chemoreceptors can lead to integration in routine analytical procedures. In addition, an application as a multianalytical sensor with a flow injection system is planned. When associated with highly diverse cyclopeptide libraries[12] that may also be immobilized electrochemically, the method offers particular possibilities in chemosensoring for rapid analysis by pattern recognition. The spatially resolved adhesion of nerve cells and the growth of axons are possible with peptides derived from laminin that may also be polymerized electrochemically with retention of their biological function.[13]

Experimental Section

2, 3: Multiple parallel peptide syntheses (each 15 μmol) on Wang resin with 9-fluorenylmethoxycarbonyl (Fmoc) amino acids and activation with *N,N'*-diisopropylcarbodiimide (DIC). Product **1**[11] (8.6 mg, 0,03 mmol) and 1-hydroxybenzotriazole (HOBT; 4.6 mg, 0.03 mmol) in DMF (200 μL) were coupled with 1.5 M DIC in DMF (20 μL, 0.03 mmol) for 4 h.

Cleavage from the resin: trifluoroacetic acid/water/triisopropylsilane (95/2.5/2.5; 500 μL; 3 h), Nbz–HPA–peptide precipitated with diethyl ether, centrifuged, dissolved in *tert*-butyl alcohol/water (4/1), and lyophilized. **2**: ESI-MS: $m/z = 1467.7$ $[M+2H]^{2+}$; **3**: ESI-MS: $m/z = 1523.6$ $[M^{+}+H]$.

The photochemical cleavage of **3** was investigated by UV spectroscopy and HPLC. At the start of the reaction an absorption maximum for the peptide is present at 214 nm, and for the protecting group at 275 nm. During photolysis a further maximum for the cleaved protecting group appears at 315 nm. Within 1 min 70 % of protected peptide **3** is cleaved, and after 5 min complete conversion into the free phenolic end group had occurred. These results indicate a stoichiometric formation of **4** (Scheme 1). A homogeneous product was also obtained with **2**; that is, the amino acids do not participate in detectable side reactions. However, Trp, Tyr, and His residues are partially oxidized during electropolymerization, but this does not have consequences for the practical use of peptide-functionalized surfaces, for example, for ELISA. To ensure that the Nbz–HPA–peptides **2** and **3** and the by-products released during photolysis cannot be oxidized, peptides **2**–**4** were measured by differential pulse voltammetry. This showed that electrochemical oxidation occurred with the photochemically deprotected HPA–peptide **4** at 0.6 V, whereas **2** and **3** were electrochemically inert at this potential.

After electrochemical polymerization by photolytic cleavage of the protecting group of the Nbz–HPA–peptide (1.0 V, 5 min), the GCE is rinsed with water. To block nonspecific binding sites 1 % BSA/PBS (BSA = bovine serum albumine) is applied (500 μL per electrode). After 2 h a solution of streptavidin (rhodamine- or fluorescein-labeled) in 0.1 mg mL^{-1} PBS is applied. After 2 h incubation the electrode is washed. The antibody-coated electrode is treated with a secondary antibody (rhodamine-labeled) in 0.1 mg mL^{-1} PBS and washed after 2 h.

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Solution of the Crystal and Molecular Structure of Complex Low-Symmetry Organic Compounds with Powder Diffraction Techniques: Fluorescein Diacetate**

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Data from modern X-ray and neutron diffractometers and improvements in computational techniques have allowed increasingly complex crystal structures to be solved from powders. The use of synchrotron radiation has had a marked impact, and provides very narrow and accurately positioned diffraction peaks. The largest structures solved are of inorganic compounds, and sequential radiation with X-rays and neutrons was exploited to locate heavier and then lighter atoms. Example include $Ga_2(HPO_3)_3 \cdot 4H_2O$[1] and $La_3Ti_5Al_{15}O_{37}$[2] with 29 and 60 atoms, respectively, in the asymmetric unit. For organic compounds, the structures solved to date are simpler; for example, the structure of chlorothiazide[3] with 17 atoms was determined by direct methods. Various algorithms have also been developed to locate the positions of known molecular fragments within the unit cell.[4–9] Here we report on the structure solution of fluorescein diacetate ($C_{24}H_{16}O_7$). All 31 carbon and oxygen atoms were accurately located, without employing any prior knowledge of the molecular

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structure, using direct methods and Fourier recycling. This represents a considerable advance in the size of an organic molecule solved ab initio from powder diffraction data. The data were collected at a third-generation synchrotron radiation source, and we believe that quite large organic structures can be solved routinely from powders using such facilities.

The determination of the crystal structure of any material or compound can usually be achieved if a single crystal, even a very small one, can be grown. Many compounds do not form single crystals, however, and exist only in the form of microcrystalline powders. Powder diffraction techniques must then be used to obtain information about the structure. However, owing to the projection of the data onto a single angular scale of 2θ, coincidence or overlap of peaks will occur, making determination of the individual integrated intensities impossible or much more difficult. When overlap becomes severe, even the 2θ positions of the individual peaks may be hard to measure with certainty. These problems make the solution of crystal structures from powders much less certain than from single crystals.

The solution of structures from powders requires data of the highest quality. Modern diffractometers, and especially those exploiting synchrotron radiation, can yield diffraction patterns from which structures can be solved without any prior knowledge. The keys to success are high angular resolution and hence narrow peak widths, accurate determination of peak positions, and good counting statistics throughout the diffraction pattern. Improved resolution reduces the overlap between adjacent reflections, so that individual peak intensities can be extracted with the highest level of reliability. With narrow peak widths, small peak splittings may also be resolved, perhaps revealing a lower symmetry than would be apparent with data measured at lower angular resolution. Accurate peak positions are required for indexing the unit cell, that is, recovering the three-dimensional crystal lattice from the one-dimensional powder pattern. Good counting statistics are essential for accurately fitting diffraction peaks during the extraction of the integrated intensities, which occurs prior to structure determination by traditional single-crystal techniques such as direct methods or Patterson synthesis. Synchrotron radiation offers all these attributes owing to the abundant photon flux, the low beam divergence, and the small source size. The combination of the low beam divergence and high flux also makes it feasible to incorporate an analyzer crystal in the diffracted beam in order to further enhance the angular resolution of the powder diffractometer. A sample of good crystallinity, free from strain and particle-size broadening, is also advantageous to exploit the capabilities of synchrotron radiation fully.

An early example of the power of high-resolution powder diffraction for structure solution was provided by McCusker[10] in 1988, when the structure of a clathrasil (Sigma-2, 17 non-hydrogen atoms in the asymmetric unit) was solved using data collected at NSLS in Brookhaven. This publication also gives a general overview of the procedures involved in structure solution. More recently, synchrotron radiation has been increasingly employed for powder diffraction studies. Most of the studies have been carried out on inorganic compounds, and there are fewer examples of organic structures solved from powder data. One reason for this scarcity, apart from the increased molecular complexity, is the poorer scattering at high angle due to the fall-off in the form factor of the light atoms which make up the organic structure. In addition, larger thermal displacement parameters in organic molecules lead to a stronger exponential decay of the scattered intensities with $\sin\theta/\lambda$.

Here we report the complete solution of the crystal and molecular structure of fluorescein diacetate ($C_{24}H_{16}O_7$), a molecule with 31 carbon and oxygen atoms, without any prior structural knowledge. Some organic structures of comparable complexity have been solved before from synchrotron radiation data, though these have required the input of the known molecular structure, or at least part of it. For example, for the red form of fluorescein, a molecule closely related to fluorescein diacetate, the complete molecule was moved around the unit cell using a Monte Carlo approach that seeks to minimize the differences between observed and calculated diffraction patterns.[9] Genetic algorithms,[4] simulated annealing,[5] or a grid search[6–8] (for structures with few degrees of freedom) are alternative approaches to the solution of crystal structures; in this case the position, orientation, and conformation of known molecular fragments, or whole molecules, are adjusted.

Fluorescein diacetate powder (Sigma, 98% purity) was loaded into a thin-walled borosilicate glass capillary with a diameter of 1.5 mm. A powder diffraction pattern was measured at room temperature with a wavelength of 1.0 Å at the Swiss–Norwegian beam line (BM1) at the European Synchrotron Radiation Facility (ESRF) in Grenoble. This diffractometer is equipped with a Si 111 analyzer stage, yielding intrinsic peak widths of about 0.012°. The diffraction pattern could be indexed with a triclinic lattice with the program ITO[11] from the position of 20 low-angle reflections with a figure of merit M_{20} of 118. The volume of the unit cell indicates that there are two molecules per cell, and hence a possible space group is $P\bar{1}$. However the structure could not be solved, possibly because of the low intensities of the reflections at high angle, a region of vital importance if direct methods are to be successful. A new diffraction pattern was measured on the powder diffraction beam line BM16 at the ESRF at a wavelength of 0.6006 Å while the sample was cooled to 100 K with a stream of cold nitrogen gas. The high-angle part of the diffraction pattern ($2\theta = 20-50°$) was scanned twice as often as the low-angle part to improve the statistical quality of this region. The beam line BM16 is equipped with nine detectors that are spaced at intervals of approximately 2° behind a Ge 111 analyzer crystal. In effect, nine diffraction patterns offset from one another by 2° are collected simultaneously. Following data collection, the counts from the nine channels are combined and normalized, taking into account the exact angular separation between channels, their different efficiencies, and the fall-off in the incident intensity as the electron current in the storage ring gradually decays with time. This provides an efficient method for measuring a high-resolution powder diffraction pattern.

A total of 2905 individual values of F^2_{hkl} were extracted from the normalized data with the program EXTRA;[12] this is equivalent to 511 statistically independent observations.[13]

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The program EXTRA performs a fit to the observed diffraction profile, using as input the unit-cell parameters to calculate peak positions, but without any structural model. A pseudo-Voigt function was used to fit the profiles. The low-temperature data confirmed the presence of a $P\bar{1}$ cell. In EXTRA, intensities are obtained following the method initially described by Le Bail.[14] The extracted intensities, together with refined lattice parameters, wavelength, space group, and information on the cell contents were input to the associated direct-methods program SIRPOW.[15] The distribution of normalized E values was compatible with the assumption of a centrosymmetric structure. In the phasing section, 32 permutations were performed to produce 10 possible sets, which were ranked according to their combined figure of merit. These were then used for the Fourier transform and generation of the E map. With the default settings, the structure was not successfully solved with SIRPOW. However, after the parameter that controls whether SIRPOW considers neighboring reflections as overlapped was changed from the default value of 0.1 to 0.2, the E map revealed a fragment of the molecule comprising 14 atoms (part of the xanthenone group). The combined figure of merit at this stage was 0.988. In subsequent cycles of Fourier synthesis and least-squares refinement, the rest of the structure was located with SIRPOW. The complete molecule was found, with carbon and oxygen atoms correctly assigned, and all bond distances were within about 0.1 Å of what would be expected.

The structure was refined by the Rietveld method, using the PC version of the program GSAS.[16] This program incorporates the correction described by Finger et al.[17] for peak asymmetry caused by axial divergence. This tends to be quite pronounced with BM16 data because of the narrow peak widths and because the short wavelength used pushes more peaks to lower diffraction angles, where the asymmetry is most marked. First the background, overall scale factor, zero point, and lattice parameters were refined, and then the profile parameters. The atomic positions were refined with the bond lengths restrained to the values found in a single-crystal diffraction study of a related system.[18] Hydrogen atoms were included in the model at their expected positions making use of the XP utility in the SHELXTL program.[19] At the end of the refinement, assuming an overall isotropic temperature factor, the resulting R values were $R_p = 7.1\,\%$ and $R_{wp} = 9.7\,\%$ ($\chi^2 = 4.1$). One molecule of fluorescein diacetate placed in the unit cell is shown in Figure 1.[21] The final atomic coordinates are given in Table 1, and the fit between the observed and calculated powder diffraction patterns is shown in Figure 2. In contrast to the case of fluorescein[9] or the 1:1 complex of acetone with the lactoid form of fluorescein,[18] where a network of intermolecular C=O···H–O hydrogen bonds is present, fluorescein diacetate forms weaker C–H···O bonds

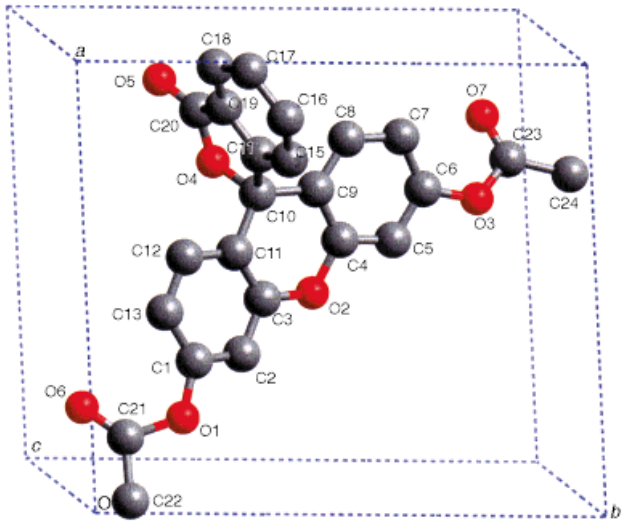

Figure 1. One molecule of fluorescein diacetate in the unit cell. Hydrogen atoms are not shown.

Table 1. Final atomic coordinates for fluorescein diacetate at 100 K after refinment in the triclinic spacegroup $P\bar{1}$ by the Rietveld method. The atom numbering scheme is shown in Figure 1. $U_{iso} = 0.013$ Å^2; $a = 11.044$, $b = 11.730$, $c = 7.371$ Å, $\alpha = 95.759$, $\beta = 97.429$, $\gamma = 94.830^\circ$, $V = 938$ Å^3; $\rho_{calcd} = 1.48$ g cm^{-3}; $Z = 2$.

O(1)	0.4604(6)	0.4752(6)	0.2509(9)
O(2)	0.8007(5)	0.8828(5)	0.7038(8)
O(3)	0.2246(5)	0.1647(5)	– 0.1026(9)
O(4)	0.9035(5)	0.2219(5)	0.4728(8)
O(5)	0.6440(6)	0.8359(5)	0.4809(9)
O(6)	0.7331(6)	0.3090(5)	0.3879(9)
O(7)	0.2458(5)	– 0.0303(5)	– 0.0977(9)
C(1)	0.0548(6)	0.0382(11)	– 0.2296(17)
C(2)	0.7863(8)	0.5715(9)	0.3815(16)
C(3)	0.3445(7)	0.3089(8)	0.0794(15)
C(4)	0.6694(8)	0.7252(6)	0.4429(15)
C(5)	0.4566(9)	0.3648(6)	0.1638(14)
C(6)	0.6837(9)	0.4972(7)	0.3050(14)
C(7)	0.6837(12)	1.0387(6)	0.6179(19)
C(8)	0.8945(9)	0.3843(9)	– 0.1474(13)
C(9)	0.5612(8)	0.2014(8)	0.0495(16)
C(10)	0.3407(6)	0.2025(8)	– 0.0210(13)
C(11)	0.7164(8)	0.9170(6)	0.6117(12)
C(12)	0.5620(8)	0.3113(7)	0.1401(13)
C(13)	0.5637(7)	0.6538(8)	0.3775(17)
C(14)	0.8775(8)	0.3082(9)	0.1919(12)
C(15)	0.7850(7)	0.6864(9)	0.4428(15)
C(16)	0.9772(8)	0.2837(8)	0.1023(14)
C(17)	0.1844(6)	0.0471(5)	– 0.1347(15)
C(18)	0.5708(8)	0.5410(7)	0.3091(14)
C(19)	0.7823(9)	0.3639(9)	0.1125(12)
C(20)	0.4478(8)	0.1484(6)	– 0.0246(15)
C(21)	0.7931(9)	0.4006(8)	– 0.0574(12)
C(22)	0.8487(7)	0.2813(8)	0.3718(12)
C(23)	0.6824(7)	0.3730(7)	0.2316(12)
C(24)	0.9884(9)	0.3281(10)	– 0.0619(15)
H(1)	0.0331(62)	– 0.0448(15)	– 0.233(12)
H(2)	– 0.0018(54)	0.0600(62)	– 0.143(7)
H(3)	0.0147(68)	0.0075(68)	– 0.354(4)
H(4)	0.8723(23)	0.5644(61)	0.366(10)
H(5)	0.2718(32)	0.3386(48)	0.123(8)
H(6)	0.7209(64)	1.0999(50)	0.714(8)
H(7)	0.6998(63)	1.0927(56)	0.529(8)
H(8)	0.6064(33)	1.0478(64)	0.667(10)
H(9)	0.9032(61)	0.4282(52)	– 0.252(6)
H(10)	0.6397(26)	0.1774(50)	0.019(9)
H(11)	0.4837(24)	0.6815(47)	0.391(9)
H(12)	0.8684(27)	0.7162(56)	0.428(10)
H(13)	1.0646(19)	0.2983(55)	0.151(9)
H(14)	0.4607(63)	0.0683(20)	– 0.062(9)
H(15)	0.7802(58)	0.4773(21)	– 0.001(8)
H(16)	1.0689(32)	0.3221(60)	– 0.102(10)

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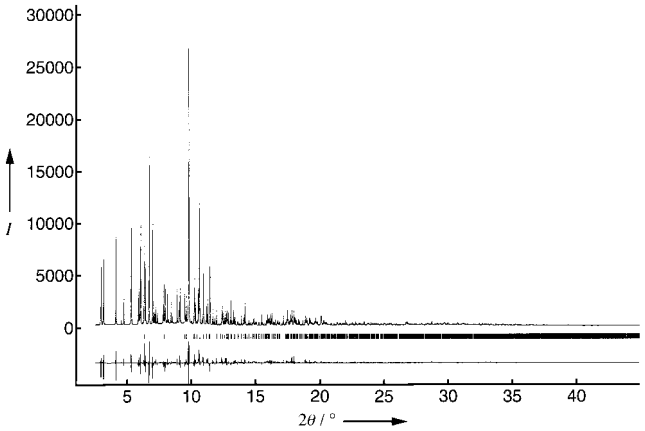

Figure 2. Results from the data collection on polycrystalline fluorescein diacetate at the beam line BM16 at the ESRF: experimental (dots) and calculated powder diffraction data (solid line, final result from the Rietveld refinement) together with the difference curve (bottom, observed minus computed intensities).

between the molecules. There is general consensus that C–H···O bonds influence crystal packing, especially when stronger hydrogen bonding is absent.[20] A summary of potential hydrogen bonds is given in Table 2 along with

Table 2. Analysis of the C–H···O hydrogen bonds in fluorescein diacetate.[a]

D–H···A	Distance D–A [Å]	Angle D-H-A [°]
C(22)–H(1)···O(7)	3.189	127.7
C(12)–H(10)···O(6)	3.053	152.3
C(7)–H(12)···O(5)	3.484	151.4
C(18)–H(13)···O(7)	3.510	179.7
C(15)–H(15)···O(2)	3.475	173.1

[a] D = donor, A = acceptor.

donor–H···acceptor distances and bond angles. The acetate groups on both sides of the xanthenone moiety play an important role in the formation of hydrogen bonds in this system. The three rings in the xanthenone group each have a high degree of planarity, and the end rings are inclined to each other at an angle of about 10°, which is in agreement with the results of the single-crystal study of the lactoid form of fluorescein.[18]

The collection of data combining high resolution, accurate peak positions, and good statistics by use of the powder diffractometers at the ESRF—the world's first third-generation synchrotron radiation source—has led to the solution of a complex organic structure without any prior structural knowledge. Only the chemical formula was used as input information in the procedure, as is usually the case for single-crystal studies. The collection of data at 100 K and additional scanning of the high-angle region to improve the statistics has without doubt improved the quality of the data, but apart from that no special procedures were followed. The structure was obtained from direct methods with only one parameter in SIRPOW being changed from its default value. It seems probable that by using more optimized methods of data collection, such as those advised by David and co-workers,[3] and by exploiting the many options available in program packages for carrying out direct methods, structures of significantly greater complexity may be obtainable solely from high-quality powder diffraction data. This is of clear importance for characterizing the large number of compounds that do not easily form single crystals, and leads us to believe that the goal of solving the structures of complex polymorphs of organic compounds, perhaps with specific pharmacological activity, is now attainable.

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