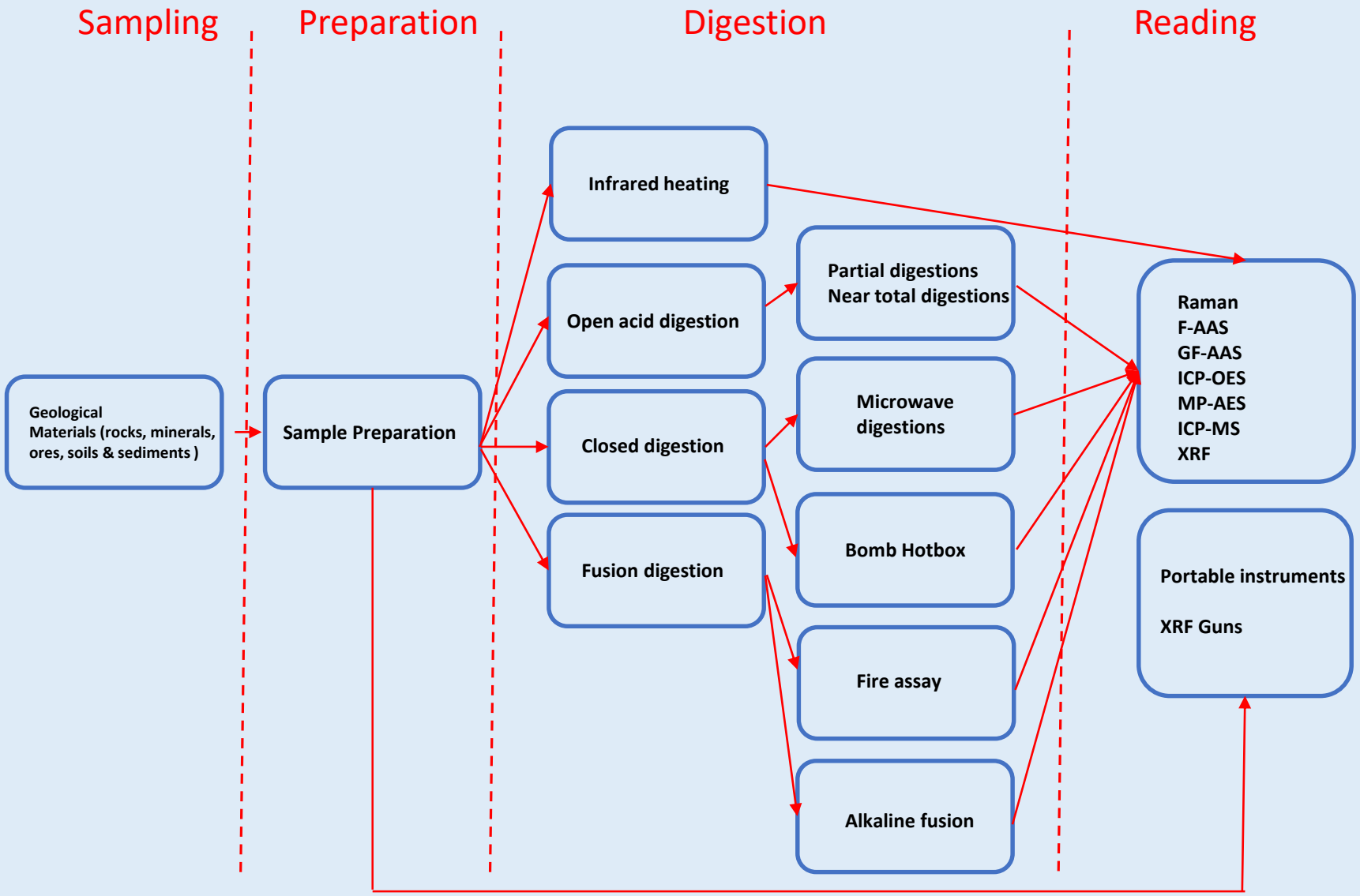
ZARAZMA

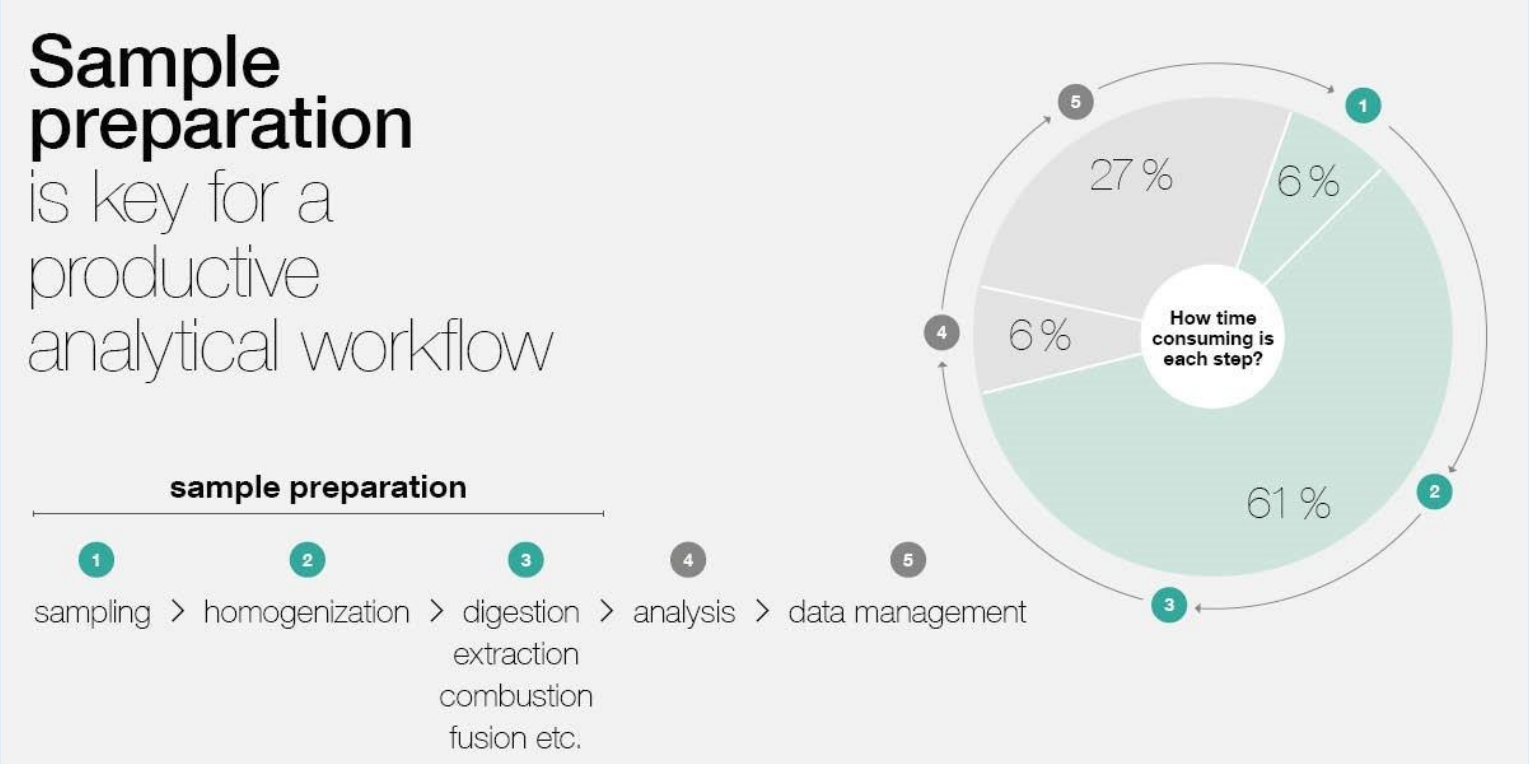
Microwave Digestion AuME+

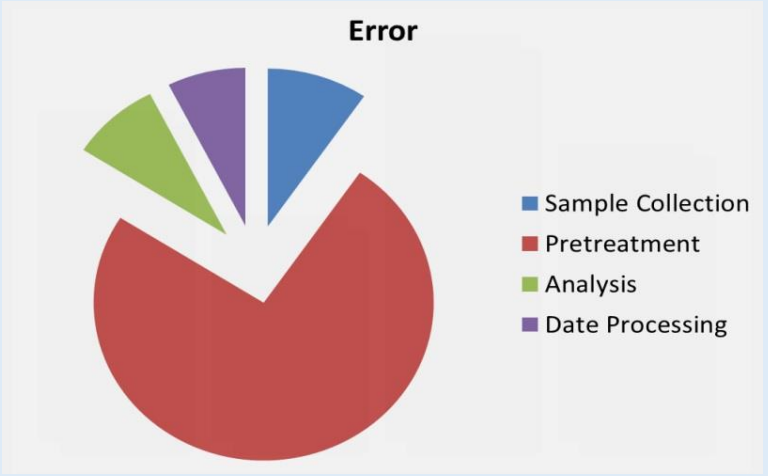
Introduce the new method for analysis of gold and paragenesis.

Steps of analysis

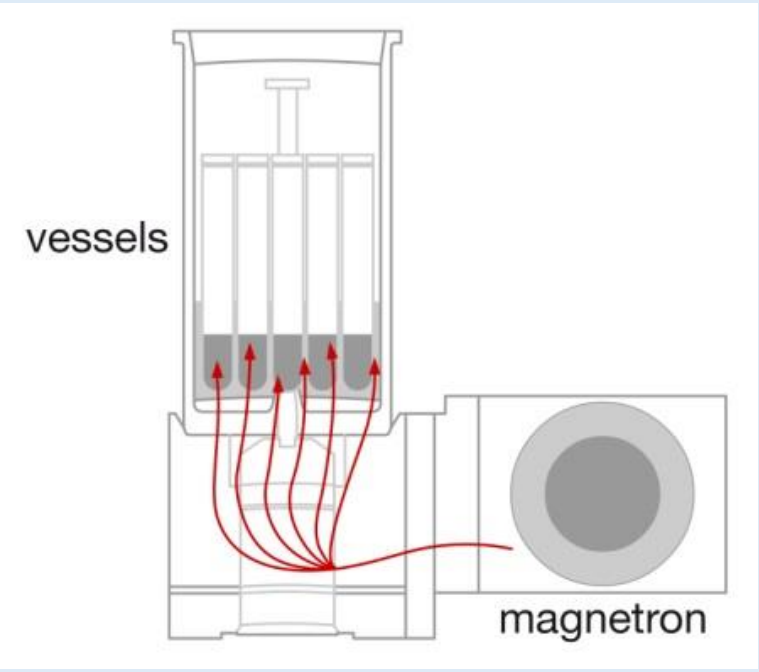


Despite many advances in recent years, sample preparation is still often considered to be the bottleneck in the analytical workflow since it takes up more than 60 % of the time needed from sampling to the end result



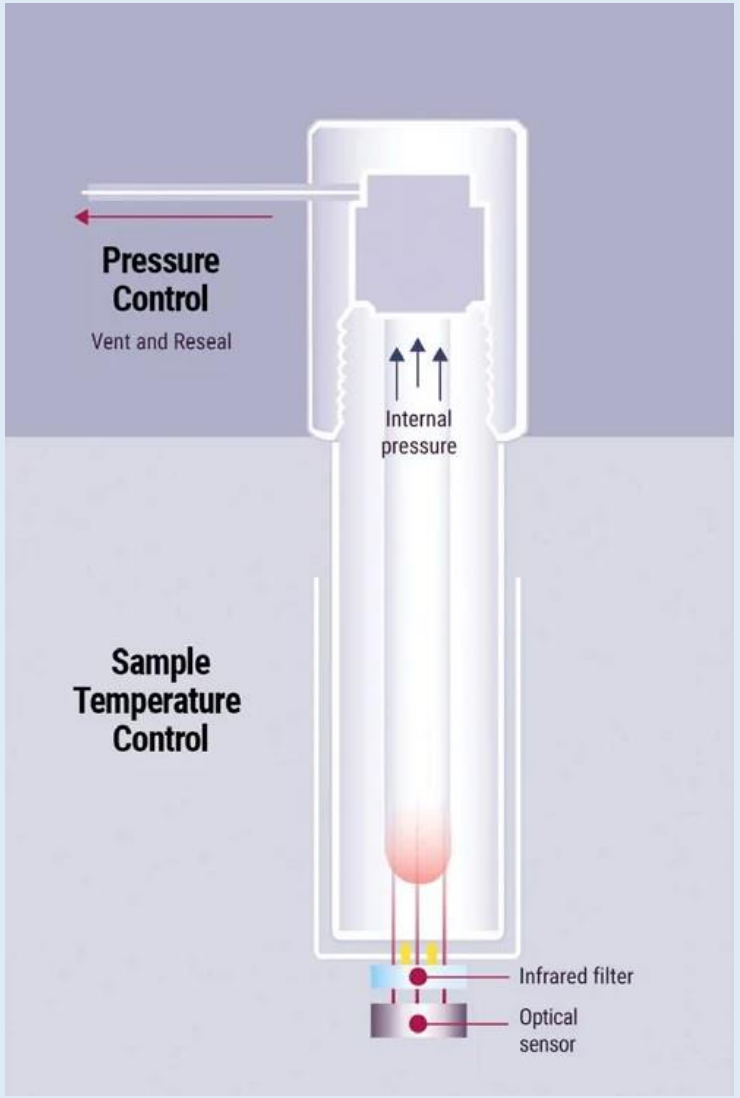


Microwave instrument parts



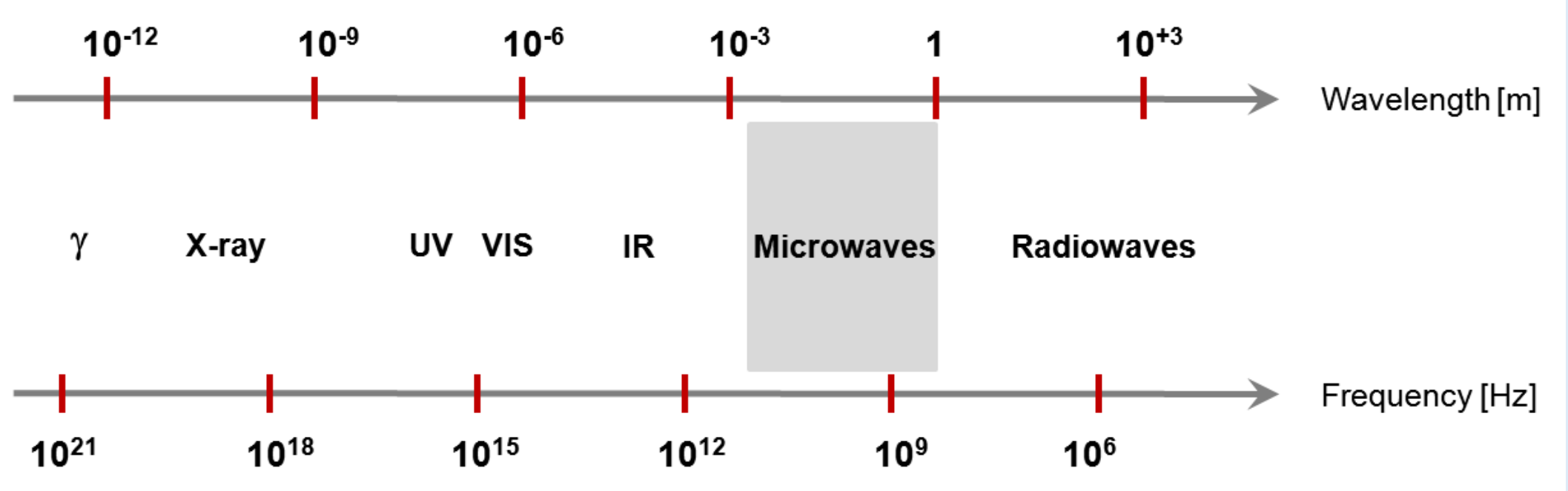
Tubes and rotors

Tubes and rotors

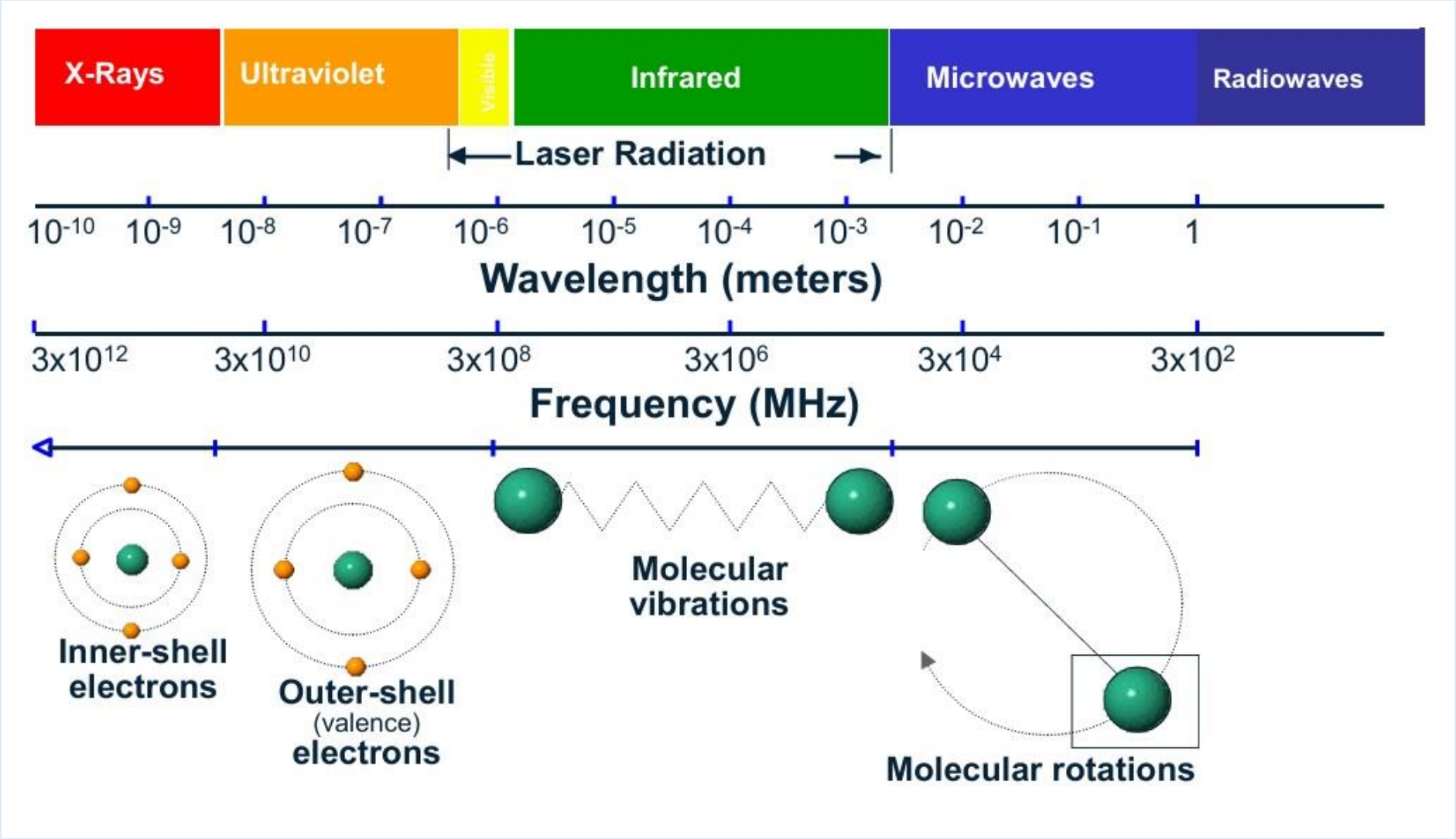


Wavelength

Microwaves are electromagnetic waves in the range of 300 MHz (0.3 GHz) to 300 GHz



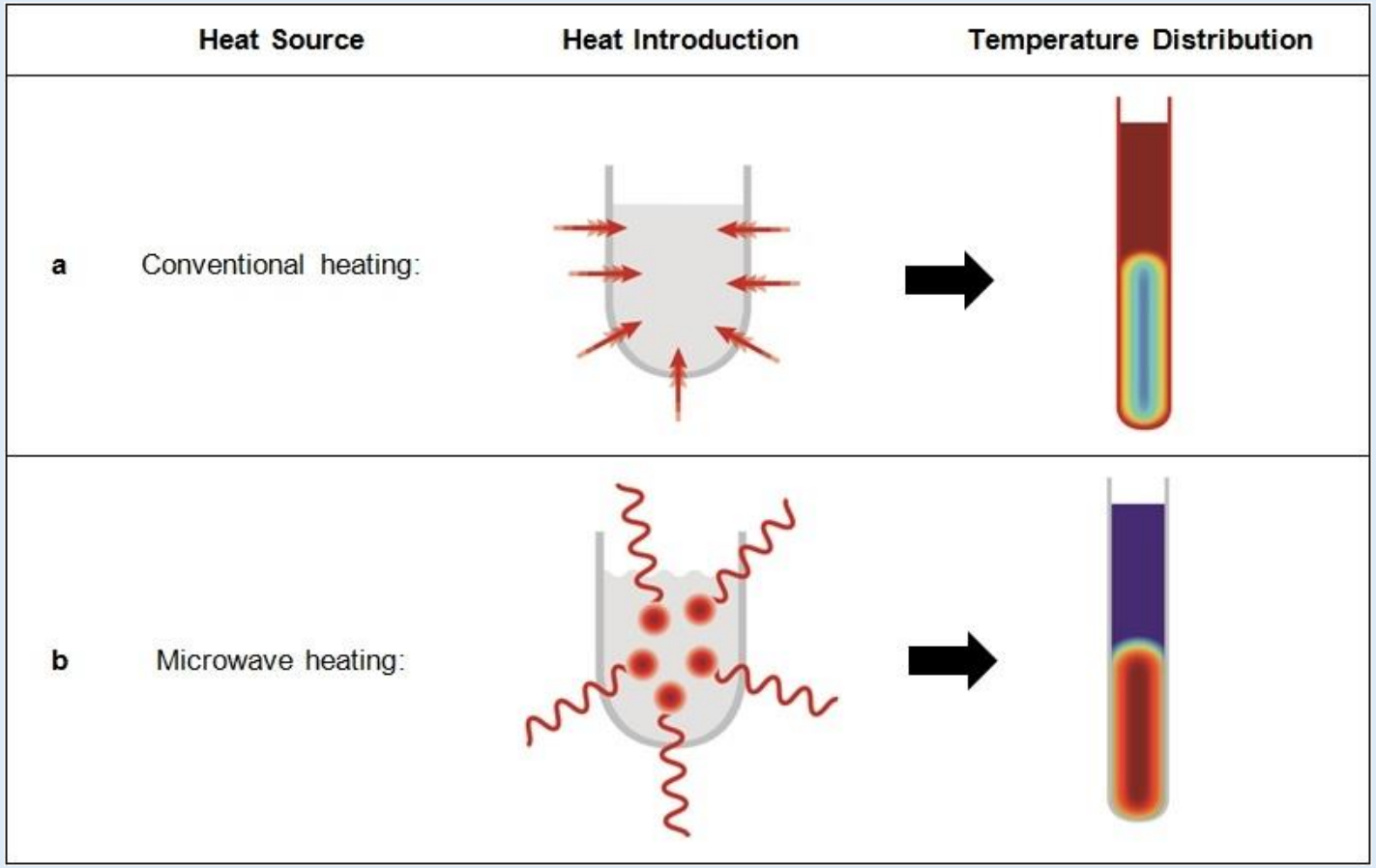
Wavelength


X-Rays
Ultraviolet
Visible
Infrared
Microwaves
Radiowaves
Laser Radiation
10-10 10-9 10-8 10-7 10-6 10-5 10-4 10-3 10-2 10-1 1
Wavelength (meters)
3x1012 3x1010 3x108 3x106 3x104 3x102
Frequency (MHz)
Inner-shell electrons
Outer-shell (valence) electrons
Molecular vibrations
Molecular rotations

What is the difference with microwave digestion for sample preparation?

- Fast heating rates
- Instant turn on and turn off
- No contact to heating core required
- High temperatures and pressures can be reached
- Rapid preparation sample
- Closed vessel

- Fast heating rates
- Instant turn on and turn off
- No contact to heating core required



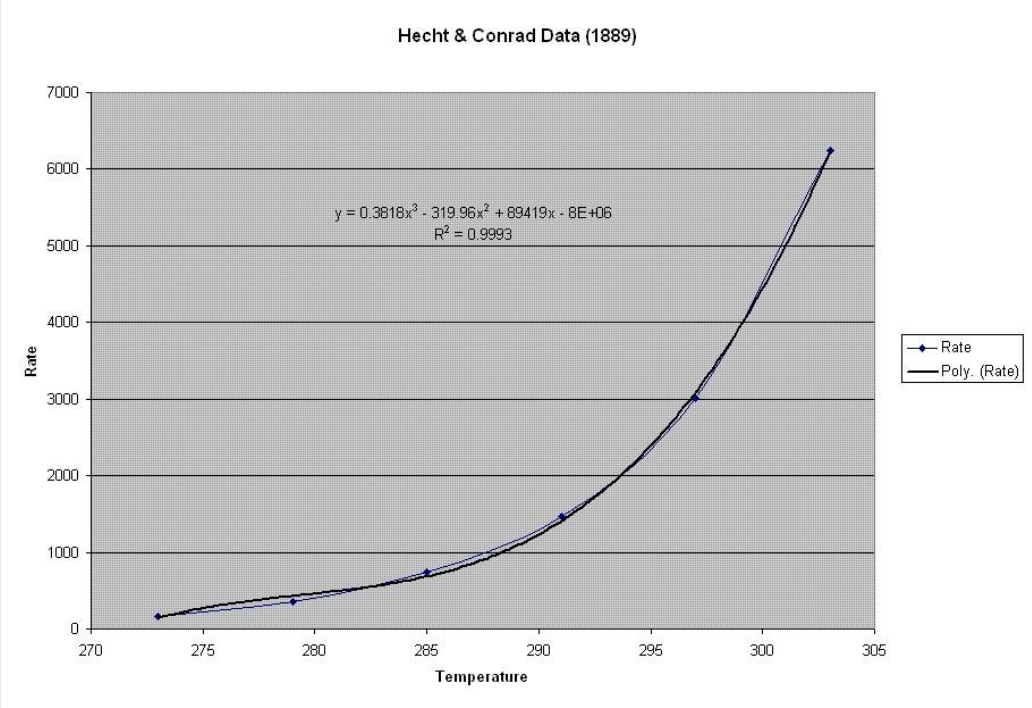
Simplified schemes of the heat distribution in a conventionally heated reaction mixture (a) and in a microwave heated reaction mixture (b).

- <u>High temperatures and pressures can be reached</u>
- Rapid preparation sample

Arrhenius Equation: $k = A \cdot e^{\frac{-E_a}{RT}}$

Rule of thumb:
10 °C temperature increase doubles the reaction rate

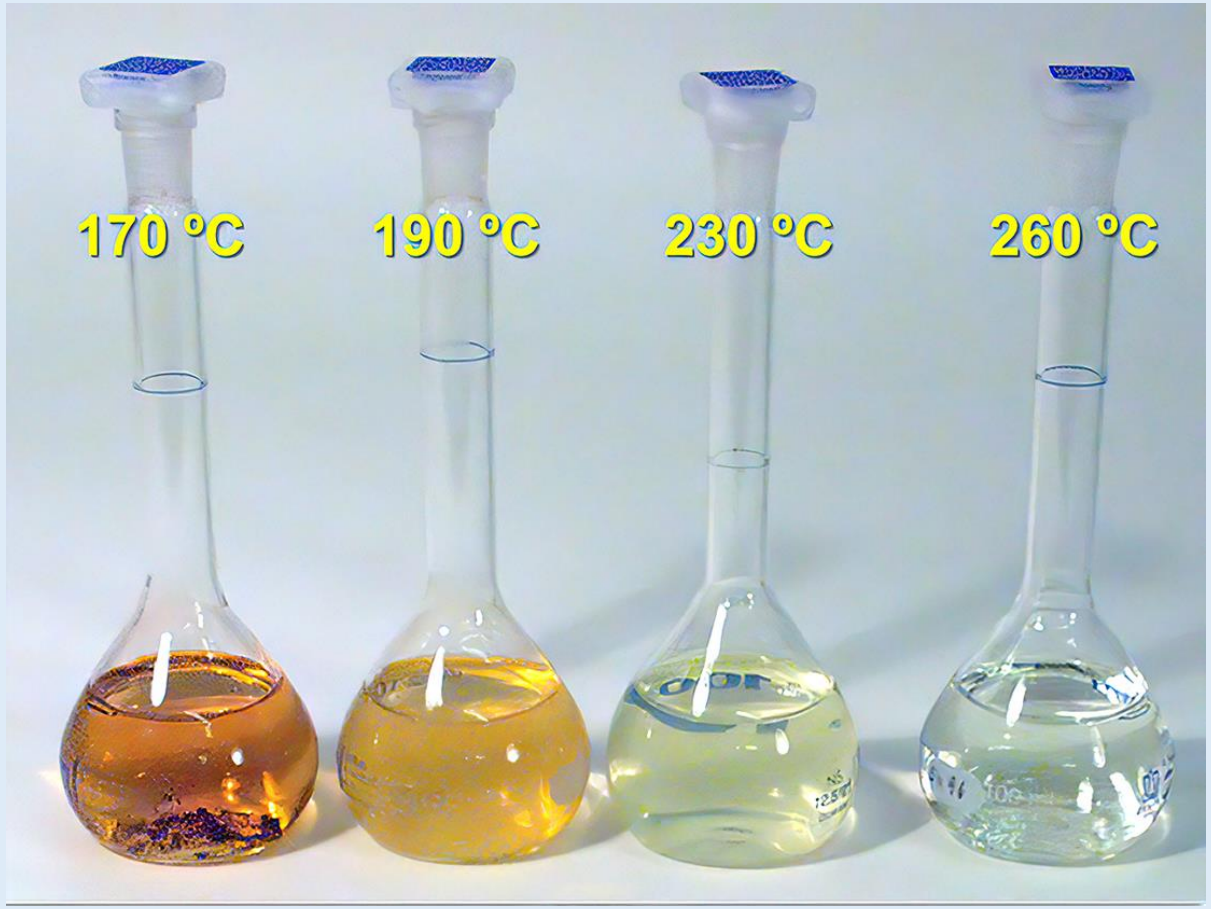
100 °C	110 °C	120 °C	130 °C	140 °C	150 °C	160 °C	170 °C	180 °C
8 h	4 h	2 h	1 h	30 min	15 min	8 min	4 min	2 min



- **Temperature on digestion**

	t_1（h）	Digestion Solution	t_2（min）
Biological Samples	6	HNO3/HCl，HNO3/HF	10～30
Environmental samples	6	HNO3, HCl	20
Al2O3	24	H2SO4,H3PO4	40
Irons	1	HCl, HF, HNO3	5
Soil	8	HNO3, HF	20
Waste Water	4	HNO3	10

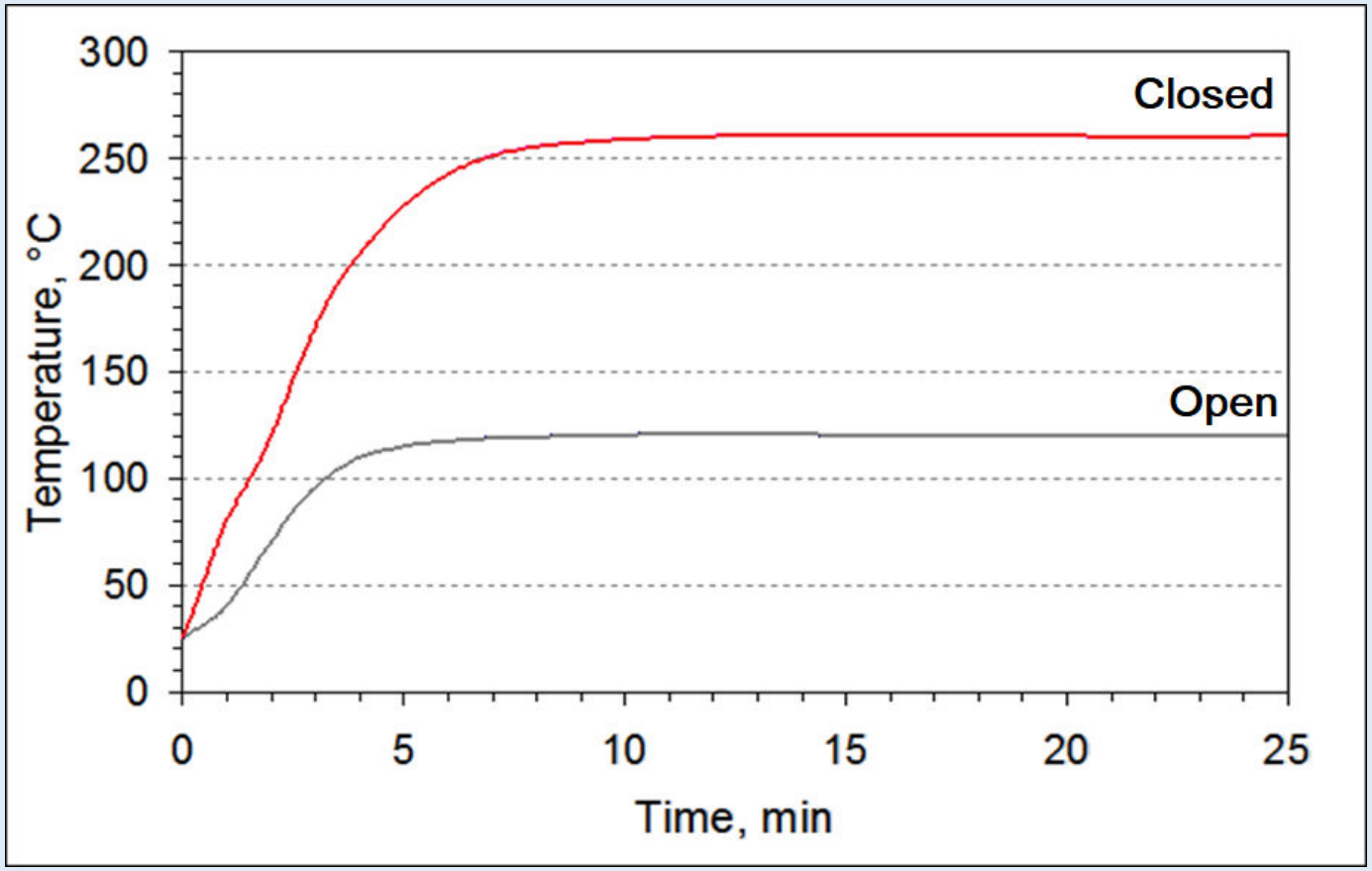
- Temperature on digestion



Kinds of acids in microwave digestion?

- Nitric acid, HNO_3 (65 %)
- Hydrochloric acid, HCl (30 % to 37 %)
- Hydrofluoric acid, HF (40 % to 48 %)
- Sulfuric acid, H_2SO_4 (95 % to 98 %)
- Perchloric acid, $HClO_4$ (70 % to 72 %)
- Phosphoric acid, H_3PO_4 (85 %)
- Hydrogen peroxide, H_2O_2 (30 %)
- Aqua regia, HCl + HNO_3 (volume ratio 3:1)
- Reverse aqua regia, HCl + HNO_3 (volume ratio 1:3)
- Boric acid, H_3BO_3 (approx. 5 %)

- Closed Vessel



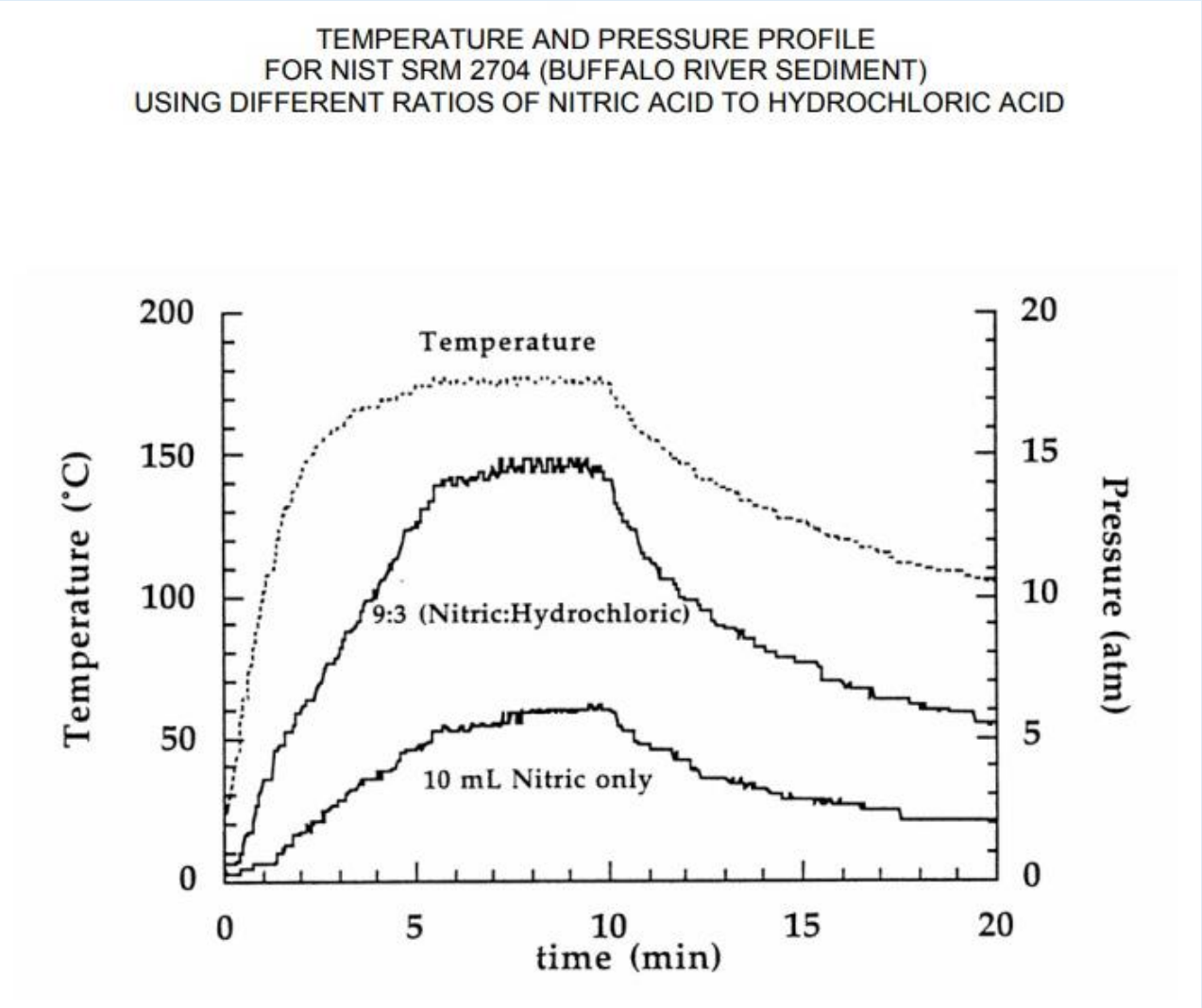
Nitric acid heated under open-vessel (reflux heating) and under closed-vessel conditions

🚫 Common Volatile / Partially Los Elements

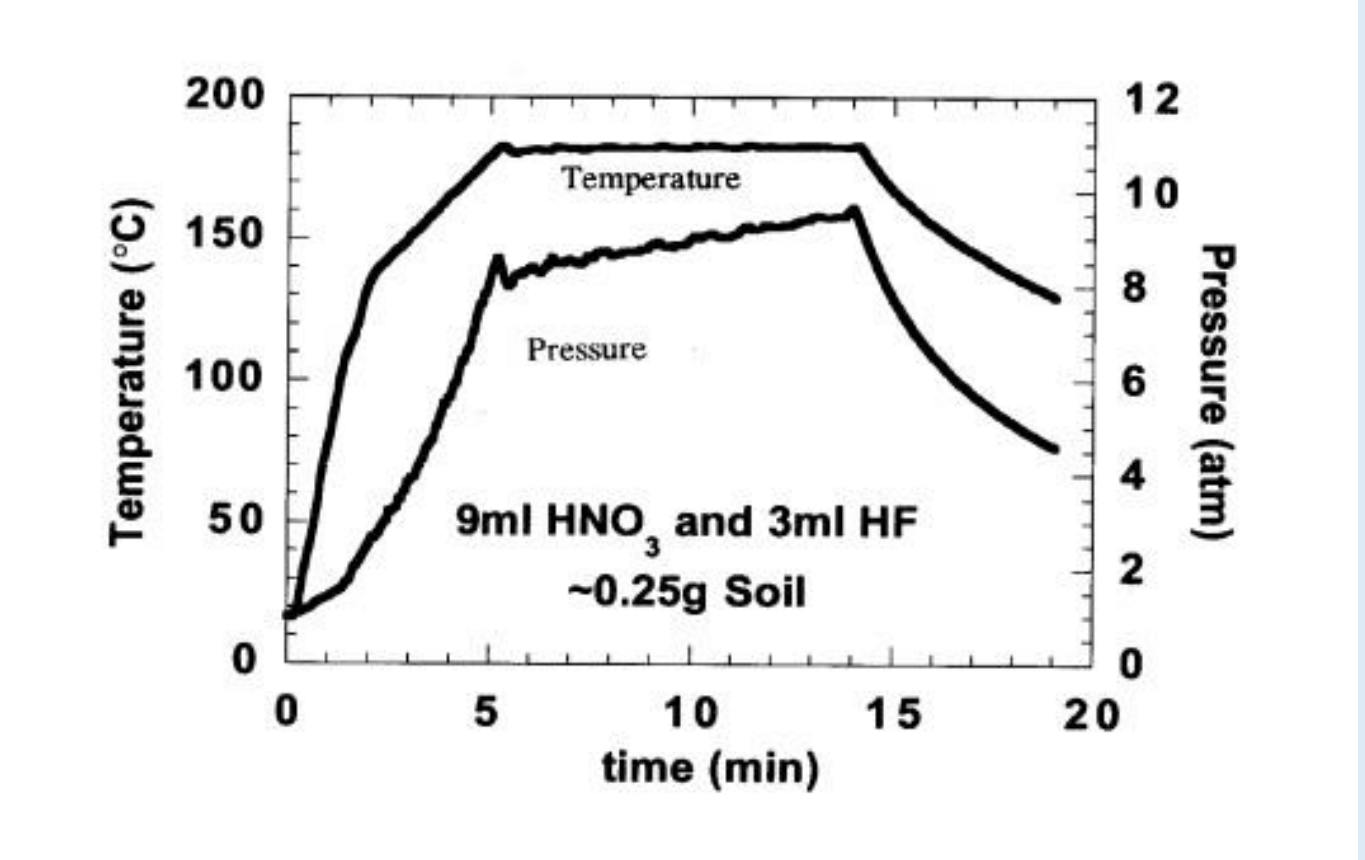
The elements that may be **lost (volatile)** four-acid digestion include:

Element	Reason for Loss
As (Arsenic)	Forms volatile $AsCl_3$
Sb (Antimony)	Forms volatile $SbCl_3$
Se (Selenium)	Forms volatile oxides (SeO_2)
Te (Tellurium)	Forms volatile halides/ oxides
Hg (Mercury)	Very volatile; easily lost
S (Sulfur)	Volatile as SO_2, SO_3
Br, Cl, I (Halogens)	Lost as gases
Cd, Pb (minor)	Slight losses possible a high temperature

The effects of T, P & Acids



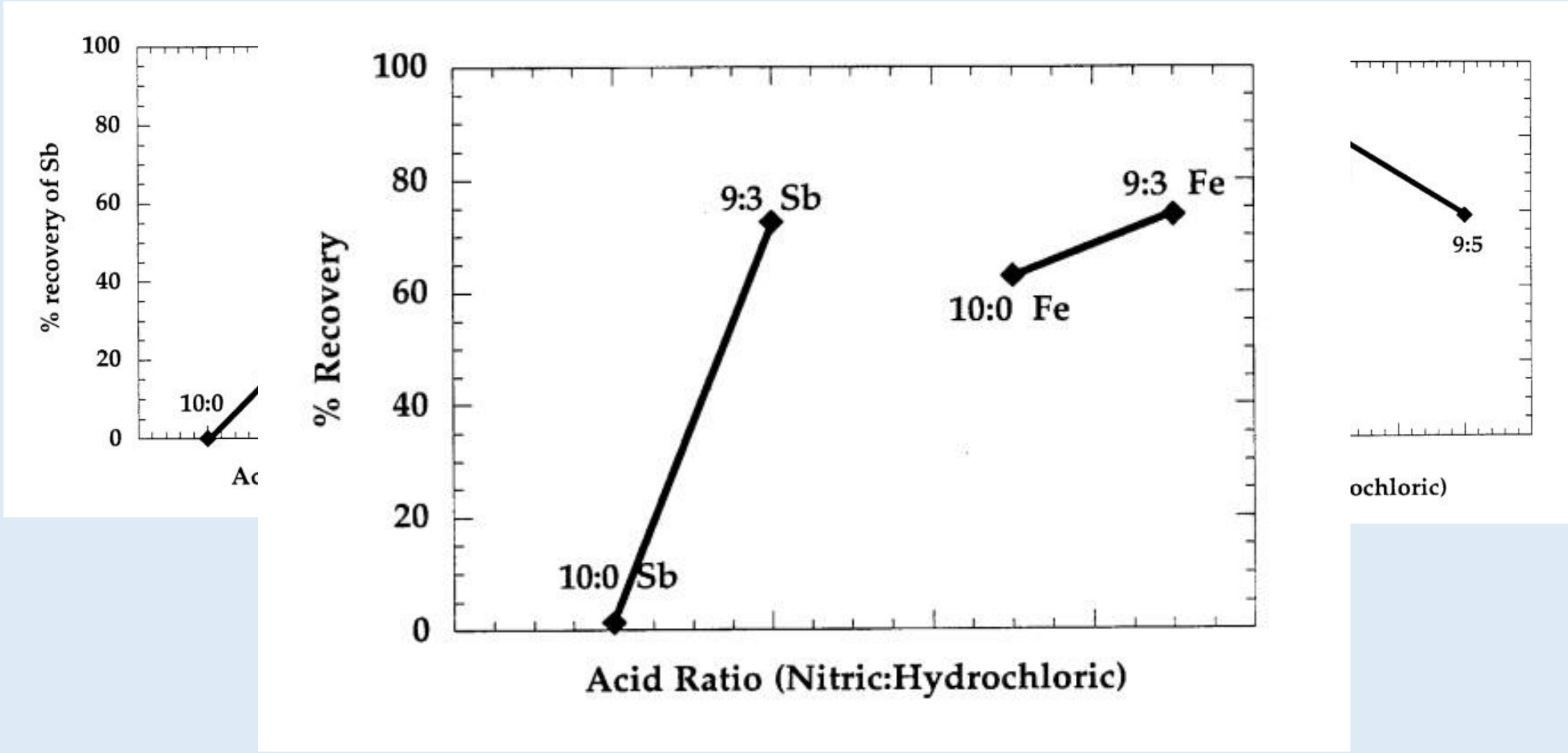
The effects of T, P & Acids



TYPICAL REACTION PROFILE FOR THE DIGESTION OF A SOIL

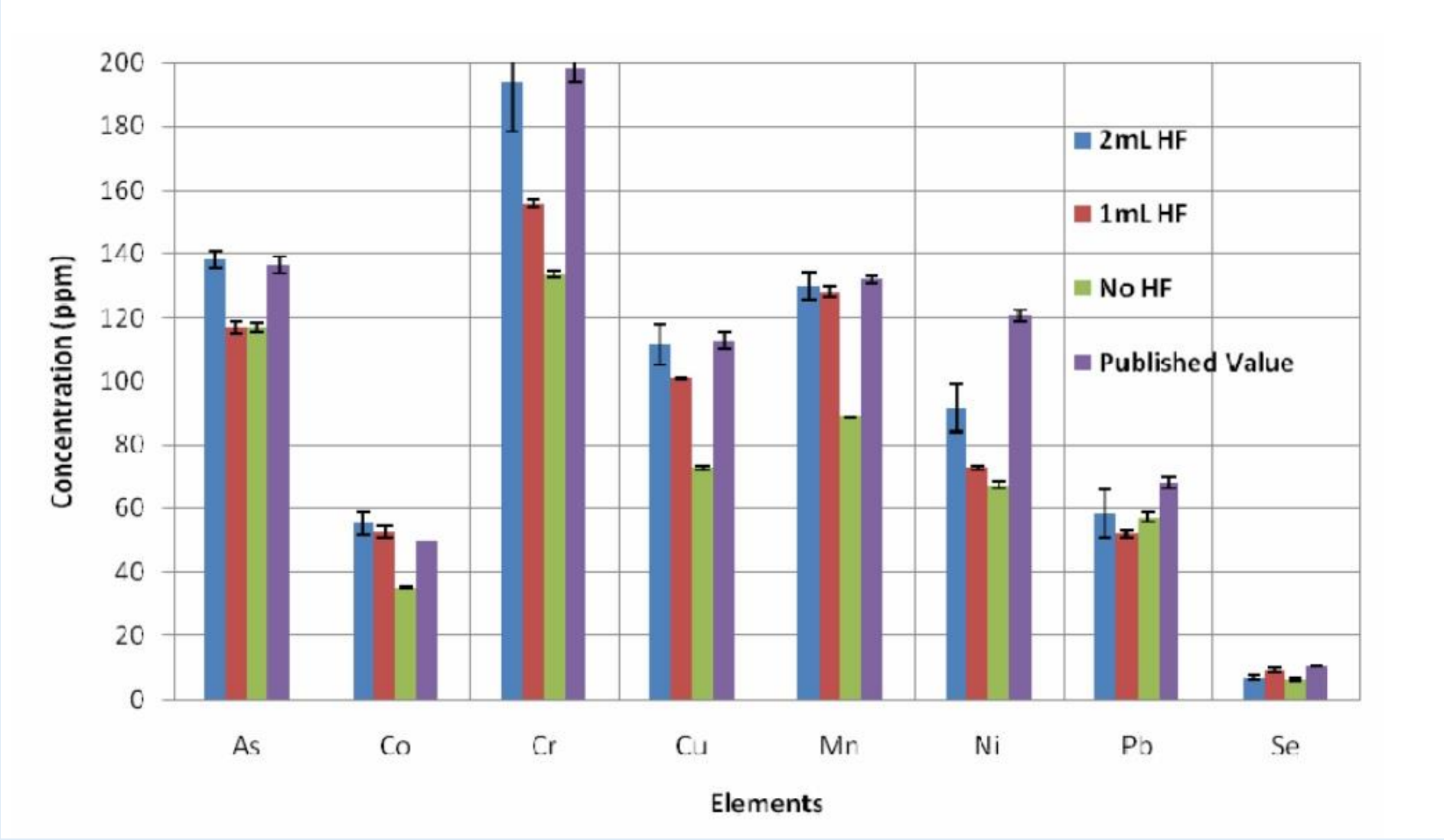
Recovery & Reproducibility

(Method development)



Recovery & Reproducibility

(Method development)

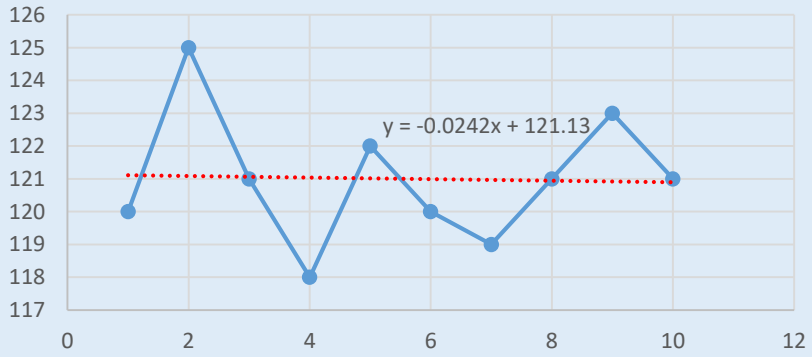


Elements of AuME+

AuME+ Method								
Element	**Au**	**Ag**	**As**	**Bi**	**Cd**	**Cu**	**Hg**	**Mo**
Unit	ppb	ppm	ppm	ppm	ppm	ppm	ppm	ppm
DL	20	0.50	0.5	1	0.10	1	1	0.5
UL	500,000	5,000	50,000	1,000	5,000	150,000	1,000	30,000
Method	AuME+	AuME+	AuME+	AuME+	AuME+	AuME+	AuME+	AuME+

Element	**Pb**	**S**	**Sb**	**Se**	**Te**	**Tl**	**W**	**Zn**
Unit	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm
DL	1	50	0.50	1	0.5	0.5	0.5	1
UL	150,000	300,000	5,000	1,000	1,000	1,000	1,000	150,000
Method	AuME+	AuME+	AuME+	AuME+	AuME+	AuME+	AuME+	AuME+

%CV = 1.65



Advantages of AuME+

- **Speed**: Complete digestion is achieved in minutes to about an hour, compared to several hours needed for traditional methods.
- **Enhanced Recovery**: Closed-vessel digestion minimizes the loss of volatile elements (e.g., mercury, arsenic).
- **High Reproducibility**: Automated temperature and pressure control ensure consistent digestion conditions across samples.
- **Improved Safety & reduce systematic errors**: Enclosed vessels and sophisticated monitoring systems reduce the risk of acid exposure and accidents also automatic procedure due to programming digestion cause to reduce the risk of systematic errors.
- **Low Acid Consumption**: Efficient digestion often requires smaller volumes of acid, reducing chemical costs and waste.
- **Handling of Difficult Samples**: Capable of digesting tough matrices such as soils, biological tissues, polymers, and industrial materials (High pressure).
- **Gold & Paragensis:** Possibility of measuring gold and associated elements in a single package.
- **Mercury detect:** Detecting Mercury as critical element for gold.
- **Cost:** competitive price

Limits:

- **High Detection limit:** compare to fireassay (1-5 ppb), it is about 20 ppb
- **Small sample weight:** below 5 gr needed for analyze.

Compare to Std methods in the ASTM.

EPA 3051a

Element		CAS Registry No. [a]
Lead	(Pb)	7439-92-1
*Magnesium	(Mg)	7439-95-4
Manganese	(Mn)	7439-96-5
Mercury	(Hg)	7439-97-6
Molybdenum	(Mo)	7439-98-7
Nickel	(Ni)	7440-02-0
Potassium	(K)	7440-09-7
Selenium	(Se)	7782-49-2
*Silver	(Ag)	7440-22-4
Sodium	(Na)	7440-23-5
Strontium	(Sr)	7440-24-6
Thallium	(Tl)	7440-28-0
*Vanadium	(V)	7440-62-2
Zinc	(Zn)	7440-66-6

EPA 3052

Element		CAS Registry No. [a]
*Aluminum	(Al)	7429-90-5
*Antimony	(Sb)	7440-36-0
Arsenic	(As)	7440-38-2
*Barium	(Ba)	7440-39-3
*Beryllium	(Be)	7440-41-7
Boron	(B)	7440-42-8
Cadmium	(Cd)	7440-43-9
Calcium	(Ca)	7440-70-2
*Chromium	(Cr)	7440-47-3
Cobalt	(Co)	7440-48-4
Copper	(Cu)	7440-50-8
*Iron	(Fe)	7439-89-6

Upper limits (UL):

Element	Ag	As	Cu	Fe	Mo	Pb	S	Sb	Zn	Hg	Au
Unit	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	20
DL	0.5	0.5	1	100	0.5	1	50	0.5	1	1	ppb
Test 1	4.10	14.90	10,931	83,540	<0.5	214	19,000	0.90	759	<1	< 20
CRM-1	3.95	15.80	10,629	79,286	1.07	203	16,704	< 1	717	< 0.13	< 10
Test 2	0.80	20.30	3,282	51,744	3.70	25	374	0.95	99	<1	59.00
CRM-2	0.73	20.40	3,150	49,755	3.85	23	360	1.52	89	< 0.05	50.75
Test 3	67.50	80.00	3,826	35,999	15.10	13,792	46,892	114.20	52,217	3.70	1,279
CRM-3	68.05	75.00	3,661	34,282	13.32	13,589	45,019	106.62	51,721	3.93	1,228
Test 4	1.20	23.70	6,615	70,599	6.90	29	888	0.80	118	<1	132
CRM-4	1.13	28.10	6,248	66,021	6.98	24	815	1.79	100	-	119
Test 5	1.40	39.80	7,979	37,660	265.60	65	13,400	3.01	323	<1	480
CRM-5	1.70	38.80	7,780	35,250	239.96	66	11,500	3.39	298	0.06	499
Test 6	1.50	33.50	5,555	41,712	356.20	59	9,700	233.30	270	72	721
CRM-6	1.49	28.60	5,300	39,386	333.30	62	8,634	186.07	255	76	700
Test 7	73.90	139.40	901	51,503	12.20	39,848	56,800	86.00	56,019	1.88	79
CRM-7	72.14	142.19	793	50,158	9.59	37,587	54,979	71.89	54,208	2.09	84
Test 8	46.90	278.40	279	>10%	8.60	13,154	131,500	30.30	84,300	2.50	8.60
CRM-8	45.14	277.59	263	1,100,766	10.40	12,406	120,409	28.19	81,061	2.49	-
Test 9	<0.5	9.80	30	13,846	3.20	28	2,645	1.07	34	<1	2,170
CRM-9	-	-	-	-	-	-	-	-	-	-	2,194
Test 10	1.30	238.60	100	32,243	7.10	6	726	7.50	64	<1	11,984
CRM-10	1.57	244.34	98	32,935	5.62	12	700	8.98	59	0.20	11,500
Test 11	26.20	47,284	33	18,153	<0.5	5	298	0.91	41	455	47,677
CRM-11	-	47,538	-	-	-	-	-	-	-	471	48,370

Upper limits (UL):

AuME+

Element	Au	Ag	As	Bi	Cd	Cu	Hg	Mo
Unit	ppb	ppm	ppm	ppm	ppm	ppm	ppm	ppm
DL	20	0.50	0.5	1	0.10	1	1	0.5
UL	500,000	5,000	50,000	1,000	5,000	150,000	1,000	30,000
Method	AuME+	AuME+	AuME+	AuME+	AuME+	AuME+	AuME+	AuME+

Element	Pb	S	Sb	Se	Te	Tl	W	Zn
Unit	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm
DL	1	50	0.50	1	0.5	0.5	0.5	1
UL	150,000	300,000	5,000	1,000	1,000	1,000	1,000	150,000
Method	AuME+	AuME+	AuME+	AuME+	AuME+	AuME+	AuME+	AuME+

ZARAZMA

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نوع نمونه:	سنگ	تاریخ درخواست:	۱۴۰۲/۰۷/۰۶ ۱۲:۰۷:۲۷
نام شرکت:		تاریخ دریافت نمونه:	۱۴۰۲/۰۷/۰۷ ۰۹:۲۵:۳۱
شماره پرونده:	۱۴۰۲-۲۹۷۱	تاریخ انجام آزمون:	۱۴۰۲/۰۷/۰۷ ۱۰:۱۸:۲۹
نام آزمایشگاه:	موسسه مطالعات معدنی زرآزما	تاریخ صدور نتیجه:	۱۴۰۲/۰۷/۰۹ ۱۱:۱۹:۳۷
آدرس مشتری:		روش آزمون:	Au-Me Plus
امضاء مدیر فنی:		هزینه آزمون (ریال):	۱۱,۰۰۰,۰۰۰
نام و سمت تصدیق کننده:	صالح صیحدل - مدیر عامل	آدرس آزمایشگاه:	تهران، پانین‌تر از فلکه دوم صادقیه، بزرگراه محمد علی جناح، خیابان طاهریان، کوی ارغوان، پلاک ۲۳، واحد۶ - تلفن:۰۲۱-۴۹۷۳۵

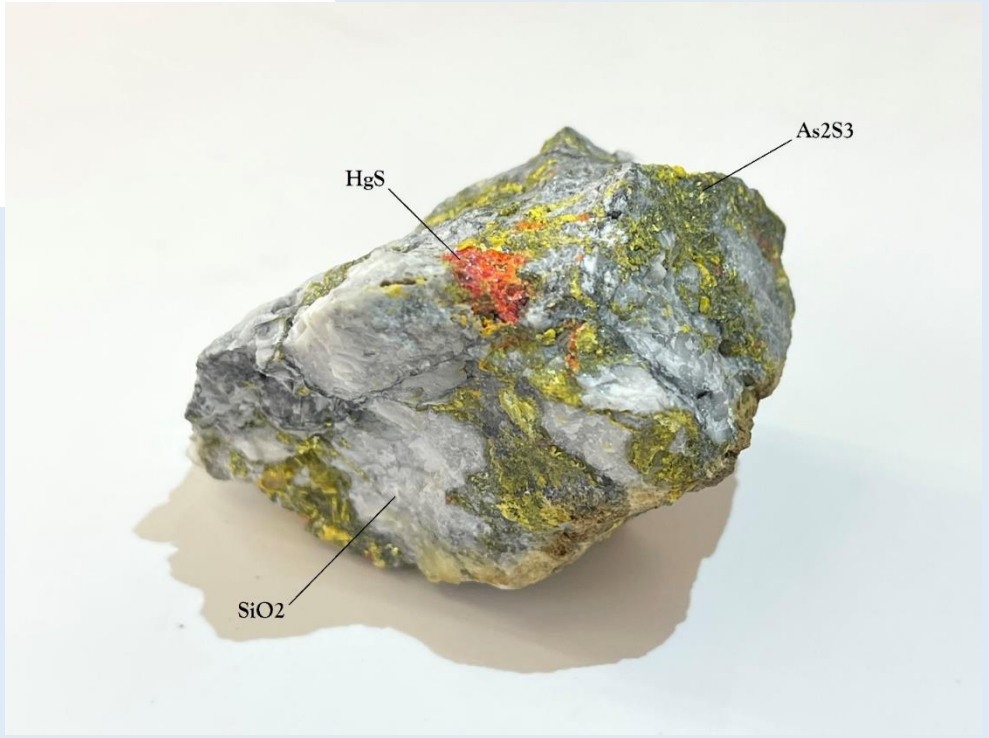
واحد منتخب معدنی سال ۹۳ | دارای گواهینامه iso 17025:9001 | آزمایشگاه معتمد اداره استاندارد | آزمایشگاه معتمد اداره محیط زیست

توجه : نتایج فقط بازگو کننده نمونه ارسالی به آزمایشگاه می باشد.

CERTIFICATE OF ANALYSIS 1404-4971

1404-4971

Element	Au	Ag	As	Bi	Cd	Cu	Hg	Mo	Pb	S	Sb	Se	Te	Tl	W	Zn
DL	ppb	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm
Unit	20	0.5	0.5	1	0.1	1	1	0.5	1	50	0.5	1	0.5	0.5	0.5	1
Method	AuME+	AuME+	AuME+	AuME+	AuME+	AuME+	AuME+	AuME+	AuME+	AuME+	AuME+	AuME+	AuME+	AuME+	AuME+	AuME+
CO																
Sample 1	364	<0.5	>5%	<1	<0.1	15	15.4	14.6	7	89158	194	24	0.6	5.4	1.5	19
Sample 2	387	0.6	>5%	<1	<0.1	15	16.8	14.4	6	91080	203	28	0.7	5.7	1.5	17
Sample 3	411	<0.5	>5%	<1	<0.1	15	17.3	15.6	5	90892	183	27	0.7	5.8	1.5	17



ZARAZMA

شماره پرونده:	1404-5817	تاریخ انجام ازمون:	1404/08/02 11:36:06
نام آزمایشگاه:	موسسه مطالعات معدنی زرآزما	تاریخ صدور نتیجه:	1404/08/05 12:27:43
آدرس مشتری:		روش آزمون:	Au-Me Plus
امضاء مدیر فنی:		هزینه آزمون (ریال):	11,000,000
نام و سمت تصدیق کننده:	صالح صبحدل - مدیر عامل	آدرس آزمایشگاه:	تهران، پائینتر از فلکه دوم صادقیه، بزرگراه محمد علی جناح، خیابان طاهریان، کوی ارغوان، پلاک ۲۳، واحد۴ - تلفن:021-49735

واحد منتخب معدنی سال ۹۳ | دارای گواهینامه iso 17025:9001 | آزمایشگاه معتمد اداره استاندارد | آزمایشگاه معتمد اداره محیط زیست

توجه : نتایج فقط بازگو کننده نمونه ارسالی به آزمایشگاه می باشد.

CERTIFICATE OF ANALYSIS 1404-5817

1404-5817

Element	Au	Ag	As	Bi	Cd	Cu	Hg	Mo	Pb	S	Sb	Se	Te	Tl	W	Zn
DL	20	0.5	0.5	0.5	0.1	1	1	0.5	1	50	0.5	1	0.5	1	1	1
Unit	ppb	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm
Method	AuMe+	AuMe+	AuMe+	AuMe+	AuMe+	AuMe+	AuMe+	AuMe+	AuMe+	AuMe+	AuMe+	AuMe+	AuMe+	AuMe+	AuMe+	AuMe+
CO																
CO																
SM7685	423	0.8	>5%	1.2	4349	72	16.52	5.8	328	>30%	2548	7.2	84.1	9.7	0.8	1223

Future plans:

- Practical workshops
- New microwave methods
- Evaluation of mineral analysis field and developing new packages
- REE packages

Thank you