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Phenols, Aromatic Carboxylic Acids, and Indoles

John H. P. Tyman

Brunel University, Uxbridge, Middlesex, England

I. PHENOLS

This chapter reviews developments in the thin-layer chromatography (TLC) of phenols, aromatic carboxylic acids, and indoles that have taken place since publication of the previous definitive text (1). Both natural and technical phenolic compounds are discussed, and the approach adopted is similar to that in other chapters. The phenolic group occurs in a vast array of natural products, extending from the alkaloids (e.g., morphine), through the lipids (e.g., polyunsaturated alkenylphenols), steroids (e.g., estrone), glycosides (arbutin, carminic acid), and cannabinoids to numerous other groups. At the risk of some duplication, this review therefore includes not only monocyclic phenols and phenolic acids but also some references to more complex systems. The majority of the investigations reviewed have been fundamental, and applied studies concerned with environmental, pollution, and toxicity problems have been relatively limited. With the increasing number of separatory processes available, their speed of application, and new commercial equipment, there has been a significant increase in quantitative studies aimed at improving detection limits. Impregnated layers have been more widely used in recent work, and with the availability of good chemically bonded commercial plates, the application of partition methods has increased. In general, standard methodologies have been used by workers, and the TLC of phenolic compounds has not been at the threshold of a novel "breakthrough" in technique. The development of high-performance thin-layer chromatography (HPTLC) and the potential for other innovations such as overpressured thin-layer chromatography (OPTLC) with this group of compounds is of considerable interest and counteracts an earlier feeling that high-performance liquid chromatography (HPLC) might displace planar techniques. To the contrary, in advanced work both approaches have often been used together. The art of chromatography is demonstrated in TLC, and the science in HPLC would have been true a decade ago. Now they are more indistinguishable. It is perhaps significant that one of the major scientific advances, "genetic fingerprinting," is a planar technique.

A. Preparation of Samples

The isolation of a concentrate of a natural or technical product is a vital operation requiring skilled application, and its importance cannot be overemphasized. At all stages of isolation processes, thin-layer chromatography can be used as a monitoring aid to follow the active material. At the point of sample application to the plate, the sample solvent can be of great importance. In TLC with liquid stationary phases on the support medium, the sample solvent should be immiscible to avoid streaking (2).

The unit operations of representative sampling, solvent extraction, steam distillation, column chromatography (cleanup), vapor absorption, freeze-drying, and chemical reaction followed by solvent extraction are but a few of the procedures discussed in the following examples.