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(54) **AGRICULTURAL SAMPLING SYSTEM AND RELATED METHODS**

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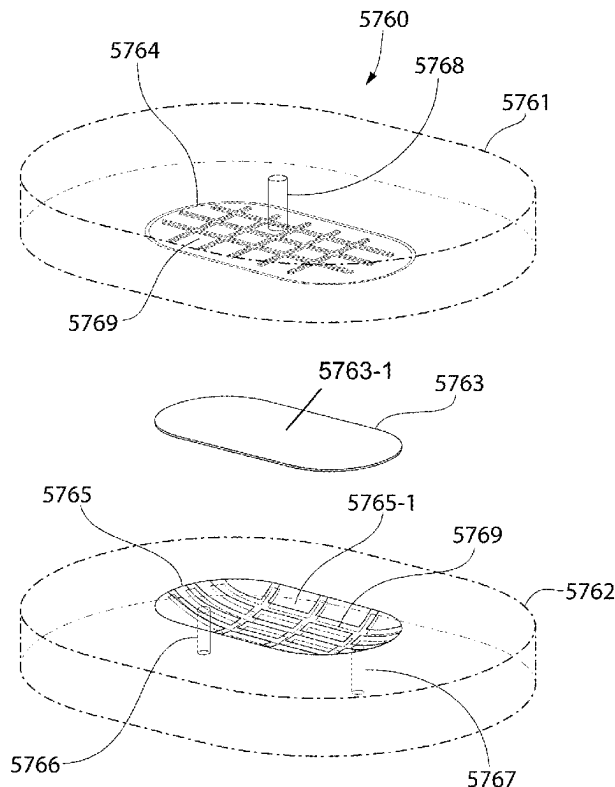
(63) Continuation of application No. 18/756,467, filed on Jun. 27, 2024, now Pat. No. 12,313,619, which is a continuation of application No. 17/343,536, filed on Jun. 9, 2021, now Pat. No. 12,031,970, which is a continuation-in-part of application No. PCT/IB2021/052872, filed on Apr. 7, 2021, which is a continuation-in-part of application No. PCT/IB2021/052874, filed on Apr. 7, 2021, which is a continuation-in-part of application No. PCT/IB2021/052875, filed on Apr. 7, 2021, which is a continuation-in-part of application No. PCT/IB2021/052876, filed on Apr. 7, 2021.
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F04B 43/073 (2006.01)
F16K 99/00 (2006.01)
 (52) **U.S. Cl.**
 CPC **G01N 33/24** (2013.01); **A01B 79/005** (2013.01); **B01L 3/50273** (2013.01); **B01L 3/502738** (2013.01); **F04B 19/006** (2013.01); **B01L 2200/0605** (2013.01); **B01L 2300/0816** (2013.01); **B01L 2300/0877** (2013.01); **B01L 2300/0883** (2013.01); **B01L 2300/0887** (2013.01); **F04B 13/02** (2013.01); **F04B 43/073** (2013.01); **F16K 99/0015** (2013.01); **F16K 99/0059** (2013.01); **F16K 2099/0084** (2013.01); **G01N 33/245** (2024.05)

(57) **ABSTRACT**

A microfluidic manifold for processing an agricultural sample fluid having: a micropump codefined between a liquid layer and an air layer of the manifold, the micropump having a pump chamber collectively formed by an air-side recess in the air layer and a liquid-side recess in the liquid layer, and a resiliently deformable diaphragm separating the air-side and liquid-side recesses; wherein the liquid-side recess comprises a plurality of anti-stall grooves recessed into the liquid layer.



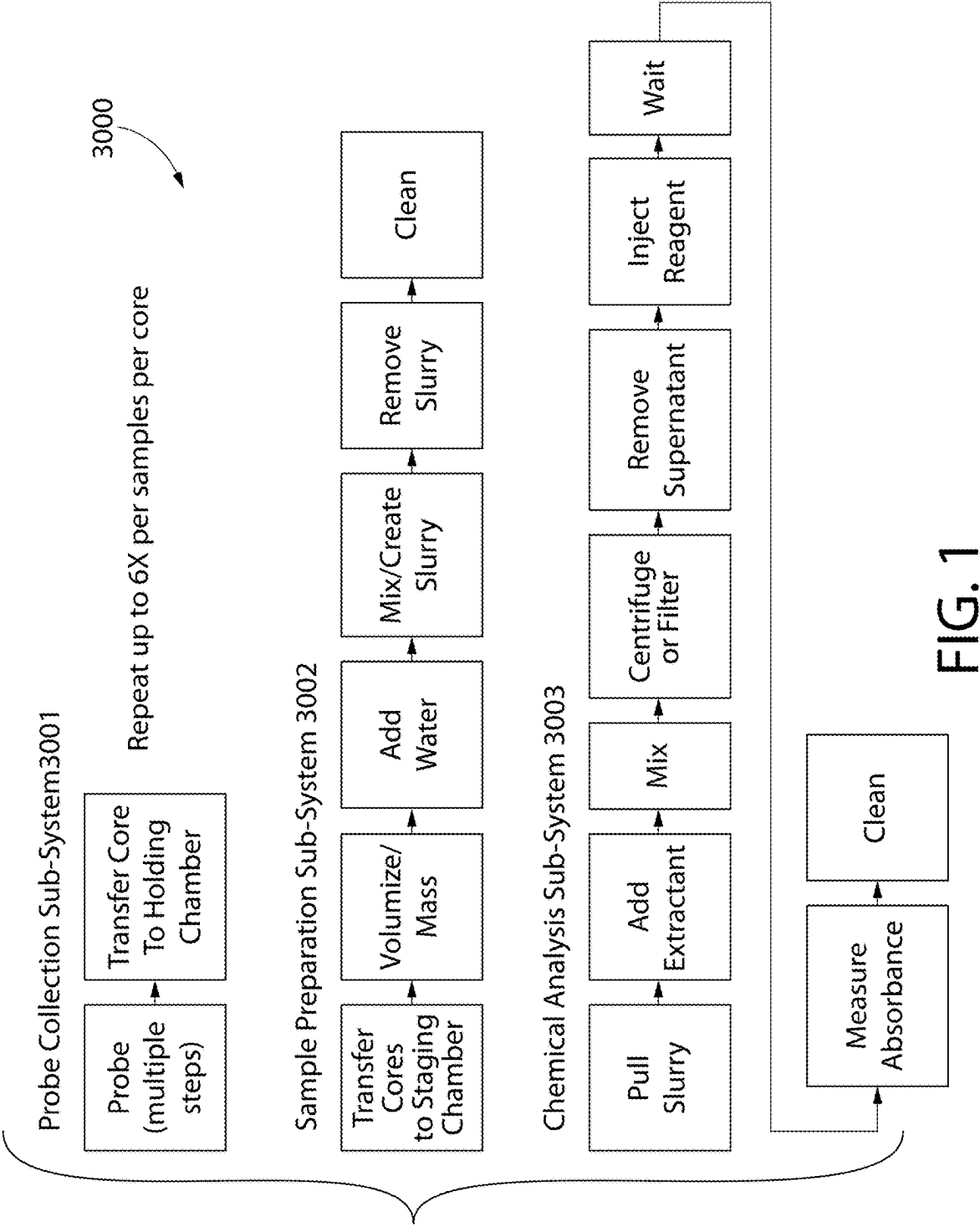


FIG. 1

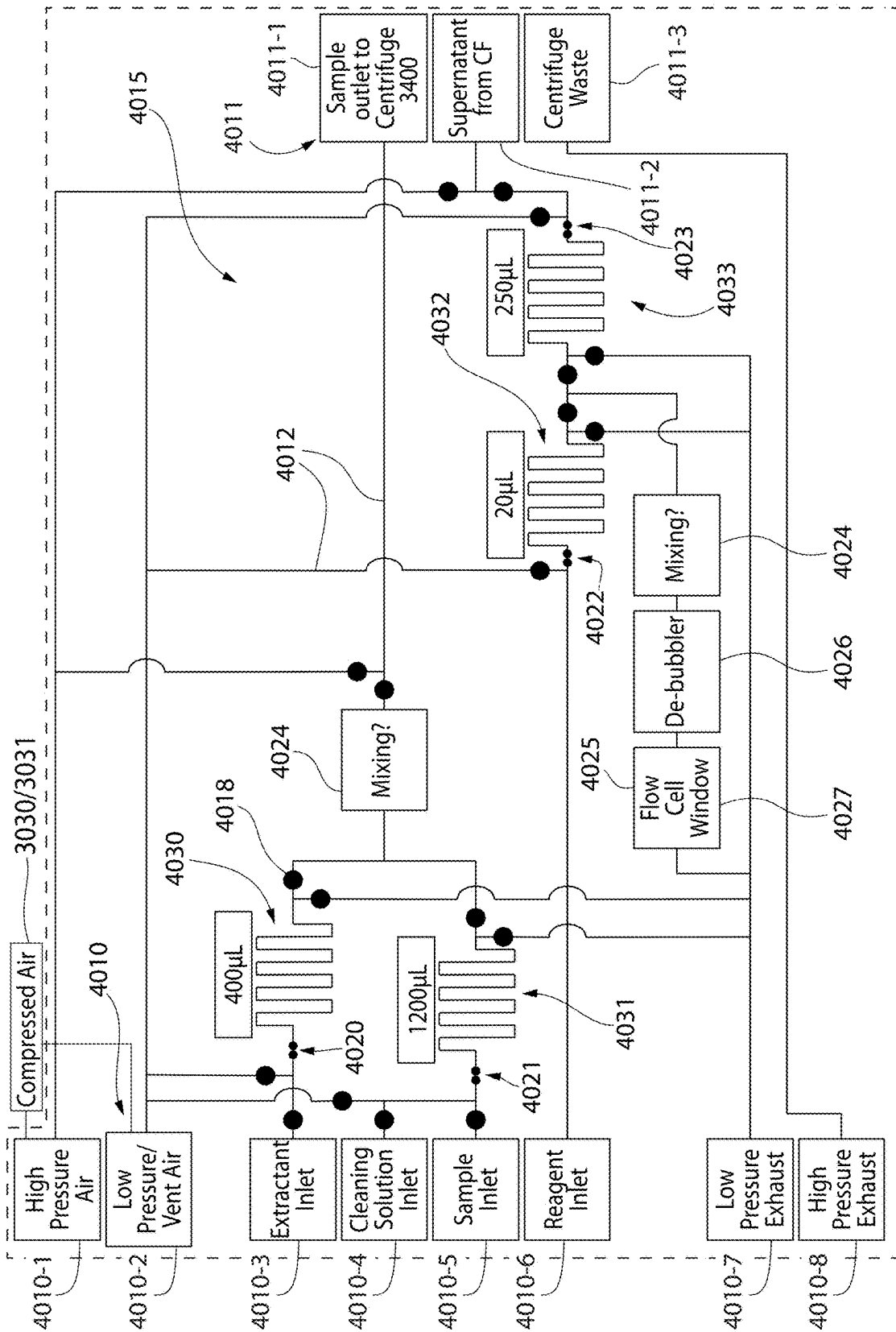


FIG. 2

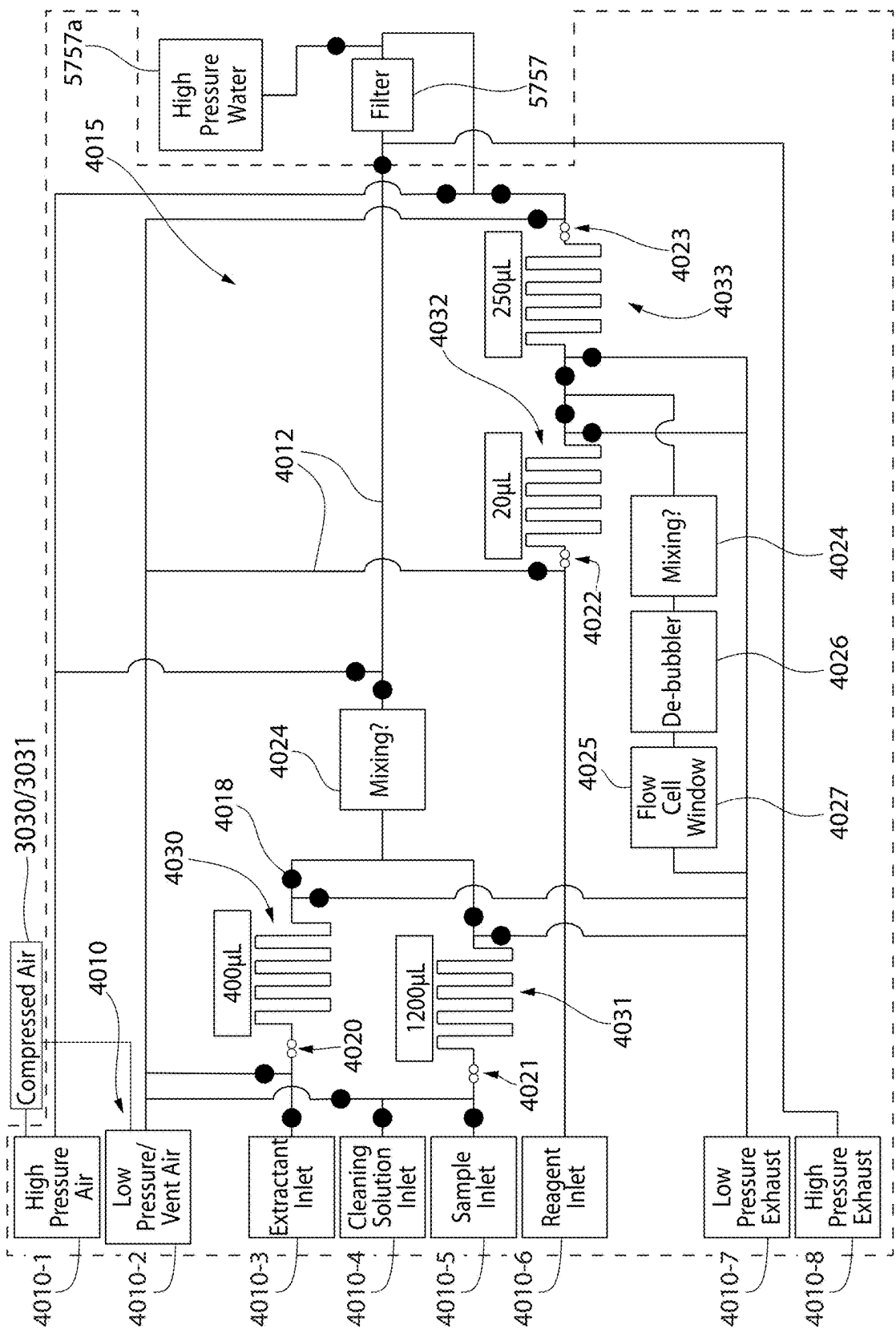


FIG. 3

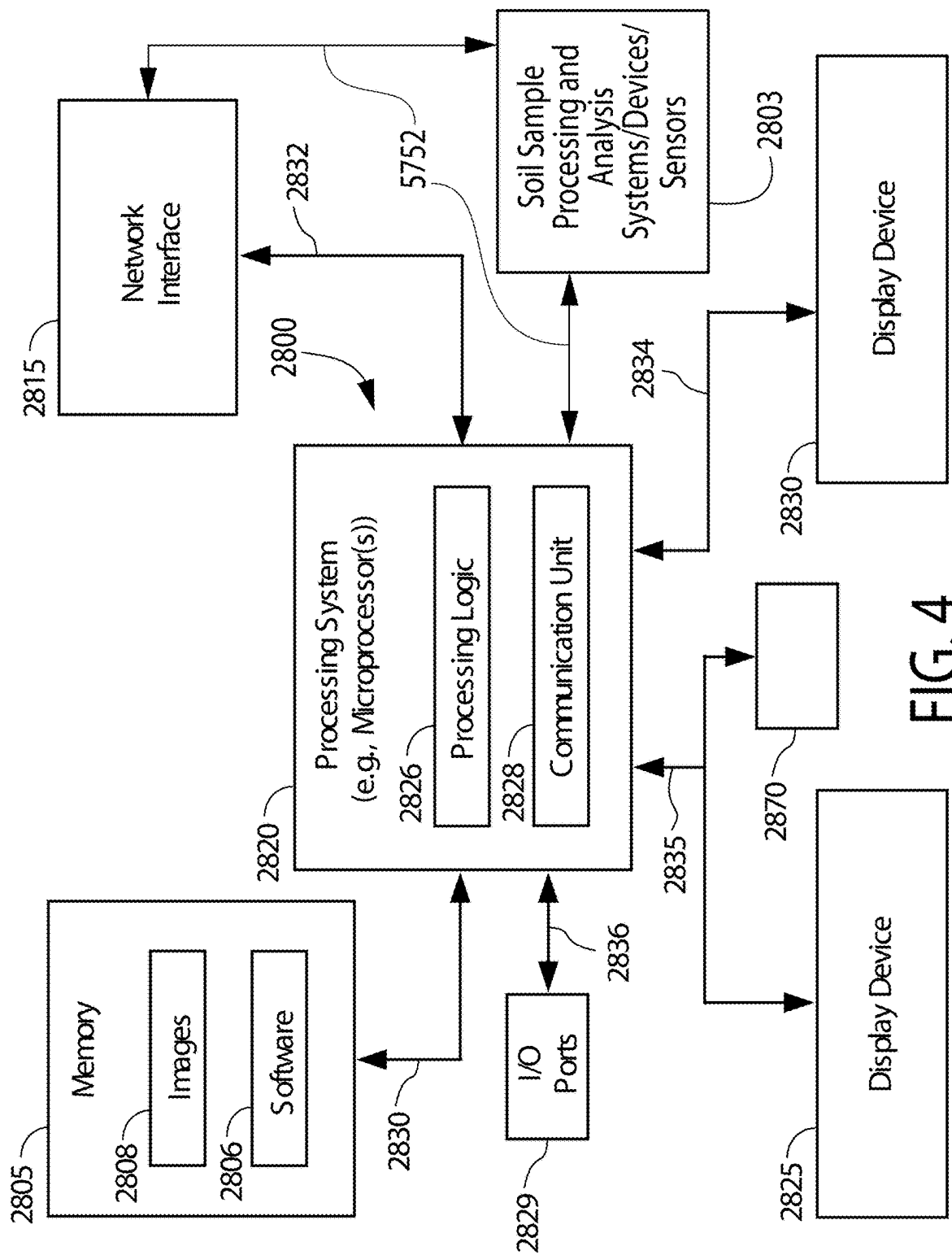


FIG. 4

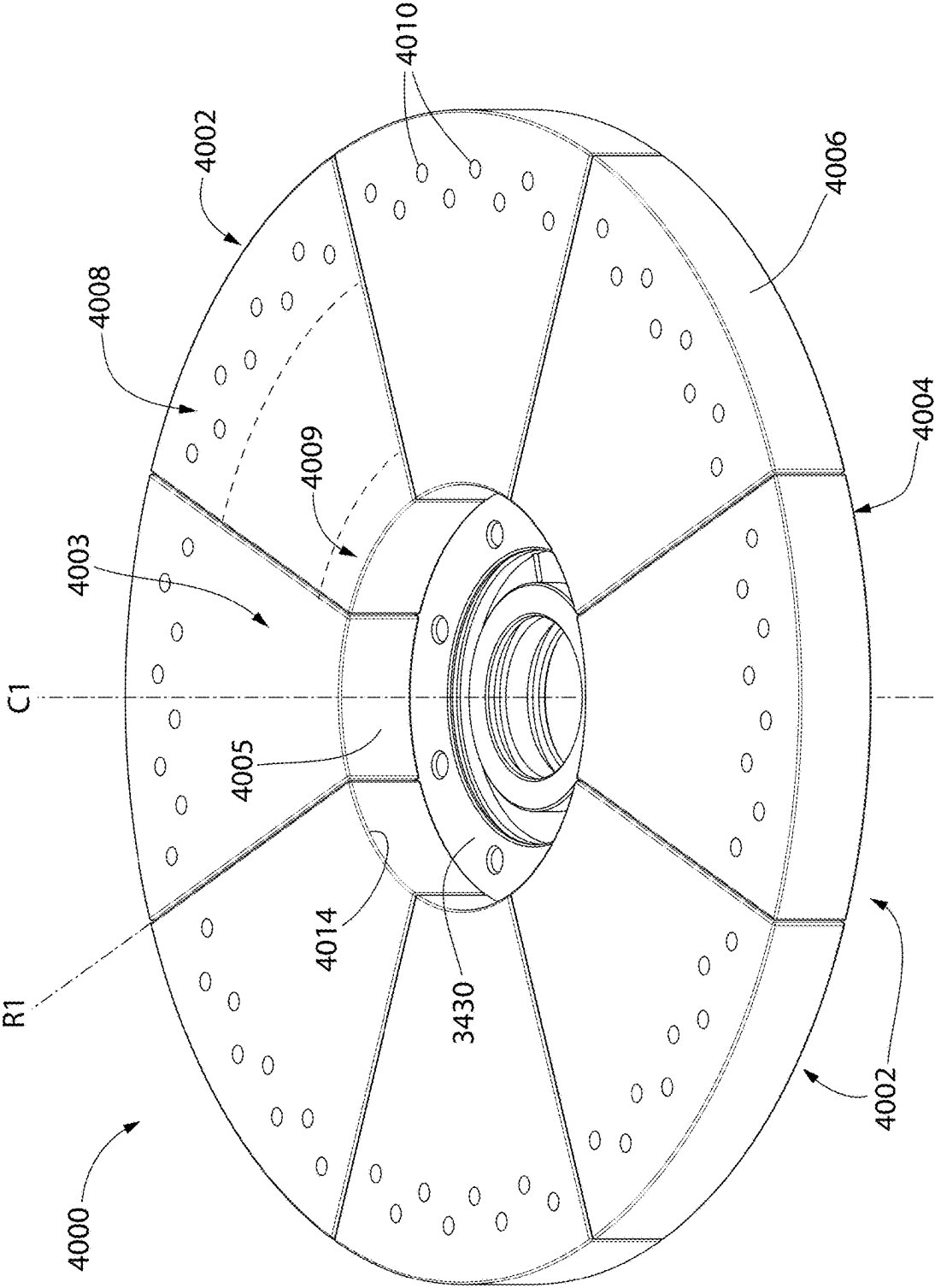


FIG. 5

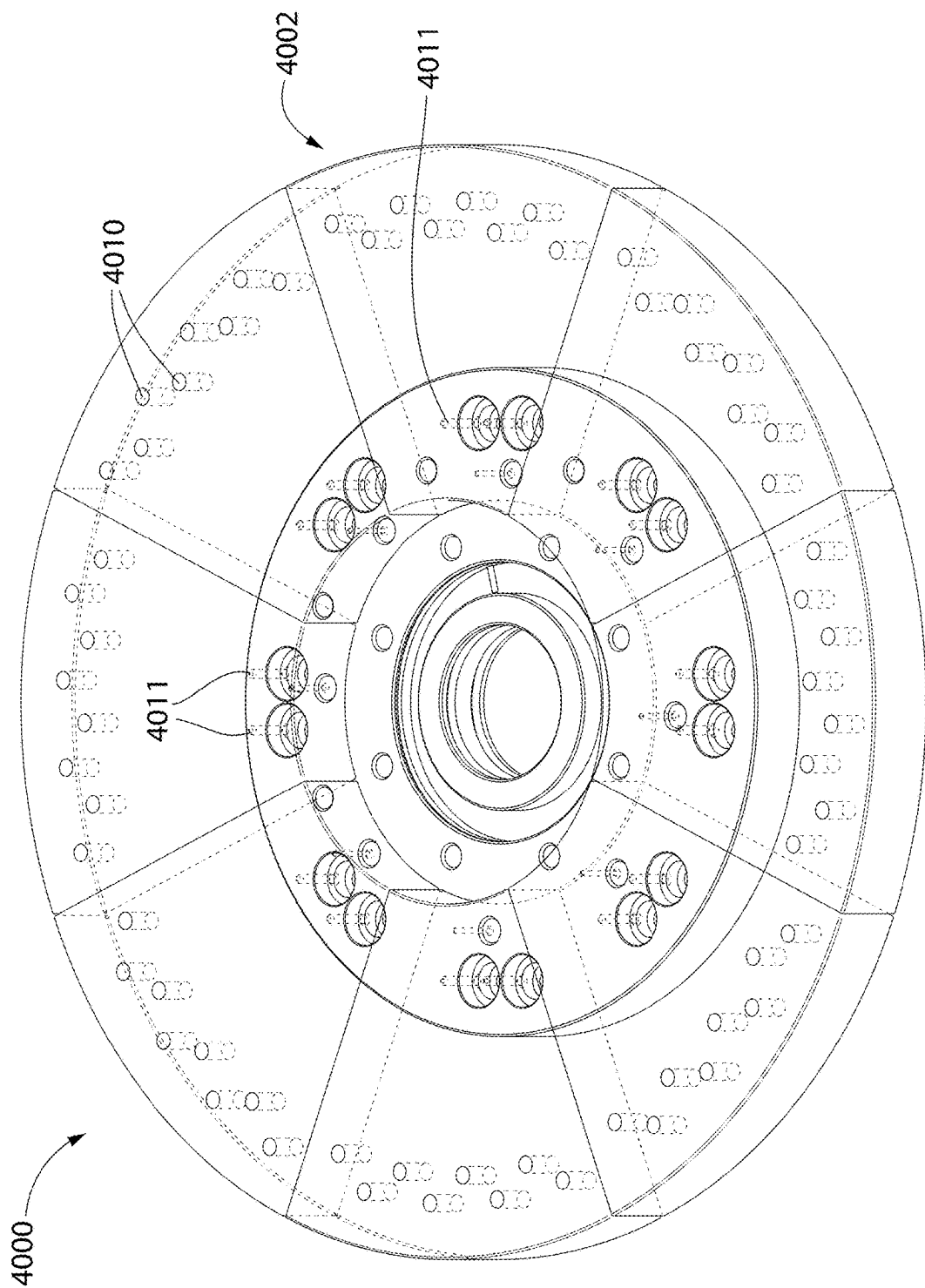


FIG. 6

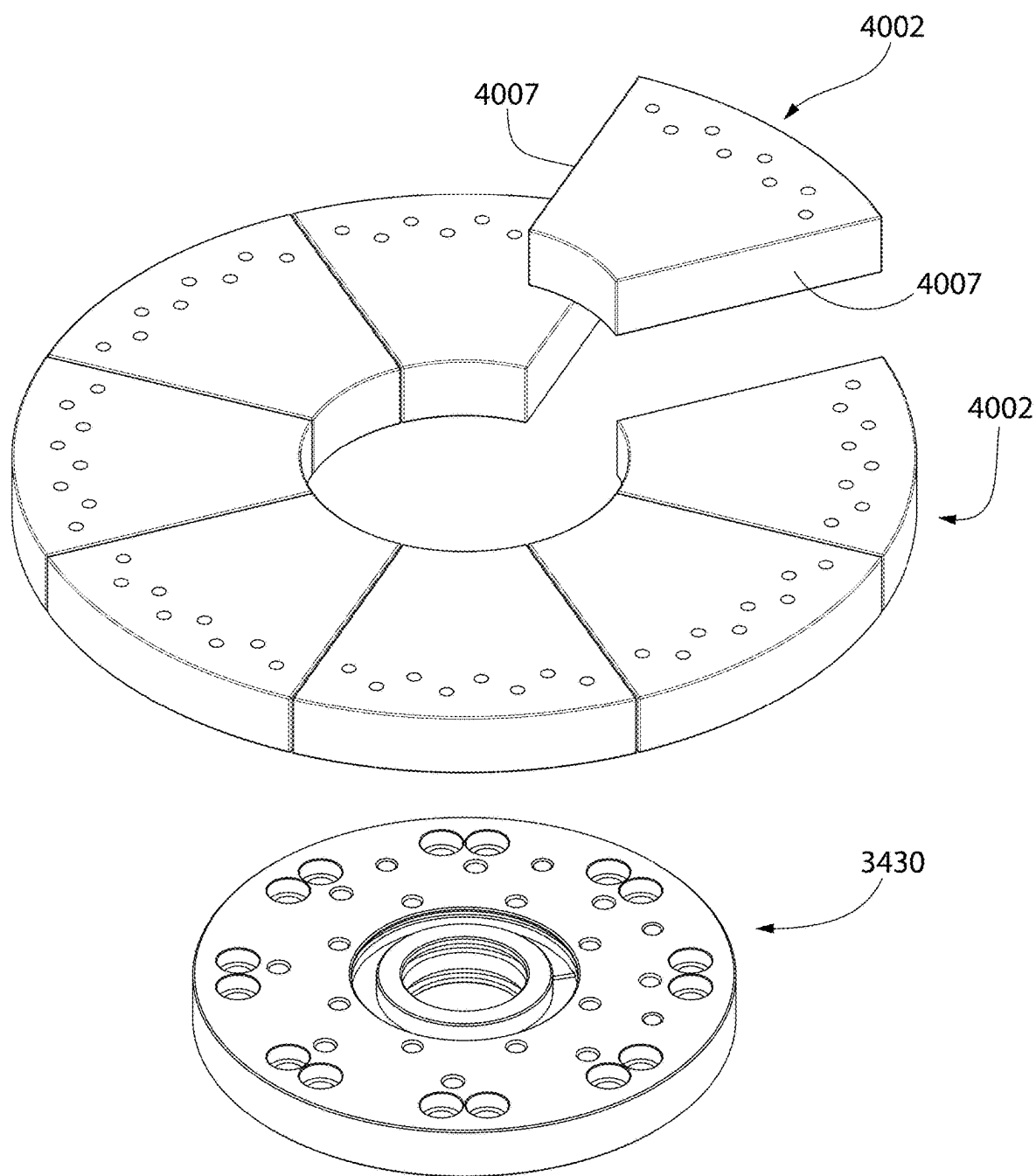


FIG. 7

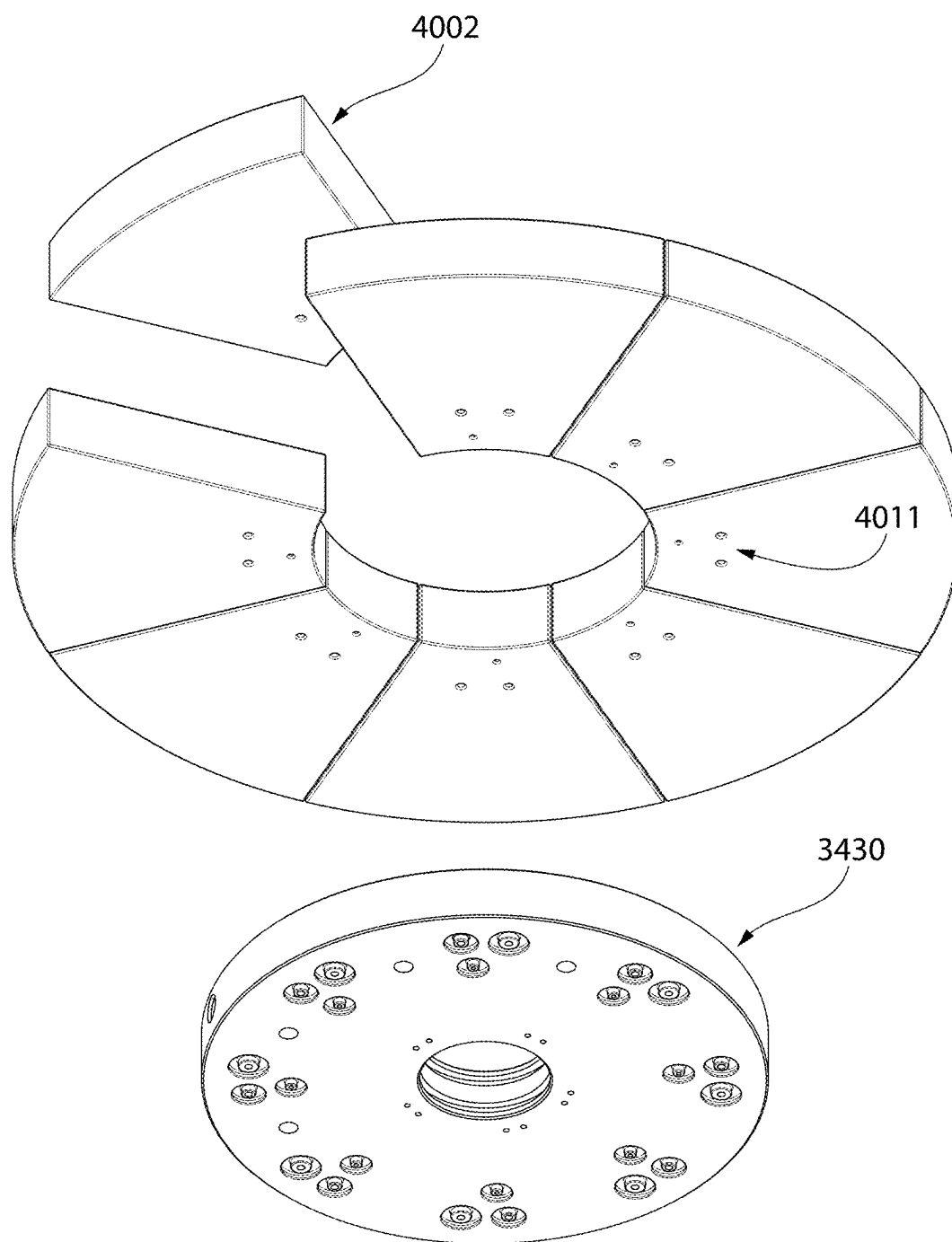


FIG. 8

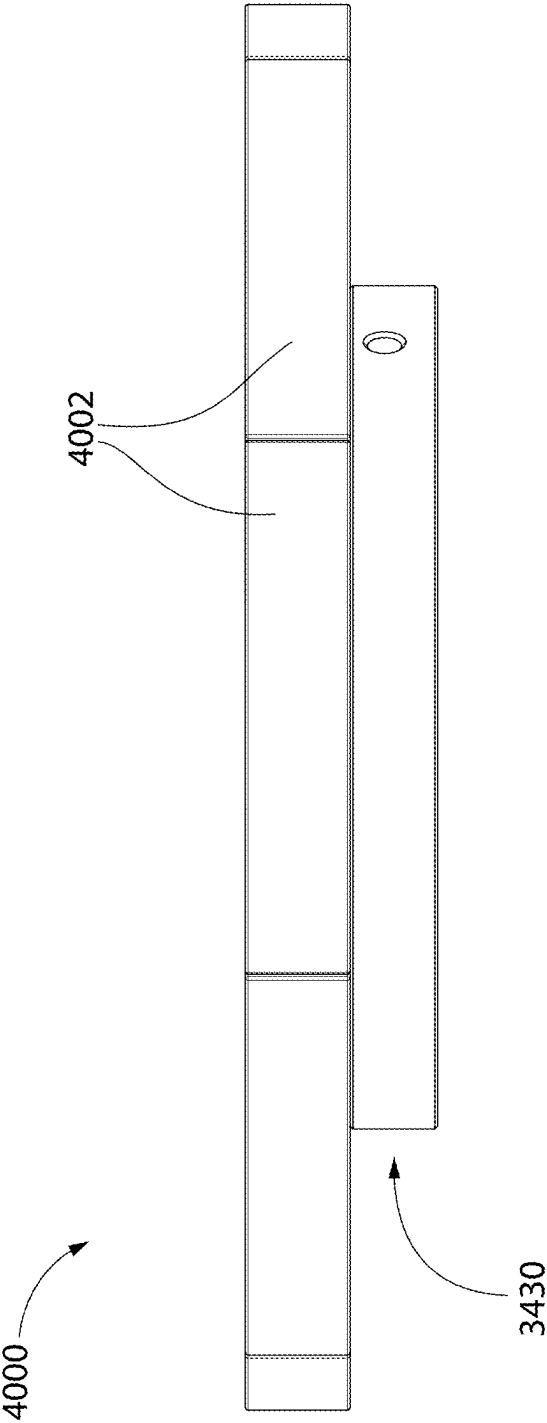


FIG. 9

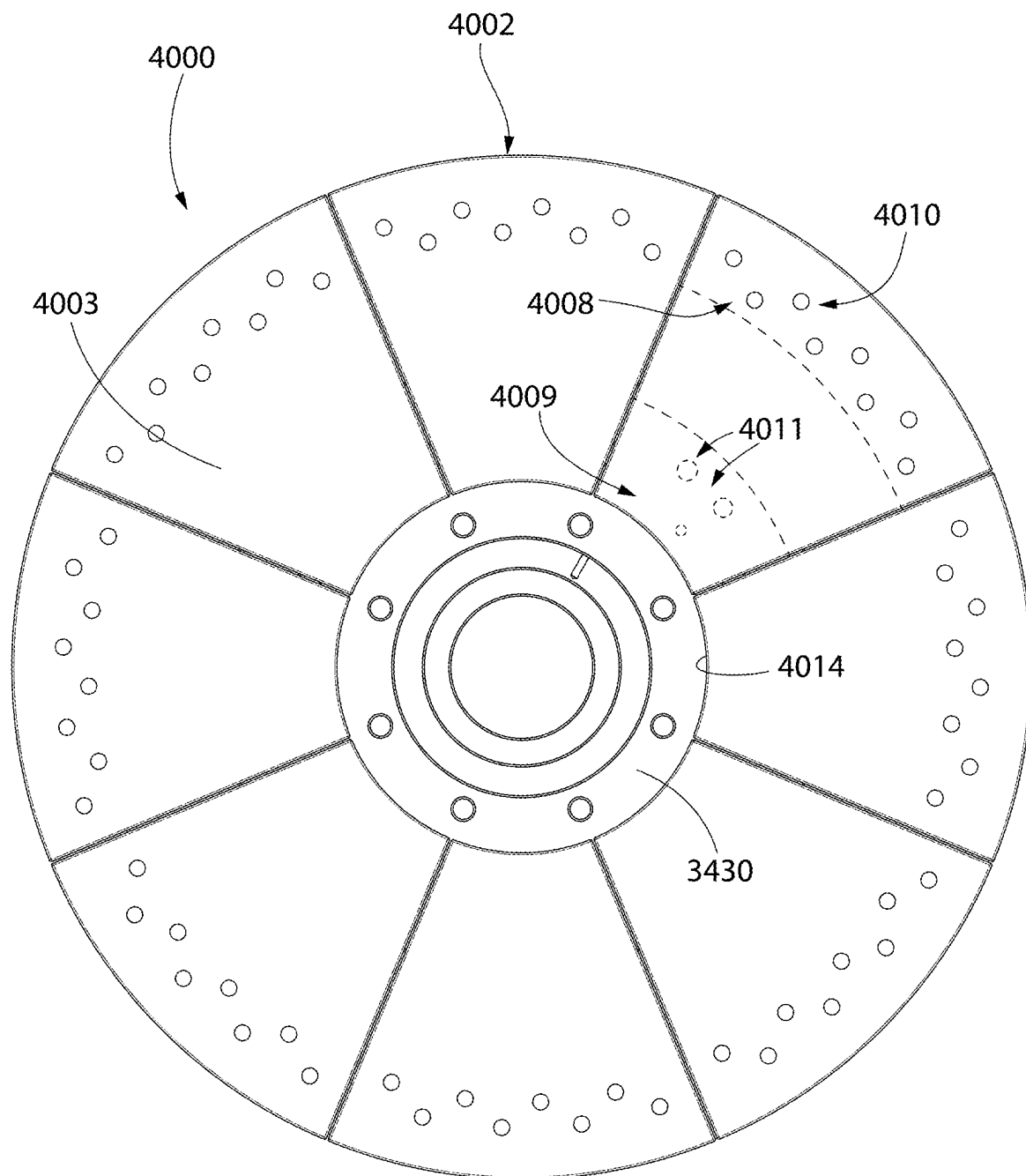


FIG. 10

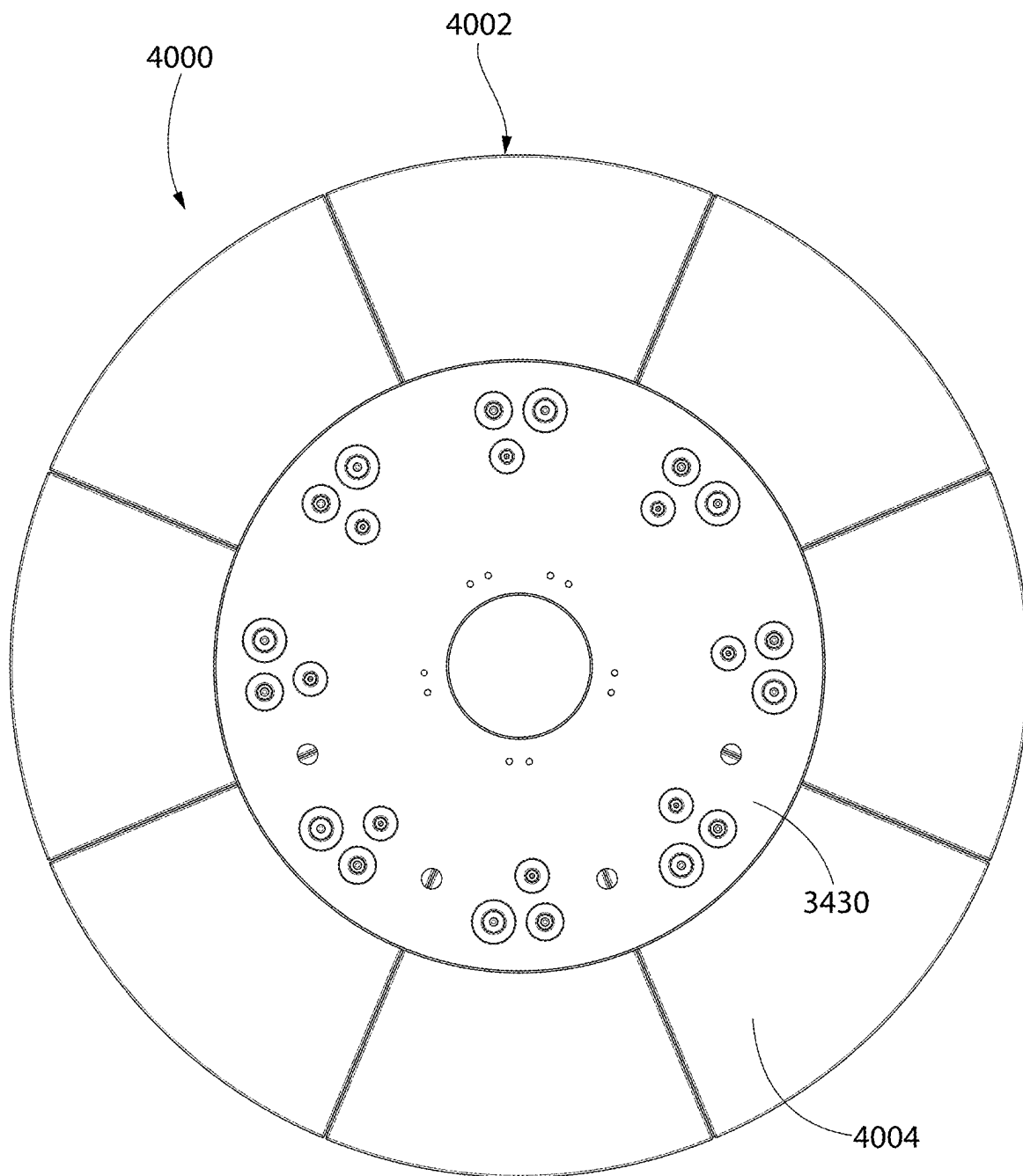


FIG. 11

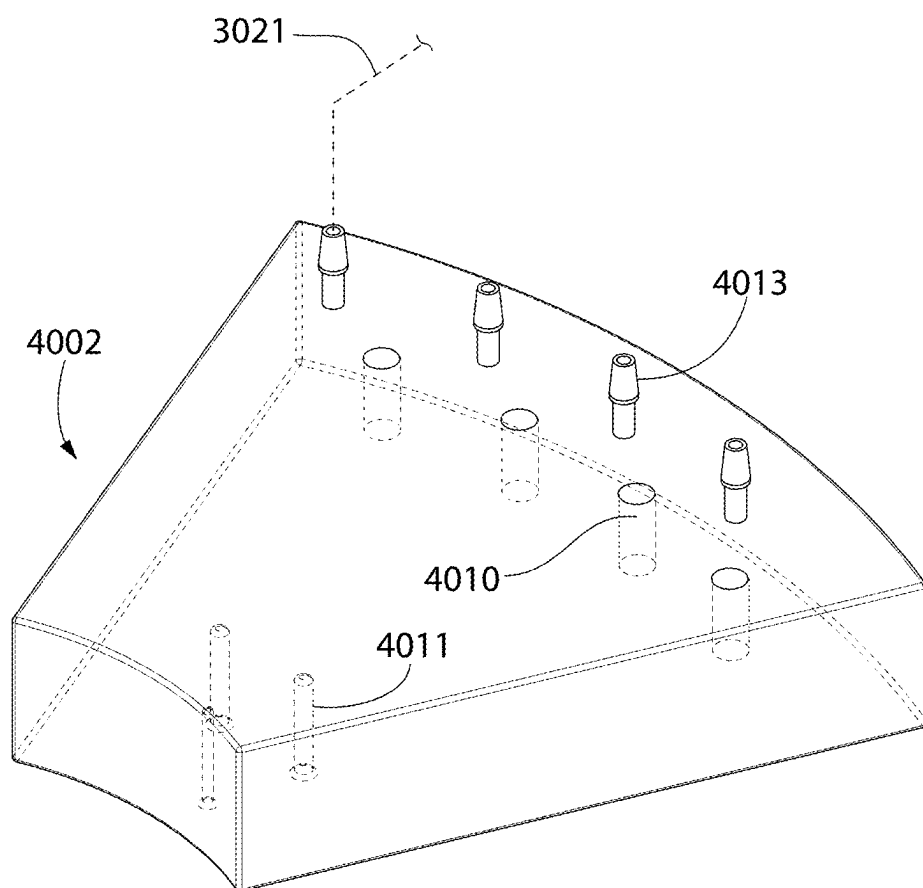


FIG. 12

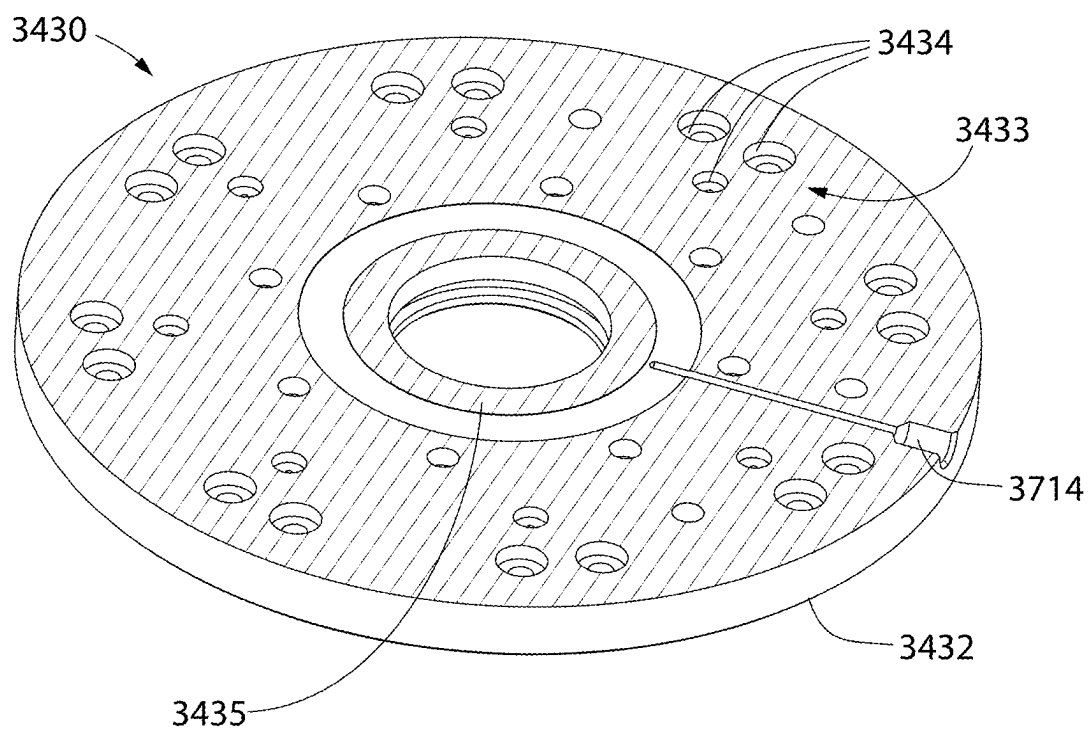


FIG. 13

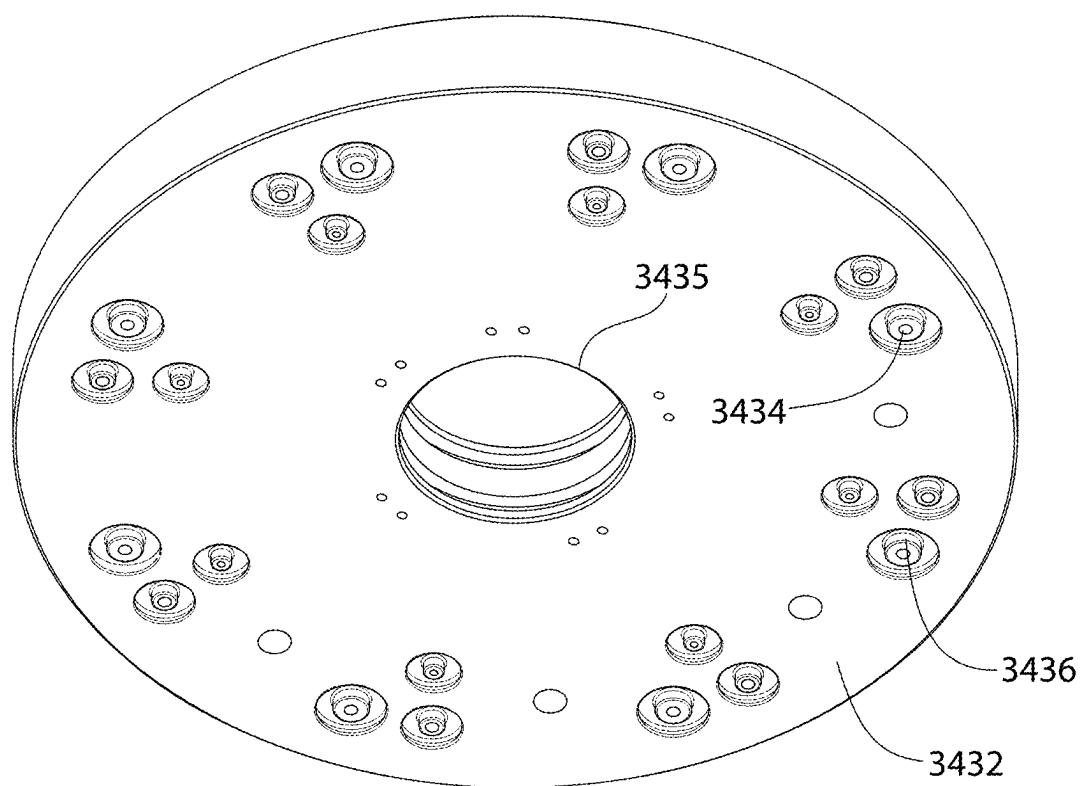


FIG. 14

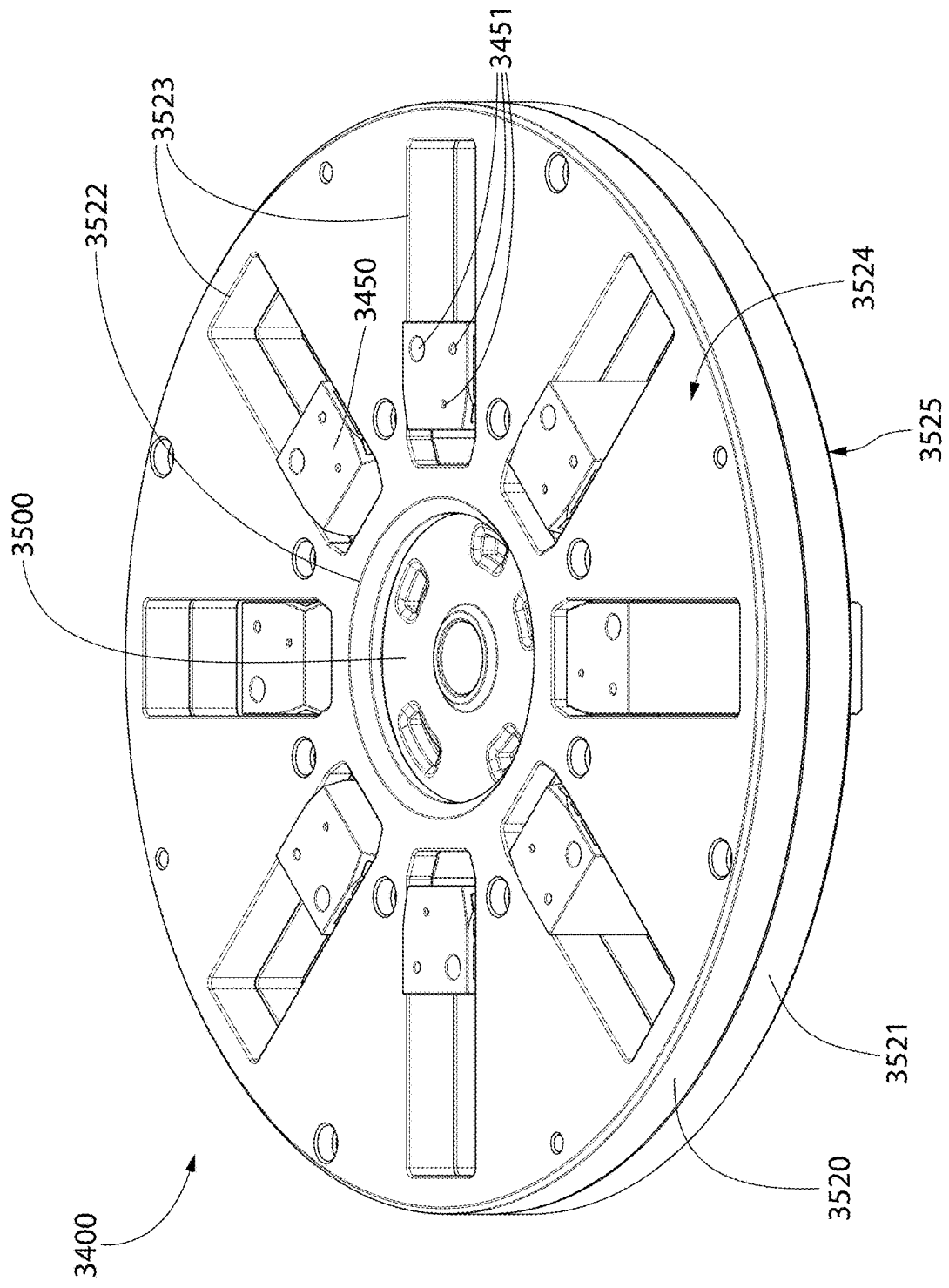


FIG. 15

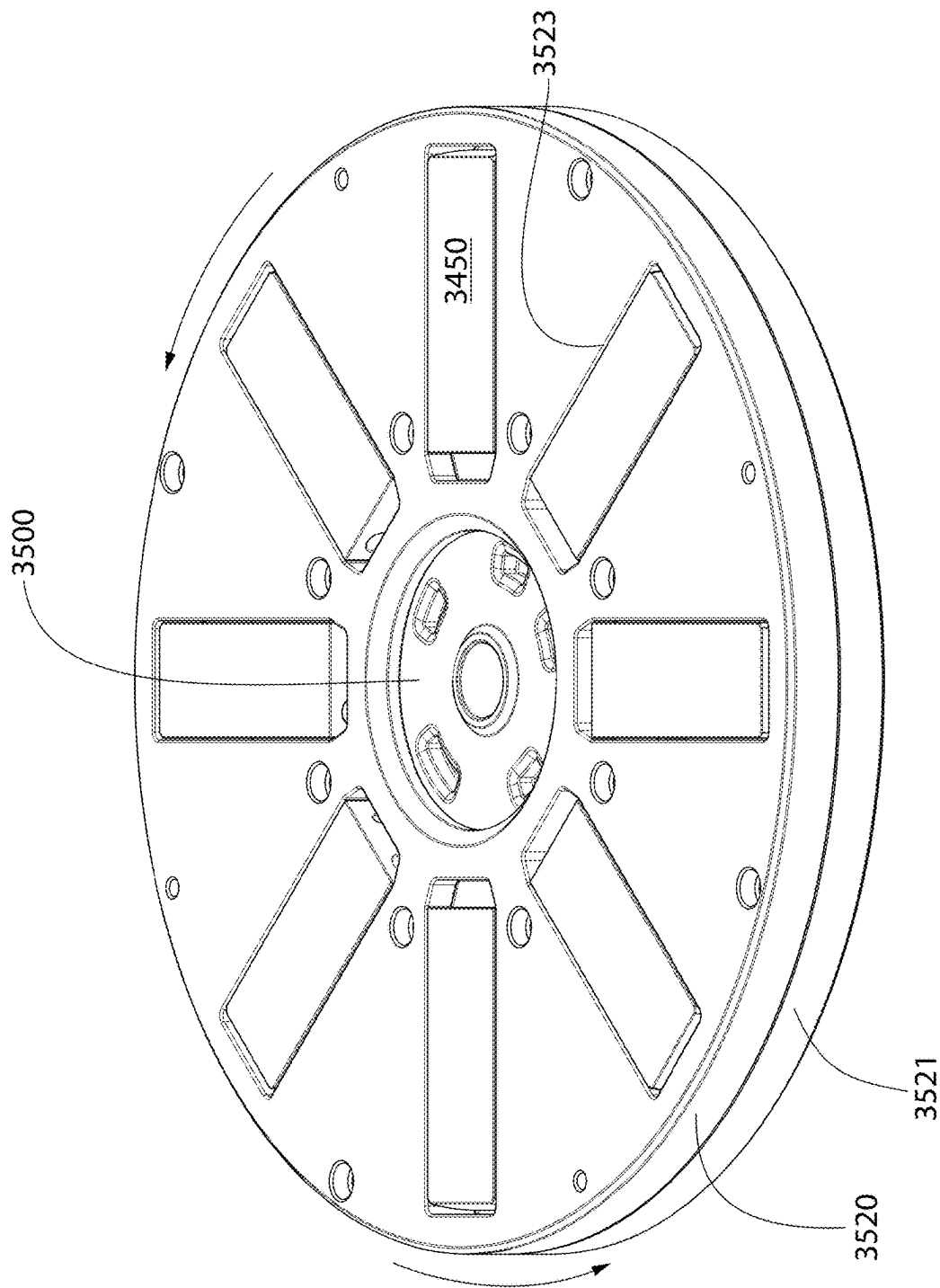


FIG. 16

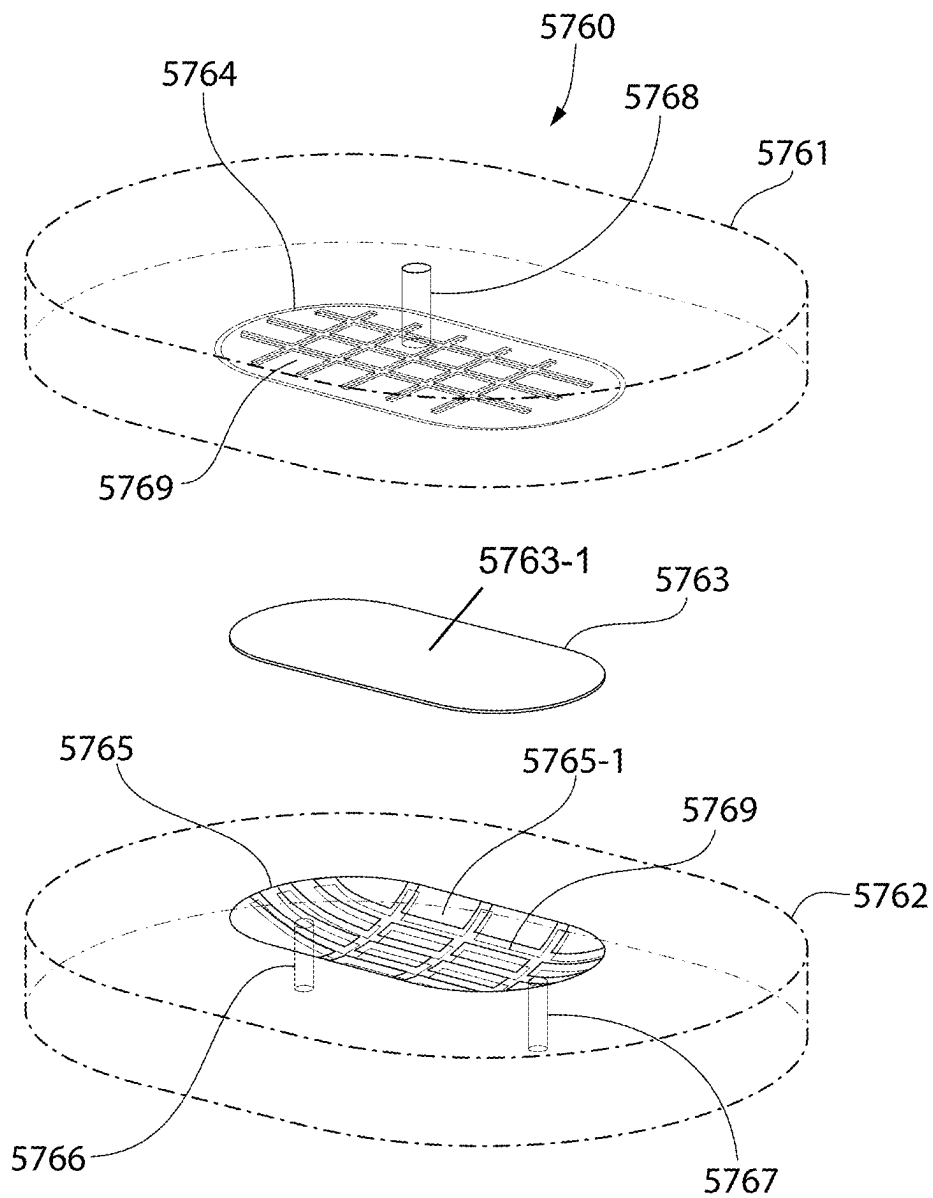


FIG. 17

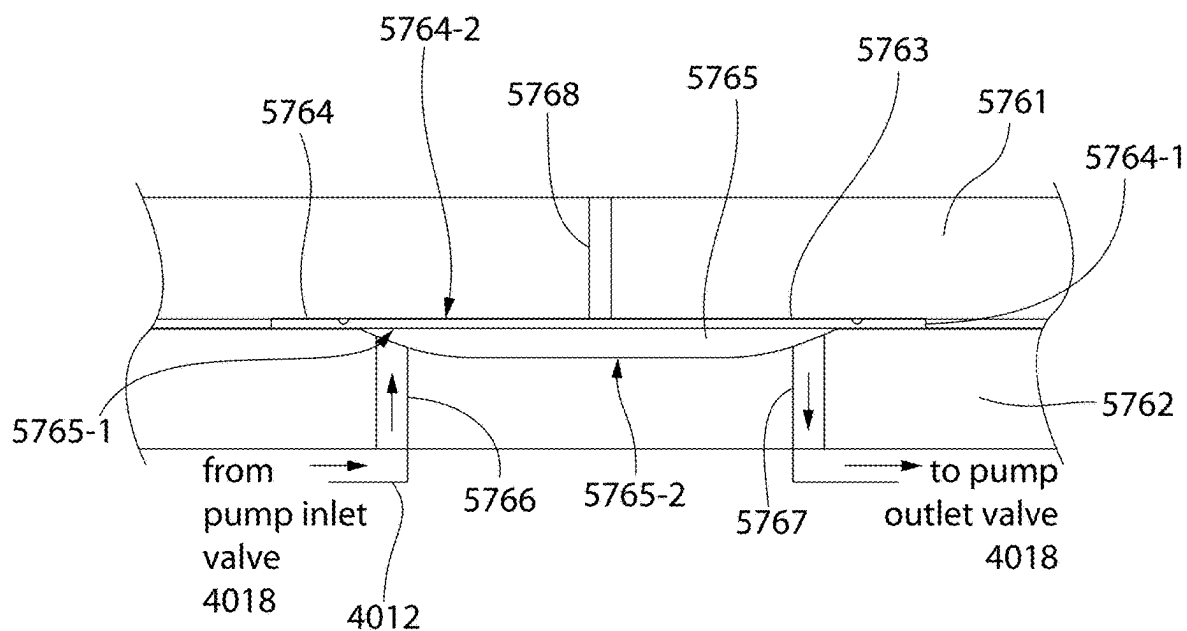


FIG. 18

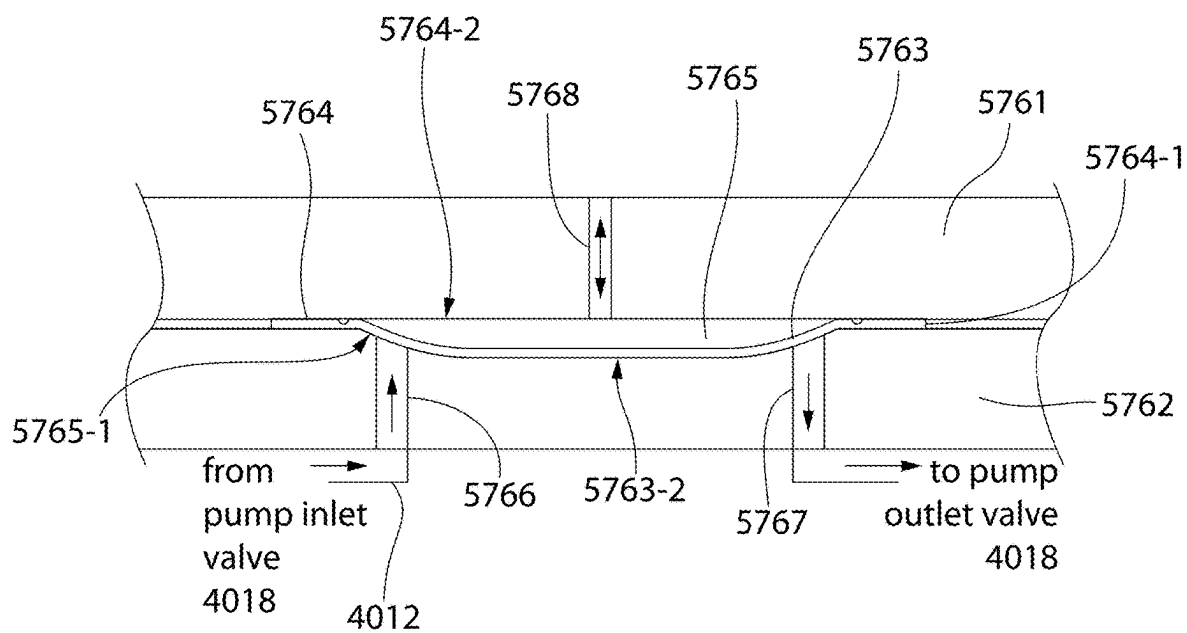


FIG. 19

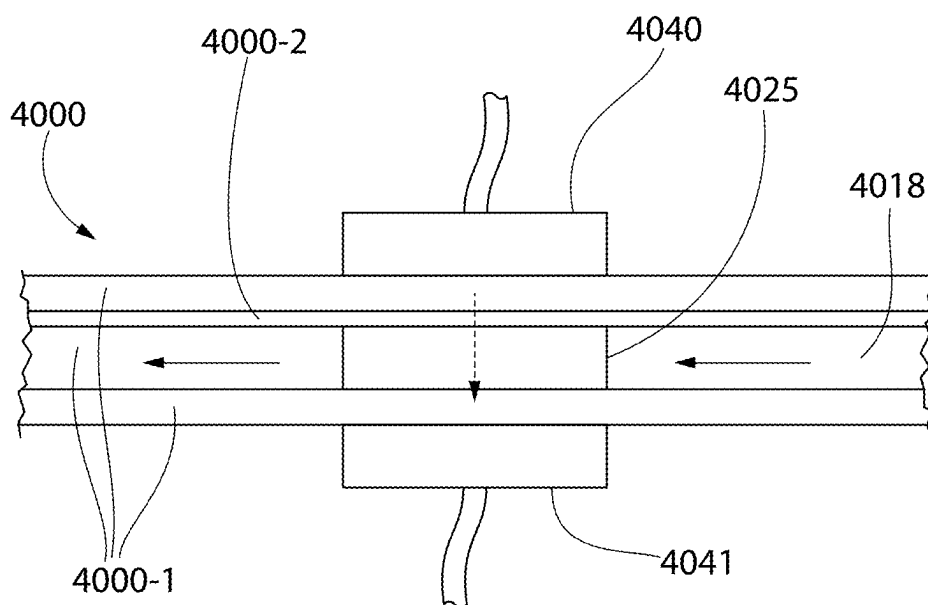


FIG. 20

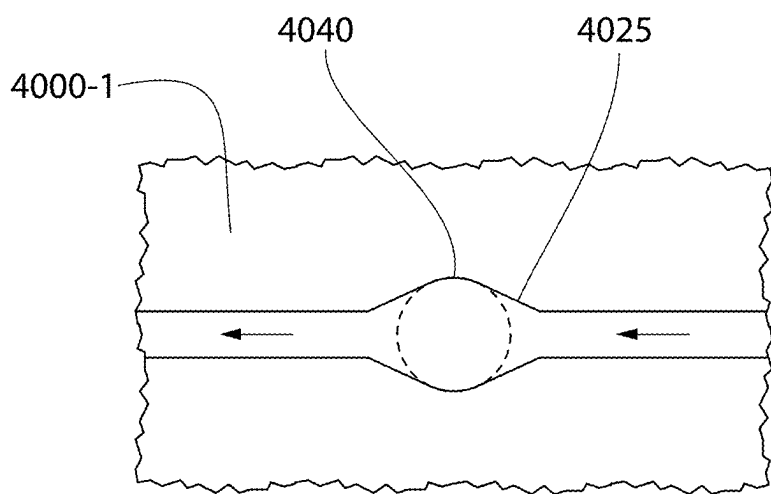


FIG. 21

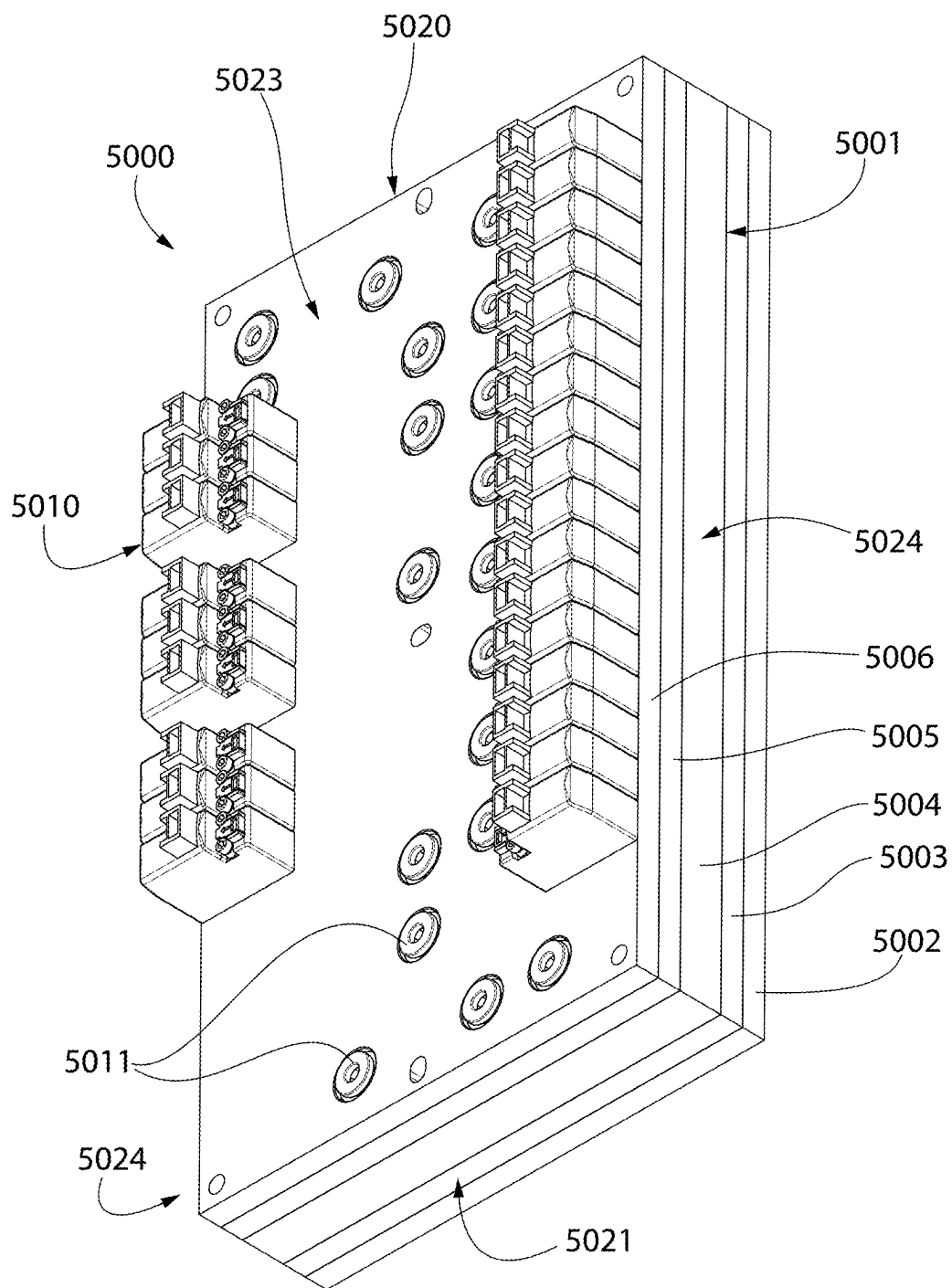


FIG. 22

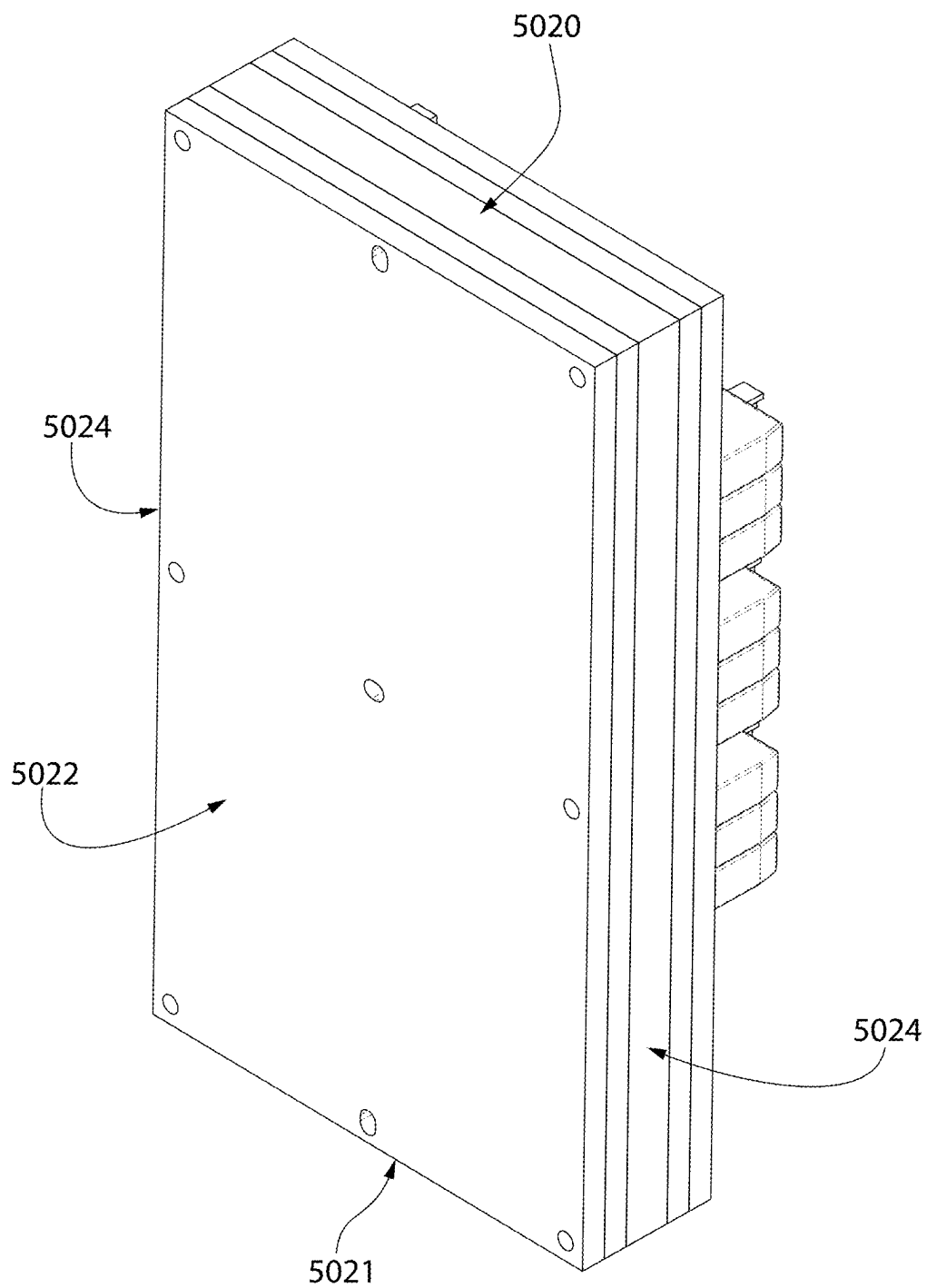


FIG. 23

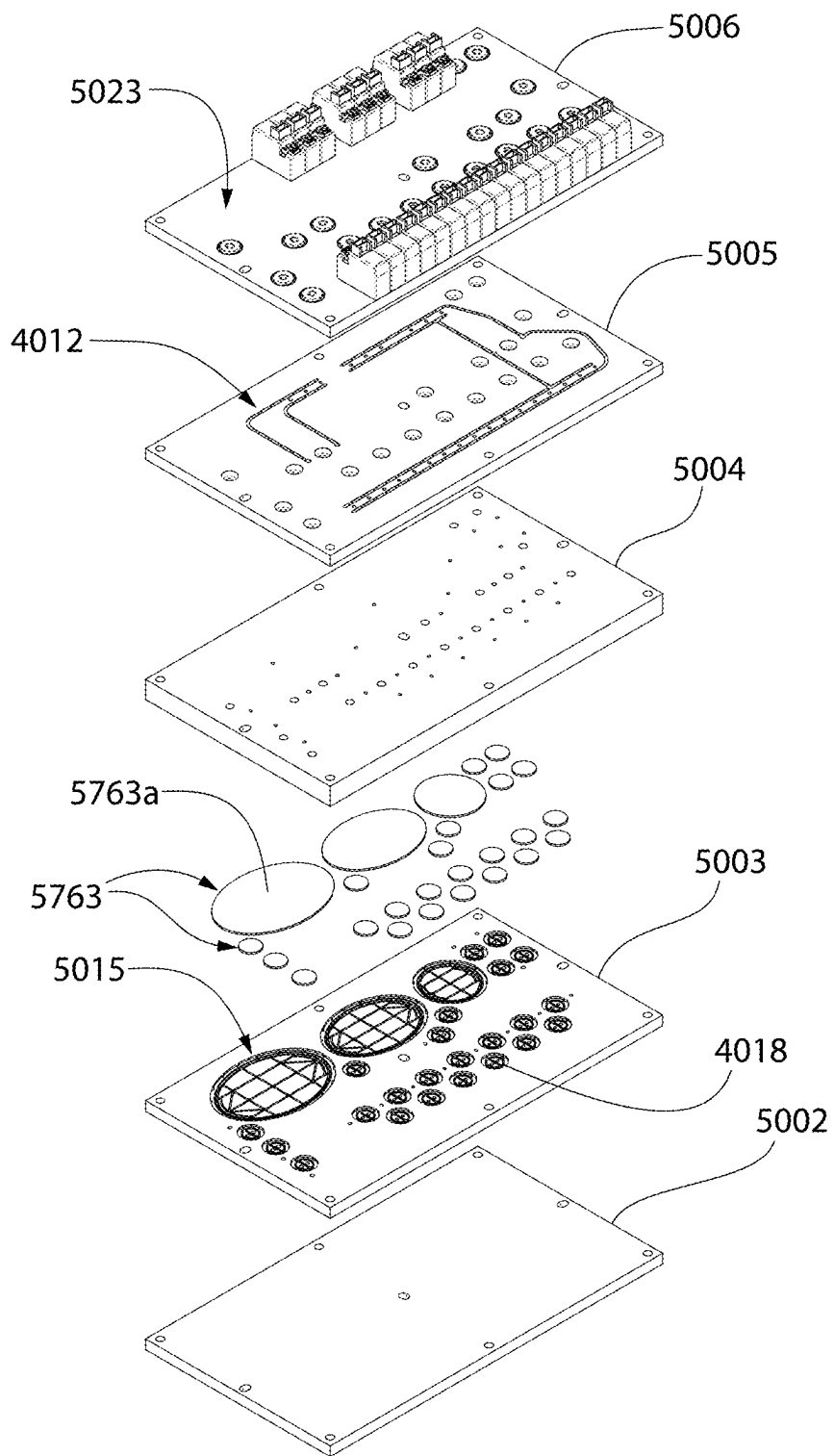


FIG. 24

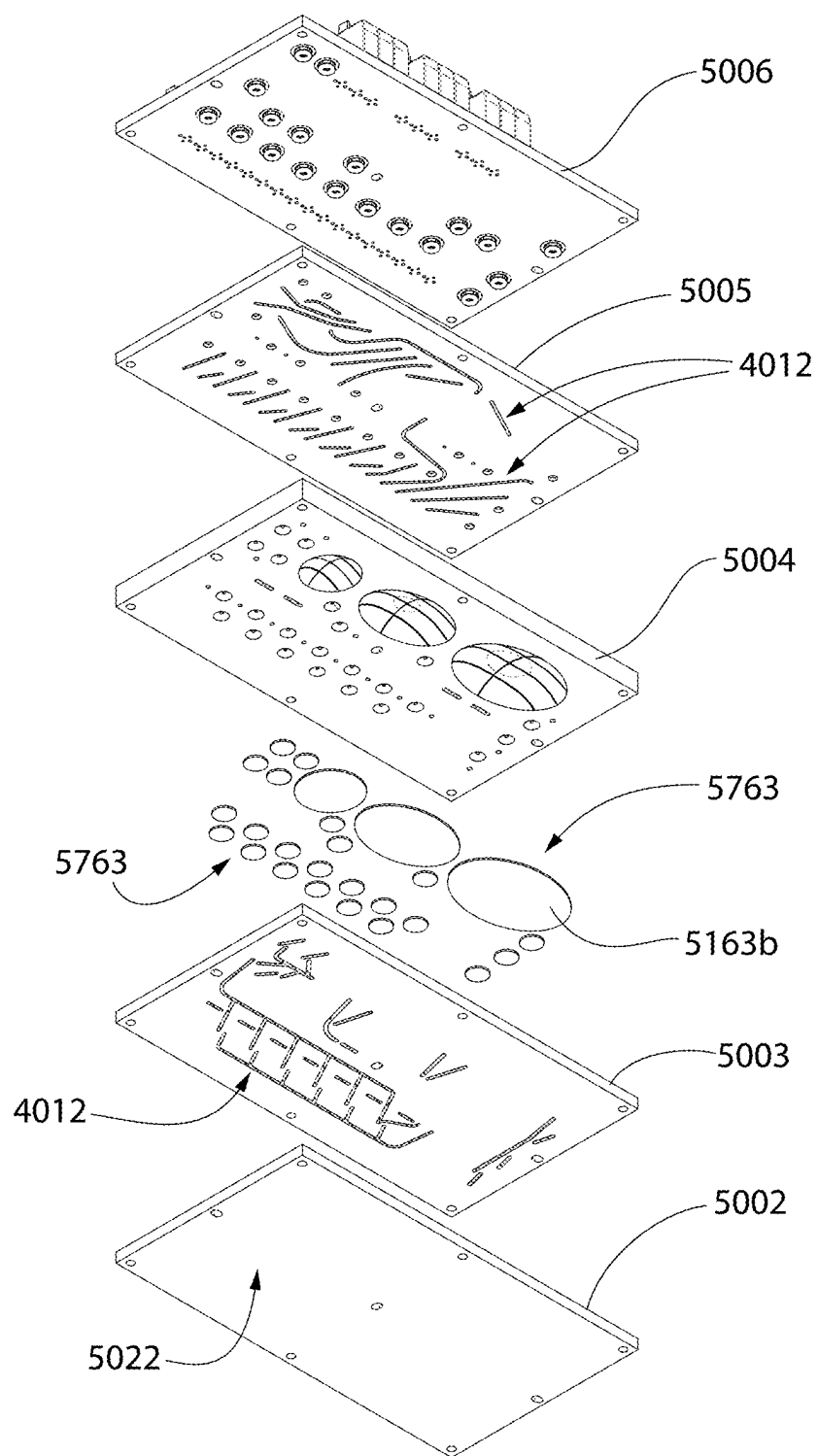


FIG. 25

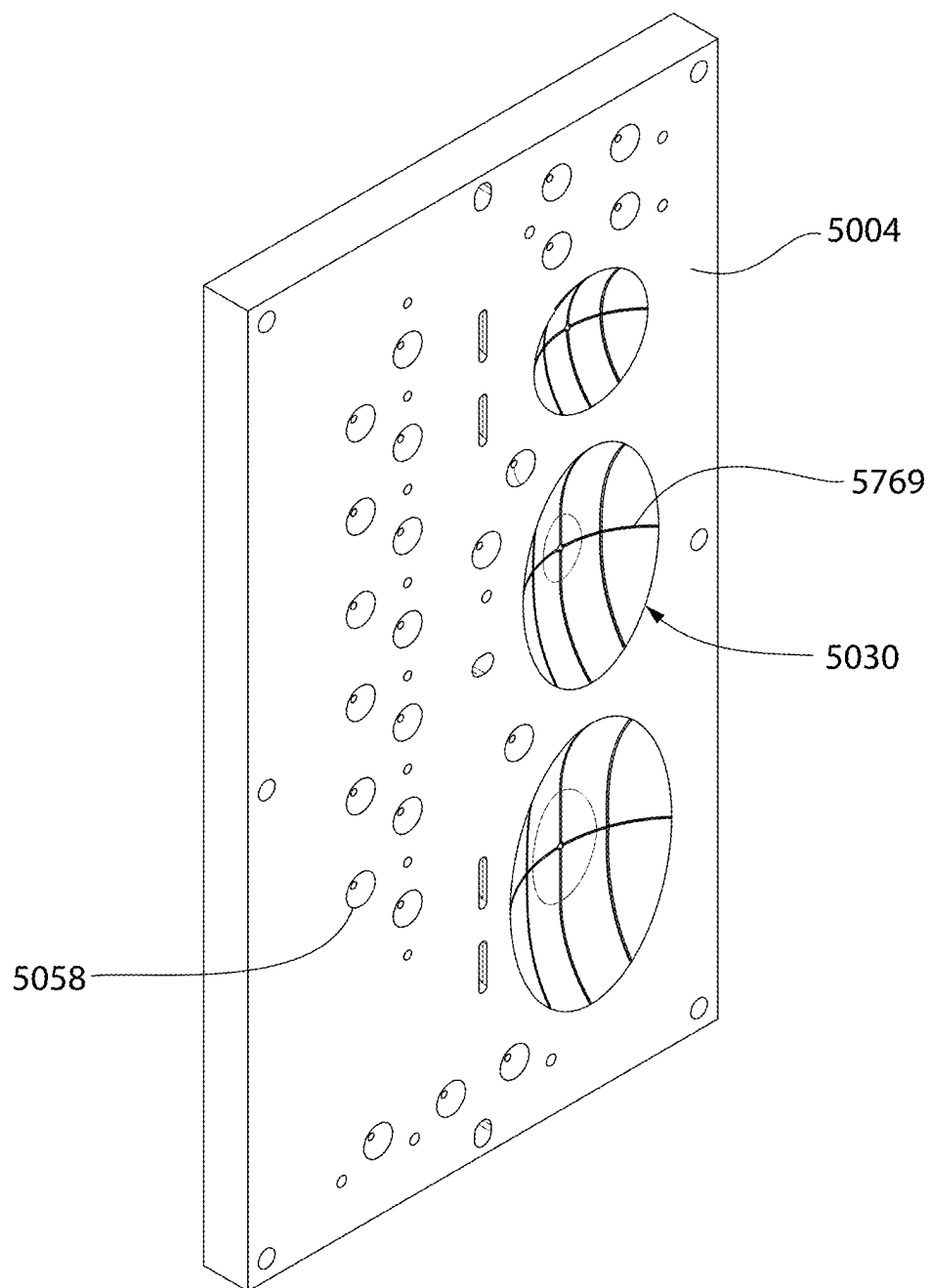


FIG. 26

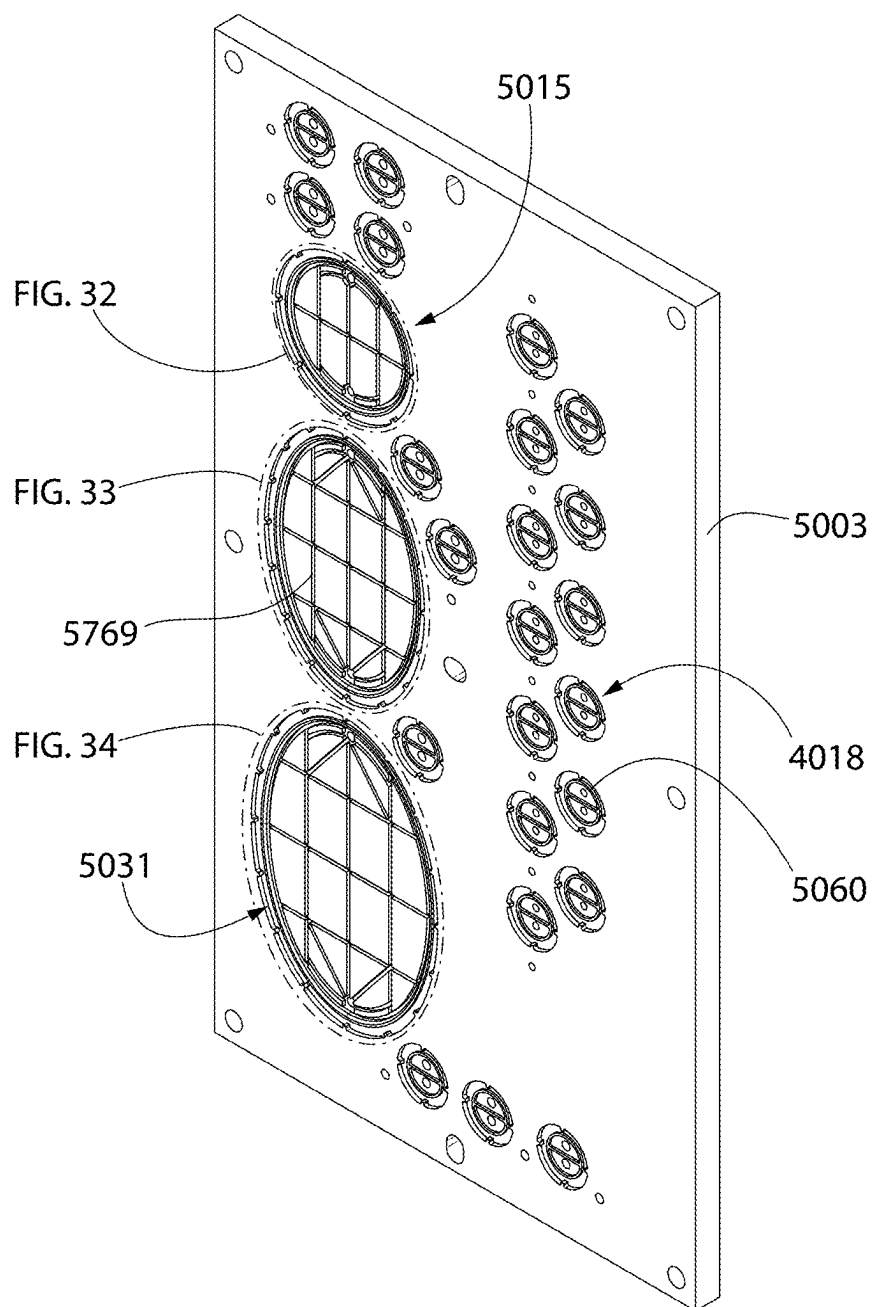


FIG. 27

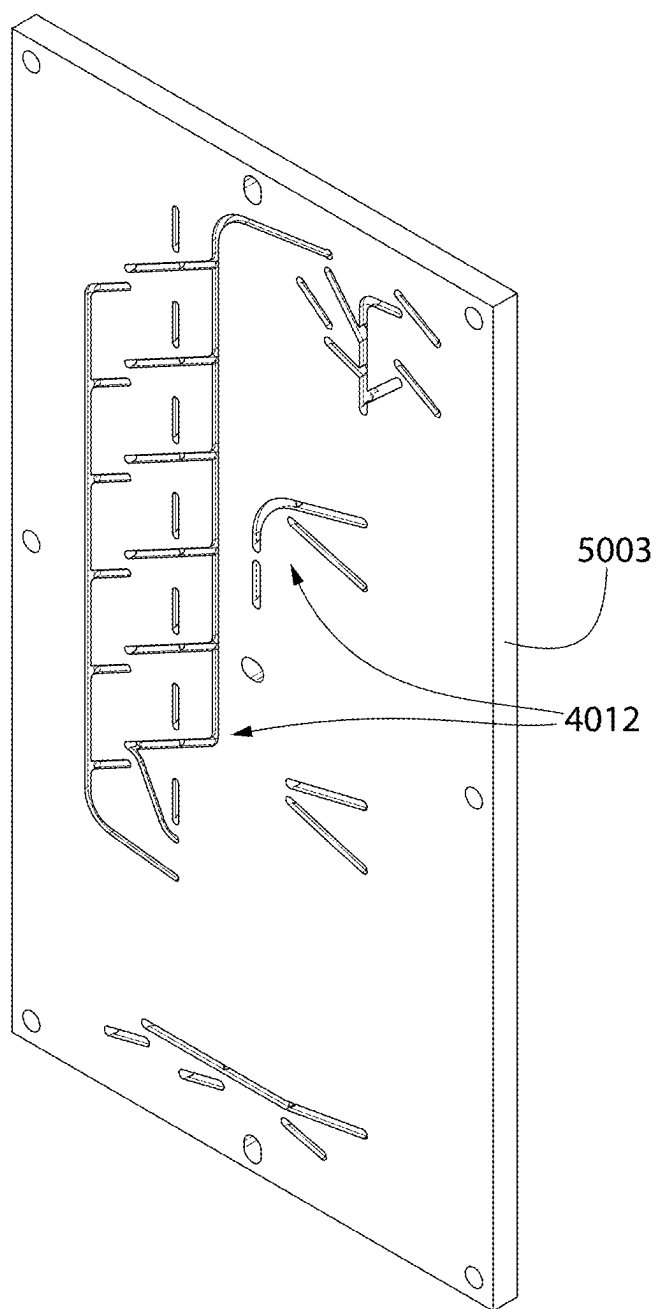


FIG. 28

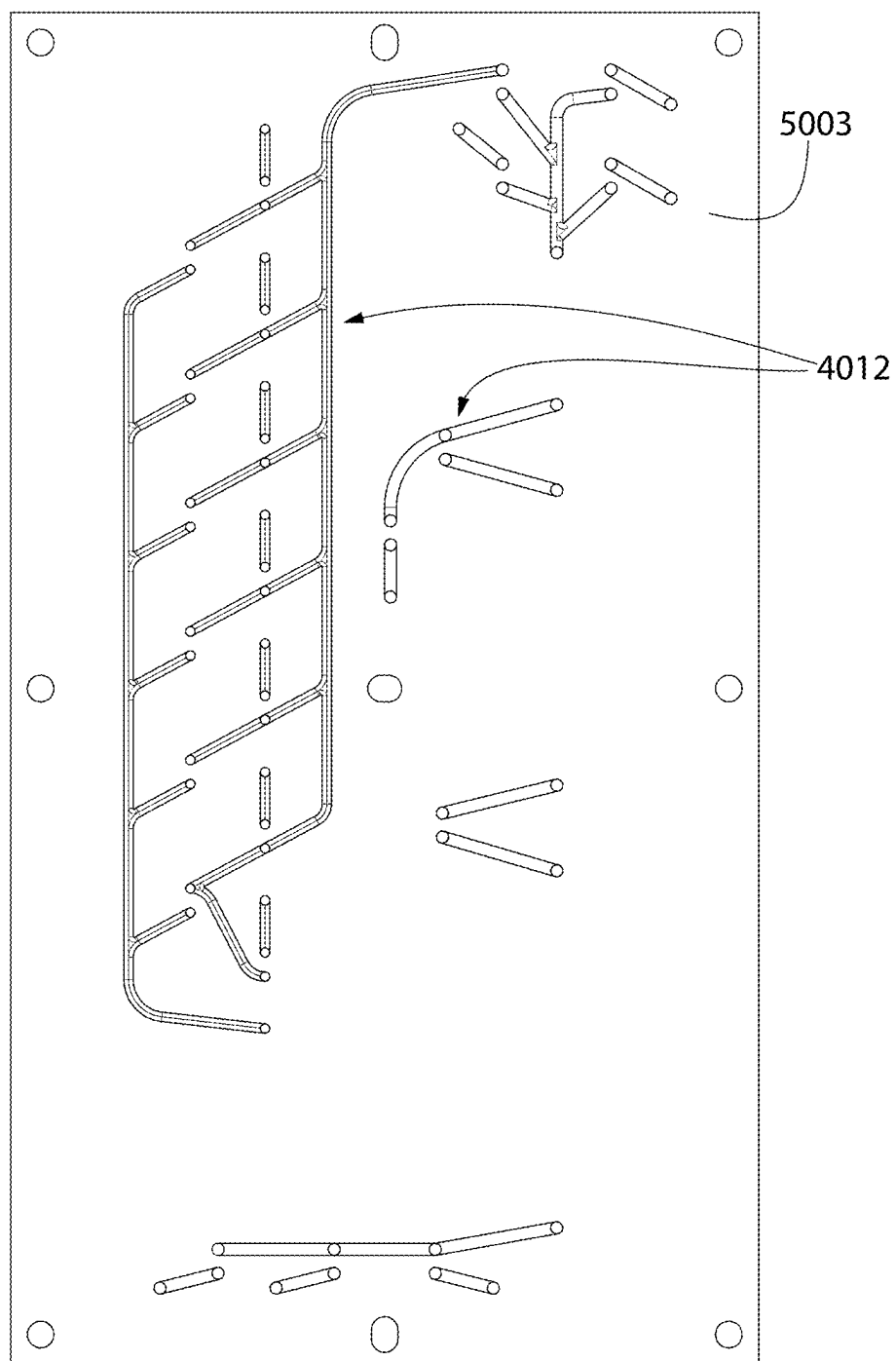


FIG. 29

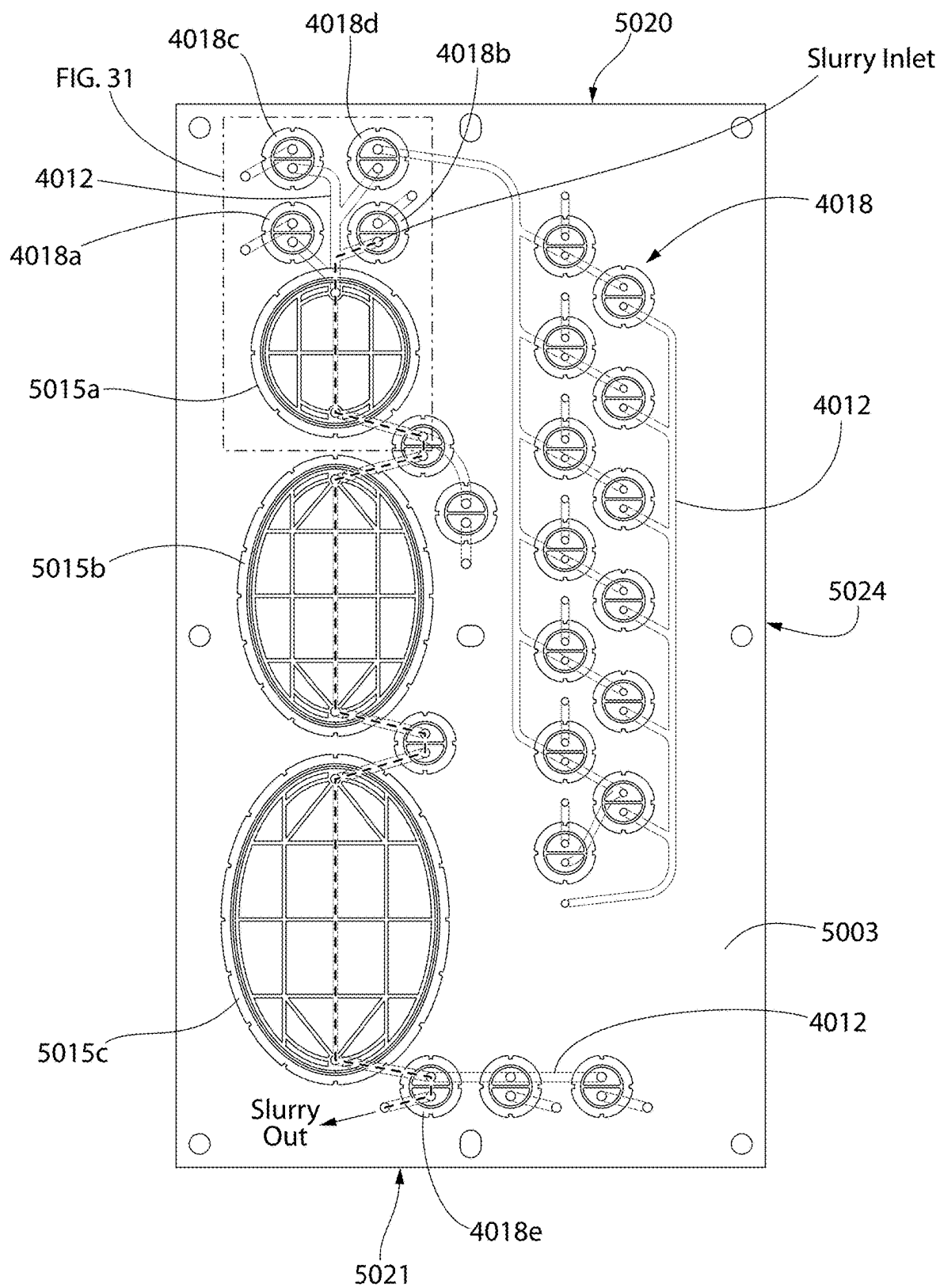


FIG. 30

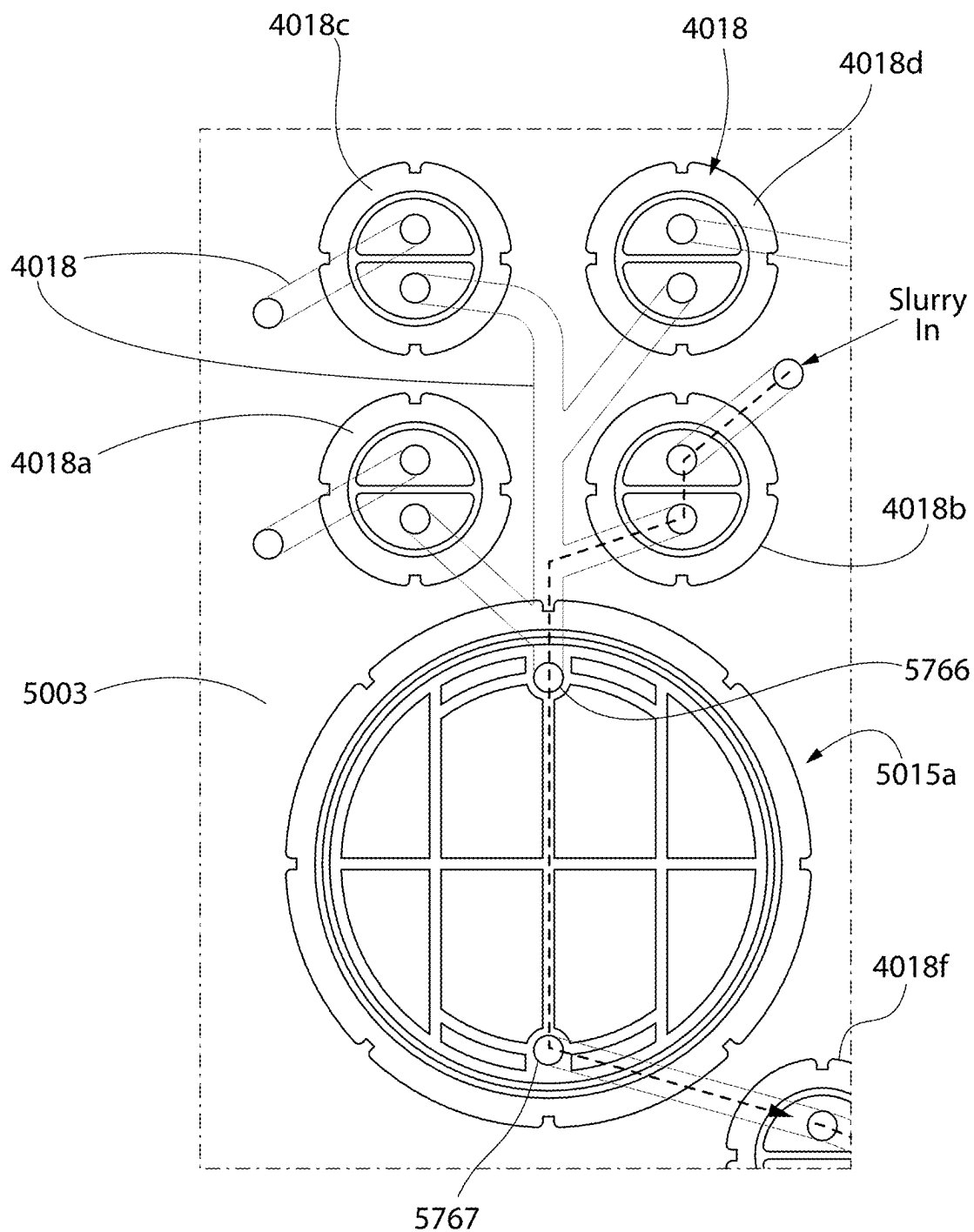


FIG. 31

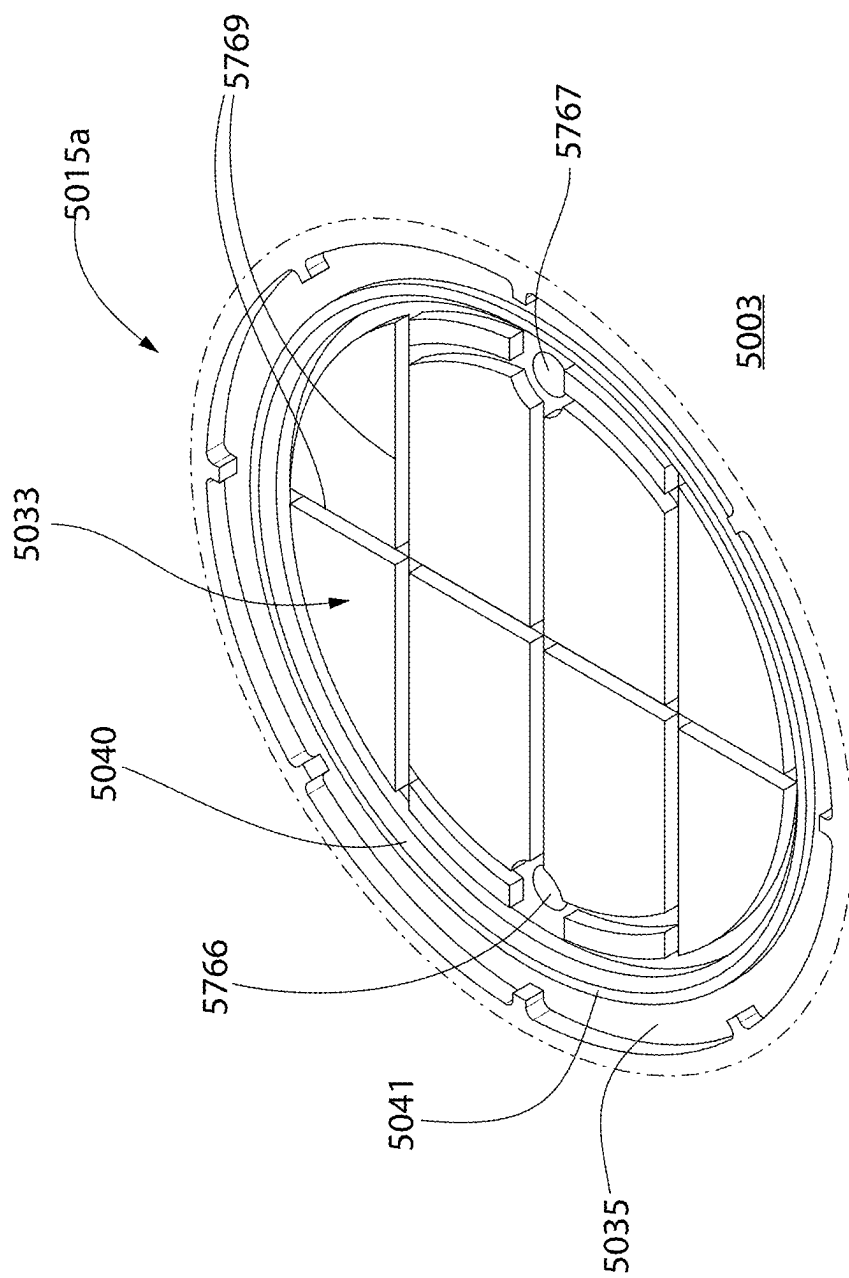


FIG. 32

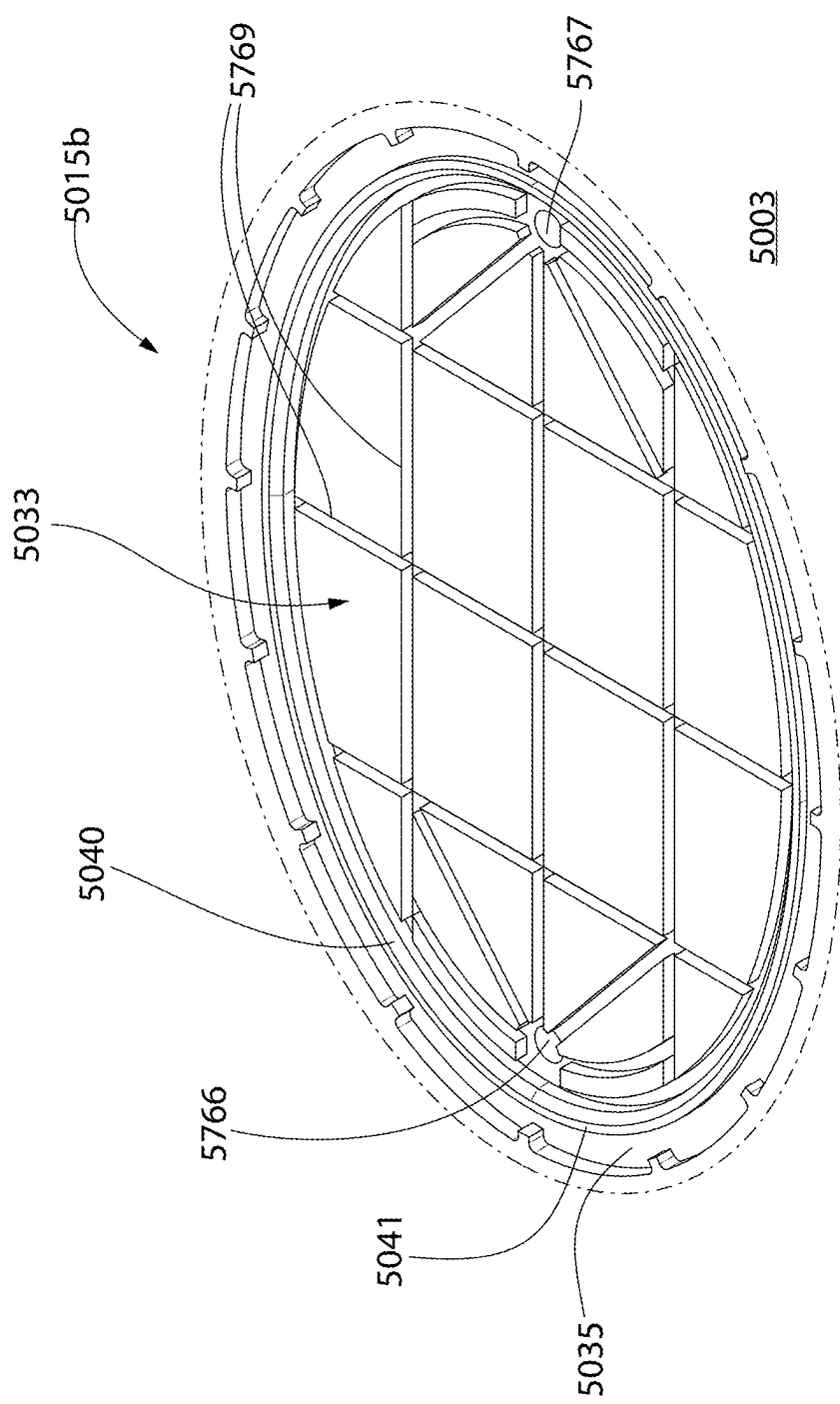


FIG. 33

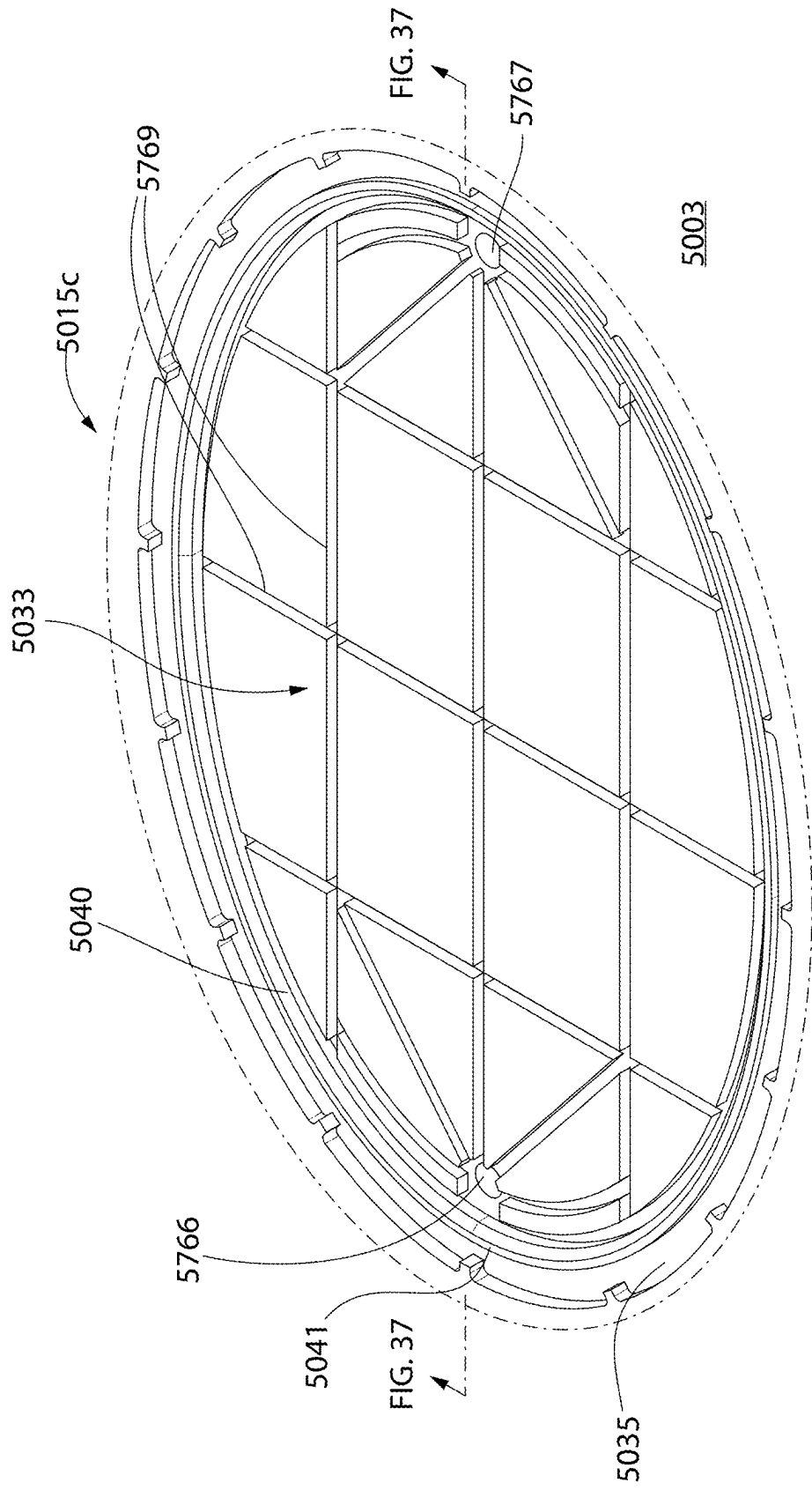


FIG. 34

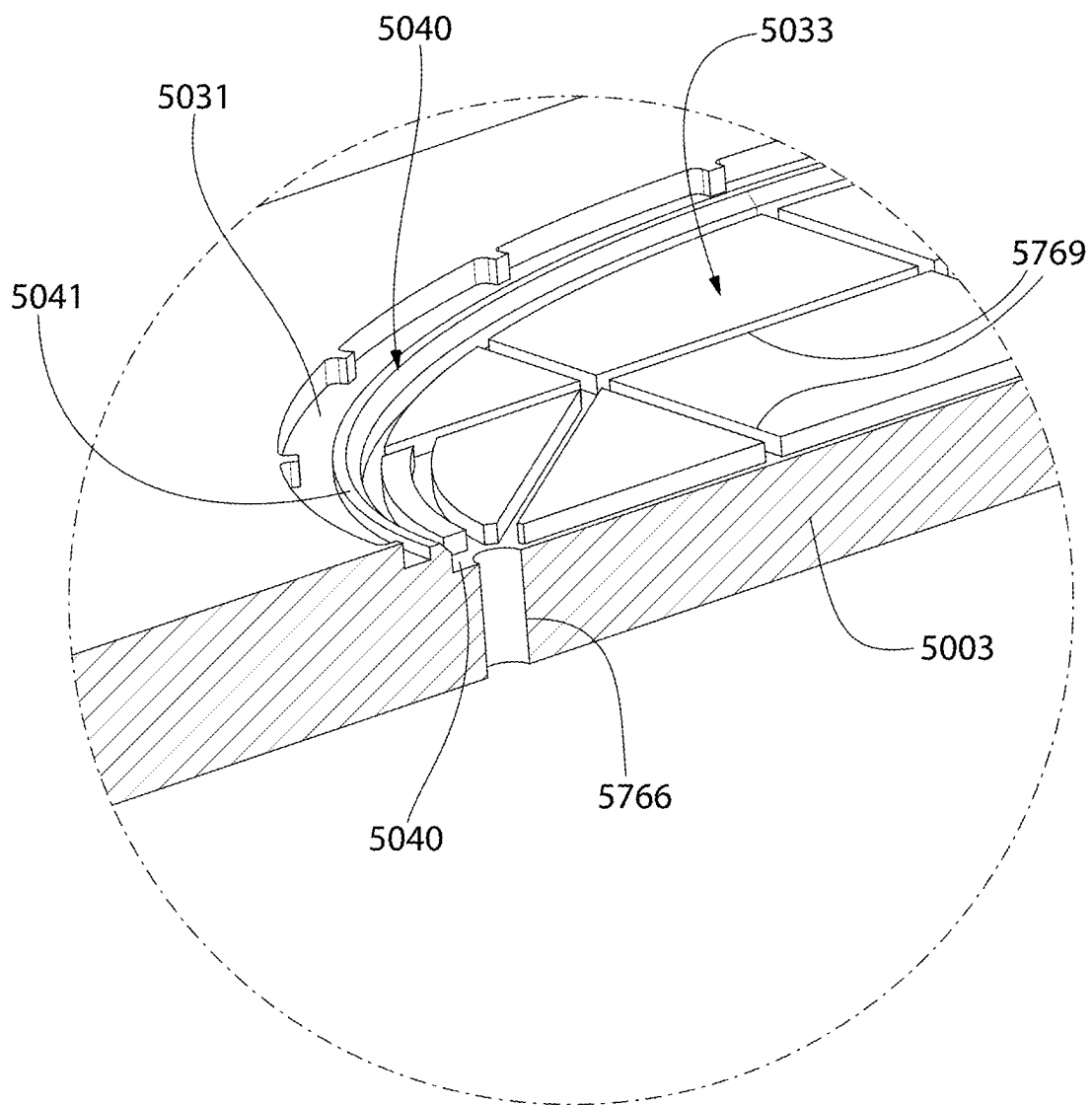


FIG. 35A

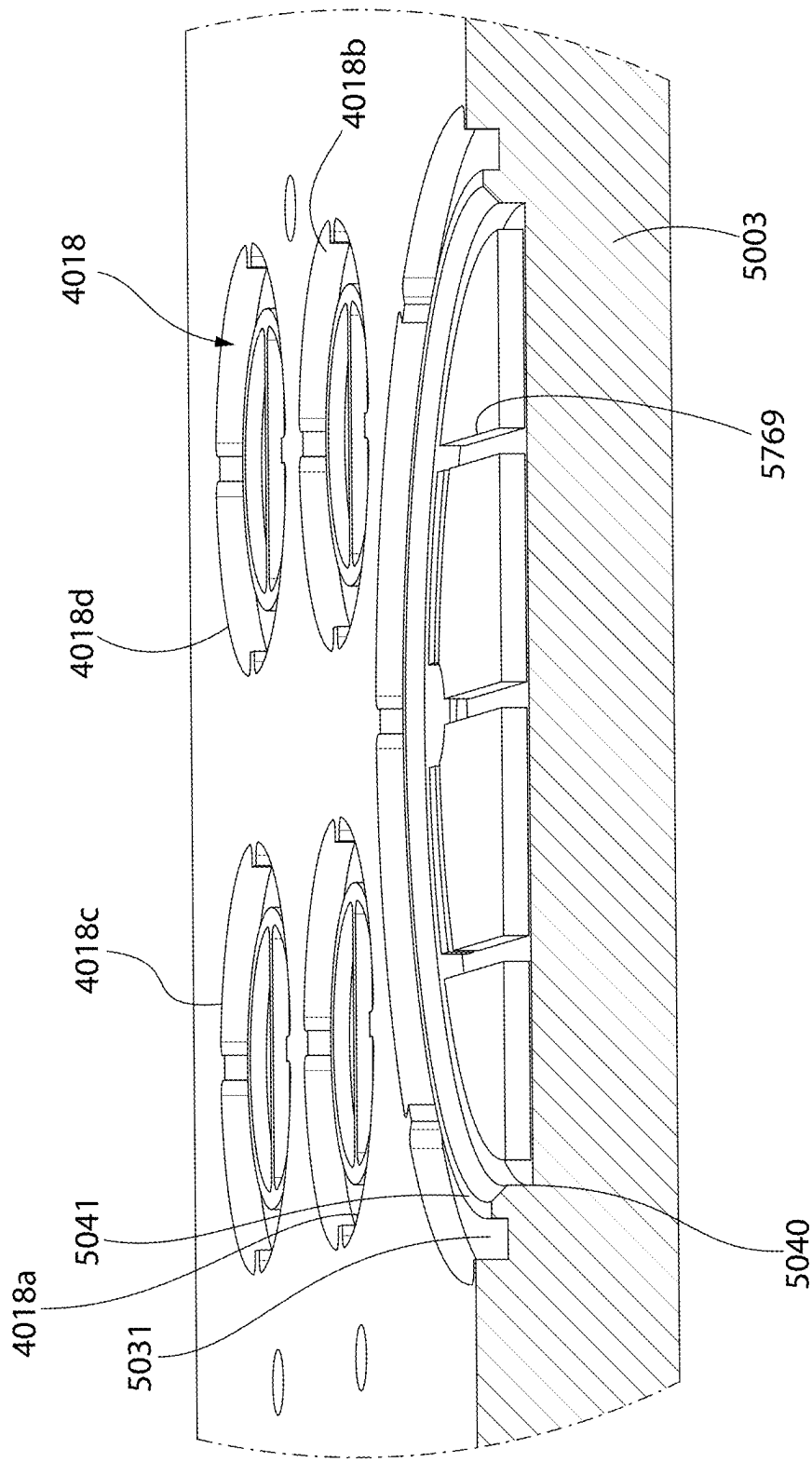


FIG. 35B

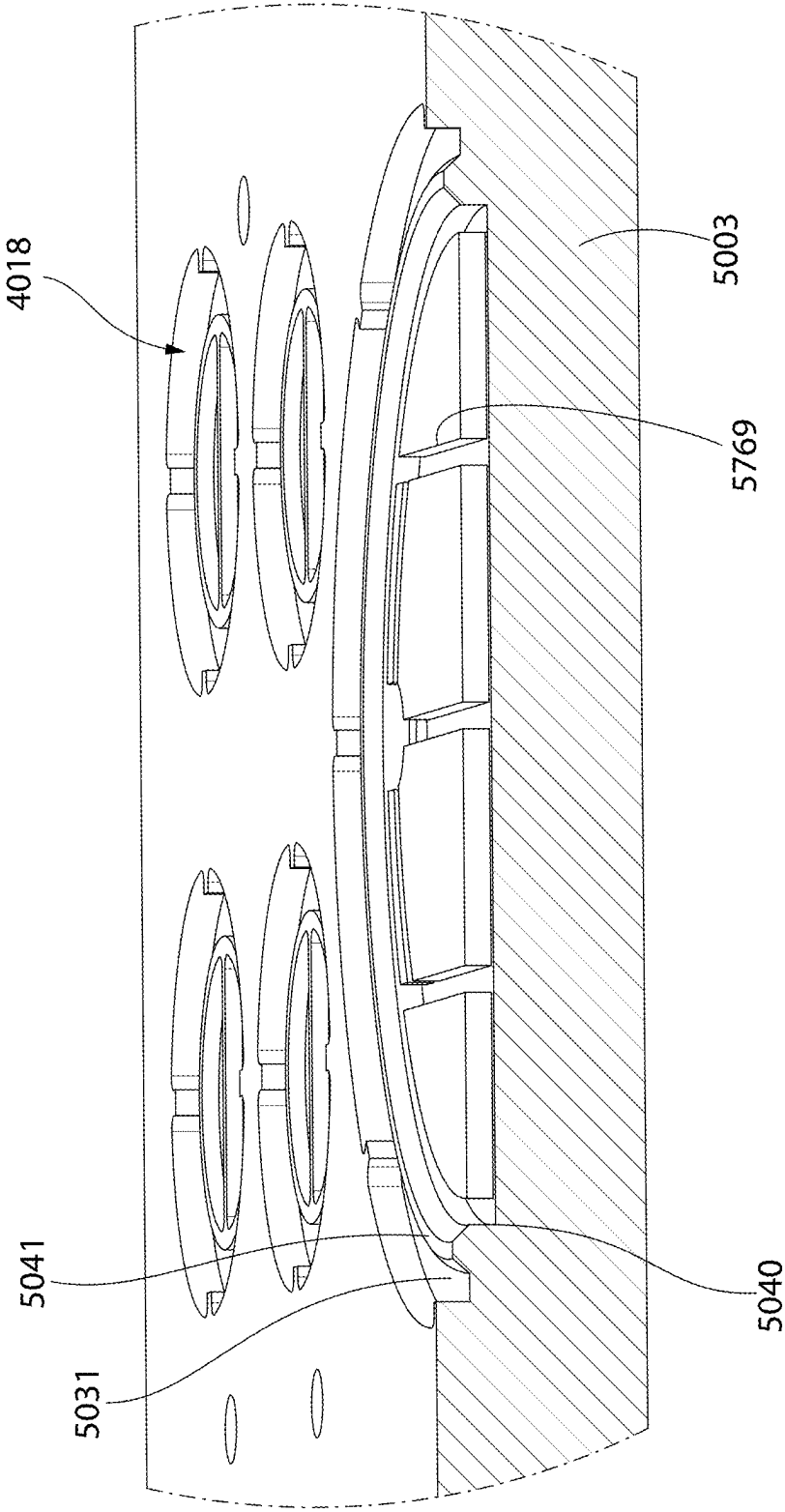


FIG. 35C

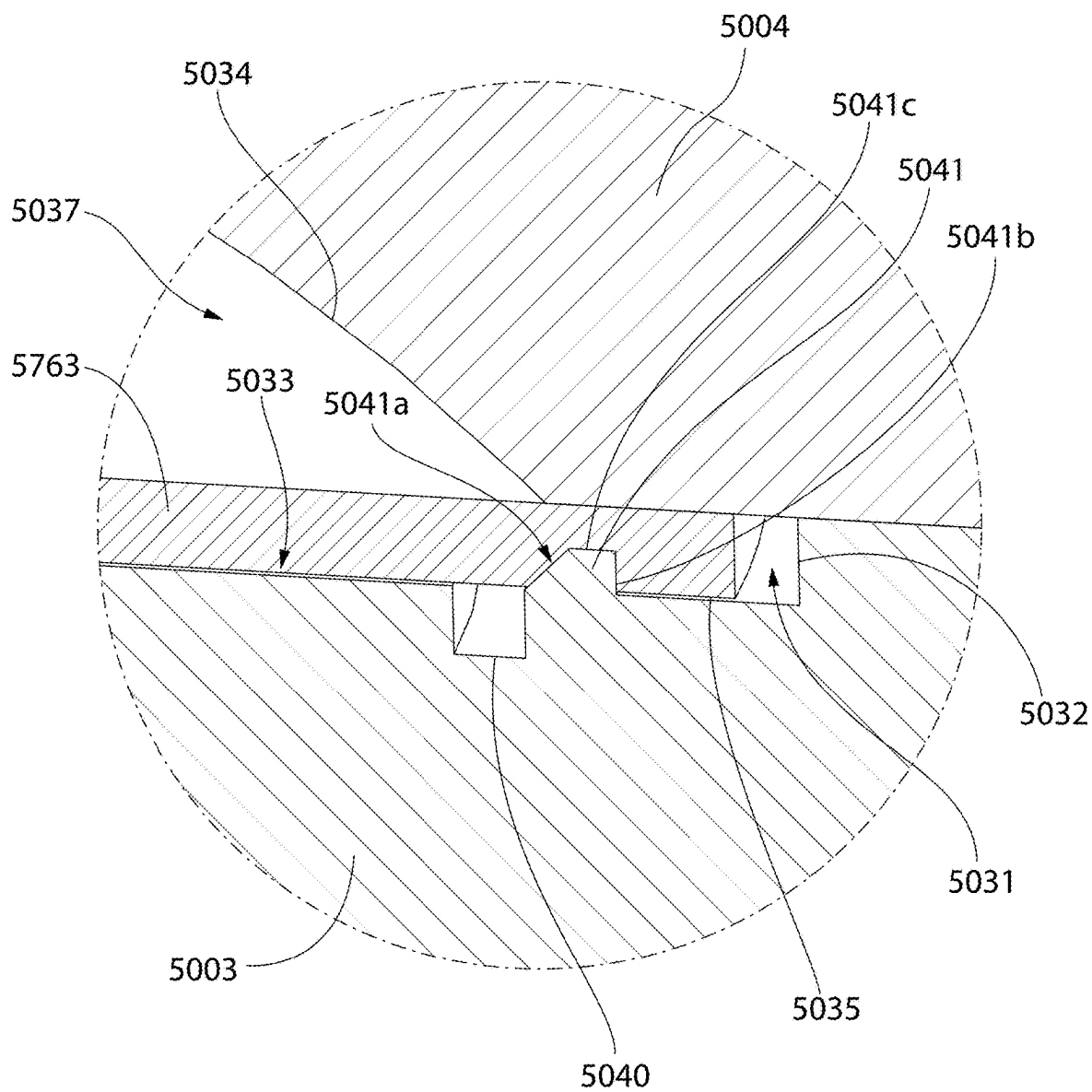


FIG. 36

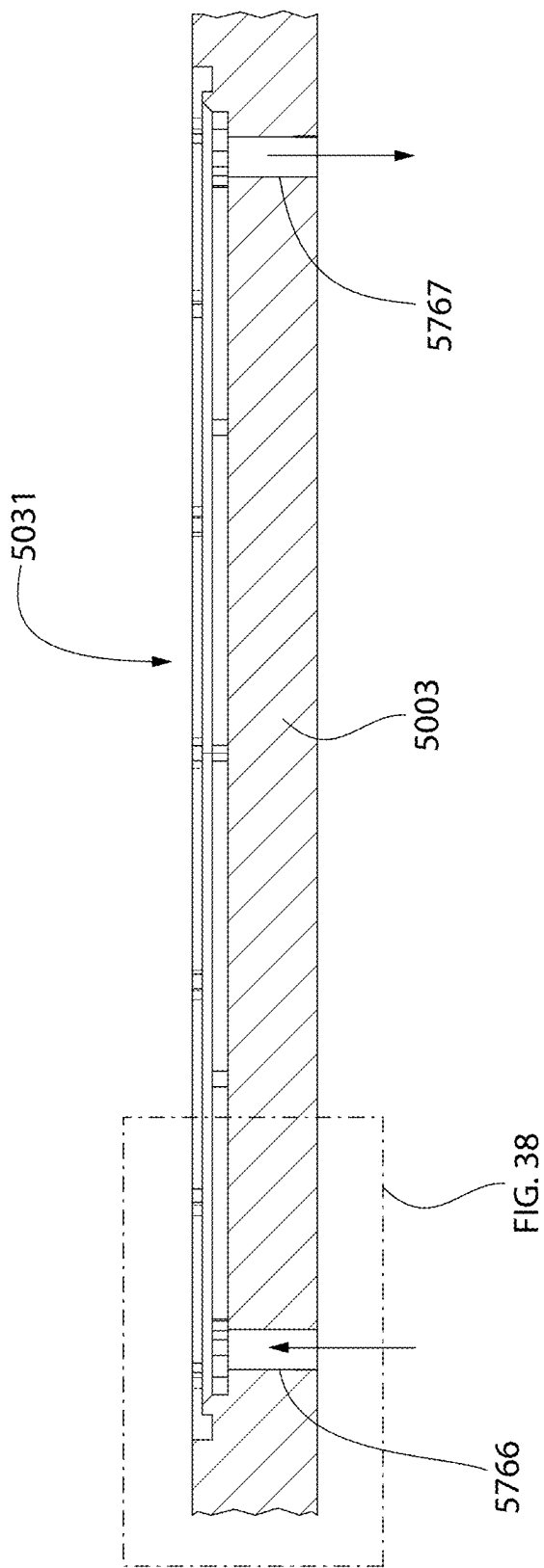


FIG. 37

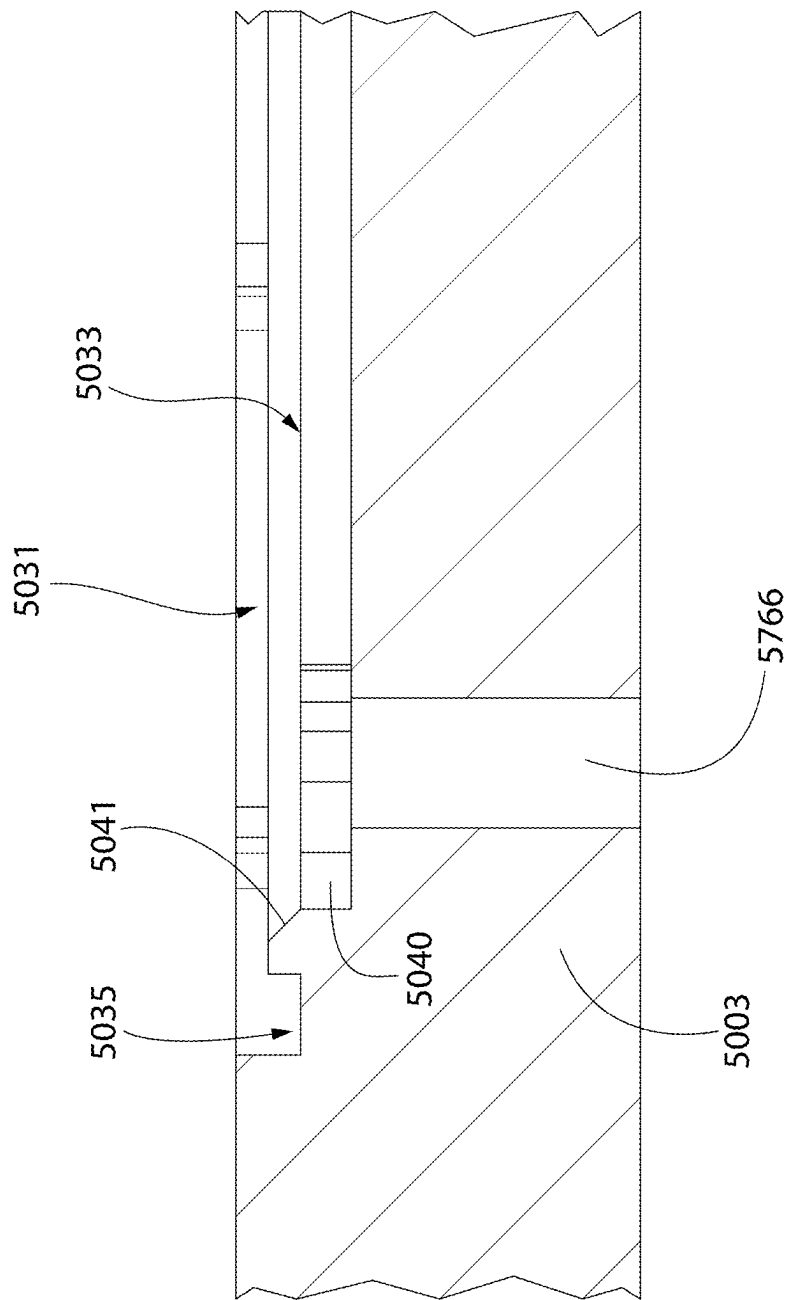


FIG. 38

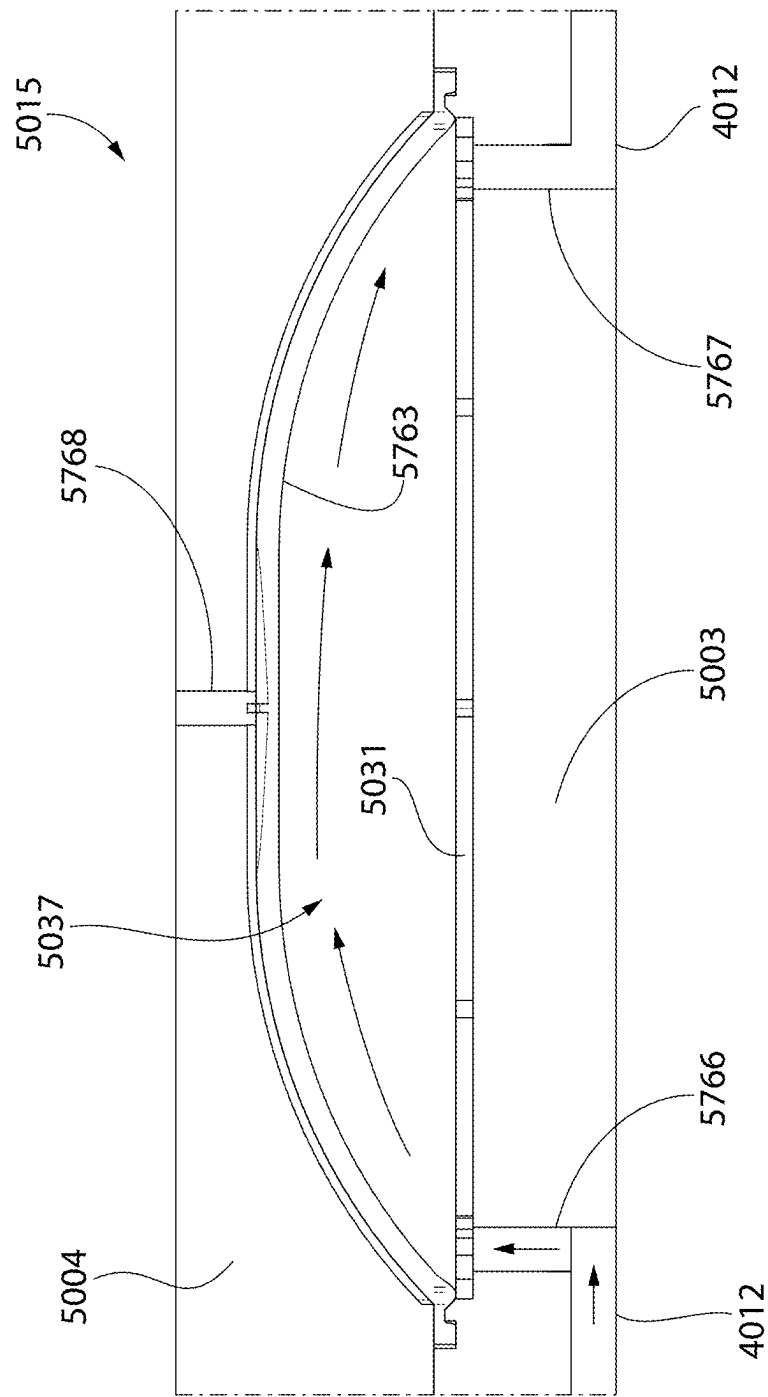


FIG. 39

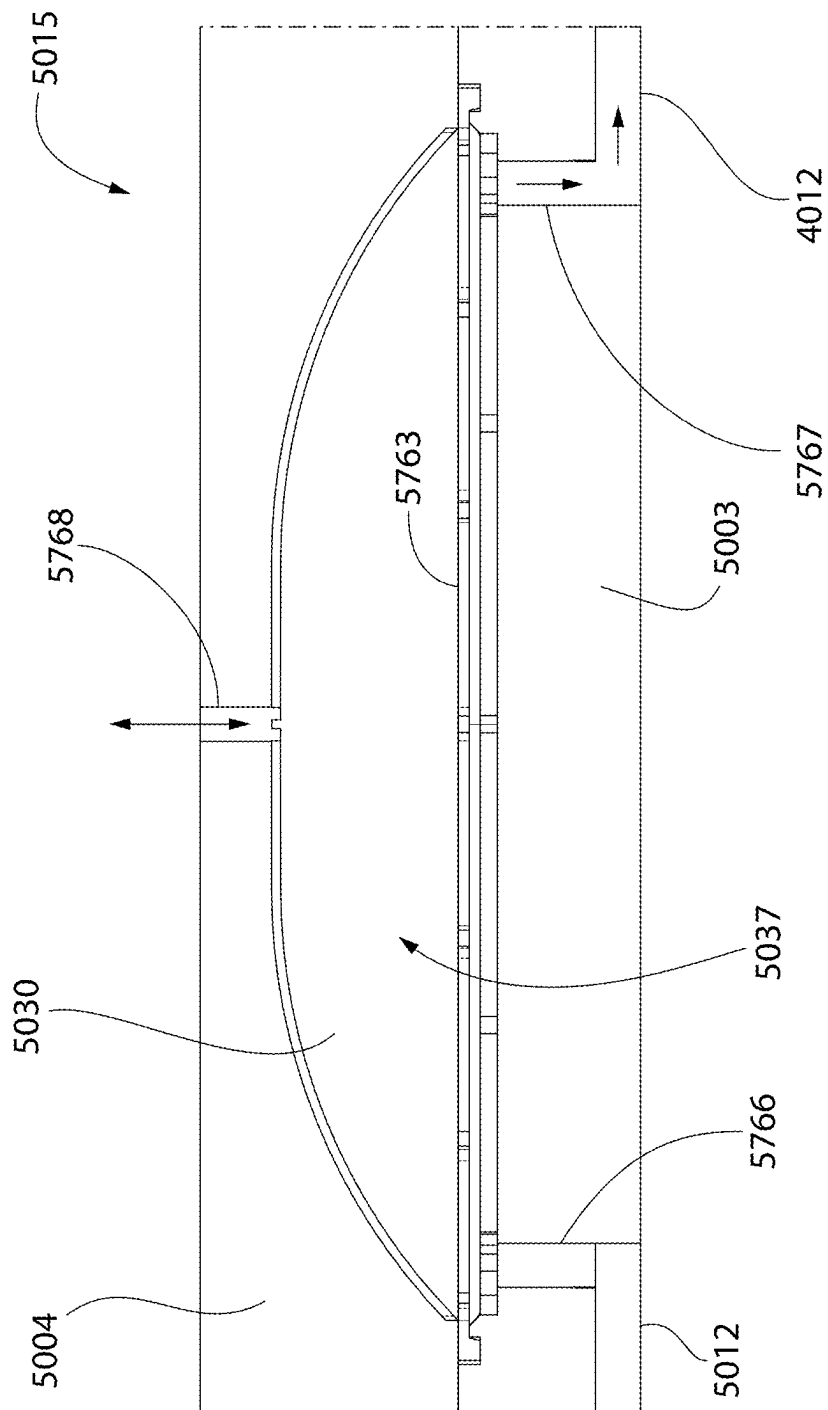


FIG. 40

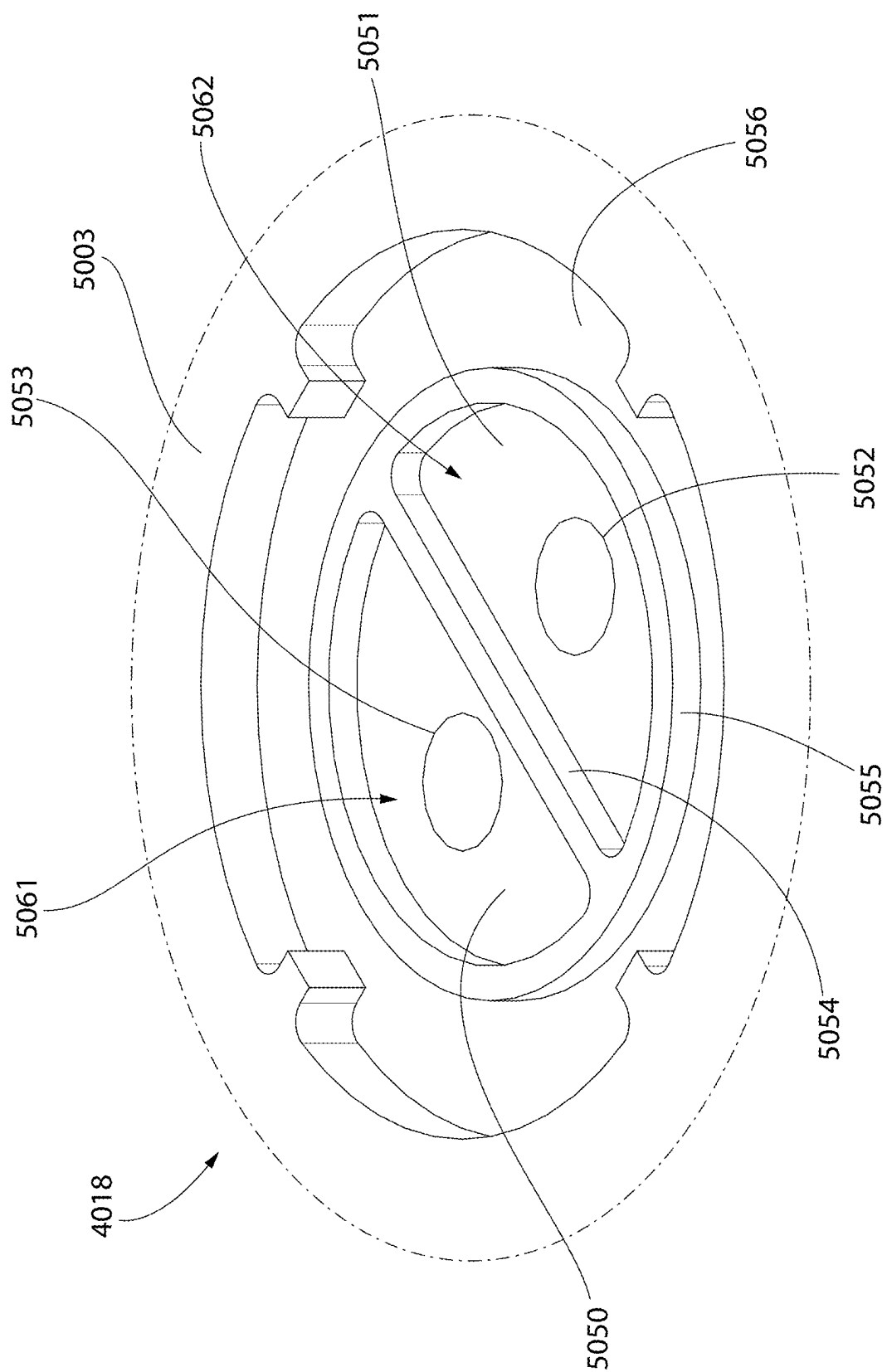


FIG. 41

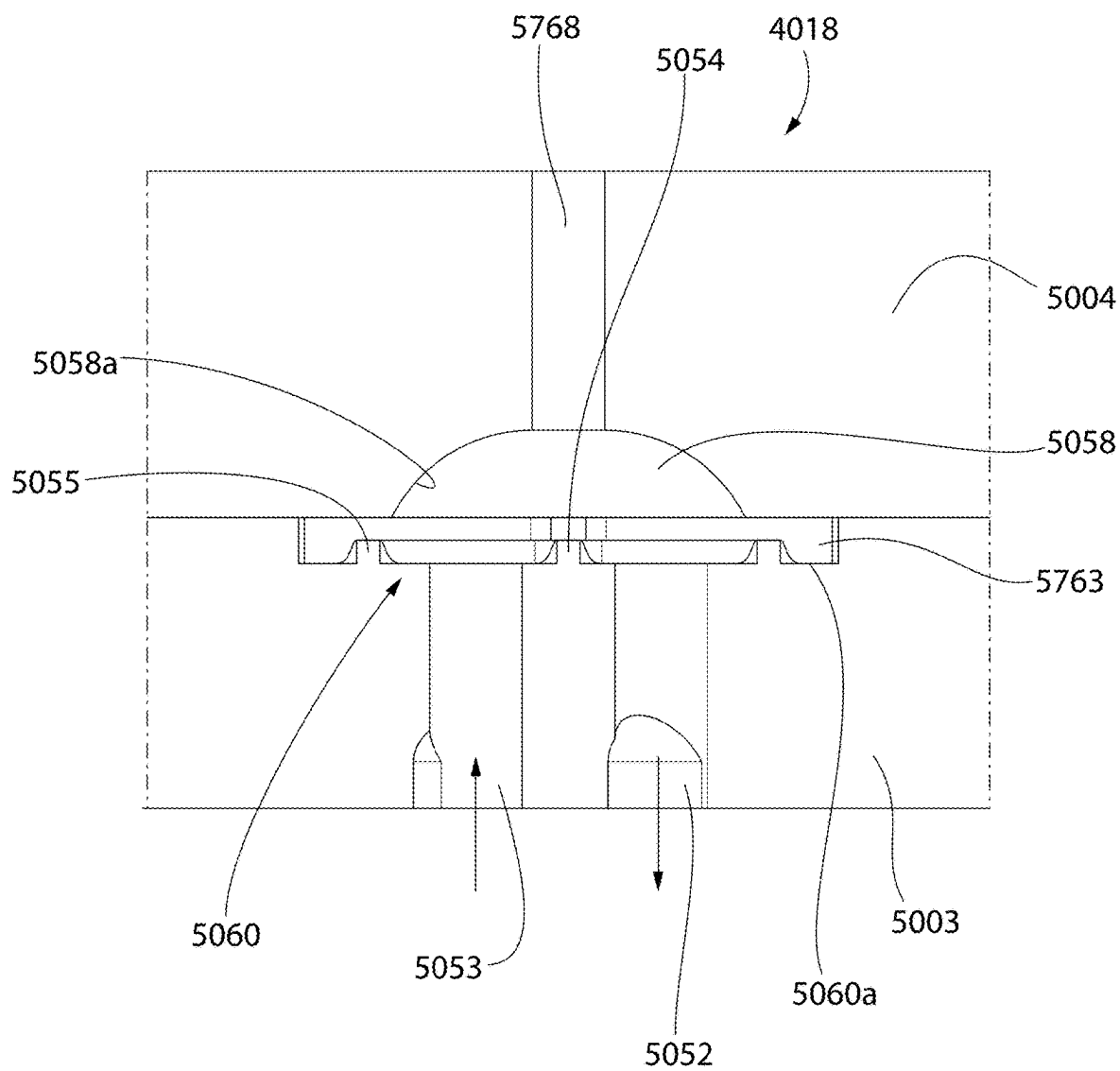


FIG. 42

AGRICULTURAL SAMPLING SYSTEM AND RELATED METHODS

CROSS REFERENCE TO RELATED APPLICATIONS

[0001] This application is a continuation of U.S. application Ser. No. 18/756,467, filed 27 Jun. 2024, which is a continuation of U.S. application Ser. No. 17/343,536, filed 9 Jun. 2021, which is a continuation in part of International Application Nos. PCT/IB 2021/052872, filed 7 Apr. 2021; PCT/IB2021/052874, filed 7 Apr. 2021; PCT/IB 2021/052875, filed 7 Apr. 2021; and PCT/IB2021/052876, filed 7 Apr. 2021 each of which claim priority to U.S. Provisional Application Nos. 63/017,789, filed 30 Apr. 2020; 63/017840, filed 30 Apr. 2020; 63/018120, filed 30 Apr. 2020; and 63/018153, filed 30 Apr. 2020, the disclosure of all of the preceding applications are incorporated herein by reference in their entireties.

BACKGROUND

[0002] The present invention relates generally to agricultural sampling and analysis, and more particularly to a fully automated system for performing soil and other types of agricultural related sampling and chemical property analysis.

[0003] Periodic soil testing is an important aspect of the agricultural arts. Test results provide valuable information on the chemical makeup of the soil such as plant-available nutrients and other important properties (e.g. levels of nitrogen, magnesium, phosphorous, potassium, pH, etc.) so that various amendments may be added to the soil to maximize the quality and quantity of crop production.

[0004] In some existing soil sampling processes, collected samples are dried, ground, water is added, and then filtered to obtain a soil slurry suitable for analysis. Extractant is added to the slurry to pull out plant available nutrients. The slurry is then filtered to produce a clear solution or supernatant which is mixed with a chemical reagent for further analysis.

[0005] Improvements in testing soil, vegetation, and manure are desired.

BRIEF SUMMARY

[0006] The present invention provides an automated computer-controlled sampling system and related methods for collecting, processing, and analyzing soil samples for various chemical properties such as plant available nutrients (hereafter referred to as a “soil sampling system”). The sampling system allows multiple samples to be processed and analyzed for different analytes (e.g. plant-available nutrients) and/or chemical properties (e.g. pH) in a simultaneous concurrent or semi-concurrent manner, and in relatively continuous and rapid succession. Advantageously, the system can process soil samples in the “as collected” condition without the drying and grinding steps previously described.

[0007] The present system generally includes a sample preparation sub-system which receives soil samples collected by a probe collection sub-system and produces a slurry (i.e. mixture of soil, vegetation, and/or manure and water) for further processing and chemical analysis, and a chemical analysis sub-system which receives and processes the prepared slurry samples from the sample preparation

sub-system for quantification of the analytes and/or chemical properties of the sample. The described chemical analysis sub-system can be used to analyze soil, vegetation, and/or manure samples.

[0008] In one embodiment, the sample preparation system generally includes a mixer-filter apparatus which mixes the collected raw soil sample in the “as sampled” condition (e.g. undried and unground) with water to form a sample slurry. The mixer-filter apparatus then filters the slurry during its extraction from the apparatus for processing in the chemical analysis sub-system. The filter may be separate. The chemical analysis sub-system processes the slurry and performs the general functions of extractant and color-changing reagent addition/mixing, centrifugating or filtering the slurry sample via microporous filter to yield a clear supernatant, and finally sensing or analysis for detection of the analytes and/or chemical properties such as via colorimetric analysis. In various embodiments, all or part of the chemical analysis sub-system may be incorporated into one or more microfluidic devices of suitable configuration.

[0009] Although the sampling systems (e.g. sample collection, preparation, and processing) may be described herein with respect to processing soil samples which represents one category of use for the disclosed embodiments, it is to be understood that the same systems including the apparatuses and related processes may further be used for processing other types of agricultural related samples including without limitation vegetation/plant, forage, manure, feed, milk, or other types of samples. The embodiments of the invention disclosed herein should therefore be considered broadly as an agricultural sampling system. Accordingly, the present invention is expressly not limited to use with processing and analyzing soil samples alone for chemical properties of interest.

BRIEF DESCRIPTION OF THE DRAWINGS

[0010] The present invention will become more fully understood from the detailed description and the accompanying drawings, wherein like elements are labeled similarly and in which:

[0011] FIG. 1 is a schematic flow diagram of an agricultural sampling analysis system according to the present disclosure showing high-level functional aspects of each sub-system of the sampling analysis system;

[0012] FIG. 2 is a schematic flow diagram showing a first embodiment of a microfluidic flow distribution network of a microfluidic manifold and its microfluidic devices for processing an agricultural sample slurry;

[0013] FIG. 3 is a schematic flow diagram showing a second embodiment of a microfluidic flow distribution network of a microfluidic manifold and its microfluidic devices for processing an agricultural sample slurry;

[0014] FIG. 4 is a schematic system diagram of a programmable processor-based central processing unit (CPU) or system controller configured and operable for controlling the microfluidic devices of the microfluidic processing manifold systems and apparatuses disclosed herein;

[0015] FIG. 5 is a top perspective view of a microfluidic processing disk with plurality of chemical analysis processing wedges each configured as a stand alone processing training for performing complete soil slurry processing and chemical analysis;

[0016] FIG. 6 is a bottom perspective view thereof;

[0017] FIG. 7 is a partially exploded perspective view thereof with fluid exchange dock which fluidly couples to the microfluidic processing disk shown below;

[0018] FIG. 8 is a bottom perspective view thereof;

[0019] FIG. 9 is a side view of the microfluidic processing disk;

[0020] FIG. 10 is a top view thereof;

[0021] FIG. 11 is a bottom view thereof;

[0022] FIG. 12 is a perspective view of one processing wedge showing its flow conduits and external fluid connections;

[0023] FIG. 13 is a top perspective view of a fluid exchange dock of the centrifuge;

[0024] FIG. 14 is a bottom perspective view thereof;

[0025] FIG. 15 is a top perspective view of a cover assembly for the tube hub showing the centrifuge tubes in a non-centrifugated vertical position;

[0026] FIG. 16 is a view thereof showing the centrifuge tubes in a pivoted centrifugated horizontal position;

[0027] FIG. 17 is an exploded perspective view of an on-disk pneumatically-actuated diaphragm micropump mountable in the microfluidic processing disk of FIG. 96;

[0028] FIG. 18 is a side cross-sectional view thereof showing the micropump in an unactuated position;

[0029] FIG. 19 is a view thereof showing the micropump in an actuated position;

[0030] FIG. 20 is a side cross sectional view of an light emitting diode (LED) emitting diode assembly and LED receiving diode assembly associated with the flow analysis cell window shown in FIG. 2 or 3 for measuring an analyte;

[0031] FIG. 21 is a top cross sectional view thereof;

[0032] FIG. 22 is a first perspective view of a multi-layered microfluidic manifold slurry processing substrate for processing agricultural slurry sample;

[0033] FIG. 23 is a second perspective view thereof;

[0034] FIG. 24 is a first exploded perspective view thereof;

[0035] FIG. 25 is a second exploded perspective view thereof;

[0036] FIG. 26 is a perspective view of an air layer thereof;

[0037] FIG. 27 is a perspective view of the first side of a liquid layer thereof showing a plurality of microfluidic devices for processing the agricultural slurry sample;

[0038] FIG. 28 is a perspective view of the opposite second side of the liquid layer showing a microchannel flow network which is fluidly coupled to the microfluidic devices;

[0039] FIG. 29 is a plan view thereof;

[0040] FIG. 30 is a plan view of the first side of the liquid layer showing the microfluidic flow network superimposed to show fluidly connections between the microfluidic devices;

[0041] FIG. 31 is an enlarged detail taken from FIG. 30;

[0042] FIG. 32 is a perspective view of a microfluidic device comprising a first micropump formed in part by the liquid layer;

[0043] FIG. 33 is a perspective view of a microfluidic device comprising a second micropump formed in part by the liquid layer;

[0044] FIG. 34 is a perspective view of a microfluidic device comprising a third micropump formed in part by the liquid layer;

[0045] FIG. 35A is a cross-sectional perspective view taken through the fluid inlet port of any of the first, second,

or third micropumps showing a perimeter flow groove, diaphragm sealing ring, and anti-stall grooves of the pump chamber;

[0046] FIG. 35B is a cross-sectional perspective view of the first micropump showing a first embodiment of the diaphragm seal ring;

[0047] FIG. 35C is a cross-sectional perspective view of the first micropump showing a second embodiment of the diaphragm seal ring;

[0048] FIG. 36 is an enlarged partial cross-sectional perspective view of the first micropump showing the first embodiment of the diaphragm seal ring in greater detail;

[0049] FIG. 37 is a cross-sectional view of the third micropump taken through the liquid layer and the fluid inlet and outlet ports of the micropump;

[0050] FIG. 38 is an enlarged detail taken from FIG. 37;

[0051] FIG. 39 is a first cross-sectional view of one of the micropumps showing the micropump in the fill or intake stroke of the pump during operation;

[0052] FIG. 40 is a second cross-sectional view thereof showing the micropump in the discharge stroke of the pump during operation; and

[0053] FIG. 41 is a perspective view another microfluidic device comprising a microvalve formed in part in the liquid layer;

[0054] FIG. 42 is a cross-sectional view of the fully assembled microvalve embedded between the liquid layer and an air layer of the microfluidic manifold.

[0055] All drawings are not necessarily to scale. Components numbered and appearing in one figure but appearing un-numbered in other figures are the same unless expressly noted otherwise. A reference herein to a figure by a whole figure number which appears in multiple figures bearing the same whole number but with different alphabetical suffixes shall be construed as a general reference to all of those figures unless expressly noted otherwise.

DETAILED DESCRIPTION

[0056] The features and benefits of the invention are illustrated and described herein by reference to exemplary (“example”) embodiments. This description of exemplary embodiments is intended to be read in connection with the accompanying drawings, which are to be considered part of the entire written description. Accordingly, the disclosure expressly should not be limited to such exemplary embodiments illustrating some possible non-limiting combination of features that may exist alone or in other combinations of features.

[0057] In the description of embodiments disclosed herein, any reference to direction or orientation is merely intended for convenience of description and is not intended in any way to limit the scope of the present invention. Relative terms such as “lower,” “upper,” “horizontal,” “vertical,” “above,” “below,” “up,” “down,” “top” and “bottom” as well as derivative thereof (e.g., “horizontally,” “downwardly,” “upwardly,” etc.) should be construed to refer to the orientation as then described or as shown in the drawing under discussion. These relative terms are for convenience of description only and do not require that the apparatus be constructed or operated in a particular orientation. Terms such as “attached,” “affixed,” “connected,” “coupled,” “interconnected,” and similar refer to a relationship wherein structures are secured or attached to one another either directly or indirectly through intervening structures, as well

as both movable or rigid attachments or relationships, unless expressly described otherwise.

[0058] As used throughout, any ranges disclosed herein are used as shorthand for describing each and every value that is within the range. Any value within the range can be selected as the terminus of the range. In addition, all references cited herein are hereby incorporated by referenced in their entireties. In the event of a conflict in a definition in the present disclosure and that of a cited reference, the present disclosure controls.

[0059] FIG. 1 is a high level schematic diagram flow chart describing the functional aspects of an agricultural sampling system **3000** according to the present disclosure. The system includes multiple sub-systems which operate in concert and sequence. The sub-systems disclosed herein collectively provide complete processing and chemical analysis of soil samples from collection in the agricultural field, sample preparation and processing, and final chemical analysis. The agricultural material sampled may be soil in one embodiment; however, other types of agricultural materials may be processed and analyzed in the same system including without limitation vegetation/plants, crop residues, forage, manure, feed, milk, and other agricultural related materials of interest in the agricultural, livestock, dairy or similar arts. In the context of soil sampling for example which is important to crop production and yield, the agricultural sampling system **3000** advantageously allows multiple samples to be processed and chemically analyzed simultaneously for different various plant-available nutrients or other parameters such as for example without limitation pH, BpH (buffer pH), etc. This information may be used to generate nutrient/parameter maps for the agricultural field to determine the appropriate quantities of soil amendments needed in different regions of the field to maximize overall crop production.

[0060] In one embodiment, portions of the agricultural sampling system **3000** may be incorporated onboard a motorized sampling vehicle configured to traverse an agricultural field for collecting and processing soil samples from various zones of the field. This allows a comprehensive nutrient and chemical profile of the field to be accurately generated “on-the-fly” in order to quickly and conveniently identify the needed soil amendments and application amounts necessary in real-time for each zone or region of the field based on quantification of the plant-available nutrient and/or chemical properties in the sample.

[0061] The soil sampling system **3000** generally includes a sample probe collection sub-system **3001**, a sample preparation sub-system **3002**, and a chemical analysis sub-system **3003**. The sample collection sub-system **3001** and motorized sampling vehicle are fully described in U.S. Patent Application Publication No. 2018/0124992A1. Sample collection sub-system **3001** generally performs the function of extracting and collecting soil samples from the field. The samples may be in the form of soil plugs or cores. The collected cores are transferred to a holding chamber or vessel for further processing by the sample preparation sub-system **3002**. Other sampling systems are described in U.S. Application Nos. 62/983,237, filed on 28 Feb. 2020; 63/017789, filed on 30 Apr. 2020; 63/017840, filed on 30 Apr. 2020; 63/018120, filed on 30 Apr. 2020; 63/018153, filed on 30 Apr. 2020; PCT/IB 2021/051076, filed on 10 Feb. 2021; and PCT Application Nos. PCT/IB 2021/051077, filed on 10 Feb. 2021; PCT/IB 2021/052872, filed on 7 Apr. 2021; PCT/IB

2021/052874, filed on 7 Apr. 2021; PCT/IB 2021/052875, filed on 7 Apr. 2021; PCT/IB 2021/052876, filed on 7 Apr. 2021.

[0062] The sample preparation sub-system **3002** generally performs the functions of receiving the soil sample cores in a mixer-filter apparatus, volumetric/mass quantification of the soil sample, adding a predetermined quantity or volume of filtered water based on the volume/mass of soil, and mixing the soil and water mixture to produce a soil sample slurry, removing or transferring the slurry from mixer-filter apparatus, and self-cleaning the mixer-filter apparatus for processing the next available soil sample. In some embodiments, the filter may be separate from the mixer.

[0063] The chemical analysis sub-system **3003** generally performs the functions of receiving the soil slurry from a mixer-filter apparatus of sub-system **3002**, adding extractant, mixing the extractant and slurry in a first chamber to pull out the analytes of interest (e.g. plant available nutrients), centrifuging the extractant-slurry mixture to produce a clear liquid or supernatant, removing or transferring the supernatant to a second chamber, injecting a reagent, holding the supernatant-reagent mixture for a period of hold time to allow complete chemical reaction with reagent, measure the absorbance such as via colorimetric analysis, and assist with cleaning the chemical analysis equipment. In some embodiments, the chemical analysis sub-system **3003** may be embodied in a microfluidic device or apparatus, as further described herein.

[0064] The process described below and in the flow diagrams may be automatically controlled and executed by the programmable system controller **2820**. The controller may be part of a controller processing system such as that further described herein and shown in FIG. 4, or as disclosed in copending U.S. Patent Application Publication No. 2018/0124992A1. The controller **2820** is operably coupled to the components of the chemical analysis sub-system **3003** disclosed herein (e.g., pumps, valves, centrifuge, compressor (air supply), etc.) for controlling the process sequence and flow of fluids (e.g., water, air, slurry, extractant, reagent, supernatant, etc.) through the system to fully process and analyze the soil or other type agricultural sample. FIG. 4 depicts one embodiment of a programmable system controller **2820** applicable to the present application.

Supernatant Separators

[0065] In some alternative embodiments, the liquid portion may be separated from the soil sample slurry and extractant mixture to produce clear supernatant for chemical analysis using a centrifuge or suitable filter media such an ultrafine microporous filter **5757** in lieu of the centrifuge. Suitable centrifuges include centrifuge **3400** and centrifuge tubes **3450** described in commonly-owned WO2020/012369. FIG. 2 shows chemical analysis sub-system **3003** of the agricultural system **3000** operably interfaced with a centrifuge. FIG. 3 shows the same but operably interfaced with microporous filter **5757** instead for producing the supernatant.

[0066] In some embodiments, a microporous sintered metal filter media of suitable shape and structure may be used for the microporous filter **5757**. Of course, numerous other suitable types and configurations of filters may be used to suit the apparatus used to mount and retain the filter (e.g. disk shaped, cone shaped, solid cylinder shape, etc.). Other types of porous filter media may be used which are suitable

for pressure requirements of the system (e.g., polymeric, etc.). Preferably, the filter media material and shape selected are suitable for backwashing. The microporous filter media selected is configured to produce a clear supernatant suitable for chemical analysis from the slurry and extractant mixture. [0067] Once the supernatant is separated from the soil slurry, the filter may be back-flushed with clean high pressure liquid (e.g. filtered water) to clean the filter media for reuse during the next soil sample run. To accomplish a backwash cycle, the flow paths formed in the system may be reconfigured by selectively opening/closing certain valves in combination to reverse filtered water flow through the filter media of filter 5757. The filter backwash is exhausted from the system.

Microfluidic Chemical Analysis Devices

[0068] FIGS. 3 and 4 previously described herein are flow diagrams showing the fluid flow paths (e.g., slurry, water, air, chemicals, etc.) and fluidic components of the chemical analysis sub-system 3003 incorporated in an analysis processing device such as a microfluidic manifold in the form of a processing wedge 4002 in one non-limiting embodiment. When using the microporous filter 5757 in FIG. 4 in lieu of the centrifuge shown in FIG. 3, the filter may be separate from and not incorporated into the processing wedge 4002 in one implementation as an integral part thereof (dashed lines schematically connoting the boundary of the wedge). In alternative implementations, however, the filter 5757 may be configured and constructed for integrating directly into the processing wedge 4002. Since the layers of microfluidic devices are typically permanently bonded together, the non-integrated filter arrangement allows the filter media to be readily replaced as needed without having to discard the entire processing wedge. Where a compact form factor is desired due to spatial constraints, however, the integrated filter arrangement may be preferable.

[0069] A suitable external off-disk high pressure filtered water source 5757a as shown in FIG. 3 may be provided for the filter backwash operations to clean the filter between different agricultural sample runs for chemical analysis. The microporous filter 5757 may be cleaned in a similar manner to that already described herein by reversing flow instead through the filter media of filter 5757 using high pressure water in a direction opposite that that used for filtering the agricultural slurry.

[0070] FIGS. 5-12 generally depict one non-limiting embodiment of a microfluidic processing disk 4000 comprised of a plurality of chemical analysis processing devices such as wedges 4002 in one non-limiting embodiment and configuration. Each wedge 4002 comprises a fluidly isolated chemical analysis sub-system 3003 which may be operably interfaced with either a centrifuge 3400 or microporous filter 5757 previously described herein for filtering the agricultural slurry and extractant mixture to produce the clear supernatant for chemical analysis. Advantageously, the microfluidic processing disk 4000 is a microfluidic device (e.g. M2D2) which is configured and operable to integrate and incorporate the entire slurry analysis system including substantially all aspects of fluid pumping, mixing, valving, and flow distribution and control shown in FIGS. 2 and 3 associated with handling the air, water, slurry, extractant, reagent, and supernatant fluids. The pumps, valving, mixing, and flow distribution functions for example are thus integrated into each processing wedge 4002 of the microfluidic

processing disk 4000 in a known manner of constructing such microfluidic devices with active micro-components (e.g. pumps, valves, mixing chambers, etc.). This eliminates the need for the multiplicity of physically discrete and separate flow control devices (e.g. pumps, valves, mixing chambers, etc.) which conventionally need to be fluidly interconnected via extensive runs of tubing and tube connection, thereby allowing for improved compactness of the chemical processing analysis portion of the system.

[0071] The microfluidic processing disk 4000 advantageously provides single unified platform or device for processing and controlling flow of all the foregoing fluids in addition to chemical analysis and quantification of the analytes of interest extracted from the agricultural sample. The microfluidic processing disk 4000 further provides parallelization of the agricultural sample processing to reduce analysis time and quantification of all chemical parameters associated with the sample. Accordingly, the sample may be processed and chemically analyzed simultaneously in the plurality of processing wedges 4002 for all analytes of interest. Pressurized air provided by air compressor 3030 provides at least part the motive force for flowing and processing the foregoing fluids through each processing wedge of microfluidic processing disk 4000 in accordance with the flow charts of FIGS. 2 and 3, as further described herein. The air is also used to operate the micro-valving and micropumps which may be pneumatically actuated, as further described herein.

[0072] Referring initially to FIGS. 5-12, the microfluidic processing disk 4000 may have a generally annular disk-shaped composite body in one embodiment formed from multiple layers of material bonded or laminated together by any suitable means used in the art (e.g. adhesives, heat fusion, etc.). Each layer may be substantially planar or flat in the sandwiched construction, typical of such microfluidic devices (e.g. M2D2) sometimes referred to a "lab-on-a-chip." The analysis processing wedges 4002 of microfluidic device are configured and operable to meter/measure, pumps, mix, and de-bubble the various fluids (air, water, slurry, extractant, reagent, etc.). One or more of the layers of each processing wedge are configured and patterned to create micro-sized channels, chambers/reservoirs, and air actuated diaphragm-operated valves and pumps embedded in the microfluidic device.

[0073] The materials used to construct the layers of the processing wedges 4002 of microfluidic processing disk 4000 may include a combination of rigid thermoplastics and flexible elastomeric material sheets. Transparent polymeric materials may be used in one embodiment to permit visual observation of the fluids being processed in the microfluidic processing disk 4000. The rigid plastics may be used to form the overall rigid substrate or body of microfluidic processing disk 4000 which defines its exposed exterior surfaces and includes an interior patterned to create a plurality of internal microchannels 4012 and chambers for creating the active microfluidic flow control devices (e.g. diaphragm-operated pumps, valves, mixing chambers, etc.). Examples of thermoplastics (polymers) which may be used include for example without limitation PMMA (polymethyl methacrylate commonly known as acrylic), PC (polycarbonate), PS (polystyrene), and others. Examples of suitable elastomeric materials which may be used include for example without limitation silicone, PDMS (polydimethylsiloxane), fluoro-silicone, neoprene, and others. The pressurized air used to

hold the microfluidic valves/pumps closed will permeate through elastomeric diaphragms over time, causing bubbles to develop in the liquid side of the device. These bubbles negatively affect the ability to volumize liquids properly, as the air bubbles displace the otherwise precise fluid volumes that are being manipulated. Fluorosilicone is one preferred non-limiting material due its low gas permeability property which aids in decreasing gas diffusion through the diaphragm over time to combat the foregoing problem.

[0074] The elastomeric materials used in each chemical analysis processing wedge **4002** may be used to form the flexible and deformable active portions of the microfluidic flow control devices such as the movable diaphragms of the micropumps and microvalves which are acted upon by air pressure (alternatively water pressure) to operate these pumps and valves for controlling fluid flow within the microfluidic processing disk **4000**. This is typically achieved by forming a thin flexible elastomeric layer (e.g. silicon, PDMS, etc.) above a layer of the more rigid thermoplastic layer in disk **4000** which is patterned with the microchannels and microchambers associated with the pumps, valves, or mixing chambers, thereby forming a flexible roof portion thereof.

[0075] It bears noting that in some embodiments, the elastomeric material may be individually cut or otherwise formed to fit and complement the shape and size of each active microfluidic flow control devices in lieu of using an entire sheet or layer of the elastomeric material.

[0076] In one embodiment, the disk-shaped microfluidic processing disk **4000** comprises a plurality of generally interchangeable and separable triangular or “pie-shaped” chemical analysis processing wedges **4002**. The wedges **4002** may be detachably interlocked together such as via suitable mechanical interlock features (e.g. snap-fit tabs/slots, etc.) and/or fasteners to collectively form the annular disk-shaped body of the processing disk **4000**. In other embodiments, the wedges **4002** may be permanently joined together such as via adhesives or ultrasonic welding as some examples.

[0077] Each processing wedge **4002** of microfluidic processing disk **4000** is a discrete microfluidic device which may be fluidly isolated from every other processing wedge in one embodiment within the confines of the processing disk structure (i.e. no cross flow through the disk between wedges). Beyond the microfluidic processing disk physical boundary, however, individual processing wedges may fluidly share common inlet manifolds connected to a source fluid flow (e.g. water, slurry, air, chemical, etc.) or outlet manifolds (e.g. waste/exhaust manifold) for convenience of construction and cost efficiency. Each processing wedge **4002** is a complete chemical processing device or train operable to process and analyze a soil sample initially provided in slurry form (from one of the mixing stations previously described herein) for a different analyte. Advantageously, this provides a plurality of chemical processing trains (i.e. wedges **4002**) capable of processing and analyzing soil samples simultaneously in parallel for different analytes (e.g. plant-available nutrients or other chemical constituents/properties) in conjunction with the centrifuge **3400**. This parallelization reduces the time required for completely processing and analyzing a soil sample for multiple analytes.

[0078] With additional reference to FIGS. 13-16, it bears noting that when a centrifuge **3400** is used for producing the

supernatant, microfluidic processing disk **4000** may be configured and operable to form a detachable fluid coupling to the centrifuge through intermediary fluid exchange dock **3430**. Fluid exchange dock **3430** is fluidly coupled and interposed between the microfluidic processing disk **4000** and centrifuge **3400**.

[0079] Centrifuge **3400** includes an aerodynamic cover assembly to streamline the tube hub **3500** assembly as it spins to reduce power input and noise due to aerodynamic losses since the tube hub which carries pivotable centrifuge tubes **3450** would act as an air impeller otherwise. The cover assembly comprises an upper cover **3520** and lower cover **3521** which are affixed to the hub such as via threaded fasteners in one embodiment or other mechanical fastening methods. The hub **3500** is thus sandwiched and compressed between the covers, as further shown in FIG. 66 which depicts the completed hub assembly.

[0080] With continuing reference to FIGS. 13-16, each of the upper and lower covers **3520**, **3521** may have a disk-shaped body including a central opening **3522** and plurality of rectangular tube openings **3523** formed completely through the cover between their top and bottom surfaces. Tube openings **3523** may be arranged in a circumferential pattern around central opening and are radially elongated as shown. The tube openings **3523** are arranged such that the mounted centrifuge tubes **3450** are exposed within the covers. Tube openings **3523** preferably have a radial length sized to allow the mounted centrifuge tube to fully swing outwards and upwards within the opening when rotated by the centrifuge **3400**. Centrifuge tubes **3452** are each pivotably mounted in tube openings **3523** and angularly movable between a vertical position shown in FIG. 15 when the centrifuge **3400** is stationary, and a horizontal position shown in FIG. 16 when the centrifuge (e.g., cover assembly) and hub **3500** is rotated at full speed by the rotary centrifuge drive mechanism. This ensures that the acceleration experienced by the sample due to gravity or rotational acceleration is always away from the tube ports. The tubes **3450** are preferably configured such that the top surface is substantially flush with the top surface **3524** of the upper cover **3520**, or preferably slightly raised and protruding above the top surface as seen in FIG. 15 to be engaged by the bottom surface **3432** of the fluid exchange dock **3430** to form a sealed connection between the flow ports **3451** of the tubes and flow passages **3434** of the dock **3500**. In the vertical position, the centrifuge tubes **3450** project downwards below the bottom surface **3525** of the lower cover **3521** such that a majority of the height of the tube extends beneath the bottom surface **3525**.

[0081] WO2020/012369 describes the centrifuge **3400** including the foregoing features and operation in greater detail.

[0082] Referring back now generally to FIGS. 5-16, each processing wedge **4002** of microfluidic processing disk **4000** may have a truncated wedge shape including a top major surface **4003**, an opposing bottom major surface **4004**, opposing arcuately curved inner and outer surfaces **4005**, **4006**, and a pair of converging radial side surfaces **4007**. Side surfaces **4007** each define radial reference lines **R1** which intersect at a geometric vertical centerline **C1** of the processing wedge **4002**. When the processing wedges **4002** are assembled together in microfluidic processing disk **4000**, they collectively define a circular central opening **4014** (for purposes similar to central opening **3435** of dock **3430**).

Processing wedge **4002** defines an outer peripheral portion or region **4008** defined as proximate to outer surface **4006**, and an inner hub portion or region **4009** defined as proximate to inner surface **4005**. Although the non-limiting illustrated embodiment includes eight analysis processing wedges **4002**, other embodiments may use more or less wedges.

[0083] A plurality of fluid exchange ports are formed in each processing wedge **4002**. The ports may include a plurality of outer ports **4010** arranged in an array in peripheral region **4008** of the processing wedge, and a plurality of inner ports **4011** arranged in an array in the inner hub region **4009**. In one embodiment, the outer ports **4010** may penetrate only the top major surface **4003** of the processing wedge **4002** and the inner ports **4010** may penetrate only the bottom major surface **4004**. In one non-limiting implementation, as an example, eight outer ports **4010** and three inner ports **4011** may be provided as illustrated. Other numbers of ports may be used in other embodiments and is not limiting of the invention. The inner ports **4011** correspond in number and arrangement to the clusters **3433** of flow passages **3434** in the fluid exchange dock **3430** (see, e.g. FIGS. 13-14), which in turn match the flow ports **3451** formed in the top surface of centrifuge tubes **3450** for exchanging fluids when the tube hub **3500** is in the upper docked position. Inner ports **4011** may be mutually configured with the top inlets to the flow passages **3434** in the fluid exchange dock **3430** to form a detachable leak-resistant sealed joint therebetween. For example, inner ports **4011** may thus be configured with the same type nozzles **3436** shown in FIG. 56 on the bottom of fluid exchange dock **3430** to form a detachable sealed therewith in a similar manner.

[0084] Outer ports **4010** are configured for fluid connection to external process tubing **3021** (see, e.g. FIG. 12). In one embodiment, outer ports **4010** may optionally include upwardly protruding tubing barbs **4013** to facilitate the coupling (see, e.g. FIG. 12). Alternatively, outer ports **4010** may instead include recessed nozzles **3436** configured similarly to the inner ports **4011** which can also facilitate fluid connection to process tubing **3021** without having a protruding tubing barb.

[0085] Referring to the microfluidic processing wedge **4002** flow diagrams of FIGS. 2-3, the inner and outer ports **4010**, **4011** are fluidly coupled together by a branched microchannel network **4015** of microchannels **4012** formed internally within the microfluidic processing disk **4000**. On the liquid side, the microchannel network forms flow paths between the inner and outer ports, and fluidly couples the flow control microfluidic devices together embedded in microfluidic processing disk **4000**. The flow network **4015** also includes air microchannels **4012** which forms air connections to the liquid microchannels and microfluidic flow control devices by the pneumatic system which may include sources of high pressure and low pressure air as shown. Pressurized air provided by air compressor **3030** (example shown in FIGS. 2-3) or another compressor/compressors which provide the motive force for flowing and processing the foregoing fluids through the microfluidic processing disk **4000** and analytical processing wedges **4002** in accordance with the flow diagrams and as described herein.

[0086] The microchannels **4012** (air and liquid) of each processing wedge **4002** are configured and patterned to form the functional layout and fluid connections represented in the flow diagrams of FIGS. 2 and 3 (recognizing that the

physical layout may differ to create the functional connections shown). The blocks on the left of this figure represent the outer ports **4010** and those on the right represent the inner ports **4011** of each processing wedge **4002**. It is well within the ambit of a microfluidic device manufacturer to create the depicted flow network (and flow control microfluidic devices shown) using computer-aided fabrication methods without undue further elaboration here. The microchannels **4012** may be formed in one or more of the layers of the microfluidic processing disk by any suitable process or combination of processes commonly used to construct microfluidic devices, such as for example without limitation micro-machining, laser milling, laser or chemical etching, lithography, hot embossing, injection molding, or other.

[0087] The microchannel network **4015** further includes a plurality of microfluidic valves, pumps, mixing chambers shown in FIGS. 2-3. In one embodiment, these microfluidic devices may be diaphragm operated and created using a flexible elastomeric flow control layer embedded within the microfluidic processing disk **4000** which is in communication with the microchannels and chambers created within the microfluidic processing disk **4000**, as described elsewhere herein. The microfluidic devices may further include pneumatically-actuated diaphragm micropumps including extractant pump **4020**, slurry pump **4021**, reagent pump **4022**, and transfer pump **4023**. The microchannels **4012** are opened/closed by a plurality of pneumatically-actuated diaphragm microvalves **4018** schematically represented by circles (solid circle=closed; open circle=open). Pneumatically-actuated micro-mixing chambers **4024** may optionally be provided as required for mixing soil sample slurry with extractant, and/or upstream of the flow analysis cell **4027** and flow cell window **4025** each integrated into the processing wedge **4002** to ensure complete mixing of the color changing reagent (also sometimes referred to as "indicator") and supernatant if required. In some embodiments, the micro-mixing chambers **4024** may be formed by two closely fluidly coupled cells connected via a narrow short microchannel which is well known construction in the microfluidic arts. The cells are alternately pressurized by air to cyclically transfer the liquid back and forth multiple times between the cells, thereby providing thorough mixing. They mixers may or may not be diaphragm operated. It will be appreciated that other types of microfluidic mixers, pumps, and valves however may be used and the invention is not limited to the disclosed non-limiting examples.

[0088] FIGS. 17-19 are exploded and side cross-sectional views respectively of an on-disk pneumatically-actuated diaphragm micropump **5760**, which may be used for the extractant pump **4020**, slurry pump **4021**, reagent pump **4022**, transfer pump **4023**, or other pumps that may be required. These pumps are incorporated into the microchannel network **4015** of each disk processing wedge **4002** and apply the motive force to the fluid to drive it through the microchannel network and various flow-related features of the disk. The micropumps and features shown are each integrally formed or molded within two adjacent layers of each wedge **4002** as unitary structural portions thereof.

[0089] Each micropump **5760** is a sandwiched structure including an upper layer **5761** of the microfluidic processing disk **4000** (e.g., processing wedge **4002**), adjacent lower layer **5762** of the disk, and a thin resiliently deformable diaphragm **5763** having an elastic memory and defining a top surface **5763-1** and opposing bottom surface **5763-2**. It

bears particular note that the upper and lower layers **5761**, **5762** are not necessary the uppermost (i.e. top) and lowermost (i.e. bottom) layers of the multi-layered microfluidic processing disk **4000**, but instead may be two adjacent intermediate layers therebetween. In one non-limiting embodiment, the upper and lower layers **5761**, **5762** are intermediate layers in a 5-layer processing disk **4000** recognizing that more or less layers may be used as needed to create the desired microfluidic devices and flow paths therein.

[0090] The diaphragm **5763** may be made of a suitable elastomeric material or polymer, such as silicone in some embodiments, and may have thicknesses less than 1 mm (0.04 inches). Diaphragm **5763** is resiliently movable between a normally flat standby condition when no pneumatic air pressure signal is applied and a deformed downwardly projecting convex actuated condition when air is applied to the top surface of the diaphragm. The diaphragm **5763** may be oval in one configuration; however, other shapes may be used.

[0091] The micropump **5760** further includes an upper pump chamber **5764** recessed into the bottom surface of the upper layer **5761** of microfluidic processing disk **4000**, and a concavely shaped lower pump chamber **5765** directly opposing and vertically aligned with the upper chamber formed in the lower layer **5762**. The upper chamber **5764** may have straight sidewall surfaces **5764-1** and a flat top surface **5764-2** in some embodiments. Lower chamber **5765** is recessed into the top surface of lower layer **5762** and may include including arcuately curved sidewall surfaces **5765-1** which extend perimetrically around the chamber to define the concavity. A flat bottom surface **5765-2** adjoins the sidewall surfaces around the perimeter of the lower chamber as shown. The curved sidewall surfaces ensure that the diaphragm **5763** does not tear or crack when actuated over multiple operating cycles. It bears noting that the lower chamber **5765** defines the volumetric pumping capacity of the micropump which is expelled with each actuation of the micropump.

[0092] The micropump **5760** further includes a pneumatic air pressure signal port **5768** formed in upper layer **5761** which in fluid communication with the upper chamber **5764**. Port **5768** is preferably centered in the top surface of the upper chamber **5764** and in fluid communication with a pneumatic or air microchannel network **4015-1** formed in the disk layer immediately above the upper layer **5761** and fluidly coupled to an air source such as those described herein. The lower layer **5762** includes a fluid inlet port **5766** for introducing fluid into the lower chamber **5765**, and a fluid outlet port **5767** for discharging fluid from the lower chamber caused by operation of the micropump **5760**. Each port **5766**, **5767** is thus in fluid communication with the lower chamber **5765**. The fluid inlet port **5766** preferably penetrates the lower chamber **5765** at an opposite end of the chamber than its outlet port **5767** at the other end. Each of the fluid inlet and outlet ports is in fluid communication with the fluid microchannel network **4015** formed in the disk layer immediately below the lower layer **5762**. In one embodiment, the upper and lower chambers **5761**, **5762** may be oval shaped; however, other shapes may be used.

[0093] Operation of micropump **5760** will be briefly described. Each micropump has an associated fluid inlet diaphragm microvalve **4018** and fluid outlet diaphragm microvalves **4018** fluidly coupled to the fluid inlet and outlet

ports **5766**, **5767** respectively which are necessary for operation of the micropump. The diaphragm valves have the same general construction and operation as the micropumps including a diaphragm, air pressure signal port, and fluid inlet and outlet ports. Operation of the valves between an open and closed position is performed in the same manner as described below for the micropumps which are thus analogous in structure and function to the valves. The valves however are generally smaller in size due to the multitude of valves arranged in the microfluidic processing disk **4000** to conserve space, and typically utilize circular diaphragms and upper and lower chambers in contrast to the elongated features of the micropumps intended to hold a predetermined volume of fluid necessary for the chemical processes and soil analysis.

[0094] FIG. **18** shows the pump in the initial flat unactuated or standby condition. Diaphragm **5763** is fully nested inside upper pump chamber **5764** and does not project downwards into lower pump chamber **5765**. The diaphragm is trapped in the upper chamber **5764** between the upper and lower disk layers **5761**, **5762**. No air is applied to the diaphragm at this stage. The fluid outlet diaphragm microvalve **4018** is first closed and the fluid inlet diaphragm valve is opened to fill the lower chamber **5765** beneath the diaphragm with the fluid to be pumped from the microchannel network **4015** (e.g. soil slurry, extractant, reagent, supernatant, or other fluid). The fluid inlet diaphragm microvalve **4018** is then closed and the fluid outlet diaphragm microvalve **4018** is opened.

[0095] To pump the fluid volume contained in the lower pump chamber **5765**, air is supplied to the top of the diaphragm **5763** via the air pressure signal port **5768** from the air source which is controlled by an air valve. The air pressure drives the diaphragm downward, which deforms and generally conforms to the shape of the lower chamber **5765**, thereby expelling the fluid through the fluid outlet port **5767** and its associated outlet microvalve **4018**. The diaphragm **5763** is now in the deformed convex actuated condition shown in FIG. **19**. After pumping is completed, the air pressure is relieved from the air pressure signal port **5768** and the diaphragm **5763** returns to its original undeformed flat standby condition ready for the next pumping cycle.

[0096] In testing, it was discovered that if smooth surfaces are provided within the lower pump chamber **5765**, the flexible diaphragm **5763** tends on occasion to get sucked into the fluid outlet port **5767** for either the pneumatic signal or fluid liquid-side communication prematurely. This unfortunately blocks fluid flow and pumping before the diaphragm is fully displaced/deformed and prevents the liquid volume in the lower chamber from being fully expelled. This causes inconsistency in the volume of fluid pumped per actuation, which can adversely affect proper slurry processing and analysis since the volumetric capacity for each pump chamber is carefully predetermined and exacting to ensure the proper ratio of chemicals (e.g. reagent, extractant, etc.) are mixed with the slurry.

[0097] To combat the foregoing diaphragm and pumping problems, the concave lower pump chamber **5765** preferably is provided with a plurality of "anti-stall" grooves **5769** which act to keep the flexible diaphragm **5763** from getting sucked into the fluid outlet port **5767** and blocking flow. This also prevents the diaphragm from attaching via formation of suction to but not fully releasing from the generally flat bottom surface **5765-2** of the lower pump chamber. The

anti-stall grooves **5769** are therefore configured to prevent adherence of the diaphragm **5763** to the lower pump chamber **5765**, thereby advantageously allowing the diaphragm **5763** to fully and reliably displace substantially the entire volumetric fluid contents of the lower chamber with each pumping cycle, thereby ensuring accuracy of the amount of fluid dispensed and ultimate soil slurry analysis. The recessed anti-stall grooves **5769** are cut or otherwise formed into preferably all surfaces within the lower chamber **5765** (e.g. sidewall surfaces **5765-1** and flat bottom surface **5765-2**), as shown in FIG. 17. In one embodiment, the grooves **5769** may be arranged in a two-directional perpendicularly intersecting grid array of grooves as shown forming a somewhat checkerboard pattern. In other embodiments, the grooves may be unidirectional and formed by a plurality of non-intersecting and spaced apart parallel grooves arranged either along the major axis or minor axis of the lower chamber **5765**, or diagonally to the axes. In some embodiments, the upper pump chamber **5764** formed in the upper disk layer **5761** may include anti-stall grooves similar to or different in configuration than the grooves in the lower chamber **5765**. Any suitable pattern and number of grooves may be provided.

[0098] The microchannel network **4015** may further include a plurality of microreservoirs of predetermined volume for holding and staging the extractant, reagent, slurry, etc. for processing. In one embodiment, this may include an extractant microreservoir **4030**, soil slurry microreservoir **4031**, reagent microreservoir **4032**, and supernatant microreservoir **4033**. The microreservoirs **4030-4033** may be formed by a series of closely spaced, undulating loops of microchannels as shown. Sample non-limiting volumetric capacities of each microreservoir are shown in FIG. 2. Other volumetric capacities, however, may of course be used.

[0099] The process described below and in the flow diagrams of FIGS. 2-3 may be automatically controlled and executed by the system programmable controller, such as for example processing system controller **2820** shown in FIG. 4 (see, e.g. system interface block **2803**). The controller is operably coupled to the low and high pressure air supply, such as provided by air compressor **3030**. The low pressure air may be created in any suitable known manner such as by employing a pressure reducing valve station taking suction from the air tank **3031** associated with compressor **3030**, which may contain high pressure air produced by the compressor. All air supply related components (compressor, tank(s), and valves) may therefore be controlled by the system programmable controller (e.g. processing system **2820**). Other sources of low and high pressure air for pneumatically controlling operation of the microfluidic processing disk **4000** such as separate compressors may of course be used. The controller **2820** via pneumatic operation of the microvalves **4018** shown in FIGS. 2-3 may further control operation of the various fluid inlets **4010-2** to **4010-6** of the processing wedge **4002**, exhaust/waste outlets **4010-7** to **4010-8**, and fluid interface with either the centrifuge **3400** or microporous filter **5757** each previously described herein.

[0100] To reiterate, as previously noted, the blocks on the left of the flow diagrams represent the outer ports **4010** of the respective processing wedge **4002** and blocks on the right represent the inner ports of the wedge. In one implementation, the outer ports **4010** may include a high pressure air inlet **4010-1**, low pressure air inlet **4010-2** also config-

ured to operate as an air vent when required, extractant inlet **4010-3**, cleaning solution **4010-4**, slurry sample inlet **4010-5**, reagent (indicator) inlet **4010-6**, low pressure exhaust outlet **4010-7**, and high pressure exhaust outlet **4010-8**. The cleaning solution provided to inlet **4010-4** may be any suitable solution including deionized water or other. The inner ports **4011** may include a slurry sample outlet **4011-1** from processing wedge **4002** to centrifuge **3400** (i.e. centrifuge tube **3450**), supernatant inlet **4011-2** from centrifuge **3400**, and centrifuge waste inlet **4011-3** from the centrifuge. Other types and numbers of outer and inner ports **4010**, **4011** may of course be provided.

[0101] FIGS. 2-3 shows the provision of the microfluidic processing disk **4000** and one of the processing wedges **4002** with integrated microchannel network **4015** at the start and readied for processing and chemically analyzing an agricultural sample such as without limitation a soil sample in slurry form. An example of processing and analyzing the slurry for analytes of interest is previous described herein and in commonly-owned WO2020/012369.

[0102] In one non-limiting embodiment, the slurry sample and extractant measurement loops (reservoirs) are pumped together into an optional first micro-mixing chamber **4024** where they are mixed. In some situations, adequate mixing of the sample and extractant may be achieved within the microchannels **4012** to obviate the need to a separate micro-mixing chamber (hence designation of the same with a “?” in the figure). Diaphragm-operated micropumps **4020**, **4021** are pressurized with low pressure air as shown to achieve pumping of the fluids. Complete mixing of the slurry sample and extractant is then performed. Next, the extractant/sample mixture is pumped from first micro-mixing chamber **4024** to the centrifuge **3400** for processing. The supernatant and reagent are staged and pumped into their respective measurement loops (i.e. microreservoirs **4033** and **4032** at a precise predetermined ratio of supernatant to reagent. Some supernatant and reagent are very briefly dumped to waste via the flow path to lower pressure exhaust outlet **4010-7** to ensure these microreservoirs are completely filled. The supernatant and reagent are then pumped to a second micro-mixing chamber **4024**. Note that the microchannel flow path comprising the micro-mixing chamber **4024**, de-bubbler **4026**, and flow cell window **4025** are active and fluidly connected to low pressure exhaust outlet **4010-7**. Complete mixing of the supernatant and reagent is performed in the second micro-mixing chamber **4024**, thereby causing a color change in the solution for detection by the absorbance analysis flow cell **4027** via downstream flow cell window **4025**. The supernatant and reagent mixture incorporating the analyte therein is then pumped through the de-bubbler **4026** in the de-bubbling station which removes any residual air bubbles entrained in the mixture. Bubbles in the liquid stream may cause volume anomalies in the downstream flow analysis cell **427** and adversely affect analytical accuracy. De-bubblers are well known devices in the art without further undue elaboration.

[0103] The supernatant/reagent mixture incorporating the analyte is then pumped into flow cell window **4025** of absorbance flow analysis cell **4027** for colorimetric measurement by the absorbance flow analysis cell **4027**. The present flow analysis cell **4027** may be formed integrally with and incorporated directly into a portion of processing wedge **4002**. FIGS. 20 and 21 schematically depict the portion of wedge **4002** containing absorbance flow analysis

cell **4027** and flow cells window **4025** formed within the bonded layer structure of the processing wedge. In the exemplary non-limiting construction shown, the layers comprises three hard plastic layers **4000-1** (e.g. PC, etc.) forming a top layer, bottom layer, and intermediate patterned with the foregoing microchannels and other fluid control devices such as the micropumps, microvalves, and micro-mixing chambers. The thin flexible elastomeric layer **4000-2** (e.g. silicon, etc.) is formed immediately on top of the intermediate hard layer **4000-1** for functioning as a diaphragm of the fluid control devices. In one embodiment, flow analysis window **4025** may be a laterally widened diamond-shaped chamber (see, e.g. FIG. 21). An LED emitting diode assembly **4040** and LED receiving diode assembly **4041** are mounted above and below the flow analysis window **4025** respectively. Diode assemblies **4040**, **4041** are attached to the outmost top and bottom surfaces of processing wedge **4002** above and below window **4025** as shown, but fluidly isolated from the window and liquid flow stream in the processing wedge **4002**. Layer **4000-2** may have a cutout formed directly above flow analysis window **4025** corresponding in size and shape to the emitting diode assembly **4040** to avoid possible reflective/refractive interference with the emitted analysis light beam.

[0104] In operation, the liquid reagent and supernatant mixture flows through flow analysis window **4025** (see, e.g. solid liquid flow arrows). As the flow passes through the window **4025**, the emitting diode assembly **4040** transmits and shines light through the window and liquid therein to the receiving diode assembly **4041** for colorimetric measurement in a known manner. The measurement of the analyte in the sample mixture liquid stream is transmitted to the system programmable controller for analysis and quantification. During the analysis, it bears noting that the sample mixture flows continuously through the flow cell window **4025** to the low pressure exhaust outlet **41010-7** where it is then dumped to waste.

[0105] It bears noting that the micro-mixing chambers **4024** described above may be omitted in some instances if complete mixing can be achieved within the microchannels themselves. The micro-mixing chambers **4024** are therefore optional for use when required.

[0106] After the soil sample has been fully processed in the above manner, the system programmable controller **2820** is configured to initiate a cleaning cycle to prepare the microfluidic processing disk **4000** for processing a new soil sample. Cleaning solution and low pressure air are each selectively and alternately pumped into and through the emboldened active sample loop microchannels **4012** and through the centrifuge **3400** to the high pressure exhaust outlet **4010-8** as shown (FIG. 2). This clears residual soil slurry and chemicals from these components and microchannels. After several cycles of alternating cleaning solution and purge air is processed through the microchannels and centrifuge, at this point, the sample loop and flow paths upstream of the of the sample loop microchannels has only air in it. There is a column containing a mixture of air and cleaning solution remaining in the emboldened section of flow path. The microvalves **4018** open to allow high pressure air from high pressure air inlet **4010-1** to force the air/cleaning solution mixture column through the centrifuge **3400**. The high pressure air then purges the centrifuge and flows to the high pressure exhaust outlet **4010-8**, which completes the cleaning cycle.

[0107] The same microfluidic process described above with respect to FIG. 2 for processing wedge **4002** is generally applicable to a processing wedge which operably and fluidly interfaces with ultrafine microporous filter **5757** shown in FIG. 3 in line of a centrifuge for producing the supernatant for chemical analysis.

[0108] As already noted herein, the agricultural sampling system, sub-systems, and related processes/methods disclosed herein may be used for processing and testing soil, vegetation/plants, manure, feed, milk, or other agricultural materials for related parameters of interest. Particularly, embodiments of the chemical analysis portion of the system (chemical analysis sub-system **3003**) disclosed herein can be used to test for multitude of chemical-related parameters and analytes (e.g. nutrients/chemicals of interest) in other areas beyond soil and plant/vegetation sampling. Some non-limiting examples (including soil and plants) are as follows.

[0109] Soil Analysis: Nitrate, Nitrite, Total Nitrogen, Ammonium, Phosphate, Orthophosphate, Polyphosphate, Total Phosphate, Potassium, Magnesium, Calcium, Sodium, Cation Exchange Capacity, pH, Percent Base Saturation of Cations, Sulfur, Zinc, Manganese, Iron, Copper, Boron, Soluble Salts, Organic Matter, Excess Lime, Active Carbon, Aluminum, Amino Sugar Nitrate, Ammoniacal Nitrogen, Chloride, C:N Ratio, Electrical Conductivity, Molybdenum, Texture (Sand, Silt, Clay), Cyst nematode egg counts, Mineralizable Nitrogen, and Soil pore space.

[0110] Plants/Vegetation: Nitrogen, Nitrate, Phosphorus, Potassium, Magnesium, Calcium, Sodium, Percent Base Saturation of Cations, Sulfur, Zinc, Manganese, Iron, Copper, Boron, Ammoniacal Nitrogen, Carbon, Chloride, Cobalt, Molybdenum, Selenium, Total Nitrogen, and Live plant parasitic nematode.

[0111] Manure: Moisture/Total Solids, Total Nitrogen, Organic Nitrogen, Phosphate, Potash, Sulfur, Calcium, Magnesium, Sodium, Iron, Manganese, Copper, Zinc, pH, Total Carbon, Soluble Salts, C/N Ratio, Ammoniacal Nitrogen, Nitrate Nitrogen, Chloride, Organic Matter, Ash, Conductance, Kjeldahl Nitrogen, E.coli, Fecal Coliform, Salmonella, Total Kjeldahl Nitrogen, Total Phosphate, Potash, Nitrate Nitrogen, Water Soluble Nitrogen, Water Insoluble Nitrogen, Ammoniacal Nitrogen, Humic Acid, pH, Total Organic Carbon, Bulk Density (packed), Moisture, Sulfur, Calcium, Boron, Cobalt, Copper, Iron, Manganese, Arsenic, Chloride, Lead, Selenium, Cadmium, Chromium, Mercury, Nickel, Sodium, Molybdenum, and Zinc

[0112] Feeds: Alanine, Histidine, Proline, Arginine, Isoleucine, Serine, Aspartic Acid, Leucine, Threonine, Cystine, Lysine, Tryptophan, Glutamic Acid, Methionine, Tyrosine, Glycine, Phenylalanine, Valine (Requires Crude Protein), Arsenic, Lead, Cadmium, Antimony, Mercury

[0113] Vitamin E (beta-tocopherol), Vitamin E (alpha-tocopherol), Vitamin E (delta-tocopherol), Vitamin E (gamma-tocopherol), Vitamin E (total), Moisture, Crude Protein, Calcium, Phosphorus, ADF, Ash, TDN, Energy (Digestible and Metabolizable), Net Energy (Gain, Lactation, Maintenance), Sulfur, Calcium, Magnesium, Sodium, Manganese, Zinc, Potassium, Phosphorus, Iron, Copper (not applicable to premixes), Saturated Fat, Monounsaturated Fat, Omega 3 Fatty Acids, Polyunsaturated Fat, Trans Fatty Acid, Omega 6 Fatty Acids (Requires Crude or Acid Fat), Glucose, Fructose, Sucrose, Maltose, Lactose, Aflatoxin (B1, B2, G1, G2), DON, Fumonisin, Ochratoxin, T2-Toxin, Zearalenone, Vitamin B2, B3, B5, B6, B7, B9, and B12,

Calories, Chloride, Crude fiber, Lignin, Neutral Detergent Fiber, Non Protein Nitrogen, Selenium U.S. Patent, Total Iodine, Total Starch, Vitamin A, Vitamin D3, and Free Fatty Acids.

[0114] Forages: Moisture, Crude Protein, Acid Detergent Fiber ADF, NDF, TDN, Net Energy (Gain, Lactation, Maintenance), Relative Feed Value, Nitrate, Sulfur, Copper, Sodium, Magnesium, Potassium, Zinc, Iron, Calcium, Manganese, Sodium, Phosphorus, Chloride, Fiber, Lignin, Molybdenum, Prussic Acid, and Selenium USP.

[0115] Milk: Butterfat, True Protein, Somatic Cell Count, Lactose, Other Solids, Total Solids, Added Water, Milk Urea Nitrogen, Acidity, pH, Antibiotic tests, and Micro-organisms.

Control System

[0116] FIG. 4 is a schematic system diagram showing the control or processing system **2800** including programmable processor-based central processing unit (CPU) or system controller **2820** as referenced to herein. System controller **2820** may include one or more processors, non-transitory tangible computer readable medium, programmable input/output peripherals, and all other necessary electronic appurtenances normally associated with a fully functional processor-based controller. Control system **2800**, including controller **2820**, is operably and communicably linked to the different soil sample processing and analysis systems and devices described elsewhere herein via suitable communication links to control operation of those systems and device in a fully integrated and sequenced manner.

[0117] Referring to FIG. 4, the control system **2800** including programmable controller **2820** may be mounted on a stationary support in any location or conversely on a translatable self-propelled or pulled machine (e.g., vehicle, tractor, combine harvester, etc.) which may include an agricultural implement (e.g., planter, cultivator, plough, sprayer, spreader, irrigation implement, etc.) in accordance with one embodiment. In one example, the machine performs operations of a tractor or vehicle that is coupled to an implement for agricultural operations. In other embodiments, the controller may be part of a stationary station or facility.

[0118] Control system **2800**, whether onboard or off-board a translatable machine, generally includes the controller **2820**, non-transitory tangible computer or machine accessible and readable medium such as memory **2805**, and a network interface **2815**. Computer or machine accessible and readable medium may include any suitable volatile memory and non-volatile memory or devices operably and communicably coupled to the processor(s). Any suitable combination and types of volatile or non-volatile memory may be used including as examples, without limitation, random access memory (RAM) and various types thereof, read-only memory (ROM) and various types thereof, hard disks, solid-state drives, flash memory, or other memory and devices which may be written to and/or read by the processor operably connected to the medium. Both the volatile memory and the non-volatile memory may be used for storing the program instructions or software. In one embodiment, the computer or machine accessible and readable non-transitory medium (e.g., memory **2805**) contains executable computer program instructions which when executed by the system controller **2820** cause the system to perform operations or methods of the present disclosure

including measuring properties and testing of soil and vegetative samples. While the machine accessible and readable non-transitory medium (e.g., memory **2805**) is shown in an exemplary embodiment to be a single medium, the term should be taken to include a single medium or multiple media (e.g., a centralized or distributed database, and/or associated caches and servers) that store the one or more sets of control logic or instructions. The term “machine accessible and readable non-transitory medium” shall also be taken to include any medium that is capable of storing, encoding or carrying a set of instructions for execution by the machine and that cause the machine to perform any one or more of the methodologies of the present disclosure. The term “machine accessible and readable non-transitory medium” shall accordingly also be taken to include, but not be limited to, solid-state memories, optical and magnetic media, and carrier wave signals.

[0119] Network interface **2815** communicates with the agricultural (e.g. soil or other) sample processing and analysis systems (and their associated devices) described elsewhere (collectively designated **2803** in FIG. 4), and other systems or devices which may include without limitation implement **2840** having its own controllers and devices.

[0120] The programmable controller **2820** may include one or more microprocessors, processors, a system on a chip (integrated circuit), one or more microcontrollers, or combinations thereof. The processing system includes processing logic **2826** for executing software instructions of one or more programs and a communication module or unit **2828** (e.g., transmitter, transceiver) for transmitting and receiving communications from network interface **2815** and/or agricultural sample processing and analysis system **2803** which includes sample preparation sub-system **3002** and the components described herein further including the closed slurry recirculation flow loop **8002** components. The communication unit **2828** may be integrated with the control system **2800** (e.g. controller **2820**) or separate from the programmable processing system.

[0121] Programmable processing logic **2826** of the control system **2800** which directs the operation of system controller **2820** including one or more processors may process the communications received from the communication unit **2828** or network interface **2815** including agricultural data (e.g., test data, testing results, GPS data, liquid application data, flow rates, etc.), and soil sample processing and analysis systems **2803** generated data. The memory **2805** of control system **2800** is configured for preprogrammed variable or setpoint/baseline values, storing collected data, and computer instructions or programs for execution (e.g. software **2806**) used to control operation of the controller **2820**. The memory **2805** can store, for example, software components such as testing software for analysis of soil and vegetation samples for performing operations of the present disclosure, or any other software application or module, images **2808** (e.g., captured images of crops), alerts, maps, etc. The system **2800** can also include an audio input/output subsystem (not shown) which may include a microphone and a speaker for, for example, receiving and sending voice commands or for user authentication or authorization (e.g., biometrics).

[0122] The system controller **2820** communicates bi-directionally with memory **2805** via communication link **2830**, network interface **2815** via communication link **2832**, display device **2830** and optionally a second display device

2825 via communication links **2834**, **2835**, and I/O ports **2829** via communication links **2836**. System controller **2820** may further communicate with the soil sample processing and analysis systems **2803** via wired/wireless communication links **5752** either via the network interface **2815** and/or directly as shown.

[0123] Display devices **2825** and **2830** can provide visual user interfaces for a user or operator. The display devices may include display controllers. In one embodiment, the display device **2825** is a portable tablet device or computing device with a touchscreen that displays data (e.g., test results of soil, test results of vegetation, liquid application data, captured images, localized view map layer, high definition field maps of as-applied liquid application data, as-planted or as-harvested data or other agricultural variables or parameters, yield maps, alerts, etc.) and data generated by an agricultural data analysis software application and receives input from the user or operator for an exploded view of a region of a field, monitoring and controlling field operations. The operations may include configuration of the machine or implement, reporting of data, control of the machine or implement including sensors and controllers, and storage of the data generated. The display device **2830** may be a display (e.g., display provided by an original equipment manufacturer (OEM)) that displays images and data for a localized view map layer, as-applied liquid application data, as-planted or as-harvested data, yield data, controlling a machine (e.g., planter, tractor, combine, sprayer, etc.), steering the machine, and monitoring the machine or an implement (e.g., planter, combine, sprayer, etc.) that is connected to the machine with sensors and controllers located on the machine or implement.

Microfluidic System Modifications

[0124] The sections which follow describe various aspects to the foregoing agricultural sample analysis systems and associated devices previously described herein which process and analyze/measure the prepared agricultural sample slurry for analytes of interest (e.g. soil nutrients such as nitrogen, phosphorous, potassium, etc., vegetation, manure, etc.). Specifically, the modifications relate to microfluidic devices and manifolds previously described herein which comprises part of chemical analysis sub-system **3003** of agricultural sampling system **3000** shown in FIG. 1. These microfluidic devices include microfluidic processing disk **4000** and individual analysis processing devices such as processing wedges **4002**, and alternative embodiments thereof described below.

[0125] FIGS. 22-41 show an alternative embodiment of a microfluidic manifold comprising a polygonal shaped microfluidic manifold slurry processing substrate **5000** for processing agricultural slurry sample. Processing substrate is constructed of a plurality of layers **5001** which may be permanently bonded together via adhesives, thermal/heat bonding, or other fabrication techniques previously described herein. Processing substrate **5000** may generally include the same internal pneumatically-actuated microfluidic devices as previously described herein for processing wedges **4002** such as diaphragm-operated micropumps **5015** including extractant pump **4020**, slurry pump **4021**, reagent pump **4022**, and transfer pump **4023**, microvalves **4018**, optional micro-mixing chambers **4024**, flow analysis cell **4027**, and branched microchannels **4012** fluidly coupling the devices together. The arrangement/layout and fluidic pas-

sageways however may be different due to the block-shaped form factor of support structure **5000** in lieu of triangular wedge shape.

[0126] It bears noting that the chemical analysis sub-system **3003** is comprised of a plurality of processing substrates **5000** operable in parallel to process and analyze slurry samples simultaneously. In contrast to annular and circular microfluidic processing disk **4000** adapted for use with a centrifuge **3000**, the block shape form substrate is amenable for use in any type and configuration of housing in a space efficient manner which may be provided when ultrafine microporous filter **5757** is used to produce the supernatant.

[0127] Manifold processing substrate **5000** may have a rectangular cuboid configuration in one as shown; however, other polygonal shapes may be used. Substrate **5000** is intended to be used in an upright vertical orientation as shown in FIGS. 22-23 in one implementation for reasons further described herein with respect to slurry flow paths. Other orientations are possible however for operation of the microfluidic manifold processing substrate **5000**. Similar to processing wedges **4002** previously described herein, each processing substrate is a standalone slurry analysis device or train which is fluidly isolated from every other processing substrate. This allows each substrate **5000** to process the slurry simultaneously in parallel for different analytes which advantageously decreases processing time and completion of chemical analyses.

[0128] A non-limiting example of a five-layer construction of microfluidic manifold processing substrate **5000** is shown recognizing that more or less layers may be provided in other embodiments as needed depending on the type of agricultural slurry processing intended to be performed. In order from the planar outer first major surface or side **5022** to opposite planar outer second major surface or side **5023**, the adjacent layers of the packaged processing substrate **5000** include first outer layer **5002**, liquid layer **5003** thereon, air layer **5004** thereon, fluid distribution layer **5005** (e.g., air and liquid—extractant, supernatant, slurry, etc.) thereon, and second outer layer **5006** thereon. Outer layer **5002** defines first major side **5022** while opposite outer layer **5006** defines second major side **5023**. The remaining layers are inner layers. The substrate further includes top side **5020**, opposite bottom side **5021**, and pair of opposed lateral sides **5024**. Major surfaces or sides **5022**, **5023** have a greater surface area than other sides of substrate **5000**.

[0129] Outer layer **5006** includes a plurality of quick-connect liquid fittings **5011** and quick connect air valves **5010**. Liquid fittings **5011** are configured for detachable connection to liquid tubing from various liquid sources used in microfluidic manifold substrate **5000** (e.g., extractant, cleaning/flushing water, calibration standard liquid, etc.). Air valves **5010** are configured for detachable connection to air tubing for applying pneumatic pressure signals or vacuum signals to the microfluidic devices embedded in substrate **5000**.

[0130] Fluid distribution layer **5005** is adjacent outer layer **5006** and includes a plurality of both fluidly separate and/or interconnected microchannels **4012** for transferring the air and liquids from their applicable sources via fittings **5010**, **5011** to the and in turn the microfluidic devices (e.g., microvalves **4018** and micropumps **5015** in microfluidic manifold substrate **5000** seen in the flow diagram of FIGS. 2-3). Each micropump **5015** and microvalve **4018** comprises

an individual thin and resiliently deformable elastomeric diaphragm **5763** having an elastic memory. The diaphragms are sandwiched and trapped between liquid layer **5003** and air layer **5004** when the processing substrate **5000** is fully assembled compressing the plural layers together.

[0131] The underside of liquid layer **5003** comprises a plurality of microchannels **4012** which fluidly couple the microvalves **4018** and micropumps **5015** together. FIGS. **30-31** show the fluid interconnections between these microfluidic devices/components formed by the microchannels **4012**. In one non-limiting embodiment, the devices and microchannels of the analysis processing substrates **5000** may be configured for mixing extractant with slurry to extract the analyte of interest for further chemical analysis processing to quantify the concentration of analyte (e.g., soil nutrients or other) and chemical properties such as pH and/or Bph. Substrate **5000** may have other used in the processing and analysis of the agricultural slurry. One non-limiting preferred vertical orientation of processing substrate **5000** for operation is shown in FIGS. **30-31**.

[0132] The micropumps **5015** may include a group of three fluidly interconnected micropumps including first micropump **5015a**, second micropump **5015b**, and third micropump **5015c** in one embodiment as shown. Micropumps **5015** are generally similar in construction and operation to pneumatically actuated micropumps **5760** previously described herein. Each micropump **5015** is a sandwiched structure including an air layer **5004**, adjacent liquid layer **5003**, and thin resiliently deformable diaphragm **5763** having an elastic memory and defining opposing top and bottom surfaces **5763a**, **5763b**.

[0133] In contrast to micropumps **5760**, the present micropumps **5015** (which collectively refers to each of micropumps **5015a-5015c**) includes a pump chamber **5037** collectively formed in one embodiment by concavely shaped air-side recess **5030** formed in the portion the air layer **5004** facing diaphragm **5763**, and a liquid-side recess **5031** formed in liquid layer **5003**. This arrangement is opposite to micropumps **5760**. The inventors have discovered that with respect to the prior micropump **5760** design, there is a certain amount of air pressure required to deform the flat diaphragm into the concavity, followed by an additional amount of air pressure required to seal the diaphragm enough to prevent fluid flow through a valve when in the closed position. By inverting the design, the pressure advantageously required to effectively seal the valves when closed is less as the diaphragm is not forced to deform and stretch into the concavity prior to sealing. Recess **5031** is arranged directly opposing and vertically aligned with the air-side recess **5030**. Liquid-side recess **5031** may have a circumferentially-extending peripheral sidewall **5032** extending perimetrically around the chamber and a flat base wall defining a flat top surface **5033** on which the diaphragm becomes engaged and seated between the pumping stroke. A flat-to-flat interface is formed between diaphragm **5763** and top surface **5033** during the pumping stroke. Air-side recess **5030** may include domed arcuately curved walls **5034** extending from side to side and circumferentially around the chamber to define the concavity. The curved sidewall surfaces ensure that the diaphragm **5763** does not tear or crack when actuated over multiple operating cycles. It bears noting that the air-side recess **5030** defines the volumetric pumping capacity of the micropump which is expelled with each diaphragm actuation of the micropump.

[0134] Air layer **5004** includes pneumatic air pressure signal port **5768** which in fluid communication with the air-side recess **5030** for pressuring the chamber and actuating the micropump **5015** during the slurry intake and discharge pumping stroke. Liquid layer **5003** includes fluid inlet port **5766** and fluid outlet port **5767** which are in fluid communication with the liquid-side recess **5031** for introducing and discharging fluids such as the slurry, extractant, flushing water, calibration standard liquids, etc. Similarly to micropump **5760** previously described herein, the inlet and outlet ports are formed at diametrically opposite ends of the recess **5031**.

[0135] In the present embodiment, micropumps **5015** also include anti-stall grooves **5769** recessed into the wall surfaces of both the air-side and liquid-side recesses **5030** and **5031**. The grooves in the air-side recess may be shallower in depth than those in the liquid-side recess. As previously noted herein, the anti-stall grooves **5769** are configured to prevent adherence of the diaphragm **5763** to the pump chambers **5030**, **5031** during operation of the pump. This advantageously allows the diaphragm **5763** to fully and reliably displace substantially the entire volumetric fluid contents of the liquid-side chamber with each pumping cycle, thereby ensuring accuracy of the amount of fluid dispensed and ultimate soil slurry analysis. The anti-stall grooves **5769** may be generally patterned in a two-directional perpendicularly intersecting rectilinear grid array of grooves as shown as previously described herein in relation to micropump **5760**.

[0136] In one embodiment, it bears noting that the anti-stall grooves **5769** intersect both the perimeter flow groove **5040** and fluid inlet and outlet ports **5766**, **5767** to better flush the slurry through and out of the micropumps **5015** during the discharge pumping stroke to prevent particulate retention and accumulations in the liquid-side recess **5031**.

[0137] Structurally, the present micropumps **5015** may further include two additional features including a perimeter flow groove **5040** and wide-base diaphragm seal ring **5041** (see, e.g. FIGS. **31-36**). The perimeter flow groove facilitates handling slurries such as soil slurries which contain a heavy particulate or solids content within the microfluidic manifold formed by processing substrates **5000**. During testing, the inventors observed that the area of lowest diaphragm physical displacement/movement and tight clearance around the perimeter of the diaphragms can be a place for the slurry particulates to become lodged. (e.g., sand-like particles). This may adversely affect proper operation and full volumetric pumping capacity with each pumping stroke. Adding the perimeter flow groove on the liquid side of each pump creates a large open cross sectional area which promotes flow in these otherwise tight areas. This advantageously continually flushes out the peripheral portions of the liquid-side recess **5031** with each pump stroke to prevent particulate and sediment buildup around the perimeter of the chamber.

[0138] Perimeter flow groove **5040** may be a continuous open structure which extends around the entire circumference and perimeter of the liquid-side recess **5031**. In one embodiment, the flow groove may have a rectilinear (rectangular or square) transverse cross-sectional shape as best shown in FIG. **36**. Flow groove **5040** may be inset from and inwardly spaced apart from peripheral sidewall **5032** and recessed into the flat top surface **5033** of the recess **5031**.

[0139] The wide-base diaphragm seal ring **5041** functions to prevent the elastomeric pump diaphragm **5763** from plugging the perimeter flow groove **5040** when the diaphragm is sandwiched between the pumping and air layers **5003**, **5004** as the layers of the analysis processing substrate **5000** are compressed and sealed together during assembly. The inventors discovered that once the flow groove was implemented, a problem was encountered with the peripheral portion of the diaphragm **5763** creeping down into the flow groove **5040** due to deformation of the diaphragm during the high temperatures encountered in the manifold fabrication process necessary to bond the layers of processing substrate together. This caused the diaphragm to “cave” into the perimeter flow groove **5040** which results in two problems: (1) obstruction of the perimeter flow grooves, allowing particulate to become trapped at the perimeter of the micropump; and (2) inadequate retention of the diaphragm in its peripheral sealing pocket **5035** formed adjacent side-wall **5032** and recessed into liquid layer **5003** in the liquid-side recess **5031**. This latter condition can lead to the diaphragm pulling out of its sealing pocket thereby adversely creating a leak path between the pneumatic and liquid sides of the micropump **5015**. The manifold processing substrate **5000** is no longer functional at this point due to the leakage path.

[0140] To overcome the foregoing problems, perimetrical extending seal ring **5041** is provided which prevents ingress of the peripheral region of the diaphragm **5763** into the perimeter flow groove **5040** to keep it clear for flushing slurry sediment/particulate during pumping (see e.g. FIG. 36). In one preferred but non-limiting embodiment, seal ring **5041** is disposed outboard of and immediately adjacent to the flow groove **5040**. Seal ring is a raised protrusion which projects upwards from top surface **5033** of the liquid-side recess **5031** to support the diaphragm at the location adjacent to the groove **5040**, thereby preventing creep into the groove.

[0141] In one embodiment, seal ring **5041** preferably has a wider base **5041b** than a terminal top end portion **5041c** terminated and formed by one or more obliquely angled surfaces **5041a**. The inventors discovered that the narrower top end portion helps penetrate the diaphragm **5763** to a greater degree than a broad top end. This readily pinches and deforms the diaphragm in the area adjacent to flow groove **5040** which precludes creep and incursion into the groove when liquid layer **5003** is compressed against and thermally bonded to opposing air layer **5004**. In addition, the seal ring **5041** ensures proper positioning of the peripheral portions or regions of the diaphragm into its circumferentially extending retention pocket **5035**. FIGS. 35A-B and 36 shows an embodiment of seal ring **5041** have a single angled surface **5041a** forming a partial trapezoidal shaped ring in cross-section. In other embodiments, opposing angled surfaces **5041a** may be provided forming a full trapezoidal shaped ring in cross-section (see, e.g. FIG. 35C). The angles surface (s) **5041a** create a cross-sectional profile of the seal ring **5041** which is narrower at top than at the base.

[0142] FIGS. 39 and 40 shows one of micropumps **5015** in operation for pumping a liquid such as agricultural slurry or other process liquid through the microfluidic manifold processing substrate **5000**. The method of operation will be briefly described. FIG. 39 shows micropump **5015** during the fill or intake stroke. A vacuum is applied to pump chamber **5037** via pneumatic air pressure signal port **5768**.

Diaphragm **5763** is displaced and drawn upwards into air-side recess **5030** of the micropump, which in turn draws a liquid (e.g., extractant, cleaning/flushing water, calibration standard liquid, etc.) into and fills pump chamber **5037** from fluid inlet port **5766**. The inlet port is fluidly coupled to a suction/intake side microchannel **4012** in the substrate. Diaphragm **5763** deforms into an arcuately curved profile and engages dome-shaped wall **5034** in the air-side recess and anti-stall grooves **5769** therein.

[0143] FIG. 40 shows micropump **5015** during the subsequent discharge or pumping stroke. Pneumatic (air) pressure is applied to pump chamber **5037** through the pneumatic air pressure signal port **5768**. This flattens and forces diaphragm **5763** downwards into contact and engagement with surface **5033** of the liquid-side recess **5031** and anti-stall grooves **5769** therein. The liquid is forced outwards from pump chamber **5037** under pressure through fluid outlet port **5767** and enters a discharge side microchannel **4018** for further processing in microfluidic manifold substrate **5000**. This completes one full pumping cycle, which can be repeated each time the slurry or another liquid (e.g., extractant, cleaning/flushing water, calibration standard liquid, etc.) is pumped.

[0144] According to another aspect of the microfluidic manifold processing substrate **5000**, the arrangement of microfluidic devices (e.g., micropumps **5015**, microvalves **4018**, etc.) and microchannels **4012** are configured to create a constant downward slurry flow path through the substrate from the initial slurry inlet to the substrate via microvalve **4018b** to the final slurry outlet via microvalve **4018e** (see, e.g. FIG. 30). In general, due to gravity, particulates or solids in the slurry are of higher density (weight) than the water carrier fluid. Due to this, the particulates have a tendency to settle out of suspension in the mixture in all fluid chambers and passageways they encounter while flowing through the microfluidic devices and microchannels. Designing a slurry manifold where slurry flows in a “generally” downward direction assisted by gravity continually promotes good cleaning of the flow passages to deter and minimize leftover particulate/sediment deposits. Such an arrangement is shown for example in FIGS. 30-31. The term “generally” is intended to connote that there may be some portions of the slurry flow passage where a slight deviation from vertical may occur. However, the slurry will still flow assisted by gravity in a generally downward direction from the slurry inlet port to the slurry outlet port to prevent any significant accumulation of particulates within the flow passage.

[0145] Referring to FIGS. 30-31, microfluidic manifold processing substrate **5000** is preferably vertically oriented in use as shown to form the continuously downward slurry flow path through the substrate. The slurry flow path is shown in emboldened dashed lines. Slurry enters microvalve **4018b** and flows downwards into the inlet port **5766** of first micropump **5015a** and out from outlet port **5767** to intermediate microvalve **4018f** to continue to second and third micropumps **5015b**, **5015c**. Extractant may then be drawn into the first micropump via extractant valve **4018a** in a similar manner to mix with the slurry for forming the slurry extractant mixture. Flushing water microvalve **4018c** and calibration standard liquid microvalve **4018d** are also located upstream of the first micropump **5015a** similarly to the slurry and extractant microvalves **4018b**, **4018a**. Water is used between slurry processing runs to clean and flush out the system. The calibration standard liquid is used to per-

form system calibration runs of the system for test extraction of analytes to ensure accuracy.

[0146] According to another aspect, the microvalves 4018 of the microfluidic manifold in the form of processing substrate 5000 are specially configured to form a positive seal between the inlet and outlet sides of the valves. This prevents flow leaking through the microvalves when in the closed position. Each microvalve is formed between liquid layer 5003 and air layer 5004 of the manifold. Microvalve 4018 may have a circular configuration comprising an air-side valve chamber 5058 recessed into air layer 5004, liquid-side valve chamber 5060 recessed therebelow into liquid layer 5003, and a resiliently deformable diaphragm 5763 separating the air-side and liquid-side valve chambers. In one embodiment, air-side valve chamber 5058 may be dome shaped forming a concavity defined by an arcuately curved wall 5058a and liquid-side valve chamber 5060 may comprise a flat base wall 5060a. Liquid-side valve chamber 5060 comprises an inlet side 5061 including recessed inlet side sub-chamber 5050 having a flat bottom wall penetrated by an inlet port 5053 for introducing a liquid into the microvalve from the microchannel network. Liquid-side valve chamber 5060 further comprises an outlet side 5062 including recessed outlet side sub-chamber 5051 having a flat bottom wall penetrated by an outlet port 5052 for discharging the liquid back into the microchannel network. Air-side valve chamber 5080 includes a pneumatic air pressure signal port 5057 for actuating the valve. The microvalve is changeable between open and closed positions to allow or shutoff/block liquid flow through the valve (e.g., slurry, extractant, cleaning water, calibration standard liquid, etc.), respectively. Pneumatically operated microvalve 4018 may operate in the same manner as micropump 4018 previously described herein (e.g., vacuum applied to open valve, pressure applied to close the valve). In other embodiments, the microvalve may alternatively open under a pressurized entry of liquid into the valve via inlet port 5053 and close via application of pressure via pneumatic air pressure signal port 5768 to air-side valve chamber 5060.

[0147] Each microvalve 4018 may further include a sealing bar 5054 fluidly separating and isolating the inlet and outlet sides 5061, 5062 of the valve when the diaphragm 5763 engages the sealing bar when the microvalve is in a closed position. Sealing bar advantageously creates a positive leak-resistant seal between the liquid inlet and outlet sides of the microvalve to promote positive shutoff in the closed position. Liquid is transferrable from the inlet side to the outlet side of the liquid-side valve chamber when the microvalve is in an open position in which the diaphragm disengages the sealing bar. Slight displacement allows flow to be established through the microvalve. Liquid-side valve chamber 5060 further comprises an annular protruding sealing ring 5055 circumferentially extending around the inlet and outlet sides of the liquid-side valve chamber. The sealing bar may be a linear structure which extends inside and is connected to the sealing ring between diametrically opposite points of the sealing ring as shown. In one embodiment, sealing ring and sealing bar have the same height such that the diaphragm 5763 engages the top surfaces of the sealing ring and bar when the valve is in the closed position. The sealing ring 5055 is surrounded by an annular diaphragm retention pocket 5056 recessed into the liquid layer 5003 which comprise an integral part of the microvalve 4018.

EXAMPLES

[0148] The following are nonlimiting examples.

[0149] Example 1—a microfluidic manifold for processing an agricultural slurry comprising: a micropump formed between a liquid layer and an air layer of the manifold, the micropump comprising a pump chamber collectively formed by an air-side recess and a liquid-side recess, and a resiliently deformable diaphragm separating the air-side and liquid-side recesses; the micropump further comprising a perimeter flow groove extending circumferentially around peripheral portions of the pump chamber.

[0150] Example 2—the microfluidic manifold according to Example 1, wherein the perimeter flow groove extends circumferentially around the liquid-side recess and is recessed into a top surface of the liquid layer.

[0151] Example 3—the microfluidic manifold according to Examples 1 or 2, wherein the diaphragm is formed of fluorosilicone.

[0152] Example 4—the microfluidic manifold according to any one of Examples 1-3, wherein the perimeter flow groove intersects and is fluidly coupled to a fluid inlet port and fluid outlet port of the micropump.

[0153] Example 5—the microfluidic manifold according to any one of Examples 1-4, wherein the perimeter flow groove is spaced inwards from a circumferentially-extending peripheral sidewall of the liquid-side recess.

[0154] Example 6—the microfluidic manifold according to Example 5, further comprising a protruding diaphragm seal ring disposed adjacent to the perimeter flow groove.

[0155] Example 7—the microfluidic manifold according to Example 6, wherein the seal ring extends circumferentially around and outboard of the flow groove.

[0156] Example 8—the microfluidic manifold according to Example 7, wherein the seal is configured to prevent ingress of the diaphragm into the perimeter flow groove when the liquid layer is bonded to the air layer.

[0157] Example 9—the microfluidic manifold according to Example 8, wherein the seal ring has a wider base than a terminal top end portion.

[0158] Example 10—the seal ring according to Example 9, wherein the seal ring comprises one or two obliquely angled surfaces at the top end portion forming a cross-sectional profile which is narrower than the base.

[0159] Example 11—the microfluidic manifold according to Example 9, wherein the seal ring has a trapezoidal or partial trapezoidal cross-sectional shape.

[0160] Example 12—the microfluidic manifold according to any one of Examples 6-11, wherein the seal ring is disposed between the peripheral sidewall of the liquid-side recess and the perimeter flow groove.

[0161] Example 13—the microfluidic manifold according to Example 12, further comprising a diaphragm seating pocket formed between the seal ring and the peripheral sidewall of the liquid-side pump chamber.

[0162] Example 14—the microfluidic manifold according to any one of Examples 1-13, wherein the liquid-side recess comprises a plurality of anti-stall grooves recessed into the liquid layer.

[0163] Example 15—the microfluidic manifold according to Example 14, wherein the anti-stall grooves are arranged in an orthogonally intersecting grid array.

[0164] Example 16—the microfluidic manifold according to Examples 14 or 15, wherein the anti-stall grooves intersect the perimeter flow groove.

[0165] Example 17—the microfluidic manifold according to any one of Examples 14-16, wherein the air-side recess comprises a plurality of intersecting anti-stall grooves recessed into the air layer.

[0166] Example 18—the microfluidic manifold according to Example 1, wherein the air-side recess has a domed shape wall and the liquid-side recess has a flat shaped wall.

[0167] Example 19—the microfluidic manifold according to any one of Examples 1-18, wherein the microfluidic manifold comprises a block shaped substrate, the liquid and air layers being formed internally within the substrate.

[0168] Example 20—a microfluidic manifold for processing an agricultural slurry comprising: a microvalve formed between a liquid layer and an air layer of the manifold, the microvalve comprising an air-side valve chamber, a liquid-side valve chamber, and a resiliently deformable diaphragm separating the air-side and liquid-side valve chambers; the liquid-side valve chamber comprising an inlet side comprising an inlet port for introducing a liquid into the microvalve, and an outlet side comprising an outlet port for discharging the liquid; the microvalve changeable between open and closed positions; and a sealing bar fluidly separating and isolating the inlet and outlet sides when the diaphragm engages the sealing bar when the microvalve is in a closed position.

[0169] Example 21—the microfluidic manifold according to Example 20, wherein the liquid is transferrable from the inlet side to the outlet side of the liquid-side valve chamber when the microvalve is in an open position in which the diaphragm disengages the sealing bar.

[0170] Example 22—the microfluidic manifold according to Examples 20 or 21, wherein the liquid-side valve chamber further comprises a protruding sealing ring circumferentially extending around the inlet and outlet sides of the liquid-side valve chamber, the sealing bar extending inside the sealing ring between diametrically opposite points of the sealing ring.

[0171] Example 23—the microfluidic manifold according to any one of Examples 20-22, wherein the sealing ring and sealing bar have the same height.

[0172] Example 24—the microfluidic manifold according to any one of Examples 20-23, wherein the air-side valve chamber has a dome shaped wall and the liquid-side valve chamber has a flat shaped wall.

[0173] Example 25—the microfluidic manifold according to any one of Examples 20-24, wherein the liquid is a slurry containing particulates.

[0174] Example 26—a microfluidic manifold for processing an agricultural slurry comprising: a substrate; a slurry inlet port formed in the substrate; a slurry outlet port formed in the substrate; and a slurry flow path formed internally within the substrate and fluidly coupling the slurry inlet port to the slurry outlet port; wherein the slurry flow path is configured such that the slurry flows assisted by gravity in a generally downward direction from the slurry inlet port to the slurry outlet port.

[0175] Example 27—the microfluidic manifold according to Example 26, wherein the slurry flow path is defined by a plurality of microchannels formed in the substrate.

[0176] Example 28—the microfluidic manifold according to Example 27, further comprising a plurality of microfluidic devices fluidly coupled together by the microchannels between the slurry inlet and outlet ports.

[0177] Example 29—the microfluidic manifold according to Example 28, wherein the microfluidic devices include at least one micropump and at least one microvalve.

[0178] Example 30—the microfluidic manifold according to Example 29, wherein the at least one micropump includes an inlet port and an outlet port, wherein the inlet port is fluidly coupled to a plurality of upstream microvalves by the microchannels.

[0179] Example 31—the microfluidic manifold according to any one of Examples 27-30, wherein the substrate has a rectangular cuboid form including opposing first and second major sides which are vertically oriented to produce the downward direction of the slurry in the flow path.

[0180] Example 32—the microfluidic manifold according to any one of Examples 27-31, wherein the slurry outlet port is lower in the substrate than the slurry inlet port.

[0181] Example 33—the microfluidic manifold according to any one of Examples 27-32, wherein the substrate is comprised of a plurality of polymeric layers bonded together.

[0182] Example 34—the microfluidic manifold according to Example 33, wherein the microchannels are formed between adjacent internal layers of the substrate.

[0183] While the foregoing description and drawings represent some example systems, it will be understood that various additions, modifications and substitutions may be made therein without departing from the spirit and scope and range of equivalents of the accompanying claims. In particular, it will be clear to those skilled in the art that the present invention may be embodied in other forms, structures, arrangements, proportions, sizes, and with other elements, materials, and components, without departing from the spirit or essential characteristics thereof. In addition, numerous variations in the methods/processes described herein may be made. One skilled in the art will further appreciate that the invention may be used with many modifications of structure, arrangement, proportions, sizes, materials, and components and otherwise, used in the practice of the invention, which are particularly adapted to specific environments and operative requirements without departing from the principles of the present invention. The presently disclosed embodiments are therefore to be considered in all respects as illustrative and not restrictive, the scope of the invention being defined by the appended claims and equivalents thereof, and not limited to the foregoing description or embodiments. Rather, the appended claims should be construed broadly, to include other variants and embodiments of the invention, which may be made by those skilled in the art without departing from the scope and range of equivalents of the invention.

What is claimed is:

1. A microfluidic manifold for processing an agricultural sample fluid comprising:

a micropump codefined between a liquid layer and an air layer of the manifold, the micropump comprising a pump chamber collectively formed by an air-side recess in the air layer and a liquid-side recess in the liquid layer, and a resiliently deformable diaphragm separating the air-side and liquid-side recesses; wherein the liquid-side recess comprises a plurality of anti-stall grooves recessed into the liquid layer.

2. The microfluidic manifold according to claim 1, wherein the anti-stall grooves are arranged in an orthogonally intersecting grid array.

3. The microfluidic manifold according to claim 2, wherein the air-side recess comprises a plurality of intersecting anti-stall grooves recessed into the air layer.

4. The microfluidic manifold according to claim 3, wherein the anti-stall grooves in the air layer are arranged in an orthogonally intersecting grid array.

5. The microfluidic manifold according to claim 2, wherein the air-side recess comprises a dome shaped wall and the liquid-side recess comprises a flat shaped base wall.

6. The microfluidic manifold according to claim 5, wherein the anti-stall grooves are recessed into the base wall.

7. The microfluidic manifold according to claim 1, wherein the air-side recess comprises a plurality of intersecting anti-stall grooves recessed into the air layer.

8. The microfluidic manifold according to claim 7, wherein the air-side recess has a dome shaped wall and the liquid-side recess has a flat shaped wall.

9. The microfluidic manifold according to claim 1, wherein the liquid-side recess comprises a fluid input port and a fluid outlet port configured to flow the sample fluid through the pump chamber.

10. The microfluidic manifold according to claim 9, wherein the air-side recess comprises an air pressure signal port configured to admit air to actuate the diaphragm.

11. The microfluidic manifold according to claim 1 further comprising a perimeter flow groove extending circumferentially around peripheral portions of the pump chamber.

12. The microfluidic manifold according to claim 11, wherein the anti-stall grooves intersect the perimeter flow groove.

13. The microfluidic manifold according to claim 12, wherein the perimeter flow groove extends circumferentially around the liquid-side recess and is recessed into a top surface of the liquid layer.

14. The microfluidic manifold according to claim 13, wherein the perimeter flow groove intersects and is fluidly coupled to a fluid inlet port and fluid outlet port of the micropump.

15. The microfluidic manifold according to claim 12, wherein the perimeter flow groove is spaced inwards from a circumferentially-extending peripheral sidewall of the liquid-side recess.

16. The microfluidic manifold according claim 15 further comprising a protruding diaphragm seal ring disposed adjacent to the perimeter flow groove, the seal ring extending circumferentially around and outboard of the flow groove.

17. The microfluidic manifold according to claim 16, wherein the seal ring is configured to prevent ingress of the diaphragm into the perimeter flow groove when the liquid layer is bonded to the air layer.

18. The microfluidic manifold according to claim 17, wherein the seal ring has a wider base than a terminal top end portion.

19. The microfluidic manifold according to claim 17, wherein the seal ring is disposed between a peripheral sidewall of the liquid-side recess and the perimeter flow groove.

20. The microfluidic manifold according to claim 19 further comprising a diaphragm seating pocket formed between the seal ring and the peripheral sidewall of the liquid-side pump chamber.

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