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ICP Test Sample Technology Reserve

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Abstract

The full name of ICP is the INDUCTIVELY COUPLED PLASMA, that is, the inductive coupling plasma technology, which is a very effective analysis method for measuring marks (PPM, PPB level). Under normal circumstances, it can be used with MS, AES, etc., which can realize various uses such as analysis of the amount of inorganic element marks, isotopes, unit elements, multi -element analysis, and multi -morphological analysis in organic matter.

Keywords

Trace Element Analysis; Dissolving Samples; Dry Gray.

1. Introduction

Because trace element analysis is very important in research, for example, during the preparation of photoresia, the content of some metal elements is required to be strictly controlled. Otherwise, the resistance and capacitance may increase during use, which will make the photoresist fail to fail; For another example, the content of precious metal elements in the electrical catalytic has a great impact on its performance. It is often used as the evaluation criteria. At this time, the quantity of the proportion of precious metal loads is very important for the quality of performance. Some elements in the load -type catalyst are often quantitatively encountered. So we can often see its figure in many literatures.

Taking the quantity of the load catalyst element as an example, in the ICP test, there are usually several steps. First, the catalyst sample is fully dissolved, and the second is to dilute the sample as much as possible to the appropriate level to reduce the impact of interference ions.

Solubbing sample preparation is a very important link. Because the metal ions in the sample need to be fully dissolved and ensure that there is no solid existence, the metal ion content of the final test can be accurate.

To this end, the principle of ICP testing is summarized here and some attention points for the preparation of samples.

2. ICP Spectrum Instrument Working Principle:

The generated plasma must have the next four conditions: high frequency, high -pressure, ravioli, and ion source. First of all, the power supply provides 220V and 50Hz electricity to the ICP spectrum through the voltage regulator. Essence For example, 27.14MHz and 40MHz are generated through RF Source (high -frequency source), entering 0 to 3W power into Driver The power magnification becomes 0 to 2000W provided to the working coil; in addition, it provides

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a high -voltage of about 3800VDC through the transformer and silicon control filtering stream, and adds to high -power tubes; high -voltage and high -frequency pass through the working coil through high -power tube. In this way, high -frequency and high power are added to the three -layer concentric quartz torch. When the sample outside the sample is peristalized through the peristaltic pump, it enters the atomization effect of the atomizer, forming an air -solution and the carrier into the central tube of the inner layer of the quartz torch. High -frequency, high -voltage current couples the energy into the ionizing of the inner layer of the torch through the working coil. When there is a high -frequency current on the coil, the axial direction of the coil produces a strong oscillating ring magnetic field. The atomic pupa in the torch is not conductive, and it will not form discharge. When the high -frequency spark discharge of the fireware is in the torch pipe to leave a small amount of pyradic electricity, once the conductive particles appear in the torch tube, due to the effect of the magnetic field, the movement direction oscillates with the frequency of the magnetic field, and forms the same shaft as the torch pipeline. The ring current. Atoms, ions, and electrons collide with each other in strong oscillations to produce more electronics and ions. Finally, the bright white Ar-ICP discharge, its shape is like a drop of water droplets. The circular vortex that can be formed inside the highly ionized ICP can look at the secondary coil of the transformer, while the water -cooled working coil is equivalent to the primary coil of the transformer. Accelerated electronics and ions are constantly changing its movement direction, resulting in the heating effect of coke ear and an ionization effect. This gas forms a stable "electric flame" light source in the torch tube of the quartz in a short time. The light source enters a two -dimensional spectrum into the slit, reflector, prism, mid -step light grids, and quasi -borders through the lighting tube. The spectrometer measured the element concentration by measuring the number of light quantity drops on the pixel. The light quantity signal is converted into a digital signal through the circuit to print the result through a computer display and printer.

3. Precautions and Skills of Dissolving Samples

(1). The dissolved sample is to dissolve. In principle, as long as the solid sample can be fully dissolved, any solvent can be used.

Common solubility includes acid, alkali, and even some organic solvents. For soluble inorganic compounds, water can be dissolved directly; for most inorganic compounds, such as metal, alloys, and even ore samples, it can be dissolved in acids. Common acids such as hydrochloric acid, nitrate, golchen, sulfate, phosphoric acid, hydrogen phosphate, hydrogen hydrogen Flobic acid and various hybrid acids.

(2). For example, hydrochloric acid can dissolve the metal that is more negative than hydrogen, iron, magnesium, calcium, cricket, aluminum, zinc, tin, rare earth, etc. with metal, iron, magnesium, magnesium, calcium, crickets, crickets, aluminum, aluminum, aluminum, aluminum, crickets, zinc, tin, rare earth; carbonate; carbonate; carbonate; Class material; metal vulcanization ore.

(3). Nitric acid has strong oxidation. In addition to the above metals, some oxidation and restore potential can also be dissolved with nitric acid, such as silver, copper, lead, cobalt, nickel, mercury, manganese, etc.

(4). Hydrofluoric acid is a complexing agent of many elements. Metals that cannot be dissolved in hydrochloric acid and nitrate can be dissolved with hydrochloric acid.

(5). For some precious metal samples, such as gold, silver, platinum, palladium (chemistry), etc., can be dissolved in king water, that is, a mixture of three concentrated hydrochloric acid and a concentrated nitric acid.

(6). In optical catalysts, the most commonly used catalysts are titanium dioxide catalysts. There are often methods such as titanium dioxide loads or other oxides to form heterogeneous knots.

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If you want to test this type of combination, such as titanium dioxide with precious metal loads, the mixing of hydrofluoric acid and Wang Shui seems to be a very good solution. In my previous attempts, other acid mixing can not be fully dissolved in this type of sample well, and you can learn from it. However, it is necessary to pay attention to the use of hydrofluoric acid to dilute as much as possible to avoid damage to ICP glass instruments. (But the impact is minimal, because during the heating process, the hydrofluoric acid goes home and basically volatilizes).

(7). As a kind of thinking, for samples such as aluminum, tungsten, the dissolution speed in the acid is relatively slow, and you can also try to dissolve it with alkali.

(8). During the dissolution, crucible is generally used as a container because it can withstand most acid. However, when hydrofluoric acid is used, it is not possible to use the material of polytrafluoroethylene as a container.

(9). Use oil bath appropriately to speed up the solubility steps.

(10). Because the ICP test is very sensitive, the test is limited to PPM or even PPB levels, so pay attention to prevent other metal interference or pollution. It is best to clean it in a pickled or other way before using the container.

(11). In addition, you can remove the interference elements by burning and other methods. For example, some materials for carbon load can be burned to make carbon decomposition, leaving only metal elements. After investigation, there are similar methods in organic sample processing, called dry gray. The organic matter is about to burn, the metal is transformed into inorganic salts, and then it is dissolved with acid.

4. ICP is a Quantitative Test of Sample Element Content. ICPOES Can Measure PPM Level, and ICPMS Can Measure PPB Level.

(1). Containing AG sample special session

Because AG ions will produce AGCL precipitation when encountering CL ions! When focusing on AG alone, the sample should be solved with pure nitric acid as much as possible, and any HCL and other CL ions cannot be introduced. If you contain ingredients that cannot be solved by silicate, you can add a small amount of hydrofluoric acid. If the sample is nitric acid and hydrogen When fluoric acid cannot be completely eliminated, when HCL must be used, you can consider an AGCL precipitation generated by ammonia water treatment.

When the sample is a combination of AG and other elements, it is necessary to pay attention to -Special element combinations, for example, at the same time, the solution of AG and AUAG should not be introduced as much as possible, but the elimination of AU must use Wang Shui (a component of Wang Shui is a component of Wang Shu Hydrochloric acid) So the question comes, how to deal with pre -processing, this must be considered in front of the two elements separately, and when testing AG (do not consider AU) test AU (do not consider AG) -Saldl chloride sedimentation is filtered out and filtered out Essence

(2) Smoofin Class

It contains Si, ZR, HF, TA, NB, Ti, Al and other compounds, and hydrofluoric acid is generally needed. However, when measuring AU, LA, Y, etc., fluorine and it will produce compounds insoluble in acid and water, knock on the blackboard! Try to consider the amount of excessive boric acid and high temperature heating samples after the amount of the sample is completely eliminated as much as possible, so that the fluoride ions that are excessive fluoride are drove at the same time. Move left, and finally achieved completely dissolved.

(3) why isn't SI measure?

When the HF and SI react, the SIF4 is easily overflowing in the elimination system and the loss of the SI element. There is a corrosive effect, and the test solution is introduced additional SI

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elements! To sum up, the previous treatment may cause the SI loss instrument test may introduce additional Si element test SI is risky and cherished.

(4). Some materials TIPS

When the main component of the sample is the metal of Fe or Al, the Wang Shui dispels it because! The oxidation of nitric acid will form a dense oxidation layer on the surface, which will prevent the suitable concentration of the ICP and the pre -processing of the further reflection of the acid and the sample.

When it comes to quantitative analysis of the content of certain elements in the sample, everyone often thinks of XPS, EDS, and XRF. These are indeed possible. These methods are often semi -quantitative results for reference only if you want to accurately accurately. ICP can basically meet your needs, whether it is PPM or PPB content level, but even if it is accurate, we will still encounter a classmate why the results tested are very different from my expectations? No test means are more or less limited to ICP, which is no exception.

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