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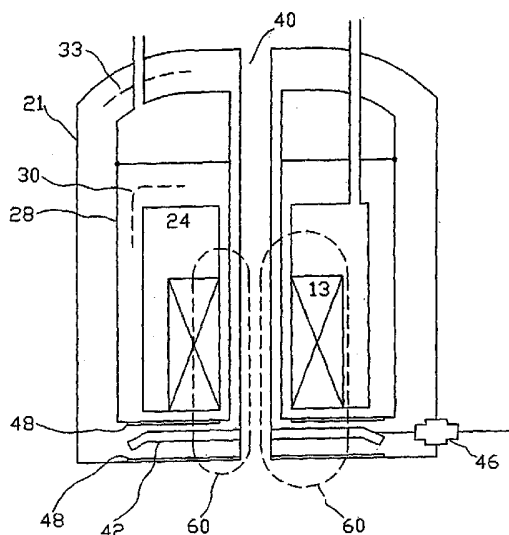
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(54) Title: ION PUMP FOR CRYOGENIC MAGNET APPARATUS



(57) Abstract: Ion pumping modules without ion pump magnets are disposed within evacuated spaces located within the fringing fields of cryostatic housed high field magnets. Improved vacuum conditions are obtainable both within superconducting magnet cryostats and for evacuated auxiliary apparatus proximate the magnet cryostat, particularly where such auxiliary apparatus is sensitive to RF noise.

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ION PUMP FOR CRYOGENIC MAGNET APPARATUS

FIELD OF THE INVENTION

The invention relates to the field of vacuum ion pumps and particularly pertains to
5 vacuum pumping applications with cryostat housed magnets.

BACKGROUND OF THE INVENTION

Vacuum ion pumps have been well studied over a period of more than 40 years, are the
subject of a vast literature and have proven to be successful commercial products for
10 maintenance of high vacuum. A review of such apparatus may be found in M. Hablanian, High
Vacuum Technology: A Practical Guide, pp. 360-372, (Marcel Dekker, Inc., 1990).

Although ion pumping apparatus is not limited to a particular geometry, the vacuum ion
pump ordinarily includes an anode formed from an array of close packed, axially symmetric
(tubular) metal elements and a cathode surface normal to the axis of the tubular array and spaced
15 apart from the anode. ("Tubular" includes hexagonal, rectangular, circular, square and other
cross sections for the purposes of this work.) The cathode(s) and anode(s) are disposed within a
hermetic sealed housing. A schematic representation of a single elementary pumping cell of
prior art is illustrated in figure 2. The cathode 58 is a chemically active, non-magnetic metal,
typically titanium, vanadium, tantalum, or zirconium. The anode 50 is frequently stainless steel.
20 In a typical design the cathode 58 may face open ends of the tubular anode or form a vacuum
tight outer housing surrounding the anode. The housing further includes a hermetically sealed
connector (not shown) for applying an electric potential from a power source between anode 50
and cathode 58. The prior art pump is completed by a magnet 54 disposed about the housing for
imposing the requisite magnetic field 52 parallel to the anode axis 56. In operation the combined
25 electric and magnetic fields act to substantially increase the density of energetic electrons. These
electrons ionize residual gases and the resulting positive ions fall through a potential difference
of the order of KV and thus accelerate to the cathode where these ions are chemically or
physically captured. The cathodes provide an electric field mirror for returning the electron
trajectories to the vicinity of the anode. Particularly light mass energetic ions (hydrogen)
30 penetrate the cathode surface and remain buried therein. In various forms and sizes such
vacuum ion pumps have been used as main pumping elements and as simple appendages to
sealed units of various apparatus. Of particular importance to the present invention is the
employment of such vacuum ion pump apparatus with cryostat- housed magnets.

The superconducting magnet that is a central component of modern NMR (including magnetic resonance imaging) apparatus is housed in an axially symmetric cryostat having a room temperature bore aligned along the axis of the magnet. A typical cryostat may include one or more heat shields and one or more reservoirs containing a liquefied gas (cryogen), the latter being vented to atmosphere or arranged in an appropriate closed cycle arrangement. The heat shields and reservoirs are contained in a vacuum insulated environment to thermally isolate these components from the ambient environment. The magnet is carefully designed to create a region of extremely homogeneous or (controlled gradient) magnetic field along the bore. This is a dipole field with the field distribution substantially confined to a central region, then flaring out at the ends of the bore to close by return from flared-out end region to the opposite flared-out end region. The field proximate to the ends of the bore flares out, that is, the field disperses radially and curves around the exterior of the magnet windings to return via the other end of the magnet windings. The field is thus diminished in density in the region external to the magnet windings and is termed a "fringe", or "stray" field. While these magnets are designed with means to protect the inner region of the bore from outside magnetic influences and to minimize the magnetic field in the environment of the magnet, it is sufficient to observe that there is a substantial fringe field in the environs of the magnet. Because these magnets are capable of very high field intensity, the fringe fields, even when minimal, are far from negligible in proximity to the magnet/cryostat. The fringe field of such apparatus is a source of possible damage or anomalous behavior to some instrumentation, credit cards and possibly dangerous to human beings with pacemakers or defibrillators implanted.

It is apparent that the thermal performance of the cryostat is adversely affected if the gas pressure in the evacuated region should rise to the point that such higher pressure supports non-radiative mechanisms for heat transport, compromising the function of the cryostat.

Conventional arrangements to maintain satisfactory vacuum conditions in such apparatus typically include a "getter" or sorbing agent within the vacuum space to trap gases.

Superconducting magnets are often identified with magnetic resonance instruments, a major application thereof. In the particular case of NMR apparatus, a homogeneous magnetic field is a basic requirement. The NMR phenomena is also critically dependent upon an RF probe coil (or coils) for exciting resonance and for coupling the resonance fields to sensitive detecting apparatus. A very much improved signal-to-noise ratio (SNR) is achievable for NMR data acquired with the RF coil maintained at low temperature. Several factors contribute to this temperature dependence of the signal. It is understood that cooling the RF coil to the superconducting phase transition of the coil conductor represents one goal, but it is still quite

desirable to operate the RF coil at depressed temperatures that need not reach as far as a superconducting transition. Accordingly, there is a need for thermal isolation of a cooled coil, and enclosure in vacuo is necessary.

The apparatus under discussion may be regarded as including as many as two separate cryostat structures. One of these is the housing (cryostat) surrounding the actual magnet windings and the other is the cryo-housing or dewar of a low temperature RF coil. The interior regions of these cryostats include spaces maintained at extremely low pressure to provide thermal insulation. In the example of a common magnet cryostat, one or more cryogen(s) boil slowly from respective cryogen reservoirs thereby removing heat from the local environment and the resulting vapor escapes to the atmosphere through a necktube. Closed cycle systems are also available to provide refrigeration apparatus to remove heat from the magnet windings and radiation shields without loss of the cryogen(s) or, without the need for cryogen(s) at all. In any case, the degree of thermal isolation from ambient environment requires satisfactory vacuum for the cryostat interior between the cryostat outer walls and the cryogen reservoir. An RF coil cryostat, or dewar, similarly employs a vacuum jacket containing a refrigerating mechanism removing heat from a cold heatsink ("cold head") cooled by cryogen circulated therethrough. The RF coil thermally communicates with the heatsink. In prior art, the dewar is maintained at the desired low pressure by a mechanical pump communicating with this vacuum jacket. It is also known to employ a sorb to sequester gases evolving (outgassing) from the walls of the jacket and other components located therein. Mechanical pumps pose a problem in that the vibration spectrum must be carefully suppressed to prevent introduction of artifacts into the NMR spectrum. Sorbs do not enjoy a very long useful life and must be renewed or re-activated.

It is also known that dewar housed RF coils for NMR studies are a source of pressure dependent RF noise triggered by high power RF pulses. This effect is described in US 4,240,033, commonly assigned. That work taught moderation of the effect to some degree by insertion of a material to serve as a baffle for reducing the probability of collisions involving ions generated in the dewar. The effect is thus attributed to breakdown of residual gasses in the vacuum space of the RF coil dewar.

SUMMARY OF THE INVENTION

The present invention, in a first embodiment, achieves vacuum ion pumping in and close to a magnet cryostat with a vacuum ion pump that utilizes the fringe field of the cryostat-housed magnet. Such vacuum ion pump represents a less expensive and less weighty pump than prior art vacuum ion pumps. In one embodiment the elements of the pump form internal structure

within the space to be evacuated, such as within the evacuated volume of the magnet cryostat, disposed in a region thereof where a significant component of the fringe field is oriented substantially parallel to the axis of the anodes. In a variation of this embodiment, the pump is located outside the main structure of the magnet cryostat with gas communication to the evacuated region, but in sufficient proximity to regions of the fringe field where the orientation and intensity is sufficient for vacuum ion pump operation. In yet another embodiment, another evacuated volume, in close proximity to the cryostat structure but independent thereof, is maintained at a desired vacuum. An example of the latter embodiment is a dewar enclosing the cooled RF coil of an NMR apparatus where such dewar is located in the bore of the cryostat and the supporting apparatus housing a suitable cathode/anode array is necessarily located proximate one end of that bore intercepting a substantial fringing field of the magnet. The latter embodiment is especially advantaged over conventional mechanical pumping for this purpose because the issue of vibration isolation from such mechanical pump is avoided, and the particular pumping properties of the vacuum ion pump aid in suppression of RF noise in the acquisition of NMR data.

BRIEF DESCRIPTION OF THE FIGURES

Figure 1 is a schematic illustration of first and second embodiments of the invention.

Figure 2 is a conceptual illustration of the typical vacuum pump cell of prior art.

Figure 3 is another embodiment of the invention.

Figure 4 is a superposition of pressure as a function of time and a scatter plot of NMR spectral noise measurements at different times in that environment.

Figure 5a shows a portion of an NMR spectrum of a sample at a selected high value of the probe dewar pressure.

Figure 5b is the same NMR spectral region as figure 5a at a relatively low probe dewar pressure.

DETAILED DESCRIPTION OF THE INVENTION

Figure 1 includes a symbolic representation in section of a (simplified) conventional cryostat 20 in which there are located superconducting windings 13 forming a cryogenic magnet. The cryostat 20 includes heat shields 30 surrounding low temperature cryogen reservoir 24, an intermediate temperature cryogen reservoir 32 establishing constant temperature (heat shield) surface 28 and a higher intermediate temperature heat shield 33 surrounding all of the above components, enclosed within hermetically sealed housing 21. The entire interior

communicating space of housing 21, exclusive of cryogen reservoirs 32 and 24, is evacuated to a pressure of the order of 10^{-4} torr at ambient temperature. When the cryostat is put into service by filling the cryogen reservoirs, most residual gases are condensed to a great degree and immobilized on the cold interior surfaces of the cryostat members, lowering the pressure to the order of 10^{-8} torr. While this results in satisfactory vacuum condition for the purposes of thermal isolation during cryogenic service, a significant partial pressure of residual light gases, principally H_2 and perhaps He, will generally remain. However, the partial pressure of these light gasses will continue to increase as they diffuse ("outgas") from cryostat components and accumulate in the vacuum space. (Moreover, in another embodiment discussed below, free hydrogen is produced during operation of that embodiment.)

The geometry of the cryostat typically includes a bore 40 for insertion of additional investigative apparatus or sample handling at ambient temperature. Such magnets are very frequently employed for NMR studies. Superconducting magnets are designed to produce extremely homogeneous magnetic field intensity within the bore 40 but the field distribution includes a portion external to the bore, representative magnetic field "lines" 60, 60' of constant magnitude being shown. It is understood that the magnetic field is characterized by a continuous spatial distribution. Outside of the central region of the bore 40, the field is referenced as "fringe field" having direction and intensity that depends upon the spatial region of interest. Many such superconducting magnets include windings specifically designed to attenuate the fringe fields in regions external to the magnet or to shield the interior of the magnet bore 40 from magnetic influences arising externally. As a practical matter, the fringe field within and proximate to the cryostat remain substantial, at least in certain such regions. For example, a typical (unshielded) 4.12 Tesla magnet exhibits a fringe field magnitude of about 1200 gauss at the lower cryostat housing surface and about 6 inches off axis. The fringe field distribution is a property of the specific magnet design and varies rapidly with position. Fortunately, functional ion pump operation is not unusually sensitive to magnetic field intensity and direction beyond certain minimal requirements.

In figure 1 this external, or "fringe" field is shown intercepting an ion pump 10 comprising a tubular anode (or anode array) 42 approximately along the anode axis 44. This is the preferred pump geometry. In this embodiment, the anode 42 is 1 and cathodes are located within the body of the cryostat. A hermetically sealed power connector 46 permits application of power to the anode 42. It is preferred to locate the pumping array in a region of the cryostat close to ambient temperature, whereby the power connector presents a negligible heat leak.

Cathode plates 48 are shown in electrical contact with the body of the cryostat. A circular pumping array, coaxial with the cryostat bore 40, provides a convenient geometry.

The separation of anode and cathode may be conveniently termed a line of separation. While the actual shape and relative orientation of these electrodes determines the actual electric field distribution between them at a given potential difference, it is sufficient for descriptive purposes to recognize a gross electric field direction along a line of separation. The anode and cathode are disposed to present a substantial component along the local fringe field direction. A substantially parallel orientation for these electric and magnetic (fringe) fields is desirable to produce a magnetically confined plasma discharge with axial extraction of positive ions from the tubular anode. The (average) angle to which the fields intersect will determine the efficiency of pumping.

The required pumping speed of the pump 10 is estimated from the cryostat dimensions and thermal design. Figure 1 depicts a common conventional LN₂ shielded liquid helium cryostat. Thus one has the interior surface of the containment housing 21 at ambient temperature and a nested structure of heat shields surrounding the exterior surface (inwardly to the cryostat) of the helium reservoir 24. Typically, one of the heat shields (32) is at LN₂ temperature. These structural components (usually composed of stainless steel or copper) outgas at rates depending upon the material properties, temperature and pressure as a function of time as

$$Q = Q_0 e^{-t/\tau}$$

An estimate of the initial outgassing rate Q_0 provides guidance for the requirements for the pumping element. Consider that the interior surface of the containment vessel remains an outgassing source, whereas the thermal shields and cryogen reservoirs are at very reduced temperatures and contribute negligibly to outgassing. These surfaces stabilize most residual gasses through the phenomena of cryopumping. The principle contribution therefore remains the interior surface of the containment vessel. For stainless steel, outgassing has been quoted at 2×10^{-7} torr•liters/sec/cm².

One commercially available vertical 600 MHz superconducting magnet cryostat has gross dimensions of about 1 meter (o.d.) by 1.4 meter high, of conventional construction as suggested above, and is typically evacuated to about 5×10^{-5} torr and sealed off. When filled with the LN₂ and He cryogens the internal pressure is typically about 5×10^{-8} torr. From the (internal) surface area of the containment vessel and the quoted initial outgassing rate for stainless steel, the value of Q_0 is estimated at 0.017 torr• liter/sec. As noted above the outgassing contribution to pump load decreases exponentially with time. A typical 8 liter per

second pumping array therefore pumps at about 340 times the maximum (initial) outgassing rate at ambient temperature. Such a pumping array may be disposed as an integral cryostat component. A typical 8 liter/second ion pump array such as Varian part 87-900-064-01(A) is quoted to exhibit an operational life of 40,000 hours at about 10^{-6} torr. Except during the initial cryogen filling, the internal pressure is about 2 orders of magnitude lower pressure than quoted for this performance parameter. Accordingly, the operational lifetime for the pump is expected to exceed the useful lifetime for the magnet.

Returning to figure 1 and figure 3, another embodiment is depicted. A separate dewar is arranged for purposes of experiments carried out within the controlled magnetic field of the bore 40 in the neighborhood of the central region of windings 13 where the designed magnetic field distribution obtains. It is emphasized that a variety of phenomena are investigated with superconducting magnets and these investigations are necessarily within the bore of the magnet and very frequently require a vacuum environment. The present inventive application of vacuum ion pumping is therefore facilitated by these conditions. For illustrative purposes, and particularly relevant to cryo-probe NMR investigations, the dewar includes annular portion 72 that provides room temperature space 74 for manipulation of subject matter of the experiment, i.e., introducing and/or rotating samples. In the particular application to NMR apparatus, the annular portion 72 houses a cooled RF coil 77 that closely couples to samples in the central room temperature space for excitation and detection of NMR phenomena. A "bare" ion pump 76 (without a dedicated magnet) is disposed in a selected portion of the fringe field 60 and in gas communication with dewar 70. The internal structure of the ion pump 76 is conventional. A power supply 75 is provided for communication with the pump 76 through appropriate vacuum tight couplings. While location of the ion pump 76 is limited by dimensions of the investigative apparatus, viz. dewar 70, it is only required that the fringe field 60, intercepting the ion pump 76, has average magnitude and direction for effective pump operation. For example, a standard 8 l/s ion pump body (Varian, Inc. model 9115005) operated at 5 KV requires a minimum of 400 gauss magnetic field intensity along the anode axis. It is useful to provide an angular degree of freedom for support for the pump to optimize by adjustment the average orientation of the pump structure in respect of the fringe field.

In one example of this apparatus, the vacuum space of the dewar 70 represents a volume of about 1.5 liter. Communicating with this vacuum space, an 8 liter/sec ion pump (without a pump magnet) was disposed about 6 inches under a cryostat housing an unshielded 4 Tesla magnet and radially displaced by about 3 inches from the outer periphery of the bore 40 of the magnet. The intensity of the fringe field was found to vary by about 30% over the space

occupied by the ion pump. Satisfactory vacuum conditions for thermal isolation were readily achieved. In operation over a period of weeks, ion pump current indicated a vacuum pressure of $< 10^{-6}$ torr.

Another benefit has been found for the inventive embodiment of cryo-dewar housed RF coils for NMR measurements. Turning now to figure 4, there is shown a scatter plot of observed noise data superimposed on a plot of the pressure as a function of time as the NMR dewar is pumped. Residual hydrogen is very effectively removed from the vacuum environment by burial and diffusion in the lattice of the cathode through ion pumping. Consequently, the electrical discharge noise, incident upon occurrence of the RF pulse for an NMR measurement, decreases as the partial pressure is further reduced by ion pumping. For a clear showing of the benefit of the ion pumping in this environment, portions of NMR spectrometer system noise corresponding to the scatter plot points A and B are shown at figure 5a and 5b. The solid line of figure 4 represents a trace of ion pump current (proportional to pressure) and expresses the steady pressure reduction as a function of time as a result of operation of the invention. At different pressures, NMR system noise was acquired and the baseline noise parameter was extracted by operation on receiver response (but otherwise devoid of sample response). The noise parameter was extracted consistently through operation of the "noise command" algorithm whereby the RMS background signal is acquired over a large number of points (typically, 10^5) at regular time intervals. As an initial step, the NMR system receiver establishes thermal noise amplitude in a quiescent receive mode. This reference amplitude then defines marker amplitudes selected for 10% added noise, e.g. 110% of thermal noise (square data points) and 200% thermal noise (round data points). The noise-relative attenuation and the pressure are the coordinates of the scatter plot distribution of points appearing in figure 4. This data was obtained by increasing the RF power applied to a decoupler channel (in db) from 16 db to 48 db in 1 db increments (with no sample present). The (transient) noise was then recorded in the observe channel (and appears as peaks in figures 5a and 5b). The lowest power level where the noise level exceeded 110% of the background noise is taken as the 10% added noise crossing. Similarly, when the noise level exceeds 200% of the background noise, the power level is recorded. These operations were repeated many times while continuously pumping on the NMR probe dewar and the vacuum pressure (ion pump current) recorded for each such noise determination. The 110% noise threshold (square data points) and the 200% noise threshold (round data points) are each then plotted (right ordinate scale) against time (as a proxy for pressure). The vacuum pressure (left ordinate) is likewise plotted against time and it is evident that as the vacuum pressure drops, both the 10% and 200% added noise thresholds tend to increase. That is, incremental noise over

and above the prevailing system thermal noise is demonstrated with greater pulse power: Consequently, it is the (incremental) transient noise that is decreasing as the vacuum pressure is continuously reduced. It is believed that such transient noise is accounted for by the instantaneous (very) dilute plasmas of light gases formed during RF pulse intervals in the decoupler channel of the NMR transmitter. As the partial pressures of these gasses are further pumped, this measurable noise source is reduced as demonstrated.

The reduction in noise contribution is readily recognized in comparison of noise phenomena corresponding to the high pressure datum A and the relatively low pressure datum B as shown in figures 5a and 5b. These figures directly exhibit the noise amplitude in the observe channel of the NMR receiver. The ion pump itself had no effect upon actual NMR spectra as determined from pump on /pump off comparison.

The application described above for this embodiment particularly benefits from active pumping. It is believed that free hydrogen is produced from the dissociation of residual water vapor through collisions of energetic molecules deriving kinetic energy from the RF power pulses. The present invention provides the desired pumping without imposition of the magnetic field of an additional magnet, specific to the pump.

The exploitation of the fringe field of high field magnets for operation of vacuum ion pump apparatus is found to provide the necessary magnetic field for effective pump operation. In this manner there is no requirement for a separate, bulky and heavy ion pump magnet specific to ion pump operation. The invention is not limited to cryo-statically housed magnets: fringe fields of room temperature magnets in diverse applications may supply the magnetic fringe field environment suitable to ion pump operation in a vacuum vessel proximate said magnet. Although NMR apparatus has been discussed here, other analytic instruments requiring substantial magnetic fields will also benefit from the invention.

Although this invention has been described with reference to particular embodiments and examples, other modifications and variations will occur to those skilled in the art in view of the above teachings. It should be understood that, within the scope of the appended claims, this invention may be practiced otherwise than as specifically described.

WHAT IS CLAIMED IS:

1. A vacuum ion pump in combination with a cryogenically cooled magnet, comprising:
 - (a) a non magnetic axially symmetric anode having at least one aperture therein for defining a glow discharge;
 - 5 (b) a non-magnetic chemically active cathode spaced apart from said anode and having a surface opposite said at least one aperture;
 - (c) a non-magnetic evacuable housing in gas communication with a region to be evacuated, said housing surrounding said anode and cathode; and
 - (d) a power source that apply a potential difference between said anode and said cathode
 - 10 establishing an electric field therebetween,
said housing disposed intercepting a portion of the magnetic field intensity of said magnet along a direction substantially parallel to said electric field and said portion characterized by sufficient magnetic field intensity magnitude that operate said ion pump.
- 15 2. The vacuum ion pump of claim 1, wherein said magnet is located within a cryostat comprising at least one evacuated volume.
3. The vacuum ion pump of claim 2, wherein said housing comprises said at least one evacuated volume.
- 20 4. The vacuum ion pump of claim 1, wherein said region to be evacuated is independent of said cryostat and said region to be evacuated is disposed proximate said cryostat.
5. The vacuum ion pump of claim 4, wherein said another evacuated region is in gas
- 25 communication with a dewar enclosing an RF coil for use in acquisition of NMR data.
6. The vacuum ion pump of claim 5, wherein at least a portion of said cryostat comprises a bore surrounded by windings comprising said magnet and said dewar is disposed in said bore.
- 30 7. An NMR probe for use in the bore of a superconducting magnet having a substantial fringing field external to said bore, said probe comprising:
an RF coil;

an evacuated cryostat housing comprising an annular portion which receipt in said bore, wherein said RF coil is disposed within the annular portion, said annular portion further comprising an a probe bore accommodating a sample of investigation;

refrigeration apparatus disposed in said housing that remove heat from said RF coil;

5 a tubular anode disposed in said housing, said anode having an axis and said anode in substantial parallel alignment with a local portion of said fringing field; and

a cathode oriented to present a surface having a normal substantially coincident with said axis, said cathode and anode arranged in support a potential difference therebetween.

10 8. The NMR probe of claim 7, wherein said cathode comprises a chemically active metal selected from the group consisting of titanium, vanadium, tantalum, and zirconium.

9. The NMR probe of claim 8, further comprising a power supply to supply said potential difference.

15

10. The NMR probe of claim 7, wherein said cathode and anode are disposed in another portion of said housing in open communication with said annular space.

20 11. A method of maintaining a vacuum condition within a cryostat housing for thermally isolating a cooled magnet, said magnet having a field distribution consisting of a major portion and a fringe field portion, comprising the steps of:

disposing a non-magnetic chemically active anode spaced apart from a nonmagnetic chemically active cathode in gas communication with said housing and within said fringing field;

25 maintaining an electric field between said anode and cathode, and

orienting said anode and cathode such that said electric field is substantially parallel to said fringe field, whereby residual gasses are sequestered under the combined influence of said fringe field and said electric field.

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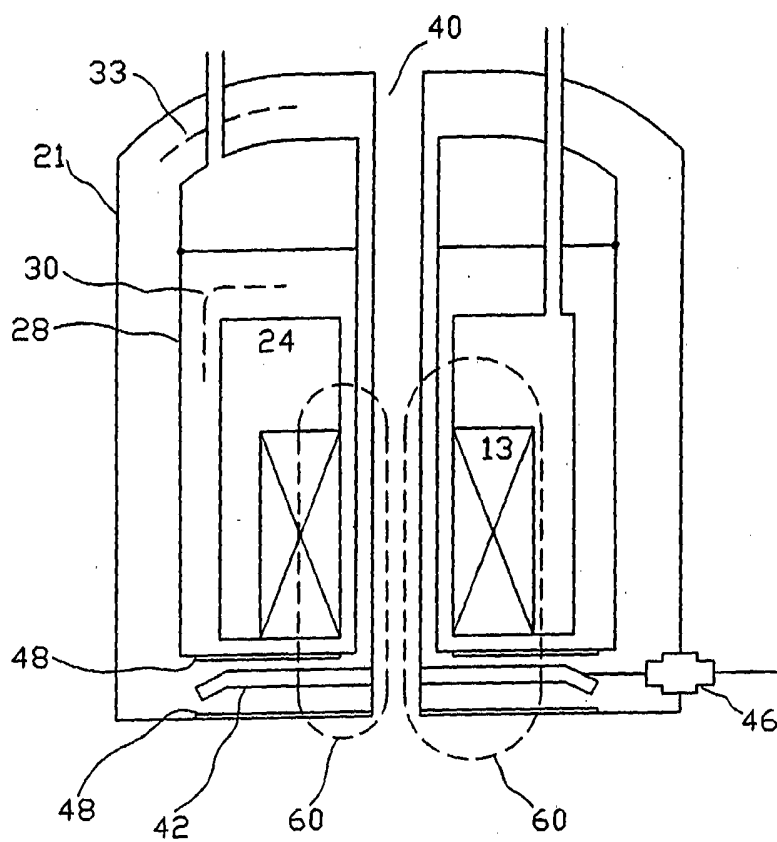


Fig. 1

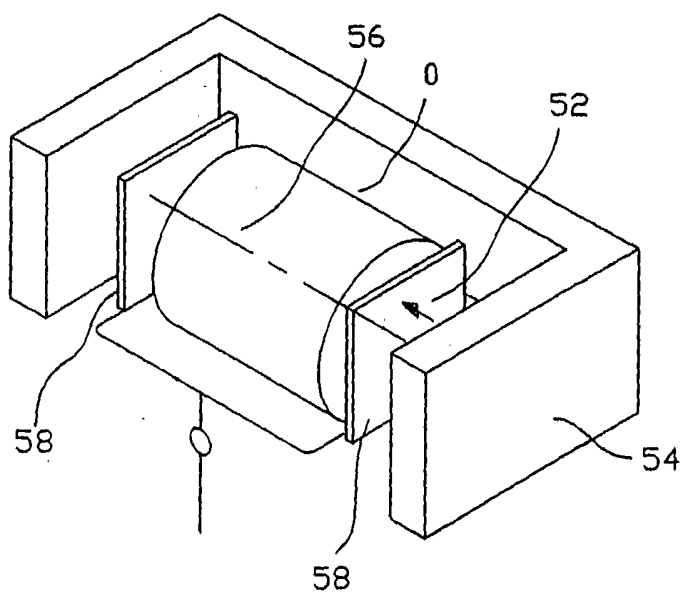


Fig. 2

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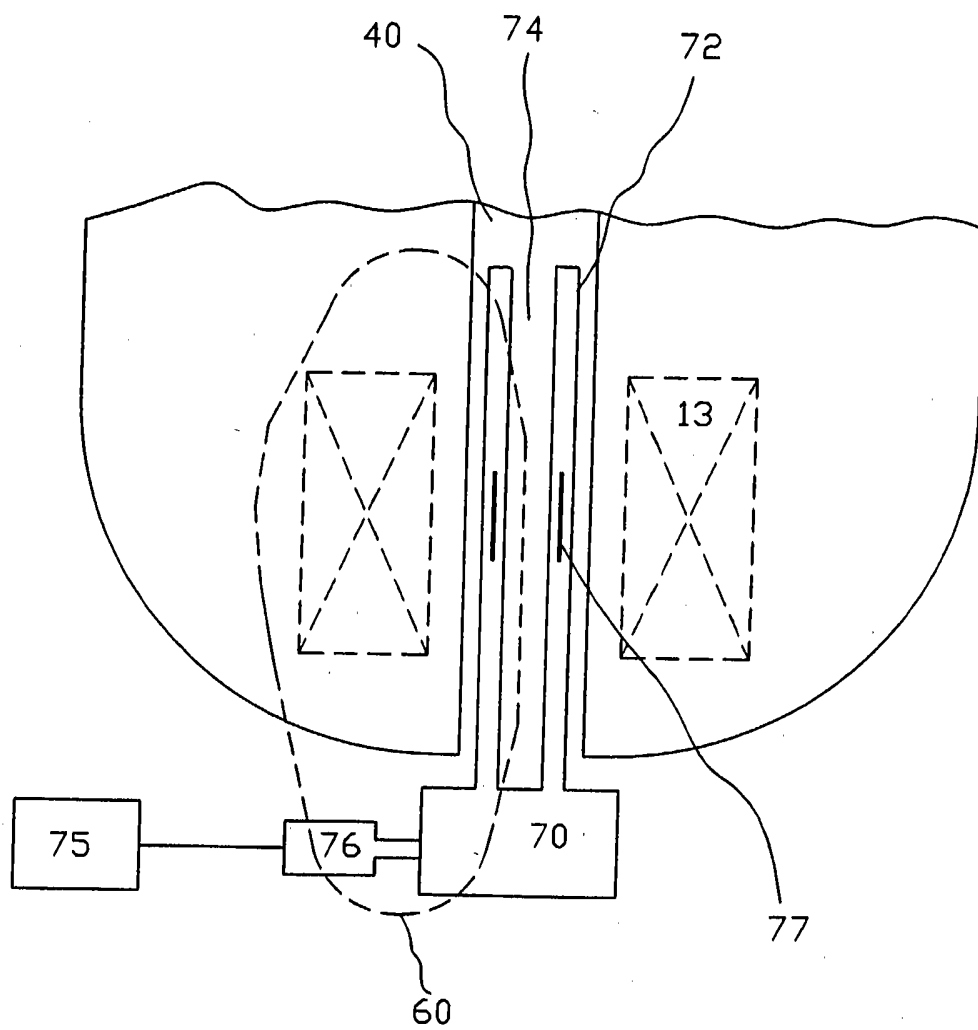


Fig. 3

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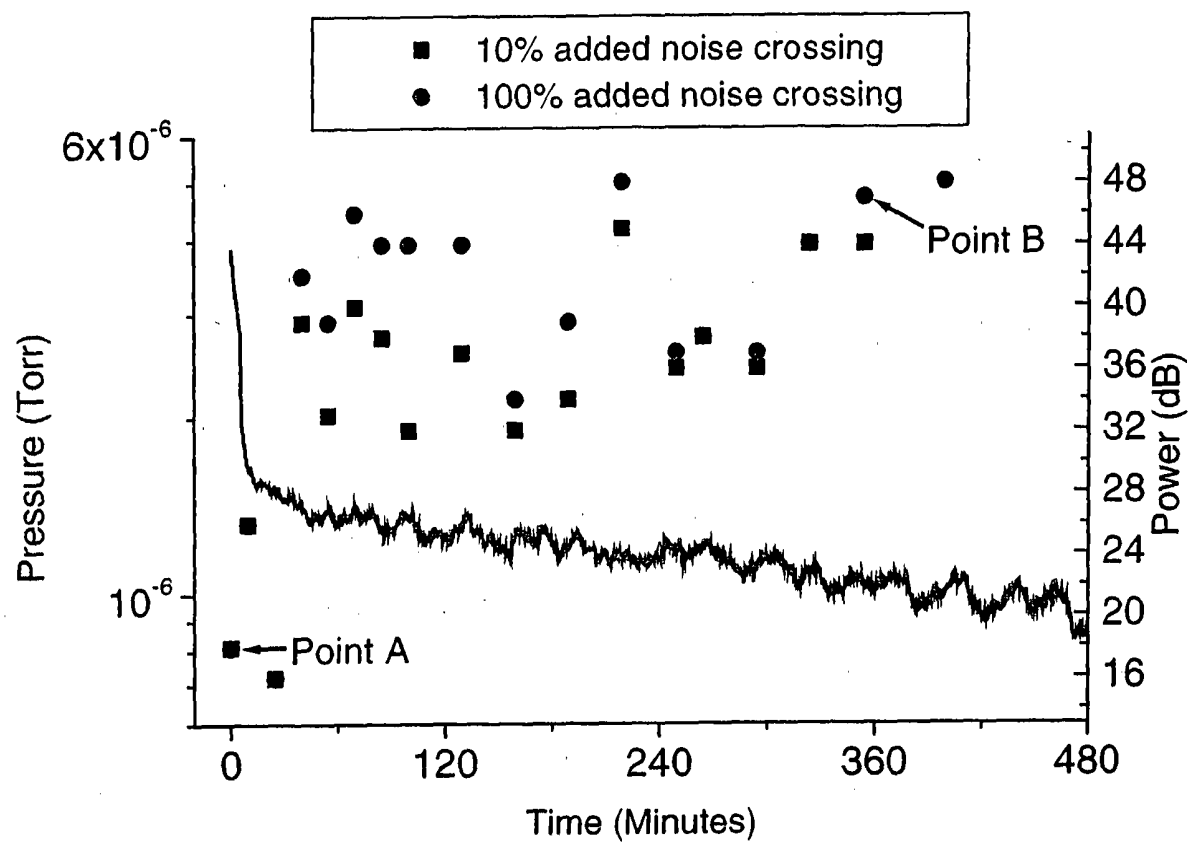


Figure 4

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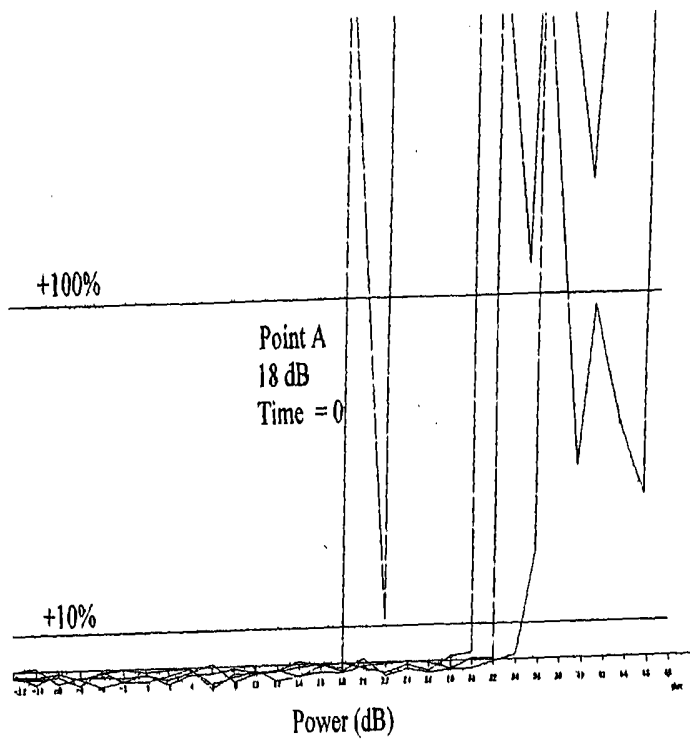


Figure 5a

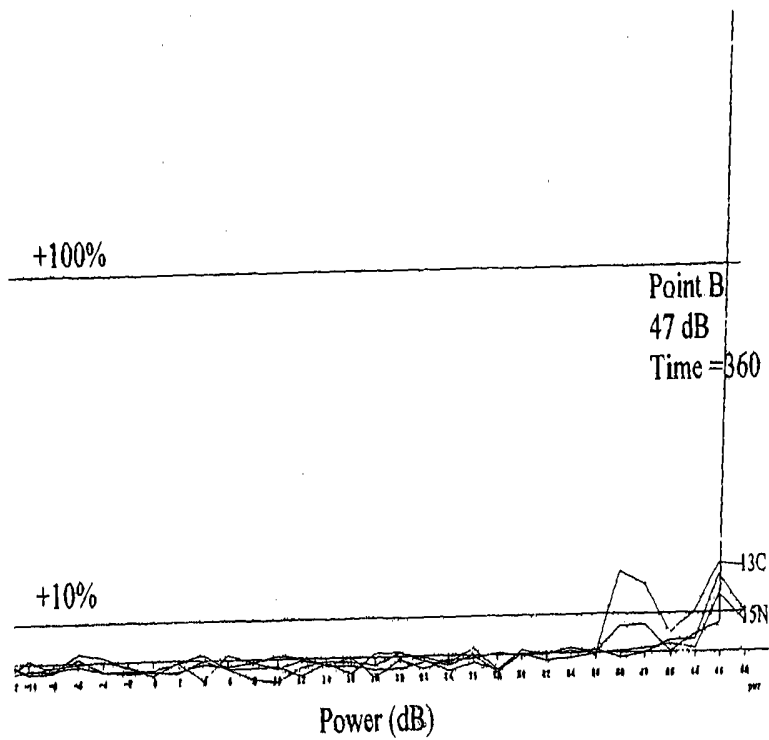


Figure 5b