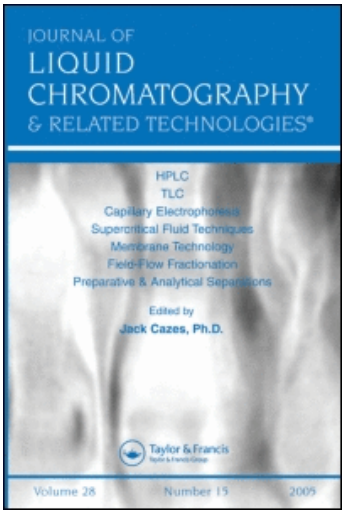

Journal of Liquid Chromatography & Related Technologies

Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713597273

Use of Aqueous Micellar Mobile Phases in Reverse Phase TLC

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To cite this Article Armstrong, D. W. and Bui, K. H.(1982) 'Use of Aqueous Micellar Mobile Phases in Reverse Phase TLC', Journal of Liquid Chromatography & Related Technologies, 5: 6, 1043 — 1050
To link to this Article: DOI: 10.1080/01483918208067567
URL: http://dx.doi.org/10.1080/01483918208067567

JOURNAL OF LIQUID CHROMATOGRAPHY, 5(6), 1043-1050 (1982)

USE OF AQUEOUS MICELLAR MOBILE PHASES IN REVERSE PHASE TLC

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ABSTRACT

Aqueous micellar solutions can be used in reverse phase TLC providing the ionic strength of the solution is sufficiently high to prevent the destruction of the stationary phase. Stability curves have been determined for sodium dodecyl sulfate and cetyltrimethylammonium chloride containing aqueous mobile phases. These "pseudophase" solutions allow the unique separation of hydrophobic from hydrophilic compounds. Indeed one can estimate the relative hydrophobicity of a compound by observing its chromatographic behavior in this system.

INTRODUCTION

Solutions containing normal and reversed micelles have been shown to make interesting and often effective mobile phases in liquid chromatography (1-5). In TLC one can use aqueous solutions of micelles and cyclodextrins as mobile phases in conjunction with polyamide and alumina stationary phases (1, 2, 5-7). It was previously impossible to use aqueous "pseudophase solutions" in reverse phase TLC because excess water caused the destruction of the stationary phase. Consequently only reversed micellar solutions (or other traditional, largely organic mobile phases) could be used in reverse phase TLC.

Recently, several bonded-reverse phase TLC plates have been introduced which can withstand a high percentage of water in the

mobile phase. This allows, for the first time, the use of aqueous micellar solutions in reverse phase TLC. Previously, aqueous micellar solution have been shown to make excellent mobile phases in reverse phase HPLC (3, 5).

EXPERIMENTAL

Materials

Chemically bonded octadecylsilane reverse phase TLC sheets (KC18F) were obtained from Whatman Chemical Separation Division, Inc. Highest available purity catechol, resorcinol, o, m and p-aminophenol were obtained from Aldrich and used as received. Highest available purity sodium chloride, phenol, methylene blue, methyl orange, fluorescence, bromocresol green and pyrogallol were obtained from Baker and used as received. Electrophoresis purity sodium doecylsulfate (SDS) was obtained from Bio Rad and cetyltrimethylammonium chloride (CTAC) was obtained from Pfaltz and Bauer. Deionized water was used in making up all aqueous solutions.

Methods

The stability of Whatman KC18F TLC sheets in aqueous surfactant-containing solutions was obtained as follows. Two by five centimeter pieces of the TLC plates were placed into separate stock solutions of 0.4 M, 0.3 M, 0.2 M, 0.175 M, 0.15 M, 0.125 M, 0.075 M, 0.025 M, 0.01 M and 0.001 M SDS and CTAC. The plates were allowed to soak between 5 and 48 hours (depending on the stationary phase stability). The time it took the stationary phase to fall from the backing was recorded. If the stationary phase remained on the backing for 48 hours, the plate was removed and scratched with the tip of a stainless steel spatula. If the stationary phase was no more easily scratched than in a new unused plate, it was deemed stable. If the stationary phase was easily removed upon scratching it was deemed intact but unstable. NaCl was then added (final [NaCl] = 0.1 M) to each of the stock surfactant solutions and the stability test was repeated. Identical stability tests were performed in surfactant solutions con-

taining 0.2 M, 0.3 M, 0.4 M, and 0.5 M NaCl. It took approximately four hours to completely develop a 20 cm chromatogram with a micellar mobile phase.

RESULTS AND DISCUSSION

The results of the stability tests are illustrated in Figures 1 and 2. It appears that the cationic surfactant (CTAC) is somewhat more compatible with the stationary phase than is SDS. A 0.4 M CTAC aq mobile phase can be used without adding extraneous NaCl. SDS mobile phases, however, always need a small amount of salt to stabilize the stationary phase. Using data from these tests, in conjunction with several trial separations, it was found that either 0.4 M CTAC aq or 0.2 M CTACl + 0.2 M NaCl aq mobile phases produced the most efficient separations on Whatman KC18F TLC sheets. As a general rule, one must keep the combined concentration surfactant plus NaCl $\geq$ 0.4 M. For any decrease in the concentration of surfactant in the mobile phase there must be an equivalent increase in the concentration of NaCl to maintain the stability of the stationary phase.

Aqueous micellar mobile phases produce unique separations when used in reverse phase TLC. The most unusual feature of this technique is the existence of two "solvent" fronts (see Figure 3). The first solvent front consists mainly of water, salt and perhaps some trace of surfactant monomer. The second front (referred to as the surfactant front) contains micelles as well. It is apparent that the stationary phase strongly adsorbs the surfactant from the mobile phase. As a result, there are no micelles in the initial solution moving up the TLC plate. Once the stationary phase is saturated with surfactant the micellar mobile phase begins to move up the TLC plate. Relatively water-insoluble, hydrophobic molecules cannot migrate further than the surfactant front (Figure 3A and 3B). While separations in this region are very good, one is limited to using 50% or less of the length of the chromatogram for the separation of hydrophobic compounds. Hydrophilic compounds, however, tend to migrate in the

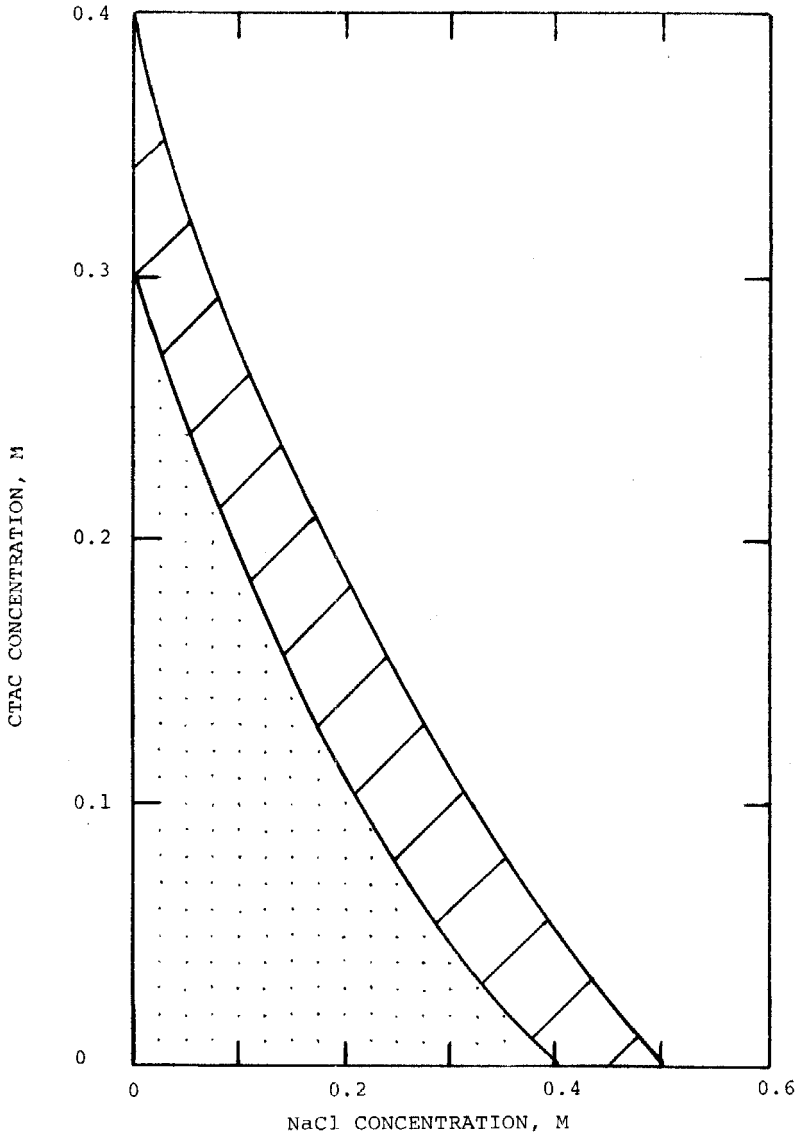

Figure 1. Stability curve which indicates the concentration(s) of CTAC and/or NaCl in an aqueous micellar mobile phase needed to maintain the integrity of the Whatman KC18F stationary phase. The left-hand curve of the cross-hatched area (/////) delineates the stable region. Use of mobile phase compositions falling in the stippled region (:::::) cause the stationary phase to come off the support. Use of mobile phase compositions falling in the cross-hatched areas result in a "metastable" stationary phase.

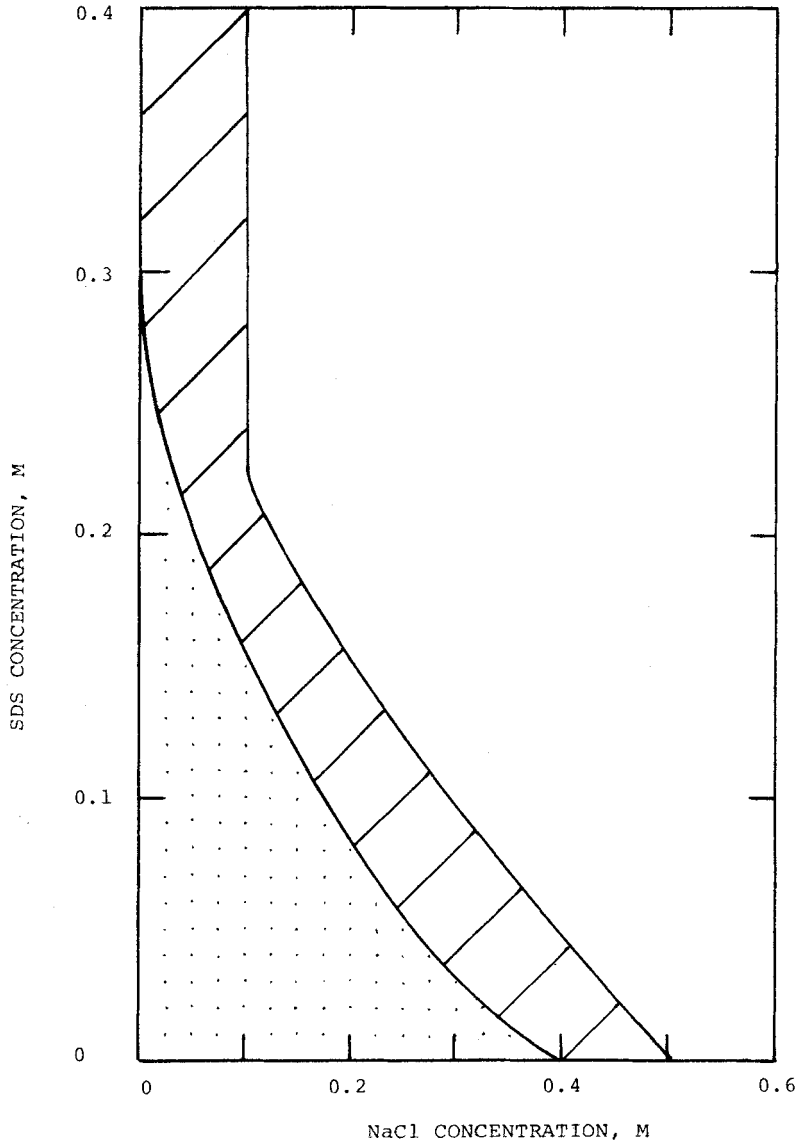

Figure 2. Stability curve which indicates the concentration(s) of SDS and/or NaCl in an aqueous micellar mobile phase needed to maintain the integrity of the Whatman KC18F stationary phase. The left-hand curve of the cross-hatched area (////) delineates the stable region. Use of mobile phase compositions falling in the stippled region (⁘⁘⁘) cause the stationary phase to come off the support. Use of mobile phase compositions falling in the cross-hatched areas result in a "metastable" stationary phase.

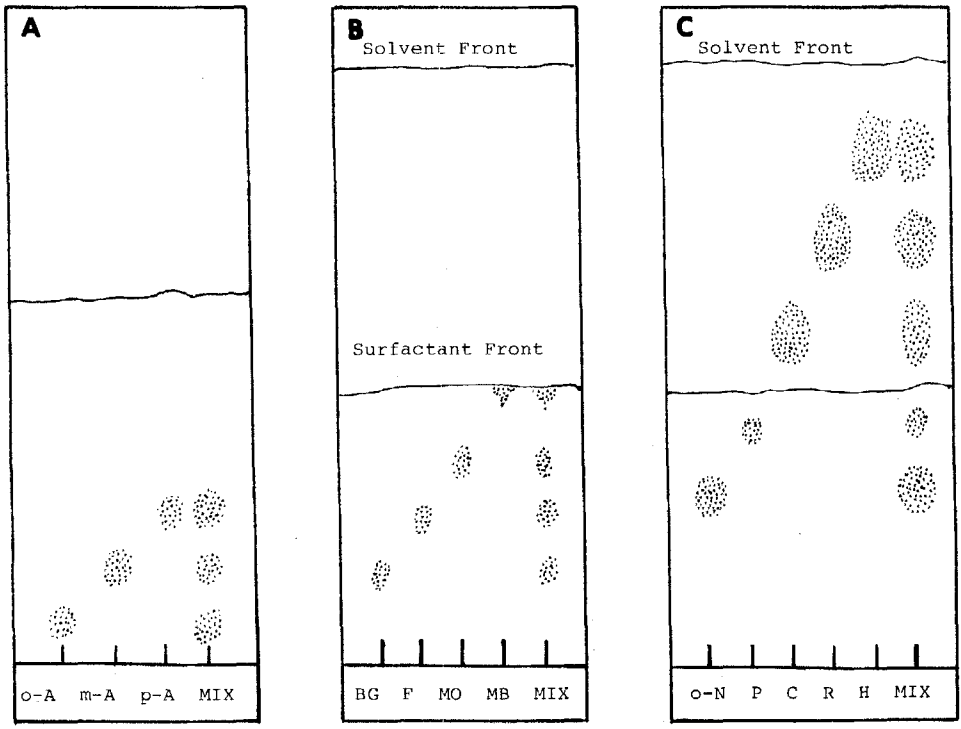

Figure 3. Reverse phase TLC chromatograms developed with aqueous micellar mobile phases. In chromatogram A the mobile phase consisted of 0.1 M CTAC + 0.3 M NaCl. o-A = o-aminophenol, m-A = m-aminophenol, p-A = p-aminophenol and MIX = a mixture of the three compounds. In chromatogram B the mobile phase consisted of 0.2 M CTAC + 0.2 M NaCl. BG = bromocresolgreen, F = fluoroscein, MO = methyl orange, MB = methylene blue and MIX = a mixture of the four compounds. In chromatogram C the mobile phase consisted of 0.2 M CTAC + 0.2 M NaCl. o-N = o-nitrophenol, P = phenol, C = catechol, R = resorcinol and H = hydroquinone, and MIX = a mixture of the five compounds.

region between the "solvent" front and the "surfactant" front (Figure 3C). Thus one has a potentially useful technique for separating hydrophilic from hydrophobic compounds. Furthermore, hydrophilic compounds (like hydrophobic ones) are separated from one another in their own section of the TLC plate. This phenomena is illustrated in Figure 3C. Catechol, resorcinol and hydroquinone are separated in the upper hydrophilic region of the TLC plate while phenol and o-nitrophenol are separated in the lower region containing the micellar mobile phase. Thus one not only has a means of simultaneously separating a range of different polarity compounds, but one can also estimate the relative hydrophobicity of a compound by observing its chromatographic behavior in this system.

ACKNOWLEDGEMENT

Support of this work by Whatman Chemical Separation Division is gratefully acknowledged.

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