Research Article

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GC – MASS: A tool for rapid Identification of Synthesized Benzopyrans

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Abstract

The synthesis of benzopyrans is an important reaction in organic chemistry because of their wide application in medicinal chemistry. In present communication, synthesized benzopyrans alongwith their different derivatives have been identified by using a rapid identification method of GC-Mass spectrometry. We have developed an efficient separation technique for the identification of such type of synthesized benzopyrans.

Keywords: Benzopyran, Chromakalim, Microwave Irradiation, Synthesis, GC-MS, Method

Introduction

Chromane, chromosomes & chromanones are associated with interesting chemistry form the core ring structure of a number of natural products like flavanoids, isoflavanoids, coumarins, homo-, iso- and neo-flavonoids [1, 2]. Compounds representing these types of classes are manifested with a variety of remarkable biological activities such as anti-allergic activity, tyrosine kinase inhibitory activity, estrogen receptor agonist or antagonist activity or inhibitor activity of steroidegenic enzymes [3,4]. Chromones/chromanones or in simple terms benzopyranones can be potential intermediates for the synthesis of heterocyclic analogs of steroids and or important building blocks for the preparation of pterocarpans and isoflavones with strong fungicidal activity [5]. Another important activity associated with chromans having substitution at e. g 2- & 4-positions have been that of its smooth muscle relaxant activity and these compounds have been used for the treatment of disorders such as asthma and hypertension with chromakalim being the best representative example of these anti-hypertensives [6].

The 1-benzopyran ring system constitutes the basic skeleton of a variety of natural compounds that show interesting biological activities. The synthesis of benzopyrans is an important reaction in organic chemistry because of their wide application in medicinal chemistry. The applicability of these type of compounds as potassium channel openers (PCO's) antihypertensive antiasthematic urinary incontinence etc is well documented [5-18].

Thin layer chromatography is the only tool for the detection of conversion of reactant into product but synthesis of benzopyrans can also be monitored by the use of GC-MS which can give a valid confirmation about the synthesis of the product [19]. Therefore present study has been undertaken to contribute a better knowledge of separation as well as identification of synthesized benzopyrans due to the lack of data on qualitative analysis of synthesized benzopyrans. GC-Mass plays a very important role for the identification of volatile as well as semi-volatiles compound. To the best of our knowledge this is the first report on the method of rapid identification of benzopyrans through GC-MASS. In this communication we have developed a method for rapid identification of different derivatives of benzopyrans.

Experimental

Materials and Methods

Benopyrans has been synthesized by using microwave irradiation in presence or absence of solvent and with or without the use of solid support. Solvents and other chemicals of reagent grade were used without further purification. Laboratory grade solvents were purified and dried by reported methods. All melting points were determined by capillary method on a Buchi technical apparatus (BUCHI-510) and are uncorrected. NMR spectra (^{1}H NMR and ^{13}C NMR) were obtained on Bruker Supercon 200 MHz and 500 MHz instruments and are expressed in d values down field from tetramethylsilane (TMS) as the internal standard. Mass spectra were recorded with a JEOL MS-D 300 mass spectrometer, IR (KBr pellet or neat sample) were recorded on Perkin Elmer-377 and Shimadzu IR-435 spectrophotometers. Column chromatography was performed on silica gel (100-200 mesh) and TLC was performed on Silica gel 60-120 mesh F_{254} (Merck) plates. For the visualization of spots either UV or iodine vapor, or 10% aqueous sulfuric acid containing 2% Ceric ammonium sulfate or 2, 4-dinitrophenyl hydrazine (5% ethanol solution) were used.

Synthesis

The compounds AKT-1 to AKT-5 has been synthesised by using microwave irradiation techniques and their structure has also been elucidated through spectroscopic data (^{1}H NMR,^{13}C NMR, Mass & IR).

Gas Chromatography - Mass Spectrometry Analysis

GC-MS analysis was performed on a Varian GC 3800 hyphenated to Varian 4000 mass spectrometer. Compounds were separated on CP-SIL 8 CB MS fused silica capillary column (30 X 0.32 mm id., film 1.00 um). The injector and detector temperature were 230 and 300°C respectively. Temperature programming of oven was from 60°C to 250°C at 3°C/ min rising rate and was then held constant for 10 minutes. Injection was performed using a split mode of 1: 100. High purity helium employed as a carrier gas with a constant flow rate of 1 mL/min. An electron impact mass spectral (EI-MS) analysis was carried out at ionization energy of 70 ev at 250°C. The detection was performed in the scan mode between 40 to 500 amu at 3 scan / s

Qualitative analysis

Identification of the constituents was based on comparison of the obtained MS with those of references compounds in the WILEY and NIST.

Result and Discussion

In the present study among the five different derivatives of synthesized benzopyranes has been separated and identified by developing a novel and an efficient method. The compound has been identified by the comparison of obtained MS with its fragmenting pattern. The compounds AKT-1 to AKT-5 appear at definite retention times (RTs) i.e. 62.639, 58.727, 48.369, 33.699 and 51.312 respectively which reveals that those compounds having high MW and less volatiles appear at the delayed RT viz. AKT-1 appears at 62.639 min. whereas AKT-4 appears at 33.699 min. which is having low MW. Results summarized in Table 1. Chromatogram of the compounds along with their spectra is also shown which revealed that all these compounds are separated in a good manner. The MS obtained by the GC-MS gives about its fragmenting pattern which can be easily understand by the scheme of the fragmenting pattern of the compounds AKT-1 to AKT-5. The compound AKT-5 appears at 51.312 min. while having 230 molecular weight where as the compound AKT-2 appears at 58.727 having molecular weight 218 due to the volatile nature of the compound AKT-5 than AKT-2.

The separation of compounds depends upon the polarity of the compounds as well as molecular weight (MW) of the compounds. The compound AKT-5 appears prior to AKT-2 due to the attachment of ethereal linkage at C-7 position of benzopyran which make its high volatile as compared to AKT-2. The peaks appear in the mass spectra and theoretical fragmentation pattern of these compounds are in good agreement. Fragmentation pattern of AKT-1 showed 232,190,177,137 and 108 these are in good agreement with obtained Mass spectra of the compound.

The results obtained in GC-MS showed that this technique can be used for rapid separation and identification of synthesized benzopyrans and their derivatives.

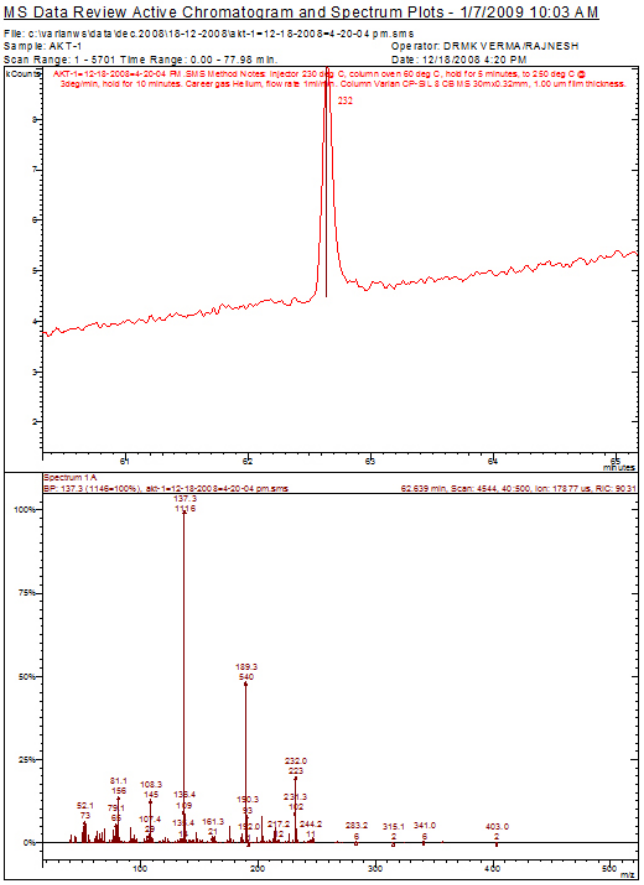


Figure 1: Chromatogram and mass spectrum of Comp. AKT-1

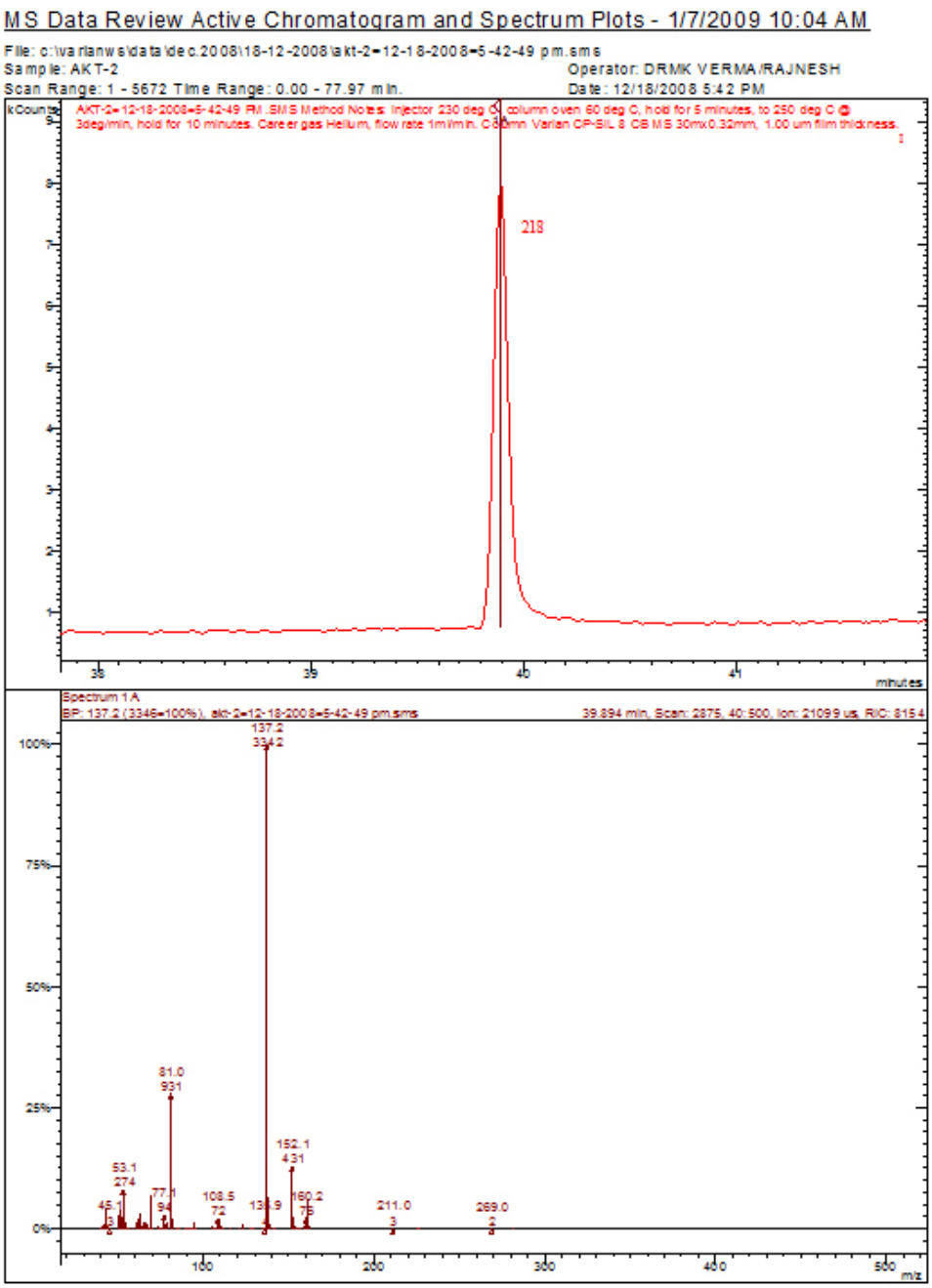


Figure 2: Chromatogram and mass spectrum of Comp. AKT-2

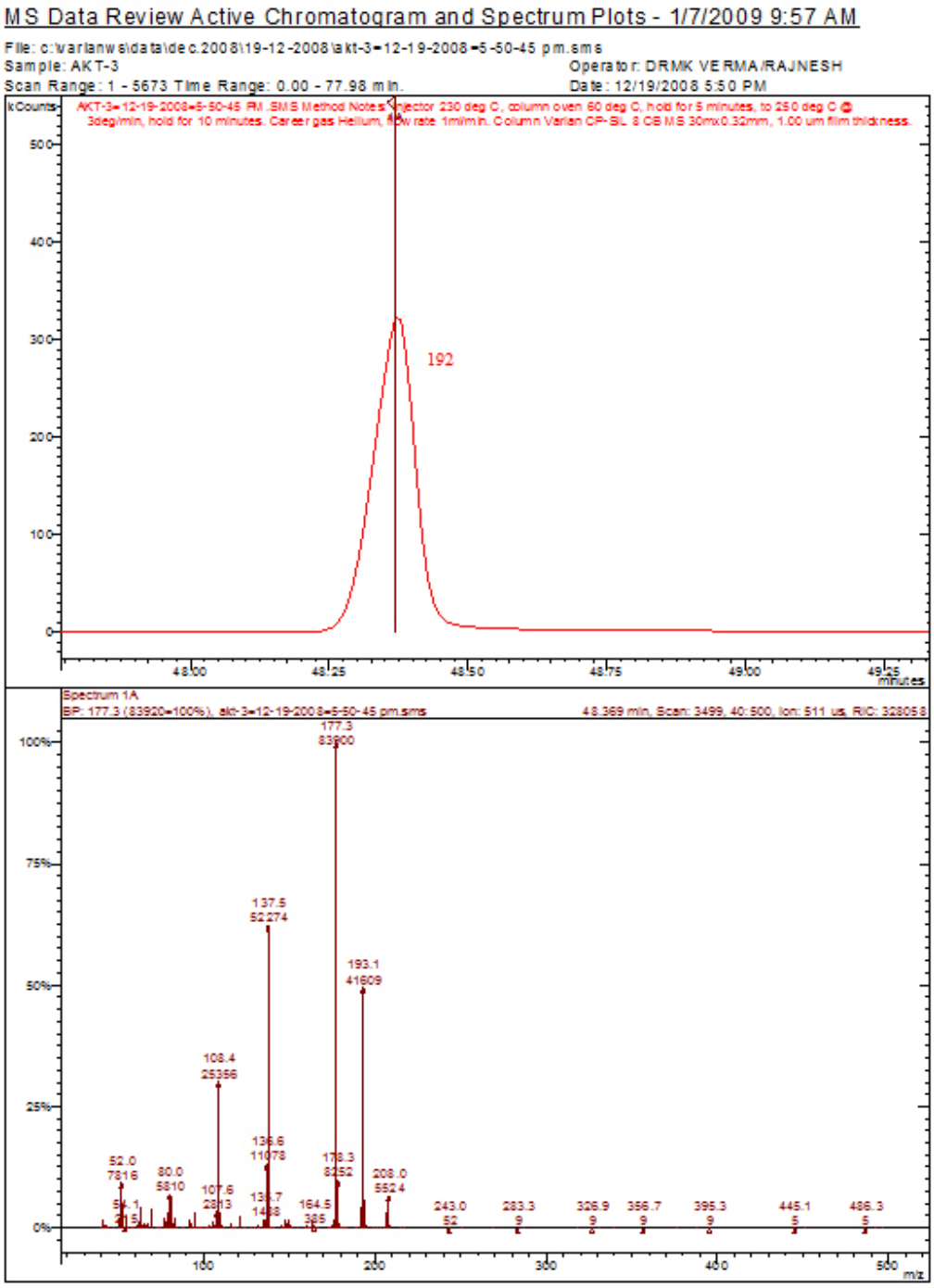


Figure 3: Chromatogram and mass spectrum of Comp. AKT-3

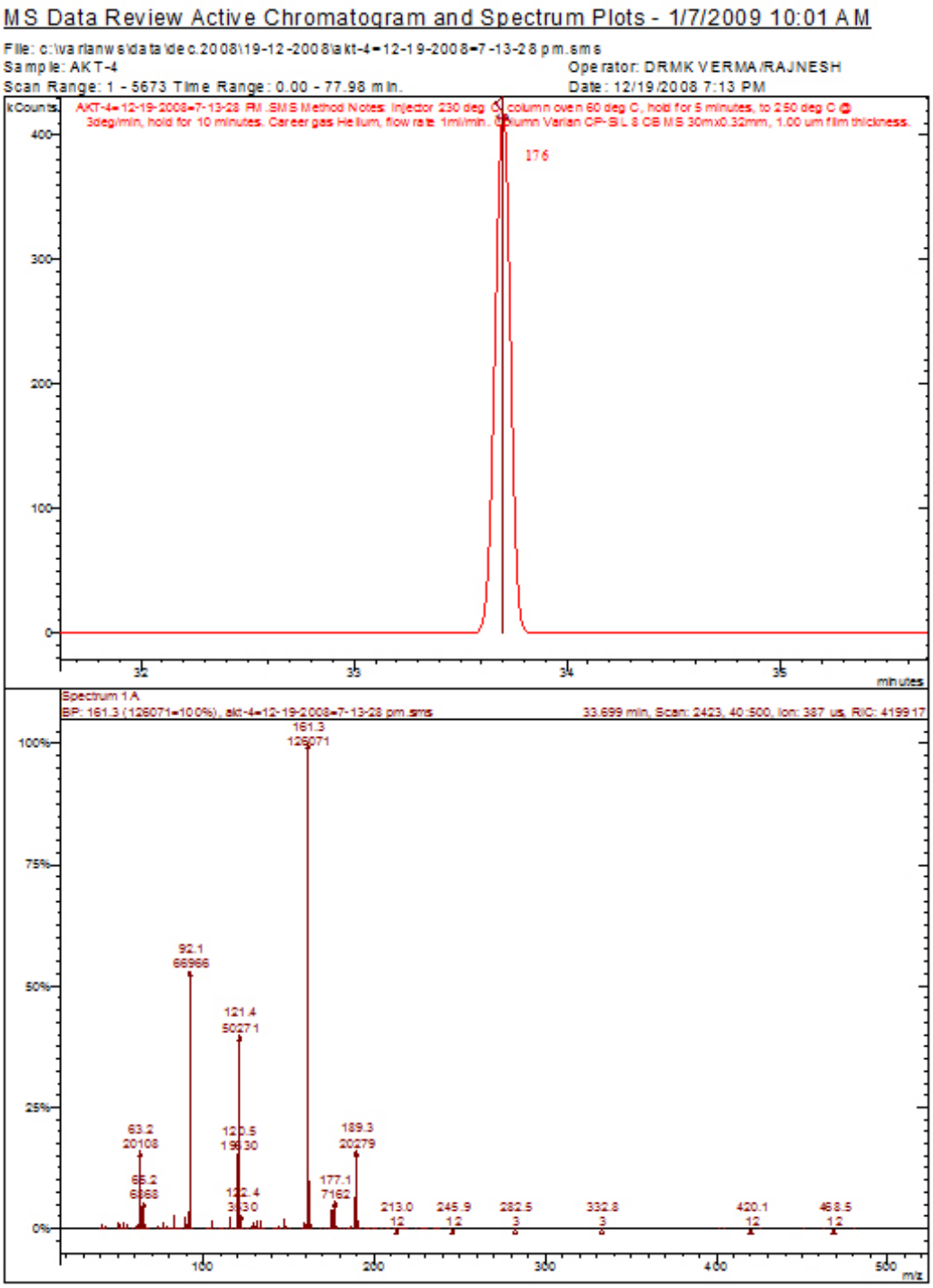


Figure 4: Chromatogram and mass spectrum of Comp. AKT-4

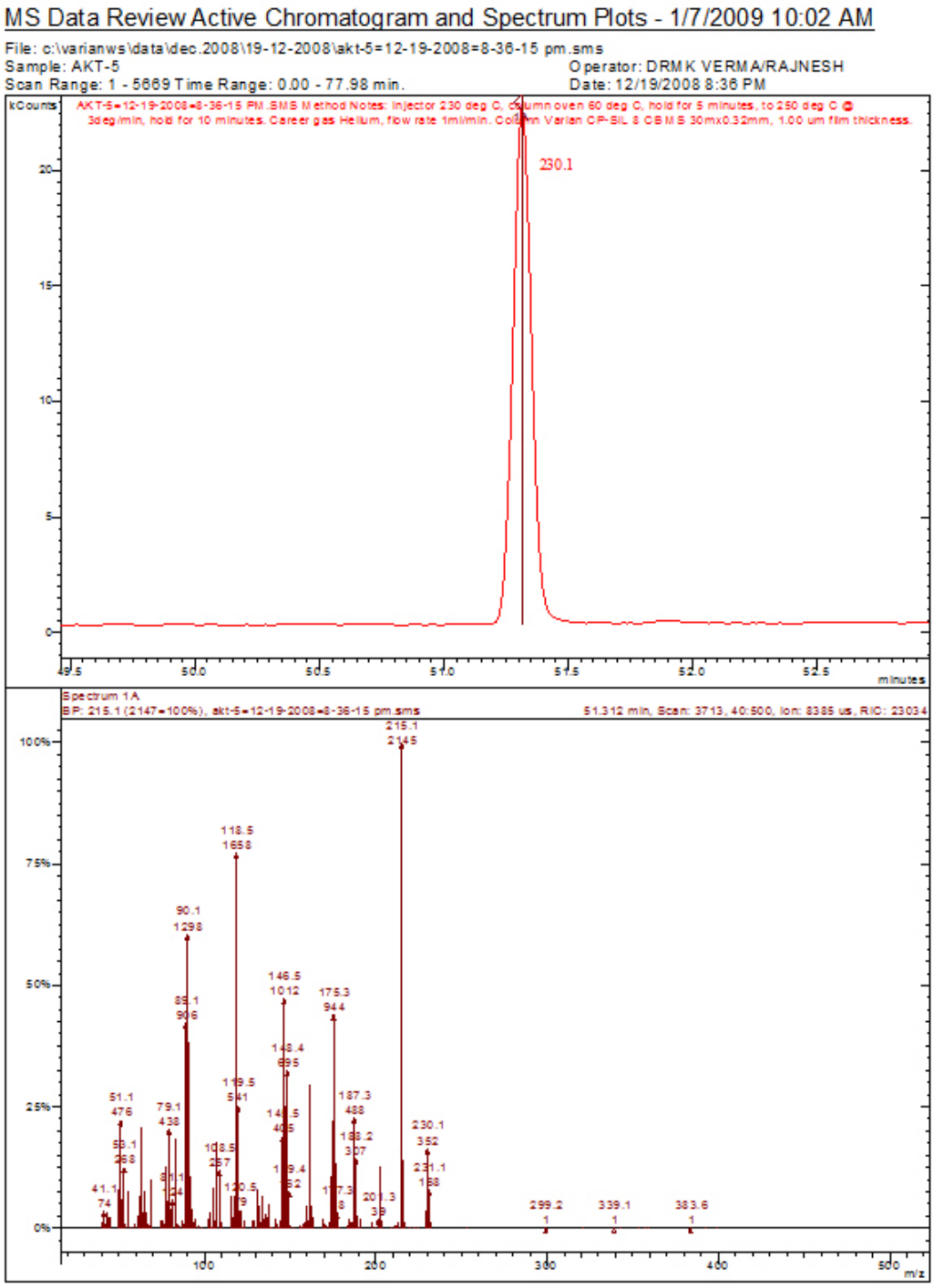
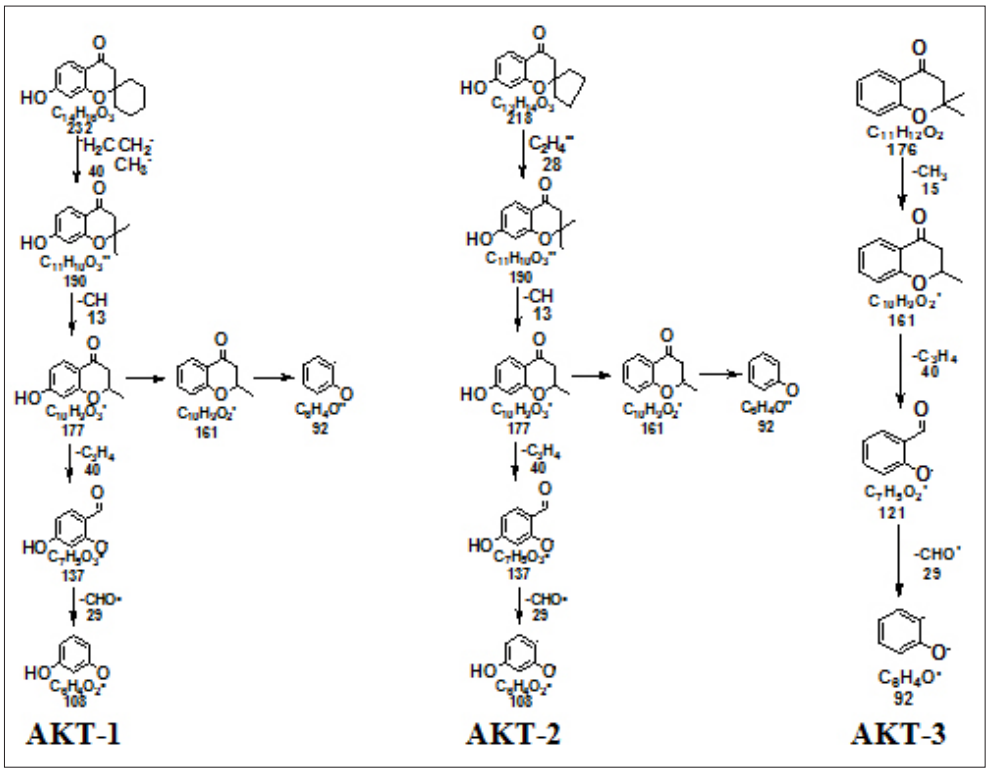
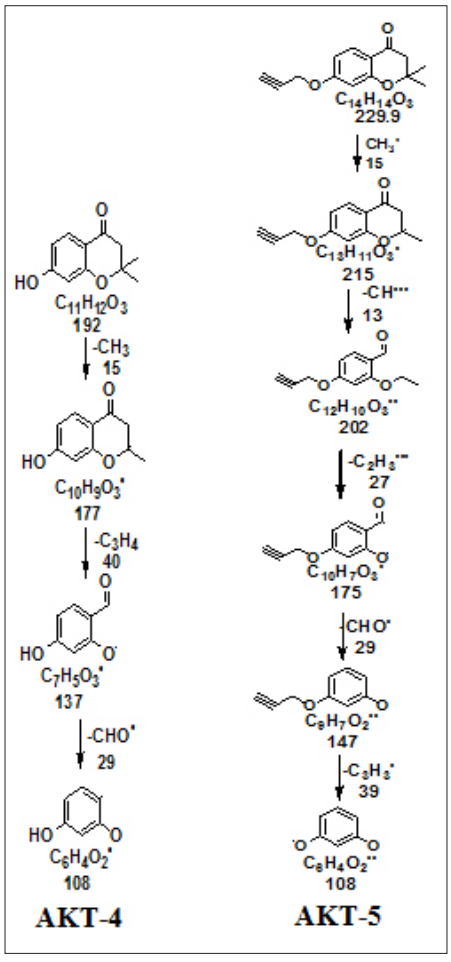


Figure 5: Chromatogram and mass spectrum of Comp. AKT-5

Fragmentation pattern of the compounds





Conclusions

Gas chromatography-mass spectrometry (GC-MS) was shown to be a suitable for this study as it is very accurate, effective and reliable technique for the rapid analysis and characterization of synthesized benzopyrans.

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