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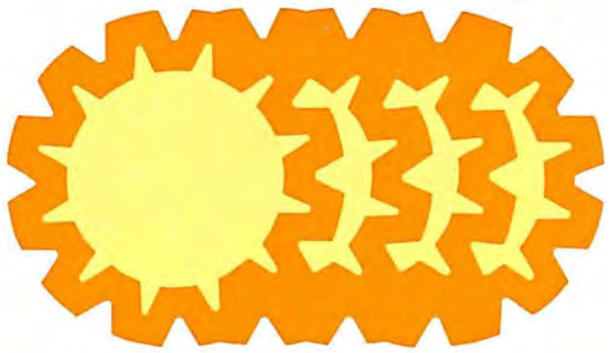
Trace Element Analysis of Normal Lung Tissue and Hilar Lymph Nodes by Spark Source Mass Spectrometry



TRACE ELEMENT ANALYSIS OF NORMAL LUNG TISSUE AND HILAR LYMPH NODES BY SPARK SOURCE MASS SPECTROMETRY

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ABSTRACT

This report describes an analytical study of twenty-one trace metals in human lung and hilar lymph nodal tissue. The samples studies were obtained from random autopsy cases at a large urban hospital.

Twenty of the metals were determined using a double focusing spark source mass spectrometer equipped both for photographic and electrical detection. The remaining metal, mercury, was determined by a flameless atomic absorption technique.

Four methods of standardization were employed in the mass spectrographic analysis: (1) National Bureau of Standards bovine liver (SRM-1577) was used as a reference standard, (2) isotope dilution methods, (3) Standard additions to bovine liver for elements not certified, and (4) synthetic standards for elements not present in the bovine liver.

The high sensitivity of the SSMS coupled with the high resolving power of the instrument make this a technique well suited to the trace analysis of biological materials. Concentrations in the ppb range (dry weight) have been detected.

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I. INTRODUCTION

This is the final report in the series pertaining to the NIOSH contract number HSM 99-73-46, Trace Element Analysis of Normal Lung Tissue and Hilar Lumph Nodes by Spark Source Mass Spectrometry. 512 samples of dried lung tissue, ashed lung tissue, dried lymph nodes and ashed lymph nodes have been received by Accu-Labs Research to be examined in this study.

The high sensitivity and unique comprehensive trace element coverage of spark source mass spectrometry has been applied to determine 20 of the 21 listed elements examined in this study. The remaining trace element, mercury was determined using a method of combustion in a stream of oxygen, collection of the mercury vapors on a gold wire amalgamator, subsequent thermal release and determination by a modified Atomic Absorption Spectrometer. This method will be adequately described in a later section.

II. SCOPE OF NIOSH CONTRACT HSM 99-73-46

1. Accu-Labs was provided with 512 samples of dried lung, ashed lung, dried lymph node, and ashed lymph node by NIOSH. The spark source mass spectrometer capabilities had previously been examine in Contract Number HSM 099-71-33 on a wider variety of materials such as lung tissue, bulk dust, coal dust, and various type of particulate matter on membrane filters [1].

2. In this instance it had been decided to limit the investigation to lung tissue and lymph node tissue samples provided, dried and ashed. In addition, the scope of analysis was restricted to 21 elements which were considered by NIOSH to be the most significant.

3. Whilst provision was made to utilize electrical detection methods where applicable, photographic plate detection was to be employed as the primary method due to the requirement for high resolving power to separate elemental spectra from molecular spectra which in biological samples always constitute a potential problem [2].

4. Investigations were to be conducted at Accu-Labs Research into the sample handling techniques and also the combustion/collection method of sample preparation. A short report is given on this method in a later section.

5. The elements to be examined in this work are as follows:

   Iodine, mercury, selenium, arsenic, tellurium, cadmium, molybdenum, chromium, copper, manganese, beryllium, aluminum, thallium, germanium, zinc, nickel, cobalt, lead, bismuth, vanadium, and titanium.

   At a later date, nickel was deleted from the list and replaced by magnesium, at the request of NIOSH.

III. INSTRUMENT DESCRIPTION

One instrument used in this work was the MS 702R Spark Source Mass Spectrometer.

The equipment is manufactured by Associated Electrical Industries Ltd., Manchester, England. It is the most widely used spark source mass spectrometer in existance today.

The MS 702R is a double focusing instrument having Mattauch Herzog type ion optics and is equipped both for photographic detection and electrical detection.

The available resolution on this equipment is quoted at 10,000 and it is this feature coupled with its high sensitivity that makes the method so applicable to the analysis of biological tissue [3].

The mass range normally covered by the mass spectrometer is 40:1 and this allows the simultaneous detection of all elements with the exception of hydrogen and helium. This mass range is quite sufficient to determine all elements listed in this study.

The other instrument is a Varian Model AA5 atomic absorption spectrophotometer equipped with a hydrogen continuum background corrector. This instrument was used to analyze mercury by the combustion-gold amalgamation flameless technique.

IV. SAMPLE IDENTIFICATION

Sample identification was listed in the first of the bi-monthly reports (4).

V. SAMPLE PREPARATION FOR SPARK SOURCE MASS SPECTROMETRY AND FOR MERCURY DETERMINATION BY FLAMELESS ATOMIC ABSORPTION SPECTROPHOTOMETRY

The samples of lung and lymph nodes were homogenized, then freeze dried by personnel at NIOSH, Cincinnati. A portion of each of these samples was low temperature ashed using the method of plasma ashing [5]

The equipment used for the ashing process was manufactured by International Plasma, Inc. The power dissipation on each of the samples and the NBS bovine liver standard was 200 watts with an oxygen flow of 200 cc per minute.

Half of the material was left in a freeze dried condition to enable Accu-Labs Research to perform two operations:

(a) Investigate the method of combustion/collection ashing as a sample preparation technique.

(b) Carry out a determination of mercury, which may be lost during any sample preparation procedures.

For mass spectrometry each of the ash samples was thoroughly homogenized then mixed with an appropriate quantity of ultra pure graphite (produced by Ultra Carbon Corporation, Bay City, Michigan), and an indium internal standard, mixing was carried out using a previously cleaned agate mortar and pestle. The mixture of sample ash and graphite was compacted into electrodes in a pre-cleaned polyethylene slug using a method of hydrostatic pressing specially designed for the preparation of powder material for mass spectrography analysis [1]

VI. THE METHOD OF STANDARDIZATION AND ANALYSIS FOR TRACE ELEMENTS IN ASHED TISSUE

Four methods of standardization were employed in the mass spectrographic analysis.

(1) The NBS bovine liver standard SRM 1577 was used as a reference material for elements listed in its certification. This standard was ashed using the same conditions as those for ashing of the samples. In this way it is anticipated that any losses of volatile elements in the low temperature ashing of both samples and standard would be of the same magnitude. A copy of the NBS certificate is attached to this report.

(2) Isotope dilution methods (1) were employed for elements having two or more naturally occurring isotopes which are uninterfered in the mass spectra.

(3) Standard addition methods were employed for elements which were indigenous in the bovine liver standard, but are not certified and could not be determined by isotope dilution.

(4) Preparation of synthetic standards was employed for elements which were not present in the bovine liver standard.

The first step in the interpretation and analysis of spectra is to ascertain the sensitivity of the photographic plate spectra, Sp.

$$Sp = \frac{Es}{Emax} \times \frac{X}{100} \times \frac{Is}{100} \times 10^{-6} \quad \text{(Equation 1)}$$

Where Es = "just detectable" exposure of the internal standard isotope (indium)
Emax = longest exposure on the photoplate
X = Atomic % concentration of the internal standard
Is = Isotopic abundance of the chosen isotope of the internal standard.

The value Sp represents the relative concentration (ppm atomic) of any isotope which is "just detectable" on the longest exposure (Emax), assuming unit sensitivity for all elements.

The following method is employed to estimate trace element concentration.

The exposure Ei, at which a line due to an isotope of the trace element becomes "just detectable" is first estimated. The concentration of the

trace element Ci in ppm atomic is calculated from the equation

$$Ci = Sp \times \frac{Emax}{Ei} \times \frac{100}{Ii} \text{ ppm atomic} \quad \text{(equation 2)}$$

where Sp = plate sensitivity
Emax = longest exposure on the photoplate
Ei = "just detectable" exposure of the trace element isotope
Ii = Isotopic abundance of the chosen isotope of the trace element.

The above procedures were carried out for all elements in both the standard bovine liver ash and the sample ash.

The values obtained for concentration in the bovine liver ash were derived and compared to those in the certificate, a relative sensitivity value was calculated as follows:

$$RS = \frac{\text{Listed Value}}{\text{Observed Value}} \quad \text{(Equation 3)}$$

This factor Rs was then applied to correct the observed concentrations in the sample ash.

$$CTrue = Ci \times RS \quad \text{(Equation 4)}$$

In order to arrive at a concentration in parts per million relative to the dried tissue the corrected concentration CTrue (from equation 4) was multiplied by the ration of $\frac{\text{ash weight}}{\text{dry weight}}$ to give the final reported value.

All values from all methods of standardization are reported relative to the dry weight.

1. Isotopic Dilution

The method of isotopic dilution may be used for elements having two or more naturally occurring isotopes, at least two isotopes must be free from interference.

The sample ash of the material under analysis is accurately weighed. To this is added an appropriate amount of ultra pure graphite and a known amount of an enriched isotope of the element under analysis is added in solution. The whole of the material is

thoroughly homogenized and dried under infra red heating. The sample is then pressed into electrodes and subjected to mass analysis at high resolving power.

The photoplate is then examined and the ratio of the two isotopes under consideration is measured.

After determining the ratio of the natural isotope and the enriched isotope the concentration of the element indigenous to the sample is calculated as follows:

$$\text{ppm weight} = WR \frac{(Asp - Bsp\ R)}{M\ (B\ R - A)}$$

where W = weight of the stable isotope "spike" added in Mg.
M = weight of the sample in grams
R = is the measured "altered" isotope ratio, (isotope a/isotope b)
K = ratio of the natural atomic weight/atomic weight of "spike"
A = natural abundance isotope a
B = natural abundance isotope b
Asp = abundance of isotope a in "spike"
Bsp = abundance of isotope b in "spike"

A complete description of the isotope dilution (spiking) method is given in the paper "Determination of Trace Elements in Zinc by Isotope Dilution Spark Source Mass Spectrometry" by Paul J. Paulsen et al (1).

2. STANDARD ADDITION

The method of standard addition is simply to add several aliquots of different amounts of element in solution to a series of samples of ash. The method allows the subsequent determination of the increase of spectral intensity of the element isotope added so that a plot of observed intensity versus concentration may be established. In the attached plot, for Al,V, and I it can be seen that the concentration of the element indigenous to the sample may be established by extrapolation. The method of standard addition is particularly applicable to spark source mass spectrometry since the analytical function is linear (i. e. concentration is directly proportional to ion intensity over a wide concentration range).

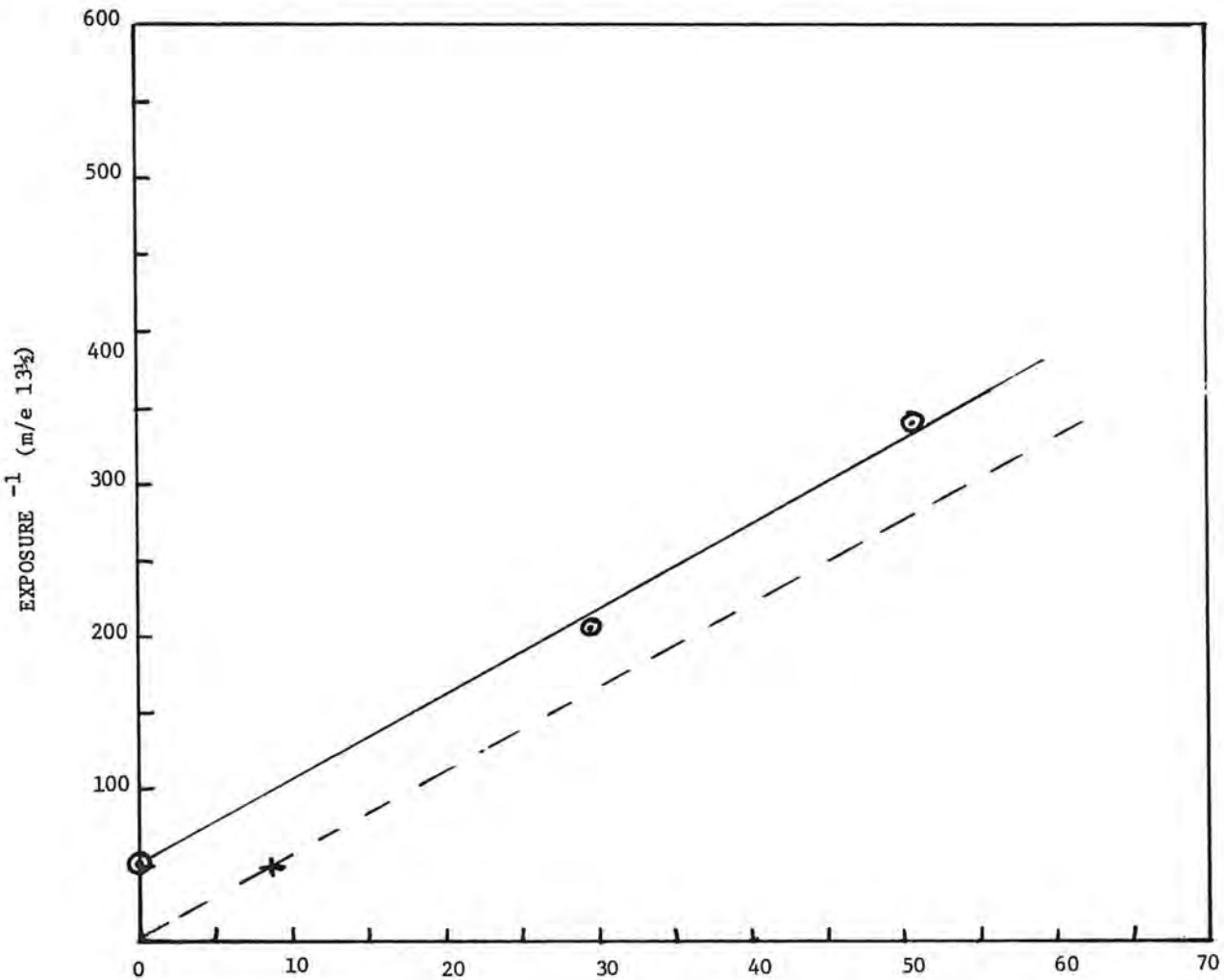
3. Synthetic Standard

Artificial standards were produced for elements which were not found in the bovine liver standard (SRM 1577) and were to be analyzed in the samples.

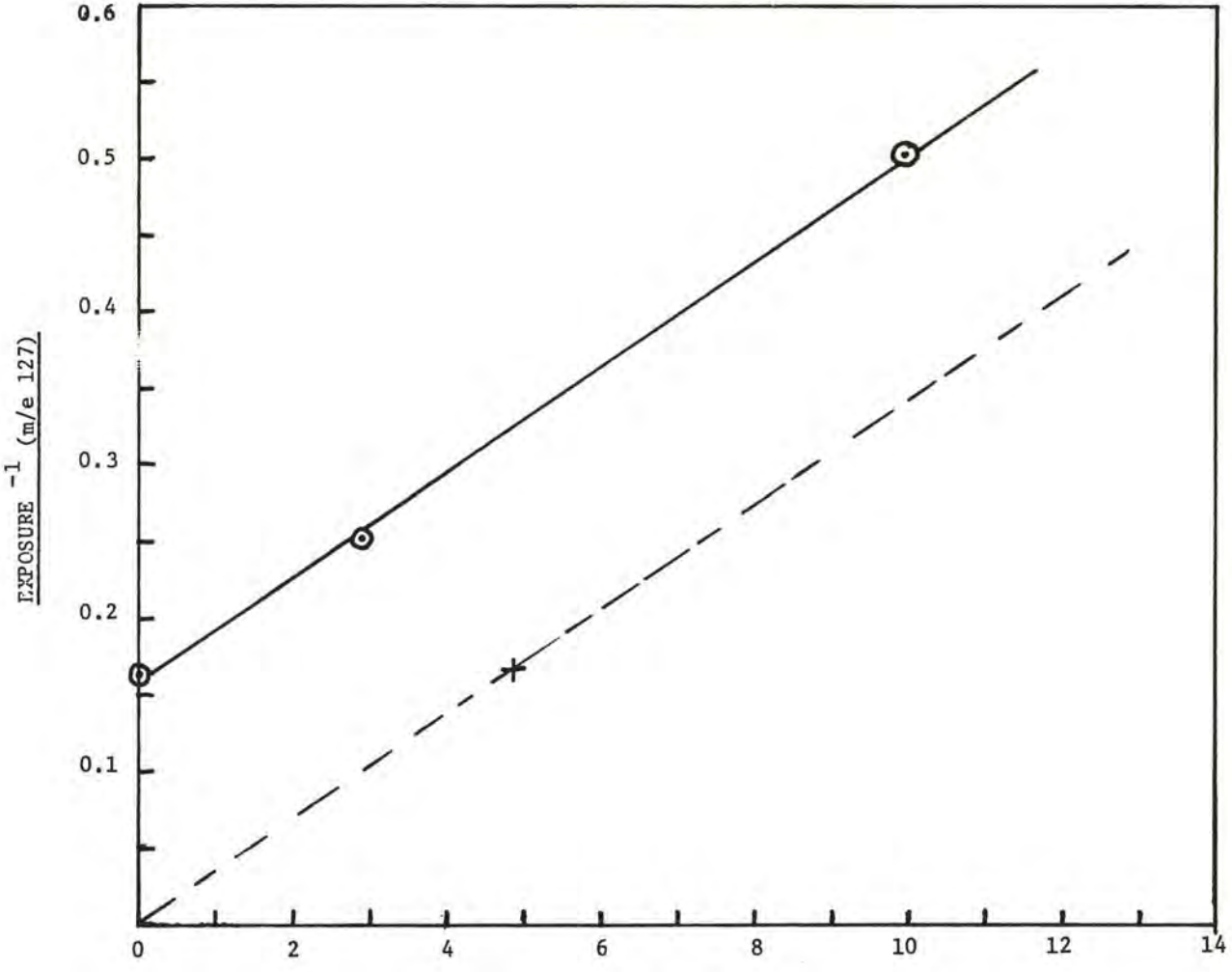
The method simply used was to add a known amount of the element in solution to the bovine liver standard ash, and to an ashed lung sample.

The relative ion intensity of the element versus the indium internal standard was measured in order to determine the relative sensitivity coefficient of the trace element studied.

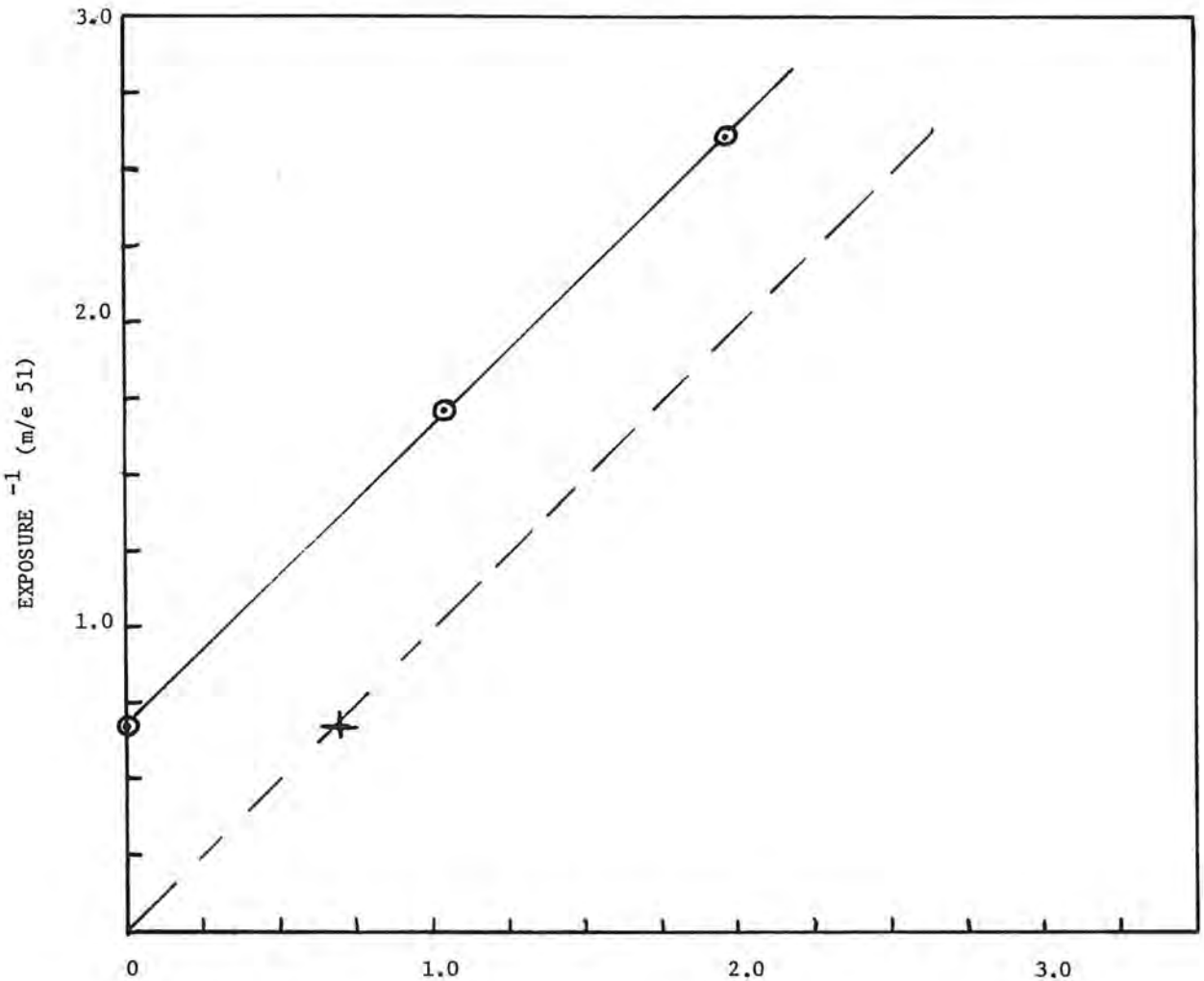
STANDARD ADDITION OF ALUMINUM TO ASHED BOVINE LIVER



STANDARD ADDITION OF IODINE TO ASHED BOVINE LIVER



STANDARD ADDITION OF VANADIUM TO ASHED BOVINE LIVER



VII. TABLES OF RESULTS

The following tables list all the values established on the 20 elements determined by mass spectrography as described previously, and mercury, the analytical procedure for mercury is described in appendix 4.

Sample No.	Concentration in parts per million by weight (dry basis)										
	Bi	Pb	Tl	I	Te	Cd	Mo	Se	As	Ge	Zn
6001	0.03	2.4	0.01	13	0.007	1.0	0.28	2.3	<0.09	0.53	200
6002	<0.02	6.5	<0.007	9.4	0.006	6.5	0.36	4.5	<0.07	0.27	300
6003	<0.02	2.2	<0.005	12	<0.004	0.20	0.69	2.8	<0.05	0.40	270
6004	0.18	12	<0.001	80	<0.008	1.7	0.64	4.9	<0.07	0.65	45
6005	<0.009	1.9	0.002	8.6	<0.002	0.10	0.31	1.2	<0.02	0.21	140
6006	<0.02	41	<0.006	6300	<0.005	0.68	0.53	16	<0.06	1.1	200
6007	<0.02	1.6	<0.005	3300	0.005	1.1	0.31	3.1	<0.05	0.22	32
6008	<0.02	12	0.008	53	0.006	0.17	0.40	7.6	<0.08	0.90	230
6009	<0.02	1.7	<0.004	12	<0.004	0.51	0.34	3.0	<0.05	0.40	58
6010	<0.02	8.2	<0.005	28	0.004	0.22	0.46	3.2	<0.05	0.61	230
6011	<0.01	2.8	<0.004	3.6	0.003	2.9	0.23	2.4	<0.03	0.31	52
6012	<0.003	0.83	<0.002	21	<0.002	1.4	0.26	2.0	<0.02	0.31	44
6013	<0.008	0.16	<0.003	180	0.003	0.15	0.21	5.1	<0.07	0.23	100
6014	0.34	4.1	0.02	41	<0.006	0.29	0.36	8.8	<0.07	0.27	220
6015	0.009	1.4	0.005	8.5	<0.001	0.39	0.31	3.2	0.03	0.11	110
6016	0.01	1.6	0.005	5700	0.005	0.26	0.41	5.6	<0.03	0.33	80
6017	<0.01	1.6	0.01	690	<0.002	0.33	0.23	3.0	<0.03	0.21	55
6018	<0.05	0.45	<0.002	84	0.02	0.16	0.08	0.62	<0.02	0.56	9.4
6019	<0.04	7.9	0.01	34	0.01	1.5	0.52	8.4	<0.09	1.3	370
6020	<0.007	0.67	0.003	7.7	0.002	0.24	0.14	2.1	<0.02	0.50	140
6021	<0.01	2.3	<0.003	2.3	<0.002	1.4	0.58	4.1	<0.03	0.78	180
6022	0.03	4.9	<0.008	49	<0.008	0.80	1.2	6.1	0.11	0.72	150
6023	<0.04	3.3	<0.01	280	<0.006	3.5	0.70	8.9	<0.12	0.49	350
6024	0.009	7.9	0.002	350	<0.001	3.4	0.91	3.9	<0.02	0.59	120
6025	0.01	5.8	0.005	1300	<0.003	0.24	0.84	4.6	<0.04	0.29	61

Sample No.	Concentration in parts per million by weight (dry basis)									
	Cu	Co	Mn	Cr	V	Ti	Al	Mg	Be	Hg
6001	5.4	0.01	0.90	0.15	0.03	8.4	44	110	<0.01	< 0.05
6002	8.1	0.04	1.9	0.56	0.12	12	34	130	<0.01	< 0.05
6003	7.3	0.01	2.4	0.33	0.05	14	23	65	<0.008	< 0.05
6004	12	0.07	1.4	1.4	0.15	31	36	100	<0.001	< 0.05
6005	3.7	0.06	0.09	0.02	0.23	100	23	30	<0.005	< 0.05
6006	14	0.06	5.8	0.23	0.11	4.9	36	100	<0.01	< 0.05
6007	5.6	0.05	4.5	0.94	0.28	50	46	47	<0.008	< 0.05
6008	9.1	0.13	1.4	0.30	0.29	54	12	63	<0.01	< 0.05
6009	15	0.11	0.44	3.3	0.07	3.6	44	75	< 0.008	>> 1
6010	6.2	0.009	1.0	0.10	0.02	4.1	9.5	43	<0.009	< 0.05
6011	0.83	0.06	0.66	0.13	0.03	22	6.4	43	<0.006	< 0.05
6012	4.4	0.02	0.73	0.25	0.08	20	32	19	<0.002	0.10
6013	10	0.08	2.3	0.21	0.13	21	0.64	110	<0.005	< 0.05
6014	18	0.05	3.3	26	0.01	15	9.7	68	<0.01	>> 1
6015	11	0.04	1.2	0.22	0.23	74	4.3	19	<0.004	0.1?
6016	12	0.05	2.6	0.66	0.46	120	46	91	<0.005	0.07
6017	5.1	0.03	1.1	0.87	0.04	8.3	22	42	<0.005	>> 1
6018	1.7	0.02	0.41	0.62	0.12	86	24	24	<0.03	< 0.05
6019	26	0.05	6.4	1.1	0.25	65	97	330	<0.02	< 0.05
6020	6.7	4.2	1.2	0.12	0.21	45	1.1	18	<0.004	0.14
6021	11	0.03	1.2	0.21	0.02	7.3	6.9	30	< 0.006	0.15
6022	30	0.41	2.7	0.91	0.41	120	81	120	<0.01	< 0.05
6023	17	0.18	3.0	4.0	1.1	560	19	88	<0.02	>> 1
6024	7.4	0.03	2.4	0.53	0.46	120	46	62	<0.004	< 0.05
6025	9.4	0.08	1.4	1.5	0.60	150	67	40	<0.006	< 0.05

Sample No.	Concentration in parts per million by weight (dry basis)										
	Bi	Pb	Tl	I	Te	Cd	Mo	Se	As	Ge	Zn
6026	<0.01	0.63	<0.003	1.1	0.003	0.34	0.73	4.0	<0.05	0.73	150
6027	<0.02	8.3	0.006	36	0.007	2.0	0.34	6.4	1.3	0.24	130
6028	<0.02	1.6	0.007	3.9	0.007	0.53	0.32	5.6	<0.07	0.39	140
6029	<0.01	5.0	0.005	2.3	0.003	2.7	0.48	1.7	<0.04	0.08	70
6030	<0.02	0.33		1.5	<0.005	0.21	0.37	5.6	<0.07	2.2	54
6031	<0.02	1.6	<0.005	1.5	<0.004	0.12	0.11	2.3	0.06	0.86	73
6032	<0.02	1.5	<0.005	8.6	0.005	0.53	0.21	2.7	<0.05	0.43	54
6033	0.36	2.0	<0.005	1.1%	0.007	0.61	0.57	4.2	<0.06	0.45	150
6034	<0.02	2.2	<0.005	12	0.009	1.9	0.37	4.3	<0.04	0.53	100
6035	<0.02	17	0.006	7.2	<0.004	2.2	0.34	2.2	<0.06	0.62	110
6036	<0.009	1.6	<0.003	12	<0.002	1.2	0.20	3.9	<0.03	0.44	94
6037	<0.01	0.57	<0.003	5.0	<0.003	0.20	0.12	2.4	<0.04	0.45	79
6038	<0.02	0.93	<0.006	4.1	0.01	1.6	0.88	3.9	<0.07	0.65	70
6039	<0.01	1.1	<0.003	4.8	<0.003	2.0	0.18	1.1	<0.03	0.29	52
6040	<0.01	1.5	0.009	5.1	0.004	0.08	0.24	2.6	<0.04	0.31	47
6041	<0.01	1.1	<0.003	4.6	0.002	0.14	0.11	4.0	0.16	0.34	55
6042	<0.02	2.4	<0.005	3.2	<0.004	2.9	0.50	4.9	<0.08	0.73	160
6043	<0.005	0.37	<0.001	0.16	<0.001	0.30	0.04	1.5	0.02	0.14	23
6044	<0.01	1.3	<0.004	17	<0.004	0.28	0.54	4.4	3.4	0.61	150
6045	0.02	1.0	<0.003	46	<0.003	0.14	0.12	2.1	<0.03	0.40	35
6046	0.10	3.9	<0.005	0.68	<0.004	2.8	0.28	6.4	<0.08	1.1	110
6047	<0.004	0.08	<0.002	9.4	<0.004	0.14	0.23	1.7	<0.02	0.41	45
6048	<0.02	0.46	<0.006	30	<0.005	0.10	0.17	5.4	<0.10	0.48	100
6049	<0.01	0.19	<0.007	2.3	<0.005	0.37	0.06	0.99	<0.03	0.08	41
6050	<0.02	3.1	<0.006	8.1	<0.007	0.82	0.85	3.7	<0.10	1.3	170

Sample No.	Concentration in parts per million by weight (dry basis)									
	Cu	Co	Mn	Cr	V	Ti	Al	Mg	Be	Hg
6026	19	0.13	1.9	0.38	0.04	11	0.59	20	<0.007	< 0.05
6027	10	0.13	7.5	1.4	0.86	290	260	86	0.009	>> 1
6028	19	0.03	2.7	0.34	0.42	140	200	190	<0.009	0.06
6029	11	0.02	0.61	0.11	0.16	9.9	12	63	<0.005	0.07
6030	4.5	0.14	2.7	0.19	0.05	1.4	41	190	0.01	< 0.05
6031	6.6	0.005	1.0	0.11	0.09	15	39	65	<0.009	0.06
6032	6.0	0.02	1.3	0.30	0.09	58	46	78	<0.008	< 0.05
6033	8.5	0.03	0.62	0.31	0.04	16	53	67	<0.009	< 0.05
6034	9.4	0.08	2.7	0.76	0.21	90	410	140	0.01	0.07
6035	5.6	0.02	1.6	0.40	0.26	73	380	86	<0.008	< 0.05
6036	4.7	0.03	0.78	0.94	0.14	41	27	45	<0.005	0.07
6037	6.7	0.09	1.0	0.12	0.04	8.9	35	59	< 0.006	< 0.05
6038	8.6	0.07	1.6	0.33	0.61	4.4	29	970	<0.01	0.08
6039	1.9	0.11	0.66	0.17	0.01	20	18	57	0.09	< 0.05
6040	5.8	0.02	0.80	0.21	0.18	200	88	37	0.007	< 0.05
6041	3.9	0.08	1.2	0.23	0.25	37	120	110	0.009	0.17
6042	11	0.02	2.7	0.17	0.02	21	33	250	<0.008	0.07
6043	2.5	0.03	0.34	0.08	0.02	15	18	58	<0.002	<0.05
6044	9.6	0.03	1.6	0.19	0.05	16	48	270	< 0.008	0.06
6045	1.1	0.02	0.48	0.13	0.12	23	160	55	<0.005	< 0.05
6046	30	0.05	0.79	0.70	0.02	14	72	510	< 0.008	<0.05
6047	62	0.02	1.9	0.04	0.007	3.7	42	43	<0.003	<0.05
6048	6.5	<0.006	0.92	0.16	0.03	21	56	160	< 0.009	<0.05
6049	3.1	0.009	0.31	0.06	0.03	3.6	12	54	< 0.005	<0.05
6050	33	0.29	1.8	0.74	0.06	25	110	160	< 0.01	<0.05

Sample No.	Concentration in parts per million by weight (dry basis)										
	Bi	Pb	Tl	I	Te	Cd	Mo	Se	As	Ge	Zn
6051	<0.02	34	<0.006	8.9	0.01	1.1	0.37	14	0.21	7.4	290
6052	<0.008	1.3	<0.004	3.4	<0.002	0.54	0.16	1.8	<0.002	0.77	57
6053	<0.02	1.6	<0.005	0.7	<0.004	1.3	0.22	3.9	<0.005	0.45	110
6054	<0.01	0.92	<0.003	0.99	0.003	0.12	0.10	2.0	<0.003	0.13	40
6055	<0.02	1.2	<0.006	1.4	0.004	1.4	0.39	3.4	<0.005	0.58	110
6056	<0.01	1.0	<0.004	6.2	<0.004	0.44	0.20	2.7	<0.004	1.2	120
6057	0.02	1.4	0.01	46	<0.003	0.50	0.29	3.1	<0.004	0.44	96
6058	<0.006	0.43	<0.002	1.9	<0.002	0.10	0.10	1.3	<0.002	0.17	22
6059	0.07	0.75	<0.01	50	0.02	0.60	0.92	9.4	<0.01	2.1	420
6060	<0.03	0.52	<0.009	120	<0.007	0.50	0.30	4.9	<0.009	1.2	120
6061	<0.02	2.4	<0.007	7600	<0.005	0.26	0.16	6.6	<0.007	0.38	82
6062	<0.01	0.98	<0.003	2.1	<0.002	0.27	0.11	2.4	<0.003	0.19	38
6063	<0.05	4.6	0.01	95	<0.01	1.6	0.75	17	<0.01	0.91	48
6064	<0.02	4.8	<0.006	830	<0.006	0.51	0.31	26	<0.06	2.0	160
6065	0.02	1.5	<0.006	1900	<0.005	0.70	0.47	2.8	0.76	0.41	110
6066	<0.01	3.0	<0.003	490	<0.003	3.0	0.31	9.9	0.006	0.22	70
6067	<0.02	0.33	<0.006	1.2%	<0.005	2.0	0.39	0.46	<0.006	0.51	490
6068	<0.02	1.1	<0.005	1200	<0.004	0.16	0.16	4.7	0.005	0.27	51
6069	<0.007	0.34	<0.002	1.1	<0.001	0.24	0.06	1.2	<0.002	0.21	29
6070	<0.008	0.40	<0.003	1.8	<0.002	0.11	0.07	2.0	<0.003	0.19	35
6071	<0.008	0.50	<0.002	0.88	<0.002	0.27	0.09	5.6	<0.02	0.33	30
6072	<0.04	3.9	<0.01	7.1	<0.01	0.63	0.85	4.0	<0.13	1.1	120
6073	<0.02	0.87	<0.005	390	<0.005	0.62	0.20	4.0	<0.06	0.30	48
6074	0.05	1.9	<0.01	8.7	<0.009	0.58	2.1	7.3	<0.12	2.0	210
6075	<0.01	0.5	<0.004	0.62	<0.004	0.50	0.16	3.2	<0.04	0.28	100

Sample No.	Concentration in parts per million by weight (dry basis)									
	Cu	Co	Mn	Cr	V	Ti	Al	Mg	Be	Hg
6051	40	0.59	100	3.5	0.64	90	320	180	<0.008	0.08
6052	4.4	0.02	1.4	1.4	0.10	15	45	96	<0.004	0.15
6053	6.8	0.03	0.73	0.24	0.08	25	74	170	<0.008	< 0.05
6054	8.5	< 0.009	0.69	0.21	0.04	13	33	92	<0.005	0.13
6055	12	0.21	0.95	0.20	0.13	65	38	130	< 0.008	< 0.05
6056	13	0.04	1.9	0.38	0.11	62	160	150	<0.007	0.08
6057	11	0.10	0.84	0.17	0.05	42	22	110	<0.007	< 0.05
6058	2.8	0.02	0.17	0.03	< 0.004	5.1	6.6	36	<0.003	0.07
6059	43	0.17	2.7	0.55	0.14	130	35	240	<0.02	< 0.05
6060	13	0.05	1.7	0.21	0.04	11	86	150	<0.01	< 0.05
6061	8.6	0.04	2.1	0.35	0.07	31	350	62	<0.01	< 0.05
6062	4.9	0.03	0.89	0.20	0.11	21	31	52	<0.005	< 0.05
6063	22	0.02	2.1	0.42	0.07	100	35	250	<0.02	< 0.05
6064	15	0.09	3.1	1.2	0.60	160	130	78	<0.01	< 0.05
6065	8.9	0.04	1.2	3.8	0.06	37	31	110	<0.009	< 0.05
6066	5.1	0.05	0.64	0.42	0.09	29	26	29	<0.006	< 0.05
6067	8.5	0.13	0.55	0.80	0.06	9.3	15	73	<0.01	< 0.05
6068	7.8	0.03	0.86	0.18	0.12	39	46	78	<0.008	< 0.05
6069	7	0.006	0.11	0.03	0.01	17	11	36	<0.004	< 0.05
6070	4.4	0.01	0.13	0.05	0.01	15	13	84	<0.004	< 0.05
6071	1.9	0.04	0.70	0.15	0.4	49	50	80	0.008	< 0.05
6072	23	0.09	2.5	0.50	0.15	86	130	220	0.03	< 0.05
6073	10	0.03	1.1	0.52	0.12	34	55	95	<0.009	< 0.05
6074	17	0.12	4.0	1.1	0.06	46	5.8	40	<0.02	< 0.05
6075	13	0.11	0.88	0.09	0.02	95	7.6	76	<0.007	< 0.05

Sample No.	Concentration in parts per million by weight (dry basis)										
	Bi	Pb	Tl	I	Te	Cd	Mo	Se	As	Ge	Zn
6076	< 0.02	8.2	< 0.005	15	< 0.004	5.9	1.8	8.0	0.06	1.0	85
6077	< 0.02	3.3	< 0.005	68	< 0.004	2.3	3.7	7.6	< 0.05	0.37	120
6078	< 0.02	1.6	< 0.005	3.2	< 0.004	1.1	0.31	5.6	< 0.005	0.67	110
6079	< 0.02	1.2	< 0.005	7.2	< 0.004	0.22	0.34	4.0	< 0.005	0.33	160
6080	0.79	4.1	< 0.006	630	< 0.005	0.67	1.0	8.3	< 0.06	0.82	94
6081	< 0.02	2.7	< 0.006	3.9	0.006	2.7	0.50	6.0	< 0.06	1.2	80
6082	< 0.03	0.76	< 0.01	2.7	0.01	0.23	0.27	3.6	< 0.01	3.9	230
6083	< 0.02	0.37	< 0.006	0.65	0.006	0.14	0.22	4.8	< 0.006	0.32	38
6084	< 0.02	2.7	< 0.006	5.9	0.03	0.63	0.73	11	< 0.006	0.76	180
6085	< 0.04	0.62	< 0.01	8.6	< 0.009	0.27	0.41	13	< 0.01	0.76	780
6086	< 0.03	2.8	< 0.009	12	< 0.007	0.95	0.57	6.8	< 0.009	0.86	250
6087	0.03	3.3	< 0.004	5.8	< 0.003	0.97	0.27	2.3	0.005	0.66	46
6088	< 0.01	2.8	< 0.004	14	< 0.003	0.89	0.14	12	< 0.004	0.50	55
6089	0.04	3.7	< 0.01	2700	< 0.008		0.69	6.9	< 0.01	2.3	490
6090	< 0.01	1.5	< 0.004	4.4	< 0.004	0.36	0.28	2.9	< 0.004	0.61	60
6091	< 0.02	0.25	< 0.006	0.82	< 0.005	0.98	0.21	1.5	< 0.006	0.25	37
6092	< 0.02	0.79	< 0.007	3.5	< 0.006	0.32	0.24	2.2	< 0.07	0.25	85
6093	< 0.04	3.1	< 0.01	1.8	< 0.01	1.7	0.22	3.1	< 0.01	1.2	64
6094	< 0.009	1.8	< 0.003	18	< 0.002	0.30	0.20	1.8	< 0.03	0.29	39
6095	< 0.02	1.9	< 0.006	4.2	< 0.005	2.9	0.45	5.4	< 0.06	1.2	110
6096	< 0.009	4.1	< 0.003	7.7	< 0.002	0.29	0.20	3.6	< 0.03	0.31	60
6097	< 0.02	8.0	< 0.007	20	< 0.01	5.7	0.50	10	< 0.07	2.2	220
6098	0.009	0.86	0.003	8.1	0.002	1.4	0.21	4.2	< 0.03	0.23	59
6229	<0.07	2.5	< 0.02	14	< 0.03	0.46	1.0	7.1	< 0.02	2.1	720
6230	0.02	3.9	<0.003	11	<0.003	0.55	0.57	11	< 0.003	0.89	190
6231	< 0.008	0.75	0.002	3.3	0.002	0.78	0.12	1.9	< 0.002	0.15	86

Sample No.	Concentration in parts per million by weight (dry basis)									
	Cu	Co	Mn	Cr	V	Ti	Al	Mg	Be	Hg
6076	8.5	1.4	10	1	0.31	1400	50	85	< 0.009	< 0.05
6077	11	0.16	0.97	1.1	0.10	280	83	16	< 0.008	< 0.05
6078	7.5	0.05	2.2	0.32	0.05	56	8.5	23	< 0.008	0.13
6079	25	0.03	0.95	0.15	0.03	7.3	2.6	33	< 0.009	< 0.005
6080	8.5	0.09	2.0	0.94	0.30	94	130	100	< 0.009	< 0.05
6081	13	0.08	1.1	0.22	0.10	34	55	96	< 0.009	< 0.05
6082	4.8	0.03	0.76	0.30	0.05	32	19	90	< 0.02	< 0.05
6083	8.4	0.04	0.60	0.08	0.01	11	6.2	100	< 0.01	< 0.05
6084	15	0.08	2.0	4.1	0.21	62	28	47	< 0.01	< 0.05
6085	35	0.18	4.8	0.66	0.30	270	58	75	< 0.02	< 0.05
6086	14	0.05	1.6	0.58	0.65	80	42	55	< 0.01	< 0.05
6087	11	0.05	0.83	0.12	0.04	28	21	26	< 0.007	< 0.05
6088	6.8	0.12	1.2	0.26	0.04	27	7.2	25	< 0.007	< 0.05
6089	27	0.16	3.1	2.3	0.27	130	52	180	< 0.02	< 0.05
6090	6.5	0.03	0.77	0.29	0.21	39	41	30	< 0.007	< 0.05
6091	3.3	0.07	0.56	0.17	0.02	4.4	2.2	29	< 0.01	< 0.05
6092	24	0.04	0.64	0.35	0.07	30	8.1	110	< 0.01	< 0.05
6093	8.3	0.04	1.3	0.49	0.13	78	63	76	< 0.02	0.26
6094	6.3	0.02	0.10	0.04	0.01	9.6	9.6	44	< 0.004	<0.05
6095	14	0.09	1.2	0.42	0.13	140	16	35	< 0.009	<0.05
6096	12	0.04	1.4	0.60	0.65	72	13	43	< 0.004	< 0.05
6097	20	0.22	3.2	2.6	0.05	31	11	61	< 0.01	< 0.05
6098	2.3	0.03	0.26	0.15	0.02	42	27	24	< 0.005	< 0.05
6229	54	0.02	2.0	0.40	0.21	330	38	100	< 0.03	< 0.05
6230	5.6	0.05	3.2	0.39	0.81	23	60	28	0.008	< 0.05
6231	4.1	0.02	0.86	0.20	0.44	42	34	40	0.006	<0.05

Sample No.	Concentration in parts per million by weight (dry basis)										
	Bi	Pb	Tl	I	Te	Cd	Mo	Se	As	Ge	Zn
6099	<0.019	1.8	<0.006	8.1	<0.005	1.3	0.77	7.1	<0.06	0.37	76
6100	<0.007	1.5	<0.002	6.3	<0.001	0.48	0.16	3.3	<0.02	0.22	70
6101	0.03	3.3	<0.002	6.2	<0.001	0.47	0.16	3.2	0.03	0.21	38
6102	<0.007	0.33	<0.002	2.9	0.01	0.16	0.16	3.2	<0.02	0.49	97
6103	0.01	3.8	<0.003	9.6	<0.002	1.2	0.18	3.8	<0.02	0.42	150
6104	<0.004	0.77	<0.002	1.6	<0.001	0.56	0.15	1.8	<0.01	0.19	40
6105	<0.009	0.83	<0.003	6.8	<0.002	1.3	0.20	4.0	<0.03	0.16	69
6106	<0.008	1.6	<0.02	6.8	<0.002	1.1	0.17	4.6	<0.02	0.31	76
6107	0.007	1.3	<0.002	120	0.001	0.08	0.72	2.1	<0.02	0.45	57
6108	<0.01	1.7	0.005	300	<0.002	0.14	0.22	5.7	<0.003	0.39	71
6109	0.04	18	<0.003	9.5	0.003	1.6	2.4	5.0	0.13	1.0	100
6110	<0.02	1.8	<0.006	4.0	<0.004	0.11	0.16	3.3	<0.02	0.63	48
6111	<0.02	1.9	<0.006	85	<0.005	0.69	0.27	8.5	<0.06	5.8	110
6112	<0.01	2.7	<0.004	280	<0.003	0.27	0.29	4.8	<0.04	0.25	130
6113	<0.008	8.4	<0.005	73	<0.004	1.7	0.96	17	<0.05	0.54	290
6114	0.02	2.8	<0.004	700	<0.004	3.5	0.31	9.6	<0.04	0.24	87
6115	<0.007	0.67	<0.002	0.63	<0.001	1.1	0.16	2.6	<0.02	0.28	38
6116	<0.02	1.9	<0.006	8.4	<0.005	0.29	0.46	18	<0.06	0.83	63
6117	<0.03	1.4	<0.009	0.69	<0.008	1.1	0.69	11	<0.09	0.46	130
6118	0.03	4.0	<0.005	66	<0.004	4.6	0.35	12	<0.005	0.41	240
6119	0.18	2.6	<0.005		<0.004	0.53	0.24	8.4	<0.005	0.47	120
6120	<0.02	1.1	0.007	120	<0.005	0.32	0.58	3.3	<0.006	0.44	130
6121	<0.009	5.8	<0.003	42	<0.003	1.7	0.95	5.8	2.0	0.58	120
6122	<0.02	9.1	<0.007	460	<0.005	4.1	0.73	4.5	<0.007	0.70	220

Sample No.	Concentration in parts per million by weight (dry basis)									
	Cu	Co	Mn	Cr	V	Ti	Al	Mg	Be	Hg
6099	9.1	0.04	1.2	0.45	0.09	110	10	34	<0.009	< 0.05
6100	5.5	0.53	1.1	0.32	0.11	32	39	19	<0.004	< 0.05
6101	4.1	0.01	0.12	0.55	0.02	20	10	6.9	<0.003	< 0.05
6102	5.4	0.007	0.69	1.2	0.08	32	10	12	<0.003	< 0.05
6103	11	0.08	1.3	0.23	0.07	32	16	14	<0.006	< 0.05
6104	4.0	0.01	0.85	0.07	0.01	35	0.85	9.6	<0.002	0.09
6105	3.8	0.009	0.86	0.19	0.04	18	2.6	15	0.03	< 0.05
6106	7.2	0.008	1.2	0.04	0.02	44	6.2	21	<0.004	0.23
6107	5.7	0.02	0.86	0.24	0.16	41	41	55	<0.003	0.11
6108	4.6	0.003	0.83	0.14	0.03	14	14	25	<0.005	< 0.05
6109	9.5	0.05	61	1.3	0.56	96	190	29	0.005	< 0.05
6110	7.3	0.01	0.85	0.63	0.06	55	16	19	<0.009	0.19
6111	11	0.04	1.2	0.23	0.13	58	17	56	<0.01	< 0.05
6112	11	0.06	2.1	0.15	0.08	37	19	49	<0.007	< 0.05
6113	8.7	0.38	2.2	0.20	0.05	36	8.7	48	<0.009	0.10
6114	5.5	0.13	1.4	0.64	0.09	32	20	37	<0.006	0.07
6115	7	0.01	0.31	0.05	0.01	13	2.1	7	<0.004	0.17
6116	4.7	0.03	0.58	0.17	0.13	33	30	55	<0.009	0.09
6117	21	0.19	1.8	0.28	0.05	76	9.5	32	<0.02	< 0.05
6118	8.6	0.17	2.3	1.8	0.47	120	53	47	<0.008	0.11
6119	6.1	0.02	0.98	0.39	0.01	13	16	53	<0.009	< 0.05
6120	17	0.12	1.1	0.14	0.03	42	2.8	29	<0.009	0.11
6121	5.2	0.08	2.9	0.78	0.50	170	56	31	0.009	>> 1
6122	16	0.07	3.2	1.2	0.40	170	120	56	0.01	0.43

Sample No.	Concentration in parts per million by weight (dry basis)										
	Bi	Pb	Tl	I	Te	Cd	Mo	Se	As	Ge	Zn
6123	< 0.03	2.5	0.02	7.4	< 0.007	1.2	0.36	4.4	< 0.009	1.1	120
6124	0.03	1.8	0.009	230	< 0.006	0.53	0.41	3.5	< 0.008	0.34	100
6125	0.01	1.2	< 0.003	6.5	< 0.002	0.13	0.27	2.7	< 0.004	0.42	120
6126	< 0.01	0.52	0.004	460	< 0.003	0.72	0.43	2.3	< 0.003	0.40	160
6127	< 0.02	5.8	0.006	140	0.006	0.25	0.23	6.3	< 0.005	0.49	210
6128	0.02	1.1	< 0.005	1000	< 0.004	2.0	0.35	11	< 0.005	0.44	130
6129	0.04	10	< 0.004	120	< 0.004	10	0.33	6.9	< 0.004	0.60	200
6130	< 0.01	1.6	< 0.004	15	< 0.003	6.49	0.08	2.2	0.009	0.3	78
6155	0.02	0.39	0.004	3.4	0.002	0.11	0.16	3.4	< 0.007	0.33	76

Sample No.	Concentration in parts per million by weight (dry basis)									
	Cu	Co	Mn	Cr	V	Ti	Al	Mg	Be	Hg
6123	11	0.009	0.85	0.30	0.01	7.3	7.7	45	<0.01	0.19
6124	7.8	0.07	1.1	0.46	0.11	52	19	55	<0.01	< 0.05
6125	5.3	0.07	1.5	0.3	0.23	55	30	52	0.006	< 0.05
6126	11	0.04	0.72	0.25	0.06	33	16	28	<0.005	< 0.05
6127	8.6	0.01	0.59	0.13	0.02	25	26	86	<0.009	< 0.05
6128	11	0.08	4.4	1.2	0.47	94	69	30	0.01	0.13
6129	13	0.06	0.80	2.8	0.26	53	26	37	<0.008	< 0.05
6130	6.6	0.12	0.66	0.14	0.05	30	9	30	<0.006	0.09
6155	5.6	0.02	1.8	0.28	0.73	79	180	25	0.004	< 0.05

Sample No.	Concentration in parts per million by weight (dry basis)										
	Bi	Pb	Tl	I	Te	Cd	Mo	Se	As	Ge	Zn
6133	<0.01	1.0	<0.009	5.9	<0.008	0.10	0.20	2.9	<0.19	1.2	59
6140	<0.01	1.7	0.02	8.4	<0.01	4.0	0.55	4.1	0.53	2.1	110
6141	<0.03	1.1	0.06	5.4	6.0	2.7	0.33	5.3	0.51	1.2	59
6143	<0.03	1.6	0.04	82	0.01	2.2	0.95	5.9	0.39	1.4	110
6144	<0.02	0.38	0.03	1.1	<0.007	0.68	0.16	1.6	<0.15	0.08	18
6145	<0.01	0.32	<0.007	3.3	<0.009	0.15	0.19	2.5	0.42	1.0	19
6146	<0.03	10	0.06	54	0.02	0.56	7	16	6.7	8.1	140
6147	<0.02	0.47	<0.003	1.8	0.01	0.21	0.22	1.9	<0.15	0.56	62
6148	0.01	0.27	<0.002	14	0.004	0.12	0.06	1.4	0.73	0.60	10
6149	0.01	0.64	0.02	5.1	0.008	0.35	0.14	2.2	0.83	0.76	67
6150	0.03	1.0	<0.004	4.8	0.01	0.41	0.28	7.4	1.3	1.2	70
6151	<0.09	56	0.04	27	0.04	1.9	4.0	13	<0.80	5.5	300
6152	<0.15	2.0	0.20	10	0.11	1.3	1.4	7.2	5.9	3.6	120
6153	<0.02	2.2	0.02	53	0.009	0.61	0.27	8.2	0.16	4.1	83
6154	<0.06	15	<0.01	7.1	0.01	2.8	0.58	8.4	1.5	2.5	150
6156	0.03	0.90	0.03	90	0.006	1.1	0.35	4.4	<0.29	1.5	70
6157	<0.04	2.5	0.04	120	0.008	1.8	0.39	5.7	1.6	3.2	120
6158	0.11	3.4	0.01	780	0.01	0.45	0.51	12	<0.23	1.7	110
6159	<0.01	0.91	0.003	440	0.008	1.3	0.29	3.1	<0.13	0.65	130
6160	<0.03	1.8	0.01	89	<0.01	31	0.59	6.4	2.5	1.4	60
6161	0.02	1.9	0.007	77	0.007	1.2	0.28	4.9	<1.3	1.1	93
6162	<0.009	0.27	<0.002	2.7	<0.004	0.31	0.18	3.1	<0.9	0.31	53
6163	0.20	2.6	<0.004	3200	0.02	1.3	0.29	5.4	0.40	0.97	94
6164	0.71	11	<0.009	490	0.02	2.2	1.2	29	1.6	5.3	570
6166	<0.03	1.8	<0.003	9.3	<0.005	5.4	0.72	6.1	<0.3	1.7	200

Sample No.	Concentration in parts per million by weight (dry basis)									
	Cu	Co	Mn	Cr	V	Ti	Al	Mg	Be	Hg
6133	7.0	0.10	7.2	1.4	2.0	1800	210	1000	0.02	< 0.05
6140	170	0.29	6.4	3.8	2.0	1900	570	4900	0.03	< 0.05
6141	15	0.25	5.4	4.9	5.7	1500	1500	3800	0.02	< 0.05
6143	6	0.05	1.1	0.95	6	760	560	5800	0.02	0.10
6144	3.8	0.09	0.71	1.8	0.63	250	99	990	0.008	< 0.05
6145	4.7	0.86	2.3	3.7	1.7	810	220	1400	0.06	0.2
6146	38	0.81	30	9.6	26	6500	7200	5600	0.14	< 0.05
6147	3.6	0.22	2.0	1.1	2.5	2000	660	1700	0.008	< 0.05
6148	2.1	0.13	1.4	0.50	0.34	810	380	2000	0.02	0.13
6149	5.5	0.14	2.3	1.4	2.1	870	420	1600	0.03	< 0.05
6150	26	0.28	2.2	0.78	2.2	1600	650	6200	0.03	< 0.05
6151	420	0.12	0.37	0.97	0.33	380	66	1.9%	<0.04	< 0.05
6152	15	1.6	13	4.2	8.5	6500	6500	8800	0.29	< 0.05
6153	7.5	0.06	7.3	2.4	3.9	7500	790	1800	0.03	0.20
6154	15	0.73	6.1	5.5	4.6	37	0.32	1.3%	0.06	< 0.05
6156	21	0.10	4.5	4.2	6.1	2600	500	6400	0.06	0.26
6157	23	0.22	18	5.8	8.5	3600	1.7%	4500	0.08	0.20
6158	58	0.35	5.9	3.5	9.5	2200	1100	5500	0.05	< 0.05
6159	16	0.05	3.3	1.6	1.0	620	230	3100	0.01	0.13
6160	27	0.20	13	4.1	6.0	1900	610	1200	0.03	< 0.05
6161	17	0.13	6.0	1.9	1.0	1200	190	1500	0.01	
6162	11	0.09	0.38	0.25	0.10	77	25	950	0.005	< 0.05
6163	9.1	0.24	2.6	5	0.92	890	430	2200	0.04	< 0.05
6164	66	5.1	44	11	16	9900	7100	9100	0.16	< 0.05
6166	35	0.66	4.9	4.9	2.3	850	250	2000	0.02	0.13

Sample No.	Concentration in parts per million by weight (dry basis)										
	Bi	Pb	Tl	I	Te	Cd	Mo	Se	As	Ge	Zn
6167	0.006	0.73	0.002	2.6	<0.002	0.43	0.1	0.34	0.07	0.59	59
6168	<0.02	8.8	<0.004	4.9	<0.007	0.88	0.39	2.9	<0.18	0.84	100
6169	0.03	1.1	<0.007	1.1	0.009	1.4	0.21	6.2	<0.17	1.3	120
6170	0.03	2.9	0.009	68	0.01	1.6	0.45	7.9	8.6	3.3	12
6171	0.02	0.77	<0.001	2.1	0.003	0.52	0.05	2.2	3.1	0.83	29
6172	0.10	0.72	<0.005	5.3	<0.01	2.8	0.22	2.3	<0.21	1.2	140
6173	0.13	2.1	0.01	6.8	<0.005	2.4	0.24	8.1	0.22	0.95	110
6174	0.02	10	0.02	30	0.009	0.83	0.66	7.7	<1.8	2.2	200
6175	0.10	7.1	0.02	70	0.01	0.41	0.46	11	<0.21	5.3	140
6176	0.39	11	0.007	11	0.007	1.6	0.36	8.4	0.34	1.7	230
6177	<0.01	0.41	0.02	4.1	<0.005	0.47	0.27	2.0	0.52	0.67	64
6178	<0.03	0.97	0.02	270	<0.006	0.93	0.23	22	<0.32	1.3	160
6179	<0.008	2.7	0.004	48	0.004	1.7	0.42	28	<0.9	0.68	80
6180	<0.02	4.4	0.007	7.1	0.02	1.5	0.82	11	1.5	4.1	290
6181	0.06	6.5	0.03	5.2	0.02	2.7	0.98	16	0.66	6.1	170
6182	<0.03	1.4	0.06	8	0.02	1.1	0.63	7.3	2.6	0.75	20
6183	<0.05	6.0	0.04	14	0.03	3.5	0.81	5.4	<0.41	4.9	110
6184	0.03	0.85	0.02	4.4	0.005	1.2	0.57	2.7	0.28	3.2	67
6185	<0.02	1.5	0.009	7.0	<0.009	0.01	0.92	9.3	<0.21	1.7	230
6186	<0.02	2.9	0.009	3.3	0.01	0.77	0.44	4.8	0.36	2.6	150
6187	0.10	7.3	0.02	350	<0.01	2.5	0.42	19	0.91	4.1	110
6188	<0.03	25	<0.005	16	<0.01	0.51	1.1	0.67	<0.26	0.87	85
6189	<0.006	0.81	0.005	92	0.003	0.11	0.12	2.3	0.24	0.46	41
6190	0.05	2.8	0.009	120	<0.009	2.9	0.44	22	0.25	1.1	150
6191	<0.02	1.3	0.008	6.2	<0.008	0.36	0.4	3.0	<0.18	1.5	130
6192	0.21	27	0.08	29	0.02	0.90	1.2	18	1.0	2.9	170

Sample No.	Concentration in parts per million by weight (dry basis)									
	Cu	Co	Mn	Cr	V	Ti	Al	Mg	Be	Hg
6167	7.1	0.07	4.3	1.1	1.3	1000	480	1100	0.005	< 0.05
6168	56	0.07	0.26	0.17	1.9	140	46	6700	<0.007	< 0.05
6169	41	0.22	15	4.9	2.3	3300	190	810	1.7	0.07
6170	16	0.72	19	6.3	4.6	4200		2400	0.04	< 0.05
6171	6.3	0.13	2.9	1.4	1.3	1900	55	1500	0.008	0.23
6172	11	0.07	3.0	1.2	1.7	1500	960	5000	0.01	< 0.05
6173	27	0.32	5.1	2.5	1.9	1900	490	1200	0.009	0.13
6174	20	0.16	6	7.8	6	2800	2700	3100	0.02	< 0.05
6175	28	0.07	5.3	2.7	8.5	2900	1400	2400	0.01	0.13
6176	17	0.06	1.6	3.7	1.3	780	380	1900	0.009	< 0.05
6177	7.3	0.06	1.2	0.79	0.98	580	180	1400	<0.007	< 0.05
6178	39	0.04	4.9	0.97	2.4	9.7	270	3500	< 0.02	
6179	12	0.43	2.1	1.9	0.67	780	94	960	0.005	< 0.05
6180	110	0.25	0.55	6.5	4.8	4400	490	2500	0.04	0.26
6181	40	0.24	300	36	55	1.5%	2800	3600	0.07	0.2
6182	12	0.1	14	23	2.4	1000	650	5000	0.02	
6183	31	0.31	19	13	9.3	8500	950	4800	0.04	< 0.05
6184	24	0.04	4.3	2.1	3.8	2500	170	3100	0.01	< 0.05
6185	41	0.12	5.3	4.0	4.7	3600	140	2400	0.01	0.13
6186	22	0.14	19	11	8.2	4100	910	2300	0.04	< 0.05
6187	41	0.55	10	6.5	4.8	4400	490	2500	0.04	< 0.05
6188	19	0.09	0.2	0.24	0.09	100	12	2600	< 0.01	< 0.05
6189	4.2	0.04	0.92	0.84	1.3	530	100	640	0.006	< 0.05
6190	22	0.32	5.1	3.0	8.2	2400	300	1300	0.02	< 0.05
6191	24	0.21	8.6	5.6	7.5	3800	280	2100	0.03	< 0.05
6192	30	0.43	11	7	9.5	3700	540	2800	0.05	< 0.05

Sample No.	Concentration in parts per million by weight (dry basis)										
	Bi	Pb	Tl	I	Te	Cd	Mo	Se	As	Ge	Zn
6193	0.01	2	0.01	91	0.005	0.86	0.54	5.3	0.45	2	110
6194	0.05	27	0.02	390	0.01	1.9	1.2	18	9.8	8.9	170
6195	< 0.05	14	< 0.01	150	< 0.02	3.6	2.2	15	< 0.5	3.6	170
6196	0.35	23	0.01	2300	0.009	0.54	0.72	13	1.5	1.8	180
6197	0.04	2.5	0.02	3400	< 0.008	1.5	0.86	4.1	< 0.17	5.4	120
6198	< 0.03	1.4	0.03	9.2	0.02	1.1	0.47	10	0.35	4.9	140
6199	0.03	0.005	0.01	1	0.008	1.2	0.32	3.0	0.18	1.5	76
6200	< 0.05	1	0.02	8.6	< 0.01	0.96	1.1	8.3	< 0.55	4.3	200
6202	< 0.02	1.4	0.02	3.6	< 0.01	0.85	0.01	5.3	< 0.23	2.6	120
6203	0.01	18	0.007	12	< 0.007	1.4	0.82	6.3	< 0.17	1.3	86
6204	< 0.02	1.2	0.02	3.6	< 0.008	1.8	0.35	2.7	1.3	0.58	89
6205	0.06	11	0.02	5.8	< 0.01	3.6	0.90	9.9	< 3.6	2.9	180
6206	0.08	26	0.02	13	0.04	1.7	3.1	12	< 0.83	3.0	2.0
6207	< 0.03	9.0	0.01	19	< 0.01	2.4	1.4	6.7	0.58	3.3	200
6208	< 0.02	11	0.004	560	< 0.008	6.5	0.84	6.4	< 0.18	5.3	180
6210	2.2	7.4	0.11	1800	0.05	8.3	2.8	13	2.2	10	100
6211	< 0.07	12	< 0.02	43	< 0.03	3.1	1.4	10	0.71	5.1	140
6212	< 0.02	2.4	0.008	2.9	0.009	1.3	0.38	3.0	0.55	1.4	94
6213	< 0.02	0.59	0.02	6.1	< 0.008	0.43	1.9	3.0	0.23	1.5	130
6214	0.08	5.1	0.02	6	< 0.02	0.71	0.8	5.9	< 0.38	7	150
6215	0.05	3.5	0.05	17	0.04	2.9	0.55	9.4	< 0.26	4.7	160
6216	0.02	2.5	0.04	60	0.01	0.69	0.45	9.1	0.28	2.1	130
6217	3.5	27	< 0.02	490	< 0.04	3.2	8.6	21	< 0.86	3.1	160
6218	< 0.10	32	< 0.02	9.9	< 0.05	2.3	3.8	13	< 0.10	12	250
6219	< 0.01	4.0	0.01	200	< 0.005	4.2	0.61	6.2	0.52	2.2	88

Sample No.	Concentration in parts per million by weight (dry basis)									
	Cu	Co	Mn	Cr	V	Ti	Al	Mg	Be	Hg
6193	26	0.14	4.8	3.2	4.3	1500	240	910	0.01	< 0.05
6194	25	0.58	11	12	19	4000	3500	4000	0.1	< 0.05
6195	210	0.07	2	2	0.76	210	130	5100	< 0.03	< 0.05
6196	44	0.41	6.5	11	11	3300	320	1200	0.02	
6197	23	0.07	8.0	8.6	7.1	6100	310	2000	0.04	< 0.05
6198	16	0.21	13	4.1	6.2	5700	500	3200	0.03	0.82
6199	19	0.36	6.7	2.2	3.2	1400	220	1700	0.02	< 0.05
6200	40	0.36	24	3.4	11	4900	1200	6000	0.05	< 0.05
6202	29	0.33	10	6.7	9	4500	400	1400	0.05	< 0.05
6203	9.3	< 0.004	1.5	1.3	2.3	1500	290	1900	0.01	< 0.05
6204	8.7	0.19	3.2	2.2	1.5	850	410	2500	0.02	< 0.05
6205	18	0.12	1.6	1.5	2.9	580	230	8000	0.02	< 0.05
6206	130	0.04	0.75	0.52	0.64	1300	440	4.0%	0.04	< 0.05
6207	25	0.04	4.6	4.2	2.2	2600	500	3200	0.06	< 0.05
6208	130	0.06	4.3	2.5	2.4	1600	92	3900	0.01	< 0.05
6210	57	0.37	24	12	13	2.5%		3300	0.24	< 0.05
6211	67	0.03	1.2	0.93	0.50	590	190	7100	< 0.03	0.13
6212	13	0.15	4.4	2.6	2.5	800	310	4000	0.04	0.75
6213	13	0.07	0.36	0.27	0.15	86	56	2100	< 0.03	< 0.05
6214	48	0.36	6	11	5.3	3500		4200	0.06	1.3
6215	15	0.14	12	3.7	10	5000	1100	2400	0.04	0.26
6216	11	0.10	4.5	5.4	4.0	1700	410	3100	0.04	< 0.05
6217	88	0.02	1.6	1.9	1.4	1400	250	1.9%	< 0.05	< 0.05
6218	250	<0.02	94	9.0	4.8	4700	84	2.3%	< 0.05	< 0.05
6219	16	0.09	3.1	3.7	1.8	580	110	1400	0.01	< 0.05

Sample No.	Concentration in parts per million by weight (dry basis)										
	Bi	Pb	Tl	I	Te	Cd	Mo	Se	As	Ge	Zn
6220	0.05	7.3	0.05	140	0.04	3.0	0.87	8.5	0.47	7.3	85
6221	0.01	0.73	<0.003	11	0.006	1.3	0.22	2.6	<0.12	0.58	71
6222	<0.07	43	<0.02	11	<0.03	3.5	340	11	6.9	2.1	150
6223	<0.02	7.6	0.01	16	0.02	8.1	0.51	8.9	0.99	2.8	75
6224	<0.02	3.6	0.01	12	0.008	1.5	6.5	4.1	5.3	1.9	110
6225	<0.01	0.60	0.02	4.1	0.01	1.0	250	1.9	<0.26	0.37	140
6226	0.01	0.60	0.03	4.6	0.007	2.1	0.47	4.7	2.3	2.0	130
6227	<0.03	3.5	0.05	79	0.02	24	0.54	19	<0.26	6.4	180
6228	0.02	2.4	0.02	12	0.008	7.6	340	11	0.73	6.5	230
6232	0.08	0.39	0.03	20	3.0	2.7	1.6	9.8	<0.64	4.9	300
6233	0.04	5.7	0.03	97	0.01	0.68	130	7.9	0.22	3.6	110
6234	0.02	1.8	0.01	24	0.009	1.7	740	8.1	<0.15	1.8	410

Sample No.	Concentration in parts per million be weight (dry basis)									
	Cu	Co	Mn	Cr	V	Ti	Al	Mg	Be	Hg
6220	18	0.26	21	6.9	9.3	1.2%	520	4400	0.05	< 0.05
6221	28	0.03	2.6	1.1	0.73	400	34	1300	< 0.006	< 0.05
6222	35	< 0.01	0.30	0.69	0.53	310	140	1.3%	< 0.03	< 0.05
6223	18	0.27	6.0	7.1	5.3	2200	540	27%	0.11	< 0.05
6224	15	0.51	13	4.4	2.6	2100	1300	3400	0.03	< 0.05
6225	26	0.04	0.48	0.94	0.43	410	37	2100	< 0.01	< 0.05
6226	13	0.32	2.6	3.2	1.2	1600	210	3200	0.04	0.20
6227	24	8.6	6.3	72	2.0	4400	370	2900	0.04	< 0.05
6228	25	0.12	4.4	2.6	3.9	4900	260	2000	0.02	0.13
6232	70	0.16	5.6	9.1	4.9	2800	780	3800	0.06	< 0.05
6233	15	0.34	9.7	6.4	7.2	3100	400	2000	0.04	0.13
6234	19	0.13	3.4	1.5	1.9	2900	660	2000	0.01	

VIII. SPECIFIC DETAILS PERTINENT TO EACH ELEMENT EXAMINED IN THE STUDY

Arsenic

This element is one of those standardized in NBS bovine liver SRM 1577.

The bovine liver standard was ashed using the method of plasma ashing using an r.f. power of 200 watts and an oxygen flow of 200 cc/min. These conditions were used in the ashing of all the submitted samples.

Isotopes Examined m/e 75 and 75^{2+} which occurs at m/e 37.5

Possible Interferences

m/e 75 is interfered by $^{40}Ca\ ^{35}Cl^{+}$ species
m/e 37.5 is not interfered

Aluminum

Aluminum is an element present but not certified in the bovine liver standard SRM 1577.

Aluminum therefore being an element having only one isotope was standardized using the method of standard addition.

Isotopes Examined m/e 27 and $^{27}Al^{2+}$ at m/e 13.5

Possible Interferences

$^{54}Fe^{2+}$ and $^{54}Fe^{4+}$

Aluminum is at a rather high concentration in the samples and consequently, the degree of interference from the ion species listed above is small.

A problem encountered on the determination of aluminum is due to its high concentration. The accuracy in the determination of this element may be limited.

Beryllium

Beryllium is not present in the NBS bovine liver standard. Standardization was not possible versus the bovine liver SRM 1577 and since beryllium has only one isotope at m/e 9 it is not possible to use the method of isotopic dilution.

Consequently, a synthetic standard was prepared by adding a known quantity of beryllium in solution to an aliquot of the ashed bovine liver standard.

Isotope Examined m/e 9

Possible Interferences

$^{18}O^{2+}$, $^{27}Al^{3+}$,

The concentration of beryllium in the samples analyzed is below the limit of detection of the method used. The synthetic standard allowed estimation of sensitivity to ascribe a limit of detection for this element.

Bismuth

Bismuth is not standardized in SRM 1577, consequently, individual standardization for this element was carried out.

In this case, since bismuth is undetectable in the bovine liver standard, a synthetic standard was prepared by adding a bismuth solution to the bovine liver ash.

Bismuth is mono-isotopic, consequently, isotope dilution techniques could not be applied, either to assess recovery on ashing or the relative sensitivity of the element.

Isotope Examined m/e 209

Possible Interferences

None

Comments:

Bismuth has proved to be at extremely low concentration in most samples, particularly the lung tissue.

Cadmium

Cadmium is one of the elements standardized in the NBS bovine liver standard SRM 1577.

The bovine liver standard was ashed by the method of plasma ashing using a r.f. power of 200 watts with an oxygen flow of 200 cc/min. These conditions were used in the ashing of all samples submitted.

Isotope Examined m/e 114 and m/e 111

Possible Interferences

$^{114}Sn^{+}$ on $^{114}Cd^{+}$, Since Sn is at variable concentration, this did present problems on m/e 114 and consequently m/e 111 was also examined, in order to check isotopic abundance ratios.

Another possible interfering ion is $^{39}K^{40}Ca^{35}Cl^+$, whilst the probable intensity of this ion species is low, it must be considered as a potential source of error.

Chromium

Chromium is an element not standardized in the bovine liver standard SRM 1577. Chromium has isotopes at m/e 50, m/e 52, m/e 53 and m/e 54.

Chromium is a volatile element and it was hoped to use the combustion/collection method to assess recovery by "spiking" with ^{53}Cr before combustion. The combustion/collection process was not successful due to variable recovery efficiency.

Standardization for chromium was carried out by isotope dilution using a ^{53}Cr isotope solution additional to the ashed bovine liver.

Isotopes Examined $^{52}Cr^+$, $^{53}Cr^+$

Possible Interferences

$^{28}Si^{24}Mg^+$, $^{40}Ca^{12}C^+$, $^{26}Mg^{2+}$, $^{25}Mg^{27}Al^+$

$^{52}Cr\ H^{37}Cl^{16}O^+$, $^{41}K^{12}C^+$, $^{26}Mg^{27}Al^+$, $^{40}Ca^{13}C^+$

The isotope dilution experiments were carried out at a high resolving power, in order to aleviate these interferences.

Cobalt

Cobalt is one of the elements standardized in the NBS bovine liver standard SRM 1577.

Cobalt is a mono-isotopic element and may be subject to some interference particularly in the presence of high concentration of fluoride.

Isotopes Examined m/e 59 and m/e 29.5

Possible Interferences

$^{40}Ca^{19}F^+$, $^{24}Mg^{35}Cl^+$, $^{12}C_2\ ^{35}Cl^+$, $^{43}Ca^{16}O^+$, $^{118}Sn^{2+}$

Notes:

It is essential in most biological materials that cobalt be examined only in high resolution conditions due to the potential problems with spectral overlap.

Copper

Copper is one of the elements standardized in the NBS bovine liver standard SRM 1577.

Both isotopes of copper appear to be clear of any interfering molecular ions at our available resolving power.

Isotopes Examined m/e 63 and m/e 65

Possible Interferences

$^{39}K^{12}C_2^+$, $^{39}K^{24}Mg^+$, $^{31}P^{16}O_2^+$, on m/e 63.

Germanium

Germanium is an element not certified in the NBS bovine liver standard SRM 1577.

In this case isotopic dilution methods were employed to determine the concentration and relative sensitivity of this element.

Isotopes Examined m/e 73 and m/e 74

Possible Interferences

$^{57}Fe^{16}O^+$, $^{56}Fe^{16}OH^+$, $^{28}Si^{29}Si^{16}O^+$, $^{41}K^{16}O_2^+$,

$^{24}Mg^{24}Mg^{25}Mg^+$, $^{37}Cl^{12}C_3^+$, $^{25}Mg^{16}O_3^+$, $^{24}Mg^{12}C\ ^{13}C^+$,

$^{24}Mg^{12}C_3{}^{13}C^+$, all on m/e 73

$^{37}Cl_2^+$, $^{39}K^{35}Cl^+$, $^{57}Fe^{16}OH^+$, $^{24}Mg^{25}Mg_2^+$,

$^{26}Mg^{16}O_3^+$, $^{24}Mg_2{}^{13}C_2^+$

All of the above molecules are probably at low intensity in the spectrum but they would constitute a definite problem at low resolving power. However the use of high resolving power alleviates the problem of serious interference.

One possible problem is the fact that in order to produce of solution of the ^{73}Ge isotope, which was as GeO_2, alkali fusion was employed. The quantitative transfer of ^{73}Ge is more open to doubt than in the case of a directly soluble material.

Lead

Lead is one of the elements standardized in the NBS bovine liver standard SRM 1577.

The probability of interference by molecular species on the isotopes of lead is very low due to the high atomic number of lead.

Isotopes Examined m/e 208

Possible Interferences

None

Manganese

Manganese is one of the elements standardized in the NBS bovine liver standard SRM 1577.

Manganese is mono-isotopic and fortunately in this case no gross interference is expected.

Isotopes Examined m/e 55

Possible Interferences

$^{31}P^{24}Mg^{+}$, $^{31}P^{12}C_2^{+}$

Magnesium

Magnesium is one of the elements standardized in the NBS bovine liver standard SRM 1577.

Magnesium has three naturally occurring isotopes

Isotopes Examined

m/e 24, m/e 25 and m/e 26 were examined to confirm identity of magnesium. However the concentration of magnesium was too high in all cases to make any concentration assessment.

Consequently, the line at m/e 12.5 which is the second order spectrum line of 25 Mg was examined.

Possible Interferences

None at m/e 12.5 and at the high concentration of magnesium.

Comments:

The very high concentration of magnesium in all materials introduced a somewhat unusual problem since it is not a trace element, the line spectra were of such high intensity that accurate measurement was very diffucult and in some cases virtually impossible. It is recommended that chemical or emission spectrographic determination of this element be considered rather than mass spectrometry. In order to determine magnesium more accurately by mass spectrometry, at least one of the following courses of action would have to be followed:

a. Employ greater sample dilution.
b. Use electrical analysis at very low gain
c. Employ isotopic dilution and electrical detection "peak switch" methods.

However, the analysis scheme chosen was for the determination of "trace elements", as indicated by contract title and the proposal. The determination of magnesium by a more appropriate method actually means re-analyses of each sample, this was not possible within the structure of time allottment or negotiated price of the contract.

Molybdenum

Molybdenum is one of the elements standardized in the NBS bovine liver standard SRM 1577.

The bovine liver standard was ashed by the method of plasma ashing using a r.f. power of 200 watts with an oxygen flow of 200 cc/min. These conditions were used in the ashing of all the samples submitted.

Isotopes Examined m/e 98

Possible Interferences

$^{66}Zn^{16}O_2^+$

The probability of interference in this case is extremely low.

Selenium

Selenium is one of the elements standardized in the NBS bovine liver standard SRM 1577.

Selenium isotopes were examined in each of the samples and their relative intensity assessed versus the ashed bovine liver spectra.

Isotopes Examined m/e 80 m/e 82

Possible Interferences

$^{40}Ca_2^+$ at m/e 80

$^{41}K_2^+$ at m/e 82

Tellurium

Tellurium is not detectable in the bovine liver standard. Consequently the method of isotope dilution or standard addition could not be employed. The bovine liver standard was used as the matrix and a known amount of tellurium in solution was added to produce a synthetic standard.

Isotopes Examined m/e 126, m/e 128 m/e 130

Possible Interferences

$^{63}Cu_2^+$, $^{63}Cu^{65}Cu^+$, $^{65}Cu_2^+$
at m/e 126,128, and 130 respectively

The production of molecular ions is dependant upon the concentration of copper in the materials under analysis.

In the samples submitted, the copper concentration is sufficiently low to give negligible contribution on the tellurium spectrum.

Thallium

Thallium is an element not standardized in SRM 1577, consequently standardization was considered by isotopic dilution. The natural isotopes are at m/e 203 and m/e 205, isotope dilution was not practical due to the interference on the m/e 205 isotope by the $^{13}C^+$ isotope at the C_{17}^+ molecule.

Standardization was therefore carried out using the method of addition of a standard solution of thallium to the ashed bovine liver, and one of the lung samples in which thallium was not detected. This allowed an assessment of the relative sensitivity of thallium.

Isotopes Examined m/e 203 and m/e 205

Possible Interferences

No interference on m/e 203
m/e 205 Tl interfered by 12.13 C_{17}^+ ions

Comments:

Thallium has proven to be at extremely low levels in all samples, normally below the limits of detection.

Iodine

Iodine is an element which had to be standardized by an independent method since it is not standardized in the bovine liver SRM 1577.

Iodine is mono-isotopic and of course is also very volatile, consequently it is impossible to assess recovery on this element using isotope dilution, and some undefined losses may occur in the plasma ashing.

Standardization was carried out by the method of standard addition of a sodium iodide solution in several aliquots to the ashed bovine liver standard SRM 1577. Analysis of the doped bovine liver ash gave a linear plot of density versus concentration and allowed an assessment of the absolute sensitivity for iodine.

Isotopes Examined m/e 127 and m/e 63.5 ($^{127}I^{2+}$)

Possible Interferences

$^{115}In\ ^{12}C^+$

The degree of potential interference on this element is extremely low and is assessed to be insignificant.

Comments:

There was considerable variation in the observed concentration of this element in many samples, and the question to be answered at this time is that of iodine contamination during autopsy.

Titanium

Titanium is an element that is not certified in the NBS bovine liver standard SRM 1577.

In the investigation it was assessed using the method of isotopic dilution in a ^{47} Ti isotope "spike".

Isotopes Examined m/e 47, m/e 48, m/e 49.

Possible Interferences

$^{31}P^{16}O^+$, $^{35}Cl^{12}C^+$, $^{29}Si^{18}O^+$ on m/e 47

$^{48}Ca^+$, $^{32}S^{16}O^+$, $^{24}Mg_2^+$, $^{35}Cl^{13}C$, $^{30}Si^{18}O$, $^{16}O_3^+$, $^{24}Mg^{12}C_2^+$, $^{12}C_4^+$ on m/e 48

$^{37}Cl\ C^+$, $^{24}Mg^+$, $^{16}O_2\ ^{17}O^+$, $^{12}C_3\ ^{13}C^+$ on m/e 49

Comments:

Isotopic dilution was carried out on the m/e 47 isotope, and this was ratioed to the m/e 48 isotope, and checked against the m/e 49 isotope, The ratios of m/e 23.5 and m/e 24.5, were checked to validate identification of the m/e 47, and 49, isotopes of titanium.

Bovine liver contains 123 ppm of calcium and therefore maximum contribution on the m/e 48 is 0.22 ppm.

Vanadium

Vanadium is an element present but not standardized in the NBS bovine liver standard SRM 1577.

Vanadium therefore was standardized using standard addition methods.

Isotopes Examined m/e 51

Possible Interferences

$^{24}Mg^{27}Al^{+}$, $^{28}Si^{23}Na^{+}$, $^{25}Mg^{26}Mg^{+}$, $^{27}Al^{12}C_2^{+}$

Some of the molecules listed above have quite a high probability of formation. However at high resolving power the $^{51}V^{+}$ isotope is separated.

Zinc

Zinc is one of the elements standardized in the NBS bovine liver standard SRM 1577.

The probability of interference by molecular species on the isotopes of zinc is rather large and high resolving power must be used to obviate possible errors due to misidentification.

Isotopes Examined m/e 66 m/e 67

Possible Interferences

$^{40}Ca^{26}Mg^{+}$, $^{54}Fe^{12}C^{+}$, $^{33}S_2^{+}$, $^{39}K^{27}Al^{+}$, $^{34}S^{16}O_2^{+}$, $^{25}Mg_2^{16}O^{+}$

$^{27}Al_2^{12}C^{+}$, on m/e 66

$^{33}S^{34}S^{+}$, $^{28}Si^{39}K^{+}$, $^{54}Fe^{13}C^{+}$, $^{40}Ca^{27}Al^{+}$ on m/e 67

APPENDIX I

USE OF THE FLUORIDE MODIFIED LOW TEMPERATURE PLASMA ASHING METHOD

One sample of ashed lymph node was supplied by NIOSH as an additional piece of work outside the normal scope of the contract. The work was undertaken with the mutual aggreement of Mrs. Sweet of NIOSH and R. Brown of Accu-Labs Research, no additional expense was involved to NIOSH.

The object of this work was to examine what factors are influenced by the use of fluorine low temperature plasma ashing.

The results of the analysis are included in this appendix of this final report, and it may be seen that there is nothing very unusual about the reported values.

The comment that can be made at this time is that for mass spectrometric analysis the use of this method of ashing is not as suitable as the conventional excited oxygen ashing.

The presence of excited fluorine atoms results inthe formation of stable fluoride containing compounds which produced rather troublesome interference on some possibly critical elements. The combinations listed below indicate some of the overlaps:

a. $^{40}Ca^{19}F^{+}$ at m/e 59 which is the only available isotope of cobalt.

b. $^{56}Fe^{19}F^{+}$ at m/e 75 which is the only available isotope of arsenic.

c. $^{39}K^{19}F^{+}$ at m/e 58, this is a major isotope of nickel.

d. $^{88}Sr^{19}F^{+}$ which occurs at m/e 107, this is one of the isotopes of silver.

These examples are but a few of the possible interferences resulting from the presence of large concentrations of fluoride in the samples ashed by this method.

It should be stressed however that these problems may be perculiar only to mass spectrometry.

Due to the fact that information on the ratio of ash weight/dry weight was not available on the node tissue, values are reported relative to the ash.

EXCITED FLUORINE PLASMA ASHED LYMPH NODE

(Reported on Ash Basis)

Element	Conc. in ppm weight
Bismuth	4.3
Lead	56
Thallium	0.24
Iodine	8.6
Tellurium	0.09
Cadmium	40
Molybdenum	18
Selenium	60
Arsenic	27
Germanium	35
Zinc	1900
Copper	210
Manganese	96
Chromium	195
Vanadium	23
Titanium	3%
Aluminum	3900
Magnesium	4%
Beryllium	0.27

APPENDIX II

COMBUSTION/CONDENSATION SAMPLE PREPARATION

This method was investigated to provide an alternative to the low temperature plasma ashing used on the dried lungs and lymph nodes.

It was felt that it might provide a method giving better recovery for the volatile elements, and elements with volatile chlorides and oxychlorides.

In order to assess its efficiency, chromium was the element examined. Several samples of lung tissue and the bovine liver standard were spiked with a ^{53}Cr isotope in order to distort the natural isotopic abundance ratio of $^{52}Cr^{53}Cr$. The amount of ^{53}Cr isotope was added so as to produce equal intensities of ^{52}Cr and ^{53}Cr based upon a rough estimate of the chromium indigenous in the samples.

Our current report on this effort is negative in that it appears that the integrity of ratio $^{52}Cr/^{53}Cr$ in the sample and the ^{53}Cr from the "spike" is not maintained sufficiently to allow routine use of the method.

It is felt however that under more controlled conditions and with more investigation of the parameters involved success may be achieved, the time allotted in this project precluded any more extensive investigation at this time.

APPENDIX III

During the course of the analysis for the 21 elements specified in the contract, certain observations were made throughout the range of samples. Many elements showed a very wide range of concentration throughout the lungs and the lymph nodes. Whilst the levels of concentration for most elements were higher in the hilar lymph nodes than in the lungs, in both classes there were significant concentration differences.

The following elements were the ones which showed the greatest variation in concentration, tantalum, niobium, antimony and tin, mainly in the lungs. In the hilar lymph nodes variation was noted in the following additional elements uranium, thorium, samarium, neodymium, cerium, lanthanum, barium, etc.

Whilst an exhaustive study on these elements would have significantly increased the comittments on interpretation it may be worth future consideration. All the raw data is available as photoplate spectra and at any future date, re-examination of the plates may yield information on the elements not included in this present study.

APPENDIX IV

The analysis of mercury was carried out by burning a weighed portion of the freeze dried lungs and/or lymph nodes in a stream of oxygen. The mercury is then pre-concentrated by passing the vapor through a gold coil packed tube. The tube is heated to deamalgamate the mercury, which is then passed through a quartz cell mounted in an atomic absorption spectrophotometer.

The quantity of mercury vapor is actually determined by measuring the absorbance of a beam of mercury radiation at it's most intense wave length (i.e. 2536Å). The system is calibrated by running the standard in a similar fashion. Since Beer's law is obeyed, a direct ratio of obsorbances yields the concentration of mercury in the sample.

In addition, a hydrogen continuum, double beam, background connection system was employed to optically subtract hydrocarbon, water vapor, or any other species that obsorbs at the same wave length as mercury.

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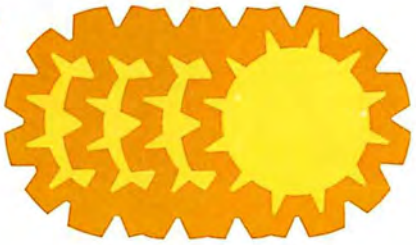

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