

Spectroscopic Studies of Iso-Saccharinic Acid and Determination of Calcium by X-Ray Fluorescence Spectroscopy

G. Getahun¹, P. Warwick¹, S. Zauner², N. Trautmann²

¹ Dep. of Nuclear Chemistry, Loughborough University

² Institut für Kernchemie, Universität Mainz

Low- and intermediate-level radioactive wastes contain a large amount of cellulose. The main degradation product of cellulose in underground repositories is iso-saccharinic acid (ISA). The ISA is a lactone in acidic medium and a straight chain molecule in basic medium [1].

According to this a strong base like KOH opens the ring of the lactone rapidly. The main aim was to study the ring opening characteristic of the iso-saccharinic acid.

For that about 0.1 g of the iso-saccharinic acid was dissolved in water, four samples were taken and their pH was adjusted in the pH range from 2-10 using KOH solution. UV spectroscopy was applied to study the ring opening of the lactone molecule. The iso-saccharinic acid lactone ring has a carbonyl functional group (C=O) at low pH [2]. As soon as the pH is raised to the alkaline region (pH=8 and above) the ring breaks and becomes an open chain due to the formation of the carboxylate group (COOH) as it is shown in Fig. 1. These results were obtained with UV absorption spectroscopy (Fig. 2). As alternative methods optical rotation and NIMR can be applied.

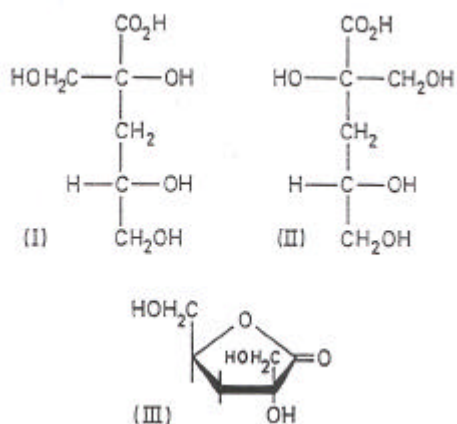


Fig.1: Structure of iso-saccharinic acid at pH values = 8 (I and II) and at pH values below 8 (III)

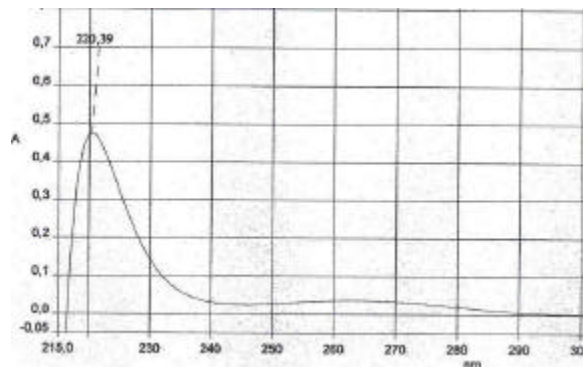


Fig. 2: UV absorption spectrum of ISA at pH=9

Furthermore, a determination of calcium in the ISA was performed by means of X-ray fluorescence spectroscopy. As can be seen from Fig. 3 samples which were treated with oxalic acid and run through a DOWEX 50X8 column several times are free of calcium (samples 5-10). Whereas in the other samples with minor treatment calcium can still be observed.

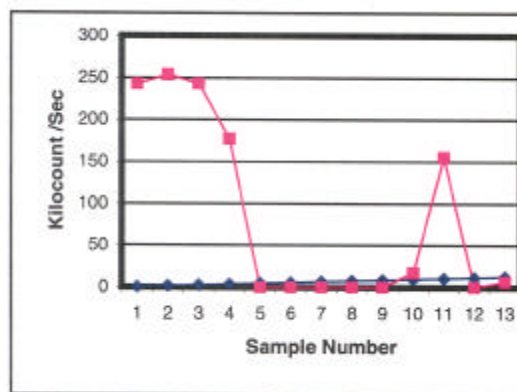


Fig.3: X-ray fluorescence measurements to determine calcium in ISA samples

REFERENCES:

- [1] Greenfield, B.F.; Hurdus, M.H.,
Mat. Res. Soc. Symp. Proc; Vol.333 (1994)
- [2] Robertson, G. P.; Somers, P. J.; Harrison, N.,
Radioactive Waste Disposal Report (1993)