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IONIZING A CARRIER GAS

3,430,040

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Sheet 1 of 2

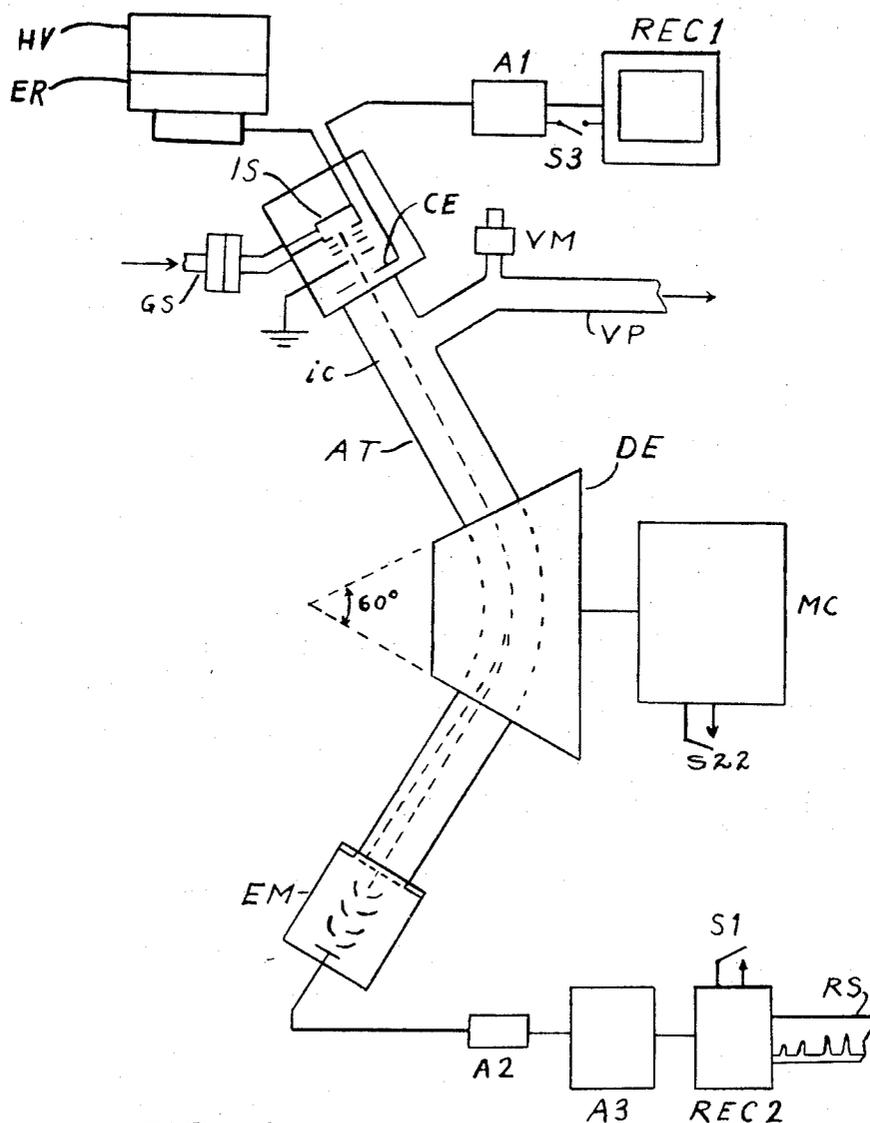


FIG. 1

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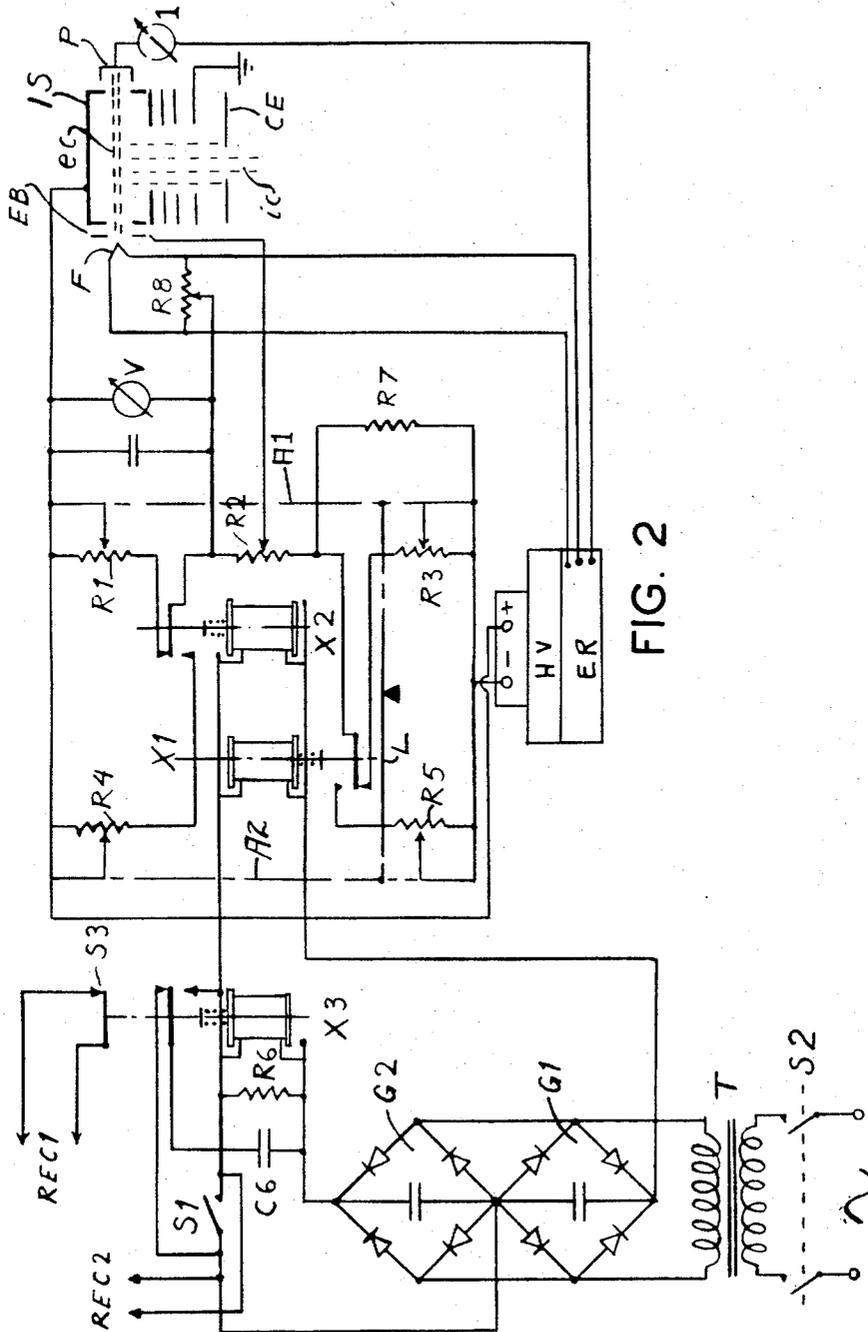


FIG. 2

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**APPARATUS FOR RECORDING MASS SPECTRA WITHOUT IONIZING A CARRIER GAS**

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2 Claims

Int. Cl. H01j 39/36; B01d 59/44

**ABSTRACT OF THE DISCLOSURE**

Apparatus for recording mass spectra of gases such as organic gases, using but a single spectrometer having a magnetic analyzer and mass spectra indicator, by initially introducing the gas into the ionization chamber along with a carrier gas, and passing an electron beam, set at a suitable low voltage by use of a voltage divider means, through the gases so as to ionize the organic gas but not the carrier gas, an ion collector being positioned in advance of the analyser to determine the total ion current, and thereafter resetting the voltage divider means to raise the electron beam energy to a higher value for ionizing both gases, to determine the mass spectrum.

This invention is related to a method and means for recording mass spectra, involving a gas, the mass spectrum of which is to be determined, the said gas being introduced into an ionization chamber of a mass spectrometer to be ionized within this chamber by means of electrons of a certain energy. A common value of the electron energy in the ionization chamber is 70 ev. The electrons are emitted from a filament, accelerated to the desired energy and captured by an electrode after the passage through the ionization chamber. The ions produced are extracted from the chamber, focused, accelerated and transmitted into a magnetic analyzer.

To be able to record the total ion current, produced in the ionization chamber, a collector is normally arranged at a certain distance from the exit slit of the ionization chamber but in front of the magnetic analyzer. This electrode will catch part of the ion beam current. The caught part of the ion current is led from the collector to an electrometer amplifier, the output of which is connected to a recording apparatus. The part of the ion current caught by the collector is proportional to the total ion current and by means of this device it is possible to record variations occurring in the ion current, when different quantities and kinds of atoms and molecules are ionized.

This method of measuring the total ion current, however, has a certain drawback in cases when a small amount of the gas to be investigated, e.g., an organic compound, for example, is introduced into the ionization chamber in a mixture of carrier gas, e.g., helium. In this case, namely, if a normal electron energy (i.e. 70 ev.) is used in the ionization chamber, then also the carrier gas will be ionized. The part of the ion current which originates from the gas to be investigated will be small in comparison with the part of the ion current originating from the carrier gas, thus causing difficulties in significantly indicating the ion current from the gas to be investigated.

It is possible to eliminate this drawback by giving the electrons in the ionization chamber a low energy, permitting only the organic compound to be ionized but not the carrier gas. The ionization potential e.g. of helium is 24.8 ev., while the ionization potential for organic compounds is lower and usually of the order of 8–15 ev. Thus, if the electrons in the ionization chamber are given an energy, which is between 15 and 24.8 ev., the ion cur-

rent produced will originate substantially entirely from the organic compound. By this method the mentioned drawback is thus avoided, but instead will occur another drawback due to the fact that the ionization potentials of the fragments of organic compounds differ from each other very much, which means that a decrease of the electron energy will cause a decreased ionization of certain fragments. Mass spectra recorded at a low electron energy will thus not be quite comparable with mass spectra recorded at normal electron energy (70 ev.).

To be able to avoid also the last mentioned drawback it is possible that the gas stream could be split into two parts being separately investigated in two ionization chambers. One part of the gas stream will be introduced into a normal ionization chamber and the ion beam originating from this chamber will pass the analyzer tube where the ions are separated according to their mass/charge ratio, upon which they are recorded in a form of a mass spectrum. The electron energy in this first ionization chamber could be varied as desired but is normally set at 70 ev. The other part of the gas stream is introduced into a second ionization chamber, and the ions produced within this chamber are attracted to an electrode which is connected to an instrument for recording the ion current. The electron energy in the second ionization chamber say 24.8 ev. is normally lower than the ionization potential for e.g. the carrier gas helium but higher than the ionization potential for organic compounds (8–15 ev.). By means of the last mentioned ionization chamber a change of the concentration of organic compounds could easily be indicated, while simultaneously a normal mass spectrum could be obtained by means of the first mentioned ionization chamber. The use of a second ionization chamber and the necessary auxiliary equipment involves a considerable increase in complexity and cost of the apparatus however.

An object of the present invention is to provide a novel method and means for making it possible, while using but a single ionization chamber, to obtain a normal mass spectrum based on a safe indication of the total ion current.

The method and means according to the invention is accomplished firstly by setting the electron energy in the ionization chamber to a first value before recording a mass spectrum which, at the beginning of the recording of the mass spectrum is changed to a second value which is maintained until the desired mass range has been recorded. The first value of the electron energy is suitably set at substantially 20 ev., while the second value which is used when recording a mass spectrum suitably is approximately 70 ev. Thus a normal mass spectrum will be obtained. The electron energy used when recording mass spectra could, however, also be set for some gases at other values between 10–100 ev.

The invention will be more apparent after a perusal of the following specification taken in consideration with the accompanying drawings wherein:

FIG. 1 schematically shows the main parts of the mass spectrometer apparatus for recording mass spectra; and

FIG. 2 shows a circuit diagram of the novel apparatus of this invention for carrying out the method of recording mass spectrum according to the invention.

Similar characters of reference are used in the above figures and designate corresponding parts.

The ion source is designated in the figures as IS. It receives the necessary voltages from a high tension regulator HV and an emission regulator ER. The gas, to be investigated, is introduced into the ion source through a tube GS. The ions produced are directed by means of focusing and accelerating electrodes into the analyzer tube AT (FIG. 1). A part of the ion current is captured

by a collector CE before entering tube AT and is fed into an electrometer amplifier A1, the exit of which is connected to a recorder REC1 for the recording of the total ion current. The remainder of the ion current will pass through the analyzer tube AT and the magnetic analyzer DE. The analyzer tube AT is connected to a high vacuum pump and cold traps by the tube VP. A vacuum gauge VM is connected to the tube VP.

In this analyzer DE, the ion beam will pass through a magnetic field, in which the ions are deflected to a varying extent according to the mass/charge ratio. The magnetic field in the analyzer DE is produced by an electromagnet which is fed with direct current from a magnet current regulator MC. The magnet current is linearly varied versus time, causing the ion beams, originating from the magnetic analyzer DE, to sweep over the entrance of an electron multiplier EM.

The output current from the electron multiplier EM is amplified in two amplifiers A2 and A3, A3 being a wideband amplifier feeding a photographic recorder REC2, in which the mass spectra will be recorded on a paper chart RS. The recorder REC2 is started by means of a switch S1. The recorder can also be combined with a switch S22 for starting the sweep current from the magnet current regulator MC, causing this regulator to be started after the paper chart RS in the recorder has been fed a certain length.

The electron current  $ec$  in FIG. 2, as indicated by ammeter I, passing through the ionization chamber is emitted from a filament F and captured after the passage through the ionization chamber by an electrode P. The energy of the electrons is prescribed or determined by the voltage across the filament F and the ionization chamber IS as indicated by voltmeter V. This voltage is taken from a potentiometer or voltage divider, including the resistor R2 and alternatively the resistors R1 and R3 or the resistors R4 and R5. The end connections of the resistor R2 are connected to make-and-break contacts on two relays X1 and X2. When these relays are not energized, the resistors R1 and R3 are kept connected to the voltage divider as shown in FIG. 2. When the relays are energized the resistors R4 and R5 are instead kept connected to the voltage divider. A suitable voltage (D.C.) is fed to the voltage divider from the voltage supply HV, which in FIG. 2 is marked with plus and minus signs. The ionization chamber IS is directly connected to the positive end of the voltage divider, while the filament F is connected to the connection between resistor R2 and the make-and-break contact on relay X2 via the middle point of a resistor R8, which is connected between the filament current leads.

The resistors R1, R3, R4 and R5 are made in the form of rheostats, the sliding contacts of R1 and R3 being interconnected by movable arm A1 and the sliding contacts of R4 and R5 being interconnected by movable arm A2 as shown in FIG. 2. The sliding contacts on R1, R3 and R4, R5, respectively, are connected with each other as by pivotal link L so that a decrease of resistance within one resistor R1 or R4 will cause a corresponding increase of the resistance in the other resistor R3 and R5, respectively. By this arrangement, the total resistance of this voltage divider is kept approximately constant, independent of the setting of the sliding contacts of the resistors. The resistor R2 is made in the form of a potentiometer, and to the movable arm of this potentiometer is connected an electron aperture EB, which is placed between the filament F and the ionization chamber IS.

In operation, the coils of relays X1 and X2 are connectable in parallel with the output of a rectifier G1, which is fed from an A.C. supply via the switch S2 and the transformer T. To the secondary winding of the transformer T is connected another rectifier G2, intended for supplying operation current to a third relay X3. Relays X1, X2 and X3 can be connected to their rectifiers by means of the switch S1, which, as mentioned earlier, is

also connected in the drive circuit of the photographic recorder REC2 (FIG. 1). This recorder is fed from a separate, not shown, current supply. As long as the switch S2 is open, the emission regulator will work in the normal way, and mass spectra can be recorded by closing the contact S1. The closing of S1 will have no other effect than the starting of the photographic recorder REC2 and the sweep for the magnetic analyzer DE. The electron energy in the ionization chamber is, however, not effected by a closing of S1. The electron energy will be prescribed by the voltage across resistor R1.

In using the method according to this invention, that is to say, to change the electron energy when recording mass spectra according to this invention, the switch S2 is closed. When the contact S1 thereupon is closed, the photographic recorder and the magnet sweep will be started as before, and the relays X1, X2 and X3 will also be energized. The relays X1 and X2 will disconnect the resistors R1 and R3 from the voltage divider and instead connect in the resistors R4 and R5. The electron energy in the ionization chamber will now be prescribed by the voltage across the resistor R4, this resistor is premised to be adjusted so that the voltage across the same is considerably higher than the voltage prescribed by R1. Thus, the electron energy for the ionization will be increased. When the electron energy is thus increased, a considerably increased ion current will be obtained resulting in an increase of the reading of the recorder REC1 (FIG. 1), indicating the total ion current.

To prevent the recorder from getting overdriven (and go off scale) the input of the recorder is disconnected by means of the contact S3 on relay X3, when change over to higher electron energy is effected. When the contact S1 is disconnected again, the relays X1, X2 and X3 are de-energized, causing the electron energy to return to its previous value and the recorder for total ion current is switched in again. It is desirable, however, that the switching in of the recorder is somewhat delayed. The relay X3 is therefore, as appears in FIG. 2, combined with a RC-circuit R6 and C6, having a suitable time constant for delaying the disengaging of relay X3.

In the described apparatus it is presupposed that the change-over from one electron energy level to another is carried out manually by operating the switch S1. The device could also, if desired however, be arranged to automatically effect this change-over.

Since many changes could be made in the above construction and many apparently widely different embodiments of this invention could be made without departing from the scope thereof, it is intended that all matter contained in the above description or shown in the accompanying drawings shall be interpreted as illustrative and not in a limiting sense.

What is claimed is:

1. Apparatus for mass spectrum determination comprising a mass spectrometer having an ionization chamber, a magnetic analyser and an indicator for obtaining a mass spectrum of a gas, means for passing a gas to be investigated into said chamber together with a carrier gas, means for passing an electron beam through said chamber, and voltage divider means connectable to a voltage supply for setting the electron beam energy of said electron beam (firstly) at a first relatively low value suitable for ionizing the gas to be investigated but not the carrier gas, and at a second and higher value suitable for ionizing both gases, and for determining the mass spectrum, said voltage divider means comprising two sets of resistors, each set of resistors being adapted to be connected in series across said voltage supply, relays for selectively connecting said sets of resistors across said voltage supply, and means connecting said resistors to said electron beam passing means for determining the electron voltage applied to the beam, and an ion collector disposed before said analyser to permit the determination of the total ion current when said beam energy is set at said first value.

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2. Apparatus for mass spectrum determination as defined in claim 1 wherein said first value of electron beam energy is insufficient to effect ionization of the carrier gas, and wherein said indicator is a recording instrument for the mass spectrum, and means for disconnecting said recording instrument while the electron energy is being changed over from the first to the second value, said disconnecting means incorporating time relay means for delaying the reconnection of said recording instrument in changing over from the second value of electron beam energy back to said first value.

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