

solid AA[®]

Direct. Fast. Easy.



Direct Solid AAS



Intelligent innovation for fast and precise analysis

Conventional methods of elemental analysis can only process liquid samples. Solids, therefore, are subject to a time-consuming sample preparation process. Analytik Jena's solid AA[®] technology offers an interesting alternative: The direct solid AAS.

Solid samples in powder form, granulates and fibers, but also paste-like materials such as cream, sludge or viscous oils can usually be analyzed directly in the graphite furnace without any need for sample preparation. Heterogeneous samples are merely reduced to a smaller size and homogenized prior to the analysis.

The typical sample quantity is usually between 100 µg and 10 mg. The actual sample weight depends on the sample matrix and the concentrations to be determined. The sample is weighed onto a sample carrier and brought to the graphite furnace. After the analysis is complete, the sample carrier may be reused.

In the direct solids analysis, the decomposition of the sample matrix by means of an acid digestion is replaced by the temperature program of the graphite furnace. Combined with a powerful background correction, the interference-free and precise determination of almost all elements in numerous materials is possible. The graphite furnace technique is subject to few interferences caused by the sample matrix. Therefore, even liquid calibration standards may be used in most cases. A calibration with solid matrix reference materials, which are expensive and often not available in the correct composition, is usually not necessary.

The Analytik Jena graphite furnace systems can be converted from liquid to solids analysis in just a few minutes – the unlimited use of both techniques is guaranteed. All functions are integrated in the operating software. Additional modules are not required. Easy, software-guided routines allow a simpler adjustment of the autosamplers and guarantee a reliable sample supply.

solid AA® – Fast, precise, easy

If solid samples are analyzed directly, the amount of work and time required for the sample preparation is reduced to a minimum. Apart from saving time, there are several other main advantages:

Analysis of the original sample

With solid AA® your samples can be analyzed without additional reagents. Consequently, the risk of contamination of these samples caused by blank values is reduced significantly. Dilution errors or analyte loss during sample preparation and storage are impossible.

Wide measurement range

The dilution, which invariably accompanies a digestion, is not required in the solid AA® technique. As a result, the relative measurement sensitivity can easily be increased by at least a factor of 10 compared to the solution analysis. Higher concentrations up to the percent range can also be determined precisely.

Analysis of small sample quantities

In the clinical and biological field of application, the available sample quantities are often very small. solid AA® is a micro-method. Therefore, a sample weight of approximately 50 µg is sufficient for element determination. Consequently, precise measurements can be carried out for such applications as well. This is also an advantage for the examination of element distribution in a sample, since the analytical samples can specifically be taken with a higher spacial resolution and inhomogeneities can be detected.

Avoiding harmful reagents

Since the analysis via solid AA® usually occurs without reagent addition, not only costs are reduced. The use of digestion reagents that may pose a health risk or a risk to the environment, as is often necessary for the dissolution of refractory samples, is not required. Chemical waste is thus kept to a minimum.

Simple handling

Paste-like, sticky materials that are difficult to dose are simply placed on the sample carrier and weighed in directly. Weighing errors due to particles left on the weighing boat can thus be avoided. Thanks to the high degree of automation of the solid AA® errors due to sample handling become practically impossible.

Fast results

With solid AA® many samples can be analyzed directly without any additional preparation. As a result, analysis results are available after only a few minutes. The monitoring of incoming goods and the production process is carried out promptly; any waiting time that occurs in conventional wet-chemical sample preparation can be avoided.

Solid analysis technology

Direct. Fast. Easy.

All graphite furnace systems by Analytik Jena can be upgraded with the solid AA[®] technology. Three different sample supply systems are available:



SSA 6 – manual solid sampler

The manual solid sampler is suitable for the occasional solid analysis. The sample is weighed on a separate microbalance and is brought into the graphite furnace by means of the sampler. After the analysis is complete, the sample carrier is immediately ready for use for the next sample.



SSA 600 – fully automated solid sampler

An autosampler for the fully automated analysis of up to 84 samples. Simply dose the samples onto the sample carriers. The autosampler automatically weighs in the sample using the integrated microbalance with a precision of 1 µg and transports it into the graphite furnace and back to the sample tray. The process does not require supervision. Dosing and weighing of the sample can be controlled comfortably via the operating keys on the autosampler. The sample mass is imported directly into the software. Additional software routines, such as the automatic clean-out and taring of all sample carriers, simplify the operation in the daily routine.



SSA 600L – fully automated solid sampler with liquid dosing unit

Equipped with an additional liquid dosing unit, the SSA 600L offers the highest possible degree of automation for routine and research. The autosampler does not just weigh and transport the samples automatically, it also carries out the calibration with liquid standards and the addition of a modifier, reducing the user's effort to a minimum.



Technical Data

Sample carrier

Material	Graphite, pyrolytic-coated
Weight	approx. 90 mg
Sample capacity (solid)	approx. 50 mg (depending on sample characteristics)
Sample capacity (liquid)	max. 30 μ L
Size (W x H x D)	14.1 x 4.6 x 2.0 mm

SSA 600

Sample capacity	42 samples (84 samples with twin sample tray)
Speed levels	3
Adjustment	Software-guide, 0.1 mm steps
Microbalance	1 μ g

SSA 600L (in addition to the SSA 600)

Dosing module	500 μ L syringe pump, mounted vertically
Dosing volume	1 – 30 μ L, adjustable in 1 μ L steps
Positions	6 x 1.5 mL, 2 x 5 mL, wash cup

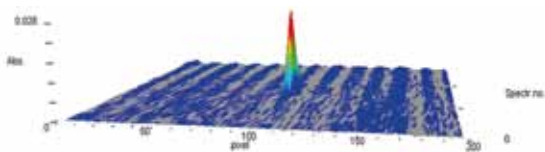
solid AA[®] – For a broad application spectrum

Food analysis

The determination of heavy metals in fish, meat and sausage products or in vegetables, grain and milk powder are typical application fields of atomic spectrometry. The solid AA[®] technology is an interesting alternative to the conventional analysis of digested samples, for the determination of, for example, cadmium, lead and arsenic in rice. After the representative sample has been ground and homogenized, it is ready for fast and direct analysis. This process allows a high sample throughput with less effort and therefore a consistent food control. In this context, raw material and production monitoring in the food processing industry can also be carried out promptly with solid AA[®].

♥ Temperature program cadmium in rice

Step	Temp. [°C]	Heating rate [°C/s]	Time [s]	Gas
Drying	150	10	15	Argon
Drying	200	10	15	Argon
Ashing	400	25	10	Argon-O ₂
Ashing	600	25	15	Argon-O ₂
Purge	600	0	5	Argon
Pyrolysis	800	100	15	Argon
Atomize	2300	1400	3	Stop
Clean-out	2500	500	4	Argon



▲ Time-resolved absorption spectrum: Cadmium in a rice sample, recorded with the HR-CS AAS contraAA[®] 700



Application examples

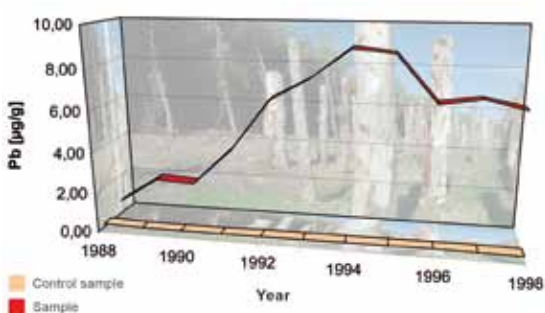
Margarine, fats, oils	Fe, Ni
Grain, vegetables, meat	Cd, Pb, Cu, Ni, Zn, Se, Na, Fe
Ice cream, milk products	Ag
Honey, jam	Cd, Pb, Fe, Ni

Environmental analysis

Whether in soils or plants – heavy metal determination is an important part of environment-related examinations as well. With solid AA[®], users can obtain precise results fast and easy – even if no fully equipped laboratory is available. Airborne dust and particle samples, for example, can be placed on the sample carrier and analyzed directly from the filter.

As part of a study, the heavy metal distribution in trees near a landfill was determined. Samples were taken directly from individual annual rings and analyzed with regard to different characteristic elements. In the process, the measurement values could be correlated with further relevant factors, such as the yearly rainfall and the delivery of specific wastes.

♥ Concentration of lead in wood over a time period of 10 years
orange: control sample, red: sample



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Application examples

Plants, tissue, bones, fur	Cd, Pb, As, Se, Ni, Cr, Co, Mn
Soil, sediment, ash, waste, dust	Cd, Pb, Cu, Fe, Mn, Ni, Cr, Sn

solid AA[®] – For a broad application spectrum

Analysis in cosmetics

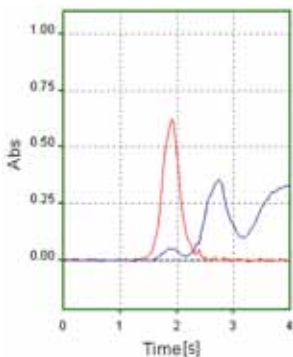
Cosmetic products must be tested for heavy metals on a regular basis. The formula component content must be verified as well.

With solid AA[®] such analyses are carried out promptly during the production process. Since the measurement results are available in a very short time, the quality of the raw materials can already be assured during the delivery.

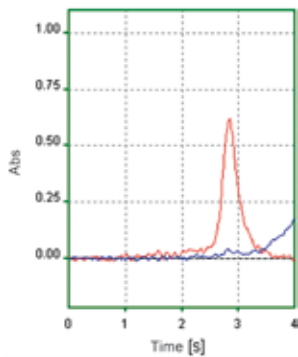
The determination of lead, cadmium, arsenic, nickel and other elements is part of the daily routine in manufacturing cosmetics and its raw materials, such as, for example, pigments. The example of mica and metal pigments, for instance, shows how efficient the solid AA[®] technology is in this field. A wide variety of samples are taken from production and analyzed directly. Since they are already in powder form and homogenized during the production process, no further preparation is required. Finished products such as makeup and lipstick can also be examined fast and reliably with the new screening method. Only samples that exceed a limit value must be digested and characterized more precisely with the help of solution analysis.



♥ 5.7 mg/kg Pb in mica pigment



♥ 3.1 mg/kg Sb in metal pigment



Pharmaceutical analysis

Raw materials and products of the pharmaceutical industry can also be examined fast and reliably. Next to the prompt availability of the results, the detection sensitivity, which is considerably improved compared to the fluid analysis, is another important criteria here. Analyses in the lower $\mu\text{g}/\text{kg}$ range can easily be carried out and the high product quality can be guaranteed.

solid AA[®] is particularly well suited for a homogeneity test as well. The small sample quantity allows for the detection of the uneven distribution of one or more characteristic elements with only very few repeat measurements. The result is an optimized production process and an increased production quality.

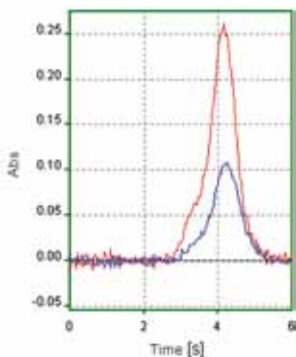


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Application examples

Pigments	Cd, Pb, As, Se, Ni, Cr, Hg, Sb
Fats, waxes	Fe, Ni, Cd, Cr, Pb, Cu
Herbs	Pb, Cd, As, Fe, Cr
Pharmaceutic raw materials	Pb, Cd, As, Fe, Cr, Cu, Ni
Insulin	Zn

♥ 1.8 mg/kg Cd in metal pigment



red = specific signal

blue = Background signal

solid AA[®] – For a broad application spectrum

Analysis in metallurgy

Modern high performance materials, such as ceramics, glasses and specific alloys are subject to the highest purity requirements. Even contaminations in the lower mg/kg range can significantly limit the material's performance for the desired purpose of use. A highly sensitive and precise analysis method is required – solid AA[®]!

The analysis of specific trace elements in alloys that are used to manufacture aircraft turbines is decisive in ensuring the reliability of the components. Due to the extreme centrifugal forces that act on turbine blades, specific elements build up in different zones where they can lead to material fatigue. This analysis is a difficult challenge for many methods.



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While preparing the sample by conventional methods, make sure that it is not contaminated by reagent blank values. Element loss due to the formation of volatile species or due to adsorption must be excluded. In the case of a wet-chemical digestion, moreover, the sample is diluted to a degree that often makes it quite difficult to attain the required determination limit.

With solid AA[®], the unaltered original sample is analyzed to avoid any risk of contamination. The measurement sensitivity is increased, since a dilution of the samples is not required. The resulting advantages are valuable for the material analysis.

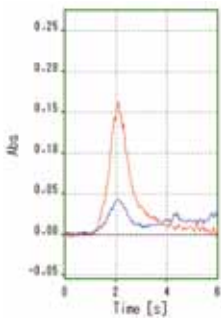
Material analysis

Special highly stable ceramics, such as carbide and oxide, are often developed with regard to mechanical and chemical stability. The degree of difficulty to mineralize such materials for the conventional liquid analysis is accordingly high. Often, lengthy digestion methods with complex acid mixtures are required here. In addition, the sample weight for such digestions is usually limited. As a result, the detection sensitivity is often insufficient for the trace range.

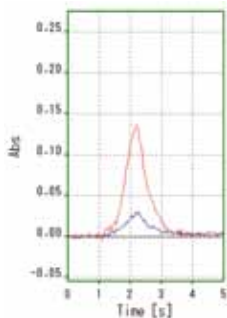
This can be avoided with the solid AA[®] technology, since the sample merely has to be reduced to a smaller size. The determination of the trace elements then usually occurs without any problems and without much effort. Without diluting the sample, even the smallest concentrations can be determined reliably.



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▲ 57 mg/kg Cr in SiC
(secondary wavelength)
Red = analyte signal
Blue = background signal



▲ 1.5 mg/kg Mn in SiC
Red = analyte signal
Blue = background signal

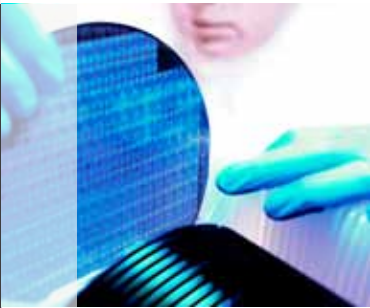
Application examples

Plastics, rubber	Cd, Pb, Cr, Cu, Fe, Mn
Textiles	Ag, P, Sb, Cd, Pb
Reference material (homogeneity)	Cd, Pb, Fe, Zn
Refractory glasses, ceramics	Pb, Mg, Na, Cr, Mn, Fe
Alloys	Se, Sb, Bi, Cd, Pb
Ion exchangers	Pd, Au, Ag, Ni, Co, Zn

solid AA[®] – For a broad application spectrum

Analysis in electronics

In the semiconductor industry, high-purity materials are used, which are specifically spiked with particular elements with the goal to modify the material properties. Even the slightest contamination is sufficient to render the material useless. This is why the detection of the smallest traces of elements is part of the daily routine in the semiconductor production – a task that requires the highest level of detection sensitivity and precision.



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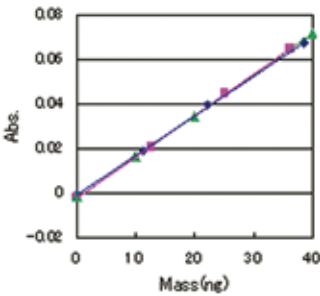
With solid AA[®] a relatively large sample quantity can be analyzed in undiluted form. Therefore, the solid AAS is the routine analysis method with the best detection results for many elements. Compared to alternative methods with similar sensitivity, it offers significantly easier handling and exhibits less interferences and sources of error.

♥ Calibration for cadmium in plastic:

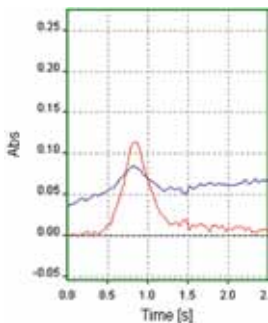
Blue: similar sample weight of different reference materials

Pink: different sample weights of the same reference material

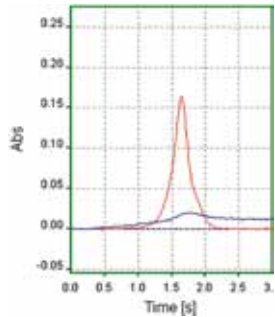
Green: aqueous calibration standards



♥ Signal profile Cr, 17.7 mg/kg



♥ Signal profile Cd, 21.7 mg/kg



RoHS

The European legislation has banned the content of particular hazardous substances in electronic devices in order to conform to environmental standards and to simplify recycling (Restrictions of certain Hazardous Substances – RoHS). Aside



from different organic substances, the ban includes cadmium, lead, chromium-(VI) and mercury. Conventional solder has so far contained a considerable amount of lead. Since the RoHS Directive was introduced, these types of solders are prohibited and, as a result, the corresponding processes were converted to include lead-free solders. Different polymers that are used for cable insulation and housing parts may also include these elements in the form of pigments or contamination due to recycling materials. To prove the conformity of the products with the European standard, raw material and products must be tested for these substances.

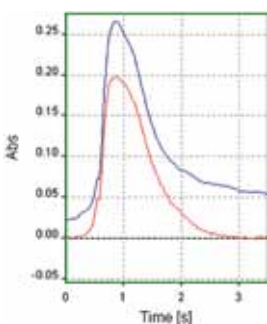
Since plastics are homogeneous due to production-related reasons and mostly occur as granulates, solid AA[®] is the method of choice here.

The analysis is simplified further as no matrix effects occur and liquid calibration standards can thus be used – in contrast to other solid methods, which usually require solid calibration standards and certified reference material.

Application examples

Plastics	Cd, Pb, Cr, Cu, Fe, Mn, Sn, Si
Semiconductors, ceramics	Pb, Mg, Na, Cr, Mn, Fe, K
Alloys, brazing solders	Pb

♥ Signal profile Pb, 13.8 mg/kg



Sample weight 1.5 – 2 mg plastic each, sensitivity reduced by selection of secondary wavelengths
 Red = analyte signal
 Blue = background signal

Analysis in medicine

In the context of clinical tests, hair or finger nails but also bones and tissue are occasionally tested for heavy metals. Since the quantity of such samples is usually quite small, the concentration after a wet-chemical digestion is often too low to obtain reliable results.

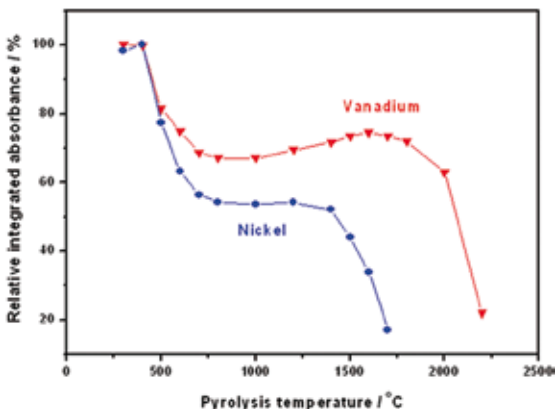
With solid AA[®] these types of examinations can be greatly simplified. A small sample quantity, usually 0.1 - 0.5 mg, can be analyzed directly. The prior digestion and the associated dilution become unnecessary, which means that the detection sensitivity with regard to the original sample is increased significantly.

Petrochemical analysis

Crude oil and the fractions contained therein are often difficult to mineralize, since they contain both very reactive and very stable components. The sample quantity that can be used is small, because a high pressure is generated during the digestion. Accordingly, the determination of element traces often requires a high analytical effort. Good examples are the elements nickel and vanadium, that act as a catalyst poison and increase the corrosion of unit parts and usually need to be determined in the lower mg/kg range.

With solid AA[®] crude oil and similar samples can be analyzed directly without any additional preparation. Additionally, the direct solid analysis has the advantage that volatile organic compounds, such as nickel and vanadylporphyrin, remain in their original form. Therefore, selectively volatile and thermally stable element compounds can be determined with the solid AAS, a process that cannot be carried out after a mineralization.

♥ *Signal intensity for nickel and vanadium in crude oil at different pyrolysis temperatures. Loss of volatile species from approx. 500°C*



Application examples

Tissue, liver, kidney	Cd, Pb
Hair, finger nails	Hg, As, Se, Pb, Cd, Si
Blood (on filter paper)	Pb
Crude oil	Ni, V, Cd, Pb, Cr
Catalysts	Fe, Cu, Pd, Pt, Rh, Pb, Co, Na

solid AA[®] – Fully integrated into the software

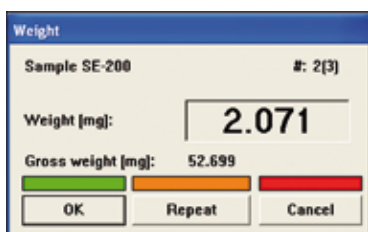
All functions of the solid AA[®] technology are pre-integrated into the basic software. The purchase and installation of additional software modules is not required. The fully automated analysis run is controlled and managed through an intuitive software concept that is adapted to the specific of solid sampling. First, the tare weights are recorded in an automatic routine. Then the sampler brings the sample carrier to the dosing position. Now the user can dose the calibration standards, the samples and, if applicable, the modifier. Dosing and weighing can be confirmed by mouse click or via the function keys on the autosampler. All sample weights are automatically transferred to the sample table and used for calculating the concentration.

Simple, software-guided routines are available for adjusting the autosampler. The switch between liquid and solid analysis occurs within a few minutes. Both methods are available without restrictions.

The integrated microbalance can be checked and calibrated with the help of an integrated calibration weight. An external certification with certified balance weights can be carried out by the user or by service engineers from Analytik Jena AG.

solid AA[®] – Direct solid AAS

The easy way to reliable analysis results in research and routine.



▲ *Display of the sample weight*

▼ *Sample table with sample weights*

Pos	#	Type	Name	#	Weight mg	Tare mg	Des.	Std. Modif.	Rely.
1		Cal-Zero	Cal-Zero	1					
2		Cal-Std1	Cal-Std1	1	1.811	50.860	*	*	
3		Cal-Std2	Cal-Std2	1	2.860	50.864	*	*	
4		Cal-Std3	Cal-Std3	1	3.863	50.862	*	*	
5		Cal-Std4	Cal-Std4	1	4.884	49.350	*	*	
6		Cal-Std5	Cal-Std5	1	5.888	49.187	*	*	
7		Sample	SE-200	1	2.848	50.450	*	*	
8				2	2.871	50.628	*	*	
9				3	2.812	50.396	*	*	
10		Sample	Stc-254	1	2.880	49.120	*	*	
11				2	2.894	49.274	*	*	
12				3	2.847	49.386	*	*	
13		Sample	SE-297	1	2.877	49.954	*	*	
14				2	2.892	50.820	*	*	
15				3	2.887	49.767	*	*	

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Design and scope of supply as well as technical specifications are subject to modification!