Sudan University of Science and Technology

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كلية الدراسات العليا

Chemical Characterization of *Hyphaenethebaica* Fruits by GC-MS and XRF Spectroscopy

توصيف كيمائي لثمار الدوم بكروماتوغرافيا الغاز-مطايفيه الكتله وفلوره الأشعه السينيه

A Thesis Submitted inPartial Fulfillment for the requirementsofM.Sc.Degreein Chemistry

By

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إستهلال

بسم الله الرحمن الرحيم

قال تعالى:

(وَمَا أُوتِيتُم مِّنَ الْعِلْمِ إِلّا قَلِيلاً)

صدق الله العظيم

سورة الإسراء الآية 85

Dedication

To my parents,my sisters and brothers.

Acknowledgement

Praise and gratitude to AllahAlmightyfor giving me help, health, and strength to complete this work.

My appreciation and thanks to my supervisor Dr. Omer Adam Gibla for his guidance and encouragement during the performance of this work.

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Abstract

GC-MS technique was used for determination of chemical constituents of the doum fruit extract. Doum fruits Powder was analyzed by an X-ray fluorescence to determine the major available elemental constituents.

The results of GC-MS analysis showed presence of (34) chemical components. The main constituents were Tris(hydroxymethyl) nitromethane (27.67%), 3-o-methyl-D-glucose (15.33%), 5-Hydroxymethylfurfural (7.49%), dl-Glyceraldehde dimer (4.19%)and Maltol (3.24%).

XRFanalysis indicates the presence of (9) elements (K, Ca, Fe, Cu, Zn, Pb, Br, Rb and Sr).potassium showed the highest concentration (2.66%), calcium (0.373%), iron (0.00596%) and bromine (0.00447%).

Other elements were detected at trace levels, including strontium (0.00163%), cupper (0.0016%), rubidium (0.0015%), zinc (0.000549%) and lead (0.000126%).

المستخلص

في هذه الدراسه تم إستخدام تقنيه كروماتوغرافيا الغاز- ومطيافيه الكتله لتحديد المكونات الكيميائيه لمستخلص ثمار الدوم .كذلك إستخدمت تقنيه فلورة الأشعة السينيه لتقديرالمحتوى المعدني لبعض العناصر.

التحليل بكروماتوغرافيا الغاز- ومطيافيه الكتله أظهر وجود (34) مكون كيميائي.

المكونات الرئيسيه كانت Tris(hydroxymethyl)nitromethan باعلى تركيز(27.67%) ,

, (%7.49) 5-Hydroxymethylfurfural , (%15.33) 3-O-methyl-D-glucose

.(%3.34)Maltol و(%4.19)dl-Glyceraldehde dimer

التحليل بتقنيه فلورة الأشعة السينيه اشار وجود (9) عناصرشملتK, Ca, Fe,Cu,Zn, Pb, Br,Rb,Sr

البوتاسيوم أظهر أعلى تركيز (2.66%), ثم الكالسيوم(0.373%), الحديد(0.00596%) والبروم(0.00447%).

العناصر الأخرى التي أظهرت تراكيز منخفضه(traces) شملت الاسترانشيوم(0.00163%),النحاس(0.0016%),الروبيديم(0.0015%),الخارصين (0.000549%)والرصاص(0.000126%).

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Chapter One

Introduction

1-Introduction

1.1Taxonomy

Kingdom *Plantae*: Plants.

Subkingdom *Tracheobionta*: Vascular plants.

Superdivision*Spermatophyta*: Seed plants.

Division *Magnoliophyta:* Flowering plants.

Class *Liliopsida*: Monocotyledons.

Subclass: *Arecidae*.

Order: *Arecales.*

Family *Arecaceae:* Palm family.

Genus: *HyphaeneGaertn*.

Species: *H. thebaica (L.) Martius*.

1.2 Common Names

The common names of*Hyphaenethebaica*Arecaceaein Amharic (zembaba); Arabic(dom); English (gingerbread palm,Egyptiandoumpalm,doum palm);French(palmierdoum);Swahili (mkoma); Tigrigna (kambash,arkobkobai) (Orwaetal; 2009).

1.3Botanic Description

Hyphaenethebaica is a deciduous palm 10-17 m high, with a girth of 90 cm. Trunk is Y-shaped, and the tree is easily recognizable by the dichotomy of its stem forming up to 16 crowns. The bole fairly smooth but clearly showing the scars of the fallen leaves. Bark dark grey. Leaves 120 x 180 cm, fan shaped, in tufts at the ends of branches with the blade divided into segments about 60 cm long, margins entire; leaf stalk about 60 cm long, armed with curved thorns; petiole more than 1 m long, sheathing at the base with numerous upwardly curving hooks. Male and female flowers on separate trees. The inflorescence is similar in both sexes, up to 1.2 m long, with short branches at irregular intervals and 2-3 spikes arising from each branch. Male flowers shortly stalked, solitary in pits of the spadix, spathe-bracts encircling the spadix, pointed. Branches of female spadices stouter, in the fruiting stage marked by densely tomentose cushions after the fall of the fruit. The female palm produces woody fruits that persist on the tree for a long time. They are 6-10 x 6-8 cm, smooth, rectangular to cubical, with rounded edges, shiny brown when ripe, about 120 g each when fresh, 60 g when dry, each containing a single seed. Seeds 2-3.5 x 3 cm, ivory in colour, trunkate at base, apex obtuse.Hyphaene is derived from the Greek word 'hyphaino' (web), referring to the fibres from the leaves, which are used for weaving(Orwaetal;2009).

1.4 Biology

Male and female flowers are on different trees; hermaphrodite trees do occur rarely, but their fruits are smaller and sterile. In Sudan, flowering occurs from February to April and fruiting from November onwards. First fruiting is after 6-8 years age. Fruit ripens after 6-8 months, and fruiting takes place at the end of the dry season. In Nigeria, fruit appears in March and persists until the following season's flowers appear(Orwaetal;2009).

1.5 Ecology

Hyphaenethebaica is one of the eleven species of the genus found in Africa. Widespread in the Sahel, it grows from Mauritania to Egypt, from Senegal to Central Africa and east to Tanzania. The tree tends to grow close to groundwater but can also grow farther away. It is also found in parts of the Sahara where water occurs, in oases and wadis, and is widely distributed near rivers and streams, sometimes on rocky slopes. It does not do well in waterlogged areas; it is very resistant to bush fires. Trees occur on silty soils on river and stream banks and on rocky hilly slopes throughout the Sudan(Orwaetal;2009).

Hyphaenethebaica occurs in arid and semi-arid areas with an average annual rainfall of (200–600)mm, from100m below sea level up to 1000 m altitude. It is found in river valleys, around oasis, in moist places in grassland and woodland and on floodplains, but also in drier locations. It, usually, grows on light soils and prefers a pH of 6.5–7.5. *Hyphaenethebaica* withstands fire and tolerates mild frost and moderate salinity, but does not tolerate stagnant water. The presence of the tree is considered an indicator of good soil with high groundwater table. Although more than 40 *Hyphaene* species have been recorded, the genus, probably, comprises only about 10 species, occurring in the drier parts of tropical and subtropical Africa, with a few species extending into the Middle East and the western coast of India. Its taxonomy is much confused and often misunderstood. It has been revised for East Africa, but a further revision for the other parts of Africa is badly needed. Records of *Hyphaenethebaica* in East Africa usually refer to*HyphaeneCompressa* H.Wendl(Edwards;1997).

1.6 Origin and geographic distribution

Hyphaenethebaica is native to Benin, Burkina Faso, Cameroon, Central African Republic, Chad, Congo, Cote d'Ivoire, Democratic Republic of Congo, Egypt, Eritrea, Ethiopia, Gabon, Gambia, Ghana, Guinea, Kenya, Liberia, Mali, Mauritania, Niger, Nigeria, Senegal, Sierra Leone, Sudan, Tanzania and Togo(Shambel;2015).

Hyphaenethebaica is also distributed from Senegal and Gambia eastwards to Somalia, and is especially common between latitudes 8°N and 12°N. It also occurs in Libya, Egypt, Israel, the Arabian Peninsula and westernIndia.

Hyphaenethebaica is often planted. It was already cultivated in ancient Egypt, where it was considered sacred(Edwards;1997).

1.7 Properties and Composition

Fresh leaves contain about 20% fibre, but the yield after decortication is only 12–13%. The fibre is about 40 cm long, and weaker and coarser than jute fibre. The ultimate fibres are (0.5–1.5) – (2.1–3.6) mm long and (10–13) – (15–25) μm wide. Leaf pulp can be made into good-quality paper, but available quantities are too low for commercial production. The ultimate fibres of the rachis are on average 0.8 mm long and 13.7 μm wide, with a cell wall thickness of 3.5 μm and a lumen width of 6.8 μm. Pulps obtained from the rachis by the soda-anthraquinone (soda-AQ), alkaline sulphite-anthraquinone (AS-AQ) and alkaline sulphite-anthraquinone-methanol (ASAM) processes had low strength properties and seemed unsuitable for competitive papermaking. However, blending with kenaf bark pulp gave paper suitable for writing and printing.

The wood of male trees is said to be hard, tough, durable and resistant to termites, but the wood of female trees is recorded to be more fibrous, less durable and more susceptible to attacks by termites and borers. The wood fibre cells are 0.8–1.5 mm long and 25–45 μm wide. Paper made from pulp obtained from the wood is of inferior quality(Beinetal;1996).

Flour made from the pericarp contains per 100 g edible portion; moisture 10.7g, energy 1239 kJ, protein 2.6 g, fat 0.4 g, carbohydrates (including fibre) 79.0g, fibre 14.0g, ash 7.3g, Ca 68mg, thiamine 0.05 mg, riboflavin 0.10 mg and niacin 3.4mg. The kernel contains per 100g edible portion: moisture 5.7–6.2 g, energy1654 kJ, protein 2.4–5.0 g, fat 4.9–8.0 g, carbohydrates (including fibre)80.6g, fibre 6.5–11.0 g, ash 1.9–5.4 g,Ca 121–168 mg and P 170–281 mg.Per 100 g dry matter the seeds contain 1.11 g tannic acid and 330 mg phytic acid.

The dried and ground fruit has shown hypotensive and hypolipidaemic effects. An aqueous extract of the fruit mesocarp stimulated contractions of frog's heart and rat intestine, but inhibited uterine contractions in rats. The extract was capable of lowering the blood pressure in normal and hypertensive dogs, but had no diuretic effects in rabbits, and no oestrogenic and androgenic effects in rats. The aqueous extract contained alkaloids, reducing sugars and glucosides. Ethyl acetate, methanolic and aqueous extracts of the fruit shave shown antibacterial and antifungal activity. Aqueous and methanolic extracts of the fruit have shown antioxidant activity, due to the presence of phenolic constituents. An aqueous extract of the leaf showed strong radical scavenging activity.

Aqueous root suspensions showed hypolipidaemic activity in rats, but were also found to be toxic to both the liver and the kidneys(Beinetal;1996).

1.8Chemical composition and nutritional value

Chemical composition of a Kernels, dry nuts and fresh leaves as currently derived from FAO's Animal Feed Resources Information System (1991-2002) and from Bo Göhl's Tropical Feeds (1976-1982) are shown in tables (1-1, 1-2, and 1-3)(Maymoneetal;1961).

Table (1-1)Doum palm kernelsconstituents

Main analysis	**Unit**	**Avg**	**SD**	**Min**	**Max**	**Nb**	
Dry matter	% as fed	90.4				1	
Crude protein	% DM	9.0				1	
Crude fibre	% DM	7.3				1	
Ether extract	% DM	7.0				1	
Ash	% DM	2.8				1	
Gross energy	MJ/kg DM	19.2					*
Ruminant nutritive values	Unit	Avg	SD	Min	Max	Nb	
OM digestibility, Ruminant	%	76.2				1	
ME ruminants (FAO, 1982)	MJ/kg DM	12.3				1	
Nitrogen digestibility, ruminants	%	36.4				1	
Pig nutritive values	Unit	Avg	SD	Min	Max	Nb	
Energy digestibility, growing pig	%	78.6					*
DE growing pig	MJ/kg DM	15.1					*

- Avg: average or predicted value; SD: standard deviation; Min: minimum value; Max: maximum value; Nb: number of values (samples) used
- The asterisk * indicates that the average value was obtained by an equation.

Table (1-2)Doum palmdry nutsconstituents

Main analysis	**Unit**	**Avg**	**SD**	**Min**	**Max**	**Nb**	
Dry matter	% as fed	93.2	1.5	91.4	95.0	4	
Crude protein	% DM	4.2	0.8	3.5	5.2	4	
Crude fibre	% DM	36.6	10.1	24.7	46.1	4	
Ether extract	% DM	4.1	2.1	2.6	7.2	4	
Ash	% DM	2.8	0.9	1.8	3.6	4	
Gross energy	MJ/kg DM	19.4					*
Minerals	Unit	Avg	SD	Min	Max	Nb	
Calcium	g/kg DM	0.7	0.2	0.5	0.9	3	
Phosphorus	g/kg DM	1.7	0.7	1.0	2.4	3	
Potassium	g/kg DM	6.5	4.6	3.4	11.8	3	
Sodium	g/kg DM	0.1		0.1	0.1	2	
Magnesium	g/kg DM	1.0				1	
Ruminant nutritive values	Unit	Avg	SD	Min	Max	Nb	
OM digestibility, Ruminant	%	42.5				1	
ME ruminants (FAO, 1982)	MJ/kg DM	7.3				1	
Nitrogen digestibility, ruminants	%	0.0				1	
Pig nutritive values	Unit	Avg	SD	Min	Max	Nb	
Energy digestibility, growing pig	%	32.6					*
DE growing pig	MJ/kg DM	6.3					*

- Avg: average or predicted value; SD: standard deviation; Min: minimum value; Max: maximum value; Nb: number of values (samples) used.
- The asterisk * indicates that the average value was obtained by an equation.

Table (1-3)Doum palmfresh leavesconstituents

Main analysis	Unit	Avg	SD	Min	Max	Nb	
Dry matter	% as fed	48.0				1	
Crude protein	% DM	6.7	1.0	4.9	8.9	19	
Crude fibre	% DM	38.8	3.8	34.4	46.8	18	
NDF	% DM	64.2	4.5	57.6	73.0	18	
ADF	% DM	44.5	3.0	40.1	51.9	18	
Lignin	% DM	10.7	1.3	8.9	14.2	18	
Ether extract	% DM	1.6	0.3	1.0	2.1	19	
Ash	% DM	10.0	2.1	6.5	14.5	19	
Gross energy	MJ/kg DM	17.7					*
Minerals	Unit	Avg	SD	Min	Max	Nb	
Calcium	g/kg DM	4.1	0.9	2.1	4.7	7	
Phosphorus	g/kg DM	1.1	0.3	0.8	1.8	7	
Potassium	g/kg DM	11.9	2.5	9.6	17.1	7	
Magnesium	g/kg DM	3.2	0.7	2.4	4.3	7	
Secondary metabolites	Unit	Avg	SD	Min	Max	Nb	
Tannins (eq. tannic acid)	g/kg DM	20.8	4.7	10.2	27.6	16	

- Avg: average or predicted value; SD: standard deviation; Min: minimum value; Max: maximum value; Nb: number of values (samples) used.
- The asterisk * indicates that the average value was obtained by an equation.

1.9 Productsand Uses

1.9.1Products

The covering of the fruit is edible and can either be pounded to form a powder or cut off in slices; the powder is often dried then added to food as a flavouring agent. Young shoots produce tasty palm cabbage; the hypocotyl is edible, and so are the immature seeds if well prepared.Trees are browsed to a limited extent by livestock, especially in dry periods.Palms are occasionally used for firewood and charcoal; leaves may also be used as fuel.Leaves are probably the most important part of the palm, providing the raw material used in basketry, making mats, brooms, coarse textiles, ropes, thatching and string. Root fibres obtained after 2-3 days of soaking and beating of the roots are used for making fishing nets.Wood can be cut using an axe, but is difficult to saw due to the many fibres that constitute the wood. Timber from the male palm is said to be better than that from the female, as it is borer and termite proof, decorative and durable. It is often used for construction, providing supports and rafters for houses, water ducts and wheels, railway sleepers, planks, fence posts and raft construction. Tannin or dyestuff; Dried bark is used to produce a black dye for leatherwear.In Turkana, Kenya, the powder made from the outer covering of the fruit is added to water and milk and left to stand to make a mild alcoholic drink in other countries, the terminal meristem is tapped for making palm wine.Roots are used in the treatment of bilharzia, while fruit pulp is chewed to control hypertension. Sore eyes in livestock are treated using charcoal from the seed kernel. Other productslike hard seed inside the fruit, known as ‘vegetable ivory’, is used to make buttons and small carvings, and as artificial pearls. Ashes from the stipes of trees can be used as a substitute for salt(Orwaetal;2009).

1.9.2 Uses

1.9.2.1General uses

Strips from the young, unexpanded leaves are widely used for weaving mats, bags, baskets, hats, fans, strainers, bowls, rope, string, nets and coarse textiles. In the Sahel ropes for wells are often made from *Hyphaenethebaica*. The midveins of the leaf segments are used as frames for woven objects and tied together to be used as brooms. Whole leaves are used for thatching. In Eritrea the petioles are woven into bed mats and used in the construction of houses, fences and bridges. Fibres from the leaf blade are used for making rough bags, but extraction is laborious and the quality not high. Waste material from fibre extraction is used in Eritrea for stuffing and reinforcing cement. Fibre from the petiole is used for making sponges and brushes. The roots yield a fibre used for making snares and fishing nets and traps.

The trunk is used for house construction, fences, railway sleepers and canoes. Cut into planks it is made into canoes and water wheels. Hollowed trunks are used as water troughs and irrigation pipes. In Mali the wood is used for poles, shafts and harpoons. It is also used as fuel and for charcoal making. The leaves, rachis and fruits are used as fuel as well. Ash from the stem is used as a vegetable salt.

The apical bud is eaten as a vegetable (‘palm cabbage’). The heart at the base of the trunk is sometimes eaten cooked, and the hypocotyl of seedlings is also cooked and eaten. Sap is extracted from the tree just before flowering for the production of palm wine.

Young leaves and stems are browsed by livestock. In Sudan the young leaves are cut and dried to be used as fodder during the dry season. Older leaves are bitter and unpalatable. The male inflorescence is a fodder in Sudan. The fruit mesocarp of some trees is inedible, but that of other trees is very palatable, with a sweet, gingerbread-like taste. It is, sometimes, made into syrup or ground into meal, which is made into cakes and sweetmeat. The fruits have a hard endocarp, and the 'nuts' are made into balls, toys and weapons. The endocarp is made into small containers. The hard kernel of mature seeds was formerly used as vegetable ivory for the production of buttons, beads and small carvings. The endosperm of unripe seeds is soft and has a cavity holding a liquid which is a much-savoured drink in northern Nigeria. The endosperm of unripe seeds is eaten raw or boiled. Dry fruits yield a black dye, used fur dyeing leather. Another interesting side use is for pleasure in Kenya where they use it to make palm wine by way of sapping or drawing the moisture inside the tree just before it flowers(Shambel;2015)

In Eritrea and Ethiopia, besides using them as edible fruit, children burn these hard-shelled nuts in a firewood inside a mogogo stove (a traditional firewood stove) where they crack them open and remove the hard outer shell-covering. They then take out the inner kernel, hammer in a nail for a tip and use their handiwork as a traditional spin top. Sometimes a hole is drilled in on the side to make it create the special humming sound effect. Just like all spin tops across the rest of the world, a string is coiled around the top and pulled hard while unwinding it and letting it go to make it spin through inertia. Children play this fun outdoorsy game seasonally.Other practical uses of doum dates include the production of molasses, cakes and sweetmeats (halewat) which are made from the rind of the seeds; for creating buttons, beads and small carvings which are made from the hard seed inside the fruit; for creating black leather dye (tinta) which is made from the dry fruits. And finally, one should not also forget its last occasional use which is that of firewood.(Shambel,2015).

1.9.2.2Medical uses

In traditional medicine in Mali a paste of the root is massaged on the chest to relieve chest-pain. In Benin a decoction of the leaves of *Hyphaenethebaica* and *Elaeisguineensis* Jacq., the aerial parts of *Indigoferasuffruticosa* Mill, and the fruit of *Xylopiaaethiopica* (Dunal) A.Rich. is drunk for the treatment of jaundice. A maceration of the root bark is taken for the treatment of intestinal colic and inguinal hernia. The mesocarp is credited with diuretic properties, and a root extract is drunk in case of blood in the urine(von Maydell;1986).

The fruit is sold in herbalist shops in Egypt for its therapeutic use. People use it as a herbal tea drink to treat hypertension. While the roots are used in the treatment of bilharzia, the ground nuts themselves have also been used for dressing wounds.A drink made from it is used for the treatment of jaundice (weyba). When softened and dissolved in liquid, the root bark of the tree is used for the treatment of intestinal colic (qirxet) and inguinal hernia. A solution from the leaf fibre (after being boiled and soaked in water) is used as an eyewash to treat conjunctivitis (himamayni) in Sudan. The fruit itself when eaten raw is also good for the treatment of stomach pain and bladder infection. The fleshy middle part of the fruit is believed to increase the flow of urine (diuretic) and thereby treat ailments that include blood pressure, heart failure, kidneys, liver disease and diabetes(Shambel;2015).

1.10 Tree Management

Hyphaenethebaica is propagated by seeds and suckers. The 1000-seed weight is 20–50 kg. Direct sowing instead of sowing in nurseries is recommended, as the radicle and plumule are buried deep before germination. Germination may start 1 month after sowing, but can also take up to a year.After sowing, the soil must be kept moist for 2–3 months, but after that seedlings are able to withstand as much as 10 months drought. In germination trials in Niger freshly collected seeds germinated much better than 13-month-old seeds. Furthermore, untreated seeds had much lower germination than seeds with the pericarp removed and bare nuts (pericarp and endocarp removed). Fresh or 13-months-old seeds that had been soaked in water before sowing had higher germination than unsoaked seeds. The highest germination for freshly-collected seeds was obtained with mechanically-scarified seeds soaked in water for 3 days Elephants and baboons eat the fruits and disperse the seeds. *Hyphaene* spp.Are pleonanthic, i.e. the stems do not die after flowering. Stems cut for tapping palm wine die, but the tree coppices from the root. The tree may produce suckers.The harvesting by Cutting palm heart or tapping palm wine kills the branch(Bekele-Tesemmaetal;1993).

Growth is known to be relatively slow, and after germination a single strip-shaped leaf is produced, with fan-shaped leaves being produced at ground level 2-3 years after germination. At this stage, a new leaf is produced every 7 days and the stem is produced after 18-20 years. This fire-resistant species is managed by coppicing and lopping. Mature trees of 6-8 years produce 50 kg of fruit/year. Palm trees that are tapped for production of wine need to be protected from browsing camels. Wood from female palms is susceptible to attack by termites and borers(Orwaetal;2009)

1.11 Handling after harvest

To obtain weaving material, the segments are separated and are made supple by wetting them, the midvein is removed, and the blade is divided into strips, with the width depending on the article to be made. In Eritrea fibre has been extracted from the leaves with machines. Before being decorticated, the leaves were soaked in water for 24 hours. Fibre is obtained from the roots by soaking them for 2–3 days and beating them with pieces of wood(Bekele-Tesemmaetal;1993).

1.12 Production and international trade

The ‘nuts’ were formerly exported, e.g. from Sudan and Eritrea, for the production of buttons and beads from the kernel and small boxes from the endocarp, but nowadays buttons are mainly made from plastic. The fruits are collected to be sold in towns. Bundles of leaf strips to be used for weaving, as well as woven products are traded on local markets(Kirby;1963).

1.13Genetic resources and breeding

Hyphaenethebaica has a wide distribution area and is often common, but it is locally overexploited and endangered. In Niger, for instance, entire juvenile leaves are cut from young palms too regularly and intensively, resulting in a change of the normal arborescent habit into a subterranean-creeping habit. In Eritrea the felling of living trees is illegal(Edwards;1997).

1.14Prospects

Hyphaenethebaica is an extremely useful multipurpose tree for arid and semi-arid regions, yielding fibre, construction material, food, fodder and a range of other products. In Eritrea it plays an important role in food security: it provides food in times of food shortage and the leaves are a source of income. To assure its availability in the future, care should be taken that the species is exploited in a sustainable way. The variability in the fruit quality may offer scope for selection and breeding(Edwards;1997).

1.15Techniques Used in DoumFruit Analysis

1.15.1Gas Chromatography-Mass Spectrometry (GC-MS)

Gas Chromatography (GC), is a type of chromatography in which the mobile phase is a carrier gas, usually an inert gas such as helium or an un-reactive gas such as nitrogen, and the stationary phase is a microscopic layer of liquid or polymer on an inert solid support, inside glass or metal tubing, called a column. The capillary column contains a stationary phase; a fine solid support coated with a nonvolatile liquid. The solid can itself be the stationary phase. The sample is swept through the column by a stream of helium gas. Components in a sample are separated from each other because some take longer to pass through the column than others. Mass Spectrometry (MS), the detector for the GC is the Mass Spectrometer (MS). As the sample exits the end of the GC column it is fragmented by ionization and the fragments are sorted by mass to form a fragmentation pattern. Like the retention time (RT), the fragmentation pattern for a given component of sample is unique and therefore is an identifying characteristic of that component. It is so specific that it is often referred to as the molecular fingerprint. Gas chromatography-mass spectrometry (GC-MS) is an analytical method that combines the features of gas-liquid chromatography and mass spectrometry to identify different substances within a test sample. GC can separate volatile and semi-volatile compounds with great resolution, but it cannot identify them. MS can provide detailed structural information on most compounds such that they can be exactly identified, but it cannot readily separate them.

Principle of GC-MS: GC/MS-a combination of two different analytical techniques, (GC) and Mass Spectrometry (MS), is used to analyze complex organic and biochemical mixtures (Skoog et al., 2007). The GC-MS instrument consists of two main components. The gas chromatography portion separates different compounds in the sample into pulses of pure chemicals based on their volatility Oregon State University, 2012) by flowing an inert gas (mobile phase), which carries the sample, through a stationary phase fixed in the column (Skoog et al., 2007). Spectra of compounds are collected as they exit a chromatographic column by the mass spectrometer, which identifies and quantifies the chemicals according their mass-to-charge ratio (m/z). These spectra can then be stored on the computer and analyzed(Syed ZameerHussainetal;2014)

1.15.2 X-ray Fluorescence (XRF)

X-ray fluorescence is the emission of characteristic "secondary" (or fluorescent) X-rays from a material that has been excited by bombarment with high-energy X-rays or gamma rays.

The phenomenon is widely used for elemental analysis and chemical analysis, particularly in the investigation of metals, glass, ceramics and building materials, and for research in geochemistry, forensic science, archaeology and art objects such as paintings and murals(De Viguerie etal;2009).

1.15.2.1 Underlying Physics

When materials are exposed to short-wavelength X-rays or to gamma rays, ionization of their component atoms may take place. Ionization consists of the ejection of one or more electrons from the atom, and may occur if the atom is exposed to radiation with energy greater than its ionization energy. X-rays and gamma rays can be energetic enough to expel tightly held electrons from the innerorbitals of the atom. The removal of an electron in this way makes the electronic structure of the atom unstable, and electrons in higher orbitals "fall" into the lower orbital to fill the hole left behind. In falling, energy is released in the form of a photon, the energy of which is equal to the energy difference of the two orbitals involved. Thus, the material emits radiation, which has energy characteristic of the atoms present. The term *fluorescence* is applied to phenomena in which the absorption of radiation of a specific energy results in the re-emission of radiation of a different energy (generally lower)(Penner-Hahn etal;2013).

1.15.2.2 Chemical Analysis

The use of a primary X-ray beam to excite fluorescent radiation from the sample was first proposed by Glocker and Schreiber in 1928. Today, the method is used as a non-destructive analytical technique, and as a process control tool in many extractive and processing industries. In principle, the lightest element that can be analysed is beryllium (Z = 4), but due to instrumental limitations and low X-ray yields for the light elements, it is often difficult to quantify elements lighter than sodium (Z = 11), unless background corrections and very comprehensive inter-element corrections are made(David Bernard Williams,etal;1928).

1.16Previous Studies

A. K., Aremuand O.K., Fadele;(2011) have determinedthe physical properties of doum palm fruit as a function of moisture content. The average dimension of doum palm fruit in the three principal axes (namely, length, width, and thickness) and its equivalent diameter, projected area, sphericity, porosity, bulk and true densities were determined for moisture contents ranging from 24.05 to 67.59% d.b and were found to be 60.65, 48.78, 47.09 and 51.61 mm, 19.94 cm^2 and 0.85 respectively, while the true and bulk densities increased from 711.05 to 958.53 kg/m^3 and 370.51 to 483.77 kg/m^3 respectively with moisture content but the porosity increased from 46.45 to 51.66% and later decreased to 49.53%. The dimension of doum palm fruit with its equivalent diameter, projected area and sphericity were found to be constant with moisture content variation. It was found that the relationships between true and bulk densities and moisture content of doum palm fruit followed a linear pattern while that of porosity exhibited a non-linear relationship with the moisture content at 0.05 significance level.

Nahla A. Ayoub etal;(2013), used GC and GC-MS analysis of essential oil isolated from doum fruits (*Hyphaenethebaica*) revealed the presence of fifty-seven compounds. Fifty of them could be identified with monoterpenes represent 15.97 % including compounds such as sabinene (0.82 %), β-pinene (1.98 %), limonene (2.42 %), terpinen 4-ol (1.77 %) %), α-terpineol (0.95 %). While diterpenes represent 40.49 %, of which incensole (17.52 %) and incensole acetate (19.81 %) were found to be the main components. Oxygenated compounds constituted 66.78 % of the total compounds identified which indicated the economical value of this oil. They concluded that, the scent of doum oil fruits may be attributed to the presence of volatile diterpenes as cembreneA, cembrene C, incensole and incensole acetate; they discraib compounds to be reported for the first time in family Palmae.

Phytochemical Analysis and Anti-inflammatory Potential of Hyphaenethebaica L. Fruit was carried by Mohamed A. Farag and Paul W. Par´e,;(2013) in their study, Metabolite profiling and biological activity are reported from organic and aqueous extracts of the fruit from the desert palm *Hyphaenethebaica*. In their analysis Phenolics and oxylipids profiles were determined using UPLC-PDA-TOF (ultra performance-photodiode array-time of flight) high-resolution mass spectrometry in order to obtain the molecular formula and exact mass Under optimized conditions, 17 compounds were simultaneously identified and quantified including 2 cinnamic acid derivatives, 5 flavonoids, 6 fatty acids, 2 sphingolipids, a lignan, and a stilbene. Sugars composition in the fruit was characterized and quantified by 1H-NMR (nuclear magnetic resonance) with sucrose detected as the major component in fruit at a level of 219 mg/g. Fruit organic extracts anti-inflammatory potential was assessed in vitro by cyclooxygenase-1 enzyme inhibition.

El-Gendy, Ahmed etal;(2009) prepared study to clarify the role of *Hyphaenethebaica*(Doum), as a medicinal plant containing flavonoids, saponins and tannins, on blood pressure, blood lipids and lipoproteins in hypertensive patients.Thirty female patients who were hypertensive, obese and hyperlipidemic were used in the study that extends to 3 months. They were divided into 2 groups each group consisted of 15 patients (control group received antihypertensive drug with similar dose, and Doum group received the same antihypertensive drug with the same dose, and agreed to consume an oral supplement of Doum in a daily dose of 25 mg/kg body weight). Blood pressure, plasma lipids and lipoproteins were followed up in all patients for 3 months. Blood samples were obtained after a 12 to 14 h fast. Concentrations of total cholesterol, triglycerides, HDL, apoA-I and apoB were measured. LDL was calculated according to the Friedewald equation. The recorded values were expressed as means and standard deviations (Mean Â±SD). The statistical analyses were performed using the SPSS statistical Package version 12. The obtainedresults compared with the control patients, supplementation with Doum caused a significant decrease in systolic blood pressure, diastolic B.P., total cholesterol, triglycerides, LDL, apoB-concentrations and LDL/HDL ratio, while significantly increasing the concentrations of HDL and apoA-I. the study concluded that, the results confirm the benefits of Doum including lowering blood pressure in hypertensive patients and changing blood lipids and lipoproteins in a manner that decreases the risk on the cardiovascular system. More studies are needed support these benefits of Doum.

Joslineetal;(2013), evaluatedthe anti-diabetic activity together with the accompanying biological effects of the fractions and the new natural compounds of *Hyphaenethebaica*epicarp. 500 g of coarsely powdered of *Hyphaenethebaica* fruitsepicarp were extracted using acetone. The acetone crude extract was fractionated with methanol and ethyl acetate leaving a residual water-soluble fraction **WF**. The anti-diabetic effects of the **WF** and one of its compounds of the acetone extract of the *Hyphaenethebaica*epicarp were investigated in this study using 40 adult male rats.The Phytochemical investigation of active WF revealed the presence of ten different flavonoids, among which two new natural compounds luteolin 7-O-[6”-O-α-Lrhamnopyranosyl]-β-D-galactopyranoside 3 and chrysoeriol7-*O*-β-D-galactopyranosyl(1→2)-α-L-arabinofuranoside 5 were isolated. Supplementation of the**WF** improved glucose and insulin tolerance and significantly lowered blood glycosylated hemoglobin levels. On the other hand, compound 5 significantly reducedaspartate amino transferase(AST) andalanine amino transferase(ALT) levels of liver, respectively. Likewise, the kidney functions were improved for both **WF** and compound 5, whereby both urea and creatinine levels in serum were highly significant. the concluded that, the results opteainded justify the use of water-soluble fraction (WF) and compound 5 of the (HT) epicarp as anti-diabetic agent, taking into consideration that the contents of **WF** were mainly flavonoids.

Betty Hsu etal;(2005) prepared fruit hot water extract*Hyphaenethebaica* and examined for its (i) hydrogen donating activity, (ii) Fe2+-chelating activity, (iii) hydroxyl radical-scavenging activity, (iv) inhibition of substrate site-specific hydroxyl radical formation, (v) superoxide radical-scavenging activity, and (vi) reducing power. The total phenolic content of the fruit extract was also determined in order to quantify antioxidant activity as gallic acid equivalent (GAE) per ml reaction. The total phenolic content of the Doum fruit is low, but the extract exhibited potent antioxidant activity in terms of GAE. The activities expressed as mmol pure compound equivalent per g GAE content of extract are: (i) 2.85 mmol ascorbic acid equivalent, (ii) 1.78 mmolethylenediaminetetraacetic acid equivalent, (iii) 192 mmolgallic acid equivalent, (iv) 3.36 mmolgallic acid equivalent, (v) 1.78 mmolgallic acid equivalent and (vi) 3.93 mmol ascorbic acid equivalent. These values were of the same magnitude as antioxidant activity in black tea except for Fe2+-chelating activity which was about 14 times more potent. The results show that the fruit of *Hyphaenethebaica* fruit is a source of potent antioxidants.

The specific heat, thermal conductivity and thermal diffusivity of doum palm fruit were determined by Aremuetal;(2010) as a function of moisture content which varies from 24.05 to 67.59%. The specific heat and thermal conductivity were found to have a range of 1496.46 – 2966.67 J/kg K and 0.1671 - 0.3338 W/mK respectively. Their values increased linearly with increasing moisture content values at 0.05 level of significance. Specific heat and thermal conductivity were found to be moisture dependent. A non-linear relationship was established between thermal diffusivity and moisture content in the above moisture range, within the temperature range of 334 - 337K. The values obtained for these thermal properties could be useful in heat treatment of Doum palm fruit**.**

The widespread use of medicinal plants for health purposes has increased dramatically due to their great importance to public health. The levels of phenolic, flavonoid, β-carotene and lycopene compounds of Anastaticahierochuntica and Hyphaenethebaica were determined by Amaletal; (2010).The plant extracts were evaluated for their antioxidant activities using various antioxidant methodologies: (i) scavenging of free radicals using 2, 2-diphenyl-1-picrylhydrazyl, (ii) metal ion chelating capacity, and (iii) scavenging of superoxide anion radical. The antimicrobial activity of both plant extracts was evaluated against a panel of microorganisms using the agar disc diffusion method. The total phenolic content (51.97 and 64.9 mg/g dry weight in *A. hierochuntica* and *H. thebaica*, respectively) was significantly (p0.05) different. The antioxidant activity increased with an increase in concentration. The plant extracts were more active against Gram-positive bacteria than Gram-negative bacteria. Also, the antimicrobial activity of *H. thebaica* was higher than that of *A. hierochuntica*methanolic extracts. This study reveals that the consumption of these plants would exert several beneficial effects by virtue of their antioxidant and antimicrobial activities.

The antioxidant capacity of doum fruit extract and the total phenolic content were analyzed byFaten(2009). The antioxidant capacity was estimated by DPPH and iron chelating assays. Quercetin, ascorbic, BHT and tannic were used as positive controls. Also the effect of doum extract on viability of acute myeloid leukemia was studied. The results showed that the total phenolic content was 0.5 µg/ 3 µg dried extract sample as quercetin. In iron chelating assay the result showed that

800 µg/ml doum extract gave the best antioxidant activity (21% inhibition). In DPPH assay the 1000 µg/ml extract exhibited 50% antioxidant activity (IC50) but 1500 µg/ml extract exhibited 80% antioxidant activity. In the viability test, the results showed that half maximal inhibitory concentration (IC50) of doum extract was found to be 3 µg/ml. The result indicated that the doum extract could be an important dietary source of phenolic compounds with high antioxidant and anticancer activities.

According to Amwatta;(2004), the *Hyphaenecompressa* H. Wendl. (doum palm) is a widespread palm in eastern Africa, being particularly abundant along the coast of Kenya and Tanzania. In the dry lands of Kenya it occurs in isolated populations and is mainly confined to riverine forests (Fig. 1). The palm is common along the coastal strip, riverine ecosystems of the drylands and scattered within open savannah grasslands (Dale and Greenway 1961, Beentje 1994). It is most abundant in low sandy places and secondary forests. In the northern and eastern regions of the country, the species contributes significantly to the livelihood and welfare of the local communities, who are mainly nomadic pastoralists and agro-pastoralists. The species is used in several ways. Products ranging from thatch, ropes, baskets, nuts, dye and medicine are derived from the species. The doum palm dominates forests and woodlands, serves as sources of dry season grazing during drought and also protects the riverbanks. Along the shores of Lake Turkana fourteen different economic uses of the species have been recorded (Awuondo 1990). Among them leaf was the most used part of the palm. Leaves and petioles are the major building materials for the manyattas (traditional domeshaped houses) and makuti (strips of mature green palm leaflets tied in a dense row along a leaf stalk) for the modern houses (see below). Leaves are used to make mats, carpets and baskets for sale. Ropes, webbed stick wheels (used for bundling fish) and wicker baskets (for catching fish) are made and sold to fishermen. However, information about the full array and magnitude of products derived from the leaves among communities living in the dry lands of Kenya is incomplete. Although palm leaf use by Turkana has been reported (Hoebeke 1989, Awuondo 1990), this is the first time the different uses by Tharaka, Kamba and Borana communities have been recorded. The climate of the study area is characterized by erratic annual rainfall. Apart from famine relief, the availability of resources that support life, such as water and forage for livestock, is highly variable in time and space. When livestock productivity and herd numbers are low, the pastoralists will seek out alternative livelihoods. In most cases these will involve activities such as rain-fed agriculture, the making of makuti and the weaving of baskets. For instance, the drought of 1992 pushed most Turkana pastoralists to resort to craft activity. The creation of a United Nations High Commissioner for Refugees (UNHCR) refugee camp in the district fuelled the demand for palmbased crafts. With ever-increasing droughts, more pastoralists resorted to weaving, leading to overharvesting of woodlands near urban centers (Amwatta, 1993). Consequently this has led to reduction in quality of basketry. In this study, I wasexamined the diversity of use of the palm leaves among the four ethnic communities. The first objective was to document the uses of leaves for both domestic and commercial needs. The second objective was to assess the availability of the leaves for the various needs.

According to Nawaletal;(2014),th**e** Non-wood forest products (NWFPs) have received great attention during the last years since they have important usages throughout the world. Forest fruits are one of these NWFPs. Sudanese forest fruits are used traditionally as foods as well as medicines. Doum (*Hyphaenethebaica*L), kirkir (*Randiageipaeflora*), karmadoda (*Naucleaelatifolia*) and godeim (*Grewiatenax*) are some of the indigenous fruits of the Sudan. The minerals profile, essential and non-essential amino acids values of these fruits were studied. These forest fruits were found containing adequate amount of minerals. Doum and kirkir are rich in P and K, karmadoda is rich in P, K, Mg and Ca; while godeim is rich in Mg, K, Ca and Fe. Karmadoda was found rich in the essential amino acids, leucine (318.59 mg/100g), isoleucine (167.28 mg/100g) and valine (214.93 mg/100g), however, kirkir was found rich in arginine (543.71 mg/100g). karmadoda was also rich in the non-essential amino acids alanine, aspartic acid, glutamic acid, glycine, serine and proline of 237.46, 421.43, 782.76, 183.70, 156.23 and 165.98 mg/100g, respectively. Therefore, these fruits can be used in several foods as ingredients. Karmadoda can be used as a supplement for minerals and essential and non-essential amino acids.

The Objective of the Study

The objective of this study is:

- To extract the methanol soluble ingredients from*Hyphaenethebaica*fruit.

-To determine the main chemical constituents of doum fruit extract using GC-MS analysis.

-Toidentify the main elementsavailable in doum fruit powder using XRF technique.

Chapter Two

Materials and Methods

2- Materials and Methods

2.1Collection of Samples

The fruits of *Hyphaenethebaica* were collected from doum palm trees and local markets atKhartoum state.The outer part of the fruits samples wereseparated, dried and grind into fine powder.

2.2 chemicals

-95%Methanol

2.3 Instruments

23.1Gas Chromatography–Mass Spectrometer

Gas chromatography–mass spectrometer model: GC.MS-QP2010 Ultra, Detector: Mass spectrometer, Company: Shimadzu, Country: Japan, Carrier gas: Helium, Column:Rtx-5MS…Length(30m)…Diameter(0.25mm)…Thickness(0.25ul).

2.3.2X-Ray Fluorescence Spectrometer

X-ray fluorescence spectrometermodel: CANBERRA series 35plus, with Cd109 source and pure silicon detector, designed and manufactured in USA.

2.4 Methods of Analysis

2.4.1 Methanol Extract of*Hyphaenethebaica*Fruit

1kg of air-dried fruits Powder of*Hyphaenethebaica*was extracted using5Lof 95% methanol at room temperature for 72 hours. The insoluble portion of the fruits was separated by filtration, and the filtrate asmethanol extract was evaporated under reduced pressure up to dryness(paste).GC-MS technique was then employed for fractionation and analysis.

2.4.2GC-MSAnalysis ofDoumFruitExtract

0.5gof the dried Extract from the fruit was dissolvedin20ml of 95% methanol and sodium sulfatepowderwas added to the solutionwith stirring. The solution was then filtered, and 1µm from the filtrate was injected into the GC-MSInstrument. The spectra were then analyzed and components of the sample wereidentified.

2.4.3 XRF Determination of Metals in DoumFruit

The sample were first crushed into fine powder and then pressed into pellets using a 10 tonpressing machine. The diameter of each pellet was about 4.9 cm and the mass about 1g. The pellets were introduced to the XRF spectrometer. The spectra were then analyzed and concentration of the sample was calculated.

Chapter Three

Results and Discussion

3-Results and Discussion

3.1GC-MS Results

Figure(3-1)showsGC-MS chromatogram of *Hyphaenethebaica*fruit extract. The corresponding compound names for peaks are shown in Table(3-1).Compounds included phenolic cmpounds as well as saccharidesand organic-acids.GC-MS was adopted for methanolic extract from doum fruit profiling resulting in the detection of 34 compounds.

Tris(hydroxymethyl)nitromethane represented the higherpercentage in the sample (27.67%) and N-Acetyl-dl-penicillamine and 2,6-dimethoxy-phenol both of them represented the lowest percentage(0.24%).

The other more available compounds were found to be 3-O-methyl-D-glucose (15.33%),5-Hydroxymethylfurfural (7.49%), Maltol (3.24%), Pentadecanoic acid (2.28%),2-Hydroxy-gamma-butyrolactone (2.24%).

Tris(hydroxymethyl)nitromethane (figure 3.2 and3.3) was found to be a major component (27.67%),Tris(hydroxymethyl)nitromethane Has a formula ($C_4H_9NO_5$) and Molecular Weight (151).

Tris(hydroxymethyl)nitromethane products are used as antimicrobial agents to control bacteria and slimes in industrial water treatment and oil and gas treatment applications and to formulate other biocide or preservative products.At high concentrations in doum fruit(Huberetal;2006).

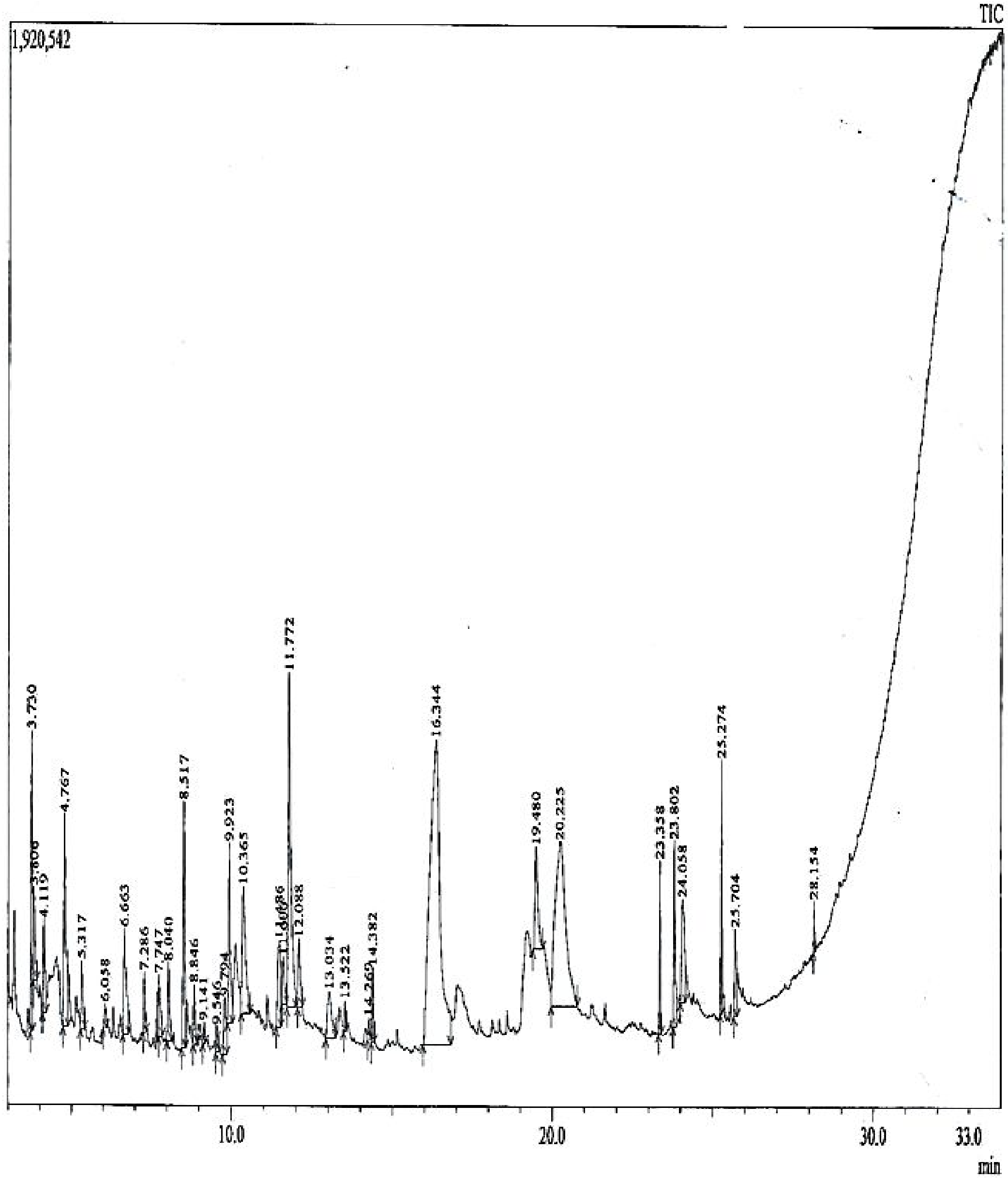


Figure (3.1) GC-MS chromatogram of *Hyphaenethebaica*fruit extract

Table(3-1)The corresponding compound names for peaks of *Hyphaenethebaica* fruit extract in GC-MS chromatogram

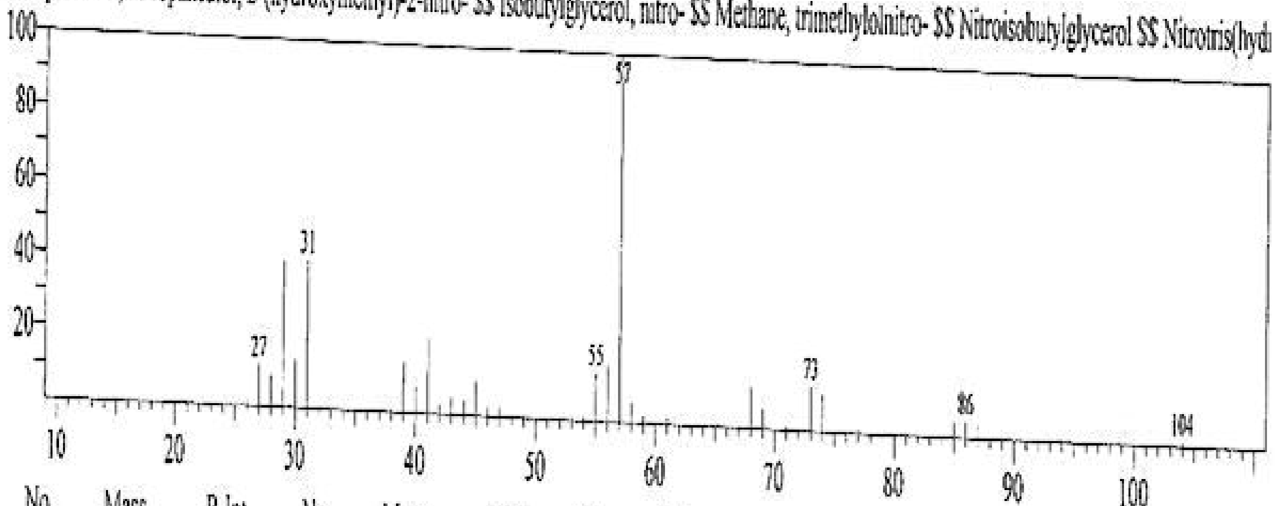
Peak#	R.Time	Area	Area%	Name
1	3.730	1551872	3.57	Ethane, 1,2-bis[(4-amino-3-furazanyl)oxy]-
2	3.806	594709	1.37	Furan, 2,5-dimethyl-
3	4.119	606355	1.39	2-Furanmethanol
4	4.767	1823388	4.19	dl-Glyceraldehyde dimer
5	5.317	459458	1.06	2-Cyclopenten-1-one, 2-hydroxy-
6	6.058	151001	0.35	2-Furancarboxaldehyde, 5-methyl-
7	6.663	974225	2.24	2-Hydroxy-gamma-butyrolactone
8	7.286	303358	0.70	3-Methyl-3-oxetanemethanol
9	7.747	423287	0.97	6-Oxa-bicyclo[3.1.0]hexan-3-ol
10	8.040	464685	1.07	2,5-Dimethyl-4-hydroxy-3(2H)-furanone
11	8.517	1408271	3.24	Maltol
12	8.846	356348	0.82	Butanal, 3-methyl-
13	9.141	104347	0.24	N-Acetyl-dl-penicillamine
14	9.546	150648	0.35	2(3H)-Furanone, 5-acetyldihydro-
15	9.794	482907	1.11	.alpha.-Amino-.gamma.-butyrolactone
16	9.923	1217072	2.80	4H-Pyran-4-one, 2,3-dihydro-3,5-dihydrox
17	10.365	1643077	3.78	d-Gluconic acid dimethylamide
18	11.486	1064455	2.45	1-Deoxy-d-arabitol
19	11.606	351932	0.81	Benzofuran, 2,3-dihydro-
20	11.772	3261234	7.49	5-Hydroxymethylfurfural
21	12.088	507005	1.17	1,2,3-Propanetriol, 1-acetate
22	13.034	801722	1.84	Heptanoic acid, 6-oxo-
23	13.522	126397	0.29	2-Methoxy-4-vinylphenol
24	14.269	103603	0.24	Phenol, 2,6-dimethoxy-
25	14.382	321232	0.74	Eugenol
26	16.344	12044663	27.68	1,3-Propanediol, 2-(hydroxymethyl)-2-nitr
27	19.480	1220410	2.80	Methyl .beta.-d-galactopyranoside
28	20.225	6672098	15.33	3-O-Methyl-d-glucose
29	23.358	589084	1.35	Hexadecanoic acid, methyl ester
30	23.802	993617	2.28	Pentadecanoic acid
31	24.058	1190947	2.74	.alpha.-D-Mannofuranoside, 1-O-decyl-
32	25.274	830395	1.91	9,12-Octadecadienoic acid (Z,Z)-, methyl e
33	25.704	548943	1.26	9,12-Octadecadien-1-ol, (Z,Z)-
34	28.154	172442	0.40	Hexanedioic acid, bis(2-ethylhexyl) ester
		43515187	100.00	

Compound Information

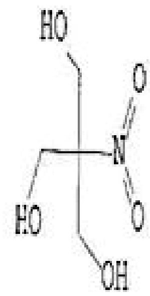
Entry:9072 Library:NIST11S.LIB

Formula:C4H9NO5 CAS:126-11-4 MolWeight:151 RetIndex:1444

CompName:1,3-Propanediol, 2-(hydroxymethyl)-2-nitro- $$ Isobutylglycerol, nitro- $$ Methane, trimethylolnitro- $$ Nitroisobutylglycerol $$ Nitrotris(hyd



No.	Mass	R.Int	No.	Mass	R.Int	No.	Mass	R.Int	No.	Mass	R.Int
1	15.00	0.60	12	41.00	21.10	23	56.00	15.30	34	74.00	10.40
2	26.00	2.10	13	42.00	2.80	24	57.00	100.00	35	77.00	1.80
3	27.00	11.50	14	43.00	5.70	25	58.00	5.70	36	85.00	4.30
4	28.00	8.80	15	44.00	4.20	26	59.00	1.80	37	86.00	4.40
5	29.00	39.70	16	45.00	9.60	27	60.00	1.00	38	87.00	4.20
6	30.00	13.20	17	46.00	2.40	28	61.00	2.10	39	88.00	0.30
7	31.00	40.30	18	47.00	2.50	29	68.00	11.30	40	102.00	0.30
8	37.00	0.60	19	49.00	1.10	30	69.00	5.40	41	104.00	1.50
9	38.00	0.90	20	53.00	1.40	31	71.00	1.00			
10	39.00	13.40	21	54.00	1.30	32	72.00	0.20			
11	40.00	7.70	22	55.00	13.30	33	73.00	12.20			



Figure(3.2) CompoundInformation ofTris(hydroxymethyl)nitromethane

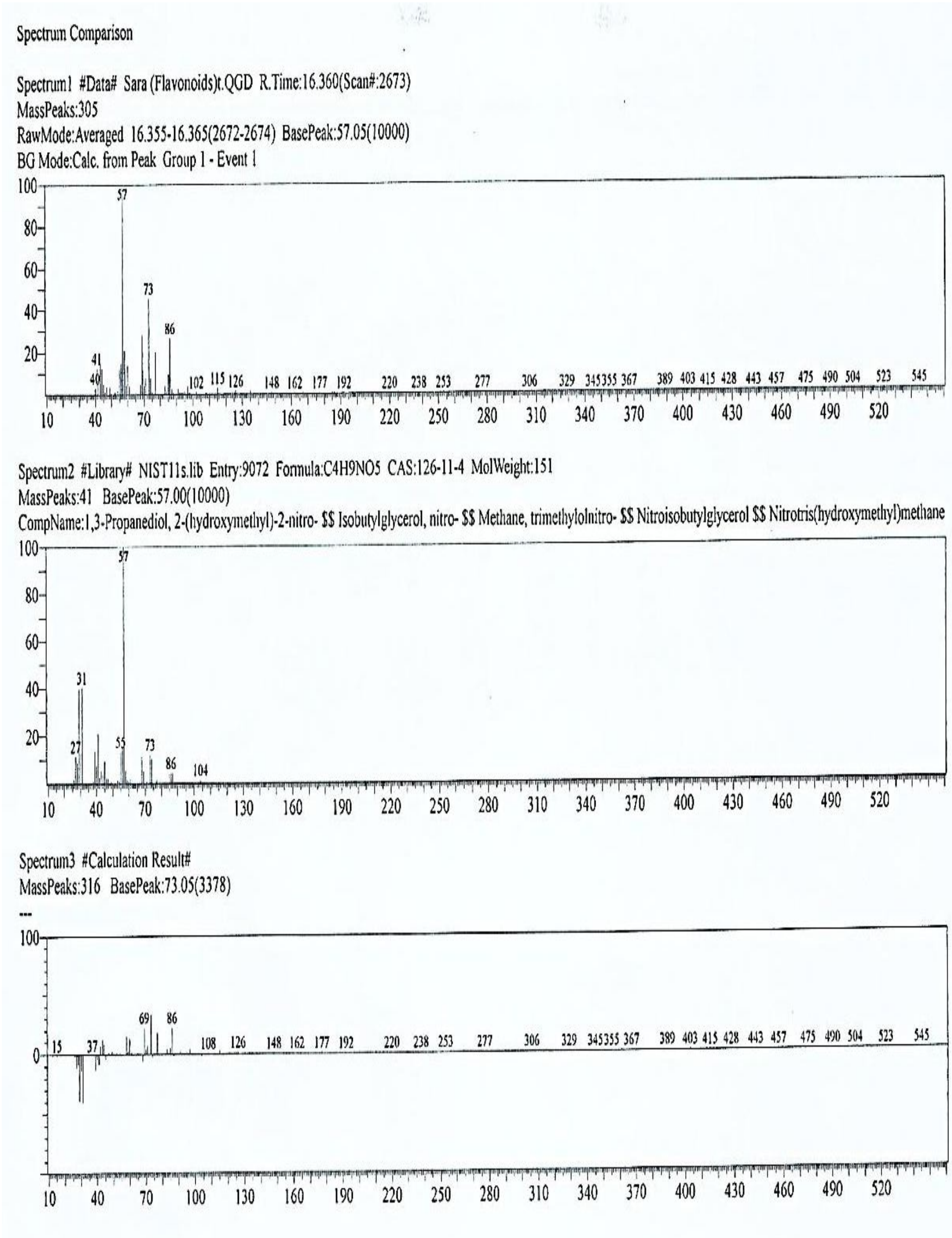


Figure (3.3) Spectrum comparison of Tris(hydroxymethyl)nitromethane

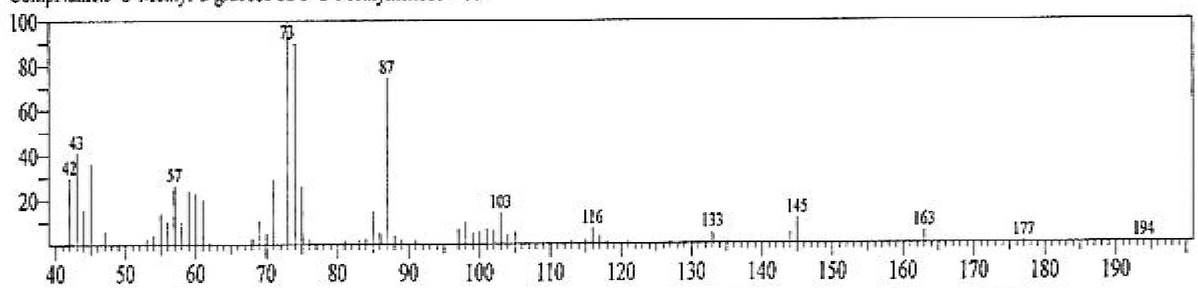
3-O-methyl-D-glucose (Fig 3.4and3.5) has relatively high percentage (15.33%). 3-O-methyl-D-glucose has a formula (C7H14O6) and Molecular Weight (194).Uptake of 3-O-methylglucose by islet cells was less than 19%of controls. Accordingly, caffeine inhibited 3-O-methylglucose uptake during contractions only in oxidative muscle fibers that are characterized by a high sensitivity to insulin(Vergauwenetal;1994).

Compound Information

Entry:39438 Library:NIST11.LIB

Formula:C7H14O6 CAS:0-00-0 MolWeight:194 RetIndex:1647

CompName:3-O-Methyl-d-glucose $$ 3-O-Methylhexose # $$



No.	Mass	R.Int	No.	Mass	R.Int	No.	Mass	R.Int	No.	Mass	R.Int
1	42.00	29.88	18	68.00	2.37	35	88.00	3.86	52	118.00	0.94
2	43.00	41.11	19	69.00	10.88	36	89.00	2.12	53	119.00	0.34
3	44.00	15.56	20	70.00	4.96	37	91.00	1.73	54	121.00	1.55
4	45.00	36.32	21	71.00	29.09	38	97.00	6.89	55	127.00	0.86
5	47.00	6.24	22	73.00	100.00	39	98.00	10.12	56	129.00	0.29
6	49.00	0.39	23	74.00	90.06	40	99.00	5.28	57	131.00	0.81
7	51.00	0.37	24	75.00	25.96	41	100.00	5.85	58	133.00	5.21
8	53.00	2.45	25	76.00	2.38	42	101.00	7.09	59	135.00	1.31
9	54.00	4.26	26	77.00	0.43	43	102.00	6.56	60	144.00	4.94
10	55.00	13.88	27	78.00	0.32	44	103.00	14.23	61	145.00	11.37
11	56.00	10.45	28	81.00	1.35	45	104.00	4.19	62	146.00	0.84
12	57.00	26.24	29	82.00	0.33	46	105.00	5.47	63	158.00	0.36
13	58.00	10.42	30	83.00	1.89	47	106.00	0.37	64	159.00	0.45
14	59.00	23.94	31	84.00	2.47	48	113.00	1.36	65	163.00	5.62
15	60.00	23.02	32	85.00	15.03	49	115.00	2.07	66	177.00	0.68
16	61.00	20.09	33	86.00	4.98	50	116.00	7.00	67	194.00	0.32
17	62.00	0.78	34	87.00	74.40	51	117.00	3.71			

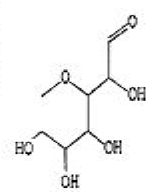


Figure (3.4)Compound Information of 3-O-methyl-D-glucose

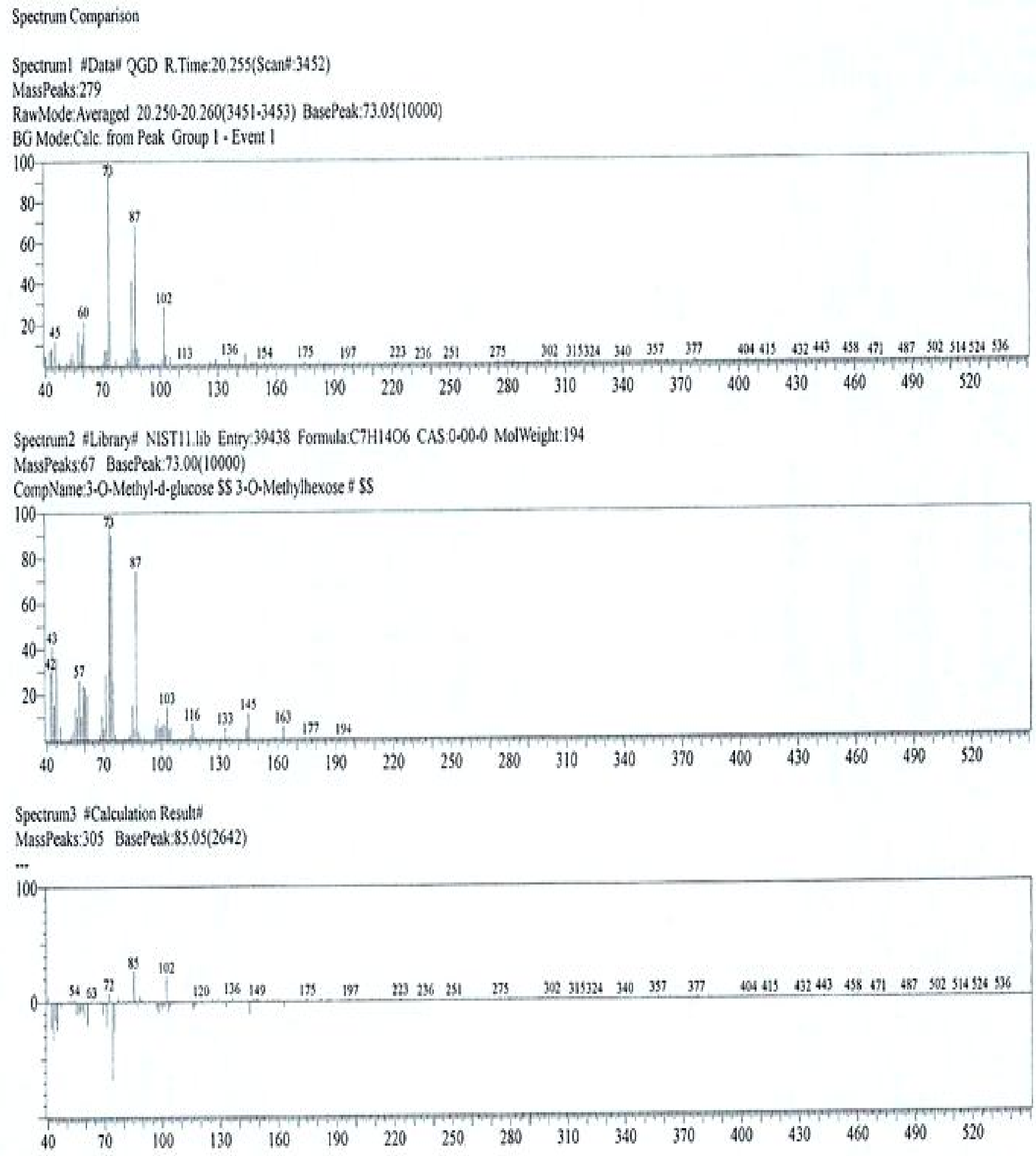


Figure (3.5) Spectrum comparison of 3-O-methyl-D-glucose

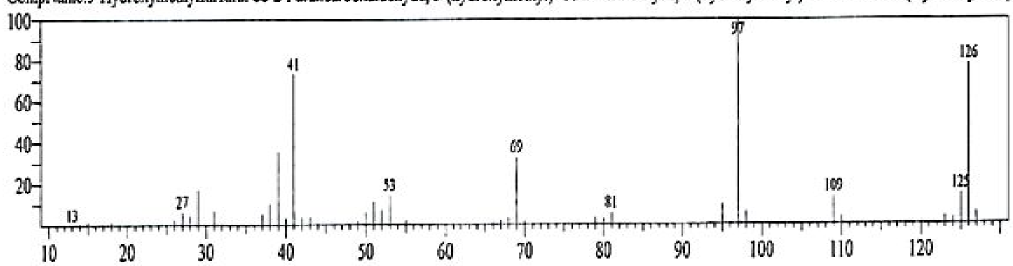
5-Hydroxymethylfurfural(Fig 3.6and3.7)has a (7.49%) in sample,has a formula ($C_6H_6O_4$) and Molecular Weight (126). Hydroxymethylfurfural (HMF), also 5-(Hydroxymethyl)furfural, is an organic compound derived from dehydration of certain sugars. This yellow low-melting solid is highly water-soluble. The molecule consists of a furan ring, containing both aldehyde and alcohol functional groups. HMF has been identified in a wide variety of baked goods. HMF, which is derived from hexoses, is a potential "carbon-neutral" feedstock for fuels and chemicals(Zakrzewskaetal;2011).

Compound Information

Entry:6369 Library:NIST11.LIB

Formula:C6H6O3 CAS:67-47-0 MolWeight:126 RetIndex:1163

CompName:5-Hydroxymethylfurfural $$ 2-Furancarboxaldehyde, 5-(hydroxymethyl)- $$ 2-Furaldehyde, 5-(hydroxymethyl)- $$ HMF $$ 5-(Hyddroxymeth



No.	Mass	R.Int	No.	Mass	R.Int	No.	Mass	R.Int	No.	Mass	R.Int
1	12.00	0.20	21	37.00	5.39	41	60.00	0.20	61	91.00	0.10
2	13.00	0.30	22	38.00	10.19	42	61.00	0.40	62	93.00	0.30
3	14.00	0.80	23	39.00	35.89	43	62.00	0.40	63	94.00	0.30
4	15.00	1.59	24	40.00	3.19	44	63.00	0.20	64	95.00	9.99
5	16.00	0.10	25	41.00	73.30	45	64.00	0.10	65	96.00	1.29
6	17.00	0.40	26	42.00	3.69	46	65.00	0.40	66	97.00	100.00
7	18.00	1.59	27	43.00	3.79	47	66.00	1.79	67	98.00	6.29
8	24.00	0.20	28	44.00	0.60	48	67.00	1.99	68	99.00	0.70
9	25.00	0.70	29	45.00	0.20	49	68.00	3.69	69	108.00	0.30
10	26.00	3.09	30	48.00	0.30	50	69.00	32.69	70	109.00	13.49
11	27.00	6.69	31	49.00	1.99	51	70.00	1.99	71	110.00	3.49
12	28.00	4.59	32	50.00	6.19	52	71.00	0.50	72	111.00	0.30
13	29.00	17.19	33	51.00	11.59	53	75.00	0.20	73	112.00	0.20
14	30.00	0.90	34	52.00	7.19	54	77.00	0.20	74	123.00	3.69
15	31.00	7.49	35	53.00	14.59	55	79.00	2.99	75	124.00	2.49
16	32.00	0.99	36	54.00	0.99	56	80.00	2.89	76	125.00	14.59
17	33.00	0.20	37	55.00	2.29	57	81.00	5.59	77	126.00	78.00
18	34.00	0.40	38	56.00	0.30	58	82.00	0.50	78	127.00	5.69
19	35.00	0.10	39	57.00	0.10	59	83.00	0.10	79	128.00	0.70
20	36.00	0.80	40	58.00	0.20	60	84.00	0.30			

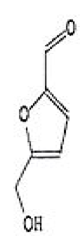


Figure (3.6)Compound Information of5.Hydroxymathylfurfural

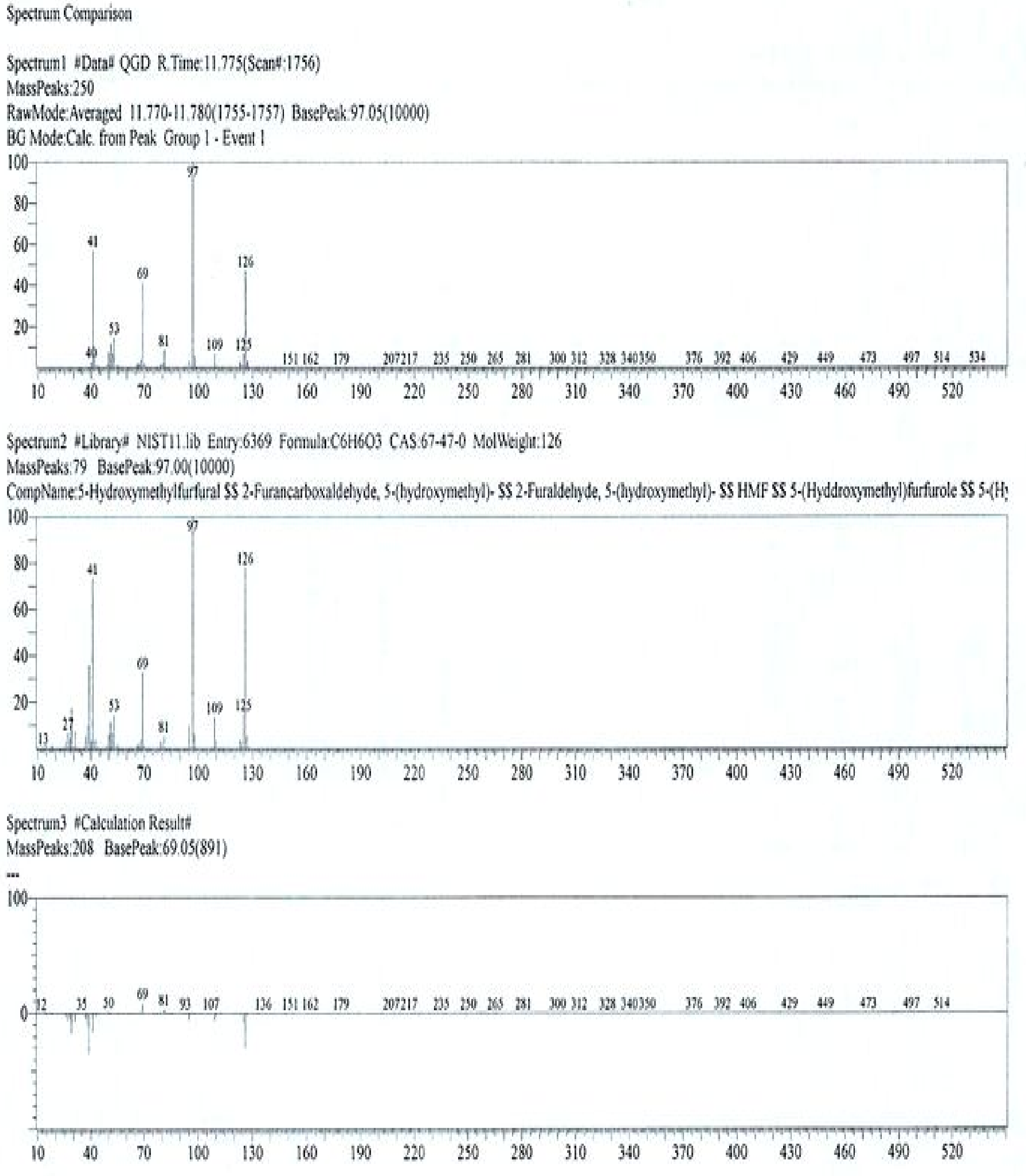


Figure (3.7) Spectrum comparison of 5-Hydroxymethylfurfural

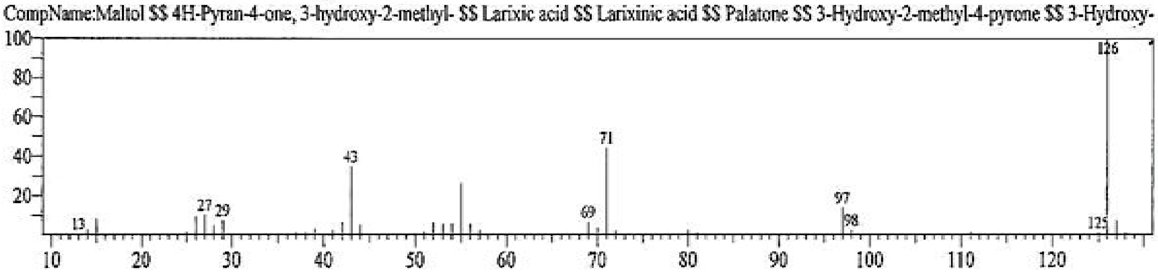
Maltol (Fig 3.8and3.9) represent (3.24%) in sample,maltolhas a formula ($C_6H_6O_3$) and Molecular Weight (126).Maltol is a naturally occurring organic compound that is used primarily as a flavor enhancer. It is found in the bark of larch tree, in pine needles, and in roasted malt (from which it gets its name). It is a white crystalline powder that is soluble in hot water, chloroform, and other polar solvents. Because it has the odor of cotton candy and caramel, maltol is used to impart a sweet aroma to fragrances. Maltol's sweetness adds to the odor of freshly baked bread, and is used as a flavor enhancer (INS Number 636) in breads and cakes. It is not registered as a food additive in the EU and thus has no E-number. Instead, maltol is registered as a flavor component in the EU(Official Journal of the European Union,2011-2012).

Compound Information

Entry:6377 Library:NIST11.LIB

Formula:C6H6O3 CAS:118-71-8 MolWeight:126 RetIndex:1063

CompName:Maltol $$ 4H-Pyran-4-one, 3-hydroxy-2-methyl- $$ Larixic acid $$ Larixinic acid $$ Palatone $$ 3-Hydroxy-2-methyl-4-pyrone $$ 3-Hydroxy-



No.	Mass	R.Int	No.	Mass	R.Int	No.	Mass	R.Int	No.	Mass	R.Int
1	12.00	0.20	19	38.00	1.49	37	58.00	0.50	55	84.00	0.40
2	13.00	0.60	20	39.00	2.89	38	61.00	0.10	56	85.00	0.10
3	14.00	2.69	21	40.00	0.70	39	62.00	0.20	57	92.00	0.10
4	15.00	8.29	22	41.00	2.29	40	63.00	0.10	58	93.00	0.40
5	16.00	0.10	23	42.00	6.79	41	65.00	0.10	59	96.00	0.20
6	17.00	0.10	24	43.00	34.79	42	66.00	0.30	60	97.00	13.79
7	18.00	0.30	25	44.00	4.99	43	67.00	0.40	61	98.00	2.49
8	24.00	0.20	26	45.00	0.30	44	68.00	0.30	62	99.00	0.30
9	25.00	1.39	27	48.00	0.10	45	69.00	6.59	63	109.00	0.20
10	26.00	9.39	28	49.00	0.50	46	70.00	3.19	64	111.00	1.89
11	27.00	10.29	29	50.00	0.80	47	71.00	44.39	65	112.00	0.10
12	28.00	4.49	30	51.00	1.69	48	72.00	1.99	66	125.00	1.09
13	29.00	7.49	31	52.00	6.39	49	73.00	0.30	67	126.00	100.00
14	30.00	0.80	32	53.00	5.49	50	79.00	0.70	68	127.00	6.99
15	31.00	0.30	33	54.00	5.59	51	80.00	2.19	69	128.00	0.80
16	32.00	0.30	34	55.00	26.09	52	81.00	0.90			
17	36.00	0.20	35	56.00	5.79	53	82.00	0.30			
18	37.00	1.19	36	57.00	1.99	54	83.00	0.40			

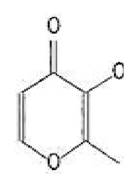


Figure (3.8)Compound Information ofMaltol

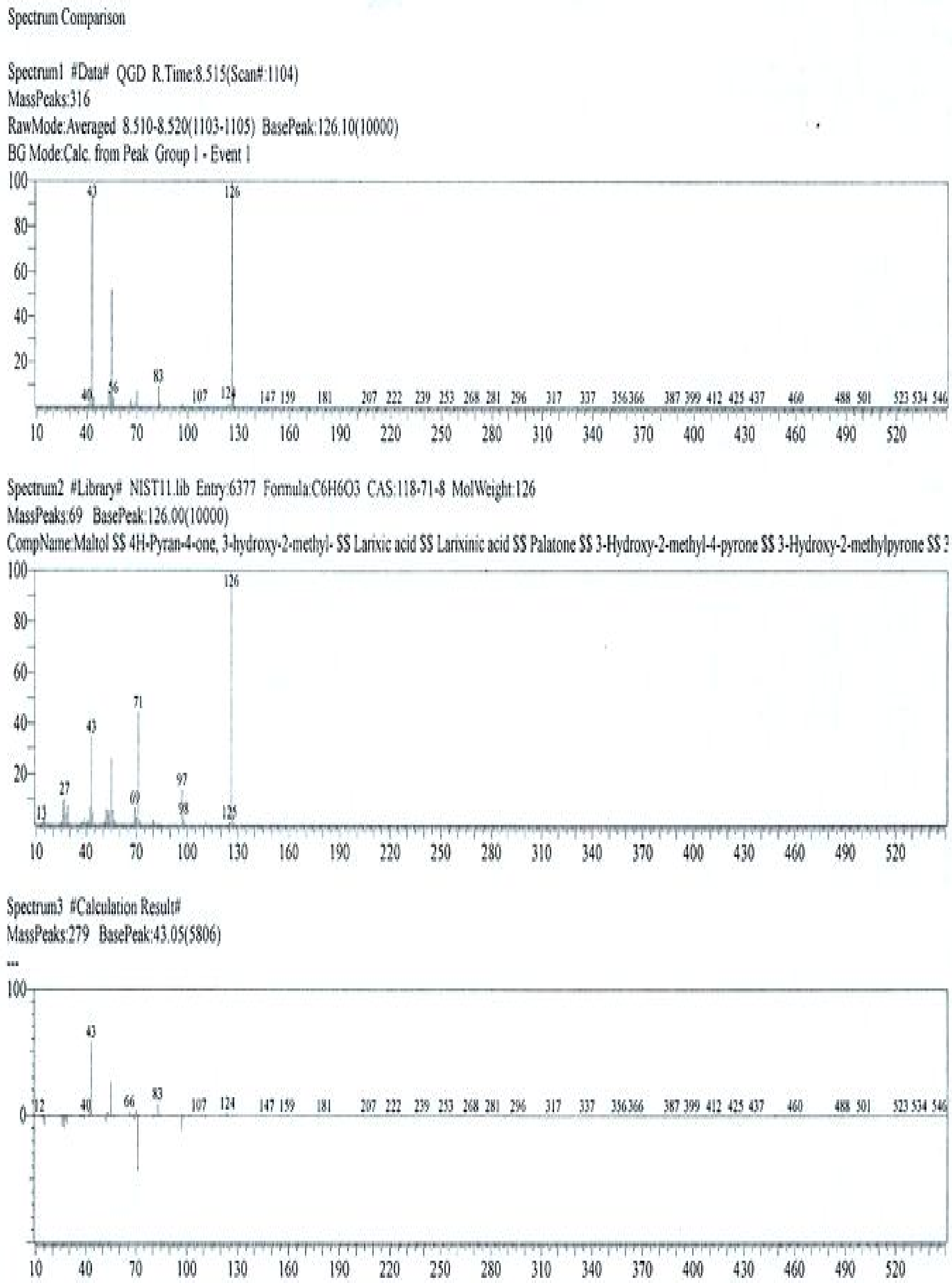


Figure (3.9) Spectrum comparison of Maltol

Pentadecanoicacid (Fig 3.10and3.11)represent(2.28%),Pentadecanoic acid is a saturated fatty acid. Its molecular formula is $CH_3(CH_2)_{13}COOH$ and Molecular Weight (242).

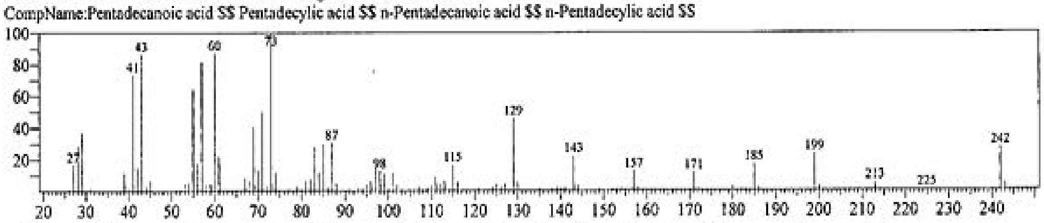
It is rare in nature, being found at the level of 1.2% in the cow's milk fat(Rolf Jost,2002).

Compound Information

Entry:22188 Library:NIST11S.LIB

Formula:C15H30O2 CAS:1002-84-2 MolWeight:242 RetIndex:1869

CompName:Pentadecanoic acid $$ Pentadecylic acid $$ n-Pentadecanoic acid $$ n-Pentadecylic acid $$



No.	Mass	R.Int	No.	Mass	R.Int	No.	Mass	R.Int	No.	Mass	R.Int
1	26.00	1.00	34	70.00	12.80	67	106.00	0.70	100	144.00	3.20
2	27.00	16.70	35	71.00	49.30	68	107.00	2.00	101	145.00	0.40
3	28.00	28.50	36	72.00	2.60	69	108.00	0.70	102	149.00	1.10
4	29.00	36.70	37	73.00	100.00	70	109.00	1.90	103	151.00	0.40
5	31.00	1.70	38	74.00	11.70	71	110.00	3.20	104	152.00	0.90
6	36.00	0.40	39	75.00	0.90	72	111.00	9.10	105	153.00	1.10
7	38.00	0.40	40	76.00	0.30	73	112.00	4.30	106	154.00	1.50
8	39.00	11.30	41	77.00	1.00	74	113.00	6.60	107	155.00	0.30
9	40.00	2.20	42	78.00	0.50	75	114.00	0.90	108	157.00	12.50
10	41.00	73.50	43	79.00	2.70	76	115.00	16.70	109	158.00	1.80
11	42.00	14.70	44	80.00	1.00	77	116.00	5.90	110	163.00	0.50
12	43.00	86.60	45	81.00	6.70	78	117.00	0.60	111	167.00	0.40
13	44.00	2.80	46	82.00	7.40	79	121.00	1.60	112	168.00	0.40
14	45.00	6.60	47	83.00	27.80	80	122.00	0.50	113	171.00	11.70
15	50.00	0.30	48	84.00	11.60	81	123.00	1.00	114	172.00	1.60
16	51.00	0.60	49	85.00	30.20	82	124.00	2.20	115	177.00	0.30
17	52.00	0.40	50	86.00	2.10	83	125.00	4.30	116	180.00	2.60
18	53.00	4.40	51	87.00	30.60	84	126.00	2.00	117	181.00	0.60
19	54.00	5.00	52	88.00	4.90	85	127.00	4.20	118	182.00	1.50
20	55.00	64.20	53	89.00	0.50	86	128.00	0.60	119	183.00	1.00
21	56.00	17.40	54	91.00	0.80	87	129.00	45.70	120	185.00	16.90
22	57.00	81.50	55	93.00	1.90	88	130.00	5.90	121	186.00	2.20
23	58.00	3.60	56	94.00	1.00	89	131.00	0.60	122	187.00	0.30
24	59.00	4.00	57	95.00	4.00	90	133.00	0.40	123	199.00	23.30
25	60.00	87.20	58	96.00	6.20	91	135.00	1.20	124	200.00	3.20
26	61.00	21.90	59	97.00	20.10	92	136.00	0.30	125	201.00	0.40
27	62.00	0.80	60	98.00	12.40	93	137.00	0.50	126	206.00	0.50
28	63.00	0.30	61	99.00	10.40	94	138.00	1.40	127	213.00	5.10
29	65.00	1.10	62	100.00	1.00	95	139.00	1.90	128	214.00	0.80
30	66.00	0.90	63	101.00	11.40	96	140.00	1.20	129	225.00	0.60
31	67.00	8.30	64	102.00	4.10	97	141.00	1.80	130	242.00	27.20
32	68.00	6.10	65	103.00	0.50	98	142.00	0.30	131	243.00	4.60
33	69.00	40.50	66	105.00	0.30	99	143.00	22.00	132	244.00	0.50

Figure (3.10)Compound Information ofPentadecanoic acid

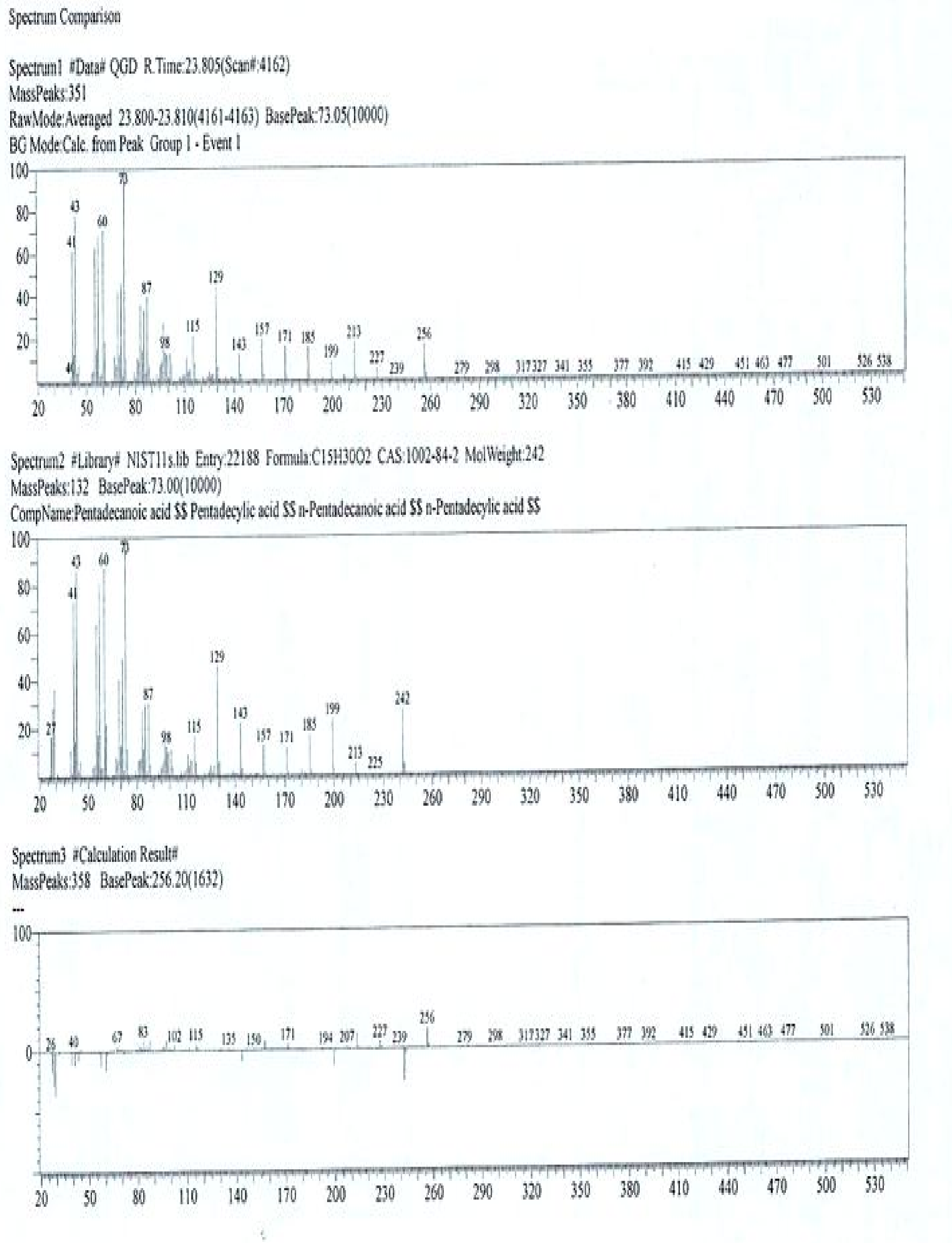


Figure (3.11) Spectrum comparison ofPentadecanoic acid

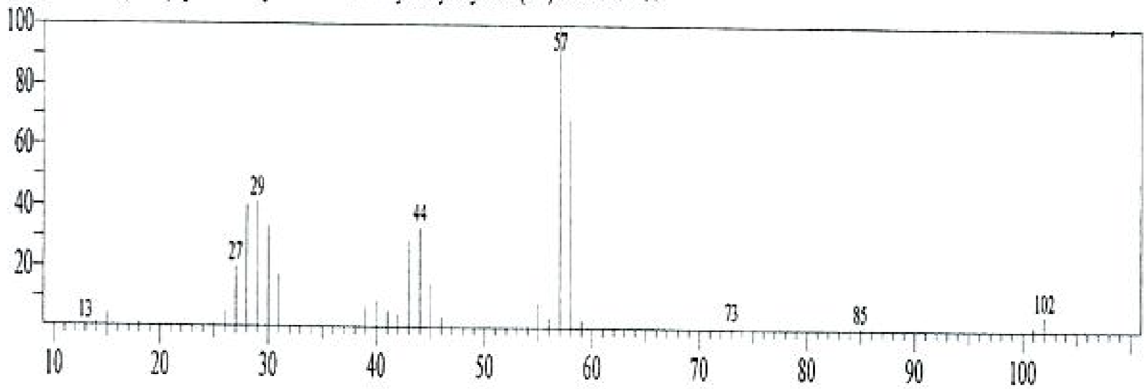
2-Hydroxy-gamma-butyrolactone (Fig 3.12 and 3.13)represented (2.24%) in sample. It has the Formula ($C_4H_6O_3$) and Molecular Weight (102).

Compound Information

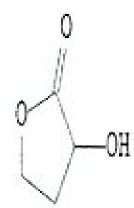
Entry:2215 Library:NIST11.LIB

Formula:C4H6O3 CAS:19444-84-9 MolWeight:102 RetIndex:1013

CompName:2-Hydroxy-gamma-butyrolactone $$ 3-Hydroxydihydro-2(3H)-furanone # $$



No.	Mass	R.Int	No.	Mass	R.Int	No.	Mass	R.Int	No.	Mass	R.Int
1	12.00	0.10	15	31.00	17.19	29	52.00	0.10	43	74.00	0.20
2	13.00	0.30	16	32.00	0.60	30	53.00	0.70	44	75.00	0.30
3	14.00	1.29	17	36.00	0.20	31	54.00	0.70	45	76.00	0.10
4	15.00	4.29	18	37.00	0.70	32	55.00	7.99	46	80.00	0.10
5	16.00	0.30	19	38.00	0.90	33	56.00	3.39	47	82.00	0.10
6	17.00	0.30	20	39.00	6.69	34	57.00	100.00	48	84.00	0.60
7	18.00	0.99	21	40.00	8.79	35	58.00	69.20	49	85.00	0.90
8	24.00	0.10	22	41.00	5.39	36	59.00	2.59	50	86.00	0.60
9	25.00	0.80	23	42.00	4.19	37	60.00	0.20	51	99.00	0.10
10	26.00	5.19	24	43.00	28.49	38	68.00	0.20	52	100.00	0.10
11	27.00	19.79	25	44.00	33.09	39	69.00	0.20	53	101.00	1.49
12	28.00	40.39	26	45.00	14.39	40	71.00	0.50	54	102.00	5.09
13	29.00	41.29	27	46.00	3.39	41	72.00	0.40	55	103.00	0.20
14	30.00	33.29	28	47.00	0.80	42	73.00	1.19			



Figure(3.12)Compound Information of 2-Hydroxy-gamma-butyrolactone

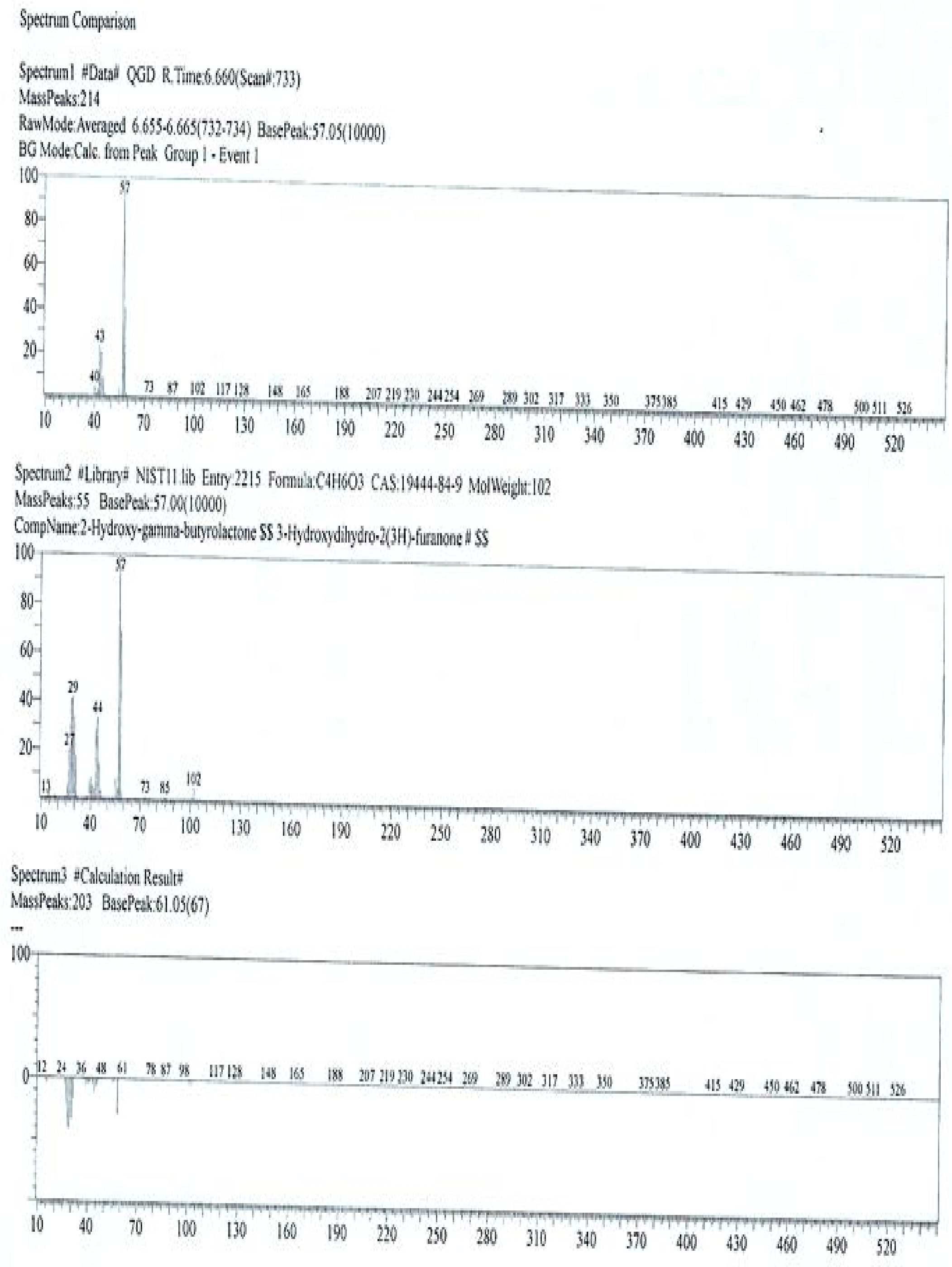


Figure (3.13) Spectrum comparison of2-Hydroxy-gamma-butyrolactone

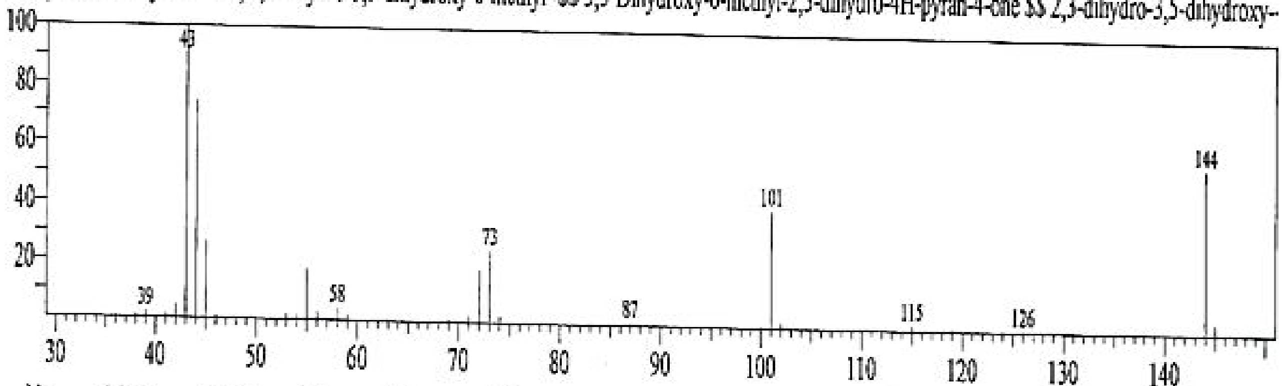
4H-pyran-4-one, 2,3-dihydro-3,5-dihydroxy-6-methyl (Fig 3.14 and 3.15) also known as 2,3-Dihydro-3,5-dihydroxy-6-methyl-4-pyrone,represented (2.80%).It has theFormula($C_6H_8O_4$)and Molecular Weight(144).

Compound Information

Entry:7819 Library:NIST11S.LIB

Formula:C6H8O4 CAS:28564-83-2 MolWeight:144 RetIndex:1269

CompName:4H-Pyran-4-one, 2,3-dihydro-3,5-dihydroxy-6-methyl- $$ 3,5-Dihydroxy-6-methyl-2,3-dihydro-4H-pyran-4-one $$ 2,3-dihydro-3,5-dihydroxy--



No.	Mass	R.Int	No.	Mass	R.Int	No.	Mass	R.Int	No.	Mass	R.Int
1	36.00	1.05	13	54.00	1.90	25	73.00	24.90	37	115.00	2.02
2	38.00	1.20	14	55.00	17.76	26	74.00	2.15	38	116.00	0.49
3	39.00	2.19	15	56.00	2.80	27	83.00	0.67	39	119.00	0.76
4	40.00	0.67	16	57.00	0.53	28	84.00	0.48	40	124.00	0.53
5	41.00	1.70	17	58.00	4.32	29	85.00	0.99	41	126.00	0.60
6	42.00	4.80	18	59.00	1.52	30	87.00	1.01	42	134.00	0.37
7	43.00	100.00	19	60.00	0.35	31	97.00	0.49	43	144.00	56.34
8	44.00	74.24	20	68.00	0.35	32	100.00	0.88	44	145.00	3.70
9	45.00	26.48	21	69.00	1.06	33	101.00	40.28	45	146.00	0.35
10	46.00	1.05	22	70.00	0.42	34	102.00	1.98			
11	47.00	0.45	23	71.00	1.98	35	103.00	0.52			
12	53.00	1.97	24	72.00	18.20	36	105.00	0.26			

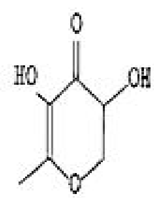


Figure (3.14)Compound Information of 4H-pyran-4-one, 2,3-dihydro-3,5-dihydroxy-6-methyl

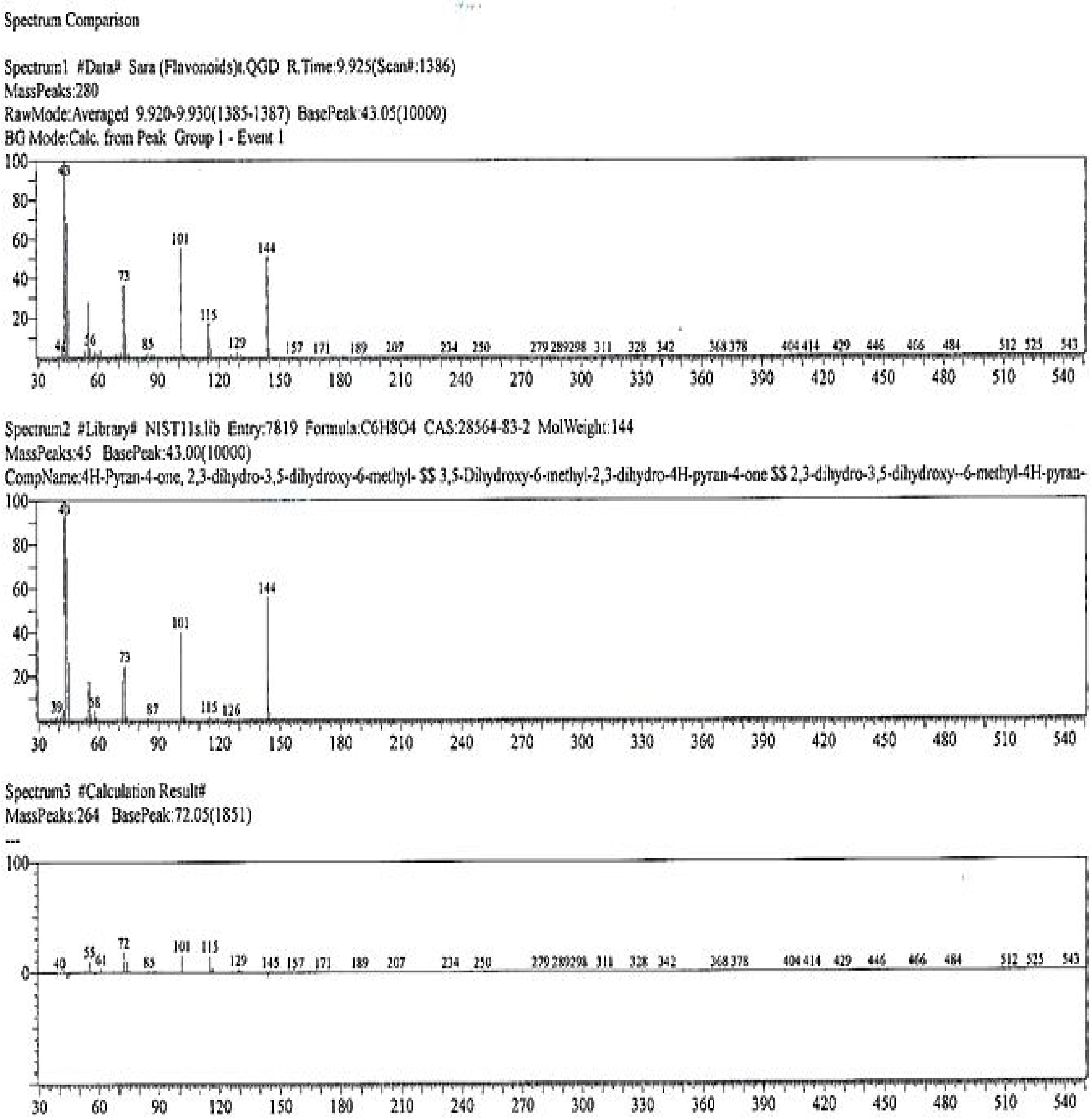


Figure (3.15) Spectrum comparison of4H-pyran-4-one, 2,3-dihydro-3,5-dihydroxy-6-methyl

3.2XRF Results of dry fruits of *HyphaeneThebaica*

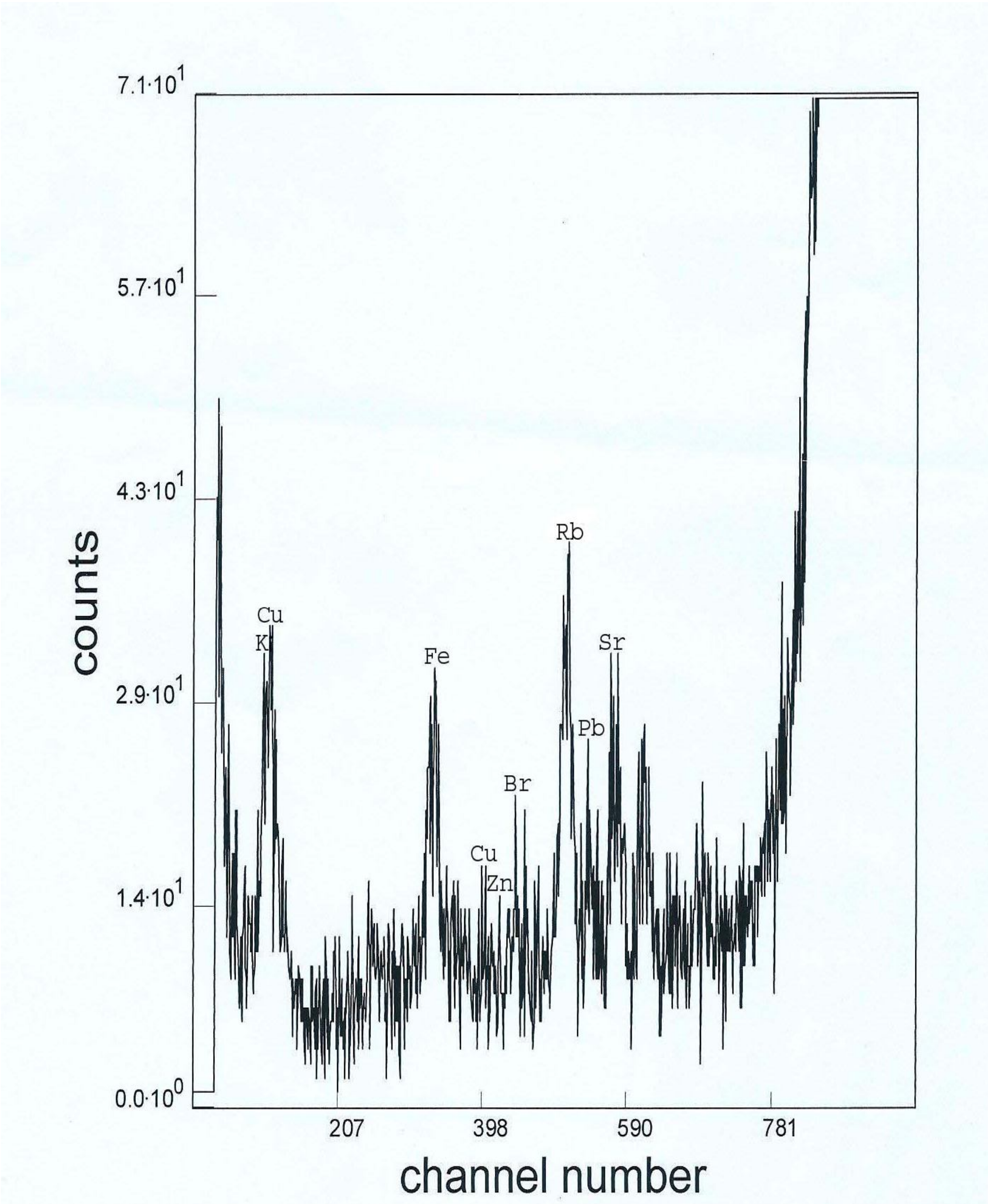
Table (3.2) and Figure (3.16) shows theMinerals content of the doum fruits.The XRFanalysis indicates to presence of (9) elements (K, Ca, Fe, Cu,Zn, Pb, Br, Rb and Sr).potassium showed the highest concentration (2.66%),calcium(0.373%), iron(0.00596%) and Bromine (0.00447%).

The other element showed low concentration, strontium(0.00163%),cupper (0.0016%), rubidium (0.0015%),zinc (0.000549%) and lead (0.000126%).

When comparing this results with that obtained byNawaletal (2014)There were some elements did not appeared in this study like (P,Mg,Mn).Nawal A Abdel-Rahman todetermined Minerals used atomic absorption spectrophotometer and flame photometer. Potassium content was almost found to be the same in the two studies.

Table(3.2) X-Ray fluorescence results of doum fruit

EL	CONC %	CONC% ppm
K	2.66	26600
Ca	0.373	3730
Fe	0.00596	59.6
Br	0.00447	44.7
Sr	0.00163	16.3
Cu	0.0016	11.6
Rb	0.0015	15.0
Zn	0.0005	5.49
Pb	0.000127	1.27



Figure(3.16)X-Ray fluorescence spectrum of doum fruit powder

Conclusion

- This study showed that*Hyphaenethebaica*doum fruits contain a wide range of fragmented chemical compounds.
- The presence of so many components, with different biological and medicinal properties makes the fruit essential for human use.
- The literature review of the previous studies indicates a worldwide interest in doum uses.
- In Sudan very few studies were carried on this important fruit.
- GC-MS analysis showed a considerable content of chemical fragments, such asTris(hydroxymethyl) nitromethane , 3-O-methyl-D-glucose,dl-Glyceraldehdedimer, 5-Hydroxymethyl furfuraland Maltol.
- X-ray fluorescence analysis indicates that doum fruit is rich in potassium, calcium and iron.

Recommendation

- More and further studies may be needed, about the benefits of doum palm fruits, because these fruits are used by Sudanese children as one of the popular foods.
- The previous studies in other countries showed the presence of many chemical constituents especially phenolic and flavonoid compounds, such studies may need to be applied to Sudan *Hyphaenethebaica*(doum) which is a wild plant.
- According to its essential uses it may need to be cultivated as a domestic tree.
- The production may be in large quantities, since the tree can grow almost in all climates of Sudan.

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