

Rapid and Sensitive Analysis of Aluminum Ions using On-column Derivatization (Part 2) ... Application to the Analysis of Aluminum Ions in Total Parenteral Nutrition Solutions

The effects of trace amounts of aluminum in medications on long-term dialysis patients and total parenteral nutrition (TPN) patients have been the topic of discussion in recent years. The aluminum ions in TPN solutions can be analyzed by AA, ICP, ICP/MS, or HPLC method.

This Application News presents a method for the rapid and sensitive analysis of aluminum ions in TPN solutions by applying Shimadzu's unique on-column fluorescent derivatization HPLC using 8-hydroxyquinoline (8-HQ) that was featured in Application News No. L277.

■ What is On-column Derivatization?

On-column derivatization involves pre-addition of the reaction reagent to the mobile phase, such that the derivatization reaction occurs inside the instrument at the time the sample is injected.

As 8-HQ begins to fluoresce when it reacts with aluminum ions to form a chelate, the addition of 8-HQ to the mobile phase has no effect on the fluorescence detector background. Due to the rapid reaction rate between 8-HQ and the aluminum ions, the injected aluminum ions react to form aluminum/8-HQ chelate

before they reach the column.

As the reaction forming the chelate occurs inside the HPLC, the on-column derivatization method avoids the problems of contamination, errors, and human mistakes that can occur during pretreatment for pre-column derivatization. The on-column derivatization method offers easy, rapid, and highly sensitive analysis of aluminum ions in TPN solutions. (See Application News No. 277 for details of this reaction.)

■ Standard Analytical Conditions

Table 1 shows the optimal analytical conditions.

Fig. 2 shows the chromatograms superimposed for 0 μ g/L, 10 μ g/L, 25 μ g/L, and 50 μ g/L aluminum standard solutions. The FDA regulations prescribe a 25 μ g/L upper limit for aluminum content in LVP solutions. This method achieves highly sensitive analysis to a few μ g/L. The fluorescence of the aluminum/8-HQ chelate is observed to fluctuate with temperature. Consequently, this HPLC system incorporates a

fluorescence detector with a cell temperature-control function to enhance the accuracy of analysis.

Table 1 Analytical Conditions

Column	:Shodex Asahipak ODP-50 (150mmL. \times 4.6mmI.D.)
Mobile Phase	:A/B = 4/6 A:12mM 8-hydroxyquinoline acetonitrile solution B:100mM imidazole buffer (pH=7.1) adjusted by perchloric acid
Flow Rate	:0.6mL/min
Inj. Volume	:20 μ L
Temperature	:40 $^{\circ}$ C
Detection	:RF-10AXL Super at Ex=380nm, Em=520nm (Cell Temperature 25 $^{\circ}$ C)

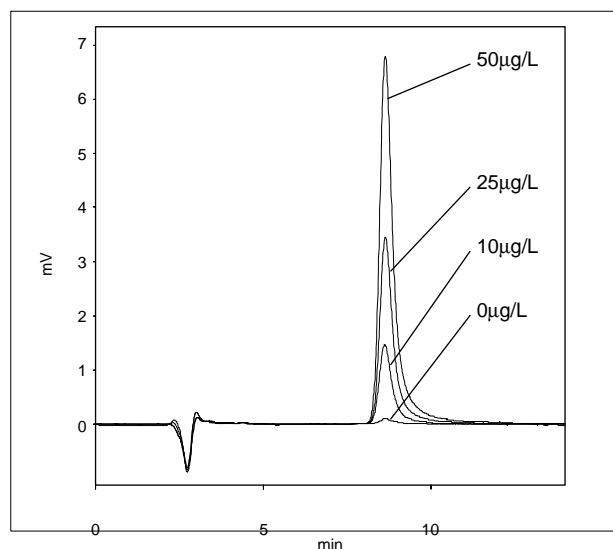


Fig. 1 Chromatograms of Aluminum Standard Solutions

Examples of TPN Solution Analysis

Figs. 2 to 5 show the analysis results for four types of TPN solution with different aluminum contents. Samples with high concentrations of active ingredients or with high viscosity are diluted 2x to 10x with the

mobile phase before injection. In these examples, the solutions were diluted with an equal volume of mobile phase and 20 μ L directly injected. Table 1 shows the analytical conditions.

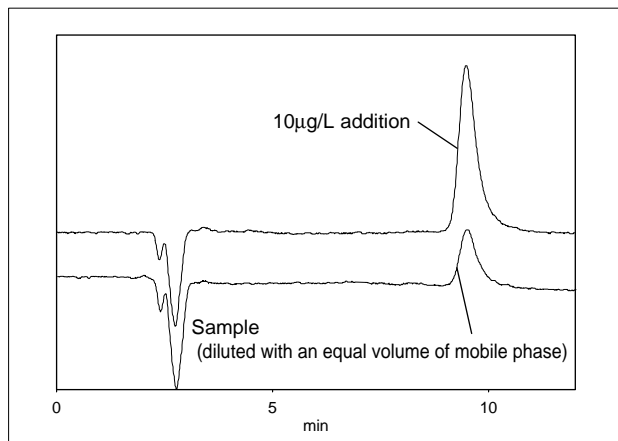


Fig. 2 TPN Solution A

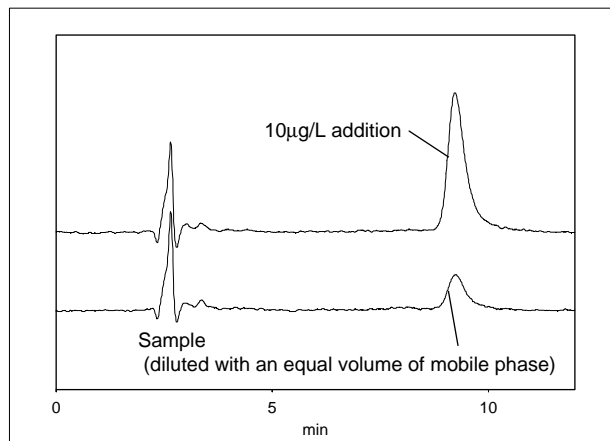


Fig. 3 TPN Solution B

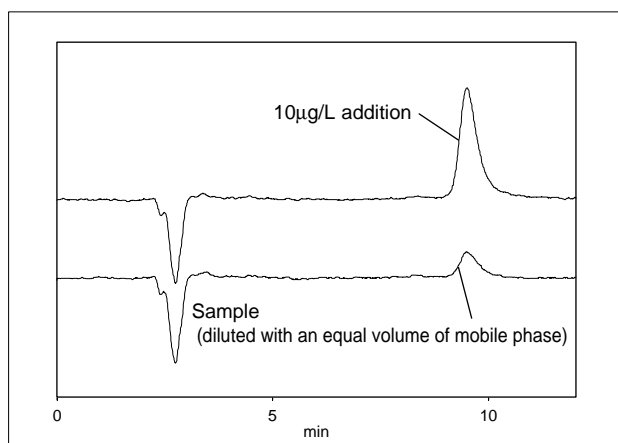


Fig. 4 TPN Solution C

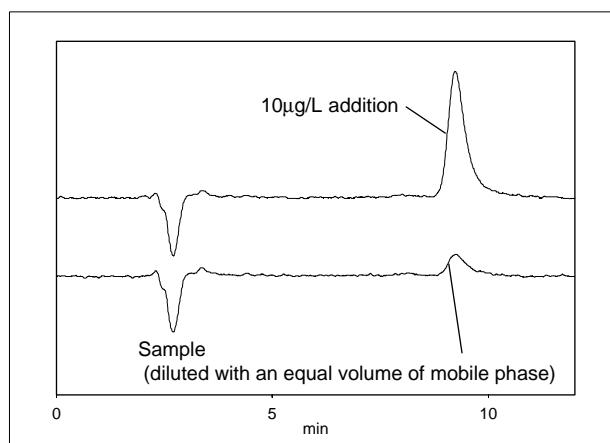


Fig. 5 TPN Solution D

Recovery

Table 3 shows the results of recovery tests on the four types of TPN solution after the addition of 10 μ g/L aluminum ions.

Table 3 Recovery

	TPN Solution A	TPN Solution B	TPN Solution C	TPN Solution D
TPN concentration (μ g/L)	6.8	2.4	2.6	3.8
10 mg/L addition (μ g/L)	18.2	13.4	13.8	14.3
Recovery (%)	114.5	110.6	112.6	105.5



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Printed in Japan 3100-06323-10A-1K