

### XRF analysis of liquids using sample cups and films



#### Introduction

The preparation of liquids using X-ray fluorescence analysis is done by simply pouring the sample into a sample cup.

But measuring samples in sample cups can be a risk to the XRF instrument. Leaks in the film and damaged or poorly prepared samples cups can lead to liquids dripping onto the X-ray tube. This can cause destruction of the tube. For this reason, only trained personnel should conduct these measurements. Sample cups should only be measured after a given waiting period, in which it can be determined whether there is leakage or not. The films should never be reused.

Liquids or loose powders in sample cups cannot be measured under vacuum, as contamination of the instrument may occur. Such measurements are generally conducted under a helium atmosphere to protect the XRF instrument. With EDXRF instruments it is also possible to conduct the measurements in an air or nitrogen atmosphere.

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### Available sample cups and types of film.

Many different sample cup types are available and can be used depending on the instrument's holder type. You can find a list of available sample cups here:

<https://www.fluxana.com/liquid-analysis-sample-cups/sample-cups>

The bottom of the sample cup is covered with an X-ray transparent film. The chemical resistance depends on the material and the thickness of the film:

Film	Thickness / $\mu\text{m}$	Suitable for	Unsuitable for
Mylar®	1,5		Acids, Bases
	2,5	Gasoline, diesel, solvents	
	3,5		
	6		
Polypropylene	4		Gasoline, Diesel
	6	Diluted acids	
	12		
Polycarbonate	5	Gasoline, diesel	
Kapton®	7,5	Aromates	

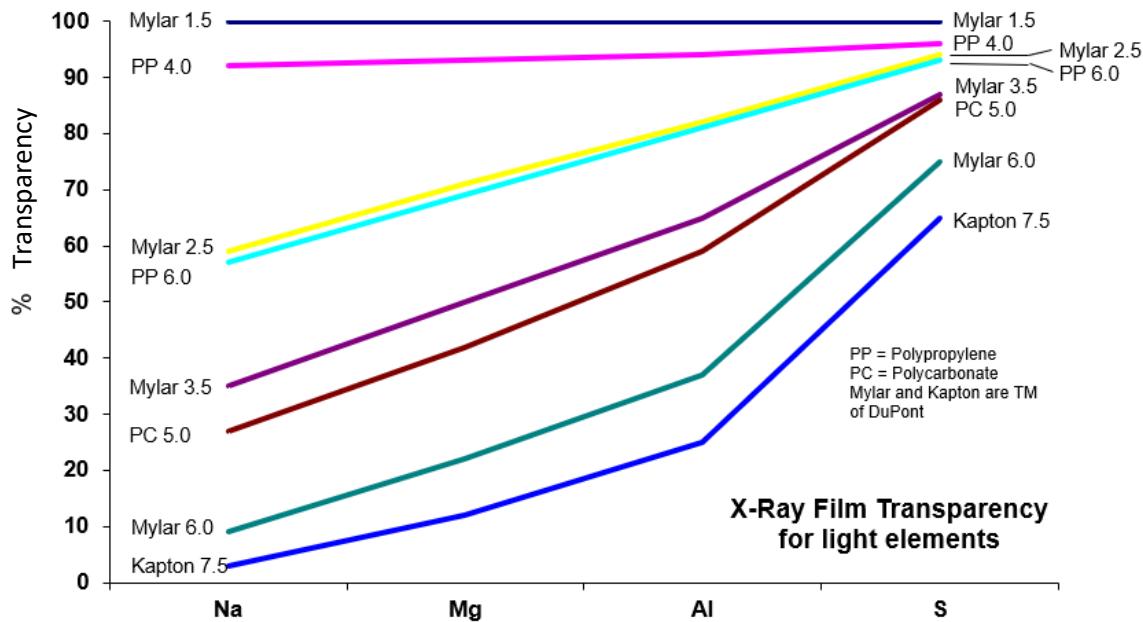
This table gives an overview of the commercially available films. In principle, two materials are used: Polyester (Mylar) und polypropylene (films with trade names that contain fragments of the name "propylene," e.g., that end with "lene", are usually the same material as the much less expensive polypropylene film). The film materials differ in the resistance to chemicals. While polyester is stable with respect to solvents, aliphatics and fuels, polypropylenes provide more stability with respect to fluids rich in oxygen (e.g., water, polyglycols and high-boiling oils).

You can find a list of available films here:

<https://www.fluxana.com/liquid-analysis-film/thin-films>

Transparency for the fluorescence radiation of the light elements from sodium to sulfur is the deciding factor for use of a film. The thinnest Mylar film with 1.5  $\mu\text{m}$  absorbs 50% of the sodium radiation; 6  $\mu\text{m}$  Mylar almost 100%. Analysis of the even lighter element, fluorine, in samples measured with a film is thus impossible. This image provides an overview:

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**Polypropylene** is especially suited to the trace analysis of solid samples, as it has no major contamination in it. In contrast, **Mylar** contains the undesirable phosphorus and calcium (3.5  $\mu$ m Mylar corresponds to 50 ppm Ca and 250 ppm P in a blank oil, e.g., clean white oil).

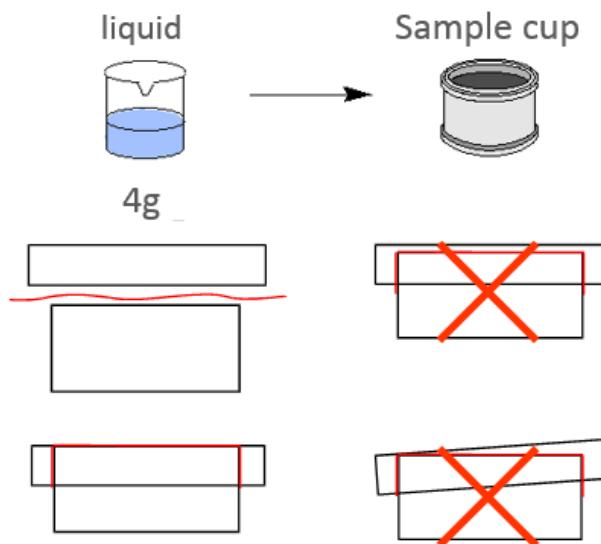
Films such as **Hostaphan** (polyester with a silicon contamination) or polycarbonate (without contamination, preferred for fuel analysis) are restricted or no longer available.

**Kapton** is not very transparent for the fluorescence radiation of light elements and is, therefore, only useful for the analysis of elements with a higher atomic number.

### Correct preparation of the sample cup

With sample cups, the analytical error depends on the preparation of the sample cups. The exact fit of the film, the outer ring and a defined height are decisive. The distance of the X-ray tube from the film on the cup must always be constant. For this reason, it is important to be sure that the outer ring of the cup, which holds the film, must be pulled up enough so that the film touches the bottom of the holder or is at the same height.

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If available, it is advisable to use a lid, as this makes handling easier and minimizes the risk of spillage. In addition, some liquid samples evaporate during measurement, and a lid can prevent liquid from entering the measuring device.

If you do not want to insert your sample cup yourself and would like to save time, you can order ready-prepared sample cups as an alternative.

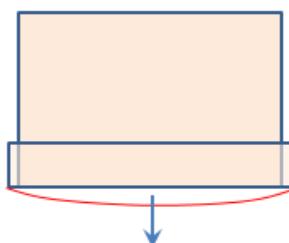
Further information can be found here:

<https://www.fluxana.com/liquid-analysis-prepared-cups/prepared-sample-cups>

#### High power WDXRF X-ray radiation shows interaction with the film:

- Polypropylene sags a little
- Polyester shows no changes

The radiation contamination of the film, which leads to sagging, can be greatly reduced through use of a thin primary filter (e.g., Al).



EDXRF and WDXRF instruments with lower power (<50 Watt) do not show this effect.

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### How much sample material must be placed in the sample cups?

Depending on the XRF instrument used, different geometries arise that determine the analyzed volume and the penetration depth. In addition, the quantity depends on the diameter of the sample cup. It is important that the filling height remains constant and that sufficient sample quantity is ensured.

### Special cases for liquid samples

Liquids containing particles and liquids with multiple phases, e.g., liquid waste samples (recycling sector):

In this case, absorption by a binding agent (CELLEOX®) mixed with the sample is recommended. This results in a solid paste that is put into the sample cup with a stamp and then measured.

Solid fat samples (lubricating greases):

Here, the use of so-called BOREOX® cups (small pressed pellets with a recess) is recommended, into which a defined amount of the fat is applied, e.g., with a spatula. The BOREOX® cup is then placed into a sample cup and can be measured.

### Summary

To successfully measure liquids, the correct

- sample cup
- foil
- procedure

must be selected.

### Literature

- [1] Rainer Schramm, X-Ray Fluorescence Analysis: Practical and Easy, 2<sup>nd</sup> edition, FLUXANA (2017).
- [2] [www.fluxana.com](http://www.fluxana.com)