S4 EXPLORER
HIGHEST PRECISION OIL ANALYSIS WITH 1 KW

Introduction

XRF is a well developed and established method for the analysis of oils. Several norms in Europe and the US are based on XRF and have a long and successful history using round robin tests and inter-laboratory cross checks.

The elements of interest for different kind of oils are:

Oil	Elements of interest	Source
Raw oil	S, V and Ni	Geogenic (formation)
Lube oils	Mg, P,(S,) Ca and Zn	Additives
Used oils	Al, Si, Cr, Fe, Ni, Cu, Mo, Sn and Pb	Wear metals

Most of these norms require excitation conditions of more than 2 kW. Others allow the application of so called "low power" instruments in case the repeatability is proven to be the same as those for norm compliant "high power" spectrometers (which for the norms tested up to now was successfully done).

This report shows the excellent instrument reproducibility of S4 EXPLORER (1 kW) analysis results of standard oil samples. The results of these measurements of course are much better than the reproducibility of "real" used oil results as typical effects like sedimentation and inhomogeneities are excluded. Nevertheless one has to use ideal samples to exclude preparation effects as much as possible and therefore examine the instrument performance.

Measurement and Results

The measurements were carried out using an S4 EXPLORER with a standard configuration. The measurement parameters are taken from a precalibrated solution "Bruker AXS OilQuant" analyzing up to 26 elements down to the low µg/g range in less than 30 min.

In this case the elements Mg, Al, Si, P, S, Ca, Ti, V, Cr, Mn, Fe, Ni, Cu, Zn, Mo, Sn and Pb were analyzed. The measurement times were optimized to ensure a standard deviation of less than 1.5 µg/g leading to an analysis time of 13.5 min.

Two samples were prepared by diluting a Conostan S21-100 reference oil with corresponding Base Oil 75 to a concentration of 10 µg/g and 5 µg/g for all elements but S. As these Conostan standards are made of sulfonatic compounds the sulfur concentration itself is much higher. The samples were stirred thoroughly and individually prepared fresh prior to each measurement. The film used for preparation of the sample cups was Prolene with a 4 µm thickness. 7.0±0.1 g of the well stirred solution were poured into the liquid cup, checked 30 s for leakage on a tissue and immediately measured.

The following two tables show the results of these reproducibility measurements:

10µg/g (n=10)	Mg (PPM)	Al (PPM)	Si (PPM)	P (PPM)	Ca (PPM)	Ti (PPM)	V (PPM)	Cr (PPM)
average	10.5	9.0	9.1	8.4	9.6	10.9	9.1	10.9
abs.std.dev.	1.0	0.6	1.2	1.4	0.6	1.4	0.8	0.7
rel.std.dev	9.4	7.1	12.9	16.1	6.1	12.6	8.5	6.8
	Mn (PPM)	Fe (PPM)	Ni (PPM)	Cu (PPM)	Zn (PPM)	Mo (PPM)	Sn (PPM)	Pb (PPM)
average	9.8	9.8	10.0	10.1	9.7	9.1	10.8	10.0
abs.std.dev.	0.7	0.4	0.4	0.4	0.3	0.4	1.6	0.4
rel.std.dev	7.1	4.3	4.2	4.0	3.1	4.1	14.6	4.0

5µg/g (n=10)	Mg (PPM)	Al (PPM)	Si (PPM)	P (PPM)	Ca (PPM)	Ti (PPM)	V (PPM)	Cr (PPM)
average	6.8	4.4	6.2	4.3	4.5	5.2	4.3	5.9
abs.std.dev.	1.0	0.8	2.0	1.1	0.8	1.0	0.9	0.6
rel.std.dev	14.8	18.0	33.1	25.4	18.8	18.8	20.6	9.8
	Mn (PPM)	Fe (PPM)	Ni (PPM)	Cu (PPM)	Zn (PPM)	Mo (PPM)	Sn (PPM)	Pb (PPM)
average	5.2	5.1	5.2	5.2	5.0	4.2	5.4	4.7
abs.std.dev.	0.7	0.5	0.4	0.3	0.3	1.0	1.6	0.4
rel.std.dev	12.7	9.6	7.1	6.5	5.1	23.5	29.8	7.9

Conclusion

These data impressively show the excellent reproducibility of the S4 EXPLORER that one normally only expects from state-of-the-art 3-4 kW spectrometers.

Obviously

- no counter gas
- no compressed air
- no external cooling water
- no large footprint and
- no high power tube

are required to achieve excellent analytical results.

All that is required is an innovative well designed 1 kW spectrometer with

- a vacuum seal for highest precision by keeping the spectrometer chamber in vacuum (further more reducing the mode change time and the He consumption)
- a high performance sealed proportional counter
- a tube and generator with long term stability
- an optimized beam path and
- an outstanding internal temperature control

– which is the Bruker AXS S4 EXPLORER.

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The following two tables show all data of these reproducibility measurements:

	Mg (PPM)	Al (PPM)	Si (PPM)	P (PPM)	Ca (PPM)	Ti (PPM)	V (PPM)	Cr (PPM)
10µg/g run 01	9.0	9.8	9.8	10.2	10.9	9.8	9.9	11.8
10µg/g run 02	10.2	8.5	9.5	9.0	9.7	12.1	9.2	11.5
10µg/g run 03	9.2	9.9	8.4	9.5	9.2	9.9	8.7	11.0
10µg/g run 04	11.2	9.6	9.0	8.2	9.8	11.5	9.0	10.9
10µg/g run 05	11.5	8.9	7.7	8.6	10.1	11.8	10.2	10.5
10µg/g run 06	9.9	8.7	11.5	5.4	9.7	9.5	10.1	10.2
10µg/g run 07	11.2	8.8	9.9	9.4	9.0	11.9	8.9	11.7
10µg/g run 08	10.5	8.6	7.7	7.2	9.0	12.6	7.8	9.4
10µg/g run 09	10.2	7.9	8.8	8.8	9.2	8.4	9.1	11.4
10µg/g run 10	12.0	9.3	8.2	8.1	9.5	11.1	8.3	11.0
average	**10.5**	**9.0**	**9.1**	**8.4**	**9.6**	**10.9**	**9.1**	**10.9**
abs.std.dev.	**1.0**	**0.6**	**1.2**	**1.4**	**0.6**	**1.4**	**0.8**	**0.7**
rel.std.dev	**9.4**	**7.1**	**12.9**	**16.1**	**6.1**	**12.6**	**8.5**	**6.8**
max	**12.0**	**9.9**	**11.5**	**10.2**	**10.9**	**12.6**	**10.2**	**11.8**
min	**9.0**	**7.9**	**7.7**	**5.4**	**9.0**	**8.4**	**7.8**	**9.4**
range	**3.0**	**2.0**	**3.8**	**4.8**	**1.9**	**4.2**	**2.4**	**2.4**

	Mn (PPM)	Fe (PPM)	Ni (PPM)	Cu (PPM)	Zn (PPM)	Mo (PPM)	Sn (PPM)	Pb (PPM)
10µg/g run 01	9.1	9.7	10.2	10.5	9.9	9.1	10.3	9.3
10µg/g run 02	9.9	10.2	9.8	9.6	9.8	9.2	10.9	10.1
10µg/g run 03	10.6	9.8	9.6	10.4	9.6	9.0	7.7	10.2
10µg/g run 04	11.0	10.2	9.6	9.6	9.7	9.3	11.1	9.7
10µg/g run 05	8.9	9.7	10.2	9.8	9.5	9.8	9.4	10.0
10µg/g run 06	9.3	10.3	10.1	10.2	10.4	8.4	11.7	10.6
10µg/g run 07	9.6	9.3	10.7	10.9	9.8	8.7	13.7	9.8
10µg/g run 08	9.4	10.3	10.4	10.1	9.5	9.1	10.2	10.0
10µg/g run 09	10.5	9.3	10.4	10.0	9.5	9.3	11.3	10.5
10µg/g run 10	9.8	9.3	9.4	10.1	9.3	9.2	11.4	9.6
average	**9.8**	**9.8**	**10.0**	**10.1**	**9.7**	**9.1**	**10.8**	**10.0**
abs.std.dev.	**0.7**	**0.4**	**0.4**	**0.4**	**0.3**	**0.4**	**1.6**	**0.4**
rel.std.dev	**7.1**	**4.3**	**4.2**	**4.0**	**3.1**	**4.1**	**14.6**	**4.0**
max	**11.0**	**10.3**	**10.7**	**10.9**	**10.4**	**9.8**	**13.7**	**10.6**
min	**8.9**	**9.3**	**9.4**	**9.6**	**9.3**	**8.4**	**7.7**	**9.3**
range	**2.1**	**1.0**	**1.3**	**1.3**	**1.1**	**1.4**	**6.0**	**1.3**

	Mg (PPM)	Al (PPM)	Si (PPM)	P (PPM)	Ca (PPM)	Ti (PPM)	V (PPM)	Cr (PPM)
5µg/g run 01	5.1	5.1	4.7	4.8	4.0	5.5	5.6	5.5
5µg/g run 02	6.2	3.6	5.1	5.9	5.8	3.5	2.7	5.3
5µg/g run 03	7.8	5.2	10.5	2.6	3.3	4.9	4.9	5.9
5µg/g run 04	6.4	3.6	6.7	4.7	3.6	6.1	4.4	6.7
5µg/g run 05	6.7	4.8	8.7	4.0	4.1	6.6	4.7	6.5
5µg/g run 06	6.3	5.2	6.0	5.0	5.4	4.2	5.2	6.2
5µg/g run 07	6.7	4.3	5.3	3.5	4.2	4.8	3.5	5.0
5µg/g run 08	6.0	3.9	3.6	2.6	4.1	4.7	4.1	5.4
5µg/g run 09	8.2	5.3	4.9	5.0	5.1	5.7	3.9	6.3
5µg/g run 10	8.1	3.2	6.3	4.7	5.3	6.3	3.6	6.3
average	**6.8**	**4.4**	**6.2**	**4.3**	**4.5**	**5.2**	**4.3**	**5.9**
abs.std.dev.	**1.0**	**0.8**	**2.0**	**1.1**	**0.8**	**1.0**	**0.9**	**0.6**
rel.std.dev	**14.8**	**18.0**	**33.1**	**25.4**	**18.8**	**18.8**	**20.6**	**9.8**
max	**8.2**	**5.3**	**10.5**	**5.9**	**5.8**	**6.6**	**5.6**	**6.7**
min	**5.1**	**3.2**	**3.6**	**2.6**	**3.3**	**3.5**	**2.7**	**5.0**
range	**3.1**	**2.1**	**6.9**	**3.3**	**2.5**	**3.1**	**2.9**	**1.7**

	Mn (PPM)	Fe (PPM)	Ni (PPM)	Cu (PPM)	Zn (PPM)	Mo (PPM)	Sn (PPM)	Pb (PPM)
5µg/g run 01	4.8	5.3	5.1	5.6	5.2	4.9	5.1	4.9
5µg/g run 02	3.9	6.0	4.9	5.4	5.3	3.5	7.1	4.4
5µg/g run 03	6.1	4.6	4.9	4.9	5.1	4.5	8.1	3.8
5µg/g run 04	5.4	4.5	5.0	5.4	4.7	5.2	4.1	4.7
5µg/g run 05	5.8	4.8	5.5	5.2	4.7	2.6	5.4	5.1
5µg/g run 06	5.0	5.0	5.6	4.7	5.3	5.7	6.4	4.7
5µg/g run 07	5.9	5.0	5.2	5.2	4.9	2.9	6.0	4.9
5µg/g run 08	5.1	4.7	4.7	5.8	5.0	3.9	3.1	4.9
5µg/g run 09	4.7	5.6	5.1	4.9	5.0	4.5	3.3	4.6
5µg/g run 10	5.6	5.5	5.9	5.1	4.6	4.3	5.2	4.5
average	**5.2**	**5.1**	**5.2**	**5.2**	**5.0**	**4.2**	**5.4**	**4.7**
abs.std.dev.	**0.7**	**0.5**	**0.4**	**0.3**	**0.3**	**1.0**	**1.6**	**0.4**
rel.std.dev	**12.7**	**9.6**	**7.1**	**6.5**	**5.1**	**23.5**	**29.8**	**7.9**
max	**6.1**	**6.0**	**5.9**	**5.8**	**5.3**	**5.7**	**8.1**	**5.1**
min	**3.9**	**4.5**	**4.7**	**4.7**	**4.6**	**2.6**	**3.1**	**3.8**
range	**2.2**	**1.5**	**1.2**	**1.1**	**0.7**	**3.1**	**5.0**	**1.3**

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