

利用拉曼光谱定量乙醇中的尿素

Urea is widely employed as a nitrogen-release fertilizer with more than 90% of urea production destined for agricultural applications[1]. Urea is also known to form complexes with fatty acids[2], which have been employed for separation of complex mixtures and purification

processes[3]. In this application note, we present the quantification of the concentration of urea in ethanol by Raman Spectroscopy and show how this method can be employed for determining the percentage of urea in a solid inclusion compound with stearic acid[4].

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EXPERIMENTAL

Chemicals: Urea (Aldrich, >99%); Stearic acid (SA) (Aldrich, >95%); Ethanol (Soria)

Instrument: [i-Raman® Plus 785S](#)

Stock solutions of urea (0.0420 gr/gr ethanol) and SA (0.04126 gr/gr ethanol) were prepared. Standard solutions were prepared by mixing these stock solutions in different proportions in order to obtain urea concentrations between 0 and 0.042 gr/gr ethanol and keeping the total mass concentration (urea + SA) approximately

constant. Raman spectra of 0.5 mL of each solution in aluminum containers were measured employing a 785 nm laser (power: 30%, ~ 90 mW) and 5000 msec of acquisition time (20 repetitions). No effects of heating or evaporation were observed. Spectra of the standard solutions that were background corrected employing the [BWSpec®](#) software are shown in **Figure 1**.

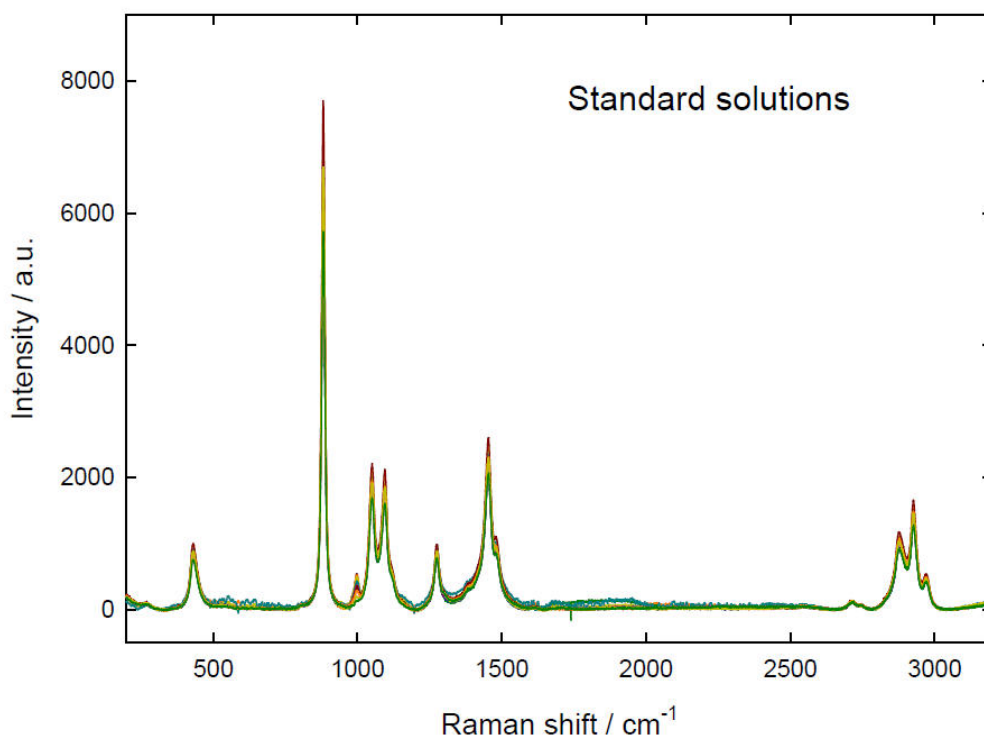


Figure 1. Dark-subtracted baseline-corrected Raman spectra of the standard solutions of urea and SA in ethanol.

RESULTS

Spectra were normalized by employing the intensity of the ethanol band at $1049\text{--}1050\text{ cm}^{-1}$. Normalized spectra (**Figure 2**) clearly show that the only appreciable change due to the increment in relative urea concentration occurs at the band at $996\text{--}997\text{ cm}^{-1}$, which is attributed to urea. This band corresponds to the symmetric C-N stretching[5] that is experimentally and theoretically reported at about 1010 cm^{-1} for the solid urea,[6] but shifts to lower wavenumbers in solution.[5,7]

For quantification purposes, spectra were deconvoluted, fitting the experimental results in the region $950\text{--}1200\text{ cm}^{-1}$ by 4 Lorentzian functions. These curve fitting results are shown in **Figure 3** for some of the standard solutions.

The ratio of the intensities of the fitted peaks assigned to urea at 996 cm^{-1} (peak 1, a1) and ethanol at 1049 cm^{-1} (peak 2, a2) was employed

as the analytical parameters. The dependence of this ratio on the urea concentration of the samples is presented in **Figure 4**. The calibration curve plotted in this figure shows a good linear behavior which indicates this parameter could be employed for urea quantification.

For the determination of the urea content of real samples containing both urea and SA, the solid sample was dissolved in ethanol (0.04299 gr/gr ethanol) and Raman spectra were recorded in the same conditions. From the values of the ratio $a1/a2$ for this sample, obtained by fitting of the peaks at 996 and 1049 cm^{-1} (**Figure 5**), the urea concentration of the solution was determined to be $0.03274\text{ gr urea /gr ethanol}$. Thus, the urea content of the sample resulted to be $76\text{ \% w/w}$. This value is consistent with other reported values for the inclusion compounds formed by stearic acid and urea (ca. $75\text{ \%}$).[2,4]

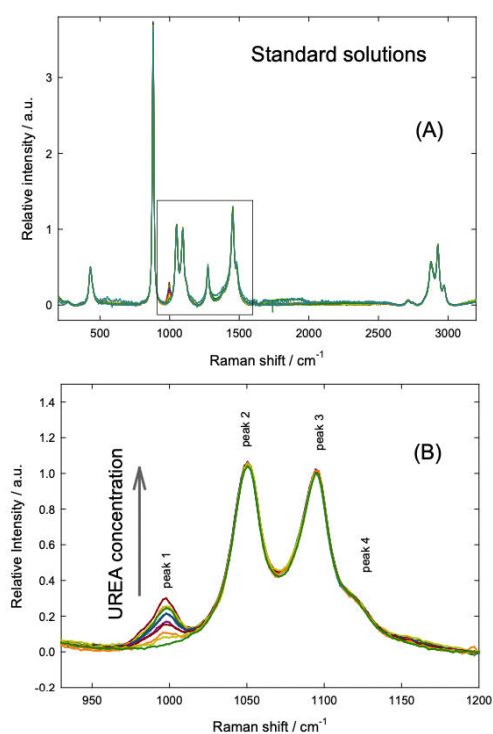


Fig.2: Normalized spectra of the standard solutions of urea + SA in ethanol. (A) Complete spectra (B) Analyzed region.

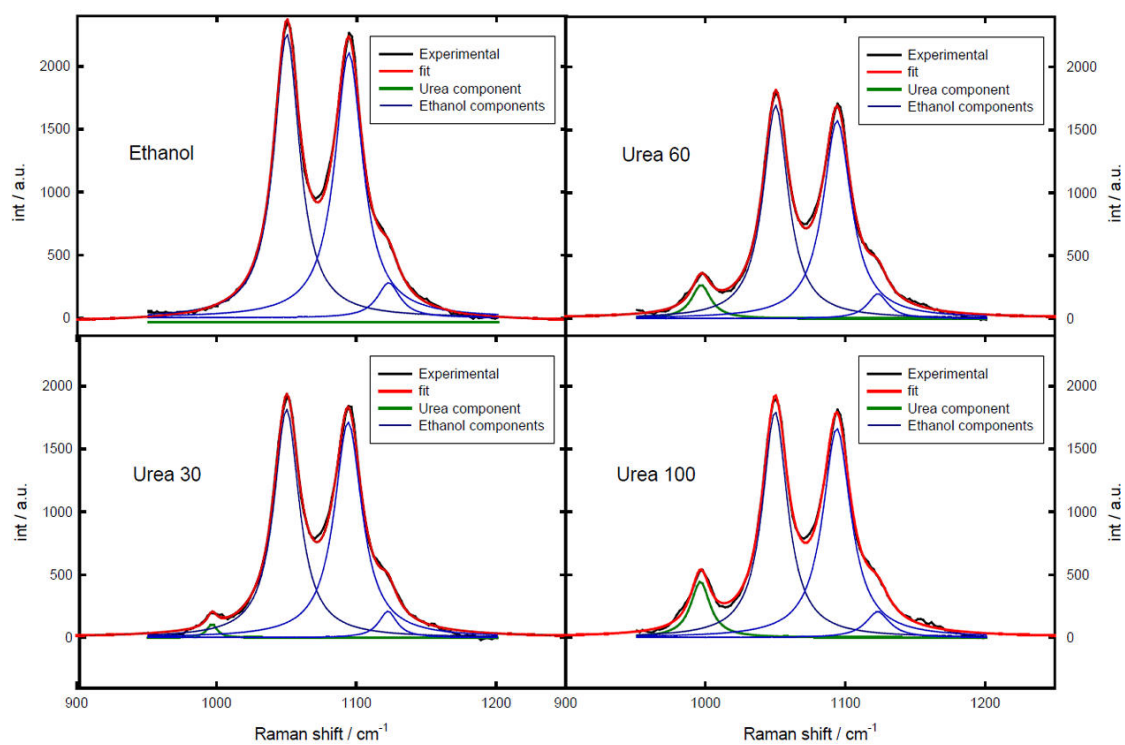


Figure 3. Fitting of the Raman spectra in the region 950-1200 cm⁻¹. Urea 30, Urea 60 and Urea 100 mean solutions 0.0123, 0.0248 and 0.0413 gr urea/gr ethanol respectively.

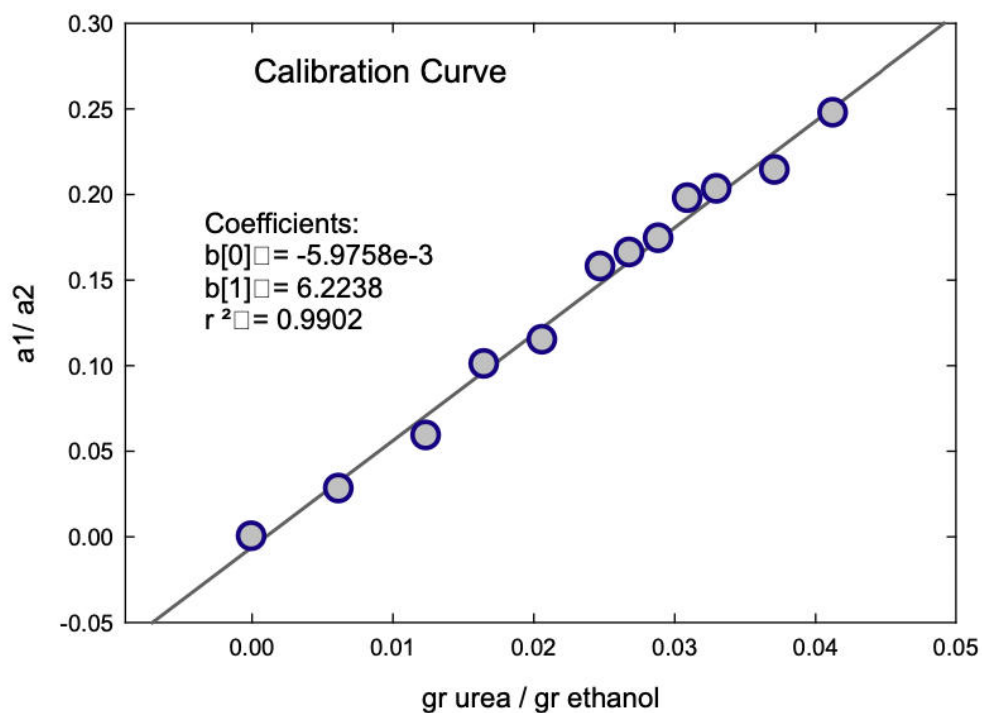


Figure 4. Calibration curve for the urea quantification in ethanol. Ratio of the intensities of the fitted bands of urea (a1) and ethanol (a2) as a function of the urea content of the standard solutions.

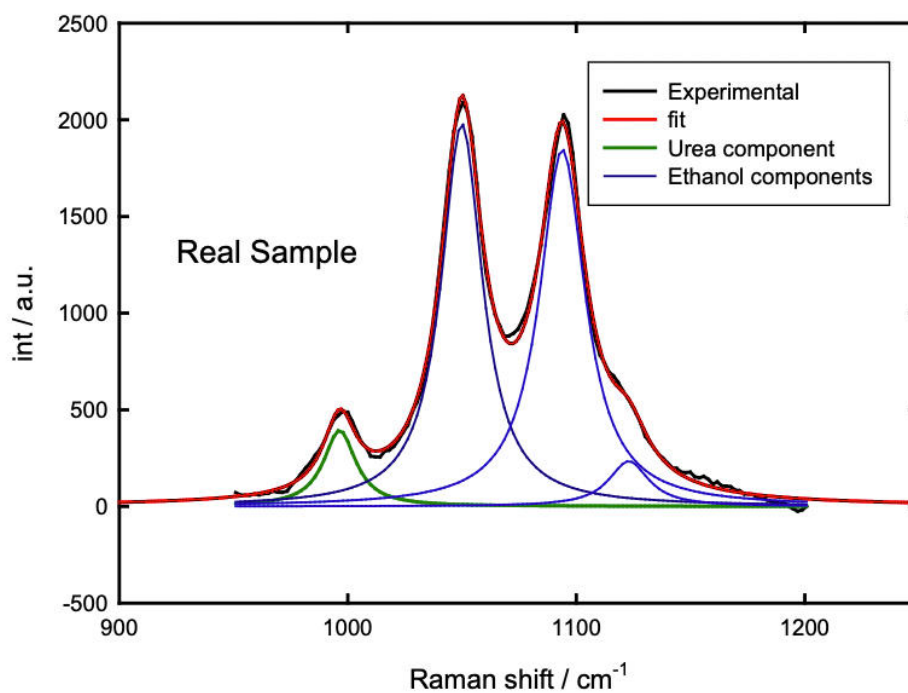


Figure 5. Fitting of the sample spectrum.

CONCLUSION

We presented a simple method for quantification of urea concentration in ethanolic solutions by Raman spectroscopy. The calibration curve presents good linearity in the concentration range analyzed (up to 0.042 gr urea / gr ethanol). The presence of stearic acid in

the samples does not modify appreciably the Raman spectra (at least up to 0.042 gr / gr ethanol), so this method allows the quantification of urea in solid binary samples containing both urea and stearic acid.

FURTHER INFORMATION

Related application notes

[Choosing the Most Suitable Laser Wavelength](#)

[Quantification of methanol in contaminated spirits with Raman](#)

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CONFIGURATION



i-Raman Plus 785S

该 i-Raman[®] Plus 785S 是我们屡获殊荣的 i-Raman 便携式拉曼光谱仪系列的一部分,其采用我们创新的智能光谱仪技术。这款便携式拉曼光谱仪使用了具有高量子效率、TE 冷却功能和高动态范围的 CCD 阵列检测器,即使集成时间长达 30 分钟,也能提供出色的低噪声性能。因此,还可以测量较弱的拉曼信号。

i-Raman Plus 785S 具有宽光谱范围和高分辨率的特点,其配置允许在 65 cm^{-1} 至 3350 cm^{-1} 之间进行测量。该系统基础面积小,结构形式轻巧并且能耗低,故此可随时随地进行研究级别的拉曼分析。该 i-Raman Plus 配备有便于采样的光纤探头,并可以与一个比色皿支架、一个视频显微镜、一个带探头支架的 XYZ 平移台、我们公司内部的 BWIQ[®] 多变量分析软件和鉴定软件 BWID[®] 搭配使用。有了 i-Raman Plus,您始终可以使用高精度拉曼解决方案进行定性和定量分析。