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Note

Analysis of cannabinoids in cannabis by means of gas–liquid chromatography and solid injection

Improvements to the method

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A method based on gas–liquid chromatography (GLC) and solid injection has been described for the qualitative and quantitative determination of cannabinoids in milligram amounts of cannabis[1,2]. In this method, plant material is injected directly into the flash heater of the gas chromatograph by means of a solids injector. In order to improve the reliability of this method, two important aspects have been considered. One aspect concerns the choice of stationary phase. A wide variety of stationary phases have been used for cannabinoid analysis, but those of medium polarity (*e.g.*, OV-17) seem to be the most popular[3]. Stationary phases are often selected without bearing in mind the cannabis alkanes, which, under unfavourable conditions, may interfere in the analysis of cannabinoids[4]. Heptacosane and nonacosane are the major cannabis alkanes and, in Norwegian-grown *Cannabis sativa* L., heptacosane was found in concentrations ranging from 0.01 to 0.03% and nonacosane in concentrations ranging from 0.06 to 0.14% (ref. 5). It was shown that nonacosane may seriously interfere in the analysis of cannabinol on an OV-17 column. In order to avoid this interference, a non-polar column (SE-30) is preferred in combination with the solid injection technique. On such a column, heptacosane elutes after cannabinol and no interference of the major alkanes takes place. The C_{20}–C_{26} alkanes have retention times similar to those of the cannabinoids but the content of these is small and serious interference in the analysis of the major cannabinoids will not occur.

The other aspect concerns the injection technique. In the methods described, plant material was injected for 30 sec into the flash heater, which was held at 300°. The column was held isothermally at the operating temperature. In a quantitative analysis, accurately weighed plant material was injected for 30 sec into the flash heater and then for another 30 sec after the analysis of the constituents that evaporated during the first injection was completed. If complete evaporation not was achieved during the second injection, the plant material was injected for a third time. The percentage of the major cannabinoids evaporated during the first injection was then calculated and the content of the major cannabinoids determined by comparing their peak areas with a calibration graph.

This procedure is simplified by using cold trapping in combination with the solid injection technique. The plant material is then heated for a prolonged period

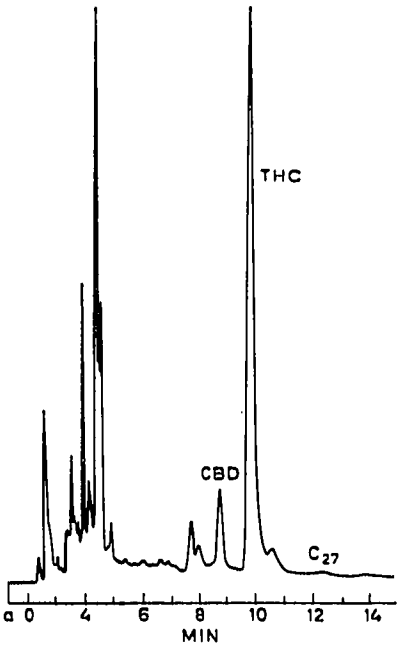

Fig. 1. Gas chromatogram of 1 mg of plant material analyzed by solid injection and cold trapping. The plant material was injected at (a) and the increase in column temperature started at zero on the time scale. CBD = Cannabidiol; THC = tetrahydrocannabinol. The sample did not contain detectable quantities of cannabinol.

in the flash heater until the cannabinoids are evaporated completely and trapped in the column. The column temperature is then rapidly increased and the volatiles are eluted. Peak broadening due to the injection is then eliminated and the peak areas can be compared directly with the calibration graph for quantitative analysis. Complete evaporation is achieved by injecting dried plant material for 1 min into the flash heater, which is held at 300°. Evaporated components are trapped in the column by using a column temperature of 40–50°.

Fig. 1 shows a gas chromatogram of 1 mg of plant material analyzed by solid injection and cold trapping. A Carlo Erba Fractovap 2300 gas chromatograph equipped with a flame ionization detector was used. The column was a 2 m long glass coil with an I.D. of 2 mm, filled with 3% SE-30 on Chromosorb W AW DMCS. The flash heater temperature was 300° and the column temperature was 40° during the injection. The column temperature was then increased to 250° in 3 min and held isothermally.

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