DOSING DEVICE FOR ANALYZING APPARATUS

TECHNICAL FIELD

The invention refers to a dosing device for analyzing apparatus, particularly for liquid chromatography, comprising

a) a dosing loop,

b) a change-over valve with a stationary element and an element which is movable between a first and a second position relative to the stationary element, which has a sample inlet and which engages the stationary element with a sealing body, with the change-over valve

in a first position, connecting the sample inlet to one end of the dosing loop and the other end of the dosing loop to a waste port, and

in the second position, connecting one end of the dosing loop to a carrier liquid port and the other end of the dosing loop to an analyzing apparatus port,

c) a dosing needle which, by an actuating mechanism, can be introduced with its front end into a sample vessel as well as sealingly into the sample inlet of the change-over valve, and

d) a sample pump which is connected to the rear end of the dosing needle and by which the sample liquid can be aspirated from the sample vessel into the dosing needle when the front end of the dosing needle is in the sample vessel, and by which sample liquid can be pressed out of the dosing needle and can be transferred into the dosing loop when the dosing loop is in the sample inlet of the change-over valve in its first position.

The analyzing apparatus can be a chromatographic separation column for liquid chromatography with a detector which is connected downstream. However, the analyzing apparatus can also be an atomic absorption or atomic emission spectro-photometer, for example, operating with the flow-injection technique. The invention can be applied anywhere where sample liquid shall be introduced into a dosing tube by means of a dosing needle.

BACKGROUND ART

In a prior art dosing device, samples are arranged in sample vessels in a magazine. The sample vessels consist of sample bottles which are closed by a septum. A sample-taking unit is provided on a conveying arm which can be moved according to a program above each of the sample bottles, a rinsing vessel, or the sample inlet. The sample-taking unit comprises a dosing needle which can be introduced into a sample bottle, by penetrating the septum, into the sample inlet or into the rinsing vessel. The change-over valve comprises a stator and a rotor which can be rotated relative to the stator between a first and a second position. The rotor has the sample inlet. The rotor engages the stator with the sealing body. In the first position, the change-over valve connects the sample inlet to one end of a dosing loop and the other end of the dosing loop to a waste port. In the second position, the change-over valve connects one end of the dosing loop to a carrier liquid port and the other end of the dosing loop to an analyzing apparatus port, i.e., a port which leads to a chromatographic separation column, for example. The rear end of the dosing needle communicates through a capillary with a sample pump and a rinsing pump. The rinsing pump has check-valves and aspirates carrier liquid which can be passed by a rinsing process through the dosing needle to the rinsing vessel. Thus, the dosing needle and the capillary are filled up to the tip of the dosing needle by a carrier liquid. Further, the capillary is connected to a sample pump. The sample pump aspirates a volume of a carrier liquid. Thereby, the carrier liquid is sucked back out of the dosing needle so that another medium, air, or sample liquid is aspirated from the tip into the dosing needle.

During operation, the dosing needle is moved above the sample bottle which is located in a predetermined magazine position. First, the dosing needle penetrates only the septum of the sample bottle without dipping into the liquid. Then, the dosing needle is again moved out of the sample bottle. After returning to the starting position, a small volume of air is aspirated by the sample pump. This volume of air serves as a separating layer between the carrier liquid and the sample liquid which shall be aspirated. After a waiting period, the dosing needle penetrates the septum for a second time and now, dips into the sample liquid. The sample pump aspirates a predetermined sample volume and, in addition, a predetermined excess volume into the dosing needle and the dosing capillary. Then, the dosing needle is lifted. The conveying arm moves the dosing needle above the sample inlet of the change-over valve.

In the prior art dosing devices, first, the change-over valve is in its second position in which the dosing loop communicates with a carrier liquid port and an analyzing apparatus port. In this position a part of the excess volume is pressed into the sample inlet in order to rinse the sample inlet with sample liquid. This sample liquid is used for rinsing and runs to a waste vessel. After a waiting period, the change-over valve is rotated to the first position. The sample pump presses the predetermined sample volume into the dosing loop. The displaced volume of carrier liquid is discharged through a capillary. After a further waiting period, the change-over valve is rotated to the second position. The dosed sample volume is conveyed to the analyzing apparatus, here a chromatographic separation column, by a carrier liquid flow which is supplied through the carrier liquid port by a carrier liquid pump. Then, the sample pump pressees the sample liquid and the aspirated air into the sample inlet and through this to a waste port. Subsequently, the rinsing pump carries out a stroke cycle. Hereby, the dosing needle and the sample inlet are rinsed with carrier liquid. This carrier liquid pumped by the rinsing pump also runs through the sample inlet to the waste port. After that, the dosing needle is lifted. The conveying arm moves the dosing needle to a position of rest above the rinsing vessel.

Such arrangements are known from German Patent No. 30 30 3963, which is cognate with U.S. Pat. No. 4,413,534, granted Nov. 8, 1983, and German Patent No. 30 37 014 which is cognate with U.S. Pat. No. 4,393,726, granted Jul. 19, 1983.

Concerning prior art, in the aforementioned German Patent No. 30 30 396 valve of the present type is described which serves as a sample inlet valve and in which a dosing needle is introduced into the rotor up to the engaging surface of the stator, such that it does not penetrate into the stator beyond the engaging surface. For this purpose, the dosing bore in the stator has a smaller diameter than the dosing needle. After supplying the sample to the dosing loop, the rotor is rotated to the second position of the change-over valve. During