Use of Microcrystalline Waxes in Candles

Presented to ALAFAVE and the NCA by
The International Group, Inc (IGI)

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THE INTERNATIONAL GROUP, INC.

Presentation Overview

- Part I: Chemical Makeup, Manufacturing and Product Origins, Commercial Use and Markets
- Part II: Manufacturing, commercial uses, and market conditions
- Part III: Review of Lab Work and Testing Utilizing Microcrystalline Waxes in Candle Applications
- Part IV: Recommendations

Discussion of Base Properties and Fundamentals

Paraffin compared to microcrystalline wax

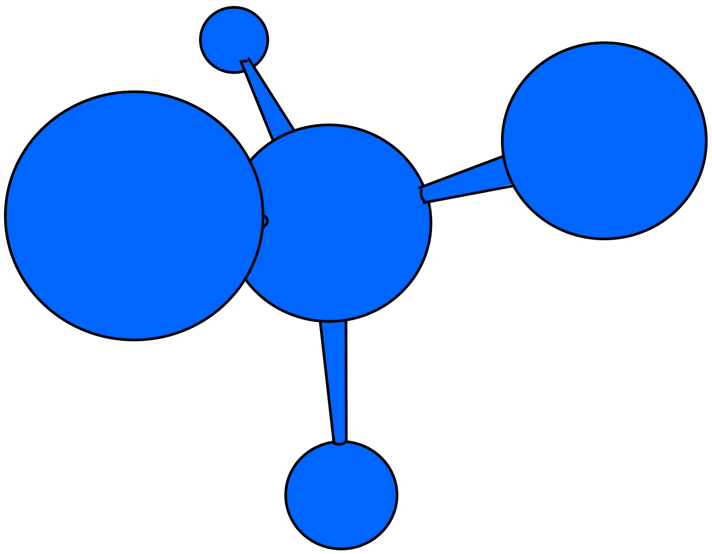
Petroleum Wax-Basic Properties

- All petroleum derived waxes are complex mixtures of many different hydrocarbons. Each individual component possesses its own melting point, viscosity, penetration, etc.
- The physical properties of a wax are an average of the physical properties of all of these components

Wax Classifications

- **Paraffin**
  - Low melting
  - White
  - Hard
  - Brittle
  - Translucent
  - Crystalline
  - Glossy
- **Microcrystalline**
  - Higher melting
  - Colored
  - Soft
  - Malleable
  - Opaque
  - Amorphous
  - Adhesive

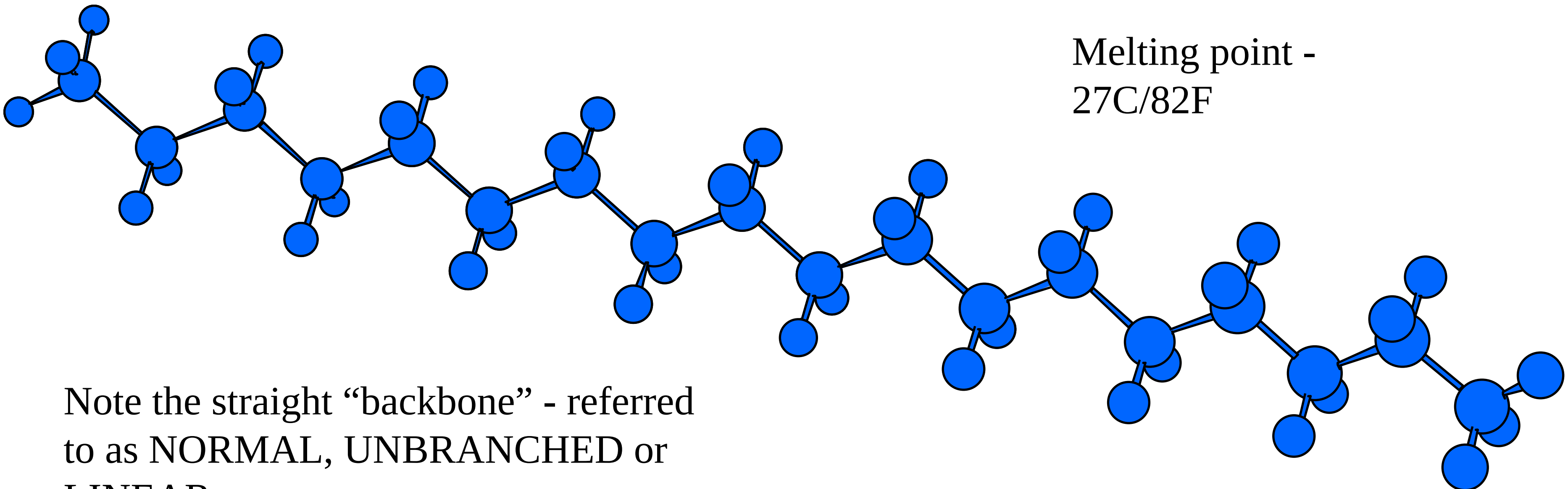
The simplest paraffin or “alkane” is METHANE with the molecular formula CH_4 (1 Carbon, 4 Hydrogen Atoms)



Paraffin, Intermediate & Microcrystalline waxes are all fully saturated hydrocarbon mixtures with the formula:

$$C_nH_{2n+2}$$

Alkanes with 6 - 16 carbon atoms are typically liquids at room temperature **- the first real waxy solid is OCTADECANE - $C_{18}H_{38}$**



Melting point - 27C/82F

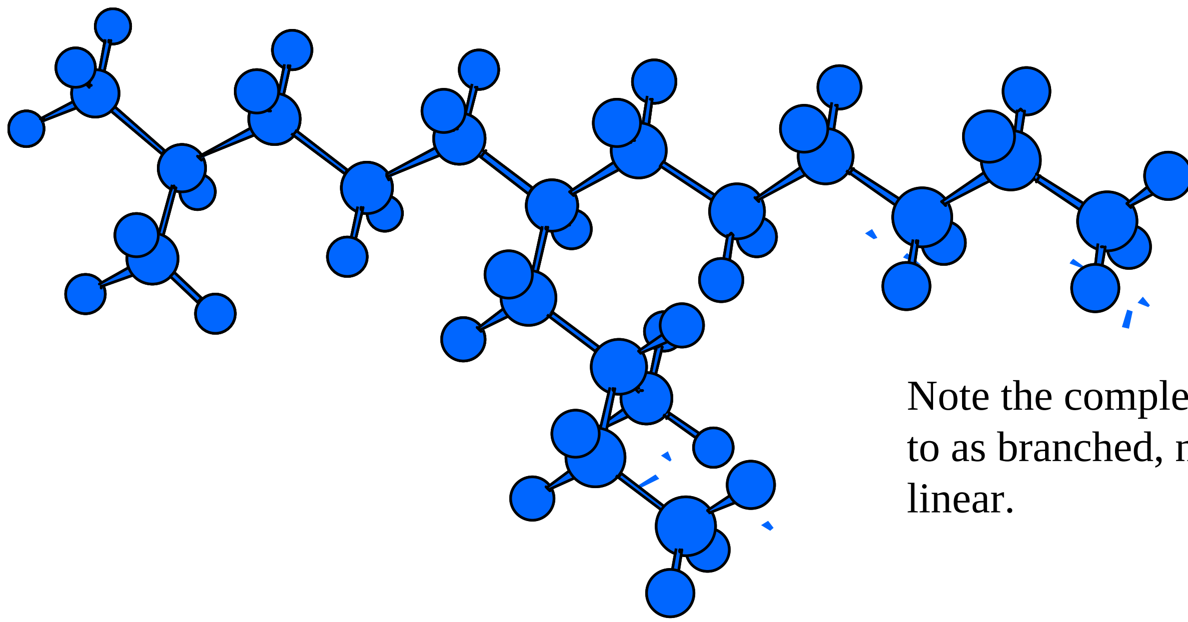
Note the straight “backbone” - referred to as NORMAL, UNBRANCHED or LINEAR

Typical of PARAFFIN waxes

PARAFFIN MICROPHOTOGRAPH x200

As the molecular weight increases, there are many more ways to arrange the carbon and hydrogen atoms and still satisfy the bonding requirements of the elements, i.e. Carbon = 4

Hydrogen = 1

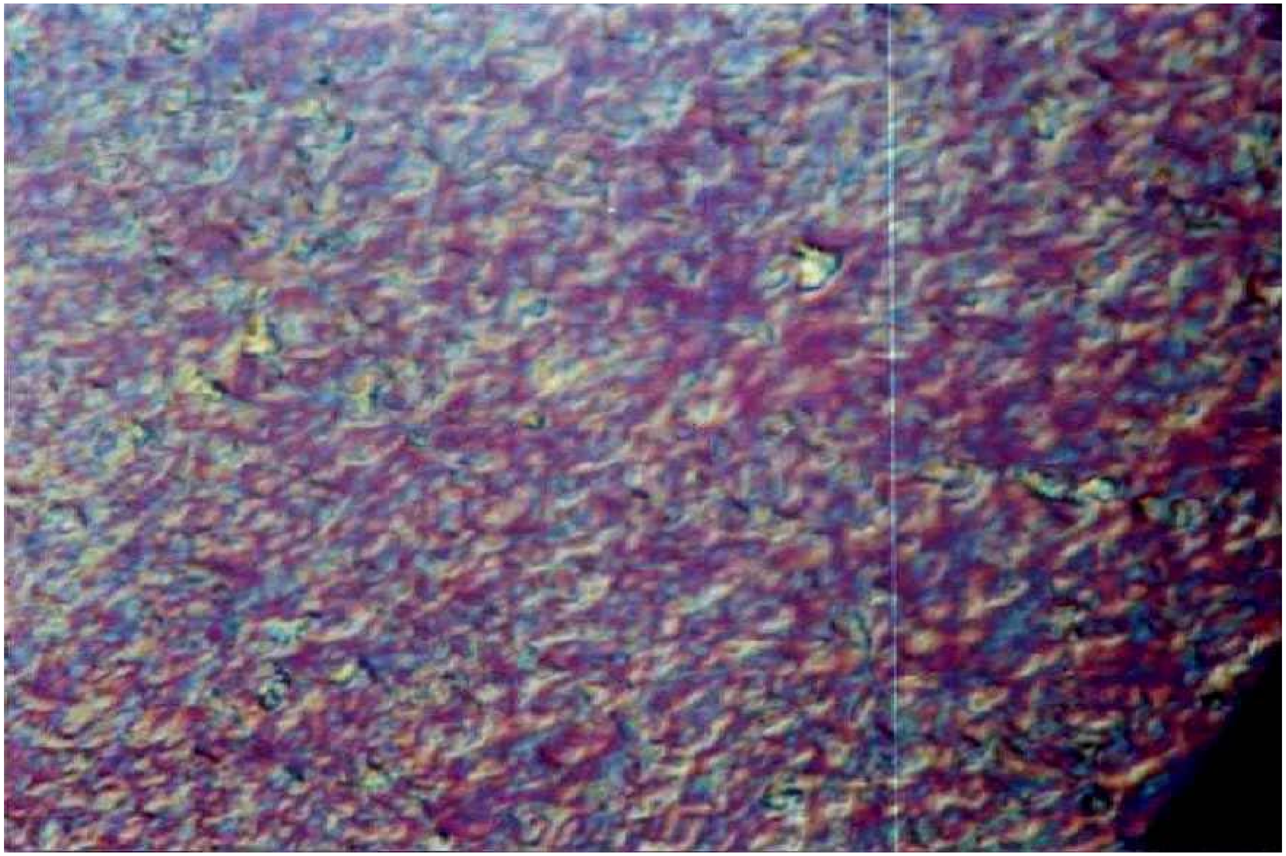


Note the complex structure - referred to as branched, non-normal or non-linear.

The 18 carbon atoms and 38 hydrogen atoms can arrange themselves - eg

Typical of <u>MICROCRYSTALLINE</u> waxes

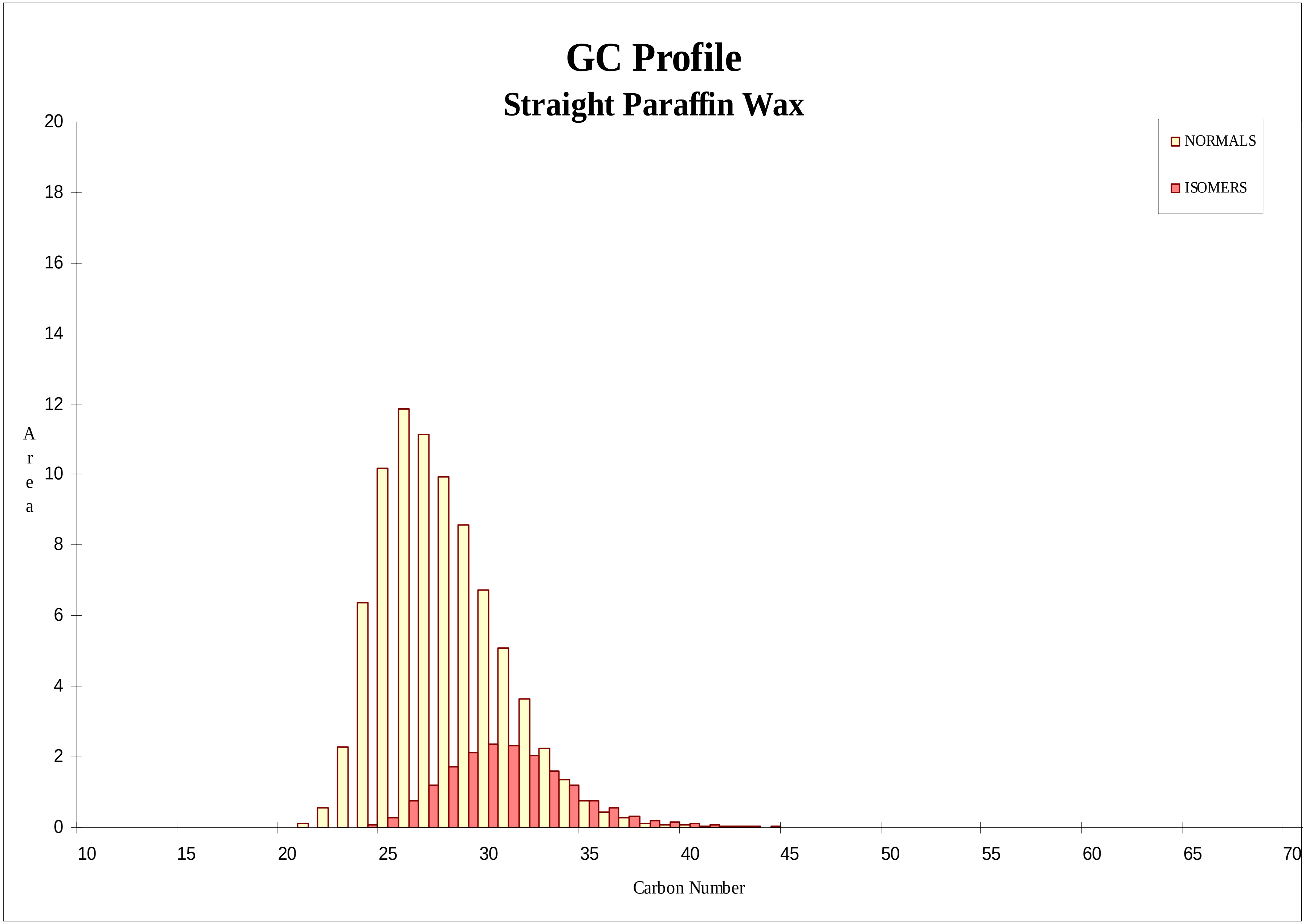
MICROCRYSTALLINE PHOTO X200

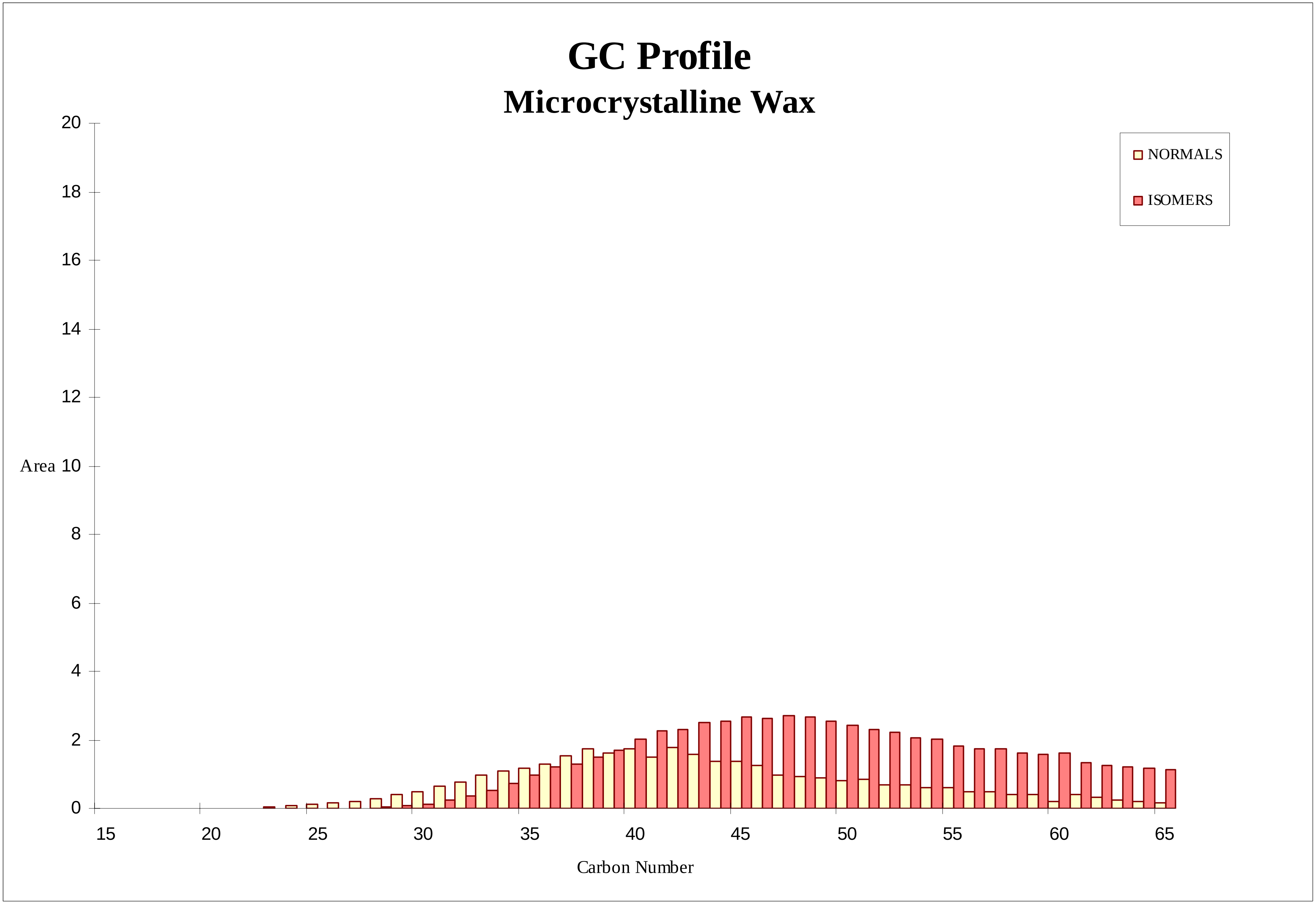


Effect of Branching

		Melting Point	Viscosity
Linear C24	**=**	**51.5C / 125F**	**2.4cps @ 90C**
2 methyl C23	**=**	**42C / 108F**	**2.5cps @ 90C**
2,2 dimethyl C22	**=**	**34.6C / 94F**	**2.7cps @ 90C**

A very minor amount of branching SIGNIFICANTLY effects all the physical properties - e.g. melting point, viscosity and penetration (hardness)


GC Profile
Straight Paraffin Wax
NORMALS
ISOMERS
Area
0
2
4
6
8
10
12
14
16
18
20
10
15
20
25
30
35
40
45
50
55
60
65
70
Carbon Number


GC Profile
Microcrystalline Wax
NORMALS
ISOMERS
Area
20
18
16
14
12
10
8
6
4
2
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15
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25
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35
40
45
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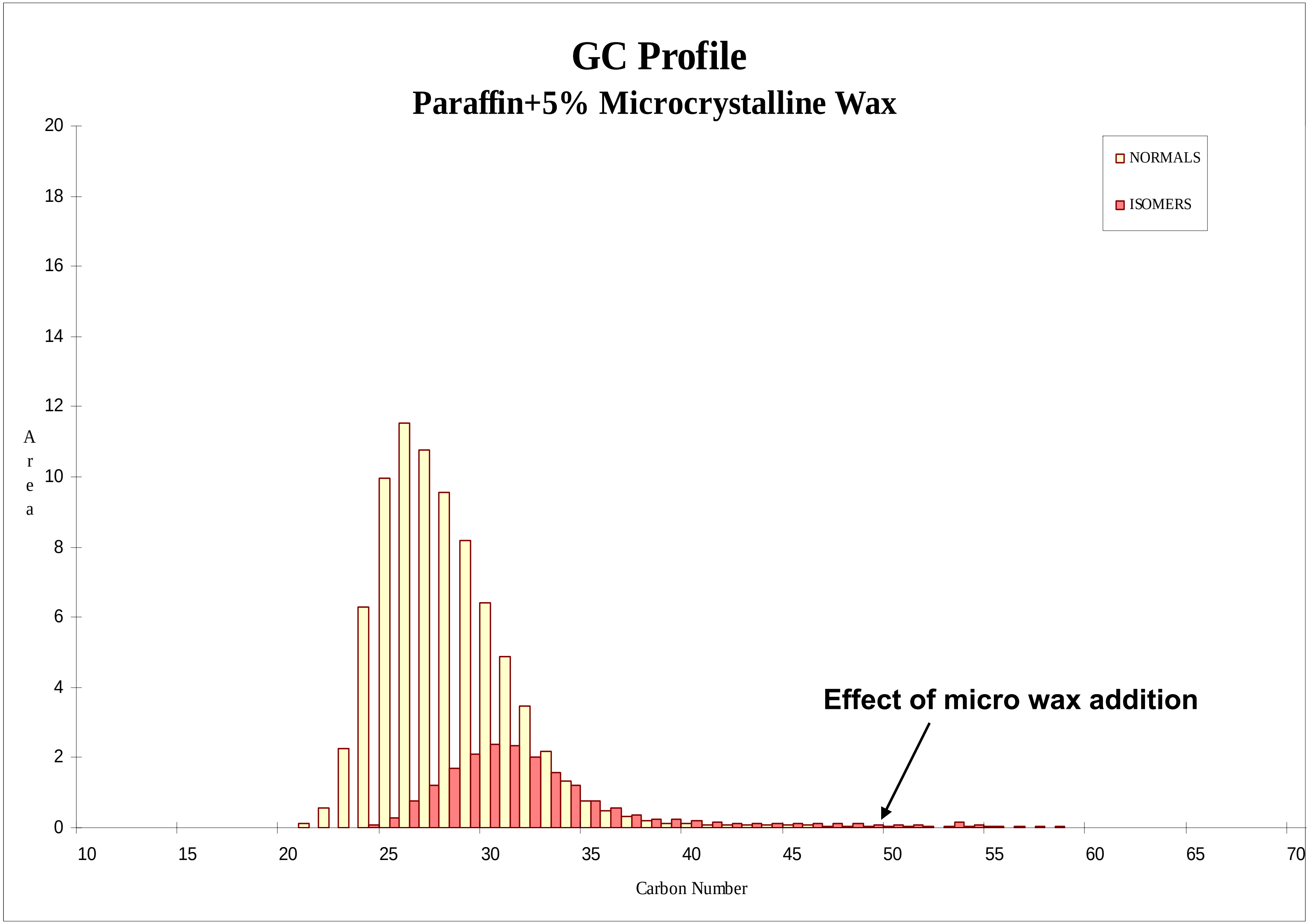
55
60
65
Carbon Number


GC Profile
Paraffin+5% Microcrystalline Wax
NORMALS
ISOMERS
Area
Carbon Number
Effect of micro wax addition
0
2
4
6
8
10
12
14
16
18
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45
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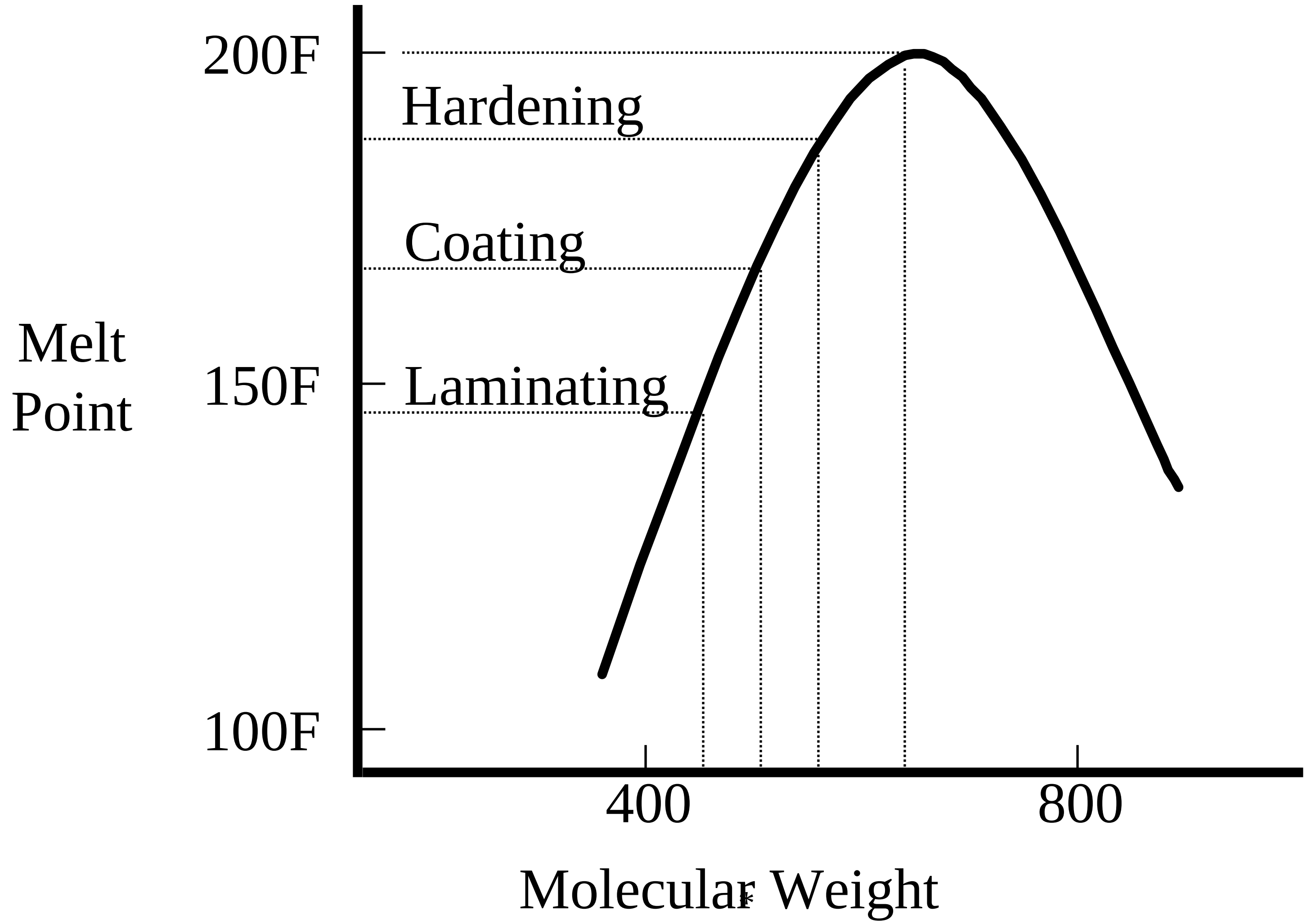
65
70

Microcrystalline Wax

Melt Point versus Molecular Weight



In General

Paraffin Waxes - mainly linear C18 to C40
Intermediate Waxes - Increased branching C25 to C60
MicroWaxes - Little or no linear HC
Complex, branched structure
Many components C25 to C85

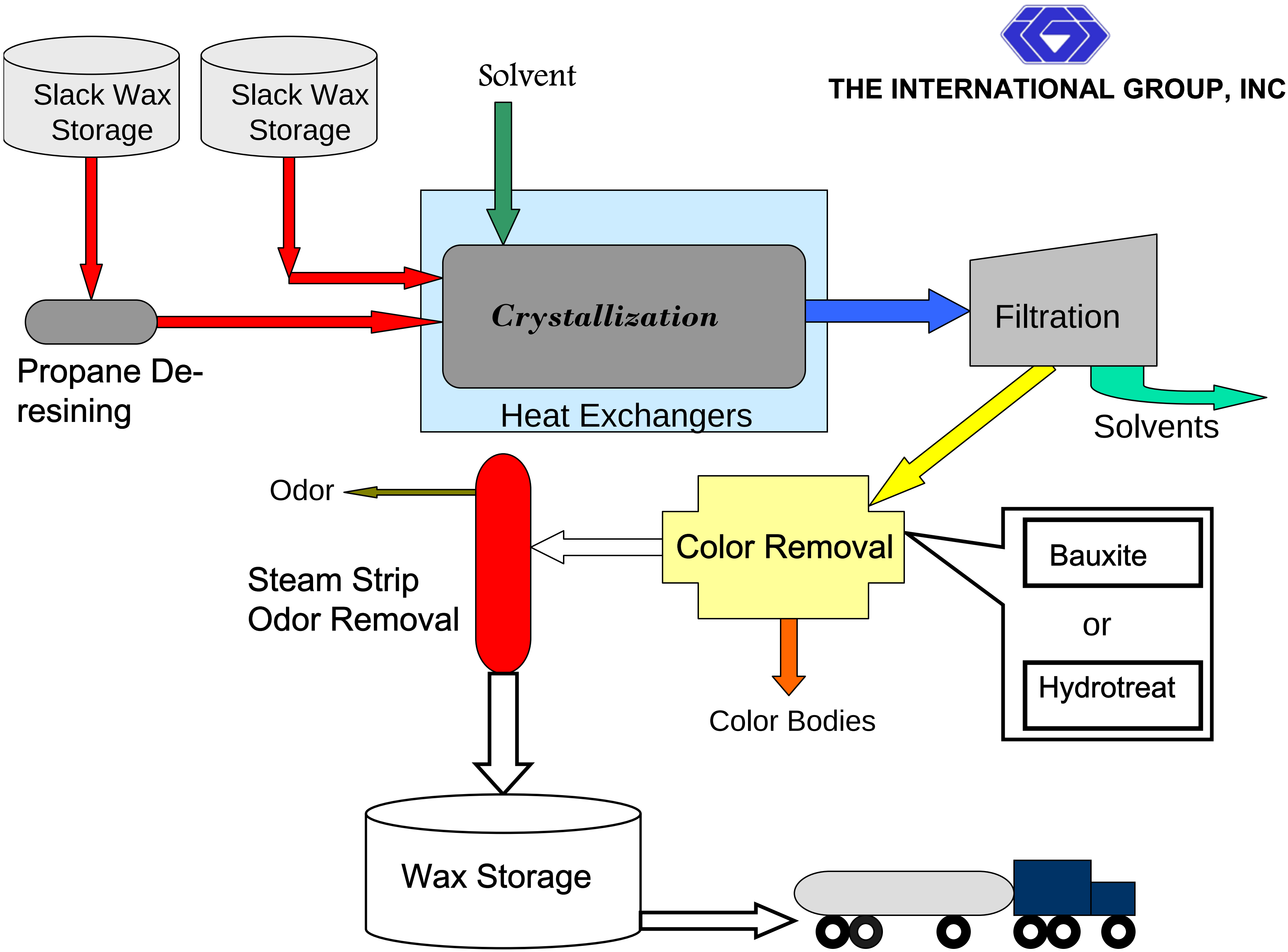
The properties of “intermediate” waxes are between those of paraffins & micro’s.

Manufacture of Microcrystalline Waxes

Methods and Processes

Raw Materials Origins

- Microcrystalline waxes are derived from by-products of other petroleum processes
- Petroleum lubricant manufacture is often the source of these by products
- This by-product stream, or “slack wax”, is the residual lubricant stock, or “bottoms” product of the vacuum distillation process used at the oil refinery


THE INTERNATIONAL GROUP, INC.
Slack Wax Storage
Slack Wax Storage
Solvent
Crystallization
Heat Exchangers
Filtration
Solvents
Propane De-resining
Odor
Steam Strip Odor Removal
Color Removal
Bauxite
or
Hydrotreat
Color Bodies
Wax Storage

Commercial Use of Microcrystalline Waxes

Industries, Applications, and
Market Conditions

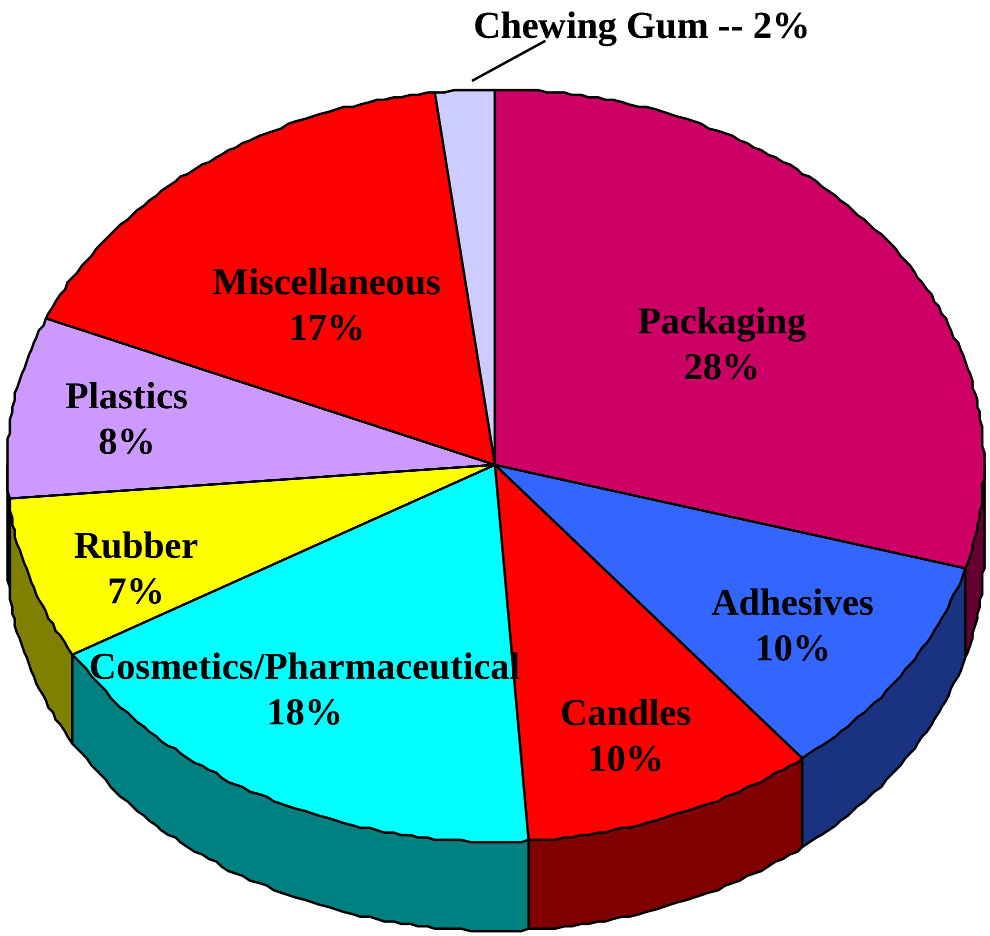
Microcrystalline Wax Classifications

Micro Type	**Melt Point** (°F/°C)	**Needle Penetration** (dmm)	**Properties**
Laminating Grade	130-170°F 54.4-76.7° C	20-40	Flexible, Tacky
Coating Grade	170-185°F 76.7-85°C	15-25	Harder, Low Tackiness
Hardening Grade	185-200°F 85-93.3°C	5-12	Very Hard, Higher Viscosity

Uses of Micro Grades

Laminating Grades	Packing, Adhesives, Cosmetics, Rubber, Candles
Coating Grades	Adhesives, Packaging, Chewing Gum, Inks, Plastics, Rubber
Hardening Grades	Adhesives, Inks, Chewing Gum, Candles, Specialty

Market Division of Microcystalline Waxes



Global Microwax Supply

(Production - MT's)

	Laminating	Coating	Hardening	Total
South America	3,000	15,000	0	18,000
Asia	4,000	25,000	2,000	31,000
Africa/ Middle East	2,000	4,000	0	6,000
Europe	38,000	25,000	4,000	67,000
North America	15,000	15,000	6,000	36,000
Totals	62,000	84,000	12,000	158,000

Using Microcrystalline Waxes in Candle Applications

Benefits and Lab Work

Benefits of Microcrystalline Waxes in Candle Systems

- Appearance
  - Increased opacity
  - Aides in obtaining smooth surface, eliminates mottling/crystallization spots
  - In jar candles, can aide and promote adhesion to glass
- Fragrance Properties
  - Retention, reduction in fragrance bleed
- Structural Properties
  - Strengthens crystal structure, imparted properties on candle molecular structure
  - Increased rigidity and/or flexibility (for wick waxes)

Lab Work Focus on Microcrystalline Waxes

- Reviewed three main applications:
  - Container Candles
  - Pillar/Votive Molded Candles
  - Compression Candles
- Utilized various types and % of micros to examine/quantify the following properties:
  - Opacity
  - Structural Properties
  - Fragrance Bleed
  - Compression Properties

Microcrystalline Waxes Evaluated

Designation	Type	Congeal Point, °F (ASTM D938)	Color (ASTM D6045)	Needle Pen, dmm (ASTM D1321)
A	Laminating Grade	140	+16	35
B	Laminating Grade	170	+16	30
C	Laminating Grade	175	+16	30
D	Hardening Grade	185	+16	18

Quantifying and Examining Opacity

- Purpose:
  - Demonstrate how the branching effect of a microcrystalline wax will effect the crystalline structure of a paraffin and its opacity
  - Demonstrate the effect microcrystalline wax will have on the opacity of a candle system
  - Allow candle manufacturers to predict and determine how changes to their formula will alter the overall opacity of their color system

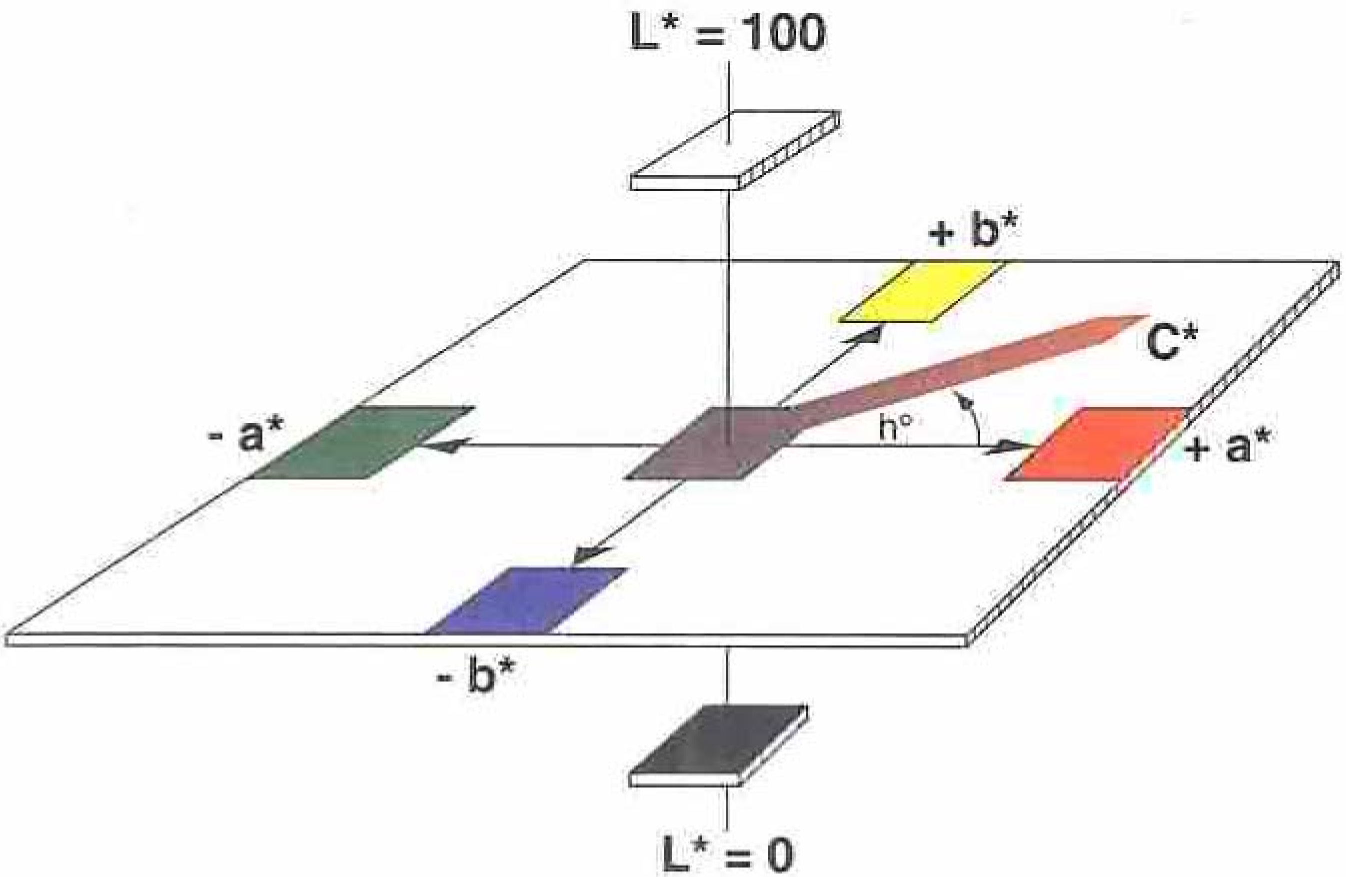
Quantifying and Examining Opacity

- Same system as utilized in 2004 NCA presentation
- System setup: Lovibond RT 100 Reflectance Color Measurement System
  - Measurements Done Using the D65 Illuminant
  - Theoretical Average Daylight with a Colour Temperature of approx 6500°K
  - Analysis of Data Designed to Express Results Measured in Co-ordinate Systems as well as Differences

Opacity/Color Measurement Ranges

- Basis: Eye Cone Receptor Information Coded into Light-Dark, Red-Green and Yellow-Blue Signals Before Reaching Brain

- Color Co-ordinates in CIE L*a*b* system:
  - **L* - Lightness Co-ordinate**
  - a* - Red/Green Co-ordinate
  - b* - Yellow/Blue Co-ordinate



L* = 100
+ b*
C*
- a*
h°
+ a*
- b*
L* = 0

Procedure

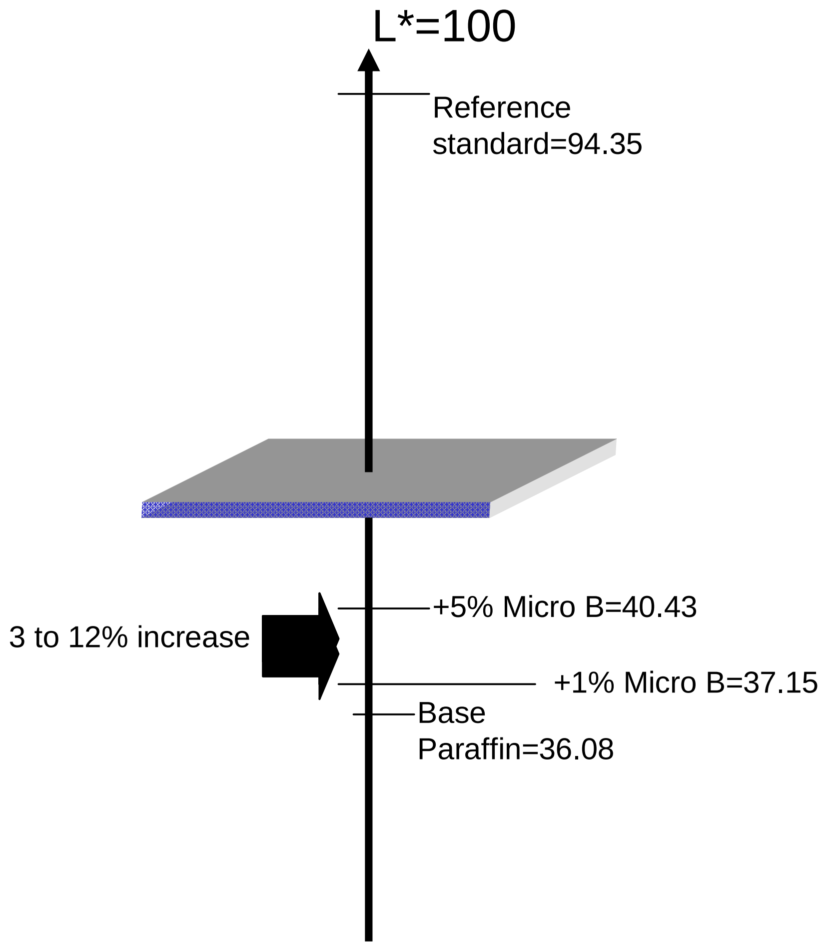
- Mid mp paraffin (135 F) blended with the following microcrystalline waxes:
  - 1, 3, and 5% “Micro B” (Laminating Grade)
  - 1, 3, and 5% “Micro D” (Hardening Grade)
- Low melt point scale wax (125 F mp) blended in the following combinations:
  - 1, 3, and 5% “Micro A” (low mp laminating grade)
  - 1, 3, and 5% “Micro B” (laminating grade)
  - 1, 3, and 5% “Micro C” (laminating grade)
- Base paraffin and blend measured on Lovibond RT-100 measurement system

Opacity Metering Equipment

- Lovibond RT100 Reflectance Tintometer

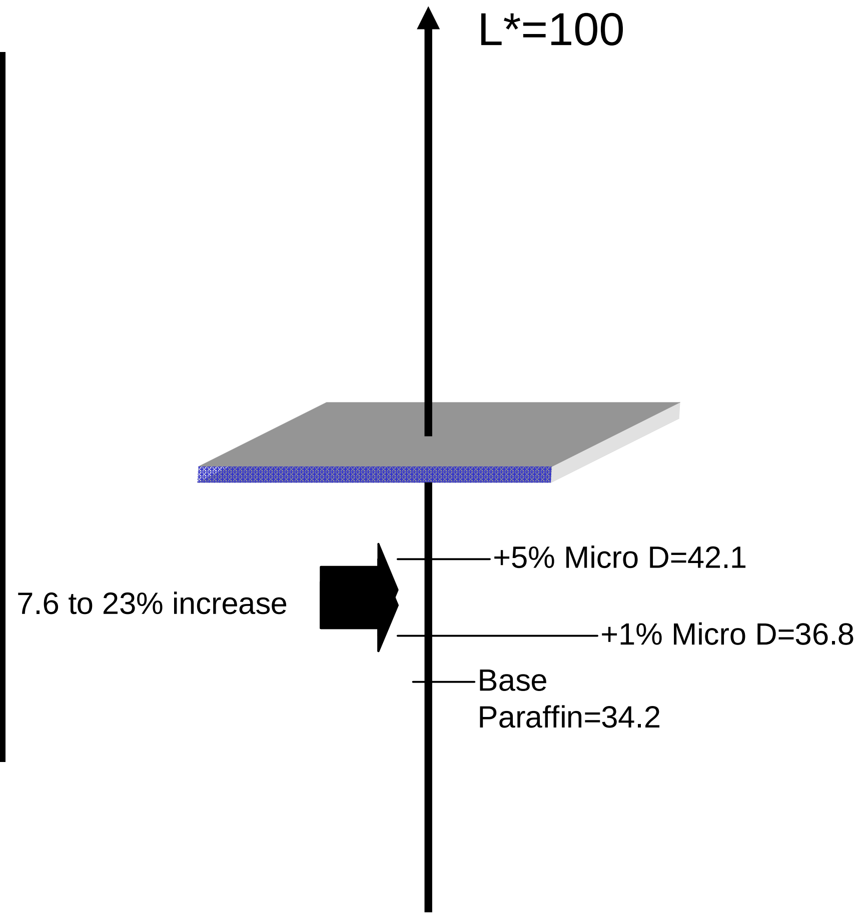
Measured Opacity Results-Low MP

Blend	Average L* Value
100% 125 mp paraffin	36.08
+1% Micro A	37.34
+3% Micro A	38.45
+5% Micro A	40.31
+1% Micro B	37.15
+3% Micro B	38.88
+5% Micro B	40.43
+1% Micro C	37.39
+3% Micro C	38.35
+5% Micro C	40.24



Measured Opacity Results-Mid MP

Blend	Average L* Value
100% 135 mp paraffin	34.2
+1% Micro B	37.8
+3% Micro B	39.8
+5% Micro B	41.1
+1% Micro D	36.8
+3% Micro D	40.1
+5% Micro D	42.1



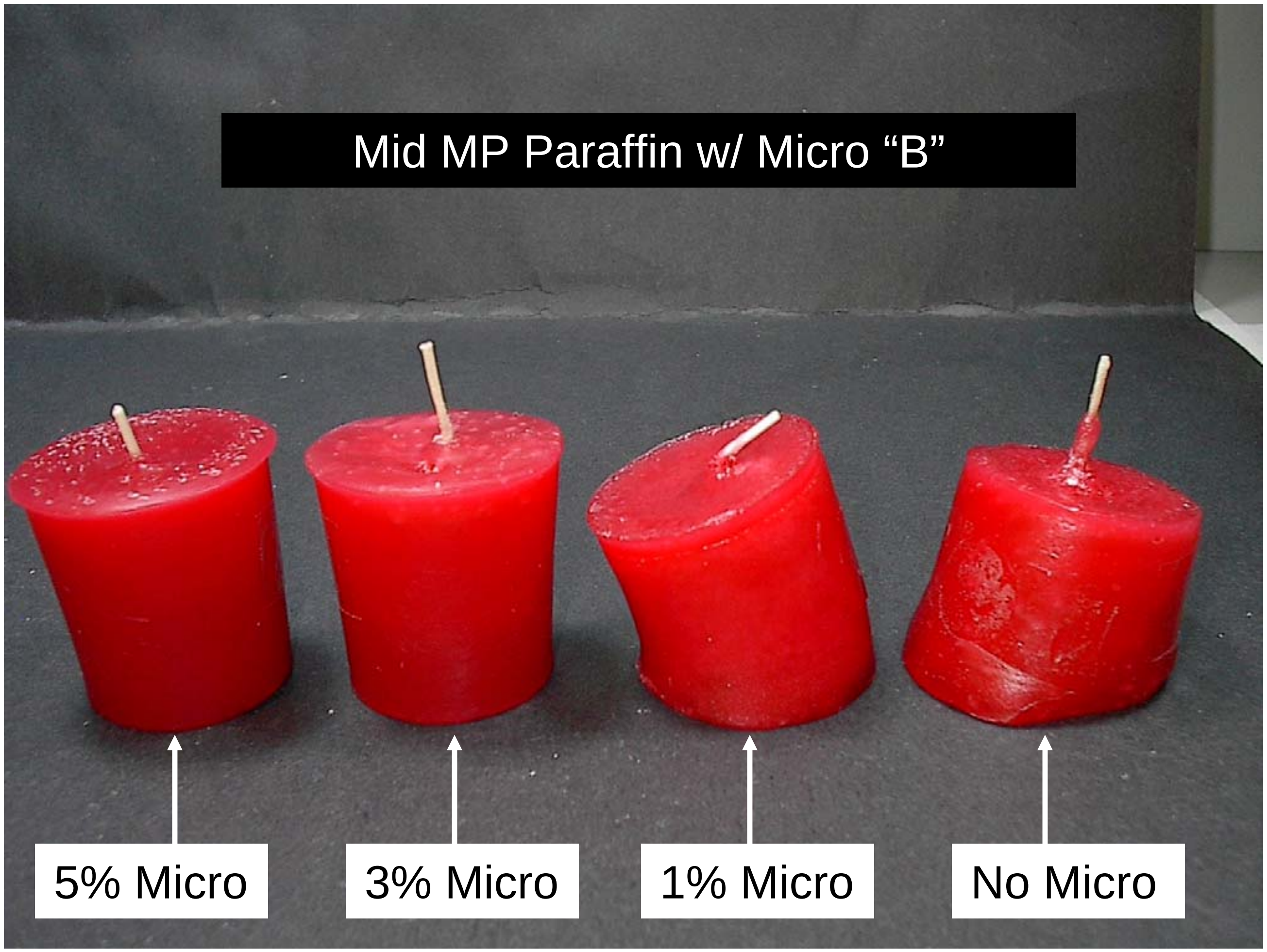
Examining Structural Properties

- **Purpose:**
  - to demonstrate how the addition of microcrystalline wax can benefit the structural integrity of molded candles
- Microcrystalline wax, by introducing branching to the carbon chain, increases bond strength and thus strengthens the overall structure in poured molded candles
- The improved strength allows for improved resistance to heat and shipments in hot conditions

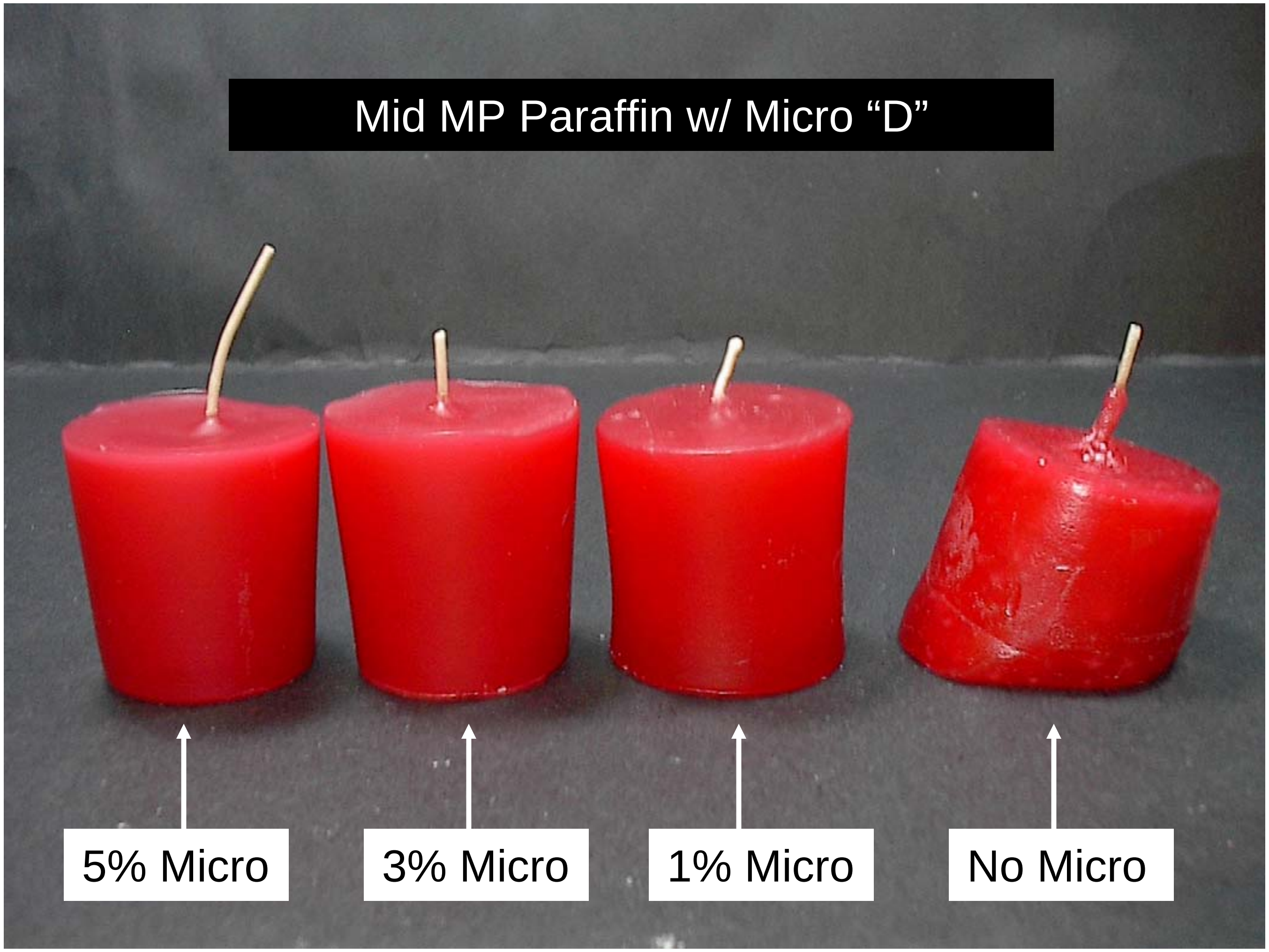
Evaluation Procedure

- Candles utilized:
  - Molded votive candles with 130°F mp paraffin wax
  - Molded votive candles modified with 1, 3, and 5% of micro wax "B" (laminating grade)
  - Molded votive candle modified with 1, 3, and 5% of micro wax "D" (hardening grade)
- Candles placed in an oven at 40°C (104°F) for a 24 hr period
- Resulting votive candle visually evaluated for structural defects

Visual Results-Votive Candles



Visual Results-Votive Candles



Use of Microcrystalline Wax for Fragrance Retention

- Purpose:
  - To demonstrate how microcrystalline wax can inhibit fragrance bleeding

- Introduction of isomers to the candle system results in a smaller crystal structure and thus improved fragrance retention

Evaluation Procedure

- Two systems evaluated
  - Container Candle-low mp paraffin wax vs. low mp paraffin wax blended with micro (A, B, and C)
  - Votive Candle-mid melt point paraffin wax vs. mid melt point paraffin wax blended w/ micro (A, B, and C)
- Each system purposely loaded with a high (10%) fragrance oil
- Container Candles examined visually only for fragrance bleed
- Votive candles weighed before and after fragrance bleed to determine % loss

Container Candles-Base Wax

Container Candle w/ Addition of Micro A

Container Candle w/ Addition of Micro B

Container Candle w/ Addition of Micro C

Fragrance Bleed in Votive Candles

- Mid melt point paraffin combined with 10% fragrance
- Votives made and set for 24 hours. Votives then weighed, de-molded, and cleaned of excess fragrance oil
- Votives re-weighed to determine % fragrance loss
  - Average fragrance loss=20% of added fragrance
  - Micros B, C, and D added at 1, 3, and 5% all stopped the resulting fragrance bleed

Compression Candle Testing

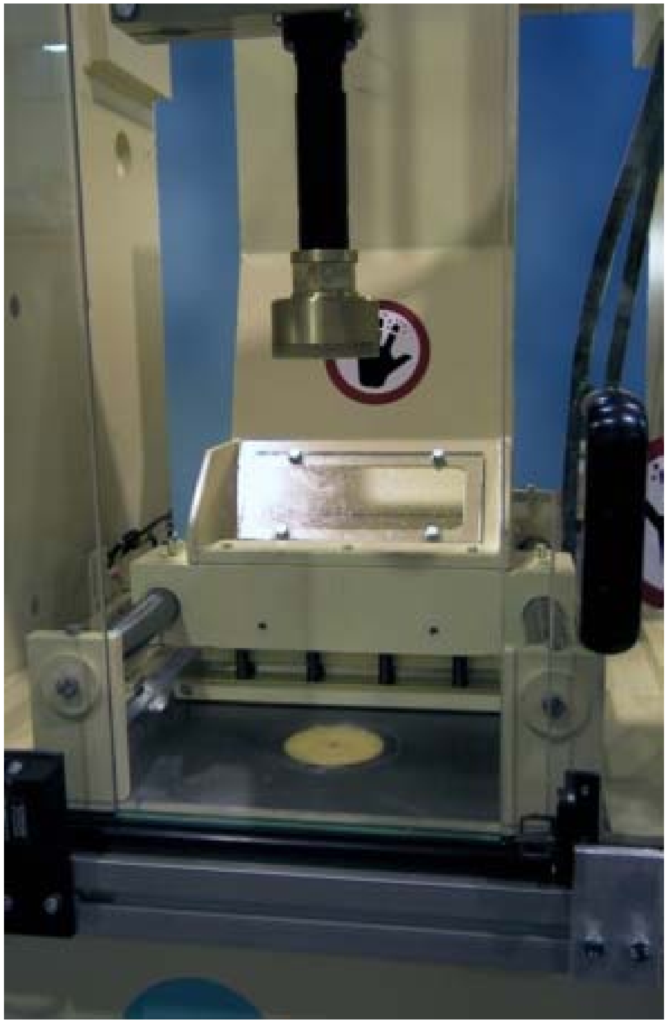
- Purpose: to determine the effect micro has on compression candle structure
- Two subsets examined:
  - Unscented compressed votives
  - Scented compressed votives
- Desired Properties from adding micro wax:
  - Reduce migration of residual oil and/or fragrance oil
  - Reduce cold flow properties
  - Strengthens the candle structure
- Waxes used
  - Micro waxes “B” (lam. 170 CP) and “C” (lam. 175 CP)
  - 130°F FRP and 135°F FRP

Equipment and Testing Setup

- Testing performed by SMS Marketing, Inc (Durham, NC)
- Equipment Used
  - Pilot compression machine
  - Imanda, Model PS (resolution +/- 0.2%) compression gauge
- Test Formulations
  - Micro wax at 1% and 2% by weight
  - Fragrance oil at 3% and 5% by weight
  - Votive candles manufactured with pilot compression machine
- Repeatability
  - Each test progression run 12 times

Picture of Pilot Compression Machine-Granulator Drum

Pilot Compression Unit



Compressed Candles

Imanda, Model PS Procedure

- Gauge applied to candle wick hole using bottom probe
- Force applied until candle cracks and breaks
- Gauge “locks on” highest force measurement, in lbs

Compression Testing

Testing Progression-Votives

TEST #	% PAR, type	% MICRO, type	COLOR	% FRG Oil
1	100% 130 mp	0%	No	0%
2	99% 130 mp	1% B	No	0%
3	98% 130 mp	2% B	No	0%
4	96% 130 mp	1% B	Yes	3%
5	93% 130 mp	2% B	Yes	5%
6	100% 130 mp	0%	No	0%
7	99% 130 mp	1% C	No	0%
8	98% 130 mp	2% C	No	0%
9	96% 130 mp	1% C	Yes	3%
10	93% 130 mp	2% C	Yes	5%

Testing Progression-Votives w/ Mid-MP Wax

TEST #	% PAR, type	% MICRO, type	COLOR	% FRG Oil
1	100% 135 mp	0%	No	0%
2	99% 135 mp	1% B	No	0%
3	98% 135 mp	2% B	No	0%
4	96% 135 mp	1% B	Yes	3%
5	93% 135 mp	2% B	Yes	5%
6	100% 135 mp	0%	No	0%
7	99% 135 mp	1% C	No	0%
8	98% 135 mp	2% C	No	0%
9	96% 135 mp	1% C	Yes	3%
10	93% 135 mp	2% C	Yes	5%

Results w/ Low Melt Point Wax

Candle Blend	Burn Rate (g/hr)	Force (lbs)
130 mp par + Color	3.60	25.9
130 mp par + Color + 1% Micro B	3.58	**26.7**
130 mp par + Color + 2% Micro B	3.20	**30.6**
130 mp par + Color + 1% Micro B + 3% Frag.	3.59	22.9
130 mp par + Color + 2% Micro B + 5% Frag.	3.72	22.5
130 mp par + Color + 1% Micro C	2.93	**21.2**
130 mp par + Color + 2% Micro C	2.80	**24.1**
130 mp par + Color + 1% Micro C + 3% Frag.	3.04	18.5
130 mp par + Color + 2% Micro C + 5% Frag.	3.07	18.1

Results with Mid Melt Point Wax

Candle Blend	Burn Rate (g/hr)	Force (lbs)
135 mp par + Color	3.90	24.9
135 mp par + Color + 1% Micro B	3.80	**29.1**
135 mp par + Color + 2% Micro B	2.78	**31.1**
135 mp par + Color + 1% Micro B + 3% Frag.	4.01	24.7
135 mp par + Color + 2% Micro B + 5% Frag.	4.53	28.0
135 mp par + Color + 1% Micro C	2.89	**28.0**
135 mp par + Color + 2% Micro C	2.82	**30.6**
135 mp par + Color + 1% Micro C + 3% Frag.	3.06	23.5
135 mp par + Color + 2% Micro C + 5% Frag.	3.13	24.0

Recommendations

- Must be balanced with major candle components and additives
- Micro wax for modifying opacity
  - Varying micro % when balancing the candle formula will alter color values slightly
  - Use of 3-5% will improve fragrance retention and opacity
- Micro wax for molded candle applications
  - 1% hardening grade to improve structural/heat integrity
  - 1% to improve fragrance retention, more if used also for heat resistance
- Micro wax for compression applications
  - Compression applications: 1-2% will improve candle strength as well as fragrance retention
  - Wax used must balance needle pen value which is hard enough to improve strength but not too hard to adversely affect compressibility
- Micro wax for Jar Candles
  - 5% laminating grade will improve adherence and allow improved fragrance retention

Selecting Microcrystalline Wax vs. Other Candle Additives

Compare To	Cost / lb	Processing Ease	Molecular Weight	% Required	Compat. w / paraffin
Veg Wax	↓	↓	-	-	↓
High MP Fischer Tropsch	↑	↓	↑	↓	↓
Poly Alpha Olefin	↑	↓	↑	↓	↓
Modified Polyethylene	↑	↓	↑	↓	↓

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