Waters
Lab Highlights
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ANALYSIS OF FAT- AND WATER-SOLUBLE VITAMINS IN PHARMACEUTICAL PREPARATIONS II: APPLICATION TO OVER-THE-COUNTER VITAMIN PRODUCTS

The capability of the QA-1™ Analyzer to simultaneously determine multiple vitamin constituents has been applied to several over-the-counter multivitamin, multimineral formulations. The data illustrates the potential for the instrument to accurately measure several vitamins over a wide concentration range in a single chromatographic run. Because of their speed and selectivity, these methods are ideal for quality control of both premixes and final products. The analyses will also be applicable in the formulation process itself where manufacturing efficiencies can be realized by monitoring raw material inputs.

Sample preparation procedures documented in the literature have been used in conjunction with the HPLC analyses. The number of tablets or volume of premix used has been scaled down to control the consumption of reagents. In many cases, larger samplings are taken in order to maximize statistical accuracy. The methodologies should be scaled according to the statistical needs of the user and to the composition of the particular matrix to be analyzed.

FAT-SOLUBLE VITAMINS

Sample Preparation[1]:

- Grind 3 tablets; transfer to 40ml capped tube
- Solubilize in 10ml DMSO/1% (w/v) Ammonium 1-Pyrrolidine Dithiocarbamate
- Heat 60 mins at 60°C with occasional shaking; cool
- Add 5ml hexane; Vortex
- Add 3ml 50% saturated NaCl; Vortex
- Shake 30 mins at ambient temperature
- Centrifuge to separate phases
- Remove 1ml aliquots of hexane layer
- Dry under N_2 stream
- Reconstitute with 1ml 2-Propanol

Analysis Conditions:

- Injection volume: 10 µl
- Column: Radial-PAK™ C_{18}, 5µm, 8mm X 10cm
- Mobile Phase: $CH_3CN/THF/H_2O$, (55:37:8)
- Flow Rate: 4.0ml/min
- Flow Volume: 40ml
- Wavelength: 280nm
- Attenuation: 2 ↑ 5

An example of the application of the method to a multivitamin tablet is shown in Figure 1.

Waters
Division of MILLIPORE 34 Maple St. / Milford, MA 01757 / 617-478-2000
Robert Burgoyne

WATER SOLUBLE VITAMINS

Sample Preparation[2]:

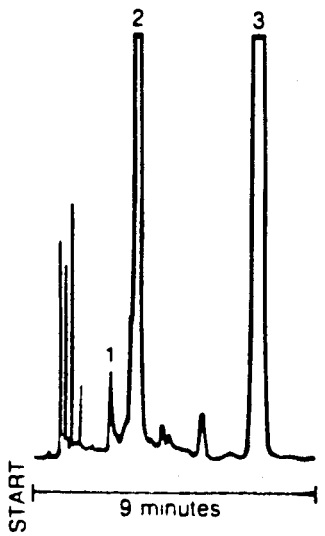
- Grind 1 tablet; transfer to 40ml capped tube
- Solubilize in 20ml DMSO/1% (w/v) Citric Acid
- Heat 45 mins at 60°C with occasional shaking; cool
- Centrifuge
- Inject supernate

Analysis Conditions:

- Injection Volume: 10 µl
- Column: Radial-PAK™ C_{18}, 5µm, 8mm X 10cm
- Mobile Phase: 25% CH_3OH
  75% H_2O containing 60% 0.01M PIC® Reagent B7
  and 40% RCSS Reagent D-4
- Flow Rate: 2.0ml/min
- Flow Volume: 40ml
- Wavelength: 254nm
- Attenuation: 2 ↑ 4

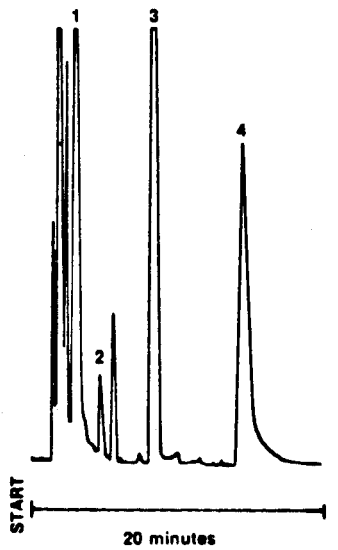
An example of the application of the method to a multivitamin tablet is shown in Figure 2.

FIGURE 1



1.	D_2	13	µg/TABLET
2.	E ACETATE	15.75	mg/TABLET
3.	A PALMITATE	1.09	mg/TABLET

FIGURE 2



1.	NIACINAMIDE	13.5	mg/TABLET
2.	B_6	1.4	mg/TABLET
3.	B_2	1.25	mg/TABLET
4.	B_1	1.2	mg/TABLET

1. R.P. Kwok, T. Hudson, and S. Subramanian, in "Application of High Pressure Liquid Chromatographic Methods for Determination of Fat-Soluble Vitamins, A, D, E and K in Foods and Pharmaceuticals", Association of Vitamin Chemists, Chicago, IL, 1978.
2. R.P. Kwok, W.P. Rose, R. Tabor and T.S. Pattison, J. Pharm. Sci. 70 (9), 1014 (1981).