

# Tabacco

Determinazione di benzene, toluene, xileni, piridina, limonene, naftalene, fenolo e nicotina nelle dieci marche di sigarette più vendute in Italia

**Metodo: SPME-GC/MS in diluizione isotopica**

# Scopo del lavoro:

Sviluppare un metodo SPME per l'analisi qualitativa e quantitativa dei composti volatili presenti nel fumo diretto e laterale di sigaretta.

Il campionamento deve rispettare i parametri utilizzati nelle macchine fumatrici.

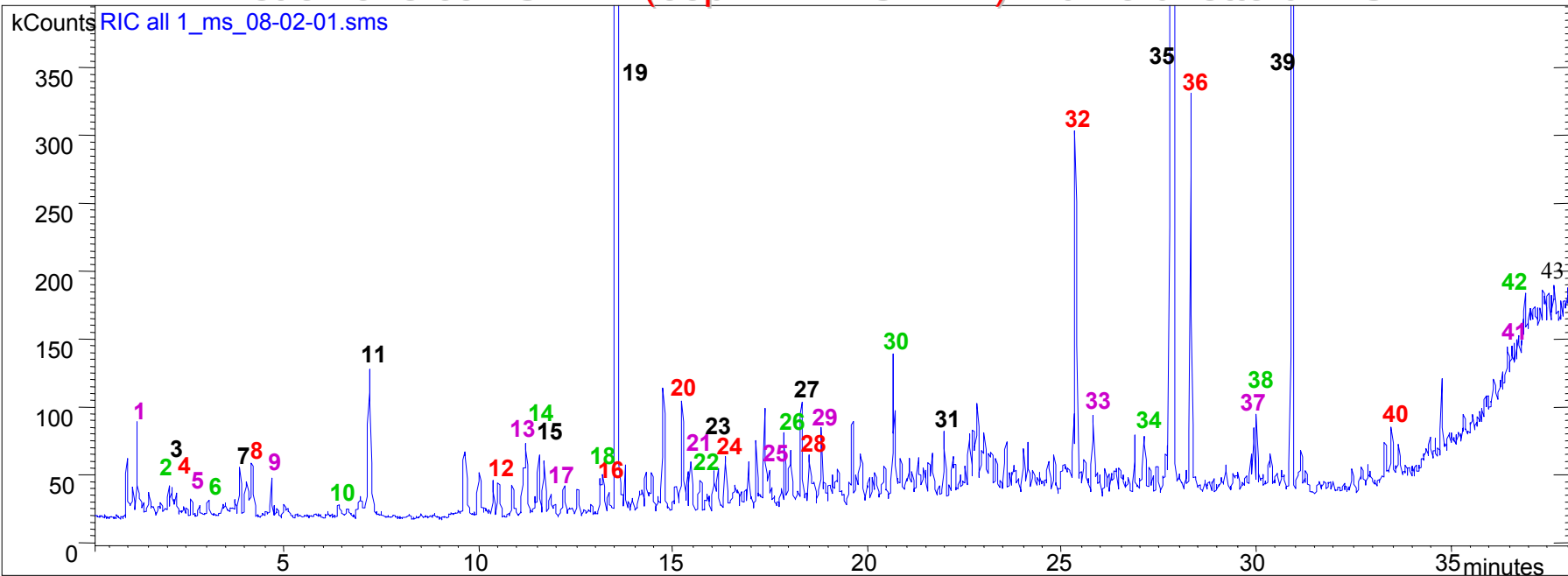
## Parametri analitici

fibra SPME: PDMS/DVB

Temperatura del campione: 40°C

Tempo di esposizione: 1 min

# Estrazione con SPME (65µm PDMS/DVB): Fumo diretto di MS



	NOME	PM	CAS		NOME	PM	CAS
1	cyclopentene	68(67)	142-29-0	23	1,2,4-trimethylbenzene	120*	95-63-6
2	acetone	58(59)	67-64-1	24	2-methyl pyridine	93*	109-06-8
3	1,3 cycloesadiene	80(79)	592-57-4	25	1,3,5-trimethylbenzene	120*	108-67-8
4	2,4 hexadien-1-ol	98	111-28-4	26	furan-3-methyl	82*	930-27-8
5	2-methyl furan	82*	534-22-5	27	beta ocimene	136	13877-91-3
6	2-butanone	72*	78-93-3	28	3-ethyl pyridine	107*	536-78-7
7	benzene	78*	71-43-2	29	allo-ocymene	136*	7216-56-0
8	2-5 dimethyl furan	96*	625-86-5	30	pyridine-3-ethenyl	105*	1221-55-7
9	2-pentanone	86*	107-87-9	31	2-cycloppen-1-one,2,3 dimethyl	110*	1121-05-7
10	cyclopentane,1-methylene-3-(1-methylethyl)	126	53771-88-3	32	solanone	194*	54868-48-3
11	toluene	91	108-88-3	33	naphtalene	128*	91-20-3
12	2,6-dimethyl-2,6-octadiene	138	2492-22-0	34	3-acetoxypyridine	133*	17747-43-2
13	p-xylene	106	106-42-3	35	nicotine	162*	54-11-5
14	m-xylene	106	108-38-3	36	9-eicosyne	278*	71899-38-2
15	p-menth-1-ene	138	1195-31-9	37	phenol 2-methyl	108*	95-48-7
16	pyridine	79*	110-86-1	38	phenol	94*	108-95-2
17	1-methyl pyrrole	81*	96-54-8	39	triacetina	218	102-76-1
18	o-xylene	106	95-47-6	40	2-methoxy-4-vinylphenol	150*	7786-61-0
19	limonene	136*	5989-54-8	41	2-pyridone	95*	142-08-5
20	cis ocimene	136*	3338-55-4	42	indole	117*	120-72-9
21	1,3,5,7-cyclooctatetraene	104*	629-20-9	43	hydroquinone	110*	123-31-9
22	benzene 1-methyl-3-(1-methylethyl)	134	535-77-3				

# Analisi Quantitativa

analita

standard interno

Benzene



Benzene d6

Toluene



Toluene d8

m-Xylene



o-Xylene d10

p-Xylene



o-Xylene d10

Pyridine



Pyridine d5

o-Xylene



o-Xylene d10

Limonene



Toluene d8

Naphtalene



Naphtalene d10

Nicotine



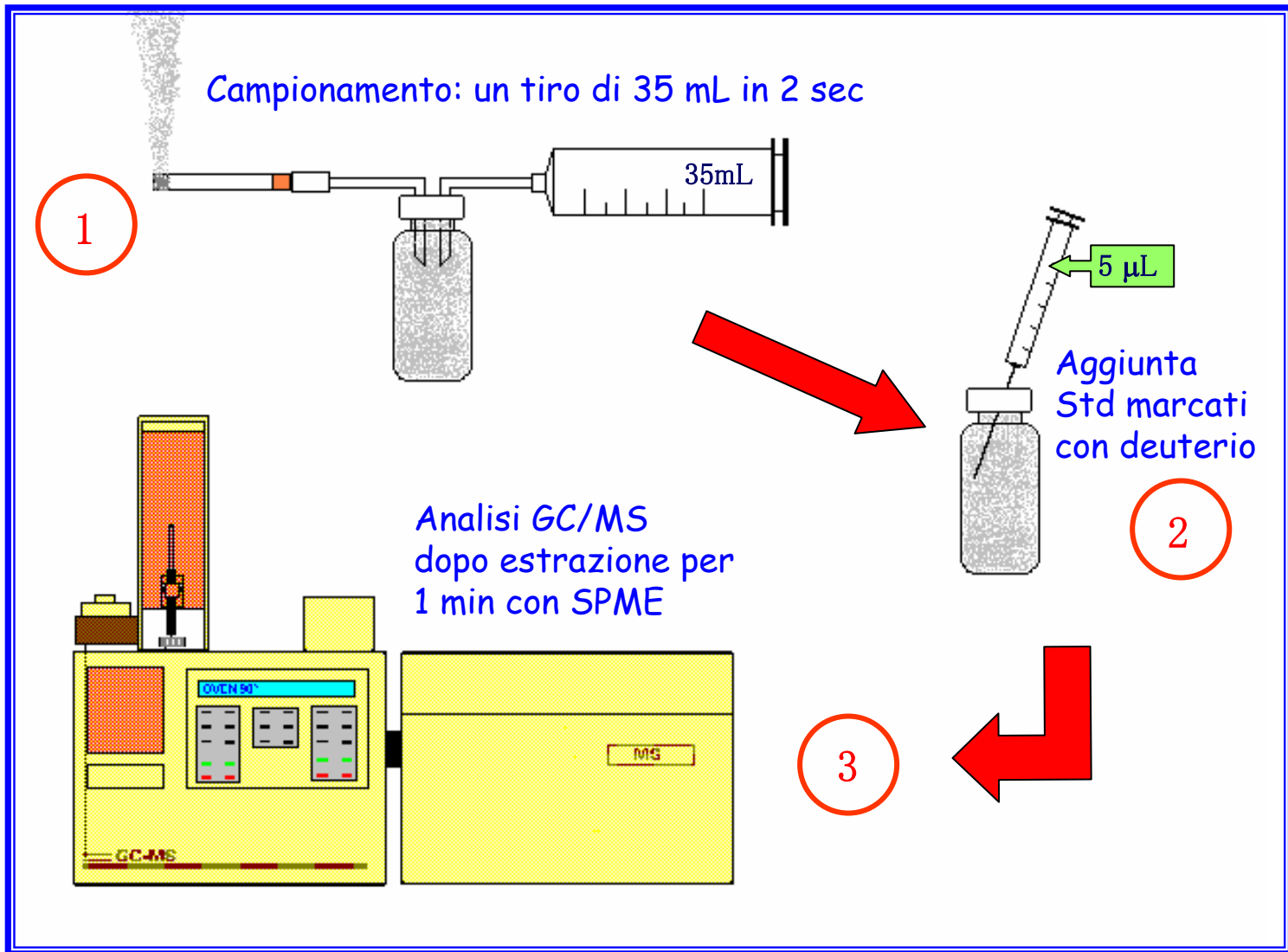
Nicotine d3

Phenol

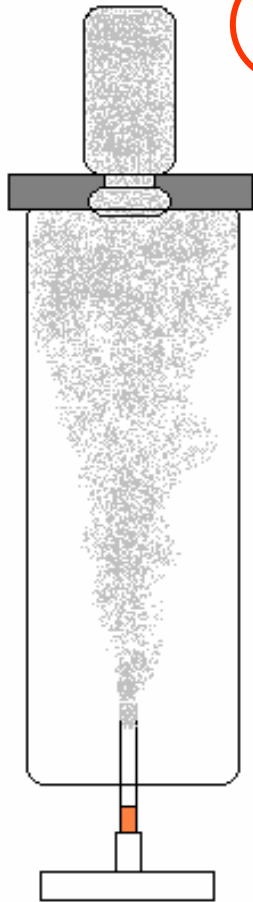


Phenol d6

# Campionamento fumo diretto

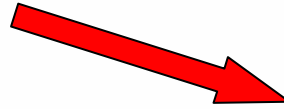


# Campionamento fumo laterale

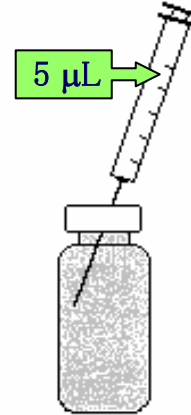


1

Campionamento: esposizione vial per 2 min



5  $\mu$ L



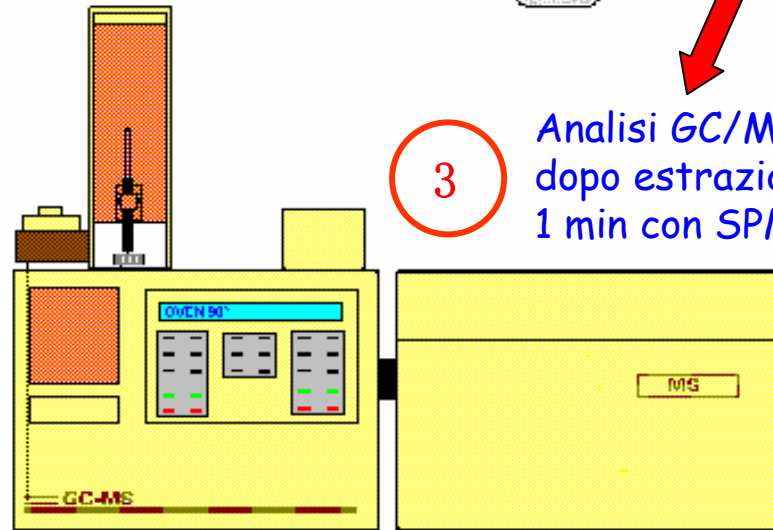
2

Aggiunta Std marcati con deuterio

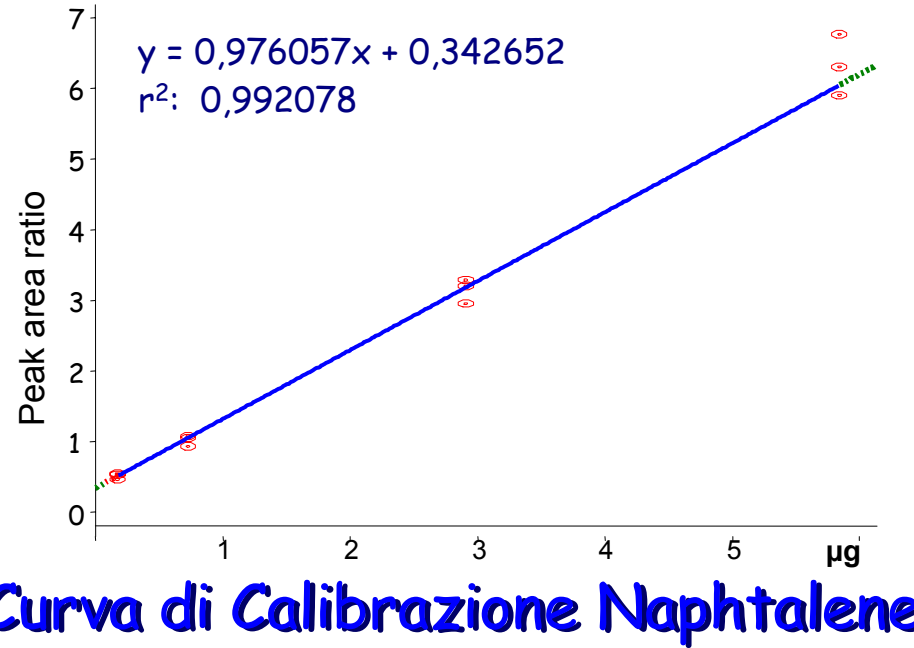
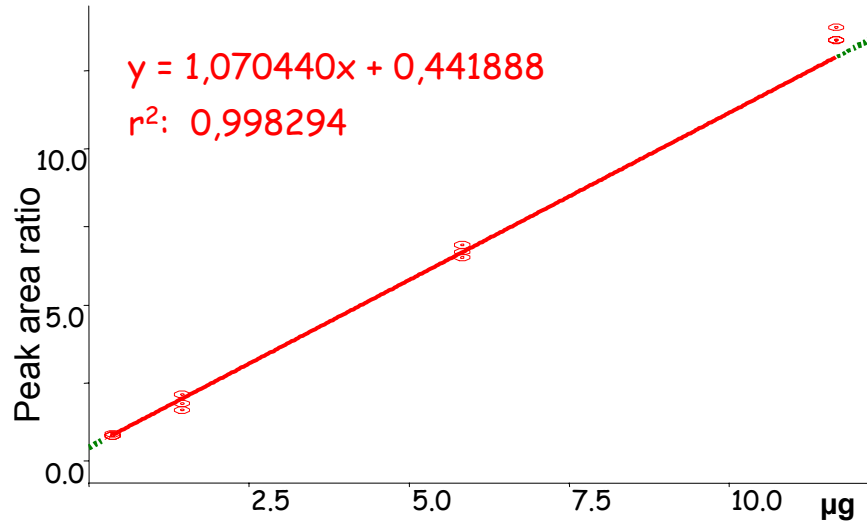


3

Analisi GC/MS dopo estrazione per 1 min con SPME



# Curva di Calibrazione o-Xylene



# Curva di Calibrazione Naphtalene

# Le dieci sigarette più vendute in Italia

MS

Diana KSF

Marlboro

Merit 100's

Multifilter 100's

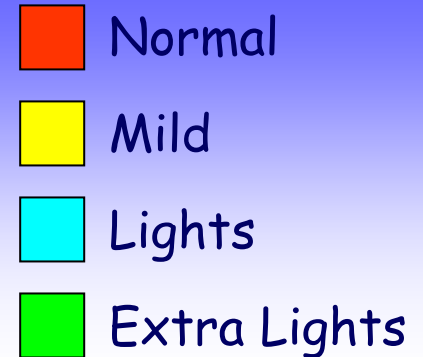
MS Mild

Diana Specially Mild

Marlboro Medium

Marlboro Lights

Multifilter 100's Extra Lights

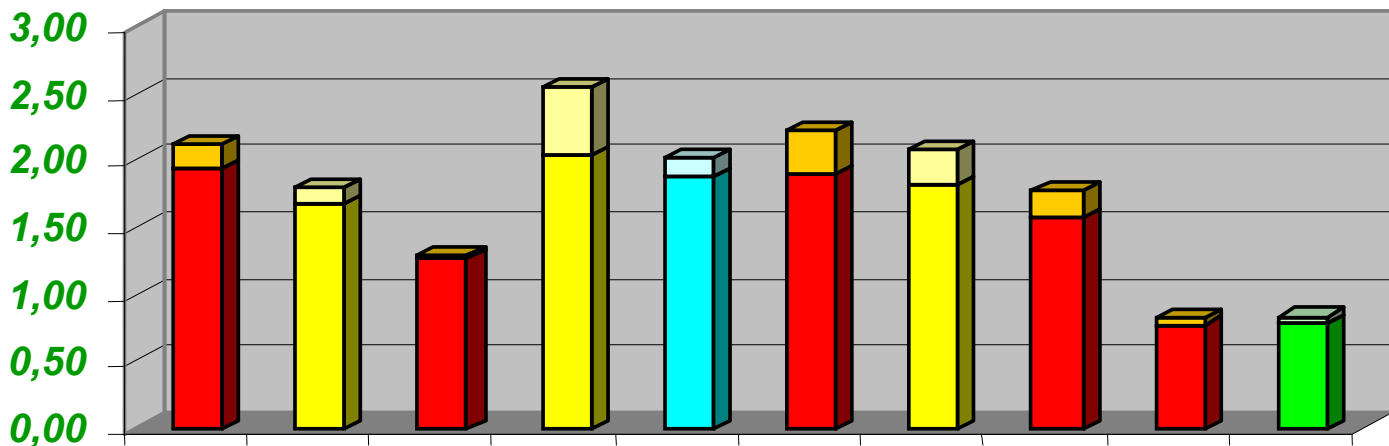




# Benzene

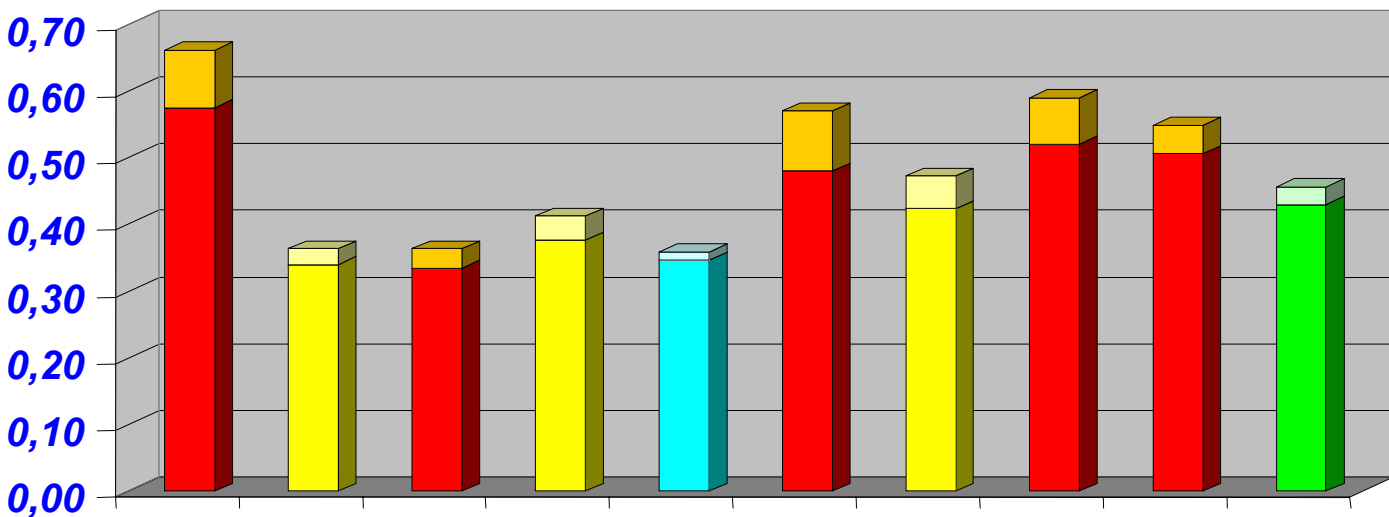
Mainstream  
Smoke

$\mu\text{g}/\text{drag}$



Sidestream  
Smoke

$\mu\text{g}$  (2min)

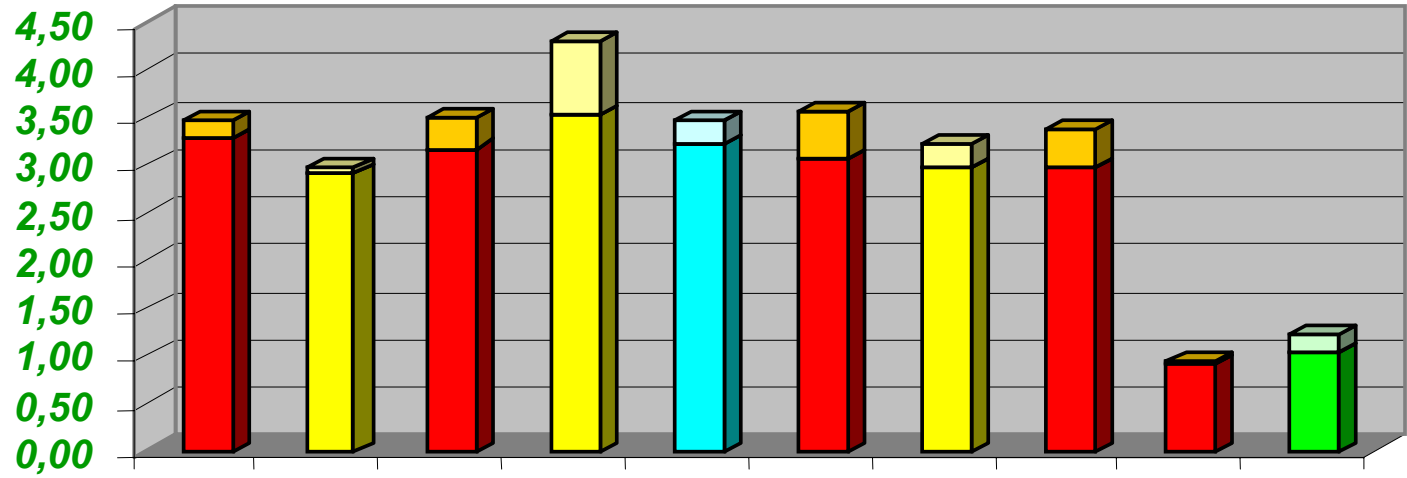


MS  
MS Mild  
Marlboro  
Marlboro Med  
Marlboro Lights  
Diana KSF  
Diana SM  
Merit 100's  
Multifilter 100's  
Multifilter 100's EL

# Toluene

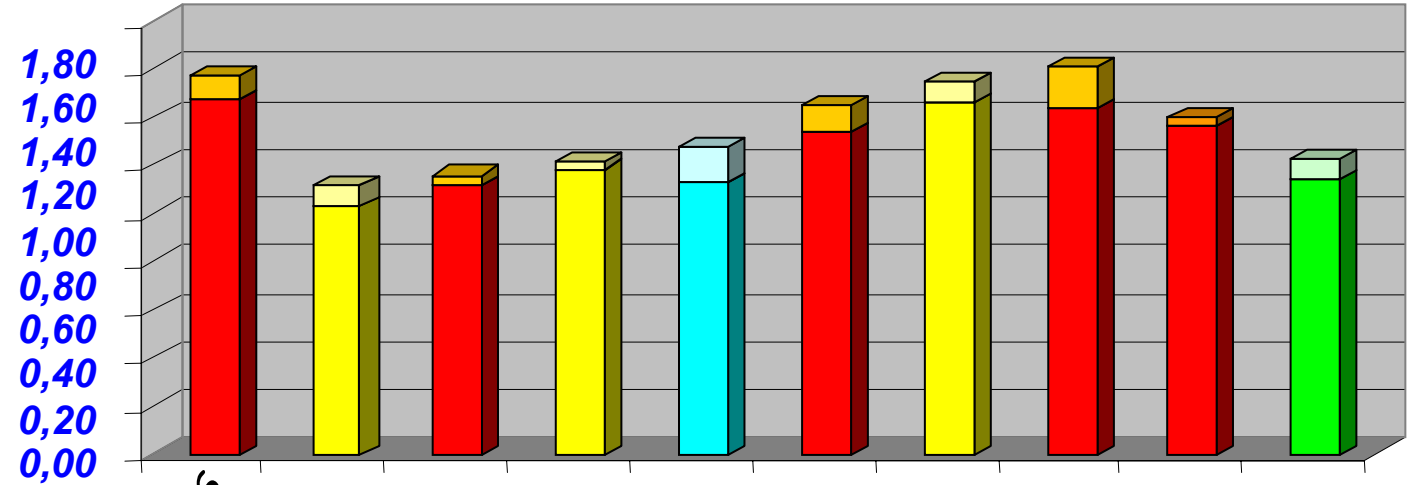
Mainstream  
Smoke

$\mu\text{g}/\text{drag}$



Sidestream  
Smoke

$\mu\text{g}$  (2min)

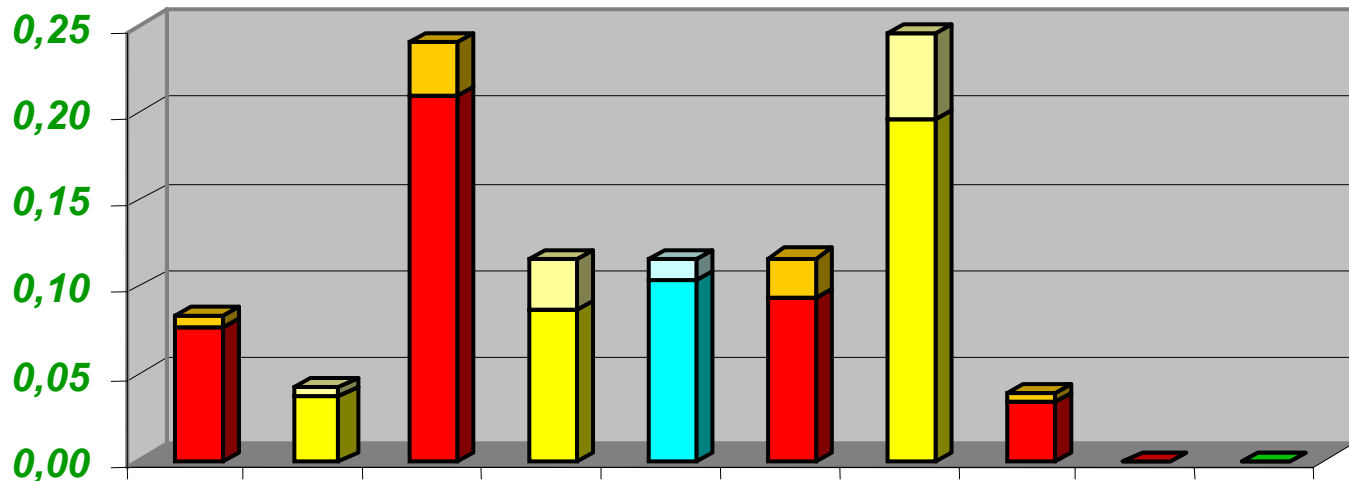


MS  
MS Mild  
Marlboro  
Marlboro Med  
Marlboro Lights  
Diana KSF  
Diana SM  
Merit 100's  
Multifilter 100's  
Multifilter 100's EL

# Pyridine

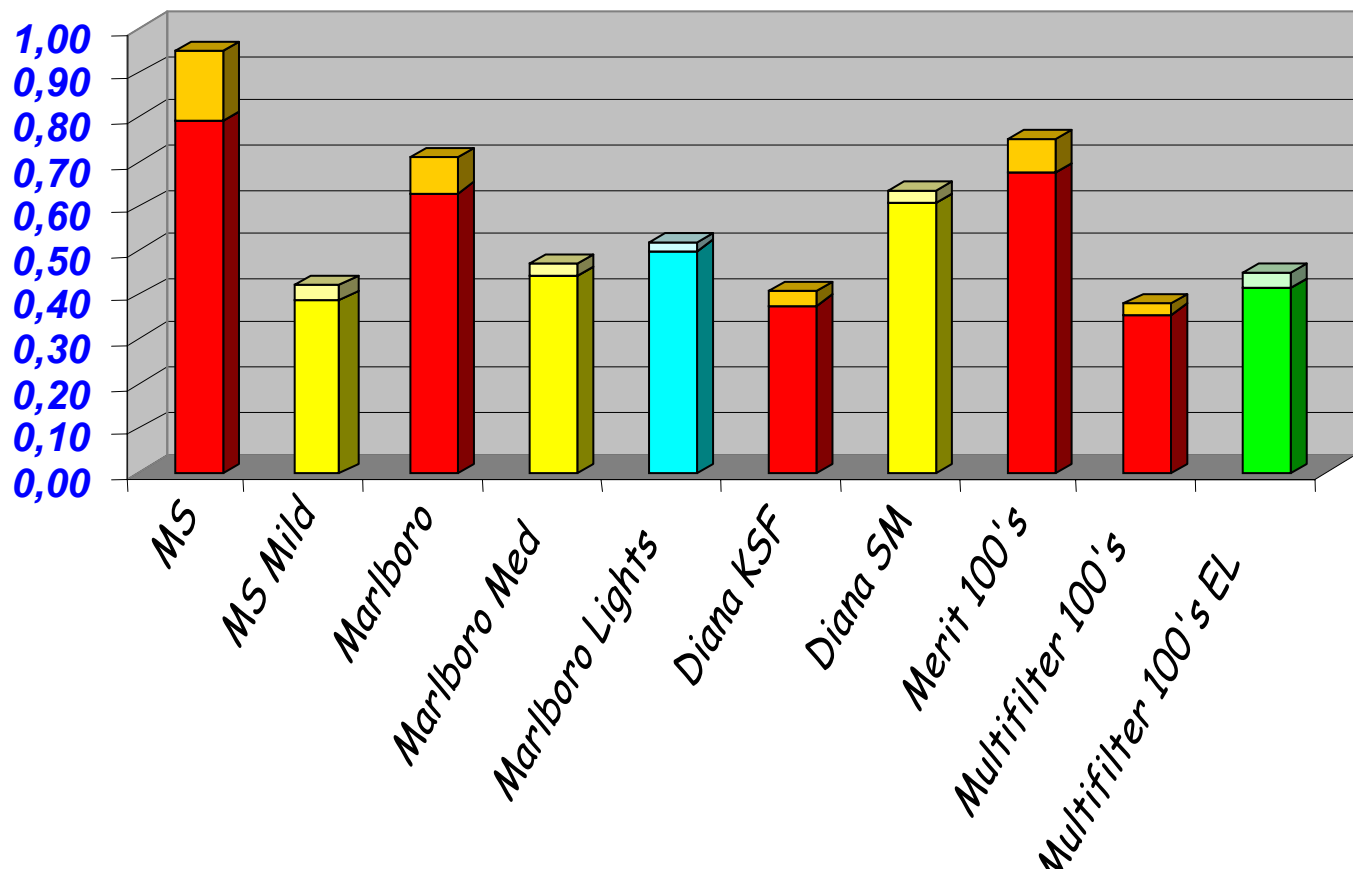
Mainstream  
Smoke

$\mu\text{g}/\text{drag}$



Sidestream  
Smoke

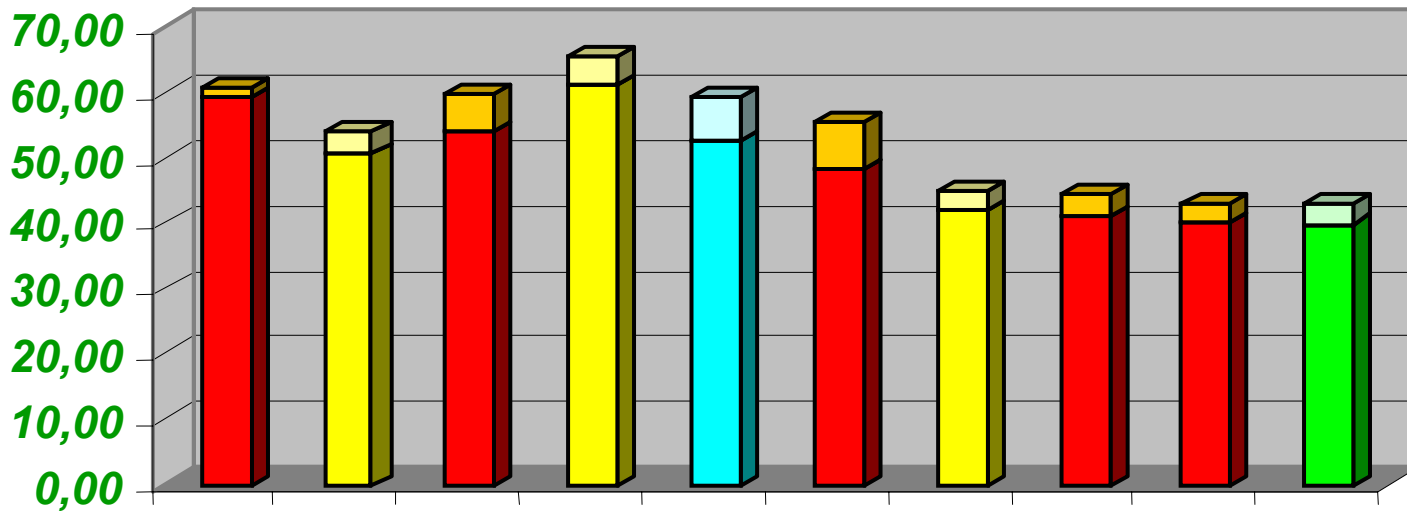
$\mu\text{g}$  (2min)



# Nicotine

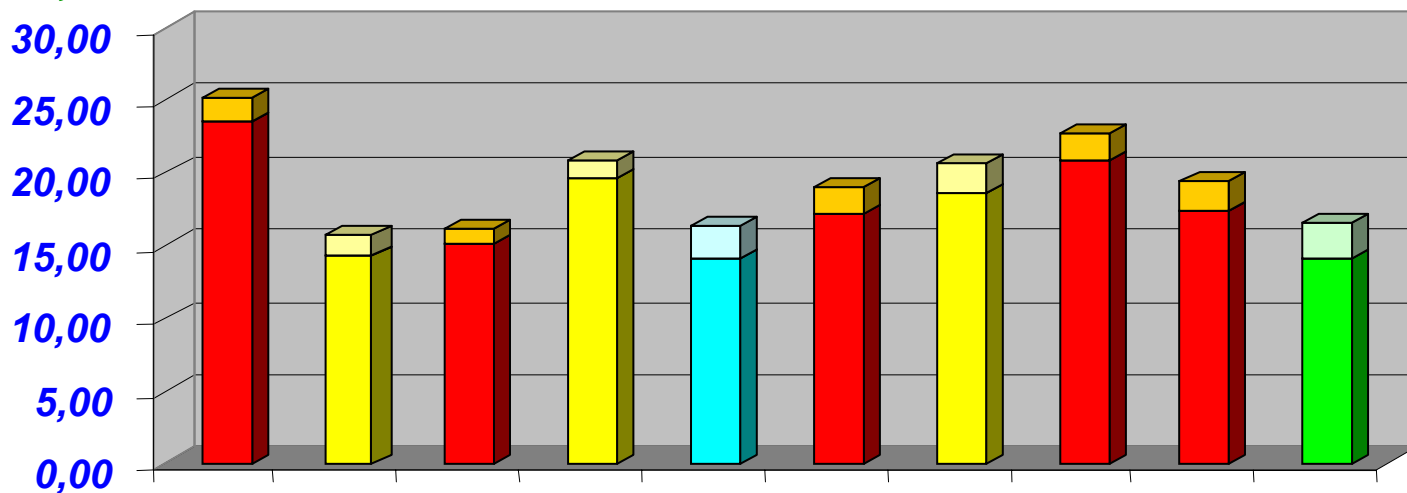
Mainstream  
Smoke

$\mu\text{g}/\text{drag}$



Sidestream  
Smoke

$\mu\text{g}$  (2min)



MS

MS Mild

Marlboro

Marlboro Med

Marlboro Lights

Diana KSF

Diana SM

Merit 100's

Multifilter 100's

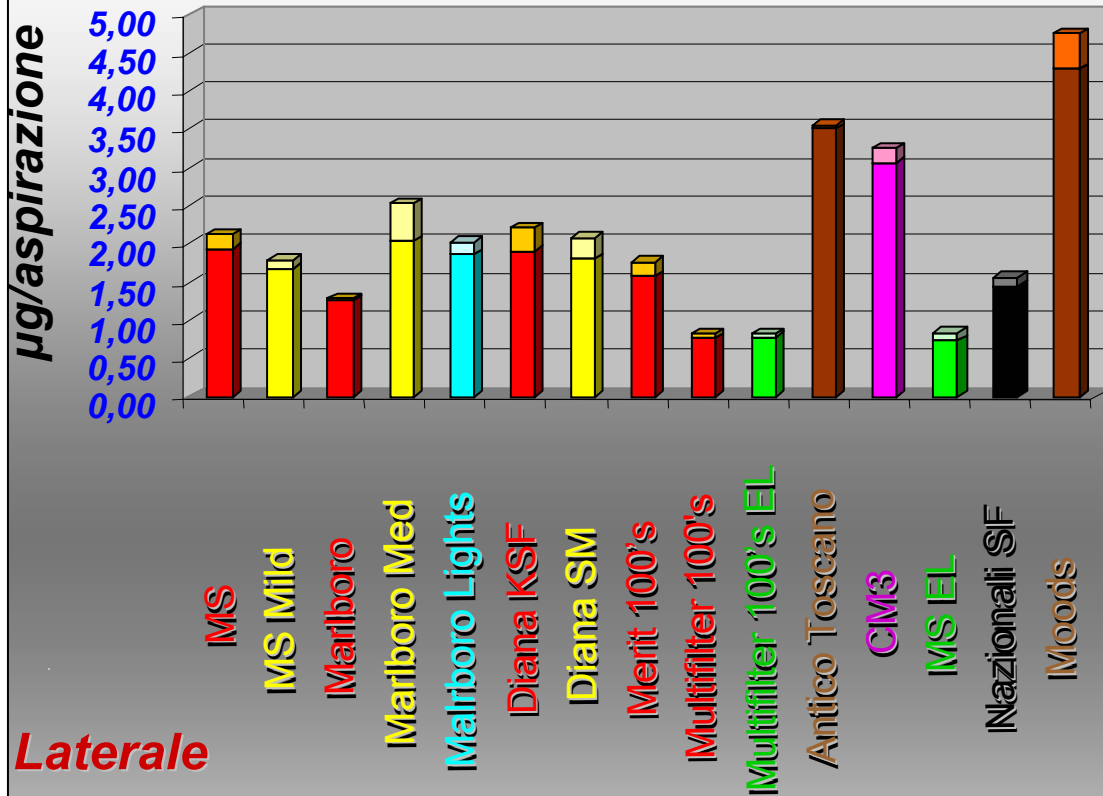
Multifilter 100's EL

...Analisi quantitativa...

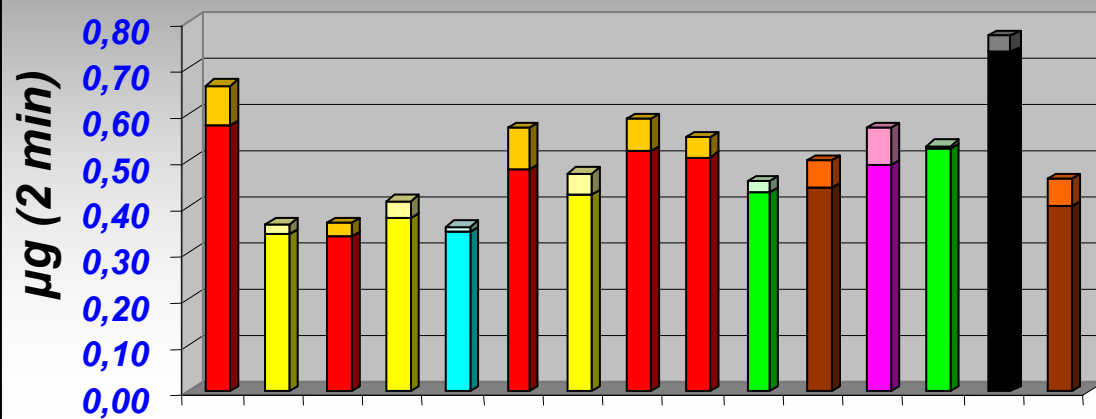


## Diretto

# Benzene



## Laterale



# Precisione fumo diretto (n=3)

SIGARETTA	Benzene		Toluene		m-Xilene		Limonene		Fenolo		Nicotina	
	µg/asp	CV	µg/asp	CV	µg/asp	CV	µg/asp	CV	µg/asp	CV	µg/asp	CV
MS	1,9	9,7	3,3	5,8	0,4	12,1	3,2	3,6	0,6	7,8	59,1	2,6
MS Mild	1,7	6,9	2,9	1,6	0,3	5,2	3,0	6,8	0,7	23,9	50,8	6,3
Marlboro	1,3	1,6	3,2	10,2	0,4	7,1	2,5	16,5	0,4	4,6	54,0	10,2
Marlboro M	2,0	24,9	3,5	22,0	0,4	16,0	3,4	17,1	0,5	10,6	60,9	7,5
Marlboro L	1,9	8,5	3,2	7,2	0,3	9,2	2,4	10,7	0,4	33,3	52,7	12,6
Diana KSF	1,9	16,3	3,0	16,4	0,4	23,9	3,1	17,0	0,7	22,2	48,0	15,0
Diana SM	1,8	15,2	3,0	8,1	0,4	1,6	3,8	0,7	0,4	20,4	41,8	7,0
Merit 100's	1,6	12,3	3,0	13,7	0,3	7,5	3,5	5,7	0,1	14,4	41,0	8,0
Multifilter 100's	0,8	7,2	0,9	6,2	0,1	12,5	1,4	6,7	0,3	31,2	40,0	6,9
Multifilter 100's EL	0,8	6,0	1,0	17,5	0,1	14,3	1,4	19,8	0,4	6,4	39,8	7,9
MS EL	0,7	15,5	0,9	25,1	0,1	21,8	2,3	12,5	0,2	38,6	24,1	16,3
Nazionali SF	1,4	8,4	1,8	10,4	0,2	9,4	2,8	9,6	1,6	12,8	49,2	5,4
Antico Toscano	3,5	0,8	13,0	9,2	1,8	15,5	19,1	6,6	0,8	26,5	86,9	3,8
CM3	3,1	6,2	5,1	6,1	0,7	2,1	5,5	2,7	1,3	14,7	98,2	10,5
Moods (Sigari)	4,3	11,1	8,8	1,4	1,4	1,0	11,2	3,2	0,6	4,6	41,2	13,3

# Accuratezza:

Fumo diretto su 2 marche di sigarette (n=3)

	Benzene	Toluene	m-Xilene	Limonene	Fenolo	Nicotina
MS+ Punto 2 Curva	96.1%	101.8%	103.0%	107.2%	103.2%	99.6%
Marlboro L+ Punto 2 Curva	99.0%	101.3%	106.4%	107.4%	109.7%	98.0%