

AN ATMOSPHERIC SOLIDS ANALYSIS SOURCE ASSEMBLY

5 Description of Invention

The present invention relates to a nozzle for directing heated gas onto the distal end of a capillary. The invention further relates to a source assembly
10 including the nozzle.

Background of the invention

The invention generally relates to an atmospheric solids analysis probe
15 (ASAP). Such probes, and the associated instrument for use with ASAP, are provided by several manufacturers, including Waters Corporation, Milford, MA, U.S.A.

ASAP is a useful and relatively cheap tool for use in the direct analysis of
20 volatile and semi-volatile, solid and liquid samples and may be used in the analysis of speciality chemicals, synthetic polymers, energy sources and food.

A sample is introduced into an ion source housing (e.g. an API source), in which the sample is volatilised using a heated gas, such as nitrogen, and the
25 sample is then ionised using, for example, a corona discharge pin. The ionised sample may subsequently be analysed in a mass spectrometer.

The sample is introduced into the source by loading it onto the tip of a capillary. The capillary may comprise a conventional glass capillary. The
30 capillary may be a solid rod, or a tube, with open ends.

Capillaries are fragile and susceptible to contamination. To ensure reliable and accurate analysis, the tip of the capillary must be inserted into the source in a repeatable manner.

- 5 To assist in the loading of a capillary into a source, it is known to provide a holder comprising a clamp mechanism which serves to retain the proximal end of the capillary (opposite the tip at the distal end which carries a sample) in the capillary holder. This may provide a user with a more robust method of handling the capillary, and may also assist in the guiding of the capillary into
10 the source. The capillary holder, and/or the source instrument, may comprise a guide mechanism to ensure the correct alignment of the capillary as it is loaded into the source.

When the capillary is arranged in the ion source housing, the distal end of the
15 capillary is arranged adjacent the outlet of a nozzle for directing heated gas onto the capillary. The outlet may comprise a single circular aperture, which may be configured to optimise the velocity and flow rate of a heated gas exiting the outlet and being directed onto the distal end of the capillary. However, the "beam" of hot gas leaving a single circular aperture may not be
20 broad enough so as to effectively heat and vaporise substantially all of a sample provided on the distal end of the capillary.

If a significant amount of the sample is not adequately heated by the nozzle, it may subsequently not be effectively volatilised into the gas phase and hence
25 would not be ionised by the corona discharge pin. This may then reduce the speed and accuracy of the subsequent measurement. Ineffective volatilisation through incomplete heating of the sampling region may result in extended analysis times, with high mass high boiling point compounds being volatilised over such an extended period that it may manifest as undesirable background
30 in the mass spectrum and subsequent analyses.

Increasing the size of the single circular aperture, and thus the diameter of the beam of hot gas, may adversely affect the velocity and/or flow rate of the heated gas, and may also adversely affect the temperature of the heated gas as it leaves the outlet of the nozzle. In turn, this may adversely affect the measurements derived from the ionised sample.

Moreover, in use, it may not be possible to achieve positional accuracy of the distal end of the capillary tip relative to the outlet of the heater. This may be due to tolerance stack-up of the various mechanical arrangements of the ASAP assembly (e.g. the clamp, the guide mechanism and/or the ion source housing). Accordingly, even if the capillaries used are of substantially the same length, the position of the distal end of one capillary relative to the outlet of the heater may differ to the position of another capillary relative to the outlet of the heater. Thus, even if the position of a single aperture outlet relative to the distal end is optimal for one capillary, it may not be optimal for a subsequent capillary loaded into the source assembly.

The present invention seeks to address at least one of the aforementioned problems.

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Accordingly, the present invention provides a nozzle for directing heated gas onto the distal end of a capillary arrangable adjacent the nozzle, the nozzle comprising:

a housing defining a plenum for heated gas; and
at outlet comprising at least one aperture fluidly connected to the plenum, the outlet configured to direct a curtain of the heated gas onto the distal end of a capillary in use, such that the curtain of heated gas is substantially aligned with the longitudinal axis of the capillary.

30 In at least one embodiment, the outlet comprises a plurality of apertures arranged substantially linearly.

In at least one embodiment, the outlet comprises four apertures.

5 In at least one embodiment, each of the apertures has substantially the same surface area.

In at least one embodiment, each of the apertures is substantially circular.

10 In at least one embodiment, the aperture(s) is/are non-circular.

In at least one embodiment, the aperture(s) is/are elongated.

In at least one embodiment, the outlet comprises a single elongate aperture.

15 In at least one embodiment, the outlet comprises a plurality of elongate apertures, wherein the direction of elongation is substantially perpendicular to the plane of the curtain of heated gas.

20 In at least one embodiment, the nozzle further comprises a heater arrangement, for heating gas in the plenum.

In at least one embodiment, the housing comprises a cylindrical outer sleeve and a substantially planar plate at the distal end of the sleeve, wherein the outlet is provided in said plate.

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In at least one embodiment, the nozzle further comprises a gas source, for delivering a gas to the plenum.

30 In at least one embodiment, the gas is nitrogen.

The present invention further provides a source assembly comprising:

a source housing, configured to receive at least the distal end of a capillary therein; and

a nozzle according to the invention, received in the source housing, configured such that, in use, the curtain of heated gas is substantially aligned
5 with the longitudinal axis of the capillary.

In at least one embodiment, the source assembly further comprises a capillary, wherein the distal end of the capillary is received in the source assembly and the curtain of heated gas is substantially aligned with the longitudinal axis of
10 the capillary.

Embodiments of the present invention will now be described, by way of non-limiting example only, with reference to the following figures in which:

15 Figure 1 illustrates a nozzle embodying the present invention;

Figure 2a illustrates a source assembly including a nozzle embodying the present invention;

20 Figure 2b illustrates the detail C of Figure 2a;

Figures 3a to 3d illustrate various embodiments of the outlet of a nozzle embodying the present invention;

25 Figures 4a to 4c illustrate a nozzle embodying the present invention;

Figure 5 illustrates a cross-sectional view of a nozzle embodying the present invention; and

30 Figure 6 illustrates the positioning of the distal end of a capillary relative to the outlet of the nozzle in use.

Figure 1 illustrates a nozzle 1 embodying the present invention. The nozzle 1 is for directing a heated gas onto the distal end 51 of a capillary 50 arrangeable adjacent the nozzle 1, shown in Figure 6.

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The nozzle 1 comprises a housing 2 defining a plenum 3 for a heated gas.

The nozzle 1 further comprises an outlet 4 comprising at least one aperture 5. The outlet 4 is fluidly connected to the plenum 3. The outlet 4 is configured to
10 direct a curtain 6 of the heated gas onto the distal end 51 of the capillary 50. The curtain 6 of heated gas is substantially aligned with the longitudinal axis 52 of the capillary 50, which is shown in Figures 6 and 2b.

The curtain 6 of heated gas has a plane and the longitudinal axis of the
15 capillary 50 is substantially aligned with that plane. Although absolute alignment may be preferable, substantial alignment of the curtain 6 of heated gas with the longitudinal axis 52 of the capillary 50 may be acceptable. For example, the angle of the longitudinal axis 52 of the capillary 50 relative to the plane of the curtain 6 of heated gas may be within a tolerable range. The
20 range may be between -10° to $+10^{\circ}$, -5° to $+5^{\circ}$, -2.5° to $+2.5^{\circ}$, or between -1° and $+1^{\circ}$.

The curtain 6 of heated gas has a width and a length. The length is longer than the width. Accordingly, the curtain 6 of heated gas directed from the
25 outlet 4 of a nozzle 1 embodying the present invention is effectively "elongated". This contrasts to the circular beam of hot gas provided by a single circular aperture of an arrangement falling outside of the claimed invention (discussed above). A benefit of an outlet 4 of the claimed invention providing a curtain 6 of heated gas is that it may provide more effective
30 coverage of the distal end 51 of the capillary 50, ensuring that more of a sample provided on the distal end 51 of the capillary 50 is effectively heated

(as compared to a single circular aperture of an arrangement falling outside of the scope of the claimed invention). Without a curtain of heated gas being provided by embodiments of the present invention, there may not be effective coverage of the heated gas on the distal end 51 of the capillary 50. Some of the sample may not then be heated directly by the heated gas stream, but instead through latent heat conduction in the capillary, sample or any coating which may be on the capillary tip. This may skew the measurements obtained.

The width of the curtain 6 may be configured so as to be substantially equal to the diameter/width of the capillary 50, and the length of the curtain 6 may be configured to be substantially equal to the length of the distal end 51 of the capillary 50 provisioned to retain sample.

Figures 3a to 3d illustrate various arrangements of an outlet 4 of a nozzle 1 embodying the present invention.

In Figure 3a, the outlet 4 comprises three apertures 5. Each of the apertures 5 is substantially circular. The apertures 5 are arranged in a line and evenly distributed therealong. This is not essential. In alternative embodiments, not shown, each of the apertures 5 may be of a different size and/or shape. The middle of the three apertures 5 may be closer to one of the outer apertures 5 than to the other.

The outlet 4 in Figure 3b comprises four circular apertures 5, all of substantially the same size, and linearly distributed. As with the arrangement in Figure 3a, the apertures 5 may alternatively be of different size and/or shape, and may be distributed differently to that shown in Figure 3a.

In other embodiments of the present invention, there may be provided two circular apertures 5. In other embodiments, there may be provided five, six, seven, eight or more apertures 5.

In Figure 3b, an aperture 5 at one end of the series of apertures 5 is substantially aligned with the central axis of the nozzle 1. This is not essential. In the arrangement shown in Figure 3a, the series of apertures 5 extends
5 across the central axis of the nozzle 1. The outlet 4 of a nozzle 1 embodying the present invention may be configured such that the curtain 6 of heated gas is offset from the central axis of the nozzle 1, or it may be centrally aligned.

Figure 3c illustrates an outlet 4 of another nozzle 1 embodying the present
10 invention, comprising a single aperture 5. The aperture 5 is non-circular and elongated. As will be noted from Figure 3c, the length of the aperture 5 is greater than the width. Consequently, the outlet of the arrangement shown in Figure 3c produces a curtain 6 of heated gas. This is in contrast to the circular beam of heated gas that would be produced by a single circular aperture, such
15 as discussed above.

Figure 3d shows an outlet 4 of a nozzle 1 according to another embodiment of the present invention. In this embodiment, the outlet 4 comprises a plurality of apertures 5 (four in this example), and each of the apertures 5 is elongated.
20 The direction of elongation is substantially perpendicular to the plane of the curtain 6 of heated gas produced by the outlet. It will be appreciated that with the configuration of the outlet 4 illustrated in Figure 3d, the elongate apertures 5 increase the width of the curtain 6 as compared to an arrangement comprising four circular apertures 5.

25 In other embodiments falling within the scope of the claims, the apertures 5 may take different forms to those illustrated and/or a combination of those illustrated in figures 3a to 3d. For example, the outlet 4 may comprise a plurality of apertures 5, wherein at least one of the apertures 5 is circular and
30 at least one of the apertures 5 is elongate. In an embodiment comprising a

plurality of elongate apertures 5, the direction of elongation of one aperture 5 may differ to the direction of elongation of another aperture 5 of that outlet 4.

5 In at least one embodiment, the total surface area of the aperture(s) 5 of an outlet 4 of a nozzle 1 embodying the present invention may be in the range 8 to 8.5 mm². In one embodiment, the total surface area may be within the range 8.1 to 8.4 mm². In another embodiment, the range may be between 8.2 to 8.3 mm². In another embodiment, the total surface area may be 8.245 mm².

10 In embodiments of the present invention, the nozzle 1 may be configured so as to deliver heated gas through the outlet 4 at a flow rate of between 1 and 4 litres per minute. In another embodiment, the flow rate may be between 2 and 3 litres per minute. In another embodiment, the flow rate may be between 2.3 and 2.7 litres per minute. In another embodiment, the flow rate may be
15 substantially 2.5 litres per minute. In another embodiment, the flow rate may be substantially 4 litres per minute.

As shown in Figure 5, the nozzle 1 may further comprise a heater arrangement 7, for heating gas in the plenum 3. The nozzle 1 may further comprise a gas
20 source 8. The gas is preferably nitrogen. The gas source 8 may be configured so as to introduce gas into the plenum 3 of the nozzle 1. The heater arrangement 7 is arranged so as to heat the gas introduced into the plenum 3. The heater arrangement 7 may be arranged in the path of the gas entering the plenum 3 from the gas source 8, so as to effectively heat the gas
25 using the heater arrangement 7.

The housing 2 comprises a cylindrical outer sleeve 9 and a substantially planar plate 10 at the distal end of the sleeve 9. The outlet 4 is provided in the plate 10. The cylindrical outer sleeve 9 and the plate 10 are hermetically sealed so
30 as to define the plenum 3 therein. Preferably, any heated gas within the plenum 3 may only leave the plenum 3 through the outlet 4 (assuming that the

gas source 8 communicating with the plenum 3 provides a positive pressure and/or a one-way valve).

5 The planar nature of the plate 10 is not essential. The reader will appreciate that it may take other forms, such as concave or convex. The cylindrical nature of the outer sleeve 9 is also not essential. In other forms it may be non-circular, such as having a square or rectangular cross-section. Correspondingly, the shape of the plate 10 may be non-circular, depending on the cross-sectional shape of the outer sleeve 9.

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As shown in Figures 4a to 4c, a nozzle 1 may further comprise a mounting flange 12, which may be substantially planar. The outer sleeve 9 of the nozzle 1 may be received in an aperture 13 in the mounting flange 12, as shown in Figure 4a.

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In at least one embodiment, as best illustrated in Figures 4a to 4c, the outer sleeve 9, rather than being entirely cylindrical, may comprise at least one flat section 13 extending along the longitudinal length of the sleeve 9. The aperture 13 in the mounting flange 12 may be similarly shaped, so as only to
20 receive the outer sleeve 9 of the nozzle 1 therein in a predetermined orientation. The mounting flange 12 further comprises an alignment feature 14, in the form of a pin, seen in Figures 4a to 4c.

In at least one embodiment, the source housing 20 comprises a corresponding
25 aperture which receives the locating pin 14. Accordingly, since the angular alignment of the locating pin 14 is set in relation to the outlet 4 of the nozzle 1, by virtue of the flat portion 13 on the outer sleeve 9, the position of the outlet 4 of the nozzle 1 is consequently fixed relative to the source housing 20. Accordingly, as shown in Figure 2b, the locating pin 14 ensures that the nozzle
30 1 is inserted into the source housing 20 in a repeatable fashion such that the

curtain 6 of heated gas is substantially aligned with the longitudinal axis 52 of the capillary 50 in use.

In at least one embodiment, the nozzle 1 is configured such that it can be only
5 inserted into the source housing 20 in a single orientation.

A benefit of the provision of the flat portion 13 on the outer sleeve 9 is that it aids assembly and manufacture of the nozzle 1. During manufacture, the outer sleeve 9 may be inserted into the aperture 13 in the mounting flange 12 in a
10 particular orientation, and then the outer sleeve 9 can be secured to the mounting flange 12, for example by welding or otherwise.

As shown in Figure 5, the nozzle 1 may further comprise an inner sleeve 15 and an intermediate sleeve 16 within the plenum. The intermediate sleeve 16
15 is arranged between the outer 9 and inner 15 sleeves. The outer 9, intermediate 16 and inner 15 sleeves create a labyrinthine/tortuous path for the passage of gas from the gas source 8 to the outlet 4. In the embodiment shown in Figure 5, a heating element of the heater arrangement 7 is provided between the outer 9 and intermediate 16 sleeves. Gas may enter one end of a
20 first section 21, between the outer 9 and intermediate 16 sleeves, and pass through to the end of the first section 21. From there, the gas may enter a second section 22 between the intermediate 16 and inner 15 sleeves. As it does so, the gas passes through the heater arrangement 7 once more. In at least one embodiment, the intermediate sleeve 16 may be conductive and
25 promote heat transfer into the gas. Finally, the gas may pass into a third section 23, inside the inner sleeve 15, again passing through the heater arrangement 7, further heating the gas.

The heated gas then exits the inner sleeve 15 through the outlet 4. A benefit
30 of the provision of the inner 15 and intermediate 16 sleeves is that the path

over which the gas is in contact with the heater arrangement 7 is increased, thereby increasing the efficiency of the heater arrangement 7.

When used in this specification and claims, the terms "comprises" and
5 "comprising" and variations thereof mean that the specified features, steps or integers are included. The terms are not to be interpreted to exclude the presence of other features, steps or components.

The features disclosed in the foregoing description, or the following claims, or
10 the accompanying drawings, expressed in their specific forms or in terms of a means for performing the disclosed function, or a method or process for attaining the disclosed result, as appropriate, may, separately, or in any combination of such features, be utilised for realising the invention in diverse forms thereof.

15 Although certain example embodiments of the invention have been described, the scope of the appended claims is not intended to be limited solely to these embodiments. The claims are to be construed literally, purposively, and/or to encompass equivalents.

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