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[54] **DETERMINATION OF THE MEAN SIZE OF SOLID PARTICLES CONTAINED IN A FLUID BY SCATTERING OF X-RADIATION**

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[52] **U.S. Cl.** **250/43.50**

[51] **Int. Cl.** **G01n 23/12**

[58] **Field of Search**..... 250/43.5 D, 43.5 MR, 250/218; 340/237 R; 356/102, 103, 104

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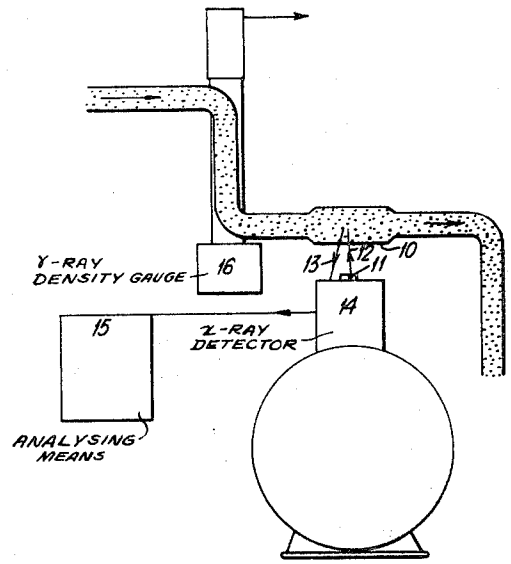
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[57] **ABSTRACT**

When electromagnetic radiation is passed through a heterogeneous material such as a slurry or suspension, part of the radiation will be scattered by the suspending medium and part by the solid particles contained in said medium. If, for example, the radiation used is X-radiation, and if the suspending medium consists of a relatively low atomic number material while the particles consist of a relatively high atomic number material, then due to the fundamental properties of scattered X-rays the bulk of the Compton or modified scattered X-rays will come from the suspending medium, and the Rayleigh or unmodified scattering will come both from the particles and the suspending medium. If the X-ray energy is suitable, the Rayleigh scattering will be independent of the size of the particles, but the Compton scattering will be markedly dependent on the size of the particles. Comparison of the Compton scattering or of the Rayleigh and Compton scattering provides a measure of the size of the solid particles.

11 Claims, 2 Drawing Figures



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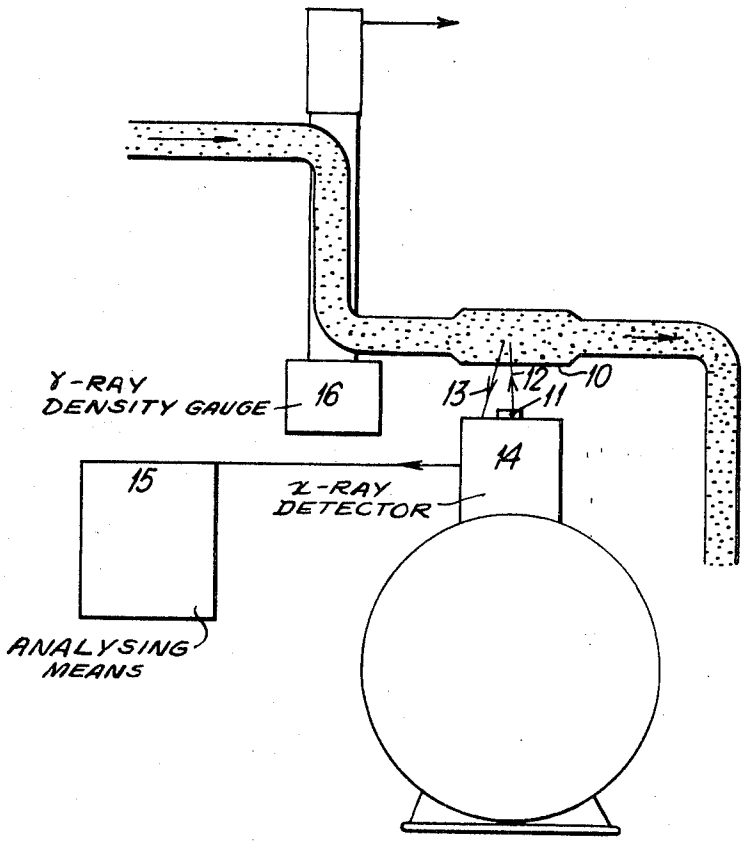


Fig. 1.

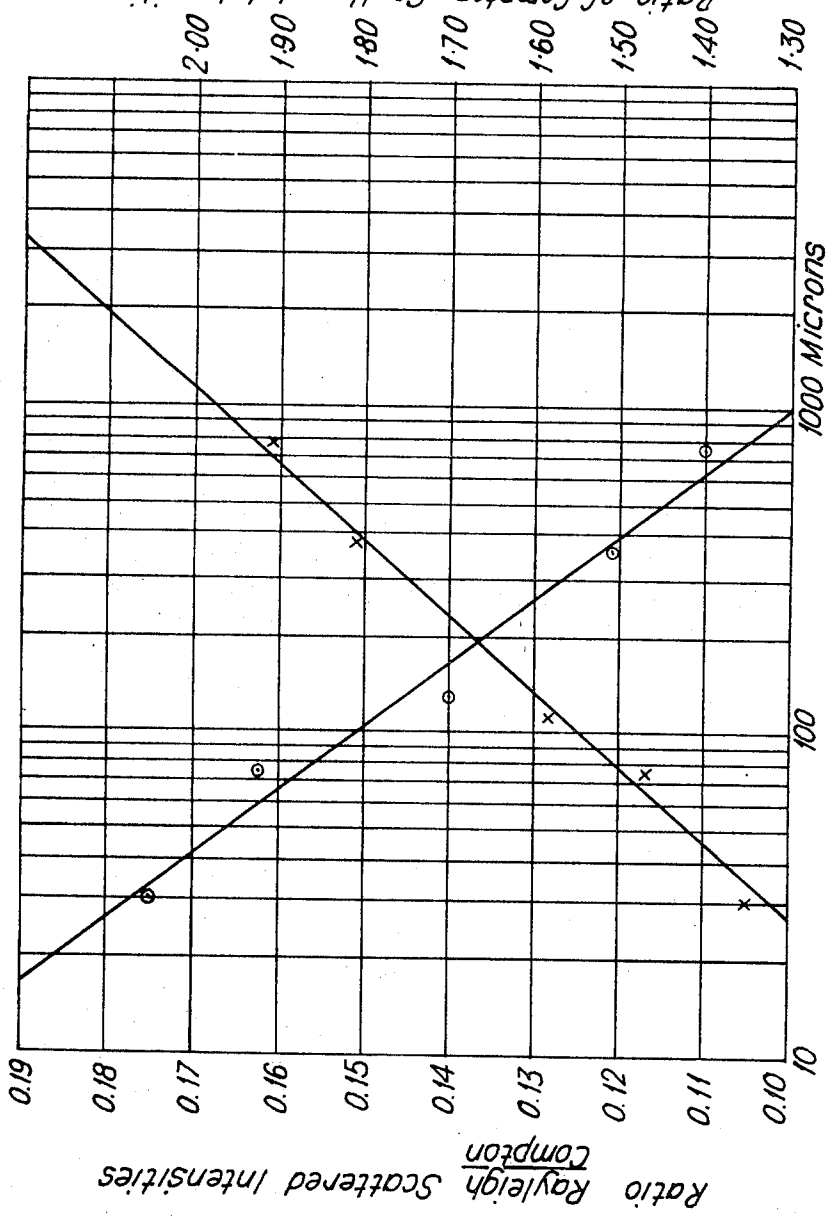
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Fig. 2



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DETERMINATION OF THE MEAN SIZE OF SOLID PARTICLES CONTAINED IN A FLUID BY SCATTERING OF X-RADIATION

This invention relates to a method of determining the mean size of solid particles of relatively high atomic number contained in a medium, particularly a fluid, of relatively low atomic number, and to an apparatus for carrying out the said method.

When X-radiation is passed through a heterogeneous material such as a slurry or suspension, part of the radiation will be scattered by the suspending medium and part by the solid particles contained in said medium. If, for example, the suspending medium consists of a relatively low atomic number material while the particles consist of a relatively high atomic number material, then due to the fundamental properties of scattered X-rays the bulk of the Compton or modified scattered X-rays will come from the suspending medium, and the Rayleigh or unmodified scattering will come both from the particles and the suspending medium.

The following simplified theory indicates the intensity of the scattered X-rays sufficiently accurately for discussion of the invention. The theory assumes that the solid particles consist of cubes of side x, arranged in layers of depth x parallel to a surface of the medium through which the radiation enters. The expression "X-radiation" as used herein is to be understood as comprehending X-rays and gamma rays.

If the solid material occupies a volume fraction V_g of the heterogeneous material then basic x-ray theory gives the following equation relating the scattered intensity from the solid material of the first layer, I_g^s to x:

$$I_g^s = K_1 I_o V_g S_g / 2\mu_g (1 - e^{-2\mu_g \rho_g x}) \quad (1)$$

where subscript g applies to the solid material

ρ = density

s = scattering coefficient

μ = x-ray mass attenuation coefficient

K_1 = geometrical factor

I_o = incident x-ray intensity at the said surface

Similarly, the scattered intensity from the suspending medium is given by:

$$I_B^s = K_1 I_o (1 - V_g) S_B / 2\mu_B (1 - e^{-2\mu_B \rho_B x}) \quad (2)$$

where subscript B applies to the suspending medium.

By combining (1) and (2) the total scattered intensity from the first layer ${}^1I^s$ will be given by:

$${}^1I^s = I_g^s + I_B^s \quad (3)$$

The scattered intensity emerging at the said surface from the second layer will be less, since both it and the incident x-rays will be attenuated by the first layer. Assuming that the probability of any x-ray being incident upon a solid or a medium component is equal to the volume fraction of that component, that the incident and scattered x-rays have the same mass attenuation coefficients and that they are both normal to the layer,

the attenuation A caused by the layer will be given by:

$$A = [V_g e^{-\mu_g \rho_g x} + (1 - V_g) e^{-\mu_B \rho_B x}]^2 \quad (4)$$

and the scattered intensity at the surface from the second layer ${}^2I^s$ will be given by:

$${}^2I^s = {}^1I^s A \quad (5)$$

and from the N^{th} layer by:

$${}^N I^s = {}^1I^s A^{N-1} \quad (6)$$

The total scattered intensity I^s can then be obtained by summing the intensities from all the layers. In practice, the intensities are summed until the scattered intensity from the N^{th} layer is less than 0.01 of the sum from $N-1$ layers. The Compton and Rayleigh scattered intensities are derived by using the appropriate scattering coefficients, assuming the scattering angle to be 180°.

It has been found that if the x-ray energy is suitable the Rayleigh scattering will be independent of the size of the particles, but the Compton scattering will be markedly dependent on the size of the particles. This discovery forms the basis of the present invention.

The present invention in one aspect provides a method of determining the mean size of solid particles of relatively high atomic number contained in a medium of relatively low atomic number the said method comprising measuring the proportion of solid particles in the medium, irradiating the medium with the solid particles therein with x-radiation such that appreciable scattering of the radiation occurs, the incident radiation including at least one component having an energy such that the intensity of the Compton scattered radiation corresponding to said component exhibits an appreciable dependence on the size of the particles, and measuring the respective intensities of two parts of the scattered radiation which differ appreciably in respect of their dependences on the size of the particles, one of said parts comprising Compton scattered radiation corresponding to said component, and the ratio of said respective intensities for the said proportion of particles in the medium giving a measure of the mean size of the particles.

The medium normally is a liquid but may be a solid, for example a paraffin wax.

Preferably, the measurements of said respective intensities are made using a common detector capable of resolving the energies of the relevant parts of the scattered radiation.

In one embodiment of the invention, the two parts of the scattered radiation whose intensities are measured respectively consist of Compton and Rayleigh scattered radiation corresponding to said one component of the incident radiation. In this case the ratio of the respective intensities, which will be referred to as the Compton to Rayleigh ratio, is dependent upon the proportion of solid particles in the medium as well as the mean size of the particles, so that to determine the latter absolutely this solid content must either be known or measured separately, for example by means of a gamma ray density gauge.

In another embodiment of the invention the two parts of the scattered radiation whose intensities are measured respectively consist of Compton scattered radiation corresponding to said one component of the incident radiation and Compton scattered radiation corresponding to a second component of the incident radiation having an energy considerably different from that of said one component. In this case the ratio of the respective intensities, which will be referred to as the Compton to Compton ratio, is substantially independent of the proportion of solid particles in the medium.

The preferred electromagnetic radiation is X-ray radiation. Moreover, it is preferred to use primary X-rays, although secondary X-rays with components having discrete energies could be used. In one practical embodiment, the source of radiation is a ^{238}Pu source which emits Uranium L radiation.

In the case where it is desired to measure the Compton to Rayleigh ratio, the L_β radiation is used, but in the case where it is desired to measure the Compton to Compton ratio, use is made of the $L_{\alpha\ 1}$ and $L_{\nu\ 1}$ radiations which are sufficiently far apart in energy for the Compton scattering from one to be particle size sensitive and from the other to be relatively particle size insensitive.

Preferably, the X-ray detector is a solid state detector, and in a preferred embodiment it is a lithium drifted silicon X-ray detector.

In another aspect of the present invention, there is provided apparatus for carrying out the method set forth above, the apparatus including a radiation source for irradiating the medium with the solid particles therein with X-radiation such that appreciable scattering of the radiation occurs, the incident radiation including at least one component having an energy such that, in operation, the intensity of the Compton scattered radiation corresponding to said component exhibits an appreciable dependence on the mean size of the particles, and means for measuring the respective intensities of two parts of the scattered radiation which differ appreciably in respect of their dependences on the size of the particles, one of said parts comprising Compton scattered radiation corresponding to said component, and the ratio of said respective intensities giving a measure of the mean size of the particles for a given proportion of the particles in the medium.

The invention will be described, merely by way of example, with reference to the accompanying drawings, in which:

FIG. 1 is a diagrammatic view of an apparatus for measuring the mean particle size of given particles in a fluid by measuring the Compton to Rayleigh or Compton to Compton scattered ratio, and

FIG. 2 is a graph of the Rayleigh-Compton ratio and of the Compton-Compton ratio of scattered intensities against mean particle size.

Referring first to FIG. 1, there is shown a cell with an X-ray transmitting window 10 past which in operation a slurry or suspension flows, the suspending medium being a liquid such as hydrocarbon which has a low atomic number, in which are suspended fine particles of an ore of tin called cassiterite which has a relatively high atomic number.

A radioisotope X-ray source 11 is provided to pass primary X-rays into the slurry. In a practical example, the X-ray source is a 30 mCi ^{238}Pu source emitting Uranium L radiation, the emitted and scattered X-rays being shown schematically by arrows 12 and 13 respectively.

To measure the intensity of the scattered X-ray radiation, there is provided a solid state X-ray detector 14. In the selected practical example, the X-ray detector 14 is a lithium drifted silicon X-ray detector having a resolution of about 320 eV at 6.4 KeV.

In order to determine the Compton to Rayleigh ratio, the detector 14 is associated with an analysing means generally indicated at 15, which may be a multichannel analyser or two single-channel analysers capable of separately indicating the intensities of Compton and Rayleigh scattered corresponding to a component of appropriate energy in the radiation emitted by the source 11; with a ^{238}Pu source this component may suitably be the Uranium L_β radiation. If desired, the outputs from the analysing means 15 may be fed to a computing device (not shown) arranged to calculate the Compton to Rayleigh ratio.

It will be appreciated from the foregoing that the ratio of the Compton to Rayleigh scattering will depend on the particle size of the heterogeneous material but it will also depend on the solid content of the system, i.e., on the amount of particles present to absorb the Compton scattering, and this must be determined separately. This can be done either by a previous measurement or it can be done substantially simultaneously by providing a gamma ray density gauge 16 located upstream of the X-ray source 11.

Referring now to FIG. 2, which shows the measurements of the Rayleigh-Compton ratio for a given solid content of the suspension with an encircled dot, it will be seen that the ratio of the Rayleigh to Compton scattered intensities plotted against particle mean size in microns follows a substantially exponential relationship, namely the greater the mean size of the particles, the smaller the said ratio.

In another preferred embodiment of the present invention, it is desired to measure the Compton-Compton ratio. In general, it will be understood that if the Rayleigh plus Compton back-scattered intensities from one relatively high and one relatively low energy X-ray source are measured, the ratio with the slurries or suspensions will not be very particle size sensitive. However, if the Rayleigh and Compton back-scattered intensities are measured separately, it is found that the Compton-Compton ratio is considerably more particle size sensitive, while the Rayleigh-Rayleigh ratio appears to be relatively particle size insensitive.

To measure the mean particle size by determining the Compton-Compton ratio, the apparatus of FIG. 1 may again be used, the analysing means being modified, if necessary, so as to be capable of separately indicating the intensities of Compton scattering for two different X-ray energies sufficiently far apart for the Compton scattering from one to be particle size sensitive and for the other to be particle size insensitive. It has been found that a suitable source is a 30 mCi ^{238}Pu source emitting Uranium L X-rays of which the $L_{\alpha 1}$ and $L_{\gamma 1}$ X-rays are suitable. In this embodiment, the gamma-ray density gauge 16 is not used.

In FIG. 2, the graph shows by a small "x" the measurements for the Compton-Compton ratio. The suspending medium was once again a suitable hydrocarbon, and the particles were once again those of cassiterite. As the graph shows, the Compton-Compton ratio

rises substantially exponentially with particle size for a given solid content.

The invention may also be employed for particle size determination when the particles are dispersed in a solid medium. For example, experimental particle size determinations have been made for galena particles dispersed in solid boric acid, and for cassiterite particles dispersed in paraffin wax.

In one such experiment galena particles were separated into size fractions by sieving, the size of each fraction being taken as the mean of the upper and lower size limits. The sized particles were suspended in boric acid at 20 percent concentration and compressed to form discs which could be readily handled. These materials were chosen to ensure maximum difference in relevant physical parameters.

The discs were irradiated at approximately normal incidence by Uranium L X-rays obtained from a sealed 30 mCi 238 plutonium source. The Compton and Rayleigh scattered X-rays were separated and their intensities measured at approximately normal take off angle with a lithium drifted silicon X-ray detector showing a half height peak width resolution in this energy region of about 380 eV. The spectra of the scattered X-rays were recorded on a 400 channel pulse height analyser and the particle sizes were determined from these records. Sufficient measurements were made with each disc to ensure that effects caused by random segregation could be neglected.

Subsequent chemical analysis of the size fractions confirmed that the results obtained were not due to variation in the composition of the grains in the different size fractions.

We claim:

1. A method of determining the mean size of solid particles of relatively high atomic number contained in a medium of relatively low atomic number, the said method comprising:

measuring the proportion of solid particles in the medium; irradiating the medium with the solid particles therein with X-radiation whereby appreciable scattering of the radiation occurs, the incident radiation including at least one component having an energy such that the intensity of Compton scattered radiation corresponding to said component exhibits an appreciable dependence on the size of the particles, and

measuring the respective intensities of two parts of the scattered radiation which differ appreciably in respect of their dependences on the size of the particles, one of said parts comprising Compton scattered radiation corresponding to said component,

the ratio of said respective intensities for the said proportion of particles in the medium giving a measure of the mean size of the particles.

2. A method as claimed in claim 1 wherein the medium is a fluid.

3. A method as claimed in claim 1 wherein the measurements of said respective intensities are made using a common detector capable of resolving the energies of th relevant parts of the scattered radiation.

4. A method as claimed in claim 1 wherein the two parts of the scattered radiation whose intensities are measured respectively consist of Compton and Rayleigh scattered radiation corresponding to said one component of the incident radiation, the ratio of the respective intensities being dependent upon the proportion of solid particles in the medium as well as the mean size of the particles.

5. A method as claimed in claim 4 wherein said proportion is determined by an γ-ray density gauge.

6. A method as claimed in claim 1 wherein the two parts of the scattered radiation whose intensities are measured respectively consist of Compton scattered radiation corresponding to said one component of the incident radiation and Compton scattered radiation corresponding to a second component of the incident radiation having an energy considerably different from that of said one component.

7. A method as claimed in claim 1 wherein the X-radiation comprises X-rays.

8. A method as claimed in claim 7 wherein the source of radiation is a ^{238}Pu source which emits Uranium L radiation.

9. Apparatus for determining the mean size of solid particles of relatively high atomic number contained in a known proportion in a medium of relatively low atomic number, said apparatus comprising:

means containing the medium with the solid particles therein and having a window transparent to X-radiation;

a radiation source for irradiating the medium through said window with X-radiation such that appreciable scattering of the radiation occurs, said source providing radiation including at least one component having an energy whereby the intensity of the Compton scattered radiation corresponding to said component exhibits an appreciable dependence on the main size of the particles, and

means for measuring the respective intensities of two parts of the scattered radiation which differ appreciably in respect of their dependences on the size of the particles, one of said parts comprising Compton scattered radiation corresponding to said component, the ratio of said respective intensities giving a measure of the mean size of the particles for said given proportion of the particles in the medium.

10. Apparatus as claimed in claim 9 wherein said means for measuring includes a solid state X-ray detector.

11. Apparatus as claimed in claim 10 wherein the detector is a lithium drifted silicon X-ray detector.

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UNITED STATES PATENT OFFICE

CERTIFICATE OF CORRECTION

Patent No. 3,749,910 Dated July 31, 1973

Inventor(s) Kenneth Garfield Carr-Brion and Susan Elizabeth Bramwell

It is certified that error appears in the above-identified patent and that said Letters Patent are hereby corrected as shown below:

In the Heading:

Item [30[Foreign Application Priority Data

October 2, 1969 Great Britain 48563/69

Signed and sealed this 23rd day of April 1974.

(SEAL)
Attest:

EDWARD M.FLETCHER,JR.
Attesting Officer

C. MARSHALL DANN
Commissioner of Patents