PERKIN ELMER

THE PERKIN-ELMER CORPORATION
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AN IMPORTANT ADVANCE FOR INDUSTRIAL HYGIENISTS

As an Industrial Hygienist, one of your concerns is the determination of trace quantities of potentially toxic metallic elements.

You are undoubtedly familiar with the new instrumental technique called Atomic Absorption which enables you to measure more than 38 metallic and semi-metallic elements at the trace level, quickly and simply.

As an example, lead in water solution has a detection limit by Atomic Absorption of 0.15 ppm (this translates to 0.15 mg/liter, or 0.015 mg/100g, or 0.06 mg/m^3 in air.) With the use of a single, non-specific extraction and concentration step, the detection limit can be improved by a factor of about 15.

The basic advantages of Atomic Absorption are speed, sensitivity, precision and relative freedom from chemical and spectral interferences. To illustrate the technique and some of its benefits, we invite your attention to the attached articles:

1. "Detecting One Part in a Million," an article describing the technique and its capabilities in simple and general terms.
2. "Detection Limits by Atomic Absorption," a table giving the current detection limits of the technique.
3. "Atomic Absorption Newsletter No. 17," one of a monthly series of newsletters published by Perkin-Elmer. This issue deals with trace metals in body fluids.
4. "Summary of Newsletters," which briefly describe the newsletters to date. Any or all may be obtained free of charge.

Atomic Absorption Spectroscopy is the newest, fastest growing analytical technique not only in your field, but in Metallurgy, Clinical Chemistry, Biochemistry, and throughout general chemical research. It is our belief that if this technique is not already included among your analytical capabilities, it is rapidly going to become one of your most important tools.

For further information please use the enclosed reply card.

Very truly yours,

THE PERKIN-ELMER CORPORATION

Herbert L Kahn

Herbert L. Kahn

Detecting One Part in a Million . . . Super Sleuthing

By HERBERT L. KAHN, The Perkin-Elmer Corporation

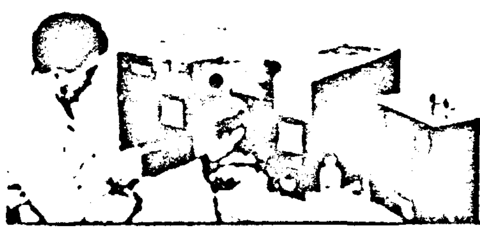
Metals or metal compounds, present in substances at concentrations of less than one part per million, can perform some astonishing tricks. They can mean the difference between an insulator and a semi-conductor, they can spoil the taste of canned foods, they can indicate the presence of wear in an engine, they can poison the catalyst in a petroleum refining process, they can poison you.

No wonder, therefore, that science and industry have been seeking to detect and measure these metals for decades, and that they have brought a multitude of tools to the task. Wet chemistry, flame and arc emission, X-ray fluorescence and neutron activation analysis have been among the means employed.

Recent years have seen the development of yet another technique to detect metals. Atomic absorption, as it is called, combines simplicity of operation, sensitivity, versatility, and precision, to make it at once an important supplement and a powerful rival to the established means of measuring the concentration of metals. In atomic absorption, the sample is vaporized, usually by a flame. Light is passed through the vapor—light emitted by a lamp whose cathode is made of the metal being sought. The lamp emits energy only at certain wavelengths, which comprise the so-called excitation spectrum of the metal. The metal atoms in the sample, on the other hand, can absorb energy at only those wavelengths. The percentage of the light absorbed by the sample, therefore, is a direct and specific measure of the concentration of the desired metal in it.

Old And Yet New

Since the principle of atomic absorption is so simple, one might suppose that apparatus has been under development ever since Fraunhofer made his first spectral observations early in the 19th century.



Extremely minute concentrations of metals or metal compounds in such diversified substances as piston rings for tiny internal combustion engines developed for moon-space vehicles, simple insulating materials, or petroleum catalysts can wreak havoc for the research scientist; to his aid, however, comes the atomic absorption technique, which is at once an important supplement and powerful rival to the established means of measuring the concentration of metals in materials.

If not, one might certainly theorize that it began when Kirchhoff explained what it was that Freunhofer saw.

Actually, references to the atomic absorption phenomenon do appear in the technical literature of 100 years ago, together with some hints of what applications the principle might have. Astronomers built a few instruments for their own special uses. However, the analytical chemists, the most numerous group to whom atomic absorption might have been useful, paid it little attention. A chance hoof-beat from our era's galloping technology knocked atomic absorption into a crevice, where it remained until the Australian physicist Alan Walsh happened to notice its glitter in the early 1950's. Walsh realized the importance of the technique, and developed the first instrumentation that could give general and useful analytical results. It soon became evident that atomic absorption had unparalleled sensitivity to zinc and magnesium, which had been difficult or impossible to measure by other means.

As the years passed, other metals were also determined with a sensitivity and precision which first rivalled, and then in many cases surpassed, other techniques. However, many of the initial applications were buried in Australian agricultural and mining journals, with the result that atomic absorption took several years to make any impression "up over." Even now, the estimated hundred atomic absorption instruments in Australia are double the number in use in the USA, although this ratio is changing very rapidly.

Advantages Of Atomic Absorption

By atomic absorption, whose basic system is shown in Figures 1 and 2, it is now possible to determine the concentration of over 40 metals at levels of less than one part-per-million. Table 1

FIGURE - 1 THE ATOMIC ABSORPTION SYSTEM

The hollow cathode lamp emits the spectrum of the elements being sought. Its output is split into two beams, one of which passes through the flame in which the sample is being vaporized. The degree of absorption depends on the concentration of the desired element in the sample. The sample beam is then recombined with the reference beam, passed through a monochromator which screens out unwanted radiation, and brought to a photodetector and to the electronics.

FIGURE - 2 ATOMIZER AND BURNER SYSTEM

In this premix burner, the sample is aspirated through a thin capillary tube by the air flowing into the atomizer section. The air/sample mixture emerges from the atomizer as a fine spray of droplets, which is then mixed with the fuel, usually acetylene. The mixture is rendered turbulent by the flow spoilers, and is then forced up into the burner head.

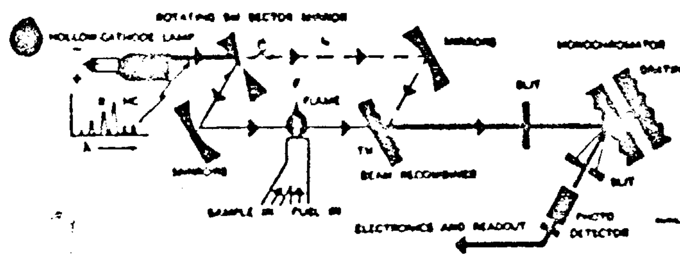


Figure 1

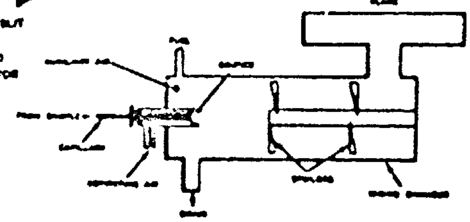


Figure 2

shows approximate detectability limits for some of these. A few of the characteristics that give atomic absorption certain advantages over previous methods are:

Simplicity—The essential simplicity of the technique makes it possible to install equipment at a modest first cost, to run it without highly-trained personnel, and to produce analyses at considerable speeds.

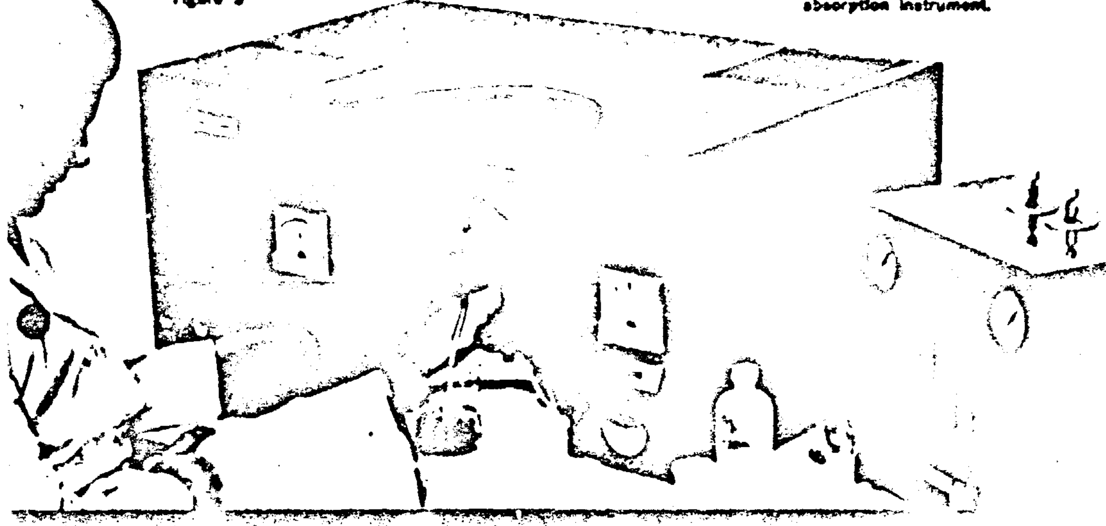
Sensitivity—In contrast to the requirements of *emission* spectroscopy, the burner shown in Figure 2 need only vaporize the sample. There is no need for the atoms to reach a high-energy, or "excited" state. For many metals, it is considerably easier to produce a vapor than to excite the atoms. Such metals are detected far more sensitively by absorption than by any emission technique.

Precision—The fact that the metallic atoms need not be excited, together with the possibility of using a double-beam technique (Figure 1), makes it possible to obtain highly reproducible and dependable results with a minimum of adjustments.

Specificity—Atomic absorption is remarkably free from interferences, which are defined as any factor in the sample tending to complicate an analysis. Only the metal being sought can absorb the emission lines of the source. There is, therefore, no possibility of *optical* interference. The possibility of *chemical* interference still remains, but is considerably smaller and easier to remove than in most other techniques. (When chemical interference takes place, other constituents in the sample affect the results from the metal being sought. This problem arises in all element detection methods, but

Determination of metal concentrations in parts per million possible with this atomic absorption instrument.

Figure 3



seems to affect atomic absorption very little).

Applications for atomic absorption are increasing at an accelerating pace. A few of the uses reported during the past year appear below:

1. It is possible to obtain precise information about the wear of engine parts by measuring the concentration of various metals in the lubricating oil. In a recent study, it was shown to be quite easy to determine the levels of copper, iron, chromium, lead, silver, barium, and sodium in the crankcase oil of railroad engines.
2. Atomic absorption now furnishes a simple, precise method of measuring lead in gasoline.
3. The presence in crude oils of iron, copper, nickel and sodium eventually poisons the catalyst during the cracking cycle. The ability of an atomic absorption instrument to perform 450 analyses per day has realized important savings in the cracking process for one firm.
4. Atomic absorption has entered the forefront of semi-conductor research. Tellurium and other trace impurities are studied in a variety of semi-conductor materials.
5. Atomic absorption has also been used successfully to measure tin, antimony, and copper in glass.
6. Metallurgy is another field where atomic absorption is coming into its own. A recent study disclosed that it was easy to determine low-level concentrations of zinc in bauxite. In England, magnesium is being determined routinely in nickel and nickel alloys. It is little or no problem to determine aluminum concentration in steel.
7. It has been found possible to detect concentrations of less than one part per billion of copper, iron, manganese, nickel and zinc in sea-water, without any sample pre-treatment, according to a recent article.
8. Sodium, iron, magnesium, copper and bismuth have been determined at low levels in a synthetic sweetener, with no sample preparation except solution in distilled water.

Monthly Newsletter Available

In order to provide a convenient way to keep up with the mushrooming literature on atomic absorption, our firm publishes a monthly newsletter, which contains up-to-date bibliographies, summaries, and other items of interest.

Much of the early work on atomic absorption was done by adapting existing instrumentation on a do-it-yourself basis. In 1957, articles describing different designs began to appear in technical journals, and shortly thereafter, combinations of "building-block" components were offered commercially.

TABLE I – APPROXIMATE DETECTION LIMITS FOR SOME METALS

METAL	CONC. IN PARTS/MILLION
Cadmium	.01
Chromium	.01
Copper	.005
Iron	.05
Manganese	.01
Strontium	.04
Zinc	.005

Atomic absorption spectroscopy went through the same development process as conventional absorption spectroscopy had done, although at an accelerated pace. The first units were of the single-beam type, in which the results were directly dependent upon source and detector stability. In many, the radiation from the source was not chopped, which made it necessary for the operator to compensate in some way for emission from the flame.

Modern instrumentation employs a double-beam, chopped-light system to maximize stability and operator convenience. With one of the newer atomic absorption spectrophotometers such as our Model 303 (Figure 3), it is possible to set up an analysis in under 10 minutes, not counting sample preparation. A determination is typically made in less than one minute.

The immediate future can be expected to show a big increase in the number of atomic absorption applications in the detection of metals. Many scientists have already shown a desire to use atomic absorption to test also for phosphorus, sulphur, and iodine, whose resonance lines lie in the far ultraviolet spectral region. In this region it is necessary to purge the optical path with nitrogen, which requires the replacement of the flame by some kind of heating element able to work without oxygen. Experiments with several types of devices are under way, and sufficiently encouraging data are on hand to ensure eventual success.

In the more distant future, there probably lies a vacuum instrument capable of checking for oxygen, nitrogen, and hydrogen. Another strong possibility is an atomic absorption direct reader, capable of testing for many elements at one time. One thing is sure, the young field of atomic absorption spectroscopy will continue to be exciting for a long time to come. ■

Reprinted from RESEARCH/DEVELOPMENT Magazine, June, 1963

LIA11551

DETECTION LIMITS IN ATOMIC ABSORPTION SPECTROPHOTOMETRY

The numbers below represent the present analytical detection limits of elements by atomic absorption spectrophotometry. Except where noted, the limit is that obtained in water solution and with an air-acetylene flame. The use of organic solvents can produce an improvement of 3-10 times in the detection limit.

All the values for detection limit for a given element shown on this sheet are identical--merely expressed in different units. The detection limit is defined as the concentration giving a signal equal to twice the variability of the background. The detection limits in air are given in mg/m^3, and assume an analysis where 1 $ft.^3$ of air is drawn through an impinger, and its content dissolved in 10 cc of water.

ELEMENT	DETECTION LIMIT μg/ml mg/liter ppm	mg/100g	mg/m^3 Air
Ag	0.02	0.002	0.008
Al*	0.5	0.05	0.2
As	1	0.1	0.4
Au	0.1	0.01	0.04
Ba	1	0.1	0.4
Be*	0.05	0.005	0.02
Bi	0.2	0.02	0.08
Ca	0.01	0.001	0.004
Cd	0.01	0.001	0.004
Co	0.15	0.015	0.06
Cr	0.01	0.001	0.004
Cs	0.05	0.005	0.02
Cu	0.005	0.0005	0.002
Fe	0.05	0.005	0.02
Hg	0.5	0.05	0.2
K	0.005	0.0005	0.002
Li	0.005	0.0005	0.002
Mg	0.003	0.0003	0.0015
Mn	0.01	0.001	0.004
Mo	0.2	0.02	0.08
Na	0.005	0.0005	0.002
Ni	0.05	0.005	0.02
Pb	0.15	0.015	0.06
Pd	1	0.1	0.4
Pt	0.5	0.05	0.2
Rb	0.02	0.002	0.008
Rh	0.3	0.03	0.15
Sb	0.2	0.02	0.08
Se	1	0.1	0.4
Sn	2	0.2	0.8
Sr	0.02	0.002	0.008
Te	0.5	0.05	0.2
Ti*	1	0.1	0.4
Tl	0.2	0.02	0.08
V*	0.5	0.05	0.2
Zn	0.005	0.0005	0.002

* An organic solvent and oxyacetylene flame are required.

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SUMMARY OF ATOMIC ABSORPTION NEWSLETTERS AND APPLICATIONS REPRINTS

This describes the Atomic Absorption Newsletters published at Perkin-Elmer. All of them are available free of charge on request. Unless specified, the authors are employees of the Applications Laboratory at Perkin-Elmer.

No. 1 - March, 1962
"BIBLIOGRAPHY AND GENERAL APPLICATIONS"
Presents a very brief outline of atomic absorption applications to biological, agricultural, metallurgical, and miscellaneous problems. Also includes a bibliography of 58 articles published on atomic absorption up to early 1962.

No. 2 - April, 1962
"BIOLOGICAL APPLICATIONS OF ATOMIC ABSORPTION SPECTROPHOTOMETRY"
This surveys briefly methods for the determination of Ca, Mg, Na & K in blood, various metals in urine, and other biological applications.

No. 3 - May, 1962 Superseded by Newsletter No. 13

No. 4 - June, 1962
"AGRICULTURAL APPLICATIONS OF ATOMIC ABSORPTION SPECTROPHOTOMETRY"
This is a survey of the work done on the analysis of metals is soils and plant materials. Particular success is reported with zinc, magnesium, calcium, sodium and potassium; also copper, iron, manganese and molybdenum are found to have few or no interferences.

No. 5 - July, 1962
"THE APPLICATION OF ATOMIC ABSORPTION SPECTROPHOTOMETRY TO METALLURGY"
This is a survey of the determination of constituents of alloys. High sensitivity and precision are found for the detection of zinc. Good agreement is found with Certificate values for lead in various standard alloys. Studies are also quoted on magnesium in nickel and aluminum alloys and the determination of manganese, cadmium, molybdenum, platinum and others.

No. 6 - September, 1962 Superseded by Newsletter No. 16

No. 7 - October, 1962
"ATOMIC ABSORPTION SPECTROPHOTOMETRY WITHOUT THE USE OF FLAMES"
Also "ANALYTICAL OPERATING DATA FOR THE MODEL 214 SPECTROPHOTOMETER"
A discussion of various proposed methods to avoid the use of flames in sampling. At present none of the methods are yet commercial.

No. 14 - August, 1963
"THE MODEL 303 ATOMIC ABSORPTION SPECTROPHOTOMETER"
A technical description of the new instrument. Also, "CEMENT ANALYSIS" - the description of an analysis for calcium in cement, performed to great precision by the use of scale expansion. Also brief descriptions of the determination of other metals in cement.

No. 15 - September, 1963
"THE DETERMINATION OF CALCIUM BY ATOMIC ABSORPTION SPECTROPHOTOMETRY"
A discussion in considerable detail of the instrumental parameters, interferences and precisions obtainable in the determination of calcium by atomic absorption.

No. 16 - October, 1963
"BIBLIOGRAPHY TO APPLICATIONS OF ATOMIC ABSORPTION SPECTROPHOTOMETRY"
A listing of 79 further articles on atomic absorption, together with a brief breakdown of articles according to their various fields of interest.

No. 17 - January, 1964
"TRACE METALS IN BLOOD AND URINE" also "CERTAIN TOXICOLOGICAL TRACE METALS"
This describes successful determinations of trace quantities of various metals, particularly Cu and Fe, in blood and urine, and gives detection limits for 13 metals in urine without sample preparation. There is also a paper on toxicological trace metal determination by Perkin-Elmer personnel together with F. Rieders and V. Cordova of the Medical Examiners office in Philadelphia. This describes methods for the determination of small quantities of cadmium, chromium, thallium, cobalt, and lead in blood and urine.

No. 18 - February, 1964
"DETECTION LIMITS IN ATOMIC ABSORPTION SPECTROPHOTOMETRY"
Detection limits with the Model 303 are given for presently determinable elements. Careful discussions are presented of the difference between sensitivity and detection limit, enhancement by organic solvents, and requirements for refractory metals. A short evaluation of alternative sampling methods is also given.

No. 19 - March, 1964
"DETERMINATION OF THE MAJOR METALS IN GRANITIC AND DIABASIC ROCKS" also "Cu AND Zn IN BAYER PROCESS LIQUOR" by C. B. Belt, St. Louis University.
A description of experiments leading to the determination of sodium, potassium, calcium, magnesium, manganese and iron in silicate rocks, with good agreement demonstrated with spectrochemical and chemical methods. There is also an article on the determination of copper and zinc in Bayer process liquor by C.B. Belt, Jr. of St. Louis University.

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No. 8 - November, 1962
"LITHIUM ISOTOPE ANALYSIS BY ATOMIC ABSORPTION SPECTROPHOTOMETRY"
A preliminary report on the determination of the Li^6 and the Li^7 isotopes. Initial results look promising.

No. 9 - December, 1962
"ATOMIC ABSORPTION SPECTROCHEMICAL ANALYSIS OF PLANT MATERIALS WITH PARTICULAR REFERENCE TO MANGANESE AND IRON" by D.J. David, C.S.I.R.O. Australia.
A description of successful determinations of iron, manganese, sodium, potassium, calcium, magnesium, zinc, iron and strontium in plant materials, with indications of the method in each case.

No. 10 - February, 1963
"A BURNER ATOMIZER FOR ATOMIC ABSORPTION SPECTROPHOTOMETRY"
A description of the design and performance of the burner system developed for the Model 303.

No. 11 - March, 1963
"SOME BIOLOGICAL APPLICATIONS OF ATOMIC ABSORPTION SPECTROPHOTOMETRY"
A compilation of several brief articles. "TRACE METALS IN BLOOD PLASMA", "ZINC IN URINE", "IRON IN PROTEIN SOLUTIONS", and "RECOVERY OF Ca, Mg, and Zn in BIOLOGICAL MATERIALS".

Describes the determination of cadmium, chromium, manganese and molybdenum at their natural levels in plasma. There are also discussions of zinc in urine, iron in protein solutions and calcium, magnesium and zinc in various biological materials. A preliminary analysis is also described for calcium, magnesium, sodium and potassium in bone ash.

No. 12 - April, 1963
"THE APPLICATION OF ATOMIC ABSORPTION SPECTROSCOPY TO THE ANALYSIS OF PETROLEUM PRODUCTS" also "DETERMINATION OF THE METAL CONTENT OF LUBRICATING OILS".
This includes a review of published work on the determination of lead in gasoline, copper, iron and nickel in crude oils, and sodium in oils. There is also a paper on determination of the metal content of worn lubricating oils with results for iron, copper, lead, silver, barium and sodium.

No. 13 - May, 1963
"MAGNESIUM, CALCIUM AND ZINC IN ANIMAL NUTRITION" by H.E. Parker of Purdue University, Also "SOME DETERMINATIONS OF Ca and Mg IN BLOOD".
Successful methods of analysis for the metals named in various minerals, feeds, and tissues. There is also a description of determinations made at Perkin-Elmer of calcium and magnesium in blood.

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No. 20 - May, 1964
"DETERMINATION OF SELENIUM AND TELLURIUM IN COPPER BY ATOMIC ABSORPTION SPECTROPHOTOMETRY" and " DETERMINATION OF VERY SMALL AMOUNTS OF COPPER AND LEAD IN KCl BY ORGANIC EXTRACTION AND ATOMIC ABSORPTION SPECTROPHOTOMETRY" also "CESIUM AND RUBIDIUM ON THE MODEL 214 ATOMIC ABSORPTION SPECTROPHOTOMETER".

Methods are given for the determination of selenium and tellurium in copper, with detection limits of .005% selenium and .005% tellurium. Analysis time is less than 10 minutes per sample.

Also, an extraction procedure for traces of lead and copper from solid KCl is given. Applicability of the method is demonstrated for several other elements.

No. 21 - June, 1964
"THE DETERMINATION OF STRONTIUM IN BIOLOGICAL MATERIALS BY ATOMIC ABSORPTION SPECTROPHOTOMETRY" also "COOKBOOK FOR ATOMIC ABSORPTION SPECTROPHOTOMETRY".

Methods are given for the determination of strontium in ashed samples of milk, bone, plant materials and shellfish. An extensive study is presented of the methods of controlling strontium analytical interferences.