DiamondAnalytics
tools for tomorrows chemistry

Diamond Analytics Application Note: DA1000-A

Flare Mixed-Mode Column: Separation of 2,4-D, MCPA, and Dicamba

Bhupinder Singh[1]; David S. Jensen[2]; Andrew J. Miles[2]; Andrew E. Dadson[2]; Matthew R. Linford[1]
[1] Department of Chemistry and Biochemistry, Brigham Young University, Provo, UT-84602, USA
[2]Diamond Analytics, 1260 S 1600 W, Orem, UT-84058, USA

Introduction

2,4-D (2,4-dichlorophenoxyacetic acid), MCPA (2-methyl-4-chlorophenoxyacetic acid), and dicamba constitute a major portion of the broad leaf herbicides used in agriculture.[1,2] In addition, major agrochemical companies are planning to introduce 2,4-D and dicamba resistant crops into the market, which will be associated with increased use of these herbicides. The environmental impact of these herbicides is also of great interest to various regulatory bodies.[3-5] If present in 2,4-D formulations, MCPA, a close chemical analogue of 2,4-D, can ruin the growing seedlings of 2,4-D resistant crops. Unfortunately, 2,4-D and MCPA are a critical pair – difficult to separate quickly and economically by conventional LC.[1, 6] It is very important to have a method that can completely separate these analytes. Here we present a rapid, baseline separation of 2,4-D, MCPA, and dicamba on the Diamond Analytics Flare mixed-mode column.

Experimental

General: Isocratic elution was used to separate a mixture of 2,4-D, MCPA, and dicamba. A mobile phase of water and acetonitrile was spiked with formic acid as an additive. Analytes were purchased from Sigma-Aldrich (St. Louis, MO).

Chromatograph: Agilent 1290 Infinity Binary LC, DAD, ChemStation software

Sample: 2 mg/mL of 2,4-D, MCPA, and dicamba in a mixture of 1:1 (Acetonitrile and H_2O)

Column: Flare Mixed-Mode Column (4.6 × 3.3 mm, 4.0 µm)

Injection volume: 2.0 µL

Temperature: 60 °C

Flow rate: 1.0 mL/min

Detection: UV detection at 280 nm

Mobile Phase:

A: 1.5 % formic acid in H_2O (pH 2.0)

B: 1.5 % formic acid in Acetonitrile

Elution: Isocratic: A:B :: 10:90

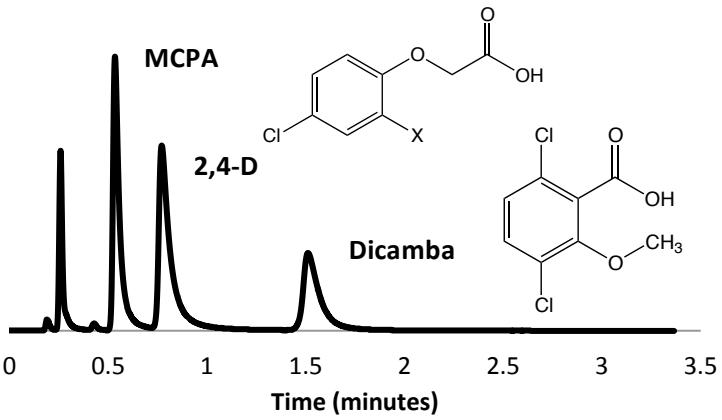

Figure 1. Isocratic separation of MCPA (-X = -CH_3), 2,4-D (-X = -Cl), and dicamba (280 nm).

Results and Discussion

2,4-D, MCPA, and dicamba were baseline-separated in less than 2 minutes by isocratic elution on the Diamond Analytics Flare column, where 2,4-D and MCPA are a critical pair. Simple UV detection of the analytes at 280 nm was employed. The mobile phase was based on acetonitrile and water with formic acid as an additive. The aqueous mobile phase was at a relatively

low pH (2.0). The Flare column can be used in a straightforward manner to separate these analytes.

References

1. A. Balinova, J Chromatogr A **728** (1-2), 319-324 (1996).
2. R. J. Flanagan and M. Ruprah, Clin Chem **35** (7), 1342-1347 (1989).
3. W. G. H. Johnson, S.G.; Legleiter, T.R.;Whitford, F.; Weller, S.C; Boredelon, B.P.; Lerner, B.R., (2012).
4. http://www.centerforfoodsafety.org/wp-content/uploads/2012/02/FSR_24-D.pdf, B. Freese, (2012).
5. N. P. I. Center, in *National Pesticide Information Center* (npic.orst.edu/factsheets, http://npic.orst.edu/factsheets/2,4-DTech.pdf).
6. http://www.restek.com/pdfs/pres-2005-260-13p.pdf, J. S. Kowalski, K; Nolan, L. and Wittrig, R. (Restek Corporation).