

blocks the counter and orders transfer of the number of pulses counted to a store, resetting of the counter and the start of scanning of the store. Each figure read is immediately transferred to the punched card or magnetic tape. The reading-writing operation lasts approximately 0.3 seconds, ending 2.3 seconds after the start of the time unit.

The starting impulse for the first diffractogram-counting time unit is given by the first pip from the angle marker on the goniometer, after it changes direction.

The time units follow one another every two seconds, until arrival at the limit stop on the goniometer arm. This stop causes blocking of the counting and data-output system.

The different phases of diffraction and scattering measurements take place according to the process selected, the limits being fixed by a clock connected to the supply current, which thus controls the duration of the measurement phases.

The purpose of using two time bases, one absolute, using a quartz clock for instance, and the other relative to the current, is to ensure that despite variations in the current frequency (± 2 percent), there is both a measurement of the number of pulses counted for a given time unit, which must be independent of these variations (the effective duration of counting must be absolute) and a correspondence between the goniometer angle and the serial number of the time unit involved, which remains perfect despite these variations, in order to determine the precise angle at which the diffractogram peaks appear.

Since the velocity of the motor driving the goniometer varies with the frequency of the current, the order of the time lapses must vary in the same way, and this is why the total duration of the cycle is set to the current.

A computing programme has been drawn up, for functions culminating in the recording of mass concentrations. This involves the following operations.

A diffractogram, cleared of stray impulses, and with the tops of the peaks smoothed into parabolae, is drawn. The beginnings and ends of the peaks are determined by detecting the slope variation after smoothing of the bottom. A sub-programme allows peaks that are not completely separated to be distinguished. After subtracting the base, one obtains a precise measurement of the net surface-area of each peak.

The total number of Compton pulses is calculated after removal of stray impulses.

The surface-area of each peak is divided by the Compton measurement, giving the equation defined above

$$(C = K I_d/I_c)$$

This quotient is compared with the reference curve for the corresponding constituent element, identified by means of the abscissa at the top of its characteristic peak. The percentage finally obtained is sent to the printing machine.

Analyses using copper radiation ($K \alpha$ Cu) or molybdenum ($K \alpha$ Mo) have been carried out on samples prepared with 20 percent quartz in a wide variety of materials, ranging from lithium tetraborate ($\mu = 8\text{cm}^2 \cdot \text{g}^{-1}$) to ferric oxide ($\mu = 215\text{cm}^2 \cdot \text{g}^{-1}$) whereas, with very few exceptions, μ is between 20 and $150\text{cm}^2 \cdot \text{g}^{-1}$ for

rocks. The results obtained are at most about 2.5 away from the real value.

Analysis of the non-crystalline fraction, using the process proposed by the invention, is carried out by taking surface-area measurements of the halo for values of 2μ between 0° and 180° .

In practice, measurement from 10° to 100° is satisfactory and in some cases a much smaller range, for example 15° to 40° , is enough.

FIG. 2a corresponds to an organic material with an absorption coefficient μ of $7\text{cm}^2 \cdot \text{g}^{-1}$ and 2b corresponds to an amorphous silica with an absorption coefficient μ of $32 \text{cm}^2 \cdot \text{g}^{-1}$

Analysis of the amorphous fraction of samples containing these constituent elements is possible with an error of 30 percent, whereas earlier results were barely qualitative.

Insufficiently thick samples, which means that the thickness cannot be regarded as infinite for Compton scattering, like samples with a smaller surface-area than the sample-holder, affect the two measurements, of the intensity of diffracted energy and of Compton scattered energy, in the same proportion. This results in compensation for these effects at the stage of calculation of the percentage of constituent element in the sample, since this percentage is obtained by the ratio between the two energy intensities:

$$I_d = K' m' C / \mu \text{ and } I_c = K'' m / \mu, \text{ whereas } I_d/I_c = K''' C$$

The process and device offered by this invention can be used both for reflection diffractometry, as described earlier, and for transmission diffractometry.

What is claimed is:

1. An x-ray crystallographic process comprising, measuring the intensity of the radiation (I_d) diffracted by each constituent element of a sample, identified qualitatively by its angle of diffraction, measuring the intensity of the Compton radiation (I_c) scattered by the whole sample, and determining the content C of each element as the ratio of the intensity I_d with respect to the intensity I_c .

2. A process for analyzing the amorphous fraction of a sample, as defined in claim 1, in which the value of the ratio of I_d to I_c , namely, I_d/I_c is entered on a diagram in a continuous curve, related to the angle of diffraction, and the ratio of the mass of non-crystalline matter to the mass of crystalline matter in the sample is determined as the ratio of the surface area between the scatter halo curve and the abscissa axes to the surface area between the diffraction peaks and the same abscissa axes.

3. An x-ray crystallographic apparatus, comprising an x-ray glow-tube for irradiating a sample with an incident beam, a sample holder, and instrument to measure the intensity of the diffracted radiation, comprising a diaphragm, monochromating crystal and radiation counter, attached to a goniometer arm with the same rotational axis as the irradiated sample, means for adjusting the angle of the monochromating crystal and radiation counter in relation to the goniometer arm, an instrument to measure the intensity of the Compton scattered radiation, comprising a diaphragm, frequency-band selector and radiation counter, attached to a second arm means for adjusting the second arm in the direction of the irradiated sample to a certain angle with the incident beam, means for adjusting the angle