

palladous chloride solution. Spots colored by spraying were scanned at 380 nm, compensating background at 600 nm, for sulfur compound analyses. An orange spot indicated the presence of dibenzothiophene, and a yellow one indicated the presence of sulfides.

Alkyl-phenyl sulfides, which are compounds typically present in fossil fuels, were separated on either cadmium acetate or silver nitrate–silica gel impregnated TLC plates with a salt/support concentration of 25% (45).

Other polar functional groups in PACs have been detected by spraying the plates with specific reagents. Tyrpien et al. (46) described the characteristics of these compounds before and after spraying with Fast Blue salt B.

5. Fullerenes

The retention and separation selectivity of C₆₀ and C₇₀ fullerenes have been evaluated on silica gel neutral and basic alumina, amino, C-18, diol plates, and silica gel impregnated with aqueous solutions (0.5–2.5%) of polymers such as polyvinylalcohol (PVA), polyvinylpyrrolidone, and poly(ethylene oxide) (47–49). The ability of fullerenes to form complexes with various polymers is useful for separating, purifying, and transforming them into water-soluble moieties.

Among the systems studied, the use of diol HPTLC plates and development with isooctane and of HPTLC silica gel plates and development with hexane–pyridine (95:5) gave successful separations. Although all the studied polymers increased retention of fullerenes with regard to silica gel, the use of PVA-impregnated plates and hexane elution improved separation selectivity. In all cases, separated peaks were inspected under UV light at 254 and 366 nm.

B. Applications

1. Petro-, Carbo-, and Geochemistry

Almost all coal, petrochemical, and geochemical applications of TLC involve the use of normal-phase adsorbents, usually silica gel (50). Usually, the separation of compounds is carried out by developing TLC plates with solvents of increasing or decreasing eluotropic strengths.

Applications of TLC involve (a) preparative fractionation of products, (b) qualitative identification of functional groups, (c) semiquantitative or quantitative hydrocarbon type analysis, and (d) planar size-exclusion chromatography.

a. Preparative Fractionation of Products. Sample fractions are collected either for further analysis by other analytical techniques (51–53) or for their use as external standards for quantitative determination of hydrocarbon types (17). Preparative fractionation is sometimes used as a rapid way to obtain quick comparative class separation of fossil fuel–derived products. Table 5 shows examples of preparative TLC of typical hydrocarbon-containing samples.

The thickness of the preparative silica gel layers used depends on the amount and nature of the sample. For fractionation of sedimentary organic matter this is about 0.25 mm for 10–20 mg and 2.5 mm for 20–90 mg (52). Shale oil (150–200 mg) was fractionated using a 0.75 mm layer (4). A gas oil (500 mg) was applied as a 180 mm band using the Linomat IV applicator and fractionated using a 2 mm thick silica gel layer (17).

Preparative TLC was useful for obtaining separation of bitumens with quantitative recovery of sample (52). This procedure is an alternative to traditional methods based on extraction. In this case, cyclohexane was used as developing solvent. Development time was 1–5 h depending on the thickness of the layer used. Spraying half of the layer with a solution of berberine sulfate in methanol and subsequent inspection under UV light gave the location of the following separated fractions: (a) saturated and unsaturated compounds; (b) aromatics, naphthoaromatics, and thiophenic compounds/ and (c) N, S, and O compounds including resins and asphaltenes. As usual, fractions were scratched, extracted with solvents, and further analyzed by GC/MS to identify the particular compounds of each fraction. It is possible to carry out a further separation between saturates and olefins on a second silica gel plate (0.5 mm thickness) that has been impregnated with an aqueous solution of silver nitrate (5 g/120 mL).

A similar scheme was followed to fractionate a shale oil (53). A rapid and reproducible separation into 14 fractions was obtained for this sample without requiring prior extraction of