

HIGH PURITY PENTAALKOXYTANTALUM,
METHOD FOR MANUFACTURING THE SAME,
PARALLEL SEPARATING ANALYZER AND ANALYSIS METHOD

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BACKGROUND OF THE INVENTION

1. Field of the Invention

The present invention relates to a high purity pentaalkoxytantalum and a method for manufacturing the same. The invention also relates to a separating analyzer for rapid and efficient analysis of impurity elements in materials, a continuous flow analysis method, high purity tantalum or high purity tantalum compounds and a ferroelectric thin-film formed using them.

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2. Description of Related Art

Pentaalkoxytantalums are semiconductor materials and, particularly, pentaethoxytantalum is widely used as a film formation starting material in the manufacture of semiconductors. With respect to the method for manufacturing it, for example, (1) Japanese Unexamined Patent Publication No. 6-192148 describes a method of treating a chlorine element-containing tantalum alkoxide with an alkoxide of an alkali metal or an alkaline earth metal to reduce the amount of the chlorine element to 10 ppm or less. Also, (2) Japanese Unexamined Patent Publication No. 10-139788 describes a method of dissolving a tantalum alkoxide containing Fe, Ca, Sr, Na and U, hydrolyzing from 1 to 20% of the alkoxide while stirring to produce a solid reaction product, and distillation-separating the reaction product from the unreacted alkoxide to recover the objective alkoxide.

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Recently, the material has been required to have a higher purity so as to improve the yield of semiconductors. In particular, the development of a high purity pentaalkoxytantalum with reduced amounts of various metals, as impurities, has been keenly demanded. According to the method (1), the chlorine element is

reduced to 10 ppm or less, however, the method for removing metal impurities is not described. The method (2) has a problem in that the numerical values of Al, Cd, Cr, Cu, K, Mg, Mn, Ni, Pb, Zn and Th except for Fe, Ca, Sr, Na and U are not described and since metals such as Fe are removed by hydrolyzing the tantalum alkoxide, the yield is inevitably reduced, though the subsequent distillation can be single distillation. For removing these metal impurities, precise distillation may be easily thought out, where, however, vacuum distillation is necessary because the tantalum alkoxide is low in the vapor pressure, therefore, the separation and purification using the difference in the vapor pressure from impurity compounds is fairly difficult as described in the patent publication.

On the other hand, for separation of necessary target components from component combinations, it is most common to employ methods that yield only the target components, whereby a sample containing a component combination is passed through a separating apparatus using a separation method such as an ion-exchange separation method, a solvent extraction method, a precipitation separation method, a gas/liquid separation method, a distillation method and the like, employed either alone or in combination. The separation is often accomplished within the short time of a few seconds to a few minutes, but longer periods of a few hours to a few days are sometimes required. After separation of the target components, steps for washing and regeneration of the separating apparatus are necessary, and such steps often require a long period comparable to that of the separating procedure. Regardless of the length of the analysis time, however, usually only one separating apparatus is present in the analyzer, and therefore an analyzer or an analysis method that can shorten the analysis time has been desired.

When long periods are required for separation

of target components or for washing and regeneration of separating apparatuses, a single separating apparatus is inefficient because it cannot simultaneously accomplish the separation procedure and the washing and regeneration procedures after separation. In particular, when the object is that of analyzing specific elements or trace impurity elements in materials, several minutes to several dozen minutes may be required depending on the type of the main component or target component to be separated, and it is extremely important to efficiently carry out the washing and regeneration steps for the separating apparatus after the separation procedure.

An example is the analysis of impurity elements contained in tantalum compounds, typically tantalum and tantalum alkoxides, of which the demand has been increasing in recent years for semiconductor uses; tantalum, as a constituent element of ferroelectric thin-films, is obtained from tantalum alkoxide starting materials, and since the thin-film performance largely depends on the impurity concentration, the amount of impurities in the tantalum alkoxide starting material must be no more than 1 $\mu\text{g/g}$. The impurities of interest here are transition metals such as iron, chromium and nickel, alkali metals and alkaline earth metals such as sodium and magnesium, heavy metals such as cadmium and lead, and non-metallic elements such as boron, phosphorus and their compounds.

In order to obtain highly precise measurement of trace amounts of impurity elements at no greater than 1 $\mu\text{g/g}$ in tantalum or tantalum compounds, it is essential to carry out a procedure of separation and removal of the main component of tantalum prior to detecting the impurity elements with the measuring device, and a variety of separation methods and measurement methods for impurity elements have been reported.

For example, metal elements are specified

according to JIS H1683 (Method for determination of iron in tantalum). However, the JIS analysis method is problematic in that:

5 (1) it is designed for analysis of the $\mu\text{g/g}$ level and higher, and

(2) it is a batch method, and the procedure is therefore time consuming and difficulties are encountered in strictly controlling the conditions such as the flow rate of the carrier solution, etc.

10 A widely known method of separating and measuring the non-metal element boron involves adding an alcohol (methanol) to the sample solution, distilling off the boron as trimethyl borate and absorbing it in an aqueous sodium hydroxide solution, and then measuring the
15 amount of boron by titration or spectrophotometry (JIS G1227: Method for determination of boron in iron and steel). However, when this method is applied to the determination of boron in tantalum, it is problematic in that:

20 (1) distillation of the boron as trimethyl borate is very difficult when fluorine is present during the distillation, and it is poorly suited as a means of separating boron from tantalum samples that will not easily dissolve without using hydrofluoric acid;

25 (2) the distillation apparatus and distillation procedure are complicated and involved;

(3) time is required for the distillation procedure and measuring procedure, and contamination from the environment becomes significant; and

30 (4) it is not sensitive enough to the determination on the level of below $1 \mu\text{g/g}$ by titration or spectrophotometry.

Another reported method involves production of ion pairs of boron in a sample solution with methylene
35 blue or the like in the presence of hydrofluoric acid, extraction in dichloroethane and measurement by

spectrophotometry (JIS G1227: Method for determination of boron in iron and steel), but this method is also problematic in that:

5 (1) the tantalum to be separated is also extracted in addition to boron, and

(2) the extraction procedure invites inclusion of contaminants from the reagents and apparatus.

10 As explained above, therefore, analysis of impurity elements in tantalum and tantalum compounds using conventional analysis methods entail the following problems:

15 (1) when longer periods are required for separation of the target components and washing and regeneration of the separating apparatus, the procedures are inefficient because the separation procedure and the washing and regeneration procedures after separation cannot be simultaneously accomplished;

20 (2) because they are batch methods, the procedures are time consuming and difficulties are involved in strictly controlling the conditions such as the flow rate of the carrier solution, etc.;

(3) distillation of the boron as trimethyl borate is very difficult when fluorine is present in the sample solution;

25 (4) the apparatuses and distillation procedures are complex and involved;

30 (5) the contamination from apparatus and reagents is common, while the analysis procedures are time consuming, and contamination from the environment becomes significant;

(6) they are not sensitive enough for analysis on the level of 1 $\mu\text{g/g}$; and

(7) tantalum cannot be removed by extraction.

BRIEF SUMMARY OF THE INVENTION

35 The present invention has been made under these circumstances and the object of the present invention is to provide a high purity pentaalkoxytantalum extremely

low in the contents of various metal impurities and further reduced in the chlorine element content, which is required of a semiconductor material, and also an industrially advantageous method for producing a high purity pentaalkoxytantalum in a good yield by vacuum distillation, which has heretofore been considered difficult.

As a result of extensive investigations to overcome the above-described problems, the present inventors have found that when a method of distilling a crude solution containing pentaalkoxytantalum using a distillation column is used, a high purity pentaalkoxytantalum reduced in the contents of various metals and also reduced in the chlorine element content can be produced and the object of the present invention can be attained. The present invention has been accomplished based on this finding. The present invention relates to the following means (1) to (17):

(1) a high purity pentaalkoxytantalum having Al, Ca, Cd, Cr, Cu, Fe, K, Mg, Mn, Na, Ni, Pb, Zn, Th and U contents each of less than 50 ppb;

(2) the high purity pentaalkoxytantalum as described in (1) above, wherein the contents of Al, Ca, Cd, Cr, Cu, Fe, K, Mg, Mn, Na, Ni, Pb, Zn, Th and U are each less than 20 ppb;

(3) the high purity pentaalkoxytantalum as described in (1) above, wherein the contents of Al, Ca, Cd, Cr, Cu, Fe, K, Mg, Mn, Na, Ni, Pb, Zn, Th and U are each less than 5 ppb;

(4) the high purity pentaalkoxytantalum as described in any one of (1) to (3) above, wherein the chlorine element content is less than 1 ppm;

(5) the high purity pentaalkoxytantalum as described in any one of (1) to (4) above, wherein the pentaalkoxytantalum is pentaethoxytantalum;

(6) a method for manufacturing a high purity pentaalkoxytantalum having Al, Ca, Cd, Cr, Cu, Fe, K, Mg,

Mn, Na, Ni, Pb, Zn, Th and U contents each of less than 50 ppb, comprising distilling a pentaalkoxytantalum crude solution using a distillation column;

5 (7) the method for manufacturing a high purity pentaalkoxytantalum as described in (6) above, wherein the high purity pentaalkoxytantalum has a chlorine element content of less than 1 ppm;

10 (8) the method for manufacturing a high purity pentaalkoxytantalum as described in (6) or (7) above, wherein the distillation column is a packed column;

(9) the method for manufacturing a high purity pentaalkoxytantalum as described in (8) above, wherein the packed column is packed with Sulzer packing;

15 (10) the method for manufacturing a high purity pentaalkoxytantalum as described in any one of (6) to (9) above, wherein the distillation is performed through a theoretical plate number of from 2 to 100 stages;

20 (11) the method for manufacturing a high purity pentaalkoxytantalum as described in any one of (6) to (10) above, wherein the distillation is performed at a bottom temperature of 300°C or less;

25 (12) the method for manufacturing a high purity pentaalkoxytantalum as described in any one of (6) to (11) above, wherein the distillation is performed at a top column temperature of 300°C or less;

(13) the method for manufacturing a high purity pentaalkoxytantalum as described in any one of (6) to (12) above, wherein the distillation is performed at a top column pressure of 1,500 Pa or less;

30 (14) the method for manufacturing a high purity pentaalkoxytantalum as described in any one of (6) to (13) above, wherein the pentaalkoxytantalum crude solution is obtained through a first step of reacting a tantalum halide with an alcohol in an inactive solvent and removing the hydrogen halide produced on the reaction
35 by bubbling an inert gas, and a second step of adding

ammonia or an amine to the reaction solution resulting from the first step to obtain a pentaalkoxytantalum crude solution;

5 (15) the method for manufacturing a high purity pentaalkoxytantalum as described in any one of (6) to (14) above, wherein the pentaalkoxytantalum crude solution is obtained through a first step for performing the reaction of a tantalum halide with an alcohol in an inactive solvent under atmospheric pressure and then
10 reducing the pressure or performing the reaction under reduced pressure, thereby removing the hydrogen halide produced on the reaction, and a second step for adding ammonia or an amine to the reaction solution resulting from the first step to obtain a pentaalkoxytantalum crude
15 solution;

(16) the method for manufacturing a high purity pentaalkoxytantalum as described in (14) or (15) above, wherein the inactive solvent is toluene; and

(17) the method for manufacturing a high purity
20 pentaalkoxytantalum as described in any one of (6) to (16) above, wherein the pentaalkoxytantalum is pentaethoxytantalum.

Further, it is an object of the present invention to provide an analyzer with separating apparatus capable of
25 rapidly and efficiently analyzing impurity elements in samples, a continuous flow analysis method, high purity tantalum and high purity tantalum compounds, and a ferroelectric thin-film formed using them.

As a result of diligent research aimed at overcoming
30 the above-mentioned problems, the present inventors completed the present invention upon finding that the problems can be solved and impurity elements can be analyzed at a high sensitivity in a short period of time by using an analysis method whereby a plurality of
35 separating apparatuses are connected in parallel, and at least one of the separating apparatuses connected in parallel is used for analysis while at least one of the

other separating apparatuses is washed and regenerated, so that analysis and washing are carried out simultaneously. Thus, the present invention also provides a parallel separating analyzer, a continuous
5 flow analysis method, high purity tantalum and high purity tantalum compounds, and a ferroelectric thin-film formed using them, as laid out in (18) to (32) below.

(18) A parallel separating analyzer, which is a continuous flow analyzer equipped with a liquid
10 transporter, a separating apparatus, a sample introduction valve, a solution switching valve, an impurity remover and a measuring device, characterized in that a plurality of separating apparatuses are provided, each separating apparatus is connected in parallel, and
15 the separating apparatuses are connected in such a manner that at least one of the separating apparatuses is used for analysis while at least one of the other separating apparatuses is washed and regenerated, so that analysis and washing and regeneration are carried out
20 simultaneously.

(19) A parallel separating analyzer according to (18) above, wherein the separating apparatuses are connected in such a manner that solutions of different compositions are simultaneously supplied to the
25 separating apparatus used for analysis and the separating apparatus being washed and regenerated.

(20) A parallel separating analyzer according to (18) or (19) above, wherein the separating apparatuses employ, as the separating means, a method selected from
30 among ion-exchange separation methods, solvent extraction separation methods, precipitation separation methods, gas/liquid separation methods and distillation methods.

(21) A parallel separating analyzer according to any one of (18) to (20) above, wherein the impurity remover
35 employs, as the means for impurity removal, a method selected from among ion-exchange separation methods, solvent extraction separation methods, precipitation

separation methods, gas/liquid separation methods and distillation methods.

5 (22) A parallel separating analyzer according to any one of (18) to (21) above, wherein the measuring device employs an inductively coupled plasma mass spectrometer.

(23) A parallel separating analyzer according to any one of (18) to (22) above, which is used for analysis of impurity elements in a metal material.

10 (24) A continuous flow analysis method characterized in that a plurality of separating apparatuses are connected in parallel, and at least one of the plurality of separating apparatuses connected in parallel is used for analysis while at least one of the other separating apparatuses is washed, so that analysis and washing are
15 carried out simultaneously.

(25) A continuous flow analysis method according to (24) above, wherein solutions of different compositions are simultaneously supplied to the separating apparatus used for analysis and the separating apparatus being
20 washed and regenerated.

(26) A continuous flow analysis method according to (24) or (25) above, wherein the separating apparatuses employ as the separating means a method selected from among ion-exchange separation methods, solvent extraction
25 separation methods, precipitation separation methods, gas/liquid separation methods and distillation methods.

(27) A continuous flow analysis method according to any one of (24) to (26) above, which is used for analysis of impurity elements in a metal material.

30 (28) A continuous flow analysis method according to (27) above, wherein the metal material is tantalum or a tantalum compound, or niobium or a niobium compound.

(29) A continuous flow analysis method according to (27) or (28) above, wherein an inductively coupled plasma
35 mass spectrometer is used for the analysis of impurity elements.

(30) High purity tantalum or a high purity tantalum

compound, characterized by having a total impurity element concentration of no greater than 1000 ng/g.

(31) High purity tantalum or a high purity tantalum compound according to (30) above, wherein the high purity
5 tantalum compound is high purity pentaethoxytantalum.

(32) A ferroelectric thin-film characterized by being formed using high purity tantalum or a high purity tantalum compound with a total impurity element concentration of no greater than 1000 ng/g.

10 BRIEF DESCRIPTION OF THE DRAWINGS

Fig. 1 is a schematic view of a separating apparatus for tantalum and boron according to an embodiment of the invention.

Fig. 2 is a graph showing the relationship between
15 eluent (hydrochloric acid) concentration and amount of eluted boron, according to an embodiment of the invention.

Fig. 3 is a graph showing the relationship between eluent (hydrochloric acid) concentration and
20 concentration of eluted tantalum, according to an embodiment of the invention.

Fig. 4 is a schematic of a parallel separating analyzer showing one case of a separating analyzer used to carry out the invention.

25 DETAILED DESCRIPTION OF THE INVENTION

That is, the present invention provides a high purity pentaalkoxytantalum having Al, Ca, Cd, Cr, Cu, Fe, K, Mg, Mn, Na, Ni, Pb, Zn, Th and U contents each of less than 50 ppb and having a chlorine element content of less
30 than 1 ppm and a method for manufacturing a high purity pentaalkoxytantalum having Al, Ca, Cd, Cr, Cu, Fe, K, Mg, Mn, Na, Ni, Pb, Zn, Th and U contents each of less than 50 ppb and having a chlorine element content of less than 1 ppm, comprising distilling a pentaalkoxytantalum crude
35 solution using a distillation column.

The high purity pentaalkoxytantalum and the method for manufacturing the compound according to the present

invention are described below.

The high purity pentaalkoxytantalum of the present invention is preferably high purity pentaethoxytantalum extremely reduced in various metal impurities, which can be preferably used as a semiconductor material. The contents of metal impurities Al, Ca, Cd, Cr, Cu, Fe, K, Mg, Mn, Na, Ni, Pb, Zn, Th and U each is less than 50 ppb, preferably less than 20 ppb, more preferably less than 5 ppb. The chlorine element content is less than 1 ppm and this means that the pentaalkoxytantalum contains chloride, chloride ion or free chlorine in an amount of less than 1 ppm in terms of chlorine element.

The method for manufacturing a high purity pentaalkoxytantalum according to the present invention is described below.

The present invention is a method of distilling a crude solution containing a pentaalkoxytantalum and in particular, the method can be preferably used for manufacturing high purity pentaethoxytantalum. The method for producing a crude solution containing pentaalkoxytantalum is not particularly limited but, for example, the crude solution may be produced by the following method (1) or (2):

(1) a method comprising a first step for reacting a tantalum halide and an alcohol in an inactive solvent and removing the hydrogen halide produced on the reaction by bubbling an inert gas, and a second step for adding ammonia or an amine to the reaction solution resulting from the first step to obtain a pentaalkoxytantalum crude solution; or

(2) a method comprising a first step for performing the reaction of a tantalum halide with an alcohol in an inactive solvent under atmospheric pressure and then reducing the pressure or performing the reaction under reduced pressure, thereby removing the hydrogen halide produced on the reaction, and a second step for adding ammonia or an amine to the reaction solution resulting

from the first step to obtain a pentaalkoxytantalum crude solution. In both methods, toluene is preferably used as an inactive solvent, insoluble salt deposited after adding ammonia or an amine is filtered if necessary, and
5 more preferably, distillation is performed after concentrating moderately.

The distillation is performed using a distillation column by placing a moderately concentrated pentaalkoxytantalum crude solution in a bottom container equipped with a heating device. The distillation column
10 is preferably a packed column using a packing. Various high performance packing materials heretofore developed may be used as the packing for the packed distillation column used in the present invention, however, Dikson
15 rings as a random packing material or a Sulzer packing as a structured packing material may be suitably used. Of these, a Sulzer packing is preferred.

With respect to the theoretical plate number for use in the present invention, if it is too small, impurities
20 cannot be satisfactorily separated and the concentrations of various metals increase, whereas an excessively large theoretical plate number is not preferred in view of profitability, though there is no adverse effect on the quality. The optimal plate number is selected by taking
25 account of the structure of alkoxy group in the pentaalkoxytantalum or the purity before distillation, however, the plate number is preferably from 2 to 100 stages, more preferably from 2 to 50 stages, and still more preferably from 2 to 10 stages, which is the number
30 used practically.

With respect to the bottom temperature at the distillation in the present invention, if it is excessively high, decomposition of the pentaalkoxytantalum takes places and the yield decreases,
35 whereas if the bottom temperature is low, although there is no adverse effect on the quality, a high vacuum is necessary and a special device is required therefor,

which is disadvantageous in view of profitability. The optimal bottom temperature varies depending on the structure of alkoxy group in the pentaalkoxytantalum but the bottom temperature is preferably from 150 to 300°C.

5 In the case of distillation of pentaethoxytantalum as a specific example, the bottom temperature is preferably from 170 to 230°C.

10 With respect to the top column temperature at the distillation in the present invention, if it is excessively high, decomposition of the pentaalkoxytantalum takes place and the yield decreases, whereas if the top column temperature is low, although there is no adverse effect on the quality, a high vacuum is necessary and a special device is required therefor, 15 which is disadvantageous in view of profitability. The optimal top column temperature varies depending on the structure of alkoxy group in the pentaalkoxytantalum but the top temperature is preferably from 150 to 300°C. In the case of distillation of pentaethoxytantalum as a 20 specific example, the top temperature is preferably from 160 to 230°C.

25 At the distillation in the present invention, if the top pressure is high, both the bottom temperature and the top temperature must be high but if these temperatures are excessively high, decomposition of the pentaalkoxytantalum takes place and the yield decreases, whereas if the top pressure is excessively low, although there is no adverse effect on the quality and it is not limited, a special device is required and this is 30 disadvantageous in view of profitability. The optimal top pressure varies depending on the structure of alkoxy group in the pentaalkoxytantalum but the top pressure is preferably from 10 to 1,500 Pa, more preferably from 10 to 700 Pa.

35 The contents of various metals Al, Ca, Cd, Cr, Cu, Fe, K, Mg, Mn, Na, Ni, Pb, Zn, Th and U referred to in

the present invention can be analyzed, for example, by an inductively coupled plasma mass spectrometer (ICP-MS). In the analysis by ICP-MS, the limit of determination of Al, Ca, Cd, Cr, Cu, Fe, K, mg, Mn, Na, Ni, Pb and Zn is 5 ppb, and the limit of determination of Th and U is 0.5 ppb.

The chlorine element content can be analyzed, for example, in such a manner that a pentaalkoxytantalum is mixed with chlorine-free water and thereby hydrolyzed and the supernatant is filtered and analyzed by ion chromatography.

According to the other aspect of the invention, at least two separating apparatuses most suited for separation of the target component and the non-target components are connected in parallel, and the solution is supplied simultaneously to the separating apparatus used for separation and the separating apparatus being washed and regenerated in order to simultaneously accomplish separation of the target and non-target components and washing and regeneration of the separating apparatus, thereby shortening the analysis time and accomplishing a more efficient separation procedure. When the analysis method of the invention is used for analysis of impurity elements in a sample, it is possible to accomplish rapid separation of trace impurity elements in high purity metals or high purity metal compounds using a continuous flow system while preventing contamination from the environment, and then to directly introduce them into a high sensitivity measuring apparatus which is sensitive enough for analysis of below 1 $\mu\text{g/g}$, for quantitative analysis of the impurity elements.

According to the invention, a parallel separating apparatus with a plurality of separating apparatuses connected in parallel is an apparatus in which a plurality of separating apparatuses most suited for separation of target components are connected in parallel, and the solution used for separation and the

solution used for washing and regeneration may be supplied simultaneously to at least two of the separating apparatuses. In this case, the target component may be in the form of a liquid, gas or solid. A trace impurity in a high purity metal or high purity metal compound according to the invention is an impurity element included in the high purity metal or high purity metal compound at no greater than 1 $\mu\text{g/g}$, and the sample may be in the form of a solid, liquid or gas. In this case, it is possible to supply the sample solution that has been liquefied with mineral acids or the like to the analyzer for analysis instead of simply supplying the sample directly to the analyzer. The sample solution supplied to the analyzer has the metals or metal compounds separated from the impurity elements by the separating apparatus, and all or a portion of the separated impurity elements are then introduced to a high sensitivity measuring apparatus for high sensitivity analysis of the impurity elements.

First, the parallel separating analyzer of the invention will be explained. The following explanation assumes a separating analyzer for trace impurities in tantalum which is a Group 5 element, but it is needless to mention that the parallel separating analyzer of the invention is not limited to a separating analyzer for trace impurities in tantalum.

The separating analyzer for trace impurities in tantalum comprises a liquid transporter, a separating apparatus, a sample introduction valve, a solution switching valve, an impurity remover, piping and a measuring device. The liquid transporter transports solutions such as mineral acids and standard solutions used for (1) separation of tantalum and impurity elements, (2) washing of the system of a measuring apparatus, (3) correction of the measuring apparatus sensitivity, or the like, at a fixed rate or at an appropriately adjusted flow rate, and when the

transported liquid is a highly corrosive mineral acid, it is preferred to use a liquid transport system based on a peristaltic pump using a Tygon tube. The pump flow speed can be altered as desired to match the necessary flow rate for the solution. The separating analyzer of the invention transports liquid to at least two separating apparatuses to accomplish separation and washing/regeneration simultaneously, and solutions of different compositions may also be transported separately at different desired flow speeds. Thus, multiple pumps may be used when different solutions are used.

The separating apparatus is an apparatus for separation of tantalum and impurity elements in a sample solution, and the separating method may be any separating method that allows effective separation of tantalum and impurity elements, selected from among ion-exchange separation methods, solvent extraction separation methods, precipitation separation methods, gas/liquid separation methods and distillation methods, and it is carried out with a plurality of separating apparatuses suited for the method used, arranged in parallel. Using ion-exchange separation as an example, an ion-exchange resin is packed into small tubes preferably of Teflon or polypropylene, and both ends of the resin are packed with Teflon wool or the like to prevent escape of the resin as the carrier solution flows through. When an ion-exchange separation method is used for separation of a matrix component and the target analysis component, the separation performance will vary depending on the inner diameter and length of the Teflon or polypropylene tubes, and the inner diameter and length of the tubes may be of any desired value depending on the nature of the analysis sample. Preferred for ease of use are tubes with an inner diameter of 1-10 mm and a length of 10-300 mm. The type of ion-exchange resin may be any type of resin selected according to the sample to be separated.

In the step of separating the tantalum and impurity

elements using this separation apparatus, a few dozen minutes may be required for washing and regeneration of the system after separation of the tantalum and impurity elements. Conventional analyzers using only one
5 separating apparatus have therefore been inefficient as they can analyze only a few samples per unit of time. The separating analyzer of the invention provides a plurality of separating apparatuses with the aim of maximizing the number of samples that can be analyzed per
10 unit of time. The parallel separating analyzer of the invention is designed with a plurality of separating apparatuses connected in parallel, and at least one of the plurality of separating apparatuses is used for analysis while at least one of the other separating
15 apparatuses is washed and regenerated after separation of the tantalum and impurity elements, so that the step of separating the tantalum and impurity elements and the step of washing and regeneration the system after separation of the tantalum and impurity elements can be
20 carried out simultaneously, thus increasing the number of samples that can be analyzed per unit of time. The number of apparatuses used here may be selected as desired, depending on the analysis time required for one step.

25 The sample introduction valve introduces the sample solution into the carrier solution that is transported from the peristaltic pump, and it may be situated at any desired location depending on the need, so long as it is before the separating apparatuses. The sample
30 introduction valve is preferably made of highly corrosion resistant Teflon or polypropylene at the liquid-contacting sections, and the amount of sample introduced may be determined by the inner diameter and length of the attached Teflon tubes.

35 The solution switching valve serves to allow a flow of the carrier solution to any desired separating apparatus by switching of the carrier solution flow, and

it is preferably made of highly corrosion resistant Teflon or polypropylene at the liquid-contacting sections. The solution switching valve is normally used for switching of the carrier solution, but it may be set
5 before the inlet of the measuring apparatus so as to introduce the washing/regeneration solution in the system after separation of the tantalum and impurity elements and prevent contamination of the measuring apparatus system so that the measuring apparatus system is always
10 washed with a clean solution in order to maintain a system of the measuring apparatus with a low baseline. Any desired number of sample introduction valves and solution switching valves may be provided depending on the type of carrier solution for the intended analysis
15 and the configuration of channels in the continuous flow system.

The impurity remover is provided for the purpose of preventing an increase in the baseline, during analysis, that can hamper or prevent analysis of trace amounts when
20 the element to be analyzed is present as an impurity in the carrier solution. The impurity removing means used may be any removing means that can effectively separate impurity elements, such as ion-exchange separation, solvent extraction separation, gas/liquid separation or
25 precipitation separation, but a column packed with an ion-exchange resin is preferred as it is effective and suitable for the purpose mentioned above. The inner diameter and length of the ion-exchange resin column may be altered as desired depending on the impurity element
30 concentration in the carrier solution to be separated, the carrier flow speed and the target baseline level. The location where the impurity remover is installed may be any desired location so long as it is upstream from the sample introduction valve.

35 The piping may be selected to match the type and nature of the carrier solution used. In particular, Teflon and polypropylene piping exhibit excellent

corrosion resistance against highly corrosive mineral acids such as hydrochloric acid, nitric acid and hydrofluoric acid, and they are preferred. The measuring apparatus is also preferably an inductively coupled plasma emission spectrometer or inductively coupled plasma mass spectrometer, with a high sensitivity inductively coupled plasma mass spectrometer being more preferred.

The analysis method of the invention will now be explained with reference to the parallel separating analyzer illustrated in general schematic form in Fig. 4. Fig. 4 is a schematic showing an embodiment of an apparatus for analysis of trace boron in tantalum, but the analysis method of the invention is not limited to the analysis of boron.

(1) The carrier solution 1 (14) is introduced using a peristaltic pump 9a for preliminary washing of the system. Here, an impurity remover 13 is provided at the inlet or outlet of the peristaltic pump 9a if necessary to prevent an increasing baseline of the measuring apparatus due to the component to be analyzed in the carrier solution 1 (shown in Fig. 4 at the inlet of the peristaltic pump). An anion-exchange resin column 12 through which the carrier solution 1 flows is used as at least one of the plurality of columns (any number of columns may be provided, and three are shown in Fig. 4). (Fig. 4 represents the boron separation flow path as a thick solid line.)

(2) The washing/regeneration solution 17 is introduced into the other column(s) through which the carrier solution 1 does not flow in (1). (In Fig. 4, the washing/regenerating solution flow path is indicated by a dotted line.)

(3) The sample introduction valve 10b is switched to introduce the sample solution 18 at a fixed rate into the flow of the carrier solution 1.

(4) The sample solution 18 introduced in (3) is

introduced into the anion-exchange resin column 12 at a fixed flow speed, for separation of the tantalum and impurity elements.

5 (5) If necessary, the tantalum and impurity elements in the sample solution introduced in (3) are introduced into the anion-exchange resin column 12 for adsorption of the entire amount, and then the solution switching valve 10a is switched to allow flow of a separate carrier solution 2 (15) for elution of only the impurity
10 elements.

(6) The carrier solution containing the impurity elements separated and eluted from the tantalum in (4) or (5) is directly introduced into the inductively coupled plasma mass spectrometer 16. If the sensitivity is
15 sufficient enough for analysis of trace amounts of under 1 $\mu\text{g/g}$, it is not necessary to introduce the full amount of the separated and eluted impurity elements into the measuring apparatus.

(7) After completion of the quantitative analysis by
20 the measuring apparatus, the solution switching valve 10g is switched to allow the washing/regeneration solution 17 to flow into the column used for analysis, while allowing the carrier solution 1 to flow into a different column through which the washing/regeneration solution 17 had
25 been flowing, and procedures (1) to (7) are then repeated.

As explained above, the separation and assay method for trace impurity elements in tantalum using the analysis method of the invention is characterized in
30 that:

(1) separation can be repeatedly carried out because the separation of tantalum and impurity elements in the anion-exchange column is carried out with transport of the carrier solution at a fixed flow rate by a
35 peristaltic pump;

(2) separation of tantalum and impurity elements can be accomplished in a short time because procedures such

as distillation separation and solvent extraction are not necessary;

(3) introduction of the sample once into the carrier solution of the continuous flow analysis system allows automatic separation of tantalum and impurity elements and quantitative analysis with the measuring apparatus;

(4) the parallel connection of separating apparatuses allows washing/regeneration of the column to be carried out in parallel with the separation procedure, thus permitting a greater amount of analysis per unit time;

(5) appropriate use of the solution switching valves allows a low baseline to be constantly maintained in the measuring apparatus system; and

(6) provision of an impurity remover allows the quantitative analysis to be carried out with very low inclusion of impurities by the elements to be analyzed in the carrier solution, for a constantly low baseline.

It is thereby possible to confirm total impurity element contents of 1000 ng/g or less in tantalum.

The parallel separating analyzer and analysis method of the invention were explained above with the assumption of an apparatus and method used for trace impurities in tantalum, but there are no particular restrictions on the targets for analysis using the parallel separating analyzer and analysis method of the invention. They are preferably used for analysis of impurity elements in metals and metal compounds, and examples of preferred compounds to be analyzed in addition to tantalum include tantalum (III) oxide, tantalum (V) oxide, pentaethoxytantalum, tantalum (III) chloride, tantalum (V) chloride, pentaisopropoxytantalum, pentamethoxytantalum, tantalum boride, tantalum nitride, tantalum silicide, niobium, niobium (III) oxide, niobium (V) oxide, pentaethoxyniobium, niobium (III) chloride, niobium (V) chloride, pentaisopropoxyniobium, pentamethoxyniobium, niobium boride, niobium nitride and

niobium silicide.

As examples of preferred impurity elements to be analyzed using the parallel separating analyzer and analysis method of the invention there may be mentioned

5 Li, Na, K, Rb and Cs of Group 1, Be, Mg, Ca and Ba of Group 2, Sc and Y, lanthanoids (La, Ce, Pr, Nd, Sm, Eu, Gd, Tb, Dy, Ho, Er, Tm, Yb, Lu), actinoids (Th, U) of Group 3, Ti, Zr and Hf of Group 4, V and Nb of Group 5, Cr, Mo and W of Group 6, Mn of Group 7, Fe of Group 8,

10 Co, Rh and Ir of Group 9, Ni of Group 10, Cu and Ag of Group 11, Zn and Cd of Group 12, B, Al, Ga, In and Tl of Group 13, Si, Ge, Sn and Pb of Group 14, P, As, Sb and Bi of Group 15, and Se and Te of Group 16, in the long form of the Periodic Table.

15 The present invention also provides a ferroelectric thin-film which is formed using high purity tantalum or a high purity tantalum compound with a total impurity concentration of no greater than 1000 ng/g as determined by the aforementioned analysis method, and the

20 ferroelectric thin-film is preferably formed using high purity pentaethoxytantalum with a total impurity concentration of no greater than 1000 ng/g. The ferroelectric thin-film is used as a base material for fabrication of surface acoustic wave devices or optical

25 devices, and it may be fabricated using the fabrication process described in Japanese Unexamined Patent Publication No. 4-301329, for example. The ferroelectric thin-film formed using the high purity tantalum or high purity tantalum compound has a very low impurity content

30 and therefore can provide improved performance, including reduced film crystal defects and an enhanced oxide film breakdown voltage, while it can also be suitably used as the aforementioned base material.

35 The present invention will be further illustrated below by referring to the Examples, however, the present invention should not be construed as being limited to these Examples.

Synthesis of Pentaethoxytantalum Crude Solution

To 250 g (0.70 mol) of tantalum pentachloride, 500 g of toluene was added and while keeping the temperature at from 45 to 55°C, 460 g (10 mol) of ethanol was added.

5 After the addition of ethanol, while maintaining the liquid temperature, a nitrogen gas was bubbled at 120 ml/min for 30 minutes. Subsequently, while keeping the liquid temperature, 60 g (3.5 mol) of ammonia was added. By the addition of ammonia, ammonium chloride was
10 produced as a solid, therefore, after the addition of ammonia, the reaction solution was cooled to room temperature and the ammonium chloride was filtered. Thereafter, the solvent was distilled off under reduced pressure and the residue was used as a
15 pentaethoxytantalum crude solution. The amount of chlorine element contained in the pentaethoxytantalum crude solution was analyzed by ion chromatography and found to be 1,000 ppm. Also, the contents of various metals were analyzed and the results are shown in Table
20 1.

Example 1

A distillation apparatus comprising a glass bottom flask (2L), a glass distillation column (column length: 1,200 mm, column diameter: 35 mm), a glass condenser, a
25 glass distillate receiver, a stainless steel Dikson ring packing, a bottom flask heater and a vacuum pump was used. A pentaethoxytantalum crude solution prepared by the above-described method was charged in the bottom flask and distilled. The distillation conditions were
30 set such that the bottom temperature was 220°C, the top temperature was 180°C and the top column pressure was 140 Pa. The distilled pentaethoxytantalum had a chlorine element content of less than 0.1 ppm. The contents of various metals were analyzed and the results obtained are
35 shown in Table 1.

Example 2

A distillation apparatus comprising a glass bottom flask (2L), a glass distillation column (column length: 1,200 mm, column diameter: 110 mm), a glass condenser, a glass distillate receiver, a stainless steel Sulzer packing, a bottom flask heater and a vacuum pump was used. A pentaethoxytantalum crude solution prepared by the above-described method was charged in the bottom flask and distilled. The distillation conditions were set such that the bottom temperature was 190°C, the top column temperature was 180°C and the top pressure was 140 Pa. The distilled pentaethoxytantalum had a chlorine element content of less than 0.1 ppm. The contents of various metals were analyzed and the results obtained are shown in Table 1.

Comparative Example

A pentaethoxytantalum crude solution prepared by the above-described method was distilled under conditions such that the distillation temperature was 180°C and the pressure was 270 Pa. The resulting pentaethoxytantalum single distillation product had a chlorine element content of 200 ppm. The contents of various metals were analyzed and the results obtained are shown in Table 1.

Table 1

Kind of Metal	Crude Solution	Example 1	Example 2	Comparative Example
Al	340	<5	<5	29
Ca	1300	<5	<5	22
Cd	10	<5	<5	<5
Cr	970	<5	<5	<5
Cu	10	<5	<5	<5
Fe	208,000	<5	<5	490
K	1,100	<5	<5	<5
Mg	750	<5	<5	<5
Mn	30	<5	<5	<5
Na	32,100	<5	<5	29
Ni	10	<5	<5	<5
Pb	10	<5	<5	<5
Zn	28	<5	<5	<5
Th	4	<0.5	<0.5	<0.5
U	5	<0.5	<0.5	<0.5

unit: ppb

As apparent from the results shown in Table 1, when the method of distilling a pentaalkoxytantalum crude solution using a distillation column of the present invention is used, the contents of various metals in the pentaalkoxytantalum can be extremely reduced and the chlorine element content can also be reduced.

Example 3

Fig. 1 shows a continuous flow tantalum and boron separating analyzer constructed using a peristaltic pump 1, a solution switching valve 2, a sample introduction valve 3 (0.15 ml sample introduction volume), an anion-exchange resin column 4 (inner diameter = 3 mm, length = 160 mm) and a Teflon tube (inner diameter = 0.8 mm), and

the apparatus shown in Fig. 1 was used to determine the adsorption of tantalum and boron onto the anion-exchange resin column 4. The anion-exchange resins used were commercially available strongly basic and weakly basic types (both 50-100 mesh), and the carrier solution 1 (5 in Fig. 1) was a hydrofluoric acid solution at a concentration of 0.1 mol/L transported at a flow speed of 2.0 ml/min.

Test solution 6 (a solution prepared by dissolving a tantalum sample in hydrofluoric acid and nitric acid, adding a boron standard solution and then diluting to a tantalum concentration of 100 mg/ml and a boron concentration of 40 µg/ml) was introduced into the anion-exchange resin column 4 of the separating apparatus through the sample introduction valve 3, and the solution eluting from the anion-exchange resin column 4 was fractionated out into a polypropylene bottle 7 every minute over a period of 6 minutes immediately after the sample solution introduction. The tantalum and boron contained in the fractionated out sample were each measured, the tantalum by inductively coupled plasma emission spectrometry and the boron by inductively coupled plasma mass spectrometry, giving the results shown in Table 2.

Table 2

Time/min	Strongly basic		Weakly basic	
	Tantalum/µg	Boron/ng	Tantalum/µg	Boron/ng
0-1	ND	ND	10300	1050
1-2	ND	ND	940	190
2-3	ND	ND	450	61
3-4	ND	ND	370	33
4-5	ND	ND	320	27
5-6	ND	ND	280	19

Note: ND indicates a tantalum elution of less than 0.2 µg and a boron elution of less than 0.2 ng.

With the strongly basic resin, no tantalum or boron was detected. This indicates adsorption of both elements to the resin. With the weakly basic resin, however, both tantalum and boron eluted, demonstrating that separation

of the two elements is difficult.

Example 4

The strongly basic anion-exchange resin column 4 used in Example 3 (inner diameter and length the same as Example 3) was used for introduction of test solution 6 in the same manner as Example 3, for adsorption of tantalum and boron onto an ion-exchange resin. The solution switching valve 2 was then switched to allow flow of carrier solution 2 (8 in Fig. 1) at four different concentrations (hydrochloric acid at 2, 3, 4 and 5 mol/l), and the solution eluting from the anion-exchange resin column 4 was fractionated out into a polypropylene bottle 7 every 20 seconds for a period of 5 minutes. The tantalum and boron contained in each fractionated out sample were each measured, the tantalum by inductively coupled plasma emission spectrometry and the boron by inductively coupled plasma mass spectrometry; the results for the boron measurement are shown in Fig. 2, and the results for the tantalum measurement are shown in Fig. 3.

From the results in Figs. 2 and 3 it is seen that a greater concentration of hydrochloric acid resulted in greater elution of boron, and although a greater amount of tantalum eluted as well, the proportion was smaller than that of the elution of boron. The tantalum/boron ratio in the test solution was 2500:1, while the tantalum/boron ratio in the hydrochloric acid eluates were approximately 32:1 (elution for about 2-3 minutes after switching the carrier solution to hydrochloric acid).

Example 5

The parallel separating analyzer shown in Fig. 4 was used for quantitative analysis of trace boron in tantalum. In Fig. 4, the amount of sample introduction through the sample introduction valve 11 was 0.15 ml, and three strongly basic anion-exchange resin columns 12 (inner diameter = 3 mm, length = 160 mm) were provided.

The measurement of boron was accomplished by switching each of the valves according to the time program shown in Table 3.

Table 3

Time/min	Procedure
Before analysis	(1) The peristaltic pump 9a is operated to allow a flow of 2.0 ml/min. (2) The solution switching valve 10a is switched to introduce the carrier solution 1 (14). It is passed through the impurity remover 13 to eliminate boron in the carrier solution 1 (14). (3) The solution switching valves 10b, 10c are switched to allow flow of the carrier solution 1 (14) to any one of the anion-exchange resin columns 12. (4) The solution switching valves 10g, 10j are then closed to prevent flow of the washing/regeneration solution 17. (5) The solution switching valve 10d is switched to allow flow of the carrier solution 1 (14). (6) The washing/regeneration solution is introduced by the peristaltic pump 9b. The solution switching valves 10e, 10f, 10h and 10i may be switched to allow flow of the washing/regeneration solution at this time to allow washing and regeneration simultaneously with separation of the target component. The waste solution is discharged to 19.
Start of analysis (0 min)	(1) The sample solution 18 is introduced from the sample introduction valve 11, and passed through the anion-exchange resin column 12 for adsorption of tantalum and boron in the sample solution.
1 min	(1) The solution switching valve 10d is switched to allow flow of the carrier solution 1 (14). (2) The solution switching valve 10a is switched to introduce the carrier solution 2 (15) and initiate elution of boron. At the same time, the measuring apparatus 16 is operated to measure the boron. The waste solution is discharged to 19.
6 min	(1) The solution switching valves 10b and 10c are switched and the solution switching valves 10g, 10j are also switched to introduce the washing/regeneration solution 17 into the column after separation, while the carrier solution 2 (15) is introduced into the anion-exchange resin column 12 to be used for the subsequent analysis. The solution switching valves 10f, 10i are closed at this time to prevent flow of the washing/regeneration solution.
9 min	(1) The solution switching valve 10a is switched to introduce the carrier solution 1 (14).
10 min	(1) The analysis procedure (0 min - 10 min) is repeated.

5

By using a parallel separating analyzer according to the invention it was possible to simultaneously carry out separation of the target component and

washing/regeneration of the column. The analysis speed was 6 samples per hour, which was about 6 times the number of samples that can be analyzed per hour with a single separating apparatus (one sample per hour). When the apparatus shown in Fig. 4 was used for measurement of boron contents in commercially available tantalum, the value was 150 ± 10 ng/g (as the average value of three repeated measurements with standard deviation) in terms of the original metal, and when used for measurement of boron contents in commercially available tantalum oxide, it was 26 ± 3 ng/g (same) in terms of the original oxide sample; the present invention can therefore be applied to make efficient measurements of trace boron in tantalum and tantalum compounds.

As described hereinabove, by using the manufacturing method of the present invention, a high purity pentaalkoxytantalum useful as a semiconductor material can be manufactured. Furthermore, by using the pentaalkoxytantalum extremely reduced in the contents of various metals and also reduced in the chlorine element content of the present invention, the yield of semiconductors is improved. Thus, the present invention is very useful.

Further, the use of a parallel separating analyzer according to the invention allows separation of non-target components and target components in a sample to be accomplished simultaneously with washing and regeneration of the column, for rapid and efficient separation of trace impurities in samples, thus allowing a greater number of samples to be treated per unit of analysis time. Furthermore, using an impurity removing apparatus maintains a state of minimal increase in the baseline due to the element to be analyzed in the carrier solution, while the use of a continuous flow system has the advantage of allowing contamination from the environment to be absolutely minimized during the analysis procedure. By using the analysis method of the invention, it is

possible to analyze the contents of trace impurity
elements in materials on the level of 1 $\mu\text{g/g}$ or lower,
and particularly in the case of ferroelectric thin-films
that are used as base materials for fabrication of
5 surface acoustic wave devices or optical devices, where
it is important to form them with materials having very
low impurity contents in order to reduce film crystal
defects and oxide film breakdown voltage problems, the
parallel separating analyzer and analysis method of the
10 invention is useful for analysis of pentaethoxytantalum
and the like that are used as materials for ferroelectric
thin-films.



CLAIMS

1. A high purity pentaalkoxytantalum having Al, Ca, Cd, Cr, Cu, Fe, K, Mg, Mn, Na, Ni, Pb, Zn, Th and U contents each less than 50 ppb.

5 2. The high purity pentaalkoxytantalum as claimed in claim 1, wherein the contents of Al, Ca, Cd, Cr, Cu, Fe, K, Mg, Mn, Na, Ni, Pb, Zn, Th and U are each less than 20 ppb.

10 3. The high purity pentaalkoxytantalum as claimed in claim 1, wherein the contents of Al, Ca, Cd, Cr, Cu, Fe, K, Mg, Mn, Na, Ni, Pb, Zn, Th and U are each less than 5 ppb.

15 4. The high purity pentaalkoxytantalum as claimed in any one of claims 1 to 3, wherein the chlorine element content is less than 1 ppm.

5. The high purity pentaalkoxytantalum as claimed in any one of claims 1 to 4, wherein said pentaalkoxytantalum is pentaethoxytantalum.

20 6. A method for manufacturing a high purity pentaalkoxytantalum having Al, Ca, Cd, Cr, Cu, Fe, K, Mg, Mn, Na, Ni, Pb, Zn, Th and U contents each less than 50 ppb, comprising distilling a pentaalkoxytantalum crude solution using a distillation column.

25 7. The method for manufacturing a high purity pentaalkoxytantalum as claimed in claim 6, wherein the high purity pentaalkoxytantalum has a chlorine element content less than 1 ppm.

30 8. The method for manufacturing a high purity pentaalkoxytantalum as claimed in claim 6 or 7, wherein said distillation column is a packed column.

9. The method for manufacturing a high purity pentaalkoxytantalum as claimed in claim 8, wherein said packed column is packed with Sulzer packing.

35 10. The method for manufacturing a high purity pentaalkoxytantalum as claimed in any one of claims 6 to 9, wherein the distillation is performed through a theoretical plate number of from 2 to 100 stages.



11. The method for manufacturing a high purity pentaalkoxytantalum as claimed in any one of claims 6 to 10, wherein the distillation is performed at a bottom temperature of 300°C or less.

5 12. The method for manufacturing a high purity pentaalkoxytantalum as claimed in any one of claims 6 to 11, wherein the distillation is performed at a top column temperature of 300°C or less.

10 13. The method for manufacturing a high purity pentaalkoxytantalum as claimed in any one of claims 6 to 12, wherein the distillation is performed at a top column pressure of 1,500 Pa or less.

15 14. The method for manufacturing a high purity pentaalkoxytantalum as claimed in any one of claims 6 to 13, wherein said pentaalkoxytantalum crude solution is obtained through a first step for reacting a tantalum halide with an alcohol in an inactive solvent and removing the hydrogen halide produced on the reaction by bubbling an inert gas, and a second step for adding
20 ammonia or an amine to the reaction solution resulting from the first step to obtain a pentaalkoxytantalum crude solution.

25 15. The method for manufacturing a high purity pentaalkoxytantalum as claimed in any one of claims 6 to 14, wherein said pentaalkoxytantalum crude solution is obtained through a first step for performing the reaction of a tantalum halide with an alcohol in an inactive solvent under atmospheric pressure and then reducing the pressure or performing the reaction under reduced
30 pressure, thereby removing the hydrogen halide produced on the reaction, and a second step for adding ammonia or an amine to the reaction solution resulting from the first step to obtain a pentaalkoxytantalum crude solution.

35 16. The method for manufacturing a high purity pentaalkoxytantalum as claimed in claim 14 or 15, wherein



said inactive solvent is toluene.

17. The method for manufacturing a high purity pentaalkoxytantalum as claimed in any one of claims 6 to 16, wherein said pentaalkoxytantalum is pentaethoxytantalum.

18. A parallel separating analyzer, which is a continuous flow analyzer equipped with a liquid transporter, a separating apparatus, a sample introduction valve, a solution switching valve, an impurity remover and a measuring device, characterized in that a plurality of separating apparatuses are provided, each separating apparatus is connected in parallel, and the separating apparatuses are connected in such a manner that at least one of the separating apparatuses is used for analysis while at least one of the other separating apparatuses is washed and regenerated, so that analysis and washing and regeneration are carried out simultaneously.

19. A parallel separating analyzer according to claim 18, wherein the separating apparatuses are connected in such a manner that solutions of different compositions are simultaneously supplied to the separating apparatus used for analysis and the separating apparatus being washed and regenerated.

20. A parallel separating analyzer according to claim 18 or 19, wherein the separating apparatuses employ, as the separating means, a method selected from among ion-exchange separation methods, solvent extraction separation methods, precipitation separation methods, gas/liquid separation methods and distillation methods.

21. A parallel separating analyzer according to any one of claims 18 to 20, wherein the impurity remover employs, as the means for impurity removal, a method selected from among ion-exchange separation methods, solvent extraction separation methods, precipitation separation methods, gas/liquid separation methods and distillation methods.



22. A parallel separating analyzer according to any one of claims 18 to 21, wherein the measuring device employs an inductively coupled plasma mass spectrometer.

23. A parallel separating analyzer according to any
5 one of claims 18 to 22, which is used for analysis of impurity elements in a metal material.

24. A continuous flow analysis method characterized in that a plurality of separating apparatuses are connected in parallel, and at least one of the plurality
10 of separating apparatuses connected in parallel is used for analysis while at least one of the other separating apparatuses is washed, so that analysis and washing are carried out simultaneously.

25. A continuous flow analysis method according to
15 claim 24, wherein solutions of different compositions are simultaneously supplied to the separating apparatus used for analysis and the separating apparatus being washed and regenerated.

26. A continuous flow analysis method according to
20 claim 24 or 25, wherein the separating apparatuses employ, as the separating means, a method selected from among ion-exchange separation methods, solvent extraction separation methods, precipitation separation methods, gas/liquid separation methods and distillation methods.

25 27. A continuous flow analysis method according to any one of claims 24 to 26, which is used for analysis of impurity elements in a metal material.

28. A continuous flow analysis method according to
30 claim 27, wherein the metal material is tantalum or a tantalum compound, or niobium or a niobium compound.

29. A continuous flow analysis method according to claim 27 or 28, wherein an inductively coupled plasma mass spectrometer is used for the analysis of impurity elements.

35 30. High purity tantalum or a high purity tantalum compound, characterized by having a total impurity element concentration of no greater than 1000 ng/g.

31. High purity tantalum or a high purity tantalum compound according to claim 30, wherein said high purity tantalum compound is high purity pentaethoxytantalum.

5 32. A ferroelectric thin-film characterized by being formed using high purity tantalum or a high purity tantalum compound with a total impurity element concentration of no greater than 1000 ng/g.

