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Applications of Flame Ionization Detectors in Thin-Layer Chromatography

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I. INTRODUCTION

Conventional methods of quantification of fractions resolved by thin-layer chromatography (TLC) using techniques such as in situ spectrophotometry or photodensitometry are of limited utility for substances that contain weak or no chromophoric groups (1). Such fractions can be conveniently detected and quantified by sensitive vapor-phase detectors that are commonly used in gas chromatography (2,3). Several systems for quantitative TLC using vapor-phase detectors have become known in recent years.

In one type of system, the substances fractionated on the adsorbent layer are vaporized, fraction by fraction, by successive application of heat, and the products formed are driven to a thermal conductivity detector (TCD) or a flame ionization detector (FID). In an early device working on this principle (4,5), narrow quartz plates coated with an adsorbent such as silica gel are used for fractionation by TLC. After removal of the developing solvent, the chromatogram is encased in a rectangular quartz chamber and driven through a furnace, while nitrogen carrier gas flows through the chamber to an FID. Thereby, the fractions on the chromatoplate are vaporized consecutively by pyrolysis and/or evaporation, and the gaseous products from the various fractions are recorded as separate peaks.

In a recent modification of such a system, the substances separated on a TLC plate are consecutively vaporized by laser pyrolysis, and the resulting products are transported by a suitable carrier gas mixture to an FID or an electron capture detector for quantification (5a).

In my laboratory, the chromatoplate encased in a quartz chamber has been replaced by a chromatotube, i.e., a quartz tube whose inner surface is coated with a layer of silica gel or some other inorganic adsorbent (6). Fractionation on such chromatotubes is carried out as in conventional TLC and is followed by removal of the developing solvent by heating in a stream of an inert gas. Thereafter, the chromatotubes are scanned by driving them through a narrow furnace (800°C) while nitrogen, the carrier gas, flows through the tube to an FID. During scanning, the individual fractions are vaporized consecutively and monitored by the FID. The technique of TLC using chromatotubes, also termed tubular TLC (2,3,7), was later modified by using different principles of vaporization of the fractions, i.e., combustion in situ on an adsorbent containing cupric oxide and detection of the carbon dioxide formed in a TCD with the aid of helium as carrier gas (8,9). The techniques of pyrolysis and evaporation on an adsorbent such as silica gel and combustion on an adsorbent containing cupric oxide were subsequently integrated into a single instrumental system using the more sensitive vapor-phase detector, i.e., FID (10–12).

Tubular TLC-FID systems have been used so far mainly for the analysis of lipids and related substances. In this context, it should be of interest to note that tubular TLC systems have also been coupled with vapor-phase radiation detectors (2,9) and with a mass spectrometer (13,14).