Application Note

# Automated Online Dissolution with the ADS 100 and the Agilent 1260 HPLC-System

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## Introduction

Prednisone is commonly used in laboratories as a dissolution bath calibrator. Manual dissolution is a very time intensive process; because of that automation of this technique is highly desirable. In this study, the ADS 100 (Figure 1) is used as a tool to automate the dissolution sampling process at the appropriate intervals and initiating the analysis with HPLC. In addition to the reduction of the hands-on time involved in dissolution, ADS Control software provides the documentation of the automated dissolution process. The objective of this study is to determine the equivalency between manual and automated dissolution sampling and analysis using the prednisone calibrator tablet.

## Material and Methods

The study was run using USP prednisone tablets Lot P1I300 containing 10 mg prednisone. HPLC grade degassed water was used for the dissolution media according to the USP specifications. The samples were taken 30 minutes after the start of the dissolution experiment manually with glass pipettes and automatically. The samples automatically taken by the ADS 100 were filtered under pressure. Both the manual and automated samples were analyzed according to the procedure contained in the USP method manual on the same UV-spectrophotometer (HPLC-DAD). The devices used in this application note are described in Table 1. The samples were analyzed using a restriction capillary (see Table 1) at 40°C in a thermostated column compartment.

Figure 1: AnaTox ADS 100

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Table 1: Devices

| Device | Description |
|---|---|
| Dissolution-Bath | SR8 PLUS Dissolution Test Station, Hanson Research, USA |
| Dissolution-Sampler | ADS 100, AnaTox, Germany |
| Degasser | 1260 µ-Degasser, Agilent Technologies, Germany |
| Pump | 1260 Binary Pump, Agilent Technologies, Germany |
| Injector | 1260 WellPlate-ALS, Agilent Technologies, Germany |
| Colum oven | 1290 Infinity TCC, Agilent Technologies, Germany |
| Detector | 1260 DAD, Agilent Technologies |
| Vacuum pump | Alcatel, Germany |
| Restriction capillary | 2m * 0,12mm stainless steel capillary |

The flow rate of the binary HPLC pump was set isocratic (100% water) to 1ml/min, the detection wavelength of the DAD to 242nm (absorption maximum of prednisone). The injection of the samples (50µl) was carried out by using the same HPLC autosampler already used for sample collecting (1260 wellplate sampler). The ADS 100 configuration consists of 6 circulating pumps, one flushing pump, a 8-position valve and a flow sensor. The ADS 100 settings for transferring samples are described in Table 2. The samples (250µl) from the ADS 100 were collected in a 96-position wellplate and analyzed by HPLC after collection of all samples (asynchronous).

Table 2: ADS 100 Settings

| Setting | Description |
|---|---|
| Flow rate | 5 ml/min |
| Sampling volume | 250 µl |
| Analysis | asynchron |

## Results

At the 30 minute sampling event, the mean results of the samples taken with ADS 100 and of the manually taken samples for 6 vessels are essentially identical. The dissolved prednisone concentration was in the USP range (see Figure 2).

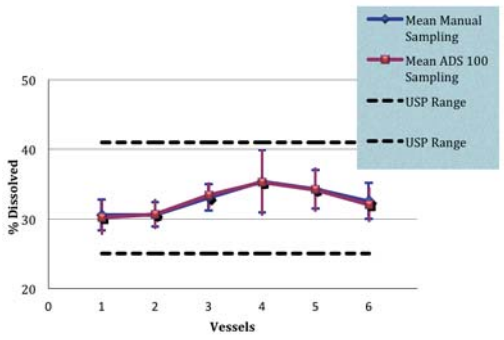


Figure 2: Dissolution rates depending on manual or automatic taken samples

## Conclusion

Equivalency between manual and automated dissolution sampling with the ADS100 and analysis by HPLC using the prednisone calibrator tablets has been demonstrated.

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