Gas Chromatography Mass Spectroscopy of Hydroxyfulvenes

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Abstract

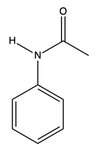
This project was conducted to determine the conditions necessary in Gas Chromatography Mass Spectroscopy (GCMS) for various hydroxyfulvenes. Several samples each having unique characteristics, such as high molar mass or specific functional groups, were used to determine the optimal conditions for each compound. Different GC parameters were varied including the inlet temperature, initial oven temperature and oven temperature ramp. Compounds were also studied at varying concentrations in order to determine the optimal conditions needed to produce reliable molecular ion data in the MS.

Introduction

Gas Chromatography Mass Spectroscopy (GCMS) is an instrument that is used to analyze specific compounds of interest. This instrument is used in fields such as forensic science to test for drugs, food and beverage analysis, to determine its purity and presence of pesticides; and environmental analysis, to monitor contaminants within nature. Specifically, GC is used to separate sample into individual components. The sample must first readily dissolve in solvent, usually an organic one. Only one microliter of the solution needs to be injected into the inlet. The sample is then pushed down the capillary tube with helium into the capillary column. Once in the capillary tube, the sample can interact with the stationary phase on the column and separate into components based on mass and volatility. MS is used to produce mass to charge ratio of abundance of each component that is isolated during GC. Electrons are shot at the components to break them down into smaller pieces called fragments and produce multiple masses to identify the original sample. Only ions can pass through the filter to the electromagnetic field created by the charged rods. The rods can either create a positive voltage which repels positive ions, or the rods can create a negative voltage which attracts positive ions. The chance of the ion hitting the rods depends on the mass and the charge of the ion as well as the strength of the field and frequency of oscillation. They are then separated by mass. Ions with a high mass and low charge readily pass through the electromagnetic field to the detector. The detector then counts how many of each ion is present and displays the quantity through the intensity of each peak.

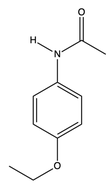
Structures

Acetanilide: 135.17 g/mol



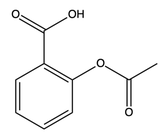
Acetanilide

Phenacetin: 179.22 g/mol



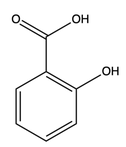
Phenacetin

Aspirin: 180.1 g/mol



Aspirin

Salicylic Acid: 138.1 g/mol



Salicylic acid

Hydroxyfulvene cc500: 343.19 g/mol

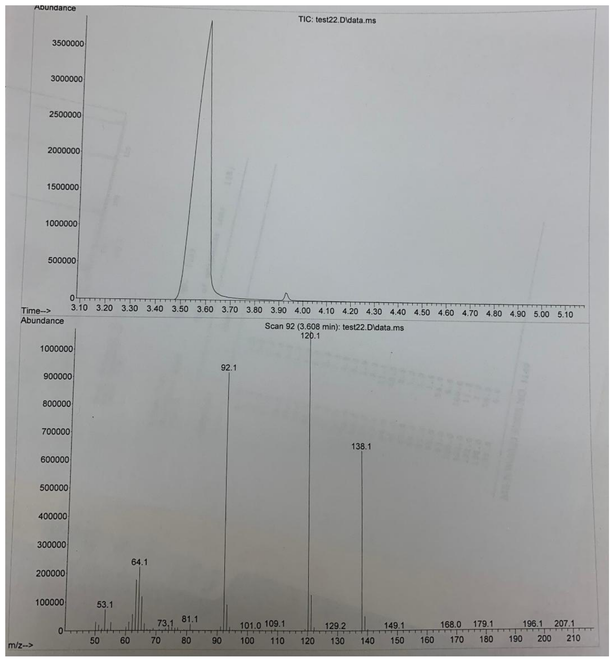
(Z)-(4-chlorophenyl)(5-((4-chlorophenyl)(hydroxy)methylene)cyclopenta-1,3-dien-1-yl)methanone, CC500

Hydroxyfulvene cc900: 310.286 g/mol

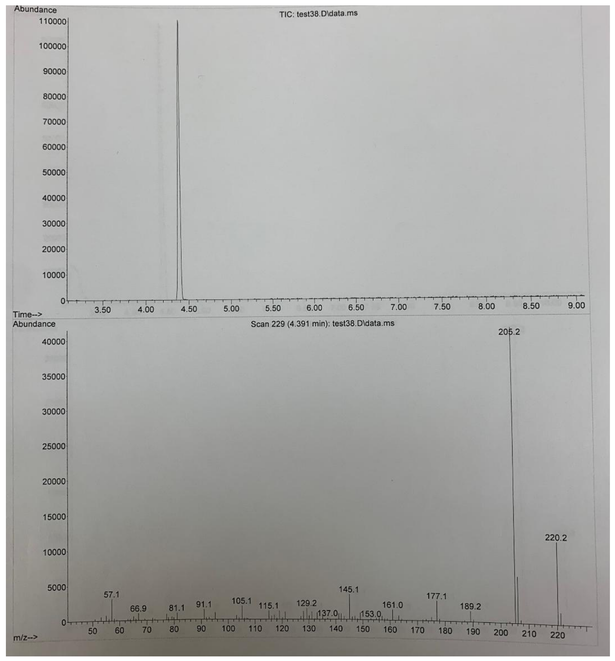
(Z)-(4-fluorophenyl)(5-((4-fluorophenyl)(hydroxy)methylene)cyclopenta-1,3-dien-1-yl)methanone, CC 900

Discussion & Results

The proper condition for hydroxyfulvenes has still not been determined. The structure of the thermal decomposition product has also not been determined. More research will need to be conducted in order to conclude whether hydroxyfulvenes are stable enough to be analyzed using GCMS.



Salicylic Acid



Hydroxyfulvene

Methods and GCMS Conditions

Ideal Conditions for Acetanilide, Phenacetin, Aspirin and Salicylic Acid: oven temperature of 60ºC and inlet temperature of 175ºC and a ramp of 40ºC per minute to a max of 220ºC. The high inlet temperature allows all sample to be cleared, leaving no residue behind. This reduces the chance of the previous sample bleeding through to the next run.

Using these conditions, the hydroxy-fulvenes were not detected by the MS. This may have been the result of thermal decomposition, so the temperature of the inlet was lowered to 100ºC, then the ramp was brought down to 20ºC per minute to allow the analyte to warm slowly. The starting oven temperature was also lowered to 40ºC to further reduce the chance of thermal decomposition. Both hydroxyfulvenes produced a peak of 220 m/z at a little less than four and a half minutes.

References

Gas Chromatography Mass Spectrometry (GC-MS) Information: Thermo Fisher Scientific - US. https://www.thermofisher.com/us/en/home/industrial/mass-spectrometry/mass-spectrometry-learning-center/gas-chromatography-mass-spectrometry-gc-ms-information.html#:~:text=GC%2DMS%20is%20a%20powerful,in%20air%2C%20water%20and%20soil (accessed Apr 7, 2021). MaidoMerisalu. Quadrupole Mass Spectrometer Working Principle Animation - How to Measure Vacuum. https://www.youtube.com/watch?v=qxPb9vFWdqo (accessed Apr 7, 2021). TheNFSTC. Gas Chromatography/Mass Spectrometry. https://www.youtube.com/watch?v=bVKASwadjQY (accessed Apr 7, 2021).