Cromatografia de gases y gases masas pdf

GC/MS is the method of choice for the analysis of small and volatile organic molecules such as benzene, alcohol and aromatic substances, and simple molecules such as steroids, fatty acids and hormones can also be used in the study of liquid, gas and solid samples. Using GC/MS for integrated analysis has many advantages, such as its ability to separate complex mixtures, quantify and identify analyses, and determine levels of traces of organic contamination. The technique begins with a gas chromatographer, where the sample is volatilized. This effectively vaporizes the gaseous phase sample and separates its various components using a capillary column filled with stationary (solid) phase and inert carrier gas such as argon, helium or nitrogen. Because the components are separated, they elude the column at different times, known as the time they are retentiond. The components come out of the SC column and are ionized by a mass spectrometer that uses chemical or electronic sources of ionization. Ionized molecules are accelerated through a mass analyzer of the instrument, which is a quadripolis or ion trap. Here the ions are divided depending on their different mass/load ratios (m/z). At the end of this process, ions are detected and analyzed, and composite peaks appear based on their m/z ratios. The complex sample will produce several different peaks generating a mass spectrum. The quantification and identification of unknown connections and analytes is possible with the help of automated search in libraries. GC/MS can be used in both full MS scanning and ion monitoring (SIM) mode to cover a wide range of m/z ratios or to collect data for specific masses of interest, respectively. Gas chromatography team. Gas chromatography is a chromatographic technique in which the sample is volatilized and injected into the head of the lighter in the chromatographic column. The elusation is caused by the flow of the moving phase of inert gas. Unlike other types of chromatography, the moving phase does not interact with analytic molecules; its only function is to transport the analite through the column. There are two types of gas chromatography (GK): gas-solid chromatography (GSK) and gas-liquidity chromatography (GLA), the latter is the most widely used and which can simply be called gas chromatography (GK). In GSC the stationary phase is solid and the retention of analytics in it occurs through the asorptation process. It is this non-linear process of adsorption that is the one that has that this type of chromatography has limited use, since the retention of analitis on the surface is semi-permanent and the peaks of the eleuzia are obtained by tails. Its only application is the separation of low molecular mass of gas species. GLC uses immobilized liquid molecules on the surface of an inert solid as a stationary phase. The GK is performed in a gas chromatographer. It consists of various components such as the carrier gas, sample injection system, column (usually inside the furnace) and detector. The history of chromatography dates back to 1903 in the work of the Russian scientist Mikhail Tsvet. German phD student Fritz Pryor developed solid-fuel gas chromatography in 1947. Archer John Porter Martin, awarded the Nobel Prize for his work in the development of liquid-liquid chromatography (1941) and paper (1944), laid the foundation for the development of gas chromatography, and then liquid-gas chromatography (1950). Erica Kremer laid the groundwork and controlled most of Pryor's work. The gas carrier Diagram of the gas chromatographer Gas Carrier basically serves two purposes: transporting exemplary components, and creating a suitable matrix for the detector. The carrier's gas must meet certain conditions: it must be inert to avoid interaction (both with the sample and with the stationary phase) It must be able to minimize the spread of gas easily accessible and purely economic suitable for the detector to use ... The carrier's gas should be inert gas to prevent its reaction with analytic or spine. Gases such as helium, argon, nitrogen, hydrogen or carbon dioxide are usually used, and the choice of this gas sometimes depends on the type of detector used. Gas storage can be in normal bullets or using a generator, especially in the case of nitrogen and hydrogen. Then we have a system of pressure sensors and flow controllers to ensure a stable flow and a system of gas dehydration such as a molecular sieve. Pressure control is usually carried out at two levels: the first pressure sensor is located at the exit of a bullet or gas generator, and the other at the entrance to the chromatographer, where the flow is regulated. The input pressure varies from 10 to 25 psi, resulting in a flow rate of 25 to 150 ml/min in filler columns and 1 to 25 ml/min in capillary columns. You can use a rotary counter or a simple soap pump counter to check the flow rate, which gives a very accurate measurement of the volume of flow entering the column. Gas purity is extremely important, requires levels of 4.5 and above, i.e. 99.995% purity. However, because of the care that needs to be taken with The active phase of the column becomes absolutely necessary to set traps at the entrance to the gas, these traps clearly have a limited capacity, but they are very important when using a chromatographer. These traps prevent hydrocarbons, water and CO from entering among others. The injection of a sample sampling system is a critical section, as enough must be injected, and must be inserted in such a way (as a steam cork) that it is quick to avoid extending the output lanes; this effect occurs with a lot of analytic. The most commonly used method uses micro-jerking (multi-million-dollar capabilities) to insert analitis into the instant evaporation chamber. This chamber is 50oC above the boiling point of less volatile components, and is sealed with a partition or partition silicone rubber pad. Sample injector for GC If the reproducibility of the input sample size is required, a six-sided valve or injection valve can be used where the amount to be introduced is constant and determined by the size of the injection valve cycle. Automatic sampler for use Headspace or Head Space for GC (accessory) technology. If the column used is filled, the volume to be entered will be about 20 liters, and in the case of capillary columns this number is less than 1 litre, and depending on the type of capillary column (since there are columns of different internal diameters) is that if the entire volume of the injected sample is used. To get a smaller volume, a thread divider is used when you insert a column that is discarded, the flow divider (the injection is known as separation mode) is used. If you use the entire sample, the injection is no split. Splitless mode was used more to identify small quantities or traces (environmental definitions). When 1 microliter of solvent, such as water, is introduced, when it enters the steam phase, its volume will be multiplied by a thousand. That is, the microlitre of water will become 1 ml of gas water; Because the volume of the injection port is limited, pulse split or other configurations are used to ensure that the sample enters correctly. In the case of solid samples, they are simply introduced as dissolution, as in the instant evaporation chamber the solvent is lost in the cleaning current and does not interfere with the elyu. According to Van Deuter's Curves (HEPT vs. Linear Speed), the best gas to use in the chromatographic column as anity carrier is hydrogen, but given its danger, it is most commonly used as an ignition gas in the FID detector, along with air. Then come, respectively, helium and nitrogen. Hydrogen gas Better carrier and streams handled by chromatographers are not dangerous, in addition to exiting them there are usually flame limiters that prevent the spread of a possible fire. The use of hydrogen may be recommended in connection, first, for its low price compared to other gases and for the resolution of peaks shown in chromatograms. The ignition-to-air ratio is 4.1% for the lower limit and 74.8% for above 101.3Kpa and 298K (NASA's Hydrogen and Hydrogen Safety Standard), and should be in the presence of a spark or high heating zone (from 520oC). Columns and temperature control systems in GC use two types of columns: packaged or filling columns and open or capillary tubular columns. The latter are more common today (2005) because of their greater speed and efficiency. The length of these columns is variable, from 2 to 60 meters, and are built of stainless steel, glass, molten silica or Teflon. Because of their length and the need to put them in the oven, the columns are usually rolled up in a helicoid form of 10 to 30 cm long, depending on the size of the oven. Temperature is an important variable, as it will depend on the degree of separation of different analitis. To do this, it must be adjusted with the accuracy of tenths of a tenth. This temperature depends on the boiling point of analitis or analitis, as well as on the maximum temperature of the column (stationary phase) and usually corresponds to the value equal or slightly above it. For these values, the elusion time will vary from 2 to 30-40 minutes. If we have several components with different boiling points, the so-called temperature ramp is adjusted so that it increases either continuously or in stages. In many cases, correcting the ramp can mean separating the different analitis well or not. It is advisable to use low temperatures for elution, because - although the higher elution temperature is faster - you run the risk of breaking down analyte. You can promote the ramp both to increase and to reduce the temperature of the furnace, so that there was no overlap of peaks. Detectors Detector is part of the chromatographer that is responsible for determining when the analysis came out towards the end of the column. Ideal Detector Features: Sensitivity: It is essential that you can accurately determine when the analyte comes out and when only the carrier gas comes out. They have sensitivity between 10-8 and 10-15 g/s analytic. A linear response to analytic with a range of several orders of magnitude. Short response time, regardless of output flow speed. A wide range of working temperatures, for example, from ambient temperature to 350-400oC, typical working temperatures. Stability and reproducibility, i.e. an equal amount of analytic should give equal signal outputs. High reliability and simple handling, or proof of inexperienced operators. A similar answer for all analitis, or a selective and highly predictable response for a small number of analytics. Some types of detectors: the Flame Ionization Detector (FID). Thermal conduction detector (TCD). Thermoion detector (TID). Electron Capture Detector (ECD). Atomic Emissions Detector (AED). View of the fiD (disassembled) GC detector. Other minority detectors are the photometric flame detector (PFD) used in compounds such as pesticides and hydrocarbons containing phosphorus or sulfur. In this detector, the gas is transmitted by a hydrogen-oxygen flame, where part of the phosphorus becomes an HPO species that emits 510 and 526 nm, and simultaneously the sulfur becomes S2, with an emissions of 394 nm. This emitted radiation is detected by the appropriate photometer. Other elements were found, such as some halogens, nitrogen, tin, germanium and others. In the photoionization detector (PID), the gas that escapes at the end of the column is exposed to ultraviolet radiation with energies from 8.3 to 11.7 eV, which corresponds to 0.106-149 nm. Applying the potential to the ionization cell generates a flow of ions that is amplified and recorded. Columns and types of stationary phases Filling columns Filling or packaged columns consist of glass, metal (inert to be possible, like stainless steel, nickel, copper or aluminum) or Teflon, length from 2 to 3 meters and internal diameter from a few millimeters, usually from 2 to 4. The interior is filled with solid material, thinly separated to have a maximum surface of interaction and covered with a layer of thickness from 50 nm to 1 m. In order to put them in the oven, they are conveniently rolled. The ideal filler material consists of small particles, spherical and homogeneous, with good mechanical strength to have a maximum surface where the stationary phase and analytic interact. The minimum specific area should be 1 m2/g. Like all components of the GC column, it should be inert at high temperatures (-400oC) and evenly moisturized with a stationary liquid phase during the production process. Currently, the preferred material (2005) is natural diatom soil, due to its natural pore size. These species, already extinct, molecular diffusion system to simulate nutrients from the environment and expel their waste. Therefore, they are particularly useful materials, as the system of absorption of the surface of analytic and stationary phase is similar. Size is crucial when it comes to the process of interacting with analitis, and at lower sizes the effectiveness of the spine is better. But there is a problem of pressure required for the circulation of a stable flow of carrier gas through the column, as this pressure is inversely proportional to the square diameter of these particles. Thus, the minimum size for the use of the maximum pressure of 50 psi is from 250 to 149 m. Capillary columns of capillary columns have two main types: columns with coating (WCOT) and support with coating (SCOT). WCONs are simply capillary tubes where the inner wall was covered with a very thin layer of stationary phase. THE SCOT columns have a thin layer of absorbent material in the inner part, like the one used in the filler columns (diatom soil), where the stationary phase stuck. The advantage of WCOCs over SCOTPs is to increase capacity, as they use more stationary phases, as it is the largest exchange zone. In order of efficiency, first there are WCONs, then SCOT and finally fill the columns. WCOT columns are made of molten silica, known as open tubular columns of molten silica or FSOT. These columns are made of particularly pure silica, with hardly any metal oxide content. Because of the fragility inherent in this material, in the same process of receiving the tube is covered with a layer of polyimide, so the column can be wounded with a diameter of several centimeters. These columns, with properties such as low reactivity, physical stability, and flexibility, replaced the classic WCONs. FSOT columns have variable internal diameters, 250 to 320 mm (for conventional columns) and 150-200 mm for high-resolution columns. The latter require less analytics and a more sensitive detector, by eluiring less gas. There are also macro-capillaries of columns up to 530 mm in diameter that support the amount of analytic comparable to the filling, but with better performance. In these columns there is a problem due to the asorptation of analytic on the surface of molten silica, asorptium due to the presence of silanole groups (Si-OH), which strongly interact with organic polar molecules. This problem is usually olving by inactivating the surface by siliating with dimethylchloylyan (DMCS). Adsorption due to metal oxides is largely softened purity of the silica used. The stationary phase of properties required for the immobilized liquid stationary phase: Distribution characteristics (capacity factor and α selectivity factor) suitable for analytic. Low volatility, the boiling point of the stationary phase should be at least 100oC above the maximum temperature reached in the oven. Low reactivity. Heat stability to prevent decomposition during elulation. There are no more than a dozen solvents with these characteristics. To choose one, the polarity of the analytic should be taken into account, since the greater the polarity of the analytic, the greater the polarity of the stationary phase must be taken into account. Some stationary phases currently in use (2005) are: Polydimethylsiloxane, a non-polar phase for general use for hydrocarbons, aromatic substances, polyenecular substances, drugs, steroids and PCD. Poly (phenylmethyldiphenyl)siloxane (10% phenyl), for methylester fatty acids, alkaloids, medications and halogenated compounds. Poly (phenylmethyl)siloxane (50% phenyl), for drugs, steroids, pesticides and glycoles. Poly (triforopropildimet methyl)siloxane, for chlorinated aromatic substances, nitroaromatics, alkylsustytut benzene. Polyethylene glycol, serves for polar compounds, as well as for compounds such as glycol, alcohol, esters, essential oils. Poly (dicianoalildimethyl) siloxane, for polyunsaturated fatty acids, free acids and alcohol. Typically, in commercial columns, the stationary phase is tied and intersected to prevent its loss during elation or washing operations. This results in the chemical attachment of the monolayer to the surface of the column. The reaction involved is usually adding peroxide to the liquid to be corrected, starting a free radical reaction, leading to the formation of a carbon-carbon bond that also increases its thermal stability. Another way is gamma radiation. Another type of stationary phase is the chiral phase, which allows you to solve enantiometric mixtures. These types of phases are usually chiral amino acids or some derivatives adapted to the operation of the spine. The thickness of the film varies from 0.1 to 5 m; thickness depends on the volatility of analytic. Thus, a very volatile analytic will require a thick layer to increase the interaction time and more effectively

separate the different components of the mixture. Typical columns (internal diameters of 0.25 or 0.32 mm) use thicknesses of 0.25 mm, and in macro-capiillaria columns the thickness rises to 1 m. Maximum thickness, usually 8 m OF Applications has two important fields of application. On the one hand, its ability to separate complex organic mixtures, organetal compounds and biochemical systems. Your other app is a method for quantitatively and qualitatively the components of the sample. For qualitative analysis, a retention time is usually used, which is unique to each connection under certain conditions (same carrier gas, temperature and ramp flow), or retention volume. In quantitative applications, integrating the areas of each compound or measuring its height, with appropriate calibration, the current concentration or number of each analysis is obtained. The CG Analytical Assembling Technique technique is purely empirical, the profile of the analyses that need to be determined, the choice of mobile phase, the retention time (eleusation) will be determined solely by the specific conditions of the column (stationary phase) before the equipment. Temperature ramps to choose from can either beothermal or staggered. The choice of phase will depend on the type of detector, the choice of column (stationary phase) will depend on the polarity of the connections that will be separated, the detector will depend on the type of connections detected. Typically, the GC analytical method consumes many hours of chromatographer, which will be developed and installed by trial and error before being tested as real. The choice of standards is fundamental to the development of technology. Stabilizing the base line of the mobile phase in the stationary phase (post-soluble front) over time is crucial for the creation of the method. An unsealed or irregular base (solution) that changes the intensity before the detector as it slips away must be configured and stabilized before the introduction of the tests. The location of the furnace's temperature range, the correct choice of column and its stationary phase (includes type, length and diameter), correct choice of the type of detector, detector temperature and injector, the volumes of the analite should be set in such a way as to get the maximum efficiency in the separation of analytics and with the best possible resolution. The purity of the sample will depend on the pre-preparation of the sample. CG is a highly effective methodology and its performance allows for a wide range of possibilities for analytical chemistry in organic compounds. The result of this method is HPLC chromatography, which works based on analytic affinity to the liquid moving phase instead of soda. The sensitivity of the GC method can even detect micrograms of analytic if it is well established. The quantitative estimate is based on calculations of the area under the curve, proportional to the concentration of analitis. It is widely used in the internal working standard. See also a translation of the bibliography of Skug, Douglas A. and Leary, James J. (1994). Analysis Armenia: McGraw Hill. ISBN 84-481-0191-X. McNair, Harold M. and Miller, James M. (1998). Basic gas chromatography. Canada: John Wylie and Sons, Inc. ISBN 0-471-17260-X (paper); ISBN 0-471-17261-8 (pbk.: alk. paper). Datos: Multimedia No677065: Gas chromatography Obtenido de