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(54) Benævnelse: **Analysesystem til analyse af vand og spildevand**

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US-A- 4 074 973

Analysis system for analyzing water and wastewater

Description

The invention relates to an analysis system for analysing water and waste water, comprising an analysis apparatus with an apparatus housing and an injection port for introducing a sample into the apparatus, and an injection syringe.

Such analysis systems are known per se, and are also produced and marketed by the Applicant.

US 3 560 156 A discloses an analysis system having the features of the preamble of Claim 1.

The documents US 4 074 973 A, EP 2 662 690 A1 and DE 20 2061 456 A1 each disclose an analysis system having a cylindrical reaction vessel and having controllable means for stabilizing the pressure and throughflow rate of a carrier gas, by way of which carrier gas a sample is supplied to the reaction vessel.

The object of the invention is to provide an improved analysis system of this type which in particular can provide reliably precise and precisely reproducible analysis results.

This object is achieved with an analysis apparatus having the features of Claim 1.

According to the invention, the analysis apparatus has an injection port which is connected to a cylindrical reaction vessel for thermal digestion of the substance to be analysed and which has guide means for guiding an injection syringe into a predetermined injection position.

The invention involves the notion of a carrier gas supply being connected to the reaction vessel at the input side, which carrier gas supply has controllable means for determining the pressure and throughflow rate of a carrier gas to be supplied to

the reaction vessel. Besides a sample supply which is precisely dimensioned in terms of the quantity thereof and is precisely predetermined in terms of the location and direction of the ejection of the sample into the reaction vessel, for exact and reproducible analysis results, accurate and reproducible control of the carrier gas flow for transporting the sample to a detection device is also of great importance. It has been found that simple throughflow limitation, for example by means of a reducing valve, is not sufficient to meet the accuracy requirements of an analysis system which is suitable for laboratory use.

For this purpose, it is in particular provided that the carrier gas supply comprises a first branch (partial flow) for supplying nitrogen and a second branch (partial flow) for supplying oxygen, wherein, in the first branch, a first pressure regulator and throughflow regulator for pressure regulation and throughflow regulation of supplied nitrogen and, in the second branch, a second pressure regulator and throughflow regulator for pressure regulation and throughflow regulation of oxygen to be mixed with the nitrogen are provided. In this case, it is in particular the case that the first and second pressure and throughflow regulators are designed and matched in such a way that, when the oxygen is mixed with the nitrogen, a dilution by a factor of at least 10, particularly at least 100, more particularly of approximately 1000, is able to be realized. It goes without saying that, for realizing such low dilutions with sufficient accuracy, precision throughflow regulators are to be used.

According to the aforementioned invention, this is based on the consideration of permitting, in dependence on particular features of the analysis results, post-processing or influencing of the specific evaluation mode. The inventors have established that, with the determination of relevant constituents of water or waste water samples, the specific sample constitution as well as characteristics of the procedure of the analysis can considerably influence the signal form of a time-dependent detection signal, and that this indicates the expediency of “manual” influencing of the evaluation process.

Since, specifically, the temporal signal profile for oxygen concentration, and furthermore carbon dioxide, nitrogen or phosphorus concentrations, measured in a time-dependent manner, of the water or waste water differ greatly, useful and comparable analysis results can be achieved only conditionally with a fixed integration time. It is therefore preferably provided that the post-processing device is configured for manual readjustment of an integration time for integration of the concentration values detected in each case in a time-dependent manner.

Advantages and features of the invention will further emerge from the following short description of an embodiment with reference to the Figures, in which:
Figure 1 is a schematic illustration of significant apparatus components of an analysis apparatus according to an embodiment,
Figure 2 is a perspective illustration of that analysis apparatus,
Figure 3 is a schematic longitudinal sectioned illustration of an injection syringe with an injection needle introduced into the injection port according to an exemplary embodiment of the analysis system, and
Figures 4a and 4b are graphs for illustrating an aspect of the invention.

Figures 1 and 2 show schematically significant components of an analysis apparatus 1 for determining the COD (chemical oxygen demand) of water or waste water. The basic structure of such apparatuses and the operation thereof are known to the person skilled in the art and are therefore not described here in greater detail, especially since it is not required for the understanding of the present invention.

The analysis apparatus 1 comprises a thermal reaction vessel or a furnace EB, in which by means of an injection syringe MM via an injection port P which is arranged on the furnace upper side a water sample can be injected and in which this sample is thermally processed. There is supplied to the furnace via a non-return valve RM1 a carrier gas flow which is composed of air and nitrogen. The carrier gas flow is controlled by means of an air pressure regulator KH1, a nitrogen pressure regulator KH2, an air throughflow regulator KH4 and a nitrogen throughflow regulator KH5 and filtered by means of a first and second fine filter HQ1, HQ2 at the input side of the furnace. At the input side of the furnace, an air

pressure display BP1 and a nitrogen pressure display BP2 are also provided.

At the output side of the furnace, the gas flow first reaches a condensate vessel CM1 and the non-condensed portion subsequently passes through a quartz wool filter HQ3 and an acid trap HS1 before it reaches the acid detector B1, which finally outputs a (an electrical) measurement value to an adjustable evaluation device A, in which in particular an integration time for integration of a time-dependent-detected oxygen detection signal is provided, see below for further details. The analysis apparatus according to the invention also comprises a carbon dioxide and/or a nitrogen and/or a phosphorus detector, which is however not described in more detail here.

Figure 2 is a perspective view of the analysis apparatus 1 in a state in which the apparatus housing 1' is partially open and a portion of the apparatus components has been removed from the housing. The apparatus housing is substantially in the form of a square prism and in the apparatus front 1A' there is provided an opening 1B' which is intended to be closed by means of a perforated door 3 which is mounted on the left lateral edge of the apparatus front.

A carriage 5 which is adapted in terms of its dimensions to the opening 1B' and on which the furnace EB, the condensate vessel CM1, the quartz wool filter HQ3 and the acid trap HS1 are arranged, can be pulled so far out of the housing that the components mentioned become freely accessible. In the retracted state of the carriage 5, the apparatus housing 1' is closed with the door 3.

At the right side of the apparatus front 1A’ is an operation field 1C' on which a plurality of operation and display elements are arranged, including a temperature display/control TC and the adjustment regulator KH1 and KH2 for the air or nitrogen pressure and the associated display elements BP1 and BP1.

Located at the apparatus upper side 1D' is the injection port P whose structure and dimensions are adapted to those of the injection syringe MM shown in Figure 1 and which communicates in the apparatus inner space with a corresponding injection

valve EB1 of the furnace EB when the furnace is in the normal position for use thereof inside the apparatus housing.

Figure 3 is a schematic longitudinal section of the basic structure and the mutually adapted geometric configurations of the injection syringe MM and of the injection port P of the furnace EB of the analysis system. The injection port P comprises a guide sleeve P1 which is constructed in a substantially cylindrical manner in the longitudinal extent thereof and whose diameter and length are adapted to the corresponding dimensions of an injection needle MM1 of the injection syringe MM and whose longitudinal axis coincides with a longitudinal axis LA1 of the furnace EB which is constructed to be cylindrical in its basic form. At the upper side of the injection port P there is provided a hole P2 with an increased diameter whose dimensions are adapted to those of a needle hub MM2 of the injection syringe and whose lower end face acts as a stop for depth limitation when the injection syringe is introduced. A precisely predetermined position of the needle end which is cut at right-angles relative to the longitudinal needle axis LA2 of the injection syringe in the furnace EB and consequently a precisely predetermined injection location are ensured with this stop.

In the injection reservoir MM3, there is supported so as to be able to be longitudinally displaced a syringe piston MM4 whose free end is constructed in conventional manner for manually drawing up a sample. At the upper end of the injection reservoir there is embedded therein a pressure spring MM5 whose upper end is supported against the upper end wall of the injection reservoir and whose lower end acts on the end of the syringe piston MM4. After the syringe has been drawn up, using a locking lever MM6 the syringe piston is locked with a tensioned spring MM5. After the locking MM6 has been released, the syringe piston MM4 is pressed downwards by the force of the pressure spring MM5 and the sample contained in the injection reservoir MM3 is injected in a predetermined time range or with a predetermined ejection speed into the furnace.

This ejection of the predetermined sample quantity at a precisely predetermined speed or in a precisely defined time range is just as important for reproducible

analysis results as the precise injection position and direction, which are ensured by the specific configuration of the injection needle and the injection port.

Figures 4a and 4b illustrate the effect of an adjustable integration time of the post-processing device A for processing an oxygen measurement signal which has been recorded in a time-dependent manner. Figure 4a shows in this instance three different curve shapes with (according to the prior art) fixedly adjusted integration time t_i. It can be seen that in this instance only the integration of the measurement signal S_1 illustrated with the solid line leads to a correct result, whilst the adjusted integration time for the measurement signals S_2 and S_3 is clearly too short and significant signal portions are not detected. Remedial action is provided in this instance by the adjustment of a longer integration time $t_{i2,3}$ for the signals S_2 and S_3, as shown in Figure 4b, which the operator of the analysis system can carry out in accordance with the fixed signal curve shape on the post-processing device A.

PATENTKRAV

1. Analyseapparat til analyse af vand og spildevand, med en cylindrisk reaktionsbeholder for termisk disintegration af stoffet, der skal analyseres, hvorved der til indgangssiden af reaktionsbeholderen er forbundet en injektionsport, der omfatter føringsmidler til føring af en injektionssprøjte for stoffet, som skal analyseres, samt en bæregasforsyning, der omfatter styrbare midler til bestemmelse af tryk og strømningshastighed for en bæregas, som skal leveres til reaktionsbeholderen,

hvorved bæregasforsyningen omfatter en første gren for tilførsel af nitrogen og en anden gren for tilførsel af oxygen,

hvorved der i den første gren er tilvejebragt en første trykregulator og flowregulator til trykregulering henholdsvis flowregulering for tilført nitrogen og i den anden gren en anden trykregulator og flowregulator til trykregulering henholdsvis flowregulering for oxygen, der skal blandes med nitrogenet, og

hvorved analyseapparatet ydermere omfatter et detekterings- og evalueringsapparat til detektering af en forudbestemt bestanddel af stoffet, som skal analyseres,

kendetegnet ved, at

detekterings- og evalueringsapparatet ydermere omfatter en indstillelig efterbearbejdningsindretning til manuelt styret efterbearbejdning af detekteringsresultatet i afhængighed af en bestemt signalkurveform for et tids-afhængigt registreret målesignal for stoffet, der skal analyseres, og at

detekteringsindretningen omfatter en oxygendetektor og ydermere en detektor for carbondioxid og/eller nitrogen og/eller phosphor med henblik på tidsafhængig detektering af oxygenkoncentration og ydermere af koncentrationen af carbondioxid, nitrogen og/eller phosphor i bæregasstrømmen, som forlader reaktionsbeholderen, og

efterbearbejdningsindretningen er konfigureret til manuel efterindstilling af en integrationstid med henblik på integrationen af de respektive detekterede tidsafhængige koncentrationsværdier af oxygen og carbondioxid og/eller phosphor.

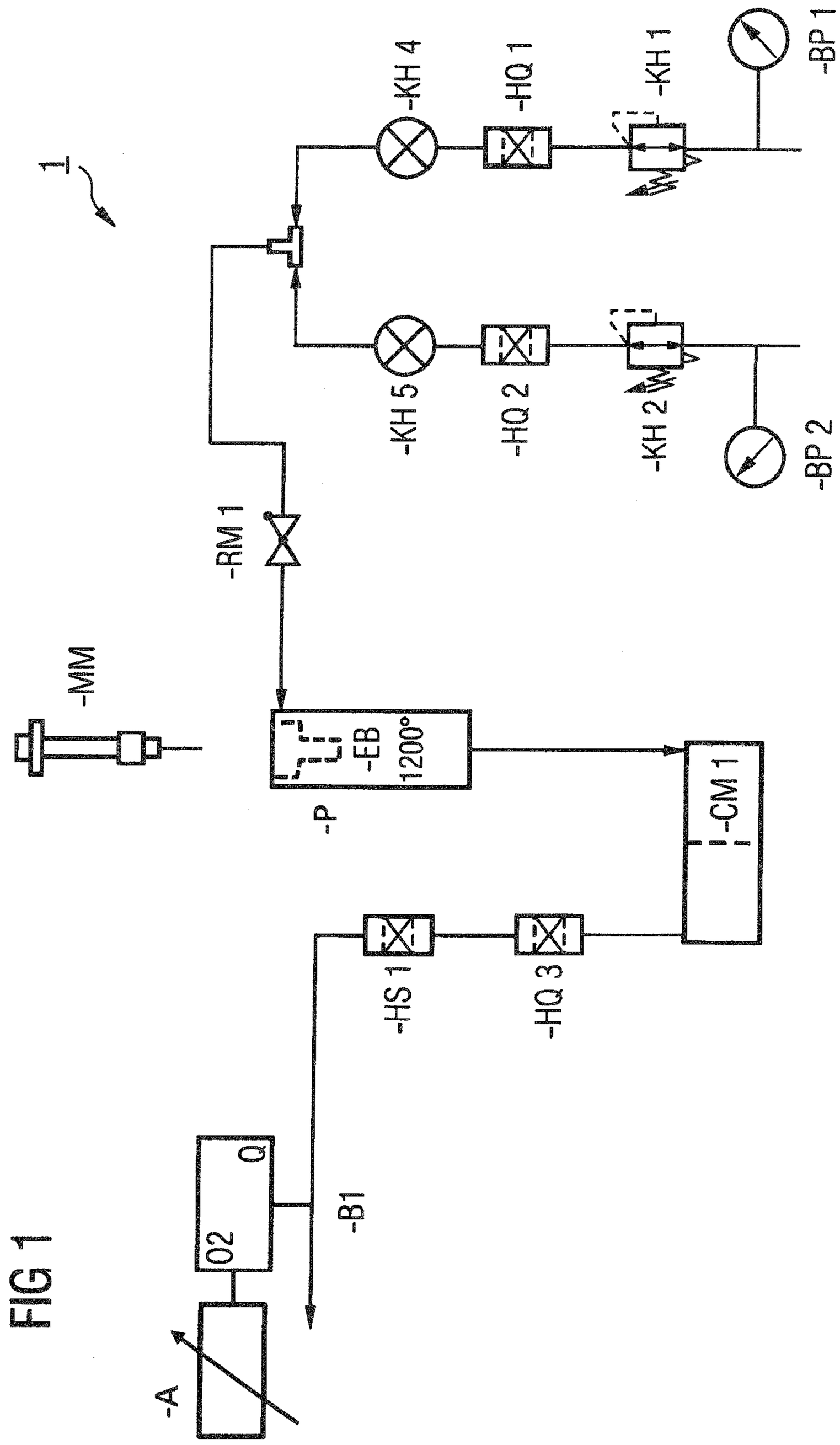
-KH 4
-HQ 1
-KH 1
-BP 1
-KH 5
-HQ 2
-KH 2
-BP 2
-RM 1
-MM
-EB
1200°
-P
-CM 1
-HS 1
-HQ 3
-B1
Q
02
-A

FIG 1

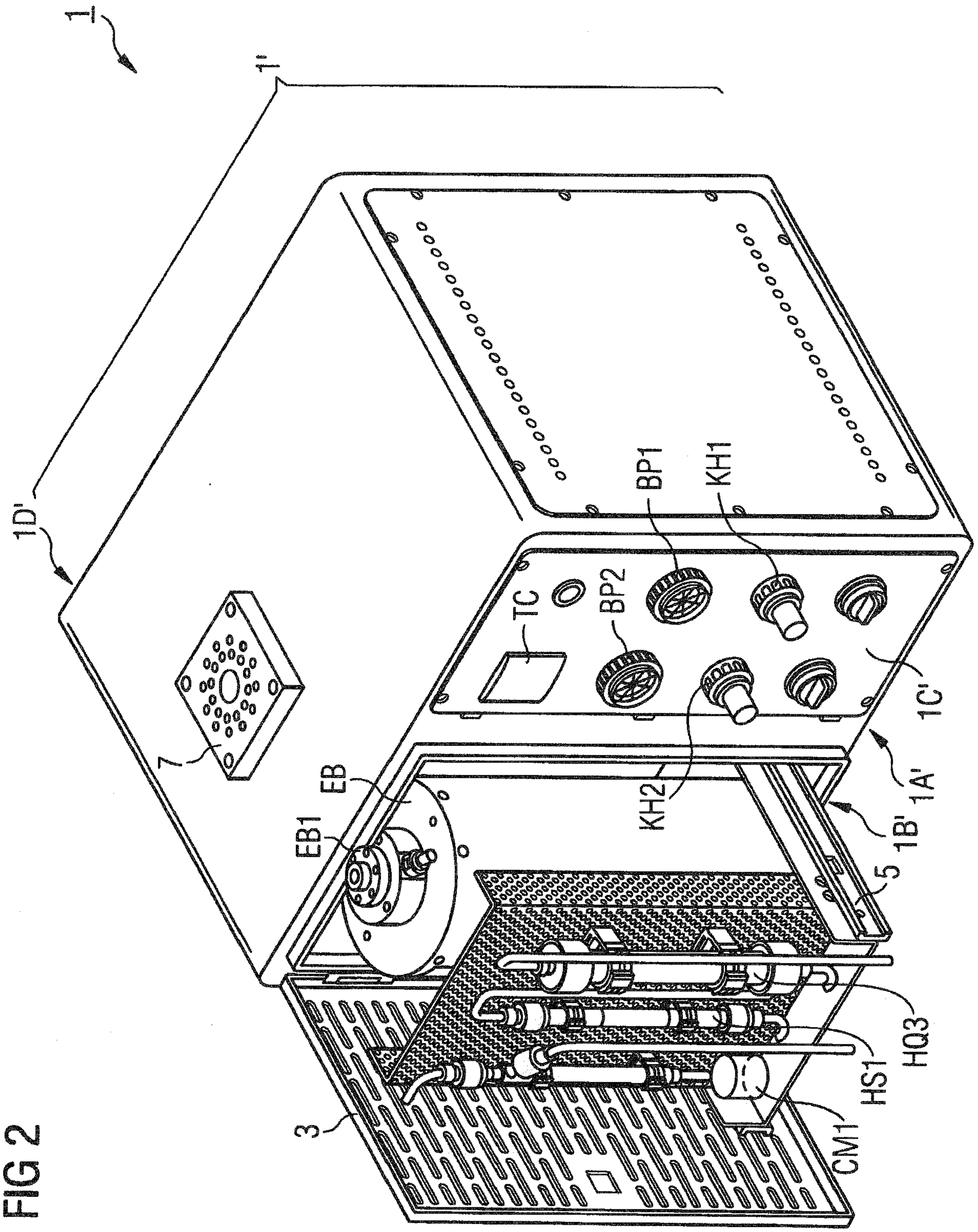

FIG 2

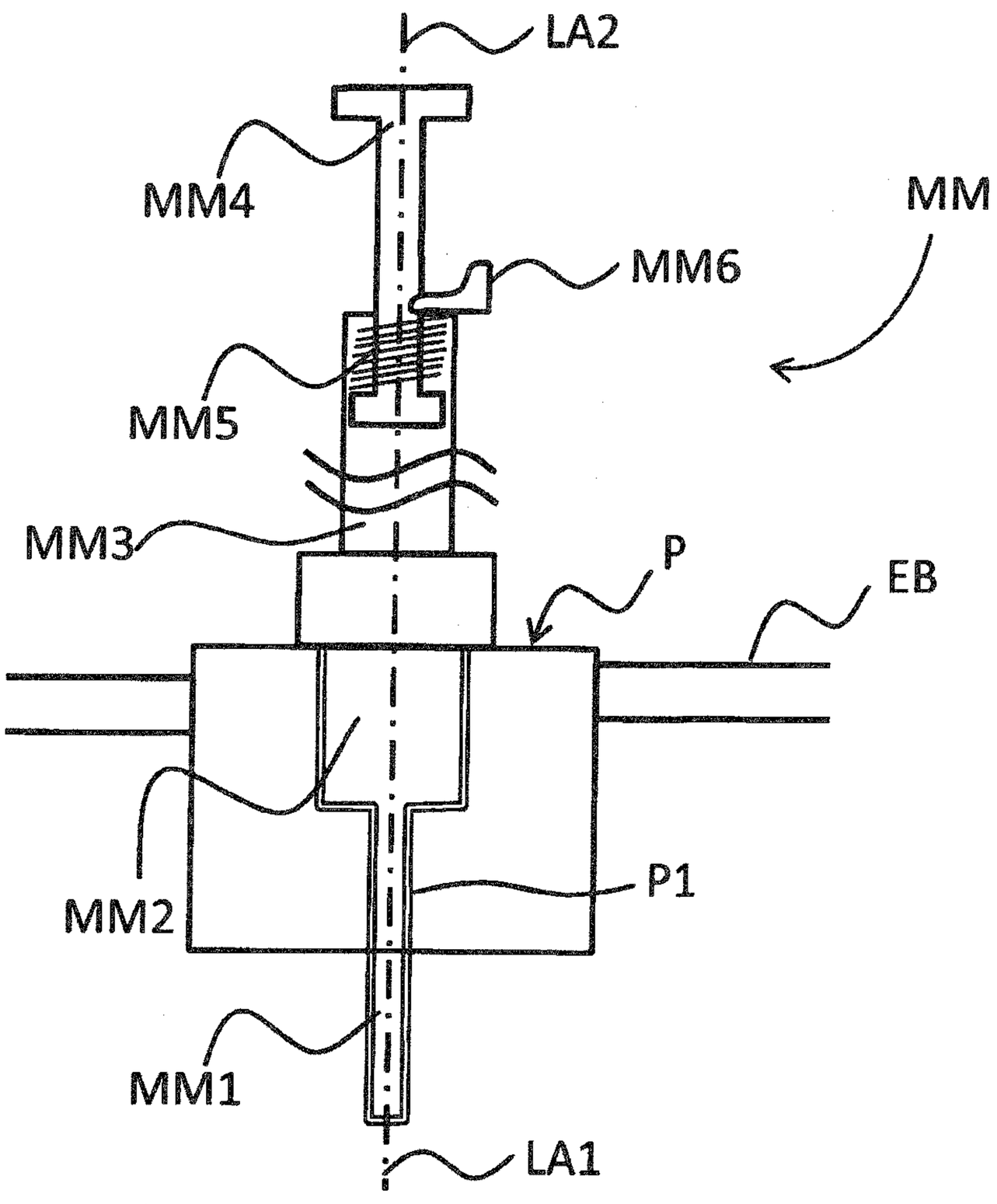

Fig. 3

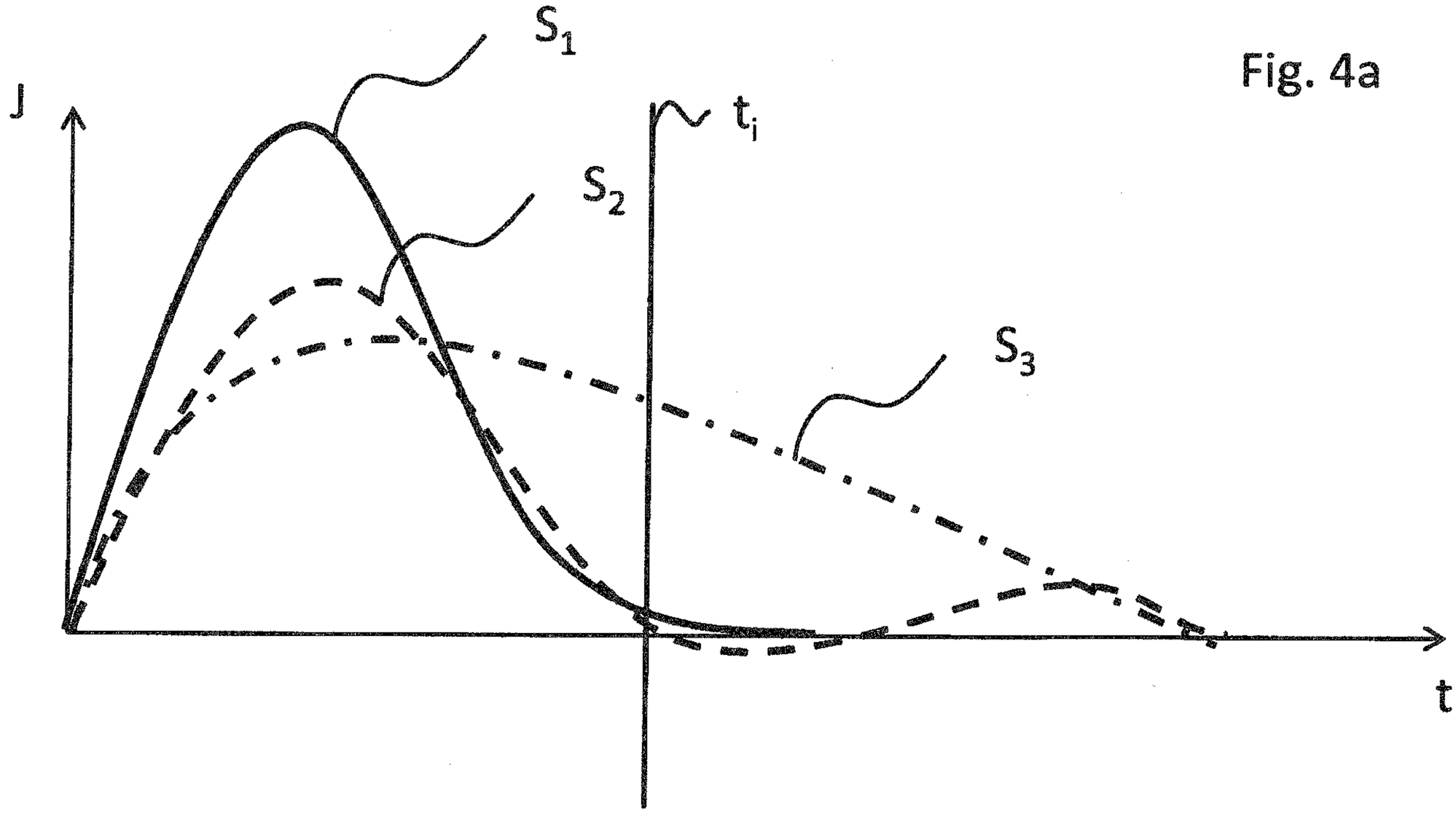

Fig. 4a

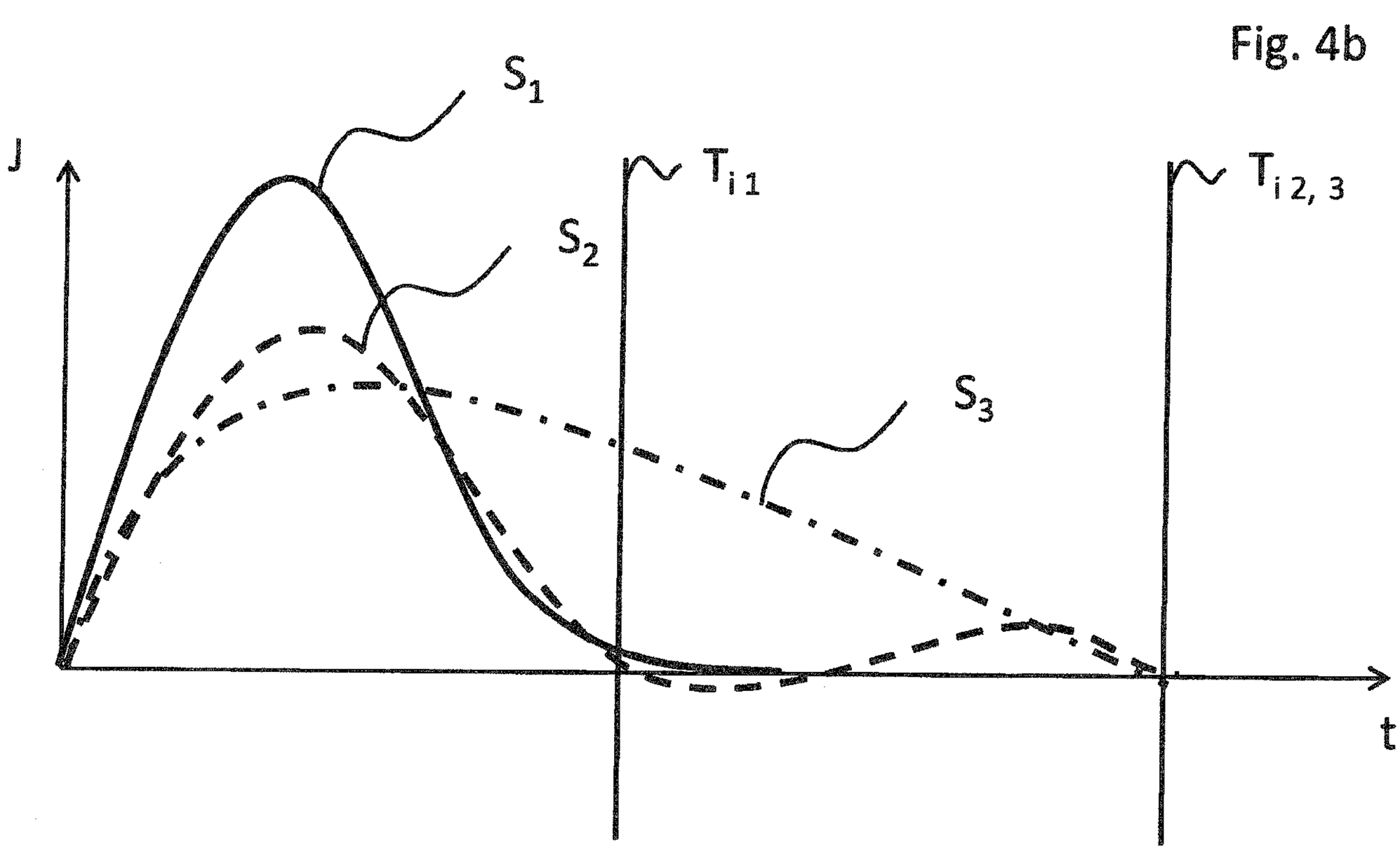

Fig. 4b