COMMONWEALTH OF AUSTRALIA
PATENTS ACT 1952
APPLICATION FOR A STANDARD PATENT

Ringsdorff-Werke GMBH, of Drachenburgstr. 1, D-5300 Bonn 2, FEDERAL REPUBLIC OF GERMANY, hereby apply for the grant of a standard patent for an invention entitled:

Graphite Tube Furnace with Specimen Support for Atomic Absorption Spectroscopy

which is described in the accompanying complete specification.

Details of basic application(s):

Basic Applic. No: P 38 23 346.0
Country: DE
Application Date: 9 July 1988

The address for service is:
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DATED this FOURTEENTH day of JUNE 1989
Ringsdorff-Werke GMBH

By: Registered Patent Attorney

TO: THE COMMISSIONER OF PATENTS
OUR REF: 97282
S&F CODE: 61910

S008181 15/06/89
5845/2
In support of the Convention Application made for a patent for an invention entitled:

Graphite Tube Furnace with Specimen Support
For Atomic Absorption Spectroscopy

Dr. Walter Ulsamer and Willy Bahrs

I/we, .......................................................

[full name of declarant(s)]

of ...........................................................

[full address of declarant(s) - not post office box]

Drachenburgstr. 1, D-5300 Bonn 2, Germany

...(respectively)

do solemnly and sincerely declare as follows:-

1. I/we are authorised by Ringsdorff-Werke GMBH, the applicant for the patent to make this declaration on its behalf.

2. The basic application as defined by Section 141 of the Act was made in Federal Republic of Germany on 9 July 1988 by Ringsdorff-Werke GMBH.

3. Bruno Hutsch and Bernd Schmidt, of Flerzheimer Allee 11, D-5300 Bonn 1, Federal Republic of Germany and Zanderstr. 8, D-5300 Bonn 2, Federal Republic of Germany [respectively], are the actual inventors of the invention and the facts upon which the applicant is entitled to make the application are as follows:

Ringsdorff-Werke GMBH is entitled by Contract of Employment between the inventors as employees and Ringsdorff-Werke GMBH as employer, as a person who would be entitled to have the patent assigned to it if a patent were granted upon an application made by the inventors.

4. The basic application referred to in paragraph 2 of this Declaration was the first application made in a Convention country in respect of the invention the subject of the application.

DECLARED at Bonn this 20th day of September 1989

Signature of Declarant

TO: THE COMMISSIONER OF PATENTS

AUSTRALIA

ppa Dr. Walter Ulsamer ppa Willy Bahrs

Procurist Procurist

JED/134D
1. A graphite tube furnace with a specimen support for atomic absorption spectroscopy arranged in the furnace, the inside surface of the tube having at least two peripheral flanges on which the specimen support is supported at a distance from the tube wall.

2. A graphite tube furnace according to claim 1, wherein the flanges are formed in the shape of knife edges.

5. A graphite tube furnace according to any one of claims 1 to 4, wherein one flange has no recesses and limits lengthwise movements of the specimen support like a buffer.

6. A graphite tube furnace according to any preceding claim, wherein the interior surfaces of the furnace are coated with pyrocarbon.
FORM 10
COMMONWEALTH OF AUSTRALIA
PATENTS ACT 1952
COMPLETE SPECIFICATION

(ORIGINAL)

FOR OFFICE USE:

Class    Int Class

Complete Specification Lodged:
Accepted:
Published:

Priority:

Related Art:

Name and Address of Applicant:
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Complete Specification for the invention entitled:

Graphite Tube Furnace with Specimen Support for Atomic Absorption Spectroscopy

The following statement is a full description of this invention, including the best method of performing it known to me/us
GRAPHITE TUBE FURNACE WITH SPECIMEN SUPPORT FOR ATOMIC ABSORPTION SPECTROSCOPY

A graphite tube furnace with specimen support for use in atomic absorption spectroscopy has the wall inside the tube furnace provided with preferably knife-edged shaped flanges on which the specimen support is held at a distance from the wall thereby to avoid production of heat in the specimen support and heat flow from the tube furnace to the specimen support, the latter being held at a distance from the tube wall.
GRAPHITE TUBE FURNACE WITH SPECIMEN SUPPORT FOR ATOMIC
ABSORPTION SPECTROSCOPY

This invention relates to a graphite tube furnace with a specimen support for atomic absorption spectroscopy arranged in the furnace.

In flameless atomic absorption spectroscopy (AAS), graphite tube furnaces which are heated by direct resistance heating to the necessary temperature are used for vaporisation and atomising of the specimen substance. The specimen is applied directly through an opening in the tube wall directly onto the wall surface of the tube furnace or is introduced into the furnace from the ends with the assistance of a specimen support provided with a small recess for receipt of the specimen, designated also as a platform. As a result of use of special specimen supports, the position of the specimen can be localised exactly and errors as a result of possible temperature gradients along the tube axis are largely avoided. On the other hand, it has been found that the heating rate of the specimen support consisting likewise of graphite is changed essentially by contact between tube wall and the specimen support, since one part of the flow of heat flows through the specimen support (Fresenius Z. Anal. Chem. 1986, 323, 748-753). The effect of different heating rates and temperature differences between tube and specimen support has been investigated by I.L. Shuttler and H.T. Delves for the example of measurement of small lead contents in blood (J. Analyt. Atomic Spectroscopy 1987, 2, 171). They found, above all, large differences in the time lag of the signal, the integral absorption and there was so large a variation in the measurement values that the method was not suitable for this purpose. A solution to the problem is expected in specimen supports which are heated exclusively by irradiation and not by Joules heat, no bodily contact
thus being had with the graphite tube furnace and the sample being held in a reproducible position in the graphite tube.

Essentially, three arrangements of specimen support have become known, which are distinguished by comparatively small contact surfaces between specimen support and tube wall and which significantly restrict electrical conduction and heat conduction between wall and support. Typical of the first group is the arrangement according to German Patent Specification 29 24 123. The specimen support has a trapezoidal cross-section and the edges of the wider side engage in flat dovetail shaped channels milled in the tube wall. In the second arrangement, the specimen support is provided with a lug which has a small cross-sectional area, is inserted in a bore in the graphite tube and the specimen support is held at a distance from the tube wall (DE-A-35 45 635). Typical of the third group is a specimen support whose widened end section engages in slots which extend from one end of the graphite tube. The specimen supporting part of the support is held extending freely at a distance from the tube wall (DE-A-37 22 379).

All described solutions reduce the flow of heat between graphite tube and specimen support and the production of heat in the specimen support, make possible the precise establishment of the specimen support relative to the graphite tube and reduce to a large part the further above described disadvantages. When rapid heating of arrangements of the first and third types takes place many times, damaging, however, occurs which restricts very much use of the graphite tubes. Fine cracks emanate from the grooves or slots serving for the guiding and fixing of the specimen supports, the cracks giving rise, after heating many times, to rupturing of the graphite tube. Non identical thermal coefficients of expansion of specimen support and tube impose additional
pressure stresses on heating which can lead to tearing
off of the slotted section. Breakdowns as a result of arcing are frequent if the mechanical stresses are
restricted by a larger clearance. In this case, there are undesired vibrations too, on proximity to magnetic
tields. Graphite tubes provided with grooves can also be used, only in combination with the specimen support, since specimen substance supplied directly to the wall surface collects in the grooves and is distributed over the entire wall surface. With arrangements of the second type, the production cost of specimen support with turned plugs is very large, on account of the small amount and brittleness of the graphite material. In fact only vitreous carbon is suitable for specimen supports of this type, on account of its high strength.

The object underlying the invention is to form graphite tube furnaces so that the specimen supports are held at a distance from the tube surface, the strength of the tube furnace is not influenced by grooves or slots and the production of specimen supports is simple. Another object is the equalisation of temperature profile in the graphite tube furnace.

The object is solved by a graphite tube furnace whose internal wall surface is provided with at least two circular flanges on which the test support lies at a distance from the tube wall.

The cross-section of the flanges is appropriately wedge-shaped and is formed preferably as a knife edge for reduction of the contact surfaces. Their base width amounts to about 0.5 to 1.0 mm. In the production of the tube furnace provided with flanges, graphite cylinders are bored out and worked up by backed off drilling of the flanges which are arranged conveniently symmetrically in the tube, that is are spaced equally distanced as a pair from both end surfaces of the tube. The abrasive resistance of the flanges, which in general is comparatively small, is, as a result of a coating of
the wall surfaces of the tube surface with pyrocarbon, which is known and usual, matched to known stress requirements so that the flanges are not damaged by operation of the furnace. The positioning of the specimen support is established reproducibly by slot-shaped recesses in the flanges which are produced conveniently by reaming or high frequency chiselling and is fixed by means of a buffer. Serving conveniently as a buffer is a flange which is not provided with recesses and is adjacent one end surface of the tube furnace.

The slotting of the graphite tube is limited exclusively to the webs and does not extend as with a previously known construction over the entire length of the tube so that, for geometric reasons already, the probability of the formation of critical cracks destroying the tube must be very much less. In fact hair-line cracks extending from the slots end in the roots of the flange and do not affect the operating life of the tube furnace. Since a bodily contact between tube furnace and specimen support likewise only occurs in the region of the flanges, electrical and thermal resistance are so large that current and heat flow between tube furnace and specimen support are predominantly excluded and the heating of the specimen substance takes place practically exclusively by irradiation. The flanges arranged symmetrically with respect to the tube ends delimit in addition a central part of the furnace in which approximately isothermal conditions prevail. Such conditions also guarantee reproducible results when atomisation of the specimen substance takes place at the tube wall of the furnace.

An arrangement consisting of a graphite tube furnace and a specimen support with a radially symmetrical flange arranged in the interior of the tube is known from DE-A- 37 22 379. This flange is provided with grooves through which the holder part of the specimen support is slid. With too small a clearance,
like in the fixing of the specimen support in slots at
the end, the tube can rupture as a consequence of
differential thermal expansion of both parts. Different
expansions, which are hardly to be avoided with the usual
range of graphite products, does not however affect the
functioning of a graphite furnace in which the specimen
support lies freely on flanges and which must be provided
correspondingly at least as a pair.

Tube furnaces and specimen supports consist of any
desired type of graphite like electrographite,
pyrographite or vitreous carbon. Preferred is purest
graphite obtained from electrographite, which is easily
workable and only slightly rendered impure by foreign
elements. Tube furnaces and supports are conveniently
coated with a thin pyrographite layer which seals the
graphite parts and improves their abrasion resistance.

For a better understanding of the invention and to
show how the same can be carried into effect, reference
will now be made, by way of Example only to the
accompanying drawings, wherein:

FIGURE 1 shows a cross-section through a graphite
tube furnace with specimen support inserted.
FIGURE 2 shows the section II-II in Figure 1
FIGURE 3 shows the section III-III in Figure 1 and
FIGURE 4 shows the section IV-IV in Figure 1.

The graphite tube furnace 1 is provided with
circular flanges 2 in which slot-like grooves 3 are
worked and with the opening 6 for supply of the specimen.
The specimen support 4, which is provided with the recess
5 for receipt of the analysis specimens and with
prong-shaped extensions 7, rests on the flanges 2 and is
guided in the slot-like grooves 3. The unslotted flange
2' limits, like a buffer, movement of the specimen
support engaging in the slot-shaped grooves parallel to
the longitudinal extent of the graphite tube furnace.

The size of the support surfaces of the specimen
support 4 is determined by the width of the flanges 2 and
the depth of the slot-shaped grooves. With knife-edged shaped formation of the flanges, in particular, the support is almost pointlike and correspondingly the energy flow through the supports on operation of the furnace is so small that temperature and temperature distribution of the specimen support are not influenced.
CLAIMS
The claims defining the invention are as follows:

1. A graphite tube furnace with a specimen support for atomic absorption spectroscopy arranged in the furnace, the inside surface of the tube having at least two peripheral flanges on which the specimen support is supported at a distance from the tube wall.

2. A graphite tube furnace according to claim 1, wherein the flanges are formed in the shape of knife edges.

3. A graphite tube furnace according to Claim 2, wherein the knife edges have a base width of 0.5 to 1.0 mm.

4. A graphite tube furnace according to claim 1, 2 or 3, wherein the flanges are provided with slot shaped recesses for guiding the specimen support.

5. A graphite tube furnace according to any one of claims 1 to 4, wherein one flange has no recesses and limits lengthwise movements of the specimen support like a buffer.

6. A graphite tube furnace according to any preceding claim, wherein the interior surfaces of the furnace are coated with pyrocarbon.

7. A graphite tube furnace substantially as hereinbefore described, with reference to, and as shown in, the accompanying drawings.

DATED this SIXTH day of JUNE 1989
Ringsdorff-Werke GMBH

Patent Attorneys for the Applicant
SPRUSON & FERGUSON
END