TXRF

Ultratrace Element Analysis of Nanoparticles

Application Note # XRF 438

Introduction

Modern materials, like nanoparticles, are largely established for the production of industrial and consumer products. Ongoing product development accompanied by increasing concerns about the impact on human health and the environment lead to a growing demand for versatile analytical technologies. This report describes the use of **T**otal reflection **X**-**R**ay **F**luorescence (TXRF) analysis for nanoparticles in different areas.

Instrumentation

All measurements were performed using the benchtop TXRF spectrometer S2 PICOFOX. This instrument is equipped with an air-cooled low power X-ray tube (Mo target), a multilayer monochromator with 80% reflectivity and the liquid nitrogen-free XFlash® Silicon Drift Detector (SDD) with an energy resolution of < 150 eV (Mn Kα).

Characterization of nanoparticles

TXRF analysis requires extremely small sample amounts and is therefore most suitable for R&D projects. For semiquantitative analysis, e.g. the determination of element ratios, TXRF is non-destructive and allows almost full recovery of the sample.

The following example describes the analysis of element ratios in CdSe nanoparticles coated with ZnS. The sample preparation was quite simple: with the use of a cotton bud, a few particles were transferred to a quartz sample carrier and TXRF measurement was then applied. Figure 1 shows the results for three samples with comparable compositions and good concordance to the target values.

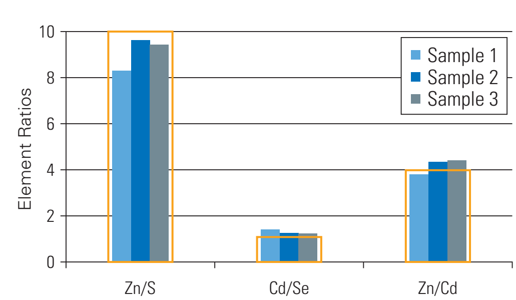

Figure 1
Element ratios of coated nanoparticles. The orange frame (□) shows the target values

Detection limits of nanoparticles in solution

Monitoring of nanoparticles released to the environment requires the most sensitive analytical technologies for accurate detection of these particles. The following example describes the detection of TiO_2 nanoparticles after dilution.

10 µl Ga standard solution (100 mg/l) was added to 1 ml of a nanoparticle solution containing approximately 1 mg/l TiO_2. Different dilution steps up to a factor of 100 were applied. 10 µl of each solution were transferred to a quartz carrier and measured for 1000 s.

Table 1 shows the concentrations of TiO_2 in all solutions. Under these conditions, the detection limit for the nanoparticles was calculated to 3.7 µg/l.

Dilution factor	Concentration (µg/l)
0	852
2	331
5	159
100	8.4
LOD	**3.7**

Table 1
Measurement of the concentration of TiO_2 nanoparticles after dilution

Analysis of nanoparticle coating

In a subsequent experiment, the amount of a Ru coating of TiO_2 nanoparticles was determined.

About 40 mg (exact weight required) were resuspended in 2.5 ml Triton X-100. After addition of 10 µl internal Ga standard (1 g/l) and careful homogenization 10 µl suspension were transferred to a quartz carrier and measured for 120 s.

Figure 2 shows a spectrum of this sample measurement. The amount of Ru was calculated to 72 µg/l, which was in accordance with ICP results (65 µg/l).

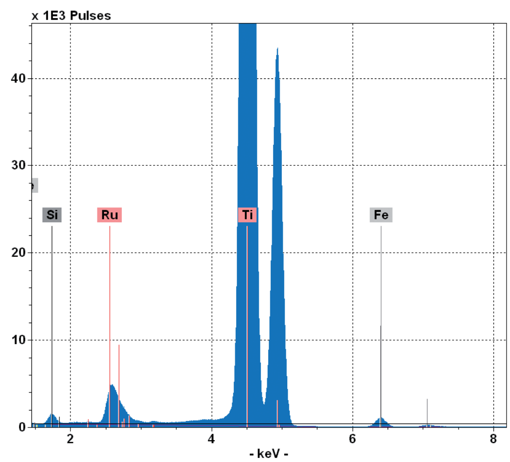

Figure 2
Spectrum of TiO_2 nanoparticles coated with Ru

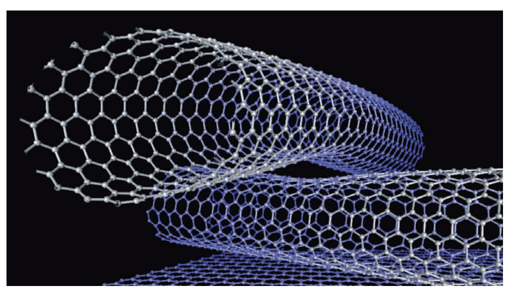

Figure 3
Nanotube

Conclusion

The examples in this report clearly demonstrate the suitability of TXRF analysis for ultra-trace element analysis of nanoparticles. Due to its versatility, TXRF can be applied to R&D, quality control or environmental monitoring.

Acknowledgement
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