New Evidence for β′-2 p.p+2.p Triacylglycerol Crystal Structure

Sir:

In a recent paper (1) we proposed a crystal structure for the homologously isomorphous β′-2 p.p+2.p triacylglycerol series. Another recent paper (2), by different authors, contains three X-ray powder diffraction (XRPD) patterns that confirm the correctness of our proposed structure. Unfortunately, only one of these XRPD patterns was available to us when submitting our paper (1). Figure 1 shows the published experimental XRPD patterns. Figure 2 shows the XRPD patterns calculated from our proposed structure. The alternating intensities of the first three experimental patterns in region A (31*l* reflections with *l* = 4, 6, 8, or 10) and peak B (the 600 reflection) are very well reproduced by our structure.

In our opinion, the fourth experimental pattern does not correspond to a β′ polymorph. Peak C at *d* = 4.57 Å, i.e., at 2θ = 19.4° (see Table 1 in Ref. 2) indicates that this is a β polymorph, which has been described for this compound by DeJong (3), among others. This explains why the XRPD pattern calculated from our β′ structure does not match the experimental pattern.

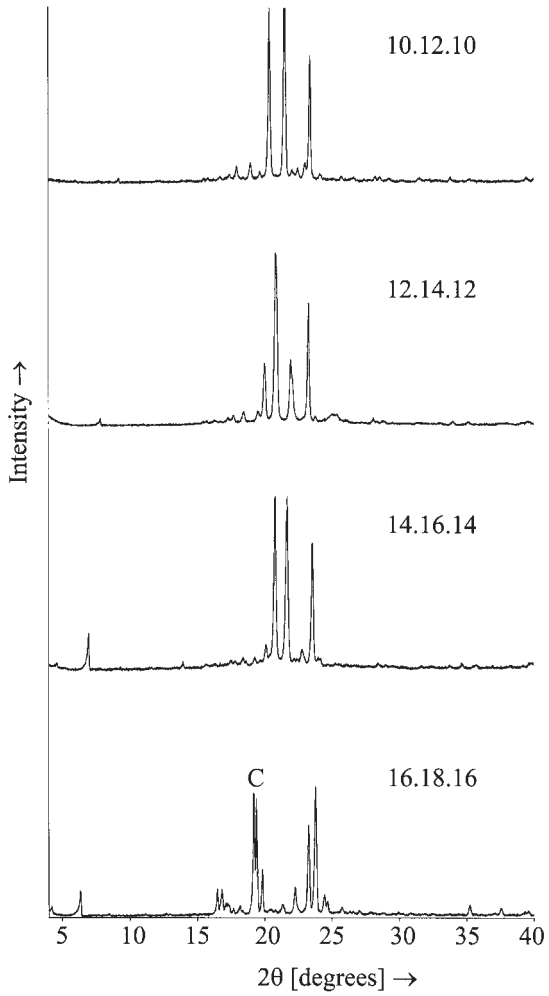

FIG. 1. Experimental X-ray powder diffraction (XRPD) patterns for 10.12.10, 12.14.12, 14.16.14, and 16.18.16, from Reference 2.

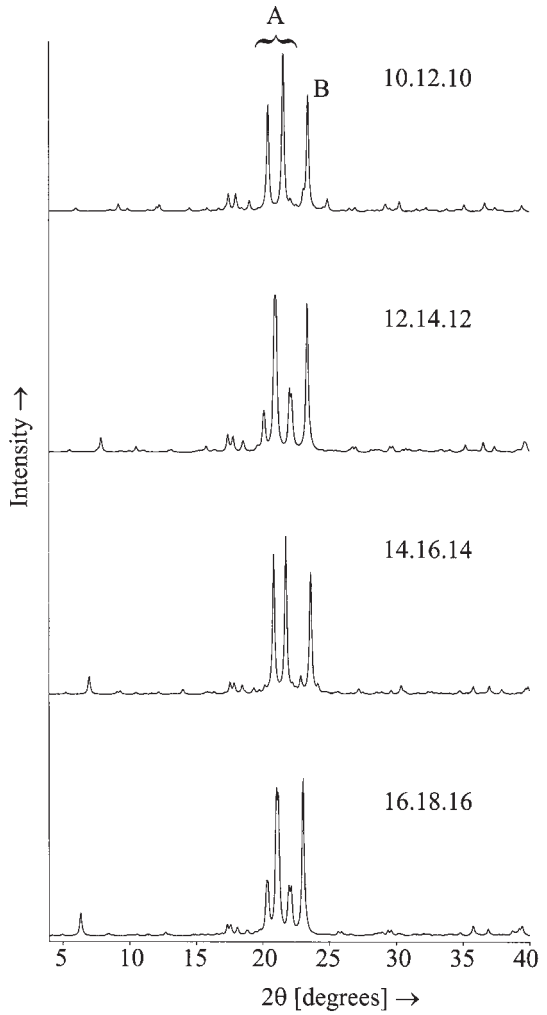

FIG. 2. XRPD patterns for 10.12.10, 12.14.12, 14.16.14, and 16.18.16, calculated from our proposed crystal structure. See Figure 1 for abbreviation.

The packing concept proposed by the authors in Reference 2 involves acyl chains parallel to the **c**-axis. We think that it is improbable that the increase in the length of the c-axis going from 10.12.10 to 14.16.14 (75.93 Å – 57.67 Å = 18.26 Å) is sufficient to accommodate the additional 4 × 2 = 8 $-C_2H_4-$ groups along this axis. The length of each $-C_2H_4-$ unit being 2.54 Å, the increase in that case should be 20.32 Å. The "missing" 2.06 Å is, however, the consequence of a 64° tilt of the acyl chains with respect to the **ab**-plane. Since the **c**-axis is then lengthened only by the projection of the $-C_2H_4-$ unit onto this axis, the increase should then be $\sin(64°) \cdot 20.32$ Å = 18.26 Å, as observed.

REFERENCES

1. Van de Steek, J., P. Verwer, R. de Gelder, and F. Hollander, Structural Analogy Between β′ Triacylglycerols and *n*-Alkanes: Towards the Crystal Structure of β′-2 p.p+2.p Triacylglycerols, *J. Am. Oil Chem. Soc. 76*:1333–1341 (1999).
2. Van Langevelde, A., K. van Malssen, E. Sonneveld, R. Peschar, and H. Schenk, Crystal Packing of a Homologous Series β′-Stable Triacylglycerols, *Ibid. 76*:603–609 (1999).
3. De Jong, S., and T.C. van Soest, Crystal Structures and Melting Points of Saturated Triglycerides in the β-2 Phase, *Acta Crystallogr. B34*:1570–1583 (1978).

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