Doc. No. PCD 18 (22269) WC
April 2023

BUREAU OF INDIAN STANDARDS

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Draft Indian Standard

OIL OF LEMONGRASS — SPECIFICATION

(*Third Revision of* IS 327)

(ICS No. 71.100.60)

Fragrance and Flavour Sectional Committee	Last date for comment is
PCD 18	**09 June 2023**

FOREWORD

(*Formal clauses shall be added later*)

In India, oil of lemongrass is produced in the South-Western State of Kerala by the distillation of lemongrass (*Cymbopogon flexuosus* Stapf family Poaceae). This essential oil produced largely in India since ages has come to be known as the East Indian Oil of Lemongrass and has found favor in the world market particularly because of its uniformly high citral content and the solubility in 70 percent alcohol of most of the oil produced in the country. This solubility in alcohol is regarded as synonymous with freshness and freedom from adulterants; though investigations have disclosed that some specimens of freshly distilled oil are also found to be insoluble in alcohol, if there is adventitious mixture, even in small proportions, with a similar but botanically different white-stemmed grass, locally known as '*wella poolu*' found in the fields. The white-stemmed grass now identified as *Cymbopogon flexuosus* Stapf Var. *albescens* has, however, been almost rooted out of the lemongrass fields by intensive efforts of the State Departments of Agriculture and Forests. The solubility of all specimens of the oil is known to be reduced on storage.

This standard (Third Revision) was first published in 1952 and subsequently revised in 1961 and 1991. The Sectional Committee responsible for its preparation felt that the standard should be revised with a view to bring it in line with trade practices prevalent in perfumery technology and also to align the quality level of material currently being produced, sold in the country and exported.

In this (third) revision, the values for Refractive Index and Relative Density have been reported at 20°C in addition to 27°C in order to facilitate international trade, since the fragrance and flavor ingredients are traded at international level quoting the relative density and refractive index at 20ºC rather than at 27ºC as prescribed in the Indian standards. Besides, the requirement for flash point and Simultaneous Thermogravimetric Analysis (STA) have been incorporated in this revision to detect the presence of common adulterants such as castor oil, liquid paraffin, vegetable oils, etc. The Citral percent has also been updated in this revision.

This revision represents the oil quality parameters of some of the notable species like *C. flexuosus, C. pendulus, C. khasianus*, etc cultivated in different parts of India.

The composition of the Committee, responsible for the formulation of this standard is given at Annex C. (*will be added later*)

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 2022 'Rules for rounding off numerical values (*second revision*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1 SCOPE

This standard prescribes the requirements and the methods of test for the material commercially known as the oil of lemongrass or the East Indian Oil of lemongrass. The material is largely used in the extraction of citral, the chief constituent of the oil and the starting material for the manufacture of important ionones. It is also employed in the preparation of lemon-like perfumes, germicides, etc.

2 REFERENCES

The following Indian Standards contain provisions which, through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. The following Indian Standards are necessaryadjuncts to this standard:

IS. No.	*Title*
326	Methods of sampling and test for natural and synthetic perfumery materials:
(Part 1): 2022	Sampling (*Third Revision*)
(Part 2): 1980	Preliminary examination of perfumery materials and samples (*Second Revision*)
(Part 3): 2006	Relative density (*Third Revision*)
(Part 4): 2005	Determination of optical rotation (*Third Revision*)
(Part 5): 2006	Determination of refractive index (*Third Revision*)
(Part 6): 2005	Determination of solubility (*Third Revision*)
(Part 7): 2006	Determination of acid value (*Third Revision*)
(Part 8): 2005	Determination of ester value, content of esters and combined alcohols (*Third Revision)*
(Part 9/Sec 1) : 2017	Part 9 determination of ester value and free alcohols section 1 determination of ester values, before and after acetylation, and evaluation of the contents of free and total alcohols (*Third Revision*)
(Part 9/Sec 2) : 2017	Part 9 determination of ester value and free alcohols section 2 estimation of free alcohols content by determination of ester value after acetylation (*Third Revision*)
(Part 11/Sec 1) : 2017	Part 11 determination of carbonyl value section 1 free hydroxylamine method (*Third Revision*)
(Part 11/Sec	Part 11 determination of carbonyl value section 2 potentiometric

2) : 2017	methods using hydroxylammonium chloride (*Third Revision*)
1070: 1992	Water for general laboratory use (*Third Revision*)
2284: 1988	Methods for olfactory assessment of natural and synthetic perfumery materials *(First Revision*)
6597: 2001	Glossary of terms relating to natural and synthetic perfumery materials *(Second Revision)*
ISO/ TR 11018 : 1997	Essential oils — General guidance on the determination of flashpoint

3 TERMINOLOGY

For the purpose of this standard definitions given in IS 6597 shall apply.

4 SAMPLING

Representative samples of the material shall bedrawn as prescribed in IS 326 (Part 1).

5 REQUIREMENTS

5.1 Description

Oil of lemongrass shall be obtained by hydro or steam distillation of the freshly cut and partially dried grass which belongs to the genus *Cymbopogon* from Poaceae family.

5.1.1 Oil of lemongrass shall be a clear liquid, free from sediment, suspended matter, separated water and added adulterants.

5.1.2 The oil shall be examined for its color clarity separated water, by-notes and sediment as prescribed in IS 326 (Part 2).

5.2 SOLUBILITY

Oil of lemongrass shall be soluble in 3 volumes of ethyl alcohol (70 percent by volume), occasionally with slight turbidity, when tested as prescribed in IS 326 (Part 6): 2005.

5.3 Oil of lemongrass shall also comply with the requirements given in Table **1**.

Table 1 Requirement for Oil of Lemongrass
(*Clause* 5.3)

SI No.	Characteristics	Oil of lemongrass	Method of Test Ref to
(1)	**(2)**	**(3)**	**(4)**
i)	Appearance and color	Clear, mobile liquid, Pale yellow to yellowish brown	IS 326 (Part 2)
ii)	Odour	Strong, lemon/ citrus like	IS 2284
iii)	Relative Density(1)		IS 326 (Part 3)
	at 20°C	0.890-0.988	
	at 27°C	0.8851 (*Min*), 0.8895 (*Max*)	
iv)	Refractive Index(2)		IS 326 (Part *5*)

	at 20°C	1.482-1.490	
	at 27°C	1.4817 (*Min*), 1.4851 (*Max*)	
v)	Optical rotation, $[\alpha]_D^{20}$	(-) 4.0 – (-) 0.5	IS 326 (Part 4)
vi)	Citral content, percent by volume	*min.* 66.4 *max* 87.7	Annex A
vii)	Flash Point	(+) 87.8 ºC	ISO/ TR 11018
viii)	STA analysis	>99% weight loss at ≤ 250 ºC	Annex B

NOTES –

1 The correction factor for relative density for each degree Celsius change in temperature is 0.00062.
2 The correction factor for refractive index for each degree Celsius change in temperature is 0.00044.

6.2 Number of Tests

Tests for the determination of all the characteristics shall be conducted on the composite sample.

6.3 Criteria for Conformity

The lot shall be considered as conforming to the specification if the composite sample satisfies all the requirements specified in Table **1**.

7 PACKING AND MARKING

7.1 Packing

7.1.1 The material shall be supplied in well-closed containers as agreed to between the purchaser and the supplier.

7.1.2 The material shall be well protected from light and stored in a cool place.

7.2 Marking

Each container so filled shall be clearly marked with the following information:

6.2.1 *Name of the material, indicating the source of manufacture;*

6.2.2 *Name of Manufacturer*

6.2.3 *Net and Gross Mass of the Material, including percentage of total Citral content;*

6.2.4 *Net Volume of the Material*

6.2.5 *Batch no.*

6.2.6 *BIS Certification Marking*

The containers may also be marked with the Standard Mark.

NOTE — The use of the Standard Mark is governed by the provisions of the *Bureau of Indian Standards Act, 2016* and the Rules and Regulations made thereunder. Details of conditions under which a licence for the use of Standard Mark may be granted to manufactures or producers, may be obtained from the Bureau of Indian Standards

8 TEST METHODS

8.1 Tests shall be conducted as prescribed in clause **5.1.3** and column **4** of Table **1**.

8.2 QUALITY OF REAGENTS

Unless specified otherwise pure chemicals, solvents and distilled water (*See* IS 1070) shall be used.

NOTE - 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

8.3 SYRINGE TYPE

A 0.5 to 1.0 micro liter volume syringe shall be used for manual injection of essential oils obtained from both the types of vetivers. For analysis where an auto sampler is used for sample injection, an equivalent and autosampler compatible syringe shall be used for sample injection. Syringe cleaning with non-polar solvents like *n*-hexane is recommend before and after the sample injection.

ANNEX A

[*Clause* 5.3, *Table* 1, *Sl.No.*(vi)]

CAPILLARY GAS CHROMATOGRAPHIC ANALYSIS OF OIL OF LEMONGRASS

A-1 GENERAL

The chromatographic conditions given here are for guidance only.

A-2 OUTLINE OF THE METHOD

An essential oil sample is dissolved in a suitable solvent (for example, acetone, cyclohexane, *n*-hexane) and is injected into the heated injector (split/splitless) of a gas chromatograph coupled with fused silica capillary column coated with 5% diphenyl and 95% dimethylpolysiloxane as stationary phase. The sample vapors travel along with the carrier gas or mobile phase by the mechanism of adsorption and desorption phenomena from one end of the capillary column to the other end connected with detector (Flame Ionization Detector). During their travel through carrier gas, the constituents of the sample undergo distribution depending upon their chemical affinity at different rates and ultimately get separate from one another. The separated constituents emerge from the detector end of the column one after another and are detected by suitable means whose response is related to the amount of a specific component leaving the capillary column. The amplified detector

signals are recorded and stored in a computer-controlled software programme. Furthermore, the area corresponds to each baseline separated peak is reported as the area percent of each constituent present in the oil sample.

A-3 APPARATUS

A-3.1 Any suitable capillary gas chromatograph and column (non polar) capable of being operated under conditions suitable for resolving the individual constituents into distinct peaks may be used. The typical chromatograms for oils of lemongrass with the following chromatographic conditions are shown in Fig. **1** and **2**.

Table 2 Column operating conditions

Sample size	About 0.1 µl (diluted in hexane/ DCM)
Capillary column:	5% diphenyl in polydimethyl siloxane
Material	polydimethyl siloxane
Length	30 m
Internal diameter	0.25 mm
Film thickness	0.25 µm
Carrier gas	Hydrogen (1 ml/min)
Flow split ratio	1: 40
Injector Type	split/splitless
Injection temperature	280°C
Detector:	
Type	Flame ionization detector
Temperature	280°C
Oven Temperature Programming:	
Temperature 1	60°C
Ramp 1	3°C/min
Temperature 2	240°C isothermal for 2 min.
Ramp2	5°C/min
Temperature 3	280°C

Table 3 Chromatographic Profile[1)]

Peak No.	**Component[2)]**	**Percent[3)]**	
		Min.	Max.
1	6-Methyl-5-heptene-2-one	0.3	2.4
2	Limonene	0.2	0.9
3	Linalool	0.3	1.5
4	Neral	29.2	35.8

5	Geraniol[4)]	0.6	3.2
6	Geranial	37.2	51.9
7	Geranyl acetate	0.5	4.7
8	ß-Caryophyllene	0.3	1.3
9	Caryophyllene Oxide	0.2	1.8

NOTES –
1 The chromatographic profile is normative, contrary to typical chromatograms given for information in Annex A.
2 Components are listed according to their elution order on a non-polar column (*see* Fig **1**).
3 Area percent values are based on non-polar column data (*see* Fig **1**).
4 Co-elution with piperitone in 5% diphenyl Fused Silica Capillary Column is observed.

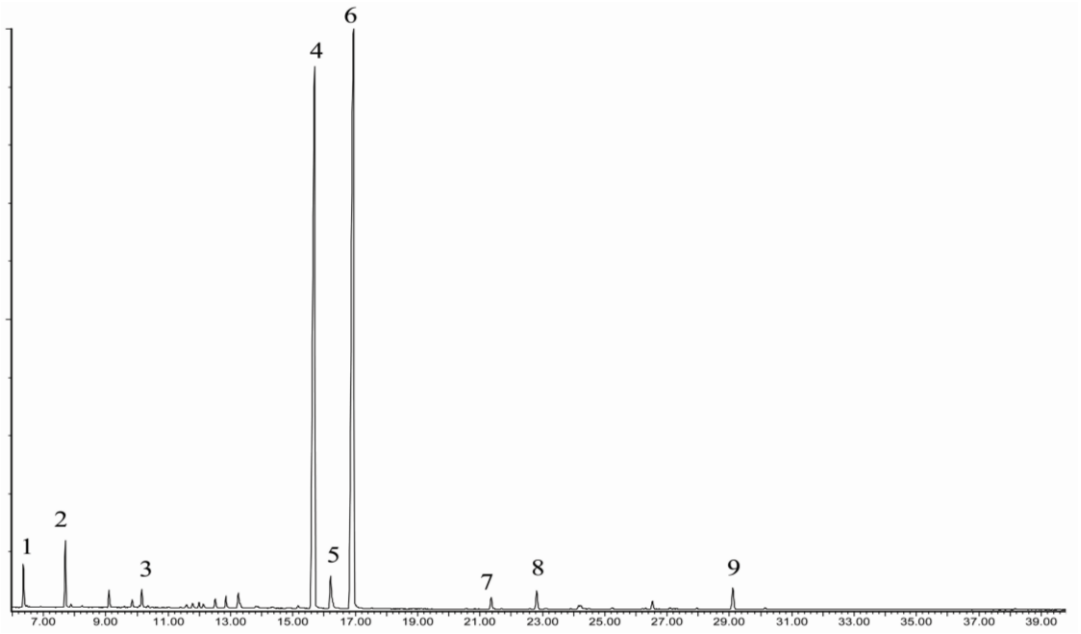

Fig 1 Typical Chromatogram of Oil of lemongrass in 5% diphenyl stationary phase

ANNEX B
[*Clause* 5.3, *Table* 1, *Sl.No*. (viii)]
SIMULTANEOUS THERMOGRAVIMETRIC ANALYSIS

C.1 GENERAL

Essential oil is frequently adulterated in recent times. Some of the most common adulterants are castor oil, liquid paraffin, vegetable oils, etc. For the quality control comparison, pure essential oil, one high boiler adulterant (coconut oil as adulterant), and one deliberately spiked lemongrass essential oil sample (15% with coconut oil) was subjected to Thermogravimetric Analysis (*fig* **2-4**).

It was concluded by a comparative study that the weight loss measurement can provide vital information about the oil constitution quantitatively. The results obtained were interpreted as a function of Delta Y, which determines the percent weight loss between two temperatures on a

Thermo-Gravimetric Analysis (TGA) curve. The oil constitution is depicted as per the following points:

a) Complete mass loss (Delta Y = 98.45%) of Lemongrass essential oil is observed at 250°C, when subjected to SGA analysis and is marked by a single-stage decomposition pattern.

b) It is observed that a high boiler adulterant (Coconut Oil) can remain thermally stable up to 350°C, complete weight loss (Delta Y = 99.572%) is visible only above 500°C leading to a single-stage decomposition pattern.

c) If an adulterated sample (Oil of Lemongrass spiked with 15% Coconut Oil) is subjected to STA analysis, a well-defined two-stage decomposition pattern is observed. The first weight loss (Delta Y = 84.71%) for a 15% spiked sample is observed at 230°C for the Oil of Lemongrass (Essential Oil) and the second weight loss (Delta Y = 15.0%) is observed at 400°C for the spiked adulterant (Coconut Oil). Each weight loss corresponds to the total proportion of an individual substance that differs from another by its volatility pattern.

d) The amount of residue present (if any) is differentiated above 600°C temperature.

TABLE 4 FURNACE HEAT OPERATING PARAMETER IN STA

Sample size	0.1 µl (diluted in hexane/ DCM)
Sample weight	Approx. 10-15 mg (solid or liquid)
Sample Holder	Silica crucible
Purge gas	Nitrogen (20 ml/min)
Furnace Heat programming *:*	
Temperature 1	60°C
Ramp 1	3°C/min
Temperature 2	690°C
NOTES – 1 Every sample must be accompanied by a sample blank/ run with an empty silica crucible. 2 Weight loss is shown in percent in thermograms of lemongrass and adulterant (see *Fig* **2-4**).	

NOTE – The thermograms were obtained using a PerkinElmer STA 8000 analyzer system.

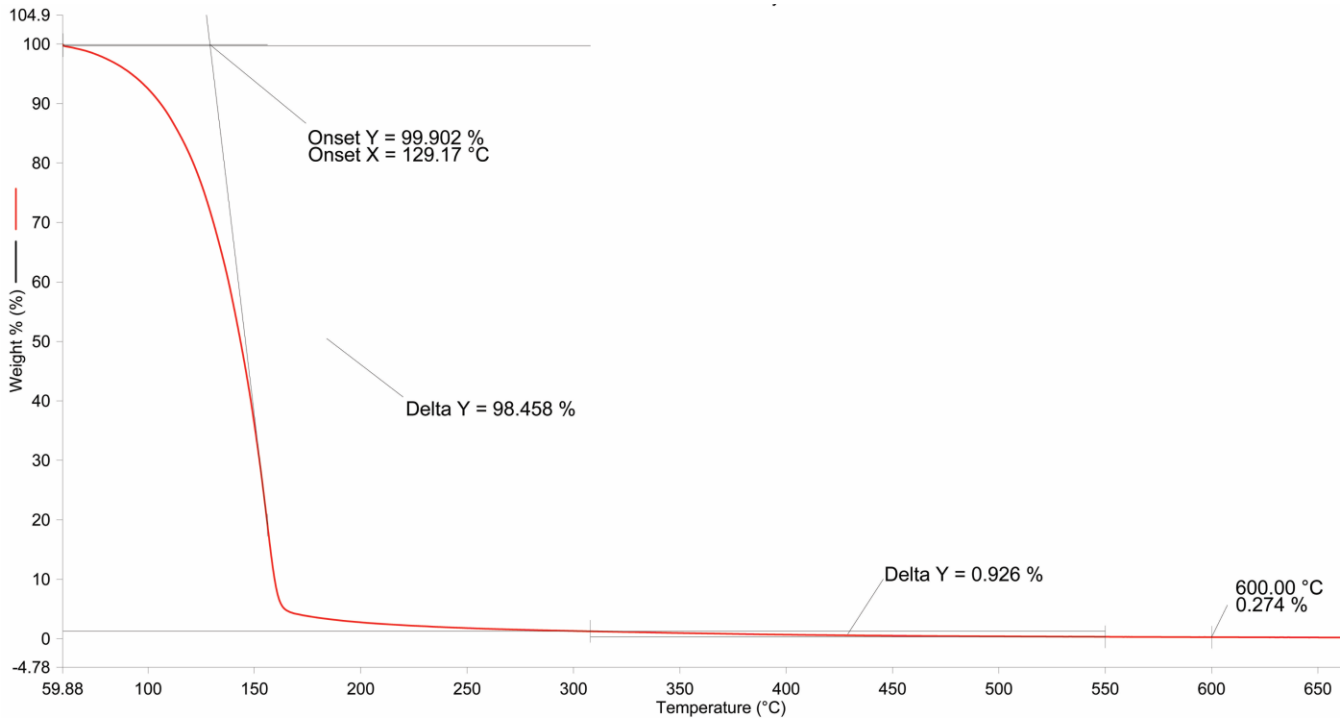

Fig 2. STA thermogram of lemongrass essential oil

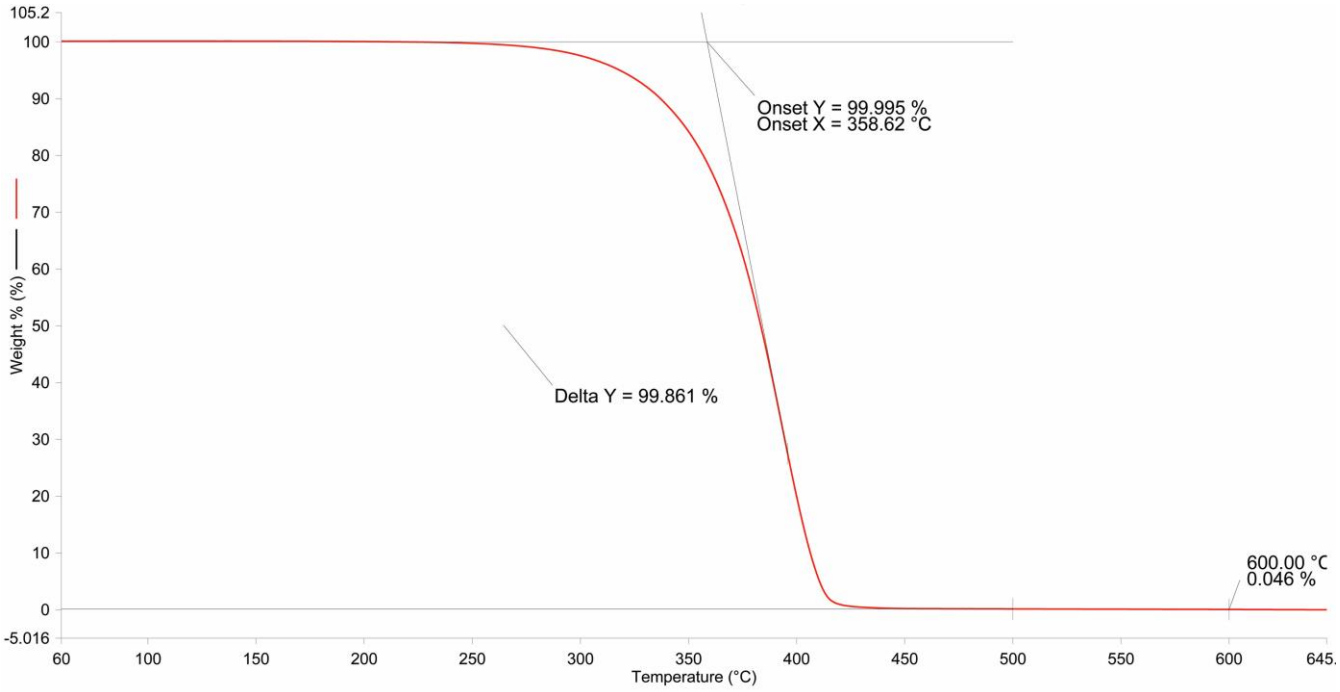

Fig 3. STA thermogram of adulterant coconut oil

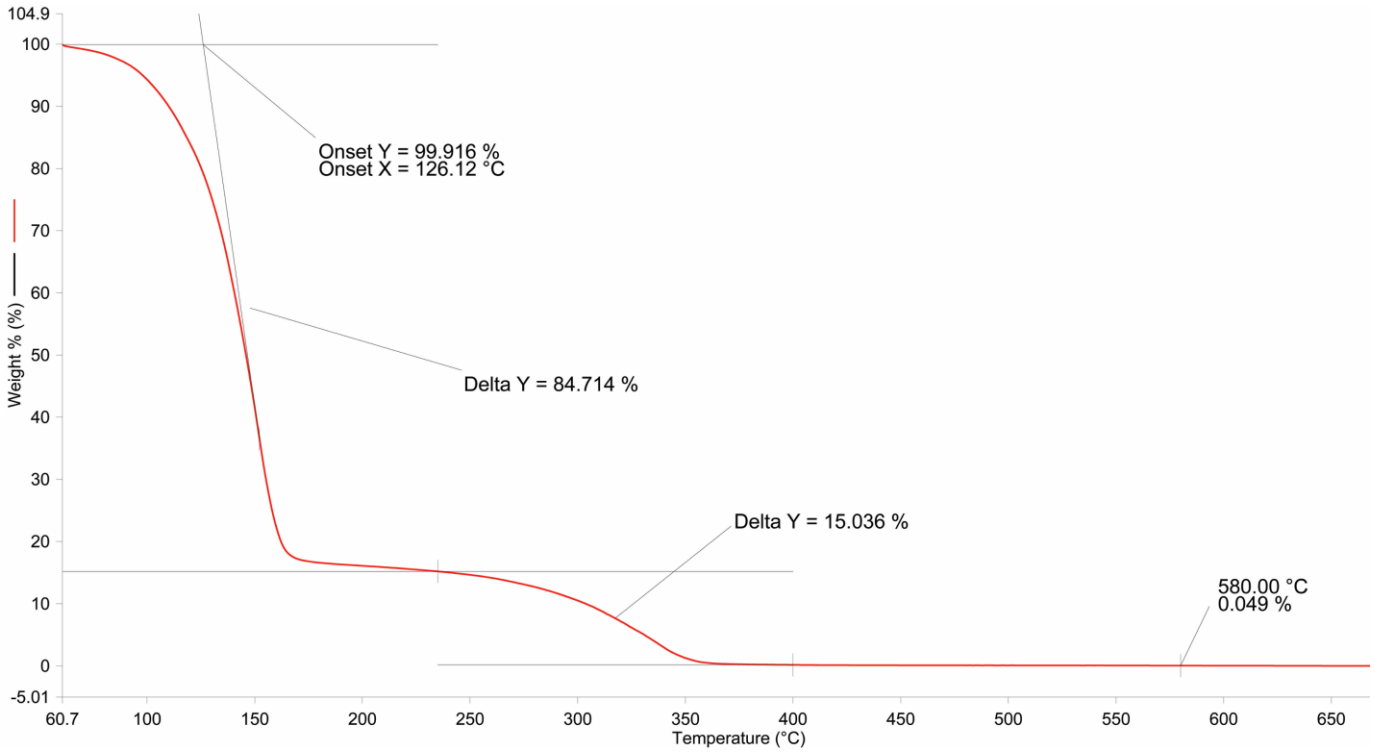

Fig 4. STA thermogram of lemongrass essential oil spiked with 15% coconut oil