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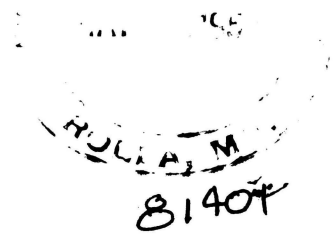
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A LINEAR POLYMER
X-RAY SCATTERING MODEL

BY

BERNARD COHEN

A
THESIS



submitted to the faculty of the
SCHOOL OF MINES AND METALLURGY OF THE UNIVERSITY OF MISSOURI
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Degree of
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1952

Approved by *Bernard N. Lund*
Professor of Physics

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B.C.

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INTRODUCTION

In the development of X-ray scattering intensity curves physical models are necessary for the theoretical mathematical development involved. The purpose of this paper is the development of a physical model suitable for X-ray diffraction analysis of a linear polymer.

X-ray intensity patterns may be used to give a greater insight as to the atomic structure, configuration of atoms, molecular weight, and therefore a greater knowledge of the structure of matter, both microscopic and macroscopic. The converse may sometimes be used. An atom or molecular model is constructed, from this model X-ray scattering curves may be derived. These curves may be compared with known physical curves and if correct give us a greater insight into the mechanics of the interaction of matter.

The success or failure of the model depends on the details involved in constructing the model. The model should have characteristics that represent the outstanding property of the real material. If potential energy is assumed to be of prime importance, then the model should be constructed so that potential energy is the predominate parameter in describing the system.

The following model takes the chemical-structure unit of the polymer as a unit or cell, and assumes the displacements of the cells are small enough such that the force between two adjacent structural-units is proportional to their separation distance.

REVIEW OF LITERATURE

The application of light scattering to molecular weight determination has been applied by Debye⁽¹⁾ in the case of polymers. Debye's polymer model consists of a flexible polymer of N links each of length A, which can rotate freely. Any interaction of parts of the chain with each other are neglected.

Light intensity scattering formula are derived by Debye for a flexible polymer as follows:

$$I = \frac{2}{X} \left[(\exp-X) - (1-X) \right] \quad (1)$$

where

I = average intensity scattered at angle α

$X = KS^2R^2/6$

$S = 2 \sin \frac{\alpha}{2}$

$K = 2\pi/\lambda$

R = average square of the length of a single polymer chain

α = angle between primary and secondary radiation

For rigid polymers having spherical shape

$$I = \frac{3}{X} \left[(\sin X) - (X \cos X) \right] \quad (2)$$

For the special case of low angle X-ray scattering Kratky⁽²⁾ used a very simple model. The model consists of an

(1) Debye, P. J. of Phys. & Colloid Chem. 1, 27, (1937)

(2) Kratky, O. J. of Polymer Science, 3, 195, (1948)

infinite, rigid chain of spheres, each sphere being in contact with its neighbors. Using this simple model he derived the following X-ray scattering formula:

$$I = f^2 \left[1 + 2 \left[\frac{\sin X}{X} + \frac{\sin 2X}{2X} + \frac{\sin 3X}{3X} + \dots \right] \right] \quad (3)$$

where

r = radius of the sphere

$f = (\sin u - u \cos u) / u^3$, X-ray scattering form factor for a sphere

$X = 8\pi\theta r / \lambda$

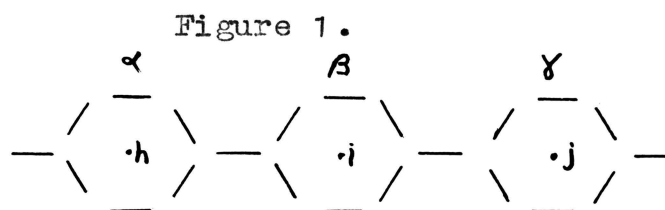
λ = wavelength of incident X-ray

θ = one half scattering angle

Kratky's method may be used to compute the molecular weight of the polymer. Both methods give theoretical results which are compatible with known data.

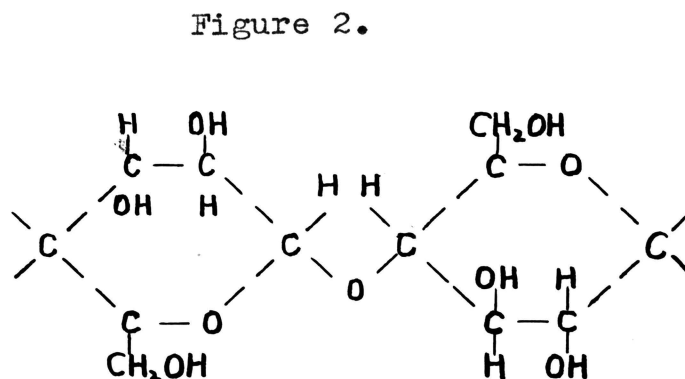
THEORY

In this section we are going to derive X-ray scattering formulas from a simplified linear polymer model. Evidence indicates that the structure of linear polymers are built up by successive repetition of small simple units. These cells can be diagrammatically shown as follows:



(Diagram of hypothetical linear polymer where h, i, j, represent mass center of cells α , β , γ , respectively.)

This preceding diagram is an over simplified version of the following monomer.

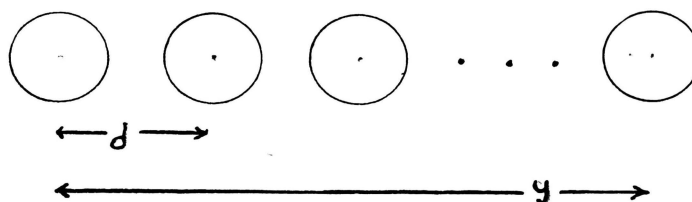


(Cellulose-Fiber)

Considering the fact the unit cell may have sensibly free rotation around the linear axis, we may visualize the unit as presenting to the X-ray beam "on the average" a spherical scattering region. A cell is then defined as a spherical scattering region, and the cell center is merely a geometric mass center of the monomer.

Figure 1 can be represented by the following simple one dimensional model.

Figure 3.



(One dimensional model of a linear polymer.)

Where d is the cell separation corresponding to the equilibrium distance between two adjacent cells. This is assumed to be a stiff model where neither bending or coiling occur.

Now the X-ray scattering will be characterized by a function $P(y)$ which is the probability of finding a polymer cell in a unit length at y from any cell in the polymer. Using the model already described we shall develop an expression for the function $P(y)$.

The above model is crude from the view point of geometric symmetry, as far as X-ray scattering is concerned. A correction factor or form factor⁽³⁾ will be introduced to compensate

(3) Kratky, O. J. of Polymer Science, 3, 195, (1948)

for the lack of symmetry. This correction need not be applied to the subsequent development.

Now consider the following:

$$\int \dots \int (\exp \sum_{i=1}^j [-K/kT] x_i^2) dx_1 \dots dx_j \quad (4)$$

where

R = diameter of cell

K = force-constant, i.e. force per unit relative displacement

k = Boltzmann constant

T = absolute temperature

$\sum_{i=1}^j x_i = y - jd = \text{a constant}$

It should be remembered that the displacements are small enough such that force is proportional to the displacement between two adjacent cells.

The above integral expresses the probability of finding the j-th cell between y and y + dy and is subject to the following condition.

$$\sum_{i=1}^j x_i = y - jd = \text{a constant} \quad (5)$$

In inserting the integral limits we notice the limits are different for each particular integral, in general

$$\int_R^{y-jR} \int_{2R}^{y-(j-1)R} \dots \int_{jR}^y (\exp \sum_{i=1}^j [-K/kT] x_i^2) dx_1 \dots dx_j \quad (6)$$

The extreme complexity of the integral limits necessitates an artifice to circumvent this difficult integration. Consider the following:

$$\frac{dy}{2\pi} \int_{-\infty}^{+\infty} (\exp[i(y-p)u]) du = \begin{cases} 1 & y - \frac{dy}{2} < p < y + \frac{dy}{2} \\ 0 & \text{outside these limits} \end{cases} \quad (7)$$

We note that if we take the Fourier transform

$$\frac{dy}{2\pi} \int_{-\infty}^{+\infty} (\exp[i(y-jd-p)u]) du \quad (8)$$

and identify p with $\sum_{i=1}^j x_i$, the integral takes on the value one in the prescribed range of integration and is zero outside. The integrand is then multiplied by equation (8). The integration over the x 's will now be taken from minus infinity to plus infinity, where we will assume the inter-cell forces are large. If the inter-cell forces are large it will not be very probable that the j -th cell will be found far from its equilibrium position, hence our integration over all configuration space will involve no serious error. The error in allowing the integration to extend over regions inaccessible in configuration space to the physical system will be negligible since if the inter-cell forces are large the possibility of finding the system in these regions of configuration space is quite small.

Equation six now reads

$$P_j(y)dy = \frac{dy}{2\pi} \int_{-\infty}^{+\infty} \dots \int_{-\infty}^{+\infty} dx_1 \dots dx_j \int_{-\infty}^{+\infty} (\exp[iyu] - i \left[\sum_{i=1}^j x_i + jd \right] - [K/kT] \sum_{i=1}^j x_i) du \quad (9)$$

Let $B = K/kT$, then

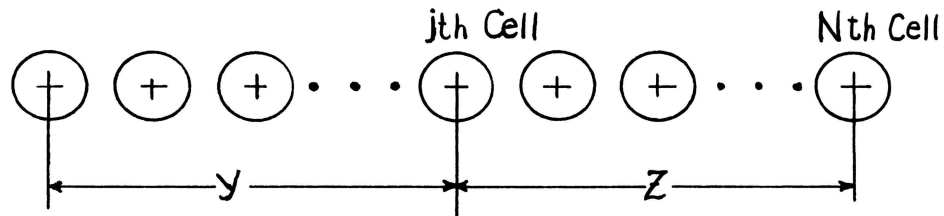
$$P_j(y)dy = \frac{dy}{2\pi} \int_{-\infty}^{+\infty} \dots \int_{-\infty}^{+\infty} (\exp-Bx_1^2)(\exp-ix_1)(\exp-Bx_2^2)(\exp-ix_2) \dots dx_1 \dots dx_j \int_{-\infty}^{+\infty} (\exp iu[y-jd]) du \quad (10)$$

After some reduction, one finds

$$P_j(y)dy = \frac{dy}{2\pi} \left[\frac{\pi}{B} \right]^{1/2} \left[\frac{4\pi B}{j} \right]^{1/2} (\exp-B[y-jd]^2/j) \quad (11)$$

Equation eleven is Gaussian about the mean position of the j-th cell. In the preceding development the j-th cell was the last in the array. We may generalize this restriction and allow the j-th cell to be any cell in the array and then calculate the type of probability distribution for the cell.

Figure 4.



(Linear model where z is fixed and where the N -th cell is free.)

In the above we shall first calculate the probability that the j -th cell be in dy at y with fixed z .

$$\int \int \dots \int (\exp[-B \sum_{i=1}^N x_i^2]) dx_1 \dots dx_N \quad (12)$$

Integral expresses the probability that the j -th cell is between y and $y + dy$ with z fixed. The integration is subject to the following conditions:

$$\sum_{i=1}^j x_i = y - jd = \nu = \text{a constant}$$

$$z = (N-j)d + \sum_{i=j+1}^N x_i = (1-y)$$

$$\int \dots \int \left(\exp -B \sum_{i=1}^j x_i^2 \right) dx_1 \dots dx_j \int \dots \int \left(\exp -B \sum_{i=j+1}^N x_i^2 \right) dx_{j+1} \dots dx_N \quad (13)$$

The first j integrations give

$$\left[\frac{\pi}{B} \right]^{j-1/2} \left[\frac{1}{j} \right]^{1/2} \cdot \int \left(\exp -B [y-jd]^2 / 2j \right) dy \quad (14)$$

To effect the remaining $(N-j)$ integrations, consider

$$\frac{dz}{2\pi} \int_{-\infty}^{+\infty} \left(\exp \left[i(z - [N-j]d - \rho)v \right] \right) dv = \begin{cases} 1 & z - \frac{dz}{2} < \rho < z + \frac{dz}{2} \\ 0 & \text{outside these limits} \end{cases} \quad (15)$$

and identify ρ with $\sum_{i=j+1}^N x_i$. Then the above, for the remaining $(N-j)$ integrations, is one over the prescribed range of

intergration and zero elsewhere. As in the preceding development we now write

$$\frac{dz}{2\pi} \int_{-\infty}^{+\infty} \dots \int_{-\infty}^{+\infty} \left(\exp -B \sum_{i=j+1}^N x_i^2 \right) dx \dots dx_N \left(\exp \left[i(z - [N-j] d) \right] \right) \times \left(\exp -i \sum_{i=j+1}^N x_i v \right) dv \quad (16)$$

After some reduction one finds for the remaining (N-j) integrations

$$\frac{dz}{2\pi} \left[\frac{\pi}{B} \right]^{(N-j)/2} \left[\frac{4\pi B}{N-j} \right]^{1/2} \left(\exp -B(z - [N-j]d)^2 / (N-j) \right) \quad (17)$$

The probability that the j-th cell be at y with fixed z is thus

$$dz \left[\frac{\pi}{B} \right]^{(N-2)/2} \left[\frac{1}{j(N-j)} \right]^{1/2} \left(\exp -B(y-jd)^2 / 2j \right) \times \left(\exp -B(z - [N-j] d)^2 / 2(N-j) \right) dy \quad (18)$$

The length z is obviously not fixed in the physical system we integrate equation (18) with respect to z between the limits of zero and infinity. We note again that if the

inter-cell forces are large substitution of the lower limit jR by zero will involve no serious error. Upon reduction we find

$$P_j(y)dy = \left[\frac{\pi}{B} \right]^{(N-1)/2} \left[\frac{1}{2j} \right]^{1/2} (\exp-B[y-jd]^2/2j)dy \quad (19)$$

This function is again Gaussian about the mean position of the j -th cell (relative probability) for the above equation has not been normalized. To recapitulate, $P_j(y)dy$ is the probability of locating the j -th cell between y and $y + dy$. The normalized form of equation (19) is

$$P_j(y)dy = \left[\frac{B}{\pi} \right]^{1/2} \left[\frac{1}{2j} \right]^{1/2} (\exp-B[y-jd]^2/2j)dy \quad (20)$$

To find the probability that a cell be in dy at y , we clearly must sum equation (20) over all distribution functions contributing to the probability at y . We thus find

$$P(y)dy = \sum_{j=1}^{y/A} P_j(y)dy \quad (21)$$

We now wish to find the limit of this sum as y approaches infinity in equation (20). The limit will be needed in the development of the X-ray scattering equations. We shall call the limiting value of $P(y)$, P_0 .

Equation (21) is integrated by allowing the finite sum of the increments to go to infinity while the increments approach zero.

$$P_0 = \left[\frac{B}{2\pi} \right]^{1/2} \int_0^{+\infty} \frac{(\exp - B[y-jd]^2/2j) dj}{j^{1/2}} \quad (22)$$

The cells are permitted to pass each other if the integral limits are taken from zero to infinity. For large values of y , the allowing of the cells to pass each other, and hence allowing all the cell to contribute to the probability of finding a cell at y is physically tenable. A large y , the contribution of the cells situated at small y is vanishingly small. Using the following substitution

$$j = \frac{2x^2}{Bd^2}$$

Integral (22) becomes

$$P_0 = 1/d \quad (23)$$

Recalling that d is the separation distance between any two adjacent cells centered around their equilibrium position, the derived P_0 is consistent with the notation that for large y there are $1/d$ particles per unit length.

Graphs five, six, seven, and eight display $P(y)$ for various values of d where the following parameters are used.

$$P(y)dy = dy \left[\frac{B}{2\pi} \right]^{1/2} \sum_{j=1}^{y/R} \frac{(\exp-B[y-jd]^2/2j)}{j^{1/2}} \quad (20)$$

$$d = 4R, 3R, 2R, R$$

$$k = 1.3 \times 10^{-16} \text{ ergs per degree per molecule.}$$

$$2\pi = 6.28$$

$$T = 300^\circ \text{ absolute}$$

$$K = 7 \times 10^{-14} \text{ dynes per cm.}$$

The value of K was chosen from the proportionality constant in the interaction of two argon atoms, where the proportionality constant is defined by the equation ($F = -Kx$). " x " is the relative separation of the two atoms, and is in the neighborhood of the separation corresponding to the minimum of their interaction potential energy.

It will be observed that after a certain distance, say y_c which corresponds to y_c/d cells the function is substantially constant or stable. This distance is known as the correlation distance and is denoted by the symbol r_c . The correlation distance defines a distance which is essentially a

FIGURE 5

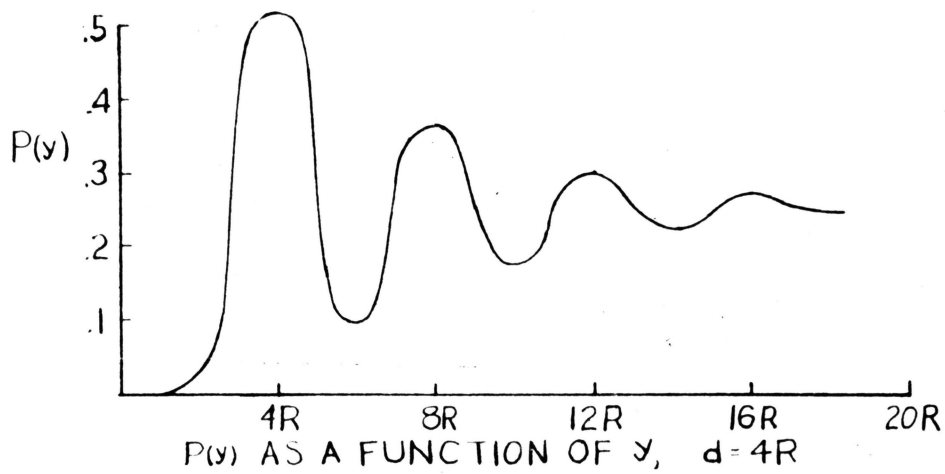


FIGURE 6

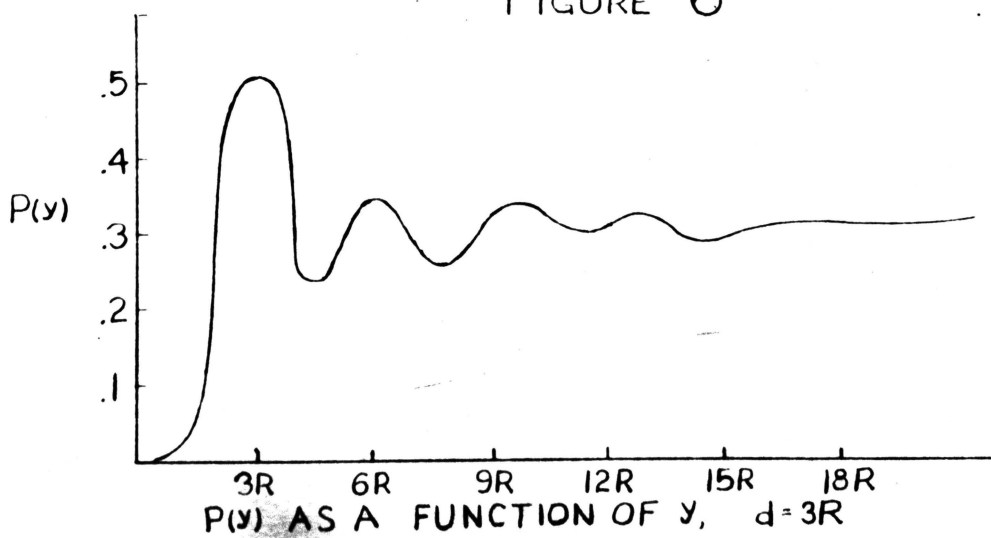


FIGURE 7

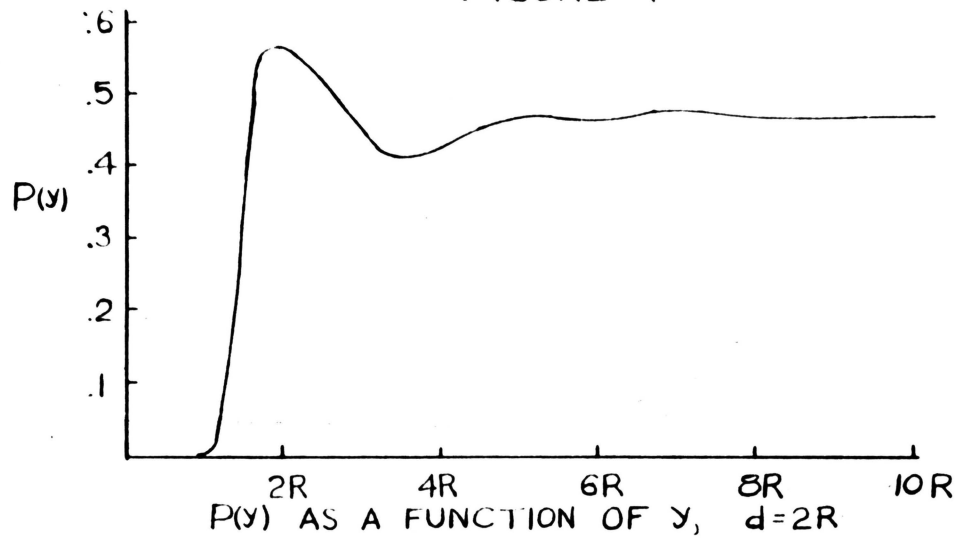
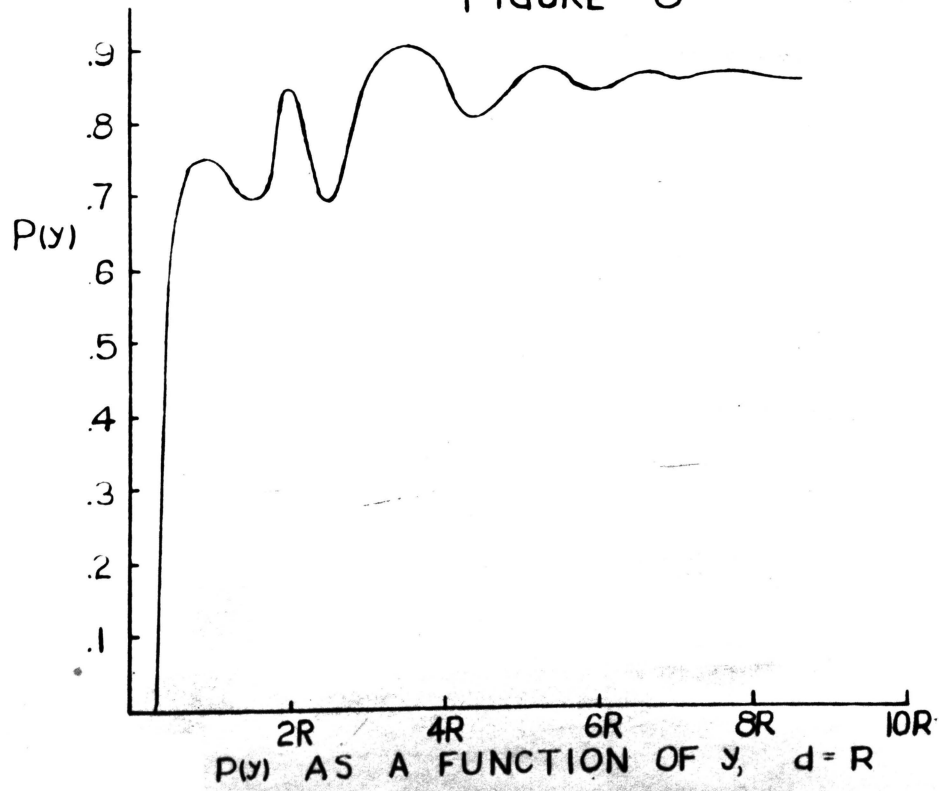


FIGURE 8



measure of the long range order of the structure, i.e. the linear polymer. The correlation distance is apparently a function of d and decreases for small values of d . This result is of prime importance for small angle X-ray scattering in determining the minimum number of cells needed to compute the X-ray intensity curves. The value of $P(y)$ after the correlation distance is approximately $1/d$, an error is introduced by using a finite number of cells in the summation for the determination of $P(y)dy$.

Debye's⁽⁴⁾ theory for X-ray scattering from a molecular gas leads to the following formula

$$I = \sum_{i=1}^n \sum_{k=1}^n f_i f_k (\sin ky_{ik}/ky_{ik}) \quad (24)$$

where

$$k = 4\pi (\sin \theta/2)/\lambda$$

I = the average intensity scattered by a molecule as a function of scattering angle

f_i = atom structure factor of the i -th atom in the molecule

n = number of atoms in the molecule

y_{ik} = distance between centers of the i -th and k -th atom

θ = angle between incident and scattered radiation

λ = wavelength of the X-rays

If we calculate the average intensity of the X-rays

(4) Debye, P. Ann. d. Physik, 46, 809, (1915)

scattered by a molecule, or here a linear macro-molecule i.e., the polymer of n identical spherical scattering regions, each sphere having a radius of $R/2$ and uniform electronic density, we are led to an equation analogous to equation (24), where f is not an atomic structure factor but is the so called X-ray scattering form factor for a sphere. The X-ray scattering form factor is defined by the following equation

$$\bar{f} = \frac{(\sin u) - u(\cos u)}{u^3} \quad (25)$$

where

$$u = 4\pi(R/2)(\sin \theta/2)/\lambda$$

and θ has been previously defined.

When "I" is summed over all possible combinations of i and k , the summation will of necessity include the summation of a particular cell with itself, remembering that the limit of $(\sin ky)/ky$ as $(y \rightarrow 0)$ is one. If there are N cells, we will then have

$$I(k) = N \bar{f}^2 \left[1 + \sum_{j=1}^N (\sin ky_j)/ky_j \right] \quad (26)$$

Replacing the discrete summation by an integration and integrating over y we have for the intensity of the X-rays

scattered by one of the linear polymers

$$I(k) = N \phi^2 \left[1 + \int_0^k [P(y) - P_0] \left[\frac{\sin ky}{ky} \right] dy \right] \quad (27)$$

Where $P(y)dy$ is the probability of finding a cell in an element of length dy at y from an average cell in the polymer, and P_0 is the asymptotic value of $P(y)$ for length y . Recalling that the expression for $P(y)$ derived earlier is

$$P(y) = \left[\frac{B}{2\pi} \right]^{1/2} \cdot \sum_{j=1}^{y/R} \frac{(\exp[-B[y-jd]^2]/2j)}{j^{1/2}} = \sum_{j=1}^{y/R} \theta(j,y) \quad (20)$$

we now can obtain an expression for the intensity of the X-rays scattered by a polymer by the introduction of the above expression for $P(y)$ into equation (27).

We begin by noticing that the number of terms in the sum for $P(y)$ depends on y . We then define $P(y)$ appropriately in the intervals $0 < y < R$, $R < y < 2R$, etc. as shown below:

$$\begin{array}{ll} P(y) = 0 & 0 < y < R \\ P(y) = \sum_{j=1}^1 \theta(j,y) & R < y < 2R \\ P(y) = \sum_{j=1}^2 \theta(j,y) & 2R < y < 3R \\ \dots & \dots \\ P(y) = \sum_{j=1}^{n-1} \theta(j,y) & (n-1)R < y < nR \end{array}$$

In the region $R < y < 2R$, the contribution to $P(y)$ is due to the first cell only. For region $2R < y < 3R$, the contribution to $P(y)$ is due to the first two cells, and in general for the region $(n-1)R < y < nR$, the first $(n-1)$ cells contribute to $P(y)$. This concept is made clearer by observing that for any particular length y , all the cells in the length y can also occupy the end position of y , therefore y/R cells must contribute to $P(y)$.

The values of $P(y)$ are then substituted in equation (27) and integrated piece-wise in the following manner.

$$\int_0^R \sum_{j=0}^{\cdot} \phi(j, y) Y dy + \int_R^{2R} \sum_{j=1}^{\cdot} \phi(j, y) Y dy + \dots + \int_{(n-1)R}^{nR} \sum_{j=1}^{(n-1)} \phi(j, y) Y dy \quad (28)$$

where $Y = (\sin ky)/ky$. The above series may be represented by the following summation

$$\sum_{n=1}^{\infty} \int_{(n-1)R}^{nR} \sum_{j=1}^{(n-1)} \phi(j, y) [(\sin ky)/ky] dy \quad (29)$$

Integration of equation (28) may be represented functionally by an equation of the form

$$\sum_{n=1}^{\infty} \left(\sum_1^n [G(k, nd) - G(k, [n-1]d)] \right) \quad (30)$$

Where $G(k,nd)$ and $G(k,[n-1]d)$ are the functional notations for the resulting integration of equation (28). The terms (nd) and $(n-1)d$ refer to the upper and lower limits of the integral. It will be observed that the upper limit of any integral is the same as the lower limit of the preceding integral and in general will cancel. The summation of equation (30) is then

$$\sum_{j=1}^n \int_{jR}^{nR} [F_j(\sin ky)/ky] dy = P(y) \quad (31)$$

where

$$F_j = \sum_{j=1}^{n-1} \phi(j,y)$$

Using the same schedule of limits as in equation (29) the integral series of $P(\sin ky)/ky$ dy may be represented by the following summation

$$-\sum_{j=1}^{\infty} P_0 \int_{jR}^{+\infty} [(\sin ky)/ky] dy - P_0 \int_0^R [(\sin ky)/ky] dy \quad (32)$$

Our intensity curve now becomes

$$I(k) = N \bar{\Phi}^2 \left[1 + \sum_{j=1}^{\infty} \int_{jR}^{\infty} (F_j - P_0) \cdot \frac{(\sin ky)}{ky} \cdot dy \right. \\ \left. - \int_0^R P_0 (\sin ky) / ky \, dy \right] \quad (33)$$

The above formula expresses the X-ray scattering intensity for a linear polymer where the separate contributions from each cell, in any prescribed interval, is computed by the summation of equation (20) when the appropriate values of j are substituted in equation (20).

Remembering that the contribution of cells past the r_c distance is negligible equation (33) then becomes:

$$I(k) = N \bar{\Phi}^2 \left[1 + \sum_{j=1}^{r_c/R} \int_{jR}^{r_c} (F_j - P_0) \left[\frac{(\sin ky)}{ky} \right] dy \right. \\ \left. - \int_0^R P_0 (\sin ky) / ky \, dy \right] \quad (34)$$

For the special case ($r_c = R$), we have

$$I(k) = N \cdot f^2 \left[1 - \frac{1}{d} \int_0^R \left[\frac{\sin ky}{ky} \right] dy \right] \quad (35)$$

The following graphs display X-ray scattering intensity as a function of r_c .

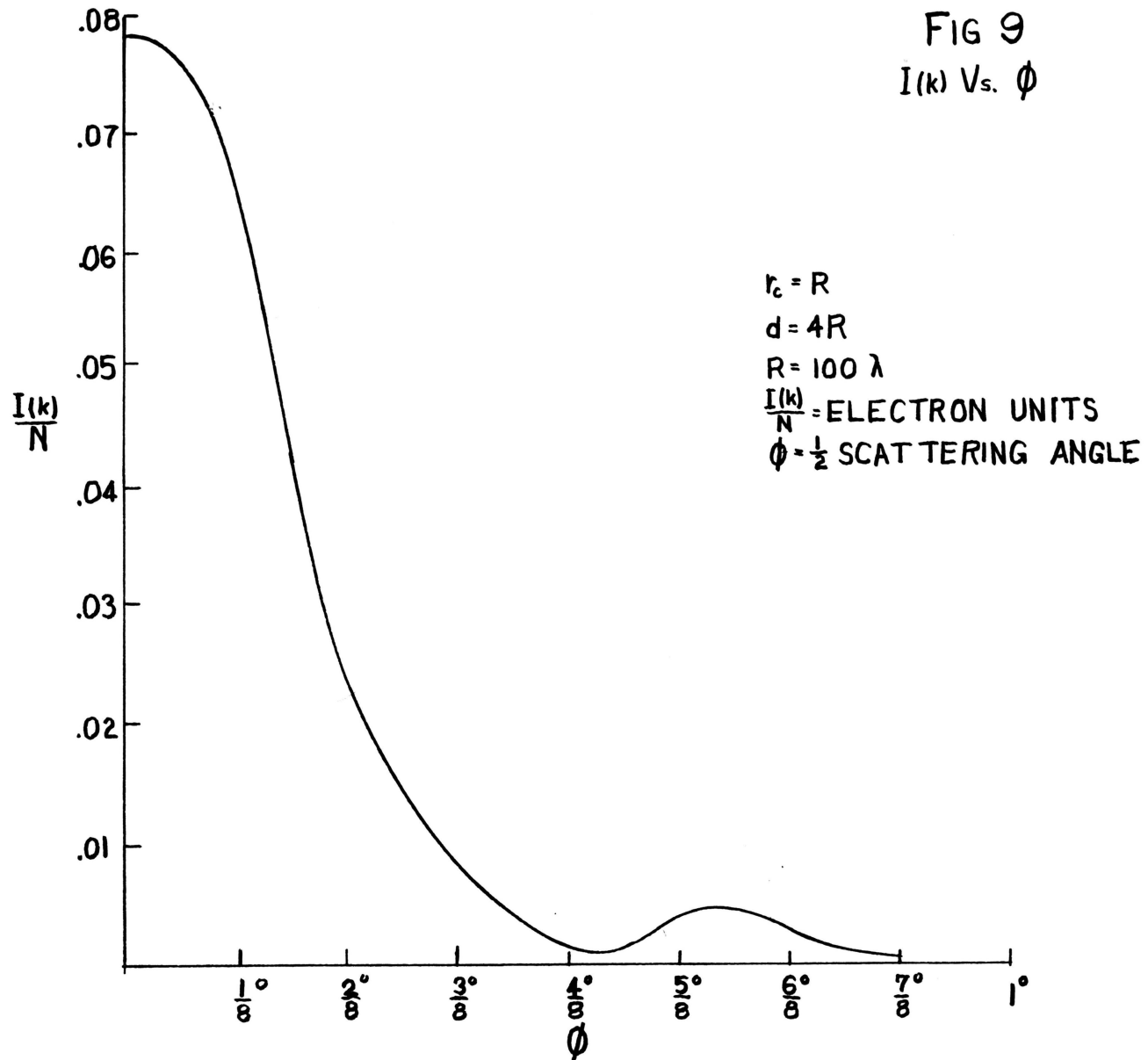
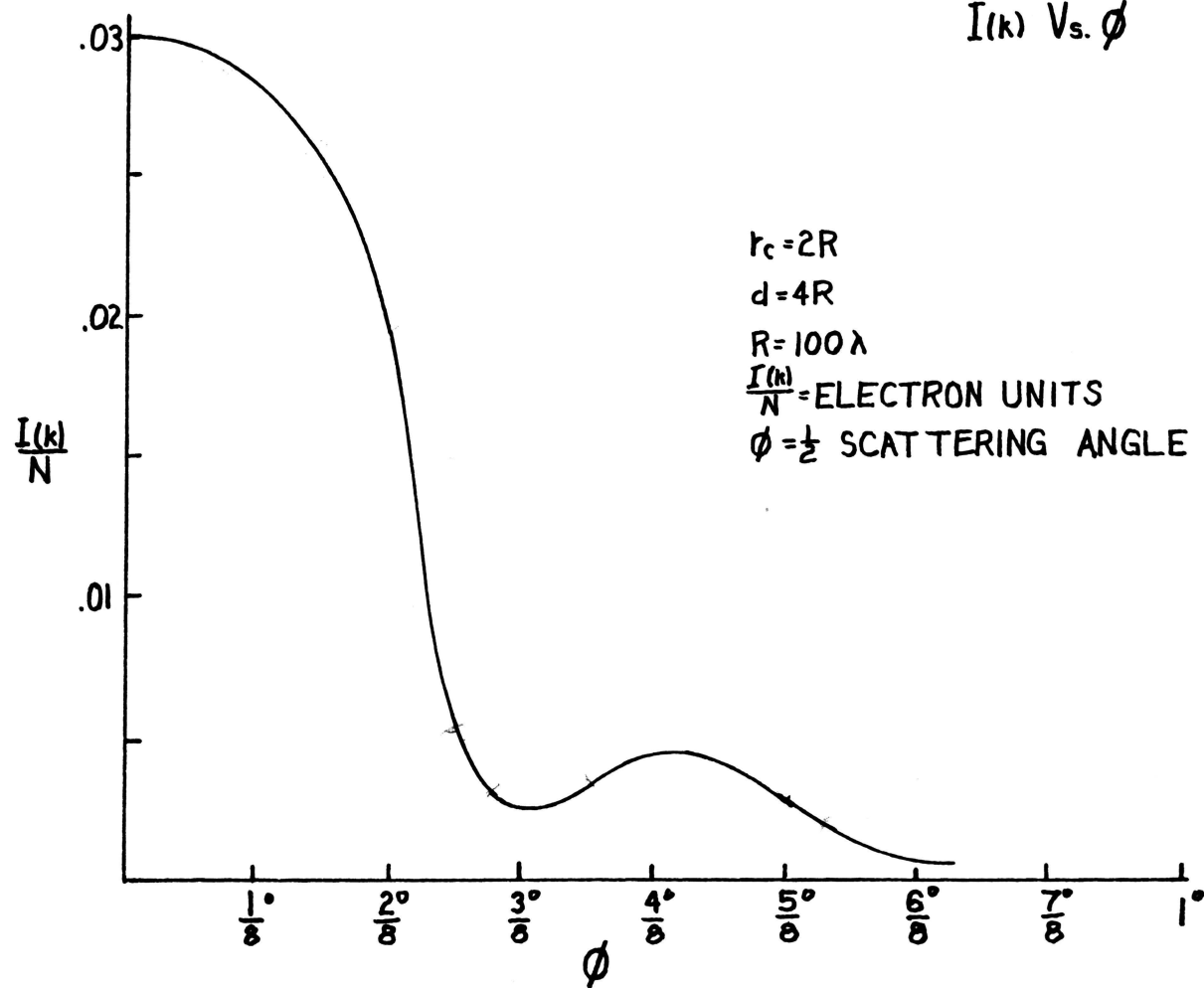


FIG. 10
 $I(k)$ Vs. ϕ



CONCLUSION

The polymer model has not been tested by physical experiments. Graphs five, six, seven, and eight display correlation distance as a function of cell spacing. From the graphs it may be concluded that the r_c distance approaches R for small cell spacing. The values of P_0 as read from the abscissa of graphs five, six, and seven is approximately $1/d$, being very close for ($d = 4R$). The error is assumed to be caused by summation over a finite number of cells.

Graphs nine and ten indicate that the X-ray scattering intensity decreases rapidly with scattering angle. The intensity being negligible at less than one degree. The first maximum occurring at zero degrees scattering angle. Intensity of the first maximum also decreases with increased correlation distance. The second maximum while small should be readily detectible by the experimenter.

When r_c is assumed equal to R the small angle X-ray scattering patterns are of the same general form as Kratky's. For large values of r_c the more general X-ray scattering intensity formula must be used. Construction of the F_j function as shown in the preceding section permits the evaluation of the intensity function.

The general X-ray scattering intensity function is temperature dependent. For the special case of ($r_c = R$) the X-ray scattering function is independent of temperature.

This result is similar to Kratky's scattering formula.

The X-ray scattering intensity decreases very rapidly as the scattering angle increases. With the parameters used in graphs nine and ten, the intensity is negligible at a scattering angle of two degrees.

SUMMARY

A stiff linear polymer suitable for X-ray scattering analysis has been developed. The model takes the chemical-structure unit, as a unit or cell and assumes the displacements of the cells are small enough that the force between two adjacent structural units is proportional to their separation distance.

The probability function which characterizes the X-ray scattering by a linear polymer has been found. Graphs nine and ten display $I(k)$ as a function of the scattering angle.

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