

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

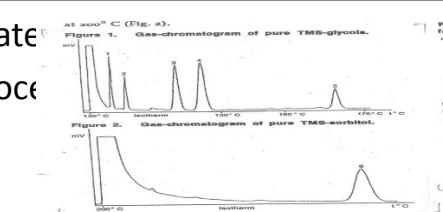
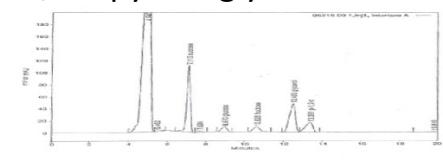
- Propylene glycol, Glycerol and Sorbitol are humectants.
- Humectants have been used in tobacco and tobacco products to maintain the moisture level.
- Humectants are used in the range of 0.5 – 5 % w/w in tobacco and tobacco Products.

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

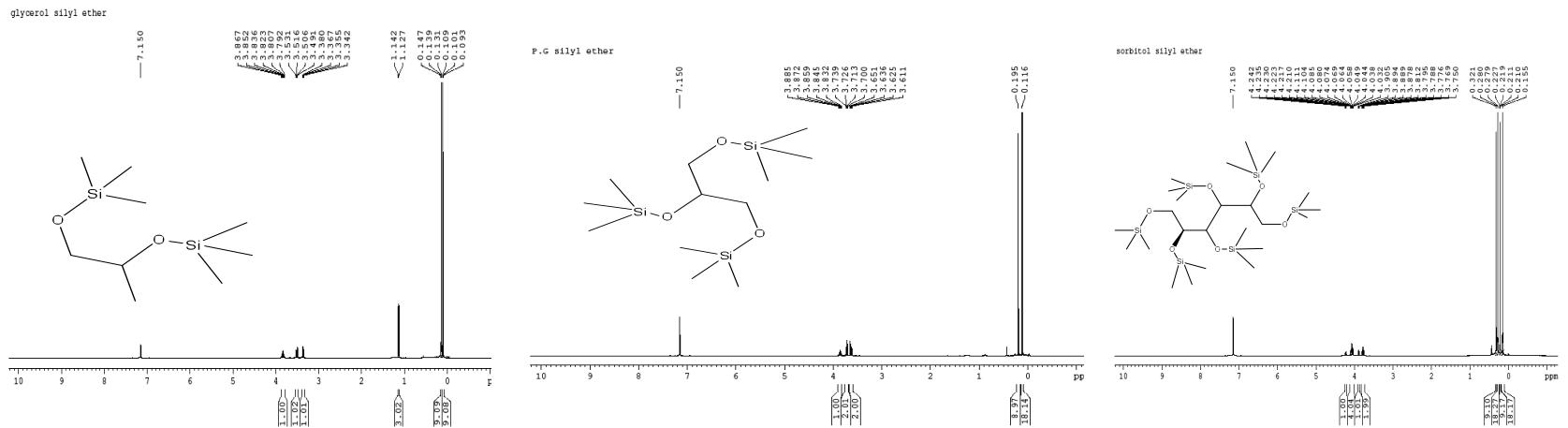
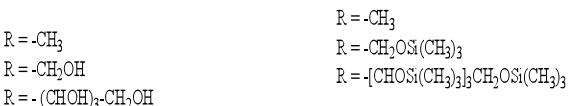
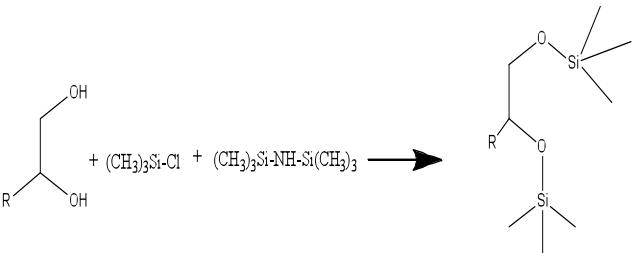
METHODS REPORTED IN THE LITERATURE

Method	Reference	Limitations
A	AOAC Official Methods of Analysis (1995), Ch 3, p.3	<ul style="list-style-type: none"> Only Propylene glycol and Glycerol can be analyzed.
B	Beitrage Zur Tabakforschung. Band 6 . Heft 2. 1971	<ul style="list-style-type: none"> Sorbitol analyzed separately Laborious extraction procedure  <p>The figure consists of two side-by-side gas chromatograms. The left chromatogram, labeled 'Figure 1. Gas-chromatogram of pure TMS-glycol.', shows several sharp peaks at different retention times. The right chromatogram, labeled 'Figure 2. Gas-chromatogram of pure TMS-sorbitol.', shows a single prominent peak at a specific retention time. Both chromatograms have temperature scales (100°C, 110°C, 120°C, 130°C, 140°C, 150°C, 160°C, 170°C, 180°C) and 'Isotherm' labels.</p>
C	Journal of Chromatography, 140 304-309 (1977)	<ul style="list-style-type: none"> Pyridine used for extraction. Only Propylene glycol and Glycerol can be analyzed.
D	CORESTA Recommended method N° 61	<ul style="list-style-type: none"> Separation of Glycerol, Propylene glycol and Sorbitol.  <p>This is a gas chromatogram showing the separation of humectants in snus. The x-axis represents retention time in minutes, ranging from 0 to 8. The y-axis represents relative intensity from 0 to 100%. The chromatogram displays several distinct peaks corresponding to different components. The peaks are labeled with their respective retention times: 4.0 min (Glycol), 5.0 min (Sorbitol), 6.0 min (Glycerol), 7.0 min (Propylene glycol), and 8.0 min (Trehalose).</p>

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

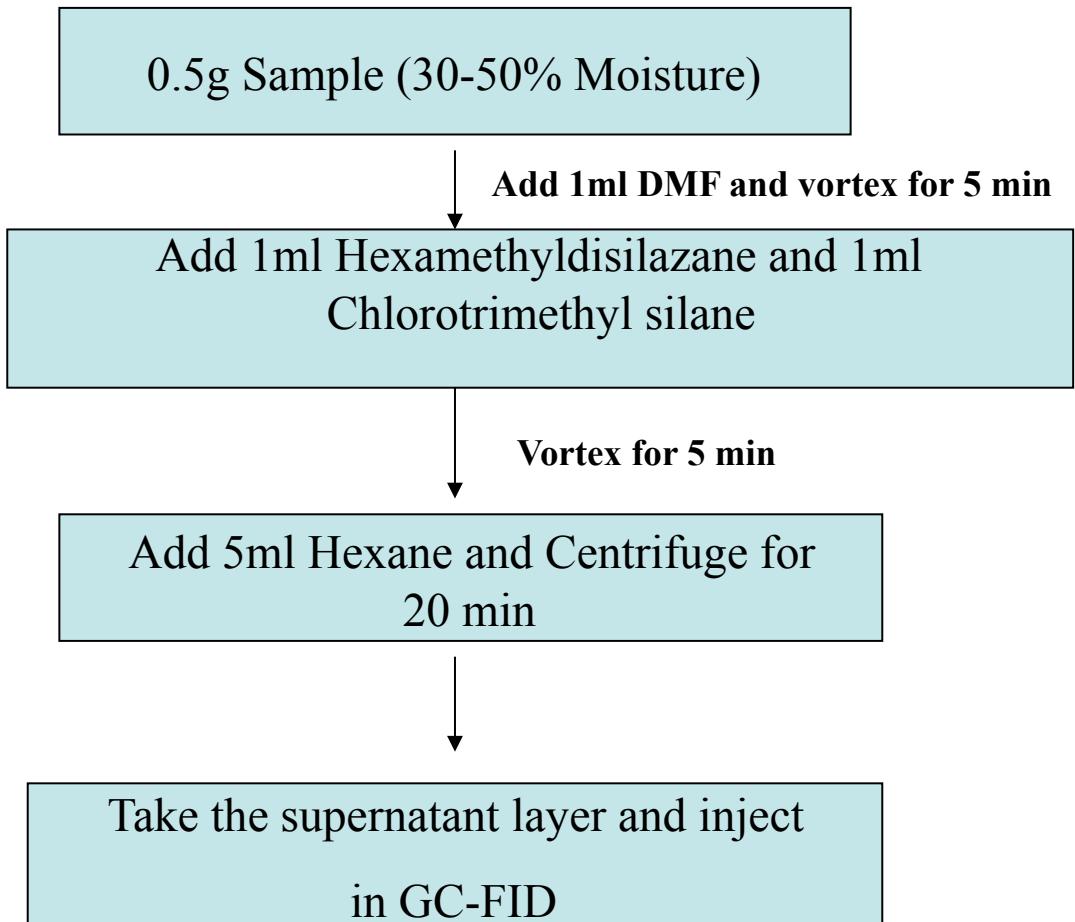
PRINCIPLE OF THE METHOD

The humectants (Propylene glycol, Glycerol & Sorbitol) are converted into their trimethylsilyl ether derivatives and these silylethers have less polarity, making them more volatile compounds.



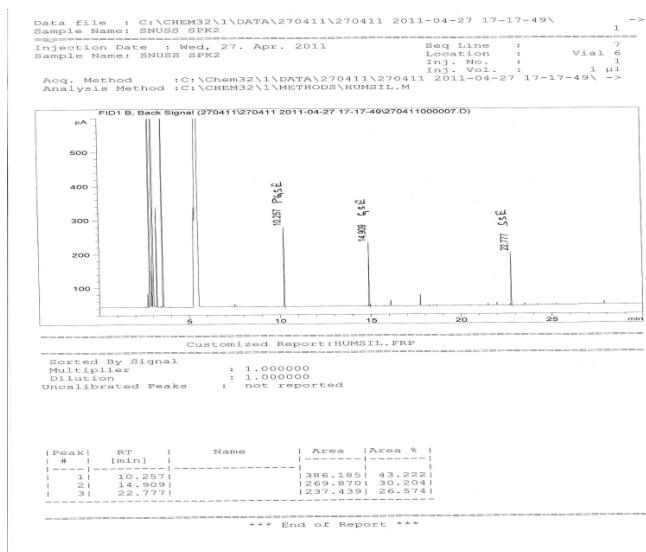
^1H NMR OF 1,2 - PROPYLENE GLYCOL, GLYCEROL & SORBITOL SILYL ETHER

FLOW DIAGRAM

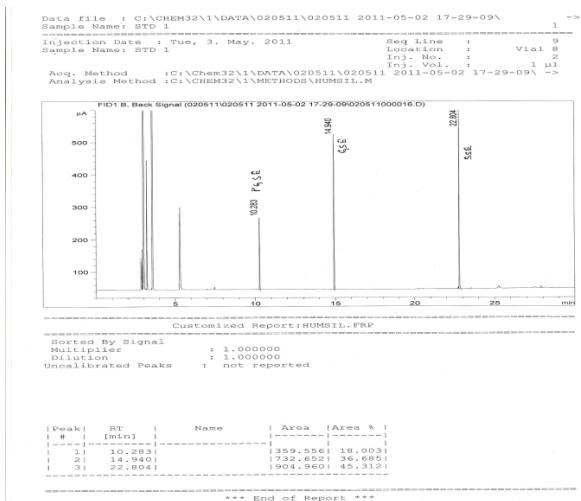


GC-FID METHOD CONDITION

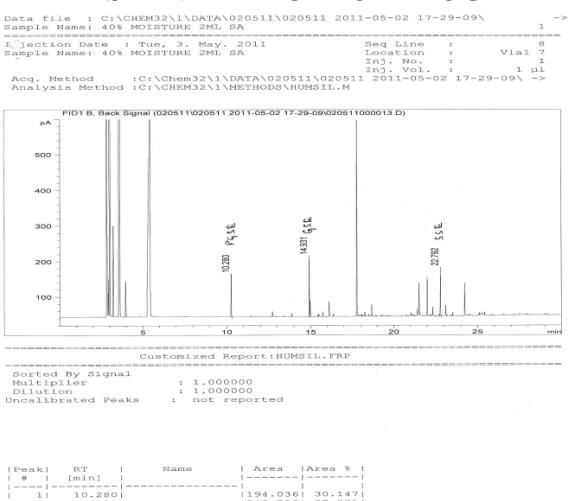
Column : DB-5
 Dimension : 30 m X 0.25 mm X 0.25 micron
 Flow : 1.0 ml/min
 Injector Temperature : 250° C
 Detector Temperature: 280° C (FID)
 Oven : Initial 60° C hold 3 min
 Ramp1 increase @ 10° C per min to 280° C hold for 3 min
 Split ratio : 100:1
 Injection volume : 1 μ l
 Run Time : 30 minutes



SPIKE SAMPLE CHROMATOGRAM



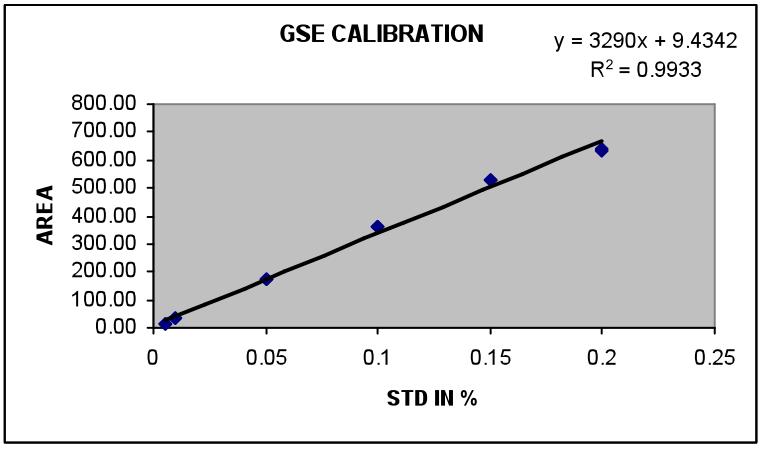
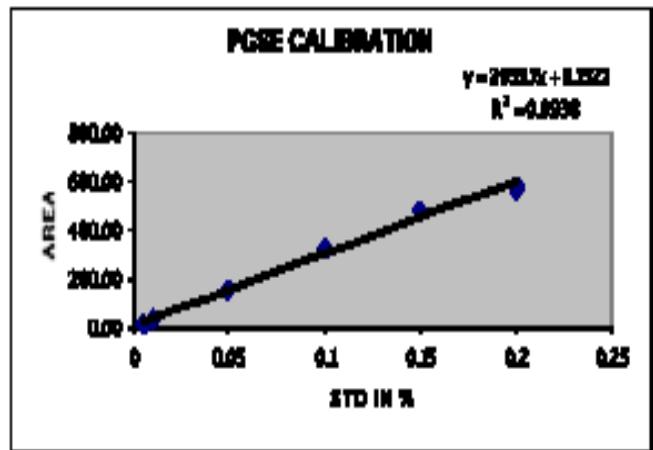
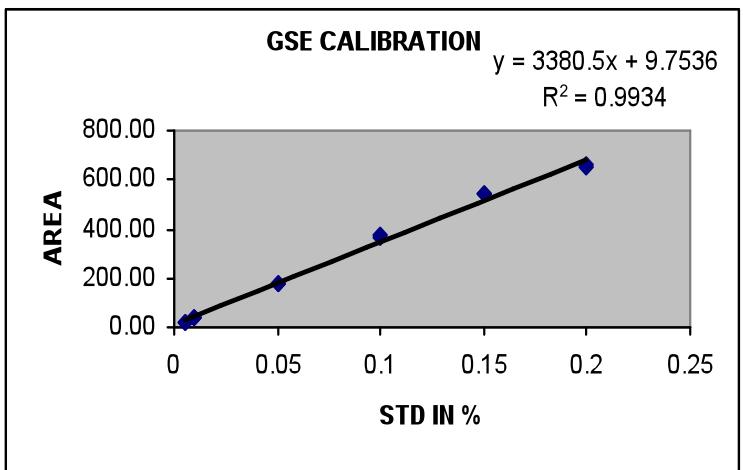
STANDARD CHROMATOGRAM



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

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

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

% RSD	1.36
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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

% RSD	1.44
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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

% RSD	1.69
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RECOVERY STUDIES

LOW SPIKE RECOVERY: SPIKED AMOUNT 1%

PROPYLENE GLYCOL		
TRIALS	OBTAINED	% RECOVERY
1	0.97	97
2	0.96	96
3	0.98	98
% RSD	1.03	

GLYCEROL		
TRIALS	OBTAINED	% RECOVERY
1	1.01	101
2	1.00	100
3	1.03	103
% RSD	1.51	

SORBITOL		
TRIALS	OBTAINED	% RECOVERY
1	0.93	93
2	0.93	93
3	0.95	95
% RSD	1.23	

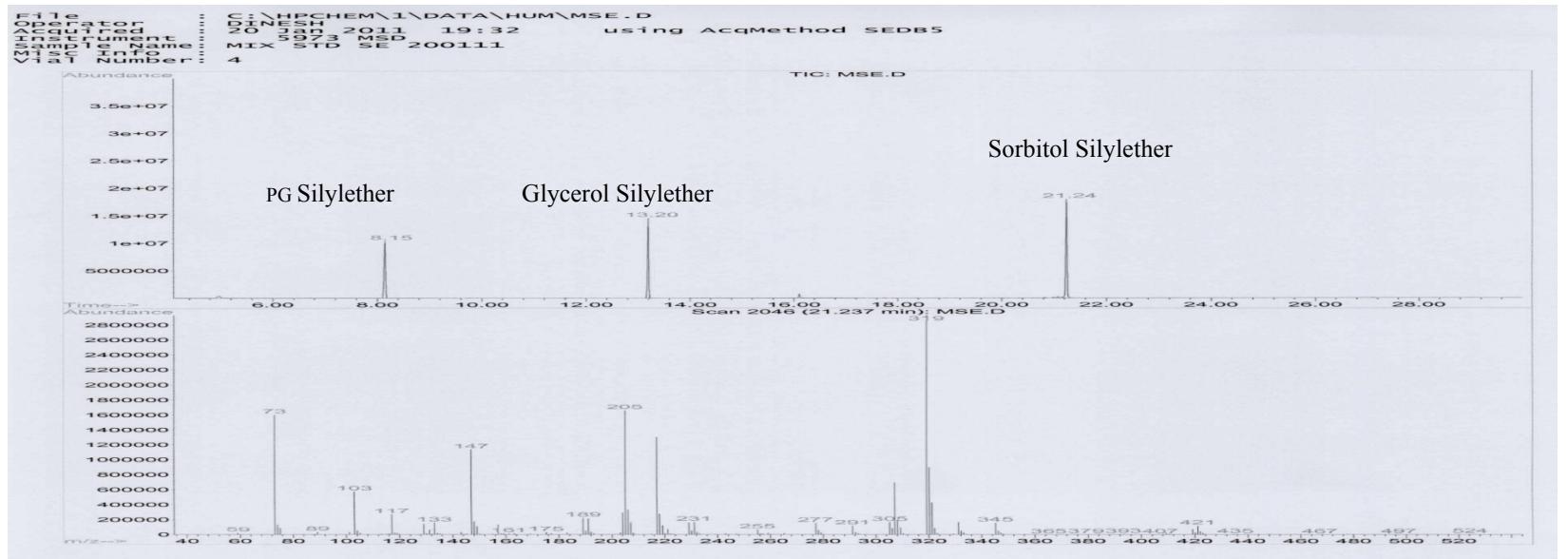
HIGH SPIKE RECOVERY: SPIKED AMOUNT 2%

PROPYLENE GLYCOL		
TRIALS	OBTAINED	% RECOVERY
1	1.96	98
2	1.94	97
3	2.01	101
% RSD	1.83	

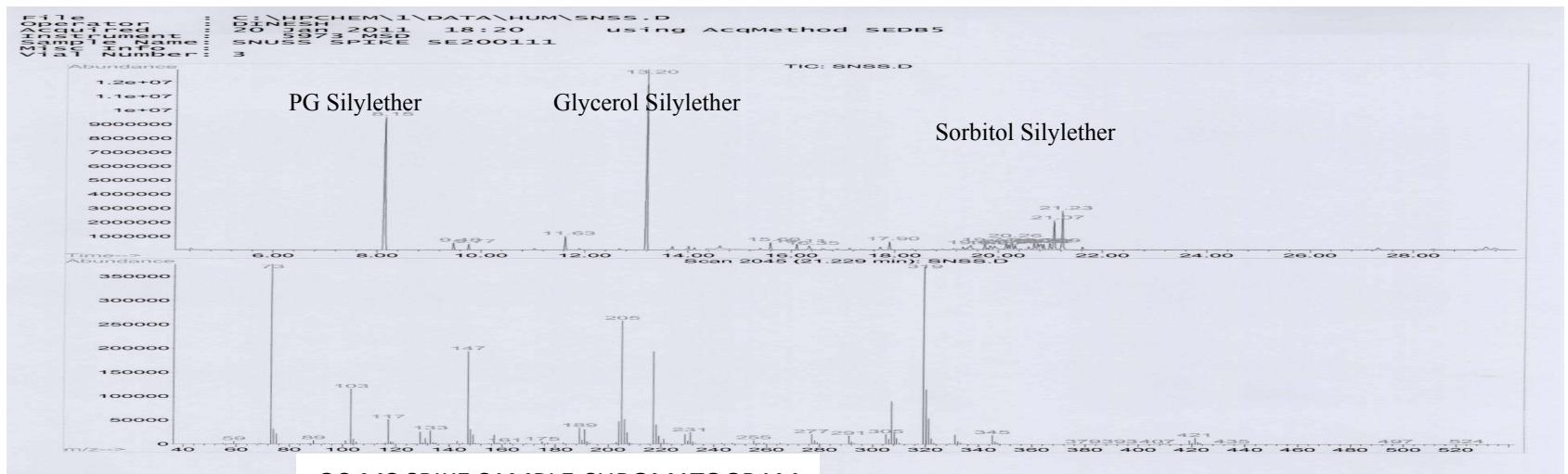
GLYCEROL		
TRIALS	OBTAINED	% RECOVERY
1	2.00	100
2	1.97	99
3	2.03	102
% RSD	1.50	

SORBITOL		
TRIALS	OBTAINED	% RECOVERY
1	1.90	95
2	1.91	96
3	1.97	99
% RSD	1.97	

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