

# Quantification of 1,2 Propylene glycol, Glycerol and Sorbitol in Tobacco Products

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# OBJECTIVE

To develop a single method for analysis of humectants (Propylene glycol Glycerol and Sorbitol) in tobacco products

# INTRODUCTION

- Propylene glycol, Glycerol and Sorbitol are humectants.
- Humectants have been used in tobacco and tobacco products to maintain the moisture level.
- Humectants are used in the range of 0.5 – 5 % w/w in tobacco and tobacco Products.

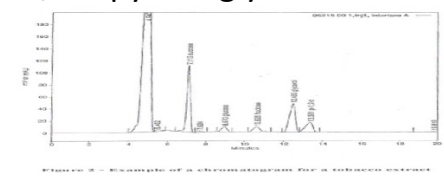
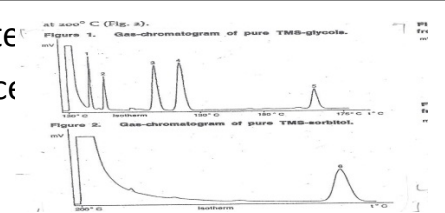
# NEED FOR NEW METHOD

All the literature methods are for tobacco and cigarettes and no methods reported for snus.

The matrix in snus interferes with the other analytes and hence making quantification difficult

# METHODS REPORTED IN THE LITERATURE

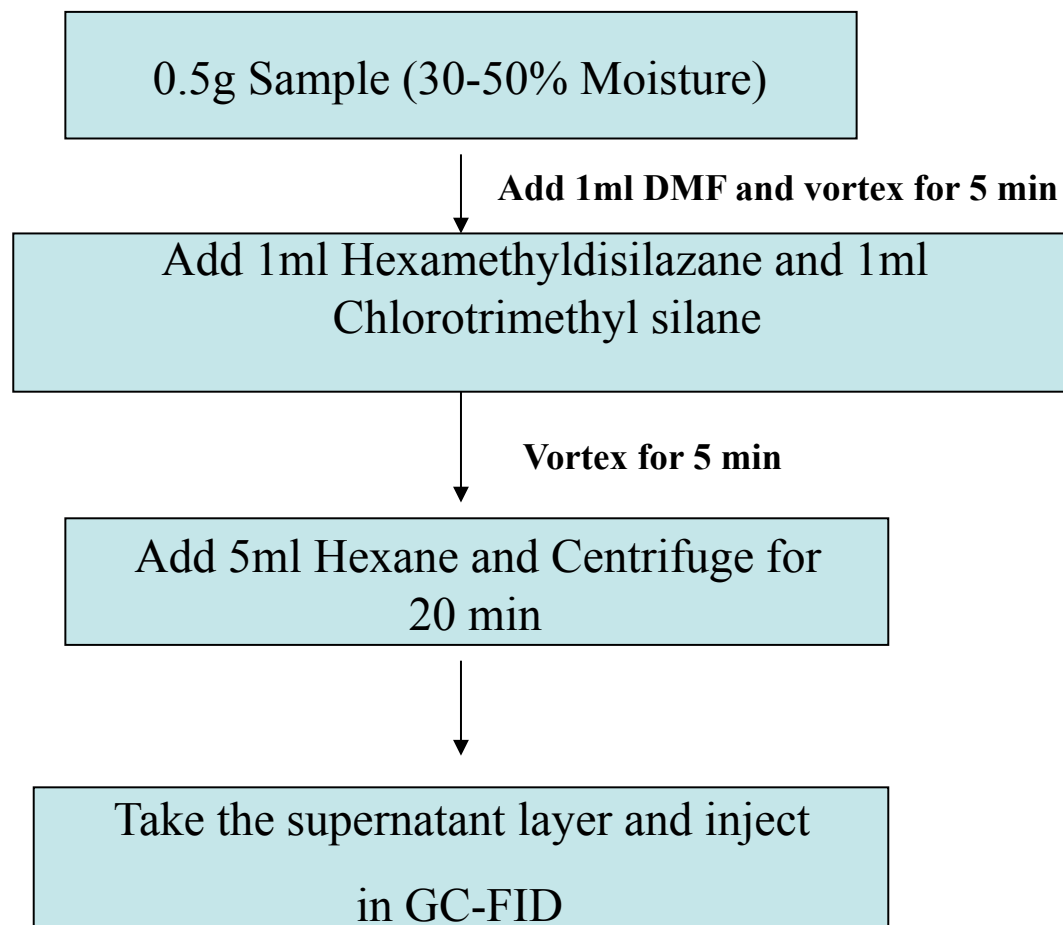
Method	Reference	Limitations
A	AOAC Official Methods of Analysis (1995), Ch 3, p.3	<ul style="list-style-type: none"> <li>• Only Propylene glycol and Glycerol can be analyzed.</li> </ul>
B	Beitrage Zur Tabakforschung. Band 6 . Heft 2. 1971	<ul style="list-style-type: none"> <li>• Sorbitol analyzed separately</li> <li>• Laborious extraction process</li> </ul>
C	Journal of Chromatography, 140 304-309 (1977)	<ul style="list-style-type: none"> <li>• Pyridine used for extraction.</li> <li>• Only Propylene glycol and Glycerol can be analyzed.</li> </ul>
D	CORESTA Recommended method N° 61	<ul style="list-style-type: none"> <li>• Separation of Glycerol, Propylene glycol and Sorbitol.</li> </ul>



ALL THE ABOVE METHODS ARE NOT USED FOR ANALYSIS OF HUMECTANTS IN SNUS

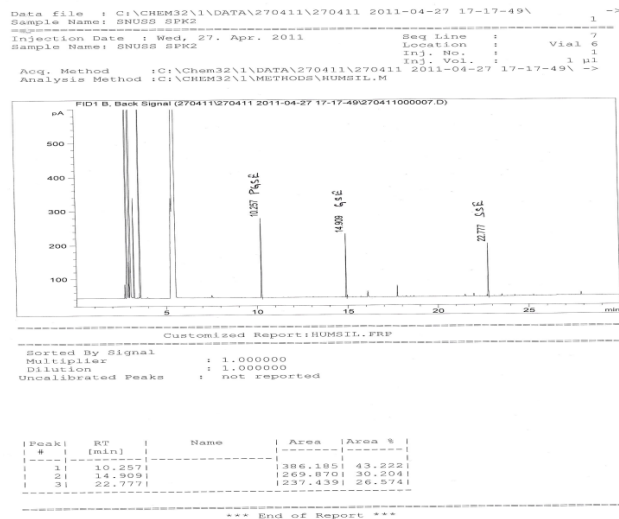


# FLOW DIAGRAM

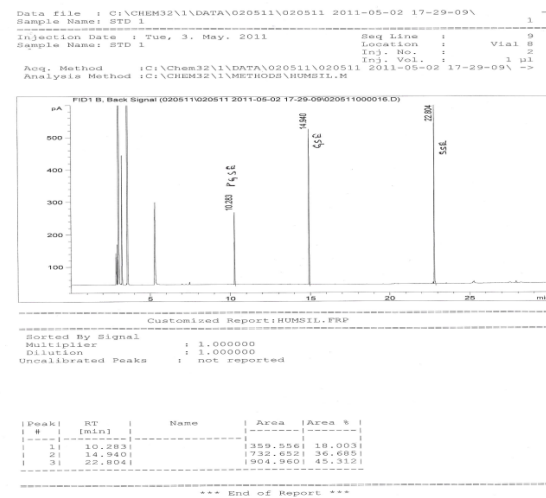


# GC-FID METHOD CONDITION

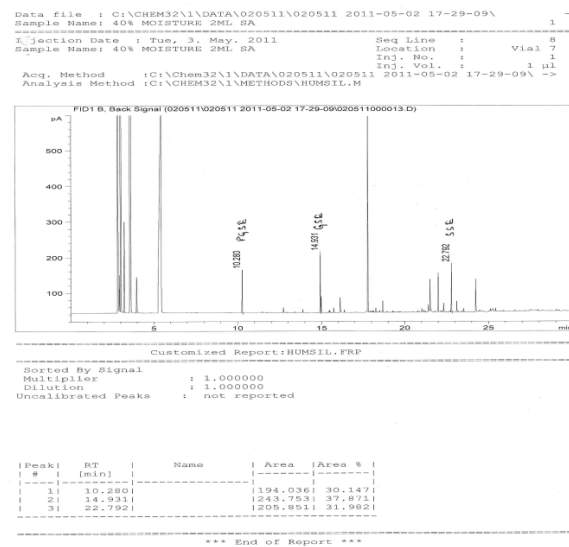
Column : DB-5  
 Dimension : 30 m X 0.25 mm X 0.25 micron  
 Flow : 1.0 ml/min  
 Injector Temperature : 250° C  
 Detector Temperature: 280° C (FID)  
 Oven : Initial 60° C hold 3 min  
       Ramp1 increase @ 10° C per  
       min to 280° C hold for 3 min  
 Split ratio : 100:1  
 Injection volume : 1 µl  
 Run Time : 30 minutes



SPIKE SAMPLE CHROMATOGRAM



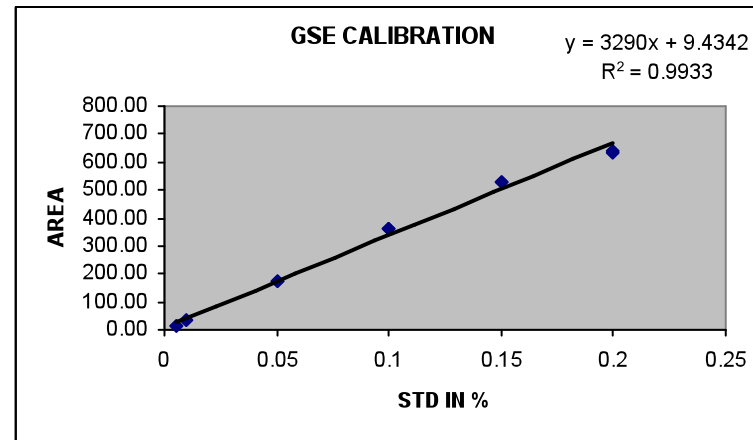
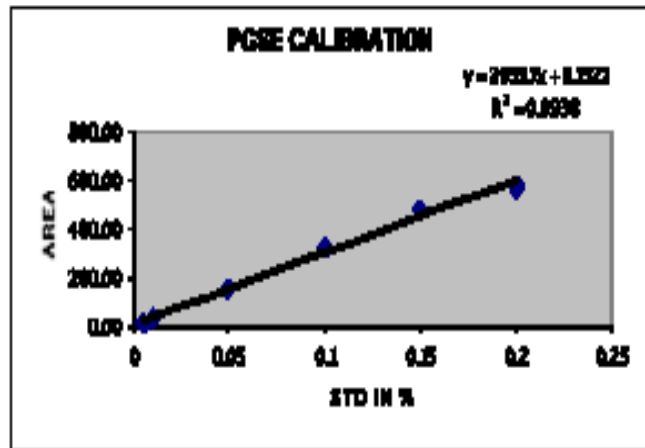
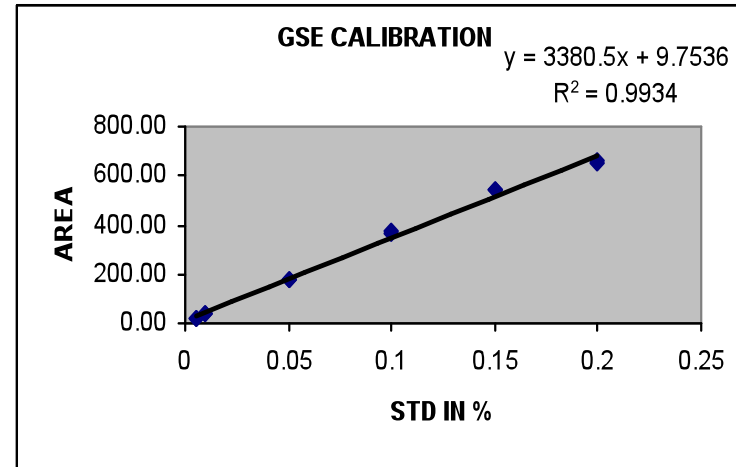
## STANDARD CHROMATOGRAM



GC-FID SNUS SAMPLE CHROMATOGRAM

# LINEARITY

CALIBRATION FOR PROPYLENE GLYCOL, GLYCEROL & SORBITOL					
	STD	%	PGSE	GSE	SSE
	1	0.005	13.59	15.47	14.66
	1	0.005	13.88	15.70	14.89
	2	0.01	31.38	35.98	34.86
	2	0.01	31.49	36.20	34.80
	3	0.05	156.54	179.88	175.18
	3	0.05	154.45	177.29	173.13
	4	0.1	320.72	368.39	360.06
	4	0.1	325.10	373.06	362.62
	5	0.15	473.00	541.85	527.60
	5	0.15	475.34	544.38	528.96
	6	0.2	571.64	653.21	633.59
	6	0.2	575.08	657.53	641.52





# REPEATABILITY

<b>TRIALS</b>	<b>EXPECTED</b>	<b>OBTAINED</b>	<b>% RECOVERY</b>
1	1	0.97	97
2	1	0.96	96
3	1	0.98	98
		<b>% RSD</b>	<b>1.03</b>
<b>GLYCEROL</b>			
<b>TRIALS</b>	<b>EXPECTED</b>	<b>OBTAINED</b>	<b>% RECOVERY</b>
1	1	1.01	101
2	1	1.00	100
3	1	1.03	103
		<b>% RSD</b>	<b>1.51</b>
<b>SORBITOL</b>			
<b>TRIALS</b>	<b>EXPECTED</b>	<b>OBTAINED</b>	<b>% RECOVERY</b>
1	1	0.93	93
2	1	0.93	93
3	1	0.95	95
		<b>% RSD</b>	<b>1.23</b>

# REPRODUCIBILITY

PROPYLENE GLYCOL			
ANALYST-1			
TRIALS	EXPECTED	OBTAINED	% RECOVERY
1	1	0.97	97
2	1	0.96	96
3	1	0.98	98
ANALYST-2			
TRIALS	EXPECTED	OBTAINED	% RECOVERY
1	1	1.00	100
2	1	0.98	98
3	1	0.98	98
		<b>% RSD</b>	<b>1.36</b>

GLYCEROL			
ANALYST-1			
TRIALS	EXPECTED	OBTAINED	% RECOVERY
1	1	1.01	101
2	1	1.00	100
3	1	1.03	103
ANALYST-2			
TRIALS	EXPECTED	OBTAINED	% RECOVERY
1	1	1.04	104
2	1	1.03	103
3	1	1.02	102
		<b>% RSD</b>	<b>1.44</b>

SORBITOL			
ANALYST-1			
TRIALS	EXPECTED	OBTAINED	% RECOVERY
1	1	0.93	93
2	1	0.93	93
3	1	0.95	95
ANALYST-2			
TRIALS	EXPECTED	OBTAINED	% RECOVERY
1	1	0.97	97
2	1	0.95	95
3	1	0.96	96
		<b>% RSD</b>	<b>1.69</b>

# RECOVERY STUDIES

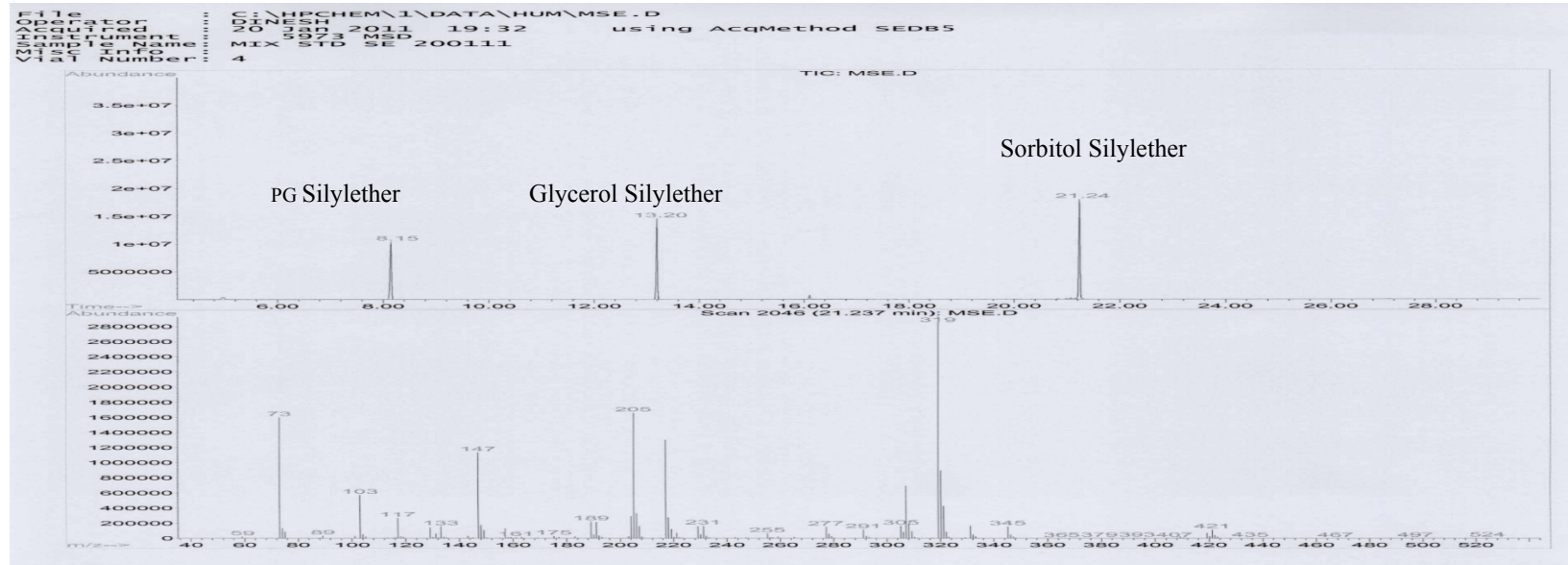
## LOW SPIKE RECOVERY: SPIKED AMOUNT 1%

<b>PROPYLENE GLYCOL</b>		
<b>TRIALS</b>	<b>OBTAINED</b>	<b>% RECOVERY</b>
1	0.97	97
2	0.96	96
3	0.98	98
<b>% RSD</b>	<b>1.03</b>	
<b>GLYCEROL</b>		
<b>TRIALS</b>	<b>OBTAINED</b>	<b>% RECOVERY</b>
1	1.01	101
2	1.00	100
3	1.03	103
<b>% RSD</b>	<b>1.51</b>	
<b>SORBITOL</b>		
<b>TRIALS</b>	<b>OBTAINED</b>	<b>% RECOVERY</b>
1	0.93	93
2	0.93	93
3	0.95	95
<b>% RSD</b>	<b>1.23</b>	

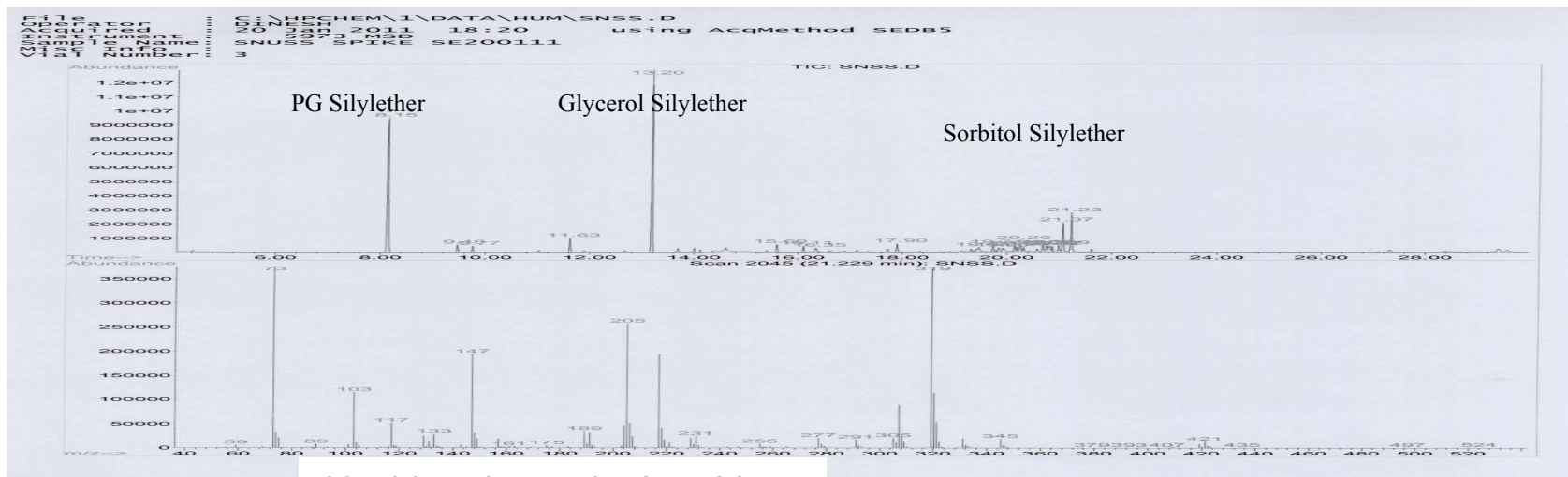
## HIGH SPIKE RECOVERY: SPIKED AMOUNT 2%

<b>PROPYLENE GLYCOL</b>		
<b>TRIALS</b>	<b>OBTAINED</b>	<b>% RECOVERY</b>
1	1.96	98
2	1.94	97
3	2.01	101
<b>% RSD</b>	<b>1.83</b>	
<b>GLYCEROL</b>		
<b>TRIALS</b>	<b>OBTAINED</b>	<b>% RECOVERY</b>
1	2.00	100
2	1.97	99
3	2.03	102
<b>% RSD</b>	<b>1.50</b>	
<b>SORBITOL</b>		
<b>TRIALS</b>	<b>OBTAINED</b>	<b>% RECOVERY</b>
1	1.90	95
2	1.91	96
3	1.97	99
<b>% RSD</b>	<b>1.97</b>	

# GC-MS CONFIRMATION



GC-MS MIX STANDARD CHROMATOGRAM



GC-MS SPIKE SAMPLE CHROMATOGRAM

# ADVANTAGE OF OUR METHOD

- Single step silylation for humectants
- Silylation of humectants not involving use of pyridine
- Method is very simple and requires only 1 hr for complete analysis

THANKYOU