



**UvA-DARE (Digital Academic Repository)**

**Triacylglycerol structures and the chocolate fat bloom mechanism**

van Mechelen, J.B.

[Link to publication](#)

*Citation for published version (APA):*

van Mechelen, J. B. (2008). Triacylglycerol structures and the chocolate fat bloom mechanism

**General rights**

It is not permitted to download or to forward/distribute the text or part of it without the consent of the author(s) and/or copyright holder(s), other than for strictly personal, individual use, unless the work is under an open content license (like Creative Commons).

**Disclaimer/Complaints regulations**

If you believe that digital publication of certain material infringes any of your rights or (privacy) interests, please let the Library know, stating your reasons. In case of a legitimate complaint, the Library will make the material inaccessible and/or remove it from the website. Please Ask the Library: <http://uba.uva.nl/en/contact>, or a letter to: Library of the University of Amsterdam, Secretariat, Singel 425, 1012 WP Amsterdam, The Netherlands. You will be contacted as soon as possible.

# Contents

<b>Chapter 1</b>	<b>Introduction</b>	<b>1</b>
1.1	Fats	1
1.2	Crystals and diffraction principles	4
1.3	X-ray diffraction of TAGs	8
1.4	Structure determination using powder data	11
1.4.1	Sample preparation and data collection	11
1.4.2	Indexing and space group determination	13
1.4.3	Structure determination using direct space global optimization.	14
1.4.4	Structure refinement	15
1.5	Bibliography	18
1.6	References	18
<b>Chapter 2</b>	<b>Structures of mono-unsaturated triacylglycerols Part I.</b>	<b>21</b>
	<b>The <math>\beta_1</math> polymorph</b>	
2.1	Abstract	22
2.2	Introduction	22
2.3	Experimental methods	24
2.3.1	Samples, sample preparation and data collection	24
2.3.2	Indexing	25
2.3.3	Model building and analysis	28
2.4	Results and discussion	29
2.4.1	Data collection	29
2.4.2	Indexing, structure determination and refinement of the $\beta_1$ polymorphs	31
2.4.3	Influence of data resolution	34
2.4.4	The $\beta_1$ polymorph crystal-structure models	35
2.4.5	The relation between melting point and Methyl end-plane packing	39
2.5	Conclusions	40
2.6	Acknowledgements	41
2.7	References	41
2.8	Appendix Rietveld refinement results of $\beta_1$ structures	44

<b>Chapter 3</b>	<b>Structures of mono-unsaturated triacylglycerols Part II</b>	<b>49</b>
	<b>The <math>\beta_2</math> polymorph</b>	
3.1	Abstract	50
3.2	Introduction	50
3.3	Experimental methods	51
	3.3.1 Samples, sample preparation and data collection	51
	3.3.2 Indexing, model building, structure determination and refinement	52
3.4	Results and discussion	54
	3.4.1 Data collection	54
	3.4.2 Anisotropic cell-parameter contraction	56
	3.4.3 The novel $\beta_2$ -POS and $\beta_2$ -SOA polymorphs	57
	3.4.4 Indexing, structure determination and refinement of the $\beta_2$ polymorphs	58
3.5	The novel $\beta_2$ polymorph crystal structure model	59
	3.5.1 Conformation of TAGs	59
	3.5.2 Built-up and stacking of 'three packs'	60
	3.5.3 Subcell	60
	3.5.4 Fingerprint area interpretation	61
	3.5.5 Methyl end-plane packing and melting points	62
	3.5.6 The $\beta_2$ to $\beta_1$ phase transition	63
3.6	Conclusions	65
3.7	Acknowledgements	66
3.8	References	66
3.9	Appendix Rietveld refinement results of $\beta_2$ structures	68
<b>Chapter 4</b>	<b>Structures of mono-unsaturated triacylglycerols Part III</b>	<b>71</b>
	<b>The <math>\beta</math>-2 polymorphs of <i>trans</i>-mono-unsaturated triacylglycerols, and related fully saturated triacylglycerols</b>	
4.1	Abstract	72
4.2	Introduction	72
4.3	Experimental	73
	4.3.1 Samples, sample preparation and data collection	73
	4.3.2 Indexing, model building, structure determination and refinement	74
4.4	Results and discussion	78
	4.4.1 Packing of the $\beta$ -2 TAGs	78
	4.4.2 The methyl end-plane	82
	4.4.3 The packing of LMM and PSS	86
	4.4.4 The problem of local minima	87
4.5	Conclusions	89

4.6	Acknowledgements	90
4.7	References	90
4.8	Appendix Rietveld refinement results of $\beta$ -2 structures	92
<b>Chapter 5</b>	<b>Structures of mono-unsaturated triacylglycerols Part IV</b>	<b>99</b>
	<b>The highest melting <math>\beta'</math>-2 polymorphs of <i>trans</i>-mono-unsaturated triacylglycerols and related TAGs and their polymorphic stability</b>	
5.1	Abstract	100
5.2	Introduction	100
5.3	Experimental	102
	5.3.1 Samples, sample preparation and data collection	102
	5.3.2 Indexing, model building, structure determination and refinement	103
5.4	Results and discussion	107
	5.4.1 The structure determination process	107
	5.4.1.1 $\beta'_{1-2}$ PEP, $\beta'_{1-2}$ PSP, $\beta'_{1-2}$ PPE and $\beta'_{1-2}$ PPS	107
	5.4.1.2 $\beta'_{0-2}$ PSS	108
	5.4.2 The role of temperature in the interpretation of XRPD patterns	109
	5.4.3 Phase transitions and stability of polymorphs	111
	5.4.3.1 The $\alpha \rightarrow \beta'_2$ Phase transition	111
	5.4.3.2 The $\beta'_2 \rightarrow \beta'_1$ phase transition	112
	5.4.3.3 Stability of $\beta'_{1-2}$	112
	5.4.3.4 $\beta'_{0-2}$ PSS	114
	5.4.4 Packing and methyl end-plane	115
	5.4.4.1 $\beta'_{1-2}$ PEP, $\beta'_{1-2}$ PSP, $\beta'_{1-2}$ PPE and $\beta'_{1-2}$ PPS	115
	5.4.4.2 $\beta'_{0-2}$ PSS	118
	5.4.5 Comparison of $\beta'$ structures	119
	5.4.6 Comparison of $\beta'$ and the $\beta$ structures	121
5.5	Conclusions	121
5.6	Acknowledgements	123
5.7	References	123
5.8	Appendix Rietveld refinement results of $\beta'$ -2 structures	125
	Summary	129
	Samenvatting	131
	List of publications	133
	Nawoord	135