

# ULTRAHIGH VACUUM: A SURVEY

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Somewhat over a decade ago certain problems in the physics laboratory instigated a major step forward in vacuum technology. These problems were in the fields of atomic collision processes and surface physics in which it was impossible to obtain either high gas purity or atomically clean surfaces with the experimental techniques available. For example, at a pressure of 10<sup>-7</sup> Torr (1 Torr is approximately = 1 mm Hg) of molecular gas, a surface which had been previously cleaned would adsorb a complete monolayer of gas in a matter of seconds. The combined efforts of a number of physicists<sup>1</sup> resulted in a new set of tools which made it possible to achieve and measure pressure two or three orders of magnitude lower than was previously possible, among them Nottingham of MIT and groups at three major laboratories including Apker at General Electric, Lander and Becker at Bell Telephone Laboratories, and several of us at Westinghouse Research Laboratories. That is, pressures down to 10<sup>-10</sup> or somewhat lower.

While we immediately recognized the significance of the ultrahigh vacuum for the fields in which we worked, for example we could now maintain and therefore investigate atomically clean surfaces for hours, days or even weeks, we could not have anticipated the much wider implications of the new vacuum technology in such fields as plasma physics, high voltage accelerators, vacuum metallurgy, semiconductor surfaces and many other applications. Nor could we anticipate, for example, that within a few years we would be talking seriously of sending a man to the moon and of directly studying the properties of the low pressure region in between. As it has turned out, it seems that in every application, if high vacuum is good, ultra-high vacuum is far better, and it is quite commonplace for vacuum equipment manufacturers to vie with each other as to who holds the record for low pressure attainment. It should not surprise us, therefore, if at times the claims for certain instruments or pumps should reflect a commercial enthusiasm rather than a candid appraisal. In a number of cases the stakes are scientific rather than commercial; the validity of a given experiment may hinge on the reliability of the instruments used for pressure measurement. Since the last several years have witnessed not only a number of advances in the state of the art but also a new recognition of the limitations of our knowledge in the field, it should not be surprising if people not experts in the field are confused as to where the limits of low pressure attainment stand today. It is therefore my intent to try to summarize what has happened in this decade of technological development. This is a very ambitious project, and I cannot hope to do justice to all the contributors in the field; however, I will try to outline the major directions of activity. I will also try to identify some of the problems of low pressure physics and chemistry which are currently tied in with these questions.

To review for a moment: what were the principle advances of a decade ago which introduced ultra-high vacuum? First of all, there was a recognition that the limitation which prevented us from going below  $10^{-8}$  lay in the measuring instruments and not in the means for producing high vacuum. This soon led to the invention of at least three gauges for measuring lower density. Secondly, there was a recognition of the principle sources of gas in a vacuum system. These were (1) the desorption of gases from contaminated surfaces, (2) the diffusion of gas through the solid walls of the enclosure, and (3) and perhaps most painful, the backstreaming

of gases and vapors into the vacuum system from the diffusion pumps, used almost universally to achieve high vacua. The third set of advances involved what I call "system techniques". These included new vacuum components such as all-metal valves, traps, demountable seals, and manometers as well as a method of putting them together which made it possible to reproduce ultra-high vacuum conditions in a straightforward manner. An example of a typical system of the early type is shown in Figure 1. Many of them are, of course, in use today.

What has happened since 1953? Among the truly impressive contributions has been the development of all-metal system techniques which are flexible, demountable and capable of almost any size you can pay for. Whereas the size of such systems as shown in Figure 1 are obviously limited by the glassblowers' art, systems of the type shown in the next figure, Figure 2, can be built in almost any size, and ultra-high vacuum systems are being built in which you can place an entire satellite for test, and in some cases the whole rocket vehicle as well. The development of these techniques was strongly accelerated by the needs of the Sherwood plasma physics program, and particular credit should be given to Don Grove and John Mark of the Princeton-Westinghouse-RCA group; 2a, b but there were many other contributors, among them, Lange at Westinghouse,<sup>3</sup> Bills of Granville-Phillips, <sup>4</sup> Wheeler, Lloyd and Zaphiropolous of Varian Associates, <sup>5a,b,c</sup> and others. During these years there have also been very significant contributions in pumping methods, both in standard approaches and in new ones. For example, among the standard approaches, the design of diffusion pumps, both oil and mercury pumps, has been significantly improved, to reduce backstreaming. Primarily, these advances

have been due to the application of good common sense and ingenuity. There have also been developed new organic fluids with less cracking into molecular contaminants. We also have new traps, particularly so-called molecular sieve traps proposed by Biondi,<sup>6</sup> which operate at room temperature to reduce the backstreaming of oil.

Among the new approaches, perhaps not new in principle but certainly new in broad utilization, are two classes of pumps in which the gas is not removed from the system, but is transferred from one part of the vacuum chamber to another part of the same enclosure. In one class of such pumps, molecular gases are adsorbed on surfaces, either on active metals like titanium at room temperature or on any surface at very low temperatures. While the pumping speed for such gases is highly selective as to the gas and dependent on the nature and condition of the solid surface, speeds of several liters per second per square centimeter are possible. This represents a very high rate of gas removal since total speeds of hundreds of thousands of liters per second can be achieved in systems of modest size.

A second class of more recently developed devices combines the removal of gases due to chemical attachment with the removal of gases in ionized form, that is by electrically driving the ionized gas into metal surfaces. Noteworthy is the sputter-ion pump, now widely used due to the contributions of Hall<sup>7</sup> and Jepsen.<sup>8</sup>

What are the ultimate pressures which can be achieved with these various methods? I have tried to summarize these in Figure 3, though I present this listing with some trepidation lest it be misinterpreted as a comparison of the absolute merits of the various pumping methods. In

general, the lowest ultimate pressure which has reliably been reported, and that is what is listed here, is only one of the parameters used in the selection of a given system. In every case but one, that of large metal oil diffusion pumps, the ultimate pressure is at or below the ultimate limitation of the Bayard-Alpert gauge, the only instrument widely used in every major laboratory. Hence, the ultimate pressure reported is not necessarily attributable to the given method of producing low pressures but rather to the method used for measuring it. For these reasons I have listed also the type of manometer used. These include, in addition to the Bayard-Alpert gauge, the suppressor ion gauge due to Schuemann,<sup>9</sup> the improved omegatron due to Klopfer,<sup>10</sup> the Davis and Vanderslice magnetic deflection mass analyzer, <sup>11a,b</sup> and the Lafferty ionization gauge.<sup>12</sup> will discuss these in detail in a moment but will comment in passing that pressures below 5 x 10<sup>-11</sup> Torr have been reliably measured in only a small number of laboratories, and in each of the cases listed here by the person who designed the manometer himself.

My summery comments are these: in several instances the lowest pressures have been achieved by a combination of two or more pumping techniques. For example, Davis reached a total pressure of 10<sup>-12</sup> Torr by combining the sputter-ion pump with the adsorption pumping of a clean tungsten surface. It seems reasonable to believe that in combination with other methods cryogenic techniques offer the possibility of reaching the lowest pressures of all. Experiments by Gomer<sup>13</sup> and by Hobson,<sup>14</sup> in which the entire vacuum chamber was immersed in liquid helium, indicated extremely low pressures as inferred from other measurements such as those of field emission. However, the lowest direct measurements of total pressure

of which I am aware were made by Lafferty, who combined ion pumping with refrigeration at liquid nitrogen temperature to achieve a value of approximately  $4 \times 10^{-13}$  Torr.<sup>11</sup>

So we see that as was the case a decade ago the state of the art has advanced to the limits of the ability of widely accepted gauges to measure pressures. Many experiments in ultra-high vacuum demand five or six reliable gauges on a single vacuum system, but for some of the new gauges I have listed here there do not exist five or six instruments in the world. Yet they clearly determine the next steps forward in this field, and it is thus desirable to review what has happened in pressure measurement since the introduction of the inverted ionization gauge and the simplified omegatron by Bayard, Buritz and others of our group in the early 1950's.

Let us recall the considerations which led to the Bayard-Alpert gauge. Figure 4 shows a schematic diagram of the old triode ionization gauge, commonly used for the measurement of pressure before 1950. In this device electrons from a hot filament cathode are accelerated through a grid and form ions whose number is proportional to the density of the neutral molecules in the grid-collector volume. The ion current to the negatively charged collector is thus a measure of the density and hence the pressure within the enclosed volume. However, over many years of experience, it was found that no matter how long one outgassed the gauge or how carefully one designed and prepared the vacuum system, the reading of such a gauge never fell below a value of  $10^{-8}$  Torr, and a number of workers became aware of the fact that there was a residual current which did not seem to be related to the pressure. It was Nottingham who first

proposed the so-called X-ray hypothesis to explain this residual current. He suggested that when the ionizing electrons impinge on the grid, they produce soft X-rays which in turn release photoelectrons from the collector. The flow of electrons from the collector thus produces a current of the same sign as ions arriving at the collector. With the intent of verifying the X-ray hypothesis and at the same time reducing the X-ray effect, Bayard and I proposed the gauge of the type shown in Figure 5 in which the elements are inverted, and the ion collector is a fine wire maintained at a negative potential and forming a potential well within the positively charged grid. In this case the residual current was reduced by the ratio of the geometrical cross-section for the capture of X-rays, and a lower limit of approximately 5 or  $6 \times 10^{-11}$  Torr was achieved. It should be obvious that the ultimate pressure which can be measured is limited by the ratio of the ion current to the residual electron current, which in turn is proportional to the ratio of the gauge sensitivity to the X-ray current.

Since the introduction of the inverted Bayard-Alpert gauge, a number of manometers have been proposed which utilize a magnetic field to increase the electron path and hence increase the sensitivity of the gauge. These include a modified Penning gauge proposed by Houston in 1956,<sup>15</sup> the inverted magnetron gauge by Redhead in 1958,<sup>16</sup> and the Lafferty magnetron gauge in 1960.<sup>11</sup> Of these I will discuss only one, the Lafferty gauge, which has been shown to be linear over a much larger range of pressure than the others, particularly in the very low pressure regions. In its simplified form it is a magnetron operated beyond cutoff. As shown in Figure 6, the mean electron path and hence the sensitivity is greatly

extended over that of the Bayard-Alpert gauge, by approximately a factor of 1,000,000 or larger, though the full increase in sensitivity cannot be utilized. The electron current must be maintained at a relatively low value to prevent nonlinear space-charge effects. To capitalize on the low pressure possibilities of his gauge, Lafferty has inserted an electron multiplier to amplify the ion current and hence increase the sensitivity still further. The sensitive Lafferty gauge is shown in the next figure, and it is with such gauges that he has estimated an X-ray limitation below  $10^{-15}$  Torr. In fact, the arrival of individual ions can be detected by his sensitive amplification system.

In a certain sense what I have to say hereafter about pressure measurement might be considered anticlimactic since I will deal with devices which do not have a comparable ultimate limitation. However, consideration of the complexity of the Lafferty gauge and the related fact that it has not as yet reached widespread use both serve to indicate why I believe that certain other recent developments deserve equal notice. These developments are the results of efforts in several laboratories directed toward a reduction or elimination of the X-ray effect while maintaining the basic simplicity of the inverted gauge.

The first of these is a modification of the Bayard-Alpert gauge proposed by Redhead, <sup>16</sup> which is shown in the next figure, Figure 8. In this gauge a second electrode, a so-called modulator, is inserted into the grid volume. By alternately placing this electrode at two selected voltages, the ion current to the collector is modulated while presumably the photoelectric current from the collector remains the same. Thus, by calibrating at higher pressures where the ion current predominates, one

can measure the electrons and ion components separately and hence obtain a correct value for pressure even at values comparable to or lower than the X-ray limit.

One of my colleagues at the University of Illinois, Mr. Don Lee, has proposed another elegant and easily used gauge based on a similar principle.<sup>17</sup> As shown in Figure 9, his modification has two identical collector electrodes. Biasing one of the electrodes more negatively than the other increases its share of ion current while the X-ray current from both electrodes remains equal. By using a differential electrometer he reads directly and on a continuous basis the difference between the two collector currents. This gives a value attributable only to the ions since the X-ray current is subtracted out to first order. With this gauge as with the Redhead modification, pressures at least one order of magnitude below the X-ray limit can be reached.

Another member of our vacuum group at the University of Illinois, Mr. Wilfred Schuemann, has proposed still another gauge,<sup>9</sup> which is shown schematically in Figure 10. This gauge, in which the X-ray current is electrostatically suppressed, is a major step forward from an earlier proposal by Metson.<sup>18</sup> In this device ions are formed as usual within the grid of the gauge and are then focussed toward the collector by an electrostatic lens. By using a negatively charged suppressor grid which is hidden behind an optical barrier to prevent a photoelectric current from the suppressor, it is possible in principle, and in actual practice, to prevent electrons from leaving the collector. Using such gauges he has reliably measured pressures as low as  $2 \times 10^{-12}$  Torr, the lowest he has thus far been able to produce. Thus, quite a bit has happened in the field of pressure measurement. Relatively simple gauges have been devised to measure pressures to  $10^{-12}$  and possibly to  $10^{-13}$  Torr; more complex gauges have been made with a lower limit below  $10^{-15}$  Torr.

Does this mean that all the problems of pressure measurement have been solved? In a narrow sense, perhaps yes, but in a broader sense many questions remain. For example, I have devoted considerable time to the description of efforts to eliminate or reduce the X-ray effect in ionization gauges, but even for this effect the physics is not fully understood. Using his modification of the inverted gauge, Redhead 19 recently discovered when the gauge surfaces are contaminated, an effect which he interpreted as a very large change in the X-ray effect. I say contaminated but I mean that in a broad range of experiments gas is introduced to the system either purposely or otherwise. When he introduced either oxygen or carbon monoxide into the volume at an appreciable pressure, Redhead discovered that the electronic component of the collector current went up by one or two orders of magnitude. Ackley, <sup>20</sup> Lothrup and Wheeler independently observed a similar effect and demonstrated that it was a strong function of the ionizing electron current. Experiments which we have carried out recently have reproduced both of these effects. The results are shown in Figure 11. In the upper curve the electron component is plotted as a function of time after gas was first introduced at time t = 0. It is seen that within a few seconds the electron component of the circuit rose to an equivalent pressure of over 10<sup>-8</sup> Torr. It is not clear at this point whether the effect is due to an enhanced photon production at the grid, to an enhanced photoelectric effect at the collector or to a third

alternative hypothesis.\* If the ionizing electron current to the grid is increased, the anomalous effect disappears or is greatly reduced. This probably explains why the effect was not identified for ten years despite worldwide use of the gauge in hundreds of laboratories. As a matter of fact, in the course of our studies at the laboratory in the last few months we have discovered still another anomalous effect which is clearly related to the effect observed by Redhead. As shown in the lower part of the same figure, when the oxygen was introduced and maintained in the system at a background pressure of  $10^{-8}$ , one observed not only the anomalous electron current from the collector but, simultaneously, an anomalous ion current which in most cases was considerably larger in magnitude than that due to X-rays. This is shown in the lower portion of Figure 11, in which the positive ion current in the same gauge is shown on the same time scale as the X-ray current above. For reference is a plot of the background pressure as measured on an auxiliary manometer which did not exhibit the anomalous effect. Note that these effects do not manifest themselves at the very lowest pressures, but do show up at pressures where we wish to carry out a number of experiments. Although the explanation of these anomalous effects still represents an important unanswered problem, it is one which I feel virtually certain will be solved in the near

future.<sup>\*</sup> The availability of gauges which can differentiate between the electronic and ion components now provide the tools with which to interpret the readings of our gauges at very low gas densities.

In the course of our studies in low pressure measurement it has become evident that a number of other surface effects may take place in

\*Since this paper was presented, additional experimental observations give strong support to the following picture:

- (1) The anomalous ion current in a Bayard-Alpert gauge is due to surface ionization of gas adsorbed on the molybdenum electron collector, the ions are probably atomic 0<sup>+</sup> produced by dissociation of adsorbed molecules.
- (2) The associated anomalous electron component is due to secondary electrons ejected from the ion collector by the ions produced both at the surface and in the volume. The resulting current may be of the order of several percent of the total ion current.
- (3) The magnitude of (1) is determined by the surface coverage of adsorbed gas and the electron current. In the steady state, the value of the surface coverage is established by the equilibrium between the adsorption of gas from the volume and the removal of adsorbed gas by one or more electron collision processes.

Another result of these observations is that the use of a modified gauge of either the Redhead of Lee type is open to serious question when the surface ionization is comparable to the volume ionization. These results will be presented in detail in a forthcoming publication.

any measuring device. Each of these must be quantitatively understood before one can appreciate whether the gauge is measuring the volume density or is being dominated by other effects. In the next figure, Figure 12, we have shown in schematic form several more or less related gas surface phenomena which may play a significant role in any gauge for measuring pressure. First of all, adsorption and desorption of molecular gases at a gauge surface can change the volume density either by the removal of gas if the surfaces of the gauge are previously clean, or conversely by the release of gas from contaminated surfaces. This is not a trivial effect; even the Bayard-Alpert gauge, with less metal surface area than most, is capable of high pumping speeds for certain gases, particularly if the surfaces are atomically clean. A related phenomenon is that of substitutional or replacement adsorption, as schematically represented in the second portion of the figure. It has been found experimentally that certain gases such as nitrogen or carbon monoxide may preferentially adsorb on metal surfaces, displacing previously adsorbed molecules or atoms attached with a weaker binding energy. Indeed, one often observes with a mass analyzer that upon introducing CO or N into a system the hydrogen content of the system is greatly increased; thus in some circumstances the composition of the gas may be seriously altered although the pressures as measured may remain relatively constant. Since it now appears that surface effects at the electrodes may dominate at extremely low pressures, we must know what gases are most likely to be attached to the surfaces. We must also know the surface mobility and the binding energy for various combinations of gases and metals.

A third surface interaction, which has been experimentally

investigated recently by Petermann<sup>21</sup> of the Swiss Batelle Institute, is the electronic desorption of molecules or atoms as schematically shown. This desorption is due to an electronic interaction rather than a thermal heating of the surfaces by the electron bombardment.

The fourth class of surface phenomena which I have indicated here is dissociative ionization of atomic ions from surfaces in a process analogous to the dissociation of free molecules. In the case shown, the molecule represented is carbon monoxide on molybdenum, a system which has been studied by Moore.<sup>22</sup> He found a very sizable cross-section for the production of  $0^+$  ions due to electron bombardment of the surface; in fact, the cross-section for the interacton is so large as to predict an ion current larger than the X-ray current of a Bayard-Alpert gauge, even if the amount of carbon monoxide on the moly grid were less than 1/100 of one percent of a monolayer. For both of the interactions shown which involve electron bombardment, the effects may be large unless the surfaces of the gauge are kept atomically clean. On the other hand, to determine the quantitative cross-section for such a process, it is typically necessary to carry out the experiment at pressures considerably above the lowest attainable pressures. Hence, to be of value in a broad sense, a gauge must also be usable and reliable at pressures well above the ultimate limitation.

The surface interactions which I have thus far discussed are those which take place at or near room temperatures. In addition, we must understand interactions which take place well above and well below room temperature. For example, it has been known for years that the chemical interactions which take place at a hot cathode may sometimes

change both the composition and density of the gas during the course of pressure measurement. With this in mind a large number of research efforts have recently been directed toward the development of cathodes operating at lower temperatures. In his magnetron gauge Lafferty utilized a lanthanum boride cathode which operates at a temperature significantly below that of a clean tungsten surface. Lange and Fox<sup>23</sup> are experimenting with a cold electron source utilizing electron multipliers, and a number of other research workers are considering thin film devices as cold electron emitters to eliminate the effects of high temperature surfaces on pressure measurement.

This survey of surface interactions indicates that there are challenging problems involving a whole realm of surface physics interactions comparable to the molecular interactions in gaseous form. It is clear that to study these interactions there is an increasing requirement for instruments which measure partial pressure, a requirement that has been recognized for many years. The past several years have seen the development of a number of high sensitivity partial pressure mass spectrometers. These include the improved omegatron of Klopfer,<sup>10</sup> the cycloidal mass spectrometers which have been used by Lange and Trendelenburg, and a number of magnetic deflection instruments. Perhaps the most sensitive of these is an instrument recently reported by W. D. Davis<sup>11a</sup> of General Electric. He has improved an earlier commercial instrument (Davis and Vanderslice, 11b capable of measuring partial pressures down to 10<sup>-12</sup> Torr) to measure partial pressures as low as to  $10^{-16}$  Torr. This corresponds to a density of one molecule per cubic centimeter, comparable to that in outer space. I will not give a detailed discussion of these instruments but will restrict

myself to one or two editorial comments.

We have been making a direct comparison between the Klopfer omegatron and the Davis and Vanderslice instrument, and Figure 13 shows the experimental arrangement with which one of our people is doing the experiment. My editorial comment is that these instruments are so complex as to require a trained and talented experimenter. An analogy of the relationship of a musical instrument to the performer is quite in order. The analogy had nothing to do with the fact that in this case the performer's name is Mr. Segovia, one of the few people in our laboratory who can operate both instruments. We do not have time for a detailed comparison of the two mass spectrometers. Suffice to say, they do not give identical results. Certain peaks appear on one instrument which are absent on the other and vice versa. It is probably more informative to show in Figure 14 a typical spectrum observed with one of the instruments, the Davis and Vanderslice deflection mass spectrometer. This spectrum, taken with a background pressure of approximately  $5 \times 10^{-9}$  Torr, shows evidence of a number of the surface effects I have previously discussed; for example, the large carbon monoxide peak is probably due to the desorption of that gas from the surfaces of the instrument. The size and the structure of the 16 peak suggests that a considerable amount of surface dissociation of adsorbed carbon monoxide is taking place, the double peak probably representing the volume and surface contributions. In addition, there are such peaks as mass 19, attributed to florine, which is also due to surface interactions. The mass 20 peak in this case is a so-called test gas, in this case neon, for use in calibrating the gauge. With several improvements over the gauge used in these experiments, Davis has shown that

partial pressures as low as 10<sup>-16</sup> may be measured. However, it is clear from these results that we must understand the various ways in which surface effects may change the size of the peaks in the course of making the measurement.

How can we summarize the present situation? First of all, methods for producing low pressures have now caught up with our means of measuring them, and pressures down to 10<sup>-10</sup> Torr are standard in a broad technological sense. Total pressures as low as 10<sup>-12</sup> Torr at room temperature have been achieved in a few laboratory experiments. Two new classes of ionization instruments have recently been developed which have ultimate sensitivity below 10<sup>-11</sup> Torr, one class for the measurement of total pressures with ultimate sensitivities in the  $10^{-12}$  to  $10^{-15}$  Torr range and a second class which involves mass analyzers capable of measuring partial constituents as low as 10<sup>-16</sup> Torr. However, in the range of pressures below 10<sup>-11</sup> Torr (and sometimes considerably higher) the surface effects, that is, the chemical and physical interactions which take place at the electrode surfaces of the instruments, begin to be comparable to or to dominate the volume effects which they are intended to measure. Since the study of these physical and chemical phenomena can only be carried out by using the best ultra-high vacuum techniques we can devise, there is a merging of the scientific and technological motivations to study and understand these processes. These include (1) the kinetics of gas surface interactions at the interface, (2) the interaction of atomic particles, electrons and photons with surfaces, and (3) the nature of the electronic bonds between adsorbed molecules and surfaces. This field of physics, like many others, is one in which the experimentalist is challenged to design meaningful experiments before the full stalents of the theorist can be brought to bear.

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2

Figure 1

Glass-Metal Ultrahigh Vacuum System



Electron Ejection from Surface by lons. Mercury Diffusion Pumps with LN Traps

# Ultimate Pressures Attainable

Pressure in Torr	Gauge used
~5×10-11	B.A.G.
ds)	
~5×10 <sup>-10</sup>	B.A.G.
~2×10-11	S.I.G.
~6×10-12	$\Omega$ tron
~ 10-12	D.V.M.S.
< 10 <sup>-12</sup>	L.I.G.
	Pressure in Torr $\sim 5 \times 10^{-11}$ ds) $\sim 5 \times 10^{-10}$ $\sim 2 \times 10^{-11}$ $\sim 6 \times 10^{-12}$ $\sim 10^{-12}$ $< 10^{-12}$

Figure 3



•...



Schematic of Conventional Ion Gauge





Schematic of Bayard-Alpert Gauge



I









High Sensitivity Lafferty Gauge





Redhead Modification of Bayard-Alpert Gauge





Lee Modification of Inverted Gauge





Schuemann Suppressor Gauge



Figure 11





Schematic Representation of Significant Gas-Surface Reactions



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# Figure 13

Experimental Arrangement for Comparison of Omegatron with David & Vanderslice Deflection Mass Spectrometer





Partial Pressures Measured by a Davis-Vanderslice Mass Spectrometer

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