

# ANALYSIS OF TWO REACTION PRODUCTS

by Direct Analysis Probe on a Compact Mass Spectrometer

Advion

## INTRODUCTION

The expression CMS provides essential compound information quickly and improves the chemist's workflow. The Atmospheric Solids Analysis Probe (ASAP<sup>®</sup>) permits fast analysis of solid and liquid samples and is a simple, low cost alternative to LC/MS methods. This direct analysis probe method has been shown to be useful for the analysis of volatile and semivolatile compounds. ASAP is available as a combined ASAP-APCI source, or existing APCI sources can be easily modified to become dual APCI-ASAP capable.

## EXAMPLE

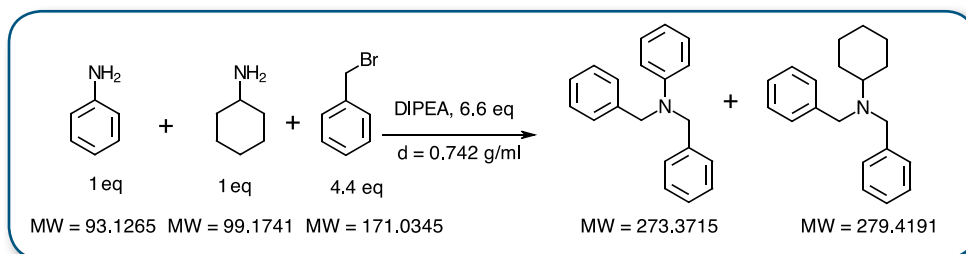


Figure 2: Alkylation of Aniline and cyclohexylamine with benzylbromide

To show the utility of the ASAP probe in identification of the products of a reaction where two products are possible, the alkylation of both aniline and cyclohexylamine and benzylbromide, following the method of Moore et al is demonstrated. To a 5 ml V-vial containing 3 ml of anhydrous acetonitrile was added 1 equivalent of aniline and cyclohexylamine. Then 6.6 equivalents of diisopropylethylamine (DIPEA) was added and the solution was allowed to mix well with stirring. Then 4.4 equivalents of benzylbromide was added quickly and a timer was started. At each timepoint, 10  $\mu$ l of the reaction mixture was diluted in 1 ml of 30% acetonitrile and 70% 0.1M ammonium formate and vortexed to mix it well. The ASAP probe was then dipped in the diluted solution and analyzed in triplicate. Samples were taken at 1, 5, 10, 30, 60, 120 and 180 minutes. The samples were analyzed by ratio of the product ion / sum of the starting amine and product ion \* 100 to give the percent of product formed. Because of the fast reaction seen for cyclohexylamine, the reaction was repeated using an ice bath. The results of the study are shown in the Table 1.



Figure 1: Photos of ASAP probe and glass capillary tip which supports the sample for analysis

The results shown are the average  $\pm$  standard deviation of the results of three experiments where each timepoint was analyzed in triplicate. (ND = sample not analyzed.)

To compare the results obtained using full scan versus selected ion monitoring (SIM), the reaction of aniline and benzylbromide was repeated. Data were collected using full scan mode and the selected ion monitoring of the key ions of aniline and dibenzylphenylamine. The results of the comparison are shown in the Figure 3.

## SUMMARY

- Direct analysis of a reaction mixture by a modified APCI source with the ASAP probe provided unequivocal reaction identification.
- Easy-to-use and reliable mass analysis system
- Results are generated in less than 30 seconds

TIME	CYCLOHEXYLAMINE REACTION		ANILINE REACTION
	ROOM TEMPERATURE	ICE BATH	ROOM TEMPERATURE
1	95.91 $\pm$ 2.20	0.00 $\pm$ 0.00	1.66 $\pm$ 1.46
5	99.44 $\pm$ 0.13	74.53 $\pm$ 10.39	11.11 $\pm$ 2.98
10	99.60 $\pm$ 0.09	95.38 $\pm$ 1.79	18.55 $\pm$ 2.52
30	98.91 $\pm$ 0.59	97.82 $\pm$ 1.03	60.66 $\pm$ 8.13
60	99.47 $\pm$ 0.23	98.85 $\pm$ 1.22	73.24 $\pm$ 5.29
120	99.68 $\pm$ 0.21	99.70 $\pm$ 0.03	82.15 $\pm$ 14.87
180	99.76 $\pm$ 0.04	ND	89.81 $\pm$ 5.95

Table 1: Aniline and cyclohexylamine with benzylbromide analyzed at time points by ASAP enabled APCI source on CMS.

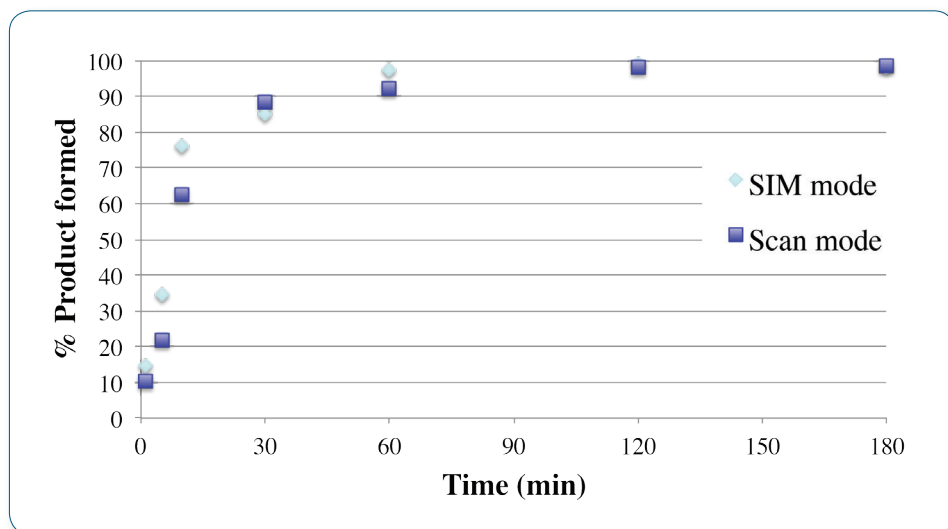


Figure 3: The analysis indicates that there is close correspondence between the results obtained using either full scan or SIM monitoring.

## REFERENCES

<sup>1</sup> Jason L. Moore, Stephen M. Taylor and Vadim A. Soloshonok, Archive for Organic Chemistry, 2005 Vol 6, p 287-292

Charles N. McEwen,\* Richard G. McKay, and Barbara S. Larsen, Anal. Chem. 2005, 77, 7826-7831

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