QUANTIFICATION OF CITRATE SALT IN CIGARETTE PAPER BY HIGH PERFORMANCE LIQUID CHROMATOGRAPHY

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ABSTRACT

Citrate salts present in cigarette paper are converted to citric acid by dissolving the cigarette paper in 0.1% H_3PO_4 and quantified as % Citric acid using HPLC-UV/VIS.

INTRODUCTION

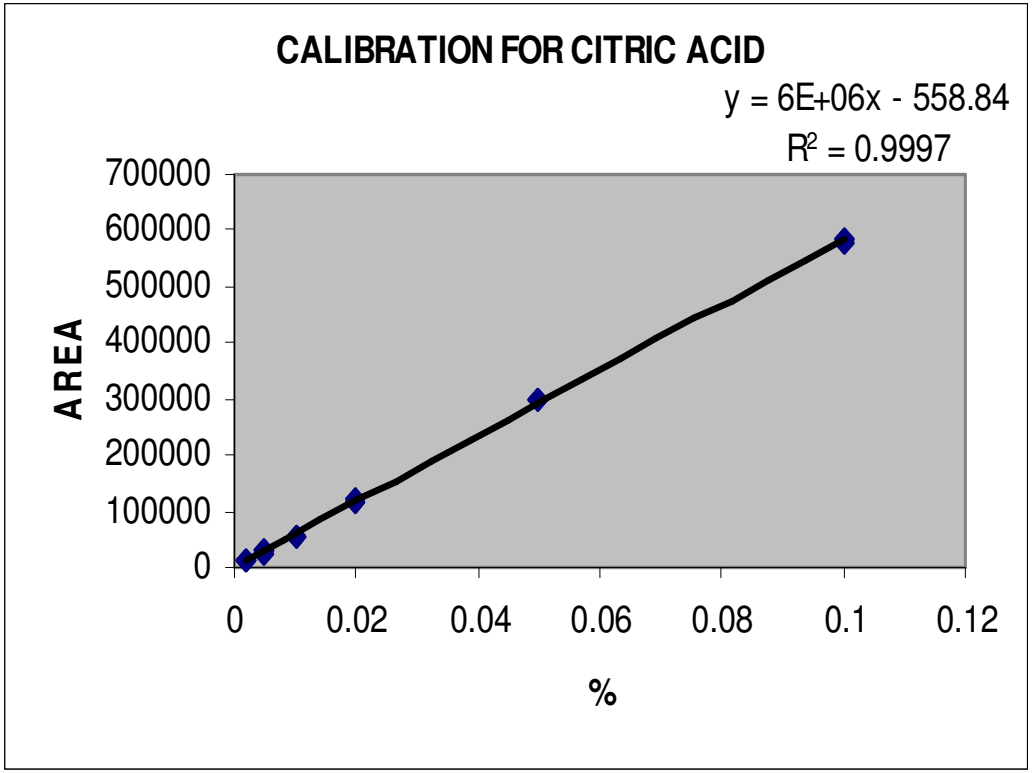
- Alkalimetal salts of Organic acids can be applied as burn modifiers to the paper.
- Levels of these additives are typically between 0.5% and 3% of the paper weight.
- These additives also play a role in taste and flavour of the smoke.
- The alkalimetal salts provide a whiter ash and increase static burn rates.
- Citrate is usually added to cigarette paper as trisodium salt or as tripotassium salt or as mixture of trisodium and tripotassium salts..
- Citrate salts are quantified to monitor the quality of the cigarette paper.
- There are several methods for quantification of citrate in cigarette paper reported in the literature namely titration method and enzymatic method.
- All the methods involve lengthier and laborious procedures.
- Hence we have developed a simple and fast method for the analysis of citrate in cigarette paper.

LITERATURE METHODS

METHOD	KEY FEATURES	LIMITATIONS
1) Determination of Citrate in Cigarette paper by Volumetric analysis	Citrate is determined by extracting cigarette paper and titrating the aqueous extract with standard potassium permanganate solution.	• Manual titration • Subjective • Manual error
2) Determination of Citrate in Cigarette paper by citrate lyase (enzyme)(CORESTA METHOD N^{O} 34)	• Citrate is converted to oxaloacetate and acetate in the reaction catalysed by citrate lyase. • In the presence of the enzyme malate dehydrogenase and lactate dehydrogenase, oxaloacetate and its decarboxylation product pyruvate are reduced to l-malate and l-lactate respectively, by reduced nicotinamide adenine dinucleotide (NADH). The amount of NADH oxidised in the reaction is stoichiometric with the amount of Citrate. NADH is determined by means of its absorbance at 340 nm.	• Expensive • Time consuming • Stability of enzyme
3) Determination of citrate in cigarette paper using conductivity	• Citrate is determined in cigarette paper by measuring the conductivity of citric acid.	• Not Specific

LINEARITY

STD (%)	AREA
0.002	12225
0.002	12066
0.002	12367
0.005	27299
0.005	28892
0.005	28917
0.01	54876
0.01	53609
0.01	53670
0.02	114746
0.02	119662
0.02	116708
0.05	297661
0.05	297130
0.05	297139
0.1	580722
0.1	583016



REPEATABILITY STUDIES

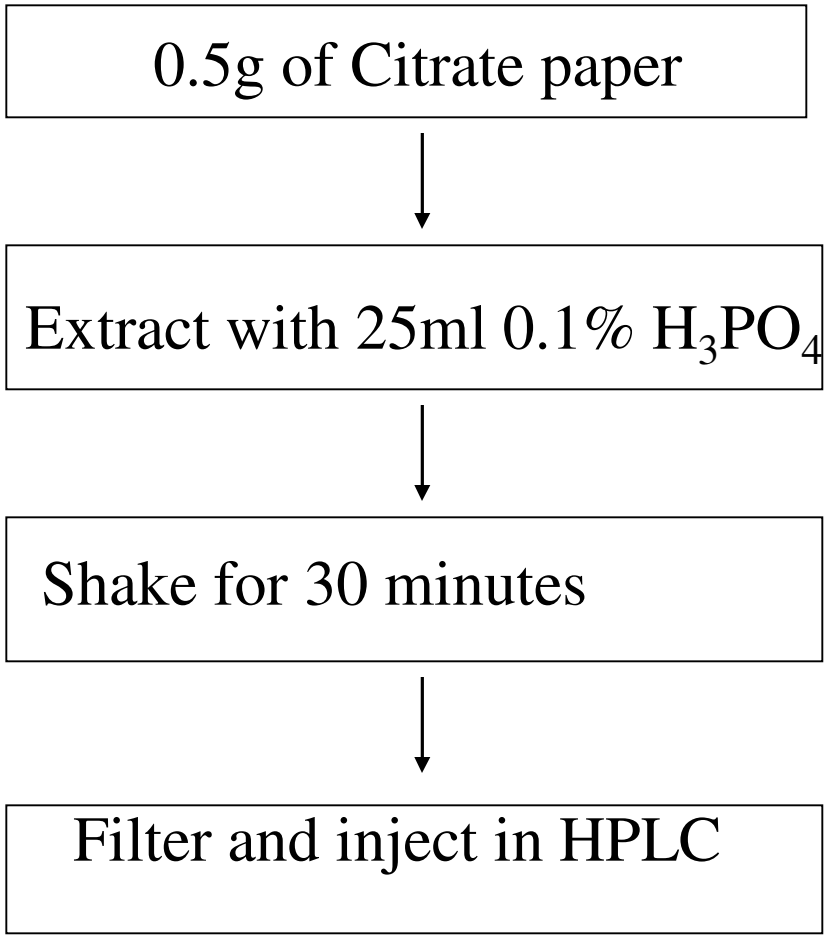
TRAIL NO.	SAMPLE	CITRIC ACID %
1	Citrate paper	1.16
2	Citrate paper	1.18
3	Citrate paper	1.17
4	Citrate paper	1.14
5	Citrate paper	1.18
6	Citrate paper	1.16
	MEAN	**1.17**
	%RSD	**1.30**

HPLC CONDITIONS

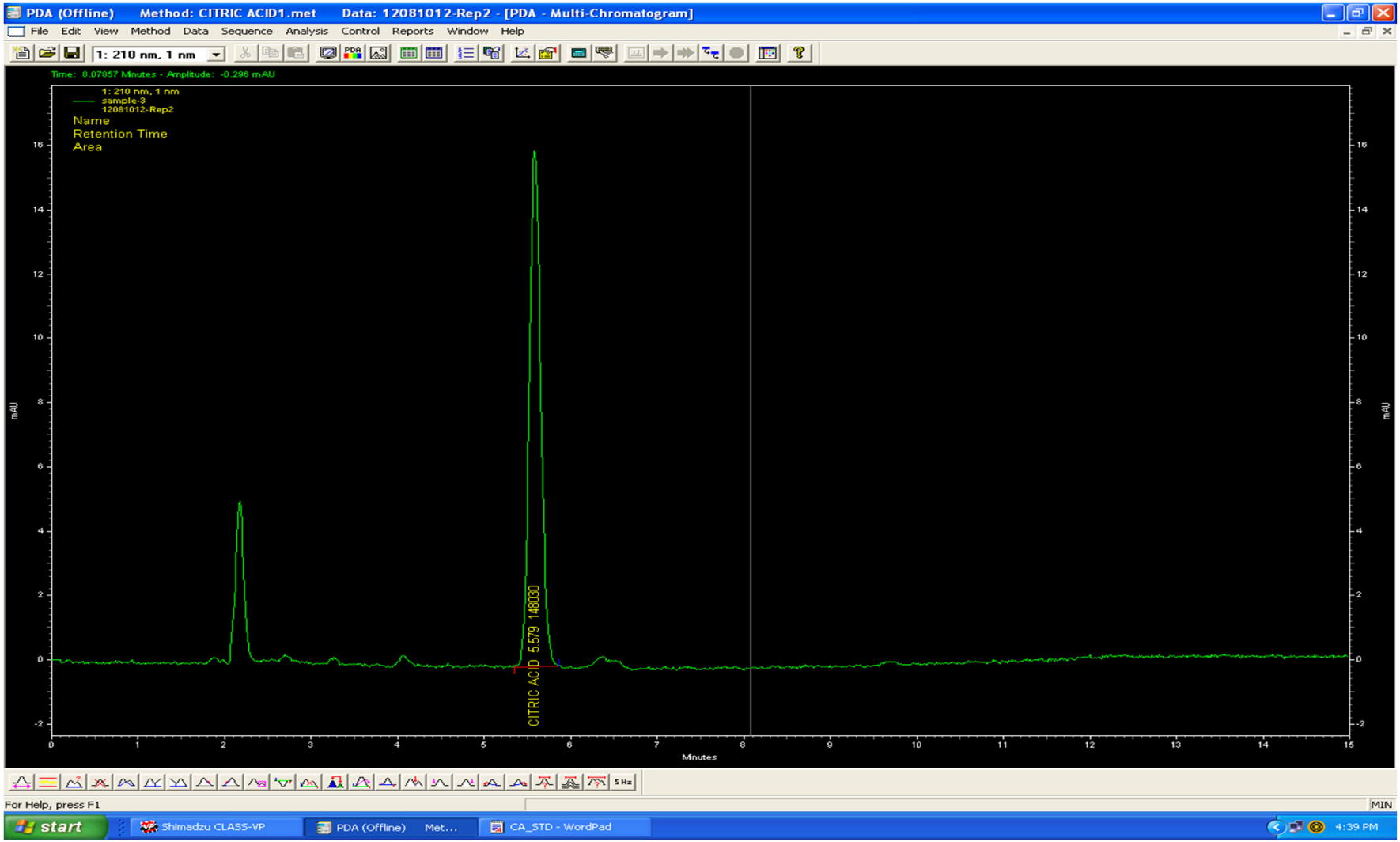
MOBILE PHASE: 0.1% Orthophosphoric acid
COLUMN: Lichrospher RP-18e 250mm X 4mm X 5um
COLUMN FLOW: 1ml/min
COLUMN TEMPERATURE: 40^0 C
DETECTOR WAVE LENGTH : 210 nm
INJECTION VOLUME: 10 ul

OUR APPROACH

SAMPLE PREPARATION



SAMPLE CHROMATOGRAM

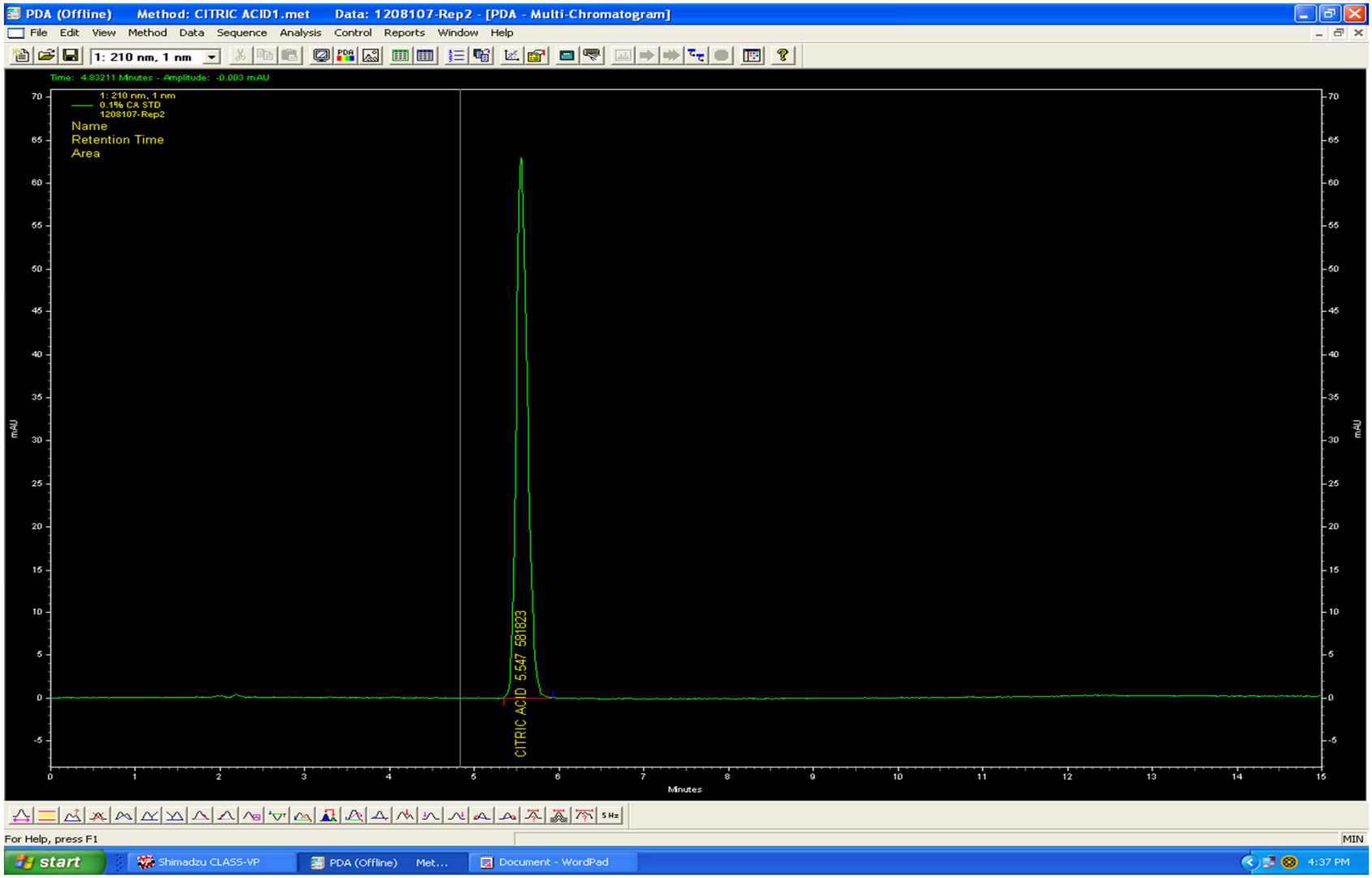


REPRODUCIBILITY STUDIES

ANALYST-1		
TRAIL NO.	**SAMPLE**	**CITRIC ACID %**
1	Citrate paper	1.16
2	Citrate paper	1.18
3	Citrate paper	1.17
4	Citrate paper	1.14
5	Citrate paper	1.18
6	Citrate paper	1.16
ANALYST-2		
TRAIL NO.	**SAMPLE**	**CITRIC ACID %**
1	Citrate paper	1.14
2	Citrate paper	1.18
3	Citrate paper	1.16
4	Citrate paper	1.17
5	Citrate paper	1.16
6	Citrate paper	1.18
	MEAN	**1.17**
	%RSD	**1.24**

LOQ: Limit of quantification is found to be 0.2%

STANDARD CHROMATOGRAM



COMPARISON OF CITRATE CONTENT ANALYZED AT DIFFERENT LABS

Citrate content in Cigarette paper in %

SAMPLES	LAB-1	LAB-2	LAB-3
CITRATE PAPER-1	1.13	1.07	1.21
CITRATE PAPER-2	0.85	0.94	1.03
CITRATE PAPER-3	1.97	1.99	2.02

LABS	METHOD
1	Conductimetric
2	Volumetric Titration
3	HPLC

CONCLUSION

- HPLC method was optimized for analysis of citrate in cigarette paper.
- Recovery studies have been done and found to be greater than 95%.
- Repeatability studies have also been done and RSD is found to be less than 2%.
- Limit of quantification is 0.2% w.r.t sample.
- Interlab results indicate that the method is rugged, accurate and reliable for the analysis of citrate in cigarette paper.