ters have been successfully summated by this method without difficulty. A typical application is the use of four Atcotran load cells to support a weighing platform and a weighing servo to indicate the average signal from these cells. In this way the weighing platform becomes independent of load position.

(4) Multiplication. If the input of one differential transformer is used as a source of input voltage for a second differential transformer, multiplication results. Since adequate power is not available in one transformer to drive another, the use of an electronic amplifier is recommended. See Fig. 6.

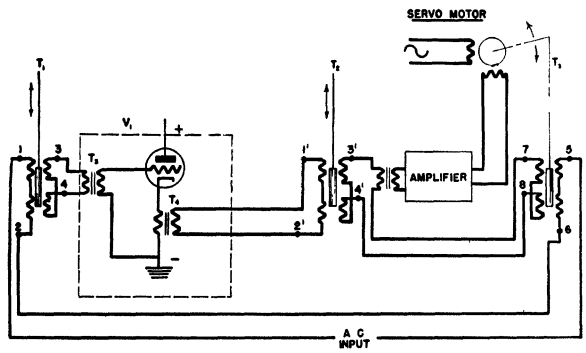

FIG. 6. Multiplying circuit.

This device has been used for compensating the output of a flow meter for changes in pressure and for other similar uses.

(5) Use of null balance circuits. In making connections between the various components of differential transformer null balance circuits, the author has standardized on the following procedures: (a) use of twisted pairs for all primary circuits; (b) use of shielded, twisted pairs for all secondary circuits, for runs over 50 feet; (c) number 20 BS gage, stranded wire is of ample size for most installations. The wires should be run in conduit but never in the same conduit with power lines.

If properly installed, transmitters and receivers may be several thousand feet apart. Special 400-cycle systems requiring only a *50 millivolt input* signal to the transmitter have been used for hazardous locations.

Although the differential transformer is still a newcomer in the instrument field, there are some standard instruments available. These include circular scale indicators with ¼% of reading accuracy (above 10% of scale) and a ten-to-one sensitivity switch; multichannel optical indicators with three automatically selected scales, a scale length of 84 in. and a 2-sec speed of response; strip chart, round chart single and multipoint recorders, controllers, etc. The switching of various input circuits into one indicator, or recorder, such as is normally done with thermocouple circuits, is standard practice with most differential transformer instruments. Transmitters, for pressure, flow, density, level, motion, etc., are also available as either standard, or custom built devices.

While it is obvious that a direct indicating, meter type of instrument may be used with a differential transformer instead of a servodriven, null balance device, no time will be devoted to these units at this time. It is only with the null balance systems that the remarkable accuracy and stability of the differential transformer can be realized.

In general, differential transformer instrument systems should be used (1) whenever highly accurate measurements of motions are required, (2) where minute forces are available and friction of any kind would be a disadvantage, (3) where electrical transmission of various variables is a requisite, and (4) where it is desired to express certain mathematical functions electrically.

Summaries of Selected Papers on Instrumentation Presented at the 1949 Gordon Research Conferences

X-Ray Powder Diffraction Analysis Film and Geiger Counter Techniques[1]

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In this note a few of the techniques and new instrumentations for X-ray powder diffraction which have been developed at this laboratory during the past six years will be briefly outlined. General descriptions of the applications of the powder method are given by Bunn (*4*), Guinier (*7*), Hanawalt, Rinn and Frevel (*9*) and Straumanis (*12*).

[1] Based on papers given at meetings of the American Society for X-Ray and Electron Diffraction, 1947–49.

In the Norelco X-ray diffraction aparatus the X-ray tube is sealed off, thus eliminating vacuum problems, vertically mounted, water cooled and full-wave rectified to aid Geiger tube work. The voltage and/or current may be continuously varied. One feature of the apparatus is that four pieces of accessory apparatus, including one or two spectrometers, can be operated simultaneously.

Powder Cameras. The design and use of Debye-Scherrer powder cameras are deceptively simple. In order to ob-

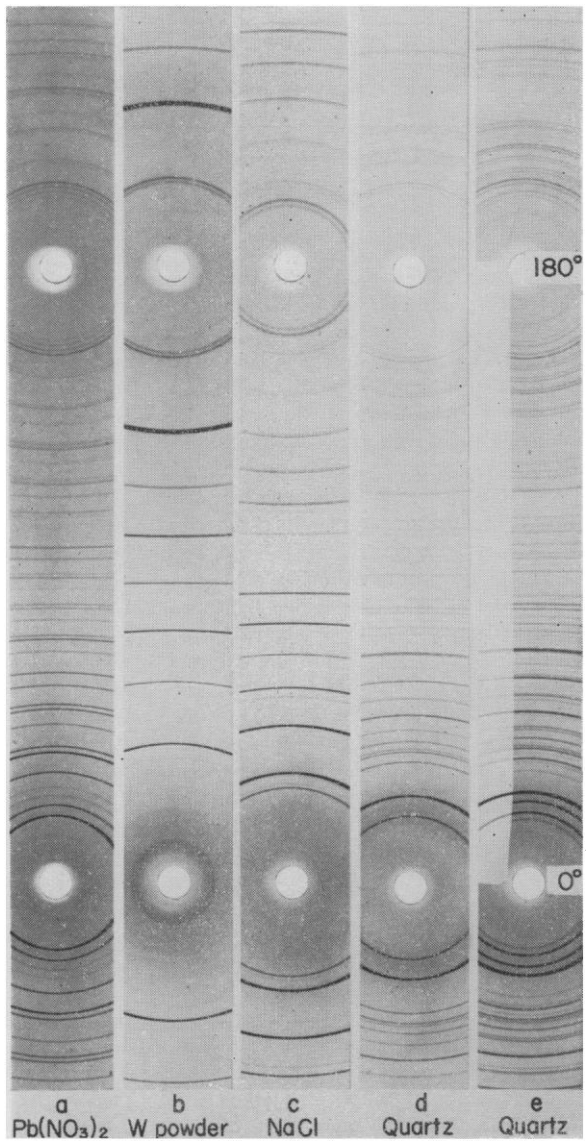

FIG. 1. X-ray powder photographs, 114.6 mm diam powder camera, Straumanis film mounting, CuK, 40 kvp, 20 ma, $\psi = 2°$. *a–d*, 0.0006-in. nickel filter, specimen mounted on 0.003-in. Lindemann glass fibre, 8 hr exposure; *e*, filter on side of film, specimen packed in Lindemann glass capillary with 0.012-in. bore, 1 hr exposure.

tain the excellent films shown in Fig. 1, certain techniques, all quite well known but not commonly practiced, are required, as much as a good camera. The mechanical design of the camera allows rapid and exact centering of the specimen and mounting of the film, exact alignment and repositioning with the X-ray tube, and easy manipulation in practice (*3*). The collimator system has been carefully computed and designed to give a minimum and almost uniform background from air scatter, no "blind" region on the film from about 5° to 175° 2θ, complete absence of scatter from metal parts of the camera or collimator, excellent line shape, and good intensity and resolution (*11*).

Precise alignment of the collimator and exit port axes with the specimen rotation axis is essential and can be tested by photographing a very thin fibre of a heavily absorbing specimen. If the alignment is accurate, the diffraction arcs will be either evenly split doublets or smooth lines of equal thickness on both sides of the 0° film hole along the equator of the film. If the collimator system is misaligned, the arc will appear as a doublet on one side of the hole and a smooth line on the other or the lines of the doublets will be unequally spaced. This can be corrected by mounting both the collimator and exit port tubes in bushings, preferably of the cone-in-cone type, whose position can be adjusted in the camera body. The adjustment is made, using a very thin glass fibre for a specimen and a magnifier fitted over the end of the exit port tube. We have found it useful to translate and rotate the specimen during exposure to obtain smoother lines and better relative intensities (*3*).

TABLE 1

PROJECTED SIZES OF 10 × 1.2 MM FOCAL SPOT

ψ	Line focus	Spot focus
2°	0.04 × 10 mm	0.35 × 1.2 mm
3°	0.06 × 10 "	0.5 × 1.2 "
6°	0.12 × 10 "	1.0 × 1.2 "

The focal spot of the new Norelco X-ray tube is 10 × 1.2 mm and the tube is constructed to allow viewing the target at grazing incidence. If ψ, the angle between the target surface and the axis of rotation of the specimen, is decreased from the conventional 6° to 3° or less, the projected size of the focal spot becomes small enough so that it can be used as the effective source slit and an actual slit is not required (*7*). The projected sizes are shown in Table 1.

The intensity as a function of ψ reaches nearly a maximum at 2°, with practically no increase at larger angles. For a given collimator with fixed divergence aperture the divergence of the beam increases as the projected size of the focal spot increases and hence the resolution decreases. The sharpest lines with maximum *peak* intensity are thus obtained at $\psi = 2°$. The divergence of the beam is limited by a circular aperture 0.5 mm diam, which in the case of the 114.6-mm-diam camera is 14 mm in front of the specimen. The full angular aperture with this arrangement is 0.4° in the vertical plane and 0.9° in the horizontal plane along the specimen axis. Although most camera tracks are set for $\psi = 6°$, the method can be applied by changing the track height and rocking the camera around the specimen axis of rotation.

Considerable care must be taken in specimen preparation (*12*). The crushed sample is passed through 200-mesh bolting cloth and is mixed with a few drops of collodion diluted 1: 10 with amyl acetate. If the material has a high absorption it is mounted on a 0.003-in. Lindemann glass fibre which is rolled in the *wet* mix. Petroleum jelly may be used if the collodion dissolves the specimen. A 0.005-in.-to-0.008-in. fibre is used for materials with moderate absorption and a capillary for ma-

terials with low absorption. The decrease in exposure time and resolution caused by using a capillary instead of a thin fibre for a quartz specimen may be seen by comparing Fig. 1d and 1e. All of these films have been purposely overexposed for the illustration and usable films may be obtained with shorter exposures.

Geiger Counter X-Ray Spectrometer. This instrument, based on the original design by the Braggs (*2*), was introduced about seven years ago and has found wide use in university and industrial laboratories where relatively accurate measurements of intensity and high resolution must be conveniently made. Its usefulness for quantitative analysis has also been demonstrated (*5*). A divergent beam of X-rays and a relatively large flat specimen 10×20 mm are used with a Geiger tube replacing the film as detector. The specimen is equidistant between the focal spot, which is the source slit, and the receiving slit in front of the Geiger tube (*1, 6*).

The X-ray optical arrangement of the newest instrument (*8*) is illustrated in Fig. 2. The line focus ($\psi = 3°$) is

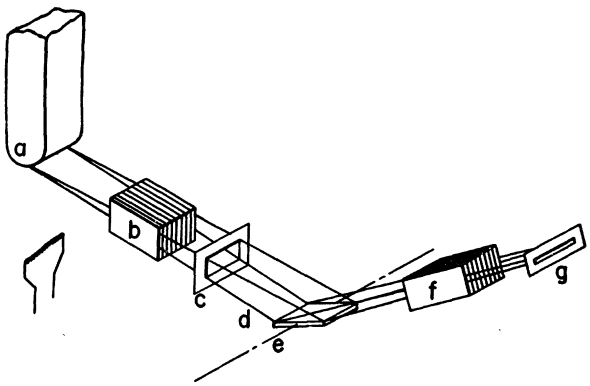

FIG. 2. Schematic X-ray optical arrangement of new spectrometer. *a*, X-ray tube line focus; *b*, *f*, Soller slits; *c*, divergence slit; *d*, Bragg focusing; *e*, specimen axis of rotation; *g*, receiving slit.

used with Soller slits (thin absorbing metal foils stacked in a parallel arrangement) to limit the divergence of the beam to a full angular aperture of 2¼° in the plane perpendicular to the plane of Bragg focusing. The best resolution is obtained with a receiving slit at least as small as the projected size of the focal spot. Scatter slits (not shown) prevent the Geiger tube from detecting radiation not scattered from the specimen. The Geiger tube arm scans in the vertical plane, the radius of the goniometer is 170 mm, and the scanning range is −38° to +160°2θ. The diffraction angles may be read to 0.01° in manual operation or angular intervals marked on the strip chart in automatic recording. The goniometer gear arrangement rotates the specimen at one-half the angular speed of the Geiger tube so that the center of the specimen is always tangent to the focusing circle. The use of a focusing arrangement gives high resolution and high intensities, since large angular apertures in the plane of Bragg focusing may be employed. For the range 20°–80°2θ the angular aperture is 1° and it is increased to 4° in the back reflection region where the intensities are very

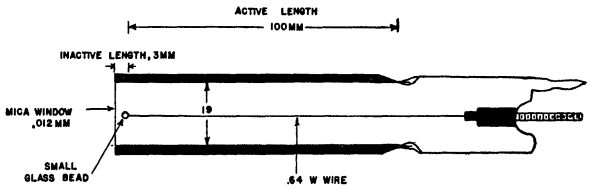

FIG. 3. Geiger counter for X-ray diffraction.

much lower. The sensitivity of the Geiger tube is so high that very small angular apertures may be used to measure large *d* spacings.

Geiger Counter Tube. The Geiger tube used for this application is shown in Fig. 3. The focused beam passes through the thin mica window along the wire anode for maximum absorption. The argon-filled counters give smoother and lower background than film (where the background is due to the shorter wavelength general radiation) because of the lower sensitivity in this region, whereas it is strongly absorbed by the film, particularly at low angles in the region of the Ag and Br absorption edges, as can be seen by broad bands around the 0° hole of the films in Fig. 1.

Electronic Circuits. A block diagram of the new circuits designed for measuring X-ray intensities with the Geiger tube is shown in Fig. 4. It is particularly important to regulate the X-ray supply in order to make correct relative intensity measurements, because the lines are scanned successively, whereas in the film method all lines are recorded continuously during the exposure.

The scaling circuit comprises eight scales-of-two which are plug-ins and are based on a modification of the conventional Eccles-Jordan type. The circuit has a resolving time of 6 to 7 microseconds (which is several times faster than currently available Geiger tubes) and will detect a pulse as small as −0.25 volt at the Geiger tube wire. For manual operation, the X-ray intensity can be measured either over a fixed time interval or the time measured to count a predetermined number of pulses. The advantage of the latter method is that the probable error (for counting alone) is the same for each measurement but a long time is required at a low counting rate.

The rate meter has a fixed full-scale range and operates from any selected stage of the scaling circuit. Therefore

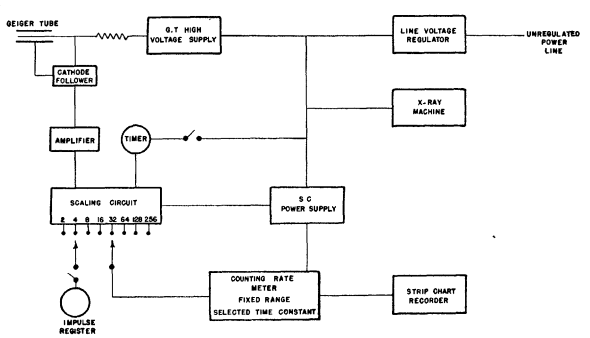

FIG. 4. Block diagram of circuits for Geiger tube operation.

each of the ranges is related to the other by an exact factor of two. A selector switch is provided to change the full-scale range from 50 to 40 or 30 cps, giving a total of 24 amplitude ranges with maximum counting rate of 12,800 cps. Five values of time constant (1 to 16 sec) are provided. The output of the rate meter can be read directly on a meter for survey work or recorded on a strip-chart recorder.

Intensity Calibration. Due to the limited resolving time of the Geiger tube and additional errors caused by automatic recording, it is necessary to calibrate the intensity measurement. This is easily done by means of a crystal plate and absorbing foils of equal thickness. The crystal plate is used in the specimen holder to monochromatize the beam. If a quartz plate (cut parallel to $10 \cdot 1$) and nickel foils (0.0007 in. thick) are used and the X-ray tube operated below 28 kvp, no difficulty will be caused by subharmonics ($\lambda/2$, $\lambda/3$, etc.) of the reflected beam. However, the calibration is accurate only for the kvp applied to the tube because the dead time of the Geiger tube measured in this way decreases with increasing kvp. Thus the difference in absorption of foils at the subharmonic wavelengths must be taken into account, or a crystal used having no reflection of higher orders.

For manual operation the intensity is measured with either the fixed count or fixed time method, depending upon the accuracy desired, for 1, 2 . . . *n* foils until the background is reached. The measured intensity in the form of counts per second is plotted on a logarithmic scale as a function of the number of foils on a linear scale. A straight line drawn through the points on the lower end of the intensity scale will then show where the measurements depart from linearity. For automatic operation the line is scanned from the same direction a few times for each number of foils and the average peak height is plotted instead of counts per second. A new calibration must be made if the scanning rate, time constant, amplitude range, or X-ray optics are changed, for these vary the measured intensity, peak position, and line shape. Unpublished work of Hamacher has shown that the peak counting rate is increased by a factor of two using full-wave rather than a half-wave rectified X-ray tube for the the same power input, and by three in operating the Geiger tube 300 v instead of 100 v above threshold.

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Two Radioactive Methods for Studying Certain Gas-Metal Reactions

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There are certain reactions between gases and metal surfaces yielding a solid reaction product which is deposited as a film over the surface of the metal. For example, the reactions between a clean aluminum surface and oxygen result in a thin oxide film spread over the metal surface. The usual method of studying the extent and rate of such film formation is the cumbersome and tedious "weight gain" technique. Two alternative methods, applicable to cases involving radioactive components, have been developed at K-25[1] for studying certain reactions of this type. These techniques, to be described, have proved extremely useful and convenient in studying the rate, the time dependence of the rate, the total magnitude of the reactions, and certain properties of the deposited films, and may have extension to other specialized investigations of similar nature.

Three criteria the particular reactions must satisfy for these methods to be applicable are: (a) The reacting gas must contain a radioactive component, (b) One product of the reaction must be a solid which adheres more or less firmly to the metal surface, and (c) The radioactive component of the gas must become a component of the solid reaction product. The number of reactions satisfying these requirements is not large, but such reactions are rather difficult to follow by regular methods.

An example of the type of reaction which may be studied by these methods is that which occurs between gaseous ruthenium pentafluoride and a clean metal surface, e.g., copper, under appropriate conditions. The pentafluoride is reduced to the nonvolatile trifluoride, which is deposited on the metal surface. The ruthenium may be obtained in a radioactive form (106) which emits beta particles at a satisfactory rate. Thus, all the stipulated requirements are met by this reaction.

Method 1. The metal involved in the gas-metal reaction to be studied is made into a Geiger-Müller or a proportional counting tube, say, 1 in. in diam and 10 in. long. A fine wire of the same metal (or alternately, of another metal inert to the gas involved), say 0.003 in. in diam, is suspended along the axis from insulators at the ends of the tube, to serve as the high voltage collector electrode of the counting tube. Suitable tubing, equipped with valves and filters, is provided for admitting and removing the corrosive gas.

The first step in studying a reaction is measuring the background counting rate, resulting from cosmic rays, contamination, etc. Then the radioactive, corrosive gas, suitably filtered, is admitted into the counting tube under the desired condition, left for a predetermined period of

[1] This work performed under contract No. W-7405-Eng-26 for the Atomic Energy Commission at Carbide and Carbon Chemical Corporation plant K-25, at Oak Ridge, Tennessee.