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LIMITING POTENTIALITIES OF MICROCALORIMETERS

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An analysis is made of the limiting values of the minimum detectable power for conductive microcalorimeters with thermoelastic converters.

1. Statement of the Problem

Since any energy conversion is connected with heat release, the universal character of the information obtained as a result of measurements of thermal processes dictates the wide distribution of microcalorimetric methods in investigations of the thermodynamics and kinetics of physical, chemical, and biological processes, including manufacturing processes, in medicine and other fields. Rather sensitive and high-speed apparatus for general and special purposes have been developed to these ends.

The individual parameters and characteristics of microcalorimeters and the problems of the design and optimization of the constructions have been discussed in a number of reports [1-18]. Up to now, however, different and frequently contradictory concepts have been used in choosing the main parameters characterizing the relative potentialities of macrocalorimeters. In this connection, in the light of the modern theory of measuring devices [19-22], it is desirable to start from an analysis of the noise arising primarily as a consequence of thermodynamic fluctuations. Such an approach was taken in [6] for isothermal and in [14] for continuous-flow microcalorimeters. The results of these reports provide an estimate of the minimum detectable power, but the interrelationship between this parameter and the energetic efficiency of the measuring converter being used and with the speed of response and accuracy of the instrument requires further investigation.

In the present report an analysis is made of the minimum detectable power for conductive microcalorimeters, the connection between this parameter and the speed of response is investigated, and the limiting potentialities of microcalorimeters, limited by thermodynamic fluctuations both in the instrument itself and in the recording apparatus, are determined.

The analysis of the limiting potentialities of a conductive microcalorimeter was carried out for a model (Fig. 1) which contains the following: A reaction chamber where the thermal power W_t, which varies with time, is released after being measured; a converter of thermal energy into a recordable signal, consisting of a thermoelastic battery, for example; a thermostat for the temperature T in which the reaction chamber and the converter are placed; a recorder of the microcalorimeter signal with an input resistance R_r, consisting of a linear measurement system such as a mirror galvanometer of photogalvanometric amplifier.

The calculations were made with allowance for the following limitations which occur in microcalorimeters.

1) The heat capacity of the converter is far less than the heat capacity of the reaction chamber filled with the test substance. This limitation means that the relation

$$\tau_c^0 \gg \tau_t^0. \tag{1}$$

between the time constant τ_t^0 of the converter itself and the time constant τ_c^0 of a converter having a heat capacity negligibly small in comparison with the heat capacity of the reaction chamber is valid. Since

$$\tau_t^0 = \frac{l^2}{\varkappa c\delta}, \quad \tau_c^0 = \frac{Cl}{\varkappa S}, \tag{2}$$

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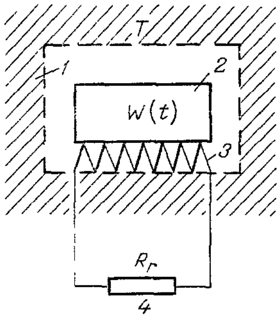

Fig. 1. Microcalorimeter model: 1) thermostat; 2) reaction chamber; 3) converter; 4) input resistance of recorder.

the condition (1) comes down to the inequality

$$C \gg C_t = cSl. \tag{3}$$

When the condition (3) is satisfied the distortions introduced by the thermal inertia of the converter are reduced to a minimum in measurements of time dependences of the thermal power. For this reason one strives to satisfy the condition (3) in the development of modern microcalorimeters [25-27].

2) The thermal conductivity of the casing of the reaction chamber is high enough so that temperature differentials along the casing can be neglected. One also strives to realize this limitation in microcalorimeters, since violation of this condition leads to an apparent increase in the heat capacity of the reaction chamber, as shown in [1].

3) The converter has a lower speed of response than the recorder. Such a limitation expresses the conditions for conformity of the speeds of response of the converter and recorder. In actual systems [25-27] the speed of response for the converter is chosen as less than but on the same order as the speed of response of the recorder.

With such limitations the microcalorimeter system presented in Fig. 1 can be represented by a model with lumped parameters, among them a heat capacity C lumped into the reaction chamber connected with the thermostat by inertialess heat removal. In this case the thermal inertia of the heat removal, possessing a thermal conductivity Λ, is allowed for as an additional term entering into C. The analysis of the processes in such a model is based on the heat-balance equation.

2. Heat-Balance Equation

The equation of heat balance at the chamber–converter contact has the form

$$W(t) = \Lambda\Theta + C\frac{d\Theta}{dt} + f(\Theta), \tag{4}$$

where $\Lambda = \Lambda_t + \Lambda_r$; Θ is the temperature difference between the reaction chamber and the thermostat; $f(\Theta)$ is the thermal reaction of the converter–recorder system to a heat flux W(t).

For a thermoelectric converter containing n thermocouples the function $f(\Theta)$ is nonlinear with respect to Θ, generally speaking, even in the case when the coefficient α of thermal emf of the thermocouples does not depend on the temperature:

$$f(\Theta) = \frac{\alpha^2 n^2 (T+\Theta)\Theta}{R+R_r} - \frac{1}{2}\cdot\frac{\alpha^2\Theta^2 n^2 R}{(R+R_r)^2}. \tag{5}$$

In Eq. (5) the first term represents the contribution of Peltier heat and the second term represents that of Joule heat. By introducing the parameter

$$z = \frac{\alpha^2 n^2}{\Lambda_t R} \tag{6}$$

of thermal efficiency of the battery and the relations

$$m = p^{-1} = \frac{R_r}{R}, \quad (7)$$

$$\mu = \nu^{-1} = \frac{\Lambda_t}{\Lambda_r}, \quad (8)$$

the function $f(\Theta)$ can be represented in the form

$$f(\Theta) = \Lambda_r \mu \left[\frac{zT}{1+m} \Theta + z \frac{1+2m}{2(1+m)^2} \Theta^2 \right]. \quad (9)$$

When the condition

$$\Theta \ll \left(1 + \frac{1}{1+2m} \right) T, \quad (10)$$

practically always realized in microcalorimetry (owing to the fact that the temperature drop in the converter is far less than the absolute temperature T at which the measurements are made), is satisfied the thermal reaction $f(\Theta)$ is proportional to Θ and Eq. (1) acquires the form of a first-order linear equation,

$$C \frac{d\Theta}{dt} + \Lambda_r \left(1 + \mu + \mu \frac{zT}{1+m} \right) \Theta = W(t), \quad (11)$$

which, for a stepwise change in W(t), gives an exponential dependence $\Theta(t)$ with a time constant

$$\tau = \frac{C}{\Lambda_r} \left(1 + \mu + \mu \frac{zT}{1+m} \right)^{-1}. \quad (12)$$

3. Minimum Detectable Power for an Ideal Converter – Detector System

Since the noise sources are lumped in different elements of the model under consideration, their combined action can be described by the equivalent power released in the reaction chamber, i.e., by the thermal input of the model. The mean value of the square of the noise power of the instrument brought to the thermal input, W_n^2, can serve as the measure of the minimum detectable power:

$$W_n^2 = W_T^2 + \eta_n^{-2} W_r^2. \quad (13)$$

The dependence $\eta(W)$ in the case under consideration has the form

$$\eta(W) = \frac{1}{WL} \cdot \frac{2}{1+2m} (1 + WL - \sqrt{1 - 2WL}), \quad (14)$$

where

$$L = (1+2m)\{\Lambda_r T [(1+m)(1+\nu)(zT)^{-1} + 1]^2\}^{-1}, \quad (15)$$

which, with allowance for (10) with $W = W_n$, gives

$$\eta_n = W_n F^{-1}. \quad (16)$$

In Eq. (16)

$$F = \frac{1}{m} \Lambda_r T \mu (zT) [1+m)(1+\nu)(zT)^{-1} + 1]^2. \quad (17)$$

With allowance for (16) and (17), Eq. (13) takes the form

$$W_n^2 = \frac{1}{2} W_T^2 (1 + \sqrt{1 + 4F^2 W_r^2 (W_T^2)^{-2}}). \quad (18)$$

From (18) it is seen that the lower limit for the quantity W_n^2 is set by the temperature fluctuations of the reaction chamber. Actually, even in the absence of noise in the converter–recorder circuit ($W_r^2 = 0$) the mean square of the noise power of a microcalorimeter is not equal to zero, but comprises

$$W_n^2 = W_T^2; \tag{19}$$

i.e., it equals the mean square of the temperature noise at the thermal input of the instrument.

The amount of temperature noise W_T^2 is determined by the expression

$$W_T^2 = 4kT^2\Lambda\Delta f, \tag{20}$$

where Δf is the effective frequency band of the recorder which, by virtue of the limitation (3), must satisfy the equation

$$\Delta f = \beta\tau^{-1} \quad (\beta \geqslant 1). \tag{21}$$

Thus, if one assumes that the converter and recorder are free of noise and do not exert any action on the reaction chamber [i.e., $\Lambda_t = 0$, $\Lambda = \Lambda_r$, and $f(\Theta) = 0$], then for such an ideal instrument

$$W_n^2 = W_0^2 = 4\beta kT^2 \frac{C}{\tau^2}. \tag{22}$$

At T = 300°K this corresponds to a power (in watts)

$$W_0 = 2.23\cdot 10^{-9} C^{1/2}\tau^{-1}, \tag{23}$$

which agrees with the equation for an ideal calorimeter given in [6].

4. Allowance for Noise in the Converter – Recorder Circuit

The noise coefficient N is introduced to describe the noise in the converter–recorder circuit,

$$N^2 = \frac{W_r^2}{W_J^2}, \tag{24}$$

where W_J^2 is the mean square of the noise power in the input stage of the recorder, produced solely by Johnson noise in the circuit. According to the theory of noise in electrical circuits,

$$W_J^2 = 16k^2T^2 \frac{m}{(1+m)^2} (\Delta f)^2. \tag{25}$$

With allowance for (24) and (25), Eq. (18) takes the form

$$W_n^2 = 2kT^2\Lambda_r (1+\mu)\Delta f \{1 + \sqrt{1 + 4N^2 (zT)^2\mu^2 p^2 (1+\mu)^{-2} (1+p)^{-2} [1+(zT)^{-1}(1+\nu)(1+m)]^4}\}, \tag{26}$$

where Δf is a function of the parameters of the converter and the recorder, subject to determination.

5. Effective Frequency Band of the Recorder

The width Δf of the effective frequency noise band for a linear recorder was calculated in [7]. For a mirror galvanometer with electromagnetic damping

$$\Delta f = R_r \frac{G}{4\psi^2} (1+p). \tag{27}$$

For a photogalvanometric amplifier in the mode of voltage compensator

$$\Delta f = R_r \frac{G}{4\psi^2} (1+p)\, v(p), \tag{28}$$

$$v(p) = 1 + \frac{KR_k}{(R_k+R_r)(1+p)}, \tag{29}$$

where $R_r = R_k + R_g$. Equations (27) and (28) allow one to represent the effective noise band for a galvanometer and a photogalvanometric amplifier in the form of a single expression

$$\Delta f = R_r \frac{G}{4\psi^2} (1+p)\, a, \tag{30}$$

where $a = 1$ for a galvanometer and $a = v(p)$ for an amplifier.

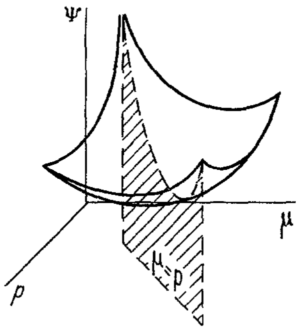

Fig. 2

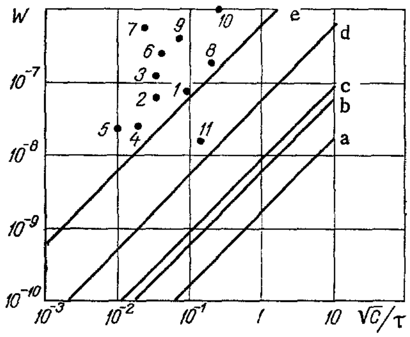

Fig. 3

Fig. 2. Dependence of the function $\Psi(p, \mu, zT, N)$ on the parameters p and μ for $a = 1$.

Fig. 3. Dependence of minimum detectable power of an optimized microcalorimeter on the parameter $\sqrt{C}/\tau$: a) ideal converter–recorder system in which only temperature noise with a power W_T acts; b) galvanometer with N = 1; c) photomultiplier with N = 1; d) F 17/1 photomultiplier; e) M-17/12 galvanometer; 1) YaK-4; 2) DAK-22; 3) DAK-12; 4) YaK-V; 5) DAK-21; 6) SRMT; 7) DAK-13; 8) YaK-2V; 9) DAK-14; 10) YaK-2B. W_n, W; $\sqrt{C}/\tau$, $J^{1/2}/K^{1/2} \cdot$ sec.

6. Optimization of the Converter and Recorder

With allowance for (30), one can write Eq. (26) for the mean-square noise power in the form

$$W_n^2 = 2kT^2\Lambda_r R_r \frac{G}{4\psi^2} \Psi(p, \mu, zT, N), \tag{31}$$

where

$$\Psi(p, \mu, zT, N) = a(1+p)(1+\mu)\{1 + \sqrt{1+4N^2(zT)^2\mu^2p^2(1+\mu)^{-2}(1+p)^{-2}[1+(zT)^{-1}(1+\nu)(1+m)]^4}\}. \tag{32}$$

From Eq. (32) it is seen that the minimum detectable power for a given recording device essentially depends on the choise of the ratio p of the electrical resistances of the recorder and converter and the ratio μ of the heat conduction of the converter to the heat conduction of radiation and of the structural elements. The character of the dependence of the function Ψ on these variables is presented in Fig. 2. For the case of a galvanometer ($a = 1$) the function (32) is symmetrical relative to the variables μ and p. As seen from the figure and Eq. (32), the function $\Psi(p, \mu, zT, N)$ reaches a minimum at some values of $p = p_0$ and $\mu = \mu_0$. These minimum values, henceforth denoted by $\Psi_0^g(zT, N)$ for the galvanometer and $\Psi_0^a(zT, N)$ for the photogalvanometric amplifier, were calculated on a BESM-4 computer. The calculated values of Ψ_0^g and Ψ_0^a for values of the variables zT and N and the parameter a realized in practice are presented in Table 1.

Thus, if a recorder with known R_r and N assigned in advance must be used in the construction of a microcalorimeter then the structural parameters of the converter must be determined from the condition of minimization of the noise level, i.e., the minimum of $\Psi(p, \mu, zT, N)$. In this case the converter parameters (such as R and Λ_t) must be calculated from the optimum values of p_0 and μ_0, which generally speaking, are not equal to unity, in contrast to the requirements of the traditional method of matching the converter and recorder [1].

The total optimization of the microcalorimetric system must be performed both with respect to the converter parameters and through the proper choice of the recorder. Two independent requirements must be satisfied in the choice of the recorder. First, the recorder must operate in the critical mode (or close to it). This condition can be written in the form

$$2R_r \frac{\sqrt{GI}}{\psi^2}(1+p_0)a_0 = 1, \tag{33}$$

TABLE 1. Values of the Functions $\Psi_0^g(zT)$ and $\Psi_0^a(zT)$

zT	$\Psi_0^g(a=1)$			$\Psi_0^a\left(a=1+\frac{1}{1+m}\right)$		
	$N=1$	$N=10$	$N=100$	$N=1$	$N=10$	$N=100$
0,1	339	$3,36\cdot10^3$	$3,36\cdot10^4$	490	$4,85\cdot10^4$	$4,85\cdot10^5$
0,3	126	$1,22\cdot10^3$	$1,22\cdot10^4$	178	$1,74\cdot10^3$	$1,74\cdot10^4$
0,5	82,5	795	$7,92\cdot10^3$	115	$1,11\cdot10^3$	$1,11\cdot10^4$
0,7	63,8	610	$6,01\cdot10^3$	88,2	848	$8,45\cdot10^3$
0,9	53,2	505	$5,03\cdot10^3$	72,9	697	$6,93\cdot10^3$
1,0	49,6	469	$4,66\cdot10^3$	65,7	643	$6,40\cdot10^3$
1,5	38,2	358	$3,56\cdot10^3$	51,1	482	$4,79\cdot10^3$
2,0	32,4	301	$2,99\cdot10^3$	42,6	399	$3,11\cdot10^3$

where I is the moment of inertia of the galvanometer loop. As seen from (33), the critical mode can always be achieved, and by various means – through the choice of the parameters R_r, G, I, and ψ and also R_k in the case of a photogalvanometric amplifier. First, according to the condition (21),

$$R_r \frac{G}{4\psi^2}(1+p_0)a_0 = \beta\frac{\Lambda_r}{C}\left(1+\mu_0+\mu_0\frac{zT}{1+m_0}\right). \quad (34)$$

Thus, for a fixed value of β the condition (33) for the critical mode and the condition (34) for making the speeds of response of the recorder and the converter conform can be satisfied jointly if the ratio $\sqrt{G/I}$ is chosen in the form

$$\sqrt{\frac{G}{I}} = 2\beta\frac{\Lambda_r}{C}\left(1+\mu_0+\mu_0\frac{zT}{1+m_0}\right). \quad (35)$$

7. Limiting Potentialities of Microcalorimeters

With the optimum choice of the parameters in accordance with the requirements of Sec. 6, the expression for W_n^2 has the form

$$W_n^2 = 2kT^2\beta\Phi_N(zT)\frac{C}{\tau^2}, \quad (36)$$

where

$$\Phi_N(zT) = \left(1+\mu_0+\mu_0\frac{zT}{1+m_0}\right)(1+p_0)^{-1}\Psi_0(zT, N).$$

From (36) for the case of W(t) = const one can obtain the following relationship between the rms relative error $\gamma = W_n/W$, the thermal power W taken from the measurement subject, and the time constant τ determining the speed of response of the instrument:

$$\gamma W\tau = \sqrt{2kT^2\beta\Phi_N(zT)C}. \quad (37)$$

As follows from Eq. (36), the value of the minimum detectable power for the given C and Λ_r can be reduced in three ways: by decreasing β, decreasing the coefficient N, and increasing the thermal efficiency z of the converter. These possibilities for reducing the noise level are limited, however,

The parameter β can be reduced to a value of $\beta = 1$; upon its further decrease the condition 3) is violated and the measurement errors start to grow owing to the nonconformity of the speeds of response of the converter and the recorder. In an extremely favorable case the noise coefficient is equal to one, which corresponds to the case when all the other noise except for Johnson noise, unremovable in principle, is absent from the the converter–recorder circuit. The thermal efficiency zT is limited by the current level of the technology of thermoelectric materials. At room temperature the best values of zT are 0.9–1.0 [24]. These limitations establish a higher level of microcalorimeter noise than that determined by Eq. (29). This level, corresponding to $\beta = 1$, N = 1, and zT = 0.9 at T = 300°K, is determined by the equation

$$W_n^2 = 2kT^2\Phi_1(0.9)\frac{C}{\tau^2}. \quad (38)$$

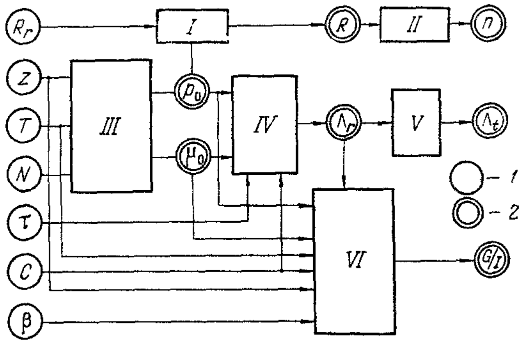

Fig. 4. Scheme for calculation of macrocalorimeter with the limiting parameters: 1) assigned parameters; 2) parameters determined as a result of the calculation; I) Eq. (7); II) Eq. (6); III) minimization of the function (32); IV) Eq. (12); V) Eq. (8); VI) the condition (35).

The results of the calculations of the limiting potentialities of microcalorimeters and a comparison of the literature data on W_n for T = 300°K are presented in Fig. 3. To the right of the line *a*, giving the dependence of the temperature noise W_0 on the quantity $\sqrt{C}/\tau$ calculated from (23), lies a region of values of the parameters fundamentally unattainable at the given temperature for a calorimeter with any converters, including a converter possessing as high a thermal efficiency as desired. In a similar way the lines b and c, in accordance with (38), bound the regions of limiting values of optimized microcalorimeters having recorders in the form of a galvanometer and of a photogalvanometric amplifier with $z = 3 \cdot 10^{-3} \,^\circ K^{-1}$. For real recorders – an F 17/1 photoamplifier and an M 17/12 galvanometer (lines d and e) – the value of W_n calculated from (38) is one and two orders of magnitude, respectively, higher than the limiting values. The values of W_n for the instruments described in [25-27] (points 1-10) are three to four orders of magnitude higher than the limiting values, which is due to departures of the construction parameters from the optimum values and to the use of recorders with high noise coefficients.

The results presented show that even an unlimited increase in thermal efficiency cannot reduce the limiting values of W_n^2 by more than two to three times. Therefore, the most urgent task of modern microcalorimetric instrument building is to decrease the gap between the minimum detectable power of existing instruments and the limiting values of W_n determined by (36) by choosing a recorder with the minimum N through conformity of the converter and the recorder and by optimizing the construction.

To design a microcalorimeter with the limiting parameters in this case one must have the assigned values of the heat capacity C of the reaction chamber, the time constant τ, the recorder parameters R_r, N, and β, the thermal efficiency z of the converter, and the working temperature T. The calculation is performed by the scheme presented in Fig. 4, as a result of which one determines the values of p_0, μ_0, R, Λ_t, n, and G/I needed to build a microcalorimeter with the limiting parameters.

A microcalorimeter (17/1 recorder–photomultiplier) with the following parameters was calculated by the given procedure and built: minimum detectable power $2.3 \cdot 10^{-8}$ W, speed of response 25 sec, volume of reaction chamber 3 cm^3 (point 11 in Fig. 3). The results of tests of the instrument show that the proposed calculation procedure, in contrast to the standard one, permits the minimum detectable power to be brought closer to the theoretical limit.

NOTATION

- a is the parameter determining the effective frequency band of the recorder;
- c is the specific heat capacity of converter material;
- C is the heat capacity of the filled reaction chamber;
- C_t is the total capacity of converter;
- G is the rigidity of galvanometer suspension;
- $f(\omega)$ is the thermal reaction of converter–recorder system to heat flux from the reaction chamber;
- F is the ratio W_n/η_n;

K is the gain of photomultiplier;
k is the Boltzmann constant;
l is the effective length of converter;
L is the parameter efficiency of the converter;
m is the ratio of resistances of recorder and converter;
n is the number of thermocouples;
N is the noise coefficient;
p is the ratio of converter resistance to recorder resistance;
R is the converter resistance;
R_r is the input resistance of recorder;
R_k is the feedback resistance of photomultiplier;
R_g is the galvanometer resistance of photomultiplier;
S is the effective cross-sectional area of converter;
t is the time;
T is the thermostat temperature;
v is the parameter determining the effective frequency band of the photomultiplier;
W_0 is the rms noise power of an ideal microcalorimeter;
W_T is the rms temperature noise power at thermal input of converter;
W_r is the rms noise power at input stage of recorder;
W_n is the rms noise power of microcalorimeter;
W is the thermal power released in the reaction chamber;
z is the thermal efficiency of converter;
α is the coefficient of thermal emf of thermocouple;
β is the constant coefficient;
δ is the density of converter material;
η is the efficiency of converter;
η_n is the efficiency of converter at $W = W_n$;
Θ is the temperature drop in the converter;
$\varkappa$ is the effective specific thermal conductivity of converter material;
Λ is the heat removal from reaction chamber;
Λ_t is the thermal conductivity of converter;
Λ_r is the heat removal from reaction chamber by radiation and through structural elements;
μ is the ratio of thermal conductivities of converter to heat removal by radiation and by structural elements;
ν is the ratio of heat removal by electromagnetic radiation and structural elements to thermal conductivity of converter;
τ is the time constant of microcalorimeter;
τ_t^0 is the time constant of a converter connected to a reaction chamber of zero mass;
τ_c^0 is the time constant of a converter with an infinitely small heat capacity;
$\Phi_N(zT)$ is the function determining the rms noise power for the optimum choice of the parameters of the converter and recorder;
ψ is the flux linkage of galvanometer loop;
$\Psi(p, \mu, zT, N)$ is the function determining the mean-square noise power.

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EXPRESSING THE COEFFICIENTS OF THE EXPANSION OF A SCATTERING INDICATRIX THROUGH MIE COEFFICIENTS

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Equations are given expressing the coefficients of the expansion of a spherical-particle scattering indicatrix by Legendre polynomials directly through Mie coefficients. The equations are converted to a form suitable for use in a computer.

In some methods of solving the equation of radiant energy transfer the scattering indicatrix for a two-phase medium is represented in the form of a series by Legendre polynomials:

$$g(\mu) = 1 + \sum_{n=1}^{\infty} (2n+1)\, g_n P_n(\mu). \tag{1}$$

The expansion coefficients g_n can be obtained from the known values $g(\mu)$ using the orthogonality properties of Legendre polynomials:

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