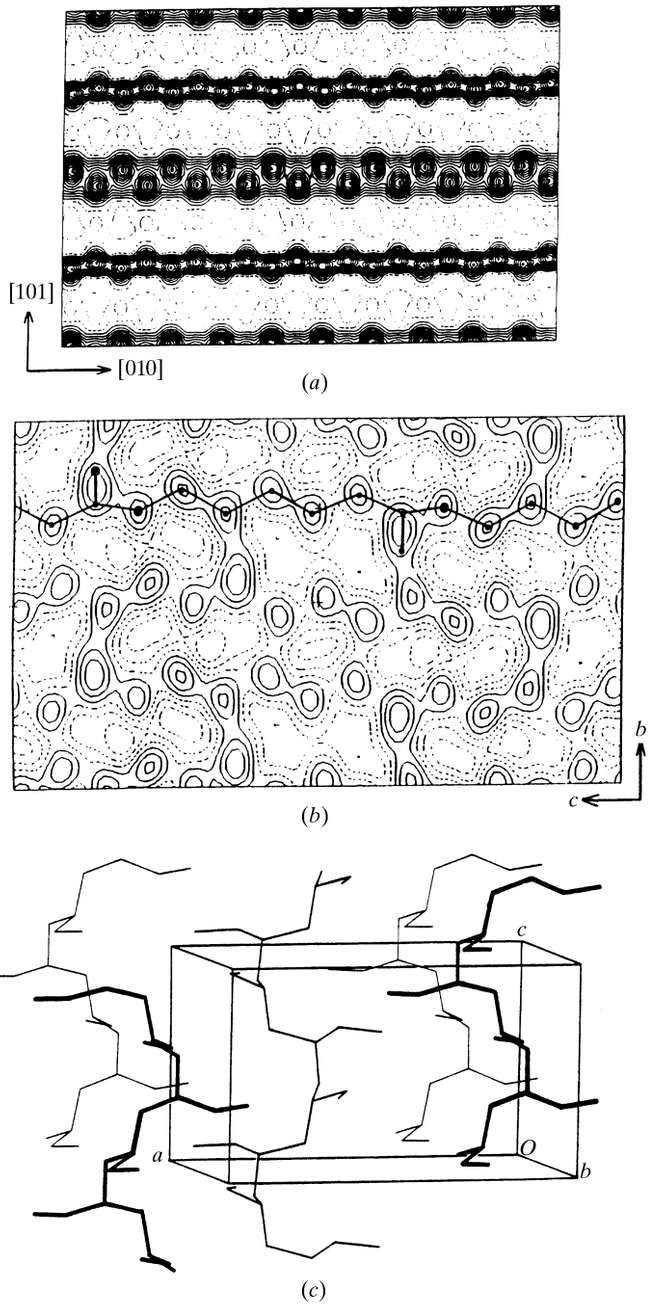

Fig. 4.5.3.2. Crystal structures of linear polymers determined from three-dimensional data. (*a*) Polyethylene; (*b*) poly(ε-caprolactone); (*c*) poly(1-butene), form (III).

orthogonal projections of the same polymer polymorph can be obtained, respectively, by self-seeding and epitaxic orientation. While tilting these specimens, all of reciprocal space can be sampled for intensity data collection.

Polyethylene crystals were used to collect 50 unique maxima (Hu & Dorset, 1989) and, *via* symbolic addition, the centrosymmetric phases of 40 reflections (space group *Pnma*) could be readily determined (Dorset, 1991*b*). The structural features were readily observed in the three-dimensional potential maps (Fig. 4.5.3.2*a*), and atomic coordinates (with estimated values for hydrogen-atom positions) could be refined by least squares (Dorset, 1995*b*) to give a final R value of 0.19.

Poly(ε-caprolactone) was epitaxically crystallized on benzoic acid and, with *hk*0 data from solution-crystallized samples, a unique set of 47 intensities was collected for the noncentrosymmetric orthorhombic unit cell (space group $P2_12_12_1$) (Hu & Dorset, 1990). Direct phase determination was achieved *via* symbolic addition, using one algebraic unknown to assign values to 30 reflections (Dorset, 1991*c*). Atomic positions along the chain repeat, including the carbonyl position, were clearly discerned in the [100] projection (Fig. 4.5.3.2*b*) and the three-dimensional model was constructed to fit to the map calculated from all phased data, yielding a final crystallographic residual R = 0.21. This independent determination was able to distinguish between two rival fibre X-ray structures, in favour of the one that predicted a nonplanar chain conformation. Because of the methylene repeat, this is actually a difficult structure to solve by automated techniques. For example, the tangent formula and *SnB* (Miller *et al.*, 1993) could only find chain zigzag positions and not the position of the carbonyl oxygen atom (Dorset, 1995*b*).

The most complicated complete polymer crystal structure solved so far by direct methods using electron diffraction data (Dorset *et al.*, 1994) was based on 125 unique data (space group $P2_12_12_1$) from isotactic poly(1-butene), form (III), using orthogonal molecular orientations crystallized in Strasbourg (Kopp *et al.*, 1994). Initially, the standard NQEST figure of merit (FOM) (De Titta *et al.*, 1975) was not suitable for identifying the correct solution among the multiple sets generated with the tangent formula. A solution could only be found when a separate phase determination was carried out with the *hk*0 data to compare with the multiple solutions generated. More recently, the minimal principle (Hauptman, 1993), used as a FOM with the tangent formula or with a multiple random structure generator, *SnB*, correctly identified the structure on the first try (Dorset, 1995*b*). The maps clearly show individual carbon-atom positions in a 4_1 helix that parallels 2_1 helices of the space group (Fig. 4.5.3.2*c*). After Fourier refinement, the crystallographic residual was R = 0.26. The previous powder X-ray diffraction determination was based on only 21 diffraction maxima, some of which had as many as 15 individual contributors.

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