

Polymeric Photoconductors— New Concepts**

By Dietrich Haarer* and Alexander Blumen*

**Charge Carrier Transport
Amorphous Materials
Fractals**

1. Introduction

The use of polymeric photoconductors in printing and copying techniques represents one of the most sophisticated applications of polymeric materials. However, there is a considerable lack of understanding of the basic electronic mechanisms involved in photoconduction. Recently, new theoretical concepts and improved experimental techniques have given new impetus to the complex problem of dispersive transport in polymeric photoconductors. On the experimental side one has succeeded in monitoring photocurrents over ten decades in time and the decay over many orders of magnitude. On the theoretical side one has been able to model not only the temporal disorder via random walks in continuous time but also the geometric disorder via fractals and the energetic disorder via ultrametric spaces. An introduction to these new concepts will be given and some topical aspects will be discussed in this article.

2. Historical Overview

The rapid growth of the electronic industry, and especially the almost exponential increase of the performance of its products, have mostly been attributed to technical advances in the field of single crystalline silicon devices. Yet, it was not until the mid-seventies and early eighties, that it became obvious that in the silicon technologies as well as in other domains of the electronic industry, sophisticated or so-called "intelligent" polymers have silently made an almost comparable progress. For instance, in the field of passive dielectric components the "natural polymer" paper (which had been widely used as dielectric medium in capacitors) was replaced by thin polymer films. In the field of "active applications"—here the word active is used to characterize "gain" or "electronic" mechanisms—the achievements are enormous, much beyond the expectation level set by research activities on organic molecular

crystals, a typical field of research on organic materials in the sixties and seventies.

One of the best examples of this change is the development of photoconductive materials. This field originated with experiments on anthracene crystals where, for the first time, electron and hole mobilities were measured by the time-of-flight method.^[1,2] From a historical standpoint it is interesting to note that in early work of the Göttingen school on the photoconductivity of color centers in alkali metal halide crystals similar techniques were employed half a century ago, using the modest experimental equipment of the time.^[3] These investigations, however, were hampered by the lack of applications which was typical of most of the color center work in general. Even research on simple crystalline organic materials did not lead to any practical results, because nobody could think of ways to use anthracene or naphthalene crystals in technical devices, and because the quantum yields for charge carrier production were, at the normal electrical field strengths, on the order of 10^{-3} and therefore not attractive for applications.

It was several years after the discovery of the xerographic process based on the inorganic photoconductor selenium,^[4] that the first patent on organic photoconductive systems was published.^[5] Several key ingredients can, in hindsight, account for the success of polymeric photoconductors:

- a) Polymeric systems can be easily fabricated in large area devices and can be produced perfect enough to withstand high electric fields (low pinhole densities).
- b) The role of donor-acceptor interactions was discovered early as a key mechanism to enhance carrier production yields.

The first example of technical applications used the donor polymer polyvinylcarbazole (PVK) and the acceptor compound trinitrofluorenone (TNF) in low concentrations^[5] or in a 1:1 stoichiometry.^[6]

With the above-mentioned advantages of polymeric materials and with complex dye-matrix or dye-pigment systems replacing the donor-acceptor based polymers, the organic photoconductors were perfected between 1970 and 1980 to the extent that they are nowadays equivalent or superior to their inorganic counterparts. The yields for charge carrier production are on the order of ten percent,^[7] depending on the limiting field strengths, and it is only the

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mobility aspect of the presently used systems which still favors inorganic materials like the chalcogenides.^[8]

In spite of the large technical progress in photoconductive systems, many basic aspects of charge carrier generation and transport are still not well understood. In this article we focus on some recent experimental and theoretical aspects of charge carrier transport. Improved techniques to measure photocurrents over ten or more decades allow one to monitor the dispersive transport with previously unknown accuracy. Since the photocurrent can be determined over a wider time regime than most other properties (such as mechanical relaxation), photoconductors are a good model system for transport phenomena in disordered materials. New experimental data allow crucial tests for recent theories of non-Gaussian behavior.

From the theoretical viewpoint, the issue at stake in amorphous photoconductors is the dispersive transport, as contrasted to the familiar diffusive, Gaussian behavior. Diffusive motion, in its many facets, ranging from the transfer of heat (macroscopic) to Brownian motion (microscopic) is a well-understood phenomenon and may be modelled using the mathematical theory of Markov processes, in which the system keeps no memory of past events. Typical discrete examples are random walks on regular lattices—first introduced by *Bachelier* at the turn of the century for monitoring the fluctuations of the stock-market^[9]—which nowadays comprise a vast mathematical literature.^[10] Simple random walks on regular lattices of arbitrary dimensions lead to a time-independent diffusion coefficient, or, expressed differently, in the absence of a biasing field, to a linear increase in the mean squared displacement as a function of time.

In amorphous photoconductors the situation is different, since one encounters dispersive transport: the motion of the carriers through a sample becomes slower and slower with the passage of time, so that no diffusion constant in the usual sense can be defined. Here the diffusion coefficient may display an algebraic dependence on time:

$$D(t) \sim t^{\alpha-1} \quad \text{with } \alpha < 1 \quad (\text{a})$$

with a similar finding for the current $I(t)$ flowing in a biasing field. Such behavior is incompatible with the usual diffusion equation.

Progress in the advancement of theoretical models for dispersive transport was slow, due to the Gaussian character of most probability distribution functions put forward. Here we have to recall the overwhelming role of the “central limit theorem” of probability; under very general conditions, a sum of random variables will tend to form a normal (Gaussian) distribution when the number of elements in the sum increases. The position vector of a particle may be viewed as the sum of distance increments due to past events, say collisions. If energies, directions etc., are randomized after such events, most distributions will lead to regular diffusion. Hence, for a successful modeling of dis-

persive transport in such a framework one has to look for the (relatively rare) distributions for which the central limit theorem does *not* hold.

The basic theoretical problem which has to be solved for the understanding of the transport of light-generated charge carriers through an amorphous medium is to model the motion of individual carriers in a “hopping model”, in which these charge carriers perform a specific type of random walk in the direction of the external electric field. In this random walk the probability for performing the next hopping process does not decay exponentially with *one* given decay constant but shows a much broader distribution. This is due to the fact that an amorphous material has rather irregular lattice spacings (geometrical disorder), which lead to large variations in energy levels (energetic disorder).

Mathematically the most important parameter characterizing the transport is the waiting time distribution, i.e., the distribution of waiting times between subsequent carrier jumps. Here a theoretical breakthrough occurred with the realization by *Scher, Lax* and *Montroll* that long tails in the waiting-time distributions $\psi(t)$ between the microscopic events can qualitatively describe the phenomenon of dispersive transport.^[11,12] Such distributions lead to the so-called Lévy processes when

$$\psi(t) \sim t^{-1-\alpha} \quad \text{with } 0 < \alpha < 1 \quad (\text{b})$$

so that the zeroth but not the first moment of the distribution $\psi(t)$ exists. This field was long considered to be a domain of mathematical curiosities.^[13,14] The main technical point in investigating random walks in continuous time (CTRW) with such distributions was the realization that the basic CTRW framework developed ten years earlier by *Montroll* and *Weiss*^[15] could easily incorporate these, less usual, $\psi(t)$ forms. However, although the theory developed^[11,12] was successful in providing a scheme for a qualitative understanding of the dispersive transport, the nature of the microscopic parameters leading to $\psi(t)$ was at the time only conjectured.

Today we know that this is only one of the possible ways of obtaining algebraic time dependencies of $D(t)$, and that the key ingredient in the CTRW framework is the idea of temporal scaling, or of time fractals, as formulated by *Shlesinger*.^[16] Other approaches that invoke scaling are based on geometrical and on energetic aspects: for the former we mention in this context fractals and percolation models and for the latter ultrametric (hierarchical) structures (UMS). All these concepts lead (for certain values of the parameters) to dispersive transport, as we discuss in the next Section.

3. New Experimental Data and Theoretical Aspects

We discuss charge carrier transport (mobility) by focussing on one of the historic hole-transport polymers, namely

polyvinylcarbazole (PVK).^[17a] Here, recent experimental data have given rise to new questions concerning the general theoretical concept of transport in disordered media. The basic question is the nature of the disorder: Is it mainly geometric or mainly energetic or both?^[17-20] Furthermore, which are the microscopic parameters that control the macroscopic mobility in polymeric materials?

PVK is prepared in 10 μm thick films in which both solvent and residual monomers have been removed.^[17a] The polymer films are mounted between aluminum electrodes produced by vapor deposition (see Fig. 1). The top electrode is semi-transparent and allows illumination with laser pulses of a time duration of typically 10 ns and a wavelength of 337 nm (N_2 laser). At this wavelength the polymer absorption is characterized by the carbazole moiety; therefore the typical penetration depth of the light is less than 0.5 μm . The light pulse thus creates electron-hole pairs within a thin surface layer of less than 1/20 of the sample thickness. With the positive electrode as front electrode, one therefore observes photocurrents, where holes (positive charges) are transported through the 10 μm -thick bulk PVK sample.

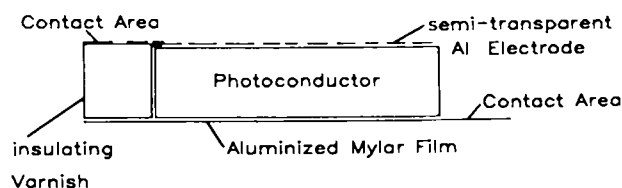


Fig. 1. Experimental arrangement for the photoconductivity measurements. The top electrode is semi-transparent aluminum. The insulating varnish prevents electrical breakthrough of the sandwich-type sample (see [17b]).

Even though the light pulse has a duration of only 10 ns and thus limits the time resolution of the experiment to the 10^{-8} s range, it triggers a photocurrent which traverses the 10 μm -thick sample in roughly 10 ns and which can be experimentally monitored up to the time regime of 10 to 100 s. This wide sampling range is important for a meaningful evaluation of the experimental data.

Figures 2 and 3 show transient hole-photocurrents in PVK as a function of field and temperature taken over about nine decades in time (10 ns to 10 s) and seven or eight decades in intensity.

The gross features of the photocurrent show an initial rise time in the range of 10^{-8} s, followed by an algebraic slow decay into the 10^{-3} s time regime ($I \sim t^{-\alpha-1}$, see below). At this point the first charge carriers arrive at the back electrode after having traversed the sample, and thus the slope of the photocurrent changes abruptly (to $t^{-\alpha-1}$; see below). The time at which this break in the slope occurs is called "transit time". It characterizes the macroscopic response time of the polymer sample of 10 μm thickness. For technical applications the material is some 10 μm thick and thus typical transit times are on the order of 10^{-3} to 10^{-2} s. These times are sufficiently short for tech-

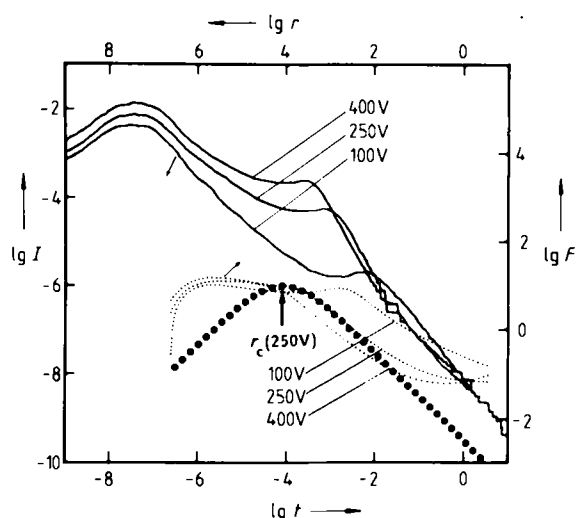


Fig. 2. Solid lines: Photocurrent of PVK (10 μm thick) at constant temperature (61.5°C) and various field strengths. Dotted lines: Analysis of rate distribution. For details see [17a].

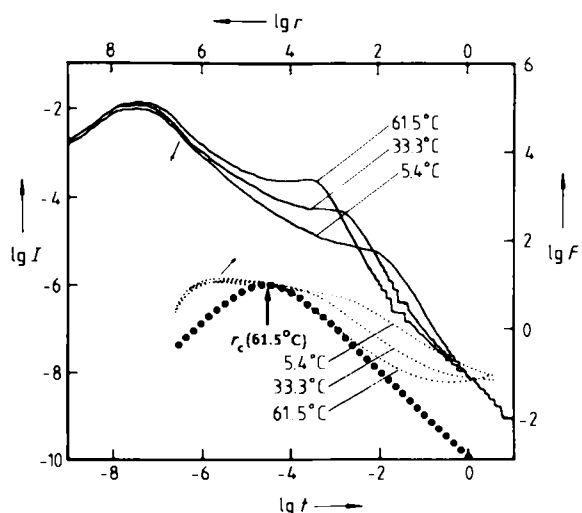


Fig. 3. Solid lines: Photocurrent of PVK (10 μm thick) at constant voltage (400 V) and various temperatures. The experimental data range over nine decades (left and lower axis). Dotted lines: Rate distributions. For details see [17a].

nical applications in electro-photography and electro-printing.

The difference between the data presented in Figures 2 and 3 and those published earlier (experiments on related systems) is the fact that the slope of the current (which characterizes the transport properties of the material and may be related to α) varies, as a function of time, over its full range, from 1 at short times to 0 at long times.^[17a] Such a behavior can only be noticed when a sufficiently large dynamic range is monitored. If, as in many previous investigations, only four decades or less are accessible, a definite analysis to discriminate between the various microscopic processes is hardly possible.

The analysis of the rate distribution in Figures 2 and 3^[17a] is rooted in the multiple trapping (MT) formalism put forth by Schmidlin.^[21] MT can be viewed as a phenomenological basis to characterize microscopic processes in which the main source of dispersion are energy-level fluctuations (see below). Hence, microscopic rates can be extracted from the experiment, but only when the current is monitored over many orders of magnitude in time; from measurements over nine decades one can determine rate distributions over four to five decades.^[17a]

Let us remark that theoretically one usually goes the opposite way and assumes a $\psi(t)$ distribution, from which the transient currents are then calculated. This is somewhat arbitrary, but the procedure can be used to demonstrate that the current is not very sensitive to the details of the model. In fact one can show that MT and CTRW give the same result.^[21-23] For a finite sample one finds^[23] the following relation between the Laplace transformed current $I(u)$ and waiting-time distribution $\psi(u)$:

$$I(u) = \frac{\psi(u)}{1 - \psi(u)} (1 - [\psi(u)]^N) \quad (c)$$

Nd is the thickness of the sample and d the distance between neighboring traps.

The advantage of Eq. (c) is its relatively simple structure, so that the numerical effort involved in obtaining $I(t)$ is small; only an inverse Laplace transformation is required. Our analysis^[23] highlights several points, by using $\psi(t)$ forms as in Eq. (b):

1. Well inside the dispersive regime (i.e. for α around 0.5) one obtains for not-too-short times the classical Scher-Montroll behavior:^[12]

$$I(t) \sim \begin{cases} t^{\alpha-1} & \text{for } t < t_T \\ t^{-\alpha-1} & \text{for } t > t_T \end{cases} \quad (d)$$

where t_T is the effective transit time.

2. For values of α larger than 1.0 one has, after an initial dispersive regime, Gaussian behavior.
3. The value $\alpha = 1$ is marginal; the plot of the current versus time continuously changes its slope, crossing over from dispersive to non-dispersive (Gaussian).

These findings make clear that a careful analysis of the whole current behavior is required in order to determine $\psi(t)$ from $I(t)$, since even simply structured $\psi(t)$ distributions may lead to complex $I(t)$ forms, and one cannot always rely on expressions like Eq. (d) to determine α . Moreover, up to now the microscopic mechanisms which lead to $\psi(t)$ were not discussed. The original CTRW proposition^[12] was that in a chemically pure, amorphous solid the distribution of distances between the atoms gives rise to dispersive $\psi(t)$ forms, and that geometrical disorder is therefore involved. Careful computer simulations^[24,25] indicate, however, that in most chemically pure solids the

variation of distances can hardly lead to quite low α -values, such as $\alpha \leq 0.5$. The counterproposal, which leads to MT, was to invoke energetic disorder:^[21,22] here the motion of the carriers is slowed down by trapping. In fact, an exponential distribution of traps^[26,27] leads directly to $\psi(t)$ forms such as Eq. (b).

Due to the mathematical equivalence between CTRW and MT it is impossible to decide from a single $I(t)$ curve whether $\psi(t)$ is dominated by geometric or energetic disorder. One needs additional information. As an example, if the disorder is energetic and if the intersite transitions are thermally activated, then $\psi(t)$ will be strongly influenced by the temperature. However, the effect of temperature variation on the intersite distances is considerably smaller. As a consequence, it is very important to monitor the temperature dependence of the current. These experiments, however, are extremely difficult. On the one hand the "temperature window" in which photoconductivity experiments can be performed is rather small (typically 300 K to 400 K). At low temperatures charge carrier trapping becomes dominant and at high temperatures the polymeric materials show irreversible changes due to chemical or structural changes. On the other hand the temperature dependence of the $I(t)$ curves often does not yield a simple characteristic time exponent α (slope) if measured over a wide enough range (see Fig. 3). Here only a detailed mathematical analysis combined with measurements over more than four decades in time is sufficient to decide on the nature of the phototransport mechanism. However, measurements over such large time regimes are scarce.

The theoretical aspects of the problem are further complicated when the substrate is not chemically pure, but consists of impurity centers embedded in an inert matrix. A recent investigation used a benzotriazole derivative embedded in a polycarbonate matrix.^[28] In such a situation, especially at relatively low impurity concentrations ($\leq 10\%$), the fluctuations in the intersite distances become very drastic. As a result, the influence of the geometric disorder may become dominant.

The prototype problem for geometric disorder in a binary mixture is percolation. In the standard model^[29]—there are many variations—sites on a lattice are conducting (with probability p) or non-conducting (with probability $1-p$) and the intersite interaction is of a short-range nature (nearest neighbors only). The current flowing through the sample depends on p . For values less than a critical value p_c no current flows at all, whereas slightly above p_c one has

$$I \approx I_0 (p - p_c)^t \quad (p \geq p_c) \quad (e)$$

Here p_c depends on many details of the model (interaction range, lattice type, number of nearest neighbors), whereas t is less influenced by details, and depends mainly on the lattice dimension ($d=2$ or 3). Note that above p_c the cur-

rent should be Gaussian (beware, however, of crossover effects near p_c). Figure 4 shows the carrier mobilities μ_T found for a mixture consisting of a charge transport component in an insulating substrate (benzotriazole in polycarbonate matrices). The experimental data seem to be consistent with the percolation picture.

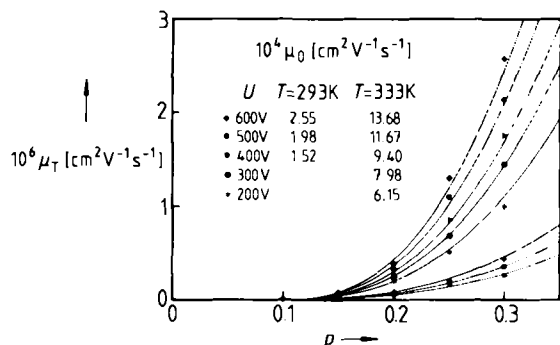


Fig. 4. The effective mobilities μ_T as a function of the benzotriazole concentration p . The μ_T data were measured at the U and T values given in the inset. All curves are fits to the percolation model, Eq. (e), with $p_c = 0.1$ and $i = 2.5$. The resulting μ_0 values are listed in the inset.

The situation at p_c is, in the standard percolation, most interesting. Here one encounters the incipient infinite cluster on which transport is *dispersive*. This is due to the large fluctuations on connectivity at p_c , so that the incipient infinite cluster contains voids of all sizes. The structure is self-similar, or, in modern language, fractal.^[14] Fractals display a subtle, lower symmetry: they are dilatationally invariant, but, in general, they lack translational symmetry.

As far as the total photocurrent is concerned, fractals can be specified by several parameters; in fact one can dispense with the stochastic intricacy of the incipient infinite cluster and model it through regular looking, deterministic fractals.^[30] For diffusion on fractals one again has Eq. (a), where now α is given by:

$$\alpha = \bar{d}/d \quad (f)$$

\bar{d} being the fractal (Hausdorff) and d the spectral dimension of the underlying structure.^[31] Use of the Einstein relation, which also holds for fractals^[32,33] implies that the time dependence of the current also follows Eq. (a). In general $\bar{d} \leq d$; thus at p_c one finds $\bar{d} \approx 4/3$ whereas $d = 1.9$ for $d = 2$ and $\bar{d} \approx 2.5$ for $d = 3$.^[29] Hence, the current is dispersive on percolating clusters at p_c .

It should be evident to the reader by now that the root of dispersive behavior, Eq. (a), lies in some self-similarity (scaling) of the underlying model. In the case of fractals the scaling aspect is *geometrical* (dilatation). In the CTRW model the scaling is connected to the *temporal* aspect, as expressed by Eq. (b), which is form-invariant under time-dilatation. In fact, one may use for $\psi(t)$ the Weierstrass form,^[19,27] which is defined in the whole time domain:

$$\psi(t) \sim \sum_{k=1}^{\infty} (ab)^k \exp(-b^k t) \quad (a < 1, b < 1) \quad (g)$$

At longer times $\psi(t)$ behaves like Eq. (b), scales, and has $\alpha = \ln a / \ln b$; this prompted *Shlesinger* to call such forms time fractals.^[16]

Furthermore, one may also use *energetic* self-similarity in order to obtain dispersive transport. The idea is somehow inherent in the exponential density of states, but becomes evident through model descriptions such as ultrametric spaces (UMS). Here the basic idea is to start from a Cayley tree (Bethe lattice), where the tips correspond to different sites, and the energetic separation between sites is given by the length of the minimal path connecting them.^[19,34] One can now show that at low temperatures the activated transport is dispersive, the parameter α being now a function of temperature, barrier-height and connectivity of the lattice.

4. Outlook

The progress that has been made recently in the theoretical description of non-Gaussian transport phenomena has created challenges for the experimentalist. It is evident that the dynamic range of most available transport data is not large enough to answer even rather basic questions such as:

- Is disorder mainly of geometric or of energetic nature?
- How well are the basic distribution functions known? (Is the trap distribution exponential, or Gaussian, or of some other type?)
- What are typical "cut-off frequencies"? Are they intrinsic or are they imposed by the nature of the experiment?

For the materials scientist the open questions are:

- What are the intrinsic limits of feasible mobilities? Can amorphous materials match the properties of crystalline materials? (Mobilities on the order of $\mu = 1 \text{ cm}^2/\text{Vs}$?)
- Is the performance of polymers limited by chemical or by structural imperfections?

All of the above questions can only be dealt with if one uses modern spectroscopic tools with high time resolution. One also has to vary the materials parameters from amorphous to partially crystalline to liquid crystalline and thus utilize the flexibility offered by the wealth of organic synthetic chemistry and polymer chemistry.

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Conference Reports

Organic Materials for Nonlinear Optics

The study of nonlinear optical effects in organic materials has been an active area of research for over ten years. These materials are now attracting considerable industrial interest through their potential for controlling and transforming laser radiation. The International Conference on Organic Materials for Nonlinear Optics, organized by the Royal Society of Chemistry and held at the University of Oxford, England, on 29–30 June 1988 (OMNO '88) was only one of several this year; there have already been meetings at Virginia Beach, USA, and Antibes, France, and the SPIE meeting in San Diego is yet to come. The roughly 160 delegates to OMNO '88 came from eight different countries and almost half were from industry. Its format of a single session over two days was just about right for covering the subject in depth while avoiding fatigue; the atmosphere was friendly, and the charming conference accommodation in historic Balliol College right in the center of Oxford assisted the lively evening socializing.

Fittingly, the first speaker (J. Zyss, CNET, France), who was among the founders of the field, spoke about the realization of one of his long-term goals, that of achieving parametric amplification in a single-crystal material. He has applied the technique as a very sensitive way of studying sub-picosecond phenomena. From his highly professional, extensively characterized and beautiful results, as well as those of J. N. Sherwood (Univ. of Strathclyde, UK) on crystal growth and S. Allen (ICI, UK) on electro-optic effects, it was apparent that second-order effects in single crystals are much better understood than any other aspect of the field. In fact, the conference heard the news that large, optical-quality crystals of 3-methyl-4-nitro-pyridine

1-oxide (POM) are now available on a commercial basis. A. Gavezzotti (Univ. of Milan, Italy) and R. W. Munn (UMIST, UK) had the design of future high-performance second-order crystalline materials in mind with their theoretical studies of molecular packing and local field effects, respectively.

Other excitement was generated by work on electro-optic effects in poled polymers. D. R. Ulrich (US Air Force Office of Scientific Research) gave well-reasoned arguments for his continued support of basic research in this area, and some experiments on these materials were reported by G. R. Möhlmann (Akzo, Netherlands) and N. Carr (Plessey, UK). However the most promising results were those of J. R. Hill (British Telecom, UK), and especially R. Lytel (Lockheed, USA), who appeared close to complete mastery of the technology. The electro-optic figures of merit reported are comparable with the best alternative materials, and the dispersion of dielectric constant much smaller. The major remaining task is to reduce levels of scattering.

In the area of third-order effects in conjugated polymers, there appears to have been a strategic retreat to understand the basic science. These materials, which were initially the front runners in the field, have now been faced with very effective competition from multi-quantum wells and other inorganic semiconductor materials. P. Prasad (SUNY, Buffalo, USA), F. Charra (CEN Saclay, France), A. F. Garito (Univ. of Pennsylvania, USA) and D. Pugh (Univ. of Strathclyde, UK) all attempted to understand the optical Kerr effect in terms of the interplay of molecular states, while G. R. Meredith (Du Pont, USA) also emphasized the contribution from cascaded second-order re-