AFWAL-TR-86-4143

CRYSTALLINE COLLOIDAL ARRAY FILTERS: THEORY AND DESIGN

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Interim Technical Report for Period June 1985 -- October 1986

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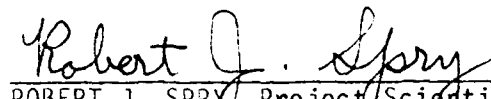AIR FORCE SYSTEMS COMMAND
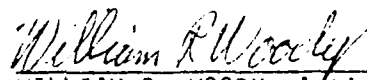WRIGHT-PATTERSON AIR FORCE BASE, OHIO 45433-6533

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# REPORT DOCUMENTATION PAGE

| 1a REPORT SECURITY CLASSIFICATION UNCLASSIFIED | | | 1b RESTRICTIVE MARKINGS |
|---|---|---|---|
| 2a SECURITY CLASSIFICATION AUTHORITY | | | 3 DISTRIBUTION/AVAILABILITY OF REPORT Approved for public release; distribution unlimited. |
| 2b DECLASSIFICATION/DOWNGRADING SCHEDULE | | | |
| 4. PERFORMING ORGANIZATION REPORT NUMBER(S) AFWAL-TR-86-4143 | | | 5 MONITORING ORGANIZATION REPORT NUMBER(S) |
| 6a. NAME OF PERFORMING ORGANIZATION Laser Hardened Materials Branch Electromagnetic Materials Division | 6b OFFICE SYMBOL (If applicable) AFWAL/MLPJ | | 7a NAME OF MONITORING ORGANIZATION |
| 6c. ADDRESS (City, State, and ZIP Code) Wright-Patterson Air Force Base, OH 45433-6533 | | | 7b ADDRESS (City, State, and ZIP Code) |
| 8a NAME OF FUNDING/SPONSORING ORGANIZATION | 8b OFFICE SYMBOL (If applicable) | | 9 PROCUREMENT INSTRUMENT IDENTIFICATION NUMBER |

| 8c ADDRESS (City, State, and ZIP Code) | 10 SOURCE OF FUNDING NUMBERS | | | |
|---|---|---|---|---|
| | PROGRAM ELEMENT NO 62102F | PROJECT NO 2422 | TASK NO 04 | WORK UNIT ACCESSION NO 01 |

**11 TITLE (Include Security Classification)**
Crystalline Colloidal Array Filters: Theory and Design

**12 PERSONAL AUTHOR(S)**
Robert J. Spry and David J. Kosan

| 13a TYPE OF REPORT Interim | 13b. TIME COVERED FROM 85 Jun TO 86 Oct | 14. DATE OF REPORT (Year, Month, Day) 1987 January | 15 PAGE COUNT 29 |
|---|---|---|---|

**16 SUPPLEMENTARY NOTATION**

| 17 COSATI CODES | | | 18 SUBJECT TERMS (Continue on reverse if necessary and identify by block number) |
|---|---|---|---|
| FIELD | GROUP | SUB GROUP | Optical filters; dynamical X-ray diffraction; Bragg diffraction; light scattering; Rayleigh-Gans theory; colloidal crystals. |
| 20 | 06 | | |

19 ABSTRACT (Continue on reverse if necessary and identify by block number) The colloidal array filter consists of an aqueous suspension of polystyrene spheres arranged in a crystalline lattice, thereby producing Bragg diffraction of incident light. Recent measurements have shown that a rejection filter incorporating a thin layer of the colloidal suspension has a moderately narrow bandwidth and unusually large peak absorbance. We have successfully explained these results using both dynamical X-ray diffraction theory and optical scattering theory. The theoretical bandwidth is in good agreement with the experimental value, while the theoretical attenuation results are consistent with measured transmittance values. The theoretical attenuation function indicates that actual filters may be constructed that are thinner and more efficient than the first experimental ones. The theoretical angle shift of the rejection band maximum has also been calculated, and is in excellent agreement with experimental data.

| 20 DISTRIBUTION/AVAILABILITY OF ABSTRACT [ ] UNCLASSIFIED/UNLIMITED [X] SAME AS RPT [ ] DTIC USERS | 21 ABSTRACT SECURITY CLASSIFICATION UNCLASSIFIED | |
|---|---|---|
| 22a NAME OF RESPONSIBLE INDIVIDUAL Robert J. Spry | 22b TELEPHONE (Include Area Code) (513) 255-6671 | 22c OFFICE SYMBOL AFWAL/MLPJ |

**DD FORM 1473, 84 MAR**   83 APR edition may be used until exhausted
All other editions are obsolete

## FOREWORD

This report describes an in-house study conducted by Robert J. Spry
and David J. Kosan of the Laser Hardened Materials Branch, Electromagnetic
Materials Division, Materials Laboratory, Air Force Wright Aeronautical
Laboratories, Wright-Patterson Air Force Base, OH 45433 under Project 2422,
Task No. 242204, Work Unit 24220401. The work reported herein was
performed during the period June 1985 through October 1986 by the authors.
The report was released on 16 October 1986.

The authors wish to thank Dr. Wade Adams of the Air Force Wright
Aeronautical Laboratories/Materials Laboratory, and Drs. Dan McLean and
Richard Sutherland of Scientific Applications International Corporation for
many beneficial discussions.

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# SECTION I

## INTRODUCTION

Colloidal suspensions of small particles in electrolytes are known to form crystalline arrays. One of the systems receiving the greatest attention is that of monodisperse latexes, or charged polystyrene spheres *in water and other solvents* (References 1-3). These systems arrange themselves in body-centered cubic (bcc) (Figure A-1) or face-centered cubic (fcc) structures (Figure A-2) depending upon the particle concentration (Reference 4). These crystalline patterns are commonly discerned through ordinary optical microscopy, as illustrated in Figure 1. There is also evidence for the existence of *hexagonal close-packed (hcp) structures* (Figure A-1) under certain conditions (Reference 5).

These arrays are sufficiently well-ordered to produce Bragg diffraction of light in the optical region (References 5 and 6). The measured performance of a very high optical density rejection filter made from a crystalline colloidal array of polystyrene spheres has recently been reported (Reference 7). The major filter data from this reference are summarized in the following table:
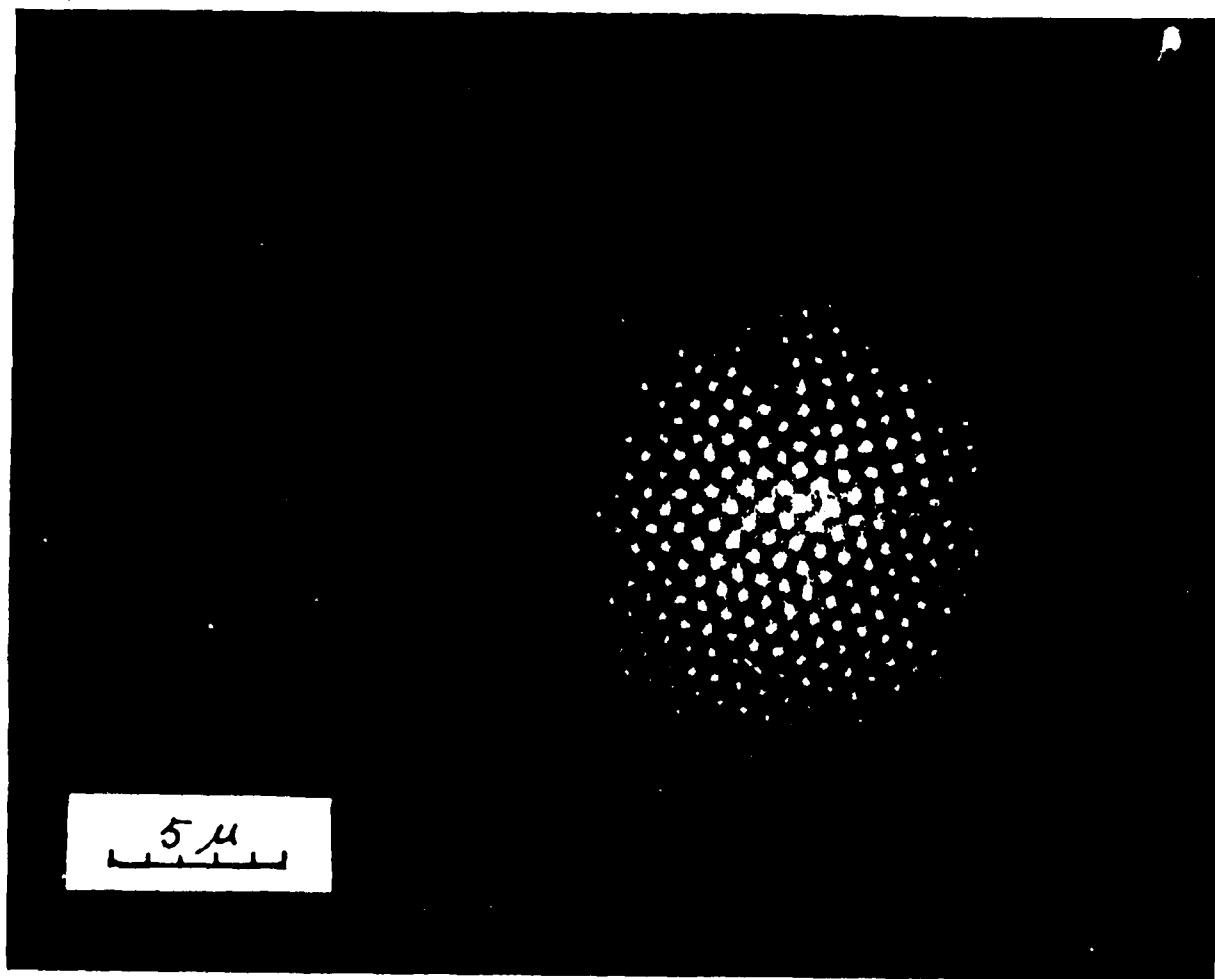
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Figure 1.  Coexistance of Hexagonal and Cubic Structures,
with a Stacking Fault between the Two Phases.
Particle Diameter 3410 A, Volume Fraction 8
Per Cent.  (After Reference 1).

## TABLE I

#### Performance Data of the Filter in Reference 7

- Minimum Transmittance                              $< 0.01\%$

- Out of Band Transmittance                          $>\ 90\%$

- Bandwidth (F.W.H.M.)                               $<\ 12$ nm

- Wavelength Shift for $10^{\circ}$ Rotation         $-4.4$ nm $\pm\, 0.3$ nm  *

 * (Obtained from Figure 2 of Reference 7)


We have successfully explained the performance data of this new

rejection filter using dynamical X-ray diffraction theory (References 8 and 9)

and optical scattering theory (Reference 10), as well as simple considerations

from thin film optics (Reference 11 and 12). Extensive expressions are

derived for the bandwidth, and attenuation of the filter, in which case

very large absorbance values are predicted. Some of our work in shortened

form has been previously presented (References 13 and 14).

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# SECTION II

## CRYSTAL PARAMETERS

The crystalline colloidal array filter of Flaugh et al. (Reference 7) was made with a rejection wavelength maximum of 514.5 nm. We shall designate this wavelength maximum in air as $\lambda_o$, which is related to the distance $\underline{d}$ between the planes of the crystalline array by the Bragg condition

$$M \lambda_o = 2nd \sin \theta \quad . \tag{1}$$

Here, $\underline{M}$ is the diffraction order, $\underline{n}$ is the refractive index of the colloidal suspension, and $\underline{\theta}$ is the usual Bragg angle <u>within</u> the suspension. The geometric and parametric relationships for the incident and diffracted light rays are depicted in Figure 2. Taking the refractive index to be equal to 1.333, that of water, we obtain $\underline{d}$ = 193.0 nm, for first-order diffraction. Flaugh et al. state that the filter crystal structure was face-centered cubic, and the filter planes are therefore (111) planes. The fcc unit cell is illustrated in Figure 3, from which we may evaluate the lattice parameter as $\underline{a} = \underline{d}\sqrt{3}$, or 334.3 nm. Our values for $\underline{d}$ and $\underline{a}$ do not agree with those of Flaugh et al. because they neglected the refractive index of the liquid, and in the case of $\underline{a}$, they seem to have a typographical error.

The diameter $\underline{D}_0$ of the polystyrene spheres used by Flaugh et al. was 91 nm. The volume fraction $\underline{\phi}$ occupied by the spheres is therefore
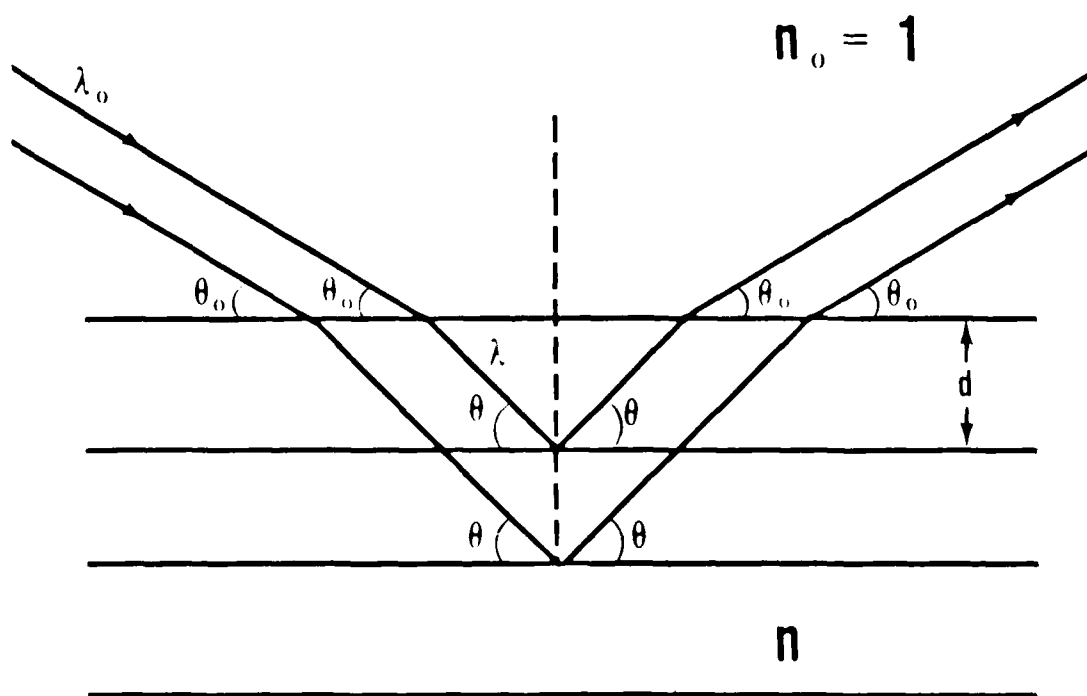
Figure 2. Geometric and Parametric Relationships for the
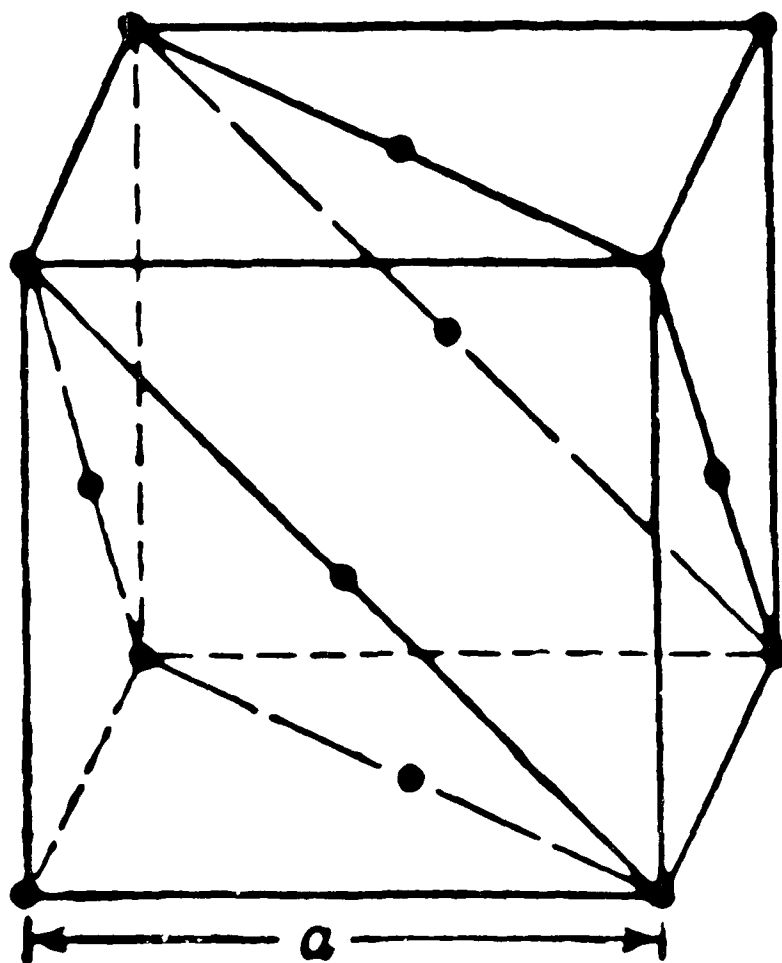Incident and Diffracted Light Rays.

Figure 3. A Pair of Parallel Adjacent (111) Planes
(Separation "d") in a fcc Lattice.

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$$\phi = \frac{2\pi}{3} \left(\frac{D_o}{a}\right)^3 \qquad , \qquad (2)$$

or 4.22 percent.

As found experimentally by Hiltner et al. (References 2 and 6), the refractive index varies according to

$$n = 1.333 + 0.27\phi \qquad (3)$$

with the result that $\underline{n}$ = 1.344, for the present case. Thus, there is only a slight error in using the refractive index of water in calculating $\underline{d}$ and $\underline{a}$.

Alternatively, one may combine Eqs.(1-3) and solve the resulting cubic equation for $\underline{d}$, yielding $\underline{d}$ = 191.3 nm. This is close enough to the result of the simplified calculation for $\underline{d}$, that our first result is sufficient for use in the rest of our calculations.

Another parameter we shall need is $\underline{D}$, the next nearest neighbor spacing (Figure 4). This is obtained by $\underline{D} = \underline{a}/\sqrt{2}$, or 236.4 nm. Also, the colloidal array thickness was $3.97 \times 10^5$ nm, equivalent to 2057 (111) planes (Figure 5).
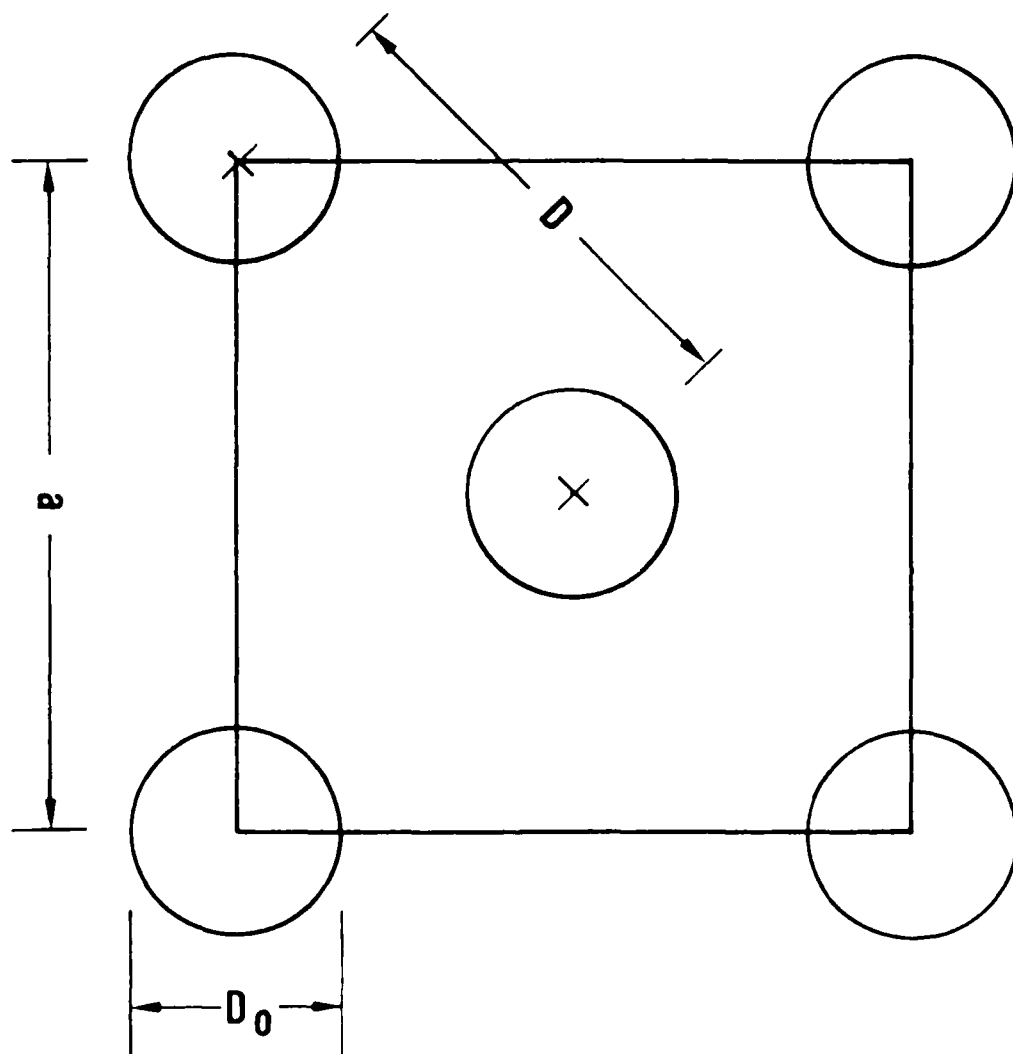
Figure 4. Details of the (100) Face in a fcc Colloidal Lattice.
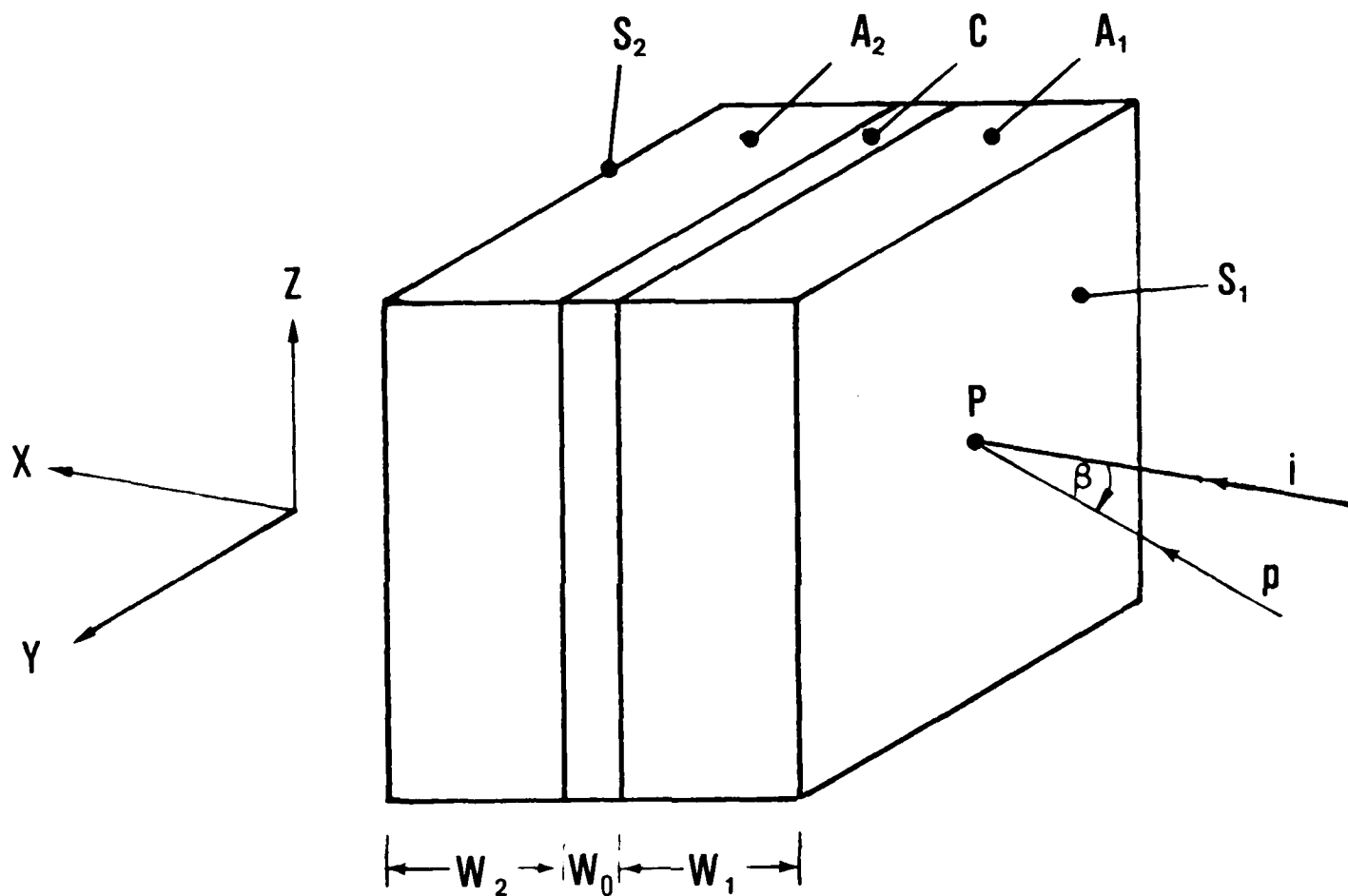
Figure 5.    Macroscopic Arrangement of the Colloidal
             Array Filter.  $A_1$ and $A_2$ are Plexiglass
             Plates; C is the Colloidal Array Held in
             Place by a Teflon Spacer.

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# SECTION III

## ANGLE SHIFT OF THE REJECTION BAND MAXIMUM

The angle shift of the reflection band maximum is shown in Figure 6. If we designate $\delta\lambda_o$ to be the wavelength shift of the rejection band maximum upon rotation of the filter by an angle $\beta$ relative to the incident radiation (See also Figure 5), application of Snell's Law produces the following relation between $\delta\lambda_o$ and known quantities:

$$\frac{\delta\lambda_o}{\lambda_o} = \sqrt{1-\left(\frac{\sin\beta}{n}\right)^2} - 1 \quad . \tag{4}$$

For small values of $\beta$, this expression reduces to

$$\frac{\delta\lambda_o}{\lambda_o} \approx - \frac{\beta^2}{2n^2} \quad , \tag{5}$$

a result well-known in interference filter theory (References 11 and 12).

Flaugh et al. state that $\delta\lambda_o$ is 506.7 nm minus 514.5 nm, or -7.8 nm, for an angle of incidence of $10^0$. However, careful measurement of the data from their Fig. 2 yields a value of $\delta\lambda_o = -4.4 \pm 0.3$ nm, where the error indicated is the precision in reading $\delta\lambda_o$ from their figure. If we use $\underline{n} = 1.344$, we calculate $\delta\lambda_o$ to be -4.3 nm using Eq. 4, consistent with the value we measure from their data. We believe the stated wavelength shift of Flaugh et al. to be in error because it deviates greatly from the theoretical value.
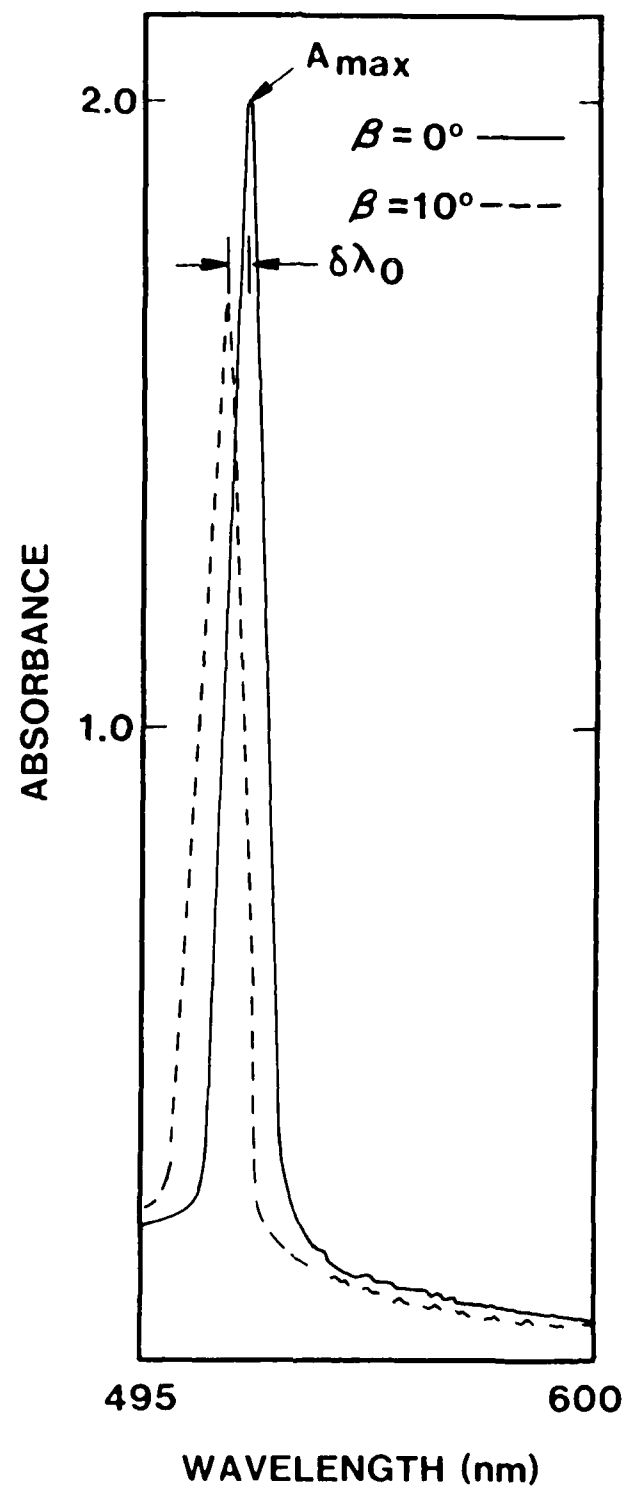
Figure 6.    Absorption Spectrum of the Colloidal Filter
at $\beta=0^{\circ}$ and $\beta=10^{\circ}$ (After Reference 7).

# SECTION IV

## BANDWIDTH

By bandwidth, we shall use the definition of the full width of the linear transmission band at one-half the maximum intensity (F.W.H.M.) which we shall label $\Delta\lambda_o$. To calculate the bandwidth, we must employ the dynamical theory of X-ray diffraction (References 8 and 9). The necessity for this approach is readily seen by referring to Figure 7. The incident beam of intensity $I_0$ loses a small fraction of its energy by reflection from each successive lattice plane. In addition, the initially reflected beam is reflected a second time to a direction parallel to the incident beam. These effects are neglected in the kinematic theory of x-ray diffraction usually used by crystallographers to determine the structure of matter.

We shall make use of the development of this theory by Ewald as later expounded by Zachariasen (Reference 8). The starting point shall be his Eq. (3.93) relating the dielectric constant $\epsilon(\vec{r})$ and the polarizability per unit volume $\alpha(\vec{r})$, both a function of the lattice position vector $\vec{r}$:

$$\epsilon(\vec{r}) = 1 + 4\pi\alpha(\vec{r}) \qquad .$$

(6)

We may further express $\alpha(\vec{r})$ as the product of $\alpha_s$, the polarizability of a single sphere, and $N(\vec{r})$, the sphere spatial distribution. The sphere polarizability is:

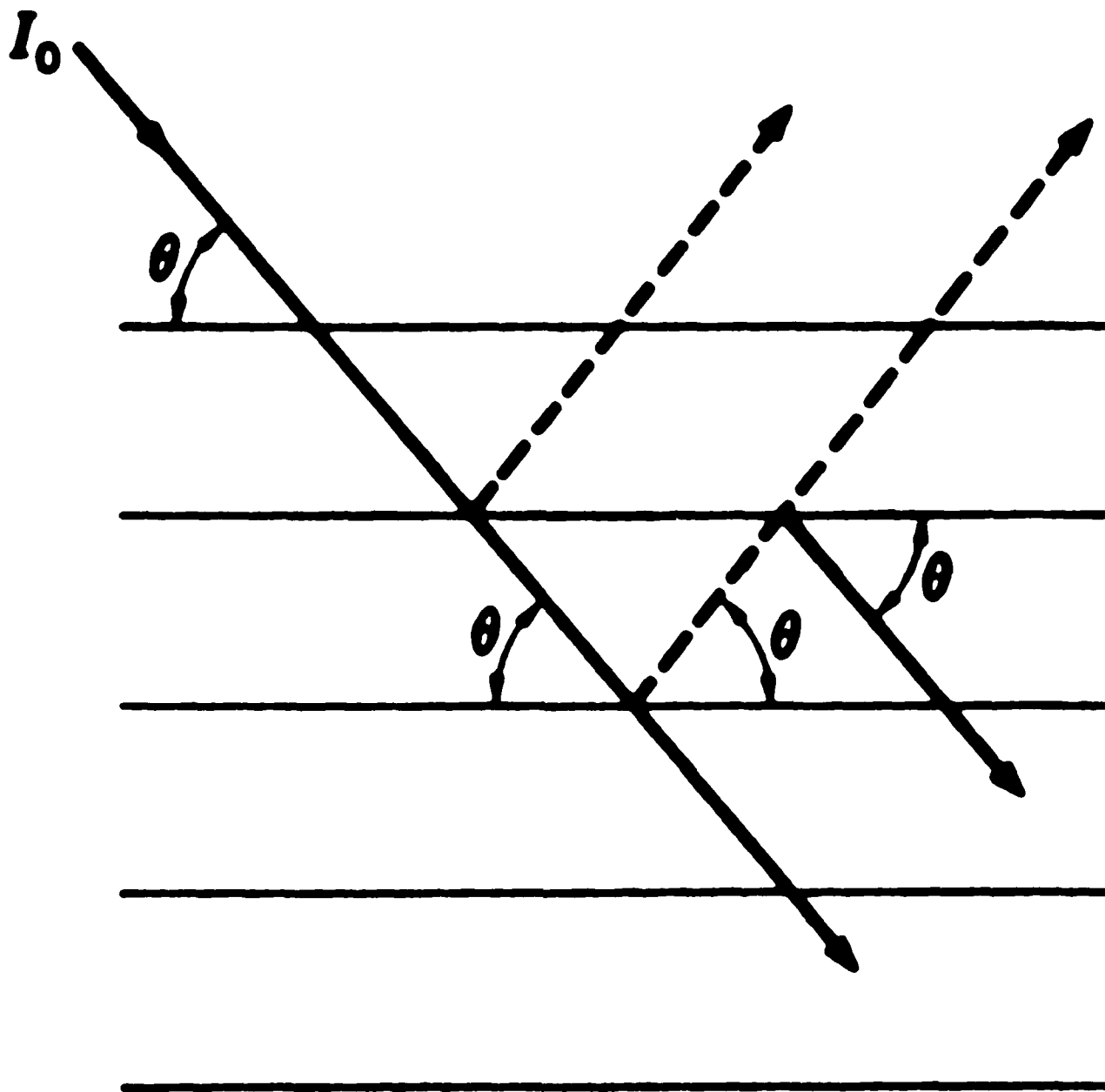$$\alpha_s = \frac{3(m^2-1)}{4\pi(m^2+2)} V_s \qquad ,$$

(7)

Figure 7. Attenuation of the Incident Beam by Reflection from Successive Planes within the Lattice (After Reference 9).

where $\underline{m}$ is the ratio of the refractive index of the spheres to that of the surrounding liquid medium, and $\underline{V_s}$ is the volume of a sphere (Reference 10). The quantity $\underline{4\pi\alpha(\vec{r})}$ may be replaced by a new function $\underline{\Psi(\vec{r})}$, which, being periodic, can be expanded as a Fourier series:

$$\Psi(\vec{r}) = 4\pi\alpha(\vec{r}) = \sum_H \Psi_H \, e^{-i\left(2\pi\vec{B}_H \cdot \vec{r}\right)} \quad . \tag{8}$$

In this equation $\underline{\vec{B}_H}$ is the reciprocal lattice vector satisfying the Laue equation

$$\vec{B}_H = \vec{k}_1 - \vec{k}_2 \quad , \tag{9}$$

and $\vec{k}_1$ and $\vec{k}_2$ are the incident and scattered wave vectors. It can then be shown that

$$\Psi_H = \frac{4\pi\alpha_s F_H}{V} \quad , \tag{10}$$

where $\underline{F_H}$ is the crystal structure factor when the Laue equation is satisfied, and $\underline{V}$ is the volume of a crystalline unit cell, equal to $a^3$ for the fcc lattice.

For waves diffracted off (111) planes in the fcc lattice, $\underline{F_H}$ is evaluated to be $\underline{4f}$, $\underline{f}$ being the scattering factor for a sphere. To evaluate $\underline{f}$, we make use of the scattering theory of small spheres developed by Van De Hulst (Reference 10). With the definition

$$x \equiv \frac{\pi D_o}{\lambda} \quad , \tag{11}$$

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the condition for ordinary Rayleigh scattering is (Reference 10)

$$x \ll 1 \quad , \tag{12}$$

which is true of Region 61 of Figure 8 and Table II. If Eq. (11) does not hold, some researchers believe that Mie scattering theory must be immediately utilized. However, in Region 1, one is yet able to use a modified Rayleigh theory, known as Rayleigh-Gans theory, provided

$$x(m-1) \ll 1 \quad . \tag{13}$$

To further understand the application of the above conditions to the case of the colloidal array filter, we have plotted x(m-1) and x vs. Do/d in Figure 9. For our case, Do/d is 0.472 and x is 0.73, which would be too large for ordinary Rayleigh scattering theory. Additional justification for using Rayleigh-Gans theory may be found in Reference 10, Sections 18.4 and 19.12.

Continuing to follow the development of Van De Hulst, we obtain

$$f = G(u) = \frac{3}{u^3}(\sin u - u \cos u) \quad , \tag{14}$$

where

$$u = \frac{2\pi D_o}{\lambda} \sin \theta \quad . \tag{15}$$

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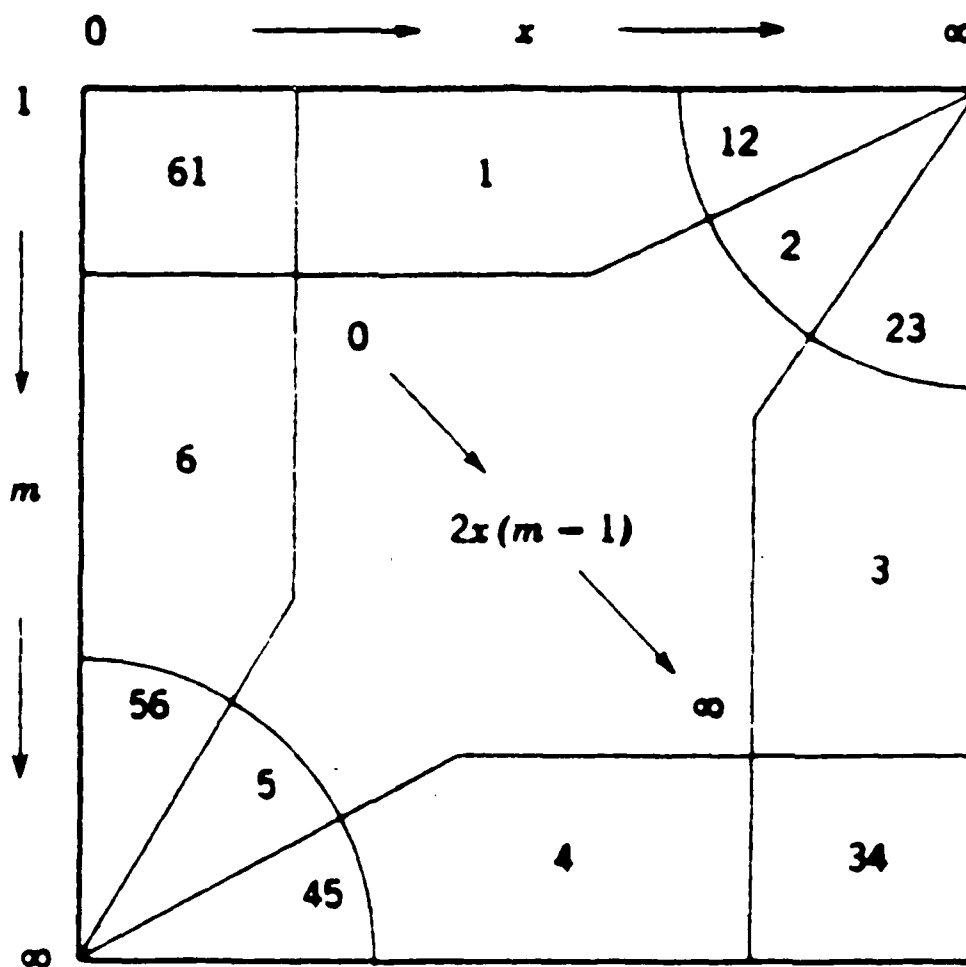
Figure 8. Regions of Validity of Various Scattering
Theories in the m-x Domain (After
Reference 10).

TABLE II

Boundary Regions of the m-x Domain (After Reference 10)

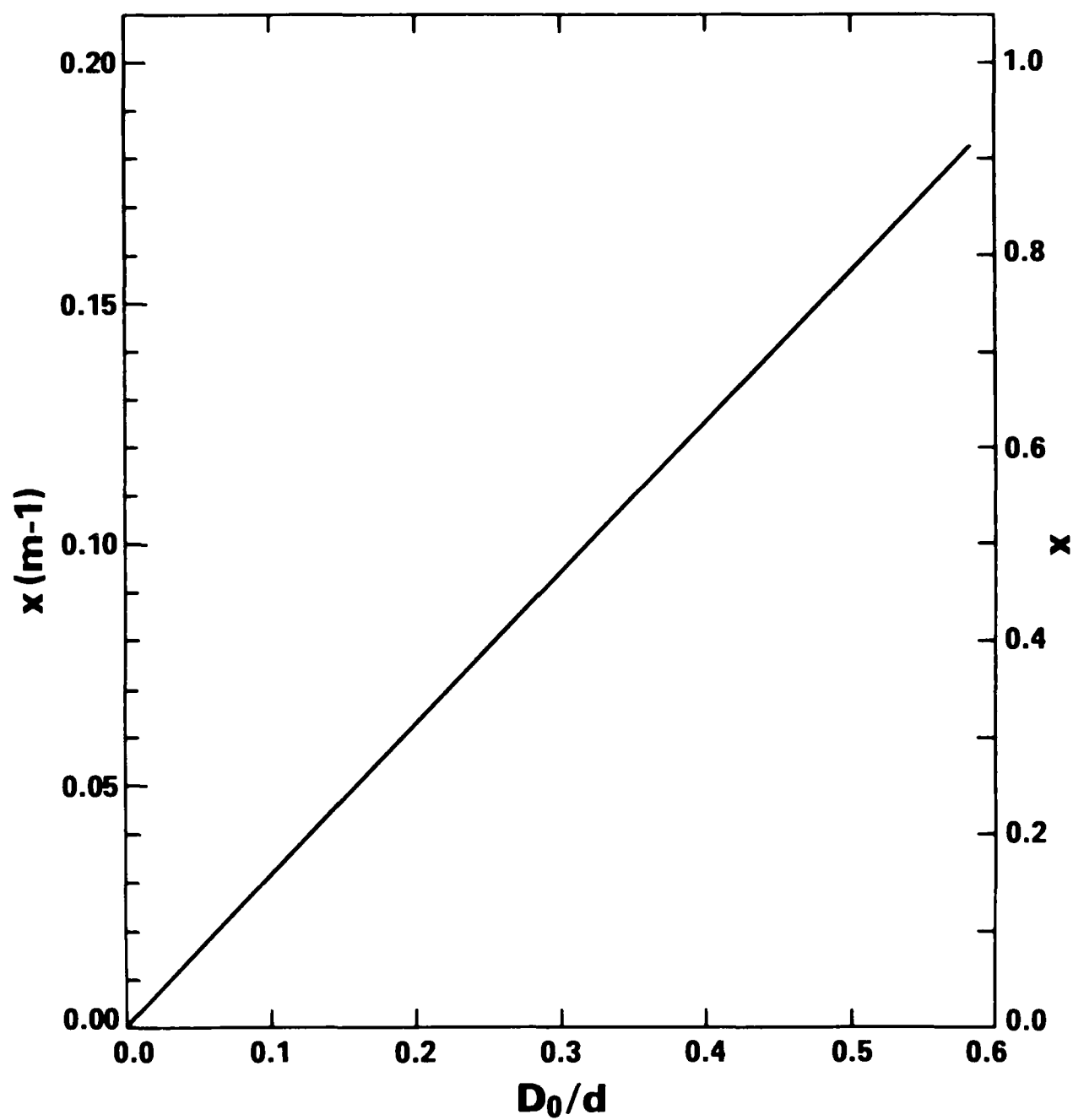| Region | $x$ | $m-1$ | $x(m-1)$ | Chapter or Section | Extinction Formula |
|---|---|---|---|---|---|
| 61 | e | e | e | | $Q = (32/27)(m-1)^2 x^4$ |
| 1 | arb | e | e | 7.2 (Rayleigh-Gans) | |
| 12 | 1 | e | e | | $Q = 2(m-1)^2 x^2$ |
| 2 | 1 | e | arb | 11 (anomalous diffraction) | |
| 23 | 1 | e | 1 | | $Q = 2$ |
| 3 | 1 | arb | 1 | 12 (large spheres) | |
| 34 | 1 | 1 | 1 | | $Q = 2$ |
| 4 | arb | 1 | 1 | 10.6 (total reflector) | |
| 45 | e | 1 | 1 | | $Q = (10/3)x^4$ |
| 5 | e | 1 | arb | 10.5 (optical resonance) | |
| 56 | e | 1 | e | | $Q = (8/3)x^4$ |
| 6 | e | arb | e | 6.3 (Rayleigh scattering) | |

Figure 9.  x(m-1) and x vs. the Ratio of Sphere
           Diameter to Diffraction Plane Spacing.

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In this equation, $\lambda$ is the wavelength of light in the colloidal suspension, equal to $\lambda_0/n$. Note that $\theta$, the Bragg angle, is one-half the "scattering angle" usually defined in optical scattering (Reference 10). Combining Eqs. 1, 7, 10, 14, and 15, we find

$$\Psi_H = -\frac{2}{\pi^2 (3)^{3/2}} (m^2 - 1) \left(\frac{3}{m^2 + 2}\right) \left(\sin u - u \cos u\right) \quad (16)$$

where

$$u = \pi \sqrt{3/2} \left(\frac{D_0}{D}\right) \quad . \quad (17)$$

Zachariesen (Reference 8) has shown that for a thick crystal and no absorption, the bandwidth is

$$\left(\Delta \lambda_0\right)' = \frac{w_y \left| K \Psi_H \right| \lambda_0}{\sqrt{b} \, \sin^2 \theta} \quad , \quad (18)$$

where $w_y$ is a numerical factor $2/3 \sqrt{3} = 1.155$ for Ewald theory and $b$ is the ratio of direction cosines of the light rays, which is unity for Bragg diffraction. The polarization factor $K$ is unity for $\sigma$ polarization and $|\cos 2\theta|$ for $\pi$ polarization. The correctness of using $\lambda_0$ instead of $\lambda$ in this equation is readily ascertainable from Eq. 1, and the expression of Zachariesen for $\Delta\theta$, the angular bandwidth. Inserting Eq. 16 into Eq. 18 yields

$$\left(\Delta \lambda_0\right)' = \frac{8}{9 \pi^2} K \left(\frac{\lambda_0}{2}\right) \left(m^2 - 1\right) \left(\frac{3}{m^2 + 2}\right) \frac{\left(\sin u - u \cos u\right)}{\sin^2 \theta} \quad . \quad (19)$$

This expression resembles the bandwidth $\Delta \theta$ on the glancing angle scale derived by other authors (References 2, 6, and 15), but with some important differences. The $\lambda_0/2$ and $\sin^2 \theta$ terms are unique to $(\Delta \lambda_0)'$, and

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the previous derivations have not considered polarization. Hiltner et al. (References 2 and 6), used a value of $w_y$ = 3/4 $\sqrt{2}$ = 1.061, following the Darwin theory of dynamical diffraction (Reference 8). They also used the approximation in the polarizibility that $(m^2 + 2) \approx 3$. Melone and Rustichelli (Reference 15) used the approximations $f$ = 1 and $w_y$ = 1.

Eq. 19 is accurate for the bandwidth if the colloidal-air interface is neglected. A correction based on Snell's law must be included for the bandwidth measured in air. If $n_0$ is the refractive index of air and $\theta_0$ is the glancing angle in air, it can be shown that the corrected bandwidth is

$$\Delta\lambda_o = \frac{8}{9\pi^2} K\left(\frac{\lambda_o}{2}\right)\left(m^2-1\right)\left(\frac{3}{m^2+2}\right)\left(\frac{n}{n_o}\right)\frac{(\sin u - u\cos u)}{\sin\theta \ \sin\theta_o} \quad (20)$$

where

$$\sin\theta = \left[1 - \left(\frac{n_o}{n}\cos\theta_o\right)^2\right]^{1/2} \quad . \quad (21)$$

Using $m$ = 1.60/1.333 and other values previously stated, we calculate $\Delta\lambda_o$ = 10.3 nm. This is in excellent agreement with the $<$ 12 nm bandwidth claimed by Flaugh et al. (Reference 7). Reasons for lack of closer agreement between theory and the measured value may include absorption, crystalline mosaicity, lattice distortions, and stacking faults.

It is appropriate to consider how to design a filter with a tailorable bandwidth. From Eq. 20, we can see that there is some freedom in choosing the refractive index of the spherical particles or of the host medium. The number of materials for the spheres is quite limited, but

there are several liquids known to support crystalline structures (Reference 2). However, the refractive indices of all these liquids are greater than that of water, which would only broaden a filter rejection band. We are thus led to consider the effect of smaller particle diameters upon the filter bandwidth. We shall define

$$H(u) \cong (\sin u - u \cos u) \qquad (22)$$

and plot this as a function of $\underline{Do/d}$ in Figure 10. This graph demonstrates that a narrower bandwidth is most easily obtained by using smaller diameter polystyrene spheres.

For the crystalline colloidal array filter of Flaugh et al. (Reference 7), $\underline{u}$ was 1.48, corresponding to $\underline{H(u)} = 0.864$. In this region, it can be readily seen from Figure 10 that the cubic approximation for $\underline{H(u)}$ should not be used.
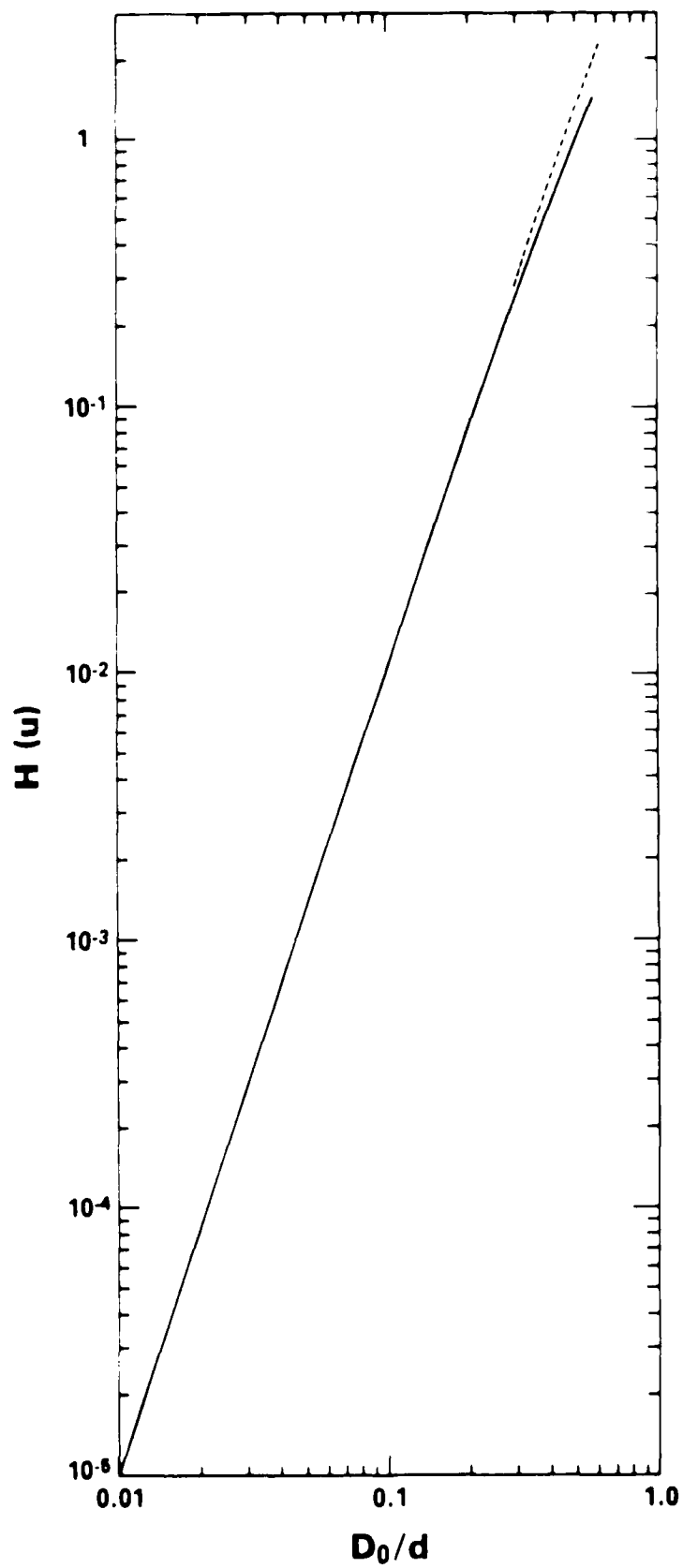
21

Figure 10. H(u)=(sinu-ucosu) vs. the Ratio of Sphere
Diameter to Diffraction Plane Spacing. The
Dotted Curve is the Straight Line
Approximation: $H(u) \approx 1/3 \ u^3$.

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# SECTION V

## ATTENUATION

Zachariasen (Reference 8) has shown that the maximum reflectance $R_{\lambda_o}$ for Bragg diffraction takes the form

$$R_{\lambda_o} = (\tanh A)^2 \quad . \tag{23}$$

In the absence of absorption,

$$A = \frac{\pi K |\Psi_H| t_o}{\lambda \gamma_o} \tag{24}$$

where $t_0$ is the depth of the wave into the crystal along a normal to the crystal surface, and $\gamma_o = \sin \theta$. Combining Eqs. 16 and 24 gives

$$A = \frac{2}{\pi(3)^{3/2}} K \left(\frac{n}{n_o}\right)\left(m^2-1\right)\left(\frac{3}{m^2+2}\right)\left(\sin u - u \cos u\right)\frac{t_o}{\sin \theta} \quad . \tag{25}$$

The transmittance $T_{\lambda_o}$ at the wavelength $\lambda_o$ may then be expressed as

$$T_{\lambda_o} = 1 - R_{\lambda_o} = \left[\cosh\left(\frac{t_o}{t}\right)\right]^{-2} \quad , \tag{26}$$

where

$$t = \frac{w_y (\lambda_o)^2 \sin \theta}{\pi (\Delta \lambda_o)} \quad . \tag{27}$$

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For normal incidence, $\underline{t}$ is calculated to be $9.42 \times 10^3$ nm for the colloidal filter of Flaugh et al. This value of $\underline{t}$ is equivalent to 48.8 layers which may be compared to the total thickness of Flaugh's filter which is 2057 layers. The transmittance may further be configured as

$$T_{\lambda_o} = \left[ \cosh\left(\frac{\ell}{L}\right) \right]^{-2} \qquad , \qquad (28)$$

where $\underline{L} = 48.8$ and $\underline{1}$ is the numbers of layers penetrated by the incident beam. Eq. 28 is plotted in Fig. 11, where the results may be seen to be consistent with the experiments of Flaugh et al. who could measure transmittance values only as low as $10^{-4}$. Also, the theory demonstrates that extremely large absorbance values are possible in colloidal filters much thinner than that of Flaugh et al.

Since the time of our original reports (References 13 and 14), Prof. Asher (Reference 16) has measured optical densities greater than eleven, in the direction of extremely large attenuation predicted by our theory.
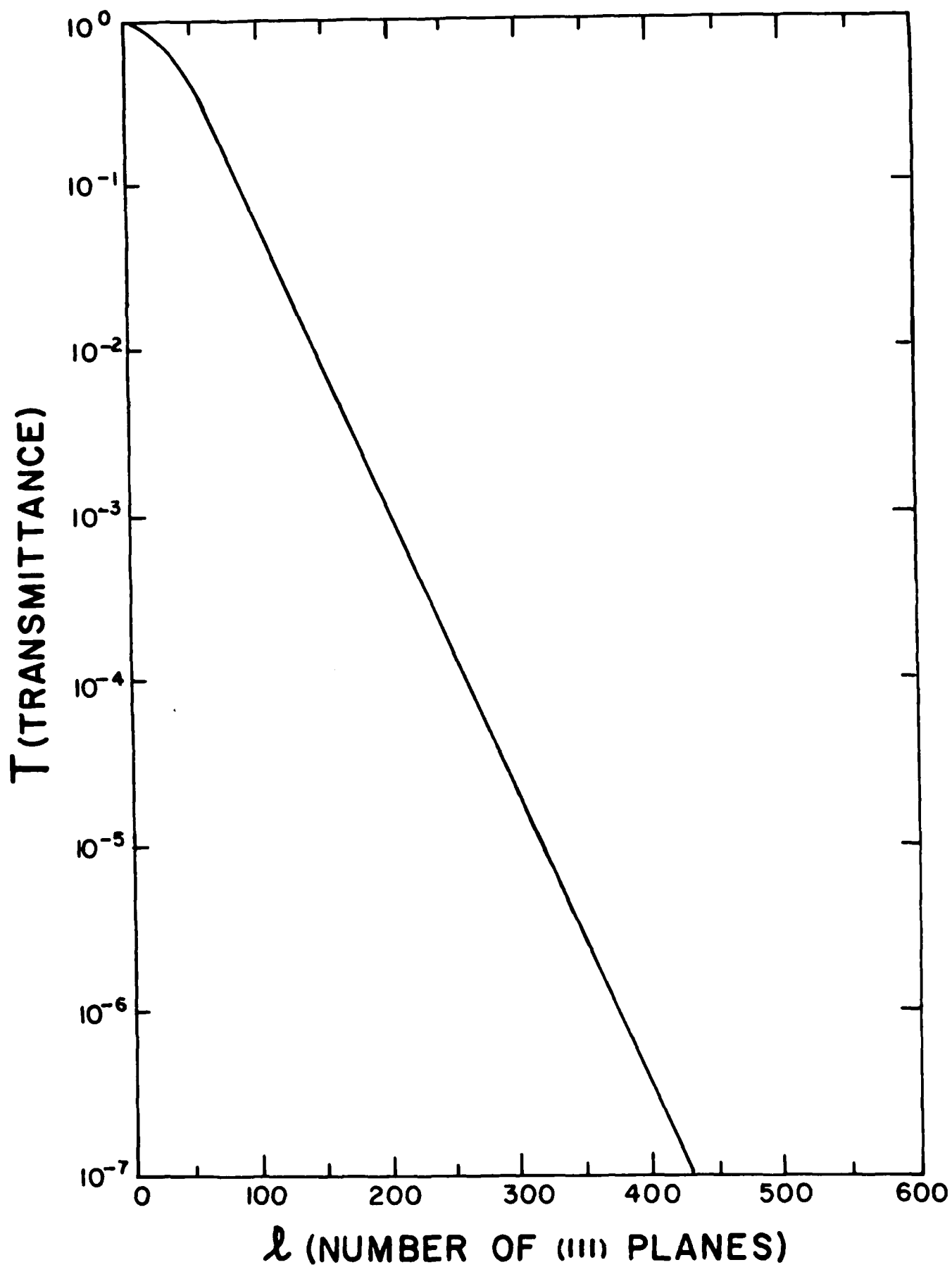
Figure 11. Theoretical Transmittance of a Crystalline Colloidal Array Filter as a Function of the Number of Crystalline Layers Traversed.

# SECTION VI

## SUMMARY

The dynamical theory of X-ray diffraction and light scattering theory have been applied to the calculation of the bandwidth and attenuation of a crystalline colloidal array optical filter. The limits of the scattering theory dependent upon particle diameter and refractive index have been thoroughly defined. The theoretical bandwidth is in excellent agreement with the previously published experimental value. Design parameters for narrower bandwidth filters are discussed.

The theoretical attenuation results are consistent with measured transmittance values. The theoretical attenuation function predicts that actual filters may be constructed that are thinner and more efficient than the first experimental ones, as has been substantiated by the most recent transmittance measurements.
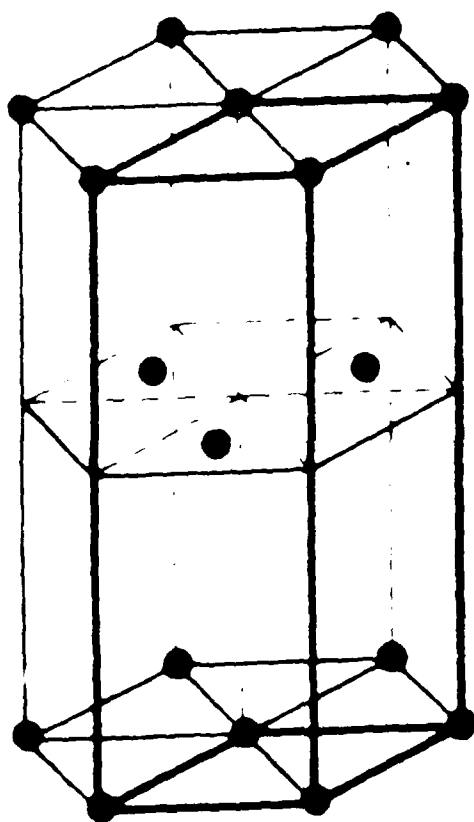
The theoretical angle shift of the rejection band maximum has also been calculated, and is in excellent agreement with experimental data.

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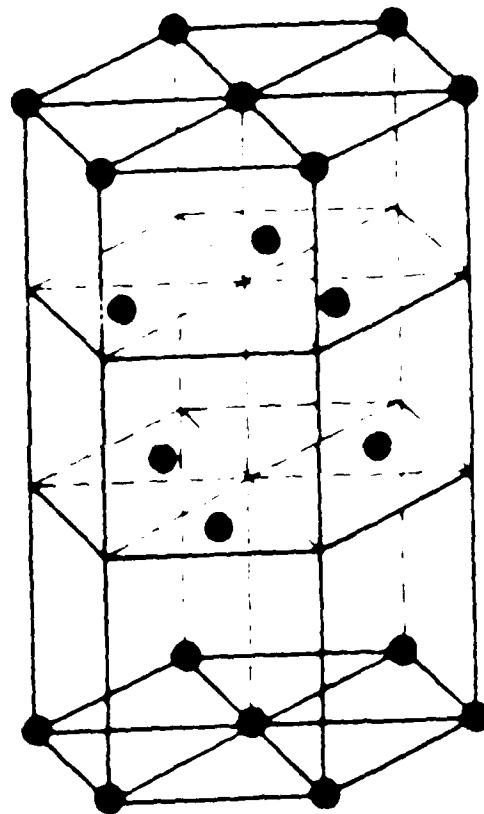
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# APPENDIX A

## ADDITIONAL CRYSTAL STRUCTURES



hcc

**Hexagonal close-packed**

fcc

**Faced-centered cubic**

Figure A-1. Close-Packed Crystal Structures. The
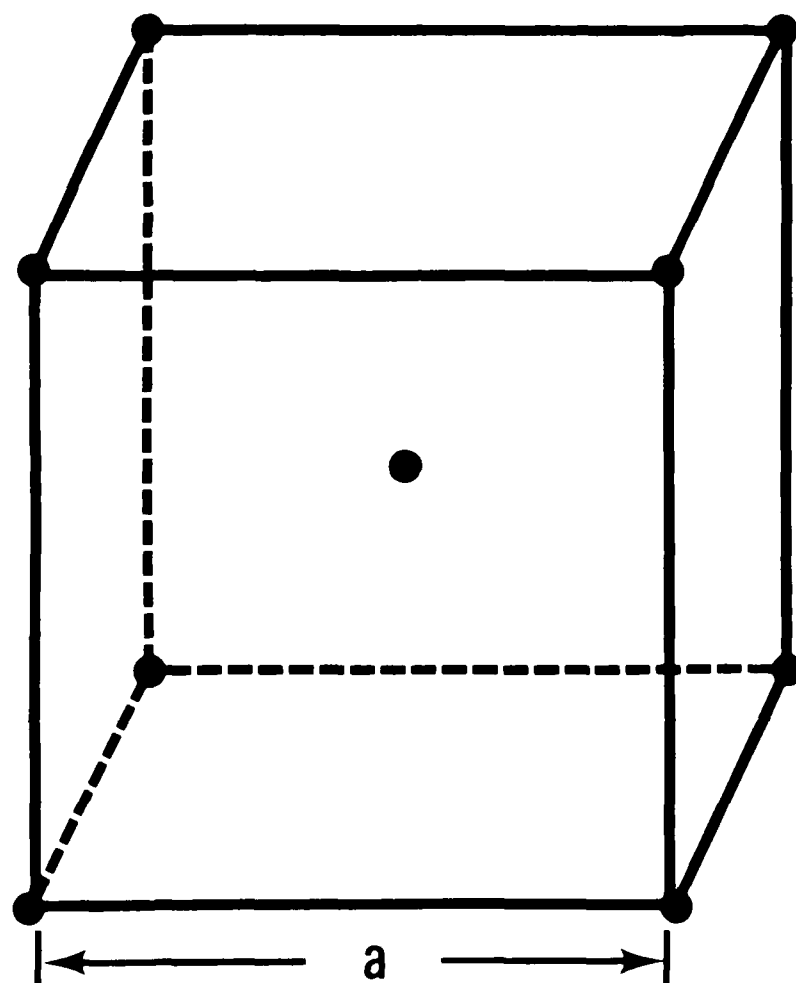(111) Axis is in the Vertical Direction.

Figure A-2. Body-Centered Cubic Unit Cell.

# END

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# DTIC