Application Note 410000016-A

利用拉曼光定量乙醇中的尿素

Urea in 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].

Find more information in the video:

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.

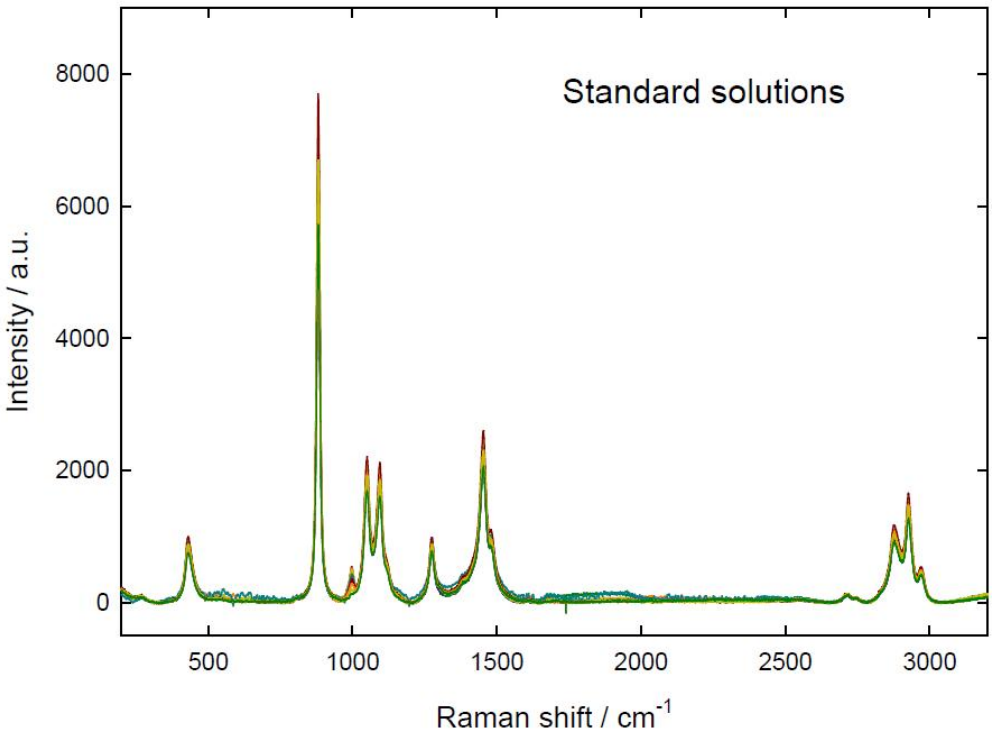

Figure 1. Dark-subtracted baseline-corrected Raman spectra of the standard solutions of urea and SA in ethanol.

RESULTS

Spectra were normalized by employing the intensity of the ethanol band at 1049-1050 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 996-997 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 950-1200 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 gr urea /gr ethanol. Thus, the urea content of the sample resulted to be 76 % w/w. This value is consistent with other reported values for the inclusion compounds formed by stearic acid and urea (ca. 75 %).[2,4]

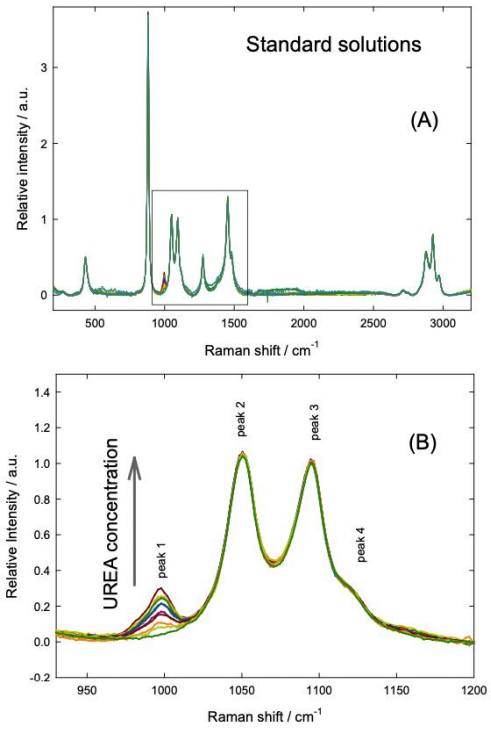

Fig.2: Normalized spectra of the standard solutions of urea + SA in ethanol. (A) Complete spectra (B) Analyzed region.

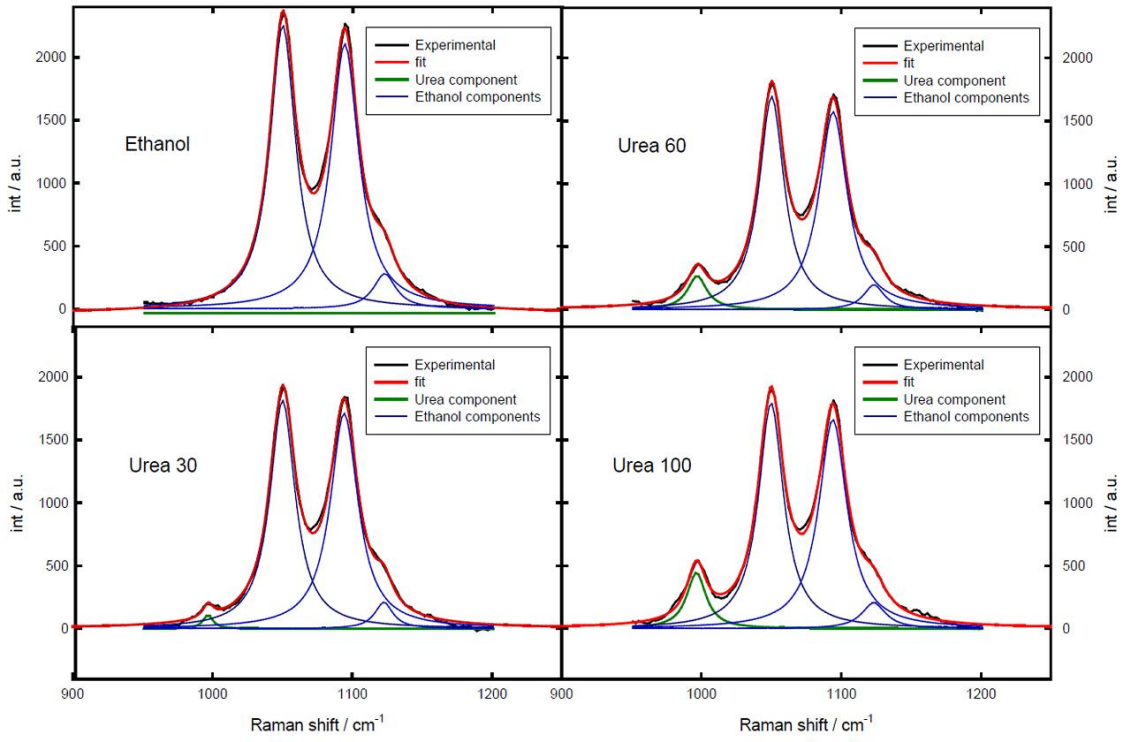

Figure 3. Fitting of the Raman spectra in the region 950-1200 cm-1. Urea 30, Urea 60 and Urea 100 mean solutions 0.0123, 0.0248 and 0.0413 gr urea/gr ethanol respectively.

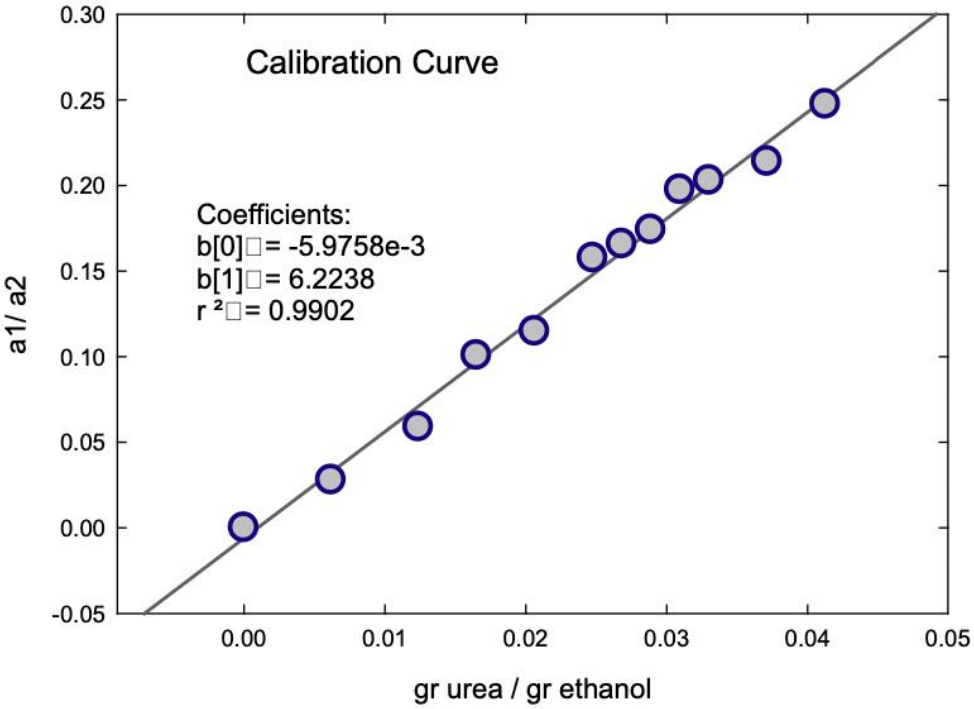

Figure 4. Calibration curve for the urea quantification in ethanol. Ratio of the intensities of the fitted bands of urea (a1) and ethanol (a2) as a function of the urea content of the standard solutions.

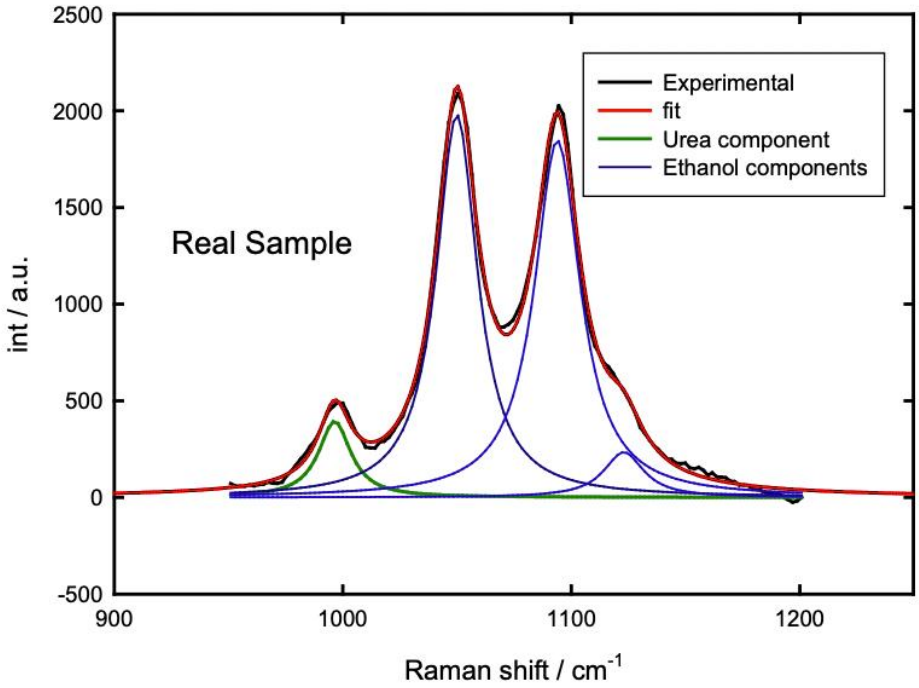

Figure 5. Fitting of the sample spectrum.

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

ACKNOWLEDGEMENTS

We would like to thank Dr. Waldemar A. Marmisollé of the Soft Matter Laboratory INIFTA – Universidad Nacional de La Plata (UNLP), Buenos Aires, Argentina – CONICET for sharing these research results.

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CONFIGURATION

i-Raman Plus 785S

i-Raman® Plus 785S 是我屡殊的 i-Raman 便携式拉曼光系列的一部分,其采用我新的智能光技。款便携式拉曼光使用了具有高量子效率、TE 冷却功能和高范的 CCD 列器,即使集成 30 分,也能提供出色的低噪声性能。因此,可以量弱的拉曼信号。

i-Raman Plus 785S 具有光范和高分辨率的独特合,其配置允在 65 cm^{-1} 至 3350 cm^{-1} 之行量。 系基面小,形式巧并且能耗低,故此可随随地行研究的拉曼分析。 i-Raman Plus 配有便于采的光探,并可以与一个比色皿支架、一个微、一个探支架的 XYZ 平移台、我公司内部的 BWIQ® 多量分析件和定件 BWID® 搭配使用。有了 i-Raman Plus,始可以使用高精度拉曼解决方案行定性和定量分析。

– 9.5 mm

使用 BCR100A 拉曼比色皿支架,便可通将拉曼探固定到支架上松量液体和粉末的拉曼光。附件采用三点式精密塞盖的内部子,具有无与比的重性,并且拉曼信号的放大倍数因此是比色皿的三倍。在其行的候,保了探不直接与比色皿接触,并包含了一个光捕器,以少背景光。BCR100A 用于直径 9.5 mm 或 12 mm 的探的型号格,并可与每一款 12.5 mm x 12.5 mm 外直径(1 cm 路径度)的准比色皿搭配使用,用于液体或粉末的取。