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GAS CHROMATOGRAPHIC PROFILES OF PROSTAGLANDINS A, B, E AND F (SERIES I AND II) AND HISTIDINE METABOLITES

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SUMMARY

The possible applications of gas chromatography for the metabolic profiling of prostaglandins and histidine metabolites in biological samples have been explored, thus expanding our previous work on biogenic amines[1] to this important class of physiological components.

Authentic samples of the prostaglandins of series I and II (PGAs, PGBs, PGEs, PGFs) have been silanized by a one-step reaction with BSTFA–piperidine (1:1) at various temperatures (from room temperature to 60°) and times (0–4 h). The TMS-PG derivatives are stable for up to 40 h and can be chromatographed using 5% OV-17 and 5% OV-225 on Gas-Chrom Q, 100–120 mesh. The four prostaglandins belonging to series I can be resolved on OV-17 and OV-225 at 250° in 30 and 20 min, respectively. Series II prostaglandins can also be separated on OV-225 but PGE_2 and PGF_2 are not resolved on OV-17. Retention index data are presented. During the course of this work, evidence was obtained that significant quantitative losses can occur in the syringe itself prior to injection into the gas chromatograph, depending whether 1-μl (sample in metal needle) or 10-μl (sample in glass barrel) syringes are used. Proof is given of the quantitative importance of this problem.

The method has been applied to the study of prostaglandin profiles in extracts of samples of human seminal fluid. The extraction procedure has been significantly simplified by direct ultrafiltration of the samples. The gas chromatographic traces obtained show a consistent pattern that proves the reproducibility of the method.

Along the same lines, we have set up a method suitable for the simultaneous determination of the following histidine metabolites: histamine, 1,4-methylhistamine, 1,4-Methylimidazoleacetic acid and imidazoleacetic acid. These substances are directly silanized with a BSA–TMCS mixture containing 4% of TMCS. A study of the reaction yields has shown that a maximum response is obtained after 30 h at 60°. The TMS derivatives are injected into glass columns packed with 5% OV-17 on Gas-Chrom Q, 80–100 mesh, in which both amines and acids are completely resolved in less than 8 min. Retention index data are presented. The gas chromatographic traces thus obtained permit the quantitative and qualitative determination of the chromatographic profiles of these interrelated substances. Structures have been verified in all instances by mass spectrometry. The mass spectrometric patterns of all TMS derivatives provide various characteristic ions suitable for single- or multiple-ion monitoring[2]. An evaluation of the high specificity of this technique as applied to this type of samples is presented.

REFERENCES

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