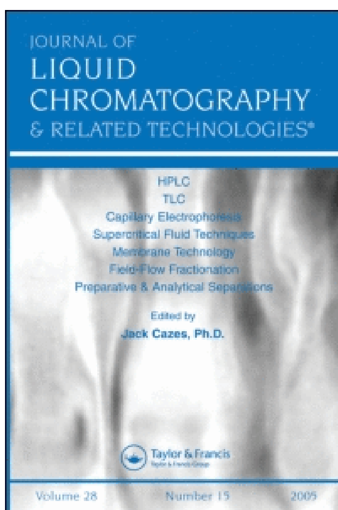
This article was downloaded by:
On: *25 January 2011*
Access details: *Access Details: Free Access*
Publisher *Taylor & Francis*
Informa Ltd Registered in England and Wales Registered Number: 1072954 Registered office: Mortimer House, 37-41 Mortimer Street, London W1T 3JH, UK



Journal of Liquid Chromatography & Related Technologies
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713597273

Quantitative HPTLC Determination of Elemental Sulfur in Sulfur Topical Medications

Jason R. McLaughlin[a]; Joseph Sherma[a]
[a] Department of Chemistry, Lafayette College, Easton, Pennsylvania

To cite this Article McLaughlin, Jason R. and Sherma, Joseph(1994) 'Quantitative HPTLC Determination of Elemental Sulfur in Sulfur Topical Medications', Journal of Liquid Chromatography & Related Technologies, 17: 20, 4445 — 4450
To link to this Article: DOI: 10.1080/10826079408013628
URL: http://dx.doi.org/10.1080/10826079408013628

PLEASE SCROLL DOWN FOR ARTICLE

Full terms and conditions of use: http://www.informaworld.com/terms-and-conditions-of-access.pdf

This article may be used for research, teaching and private study purposes. Any substantial or systematic reproduction, re-distribution, re-selling, loan or sub-licensing, systematic supply or distribution in any form to anyone is expressly forbidden.

The publisher does not give any warranty express or implied or make any representation that the contents will be complete or accurate or up to date. The accuracy of any instructions, formulae and drug doses should be independently verified with primary sources. The publisher shall not be liable for any loss, actions, claims, proceedings, demand or costs or damages whatsoever or howsoever caused arising directly or indirectly in connection with or arising out of the use of this material.

JOURNAL OF LIQUID CHROMATOGRAPHY, 17(20), 4445-4450 (1994)

QUANTITATIVE HPTLC DETERMINATION OF ELEMENTAL SULFUR IN SULFUR TOPICAL MEDICATIONS

JASON R. MCLAUGHLIN AND JOSEPH SHERMA
Department of Chemistry
Lafayette College
Easton, Pennsylvania 18042-1782

Downloaded At: 07:45 25 January 2011

ABSTRACT

A method was developed for determination of elemental sulfur in sulfur topical pharmaceutical preparations involving separation on a preadsorbent HPTLC silica gel plate with fluorescent phosphor, detection by fluorescence quenching, and quantification by densitometric scanning. Sulfur was directly detected and quantified on the plate at levels as low as 800 ng, and no interference was encountered from other ingredients in the medication formulations. The method was applied to the analysis of commercial liquid and cream preparations having respective label values of 5% and 8% sulfur, and recoveries averaged 99.3 and 99.5%. An unknown cream medication was assayed using the method and the accuracy of the result was validated by standard addition. All analyses were carried out with 5 or 6 replicates to evaluate precision, and coefficients of variation ranged from 2-4%.

INTRODUCTION

Elemental sulfur is an active ingredient in a variety of topical ointment, cream, and liquid pharmaceutical preparations for control of acne. The official methods for determination of sulfur in these products involve chemical

Copyright © 1994 by Marcel Dekker, Inc.

oxidation or reduction followed by titration or gravimetric analysis (1). These methods are slow and are not sufficiently selective for many of the complex formulations currently being marketed. A method involving separation of sulfur by TLC on homemade silica gel G plates, recovery by scraping off of the sulfur band and elution with chloroform, and analysis by spectrometry at 265 nm was recently reported (1). In this paper we report a quantitative method that is based on separation of sulfur on commercial high performance preadsorbent silica gel plates containing fluorescent indicator, detection by fluorescence quenching, and in situ densitometric scanning at 277 nm. This direct method is faster, more convenient, and less prone to random loss of analyte during the scraping and elution operations compared to the earlier method.

Downloaded At: 07:45 25 January 2011

EXPERIMENTAL

Standard Solution

A standard solution (200 ng/ul) was prepared by adding 10.0 mg of reagent grade elemental sulfur (99.999% purity, Janssen Chimica) to 25 ml of acetone in a 50 ml volumetric flask. The solution was boiled on a hot plate for ca. 30 min, with additions of acetone as needed to maintain the volume, until the sulfur was completely dissolved. The solution was cooled to room temperature and the flask was filled to the line with acetone and mixed thoroughly by repeated inversion for 45 sec.

Thin Layer Chromatography

TLC was carried out on 10 x 20 cm Whatman LHPKDF high performance silica gel plates with 19 lanes, preadsorbent spotting area, and fluorescent phosphor (catalog no. 4806-711). Standard and sample solutions were applied to the preadsorbent using a 10 ul Drummond digital microdispenser. Plates were developed with petroleum ether for a distance of 7 cm beyond the silica gel-preadsorbent junction in a paper-lined, solvent-saturated Camag twin-trough chamber and dried in a fume hood. Separated sulfur zones were detected by inspection under 254 nm UV light in a viewing cabinet and scanned using a Shimadzu CS-930 densitometer in the single beam, reflectance mode at 277 nm.

Downloaded At: 07:45 25 January 2011

Analysis of Samples

The samples analyzed to test the new method were an acne medication cream with a label declaration of 8% sulfur, a liquid medication with a label value of 5%, and another cream containing an unspecified amount of sulfur, all purchased in a local pharmacy without prescription. Approximately 250 mg of the 5% liquid was poured into a tared 25 ml volumetric flask, the flask was reweighed to obtain the exact sample weight by difference, the sulfur in the sample was dissolved by boiling with ca. 15 ml of acetone, and the solution was cooled to room temperature and diluted to the line with acetone. Approximately 156 mg of the 8% cream or 250 mg of the unknown were accurately weighed by difference into a tared 50 ml

beaker and quantitatively transferred into a 25 ml volumetric flask with acetone, and the solution was boiled and diluted to the line as described above.

Recovery from the known samples was determined by spotting 4.00, 8.00, and 12.00 ul of standard (containing 0.800, 1.60, and 2.40 ug of sulfur, respectively) and duplicate aliquots of sample solution that represented a theoretical weight equal to the 8.00 ul of standard. Based on the sample weights specified above, 3.20 ul was spotted for both samples (containing 1.60 ug of sulfur for 100% recovery). A calibration curve was constructed from the scan areas and spotted weights of the standards using a Quattro curve-fitting program on an IBM PC, the weight of sulfur in the sample was interpolated from the curve using the average scan area of the duplicate sample aliquots, and percent recovery was calculated by comparing the experimental and theoretical sample weights.

The percent sulfur in the unknown was determined by spotting 4.00, 8.00, and 12.00 ul aliquots of standard and 2.00, 3.20, 5.00, and 8.00 ul of the sample. The scan areas and weights of the standards were used to produce the calibration curve, and the weight of sulfur was interpolated from the curve using the scan area of the 5.00 ul sample zone, which most closely matched the 8.00 ul standard. The percent sulfur was calculated based on the weights of sulfur and sample (250 mg) and the volumes of the total sample (25 ml) and the aliquot spotted (5 ul).

Downloaded At: 07:45 25 January 2011

RESULTS AND DISCUSSION

Boiling with acetone completely dissolved the sulfur in the standard and samples, but other insoluble sample ingredients settled to the bottom of the volumetric flasks and did not interfere with spotting. Sulfur produced a tight band with R_f 0.63 across the lane of the high performance preadsorbent silica gel plate when developed with petroleum ether. Despite the presence of many ingredients in the medications analyzed, including resorcinol, fragrances, and methyl and propyl parabens, zones other than sulfur were not detected by fluorescence quenching under 254 nm UV light in any of the chromatograms. Standards and samples were always chromatographed together to correct for the inevitable slight variations in the slope and intercept of the calibration curve on different plates. The calibration curve typically had a linearity correlation coefficient (R value) of 0.99.

Downloaded At: 07:45 25 January 2011

The liquid medication with a 5% label value of sulfur was analyzed 5 times, and the percent recovery values obtained were 97.0, 96.9, 98.8, 104, 99.5 (99.3 average, 3.0 standard deviation). Percent recovery values for 6 analyses of the cream with an 8% label value were 98.3, 96.3, 102.3, 102.1, 98.3, 99.5 (99.5 average, 2.3 standard deviation).

The analysis of the unknown cream was replicated 5 times with the following results: 3.20, 3.16, 2.93, 3.28, 3.00%; average 3.11%; standard deviation 0.14%. A standard addition analysis was performed in duplicate using a spiked sample to validate these results for the unknown. A weighed amount of solid sulfur was added to 25.0 ml of preanalyzed sample

solution to exactly double the content of sulfur, and the solution was heated to dissolve the sulfur and diluted to 50.0 ml with acetone. Duplicate 5.00 ul aliquots of the spiked and unspiked sample solutions were chromatographed, and the average scan areas were found to agree within 0.05 and 0.20% (relative error), thereby verifying the original analysis of the unknown.

The quantitative HPTLC method described is simple because layer preparation, sample preparation, or scraping and elution of the separated sulfur zones are not required, and it has high sample throughput because up to 16 samples can be analyzed on a single plate along with the three required standards. It has been shown that the method has the necessary sensitivity, selectivity, accuracy, and precision for routine use in a pharmaceutical analytical laboratory.

Downloaded At: 07:45 25 January 2011

REFERENCE

1. A.K. Sanyal, B. Chowdhury, and B. Banerjee, J. AOAC Int. 76, 1152 (1993)

Received: June 12, 1994
Accepted: August 8, 1994