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ELECTRON IONISATION MASS SPECTRA OF SOME SUBSTITUTED CHALCONES

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SUMMARY

Electron ionisation mass spectra of some substituted chalcones and related epoxy ketones were investigated with the intention to characterise newly synthesized compounds, having biologic potential as antibacterial and antiinflamatory products.

INTRODUCTION

The heterocyclic chalcones under investigation were based on aryl-thiazolo[3,2-b][1,2,4]triazole or aryl-thiazole heterocycles that were condensed with ortho or para hydroxyacetophenone. The aryl group had the following substituents in the para position: R_1 = H, Cl and CH_3. The related heterocyclic epoxy ketones had as substituents of the aryl group R_1 = H, Cl and CH_3 and the phenyl group of the benzoyl moiety was either unsubstituted or had as substituent R_2 = Br.

EXPERIMENTAL

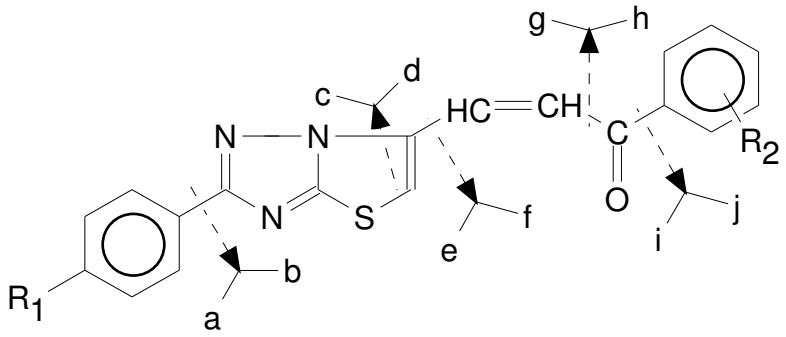
Mass spectra were recorded on a MAT 311 mass spectrometer with EI ion source, at an ionisation energy of 70 eV. The solid samples were introduced using the direct inlet probe and the mass spectra were recorded and digitalised under computer control by repetitively scanning the mass range under temperature programming of the sample crucible from room temperature to 300 °C.

High resolution exact mass determinations were made using the peak matching technique at a resolution of 6000.

RESULTS AND DISCUSSION

All substituted chalcones showed intense molecular ions, supporting the structure of the synthesized compounds. In fact, for all three substituents R_1, the molecular ion was the base peak of the spectrum.

Fragmentations of the molecules under electron impact, common to all substituted chalcones investigated, could be observed. The structure of substituted heterocyclic chalcones and possible fragmentations of the molecule under electron impact are shown in Scheme 1.



Scheme 1. Substituted heterocyclic chalcones

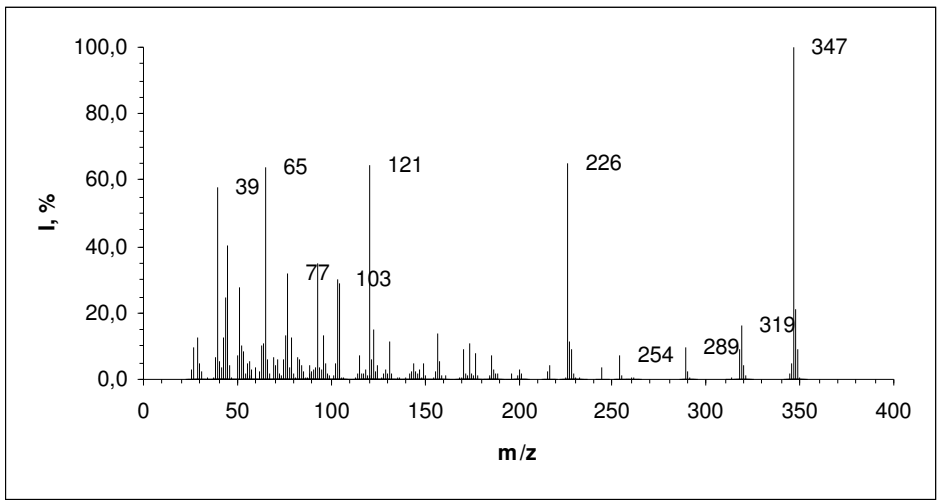


Figure 1. Substituted chalcone, R_1 = H, R_2 = p-OH, M = 347

Typical mass spectra of three substituted heterocyclic chalcones are shown in Figures 1 – 3. Fragmentation of the single bond between the triazole ring and the adiacent phenyl ring produces very few ions, if any (ions **a**, **b**, Scheme 1).

Fragmentation of the single bond between the thiazole ring and the ethylene group (ion **e**) is also weak, producing the ion m/z 199 + R_1 +H transposition from the right side, of low intensity.

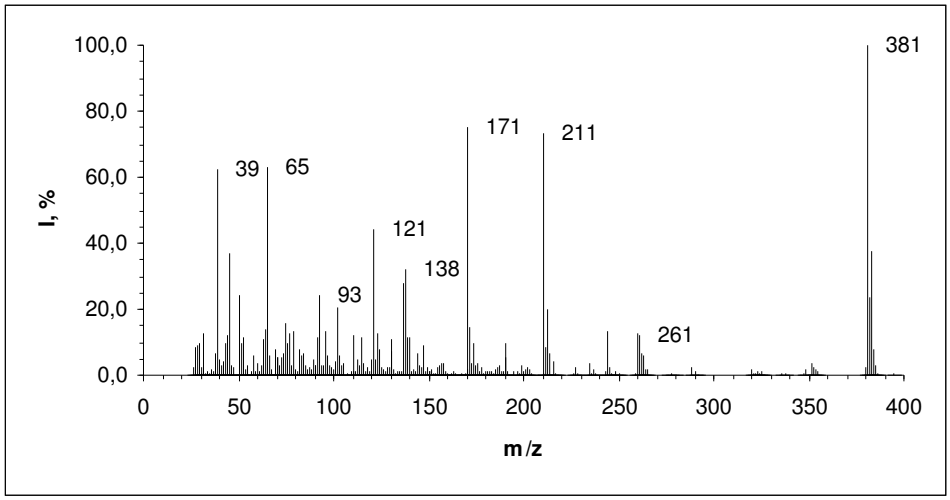


Figure 2. Substituted chalcone, R_1 = Cl, R_2 = o-OH, M = 381

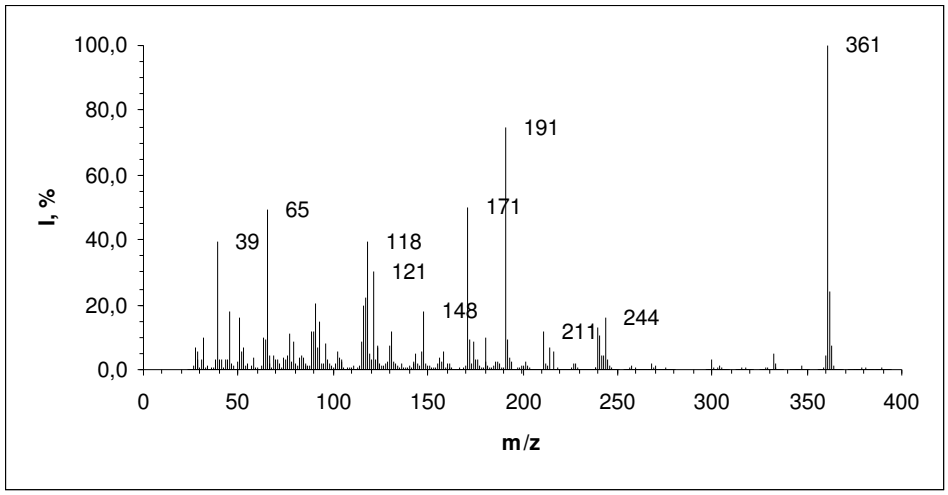


Figure 3. Substituted chalcone, R_1 = CH_3, R_2 = o-OH, M = 361

Abundant fragment ions appear at the carbonyl group on the phenyl side, with ion **j**, m/z = 93. Very important are the fragment ions on the bond between the

carbonyl and ethylene groups, m/z = 225 + R_1 (ion **g**) and m/z = 121, (ion **h**) and these ions are much more intense for the structures having the hydroxy group of the acetophenone ring in the para position. For the ortho substituent the fragment m/z = 225 + R_1 is less intense and is accompanied by a m/z = 225 + R_1 + H ion. A very important fragmentation appears by breaking two bonds of the thiazole ring accompanied by proton transfers. Ions m/z 174 + R_1 +2H (ion **c**) and m/z 172 -H (ion **d**)are very intense for the ortho substituent, but significantly less intense for the para substituent and permit therefore to distinguish between the ortho and para hydroxy substitution of acetophenone.

Mass spectra of two substituted epoxy ketones, Scheme 1, with epoxydated ethylene group, are shown in Figures 4 and 5. In all compounds of this type the substituted benzoyl group (ion **h**) is the base peak. Characteristic fragment ions are ion **g**, (M – benzoyl group) and triazole fragmentation, M – benzoyl – HCN, all confirmed by high resolution exact mass determination.

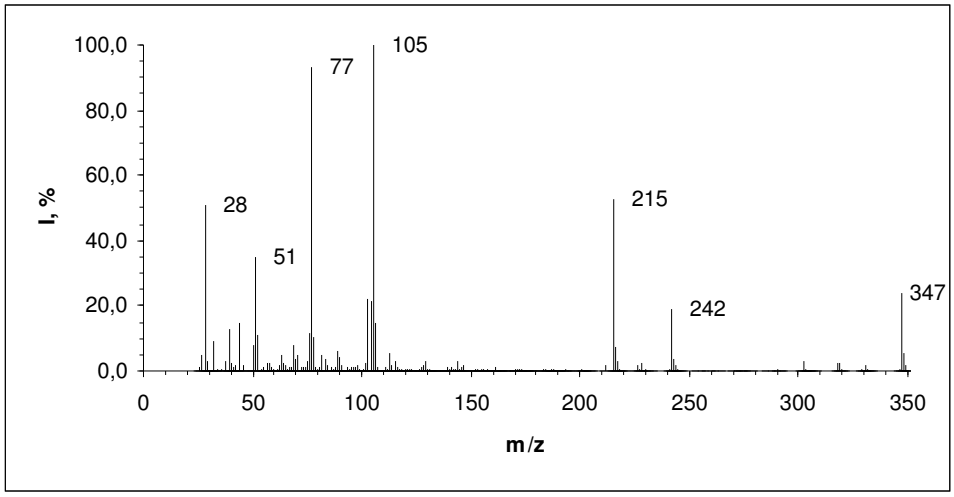


Figure 4. Substituted epoxy ketone, R_1 = H, R_2 = H, M = 347

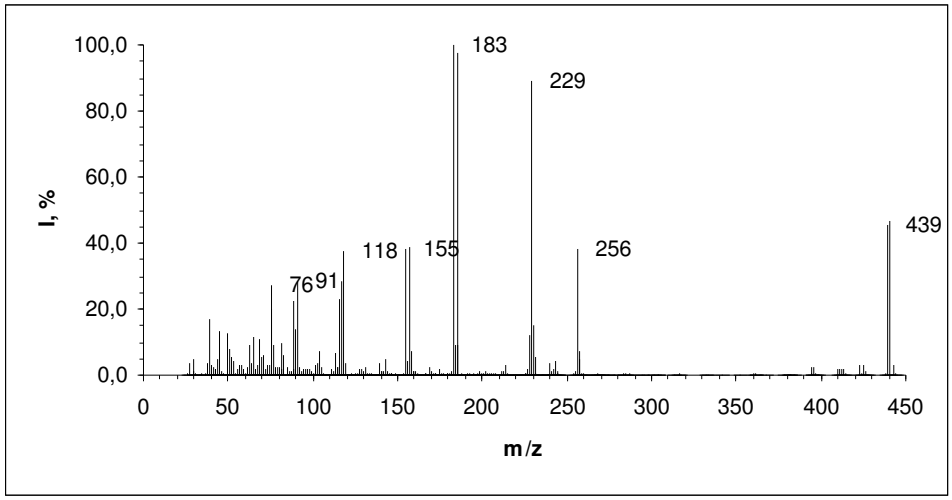


Figure 5. Substituted epoxy ketone, $R_1 = CH_3$, $R_2 = Br$, M = 439