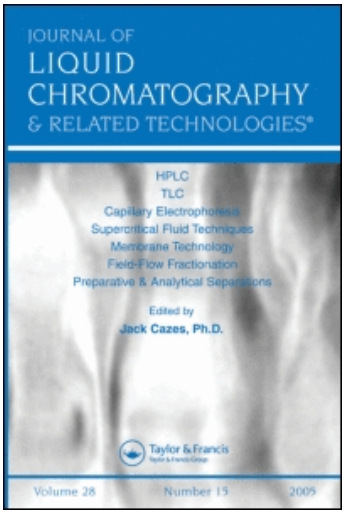

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ANALYSIS OF ANALGESIC TABLETS BY QUANTITATIVE HIGH-PERFORMANCE REVERSED PHASE TLC

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ABSTRACT

Aspirin, phenacetin, and caffeine in two commercial analgesic tablets were determined by scanning of fluorescence-quenched zones after separation on high performance TLC plates. Assays for the components ranged from 100 to 88% of label values, and agreement between duplicate samples was better than 5%. Use of phenacetin as an internal standard for determination of aspirin and caffeine was not advantageous. The analysis serves well as an introduction to quantitative TLC in undergraduate laboratory courses.

INTRODUCTION

Quantitative high performance TLC is a very useful method for content assays of pharmaceutical preparations. The ability to spot multiple samples along with bracketing standards leads to high

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sample throughput, and an internal standard is usually not required for accurate and precise determinations as is common in HPLC analyses. This paper demonstrates the analysis of commercial analgesic tablets containing aspirin and caffeine (designated AC) and aspirin, phenacetin, and caffeine (APC) on phosphor-containing reversed phase layers by scanning of fluorescence-quenched zones of the separated ingredients. Quantitative high performance TLC has recently received expanded coverage in educational journals (1) and analytical chemistry textbooks (2), and this analysis can readily serve as the basis for an undergraduate laboratory introduction to this increasingly important field in organic chemistry or instrumental analysis courses.

EXPERIMENTAL

Standard stock solutions were prepared by dissolving 790 mg of aspirin (acetylsalicyclic acid) in methanol in a 10.0 ml volumetric flask and 135 mg of caffeine and 500 mg of phenacetin in separate 25.0 ml volumetric flasks. An internal standard solution of phenacetin was prepared by dissolving 125 mg of phenacetin in methanol in a 100 ml volumetric flask. Working standard solutions for the AC tablet analyses were prepared by pipeting into three 25.0 ml volumetric flasks the following volumes (ml) of the aspirin and caffeine stock solutions, respectively: I-1.10 aspirin, 0.90 caffeine; II-1.40 aspirin, 1.60 caffeine; III-1.70 aspirin, 2.30 caffeine. After dilution to the line with methanol, the flasks

contained the following respective concentrations (μg/μl) of aspirin and caffeine: I-3.48, 0.190; II-4.42, 0.301; III-5.37, 0.366. For preparation of the sample, an AC tablet was placed in a 100 ml volumetric flask and crushed using a flattened glass rod. About 50 ml of methanol and a magnetic stirring bar were added to the flask, and the solution was stirred for 10 minutes. The bar was removed and the flask was filled to the line with methanol. The solution remained cloudy due to insolubility of inert binder ingredients, which were allowed to settle to the bottom of the flask.

For analysis of APC tablets, 0.50, 0.90, and 1.30 ml of aspirin, caffeine, and phenacetin, respectively, were measured into 25.0 ml volumetric flask I; 0.80, 1.60, and 2.50 ml into flask II; and 1.10, 2.30, and 3.80 ml into flask III. A 250 ml volumetric flask was used for dissolving the weighed APC tablet.

Analyses were performed on 20 cm x 20 cm chemically bonded C_{18} reversed phase layers containing a fluorescent phosphor and preadsorbent spotting strip and divided into nineteen 8-mm wide channels. Plates were prewashed by development with methylene chloride-methanol (1:1 v/v). For analysis of AC tablets, the three aspirin plus caffeine standard solutions and the sample were applied on adjacent lanes by streaking onto the preadsorbent using 10 μl Drummond microcap micropipets. For the determination of aspirin and caffeine in APC tablets, 10 μl of the three 3-component standards and the sample were applied to adjacent lanes. For the determination of phenacetin, 2 μl of the standards and sample were applied.

Plates were developed in a paper-lined, vapor-saturated rectangular chamber with methanol-0.5 M NaCl in 1% aqueous acetic acid (2:3 v/v) for a distance of 12 cm beyond the preadsorbent-C_{18} interface. The layer was air dried for 25 minutes and the separated zones were scanned in the single beam, transmission mode with a Kontes Model 800 scanner equipped with a 254 nm UV source (Co glass filter) and interfaced with a Hewlett-Packard 3390A recorder/integrator. The scanner light beam length was 8 mm (to match the lane width) and scan speed was 5 cm/minute. The recorder/integrator settings were attenuation 6, chart speed 5.0 cm/min, peak width 0.01, integrator function 2. Integrated peak areas from the standards were used to determine a linear least squares equation using a Commodore 64 computer program, and the micrograms of compounds represented by the sample peak areas were calculated by the computer from the calibration equation. Recoveries were calculated by comparing experimental and label values.

RESULTS AND DISCUSSION

Aspirin, caffeine, and phenacetin were completely resolved by a 12 cm development (ca. 1.5 hours) on the reversed phase layer, with respective R_F values of 0.65, 0.29, and 0.10. The plate number (N) for the phenacetin zone was ca. 1500. Figure 1 shows typical scans of the three separated compounds. Calibration curves for aspirin (30-55 µg), caffeine (1.5-4.0 µg), and phenacetin (1.5-7.0 µg) always had linear correlation coefficient (R) values greater than 0.98 and usually greater than 0.99.

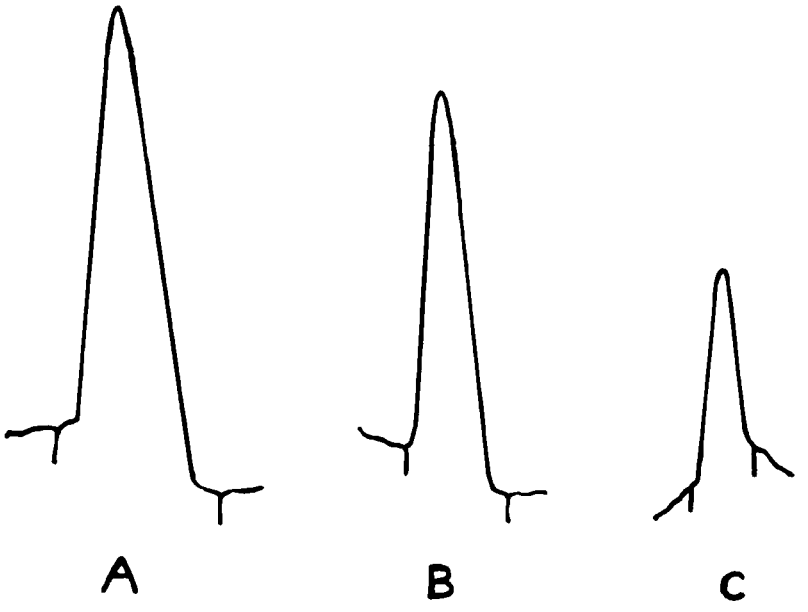

Figure 1. Densitometer scans of (A) aspirin (34.8 g), (B) caffeine (30.1 g), and (C) phenacetin (0.50 g) at 254 nm on phosphor-containing silica gel HPTLC plates. The attenuation setting of the integrator/recorder was x6 in each case.

Analyses of separate AC tablets gave the following results for aspirin (label value 454 mg/tablet) and caffeine (32.4 mg/tablet), respectively: Sample 1 - 453 (-0.2% error), 33.7 (+4.0%); Sample 2 - 424 (-6.6%), 32.2 (-0.6%); Sample 3 - 459 (+1.1%), 36.0 (+11%); Sample 4 - 470 (+3.5%), 29.8 (-8.0%); Sample 5 - 422 (-7.0%), 31.6 (-2.3%). When duplicate samples were spotted, individual results agreed within 0.2-2.4% for aspirin and 1.8-4.7% for caffeine. Analysis of three APC tablets gave the following respective results for aspirin (label value 227 mg/tablet), phenacetin (162 mg), and caffeine (32.4 mg): Sample 1 - 210 (-7.5%

error), 177 (+9.2%), 30.6 (-5.5%); Sample 2 - 215 (-5.3%), 153 (-5.5 %), 29.0 (-10%); Sample 3 - 199 (-12%), 170 (+4.9%); 29.6 (-8.6%). Reproducibility between duplicate samples of phenacetin was in the 1.9-4.5% range and for aspirin and caffeine was similar to the values given above for the AC tablet analysis.

The use of phenacetin as an internal standard for the determination of aspirin and caffeine in AC tablets was studied. Exactly 1.00 ml of internal standard solution was added to each standard flask and 4.00 ml to each sample flask before diluting to the line (0.05 mg/ml phenacetin in each solution). When the ratios of the scan areas (aspirin/phenacetin and caffeine/phenacetin) were used in the calculations, results agreed less closely with the label values than those given above. It is possible that phenacetin is not the optimum internal standard for this analysis, but other compounds were not tested. In general, internal standardization is less beneficial in TLC than in column chromatography because a unique calibration line is generated on each plate by processing samples and bracketing standards under the same conditions. The ability to spot multiple samples with standards on the 19 available lanes on each plate provides rapid analyses on a per-sample basis. Analyst attention is not required during the development and drying steps, and sample application onto the preadsorbent spotting area is relatively rapid compared to conventional layers.

The analysis could be completed by scanning UV absorbance of the three components directly on a layer without fluorescent

phosphor. A reflection scanner with a monochromator for selection of the optimum wavelength for each compound should be used in this case.

The procedure described is ideal for a six-hour undergraduate laboratory experiment illustrating quantitative TLC. In the first three-hour period, standard and sample solutions are prepared, the layer is prewashed, and spotting of the layer and operation of the densitometer are described and demonstrated. In the second period, samples are applied and dried, the plate is developed and dried, and zones are scanned. The report prepared by students can include calculations of the calibration equation, recoveries of ingredients compared to label values, and agreement between duplicate samples, as well as N (plate number), k' (capacity factor), α (selectivity), and R_s (resolution) values for the chromatographic zones.

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