A Practical Applications Guide for Analytical Pyrolysis -GC/MS

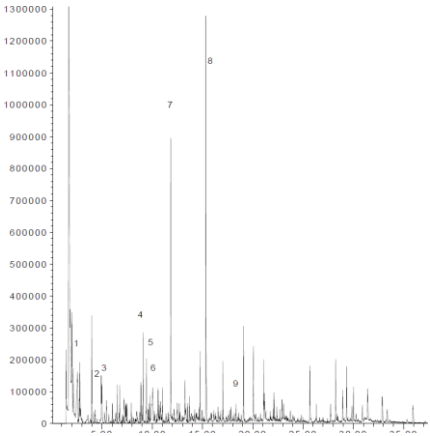
Tobacco

CDS Analytical

analytix

Analysis of Menthol Cigarettes

The material used in cigarettes is a carefully formulated product of natural tobacco together with a variety of additives. Analytical pyrolysis can reveal both the materials produced from the tobacco at high temperatures as well as the volatile additives. Pyrolysis has also been applied to the study of the papers used in cigarette manufacture, as well as the filter material. A small (~250 µG) sample of tobacco from a mentholated cigarette was pyrolysed at 700°C for 15 seconds. The resulting pyrogram shows a wide range of natural products, including nicotine and levoglucosan (a product of cellulose) as well as additives, including glycerin and menthol. Selected peaks from the pyrogram are identified in Table I.



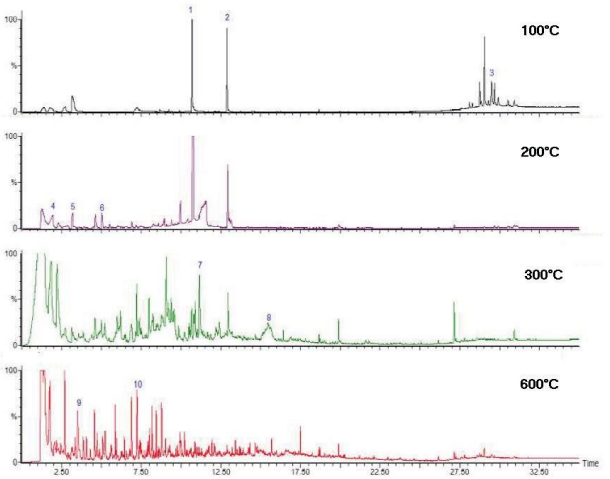
Pyrogram of mentholated cigarette

Pyroprobe Setting	
Set-point:	700°C 15 sec
Valve Oven:	300°C
Transfer Line:	325°C
GC/MS Setting	
Column:	5% phenyl
Carrier:	Helium, 50:1 split
Injector:	300°C
Oven:	40°C for 2 min
	8°C/min to 300°C hold 12 min
Mass Range:	35-550

Peak number	Compound
1	Acetic acid
2	Propylene glycol
3	Toluene
4	Limonene
5	Phenol
6	Glycerin
7	Menthol
8	Nicotine
9	Levoglucosan

Multi-step Pyrolysis on Smokeless Tobacco

The tobacco tested here is a wintergreen flavored smokeless product. It is sampled using of 4 separate temperatures to examine both volatile compounds and pyrolysis products. At 100°C, nicotine and methyl salicylate, (wintergreen flavoring) are apparent. In addition, vitamin E, a natural product of tobacco is present. At 200°C, acetic acid and propylene glycol evolve, and tobacco begins to degrade, so pyrolysis products are also seen. At 300°C, degradation products of cellulose become significant, including furans and levoglucosan. Performing multiple thermal steps provides several advantages. Some of the minor constituents (like the vitamin E) would be difficult to identify if only performing single pyrolysis run. Secondly, it clear that the nicotine is quite volatile. Finally, additives are determined easily, apart from the products generated when tobacco is pyrolysed.



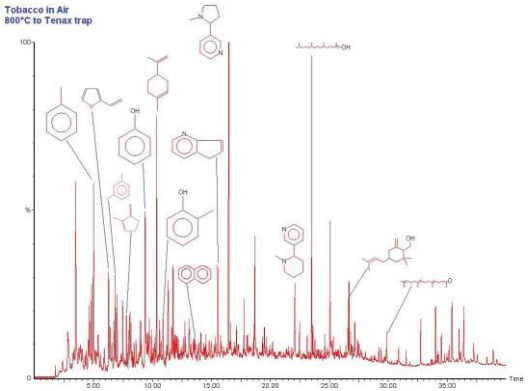
Peak Identification
1. Methyl salicylate
2. Nicotine
3. Vitamin E
4. Acetic acid
5. Propylene glycol
6. 2-Furanmethanol
7. Hydroxymethyl furan carboxaldehyde
8. Levoglucosan
9. Toluene
10. Phenol

Analysis of wintergreen flavored smokeless tobacco at 100, 200, 300 and 600°C.

Pyroprobe Setting		GC/MS Setting	
Interface:	100,200,300,600°C for 15 sec	Column:	5% phenyl
Valve Oven:	300°C	Carrier:	Helium, 50:1 split
Transfer Line:	325°C	Injector:	300°C
		Oven:	40°C for 2 minutes
			10°C/min to 300°C
		Mass Range:	35-550

Combustion Products

Using the CDS Model 6200 Pyroprobe, burning of tobacco can be studied. Tobacco can be pyrolysed under a reactant gas, such as air, and the pyrolysing reaction products collected onto a trap which is then desorbed to the GC/MS. When tobacco burns, the products include a wide range of compounds, some of which are pyrolysis products, some have been oxygenated, and some just desorbed. The largest peak, nicotine, is simply volatilised from tobacco by heating. Some of the other compounds are pyrolysis products of the cellulose, including furfural and methyl cyclopentenone. Aromatic hydrocarbons produced include toluene, xylene, naphthalene and many others, plus phenolic compounds including phenol and methyl phenol. Aliphatics, especially branched and unsaturated compounds are also generated, including limonene and long-chain alcohols. Because the heating rate, temperature and time for pyrolysis with the CDS Pyroprobe are all selectable, experiments may be designed to simulate the various burning processes involved when tobacco is smoked.



Pyrogram of Tobacco in Air

Pyroprobe Setting		GC/MS Setting	
Pyrolysis:	800°C for 25 sec	Column:	5% phenyl
Valve oven:	325°C	Carrier:	Helium, 50:1 split
Interface:	300°C	Injector:	300°C
Transfer line:	325°C	Oven:	40°C for 2 minutes
Trap rest:	40°C (Tenax)		8°C/min to 300°C
Trap desorption:	325°C for 4 minutes	Mass Range:	35-600 amu
Reactant gas:	Air at 30 ml/minute		

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