TXRF

Why Wait for Sample Prep! Shortest Time-to-Result through TXRF

Application Note # XRF 435

Introduction

The accurate chemical analysis of any sample requires a time-consuming procedure with a number of steps depending on the type of sample:

- Sampling, filtration, grinding, homogenization, chemical stabilization
- Digestion and/or extraction with hazardous chemicals, dilution or enrichment, pellet pressing, melting
- Instrument setup, calibration of the analytical instrument
- Measurement, data aquisition
- Data evaluation, reporting, data export, archiving

Each step raises costs for materials and labour, increases the risk of analytical errors and contaminations and requires operator training and instrument maintenance.

Simplify your sample preparation with TXRF

Total Reflection X-ray Fluorescence (TXRF) analysis is a versatile analytical method, which is suitable for the multielement analysis of different kinds of sample types. For most analytical tasks sample digestion can be avoided and sample preparation can be reduced to a few simple steps. The operation of the instrument does not require any consumables or replacement parts. Due to a factory-built one time calibration the spectrum evaluation and quantification is fast and simple.

This report explains the easy procedures required for the preparation of different sample types. Furthermore, it highlights the short time-to-result achieved by the TXRF method.

Why Wait for Sample Prep!

Just a few steps are required for the sample preparation prior to an elemental analysis with TXRF. The preparation procedures for the most common sample types are described here in detail. A variety of samples can be applied directly or after a simple dilution step:

- **Particles:**
  Nanoparticles, contaminations, proteins, gunshot residues.

In addition, procedures for the preparation of samples like filters, wafer pieces, thin films, aerosols etc. are available on request.

- **Liquids:**
  tap water, freshwater, beverages, urine and other body fluids, organic solvents.

- **Suspensions and matrix rich liquids:**
  sewage, sea water, whole blood, blood serum, tissue homogenates, dyes.

Solid samples can be quantitatively analyzed after grinding and resuspending. Time-consuming digestion with hazardous chemicals can be avoided.

- **Solids, powders:**
  soils, sediments, tablets, polymers, food, lubricants, catalyzer, glass splinter, ashes.

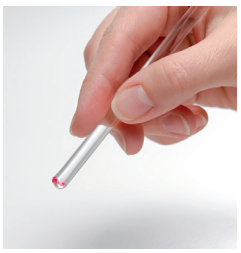
Direct application of particles allows a standardless analysis of the relative element composition.

Your benefits

- Direct analysis of liquids, suspensions and particles
- Precalibrated instrument – simple quantification by internal standardization
- Save method - almost no hazardous chemicals needed
- Low cost of operation - no media or consumables required

Particles

1. Dab vacuum grease on carrier



2. Pick-up some particles with (glass) rod

3. Pipette 5 to 20 µl on carrier

Liquid samples

1. Micro reaction tube with liquid sample

2. Add internal standard

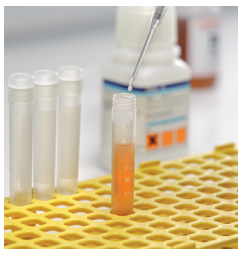
3. Homogenize carefully

4. Pipette 5 to 20 µl on carrier

Suspensions

1. Tube with raw suspension

2. Dilute sample with distilled water



3. Add internal standard

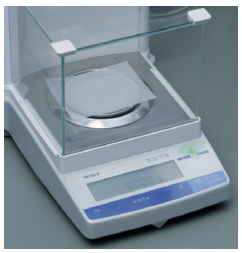
4. Homogenize carefully

5. Pipette 5 to 20 µl on carrier

Solid samples

1. Fill powder in an agate mortar or micro mill

2. Grind or mill carefully (e.g. Retsch MM400)



3. Weigh about 20 to 50 mg, note exact amount

4. Transfer quantitatively to a tube

5. Suspend in 1 to 2 ml detergent solution

6. Add standard

7. Homogenize carefully

8. Pipette 5 to 20 µl on carrier

Dry 10 min

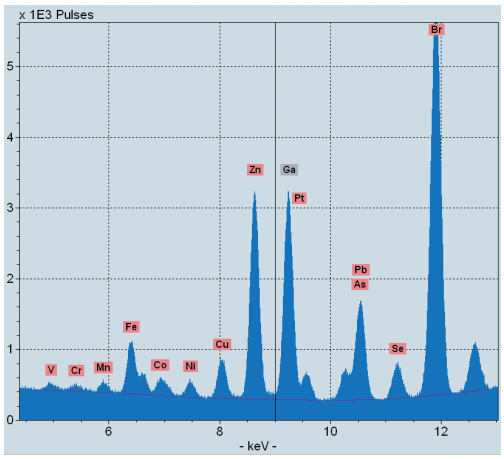
Drying process

1. Dry through heat or vacuum

2. Load the instrument

Measure 5 – 10 min

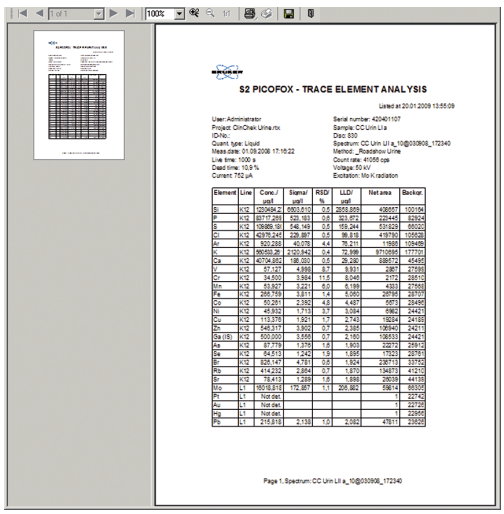
Results



TXRF spectrum

No.	Element	Net	Conc./(µg/l)	LLD/(µg/l)
1	Si	415518		
2	P	212872	80261,960	353,861
3	S	541046	112482,536	200,463
4	Cl	419241	43192,340	100,655
5	Ar	17615		
6	K	9846937	572004,830	51,945
7	Ca	861918	39689,771	35,851
8	V	3207	64,303	10,016
9	Cr	3173	50,723	7,985
10	Mn	6509	81,518	6,223
11	Fe	29299	293,543	4,923
12	Co	7977	71,120	4,332
13	Ni	6792	44,969	3,115
14	Cu	19170	113,420	2,764
15	Zn	106723	548,666	2,407
16	Ga	107849	500,000	2,179
17	Ga	20783		
18	As	12333	48,916	1,915
19	As	5599		

Table with quantitative results



Create PDF file or print report

TXRF Statistics

	Mean CC Urin LII a_10@030908_172340 CC Urin LII b_11@030908_172714	CC Urin LII a_10@030908_172340	CC Urin LII b_11@
	µg/l	µg/l	µg/
Si	1238676,218	1230484,237	
P	87275,633	83717,269	
S	109753,865	109869,186	
Cl	42437,420	42976,245	
Ar	745,148	920,288	
K	610361,966	560533,261	
Ca	39615,410	40704,862	
V	58,822	57,127	
Cr	37,032	34,500	
Mn	45,267	53,927	
Fe	281,130	266,759	
Co	61,612	50,261	
Ni	43,069	45,932	
Cu	110,832	113,376	
Zn	537,747	546,317	
Ga		500,000	
As	85,860	87,779	
Se	62,553	64,513	
Br	784,170	826,147	
Rb	431,168	414,232	
Sr	72,483	78,413	

Export data for archiving, LIMS etc.

Shorten your Time-to-Result

Effective quality and process control requires the shortest time-to-result possible. This is the time needed from sampling to the final quantitative result. Any advantage results in:

- Higher sample throughput
- Stable industrial processes due to immediate feedback
- Constant high product quality

The sample preparation steps required for most common sample types prior to a TXRF analysis were shown on the previous pages.

Avoiding several preparation steps will shorten your time-to-result significantly as shown on the chart below.

- Digestion is not required for most samples types.
- The instrument is calibrated ex works, which will save up to 30% of your daily work time.
- The one-point quantification procedure with an internal standard gives accurate results automatically - effort for trainings and lab standardization will be minimized.

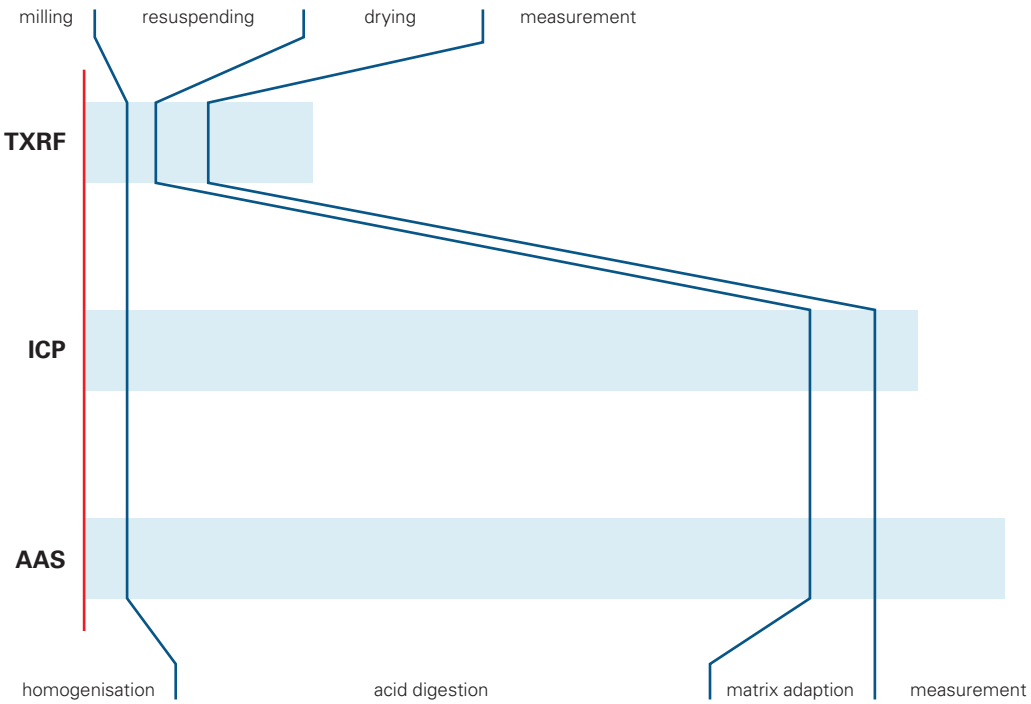


Figure 1
Comparison of the total process time for different analytical methods

In the End - Consumables

A bottomless pit

The operation of any AAS or ICP spectrometer requires the use of carrier and burning gas, standards for regular calibration and cooling water. Even at average sample throughput the costs for all consumables will sum up to about 25 % of the purchase price of an ICP-OES and 50 % of an AAS per year.

With TXRF expenses for consumables will be reduced dramatically. Inexpensive one-element standards will last for hundreds of measurements. Highest sensitivity will be achieved when using quartz sample carriers, which can be reused for more than 100 measurements. The following pictures explain the simple cleaning procedure of the carriers.

Carrier disk cleaning

- Remove sample material manually with a lintfree wipe soaked with acetone.
- Plug quartz discs in a cleaning cassette (incl. in delivery).
- Place cleaning cassette in a 1 litre glass beaker filled with 10 % nitric acid and simmer for 2 hours on a heating plate.
- Rinse the cleaning cassette with distilled water.
- Place it in a beaker filled with ultrapure water.
- Heat the beaker in a microwave oven for about 5 minutes at 800 W.
- Dry the carriers in a laboratory oven for 20 minutes at 80 °C.
- Run a blank measurement of the cleaned discs for about 180 s.
- Done! Store the clean carriers carefully without risk of contamination.

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