

# Reagents for Pharma Industry

## Chapter 2

### Spectroscopy



**PanReac**   
**AppliChem**  
ITW Reagents

## UV/VIS Spectrophotometry Standards

UV/VIS spectrophotometers must be checked periodically to ensure their correct functioning, thereby guaranteeing the reliability of their results. The European Pharmacopoeia and the USP recommend certain solutions for the control of UV/VIS equipment that can be used to detect possible deviations arising from electrical, mechanical or optical problems.

The parameters to be checked in UV/VIS spectroscopy according to the European Pharmacopoeia and USP are as follows:

- Absorbance
- Accuracy of the wavelength
- Stray light limit
- Spectral resolution

PanReac AppliChem supplies four liquid standards that allow these parameters to be verified with the following advantages:

- Compliance with Ph. Eur. and USP
- Packaged in ready-to-use, disposable ampoules
- Certificate of analysis with each pack
- Three-year expiry date



Solutions for the control of:	Composition	Code	Package
<b>Absorbance</b>	Potassium Dichromate solution 4x10 mL standard ampoules (60.06 mg/L $K_2Cr_2O_7$ in $H_2SO_4$ 0.01N) 4x10 mL blank ampoules ( $H_2SO_4$ 0.01N)	395462.2590	8 x 10 ml
<b>Spectral resolution</b>	Toluene solution in n-Hexane 4x10 mL standard ampoules (0.02 mL toluene in 100 mL n-hexane) 4x10 mL blank ampoules (n-hexane)	395461.2590	8 x 10 ml
<b>Stray light</b>	Potassium Chloride solution 8x10 mL standard ampoules (12 g/L KCl in $H_2O$ )	395460.2590	8 x 10 ml
<b>Wavelength</b>	Holmium Oxide solution according to Ph. Eur. 8x10 mL Standard ampoules (40.0 g/L $Ho_2O_3$ in $HClO_4$ 10% v/v)	396070.2590	8 x 10 ml





## Solvents for UV-Vis

When preparing the solution of the substance to be examined, the solvent should completely solubilize the sample at the desired concentration. The solvent should be UV transparent at the measuring wavelength so as not to cause interference which could affect quantitative results.

When using volatile solvents stoppered cells should be employed to eliminate evaporation which can lead to changes in the solution concentration and ultimately affect quantitation.

PanReac AppliChem offers an excellent selection of UV-Vis transparent solvents to meet the most exigent spectrophotometric needs. We assure consistent, dependable performance and extremely low levels of optical impurities. Our solvents offer the following benefits:

- More reliable sample identification, since solvents have highest chemical purity and contain no extraneous peaks
- Enhanced sensitivity, due to highest UV transmittance/lowest UV absorbance
- Reproducible absorption curve throughout the entire spectrum due to the highest batch-to-batch consistency

Product name	Assay (G.C.) min.	Non-volatile matter max.	Water max.	Fluorescence (as quinine) max.		UV transmittance (min.) at $\lambda$					Code	Pack	
				254 nm	365 nm	330 nm	335 nm	340 nm	345 nm	$\geq 350$ nm			
<b>Acetone</b>	99.9 %	0.0003 %	0.2 %	-	2 ppb	15%	60%	85%	95%	98%	361007.1611	☞ 1 L	
											361007.1612	☞ 2.5 L	
											361007.16153	☞ 4 L	
											361007.0515	☞ 10 L	
											361007.0537	☞ 30 L	
<b>Acetic Acid glacial</b>	99.8 %	0.001 %	0.1 %	-	-	25%	95%	98%	361008.1611	☞ 1 L			
									361008.1612	☞ 2.5 L			
<b>Acetonitrile</b>	99.9 %	0.0004 %	0.02 %	1 ppb	1 ppb	55%	70%	90%	98%	361881.1611	☞ 1 L		
										361881.1612	☞ 2.5 L		
										361881.0314	☞ 2.5 L		
										361881.0516	☞ 25 L		
<b>Benzene</b>	99.8 %	0.0003 %	0.01 %	-	2 ppb	25%	70%	80%	90%	95%	98%	361192.1611	☞ 1 L
<b>1-Butanol</b>	99.9 %	0.0003 %	0.03 %	-	-	25%	50%	75%	85%	94%	98%	361082.1611	☞ 1 L
<b>tert-Butyl Methyl Ether</b>	99.9 %	0.0003 %	0.03 %	2 ppb	2 ppb	20%	50%	60%	85%	98%	363312.1611	☞ 1 L	
											363312.1612	☞ 2.5 L	
<b>Carbon Disulfide</b>	99.9 %	0.0005 %	0.005 %	-	-	50%	80%	90%	98%	361244.1611	☞ 1 L		
<b>Chloroform stab. with ~ 150 ppm of amylene</b>	99.8 %	0.0003 %	0.01 %	1 ppb	1 ppb	15%	70%	85%	98%	363101.1612	☞ 2.5 L		

>>

<<

Product name	Assay (G.C.) min.	Non-volatile matter max.	Water max.	Fluorescence (as quinine) max.		UV transmittance (min.) at $\lambda$						Code	Pack
				254 nm	365 nm	245 nm	250 nm	257 nm	260 nm	270 nm	$\geq 280$ nm		
<b>Chloroform stab. with ethanol</b>	99.0 %	0.0003 %	0.01 %	1 ppb	1 ppb	245 nm 20%	250 nm 50%	257 nm 80%	260 nm 85%	270 nm 90%	$\geq 280$ nm 98%	361252.1611	☞ 1 L
<b>Cyclohexane</b>	99.9 %	0.0003 %	0.01 %	1 ppb	1 ppb	210 nm 15%	220 nm 50%	230 nm 80%	240 nm 90%	250 nm 98%	$\geq 260$ nm 99%	361250.1611	☞ 1 L
												361250.1612	☞ 2.5 L
												361250.0515	☞ 10 L
												361250.0537	☞ 30 L
<b>Dichloromethane stab. with ~ 20 ppm of amylene</b>	99.9 %	0.0003 %	0.01 %	1 ppb	1 ppb	235 nm 40%	240 nm 75%	245 nm 90%	255 nm 98%	$\geq 260$ nm 99%	361254.1611	☞ 1 L	
											361254.1612	☞ 2.5 L	
											361254.16153	☞ 4 L	
											361254.0516	☞ 25 L	
<b>Diethyl Ether stab. with ethanol</b>	98.0 %	0.0003 %	0.02 %	2 ppb	0.5 ppb	236 nm 50%	252 nm 80%	280 nm 94%	$\geq 300$ nm 99%	362551.1611	☞ 1 L		
										362551.0537	☞ 30 L		
<b>N,N-Dimethylacetamide</b>	99.9 %	0.0005 %	0.03 %	-	-	280 nm 50%	290 nm 70%	310 nm 89%	320 nm 93%	$\geq 360$ nm 98%	363145.1611	☞ 1 L	
											363145.1612	☞ 2.5 L	
<b>N,N-Dimethylformamide</b>	99.9 %	0.0003 %	0.05 %	-	-	275 nm 60%	290 nm 80%	300 nm 90%	$\geq 330$ nm 98%	361785.1611	☞ 1 L		
										361785.1612	☞ 2.5 L		
<b>Dimethyl Sulfoxide</b>	99.9 %	0.001 %	0.05 %	-	-	270 nm 30%	280 nm 63%	290 nm 70%	310 nm 80%	330 nm 94%	$\geq 350$ nm 98%	361954.1611	☞ 1 L
												361954.1612	☞ 2.5 L
<b>Ethanol absolute</b>	99.9 %	0.0003 %	0.1 %	2 ppb	1 ppb	210 nm 35%	220 nm 55%	235 nm 80%	245 nm 90%	$\geq 270$ nm 98%	361086.1611	☞ 1 L	
											361086.1612	☞ 2.5 L	
											361086.16153	☞ 4 L	
<b>Ethanol 96% v/v</b>	96 %	0.0005 %	-	-	-	210 nm 35%	220 nm 55%	230 nm 72%	240 nm 90%	$\geq 270$ nm 98%	361085.1611	☞ 1 L	
											361085.0537	☞ 30 L	
<b>Ethyl Acetate</b>	99.9 %	0.0003 %	0.01 %	2 ppb	1 ppb	255 nm 20%	257 nm 32%	260 nm 50%	263 nm 80%	265 nm 90%	$\geq 270$ nm 98%	361318.1611	☞ 1 L
												361318.1612	☞ 2.5 L
												361318.16153	☞ 4 L
												361318.0515	☞ 10 L
<b>n-Heptane</b>	99.0 %	0.0003 %	0.005 %	1 ppb	1 ppb	210 nm 50%	220 nm 80%	230 nm 92%	$\geq 245$ nm 98%	362062.1611	☞ 1 L		
										362062.1612	☞ 2.5 L		
										362062.0314	☞ 5 L		
										362062.0537	☞ 30 L		
<b>n-Hexane</b>	99.0 %	0.0003 %	0.005 %	2 ppb	2 ppb	200 nm 20%	210 nm 60%	220 nm 85%	225 nm 90%	230 nm 94%	$\geq 245$ nm 98%	362063.1611	☞ 1 L
												362063.1612	☞ 2.5 L
<b>n-Hexane 95%</b>	98.5 % (isomers)	0.0003 %	0.01 %	-	-	200 nm 20%	210 nm 60%	220 nm 80%	230 nm 94%	$\geq 245$ nm 98%	363242.1611	☞ 1 L	
											363242.1612	☞ 2.5 L	
											363242.0515	☞ 10 L	
											363242.0537	☞ 30 L	
<b>Hexane, alkanes mixture</b>	95.0 % (isomers)	0.0005 %	0.01 %	-	-	210 nm 40%	220 nm 85%	$\geq 254$ nm 99%	361347.1612	☞ 2.5 L			

>>

# Reagents for Pharma Industry

## Chapter 2



<<

Product name	Assay (G.C.) min.	Non-volatile matter max.	Water max.	Fluorescence (as quinine) max.		UV transmittance (min.) at $\lambda$				Code	Pack		
				254 nm	365 nm	210 nm	220 nm	235 nm	$\geq 245$ nm				
<b>Isooctane</b>	99.5 %	0.0003 %	0.005 %	1 ppb	1 ppb	210 nm 50%	220 nm 80%	235 nm 90%	$\geq 245$ nm 98%	362064.1611	☞ 1 L		
										362064.1612	☞ 2.5 L		
										362064.16153	☞ 4 L		
<b>Methanol</b>	99.9 %	0.0005 %	0.03 %	1 ppb	1 ppb	210 nm 30%	220 nm 55%	230 nm 75%	240 nm 90%	$\geq 260$ nm 98%	361091.1611	☞ 1 L	
											361091.1612	☞ 2.5 L	
											361091.16153	☞ 4 L	
<b>n-Pentane</b>	99.5 %	0.0003 %	0.005 %	1 ppb	1 ppb	200 nm 40%	210 nm 70%	215 nm 80%	220 nm 90%	$\geq 240$ nm 98%	362006.1611	☞ 1 L	
											362006.1612	☞ 2.5 L	
<b>1-Propanol</b>	99.8 %	0.0002 %	0.1 %	-	-	210 nm 15%	230 nm 70%	240 nm 80%	250 nm 94%	$\geq 260$ nm 98%	361885.1611	☞ 1 L	
											361885.1612	☞ 2.5 L	
<b>2-Propanol</b>	99.9 %	0.0003 %	0.05 %	-	-	217 nm 50%	232 nm 80%	242 nm 90%	250 nm 95%	$\geq 260$ nm 98%	361090.1611	☞ 1 L	
											361090.1612	☞ 2.5 L	
											361090.16153	☞ 4 L	
<b>Propionitrile</b>	99.9 %	0.0005 %	0.03 %	-	2 ppb	235 nm 94%	250 nm 97%	$\geq 290$ nm 99%	365732.1611	☞ 1 L			
<b>Tetrachloro-ethylene</b>	99.9 %	0.0005 %	0.01 %	-	2 ppb	295 nm 50%	300 nm 80%	305 nm 85%	350 nm 89%	$\geq 400$ nm 94%	361455.1611	☞ 1 L	
											361455.1612	☞ 2.5 L	
<b>Tetrahydrofuran</b>	99.9 %	0.0002 %	0.02 %	-	-	240 nm 30%	245 nm 50%	260 nm 70%	265 nm 80%	275 nm 90%	$\geq 310$ nm 99%	361736.1611	☞ 1 L
												361736.1612	☞ 2.5 L
<b>Toluene</b>	99.9 %	0.0003 %	0.01 %	-	2 ppb	288 nm 32%	290 nm 50%	293 nm 63%	300 nm 80%	310 nm 90%	$\geq 350$ nm 98%	361745.1611	☞ 1 L
												361745.1612	☞ 2.5 L
												361745.16153	☞ 4 L
<b>Water</b>	-	0.0003 %	-	1 ppb	0.5 ppb	200 nm 98%	210 nm 98%	254 nm 99%	$\geq 300$ nm 99%	361074.1611	☞ 1 L		
										361074.1612	☞ 2.5 L		

All solvents are filtered through 0.2  $\mu$ m and bottled under nitrogen atmosphere. APHA color:  $\leq 10$





## Solvents for UV-Vis in Returnable Containers

The consumption of solvents in 10 L and 30 L returnable stainless steel containers represents a fantastic opportunity to contribute to environmental sustainability.

It also involves a significant saving in the consumption of packaging materials as a result of the long-lasting and resistant nature of stainless steel containers, which therefore simplifies laboratory waste management.

Download our brochure "Solvents in Returnable Containers" and find out more at [https://www.itwreagents.com/download\\_file/brochures/A176/en/A176\\_en.pdf](https://www.itwreagents.com/download_file/brochures/A176/en/A176_en.pdf)



**Types of barrels & Withdrawal systems**

### 10 L – code BIN010TRF

Material	Stainless steel AISI 304
Total Capacity	12.2 L
Diameter	229 mm
Height	390 mm
Wall thickness	1 mm
Opening	Thread 6L 45, internal opening 28 mm
Weight	2.4 kg
Admissible working pressure	max. 0.3 bar
UN Transport approval	1A1 / X 2.0 / 900



### Accessories

Description	Code
Withdrawal system for manual pressurizing	BIS10RF

### 30 L – code BIN030RF

Material	Stainless steel AISI 304
Total Capacity	33 L
Diameter	383 mm
Height	435 mm
Wall thickness	1.5 mm
Opening	Central bung 2" with Tri-Sure screw cap
Weight	10 kg
Admissible working pressure	max. 5 bar
UN Transport approval	1A1 / X 2.0 / 900





### Accessories

Description	Code
Withdrawal system for manual pressurizing	DIN0X3RF
Hand pump ball	BOMBIXRF
Withdrawal system for compressed air or nitrogen supply pressurizing	GDIN0XRF
Metal wrench for drums with 2" and 3/4" closures Tri-Sure	100004RF



3



## Infrared Spectroscopy (IR)

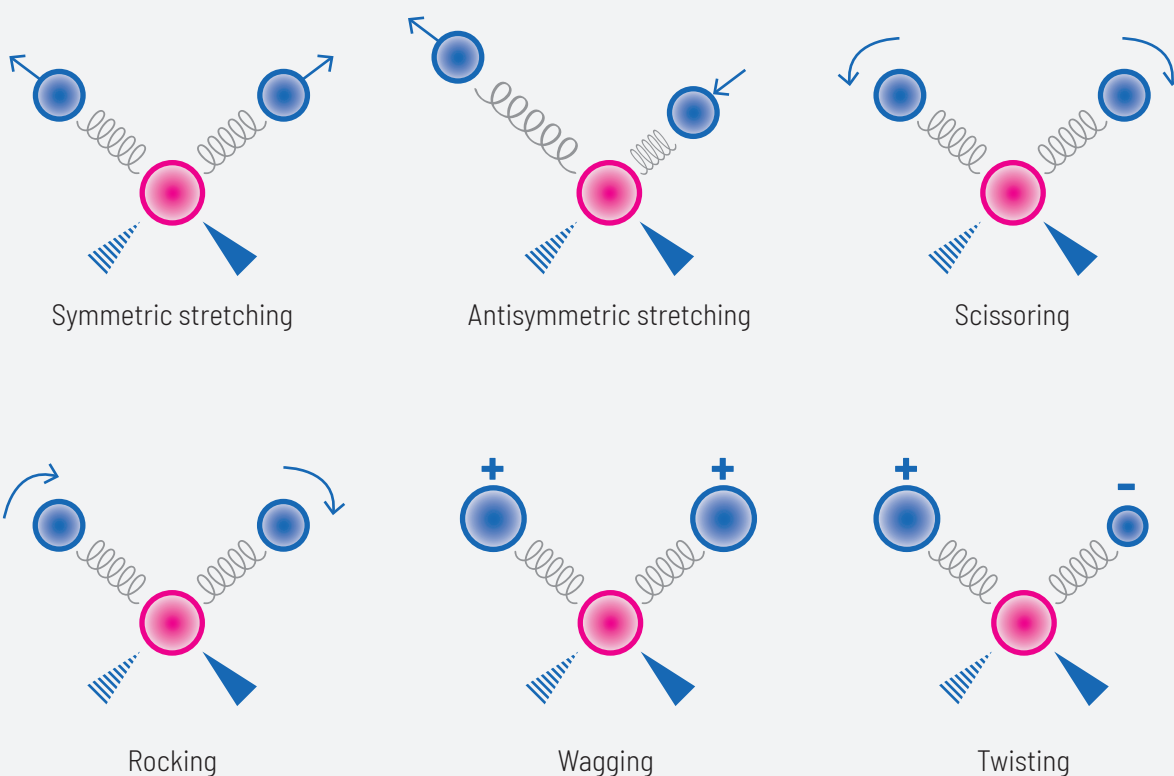
Infrared spectroscopy (IR spectroscopy or vibrational spectroscopy) involves the interaction of **infrared radiation**,  $12800 - 10 \text{ cm}^{-1}$  ( $780 \text{ nm} - 1000 \text{ }\mu\text{m}$ ), with **a molecule**.

**The IR region** of the spectrum can be divided into three zones, both from the point of view of applications and devices: **Near IR (NIR)**:  $12800-4000 \text{ cm}^{-1}$ , **Mid IR**:  $4000-400 \text{ cm}^{-1}$ ; **Far IR**:  $400-10 \text{ cm}^{-1}$ , being the Mid-IR region where most of the traditional analytical applications occur.

It covers a range of techniques, mostly based on **absorption** spectroscopy. It relies on the fact that molecules absorb **specific frequencies** that are characteristic of their structure and can be associated with a **particular type of bond**.

A molecule can **vibrate** in many ways (i.e. symmetric and antisymmetric **stretching**, **scissoring**, **rocking**, **wagging** and **twisting**), and each way is called a vibrational mode and is reflected in the **IR spectrum**. The more complex a molecule is, the more bonds it has and their vibrational spectra are correspondingly more complex.

### Molecular vibrational modes



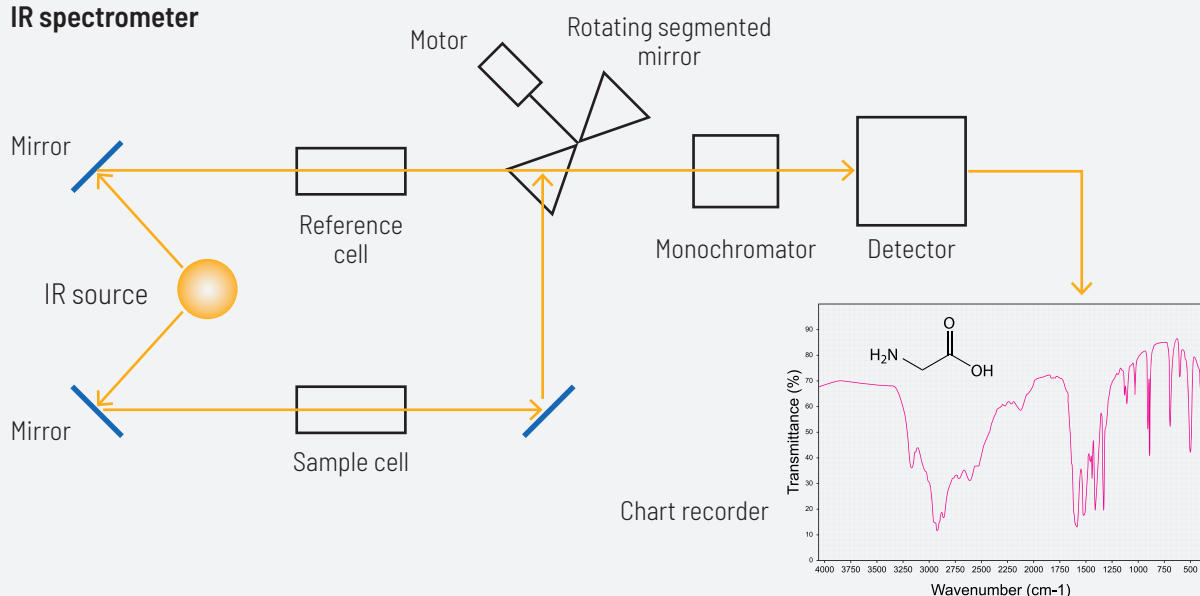
Infrared (IR) spectroscopy is one of the most common and widely used spectroscopic techniques by inorganic and organic chemists due to its usefulness in **determining structures of compounds** and **identifying them** as each functional group absorbs at a different and specific IR frequency.

## Apparatus

The **spectrophotometers** used to obtain the near and medium infrared consist of a **light source, monochromator** or interferometer and **detector**, which allow obtaining spectra in the region between 780 nm and 25000 nm ( $12800\text{ cm}^{-1}$  to  $400\text{ cm}^{-1}$ ). Currently, infrared spectrophotometers use an interferometer instead of a monochromator in which case the polychromatic radiation impinges on the sample and the spectra are obtained in the frequency domain with the help of the Fourier transform.



### IR spectrometer



Normally the spectrum is given as a function of transmittance, the quotient of the intensity of the transmitted radiation and the incident radiation. It may also be given in absorbance.

The absorbance (A) is defined as the logarithm to base 10 of the reciprocal of the transmittance (T):

$$A = \log_{10} \left( \frac{1}{T} \right) = \log_{10} \left( \frac{I_0}{I} \right)$$

$$T = I/I_0$$

$I_0$  = intensity of incident radiation

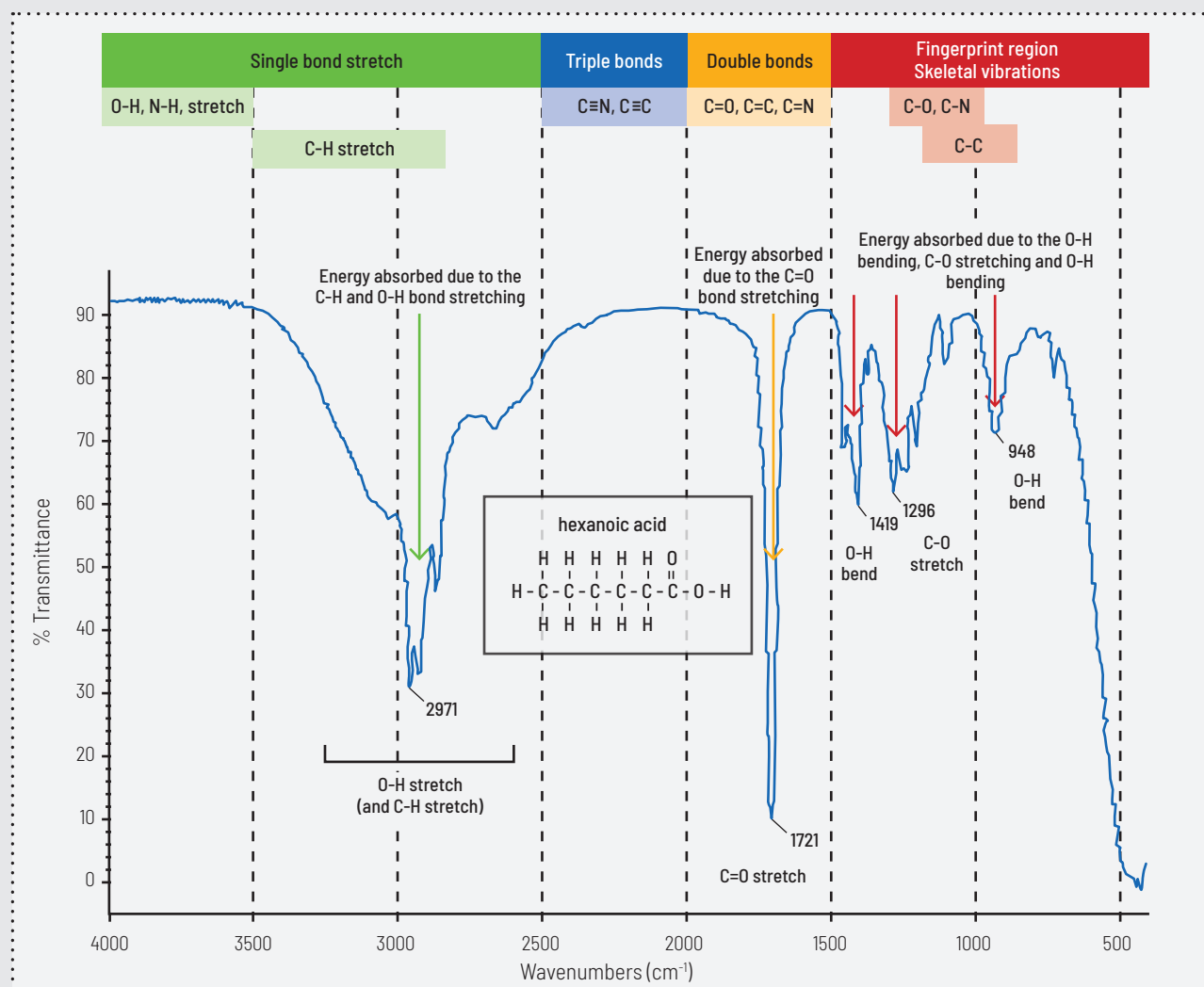
$I$  = intensity of transmitted radiation





## IR Spectrum

The following graphic shows an example of an infrared spectrum for hexanoic acid with five different absorptions characteristics of different bond vibrations.



## Sample preparation

IR Spectroscopy is a versatile technique that allows to obtain spectra of solids, liquids and gases using in each case the appropriate cells or supports that must be transparent to the incident radiation. Alkali halides are the most commonly used in the transmission methods (NaCl, KBr, KCl, etc.). In comparison with other instrumental techniques, the samples to be analyzed require little or no preparation.

**Gaseous samples** require a sample cell with a long path length to compensate for the diluteness. The path length of the sample cell depends on the concentration of the compound of interest. A simple glass tube with length of 5 to 10 cm equipped with infrared-transparent windows at the both ends of the tube can be used for concentrations down to several hundred ppm.

**Liquid samples** can be sandwiched between two plates of a salt (commonly sodium chloride, although other salts such as potassium bromide or calcium fluoride are also used). The plates are transparent to the infrared light and do not introduce any lines onto the spectrum.

**Liquid or solid samples in solution** are prepared in a suitable solvent, choosing a concentration and a path length of the cell that give a satisfactory spectrum. Generally, good results are obtained with concentration of 10-100 g/L for a path length of 0.5-0.1 mm. The absorption due to the solvent is compensated by placing in the reference beam a similar cell containing the solvent used.

**Solid samples** are dispersed in a suitable liquid (mull) or in a solid (halide disc), as appropriate. If prescribed in the monograph, make a film of a molten mass between 2 plates transparent to infrared radiation.

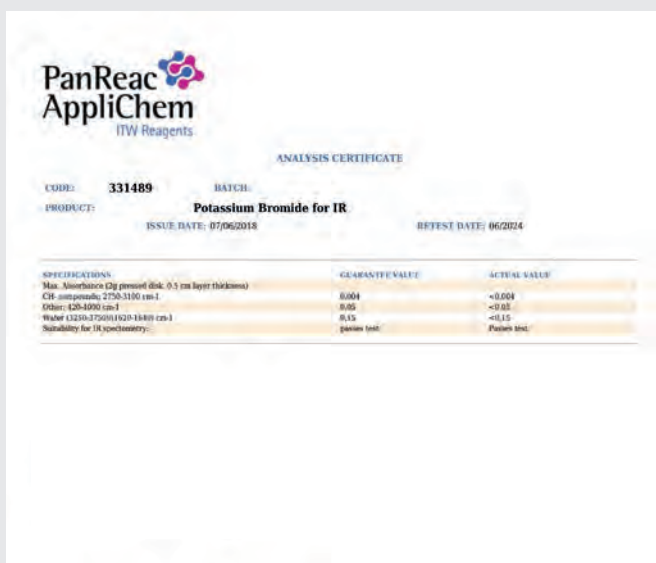
## Potassium Bromide for IR Spectroscopy

Potassium bromide (KBr) is the most common alkali halide used as infrared optical windows and components for general spectroscopy because of its transparency from the near ultraviolet to long-wave infrared wavelengths (0.25-25  $\mu\text{m}$ ) and has no significant optical absorption lines in its high transmission region.

In IR spectroscopy, solid samples which are difficult to melt or dissolve in any suitable IR-transmitting solvent are analyzed by grinding with potassium bromide powder, and pressing into a disc.

This method requires a high quality potassium bromide.

In PanReac AppliChem we produce a high quality potassium bromide specially controlled to satisfy the highest requirements in IR spectroscopy.



**PanReac AppliChem**  
ITW Reagents

ANALYSIS CERTIFICATE

CODE: **331489** BATCH: \_\_\_\_\_  
PRODUCT: **Potassium Bromide for IR**  
ISSUE DATE: 07/06/2018 REFEST DATE: 06/2024

SPECIFICATIONS	GUARANTEE VALUE	ACTUAL VALUE
Max. Absorbance (2g pressed disc, 0.1 cm layer thickness)	0.004	<0.004
Cl <sup>-</sup> impurities: 2750-3100 cm <sup>-1</sup>	0.05	<0.05
Other: 420-4900 cm <sup>-1</sup>	0.15	<0.15
Water (3230-3720) (620-1840) cm <sup>-1</sup>	0.15	<0.15
Suitability for IR Spectrometry:	Passes test	Passes test



Product name	Code	Package
<b>Potassium Bromide for IR</b>	331489.1608	100 g
	331489.1609	250 g

## Solvents for IR Spectroscopy

The solvent should not exhibit interactions or chemical reactions with the substance to be analyzed, nor should it damage the cell. Those regions of the spectrum in which the solvent has a strong absorption should not be taken into account. The organic solvents to be used must be free of water.

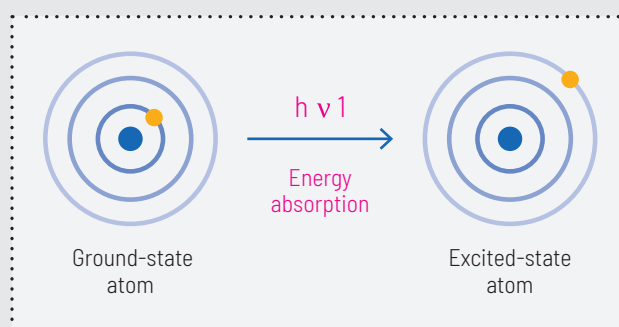
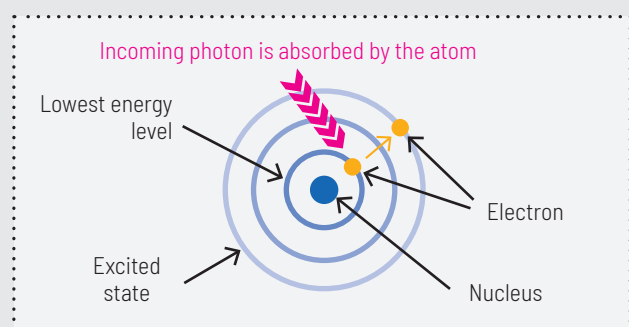
You can find the solvents that PanReac AppliChem can offer in the previous section dedicated to UV spectroscopy.





## Atomic Absorption Spectroscopy (AAS)

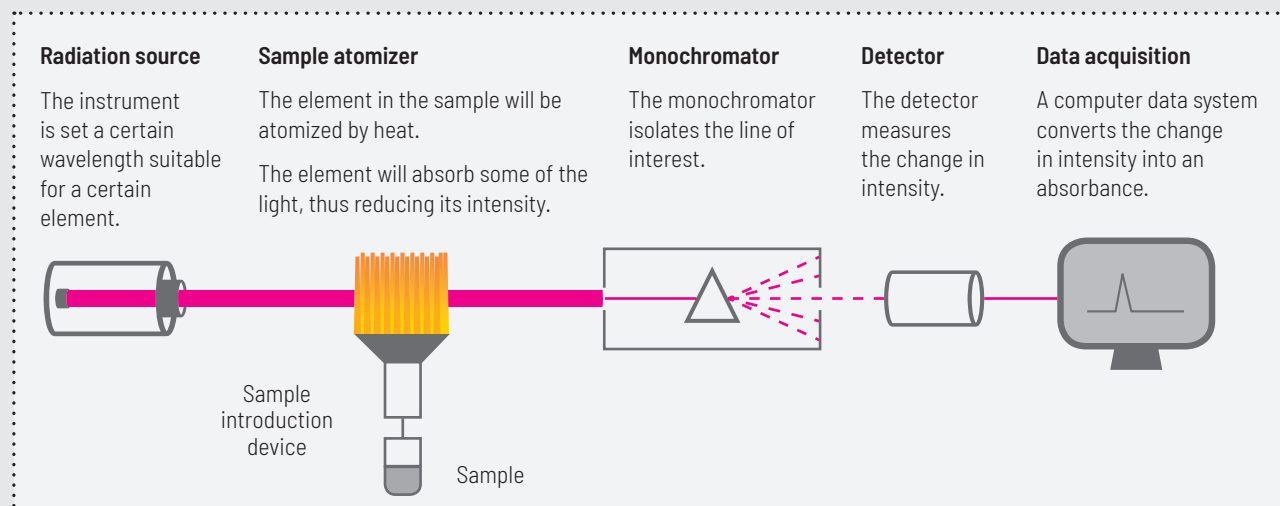
In the **Atomic Absorption Spectroscopy** the visible and ultraviolet light interacts with the atoms. This is a process that occurs when a ground-state atom absorbs electromagnetic radiation of a specific wavelength and is elevated to an excited state. This is a technique for determining the concentration of an element in a sample by measuring the **absorption of electromagnetic radiation** by the **atomic vapor** of the **element** generated from the sample. The amount of radiation absorbed is proportional to the element concentration.



### Apparatus

The apparatus consists essentially of:

- A source of radiation (e.g. hollow-cathode lamps, electrodeless discharge lamps (EDL))
- A sample introduction device
- A sample atomizer
- A monochromator or polychromator
- A detector
- A data acquisition unit





There are 3 types of sample atomizers

- **Flame technique:** a flame atomizer is composed of a **nebulization system** regulation and a **burner**. Fuel-oxidant mixtures are commonly used to produce a range of temperatures from about 2000 K to 3000 K. Samples are nebulized and acidified water is the solvent of choice.
- **Cold vapor and hydride technique:** the atomic vapor may also be generated outside the spectrometer. This is notably the case for cold-vapor method for **mercury** or for certain hydride-forming elements such as **arsenic, antimony, bismuth, selenium** and **tin**. For mercury, atoms are generated by chemical **reduction** with **stannous chloride or sodium borohydride** and the atomic vapor is swept by a stream of an inert gas into a cold quartz cell mounted in the optical path of the instrument. **Hydrides** thus generated are swept by an inert gas into a heated cell in which they are **dissociated into atoms**.
- **Electrothermal atomization technique:** an electrothermal atomizer is generally composed of a **graphite tube furnace** and an **electric power source** atomizes the entire sample and retains the atomic vapor in the light path for an extended period. This **improves the detection limit**. Samples, **liquid as well as solid**, are introduced directly into the graphite tube furnace which is heated in a programmed series of steps to dry the sample and remove major matrix components by pyrolysis and to then atomize all of the analyte.

## Interferences

Chemical, ionization, physical and spectral interferences are encountered in atomic absorption measurements.

- **Chemical interferences** are due to the formation of stable compounds at the combustion temperature of the flame. It can be compensated adding matrix modifiers (as **lanthanum salts**) or releasing agents (as interferences of P, Al or Si).
- **Ionization interferences** are due when the element is easily converted to an ion. Then the number of atoms in ground state able to absorb energy is reduced thus the atomic absorption is reduced. The use of specific ionization buffers (as **Cesium Chloride** or **Potassium Chloride**), which are much easier to ionize, compensates for this effect.
- **Physical interferences** as viscosity or high levels of salts can be eliminated by diluting the sample, for example.
- **Spectral interferences** can be avoided by using a different resonance line.

## Sample preparation

The preparation of the sample may require a dissolution, a digestion (mostly microwave-assisted), an ignition step or a combination thereof in order to clear up the sample matrix and/or remove carbon-containing material.

Sample preparation is always a critical step in atomic spectroscopy analyses. The **reagents used** for sample preparation must be **very pure** in order to make sure to reliably determine the trace elements you are analyzing for.

We offer a **range of acids** of different purity adequate depending on the type of AAS technique used. We also offer **matrix modifiers** to suppress the chemical or ionization interferences, **reduction reagents** (for the cold vapor and hydride techniques) and **standards** traceable to NIST standards reference materials.

# Reagents for Pharma Industry

## Chapter 2



### Acids

Product name	Flame	Cold Vapor	Hydrides	Graphite chamber	Code	Package
<b>Hydrochloric Acid 37%</b>				X	381020.1611	1000 ml
					381020.1612	2.5 L
<b>Hydrochloric Acid 37%</b> (low Hg content)	X	X	X		471020.1611	1000 ml
					471020.1612	2.5 L
<b>Nitric Acid 65%</b>				X	383255.1609	250 ml
					383255.1611	1000 ml
<b>Nitric Acid 65%</b> (low Hg content)	X	X	X		473255.1611	1000 ml
					473255.1612	2.5 L
<b>Sulfuric Acid 95-98%</b> (low Hg content)	X	X	X		471058.1611	1000 ml
					471058.1612	2.5 L
					471058.0716	25 L

### Matrix modifiers

Product name	Chemical suppressor	Ionization suppressor	Code	Package
<b>Buffer Solution Cesium Chloride/Lanthanum Chloride</b>	X	X	176168.1211	1000 ml
<b>Cesium Chloride</b>		X	122509.1206	25 g
			122509.1208	100 g
<b>Lanthanum(III) Chloride 7-hydrate</b>	X		122848.1208	100 g
<b>Lanthanum(III) Nitrate 6-hydrate</b>	X		122669.1208	100 g
<b>Lanthanum(III) Oxide (Reag. Ph. Eur.)</b>	X		122705.1607	50 g
			122705.1609	250 g
<b>Potassium Chloride</b>		X	131494.1210	500 g
			131494.1211	1000 g



### Reducing agents

Product name	Code	Package
<b>Hydroxylammonium Chloride</b>	471914.1209	250 g
<b>Sodium Borohydride for analysis</b>	123314.1606	25 g
	123314.1608	100 g
<b>Tin(II) Chloride 2-hydrate</b>	471303.1609	250 g

### Oxidizing agents

Product name	Code	Package
<b>Potassium Permanganate</b>	471527.1210	500 g

## Standards

Product name	Element (1 g/L)	Composition	Code	Package
<b>Aluminium</b>	Al	Al(NO <sub>3</sub> ) <sub>3</sub> ·9H <sub>2</sub> O in HNO <sub>3</sub> 0.5N	313170.1208	100 ml
			313170.1210	500 ml
<b>Arsenic</b>	As	As <sub>2</sub> O <sub>3</sub> in HNO <sub>3</sub> 2-5%	313171.1208	100 ml
<b>Barium</b>	Ba	Ba(NO <sub>3</sub> ) <sub>2</sub> in HNO <sub>3</sub> 0.5N	313172.1208	100 ml
<b>Cadmium</b>	Cd	Cd(NO <sub>3</sub> ) <sub>2</sub> ·4H <sub>2</sub> O in HNO <sub>3</sub> 0.5N	313175.1208	100 ml
			313175.1210	500 ml
<b>Calcium</b>	Ca	Ca(NO <sub>3</sub> ) <sub>2</sub> ·4H <sub>2</sub> O in HNO <sub>3</sub> 0.5N	313176.1208	100 ml
			313176.1210	500 ml
<b>Chromium</b>	Cr	Cr(NO <sub>3</sub> ) <sub>3</sub> ·9H <sub>2</sub> O in HNO <sub>3</sub> 0.5	313179.1208	100 ml
			313179.1210	500 ml
<b>Cobalt</b>	Co	Co(NO <sub>3</sub> ) <sub>2</sub> ·6H <sub>2</sub> O in HNO <sub>3</sub> 0.5N	313177.1210	500 ml
<b>Copper</b>	Cu	Cu(NO <sub>3</sub> ) <sub>2</sub> ·3H <sub>2</sub> O in HNO <sub>3</sub> 0.5N	313178.1208	100 ml
			313178.1210	500 ml
<b>Fluoride</b>	F	NaF in H <sub>2</sub> O	312682.1210	500 ml
<b>Gold</b>	Au	HAuCl <sub>4</sub> ·4H <sub>2</sub> O in HCl 1.2%	313672.1208	100 ml
<b>Iron</b>	Fe	Fe(NO <sub>3</sub> ) <sub>3</sub> ·9H <sub>2</sub> O in HNO <sub>3</sub> 0.5N	313182.1208	100 ml
			313182.1210	500 ml
<b>Lead</b>	Pb	Pb(NO <sub>3</sub> ) <sub>2</sub> in HNO <sub>3</sub> 0.5N	313189.1208	100 ml
			313189.1210	500 ml
<b>Lithium</b>	Li	LiCl in HCl ~1%	313183.1210	500 ml
<b>Magnesium</b>	Mg	Mg(NO <sub>3</sub> ) <sub>2</sub> ·6H <sub>2</sub> O in HNO <sub>3</sub> 0.5N	313184.1208	100 ml
			313184.1210	500 ml
<b>Manganese</b>	Mn	Mn(NO <sub>3</sub> ) <sub>2</sub> in HNO <sub>3</sub> 1N	313185.1208	100 ml
			313185.1210	500 ml
<b>Mercury</b>	Hg	Hg(NO <sub>3</sub> ) <sub>2</sub> in HNO <sub>3</sub> 0.5N	313186.1208	100 ml
			313186.1210	500 ml
<b>Molybdenum</b>	Mo	(NH <sub>4</sub> ) <sub>6</sub> Mo <sub>7</sub> O <sub>24</sub> ·4H <sub>2</sub> O in NH <sub>4</sub> OH 0.5N	314111.1208	100 ml
			314111.1210	500 ml
<b>Nickel</b>	Ni	Ni(NO <sub>3</sub> ) <sub>2</sub> ·6H <sub>2</sub> O in HNO <sub>3</sub> 0.5N	313187.1208	100 ml
			313187.1210	500 ml
<b>Potassium</b>	K	KCl in HCl ~1%	313190.1208	100 ml
			313190.1210	500 ml
<b>Selenium</b>	Se	SeO <sub>3</sub> H <sub>2</sub> in HNO <sub>3</sub> 1N	313191.1208	100 ml
			313191.1210	500 ml
<b>Silicon</b>	Si	Na <sub>2</sub> SiO <sub>3</sub> in H <sub>2</sub> O	312683.1208	100 ml
			312683.1210	500 ml
<b>Silver</b>	Ag	AgNO <sub>3</sub> in HNO <sub>3</sub> 0.5N	313188.1210	500 ml
<b>Sodium</b>	Na	NaCl in HCl ~1%	313192.1208	100 ml
			313192.1210	500 ml
<b>Tin</b>	Sn	SnCl <sub>4</sub> in HCl 5N	313180.1208	100 ml
			313180.1210	500 ml
<b>Titanium</b>	Ti	TiCl <sub>4</sub> in HCl 5N	313960.1208	100 ml
<b>Zinc</b>	Zn	Zn(NO <sub>3</sub> ) <sub>2</sub> ·6H <sub>2</sub> O in HNO <sub>3</sub> 0.5N	313193.1208	100 ml
			313193.1210	500 ml







## Inductively Coupled Plasma – Atomic Emission Spectrometry (ICP-AES) Mass Spectrometry (ICP-MS)

Inductively Coupled Plasma-Atomic Emission Spectroscopy (**ICP-AES**) also referred to as inductively coupled plasma-optical emission spectrometry (**ICP-OES**), is an **atomic emission spectrometry** method that uses an inductively coupled plasma (ICP) as the excitation source.

Inductively Coupled Plasma-Mass Spectroscopy (**ICP-MS**) is a **mass spectrometry** method that uses an inductively coupled plasma (ICP) as the excitation source.

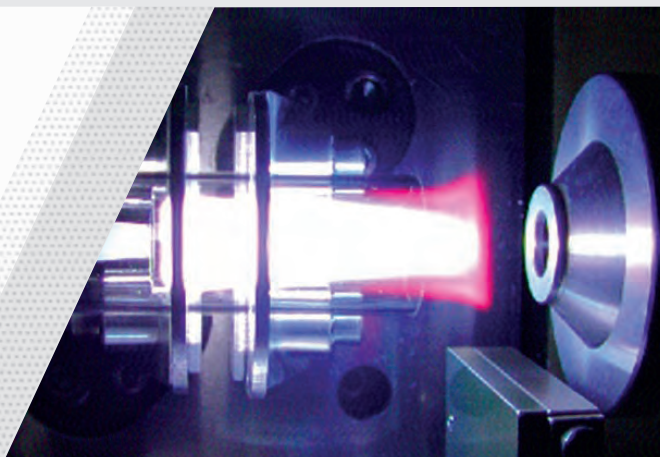


Image courtesy of Perkin Elmer

An ICP is a highly ionized inert gas (usually argon) with equal number of electrons and ions sustained by a radio-frequency (RF) field. The **high temperature** reached in the plasma (7000 °K), successively desolvates, vaporizes, excites (AES detection) and ionizes (MS detection) atoms from the sample.

### ICP-AES

ICP-AES exploits the fact that excited electrons emit energy at a given wavelength as they return to ground state after excitation. The intensity of the **energy emitted** at the chosen wavelength is proportional to the amount (concentration) of that element in the sample being analyzed.

Theoretical detection limit: approximately from 0.1 µg/L (0.1 ppb) to 50 µg/L (50 ppb) depending on the element and assuming there are no spectral interferences.

### ICP-MS

ICP-MS utilizes the ability of the ICP to generate charged ions from the element species within a sample. These ions are then directed into a mass spectrometer, which separates them according to their **mass-to-charge ratio** ( $m/z$ ). Most mass spectrometers have a quadrupole system or a magnetic sector. Ions of the selected mass/charge ratio are directed to a detector (channel electromultiplier, Faraday cup, dynodes), where ion currents are converted into electrical signals.

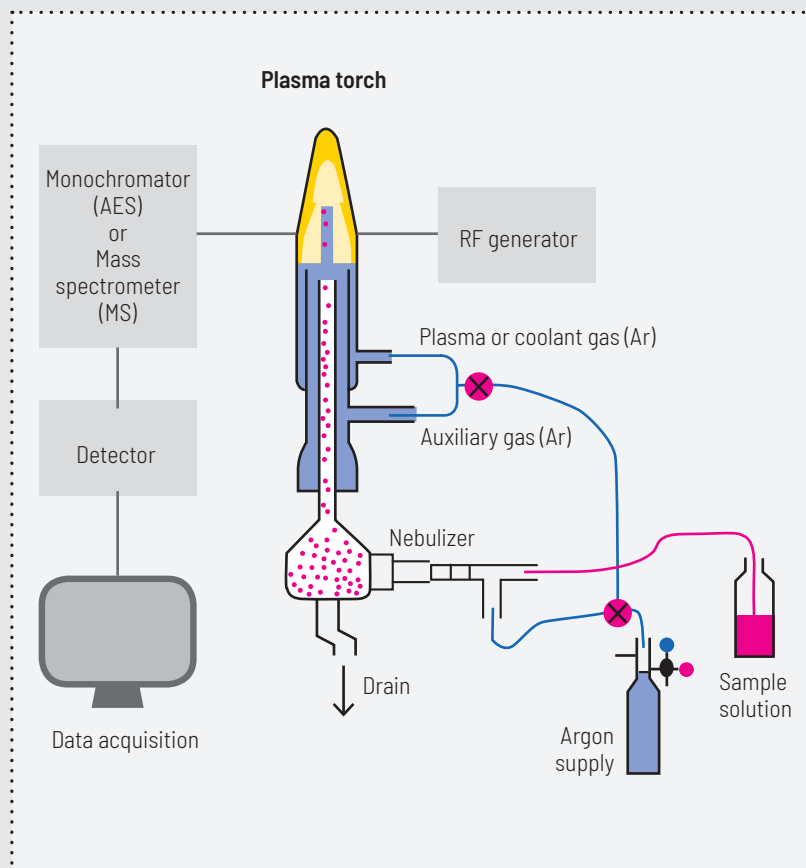
The element is quantified according to the number of ions arriving and generating electrical pulses per unit time.

Theoretical detection limit: approximately from 0.001 µg/L (1 ppt) to 0.01 µg/L (10 ppt) depending on the element and assuming there are no spectral interferences.

## Apparatus

The apparatus consists essentially of the following elements:

- Sample introduction system consisting of a peristaltic pump delivering the solution at constant flow rate into nebulizer
- Radio-frequency (RF) generator
- Plasma torch (ICP)
- Transfer optics focusing the image of the plasma at the entrance slit (ICP-AES)
- Wavelength dispersive devices consisting of diffraction gratings, prisms, filters or interferometers (ICP-AES)
- Interface region including cones to transport ions to the ion optics (ICP-MS)
- Mass spectrometer (ICP-MS)
- Detector
- Data acquisition unit



## Sample preparation

The sample preparation usually involves a step of digestion of the matrix by a suitable method, for example in a microwave oven.

Several-sample introduction systems tolerate high acid concentration, but the use of sulfuric ( $\text{H}_2\text{SO}_4$ ) and phosphoric acids ( $\text{H}_3\text{PO}_4$ ) can contribute to background emission. Therefore, nitric ( $\text{HNO}_3$ ) and hydrochloric acids ( $\text{HCl}$ ) are preferable.

The choice of acids used for the preservation of water samples and for the preparation of digested samples is very important. The acid to be used must have a very low concentration of metal in order not to contaminate the samples. Also the choice of the type of acid is very important to avoid:

- unwanted side reactions that can interfere in the analysis,
- formation of volatile compounds which are easily lost from the sample,
- damage to glass parts (special sample introduction system is necessary when using hydrofluoric acid).

Nitric ( $\text{HNO}_3$ ) and hydrochloric ( $\text{HCl}$ ) acids are typically used for the preparation of these samples, often in combination with each other to optimize digestion conditions.

Nitric acid and peroxide ( $\text{H}_2\text{O}_2$ ) are often used for organic matrices as the peroxide is an effective oxidizing agent and destroys the organic matrix.

In some cases hydrofluoric acid ( $\text{HF}$ ) will have to be added to destroy certain materials such as titanium dioxide or silica if they have been used in the final product.

# Reagents for Pharma Industry

## Chapter 2



PanReac AppliChem offers its ranges of **high purity reagents** for trace metal analysis at **ppb and ppt** levels in order to satisfy the high quality demand of reagents for trace analysis by **ICP-OES** in environmental, quality control and research laboratories.

### Acids for trace metal analysis (ppb)

This range consists of acetic, hydrochloric, nitric, sulfuric, hydrofluoric and perchloric acids in 500 mL, 1000 mL and 2.5 L high density polyethylene (HDPE) bottles.

Main advantages:

- In most cases, a metal impurity content of less than 0.1 ppb ( $\mu\text{g/L}$ ) is guaranteed for 60 different elements.

Compared to glass bottles:

- Low level of metal impurities leaching from the polyethylene.
- Light-weight bottles for easier handling and greater safety against breakage in the laboratory.

Product name	Code	Package
<b>Acetic Acid glacial</b>	721008.0011	1000 ml
	721019.0010	500 ml
<b>Hydrochloric Acid 35%</b>	721019.0011	1000 ml
	721019.0012	2.5 L
<b>Hydrofluoric Acid 48%</b>	721028.0010	500 ml
<b>Nitric Acid 69%</b>	721037.0010	500 ml
	721037.0011	1000 ml
	721037.0012	2.5 L
<b>Perchloric Acid 70%</b>	722175.0010	500 ml
	722175.0011	1000 ml
<b>Sulfuric Acid 93-98%</b>	721058.0010	500 ml
	721058.0011	1000 ml



### Acids for trace metal analysis (ppt)

These reagents are **specially purified** by multiple distillation to achieve the **lowest impurity levels**, obtaining products that can be used as a baseline for the analysis of trace metals. The greatest challenge has always been to minimize the impurities of some elements like Fe, Zn, Ni and Cu with a special analytical importance and to limit the presence of elements such as Ca, Na and Al which are ubiquitous in the environment as well as in the container materials. Thanks to this special care in the manufacturing, this range is able to offer reagents with concentrations of the above elements lower than 100 ppt ( $\text{ng/L}$ ) and most lower than 10 ppt ( $\text{ng/L}$ ). It represents the range of reagents with the lowest metal content intended for trace metal analysis. Our reagents for trace metal analysis (ppt) are available in the following sizes in both Teflon PFA and FEP: 250 mL and 500 mL. Water is available in specially washed HDPE bottles.



### Main advantages

- Our reagents for trace metal analysis (ppt) include the analysis of more than 60 metals at parts per trillion levels providing the best level of purity available on the market.
- Manufactured by sub-boiling distillation.
- The concentration level for the majority of metals is below 10 ppt, being all below 100 ppt.
- Batch-to-batch homogeneity for more reproducible results.
- Manufactured and packaged in a clean room, class 10, free of contaminants to guarantee the highest levels of purity.
- Stored in specially selected teflon bottles. The material is controlled prior to the bottle manufacture. Every bottle is leached with hot acid for two weeks in order to eliminate any contamination material due to metal traces.

Product name	Code	Package
<b>Hydrochloric Acid 35%</b>	711019.0009	250 ml
	711019.0010	500 ml
<b>Hydrofluoric Acid 48%</b>	711028.0010	500 ml
<b>Hydrogen Peroxide 30% w/w</b>	716323.0010	500 ml
<b>Nitric Acid 69%</b>	711037.0009	250 ml
	711037.0010	500 ml
<b>Water</b>	711074.0011	1000 ml



### ICP Standards

For the calibration of the ICP equipment, PanReac AppliChem offers a range of ICP element standards that meet the highest purity and quality requirements demanded by these highly sensitive techniques.

The range of ICP element standards consists of 72 elements in concentrations ranging from 1.000 to 10.000 g/L.

The concentration of each element is strictly controlled to an accuracy of  $\pm 0.2\%$  of the true value, with NIST traceability.

The traces of 69 elements are analyzed by ICP and the result is specified on the certificate of analysis.

The preparation is carried out using raw materials of the highest available purity: 99.999% for metals as well as ultra-pure water and acids. The ICP standards are available in pre-washed 100-mL PE bottles, all of which are supplied with their corresponding detailed certificate of analysis. This certificate includes the composition of the standard, its date of manufacture, the minimum expiry date, the actual value with respect to the guaranteed value, traceability and the concentration of trace impurities.














### Single Element Standards





Product name	Element	Composition	Code (1 g/L)	Code (10 g/L)	Package
<b>Aluminium</b>	Al	Al in 2-5% HNO <sub>3</sub>	766034.1208	775943.1208	100 ml
<b>Antimony</b>	Sb	Sb in 2-5% HNO <sub>3</sub>	766035.1208	-	100 ml
<b>Arsenic</b>	As	As <sub>2</sub> O <sub>3</sub> in 2-5% HNO <sub>3</sub>	766036.1208	-	100 ml
<b>Barium</b>	Ba	BaCO <sub>3</sub> in 2-5% HNO <sub>3</sub>	766037.1208	-	100 ml
<b>Boron</b>	B	H <sub>3</sub> BO <sub>3</sub> in H <sub>2</sub> O	765900.1208	-	100 ml
<b>Cadmium</b>	Cd	Cd in 2-5% HNO <sub>3</sub>	766038.1208	-	100 ml
<b>Calcium</b>	Ca	CaCO <sub>3</sub> in 2-5% HNO <sub>3</sub>	766040.1208	775950.1208	100 ml
<b>Chloride</b>	Cl	KCl in H <sub>2</sub> O	765903.1208	-	100 ml
<b>Chromium</b>	Cr	Cr in 2-5% HNO <sub>3</sub>	766043.1208	-	100 ml
<b>Cobalt</b>	Co	Co in 2-5% HNO <sub>3</sub>	766041.1208	-	100 ml
<b>Copper</b>	Cu	Cu in 2-5% HNO <sub>3</sub>	766042.1208	-	100 ml
<b>Germanium</b>	Ge	Ge in 2-5% HNO <sub>3</sub> + traces HF	765910.1208	-	100 ml
<b>Gold</b>	Au	Au in 20% HCl	766061.1208	-	100 ml
<b>Iron</b>	Fe	Fe in 2-5% HNO <sub>3</sub>	766049.1208	775967.1208	100 ml
<b>Lead</b>	Pb	Pb in 2-5% HNO <sub>3</sub>	766063.1208	-	100 ml
<b>Magnesium</b>	Mg	Mg in 2-5% HNO <sub>3</sub>	766051.1208	775976.1208	100 ml
<b>Manganese</b>	Mn	Mn in 2-5% HNO <sub>3</sub>	766052.1208	-	100 ml
<b>Mercury</b>	Hg	Hg in 2-5% HNO <sub>3</sub>	766060.1208	-	100 ml
<b>Molybdenum</b>	Mo	Mo in 2-5% HNO <sub>3</sub> + traces HF	766053.1208	-	100 ml
<b>Nickel</b>	Ni	Ni in 2-5% HNO <sub>3</sub>	766054.1208	-	100 ml
<b>Nitrogen</b>	N	(NH <sub>4</sub> ) <sub>2</sub> SO <sub>4</sub> in H <sub>2</sub> O	765920.1208	-	100 ml
<b>Palladium</b>	Pd	Pd in 20% HCl	765922.1208	-	100 ml
<b>Phosphorus</b>	P	H <sub>3</sub> PO <sub>4</sub> in H <sub>2</sub> O	766384.1208	775907.1208	100 ml
<b>Potassium</b>	K	KNO <sub>3</sub> in 2-5% HNO <sub>3</sub>	766050.1208	775989.1208	100 ml
<b>Rhodium</b>	Rh	RhCl <sub>3</sub> ·3H <sub>2</sub> O in 20% HCl	765926.1208	-	100 ml
<b>Scandium</b>	Sc	Sc <sub>2</sub> O <sub>3</sub> in 2-5% HNO <sub>3</sub>	765930.1208	-	100 ml
<b>Selenium</b>	Se	Se in 2-5% HNO <sub>3</sub>	766055.1208	-	100 ml
<b>Silicon</b>	Si	(NH <sub>4</sub> ) <sub>2</sub> SiF <sub>6</sub> in 5% HF	765997.1208	-	100 ml
<b>Silver</b>	Ag	Ag in 2-5% HNO <sub>3</sub>	766062.1208	-	100 ml

>>








<<

Product name	Element	Composition	Code (1 g/L)	Code (10 g/L)	Package
<b>Sodium</b>	Na	NaNO <sub>3</sub> in 2-5% HNO <sub>3</sub>	766056.1208	775999.1208	 100 ml
<b>Sulphur</b>	S	H <sub>2</sub> SO <sub>4</sub> in H <sub>2</sub> O	765898.1208	775946.1208	 100 ml
<b>Tin</b>	Sn	Sn in 20% HCl	766047.1208	-	 100 ml
<b>Titanium</b>	Ti	Ti in 5% HF	766057.1208	-	 100 ml
<b>Tungsten</b>	W	W in 5% HF	765939.1208	-	 100 ml
<b>Vanadium</b>	V	V in 2-5% HNO <sub>3</sub>	765938.1208	-	 100 ml
<b>Yttrium</b>	Y	Y <sub>2</sub> O <sub>3</sub> in 2-5% HNO <sub>3</sub>	765915.1208	-	 100 ml
<b>Zinc</b>	Zn	Zn in 2-5% HNO <sub>3</sub>	766058.1208	-	 100 ml
<b>Zirconium</b>	Zr	Zr in 5% HF	765940.1208	-	 100 ml

## Multi Element Standards

Product name	Elements	Matrix	Code	Package
<b>Standard solution containing 9 elements</b>	As, Be, Cd, Cr, Pb, Hg, Ni, Se, Tl (100 mg/L each)	10% HNO <sub>3</sub>	766334.1208	 100 ml
<b>Standard solution containing 21 elements</b>	As, Be, Ca, Cd, Co, Cr, Cu, Fe, Li, Mg, Mn, Mo, Ni, Pb, Sb, Se, Sr, Ti, Tl, V, Zn (100 mg/L each)	5% HNO <sub>3</sub>	766335.1208	 100 ml
<b>Standard solution containing 24 elements</b>	Al, B, Ba, Be, Bi, Ca, Cd, Co, Cr, Cu, Fe, Ga, K, Li, Mg, Mn, Na, Ni, Pb, Se, Sr, Te, Tl, Zn (100 mg/L each)	2% HNO <sub>3</sub>	766333.1208	 100 ml
<b>Standard solution containing 23 elements</b>	Ag, Al, B, Ba, Bi, Ca, Cd, Co, Cr, Cu, Fe, Ga, In, K, Li, Mg, Mn, Na, Ni, Pb, Sr, Tl, Zn (1000 mg/L each)	2% HNO <sub>3</sub>	766332.1208	 100 ml

## Package pictograms

	Glass bottle		Aluminium bottle
	Plastic bottle		Glass ampoule
	Plastic jerrycan		Fluoropolymer bottle
	Stainless steel drum (returnable)		