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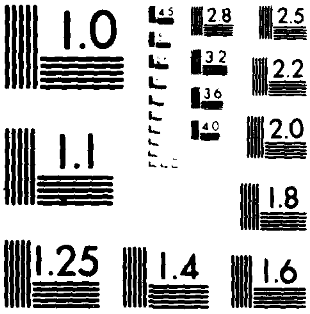

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TEMPORAL AND TEMPERATURE DEPENDENCE OF THE ENERGY TRANSFER PROCESS AMONG Eu^{3+} IN AN AMORPHOUS SOLID

by

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ABSTRACT

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INTRODUCTION

The description of the time dependence of energy transfer between like ions or molecules randomly arranged in solids or solution has recently become an area of active theoretical and experimental research. In a system of optically active species arranged in a well-defined periodic lattice, the energy migration can be described by the diffusion equation in which the diffusion coefficient is independent of time,[1] yielding an exponential decay of the excited donor species.[2-4] When the optically active molecules or ions have instead random energies and distance of separation, a distribution in coupling would exist which makes the diffusion constant a function of time[1] and the decay of the donor becomes nonexponential.[2] How nonexponential the decay of the donor becomes was the main question asked by a number of theoretical treatments in three[1-7] and one[8,9] dimensional systems transferring their energy by a phonon assisted (nonresonant) energy transfer process. In all of these treatments, the time dependence of the energy transfer process, as followed by the decay of the emission intensity of the donor, is predicted to vary during the decay and to depend on the concentration as well as the degree of randomness of the optically active species studied. These theoretical predictions suggest that at long enough time a diffusive behavior, in which the donor intensity decays as $t^{-3/2}$ for a three-dimensional system, should be observed. Recently it has been theoretically shown[10] that the diffusive behavior is reached faster at higher concentrations for transfer between like ions or molecules in solution.

On the experimental side, direct donor-donor energy transfer has been examined either indirectly by concentration quenching, by trapping, or more directly by either studying the concentration dependence of the lifetime or the changes in the polarization ratio of the fluorescence emission. The theory of the latter has been worked out and compared.[11-14] More recently, with the advent of lasers, direct like-ion transfer resulting in spectral and spacial diffusion has been studied by either time resolved fluorescence line narrowing (FLN) techniques[15] or by grating methods.[16] In the FLN studies, the time development of the site selected donor fluorescence as well as the inhomogeneous spectra for the Pr^{3+} ion in crystalline LaF_3[17-21] and Eu^{3+} in $Ca(PO_3)_2$ glass[22-28] have been examined. The results of the time resolved FLN techniques have shown[17-26] that the energy transfer in these systems is phonon assisted whose rate is independent of the energy mismatch at the temperatures used in these studies (at which KT is greater than the inhomogeneous width). The Inokuti-Hiroyama equation[5] was found to be useful in analyzing the data from which a multipolar mechanism has been tested.[17-26] The results of all the workers on these different ions suggest a dipolar mechanism except for one study[27] on Eu^{3+} that suggests a quadrupolar mechanism. In this letter, we carefully examined the temporal and temperature behavior to determine the transfer mechanisms involved in the different time regimes during the course of the transfer.

Time resolved FLN techniques involve recording the spectra at different delay times after short time pulsed excitation of a selected site. While this technique gives detailed information concerning the time development of the spectral frequency characteristics, it does not cover the details of the time domain of the donor decay, since only a few delay times are used under different excitation and detection conditions. In this communication we have used a gated photomultiplier[26] to record the fluorescence decay in resonance with the laser excitation of a site absorbing at 5760 Å within the inhomogeneous profile of the $^7F_0$ - $^5D_0$ absorption of 10 and 30 mol percent Eu^{3+} in $Ca(PO_3)_2$ glass over the 100 K - 450 K temperature range.

Three time regimes have been observed: the short time regime for which all three multipolar mechanisms describe the transfer, the time regime for which the majority of the decay (∿ 90% of it) is described by a dipolar mechanism (in agreement with previous workers[22-26, 28]), and the regime for which long time $t^{-1.5}$ fit could be made, suggesting a diffusion behavior. The range of the fit at early and at long times is examined as a function of temperature in the 100 - 450° K range.

EXPERIMENTAL

Glass samples of 10 and 30 mole percent Eu^{3+} in $Ca(PO_3)_2$ were used. Sample sizes were approximately 1x1x2 cm, were well polished and of good optical quality. The 5760 Å output from a Quanta-Ray DCR-1 pulsed Nd:YAG pumped PDL-1 dye laser with a 6 ns fwhm pulse width and ∿ 0.3 cm^{-1} spectral width was focused into the sample with a width of approximately 0.5 mm. THe excitation wavelength corresponds to the high energy side of the inhomogeneously broadened $^7F_0$ - $^5D_0$ Eu^{3+} absorption in this glass. The $^5D_0$ - $^7F_0$ fluorescence was collected at right angles to the laser beam and focused into the slits of a 1 M Jarrel-Ash monochromator with an effective slit-width that varied between 0.6 and 1.8 cm^{-1}, considerably less then the observed homogeneous width of the transition. The monochromator was carefully turned to the laser wavelength so as to follow the intensity of resonant fluorescence. Resonant detection required that the phototube be gated off during the laser pulse to prevent saturation of the phototube by the intense scattered laser light. The same gated phototube of ref. 26 was used with a 5 μs gate width. The signal was fed into a Biomation 805 waveform recorder with 5 and 10 μs sampling intervals. The digitized signal was averaged over 10,000 laser shots by a homebuilt signal averaging computer linked to a PDP 11/45 computer. Data was taken at 50 K intervals in the 100 K to 450 K temperature range.

DATA ANALYSIS

The physical quantity of interest in these experiments is the population of the excited donor ions at any time minus its equilibrium steady state value (attained when the net transfer of excitation is zero) after correcting for the population loss due to first order decay processes. The set of donor ions consists of those ions initially excited by the laser pulse at time t = 0 and those that fluoresce in resonance with the initially excited ions. In order to determine this quantity in terms of experimental parameters, the following expression[26] is used:

$$P(t) = [I(t)\exp(t/\tau_0) - P_{ss}]/(1-P_{ss}) \qquad (1)$$

where P(t) is the population of excited donor ions at time t minus its steady state value resulting when the net transfer of excitation is zero, after correcting for the first order decay. P(t) changes from 1 at t = 0 to zero when the excitation density of the donor population fits the inhomogeneous profile of the $^5D_0$ - $^7F_0$ fluorescence (i.e. reaches a steady state value). I(t) is the emission intensity at time t normalized to its value at t = 0; τ_0 is the observed first order lifetime of the

$^5D_0$ - $^7F_0$ transition, and P_{ss} is the steady state population of excited donor ions, when the relative excited donor intensity at the donor frequency fits the uniform inhomogeneous profile. The emission intensity at $t = 0$ was found by extrapolating the observed early time behavior, $\ln(I) + t/\tau_0 \propto t^{1/2}$ consistent with a dipole-dipole transfer mechanism,[5] to $t = 0$.

To determine P_{ss}, the method illustrated in Fig. 1 is used. If the long time exponential part of the donor emission is taken as the first order decay of the full inhomogeneous profile after transfer equilibrium is reached, then the $t = 0$ intercept of the line fitted to the exponential part gives P_{ss}, the steady state population of the donor (corrected for the first order decay). The slope of the line gives τ_0. For the 30 percent sample at temperatures > 250 K a value of 0.05 ± .01 is obtained for P_{ss}, independent of temperature. This value is about what is to be expected from consideration of the linewidth of the homogeneous profile at 5760 Å relative to the inhomogeneous profile. Artificially higher values for P_{ss} are observed at temperatures below 250 K for the 30 percent sample and for the 10 percent sample at all temperatures. This is the result of a decrease in the transfer probability relative to the first order decay which prevents the steady state from being reached (due to the dominance of the first order decay). A value of 0.05 has been used for both glasses at all temperatures. Eq. (1) then becomes:

$$P(t) = [I(t)\exp(t/\tau_0) - 0.05]/0.95 \qquad (2)$$

RESULTS AND DISCUSSION

The results of the time dependence of the donor fluorescence intensity are shown in figs. 2 and 3. Fig. 2 shows the results of attempting the decay of the donor excitation ($P(t)$) to the different predicated behavior for a dipole-dipole (top), dipole-quadruple (middle) and quadrupole-quadrupole (bottom) mechanisms. Fig. 3 is a $\log(P)$ vs $\log(t)$ plot of the long time behavior to test for the diffusion limit.

The theoretical results of Inokuti and Hirayama predict that, in the absence of back transfer from acceptor to donor, the donor decay can be described by $\ln(I) + t/\tau_0 \propto -t^{3/s}$, where $s = 6,8,10$ for dipole-dipole, dipole-quadrupole, and quadrupole-quadrupole coupling respectively. (Huber et al[2] have considered various approximations for back transfer where they find that the donor decay at early times can be described by $\ln[P(t)] - t^{3/s}$.) Fig. 2 shows the attempt to fit the behavior of $P(t)$ to these three mechanisms for the 30 percent sample at room temperature. As shown in the figure, at short times all three mechanisms can describe the transfer process. However, the dipole-dipole mechanism continues to describe the transfer until $\sim$ 90 percent of the excitation has been transferred. At very short times, i.e., transfer over short distances, any mechanism (even exchange mechanism) may lead to transfer. All the terms resulting from the expansion of the coulombic term could lead to transfer. At long time (large distances) the relatively strong long range dipole-dipole mechanism is found to describe the transfer. As shown in Table 1 the dipole-dipole mechanism describes $\sim$ 90% of the decay of $P(t)$ for the 30 percent sample for temperatures $\geqslant 250$ K. At lower temperatures or concentration first order decay dominated before an observation of the long time behavior could be made. This is a result of the decrease in the transfer probability relative to the first order decay rate.

At very short times an exponential decay of P(t) may be expected due to transfer to acceptors at the discrete nearest neighbor separation.[2,6] This effect has been observed in crystalline Pr:LaF$_3$ at very short times.[2] This very short time behavior was not observed on the time scale of our experiments (which might not have been sufficiently short).

At long time, the $\exp(-(t/\tau_{TR})^{1/2}$ fit of P(t) fails for both the 10 and 30 percent samples at all temperatures. Diffusion behavior has been postulated as the long time limit and has been observed in Monte Carlo calculations in both one[29] and three[6] dimensions. Diffusion in three dimensions is characterized by a $P(t) \propto t^{-3/2}$ behavior. Fig. 3 shows the fit at long time for the 30 percent sample at room temperature. Although the signal to noise is poor and the slope more sensitive to the measured value of P_{ss} than at early time, the data is consistent with a $P(t) \propto t^{-3/2}$ behavior when the donor excitation has dropped to less than 5 percent of its initial value. This behavior is reached in a shorter time than the first order lifetime and after ~ 95 percent of the excitation has been transferred. The full -1.5 slope in the log-log plot is reached only for the 30 percent sample at room temperature and above as shown in Table 1. For the slower transfer rates resulting from a decrease in temperature or concentration, the first order decay process dominates the excitation decay of the donor before an observation of the diffusion behavior can be made. These results are in good agreement with the calculations of Gochanour et al.[10]

In summary, we have found three time regimes for the decay of P(t): i) a short time regime for which three terms in the multipolar expansion can describe the transfer; ii) most of the remaining of the decay in which the dipole-dipole mechanism seems to dominate; and iii) a long time regime consistent with diffusion behavior after 95 percent of the excitation has already been transferred only in the 30 percent sample and at temperatures $\geqslant 298^0$ K. At low concentration or temperature the first order decay was found to dominate the emission before an observation of the long time behavior could be made.

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Figure Captions

Fig. 1. Semilog plot of intensity vs. time (t) for 30 mole percent $Eu^{3+}:Ca(PO_3)_2$ glass at 298 K illustrating the method used for determining the steady state value of the donor excitation probability (P_{ss}) and the donor first order lifetime (τ_0) (see text). The first order lifetime for the glasses used in this study is ∿ 1.5 msec.

Fig. 2. The electronic coupling transfer mechanism, a fit to the Inokuti-Hirayama equation for a dipole-dipole (top), dipole-quadrupole (middle) and quadrupole-quadrupole (bottom) mechansims for a 30 percent Eu^{3+} sample at room temperature. The figure shows that at short time (transfer over short distances) all the mechanisms can describe the transfer while the long range dipole-dipole mechanism continues to describe the transfer for ∿ 90 percent of the total donor excitation.

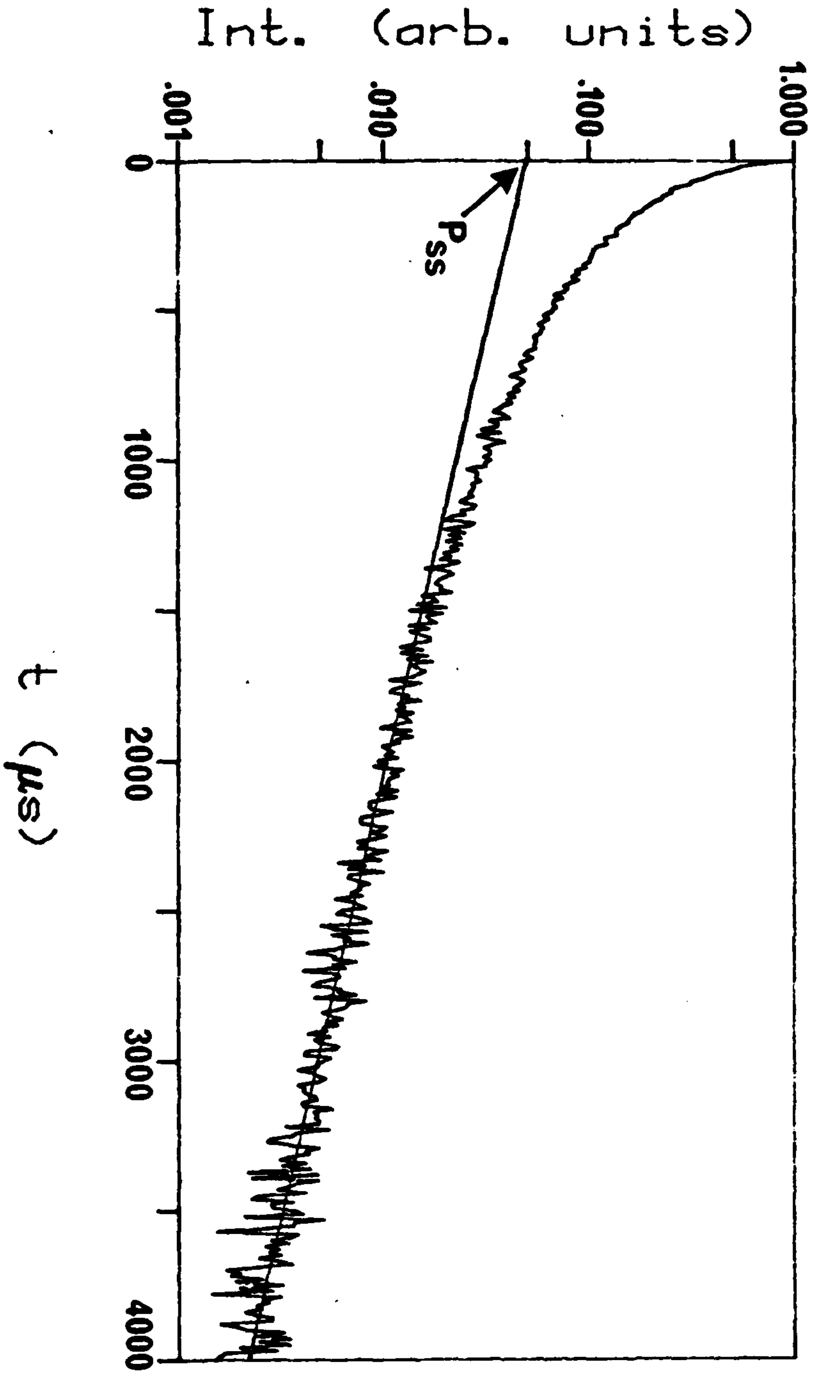
Fig. 3. The diffusion limit: The long time fit of the decay of the donor excitation to diffusion behavior ($P \propto t^{-3/2}$) for a 30 percent sample at room temperature. This limit is reached after ∿ 95 percent of the excitation is transferred for this sample at 298° K. This behavior is not seen for lower temperatures or concentrations.

Table 1. Temperature dependence of observed range for the dipole-dipole mechanism and the diffusion behavior in the 30 percent Eu^{3+} sample in calcium metaphosphate glass.

Temperatures (°K)	Dipole-Dipole Range * from P=1 to P=	Best value * for long time slope[†]	Diffusion Range from P=0 to P=
100	0.34 ± 0.10	-0.55	-
150	0.30 ± 0.10	-0.65	-
200	0.21 ± 0.07	-0.87	-
250	0.12 ± 0.02	-1.3	-
300	0.08 ± 0.02	-1.5 ± 0.3	0.04 ± 0.02
350	0.06 ± 0.02	-1.5 ± 0.3	0.05 ± 0.02
400	0.06 ± 0.02	-1.6 ± 0.3	0.03 ± 0.02
450	0.08 ± 0.03	-1.6 ± 0.3	0.03 ± 0.02

[†] For logP vs logt plot; a slope of -1.50 might suggest diffusion limit.

* For temperatures lower than 300 K, the transfer rate is decreased sufficiently that the decay of the donor was dominated by first order process which prevented observation of the long time behavior of the transfer process.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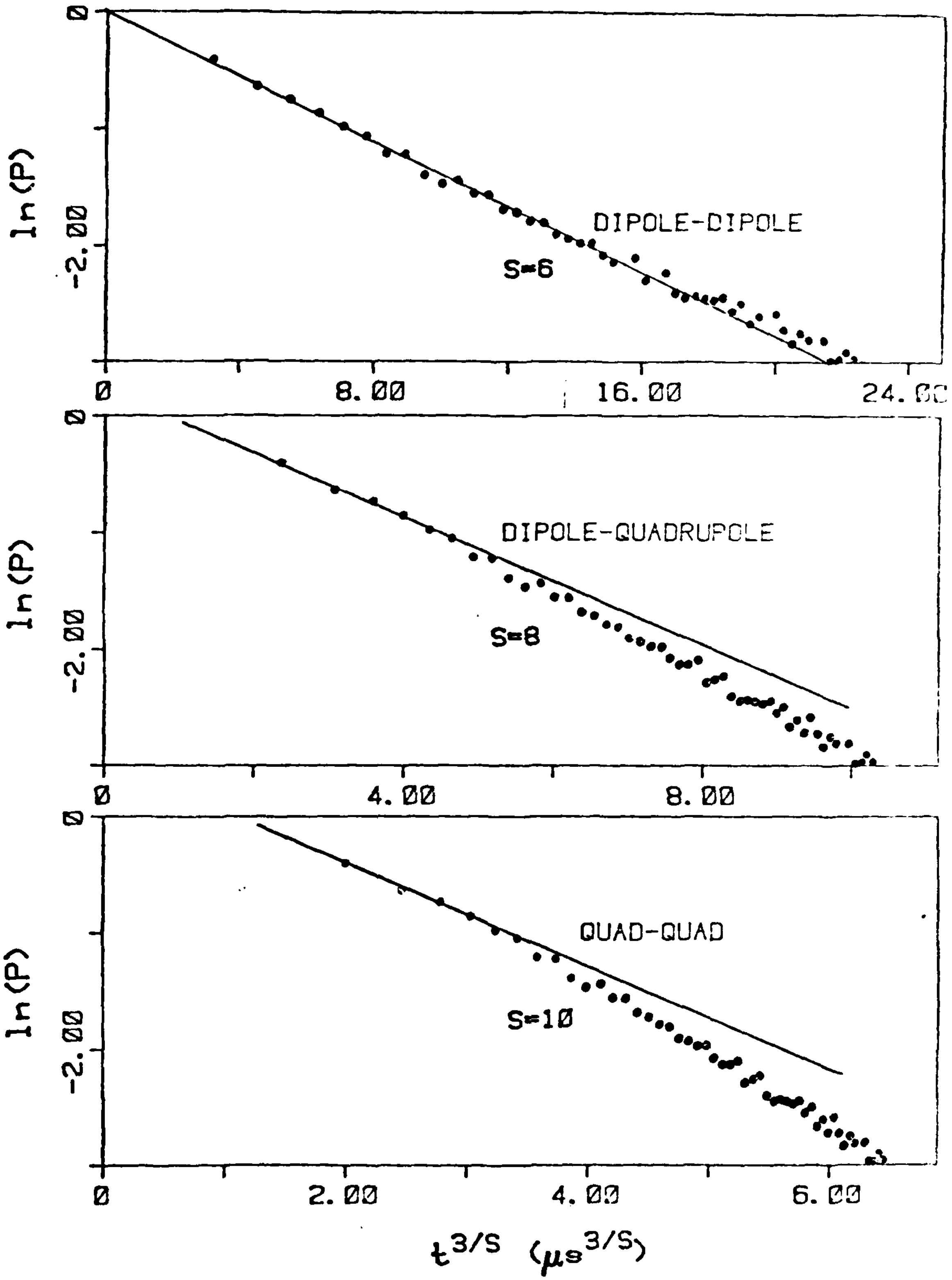

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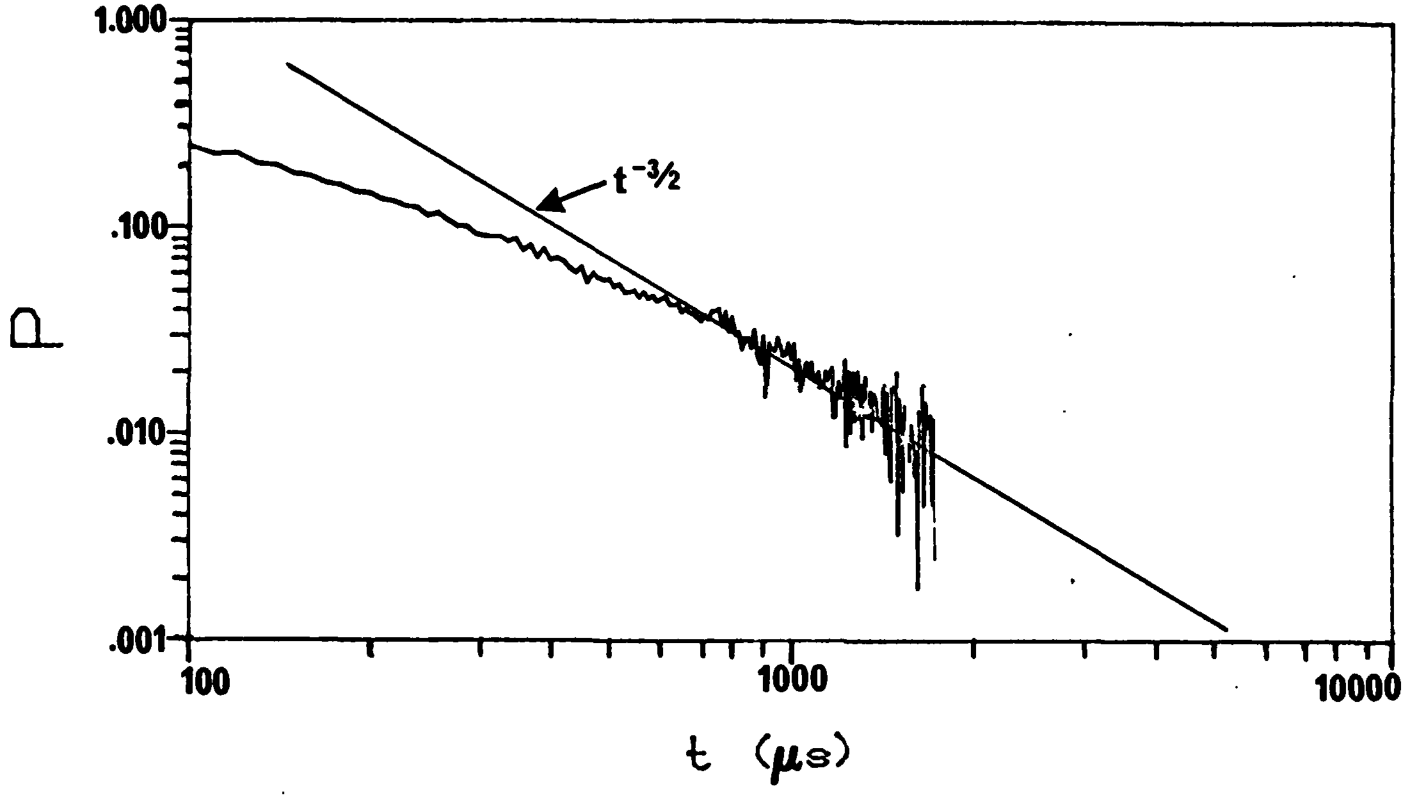

Fig. 3

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