Analysis of rosuvastatin stress degradation behavior using liquid chromatography coupled to ultraviolet detection and electrospray ionization mass spectrometry

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Supplementary data

1. Method optimization

Several modifications in the mobile phase composition were performed to achieve the best separation, detection and method performance. These modifications involved the change of the type and ratio of the organic modifier, the use of formic acid or ammonium acetate buffer with the organic modifier and changing the pH of ammonium acetate buffer (Table 1S).

Table 1S

Effect of experimental parameters on ROS chromatographic performance parameters.

Parameters	K'[a]	T[a]	N[a]
1) *Type of organic modifier*			
• 40 % acetonitrile: 60 % (0.1 % formic acid)	3.60	1.02	11450
• 60 % methanol: 40 % (0.1 % formic acid)	3.80	1.05	6590
2) *Ratio of organic modifier*			
• 50 % acetonitrile	1.05	1.06	5100
• 40 % acetonitrile	3.60	1.02	11080
• 35 % acetonitrile	7.82	1.01	12090
• 30 % acetonitrile	19.30	1.01	11970

3) Aqueous portion of the mobile phase			
• 60 % (0.1 % formic acid)	3.61	1.02	11080
• 60 % Ammonium acetate buffer at pH 4.8	2.46	1.11	8590
• 60 % Ammonium acetate buffer at pH 3.8	3.20	1.03	10040
• 60 % Ammonium acetate buffer at pH 5.8	0.46	1.28	3470

[a] K' : Capacity factor, T : USP tailing factor, N: USP plate count

2. Limit of detection and limit of quantitation

LOD and LOQ were determined experimentally according to the ICH guidelines. The LOD was taken as the lowest concentration of the analyte which can be reliably detected but not necessarily quantified (signal-to-noise ratio is 3:1) whereas LOQ was taken as the lowest concentration of the analyte that can be quantitatively determined with suitable precision and accuracy (signal-to-noise ratio is 10:1)(Figure 1S and 2S).

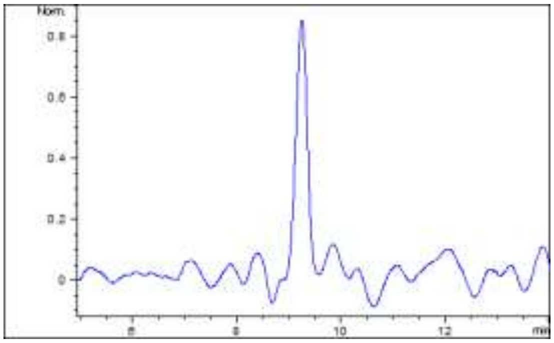

Figure 1S. Representative chromatogram for LOD using UV detector.

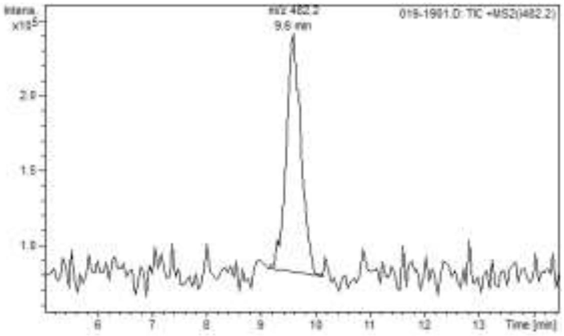

Figure 2S. Representative chromatogram for LOD using ESI-MS.

3. Degradation kinetics of ROS

The Degradation kinetics was studied using ROS sample subjected to UV irradiation. It was apparent that the peak area of entire ROS decreased over time with increase of peak areas of all DPs specially DEG-7 and DEG-8. The degradation was found to follow the first order kinetics (figure 3S-45).

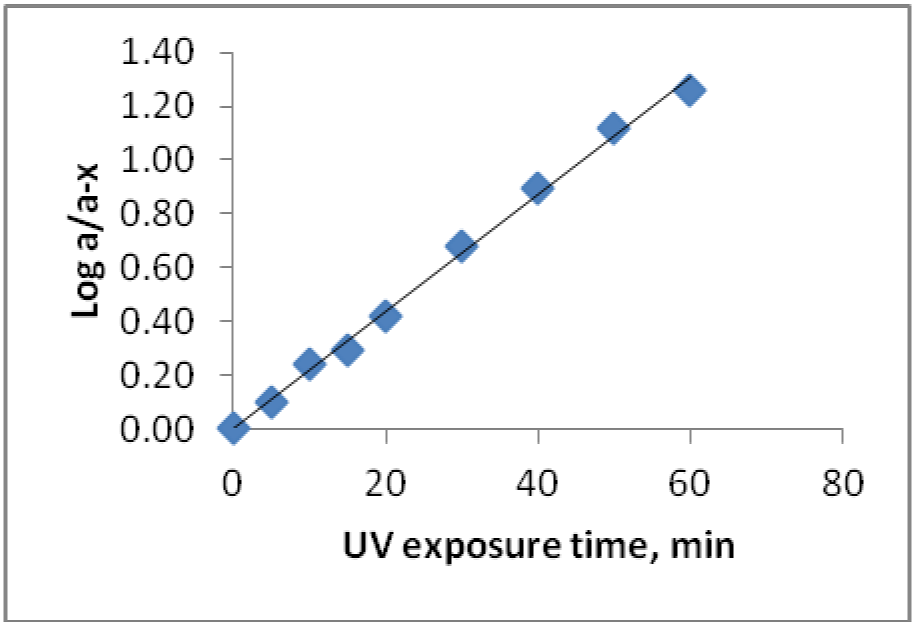

Figure 3S. Semilogarithmic plot for ROS degradation kinetics.

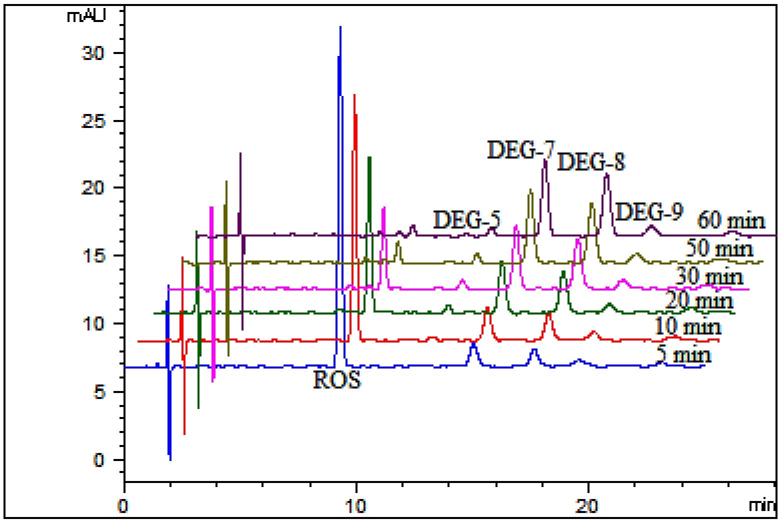

Figure 4S. Overlaid chromatograms of photo degraded ROS after different UV exposure time.