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MEASUREMENT OF OMNITRON CRYOPUMPING AND OUTGASSING BY 10-16 TORR PULSE COUNTING MASS FILTER

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April 1968

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MASS FILTER

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ABSTRACT

The Omnitron is a new type of synchrotron accelerator with storage ring that will deliver beams of light and/or heavy ions to different experiments almost simultaneously. Storage of heavy ions in a high charge state for 20 msec requires that the vacuum environment be assessed more carefully than can be done by the 'sum of the partial pressures' convention. A crude figure of merit,  $10^{14} \leq N \cdot \sum_i n_i Z_i^2$  is used where:  $N = 3 \times 10^7$  turns around the accelerator,  $n_i$  = the concentration of atoms of the  $i^{\text{th}}$  atomic number  $Z_i$ . To insure that this figure of merit is met, outgassing from materials and pumps and pumping speeds are assessed by counting ions from a quadrupole mass filter that can indicate average concentrations of light gas to  $< 10^{-16}$  torr and  $< 10^{-15}$  torr of ions with  $m/e$  up to 500. Using this together with an accumulation method, contamination coming from oil D.P. and M.P. systems having special LN traps and valves is shown to be negligible. For the 7" D.P. system, no  $m/e > 44$  could be detected at an average sensitivity of  $10^5$  molecules/sec. An average of  $10^8$  molecules/sec with  $m/e > 44$  were detected coming from the M.P. system. A 304-type stainless-steel tube, 3 in. dia. x 0.065 in. wall x 17 in. length cleaned in a chemical process made by the Diversey Co., was pumped down to the Omnitron figure of merit in  $< 5$  hrs. (without bakeout or cooling) using  $4^{\circ}\text{K}$  cryopumping and the clean D.P. system.

## Measurement of Omnitron Cryopumping and Outgassing

By  $10^{-16}$  Torr Pulse Counting Mass Filter \*

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In colloquial terms, the Omnitron<sup>1,2</sup> will require a clean  $10^{-10}$  torr vacuum. Because of the threat to the ion beam posed by small concentrations of high atomic number and high molecular weight species, we must depart from convention. Despite our concern about contamination, we give evidence here supporting our plan to use diffusion pumps and mechanical backing and roughing pumps charged with inexpensive hydrocarbon oil--but first a brief description of the Omnitron facility.

Although the Omnitron (plan view, Fig. 1) has not received final approval for construction, it seems likely that five years from now the hill site chosen within the Lawrence Radiation Laboratory will be graced by its operating presence. The name--Omnitron--was chosen because the machine will serve nuclear chemistry, biomedical and medium energy physics users, with pulsed and dc beams of different types of ions at different energies and currents delivered almost simultaneously to different target complexes. For example, for biomedical purposes small beam currents of monoenergetic, highly-collimated heavy ions in a high charge state at energies of up to 500 MeV/nucleon will be delivered. At other target locations, 1.4 GeV protons at currents up to  $10^{13}$  ions/sec will serve physics experimenters; all the while, nuclear chemists will use a wide variety of lower-energy, heavy-ion beams at currents

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\* This work was done under the auspices of the U.S. Atomic Energy Commission.

up to  $10^{11}$  ions/sec. If constructed today, the machine, together with its experimental facility would cost about \$26 000 000.

From a vacuum point of view, the Omnitron consists of injector and target systems of comparatively dirty vacuum at  $5 \times 10^{-6}$  torr, communicating through open ducts to clean vacuum rings at  $10^{-10}$  torr wherein the beam is accelerated and stored. Figures 1 and 2 show an arrangement of these parts. Beam injection at 60 pulses/sec occurs vertically downward into a 60 Hz synchrotron. Particles then orbit  $\sim 10^7$  turns before being either delivered to a target system or transferred into a beam storage ring concentric with the synchrotron. Bunches of particles can be held in the storage ring and either reinjected into the synchrotron for additional acceleration (after charge stripping through foil) or these bunches can be allowed to spread in phase space permitting a dc beam to be dribbled out of the storage ring to a target.

As a preface to specifying the vacuum required, some remarks on a common sense level may be of interest. First, the kind of gas encountered by the beam can be more important than the gas concentration (of course, the ion beam does not 'see' the gas pressure at all). How is the Omnitron vacuum to be measured?<sup>3</sup> Even using a mass spectrometer we should not be too cavalier. For example, experimental measurements<sup>4</sup> show that beam attenuation by charge exchange with hydrogen is about as serious as attenuation by charge exchange with carbon monoxide. Indeed, predicting beam attenuation by charge exchange with residual gas is not feasible from first principles. Rather, experimental determinations must be made, but until the Omnitron (or equivalent) exists, extrapolations must be relied upon. These show that beam losses by

charge exchange will be a complex function of the atomic number, charge state and energy of the beam ions, as well as the atomic number concentration of the residual gas. To err on the safe side (except in the case of hydrogen), a crude figure of merit,  $N \sum \bar{n}_i \cdot Z_i^2 \leq 10^{14}$ , serves to set upper limits on the product,  $\bar{n}_i \cdot Z_i^2$ , where  $N \cong 3 \times 10^7$ , the number of turns around the machine that the beam takes going through the average partial atomic concentration (atoms/cm<sup>3</sup>)  $\bar{n}_i$  of atomic number  $Z_i$ .

In place of a conventional approach to vacuum specification, a system's approach is adopted where the vacuum is defined as an environmental quality. The interaction among pumps, plumbing, direct and indirect beam effects, and measuring devices is taken into account. Short of cooling the whole Omnitron down to effective cryopumping temperatures (Unfortunately, superconducting magnets that operate at 10 kG peak and 60 Hz are not at hand), pumping the two ultrahigh vacuum rings (Fig. 2) must be done through the 181 gaps separating magnets. We are thus constrained to pump at intervals spaced more than 10 pipe diameters apart. Under these conditions, performance of the system is dominated by wall outgassing, especially by H<sub>2</sub>O and other molecules that linger on the walls at each encounter. Thus much larger concentration gradients of high-effective atomic-number contaminants are possible than predicted by conductance only. Clearly then, pump backstreaming and outgassing during starting, steady operation, and as a consequence of accidents or malfunctions, must be considered. Having said this, why are we proposing to use oil-diffusion pumps and oil-sealed mechanical-displacement pumps-- even to support high-speed cryopumping? We offer evidence (below) to support that we can 'make a silk purse out of a sow's ear.' Using existing hardware



that we would not wish to duplicate if we started from scratch, we have assembled an outgassing and pump backstreaming tester with in situ calibration features shown in Fig. 3. Modification of the commercial, quadrupole mass filter to permit ion counting is the core of this apparatus and we discuss this instrument next.

## II. ION COUNTING WITH THE MASS FILTER

Discussing the strengths and weaknesses of ion counting and the details of the electric circuits and mechanics involved is far beyond the scope of this paper. In a nutshell, this arrangement for counting ions gives a digital readout, and using multiple scans can provide a signal-to-noise ratio of  $> 10^6$ , can yield  $< 1$  noise count/min, can record the total number of ions collected at each  $m/e$  position as a function of time, permits observing adjacent  $m/e$  peaks that differ in total counts by  $10^6$ , and permits scanning mass spectra at 50 a.m.u./0.12 sec. (Note that signals may be followed far below the ambient level.) Based upon a year's experience ironing out troubles, the major drawback so far is that the signal-to-noise ratio from the multiplier decreases with time.

A brief sketch of essentials follows: Gas-phase molecules are ionized by electron bombardment, and the resulting ions are filtered by the quadrupole electric lens into groups according to their mass-to-charge ratio. If the numbers of ions per group is kept sufficiently small, each ion produces a discrete pulse when the ion hits the first stage of the 20-stage electron multiplier. The maximum rate at which single ions can be observed is probably  $\leq 10^8$ /sec and is determined by the integrating effect of the capacitance of the anode lead from the electron multiplier. Figure 4 is a block diagram

showing how ion pulses are sorted from noise pulses and analyzed. Most noise pulses are smaller in amplitude than ion pulses, a discriminator lets only pulses above a fixed threshold come through. Ultimately, the pulses are stored in the memory of the 400 channel pulse height analyzer operating in the multiscaling mode. The counts stored in this memory are displayed on an oscilloscope screen as a dot pattern corresponding to the mass spectrum. Contents of this memory can be printed or punched on paper tape or recorded on magnet tape. Limitations on accuracy, scan rate and sensitivity may be understood as follows: The pulse height analyzer used accepts a maximum of  $3 \times 10^5$  counts/sec and has 400 channels. If 57 mass peaks are to be recorded, 400/57 channels are then available/mass peak.<sup>8</sup> Since statistical accuracy varies as  $1/\sqrt{N}$ , where: N is the number of counts/peak, 200 counts/peak is about the minimum acceptable. A single scan of 57 peaks was done in a minimum of 0.12 sec. (Note: Commercial analyzers accepting up to  $10^8$  counts/sec and/or having far more than 400 channels are available.)

### III. USING THE TESTOR

Figure 3 is a schematic cross section of the essential features of the outgassing and backstreaming testor. The ion counting quadrupole is within side arm (1). Samples of vacuum plumbing (3) are welded on and cut off routinely with a special can opener and welding tool.<sup>3</sup> The system is let up to atmospheric pressure by sucking in liquid nitrogen through a bronze filter (not shown) to keep out ice crystals. Roughing is done by an oil-sealed mechanical pump operating through a 'U' trap (6) with heavy copper walls whose temperature is insensitive to change in LN level. This stainless

steel roughing line has all metal valves between the system and trap and is always kept at 200°C.

The first tests made examined the roughing system and D.P. system as sources of contamination. The measuring dome (Fig. 3) has an internal tubular ring (not shown) that may be heated or cooled and an all stainless steel poppet valve (4) carrying two small orifice holes. Thus by shutting this poppet valve, either one or the other, or both or none, of the holes can be open. Three known flow rates can be thus obtained in addition to isolation between the diffusion pump being tested and diagnostic gear. By heating and then cooling the tubular ring to 520°K and < 80°K, thus providing an accumulator for a known time, and opening and closing the poppet valve, contamination already in the dome can be distinguished from contamination steadily entering the dome. Approximately  $10^8$  molecules/sec of  $m/e > 44$  came from the roughing system (This amount from roughing was  $\ll$  than the amount already in the sample tube initially.) under free molecular flow. No input flux of  $m/e > 44$  could be detected coming from the D.P. at an average sensitivity of  $10^{5-8}$  molecules/sec.

#### IV. CALIBRATION OF THE ION COUNTING MASS FILTER

An in situ calibration procedure permitting a quick check of output signal linearity, sensitivity, resolution, signal-to-noise ratio and ion counts in terms of numbers of molecules is followed. The central idea is to compare apparent pumping speeds in steady state to absolute pumping speeds during free gas expansion. Two small orifices are used, each in turn to obtain three known speeds. Leak-up rates proportional to outgassing into the dome are determined by closing valve (5) and recording ion counts as a function of time. Some of the peaks thus obtained are composites that can be

identified by searching a library of known spectra with a regression<sup>6</sup> computer code. Next, apparent pumping speeds in steady state are taken using the leak-up rates. True pumping speeds are then obtained by closing (5), accumulating gas and then opening (5) quickly to achieve a free-gas expansion through the orifices. By keeping the walls of the system at  $> 200^{\circ}\text{C}$  (except for  $150^{\circ}\text{C}$  around the electron multiplier) and because the geometry is appropriate, well known simple formulas can be applied. In future, more exact expressions will be tried (for example Hobson and Earnshaw's<sup>5</sup>). Dividing the apparent speeds by the corresponding true speeds gives the calibration constants.

#### V. CRYOPUMPING MEASUREMENTS

Figure 2 shows a third ring carrying a 1 in. dia.  $4^{\circ}\text{K}$  tube shielded by an  $80^{\circ}\text{K}$  system. Pumpout ducts connect this system to the pill box pumpouts between magnets. A single full size pumping station has been tested for the past 1-1/2 years. As yet the ion counting has not been applied to this system, but many oscilloscope traces of mass spectra using the commercial mass filter have been taken. Pumpdown of this all stainless steel and alumina station using constant temperature LN traps on an oil D.P. and on an oil-sealed M.P. with a system speed on 1 liter/sec required 30 minutes from 1 atmosphere to  $5 \times 10^{-6}$  torr.  $4^{\circ}\text{K}$  cryopumping then commenced and without bakeout, Omnitron conditions were achieved 7 hours later. This result was due to treatment of the stainless steel (below) by dipping in a patent process (No. DS-9 sold by the Diversy Co., Chicago).

VI. OUTGASSING MEASUREMENTS

One Diversey-processed<sup>3</sup> piece of 304 stainless steel tubing (3 in. O.D. x 17 in. long with welded eyelet joints<sup>3</sup>) has been tested. Omnitron spec's were achieved in < 5 hours from air, without bakeout or cooling. Several other samples will be run on our testor in the immediate future.

REFERENCES AND FOOTNOTES

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4. NIKOLAEV, V. S., 1965, Sov. Phys. Usp., 8, 2, 269-94.
5. HOBSON, J. P. and EARNSHAW, J. W., 1967, J. Vac. Sci., 4, 5, 257-273.
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7. All walls and valves were at  $> 200^{\circ}\text{C}$  except  $150^{\circ}\text{C}$  around electron multiplier.
8. The E.A.I. commercial mass-filter power supply does not permit an equal number of mass peaks/channel in a given scan range, i.e., if  $m/e = 2$  uses 3 channels,  $m/e = 12$  requires 4,  $m/e = 18$  requires 6,  $m/e = 28$  requires 7,  $m/e = 44$  requires 7,  $m/e = 57$  requires 7. This sort of nonlinearity is easily adjusted for. Also, when scanning in the high range, resolution must be sacrificed to keep sensitivity within a factor of 10.

FIGURE CAPTIONS

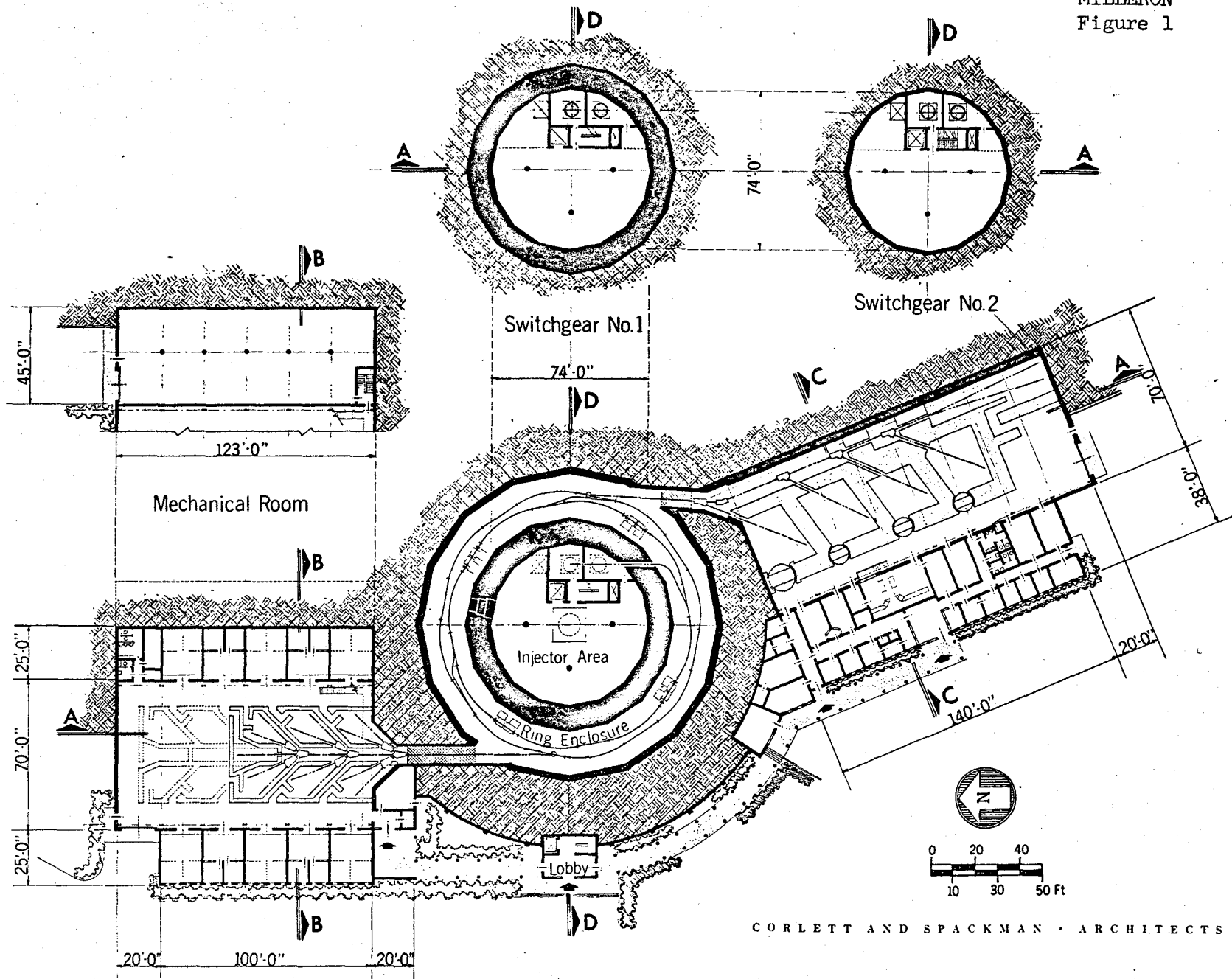
Fig. 1 Plan View of the Omnitron Facility.

Fig. 2 Vacuum System Schematic.

Fig. 3 Outgassing and Backstreaming Testor.

1. Quadrupole gas analyzer.
2. Ion gauge
3. Outgassing sample
4. Measuring orifice
5. Fast poppet valve
6. Liquid nitrogen trap
7. Water cooled baffle
8. Oil diffusion pump
9. Liquid nitrogen trap roughing line

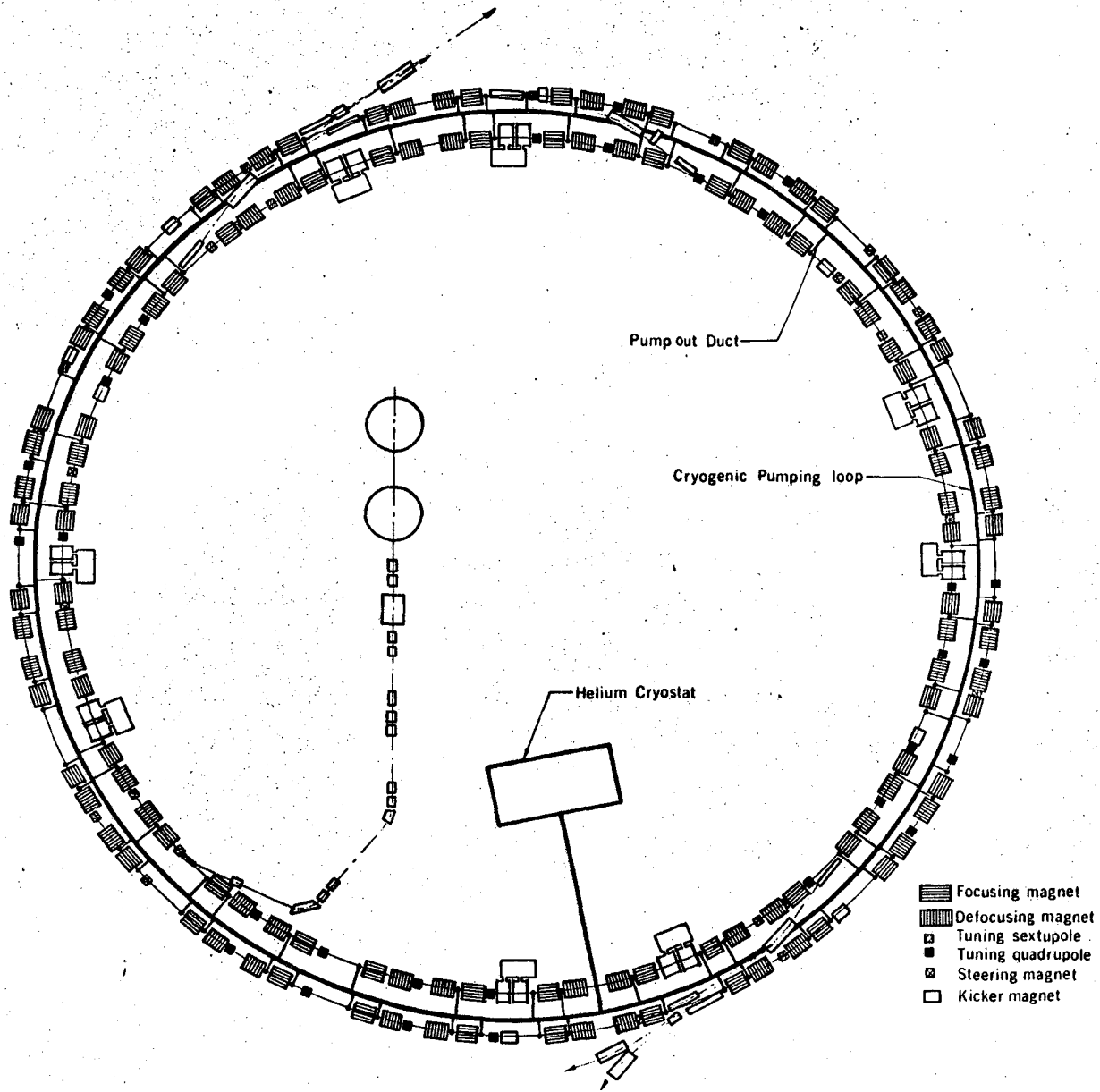
Fig. 4 Block Diagram, Ion Pulse Counting.



Plan View of the Facility

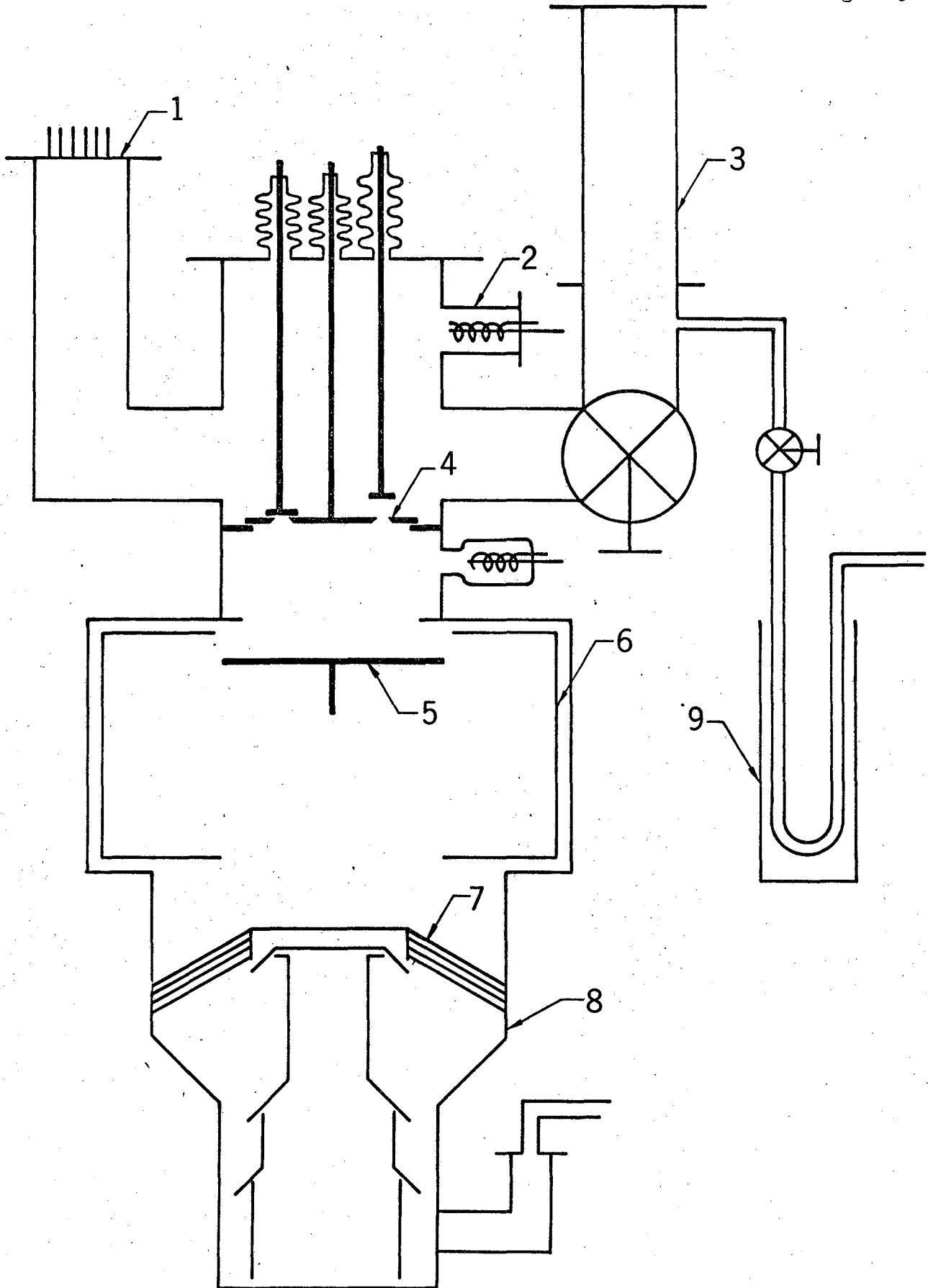
Fig. III-1

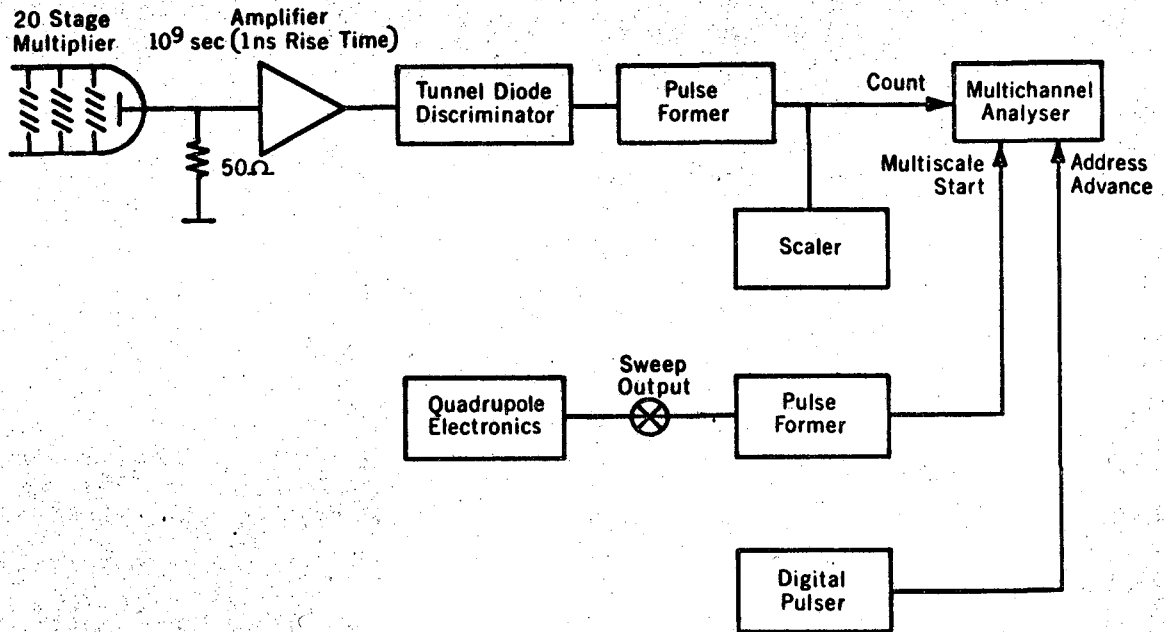




Vacuum System Schematic

MILLERON  
Figure 3





ION PULSE COUNTING

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