Gas chromatography analysis of statins

Soňa Poláková (roz. Pavlovičová)

Abstract

A new GC method for determination of statins after derivatizacis using FID detector was developed. During the experiments the following results were achieved. Optimum separation results in the analysis of statins achieved in the following temperature gradient: one minute at 250 ° C, the temperature is further increased at 10 ° C / min to a final temperature of 320 ° C with other isothermal sections. A flame ionization detector was kept at the temperature 320 ° C. The optimum derivatization reagent was chosen BSTFA + TMCS for simvastatin, and N-tert-butyl (dimethylsilyl) trifluoroacetamide for atorvastatin. Addition of anhydrous potassium carbonate was obtained in alkaline environment, which allowed the derivatization. The best temperature for the derivatization of both drugs was obtained at 90 ° C. Time that is necessary to be run derivatization is 60 minutes with simvastatin and atorvastatin for 30 minutes. Quantification was performed in both cases using mefenamic acid as an internal standard, which was also derivatized, in the same derivatization agent. IS derivatization proceeded at 60 ° C for 60 minutes. The greatest yield response was achieved using half the amount of derivatization reagent than the amount of drug. Due to a better response was used injected sample volume and high pressure 8μl injection, when the pressure at the beginning of the column was 150 kPa for one minute. After derivatization of the analytes eluted as two peaks. The limit of detection is 6.4644 mg / ml for 1 peak simvastatin, 3.1251 mg / ml for 2 peak of simvastatin and 4.6250 mg / ml for atorvastatin. The limit of quantification is 21.5479 mg / ml for 1 peak of simvastatin; 10.4171 mg / ml for 2 peak of simvastatin and 8.1254 mg / ml for atorvastatin. A partial validation of the method was carried out.