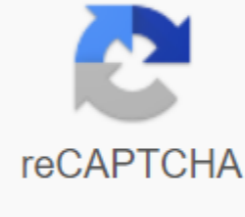# Atomic absorption spectroscopy pdf file

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Flame atom absorption spectroscopy instrument A scientist preparing solutions for spectroscopy for atomic absorption, reflected in the glass window of the AAS flame atomizer cover door Atom absorption spectroscopy (AAS) and atomic emission spectroscopy (AES) is a spectral analytical procedure for quantitative determination of chemical elements using the absorption of optical radiation (light) of free atoms in the gas state. Atomic absorption spectroscopy is based on the absorption of light by free metallic ions. In analytical chemistry, the technique is used to determine the concentration of a particular element (analyte) in a sample to be analyzed. AAS can be used to determine over 70 different elements of the solution, or directly in solid samples via electrothermal evaporation,[edit] and used in pharmacology, biophysics, archaeology and toxicology research. Spectroscopy for nuclear emissions was first used as an analytical technique, and the underlying principles were established in the second half of the 19th century by Robert Wilhelm Bunsen and Gustav Robert Kirchhoff, both professors at the University of Heidelberg in Germany. [1] The modern form of AAS was largely developed in the 1950s by a team of Australian chemists. They were led by Sir Alan Walsh of the Commonwealth Scientific and Industrial Research Organisation (CSIRO), Division of Chemical Physics, in Melbourne, Australia. [2] [3] Atomic absorption spectrometry has many applications in various areas of chemistry, such as clinical analysis of metals in biological fluids and tissues such as whole blood, plasma, urine, saliva, brain tissue, liver, hair, muscle tissue, atomic absorption spectrometry can be used in qualitative and quantitative analysis. Principles The technique makes use of the atomic absorption spectrum a sample to assess the concentration of specific analytes in it. It requires standards of known analytical content to establish the relationship between measured absorbance and analyt concentration and therefore depends on the Beer-Lambert Act. Instrumentation Atom absorption spectrometer block diagram To analyze a sample for its atomic constituents, it must be atomized. The nebulizers most commonly used today are flames and electrothermal (graphite tubes) nebulizers. The atoms should then be irradiated by optical radiation, and the radiation source may be an element-specific line radiation source or a continuum radiation source. The radiation then passes through a monochromator to separate the element-specific radiation from other radiation emitted by the radiation source, which is eventually measured by a detector. Nebulizers The atomizers most commonly used today are (spectroscopic) flames and electrothermal (graphite tubes) nebulizers. Other atomizers, such as radiant discharge, hydride atomization or atomization, can be used for special purposes. Flame atomizers The oldest and most commonly used atomizers in AAS are flames, mainly the air-acetylene flame with a temperature of about 2300 °C and nitrous oxide[3] system (N2O)-acetylene flame with a temperature of about 2700 ° C. The latter flame, in addition, offers a more reducing environment, which is ideally suited for analytes with high affinity to oxygen. A laboratory flame footmeter that uses a propane-driven flame atomizer Liquid or dissolved samples is usually used with flame atomizers. The sample solution is aspired by a pneumatic analytical atomizer, transformed into an aerosol, which is introduced into a spray chamber, where it is mixed with the flame gases and conditioned in a way that only the finest aerosol droplets (< 10 μm) enter the flame. This conditioning process reduces interference, but only about 5% of the aerosolized solution reaches the flame because of it. On top of the spray chamber is a burner head that produces a flame that is sideways long (usually 5-10 cm) and only a few mm deep. The radiation beam passes through this flame on its longest axis, and flame gas flow rates can be adjusted to produce the highest concentration of free atoms. The burner height can also be adjusted, so that the radiation beam passes through the zone with the highest atomic cloud density in the flame, resulting in the highest sensitivity. The processes of a flame include stages of destruction (drying) in which the solvent evaporates and the dry sample nanoparticles remain, evaporation (transmission to the gas phase) in which the solid particles are converted into a gas molecule, atomization in which molecules are dissociated into free atoms, and ionization there (depending on the ionization potential of analytatomes and the energy available in a particular flame) atoms can be partially converted into gas ions. Each of these stages includes the risk of interference in case the degree of phase transfer differs for the analyte in the calibration standard and in the sample. Ionization is generally undesirable, as it reduces the number of atoms available for measurement, that is, sensitivity. In the AAS flame, a steady-state signal is generated during the time period when the sample is aspirated. This technique is usually used for provisions of the mg L-1 range, and can be extended down to a few μg L-1 for some elements. Electrothermal atomizers GFAA method development Graphite tube Elektrothermic AAS (ET AAS) using graphite tube atomizers pioneered by Boris V. L'vov at St. Petersburg Polytechnical Institute, Russia,[4] since the late 1950s, and examined in parallel by Hans Massmann at the Institute of Spectroscopy and Applied Spectroscopy (ISAS) in Dortmund, Germany. [5] Although a wide variety of graphite tubes has been used over the years, the dimensions today are usually 20-25 mm in length and 5-6 mm inner diameter. With this technique, liquid/dissolved solids and gas samples can be analyzed directly. A measured volume (usually 10-50 μL) or a weighted mass (usually around 1 mg) of a solid sample is introduced into the graphite tube and exposed to a temperature program. This usually consists of stages, for example, drying - the solvent evaporates; pyrolysis – the majority of the matrix constituents are removed; atomization – the analysis element is released into the gas phase; cleaning – remove any residues in the graphite tube at a high temperature. [quote required] Graphite tubes are heated via their ohmic resistance using a low-voltage high-current power supply; The temperature in the individual stages can be checked very carefully, and temperature ramps between the individual stages facilitate the separation of sample components. Pipes can be heated transverse or longitudinal, where they previously have the advantage of a more homogeneous temperature distribution over the length. The so-called stabilized temperature platform oven concept (STPF), proposed by Walter Slavin, based on research by Boris L'vov, makes ET AAS essentially free of interference. [quote required] The main components of this concept are atomization of the sample from a graphite platform inserted into the graphite tube (L'vov platform) instead of from the pipe wall to delay the atomization until the gas phase in the atomizer has reached a stable temperature; use of a chemical modifier to stabilize the analyte to a pyrolysis temperature sufficient to remove the majority of the matrix components; and integration of the absorbance over time of the transient absorption signal instead of using peak height absorption for quantification. In ET AAS, a transient signal is generated, the area of which is directly proportional to the mass of analyte (not the concentration) introduced into the graphite tube. This technique has the advantage that any kind of sample, solid, liquid or gas, can be analyzed directly. Its sensitivity is 2-3 orders of magnitude higher than that of the flame AAS, so that provisions in the low μg L-1 range (for a typical sample volume of 20 μL) and the ng g-1 area (for a typical sample mass of 1 mg) can be performed. It shows a very high degree of freedom from interference, so ET AAS can be considered the most robust technique available today for the determination of trace elements in complex matrices. [quote required] Specialized atomization techniques While flame and electrothermal evaporators are the most common atomization techniques, several other atomization methods are used for specialized use. [6] [7] Atomization of glow a glow discharge (GD) acts as a versatile source, as it can simultaneously introduce and atomize the sample. The glow emission occurs in a low-pressure argon gas atmosphere between 1 and 10 torr. In this atmosphere lie a pair of electrodes that use an DC voltage of 250 to 1000 V to break down the argongassen into positively charged ions and electrons. These ions, under the influence of the electric field, are accelerated into the cathode surface containing the specimen, bombarding the sample and causing neutral sample atommuting through the process known as sputtering. The atomic vapor produced by this emission consists of ions, ground state

atoms and fractions of tense atoms. When the tense atoms relax in the ground again, a low-intensity glow is released, giving the technique its name. The requirement for samples of glow discharge atomizers is that they are electrical conductors. Accordingly, nebulizers are most commonly used in the analysis of metals and other conductive samples. However, with proper modifications, it can be used to analyze liquid samples as well as non-seductive materials by mixing them with a conductor (e.g. graphite). Hydride atomization Hydride generation techniques are specialized in solutions of specific elements. The technique provides a means of introducing samples containing arsenic, antimony, selenium, bismuth, and lead into an atomizer in the gas phase. With these elements, hydridatomization improves detection limits by a factor of 10 to 100 compared to alternative methods. Hydride generation occurs by adding a surized aqueous solution of the sample to a 1% aqueous solution of sodium borohydride, all of which are found in a glass vessel. The volatile hydride generated by the reaction that occurs is swept into the atomizer chamber by an inert gas, where it undergoes decomposition. This process forms an atomized form of analyte, which can then be measured by absorption or discharge spectrometry. Cold vapor atomizer The cold steam technique is an atomization method limited to mere determination of mercury, due to the fact that it is the only metallic element that has a large enough vapor pressure at ambient temperature. [quote required] Because of this, it has an important use in determining organic mercury compounds in samples and their distribution in the environment. The method initiates by converting mercury to Hg2+ by oxidation from nitric acid and sulfuric acids, followed by a reduction of Hg2+ with tin(II) chloride. The mercury, is then swept into a long-pass tube by bubbling a stream of inert gas through the reaction mixture. The concentration is determined by measuring the absorbance of this gas at 253.7 nm. Detection limits for this technique are in the area of parts per billion, making it an excellent mercury detection metatomicization method. Two types of burners are used: total consumption and premix burner. Radiation sources We must distinguish between line source AAS (LS AAS) and continuum source AAS (CS AAS). In classic LS AAS, as has been suggested by Alan Walsh,[8] the high spectral resolution required for AAS measurements is provided by the radiation source itself that emits the spectrum of the analyte in the form of lines narrower than the absorption lines. Continuum sources, such as deuterium lamps, are used only for background correction purposes. The advantage of this technique is that only a medium-resolution monochromator is required to measure AAS; However, it is downside that a separate lamp is usually required for each item that needs to be determined. In CS AAS, on the other hand, a single lamp is used, which emits a continuum spectrum across the entire spectral area of interest for all elements. Of course, a high-resolution monochromator for this technique is needed, which will be discussed later. Hollow cathode lamp (HCL) Hollow cathode lamps Cave cathode lamps (HCL) are the most common radiation source in LS AAS. [quote required] Inside the sealed lamp, filled with argon or neon gas at low pressure, is a cylindrical metal catode containing element of interest and an anode. A high voltage is applied over the anode and cathode, resulting in an ionization of the filling gas. The gas ions are accelerated towards the cathode, and by the influence on the cathode, the cathode material that is enthused in the incandescent emission to emit the radiation of the sputtered material, that is, the element of interest. In most cases, single element lamps are used, where the cathode is squeezed out of predominantly connections of the target element. Multi-element lamps are available with combinations of connections of the target elements pressed in the cathode. Multi-element lamps provide slightly less sensitivity than individual element lamps, and the combinations of elements must be carefully selected to avoid spectral disturbances. Most multi-element lamps combine a handful of elements, such as: 2 - 8. Atomic Absorption Spectrometers can contain as few as 1-2 hollow cathode lamp positions or in automated multi-element spectrometers, an 8-12 lamp positions may be usually available. Electrot flow lamps Electrolys discharge lamps (EDL) contain a small amount of analyte as metal or salt in a quartz bulb along with an inert gas, usually argon gas, at low pressure. The bulb is inserted into a coil that generates an electromagnetic radio frequency field, resulting in a low pressureductively switched off the lamp. The emissions from an EDL are higher than from an HCL, and the line width is generally narrower, but EDLs need a separate power supply and may need longer to stabilize. Deuterium lamps Deuterium HCL or even hydrogen HCL and deuterium discharge lamps are in LS AAS for background correction purposes. [9] The radiation intensity emitted by these lamps is significantly reduced by increasing wavelength, so they can only be used in the wavelength range between 190 and about 320 nm. Xenon lamp as a continuous radiation source Continuum sources When a continuum radiation source is used for AAS, it is necessary to use a high-resolution monochromator, which will be discussed later. In addition, it is necessary that the lamp emits radiation of intensity at least one magnitude over a typical HCL over the entire wavelength ranging from 190 nm to 900 nm. A special high pressure xenon short arc lamp, operating in a hot-spot mode is designed to meet these requirements. Spectrometer As already pointed out above, there is a difference between medium-resolution spectrometers used for LS AAS and high-resolution spectrometers designed for CS AAS. The spectrometer includes spectral sorting unit (monochromator) and detector. Spectrometers for LS AAS are the high resolution required for the measurement of atomic absorption provided by the narrow line emission of the radiation source, and the monochromator must only solve the analytical line from other radiation emitted by the lamp. [quote required] This can usually be achieved with a band pass between 0.2 and 2 nm, that is, a medium resolution monochromator. Another function of making LS AAS element specific is modulation of primary radiation and the use of a selective amplifier that is tuned to the same modulation frequency, as already postulated by Alan Walsh. In this way, any (unmodulated) radiation emitted for example by the atomizer can be excluded, which is important for LS AAS. Simple monochromeators of Littrow or (better) Czerny-Turner design are usually used for LS AAS. Photomultiplier pipes are the most commonly used detectors in LS AAS, although solid state detectors may be preferred due to their better signal-to-noise ratio. Spectrometers for CS AAS When a continuum radiation source is used for AAS measurement, it is indispensable to work with a high-resolution monochromator. The solution must be equal to or better than the half-width of an atomic absorption line (approx. 14:00) to avoid loss of sensitivity and linearity of the calibration graph. The research with high-resolution (HR) CS AAS was pioneered by the groups O'Haver and Harnly in the UNITED States, which also developed (until now) only simultaneously multi-element spectrometer for this technique. However, the breakthrough came when the becker-ross group in Berlin, Germany, built a spectrometer entirely designed for HR-CS AAS. The first commercial equipment for HR-CS AAS was introduced by Analytik Jena (Jena, Germany) at the beginning of the 21st century. and Florek. These spectrometers use a compact dual monochromator with a prism pre-monochromator and a echelle grating monochromator for high resolution. A linear charge-linked device (CCD) array with 200 pixels is used as a detector. The second monochromator does not have an output slot; therefore, the spectral environment on both sides of the analytical line becomes visible at high resolution. Typically, only 3-5 pixels are used to measure atomic absorption, the other pixels are available for correction purposes. One of these corrections is that for lamp fibrillation noise, which is independent of wavelength, resulting in measurements with very low noise levels; other corrections are those for background absorption, which will be discussed later. Background absorption and background correction The relatively small number of atomic absorption lines (compared to nuclear emission lines) and their narrow width (a few p.m.) makes spectral overlap rare; There are only a few examples that are known that an absorption line from one element will overlap with another. [quote required] Molecular absorption, on the other hand, is much wider, so it is more likely that some molecular absorption bands will overlap with an atomic line. This type of absorption can be caused by uncirculated molecules of concomitant elements of the sample or by flame gases. We need to distinguish between the spectra of di-atomic molecules, which show a pronounced fine structure, and those of larger (usually tri-atom) molecules that do not show such a fine structure. Another source of background absorption, especially in ET AAS, is the proliferation of primary radiation by particles generated in the atomization stage, when the matrix could not be adequately removed in the pyrolysis stage. All these phenomena, molecular absorption and radiation absorption, can result in artificially high absorption and an incorrect high (erroneous) calculation for the concentration or mass of the analyte in the sample. There are several techniques available to correct for background absorption, and they are significantly different for LS AAS and HR-CS AAS. Background correction techniques in LS AAS I LS, AAS background absorption can only be corrected using instrumental techniques, and all are based on two sequential measurements:[10] firstly, total absorption (atom plus background), secondly, background absorption only. The difference between the two measurements provides net nuclear absorption. Because of this, and due to the use of multiple devices in the spectrometer, the signal-to-noise ratio of background-corrected signals is always significantly inferior compared to uncorrected signals. It should also be pointed out that in LS AAS there is no way to correct for (the rare case of) a direct overlap of two atomic lines. Essentially, there are three techniques used for background in LS AAS: Deuterium background correction This is the oldest and still most commonly used technique, especially for flame AAS. In this case, a separate source (a deuterium lamp) with a wide discharge is used to measure background absorption across the entire width of the output slot on the spectrometer. The use of a separate lamp makes this technique the least accurate, as it can not correct for structured background. It also cannot be used at wavelengths above about 320 nm, as the emission intensity of the deuterium lamp becomes very weak. The use of deuterium HCL is preferable compared to an arc lamp due to better adaptation of the image of the former lamp with analyte HCL. Smith-Hieftje background correction This technique (named after the inventors) is based on line expansion and self-reversal of emission lines from HCL when high current is used. Total absorption is measured with normal lamp current, it will persist with a narrow emission line, and background absorption after applying a high-current pulse with the profile of the self-reined line, which has little discharge at the original wavelength but strong discharge on both sides of the analytical line. The advantage of this technique is that only a radiation source is used; Among the disadvantages is that the high power pulses reduce the life of the lamp, and that the technique can only be used for relatively volatile elements, as only they show sufficient self-reversal to avoid dramatic loss of sensitivity. Another problem is that the background is not measured by the same wavelength as total absorption, making the technique unsuitable for correction of structured background. Zeeman effect background correction Main article: Zeeman effect An alternating magnetic field is applied by the nebulizer (graphite furnace) to divide the absorption line into three components, the π component, which remains in the same position as the original absorption line, and two σ components, which are moved to higher and lower wavelengths respectively. [quote required] Total absorption is measured without magnetic field and background absorption with the magnetic field on. The π component must be removed in this case, for example, using a polarizer, and the σ components do not overlap with the lamp's emission profile, so only the background absorption is measured. The advantages of this technique are that total and background absorption is measured with the same emission profile of the same lamp, so that any kind of background, including background with fine structure, can be corrected accurately, unless the molecule responsible for the background is also affected by the magnetic field and uses a helicopter as a polarizer reduces signal-to-noise ratio. While the disadvantages are the increased complexity of the spectrometer and power supply needed to run the powerful magnet to divide the absorption line into components; Therefore, unlike LS AAS, additional background correction components are not required. Background correction using correction pixels It has already been mentioned that in HR-CS AAS lamp flickering noise is eliminated by using correction pixels. In fact, any increase or decrease in radiation intensity observed to the same extent on all pixels selected for correction is eliminated by the correction algorithm. [quote required] This obviously also includes a reduction of the measured intensity due to radiation-dispersing or molecular absorption, which is corrected in the same way. As the measurement of total and background absorption, and correction for the latter, is strictly simultaneously (unlike LS AAS), even the fastest changes of background absorption, as they can be observed in ET AAS, do not cause any problem. In addition, as the same algorithm is used for background correction and elimination of lamp noise, background-corrected signals show a much better signal-to-noise ratio compared to the uncorrected signals, which are also unlike LS AAS. Background correction using a minimum square algorithm The technique above can obviously not correct for a background with fine structure, as the absorbance in this case will be different on each of the correction pixels. In this case, HR-CS AAS offers the ability to measure the correction spectrum of the molecule(s) that is (is) responsible for the background and store them in the computer. These spectra are then multiplied by a factor to match the intensity of the sample spectrum and are drawn pixel by pixel and spectrum by spectrum from the sample spectrum using a minimum square algorithm. This may sound complicated, but first of all the number of di-atomic molecules that can exist at the temperatures of the atomizers used in AAS is relatively small, and secondly, the correction of the computer is carried out within a few seconds. In fact, the same algorithm can also be used to correct for direct line overlap of two atomic absorption lines, making HR-CS AAS the only AAS technique that can correct for this type of spectral interference. 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