Abstract

Evaporative light scattering detection (ELSD) is gaining popularity for the analysis of many difficult analytes, such as dimethicone. Its versatility enables the detection of any sample that is less volatile than the mobile phase. As a universal detector the ELSD does not depend on optical characteristics. Therefore, any compound in the sample may be detected with nearly equivalent response factors. This provides a more accurate representation of actual sample concentration. Unlike RI and low-wavelength UV, the ELSD is gradient compatible for improved resolution and faster separations. The ELSD is also more sensitive than an RI detector. It provides a more stable baseline and is not subject to temperature changes, eliminating the need for a column heater.

This Application Note focuses on the utility of an ELSD for the analysis of dimethicone. A reverse phase HPLC method was developed for the separation of dimethicone. Sample preparation involving solvent extraction and separation will also be discussed.

Introduction

Dimethicone is a silicone derived oil. It is available in a complete range of viscosities from the highly mobile 1-20cs, to the favorite cosmetic range of 50-500cs and then the 1000-200,000cs heavy oils, and silicone gels.[1] It is a common ingredient in cosmetics, hair care products, pharmaceutical applications and food processing. In cosmetics and hair care products it serves as a spreading agent and moisturizer, adding luster and sheen. In pharmaceutical processing it is used as an antifoam agent and in food processing as a defoaming agent. Although it is a widely used ingredient, the detection and analysis of dimethicone is difficult and limited. One of the current methods for analyzing dimethicone is by Fourier transform infrared spectral subtraction (FTIR). This method is complicated, time consuming and costly.

Coupled with an HPLC system, the ELSD gives a new alternative for the analysis of dimethicone. Its unique method of detection is the key to its versatility. The column effluent enters the detector’s nebulizer where it mixes with nitrogen gas to produce an aerosol spray. The fine mist travels through a heated drift tube, where the mobile phase is evaporated. The remaining sample particles pass through a flow cell where they scatter light from a laser diode. The light is then detected by a silicon photodiode, producing an electrical signal. Through this method of detection the ELSD easily detects non-volatile samples with sensitivity and precision.

Experimental

Standard Preparation

The Dimethicone standard of 200cs was dissolved in Chloroform and diluted with Acetonitrile.

Sample Preparation:[2]

Approximately 1.5g of hand lotion containing Dimethicone of 200cs was weighed out into a 50.0mL graduated cylinder with a ground glass stopper. Using a pipette, 20.0mL of 37% by wt HCL solution and 15.0mL of methylene chloride were added. The mixture was shaken vigorously, manually, for one minute and allowed to separate into layers. A 10.0mL aliquot of the bottom organic phase, was placed into a 10.0mL disposable syringe. The syringe was attached to a 25mm syringe filter (0.45µm pore size, Alltech Assoc., Part No. 2090) and a Maxi-Clean silica cartridge (600mg, Alltech Assoc., Part No. 20978). The aliquot was passed through the system and collected into a 25mL erlenmeyer flask. The collected effluent was evaporated almost to dryness using a hot plate, being careful not to let the solution boil. The residue was taken up in 75:25 acetonitrile:chloroform and diluted, volumetrically to 10.0mL. Using a 10.0mL disposable syringe the solution was then passed through a Maxi-Clean silica cartridge (600mg, Alltech Assoc., Part No. 20978).
**Discussion**

Dimethicone is a difficult ingredient to analyze. It contains no UV chromophores and depending on its viscosity, has limited solubility. For this particular sample, dimethicone had a viscosity of 200cs and was soluble in chloroform. Using the reverse phase HPLC method developed, the separation of dimethicone was easily achieved. However, the method involved a chloroform acetonitrile gradient, eliminating the possibilities of UV or RI detection.

UV detection is not possible. Due to the absence of UV chromophores dimethicone would produce no response. Also, chloroform has a high UV cutoff. At low wavelengths detector saturation will occur and at mid-range wavelengths baseline drift will occur. RI detection is also not possible with this method. Although it is a universal detector, and can detect dimethicone, it is not compatible with gradient elution. RI detectors are also not as sensitive as an ELSD. They are susceptible to ambient temperature changes and require the use of a column heater in order to maintain baseline stability. If the baseline is not stable, the limit of detection decreases.

Although the detection and separation of the dimethicone standard was easily achieved, the sample was not completely successful. As illustrated in the sample chromatogram, there are interfering components within the first two minutes of the dimethicone separation. These components could not be separated. Often samples containing dimethicone also have similar components such as cyclomethicone or dimethicone copolyol. The extraction and recovery of only dimethicone from a sample containing similar components can prove to be challenging. The sample preparation described here may be successful for some matrices. However, for this particular hand lotion additional sample preparation will be needed.

**Conclusion**

A reverse phase HPLC method was developed for the detection of dimethicone using an evaporative light scattering detector. This method showed excellent separation of dimethicone within twenty minutes. With this method it is possible to analyze dimethicone in cosmetics, pharmaceutics and food preparations. Due to interfering sample components, additional sample preparation is needed.

**References**

1. A. & E. Connock (Perfumery and Cosmetics) Ltd, website @http://www.connock.co.uk/silicone.htm.


**Acknowledgements:**

Thanks to Dr. Ed Burt from Andrew Jergens, Cincinnati OH, for providing the dimethicone standard of 200cs and a sample of hand lotion containing dimethicone of 200cs.

**Column**

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