

Feb. 18, 1969

P. K. MEHTA ETAL

3,428,802

EXTERNAL STANDARD METHOD OF X-RAY DIFFRACTION ANALYSIS FOR  
DETERMINING THE PERCENTAGE OF COMPOUNDS IN CEMENT CLINKER

Filed April 11, 1966

Sheet 1 of 2

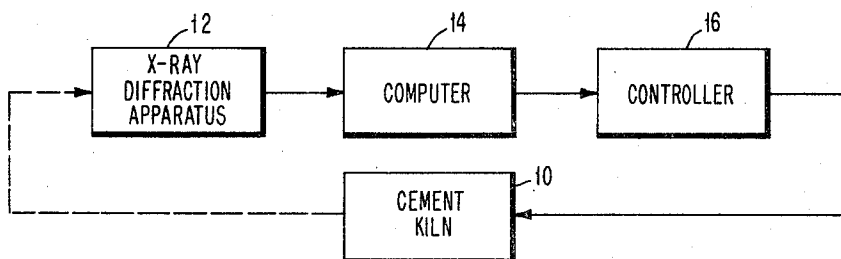


FIG. 1

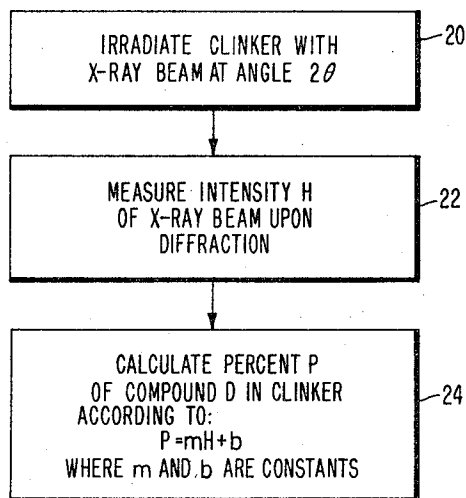


FIG. 2

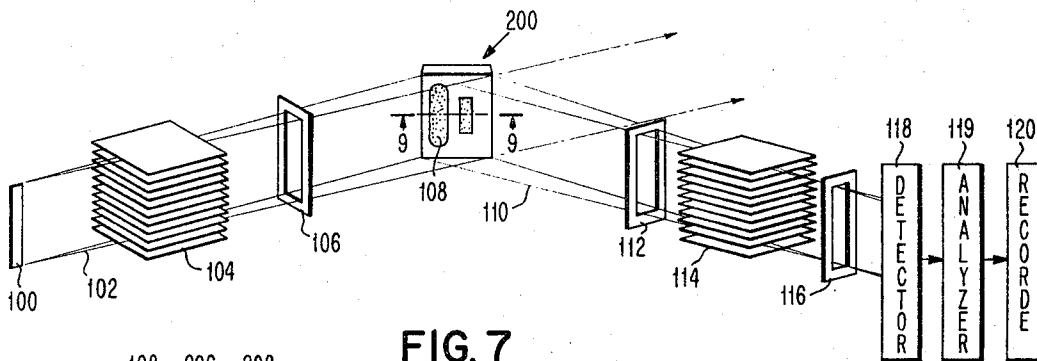


FIG. 7

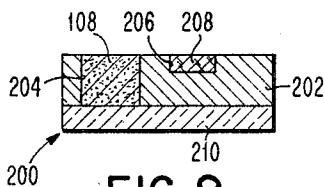


FIG. 9

INVENTORS

POVINDAR KUMAR MEHTA  
MANESH P. SHAH

BY *Edward J. McHenry*

AGENT

Feb. 18, 1969

P. K. MEHTA ETAL

3,428,802

EXTERNAL STANDARD METHOD OF X-RAY DIFFRACTION ANALYSIS FOR  
DETERMINING THE PERCENTAGE OF COMPOUNDS IN CEMENT CLINKER

Filed April 11, 1966

Sheet 2 of 2

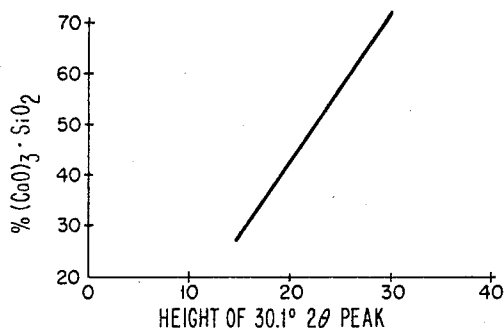


FIG. 3

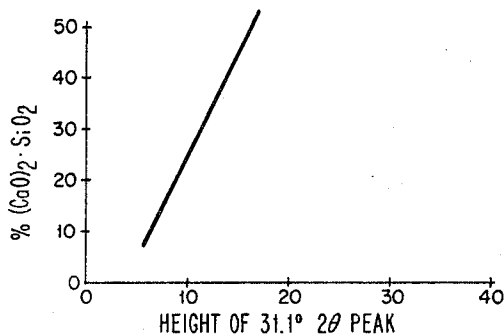


FIG. 4

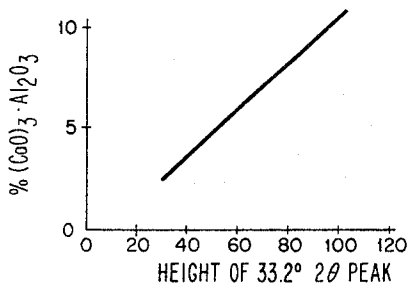


FIG. 5

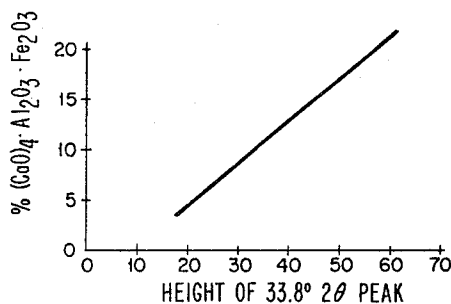


FIG. 6

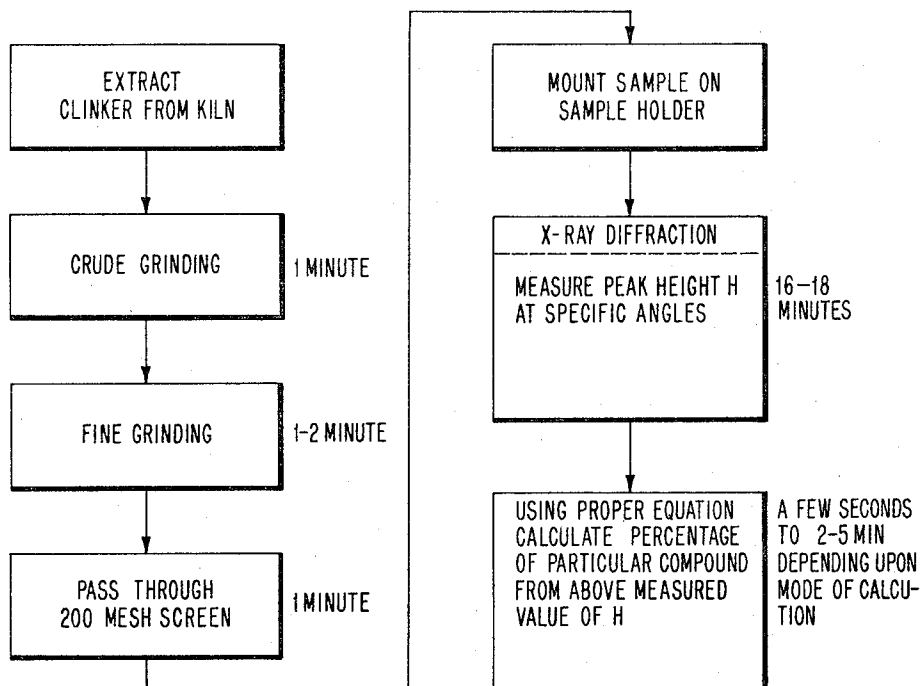


FIG. 8

1

3,428,802

## EXTERNAL STANDARD METHOD OF X-RAY DIFFRACTION ANALYSIS FOR DETERMINING THE PERCENTAGE OF COMPOUNDS IN CEMENT CLINKER

Povindar K. Mehta, Albany, and Manesh J. Shah, San Jose, Calif., assignors to International Business Machines Corporation, Armonk, N.Y., a corporation of New York

Filed Apr. 11, 1966, Ser. No. 541,822

U.S. Cl. 250—51.5

Int. Cl. G01n 23/20

6 Claims

### ABSTRACT OF THE DISCLOSURE

A method of determining the percentages of four compounds in cement clinker. The method includes an X-ray diffraction analysis coupled with a physical determination according to the formula:

$$P=mH+b$$

where:

P=percentage of compound;  $m$  and  $b$ =constants for that compound;

H=intensity of the diffracted beam.

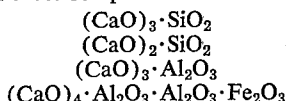
This invention relates to a method of analyzing cement and, more particularly, to the X-ray diffraction analysis of Portland cement clinker.

The manufacture of cement, although practiced for many years, is still a difficult operation.

Conventionally, cement manufacturing employs a kiln for heating the necessary raw materials. Such a kiln comprises a large, cylindrical steel shell, frequently ranging from 100 to 500 feet in length and from 8 to 15 feet in diameter. In the wet process, the necessary raw materials are generally mixed with water to form a slurry. That slurry is then introduced at one end of the kiln. In a dry kiln operation, the feed mixture is ground, dried, and then fed into the kiln. The kiln is inclined slightly with respect to the horizontal plane and revolves at a controlled rate, such as one-half to two revolutions per minute. As the kiln revolves, the feed mixture travels slowly down the length of the kiln toward the lower, or firing, end. There, an intense heat is produced by the burning of a fuel such as oil, gas, or powdered coal. In that region, the temperature of the mixture is raised to 2600–2800° F. and clinker (i.e., granular material) is formed. The clinker comprises a number of different ingredients. After exiting from the kiln, the clinker passes through a cooler, and is then available for further processing. At that stage, the clinker exists in granules ranging from approximately one-eighth to three-quarters of an inch in diameter.

The quality of the final product, as well as the profit of the manufacturer, are closely related to the degree of control which can be exerted over the manufacturing process. Cement manufacturers have been exploring the possibility of automating their plant facilities so as to increase the degree of process control available; however, for a variety of reasons, cement making processes have been extremely difficult to control effectively.

One promising way of controlling a process for manufacturing cement is to monitor the final output from the cement kiln, already referred to above as clinker. Much of the cement manufactured today has four major compounds present in the clinker. The approximate chemical composition of those compounds is:



These compounds are referred to in the cement industry by the shorthand notations  $\text{C}_3\text{S}$ ,  $\text{C}_2\text{S}$ ,  $\text{C}_3\text{A}$ , and  $\text{C}_4\text{AF}$ ,

2

respectively, and the latter notations will be employed herein for convenience.

An acceptable batch of cement clinker is produced when the first two compounds comprise roughly 80% of the clinker and the last two compounds comprise about 20% of the clinker. Any control scheme, to be effective, should be able to determine the composition of the clinker, and then adjust the process parameters accordingly so as to produce a desired clinker composition. To be truly effective, the control scheme should be fast.

Prior attempts to quantitatively determine cement clinker compounds have no proven very satisfactory. They are time consuming and also indirect, thereby yielding inaccurate results.

X-ray diffraction analysis has been attempted as one direct method of analyzing the compound composition of Portland cement; however, this has been restricted to an internal standard method of X-ray diffraction analysis. The X-ray diffraction patterns so obtained can be recorded on a strip chart, or other recording device. The pattern consists of a series of peaks (representing voltages corresponding to the intensity of the diffracted X-ray beam) which are available for qualitative as well as quantitative interpretation. The analysis of results is complicated by overlapping peaks due to different compounds.

Accordingly, it is a general object of this invention to provide an improved, rapid method of analyzing cement clinker.

A more particular object of this invention is to provide an improved method of not only identifying the compounds in cement clinker, but also calculating the percentages of each of said compounds present in the clinker.

A more particular object of this invention is to provide an improved method of quantitatively analyzing cement clinker by X-ray diffraction analysis wherein the diffraction peak for a particular compound in that clinker is so chosen as to minimize interference from other compounds in the clinker.

Still another object of this invention is to provide an improved method of relating X-ray diffraction patterns to certain equations so as to rapidly and surely analyze the compounds present in the cement clinker.

A still further object of this invention is to provide an improved method of clinker analysis which can be utilized in a closed-loop system for the control of clinker burning processes.

Briefly stated, in accordance with one aspect of our invention, we have provided a novel method of analyzing cement clinker rapidly and determining the percentage of each major compound of that clinker by utilizing diffraction techniques. In order to determine the percentage  $P$  of a compound  $D$  in cement clinker, the clinker and a standard are irradiated with an X-ray beam. The angular relationship between the incident X-ray beam and the clinker sample governs the analysis. We have determined precise angular relationships for investigating each of four major compounds in cement clinker, so that easily distinguished peaks can be obtained and correlated to the percentage of a particular compound present. The sample is irradiated with an X-ray beam incident at a specific angle as taught herein. The intensity  $H$  of the X-ray beam, upon diffraction, is measured. The percentage  $P$  of a particular compound  $D$  in the clinker can be calculated according to the relationship:

$$P=mH+b \quad (1.0)$$

where:

$$m \text{ and } b \text{ are constants} \quad (1.1)$$

The value of these constants  $m$  and  $b$  have also been discovered by us for these compounds.

The novel method of our invention provides a substantial number of advantages over the prior art, the

principal one being the vastly decreased amount of time required to analyze the clinker. It is possible, by utilizing the method disclosed herein, to determine within half an hour or less information of the type that previously, by wet chemical analysis, took twenty-four hours to calculate. In addition to the time saving aspects of the method disclosed herein, all of the precision required in the quality control of cement clinker and associated with X-ray diffraction analysis becomes available at no added cost. Thus, the analysis is not only rapid, but also definitive.

It is possible to utilize standard X-ray diffraction apparatus to perform this analysis; there is no necessity to vastly modify existing apparatus. Cost savings are thereby realized. By use of the external standard method of X-ray diffraction analysis, the necessity of intergrinding the sample with an internal standard is eliminated. It is only necessary to mount the sample of the clinker being analyzed next to a sample of reference material; irradiate both in sequence; note the results, and interpret them. Furthermore, the external standard method of our invention provides results having an accuracy equal to those obtained by the conventional, more time-consuming, internal standard method.

Since mathematical relationships have been established for each of the major compounds present in the cement clinker, it is possible to make a complete analysis of the four major clinker constituents by using the novel method of this invention. This feature becomes of increased importance when the ramifications of a closed-loop process control system are contemplated. In such a system, great economies in operation and an improved quality of the final product can be brought about, if the control system can rapidly compensate for product or process deficiencies. With an analysis method of the type disclosed herein, a closed-loop control system becomes more feasible, since the system response time is vastly improved.

The foregoing and other objects, features and advantages of the invention will be apparent from the following more particular description of preferred embodiments of the invention, as illustrated in the accompanying drawings.

FIGURE 1 is a block diagram of a control loop including a cement kiln and X-ray diffraction apparatus.

FIGURE 2 is a flow chart of the general steps used in the method of this invention.

FIGURE 3 shows a plot of the percent of  $C_3S$  present in clinker versus the peak heights on the X-ray diffraction patterns of synthetic cement clinker specimens containing different proportions of  $C_3S$ .

FIGURE 4 shows a plot of the percent of  $C_2S$  present in clinker versus the peak heights on the X-ray diffraction patterns of synthetic cement clinker specimens containing different proportions of  $C_2S$ .

FIGURE 5 shows a plot of the percent of  $C_3A$  present in cement clinker versus the peak heights on the X-ray diffraction patterns of synthetic cement clinker specimens containing different proportions of  $C_3A$ .

FIGURE 6 shows a plot of the percent of  $C_4AF$  present in cement clinker versus the peak heights on the X-ray diffraction patterns of synthetic cement clinker specimens containing different proportions of  $C_4AF$ .

FIGURE 7 shows schematically an arrangement of X-ray diffraction apparatus suitable for practicing this invention.

FIGURE 8 shows by means of a flow chart, the method of sample preparation, as well as a satisfactory testing technique, for an external standard method of X-ray diffraction analysis.

FIGURE 9 shows, in section, a sample holder suitable for utilization in the method of this invention.

With reference to FIGURE 1, cement kiln 10 is the major item in a control loop comprising X-ray diffraction apparatus 12, computer 14, and controller 16. As noted earlier, a mixture of raw materials is fired and processed

within kiln 10 in order to produce cement clinker. Samples of this clinker, after cooling, are extracted from kiln 10 for analysis. X-ray diffraction apparatus 12, in a manner more fully described subsequently, is employed to perform a qualitative and quantitative analysis of selected clinker particles; the amount of each primary compound  $C_3S$ ,  $C_2S$ ,  $C_3A$  and  $C_4AF$  can thereby be determined. The analysis results are passed onto computer 14, where they are correlated to other process parameters, such as burning zone temperature, feed rate, elemental slurry composition, etc. Computer 14 can then determine and implement whatever parameter variations are necessary through the operation of controller 16. By way of example, computer 14 may comprise an IBM 1710 or 1800 system, either of which is especially adapted to process control applications.

Before describing the group of novel curves and particular mathematical relationships utilized in our invention, the general steps of our invention will be set forth with reference to the flow chart of FIGURE 2. Box 20 indicates, as an initial step, the irradiation of a powdered clinker sample with an X-ray beam at an angle  $2\theta$  (theta), whose definition is given with reference to the description of FIGURE 3; this  $2\theta$  angle varies with the compound under investigation. An external standard material, such as silicon, is employed with the powdered clinker sample. Referring to box 22, the intensity  $H_1$  of the X-ray beam, after diffraction by the powdered sample, is then measured. Next, the intensity of the beam due to diffraction by the external silicon standard at its characteristic  $2\theta$  angle is measured. If the beam intensity (as represented by peak height) due to diffraction by the silicon has changed from a previously-established standard peak height, a correction factor  $H_2$  must be calculated. The previously-established standard peak height has been obtained by irradiating the silicon previously with the instruments adjusted in an optimum manner. The correction factor  $H_2$  then normalizes all subsequent values with reference to the previously-established standard peak height for silicon.  $H_2$  is obtained by dividing the previously-established standard peak height by the new peak height resulting from the irradiation of the silicon standard. Then, the relative intensity  $H$  for the compound under investigation in the powdered sample is given by  $H = H_1 \times H_2$ . With reference to box 24, the percentage  $P$  of a compound  $D$  in the clinker can now be calculated readily according to the equation:

$$P = mH + b \quad (1.0)$$

where:

$m$  and  $b$  are constants discovered by us. (1.1)

Equation (1.0) is that of a straight line. It results from certain observed relationships between the angle at which a sample of powdered clinker is irradiated with an X-ray beam, the intensity of the X-ray beam upon diffraction by the sample, and the percent of a particular compound present in the clinker. These relationships have been established by us. They are further illustrated in FIGURES 3 through 6 inclusive. The straight line equation for each of those figures will accordingly have different values for constants  $m$  and  $b$ , as will be described hereafter.

Looking first at FIGURES 3 through 6 inclusive as a group, it may be noted that they show a graphical representation of the novel relationships between the percent of various compounds present in the cement clinker and the results obtained from X-ray diffraction analysis of that clinker. FIGURES 3 through 6 relate to X-ray diffraction analysis using an external standard of silicon; the method of sample preparation for that technique will be described more fully with relation to FIGURE 8. Since conventional apparatus may be used to perform the X-ray diffraction analysis, a description of apparatus for practicing this invention will be postponed until reference is made to FIGURE 7. However,

it should be noted at this point that the intensity of the diffracted beam is translated into the height of an electrical signal(s) by means of, for example, a conventional scintillation counter.

With reference now to FIGURE 3, it may be used to calculate the amount of  $C_3S$  present in cement clinker when X-ray diffraction analysis with an external silicon standard is used. FIGURE 3 indicates the correlation between the peak heights generated by a scintillation counter when powdered clinker samples are irradiated with an X-ray beam a  $2\theta$  angle of approximately  $30.1^\circ$ . Angle  $2\theta$  is that angle between the undeviated incident beam and the diffracted beam. For a further discussion of this factor, see X-ray Diffraction Procedures, by Klug and Alexander; 1954; John Wiley & Sons, Inc.; pp. 117-119. For any observed peak heights, the curve indicates the percent of  $C_3S$  present in the clinker. The following equation defines the relationship between observed peak heights and the percentage of  $C_3S$  as shown in FIGURE 3:

$$P=2.85H-15.5 \quad (2.0)$$

where:

H is a value representing the observed peak heights due to irradiation of the clinker sample at the noted  $2\theta$  angle (it has been multiplied by a correction factor  $H_2$  as noted above to take into consideration any variation in peak heights due to the silicon standard); and P is the percentage of  $C_3S$  present in the clinker sample.

FIGURE 4 may be used to calculate the percentage of  $C_2S$  present in a sample of clinker when an external standard of silicon is used in the X-ray diffraction analysis of that clinker. FIGURE 4 indicates the correlation between generated peak heights (with correction factor  $H_2$ ) and the percentage of  $C_2S$  present when a clinker sample is irradiated with an X-ray beam at a  $2\theta$  angle of approximately  $31.1^\circ$ . For any observed peak heights, the curve indicates the percent of  $C_2S$  present in the clinker sample equation. The following equation defines the relationship between peak heights and percentage of  $C_2S$  that is shown in FIGURE 4:

$$P=4.0H-16.0 \quad (3.0)$$

where:

H represents the peak heights observed for the irradiation of the cement clinker at the noted  $2\theta$  angle, multiplied as explained previously by a correction factor  $H_2$ ; and P is the percentage of  $C_2S$  present in the clinker sample.

FIGURE 5 may be used to calculate the percentage of  $C_3A$  present in cement clinker when an external silicon standard is used, and the samples have been irradiated with an X-ray beam at a  $2\theta$  angle of approximately  $33.2^\circ$ . FIGURE 5 indicates the correlation between the generated peak heights and the percent of  $C_3A$  present in the cement clinker. The following equation defines the relationship between the peak heights generated and the percentage of  $C_3A$  present in the samples:

$$P=0.1167H-1.17 \quad (4.0)$$

where:

H represents the observed peak heights due to irradiation of the powdered clinker sample at the noted  $2\theta$  angle, multiplied by a correction factor  $H_2$  as explained previously; and P is the percentage of  $C_3A$  present in the clinker sample.

FIGURE 6 may be used to calculate the percentage of  $C_4AF$  present in samples of powdered cement clinker. An external silicon standard is used in the analysis. The samples are irradiated at a  $2\theta$  angle of approximately  $33.8^\circ$ . For any observed peak heights, the curve indicates the percentage of  $C_4AF$  present in the sample

under investigation. The relationships of FIGURE 6 may be set forth as the following equation:

$$P=0.4225H-3.85 \quad (5.0)$$

where:

- H is a numerical value representing the observed peak heights for irradiation of the cement clinker at the noted  $2\theta$  angle, multiplied by a correction factor  $H_2$ , as explained previously; and P is the percentage of  $C_4AF$  present in the clinker sample.

With reference to FIGURES 3 through 6 as a group once again, the general method of using them in analysis work is much the same, but the irradiation angles will vary depending upon the particular compound under investigation. For example, should it be desired to calculate the percentage  $C_3S$  present in the clinker, and should a silicon external standard be used, the graph of FIGURE 3 would be employed and the sample irradiated at a  $2\theta$  angle of  $30.1^\circ$ . The resulting peak height would then be measured and corrected, if necessary. By consulting the graph of FIGURE 3, the percentage of  $C_3S$  present could be determined readily. Alternatively, equation 2.0 could be used and the measured intensity of the X-ray beam H, as corrected, would be inserted into that equation. Then P can readily be calculated where P represents the percent of  $C_3S$  present in the cement clinker. Similarly, any of the other graphs, or any of the other equations, may be used with the requirement that the proper  $2\theta$  angles, as specified on each graph, be used during the analysis for a particular compound.

The apparatus used in practicing this invention comprises an arrangement of commercially available X-ray diffraction tools. Such apparatus was shown generally as block 12 in FIGURE 1, and will be described in more detail with reference to FIGURE 7. That figure shows schematically an arrangement of X-ray diffraction tools which can be adapted to practice this invention.

With reference to FIGURE 7, X-ray source 100 generates an X-ray beam 102 which passes through collimator 104 in order to restrict the vertical divergence of the beam. X-ray source 100 could have, for example, an input of 40 kilovolts and 35 milliamperes. Rectangular aperture 106 limits the horizontal divergence of beam 102. Aperture 106 could have, for example, an opening of 0.0006". X-ray beam 102 is thereby focused as an incident beam onto the powdered clinker sample 108, mounted in sample holder 200, shown in section in FIGURE 9.

With reference to FIGURE 9, note that sample holder 200 comprises a flat metallic plate 202 made, for example, of aluminum. Plate 202 has an aperture 204 and a groove 206. Mounted in aperture 204 is the powdered clinker sample 108, and mounted in groove 206 is a crystalline silicon standard 208. Mounted across the lower surface of a sample holder 200 is a glass slide 210.

Returning now to the description of FIGURE 7, irradiation of sample 108 by X-ray beam 102 results in the formation of a diffracted X-ray beam 110, which passes through a second rectangular aperture 112 and a second collimator 114. Beam 110 then passes through a focusing aperture 116. From focusing aperture 116, beam 110 is directed to a detector 118, whose output is responsive to the intensity of diffracted beam 110. Detector 118 may, for example, be a commercially available scintillation counter which generally comprises several transparent phosphors together with a photo-multiplied tube. The output from detector 118 provides an indication of the intensity of the diffracted X-ray beams; the beam intensities are represented by the peak heights of voltage signals generated by detector 118. The peak heights are made available from detector 118 through a pulse height analyzer 119, having a base line of 9 volts and a window of 19.8 volts to recorder 120 for storage, either transistory

or permanent. Recorder 120 may comprise, for example, a millivolt or milliamp type recording device, a punched paper tape, or an array of binary storage elements capable of providing a digital output to associated, interpretative apparatus. The measurement of the peak heights is then corrected, if necessary, and used to calculate the percent of a particular compound in clinker sample 108 according to the relationships disclosed in FIGURES 3 through 6 inclusive or the corresponding linear Equations 2.0 through 5.0 respectively.

Summarizing the description of FIGURE 7, it should be noted that the arrangement of apparatus shown is exemplary, and the method of this invention is not meant to be restricted to such a particular arrangement of apparatus. Certain substitutions and rearrangements may occur to those skilled in the art of X-ray diffraction analysis. Equipment capable of practicing this invention is, however, generally, available on the commercial market. For instance, a Norelco<sup>1</sup> X-ray Generator Type 12045B/3 may be used in conjunction with a Norelco X-ray Diffractometer Type 42202. In an operable relationship with such a diffractometer, a Norelco scintillation counter, Type 52245, may be satisfactorily employed. The noted equipment, when adjusted as described with reference to FIGURE 7, was used to obtain the curves of FIGURES 3 through 6 inclusive. Commercial equivalents of the above equipment can also be used.

In practice, the sample preparation followed for the analysis took the precaution that the preferred orientation of the crystals in the sample was held to a minimum. The method of sample preparation is described in more detail with reference to FIGURE 8.

With reference to FIGURE 8, a flow cart is set forth illustrating the major steps in sample preparation, as well as the major steps in the X-ray diffraction analysis, for our external standard method of analysis. Looking initially at the upper portion of FIGURE 8, it can be seen that a clinker sample is extracted from the kiln and then cooled. The clinker is passed through a crude grinding operation (performed either manually or mechanically) taking roughly one minute. Further fine grinding to -200 mesh is accomplished, for example, in an air swept mill; this operation takes roughly one to two minutes. The clinker sample is mounted in a sample holder, like sample holder 200 shown and described more fully in FIGURE 9. Mounted next to the powdered clinker sample is a silicon crystal, also shown more fully in FIGURE 9, which provides the external standard for the subsequent analysis.

With continued reference to FIGURE 8, it can be seen that the sample is now ready for X-ray diffraction analysis. In a preferred manner of operation, it may be desired to calculate the quantity of the four primary compounds in a continuous operation; for example, this would be desirable in the computer control of a cement kiln. The peak heights are thus measured continuously with a scanning diffractometer and recorded. It is only necessary that the sample and the standard be scanned through  $2\theta$  angles of  $35^\circ$  down to  $29^\circ$ . When a  $2\theta$  angle of  $29^\circ$  is reached, the scanning is stopped momentarily and the sample holder shifted so that the incident X-ray beam is now directed at the silicon standard. The scanning is then resumed until the angle  $2\theta$  reaches  $27^\circ$  so that the silicon peak of  $28.1^\circ$  may be noted. The various peak heights due to the four primary compounds, as well as the silicon peak height, have been noted during the scanning. The peak heights due to the four primary compounds can be corrected by a factor  $H_2$  as described before. The equations, or curves, set forth with reference to FIGURES 3 through 6 can then be used to calculate the percentage of each of the four compounds present. The entire analysis can be completed within 19 to 25 minutes.

<sup>1</sup> Trademark of North American Phillips Co.

While the invention has been particularly shown and described with reference to preferred embodiments thereof, it will be understood by those skilled in the art that the foregoing and other changes in the form and details may be made therein without departing from the spirit and scope of the invention.

We claim:

1. An external standard method of X-ray diffraction analysis for determining the percentage P of individual compounds present in cement clinker, said compounds comprising  $(\text{CaO})_3 \cdot \text{SiO}_2$ ,  $(\text{CaO})_2 \cdot \text{SiO}_2$ ,  $(\text{CaO})_3 \cdot \text{Al}_2\text{O}_3$ , and  $(\text{CaO})_4 \cdot \text{Al}_2\text{O}_3 \cdot \text{Fe}_2\text{O}_3$  and said method comprising the steps of:

irradiating said clinker with an X-ray beam at a  $2\theta$  angle characteristic of the compound under investigation;

measuring the intensity H of the X-ray beam upon diffraction; and

physically calculating the percentage P of a given one of said compounds according to an equation:

$$P = mH + b$$

where:

m and b are constants characteristic of the compound under investigation.

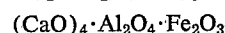
2. A method of the type set forth in claim 1 for determining the percentage P of  $(\text{CaO})_3 \cdot \text{SiO}_2$  present in said clinker wherein said  $2\theta$  angle is approximately  $30.1^\circ$ , said constant m has the value 2.85, and said constant b has the value -15.5.

3. A method of the type set forth in claim 1 for determining the percentage P of  $(\text{CaO})_2 \cdot \text{SiO}_2$  present in said clinker wherein said  $2\theta$  angle is approximately  $31.1^\circ$ , said constant m has the value 4.0, and said constant b has the value -16.0.

4. A method of the type set forth in claim 1 for determining the percentage P of  $(\text{CaO})_3 \cdot \text{Al}_2\text{O}_3$  present in said clinker wherein said  $2\theta$  angle is approximately  $33.2^\circ$ , said constant m has the value 0.1167, and said constant b has the value -1.17.

5. A method of the type set forth in claim 1 for determining the percentage P of  $(\text{CaO})_4 \cdot \text{Al}_2\text{O}_3 \cdot \text{Fe}_2\text{O}_3$  present in said clinker wherein said  $2\theta$  angle is approximately  $33.8^\circ$ , said constant m has the value 0.4225, and said constant b has the value -3.85.

6. A continuous method of X-ray diffraction analysis employing an external standard material and a powdered clinker sample for determining the percentage P of  $(\text{CaO})_3 \cdot \text{SiO}_2$ ,  $(\text{CaO})_2 \cdot \text{SiO}_2$ ,  $(\text{CaO})_3 \cdot \text{Al}_2\text{O}_3$ , and



present in said clinker sample comprising the steps of:

irradiating said clinker sample with an X-ray beam through  $2\theta$  angles of  $35^\circ$  to  $29^\circ$  inclusive;

physically noting and recording a pattern representing the beam intensities after diffraction of said beam by said sample;

irradiating said external standard material with an X-ray beam incident at the  $2\theta$  angle characteristic for said external standard material;

physically noting and recording the intensity of the X-ray beam as diffracted by said external standard material;

physically comparing the intensity due to diffraction by said external standard material to an established value of that intensity;

physically correcting said pattern for any difference present between the noted diffraction pattern for said external standard material and the established pattern for said material; and

physically calculating the percentage P of a given one of said compounds according to an equation:

$$P_{1-4} = m_{1-4}H + b_{1-4}$$

where:

P<sub>1</sub> is the percentage of  $(\text{CaO})_3 \cdot \text{SiO}_2$

$m_1=2.85$   
 $b_1=-15.5$

$P_2$  is the percentage of  $(CaO)_2 \cdot SiO_2$   
 $m_2=4.0$   
 $b_2=-16.0$

$P_3$  is the percentage of  $(CaO)_3 \cdot Al_2O_3$   
 $m_3=0.1167$   
 $b_3=-1.17$

$P_4$  is the percentage of  $(CaO)_4 \cdot Al_2O_4 \cdot Fe_2O_3$

$m_4=0.4225$   
 $b_4=-3.85$

References Cited

5 UNITED STATES PATENTS  
3,102,952 9/1963 Hendee et al. \_\_\_\_\_ 250—51.5  
3,260,845 7/1966 Duncumb \_\_\_\_\_ 250—49.58  
RALPH G. NILSON, *Primary Examiner*.  
10 A. L. BIRCH, *Assistant Examiner*.