

Description**Process and apparatus for gravimetric test gas
production by means of re-weighing**

5 A test gas is understood to be an exactly mixed
precision gas mixture with a defined composition, that is
to say one which is known in terms of the type and amount
of the constituents. It often consists of a basic gas or
basic gas mixture, which forms the main constituent of
the test gas, and of one or more adjuvants, which are
10 used directly for the purpose of testing or calibration.

There is a need for test gases with different
adjuvants, whose type, number and concentration vary
greatly. The range of application of test gases extends
from the calibration and adjustment of measuring instru-
15 ments, process optimization and plant monitoring, via
research and development, to medicine.

In principle, test gases are produced by com-
bining defined amounts of various gas proportions. The
oldest production methods are manometric and volumetric.
20 The manometric process is based on the change in pressure
after adding the individual adjuvants or the basic gas.
In order to determine the mass concentrations exactly,
recalculation with the aid of phenomenological gas-state
equations is necessary. In the case of the volumetric
25 process, the volumes of the individual gas mixture
components, are determined, for example by means of flow
meters, and transferred into a gas container, referred to
below as a bottle. However, the mixing accuracy is
relatively low in both processes, so that subsequent gas
30 analysis for the precise determination of the mixture
composition has to be carried out.

With the availability of sufficiently accurate
balances, gravimetric test gas production has rapidly
become widespread and is generally preferred nowadays.
35 The principle is that the gas mixture components are
introduced one after another into the bottle, the

increase in mass being determined by weighing after each metering operation. This gives the direct relation of the weighed-in gases to the basic variable "kg" or "mol", and recalculation using state equations is superfluous. Since
5 mass determinations by means of weighing belong to the most precise physical measuring methods, it is possible for test gases of extremely high precision to be produced with this method.

10 In practice, gravimetric test gas production is carried out in such a way that an evacuated bottle is placed onto a balance, and the connection to the gas supply, gas metering and control system is produced via a gas feed, generally a capillary, which is intended to ensure the smallest possible influence on the weighing.
15 The rest of the procedure is distinguished according to whether operations are carried out with or without re-weighing.

In the case of production without re-weighing, first of all the gas feed necessary for the gas supply is
20 connected to the bottle and a first weighing is carried out. Then the first component is introduced in accordance with the calculated formulation of the test gas to be produced. The components are customarily introduced in the sequence of increasing concentration. After the
25 filling operation has been completed, the bottle, with the gas feed connected and first component introduced, is weighed again. The difference between the two weighings of the bottle before and after filling gives the mass of the first component introduced. These operations are then
30 repeated correspondingly for all the further test gas components.

However, in the case of the weighings with the gas feed connected, the measurement result is falsified on account of the tension in the gas feed influencing the
35 balance. The values ascertained in this way therefore generally cannot be used for verifying the composition. Instead, this is achieved by means of a concluding physical/chemical analysis against a test gas standard.

In order to eliminate this disadvantage, the process of gravimetric test gas production with re-weighing is used. In this case, the gas feed is separated from the bottle, and so-called re-weighing is carried out, before and after the introduction of each component. Since the balance is in each case completely disconnected from the gas supply it is only the bottle with the appropriate contents that is weighed, without disruptive external influences. It is thus possible for the masses of the individual components to be ascertained exactly from the values determined by re-weighing. In the case of this process, the verification of the composition is carried out exclusively using the ascertained masses. Concluding gas analytical tests can thus be dispensed with.

Gravimetric test gas production with re-weighing has hitherto been carried out manually and is therefore very work-intensive and time-consuming. Carrying out the process automatically has not been possible hitherto.

DE-C-37 39 950 discloses a process for filling bottles with gases automatically in accordance with a weight specification. For this purpose, a rotary head for opening and closing the bottle valve, and a filling head for introducing the gas, are arranged on the balance. During the filling of the gas bottle, gas is introduced into the bottle, and weighing is subsequently carried out, in several steps until the predefined final weight has been reached. However, the filling method described is not suitable for the production of precision gas mixtures, since a gas hose, a compressed-air hose and a power supply cable lead away from the balance, and influence the weighing.

The object of the present invention is therefore to provide a process with which gravimetric test gas production with re-weighing can be automated. The invention is also based on the object of providing an apparatus for carrying out this process.

As far as the process is concerned, this object is achieved by the features of Claim 1.

The process according to the invention permits the automatic production of a test gas by the gravimetric process with re-weighing. The bottle valve, which is to be actuated manually and is usually a valve provided with a hand wheel, has connected to it a further, controllable valve, which makes it possible to open and close the bottle automatically under control, without actuating the hand wheel. The connection of the gas feed to the bottle is produced by means of a controllable adjusting element, that is connected to the balance.

During test gas production, first of all the bottle is evacuated and then the controllable valve is closed. By contrast, the bottle valve can remain open during the entire production process. After a first weighing of the evacuated bottle, the connection between the gas feed and the bottle is made in a gas-tight manner, and thus the connection between the gas feed and the balance is also produced. After the controllable valve has been opened, the first component of the test gas is introduced. The amount introduced is monitored automatically by weighing the bottle and, after the required amount has been reached, the gas feed is interrupted and the valve is closed. The accuracy of the test-gas-component mass ascertained in this way and the deviation of this mass from the predefined value determine the preparative accuracy of the process. In order to verify the gas composition, re-weighing is carried out. For this purpose, according to the invention the seal between the gas feed and the valve is released by the adjusting element, the gas feed is separated from the balance and re-weighing is then carried out. These process steps are repeated until all the constituents of the test gas have been introduced and re-weighed in accordance with the predefined specification.

The controllable valve is advantageously connected directly to the bottle. However, it is also possible to fit, between the valve and the bottle, further elements which permit a gas flow, for example

30.04.96 - Gellner/(ed)

lines or measuring instruments, such as manometers or flow meters. The only essential factor is for the bottle to be sealed off in a gas-tight manner from the environment when the valve is closed.

5 It is also possible for further elements which permit a gas flow to be arranged between the gas feed and the controllable valve. The only important factor is for the connection to be releasable, that is to say interruptible, and for a gas-tight seal with respect to the
10 environment to be achieved in the connected state.

 The releasable connection between the gas feed and the controllable valve is expediently made or broken by an automatically actuated lifting or pulling cylinder. For example, it is possible to press the gas feed onto
15 the valve, which is provided with a seal, by means of a pneumatically or electrically actuated cylinder in order to make the connection, and to pull the gas feed back again in order to break the connection.

 The gas feed can also be pressed onto the seal by
20 other suitable means, such as, for example, an eccentric or a toggle lever. It is likewise possible to guide the gas feed by other means, for example a linearly moveable band that is provided with a holding device for the gas feed, in such a way that the connection between the gas
25 feed and the controllable valve is made or broken.

 The gas feed is preferably designed to be flexible such that it can be advanced up to the corresponding mating piece in order to be connected to the bottle, and can be removed some way from the said mating piece at the
30 time of separation. On the other hand, however, it is expedient to design the gas feed in such a way that, when it is connected to the balance, it does not exert any additional weight on the balance. For this purpose, a flexible, but self-supporting design of the gas feed, for
35 example as a thin tube, has proven to be suitable. It is also possible to compensate for the weight of the gas feed, for example, via a deflecting means and a counterweight. In addition, the weight of the gas feed can also

be calculated from the weighing results by the control unit.

For exact re-weighing, the gas feed must be separated both from the bottle and from the balance. In the case of a self-supporting design of the gas feed, or in the case of a device for compensating for the weight of the gas feed, the release of the connection between the gas feed and the bottle is advantageously associated with the disconnection of the gas feed from the balance.

It is also advantageous to separate the gas feed from the balance, or to connect it to the balance, by a controllable lifting or pulling cylinder which is not connected to the balance. In this case, the weight of the gas feed, when the latter is separated from the balance, is borne by this cylinder.

During the re-weighing of the bottle, all the connections between the balance and the environment have to be released in order to ensure exact weighing. In addition to the gas feed, these are, in particular, lines which are used for the power supply and control of the controllable valve and to the controllable adjusting element. It is therefore advantageous if the energy to actuate the controllable valve and the adjusting element is supplied by at least one self-contained power supply that is located on the balance. This dispenses with otherwise necessary supply lines, which may have an influence on the re-weighing. For example, an electrical battery which supplies the necessary energy for all the valves and devices to be actuated can be provided on the balance.

Depending on the design of the elements to be controlled, other energy stores or combinations of different energy stores may be provided in addition to an electrical energy store. For example, the regulation and control of all the elements to be actuated can advantageously be carried out electrically, whereas the actual actuation is carried out pneumatically. Accordingly, in this case, in addition to an electrical battery, use will

30.04.96 - Gellner/(ed)

also be made of a pneumatic energy store, for example a gas cylinder under a positive pressure.

However, a self-contained power supply for all the elements that are located on the balance and are to be actuated, such as, for example, the controllable valve, the adjusting element or further adjustable valves and devices, is not absolutely necessary. It is likewise possible to supply the necessary energy for actuating the elements from outside as required. For example, such electrically adjustable elements can be designed to be self-maintaining, so that they remain in the adjusted state after the electrical energy supply has been disconnected. This makes it possible for the electrical supply lines to be removed from the balance again after the elements to be controlled have been appropriately actuated. Any negative influence on the weighing, in particular during the re-weighing, is thus likewise avoided. Such electrical supply lines can be connected to the devices located on the balance via so-called blade contacts, for example.

In order to control the overall test gas production, a control unit is provided which has the task of carrying out all the necessary control actions during the test gas production in accordance with a predefined formulation and of coordinating these actions with one another. This includes opening and closing the valve arranged between the gas feed and the bottle and the valves of the gas supply device, which is generally located alongside the balance, as well as controlling the adjusting element. In addition, the control unit expediently monitors and regulates the pressure relationships in the gas feed lines and, in the case of pneumatic control, also those in the control lines, as well as all the weighings. Numerous further monitoring and regulating tasks, as well as the arithmetic further processing of weighing results, can likewise be undertaken preferably by the control unit as well, so that a completely automatic sequence is achieved.

The entire control unit may be arranged either on the balance or alongside the balance. Furthermore, it is also possible to fit only specific parts of the control unit on the balance.

5 It is advantageous not to arrange the control unit on the balance. Since the control unit has to control both elements on the balance and those alongside the balance, a device for transmitting the control signals from and to the balance is needed in any case, 10 irrespective of the arrangement of the control unit. The arrangement of the control unit alongside the balance has the advantage that the total weight which the balance has to bear is lower, which means that a balance with a higher weighing accuracy can be chosen. Furthermore, an 15 arrangement of this type permits manual intervention, if required, in the automatically progressing production process, without the weighings being influenced as a result.

 The signal transmission between the control unit 20 located alongside the balance and the elements which are to be controlled and are located on the balance, or between the control unit located on the balance and the elements which are to be controlled and are not located on the balance, is expediently carried out without 25 contact, in order to avoid any influence on the weighing. For this purpose, optical transmission using so-called optocouplers is expedient. The light transmitters used are light-emitting diodes (LED), which for the most part radiate infra-red light or visible light. The light 30 receivers used are photodiodes, phototransistors or photo-darlington transistors.

 In order to prevent the penetration of atmospheric air, and in particular of moisture, into the gas feed, it has proven to be advantageous to flush the gas 35 feed with an inert gas when it is separated from the bottle. After the gas feed has been separated from the controllable valve, it is advantageously removed from the mating piece which is to be connected to it only to such

a small extent that, although, on the one hand, the gas feed is completely disconnected from the balance, on the other hand the mating piece is also swept by the inert gas stream carried by the gas feed. This therefore also
5 prevents the penetration of air into the gas line region as far as the controllable valve.

In addition to the process for gravimetric test gas production with re-weighing, the invention also relates to an apparatus for carrying out this process,
10 having a balance for weighing the gas container with contents and a gas feed for feeding the test gas components.

According to the invention, provision is made for a balance for weighing a gas container with and without
15 contents, a gas feed for introducing the test gas components into the gas container, a controllable valve that is connected to the gas container, and a connection which is provided between the gas feed and the controllable valve and can be released and made by a controllable
20 adjusting element that is connected to the balance.

It is expedient for the adjusting element that is connected to the balance to be constructed as a controllable lifting or pulling cylinder.

A self-contained power supply that is located on
25 the balance is advantageous. Use is expediently made of an electrical battery for the supply of electrical energy, and of a compressed-gas container for the supply of pneumatic energy.

In the event of using pneumatic control of
30 elements to be controlled, it is important that no control gas escapes, in order not to falsify the weighing. In this case, therefore, a low-pressure gas container is advantageously arranged on the balance, this container intercepting control gases exhausted by
35 elements located on the balance, for example valves.

The proposal according to the invention has numerous advantages in comparison with the prior art. As a result of the complete automation of the gravimetric

test gas production, the use of operating personnel is reduced in a cost-effective manner. In addition, the test gas production is accelerated, so that significantly more bottles can be filled with lower operating outlay. The
5 gravimetric production with re-weighing allows final gas analytical tests for verifying the composition to be dispensed with. Random gas analytical sample tests are carried out occasionally merely in order to monitor satisfactory functioning of the process.

10 Until now, it was necessary for the composition of the test gases to be ascertained, after their production, by means of a physical/chemical analysis against a test gas standard. Test gas standards of this type are generally produced by gravimetric production by means of
15 re-weighing. In the case of previous verification, the accuracy of the test gas composition is therefore determined by the accuracy of the analysis and the accuracy of the standard. In the case of the inventive method, the accuracy of the composition is determined by the re-weighing. The test gases produced therefore correspond in
20 qualitative terms to the test gas standards produced hitherto. The proposed process thus also has qualitative advantages, in addition to the quantitative advantages, in comparison with previously known processes.

25 The proposal according to the invention is to be explained below by way of example with reference to the schematic drawing.

 The single figure shows an apparatus according to the invention for gravimetric test gas production.

30 Located on the balance 1 is the bottle 2 to be filled, which is connected to the moveable connector 6 in terms of a gas path via the controllable, pneumatically actuated valve 3, the flexible line 4 and the coupling 5. Fastened to the connector 6 is a capillary 7, which
35 allows the lowest possible influence on the weighing and via the other end of which the filling gases are fed in. Alongside the balance, indicated merely by dashed lines in the drawing, are the gas supply, a gas metering

30.04.96 - Gellner/(ed)

system, an overfilling safeguard and a PC-supported control system 8.

5 The connector 6 is connected in a manner suitable for the filling pressure to the bottle 2 to be filled, by the pneumatically actuated lifting cylinder 9 closing the coupling 5. The coupling 5 comprises a chambered O-ring, which is pressed against a mating piece. The mass contact between the connector 6 and the balance 1 is eliminated by the connector 6 being lifted a few millimetres by the
10 pulling cylinder 10, which is not connected to the balance, when the coupling 5 is open. The capillary 7 is constructed as a thin metal tube, so that it is sufficiently flexible to be connected to the line 4 and released from it. On the other hand, however, the capil-
15 lary 7 is also designed to be self-supporting, so that, when the coupling 5 is made, the said capillary does not exert any additional pressure on the balance 1. By contrast, when the capillary 7 is connected, effects on the weighings as a result of stresses occurring in the
20 capillary 7 are unavoidable.

Also located on the balance 1 is a self-contained power supply, which is formed by an electrical battery 11 and a high-pressure gas cylinder 12. The high-pressure gas cylinder 12 is filled with a suitable gas, for example
25 nitrogen or compressed air, via the coupling 13 and the non-return valve 14. The pressure in the high-pressure gas cylinder 12 can be monitored by means of the manometer 15. The operating pressure for the valves 3 and 22 and for the lifting and pulling cylinders 9 and 10, respectively, is
30 set using a pressure reducer 16. The control valves 17, 18 and 19 are designed as electrically actuated 2-way valves. The control signals for controlling these valves are transmitted optically from the signal generator 20 to the switching amplifier 21, which switches the valves 17, 18,
35 19 in accordance with a program. The signal generator 20 has various light-emitting diodes, emitting infra-red light and visible light, as light transmitters. Photo-diodes are integrated in the switching amplifier 21 as

light receivers. The switching amplifier 21 is electrically connected to the battery 11. The low-pressure container 23 intercepts the control air exhausted by the valves 18 and 19 and thus prevents any reduction in weight on the balance 1. At a time that is favourable for the weighing, the control air exhausted is deliberately discharged to the atmosphere via the valve 22. The valves 3 and 22 are designed in such a way that they are closed when no pressure is applied. The lifting cylinder 9 is relieved of load when no pressure is applied.

The process according to the invention is to be explained in more detail below by way of example with reference to the production of a binary test gas mixture.

The evacuated bottle 2 to be filled is connected to the valve 3, the coupling 5 is closed by means of the lifting cylinder 9 and the valve 3 is opened. The gas path from the capillary 7 to the bottle valve 24 is evacuated and thereby tested for tightness. The bottle valve 24 is then opened.

All the following process steps proceed automatically under control, without any manual intervention being needed. The valve 3 is closed, the control air being expanded into the atmosphere via the valve 22 until pressure equalization takes place. The capillary 7, the flexible line 4 and the connector 6 are subsequently brought to atmospheric pressure or a slight positive pressure, via the capillary 7, using inert gas. The coupling 5 is released, the connector 6 is lifted by the cylinder 10 and hence separated from the balance 1. In order to avoid the penetration of atmospheric air and, above all, moisture, the connector 6 is continuously flushed with inert gas via the capillary 7. Because of the slight parallel displacement of the connector 6 of only a few millimetres, the balance-side part of the coupling 5 is also swept by inert gas. The first weighing takes place in this state.

The coupling 5 is closed using the cylinder 9. In addition, the valve 22 is closed, in order to prevent

control air from escaping. Via the capillary 7, the gas path from the gas feed 6, 7, via the flexible line 4, as far as the valve 3, is evacuated. The valve 3 is opened, and a second weighing, now with the gas feed connected, is carried out. According to the formulation, the appropriate amount of the first component of the test gas is introduced. After the desired amount has been introduced, the valve 3 is closed, the control air being intercepted in the low-pressure container 23. The capillary 7, the connector 6 and the line 4 are relieved of pressure and evacuated. Weighing is then carried out in order to monitor the amount introduced. If the filling weight has not yet been reached, the first test gas component is led in once more via the gas feed 6, 7, the valve 3 is opened and the missing amount is introduced. The valve 3 is then closed again, the gas path 4, 6, 7 is relieved of pressure and evacuated, and a monitoring weighing is carried out. If the predefined end weight has been reached, the gas feed 6, 7 and the line 4 are flooded with inert gas as far as the valve 3, and the coupling 5 is opened. The control air is once more intercepted in the container 23. The introduced amount of the first component can be determined exactly from the following re-weighing. The difference between the two weighings without the capillary 7 connected differs from the difference between the weighings with the capillary 7 connected on account of stresses in the capillary 7. Only the first is suitable to verify the mass of the component introduced. The control air is then blown out again via the valve 22. Renewed weighing supplies a lower result and thus confirms that the mass of the control air cannot be neglected.

The coupling 5 is closed and, following the evacuation of the gas path 4, 6, 7, weighing is carried out again. Introduction of the second component is carried out in a manner similar to the described process for introducing the first component. The mass of the second component introduced is obtained precisely from the final re-weighing.

Patent Claims

1. Process for gravimetric test gas production by means of re-weighing, the following process steps being carried out automatically one after another:
- 5 a. weighing an evacuated gas container (2) on a balance (1),
- b. connecting a gas feed (7) to a controllable valve (3), that is connected to the gas container (2), by means of a controllable adjusting element (9)
- 10 that is connected to the balance (1),
- c. automatically opening the controllable valve (3),
- d. introducing the first test gas component, the amount introduced being monitored automatically by weighing the gas container (2) and, after the
- 15 required amount has been reached, the controllable valve (3) being closed,
- e. releasing the connection between the gas feed (7) and the controllable valve (3) by means of the adjusting element (9), and automatically separating the gas feed (7) from the balance (1),
- 20 f. re-weighing the gas container (2),
- g. repeating the steps b. to f. for each further test gas component to be introduced into the gas container (2).
- 25 2. Process according to Claim 1, the connection between the gas feed (7) and the controllable valve (3) being made or broken by an automatically actuated lifting or pulling cylinder (9).
3. Process according to either of Claims 1 and 2,
- 30 the gas feed (7) being connected to the balance (1), or separated from it, by a controllable lifting or pulling cylinder (10) which is not connected to the balance (1).
4. Process according to one of Claims 1 to 3, the energy to actuate the valve (3) and the adjusting element
- 35 (9) being supplied by at least one self-contained power supply that is located on the balance (1).
5. Process according to one of Claims 1 to 4, the



valve (3) and the adjusting element (9) being controlled by a control unit (8) which is not located on the balance (1).

5 6. Process according to one of Claims 1 to 5, signals being transmitted from the control unit (8) to the balance (1) without contact.

7. Process according to one of Claims 1 to 6, the gas feed (7) being flushed with an inert gas when it is separated from the gas container (2).

10 8. Apparatus for gravimetric test gas production by means of re-weighing, having a balance (1) for weighing a gas container (2) with and without contents, a gas feed (7) for introducing the test gas components into the gas container (2), a controllable valve (3) that is connected
15 to the gas container (2), and a connection which is provided between the gas feed (7) and the controllable valve (3) and can be released and made by a controllable adjusting element (9) that is connected to the balance (1).

20 9. Apparatus according to Claim 8, having an adjusting element (9) constructed as a lifting or pulling cylinder for making and breaking the connection between the gas feed (7) and the valve (3).

25 10. Apparatus according to either of Claims 8 and 9, having a controllable lifting or pulling cylinder (10), which is not connected to the balance (1), for separating the gas feed (7) from the balance (1).

30 11. Apparatus according to one of Claims 8 to 10, having a self-contained power supply arranged on the balance (1).

12. Apparatus according to Claim 11, having an electrical battery (11) arranged on the balance (1).

13. Apparatus according to Claim 11, having a compressed-gas container (12) arranged on the balance (1).

35 14. Apparatus according to Claim 11, having a low-pressure container (23) arranged on the balance (1).

15. Apparatus according to one of Claims 8 to 14, having a control unit (8), which is not located on the



G 96/055 = G547

- 16 -

06.05.96 - Gellner/(ed)

balance (1), for controlling the valve (3) and the
controllable adjusting unit (9).



* C L M S *