsamples of the Allende meteorite reference sample and USGS reference sample BCR-1 by x-ray fluorescence spectrometry (total Fe reported as FeO; N/A = not applicable; ND = not determined; dash indicates not reported).

Constituent	Split 1/ Position 3	Split 4/ Position 22	SD (2σ)	BCR-1
	PERCENT			
SiO_2	33.65	33.62	0.34	ND
TiO_2	0.140	0.141	0.006	ND
Al_2O_3	3.20	3.30	0.07	ND
FeO	30.45	30.47	0.16	ND
MnO	0.198	0.199	0.006	ND
MgO	24.79	24.81	0.46	ND
CaO	2.56	2.57	0.03	ND
Na_2O	0.46	0.45	0.02	ND
K_2O	0.033	0.034	0.002	ND
P_2O_5	0.230	0.244	0.016	ND
H_2O^-	0.096	0.131	N/A	ND
S	2.07	2.07	0.02	ND
Cr_2O_3	0.55	0.54	0.02	ND
	PARTS PER MILLION			
Nb	<2.5	<2.5	N/A	12.6
Zr	8.3	8.0	1.1	193
Y	~3	<3	N/A	37.7
Sr	14.2	14.1	0.3	334
Rb	1.0	1.3	0.4	47.3
Ba	3	4	1	-

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