



(86) Date de dépôt PCT/PCT Filing Date: 2008/08/28

(87) Date publication PCT/PCT Publication Date: 2009/03/12

(45) Date de délivrance/Issue Date: 2014/10/28

(85) Entrée phase nationale/National Entry: 2010/02/16

(86) N° demande PCT/PCT Application No.: US 2008/074618

(87) N° publication PCT/PCT Publication No.: 2009/032745

(30) Priorité/Priority: 2007/08/28 (US11/846,372)

(51) Cl.Int./Int.Cl. *C08J 3/20* (2006.01),
B29B 9/00 (2006.01), *B29C 33/02* (2006.01),
B29C 41/04 (2006.01), *B29C 47/12* (2006.01),
C08J 9/34 (2006.01), *C08L 23/00* (2006.01)

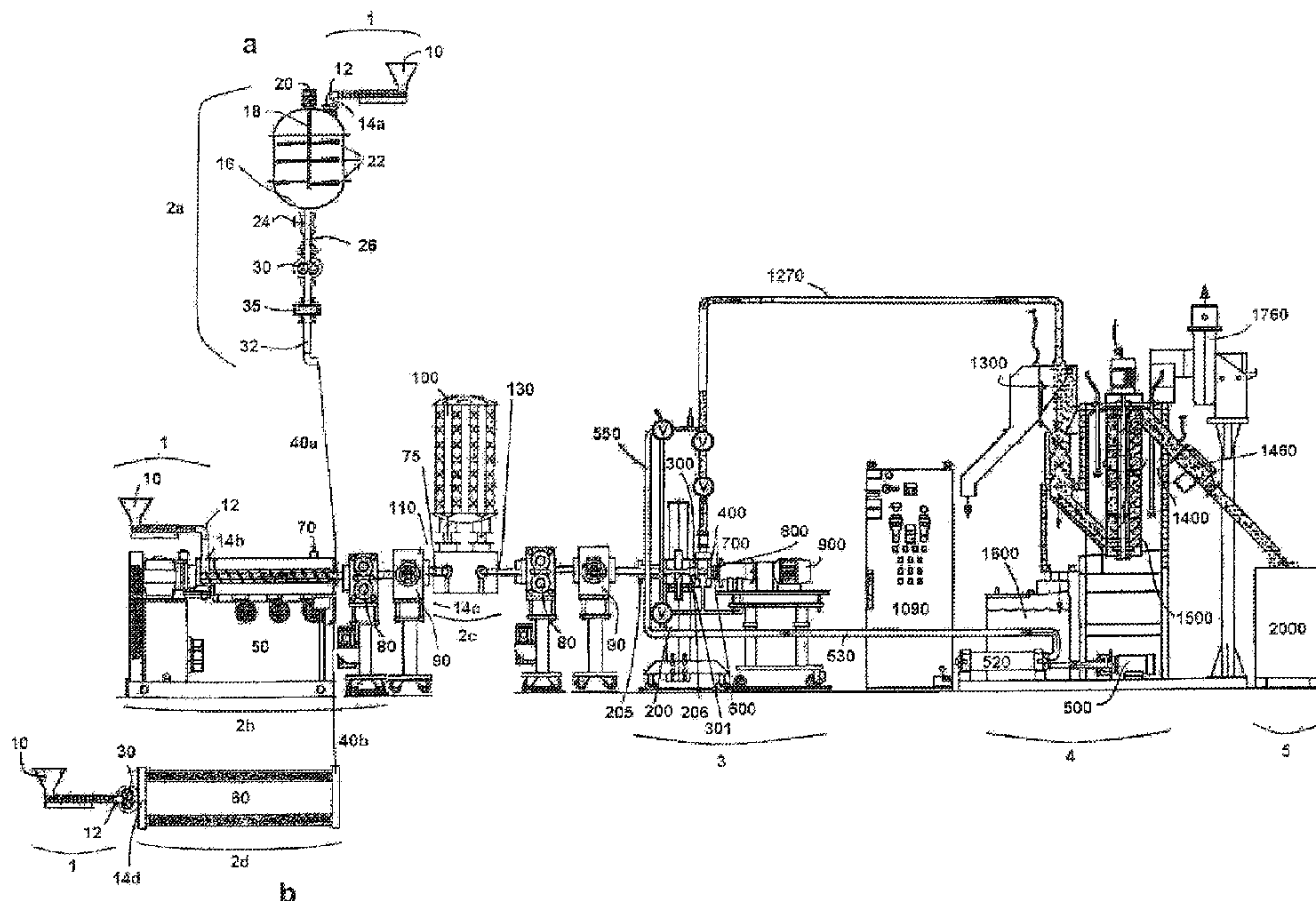
(72) Inventeurs/Inventors:
WRIGHT, ROGER BLAKE, US;
TORNOW, MATTHEW JEAN, US;
NEWBURY, SHAWN DAVID, US;
WEAVER, NATHAN EUGENE, US

(73) Propriétaire/Owner:
GALA INDUSTRIES, INC., US

(74) Agent: GOWLING LAFLEUR HENDERSON LLP

(54) Titre : PROCEDE ET APPAREIL POUR UN MOULAGE A CISAILLEMENT MINIMAL PERFECTIONNE, UTILISANT UNE EXTRUSION, UNE PELLETISATION ET UNE RHEOLOGIE A L'ETAT FONDU CONTROLEES DE PASTILLES ET DE MICROPASTILLES, ET OBJETS MOULES OBTENUS PAR CE PROCEDE ET CET APPAREIL

(54) Title: METHOD AND APPARATUS FOR ENHANCED MINIMAL SHEAR MOLDING UTILIZING EXTRUSIONAL, PELLETIZATION, AND MELT RHEOLOGICAL CONTROL OF PELLETS AND MICROPELLETS AND MOLDED OBJECTS MADE THEREFROM



(57) Abrégé/Abstract:

The various embodiments of the present invention relate to methods for enhancement of minimal shear molding processes. This can be accomplished using pellets and/or micropellets, along with optional powders. The pellets and/or micropellets can be produced through controlled extrusional and pelletization processes in combination with nonpowder melt flow rheology that incorporates reduced molding temperature, reduced cure time, reduced rotation axis ratios, and/or reduced rotation rates. Molding can be uniaxial, biaxial, or multiaxial, and rotational, oscillatory, or both. The molded items produced can have a single layer or multiple layers, and can be produced in one or more molding sequences using similar or different chemical compositions.



**(19) World Intellectual Property Organization
International Bureau**



PCT

(10) International Publication Number
WO 2009/032745 A3

(43) International Publication Date
12 March 2009 (12.03.2009)

(51) International Patent Classification:

C08J 3/20 (2006.01) **B29C 41/04** (2006.01)
C08J 9/34 (2006.01) **B29C 33/02** (2006.01)
C08L 23/00 (2006.01) **B29B 9/00** (2006.01)
B29C 47/12 (2006.01)

(21) International Application Number:

PCT/US2008/074618

(22) International Filing Date: 28 August 2008 (28.08.2008)

(25) Filing Language: English

(26) Publication Language: English

(30) Priority Data:

11/846,372 28 August 2007 (28.08.2007) US

(71) Applicant (for all designated States except US): GALA INDUSTRIES, INC. [US/US]; 181 Pauley Street, Eagle Rock, VA 24085 (US).

(72) Inventors; and

(75) Inventors/Applicants (for US only): **WRIGHT, Roger, Blake** [US/US]; 457 Middlebrook Road, Staunton, VA 24401 (US). **TORNOW, Matthew, Jean** [US/US]; 160 Lakeridge Circle, Troutville, VA 24175 (US). **NEWBURY, Shawn, David** [US/US]; 3409 Evangeline Street, Monroe, LA 71203 (US). **WEAVER, Nathan, Eugene** [US/US]; 88 Foxglove Lane, Berkeley Springs, WV 25411 (US).

(74) Agents: SHAHRIARI, Dean, Y. et al.; Troutman Sanders Llp, Bank of America Plaza, 600 Peachtree Street, N.E., Suite 5200, Atlanta, GA 30308-2216 (US).

(81) Designated States (unless otherwise indicated, for every kind of national protection available): AE, AG, AL, AM, AO, AT, AU, AZ, BA, BB, BG, BH, BR, BW, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DO, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, GT, HN, HR, HU, ID, IL, IN, IS, JP, KE, KG, KM, KN, KP, KR, KZ, LA, LC, LK, LR, LS, LT, LU, LY, MA, MD, ME, MG, MK, MN, MW, MX, MY, MZ, NA, NG, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RS, RU, SC, SD, SE, SG, SK, SL, SM, ST, SV, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, ZA, ZM, ZW.

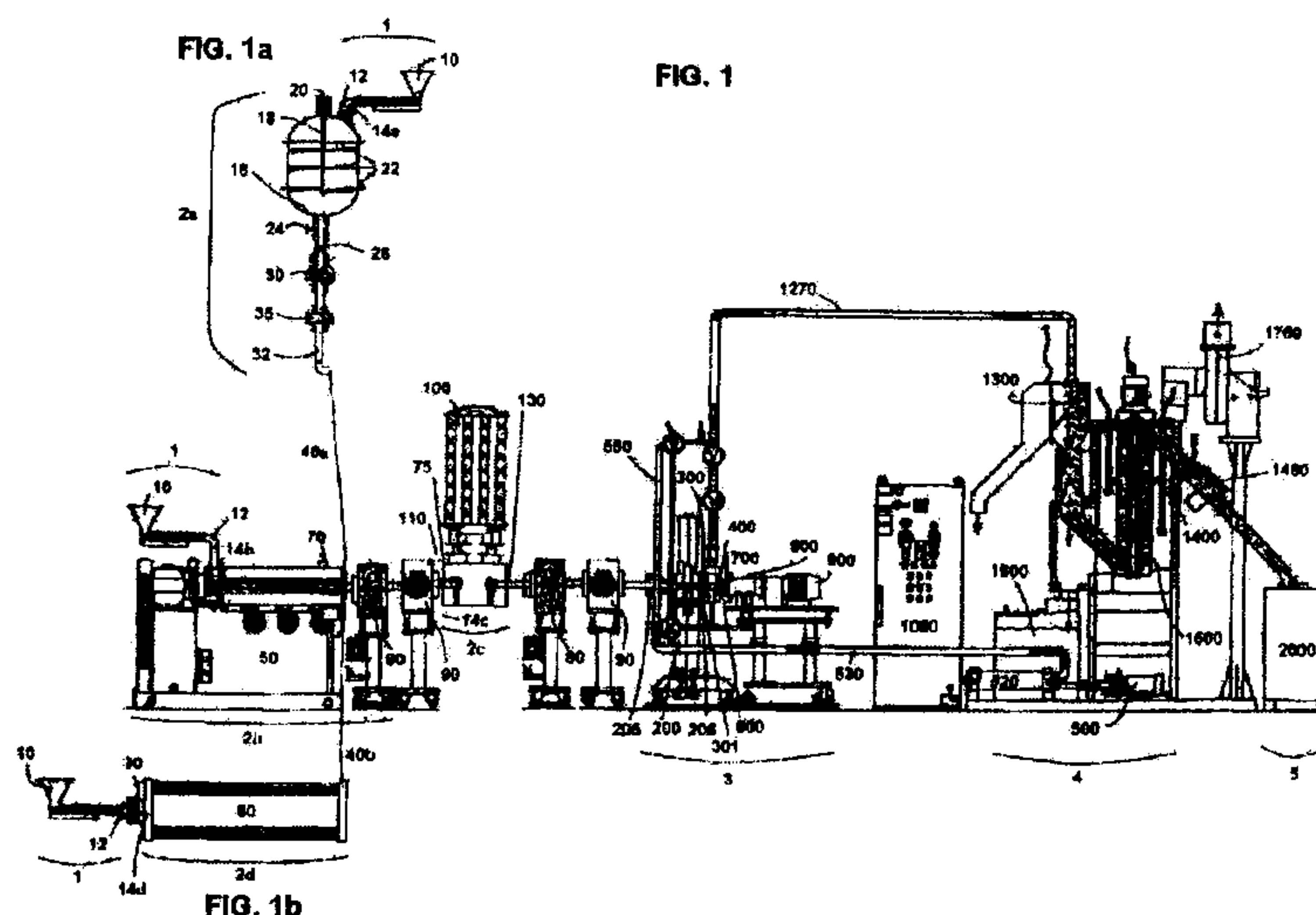
(84) Designated States (unless otherwise indicated, for every kind of regional protection available): ARIPO (BW, GH, GM, KE, LS, MW, MZ, NA, SD, SL, SZ, TZ, UG, ZM, ZW), Eurasian (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European (AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HR, HU, IE, IS, IT, LT, LU, LV, MC, MT, NL, NO, PL, PT, RO, SE, SI, SK, TR), OAPI (BF, BJ, CF, CG, CI, CM, GA, GN, GO, GW, ML, MR, NE, SN, TD, TG).

Declarations under Rule 4.17:

— *as to applicant's entitlement to apply for and be granted a patent (Rule 4.17(ii))*

[Continued on next page]

(54) Title: METHOD AND APPARATUS FOR ENHANCED MINIMAL SHEAR MOLDING UTILIZING EXTRUSIONAL, PELLETIZATION, AND MELT RHEOLOGICAL CONTROL OF PELLETS AND MICROPELLETS AND MOLDED OBJECTS MADE THEREFROM



(57) Abstract: The various embodiments of the present invention relate to methods for enhancement of minimal shear molding processes. This can be accomplished using pellets and/or micropellets, along with optional powders. The pellets and/or micropellets can be produced through controlled extrusional and pelletization processes in combination with nonpowder melt flow rheology that incorporates reduced molding temperature, reduced cure time, reduced rotation axis ratios, and/or reduced rotation rates. Molding can be uniaxial, biaxial, or multiaxial, and rotational, oscillatory, or both. The molded items produced can have a single layer or multiple layers, and can be produced in one or more molding sequences using similar or different chemical compositions.

WO 2009/032745 A3



— *as to the applicant's entitlement to claim the priority of the earlier application (Rule 4.17(iii))*

— *before the expiration of the time limit for amending the claims and to be republished in the event of receipt of amendments*

Published:

— *with international search report*

(88) Date of publication of the international search report:

28 May 2009

**METHOD AND APPARATUS FOR ENHANCED MINIMAL SHEAR MOLDING
UTILIZING EXTRUSIONAL, PELLETIZATION, AND MELT RHEOLOGICAL
CONTROL OF PELLETS AND MICROPELLETS AND MOLDED OBJECTS MADE
THEREFROM**

BACKGROUND OF THE INVENTION

1. Field of the Invention

The present invention relates generally to a non-powder process to extrude pellets, more particularly micropellets, to control the size and shape of the pellets as well as retain the chemical integrity of the pellets insuring they are as free as possible of undesirable degradation, premature oxidation, discoloration, premature reaction, expansion, or cross-linking, and to further insure they are of approximately uniform mixture or blend of composition for use in uniaxial, biaxial, and multiaxial minimal shear molding to facilitate enhancement of process temperature, rotation ratios, and rotation rates in view of conventional processes.

2. Description of the Prior Art

Individual processes and equipment for extrusion, pelletizing, drying, conveying, rotational molding, and rock-and-roll molding generally have been known and used in various applications. Separately, chemistries involved in this art have been known. The application of these processes and equipment to the possible chemistries and the extension to enhancement of the minimum shear processes of rotational and rock-and-roll molding has not been readily apparent from the prior art disclosures, and is a focus of the present invention.

WO 2009/032745

PCT/US2008/074618

Pelletization equipment and its use following extrusion processing has been introduced and/or utilized in applications by the assignee for many years as is exemplified by prior art disclosures including US Patent Nos. 4,123,207; 4,251,198; 4,500,271; 4,621,996; 4,728,176; 4,888,990; 5,059,103; 5,403,176; 5,624,688; 6,332,765; 6,551,087; 6,793,473; 6,824,371; 6,925,741; 7,033,152; 7,172,397; US Patent Application Publication Nos. 20050220920, 20060165834; German Patents and Applications including DE 32 43 332, DE 37 02 841, DE 87 01 490, DE 196 42 389, DE 196 51 354, DE 296 24 638; World Patent Application Publications WO2006/087179, WO2006/081140, WO2006/087179, and WO2007/064580; and European Patents including EP 1 218 156 and EP 1 582 327.

Similarly, dryer equipment has been introduced and used in applications following extrusion and pelletization for many years by the assignee as demonstrated in, for example, US Patent Nos. 3,458,045; 4,218,323; 4,447,325; 4,565,015; 4,896,435; 5,265,347; 5,638,606; 6,138,375; 6,237,244; 6,739,457; 6,807,748; 7,024,794; 7,172,397; US Patent Application Publication No. 20060130353; World Patent Application Publication No. WO2006/069022; German Patents and Applications including DE 19 53 741, DE 28 19 443, DE 43 30 078, DE 93 20 744, DE 197 08 988; and European Patents including EP 1 033 545, EP 1 602 888, EP 1 647 788, EP 1 650 516.

Minimal shear molding equipment including but not limited to rotation molding and rock-and-roll molding equipment and following from slush molding equipment are similarly disclosed in, for example, US Patent Nos. 2,629,131; 2,893,057; 3,134,140; 3,315,314; 3,350,745; 3,564,656; 3,676,037; 3,703,348; 3,788,792; 3,810,727; 3,822,980; 3,825,395; 3,841,821; 3,843,285; 3,914,105; 4,022,564; 4,247,279; 4,671,753; 4,705,468; 4,738,815; 4,956,133; 4,956,135; 5,022,838; 5,039,297; 5,188,845; 5,238,379; 5,705,200; 6,030,557; 6,296,792; 6,338,623; 6,511,619; 6,555,037; and 6,604,931; US Patent Application Publication No. 2006/0257518; Canadian Patent No. 2 025 267; Japanese Patent Application No. JP 2005028803; and World Patent Application Publication No.

WO 2009/032745

PCT/US2008/074618

WO 1999/056930.

Various pulverulent materials have also been utilized for minimal shear molding including polyethylenes and other polyolefins, vinyl esters and other vinyl polymers, fluoropolymers, polyamides, polyesters, polycarbonates, acrylonitrile-butadiene-styrene (ABS) copolymers, and reactive polymers such as cross-linkable polyethylene (XLPE). Additive packages have been introduced to confer antioxidant stabilization, thermal stabilization, ultraviolet (UV) stabilization, pigmentation, flow modification, processing modification, plasticization, foaming, and/or cross-linking capabilities, among others.

Le Roy Payne has introduced equipment and methodology through, for example, US Patent Nos. 4,671,753; 4,671,991; 4,749,533; 4,909,718; 4,956,133; 4,956,135; 5,011,636; 5,022,838; 5,173,221; 5,188,845; 5,238,379; 5,316,701; 5,503,780; 5,507,632; 5,705,200; 6,030,557; 6,296,792; 6,511,619; 6,555,037; Canadian Patent No. CA 2 025 267; and World Patent Application Publication No. WO 1999/056930

that demonstrate the use of pourable polymerizably reactive polyurethane or polyester rotational molding in one or more axes of rotation in optional combination with oscillatory or rock-and-roll processing to generate rotomolded parts. The use of microspheres as additives to at least one of the polymerizable liquid ingredients is demonstrated in US Patent No. 6,555,037, but this reference is silent on the use of pellets or micropellets as described in the current invention.

Early references for rotational molding compositions include use of pellets that were typically more cylindrical and often were ground to provide powders. US Patent No. 3,514,508 discloses the use of preferably 20 mesh to 50 mesh particles of polyethylene, polypropylene, and polyvinyl chloride in that oven temperatures of 350°F to 950°F (approximately 177°C to 510°C) are suggested with an inert gas such as nitrogen or carbon dioxide present inside the mold to confer a degree of positive pressure. Larger size particles, up to two (2) millimeters (mm) are disclosed. An acrylonitrile-butadiene-styrene (ABS) resin containing additive was prepared in sizes ranging from a minus 10 mesh to plus 100 mesh for rotational casting as disclosed in US Patent No. 3,935,143. Masterblends are disclosed in US Patent No. 4,508,859 wherein the resin particles and additives are mixed below the softening temperature and this blend, upon

transfer to an intensive mixer, softens the granules sufficiently without fluxing to allow the additive to be absorbed into or onto the granule. Some rounding of the resin granules is anticipated and US Patent No. 4,508,859 describes use of a 30 or 35 mesh screen to remove unacceptably large granules. US Patent No. 4,624,818 discloses nylon granules ranging in size from 12 to 35 mesh containing abrasive-resistant components preferably ranging in size from 24 mesh to 500 mesh. Similarly, US Patent No. 4,970,045 discloses the formation of pellets or powder of polyamides and ionomeric copolymers for rotational molding. Controlled pelletization to achieve reproducible particle size distributions, uniform blends, and/or optimized molded article quality or performance was not disclosed.

In US Patent No. 5,525,274, microspheres are prepared by extruding and flaking polyurethane, which is then mixed with other additives including plasticizers. The combination is then re-extruded through orifices ranging from 0.007 inches (") to 0.040" (approximately 0.178 mm to 1.02 mm). Similarly, polyurethane, polypropylene, polyethylene, polystyrene or polyvinyl chloride is blended with additives including a plasticizer, with the resin comprising 50% to 55% of the mixture with extrusion to generate particles dimensionally equivalent as described above. An objective of the US Patent No. 5,525,274 appears to be to lock the plasticizer into the resin matrix to obtain a cast part of uniform thickness and free of "backside irregularities". A 50,000 to 150,000 molecular weight range for polyvinyl chloride (PVC) is disclosed. Also disclosed is that lower molecular weight resins reduce the process temperature and decrease the cycle time.

US Patent No. 5,525,284 is a divisional patent of US Patent No. 5,525,274. In this reference, the highly plasticized microspheres are rotationally cast using a predetermined charge size equal to or greater than the weight of the part being molded or slush-molded to form thin plastic shells.

US Patent No. 5,654,102 is a divisional patent of US Patent Nos. 5,525,274 and 5,525,284. In this reference, the composition of the microspheres containing plasticizer, limited to 50% to 55% resin, are disclosed to be polyurethane, polypropylene, polyethylene, polystyrene, polyvinyl chloride, or epoxies or alloys thereof.

US Patent Nos. 5,998,030 and 6,410,141 are continuations-in-part patents to US Patents 5,525,274; 5,525,284; and 5,564,102. These references extend the molecular

weight range from 2,000 to 150,000. Plasticizer remains a component, although optional, and the resin level, in one example therein, is within the 50% to 55% range as described above. These patents require that pigment be present, at a level less than 5% by weight.

A pelletizer die for extruding micropellets is described in US Patent No. 5,597,586. The die requires a multiplicity of die holes arranged in groups concentrically about the face of the die. At least one of the groups is composed of die holes of uniform diameter but different than the other groups, or subgroups within the groups of die holes are uniform in diameter but different than the other subgroups of that group. It has been found that excessive pressure generation and freeze-off effectively block at least some of the die holes, and this has proven problematic for attempts at implementation of the die.

US Patent No. 4,238,537 discloses the use of a mixture of pellets and powder, both composed of various ethylene-vinyl-acetate copolymers for rotational molding. The specification discloses that the pellets are typically 1/8 inch by 1/8 inch (approximately 3.2 mm by 3.2 mm) and the powder is ground between 20 mesh and 50 mesh. The disclosed blend is from 50% to 80% pellets and inversely from 50% to 20% powder. Molding is generally done between 500°F and 625°F (approximately 260°C to 329°C). Complete coalescence of pellet and powder is not requisite according to US Patent No. 4,238,537, where it is disclosed that the pellets retain their particle characteristics at least in part.

European Patent No. EP 0 755 761 and US Patent No. 5,585,419 disclose the use of micropellets, 0.028 inch to 0.051 inch (approximately 0.7 mm to 1.3 mm) in diameter, in combination with powder, 0.012 inch to 0.035 inch (approximately 0.3 mm to 0.9 mm) and irregularly shaped, of similar composition for use in rotational molding. Similarly US Patent No. 5,886,068 discloses use of two different micropellet sizes of similar composition wherein the micropellets have a diameter from 0.024 inch to 0.039 inch (approximately 0.6 mm to 1.0 mm) in combination with the larger size ranging from 0.028 inch to 0.051 inch (approximately 0.7 mm to 1.3 mm). Materials disclosed include polyvinyl chloride, polyethylene, polypropylene and SURLYN. Oven temperatures of 600°F to 800°F (approximately 316°C to 427°C) are maintained where the melting temperature is listed at 400°F to 550°F (approximately 204°C to 288°C). Rotation ratios of 3.75:1 or multiple equivalents are disclosed; and large pellet sizes are discouraged as

they are disclosed to require longer times to melt and thus fuse after the powder or smaller micropellets have already begun to lay down on the surface of the mold. US Patent No. 5,886,068 is silent regarding an attempt at separation of layers or use of different materials.

Similarly, US Patent Nos. 6,433,086 and 6,682,685 describe a rotational molding process using a combination of polyethylene pellets and ground polyolefin powder of the same composition wherein the powder is from 20% to 30% or 20% to 50%, respectively, of the weight of the blend. A single layer is formed from this resin blend that, following US Patent No. 6,682,685, can include reinforcing fibers to improve structural properties. US Patent No. 6,833,410 discloses fibers including organic, inorganic, and mineral reinforcing fibers utilized at a loading level of 10% to 80% by weight of a comparable resin pellet to resin powder blend, but the resin composition is not disclosed.

US Patent No. 3,368,013 discloses sintering only the edges of various shaped pellets to make multi-vented hollow articles. Similarly, pellets of less than 1/8 inch (approximately 3.2 mm) are sintered according to US Patent No. 6,030,558 to make porous plastics. The size of the porosity is controlled by the pellet size and sintering is done at temperatures sufficient to only fuse the outside surfaces of the pellets. Mention is made of the very narrow particle distribution size range that is particularly beneficial in an application such as selective filtration. For low density polyethylene, temperatures are cycled from ambient (or room temperature) to 320°F (approximately 160°C) and then cooled over a five (5) minute period where particle sizes are 0.031 inch (approximately 0.8 mm). For particle sizes larger than this, cycling as above is followed with heating from two (2) to four (4) minutes at 320°F (approximately 160°C) prior to cooling.

WO 2000/035646 demonstrates the use of polyolefin pellets ranging from two to ten carbon monomers, particularly ethylene and propylene. According to claim 1, the distribution of the particle sizes should meet the criterion of a maximum value of one (1) for a ratio of the difference of the size distribution of an average 0.035 inch (approximately 0.9 mm) particle size and that of an average 0.004 inch (approximately 0.1 mm) particle compared to that of an average 0.020 inch (approximately 0.5 mm) particle size distribution; and the pellets should contain a water content of less than 0.1% by weight. An apparatus requiring at least one additive to be combined with the

polyolefin in a mixer, an extruder and pelletizer, a dewatering centrifuge and a fluidized bed drier is described. Rotational molding of the claimed material is also disclosed. WO 2000/035646 also describes the residual water content as being no more than 1% by weight. US Patent Application Publication No. 2005/0228118 is a divisional patent application of US Patent No. 6,894,109, which is itself a divisional patent of US Patent No. 6,573,314, and only claims minimal or no presence of pinholes in a rotationally molded article. US Patent No. 6,894,109 reduces this water content to a value less than 0.1% as disclosed in the herein cited WO 2000/035646.

US Patent No. 6,632,525 discloses the manufacture and use of a plastic particle, with a diameter ranging from 0.007 inch to 0.040 inch (approximately 0.18 mm to 1.0 mm), that has a protruding ring on the outer surface of that particle. The ring can be continuous or non-continuous and is produced in an additional heating step following extrusion, pelletization, and cooling of the original pellet without the ring.

Multilayer molded articles have been described in the literature as well. US Patent No. 3,542,912 discloses the use of a mixture of granules, 35 mesh or finer, where the components form individual layers differing in melting point by at least 10°F (approximately 5°C). The individual components are described as immiscible. Rotation rates of 20 to 100 rotations per minute (rpm) are also described. Oven temperatures are necessarily disclosed at a minimum 20°F (approximately 11°C) above the melting point of the highest melting component and typically are maintained at 550°F (approximately 288°C). In exemplary embodiments, the inner layers, and the higher melting layer, are formed from nylon, polycarbonate, acetal, polyphenylene oxide, polysulfones and polyester. The highest melting layer necessarily should also have a higher density than at least one of the other layers. The external (hence lower melting) layer for nylon is suggested to be composed of polyethylene, polypropylene, polyurethane, polystyrene, cellulose acetate, cellulose propionate, cellulose acetate butyrate, acrylonitrile-butadiene-styrene, polyphenylene oxide, polysulfone, styrene-butadiene or polyvinyl chloride. For polycarbonate inner layers only nylon is recommended. Acetal, polyester, or polysulfone inner layers complement polyethylene or polypropylene outer layers. Three-layer structures composed of nylon – acetal – polyethylene or polypropylene, or alternatively of polycarbonate – nylon – polyethylene or polypropylene, are described.

Sequential formation of layers of similar composition is disclosed in US Patent No. 3,627,869, where particles ranging from 10 mesh to 400 mesh are deemed acceptable with 16 mesh (0.047" or approximately 1.2 mm) to 50 mesh (0.012" or approximately 0.3 mm) considered suitable for rotational molding. Rotation speeds of 10 to 20 rpms are disclosed with oven temperatures given as ranging from 500°F to 700°F (approximately 260°C to 271°C) or higher, with 650°F (approximately 343°C) preferred. Polyolefin homopolymers or copolymers were described for each layer. Similarly, US Patent No. 4,548,779 generates a first higher melting outer layer then cools the mold and introduces a material to form the inner layer. Preferentially the outer layer is polyamide and the inner layer is a polyolefin.

Japanese Patent Abstract Publication No. JP 03-000216 discloses a multi-step, multi-layer article rotationally molded from powder at approximately 460°F (approximately 238°C) wherein each layer is separately charged into the mold. Japanese Patent Abstract Publication No. JP 2006-095928 similarly discloses a two-step, two-layer process wherein the second material, nylon, is introduced into the mold through a feed nozzle. US Patent No. 6,982,057 similarly discloses the two-step, two-layer process wherein polyethylene, polypropylene or nylon is used as the first layer and the inner layer should be transparent. Oven temperatures of 375°F to 650°F (approximately 191° to 343°C) are described. Linear low-density polyethylene was molded at a rotation ratio of 8:2 and 540°F (approximately 282°C). The first layer is fashioned to be sufficiently thin to allow removal of an insulating member such that the second flowable component can be poured through the exposed gap. By way of example, the clear second layer now seals over the gap to form a sight line.

Multilayer articles utilizing foamable layers have been made using rotational molding as well. US Patent No. 3,419,455 discloses a decorative object made from a thermoplastic outer shell and a rigidifying inner core can be a layer or can completely fill the inner chamber of the molded article. A skin of reactable resin is introduced into a mold containing a capsule of foaming agent and subjected to rotational molding according to US Patent No. 3,527,852. Once the skin layer has been formed the foaming agent is controllably released to generate a foamed core article. Similarly, US Patent No. 3,541,192 introduces a plastisol for formation of the skin layer and then introduces a

second foamable or expandable plastisol for controlled generation of the foamed core. US Patent No. 3,914,361 generates an outer skin and inner foamed core utilizing a plastic powder and a larger expandable particulate simultaneously charged into the sealable mold. Heating from outside melts powder to form the crust and then expands larger particulates to generate the foamed interior.

US Patent No. 3,936,565 introduces a sandwich structure in that the first layer is charged into the mold and uniformly coated onto the mold surface. The first layer is preferably a cross-linkable polyethylene and the molding temperature is sufficiently controlled to prevent complete cross-linking. On completion of the melt lay-down of the first layer, a second, preferably foamable, layer is charged into the mold and similarly layered without foaming. An optional third layer can be placed on the inner surface of the second layer and the cross-linking and foaming operations are performed simultaneously to complete the molding process.

US Patent No. 5,532,282 discloses the simultaneous introduction of a small non-expandable powder, typically 20 to 50 mesh and a considerably larger expandable granule or pellet that is typically 0.040" to 0.15" (approximately 1.0 mm to 3.8 mm) in diameter and 0.040" to 0.125" (approximately 1.0 mm to 3.2 mm) in length, and is specifically disclosed as not needing to be a sphere. Oven temperatures of 580°F (approximately 304°C) are described as exemplary. It is postulated that the smaller particulate melts preferentially allowing the larger pellets to form a second layer and subsequently foam after the first layer is formed. Both a foamed layer and a foamed core are disclosed. The skin-forming layer can be cross-linkable as presented. US Patent Nos. 5,783,611, 5,830,392, 5,922,778, and 6,038,434 utilize similar methodologies in that improvements of surface properties are achieved by carefully controlled chemical formulation and blending. This concept is further extended in US Patent No. 6,180,203 to include the use of reclaimed material in the larger, higher melting layer that forms the inner skin layer. This additionally provides optional reinforcement rods and a foamable layer between the inner and outer skin of the molded and expanded item.

A mixture of high zero-shear viscosity and low zero-shear viscosity polymeric materials, and a third foamable core-forming component are disclosed in US Patent No. 5,928,584. The patent describes the high viscosity material as a wall-forming barrier that

layers against the inner mold surface and forms a barrier to the foamable component preventing its contact with the mold wall. The low viscosity component, that is preferentially cross-linkable, serves as a binding element for the wall-forming layer and the inner foamable core-forming component.

Cross-linkable polyolefins and other reactive resins are well-known in rotational molding and include reactive residual components in the polymeric structure or incorporate “additive packages” that generate bonds, typically by free radicals or other covalent linkages, between portions of proximally located polymers. These additive packages typically include a cross-linking agent, a multifunctional material, and a facilitating agent to enhance the likelihood of cross-linking. Exemplary prior art discussion of such packages includes US Patent Nos. 3,876,613; 3,969,475; 4,029,729; 4,088,714; 4,267,080; 4,414,340; 4,526,916; 4,587,318; 4,678,834; 4,808,364; 4,857,257; 4,900,792; 5,260,381; 5,367,025; 6,528,585; and 6,610,768. Additional examples include US Patent Application Publication Nos. 2005/02024338, 2005/0261430, and 2005/0261443.

Various products are made by rotational molding. These include toys, chemical storage tanks, water tanks, vehicular fuel tanks, and automotive parts, among others. Decorative effects in polymers have been disclosed, including marbling and nacreous effects of salt additives in US Patent No. 2,330,108, as well as granite-like effects from incorporation of a multiplicity of particulates of comparable or equal density to, and suspended in, typically a transparent or translucent matrix as demonstrated in US Patent No. 5,304,592.

It is an object of the present invention to provide a cost effective method to prepare reproducible pellets and micropellets by controlled extrusion and pelletization with appropriate drying of rotomolding and extrusional grade materials, and rotomolding and extrusional formulations including additives such that minimal or no degradation or undesirable reaction of the components or pellets occurs, and to use those pellets to form quality single- or multiple-layer molded articles utilizing minimal shear rotational and oscillatory processes (including slush-molding), capable of utilizing, in view of the conventional processes, lower processing temperature, reduced processing time, reduced rotation ratios and reduced rotation rates, reducing storage volumes, facilitating in-mold

devolatilization, reduction of bridging, ease of transport, safety of operation, reduction of dust hazards, and lower shipment costs by increasing intermediate bulk density such that the nonpowder processes produce a product comparable or superior to the industry standard powder-based products in impact strength, permeability, reproducibility of production, intricacy and trueness of mold detail, uniformity of layer thickness, integrity of layers, bond strength between layers, interlayer uniformity, minimization of bubbles and pinholes and sinks or pockmarks, reduction or elimination of porosity, inner surface quality, structural integrity, gel formation, uniformity of pigmentation and decorative effects.

BRIEF SUMMARY OF THE INVENTION

Briefly described, the various embodiments of the present invention provide a nonpowder process to extrude micropellets, to control the size and shape of the micropellets as well as retain the chemical integrity of the micropellets. These controllably produced micropellets are used in accordance with the various embodiments of the present invention in minimal shear molding such as rotational or rock-and-roll processes, and they facilitate considerable savings in energy costs by reduction of the process temperature and/or process time as well as reduce the wear and tear on the equipment by allowing considerable reduction in rotation ratios and/or rotation rates of the one or more rotational support arms.

It has been found that transport of the micropellets is greatly simplified and shipping costs are tremendously reduced by virtue of the significantly increased bulk density of the micropellets as compared with the more standard bulk powders of the industry. The use of extruded micropellets extends the scope of usable materials by allowing the direct preparation of materials not traditionally marketed for minimal shear molding through uniform incorporation of antioxidant, ultraviolet, and other stabilizer packages, including thermal stabilizers for improving the preparation lifetime in the mold as well as post-molding lifetime.

The micropellets are more reproducible in structure, being more spherical in geometry, and have a more controllable surface area, which significantly impacts the rotational mechanisms by that the material is layered onto the surface of the mold in accordance with the present invention. This control allows facile extension of the one

layer molding process to the multiple layer process in that the layers can be formed sequentially or in a single charge of material into the mold. Layering of the multi-component charge can be achieved by at least one of several mechanisms including, but not limited to, melt temperature, softening temperature, pellet size, pellet density, melt density, melt viscosity, melt flow, surface properties including wettability of melt for metal surface or underlayers, and differences of polarity or chemical composition.

The use of micropellets in accordance with some embodiments of the current invention enhances control of pigmentation to achieve uniformity of coloration, to effect marbling, or to achieve other decorative effects. Use of the more spherical micropellets significantly improves the inside molded surface properties and allows greater control of bubble and pinhole removal and devolatilization of low-boiling components that can be by-products, processing aids, or excipients used in, or generated by, the extrusion, pelletization, melt formation, layering, reaction, and/or molding process or processes. The process as described herein does not negatively impact the quality of the item being molded as compared with the quality of the contemporary art of traditional powder molding processes.

Prior art has demonstrated the effectiveness of using powders in minimal shear molding to generate sundry articles from toys to pipe liners to automotive parts to boats to tanker tanks holding thousands of gallons and even to tanker liners. Limitations of the powder technology have also been documented. Exemplary limitations include irregularity and nonreproducibility of particle size and shape, variation in particle size distribution, problematic flow dynamics, irregularities of melt and subsequent lack of uniformity in lay-down during molding, problematic volumes in small or intricate molding in particular affected by bulk density constraints, undesirable bridging and inconsistency in lay-down for intricate or constrictive mold designs, nonuniformity of wall thickness in molded article, instability of powder components during grinding leading to potential degradation or discoloration, as well as maintenance, operational, and safety concerns from potential dust, inhalation, fire, and explosion hazards.

Materials that can be used and/or formulated for minimal shear molding applications have traditionally been limited in variety and particular grades or

formulations have often been unexpectedly removed from the market leading to challenges for the molder.

Pellets have also been initially introduced to the market as typically, and undesirably, cylindrical in shape and often of sufficiently (and undesirably) large size to pose limitations in their utility due in part to the challenges in melting and uniformity of lay-down, resulting in unacceptable or poor quality end-products. As pellet sizes decreased with increasingly sophisticated manufacturing equipment, their utility has been brought to the market following closely in processing typically with the original powder molding conditions, thus necessitating significant plasticization (assuming the need for uniformity of distribution of components throughout the multiplicity of particles), requiring combination with powders or pellets of differing size (assuming the need for pellet sizes comparable to powders or even smaller), and/or utilizing excessive heating or aggressive rotation sequences. The result of that has often been poor molding quality, lack of uniform lay-down, and irregular mold surfaces, significant problems with pinholes or formation of sinks and pockmarks, nonuniformity of composition or pigmentation or wall thickness, limitation of material considerations restricting the scope of useful materials and thus of useful end-products, and even necessitating risky or unsafe production manipulations to achieve desirable effects of structural or barrier properties such as multiple layer formation.

What is needed then is a method of generating relatively consistent and reproducible pellets or micropellets that are easily conveyable and can readily be incorporated into minimal shear molding processes that optionally contain additives including various stabilizer packages for enhancement of the pelletization process, the molding process, and/or the shelf-life of the end-product; or in combination, reactive polymers or formulations for improvement of structural strength, barrier properties, impact strength, and/or modification of flexural properties; pigments or ultraviolet absorbers; heat absorption; reflection components; and the like. With the pellets and micropellets thus generated, it is desirable to have a method for the molding process that is energetically attractive, reduces maintenance of the equipment through less aggressive molding techniques, improves the quality of the molding process and ultimately the

molded part, eliminates the expense and inconsistency of grinding, and is more cost effective for transport and storage.

The various embodiments of the present invention provide a composition for minimal shear molding, a process of minimal shear molding, and an apparatus for minimal shear molding.

Both the present composition and process can operate within four parameters to provide a novel and non-obvious composition and process. In exemplary embodiments, an apparatus is provided such that pelletization, transport, and drying of pellets/micropellets does not degrade, prematurely react, expand, prematurely cross-link, and otherwise alter the chemical composition of the pellets/micropellets for use in minimal shear molding.

Some embodiments of the present invention provide a composition for minimal shear molding of at least one layer, wherein the composition includes pellets, micropellets, powders, or a combination comprising at least one of the foregoing. The preferred composition comprises at least one chemical composition that can simultaneously form the at least one layer such that, for example, the following four parameters are met. Firstly, the composition for a single layer contains less than 20% of the minor component, and the size of the powder, if used, does not have to be smaller than the size of the pellet. Secondly, the pellets, micropellets, and/or powders used to form each layer in multiple layers are compositionally different such that each layer is compositionally different. Thirdly, combinations of one size pellet and one size micropellet are not used for a single layer. And lastly, a single layer is not formed from powder alone.

The chemical compositions can include polymers, copolymers, additives, and combinations thereof. Further, the polymers, copolymers, and additives can contain reactive functionalities, which can be cross-linkable. The reactive functionalities can be modified by chemical reaction, including expandable.

The polymers can be polyolefins, polyamides, polyesters, polycarbonates, polyurethanes, polyethers, cellulose, and copolymers thereof.

The polyolefins can be ultra-low density polyethylene (ULDPE), linear low density polyethylene (LLDPE), low density polyethylene (LDPE), medium density

polyethylene (MDPE), high density polyethylene (HDPE), polypropylene, alkyl- and aryl-substituted vinylics, halogenated and polyhalogenated vinylics, polyvinyl esters, polyvinyl alcohols, and copolymers thereof.

The polymers, copolymers, and additives can be amorphous, crystalline, or a combination thereof.

The additives can compositionally include rheology modifiers, cross-linking facilitating agents, antioxidant agents, ultraviolet stabilizers, thermal stabilizers, pigments, fillers, fibers, nucleating agents, expanding agents, tackifiers, detackifiers, pellet coatings, plasticizers, waxes, processing aids, and pelletizing aids. The additives can be single-component and multi-component formulations.

The pellets and micropellets can include many geometric configurations, such as from spherical to lenticular.

The pellets and micropellets can be controllably pelletized such that degradation, expansion, premature reaction, and premature cross-linking are minimized.

The pellets, micropellets, or both can contain less than approximately 0.5 weight percent fines.

The pellets can have an average longest cross-sectional dimension of approximately 0.7 millimeters (mm) to approximately 3.5 mm. In some cases this dimension can be approximately 0.7 mm to approximately 1.6 mm; and in other cases it can be approximately 0.7 mm to approximately 1.0 mm. Micropellets are generally smaller than approximately 0.7 mm in cross-section.

The pellets and micropellets can be conveyed, transported, and stored without blocking together and without being obstructive to the conveyance and transport equipment.

Further, a composition can contain one size pellet or micropellet of one chemical composition for each layer formed.

Other embodiments of the present invention are directed to methods of minimal shear molding. One method of minimal shear molding can include providing pellets, micropellets, powders, or a combination comprising at least one of the foregoing. The method can also include simultaneously forming at least one layer of a molded part with the pellets, micropellets, or powder. The composition can include at least one chemical

composition that can simultaneously form the at least one layer such that, for example, the following four parameters are met. Firstly, the composition for a single layer contains less than 20% of the minor component, and the size of the powder, if used, does not have to be smaller than the size of the pellet. Secondly, the pellets, micropellets, and/or powders used to form each layer in multiple layers are compositionally different such that each layer is compositionally different. Thirdly, combinations of one size pellet and one size micropellet are not used for a single layer. Lastly, a single layer is not formed from powder alone.

The method can be one wherein molded parts can be made that are not discolored and in which the inner surface is smooth without flow irregularities, such that the part has minimal to no surface imperfections including pinholes, bubbles, sinks, and pockmarks.

The method can utilize molding temperatures at least 25°C below molding temperatures of 35 mesh powder of comparable composition. In some cases, the method can utilize molding temperatures at least 50°C below, at least 75°C below, and, in exemplary embodiments, at least 100°C below molding temperatures of 35 mesh powder of comparable composition.

The method can provide molding ratios reduced from a range of 10:1 to 4:1 typical for 35 mesh powders of comparable composition to a range from 5:1 to 3:1.

The method can provide molding rotation rates reduced from a range of 6 rpm to 20 rpm typical for 35 mesh powders of comparable composition to a range of 1.75 rpm to 6 rpm.

The method can provide molding process times that are reduced at least 10% over those for 35 mesh powders of comparable composition; and, in some cases, the time is reduced at least 15%. The molding process times can be reduced by a combination of lower process temperatures, shorter cooling times, and more efficient transfer of heat to approximately uniform pellet surface area than to the irregular surface area typical of 35 mesh ground powders.

The method can be one in which minimal shear molding occurs rotationally in at least one axis (uniaxially), in at least two axes (biaxially), or in more than two axes (multiaxially).

The minimal shear molding can occur rotationally and be continuous and circular in at least one axis. Alternatively, the minimal shear molding can occur rotationally and be arcuate and oscillatory in at least one axis. Further, the minimal shear molding can occur rotationally and be continuous and circular in combination with arcuate and oscillatory in at least two axes. Additionally, the minimal shear molding can occur rotationally and continuously in at least two axes.

The rotation rates in different axes need not be equivalent.

The minimal shear molding can include rotation in an enclosed oven, rotation over open flame, and/or rotation in a jacketed mold. Different zones can be differently heated and heating can be achieved using at least one method including infrared radiation, electricity, gas, oil, and/or steam.

The partial layers can be formed using arcuate and oscillatory motion.

The layers can be formed by sequential addition of components.

The interface between layers can be smooth, distinct, uniform, and contain few to no bubbles.

The method can provide the sequential addition of components done manually by controlled release. The sequential addition of components can be done thermally by controlled release.

The method can limit the separation of layers on cooling and storage. Further, the process can include the addition of an adhesive layer molded between layers to prevent the separation of layers on cooling and storage.

In some cases, all components for all layers can be simultaneously charged into the mold to form layers without sequential addition of components.

The separation into layers can involve conditions including at least one of differential melt viscosity, melt density, pellet size, pellet density, softening temperature, melting temperature, polarity, component immiscibility, surface dynamics, and surface wetting, and combinations thereof.

Successful separation of layering can be achieved by variance of at least one parameter including rotation rate, rotation ratio, composition, molding process temperature, molding process time, and combinations thereof.

Adhesion of two layers can be achieved by admixture at the interface between those two layers.

The method can utilize a mold of many suitable materials, including metal and ceramic, which is compatible with the composition being molded.

The mold can be vented or unvented, and the addition of material to the mold can be achieved through the vent, an orifice, or combinations thereof.

The higher bulk density of pellets and micropellets can allow single charges of the material into a mold which commonly involve multiple charges for 35 mesh powders of comparable composition.

The reactive materials of the present process can be satisfactorily molded to effect the reaction.

The method can provide the cross-linking of the molded part.

Further, expansion of a foaming agent can be achieved.

The method can utilize a range of compositions, the choice of which can include pellet geometries, compositions, and dimensions. The choice of composition can include pellet geometries, compositions, and dimensions purely for decorative purposes. Further, the choice of materials can include materials that do not melt to provide decorative inclusions. The choice of materials and molding conditions can allow variation of surface patterning and color for decorative effects.

The choice of materials and molding conditions can include use of oversize pellets, low melt index pellets, and fractional melt pellets used singly and in combination that confer decorative and functional three-dimensional molded parts.

Other embodiments of the present invention are directed to an apparatus. The apparatus can include a highly regulated mixing stage, capable of both heating and cooling, with optional pressurization and filtration, to achieve extrusion through a multi-orifice die of approximately uniform diameter holes such that pelletization, transport, and drying of the pellets does not degrade, prematurely react, expand, prematurely cross-link, and otherwise alter the chemical composition of the polymers and copolymers for use in minimal shear molding.

The die of the apparatus, through which the chemical composition is extruded, can include a multiplicity of approximately uniform diameter holes concentrically positioned circumferentially and singularly about the face of the die.

The die of the apparatus, through which the chemical composition is extruded, can include a multiplicity of approximately uniform diameter holes concentrically positioned circumferentially in a multiplicity of groups of pods about the face of the die.

It is therefore an object of the present invention to provide a method to produce pellets and micropellets that are readily conveyable, economically transported and stored, and having a composition that includes polymers or resins, stabilizers, pigments, reactive or expandable components or component packages, cross-linking modifiers, rheology modifiers, processing agents, plasticizers, surface modifiers, and additives alone or in combination. The composition is such that the pellets and micropellets produced are of reproducible size, geometry, and composition and are readily moldable using minimal shear processes in one or more layers utilizing reduced molding temperature, reduced molding times, reduced axial or multiaxial rotation rates and ratios alone or in combination such that the molded article is structurally sound meeting the requisite criteria including acceptable appearance, uniform surface quality, impact strength, flexural qualities, gel formation or the lack thereof, barrier properties, and wall uniformity alone or in any combination.

BRIEF DESCRIPTION OF THE DRAWINGS

Figure 1 is a schematic illustration of an exemplary apparatus, including a feeding section, a mixing section, pelletization, dewatering and drying, and a post-processing section.

Figure 1a is a schematic illustration of a mixing vessel, medium pressure pump, and coarse screen changer.

Figure 1b is a schematic illustration of a feeder, gear pump, and static mixer assembly.

Figure 2 is a schematic illustration of a comparative static mixer with gear pump and bypass pipe connected by three-way valves.

Figure 3 is a schematic illustration of a vertically configured static mixer with attached bypass diverter valve.

Figure 4 is a schematic illustration of flow, purge, and drain positions for the bypass diverter valve in Figure 3.

Figure 4a illustrates the operational flow positions of valving in the bypass diverter valve with the static mixer vertically inclined.

Figure 4b illustrates a purge or bypass operation in the bypass diverter valve that bypasses the static mixer.

Figure 4c illustrates a purge or drain position in the bypass diverter valve that allows both sides of the vertically inclined static mixer to purge or drain without introducing material downstream.

Figure 4d illustrates a drain position in the bypass diverter valve that allows drainage of the upstream portion of the static mixer to drain out of the system with the downstream portion of the static mixer draining through the attached polymer diverter valve.

Figure 4e illustrates a purge position that blocks the static mixer and all downstream equipment allowing independent purge of the upstream equipment.

Figure 5 is a schematic illustration of flow and purge positions for the bypass diverter valve and to that is attached the static mixer in pendulous orientation.

Figure 5a illustrates the operational flow positions of valving in the bypass diverter valve with the static mixer pendulously attached.

Figure 5b illustrates a purge operation in the bypass diverter valve that bypasses the static mixer.

Figure 6 is a schematic illustration of flow and purge positions for the bypass diverter valve and to that is horizontally attached the static mixer.

Figure 6a illustrates the operational flow positions of valving in the bypass diverter valve with the static mixer horizontally attached.

Figure 6b illustrates a purge or bypass operation in the bypass diverter valve that bypasses the static mixer.

Figure 6c illustrates a drain position in the bypass diverter valve that allows both sides of the horizontally positioned static mixer to drain without introducing material downstream.

Figure 7 is a schematic illustration of a polymer diverter valve.

Figure 8 is a schematic illustration of a one-piece die plate with heating elements in three configurations.

Figure 9a illustrates the three configurations of the heating element extracted from the die plate.

Figure 9b illustrates the three configurations of the heating element positionally placed individually in side view.

Figure 10 is a schematic illustration of a removable-center die.

Figure 11 is an expanded view illustration of the components of a removable center-heated die.

Figure 12 is a schematic illustration of a die body with transport fluid box or waterbox.

Figure 13 is a schematic illustration of a die body and two-piece transport fluid box or waterbox.

Figure 14 is an expanded view illustration of a comparative two-piece waterbox or transport fluid box.

Figure 15a is a schematic illustration of a complete assembly of a comparative two-piece waterbox or transport fluid box.

Figure 15b is a cross-sectional illustration of an alternative waterbox or transport fluid box inlet and outlet design.

Figure 15c is a schematic face-view illustration of the alternative waterbox or transport fluid box inlet and outlet design of Figure 15b.

Figure 16 is a schematic illustration of a pelletizer with attached waterbox or transport fluid box showing the die.

Figure 17 is a schematic illustration of a die attached to a waterbox or transport fluid box containing a flow guide.

Figure 18a is a schematic illustration of a comparative flow guide.

Figure 18b is a schematic illustration of a second configuration of a comparative flow guide.

Figure 19 is a schematic illustration of a comparative flexible cutter hub with exploded view of flexible hub component.

Figure 20a is a schematic view of a portion of a streamline cutter hub.

Figure 20b is a schematic view of the streamline cutter hub rotated in perspective relative to Figure 20a.

Figure 20c is a cross-sectional view of the streamline cutter hub in Figure 20a.

Figure 21 is a schematic illustration of a steep angle cutter hub.

Figure 22a is a schematic illustration of a comparative cutter hub with attached normal angle blade.

Figure 22b is a schematic illustration of a steep angle cutter hub with attached blade.

Figure 22c is a schematic illustration of a comparative perpendicular angle cutter hub with attached non-tapered or square-cut blunted tip blade.

Figure 22d is a schematic illustration of a cutter hub with attached reduced thickness blade at normal angle.

Figure 23 is a schematic illustration of a comparative waterbox bypass.

Figure 24 is a schematic illustration of a comparative self-cleaning dryer.

Figure 25 is a schematic illustration of the dewatering portion of the self-cleaning dryer in Figure 24.

Figure 26 is a schematic illustration of a second comparative dryer with attached dewatering section.

Figure 27 is a schematic illustration of a reservoir.

Figure 28 is a schematic illustration of a dryer showing dewatering screen and centrifugal drying screen positioning.

Figure 29 illustrates a dryer screen with deflector bars.

Figure 30 is a cross-sectional illustration of the dryer screen with deflector bars in Figure 29.

Figure 31 illustrates a dryer screen of a configuration not requiring deflector bars.

Figure 32 is a cross-sectional illustration of the dryer screen of Figure 31 without deflector bars.

Figure 33 illustrates an enlarged edge-on view of a three-layer screen.

Figure 34 illustrates an enlarged edge-on view of a two-layer screen.

Figure 35 illustrates an enlarged external view of a multi-layer screen following Figure 34.

Figure 36a is a schematic drawing of an exemplary cylindrical tank with vent port.

Figure 36b is a schematic drawing of a top view of the cylindrical tank in Figure 36a illustrating an acentric vent position.

Figure 37a is an illustration of a second exemplary cylindrical tank illustrating concentric vent position.

Figure 37b is a cross-sectional view of the cylindrical tank in Figure 37a illustrating through-penetration of vent pipe into internal space of tank.

Figure 38a is a top-view illustration of a molded part showing through-penetrating orifice.

Figure 38b is a cross-sectional illustration of the molded part in Figure 38a.

Figure 39a is a schematic illustration of molded threads on a tank.

Figure 39b is a top view of the threaded tank in Figure 39a illustrating molding imperfections characteristic of powder.

Figure 39c is a top view of the threaded tank in Figure 39a illustrating reduced molding imperfections characteristic of micropellets.

Figure 40a is a cross-sectional view of a marginal contact kiss-off region of a molded tank.

Figure 40b is a cross-sectional view of a marginal contact kiss-off region of a molded tank with bridging.

Figure 40c is a cross-sectional view of an intimate contact kiss-off region of a molded tank.

Figure 41a is a cross-sectional view of an angular deformity typical of powder in molds where the long side is significantly greater in length than is the width.

Figure 41b is a cross-sectional view illustrating minimal angular deformity typical of micropellets in molds where the long side is significantly greater in length than is the width.

Figure 42a is a schematic illustration of a segment of a two-layer molded part wall with intimate and uniform contact of layers at the interface of the two layers.

Figure 42b is a schematic illustration of a segment of a two-layer molded part wall with entrainment of microbubbles at the contact interface of the two layers.

Figure 42c is a schematic illustration of a segment of a two-layer molded part wall with entrainment of microbubbles in combination with macrobubbles at the contact interface of the two layers.

Figure 42d is a schematic illustration of a segment of a two-layer molded part wall with an irregular contact interface between the two layers.

Figure 42e is a schematic illustration of a segment of a two-layer molded part wall with entrainment of macrobubbles randomly dispersed near the interface of the two layers and in an inner layer.

Figure 42f is a schematic illustration of a segment of a three-layer molded part wall with intimate and uniform contact of the layers at the interfaces of each layer.

Figure 43 is a schematic illustration of a melt cooler mounted horizontally with respect to the diverter valve in that the melt cooler inlet line enters a bottom portion of the cooler.

Figure 44 is a schematic illustration of the melt cooler mounted horizontally with respect to the diverter valve in that the melt cooler inlet line and the melt cooler outlet line are oriented in a side-by-side configuration.

Figure 45 is a schematic illustration of the melt cooler shown in Figure 43 with a top-mounted vent.

Figure 46 is a schematic illustration of the melt cooler with a bottom-mounted vent and drain.

Figure 47 is schematic illustration of the melt cooler shown in Figure 43 with a top head heated/cooled by a thermal transfer fluid.

Figure 48 is schematic illustration of a portion of the melt cooler shown in Figure 43 with a top head temperature controlled electrically.

Figure 49 is a perspective view of the diverter valve in a cooling mode of operation.

Figure 50 is perspective view of the diverter valve shown in a bypass mode of operation.

Figure 51 is perspective view of the diverter valve shown in a drain mode of operation.

Figures 52a, 52b, and 52c are schematic illustrations of melt coolers and diverter valves in various configurations.

DETAILED DESCRIPTION OF THE INVENTION

Although exemplary embodiments of the invention are explained in detail, it is to be understood that other embodiments are possible. Accordingly, it is not intended that the invention is to be limited in its scope to the details of construction and arrangement of components set forth in the following description or illustrated in the drawings. The invention is capable of other embodiments and of being practiced or carried out in various ways. Also, in describing the embodiments, specific terminology will be resorted to for the sake of clarity.

Minimal shear molding is known and, as used herein, includes rotation of the mold about a single axis (as exemplified in lining pipes); biaxial, which can include two perpendicular arms rotating independently through 360° or one arm rotating through 360° while the second arm swings back and forth in a predefined arc such that the motion can be described as oscillatory or rock-and-roll, or can be oscillatory rotation by both arms independent of each other; or can be multiaxial in that each independent rotation can be oscillatory or can circumscribe 360°. Biaxial rotation is a common process for the manufacture of storage tanks or tanker liners. Canoes and boats serve similarly as examples of common applications of the rock-and-roll methods.

Conventionally, powders have proven the workhorse of the rotational molding industry where the prior art suggests the use of sizes ranging from minus 10 mesh to 200 mesh, preferably 20 mesh to 50 mesh (approximately 0.033 inches to 0.012 inches or 0.84 mm to 0.30 mm respectively), and more preferably on 35 mesh or 0.020 inches (approximately 0.5 mm). Depending on the material, powder traditionally has been ground or cryogenically ground either of which adds considerable expense to the overall processing cost.

Grinding is well documented as introducing considerable work energy often manifested as heat into the material that potentially can lead to degradation, decomposition, and/or discoloration of the powder produced. The particle size distribution of the powders thus generated is typically of a broad range from very small to quite large; and agglomeration or adherence of these particles is not uncommon, leading

to loss of material or recycling for additional grinding steps. This wide size distribution can further require sizing processes to remove material larger and/or smaller than the requisite processing range. The grinding process is quite variable itself, over time, as blades or other grinding apparatus wear down or abrade with usage. Throughput volume is restricted to the capabilities of the grinding apparatus potentially requiring multiple units with escalation of maintenance costs and shut down of operations. The grinding process potentially generates tremendous amounts of fine particulates, or fines, which should be controlled to prevent unsafe levels of dust, leading to inhalation and slip hazards, fire or explosion hazards, and other liability and safety concerns for both the workers and the facility. The various types of grinding equipment utilized are well understood by those skilled in the art.

The conventional powder produced can have a variety of shapes (e.g., spherical, oval, egg-shaped, square, rectangular, platelike, flakes, needle-like, or acicular, or can be highly irregular with the possibility of easily fracturable points and protrusions). As a consequence of the powder geometry, and because of the rotation of the mold, powder flow into the mold can vary from a continuous mode to a slip mode where the powder remains in place until such elevation of the mold or other outside force causes it to slide along the pathway either smoothly, periodically, or to a cascade or avalanche flow mode in that a large portion of the powder gives way simultaneously much like an avalanche of snow. This is likened to pouring from a bin of sugar as opposed to pouring from a bin of extremely fine flour, conceptually. As the mold warms and the powder begins to soften, tacking occurs in the material allowing at least a portion of the powder to stick to the mold surface further altering or obstructing these flow mechanisms, potentially resulting in surface irregularities in the molded part. Ultimately, the heat applied to the mold melts the powder that coats the surface of the mold that continues to rotate and is then resolidified to produce the molded article.

Pellets, or more appropriately, strand pellets, can be prepared by extrusion of a strand of molten material that has typically been cut after cooling to generate cylindrical particles. These strand pellets often are prone to irregular fractures during or following the cutting process, and can be subjected to abrasive wear during transport. Rotational molding grades of material commonly were, and are still, marketed as powders (as

detailed above) or as pellets of this nature. These pellets can be of any length and diameter depending on the manufacturing process. Sizes of 1/8 inch (approximately 3.2 mm) in length and width are not uncommon. Pellets as described above similarly suffer, as do the powders, from irregular particle size and distribution, poor particulate flow, uneven melting, and poor reproducibility of the molded article, and are thus seldom used directly in rotational molding.

Powders and/or pellets (as previously described) for rotational molding are commonly available and are not limited to linear low density polyethylene, low density polyethylene, medium density polyethylene, high density polyethylene, cross-linkable polyethylene, polypropylene, polyester, polyamide, polycarbonate, acrylonitrile-butadiene-styrene (ABS), polystyrene, ethylene-vinyl acetate (EVA), ethylene-chlorotrifluoroethylene copolymer (ECTFE), polyvinyl chloride, and polyvinylidene difluoride (PVDF).

Pellets can also be generated in reactors. Polymerization of the pellets is commonly done as a dispersion that generates small globules of molten material in the dispersive media, which solidify on cooling to form somewhat spherical pellets of a particular size distribution that can subsequently be sized to appropriate ranges for the process and application being considered.

Pellets prepared by the above-described, or similar processes, are encompassed by the present invention by way of reference, and are preferably prepared by extrusion of a polymer melt formulation through an underwater pelletizer with subsequent drying as shown in Figure 1. The apparatus shown in Figure 1 includes a feeding or filling section 1 that provides material into a mixing, melting and/or blending section(s) 2 fittingly attached to a pelletizing section 3 that preferentially utilizes otherwise expedited fluid transport of the pellets to a dewatering and drying device 4. Afterwards, the material is conveyed to packaging, storage and/or post-processing manipulations 5.

In feeding section 1, material or component materials are fed into the mixing section 2 manually as a solid or liquid. Preferably, liquids can be pumped or metered (not shown) into the mixing apparatus 2 and solids can be added via a feed screw 10 as indicated in Figures 1, 1a, and/or 1b, or by another appropriate device. Feeding can be accomplished gravimetrically or volumetrically, and preferably is controlled through

mechanical and/or electronic feed-back mechanisms as are readily known to those skilled in the art. One or more similar or different feeding mechanisms can be required by the process and can be placed at the same or different entry points in the mixing section 2 as indicated by mixing inlet 14a, 14b, 14c, or 14d. The feeding components can be ambient in temperature, heated, or cooled and can be at atmospheric conditions or pressurized, purged with air or an inert medium (e.g., argon or nitrogen), or can be subjected to a vacuum or partial vacuum to expedite flow into the mixing section 2 near the exit port of the feeding device. An exemplary exit port is a feed screw outlet 12.

The mixing section 2 can include dynamic 2a, extrusional 2b, and/or static 2c mixing components. These components can be used individually or in combination, and can be interconnected or attached in series, in tandem, and/or in parallel.

The feed screw outlet 12 of feeding section 1, shown as Figure 1a, is attached to the dynamic mixing section 2a at one or more inlets (e.g., inlet 14a for the thermally controlled mixing vessel 16). The vessel chamber can be atmospheric or purged with air or inert gas (e.g., argon or preferably nitrogen). Components can be added continuously or in portions, with warming to temperature as desired. Mixing is achieved by rotation of the rotor 18 controlled by motor 20. Attached to rotor 18 are mixing blades 22, which can be propeller style, boat style, ploughshare style, delta style, sigma style (in single, double, or multiple configurations) or helical or helical dispersion blades. Alternatively, the mixer can be a kneader, Buss kneader, or Farrell internal mixer, or it can be a ribbon blender, Banbury-type blender, horizontal mixer, vertical mixer, planetary mixer or equivalent device known to those skilled in the art.

On reaching the appropriate pour point, valve 24 is opened and the fluid or molten material passes into and through pipe 26 and is drawn into booster pump 30. The booster pump 30 can be, for example, a centrifugal pump or a positive displacement reciprocating or rotary pump. Preferably, the booster pump 30 is rotary and can be a peristaltic, vane, screw, lobe, progressive cavity, or a gear pump. The gear pump can be high precision or open clearance, and generates an intermediate pressure (e.g., up to 500 psi, but preferably less than 150 psi). The pump pressure is sufficient to force the melt through coarse filter 35, which can be a candle filter, basket filter, or screen changer. In exemplary embodiments, the coarse filter 35 is a basket filter of 20 mesh or coarser. The coarse

filter 35 removes larger particles, agglomerates, or granular material from the melt as it flows to and through pipe 32. The dotted line 40a indicates the connection to melt pump 80.

Alternatively, the feeding section 1 in Figure 1 is connectedly attached via feed screw outlet 12 to the mixing section 2, and more specifically extrusional mixing section 2b, at one or more inlets (e.g., inlet 14b) to an extruder 50 that optionally can be but is not limited to a single screw, twin screw, multiple screw or ring extruder, or a ram extruder. The sections or zones of the screw should feed, mix, and convey the material simultaneously, providing sufficient thermal and mechanical energy to melt, mix, and uniformly disperse and distribute the material(s) for the pelletization to follow. The extruder 50, preferably a twin screw extruder, can be purged with air or an inert gas, and can also have one or more vent ports. Some or all of the vent ports can be fitted with one or more vacuum attachments or other exhaust mechanism(s) as is understood by those skilled in the art. Vent ports or appropriate exhaust mechanisms facilitate removal of gases, unwanted volatiles such as residual monomer, byproducts, and/or impurities. Venting should be used with caution and positionally placed such that any volatile components essential to the formulation are not lost or compromised after introduction to the mixing process. The configuration of the screw should be satisfactory to achieve an appropriate level of feeding, mixing dispersively and/or distributively, melting, blending, and throughput rate, which is determined by the formulation and processing requirements. The extruder 50 is coupled to the melt pump 80 as shown in Figure 1 at the location similarly identified by the dotted line 40a for dynamic mixing section 2a illustrated in Figure 1a.

Analogously, feeding section 1 can be connected via feed screw outlet 12 to inlet 14c in the static mixing section 2c, and/or to inlet 14d in the static mixing section 2d. Process operations can include use of a booster pump 30 and/or a melt pump 80 to facilitate transfer and pressurization of the material flow into the static mixer 60. Static mixer 60 is connected to melt pump 80 positionally as indicated by dotted line 40b.

Mixing sections can be used alone or in combination where dynamic, extrusional, and/or static mixing are connected in series and/or in parallel. Examples of this can be seen as dynamic mixing section 2a attached directly to static mixing section 2d at inlet

14d, extrusional mixing section 2b attached directly to static mixing section 2d at inlet 14d, and extrusional mixing section 2b attached directly to static mixing section 2c at inlet 14c, bypassing static mixer 100. Extrusional mixing section 2b alternatively can be attached to another extrusional mixing section in series and/or in parallel having a similar or different design type or configuration. Temperatures and process parameters can be the same or different in the various mixing sections, and mixing units can be attached in combinations, serially or otherwise.

Solid or liquid ingredients can be added utilizing the feeding section(s) 1 connected at one or more locations including, but not limited to, inlets 14a, 14b, 14c, or 14d. For dynamic mixing, components are added at inlet 14a or at inlet position 75 proximal to inlet 14c for situations where volatiles are involved. Where dynamic mixing is attached serially to static mixing (not shown in Figure 1), addition of the volatiles is preferably performed at the inlet of the static mixer, an example of which includes modification to inlet 14d for static mixer 60 (Figure 1b). For extrusional mixing, components are added at inlet 14b, or at an inlet positionally near the end of the extruder 50 for situations where volatiles are involved, as shown by inlet position 70 or alternatively at inlet position 75 proximal to inlet 14c. For extrusion mixing serially attached to static mixing prior to gear pump 80 (not shown in Figure 1), components can be added at the inlet of the static mixer as exemplified by a modification of inlet 14d for static mixer 60 (Figure 1b). For static mixing, components can be introduced at inlet 14d in Figure 1b, or at inlet position 75 proximal to inlet 14c in Figure 1 for situations involving volatiles.

Various levels of mixing and shear are achieved by the differing styles of mixing processes. Static mixing typically has the least shear and relies more on thermal energy. Dynamic mixing depends to a large degree on blade design and mixer design. Extrusional mixing varies with type of screw, number of screws, and the screw profile and is quite capable of significant generation of shear energy. Therefore, energy is introduced into the mixing process in terms of both shear or mechanical energy and thermal energy. Heating, and/or cooling, of the units can be achieved electrically, by steam, or by circulation of thermally controlled liquids such as oil or water. Mixing continues until a formulation reaches an appropriate temperature or other criterion of

consistency or viscosity as determined specifically for the process by those appropriately skilled in the art.

On exit from the mixing stage 2a, 2b, 2c, 2d, or any combination thereof, the molten or fluidized material optionally passes to and through a melt pump 80 that generates additional pressure on the melt, preferably at least approximately 10 bar and more preferably approximately 30 to approximately 250 bar or more. The exact pressure will be dependent on the material being processed and can be significantly affected by the pelletization process 3 that follows mixing and on the throughput rate or flow rate of the process. Melt pump 80 can be a centrifugal or positive displacement reciprocating or rotary pump. In exemplary embodiments, the melt pump is a rotary pump, which can be a peristaltic, vane, screw, lobe, progressive cavity, or gear pump, with the gear pump being preferred. Seals should be compatible with the material being processed, chemically and mechanically, the details of which are well understood by those skilled in the art.

The pressurized melt passes through a filter 90 that can be a basket filter or screen changer. Preferably, a screen changer of approximately 200 mesh or coarser is used. An exemplary screen changer is a multilayer screen changer having two or more screens of differing mesh (e.g., 20 mesh, 40 mesh, and 80 mesh screens). The screen changer can be manual, plate, slide plate, rotary plate, single or dual bolt, and can be continuous or discontinuous.

The use of melt pump 80 and/or filter 90 is strongly and optionally dependent on the containment of any volatile ingredients in the formulation. Pressures can be sufficient from extrusional mixing 2b to forego use of melt pump 80 whereas use of static and/or dynamic mixing, 2a or 2d, can require facilitation of pressurization to insure progress through, and egress of, the formulation from the apparatus. The filter 90 provides a safety mechanism, where employed, to insure oversize particles, lumps, amorphous masses, or agglomerates are not propagated to the bypass static mixer 100 or pelletization process 3. Alternatively, introduction of any volatile components can be performed at inlet position 75 proximal to inlet 14c in Figure 1 as previously delineated. Where additional pressurization and/or screening are a requisite process component, introduction via inlet position 75 proximal to inlet 14c is the preferred approach.

Static mixer 60, shown in Figure 1b, can be used to heat the mixture being formed to generate a uniform molten mass or can be used effectively as a melt cooler to reduce the temperature of the molten mass. When static mixers are used in series, each unit can be used to heat and further mix the formulation where the temperatures, design, geometries and configurations, physical size, and process conditions can be the same or different. A static mixer in the series can be heating the mixture to achieve better dispersive and distributive mixing, whereas a second static mixer can actually be cooling the mixture to facilitate further processing. The static mixer 60 or melt cooler is a heat exchanger of the coil type, scrape wall, shell and tube design, U-style tube design, or other comparable style. In exemplary embodiments, it is a shell and tube design that includes static mixing blades of appropriate configuration within the individual tubes to further mix the material and bring more of the material in intimate contact with the wall of the tube, outside of which is a flow of a fluid, such as oil or water, to provide warming or cooling as appropriate. The temperature and flow rate of the circulating medium is carefully regulated by a control unit (not shown). The important criterion for selection of conditions in static mixing or melt cooling is to do a maximum amount of work to effect mixing with a minimum pressure drop while maintaining the pressure required for proper admixture. Pressures generated by the extruder 50 and/or the melt pump 80, where present, should be sufficient to maintain flow of the molten or fluid mass through the filter 90, where applicable, into and through the bypass static mixer 100, and into and through the pelletization section 3. Alternatively, an optional melt pump 80 can be positionally attached to outlet 130 and inlet 205 to maintain or increase pressure into and through the pelletization section 3.

The optional bypass static mixer 100 shown in Figure 1 has a distinct advantage over prior art where a static mixer 60 would have to be removed physically from the melt flow pathway for maintenance or cleaning, and is not always necessary in a particular process. To simplify this challenge, a “spool” or straight large bore pipe that can or can not have a coolant connection was inserted into the pathway to allow flow effectively bypassing the unnecessary static mixer. Alternatively a bypass line 102 can be inserted into the flow path as shown in Figure 2 with a diverter valve 104 used to switch flow from the static mixer 60 into the bypass line 102. Similarly a second diverter valve 106

was required to reconnect the bypass flow back into the mainstream at or near the outlet of static mixer 60.

The outlet of optional filter 90 is connected to the bypass static mixer 100 in Figure 1 via inlet 110 of bypass diverter valve 120 as detailed in Figure 3. Inlet 110 directs melt flow into the static mixing component 150 of the bypass static mixer 100 through static mixer inlet 152. The melt flow passes through static mixing component 150, and exits through static mixer outlet 154 into the outlet 130 of the bypass diverter valve 120. A two-pass or double pass heat exchanger having the base 156 of the static mixing component 150 attached through inlet 152 and outlet 154 to the bypass diverter valve 120 is illustrated in Figure 3. The top 158 of the static mixing component 150 is distal from the bypass diverter valve 120.

Figure 4 illustrates five modes of operation of the bypass diverter valve 120 in conjunction with the static mixing component 150 as detailed in accordance with Figure 3. An "X" in a valve line of the bypass diverter valve 120 indicates that the valve line is closed. Figure 4a illustrates operation of the bypass diverter valve in conjunction with the static mixing component 150 in vertical orientation as detailed above. More specifically, bypass valve line 166 between valve components 162 and 164 is closed, as well as valve drain lines 168 and 170. As such, liquid, molten material, or extrudate entering bypass diverter valve 120 through valve entry line 165 is directed by valve component 162 to static mixing component 150 through valve static mixer entry line 172. Thermally modified material exiting the static mixing component 150 via static mixer exit line 174 is directed by valve component 164 out of bypass diverter valve 120 through valve outlet line 167 toward the pelletization section 3.

Figure 4b illustrates the bypass mode for bypass diverter valve 120. As shown, bypass valve line 166 is open, valve drain lines 168 and 170 remain closed, and valve static mixer entry line 172 (which is connected to inlet 152 of static mixing component 150) and static mixer exit line 174 are also closed. As such, liquid, molten material, or extrudate flows directly from valve entry line 165 through the bypass diverter valve 120, thus bypassing the static mixing component 150.

Figure 4c illustrates a first drain mode in that the bypass valve line 166 is closed, while valve drain lines 168 and 170 and valve static mixer entry line 172 and static mixer

exit line 174 are open. In this manner, the molten or liquid formulation in the static mixing component 150 can drain away. Similarly, valve entry line 165 and valve outlet line 167 are open so that liquid, molten formulation, or extrudate upstream and/or downstream, respectively, from the bypass diverter valve 120 can also drain out through valve drain lines 168 and 170, respectively.

In an alternate drain mode shown in Figure 4d, the bypass valve line 166 is closed. Liquid or molten material in the static mixing component 150 proximal to valve drain line 168, as well as material upstream of the bypass diverter valve 120, drain through valve entry line 165 and exit via valve drain line 168. Material, molten or liquid, in the static mixing component 150, proximal to the static mixer exit line 174, drain through that line past valve component 164, out valve outlet line 167, and then pass through diverter valve 200 (from Figure 1).

Figure 4e illustrates a purge position that redirects flow from valve entry line 165 directly to valve outlet line 168, effectively removing the static mixing component and all downstream processing.

Figure 5 illustrates an alternate arrangement of the static mixing component 150 and the bypass diverter valve 120. In this embodiment, the static mixing component 150 is vertically suspended below the bypass diverter valve 120. The inlet 152 to, and exit 154 from, the static mixing component 150 are both mounted at the top of the static mixing component 150 and the base of the bypass diverter valve 120. In Figure 5a, the liquid, molten, or extruded material enters valve component 180 through valve inlet line 165. With bypass line 166 closed and valve static mixer inlet line 172 open, valve component 180 directs the flow into the static mixing component 150. During steady state process conditions, material exiting the static mixing component 150 at outlet 154 enters the bypass diverter valve 120 through static mixer outlet line 174, and flow is directed by valve component 182 out through valve outlet line 167.

In the bypass mode, illustrated in Figure 5b, the valve static mixer inlet line 172 and static mixer outlet line 174 are both closed, while the bypass line 166 is opened, thus allowing the material to bypass the static mixing component 150 directly to valve outlet line 167.

Figure 6 illustrates a third orientation of the static mixing component 150 with respect to the bypass diverter valve 120. More specifically, static mixing component 150 is positioned horizontally relative to bypass diverter valve 120. As shown, both the inlet 152 and outlet 154 are positioned at the end of the static mixing component 150 adjacent to the bypass diverter valve 120. In the normal operating mode, the melt, liquid, or extrudate is directed by the bypass diverter valve 120, as illustrated in Figure 6a. The bypass mode follows, as Figure 6b illustrates, and the drain mode is shown in Figure 6c. In each operation, bypass diverting valve 120 operates in the same manner as described previously for valve components 162 and 164 and, therefore, is not described here. The orientation of the static mixer 100 and bypass diverter valve 120 can be pendulous, horizontal, or vertically disposed or can be positionally inclined at many angles between these positions.

The preferred design of the bypass diverter valve 120 is of a single-body housing capable of being heated by a jacket (e.g., using steam or other heat transfer fluid) or by electric heater cartridges. It has a plurality of inlet and outlet bores and connections as shown in Figures 3, 4, 5, and 6. The valve components 162 and 164, as well as valve components 180 and 182, are preferably in the form of movable bolts, with valve components 162 and 180 being upstream of the static mixing component 150 and valve components 164 and 182 being similarly downstream. The bolts can contain, but are not limited to, 2 bores. Examples of this include valve components 164, 180 and 182. The bolts can also contain 3 bores, of which valve component 162 is an example. The respective bores can be straight-through, form a 90° turn or in the shape of a “tee” or “T”, and are specifically placed along the length of the bolt. Each of these bores is positionally placed by means of a fluid-controlled cylinder or equivalent device, and will adjustably maintain good alignment with the proper inlets and/or outlets of the bypass diverter valve 120, based on the desired position required by the operator running the process, as will be understood by those skilled in the art. The positioning of the fluid powered cylinders, and thus the bolts’ position, can be controlled by manually operating a fluid flow valve, by automatic control (e.g., by PLC), or both.

Figure 43 illustrates another embodiment specifically of the melt cooler in that the orientation of the melt cooler with respect to the diverter valve is the same as is shown in

Figure 6 and identified as static mixing component 150. More specifically, melt cooler 2090 is shown positioned horizontally with respect to the diverter valve generally designated by reference numeral 2092. As shown, both the inlet 2094 and outlet 2096 are positioned at the end of the melt cooler 2090 adjacent the diverter valve 2092. In this embodiment, the inlet 2094 is positioned in the bottom portion 2093 of melt cooler 2090 and the outlet 2096 is positioned in the top portion 2091 of melt cooler 2090. The normal operating mode where the hot melt polymer is directed by the diverter valve 2092 through the melt cooler 2090 is shown in the left-hand illustration of Figure 43 marked "A". The bypass mode is shown in the center illustration of Figure 43, marked "B", and the drain mode is shown in the right-hand illustration, marked "C". In each mode of operation, the diverter valve 2092 operates in the same manner as described above.

Figure 44 illustrates another embodiment in which the orientation of the melt cooler with respect to the diverter valve is the same as is shown in Figure 6. More specifically, melt cooler 2090 is shown positioned horizontally with respect to the diverter valve 2092. Both the inlet 2094 and outlet 2096 are positioned at the end of the melt cooler 2090 adjacent the diverter valve 2092. In this embodiment, the inlet 2094 and the outlet 2096 are located in opposing portions 2097 and 2098 of the melt cooler in a side-by-side configuration. The normal operating mode where the hot melt polymer is directed by the diverter valve 2092 through the melt cooler 2090 is shown in the left-hand illustration of Figure 44, marked "A". The bypass mode is shown in the center illustration of Figure 44, marked "B", and the drain mode is shown in the right-hand illustration, marked "C". In each mode of operation, the diverter valve 2092 operates in the same manner as described above.

As shown in Figures 45 and 46, respectively, melt cooler 2030 and melt cooler 2060 can be configured to vent compressible fluids and to drain the polymeric melt and other fluids. Figure 46 illustrates a vent 2095 located on the top 2038 of melt cooler 2030. Figure 46 illustrates a vent and drain 2101 located on the bottom 2100 of melt cooler 2060.

To provide for the desired melt flow regimes in the top 2038 of melt cooler 2030, the top 2038 can be heated. For example, as illustrated in Figure 47, the top 2038 can be heated or cooled by a thermal transfer fluid that passes through flow channel 2039. In

another possible heating configuration as illustrated in Figure 48, the top 2038 can be heated electrically, such as for example, by an electric heater 2041. Controlling the temperature of the top 2038 ensures that the melt does not cool below a predetermined temperature as it turns through top 2038 from a first process side of the melt cooler to a second process side of the melt cooler.

As indicated above, Figures 49, 50, and 51 provide detailed views of the diverter valve 2040 in, respectively, the cooling mode, the bypass mode, and the drain mode of operation. The diverter valve 2040 has a body housing capable of being heated by jacket using steam or other thermal transfer fluid or by electric heater cartridges. In a preferred embodiment, the first movable valve component 2042 is a hydraulically actuatable bolt having three sets of flow channels therein, and the second movable valve component 2044 is a hydraulically actuatable bolt having two sets of flow channels therein. In other possible embodiments of the diverter valve 2040, the bolts can include two or three sets of flow channels, either as a straight-through flow channel, as a 90° turn flow channel, or as a tee-flow channel, specifically placed along the bolt length. Each of these flow channels is moved into a specific position by a fluid controlled cylinder, and aligns with the corresponding inlets and/or outlets of the diverter valve, based on the position desired by the operator running the process. The positioning of the fluid powered cylinders, and thus the bolt position, can be controlled by manually operating a fluid flow valve, by automatic control (e.g., by a PLC), or both.

In other situations, the melt cooler 2030 can be oriented perpendicular to the melt flow path through a diverter valve 2140. As illustrated in Figures 52A, 52B, and 52C, the diverter valve 2140 has a single movable valve component 2145. Movable valve component 2145 is a hydraulically actuatable bolt having three sets of flow channels therein, including a cooling flow channel 2141, a bypass flow channel 2145, and a drain flow channel 2143. The single bolt embodiment of the diverter valve provides a relatively short melt flow path and an economical valve construction.

Where employed, the bypass static mixer 100 provides a heretofore unachievable level of control of the volatile ingredients over prior art devices. Use of the bypass static mixer 100 as a replacement of the static mixer 60 is well within the scope of the invention. The gains to be realized are in the greatly facilitated purging capacity of the

bypass diverter valve. Orientation of valve component 162 allows upstream flow to be diverted prior to entry into the static mixing component 150 as illustrated in Figure 4e where inlet line 165 and drain line 168 are the only open pathways as well as the bypass variations as herein described. Should the additional modification of the process liquid, melt, or extrudate not be necessary for a particular application, the bypass pathway is of minimal distance where temperature and pressurization can be maintained. Purging processes or purging materials can be passed through the components with sufficient ease, without risk of unwanted loss of any volatiles, and/or with reduced likelihood of hazardous flammability issues, and without significant inhibition of process flow. Minimization of downtime is readily achievable through elimination of component removal for routine maintenance or repair.

The component(s) of the mixing section 2 can be connected to the diverter valve 200, as indicated in Figure 1, where the outlet 130 of the bypass static mixer 100 is attached to inlet 205. Figure 7 illustrates inlet 205 and outlet 206 attached to housing 202 of diverter valve 200. The movable diverter bolt (not illustrated) can be actuated electromechanically, hydraulically, pneumatically, or by any combination thereof.

Referring again to Figure 1, diverter valve 200 is attached at outlet 206 to the pelletization section 3 at inlet 301 of the die 320, the details of which are illustrated in Figures 8, 9, 10, and 11.

The die 320 in Figure 8 is a single-body style including a nose cone 322 attached to die body 324, into which are fitted heating elements 330 and through which are bored multiple die holes 340 that vary in number and orientation pattern. The die holes 340 can be approximately 3.5 mm in diameter or smaller, preferably approximately 1.5 mm or smaller, more preferably approximately 1.0 mm or smaller, and most preferably approximately 0.5 mm or smaller. The die holes 340 can have many design combinations including, but not limited to, increasing or decreasing taper, cylindrical, and combinations thereof. The segments can vary in length as necessitated by the process and materials. Preferably the die holes are placed singularly or collectively in groups or pods in one or more concentric rings as determined by the diameter of the outlet 206 of the diverter valve 200 to which it is attached.

Heating elements 330 can include a cartridge or a coil type element, and can be of sufficient length inside the die body 324 to remain outside the circumference of the die holes as illustrated in Figure 8 and detailed in Figures 9a and 9b as configuration 1. They also can extend into and near the center of the die body without passing the center in length (shown as configuration 2 in Figures 9a and 9b), or can extend past the center in length but not of sufficient length to contact the ring of die holes diametrically opposed (shown as configuration 3 in Figures 9a and 9b). Positioning of the die holes can vary to accommodate the appropriate configuration of the heating elements 330.

A die 320 having a removable center or insert configuration in the die body is illustrated in Figure 10. The heating elements 330 are of a cartridge or coil configuration and are inserted into the outer die body component 352, where they are constrained in length to suitably fit within the confines of the outer die body component 352. The die holes 340 are contained within removable insert 350 and are variable in design, dimension, and placement as detailed in the foregoing discussion. The removable insert 350 is fixedly attached to outer die body component 352 by ordinary mechanisms.

Figure 11 shows an alternative design of die 320 in that the die body is of a removable center or insert configuration with multiple heating zones for enhanced heating efficiency and more facile thermal transfer to the molten or liquid materials as they pass through the die holes 340. The outer die body component, not shown, is comparable to that described for Figure 10. The heated removable insert 360 of the alternative design has an open center that is fitted with a heating element 365, preferably a coiled heating element, that can be thermally controlled in common with other heating elements in the outer die body component or can be autonomously regulated thermally (thus allowing multizone heating capacity within the die 320).

The die 320 in all configurations (Figures 8, 9, 10, and 11) can contain an appropriate hardface 370 fixedly attached for a cutting surface as illustrated in Figure 11 that is preferably abrasion resistant, wear resistant, and (where desired) corrosion resistant. The die holes 340 for extrusion of the molten, liquid extrudate can pass therethrough. By way of example, tungsten carbide, titanium carbide, other ceramics, or mixtures thereof are common materials for hardface applications.

Surface treatments, surface finishing, polishing, or hard surfacing of die 320 can also be done to provide additional protection to the die body. Examples of such treatments include, without limitation, nickel phosphide, chrome plating, nitriding, or comparable physical or chemical treatment. Prior art has demonstrated the use of different die hole sizes in at least one group where several groups of die holes are present or in that a particular group of die holes is of at least two different sizes. This has not been found to be necessary or practical and often found to lead to freeze off or wrapping of the polymer around the face of the die. The instant invention does not reflect the need for simultaneous extrusion of different pellet sizes.

An exemplary bolting mechanism for the nose cone 322 is illustrated in Figure 11. A cover plate 372 is positionally attached by bolt 374 to the face of the die body 320, removable insert 350, or heated removable insert 360, as shown in Figures 8, 10, and 11 respectively. The cover plate 372 can be less than or at least equal to the height dimension of the hardface 370. Alternatively, gasket materials or other materials for sealing of the cover plate 372 can be used as desired.

Referring once again to Figure 1, the die 320 is attached to transport fluid box or waterbox 400 as detailed in Figures 12, 13, 14, and 15 (a, b, and c). Figure 12 illustrates a configuration of a one-piece transport fluid box or waterbox 400 that comprises a housing 402 connected to an inlet pipe 404 and outlet pipe 406 of similar diameter and geometry. The one-piece transport fluid box or waterbox 400 is diametrically opposed positionally and interconnectedly attached to a rectangular, square, cylindrical or other geometrically open cutting chamber 408 surrounding (with sufficient diameter to completely encompass) the die face 410 (representationally equivalent to the surface of hardface 370 in Figures 8, 10, and 11). Housing 402 has mounting flange 412 through which a plurality of mounting bolts 414 pass to sealingly attach the transport fluid box or waterbox 400 and die 320 to diverter valve 200. Flange 416 on housing 402 allows attachment to the pelletizer 900 (see Figure 1) as detailed below. Components that are free to rotate within the cutting chamber 408 are described later in this disclosure.

Similarly, Figure 13 illustrates a two-piece configuration of transport fluid box or waterbox 400 comprising a main body 450 with housing 452 that is connected to inlet pipe 454 and outlet pipe 456, which have a similar diameter and geometry and are

diametrically opposed positionally and interconnectedly attached to a rectangular, square, cylindrical, or other geometrically open cutting chamber 458 surrounding (with sufficient diameter to completely encompass) the die face 410 (representationally equivalent to the surface of hardface 370 in Figures 8, 10, and 11). Housing 452 has mounting flange 462 through which a plurality of mounting bolts or studs 464 pass. Mounting flange 462 sealingly attaches to adapter ring 470 of comparable diameter (both inside and outside dimensions). A plurality of countersink bolts 472 pass therethrough. Mounting bolts or studs 464 and countersink bolts 472 are preferably used in an alternating manner, and sealingly attach the components of the complete transport fluid box or waterbox 400 and die 320 to diverter valve 200. Flange 466 on housing 452 of the main body 450 allows attachment to the pelletizer 900 (see Figure 1) as detailed below. Components that are free to rotate within the cutting chamber 408 in Figure 12 and/or cutting chamber 458 in Figure 13 are described later in this disclosure. Separate attachment of the adapter ring 470 to, and through, the die 320 allows the main body 450 to be removed for cleaning or maintenance while leaving die body 320 sealingly attached to diverter valve 200.

An exploded view of the two-piece configuration of transport fluid box or waterbox 400 is illustrated in Figure 14 with a complete assembly illustrated in Figure 15. As with other figures throughout the disclosure, similar parts have similar numbers in Figures 13, 14, and 15a.

Figures 15b and 15c illustrate an alternative design for the transport fluid box or waterbox inlet and outlet, in that inlet 480 is fixedly attached to a rectangular or square inlet tube 482 that taperingly increases along its length as it approaches the housing 481, to which it is attachedly connected and within which is cutting chamber 484. Similarly, attached to housing 481 and diametrically opposed to inlet tube 482 is rectangular or square outlet tube 486 that taperingly decreases along its length to outlet 488 to which it is fixedly attached. Flange 483 and flange 485 in Figures 15b and 15c compare in design and purpose to flanges 462 and 466 in Figure 15a previously described.

Figures 15a, 15b, and 15c illustrate the preferred diametrically opposed inlets and outlets. Alternatively, the inlets 454 and 480 and outlets 456 and 488 can be located at an angle from approximately 20° to approximately 180° relative to, and defined by, the position of outlet to inlet. By way of example, the inlets 454 and 480 and outlets 456 and

488 can be opposingly or staggeringly attached to housing 481. Dimensions of the inlet and outlet can be the same or different and the inlet and outlet can be similar or different in design. Preferably the inlet and outlet so identified are of similar dimension and design, and are diametrically opposed.

Once again returning to Figure 1, pelletizer 900 is shown in the non-operational open position. Attached to the pelletizer is flow guide 800, and cutter hub 600 with cutter blades 700. Upon operation of the equipment, pelletizer 900 is moved into position such that it can be fixedly attached to flange 416 of the one-piece configuration of transport fluid box or waterbox 400 or flange 466 on the main body 450 of the two-piece configuration of transport fluid box or waterbox 400, as detailed in Figures 12 and 13, respectively. Attachment can be made via quick disconnects or other such mechanism. In the operating configuration, the cutter hub 600 and cutter blades 700 freely rotate within the cutting chamber 408 (Figure 12) or 458 (Figure 13). Details of all illustrated components are described in greater detail below.

The pelletizer 900 of the instant invention is shown schematically in Figure 16. The pelletizer 900 can be adjustable positionally in terms of cutter hub 600 relationally to die face 410. Figure 16 represents the pelletizer 900 in operational position, where it is sealingly attached via pelletizer flange 902 to transport fluid box or waterbox flange 466 tightly held by removable quick disconnect clamp 904. Positional adjustment of the pelletizer can be achieved manually, via spring-loading, hydraulically, pneumatically, electromechanically, or can be achieved by combinations of these mechanisms acting cumulatively in one direction or opposingly in counter-direction of forces applied to insure appropriateness of position as necessitated to achieve even wear, increased longevity, avoidance of undue extrusion leading to melt wrap around the cutter hub or the die face 410, and consistency of the pelletized product. A preferred design is of the hydraulic-pneumatic mechanism detailed in Figure 16 comprising a motor 905, housing 910, and containing hydraulic cylinder 920 engaged to coupling 922. A rotor shaft 930 connects coupling 922 to the cutter hub 600 at the die face 410, and passes through thrust bearing 940 and sealing mechanism, and preferably a mechanical sealing mechanism 950 in fluid contact with cutting chamber 458 of transport fluid box or waterbox 400. Inlet pipe 454 and outlet pipe 456 indicate flow direction of fluids (e.g., water) into the cutting

chamber 458, admixture of fluids and pellets in the cutting chamber 458, and subsequently, flow of the pellet slurry formed away from the cutter hub 600 as well as die face 410, and out of the cutting chamber 458.

Figure 17 illustrates a preferred configuration where flow guide 800 is positioned in the cutting chamber 458 effectively reducing the fluid volume of that region. This configuration can be used to increase fluid velocity through the cutting chamber 458, improve pellet quality, reduce freeze off, avoid wrapping of melt around die face 410, generate or increase head pressure, and improve pellet geometry. The die 320, transport fluid box or waterbox 400, and pelletizer 900, shown only partially, are positioned in the same fashion as shown in Figure 16. The hollow shaft rotor can be attached to cutter hub 600 in cutting chamber 458 with appropriate inlet pipe 454 and outlet pipe 456 as previously described. The pelletizer 900 is sealingly and removably attached by transport fluid box or waterbox 400 through use of quick disconnect clamp 904 on pelletizer flange 902 and transport fluid box or waterbox flange 466 as before.

Figures 18a and 18b show two possible different configurations for flow guide 800 in which sections can be of similar or different segmental length having consistent outside diameter that is less than the diameter of cutting chamber 458 and can be varied in accordance with the requisite diminution of volume desired in cutting chamber 458. Flow guide spacer sections 803 can be approximately uniform circumferentially and diametrically as indicated singly by 803a, or in a plural manner in 803b and 803c but can vary in segmental length. To direct and/or restrict flow, flow directing segments 801 singly in 801a or unlimited plurally in 801b, 801c, and 801d, for example, are modified by longitudinally extending grooves that are arcuate in transverse configuration with the deepest grooved section positioned proximal to the cutter hub 600. The preferred configuration of a series of segments is not intended to be limited as to number of segments and a single flow guide component of comparable geometry and functionality is well within the scope of the present invention.

Continuing with Figure 16, cutter hub 600 is attached by screwing onto the threaded end of the rotor shaft 930 of pelletizer 900. The cutter hub 600 can be rigidly mounted to the rotor shaft 930 and can contain any number of cutter arms 610 in balanced proportion placed circumferentially about the cutter hub 600 as illustrated in

Figure 19. Alternatively, the cutter hub 600 is flexibly attached to rotor shaft 930 using an adapter 620 in that the adapter 620 is attachedly and threadedly connected to rotor shaft 930. Adapter 620 has a partial spherical outer surface 622 matching a similar partial spherical inner surface bore 602 in the cutter hub 600. Diametrically opposed and recessed into the partial spherical inner surface bore 602 are longitudinal recesses 605 that extend to the edge of the cutter hub 600 and into which ball 640 fits. Similarly diametrical recesses 626 for ball 640 are located on adapter 620 positionally oriented such that longitudinal recess 605 and diametrical recess 626 align to interlock balls 640 once adapter is inserted orthogonally into position and rotated to a position parallel to cutter hub 600. This allows free oscillation of the cutter hub 600 about the diametrically positioned balls 640 on fixedly attached adapter 620 to rotor shaft 930 that permits rotational self-alignment of the cutter hub 600.

The cutter arms 610 and body of cutter hub 612 can be square or rectangular in cross-section as shown in Figure 19. The cutter arms 610 and body of cutter hub 612 can be more streamlined to give an extended hexagonal cross-section as illustrated in Figure 20c. Figures 20a and 20b shows segments of streamline cutter hub 650. Cutter blades (not shown) are fixedly attached by screw or similar mechanism at flattened angular groove 614 (Figure 19) or at flattened angular notch 652 (Figure 20a and 20b).

Alternatively, Figure 21 illustrates the steep-angle cutter hub 600, where cutter arms 610 (as shown in Figure 19) are optionally replaced by cutter blade support 702 with cutter blade 750 attached, preferably by screw 748 or other mechanism. Adapter 720 allows self-aligning flexibility with threaded attachment to rotor shaft 930 (Figure 16). Other cutter hub designs that are functionally equivalent are within the scope of the present invention as would be understood by those skilled in the art.

Figure 22 illustrates various angularly inclined positions and shapes of the cutter blades 750. The blade angle 755 can vary from approximately 0° to approximately 110° or greater relative to die hard face 370. See e.g., Figures 22a, b, and c. A blade angle 755 of 60° to 79° , as shown in Figure 8, is preferred. A blade angle of 75° , as shown in Figure 22b, is more preferred. The blade cutting edge 760 can be square, beveled, or angled and can be at a blade cutting angle 765 of approximately 20° to approximately 50° , with approximately 45° being preferred. Alternatively, a half-thickness blade 770, as

illustrated in Figure 22d, can be similarly attached, similarly angled, and can have comparable blade cutting angles and preferences as described above. Additionally, blade designs, dimensionally and compositionally, can prove useful depending on other process parameters.

The cutter blade 750 compositionally includes, but is not limited to, tool steel, stainless steel, nickel and nickel alloys, metal-ceramic composites, ceramics, metal or metal carbide composites, carbides, vanadium hardened steel, suitably hardened plastic, or other comparably durable material, and can be annealed, hardened, and/or surface treated as known to those skilled in the art. Wear-resistance, corrosion resistance, durability, wear lifetime, chemical resistance, and abrasion resistance are some of the factors influencing the utility of a particular blade relative to the formulation being pelletized. There are no particular constraints on the blade dimensions of length, width, and thickness as well as on the number of blades used relationally with the cutter hub design.

Figure 1 illustrates the relative position of the bypass loop 550. Water, or a comparable fluid for use in the bypass loop 550 and pellet transportation, is obtained from reservoir 1600 (or other source) and is transported toward the transport fluid box or waterbox 400 through pump 500 that can have a design and/or configuration to provide sufficient fluid flow into, and through, the optional heat exchanger 520 and transport pipe 530 to bypass loop 550. The heat exchanger 520 similarly can be of a design having a suitable capacity to maintain the temperature of the water (or other transport fluid) at an appropriate level to maintain the temperature of the pellets being formed such that pellet geometry, throughput, and pellet quality are satisfactory without tailing, and where wrap-around of molten plastic on the cutting face, agglomeration of pellets, cavitation, and/or accumulation of pellets in the transport fluid box or waterbox are avoided. Temperatures, flow rates, and composition of the transport fluid will vary with the material or formulation being processed. Transport fluid temperatures are maintained at least approximately 20°C below the melting temperature of the polymer and preferably are maintained at a temperature of approximately 30°C to approximately 100°C below the melt temperature. The transport fluid temperature is more preferably maintained from approximately 0°C to approximately 100°C, with approximately 10°C to approximately

90°C more preferred, and with approximately 60°C to approximately 85°C most preferred.

Additionally, processing aids, flow modifiers, surface modifiers, coatings, surface treatments (including antistats) and various additives known to those skilled in the art can be accommodated in the transport fluid. Piping, valving, and bypass components should be of suitable construction to withstand the temperature, chemical composition, abrasivity, corrosivity, and/or any pressure requisite to the proper transport of the pellet-transport fluid mixture. Any pressure required by the system is determined by the vertical and horizontal transport distance, pressure level needed to suppress unwanted volatilization or premature expansion of components, pellet-transport fluid slurry flow through valving, coarse screening, and ancillary process and/or monitoring equipment. Pellet-to-transport fluid ratios should similarly be of varying proportions to be satisfactorily effective in eliminating or alleviating the above-mention complicating circumstances, of which pellet accumulation, flow blockage, obstruction, and agglomeration are exemplary. Piping diameter and distances are determined by the material throughput (thus the flow rate and pellet-transport fluid ratio) and time required to achieve an appropriate level of cooling and/or solidification of the pellets to avoid undesirable volatilization and/or premature expansion. Valving, gauges, or other processing and monitoring equipment should be of sufficient flow and pressure rating as well as of sufficient pass-through diameter to avoid undue blockage, obstruction or otherwise alter the process leading to additional and undesirable pressure generation or process occlusion. Transport fluid and additive composition should be compatible with the components of the pellet formulation and should not be readily absorbed into/onto any of the components in that formulation. Excess transport fluid and/or additives should be readily removable from the pellets by such methods as rinsing, aspiration, evaporation, dewatering, solvent removal, filtration, or a similar technique understood by those skilled in the art.

A bypass loop 550, as illustrated in Figure 23, allows the transport fluid (e.g., water) from inlet pipe 530 to enter three-way valve 555 and be redirected into the bypass flow or toward the transport fluid box or waterbox 400. To bypass the transport fluid box or waterbox 400, the transport fluid is directed by three-way valve 555 into/through bypass pipe 565 into outlet pipe 570. To achieve this, blocking valve 575 is closed.

Alternatively, to allow water to flow to/through the transport fluid box or waterbox 400 the three-way valve 555 is directed to allow flow into/through pipe 560 and into pipe 580 with blocking valve 575 open and with drain valve 590 closed. Water proceeds into/through transport fluid box or waterbox 400 and transports pellets into/through sight glass 585 through blocking valve 575 and into outlet pipe 570 for down-stream processing as described below. To drain the system and allow cleaning or maintenance of the transport fluid box or waterbox 400 or die hardface 370, or to replace any of the die 320 components, three-way valve 555 directs flow into/through pipe 565 and into pipe 570. With blocking valve 575 now closed and drain valve 590 open, the water remaining entrapped below 575, in components 585, 400, 560, and 580 drains out drain 595 for recycling or disposal.

Once the pellet is sufficiently solidified for processing, it is transported via pipe 1270 to/through an agglomerate catcher/dewatering unit 1300 and into the drying unit 1400 and downstream processes 2000, as illustrated in Figure 1.

The dryer 1400 can be any apparatus for achieving a controlled level of moisture for materials that can be flake, globular, spherical, cylindrical, or other geometric shapes. It can be achieved but is not limited by filtration, centrifugal drying, forced or heated air convection or a fluidized bed and is preferred to be a centrifugal dryer, and is most preferred to be a self-cleaning centrifugal dryer 1400.

Turning now to Figure 24, the pipe 1270 discharges the pellets and fluid slurry or concentrated slurry into an agglomerate catcher 1300 that catches, removes, and discharges pellet agglomerates through a discharge chute 1305. The agglomerate catcher 1300 includes an angled round bar grid, perforated plate, or screen 1310 that permits passage of fluid and pellets but collects adhered, clumped, or otherwise agglomerated pellets and directs them toward the discharge chute 1305. The pellets and fluid slurry then optionally pass into a dewaterer 1320, as shown in Figures 24 and 25. The dewaterer 1320 includes at least one vertical or horizontal dewatering foraminous membrane screen 1325 containing one or more baffles 1330 and/or an inclined foraminous membrane screen 1335 that enables fluid to pass downwardly into a fines removal screen 1605 and therethrough to the water reservoir 1600 (Figures 1 and 27). The pellets that still retain moisture on their surfaces are discharged from dewaterer 1320

into the lower end of the self-cleaning centrifugal dryer 1400 at a slurry inlet 1405, Figure 24.

As illustrated in Figure 24, the self-cleaning centrifugal pellet dryer 1400 includes, but is not limited to, a generally cylindrical housing 1410 having a vertically oriented generally cylindrical screen 1500 mounted on a cylindrical screen support 1415 at the base of the screen, and a cylindrical screen support 1420 at the top of the screen. The screen 1500 is thus positioned concentrically within the housing 1410 in radially spaced relation from the inside wall of the housing.

A vertical rotor 1425 is mounted for rotation within the screen 1500 and is rotatably driven by a motor 1430 that can be mounted at, and/or connected to, the base (Figure 26) or the top of the dryer. The motor 1430 is preferably mounted atop the upper end of the dryer, as seen in Figure 24. The motor 1430 is connected to the rotor 1425 by a drive connection 1435 and through a bearing 1440 connected with the upper end of the housing. The connection 1445 and bearing 1440 support the rotor 1425 and guide the rotational movement of the upper end of the rotor. The slurry inlet 1405 is in communication with the lower end of the screen 1500 and rotor 1425 through the lower screen support section 1450 at connection 1448. The upper end of the housing and rotor is in communication with a dried pellet discharge chute 1460 through a connection (not shown) in the upper screen support section 1455 at the upper end of the housing. A diverter plate 1465 in outlet 1467 can divert dried pellets out of exit 1470 or exit 1475.

The housing 1410 is of sectional construction, connected at a flanged coupling (not shown) at a lower end portion of the dryer and a flanged coupling (not shown) at the upper end portion of the dryer. The uppermost flange coupling is connected to a top plate 1480, that supports bearing structure 1440 and drive connection 1435, which are enclosed by a housing or guard 1437. A coupling 1432 atop the housing 1437 supports the motor 1430 and maintains all of the components in assembled relation.

The lower end of the housing 1410 is connected to a bottom plate 1412, which is on top of a water tank or reservoir 1600, by a flange connection 1610 as illustrated in Figure 27. Apertures 1612 provide a means of communication between the lower end of the dryer housing and the reservoir 1600 to allow discharge of fluid from the housing 1410 into the reservoir 1600 as the surface moisture is removed from the pellets. This

removal is achieved by action of the rotor, which elevates the pellets and imparts centrifugal forces to the pellets so that impact against the interior of the screen 1500 will remove moisture from the pellets with such moisture passing through the screen and ultimately into the reservoir 1600.

The self-cleaning structure of the dryer includes a plurality of spray nozzles or a spray head assembly 1700 supported between the interior of the housing 1410 and the exterior of the screen 1500, as illustrated in Figure 24. The spray nozzle assembly 1700 is supported at the end of the spray pipes 1702 extending upwardly through top plate 1480 at the upper end of the housing, with the upper ends 1704 of the spray pipes 1702 being exposed. Hoses or lines 1706 feed high pressure fluid (e.g., water) at a flow rate of at least 40 gallons per minute (gpm) to the spray nozzles 1700. In some embodiments, the pressure fluid can be fed at a rate of about 60 gpm to about 80 gpm, while in others the rate can be at 80 gpm or higher. The hoses 1706 can optionally feed off a single manifold (not shown) mounted on the dryer 1400.

There are preferably at least three spray nozzle assemblies 1700 and related spray pipes 1702 and lines 1706. The spray nozzle assembly 1700 and pipes 1702 are oriented in a circumferentially spaced relation about the periphery of the screen 1500, and oriented in staggered vertical relation so that pressurized fluid discharged from the spray nozzles 1700 will contact and clean the screen 1500, inside and out, as well as the interior of the housing 1410. Thus, any collected pellets that have accumulated or are lodged in hang-up points or areas between the outside surface of the screen 1500 and inside wall of the housing 1410 are flushed through apertures 1612 into the reservoir 1600, as seen in Figure 27. Similarly, leftover pellets inside the screen 1500 and outside the rotor 1425 are flushed out of the dryer and will not contaminate or become mixed with pellets passing through the dryer during a subsequent drying cycle where a different type pellet is dried.

The region between the screen support section 1450 at the lower end of the dryer and the inner wall of the housing 1410 includes flat areas at the port openings and seams that connect the components of the dryer housing together. The high pressure water from the spray nozzle assembly 1700 effectively rinses this region as well. The base screen support section 1450 is attached to the bottom plate 1412 of the housing 1410 and

reservoir 1600 by screws or other fasteners to secure the housing and screen to the reservoir 1600. The base screen support section 1450 is in the form of a tub or basin as shown in Figure 24. Alternatively, in other dryer configurations, the base screen support section 1450 can be in the form of an inverted tub or inverted base (not shown).

The rotor 1425 includes a substantially tubular member 1427 provided with inclined rotor blades 1485 for lifting and elevating the pellets and subsequently impacting them against the screen 1500. In other dryers, the rotor 1410 can have a square, round, hexagonal, octagonal, or other cross-sectional shape. A hollow shaft 1432 extends through the rotor 1425 in concentric spaced relation to the tubular member 1427 forming the rotor. The hollow shaft guides the lower end of the rotor as it extends through an opening 1482 in a guide bushing 1488 at the lower end of the rotor 1425, as well as aligned openings in bottom plate 1412 and the top wall of the reservoir 1600, respectively. A rotary coupling 1490 is connected to the hollow shaft 1432 and to a source of fluid pressure, preferably air (not shown) through hose or line 1492 supply to pressurize the interior of the hollow shaft 1432.

The hollow shaft 1432 includes apertures to communicate the interior of the hollow rotor member 1427. These holes allow for the pressurized fluid (e.g., air) to be introduced into the interior of the rotor 1425. The rotor 1425, in turn, has apertures in the bottom wall that communicate the bottom end of the rotor 1425 with the interior of the base or tub section 1450 to enable the lower end of the rotor 1425 and the tub section 1450 to be cleaned. Pellets flushed from the rotor and inside screen 1500 are discharged preferentially through the dried pellet outlet chute 1460.

The top of the rotor 1425 inside top section 1455 is also a hang-up point and subjected to the high pressure fluid to dislodge accumulated pellets. As shown in Figure 24, a nozzle 1710 directs the high pressure fluid across the top of the rotor 1425 to drive accumulated pellets out of the top section and preferentially into the pellet outlet chute 1460. The nozzle 1710 is fed by a hose or line (not shown) that extends through top plate 1480 and is connected to a high pressure fluid source.

In addition to hang-up points or areas in the dryer structure, the agglomerate catcher 1300 can also be cleaned by a separate pipe or hose 1720 controlled by a solenoid valve that directs high pressure fluid onto the pellet contact side of the angled

agglomerate grate or catcher plate and bar rod grid 1310 to clean off agglomerates that are then discharged through the discharge tube or chute 1305.

A hose and nozzle can supply bursts of air (or other fluid) to the discharge chute or pipe 1460 in a direction such that it cleans the top of the rotor 1425 and the pellet discharge outlet 1460. The air discharge blows pellets past pipe connections and the diverter plate 1465 through outlet 1467 for discharge of dried pellets from the dryer.

The rotor 1425 preferably turns continuously during the full cleaning cycle. Solenoid valves are provided to supply air preferably at about 60 psi to 80 psi, or more, to additional hang-up points (not shown) that include the water box bypass air port, rotor air ports, top section air port, pellet outlet air port, and diverter valve air port. The solenoid valves include timers to provide short bursts (e.g., about three seconds), which cleans well and does not require a lot of time. A clean cycle button (not shown) activates the cleaning cycle with the water box bypass air port being energized first to allow air to purge the bypass with a multiplicity of air bursts (e.g., five or more). The top section air port is then activated. This is followed sequentially with activation of the diverter plate 1465. This valve closes prior to activation of the spray nozzle assembly 1700 that washes the screen for about one to about ten seconds, preferably about six seconds. The blower 1760 should be deactivated during the water spray cycles and is then reactivated when the spray nozzle pump is de-energized thus completing one cleaning cycle. The cycle as herein described is not limited in scope and each component of the cycle can be varied in frequency and/or duration as necessitated to achieve appropriate removal of the residual pellets.

The screens for the process can optionally include one or more horizontal or vertical dewatering screens 1325, inclined dewatering screens 1335, port screens 1595, and/or one or more cylindrically attachable screens 1500, as illustrated in Figure 28. The size, composition, and dimensions of the screens should accommodate the pellets being generated and can be perforated, punched, pierced, woven, or of another configuration known to those skilled in the art and can be the same or different in construction, composition, and style. As the pellet size decreases in diameter, preferably the screens will be composed of two or more layers. These layers can be of similar or different

composition, design, and size. The screens are secured in place by latches, clamps, bolts, or any other fastening mechanism.

The screens 1500 are preferably of suitably flexible construction as to be circumferentially placed around the dryer 1400 and rotor 1425 and can contain deflector bars 1550, as illustrated in Figures 29 and 30, that are bolted in placed effectively segmentalizing the screen area into approximately equal areas. Alternatively, the screens can be free of deflector bars, as seen in Figures 31 and 32. Preferably screens 1500 have two or more layers functionally incorporating an outer support screen and an inner screen that accomplishes the effective drying of the pellets and smaller micropellets. Additionally, one or more screen layers can be sandwiched between the outer support screen and the inner screen depending upon the particular application. Figure 33 illustrates an edge view of a three-layer configuration; and Figure 34 illustrates a similar edge view of a two-layer configuration. Figure 35 illustrates a surface view of a two-layer screen configuration in that the view is from the side of the support layer through which the finer mesh screen layer can be seen.

The outer support screen 1510 can be formed from molded plastic or wire-reinforced plastic. The polymer/plastic can be chosen from polyethylene, polypropylene, polyester, polyamide or nylon, polyvinyl chloride, polyurethane, or a similarly inert material that capably maintains its structural integrity under chemical and physical conditions anticipated in the operation of the centrifugal pellet dryers. Preferably the outer support screen 1510 is a metal plate of suitable thickness to maintain the structural integrity of the overall screen assembly and flexible enough to be contoured (e.g., cylindrically) to fit tightly and positionally in the appropriate centrifugal pellet dryer. The metal plate is preferably 18 gauge to 24 gauge, and most preferably is 20 gauge to 24 gauge in thickness. The metal can be aluminum, copper, steel, stainless steel, nickel steel alloy, or a similarly non-reactive material inert to the components of the drying process. Preferably the metal is a stainless steel alloy, such as Grade 304 or Grade 316 stainless steel as necessitated environmentally by the chemical processes undergoing the drying operation.

The metal plate can be pierced, punched, perforated, or slotted to form openings that can be round, oval, square, rectangular, triangular, polygonal, or other dimensionally

similar structure, to provide open areas for separation and subsequent drying. Preferably, the openings are round perforations and geometrically staggered to provide the maximum open area while retaining the structural integrity of the outer support screen. The round perforations are preferably at least approximately 0.075 inches in diameter and are staggered to provide an open area of at least approximately 30%. More preferred is an open area geometric orientation such that the effective open area is approximately 40% or more. Most preferred are round perforations having a diameter of at least approximately 0.1875 inches that are staggered to achieve an open area of approximately 50% or more.

Alternatively, the outer support screen can be an assembled structure or screen composed of wires, rods, or bars, stacked angularly, orthogonally, or interwoven, and welded, brazed, resistance welded or otherwise fixed in position. The wires, rods, or bars can be plastic, wire-reinforced plastic, or metal, and can be geometrically round, oval, square, rectangular, triangular, wedge-shaped, polygonal or other similar structure. The wires, rods, or bars across the width or warp of the screen can be the same as, or different, dimensionally as the wires, rods, or bars longitudinally contained as the weft, or shute.

Preferably the wires, rods, or bars are a minimum of approximately 0.020 inches in the narrowest dimension, more preferably are at least approximately 0.030 inches in the narrowest dimension, and most preferably are approximately 0.047 inches in the narrowest dimension. Open areas are dimensionally dependent on the proximal placement of adjacent structural elements and are positionally placed so as to maintain a percent open area of at least about 30%, more preferably above about 40%, and most preferably greater than or equal to about 50%.

The optional middle screen(s) 1520 and the inner screen 1530 are structurally similar to that described herein for the outer support screen. The screens in the respective layers can be similar or different in dimension and composition. The percent open area of the respective screens can be similar or different, but a lesser percent open area will reduce the effective open area of the screen and the least percent open area will be the most restrictive and therefore the delimiting percent open area for the screen assembly. The orientation of any screen relative to other layers of the assembly as well as the dimension and structural composition of the screens can be similar or different.

The inner screen 1530 is preferably a woven wire screen that can be in a square, rectangular, plain, Dutch, or similar weave, wherein the warp and weft wire diameters can be the same or different dimensionally or compositionally. More preferably, the inner screen is a plain square or rectangular weave wire screen wherein the warp and weft wires are similar compositionally and dimensionally, and the open area is approximately 30% or greater. Even more preferably, the inner layer screen is plain square or rectangular approximately 30 mesh or larger mesh grade 304 or grade 316 stainless steel, and the warp and weft wires are of a size to allow at least approximately 30% open area; and, most preferably, the open area is at least about 50%. Still more preferred is an inner screen of a plain square or rectangular weave of approximately 50 or greater mesh, with a percent open area of approximately 50% or greater. If incorporated, the middle screen 1520 would be of a mesh intermediate between the support screen 1510 and the inner screen 1530 and can be similar or different structurally, geometrically, compositionally, and orientationally.

Pellets discharged from the pellet discharge chute 1460 can be sized, sieved, packaged, additionally dried, or subjected to further processing such as fluidization or transported for storage or immediate manipulation in accordance with the process requirements. The processes included herein are by way of reference without limitation.

The dryer 1400 and screens 1500 can be treated or coated for optimization of performance, reduction of static charge, improved wear resistance, enhancement of corrosion resistance, better abrasion resistance, facilitation of drying, or similar process improvement.

The pellets produced by the above described process can be of many shapes, including, but not limited to, round, square, rectangular, triangular, pentagonal, hexagonal, or otherwise geometric in cross-section; star-shaped or other decorative designs; and can be the same or different when viewed in a second cross-section perpendicularly to the first. Preferably, the pellets and micropellets are spherical to lenticular for the major or preponderant rotational component. It is additionally preferred that they be free of tailing, sharp points or protrusions that can readily fracture to produce unnecessary fines. Considerations for decorative inclusions to be incorporated into the molded article need realize that such ornamentation of the pellets and micropellets can be

compromised in its three-dimensionality by contact, impact, abrasion and/or agitation, which arise potentially from the drying process, through conveyance, transport, or through the rotational process itself.

For purposes of clarification and simplification, pellet size hereinafter reflects the dimensions of the die hole used to make them rather than take into consideration any variance due to increases or decreases from die swell or potential shrinkage. Pellet size is not limited and typically ranges from approximately 3.5 mm to approximately 0.5 mm (approximately 0.138 inches to approximately 0.020 inches), or smaller, readily encompassing any conception of differentiation between pellet and micropellet. As the pellet size decreases, pressure typically increases and throughput can be necessarily compromised. It is preferred that the major or preponderant rotational component be approximately 1.6 mm (approximately 0.063 inches) or smaller, more preferably approximately 1.0 mm (approximately 0.040 inches) or smaller, and most preferably approximately 0.7 mm (approximately 0.028 inches) or smaller. Pellets 0.7 mm (approximately 0.028 inches) and smaller are also described herein as “micropellets”. For intricately detailed or small molded articles it is preferred to use approximately 0.35 mm (approximately 0.014 inches) or smaller as herein detailed.

Pellets and micropellets preferably contain less than 1% powder or fines, which are less than approximately 0.007 inches (approximately 0.18 mm) in cross-section, more preferably less than approximately 0.5% fines, and most preferably less than approximately 0.1% fines. It has been found that smaller size pellets typically contain fewer fines.

Similarly, material and pellet composition is not limited to rotational molding grades as heretofore identified. Historically, for minimal shear molding, the material has typically been represented in a range of melt flow index from 200 grams/10 minutes to 1 gram/10 minutes as evaluated using ASTM D1238 at condition E (190°C at 2.16 kilogram load) or at the appropriate standard temperature for the material in consideration. More recently, the range preferably has been 50 grams/10 minutes to 1 gram/10 minutes and more preferably is 30 grams/10 minutes to 1 gram/10 minutes. The higher the melt flow index, typically the lower the average molecular weight range, and subsequently the lower the viscosity. For a particular material, there will exist a viscosity that is too high

to allow reasonable flow for lay-down at the conditions useful for rotational molding, and similarly will also exist too low a viscosity practicably applicable. Thus the criterion for choice of materials to pelletize in this application is similarly within, but not limited to, these preferable flow constraints.

The material and, more specifically, the pellet melt temperature constraint is defined by the limitations of the minimal shear molding equipment itself more so than it is compositionally. The melt temperature should be sufficiently low as to avoid premature reaction or expansion of the material, and should be such that undue volatilization of critical ingredients is similarly avoided. Melting temperatures leading to decomposition or degradation of the material are preferably circumvented as well. Purging of processing, conveyance, storage, and molding equipment can be necessary to minimize this risk regardless of melt or processing temperatures. Inert gases such as nitrogen or carbon dioxide are preferred but in themselves are not limiting. Application in the molding process will be described later in this document.

Physical property constraints would include avoidance of materials of sufficiently low glass transition temperature to be tacky at the processing, transport and conveyance temperatures to avoid potential agglomeration, blockage, or undesirable adhesion to surfaces outside the mold. Brittleness of the material is preferably avoided to minimize or eliminate the risk of fragmentation at any stage of the processing that can lead to undue formation of fines and potential dust and other safety hazards.

Chemical composition of the material, and specifically the pellets, meeting the preferential qualifications heretofore described are not limited to the traditionally marketed rotational molding grades and the subsequent listing in itself is not limiting. Pellets and micropellets can be made of polyethylenes (PE), including ultra-low density PE, linear low density PE, low density PE, medium density PE, high density PE, cross-linkable polyolefins, polypropylenes, and other polyolefins, and copolymers thereof, amorphous or crystalline. Similarly, derivatized polyolefins including alkyl substituted polyolefins; polyvinyl esters, acids, and their derivatives; polyvinyl halides; polyvinyl alcohols and their esters; aromatic and substituted-aromatic polyolefins; and their copolymers with these or other polyolefins can be pelletized. Adhesive materials and

waxes, typically within the above described compositions are readily pelletized using the processes of the instant invention.

Additionally, polyesters, polyamides, polyethers, polycarbonates, and polyurethanes, and their copolymers, amorphous or crystalline, are capable of pelletization. Cellulosics and biodegradable polymers are within the scope of the pelletization process as well.

The pellets can incorporate the polymeric components individually or in combination with any or all of the following components or additives, in any or all levels of loading, such that the constraints of the molding criteria set forth above are accomplished. Such additives can include, and are not limited to, antioxidants and antioxidant stabilizers or formulations, ultraviolet absorbers and ultraviolet absorption stabilizers or formulations, thermal stabilizers or formulations, extrusional processing aids, flow and rheology aids, melt processing aids, viscosity modifiers, pigments including liquid or solid formulations, pellet coatings, fillers, fibers, structural modifiers, impact modifiers, chemical and/or physical expanding agents and their respective formulations, nucleating agents, rheology modifiers, cross-linking agents or formulations, cross-linking facilitating agents and catalysts or formulations containing such, waxes, tackifiers or detackifiers, and can be solid or liquid or any combination.

Additionally, the pellets for use in molding can be similar or different in composition, and can be similar or different dimensionally and/or morphologically. Pellets for use in minimal shear molding of one, two, or more layers, can be used singularly or can be a multiplicity dimensionally, compositionally and/or morphologically for each respective layer.

Although it is preferred to use pellets or micropellets in their appropriate geometries, sizes, and compositions, the various embodiments of the invention is not specifically limited to use of pellets in that additives or minor components, solid or liquid, can be included in the molding composition.

The actual design of the minimal shear molding equipment can be found in the representational list of prior art references that in themselves are not intended to be limiting in design or scope or in their application to the various embodiments of the present invention.

Minimal shear molding includes processes that inherently rely on the melting and flowing of materials to generate a molded article on cooling and resetting of that melt flow. Rotational molding, and its variant rock-and-roll molding, are non-limiting examples of this technology. The process rotationally lends itself to one, two, or multiple dimensions/axes, about which the rotation occurs either continuously and circularly or oscillatorily and arcuately relative to its motion as transcribed by the mold and/or the arms of the molding apparatus.

One-dimensional or uniaxial rotation in one example circumscribes an arc of 360° or less and oscillates back and forth in a rock-and-roll motion for the defined distance of that arc that preferentially is less than approximately 360°, more preferentially is less than approximately 270°, and most preferentially is approximately 180° or less. By example, this would be of use in molding a portion of a part in a limited area of the mold. As an alternative, and more typically, one dimensional rotation circumscribes a continuous 360° circle about a singular axis. This is commonly applied to formation of, for example, linings in a pipe.

Two-dimensional or biaxial rotation combines the options for one-dimensional rotation with the same options for a second axis of rotation circumscribing at an angle different than the first axis and preferentially perpendicular or orthogonal to that first rotation axis. Thus one two-dimensional mode involves continuous circulation of one axis 360° about its center with simultaneous rotation of the second and preferably perpendicular axis circularly 360° about its center. Alternatively, the second mode involves continuous rotation circularly 360° about the first axis and oscillatorily about the second, perpendicular axis as similarly as preferentially indicated for the one-dimensional rotation discussion. Yet a third two-dimensional or biaxial mode involves oscillatory rotations with similar preference of degree of rotation about both perpendicular axes of rotation. The rotation rates and/or the degree of arcuate oscillation can be the same or different for the two axes. Additional detail as to possible limitations or constraints to these rotations are discussed below.

Multidimensional or multiaxial rotation follows from the discussion of two-dimension rotation in that each respective axis of rotation can be continuous and circular, or can be oscillatory and arcuate, and can be of the same or different rotation rate and/or

degree of arcuate oscillation, and each respective axis is oriented differently than each or any of the other axes.

The minimal shear molding apparatus can be heated while rotating within a confined heated area, for example, by an oven. The heated area be heated electrically, by forced air convection, fuel oil, gas, steam, infrared radiation, oil, direct open flame applied to the mold, or by use of a jacketed mold where the jacket can be heated electrically, with steam, or by through-circulation of a heat transfer fluid such as oil or water. Alternatively, the mold can be variably heated in differing zones or portions.

The mold design can be of suitable metal or ceramic material that can optionally be vented, and the size of the vent can be determined by those skilled in the art. The depth into the mold the vent pipe penetrates should be such that loss of material is minimized. The vent pipe can be permeably plugged to allow efflux of gas on heating, or remain unplugged. The size and design of the mold should be appropriate to the intended article including appropriate allowances for shrinkage of the molding material and any multiplicity of the layering requisite for the intended article. Size of the mold is limited by the part needed and by the minimal shear molding apparatus such that it does not prevent or interfere with the respective rotations in any way. The mold can be provided with an inert gas purge and/or direct supply lines.

The various embodiments of the present invention are further illustrated by the following non-limiting examples.

EXAMPLES

This example relates to a discussion of the molding process optimization.

Initial evaluations were conducted on a tank, as shown in Figures 36a and 36b, that is approximately 17 inches in height 2515, approximately 9.5 inches (approximately 24.13 centimeters) in diameter 2510, and has a vent 2520 that is approximately 0.5 inches (approximately 1.27 centimeters) in outside diameter. The vent is inset approximately 1 inch (approximately 2.54 centimeters) from the outside wall of the tank. The vent was initially permeably plugged at its innermost end with fiberglass wool. Powder conditions for the tank were defined as an oven temperature of approximately 550°F (approximately 288°C), approximately 37 minute heating time, approximately 3.7:1 rotation ratio (major axis to minor axis) with a rotation rate of approximately 6 rpm. Internal air temperature

was not measured, and cooling on completion of the heating cycle was achieved with fan forced air to a temperature of no more than approximately 100°F (or approximately 38°C). Approximately five pounds (2.3 kilograms) of material, powder or micropellet was charged into the mold. Tanks were compared using 35 mesh or approximately 0.020 inch (approximately 0.5 mm) ground powder and approximately 0.014 inch (approximately 0.36 mm) micropellets produced using the apparatus described above. Evaluations were done on high density polyethylene (HDPE) and medium density polyethylene (MDPE). These preliminary tests for MDPE and HDPE showed that at equivalent temperatures, the powder tanks were satisfactory in terms of color and impact resistance while micropellet tanks were darker in color, though satisfactory in impact resistance. Reduction of time for the MDPE and HDPE of approximately 2 to approximately 5 minutes in heating time achieved comparable color to the powder tanks of longer heating duration.

Analogously, a more detailed study was conducted for black-pigmented cross-linkable polyethylene (XLPE), using approximately 0.020 inch (approximately 0.5 mm) ground powder, and micropellets of approximately 0.014 inch, approximately 0.020 inch, and approximately 0.038 inch (approximately 0.36 mm, 0.5 mm, and 0.97 mm, respectively) at oven temperatures of approximately 500°F, 475°F, 450°F, 425°F, 400°F, 375°F, and 350°F (approximately 260°C, 246°C, 232°C, 218°C, 204°C, 191°C, and 177°C, respectively). The heating time was reduced to approximately 27 minutes in accordance with the preliminary testing. It is important to note that conditions other than oven temperature and cure time were also modified to improve the uniformity of the lay-down on the inner tank surface such that the rotation ratio was adjusted to 5:1.2 and the rotation rate was increased to approximately 10 rpm. Melting temperatures for all materials were approximately 240°F to 250°F (approximately 116°C to 121°C) and cross-linking temperatures for the XLPE are approximately 350°F or 177°C.

Gel Test Results for XLPE Evaluations

Sample	Temp.	Powder	0.014"	0.020"	0.038"
Black	400°F -----		78.0%	73.3%	76.8%
	425°F -----		90.1%	89.2%	89.9%

450°F	87.2%	83.4%	85.8%	85.9%
475°F	87.5%	90.8%	90.7%	88.7%
500°F	89.3%	90.5%	91.4%	91.5%

Impact tests for the successfully gelled samples, > 70%, all passed the 200 foot-pounds (ft.-lbs.) testing level.

Gel formation and low temperature impact were determined in accordance with ASTM D 1998-97 with the powder standards defined as a minimum of 70% gel formation for XLPE and low temperature impact of a minimum 100 ft-lbs for this tank for all of the cited materials. Values for gel formation of 84% and low temperature impact of 200 ft.-lbs. are preferred for the instant tank.

It is readily apparent from this data that successful molding and cross-linking is achieved at significantly lower temperatures in micropellets than is possible in the standard powder. The quality of the inner surface of the tank actually improves as the temperature of the oven is reduced and as the pellet size increases such that a approximately 0.038 inch (approximately 0.97 mm) pellet at approximately 450°F (approximately 232°C) is comparable to a powder tank or an approximately 0.014 inch (approximately 0.36 mm) micropellet tank prepared at approximately 500°F (approximately 260°C). This phenomenon was originally attributed to competition between the rate of melting and lay-down as compared with the cross-linking rate in that it appeared both reasonable and logical that cross-linking proceeded more rapidly as the temperature increased resulting in a rougher surface due to the improper lay-down.

Gel tests were determined on the micropellets to insure no cross-linking was occurring. All sizes contained no presence of gels.

It has also been observed in making natural and white-pigmented powder XLPE tanks that discoloration decreased as the oven temperature decreased. To evaluate this, tanks were similarly prepared as above and measured for gel formation and impact.

Gel Tests for XLPE Powder Tanks

Sample	Temp.	% Gel
Black	400°F	0%

White	400°F	0%
Natural	400°F	41.2%
Black	425°F	0%
White	425°F	81.0%
Natural	425°F	90.4%
Black	450°F	87.2%
White	450°F	80.1%
Natural	450°F	90.9%
Black	475°F	87.9%
White	475°F	82.7%
Natural	475°F	91.2%
Black	500°F	89.3%
White	500°F	82.5%
Natural	500°F	82.9%

Impact tests for the successfully gelled samples, > 70%, all passed the 200 ft.-lb. testing level.

The color of the natural tanks progressively lightened and the inside surface of the white tank lightened from brown, to tan, to faintly yellow, and ultimately to white as the oven temperature was reduced from approximately 500°F to approximately 425°F (approximately 260°C to 218°C).

To investigate the color improvement and the inner surface improvement, testing was conducted on the MDPE at comparable temperature intervals and approximately 0.020 inch, approximately 0.030 inch, and approximately 0.045 inch (approximately 0.5 mm, 0.76 mm, and 1.14 mm, respectively) pellets, as well as the standard powder. Color improved with both reduction of temperature as well as with increase in pellet size. As

had been observed with the XLPE, the inner surface of the larger pellets improved as the temperature decreased, resulting in a rough and uneven surface at approximately 500°F (approximately 260°C) with improvement to a very smooth surface at approximately 425°F (approximately 218°C), comparable to both the powder tanks and the smaller size pellet tanks. In all cases, the color of the powder tank was considerably more brown than the micropellet tanks and similarly improved as the oven temperature was reduced. Of particular note, the powder tank and the approximately 0.020 inch (approximately 0.5 mm) pellet tank failed impact testing at the approximately 100 ft. lb. level, while all other testing proved successful.

From these examples it is readily apparent that micropellets can readily be made into tanks using considerably lower oven temperatures, reduced heating times, and can be of varying sizes. Additional testing allowed tanks to be made with larger MDPE pellets including approximately 0.063 inch, approximately 0.090 inch, and even the irregular and cylindrical approximately 0.125 inch (approximately 1.6 mm, 2.3 mm, and 3.2 mm, respectively) as-supplied pellets. As the pellet size increased, the lay-down of the inner surface was not as uniform, and heating times would have to have been increased to effect the requisite uniformity. Conclusively, it is reasonable to work in the approximately 0.020 inch to approximately 0.040 inch (approximately 0.5 mm to 1.0 mm) micropellet sizes with considerably reduced oven temperature and/or reduced heating times.

To investigate venting phenomena, rotation ratio, and rotation rate variance, a smaller tank, as illustrated in Figures 37a and 37b, was used. The tank was approximately 9.5 inches (approximately 24 centimeters) in height 2615, approximately 8.25 inches (approximately 20.1 centimeters) in diameter 2610, and has an approximately 2.25 inch (approximately 5.7 centimeters) vent 2620 centered in the top of the tank. Approximately two pounds (0.9 kilograms) of approximately 0.038 inch (approximately 0.97 mm), black XLPE micropellets were charged into the tank and molding conditions were set to monitor failure of tank formation. Thus, the oven temperature was evaluated at approximately 400°F to as low as approximately 350°F (approximately 204°C to 177°C), heating time was reduced to approximately 25 minutes, rotation ratios were evaluated at 5:1.2, 4:1.2, and 3:1.2 and rotation rates were reduced from the previous 10

rpm or 8 rpm to only approximately 1.75 rpm, and ultimately approximately 1.5 rpm. Tanks were successfully prepared with acceptable inner surface lay-down. Gel formation was not evaluated, as it was not anticipated to pass the requisite 70% following original data. It is important to note that the oversize venting compared to previous testing did not appear to alter the melt or lay-down processes. Powder was particularly problematic to use with the vent as it poured out readily without some obstruction, whereas the pellets were well-contained as long as the vent length was sufficiently inside the mold that the cumulative volume of pellets was contained in inverted positioning. It was found that the slower the rotation ratio the better the pellets were retained inside, and the losses of the powder were greater. This is in tremendous contrast to the early prior art that reflect rotation rates of 20 rpm to as high as 100 rpm or more that would reasonably be expected to seriously compromise and impose significant stress on the minimal shear molding equipment itself. The larger the mold design, and thus the requisite molding apparatus, the more significantly detrimental the high rotation ratios and rotation rates become.

Without intending to be bound by any theory, it seems reasonable to conclude that higher rotation ratios and higher rotation rates are necessary with the powder to overcome the various flow irregularities that are not issues with the more spherical, easily rollable pellets. These increased rates are anticipated to sufficiently agitate the powders so as to keep significant amounts suspended in the air, attributing to the improved smoothness and uniformity of the inner surface at the higher temperatures. This latter phenomenon attributes to the excessive loss of powder as it is rigorously tossed throughout the interior of the mold. Only a small amount of powder is initially in contact with the heating surface of the mold, and this appears to contribute to the need for longer heating times at higher temperatures. Since the pellets are free to roll, the rotation times and rotation ratios can readily be lowered allowing more intimate and prolonged time in contact with the heating mold surface and the better lay-down at the lower temperature. With less agitation, there is reduced proclivity for the micropellets to be tossed or to roll out of the vent even when the vent is considerably oversized.

An added benefit to the facility with which the micropellets roll is the ease of conveyance and transport from a storage facility to the molding equipment. Many means of conveyance (e.g., from pouring by hand, mechanically dumping the pellets, vacuum

conveyance, vibratory or belt conveyance, to pneumatic conveyance) can be used. Such means are often highly problematic for powders because of the various constraints on ease of flow in combination with particularly serious dust and fine generation that is a maintenance and, more importantly, a potentially serious health and safety hazard for both personnel and equipment. The use of micropellets significantly reduced or eliminated the dust issues as long as the brittleness of the compositions chosen is minimized as described above.

Pellets of various sizes were evaluated for bulk density to determine the impact this can have on storage, transport, conveyance, and mold loading. Both powders and micropellets were compared for XLPE and MDPE.

Table of Approximate Bulk Density Determinations

Material	Size	Bulk Density
Black XLPE	powder	24.14 lb./cu. ft.
Black XLPE	0.014"	33.59 lb./cu. ft.
Black XLPE	0.020"	34.35 lb./cu. ft.
Black XLPE	0.038"	34.69 lb./cu. ft.
MDPE	powder	25.60 lb./cu. ft.
MDPE	0.020"	36.14 lb./cu. ft.
MDPE	0.030"	37.24 lb./cu. ft.
MDPE	0.045"	37.59 lb./cu. ft.
MDPE	0.063"	38.18 lb./cu. ft.
MDPE	0.078"	36.14 lb./cu. ft.
MDPE (as is)	0.125"	36.48 lb./cu. ft.

Bulk determinations are considered approximate as they were made by weighing the powder or pellets in an irregular container and comparing those weights to the weight of water determined for that container with appropriate conversions.

This data illustrates that the bulk density of the micropellets in the preferred size range as previously defined is significantly greater than the comparable powder, thus

significantly reducing the storage and transportation costs as compared with that powder. Assuming *arguendo* tremendous error in the elementary technique used and for the lesser anticipated possible compaction of the pellets on transport and storage, a conservative savings of approximately 30% seems reasonable. By way of example, only two silos instead of three would be necessary for storage, or a shipment of ten railcars now requires only seven.

Bulk density and its relation to volume is of significant importance in high surface area molds that have small volumes and therefore often have required more than one charge of powder. Figure 38a illustrates a cut-away view of a portion of a small unvented mold that is only approximately 0.75 inches (approximately 1.9 centimeters) in height with orifices 2710 penetrating completely through the part with a partial cut orifice indicated as 2715. Figure 38b illustrates a cross-section of this same molded part of molded wall 2720. The charge required approximately 0.5 pounds (approximately 0.2 kilograms) of powder that physically had to be packed into the mold as tightly as possible to obtain a satisfactory part. Micropellets of approximately 0.014 inches, approximately 0.020 inches, or approximately 0.038 inches (approximately 0.36 mm, 0.5 mm, and 0.97 mm, respectively) were used without need for packing or undue challenge as the volume fit comfortably into the mold. Molded articles of the approximately 0.014 inch (approximately 0.36 mm) micropellets were at least equivalent to the powder in terms of surface quality, which was free from pinholes and other surface imperfections. Approximately 0.020 inch and approximately 0.038 inch (approximately 0.5 mm and 0.97 mm, respectively) micropellets contained pinholes that were not eliminated by variation of oven temperature, heating time, rotation ratios, or rotation rates. This is important, as it illustrates the need for specific micropellet sizing as the mold size decreases and/or intricacy increases. Importantly, no compromise was obtained by using the micropellets when chosen to be dimensionally appropriate for the molded article design while being of particular importance in satisfactorily charging the mold in only one portion without packing or other manipulations not accomplished with powders.

Additionally, pinhole reduction was evaluated using micropellets of polypropylene and copolymers of polypropylene and polyethylene. The tank, as illustrated in Figures 36a and 36b, was charged with approximately five pounds

(approximately 2.3 kilograms) of the respective material and molded at approximately 550°F (approximately 288°C) for approximately 30 minutes using a rotation ratio of 4:1.2 and a rotation rate of approximately 8 rpm. The resulting tanks were full of numerous pinholes, many of which penetrated through the wall of the tank. Pinholes are generally thought to be indicative of gas entrapment, whether from air or potentially from volatiles that can include moisture. The testing was repeated by charging a comparable amount of respective material in approximately 0.030 inch and approximately 0.020 inch (approximately 0.76 mm and 0.05 mm, respectively) micropellets into the mold and heating each at approximately 300°F (approximately 149°C) for 15 minutes and then at approximately 550°F (approximately 288°C) for approximately 18 minutes keeping rotation ratios and rates the same. The number of pinholes was drastically reduced and ultimately eliminated when the approximately 300°F (approximately 149°C) first stage heating was increased to approximately 20 minutes, followed by the same second stage heating at approximately 550°F (approximately 288°C) for approximately 18 minutes similarly rotated. This suggests that volatilization or gas entrapment can be eliminated in a vented mold effectively with reasonably appropriate modification of the molding conditions.

Another particular challenge in molding rotational grade powders is to achieve intricate threading without bubble formation and entrapment in the thinnest portions of the lead-in thread. Such a design is illustrated in Figures 39a, 39b, and 39c. Figure 39a illustrates a side view of such threads 2810 with the encircled area 2805 being the most significant point of concern. Tanks were molded with HDPE powder using standard powder conditions previously described and, as illustrated in Figure 39b, resulted in large numbers of bubbles in the circled region as indicated, many of which penetrated through the thin thread leading to a highly irregular ragged appearance. Comparable molding with approximately 0.014 inch (0.36 mm approximately) micropellets of HDPE resulted in a much whiter tank with only a few very minor granules in the thinnest portion of the thread with minimal bubbles and no penetration as noted in Figure 39c.

Figure 40a illustrates a “kiss-off” region with proximal regions that are very close to touching 2905, or physically and intimately touch 2915 as in Figure 40c. Use of rotational molding grade powders often has proven problematic leading to bridging 2910

that can be small strands or larger columns where portions of the “kiss-off” region of the two walls touch as shown diagrammatically in Figure 40b. This proved to be equally challenging for micropellets. It was discovered, however, that choice of materials can lead to the desired effect. Molding utilizing HDPE resulted in significant bridging be it in powder or micropellet form and bridging in various “kiss-off” regions was different regardless of the particulate used. However, satisfactory and reproducibly uniform “kiss-off” regions were made throughout molded articles when micropellets or powder of XLPE were used. This presents options where material selection and/or particle selection can be chosen such that the desired effect is achieved.

Long walls of a molded article that are in close proximity, as illustrated in Figures 41a and 41b, are equally difficult to mold with powders and typically result in uneven wall thickness favoring areas of angles where multiple wall junctures receive more cumulative heating and thus result in thicker walls 4010 (Figure 41a). The use of approximately 0.020 inch (0.5 mm approximately) micropellets of HDPE resulted in a significant lessening of the buildup in the angular deposition 3005 as compared with the powder but use of approximately 0.020 inch (approximately 0.5 mm) micropellets of XLPE showed considerable improvement, as illustrated in Figure 41b. Reduction of rotation rates are believed to significantly affect the lay-down of micropellets allowing more reasonable time to distribute themselves more uniformly. As detailed above, such rate reduction is not workable in the use of powders. This demonstrates a combination of use of micropellets with proper selection of material as potentially impacted by rotation rates as an effective method to achieve otherwise problematic molded articles as compared with powders.

Tanks were also made in accordance with Figure 36 using materials such as polyethylene terephthalate glycolate (PETG) in approximately 0.025 inch (approximately 0.64 mm) micropellets and mixtures of amorphous and crystalline polyesters coextruded in approximately 0.040 inch (approximately 1.0 mm) micropellets. Both materials commercially available non-rotational molding grade materials and were not modified with additives. Molding conditions included an oven temperature of approximately 550°F (approximately 288°C) for heating times of 30 minutes utilizing rotation ratios of 4:1.2 and rotation rates of approximately 10 rpm. Charges of seven pounds were used

anticipating the volume necessary at higher densities than are typical of the polyolefins discussed above. Both were successfully molded and only minimal shrink rate was observed, making removal from the mold a considerable challenge. Tapered mold design is anticipated as a reasonable solution for minimal molded article shrinkage situations as exhibited here. The appearance of the tanks will be discussed below.

In addition to the various tanks described, approximately 0.014 inch (approximately 0.36 mm) micropellets of HDPE were made into approximately 1450 gallon, approximately 3900 gallon, and approximately 12500 gallon (5488 liters, 14762 liters, 47,313 liters, respectively) tanks comparable in design to Figure 36 and were found to be much smoother on the inside wall of the tank than comparably molded powders as explained due to the various problems with powder flow and lay-down. All tanks passed impact testing and showed no ill effects fully loaded with water. No unzipping or vertical splitting of tank walls was observed as would be anticipated if the hoop stresses circumferentially about the tank were not appropriately offset by the structural integrity of the molded tank wall.

Two layer tanks have been molded for many years in that the first powder material that forms the outside wall of the part is charged into an appropriate mold, molded at the appropriate conditions for a particular heating time, optionally cooled or removed from the oven, and then an additional powder material is charged into the mold now containing the first layer. This is subjected to the same or different molding conditions. Alternatively, additional layers can be added as well. Once completed, the mold is removed from the oven, cooled appropriately, and the molded article is removed. The second and any subsequent shots can be introduced manually by opening the mold and physically removing a small portion of the first layer to introduce the second material for molding, the use of meltable containers within the mold or various designs of “drop boxes” that are mechanically or thermally opened at an appropriate time, temperature, or other appropriately determined point can be used to introduce the material for the next layer.

Traditionally each layer is made from powder that can be the same or different dimensionally and/or compositionally. The layers can also contain reactive or expandable materials, examples of which include cross-linking or foam production. In

particular, prior art has also demonstrated use of foams in large pellets introduced simultaneously with powders such that the powder melts initially to lay-down a continuous and non-perforated wall. Inside of the wall is deposited the foamable material that can form a distinct layer and is then foamed or can be foamed to completely fill the core of the part. Two layer technology can be used to add a barrier layer to a structural element, or to provide decorative enhancement, or to serve as a reinforcing layer for improved structural rigidity, among other applications.

Two layer or multi-layer tank manufacturing is not without its hazards and safety issues, as the second layer material should be added as quickly as possible while rotation and heating are effectively discontinued. With bigger tanks come greater heat retained by the molding equipment, greater worker heights from the floor, larger amounts of the material required, and greater dust hazards (e.g., while adding a powder into a hot tank while the heated and potentially hazardous or toxic gases are pouring out of the inside). Tremendous potential for safety hazards and the increased likelihood for accidents are self-evident.

As such, it is important to determine the effectiveness of micropellets in lieu of at least one material and preferably as replacement for all components. Black XLPE micropellets and natural MDPE or natural HDPE micropellets were chosen for the important color contrast for ease of visualization and because of the volume of experimentation done on these same materials. Figure 42a illustrates the idealized two-layer concept with outer layer 3110, inner layer 3105 and interface 3115. Particular problems that are common to two-layer molding include a continuous line of very small bubbles or microbubbles 3120 at the material interface (Figure 42b), a series of both large and small bubbles, macrobubbles 3125 and microbubbles 3120 respectively, at the interface (Figure 42c), an irregular juncture 3130 at the interface of the two layers (Figure 42d), or bubbles 3135 entrapped at varying depths and frequency (Figure 42e). Figure 42f illustrates a three layer tank with uniform intimate contact at all interfaces. Discussions concerning Figures 42a, 42b, 42c, 42d, 42e, and 42f are generally exclusive to expandable materials. An additional problem is complete or partial separation of the inner layer from the outer layer. Inherent in multiple layer molding is a tendency for the second layer to influence the structural integrity of the initial layer as cooling of the two

layers is not necessarily of similar rate and shrinkage rates can also be different. As a consequence pockmarks or sinks can develop on the outside wall of the tank. Note must be made here that similar phenomena can occur in one or more layer tanks if the choice of, or inappropriate application of, mold release is not prudently administered.

Using a tank, as illustrated in Figures 37a and 37b, powder conditions were optimized using approximately two pounds (0.9 kilograms) of black XLPE powder that was charged into the mold and heated at an oven temperature of approximately 425°F (approximately 218°C) for a heating time of 27 minutes at a rotation ratio of 4:1.2 and a rotation rate of approximately 10 rpm. On completion of the first layering, rotation was temporarily discontinued and the vent pipe optionally was removed. The MDPE powder was added carefully, the vent pipe reinserted, if desired, and rotation was continued at the same oven temperature, rotation ratio, and rotation rate for a heating time of approximately 15 minutes. A cross-linked black outer layer and a well-defined white inner layer were obtained as obviated by cutting the tank in half after cooling and removal from the mold. Layerage was comparable to that ideally illustrated in Figure 42a.

Previous work as detailed above demonstrated the facility of lay-down of approximately 0.014 inch and approximately 0.020 inch (approximately 0.36 mm and 0.5 mm respectively) micropellets at the lowest temperatures with the most uniform surface properties, inside and outside, for the molded tank. Optimization of conditions resulted in a tank comparable to that produced from powder when molding conditions were at an oven temperature of approximately 400°F (approximately 204°C) for approximately 19 minutes at a rotation ratio of 4:1.2 and rotation rate of approximately 10 rpm for approximately two (2) pounds of the approximately 0.014 inch or approximately 0.36 mm black XLPE micropellets. This was followed by molding conditions for the inner layer using approximately 1.25 pounds (approximately 0.57 kilograms) of approximately 0.020 inch (approximately 0.5 mm) micropellets of MDPE at similar oven temperature, rotation ratio, and rotation rate for approximately 15 minutes. Somewhat higher rotation rates are important in the second layer in particular to minimize the nonuniformity of the lay-down. Optimization using approximately 1.25 pounds (approximately 0.57 kilograms) of HDPE in lieu of MDPE yielded comparable results.

Importantly, introduction of the second material was initially done by discontinuing heating and rotation, removing the vent pipe, and charging the second material through a funnel with a layer of insulation around the base to prevent premature heating or melting of the materials being added. Introduction of micropellets was considerably easier than powder and required approximately one-half the time. Additionally, it was discovered that micropellets could easily be poured directly through the vent pipe, which significantly reduced the time the heating and rotation had to be discontinued. Optimization as described above utilized the technique of introduction of powder, though challenging and somewhat dusty, as well as the micropellets directly through the vent tube. Such direct addition without manipulation or damaging of the first layer is an important improvement over more standard two-layer molding techniques as revealed above.

Optimization of the two layer process resulted in generation of the various phenomena as described for the illustrations in Figures 42a, 42b, 42c, 42d, 42e, and 42f. Without being bound by theory, it is believed that the small bubble layer as illustrated in Figure 42b is an indication of incomplete out-gassing, devolatilization, or possible overcure of the first layer. By way of example, if the XLPE material in particular is cross-linked excessively, a liquid layer is observed on the inside surface that is not able to dissipate prior to lay-down of the second layer. Large bubbles in concert with smaller bubbles, shown in Figure 42c, is an indication of undercure, incomplete devolatilization, or incomplete outgassing of air from the lay-down of the material. Thus, material that is released after the second layer is trapped can form large bubbles as observed at the interlayer interface. Figure 42d illustrates unevenness of the inner surface of the outside layer prior to lay-down of the second or inner layer, and Figure 42e illustrates serious air entrapment, volatile outgassing, or chemical incompatibility of at least some of the ingredients in a particular formulation. This was observed in formulations where an additional amount of antioxidant package was introduced into the MDPE. Following comparable molding it was observed that bubble phenomena, as illustrated in Figures 42c and 42e, were significantly increased. Further testing revealed that formulations containing the added antioxidant package darkened considerably more than formulations without; and, as oven temperatures were reduced, tanks made from the MDPE with

additive failed impact testing at higher temperatures than did MDPE without the additive. This was considered to be additional indication of the counter-effects of the additive package as similarly observed in the two layer evaluations.

With two layer tanks accomplished in a satisfactory manner, evaluations of two-layer single-shot molding were pursued. Initially, attempts were made using black XLPE powder and approximately 1/8 inch (approximately 3.2 mm) cylindrical pellets of MDPE as received from the manufacturer. Some tendency for separation was observed in the various attempts but admixture was problematic throughout the wall of the tank with some minor penetration of the white MDPE material through to the outside layer of the wall. More problematic was the roughness of the inside surface where the uniform lay-down of the large pellets was not successful. Similar results were achieved by following this lead though utilizing pellets of MDPE in sizes not commercially available at the time of the testing.

Prior art using powder and pellets for single layer moldings and usage in foamables has favored lay-down of the powder first theorizing that the smaller particle sizes more readily absorb heat thus softening sufficient to become slightly tacky and adhere to the wall of the tank before sufficient heat energy has been absorbed by the larger pellet to begin similar softening. Based on this, work was initiated using the tank illustrated in Figures 36a and 36b. Regardless of pellet sizes, it was realized that increased rotation rates facilitated a better lay-down of a predominant layer. For a mixture of powder A and micropellet B, slower rotation rates favored lay-down of the powder more preferentially whereas with increased rotation rate the pellets became the preferential layer. Similarly, it was reasoned as follows from prior art that lower melting materials would lay down preferentially. Therefore it seemed reasonable to assume that lower melting point and smaller particle size should yield a preponderance of that material in initial lay-down. Surprisingly, even this could be countered by increase in rotation rate. It was further determined that use of a polyethylene for one layer and a higher melting material such as a polypropylene or polyester for the second layer should yield a predominant lower melting polyethylene layer with later lay-down of the higher melting layer. Once again, increase in rotation rates could reverse this depending on which material had the smaller particle size. Investigations of similar melting point

materials demonstrated that once again the smaller material could be forced to the outside with increased rotation regardless of the melting point. It followed in review that varying rotation rates could similarly reverse lay-down of higher melt density material or lower melt density and it was readily observable that pellet density could be reversed putting the lower density material down first or the heavier. Similar conflicts were observed with differing size micropellets in that variance of rotation rates could lay down the largest pellet first or the smaller pellet. Varying rotation ratios had significantly less effect than anticipated, and uniform tank formation was found to lie in a very narrow range of rotation ratios. From these initial explorations it became readily apparent that multiple variables including, but not limited to, particle or pellet size, chemical composition, melting point, softening point, polarity, melt density, particle density, rotation rates, and rotation ratios are all involved in the integral lay-down of the layers. It was also realized that the ability of a melting or softening material to lay down on a metal surface required appropriate ability to wet the surface as well as an ability to adhere to the surface. All of the materials cited herein were investigated in at least one and often many of the aspects as detailed herein.

Separation of any significance was first achieved combining approximately two pounds (approximately 0.9 kilograms) of approximately 0.014 inch (approximately 0.36 mm) black XLPE micropellets and approximately 1.25 pounds (approximately 0.57 kilograms) of approximately 0.020 inch (approximately 0.5 mm) MDPE micropellets. The oven temperature was approximately 400°F (approximately 204°C) with a heating time of approximately 27 minutes using a rotation ratio of 4:1.2 and a rotation rate of approximately 10 rpm. Rotation rates were lowered sequentially to approximately 6 rpm with additional gain in separation, thus oven temperature was lowered sequentially to approximately 350°F (approximately 177°C) this time with minimal gain. The rotation rate was again lowered to approximately 4 rpm with an increase in heating time to approximately 35 minutes with additional marginal gain. Variance of rotation ratios was marginal at best and cross-linking could not occur at the lower temperature so the temperature was raised incrementally with subsequent lowering of the rotation rate to obtain a reasonable separation. Replacing the MDPE with HDPE effected even more acceptable separation but resulted in poorer quality of the innermost layer surface, and

increasing the size of the XLPE facilitated even greater improvement. Acceptable separation was at last achieved using approximately 1.75 pounds of approximately 0.038 inch (approximately 0.8 kilograms and 0.97 mm) black XLPE micropellets and approximately 1.5 pounds (approximately 0.68 kilograms) of HDPE at an oven temperature of approximately 450°F (approximately 232°C) for a heating time of approximately 30 minutes and then increasing the oven temperature for approximately 7 minutes at approximately 475°F (approximately 246°C) at a rotation ratio of 4:1.2 at a surprising rotation rate of approximately 1.75 rpm. These conditions were repeated, but replacing the HDPE with MDPE, with even more uniform lay-down of the innermost surface area. As a result, none of the white MDPE or HDPE penetrated through to the outside surface of the black XLPE. Minor interlayering was observed at the interface of the two materials. The formation of two significant layers in which the outside surface of the tank was cross-linked XLPE and the inner tank surface was either MDPE or HDPE was achievable. An added benefit was found in using less compatible materials such as XLPE and polyester in that the small interlayering at the interface better facilitated the adhesion of otherwise challengingly incompatible materials.

Advisedly, need for absolute and distinct two-layer or multi-layer moldings, where barrier properties or other critical avoidance of any penetration of one-layer into the next, is best retained in the introduction of each individual layer. Adherence issues are markedly improved by introduction of a meltable adhesive layer to serve as bonding agent between such incompatible or limitedly compatible layers.

The evaluations leading to the formation of significant layering provides an excellent pathway to achieve extensive decorative effects. Polycarbonates and other clear materials (e.g., PETG), particularly amorphous materials, make clear molded items into that can be incorporated other shaped pellets or higher melting pellets to introduce unique and unusual decorative items. Admixture of amorphous and crystalline materials of similar melting point, such as the polyester described herein, resulted in a tank that uniquely contained a swirl pattern that was found to vary in accordance with minor variation of rotation ratios. Unusual pigmentation effects can be achieved by incorporation of pigment or of different pigments in the components of the varied layering effects described above. Utilization of constrained rock and roll in one or more

dimensions allows lay-down of one layer in a localized area, over which can be layered other materials or similar materials in other colors. Use of fractional melt and low melt index micropellets was found to give exceptionally strong tanks that were permeable to other lower melting materials to make unusually patterned items. Decorative combinations are unlimited by utilizing uniaxial, biaxial, or multiaxial capabilities with one or more of continuous and circular rotation or oscillatory and arcuate motion.

The embodiments of the present invention are not limited to the particular formulations, process steps, and materials disclosed herein as such formulations, process steps, and materials may vary somewhat. Moreover, the terminology employed herein is used for the purpose of describing exemplary embodiments only and the terminology is not intended to be limiting since the scope of the various embodiments of the present invention will be limited only by the appended claims and equivalents thereof. For example, temperature, pellet size, and time parameters may vary depending on the particular materials used.

Therefore, while embodiments of this disclosure have been described in detail with particular reference to exemplary embodiments, those skilled in the art will understand that variations and modifications can be effected within the scope of the disclosure as defined in the appended claims. Accordingly, the scope of the various embodiments of the present invention should not be limited to the above discussed embodiments, and should only be defined by the following claims and all equivalents.

What is claimed is:

1. A minimal shear molding composition for minimal shear molding at least one layer, the minimal shear molding composition comprising:
 - pellets, micropellets, powders, or a combination comprising at least one of the foregoing, having at least one chemical composition that can form the at least one layer;
 - wherein the pellets, micropellets, or powders used to minimal shear mold each of the at least one layers are compositionally different for each layer, such that each layer is compositionally different from an adjacent layer;
 - wherein no layer formed by the minimal shear molding composition consists only of powders; and
 - wherein the pellets and micropellets are spherical or lenticular in geometry.
2. The minimal shear molding composition according to Claim 1, wherein the at least one chemical composition is selected from the group consisting of polymers, copolymers, additives, and combinations thereof.
3. The minimal shear molding composition according to any one of claims 1 and 2, wherein the at least one chemical composition comprises polymers having reactive functionalities.
4. The minimal shear molding composition according to any one of claims 1 to 3, wherein the reactive functionalities are cross-linkable.
5. The minimal shear molding composition according to any one of claims 1 to 4, wherein the pellets have an average longest cross-sectional dimension of approximately up to 3.5 mm.
6. The minimal shear molding composition according to any one of claims 1 to 5, wherein the at least one chemical composition for each of the at least one layers comprises a minor component that comprises less than approximately 20 weight percent of the at least one chemical composition.

7. The minimal shear molding composition according to any one of claims 1 to 6, wherein no layer consists only of combinations of one size pellets and one size micropellets.
8. The minimal shear molding composition according to any one of claims 1 to 7, wherein one component of the combination is a micropellet having a cross-sectional dimension less than 0.6 mm.
9. A method of minimal shear molding, comprising:
 - providing pellets, micropellets, powders or a combination comprising at least one of the foregoing;
 - simultaneously forming at least one layer of a molded part with the pellets, micropellets, powders, or a combination of the foregoing;
 - wherein the pellets, micropellets, or powders used to minimal shear mold each of the at least one layers are compositionally different for each layer, such that each layer is compositionally different from an adjacent layer;
 - wherein no layer consists only of powders; and
 - wherein the pellets and micropellets are spherical or lenticular in geometry.
10. The method according to Claim 9, wherein an inner surface of the molded part is smooth without flow irregularities.
11. The method according to any one of claims 9 and 10, wherein the pellets have an average longest cross-sectional dimension of approximately up to 3.5 mm.
12. The method according to any one of claims 9 to 11, wherein simultaneously forming at least one layer of a molded part occurs at temperatures at least approximately 25°C below a molding temperature for a 35 mesh powder of a comparable composition.
13. The method according to any one of claims 9 to 12, wherein molding ratios are approximately 5:1 to approximately 3:1, as related to those for 35 mesh powders of a comparable composition.

14. The method according to any one of claims 9 to 13, wherein molding rotation rates are approximately 1.75 rpm to approximately 6 rpm, as related to those for 35 mesh powders of a comparable composition.
15. The method according to any one of claims 9 to 14, wherein minimal shear molding occurs rotationally in at least one axis.
16. The method according to any one of claims 9 to 15, wherein minimal shear molding occurs rotationally in at least two axes, and the rotation rates in different axes are different.
17. The method according to any one of claims 9 to 16, wherein partial layers are formed using arcuate and oscillatory motion.
18. The method according to any one of claims 9 to 17, wherein layers are formed by sequential addition of components.
19. The method according to any one of claims 9 to 18, wherein sequential addition of components is done manually or thermally by controlled release.
20. The method according to any one of claims 9 to 19, further comprising molding an adhesive layer between layers formed from the pellets, micropellets, or powder to limit separation of layers formed from the pellets, micropellets, or powder on cooling and storage of the molded part.
21. The method according to any one of claims 9 to 20, wherein all components for all layers are simultaneously charged into the mold to form layers without sequential addition of components.
22. The method according to any one of claims 9 to 21, wherein the separation into individual layers is achieved from at least one of differential melt viscosity, melt density, pellet

size, pellet density, softening temperature, melting temperature, polarity, component immiscibility, surface dynamics, or surface wetting.

23. The method according to any one of claims 9 to 22, wherein separation into individual layers is achieved by variance of at least one of rotation rate, rotation ratio, composition, molding process temperature, or molding process time.

24. The method according to any one of claims 9 to 23, wherein the adhesion of two adjacent layers formed is achieved by admixture at the interface between the two adjacent layers.

25. The method according to any one of claims 9 to 24, wherein a mold for the molded part is vented.

26. The method according to any one of claims 9 to 25, wherein the addition of material to the mold is achieved through the vent.

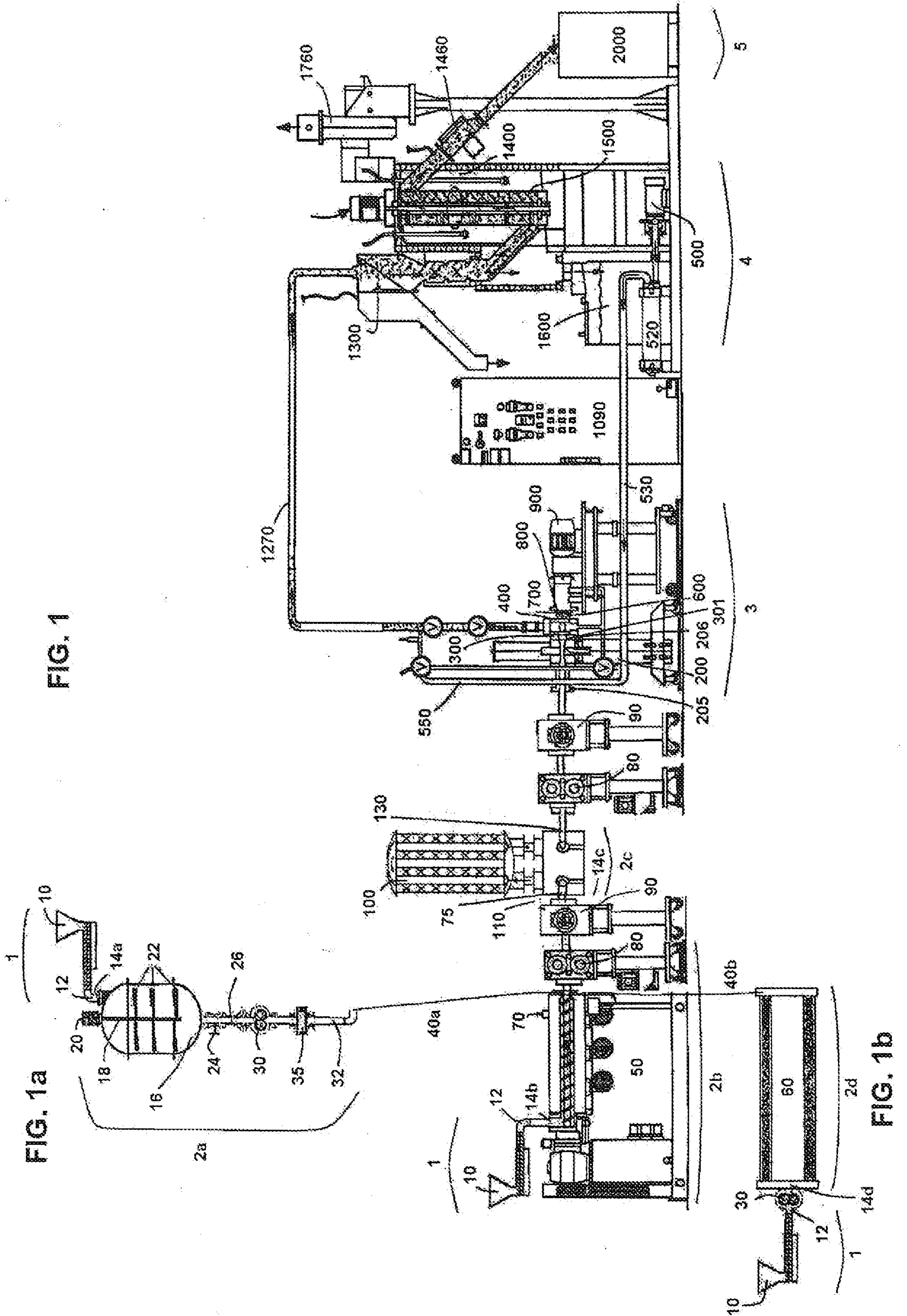
27. The method according to any one of claims 9 to 26, further comprising cross-linking of the molded part.

28. The method according to any one of claims 9 to 27, further comprising expanding a foaming agent in the at least one layer of the molded part.

29. The method according to any one of claims 9 to 28, further comprising providing at least one composition that does not melt.

30. The method according to any one of claims 9 to 29, wherein the choice of compositions and molding conditions include one or more of the use of oversize pellets, low melt index pellets, and fractional melt pellets, that confer decorative and functional three-dimensional molded parts.

31. The method according to any one of claims 9 to 30, wherein a composition for each of the at least one layers comprises a minor component that comprises less than approximately 20 weight percent of the composition.
32. The method according to any one of claims 9 to 31, wherein no layer consists of combinations of one size pellets and one size micropellets.
33. The method according to any one of claims 9 to 32, wherein one component of the combination is a micropellet having a cross-sectional dimension less than 0.6 mm.



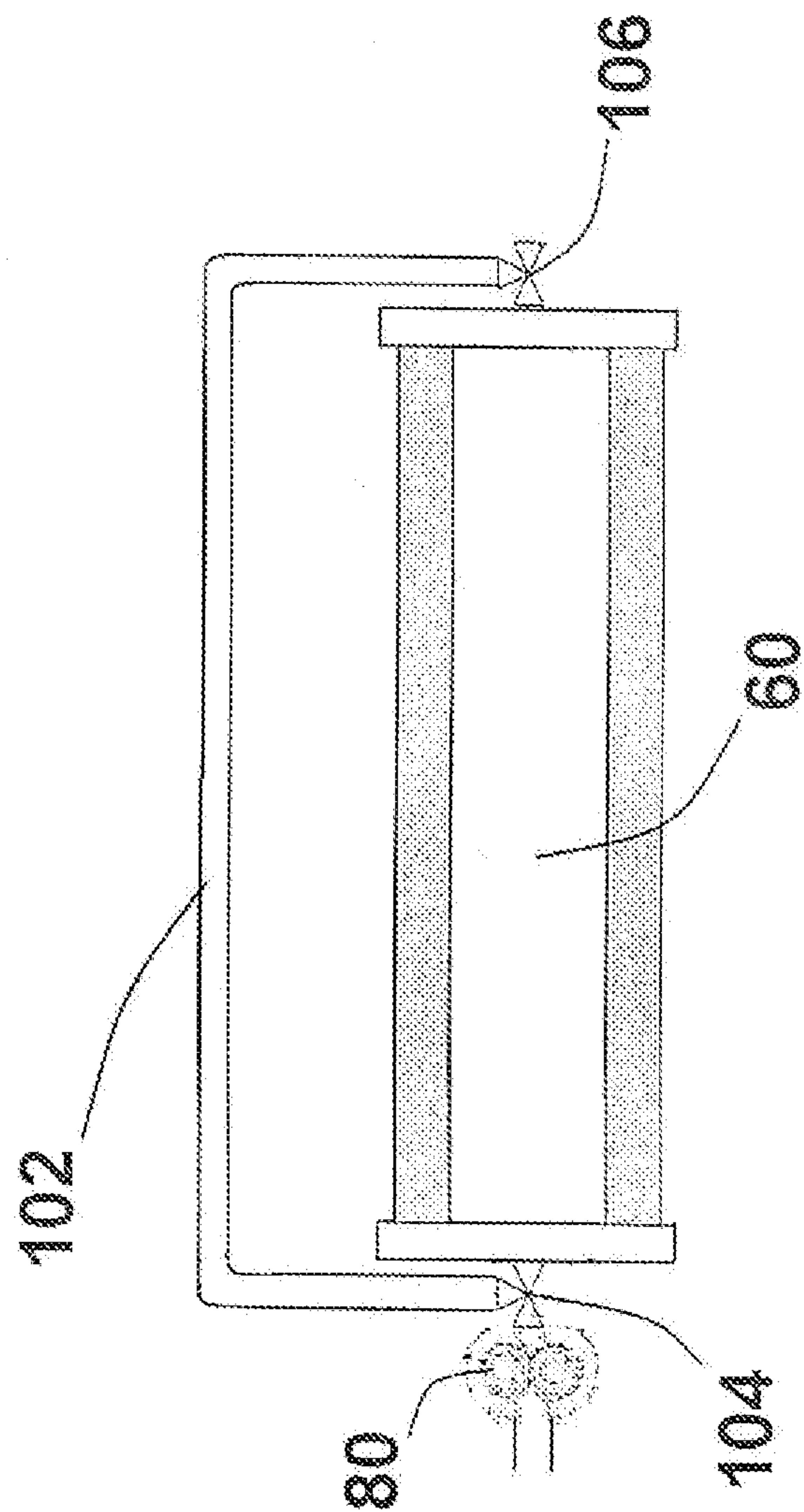


FIG. 2

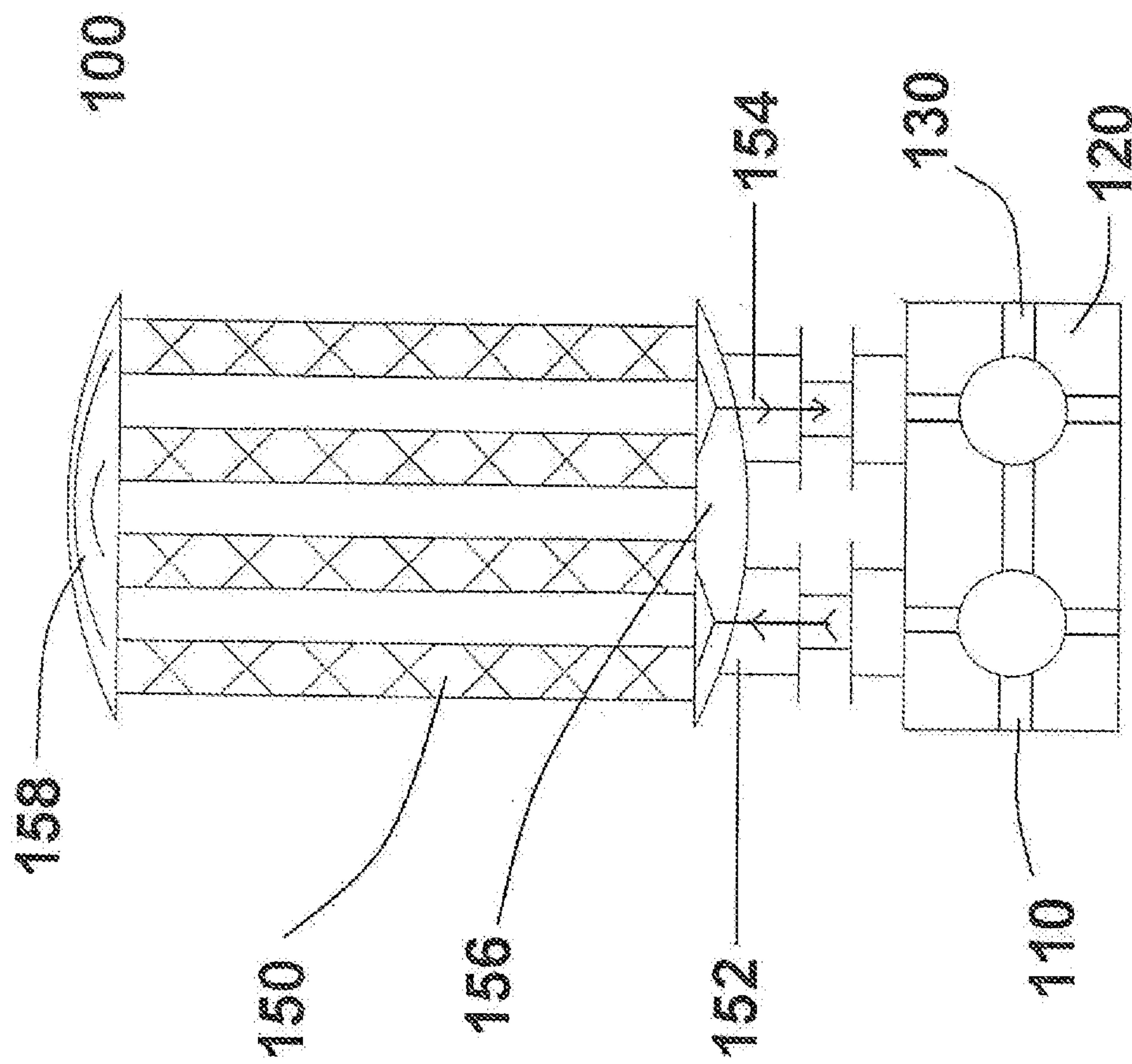


FIG. 3

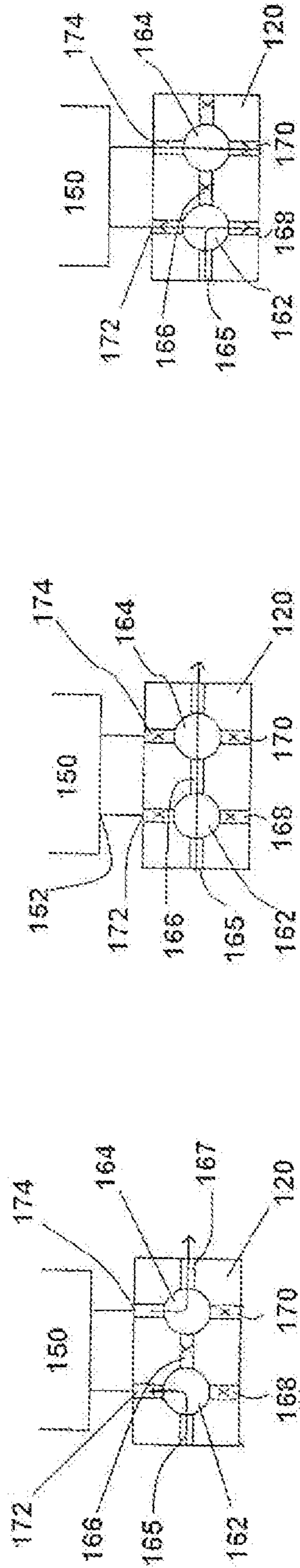


FIG. 4a

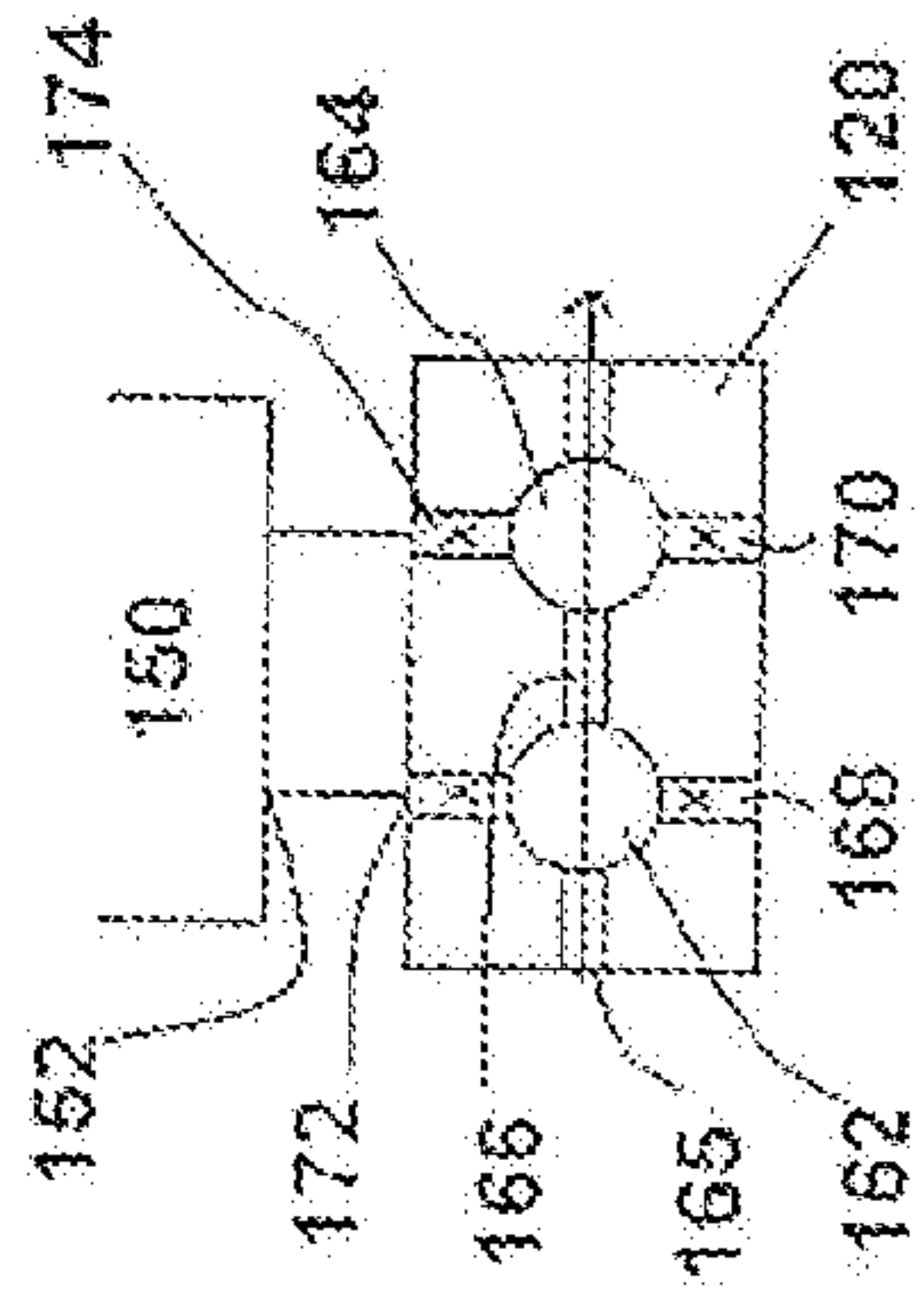


FIG. 4b

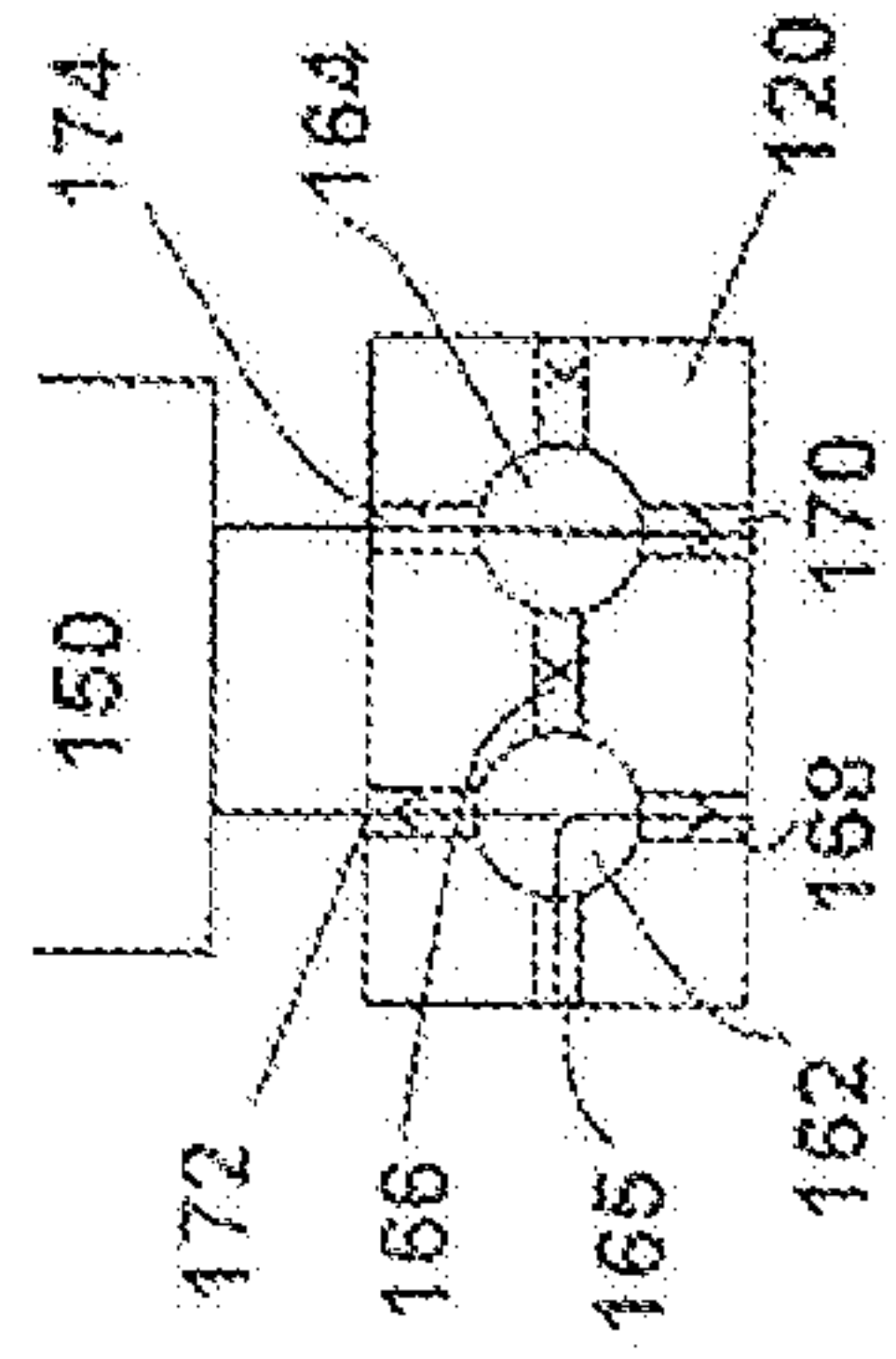


FIG. 4c

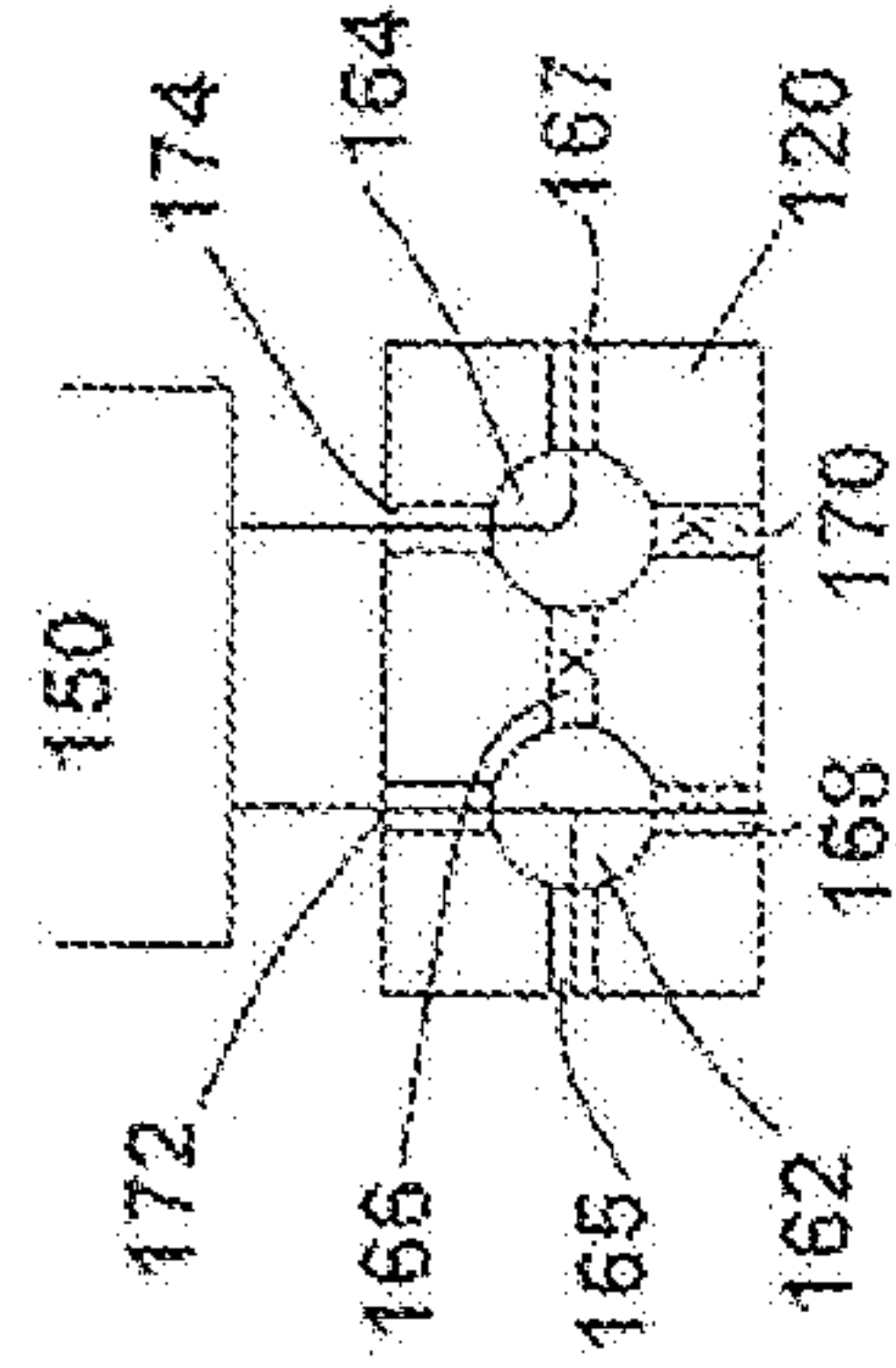


FIG. 4d

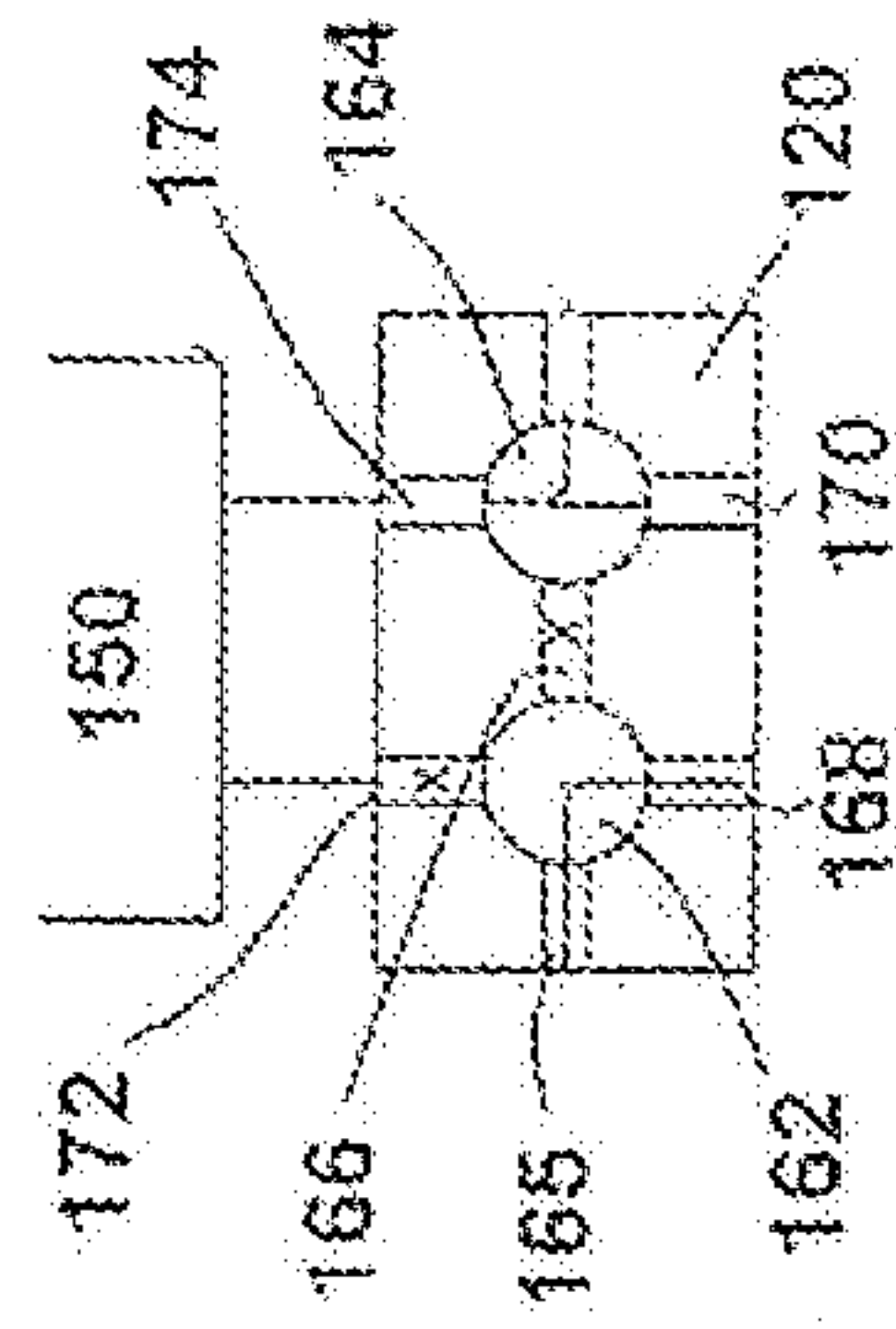
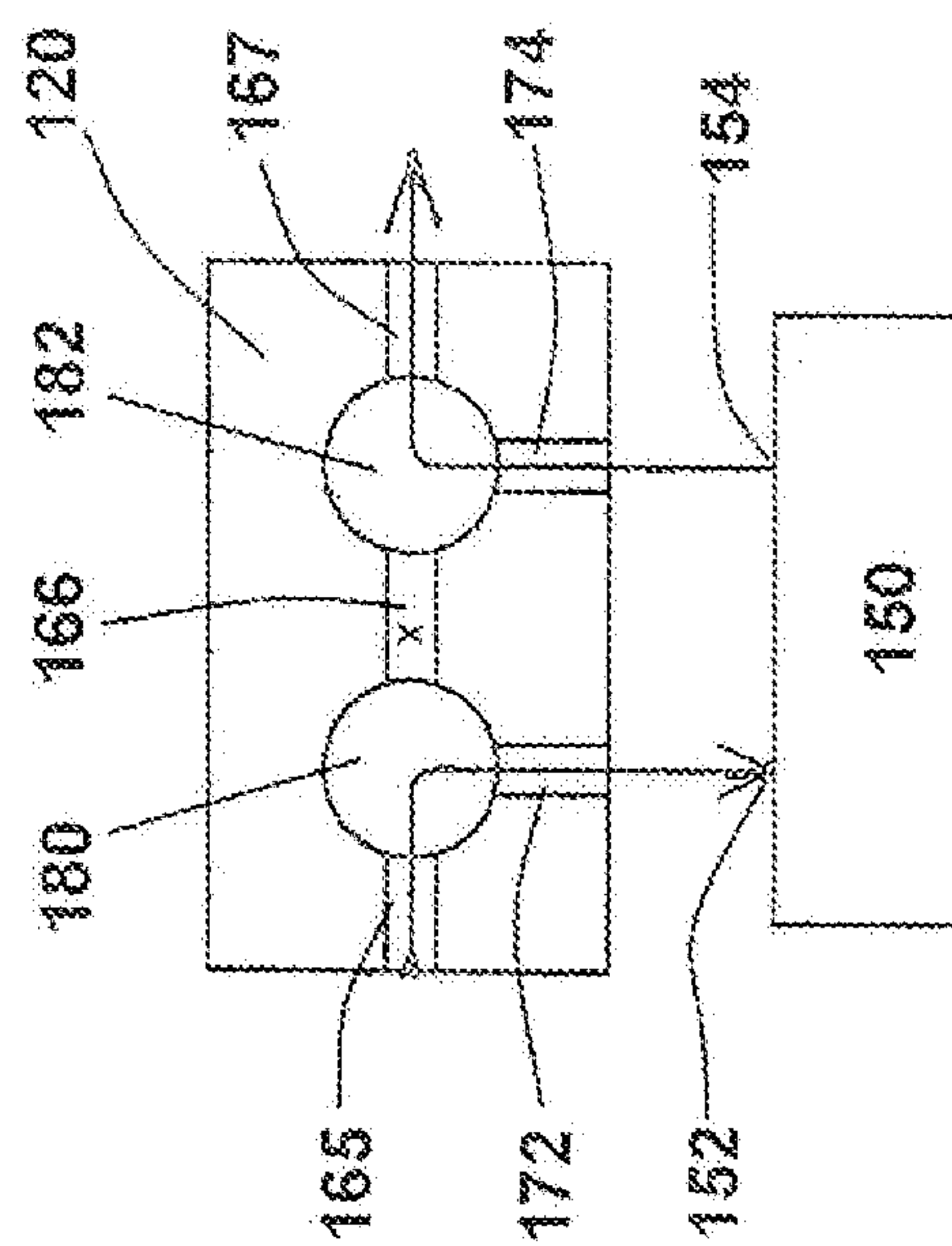
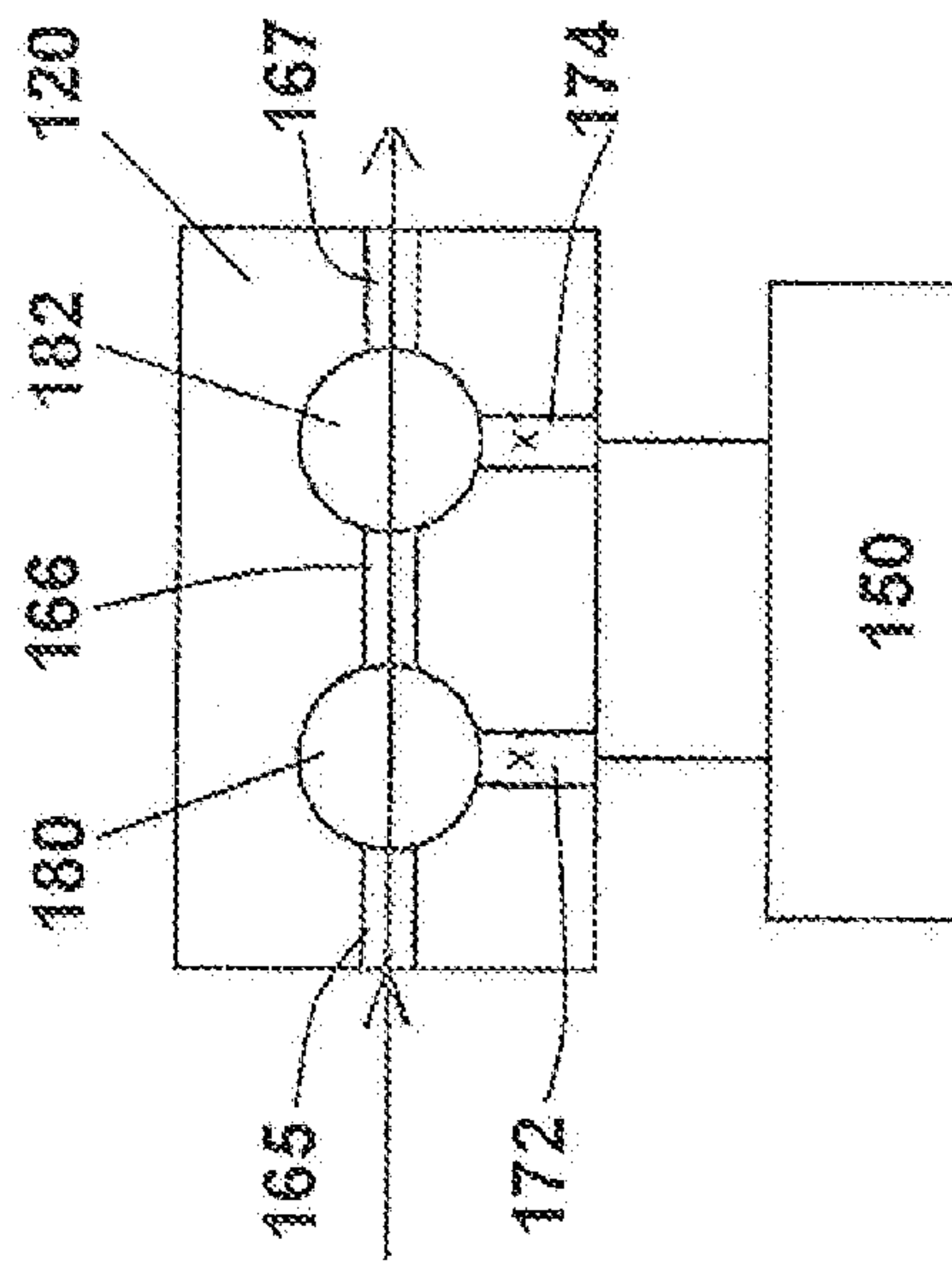


FIG. 4e

FIG. 4



50



Q. L. L.

50

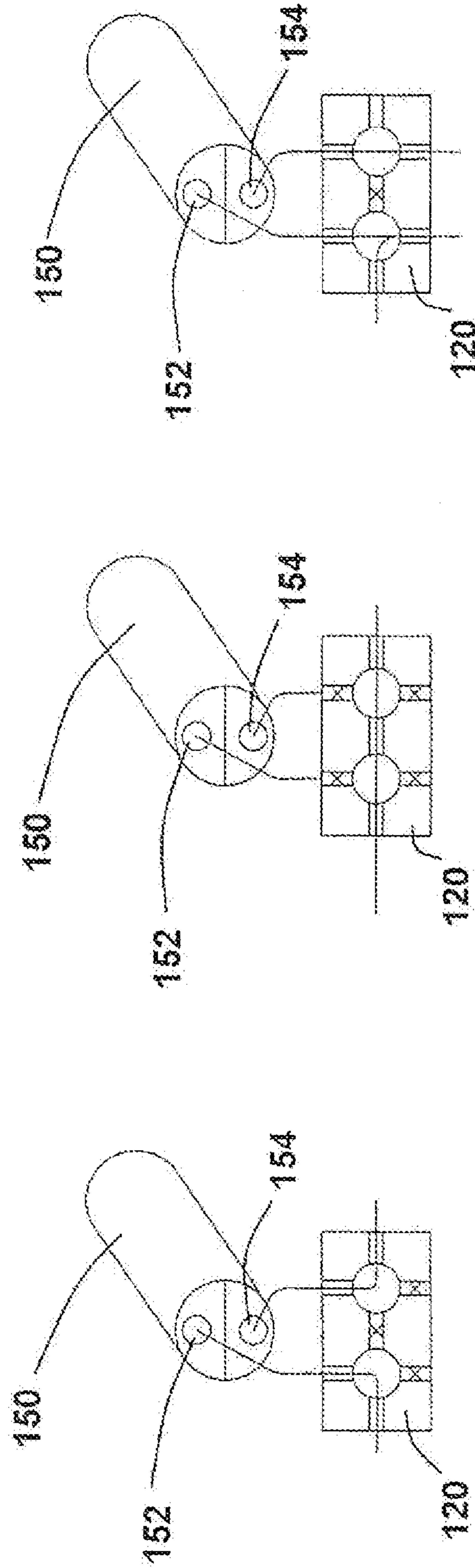


FIG. 6a

FIG. 6b

FIG. 6c

FIG. 6

701

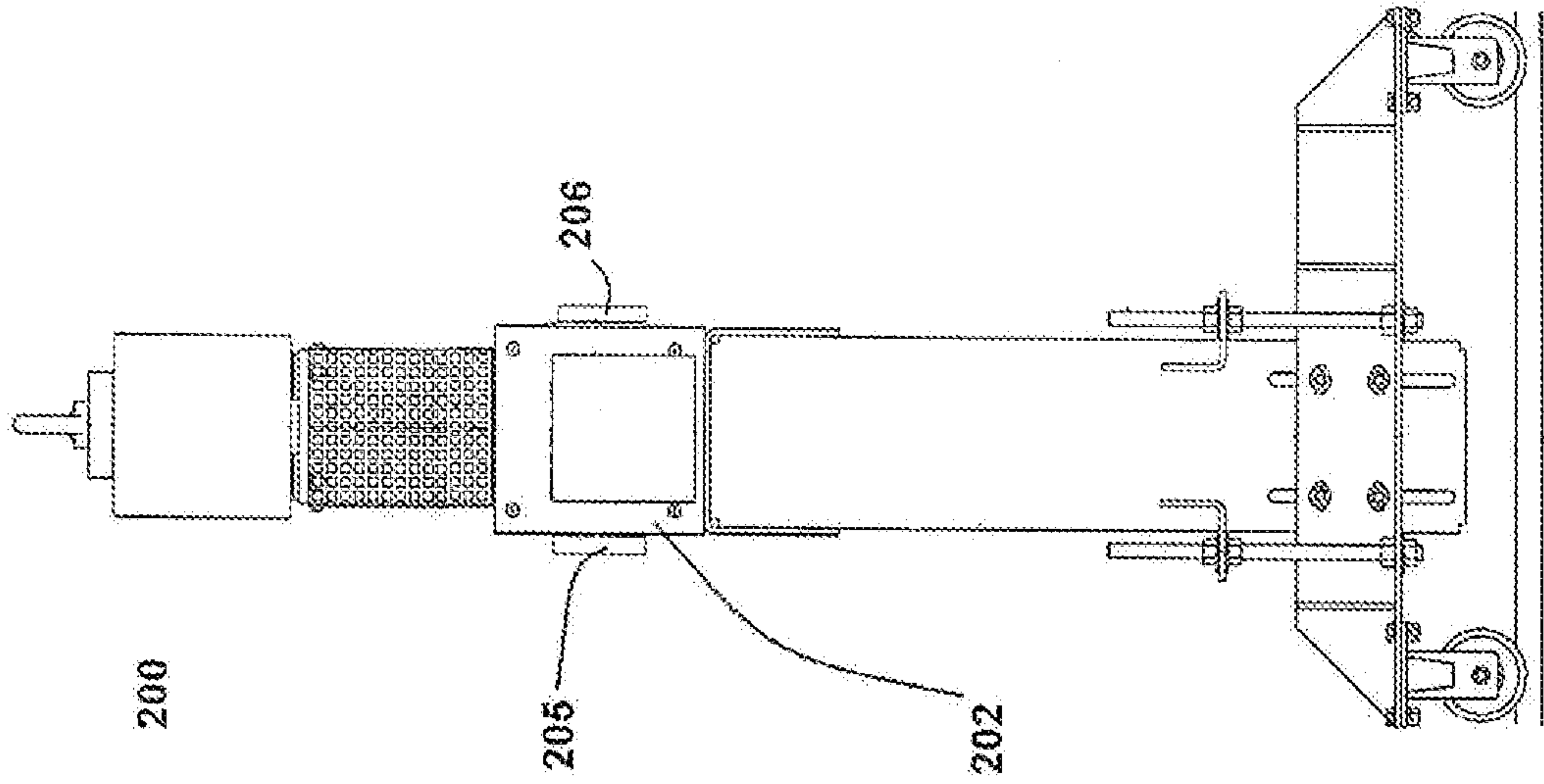
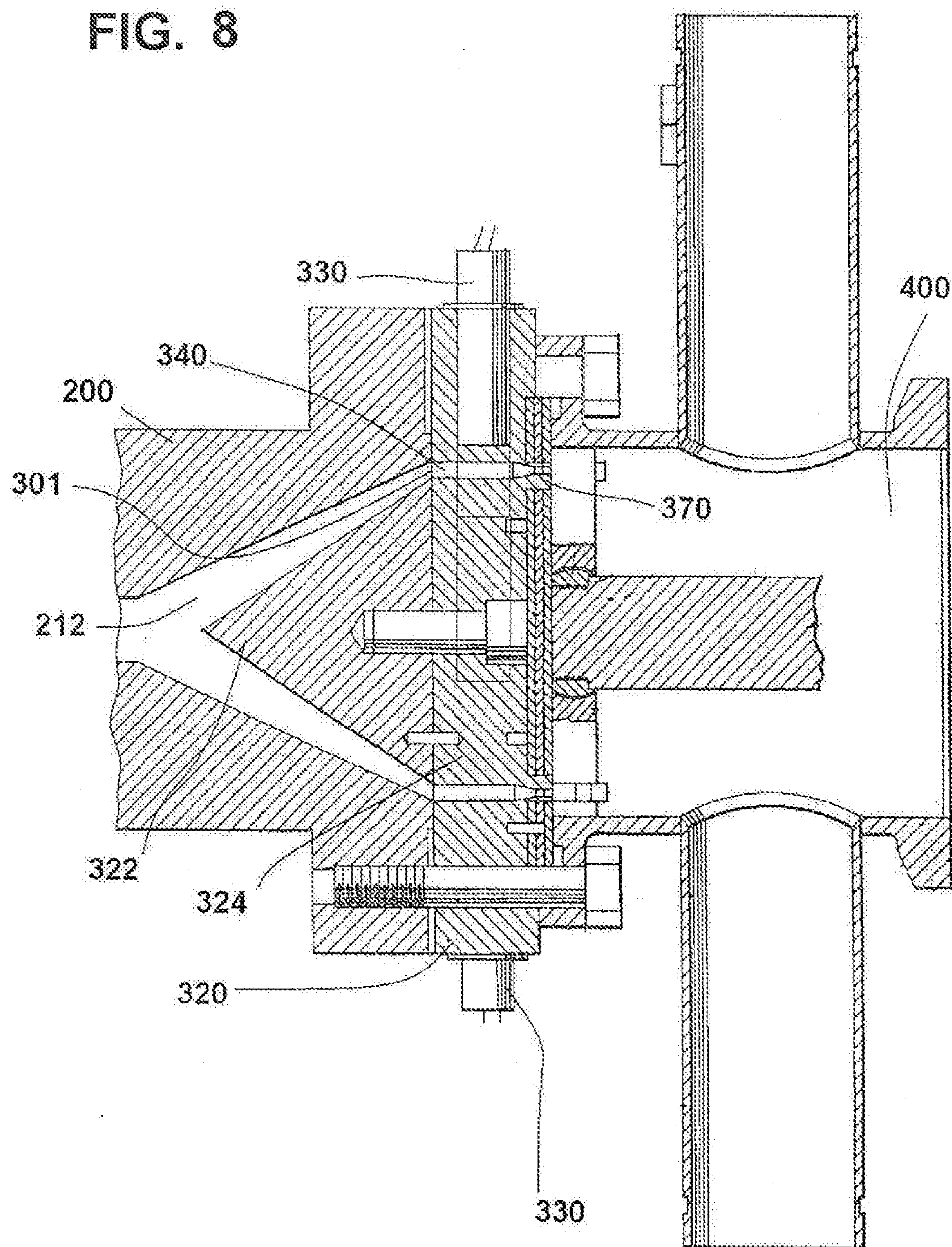
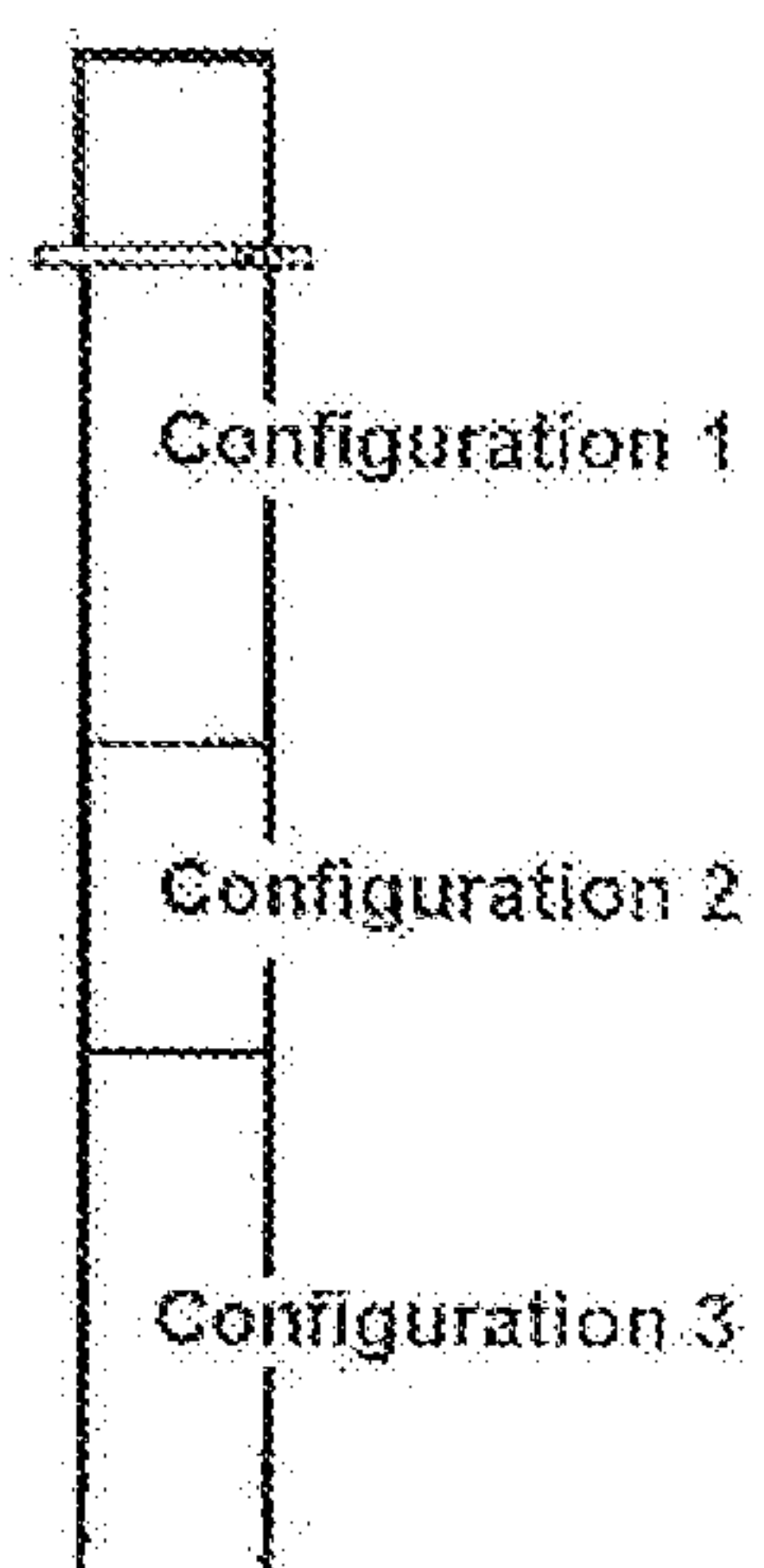
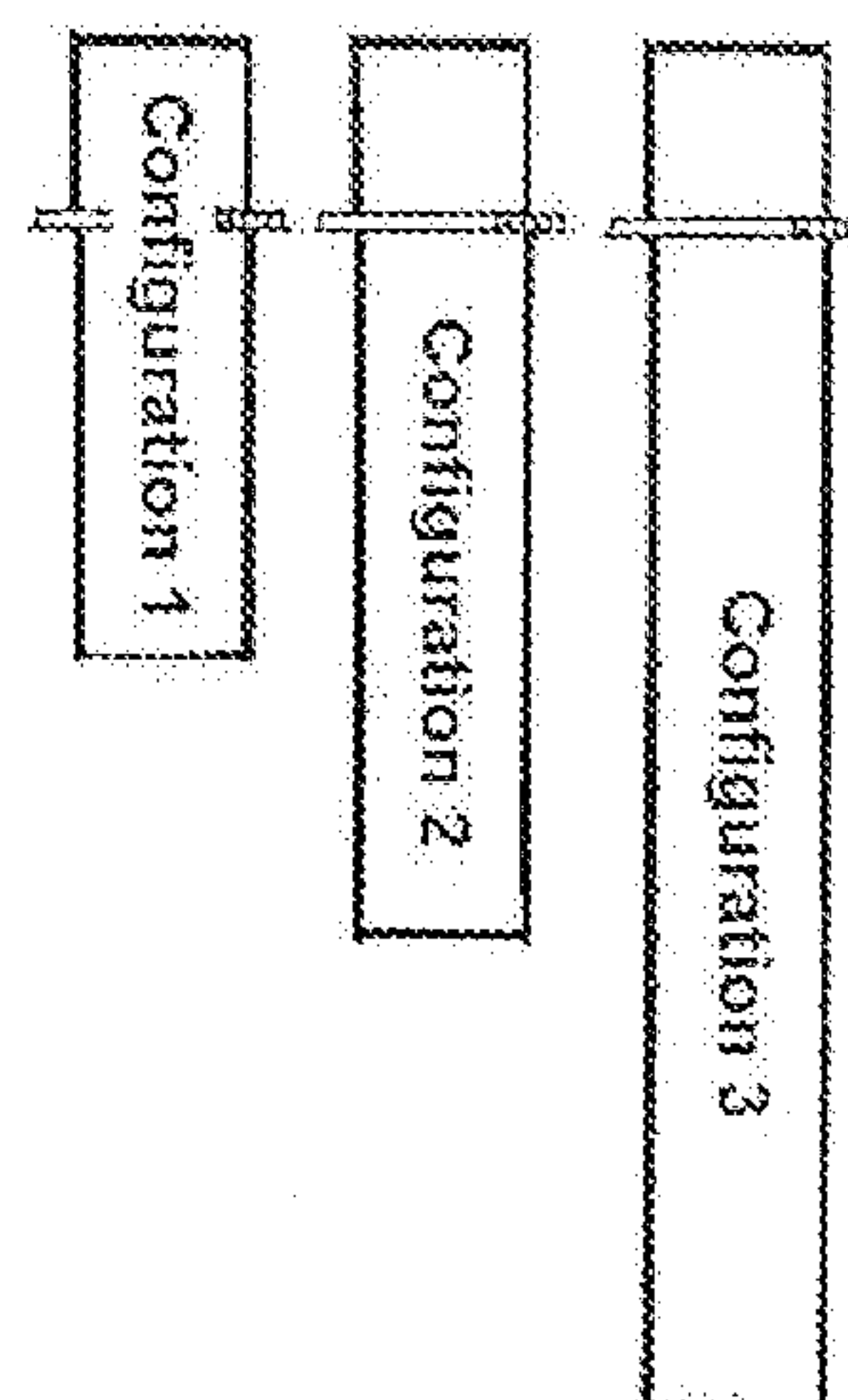


FIG. 8**FIG. 9a****FIG 9b**

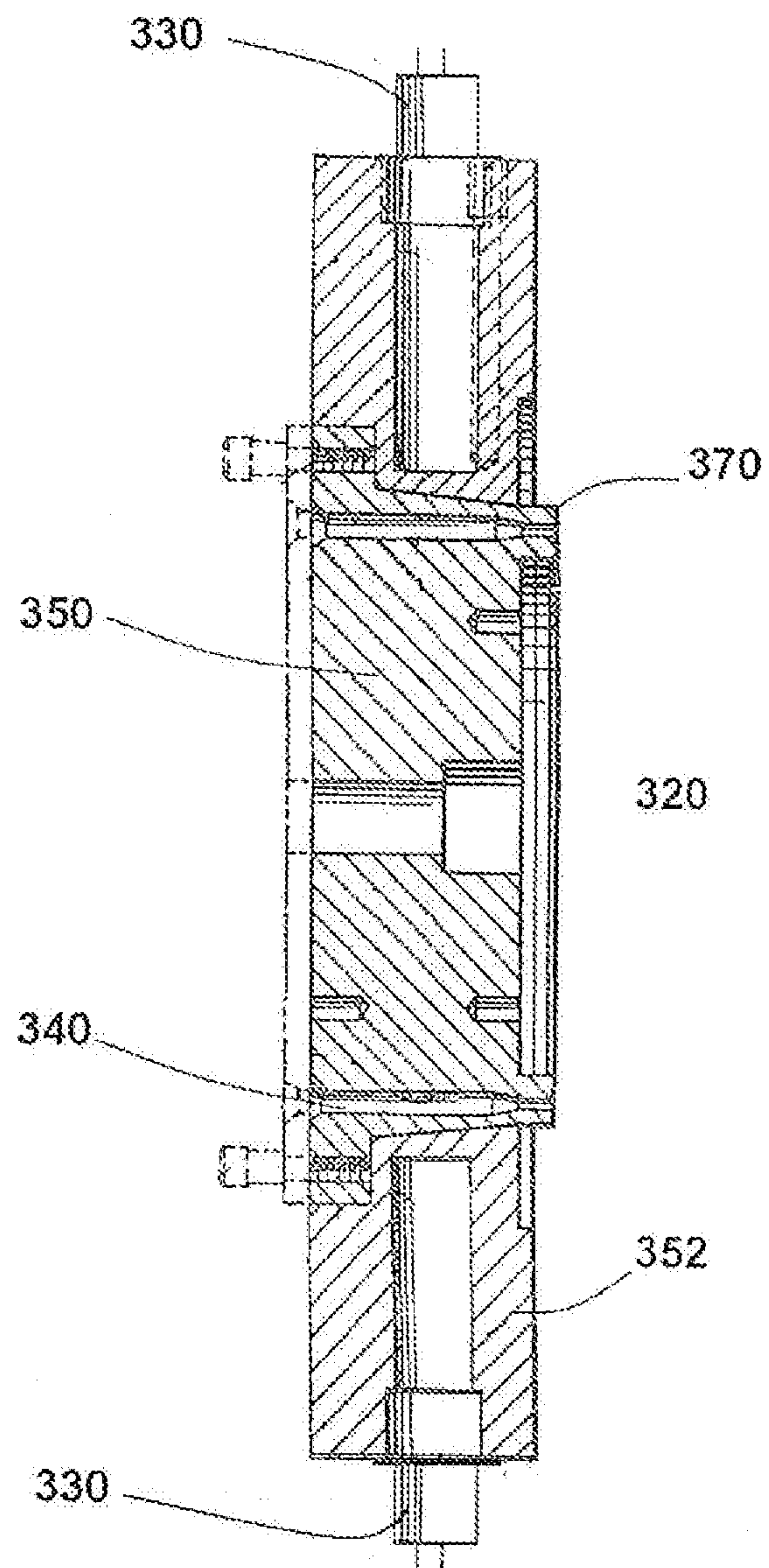
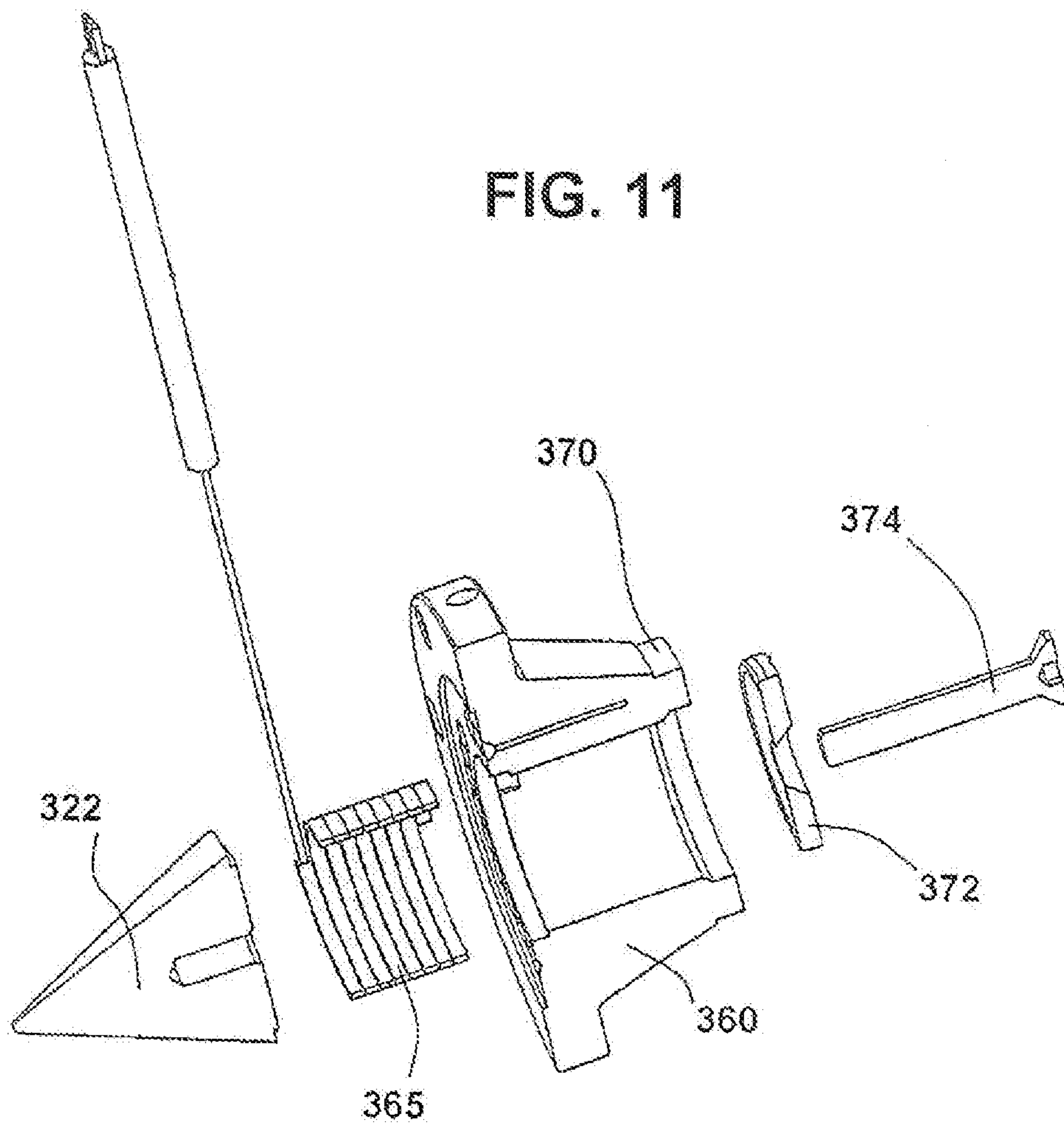


FIG. 10

FIG. 11



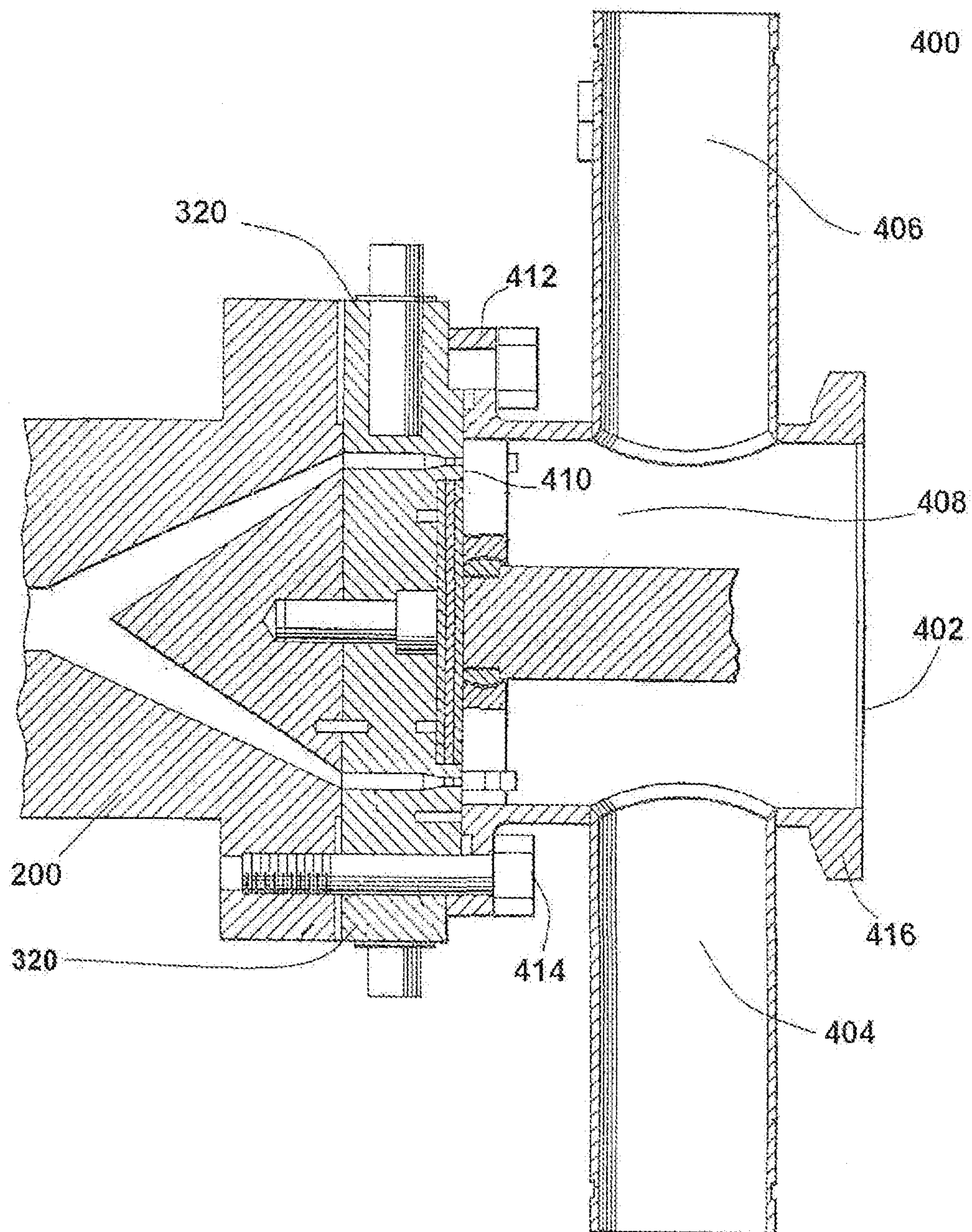
**FIG. 12**

FIG. 14

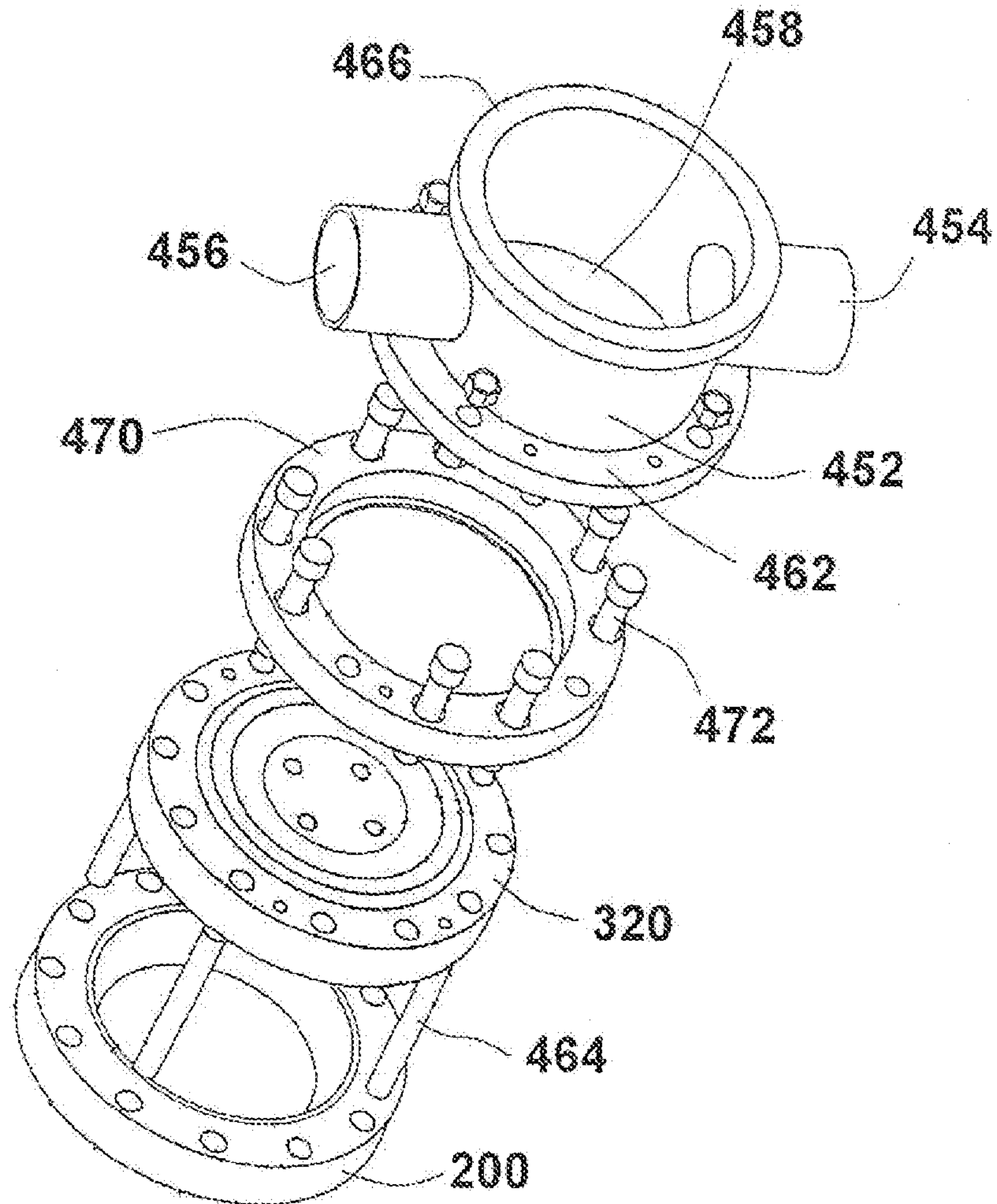


FIG. 15a

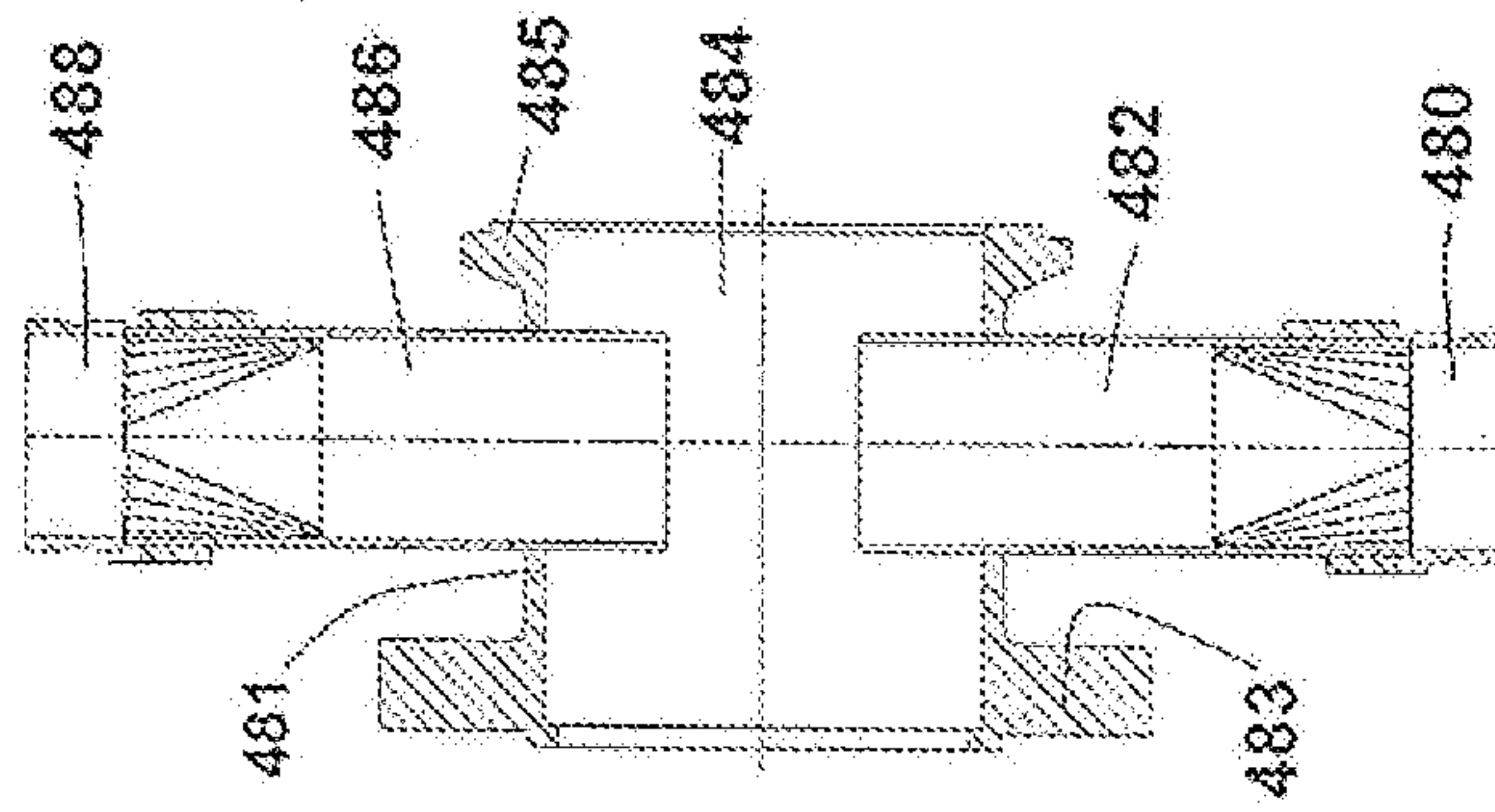
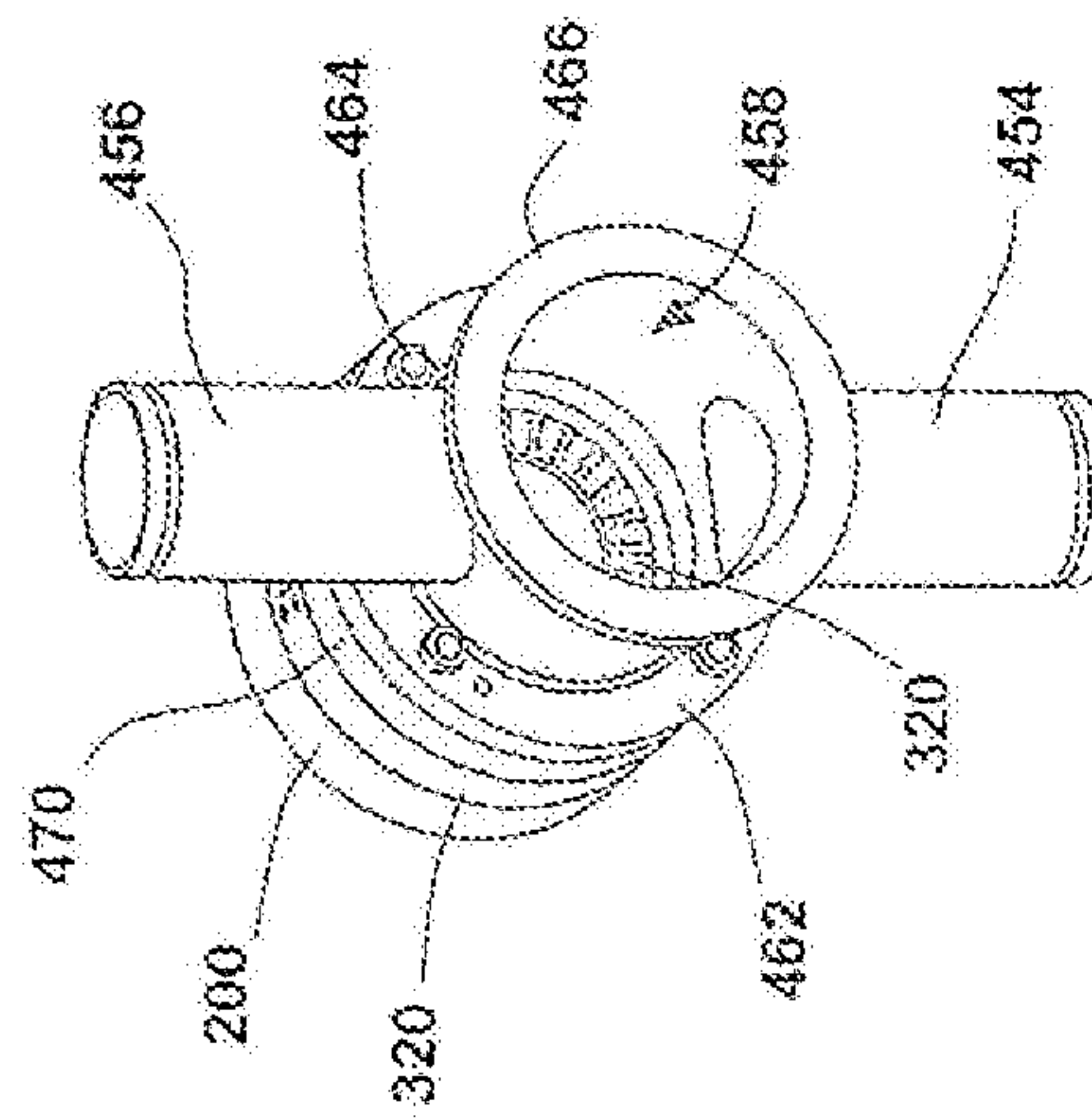


FIG. 15b

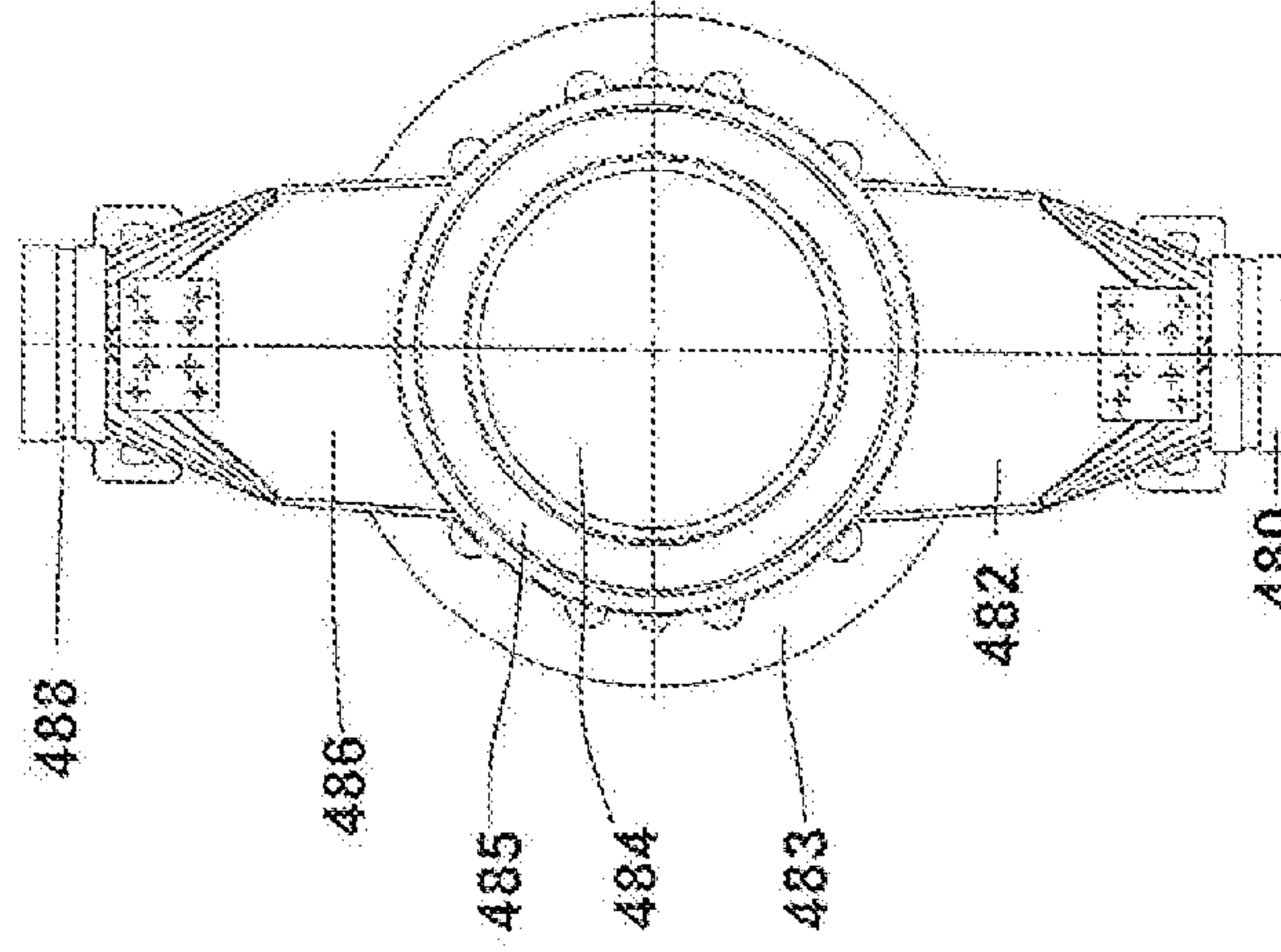


FIG. 15c

FIG. 16

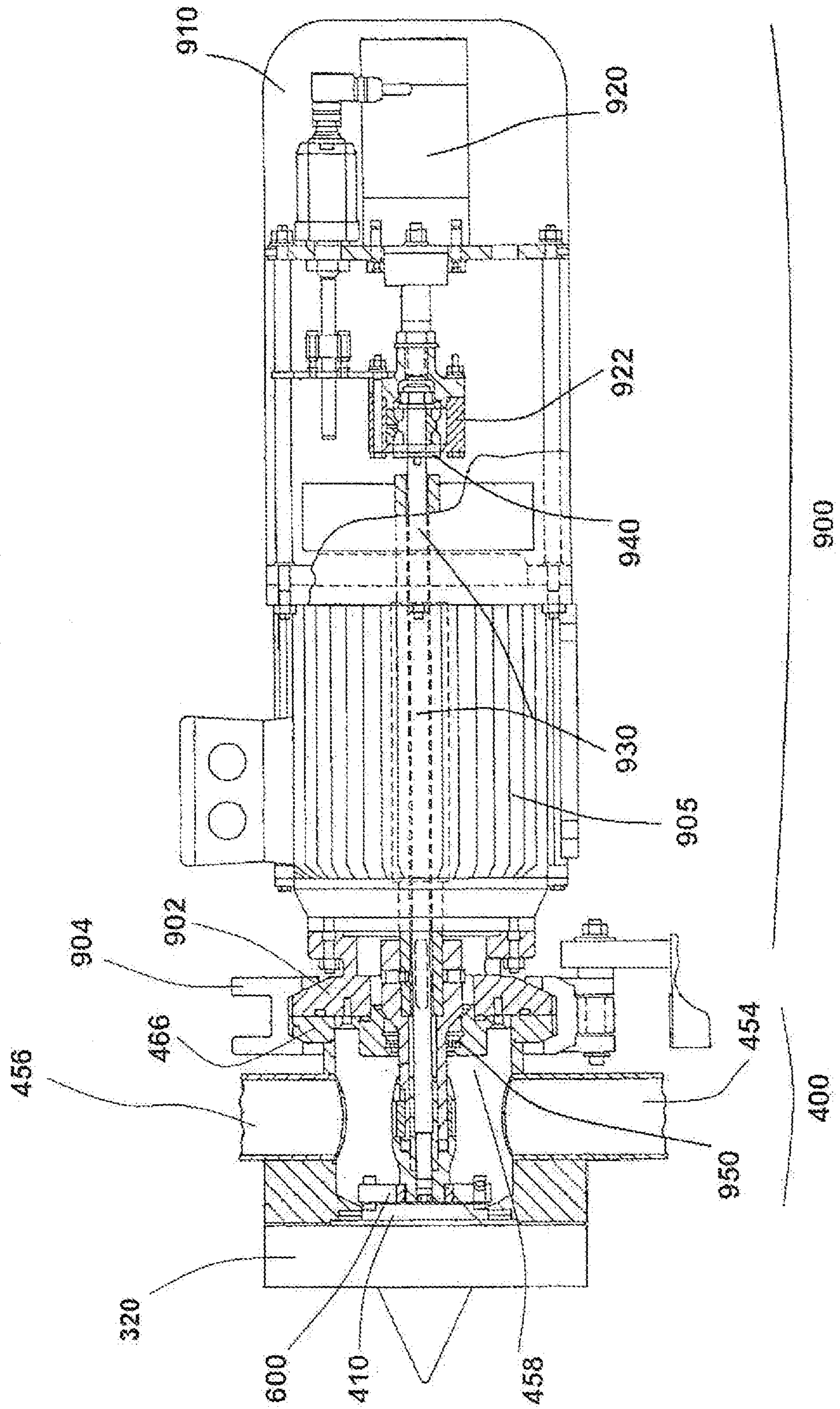
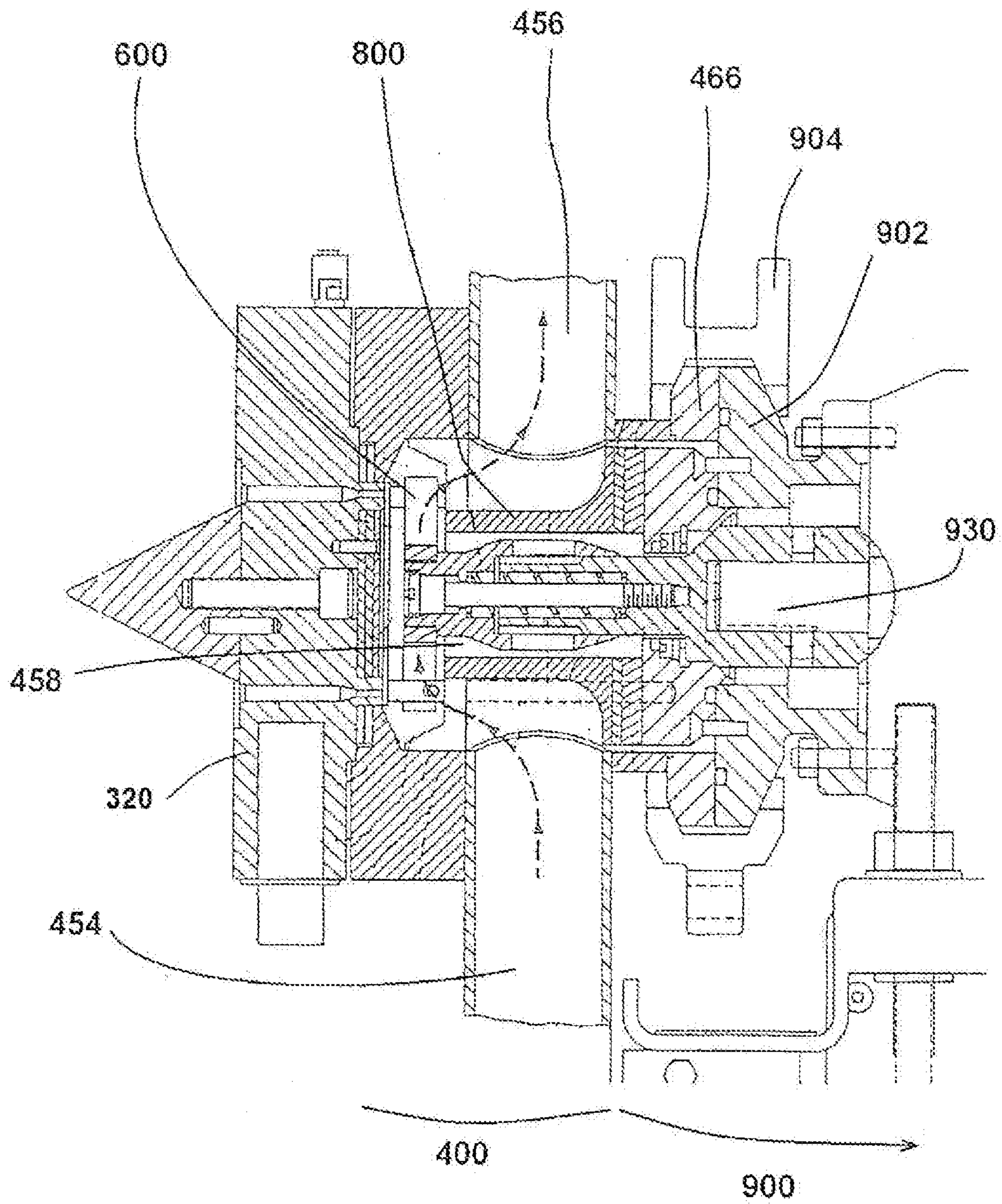
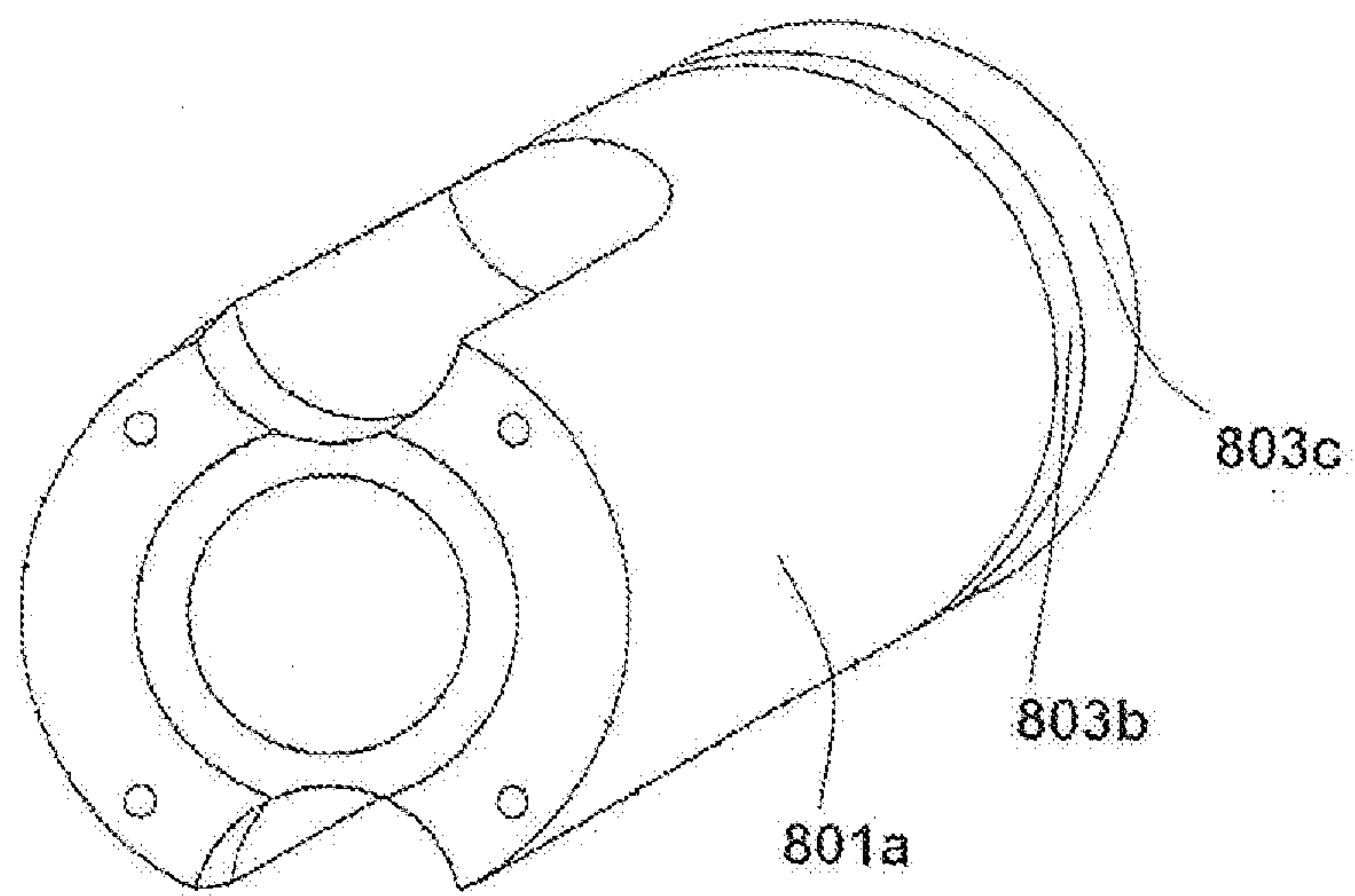
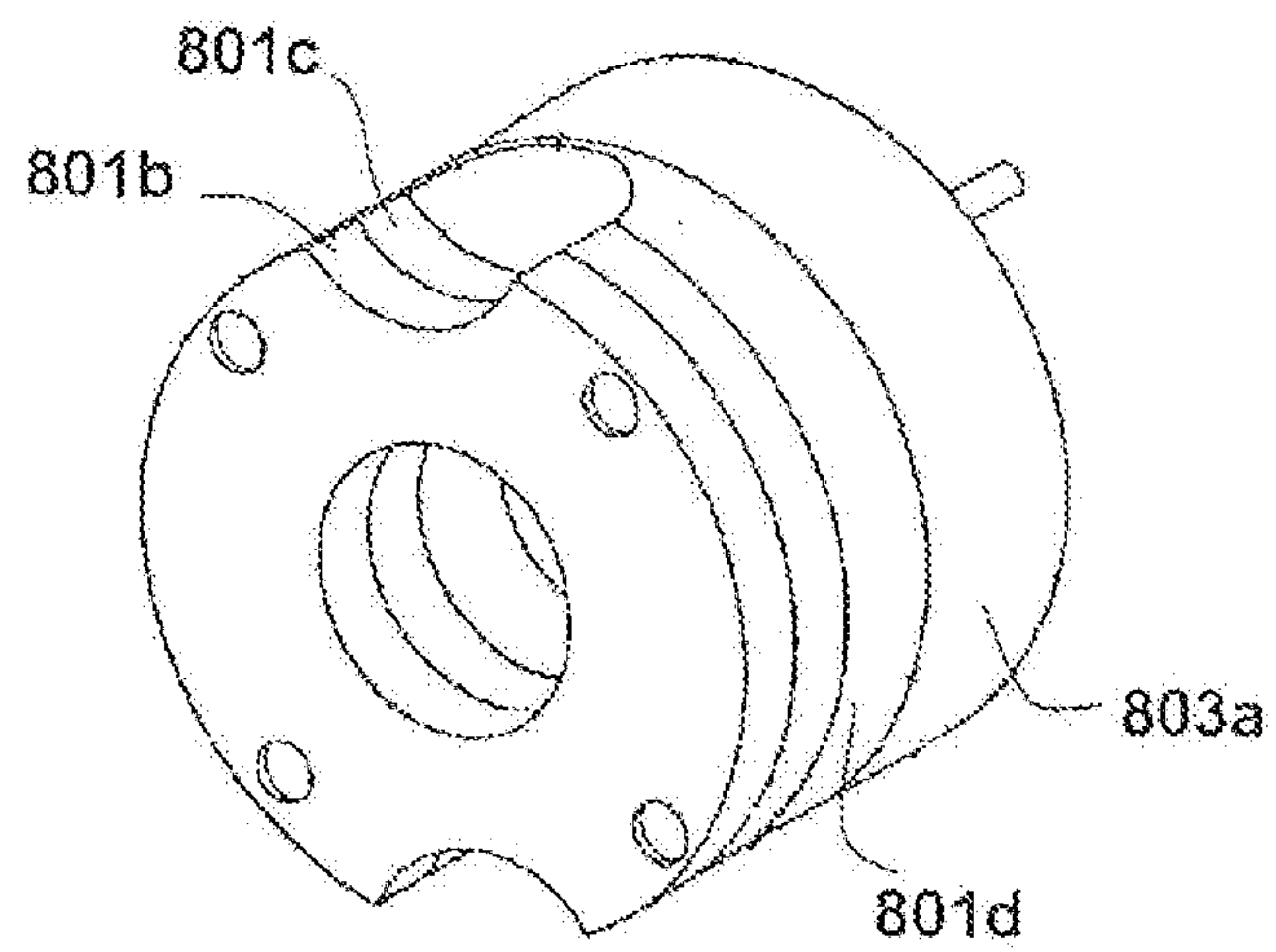
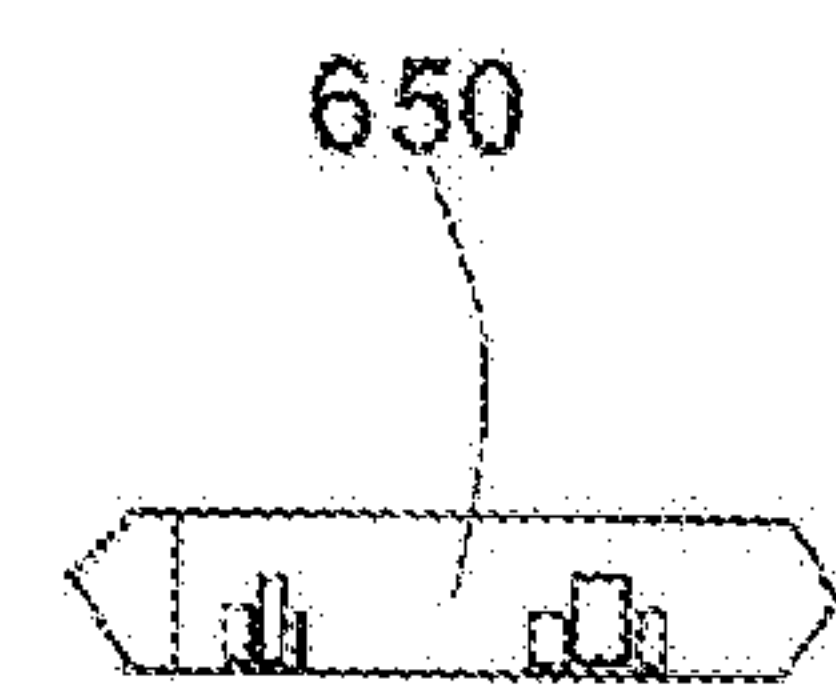
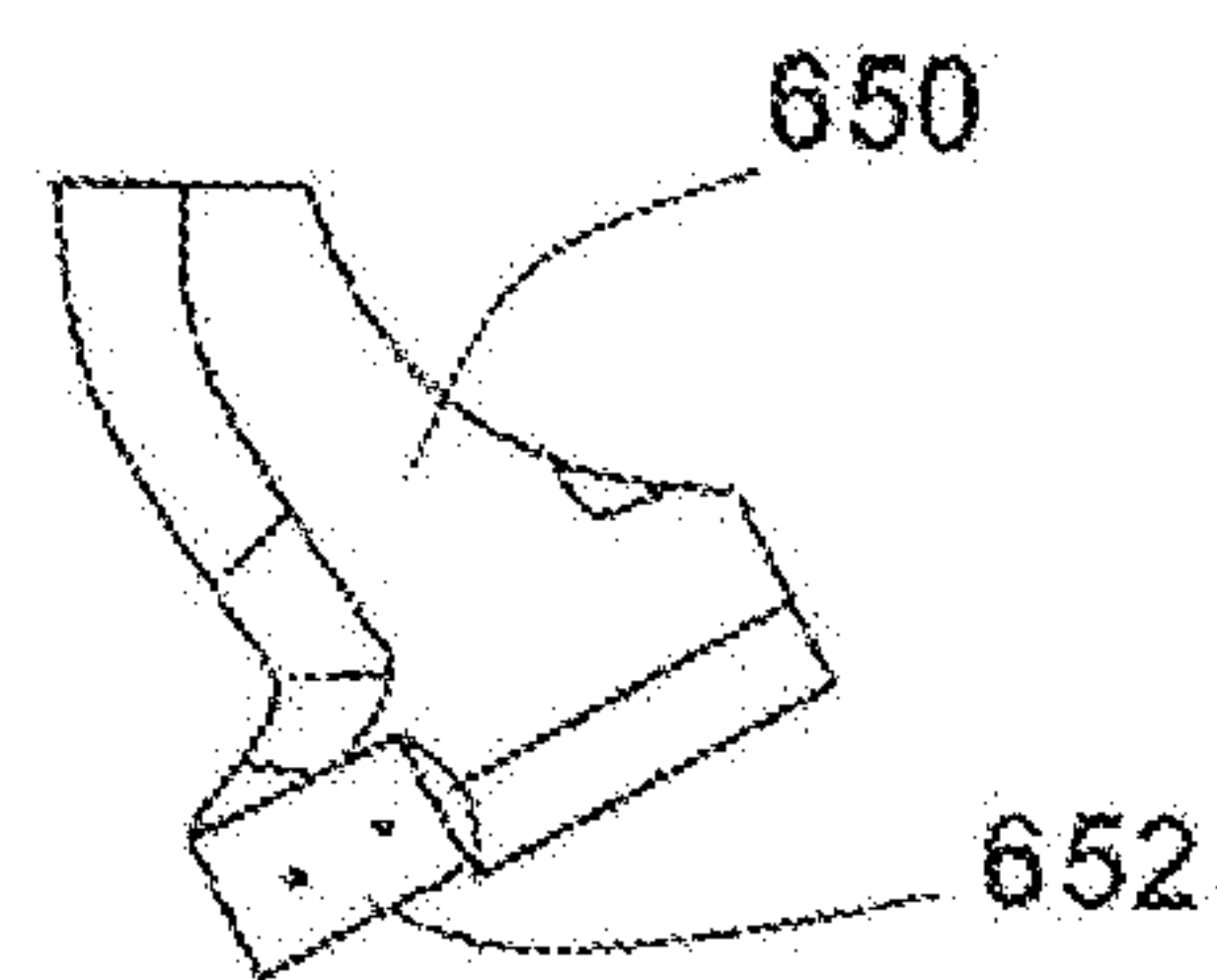
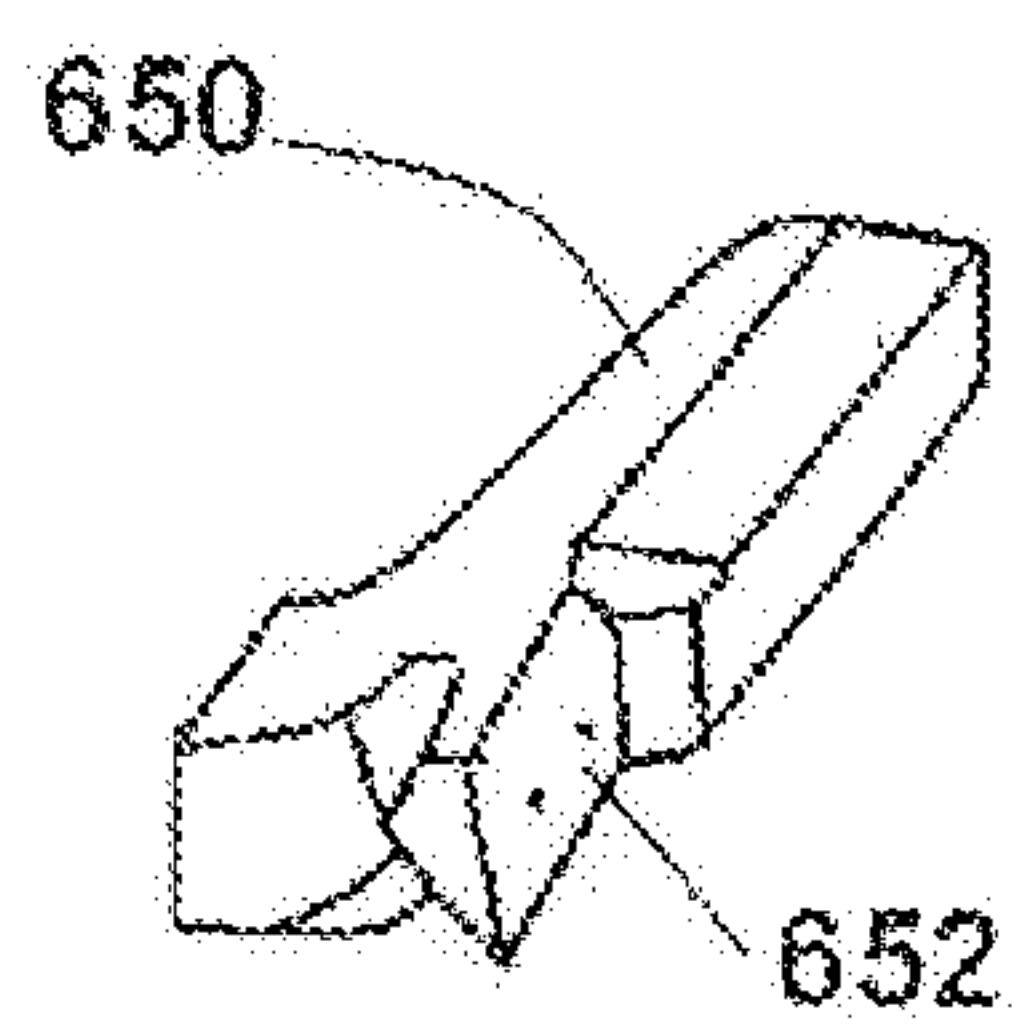
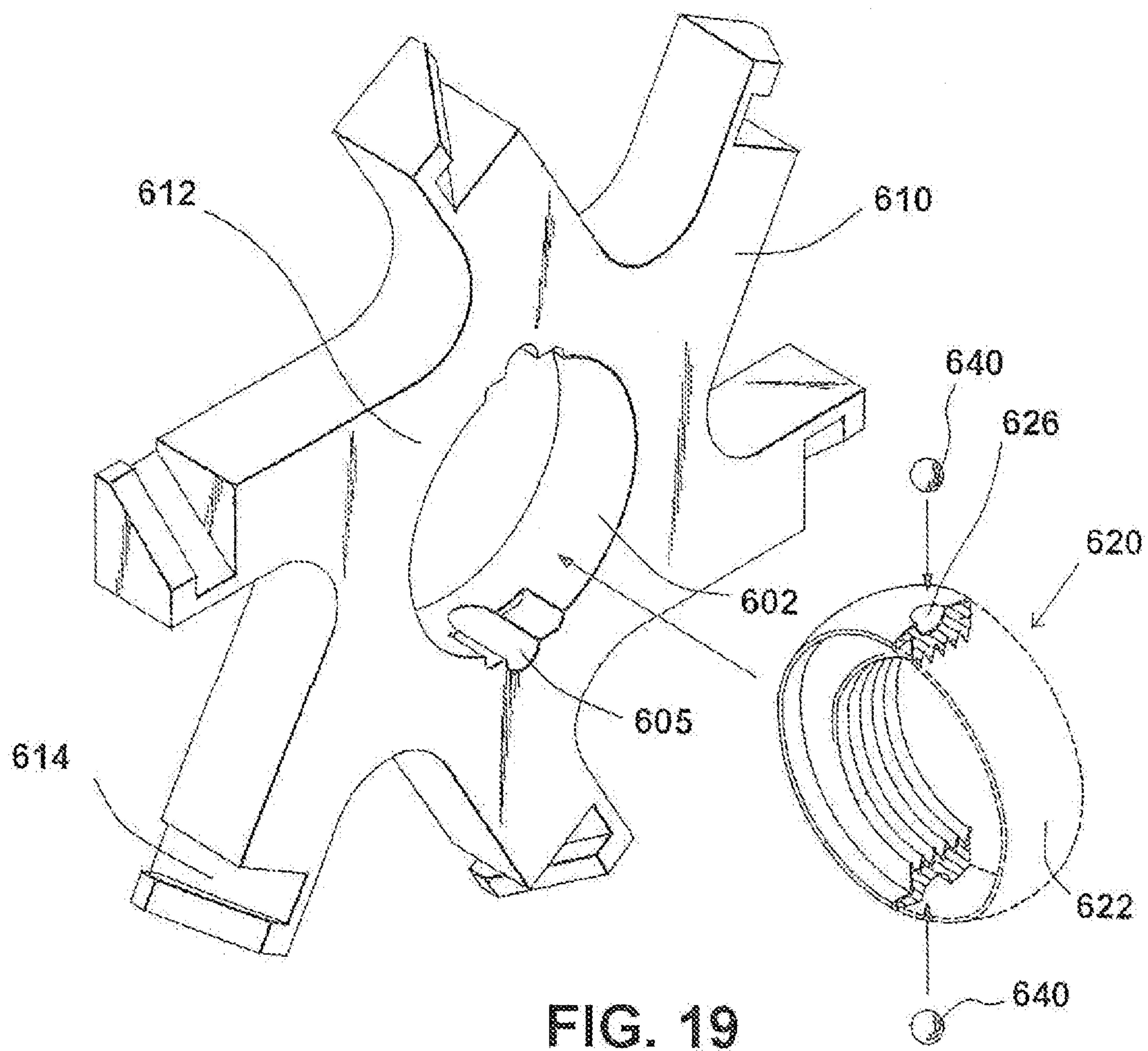


FIG. 17



**FIG. 18a****FIG. 18b**



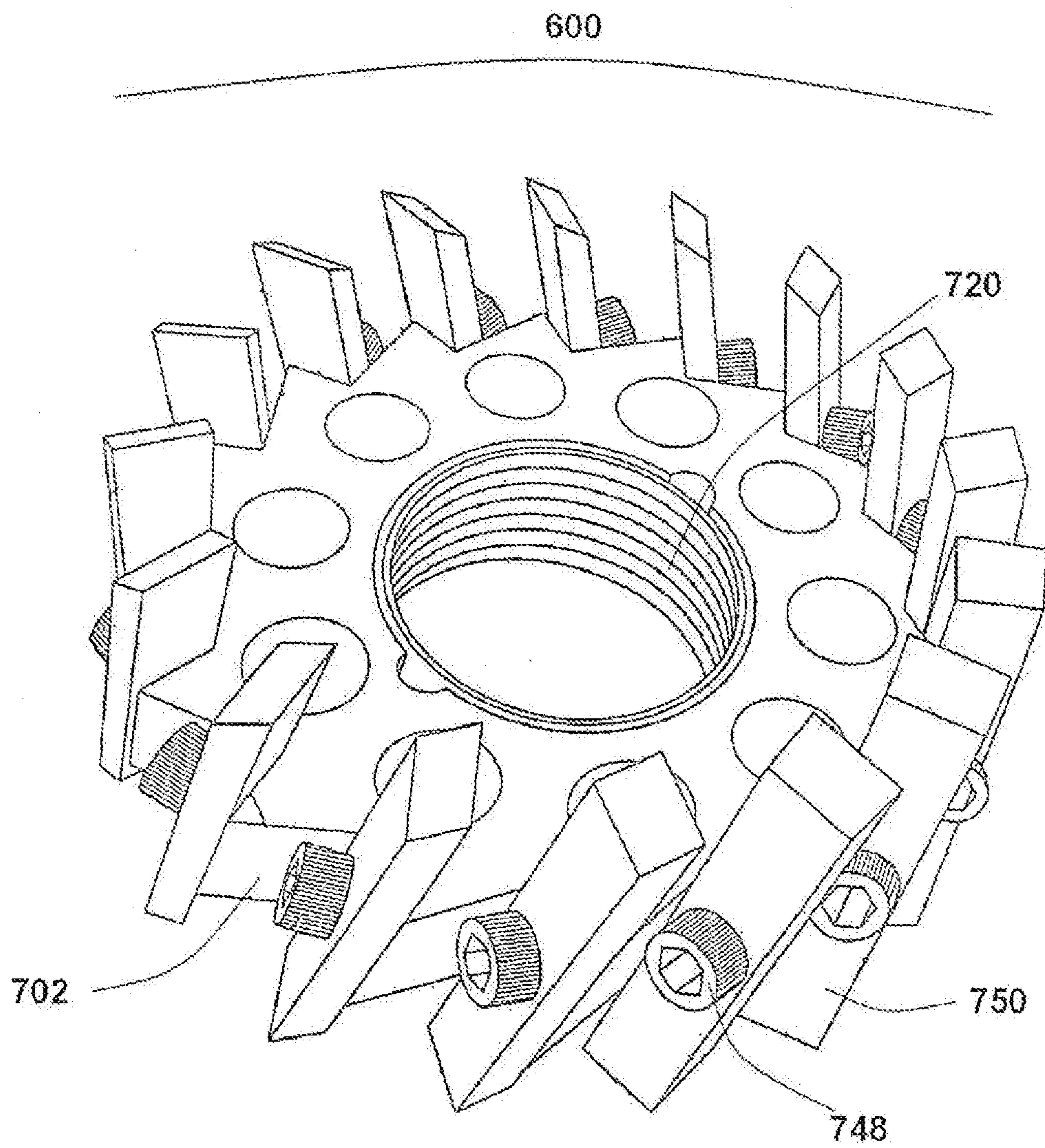


FIG. 21

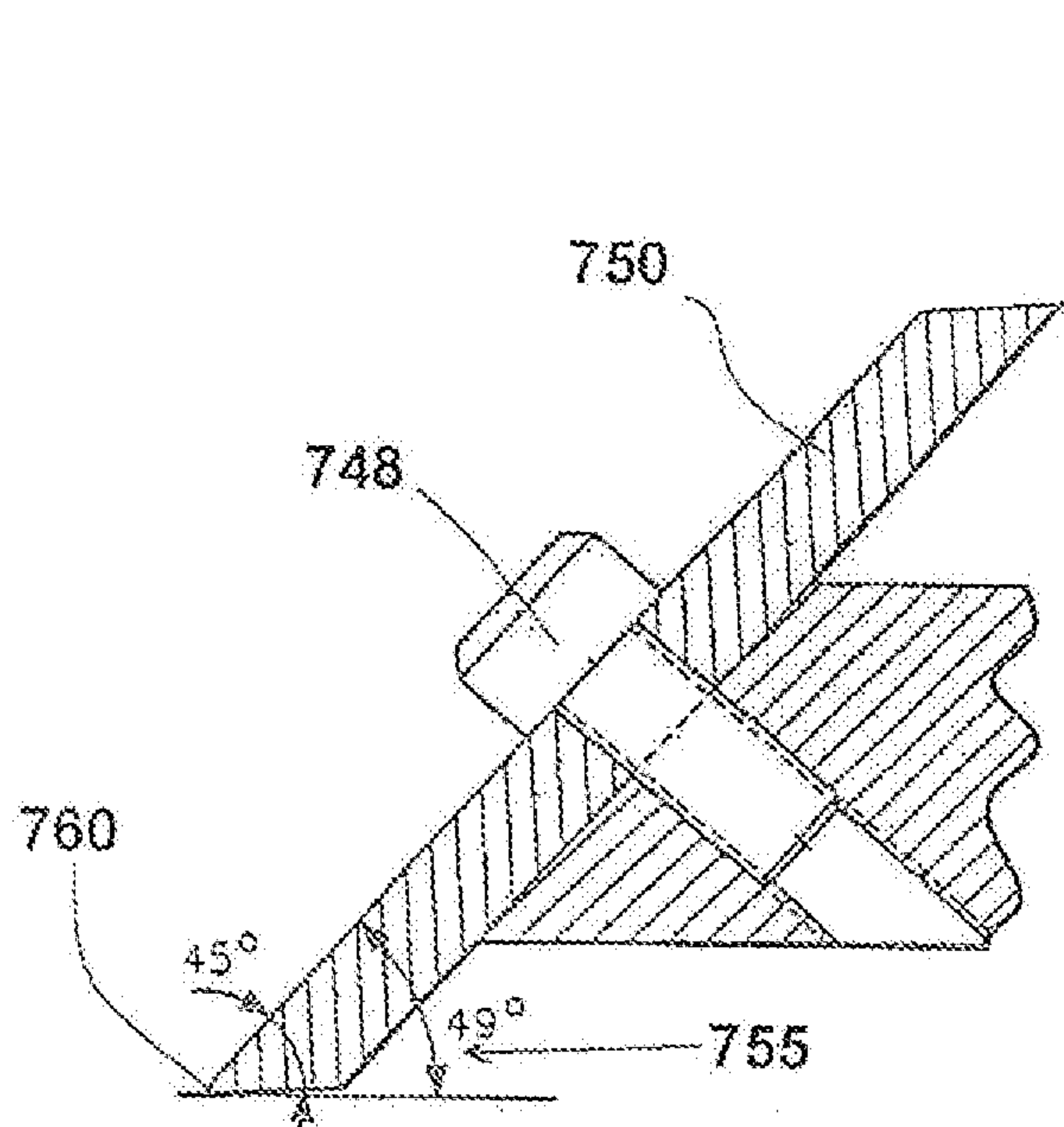


FIG. 22a

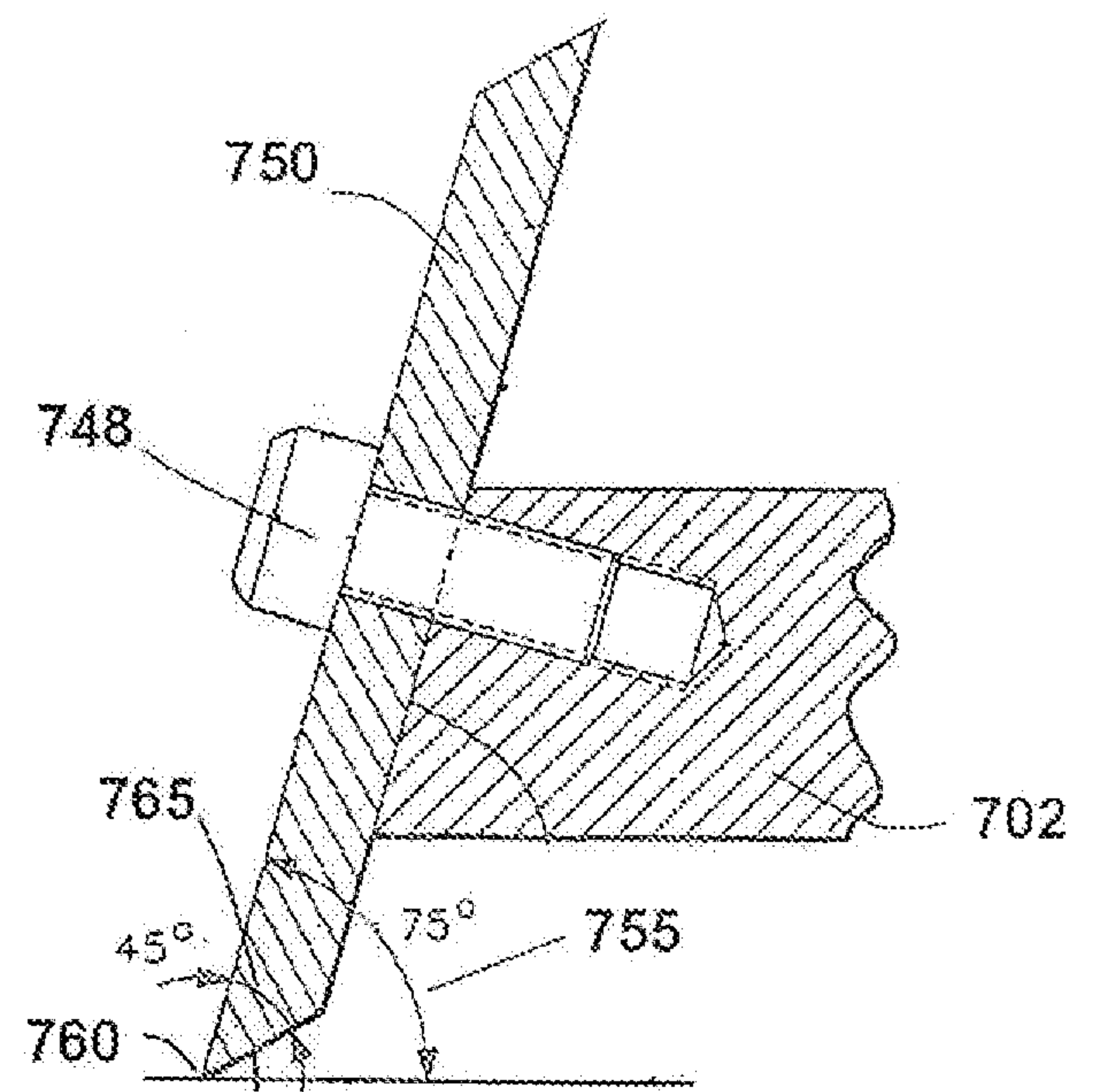


FIG. 22b

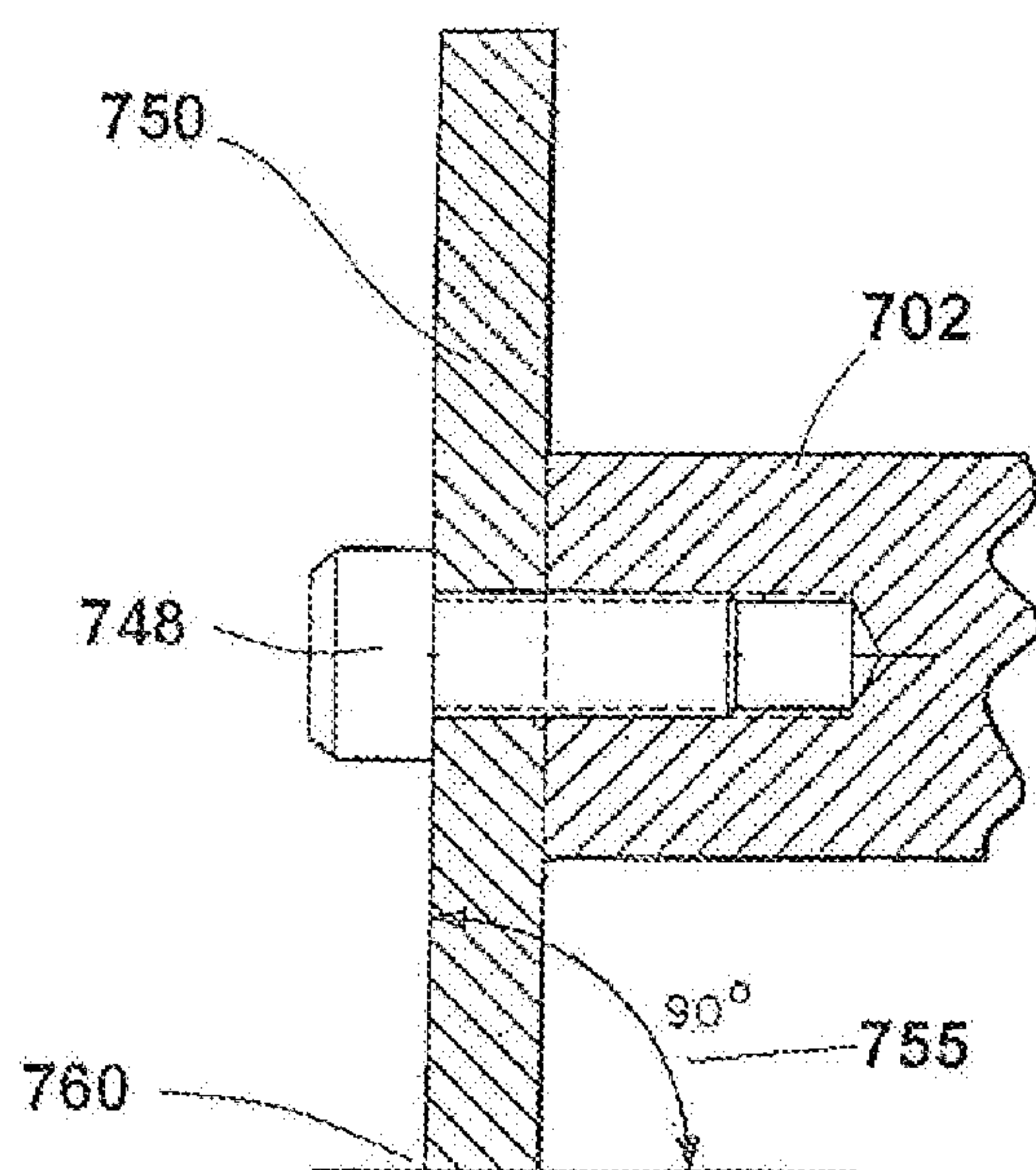


FIG. 22c

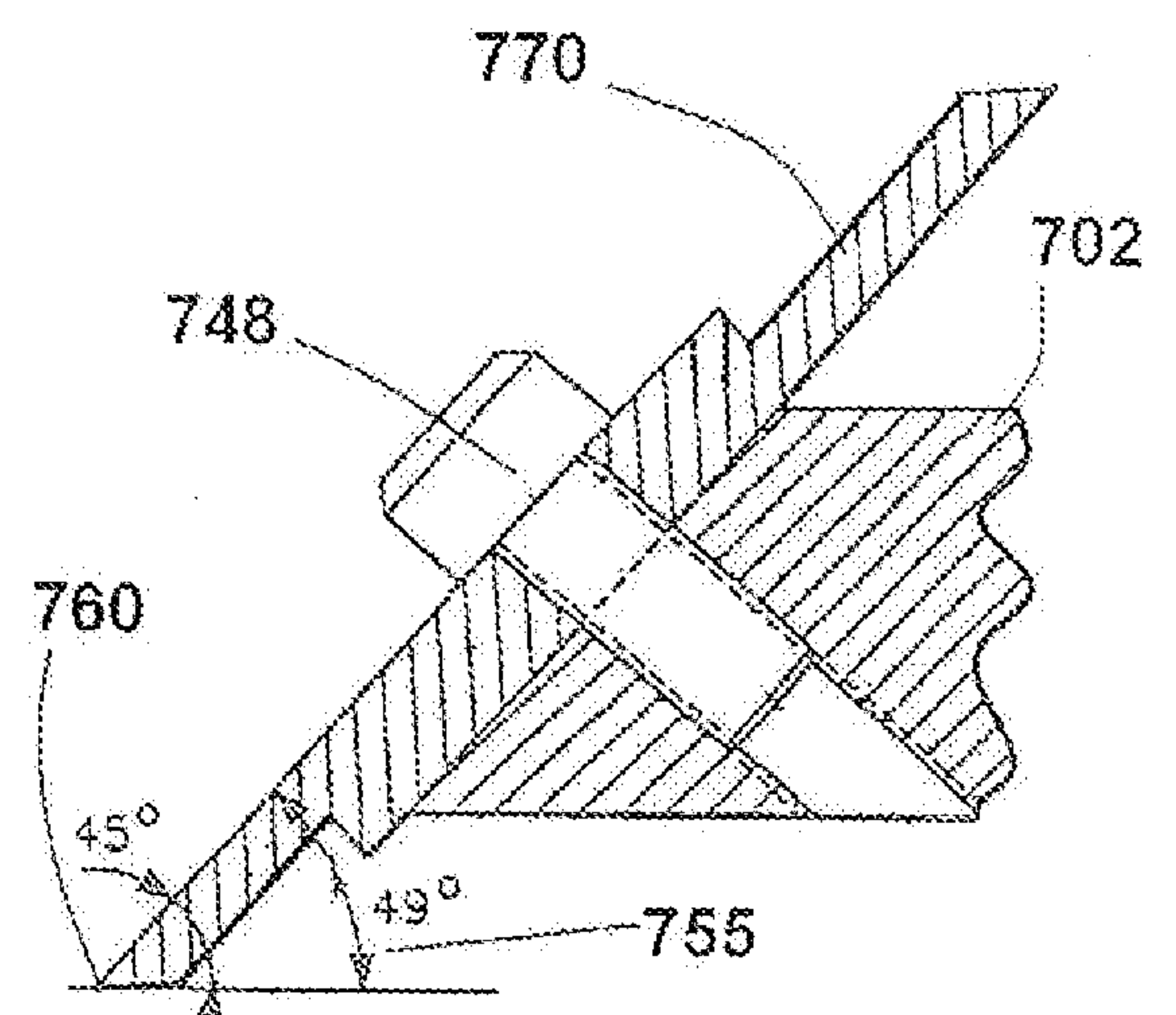


FIG. 22d

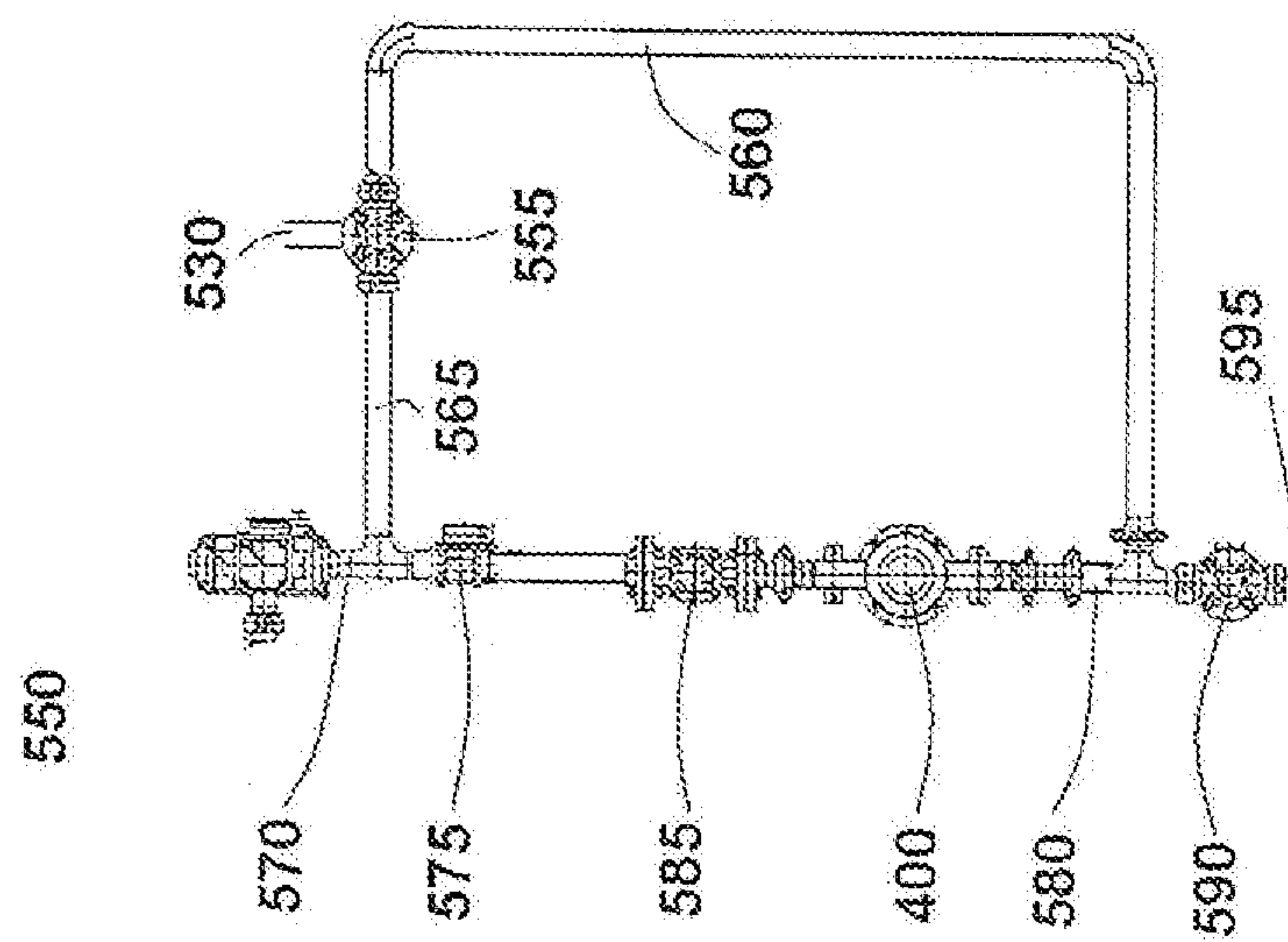


FIG. 23

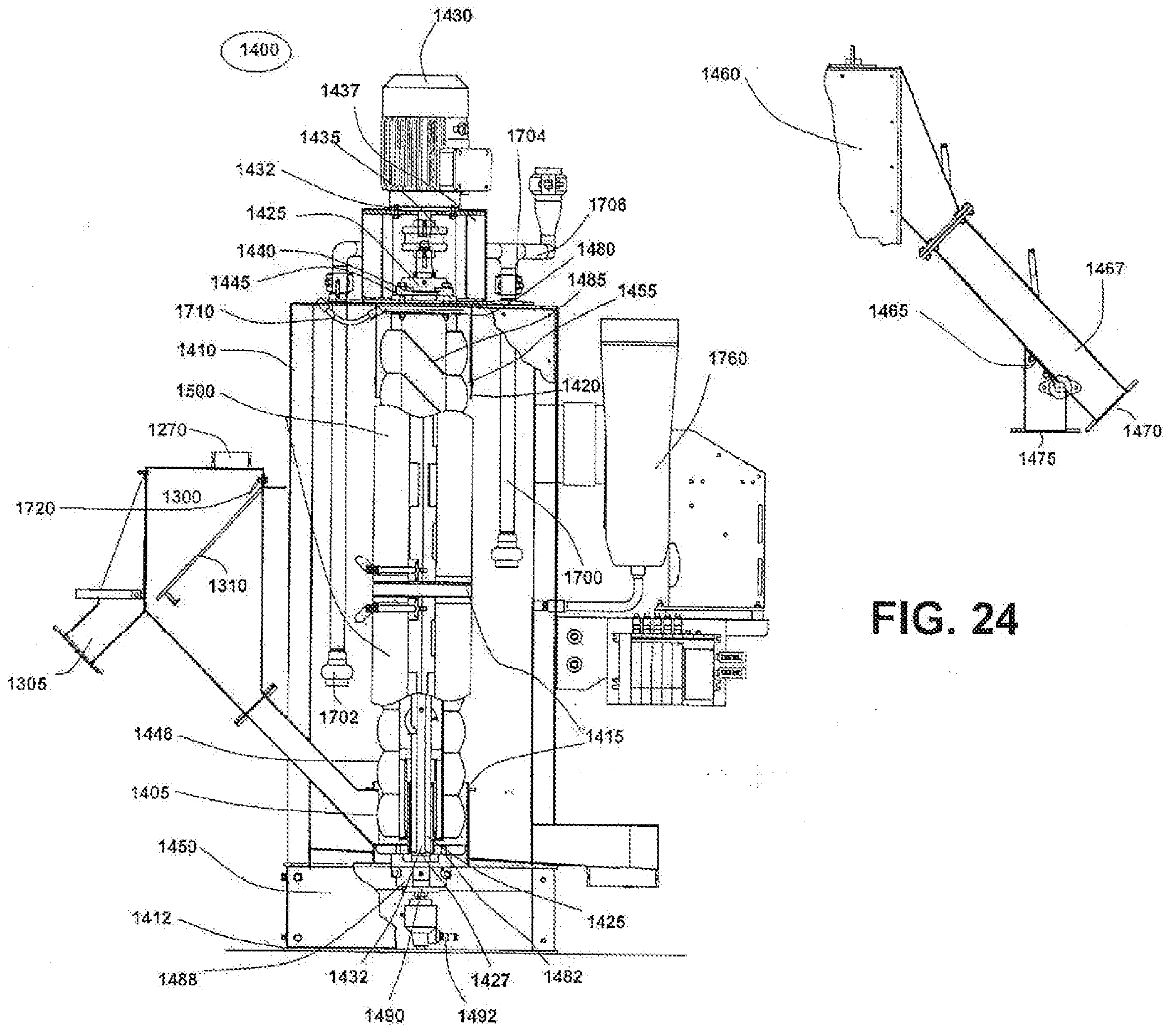


FIG. 24

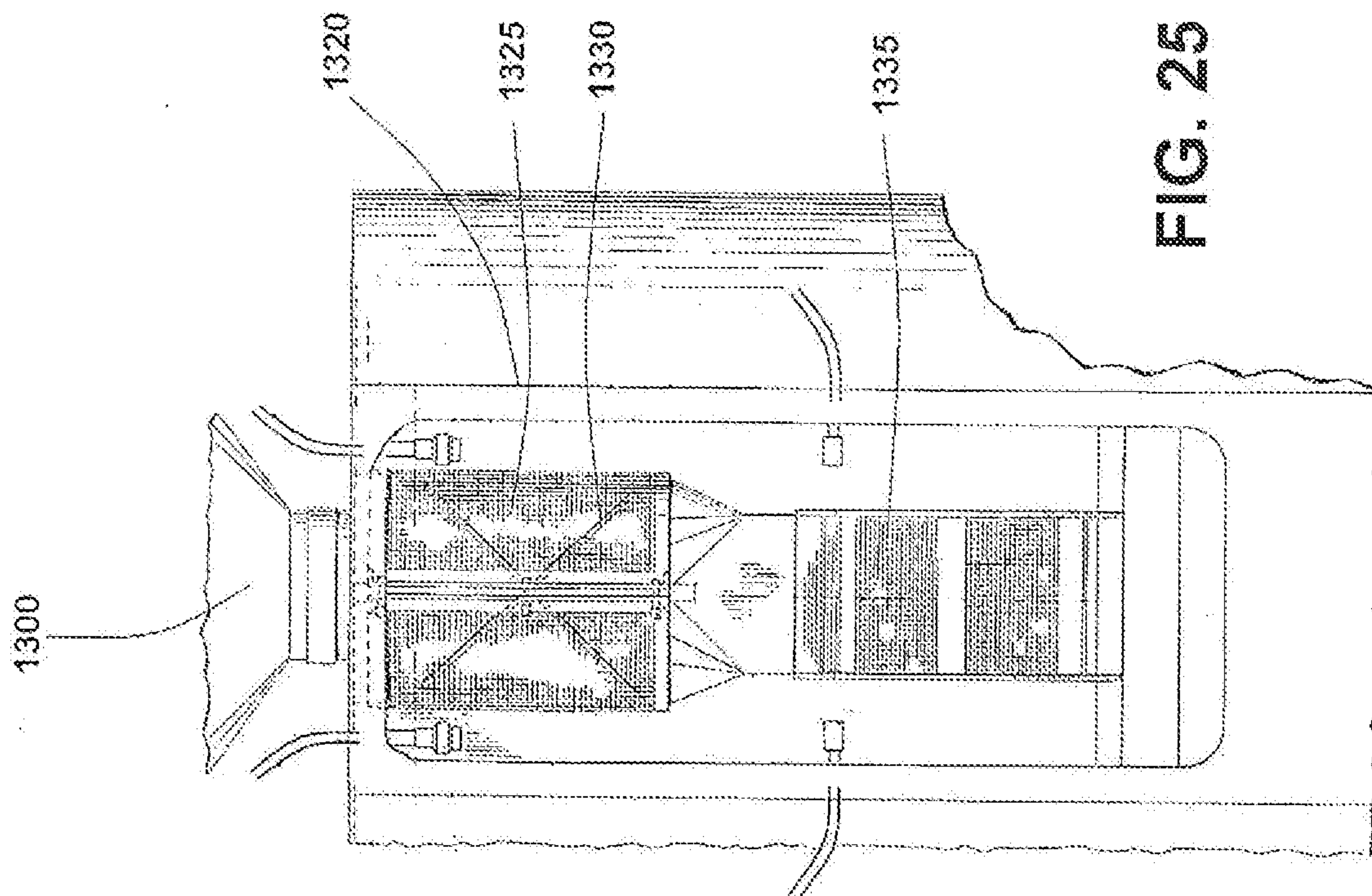


FIG. 25

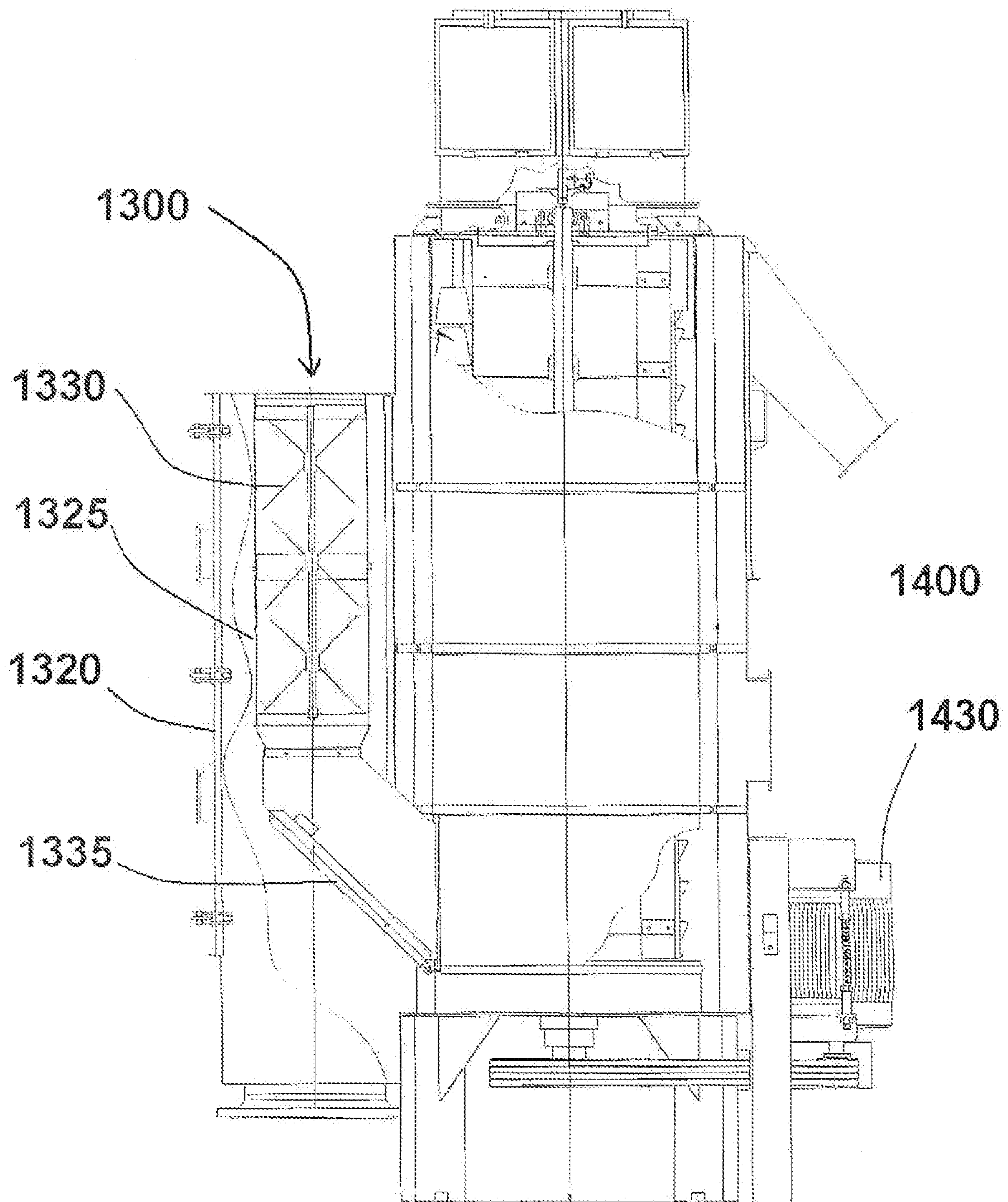


FIG. 26

FIG. 27

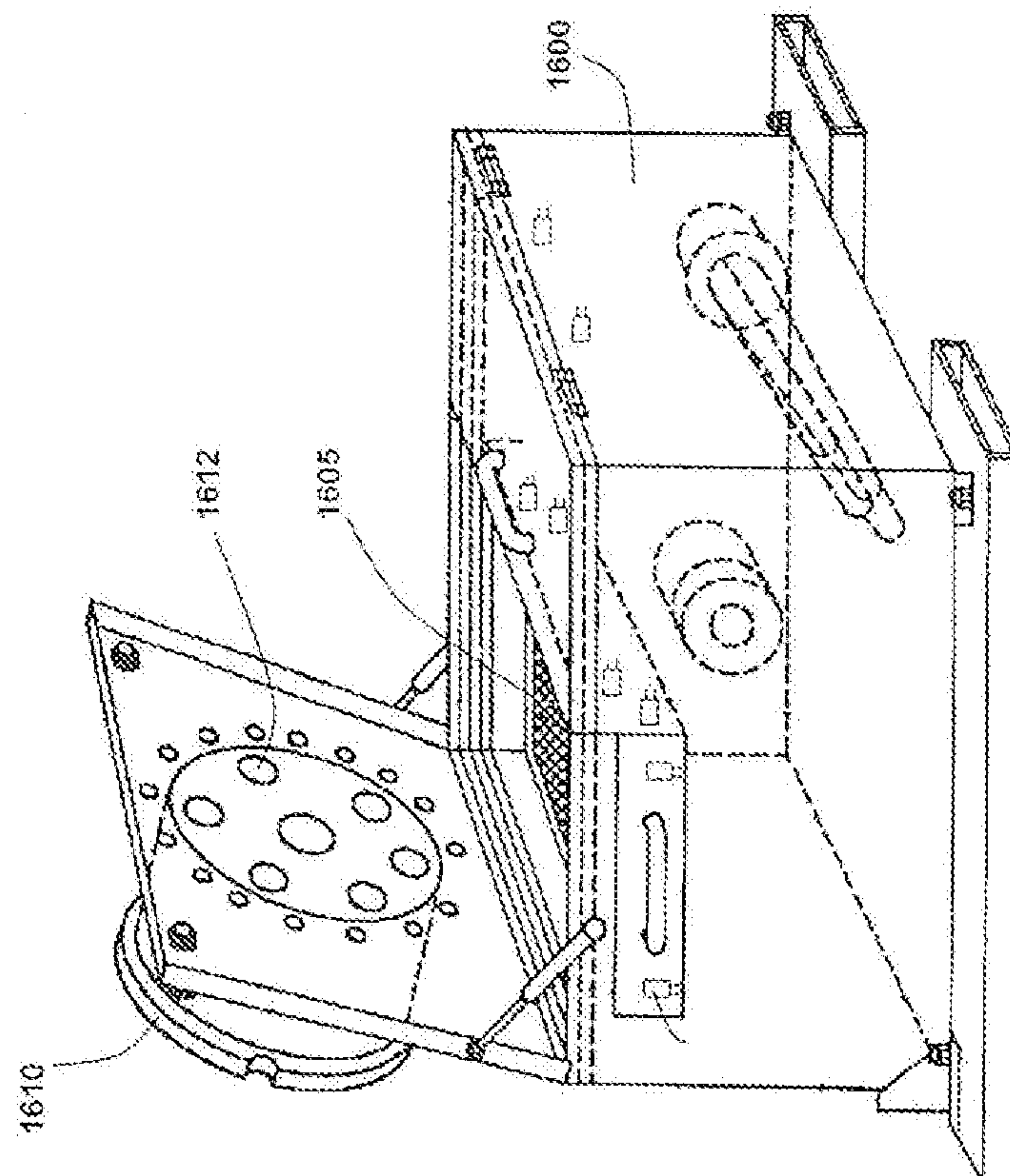


FIG. 28

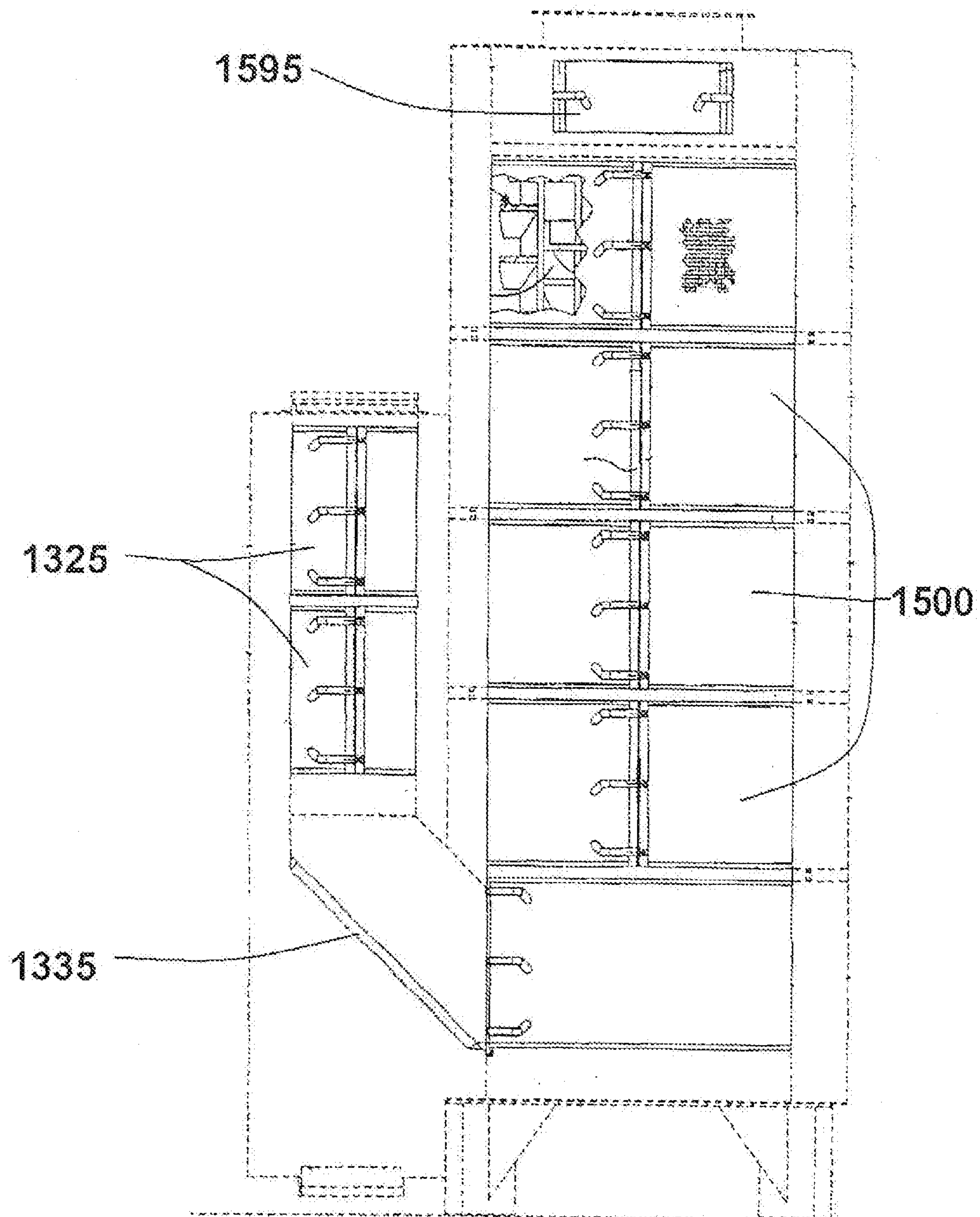
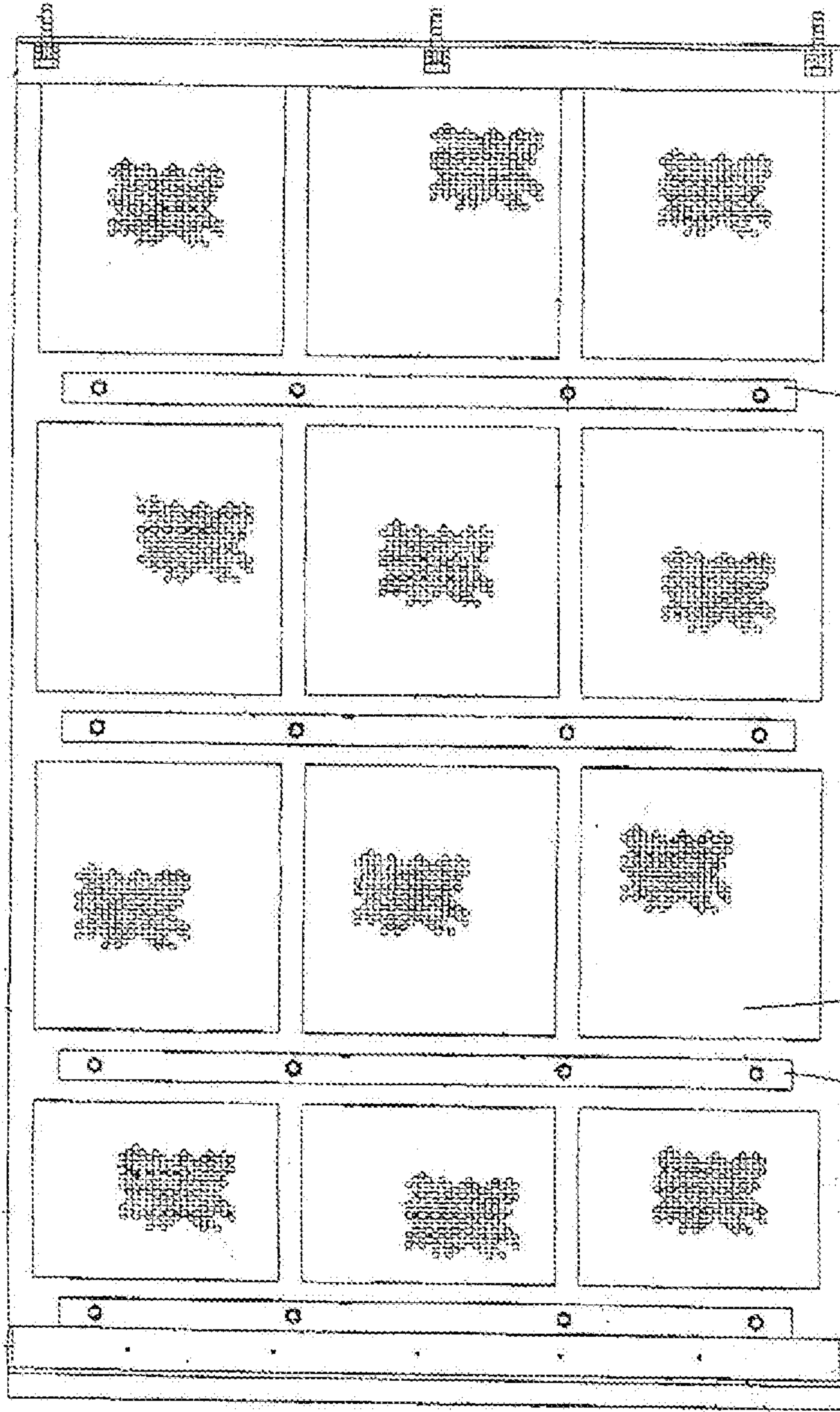


FIG. 29



1550

1500

1550

FIG. 30



FIG. 31

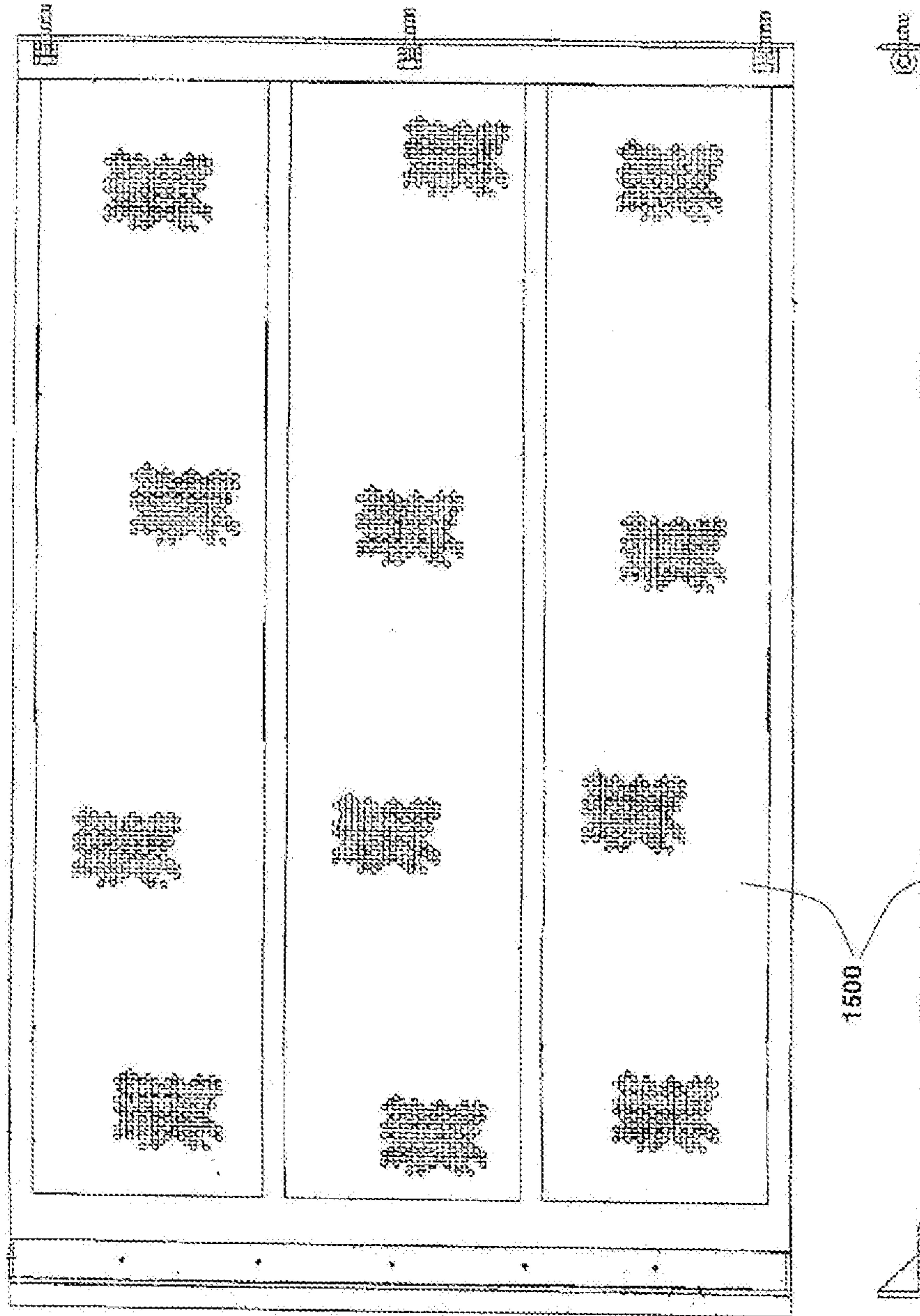


FIG. 32

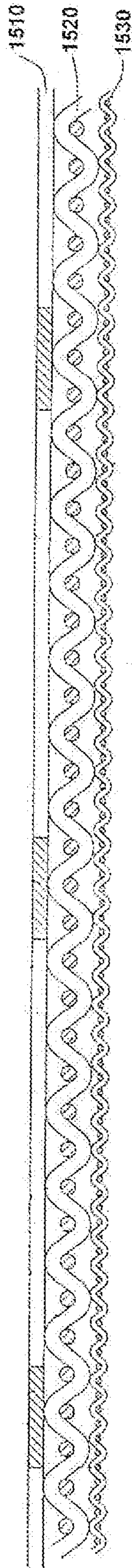
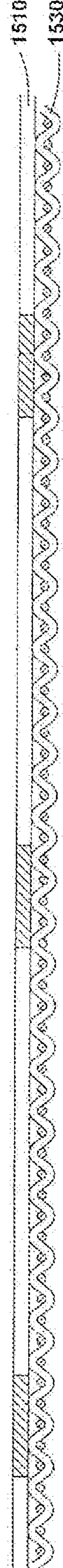
**FIG. 33****FIG. 34**

FIG. 35

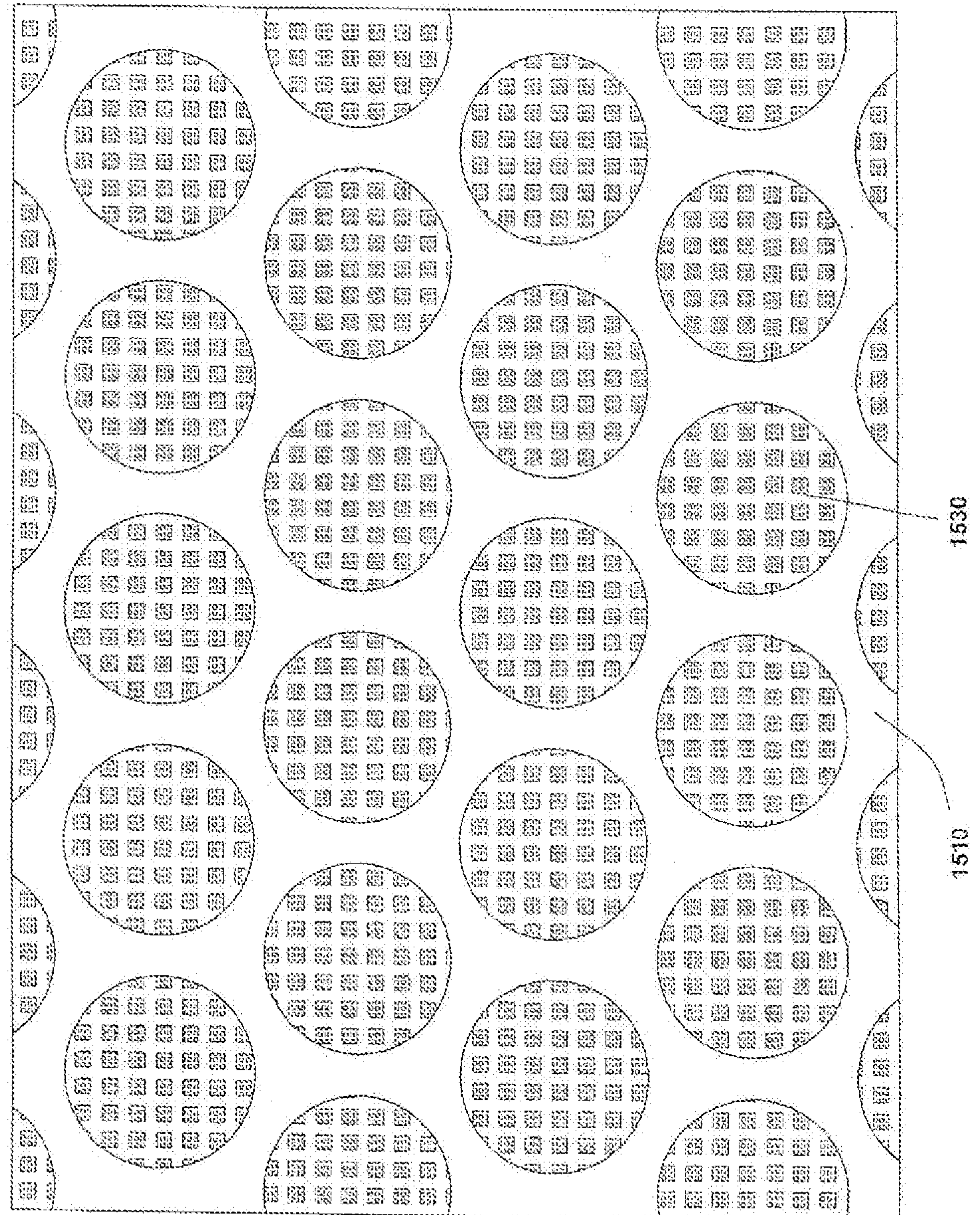


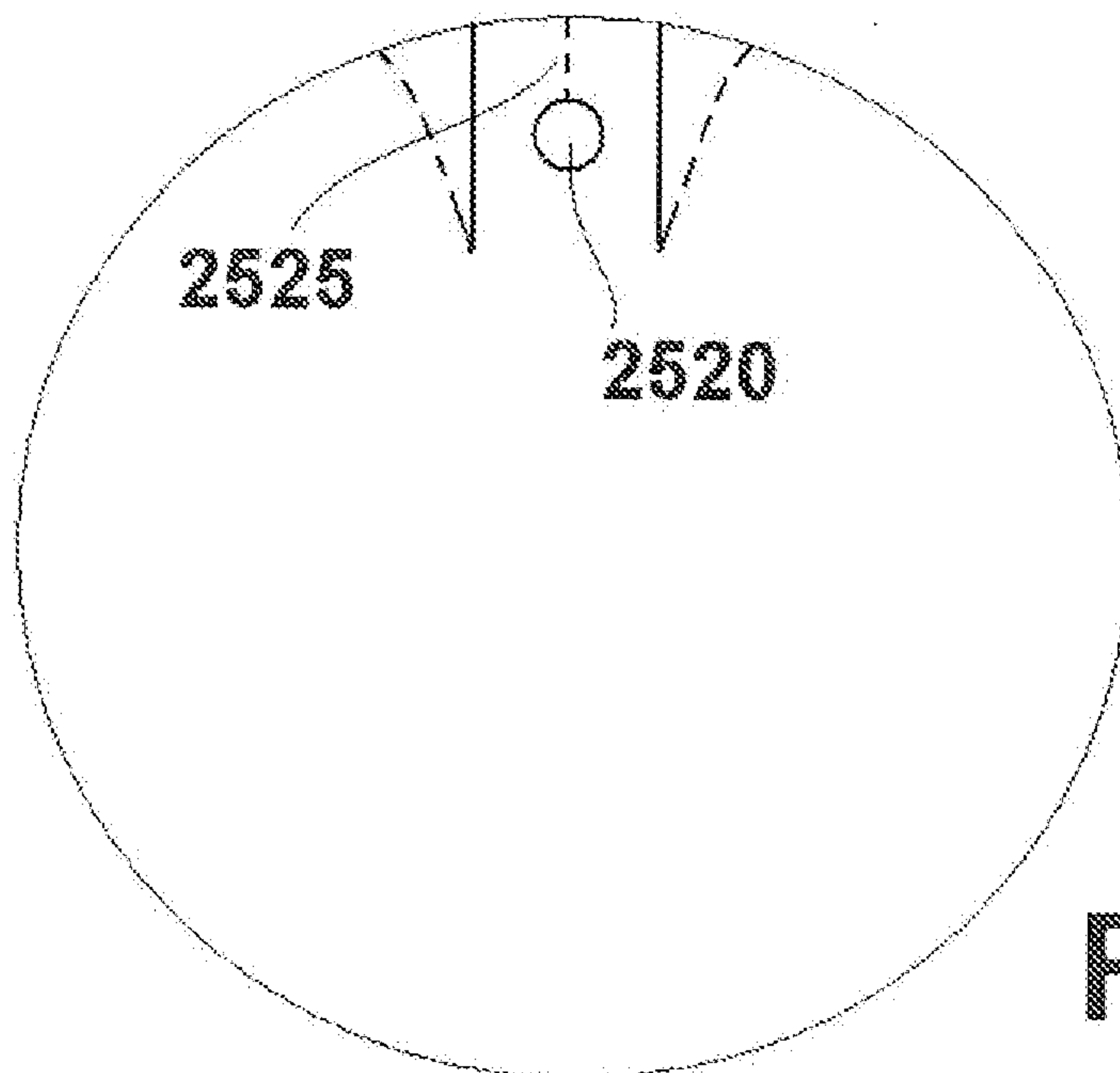
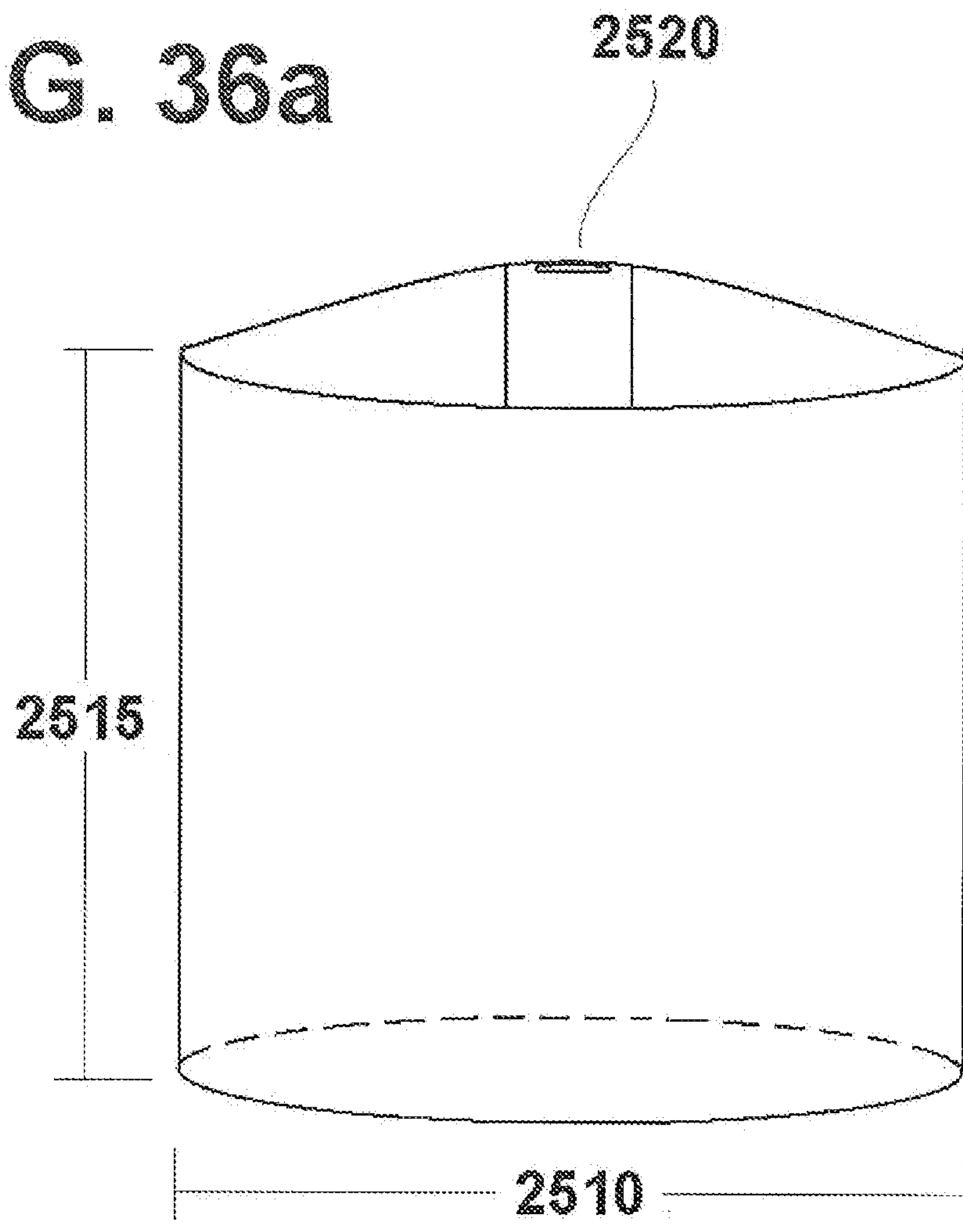
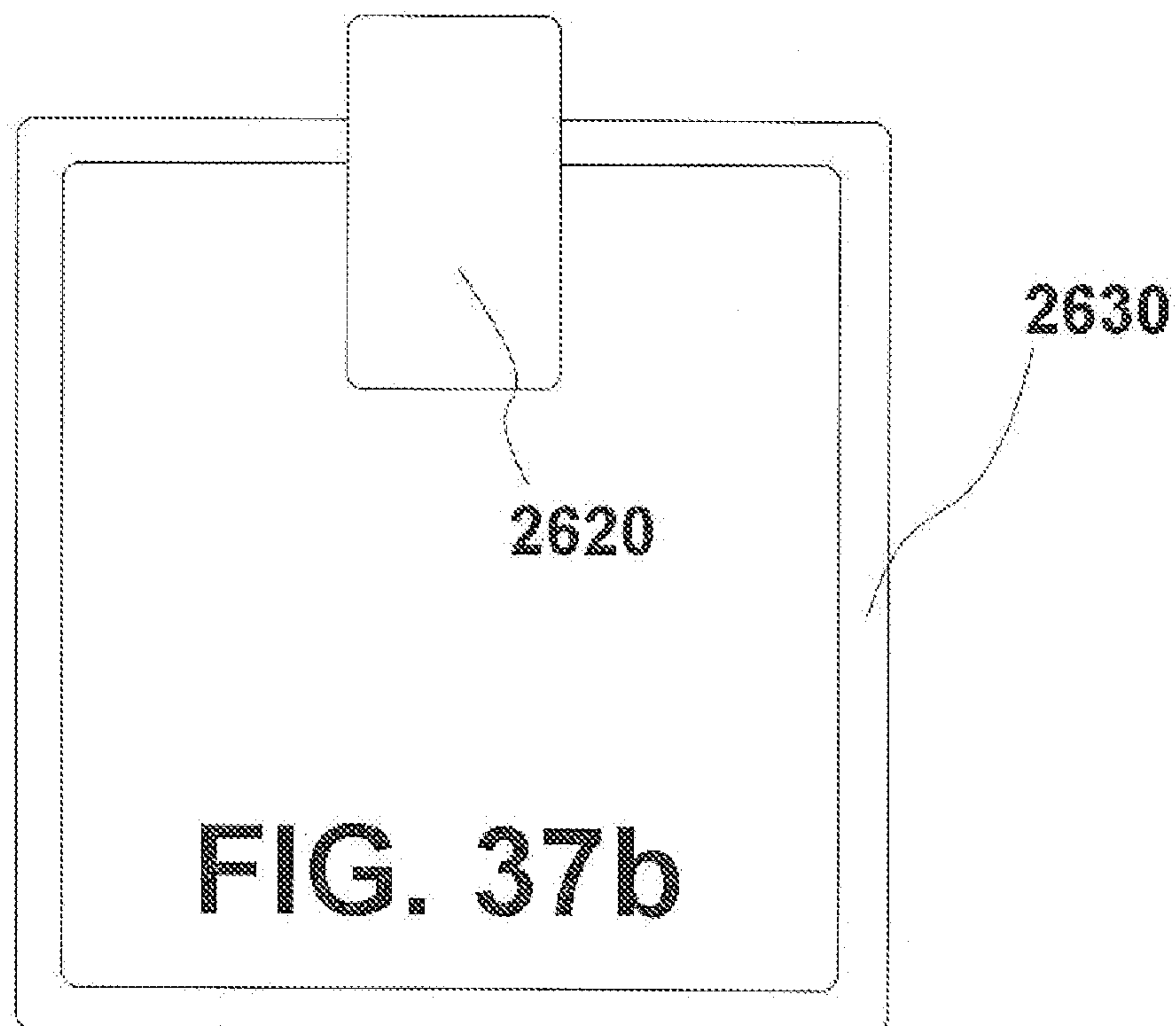
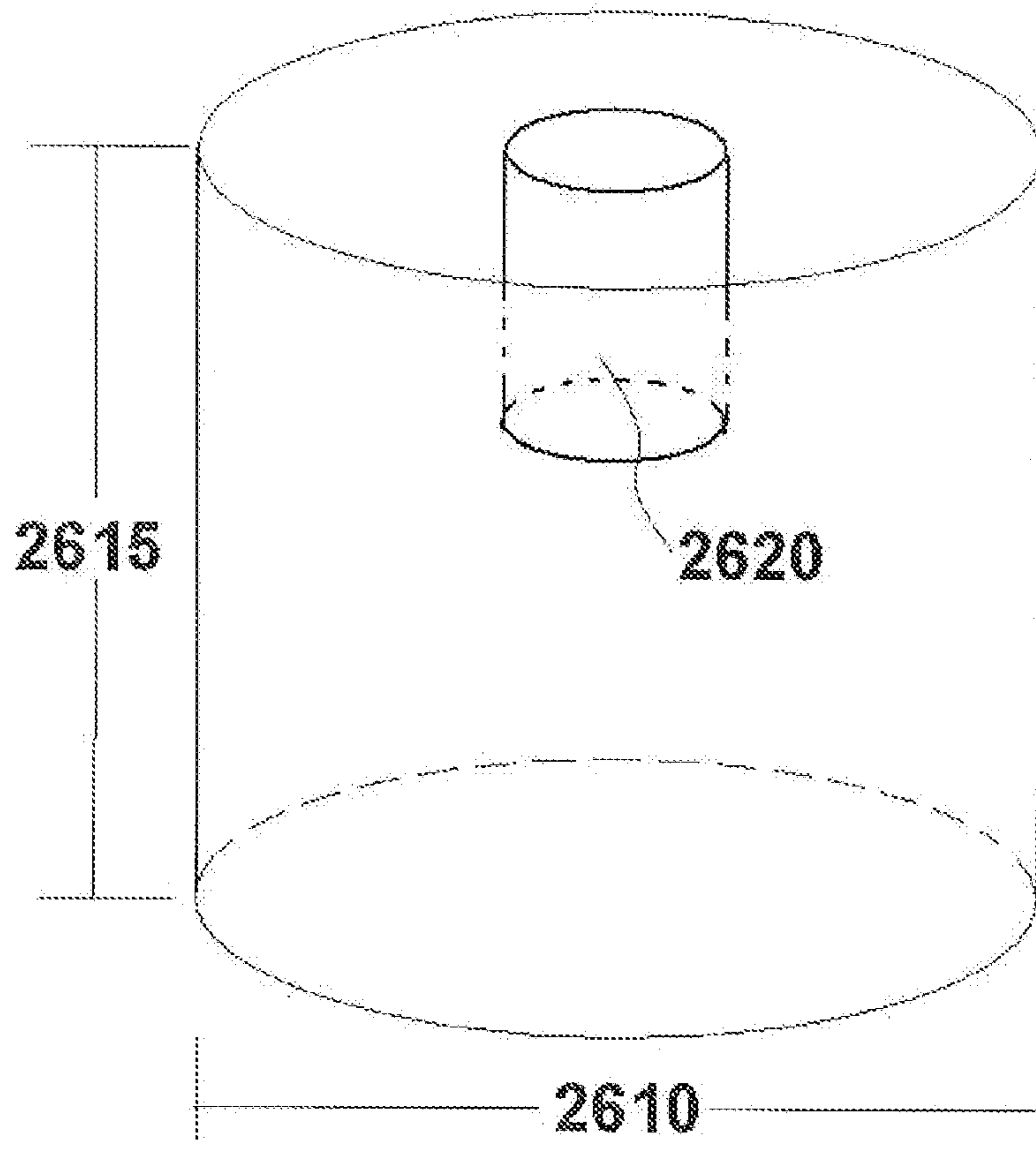
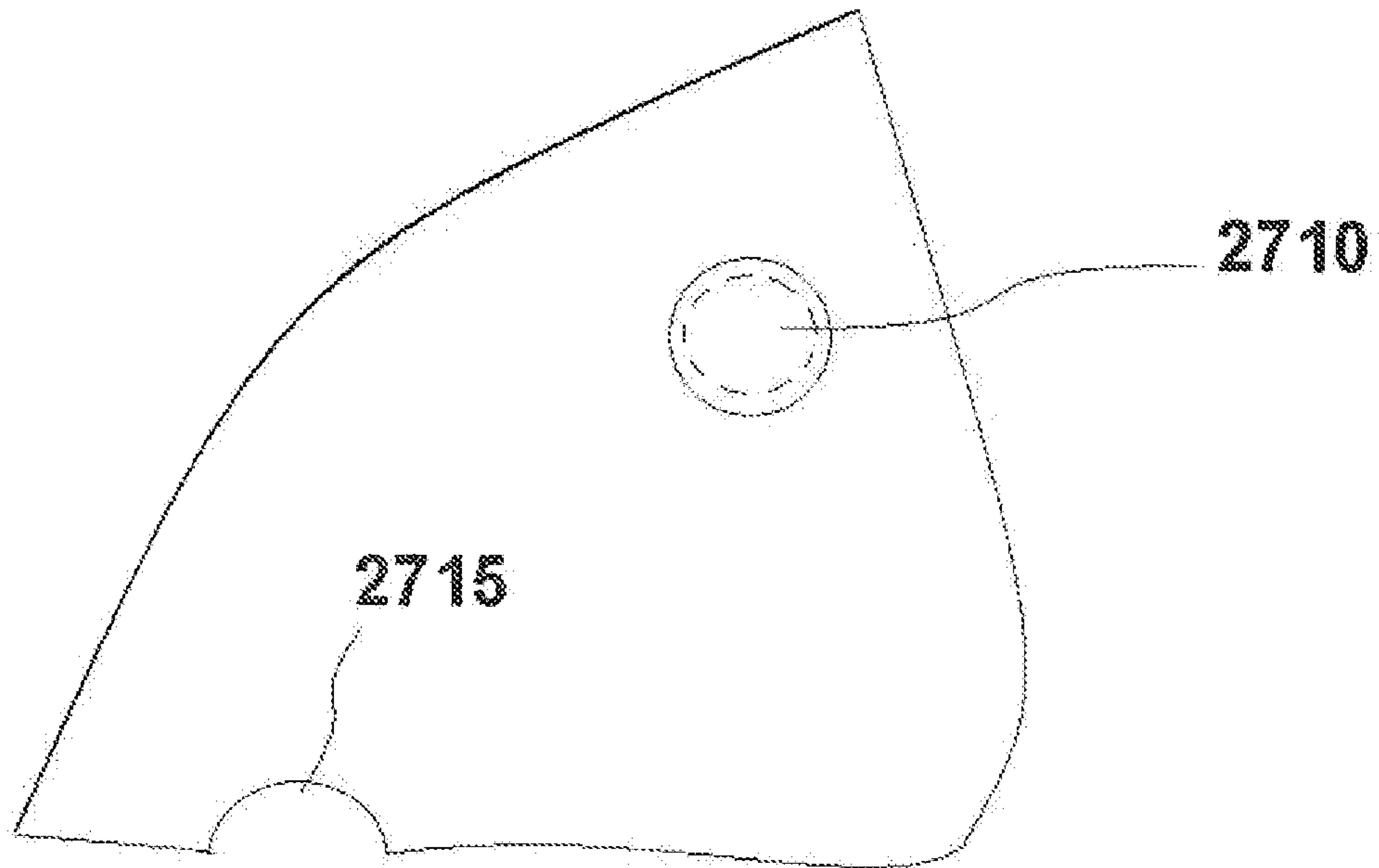
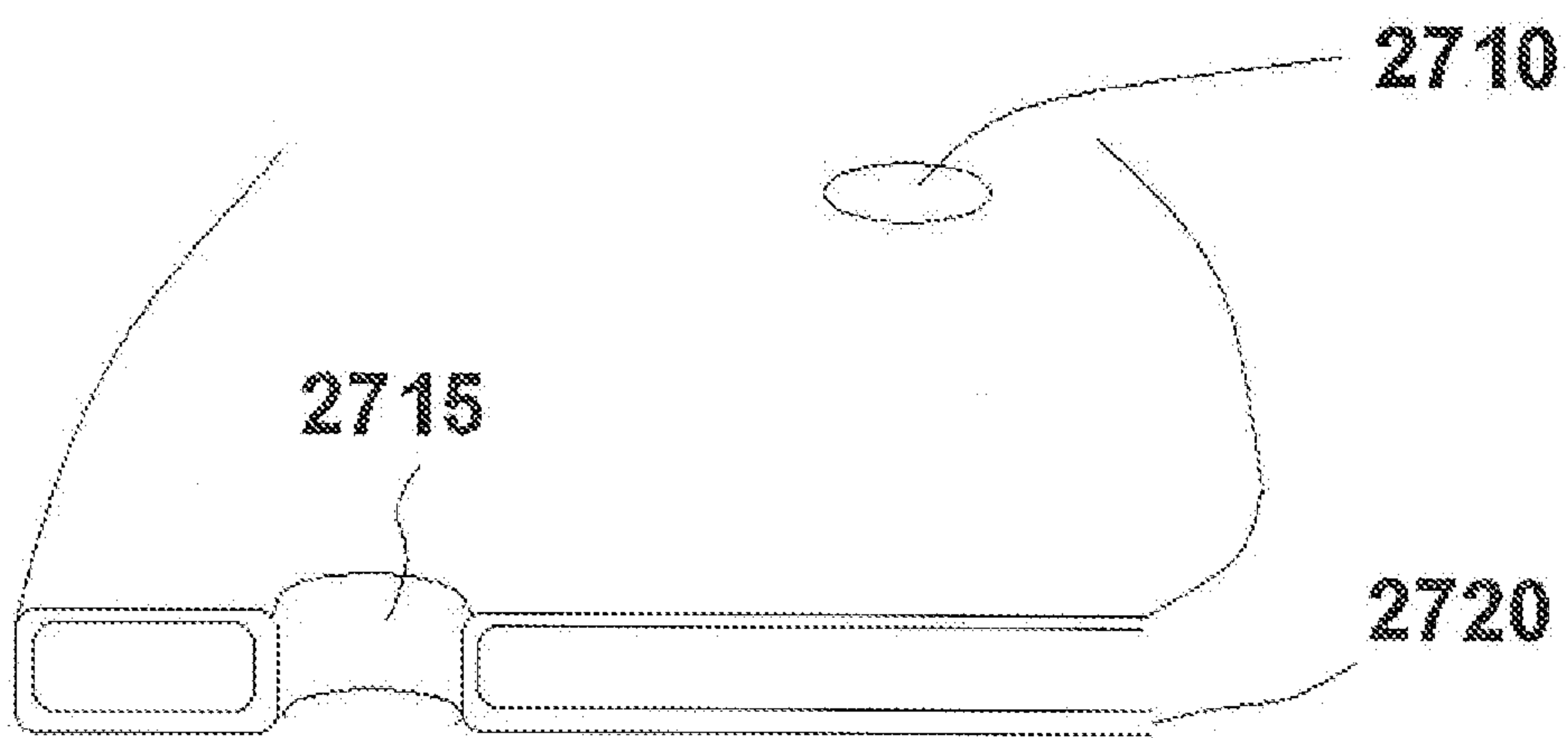
FIG. 36a**FIG. 36b**

FIG. 37a

**FIG. 38a****FIG. 38b**

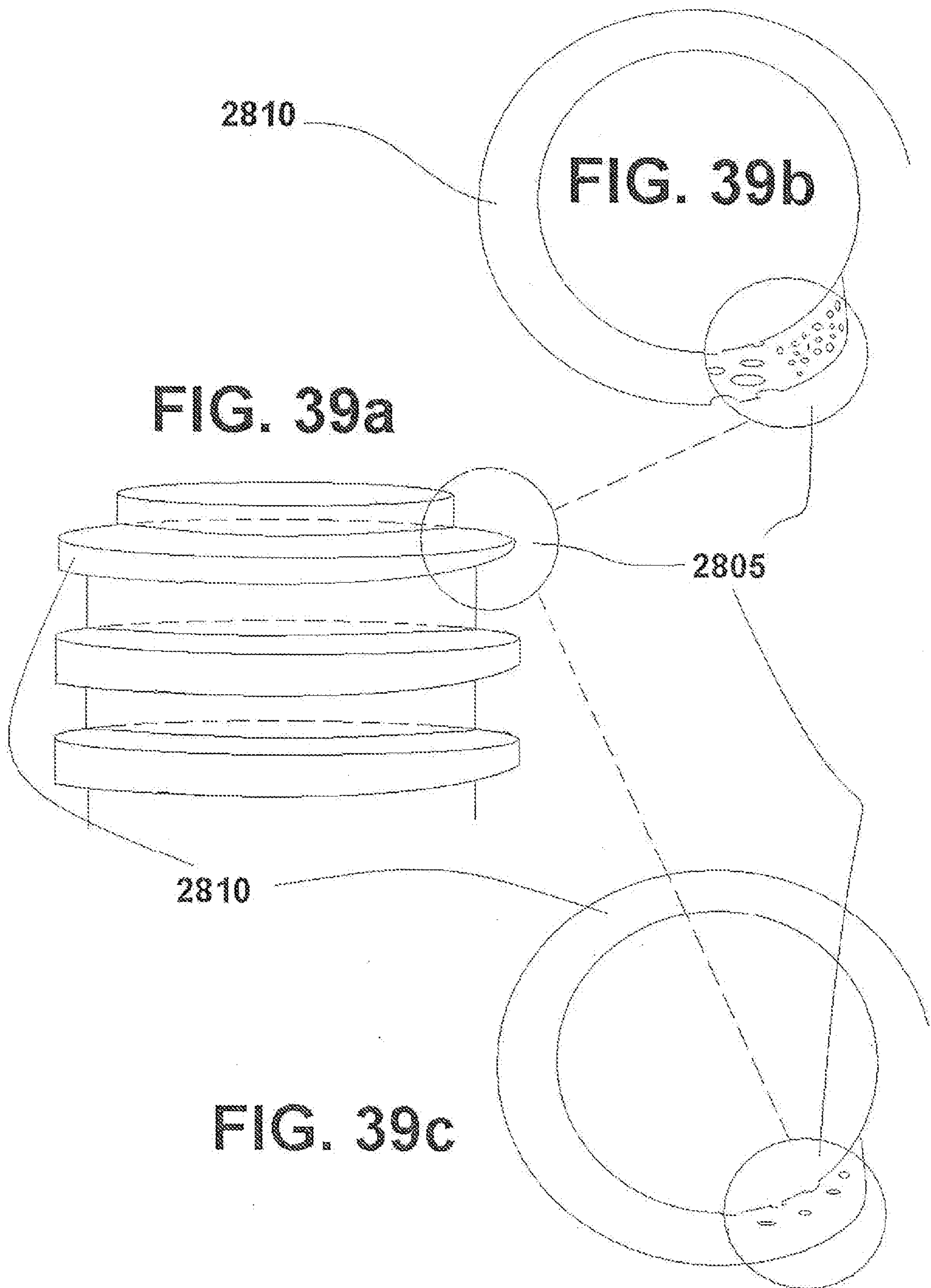
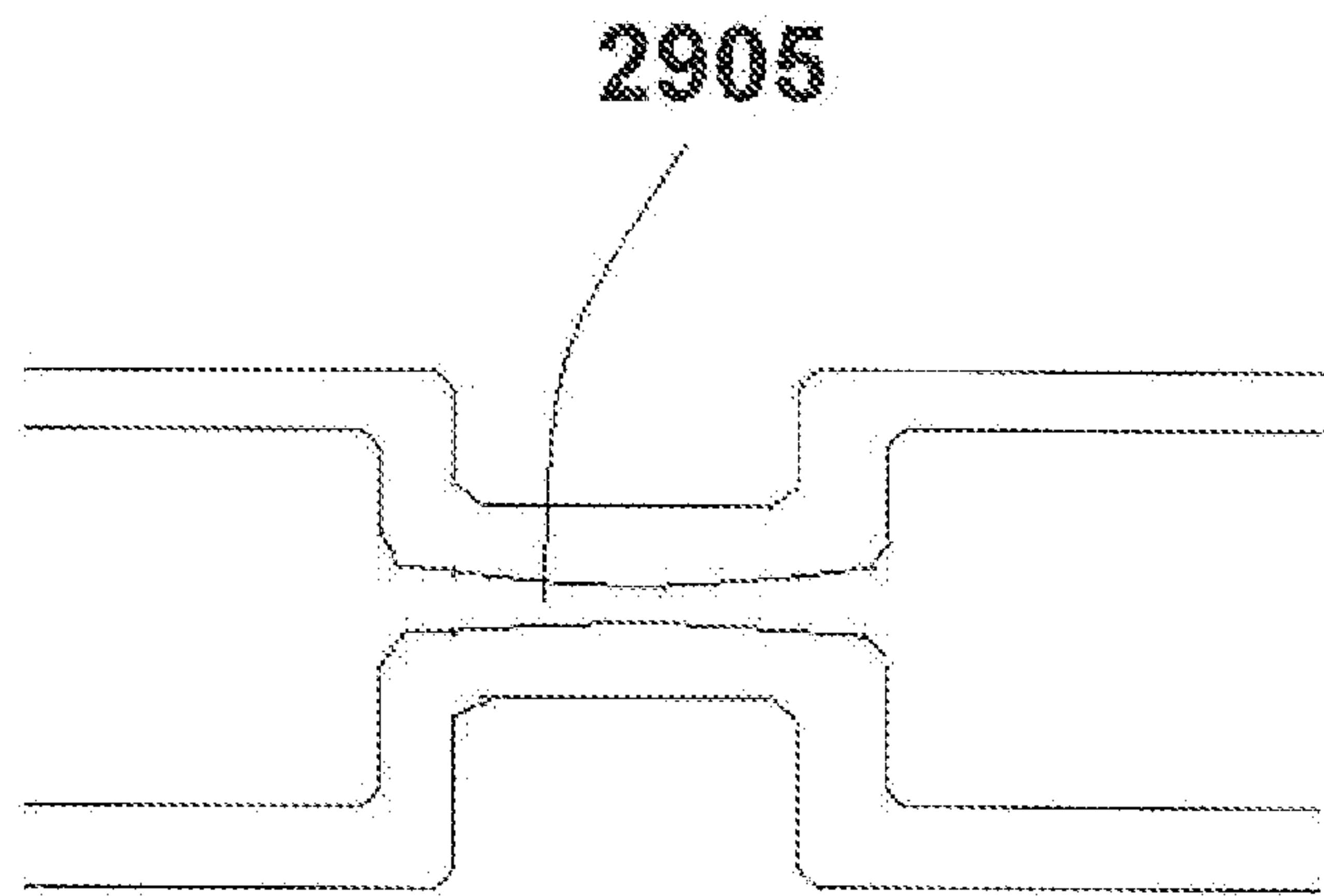
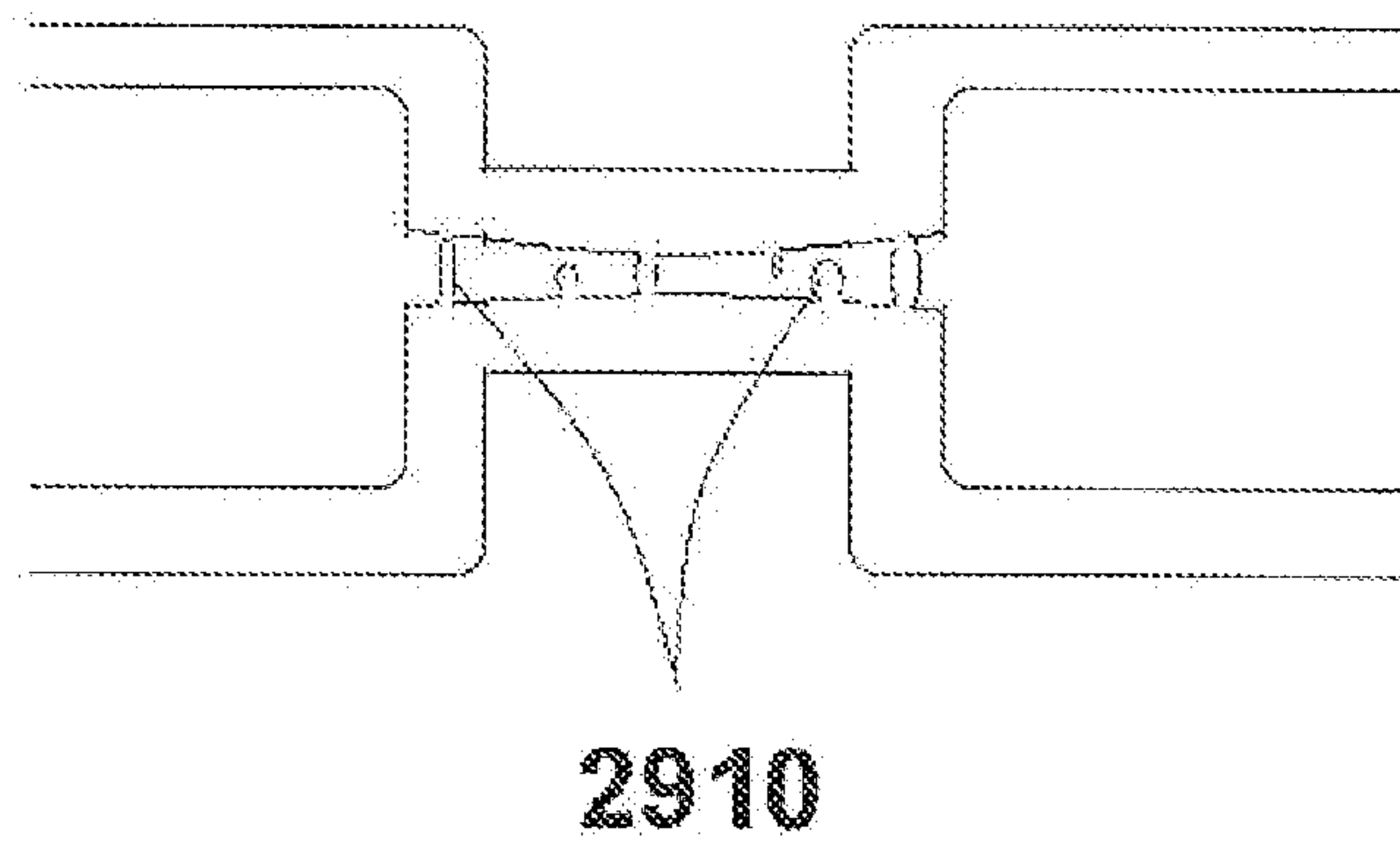
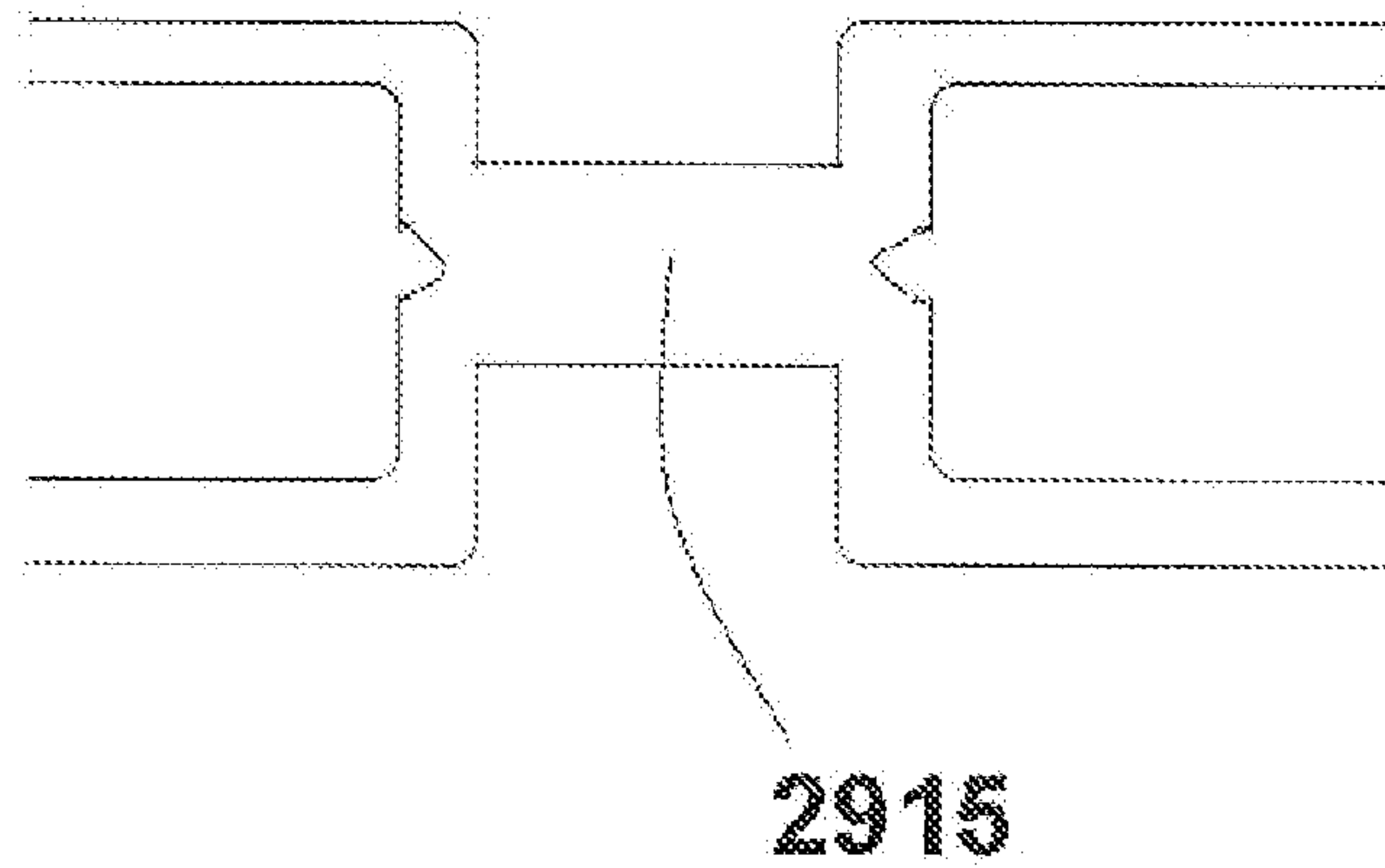
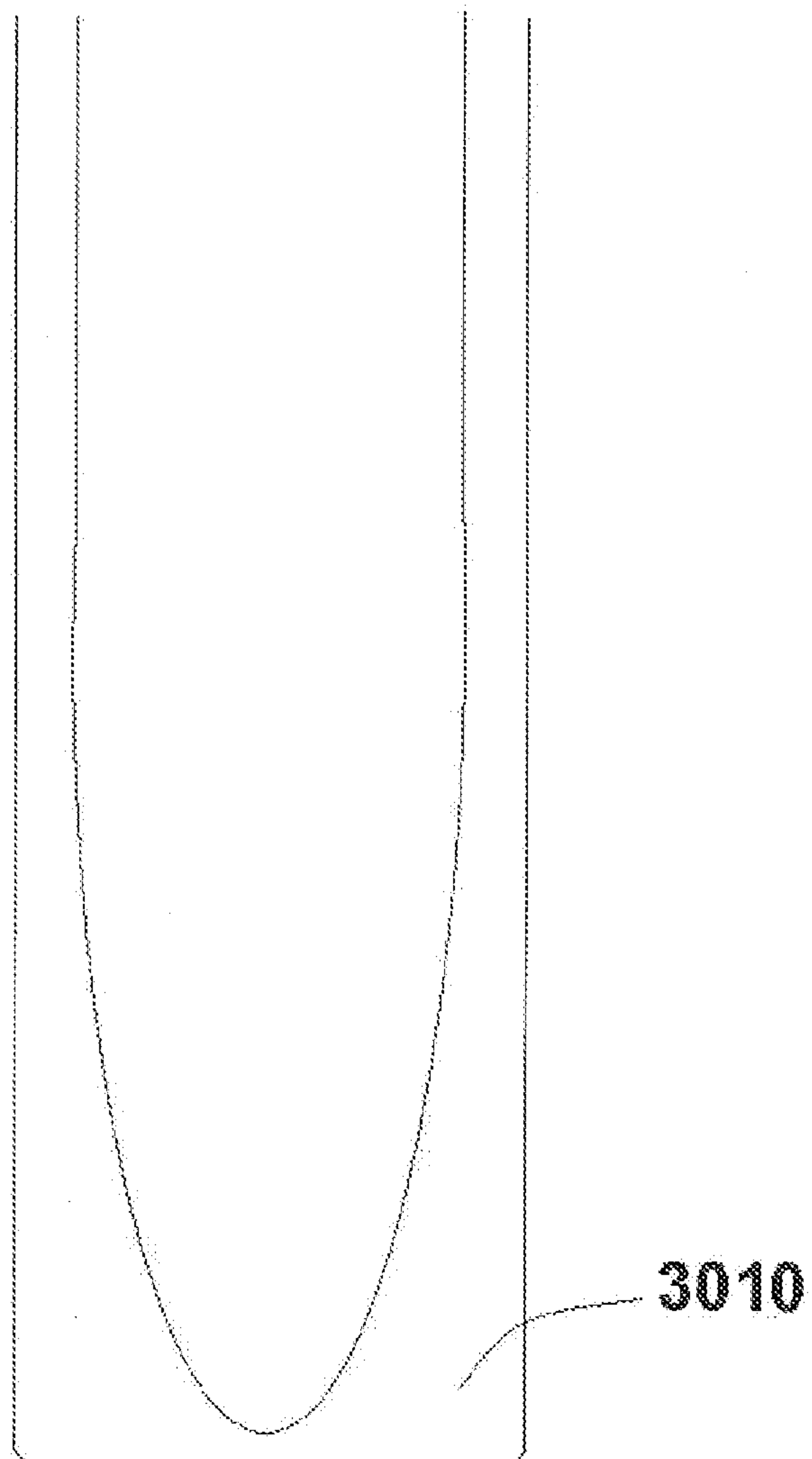
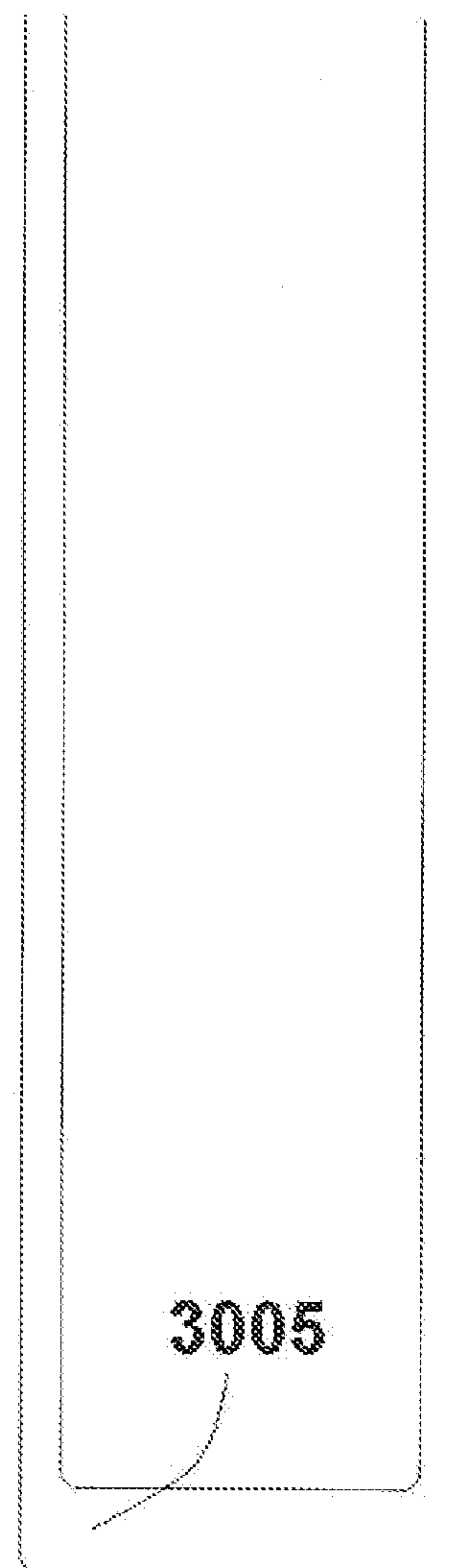


FIG. 40a**FIG. 40b****FIG. 40c**

**FIG. 41a****FIG. 41b**

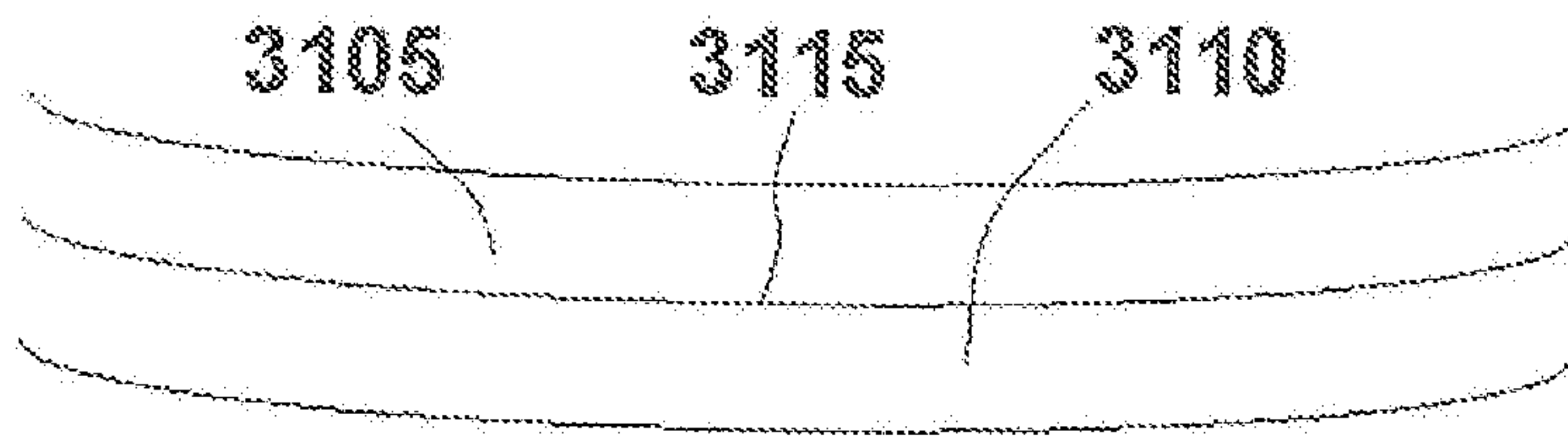


FIG. 42a

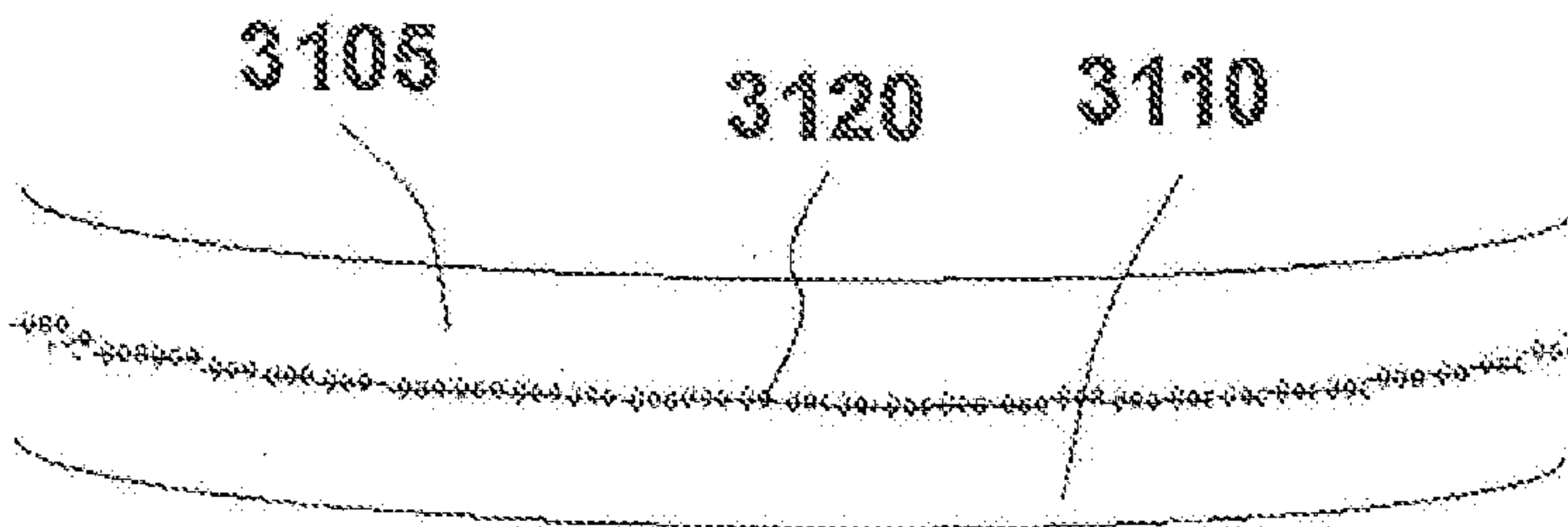


FIG. 42b

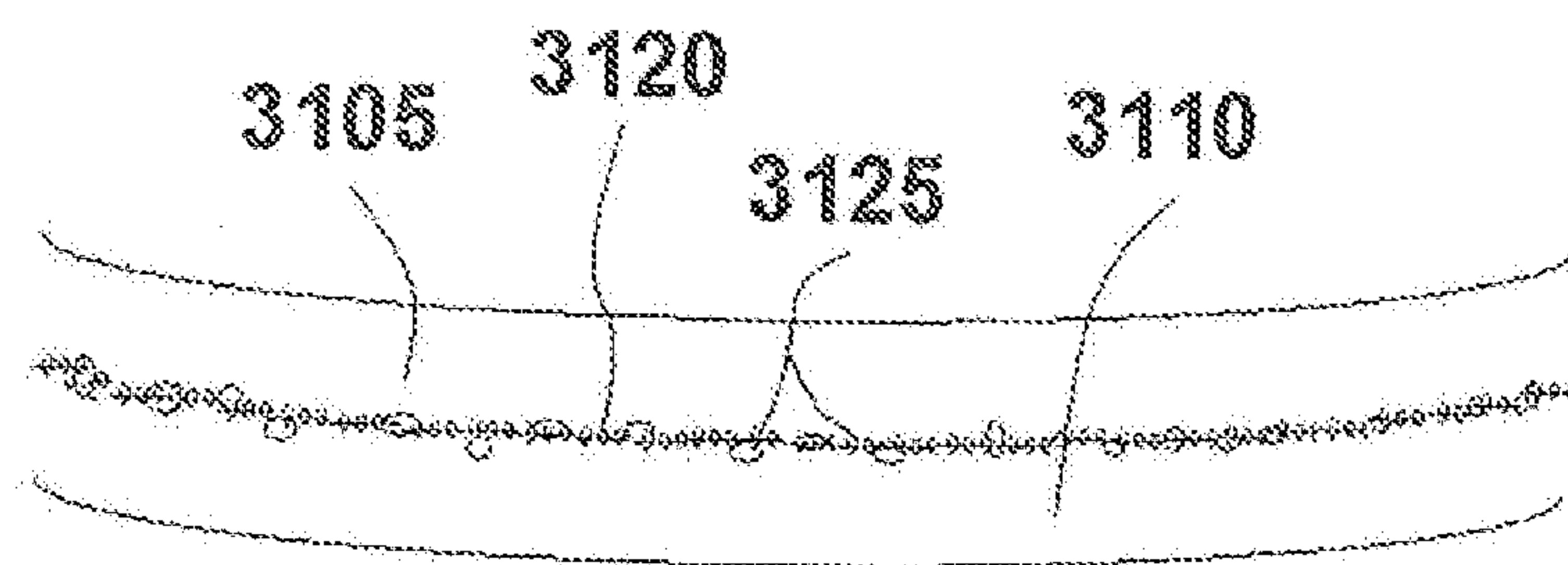


FIG. 42c

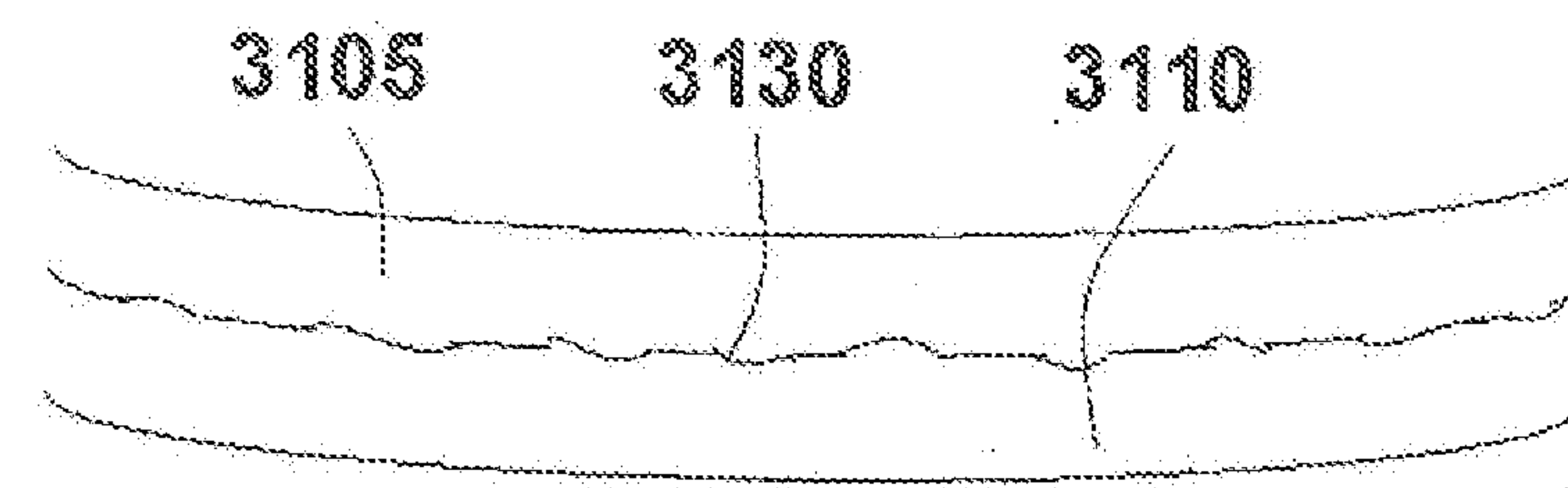


FIG. 42d

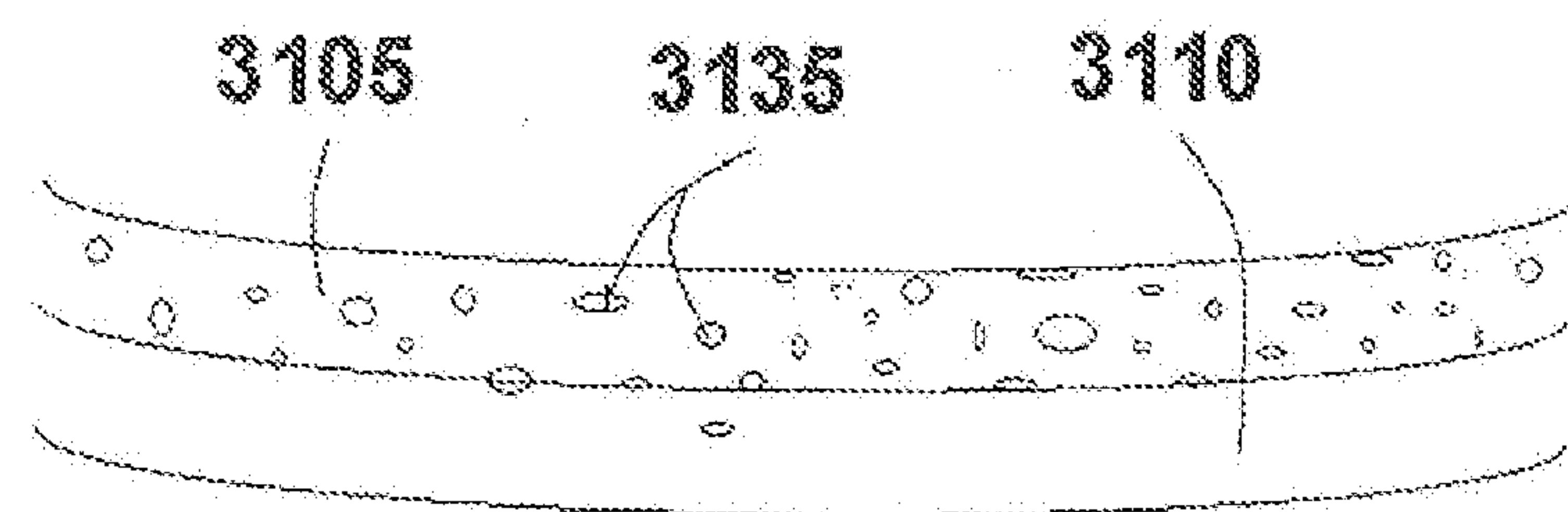


FIG. 42e

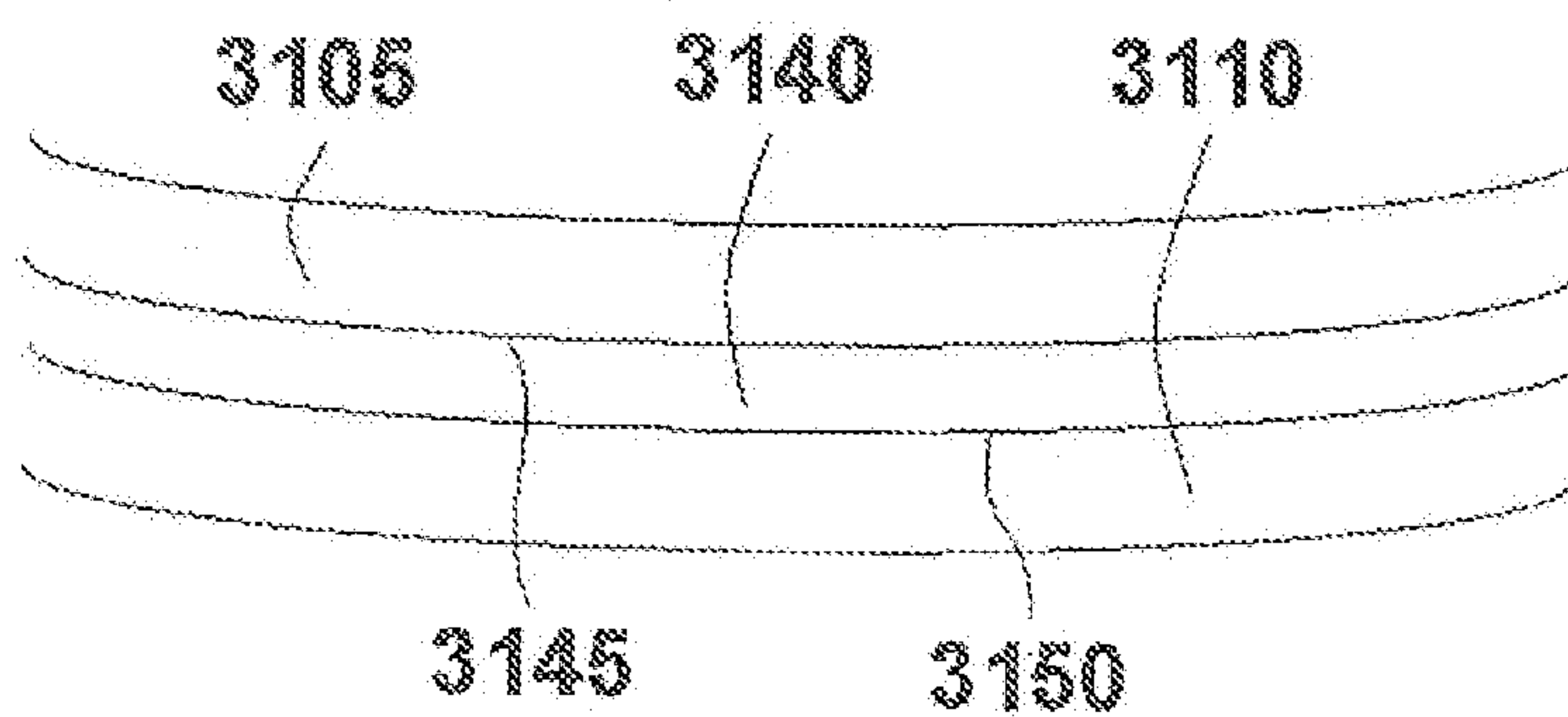


FIG. 42f

FIG. 43

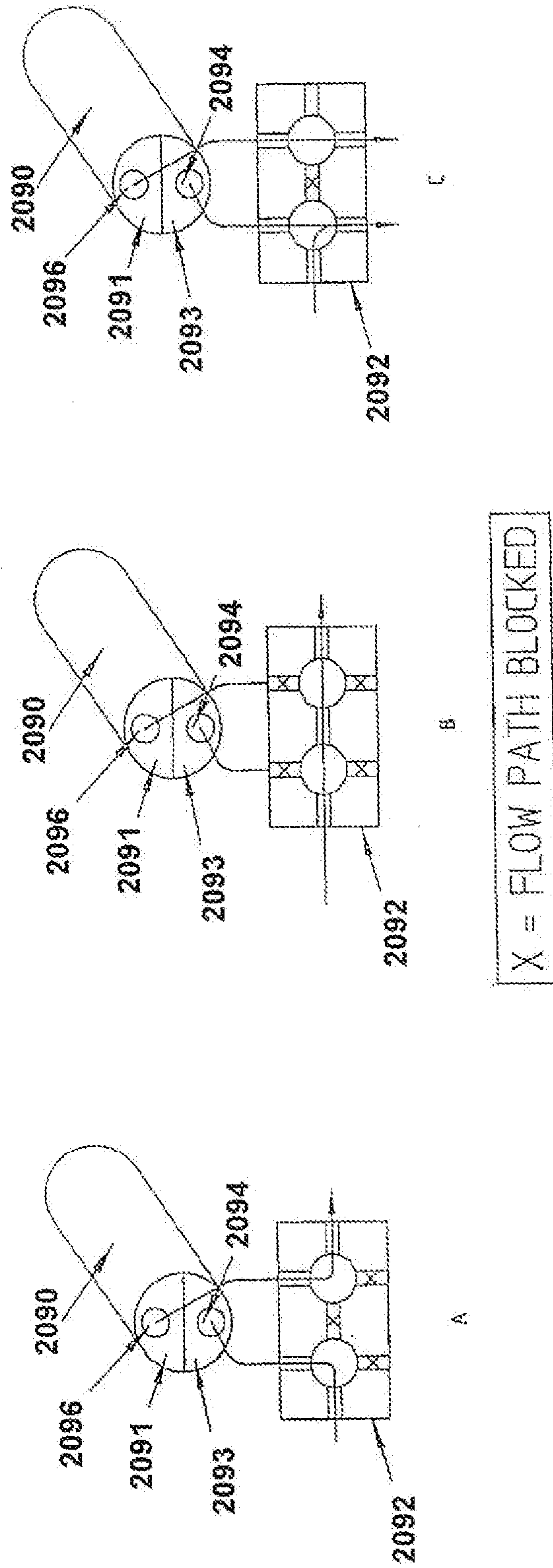
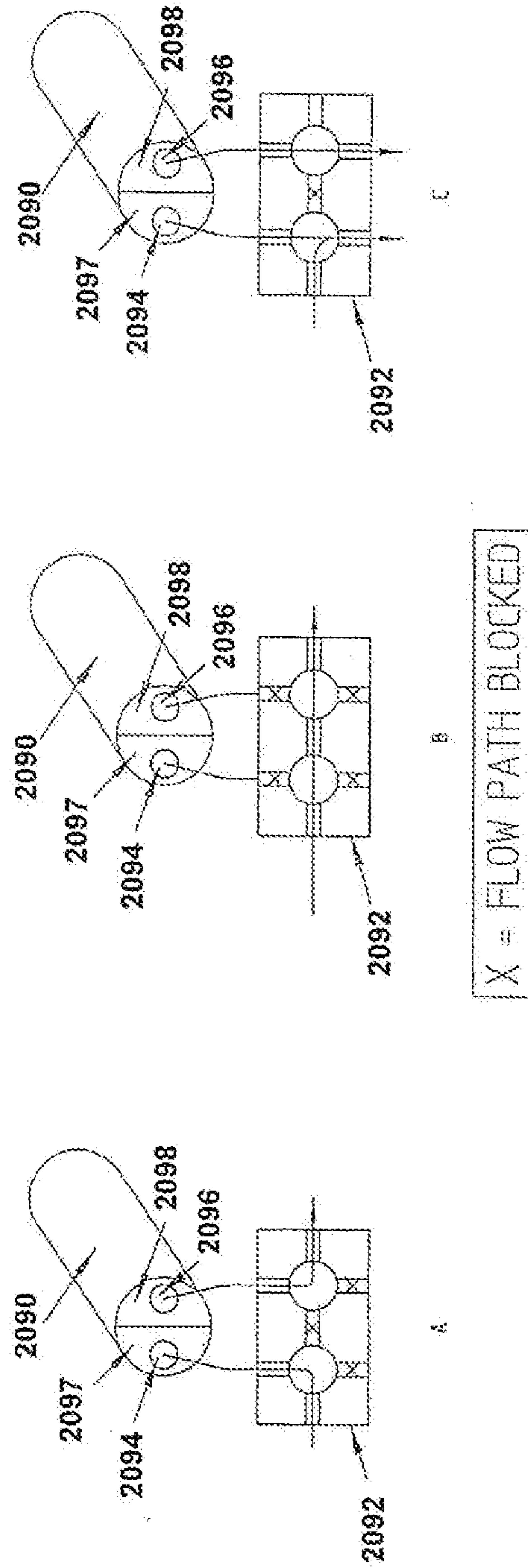


FIG. 44



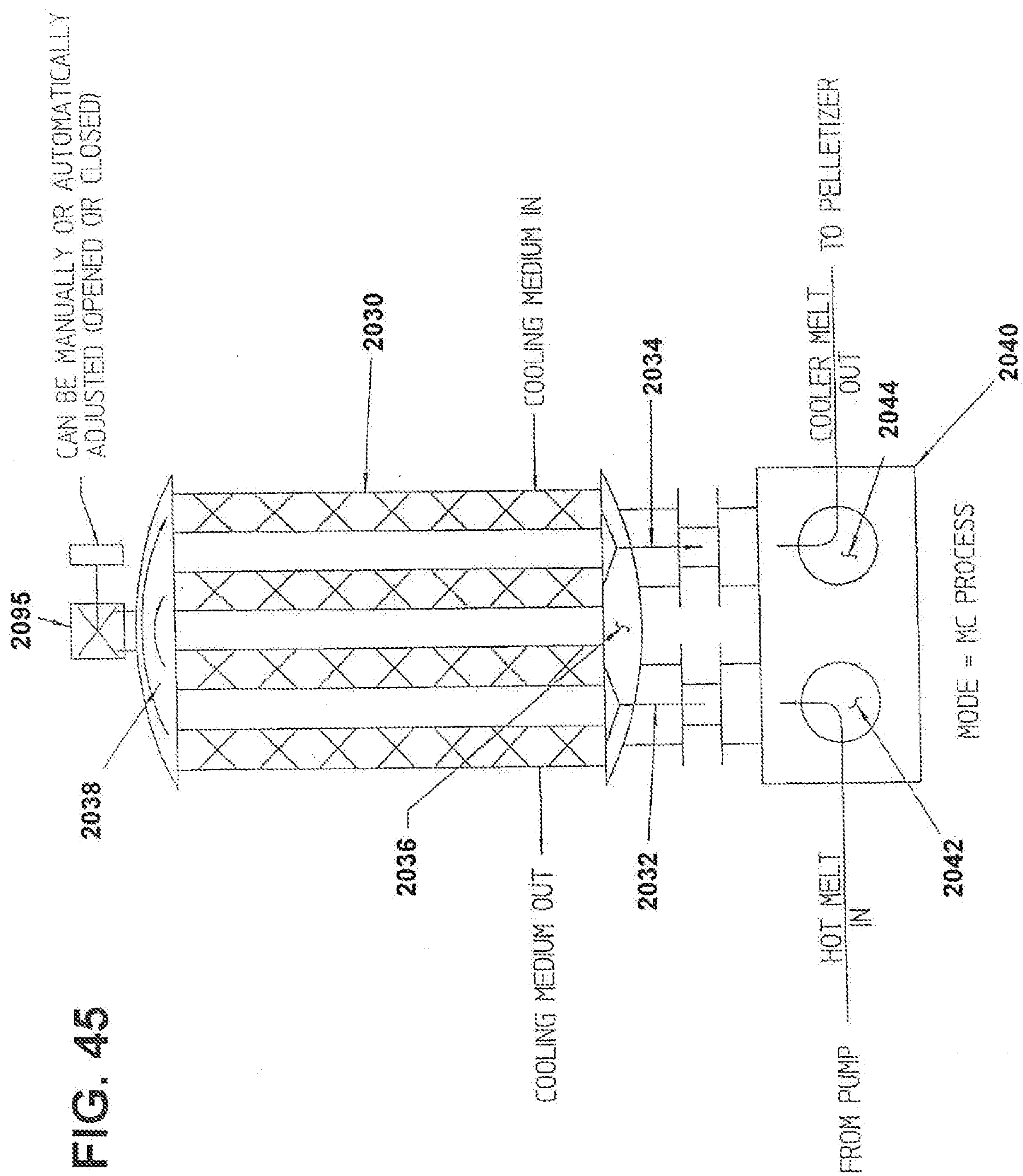


FIG. 46

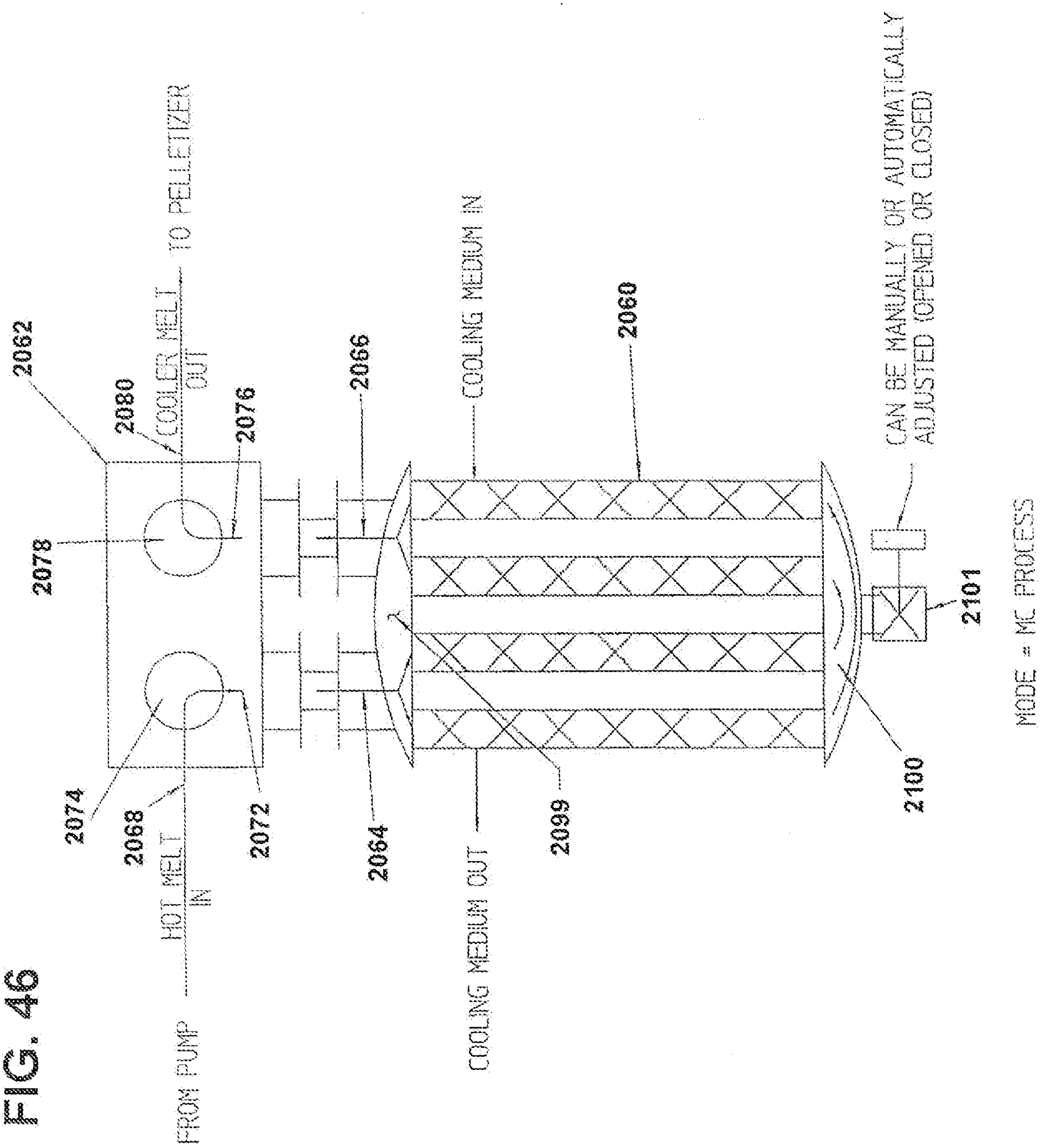


FIG. 47

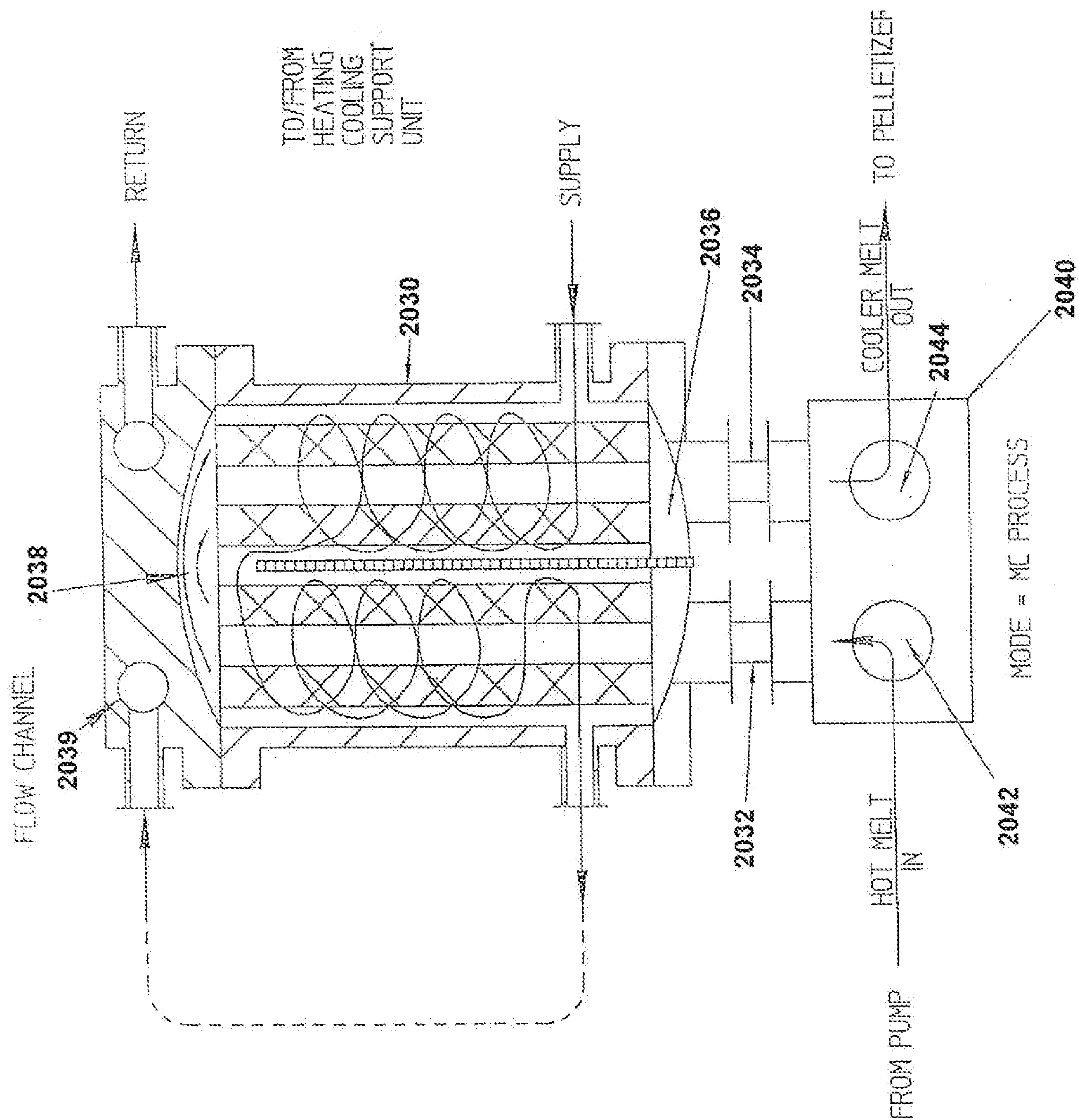


FIG. 48

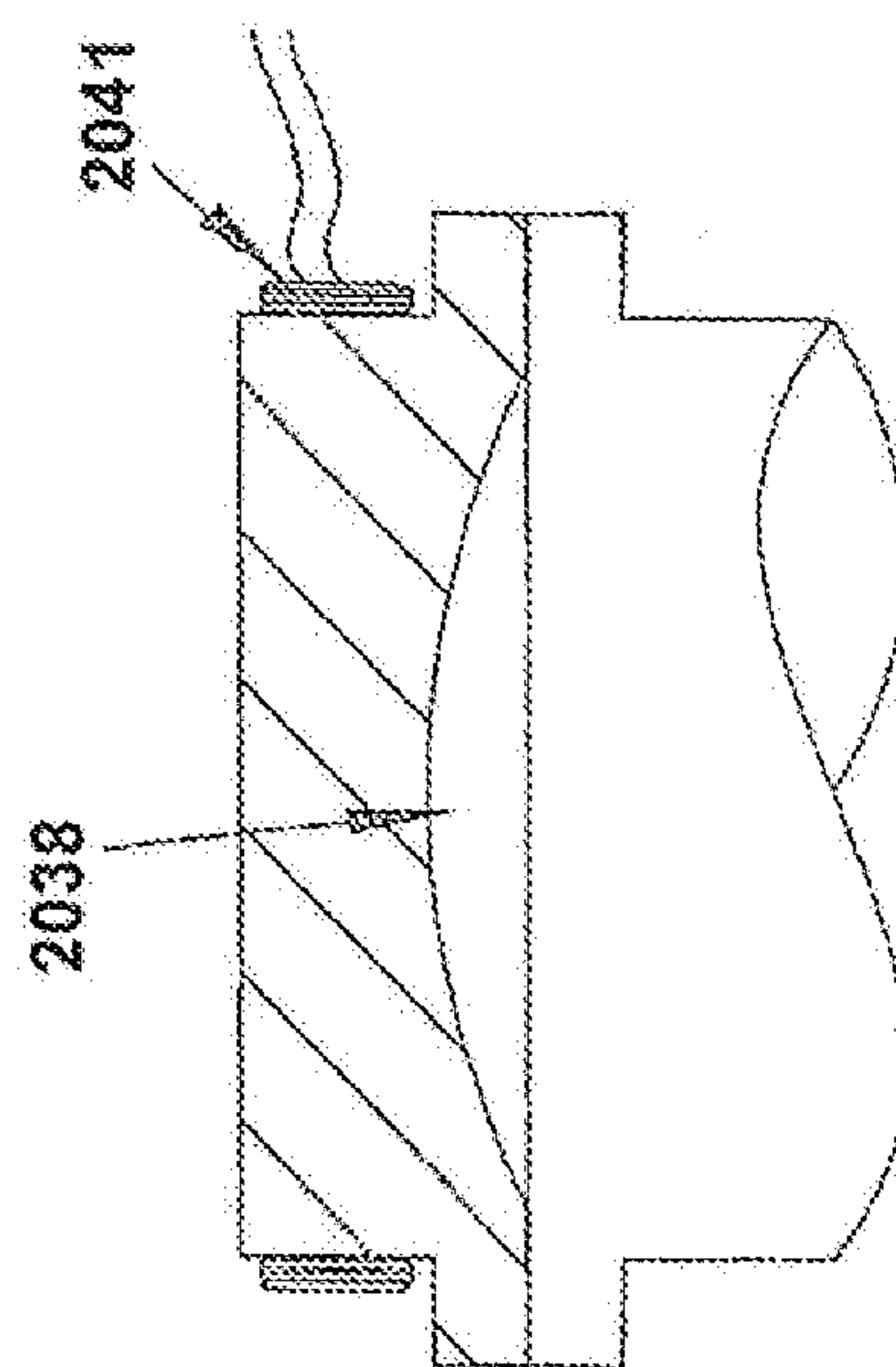


FIG. 49

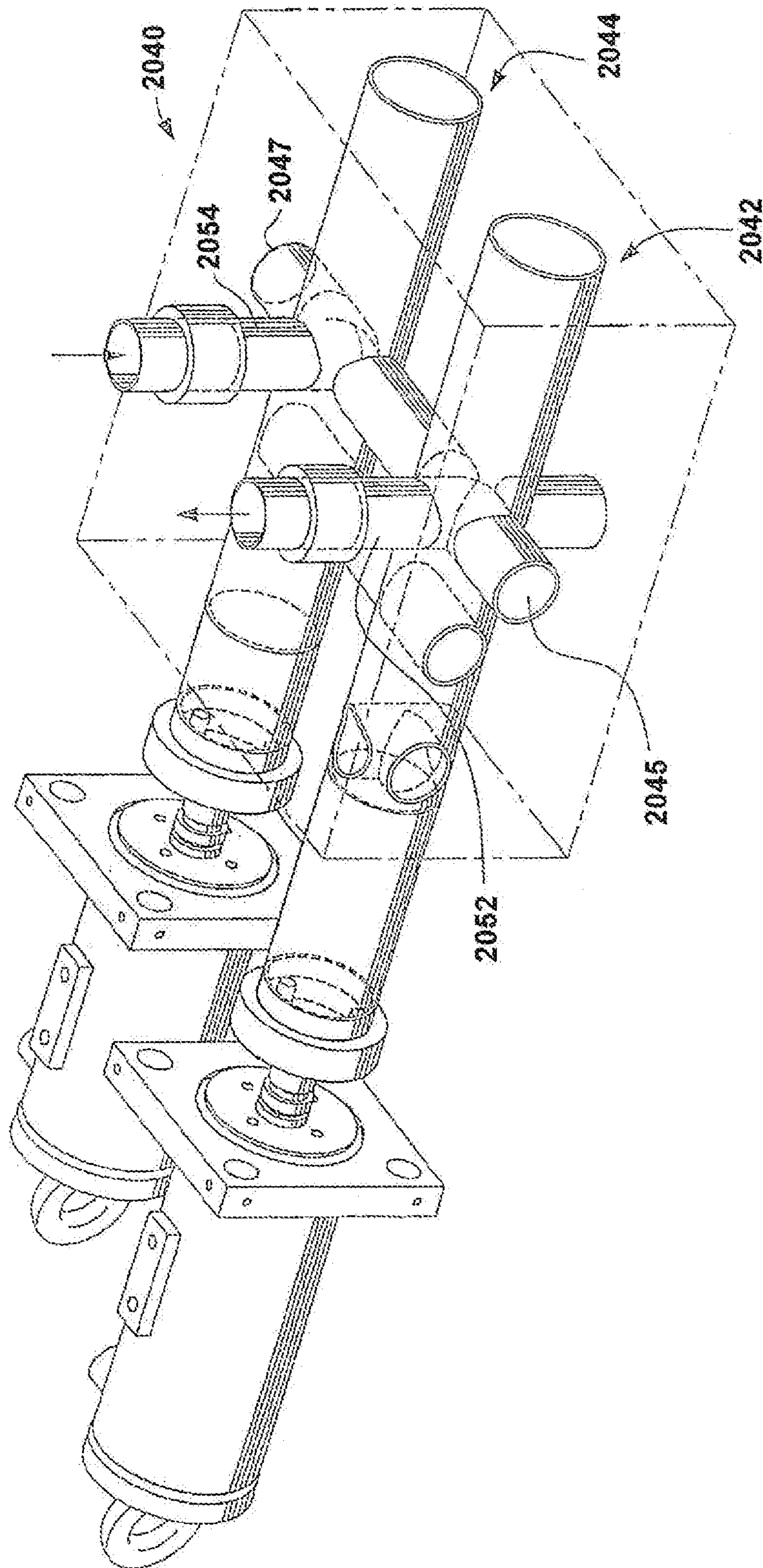


FIG. 50

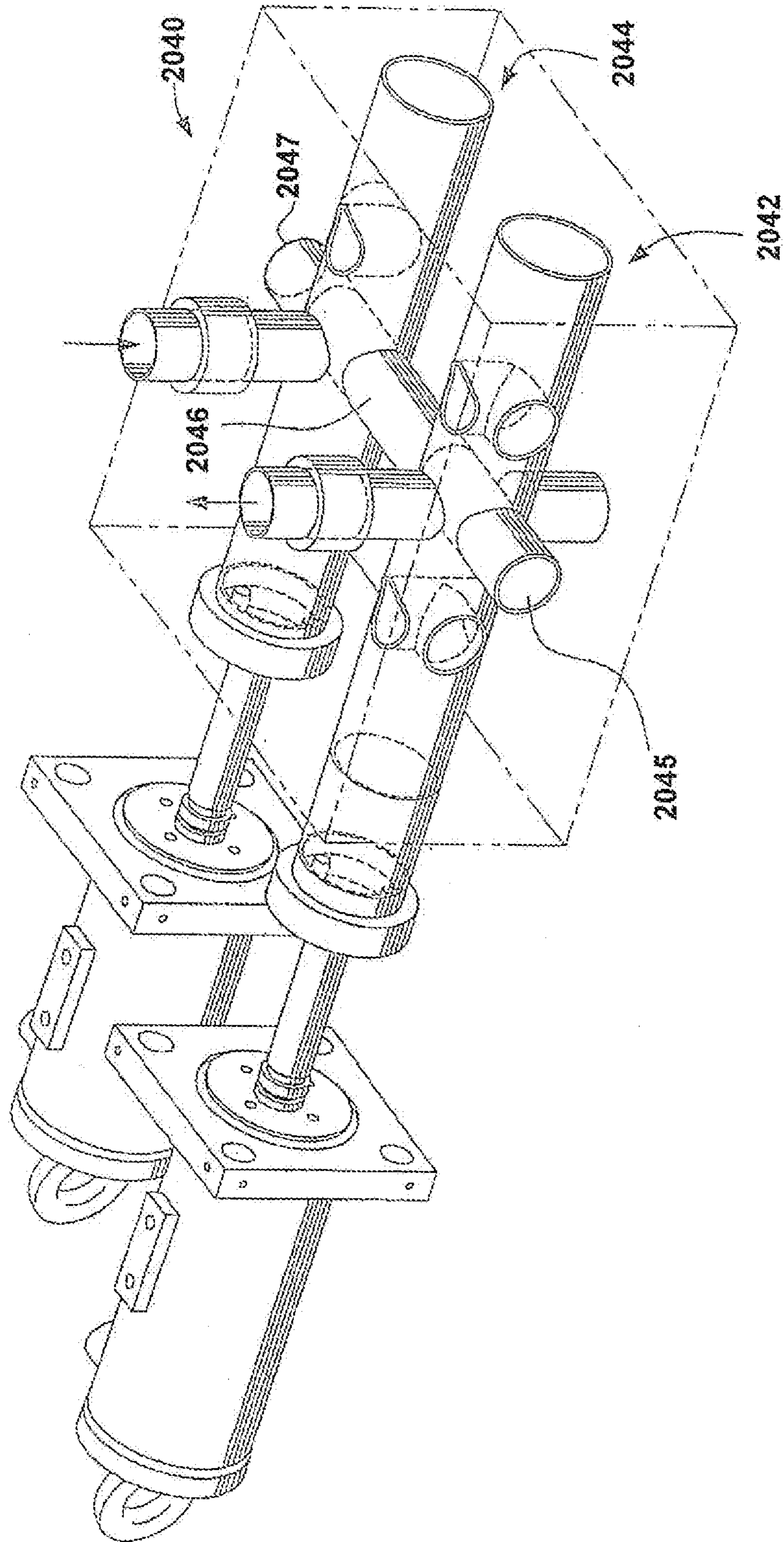


FIG. 51

