

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 10·1) 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 Unpublished work of Hamacher has shown that the peak counting rate is increased by a factor of two using full-wave rather than a half-wave rectified X-ray tube for the 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-25¹ 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

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reaction with the metal of the tube, say 1 min, and then pumped out. After purging the tube with inert gas and filling with some suitable counting gas, such as methane, the amount of deposited nonvolatile radioactive product left in the tube is determined by the counting rate.

The radioactive gas may then be readmitted for another exposure period, and the amount of increase in deposit subsequently determined. In this way, one may measure the rate of reaction, and the variation of the rate, with different parameters.

It is obvious, of course, that any of the radioactive gas left in the tube at the time of counting, whether chemically reacted with the metal surfaces or physically absorbed thereon, contributes to the total radioactive count. This constitutes a disadvantage in that care must be exercised to outgas thoroughly the adsorbed gas before determination of the reaction extent. This constitutes an advantage also, however, in that knowledge of physical adsorption and factors like temperature governing the adsorption, often highly desired, is easily obtained. That is, it is often of great assistance in interpreting a reaction to know how the deposited film may be removed, or how it behaves in other ways, as well as the rate at which the reaction originally proceeded. In this way the effects on deposited radioactive films of extended evacuation, high temperatures, treatment with hydrogen, fluorine, etc., have been studied.

Method 2. The second method to be described has not been used as extensively as the first. It is, in fact, still in the developmental-calibration stage, but it seems to offer certain advantages for the study of particular reactions.

The metal involved in the gas-metal reaction to be studied is made into the form of a fine wire, say 0.005 in. in diam. The wire is wound into a loose coil and mounted inside a chamber, where exposure to the radioactive gas can occur under the chosen conditions. One end of the wire extends from the chamber into a suitably arranged "withdrawal alley," through which, at chosen intervals, short lengths of the wire may be removed as samples. The wire samples, removed at successive intervals after the start of the exposure, contain on their surfaces increasing amounts of radioactive deposit, proportional to the extent of reaction occurring before their removal from the exposure chamber.

The wire samples are mounted in succession along the axis of a suitably designed Geiger-Müller or proportional radioactivity counting tube, where they serve both as the source of radioactivity being measured and the ion-collecting electrode of the tube. By measuring the activity of similar samples receiving increasingly long exposures to the corrosive gas, a history of the reaction may be obtained.

Each of the methods described has certain advantages and disadvantages. With the metal of interest in the form of a counter tube, the same metal surface is intermittently exposed to the corrosive gas, with periods of evacuation and exposure to inert counting gas between corrosive gas exposure. Presumably, the intermittency of

exposure and the effect of inert gas would have little influence on the gas-metal reaction being studied, but this would have to be established. The chief advantages of the method are: (a) the system, in contrast to the usual "weight gain" technique, does not have to be broken, with attendant likelihood of moisture contamination, etc., to permit periodic observations of the extent of the reaction; (b) once the system is set up, periodic observations of the extent of the reaction are quickly, easily, and reliably made; and (c) the exposure and counting system is easily set up, and with simple gas manifold and counter switching arrangement can handle many samples as easily as one.

With the metal of interest in the form of wire, individual samples are removed from the exposure chamber and not reused. The chief advantage is that the removal of a sample in wire form is easy to achieve without interrupting the course of the reaction on other samples left in the exposure chamber. Samples in the shape of disks, for example, would be extremely difficult to remove from an exposure chamber without interrupting the reaction, or opening the exposure chamber to ambient atmosphere; etc. Placing the wire sample in a clean, well-designed counter tube is rapidly accomplished, and gives suitable counting results.

Either of these methods, by adequate care in calibration, may be arranged to give absolute values of reaction rates. It is considerably easier to use the technique for obtaining relative values of the reaction rates between different metals and a given corrosive gas.

The most troublesome item to be encountered in the use of the methods described is the condition of the metal surfaces involved in the reaction. Extreme care in preparation of smooth, clean, uniform surfaces is necessary if reproducible data are desired.

Instrumental Methods of Studying Some Properties of Aerosols¹

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The general properties of aerosols were recently described elsewhere by the author (3). These colloidal dispersions of liquid or solid particles in a gas are the most ephemeral and elusive of colloidal systems. Hence they have received less study than liquid-dispersed systems, although they comprise clouds and smokes, dusts and bacterial spores of great economic and practical, as well as theoretical, importance. Recent developments of instrumental methods allow rapid investigation of the mass

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