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Application of normal fluorescence and stability-indicating derivative synchronous fluorescence spectroscopy for the determination of gliquidone in presence of its fluorescent alkaline degradation product

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#### Abstract

Simple, smart and sensitive normal fluorescence and stability-indicating derivative synchronous spectrofluorimetric methods have been developed and validated for the determination of gliquidone in the drug substance and drug product. Normal spectrofluorimetric method of gliquidone was established in methanol at  $\lambda$  excitation 225 nm and  $\lambda$  emission 400 nm in concentration range 0.2-3  $\mu\text{g/ml}$  with LOD equal 0.028. The fluorescence quantum yield of gliquidone was calculated using quinine sulfate as a reference and found to be 0.542. Stability-indicating first and third derivative synchronous fluorescence spectroscopy were successfully utilized to overcome the overlapped spectra in normal fluorescence of gliquidone and its alkaline degradation product. Derivative synchronous methods are based on using the synchronous fluorescence of gliquidone and its degradation product in methanol at  $\Delta \lambda$  50 nm. Peak amplitude in the first derivative of synchronous fluorescence spectra was measured at 309 nm where degradation product showed zero-crossing without interference. The peak amplitudes in the third derivative of synchronous fluorescence spectra, peak to trough were measured at 316,329 nm where degradation product showed zero-crossing. The different experimental parameters affecting the normal and synchronous fluorescence intensity of gliquidone were studied and optimized. Moreover, the cited methods have been validated as per ICH guidelines. The peak amplitude-concentration plots of the derivative synchronous fluorescence were linear over the concentration range 0.05-2  $\mu\text{g/ml}$  for gliquidone. Limits of detection were 0.020 and 0.022 in

Abbreviations: NF, normal fluorescence; SFS, synchronous fluorescence spectroscopy; DSFS, derivative synchronous fluorescence spectroscopy; 1DSFS, first derivative synchronous fluorescence spectroscopy; 3DSFS, third derivative synchronous fluorescence spectroscopy.

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