

CDSolutions

APPLICATIONS INFORMATION USING ADVANCED SAMPLE HANDLING TECHNOLOGY

Quantitation of IPA Using the CDS 7000 Purge & Trap with the Heater Option

Organic trace volatiles such as halogenated hydrocarbons, aromatics, and polyaromatics have been qualified and quantitated using purge and trap for many years. The analytes are usually purged from water using an inert carrier gas (either He or N₂) and trapped onto an adsorbent bed which is then thermally desorbed to a GC/MS for quantitation. Although alcohols like isopropanol are water soluble, they may also be assayed using this technique. Heating the water makes them more volatile and enhances the recovery.

A 10 μMole solution of isopropyl alcohol was prepared, and serial dilutions were made from the stock consisting of 5 μM, 1 μM, 0.5 μM, and 0.25 μM concentrations. A 5 ml sample of each dilution was placed into the standard 5 ml sparging vessel of a CDS 7000 sample concentrator, which was interfaced to a GC/MS (ion trap). A standard heater option was attached to the sparging vessel. The aqueous sample was heated to 85°C while being purged for 15 minutes, and the trap (Vocarb 3000) was desorbed at 260°C for 5 minutes. Three replicate runs were made with each concentration.

A normal mass scan of IPA shows poor peak shape at lower concentrations. The technique of SIS (selective ion storage) was used which has much greater sensitivity and gives sharper peaks. Masses m/e 44, m/e 45 (base peak), and m/e 46 were selected.

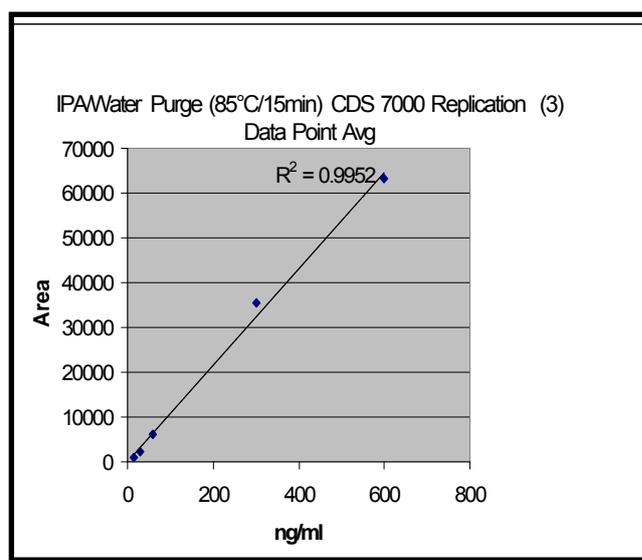


Figure 1. Linearity plot average of 3 runs.

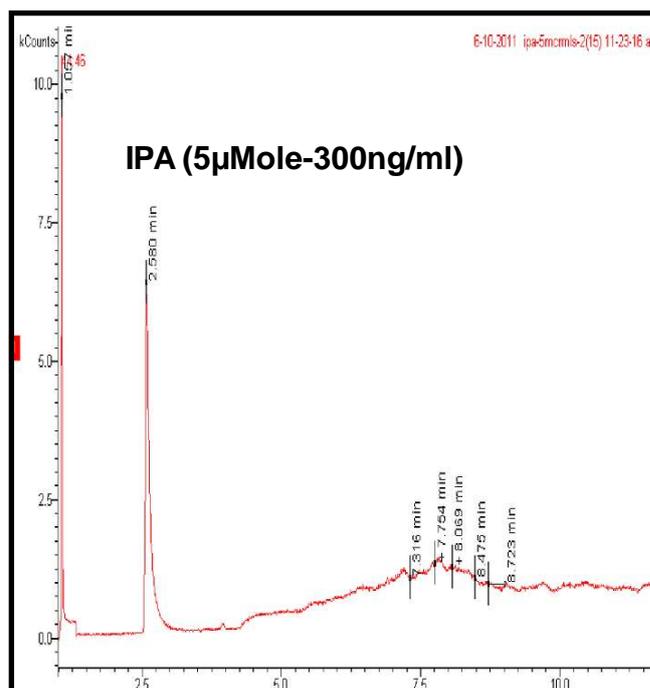


Figure 2. Peak of IPA (SIS).

Figure 1 shows a three data point average plot for each specified concentration. The area of each average was plotted against the concentration in ng/ml. The R squared value is 0.99. Figure 2 shows a well defined peak of IPA using the selective ion storage of the ion trap. Figure 3 is a picture of the CDS 7000 with the optional heater.



Figure 3. CDS 7000 with optional heater.

Experimental Conditions

CDS 7000

Purge Volume: 5ml
Purge Time: 15 minutes @ 85°C
(Heater)
Purge Flow: 60ml/minute
Desorb preheat 85°C/3 minutes
Desorb: 260°C/5minutes
Trap Bake: 260°C/10minutes
Valve Oven: 150°C
Transfer Lines: 150°C
Wet Trap Bake: 260°C

GC/MS

Column: Varian CP 624 CB
30m x 0.25mm x 1.4 µm
Flow Rate: 1.3ml/minute
Split Ratio: 20:1
Program: 40°C/2minutes,
100°C/10°C/minute, hold 5min

Mass Spectrometer: Ion Trap

CDS Analytical, Inc. has been a leader in the design and manufacture of laboratory instruments for sample preparation and analysis since 1969. We are dedicated to providing the best possible instruments for both research and routine analysis. Well known in the field of pyrolysis, CDS manufactures the Pyroprobe® 5000, 5150, 5200 and 5250 autosampler for the introduction and analysis of solid materials by GC, MS and FT-IR. CDS offers a complete line of dynamic headspace instruments for the analysis of volatile organic compounds in environmental, pharmaceutical and food applications, including the model 8400 four-position autosampler for complex, multicomponent materials investigation. Our customers, their requirements and applications are important to us. To help meet your needs, we offer a wide range of analytical information and the services of our applications laboratory. If you would like additional information, please contact us at the address below, call us at 1 800 541 6593, or log onto www.cdsanalytical.com.