# United States Patent

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[54] **DEVICE AND METHOD OF DIRECT INTRODUCTION OF MATERIALS INTO MASS SPECTROMETERS FROM ADSORBANTS**
**2 Claims, 1 Drawing Fig.**


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[50] Field of Search ..... 73/422, 421; 250/41.9 S



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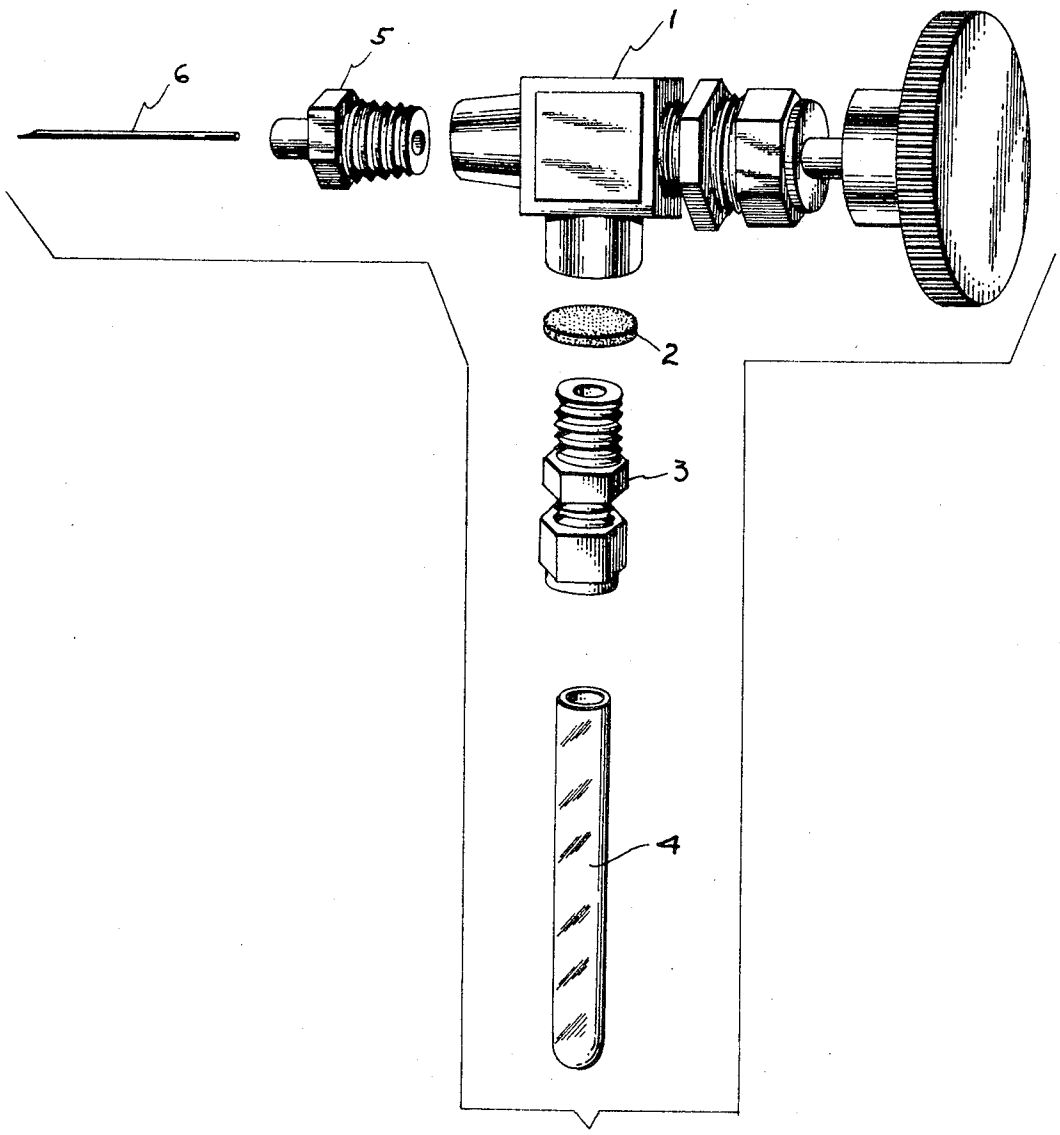
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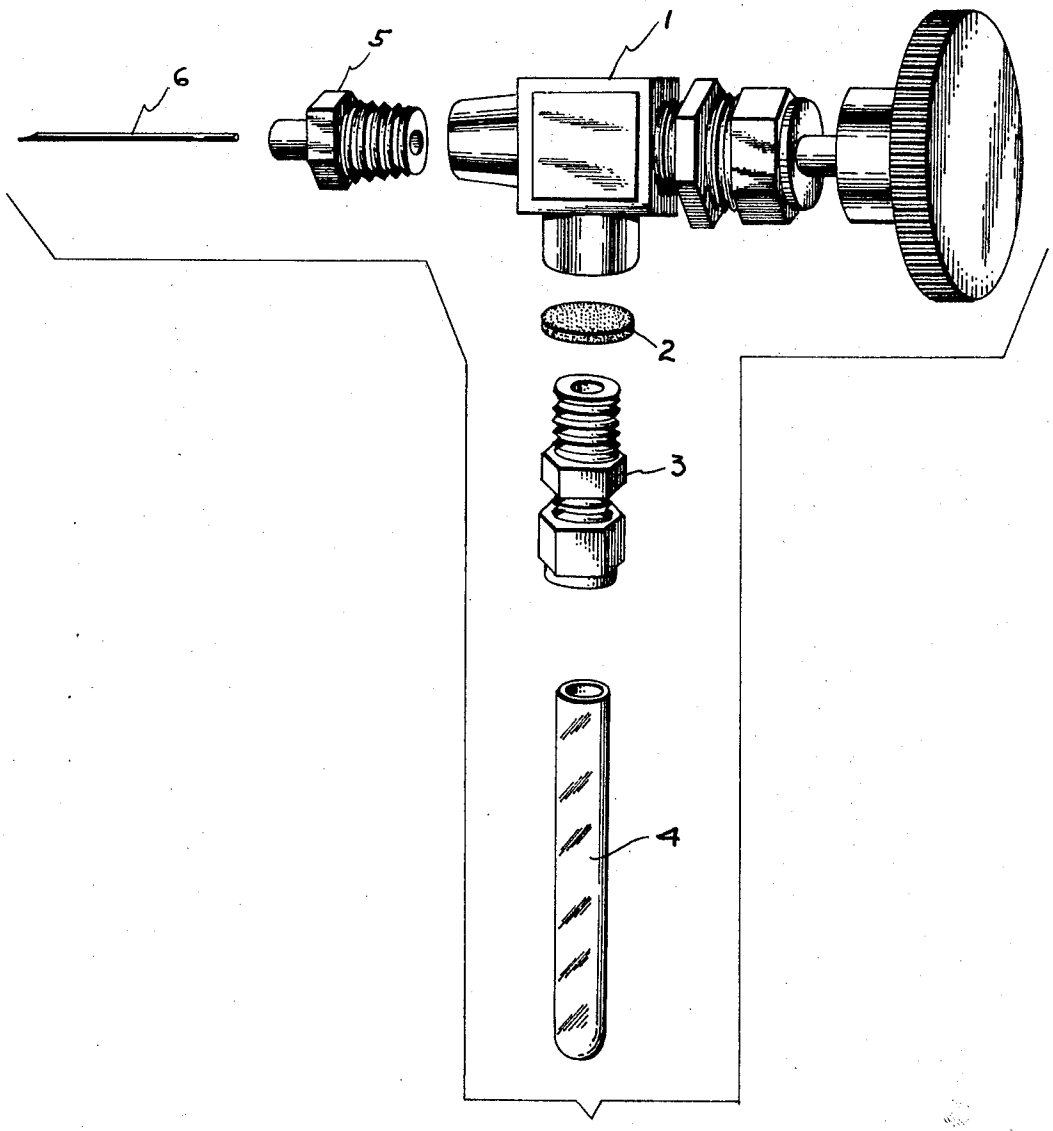


**ABSTRACT:** A device for use with the gas inlet system of a mass spectrometer to obtain mass spectra of certain organic compounds directly from the adsorbant onto which it is adsorbed. A sample to be tested is placed in a chamber which is attached by means of a fitting to a two-way valve. Attached to the valve is a needle which is inserted into a septum. The valve is opened and air is pumped drawn into the mass spectrometer. A mass spectrum of the sample is obtained thereby.

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# DEVICE AND METHOD OF DIRECT INTRODUCTION OF MATERIALS INTO MASS SPECTROMETERS FROM ADSORBANTS

A nonexclusive, irrevocable, royalty-free license device the invention herein described throughout the world for all purposes of the United States Government, with the power to grant sublicenses for such purposes, is hereby granted to the Government of the United States.

This invention relates to a devices and method of analysis pertinent to mass spectroscopy and volatile compounds. More specifically, this invention relates to a mechanical device which will make it possible to use the gas-liquid inlet system of a mass spectrometer to obtain mass spectra of certain organic compounds directly from adsorbants without requiring a preliminary extraction of the organic material from adsorbant.

Heretofore, the mass spectra of organic compounds adsorbed on the surface of a finely divided adsorbant, such as a fraction obtained by removing a zone from a thin layer chromatogram, could be obtained by a laborious procedure involving extraction of the organic material from the adsorbant with a volatile solvent, removal of the solvent by evaporation and transfer of sample into a low-volume closed container suitable for insertion into the gas-liquid sampling system of the mass spectrometer. The aforementioned procedure suffers from the disadvantage that it is relatively time consuming and there is a possibility of loss of sample and contamination of the sample during transfer.

Another method for obtaining this sort of mass spectrum makes use of a solid probe inlet system (see "Combination of Thin-layer Chromatography and Mass Spectrography," by K. Heyns and W. F. Grutzmacher, Angewandte Chemie, 74, 387 (1962). In said method the sample of adsorbant containing the organic material is placed near the ionizing zone of the mass spectrometer. The sample is vaporized and passes directly into the ionizing zone from the surface of the adsorbant This is a difficult and time-consuming method involving inserting the probe into the mass spectrometer and removing it through a series of valves requiring a number of evacuation steps. Furthermore, a background of other materials which may have condensed on the probe is usually required before a mass spectrum of the sample can be obtained.

The main object of this invention is to provide a device which enables the use of the gas-liquid inlet system of a mass spectrometer for obtaining the mass spectrum of an organic compound directly from the adsorbant onto which it is adsorbed.

A second object of this invention is to provide a device for accomplishing the aforementioned operation rapidly and easily and with a minimum of loss or contamination of sample.

A third object of this invention is to provide such a device which is simple to build, use and maintain.

The invention will be further described by reference to the single FIGURE of drawing, showing an exploded view of the device, and an illustrative experiment which was performed for the purpose of describing the preferred embodiment of the invention.

A 10 $\mu$l. sample of carvone was diluted with 90 $\mu$l. of absolute ethanol. A 10 $\mu$l. sample of this solution was applied to a 5×20 cm. Eastman thin layer chromatogram sheet, type K 301 R. The sheet was developed in a solvent system until the carvone had traveled approximately one-half the distance to the top. After evaporation of the solvent from the sheet, the area onto which the carvone was adsorbed was scraped off and placed in the glass chamber **4**.

The chamber **4** was attached to the two-way vacuum valve **1** by means of the Swagelock fitting **3**. A sintered metal disc **2** was inserted between the fitting **3** and valve **1** as shown in the FIGURE The syringe needle **6**, attached to valve **1** by means of fitting **5**, was then inserted into the septum, valve **1** was opened and the air pumped out of the system with the valve to the mass spectrometer closed. The latter valve was then



opened and chamber **4** heated until the carvone was vaporized and drawn into the mass spectrometer through the sintered disc **2**, the disc serving to prevent fine particles of the adsorbant from being carried into the inlet system. A strong mass spectrum of carvone was obtained in this manner.

The device can be made in various shapes and sizes and from a variety of materials such as, for example, steel, aluminum, brass, glass, monel or Teflon—depending on the nature of the adsorbant and organic material as well as the requirements of the various types of mass spectrometer inlet systems.

The device may be equipped with an electric heater or a jacket for circulation of coolant or a similar temperature regulation device for the purpose of adjusting the temperature of chamber **4** to suit the volatility of the organic material.

The sintered metal disc **2** can be replaced by any equivalent filtering element such as a glass wool plug. The device may also use an alternative to the syringe needle which will serve the function of providing an opening from the valve into the gas-liquid inlet system of the mass spectrometer.

A wide variety of inorganic adsorbants can be used in conjunction with this device such as silica gel, alumina, carbon and magnesium silicate as well as organic adsorbants such as cellulose and polyamides provided only that the difference in volatility between the organic material and the adsorbant is great enough to allow a clean mass spectrum of the organic material to be taken without interference from volatile substances originating from the adsorbant.

The range of organic materials suitable for application of this method includes volatile compounds such as monoterpene hydrocarbons and extends to relatively nonvolatile compounds, for example diterpenoids and other high molecular weight relatively nonvolatile compounds. The limitations as to volatility are set only by the necessity for the volatility difference between the adsorbant and the organic material adsorbed on it to be great enough to allow a clean mass spectrum of the organic material to be taken without interference from volatile substances originating from the adsorbant.

I claim:

**1**. A device for feeding volatilized uncontaminated volatile organic compounds through gas-liquid inlet systems of mass spectrometers consisting of these component parts assembled to function in unison....

a. a chamber for placing chromatographically separated adsorbant and volatile organic sample increment mixtures,

b. fitting means for attaching the chamber to a two-way vacuum valve,

c. a sintered metal disc filtering means designed to fit between the fitting means and the two-way vacuum valve to constrain a solid adsorbant and permit a volatilized volatile organic sample to pass into the ionizing zone of the mass spectrometer uncontaminated,

d. means to control the volatilized flow of sample impelled in the direction of the ionizing zone of the mass spectrometer by a heat source,

e. needle-to-valve fitting means to connect the two-way vacuum valve to a syringe needle, and

f. a syringe needle through which the sample passes to the gas-liquid inlet system of the mass spectrometer in an uncontaminated gaseous form.

**2**. A method of introducing volatile organic samples readily and uncontaminatedly through a gas-liquid inlet system having a two-way valve of a mass spectrometer having an ionizing zone, for more efficient analysis comprising.....

a. locating a volatile organic compound in a thin-layer chromatography plate,

b. scraping small increments of adsorbant from the region which contains the said volatile organic compound off the said chromatography plate and directly into a sample chamber,

c. closing the system to the atmosphere promptly thereafter,

d. coupling the chamber to the gas-liquid inlet system of said mass spectrometer,

e. turning the two-way valve to evacuate the system and make ready for analysis, and

f. applying a sufficient quantity of heat to the outside of the sample chamber to volatilize and impel the volatile organic compound being analyzed away from the adsorbant and into the ionizing zone of the mass spectrometer.